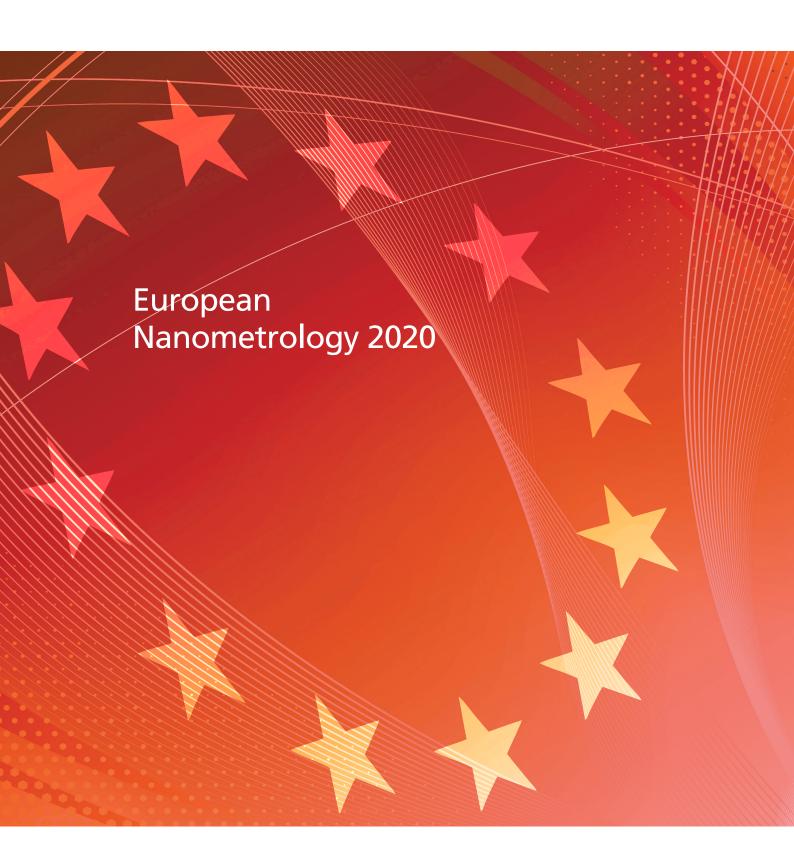
# **Co-Nanomet**

Co-ordination of Nanometrology in Europe





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# **Glossary of Acronyms**

AES	Auger Electron Spectroscopy			
AFM	Atomic Force Microscopy			
ALD	Atomic layer Deposition			
BEEM	Ballistic Electron Emission Microscopy			
BIPM	Bureau International des Poids et Mesures			
CD	Critical Dimension			
CEN	European Committee for Standardization			
CENELEC European Committee for Standardization  CENELEC European Committee for Electrotechnical Standardization				
CEOC				
CIPM	International Committee for Weights and Measures			
CMCs	Calibration Measurement Capabilities			
CMM Coordinate Measuring Machine				
CMOS	Complementary Metal-Oxide-Semiconductor			
CNT	Carbon Nanotubes			
CoD Centres of Dissemination				
CoE Centres of Excellence				
CVD Chemical Vapour Deposition				
CU Competence Units				
DFT Density Functional Theory				
DUV	Deep Ultraviolet			
EA	European Co-operation for Accreditation			
EC	European Commission			
EC	Evaluation Criteria			
ECTS	European Credit Transfer System			
EDQM	European Directorate for the Quality of Medicines			
EF	Electric Field			
EFTA	European Fair Trade Association			
EHS	Environment, Health & Safety			
EMRP	European Metrology Research Programme			
ENP	Engineered Nanoparticles			
EPMA	Electron Probe Microanalysis			
ERA	European Research Area			
ERA-NET	Networking the European Research Area			
ERM European Reference Materials initiative				
ESFRI	European Strategy Forum on Research Infrastructures			
ESRF	European Synchrotron Radiation Facility			
ETSF	European Theoretical Spectroscopy Facility			
ETSI	European Telecommunications Standards Institute			
EU-OSHA	European Agency for Health and Safety at Work			
EURAMET	European Association of National Metrology Institutes			
EUV	Extreme Ultraviolet			

FEM	Finite Element Model			
FIB	Focused Ion Beam			
FP (6,7,8) Framework Programme (6,7,8)				
, ,	Grand European Initiative on Nanoscience and Nanotechnology			
GENNESYS	using Neutron- and Synchrotron radiation sources			
GMN Gap-probed molecular nanometrology				
GMR Giant Magneto-Resistive Effect				
HOMO-LUMO	Highest Occupied Molecular Orbit - Lowest Occupied Molecular Orbit			
HPC	High Performance Computing			
IEC	International Electrotechnical Commission			
ILCs	Interlaboratory Comparisons			
iMERA-Plus	implementing metrology in the European Research Area			
IRMM	Institute for Reference Materials and Measurements			
ISO	International Organization for Standardisation			
ITNs	Initial Training Networks			
ITRS	International Technology Roadmap for Semiconductors			
JJ	Josephson Junction			
JWG	Joint Working Group			
KCDB Key Comparison Database				
KPM Kelvin Probe Microscopy				
LCA Life cycle assessment				
MBPT	Many Body Perturbation Theory			
MC	Monte Carlo			
MD	Molecular Dynamics			
MEMS	Microelectromechanical systems			
MFM	Magnetic Force Microscopy			
MLS	Metrology Light Source			
MWCNT	Multi Wall Carbon Nanotubes			
NEMS	Nanoelectromechanical systems			
NGO	Non Governmental Organisation			
NLCG	Nanotechnology Liaison Coordination Group			
NMI	National Metrology Institute			
NMR Nuclear Magnetic Resonance				
OECD Organisation for Economic Co-Operation and Development				
OEMs	Original Equipment Manufacturer			
OLED Organic Light Emitting Diode				
PC	Performance Criteria			
PP	Professional Performances			
PRINS	Pan-European Research Initiative for Nano-Structures			
PS	Professional Skills			
QA	Quality Assurance			
QHE	Quantum Hall Effect			

R&D	Research & Development			
	Registration, Evaluation, Authorisation and Restriction of			
REACH	Chemicals (REACH)			
RM Reference Material				
SAXS Small Angle X-ray Scattering				
SEA	Semiconductor Equipment Assessment Scheme			
SE Spectroscopic Ellipsometry				
SEM Scanning Electron Microscopy				
SEMPA Scanning Electron Microscopy with Polarization Analysis				
SERS Surface Enhanced Ramen Scattering				
SET Single Electron Transistor				
SFM Scanning Force Micrsocopy				
SIMS Secondary-ion Mass Spectrometry				
SMEs Small and Medium sized Enterprises				
SNAP	Strategic Nanotechnology Action Plan			
SNOM Scanning Near-field Optical Microscopy				
SPIP™	Scanning Probe Image Processor			
SPM	Scanning Probe Microscopy			
STM	Scanning Tunnelling Microscopy			
SWCNT	Single Walled Carbon Nanotube			
SWOT Strengths, Weaknesses, Opportunities, Threats				
TC Technical Committee				
TDDFT Time-Dependent Density Functional Theory				
TEM Transmission Electron Microscopy				
TM Training Module				
TOF-SIMS	Time-Of-Flight Secondary-Ion Mass Spectroscopy			
XANES	X-ray Absorption Near Edge Structure			
XAS	X-ray Absorption Spectroscopy			
XPS	X-ray Photoelectron Spectroscopy			
XRF X-ray Fluorescence				
WHO	World Health Organization			

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# **Executive Summary**

Metrology, from the Ancient Greek metron (measure) and logos (study of), is the science of measurement. Nanometrology is a subfield of metrology, concerned with the science of measurement at the nanoscale level.

Today's global economy depends on reliable measurements and tests, which are trusted and accepted internationally. As such, metrology is a natural and vital part of our everyday life: coffee and planks of wood are both bought by weight or size; water, electricity and heat are metered. The quantity of active substances in medicine and the effect of the surgeon's laser must also be precise if patients' health is not to be jeopardised.

This measurement infrastructure must be extended into the nanoscale and beyond, to bring nanotechnology based products or manufacturing processes successfully and safely into the market place. It must provide the ability to measure in three dimensions with atomic resolution over large areas. For industrial application this must also be achieved at a suitable speed/throughput.

Measurements in the nanometre range should be traceable back to internationally accepted units of measurement (e.g. of length, angle, quantity of matter, and force). This requires common, validated measurement methods, calibrated scientific instrumentation as well as qualified reference samples. In some areas, even a common vocabulary needs to be defined. A traceability chain for the required measurements in the nm range has been established in only a few special cases.

A common strategy for European nanometrology has been defined, as captured herein, such that future nanometrology development in Europe may build out from our many current strengths. In this way, European nanotechnology will be supported to reach its full and most exciting potential.

As a strategic guidance, this document contains a vision for European nanometrology 2020; future goals and research needs, building out from an

evaluation of the status of science and technology in 2010. It incorporates concepts for the acceleration of European nanometrology, in support of the effective commercial exploitation of emerging nanotechnologies.

The field of nanotechnology covers a breadth of disciplines, each of which has specific and varying metrological needs. To this end, a set of four core technology fields or *priority themes* (Engineered Nanoparticles, Nanobiotechnology, Thin Films and Structured Surfaces and Modelling & Simulation) are the focus of this review.

Each represents an area within which rapid scientific development during the last decade has seen corresponding growth in or towards commercial exploitation routes. In turn, questions of fundamental metrological understanding have emerged. The four identified thematic areas are of continuing importance for the coming decade and as key breakthrough areas with high commercial potential and societal impact.

Looking ahead to 2020, this document, sets out high level goals about each of the four priority themes. Specific objectives underpinning these goals are defined in each case plus, where relevant, underlying assumptions. This strategic analysis is captured in Vision 2020 (section 2).

Annexed to this paper is a series of more detailed reviews, supporting the Vision 2020 synopsis. These include: i) current and emerging regulatory issues (Annex B); nanometrology and standardisation (Annex C) and ii) metrology for laboratory quality assurance (Annex D). Annex E provides a detailed scientific review across eight specialist nanometrology areas (such as dimensional and chemical nanometrology). The emerging technical challenges within these areas are cross-cutting and underpinning to the four priority themes detailed above.

This document was compiled under the European Commission Framework Programme 7 project, Co-Nanomet. It has drawn together input from industry, research institutes, (national) metrology institutes, regulatory and standardisation bodies across Europe. Through the common work of the partners and all those interested parties who have contributed, it represents a significant collaborative European effort in this important field.

In the next decade, nanotechnology can be expected to approach maturity, as a major enabling technological discipline with widespread application. The principal drivers for its development are likely to shift from an overarching focus on the 'joy of discovery' towards the requirement to fulfil societal needs.

This document provides a guide to the many bodies across Europe in their activities or responsibilities in the field of nanotechnology and related measurement requirements. It will support the commercial exploitation of nanotechnology, as it transitions through this next exciting decade.

Dr Theresa Burke, Aug 2011 on behalf of the Co-Nanomet Consortium

# 1 Introduction to European Nanometrology Strategy

#### Introduction

Nanometrology is the science of measurement at the nanoscale (1 nm to 100 nm). It has a crucial role in the production of nanomaterials and the manufacturing of nanoscale devices with a high degree of accuracy and reliability.

As, worldwide, governments wish to see a return on their investment in nanoscience, the role of nanometrology comes to the fore. Anticipated advances in emerging nanotechnology industries will require revolutionary metrology with higher spatial resolution and accuracy than has previously been attained. Fundamentally new measurement techniques and standards must be developed.

The field of nanotechnology covers a breadth of disciplines each of which has specific and varying metrological needs. Moreover, the interdisciplinary nature of nanosciences and nanotechnology requires the development of new reference systems and concepts that can make the results of nanoscale measurements traceable and thereby comparable.

To this end, a set of four core technology fields (Engineered Nanoparticles, Nanobiotechnology, Thin Films & Structured Surfaces and Modelling & Simulation) have been assessed as part of the European Framework Programme 7 project Co-Nanomet.

Each of the four identified fields faces the challenge to develop or create new measurement techniques and standards to meet the needs of next-generation advanced manufacturing, which will rely on nanomaterials and nanotechnologies. The needs for measurement and characterisation of new sample structures and their behaviour far exceed the capabilities of current measurement science.

#### Framing the European Nanometrology Strategy

As strategic guidance, this document contains a vision for European nanometrology 2020; future goals and research needs, building out from an evaluation of the status

of science and technology in 2010. It incorporates concepts for an accelerated development of European nanometrology, in support of the effective commercial exploitation of emerging nanotechnologies.

#### Process for developing the Strategy

Over a two year period, the Co-Nanomet programme has carried out a comprehensive review of the current status, future challenges and opportunities.

A number of forums have been used to gather and consolidate input from key experts and interested parties including: a foresight review, the formation of five European nanometrology Action Groups including key stakeholders; targeted workshops and consultations<sup>1</sup> throughout 2009 and 2010; publication of a series of discussion papers and the launch of a nanometrology capabilities directory mapping key capacities Europe wide. Input has been drawn from industry, academia, (national) metrology institutes, standards and regulatory bodies both European and world-wide.

This work builds on previous activities, including that carried out in the European Commission specific support action, Nanostrand, which ran from August 2006 until January 2008. An output of Nanostrand was a set of roadmaps in the areas of: (i) dimensional nanometrology; (ii) nanomechanical metrology; (iii) nanochemical metrology; (iv) nanostructured materials; and (v) nanobiometrology. The roadmaps have been drawn upon in this review.

#### Background to this Strategy

Nanoscience and nanotechnology involve studying and working with matter at and around the nanoscale (1 nm to 100 nm). Astonishing progress has been made in the last decade in the field. A fundamental understanding of physical, chemical and biological processes at the nanoscale has accelerated at a pace, on the basis of new measurement capabilities. For example, femtosecond observation of nanoscale interactions (displacement of atoms) has become possible in chemical processes and single-charge, single-spin, spin excitation and bond vibrations may now be

<sup>&</sup>lt;sup>1</sup> See full publications list at <u>www.euspen.eu/nanometrology</u>

probed at the atomic scale. Meanwhile meaningful commercial exploitation has emerged: semiconductors with lateral features under 100 nm today constitute over 30 % of that market worldwide; in 2005 Abraxane® was the first US Food and Drugs Administration approved drug to use albumin nanoparticles to improve the therapeutic and safety properties of an anticancer agent $^2$ . Such progress has built on discoveries during prior decades such as  $C_{60}$ , nanotubes; and of instrument developments such as the invention of the atomic force microscope, plus developments in electron microscopy, which has enabled the imaging of surfaces and structures with sub-nanometre-level out-of-plane resolution.

Progress during the last decade has clearly established nanotechnology as a truly revolutionary technological capability, so fundamental it will impact a myriad of industrial sectors (not least chemicals, pharmaceuticals and electronics) and, no doubt, give birth to whole new areas of technology (as today's information age has grown out of the development of the semiconductor chip).

Almost sixty countries world-wide have significantly invested in nanotechnology research programmes. In the US, the cumulative investment in its National Nanotechnology Initiative to 2010 is over \$12 billion<sup>3</sup>, placing it among the largest US civilian technology investments since the Apollo Moon-landing programme. Fierce competition, therefore, exists across the globe to extract the vast economic potential of nanotechnology.

To sustain the expected considerable growth in the number of nanoproducts, major investment in technological and manufacturing/business infrastructure is needed. This includes metrology and measurement standards (documentary and material) which can be used at the nanoscale in support of conformity assessment of nanoproducts of all kinds. Quality-assured measurements in nanometrology – including metrological traceability, measurement uncertainty and standardised definitions and, if necessary, measurement methods – provides an infrastructural support throughout the innovation process – from initial idea, through design,

Website: nano.cancer.gov/action/news/nanotech\_news\_2006-01-30e.asp

<sup>&</sup>lt;sup>3</sup> Nanotechnology: Small Wonders, Nature, Sept 2010, p18

manufacture, conformity assessment and marketing – through to the finished product.

#### 2 Vision 2020

In the next decade nanotechnology can be expected to approach maturity, as a major, enabling technological discipline with widespread application. The principal drivers for its development are likely to shift from an overarching focus on the 'joy of discovery' towards the requirement to fulfil societal needs<sup>4</sup>: Addressing challenges relating to water conservation, energy management and the ageing population as well as ambitions such as those specified in Europe 2020, the EU's growth strategy for the coming decade (for a smart, sustainable and inclusive economy) is expected to come to the fore.

Key markets today are in pharmaceuticals, electronics and materials. For these, and newly emerging or assimilating ones, competitive advantage will require a rigorous understanding of the principles and methods of nanotechnology. This in turn will require significantly improved measurements with higher resolution and accuracy than has previously been envisioned. New measurement techniques and standards ((both written and material) must be developed, harmonised and characterised, combining the existing expertise of metrologists in diverse fields, to support such understanding.

As nanotechnology becomes a pervasive technology, its health and safety management throughout the lifecycle (from R&D, through manufacture to product and disposal) must become a commonplace process. So too, the reference materials, documentary standards, reference data, instruments and transferable methods and models must be made available.

Direct, non-invasive, measurement of dynamic processes will be a key step forward which will open our vision and enhance our fundamental understanding of both

<sup>&</sup>lt;sup>4</sup> Nanotechnology Research Directions 2020, NSF, WTEC report, September 2010

nanoscale systems and nanomanufacturing processes. Major challenges also lie in the preparation of test samples, with a need to preserve delicate nanoscale features.

The nanometrology community will be required to develop the empirical understanding to ensure the metrological traceability of nanoscale (and subnanoscale) measurement results across instruments, and thus their comparability across organisations, and across international boundaries.

#### Strategic priorities

The field of nanotechnology covers a breadth of disciplines, each of which has specific and varying metrological needs. To this end, a set of four core technology fields (Engineered Nanoparticles, Nanobiotechnology, Thin Films & Structured Surfaces and Modelling & Simulation) are the focus of this review.

Each represents an area within which rapid scientific development during the last decade has seen corresponding growth in or towards commercial exploitation routes. In turn, questions of fundamental metrological understanding have emerged.

The Co-Nanomet programme has assessed the current nanometrology landscape and potential within each area (see full publications list at <a href="https://www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>) through consultation with a range of key stakeholders (defined in section 4). European Nanometrology Action Groups have been formed, serving as a focal point for communication, co-operation and collaboration amongst participating parties and providing a framework for shared goals, priorities and strategies.

The four identified thematic areas have been confirmed, through the work of the Co-Nanomet programme, as being of continuing importance for the coming decade and as key breakthrough areas with high commercial potential and societal impact. Given a minimum incubation period of emerging capabilities of five years, and the envisioned transformation of nanotechnology in the next ten years, *emphasis should continue to be placed upon these areas, to build upon the momentum gained*.

With the rapid speed of development in the field, an adaptive strategy is required to respond to advances in research, new discoveries and product development. Looking ahead to 2020, this document, therefore, sets out high level goals about each of the four areas. Specific objectives underpinning these goals are defined in each case plus, where relevant, underlying assumptions.

### **Priority Theme 1: Engineered nanoparticles (ENPs)**

Nanoparticle production is in rapid growth for many different applications. Increases, in the last decade in the number of different kinds of ENPs and the broader range and understanding of their functional properties promises much for future applications. The commercial availability of off-the-shelf nanoparticles, in sizes ranging from one to one hundred nanometres, has enabled rapid advances in the fundamental science and application of particles in products ranging from medical therapeutics to hybrid electronics.

**Goal for 2020:** As nanoparticle enabled products expand further into the market place, nanoparticle EHS (Environment, Health & Safety) hazard assessment is routinely integrated into mainstream research and production activities, to support safer and more equitable progress of existing and future nanotechnology generations.

#### **Specific Objectives:**

- Development of scientifically sound classification methods to distinguish nanoparticles in terms of their basic physico-chemical characteristics and properties.
- ii) Enhanced understanding of the suitability and limitations (detection limits, calibration procedures, etc.) of measurement techniques and instrumentation (in particular for the range below 10 nm) both within the scientific and industrial community. It is particularly important to be able to select the relevant measurand and methodology for a particular industrial application, and to have a full understanding of the intercomparability of different techniques.
- iii) Beyond sizing of nanoparticles, techniques should be made available for other

- relevant measurands including i) concentration measurement, ii) shape description and iii) surface charge measurement.
- iv) Deterministic relationships between concentration metrics (number, surface, mass, etc.) and EHS related effect (dose, toxicity) of nanoparticles should be established.
- v) Instrument calibration and method validation should be routinely established for measurements in the range below 100 nm (including accuracy, uncertainty, resolution of size distributions). Where possible, easy, robust, broadly applicable methods should be made available to measure nanoparticles in complex matrices, high concentration and/or harsh environments (pH, temperature, pressure, etc.).
- vi) Improved resolution, low-noise metrology scanning probe microscopy for very small scales is required in support of nanoparticle technology development (as well as nanobiotechnology, thin films and structured surfaces). The attainment of sub-atomic resolution not only vertically (out-of-plane), but also laterally is a key objective here.
- vii) Improved resolution methods for chemical analysis of nanoparticles.

#### **Assumptions:**

Development of suitable reference measurement systems (including standard methods and reference materials) is required in support of the above objectives.

#### **Priority Theme 2: Nanobiotechnology**

This area addresses the development of nanometrology related to biomedicine, bioscience and biotechnology. This is of particular importance for the pharmaceutical industry, health care applications such as imaging technology, clinical diagnostics, and medical devices (for example, implants), as well as for food safety.

In the area of bioanalytics, medical diagnostics and biosensing there is currently an intense development of new measurement methods based on nanotechnology. Important goals of this development are increased sensitivity, selectivity, speed, cost

effectiveness and ease of use. Measurement challenges in this rapidly developing area include the in-situ measurement of dimensions of biological structures, localisation and measurement of relevant levels of biologically important substances (such as drugs, biomarkers and toxins), and the biological variability of systems. Characterising soft and wet materials at the nanoscale remains a challenging task to this community.

Goal for 2020: Achieve a further step change in measurement science to improve our fundamental understanding of complex structures and phenomena within biological systems at the nanoscale and their interface with hard matter or abiotic factors. The engineering capability achieved should enable improved quality of life within the ageing population and the development of new technologies, drugs and therapies for diagnosing, understanding and treating wide spread diseases (such as diabetes, cancers, etc.).

### **Objectives:**

- i) Nanometre resolution achieved for the in-situ measurement of the three dimensional structure of a single protein with chemical specificity.
  - ii) Detection of low concentrations of biomolecules or disease markers below  $\mu gm^{-3}$  in biological samples.
- iii) Measurement of drug molecule binding to single receptors.
- iv) Imaging of living systems with high spatial and temporal resolution, as well as three dimensional information from buried structures.
- v) Increase of metrological awareness and understanding within the bio- and medical communities. Collaboration must be enhanced between these players and the metrology community to address this. Education activities must be further established to disseminate metrology knowledge into this new sector.
- vi) Lack of traceability to SI-units should be addressed within the bio-nano field.

  Standardisation should be enabled by the development of relevant measurement methods, reference materials, and best practices.
- vii) Effective implementation of reliable, economic and easy-to-use point of care devices for diagnostics and monitoring should be progressed as a key exploitation route of emerging measurement capability in the field.

#### Priority Theme 3: Thin films, structured surfaces and critical dimensions

This theme addresses the measurement and characterisation of surfaces, layers/coatings or geometrical features decisive for nanotechnology applications that have sizes (lateral and depth) of 100 nm and less.

Inorder to achieve a desired functional property, nearly all nanotechnology applications require precise control of the dimensions. Consequently measurement techniques are necessary which allow one to measure the dimensional properties as well as the specific functional properties, turning the characterisation of nanoproducts into an interdisciplinary task, with dimensional measurements often in a leading role.

Thin films are now integral to several major technology-based industries, including: semiconductor fabrication and microelectronics, magnetic data storage, optical components and coatings and photovoltaics. As thin film technology develops – more complex layers, tighter control over parameters, an increasing diversity of applications and environments – the challenges for measurements grow.

The ability to deterministically alter the topographic structure of a surface can have a profound effect on how that surface functions. Whilst much of this work is at the research stage, the number of products that include some form of surface structure control is growing rapidly.

Scanning Probe Microscopy (SPM), being an essential measurement technique for nanotechnology, is related to all four priority themes specified in this strategic paper. However a number of specific developments in regard to SPM are specified under this priority theme.

**Goal for 2020:** Thin film technologies (and associated nanometrology) developments to have enabled further key advances to major industry sectors of societal import (including semiconductor, data storage, photovoltaics and microfluidics).

Unambiguous classification of structured surfaces and a significantly enhanced range of topographic properties made measureable through novel metrology methods. Improved understanding established between the functional behaviour of structured surfaces and its features and the related "technical" parameters describing these surfaces.

#### **Specific Objectives:**

- i) A focus should be placed on accelerating the advance of techniques where Europe currently has a lead. This includes helium ion microscopy, low and medium energy ion scattering, time-of-flight secondary ion mass spectroscopy, spectroscopic ellipsometry and microinterferometry techniques.
- ii) Instrumentation development to bridge the critical gap between 2½D to 3D scanning probe microscopy and today's industrially available coordinate measuring machines should be progressed. The achievement of subnanometre resolution scanning ranges of several 100 millimetres with speeds of 1 cm<sup>2</sup> s<sup>-1</sup> would give Europe a critical industrial advantage.
- iii) The important need for rapid non-contact measurement solutions should be addressed. Advances in sensitive optical methods such as ellipsometry and micro-interferometry have particular potential here.
- iv) Issues of physical understanding and comparability with regard to key measurement techniques (e.g. AFM and alternative optical methods, and TEM as a benchmarking technique) should be addressed and widely disseminated.
- v) Improved understanding of tip-sample interactions in SPM, including tip shape effects, to reduce measurement uncertainty and measurement artefacts.
- vi) New measurement approaches and definitions should be implemented for porous and nanoparticle thin films for the determination of parameters such as surface area, pore sizes (diameter, shape, distribution, length, etc.).
- vii) Focused efforts should be made to realise the potential of high-resolution tomography; 3D measurement parameters and extraction from data sets defined; speed and ease of use addressed.
- viii) Techniques to probe surface layers at the nanoscale (and below) in air or liquid require further development to enable their application, for example, in

biological applications where function is critically dependent on the liquid environment.

- ix) Systematic research effort is required on measurement and characterisation methods for structured surfaces addressing high aspect ratio, complex geometries and special sampling methods.
- x) Development of metrology to enable manufacturing of micro- or nanoscale structured surfaces of lateral dimensions up to several metres.

#### **Assumptions:**

Effective implementation and development of inter-technique comparisons is key in this area. Round robins should be employed:

- a) in establishing needs for standards (written or physical); and
- b) in disseminating standards to industry and embedding best practice.

Development of nanometric physical measurement standards – across a range of selected materials is also required.

#### **Priority Theme 4: Modelling and Simulation**

In the field of nanometrology, computer models assist in designing new modes of measurements by giving an insight into background physical processes. Modellers may provide missing understanding of the physical properties for simulation technologies. On the other side, nanometrology contributes to effective modelling through the provision of precise input data. Modelling and simulations for nanometrology provide aid to:

- a) fabricate and measure nanoscale devices for which numerical simulations can optimise the output, either by shortcutting device design or analysing the results of measurement;
- b) predictively calculate the physical properties of nano-objects (clusters, polymers) for a bottom up design of nano-objects;
- c) calculate the range in which a specific physical effect occurs, for example clusters of silicon atoms might become metallic in a small range. Then

metrology is necessary to measure and control such a small range; and

d) simulate measurement tasks obtained by a device under variation of internal (probe, electronic) or external (temperature, vibration, etc.) parameters to estimate measurement uncertainties.

Modelling and simulation for nanometrology is a novel field, which has recently attracted significant attention. Multiscale techniques have been developed that integrate molecular simulations with modelling techniques that can be used on the scale of micrometres and seconds. Such integrated simulations offer new possibilities to interpret experimental data and to predict the properties of new materials. The tools of theory have advanced as much as the experimental tools in nanoscience over the past decade. It has been a true revolution fuelled by increased computer power.

**Goal for 2020:** Modelling and simulation community to provide the ability to realistically predict the behaviour of objects at the nanoscale for:

- 1. fabrication and integration processes;
- three dimensional nanoscale metrology;
- 3. integration of multi-scale functional systems; and
- 4. measurement approaches for off-line and in-process production applications.

## **Specific Objectives:**

- of manufacturing processes solidification, 3D injection with or without reinforcement, fibres, anisotropy and systems (virtual and accurate products). These include combinatorial chemistry and genetic techniques that open the door to the synthesis of new biomolecular materials and the creation of nano-interfaces and nano-interconnects between hard and soft matter.
- ii) Application of new mathematic tools to bridge electronic (quantum-mechanics) through macroscopic (continuum-mechanics) length and time scales.
- iii) Devise theoretical and simulation approaches to study nano-interfaces,

- which dominate nanoscale systems and are necessarily highly complex and heterogeneous.
- iv) Establish capability to simulate, with reasonable accuracy, the optical properties of nanoscale structures and to model nanoscale opto-electronic devices.
- v) Verification of complex nanostructure simulations involving "soft" biologically or organically based structures and "hard" inorganic ones as well as nanointerfaces between hard and soft matter.
- vi) Development of enhanced self-assembly and directed assembly simulations.
- vii) Theoretical and simulation approaches devised for quantum coherence, decoherence and spintronics.
- viii) Development of self-validating and benchmarking methods.
- ix) In the area of scanning force microscopy, theoretical models are necessary to correct for probe tip/sample interaction effects in the sub-nanometre range.
- x) Develop methodologies for extracting quantitative 3D information from 2D images (preferably compatible with rapid imaging). This objective supports the 2020 goals for nanoparticles, nanobiotechnology, as well as thin films and structured surfaces as set out above.

In summary: there must be robust tools for quantitative understanding of structure and dynamics at the nanoscale and a strong link to real world and measurement values.

#### **Assumptions:**

As simulations become increasingly relevant for the planning and interpretation of experiments, so do closer links between the computer-simulation community and experimentalists working at the nanoscale level.

#### Pan-Priority Requirements:

A number of assumptions are made in regard to the coming decade for nanometrology development, which impact on each of the four strategic priority areas defined above. These assumptions are as follows:

A strategic pan-European plan and infrastructure for advanced instrumentation is required which supports the metrologically sound deployment of new nanomanufacturing capability (including but not limited to electron microscopes, scanning probe systems, and advanced optical systems)

Breakthrough initiatives towards measurements with high resolution in three dimensions should be supported; such metrology having the potential to prove transformational in each of the priority areas above.

It is important that future inter-laboratory comparisons (in the form of key comparisons and supplementary comparisons) and calibration measurement capabilities (CMCs) are registered in the Key Comparison Data Base (KCDB) of the Bureau International des Poids et Mesures (BIPM), accessible at kcdb.bipm.org. To make nanometrology more visible in the KCDB, Co-Nanomet has suggested to the BIPM to 'flag' KCDB entries with 'nano' when relevant.<sup>5</sup>

Nanoscience is a global phenomenon. In 1999, nanometrology initiated its first global inter-laboratory comparison on 1D gratings with eleven participants and, at this point in time, four supplementary CIPM comparisons have been approved and published in the KCDB Appendix B. It is important that European research continues on the dual agenda of working in global networks whilst maintaining a clear focus on specific European needs. On-going work in the field of nanoparticles is a good example of the benefit of such an approach (see Co-Nanomet report Nanometrology Discussion Papers 2011 published at www.euspen.eu/nanometrology for further details).

Whilst strengthening traceability within the National Metrology Institutes is required, it

<sup>&</sup>lt;sup>5</sup> Steele A G, Viallon J, Hatto P, Janssen J T, Knight A, Locascio L, Miles J R, Morazzani V, Prins S, Unger W (2010) Report on the BIPM Workshop on Metrology at the Nanoscale, 18-19 February, Rapport BIPM-2010/06, 22 pp

is equally important to transfer knowledge to the calibration and testing laboratories. The majority of these will be accredited laboratories operating under the Calibration Multilateral Agreement (MLA) of EA (European co-operation for Accreditation). The task of writing and disseminating the necessary calibration guides on nano-calibration at the secondary level is an important one. EURAMET could be considered the appropriate body to oversee this activity.

The collaboration between metrology and research, development, and innovation must continue to be strong as the novel field of nanotechnology matures. Metrology is closely linked to the concepts of quality control or conformity assessment in product manufacturing. Programmes aimed at encouraging small to medium sized enterprises to exploit the opportunities offered by for example new micro- and nanotechnologies, can usefully also cover nanometrology.

As industry and society place increasingly exacting demands on measurement in terms of complexity and accuracy, there is a need for more pre-normative R&D into the metrological aspects of measurement and product norms for nanotechnology.

The necessary tools for implementing legislation in Europe, in the form of (mandated) documentary standards, must be based on sound measurement concepts and methods. At present, legislation is seriously impeded by the lack of standardised terminology; standardisation is, however, progressing, and in ten years time one should see the usual relationship between standardisation and metrology, where standardisation ensures what one is manufacturing and discussing, and metrology ensures how one characterises or talks about nanotechnology issues.

Important standardisation effort has recently begun to give valuable results (see Co-Nanomet document "European Consultation on Metrological Traceability, Standards and Dissemination of Metrology in Industrial Nanotechnology" for further details). Unfortunately, documentary standards sometimes make reference to unrealistic and ambiguous measurement specifications and unclear requirements. It is suggested

<sup>&</sup>lt;sup>6</sup> L R Pendrill, O Flys, K Dirscherl and Gert Roebben 2010 "European Consultation on Metrological Traceability, Standards and Dissemination of Metrology in Industrial Nanotechnology", *CO-NANOMET* report, ISBN: 978-0-9566809-8-3 published at <a href="https://www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>

that the metrological aspects of written standards are assessed and written standards describing new measurement procedures validated by an inter-laboratory comparison before their release.

Dissemination of European capability to potential users, is important to future exploitation and may be enabled through further development of the European Capabilities Directory developed under the Co-Nanomet programme.

Public opinion on nanotechnology is already strong, both because of the eye-catching and fascinating novel features of the nano-world and fear of the dangers to public health.<sup>7</sup> A mature and convincing nanometrology infrastructure will help to give a scientifically sound picture of both the benefits and dangers of nanotechnology.

#### Instruments for implementation of priorities

Multiple mechanisms may be used to build consensus, implement priority activities and drive a common agenda at a European level. These include:

- i) The European Metrology Research Programme (EMRP) may be used to drive issues of an underpinning or empirical nature requiring collaborative work between multiple (national) metrology institutes and their partners.
- ii) Short term priorities, of a more applied or industrial nature, may be addressed through Framework Programme 7 (FP7, 2007 to 2013); medium term through Programme 8 (FP8, 2014 to 2020). Consideration should be taken to enable smaller scale projects (of limited partners or budgets e.g. <€5m) which may be more attractive for engaging smaller companies, who constitute a significant body of the industrial nanometrology community.
- iii) Long term priorities, whilst highlighted in the technical annexes, are by their nature somewhat speculative. As these clarify, the latter part of FP8 may be used as a tool to address these.

<sup>&</sup>lt;sup>7</sup> Report on the European Commission's Public Online Consultation TOWARDS A STRATEGIC NANOTECHNOLOGY ACTION PLAN (SNAP) 2010-2015; website: ec.europa.eu/research/consultations/snap/report\_en.pdf

- iv) Marie Curie Training Networks (ITNs<sup>8</sup>), the European Research Council grants and the EMRP Researcher Grant Programme may contribute to the transfer of knowledge through the promotion of multidisciplinary research.
- v) The remit to ensure that the European Research Area is populated by a wide range of significant, world class, top quality instruments open centres of excellence to inspire researchers in the major discipline of nanometrology is that of the European Strategy Forum on Research Infrastructures. In them, training of young researchers and technicians may occur in a competitive environment; companies can also be incubated at the source of new technologies. To maximise impact, nanometrology infrastructures may be linked to related nanotechnology infrastructure developments. For example, the Pan-European Research Infrastructure for Nano-Structures (PRINS) will be a distributed facility of leading centres, smaller centres and research groups from all countries that forms a large pan-European infrastructure dealing with the ultimate silicon and heterogeneous integration to support the evolution from micro to nanoelectronics.

# 3 Technical Background

A further detailed analysis of the current state of the thematic areas addressed above and their current needs is given in Co-Nanomet publication Nanometrology Discussion Papers 2011 (<a href="www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>).

In ANNEX E – Technical Challenges and Opportunities, a detailed scientific review is given across the specialist areas:

- Dimensional metrology
- Chemical nanometrology
- Thin film nanometrology
- Mechanical nanometrology
- Metrology of nanostructured materials
- Electrical nanometrology
- Biological nanometrology

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<sup>&</sup>lt;sup>8</sup> Website: cordis.europa.eu/fp7/people/initial-training en.html

#### Modelling and simulations for nanometrology

The emerging technical challenges of these areas are contained within the four priority themes detailed in Section 2: Vision 2020 (Engineered Nanoparticles, Nanobiotechnology, Thin Films and Structured Surfaces, Modelling and Simulation).

In Annex E, each area is assessed against: i) a vision for the next ten years, ii) an action plan, iii) infrastructure needs iv) R&D strategies v) emerging topics and priorities, and finally vi) a SWOT analysis. These sections provide a jump off point for those wishing to address the goals for 2020 and supporting objectives set out for the four priority themes.

Drawing from the scientific review of ANNEX E – Technical Challenges and Opportunities, Table 1, provides a further summary of the technical needs of nanometrology and key priorities mapped to the measurement objects: nanostructured surfaces, nanoscale coatings and nano-objects. For each object, five measurement aspects (dimensional, mechanical, chemical, electrical and biological) are specified. Each field lists the most urgent topics for metrological development, which may be mapped to the priority themes above. The relative importance and urgency between fields is indicated with a colour code – on a scale from red (most urgent) to white (less urgent) as: red > orange > yellow > white.

Table 1: Summary of needs for action in different technical areas (Colour code: red=most urgent > orange > yellow > white =less urgent)

	Nanostructured surfaces	Nano-scale coatings	Nano-objects
Application sectors	Precision engineering	Micro-electronics / semiconductor	Materials manufacturing
Dimensional - structural	- Ensure consistency of different physical surface topography methods to close the gap between micro- and nanoscale measurement methods Study probe-sample interactions to reduce measurement uncertainty Extend nanoscale measuring capabilities to cover larger dimensional ranges Establish improved probe characterisation methods Develop methods to measure	<ul> <li>Improve comparability of different film thickness measurement methods.</li> <li>Accelerate development and application of technique advances for multilayer measurements and non-destructive methods with potential for rapid large-area sampling.</li> <li>Advance the methods to characterise porous thin films.</li> <li>Develop methods to measure the thickness of coatings on curved surfaces as used in biomedical implants.</li> </ul>	<ul> <li>Study effect of different equivalent diameters when making and reporting particle size and morphology measurements.</li> <li>Study effect of particle size distribution on sampling efficiency and quantity conversion.</li> <li>Develop certified reference materials in the sub-50 nm region.</li> <li>Develop methods to assess dispersion of nano-objects in a matrix.</li> <li>Improve 3D-EM calibration standards and stereo-grammetric measurements and analysis methods.</li> <li>Study probe-sample interactions in SPM techniques for nano-objects.</li> <li>Improve 3D-AFM/SPM and related scanning strategies.</li> <li>Develop methods to determine in situ the size distribution of nanoparticle production batches.</li> </ul>
Mechanical	roughness of curved surfaces.  - Develop AFM methods to map the mechanical response of surfaces over larger areas.	Develop tribology methods to study wear and contact reliability at the nanoscale.     Improve nanoindentation methods to measure stiffness of constituent phases of nanostructured coatings.     Develop methods to measure residual stress and elastic properties of thin films.	Develop methods to measure the strength of nanotubes, aggregates and agglomerates.     Develop methods to assess the potential release of nano-objects from matrices in which they are embedded.
Chemical	analysis Develop methods to collect molecular	al resolution of methods in chemical surface, bonding, phase and microstructural and on crystalline and amorphous surfaces,	<ul> <li>Develop in situ and dynamic methods to measure low concentrations of nano-objects in polydisperse powders, in solids (nanocomposites), and in liquid and gaseous media (fluid nanodispersions).</li> <li>Develop methods to locally assess chemical composition of nanostructured objects (for example, coated nanoparticles and capsules).</li> </ul>
Electrical	Improve dimensional and thin film nan analysis to enable nanofabrication of se devices.  (See also ITRS Roadmap 2010 Update)	emiconductor and quantum electronic	<ul> <li>Develop methods to measure conductivity of CNT (-composites).</li> <li>Develop methods to assess electrical charge of particles in aerosols.</li> <li>Investigate possibilities to use graphene for quantum electronic devices that measure physical properties at atomic scale (electronic states, contact resistance, influence of single, line or edge defects,)</li> </ul>
Biological	Develop methods to analyse molecules on a structured or particle surface, also in biologically-relevant matrices.	- Standardise methods for measuring biological activity of coatings.	<ul> <li>Develop/improve methods to characterise nano-objects in biologically relevant matrices, and to quantify their concentration in these matrices.</li> <li>Develop methods to assess toxicological effects of high concentration short term and low concentration long term exposure to nano-objects.</li> </ul>

#### 4 The stakeholders

Given the breadth of applications and implications of nanotechnology, the nanometrology stakeholder base is likewise very broad. It is convenient to separate stakeholders into two groups: public and private stakeholders. When addressing the thematic priorities set out in Section 2 - Vision 2020, the needs of each group must be taken into account.

#### Public stakeholders

National metrology institutes (NMIs) - NMIs have the obligation to develop and maintain measurement standards, to provide worldwide traceable calibrations and to disseminate measurement related knowledge.

European Association of National Metrology Institutes (EURAMET) – EURAMET coordinates, to an extent, the work of the NMIs and acts as a focal point for metrology in Europe.

Research institutions and universities - these conduct research and teach students, and are often the starting points for work at NMIs. They frequently collaborate with NMIs and possess unique measurement instrumentation and expertise.

Regulatory bodies (National and European) – there is no, or very little, specific regulation for nanomaterials and nanotechnology. Regulatory bodies are expected to develop new regulation, or revise existing regulation, alongside developments in nanotechnology and nanometrology<sup>9</sup>.

Calibration and testing laboratories – in the long term, nanotechnology and fabrication must be supported by routine and accredited calibrations and test laboratories<sup>9</sup>. It is important that such laboratories are engaged in nanometrology from an early stage in order to be prepared for this novel task.

<sup>&</sup>lt;sup>9</sup> L R Pendrill, O Flys, K Dirscherl and Gert Roebben 2010 "European Consultation on Metrological Traceability, Standards and Dissemination of Metrology in Industrial Nanotechnology", *CO-NANOMET* report, ISBN: 978-0-9566809-8-3 published at <a href="https://www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>

Standardisation bodies - work is being undertaken in national standardisation organisations, in the European Committee for Standardization (CEN), and in the International Organization for Standardization (ISO), often in parallel. Close connections between standardisation bodies and the metrology community must be maintained, so that the new nanotechnology standards are compatible with the metrological requirements <sup>9</sup>.

Accreditation and certification bodies – accreditation and certification bodies need to indicate which quality assurance tools are needed to assess the performance of calibration and testing laboratories.

#### **End-user stakeholders**

Innovative industries - In order to profit from the strong European research position in nanosciences, industry must be in close contact with new developments in metrology so that development leads to product types that can be characterised by traceable measurement results. Production industries need access to efficient, affordable calibration and testing.

Calibration and testing laboratories – in the long term, nanotechnology and fabrication must be supported by routine and accredited calibration and testing laboratories<sup>9</sup>. It is important that such laboratories are engaged in nanometrology from an early stage in order to be prepared for this novel task.

Non-Governmental Organisations (NGOs) – many such organisations monitor developments within the nanotechnology field, and can have a significant impact on the perception held by the general public. Metrology is essential in providing robust data in regard to developing nanotechnologies and enabling potential hazards to be made specific. Metrology ensures that risks can be estimated in concrete cases and not generalised causing unjustified public fear.

The general public – several potentially beneficial technologies have been prevented from prospering because of (justified or un-justified) public fear. Factual evidence,

based on sound measurements, will promote balance in the necessary public debate about nanotechnology.

#### 5 Infrastructure Needs

In the implementation of the priority themes set out in Vision 2020 (Section 2) a number of dedicated infrastructures may be considered. Such infrastructures would work to consolidate effort at a Pan European level and enhance pathways to commercialisation of nanotechnologies. They may be applied as appropriate to the thematic area to be addressed. In some instances, a single centre may combine two or more roles of (i) measurement instrumentation hub; (ii) centre of excellence in measurement; and (iii) centre of metrology dissemination

#### Coordinated measurement instrumentation hubs

Because of the cost of the necessary instrumentation, and in order to ensure efficient use of it, instrument hubs may be considered in specific technology areas or, for example, for top class metrology tools of a value above €10m. Where possible these hubs should be positioned around either national metrology institutes or large scale nanotechnology infrastructures, with the provision that they be made available for wide industrial access.

#### Centres of measurement excellence

European centres of measurement excellence may be established in order to ensure that Europe maintains its strong research position in key areas of nanotechnology and associated metrology. The centres of excellence should be established as international, interdisciplinary centres drawing from the whole stakeholder base including industry, metrology institutes plus key research organisations. In this way nanometrology may be better positioned as an enabling part of the overall effort in contributing to major nanotechnology advances and exploitation. Such centres may provide opportunities and support for multidisciplinary research among investigators from a variety of disciplines and from different research sectors, including academia, industry and government laboratories.

The fostering of multidisciplinary research not only supports advances in knowledge, but also promotes the formation of relationships that enhance the transition of research results to a broader application base.

#### Centres of metrology dissemination

Centres of metrology dissemination may be established to ensure adequate levels of nanometrology knowledge and access to expertise for all key stakeholders across Europe. Such centres could be effectively linked with the high level priority themes defined in Vision 2020. They may foster the transfer of new technologies into products for commercial and public benefit under conditions of more rigorous understanding and controls. Hence they will also support the responsible development of nanotechnology in these fields. Linkages and input would be required from all key stakeholders including industry, NMIs, research organisations, standards and regulatory organisations and the Commission. They would provide a focal point for multiple agencies, including funding bodies, to support their planning and decision making processes for priority actions.

European industry has a further need for adequate access to calibration and test facilities and services. These centres could be established collaboratively between key partners, for example, EURAMET and the European Federation of National Associations of Measurement, Testing and Analytical Laboratories (EUROLAB) as well as relevant industry linking bodies<sup>ii</sup>.

#### 6 Summary

Through the Co-Nanomet programme, a two year review of nanometrology status, opportunities and challenges across Europe has been carried out. The current technical status of the field and the extraordinary progress made in the last decades holds much promise for the future.

During the programme, the European nanometrology community has embraced the opportunity to work together on defining priorities and we are most grateful to all interested parties who have contributed to this review either directly or through associated work within the programme<sup>iii</sup>.

Following a review of the field, captured in multiple publications (available at <a href="https://www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>) and the technical annexes herein, a set of goals and

objectives have been established for European Nanometrology for the next decade (Vision 2020). These goals are set around the four priority themes of:

- Nanoparticles
- Nanobiotechnology
- Thin Films and Structured Surfaces
- Modelling and Simulation

The overarching aim of the priorities set out is:

- a) To foster the continued growth of world class nanometrology research and development across Europe.
- b) To advance the transfer of nanotechnologies into new products for public and commercial benefit under conditions of more rigorous understanding and controls.
- c) To develop the supporting infrastructure, tools, embedded skills and expertise to support European nanometrology and hence nanotechnology exploitation.
- d) To support the responsible development of nanotechnology.

We hope this document may act as a guide to the many bodies across Europe in their activities or responsibilities in the field of nanotechnology and related measurement requirements, as it moves into its next exciting decade

# **ANNEX A - The policy-relevance of nanometrology**

# A.1 Policy areas where nanometrology can be employed

Major areas of policy in the European Union (and elsewhere), such as those identified in the recent Europe 2020 paper,<sup>10</sup> will need the development of nanometrology, since nanotechnology can usefully be employed to meet these 'Grand Challenges'. Flagship initiatives include:

 'Innovation Union' to improve framework conditions and access to finance for research and innovation, in order to ensure that innovative ideas can be turned into products and services that create growth and jobs.

The Co-Nanomet project is formulating (in this document) a strategy for future research and innovation in nanometrology, in particular efficient dissemination to the workplace. Synergy with major metrology initiatives, such as the EMRP, is to be sought, for example:

 'Resource efficient Europe' to help decouple economic growth from the use of resources, support the shift towards a low carbon economy, increase the use of renewable energy sources, modernise our transport sector and promote energy efficiency.

There are many proposals to improve energy and transport efficiency and reduce environmental emissions through the deployment of nanotechnology. Examples include ultra-light but strong nanomaterials, the new properties of which need to be carefully measured to serve as inputs to product development:

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<sup>&</sup>lt;sup>10</sup> Europe 2020: A strategy for smart, sustainable and inclusive growth, European Commission ec.europa.eu/eu2020/index\_en.htm

 'An industrial policy for the globalisation era' to improve the business environment, notably for SMEs, and to support the development of a strong and sustainable industrial base able to compete globally.

Whether traditional industries are being transformed from resource-intensive to knowledge-intensive or new, high-technological industries are initiated, nanotechnology is one emerging technology to consider.

 'European platform against poverty' to ensure social and territorial cohesion such that the benefits of growth and jobs are widely shared and people experiencing poverty and social exclusion are enabled to live in dignity and take an active part in society.

Nanotechnology has the potential to significantly improve the life of the world's poorest people: it can clean water, make transport cheaper, help in producing more food and provide novel, clean and cheap energy sources. However, to make this happen is a question of policy. Deliberate and early action can be taken by governments and non-governmental organisations (NGOs) to increase the chances that the benefits of nanotechnology will be widespread.

Whatever the challenge or sector, in order to sustain the predicted radical growth in considerable investment in nanoproducts, а technological and manufacturing/business infrastructure is needed. The global race to invest in nanotechnology R&D is already creating many new products and processes, and both the research effort and the predicted market activity is conservatively forecast to be hundreds of millions of Euros. This growing trade will generate the need for new standards and new quality-assured measurement resources and capabilities. While 'risk' is a prime mover in some areas of nanotechnology, many nanoproducts that are not dangerous rely on nanometrology to ensure general product quality and conformity compliance to facilitate trade and promote customer satisfaction and efficient production.

What are the challenges to be met?

The challenge is to develop the appropriate metrology tools (new and improved measurement instrumentation with nanoscale resolution, validated methods described in documentary standards to make proper use of the new instruments, and reference systems and materials for the assessment of methods and laboratory performance). Nanotechnology presents a unique opportunity for proactive development of these tools since the technology is still in its infancy.

# A.2 From policy to measurement laboratory

This strategy paper and annexes will address: (1) the regulations embodying policy requirements; (2) the documentary standards where (essential) requirements for regulation of product are specified; and (3) measurement quality assurance in the laboratory and the workplace, for testing of products with respect to specifications in standards set by regulation from policy.

A major role would be to provide policy support in the context of conformity assessment, particularly in a proactive way, drawing attention of policy makers to upcoming issues and becoming more involved in the early, agenda-setting part of policy cycle. This includes identifying future problems, opportunities and needs of society, including technology, watching activities that pick up signals from the scientific community and using horizon-scanning procedures based on the current state of knowledge from science and technology.

# ANNEX B - Current and emerging regulatory issues

#### **B.1** Overview

Over the past fifteen years, large resources have been invested into the emerging area of nanoscience and nanotechnology. However, there remain open questions about the unknown impacts of nanomaterials on health, safety and the environment (HSE). Due to the new properties of nanoscale materials, there are potential risks associated with certain examples. Indeed, risk is a central factor to be considered in the early stage of any emerging technology; if risks are considered in time, the expost costs of identifying important impacts on HSE and potential liability costs in case of damages can be minimised.

Regulatory issues of utmost importance are:

- occupational health and safety (laboratory and large scale manufacturing);
- product and consumer safety, (Life Cycle Assessment (LCA), waste, emission);
- environmental safety (LCA, food-chain, waste, emissions);
- guidelines for the safe and sustainable use and handling of nanomaterials;
- technical measures for the protection of health and the environment (for example filters);
- low-pressure fume hoods, etc.;
- labelling of potentially hazardous materials;
- inventories of potentially critical nanoparticles and nano-applications;
- inventories of exposure and quantities of nanoparticles;
- threshold values for potentially hazardous nanoparticles;
- LCA-studies of nanomaterials, for example nanoparticles; and
- 'Code of Conduct' regarding further product aspects.

Appropriate risk data, tailored regulations, transparent communication and open public dialogue are mandatory pre-requisites to build public trust and create conditions for the successful development and application of nanotechnology. Thus, with expected risks becoming explicit and the number of nano-enabled products on the market increasing quickly, the complexities of regulating nanotechnologies need to be considered<sup>11</sup>. There is a general opinion that a clear regulatory framework needs to be set up both to enable and constrain developments in nanotechnology, in order to develop technologies that are beneficial to the public.

An example of regulatory work in nanotechnology is that undertaken in the Organisation for Economic Co-operation and Development (OECD) Environment Directorate. A recent document<sup>12</sup> provides a global perspective of current activities regionally and nationally on the safety of manufactured nanomaterials. This document mentions, for instance, connections with standardisation and metrology organisations.

The role of nanometrology in the regulatory field, in order to handle potential risks with nanosystems as well as stimulate innovation, even if the nanoproducts are not dangerous, is mainly to provide an objective ground on which decisions of conformity to specifications can be reliably taken.

Since 2004, the EU has considered the regulatory aspects of nanotechnology. The assumption was that current legislation would suffice to cover nanotechnology. Recently, some fields have started to adapt, in particular with regard to chemicals, cosmetics and food<sup>13</sup>. Indeed, The European Parliament has stated that nanomaterials must be covered by a different normative because of the almost unlimited range of applications for nanotechnologies in various sectors with their own specific features.

13 epub.oeaw.ac.at/ita/nanotrust-dossiers/dossier017en.pdf

<sup>&</sup>lt;sup>11</sup> Communication from the Commission to The European Parliament, The Council and The European Economic and Social Committee - Regulatory Aspects of Nanomaterials [SEC(2008) 2036]; <a href="mailto:europa.eu/LexUriServ/LexUriServ.do?uri=COM:2008:0366:FIN:en:PDF">europa.eu/LexUriServ.do?uri=COM:2008:0366:FIN:en:PDF</a>

<sup>&</sup>lt;sup>12</sup> OECD 2010 'Current development/activities on the safety of manufactured nanomaterials', ENV/JM/MONO(2010)4, 24 Feb 2010 Joint meeting of the Chemicals Committee and the Working Party on Chemicals, Pesticides and biotechnology, Environment Directorate

European standardisation continues to support European policies and legislation by providing adequate tools to meet identified needs, reinforcing its cooperation with the European Commission (EC) and the European Fair Trade Association (EFTA) and maintaining its recognition as the relevant platform for the development of widely-accepted-and-implemented, consensus-based standards. The New Approach and the New Legislative Framework are a future-oriented way to use dedicated expertise to meet industrial and societal needs.

The EC tabled a proposal for a recommendation on defining the term "nanomaterial" on 21 October 2010, which was open for public consultation until November 2010. The draft proposal follows a 2009 European Parliament resolution that called for the introduction of a comprehensive science-based definition of nanomaterials in Community legislation, to allow for nano-specific amendments to relevant rules and regulations.

According to the draft recommendation, a nanomaterial either "consists of particles, with one or more external dimensions in the size range 1 nm to 100 nm for more than 1 % of their number size distribution; and/or has internal or surface structures in one or more dimensions in the size range 1 nm to 100 nm, and/or; has a specific surface area by volume greater than 60 m²/cm³, excluding materials consisting of particles with an area smaller than 1 nm². It must be noted that this draft definition combines four metrological challenges:

- 1) the measurement of the size of nanoscale particles;
- 2) the measurement of the fraction of nanoscale particles in a matrix consisting of larger particles;
- 3) the measurement of the size of nanoscale structural and surface features;
- 4) the measurement of the surface area of a collection of nanoparticulate matter.

The Committee of Occupational Health and Safety (CEOC - Confédération Européenne des Organismes de Contrôle) has created a special task force on nanomaterials and is closely following up the developments in this field. Several

meetings have already taken place on the issues, including guest speakers from EU-OSHA and regular cooperation with industry associations.

The EU executive wants to hold another public consultation on the subject by 2012. The definition may also be reviewed based on experience gained, new scientific knowledge or technological development.

## **B.2** Defining the measurand and quality characteristic

A first step towards regulation is to describe clearly and unambiguously the object to be measured in a terminology which is consistent and understood by all. The 'quality characteristic', i.e. the property of the nanoproduct that is regulated, needs to be defined.

# B.3 Measurement methods in regulatory documents

Another example of how metrology enters into regulatory issues can be cited from the Nanotech Regulatory Document Archive<sup>14</sup>. A report: 'Prioritization of Environmental, Health, and Safety Research Needs for Engineered Nanoscale Materials', contains the following in its abstract:

'High priority research needs include developing methods to detect nanomaterials in biological matrices, the environment, and the workplace; developing methods to quantify and characterize exposure to nanomaterials; understand the effects of nanomaterials in individuals of a species; and

cale\_Materials\_527\_8119.pdf

<sup>&</sup>lt;sup>14</sup> Nanotech Regulatory Document Archive, [nanotech.law.asu.edu/] is a free resource built and maintained by the Centre for the Study of Law, Science, & Technology at the Sandra Day O'Connor College of Law.

<sup>&</sup>lt;sup>15</sup> National Science and Technology Council, Report 527, Prioritization of Environmental, Health and Safety Research Needs for Engineered Nanoscale Materials, [nanotech.law.asu.edu/Documents/2010/08/Prioritization\_EHS\_Research\_Needs\_Engineered\_Nanoscale\_N

identify population groups exposed to nanomaterials.'

Thus nanometrology is essential in providing measurement methods with which nanomaterials can be detected, quantified and characterised. That in turn allows and enables studies of exposure to such materials.

The behaviour of nanomaterials may be strongly influenced by the close contact with constituents of a surrounding matrix and their apparent properties may be matrix dependent. Such composite and electronics products, as well as other materials such as nanoporous materials, present challenges in developing nanometrological methods.

# B.4 Health, safety and environmental impact

Due to the relatively different physical and chemical properties of nanomaterials, the recent innovation of synthetic nanoparticles and the mobility of aerosol and unbound nanoparticles, free nanoparticles could potentially cause negative health and environmental effects. Regulation of nanoparticles will require the development of appropriate and metrologically robust methods to assess the distribution and behaviour of nanoparticles.

Regarding occupational health and safety, the lack of appropriate measurement and monitoring tools, detailed information on hazards and exposure levels and use of nanomaterials are evident challenges.

# B.5 Vision for the next ten years

Over the next ten years it will be necessary to improve the knowledge base, via research, scientific committees, information sharing and cooperation at the international level, in order to develop the necessary regulation for the diffusion of

nanotechnology and nanomaterials in the markets. For this purpose, NMIs, standard development organisations, regulatory bodies, international and sectoral industrial organisations, and universities must be involved in collaborative projects for the definition of the necessary instruments to ensure safe and prompt actions in case of risk to HSE.

# B.6 Action plan

#### Short Term

Undertake a review of all EU legislation relevant to nanomaterials and nanotechnologies:

Given the rapid development and use of nanotechnologies and nanomaterials, it is crucial to adapt nano-relevant regulatory measures, as required, to safeguard consumer health and safety, as well as the environment. Regulations are required that do not restrict innovation.

The European Parliament has requested the EC to undertake a thorough review of all EU legislation that is relevant to nanomaterials.<sup>16</sup>

Suggested action: One of the first requirements is to review the adequacy of regulation concerning nanometrology, adapt implementation instruments and make regulatory changes where necessary. Depending on needs, regulatory changes must be approved. The review should address all major policy areas<sup>17</sup> including consumer protection policies and product safety legislation. The review should also encompass chemical legislation such as Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) and environmental and workers' protection legislation. In particular, the review should address the adequacy of specific legal safety requirements, such as limit values for certain chemicals in products. It is important to foresee the adaptation of such specific requirements in legislation while fostering

<sup>7</sup> See Co-Nanomet Consultative Document published at <u>www.euspen.eu/nanometrology</u>

<sup>16</sup> European Parliament resolution of 24 April 2009 on regulatory aspects of nanomaterials (2008/2208(INI))

standardisation developments for technical specifications, such as nomenclatures and measurement test methodologies. Further to the review, the needs for adapting existing relevant legislation (for example, specific legal requirements) must be identified.

Definition of nanomaterials for regulatory issues:

Regulations require definitions of the items that are comprehensive, science-based and harmonised.

Suggested action: measurement experts should actively participate in the discussions and consultations surrounding the preparation of definitions of the term 'nanomaterial' and related terms. The development and issuing of common metrologists' opinions about the measurability and practicability of proposed definitions, be it in legislative texts or in documentary standards, could be required to have a more significant influence.

In order to base a nanomaterials definition for regulatory purposes on size alone, the upper nanoscale limit should ideally be high enough to include all types of materials that would need particular attention for regulation due to their nanoscale. Upper limits which are often used in existing definitions, for example 100 nm, may require the introduction of one or more qualifiers based on structural features or properties other than size, in order to capture structures of concern (for example agglomerates or aggregates) with a size larger than 100 nm in the regulation. Establishing a nanoscale size range with rigid limits would be advantageous for a clear, single and broad definition, and would be important with regards to enforceability of a definition in a regulatory context. A lower limit of 1 nm and an upper limit of 100 nm or greater are reasonable choices. For a detailed discussion see the JRC reference report<sup>18</sup>, which provides practical considerations to be taken into account.

Development of reliable measurement methods for size determination and reference materials:

<sup>&</sup>lt;sup>18</sup> Report - Considerations on a Definition of Nanomaterial for Regulatory Purposes, Göran Lövestam, Hubert Rauscher, Gert Roebben, Birgit Sokull Klüttgen, Neil Gibson, Jean-Philippe Putaud and Hermann Stamm 2010

ec.europa.eu/dgs/jrc/downloads/jrc\_reference\_report\_201007\_nanomaterials.pdf

For a size-based definition to be enforceable, the availability of techniques for measuring size is crucial. Although the size of an object can be measured from less than 1 nm and upwards, such size measurements are not straightforward. Several techniques are available, but not all are suitable for routine analysis. Moreover, different methods may yield different values when measuring the same structural feature. Indeed, for materials other than perfectly spherical nanoparticles, the results of these methods are not often comparable and measured particle size values should be regarded as method-dependent. An individual nanoparticle can be precisely visualised and measured by electron microscopy; however, every measurement refers to a single nanoparticle (single particle method). Therefore, statistical significance is a problem, particularly for poly-disperse samples, where there are also difficulties in obtaining a representative sample of particles.

Suggested action: These issues can be addressed by measuring multiple samples and using automated image recognition software for size determination, but only at the expense of time and costs.

A definition of nanomaterial that is based on size should also take into account size distributions and non-uniformity of samples. For the sizing of large numbers of nanoparticles, standard methods are available at present<sup>19</sup>.

<sup>&</sup>lt;sup>19</sup> See Co-Nanomet Consultative Document published at <a href="https://www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>

#### Medium Term

#### Development of reference materials:

A wide range of other products are based on mixtures which include deliberately added nanomaterials, in order to introduce or enhance effects caused by the particular properties of nanomaterials. Well known examples here are cosmetics and sunscreens, but paints, cleaning products and lubricants can also contain nanomaterials. Such products are usually not perceived as nanomaterials but as mixtures containing nanomaterials, i.e. the nanomaterial is an ingredient and not the end product.

Suggested action: New methods and reference standards should, therefore, be developed, validated and standardised, targeting reduced measurement uncertainty and improved traceability of measurement results to internationally accepted standards<sup>20</sup>.

Development of test methods and reference materials referring to human HSE: Reliable specific tests are urgently needed to evaluate potential risks associated with nanotechnology/nanomaterials uses, mainly in relation to:

- chemicals and materials;
- cosmetics;
- foods;
- pharmaceuticals and medical devices; and
- occupational safety and environmental protection.

In particular, there are a number of concerns about consumer safety regarding sunscreen lotions and cosmetics, such as skin-care and colorant products (about one third of nanoproducts), since they are applied directly onto the human body. For sunscreens, titanium dioxide and zinc oxide nanoparticles are used as they absorb and reflect ultraviolet rays. Liposomes, i.e. tiny vesicles made out of the same

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<sup>&</sup>lt;sup>20</sup> Reference materials for measuring the size of nanoparticles, Thomas P.J. Linsinger, Gert Roebben, Conxita Solans, Roland Ramsch, Trends in Analytical Chemistry, available online October 20, 2010 at: <a href="https://www.sciencedirect.com">www.sciencedirect.com</a>

material as cell membranes, are also used in certain cosmetic products that have nanomaterial content<sup>21</sup>.

Suggested action: Development of reference materials, test methods and other standards to provide support for regulation to guarantee safe nanotechnology-based products.

As nanomaterials can have unique physical and chemical properties, risk assessment protocols and/or analytical techniques designed for conventional chemicals may not be appropriate for accurate risk assessment. At this moment, neither in vivo nor in vitro assays are sufficiently developed for a full risk assessment. Novel, high-throughput, in vitro methods need development, validation and implementation.

In particular, more research is needed to determine:

- toxicity and internal dose: investigate and determine the physical and chemical properties that influence the potential toxicity of nanomaterials (for example size, shape, solubility, surface area, oxidant generation potential, surface funtionalisation, surface charge, chemical composition);
- deposition pattern of nanoparticles in the lung and their translocation to the interstitial and to extrapulmonary organs: evaluate short- and long-term effects of pulmonary exposure to nanomaterials in various organ systems and tissues (for example, lungs, brain, cardiovascular);
- dermal effects of topical exposure to nano-objects: whether these nanoobjects can penetrate into the skin, and whether they can cause immune alterations;
- genotoxic and carcinogenic potential;
- biological mechanisms for potential toxic effects;
- reliability of in vitro screening tests: create and integrate models to help assess potential hazards;

<sup>&</sup>lt;sup>21</sup> The Woodrow Wilson Nanotechnology Consumer Products Inventory, <a href="https://www.nanotechproject.org/inventories/consumer/">www.nanotechproject.org/inventories/consumer/</a>

- metrics other than mass that are possibly more appropriate for determining toxicity;
- methods to measure the mass of respirable particles in the air and determine whether this measurement can be used to measure nanomaterials;
- field-test practical methods to accurately measure airborne nanomaterials in the workplace;
- an evaluation of the effectiveness of engineering controls in reducing occupational exposures to nanoaerosols and developing new controls when needed:
- efficient personal protective equipment; and
- physical and chemical properties that contribute to dustiness, combustibility,
   flammability and conductivity of nanomaterials;

With respect to medical devices and pharmaceutical products, the existing provisions are also generally considered adequate for nano-related products, due to the detailed authorisation procedures required. However, specific regulations are needed for advanced nanotechnologies, such as implantable biomaterials or nanoparticle-based drug delivery systems, where technologies can be defined as a device, drug and biomaterial.

## **B.7** SWOT analysis

#### **Strengths**

The base of the normative references is standards. A lot of work has been done in these years by all national and international organisations for developing standards in nanotechnology. Some of them are already available and many others are currently under discussion.

# Weaknesses

The fields of application of nanotechnology are growing and the discussion about the criticality of nanoproducts has just started. Indeed, there is still a lack of standards

and reference materials, both mandatory for setting and optimising new effective normative references.

The expectation of commercial success of nanotechnology in many different fields pushes the competition between national and pan-European strategies. As a consequence, difficulties can be foreseen for the fruitful discussion of the necessary concerted normative.

## **Opportunities**

An adequate and convincing normative will increase markets and international trades. Indeed, only the acceptance by the general public through wide dissemination will allow for the spreading of the results obtained by the research. This can be done by managing the community expectations about responsible development of nanotechnology.

#### Threats

Prohibition, restrictions or limitations regarding ethical and safety aspects because of large measurement uncertainties, as well as regulations based on an over interpretation of the Precautionary Principle, may compromise the exploitation of nanotechnology potentialities.

On the other hand, monopolies and concentrated control and ownership of new nanotechnologies may push political systems to underestimate the social risks of nanoproducts.

For the development of a good normative, the active participation in the discussion of all the Countries is mandatory. However, some Countries, most notably developing countries, may be excluded from international standards negotiations or non compliant.

# **B.8** Summary

With the exception of standardisation<sup>22</sup>, there is no common effort to elaborate regulatory issues that could be shared at an international level. Indeed, the development of a regulatory framework accepted at global level is necessary to have common rules for safety, to facilitate trade and to avoid regional divide. Therefore, the promotion of international cooperation in defining the regulation is fundamental. The political decision processes have to be supported by all stakeholders. Indeed, an evidence based risk research policy is the key for rapid and efficient results. Thus regulatory issues should be defined and coordinated at the international level, as well as coordination at the inter-departmental and inter-agency level in individual jurisdictions. Regulatory approaches to nanotechnology should be flexible and adaptive. Moreover, risk management approaches should strive to comprehensive, by incorporating a lifecycle approach to govern the potential risks of nanotechnology. In all, the stakeholders of the process for regulation development must be engaged appropriately and the regulatory system should be transparent.

In the previous paragraphs it was shown that there are many information deficits requiring research activities. Furthermore, research results need to be transcribed into standards and guidelines and then must be interrelated to the regulatory framework at the international level.

It is possible to conclude that the challenge for the regulatory process is to ask the right questions and to generate the answers needed to demonstrate safety, on a case-by-case basis, before the products enter the marketplace.

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<sup>&</sup>lt;sup>22</sup> See Co-Nanomet Consultative Document published at <a href="www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>

# **ANNEX C - Nanometrology and standardisation**

#### C.1 Overview

# C.1.1 Documentary measurement standards

Standardisation (as in the development and use of documentary standards) is widely regarded as one of the prerequisites for the successful introduction of nanotechnologies in industry. Nanotechnology in its myriad applications will need standardisation regarding:

- 1) terminology and nomenclature;
- 2) measurement and characterisation of nanomaterials and systems;
- 3) HSE; and
- 4) product and process standards.

While risk is a prime prerequisite in some areas of nanotechnology, many nanoproducts that are not dangerous also rely on nanometrology to ensure general product quality and conformity compliance, to facilitate trade and promote customer satisfaction and efficient production. Conformity assessment refers to specifications of product quality characteristics, often given in harmonised written standards (norms) or equivalent normative documents, and with regard to EU legislation. Measurement systems and laboratories can also be conformity assessed if required.

Since product quality is determined through conformity assessment, by measurement quality in testing products, standardisation is developed both for the nanoproducts themselves, as well as for the nanometrology used to test them. For example:

 harmonised definitions of nanoproduct quality characteristics are needed in regulatory areas where a producer is to be quality assurance certified; and  recommended and standardised measurement methods are needed in support of measurement quality assurance of laboratories making measurements in the workplace.

The section focuses on documentary measurement standards.

# C.1.2 Metrology in the context of standardisation

Metrology ultimately aims for the reliable assessment of material or product properties independent of a specific measurement method. The idea is that the property value carried by the measurement object is described without the need to refer to the way it was measured. This aim is in conflict with the use of documentary measurement standards if they impose unnecessarily detailed procedural steps.

Nevertheless, it is well accepted that the results of the measurement of many industrially or regulatory significant material properties are method-defined, because of the limited understanding or accessibility of the intended property. For such method-defined properties, documentary standards are actually required to prescribe the detailed steps to be taken to achieve comparable measurement results. It is only logical that for relatively new measurement fields, such as that of nanometrology, the basic scientific understanding is not yet sufficiently developed to be able to work without documentary measurement standards. Nanotechnology presents a unique opportunity for proactive development of metrology and standards, since the technology is still in its infancy.

# C.1.3 New standards and measurement techniques in nanotechnology

The expected growth in nanotechnology depends on the development and introduction of new standards and measurement techniques in nanotechnology. Few standard measurement methods exist today, and the existing measurement techniques and instrumentation will have to be developed. These measurement techniques and standards will have to ensure that newly developed products will

satisfy the exacting requirements of reliability and economy when practically implemented.

Quality-assured measurement enters essentially in the development of both generic standards and sector-specific standards. The need for measurement standards for nanotechnology will have to be continuously reviewed in consultation with key stakeholders. This will also contribute towards making nanotechnological products freely tradable and safe to use. Two main challenges for metrology in standardisation, including nanometrology, to be addressed are given below.

1) Documentary standards are increasingly not only setting basic requirements but also becoming a valuable pedagogical resource in disseminating nanometrological techniques to the workplace. However, norms sometimes make reference to unrealistic and ambiguous measurement specifications and unclear requirements.

Suggested action: metrology organisations work more closely with standards bodies and others to improve written norms in this respect.

2) Industry and society are placing increasingly exacting demands on measurement in terms of complexity and accuracy, including meeting grand challenges, such as in Energy and the Environment and Health and Safety, employing nanotechnology.

Suggested action: There is a need for more pre-normative R&D in the metrological aspects of standardisation for nanotechnology. Significant contributions are needed to pre-normative research, in developing the necessary metrological aspects at all stages of product development – from initial product design and specification, through metrological design, procurement and actual measurement, to final product conformity assessment and marketing.

One example of a recent action addressing these challenges is the signing of a Collaboration Agreement between EURAMET and the European Committee for Electrotechnical Standardization<sup>23</sup> (CENELEC).

# C.1.4 ISO/TC 229 Nanotechnologies

To what extent can existing documentary measurement standards directly cover nanotechnologies? Can they be easily adapted for use at the nanoscale?

Due to the different physico-chemical properties on the nanoscale it is evident that a simple amendment or downscaling of existing standard methods into the nanometre regime will, in many cases, be insufficient. To give an example from dimensional metrology, techniques such as coordinate measuring machines, profilometry and optical methods do not typically yield a sufficient lateral resolution to be applied in nanometrology. Instead, techniques such as scanning probe microscopy (SPM) or scanning electron microscopy (SEM) need to be applied, which need separate standards due to their different physical principles employed for imaging. Therefore, the International Organization for Standardization (ISO), the most globally recognised standardisation organisation in the field of documentary measurement standards, established in 2005 a new Technical Committee, ISO/TC 229 Nanotechnologies, for:

'Standardization in the field of nanotechnologies that includes either or both of the following:

1) Understanding and control of matter and processes at the nanoscale, typically, but not exclusively, below 100 nanometres in one or more dimensions where the onset of size-dependent phenomena usually enables novel applications.

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<sup>&</sup>lt;sup>23</sup> 'Metrology and Standardization in Europe', www.cenelec.eu/Cenelec/CENELEC+in+action/News+Centre/Press+releases/EURAMET.htm

2) Utilizing the properties of nanoscale materials that differ from the properties of individual atoms, molecules, and bulk matter, to create improved materials, devices, and systems that exploit these new properties.<sup>724</sup>

Within ISO/TC 229, Joint Working Group (JWG) 2 addresses measurement issues. Most documents currently drafted in JWG 2 focus on the application of one particular measurement technique for the characterisation of carbon nanotubes. Also related to measurements are the activities of ISO Technical Committee (TC) 229/WG 3, which is developing a set of test methods to help assess the impact of nanomaterials on health and the environment. The main topics currently addressed by WG 3 are toxicology, inhalation and migration of nanoparticles, as well as guides for handling, disposal and occupational risk management.

## C.1.5 CEN/TC 352 Nanotechnologies

The European counterpart of ISO, the European Committee for Standardization (CEN), established in 2006 the committee CEN/TC 352 Nanotechnologies. This TC has the same scope as ISO/TC 229. CEN/TC 352 members have explicitly chosen to avoid duplication of the ISO/TC 229 work. Recently, CEN has decided to assign the EC standardisation mandate M/461 to CEN/TC 352.

#### C.1.6 Other standardisation committees and related organisations

Prior to the creation of ISO/TC 229, several other ISO/TCs were already in charge of specific measurement techniques or other standardisation issues that are in some way relevant to nanotechnologies. Both ISO/TC 229 and CEN/TC 352 recognise that there is potential overlap with other TCs within ISO and CEN, but also with International Electrotechnical Commission (IEC), OECD and in other organisations, mentioned in Figure 1. Most of the organisations that have established a formal liaison with ISO/TC 229 are shown in Figure 1, except for the more recently established liaison with the Bureau International des Poids et Mesures (BIPM).

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<sup>24</sup> www.iso.org/iso/iso\_technical\_committee?commid=381983

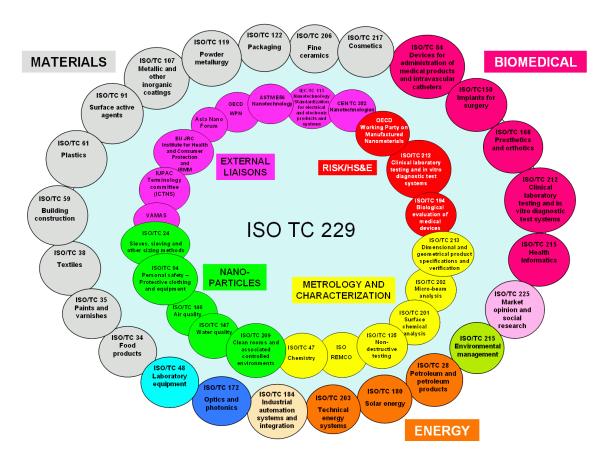


Figure 1 Current and potential future liaisons of ISO/TC 229 (source: ISO/TC 229 overview by Peter Hatto, Chairman of ISO/TC 229)

The liaison-TCs with the highest nanometrology-relevance are given below:

- ISO/TC 24/SC 4 Particle characterization;
- ISO/TC 201 Surface chemical analysis (includes scanning probe microscopy);
   and
- ISO/TC 213 Dimensional and geometric product specification and verification (includes surface texture).

# C.2 Vision for the next ten years

The ambition for the nanometrology community is to intelligently identify and select the most important needs for internationally agreed documentary measurement standards and to develop them in a metrologically reliable way, avoiding unnecessary specificity and duplication of standards.

# C.3 SWOT analysis

#### Strengths

## ISO/TC 229 Nanotechnologies:

The mission of ISO/TC 229 is broad and flexible, in the sense that it is designed to fill gaps left by other TCs. The members of the relatively young ISO/TC 229, with delegates often approaching nanotechnology from very different perspectives, have now had the time to learn to know each other better, and the TC is ready to more efficiently tackle current and future work items.

## ISO/TC 229/JWG2 Study Group on Metrology:

The work of JWG 2 is strongly supported by a number of companies and NMIs leading in nanometrology, which was reflected in the creation of the JWG2 Study Group on Metrology. The collective expertise with high-resolution measurement instruments and knowledge of uncertainty contributions helps to ensure a strong metrological foundation for the measurement standards being developed for nano-object measurement.

#### The Nanotechnology Liaison Coordination Group (NLCG):

The NLCG was established in 2008 to provide a forum to help coordinate and harmonise the work of relevant technical committees and other organisations in the field of nanotechnologies and to identify cross-cutting gaps and opportunities and ways to address them. Represented are all organisations with a formal liaison to ISO/TC 229.

# Weaknesses

#### Broad variety of measurement aspects of nanotechnology:

The measurement aspects of nanotechnology related standardisation activities are manifold in several respects and keeping a general overview is a difficult task.

Limited resources for the development of standards:

Both companies and governments attach different priorities to standardisation in different world regions and countries. European as well as national standardisation policies thus vary greatly. While in some countries governments provide full or partial funding to support national standardisation committees, the cost of maintaining committees must be borne by industry in many other countries. This, in turn, leads to a situation in which the involvement in international standardisation does not necessarily reflect a country's role in research, development and application of nanotechnologies. For example, ISO/TC 201/SC 9 (SPM) is mainly driven by Korea, Japan and China. Although the UK and recently Germany actively support its work, Europe is clearly under-represented in this TC, considering the significant number of SPM manufacturers, especially in central Europe. While European companies are sometimes thought to be less standardisation oriented due to sufficient regulation in this part of the world, several smaller companies argue that the membership fees demanded by their national standardisation committees are the major obstacles to participation in the TCs.

Absence of link between expertise and voting behaviour in ISO and CEN:

A country's official ISO/TC-status as Participating Country (P-member) does not necessarily mean that the country can convince its experts to study and judge the draft documentary standards submitted for approval. While the absence of expert judgement should lead to abstention at the ballot, it is suspected that many positive votes are cast by default, potentially affecting the quality of the released documents.

Lack of metrological understanding with standard developers:

It is surprising to see how many of the people contributing to the development of documentary measurement standards have a limited generic knowledge of basic metrology principles.

#### Opportunities

EC standardisation mandate (M/461 Nanotechnologies and nanomaterials):

Recently, the European Commission has sent to CEN, CENELEC and European Telecommunications Standards Institute (ETSI) a standardisation mandate (M/461

Nanotechnologies and nanomaterials), requesting the development of Technical Specifications and European Standards for:

- methodologies for characterisation of nanomaterials in the manufactured form and prior toxicity and ecotoxicity testing;
- sampling and measurement of workplace, consumer and environmental exposure to nanomaterials;
- methods to simulate exposures to nanomaterials; and
- guidance documents and protocols in relation to occupational handling and exposure.

These topics were selected from a more extensive list produced by CEN/TC 352 in its response to a prior programming mandate (M/409).

Recent agreement between CEN/CENELEC and EURAMET:

Following the recent signing of a Collaboration Agreement between EURAMET and CEN-CENELEC<sup>9</sup>, an action plan is now being formulated.

Suggested action: Ensure that the present Co-Nanomet Strategy is implemented in the EURAMET/CEN-CENELEC Collaboration Agreement and in similar initiatives elsewhere.

#### **Threats**

Non-realistic expectations and time pressure:

It is often stated that standards for various aspects of nanotechnology need to be introduced as soon as possible because nanotechnology offers significant potential for the economy and is growing rapidly. It must be noted that for a large number of issues, the fundamental understanding required to judge the reliability of a measurement method is not yet available, potentially leading to the premature adoption of standard methods, which can slow down the development of alternative, more effective methods.

### Duplication of work:

The perceived sense of urgency has led to many standardisation initiatives in various international organisations, as well as at the regional and national level. This has increased the risk of double-work or incompatible standards.

## Method-specific versus product-specific standards:

It must be noted that documentary measurement standards are often application - or product - specific. There is a risk of developing similar yet different standards for the measurement of the same property for different materials.

## C.4 Action plan

### Short term

A first and ongoing exercise is the careful analysis of the EC request formulated in M/461; based on this analysis, a realistic and detailed roadmap for the development of the requested deliverables needs to be developed. This will include necessarily intensive consultations between CEN/TC 352 and ISO/TC 229, and with other CEN and ISO TCs through the NLCG. In parallel, technical specifications will have to be produced for a number of already identified, newly developed or validated measurement methods.

#### Medium term

Based on previously developed technical specifications, full European Standards need to be elaborated. In addition, metrology guidance needs to be provided in a more systematic manner to the developers of documentary measurement standards.

#### Long term

Foster a more 'standardisation-friendly' attitude in European industry, regulators and academia, for example, by using FP levers.

# C.5 Summary

While the field of nanotechnology is currently searching for a harmonised, standardised language and terminology, there is also a need for internationally agreed and validated, standard measurement methods. The proposed measurement methods cannot be standardised unless they have reached a minimum degree of metrological maturity. On the other hand, these standard methods and the measurands defined therein are especially important for a young area such as nanotechnology, where lack of full scientific understanding prohibits the use of fully traceable, method-independent measurements. The interdisciplinary nanotechnologies standardisation committees ISO/TC 229 and CEN/TC 352, the TC 229 hosted Study Group on Metrology and Nanotechnologies Liaison Coordination Group, and the new EC Standardisation Mandate M/461 provide structure to this important work in the years to come.

# **ANNEX D - Metrology for laboratory quality assurance**

#### D.1 Overview

## D.1.1 Elements of measurement laboratory quality

Product quality is very much determined by the quality of the measurements made to test against specifications. The following two key aspects of metrology need to be disseminated to the workplace in order to ensure quality nanomanufacturing.

Metrological traceability – to universal measurement references of the SI system, where possible, ensures that different measurement results (those made at different times and different locations) can be compared objectively. Products and processes assessed for conformity using traceable test results will themselves be comparable and robust.

Measurement uncertainty – a declared level of quality in the measurement result; an estimate of unknown measurement errors. Measurement uncertainty is always needed when judging whether different measured values are equal, or not. It is also essential in assessing the risks of incorrect decisions of compliancy when comparing a test result with a specification limit. The latter is a key concern in nanometrology, since decisions of conformity assessment, for instance in regulatory contexts, are often of concern.

#### D.1.2 Formal quality assurance approaches

Measurement laboratories must make dedicated quality assurance (QA) efforts to create and maintain the confidence of their customers in the results of their measurements. Therefore, an increasing number of measurement laboratories

operate a formal quality system that guards and assures the quality of their measurement results.

Thus, parallel to formal quality assurance of a nanoproduct manufacturer or supplier, for instance according to ISO 9001 management standards, is the formal assurance of the measurement quality according to ISO 17025<sup>25</sup> measurement management standards – since product quality is determined by measurement quality.

At the base of all measurement laboratory QA systems are the main metrology concepts mentioned earlier: metrological traceability and measurement uncertainty<sup>26</sup>. To implement the generic metrology concepts, laboratory staff need to have a good, practical understanding of the meaning of these concepts for the measurements they are making. Measurements have to be fit-for-purpose so that measurement uncertainty is optimised in a balance between measurement costs and the consequential costs of incorrect decisions of conformity. Also required are laboratory QA tools such as method validation approaches, calibration services, ILCs, and (certified) reference materials. The lack of this practical understanding, and of the mentioned tools in the area of nanotechnology, explain why few laboratories have nanotechnology-related measurement methods in the scope of their accreditation at present.

# D.1.3 Specific metrological challenges for quality of measurements at the nanoscale

The various units of the SI for the different measurement quantities are defined at a level where the actual measurement accuracy is highest. For example, the unit of mass is set at the one kilogram level, since weighings at heavier and lighter levels are less accurate – heavier weights are more difficult to handle while smaller weights are more easily perturbed. These trends, where accuracy generally falls towards both shorter ranges and longer ranges, are similar for many measurement quantities.

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<sup>&</sup>lt;sup>25</sup> ISO/IEC 17025: 2005, General requirements for the competence of testing and calibration laboratories

<sup>&</sup>lt;sup>26</sup> Co-Nanomet Introductory Guide to Nanometrology, P E Hansen et al. 2010

Measurements at the nanoscale (1 nm to 100 nm) are no exception. Because of this, it will be increasingly more challenging in nanometrology to establish metrological traceability and to reach target measurement uncertainties.

Examples of nanometrological challenges include:

- longer chains of metrological traceability;
- the need for new nanoscale reference materials and standards;
- the need for new measurement instrumentation and test methods working at the nanoscale;
- the need to measure new characteristics unique to the nanoscale; and
- measurements in challenging environments, for example ultra-high vacuum.

For details of these technical challenges, see the consultative document (www.euspen.eu/nanometrology).

# D.1.4 General dissemination of metrological traceability to the nanotechnology workplace

Two main aspects of presenting metrology in nanotechnology for industry and in the workplace are:

- contents of dissemination; and
- best practice of dissemination.

#### Contents of dissemination:

One key observation is to place (nano-) metrology in the right context, so that when people in the workplace have to choose the relevant metrology tools and human resources, they can relate product requirements – often in terms of conformity assessment to specifications – to the corresponding metrological requirements.

The idea is that – in any field – one first (step 1) has to reach a consensus about a common terminology to aid communication, especially in multidisciplinary areas such

as nanotechnology. One needs, for example, to define what is the nanoscale, what is a nanoparticle, how does one denote the effective surface area of a nanoparticle, etc.

Once the common language is agreed, the next step (step 2) is to give a harmonised and unambiguous description of the recommended methods, instruments and laboratory capabilities for making measurements and testing nanosystems.

Thereafter, with the words and methods to describe and test products, one can then proceed to standardise requirements on nanosystems from the point of view of regulatory limits (step 3 - in which standards set the essential requirements) or even in the wider, non-regulatory area where nanosystems need to be assessed, not because they are perceived as dangerous, but rather to promote efficient and fair trade for both producer and consumer.

## Best practice of dissemination:

Earlier studies have aimed at educating small to medium sized enterprises about the opportunities offered by new micro- and nanotechnologies. The Swedish programme minST (expert competence in micro- and nanotechnology) concluded the following. The basic requirements are:

- actively-engaged people with the skills and capabilities for best knowledge exchange;
- active information dissemination, including homepage, newsletters, articles and papers about successful case studies in industry. An annual competition for the 'best product of the year' was one element with a large impact;
- division of the programme into regional parts where regional rather than central – programme leadership has ensured a local anchoring and adaptation to local needs and resources; and
- continued public financing, since the motors of the programme were the search and idea development activities, which are difficult to operate on purely commercial conditions.

# D.2 Vision for the next ten years

Parallel with an increased effort to better disseminate the generic metrology concepts, one of the ambitions of the metrology community over the next ten years must be to develop and provide the tools that will enable laboratories in the field to improve, assure and demonstrate the quality of the results of their measurements on the nanoscale and/or on nanomaterials.

## D.3 SWOT analysis

## **Strengths**

There is a well-developed structure of metrology institutes:

The NMIs are organised regionally (through EURAMET) and internationally (through CIPM – International Committee for Weights and Measures) in organisations that provide the platforms for discussing best practice and new approaches also relevant for QA in nanometrology.

The laboratory accreditation structure exists:

All European countries have an accreditation body that organises laboratory accreditations in accordance with well-established generic reference documents, which also apply to the nanometrology area (for example, ISO/IEC 17025, Good Laboratory Practice, etc.).

#### <u>Weaknesses</u>

The understanding of basic metrology concepts is limited:

A solid understanding of the basic metrology concepts is not sufficiently widespread. This is the case for many industry branches, and results from an underexposure of pupils and students to these concepts, even at the academic university level, where interest in metrology is limited.

Several views on metrology are confronted in the multidisciplinary area of nanotechnology:

It is only recently that metrology practices in laboratories from different fields (physics, chemistry, biology) have started to converge, for example, with more harmonised terminology, but also in terms of traceability systems.

Lack of basic scientific understanding of phenomena occurring at the nanoscale:

The field of nanotechnology is particularly challenging for measurement science. Many of the relevant measurement methods provide very indirect observations of the property intended to be measured. It is particularly challenging to establish QA systems in this area.

Only a few measurement methods are fully validated for application at the nanoscale: A method can only be used to produce reliable measurement results after proper validation in the laboratory that wishes to apply the method. Due to the novelty of the area, the metrological traceability systems and the laboratory QA tools required to validate methods, establish laboratory proficiency and achieve traceable results are not available.

#### **Opportunities**

It is possible to turn sound laboratory QA into a competitive advantage:

In the area of nanometrology, there is still a great deal of uncertainty about the reliability (general accuracy, but also basic comparability) of measurement results. There is plenty of scope to improve the confidence in measurements, which has the potential to be turned into a competitive advantage.

The multidisciplinary field of nanotechnology can draw from multiple QA approaches: The multidisciplinary field of nanotechnology has the potential to promote crossfertilisation of QA approaches proven in different areas (physics, chemistry, biology).

Use of EMRP to develop a deeper and more common understanding:

The recently launched EMRP can be used to further develop the basic metrological understanding required to build the QA systems also needed by non-NMI measurement laboratories and to create a more common understanding of different metrology practices.

Increased international collaboration on production of reference materials:

The European Reference Materials (ERM) initiative, which currently unites the Federal Institute for Materials Research and Testing (BAM, Germany), LGC (UK) and IRMM (EC) as reference material (RM) producers, is committed to commonly apply the ISO Guide 34 principles<sup>27</sup> in their RM certification activities. ERM partners submit their candidate RMs to peer-review by the other ERM partners. This existing structure can also be used as an example method to increase mutual confidence in QA tools in the nanometrology area.

#### **Threats**

The 'chicken-and-egg' problem of metrological traceability:

Every new measurement field has to face the same problem: to develop QA tools, one needs reliable measurements, and to prove the reliability of measurement results, one needs QA tools. For example, candidate laboratories for the characterisation of a to-be-certified reference material (CRM) need to have proven competence in the measurement of the to-be-certified properties, which is difficult without a CRM. It is clear that the area of nanometrology covers many measurements for which this 'chicken-and-egg' problem will not be solved without going through a number of iterative efforts, stepwise improving the reliability of the measurement results.

Inability to convince the stakeholders of the relevance of metrological rigour and of the necessity of their involvement:

The Co-Nanomet project has revealed a low industrial and academic interest for metrological laboratory QA developments. With only a limited number of working traceability systems in the area of nanotechnology (with the exception of dimensional

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<sup>&</sup>lt;sup>27</sup> ISO Guide 34: 2000 General requirements for the competence of reference material producers

nanometrology<sup>28</sup>), it is difficult to convince, in particular, industry to commit to rigorous metrological practice.

Limited number and variety of approaches among expert auditors:

Every accreditation system depends on the expertise of the auditors. In the field of nanometrology there are few technical experts with personal experience in QA. The number of these qualified auditors and the commonality of the approaches they use in particular areas of nanotechnology, might be insufficient to meet the future demands of the measurement laboratories. Overall confidence in, and the quality of, conformity assessments of products can be enhanced by ensuring that Conformity Assessment Bodies (CAB) for testing, certification and inspection laboratories themselves comply with relevant requirements and possess the necessary technical competence, independence and impartiality. Increased metrological support to CAB in the form of measurement advice and in assessing measurement uncertainty is required (better coordination of metrological training, conferences and knowledge exchange; programmes of technical assistance and development, cooperation with developing countries and promotion and enhancement of European metrology), as well as an enhanced access to metrological traceability (through regular calibrations of CAB measurement standards and methods with a laboratory higher in the hierarchy; better organisation of inter-comparison activities).

# D.4 Action plan

### Short term

An important action, for metrologists and metrology organisations in general, is to increase the training opportunities for generic metrology issues (metrological traceability, measurement uncertainty and use of reference materials in QA). More specifically for the nanotechnology area, methods for which QA tools do not exist need to be identified and ILCs set up for these methods. Also, the database for nanoscale RMs should be extended and improved, hosted by BAM<sup>29</sup> while also

<sup>&</sup>lt;sup>28</sup> See Co-Nanomet Consultative Document published at <a href="www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>

promoting the principles of production and use of RMs laid down in the ISO Guides developed by the ISO Committee on Reference Materials.

There is also room to harmonise QA approaches for nanometrology across NMIs (via EMRP, ERM, etc.) and between accreditation bodies, standard development organisations and sectoral organisations (for example, via ISO/TC 229 and its NLCG).

### Medium term

After a period of searching for improved mutual understanding and collection of best practices, the metrology community should agree and disseminate accepted method validation approaches. Based on such an understanding, training courses dedicated to metrology at the nanoscale can be developed, along with proficiency testing schemes, new CRMs and new matrix RMs.

### Long term

In the long term, online training tools for metrology at the nanoscale can be developed. Also, based on the experiences with earlier efforts to develop RMs, it will be necessary to develop new certified matrix RMs. Ultimately, different laboratory accreditation attitudes will need to be aligned.

# **ANNEX E – Technical Challenges and Opportunities**

# E.1 Dimensional nanometrology

#### E.1.1 Overview

In order to achieve the desired functional properties, nearly all nanotechnology applications require accurate control of the dimensions of the relevant structures. Consequently, measurement techniques are necessary which allow the measurement of the dimensional properties as well as the specific functional properties, turning the characterisation of nanoproducts into an interdisciplinary task, with dimensional measurements predominantly in the leading role. In the following pages, the strategic aspects of dimensional nanometrology are discussed.

# E.1.2 Vision for the next ten years

Over the next ten years the following points have to be improved or realised:

- high resolution, high precision, and high accuracy instruments for different scales, at different geometries and with different materials have to be established;
- better understanding of measurands, by improved knowledge of interaction between probe and samples at the nanoscale, has to be developed;
- calibration services and solutions (techniques/strategies) for problems related to nanoscale measurements have to be provided; and
- the stability of measurands by well defined standards, rules and guidelines, as well as worldwide accepted traceability, has to be assured.

## E.1.3 Action plan

### Short term

The contacts between measurement instrument manufacturers, users and NMIs need to be strengthened to determine manufacturers' and users' requirements both from each other and from the NMIs. More communication and education will lead to increased awareness of RMs, standards and guidelines, greater use of the calibration services offered by NMIs, and more opportunities for cooperation, resulting in greater scientific output for society. Also, future strategies to close the gap between micro- and nanoscale measurement methods should be developed and consistency of measurement results between instruments ensured. To enable handling of large area scans, intelligent measurement schemes need to be worked out to derive data giving an overview of the sample in combination with a betterautomated identification of the regions of interest for subsequent high-resolution measurements. This includes the implementation of automation of measurement batches, their analysis and reporting. Furthermore, 3D AFM probes and related scanning strategies need to be improved to perform 3D measurements, for example of sidewalls (MEMS/NEMS, micro-bores, sidewalls on photomasks, etc.). In addition, systematic investigations of the influence of control parameter settings in different SPM modes, on different materials are required. In-depth understanding of probesample interactions is the key to decrease measurement uncertainties in this field. The awareness of this fact should be increased and collaborations among NMIs, institutes and industry should be initiated.

The current standardisation projects, for SPM particularly in ISO/TC 201/SC 9, need stronger support both by the experienced European NMIs and especially companies active in the field in order to be completed timely and to avoid an Asian predominance. Standardisation activities need to be communicated to the stakeholders in order to get their feedback during the current drafting phase and later on to ensure a broad acceptance with uniform terminology and procedures among the user community.

The current incompatibility of data formats needs to be overcome by increasing the awareness for open data formats and, where possible, agree an a standard data exchange format as, for example, currently drafted in ISO/TC 201/SC 3.

# Medium term

Different measurement techniques should be combined to cover various measurement ranges and bring together information on different physical quantities (for example, from optical microscopy, scatterometry, coordinate measuring machines (CMMs), SPMs, SEMs and partly also analytical techniques). Physically correct data fusion methods are needed for this. For SPM, comparative research of probe characterisation methods (based on SEM, direct imaging of test structures, blind reconstruction), specific for different probe types (shape, material, funtionalisation), including modelling and simulation are required. This is essentially linked to an improvement in the understanding of probe-sample interactions including higher-resolution, lower-noise instruments combined with modelling/simulation.

Furthermore, the understanding of image formation in SEM and helium ion microscopy needs to be enhanced. In the field of RMs: topography-free resolution standards for SPM modes such as scanning near-field optical microscopy (SNOM), magnetic force microscopy (MFM) and Kelvin probe microscopy (KPM), as well as 'golden standards' for easy everyday verification made of application dependent materials (not only 'ideal' materials) and 3D standards for single-step calibrations, should be developed. In order to transfer routine standard calibration tasks from NMIs to calibration laboratories, accredited calibration laboratories need to be established. NMIs should focus on the smallest uncertainty calibrations and novel, challenging calibration tasks.

#### Long term

A systematic understanding of probe-sample interactions and their influence on dimensional metrology is required, as a prerequisite to enable the NMIs to fulfil the future demands of the nanotechnology industry. Full data fusion of results measured at difficult object geometries and by different measurement techniques is desirable. Accurate and full 3D probing by SPM methods, using a large variety of different probe types suitable for various tasks, should then be possible. This includes the

development of a large toolbox of probing strategies and methods. Additionally, high-resolution tomographic techniques based on DUV-light, x-rays, electrons and ions are required as auxiliary or supporting techniques.

#### E.1.4 Infrastructure needs

As well as international networks needed for collaborations and comparisons, national metrology centres are also necessary. Obtaining the smallest uncertainty measurements and undertaking novel, challenging calibration tasks are the role of the NMIs. Additionally, external accredited calibration laboratories are needed for routine calibrations of nanoscale reference artefacts.

The infrastructure of the NMI nanometrology centres should contain high-level, traceable measurement instruments based on different methods and different physical interactions (SPM, electron microscopy, scatterometers, analytical techniques). This is important since a comparison of data and a combination of information from a variety of methods is necessary to interpret nanoscale measurements. The instruments used should be commercial as well as specialised, purpose-built metrological systems.

In addition, highly skilled personnel are needed for operation and instrument development, as well as basic research, particularly on the physical modelling and experimental verification of probe-sample interactions and analysis of measurement results. The development of new methods and instruments with the focus on traceability is of fundamental importance because this is often neglected in commercial instruments. The experience gained in the past years has shown that development work in the field of instrumentation is of the utmost importance for the quality and understanding required for subsequent use of these devices and their calibration. No study of operating instructions or training courses can replace the knowledge acquired from this development work.

### E.1.5 R&D strategies

The long-term goal is to close the gap between macro-, micro- and nanoscale measurement methods and measure in the nano-world with relative uncertainties approaching those obtained in the macro-world. The strategy to achieve this goal is to ensure consistency of measurement results between the instruments used throughout the measurement ranges. This will be achieved by appropriate extension and improvement of the technical infrastructure in conjunction with theoretical work to underpin the interpretation of the measurement results. Comparisons of the results obtained using instruments in overlapping regions of their measurement ranges are also necessary for performance validation. As with macro- and micro-scale measurements, the effects of environmental conditions will need to be considered in addition to the extra 'nano-effects' (often quantum) that occur in the nano-world.

# E.1.6 Emerging topics and priorities

Although various trends and requirements have been highlighted, due to the continuing miniaturisation in many high-technology fields and the fast growing number of nano-enabled products, the exact nature of individual potential measurement tasks is hard to predict. This is especially true for measurements and calibrations on the product level and, therefore, the main effort is the development of a flexible metrological infrastructure meeting the general requirements already outlined in this chapter. Based on these, metrology instrumentation, together with a well-founded and coordinated development of flexible application-dependent calibration artefacts and methods, are mandatory.

The priorities for future work in the field of dimensional nanometrology are discussed above and have to be worked out in close contact with customers of calibration services from industry and academia.

# E.1.7 SWOT analysis

## **Strengths**

Reference instruments already available at several NMIs:

Contrary to most other fields of nanometrology, reference instruments are already available - at least for the basic measurement tasks in dimensional nanometrology. Some NMIs have already gained more than twenty years of experience with highly sophisticated SFMs (Scanning Force Microscopes); most of these instruments (so-called metrology SFMs) are equipped with laser interferometry for direct traceability to the SI-unit metre, and several NMIs in Europe have been offering certified calibrations for several years already. Apart from SFMs, SEMs are used, and diffractometers/scatterometers both in the visible and increasingly also in the DUV, UV and x-ray range have been developed and become available for measurement services. The latter is achieved especially by those NMIs that have access to synchrotron radiation sources, for example PTB with its own laboratories at the Berlin Electron Storage Ring "BESSY II", as well as the PTB-owned Metrology Light Source (MLS).

## Existing networks, good contact to many industries:

European (and global) collaboration in this field is rather advanced, with well-established European structures and networks (for example, within EURAMET). Furthermore, the metrological level is strengthened by the interaction between European institutions via competitions, collaborations and comparisons aided by, for example, EURAMET and EU projects. Further strategies are guided by roadmaps for dimensional metrology (through EURAMET and EMRP).

In this field, good contacts to instrument manufacturers and various users in industry already existed from the very beginning. Metrologists and different industries have worked together in a number of national and European projects and are involved in different kinds of networks.

## <u>Weaknesses</u>

Lack of awareness in academia and many industries:

While metrologists and some industries have been working together in the field for many years already, the relevance and importance of metrology in general is not sufficiently promoted in many industries and particularly in academia. Although dimensional metrology underpins all nanometrology and is a generic requirement for nanoscience and technology, it is often referred to, only in passing, in conjunction with other specific requirements for advancing nanoscience and technology. Due to this broad range it is, therefore, difficult for metrologists to address this rather heterogeneous community in an efficient way. For example, conferences and congresses, as well as scientific and technical publications, typically aim at a certain, highly specialised group. This weakness could, if not addressed, become a danger, and the importance of dimensional metrology could be overlooked.

## Physics of interaction still not sufficiently understood:

By focusing on applied research and development, NMIs may run the risk of neglecting basic metrology research, for example, into the understanding of interactions between probe and sample. This is highly important, as there is a need to qualify measurands at the nanometre scale, such as the diameter of nanoparticles, line edges, thin film interfaces, etc., that are of particular interest to several industries. However, such fundamental metrological research takes a lot of effort and can be expected to be time and labour intensive. A systematic understanding can thus only be achieved in the medium and long term, demanding continuous research that cannot be completed within the standard framework of typical three-year R&D projects with short-term employment contracts. Consequently, this potential weakness demands a higher number of long-term employed researchers that are actively involved in the research.

### **Opportunities**

NMIs in Europe and worldwide record a steady increase in the demand for calibration services at the nanoscale. In the past few years, many standardisation activities have been launched to ensure uniform terminology and procedures, both for the measurement techniques (as for example, ISO/TC 201, also comprising SPM) as well as for the characterisation and measurement of certain objects, for example in ISO/TC 229. Apart from this specific standardisation, there is a greater drive for general quality assurance as per ISO 9000 and ISO 17025. It is obvious that further growth in nanoscience will need dimensional metrology in order to determine the position and size of features and understand interactions.

Standardisation requirements and the growing demands of industry help to justify increased efforts at NMIs in this field, and should push dimensional nanometrology. EURAMET promotes collaboration and larger projects to tackle outstanding problems (for example, EMRP projects) in this field.

# **Threats**

# Tough global competition:

Particularly in the field of dimensional nanometrology, tougher competition with other regions worldwide is witnessed, especially with East Asia and partly with North America. In recent years, several NMIs and other reference institutes in these world regions have invested significant resources in building up their measurement infrastructure. Asian countries often drive standardisation in this field, and the relatively low awareness for standardisation issues in most European countries may, in the long term, prove disadvantageous for European companies once international standards have been agreed upon.

### Hard to satisfy industrial measurement needs:

While some measurands, such as mean pitch, can be determined with very low uncertainties, many other measurands, such as structure width and shape, can currently, and in the near future, only be determined with relatively large uncertainties. As these measurands are critical for the advancement of several industries, dissatisfaction among the potential customers is likely, especially if the rather long measurement times and, therefore, high costs for high-resolution scanning microscopy methods are taken into account. Furthermore, progress in this field can only be achieved by continuous long-term research

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Finally, as knowledge about calibration services and technology transfer opportunities from NMIs are hard to convey to the broad range of potential users, the services of NMIs may not be sufficiently visible.

# E.1.8 Summary

It is clear that for nanoscience and technology to be effective, they require underpinning with dimensional nanometrology. At the very least this will allow the size of nanoscale artefacts to be quantified and located in the macroscopic world.

The main technical challenges appear to be extending nanoscale measuring capabilities over a larger dimensional range, understanding probe-sample interactions and increasing the accuracy of measurements, not just the precision.

The EMRP offers an excellent opportunity for European NMIs via strengthened collaboration to address the dimensional metrology requirements at a fundamental level, thereby facilitating future European capabilities in nanoscience and metrology.

# **E.2** Chemical nanometrology

### E.2.1 Overview

The metrology activities in the chemical area at the nanoscale concern the measurement of the composition of chemical species, chemical states or structural properties. It has been noticed that such analysis, for example SNOM, AES or XPS could not alone provide a complete chemical description of the product to be measured. Thus combined information is necessary to have a complete chemical analysis.

Furthermore, in order to establish traceability, some specific efforts are needed in the development of certified reference materials (CRMs) useful for this area. The number

useful for chemical analysis at the nanoscale is relatively small and there is a strong need for further developments.

Finally, there is a lack of a metrological aspect (like uncertainties on dimensional measurements or measurements of chemical properties of nanoparticles) with chemical toxicity in human bodies. A correlation between measurements of size distributions and chemical compounds on the one hand, and tracheal diseases on the other hand, is of great interest and should be studied.

# E.2.2 Vision for the next ten years

Issues requiring development in chemical analysis of nanomaterials and at the nanometre scale have been identified and are summarised in this section.

The first concern is the characterisation of chemical compounds, which aids understanding of certain areas of the research as well as the fabrication mechanisms such as:

- understanding the first steps of nucleation in case of fabrication of nanomaterials;
- better understanding of biomass and green molecule chemistry, and development of new solvents with low trace of impurities in case of waste reduction;
- in situ structure and interfaces for the evaluation of the impact on air quality;
   and
- functionalisation of nanomaterials and development of nanoparticles for the field of bioanalytics, in the fields of biology and medicine.

An important point for these fields is the necessity to analyse one parameter together with the others. For example, in the case of air quality studies, the impact on health of species able to change depending on their environment is highly dependent on different parameters. Thus the dimension, chemical compounds and biologic characteristics of the media studied need to be analysed together.

## *X-ray tomography and imaging:*

X-ray digital radiography, computed tomography and imaging can have relevant applications to cultural heritage, medicine and industrial applications. Chemical imaging aids in the answering of difficult questions, especially when these questions occur in complex chemical environments. For example:

- understanding of corrosion processes;
- pollution control and understanding of interaction in dynamic systems;
- study of ancient specimens of plants and animals in the archaeology and palaeontology area; and
- the self-assembly of small molecular units into larger structures is a common and important occurrence in nature. In the biological realm, proteins and RNA fold into specific functional conformations.

The imaging techniques described are divided into three main categories. In addition, a section on image processing and computation, which has a bearing on virtually all chemical imaging techniques, is also included:

- optical imaging (Raman, infrared and fluorescence) and magnetic resonance;
- electron microscopy, x-rays, ions, neutrons;
- proximal probe (force microscopy, near field, field enhancement); and
- processing analysis and computation.

Consequently, new approaches have to be developed and implemented, integrating existing ones based on x-ray photoelectron spectroscopy (XPS), spectroscopy, SPM and secondary-ion mass spectrometry (SIMS) to improve spectral and spatial resolution, contrast and sensitivity for elements and molecular species. The new methods must also have capabilities to work in situ, at ambient air and/or in liquid environments.

## E.2.3 Action plan

### Short term

Incoming legislation on the registration, evaluation, authorisation and restriction of chemical substances places responsibility on the chemical industry, including downstream users of chemicals, to provide appropriate safety information with which to improve the protection of human health and the environment through the better and earlier identification of the intrinsic properties of chemical substances.

In order to understand the risk presented by the possible presence of a chemical contaminant, knowledge is required of its toxicity and of the level of exposure. Reliable exposure assessment requires robust analytical methodology. Existing standards for the validation and performance evaluation of methods have led to improved analytical capability and better interlaboratory agreement of results. However, increasing the availability of robust, cost-effective methodology should be the benchmark for future developments in the field of chemical analysis of contaminants.

Specific sensors and portable instrumentation providing sufficient sensitivity and traceable measurements must be designed, allowing on-line quality control. In the case of low concentrations of chemicals in liquid or gas, new sampling methods must be developed and evaluated by means of round-robin tests.

# Medium term

Analytical spectrometry should merge with integrated circuit (IC) technology to design novel devices such as lab-on-a-chip, miniaturised photo-spectrometers, micro-glow discharge systems and lasers integrated in waferscale CMOS (complementary metal-oxide-semiconductor) technology. Examples are the application of nuclear magnetic resonance (NMR) on a chip, chip-based mass spectrometry and chip-based infrared and Raman spectroscopy. Then there is the need to merge atomic and molecular techniques, through combinations of instrumentation, such as Raman for molecular analysis with x-ray fluorescence and x-ray absorption spectroscopy (XAS) for providing elemental information on a single sample; modulated techniques for

molecular and atomic information in plasma techniques; and edge techniques through electron and x-ray analysis for spatially resolved speciation.

Scanning near-field optical spectroscopy (SNOM) has to be validated as a tool for highly localised laser-based analysis using Raman spectroscopy/imaging, fluorescence detection and imaging and laser ablation mass spectrometry. Specific standards should be developed and standardised methods should be defined. All these techniques allow in situ molecular and elemental analysis. In SNOM combined with fluorescence and Raman spectroscopy, a continuous laser beam is delivered to a very small spot on the sample surface and scattered light is collected in transmission or reflection. SNOM–Raman usually relies on the use of surface enhanced Raman scattering (SERS) and allows only the observation of surfaces with special composition. In SNOM-laser ablation mass spectrometry, the ablated material is ionised and then analysed by a mass spectrometer.

With a few exceptions, presently available probe methods for studying matter with nanoscale lateral resolution (scanning tunnelling microscopy (STM), atomic force microscopy (AFM)) provide the shape of a nanostructure, its surface topography and its local electrical properties, but little or no elemental information. There are possibilities to integrate scanning force microscopy with ion beams.

The European Synchrotron Radiation Facility (ESRF) blue book<sup>30</sup> lists the following points that need further study for online analysis in the future:

- quantitative x-ray fluorescence (XRF) analysis by fast Monte Carlo methods;
- procedures for improved crystallographic analysis;
- enhancement of fast small angle x-ray scattering (SAXS) data analysis; and
- X-ray absorption near edge structure (XANES) micro-mapping combined with iterative transformation factor analysis.

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<sup>&</sup>lt;sup>30</sup> Anon, Science and technology programme 2008–2017, A Programme to Upgrade Europe's Strategic Centre for Science and Technology Research, 2 volumes, ESRF, Grenoble, France, September 2007, www.esrf.eu/AboutUs/Upgrade/documentation/purple-book/.

The goal in SIMS development is to achieve enhanced sensitivity and develop methodological innovations for lateral resolutions well below 100 nm. Thus, SIMS needs to attain the following capabilities:

- a lateral resolution down to 10 nm in order to keep pace with developments in microelectronics and other areas in materials science. Recently, major progress has been realised in this field and the lateral resolution is now at 50 nm;
- an ultimate depth resolution close to the atomic scale, as increasingly shallow implantations are utilised in microelectronics (the 'microelectronics roadmap') and micro-miniaturisation;
- the ability to detect all elements and their isotopic composition as well as small molecules;
- increased sensitivity with a realistic goal of one ion detected and mass analysed for every 2 to 100 atoms sputtered, in order to be able to analyse nanoscale materials at the level of 10 nm<sup>3</sup>; and
- the non-destructive chemical analysis of biological processes in the crowded intracellular environment, at cellular membranes and between cells, with a spatial resolution well beyond the diffraction limit.

#### Long term

Synchrotron based techniques must be developed and used for simultaneous elemental, structural and molecular analysis of heterogeneous samples in one single instrument. Developments in both focusing/imaging optics and in detector technology can yield enormous benefits to the fundamental understanding of phenomena spanning such diverse fields as cellular biology, chemical engineering and semiconductor physics. Synchrotron based techniques will also contribute to the quality assurance of measurements, including traceability and uncertainty analysis, and will be instrumental in the elaboration of RMs and the validation of other methods of analysis.

#### E.2.4 Infrastructure needs

The need is to provide access to an infrastructure that supports rapid advancements in science, engineering and technology at the nanoscale. This can be done by providing shared, geographically diverse laboratories, each with specific areas of technical excellence, and providing fabrication, synthesis, characterisation and integration resources to build structures, devices, and systems from atomic to complex large-scales. It should be possible for users to perform research on-site using facility equipment, training and staff support and eventually remote usage.

Generally speaking, we could determine that the metrological characterisation of instrumentation is not the priority of the end-users. In fact, as the scale is reduced, or as the sensitivity has to be improved, the determination of uncertainties appears more crucial. In this way, another need is thus to improve the dissemination of the metrological basis in institutions. However, techniques are merging in the market, becoming available to end-users, before a complete process has been efficiently established.

### E.2.5 R&D strategies

Satisfying the surge in demand for nanoscale analytical characterisation in the years ahead will depend upon a multi-disciplinary combination of methods associated with improved high-technology sample preparation skills. Simultaneously, it will be necessary to further refine existing methods for direct elemental speciation in solid samples.

Instrumentation and protocols to guarantee the metrology in the nanometre range for compositional/elemental analysis must be developed and standardised to evaluate nanomaterials, thin layers (below 100 nm) and devices. This will allow control of the chemical composition and homogeneity of impurities content (for example, in case of information storage in phase memories, diffusion properties of the impurity have to be studied and correlated).

In view of the expected industrial exploitation of nanotechnology in the future, the development of on-line chemicals are mandatory, both for the quality control of processes and to control potential environmental risks. The quantification of very low concentrations of toxic species in industrial waste and water must be assessed, in particular with respect to the compounds highlighted by European Union and REACH directives.

The evaluation of the potential risks and toxicity of nanomaterial products is urgently needed. The determination of the correlation between toxicity and the chemical composition of specified compounds should be based at the first step on the categories determined by OECD.

# E.2.6 Emerging topics and priorities

The need to address calibration issues for electron probe microanalysis (EPMA) is extremely important for industrial applications. The specific use of measurements using synchrotron or neutron measurements could greatly improve the chemical analysis.

Regarding specifically environmental and health requirements, it is necessary to perform in situ and dynamic measurements. Thus, some progress has to be made on resolution and sensitivity and the impact of background in the case of some techniques should be reduced.

Another point, which has previously been mentioned, is that in order to obtain complete information on a specific product, the analyses of the few useful parameters have to be combined together, prior to the interpretation of the results.

Finally, some quantitative measurements are accomplished using a standards/matrix correction methodology whereby the unknown is measured under identical conditions relative to a suite of standards. Earlier pilot studies (see CCQM-P80, -81 and -95) demonstrated that use of this method resulted in differences in measurements larger than the reported expanded uncertainties.

# E.2.7 SWOT analysis

### Strengths

The most important strength is the actual expertise in NMIs. BIPM participants are already developing protocols for XPS, Auger electron spectroscopy (AES) and EPMA techniques. This is the first step to elaborate the traceability of the measurements of the chemical composition of nanolayers with uncertainties of approximately 5 % relative. Matrix effects are reduced by calibration with alloy reference samples, as opposed to pure elements.

Another strength is the large number of universities and academic laboratories that provide a wide spectrum of available techniques and develop knowledge on adequate equipment for the nanometre scale. Collaboration between NMIs and the academic institutes should be increased in order to disseminate metrological protocols, while improving the core measurements.

# <u>Weaknesses</u>

Manufacturers make strong advances in the measurements and production of nanomaterials. In the meanwhile, the lack of standardisation creates a widening gap between the metrological control and the end-user utilisations.

The lack of metrological basis in the development of nanoscale techniques is another weakness of the whole process chain.

Equipment required for the improvement of the overall areas in metrology needs wide investments from NMIs, as well as specific resources adequate to the specific studies.

#### Opportunities

The cross understanding of nanometrology (interdisciplinary interactions, academic-NMIs) could yield new levels of information, for example with the standardisation and control of safe nanoparticle production.

The induced results are of great support for the development of specific industrial areas: greenhouse gases and air quality, food analyses and molecule calibrators for therapeutics and diagnostics.

### **Threats**

Each material needs a unique characterisation, depending on both the chemical compound and the matrix considered. In this way, the understanding of the mechanisms on production processes as well as the impact on human health is wide. This dependence may not easily lead to a helpful standardisation.

# E.3 Thin film nanometrology

#### E.3.1 Overview

Nanometric thin films are central to many advanced technology products and applications. The primary nanometrological requirements are for control of thickness and composition. Although thickness has the dimension of length, compositional nanometrology techniques<sup>31</sup> may alternatively measure mass per unit area or surface coverage, which are sometimes the more desirable or accessible measurands.

The sheer diversity of thin film compositions, thicknesses (monolayer to multilayer), and functions requires advances in metrology across broad fronts to enable and support continued development. The main trends and requirements allow a clear strategy to be devised for the development of appropriate nanometrology, with the ultimate aim of enhancing the development and competitiveness of European manufacturing industry.

### E.3.2 Vision for the next ten years

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<sup>&</sup>lt;sup>31</sup> See Co-Nanomet Consultative Document published at <u>www.euspen.eu/nanometrology</u>

Advances in measurement techniques and instruments have historically been the prime mechanism by which nanometrology supports advances in thin film technology. The continued development of existing techniques is expected to lead to thickness measurements with increased sensitivity, spatial resolution and applicability. The emergence of new techniques (helium ion microscopy, as a current example) is also likely, although less easy to predict.

The major capital cost and complexity of state-of-the-art instrumentation will remain a barrier to wide take-up by industry, but will be partially offset by various approaches, for example the availability of instruments with higher measurement throughput and ease of use (including developments in automation and web access) and by the establishment of expertly operated open access facilities or centres.

Modelling is expected to play an increasing role as an intrinsic element of measurement techniques, for example, the use of image simulation in transmission electron microscopy (TEM) measurement of very thin layers.

Demands for control of some thin-layer manufacturing processes using non-invasive high-throughput measurement techniques will be met, where possible, using non-contact optical techniques, such as interferometry and spectroscopic ellipsometry (SE). Confidence in these techniques will be based on careful cross-referencing to other, more direct, measurement techniques and supported by the development of written or physical standards.

All areas will benefit from closer linkages between industry, NMIs, academia and major (for example, synchrotron or accelerator) facilities.

Major progress is also expected in many specific challenges of thin film nanometrology, for example, the characterisation of nanoparticle or porous thin films, the classification and quantification of surface functional groups and molecular layers and nanoscale 3D characterisation. It is less easy to predict progress in some other longer-term challenges such as the development of techniques for application in air or liquid environments.

# E.3.3 Action plan

# Short term

A prime target is to accelerate advances in techniques where Europe has a lead. Examples here include helium ion microscopy, low energy ion spectroscopy (LEIS)/medium energy ion spectroscopy (MEIS), time-of-flight SIMS (TOF-SIMS), SE and interferometry techniques. This could be achieved by calls for projects developing new measurement applications addressing high-priority thin film measurement problems. These topics include (a) non-destructive depth-profiling; (b) quantification of thin molecular layers and surface functional groups; (c) characterisation of porous thin films; and (d) non-contact high-throughput large-area measurement of thickness and related factors. Participation of end-user industries in these projects would be highly desirable.

A powerful mechanism for achieving early take-up of technique advances by industry is to site state-of-the-art instruments in laboratories able to provide focused open access services to industry. Funding for the instrument purchaser and for applications development ensures there are benefits for the instrument manufacturer, the host laboratory and for the user industries, without market distortion.

Understanding the reasons why different techniques give different values (for example, of layer thickness) is critical to establishing measurement confidence, as well as for agreement on correct techniques and methodologies. Focused comparisons between techniques have a valuable role here, as shown by the extensive and detailed comparative measurements of ultrathin silicon oxide films<sup>32,33</sup> which have led to improved method understanding. A specific residual question relating to discrepancies in TEM measurement values should be resolved, owing to the importance of this technique to nanometrology. The principle can be extended either to new materials challenges or to new techniques.

<sup>&</sup>lt;sup>32</sup> Seah M P et al. 2004 Critical review of the current status of thickness measurements for ultra-thin SiO2 on Si: Part V, results of a CCQM pilot study *Surf. Interf. Anal.* 36 1269-1303

<sup>&</sup>lt;sup>33</sup> Seah M P et al. 2009 Ultra-thin SiO<sub>2</sub> on Si IX: absolute measurements of the amount of silicon oxide as a thickness of SiO<sub>2</sub> on Si, *Surf. Interf. Anal.* 41 430-439

Measurement round-robins have important roles to play, firstly in establishing the need for standards (whether written methodologies or physical standards), and secondly in disseminating these to industry and embedding best practice.

### Medium term

A widespread need for rapid non-contact measurement of thin film thickness uniformity and related factors (for example, conformality and defects), with realistic sampling over large areas, is likely to be best addressed by advances in sensitive optical methods such as SE and interferometry. These are indirect measurements, requiring assumptions of optical constants or modelling, and thus development work is required, including detailed comparison with direct reference techniques.

The development of nanometric thin film measurement standards is required on an ongoing basis. Careful assessment is required to decide, on a case-by-case basis, where the best solution is provided by physical thickness or coverage standards, or written methodological standards. Highly controlled deposition techniques such as atomic layer deposition (ALD) may be advantageous for preparing certain material standards.

Porous and nanoparticle thin films present new measurement challenges for solution. Some parameters, such as surface area, require new measurement approaches (for example, the integration of complementary techniques – gas adsorption and SE on a single platform), whereas others, such as pore size distribution, may also require new definitions. Application-specific measurements may also need to be developed.

## Long-term

High-resolution 3D tomography is an ideal approach for the metrology of thin films that are either nanostructured or applied in small areas (for example, patterned features). Focused ion beam (FIB) methods are already available for this, although advances in non-destructive techniques (for example, x-ray or electron tomography) and in speed and ease of use would be desirable. R&D is also required for the definition of 3D measurement parameters, and their extraction from 3D data sets with traceability and uncertainty estimation.

Many advanced nanometrology techniques (electron beam, mass spectrometric and surface analysis) are vacuum-based, and there is a significant lack of techniques able to probe surface layers in air or liquid. Work to close this gap is particularly important for certain biological applications where the surface chemistry, structure and function are intrinsically dependent on the liquid environment.

#### E.3.4 Infrastructure needs

The following European infrastructure developments are recommended in support of the action plan:

- establishment of centres or nodes allowing easy industry access to the latest advances in instrumentation (similar to the previous EC-funded Semiconductor Equipment Assessment (SEA) scheme);
- schemes to facilitate access to large facilities (for example, synchrotron and accelerator), especially for industry and SMEs; and
- a mechanism for improved reactive problem solving, for example, a pan-European measurement brokerage service and database.

### E.3.5 R&D strategies

Most of the action plan can be effected through calls for collaborative R&D proposals against the summary topics identified. Proposals that involve collaboration between instrument manufacturers, metrology institutes and industrial users (of the metrology) would be most desirable. Action plan items concerning inter-technique comparisons and measurement round-robins, and development and evaluation of standards, would also benefit strongly from funding for industry participation to ensure that the advances in best practice are taken up effectively.

## E.3.6 Emerging topics and priorities

The specific challenges and measurement gaps identified from Co-Nanomet stakeholder discussions are as follows:

- advancement of techniques for deriving layer thicknesses and related parameters non-destructively, including approaches for non-destructive depth profiling of multilayers;
- measurement of functional organic thin films (as surface monolayers or multilayers), in particular the characterisation of molecular structure and quantification of surface functional groups;
- techniques for measurement of porous thin film parameters, such as surface area, pore size and pore size distribution;
- techniques for 3D imaging of nanostructures. These may require the development of new parameters for structure definition as well as for measurement:
- rapid non-destructive large area measurement of film thickness and other parameters. Optical techniques (for example, SE and interferometry) are particularly attractive for this, being non-contact and non-invasive. Refractive index determination and initial referencing to direct measurement techniques are necessary for optical thickness measurement;
- further development of TEM methods, to achieve improved confidence and industrial throughput in measurement of film thickness and other parameters; and
- there is an identified need for more reference and written specification standards.

# E.3.7 SWOT analysis

## Strengths

Europe has notable strengths in thin film technology, namely a strong European thin film industry (deposition equipment and suppliers) as well as a strong European measurement instrument development and manufacturing base. Additionally, there is strong European NMI and academic R&D activity in these areas.

### Weaknesses

There is a relatively weak indigenous end-user manufacturing industry in many technology sectors employing nanometric thin films when compared with the scale of large-volume manufacturing capacity that exists in Asia and the USA, for example, in flat-panel displays and microelectronics industries.

## **Opportunities**

The main opportunity addressed by the action plan is to leverage European advances and infrastructure in thin film measurement and deposition to improve the competitiveness of European end-user manufacturing (especially SMEs). A prime mechanism for this would be to accelerate the development and introduction of new and improved measurement techniques and instruments within Europe. A secondary mechanism would be to improve links between NMIs and industry, and in particular to promote the take up of good metrology practice within industry.

#### Threats

The centre of gravity in end-user manufacturing lies outside Europe in many technology sectors. Since this acts as an attractor for the co-location or indigenous development of supporting industries, it threatens the existing European strengths (the converse argument being that support for these could act as an incentive for end-user manufacturers to locate in Europe).

## E.3.8 Summary

Thin films of nanometric thickness are a key functional element in many advanced technologies. There are clear trends in requirements for closer control over layer thickness, composition and numerous other parameters, and these drive the development of improved nanometrological tools and methods. An action plan is proposed which addresses the key measurement gaps identified by Co-Nanomet stakeholders, and builds on the identified European industry strengths in metrology instrument manufacture and thin film deposition. Acceleration of measurement technique development, and lowering barriers to allow rapid uptake of advances by European industry (including SMEs) is seen as the highest priority. Support for technique comparisons and development of standards is also important.

# E.4 Mechanical nanometrology

## E.4.1 Overview

Mechanical nanometrology is concerned with techniques that can be used to measure the mechanical properties of materials in the nanoscale. This includes nanoindentation, the use of AFM to measure mechanical properties, nanotribology and direct measurement of the mechanical properties of nano-objects such as carbon nanotubes.

The main priorities in the future will be measurements of localised constitutive properties; measurements under extreme conditions such as high temperatures and aqueous environments; determining the mechanical properties and performance of nanostructured coatings and the assessment of materials durability and lifetime; and measurement of the performance of soft solids, including those of importance in the bio-nano interface.

A key future emphasis will also be to combine nanomechanical information with other aspects of nanometrology such as structural and compositional information.

## E.4.2 Vision for the next ten years

Once the issues of traceability of measurements for force, displacement and tip shape have been resolved, research in the area will be focused on the development of techniques to provide a much richer set of materials property information at length scales appropriate to the user requirements under a much more extensive range of environments, including extended temperature ranges and different aqueous or gaseous environments. Particular areas where research will be a priority are mechanical property evaluation to smaller scales using AFM technology and the development of robust procedures for inverse modelling that give stress-strain constitutive relationships for materials with nanoscale spatial resolution. Fast mapping of properties with automated and optimised test systems will also come to the fore, with in situ measurement of properties (combining imaging and nanoindentation with FIB machining) being used more widely. New techniques based on multi-axis measurement will be used to achieve better materials properties measurement, including the full set of elastic constants. Another goal will be to make the instrumentation more robust and portable so that, instead of small samples being taken to the laboratory for testing, the instrument can be taken to the component in the field and on-line or in situ measurements can be made.

# E.4.3 Action plan

### Short term

The most immediate short-term requirements are the setting up of traceable routes needed for calibration of the lower force ranges and small displacement measurements that are required for nanomechanical measurements. There has already been considerable progress in this regard as outlined earlier, but it is only by making procedures straightforward and easy to use that these essential requirements are established as routine processes in laboratories. At the same time, better procedures are required for the traceable evaluation of tip shape down to the few nm

(0-5 nm or less) scale relevant for AFM probes. There is also a need to develop probes for reduced wear to give increased lifetime in terms of calibration and use.

Such developments need to be supported by the development of RMs that can be routinely used to check that instruments are working correctly. However, there will be limits to the atomic stability of materials kept in atmospheric conditions that make truly nanoscale reference materials unlikely.

Inverse modelling of nanoindentation is currently a major focus of international work in the area that promises to achieve the goal of providing full stress-strain information with high spatial resolution for a material.

The ready access to high-resolution property measurement through nanoscale contact mechanics with the availability of high-resolution displacement and force sensors has enabled significant advances in nanotribology. Here the aim is not only to develop an understanding of the wear and friction of nano- and micro-scale devices, but also to answer some of the fundamental questions behind friction and the wear of macroscopic engineering components.

Yet another pressing need is to extend nanomechanical measurements to more extreme operating conditions, such as high temperatures, controlled gasses and aqueous environments. Some progress has already been made here, but further work exploring the metrology of nanomechanical measurements in these areas is required and will increase demands for a fusion of nanomechanical, nanoimaging, nanostructural and nanochemical analyses.

## Medium term

In the medium term, it is likely that more metrological support for more complex testing, such as testing of FIB machined pillars or cantilevers, will be required. Another requirement will be to develop traceability routes for the calibration of in situ testing. As the metrology for nanomechanical measurements is improved, a key medium term requirement will be to disseminate good nanomechanical practice to stakeholders. Aspects here include cooperation with OEMs (Original Equipment Manufacturers), automation of good metrological practice within test systems, and

the education of users with the meaning and application of results which may be highly size dependent (such as yield stress or strength of a material).

### Long term

In the long term it is difficult to predict how the technology in the area will develop, but several areas are thought to be priorities for development over this time scale. The first of these is integrated 3D nanomechanical property measurement combined with technology, such as FIB, that can reveal the internal structure and chemistry of materials. This combination of technologies would yield a full structural, chemical and mechanical evaluation of a material with a fine scale, enabling a far deeper understanding of materials behaviour than is currently available.

Another goal is the development of non-contact techniques that provide nanoscale mechanical property information without any permanent damage to the sample. Finally, with the growing importance of the life sciences, it is highly likely that improved nanomechanical testing for nano-bio interface structures will be required.

#### E.4.4 Infrastructure needs

The main requirement for infrastructural support is an increasing provision of expertise and capability in nanomechanical testing. Without this support, the development of sound metrology is unlikely to occur. This will require expertise in the mechanical testing of materials at the nanoscale, as well as an understanding of force and displacement metrology.

There is also a need for support from NMIs for the basic traceable measurement and calibration of low forces and small displacements as relevant to mechanical testing at the nanoscale, but an urgent and more important calibration requirement is for support for the traceable measurement of tip shape, and evolution of tip shape with use for AFM and indentation measurements.

Thus, the supply of RMs is extremely important and NMIs will have a major role in their development since the issue of nanoscale instability is a metrological challenge.

Validation of increasingly sophisticated contact mechanics models will be a natural NMI role.

# E.4.5 R&D strategies

The focus of work in nanomechanical testing should be on areas of most relevance to end-users in industry. These include measurements under extreme conditions, such as high temperatures, gaseous and aqueous environments. This has relevance for industrial sectors, such as aerospace, land transport, civil infrastructure, minerals extraction and materials processing. Nanomechanical testing is also a key aspect in determining the mechanical properties and performance of nanostructured coatings that have the potential to improve energy efficiency and reduce impact on the environment. The assessment of materials durability and lifetime is another related area where nanomechanical testing can provide important knowledge on how materials degrade under mechanical and chemical exposure. Soft and viscous solids, including those of importance in the bio-nano interface, are also important growing areas for research in nanomechanical testing.

Effective nanomechanical testing requires a healthy metrological environment, targeting nanoscale metrology to foster this research. It is, therefore, essential that expertise and facilities are developed and maintained in European NMIs in this area. Networking between these NMIs will also be important to coordinate work for effectiveness and efficiency (for example, the elimination of undue duplication of work).

### E.4.6 Emerging topics and priorities

Much information has already been given under other headings on emerging topics and priorities, so this information will not be repeated here.

# E.4.7 SWOT analysis

# **Strengths**

- EU (SMT) funded standards have led to a good infrastructure for nanoindentation testing;
- · relatively mature in some areas; and
- some key centres of expertise, mainly in universities.

# **Opportunities**

- better information should yield improved take-up;
- new technologies could yield new levels of information;
- new application areas such as longer term materials degradation; and
- combination of technologies will enable more sophisticated virtual prototyping that relies on nanometrology and validated models.

### Weaknesses

- poorly understood by some sectors in industry;
- · uptake is poor; and
- measurement technology is expensive.

## **Threats**

- the EU-wide trend of reduction in government spending and industrial investment;
- some instrument suppliers supply proprietary measurement solutions that will slow down the spread of best measurement practice; and
- there is competition between leading experts in the area, with disagreement about the fundamentals of measurement that makes a coordinated approach less likely.

## E.4.8 Summary

Nanomechanical testing is an area of nanometrology making a major impact on industry through the development of improved materials. Such materials contribute to the important goals of more effective energy generation and use, improved sustainability and developing a more competitive European industry.

Future work in this area should be focussed on areas of highest relevance to endusers in industry. These include:

- measurements of localised constitutive properties;
- measurements under extreme conditions, such as high temperatures and aqueous environments;
- determining the mechanical properties and performance of nanostructured coatings and the assessment of materials durability and lifetime; and
- performance of soft solids, including those of importance in the bio-nano interface.

Development of improved metrology in European NMIs is key to ensuring a healthy measurement infrastructure for the area.

# **E.5** Metrology for nanostructured materials

### E.5.1 Overview

One of the drivers for the development of nanometrology is the increasing emphasis on materials with a specific structure on the nanoscale. This structure can be produced externally (for example, nano-objects such as particles, tubes and wires) then incorporated into a matrix or internally during the material's manufacture. These types of materials are now beginning to penetrate the market and are being found in a wide range of products. However, there are metrological roadblocks preventing more take-up of these types of materials. These include, but are not limited to:

· determining the dimensions of the specific nanostructures;

- detecting the position of nanostructures within the final material and ensuring good dispersion;
- measuring transport and interface properties; and
- understanding the effect of nanostructures on the final properties of the material.

In addition there have been recent well-highlighted concerns regarding the short-term health effects and long-term environmental build up of nano-objects. The public will need assurances regarding physical structure, stability within the material and safety in cases of accidental release of the nano-component. This is especially true in the disposal of such materials.

In order to meet these challenges there must be a pan-European strategy to build on the existing strong research base and infrastructure, much of which still resides on a national level. This pan-European strategy will be needed to deliver new capability, which will need to be accessible by many different organisations. The strategy includes new techniques and equipment that requires a minimal amount of training and the setting up of centres of excellence to provide access to high cost equipment (for example TEM), expertise and training.

Such a strategy will provide the ability to characterise nanomaterials and products, which is essential for European industry to fully exploit their related products and compete in the world market.

### E.5.2 Vision for the next ten years

Nanostructured products are now entering the market in many different sectors. These include high end products (for example, carbon nanotube impregnated bike frames, of which only a hundred or so will be produced per annum, at a cost of several thousand euros each), mid-range products (for example, improved car components, of which thousands will be produced at a cost of a few hundred euros each) and low end products (for example, nanoporous materials for water purification

in the third world, rolls of which may be produced at a rate of hundreds of square metres per day, at a cost of a few (euro) cents per m<sup>2</sup>).

Over the next ten years it is expected that this trend will continue with more and more nano-objects and structured materials being found in a wide variety of products, as described in the value train in Figure 2.

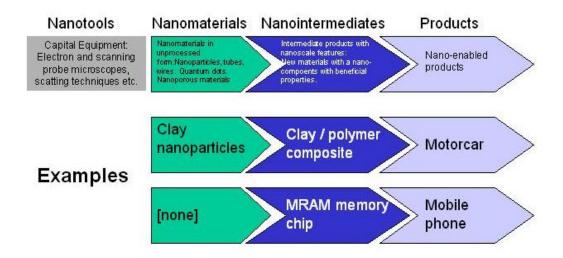


Figure 2 Nanotechnology value chain, derived from Lux Research Report on Nanotechnology

The food packaging industry is one example that highlights the potential benefits and risks posed by nanostructured materials. The use of nanomaterials in food packaging is already a reality with nanocomposites used in bottling technology that prevent the release of carbon dioxide, increasing the lifetime of the product. Silver nanoparticles, known anti-microbials, are being used in food storage products, helping to prevent the build-up of harmful bacteria. Nevertheless doubts remain about the long-term safety of such products and processes need to be developed to allow the capture and reuse of the nano-component during disposal or recycling.

There are distinct metrological challenges that will need to be overcome if this anticipated growth in nano-enabled products is to be achieved. These include, but are not limited to, the issues described below.

Researchers in both the public and private sectors are working on the development of novel nano-objects and structured materials with evermore complicated structures, for example, coated nanoparticles and doped nanotubes. These complicated structures, which might be structural or chemical in nature, will need to be identified, characterised and verified, paying close attention to the interface properties that will ultimately affect their behaviour. There is a need for methods to assess the structure and composition of nano-objects and structured materials, including:

- particle structure (porosity, fractal dimension); and
- composition of the material (core-shell, hybrid, janus-like).

Producers of these types of materials will need to ensure that their products meet tight quality control specifications. These are especially challenging for both nano-objects and nanostructured materials, where billions of specifically designed features have to be produced, a high percentage of which will need to lie within these specifications. There is a need for more on-line/in-line process control with:

- high temporal resolution and robustness (fouling, untrained personnel); and
- high concentration and harsh environments (pH, temperature, pressure).

This will mean going beyond the study of individual structures and will have consequences for the validation of instruments and techniques, as well as the requirements for RMs.

The end-users of these materials will require confirmation of both the dispersion of the nano-objects as well as the fact that they are behaving as they should. Hence sample preparation for analysis should reproduce the state of dispersion relevant for the proposed application. Sample preparation procedures can affect the accuracy and uncertainty of any test method and they will need to be validated. Related to this is dispersion stability, both during use and measurement.

There is a need to develop product and process models/property and process functions with nanoparticle properties-based measurements taken at different stages of processing and use.

The general public and regulatory bodies need reassurance that these materials are safe and pose no risk to human health or the environment. To achieve this, new metrology is required. This includes reliable and traceable methods to determine nanoparticle concentration in complex products and in the environment (air, water, soil, etc.). This is complicated by several facts, namely that it is often not clear which concentration metrics (number, surface area, mass) determine the effect of nanoparticles and difficult to distinguish between natural and engineered nanoparticles. Traceable methods for characterising the nanoparticle release from surfaces and agglomerates will become crucial for controlling the environmental exposure to nanoparticles. This will require RMs for determining the adhesive strength of nanoparticles to surfaces and agglomerates. Finally, a new nanoparticle classification/identification system will need to be devised, although the funtionalisation of nanoparticles will make a simple classification problematic.

# E.5.3 Action plan

#### Short term

In the short term, the immediate metrology roadblocks preventing exploitation of nanostructured materials need to be addressed. These roadblocks currently consist of the lack of any agreed standard measurement techniques, or transfer standards allowing cross comparison between different instrumentation, and any robust method to determine the dispersion of nano-objects within a material. Poor dispersion has been highlighted as one of the major causes of failure of nano-enabled products.

In addition, the future direction of the development of nanoscale characterisation methods needs to be defined by selecting the specific measurands that are required but beyond current instrumentation. These are discussed in section E.5.5 but will include 3D characterisation, interface and transport properties as well as dispersion/position of nanostructures.

#### Medium term

Metrology is required to enable the transfer of nanomaterials from the laboratory to the production plant, and finally to the market place. The researchers, producers and users of such materials will require full characterisation of the physical and chemical properties of their nano-objects, in 3D and with better than 1 nm accuracy. A large number of objects will need to be analysed in order for statistically reliable measurements to be made. Finally, the effects these properties have on both the behaviour of the nano-objects (interface properties, charge) and the final nanostructured material (thermal, electrical) needs to be understood.

## Long term

Metrology will concentrate on quality assurance and in-line measurement. For full commercialisation, in-line testing will be required to provide:

- size and shape measurement, plus chemical and physical structure of nanoobjects and the dispersion/position within a matrix; and
- verification of the described nanostructure, if produced within the material.

## E.5.4 Infrastructure needs

The development of capabilities in the metrology of nanomaterials should be concentrated into the two areas described below.

The first area concerns meeting the needs of researchers who are developing novel nanoscale materials. Such researchers require access to high-level capital equipment, for example transmission/analytical electron microscopy, SPM, surface analytical techniques, etc. This type of equipment requires high investment in resources, time and trained personnel. This expenditure is beyond the means of SMEs, and even some larger companies, and so it is important to improve access to these types of measurement techniques. This will require the creation of centres of excellence, but discussions will need to take place in order to determine the number, size and structure (for example, will they be incorporated into existing organisations or stand alone?), as well as to develop funding sources and a sustainable business

model. These centres should also be responsible for training and maintaining up-todate databases.

The second area is for widely accessible measurement capability, suitable for in-line measurement to be run by a non-specialised workforce. This requires the development of the capability along with the infrastructure to enable its uptake by the nanotechnology industry. This will require suitable transfer materials, international standards and training.

## E.5.5 R&D strategies

Several key capabilities will need to be developed, including 3D characterisation of materials, in terms of their chemical and physical structure, with sub-nanometre accuracy, and full characterisation of the interface properties of either nano-objects or nanostructured materials, in terms of their bioactivity, surface charge and transport properties, etc. A link needs to be established between these nanoscale properties and the macroscale behaviour of the final enabled product. Finally, this metrology capability needs to be provided in a form that is both accessible to non-trained personnel and suitable for in-line measurement (for example, by enabling real-time measurement of the size and shape of nanoparticles as they are produced). As mentioned earlier, this is particularly challenging due to the extremely small sizes and high densities involved.

# E.5.6 Emerging topics and priorities

With increasing penetration of nano-enabled products into the market place, new and justifiable concerns will arise. These will include:

Effect on public health of these materials:

Both in the short term accidental release of material from products, and long term leaching out of the nano-component and accumulation in the body.

#### Effect on the environment:

Accidental release and long term accumulation of such material may cause detrimental effects on the environment, in terms of wildlife and agriculture.

Sustainability of nanostructured material:

A strategy for the complete lifetime of a nanostructured product will need to be devised, from the production, use and final disposal, including the reclamation and recycling of the nano-component.

# E.5.7 SWOT analysis

#### Strengths

There are well-established European research activities in both the public and private sectors, including well funded public-based laboratories at both the university and government level, which provide current state of the art metrology for nanostructured materials as well as the ability to develop new measurement techniques.

Exploitation of nanometrology across the EU is enabled by both SMEs and well established large profit-making businesses based in Europe, or with a large European presence. Many of these businesses know the importance of, and have established, metrology capabilities.

There is a tradition in Europe of innovation in, and exploitation of, metrology. For example, both TEM and STM were developed in Europe.

#### Weaknesses

There is no overall strategy for the development and exploitation of nano-objects and structured materials. Health and safety concerns mean that progress has been sporadic.

There is no agreement on standard measurement techniques and no reliable transfer standards for spherical nano-objects below 50 nm, or for high aspect ratio particles.

Comparative spending in competitor countries, in particular the US, China and Japan, is much higher. The current economic climate means increased difficultly in obtaining funding.

There is a lack of specifically trained nanometrologists with the skills required to characterise nanomaterials.

#### **Opportunities**

Europe has the expertise, in both standardisation and instrumentation development, to fully exploit the new metrology capability that is required for the full potential of nanostructured materials to be realised. In turn, this will provide European industry with a competitive advantage in the exploitation of such materials.

## **Threats**

Increased penetration into the market place of materials with a nano-component will lead to concerns with respect to the safety of such materials. Strong regulations will need to be enforced to ensure the safety of nano-enabled products, which may not be possible without the necessary instrumentation and standards.

The adverse economic environment may mean a decrease in support and funding for nanometrology.

The lack of the ability to characterise nanomaterials and products, combined with strong competition from outside Europe, will harm the EU's competitiveness in the nanotechnology area.

#### E.5.8 Summary

Nanostructured materials hold great potential for the basis of products with new and beneficial properties. In order for European industry to be able to exploit these materials, new capabilities will be required in order to characterise their core components, structure and properties. These capabilities must be made available to industry both by the development of easier to use instrumentation and the setting up of an infrastructure allowing access to high value equipment, expertise and training.

In addition, the continuing safety of these materials needs to be established and monitored, for both short-term accidental exposure and long-term environmental impact.

# E.6 Electrical nanometrology

#### E.6.1 Overview

Nanotechnology applications are unimaginable without micro- and nanoelectronics. The consistent miniaturisation of the structural dimensions of semiconductor circuits, as per Moore's Law, will inevitably end in a small number of atoms defining a 'bit'. Manufacturing equipment and new research-intensive approaches for electronic components, such as molecular electronics and spintronics, are clearly assignable to nanotechnology.

Most of the NMIs requested the foresight study to examine electrical nanometrology research activities related to quantum electrical metrology.

Activities on electrical nanometrology listed can be divided into:

- support of metrology for the semiconductor industry (ITRS) working towards
   Extreme UV (EUV) lithography;
- application and improvement of nanoimprint technologies;
- metrology for the electrical quantum triangle (see Figure 3) related to measurements of, or improvements on, quantum based constants, for example, Quantum Hall effect (QHE), Josephson Junction (JJ) and Single electron transistor (SET);
- investigation into new methods to reduce the measurement uncertainty and to support traceability of electrical measurements of nanoparticles/nanoaerosols,

for example, providing traceable femtoampere measurements for nanoparticles, counting of thin films, nanostructures, organic materials and CNT (SWCNT, MWCNT, doped CNT);

- use of graphene to develop new nano-based devices;
- use of polymers and organic materials to develop new nano-based devices;
- use of AFM to measure the magnetic properties of nanostructures, for example, bits on hard disc;
- development and improvement of magnetoeletronic sensors using nanotechnological effects for higher sensitivity; and
- improvement of magnetoeletronic devices.

#### Semiconductor and Quantum electronics

The activities involved in the investigation of the electrical quantum triangle are related to the fabrication of small, precise junctions, i.e., Josephson junction (JJ) and quantum Hall effect (QHE), small nanostructures (single electron transistor (SET)). These are more related to quantised states and quantum metrology than to electrical nanometrology. The activities related to semiconductor fabrication and the challenges defined in the roadmap (ITRS) are more involved with nano-fabrication and application of dimensional nanometrology, thin film metrology and surface chemical analysis, than electrical nanometrology<sup>34</sup>.

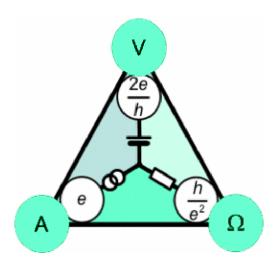


Figure 3 Electrical quantum triangle showing the relations of Ohm, ampere and voltage to fundamental constants

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<sup>&</sup>lt;sup>34</sup> See Co-Nanomet Consultative Document published at <a href="www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>

Due to the rapid development of both fields and the limits related to used and well-known materials, for example, silicon and silicon oxide, there is a strong need to investigate developments of new materials such as high- and low-k index materials, and ultra thin films as dielectric materials. Future limits end at electronic devices of molecular or atomic scale.

## Molecular or CNT based electronics

Molecular electronics uses the conduction, switching and storing of information in single molecules. Here, extremely high integration densities are achievable, which can partly be combined with new concepts, for example, error redundant circuits. Molecular electronics, however, is still the subject of basic research, for which the investigation of doped single wall carbon nanotubes (SWCNTs) and multi-wall CNTs (MWCNTs), polymers and organic materials, for example, OLEDs (Organic Light-Emitting Diodes), is necessary. Specifically graphene, the 'wonder material', has a very large potential, and not only for electrical applications and devices.<sup>35</sup>

# Magnetoelectronic Sensors

In order to enhance the reliability of mechatronic systems and non-contact magnetic field sensors, the use of the magneto resistive effect is increasingly applied. The measuring principle of a magnetic field sensor is the transformation of a position or movement information of a magnet into an electric signal, which is applied in nearly all hard disks. Here, the Giant Magneto-Resistive Effect (GMR) is utilised. The German scientists P. Grünberg and A. Fert were awarded the Nobel Prize in Physics for this in 2007. The GMR-effect occurs only in extremely thin layers and their magnetisation is a challenge for nanometrology. In the case of magnetic materials, like structures on a hard disc, the limit of the structures is becoming smaller and smaller. For a quantitative analysis of such small structures, the magnetic scanning force microscope is an essential tool. For precise and accurate measurements of magnetic structures, it is necessary to apply dimensional metrology for the structure parameters. However, the quantitative determination of magnetic forces is still a big challenge.

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<sup>&</sup>lt;sup>35</sup> A.K. Geim, Graphene: Status and Prospects, Science 324 p. 1530

# Magnetoelectronics/Spintronics

Another effect occurring in very thin films is the resistance change of ferromagnetic thin-layer systems depending on the relative direction of either their magnetisation or one of the external magnetic fields - the basis of all physical effects used in magnetoelectronics. Different sensor types and components are already commercially available that utilise this effect.

# E.6.2 Vision for the next ten years

Smaller, faster, bigger is the motto here. The sizes of the structures for semiconductor devices will be decreased in the next ten years to 16 nm nodes<sup>36</sup>.

As in the past, metrology requirements continue to be driven by advanced lithography processes and new materials, as well as the materials, structures and devices for 'Beyond CMOS'. Despite some problems related to isolated lines, CD-SEM and scatterometry will continue to be the key potential solution for in-process manufacturing control, whereas CD-AFM will be the key method for reference measurements. Referenced structures are necessary to validate scatterometry. AFM will also be well suited for line-edge and line-width roughness measurements.

Related to metrology, CD-AFM will be used to calibrate such small structures, where scatterometry could be the technique used during the process.

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<sup>&</sup>lt;sup>36</sup> ITRS 2009 Edition - Metrology

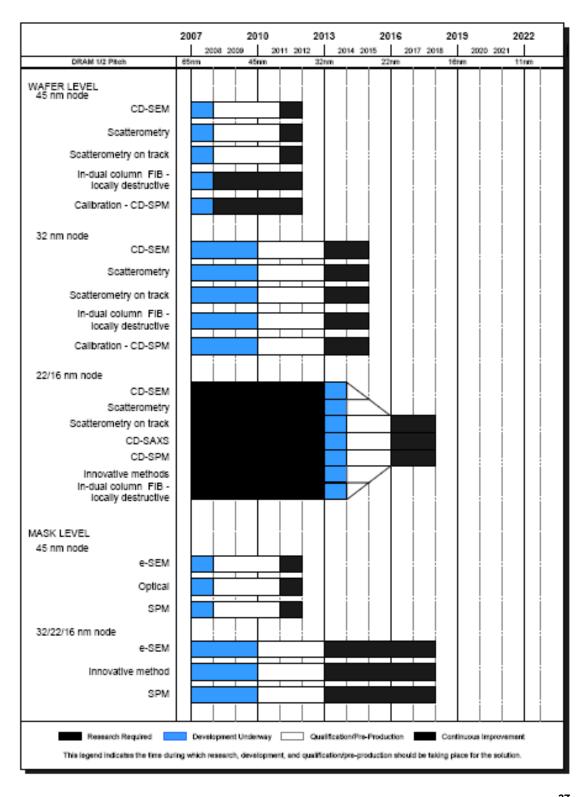
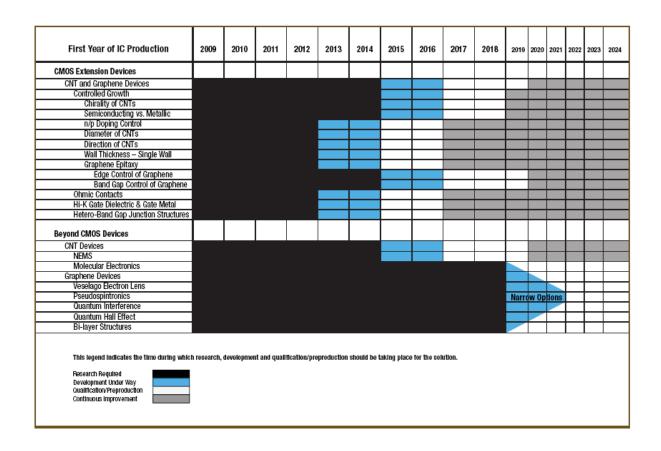


Figure 4 ITRS Roadmap 2009 Lithography Metrology Potential Solutions: CD<sup>37</sup>

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<sup>&</sup>lt;sup>37</sup> ITRS 2009 Edition - Metrology

However, metrology is needed for some other processes, such as front-end process (realisation of high-k materials and measurements, dual work function metal gates, improved starting materials, new ultra shallow junctions doping processes), interconnects (realisation of low-k materials, porosity measurements and control, barrier thickness, layer thickness, 3D interconnects and related metrology) and for 'Beyond CMOS' metrology (R&D of a large variety of materials as potential replacements for the transistor - graphene, complex metal oxides), measurements of critical electrical and magnetic interface properties (QHE at high mobility, spintronic materials: local characterisation of spin by BEEM (ballistic electron emission microscopy) and of magnetic properties by SEMPA (SEM with polarisation analysis)). Graphene layers need to be increased in size by new or improved techniques (for example, chemical vapour deposition - CVD) with better properties (electronic properties, low number of vacancies and broken bonds, improved knowledge on edge orientation and its impact on properties), improved low-ohmic contacts and the extension to applications of multi-layer systems.



<sup>&</sup>lt;sup>38</sup> A.C. Diebold, ITRS chapter: Metrology, Future Fab 32, Jan. 2010, p. 109

Table 2 R&D schedule proposed for carbon-based nanoelectronics to impact the industry's timetable for scaling information processing technologies<sup>39</sup>

There is an increase in the number of investigations in the areas of CNT and graphene, however, an overview and a future perspective are beyond the scope of the Co-Nanomet project.

Nanometrology comes into play for the research and development of semiconductor and quantum electronic devices as part of the necessary nano-fabrication in the form of:

- dimensional nanometrology;
- thin film metrology; and
- chemical nanometrology or surface chemical analysis.

All of these fields will have a role to play in successful R&D and fabrication of the electronic devices.

# E.7 Biological nanometrology

## E.7.1 Overview

The convergence of nanoscience, nanotechnology, biology and medicine - here referred to as "nanobio" — is probably the most rapidly evolving area in nanotechnology. It encompasses the use of nanotechnologies and nano-enabled approaches for studying and measuring biological systems, and for diagnosing, monitoring, treating and curing diseases in patients. In parallel to the strong knowledge and technology development in the area, there is rapid industrial growth by companies manufacturing and selling new devices, instrumentation, drugs and services. However, from a metrology point of view this area is in a much earlier development phase than any of the other areas discussed in this strategy. The reasons for this are partly associated with the novelty of the field and the complexity of biological systems, but can also be attributed to insufficiencies regarding stakeholder contacts, infrastructure and funding schemes. In order to reach its full potential the area will need the support of an improved metrological infrastructure.

 $<sup>^{39}</sup>$  J.A. Hutchby, ITRS Chapter: Emerging Research Devices, Future Fab. 32, Jan 2010, p. 29

# E.7.2 Vision for the next ten years

The future impact of nanotechnology in biology and the life sciences is only just becoming apparent. In the next ten years it can be envisaged that a wide range of nano-enabled drugs, products, diagnostic methods and treatments will be realised and successfully used in hospital clinics, leading to tremendous advances in the health care sector and improved quality of life for an ageing population. In parallel, techniques have been developed that provide researchers in life sciences with new tools for studying biological structures and processes at the nanoscale, where biomolecules are active.

Before any future visions in the area can be realised several metrological challenges need to be solved, including:

- non-invasive imaging and analysis of living systems at <10 nm spatial resolution;
- increased resolution in measurements of specific functions of biomolecules, for example, activity and binding forces;
- reliable, yet easy to use and cheap, diagnostic methods for specific physiological conditions or diseases, with improved speed, sensitivity and selectivity;
- defining the relevant measurands and developing techniques and protocols necessary for understanding the role of nanoscale particles and nanoscale material structures for toxicity and biocompatibility; and
- developing relevant regulations and standards for ensuring the quality, efficacy and safety of new nano-enabled methods and products.

Addressing the above challenges in an efficient manner is not only about solving scientific and technological problems. It will require increased collaboration and dissemination between stakeholders in the field, educational efforts and new funding schemes.

## E.7.3 Action plan

#### Short term

A successful translation of enabling nanotechnologies and measurement techniques to applications in the biomedical field and clinical practice is a challenging task that requires intense collaboration across disciplines and organisations. Much of the current development in this area is technology driven (rather than need driven), and there is low awareness of the importance of metrology aspects among many of the actors developing the technologies. The latter is also the case regarding regulatory aspects, which are of utmost importance for bringing new products all the way to clinical practice and the market. Meeting the regulatory requirements is in turn based on documentation of the properties of the products according to standardised methods that often involve the use of RMs. In many cases appropriate standards and RMs are lacking.

Important activities in the short term are to raise the metrological status of newly developed and emerging measurement techniques. This means development of appropriate RMs and establishing measurement best practice, specification standards and traceability to relevant measurement standards. Examples of RMs that need to be developed are: surfaces with controlled biomolecule coverage, nanoparticles with defined size and shape distribution, biomolecule reference formulations for comparison of different diagnostic techniques, and biological dimensional standards for imaging techniques.

Several imaging and analysis techniques originally developed for non-biological applications have promising potential in the nanobio area. New biological sample preparation techniques for vacuum based measurements need to be developed for achieving the full potential of these techniques.

To raise the general metrological status of the field, dissemination of metrology knowledge to players in the area is an important task for the NMIs. This should be carried out with the development and offering of courses on nanometrology. There is also a need to increase the awareness of regulatory aspects and identify the needs

of appropriate standards and RMs. The standardisation activities of relevant ISO working groups, as well the work on biological RMs carried out by WHO (World Health Organization), EDQM (European Directorate for the Quality of Medicines) and IRMM (Institute for Reference Materials and Measurements) should be involved in these efforts.

# Medium term and long term

Important measurement needs and challenges in life sciences to address in the medium and long term should be defined in close dialogue with the end-users, i.e. the life science community and industry. Such challenges include, for example, reliable quantitative measurements of specific markers for early detection or monitoring of wide-spread diseases, synthesis and characterisation of carriers for targeted drug delivery, quantitative characterisation of surfaces for biosensing or medical implants, and bioimaging technologies with high spatial and temporal resolution at ambient conditions.

One particularly important aspect of devices and materials used in health care is biocompatibility and toxicity. Current methods and standards for assessing these properties need to be further developed and adopted for new emerging nano-enabled products. This will require underpinning research efforts to obtain increased understanding of the role of nanoscale material properties for biological response at different levels, and for establishing relevant measurands for assessing biocompatibility and toxicity.

#### E.7.4 Infrastructure needs

Whereas several training programmes combining nanotechnology and life sciences are now established throughout Europe, there is a lack of individuals with in-depth knowledge of metrology in this field. Similarly, metrologists rarely have academic training in the life science disciplines. New educational programmes and courses are needed to provide academia, NMIs and industry with personnel who have the right combination of competencies for this new emerging area. Taking the example from other interdisciplinary areas such as biomaterials, courses of the type *Metrology for biologists* and *Biomedicine for metrologists* could be developed.

European and national research funding programmes are important resources for supporting the development of specific technological areas. In order to facilitate the necessary cross-disciplinary and cross-organisational efforts that are required for successfully advancing the nanobio area, there is a need for new funding schemes encouraging, for example, NMIs to seek collaboration with academic research groups, companies and other stakeholders from life sciences, and vice versa, for academic researchers to seek collaboration with NMIs. Centres of excellence with long-term funding (six to ten years), of the type that exist in several European countries in different technology areas, can be an appropriate way to implement this with NMIs as host organisations. The EMRP, which is now primarily aimed at NMIs, could in a future implementation have stronger requirements and provisions for involving and funding also non-NMI participants. Part of such funding schemes could also be to provide support for exchange of personnel between different NMIs and between NMIs, universities and industry.

Like any other area of modern research, the nanobio area has needs of advanced and costly instrumentation and laboratory resources. It is not realistic that all NMIs in Europe will have the necessary infrastructure to carry out leading activities in this field. It seems, therefore, appropriate that NMIs coordinate their activities in the field, specialise in particular subfields and develop schemes for task and equipment sharing for making the most efficient use of the resources available in Europe. NMIs could also benefit from increasing their use of large-scale facilities (for example, synchrotron radiation sources and neutron sources) that are today mainly used by academic researchers.

As mentioned above, contacts and collaborations across disciplines and organisations are important for advancing this field. NMIs could encourage such contacts by providing meeting places where relevant stakeholders can discuss needs and opportunities in the area.

#### E.7.5 R&D strategies

Currently the nanobio field is very active with strong developments of different nanoenabled products and measurement techniques. In contrast, in terms of metrology the field is much less developed. To advance the metrological aspects of all the technologies that are currently under development would be a huge effort. Strategically it seems appropriate that the techniques that are most highly developed and offering the highest biomedical and industrial impact are selected as case studies for focused metrological development efforts. Examples of techniques in this category are electron microscopy techniques for imaging, imaging mass spectrometry techniques, optical imaging techniques (for example, Raman and fluorescence microscopy techniques) and nanoparticle based biodiagnostics and therapeutics. Metrological efforts around these techniques should involve development of suitable RMs, best measurement practice, specification standards and establishing traceability to relevant measurement standards.

For future developments, a research strategy in this area that maximises the possibilities for reaching all the way from laboratory to market and clinical practice needs to be based on a holistic view, i.e. taking into account the entire innovation chain. Instead of the technology driven approach that prevails today, future efforts should be more driven by needs among the end-users in the health care sector (i.e. clinics and patients) and also take into account regulatory and metrology aspects, as well as commercial and market considerations, in the early stages of development. This means that additional aspects to academic or scientific/technological should be used as evaluation criteria for funding, and that projects by necessity will be interdisciplinary, involving partners from different stakeholder groups.

# E.7.6 Emerging topics and priorities

From a scientific point of view, two research topics of generic interest and of high priority can be mentioned. The first concerns the interaction between biosystems (from the biomolecule level to entire organisms) and nanoparticles or nanostructured materials. Knowledge in this area is largely lacking, and will be of key importance to developments of new devices such as biosensors, diagnostic devices and implants,

as well as for understanding toxicity of nanoparticles. A second topic of high priority and wide relevance is quantification, distribution and biological activity of biomolecules and other substances in biological systems. New concepts for measuring and imaging need to be developed to implement the next generations of biodiagnostic tools.

An important emerging topic that needs to be addressed in this field is public awareness. This is already an area of concern in food applications, and will most likely also be so in applications within the health care sector.

## E.7.7 SWOT analysis

## Strengths

Europe holds a strong global position in most of the scientific and technological disciplines required to advance the field, and has well established NMIs with excellent track records in the more traditional metrology areas. European industry is also among the leaders in several segments of the market, with several world-leading manufacturers of instrumentation for the nanobio area.

#### **Weaknesses**

An overall strategy for efficiently bringing research results and technological advances into useful products and the market is presently lacking. Metrology aspects and standardisation sometimes lag behind the development of new technologies and concepts, which may be an obstacle for further development and market introduction.

The area needs stronger interaction between the metrology and life science communities, as well as other stakeholders (industry, health care sector and regulatory authorities).

#### **Opportunities**

The area has a huge potential, both in terms of industrial development and benefits for individuals. Provided that proper strategies are implemented, Europe has the capabilities necessary to achieve a leading position when it comes to translating research and knowledge in the field into products and clinical applications.

The experiences and excellent track record of European NMIs from building metrology systems in other areas provides an opportunity for raising the metrological status of the nanobio area in an effective manner in collaboration with other stakeholders.

#### Threats

Europe faces strong competition from other countries that are investing strongly in the area.

## E.7.8 Summary

Nanometrology in biology is a rapidly developing area with expected impact on industrial sectors such as health care, food industry and agriculture. The area is rapidly evolving, with intense knowledge and technology development and strong industrial growth. To reach its full potential, the area needs a better developed metrology infrastructure as support. A strategy to achieve this and consisting of the following main components is proposed:

- focused metrology efforts around the current or emerging techniques that are most highly developed and offering the highest biomedical and industrial impact;
- future development efforts focusing on needs defined by end-users, and also taking into account regulatory, commercial and market considerations;
- underpinning research aimed at understanding the role of nanoscale material properties on biological responses, in order to identify relevant measurands for assessing biocompatibility and toxicity;
- new funding schemes encouraging collaboration projects between NMIs, industry and end-users; and
- educational efforts to raise the knowledge of metrology among other players in the field, and vice versa, the level of biomedical competence among metrologists.

# E.8 Modelling and simulations for nanometrology

#### E.8.1 Overview

Nanometrology, defined as science of measurements at the nanoscale, provides measurements that characterise processes and product performance and covers instrumentation and standards. Advances in nanometrology depend on understanding the properties of matter at the nanoscale, quality of measuring instruments, and the requirements of the industry involved in production of nanomaterials. Production of nanomaterials and nanometrology are key factors for achieving the promise of nanotechnology.

Nanomanufacturing (NM) aims at special material properties and processing capabilities at the nanoscale, and promotes integration of nanostructures to functional micro- and nanodevices, as well as the interfacing issues across dimensional scales. Nanometrology provides methods to measure and characterise process and product performance and includes topics such as instrumentation, measurement approaches for off-line and in-process production applications, and standards. Models and computer simulations assist in designing new modes of measurements by giving an insight into background physical processes. Various techniques are implemented depending on space-time scale - Figure 5.

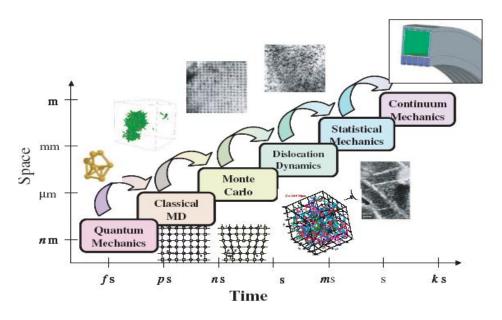


Figure 5 Models and Computer Simulations

Calculations in a given space-time region should be transferred into the neighbouring region with care because some of the object properties do not scale.

Modelling and Simulation is a discipline for developing a level of understanding of the interaction of the parts of a system, and of the system as a whole. The level of understanding that may be developed via this discipline is seldom achievable via any other discipline. To properly simulate the behaviour of a system of interest, both the experimental set-up and the theoretical model should allow reasonable changes to meet convergence requirements when the experimental and theoretical results are being compared<sup>40</sup>. As nanomaterials exhibit different properties from their bulk counterpart, due to their low dimensionality - (0D) quantum dots, (1D) carbon nanotubes, (2D) thin-film-multilayers, or due to a specific composition - complex structures, the techniques of measurements and modelling are classified in two general classes: for particle characterisation and for processes. All techniques should respect the quantum nature of the materials and the processes at the nanoscale. To design realistic models one needs reference data for the size and shape of nanomaterials, structure (inner and outer), aspect ratio, conductivity, magnetic properties, morphology and topography. These are provided by precise measurements. Although resolution of about 0.1 nm is achievable by using independent methods including tuneable and stabilised lasers, Fabry-Pérot interferometry and laser interferometry, the measurement uncertainty is in the range of a few nanometres due to non-linearity of the laser interferometer, alignment of the displacement axis and interferometer axis.

That is why inherent (systematic) sources of errors must be analysed in the case of all measurement techniques. For example, AFM sources of errors have been summarised<sup>41,42</sup>. Some errors can be reduced by introducing modelling<sup>43</sup> that presents a finite element model (FEM) for simulating the mechanical behaviour of

<sup>&</sup>lt;sup>40</sup> Ana Proykova, 2010 Challenges of Computations at the Nanoscale, *Journal of Computational and Theoretical Nanoscience* 7, 1806-1813

<sup>&</sup>lt;sup>41</sup> Danzebrink et al. 2006 Advances in scanning force microscopy for dimensional metrology, *Ann. CIRP* 55, 2

<sup>&</sup>lt;sup>42</sup> A. Yacoot et al. 2008 Aspects of scanning force microscopes and their effects on dimensional measurement, *J. Phys. D: Appl. Phys.* 41, 103001

<sup>&</sup>lt;sup>43</sup> Shaw C. Feng, Theodore V. Vorburger, Che bong Joung, Ronald G. Dixson, Joseph Fu, Li Ma, 2008 Computational Models of a Nano Probe Tip for Static Behaviors, *Scanning*, 30, 47-55

AFM cantilevers with carbon nanotubes attached. Spring constants of both the nanotube and cantilever in two directions are calculated using the finite element method with known Young's moduli of both silicon and multiwall nanotube as input data. Compliance of the nanotube-attached AFM probe tip may be calculated from the set of spring constants. Simulations of various AFMs can be performed using free software<sup>44</sup>. The commercial 'Nanosurf® easyScan control software' simulates an STM and takes full control of the microscope's functions to acquire real-time data<sup>45</sup>. Another commercial software is SPIP™ (Scanning Probe Image Processor) that allows a wide variety of functions - from 3D movie-making to batch filters<sup>46</sup>.

The most frequently used simulation techniques in nanometrology are large-scale finite element methods, multiscale Green's function methods, classical atomistic simulations (special purpose Monte Carlo Methods, Molecular Dynamics), *ab initio* quantum mechanical calculations and spin or/and time-dependent density functional theory. Each technique possesses both advantages and limitations from the point of view of nanometrology.

The finite element method converts the conditions of equilibrium into a set of linear algebraic equations for the nodal displacements. Once the equations are solved, one can find the actual strains and stresses in all the elements. By breaking the structure into a larger number of smaller elements, the stresses become closer to achieving equilibrium with the applied loads. Therefore, an important concept in the use of finite element methods is that, in general, a finite element model approaches the true solution to the problem only as the element density is increased. In spite of the significant advances that have been made in developing finite element packages, the results obtained must be carefully examined before they can be used. The most significant limitation of finite element methods is that the accuracy of the obtained solution is usually a function of the mesh resolution. Any regions of highly concentrated stress, such as around loading points and supports, must be carefully analysed with the use of a sufficiently refined mesh. In addition, there are some

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<sup>&</sup>lt;sup>44</sup> Some available here: <u>www.nanoscience.com/education/software.html</u>

<sup>45</sup> 46 www.imagemet.com/

problems that are inherently singular (the stresses are theoretically infinite). AFM data are used to generate an FEM mesh for the simulations.

Scatterometry is the investigation of micro- or nanostructured surfaces regarding their geometry and dimension by measurement and analysis of light diffraction from these surfaces. In contrast to optical methods, non-imaging metrology methods like scatterometry are not diffraction limited. They give access to the geometrical parameters of periodic structures like structure width (CD), pitch, side-wall angle or line height. However, scatterometry requires *apriori* information about the surface structure. The inverse diffraction problem has to be solved to determine the structure parameters from a measured diffraction pattern. Proper scatterometric measurements require an intense effort regarding modelling, simulation and inverse methods.

Density functional theory (DFT) transformed theoretical chemistry, surface science, and materials physics and has created a new ability to describe the electronic structure and interatomic forces in molecules with hundreds and sometimes thousands of atoms. A wide range of theoretical and computational methods to study electrons in nanostructures and their interaction with external fields and light are based on (DFT) <sup>47</sup>:

- Kohn-Sham DFT for ground-state total energy calculations, structure determination and potential energy surfaces for atomic motion;
- time-dependent DFT for study of systems excited out of the ground state, for example, optical absorption and Many Body Perturbation Theory (MBPT);
- GW and GWΓ self-energy approaches for electron addition and removal energies, spectral functions, total energy;
- Bethe-Salpeter approach for neutral electronic excitations;
- Non-equilibrium Green's function theories for Quantum Transport and Combined DFT-MBPT approaches;
- Generalised Kohn-Sham DFT for total energy calculations, incorporating elements of DFT and MBPT;
- GWF self-energy, incorporating Density Functional concepts;

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- TDDFT approaches for quantum transport; and
- TDDFT approaches for total energy calculations, via the fluctuation-dissipation theorem.

Molecular Dynamics (MD) with fast multi-pole methods for computing long-range interatomic forces has made accurate calculations possible on the dynamics of millions and sometimes billions of atoms. When combined with the DFT calculations it is used instead of the classical convolution approach to tip–sample artefacts that is not valid for measurements of nanospecimens due to the quantum-mechanical nature of small objects. As interatomic forces act on the sample and the tip of the microscope, the atoms of both relax in order to reach equilibrium positions. This leads to changes in those quantities that are finally interpreted as the AFM tip position and influences the resultant dimensional measurements. Sources of uncertainty connected with tip–surface relaxation at the atomic level have been discussed<sup>48</sup>. Results of both density functional theory modelling and classical molecular dynamics of AFM scans on typical systems used in nanometrology, for example, fullerenes and carbon nanotubes, on highly oriented pyrolytic graphite substrates are presented. Effects of tip–surface relaxation on critical measurements of the dimensions of these objects are also studied.

Monte Carlo modelling of SEM image formation has been used to generate artificial SEM images or signal profiles for the development and testing of new CD evaluation algorithms. Therefore, the generated images have to be close enough to real SEM images to allow us to transfer results of a CD evaluation at a simulated image to real SEM measurements. The electron diffusion in the sample and the excitation and emission of secondary electrons are simulated by Monte Carlo routines. The central part of the simulation procedure is the iterative (i.e. stepwise) calculation of electron trajectories in solid state<sup>49</sup>.

Time dependent Monte Carlo method simulates processes occurring in a threephase batch reactor working at isobar and isotherm conditions. It calculates the dose

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<sup>&</sup>lt;sup>48</sup> Anna Campbellová, Petr Klapetek and Miroslav Valtr, 2009 Tip-sample relaxation as a source of uncertainty in nanoscale scanning probe microscopy measurements, *Meas. Sci. Technol.* 20, 084014 <sup>49</sup> C G Frase, D Gnieser and H Bosse 2009 Model-based SEM for dimensional metrology tasks in semiconductor and mask industry, *J. Phys. D: Appl. Phys.* 42, 183001

in time-dependent geometry; the results of three-dimensional calculations are usually performed separately and combined. This approach becomes cumbersome when high temporal resolution is required, if the geometry is complex, or if interplay effects between different, independently moving systems are to be studied. Standards in energy deposition can be established by implementing this technique. Quantum Monte Carlo methods now promise to provide nearly exact descriptions of the electronic structures of molecules.

WKB models, direct tunnelling (Simmons model) and field emission tunnelling (Fowler-Nordhaim tunnelling) could be used to model conductivity in single molecular structures at low and elevated bias. Potentially, Simmons model could extract two molecular barriers, one for electrons and one for holes from conductivity spectra. Following this assumption, electrical and optical gap-probed molecular nanometrology (GMN) could be developed. The main GMN principle is the small difference between the values of the HOMO-LUMO energy gap detected by electrical and optical measurements. A comparison of experimentally derived electrical and optical probed gap and energy offsets between EF and nearest molecular orbital makes this approach feasible and applicable.

# E.8.2 Vision for the next ten years

If the final objective of modelling in nanometrology would be standardisation of drug delivery systems for health care then the computational community and instrument developers should:

- produce control devices and instruments to administer therapeutically useful compounds suggested by in-silico drug design;
- invent simulation methods and modelling techniques for toxicological and ecotoxicological research to systematically view exposure, dose and response, cellular mechanism and long-lasting effects;
- develop new hybrid techniques of known ones;
- simulate measurement tasks obtained by a device under variation of internal (probe, electronic) or external parameters (temperature, vibration, ...);

- estimate measurement uncertainties (virtual Instrument) in ambient conditions;
- assess toxicity of nanomaterials; and
- develop new methodology for governance of emerging nanotechnology.

# E.8.3 Action plan

To achieve the objectives listed in the previous section over the ten year period, research groups at the universities and research institutes, including national metrology institutes, need to cooperate with high-tech companies and the regulatory bodies including health and environmental watchdog groups.

It seems likely that fundamentally new mathematics will be needed:

- to bridge electronic through macroscopic length and time scales;
- to determine the essential science of transport mechanisms at the nanoscale;
- to devise theoretical and simulation approaches to study nanointerfaces, which dominate nanoscale systems and are necessarily highly complex and heterogeneous;
- to simulate with reasonable accuracy the optical properties of nanoscale structures; and
- to model nanoscale opto-electronic devices.

#### Short term

- the existing network EURAMET to contact universities with established graduate (Masters & PhD) programmes in Nanoscience and Nanotechnology (within 3-6 months).
- establishment of a new network (12 months); and
- deliverables of such contacts to be software enriched with features specific for nanometrology (24 months)

#### Medium term

expansion of the new network with business partners and regulatory bodies

(18-24 months);

- dissemination of the achievements via international conferences, info-days and electronic media (continuous); and
- deliverables commercial & free simulation packages 'easy for usage' (36-60 months)

#### Long term

- validation of the software developed by the NMI;
- incorporated packages into metrological devices (producers of devices); and
- continuous education of scientists to use the new techniques (universities and research institutes).

Here, a citation is helpful:

"If misuses (...of models) are to stop and if modeling is to become a rational tool of the general public, rather than remaining the special magic of a technical priesthood, a basic understanding of models must become more widespread" - Dr. John D. Sterman, Director of the MIT System Dynamics Group and Professor of Management Science at the Sloan School of Management, Massachusetts Institute of Technology, 1991.

#### E.8.4 Infrastructure needs

To this end, a transparent and global vision of European needs has been developed in the form of a roadmap for research infrastructure in Europe for the next ten to twenty years. The elaboration of this scientific roadmap has been entrusted to the 'European Strategy Forum on Research Infrastructures' (ESFRI). Distributed infrastructures, as defined by the European Strategy Forum on research Infrastructures in its last roadmap<sup>50</sup>, will be very useful for the purposes of nanometrology, as every country has already established local computational premises.

<sup>&</sup>lt;sup>50</sup> <u>ftp://ftp.cordis.europa.eu/pub/esfri/docs/esfri\_roadmap\_update\_2008.pdf</u>

The tools of theory have advanced as much as the experimental tools in nanoscience over the past fifteen years. It has been a true revolution by increased computer power. The rise of fast workstations, cluster computing, and new generations of massively parallel computers complete the picture of the transformation in theory, modelling, and simulation over the last decade and a half. Moreover, these hardware (and basic software) tools are continuing on the Moore's Law exponential trajectory of improvement, doubling the computing power available on single chips every 18 months. Computational Grids are emerging as the next logical extension of cluster and parallel computing.

From a modelling and simulation point of view, it is necessary to ensure access of the computational nanometrology community to the already existing highperformance computing centres, such as HPC-Europa<sup>51</sup>.

The main problem is that the software companies in the field are too small, with limited economical resources, and the NMIs do not have, in general, groups involved in nanomaterial modelling. This should be overcome by specific measures.

A good practice is the recently created distributed network, The European Theoretical Spectroscopy Facility (ETSF), which offers theoretical calculations and open source software code to help interpret experimental data. It is a virtual facility that supplies services, support and information to experimental and industrial users<sup>52</sup>.

The nanometrology community involved in modelling and simulations could create a similar network with the help of national and European funds.

#### E.8.5 R&D strategies

To fulfil the needs of the manufacturing community it is important that research on the upscale of nanotechnology for high rate production, reliability, efficiency and cost issues for nanoproducts is pursued. To accomplish this, new research directions

 $<sup>^{51}</sup>_{52} \frac{\text{www.hpc-europa.eu/}}{\text{www.etsf.eu/}}$ 

must involve a systems approach that encompasses nanoscale materials and structures, fabrication and integration processes, production equipment and characterisation of instrumentation, theory/modelling/simulation and control tools, biomimetic design, three dimensional nanoscale metrology, production-hardened metrology and other areas driven by industrial applications.

In a sense this is the mission of PRINS (Pan-European Research Infrastructure for Nano-Structures)<sup>53</sup>, established by three scientific partners CEA-LETI (France), IMEC (Belgium) – coordinator, Fraunhofer Group of Electronics (Germany), four industrial partners and the Flemish Government. More countries are expected to join in the future.

# E.8.6 Emerging topics and priorities

Broadly speaking, modelling and simulation techniques in nanometrology will continue to span the spectrum, from the development of new methods (longer-term, enabling research) to innovative applications of known methods to new instruments.

Application-oriented topics will require corresponding models for:

- nanotechnologies for efficient energy storage;
- compounds for drug delivery;
- nanorobots; and
- nanophotonics, including spintronics.

#### New research directions:

- modelling that encompasses nanoscale materials and structures;
- simulation of integration processes;
- design of novel equipment for nanometrology;
- modelling of open quantum systems, such as those encountered in nanometrology;

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<sup>53</sup> www.prins-online.eu/index.php?id=2

- integration of multi-scale functional systems;
- simulation of three dimensional nanoscale metrology, production-hardened metrology and other areas driven by industrial applications;
- advances in extensibility and portability of the software; and
- validation and verification of the modelling codes.

# Priorities:

faster quantum computations with a temperature control

# E.8.7 SWOT analysis

#### Strengths

Deep understanding of the quantum processes and their influence on precise measurements; Europe is a leader in scientific research

#### <u>Weaknesses</u>

Lack of interaction between the large high-tech companies (producers of devices) and the software developers; limited interaction between the regulatory bodies and universities

#### **Opportunities**

New educational programmes available and exchange programmes from university to business; cooperative efforts between EC-FP7 and National Science Funds on materials, and device development and modelling for direct thermal-to-electrical energy conversion should be undertaken in areas of thermoelectric, thermophotovoltaics, and thermionics.

## **Threats**

Delayed transfer of new techniques suitable for nanometrology from the universities and research institutes to manufacturers. The rate of transfer is much higher (in some cases within a year) in the US, Japan and China.

# E.8.8 Summary

With the advance of new manufacturing processes and close tolerance designs, the quality control and improved evaluation methods have an increasing role. In general, quality departments are investing in various methods for inspections of products. These are ranging from optical microscopes to high-end scanning electron microscopes (SEM).

Computational simulation is riding a hardware and software wave that has led to revolutionary advances in many fields represented by both continuous and discrete models.

There is a need for SME's to use easy packages – this is a challenge to be met by the developers in the near future. Another need is to embed modelling into industrial collaborative projects at national and European level, as funding authorities are unlikely to call modelling topics in isolation except for certain things, for example nanotoxicology, where the field is more academic. Effort is to be put into development of new data mining processes and procedures to cope with the sheer amount of data that can now be generated. The companies are convinced that modelling is good for the sustainability agenda as it reduces the amount of expensive experimentation for industry and academia alike.

#### In brief, modelling:

- improves the productivity of the development team (for example, models can be used for semi-automatic code-generation);
- reduces the number of defects in the final code (models facilitate early evaluation of the system);
- captures and organises the understanding of the system. Models are well suited for documentation;
- permits the early exploration of alternatives; and
- facilitates the reuse of parts of the system in new projects.

As the development of a European Strategy for Nanometrology must exist within the context of global activity in the field, international efforts will be reviewed in each of the above activities and reflected within the strategy work.

# **ANNEX F – Infrastructure**

## F.1 Overview

The development of infrastructures – that is, strongly integrated resources either at a specific site (for example, CERN) or in a distributed network – in innovation, education and research were particularly emphasised, both by experts and the general public, in a recent public consultation: "Towards a Strategic Nanotechnology Action Plan (SNAP) 2010-2015"<sup>54</sup>. The same study emphasised the need for EU policies in the new Action Plan to do more, in particularly: (a) developing education & training (b) developing better tools; (c) active communication and (d) international cooperation.

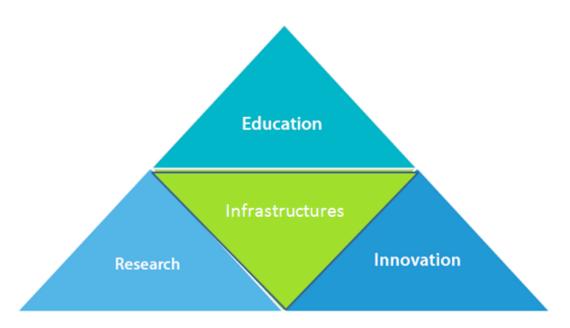


Figure 6 Infrastructures positioned within the knowledge triangle, adapted 55

Infrastructures in general lie at the heart of the knowledge triangle – the beneficial combination of research activity, specialised education and innovation that advances

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<sup>&</sup>lt;sup>54</sup> Report on the European Commission's Public Online Consultation, TOWARDS A STRATEGIC NANOTECHNOLOGY ACTION PLAN (SNAP) 2010-2015 ec.europa.eu/research/consultations/snap/report\_en.pdf

our knowledge and understanding across all scientific and societal domains, see Figure 6.

A proposed infrastructure for nanometrology contains (see section F.2):

- Instrumentation hubs;
- Centres of Excellence (CoE); and
- Centres of Dissemination (CoD)

National metrology institutes typically will form part of CoEs.

In the following we will relate this nanometrology infrastructure to the knowledge triangle.

#### F.1.1 Innovation infrastructures

It is widely acknowledged that the industrial exploitation of nanotechnology in innovative products is still lagging, despite advances in nanoscience.

In order to sustain the expected considerable growth in nanoproducts, major investment in technological and manufacturing/business infrastructure is needed<sup>55</sup>. This includes metrology and standards that can be used at the nanoscale in support of conformity assessment of nanoproducts of all kinds.

Developments in nanotechnology place special demands on the required metrological infrastructure, while at the same time, the rate of nanotechnological development continues to increase in both volume and complexity. Demands are more extensive than can be provided by individual actors, simply because all stages of the innovation process have to be covered at the same time as nanotechnology is rapidly developing. A strengthening is needed of the existing network of European measurement facilities as well as investments in the development and introduction of

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<sup>&</sup>lt;sup>55</sup> A vision for strengthening world-class research infrastructures in the ERA, Report of the Expert Group on Research Infrastructures, ISBN: 978-92-79-14214-7 (2010)

new standards and measurement techniques, as part of the necessary infrastructure to support growth in nanotechnology. Few such standards and techniques exist today that can be used across the entire nanoscale range (1 nm to 100 nm) and existing measurement techniques and instrumentation will have to be developed further as whole new ranges of nanotechnological products appear.

The nanometrology infrastructure approaches innovation in three ways:

- 1) The Centres of Excellence form the backbone of the instrumental validation that is required for metrologically supervised innovation process. Here, new instrumentation will be developed and characterised at the highest level of nanometrology. The CoEs can also advise the stakeholders regarding nanometrology for innovative production lines as well as application of new standards.
- 2) The Centres of Dissemination spread the newest advances in nanometrology and standardisation. This is essential for any innovation process. At the same time, the CoDs also keep in touch with the stakeholders in order to register any new instrumentation available for a nanometrological characterisation. The CoDs also provide support for the stakeholders with regards to questions concerning the implementation of new standards.
- 3) The Instrumental Hubs implement new techniques and allow for their innovative application. Here the stakeholders can learn about new techniques. It is essential that these be strengthened, as they assist the stakeholders to implement new instrumentation at the production sites. The Instrumental Hubs also have the facilities and the potential to invent new instrumentation that better suits the emerging measurement requirements.

This triad can also be found again in the triangle of instrumentation, metrology and standardisation, see Figure 7.

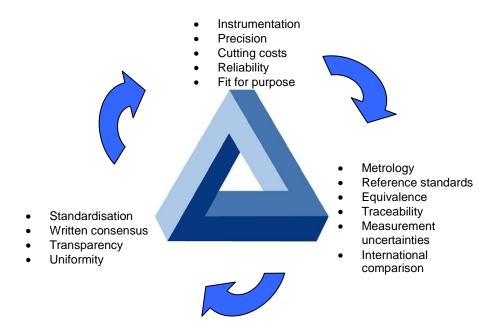


Figure 7 Triangle of standardisation, instrumentation and metrology, adapted<sup>56</sup>

This figure graphically visualises the interdependencies between state-of-the-art instrumentation, their metrological characterisation and the written agreement on how to implement a uniform, transparent and inter-comparable measurement strategy for the instruments.

#### F.1.2 Education infrastructures

One main aim of the Co-Nanomet project is to bring nanotechnology through to successful business by developing as part of the general dissemination of traceable nanometrology to the workplace:

- relevant metrology tools; and
- suitably skilled human resources able to implement appropriately such tools.

 $<sup>^{56}</sup>$  Gennesys white paper, ISBN 978-3-00-027338-4 (2009)

A relook and strengthening of a European educational infrastructure is needed in support of nanotechnological innovation through better measurements on the nanoscale.

With this in mind, one key observation is to place (nano) metrology in the right context, so that when people in the workplace have to choose the relevant metrology tools and human resources, they can relate product requirements – often in terms of specifications for conformity assessment – to the corresponding metrological requirements.

There are several means of disseminating nanometrology to the workplace. For example, new training material – in the form of courses, perhaps on the Internet (for example, webinars), and compendia – is being developed<sup>57</sup>. This is the task of the Centres of Dissemination CoDs, supported by the NMIs.

The understanding of basic metrology concepts is limited:

Beyond the NMIs, a solid understanding of the basic metrology concepts is not sufficiently well spread. This is the case for many industry branches, and results from an underexposure of pupils and students to these concepts, even at the academic, university level, where interest in the metrology subject is limited.

In making the Consultation of the Co-Nanomet task (4.3)<sup>58</sup>, additional studies have been made of:

- programmes using local networks aimed at encouraging small to medium sized enterprises to exploit the opportunities offered by, for example, new micro- and nanotechnologies; and
- development of written standards (norms) as pedagogical material, for example, guidelines.

www.co-nanomet.eu/page1014/CoNanomet-Home/Coordination-Of-Education-And-Skills

<sup>&</sup>lt;sup>58</sup> Co-Nanomet European Consultation on Metrological Traceability, Standards and Dissemination of Metrology in Industrial Nanotechnology 2010

#### F.1.3 Research infrastructures

At the national metrology level in Europe, there is already an extensive and increasing coordination of national metrological research, particularly thanks to the European Metrology Research Programme (EMRP)<sup>59</sup> implemented on behalf of the European Union by EURAMET. Nanometrological research is already included in a number of ERA-NET+ iMERAplus projects in the first phase of the EMRP. As part of the major Art185 (former Art169) EMRP, there are plenty of opportunities for the formulation of new research projects in nanometrology, for example, EMRP New Technologies Call 2011. This will have strong impact on the future structure of the NMI and their responsibility. For the future, in areas such as nanometrology, an opening up of EMRP to wider researcher and stakeholder communities would be beneficial. The competitive process of application inherently leads to a formation of Centres of Excellence within the metrological fields. The CoEs will focus the research on their fields and interact with the stakeholders in order to define nanometrological requirements and register emerging needs.

In addition to this, new multidisciplinary collaborative actions should be promoted, both for stimulating scalable application development for various nanometrological domains and for developing efficient middleware addressing outstanding issues with regard to measurement reliability, traceability, mutual recognition and further related topics within the fields of nanometrology.

In addition to EURAMET, during the Sixth Framework Programme (FP6), funding of research within nanotechnology infrastructures amounted to 40 M €<sup>60</sup>. The European Strategy Forum on Research Infrastructures (ESFRI) introduced support to Design Studies for new research infrastructures of clear European dimension and interest. A roadmap for pan-European research infrastructures was also published<sup>61</sup>.

<sup>&</sup>lt;sup>59</sup> www.emrponline.eu

<sup>&</sup>lt;sup>61</sup> ESFRI European Strategy Forum on Research Infrastructures European Roadmap for Research Infrastructures Implementation Report 2009 ISBN: 978-92-79-14602-2

Major upgrades of existing infrastructures were included, and the end result is intended to be equivalent to a new pan-European research infrastructure. All fields of science and technologies were considered. Design Studies have thus provided, during FP6, the technical tools to address the conceptual design of new facilities in a bottom up fashion. In all cases, however, this instrument is not sufficient, nor designed, to produce a fully costed, production-ready, detailed engineering design, and further commitment to advanced R&D is still required from the stakeholders.

Discussions are currently taking place within the European Commission to organise the support to selected ESFRI projects, after the successful completion of the Preparatory Phase. Such support under FP7 could take place under the budgetary year 2011 or 2012 and might be related, for example, to the strengthening of the "eco-system" of research infrastructures in specific fields. Additionally, further strategic support to the implementation of projects with substantial international cooperation could be given. Here an option presents itself for supporting the development of a comprehensive nanometrological infrastructure.

#### Funding of infrastructures F.1.4

The processes of identifying, funding, designing, developing, constructing, managing and sharing infrastructures for innovation, education and research are complex and costly. Yet the efficient and timely realisation of all these processes is vital to the healthy development and more rapid implementation of the infrastructures. The Expert group of the European Research Area (ERA) of the Commission has recently (in 2010) published a report to provide recommendations for developments and improvements of Research Infrastructures<sup>62</sup>.

In addition to addressing the needs of large-scale radiation, the white paper<sup>63</sup> of the "Grand European initiative on Nanoscience and Nanotechnology using Neutron- and Synchrotron radiation Sources" (GENNESYS) also comments on the need for nanometrology research and technology. In the foreword it is stated: "Europe needs

<sup>&</sup>lt;sup>62</sup> A vision for strengthening world-class research infrastructures in the ERA, Report of the Expert Group on Research Infrastructures, ISBN: 978-92-79-14214-7 (2010)

<sup>&</sup>lt;sup>63</sup> Gennesys whitepaper, ISBN 978-3-00-027338-4 (2009)

bright scientists, supported by efficient research infrastructures, to deliver on the promise of nanotechnology."

### F.1.5 Nanotechnological large-scale infrastructures in Europe

Amongst the European research infrastructures are the large-scale or singular facilities, scientific instruments, distributed facilities and interconnected networks, funded by Member States and supported by Community budget of the European Union, and shared widely within and between scientific research communities.

This is also valid for the infrastructures of nanotechnology. Most of the existing infrastructures are not primarily addressing nanometrology. A recent Co-Nanomet assessment has located more than 500 European measurement facilities and instrumentations. They are listed in an online accessible database<sup>64</sup>, where they can be sorted and selected with respect to their measurement capability and location.

The facilities comprise national metrology institutes (NMIs) and public, as well as private, non-governmental organisations (NGO) - see Table 3 below. The facilities can be categorised as follows:

- GOV national or regional government funded, primary function research activities;
- UNI national or regional government funded, primary function education activities;
- RTO privately funded (not-for-profit), primary function research activities;
- SME privately funded, profit making activities, <250 employees; and
- LE privately funded, profit making activities, >250 employees.

<sup>64</sup> search.co-nanomet.eu/

Country	<u>Facilities</u>	Comment	
EC States			
Austria	10	Mix of UNI and GOV capability supported by strategic initiative Austrian Nano <sup>65</sup>	
Belgium	14	Mix of RTO, GOV and UNI capability	
Bulgaria	6	All GOV institutes from Bulgarian Academy of Sciences <sup>66</sup>	
Cyprus	1	One UNI only	
Czech Republic	22	Chiefly GOV Academy of Science <sup>67</sup> centres, supported by strategic action Nanotechnology.cz <sup>68</sup>	
Denmark	9	Blend of UNI and RTO capability supported by the initiative NaNet <sup>69</sup>	
Estonia	3	Major UNIs and one GOV centre	
Finland	16	Broad spectrum of capability across UNI, RTO and GOV sectors supported by strategic initiative FinNano <sup>70</sup>	
France	59	Many mixed UNI/GOV centres with national support from CNRS	
Germany	125	Capabilities found in many GOV, RTO, LE and SME some technology mapping available <sup>71</sup>	
Greece	7	All GOV institutes	
Hungary	7	Mostly institutes from the Hungarian Academy of Sciences <sup>72</sup>	
Ireland	8	All capability UNI based	
Italy	36	Mix of mainly GOV, UNI and RTO supported by strategic initiative Nanotec IT <sup>73</sup>	
Latvia	2	Major UNIs only	
Lithuania	5	Major UNIs and GOV centres	
Luxembourg	2	Two RTO only	
Malta	1	One UNI has some capability	
Netherlands	16	Significant LE and SME capacity focused towards industrial access. Strategic support action NanoNed <sup>74</sup>	
Poland	23	Some UNI, mainly GOV Polish Academy of Sciences institutes <sup>75</sup>	
Portugal	8	Mostly UNI capability	
Romania	6	All GOV institutes of Romanian Academy of Sciences <sup>76</sup>	
Slovakia	9	Mostly UNI and branches of Slovak Academy of Sciences 77	
Slovenia	4	Mostly GOV institutes	
Spain	35	Mix of centres, strategic support through NanoSpain <sup>78</sup>	
Sweden	14	Significant RTO capability matching UNI sector	
United Kingdom	63	Mix of centres with strategic national initiatives <sup>79</sup>	
EU TOTAL	511		

www.nanoinitiative.at/
www.bas.bg/
www.cas.cz/
www.nanotechnology.cz/
www.nanet.nu/
www.aka.fi/en-gb/A/Science-in-society/Research-programmes/Ongoing/FinNano/
www.nano-map.de/
www.nano-map.de/
www.nanotec.it/
www.nanoned.nl/
www.nanoned.nl/
www.english.pan.pl/
www.acad.ro/def2002eng.htm
www.nanospain.org/nanospain\_English.htm
www.innovateuk.org/deliveringinnovation/micronanotechnologycentres.ashx

Table 3 Brief summary of identified nanometrology capabilities, continues on next page.

AC States				
Croatia	2	GOV institutes		
Iceland	1	Only one UNI has capability		
Israel	10	UNI and significant SME capability supported by strategic initiative NanoIsrael <sup>80</sup>		
Liechtenstein	0	No capability, no NMI		
Macedonia	0	No capability		
Norway	7	Capability largely with UNI some RTO		
Switzerland	16	Mostly UNI and GOV, supported by SNI initiative <sup>81</sup>		
Turkey	7	Mix of UNI and GOV centres		
AC Total	43			

Table 3 Brief summary of identified nanometrology capabilities, continued from previous page

The most significant contributions in terms of volume of facilities are apparent: Germany, United Kingdom, France, Italy and Spain account for more than 50 % of the facilities available in total. On the other hand, a number of states (Cyprus, Estonia, Latvia, Luxembourg, Malta, Slovenia, Croatia, Iceland, Liechtenstein and Macedonia) individually contribute less than 1 % to the overall capability.

The division of the nanometrology capability between the five categories is illustrated in Figure 8.

<sup>81</sup> www.nanoisrael.org/ www.nccr-nano.org/nccr/

#### **Total Facilities**

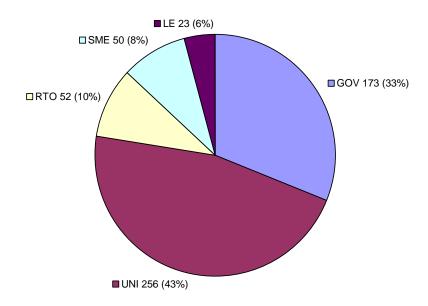


Figure 8 Contribution of different facility types to overall nanometrology capability

It is evident that public sector facilities form the core (76 %) of the nanometrology capacity available in the selected states. The remaining contribution is provided by the private sector, largely in the form of not-for-profit RTO organisations undertaking contract research. The profit-focused SME and LE sectors provide a significant contribution and two important types of relevant activity can be noted within this sector:

- manufacture of nanometrology systems; and
- contract metrology services

It can also be noted that within the SME sector, the majority of organisations featuring in this study are spin-out companies associated with universities. Such entities are only included in this study where nanometrology capacity is not owned by or situated within the university in question.

It is a challenge to integrate these nanotechnological infrastructures in order to strengthen nanometrology by installing activities that address the key concepts of metrology. The main advantage would be the development of measurement techniques and investigations related to measurement uncertainty and traceability to the international system of units: SI. As a consequence, the uncertainty of measurement could be evaluated and the measurement results would become intercomparable amongst the facilities, and mutually recognisable. First then, a nanotechnology facility becomes a nanometrological facility. For certain measurement tasks, however, a stable reproducibility of the measurements is sufficient, and the traceability is not immediately required. See further information on metrology in Section F.2.

For example, today only a few measurement methods are fully validated for application at the nanoscale. A method can only be used to produce reliable measurement results after proper validation in the laboratory that wishes to apply the method. Due to the novelty of the area, the metrological traceability systems and the laboratory QA tools required to validate methods, to establish laboratory proficiency, and to achieve traceable results, are not currently available.

A general list of metrological calibration and measurement capabilities can be accessed and searched online in the Key Comparison Database (KCDB) at the website of the International Bureau of Weights and Measures (BIPM) <sup>82</sup>. While this database undoubtedly holds a large amount of valuable information for the metrology community, when the focus is upon the nanoscale, the CMC is less useful in mapping capabilities. Aside from those NMIs of selected member states, members of the BIPM are not, in the main, focused upon the development of research-driven nanometrology techniques. It is likely that the future will see significant adjustment of this position through strategic partnerships or other means, as nanometrology becomes increasingly significant in legislative terms.

In summary, the BIPM database offers some information concerning nanometrology capabilities across the EC, but only within its member and associate organisations, thus capturing only a fraction of the capacity and capabilities that are actually available within research and other organisations throughout the EC. This is illustrated in Table 4, where NMI capabilities in the sub 100 nm dimensional

<sup>82</sup> www.bipm.org

measurement realm, as documented by the BIPM, are summarised for states encompassed by the capability mapping undertaken during this project.

YES	NO
Finland, France, Germany, Italy, Netherlands, Poland,	Austria, Belgium, Bulgaria, Croatia, Cyprus, Estonia, Greece, Hungary, Iceland, Ireland, Israel, Latvia, Liechtenstein, Lithuania, Luxembourg, Macedonia, Malta, Portugal, Norway, Slovakia, Slovenia

Table 4 NMI coverage of nanometrology capability (for example, surface texture, roundness, line spacing) from BIPM data

It should be noted that while the BIPM website features regularly updated news items concerning the developing capabilities of member organisations, much of the information contained within the CMC database itself dates back as far as 2001. Clearly, an update of structured information available through the BIPM to potential nanometrology users would be of benefit. For this purpose, BIPM has been approached by the Co-Nanomet consortium to implement the option of filtering the database with respect to the keyword 'nano'. This is currently being considered by BIPM.

The Consultation of the Co-Nanomet task  $(4.3)^{83}$  on Metrological Traceability, Standards and Dissemination of Metrology in Industrial Nanotechnology 2010 has identified a number of future needs in these areas.

## F.2 Vision for the next 10 years

Most nanometrology infrastructures are currently by the NMIs, since traceability to units at the nanoscale is primarily established at the highest national level.

<sup>&</sup>lt;sup>83</sup> CO-NANOMET European Consultation on Metrological Traceability, Standards and Dissemination of Metrology in Industrial Nanotechnology 2010

In addition to this, other nanotechnology infrastructures have been identified<sup>84</sup>. Most of them are funded by Member States and/or supported by European Community budget and the private sector. While the existing infrastructures must be strengthened, for example, by cooperation in the EMRP, the establishment of traceability for relevant nanotechnology facilities is required in order to enable intercomparability of measurement results and their mutual agreement. Appropriate methods are calibration by transfer standards such as reference materials, or by direct instrumental comparison with a Pole of Excellence in the relevant field.

Regarding training and education within nanometrology, it will be taking advantage of Marie Curie initial training networks (ITNs)<sup>85</sup>. The EMRP already reserves 10 % of its budget (40 M€) for researcher excellence and mobility grants, including specifically non-NMI actors. These aim to improve early-stage researchers' career prospects in both the public and private sectors. This will be achieved through a transnational networking mechanism, aimed at structuring the existing high-quality initial research training capacity throughout Member States and Associated Countries.

Complementary to the EMRP, which strengthens the technological part of the nanometrology infrastructure, the impact of ITN to the infrastructure is focused on the transfer and distribution of knowledge. ITN can advantageously be used to:

- strengthen interdisciplinary and international cooperation;
- initialise the transfer of knowledge;
- train PhD. students, preserving and potentially increasing knowledge; and
- bring in non-NMIs and establish new contacts, thereby enlarging the infrastructure.

Any accomplishments of the infrastructure are to be visualised appropriately. This leads to common national and international level impact studies. It is envisioned that a board of external committees shall monitor and measure the impacts of infrastructures.

<sup>84</sup> search.co-nanomet.eu/

<sup>85</sup> cordis.europa.eu/fp7/people/initial-training\_en.html

Infrastructure must encompass not only scientific, but also technological developments. This will ensure that European stakeholders have access to:

- global traceable calibrations, including inter-laboratory comparisons;
- metrology assistance in research and development;
- sound basis for forming public opinion; and
- legislation that is based on proper standardisation and metrology.

### A proposed infrastructure consists of:

- Infrastructure Management
- Advisory board
- Instrumentation hubs
- Centres of Excellence
- Centres of Dissemination

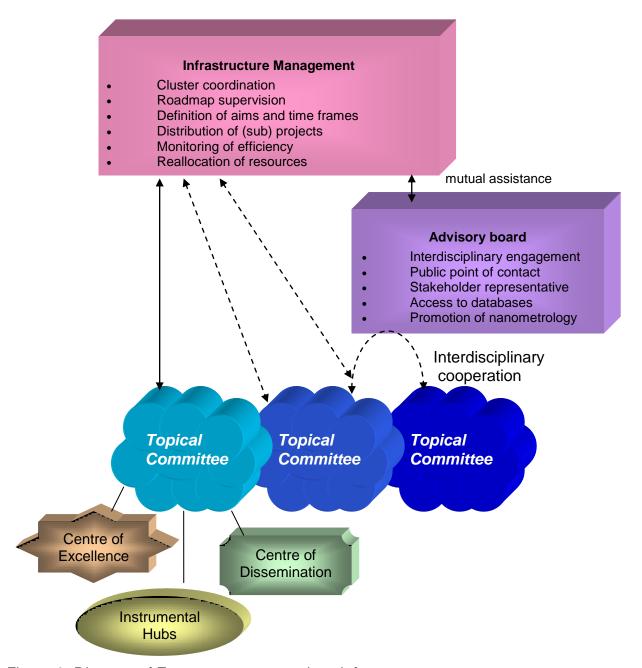


Figure 9 Diagram of European nanometrology infrastructure

Figure 9 summarises the structure and the actions point of section F.3 diagrammatically.

The strategy for the future infrastructure includes specific plans:

Methods to promote resource exchange and pooling among Member States should be piloted by the advisory board:

- concepts to utilise existing resources more efficiently by sharing critical or unique resources are to be worked out by the management;
- funding possibilities of infrastructures include Joint Programming including an extension of the EMRP; the development of Public-Private Partnerships;
   Joint Technology Initiatives and the use of ERA-NET+ and Article 185 instruments already covered by the EMRP of EURAMET; and
- calibration services that are already established or that will be developed in the further process can be used to attract customers in a free competition in Europe. This will improve the competences of the industry as well as demonstrate the created impact of the nanometrological infrastructure.

This leads to the following aims and action points regarding the nanometrology infrastructure.

### F.3 Action points

### Building a distributed infrastructure in nanometrology

- 1) To cover the large range of scientific disciplines involved in nanometrology and the diversity of application areas, a European infrastructure has to be built on a dedicated central management, which in turn integrates the clusters of regional Centres of Excellence, Centres of Dissemination and Coordinated Instrument Hubs. These Centres and Hubs should have world-class facilities and expertise within their field of nanometrology, as well as high levels of engagement between industry and academia.
- 2) The Management of the European nanometrology infrastructure, as described in this section, connects and coordinates the nanometrology committees to share knowledge and equipment, even interdisciplinary where necessary, focusing on results and assisting in the definition of the aims. It guides the distribution of nanometrology projects within the infrastructure, allocating fixed periods of time to the individual tasks. The Management monitors the critical mass of the committees, and proactively engages the infrastructure development by dynamically reallocating the affiliation of institutions within the committees. The infrastructure can be reduced

if necessary where redundancies appear, saving resources. It can also be locally expanded where a solution to a problem requires the expertise of further nanometrology facilities. An expansion of the infrastructure can be accomplished, for example, by promoting the development of more industrial-oriented research facilities, or via greater involvement of the private sector. These means allow an efficient and resource efficient usage of the infrastructure, increasing the response time to actual tasks.

It is recommended that the management, as described here, should be embedded in the EURAMET e.V. organisation to make efficient use of an already well-organised and established metrology service network.

3) In order to help the management in executing its functions, an infrastructure advisory board is set up. It improves the engagement between academic measurement disciplines, research centres and companies inside and between the involved committees. This helps problem solving within the infrastructure. Acting as a public point of contact as well as a switchboard, the advisory board relates and distributes metrology concepts, proposals or other requests from stakeholders and other parties of interest. If necessary, the advisory board also assists the efficiency of the infrastructure by subdividing larger, possibly interdisciplinary, projects.

It will be the duty of EURAMET to set up the appropriate infrastructure advisory board, both the structure and appropriate rules. The members of the advisory board should be from industry, academia, and NMIs, as well as from standardisation and regulatory bodies.

- 4) Existing technical roadmaps for each of the fields of nanometrology, such as developed by EURAMET TC-L, should be monitored and adjusted actively in order to provide a constantly updated catalyst for collaboration between industry and academia within the infrastructure.
- 5) Set-up and upgrading of the regionally distributed committees will require local, national and European political support and funding supplemented by private investments at a later, more mature stage.

### Communication and public engagement

- 6) The advisory board should also support the stakeholders, especially SMEs, to help articulate their needs and interests to regulatory and standardisation bodies.
- 7) The infrastructure should provide pools of experts and professional communication tools necessary for engagement with the public.
- 8) Promotion of the capabilities of nanometrology to stakeholders, SMEs and the public in general should be facilitated by showcasing examples of successful exploitation of nanobiotechnology.
- 9) Engagement of the European nanometrology infrastructure with research centres outside Europe should be encouraged.
- 10) Public access to nanometrology and its services must be established. This can be done by setting up a public 'infrastructure portal' that is globally accessible. It must be connected to the BIPM KCBD to allow an overview of existing services of nanometrology. The link to the KCDB must be direct, however, it should also be modified to make nanometrology services clearly visible from non-nanometrological services. The public portal should also link to the database of the "European cooperation for Accreditation" (EA) <sup>86</sup>, which lists accredited calibration services.

It should be the task of EURAMET to set up the described "infrastructure portal".

<sup>&</sup>lt;sup>86</sup> www.european-accreditation.org

### **Education and training**

- 11) The highly interdisciplinary nature of nanometrology requires the integration of dedicated academic nanotechnology modules, preferably at the MSc or PhD level. In order to promote metrology as such, education metrology should be part of academic lecturing.
- 12) Because nanometrology touches on many important wider issues especially within biological nanometrology an understanding of ethical and social aspects and training in science communication and public engagement should be taught at the MSc and PhD level.
- 13) Due to the rapid development in nanotechnology, targeted education and training programmes for in-career training need to be developed, with focus on metrological issues. This is perhaps closest to the main aim of the Co-Nanomet project to bring nanotechnology through to successful business with:
  - relevant metrology tools; and
  - suitably skilled human resources able to implement appropriately such tools

Several means of disseminating nanometrology to the workplace have been studied.

### F.4 SWOT analysis

#### **Strengths**

- metrology in Europe is already coordinated by EURAMET e.V., which is able
  to handle the task related to the establishment and operation of the proposed
  European nanometrology infrastructure; and
- nanotechnological facilities already exist.

### Weaknesses

- much of the current European nanotechnology instrumentation is not available for metrology purposes, i.e. it is not directly traceable, its measurement capability is not well characterised and it is not part of an inter-laboratory comparison programme; and
- stakeholder access to metrology equipment is limited.

### **Opportunities**

- to expand EMRP projects on nanometrology to establish the necessary nanometrology infrastructures; and
- to use available national and European funding, dedicated to infrastructures, to improve the nanometrology infrastructure.

### Threats

- the lack of public understanding of metrology in general;
- planning of activities mainly on regional level rather than on a pan-European level (lack of coordination on European-level); and
- the public fear of 'nano'. New innovative products do not receive the deserved acceptance, which in turn can hinder further advances in nanometrology.

### F.5 Summary

Regarding the current state of large-scale infrastructures and single facilities for nanometrology, the following can be summarised:

- Twenty out of thirty-four national metrology institutes have no visible nanometrology capability;
- Germany, United Kingdom, France, Italy and Spain account for more than 50 % of the total nanometrology facilities;
- more than 75 % of the nanometrology facilities are from within the public sector;
- the university research base is the single largest contributor to nanometrology capability;

- profit-focused organisations provide the clear majority of the private sector nanometrology contribution in France, Germany, Netherlands, United Kingdom and Israel;
- Government funded Academies of Science in Bulgaria, Czech Republic, Hungary, Poland, Romania and Slovakia provide significant nanometrology access; and
- most evidently in Austria, Germany, Italy and Spain, regional government support of research infrastructure has created significant nanometrology capability.

Despite the amount of available instrumentation, the number of registered nanometrological service providers is rather limited. Existing instrumentation, as well as newly developed measuring methods, requires metrological characterisation in order to achieve comparability and mutual recognition. In the field of nanometrology, this concerns instrumentation at potential Centres of Excellence as well as that implemented at stakeholders' production sites. Consequently, with an increasing amount of nanometrological services, the need for a global accessible database becomes more imminent for a structured overview. The KCDB of the BIPM is a good example of such a database. Additionally, such a database will facilitate the structuring of Instrumental Hubs, which form one base of the nanometrology infrastructure alongside the Centres of Excellence and Centres of Dissemination. An advisory board will act as point-of-contact for the stakeholders.

### **ANNEX G - Education and skills**

#### **G.1** Overview

In line with the rapid development associated with nanotechnology, training needs within nanometrology will also be under constant development. Having defined a set of key skills required within the field, these would then be mapped on to a matrix of knowledge/skill level against skill type, as a basis on which to plan the future training provision. For a given technical area, the matrix defines the level of skill required, varying from an awareness of concepts only, at one extreme, to the application of specialist knowledge, at the other. Against this, the stage at which such training should be applied is defined, for example, training delivery to degree level students. This model then differentiates between those areas that should be delivered through the education system and those that should be delivered through a vocational training route.

Qualification profiles contain characteristics of knowledge, skills and faculties that are prerequisites for certain jobs in nanometrology. They cover further education, the development of intermediate qualifications, academic degrees and higher degrees.

The current scope of research activities in nanotechnology is the reason for the high demand for personnel with high-level university degrees. The demand for staff with qualifications below university level is comparatively low at present. Nevertheless, there are studies that record cases of a lack of qualified staff below university level<sup>87</sup>.

Employees with qualifications below university level need particular interdisciplinary knowledge and strong social competences to take part in cooperation and innovation processes in the enterprises<sup>88</sup>.

<sup>87</sup> 'Identification of skill needs in nanotechnology', Lothar Abitch, Henriette Freikamp, Uwe Schumann; Cedefop Panorama series; 120. Office for Official Publications of the European Communities, 2006, Luxembourg.

<sup>88</sup> 'Approaching the labour market in the European Educational Environment', Laura Malita, Catalin Martin; 2010, Unversity of Timisoara.

The construction of qualification profiles should be modular. A corresponding acknowledgement of degrees, at the international level, can be guaranteed by the use of a reliable credit-transfer system for academic recognition, for example, the European credit transfer system (ECTS), allowing transparency and recognition of vocational education and training.

### Structure of the professional qualifications system<sup>89</sup>

The proposed structure consists of professional qualifications arranged by level of qualification. In a first approach, it is necessary to divide the qualifications into different levels depending on the final job. The three levels of professional qualification are based on the professional competency required for each productive activity, taking into account different criteria like knowledge, initiative, autonomy, responsibility and complexity, amongst others, necessary for the accomplishment of each activity.

Qualification Levels: The management of the skill levels is conducted on the basis of the professional competence required in the production systems, according to criteria of the activity to develop as knowledge, initiative, autonomy, responsibility, and complexity.

#### Level I: Vocational training and company employees

This level includes low-level employees who have a basic understanding of the occupation through education or experience. They perform routine or moderately complex tasks that require limited exercise of judgement and provide experience and familiarisation with the employer's methods, practices and programmes. They may assist staff in performing tasks that require skills equivalent to a level II (see below) and may perform higher-level work for training and development purposes. These employees work under close supervision and receive specific instructions on required tasks and the results expected. Work is closely monitored and reviewed for accuracy.

<sup>&</sup>lt;sup>89</sup> National Institute of Qualifications (INCUAL), Ministry of Education, Government of Spain, www.educacion.es/iceextranet/

### Level II: Degree level or high laboratory experience

Able to work independently on individual projects and ultimately to manage project groups, they can write reports and clearly document experimental work. At this level, employees need to be able to gather and analyse relevant information from a wide variety of sources and identify and propose solutions to problems. Besides this, they will also have technical and computing skills.

### Level III: Requires an advanced degree (Masters or PhD)

Fully competent employees who have a sufficient level of experience in the role to plan and conduct work requiring judgment and independent evaluation, selection, modification and application of standard procedures and techniques. These employees use advanced skills and diversified knowledge to solve unusual and complex problems. They may supervise or provide direction to staff performing tasks requiring skills equivalent to level II and level I. These employees will receive only technical guidance and their work will be reviewed for effectiveness in meeting the establishment procedures and expectations.

### The professional qualification definition:

A Professional Qualification (PQ) is defined as a set of professional competences that can be acquired through vocational education and training modules, or any other kind of learning structure, as well as through work experience. It is understood that a person is qualified when he or she achieves the expected outcome during his or her professional performance, with reasonable resources and quality levels. From a formal point of view, a qualification is a group of competences (knowledge and capabilities) that satisfy occupations and job posts in the labour market.

The competence of one person comprises the whole range of personal, professional and academic knowledge and capabilities of that person, acquired following different paths and at all levels available.

The Professional Qualification structure:

Each qualification has a general competence that defines briefly the employee's essential tasks and functions.

Other elements are also described, including the professional environment in which the qualification takes place, the corresponding productive sectors and the relevant occupations or posts that can be accessed with that qualification.

Every qualification consists of Competence Units (CU). The competence unit is the minimum set of professional competences that can be partially recognised and accredited.

Every competence unit is linked to a learning module that describes the necessary learning to acquire that particular competence unit.

This structure facilitates the assessment and accreditation of every competence unit acquired by an employee, both through work experience and non-formal or informal learning. Recognised and accredited competence units can be accumulated in order to obtain the accreditation of a qualification.

Each competence unit will have a standardised format, which includes its identification information and the specifications of that competence.

The competence units are divided into Professional Performances (PP). These establish the expected behaviour of one person, i.e. the expected consequences or results of the activities performed by that person. They help to show whether a person is competent in a competence unit.

The Performance Criteria (PC) express the acceptable level of one professional performance to meet the productive organisation's targets and act as reference guides for the assessment of professional competences. The Professional Qualifications structure is shown in Figure 10.

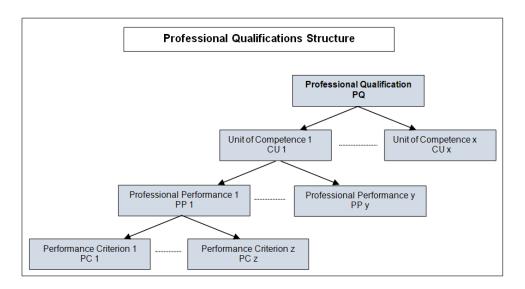


Figure 10 Professional qualifications structure

### The associated skills training

Each competence unit is associated with a learning module, which describes the necessary learning required to acquire that competence unit.

A Training Module (TM) is a coherent set of training associated with the units of competence that make up skills. It is the smallest unit of training to determine the diplomas and professional certificates.

Training specifications contain Professional Skills (PS), the expression of the expected outcomes of peoples' learning situations at the end of the training module.

The Evaluation Criteria (EC) is a set of details for each capacity that indicates the degree of detail acceptable to it. They define the scope and level of ability and the context in which it is to be evaluated. The Training Modules structure is shown in Figure 11 and the relationship between competences and educational skills is described in Figure 12.

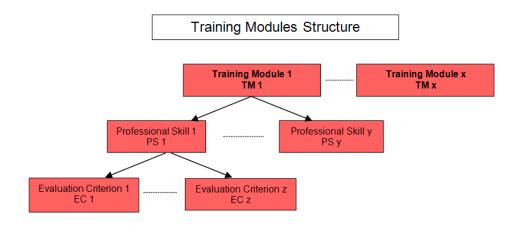


Figure 11 Training modules structure

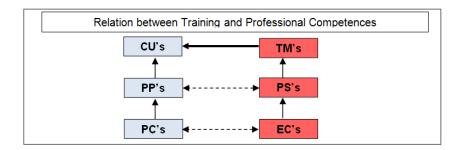


Figure 12 Relationships between training and professional competences

### G.2 Vision for the next ten years

Over the next ten years, it will be necessary to consolidate the professional qualifications and the general training structure in order to balance the training offer and implement the new professional skills into the nanotechnology market, while avoiding duplicities in nanometrology training programmes.

On the other hand, it is very important to disseminate the importance of metrology at the nanoscale, by including it in degree level subjects.

Industry should be involved, both from metrology and nanotechnology, with collaborative training projects between NMIs and universities, and with employee-level recognition.

### G.3 Action plan

#### Short term

- review the professional qualifications with respect to the professional levels.
   Once the first drafts have been developed, an expert group in each area should evaluate them in order to clearly relate the professional qualifications to the necessary level to develop each job position;
- validate experienced employees. It is very important to involve employees with experience, not only from the standpoint of motivation but also the recognition of their knowledge and work in the company; and
- start up the model with training modules as a validation plan. Once
  educational goals for a professional qualification are set, the developed
  training modules should be implemented to verify suitability for the related job.
   This pilot phase will serve as validation of the overall training plan.

### Medium term and long term

- modular training consolidation. Once the first phase of implementation has been completed, the concept of modular training should be consolidated and extended to all related areas;
- increase training offer for generic metrology and metrology at the nanoscale.
   When the overall training plan is completed, it will be possible to adapt the training offer, whether university or non-university, to meet the needs in the field of nanometrology;
- introduce metrology in regular chemistry, physics, biology or engineering nanotechnology studies to reflect, at the educational level, the impact on both industry and current research; and
- create a steering committee or a group of experts that monitor the improvements in the professional field due to the change in the training structure.

### G.4 SWOT analysis

#### Strengths

- the NMIs and the European institutions are seriously involved, both individually and collectively;
- many universities across Europe are increasing their research in nanotechnology, due to the improvement of metrology instrumentation;
- many large companies and SMEs are investing in innovation and new technologies; and
- the investment in innovation and new technologies is increasing in large companies and also in SMEs, and not only through public funding.

### Weaknesses

- no real introduction to generic metrology in training programmes;
- constantly changing targets in nanometrology because the technology develops quickly; and
- each university or community has their own training programmes and educational system.

### **Opportunities**

• European projects and consortiums, like Co-Nanomet, can successfully influence the harmonisation of approaches in nanometrology.

### Threats

- the current economic situation may hamper the provision of public funds; and
- it is very important for institutions to work together for a common goal and not to get lost in competition for similar purposes.

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<sup>&</sup>lt;sup>i</sup> The clinically relevant levels of biomolecules vary over several orders of magnitude. In certain instances such levels of detection will be required.

iii Details of individual contributors may be seen through the multiple publications of the Co-Nanomet programme at <a href="www.euspen.eu/nanometrology">www.euspen.eu/nanometrology</a>



