Publishable Summary for 20IND08 MetExSPM
Traceability of localised functional properties of nanostructures with high speed scanning probe microscopy

Overview

Today, complex nanostructures and nanodevices are used in photonics, quantum technology and nanoelectronics, and increasingly in healthcare and in novel materials research. Fast, accurate and traceable High-Speed Scanning Probe Microscopy (HS-SPM) has great potential for use in identifying faulty nanoproducts across multistage production processes and offers the benefits of increased productivity and reduced wastage. Conventional scanning probe microscopy (SPM) is either too slow to cover large sample areas or if fast, it lacks positioning accuracy. This project will develop essential scanning probe microscope components and ultimately a validated and traceable prototype HS-SPM measurement system suitable for use in industrial measurements.

Need

Nanotechnology is expected to grow as its use in new applications increases across many diverse sectors, from consumer goods and healthcare to energy production. For example, the progressive miniaturisation of advanced nanomanufacturing techniques which currently deliver nanodevices with feature sizes below 22 nm and complex nano-objects in the size range below 100 nm requires the introduction of fast, accurate and traceable measurement methods for the quality control of nanostructures and nanodevice dimensions and properties during production processes.

Industry, universities, and research institutes perform many high-resolution measurements; however, high resolution or high precision does not necessarily mean high accuracy. Without proper calibration and a good understanding of probe sample interactions, dimensional/property measurement errors may be as large as 30 %. At present, in the nanomanufacturing, semiconductor, nanometrology and quantum technology fields, a lack of measurement traceability to the SI metre, the associated uncertainties, and the effects of speed on measurement accuracy do not meet user requirements for higher speed (>1 mm/s) combined with larger scanning area (>1 mm^2) and better accuracy (~nm).

To turn high-speed SPMs from qualitative imaging instruments to high accuracy measurement instruments suitable for industrial quality control applications, requires the development of scanning microscope stages with far greater stability (Objective 2), improved probing systems (Objective 1) and advanced measurement strategies (Objective 3) that combine high-speed scanning with the possibility to collect local electrical or mechanical properties. The aim of this project is to enable the expansion of multi-functional probing microscopy, which is currently used for topography, to measurements of the localised functional electrical and mechanical properties of nanostructures and to produce guidance on the measurement methods needed to enable the demonstration of combined topography and electrical and/or mechanical properties as a starting point for future standardisation (Objective 4 and 5).

Objectives

The overall objective of this project is to design and develop technologies for transforming HS-SPM (~10 mm/s) metrology instruments for use in industrial high-speed quantitative multi-sensing metrology with a target traceable position measurement uncertainty of 1 nm. The developed instrument will be suitable for the industrial characterisation of functional nanostructure property combinations (electrical, chemical, mechanical, dimensional).

The specific objectives of the project are:

1. To design, develop, manufacture and characterise the frequency response, and noise level of multi-functional high-frequency (resonance frequency ω_0 = 1 MHz) self-sensing and/or self-actuating probes

and control electronics that will form a sub system suitable for a compact HS-SPM prototype designed for use in industrial environments.

2. To develop a new generation of scanning stages for HS-SPMs, which will be capable of high-speed motion (~10 mm/s) and large stroke (~10 mm) with inherent metrological traceability to the SI metre. The scanning stages should include high-speed 6-DoF interferometry sensors, which will enable real-time measurements in industry without dynamic position errors. The target position noise is 0.5 nm.
3. To develop open source experimental control and data processing software tools, for adaptive scanning and compressive sampling suitable for high speed SPM (~10 mm/s) surface nanometrology measurements over large areas (1 cm^2), using hybrid stage combinations (piezo-electric and/or MagLev). This will enable real-time traceable quantitative multi-sensing measurements (topography combined with electrical or mechanical properties).
4. To incorporate the probes, scanning stages and software tools developed in objectives 1–3 into at least one new fully assembled and characterised custom-designed prototype HS-SPM which will be capable of multi-sensing measurements for the industrial characterisation of functional nanostructure property combinations (topography combined with electrical or mechanical properties). In addition, to demonstrate its performance against other HS-AFM and/or HS-SPM by comparative measurements of reference samples and by measurements of industrially relevant samples (e.g.- an optical industry ultra-smooth optical surface, rectangular gratings, silicon samples relevant for the semiconductor industry, etc.).
5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain (accredited laboratories, instrumentation manufacturers), standards developing organisations (CEN, ISO) and end users (SPM manufacturers).

Progress beyond the state of the art

Objective 1: Multi-functional probes and control electronics:

Currently, SPM probes with silicon-based cantilevers typically use bulky optical beam deflection detector read-out sensors. Although of high sensitivity (<<1 nm) and low cost (~tens of euros) these require long set-up times (~10 min) and measurements suffer from interference from sample reflected light, which significantly impacts measurement accuracy. This project will deliver a multi-functional high-frequency self-sensing and self-actuating probe having an active cantilever operating at increased resonance frequency (~1 MHz) with moderate stiffness (<20 N/m). The probe will use a piezo resistive read-out sensor integrated in its cantilever, thus reducing set-up time as well as eliminating undesired optical interferences.

Objective 2: New generation of scanning stages for HS-SPMs

Conventional SPM scanners typically use polycrystalline piezo materials for fast small-area scanning. Although of low-cost and with high dynamic capabilities, these scanners have very poor positioning accuracy due to piezo material behaviour and strokes of less than 100 µm. To achieve long-stroke measurements, micro positioning stages using mechanical guiding are applied, but these suffer from poor dynamic properties and considerable motion errors (e.g. straightness and angular errors).

This project will deliver a novel SPM scanner based on a hybrid combination of three stages: a 3-axes monocrystalline piezo stage, a 6-axes polycrystalline piezo stage and a 6-axes large-stroke magnetic levitation (MagLev) stage. The advantages of a MagLev stage are its smooth and controllable accuracy and perfect cleanness during scan movements compared to conventional mechanically guided or air bearing systems.

Objective 3: Scanning strategies

Typical high-speed AFM collect data sets as a set of individual high-speed frames. If a large scanning area e.g., 1 mm^2 to 1 cm^2 needs to be covered, a coarse motion stage scans slowly over the sample slightly overlapping individual frames, and the collected data is merged to form a larger area image.

A general XYZ data handling approach developed in EMRP JRP IND58 6DOF will be implemented on the high-speed SPM systems developed in this project, to show both the benefits of the XYZ data processing and also those of having metrological traceability for the X, Y and Z coordinates. The XYZ data handling will also be extended towards multi-functional measurements, such as combining high-speed scanning with spectroscopic methods or with the collection of local current or mechanical properties.

Objective 4: High-speed SPM for industrial measurements

Practical use of SPMs in industrial quality control is limited by their slow scanning speed and small measurement range whilst high-speed variants are available only for qualitative instruments. AFM, traditionally a slow technique with few µm/s scan speeds, has been developed to have high-speed capabilities, but these instruments lack metrological positioning traceability. Currently over ten metrological low-speed AFM (<<100 µm/s) operating over a limited measurement range (~100 µm) exist at NMIs.

This project will turn high-speed scanning probe microscopy from a qualitative imaging process into traceable instrumentation that can be used for routine measurements to examine large area (up to 1 cm^2) samples at probesample speeds of up to 10 mm/s, with dimensional traceability to the SI metre through the use of a 6-DOF interferometry system.

Results

Objective 1: Multi-functional probes and control electronics

New types of active high frequency probes have been designed. Numerical finite element model (FEM) simulations of the different cantilever shapes and sides were performed to find the cantilever type suitable to fulfil the project requirements.

Silicon probes designs were selected based on the FE-simulations. The first design of the probes has been manufactured. State-of-the art CMOS, MEMS, wet chemical etching and dry chemical etching methods were used in the fabrication. The fabricated active probes are shown three times higher deflection sensitivity and frequencies in the range of 600 kHz. New Omega type probes were developed and produced. The probes are available for stakeholders.

Objective 2: New generation of scanning stages for HS-SPMs

Detailed designs for the long-stroke high speed scanner including a 3-axes monocrystalline piezo stage, a 6-axes polycrystalline piezo stage and a 6-axes large-stroke MagLev stage were created. Following the specification of fundamental optical, electrical and mechanical interfaces, further functionalities were added to the designs: both piezo stages feature a full invar construction; the 3-axes piezo stage features additional actuators for reaction force compensation to minimise excitation of the demonstrator frame; the MagLev stage allows for a decoupled mounting of actuator and metrology frames to minimise vibrations at the integrated metrology system; the mover of the MagLev stage features a thermal decoupling mechanism to account for different CTEs (coefficient of thermal expansion) of metrology components (near-zero expansion glass) and electromagnetic actuators (metal alloy); the glass-ceramic core of the MagLev mover features integrated mirrors for the reference metrology system of the instrument to minimise Abbe errors at the cantilever tip position during measurement.

The assembly and commissioning of the piezo stages has been completed. Tuning and characterisation of the 6-axes polycrystalline piezo stage confirmed a reliable performance well within specification with linearity errors of approximately 1 nm over 45 µm of travel and a position noise of < 200 pm or < 15 nrad respectively (standard deviation). Tuning and characterisation of the 3-axes monocrystalline stage and the piezo stage assembly are scheduled for early 2024. The components for the MagLev stage are in procurement, start of assembly is scheduled for Q1 2024, first results on the performance of the system are expected in Q2 2024.

Furthermore, preliminary studies were conducted to validate the performance of the interferometric metrology system for the MagLev stage. The individual sensors exhibit sub-nanometer resolution and sufficient robustness (tolerance to lateral displacement and tilting) for the specified 6 DOF motion range. Dedicated interferometer controller cards were designed and manufactured which contain the laser source, detection and interpolation electronics, as well as various interfaces for communication with the MagLev controller. Extensive experiments were conducted to ensure reliable temperature control of the laser diode for minimum measurement errors due to ambient temperature changes (<< 1 nm/K).

High speed motion requires synchronisation between detectors, stages, and AFM head. The data transfer interfaces were therefore carefully devised for the prototype instrument. In particular, a modular controller architecture was established for the scanning stages. A proof of concept has been provided for data transfer and synchronisation with sub-microsecond jitter. A general control system architecture was established and implemented within a real-time control environment of an industrial PC (IPC). Dedicated modules with high-resolution current drivers and interpolation electronics were designed and manufactured which interface with the IPC via fieldbus protocol at 20 kHz. Initial tests with preliminary firmware have been successfully completed to confirm feasibility and performance of the control system architecture.

Objective 3: Scanning Strategies

The requirements for the scanning algorithms and data formats have been discussed and decided upon. Compressed sensing approach has been developed and first measurements performed. Hardware at PTB and CMI were modified to be able to handle complex scan paths. Software simulation tool for generating virtual high-speed datasets was developed. An open hardware demonstrator system for using general XYZ data handling approach and adaptive scan paths was set up and made publicly available. The demonstrator system can use open source library Libgwyscan (to which new scan paths suitable for high-speed scanning were added) or scan path scripting via Lua programming language, optionally based on local statistical information, like surface roughness correlation length. The hardware demonstrator was successfully tested also on partner AFMs, at GUM and at National Physical Laboratory (project stakeholder). To process the data from both frame-by-frame scanning high-speed systems and general XYZ data handling instruments novel set of data processing modules was developed for the open source software for SPM data analysis Gwyddion and made public. This includes various data pre-processing options (drift correction, Fourier filtering, line levelling) and more complex data evaluation options (stitching, object shape fitting).

Objective 4: High speed SPM for industrial measurements

Modifications for the existing instruments to adopt the new active probes and scanning algorithms were designed at VTT and CMI. Fast interferometer and feedback loop module was developed at CMI for testing the different scan paths. First high-speed measurements on large area samples from optical industry were performed at CMI.

The different options for the design (C-type or bridge) of the prototype HS-SPM were investigated via FEM simulations. The selected design consists of a bridge type frame with the vertical mounted 6 DOF piezo stage. The frame and coarse z-stage with bearings are being manufactured. The prototypes for acoustic chamber and vibration isolation table and rags have been delivered and setup in the laboratory. A technical protocol, which defines the procedures to be used during prototype hybrid HS-SPM characterisation for short-term stability, long-term stability, noise level, maximum measurement speed, tip wear etc. is drafted further. The AFM head was investigated and tested in a different setup.

The scanning head and active probes were tested further. The 1st and 2nd resonance frequency of the active cantilevers, for later high-speed scanning, were tested.

CMI had reconstructed the aperture SNOM head to be compatible with high-speed measurement setup and tested functionality of the tuning fork probes with frequency modulated feedback loop in the Gwyscope open hardware controller. CMI also started measurements of the surface potential using FM-KPFM regime on test samples suitable for resolution vs. speed determination.

Test samples for the instrument testing were selected: two ultra-low roughness plane surfaces (SI and ULE) and rectangular grating.

Impact

The consortium has been actively taken part in standardisation activities participating in national/international standardisation technical committee meetings (e.g, METSTA, ISO, DIN, VDI/VDE) and giving input to normative documents (e.g ISO/NP TS 23879).

A project website has been set up to present an overview of the project. The consortium has produced four publications so far and three of them has already been published in peer-reviewed journals. The project was mentioned in Horizon Magazine in article titled "The small things make a big difference in the science of measurement".

The consortium has had active communication with the stakeholders. The communication methods have been emails, customer meetings and exhibitions. Stakeholder committee has been set up with 14 members and members are invited to participate in the project activities.

The project and first results were presented in New Trends in Metrology 2022 conference in Poland. The project objectives and results have been discussed within seven meetings with potential end users. The project partners have participated in Hannover Messe 2022 exhibition and BiOS Expo & Photonics West.Omega Active Cantilever for traceability of localised functional properties of nanostructures were presented in Micro and Nano Engineering Conference 2023 in Berlin, Germany.

The project results were presented in Nanoscale 2023 conference (Helsinki, Finland) in two MetExSPM sessions. An SPM Workshop was organised in April 2023 (Lednice, Czechia).

A summer school for researchers and students were organised at Wroclaw in June 2023.

Impact on industrial and other user communities

Future innovations in nanotechnologies and nanoscience are reliant on developments in nanometrology such as the improvements to measurement capabilities resulting from the implementation of HS-SPM developed in this project. The HS-SPM subsystems and methods will be directly implementable in industrial applications; SPM manufacturers will be able to use the developed techniques in their instruments which will improve their end-user measurements; and improved measurement capabilities generated at NMIs will be available as measurement services to end-users. This project aims to make commercially available new multi-functional probes and stage subsystems which can be easily installed on most commercial AFMs. The newly developed scanning stages, once commercially available, will be suitable for other applications such as coordinate measuring machines, optical measurement and inspection devices, or lithography tools. Improved traceability, and accuracy for evaluating the performance of new products or manufacturing procedures is a major goal of this project.

As a result of this project new NMI based nanometrology services will be commissioned and promoted to end-users based on upgrades to existing SPM instrumentation to assist with product development and the characterisation of innovative components and materials – helping to keep European industry competitive.

The gwyscope open hardware demonstrator has been made available for a company and there are ongoing discussions about the uptake with some other companies.

The active probes developed in the project are available for customers and have been uptaken by a collaborator.

Impact on the metrology and scientific communities

This project's main goal is traceability to the metre for high-speed SPM leading to more reliable, cost efficient nanomanufacturing. Project outcomes will be shared with the rest of the NMI community through dissemination activities within EURAMET TC-L and CIPM CCL, co-operation with non-European NMIs and directly to European Industry. This research will lead to new and improved CMC entries. The results of the project will be disseminated via conferences featuring dimensional nanometrology.

An open hardware demonstrator for advanced scanning was developed and it can be used by scientific community and microscope manufacturers to add the Gwyscan and Gwyddion related methodology for XYZ handling to their Scanning Probe Microscopes.

Impact on relevant standards

This project will generate measurement good practice guidance on characterisation for HS-SPM that has the potential for future incorporation into new documentary standards for 'high-speed SPM metrology'. The consortium has membership of ISO 201/TC201/SC9 "scanning probe microscopy" and will actively promote project results to this committee throughout the project's lifetime with a view to getting a new working group formed. The project will also have direct impact on several other standard development technical committees (e.g., ISO/TC 213/WG 16 surface texture, VDI/VDE GMA 3.41 surface measurement techniques in the micro- and nanometre range, and ISO/TC229/JWG2 nanotechnologies: measurement and characterisation). An example is surface roughness metrology, where the high-speed SPMs from this project will create highly accurate, traceable nano-positioning for high-speed, high resolution areal surface metrology for the first time. The project already has members on these standardisation working groups who will keep them informed of the results aiming for future incorporation into updated standards.

Longer-term economic, social and environmental impacts

Nanotechnology is a rapidly growing area with potential applications in many sectors of the global economy, namely healthcare, cosmetics, energy, and agriculture among others. The technology is revolutionising every industry, while attracting tremendous worldwide attention. It is making significant improvements in technologies for protecting the environment. Nanoscale devices are being used for enhanced sensing, and for treating and remediating environmental contaminants. From the energy point of view, the improved nanomanufacturing industry will result in products with less energy consumption or they will bring better energy harvesting capability. On the other hand, nanotechnology's unique characteristics may also lead to unforeseen environmental problems. One known issue is the eco-toxicity of some nanomaterials. The project directly underpins the metrological needs, for instance, for the EU legislation of nanomaterials.

List of Publications:

1. S. Metzner *et al*., "Assessment of Subsampling Schemes for Compressive Nano-FTIR Imaging," in *IEEE Transactions on Instrumentation and Measurement*, vol. 71, pp. 1-8, 2022, Art no. 4506208, https://doi.org/10.1109/TIM.2022.3204072

2. D Nečas *et al* 2023 „“Demystifying data evaluation in the measurement of periodic structures“ in *Meas. Sci. Technol.* 34 055015, https://doi.org/10.1088/1361-6501/acbab3

3. Valtr, Miroslav, et al. "Scanning Probe Microscopy controller with advanced sampling support." *HardwareX* 15 (2023): e00451. https://doi.org/10.1016/j.ohx.2023.e00451

4. R Krueger, R Gloess, “ Magnetically levitated planar motion stage with atomic resolution for metrological high-speed scanning probe microscopy“ in *Proceedings of the euspen’s 23rd International & Exhibition*, Copenhagen, Denmark, June 2023, https://www.euspen.eu/knowledge-base/ICE23149.pdf

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

Project start date and duration:		1 September 2021, 36 months
Coordinator: Virpi Korpelainen, VTT Tel: +358 504105504 E-mail: virpi.korpelainen@vtt.fi Project website address: https://www.ptb.de/empir2021/metexspm		
Internal Funded Partners: 1. VTT, Finland 2. CMI, Czechia 3. GUM, Poland 4. PTB, Germany	External Funded Partners: 5. CZ SMT, Germany 6. NA, Germany 7. PI, Germany 8. PWR, Poland	Unfunded Partners: -
RMG: -		