Publishable Summary for 15SIB06 NanoMag
Nano-scale traceable magnetic field measurements

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.

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.

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.

Progress beyond the state of the art

This project developed, evaluated, and validated metrological methods based on scanning magnetic field microscopy, MOIF microscopy and MFM by developing and validating traceable calibration methods models and reference materials. The project also established a European metrology infrastructure for traceable magnetic field measurements with micrometre and nanometre spatial resolution by developing high resolution spatially resolved field measurement setups at PTB, CMI, NPL, INRIM and TUBITAK. Such an infrastructure was not available prior to the start of the project. Further to this, the project developed and validated calibration artefacts for nano-scale traceable magnetic field measurements by comparative stray field imaging of calibration artefacts. This work reflects specific stakeholder needs at the start of the project and underpins traceable on-site calibrations by end-users after the end of the project.

The highest resolution traceable mapping for large areas of magnetic fields was achieved by the characterisation of individual nano-scale elements as well as of complete integrated devices. The project addressed this by developing nano-scale calibration reference materials.

Sustainable and coordinated European metrology capabilities are now provided based on these developments, traceability chains to end-users were established, and input into the relevant written standards were achieved.

Results

Objective 1: The first micro and nano scale field sensors for scanning magnetic field measurements have been designed as part of the project. Various Hall field sensors have been developed by the partners based on a range of materials such as metals, semiconductors and graphene. Characterisation of the field sensitivity, noise, and stability of these devices was completed, as well as the optimisation of Hall sensors. Complementary Metal-Oxide Semiconductor (CMOS) Hall sensors with sensor dimensions down to 1 µm and metallic Hall sensors as small as 50 nm have been fabricated and successfully tested. Giant magneto resistance (GMR) based field sensors were successfully fabricated on Silicon Nitride (SiN) cantilevers and tested for DC and AC for scanning magnetic field applications. Scanning systems with different scan range and resolution were developed, including a metrological large-scale scanning Hall system (PTB) and a scanning magneto resistive probe system at CEA.

A prototype of a nano Hall mapper system with a sliding Hall probe has been developed and tested. The system has a 3D positioning resolution of 50 nm and is equipped with CMOS Hall sensors with lateral sensor dimensions below 10 µm. The new scanning system is ready since August 2019 for commercialisation by the unfunded partner SENIS.

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 spatial resolution down to 500 nm achieving the original targets of the project. At PTB a scanning Hall probe microscope has been developed demonstrating spatial resolution down to 5 µm over millimeter scan areas. 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 since August 2019. All systems have been validated and traceably calibrated. This project achieved this objective by obtaining 500 nm spatial resolution against the starting target which included an original spatial resolution from 50 µm down to 500 nm. As consequence, this project provided new tools for providing calibrated field imaging from the centimeter to the sub-micrometer scale which is an important contribution to the European metrology landscape.

Objective 2: By the end of the project optimized magneto optical indicator films with variable thickness down to 100 nm have been developed, tested and validate. The low thickness allows an increase of the sensing volume and thus of the spatial resolution. The linearity of the new sensor films has been tested and the sensor anisotropies have been studied by ferromagnetic resonance (FMR) measurements. Additionally, different sets of micro-scale stray field reference materials have been developed, tested and validated. Quantitative modelling of the MOIF response to the stray field of the reference materials has been performed based on the measured anisotropies and magnetisation of the MOIF sensors. A good quantitative agreement with the experimental data of a calibrated MOIF microscope with 50 µm resolution has been obtained. By the end of the project two high resolution MOIF systems have been set up, characterized, traceably calibrated, compared, and validated by the partners TUBITAK and INRIM. The systems allow fast optical imaging of magnetic stray field distributions from the centimetre scale down to the optical resolution limit of 300 nm. Successful validation has been obtained as well as the comparison of measurement data on specific reference samples. The comparison took place between different partner laboratories and using different measurement techniques from MOIF to quantitative magnetic force microscopy and scanning Hall probe microscopy. This project achieved this objective by obtaining 500 nm spatial resolution against the starting target which included an original spatial resolution from 50 µm down to 500 nm. As consequence, this project provided new tools for fast calibrated field imaging from the centimeter to the sub-micrometer scale to the European metrology landscape.

Objective 3: Within the project reference materials for MFM calibration have been developed, optimized tested and validated. Hard magnetic thin films with high perpendicular magnetic anisotropy (PMA) with stripe domains were identified as the most suitable reference materials for MFM calibrations. MFM calibration algorithms based on these optimized reference materials have been developed, tested and validated by a round robin comparison. A software implementation of the optimized calibration algorithm including uncertainty analysis has been implemented in the open source scanning probe microscopy software package Gwyddion and is available for stakeholder download. The project has further delivered the world’s first large scan range metrological MFM systems at PTB and CMI.

The project further delivered the first international round robin comparison of calibrated quantitative MFM. The round robin yielded good agreement on nano-scale stray field values for 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 validated the employed reference materials as a suitable tool for nano-scale magnetic field calibrations. The results of the round robin comparison entered Guidelines for nano-scale magnetic field measurements and allowed to draft the first international standard on nano-scale magnetic field measurements. Hence, comparison, guidelines, and the international standard delivered during this project completed this objective. The results are a milestone for the industrial and scientific community of nano-magnetism as they enable for the first time internationally harmonized, quantitative and reliable nano-scale magnetic measurements.

Objective 4: During the project micro and nano-scale reference materials have been developed, fabricated, optimized and validated by traceably calibrated measurements of magnetic parameters and stray field distribution and by comparison to results of numerical stray field simulations.

As part of the project, hard magnetic thin films with high perpendicular magnetic anisotropy (PMA) with stripe domains have been fabricated and successfully used as MFM calibration reference materials. Monodomain PMA magnetic thin films have been lithographically patterned to nanometer dimensions and were tested as nano scale MFM reference materials. Electrodeposited hard magnetic materials with large film thickness and feature sizes down to 20 µm have been developed and successfully tested as large-scale stray field reference materials. These systems have been used for MFM an MOIF calibrations by all partners.

In parallel, simulation of stray field generation and sensor response was carried out for various reference materials and different field probes (e.g. Hall, MFM) to compare with experimental data and to obtain validation.

Additionally, measurements of NiFe base electrodeposited µm scale reference samples have been performed using MOIF, nano Hall mapping, and MFM, allowing for the first time a comparison of the different measurement results. A set of hard magnetic thin films with high PMA with stripe domains was selected as a reference sample for the successful round robin comparison of MFM calibrations.

With the successful MFM round robin comparison the nano-scale magnetic stray field reference materials based on hard magnetic thin films with high PMA and stripe domains have been successfully validated. These materials have also entered the guidelines and the submitted IEC standard draft document. Additionally, two further large-scale stray field reference materials based on polymer bound hard magnetic particles and on thermomagnetically patterned hard magnetic thin films were characterized and compared by different magnetic measurement techniques ranging from MOIF to scanning Hall probe microscopy and quantitative large-range MFM. Thereby, this project has successfully delivered validated stray field materials with lateral feature sizes from 250 µm down to 10 nm achieving the original objective.

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

Exploitation of high resolution scanning Hall microscopy: 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.

Exploitation of MOIF calibration software routines: 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.

Free stakeholder access to MFM calibration software: 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.

Guidelines and validated reference materials for stray magnetic field measurements: "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.

EURAMET

List of publications

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Internal Funded Partners: 1 PTB, Germany 2 CMI, Czech Republic 3 INRIM, Italy 4 NPL, UK 5 TUBITAK, Turkey	External Funded Partners: 6 CEA, France 7 GTU, Turkey 8 IFW Dresden, Germany 9 INNOVENT, Germany 10 ISC, Germany 11 Sensitec, Germany	Unfunded Partners: 12 SENIS, Switzerland
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)		