FINAL PUBLISHABLE REPORT

Grant Agreement number	15SIB06
Project short name	NanoMag
Project full title	Nano-scale traceable magnetic field measurements

<table>
<tr><td colspan="2">Project start date and duration:</td><td>01 September 2016, 36 months</td></tr>
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Project website address: http://www.ptb.de/empir/nanomag.html</td></tr>
<tr><td>Internal Funded Partners:
1 PTB, Germany
2 CMI, Czech Republic
3 INRIM, Italy
4 NPL, United Kingdom
5 TUBITAK, Turkey</td><td>External Funded Partners:
6 CEA, France
7 GTU, Turkey
8 IFW Dresden, Germany
9 INNOVENT, Germany
10 ISC, Germany
11 Sensitec, Germany</td><td>Unfunded Partners:
12 SENIS, Switzerland</td></tr>
<tr><td colspan="3">RMG1: NPL, UK (Employing organisation); PTB, Germany (Guestworking organisation)
RMG2: INRIM, Italy (Employing organisation); PTB, Germany (Guestworking organisation)
RMG3: INRIM, Italy (Employing organisation); NPL, UK (Guestworking organisation)
RMG4: INRIM, Italy (Employing organisation); NPL, UK (Guestworking organisation)</td></tr>
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1 Overview

The overall goal of this project was to develop and provide coordinated and sustainable European metrology capabilities that extend reliable and traceable measurements of spatially resolved magnetic fields down to the micrometre and nanometre length scale. The project delivered traceable scanning magnetic field microscopy and magneto optical indicator film microscopy tools with spatial resolution from 50 µm down to 500 nm. Furthermore, the project delivered validated calibration technique for magnetic force microscopy calibration with spatial resolution below 50 nm together with calibration artefacts suitable for on-site calibrations. Additionally, the project heavily contributed to the development of the first international standard for traceable nano-scale magnetic field measurements.

2 Need

Macroscopic magnetic field measurements that are traceable to nuclear magnetic resonance (NMR) quantum standards and traceability chains to industry are well established. However, prior to the start of this project, these calibration chains only related to measurements of fields that were constant and homogeneous over macroscopic volumes or surface areas down to the millimetre scale. Important European high tech industries such as magnetic sensor manufacturing, precision position control and sensing in industrial applications, information technology, and bio-medical, as well as R&D laboratories required traceable and reliable measurements of magnetic fields and flux densities on the micro- or nanometre scale e.g. for quantitative analysis and quality control. Three measurement techniques: (i) scanning magnetic field microscopy and (ii) magneto optical indicator film (MOIF) microscopy to measure and image magnetic stray field distributions on the micrometre scale together with (iii) magnetic force microscopy (MFM) for nano magnetic imaging, were available prior to the start of this project , but standards for traceable calibrations for these three techniques were unavailable and forming the need for 15SIB06 NanoMag project.

3 Objectives

The overall objective of this project was to develop, test, and validate metrology tools and methods which for the first time allows reliable and traceable measurements of spatially resolved magnetic fields over the entire range from centimetres down to the micrometre and nanometre length scales.

The project’s specific objectives were:

1. To provide metrology tools and methods based on scanning magnetic field microscopy techniques suitable for traceable measurements of the local stray field distribution of permanent magnets and magnetic encoder scales with spatial resolution from 50 µm down to 500 nm; to evaluate the measurement techniques with respect to traceability and uncertainties; and to establish traceability of the local stray field measurements to macroscopic SI standards and to evaluate their uncertainties.
2. To provide metrology tools and methods based on magneto optical indicator film (MOIF) microscopy techniques suitable for traceable measurements of the local stray field distribution of permanent magnets and magnetic encoder scales with spatial resolution from 50 µm down to 500 nm; to evaluate the measurement techniques with respect to traceability and uncertainties; and to establish traceability of the local stray field measurements to macroscopic SI standards and to evaluate their uncertainties.
3. To provide validated calibration techniques to ensure SI traceability of magnetic force microscopy (MFM) with spatial resolution below 50 nm; to develop, test and validate different calibration approaches; to establish traceability to macroscopic SI standards and to evaluate their uncertainties.
4. To provide calibration artefacts suitable for traceable on-site calibrations to underpin the reliability of micro- and nano-scale traceable magnetic field measurements by end-users.
5. To facilitate the uptake of new advanced high-resolution magnetic field metrology techniques by the measurement supply chain, ensuring traceability of measurement results to the users of metrology services and to contribute to the development of standards by the international (IEC) standardisation committees concerning nano-scale magnetic measurements or nano-electronics.

4 Results

Objective 1: scanning magnetic field microscopy techniques

Objective 1 was to develop, calibrate, and validate scanning magnetic field microscopes capable of imaging magnetic features with sizes varying from the millimetre down to the sub micrometer range.

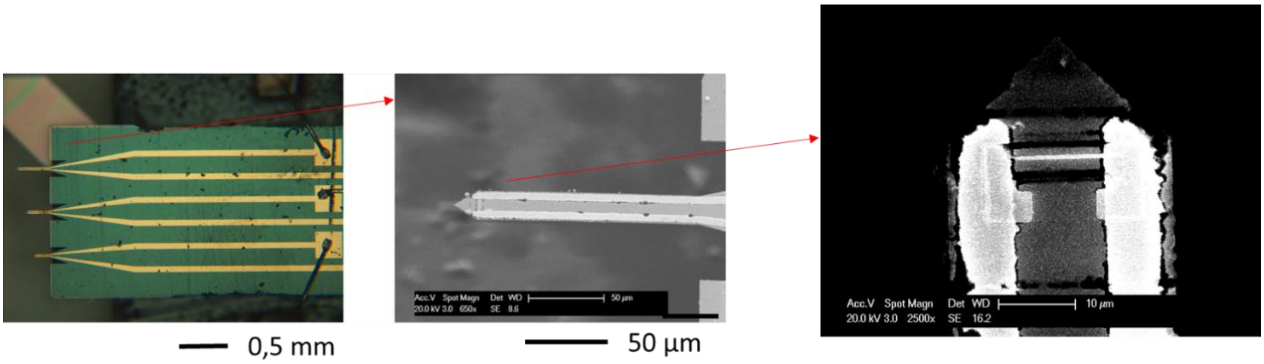
In this project, three different scanning magnetic field microscopes have been developed at three partner institutes: at CEA a scanning giant magneto resistance microscope has been developed capable of reaching the spatial resolution of 500 nm in combination with the field resolution down to 1 µT. At PTB, a scanning Hall microscope was developed suitable to image magnetic field distributions over millimeter ranges with spatial resolution down to 5 µm and 5 mT field resolution. The system relies on newly developed gold Hall sensors on Silicon Nitride (SiN) cantilevers with good long-time stability developed as part of this project and in a joint fabrication process at PTB and CEA. Additionally, the unfunded partner SENIS, has developed a prototype for a commercial nano Hall mapper, suitable to image magnetic field distributions with 1000 nm spatial resolution and large scan range of several centimeters. The system is ready to be manufactured and sold to stakeholders on demand.

All systems were calibrated in static fields. Additionally, procedures for calibrations in spatially varying fields based on suitable stray field reference samples in combination with modelling were tested and validated. Comparisons between scanning Hall probe microscopy and magneto optical indicator film (MOIF) techniques allowed cross method validation of the newly developed metrology tools.

Scanning giant magneto resistance microscope:

At CEA a scanning giant magneto resistance (GMR) microscope has been developed, tested and validated. The system can measure magnetic stray field distributions with spatial resolution of 500 nm.

A combination of UV and electronic lithography was used to fabricate 500 nm x 12 µm active size GMR sensors. Titanium gold (Ti-Au) contacts are deposited and a protective layer of 50 nm of Si_3N_4 is deposited on the GMR. The cantilevers are fabricated by a combination of back side calcium hydroxide wet etching and Reactive Ion Etching (RIE) etching Fig. 1.1).

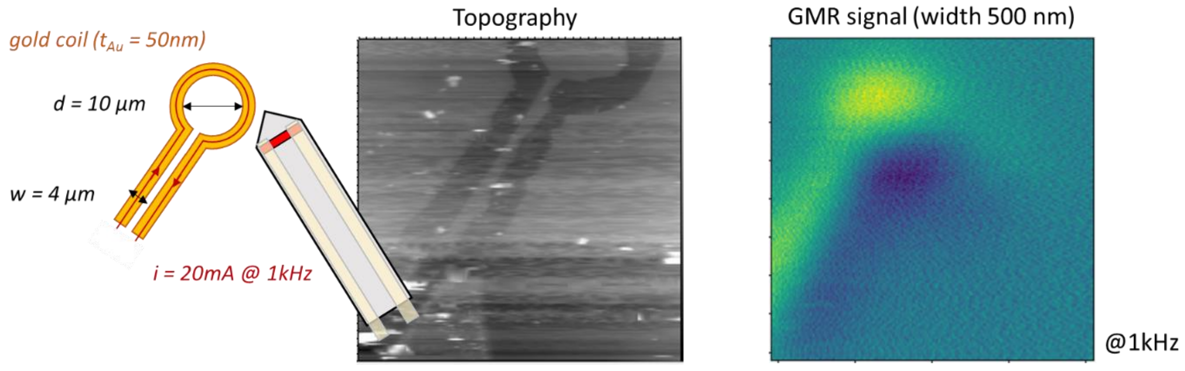


(a) (b) (c)

Fig. 1.1: Optical (a) and MEB (b, c) images at different scales (0.5 mm, 50 µm and 10 µm) of the GMR sensors integrated in flexible cantilevers of 1 µm thick Si_3N_4.

The performance of the nanoGMR has been measured in terms of sensitivity and noise level. The GMR achieved a detectivity of 1 µT at 10Hz, 400/500 nT at 100Hz and 100 µT at 1kHz. The scanning system had a scan range of 50 µm x 50 µm.

The nanoGMRs probes have been tested on micropatterned calibration coils fabricated by PTB (Fig. 1.2). The coil is fed by an AC current of 20mA at 1kHz and creates a stray field at this frequency which is measured by the nanoGMR on the cantilever. The signal of the GMR is demodulated by a lock-in amplifier to extract the magnetic signal of the coil at 1 kHz. A phase correction is applied on the magnetic images in order to get the total magnetic signal in the amplitude. The topography is obtained by the deflection of the cantilever due to the tip-sample Wan-der-Waals interactions. As the tip and the GMR are not overlapping, the topography and the magnetic image are shifted by the shift between the tip and the GMR.



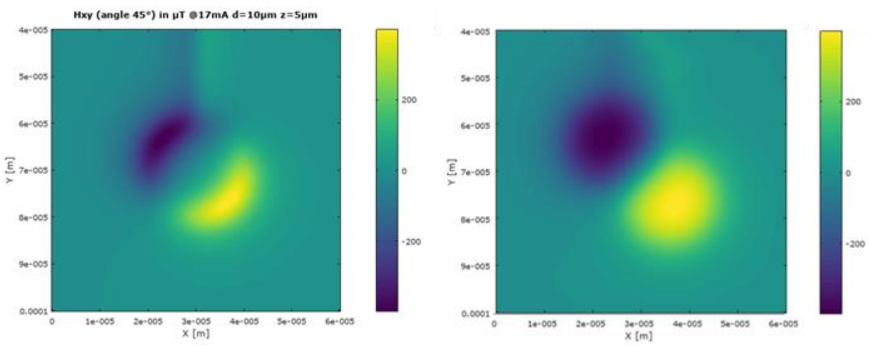
(a) (b) (c)

Fig. 1.2: (a) Schematic of the sample measured. (b) topography and (c) magnetic images (magnetic signal measured at 1 kHz) of the coil measured with the MR-SPM and the 500 nm GMR. The GMR sensitivity axis is positioned at 45° from the scanning direction. The scans size is 35 µm x 35 µm.

The lateral resolution is determined by the size of the GMR and the distance between the sample and the GMR. The width of the GMR is 500 nm determining the obtainable spatial resolution. The cantilever is tilted by 20° from the sample surface and the GMR is placed at 15 µm from the cantilever tip. Thus, the height is estimated to be 5 µm.

The lateral resolution was estimated to be well below 1 µm along one planar direction. However, smaller coils needed to be measured to verify the exact lateral resolution. The lateral resolution was obtained in the direction of sensitivity of the nanoGMR.

Simulations of the coil stray field have been performed by using Octave software (Fig. 1.3). A convolution of the lateral size of the GMR has been performed (Fig. 1.3b). The simulation results are in good agreement with the magnetic image.



(a) (b)

Fig. 1.3: Simulations of the stray field emitted by the coil. The field amplitude is given by the field projection on the diagonal of the image, which is the sensitive direction of the GMR sensor. (a) Raw data. (b) Convolution by the lateral size of the GMR. The field amplitude scale is in µT.

Scanning Hall probe microscope

At PTB a scanning Hall probe microscope (SHPM) has been developed, tested and validated. The system can measure magnetic stray field distributions with spatial resolution of 5 µm. The system has been cross validated by comparison with magneto optical indicator film microscopy.

Fabrication of Hall sensors

The Hall sensors having small and cross shaped active areas from 5 x 5 µm² down to 50 x 50 nm² were fabricated using electron beam lithography at PTB. For the gold crosses (Fig. 1.4 a) two lithography steps were needed. The first is used to define the lateral dimensions of the active area and to deposit 5 nm of titanium and 30 nm of gold as the active material by electron evaporation on a silicon substrate with a silicon nitride layer on both sides. Within the second step an additional 50 nm thick layer of gold is added in the lengthy contact region. After this, the Hall sensor fabrication cantilever chips were manufactured out of the silicon wafer (Fig. 1.4)**Error! Reference source not found.**. At CEA the cantilever shape was defined by an aluminium mask then the bottom layer of silicon nitride was removed by RIE. The silicon in the cantilever region was thinned down with wet etching. Cantilever release was performed with RIE of the top silicon nitride layer. Thereby, the Hall sensors were positioned as close as possible to the tip of the cantilever. The geometrical dimensions are visualized in Fig. 1.4 b) and c).

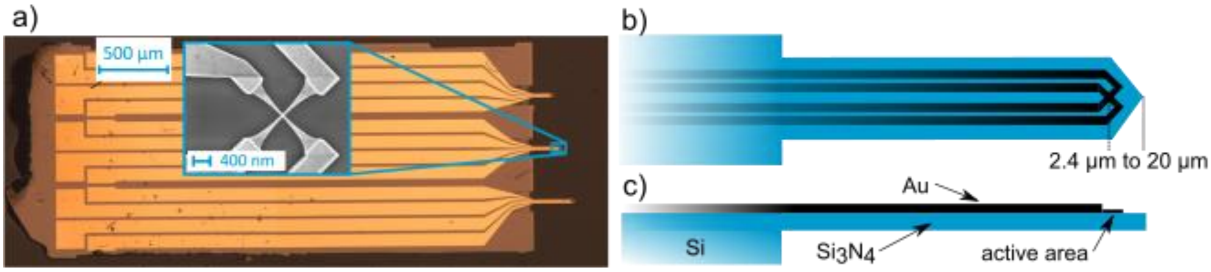


Fig. 1.4: (a) Microscopy image of a 3.4 mm x 1.5 mm cantilever chip with three gold Hall sensors for integration into commercial SPM systems. The inset shows the scanning electron microscopy image of a 50 nm gold Hall cross. (b) Top view sketch of Hall sensor on cantilever. The distance between active area and triangular tip of the cantilever depends on the sensor size, smaller sensors can be positioned closer to the tip. (c) Side view of cantilever. Dimensions are not true to scale; the cantilever is 1 µm thick.

Calibration of Hall sensors

For the Hall sensor calibration an electromagnet was used which can create a magnetic flux density of up to 450 mT. During the calibration, the magnetic flux density was simultaneously measured with a commercial reference Hall probe thus ensuring traceability to the SI. The Hall voltage was measured as a function of the magnetic flux density and corrected by the offset. The sensors typically showed a linear dependence of the Hall voltage V_{Hall} on the magnetic flux density B as expected from $V_{\mathrm{Hall}} = \frac{I*B}{n*e*t}$ where I is the supply current, n is the electron density, e is the charge of an electron and t is the thickness of the active layer. For 5 µm gold Hall sensors operated at 10 mA the output is in the micro volt range for a magnetic flux density in the order of a few tenths of milli tesla. This led to a sensitivity of 3.2 mV/AT ± 0.3 % with low long-term drifts below 0.6% over several months.

Scanning Hall probe microscope

SHPM is realized by integration of the manufactured cantilever chips with gold Hall sensors into a commercial scanning probe microscope. The Hall sensors are positioned at the bottom side of the cantilever and close to the cantilever tip to achieve a small distance between sensor and sample. This is important for the measurement of nanostructures due to the exponential decay of stray fields with increasing distance to the sample surface. Furthermore, it optimizes the lateral resolution. The SHPM measurements shown here were executed with the scanning system Nanoscope IIIa. To increase the scan area into the millimeter range additional piezo tables were added to the setup. The cantilever holders of this device have an angle of 10°. This led to a minimal measurement height of 400 nm for the 50 nm sensors and 3366 nm for the 5 µm ones. The same devices used during the calibration were utilized for the Hall sensor operation. A commercially available magnetic scale SST250HFA-04 from Sensitec was chosen as test sample. It is made of a wet pressed strontium ferrite having a remanence magnetization of 395 mT. The material was magnetized into stripes with a width of nominally 250 µm.

SHPM measurement data in combination with MOIF data and simulated field data of the magnetic scale matched well regarding the uncertainties and the fact that the datasets are not from the exact same position on the scale, thereby cross validating the calibration of the two techniques (Fig. 1.5).

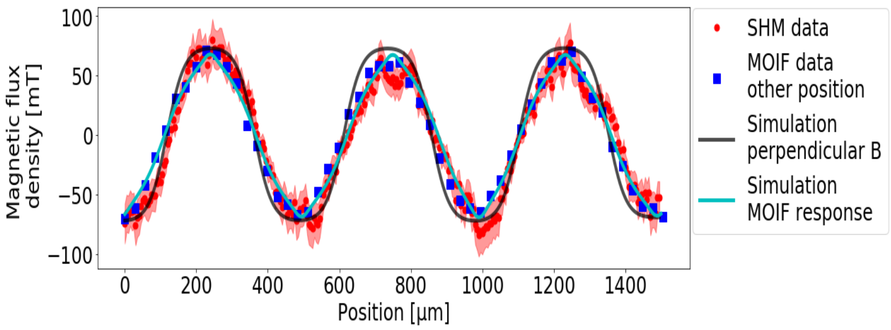


Fig. 1.5: Traceable SHPM data with uncertainty budget of scale SST250HFA-04 at a measurement height of 49 µm compared to simulated stray field values and to measured data using a MOIF device. The uncertainty of the MOIF data is ± 2.5 mT which is smaller than the data point size.

Commercial Nano Hall Mapper

At the unfunded partner SENIS a commercial nano Hall mapper has been developed which is available since May 2019 for sale to interested customers (http://www.senis.ch/products/mappers/nano-magnetic-field-mapper).

The system features a compact 2-axis Hall probe (By, Bz) with field sensitive volume of 10 µm x 10 µm x 10 µm. The measurable field range is: +/-500 mT with a minimum field resolution better than 5 µT and a probe positioning resolution around 100 nm. This system addresses the stakeholders need of fast, robust, and versatile calibrated high-resolution scanning field systems for multipurpose applications.

Conclusion

By the end of the project three complementary scanning magnetic field microscopes have been developed and set up by different partners. At CEA a high-resolution scanning giant magneto resistance microscope has been developed allowing to reach highest spatial resolution down to 500 nm. At PTB, a scanning Hall probe microscope has been developed demonstrating spatial resolution down to 5 µm over millimeter scan areas. The system is based on Hall probes fabricated in a joint clean room fabrication process by CEA and PTB. Furthermore, the nano Hall mapper prototype developed by SENIS allows spatial resolution down to 10 µm and robust customer operation over centimeter scan ranges. The latter system is ready for commercialization by SENIS. All systems have been validated and traceably calibrated and validated. Here, the cross validation relied on a joint effort of different Partners and the use of the different high-end magnetic field metrology tools. The project thus achieved the targeted resolution and completed the objective which heavily contributes to the European metrology landscape by providing calibrated field imaging form the centimeter to the sub-micrometer scale.

Objective 2: magneto optical indicator film (MOIF) microscopy

Objective 2 was to develop and validate MOIF microscopy. Among other microscopic measurement techniques, MOIF has the advantage of sub-micron resolution in combination with fast image acquisition. Magneto optic systems have been used in several labs around the world for more than two decades, however the metrological basis to the MOIF technique was not fully established. Both the traceability to the primary magnetic field standards and validation of the existing systems were not completed. Besides, some issues connected with the physical nature of MOIF sensors still needed to be addressed before the start of this project

In this project, four MOIF systems in three EURAMET member institutes, namely PTB, INRIM and TUBITAK and one externally funded partner, INNOVENT, Jena, were set up or further developed. The existing calibration procedures were made more precise and have been validated for the first time, the measurement sensitivity and uncertainty were improved, the spatial resolution was pushed down to the optical resolution limit and the magnetic field range was expanded. Comparisons between different systems and with other measurement techniques were completed thereby validating the newly developed MOIF calibration techniques.

Principe of operation

MOIF sensors constitute a Bismuth doped Yttrium Iron Garnet (YIG film grown by liquid phase epitaxy on a Gadolinium Gallium Garnet (GGG) substrate). Both GGG and YIG are transparent in the visible light range and YIG results in a strong Faraday rotation of polarized light passing through the film (Bismuth is added to enhance the Faraday effect). Without an applied external field, the magnetization vector in the MOIF lies within the film plane, not resulting in any Faraday effect. Under a normal external field, the magnetization vector turns away from the film plane and its normal component produces Faraday rotation which can be observed in a polarizing microscope(Fig. 2. *2.1*).

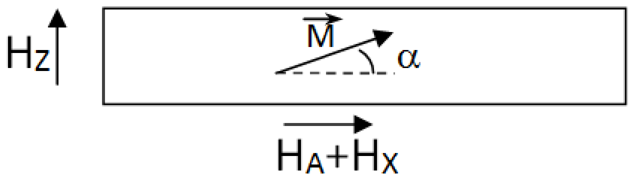


Fig. 2.1: Rotation of magnetization vector in MOIF under an external magnetic field (H_A is the anisotropy field of the MOIF and H_Z and H_X are the components of the external field)

If a MOIF sensor is placed on top of an inhomogeneously magnetized sample, its magnetization will rotate differently at each point according to the local stray field produced by the sample. Due to the Faraday effect, this inhomogeneous magnetization pattern can be observed in a polarizing microscope and can be quantified by measuring the light intensity. Thus, a quantitative distribution of the local stray field can be reconstructed after a calibration procedure.

High quality MOIF sensors with thicknesses of 0.22, 0.35, 0.66, 1.35, 2.72, and 4.5 µm were developed and produced by INNOVENT. MOIF properties such as saturation magnetization M_S, anisotropy field H_A, coercivity field H_C, Faraday rotation as a function of the applied field, and their temperature dependences were measured by INNOVENT and PTB. These parameters are required for the quantitative reconstruction of magnetic field distribution from the light intensity maps. It was observed that the MOIFs have a small, almost negligible anisotropy within the plane which needed to be considered for more accurate determination of the local field strength (Fig. 2.2). Measured dependences of the Faraday rotation on the applied normal field (Fig. 2.3) were used in some experimental setups (see below) for the direct calibration of the measured signal into the magnetic field strength. Besides, the saturation field found from these curves determines the operation field range for the MOIFs used.

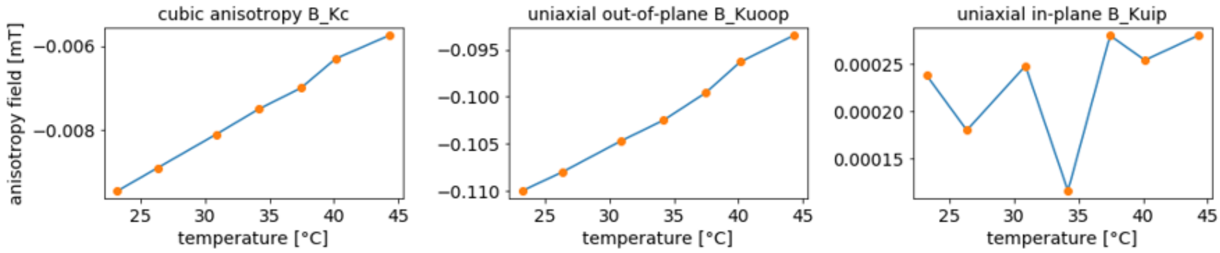


Fig. 2.2: Temperature dependence of the anisotropy fields.

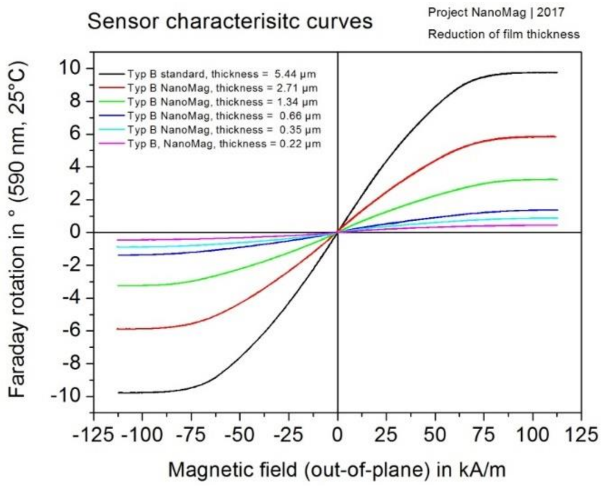


Fig. 2.3: Faraday rotation as a function of normal field for MOIFs of different thickness

Calibration procedure

Two different calibration approaches were applied depending on the measurement technique used. At INNOVENT, PTB and INRIM, the light intensity was measured by a digital camera. In order to convert the light intensity data into the magnetic field strength, pixel by pixel calibration curves (light intensity vs. field) were measured in the absence of a sample. Then these curves were applied for each pixel to find the local field strength above the sample. AT TUBITAK, the light intensity was measured using a photomultiplier and a special analog modulation technique was used which results in the signal varying linearly with the Faraday rotation. Thus, the Faraday rotation calibration curves, like those in Fig. 2.3, were used to calibrate the signal. In the range of lower fields these curves were linear and thus the measured signal was a direct measure of the local field with just one numerical constant found from the curve slope. In TUBITAK's system, the photomultiplier measurements were done at a single measurement spot, in order to obtain a 2D magnetic field map, the sample was scanned in two directions across that spot. Thus, the calibration is done at a single spot unlike the systems at INNOVENT, PTB and INRIM where the calibration is done pixel by pixel.

Magnetic fields measured were calibrated with the traceability established to the national magnetic field primary standards, the uncertainty budgets were computed for all measurement setups. Temperature dependences of MOIF parameters were measured, all parameters were found to depend only slightly on the temperature in the operational temperature range. Thus, the MOIF susceptibility (i.e. its sensitivity to the applied field) changed from 0.139 to 0.148 with changes of the temperature from 16 to 30° C. Although this dependence is minor, measurements should be done at a fixed temperature in order to decrease the measurement uncertainty.

One issue that has not been addressed previous to the present project is the effect of the in-plane component of the measured stray field on the magneto optical signal. The rotation angle of the magnetization vector $\vec{M}$ in the MOIF depends on both components of the stray field, H_Z and H_X (Fig. 2.1). However, only one parameter, the light intensity, was originally measured in the MO experiments. Thus, the problem did not have a unique solution and in all MOIF measurements done previous to this project, the effect of the in-plane component H_X was simply ignored.

Two approaches have been developed to address this issue. At PTB, a numerical model was developed that minimizes the MOIF's free energy in a given magnetic field. At TUBITAK, a second parameter, the derivative of the measured signal with respect to an additional small magnetic field applied in the plane, ΔH_X, was measured. Thus, a system of two equations with two unknowns was obtained, which is sufficient to uniquely determine both field components H_Z and H_X. The results obtained in the simulation exhibited a good agreement with the experiment. Also, it was found that in the specific case of low stray fields (compared to the MOIF's anisotropy field H_A) the MOIF response appears to be insensitive to H_X and thus the normal field component H_Z can be directly determined from the measured signal. That is, in low fields MOIF are similar to Hall sensors which also measure only H_Z.

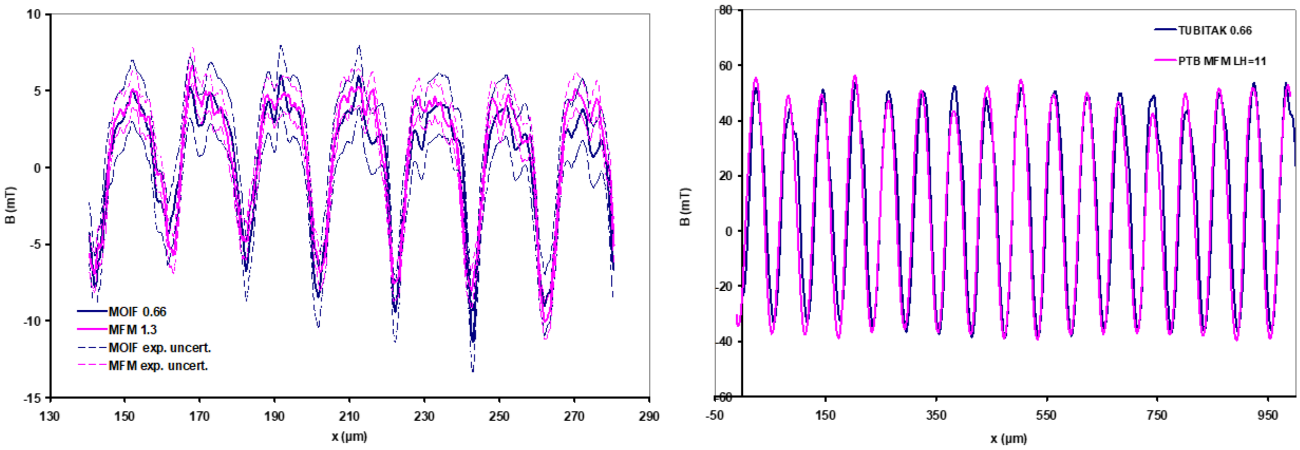
Different MOIF systems were used at different participant institutes. The systems at INRIM, TUBITAK and INNOVENT were based on high resolution optical microscopes that provide a high spatial resolution but a narrow field of view. The system at PTB has its own optics which provides a lower spatial resolution but a wider field of observation. The spatial resolution of PTB's MagView-CMOS system with a sensor size of 60 x 45 mm² is 25 µm. The system at INNOVET has a resolution of 1.5 µm. After purchasing a special 100x microscope objective at TUBITAK, the spatial resolution determined on the test magnetic patterns was found to be 0.3 µm, in line with the project target of 0.5 µm and is close to the optical resolution limit.

Resolution with respect to magnetic field is determined from the signal to noise ratio. It depends on the spatial resolution as the signal from a larger measurement spot results in a lower noise. The best field resolution achieved was 10 µT (which is the targeted value in this project) for the spatial resolution of 1.5 µm and above. Thus, magnetic features as small as 1.5 µm with field changes as small as 10 µT can be detected, which is a good achievement.

Two patterned magnetic samples where used to compare measurements done by different MOIF systems as well as to compare MOIF measurements with other techniques such as Scanning Hall Microscopy (SHM) and Magnetic Force Microscopy (MFM). These patterned magnetic samples included a thermomagnetically patterned (TMP) NdFeB thick film containing magnetic patterns with dimensions ranging from 1.5 µm to 20 µm periodicity, and a lithographically patterned NdFeB reference scale having magnetic structure with a period of 60 µm and fabricated by SENSITEC, Establishing proper conditions for comparisons was a challenging task due to (i) the difficulties of measuring exactly the same spot on the sample, and (ii) correctly relating the lift height in MFM measurements to the MOIF sensor thickness. A further comparison of MOIF with SHPM was discussed, above (Fig. 1.5). Generally, a good agreement between measurements using different techniques has been obtained (Figs. 1.5, 2.4). This provided a solid basis for the validation of the novel MOIF calibration techniques that resulted from the project.

Key outputs and conclusions

The magneto optic systems at INRIM, TUBITAK, PTB and INNOVENT have been calibrated with the traceability established to the national primary standards of magnetic field. The uncertainty budgets for the calibration have been prepared. Validation of the MOIF technique was done by comparing measurements done by different systems as well as comparing with other techniques, such as scanning Hall and Magnetic Force Microscopy. The envisaged spatial resolution from 50 µm down to 500 nm was achieved. The project thus heavily contributed to the European metrology landscape by providing calibrated fast magneto optical magnetic field metrology infrastructure from the centimeter to the sub-micrometer scale.



(a) (b)

Fig. 2.4: (a) MFM-MOIF comparison on TMP NdFeB sample with 20 µm periodicity. (b) MFM-MOIF comparison measurements on SENSITEC lithographically patterned NbFeB reference sample with 60 µm periodicity done using TUBITAK's high resolution magneto optical system and PTB's large range MFM. Both datasets show a good agreement thereby validating the calibrations of the systems.

Objective 3: magnetic force microscopy (MFM)

Objective 3 was to develop and validate quantitative magnetic force microscopy (qMFM), developing a protocol to traceably calibrate MFM probes and to implement the calibration algorithms in the open-source software Gwyddion for easy stakeholder access to quantitative and traceable nano-scale magnetic imaging.

Previous to the start of the project, MFM was able to provide qualitative images of the stray field distribution with nanoscale resolution with relatively ease, however, quantitative images were only provided in specialized laboratories. The project followed the calibration approach introduced by Hug et al in 1998 based on the use of a reference sample with calculable stray field. The measured stray field of the selected MFM tip was compared to the calculated stray field and the so-called tip transfer function (TTF) was derived by a deconvolution process. The TTF contains all information on the sensitivity and spatial resolution of the such calibrated tip and allows quantitative imaging of unknown magnetic structures by deconvolution the obtained images with the TTF.

In this project, the TTF calibration approach has been validated for the first time by an international comparison and suitable reference materials for calibrations have been identified and validated. Furthermore, an open-access version of the deconvolution algorithm has been implemented and published in an open-access scanning probe software allowing for the first time easy stakeholder access to quantitative and traceable nano-scale magnetic field measurements. These results have entered into Guidelines and the submitted international IEC standard for quantitative MFM; thereby underpinning for the first time international harmonization of nano-scale magnetic field measurements.

Modification of atomic force microscopy systems.

In order to be able to provide qMFM services, the MFM systems existing within the consortium had to be modified or upgraded to: (i) cover a larger spatial range, and (ii) provide traceability to the SI standards. Additionally, the modifications allowed testing relevant magnetic nanostructures, resilience of the TTF calibration approach to variations in field and/or temperature. The SPM systems at NPL, PTB, IFW, and CMI that were modified and upgraded to achieve in-field large-range metrological qMFM are:

- At NPL, the qMFM system includes a heating (up to 100 deg) and cooling (down to -30 deg) stage, along with in-field capability (90 mT maximum out-of-plane field, 120 mT in-plane).
- At PTB, a large-range qMFM with interferometric positioning with traceability to the SI units was developed.
- The system at IFW uses a system of permanent magnets to achieve ~ 600 mT of out-of-plane field.

- At CMI a large-range qMFM with interferometric positioning with traceability to the SI units was developed.

All the systems were compared during a Round Robin comparison, demonstrating that, within the error, all the systems agree in the measured stray fields. Different tests, linking stray field measurements across different length scales were carried out showing an agreement between the different length scales. Hence, as a result of this project, the partners of this project are now capable of providing qMFM measurements.

Calibration protocol.

The calibration process based in the TTF approach, was optimized and improved during of the project.

The principal steps of the TTF are as follows: The TTF approach starts by converting the phase change images (e.g. Fig. 3.1(a)) into pixel area force gradient (Fig. 3.1(b)), considering the spring constant and quality factor of the probe. Then the up/down domains are differentiated to create a mask to simulate the stray field of the sample (shown as a red overlay in Fig. 3.1(c)). Using the material parameters and the mask, the surface charge of the sample is calculated (Fig. 3.1(d)). The pixel area force gradient and the surface charge (i.e. Fig. 3.1(b) and (d), respectively) are then deconvolved to obtain the TTF (i.e. the gradient of the probe's stray field at sample's surface (Fig. 3.1(e)). The TTF can then be used to deconvolve MFM images taken with the same probe and obtain the stray field produced by the sample under study, or it can be used to obtain a dipole approximation of the probe (e.g. to be implemented in numerical simulations).

In order to find the dipole that best reproduces the probe's stray field, the typical small asymmetry in the stray field profile due to the cantilever tilt is neglected and the TTF matrices are circularly averaged around the central peak. The result for a typical probe (PPP-MFMR by Nanosensors) is shown in Fig. 3.1(f). Apart from the small errors introduced by noise filtering (Wiener filter) and the averaging processes, this real space TTF (RSTTF) is the correct quantification of the probe's imaging properties within the spatial frequency range provided by the reference sample and probed by the reference measurement (determined by image and pixel size). The best dipole fit is then found by calculating the field gradient created by the dipole and matching it with the profile extracted from the TTF. In the case shown here, for the PPP-MFMR Nanosensors probe the dipole has an amplitude of -3.45 × 10 -16 Am^2 and its located at a distance Z = 70 nm from the probe's apex. Using these values, the z-component of the field generated by the probe at 100 nm from the probe's apex is estimated in ~ 14 mT. It is important to notice that in order to fully reproduce the probe's field gradient a multipole expansion is required, however, as it can be seen in Fig. 3.1(f), a dipole approximation generally is insufficient to capture most of the fields features.

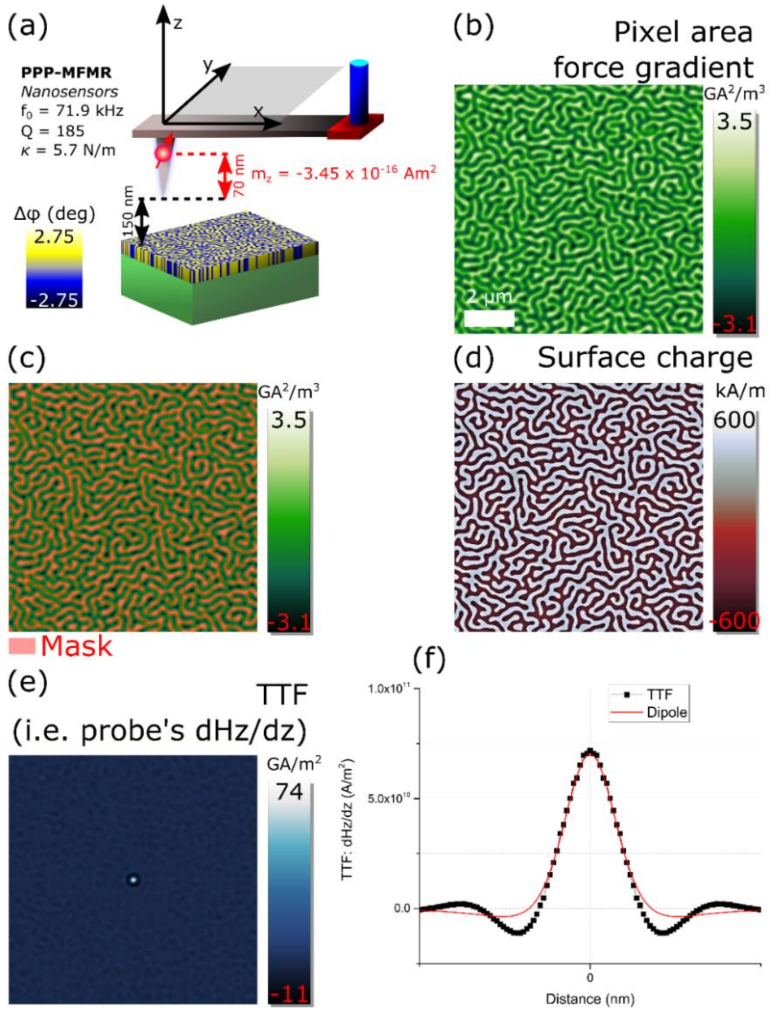


Fig. 3.1: Process followed to approximate probe's stray field by a dipole for a PPP-MFMR Nanosensors probe. (a) Schematic of MFM on a Co/Pt sample indicating the distance to the sample during the second pass (i.e. 150 nm), the mechanical properties of the probe, and the value and position of the dipole (70 nm from probe's apex). (b) Conversion of phase shift image into pixel area force gradient using probe's mechanical properties. (c) Pixel area force gradient image masked to identify up/down domains in the Co/Pt sample. (d) Reconstruction of the sample's surface charge, using the mask from (c) and the material parameters. (e) TTF obtained using the surface charge shown in (d) and the pixel area force gradient in (b). (f) Circularly averaged profile of (e) alongside with the field gradient from a dipole located as in (a) and with the values indicated in (a).

Gwyddion implementation

Previous to this project, most microscopy laboratories worldwide haven't implemented qMFM although the above calibration processes were available 20 years ago. This is due to the complexity of processing the data after capturing the qMFM images. This project aimed to ease the access to qMFM by simplifying and improving the calibration process. Consequently, the improvement consisted of implementing the calibration tools in the open software Gwyddion. Receiving input from all partners, CMI implemented the TTF-based convolution-deconvolution protocol into Gwyddion, created an online manual, and published several papers comparing the optimization done in the deconvolution/convolution process. Within a detailed analysis carried out at PTB, the full uncertainty budget of the MFM calibrations has been established for the first time. The corresponding algorithm is also implemented in Gwyddion to ensure stakeholder access to the uncertainty of their calibrations. The open access software can be found at: http://gwyddion.net/

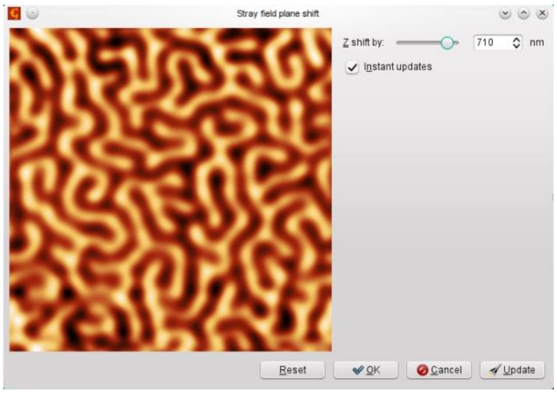


Fig. 3.2: Example of Gwyddion analysis window showing the stray field calculated from MFM phase shift data. Taken from http://gwyddion.net/documentation/user-guide-en/mfm.html

Comparison of several calibration methods.

Besides the TTF approach using Co/Pt calculable reference samples, two other calibration processes were investigated during the project, one based on nano-sized graphene Hall crosses, and one based on micron-sized field coils.

Thin Co/Pt films were fabricated and optimized by IFW, characterized by various partners and distributed in the Consortium. Micron-sized coils were fabricated by PTB and optimized by several iterations between PTB and NPL. One optimization was the planarization of the coils to reduce the effect of topography in MFM imaging. Nano-scale graphene Hall crosses were provided by external partners.

The comparison process (schematically described in Fig. 3.1) was as follows:

1. An MFM probe is used to image: 1st Co/Pt; 2nd the Hall cross; 3rd the Co/Pt again; 4th the coil; and 5th Co/Pt. The spring constant and the Q factor of the probe was measured each time the Co/Pt was imaged.
2. Using the data from imaging the Co/Pt and the TTF deconvolution, the magnetic field from the probe is approximated by a magnetic dipole.
3. With a numerical model, that uses the dipole as an input, the data from the Hall cross and the coil was simulated.

At NPL, using the comparison process described above, a total of ~30 commercial MFM probes were calibrated. During the process, electrostatic compensation was required when imaging the coils and the graphene Hall crosses. The raw data (which can be seen in Fig.3.2 for a PPP-MFMR probe) was sent to IFW, which performed the deconvolution process and approximated the stray field by a dipole. The dipole approximations and the raw data was then sent to INRIM, where they performed numerical modeling to reproduce the measured results.

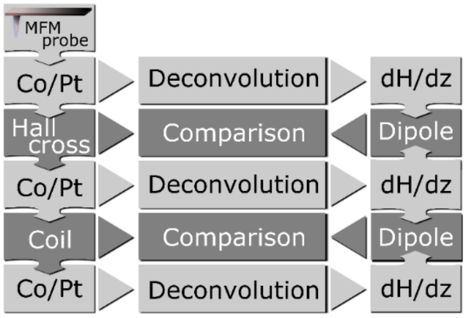


Fig. 3.3: Calibration process. For each probe the sequence of measurements consists in imaging the Co/Pt film in between imaging the Hall cross and the coil. The images from the Co/Pt were then deconvolved to obtain the probe's magnetic field gradient (i.e. $\frac{dH}{dz}$*), and subsequently, the dipole approximation. Numerical modeling, using the dipole approximations, were then compared with the experimental data from the use of the Hall cross and the coil.*

The numerical data showed that the dipole approximation obtained from the Co/Pt sample can be used to reproduce the measurement results obtained from the Hall crosses. While many different dipole approximations matched the Co/Pt results, only one approximation matched both the Co/Pt and the Coils.

Conclusion

Overall, it was shown that the TTF approach can be validated by comparison to the two other calibration methods. Furthermore, the TTF approach has proven to be more robust (e.g. against electrostatic influences) and easier to implement. The objective was to obtain an spatial resolution below 50 nm, and the results based on the TTF approach successfully achieved the targeted resolution. Therefore, the TTF approach in combination with the above Gwyddion software and the description of the reference samples established the most accessible path to nano-scale quantitative magnetic field measurements for the European and international stakeholders.

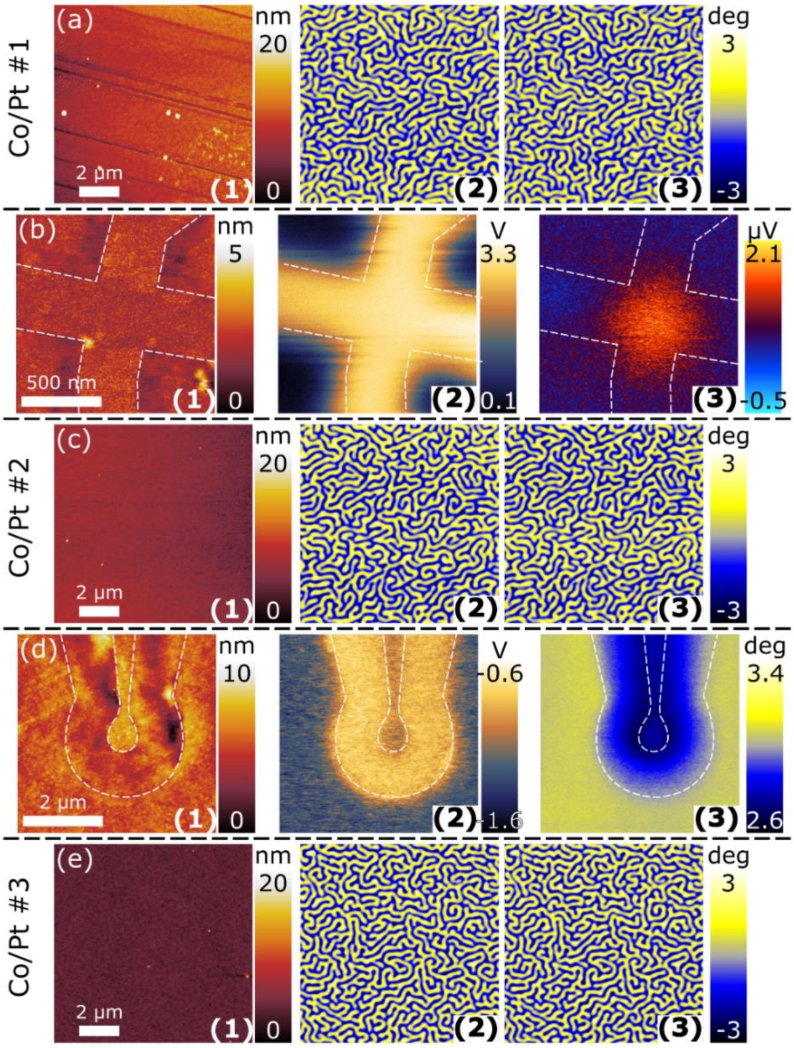


Fig. 3.4: Sequence of measurements for a PPP-MFMR Nanosensors probe. (a) MFM on a Co/Pt sample: topography (1); phase during the second pass (2); and phase during the second pass on the second repetition of measurement (3). (b) FM-KPFM/mSGM on a graphene Hall cross: topography (1); V_{CPD} during the second pass (2); and electrical voltage measured across the sensor (3). (c) MFM on a Co/Pt as in (a). (d) FM-KPFM/MFM on a micro-coil: topography (1); V_{CPD} during the first pass (2); and phase during the second pass (3). (e) MFM on a Co/Pt as in (a) and (c).

Objective 4: calibration artefacts

Objective 4 was to provide novel magnetic stray field reference materials for reliable high-resolution stray field calibrations, and high-level modelling tools and methods to support calibration and data analysis for scanning magnetic field microscopy, magneto-optical indicator film (MOIF) microscopy and magnetic force microscopy (MFM). Previous to the project, local magnetic stray field measurement techniques were usually calibrated by determining the sensor's response to a homogeneous magnetic field. However, to obtain reliable calibrations with micrometer and nanometer spatial resolution, the influence of the response of the finite sized sensor must be considered and needs to be implemented into the calibration procedures.

The project proposed a way to meet this requirement, developing a set of reference samples, which produce a well-defined and controllable spatially varying magnetic field. The samples were tailored to the spatial resolution of the considered measurement techniques, with the generation of quasi uniform magnetization regions with typical sizes ranging from some hundreds of microns down to below 10 nm. Clean-room nano-patterning techniques were also adopted to obtain materials with multiple sized features. Meanwhile, different modelling approaches (e.g. full micromagnetic modeling, solution of Poisson equation) were developed and used to calculate the spatial distribution of the stray field produced by the reference samples, enabling us to validate the considered calibration schemes and infer the obtained experimental results.

Fabrication of magnetic reference samples

Different stray field reference materials were developed to generate magnetic stray fields with well-defined spatial profile, as calibration artefacts for on-site traceable calibration and reliable micro- and nano-scale traceable magnetic field measurements. The fabricated samples include:

1) **materials with micro-scale features** generating high magnetic stray fields (up to 200 mT) and with a magnetic pattern width in the range from hundreds of microns down to 20 µm;
2) **materials with nano-scale features** (from several micrometers down to 10 nm), generating magnetic fields in the range from 1 mT to 100 mT;
3) **nano-scale structures based on planar coils**, generating magnetic fields in the range from 1 mT – 100 mT and to be used for MFM probe calibration.

Among the **materials with micro-scale features**, SENSITEC produced pulsed magnetized scales made of wet pressed hard strontium ferrite, with a remnant magnetization of 395 mT. The materials were magnetized into alternating up and down uniform magnetized stripes with a length of several millimeters and a variable nominal width (250 µm, 500 µm, 1000 µm). These samples were successfully used to prove quantitative stray field measurements by means of gold Hall sensors for scanning magnetic field microscopy.

SENSITEC also prepared a prototype wafer containing lithographically defined stripe-pitch geometry samples made of NiFe, with stripe width varying from 20 µm to 100 µm and pitch varying from 40 µm to 200 µm. These materials were initially used to test MOIF microscopy and MFM, under different magnetization configurations, from saturation to remnant state. However, such encoders with soft magnetic materials can show significant variations of the stray field distribution from device to device inhibiting their use as quantitative reference materials. In a second iteration of these lithographically patterned micro-scale reference materials a stripe geometry based on hard magnetic NdFeB was developed. These micron-scale reference materials were successfully used for comparison measurements of MOIF and MFM as shown in Fig. 2.4b) and have a high potential to be further developed into standardized reference materials.

Additionally, SiOx/Ta(100 nm)/NdFeB(5 µm)/Ta(100 nm) films with laser-thermo-magnetically patterned microstructure and out-of-plane remnant magnetization (1.3 T) were provided by the collaborator Institut Louis Néel in Grenoble, France. The pattern has a chessboard configuration and a depth of reversal estimated to be in the range of 1.2 µm. The characteristic dimensions of the mask used for the thermo-magnetically patterning range from 1.5 µm to 10 µm (Fig.4.1). These samples were employed as a reference to compare MOIF microscopy calibrations performed by different partners of the project and to compare calibrated MOIF with quantitative MFM (Fig. 2.4).

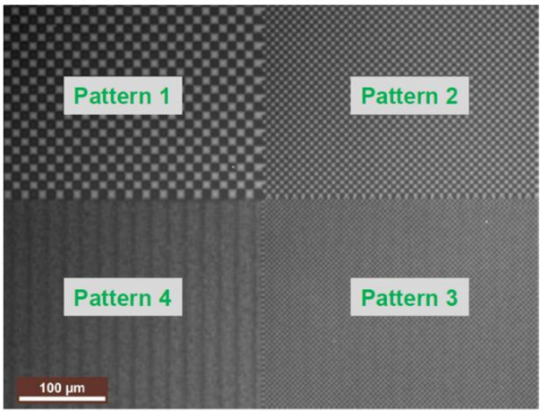


Fig. 4.1: Image of NdFeB film with different thermo-magnetically patterned microstructures from 1.5 µm to 20 µm.

Among the **materials with nano-scale features**, a Co/Pt multilayer film was prepared by IFW as a calibration sample for MFM measurements and was referenced in the Guideline for MFM calibrations described in Objective 5. The stack of Pt (2 nm)/ [(Co(0.4 nm)/ Pt(0.9 nm)]100/ Pt(5 nm)/ Ta(5 nm)/ SiOx/ Si(100) was prepared by magnetron sputtering. The total magnetic thickness is 130 nm and owing to the interface anisotropy, the film develops magnetic anisotropy perpendicular to the surface. At zero field, the magnetization of the multilayer collapses into a stripe-domain pattern with average domain width of 170 nm. Via global magnetization measurements, the saturation magnetization was estimated to be 500 kA/m (error ±30 kA/m) and perpendicular magnetization was confirmed. The Bloch type domain transition had a width of about 16 nm, as estimated from MFM imaging.

Other samples fabricated by IFW, also used for the MFM guideline definition, comprised epitaxial $SmCo_5$ thin films with a patchy domain pattern on a length scale of 70–300 nm. The film was prepared by UHV pulsed laser deposition on a heated (650 °C) Ru-buffered Al_2O_3 substrate; the full layer architecture was Ta(3 nm)/$SmCo_5$(12 nm)/Ru(9 nm)/Al_2O_3(0001). The sample exhibited a very large uniaxial magnetocrystalline anisotropy perpendicular to the film surface (perpendicular anisotropy constant higher than 6 MJ/m^3) and a small domain transition width (lower than 5 nm).

Additional hard magnetic thin films with perpendicular magnetic anisotropy were produced by GTU with sputter deposition. The samples, with variable thickness and with/without extra Ti layer, were made of Pt/Co/Pt on naturally oxidized Si(111) substrates. Via magneto-optic Kerr effect (MOKE) characterization it was found that the films with Ti layer inclusion enable the obtaining of nearly square hysteresis loops. X-ray powder diffraction (XRD) analysis conducted at the Swiss Federal Laboratories for Materials Science and Technology (EMPA) pointed out a textured Pt structure for both types of films, but a more ordered Co orientation for samples including Ti layer. The effect of cluster size on magnetic anisotropy was also investigated by changing the argon flow rate during deposition. Finally, a multilayer stack with two different Ti/Pt/Co/Pt type samples grown on each other was produced, providing a step-like hysteresis loop.

All the Pt/Co/Pt based films from GTU and the $SmCo_5$ films from IFW were processed at PTB with clean-room patterning, considering a fixed lithography pattern with variable nano-structuring scale, with feature size from about 1 µm down to below 10 nm (Fig. 4.4). The lithographic definition allowed to obtain high reproducibility, full dimensional traceability, calculable stray field properties and increased pattern resolution, enabling the overlapping validation of traceability from high resolution MFM measurements to micrometer resolved techniques, such as MOIF or scanning Hall microscopy techniques.

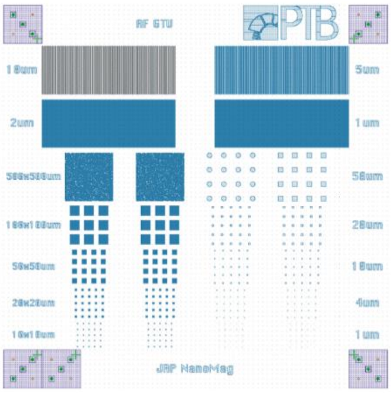
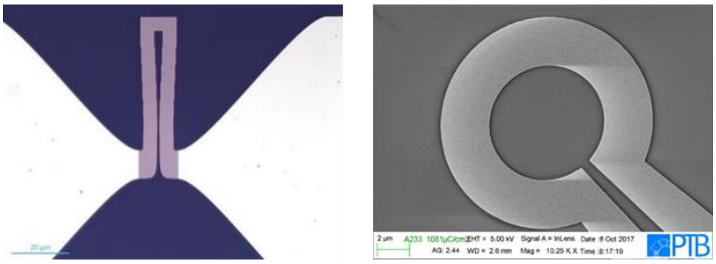


Fig. 4.2: Layout of the lithography mask with various patterns (disks, bars, squares, maze) of different length scales.



(a) (b)

Fig. 4.3: Coil geometry of first (a) and second batch (b).

Regarding **nano-scale structures based on planar coils** for MFM probe calibration, PTB produced several microstructures made of graphene, gold or aluminum, with variable shape and width ranging from 500 nm to 5 µm (Fig. 4.3). In particular, the planar coils were fabricated on a silicon wafer with 300 nm oxide using electron beam lithography and conventional lift-off processes.

For the first batch, stripe like coils with conductor widths of 500 nm, 1 µm, 2 µm and 5 µm and varying conductor distances from 0.5 µm to 2.7 µm were fabricated. Gold and aluminum layers with thicknesses of 20 nm and 30 nm were deposited by electron evaporation; 5 nm of titanium was used as an adhesion agent. On the contact pads a 50 nm thick gold layer was added.

For the second batch, the geometrical design was optimized. Gold round coils with widths from 1 µm to 4 µm and diameters from 2 µm to 10 µm were added, and the coil regions were separated from the contact regions to simplify the MFM measurements. Furthermore, the coils were embedded to minimize MFM-tip degradation. Therefore, thermal silicon oxide was deposited by plasma-enhanced chemical vapor deposition on Ti/Au (5 nm/50 nm plus 50 nm Au for the contact pads) coils and thinned using chemical-mechanical polishing. The final oxide thickness over the coils is (50 ± 20) nm, while the contact pads are oxide free. The round gold coils were successfully employed in the calibration of several MFM probes with different magnetic moment, in combination with MFM imaging.

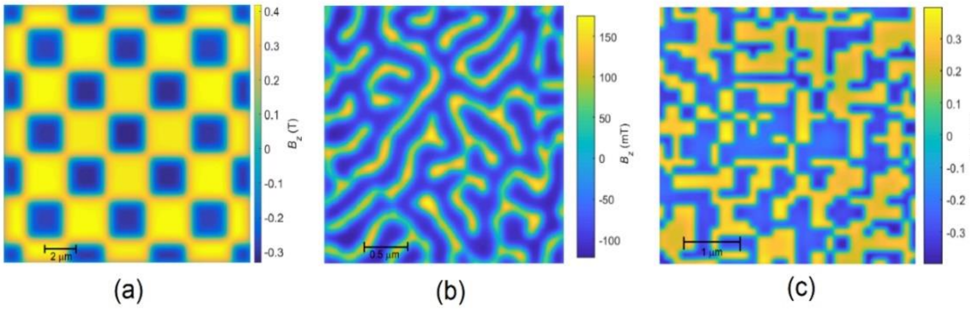


Fig. 4.4: Maps of the z-component of the stray field produced by three different reference samples: (a) thermo-magnetically patterned NdFeB film; (b) Co/Pt multilayer film; (c) nano-patterned Pt/Co/Pt based film.

Development and application of modelling tools

The definition of reliable high-resolution stray field calibration schemes was supported by validated numerical tools, aimed at (i) calculating the spatial distribution of the stray field generated by the considered reference samples and at (ii) inferring the experimental results obtained with MFM, MOIF microscopy and scanning magnetic field microscopy.

First, a **custom-made 3D computation tool for the solution of the Poisson equation** was developed and validated by INRIM, to support MFM and MOIF microscopy results. Through this numerical code, it was possible to calculate the maps of the magnetic stray field produced by large reference samples with known magnetization configuration, also in the presence of complex magnetic patterns (Fig. 4.4). As an example, the magnetostatic field solver was used to evaluate the spatial decay of the stray field produced by the materials with micro-scale features produced by SENSITEC (i.e. the strontium ferrite scales and the NiFe stripes) and by the Institut Louis Néel (CNRS) (i.e. the thermo-magnetically patterned NdFeB films). The simulations were performed at different heights from the sample surface and enabled us to investigate the effects of geometrical parameters and patterning, providing information about the peak values of the stray field. Moreover, the modelling results were compared to experimental data obtained with MOIF microscopy, considering different thicknesses of the indicator film.

Additionally, the magnetostatic field solver was used to calculate the maps of the stray field generated by the materials with nano-scale features, focusing on the Pt/Co/Pt based films produced by GTU and then lithographically patterned at PTB. The simulations were performed under the assumption of magnetically saturated samples (perpendicularly to the film plane), considering different patterning scales. The stray field maps were calculated at different heights from the sample surface. Similar numerical analysis was also performed for the Co/Pt multilayer films fabricated by IFW, which show an intrinsic stripe domain structure. In this case, the out-of-plane magnetization configuration extracted from MFM images taken at small distance from the sample surface was used as an input for the simulations.

Second, INRIM developed a **parallelized 3D micromagnetic solver** for the simulation of the magnetization configuration at equilibrium of 3D magnetic samples with size up to about 10 µm. The code was validated by comparison to the open source software OOMMF and MuMax, developed by NIST and Ghent University, respectively. The INRIM micromagnetic solver uses a 3D finite difference method for the calculation of the exchange field, an FFT-based technique for the evaluation of the magnetostatic field and a geometric integration scheme based on the Cayley transform for the time-update of the Landau-Lifshitz-Gilbert equation.

After the validation, the micromagnetic code was used to compute, in combination with software MuMax, the equilibrium magnetization configurations of high anisotropy magnetic thin films with nano-scale intrinsic stripe domain structure. The attention was focused on the perpendicular magnetic anisotropy materials composed of Co/Pt multi-layers fabricated by IFW. High spatial resolution micromagnetic simulations were performed considering mesh elements with size of 4 nm. In order to investigate the domain-wall structure, which also varies along the sample thickness, and to define the optimal discretization mesh for calculations, straight magnetic domains were first simulated. After this preliminary analysis, the real samples were modeled, considering as an initial state the magnetic configurations extracted from MFM measurements. As an output, the domain wall size was determined after the simulation of a relaxation process, varying the extension of the regions within magnetic domains, where the magnetization vector was blocked. The results from the simulations were compared to the experimental ones, obtained with MFM imaging.

Third, INRIM developed and validated a **custom-made finite element code for the calculation of the Hall voltage response of micro and nano Hall sensors**, under the effect of strongly localized magnetic fields, to be used as a support to scanning magnetic field microscopy. The code was applied to investigate the sensitivity and scanning magnetic field measurement resolution of Hall devices fabricated at PTB, made of gold or monolayer graphene and with cross width varying from 50 nm to 5 µm. The analysis was performed for different bias currents, calculating the Hall voltage response to the stray field generated by reference samples with micro-scale features, focusing on the thermo-magnetically patterned NdFeB films from the Institut Louis Néel (CNRS) and the hard strontium ferrite scales from SENSITEC. The stray field at different height scan lines was estimated by means of the custom-made 3D computation tool for the solution of the Poisson equation, developed at INRIM, as well as with the method based on transfer functions in Fourier space, implemented at PTB. The analysis was performed by calculating the maps of the Hall voltage response, obtained by moving the magnetic sample, according to the scanning procedure implemented by PTB and CEA. The effects of inclination of the scanning system, vertical distance of the film from the sensor surface and sample roughness and surface defects were investigated, analyzing in detail the sources of signal drift. A good agreement between simulations and scanning Hall microscopy measurements of the hard strontium ferrite scales was found, with small deviations that can be explained by the uncertainty budget.

The finite element code for the calculation of the Hall voltage response was also used by INRIM to model graphene Hall nano-sensors characterized at NPL. The numerical code was first employed to interpret the Hall voltage maps measured in the presence of multi-layered custom-made MFM probes with controllable multi-stable high/low magnetic moments, also considering probe-sensor electrostatic capacitive coupling. In the adopted model, each magnetic layer of the multi-layered probe was approximated as a point magnetic dipole, with parameters derived by IFW with a tip-sample deconvolution algorithm. Then, in collaboration with NPL and IFW, the Hall device simulation code was also used as a support to the calibration of several commercial MFM probes with variable magnetic moment.

Fourth, INRIM developed a **custom-made finite element code for the calculation of the electrical resistance and the magnetic stray field of planar micro-coils** was fabricated at PTB, to be used in MFM probe calibration. The computation tool was initially employed to model the stripe like coils, considering different coil widths ranging from 0.5 µm to 5 µm. The maps of the generated stray field were calculated in order to find the zones around coils characterised by the highest field values. Moreover, the modelling analysis was performed on the gold planar coils with ring shape (having width ranging from 1 µm to 3 µm and outer diameter ranging from 2 µm to 10 µm). Regarding the gold ring coils, the developed finite element code was also used to support MFM probe calibration in combination with MFM imaging and use of graphene Hall nano-sensors. In collaboration with NPL and IFW, INRIM evaluated the maps of the phase shift of MFM tip vibration, due to the magnetic interaction between the tip and the coil (Fig.4.5), considering the additional contribution of electrostatic capacitive coupling. The analysis enabled us to evaluate the reliability of the MFM probe point-dipole approximation, resulting from the application of the tip-sample deconvolution algorithm.

Finally, an analytical model was successfully employed to determine the easy-axis and hard-axis hysteresis loops and magnetoresistance response of GMR nanosensors, fabricated at CEA, under the assumption of magnetisation uniformly distributed in the ferromagnetic layers. The model takes into account the effects of the exchange coupling between free and reference layers, the antiferromagnetic interlayer exchange coupling between reference and pinned layers and the exchange bias coupling between pinned and antiferromagnetic pinning layers. Contextually, CEA performed micromagnetic simulations with the OOMMF software, considering the sensor as made of the free layer only, in turn composed of a thin CoFe layer and a thick NiFe layer. Despite the simplifying assumption, the simulations were able to reproduce the experimental magnetoresistance response of the studied GMR sensors with width from 100 nm to 1 µm, under a uniform in plane field. Both the variation in sensitivity due to shape anisotropy and the irreversible jumps, observed experimentally, were correctly predicted.

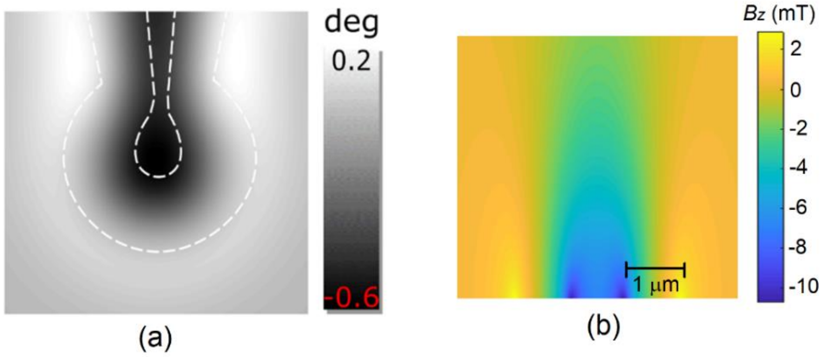


Fig.4.5: (a) Map of the phase shift of MFM tip vibration, due to the magnetic interaction with a gold ring coil (width of 1 ∝μ and diameter of 2 ∝μ); (b) Map of the z-component of the stray field generated by the coil, when a current of 10 mA is imposed.

Conclusion

The project has delivered different types of stray field reference materials which have been used to validate traceable calibrations by comparison measurements of the same method over different Partner laboratories and to validate calibrations of different methods against each other. The pattern range of the different calibration samples spans from 250 µm periodicity, over the range of few tens of micrometers down to the nanometer scale. In addition, a variety of numerical tools has been developed allowing to evaluate and compare the stray field and measurement response of measurements and calibrations of the reference materials by various high-resolution magnetic imaging techniques.

Objective 5: Ensuring traceability

The overall goal of this project was to develop and provide coordinated and sustainable European metrology capabilities that extend reliable and traceable measurements of spatially resolved magnetic fields down to the micrometer and nanometer length scale. As shown in the above description of the results of the four technical objectives a wealth of metrological infrastructure has been established at partner NMIs, allowing for the first time traceable calibrations of magnetic field measurements down to the nanometer scale. Stakeholder access to the newly available metrology infrastructure is enabling the European industry to take advantage of these unique metrology tools for quality control and quantitative evaluation of micro- and nano-scale magnetic devices.

Additionally, the nano Hall mapper which is commercialized by the unfunded partner SENIS since May 2019 (http://www.senis.ch/products/mappers/nano-magnetic-field-mapper) is allowing stakeholders easy access to fast, robust, and versatile calibrated high resolution scanning field system for multi-purpose applications.

The project had further impact by validating quantitative MFM calibrations and thus for the first time allowing calibrated quantitative and validated nano-scale magnetic field measurements to be used by stakeholders across Europe and world-wide. An MFM calibration algorithm including uncertainty evaluation has been implemented by the Partners and validated by the first international comparison of quantitative MFM with five participants in the Consortium under the lead of CMI. Based on this successful international comparison, also a set of nano-scale magnetic stray field reference samples has been validated and Guidelines for nano-scale magnetic field measurements have been drafted.

Additionally, the deconvolution algorithms required for carrying out traceable quantitative MFM calibrations by the stakeholders have been implemented in the freeware scanning probe microscopy software Gwyddion (http://gwyddion.net/news.php#v2.48). The freeware package is regularly maintained and a detailed user guide for stakeholder calibrations is available for download. Since the initial upload of a Gwyddion MFM module, the software has been downloaded by external stakeholders more than 3000 times. This clearly demonstrates the high demand for quantitative magnetic measurement tools by the international stakeholders and the impact the NanoMag Project is having world-wide.

To increase the impact of the scientific work, the Guidelines for nano-scale magnetic field measurements were used to draft the first international standard for nano-scale magnetic measurements. For the first time, guidelines for underpinning international harmonization in research and development in the high-tech field of nanomagnetism are available as a result of the present project. PTB and ISC have been leading the project group of IEC TC 113 "Nanotechnologies" to develop the standard technical specification TS 62607-9-1: Nanomanufacturing – Key Control Characteristics – Part 9-1: Spatially resolved magnetic field measurements – Magnetic Force Microscopy. The draft technical specification was submitted in July 2019 and the publication of the TS is expected after comments by the international TC members in 2020. This first international IEC standard can be considered a milestone for the industrial and scientific community of nano-magnetism as it will enable for the first time internationally harmonized, quantitative and reliable nano-scale magnetic measurements to the benefit of international R&D, industry, and society.

EMPIR Researcher Mobility Grant (RMG) results and contributions to the present project:

15SIB06 RMG 1: NPL, UK (Employing organisation); PTB, Germany (Guestworking organisation).
Investigation of magnetic scanning gate measurements using a perpendiculary magnetized nanoscale Hall cross. The RMG researcher was trained in using the magneto-transport, MFM, and MOKE systems at PTB to perform device optimization and magnetic scanning gate measurements in order to verify MFM probe calibration.
The technique proposed and developed in this RMG project uses a reference sample which consists of magnetic nanostructures with features of different sizes, where the magnetisation direction is controlled by electrical currents, and the effect of the magnetic field from the MFM probe onto the sample's magnetization can be measured as an electrical voltage. Within the RMG successful designs of the nanostructures were achieved, a protocol to perform the calibration was developed, and numerical simulations are being carried out by other partner of the NanoMag consortium (i.e. INRIM) to compare the current method with the previous ones.

15SIB06 RMG 2: INRIM, Italy (Employing organisation); PTB, Germany (Guestworking organisation).
Micromagnetic simulation of nano-scale magnetic materials and magnetization reversal processes. The RMG researcher carried out the research at PTB. The training for the RMG researcher included one-on-one training on nano scale magneto transport. PTB researchers received training on micromagnetic simulations.
During this RMG the magnetization state in the presence of defects in perpendicular magnetized materials was calculated. The defects were simulated using grains, the strength of the anisotropy axis deviation and the inter-granular exchange as parameters. The stray field of the sample was then derived from these results. This analysis was done for two different types of samples, which were a single layer of Co/Pt with composition Pt(3.46 nm)/Co(1.683 nm)/Pt(1 nm) and a multilayer composed of [Pt(0.9 nm)/Co(0.4 nm)]100. In the case of multi-layered samples, the effective parameters were calculated from the domain size distribution using domain models. The Anisotropic Magnetoresistance (AMR) signal was calculated for the samples. Due to its complexity, it was not possible to study the full MR sensors, so only a simplified version was analysed. A joint paper with researchers of PTB was published.

15SIB06 RMG 3: INRIM, Italy (Employing organisation); NPL, UK (Guestworking organisation).
Measurements of local spin seebeck effect by means of thermal scanning probe technique. The RMG researcher focused on measurements of local spin seebeck effect by means of thermal scanning probe technique. The RMG researcher trained to use MFM system and scanning thermal microscope at NPL to correlate magnetic domains with electrical signals arising from the seebeck effect.
We first focused on the local spin Seebeck effect with a measurement system based on a combination of magnetic force microscopy (MFM) and heated-tip atomic force microscopy (nanoTA). The sample under test was a bulk yttrium iron garnet (YIG) with a Platinum (Pt) thin film (5 nm) deposited on the top surface of the sample. The Pt film was electrically connected to a nanovoltmeter in order to record any inverse spin Hall effect (ISHE) signal from the YIG-Pt interface. The result of this RMG was the proven feasibility of the application of scanning probe techniques to the field of spin caloritronics, beyond the MFM. This study could stimulate some future works in the framework of spintronics and scanning probe microscopy like the design of functionalized tips with materials like high spin orbit coupling metals such as platinum or tungsten.

15SIB06 RMG 4: INRIM, Italy (Employing organisation); NPL, UK (Guestworking organisation).
Measurements of the Dzyaloshinskii-Moriya interaction and manipulation of individual skyrmions by scanning probe microscopy. The RMG researcher focused on the measurements of the Dzyaloshinskii-Moriya interaction and manipulation of individual skyrmions. The RMG researcher trained to use MFM system at NPL to perform in-field measurements and to operate in lift mode.
This RMG aimed at imaging and tuning Néel domain walls in ultrathin CoFeB films through Magnetic Force Microscopy (MFM). By imaging samples with systematic variations of the Dzyaloshinskii-Moriya interaction (DMI), we wanted to investigate the relationship between DMI strength and spin structure of Néel walls. However, the limited spatial resolution achieved, combined with the lack of custom-made MFM tips on which the project relied, prevented us from achieving these targets. Anyhow, MFM imaging of the CoFeB films allowed us to obtain interesting results. Firstly, we were able to measure the strength of the DMI for all the sample investigated. Secondly, we proved for the first time that MFM can be used to move individual magnetic skyrmions in a controlled manner. So, despite not meeting the original targets, we believe that the output of this RMG will have a strong impact on the spintronics community. A joint manuscript has been published.

5 Impact

The project outputs have been transferred to stakeholders via the project web-site. At the end of the project, 57 talks and posters were presented at conferences such as Intermag 2017 and 2018, the IEEE Conference on Advances in Magnetics, the International Conference on Magnetism 2018, CPEM 2018, the 16th European Powder Diffraction Conference (EPDIC16) and the 65th American Vacuum Society International Symposium & Exhibition.

In addition, the project has produced 18 open access publications in journals such as American Institute of Physics (AIP) Advances, Applied Physics Letters, IEEE Transactions on Magnetics and Ultramicroscopy. Members of the consortium have also given training courses to stakeholders including 'Introduction to quantitative MFM', 'Introduction to quantitative nano scale field measurements', and 'Calibrated nanoscale magnetic field measurements'. Furthermore, the project established a Stakeholder Committee and the outputs from the project were disseminated to industry and other potential end-users.

Impact on industrial and other user communities

<u>Exploitation of high resolution scanning Hall microscopy:</u> Partner SENIS, has developed and tested a high-resolution sliding probe scanning Hall microscope prototype with 50 nm positioning accuracy and micrometer spatial sensor resolution. The system is called nano Hall mapper and the commercial exploitation of the system is envisaged including a new product based on the prototype or offering traceable high-resolution calibration services to end-users.

<u>Exploitation of MOIF calibration software routines:</u> MOIF calibration software routines developed within the project could be suitable for exploitation in commercial MOIF measurement systems. PTB is in contact with an industrial stakeholder that will be encouraged to implement calibration algorithms in their analysis software to enable a traceable calibration chain to end-users of commercial MOIF stray field measurement systems.

<u>Free stakeholder access to MFM calibration software:</u> The freeware scanning probe microscopy software Gwyddion MFM software package (http://gwyddion.net/news.php#v2.48) developed in the project was published online in April 2017. Since the initial upload of a Gwyddion MFM module on April 30th, 2017 and up to the date of this final report, the software has been downloaded by external stakeholder more than 3000 times. This demonstrated the demand by the international stakeholder community for quantitative magnetic measurement tools and the impact this project had on quantitative and reliable nano-scale magnetic measurements. The freeware package is regularly improved and a detailed user guide for stakeholder calibrations is presently under development.

A range of other activities were undertaken including liaising with industrial stakeholders, namely two MFM manufacturers and one MOIF system manufacturer. An industrial audience was also specifically targeted through a project presentation at the MR Sensor Symposium Wetzlar 2017 and via the final dissemination meeting at the MR Sensor Symposium Wetzlar 2019.

Impact on the metrology and scientific communities

The quantitative and traceable measurement of local stray fields of nanomagnetic structures is important for reliable research in the field of nanomagnetism. Impact on this scientific community was achieved via presentations at specific conferences such as International Conference on Magnetics (Intermag) 2017, and the Conference on Magnetism and Magnetic Materials (MMM) and by scientific publications with high relevance for this community such as IEEE Transaction on Magnetics. The importance of nano scale magnetic calibrations was demonstrated by the large number of invited talks by consortium members at the CPEM conference in Paris in August 2018, the world leading conference on electromagnetic metrology. One of the project's talks discussed the uncertainty evaluation of MFM calibrations.

<u>Guidelines and validated reference materials for stray magnetic field measurements:</u> "Guidelines for Nano-scale Magnetic Stray Field Measurements" have been developed within the project including descriptions and specifications of calibration routines and reference materials as well as uncertainty evaluation. They enable academic and R&D laboratories to traceably calibrate their measurement systems by following the guidelines and thus immediately underpin quantitative nanomagnetic research and development.

In the field of magnetic force microscopy the project delivered the first international round robin comparison of calibrated quantitative MFM. The round robin yielded good agreement on the stray field values of all participating Partners CMI, PTB, IFW, and NPL. This successful comparison not only proves the capabilities of the so-called tip-transfer function approach for MFM calibration but also validates the employed reference materials as a suitable tool for nano-scale magnetic field calibrations. The results of the report and the employed calibration tools and methods have entered Guidelines for nano-scale magnetic field measurements which is accessible via the project's web site.

New metrology infrastructure and CMCs: New metrology infrastructure was developed within the project and established at partners PTB, CMI, INRIM, NPL, and TUBITAK. The new infrastructure is available for measurements to the metrological and scientific community. Two high resolution metrological large range MFM are now available at CMI and PTB. High resolution MOIF setups are available at TUBITAK and INRIM.

The project contributed to strengthen the European metrology infrastructure in the field of micro- and nano-scale magnetic field measurements by stablishing at the partner institutes three scanning magnetic field microscopes, two magneto optical indicator film microscopes and four validated quantitative magnetic force microscopes. This enables a wide variety of stakeholder to receive services in the field of spatially resolved magnetic field measurements.

Early impact on European R&D networks: The new advanced European metrology infrastructure for nano scale traceable magnetic field measurements at PTB, CMI, INRIM, NPL, and TUBITAK will not only benefit industry but also academic research in the wider scientific community of spintronics. Close collaboration with various research networks such as the EU funded international training network Wall the DFG priority program SpinCaT, the DFG priority program Skyrmionics, and the EMPIR 17FUN08 TOPS project will provide direct contact with the project's new metrology infrastructure. To this extent the quantitative MFM developed and validated within NanoMag has recently been used to carry out quantitative stray field measurements of skyrmion samples within the EMPIR 17FUN08 TOPS.

Impact on relevant standards

The project has provided direct input into a draft IEC Technical Specification for a relevant written standard on nano-scale magnetic field measurements thereby significantly contributing to standardisation and international harmonisation in the field of nanomagnetic measurements and devices. Additionally, the standardization committees DKD/DKE K141 Nanotechnologie, IEC TC 68 Magnetic Alloys and Steels and EURAMET TC-EM Electricity and Magnetism have been regularly updated on the results of the project.

For the development of the new IEC standard, experts from Canada, Germany, Italy, Japan, Norway, Korea and Russia have been nominated by their National Committees and cooperate with experts of the NanoMag Standardisation Group (NMSG). This represents the first approval step towards an IEC technical specification.

The compelling scientific results of the present project allowed to draft the **first international standard in the field of nano-scale magnetic measurements**. The draft technical specification has been submitted to the IEC TC 113 "*Nanotechnology standardisation for electrical and electronic products and systems*" . At the end of the project a revised Technical Specification TS has been submitted. The publication of the final revised TS is expected after comments by the international TC members in 2020. This upcoming first international IEC standard is a milestone for the industrial and scientific community of nano-magnetism as they will enable for the first time internationally harmonized, quantitative and reliable nano-scale magnetic measurements to the benefit of international R&D, industry, and society.

Longer-term economic, social and environmental impacts

The project results delivered benefits to the European stakeholders by providing metrology infrastructure for nanomagnetic calibrations at the European NMIs; ensuring traceability of micro- and nano-scale magnetic measurements to European industry and R&D laboratories to enable a rapid development and adoption of innovative nano-magnetic technology; and by defining standards for nano-scale magnetic measurements to underpin international harmonisation and mutual recognition of worldwide nano magnetic measurements and devices. On the long term these outputs are expected to:

- Increase the competitiveness of European industries in the field of nano-magnetic systems and devices and thus strengthen the economic sector.
- Strengthen consumer safety and thus the social sector by increasing the reliability of nano-magnetic applications in health and biomedical applications.
- Enable validated measurements of nano-magnetic properties in environmental data analysis thereby strengthening the environmental sector.

6 List of publications

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