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

Overview

The overall goal of this project was to meet future requirements for traceable 3-dimensional (3D) metrology at the nanometre level with measurement uncertainties below 1 nm. To achieve this goal the project set up to establish new routes for traceability, further developed existing instruments and validated 3D measurement procedures. Moreover, the project developed new calibration artefacts that can be used in 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.

Need

Nanotechnology is increasingly used in different sectors e.g. health, medicine, nanophotonics and nanoelectronics. 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 and the extensive use of complex nano-objects of different shapes (rod, star, donut shape, etc.) has 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 demanded for the measurement techniques; if measurements are not traceable, they have little value from a judicial point of view. At the start of the project, there was insufficient traceability to the SI metre for true 3D nano measurements, because the level of uncertainty in measurements (5 nm) did not meet the requirements of industry or scientific research.

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 was a large gap between these SPMs and real practical 3D measurements. To bridge the gap, instruments with lower noise, higher speed and larger range and new types of reference materials needed to be developed. Hybrid metrology is a promising way to measure complex nanostructures but it lacked data fusion software to make it a practical method.

Understanding probe-sample interactions is a key for improving uncertainty in measurements of 3D nano-structures. Prior to the start of the project, tip probe-sample interaction studies were limited to measurements of nearly flat surfaces, only. Without proper calibration and understanding of the probe-sample effects, traceability is rarely established and errors in simple measurements can be up to 30 % even with high-resolution instruments.

Objectives

The overall objective of this project was the realisation of traceable calibration services for three-dimensional nanometrology with an uncertainty less than 1 nm. The technical objectives of this project were 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 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 SEM. In particular, to realise test structures for characterising the tip geometry in AFMs

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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, 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

The project targeted to reduce the 3D measurement uncertainty from 5 nm to below 1 nm. In order to establish traceability for this challenging target, a new “bottom-up” approach was developed using the lattice of crystal silicon as an internal ruler. In this way, the critical dimension (CD) can be directly linked to the atomic spacing in the crystal lattice, which is traceably calibrated using a combined optical interferometry and XRI. A complete set of tools (including the metrology tool and software packages) for realising this traceability routine was realised. Critical dimensions of complex nanostructures can now be measured with an uncertainty < 1 nm.

ii) Noise level

Traceability for MAFMs is made via optical interferometry and typically has a noise limit of 0.7 nm (rms). The noise level in different long-range MAFMs was reduced to 0.1–0.4 nm (rms) by using e.g. vibration isolation, acoustic shielding and thermal isolation techniques as well as improved displacement measurement sensors.

iii) Scanning speed and range

Prior to the project, the limited scanning range and speed restricted the measurement capabilities of metrological AFMs. The project increased the scanning range and speed of MAFMs by redesigning them and by upgrading various key components. With these developments high-speed (in access of 1 mm/s) and large range (up to 25 mm) metrological AFMs were successfully realised.

Development of reference materials for 3D nanometrology tools

Different types of high quality reference materials are needed for characterising the tip geometry of Atomic Force Microscopes (AFMs) and the beam size of Scanning Electron Microscopes (SEMs), and for experimentally studying tip/sample interactions, which are essential for verifying theoretical modelling. In addition, reference materials with well-defined structures are needed to progress 3D-nanometrology, hybrid metrology and the data fusion of multi tools. This project designed, developed and characterised new reference materials including the Siemens star, nanopillar array and nanoparticle standards. The reference standards show good potential for new 3D reference standards for AFM and/or optical measurement methods, which were not available prior to the project.

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

Understanding probe-sample interactions is a key for improving measurement uncertainty. Prior to the start of the project, AFM tip probe-sample interaction studies were limited to measurements of nearly flat surfaces, only. In this project, tip probe-sample interaction in true 3D AFM measurement was simulated and a set of reference probes and samples developed to perform novel types of experiments for probe-sample elastic deformation analysis. Critical issues such as structure/tip deformation due to the measurement force and humidity were studied theoretically and experimentally. A physical model of SEM image formation of reference nanostructures for the quantitative analysis of SEM images for CD and shape metrology was developed.

Development of industrial solutions for hybrid metrology

New methods as well as algorithms and software for data were developed for hybrid metrology i.e. merging the measurement results from either different tools (instrument fusion) or different channels of a single tool (data fusion). In instrument fusion, optical methods were successfully combined with AFM for characterising reference materials. In data fusion, fusion of datasets from multiple SPM measurements of the sample was used to reduce the scan artefacts and methods for matching the measurement data of the Transmission

Electron Microscopy (TEM) and AFM were developed for realising the bottom-up approach. Excellent results were achieved, for instance, a combined standard measurement uncertainty of the reference critical dimension (CD) value is estimated as 0.81 nm. The results strongly support the recommendations of applying hybrid metrology in industry.

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 traceability approached was successfully developed and realised. Using this approach, the 3D dimension of complex nanostructures is determined by using the lattice of crystal silicon as an internal ruler, where the lattice constant was calibrated accurately and traceably by x-ray interferometers (XRI). High-resolution transmission electron microscope has been applied to measure sample lamellas with a true atomic resolution, where the sample lamellas are prepared from the pre-defined sample structures using a focused ion beam (FIB) facility. In addition, to overcome the problem that the sample preparation by the FIB technique is destructive, hybrid metrology method where the data fusion technique is applied to combine the AFM and TEM results was used. The final measurement uncertainty budget indicates that the critical dimension (CD) of structures can be determined at a combined standard measurement uncertainty of 0.81 nm. Results from this work have contributed to the realisation of the lattice parameter of silicon as a secondary length standard in the Mise en Pratique for the metre revised in May 2019.

In this project, various measures were studied and undertaken to reduce the noise level of MAFMs.

- at PTB, some key components were upgraded for easier adjustment, better thermal behaviour and better stability. With these improvements, the noise level of the metrology tool was reduced significantly, for instance, the positioning noise along the z-axis has been improved from $1\sigma = 0.52$ nm to $1\ \sigma = 0.13$ nm.
- at NPL, by installing a new scanning stage into its metrological AFM, the noise levels measured in the x, y and z axes are 0.25 nm, 0.55 nm and 0.31 nm. A further reduction is possible with more averaging of collected data to achieve the target of 0.1 nm rms. Noise levels in NPLs' *high-speed* metrological AFM are between 0.1 nm and 0.4 nm depending on the mode of operation, the density of data points collected and the amount of averaging. This is the first time 3 D metrology has been brought to high-speed AFMs used for non-NMI applications.
- VTT redesigned its Metrological Atomic Force Microscopy (MAFM) for 3D measurements and updated the software to reduce the noise level. A noise level of 0.2 nm in interferometric measurements of the Z-direction was achieved.
- FAU undertook various efforts to reduce the noise level of her NMM-1, which is used for the LR-AFM. So the NMM-1 was positioned on pendulum isolators to reduce vertical and especially lateral vibrations. Signal processing problems of the autocollimators for the angular control of the NMM-1 positioning stage were eliminated. A new housing for acoustic shielding, thermal isolation and temperature control was constructed and installed. With the self-sensing AFM on the NMM-1 a noise level of 7 nm is reached (z-axis), which can be improved to 0.2 nm by filtering.
- VSL demonstrated 0.1 nm rms noise level for their 3D AFM.

To raise the measurement speed of metrological long range AFMs, further improvements were undertaken:

- with the developments made at PTB, high-speed (up to 1 mm/s) and large range (up to 25 mm) metrological AFMs were successfully realised. Verification measurements indicated that the tool can calibrate the step height standards with a stability of better than 0.8 nm (p-v), and calibrate the pitch value of a 2D grating (nominal pitch of 1000 nm) with a stability better than 0.01 nm (p-v), when the measurement speeds are increased from 10 µm/s to 1000 µm/s (i.e. 100x times faster).
- NPL brought metrology to a non-NMI high-speed AFM with following results: an average speed in the fast axis that is higher than the target 1 mm/sec; in fact, approaching 9 mm/sec. The high-speed stage with a range of 4 µm × 4 µm is mounted on an x-y slip stick stage having a larger range (25 mm x 25 mm).
- VTT reconstructed its MAFM to achieve a larger measurement volume (950 µm × 950 µm × 115 µm).
- FAU developed a new compact optical AFM head, which includes a laser interferometer for measuring the height and simultaneously incorporates an optical tilt measurement to acquire the orientation of the cantilever (bending and torsion). Integrated into a NMM it offers a scanning range up to 25 mm. FAU also constructed and manufactured a new AFM head for self-sensing cantilever to be integrated into NMM. Using that MAFM, a scanning range of 10 mm by scanning speed 5 µm/s was realised.

Development of reference materials for 3D nanometrology tools
Based on the outcome of a survey among stakeholders, novel standards for AFM probe size determination and SEM beam size evaluation were designed and manufactured and also prepared from existing nanomaterials. Intermediate evaluation of the first production batches was used to improve both the manufacturing processes and the preparation procedures. The three main standards were 1) a nano Siemens star design with various star geometries, 2) a nanopillar array with various geometries and 3) nanoparticle systems with spherical particles of different diameter and nanorods.
Evaluation of the standards resulted in the development of new measurement methods and evaluation strategies for measurands like diameter, height, length, line edge roughness, line width roughness. The standards were distributed as part of a comparison to establish both the developed measurement capabilities and the comparability of the individual results. The results showed good agreement for some of the measurands like height, diameter and length for the nanoparticle systems. The equivalence of the results on the Siemens star and nanopillar standards was less mainly because of manufacturing tolerances of these highly complex designs and because of differences in the measurement and analysis methods.

The potential for use as an AFM probe radius characterisation tool was evaluated for all developed standards and compared to commercially available tip characterisers. It was concluded that the nanopillar array standard and the nanoparticle standards were the most promising reference materials for 3D nanometrology.

Improvement of the understanding of probe-sample interactions in AFM and SEM measurements
Impact of different probe-sample effects on nanoscale SPM dimensional measurements was studied. This included impact of humidity, probe-sample elastic deformation, cantilever torsion and effects of microscope operation regime. Studies on environmental conditions revealed that relative humidity and temperature do not significantly affect the results under typical AFM measurement conditions. Methods for prediction of the impact of probe-sample elastic deformation, based on analytical and simple numerical models were developed and validated by comparison to experimental results. Methodology for measurement of the cantilever torsion and separating it from tip elastic deformation was suggested and measurements of mechanical properties of specially developed high aspect ratio probes were performed. The studies revealed that the tapping mode is favourable with high aspect ratio probes. This is fortunately the most widely used approach. The uncertainty related to measurement regime settings is then below one nanometre, and if the experimental conditions are kept unchanged even smaller can be reached. The tip-sample-substrate interactions by the AFM probe and a reference plant nanostructure on mica were studied by different tips and samples to estimate the amount and uncertainty of corrections.
An improved version SEM simulation software was adapted to the experimental set-up of electron optical metrology system. SEM images for various imaging parameters were simulated and studied. LERMODEL software was further developed to generate synthesized SEM images of line/space patterns with Line Edge Roughness (LER). The simulated images with the predefined LER were used for power spectrum metrology of LER and the evaluation of methods for noise-free LER measurements.

Development of industrial solutions for hybrid metrology
Hybrid metrology methods for merging measurement results from either different tools or different channels of a single tool were developed. Fusion of datasets from multiple SPM measurements of the sample was used to reduce the scan artefacts like feedback loop defects and data anisotropy. For this data fusion, scripting available in Gwyddion open source software was used and combined with other data fusion modules (e.g. the x/y denoising). To test the reliability of this data fusion approach a set of simulated tip check samples was used together with different SPM artefacts simulation tools. Moreover, the impact of the potential probe-sample elastic deformation on such experiments was estimated; however, for most of the dimensional measurements using standard probes this effect was lower than the scan imperfections effect.

Two methods were developed for matching the measurement data of TEM and AFM in the hybrid approach for realising the bottom-up approach. One method uses alignment marks “written” near to the target feature pattern by the focus ion beam (FIB) milling. The other method uses the least square fitting of the AFM and TEM results. By applying the hybrid approach for determination of critical dimension a combined standard measurement uncertainty of the reference CD value was estimated as 0.81 nm. Optical methods including Mueller Polarimetry and scatterometry, which are fast and in-line measurement feasible but strongly model dependent, were combined with AFM, which is capable of 3D measurement with high spatial resolution and precision but slow. This hybrid metrology approach was applied for characterising reference materials, especially the Siemens star and nanopillar samples.

Impact

The project results were disseminated mainly by direct contact with industrial stakeholders. Nevertheless, ten open access peer-reviewed papers were published, and the project and its results were presented at various conferences (37 presentations in 21 conferences). Several presentations covering the developed methods, equipment as well as reference materials and artefacts were presented. Moreover, a Good Practice titled "Guide for dimensional metrology at the nanometre scale and for using the developed reference standards and methodologies" was developed and is available for download on the project website. The project results were presented also in two webinars. A software module for data fusion purposes was developed for Gwyddion, which is a free modular program for SPM data visualisation and analysis (http://gwyddion.net). This project delivered new and improved methods and reference materials for 3D measurements of nanostructures. Many of the methods and reference standards were developed in close co-operation with industry and several companies have already shown interest to exploit the project outcomes. The research outputs of the project allow traceable 3D metrology at the nanometre level with measurement uncertainties below 1 nm, which meet the requirements of industry or scientific research. By bringing traceability to the SI metre for 3D nano measurements, finally enables NMIs to offer the long-waited metrological support to industry. To assist the industrial and scientific communities, new SI traceable measurement and calibration services for CD measurements were launched.

Impact on industrial and other user communities

This project improved dimensional nanometrology at nanometre scale in order to advance the development and expansion of European high technology industries. To ensure the industrial impact, many of the methods and equipment developed in the project were characterised by measuring samples received from industrial stakeholders and the calibration standards produced in the project were designed in close cooperation with the stakeholders. The consortium 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 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. Moreover, PTB is collaborating with a world-leading equipment vendor to exploit the developed 3D nanometrology to measure three-dimensional parameters (including height, width, sidewall angle, sidewall profile etc.) of complex nanostructures. This challenging and important metrology task is required for realising e.g. the next generation lithography (NGL) technologies. VTT and PTB measured the 3D geometry of several types of optical specimens (microlens array MLA concave 6 µm lens and MLA convex 1.5 µm and 6 µm lenses) received from industrial stakeholders. This collaboration demonstrated the measurement capabilities of the developed equipment and methods for industry.

INRIM collaborated with a parent institute (CNR-IPSP Institute for Sustainable Plant Protection) for developing the preparation of samples with known plant nanostructures. DFM developed a compact scatterometry setup and delivered it to the NIL technology ApS for field measurements on nanostructured surfaces. CMI 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 during the project period indicates its great impact for public communities. NPL developed EBD AFM tips that have been trialed by a UK AFM manufacturer supplying AFM systems for the semiconductor industry. VSL has received interest from two companies regarding the developed Siemens star samples. TNO was made aware of this and contact details have been exchanged.

Impact on the metrology and scientific communities

The key impact from this project to the metrological and scientific communities is the traceability of 3D nano measurements to the SI metre, which enables NMIs to offer metrological support to industry. When the project started, the traceability chain was broken, because the uncertainty levels of traceable measurements at NMIs did not meet the requirements of the scientific community and industry.

Results from the bottom up approach and the x-ray interferometry work performed within this project contributed to the realisation of the lattice parameter of silicon as a secondary length standard in the revised *Mise en Pratique* for the metre (https://www.bipm.org/utils/en/pdf/si-mep/SI-App2-metre.pdf) and to its Guidance document *CCL-GD-MeP-1*: “Realisation of the SI metre using silicon lattice parameter and x-ray interferometry for nanometre and sub-nanometre scale applications in dimensional nanometrology” (https://www.bipm.org/utils/common/pdf/CC/CCL/CCL-GD-MeP-1.pdf).

Two universities and a research institute were actively involved in the project as colllaborators: University of

Helsinki, Technische Universität Ilmenau and Institute for Sustainable Plant Protection. University of Helsinki provided nanostar samples that the partners used for measurements with different methods. The results of these measurements are described in a co-authored peer-reviewed paper titled "Transfer standards for 3D nano-metrology" (submitted). The University of Technology Ilmenau, verified with a frequency comb and a GPS system all three frequency-stabilised lasers (axes x, y, z of the FAU nanopositioning and manomeasuring machine). Institute for Sustainable Plant Protection (IPSP) supplied 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 characteriser for metrological Atomic Force Microscopes.

Impact on relevant standards

The consortium 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 the metre on nanometre scale. The project contributed to the realisation of the lattice parameter of silicon as a secondary length standard in the revised *Mise en Pratique* for the metre. Moreover, the project results were presented in a CCL WG-N meeting.

Documentary standard ISO 11952: – “Scanning-probe microscopy – Determination of geometric quantities using SPM: Calibration of measuring systems for the dimensional calibration of SPM instruments” was revised under PTB lead. In addition, the project had contacts with an Italian standardisation body (UNI/CT 047) and the following national standardisation committees UNI, METSTA, VDI/VDE and Dutch normcommission Nanotechnologie were informed about the project results. Results

Longer-term economic, social and environmental impacts

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

The advanced nanometrology methods and reference materials developed in this project will benefit nanotechnology industries. The project established appropriate and urgently needed nanometrology capabilities and traceability for various nanoscale measurements, which provides competitive advantages for European companies and supports future innovation. Furthermore, in long term the results will enhance the growth of nanotechnology industries e.g. the semiconductor, nanomaterial, nanophotonics, microscopy, etc.

Concerning 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. The developed 3D nanometrology methods can help to 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 have not been 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

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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
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