

Title: Traceability of localised functional properties of nanostructures with high speed scanning probe microscopy

Abstract

Scanning probe microscopy (SPM) underpins dimensional measurements, imaging, manipulation, fabrication and many other aspects of nanotechnology. However, its slow scan speed ($\mu\text{m/s}$) and limited range has restricted its industrial uptake. High-speed (HS)-SPM provides real-time imaging, but it lacks traceability and it is currently unsuitable for large area surface scanning and for simultaneously measuring topography and other parameters. Therefore, HS-SPM needs to be transformed into a real-time traceable technique that can accurately sense and measure the localised functional properties of nanostructures. Advances should include new high speed scanning techniques, functionalised SPM probes, compressive sampling and the establishment of appropriate data formats and methods for processing large datasets.

Keywords

High-speed scanning probe microscopy, large area scanning, localised functional properties, modelling, multi-sensors, nanotechnology, scanning stages

Background to the Metrological Challenges

SPMs are primarily used in nanotechnology and materials science for imaging and for the measurement of dimensions. Other SPM variants are used to measure thermal, magnetic, electrical, chemical, mechanical and other physical properties. In addition, SPMs are used for manipulation, fabrication and to determine the position of measurands. Applications include measurements of solar cells, organic electronics, energy harvesting materials and photonic and optoelectronic devices. Industrial use for material characterisation is becoming more widespread, but the slow scan speed ($\mu\text{m/s}$) and limited range of SPM is restricting its uptake, despite its extremely high spatial resolution. HS-SPM offers the same resolution, but with acquisition rates of tens to thousands of frames/second providing real-time (video rate) imaging. However, there is no traceability for positioning. HS-SPM is currently mainly designed for imaging rapid phenomena over small areas (often $< 10 \times 10 \mu\text{m}^2$) unless data is stitched together. Therefore, at the moment HS-SPM is unsuitable for large area surface scanning and for simultaneously measuring topography and other parameters.

For large area measurements, in the order of a few cm^2 , scan speeds are currently comparable to conventional SPM ($\mu\text{m/s}$). HS-SPM instruments are currently unsuitable for large area measurements mainly due to their complexity, inflexibility and to the lack of tools for data interpretation. In addition, heavy data post processing is often required to build high-quality images and there are no standard data formats for this at the moment. For large area measurements, commercial HS-SPM instruments are orders of magnitude slower than conventional SPMs and they still have an insufficient scanning range (up to a few $100 \mu\text{m}$), a lack of tools for advanced data interpretation and limited accuracy. Although some specialist systems exist for the semiconductor industry, these are not widely available. Therefore, technologies need to be designed and developed for transforming HS-SPM from a qualitative imaging process into traceable instrumentation that can be used to examine large area (cm^2) industrially relevant samples at probe-sample speeds of $\sim 10 \text{ mm/sec}$ with complete uncertainty analysis.

Functionalised SPM probes can provide nanoscale electrical, chemical and optical characterisation, but these signals are not quantitative, and the dimensions and positions of features cannot always be correctly inferred. In addition, these measurements are often obtained independently from dimensional measurements via sequential scanning in different SPM modes. Therefore, to speed up the characterisation process the different SPM modes need to be used simultaneously and this data needs to be used to improve the accuracy of the dimensional image by adapting it in real time according to the data received. SPM needs to become a hybrid process adapted system that is responsive to the sample. Therefore, multi-functional high frequency

(resonance frequency $\omega_0 = 1$ MHz) self-sensing and/or self-actuating SPM probes, and the associated electronics, need to be developed for use in compact HS-SPMs in industrial environments.

In addition, a new generation of scanning stages will be required for high speed SPM in order to run more complex measurement sequences related to e.g. volume data and other physical quantities. The design of the scanning stages needs to be simplified so that they are capable of high-speed motion and large stroke with inherent metrological traceability. The scanning stages will need to include high-speed interferometry and capacitance sensors to enable real-time measurements in industry without dynamic position errors and the target uncertainty will need to be around 1 nm. One approach would be to use different variants of sparse sampling, such as adaptive sampling, which only focuses on the relevant parts of the sample, and another approach would be to use statistical methods such as compressed sensing.

Models of the probe sample interaction need to be developed in order to establish traceability. The measured data also need to be analysed to find any discrepancies between different methods and to determine the impact of humidity, surface contamination and probe wear on the results. Probe parameter calibrations and numerical corrections in real-time, or with post processing, are also essential for reliable measurements. Novel volume-based measurement regimes, e.g. PeakForceTUNA, can provide suitable datasets for such corrections, but the methodology is not sufficiently developed to perform them. Current numerical models are typically too slow, complex and computationally demanding for use in routine analysis, and reference samples are missing for such measurements. Therefore, new custom-designed fully characterised HS-SPM instruments need to be developed by the NMIs, which are capable of real-time traceable quantitative multi-sensing metrology. These systems will then be used by the industry to locally characterise the functional properties of nanostructures.

Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the protocol.

The JRP shall focus on the traceable measurement and characterisation of the localised functional properties of nanostructures using high speed scanning probe microscopy.

The specific objectives are

1. To design and develop technologies for transforming high-speed scanning probe microscopy (HS-SPM) from a qualitative imaging process into traceable instrumentation that can be used to examine large area (cm^2) industrially relevant samples at probe-sample speeds of ~ 10 mm/sec with complete uncertainty analysis.
2. To develop multi-functional high frequency (resonance frequency $\omega_0 = 1$ MHz) self-sensing and/or self-actuating probes and control electronics for compact HS-SPM systems designed for use in industrial environments.
3. To develop a new generation of scanning stages for HS-SPMs, which will be capable of high-speed motion and large stroke with inherent metrological traceability. The scanning stages should include high-speed interferometry and capacitance sensors, which will enable real-time measurements in industry without dynamic position errors. The target uncertainty should be around 1 nm.
4. To incorporate the items developed in objectives 1 - 3 into at least 1 new custom-designed HS-SPM instrument. The final fully characterised system(s) should be capable of the real-time traceable quantitative multi-sensing metrology that is needed by industry to locally characterise the functional properties of nanostructures.
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).

Proposers shall give priority to work that meets documented industrial needs and include measures to support transfer into industry by cooperation and by standardisation. An active involvement of industrial stakeholders is expected in order to align the project with their needs – both through project steering boards and participation in the research activities.

Proposers should establish the current state of the art, and explain how their proposed project goes beyond this. In particular, proposers should outline the achievements of the EMPIR project 15SIB09 3DNano and how their proposal will build on this.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 1.5 M€, and has defined an upper limit of 1.8 M€ for this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 30 % of the total EU Contribution across all selected projects in this TP.

Any industrial partners that will receive significant benefit from the results of the proposed project are expected to be unfunded partners.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community, describing how the project partners will engage with relevant communities during the project to facilitate knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the “end user” community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

- Address the SRT objectives and deliver solutions to the documented needs,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the nanotechnology sector.

You should detail other impacts of your proposed JRP as specified in the document “Guide 4: Writing Joint Research Projects (JRPs)”

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include the best available contributions from across the metrology community. Specifically, the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work.

Time-scale

The project should be of up to 3 years duration.