Publishable Summary for 15SIB09 3Dnano
Traceable three-dimensional nanometrology

Overview

The overall goal of this project is to meet current and future requirements for traceable 3-dimensional (3D) metrology at the nanometre level with measurement uncertainties below 1 nm. To achieve this goal the project will set up to establish new routes for traceability, further develop existing instruments and validate 3D measurement procedures. Additionally, this project will develop new calibration artefacts and made them available to industry as traceable reference standards to enable valid comparison of fabrication and measurement results, and establish a robust basis for design of objects with traceable nanoscale dimensions and tolerances.

Scanning Probe Microscopes (SPMs) available in national metrology institutes (NMIs) have low uncertainties, are traceable to the SI-metre and significantly outperform commercial SPMs in accuracy. However, there is a large gap between SPMs and the rest of 3D metrology. This project aims is to further develop SPM instrumentation, measurement procedures, data interpretation and reference materials to bridge this gap, as proper understanding of probe-sample interactions is crucial for the reduction of measurement uncertainty.

Need

Nanotechnology is increasingly used in different sectors e.g. health, medicine, nanoelectronics, nanophotonics and nanostructured materials, and the market for final products incorporating nanotechnology is estimated to have increased ten-fold during the current decade. The progressive miniaturisation of advanced nanomanufacturing techniques that currently deliver nanodevices with feature size below 22 nm and the extensive use of complex nano-objects e.g. wiring, surface coatings, nanoparticles in consumer products in different application e.g. semiconductors, health and medical products, has therefore driven the need for improved accuracy in 3D nanometrology.

High accuracy measurements are needed in R&D and quality control, as many health and environmental effects of nano-objects and nanoparticles are dependent on the size and shape of structures. From a regulatory perspective, traceability is a must for measurement techniques, as if measurements are not traceable, they have little value from a judicial point of view. Currently there is insufficient traceability to the SI metre for true 3D nanomeasurements, because the existing level of uncertainty in measurements (5 nm) does not meet the requirements of industry and scientific research. 3D metrology differs from one directional (1D) height and pitch metrology, where the impact of tip geometry and the tip sample interaction at the top/bottom planes are self-compensating. Instead, measurements of 3D structures are bi-directional, where the effect of tip geometry and tip sample interactions at the left and right sidewalls are different. Measurement of line width is an example of bi-directional measurements. In addition, more and more complex particles are put on the market and used in processes, and these particles have different shapes e.g. rod, star, donut shape, which require real 3D characterisation. Furthermore, although high resolution instruments exist in industry, universities and research institutes, this does not necessarily result in high accuracy, as without proper calibration and understanding of probe-sample effects traceability is rarely established and errors in simple measurements can be up to 30 %. To achieve better uncertainty for 3D measurements the existing metrological Atomic Force Microscopy (AFMs) also need to be improved and lower noise, higher speed and large range are needed.

Hybrid metrology i.e. merging the measurement results from either different tools (instrument fusion) or different channels of a single tool (data fusion) is a promising way to measure complex nanostructures, however new algorithms and software are needed for data fusion. Reference materials are also needed for the traceable calibration and characterisation of measurement instruments to enable the valid comparison of measurement results and to establish a solid basis for the design of objects with traceable nanoscale dimensions and tolerances. Furthermore, new types of reference materials are needed for 3D measurement and probe characterisation.

Objectives

The scientific and technical objectives of this project are to:

1. Reduce the 3D nanomeasurement uncertainty, by means of a bottom up approach, to less than 1 nm for nanodimensional measurands (including line width, height, pitch, and edge/width roughness) on engineered nanostructures and nanoparticles. In addition, to reduce the noise level of metrological AFMs (MAFMs) to 0.1 nm (rms) using a top-down approach, to raise the scanning speed up to 1 mm/s, and to extend the scanning range up to 25 mm/s by further developing the state-of-the-art optical and x-ray interferometry (XRI).
2. Develop reference materials for 3D nanometrology tools including AFM and Scanning Electron Microscopy (SEM). In particular, to realise test structures for characterising the tip geometry in AFMs and the beam size in SEMs and reference standards for width and sidewall roughness measurements.
3. Widen the understanding of probe-sample interactions in AFM and SEM measurements for reducing the measurement uncertainty. In particular, to study the tip-sample interaction force of AFM line width and nanoparticle measurements; to model the image formation of SEM; and to investigate the influence of humidity on AFM measurements.
4. Develop a hybrid metrology for merging measurement results from either different tools (e.g. AFM, SEM, Transmission Electron Microscopy (TEM), Mueller polarimetry and optical scatterometry) or different channels of a single tool.
5. Facilitate the take up of the technology and measurement infrastructure developed by the project by the measurement supply chain (accredited laboratories, instrument manufacturers) and end users (semiconductor industry, precision engineering, optical industry and nanoparticle researchers).

Progress beyond the state of the art

Reduction of i) 3D measurement uncertainty to less than 1 nm, ii) noise level of MAFMs to 0.1 nm (rms) and iii) raising the scanning speed up to 1 mm/s and extending the scanning range up to 25 mm

i) measurement uncertainty

This project aims to reduce 3D measurement uncertainty to below 1 nm. In order to establish traceability for this challenging target, a new “bottom-up” approach will be developed using the cross-section of TEM structures with atomic resolution. In this way, the CD can be directly linked to the atomic spacing in the crystal lattice, which is traceably calibrated using a combined optical interferometry and XRI.

Additionally, tools and measurement procedures will be optimised by the project for true 3D measurands, and AFM tips will be characterised and developed for metrology at the nanometre range. The AFM probes selected for metrological measurements include carbon nanotube (CNT) probes, CD flared probes and probes with a spherical nanoparticle tips (SPH).

ii) noise level

This project will address current noise, range and speed limitations of MAFMs. Improved displacement measurement sensors will be used in MAFMs to lower noise levels (0.1 nm rms) and reduce nonlinearity errors.

iii) scanning speed and range

To address the current speed limitations of AFM metrology and progress beyond the current state of the art, high speed AFM heads will be developed that are suitable for NMI based instrumentation as well as video rate AFMs (scanning speed over 1 mm/s). Metrology will also be applied to video rate AFMs that surpass the current speed of MAFMs. The range of MAFM positioning stages will also be increased up to 25 mm.

Development of reference materials for 3D nanometrology tools

This project will design, manufacture and characterise new LER/LWR reference materials, new CD standards and new tip characterisers. The line width samples will have either 1D line patterns, 2D pillar arrays or 2D hole arrays with different pitches (from 10 µm to 50 nm) and with a variety of line/space ratios 10:1 to 1:1. Varying edge roughness will also be included when possible. Samples for hybrid metrology will be produced, which have a pattern feature consisting of both dense lines and isolated lines, and commercially available nanoparticle samples suitable for calibration purposes will be selected and characterised. The target uncertainty for the reference materials is < 1 nm.

Improvement of the understanding of probe-sample interactions in AFM and SEM measurements

In this project, tip probe-sample interaction in true 3D AFM measurement will be simulated. Critical issues such as structure/tip deformation due to the measurement force and humidity will be theoretically and experimentally studied. A special reference material will be developed to improve the metrological characterisation of the AFM probe, and the simulation of SEM measurements will include a physical model of SEM image formation of nanodimensional reference structures for the quantitative analysis of SEM images for CD and shape metrology. Finally, the impact of SEM image noise on LER measurements will be investigated.

Development of industrial solutions for hybrid metrology

This project will develop a feasible topology for hybrid metrology. Furthermore, a method and software for data fusion from different tools (i.e. TEM, AFM, SEM and optical scatterometer) will be developed for the measurement of i) CD and roughness and ii) complex nanoparticles or agglomerates of nanoparticles.

Results

Reduction of i) 3D measurement uncertainty to less than 1 nm, ii) noise level of MAFMs to 0.1 nm (rms) and iii) raising the scanning speed up to 1 mm/s and extending the scanning range up to 25 mm/s

A new bottom-up approach will be used for the cross-section of TEM structures with atomic resolution. In such a way, the CD can be directly linked to the atomic spacing in the crystal lattice, which will be traceably calibrated using a combined optical interferometry and XRI. Additionally, tools and measurement procedures will be optimised for true 3D measurands, and AFM tips will be characterised and developed for metrology at the nanometre range.

The developed new methodology for the bottom-up traceability routes has been further improved. NPL with Queensgate has completed the design of translation stage for XRI. Test results for noise level and resolution are encouraging and show that the design should meet specifications. Vibration measurements made at NPL showed that the existing isolation was now always sufficient. A new active isolation system has been purchased and a frame for its support is under construction.

NPL has obtained an image over a 1 mm x 25 µm area demonstrating the large scanning range of the system. Work is in progress to improve the scanning range in Y from 800 µm to large area scans. . Further work will be carried out to mitigate the effects of extraneous Z motion of the scanning stages in HSAFM. Work has started on the uncertainty budget. The scan speed is in excess of 1 mm per second for the high-speed stage.

The installation of the new collimating optic for the x-ray source for the XRI is almost completed at NPL and PTB.

The development of 3D MAFMs, long range 3D/CD AFM and high speed metrological large range AFM is ongoing:

- At PTB the mechanical and electronical development of the instruments is finished. The mechanical construction and manufacturing of low-noise 3D/CD AFM and a high speed metrological large range AFM are ready. Additionally, an automatic tip changer system is ready and tested. A signal processing and controller have been designed. The development of the signal processing and controller is finished and tested; (2) the measurement software is implemented; (3) first test measurements by the developed metrology tool are performed successfully.
- The AFM head that combine an optical interferometer for measuring the tip height and an optical lever for measuring cantilever bending/torsion is under improvement. Actually all parts are delivered and mounted. The optical adjustment is in progress.
- The MAFM using self-sensing probes is improved: a new AFM head for self-sensing cantilevers was constructed and manufactured.
- A fibre optic interferometer head for CMI's metrology AFM was developed and first tests with NPL’s electronics carried out. CMI has developed data processing software based on general xyz data to simplify the multiple data fusion in the high speed AFM measurements, applicable for both high-speed systems

- Uncertainties on the NPL HSAFM are being evaluated and a paper on its design and operation showing initial results is under preparation.
- VTT has tested the designed interferometric system, designed the new long-range AFM setup with tilting option and purchased the components. The alignment of the instrument has started.

Development of reference materials for 3D nanometrology tools

Four nanoparticle samples of different size, shape and composition were selected and purchased. Supplier specifications were distributed to the relevant consortium partners.

Simulations on the Siemens star structure with different Line Edge Roughness (LER) and other parameters were performed to further optimise the structure. For the Siemens stars, a process with e-beam patterned hydrogen silsesquioxane (HSQ) was first tested. Due to some external setbacks for this process, only first tests were done with proper tool functionality and HSQ material for the Siemens stars. Hence, the process optimisation for the Siemens stars in HSQ on silicon continued. After an intermediate measurement of a test sample at PTB, it was decided to etch in the HSQ Siemens star pattern into the silicon by reactive ion etching (RIE). Three sets of samples were prepared and sent out for round robin tests at all interested consortium partners namely SMD, PTB, DFM, METAS, CMI, VSL, VTT & NPL. Fabrication of 1 spare set was included. A sample set consists of two samples where one sample has Siemens stars in HSQ on a silicon substrate and the other sample has Siemens stars etched in the silicon substrate.

From the results of the measurements of the partners, the samples with Siemens stars are considered suitable as reference sample for optical measurements but not yet for AFM measurements. Main concerns are the edges that need to be steeper and sharper, as well as the edge roughness. The partners decided to continue with developing samples where the Siemens stars are etched in the silicon as the expectation is that this will improve the side wall roughness, steepness and sharpness. For the second run, the design will be updated and a new process flow will be designed. Initial tests for lithography have started.

Processing of all layouts for nanopillar gratings was performed and preliminary Electron Beam Lithography (EBL) tests were carried out in association with pattern transfer via RIE. First results were used to optimise the fabrication process parameters. Fabrication of pillar samples on square lattice has started and the first SEM images show promising results. Hybrid metrology measurements of nanopillar samples with AFM, Mueller polarimetry and scatterometry is in progress. The preparation of samples of tobacco mosaic virus (TMV) is in progress at INRIM in cooperation with a parent Institute (CNR-IPSP), and preliminary characterisation of the samples has just started.New types of calibration samples for mostly optical methods were tested in cooperation with the University of Helsinki.

Improvement of the understanding of probe-sample interactions in AFM and SEM measurements

Limits of elastic probe-sample interaction in SPM were estimated and modelling of probe-sample interaction using mass-spring model performed. DFM has coordinated with SMD to choose silicon grating and nanoparticle samples for investigating the effect of humidity on probe-sample interactions. Measurements are in progress. Experimental studies on the probe-sample interaction of existing HAR SSS AFM tips with sidewalls having various slopes is ongoing.

AFM tip-sample interaction at different environmental conditions were investigated. AFM experiments were carried out in a climate chamber on different samples (nanogratings and nanoparticles) at different humidity levels and temperatures. The results are under discussion. The study of the tip-shape-substrate corrections has been continued by using samples of TMV on mica and by focusing on models and test runs to determine the elastic modulus of these plant nanostructures.

Development of industrial solutions for hybrid metrology

The developed data fusion methodology of AFM and TEM data for traceable hybrid true 3D nanometrology was applied in the reference metrology of industrial photomask standards, and its feasibility proved. Initial tests were conducted to combine AFM data with scatterometry data obtained on several samples (i.e. gratings).The existing data fusion module for Gwyddion open source software was tested for mechanical data interpretation (e.g. combination of topography and deformation (or adhesion) channel in PeakForceQNM regime). The developed mechanical interaction models are basis for estimation of the uncertainties in situations where an elastic response is expected between probe and sample. The methodology for combining different sources and use of different channels is being developed.

A compact scatterometry setup for industrial adoption was developed. Integration of scatterometry with AFM will commence in the next reporting period. Further development of SEM and AFM data fusion methodology with modelling of AFM tip effects on rough surfaces to be used in the validation of the method is ongoing.

Impact

Thus far, the project results have been disseminated mainly by direct contact with industrial stakeholders. Nevertheless, open access peer-reviewed papers have been published and the project and its results have been presented at different conferences. The partners have disseminated the project and its results in workshops and in one-to-one training sessions.

Impact on relevant standards

The consortium has had active communication on EURAMET Technical Committee in Length (TC-L), Consultative Committee in Length (CCL) and CCL Working Group Nano (WG-N) on the new *mise en Pratique* for realisation of metre on nanometre scale by Si lattice parameters. The bottom-up methods will be included in the new *mise en Pratique*. In addition, the consortium has contacts with an Italian standardisation body (UNI/CT 047).

Documentary standard ISO 11952: -- “Scanning-probe microscopy -- Determination of geometric quantities using SPM: Calibration of measuring systems for the dimensional calibration of SPM instruments” has been revised under PTB lead.

Impact on industrial and other user communities

The consortium has performed reference metrology of industrial photomask standards, which confirms the feasibility of the bottom-up and top-down metrology methodology developed in this project. The successful photomask standard case shows high potential that the method will be taken-up by industry.

PTB has initiated collaboration with a company to exploit the developed 3D nanometrology to investigate the new industrial CD standard. The measurements results are expected to improve the products of an industrial stakeholder.

PTB is collaborating with a world-leading equipment vendor to exploit the developed 3Dnanometrology to measure three dimensional parameters (including height, width, sidewall angle, sidewall profile etc.) of complex nanostructures. This is a challenging and important metrology task required for realizing e.g. the next generation lithography (NGL) technologies.

VTT and PTB have received several types of optical specimens (e.g. micro lens array MLA concave 6 µm lens, MLA convex 6 µm lens and MLA convex 1.5 µm from industrial stakeholders. The measurements of 3D geometry of these optical features will be carried out soon. The collaboration will be part of the demonstrations on measurement capabilities for industry.

INRIM has continued a collaboration with a parent Institute (CNR-IPSP Institute for Sustainable Plant Protection) for developing the preparation of samples with known plant nanostructures.

DFM has developed a compact scatterometry setup and delivered it to the NIL technology ApS for field measurements on nanostructured surfaces.

CMI has further developed a software module for Gwyddion for data fusion purposes. The software is free and available at http://gwyddion.net/. Thousands of new downloads in the last project period indicates its great impact for public communities.

To ensure the industrial impact the calibration standards produced in the project have been designed in close cooperation with the stakeholders.

Impact on the metrology and scientific communities

The key impact to the metrological and scientific communities, from this project, will be the traceability of 3D nano measurements to the SI metre, which will enable NMIs to offer metrological support to industry. Currently the traceability chain is broken because the uncertainty levels of traceable measurements at NMIs do not meet the requirements of the scientific community and industry. In addition, the project’s research will lead to new and improved draft Calibration and Measurement Capabilities (CMC) statements. This project will lead to progresses in advanced dimensional nanometrology that is needed for the development and expansion of European high technology industries.

Three collaborators are actively involved in the project: University of Helsinki, Technische Universität Ilmenau and Institute for Sustainable Plant Protection. University of Helsinki has provided nanostar samples that the partners will use for measurements with different methods. If possible, the results will be published in a coauthored peer-reviewed paper.

The University of Technology Ilmenau, has verified with a frequency comb and a GPS system all three frequency-stabilized lasers (axes x, y, z of the FAU nanopositioning and manomeasuring machine).

Institute for Sustainable Plant Protection (IPSP) supplies freshly grown bio-plant nanostructures, namely Tobacco Mosaic Virus (TMV). TMV has a rod shape with a diameter of about 18 nm, which represents a natural reference as tip characterizer for metrological Atomic Force Microscopes.

Longer-term economic, social and environmental impacts

Nanotechnologies and advanced materials have potential to offer valuable solutions in the health, energy, climate and environmental sectors, leading to economic growth in Europe.

Advanced dimensional nanometrology is an enabling technology for the manufacture of nanotechnologies and research and therefore needed for re-industrialisation of Europe and for creating European wealth. It is essential for European re-industrialisation that enabling technologies supporting the development of nanotech products exist for European nanotechnology R&D.

The advanced nanometrology capabilities to be developed in this project will benefit nanotechnology industries. By developing appropriate and urgently needed nanometrology capabilities and by bringing traceability for various nanoscale measurements, the project will provide competitive advantages for companies at a European and worldwide level and support future innovation. Furthermore, this project will in long term enhance the growth of nanotechnology industries e.g. the semiconductor, nanomaterial, nanophotonics/optics, microscopy and sensor industries.

Nanoscale devices are used for enhanced sensing as well as for the treatment and remediation of environmental contaminants. In the future, it might be possible to prevent pollution with the help of nanotechnology.

In regard to energy, an improved nanomanufacturing industry will result in products with lower energy consumption, and it could lead to better energy harvesting capability. For example, the use of nanotechnology, more specifically organic photovoltaic cells instead of silicon crystal solar cells is making solar power cheaper. Nanometrology could help control nanoscale morphology, which could significantly increase the conversion efficiency of photovoltaic cells.

Clinical tests for nanomedicine and nanotoxicology lack comparability of data. Much progress has been made in labs around the world, but *because the measurement results are not traceable*, they cannot be compared and few valuable conclusions can be drawn. The traceable methods developed in this project may help this. Furthermore, before any nanomedicine product is investigated in clinical tests, reliable measurement datasets are required, for which traceability is crucial.

List of publications

Gaoliang Dai, Kai Hahm, Harald Bosse and Ronald G Dixson, "*Comparison of line width calibration using critical dimension atomic force microscopes between PTB and NIST*", Meas. Sci. Technol. **28** (2017) 065010, https://doi.org/10.1088/1361-6501/aa665b.

Gaoliang Dai, Ludger Koenders, Jens Fluegge, and Matthias Hemmleb, "*Fast and accurate: high-speed metrological large-range AFM for surface and nanometrology*", Meas. Sci. Technol. **29** (2018) 054012, https://doi.org/10.1088/1361-6501/aaaf8a.

Andrew Yacoot, Petr Klapetek, Miroslav Valtr, Petr Grolich, Herve Dongmo, Giovanni Mattia Lazzerini and Angus Bridges, "*Design and performance of a test rig for evaluation of nanopositioning stages*", Meas. Sci Technol. https://doi.org/10.1088/1361-6501/aafd03

Johan Nysten, "*A characterization and uncertainty estimates for MIKES MAFM*", Master thesis, https://jyx.jyu.fi/handle/123456789/62847

Project start date and duration:	01 October 2016, 36 months	
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Internal Funded Partners: 1 VTT, Finland 2 CMI, Czech Republic 3 DFM, Denmark 4 NPL, United Kingdom 5 PTB, Germany 6 SMD, Belgium 7 VSL, Netherlands	External Funded Partners: 8 FAU, Germany 9 NRCS Demokritos, Greece 10 TNO, Netherlands	Unfunded Partners: 11 INRIM, Italy 12 METAS, Switzerland