Dimensional metrology for micro- and nano-technologies

Background
European micro- and nano-technology is reaching increasing levels of miniaturisation and complexity and encountering new issues related to health, environment, production feasibility, quality and efficiency, for manufacturing and control on the µm and nm scales. Within the different technological fields which can be distinguished in nanotechnology, a set of four core application/technology fields is assessed in this roadmap: Nanoparticles, Functionalyzed surfaces, Semiconductor & nano-electronics (Beyond Moore) and Nanobiotechnology. Metrology is needed to bring these applications into the realm of predictable, controllable manufacturing, rather than experimental new technology. Although the Semiconductor Industry is more mature than the other fields in nanotechnology, the challenges here to progress with Moore’s law are still great.

Roadmap targets and associated routes
Here we can distinguish between targets where innovation is necessary or grand challenges (nanoparticles and health) exist, and targets for basic R&D capacity building which are more general for nano- and micro-technology.

Metrology of small structures (This is a basic science and capacity building target in general for the micro- and nano-technology).
3D instrumentation based on ultra precise positioning systems are a basic technology for nanometrology, necessary for sensitive investigations of probe-sample interaction as well as a base for traceable metrology.
The potential of atomic lattices as natural rulers as well as self-organization of artificial gratings and structures will be investigated for the use as intrinsic standards for dimensional measurement instruments.
The measurement of shape and size of small structures are a more complex task to be handled. It needs an improved understanding of probe-sample interaction and the combination of different measurement methods like SPM and TEM to determine the effective size of probes. A better matching of different sensor techniques will improve the reliability of the uncertainty evaluations. Metrology in UHV for investigation of influence of surface layers on probe-sample interaction. There will be an increasing overlap between dimensional- and analytical metrology using SPM due to the fact that material contrast will influence the dimensional results as well as dimensional deviations influence the results from analytical methods like MFM, KPM, ... Investigation to use analytical modes on SPM and SEM for improved dimensional accuracy (signals at curved structures, detection of material contrasts). New sensor systems like HIM have to be investigated for improved knowledge of the imaging process.

Different metrology tools are in use today and will also be used in future to control industrial production processes with nanometre stability. In order to detect the smallest changes in the produced components the response of a metrology tool has to be well understood. A sound model for the interaction of the probe and the feature of interest has to be available, to allow cross calibration analysis of results. These models have to take into account size, shape and material parameters of the features of interest as well as the complex matrix in which these features might exist at the nanoscale. Modelling is already existing but given the large variety of sensors (especially non-contact) in combination with real (non-ideal) industrial and even more complex structures this remains a challenge at the demanded levels of uncertainty.

Nanoparticles
Nanoparticles (NP) use is in rapid growth in various products (e.g. paints, lotions, protective clothes, etc). There is a public concern on possible hazardous effects of nanoparticles which has to be addressed by standardisation, control, legislation and, therefore, metrology. CEN-352 has a mandate from the EU to develop standards which can be used for regulations. Effective control of nanoparticles requires measurement, classification and safe handling/manufacturing. There are several standard instrumental techniques currently applied for nanoparticle, and nanomaterials in general, characterization, including Electron Microscopies (TEM, SEM), Zeta Potential, Dynamic Light Scattering, Mass spectroscopies, UV-VIS spectroscopies, etc. Also, high resolution microscopy techniques for individual nanoparticle characterization are usually applied (SEM, TEM, AFM, UV, optical, EELS, HAADF, EDS, etc)), along with X-Ray Diffraction, magnetic measurements (SQUID) and even high resolution spectroscopies using synchrotron light (XAFS, EXAFS, XANES, RIXS, etc). However all these should be further developed...
(e.g. smaller wavelength, fs microscopy) in order to deal with the challenges that entail the fast development of the synthetic procedures to synthesize new types of nanomaterials and the better understanding of their reactivity in different application media.

In addition, other, integral metrology techniques also exist: PCS, scattering methods, etc. However, high quality, well defined nanoparticle standards from different materials are lacking. If available, these would help to better cross-correlate the results of local and integral methods and this would allow to achieve the 1 nm accuracy target.

Basic measurements of size and size-distribution of well-defined nanoparticles have been realized, but now we are facing different challenges regarding nanomaterial characterization and standardization: i) measurement of morphology and shape evolution of individual nanoparticles and agglomerates (and/or while incorporated in larger matrices) and in general the tracking of the evolution of the nanoparticles during their full life cycle is still not solved; ii) The influence of shape and form of the nanoparticles on integral methods needs to be determined for a more reliable measurement uncertainty; iii) New generation of nanoparticles with increased complexity in terms of morphology (nanocages, nanoboxes, double-boxes, hollow multi-chamber nanoparticles, nanorattles, etc) are in rapid development and with tremendous potential on new/improved applications, which will entail another challenge for the current characterization techniques.

Nanoparticles can exist in air, liquid, powder and solid media, all having their own specific influence on the nanoparticles themselves as well as on the measurement techniques. For example, nanoparticles in air (aerosols) are a concern for health risks in i.e. production environments. Traceable, easy, robust and efficient monitoring equipment here is lacking.

Finally, although not directly a dimensional parameters, the issue of traceability for concentration of NP has not really been addressed. The measurement of shape and form of nano-objects, like crystalline nano rods, e.g. ZnO, requires much more improved measurement and sample preparation techniques than nanoparticles. It will be a challenge here to combine the use of different methods using single objects as well as ensemble techniques for the characterization, e.g. high resolution TEM and SAXS for core shell particles.

**New triggers:**
- CEN-352 has a mandate from the EU to develop standards which can be used for regulations. Underpinning metrology is lacking. E.g. Non-spherical nano particles give large variations in measured size when using different techniques. Shop floor level traceability is lacking.
- New generation of nanoparticles with increased complexity of morphology.

**New targets:**
- Shape and form measurements of nano-particles and nano-objects, like fibres, rods, wires or plates
- Traceability for the shop floor. This will mainly be measurements in air. (Aerosols)
- Multi-parameter measurements in different media (solid, air, liquid) and state (individual or agglomerate)
  - Dimensional: (size, size-distribution, shape morphology, porosity
  - Concentration
  - Mechanical, optical electrical

**Functionalized surfaces (including membranes)**
Over the last ten years, European nanotechnology has improved the possibilities in the functionalization of surfaces by nano structuring techniques and/or special single or multiple layered films (super hydrophobic, self-cleaning, scratch resistance, low/high reflectance, low friction) and membranes used for sensing (pressure), filtering (pure water) or in energy production (gas purification) to save resources due improved lifetime and/or energy saving properties. Furthermore new functionalized surfaces have been produced: haptic, fingerprint free, etc. and some investigations are made to protect surfaces against bio-fouling to save resources and energy (sewer channels, ship surfaces).

Further necessary improvements of surface quality call for an increased 3D resolution capability of instruments combined with a more multi-instrumental characterization of nano-structured surfaces consisting of various materials. This includes the characterization of porous surfaces, thin films and thin membranes including the characterization of their filtering properties to increase the efficiency.

Based on well developed established techniques to characterize homogeneous sample surfaces, i.e. geometry of 3D micro and nano structures and 2D roughness there is a strong need to accurately characterize in three dimensions micro- and nano-structured surfaces consisting of various materials. This is challenging in several aspects: There is no unique technique which allows the characterization of all the details and properties including material properties alone. Therefore one has to combine high resolution reference instruments based on tactile 2D measurements to achieve a well defined reference basis. This has to be done with well characterized tips and small forces. To get a complete view of the
surface and their function this has to be combined with microscopy techniques (optical interference, confocal, white light, or new techniques) for faster investigations of larger areas or for control of production process and stability. For high resolution investigation of special regions of interest, scanning probe instruments have to be applied, too. Here the determination of the 3D shape of tips is a challenging task. Beside the more existing qualitative characterization of samples, new techniques have to be developed to provide a true traceable characterization of tips (certified reference standards calibrated by TEM based characterized tips).

Additionally, for complex surface functions it is mandatory to combine these more or less dimensional working instruments together with spectroscopic or analytical instruments providing right information about quality and quantity of layered systems. All the measured data must be aligned to the samples surface coordinates to allow a quite well data fusion.

In the case of thin membranes reference methods need to be developed to determine their thickness accurately with very high lateral resolution and small uncertainty. Reference membranes need to be developed for traceable calibration of optical and other instruments used in industry and at universities for fast measurements.

Suitable high resolution sensors have to be developed (e.g. fast, miniaturized probes), ensemble of different sensors in one instrumentation allowing to probe a specimen in different wavelength regimes (roughness, form, etc.) or to probe dimensional & additional functionally important parameters (multi-parametric: e.g. optical (e.g. SNOM), electrical (e.g. SCM), magnetic, (e.g. MFM)).

**New triggers**
- Improvement of surface quality of functionalized surfaces

**Semicon & nanoelectronics (Beyond Moore) (Previously metrology over several 100 mm range)**

Most prominent technology field related to this target is semiconductor production, in which already today production stability has to be realized at a sub-nm level (overlay, 2D-position) or even lower for line width (as discussed above), layer thickness. Future lithography techniques like e.g. quad patterning or EUVLitho, put even higher requirements on relative positioning of components (see ITRS) and on their thermal and long-term stability, too (e.g. thermal expansion of EUV mask has to be guaranteed to be less than $5 \times 10^{-6} \, \text{K}^{-1}$).

Calibration - inspection- and manufacturing- tools require suitable stable (dilatometry) and well characterized metrology stages (possibly requiring operation in a vacuum environment); of which guiding and positioning deviations of motion axes have to be accurately known (displacement & angle metrology) and have to be mapped or cancelled (self calibration techniques).

An open point for future developments is use of e-beam instruments for stable long range registration metrology, if structures become too small for optical microscopes. The stability of metrology frames including especially the stability of the e-beam is unsolved.

Moreover the semiconductor industry is moving from 300 mm wafers to 450 mm wafers, so the ever improving demands on position and stability need to be realized over longer ranges.

The ITRS roadmap here is the guide for the metrology and standards to be developed.

Nano-electronics (organic and inorganic) is seen as the technology for new devices of the future. When size of particles and structures reach the nanoscale, often new properties are observed, which can be used for new products or optimisation of existing ones. The relation between size, shape, material characteristics and intended properties of nanostructures have to be well understood for product development and manufacturing. Accurate dimensional metrology and material characterization, both locally at the nanoscale on individual nanostructures and over the extended dimensions of the whole nanotechnology component is one target to achieve. Suitable high resolution sensors have to be developed to probe dimensional & additional functionally important parameters (multi-parametric: e.g. optical (e.g. SNOM), electrical (e.g. SCM), magnetic, (e.g. MFM)).

**New Triggers:**
- ITRS roadmap
- Nanoelectronics is the technology for device of the future. Size, shape and material characteristics at the nanoscale determine their properties
- More efficient production control for manufacturing processes of nanostructures

**New Targets:**
- Multi parametric metrology: size(3D), shape, thickness, overlay and materials characteristics at the sub-nm level of nanostructures
- 6 DOF metrology stages with sub-nm uncertainty and thermal- and long-term stability in ambient and vacuum conditions
- Reduction of measurement uncertainty for determination of the size and the shape of nanostructures
- Better understanding of probe sample interaction and therefore improved Tool matching

**Nanobiotechnology**

A new area for metrology is in the nano-bio technology, here biological cells, tissues and layers need to be measured. So far this field has mainly been one of scientific interest, but the first applications, for example diagnostic of biomarkers in health, are requiring metrology at the nanoscale. The soft and wet biological materials are a challenging task for metrology. A step change in metrology is necessary to improve the fundamental understanding of complex structures and phenomena within biological systems.

Physicochemical changes of inorganic nanoparticles in biological environments determine their effects. Blood, lymph, mucus, complete cell culture media, and other biological fluids contain a large amount and variety of different molecules. Nanoparticles dispersed in these fluids are sensitive to such environment. One of the most significant alterations is the formation of the Nanoparticle Protein Corona as a result of the absorption of proteins onto the inorganic surface. Currently, there is an increasing awareness of the importance of the Nanoparticle Protein Corona in the field of inorganic nanoparticles, which is reflected in the increasing number of recent publications that cover different aspects of it. Largely, this is because this spontaneous coating provides the biological identity to the composite Nanoparticle–Protein Corona and determines the interactions between the nanoparticles and the host in living systems. As a result, the proper knowledge of the Nanoparticle Protein Corona has emerged as a crucial aspect to understand the evolution, biodistribution and reactivity of nanoparticles inside organisms and, therefore, for the safe design of the engineered nanoparticles. And assays and techniques to integrate the characterisation of both the biological and the inorganic material without disruption one, other or their interactions needs to be validated.

**New Triggers:**
- First applications of nanobiotechnology (biomarkers, biological coatings) are emerging. Base metrology is missing.

**New Targets:**
- Development of a metrological infrastructure for biological systems.

**Cross-links with other technical areas**

**Enabling science & technology inputs include:**
- Improved nano-fabrication techniques – envisaged as either directly developed by the NMIs (although this could be costly) or available as spin-offs or shared used of facilities from industry.
- Nano-thrusters and associated metrology from the TC-Mass area.

**Outputs from TC-Length into other technical areas:**
- New capabilities for characterisation of new materials at the nanoscale, using new probing techniques, better modelled and understood probe-surface interactions.
- Link with chemistry & materials for the physico-chemical characterisation.

**References**

Other sources of information on metrology aspects in nanotechnology are available and were taken into account in setting up this roadmap:

The need for measurement and testing in nanotechnology, position paper by High Level Expert Group: [http://www.npl.co.uk/euromet/length/docs/nano-initiative/dfm-report.pdf](http://www.npl.co.uk/euromet/length/docs/nano-initiative/dfm-report.pdf)

Technology platforms on the EU Cordis site: [http://cordis.europa.eu.int/technology-platforms/individual_en.html](http://cordis.europa.eu.int/technology-platforms/individual_en.html) especially: ENIAC, Photonics21, Nanomedicine,

**New roadmaps for the 2012 version of the micro Nano Roadmaps**

ITRS 2011 (Executive summary and Chapter on Metrology)
NANOfutures, A cross-ETP Coordination Initiative on nanotechnology
http://www.nanofutures.info/sites/default/files/NANOfutures-D2.3-20-%20H13_1.pdf

Fertigungs-messtechnik 2020 VDI/VDE

European Nanometrology 2020 Co-Nanomet
http://www.euspen.eu/content/Co-nanomet%20protected%20documents/publications%20area-European%20Nanometrology%202020.pdf

Dutch HTSM (High Tech systems and Materials) roadmaps
Nanotechnology:
http://www.htsm.nl/Roadmaps/Nanotechnologie
Semiconductor:
http://www.htsm.nl/Roadmaps/Semiconductor_Equipment
Mechatronics and manufacturing
http://www.htsm.nl/Roadmaps/Mechatronics_Manufacturing

Nano-strand
http://www.ebn.din.de/sixcms_upload/media/2929/NANOSTRAND.pdf

References:


National nanotechnology initiative strategic plan

Nanotechnology Research directions for societal Needs 2020
http://www.wtec.org/nano2/docs/Nano2-Brochure-Final-04-14-11.pdf see page 10