Action in Support of the Federal Authority's Strategic Priorities

Science Metrology for Micro and Nanotechnologies

Final Report

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Issue	Date	Modifications
Draft	November 21, 2006	initial issue
1.0	December 15, 2006	- §3.2 Updates on NANO project + [RD11] conclusions - §5 & 6 Pie presentations - Additional § 6.4 on Materials, components most studied or measured - Additional contribution on §6.5 Cost impact Complement to the conclusions

Applicable Documents

[AD1] *Contrat de recherche N° AP2/M01*
[AD2] *Annexe 1 au Contrat de recherche N° AP2/M01*

Reference documents

[RD1]	*AP2/M01 Initial report*	*060621_Nanometrology_Initial Report*
[RD2]	*RP-CSL-NAM-06001*	*060621_Nanometrology_Initial Report FR*

[RD3] *RP-CSL-NAM-06002* *060821_Follow-up board Report*

[RD4] *MT/CSL/NAM/06001* *Initial follow-on board meeting*

[RD5] *MT/CSL/NAM/06002* *Minutes of meeting at LNE*

[RD6] *T.Dziomba, L. Koenders, G. Wilkening, Standardisation in dimensional nanometrology: development of a calibration guideline for Scanning Probe Microscopy. Optical fabrication testing and metrology II SPIE Vol 5965 JENA 2005 Germany*

[RD7] *L Koenders et al, Comparison on step height measurements in the nano and micrometre range by scanning force microscopes* Metrologia ***43*** *04001 2006*

[RD8] *K Doytchinov et al, International comparison of surface roughness and step height (depth) standards, SIML-S2 (SIM 4.8)* Metrologia ***43*** *04002 2006*

[RD9] *Blind, Knut; Gauch, Stephan: Frictions in the Interface between Nanotechnology Research and Standardisation in Germany. Explanations and Solutions. In: Coenen, Heide; Gröhndahl, Jörg; Jakobs, Kai (Eds.): Standardisation and Networks. Proceedings: of 11th EURAS Workshop on Standardisation. Hamburg, 8-9 June. Aachen: Wissenschaftsverlag Mainz in Aachen, 2006, S. 61-70 (Aachener Beiträge zur Informatik Bd. 38) (ISI-P-41-06*

[RD10] *Nanometrology Eighth nanoforum Report July 2006*

[RD11] Nanoscale Calibration Standards and Methods: Dimensional and Related Measurements in the Micro- and Nanometer Range Günter Wilkening, Ludger Koenders Wiley-VCH Veerlag GmbH 2005

[RD12] European Survey on Success Factors, barriers and Needs for the Industrial Uptake of Nonmaterials in SMEs, CEE Nanoraod SME report 2005

Table of content

1. Scope

This report presents the output of the study about the Belgian needs and status on nanometrology. It presents the study approach, the questionnaire used, the list of companies contacted, an analysis of the query and conclusions.

2. Introduction

The nanometrology marketplace is fully dependent on the gauges development. These later unambiguously define the technical terminology and also test procedures to determine sample characteristics.

It is of major importance to connect the standardization and research to make our companies competitive and at the top of the technology by interconnecting research centers and department in that field.

The project aims to establish an overview of nano metrology in Belgium, pointing out the techniques used by our companies, research centers and universities in this field. This work was achieved through information enquiry at the identified entities. This way gives indications of the demand in term of instrumental gauging. Survey is one possibility that has been proposed to determine the future needs of standard for the service sector.

In parallel, a study of the international state-of-the-art in the domain through an exhaustive bibliographic research (ISO and other standards, scientific journals, documents made available by foreign bureau of standards or other international organizations) was performed. However, this part is already well known at the SPF Economie (see [RD4]), but a summary is anyway provided hereafter.

3. International state of the art

3.1. Fields of nanometrology and used instruments

A general summary on application in nanotechnology can be found on http://cordis.europa.eu/nanotechnology/src/pressroom_films.htm, “Nanotechnology: Innovation for tomorrow's world"

It is well known that these technologies cover a wide field of applications (e.g. Optic, Integrated optics, Lithography, MOEMS, MEMS, Electronics, Coating, Powder, Biology, Precision engineering, Semiconductor, Material science, Crystallography, Chemistry, Life science, Nano engineering)

This study will be limited to Belgian applications.

3.2. Standardisation in nanometrology

[RD6] indicates that hardware transfers are already commercially available. However there is no general accepted procedure how to perform SPM calibration. VDI/VDE – GMA 3.41/3.43 and ISO are discussing guidelines procedures on these aspects.

SPMet European Network on the calibration of SPMs took an important part in the SPM calibration procedures within EC for precision engineering.

Through EUROMET a lot of round robins are performed. One [RD7] shows that step height measurements with SPM are in good agreement with the reference values. This implies that SPM measurements are reliable and suitable for traceable measurements of step heights. The study shows that today step heights on samples can be measured with uncertainties in the sub-nanometre range.

Between 1999 and 2002, several round robin were performed (NANO2 (step height), NANO3 (Line scale), NANO4 (1D grating). They were quite successful either the instruments were quite new. Another international comparison of surface roughness and step height (depth) standards has been performed [RD8] and points out that the laboratories agree on all of the measurements within their stated and published uncertainties.

However a lot of other types of instruments are also used (see equipment list proposed in the questionnaire). Some of them use laser light, and the laser wavelength act as a “standard”. These lasers are generally not stabilized. It is probably one of the reasons why nanometrology at the BIPM has been transferred to Time, Frequency and Gravimetry department.

The ISO-TC Training Committee 201 “Surface Chemical Analysis” standard is mainly driven for SPM applications in the field of chemistry, life science and crystallography.

ISO – TC229 (Initiated in May 2005 with a first meeting in November 2005 and the second June 2006). This group plan to initiate international standardization activities with the goal of contributing to facilitating nanotechnology development and utilization, as well as improving public understanding. It also decided to establish working groups (WGs) covering the three areas of 1) terminology and nomenclature; 2) measurement and characterization (including calibration and certification); and 3) health, safety and the environment. Up to now, no ISO standards have been published from this TC. The IBN (Institut Belge de Normalisation) is involved in this TC.

ANSI Nanotechnology Steering Panel attempts to cover nanotechnology standardization as a whole.

ASTM E56 Committee on Nanotechnology started also in 2005. This Committee addresses issues related to standards and guidance materials for nanotechnology & nanomaterials, as well as the coordination of existing ASTM standardization related to nanotechnology needs. This coordination includes the apportioning of specific requests for nanotechnology standards through ASTM's existing committee base, as well as the maintenance of appropriate global liaison relationships with activities (internal and external) related to this subject area. There are 6 working groups, one also on nomenclature, the other addresses more nanoparticule issues and carbone nanotubes.

Pr. Massalar member of the follow-on board is representative for central Europe of NSCL (National Conference of Standards Laboratories) and is member of IMEKO (International Measurement Confederation). NCSL and IMEKO are also concerned by nano metrology standards.

Finally, an entire book dedicated to nanoscale Calibration standard has been recently edited [RD11]. Since the authors are German, the book discusses mostly development and activities performed at the PTB. This book indicates that there is no standard process for microroughness and three dimensional surfaces measuring, and that there are generally no accepted guide lines. Optical and lithography techniques allows to manufacture grating down to 300 nm. It demonstrated that the topic is complex and confirms our first statement of this paragraph.

3.3. Activities in our neighbor countries and in the EC

Since the beginning of 2000, it appears obvious for several neighbor countries and at EC level that there is a need in nanotechnology to write standards, to define scientific instrumentation for measurement, to validate measurement procedures and to measure standards. EC starts a study in 2001 (EUROMET project). In GB this type of activities have also been proposed in 2002 with programs covering 2003 - 2005 (theme 4 of Science and technology in length metrology). In France, the nanometrology project starts in 1999 - 2000 under the demand of electronic industries.

Recently, final versions of the roadmap reports of NanoroadSME and NanoRoadMap were available. Both have elaborated roadmaps in the fields of energy, health and materials, the first with a focus on SMEs.

The roadmaps of **Nanoroad SME** (www.nanoroad.net) are as follows:

- Nanoroadmap Medical and Health (http://www.nanoroad.net/download/roadmap_mh.pdf)

- Nanoroadmap Energy (http://www.nanoroad.net/download/roadmap_e.pdf)

- Nanoroadmap Automotive Industry (http://www.nanoroad.net/download/roadmap_ai.pdf)

- Nanoroadmap Aeronautics Industry (http://www.nanoroad.net/download/roadmap_as.pdf)

SME surveys and SWOT analyses for these four areas have been done and can be downloaded from http://www.nanoroad.net/index.php?topic=download.

The roadmap reports of NanoRoadMap (www.nanoroadmap.it) are the following ones:

- 4 Materials roadmap reports (* Nanoporous * Nanoparticles/Nanocomposites * Dendrimers * Thin film & coatings).

- 4 Health and Medical Systems roadmap reports (* Drug encapsulation / Drug delivery / Drug targeting * Molecular Imaging / Biophotonics / Medical Imaging * Biochips / High-throughput screening / Lab-on-a-chip devices * Biomolecular sensors).

- 4 Energy roadmap reports (* Solar cells * Thermoelectricity * Rechargeable batteries and Supercapacitors * Heat insulation and conductance).

They can be downloaded in the section reports on the www.nanoroadmap.it homepage.

3.4. Conclusions

Since July 2006 a report on Nanometrololy is available on the web and gives a good overview of international state-of-the-art in nanometrology.

The main highlights are:

1. There is only one Belgium company pointed out working in nanometrology, while countries like The Netherlands, Danemark, Czeck Republic, Switzerland have several companies. The origin of the authors (NL, Cz) biased probably this list in someway. (pg20). It has to be noted that this Belgium Company was not aware of this.
2. They propose 6 categories for classifying nanostructures according dimensionality (pg 13)

   0 – D: Nanoparticules

   1 – D: Nanowires

   2 – D: Coating and surface layer

   3 – D: Bulk

   Powders

   Other Nanostructures
3. The overview of nanometrology initiatives (pg 17) are the one we already rise up in § 3.2.
4. Papers dedicated to nanometrology comprise about 7% of all the nanotechnology papers in the last 10 years. But nanotechnology is implied in a lot of areas, and nanometrology is indirectly present in nanotechnology papers. So we wouldn't take this as a negative point.
5. The report gives also a good overview and summary of the techniques used in nanometrology, however the selected methods are bias by the expertise of the authors.

Anyway, this interesting report can be downloaded from http://www.nanoforum.org/ in publication heading.

Miss U. Dobre member of the follow-on board indicates that at the SPF Economie they have large international collaboration and by this way have a good knowledge of the international status on nanometrology standard. It is accepted that it is not relevant to go in deeper analysis about this topic.

From informal discussions the Table 1 of the nanometrology report is also valuable for Belgium partner where the interest in nano metrology is different between Research oriented and industrial players.

Nanometrology	
Research-oriented	**Industrial**
Requires highest precision possible	Requires highest effectiveness of measuring systems
Vision driven observations mostly	Quantitative parameters need to be measured
Quantity of measured parameters – as many as possible	Quantity of measured parameters – minimum acceptable
Measurement time and cost – are not important	Measurement time and cost - minimum
Significant importance of ambient conditions	Measurement under conditions in a production process (rough conditions, affected by vibration, air pollution, etc.)

Table 1

4. Questionnaire

To scan the needs of the Belgium actors in the field of nanotechnology a questionnaire was set up. It has been discussed during the initial meeting [RD4] and updated. It is presented in annex 1.

The goal was to:

1) point out the most used instruments in the nanotechnology field,

2) verify if the users are concerned by the traceability of their measurements,

3) consider the financial impact of wrong measurements,

4) evaluate further needs in nanometrology.

To avoid that people spend too much time to fill the questionnaire in, the questionnaire was simplified as much as possible and addressed mainly the instruments that the users most handle.

5. Contacted companies

First of all, we cannot be sure that all the Belgium actors involved in nanotechnology have been contacted.

The way the companies were found was through workshops (e.g. Hainova symposium in Charleroi), web search, EU web information, and CSL contacts.

About 100 actors have been identified. Several were eliminated since they seemed not really concerned by measurements as user e.g. modeling lab, simulation, doubloons,…, or because they are not really implicated in nanotechnology.

After this first selection, about 50 players remain and were contacted by email, 50% replies and 40% fill actually the questionnaire. In total our analysis is based on 18 answers by researchers and responsibles in the field. The name of the contacted company, their coordinates and their fields of activities are presented in annex 2. It has to be noticed that some companies at first view which shall be concerned by nanometrology and standard didn't reply. Oral contacts were also performed to work out some of the points addressed in the questionnaire.

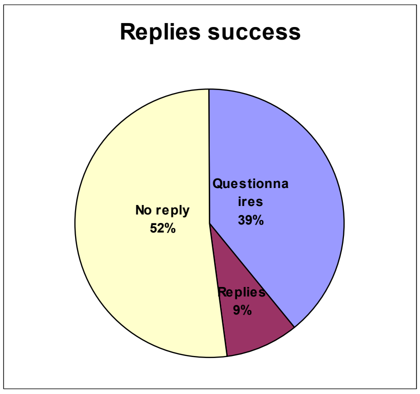

Fig 1 : Replies success

It is estimated that the sampling is large enough to provide a general overview of the nanometrology users in Belgium. The result is not so bad, compared to similar study in Germany [RD9] or at EC. In Germany a response rate of 28% was achieved. A total number of 65 questionnaires were filled. Half of the questionnaires were collected during two national conferences, opportunity that we didn't have in Belgium. Additionally, it is well known that Germany occupies a leading position in the field. At EC, 19% of replies were collected for the SME survey http://www.nanoroad.net/index.php?topic=download.

6. Questionnaire replies

6.1. Flemish/Wallonia-Brussels parity

The query is well balance between Flemish and Wallonia. The Large companies are not taken into account in this rating. The actual ratio was Flemish/Wallonia-Brussels = 82%. This does not indicate that the nanotechnology is more developed in Wallonia. A larger reply rate was encountered in the Flemish region while more entities were contacted in the Wallonia-Brussels region.

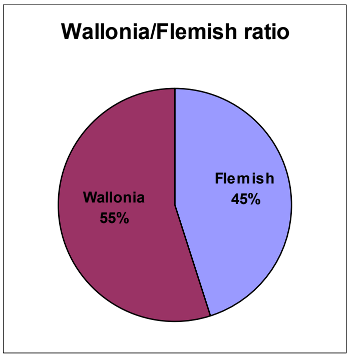

Fig 2 : Ratio between Flemish and Wallonia replies

6.2. General information about participants

It was easier to find university and research center actors in the field of nanotechnology than industries. University services working in nanotechnologies are well identified (e.g. Nanowal web site). Since the field is new, it is not a surprise that universities look like the most involved in the field. For large companies, their core business is hiding their nanotechnology business.

The sharing between the repliers is represented in the figure hereunder.

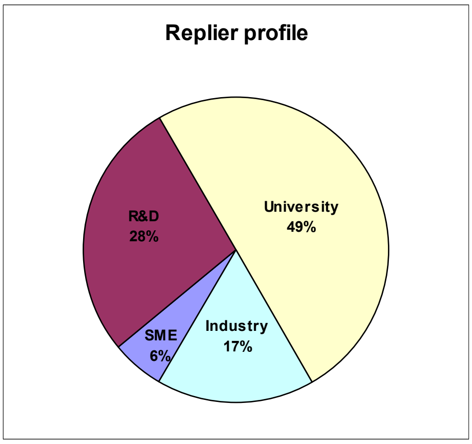

Fig 3 : Replier profile

6.3. Most addressed field

The addressed fields are rated hereafter.

Optics	16 %	Precision engineering	5 %
Electronics	16 %	Material science	81 %
Biology / Life science	31 %	Chemistry	62 %

The sum of the percents is higher than 100 %, since several fields are addressed simultaneously by some of the entities. Material science is the topics the most studied in nanotechnology. The same is observed in EC studies (e.g. [RD12]). This can be understood since it is the fundamental of all the applications. However chemistry takes also a good place in the field. Large companies are also concerned by material science. No replies were received from nanobiology industries that are probably not concerned by dimensional metrology.

6.4. Materials, components most studied or measured

This question was related to the materials most used. The replies didn't indicate precisely a chemical composition but generally a chemical group. The replies have been arranged by type of activities.

<u>Coating activity</u>

Polymer coatings, inorganic coatings, organic molecules (self-assembled monolayers), low k materials,... organic coatings with nano-particles included, fillers in polymers, metallic oxide coatings, optical thin films (like, e.g., diamond like carbon coatings), thin films of metals.

<u>Micro-nanoelectronic</u>

MicroElectroMechanical Systems (MEMS).
Nanoscale transistors as well as NanoElectroMechanical Systems (NEMS).
Analyze the doping atoms distribution in thin silicon layers after activation.
Ceramic superconductors, photovoltaics.

<u>Materials</u>

Nanomaterials (like, e.g., nanoparticles of transition and rare earth metal oxides, nanotubes(CNT)), alloys) nanostructured oxide materials (like, e.g., STO- LAO multilayers), pervoskite based materials, alloys (like, e.g., Ni-Ti shape memory) Conductive polymers, diamond, oxide nanoparticles silicon nanowires, carbon nanotubes, III-V material nanowires Aluminium alloys and ceramics.

<u>Chemical</u>

Catalysts (heterogeneous and homogeneous), zeolites, CdS-nanocolloids. Particles, metals and oxides, ceramics, nitrides and carbides, DLC (including films containing nano-precipitates) Thermoplastic polymers, Composites (thermoplastics and thermosets) Nanocomposites (TP/TD polymers + CNT, nanoclays),), Polymers (PVC, PVDC, PVDF, PA, PSu, etc.), catalysts, chemicals, additives (fillers, pigments, etc.)

Biology/life science

Proteins, DNA, dendrimers, aromatic molecules, etc. Templates for the growth of C-nanotubes, biosensors, microelectronic structures, material from third companies lipidlayers, nanovesicles (triblock polymers), proteins, protein crystals, DNA (plasmids, short strands), colloïds pharmaceuticals, polymers, proteins/peptides

Powders

Powders of metals. Nanopowders (carbides, nitrides, oxides and pure metals nanopowders)

6.5. Available and most used instrumentations

The next question is related to the tools and methods used in Belgium nanotechnology business.

The 3 most used instruments are in red, the next 3 in blue. A summary description of these devices can be found in [RD10].

	Available on site	Most used within the companies
Atomic Force Microscopy (AFM) or Scanning Force Microscopy (SFM)	11/18	**8/18**
Electron Diffraction (ED)	8/18	0/18
Scanning Tunnelling Microscopy (STM)	5/18	1/18
Scanning Electron Microscopy (SEM)	15/18	**11/18**
Transmission Electron Microscopy (TEM)	11/18	**4/18**
Scanning Transmission Electron Microscopy (STEM)	6/18	2/18
Small Angle Neutron Scattering (SANS)	0/18	0/18
Small Angle XRay Scattering (SAXS)	2/18	0/18
Scanning Near-field Optical Microscopy (SNOM or NSOM)	1/18	0/18
Electron Energy Loss Spectroscopy (EELS)	4/18	0/18
Energy Dispersive X-Ray Spectroscopy (EDX or EDS or XEDS)	9/18	**6/18**
X-Ray Diffraction (XRD)	10/18	4/18
X-Ray Absorption Spectroscopy (XAS)	0/18	0/18
Neutron Diffraction (ND)	0/18	0/18
Single Molecule Spectroscopy (SMS)	2/18	0/18
Auger Electron Spectroscopy (AES)	3/18	0/18

Polarization Spectroscopy (PS)	1/18	0/18
Photoluminescence Spectrocopy (PL), Electroluminescence Spectroscopy (EL), Cathodoluminescence Spectroscopy (CL)	2/18	0/18
Fourier Transform Infrared Spectroscopy (FTIR)	7/18	**4/18**
Raman Spectroscopy (RS)	6/18	**4/18**
X-Ray Photoelectron Spectroscopy (XPS) or Electron Spectroscopy For Chemical Analysis (ESCA)	2/18	0/18
Profilometer (PROF)	6/18	1/18
Secondary Ion Mass Spectrometry (SIMS)	2/18	0/18
Differential Scanning Calorimetry (DSC)	10/18	0/18
Mössbauer Spectroscopy (MS)	0/18	0/18
Nuclear Magnetic Resonance (NMR)	6/18	2/18
Cyclic Voltammetry (CV) and Linear Sweep Voltammetry (LSV)	4/18	0/18
Four point probe AND I-V techniques (4P)	4/18	0/18
Capacitance spectroscopy (CS)	1/18	0/18
Other : Interferometer	2/18	1/18

The table indicates that the Belgium labs are well equipped; it is possible to find nearly all types of instruments listed and additional ones that were not in the initial questionnaire. A description of most of the instruments can be found in [RD10].

SEM is available in 84% of the labs. It is also the instrument the most used. The next one is the AFM, followed by EDT.

6.6. Instrumentation environment

This question is related to the environmental conditions of the lab where the instruments are located. This is connected to the uncertainty of the measurement.

Standard lab	50 %
Clean room	37 %
Lab + Air Condition	68 %
Damping	36 %
Pressure	0 %

Most of the users work in a temperature controlled lab. The temperature control is usually +/- 1°C or better. When damping is present, it is always on a dumping system provided with the metrology instrument, and not a damped floor. Some users work in clean rooms but the latter are not always classified.

6.7. Range and incertitude

These questions are related to the performances achieved or at least announced within the classical measurement procedures. It concerns also if relative or absolute measurements are needed. Finally, the question tackles if the users are satisfied with the measurement uncertainty.

In which range do you measure (x nm to y µm)?	few nm to few 100 µm
With which uncertainty	0.1 nm to 20 %
If not sufficient, what uncertainty would you need?	Most of the answers show that the users are happy with what they have.
Absolute / relative measurements	10 absolute, 5 relative, 3 both

The range is quite large, and the uncertainty mostly depends on the instruments used. The announced uncertainties (e.g. 0.1 nm) are indications coming from the instrument manufacturer. The question indicates that there is no need for better measurements. Additionally, there is no guarantee that the question is well understood. Most people are not necessarily specialized in measurement techniques, and there is a potential mismatch between the terminologies used (e.g. uncertainty, accuracy, resolution, repeatability, absolute error …). Most of the users ask for absolute measurement, but without any care to traceability to a standard as it can be understood from the next questions.

6.8. Standard and procedure

This part tries to understand with which guarantees the previous figures are achieved.

Do you use special measurement protocols? If yes, which one (ISO, In home,..)	66% have no special protocol. The remaining ones use internal procedures. These are based on the user manual of the instrument provider.
Are you concerned only by the functionalities of the components you develop or is it required that this product is further interfaced or in interaction with a	The replies are not obvious. Nevertheless the next question indicates that interface problem do not occur frequently.

third party hardware?	
When it is required to have compatibility, how do you fix the rules?	Usually not required.
How do you ensure consistent measurements, and how do you know the part meets the specification?	Check consistency wrt other instruments, or well known pieces reference hardware or physical properties
What type of terminology do you use (scientific, commercial, in home, standard)?	All the users employ scientific terminology
Do you need accredited measurements?	Nobody needs accreditation

Most of the users declare explicitly to not use special measurement protocols. The remaining ones use internal procedures inspire from the instrument procedures, their knowledge of their tested items and code of best practice. There is no particular demand for protocol except only one for sample preparation.

The interface aspects are not critical; this sector in Belgium is not mature enough for large hardware exchange between laboratories and/or industries. When interface problems occur they are handled through test plans.

To improve the confidence in the measurement, the approach is to cross-correlate measurements with different instruments. Most of the labs having several instruments are able to do this internally. Another way to evaluate the instrument is to use known physical parameters. Only one lab indicates participating to round robin.

The terminology used is scientific, the one codified trough scientific publication. Presently, nobody required accreditation, labs have no money for it, it just can give an added value.

6.9. Measurement and calibration frequency

These questions concern the calibration process used related to the used frequency

How frequently do you measure (n°/week)?	More than 1/day with the most frequent instrument to 1/week with the other
How frequently do you perform the maintenance of the instrument (n°/year)?	At least 1/year.
How frequently do you perform a recalibration of the instrument (n°/year)?	Most 1/year
What procedure is followed for the	The calibration is generally done in

calibration?	home with manufacturer procedure and staff. Return to the manufacturer is exceptional.
What is the calibration process you use? E.g.: gauge blocks, which references, which material	Calibration grids are used

The nanometrology tools are used quite frequently. This indicates that the instrument operators have a good follow on and good understanding of the instruments. This provides additional confidence in the measurements.

Concerning the maintenance and calibration issues, these are performed once a year as advised by the manufacturers. Or even more for some instruments more sensitive to ageing effect or more frequently used.

Almost all the consulted people use calibrated grids. These are not well identified but are advised by the manufacturer. Some labs have dedicated sample for these calibration activities.

6.10. Cost impact

This question is related to the cost impact of wrong measurements.

What is the cost impact of wrong measurements	Variable

It is a point that it is difficult to evaluate. In universities the impact is mostly bad science and a loss of time. In industries, it starts from a waste of time to several 10 000 of Euros and can be really catastrophic in SME.

The reason why cost impact evaluation is difficult is that this evaluation depends on several parameters. Example of sub cost impacts are:

- Cost to redo the measurement
- Cost to redo the hardware
- Cost to improve or correct the system
- Cost link to discarding a good device
- Juridical cost
- Lost in reputation ➔ lost of potential customer

From this list of parameters it is obvious that it not always easy to provide accurate figures, knowing that these cost impact factors are also depending on the component or process involved.

Another aspect linked particularly to the nano field, is that the dimension of nano materials and nano particles is lower than the standard physical phenomenon size characteristics and by this way several physical, optical or chemical properties start to become completely different.

During the process, dimensional measurements are taken, but at the end only the final functionalities are considered. This method gives warranties again the measurement error. For this, the cost impact is only limited to hardware and the operation between the dimensional and the expected functionalities measurements.

6.11. Further needs

Final questions try to evaluate the actual and further needs in nanometrology and the opted solution when the metrology tools are not available.

When you need accurate measurements that are not available at your premises, how do you proceed?	All the users ask to another well known lab
Do you have or see other concerns or demands for nanometrology for your current or future activities?	Quantification of size and shape of nanoparticles Sample preparation Safety is currently a big concern with nanomaterials such as carbon nanotubes, nanowires and nanoparticles. Special precautions have already been taken for working with carbon nanotubes and their shipment. Profilometry

When the measurement instrument is not available on site, the user goes to another well known laboratory. However, this procedure is quite less frequent than several years ago. Now, it is not more exceptional to have a SEM in a small lab. Additionally, the dedicated instrument needed for their daily applications is also available in home. In SME or start up, where these instruments are too costly, they have a network of companies able to perform the measurements.

The further needs are quite low and some already taken in consideration in actual international developments.

Safety is currently a big concern with nanomaterials and is considered in the Nanosafe EC project.

For sample preparation, an ISO platform is taking care to establish well accepted procedures in sample preparation.

Profilometry. A lot of commercial profilometers (Veeco Dektak, nanosurf, FRT GmH, …) and in home profilometers (CSL, TNO, NPL, PTB, LNE) are becoming available. However, care must be taken in the understanding of the performances (e.g. repeatability of 1 nm is repeatability when there is no displacement of the probe, which is no what a profilometer users want (repeatability over a scan)). The same is true for quantification of size and shape of nanoparticles, where several techniques as laser granulometry are commercially available.

7. Conclusion

The enquiry indicates that in Belgium

- it is possible to find all types of instruments used in nanotechnology,
- the organizations concerned by nanotechnology work mostly with Scanning Electron Microscope (SEM) instruments, while in Germany SPM looks to be the most used [RD6]. SEM are available in 85% of the consulted labs. SEM are generally used for topographic measurements,
- none of entities required accreditation,
- when calibrations are performed, calibrated grids are employed. The calibration process generally accepted used VLSI grids (http://www.vlsistandards.com/home/default.asp),
- the instruments are generally located in a standard lab with controlled temperature, only 20% are in clean room,
- when the measurement facility is not available on site, well know labs are addressed,
- the parity between Wallonia and Flemish is well balanced but there is no indication that a region is more involved than another,
- 45 % of actors are universities. It was easier to find university actors in the field of nanotechnology than industries. Additionally, this activities is quite new and concern most research activities than industrial production,
- material science is the topics the most studied in nanotechnology,
- the measurement range is quite large between 1 nm up to 100 µm, as well as the uncertainty 0.1 nm to 20%. These are linked to the applications. The users are more concerned by absolute measurements than relative,
- the instruments are well used generally more than once a day. The calibration and maintenance are performed once a year at the user premises with the help of the instrument manufacturer staff,
- the cost impact of wrong measurements is quite variable, from bad science in university and waste of time to several 10 k€ in industry. The standard way to minimize this is to check consistency with respect to other instruments,
- the particular additional demands for future activity highlighted are already covered by international activities.

- A similar study is performed in France by the LNE. It has been proposed to compare the results.

8. Acknowledgements

We thank all the contributors that kindly replied to the questionnaire.

9. Annex 1 : Questionnaire

The concern is to know if precise control of dimensions of object (3D, layer, powder …) is a key issue in nanotechnology.

The goals of the questionnaire are :

1) To evaluate the most used instruments and methods in nanometrology by the Belgium nanotechnology community.
2) To evaluate how standard and interface are handled and needed by the Belgium nanotechnology community.

9.1. General questions on your core business in nanotechnology

Name of the company:

Name of the department:

SME : University: Large company: Research center:

Field of activity:

Optics	
Electronics	
Biology / Life science	
Precision engineering	
Material science	
Chemistry	
Other please specify	

9.2. Dedicated questions on your dimensional metrology activity

1	What are the materials, components you most study or measure? Chemical name, type of polymers, type of powder, coating material, …(e.g: CdSe/ZnS core shell nanocrystal, PbTe nanorods, ..)		
2	What types of instruments are available in your company (put a cross in the last column when available at your premises).	Atomic Force Microscopy(AFM) or Scanning Force Microscopy(SFM)	
		Electron Diffraction (ED)	
		Scanning Tunneling Microscopy (STM)	
		Scanning Electron Microscopy (SEM)	
		Transmission Electron Microscopy (TEM)	
		Scanning Transmission Electron Microscopy (STEM)	
		Small Angle Neutron Scattering (SANS)	
		Small Angle XRay Scattering (SAXS)	
		Scanning Near-field Optical Microscopy (SNOM or NSOM)	
		Electron Energy Loss Spectroscopy (EELS)	
		Energy Dispersive XRay spectroscopy(EDX or EDS or XEDS)	
		X-Ray Diffraction (XRD)	
		X-Ray Absorption Spectroscopy (XAS)	
		Neutron Diffraction (ND)	
	Other? Please fill here below.	Singlemolecule spectroscopy (SMS)	
		Auger Electron Spectroscopy (AES)	
		Polarization Spectroscopy (PS)	
		Photoluminescence Spectrocopy (PL), Electroluminescence Spectroscopy (EL), Cathodoluminescence Spectroscopy(CL)	
		Fourier Transform Infrared Spectroscopy (FTIR)	
		Raman Spectroscopy (RS)	
		X-Ray Photoelectron Spectroscopy (XPS) or Electron Spectroscopy For Chemical Analysis (ESCA)	
		Profilometer (PROF)	
		Secondary Ion Mass Spectrometry (SIMS)	
		Differential Scanning Calorimetry (DSC)	
		Mössbauer Spectroscopy (MS)	
		Nuclear Magnetic Resonance (NMR)	
		Cyclic Voltammetry (CV) and Linear Sweep Voltammetry (LSV)	
		Four point probe AND I-V techniques (4P)	
		Capacitance spectroscopy (CS)	

<table>
<tr><td></td><td></td><td colspan="2"></td></tr>
<tr><td>3</td><td>With respect to the previous list, what type of instruments are you using most frequently?
Brand name:
Type:</td><td colspan="2"></td></tr>
<tr><td rowspan="7">4</td><td rowspan="7">Are there particular environmental conditions where your instrument(s) is (are) located and used?</td><td>Standard lab</td><td></td></tr>
<tr><td>Clean room (which class)</td><td></td></tr>
<tr><td>Air Condition room (which temperature stability)</td><td></td></tr>
<tr><td>Pressure</td><td></td></tr>
<tr><td>Damping system</td><td></td></tr>
<tr><td>Other</td><td></td></tr>
<tr><td></td><td></td></tr>
<tr><td>5</td><td>In which range do you measure (x nm to y µm)?</td><td colspan="2"></td></tr>
<tr><td>6.a</td><td>With which uncertainty?</td><td colspan="2"></td></tr>
<tr><td>6.b</td><td>If not sufficient, what uncertainty would you need?</td><td colspan="2"></td></tr>
<tr><td>7</td><td>Do you need relative or absolute measurement? Why?</td><td colspan="2"></td></tr>
<tr><td>8</td><td>Do you use special measurement protocols? If yes, which one (ISO, In home,..)</td><td colspan="2"></td></tr>
<tr><td>9</td><td>How frequently do you measure (n°/week)?</td><td colspan="2"></td></tr>
<tr><td>10</td><td>How frequently do you perform the maintenance of the instrument (n°/year)?</td><td colspan="2"></td></tr>
<tr><td>11</td><td>How frequently do you perform a</td><td colspan="2"></td></tr>
</table>

	recalibration of the instrument (n°/year)?		
12	What procedure is followed for the calibration?	Return to the manufacturer	
		In home with manufacturer	
		In home with manufacturer procedure	
		In home with self established procedure	
		Other please specified	
13	What is the calibration process you use? E.g.: gauge blocks, which references, which material		
14	Are you concerned only by the functionalities of the components you develop or is it required that this product is further interfaced or in interaction with a third party hardware?		
15	When it is required to have compatibility, how do you fix the rules?		
16	How do you ensure consistent measurements, and how do you know the part meets the specification?		
17	What is the cost impact of wrong measurements?		
18	What type of terminology do you used (scientific, commercial, in home, standard)?		
19	When you need accurate measurements that are not available at your premises, how do you proceed?	Ask to another well know lab	
		Ask to an accredited lab	
		It doesn't matter unless the measurements are performed	

20	Do you need accredited measurements?	
21	Do you have or see other concerns or demands for nanometrology for your current or future activities?	

10. Annex 2: List of contacted Industries, research centers and universities

N°	Name	Contact Person	Phone/email	Type of activity	Reply
1	Agfa Gevaerts	Chris Van Roost	http://www.agfa.com/en/sp/forms/contactus.jsp	Metrology department	OK
2	Belgian Ceramic Research Centre http://www.bcrc.be/	Fabrice Petit	j.lagneau@bcrc.be	Nano composites, new concept of standardization which allows materials to be classified on the basis of their level of performance for a given mode of stress.	NR
3	Bekaert NV	Nadine Van de Velde	bekaert.engineering@bekaert.com	Material	NR
4	Biovallee	JF Dierick	xavier.barthelemy@biovallee.be	Biology, genomic	Restructuration phase concerning the R&D projects
5	Centexbel		philippe.vanacker@centexbel.be	Smart Textile	NR after indicating they concern about nanometrology
6	Certech	Henri May	info@certech.be	R&D services	OK
7	CoRI	Céline, de Lame Sophie Vonckx	vonckx.s@cori-coatings.be +32 2 652 22 49	Anticorrosion, Paint Formulation Coating	NR
8	Crif	Frederik Cambier	Umberto.baraldi@crif.be +32 498 91 93 91	Powder	OK

9	CSL	Denis Vandermael	dvandermael@ulg.ac.be		OK
10	Eurogentec		ph.boulton@eurogentec.com 32 4 366 61 61	Nanobiology	NR
11	FLAMAC	Johan Paul director	32 9 264 58 13 johan.paul@flamac.be	Flanders Materials center	OK
12	GIGA	Serge Pampfer	pampfer.giga@ulg.ac.be <pampfer.giga@ulg.ac.be>	Nanobiology	Not concerned
13	Glaverbel		DOMINIQUE.MICHIELS@glaverbel.com	Glass Coating	OK
14	IMOMEC	M.d'Olislaeger		Science of materials	OK
15	IMEC	Thomas Hantschel Operational Manager	Materials & Components Analysis Tel: +32-16-28-1056 Email: thomas.hantschel@imec.be	Electronic	OK
16	LUXILON	Herbert De Breuck	nico.vanmalderen@luxilon.be Industriepark / Vosveld 11 B-2110 Anvers, Belgique tél. +32 (0)3 326 33 88	High tec mono filaments	NR
17	MATERIA NOVA		michael.alexandre@umh.ac.be 065/37 34 81	Material analysis TGA-DTA thermogravimetry coupled with mass spectroscopy, micro and nano-hardness, dilatometry, X-Ray diffraction and X fluorescence, LASER granulometry, porosimetry and specific surface measurement, rheometry, , …, He-Ne laser ellipsometry, specific electrical charge, Langmuir-Blodgett balance, ESCA	Wait reply

				spectroscopy in XPS, Tof-SIMS spectroscopy, …	
18	NANOCYL	F. Luizi		C Nanotube	
19	Nanoshape	Marc Wielands	marc.wielandts@amos.be	Mirror manufacturing	OK
20	Solvay	Dr Antoine GHANEM	Phone : 32(0)2-2643422 mailto:antoine.ghanem@solvay.com	SOLVAY Research and Technology Materials Characterization Analytical Technologies	OK
21	VITO	Frans Snijkers	Rosita Persoons Tel. + 32 14 33 57 30 rosita.persoons@vito.be		NR
	Name	**Institution**	**e-mail**	**Type of activity**	
22	Bayot, Vincent	UCL	bayot@dice.ucl.ac.be	Nano electronic SEMCv	NR
23	Beyer, Günter	Eupen AG	gbeyer@euregio.net	Nanocomposites as flame retardants for polymers	NR
24	Buess-Herman, Claudine	ULB	cbuess@ulb.ac.be	Tailoring of electrode surfaces Characterization of surfaces by electrochemical methods, FTIR, STM, AFM…	NR
25	Delplancke, Jean-Luc	ULB	jdelpla@ulb.ac.be	Metallic nanopowder production and characterization - Matter and Materials Department	at CEE DG RTE

26	Delplancke, M.P.	ULB	mpdelpla@ulb.ac.be	Nanocomposite layers – Multifunctional coating	OK
27	Deparis, Olivier	FUNDP	odeparis@fundp.ac.be	Engineering nano-structured optical materials	NR
28	De Pauw, Edwin	ULg	E.DePauw@ulg.ac.be	NOMADE Nanoparticles for Optical and MAgnetic Detection	NR
29	Dubois, Philippe	UMH	Philippe.dubois@umh.ac.be	Preparation and characterization of polymer nanocomposites	NR
30	Dufrene, Yves	UCL	dufrene@cifa.ucl.ac.be	Exploring biosystems on the nanoscale : from single molecules to living cells	NR
31	Ferain, Etienne	UCL	ferain@poly.ucl.ac.be	Track Etching Technology OK	NR
32	Grandfils, Christian	ULg CATµ	C.Grandfils@ulg.ac.be	Nanoparticles for drug delivery and diagnostic OK 32 4 366 3506	OK
33	Jerome, Christine	ULg	C.Jerome@ulg.ac.be	Synthesis and surface functionalization of metallic nanodots	NR
34	Jonas, Alain	UCL	jonas@poly.ucl.ac.be	Nanotech Research in Alain M. Jonas' Group	OK
35	Lazzaroni, Roberto	UMH	Roberto@averell.umh.ac.be	Peptosome-based NanostructuredNetwork	NR
36	Lecomte-Beckers, Jacqueline	ULg	Jacqueline.Lecomte@ulg.ac.be	Alliage de titane / nano-structuré	NR
37	Legras, Roger	UCL	legras@poly.ucl.ac.be	Track Etching Technology	NR
38	Muller, Robert	UMH	Robert.muller@umh.ac.be	development of nanomagnetic systems as contrast agents for magnetic resonance imaging (MRI),	NR
39	Pirard, Jean-Paul	ULg	Jean-Paul.Pirard@ulg.ac.be	Nanostructured sol-gel porous materials	NR
40	Piraux, Luc	UCL	piraux@pcpm.ucl.ac.be	nanoporous media and nanowire	NR
41	Raskin, Jean-Pierre	UCL	raskin@emic.ucl.ac.be	CERIM	OK
42	Wautelet, Michel	UMH	michel.wautelet@umh.ac.be	Modele Unite de Photonique Experimental	NR

43	Ronnie Willaert	VUB	Ronnie.Willaert@vub.ac.be <Ronnie.Willaert@vub.ac.be >	Ultrastructure	OK
44	Rolf Erni	Universiteit Antwerpen	rolf.erni@ua.ac.be	Electron Microscopy for Material Science	OK
45		U Gent		Inorganic and Physical Chemistry	OK
46	Vander Auweraer	KUL		organic molecules	OK