Nanometrology and its role in the development of nanotechnology

Rob Bergmans

Nederlands Meetinstituut

Van Swinden Laboratorium

NMi Van Swinden Laboratorium

" The Art of Measurement "

- Dutch national metrology institute
  - Maintenance of national standards, provider of traceability
  - Development of novel standards, instruments, methods
  - Calibration services
  - Consultancy

Jan Hendrik van Swinden

"The meter" (1799)

Outline

- Why do we need measurement?
- Metrology
- International Metrological infrastructure
- Nanometrology
  - Roadmap
  - Trends & Outlook in the future

Trade

Supplier

wants to deliver no

more than was agreed

Customer

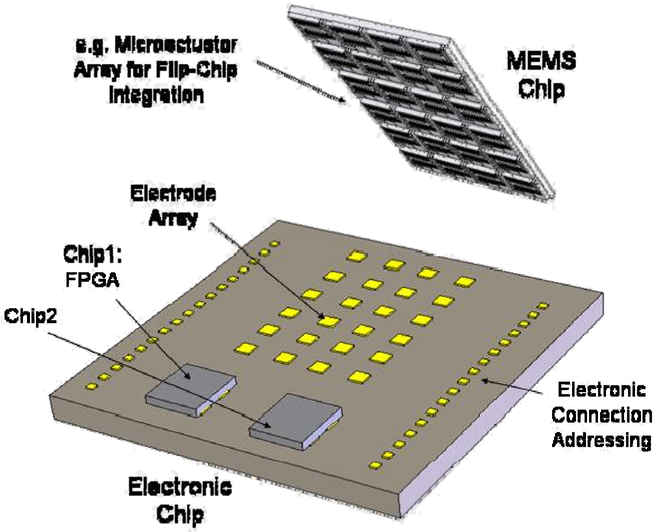
wants to receive no

less

than was agreed

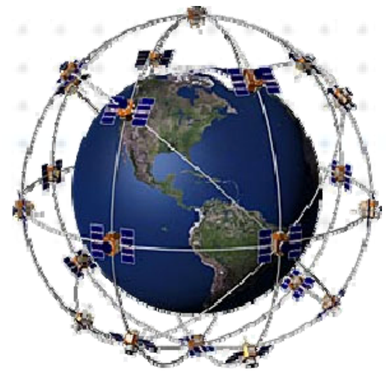
Industry

- Production processes
  - Enabling, from lab to factory
  - Efficiency
  - Less waste
- Parts from individual suppliers should always fit during assembly



Society needs

- Global positioning



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Health

- Entertainment

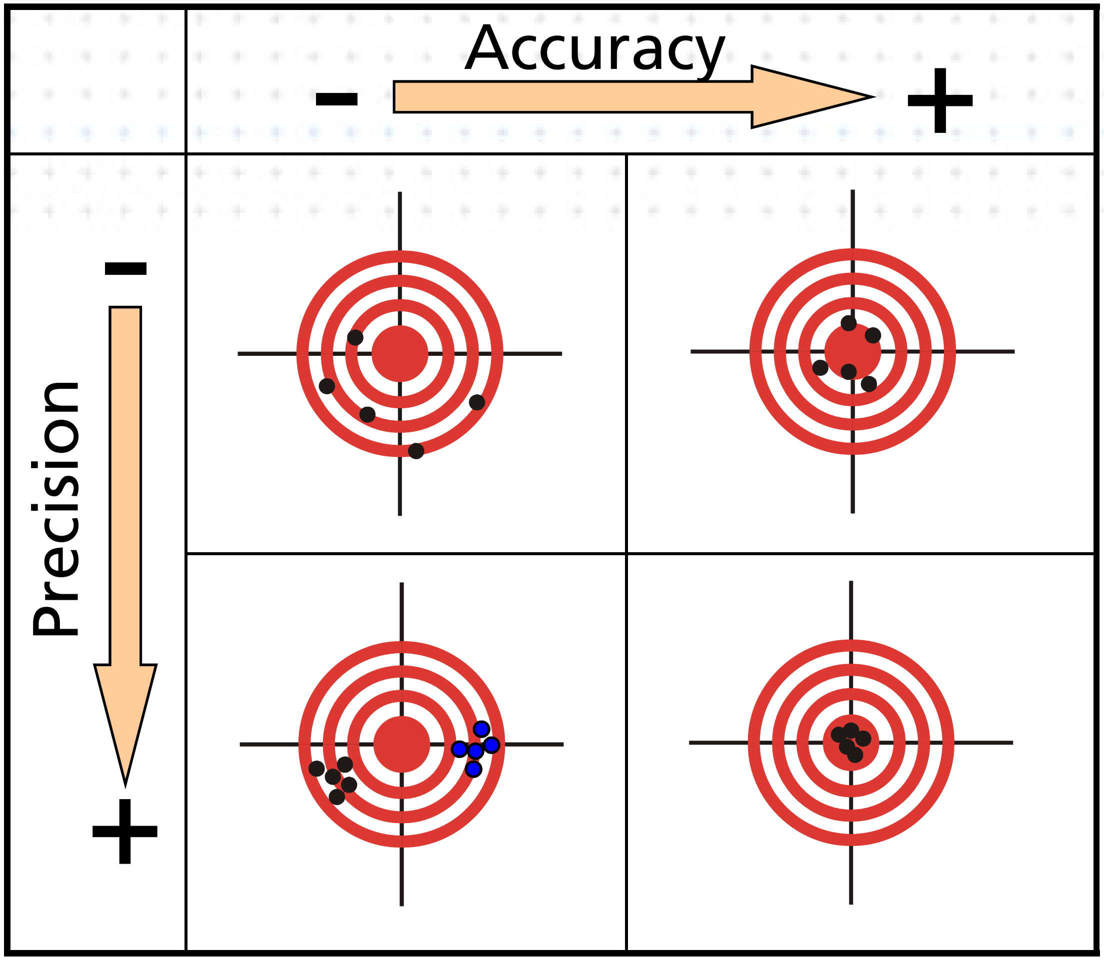
Measurement

We need ..

- Reliable
- Comparable
- Accurate
- Confidence

In measurements

Precision versus Accuracy



Accuracy ← reference value ← standards ← SI unit

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Système international d'unités (SI)

- The SI was developed in 1960 from the former metre-kilogram-second (mks) system
- Currently has 7 base units
  - The metre
  - The kilogram
  - The second
  - The ampere
  - The Kelvin
  - The mole
  - The candele

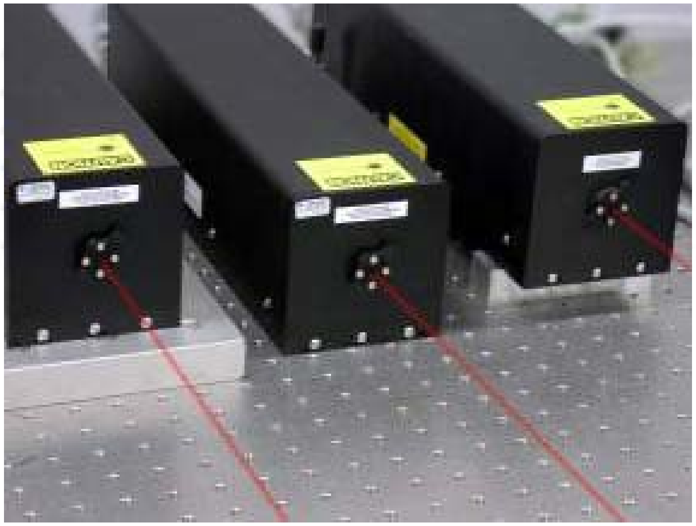
Definition of the Metre

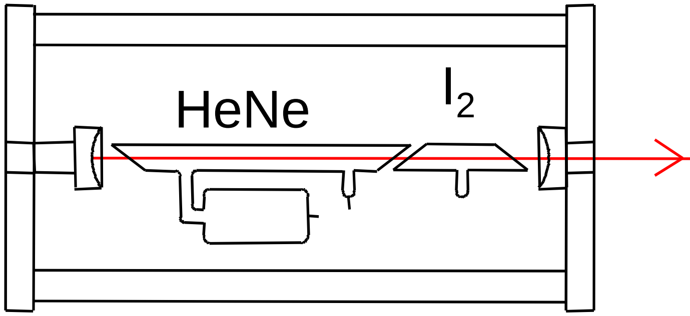
Definition of the Metre

- The metre is the length of the path travelled by light in vacuum in a time of (1/299 792 458) second (17th CGPM (1983), Res. 1)

Metre: practical realization

Primary standard of length





l = 632991398,22 fm

f = 473612214705 kHz

Iodine stabilized He-Ne laser.

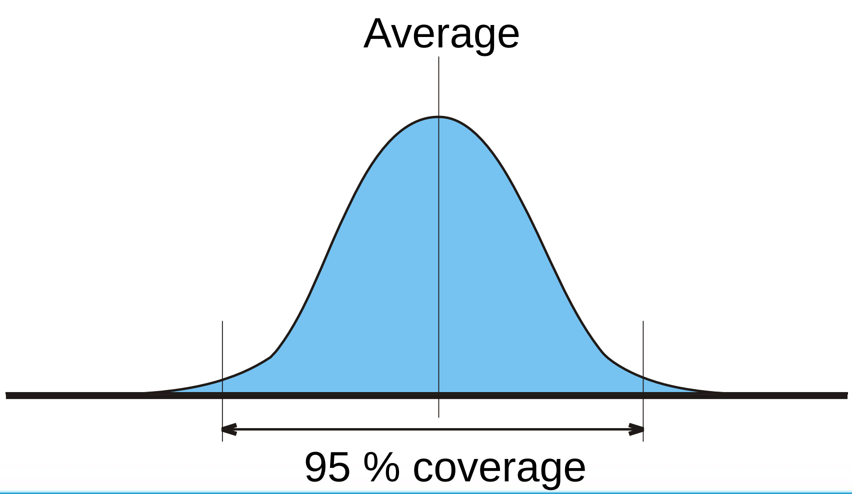
Absolute Uncertainty: 4,0 $\cdot 10^{-11}$

Traceability

- Traceability describes the way to link all measurements in a measurement chain ultimately to the SI unit
- Traceability provides measurement uncertainty and therefore accuracy

Measurement uncertainty

- Range of values assigned to a measurand to indicate the relation between the measured value and the true (but unknown) value
- Property of the measurement process



Measurement

- In a measurement chain every subsequent measurement introduces additional uncertainty
- Keep the chain as short as possible
- Use standards with an appropriate uncertainty

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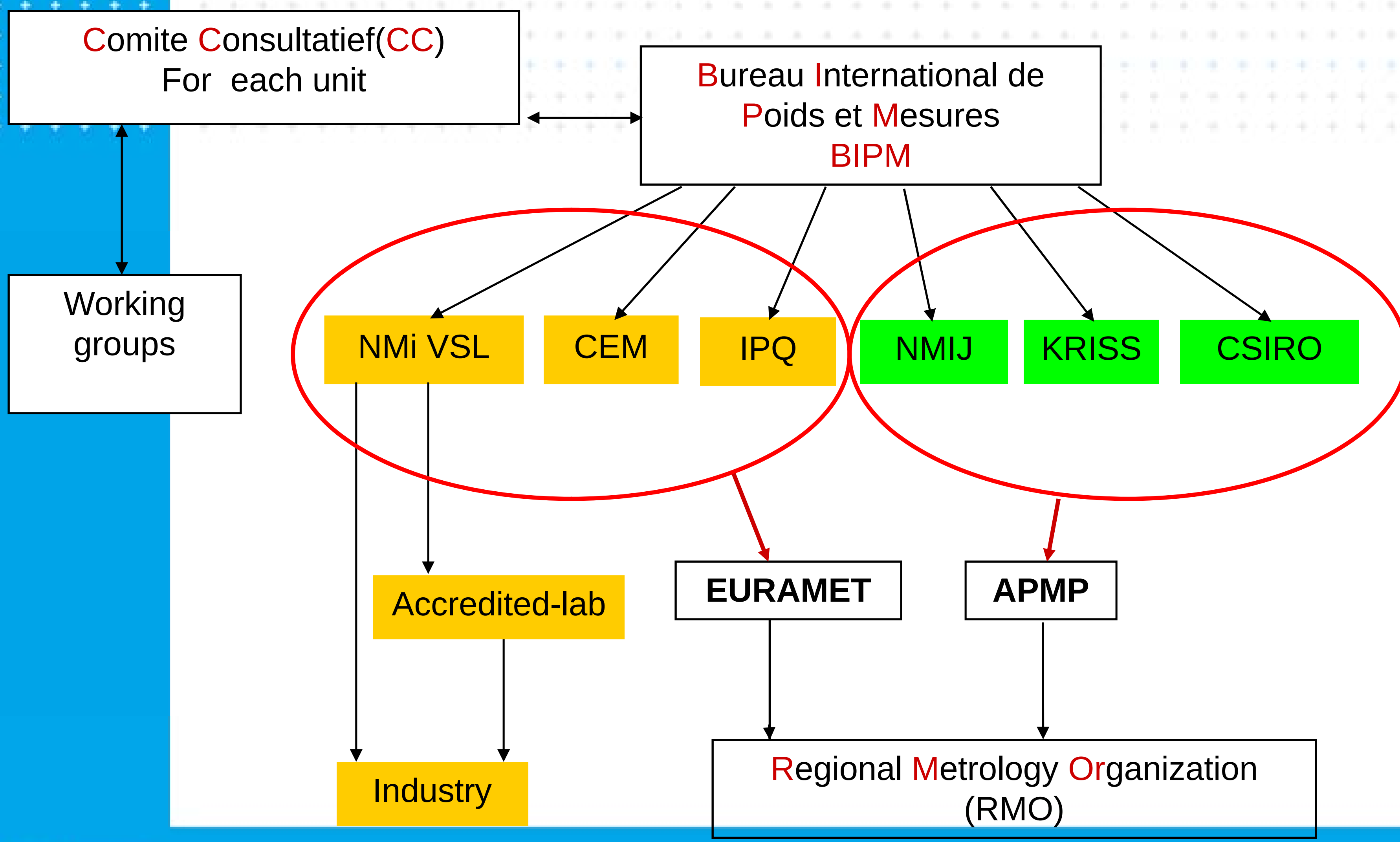
International metrological infrastructure

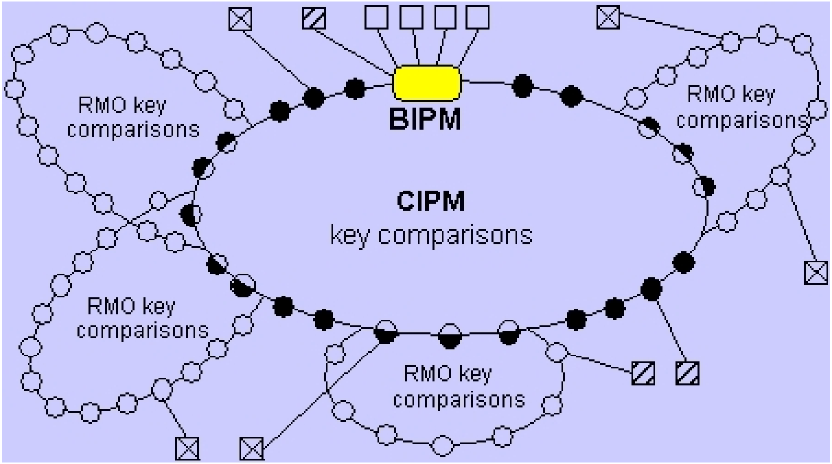


Mutual recognition

- Mutual recognition of measurement results on a national and international level is crucial for (inter)national trade
- Mutual recognition is based on demonstrated performance and evidenced by comparison results
- Performance is regularly verified by comparison

Comparisons

- Key comparisons: confirm and consolidate existing methods and performance and demonstrate equivalence
- Scientific: establish state of the art performance for novel methods and or instrumentation
- Results are publicly available on-line



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Nanometrology <-> classical metrology

- Very high accuracy demands
- New techniques and methods
- Hugh investments in money and man-hours
- Time-to-market of nanotechnology standards/instruments is much shorter

2001 start of EURAMET initiative on nanometrology

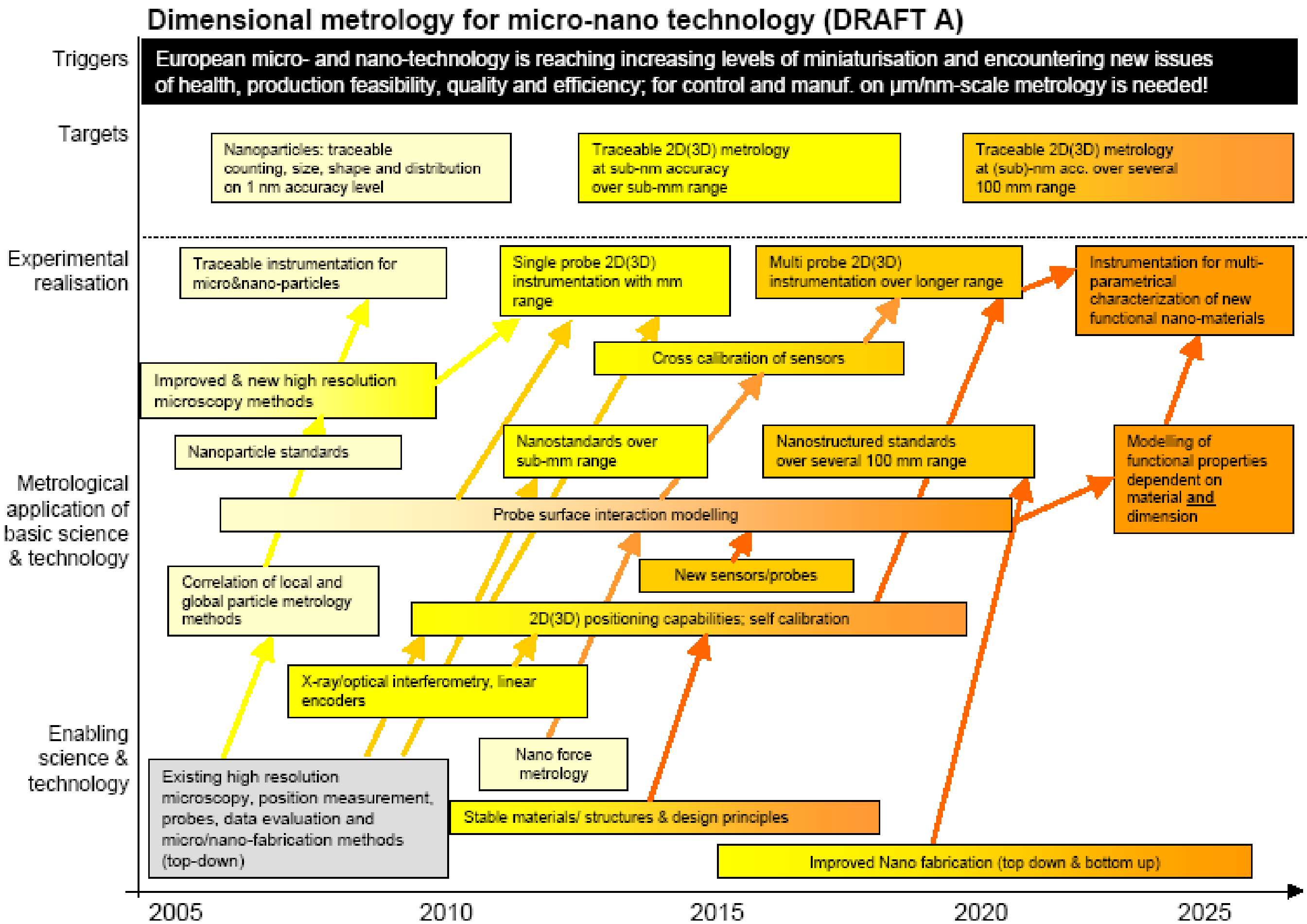
Cooperation European NMIs

- European Metrology Research Program (First formal research cooperation)

- 2006 Roadmap Nanometrology
- 2007 Definition of projects
- 2008 Start of first joint research project.

Micro nano Roadmap

EURAMET
European Association of National Metrology Institutes



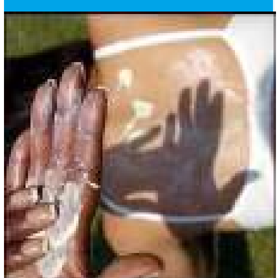
Main challenges

- Nanoparticle standards
- Scanning probe microscopy to support nanotechnology
- 2D- and 3D-instrumentation with nm uncertainty.
- Displacement metrology at the nanometer scale

Traceable characterization of nanoparticles

Why ?

- Nanoparticles have unique properties due to the reduced dimensions.
- Many innovative uses, produce, processes…
- but also toxicological concerns:



Objectives

- New traceable standards and procedures to determine the size, shape and distribution of nanoparticles with an accuracy of better than 1 nm.
- Correlated with preparation method and the end product environment (on a surface or in suspension)
- Improved instrumentation for nanoparticles analysis and new methods for the reliable characterization of nanoparticle shape.

Nanoparticle standards

National Institute of Standards & Technology

Report of Investigation

Reference Material 8011

Gold Nanoparticles, Nominal 10 nm Diameter

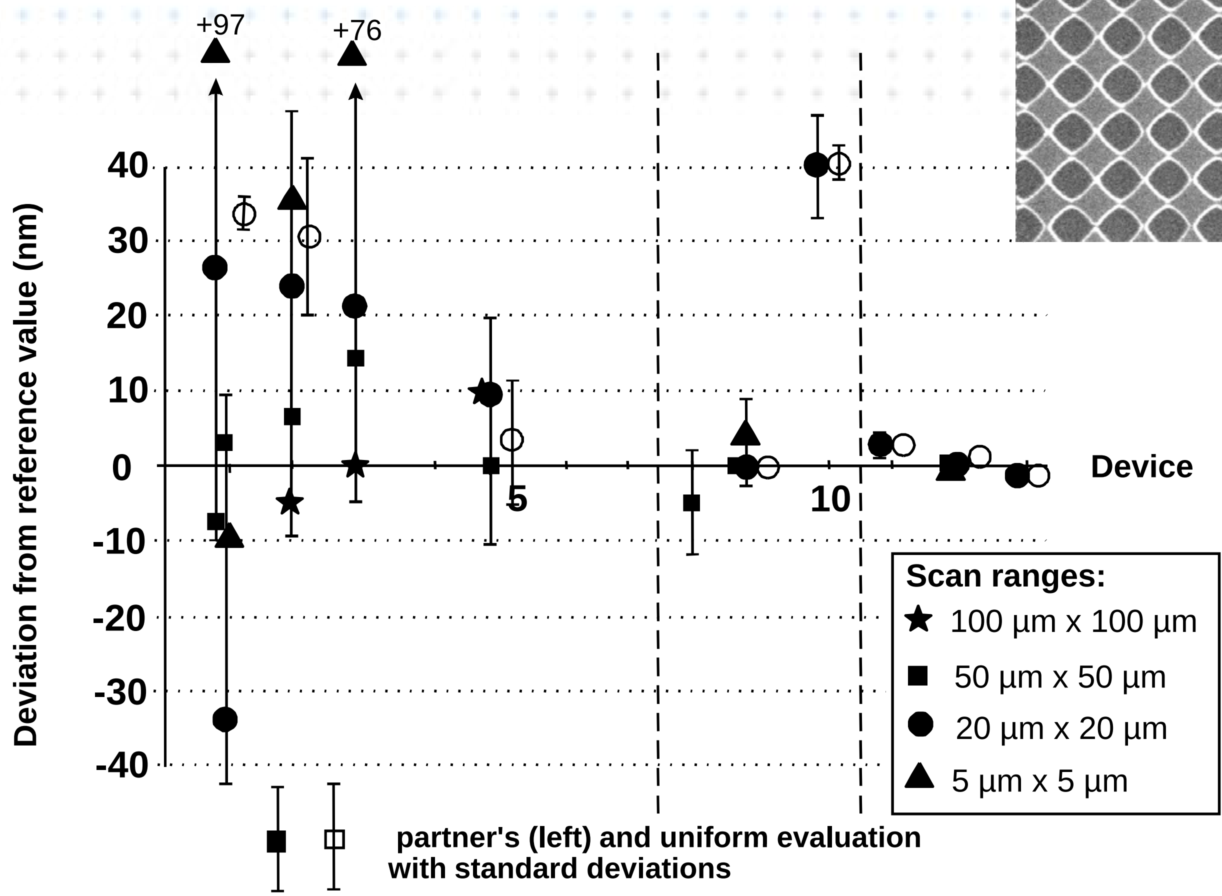
Table 1. Reference Value Mean Size and Expanded Uncertainty [a]
Average Particle Size (Diameter), in nm

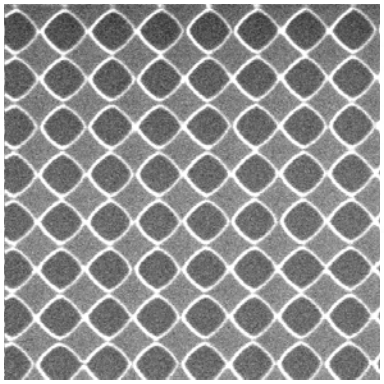
Technique	**Analyte Form**	**Particle Size (nm)**
Atomic Force Microscopy	dry, deposited on substrate	8.5 ± 0.3
Scanning Electron Microscopy	dry, deposited on substrate	9.9 ± 0.1
Transmission Electron Microscopy	dry, deposited on substrate	8.9 ± 0.1
Differential Mobility Analysis	dry, aerosol	11.3 ± 0.1
Dynamic Light Scattering	liquid suspension	13.5 ± 0.1
Small-Angle X-ray Scattering	liquid suspension	9.1 ± 1.8

Scanning Probe Microscope

- Metrological Scanning Probe Microscope
- Surface Probe interactions
- Cross correlation of different measurement techniques i.e AFM and SEM.

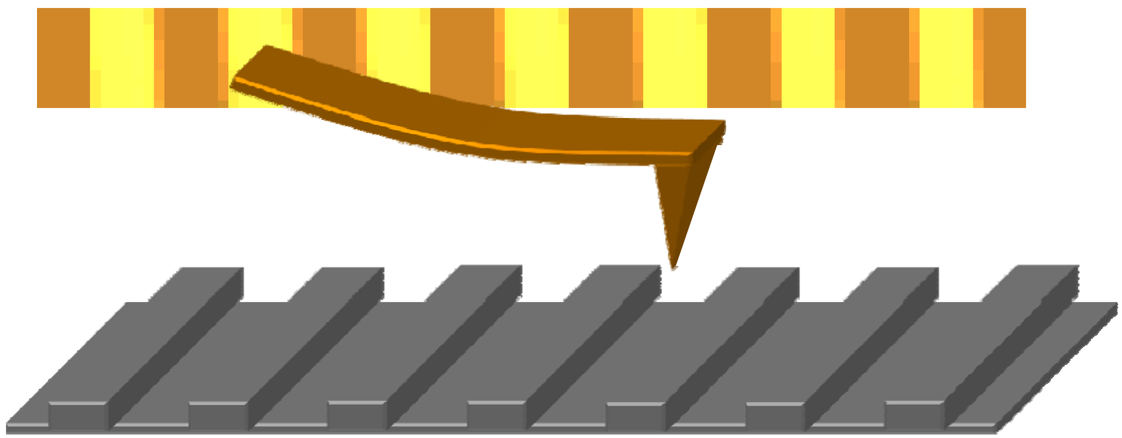
SPM international comparison results: lateral



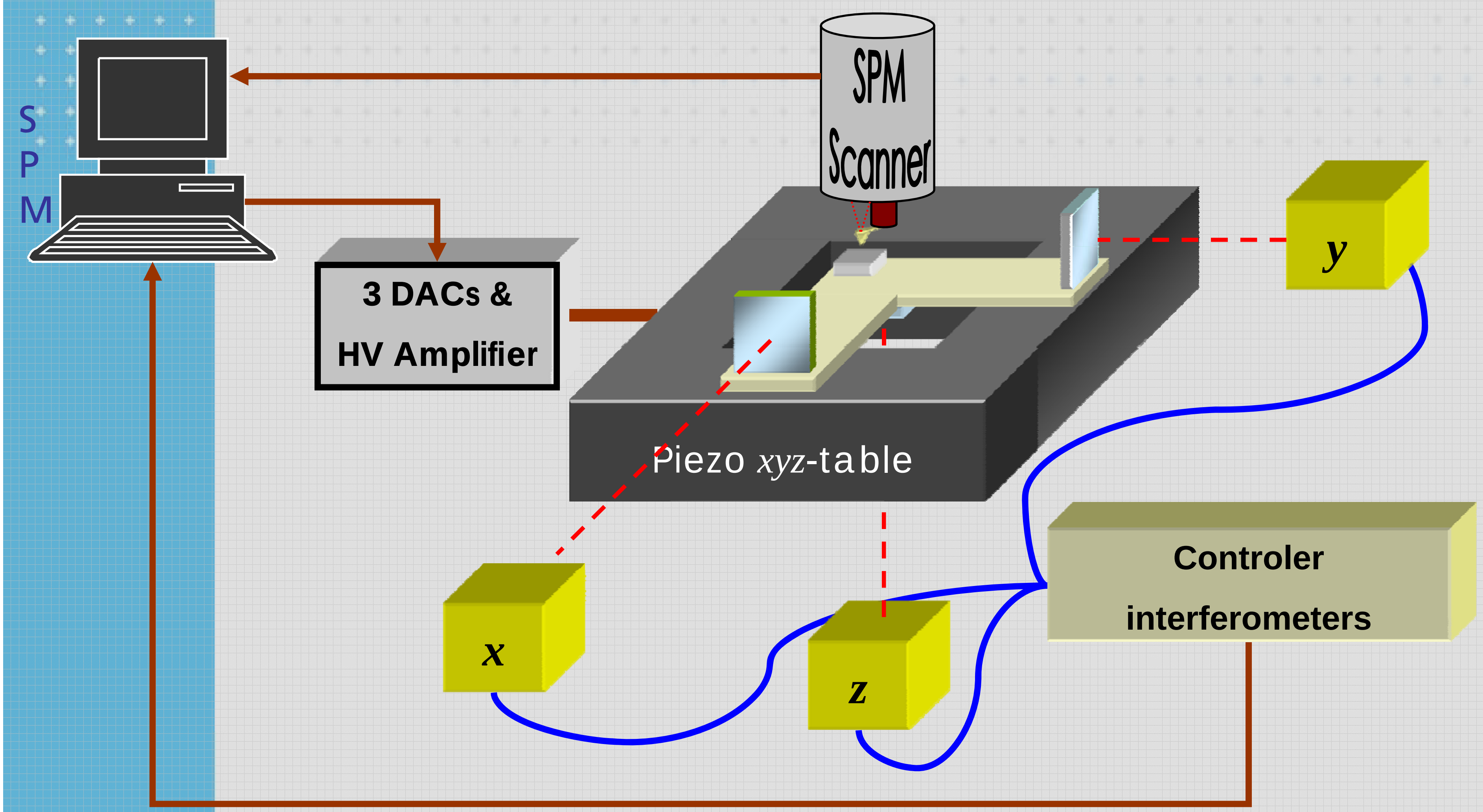


Principle Scanning Probe Microscope

- Tactile probing system
- Probe dimension 10-100 nm
- Surface profiles obtained by scanning
- Small measurement volumes
- Slow measurement process



Principal metrological SPM



NMi VSL Metrological SPM

Properties:

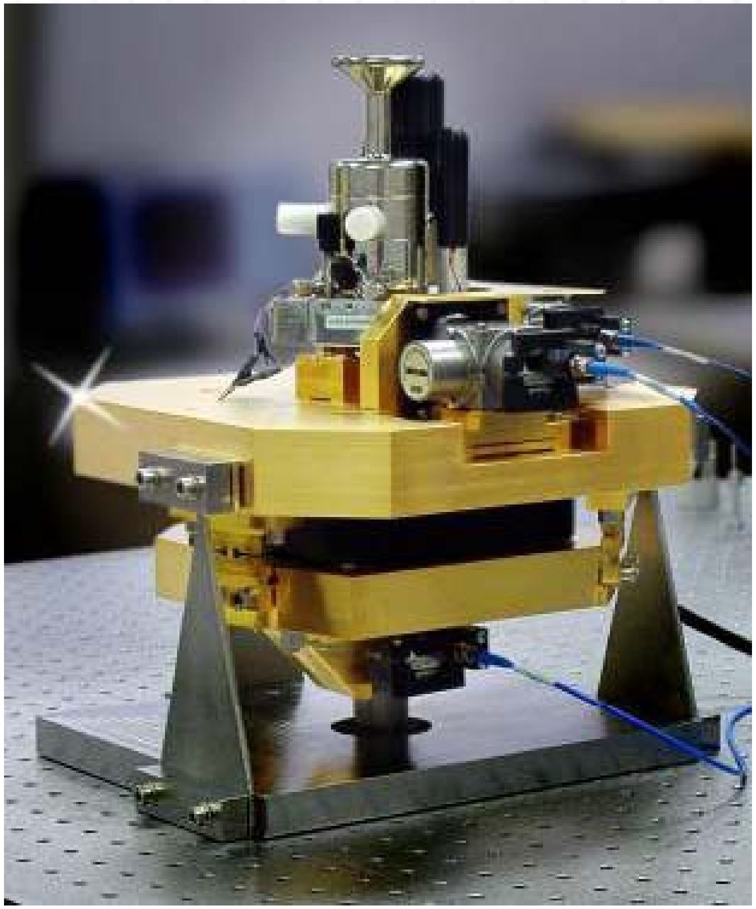
Range: 100 μm x 100 μm x 20 μm

Four pass 3-axes laserinterferometer system

Minimized thermal load

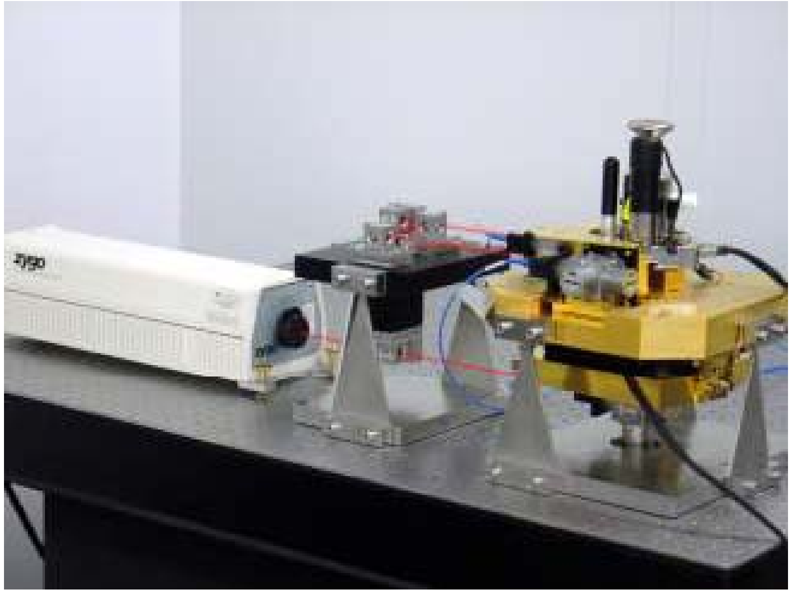
Maximized thermal inertia

Uncertainty: 1 nm



SPM set-up in the lab

- Separate foundation
- Heavy granite base
- Continuous environmental control and vibration registration



Uncertainty analysis

Sources of measurement uncertainty

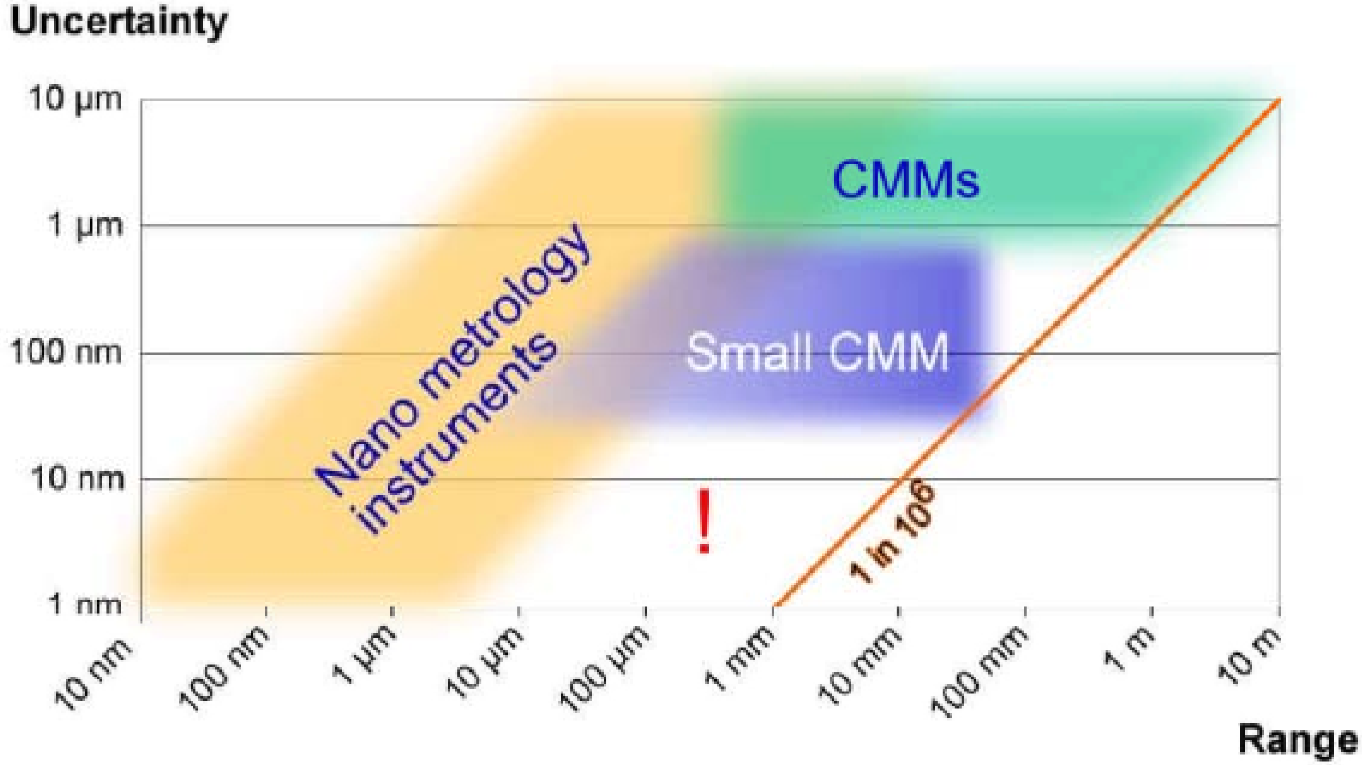
- Geometrical
  - Squareness errors
  - Mirror flatness deviations
  - Abbe error from parasitic rotations of stage
- Thermal
  - Linear expansion
  - Deformations
- Vibrations
  - Seismic
  - Acoustic

- Interferometers
  - Laser wavelength
  - Resolution
  - Refractive index
    - Air temperature
    - Air pressure
    - Relative humidity
    - CO_2 level
  - Alignment
    - Linear errors (angles of mirrors etc.)
    - Non-linear errors from polarization mixing
  - Residual dead path

- Tip-sample interaction
  - Tip shape
  - Tip wear
  - Sample deformation
  - Cantilever twist
  - Cantilever sensitivity
- Electronics
  - Drift
  - Noise

2D and 3D instrumentation

'Metrology Gap'



NPL

Long Range SPM

- Range of 1 mm x 1 mm x 1 mm,
- Measurement uncertainty 1 nm

Nano Coordinate Measuring Machine

- Truly 3D
- Measurement of i.e MEMS structures
- Novel Design

•Volume 50 x 50 x 4 mm, uncertainty <25 nm (3D)

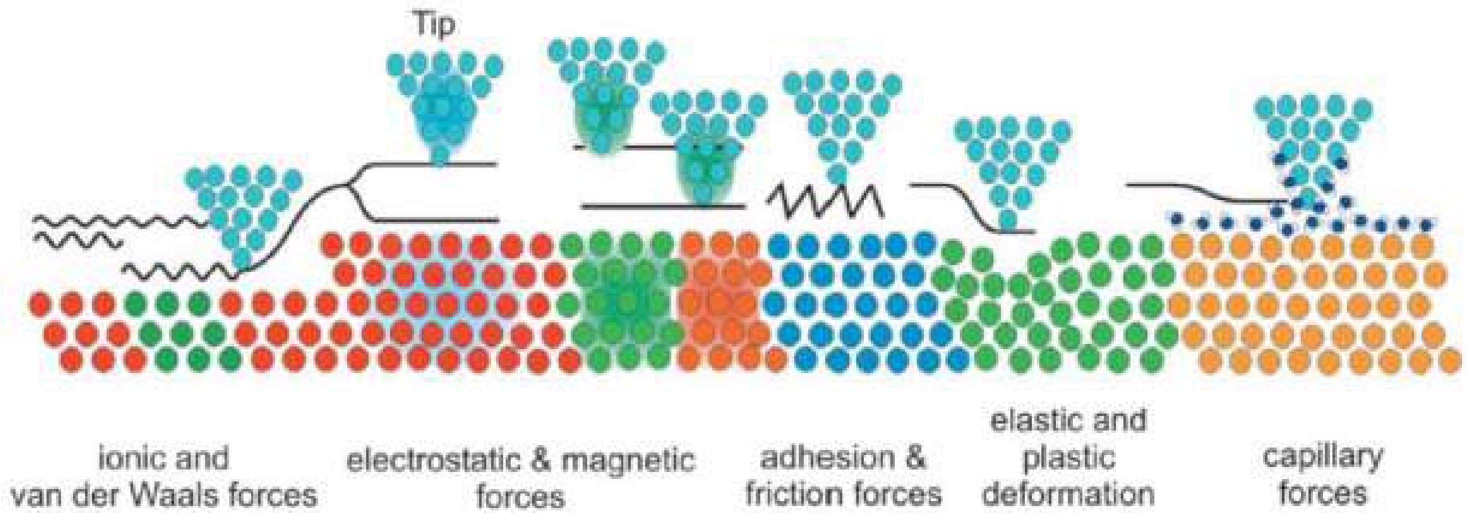
Elastically guided z-axis

Probe Sample Interactions

Why ?

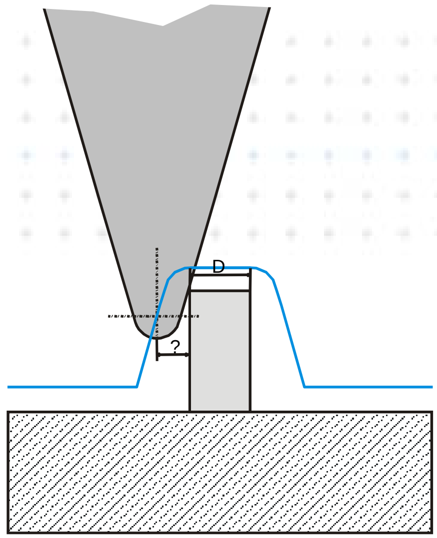
- SEM, SPM, Scatterometry – 3 main nanometrology tools – widely used……but reaching limitations: perturbation by the tool now similar to magnitude of measurand.

Probe Sample Interactions



- Physical Interaction
- Tip shape
- Cross validation SEM, STM, Scatterometry

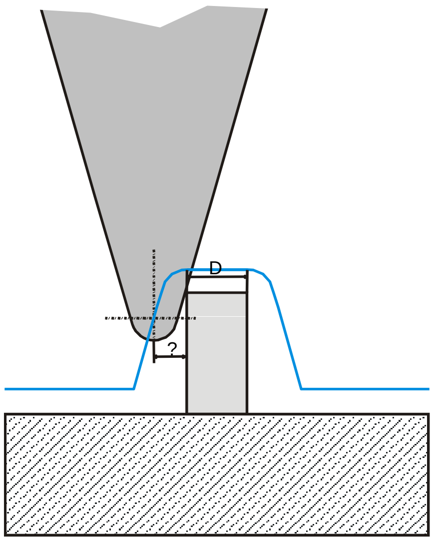
Probe Sample Interactions



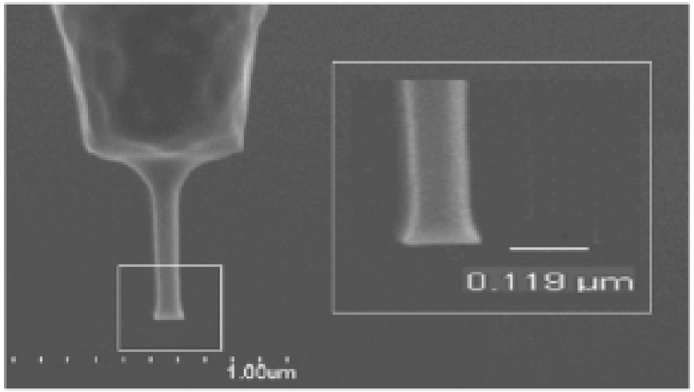
Conventional SPM probe

- Physical Interaction
- Tip shape
- Cross validation SEM, STM, Scatterometry

Line width probe

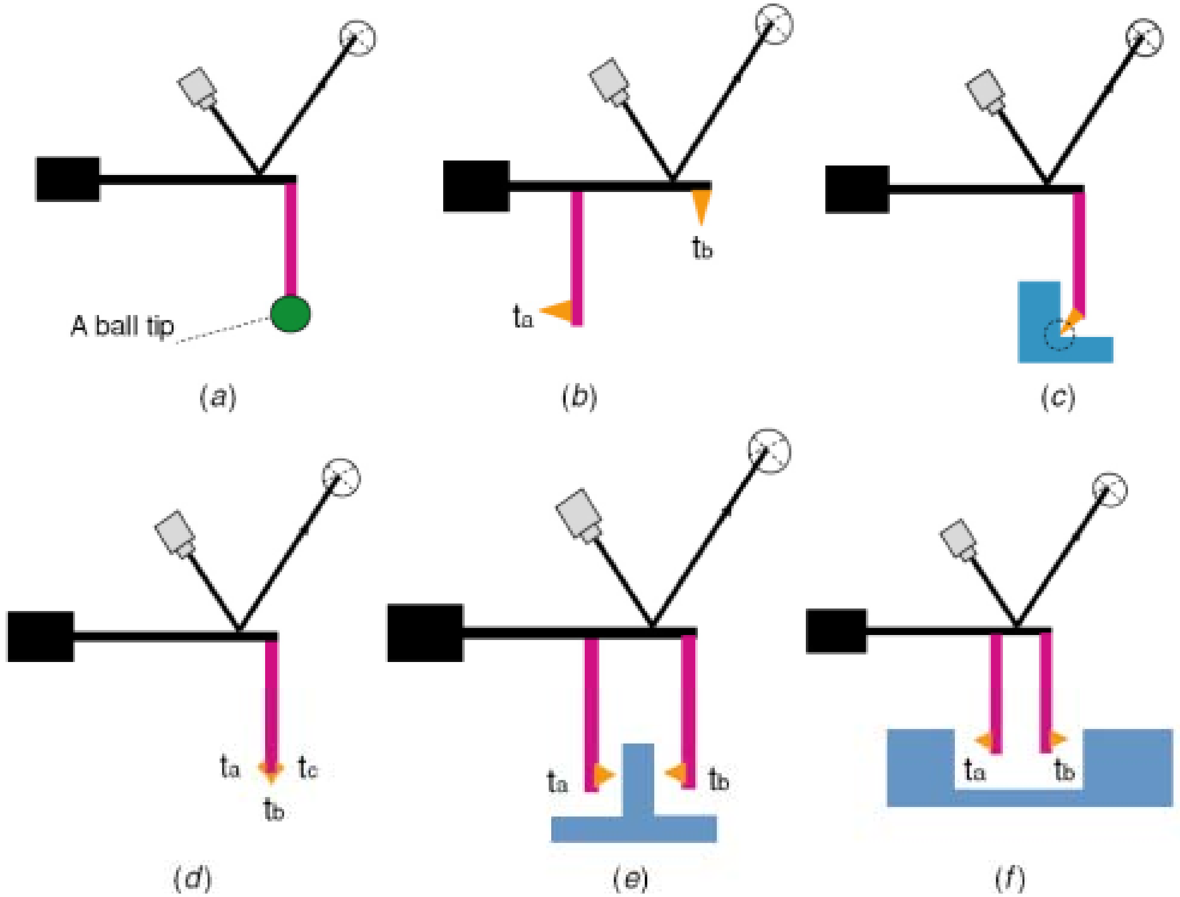


Conventional SPM probe



N.G. Orji et al, Meas. Sci. Techn. 18, 2007

Various probing concepts



G. Dai et al, Meas. Sci. Techn. 18, 2007

Nano scale pitch artefacts

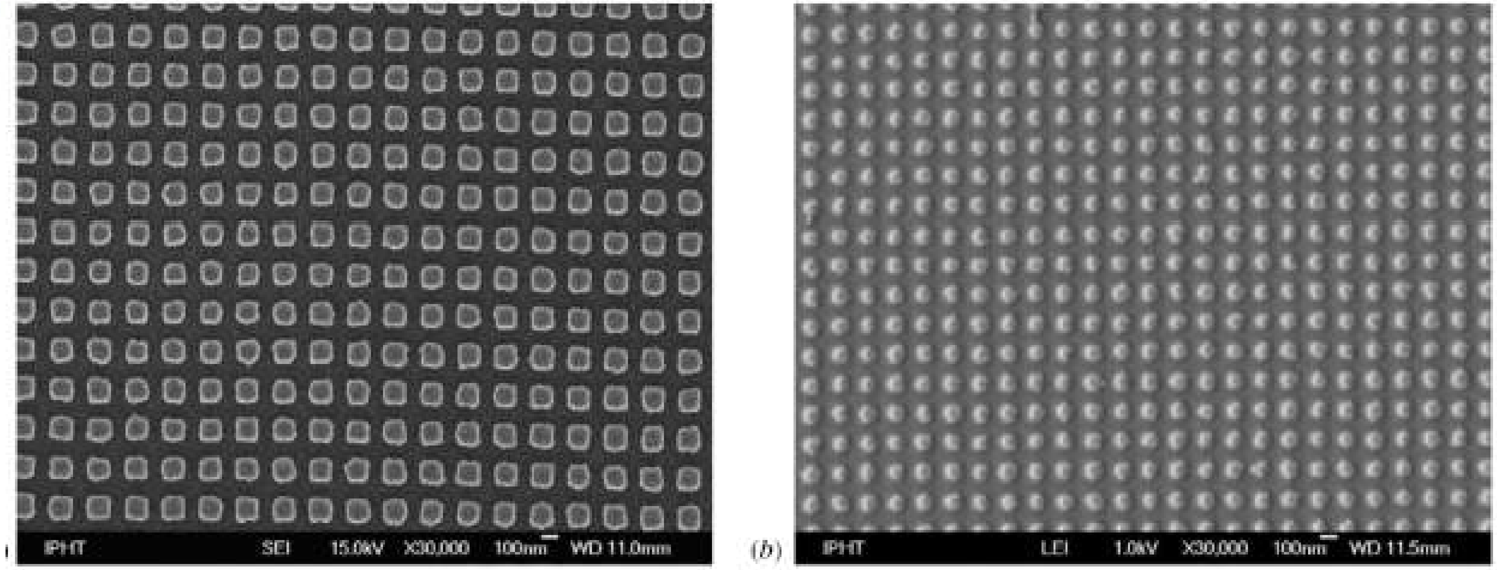
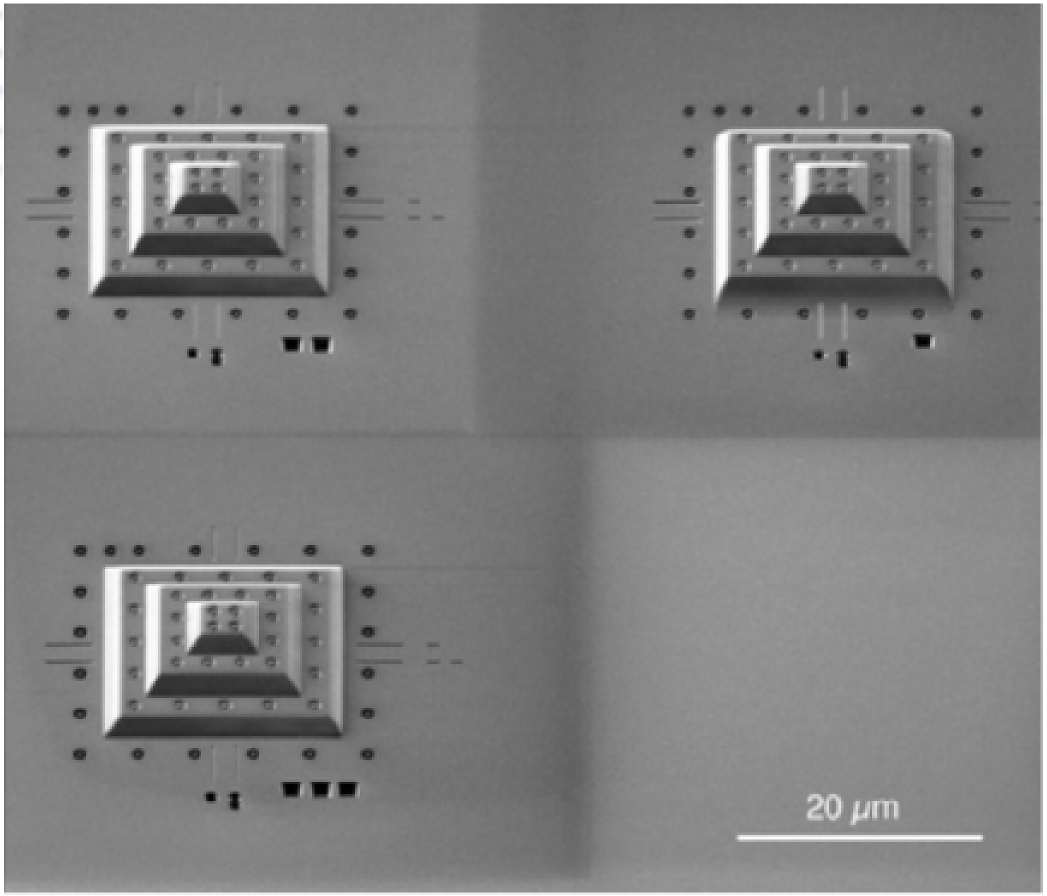


Figure 4. (*a*) Details of the 230 nm pitch cross grating and (*b*) 160 nm pitch cross grating.

U. Huebner et al, Meas. Sci. Techn. 18, 2007

3D calibration artefact



M. Ritter et al, Meas. Sci. Techn. 18, 2007

Displacement metrology at the nanometer scale

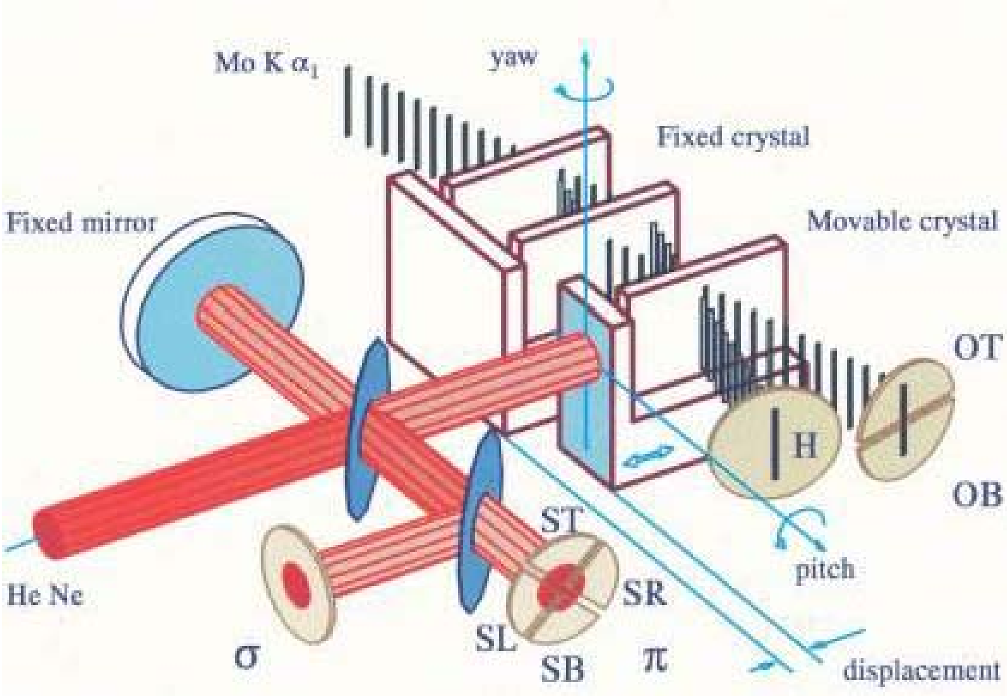
Objective

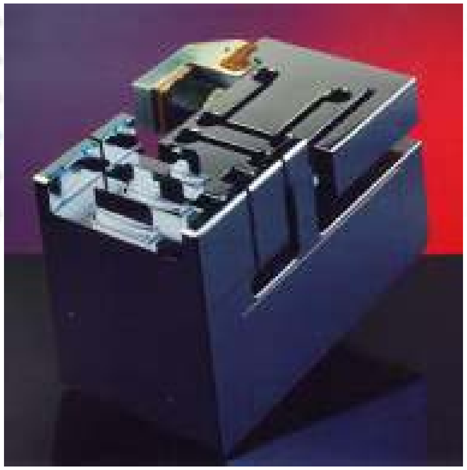
- Traceability at resolutions far beyond the nanometer

Possibilities

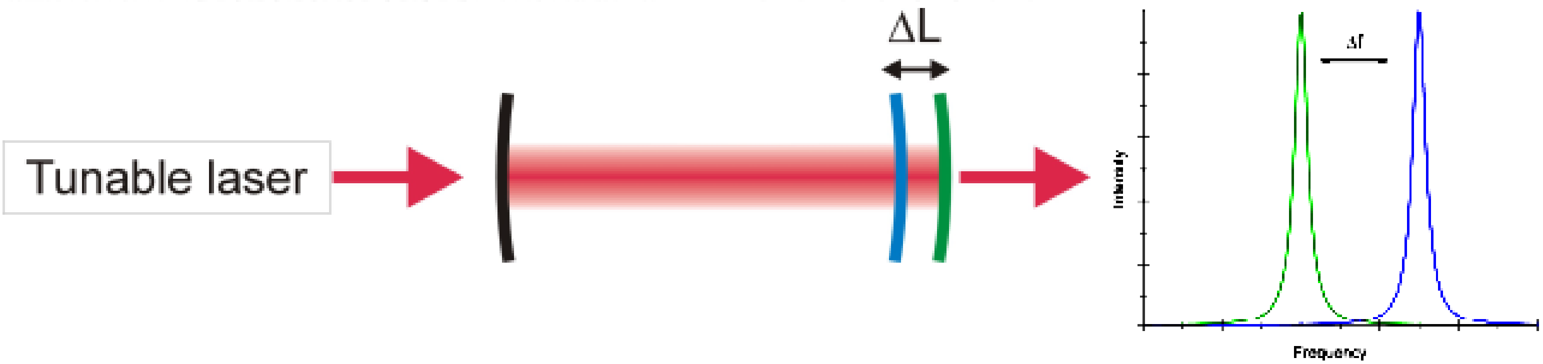
- X-ray interferometry
- F-P interferometry
- Atomic lattice (Silicon)

X-ray interferometry





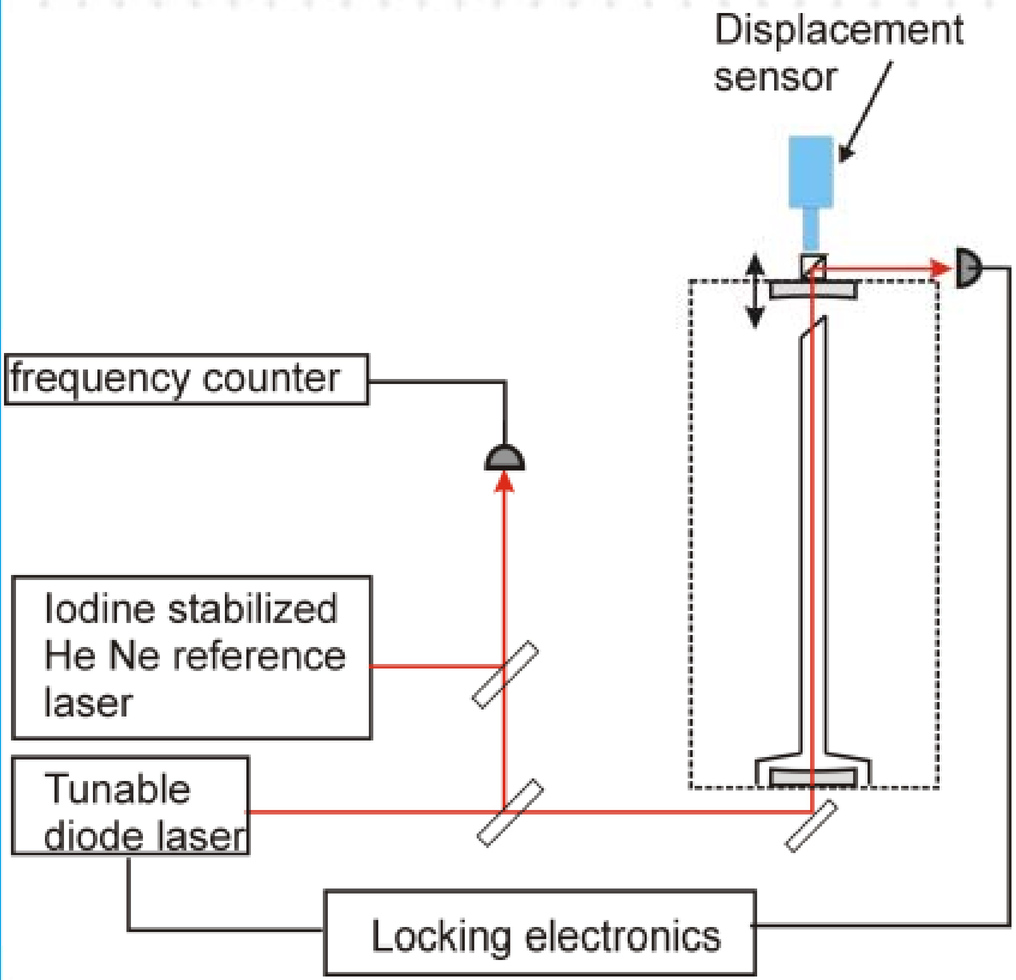
Metrological Fabry-Perot



Tunable diode laser locked to the FP-resonator.
Frequency measurement by direct comparison to the primary standard yields the displacement:

Displacement ΔL: $$\boxed{\Delta L = \left(\frac{f_{DL} - f_S}{FSR}\right) \cdot \frac{\lambda}{2}}$$

Principle of operation



Resonator:
L = 150 mm

Free Spectral Range = 1000 MHz

Sensitivity = 1000 / 316 = 3.2 MHz/nm

$$\Delta L = \left(\frac{f_e - f_s}{FSR} \right) \cdot \frac{\lambda}{2}$$

Realization

Fabry-Pérot interferometer with capacitive sensor

Conclusions

- The role of metrology
- International Metrological infrastructure
- Activities of European NMis in nanometrology
  - Roadmap & challenges
  - Trends