European Partnership on Metrology
Call 2023 –Fundamental Metrology, Industry, Normative and Research Potential

Selected Research Topic number: **SRT-i18**
Version: 1.0

Title: Automatisation of calibration at nanoscale with bottom-up traceable standards

Abstract

The calibration of microscopes at the nanoscale is still only carried out for a small fraction of the installed instruments worldwide due to the high cost and lack of technical skills, leading to unreliable measurement results. The consequence of uncalibrated microscopes on product reliability and comparability of results are dramatic, affecting almost all areas of manufacturing industry. The project will provide inexpensive standards with direct traceability, such as crystalline standards, and digital assistance systems to support end-users in the calibration process. The existing obstacles for calibration will be removed and microscopes will be used as calibrated instruments.

Keywords

Calibration, atomic force microscope, traceability, digital assistant, microscope, nanotechnology

Background to the Metrological Challenges

Nanotechnology is of paramount importance to develop solutions for the grand challenges towards energy production, environment protection, and health care. Nanotechnologies are gaining increased relevance in manufacturing industry, the backbone of European industry.

In nanotechnology, key instruments are high-resolution microscopes such as atomic force microscopes (AFM) or confocal microscopes. However, without proper calibration of these instruments, the results are only qualitative. AFMs in industry and academia are not regularly calibrated due to the high cost of traceable standards but by short cutting a long calibration chain, the price will be dramatically reduced, advancing thus European industry. A way to short cut the calibration chain is to use the lattice parameter of silicon as a length reference at the nanometre scale (< 100 nm).

Technical characteristic regarding small dimensions, reduction of uncertainty and reliability have been investigated and demonstrated in the EMRP SIB61 Crystal and EMPIR 15SIB09 3DNano projects. It is now known that to transform standards made with this bottom-up approach into fit-for-purpose industrial calibration standards, new manufacturing techniques have to be developed. In the present situation only state-of-the art microscopes can directly benefit from crystalline standards. The majority of instruments require larger feature sizes (starting at approximately 20 nm), adjusted to the specific resolution of those general-purpose instruments and close to the size of the final product, the number of general-purpose instruments is by far larger than the high-end microscopes, both in industry application and in research.

Another obstacle for the calibration of microscopes (SEM, AFM etc.) is the complexity of calibration processes requiring high technical skills. Some recent results underpinned the need for a digital assistant for supporting the end users of microscopes to achieve reliable and accurate measurements. The manufacturing process of the bottom-up standards, crystalline standards, block copolymer based artifacts or DNA nanoorigami, are marking a technological disruption compared to the traditional lithography produced artifacts and standardisation documents describing their application. The European Metrology Network for Advanced Manufacturing stipulates initiatives to make full usage of these new opportunities.

In recent years several micro-oscillator based approaches emerged in the field of biophysics to detect the masses of bio-absorbates in the pico- to nanogram range. Their primary goal is to detect and characterize biological cells in their mass and growth to optimize industrial applications (e.g., yeast, cancer cells, bacteria cells). In all applications, the mass induced shift of the cantilever resonance frequency serves as the mass read-out. To convert the resonance-frequency shift of the cantilever into the sample mass, the spring constant and the position of the particle need to be known, i.e. measured beforehand or in-situ. Therefore, a well-known mass as a calibration or a reference to test the calibration would be of great value to the field. Simple, commercially available, silicon dioxide beads are insufficient. For the creation of ng mass standards, starting point could be the Avogadro constant N_A. By using Silicon crystals with a defined spacing of the atoms in the crystal lattice, the task is to count the atoms. This can be transformed into a length measurement via the Silicon lattice parameter. Options like silicon nanowires of diameters <100 nm could be interesting for the realisation.

Standardisation is crucial for companies to overcome economical risks and to stimulate invention. Any results from the research will have an effect in the revision of standards (e.g. digital assistance systems with AI algorithms) so interaction with standardisation committees like ISO/TC 201, ISO/TC 229, CEN TC 352 and VDI-GMA FA 3.4, 3.41 should be included.

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 metrology research necessary to support standardisation in automatisation of calibration at nanoscale

The specific objectives are:

1. To create fit-for-purpose standards and manufacturing technologies and bottom-up standards. To meet industry targets for crystalline standards by improving the yield and parallel processing of several chips in a single process. To adapt the process control for the production of standards to create larger features (e.g. dimensions of 20-70 nm for calibration of microscopes). To combine crystalline standards with different technologies like organic molecules (e.g. DNA, copolymers) or microprinting to tailor the features size to the user demands.
2. To create a digital assistant for data evaluation and calibration. To use crystalline standards and combination of standards developed in objective 1 as a baseline for the digital assistant. The assistant will automatically identify regions of interest using the new bottom-up standards with machine vision systems and/or AI based algorithms. It will also use AI for automated generation of digital twins used in automated manufacturing facilities.
3. To implement appropriate documentary standard. To prepare good practice guides for the application of the new build up standards in different measurement scenarios, instruments (e.g. AFM, SEM), and level of application (e.g. NMIs, companies). Update to the Mise en Pratique for the metre on the use of silicon reference standards.
4. To investigate further candidates for “Natural scales” at nanoscale. To generate strategic search for other candidate materials such as graphene, carbon nanotubes or C60 molecules, to be used as nanoscale standards. To develop techniques for the manufacturing of nanomass or nanoforce standards, to demonstrate on the laboratory scale the options of the new SI and its defining constants.
5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain (e.g. accredited laboratories, instrument manufactures), standards developing organisations (e.g. ISO, CEN, VDI-GMA), and end users (e.g. nanoscale industry such as nanomaterials and nanosensors).

These objectives will require large-scale approaches that are beyond the capabilities of single National Metrology Institutes and Designated Institutes. 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 EMRP SIB61 Crystal and EMPIR 15SIB09 3DNano project and how their proposal will build on those.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 1.9 M€ and has defined an upper limit of 2.3 M€ for this project.

EURAMET also expects the EU Contribution to the external funded beneficiaries to not exceed 35 % of the total EU Contribution across all selected projects in this TP.

Any industrial beneficiaries that will receive significant benefit from the results of the proposed project are expected to be beneficiaries without receiving funding or associated 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,
- Facilitate improved industrial capability or improved quality of life for European citizens in terms of personal health, protection of the environment and the climate, or energy security,
- Transfer knowledge to the nanomaterials and nanosensors industry.

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 the Partnership 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.