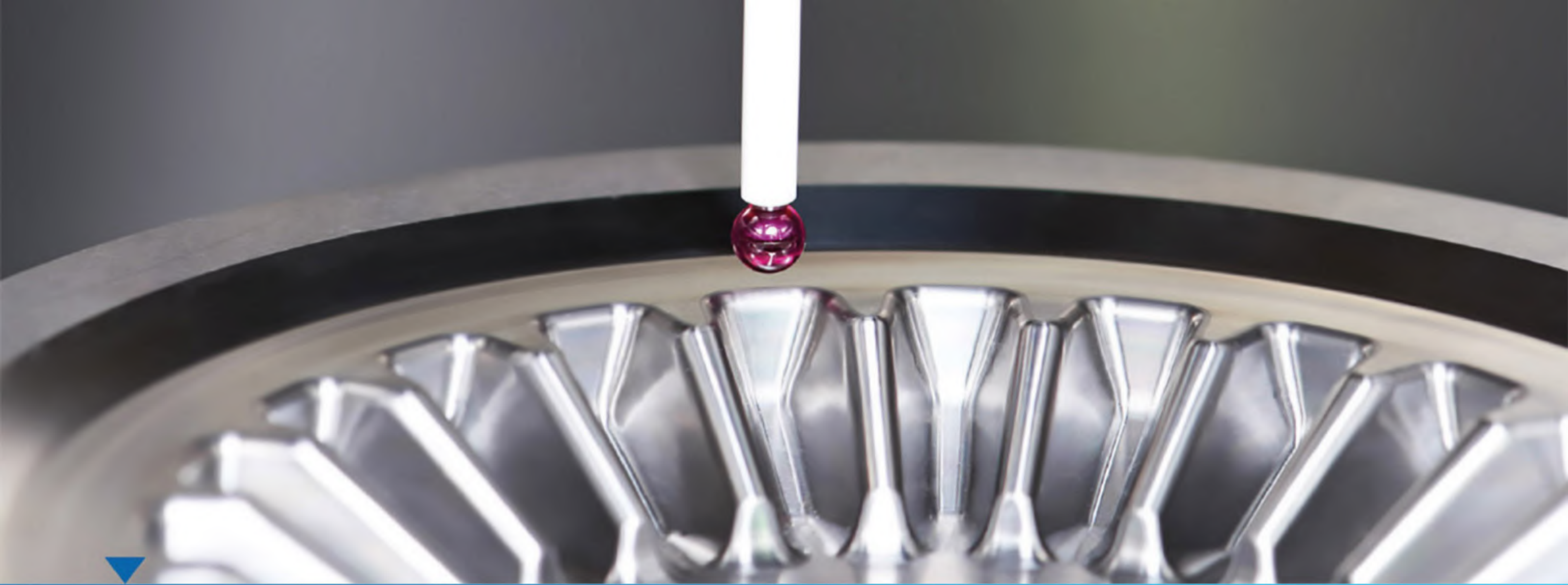


Advanced Scanning Probe Microscopy

Petr Klapetek, Český metrologický institut, Okružní 31, 638 00 Brno

CZECH
METROLOGY
INSTITUTE

SPM – scanning probe microscopy

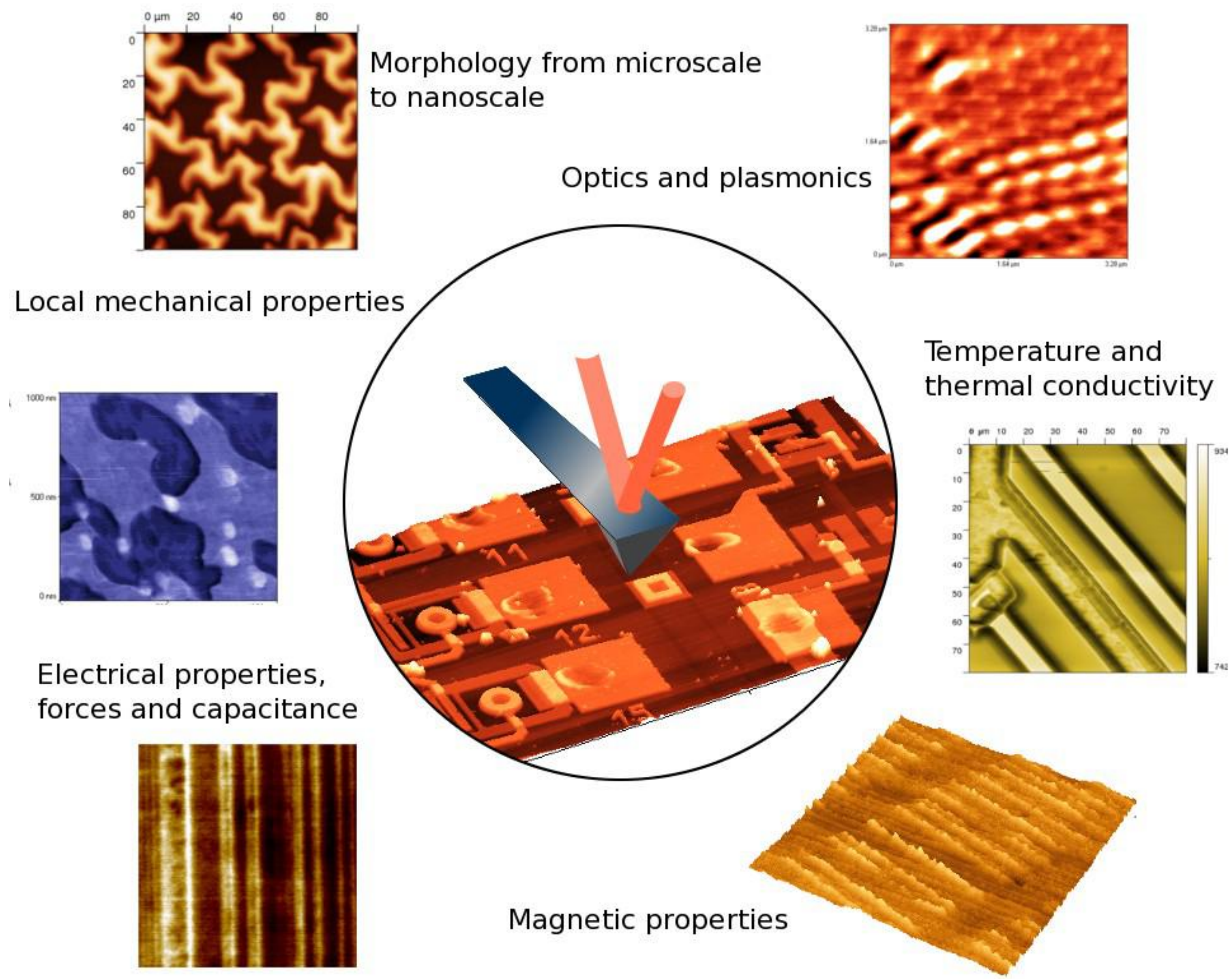
Scanning probe microscopy

Versatile surface measurement technique:

- no sample preparation
- many quantities achievable
- simple principle and construction
- cost effective

Novel regimes emerging quickly.

What about quantitative aspects?



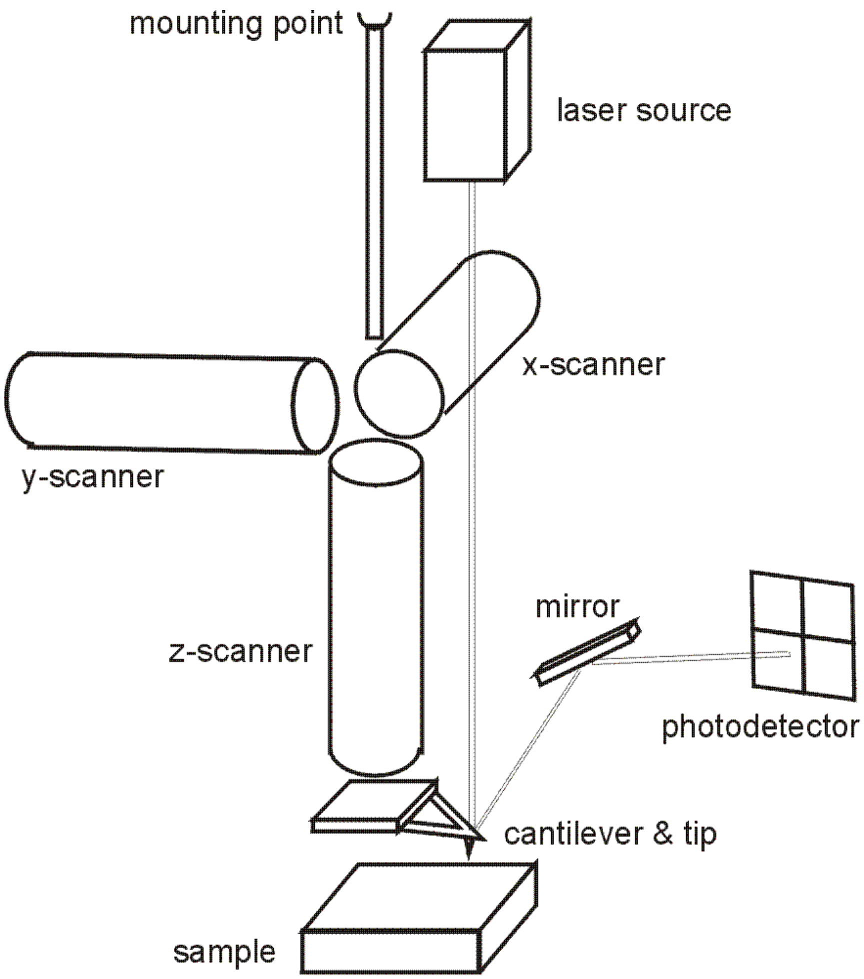
SPM – a simple device

Key concept
Small probe scanning close to surface

Small probe …
sharp tip

… scanning …
scanners, piezoelectric materials, positioning sensors

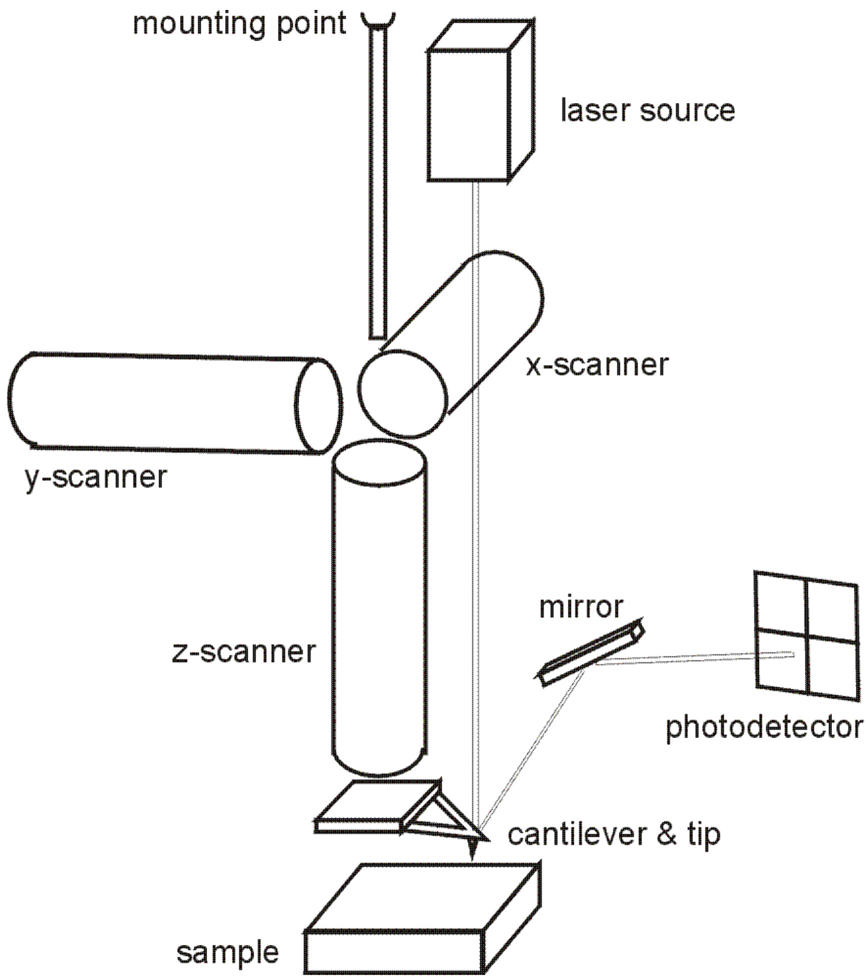
… close to surface
feedback loop, optical pickup, self-sensing probes.



SPM – a simple device

Key concept
Small probe scanning close to surface

Small probe …
sharp tip



SPM – a simple device

Key concept

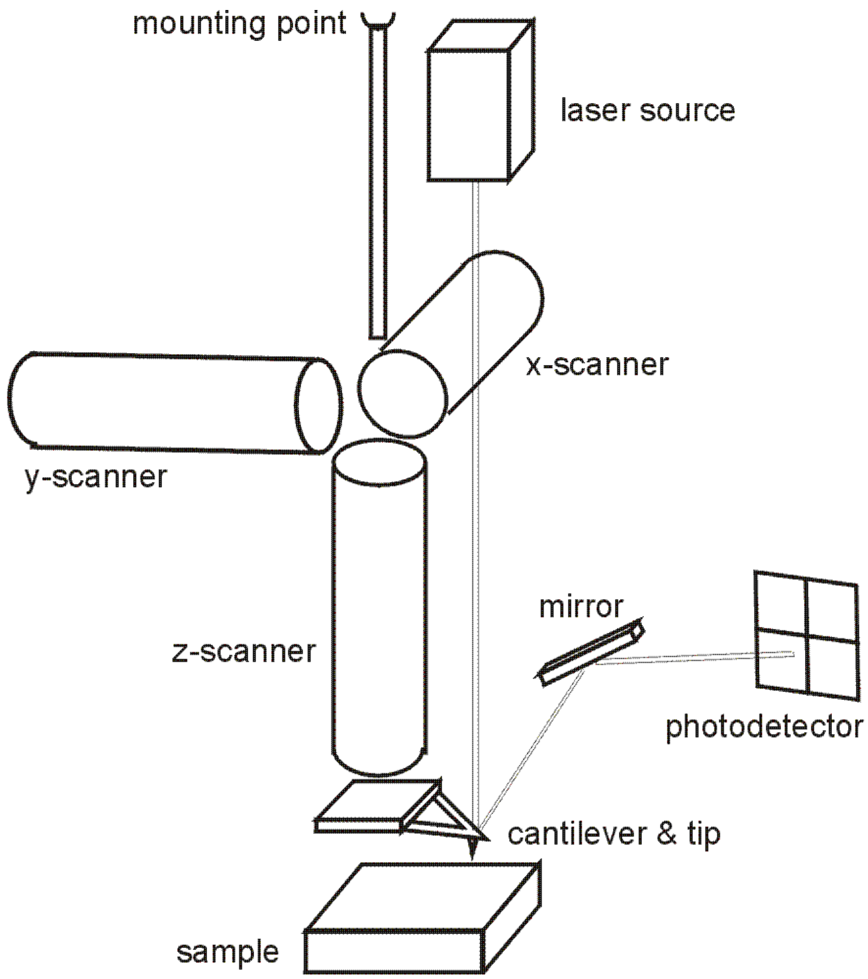
Small probe scanning close to surface

Small probe …

sharp tip

… scanning …

scanners, piezoelectric materials, positioning sensors



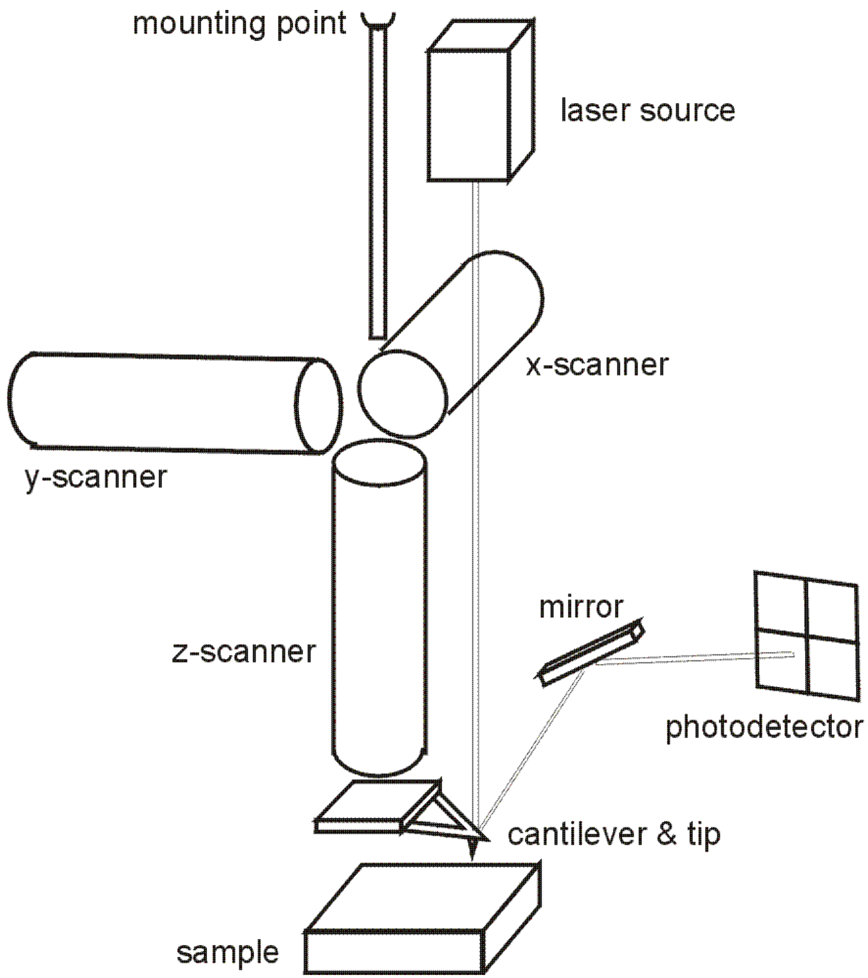
SPM – a simple device

Key concept
Small probe scanning close to surface

Small probe …
sharp tip

… scanning …
scanners, piezoelectric materials, positioning sensors

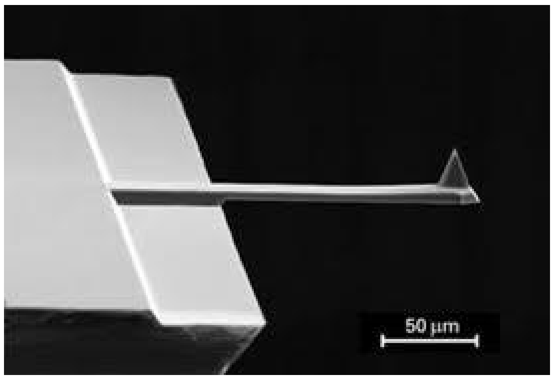
… close to surface
feedback loop, optical pickup, self-sensing probes.

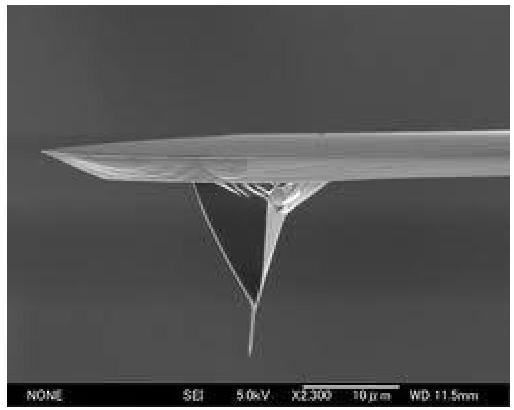


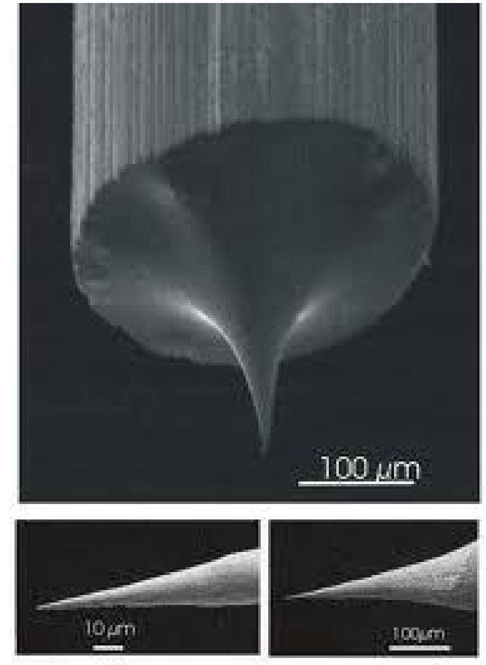
SPM probes

Many probe types:

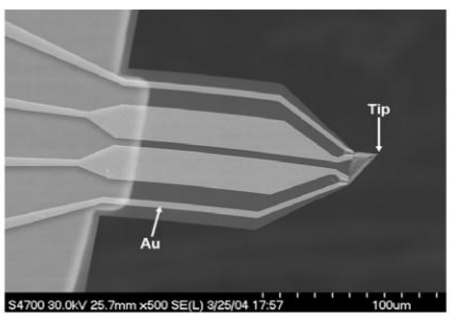
- tip geometries (super-sharp, spherical, ...)
- functionalisation (electrical, magnetic, …)
- stiffness (contact, tapping, ...)



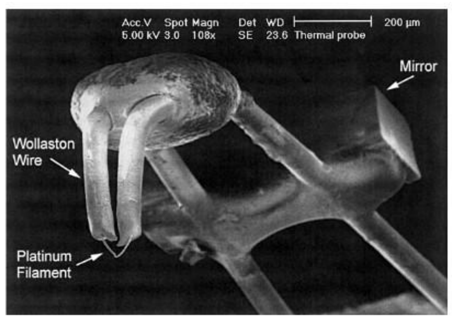


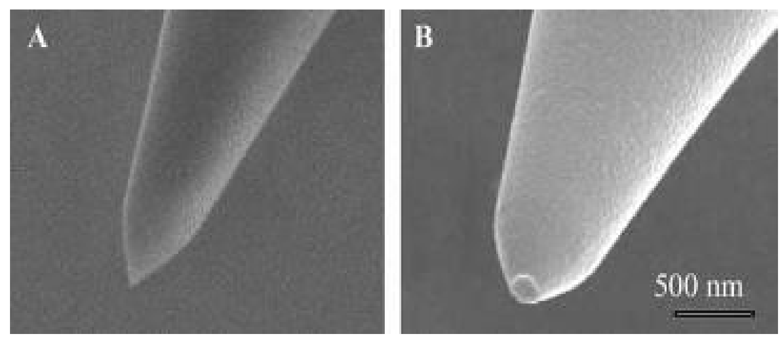


(a)



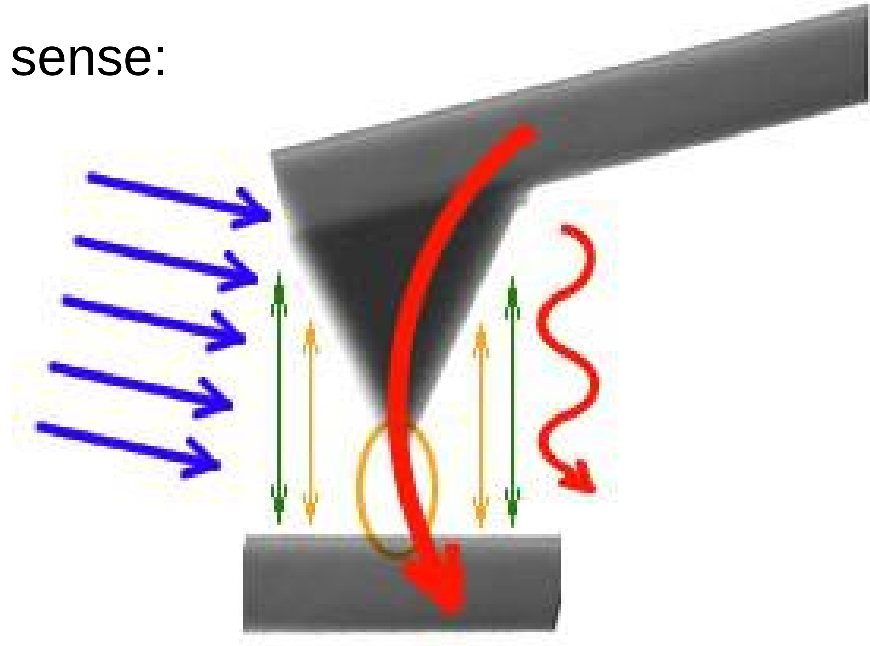
(b)





Probe-sample interaction

There are many interactions that a probe could sense:



- inter-atomic forces (AFM)
- electrostatic field (AFM, EFM, KPFM, SCM)
- magnetic field (MFM)
- temperature and heat transfer (SThM)
- electromagnetic field distribution (SNOM, SMM)

We want to make everything quantitative!

Czech Metrology Institute

National metrology institute of the Czech Republic

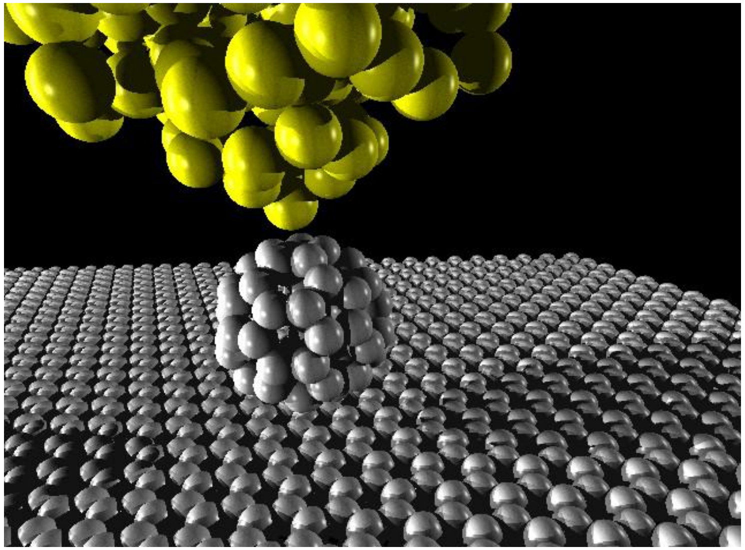
fundamental metrology: maintenance and development of national standards, R&D in metrology

dissemination of units: top level calibration of standards and measuring instruments

legal metrology: regulated sphere, type approvals of legal metrology instruments...

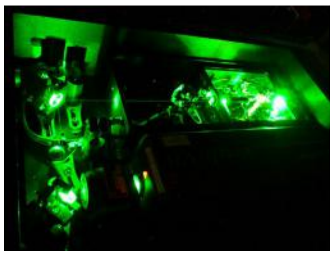
Department of nanometrology: CMI Regional Branch Brno.

- scanning probe microscopy methods
- numerical modeling at nanoscale and microscale
- advanced data processing algorithms development
- providing metrological traceability
- methodology, uncertainty analysis



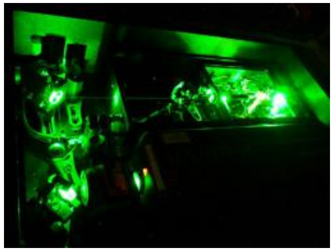
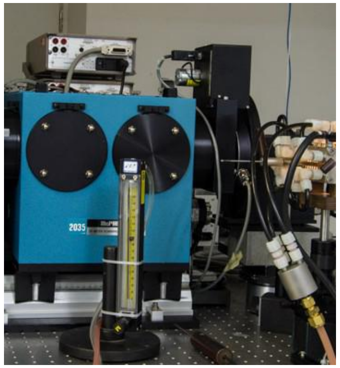
Metrology

Metrology

Mass	**Length**	**Photometry**	**Flow**	**Pressure**	**...**
					
standards	standards	standards	standards	standards	standards
methods	methods	methods	methods	methods	methods
...	...	...	...	...	...

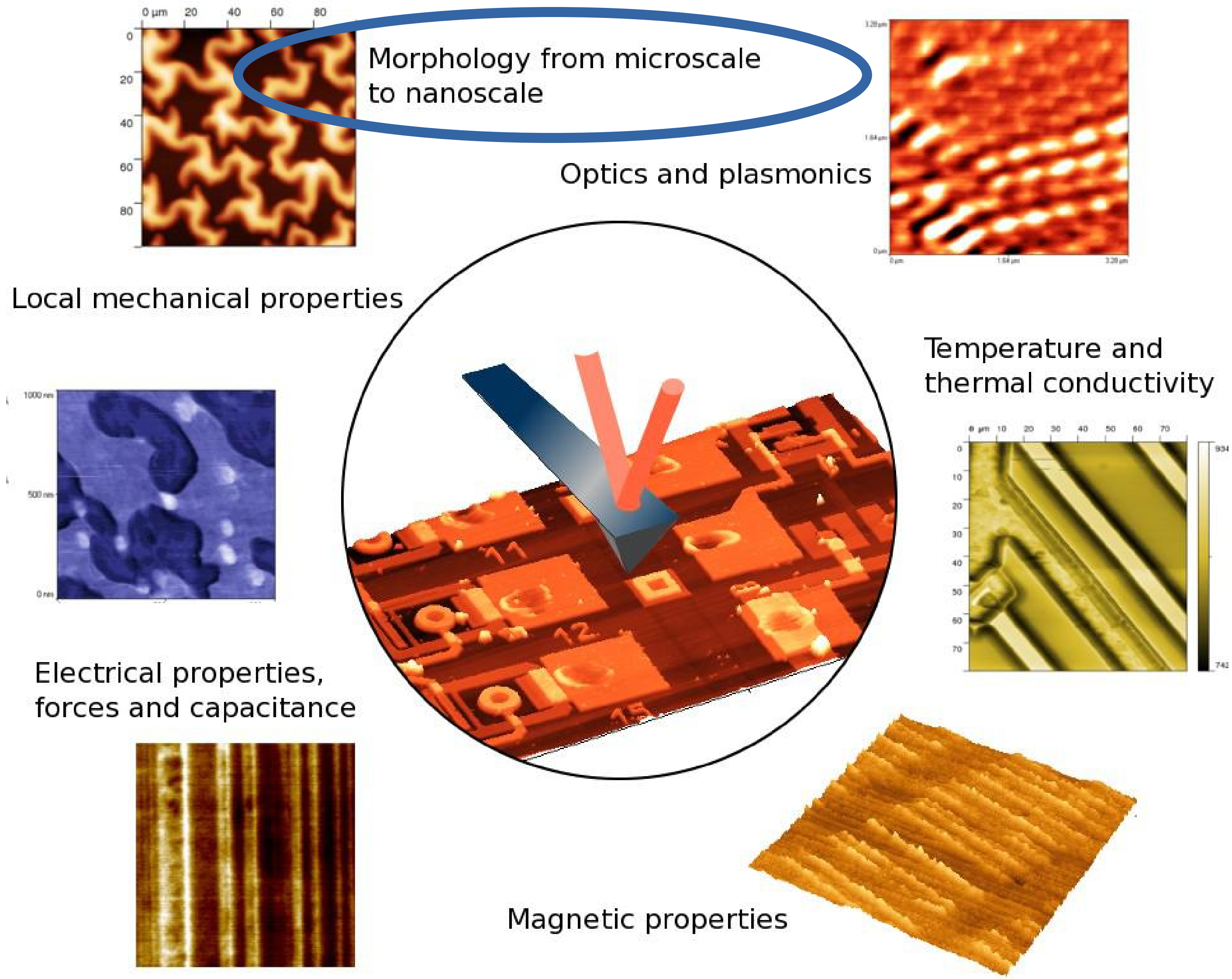
Nanometrology

Metrology

Mass	**Length**	**Photometry**	**Flow**	**Pressure**	**...**
					
standards	standards	standards	standards	standards	standards
methods	methods	methods	methods	methods	methods
...	...	...	...	...	...

Nanometrology

SPM – scanning probe microscopy

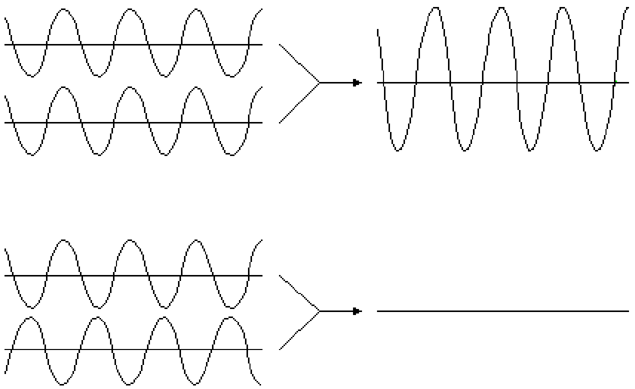


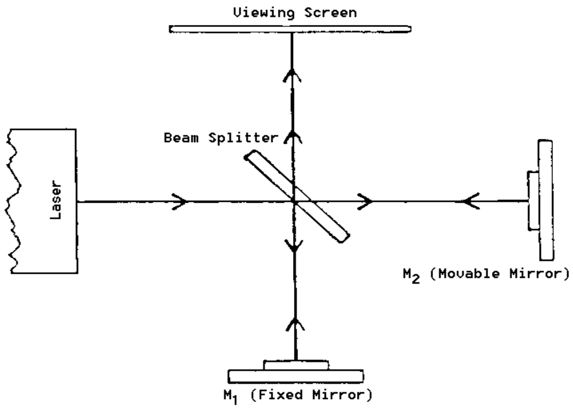
Metre definition:

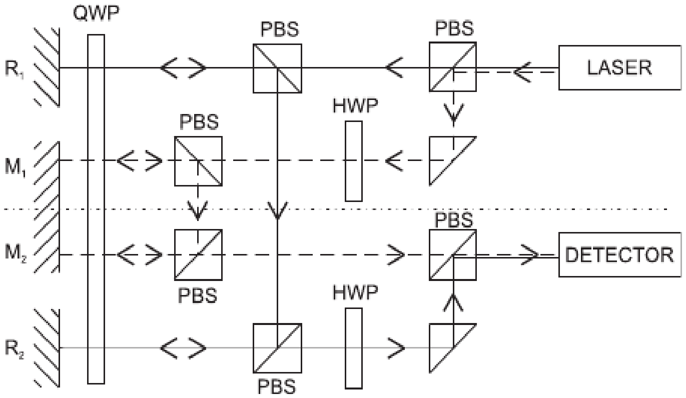
The metre, symbol m, is the SI unit of length. It is defined by taking the fixed numerical value of the speed of light in vacuum c to be 299792458 when expressed in the unit m·s−1, where the second is defined in terms of the caesium frequency ΔνCs.

Length measurements via lasers: laser interferometers

- based on monitoring the interference of monochromatic light of known wavelength
- range up to tens of meters
- resolution down to tens of picometers

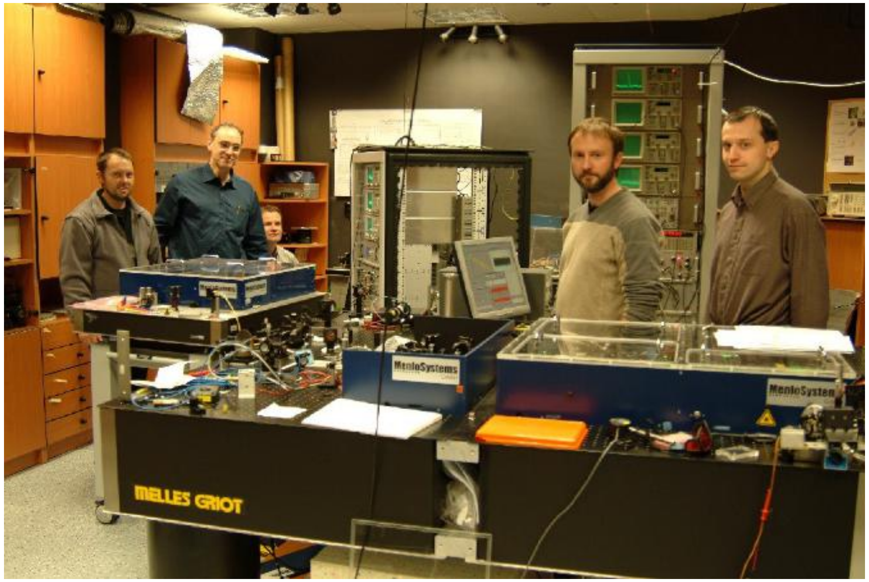






Traceability for dimensional measurements

State etalons of length: stabilised lasers



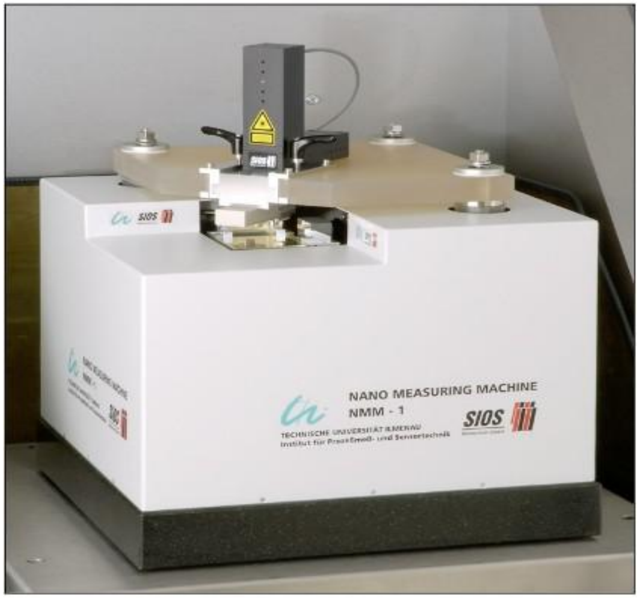
Calibration of wavelength of lasers used in **laser interferometers**.

Interferometers are then used to calibrate **other sensors**.

Going up: Large area SPMs

Interferometric calibration of positioning systems

- use of independent interferometer at calibration
- cheaper sensors used for routine operation
- time stability needs to be analyzed
- larger uncertainties



Positioning systems using interferometers

- direct traceability
- many effects can be compensated
- more expensive, more sensitive to disturbances

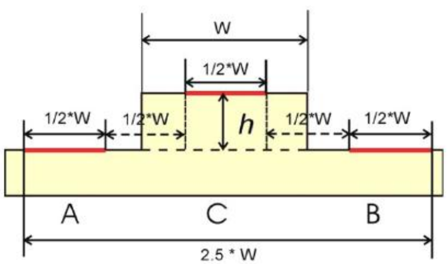
Calibration samples for dimensional AFM

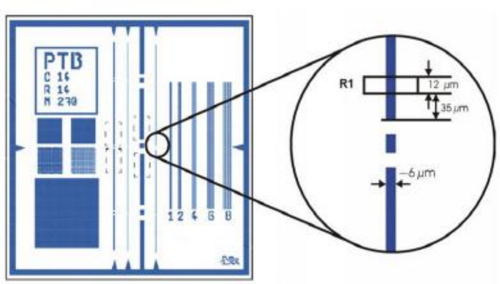
Grating: lateral scale calibration

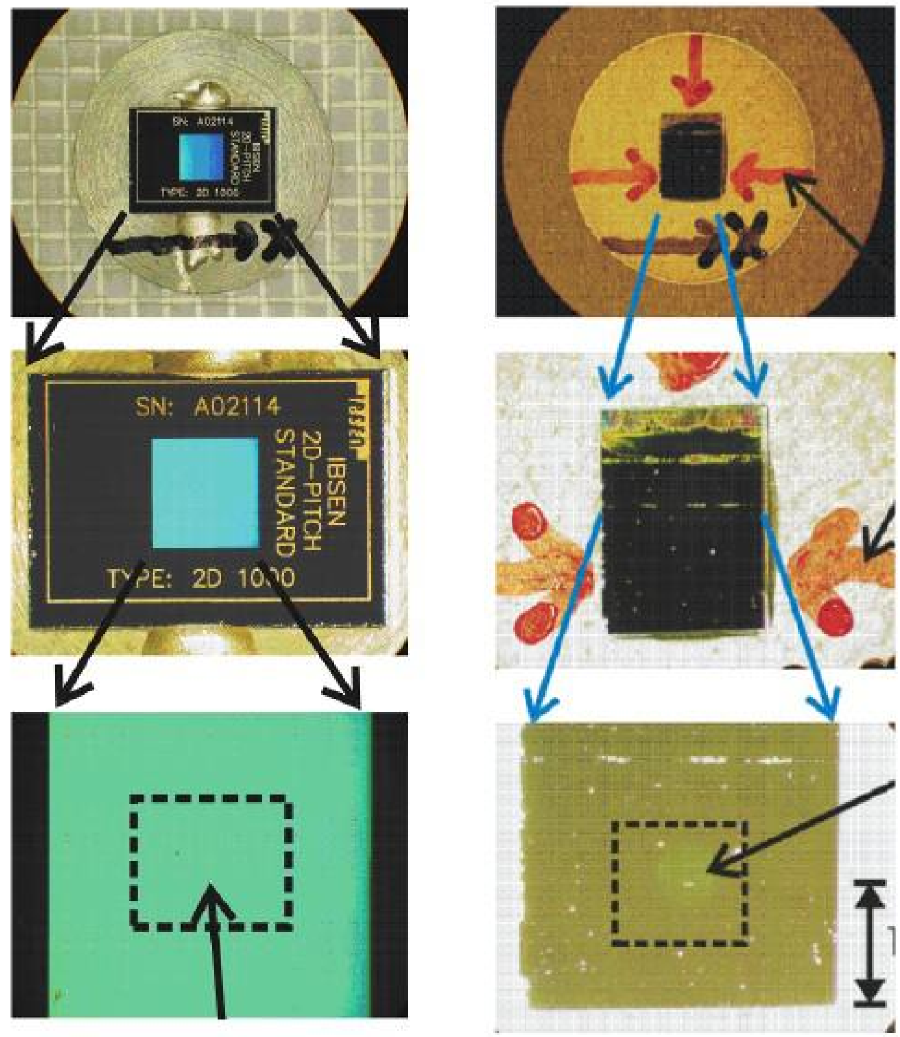
Step height: z-scale calibration

You can buy 1D and 2D gratings on many places.

The grating itself can be used as a step heigh standard (of a limited accuracy).



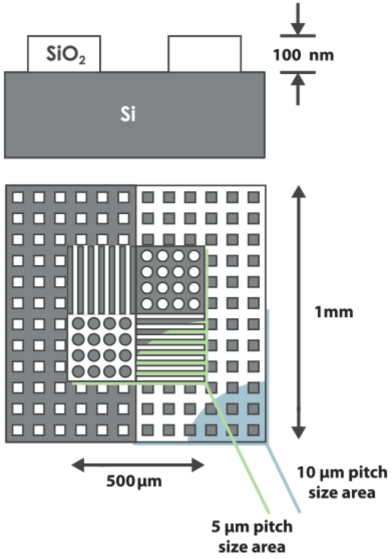


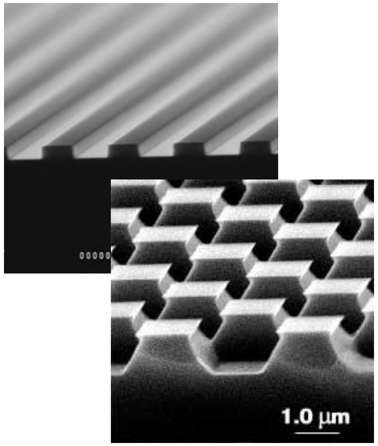


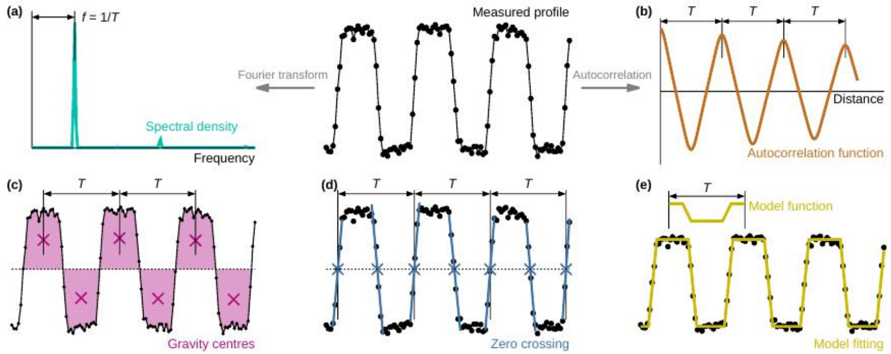
Calibration samples for dimensional AFM

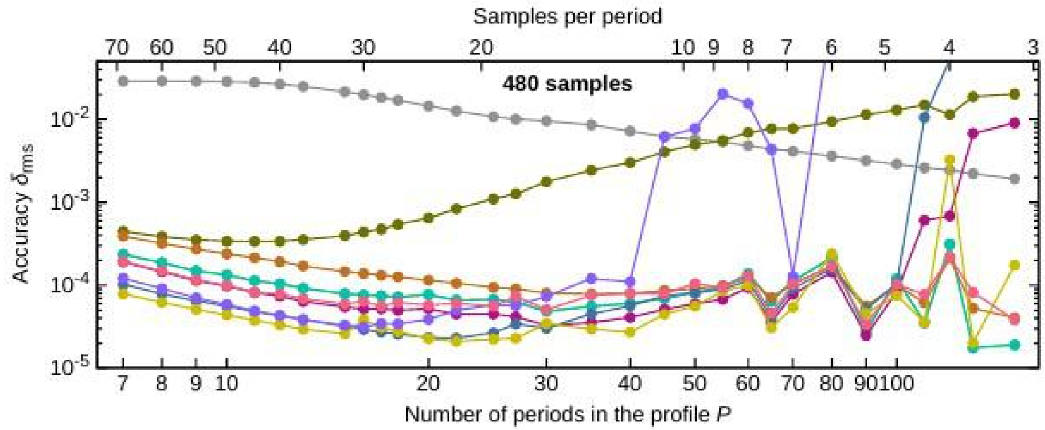
Grating: lateral scale calibration

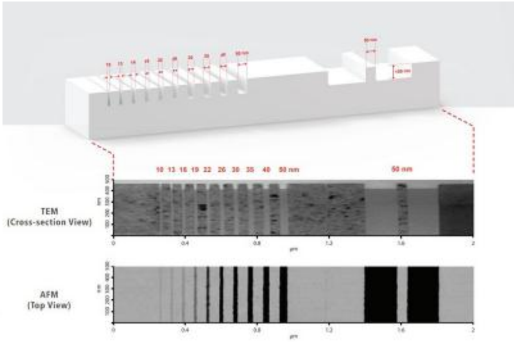
(Bruker, Tipsnano, Nanoandmore, ...)

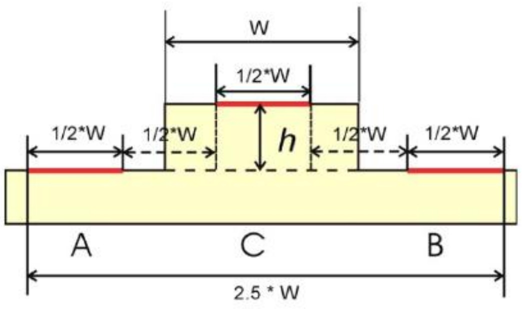












Step height: z-scale calibration

(Park Systems, Tipsnano)

Can be combined with grating.

Going up: Large area SPMs

How to perform and measure a nanometer motion over large scale?

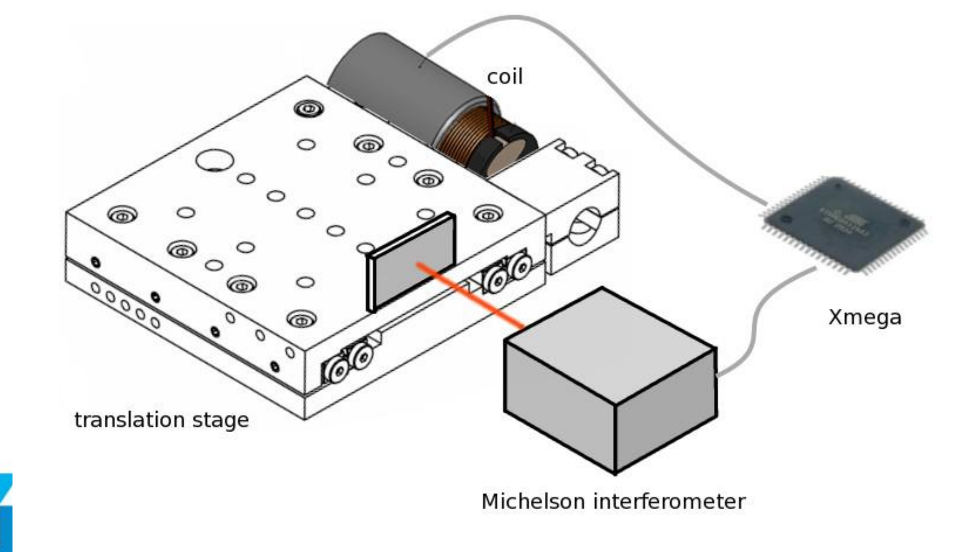
The simplest choice: piezoelectric material. However, this many disadvantages:

- small range (typically 10 microns per centimeter of actuator size)
- power demands (high voltage, large currents for fast changes)
- limited long term stability.

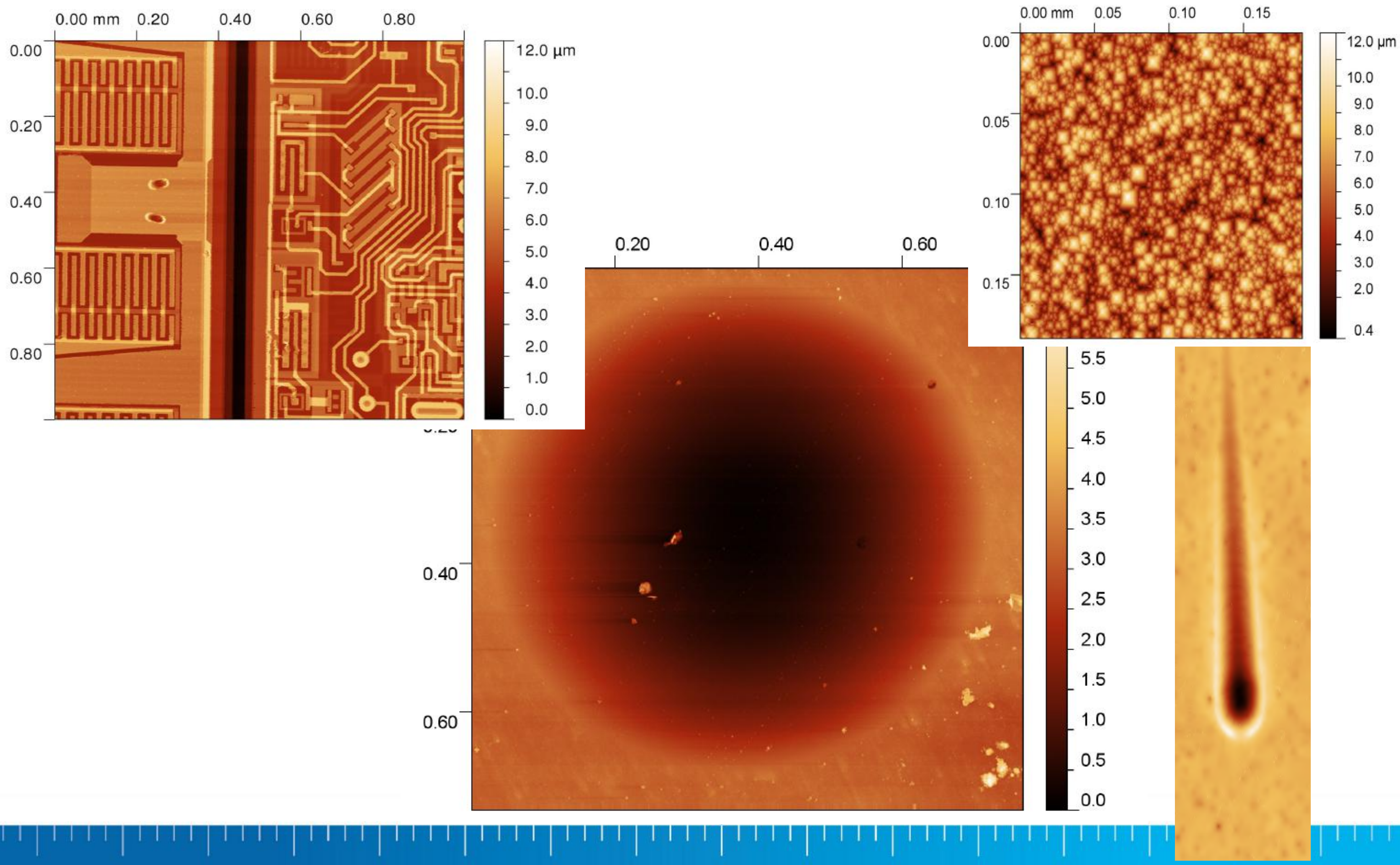
Good DA converter is necessary to be able to get both large scanning range and high resolution. This is a **voltage to position** transducer.

As an alternative, we can use

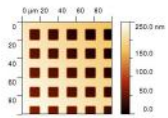
voltage-force transducer coupled to a high resolution sensor, interferometer.



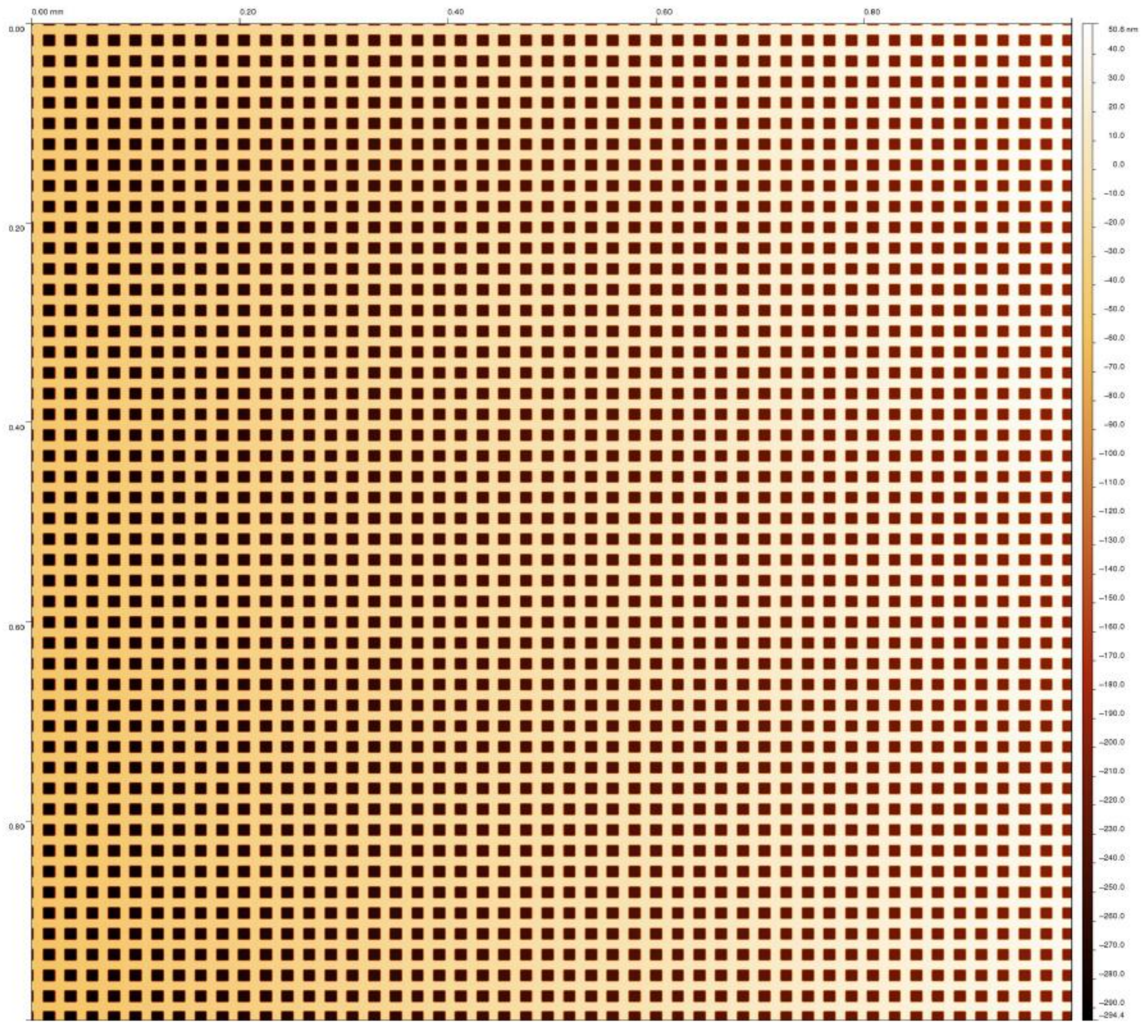
Going up: Large area SPMs



Going up: Large area SPMs



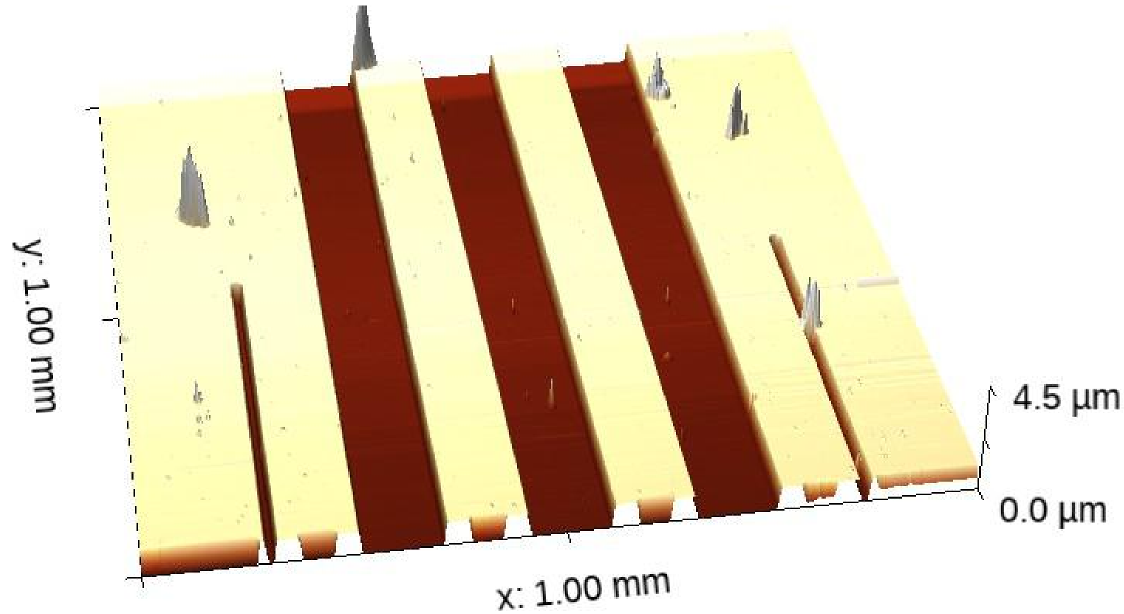
Typical SPM data

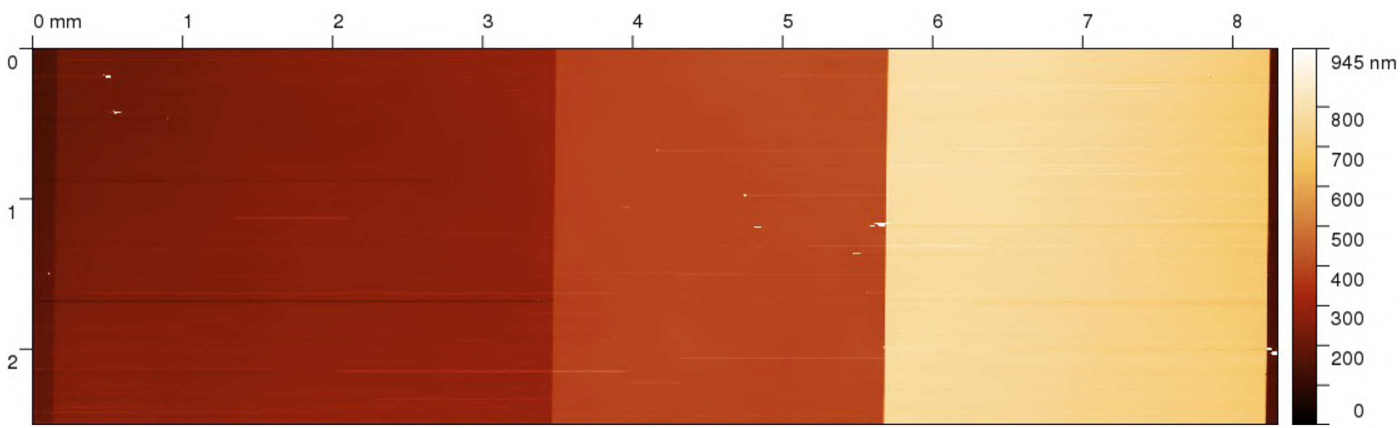


1x1 mm SPM data

Going up: Large area SPMs

Calibration of step height standards suitable for larger range measurment techniques (e.g. confocal microscopes).

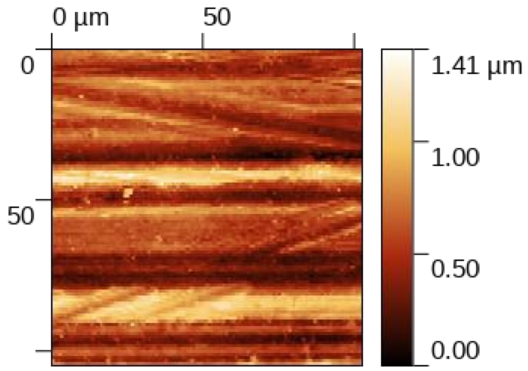


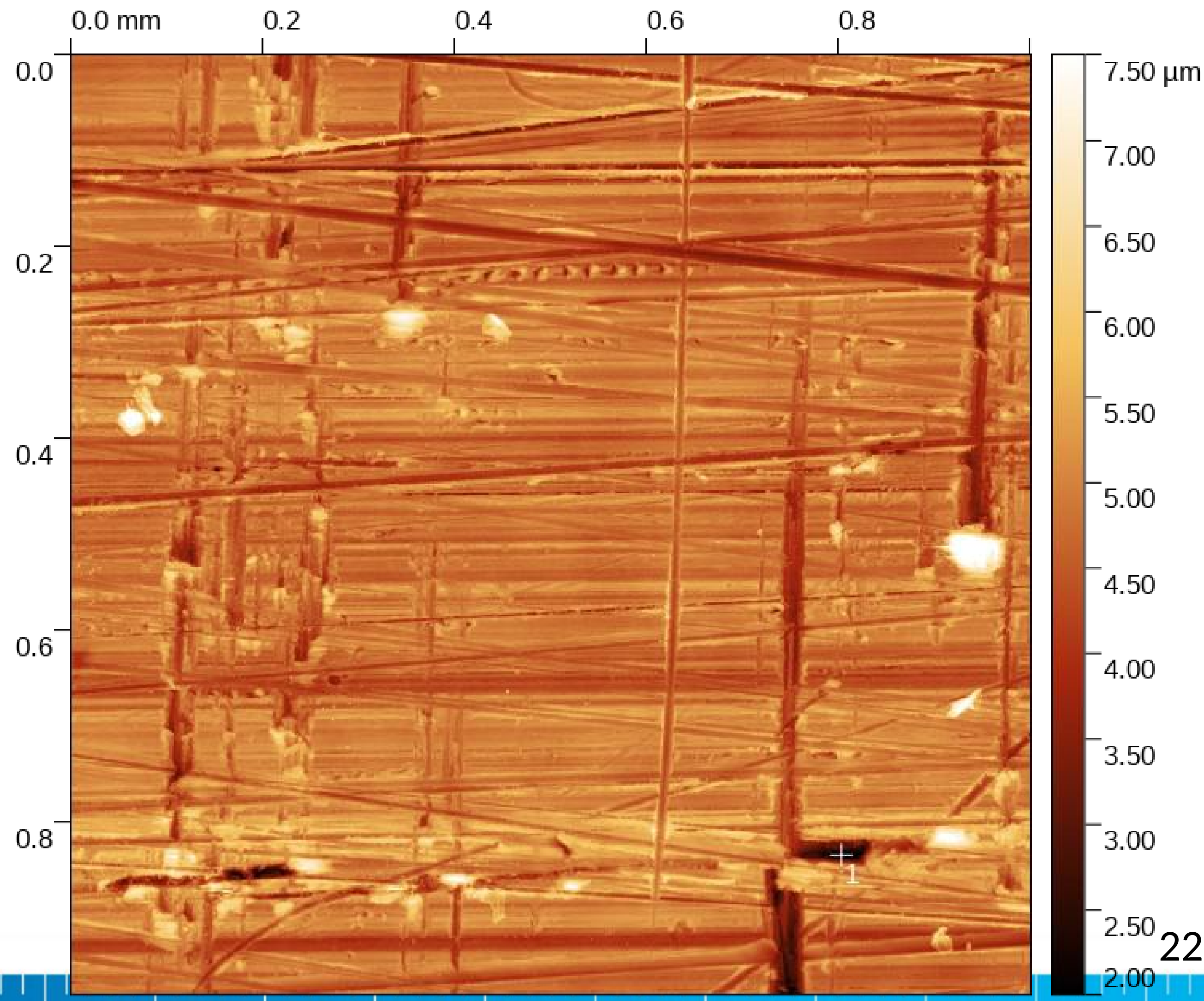


Monitoring thin film thickness variations over millimeter or centimeter areas.

Going up: Large area SPMs

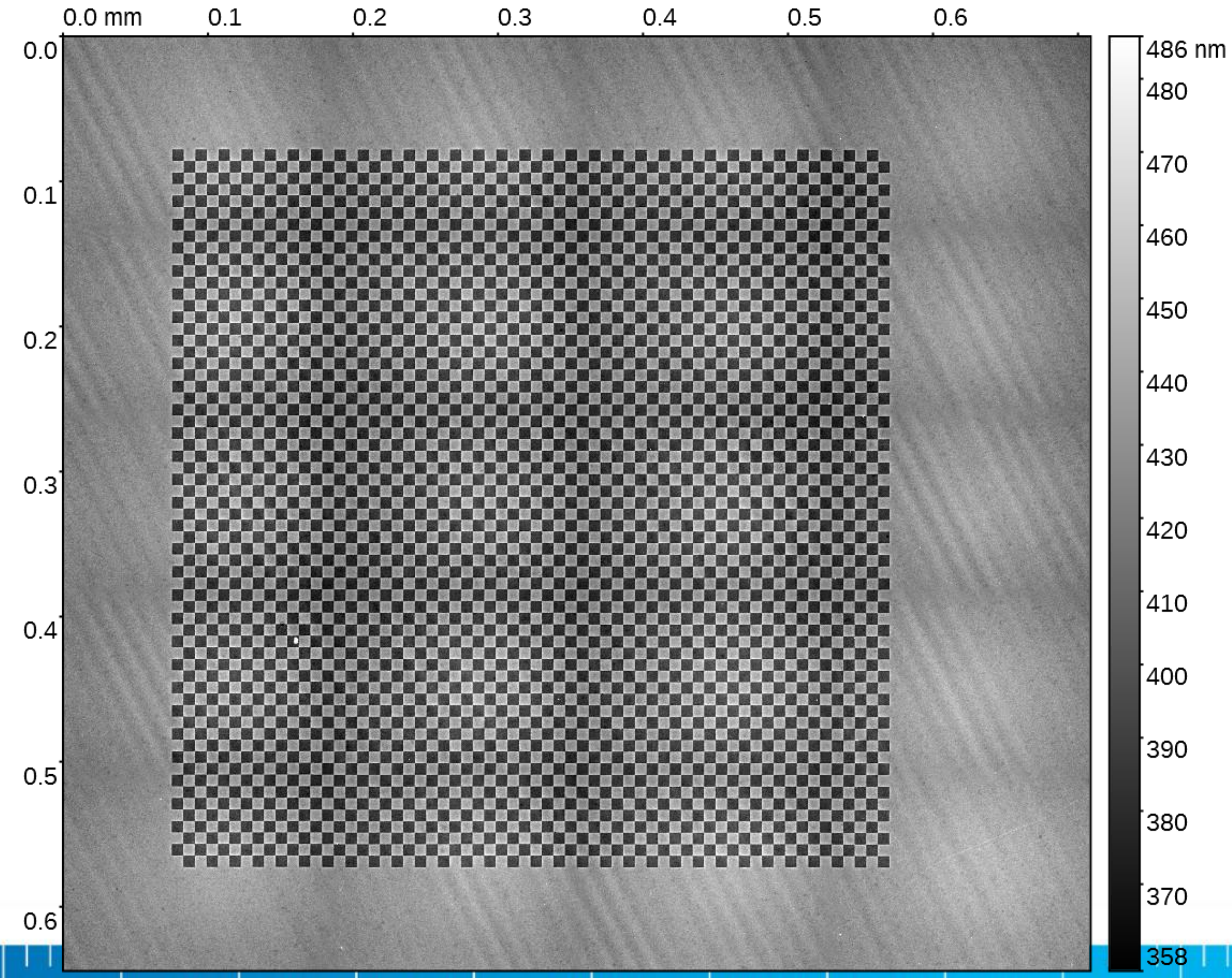
Roughness measurements on large areas: beyond the stylus measurements capabilities.





Going up: Large area SPMs

SPM has less systematic errors than optical techniques (e.g. confocal microscopy), is not sensitive to refractive index variations and less sensitive surface roughness.

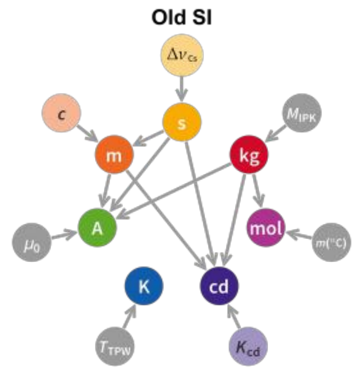


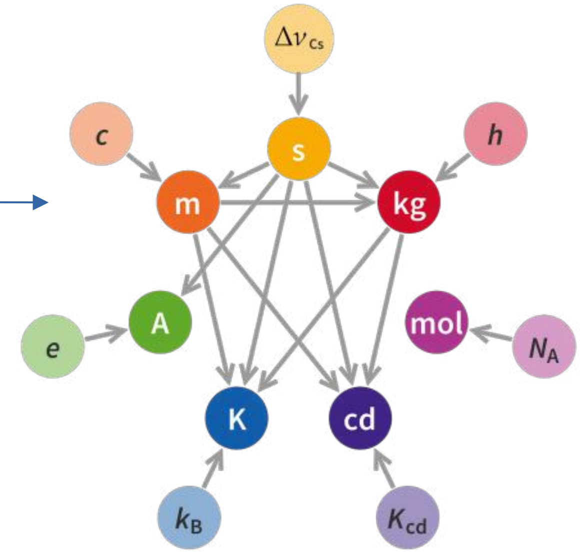
Going down: Silicon step standards

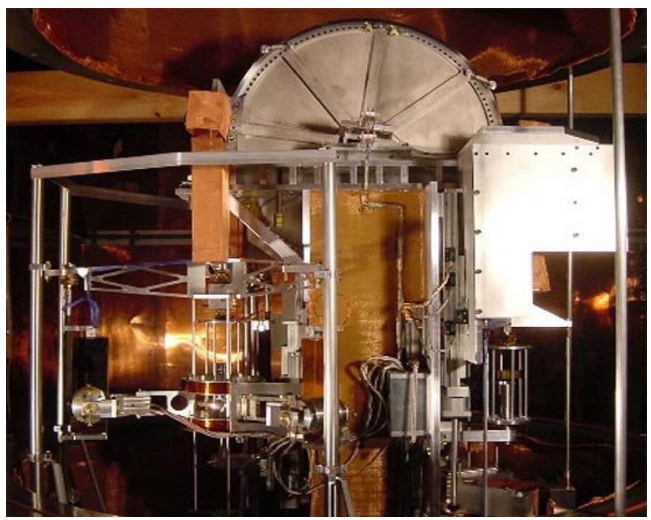
Redefinition of SI:

The biggest metrology challenge in the last years.

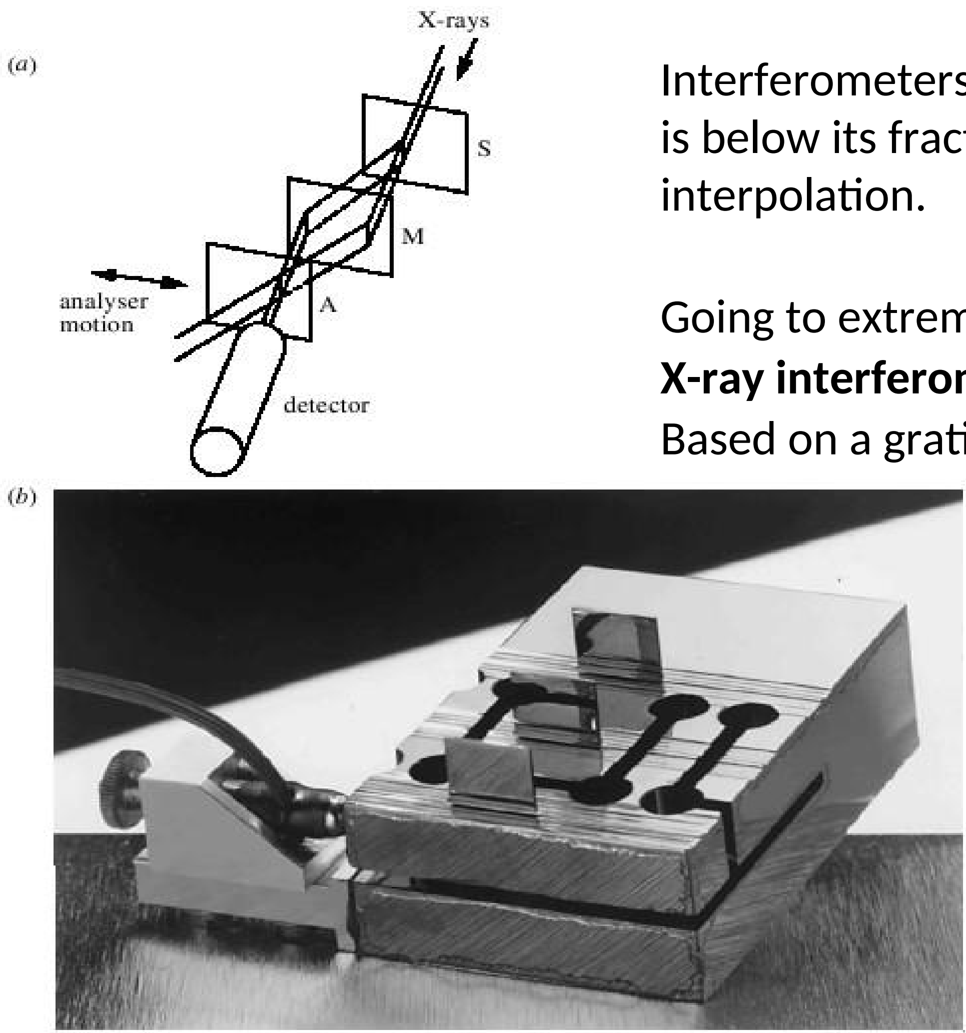
Goal was to used physical constants instead of unit prototypes.







Going down: Silicon step standards



How the length measurements benefit from SI redefinition?

Interferometers are limited by the wavelength; everything that is below its fraction (e.g. half of the wavelength) is a kind of interpolation.

Going to extremely small wavelengths:
X-ray interferometry
Based on a gratings created by silicon lattice.

COXI X-ray interfereometer based in National Physical Laboratory is being used e.g. for characterisation of non-linearities of other interferometers.

Traceability to **silicon lattice** is also a next potential realization of meter, covered to the present Mise en pratique documents by BIPM.

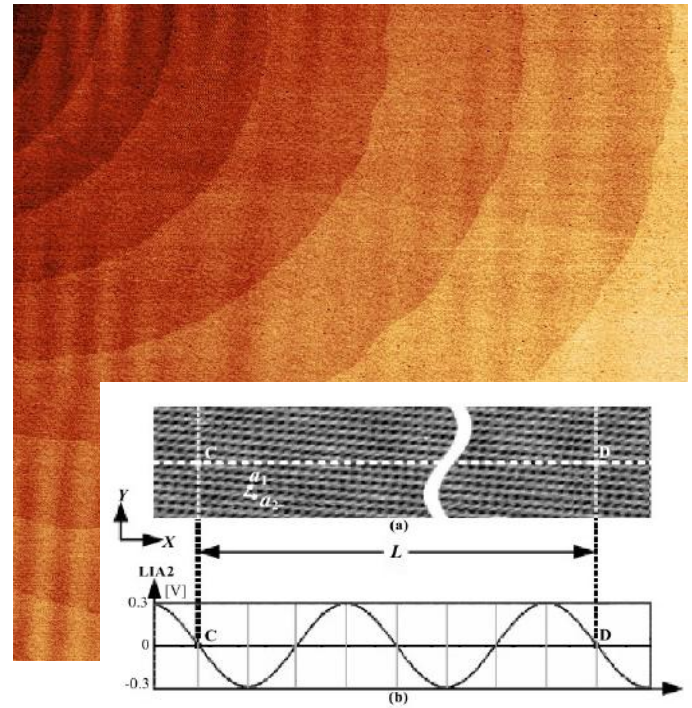
Going down: Silicon step standards

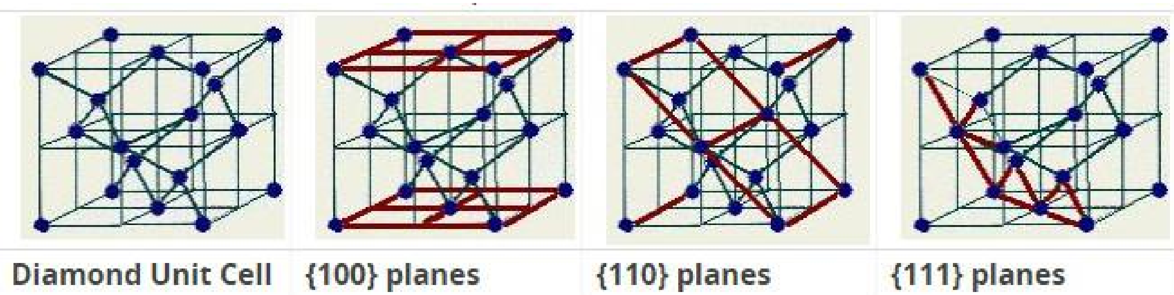
Si d_{220}, CODATA value (192,0155714 ± 0.0000032) pm

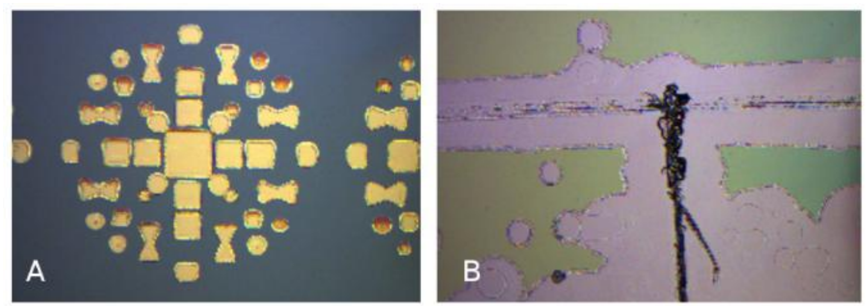
Step height for d_{111} = (313,5601151 ± 0.0000053) pm

Uncertainty in the range of 10^{-8}, comparable to the best custom built interferometric systems.

No need to care for interferometers uncertainty sources (refractie index, Abbe error, etc.).







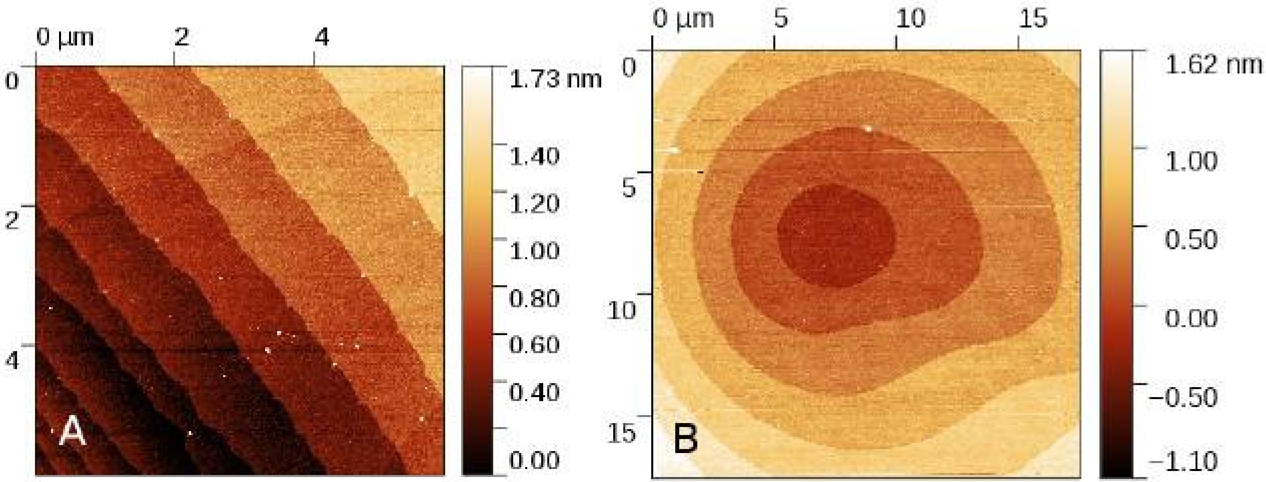
Going down: Silicon step standards

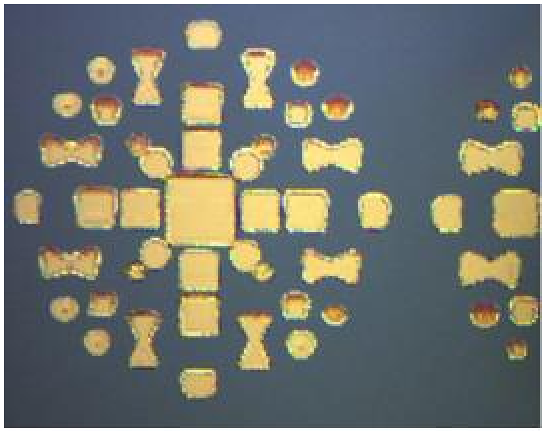
Atomární schodky v nanometrologii

Možnost využití atomárních schodků je jedním z důsledků redefinice kilogramu a dalších aktivit souvisejících s novou SI soustavou. Velmi přesná hodnota meziatomární vzdálenosti v křemíku vedla k rozpracování metodik pro sekundární realizaci metru pomocí křemíku – pro kalibraci TEM mikroskopů a pro měření výšky v různých mikroskopických metodách.

V minulosti vznikla řada slibných vzorků o různých geometriích, metodické pokyny pro jejich využití a aspekty nejistoty měření však zůstaly nepokryty.

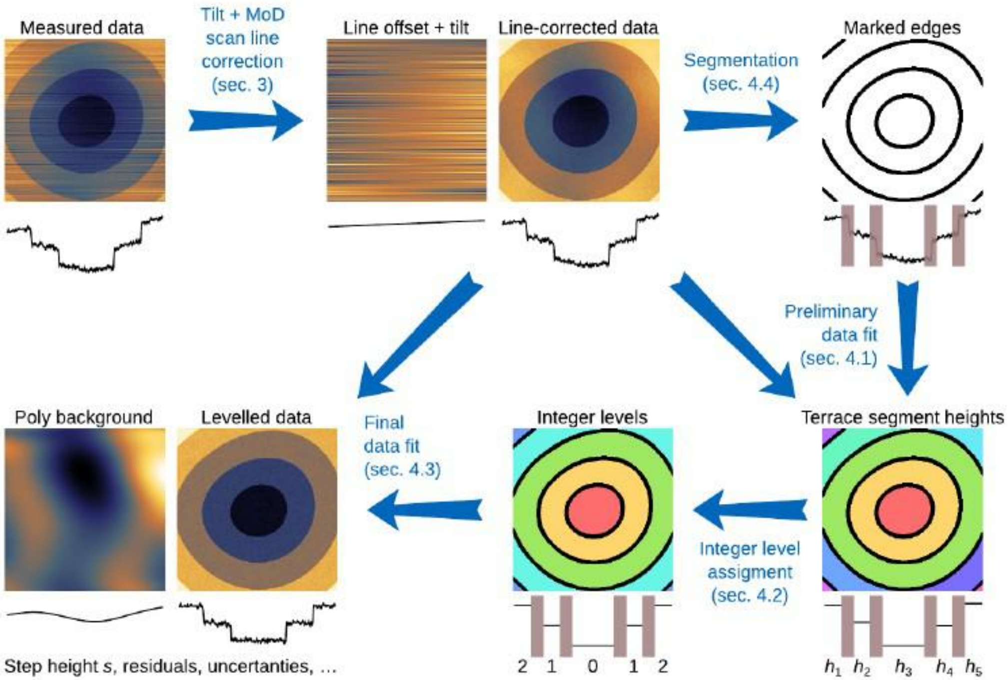
Proto jsme se podíleli na vývoji algoritmů pro analýzu křemíkových schodků.

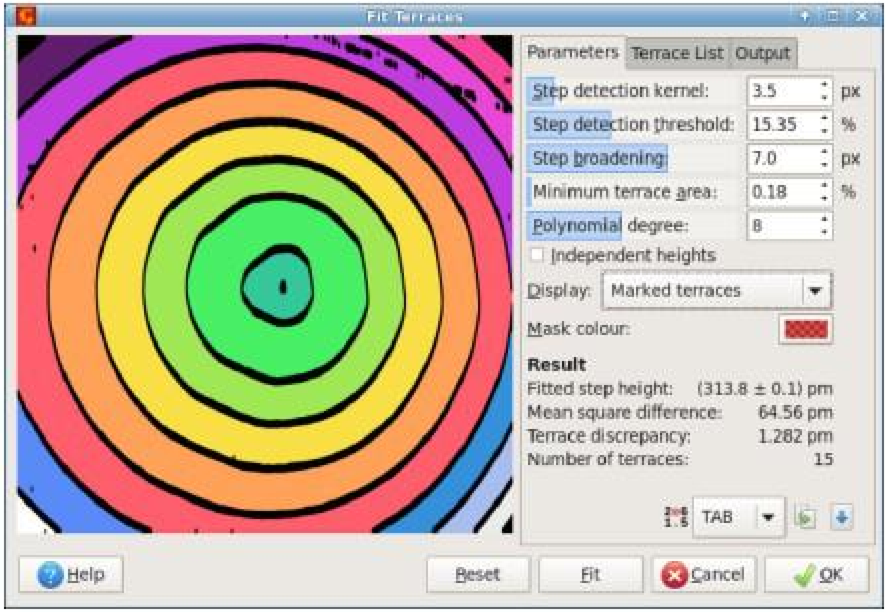




Separation of background and silicon steps data

A procedure suggested by DFM was extended to 2D by David Nečas and is now part of Gwyddion open source software.



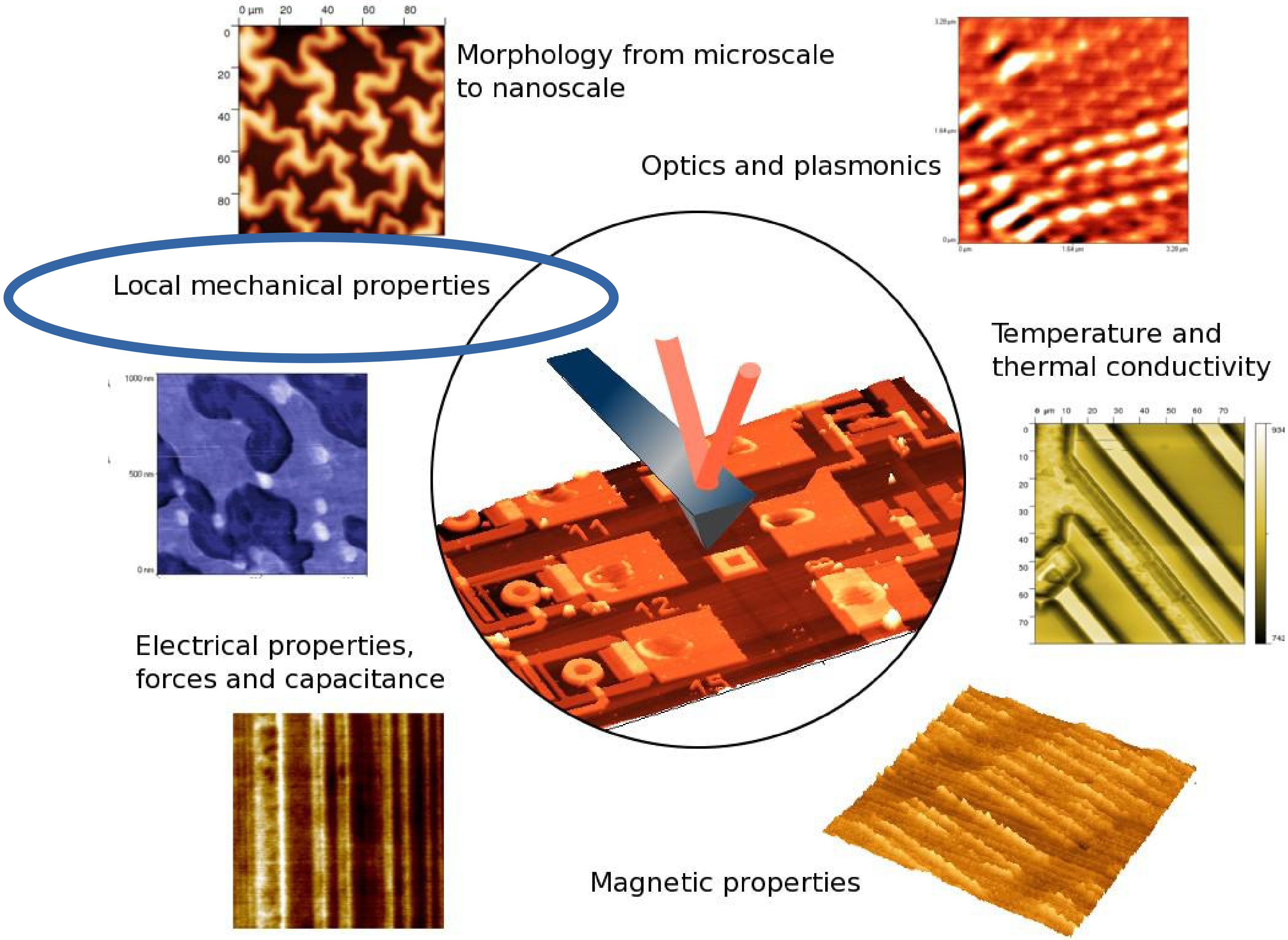


Dimensional wrap up

Dimensional measurements guidelines:

- calibrate your microscope (at least once a year)
- know your probe
- if you have doubts about your measurement, rotate the sample by 90 degrees
- follow all the data processing guidelines provided in yesterday's talk

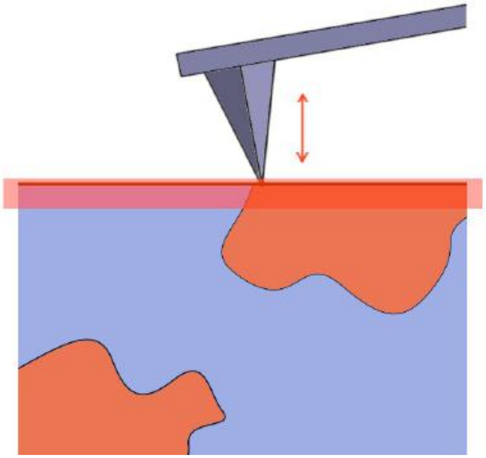
SPM – scanning probe microscopy



Mechanical measurements in SPM?

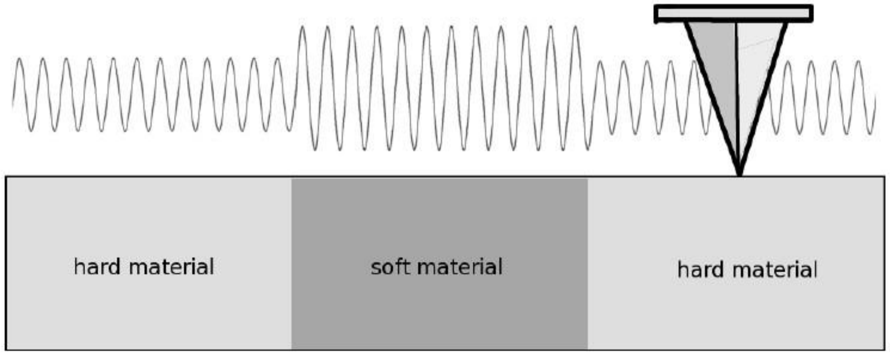
Motivation

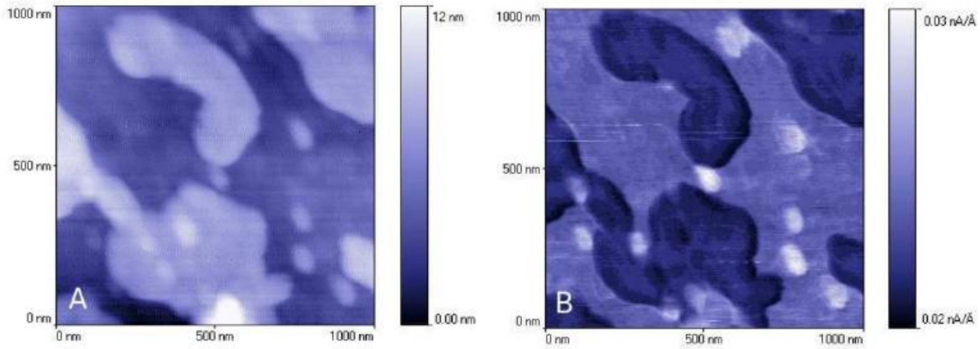
Since very beginning there were some attempts to use the force-distance data in AFM for some viscoelastic properties mapping.



Z-modulation technique was one of the first trials.

With advent of fast FPGA based controllers we can see massive improvemnts by nearly all the manufacturers.



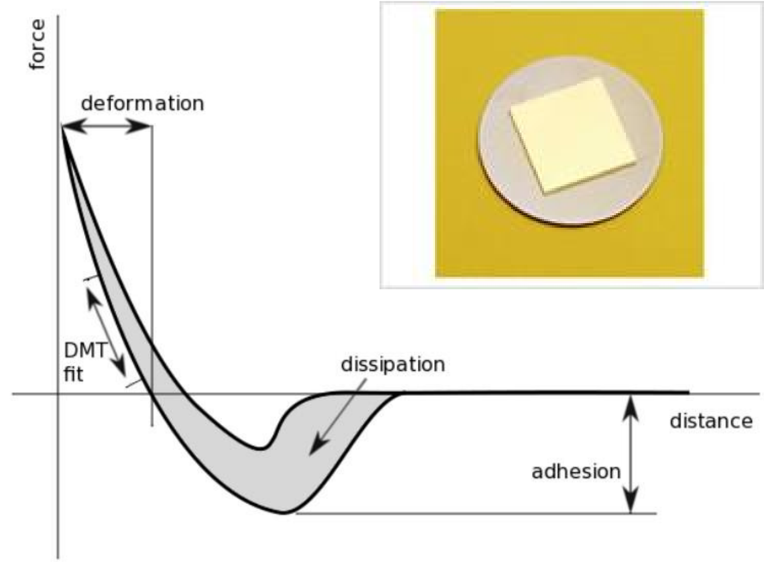


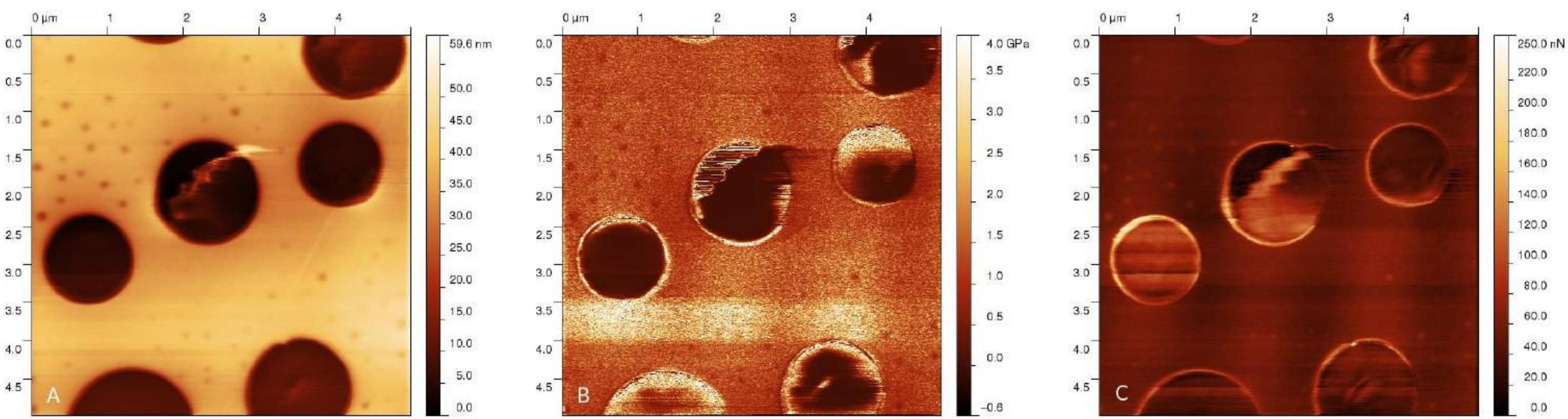
Mechanical measurements in SPM!

Various brand names:
PeakForce QNM (Bruker), Quantitative Imaging (JPK), PinPoint (Park), RSI (NT-MDT). They differ only in details

Principle: Indentation at every pixel
Benefits: small indentation depths, very high resolution

Reference samples: still a problem, two component polymer mixtures are good test sample.

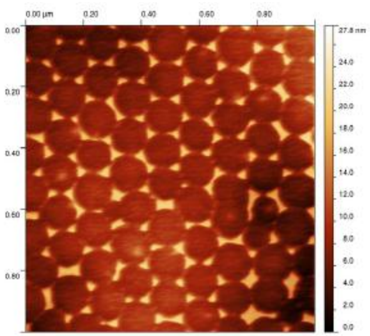


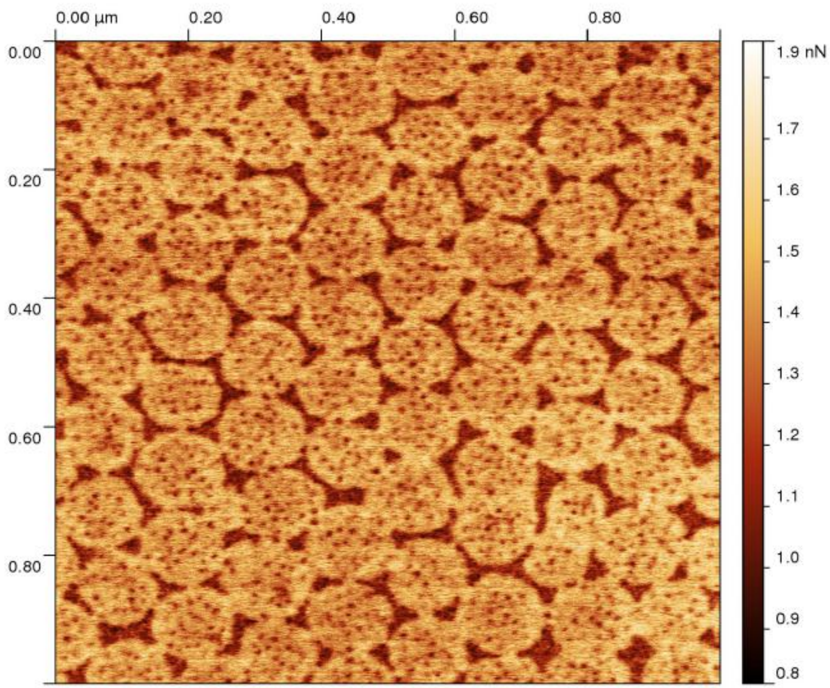


Gallery of nice mechanical measurements

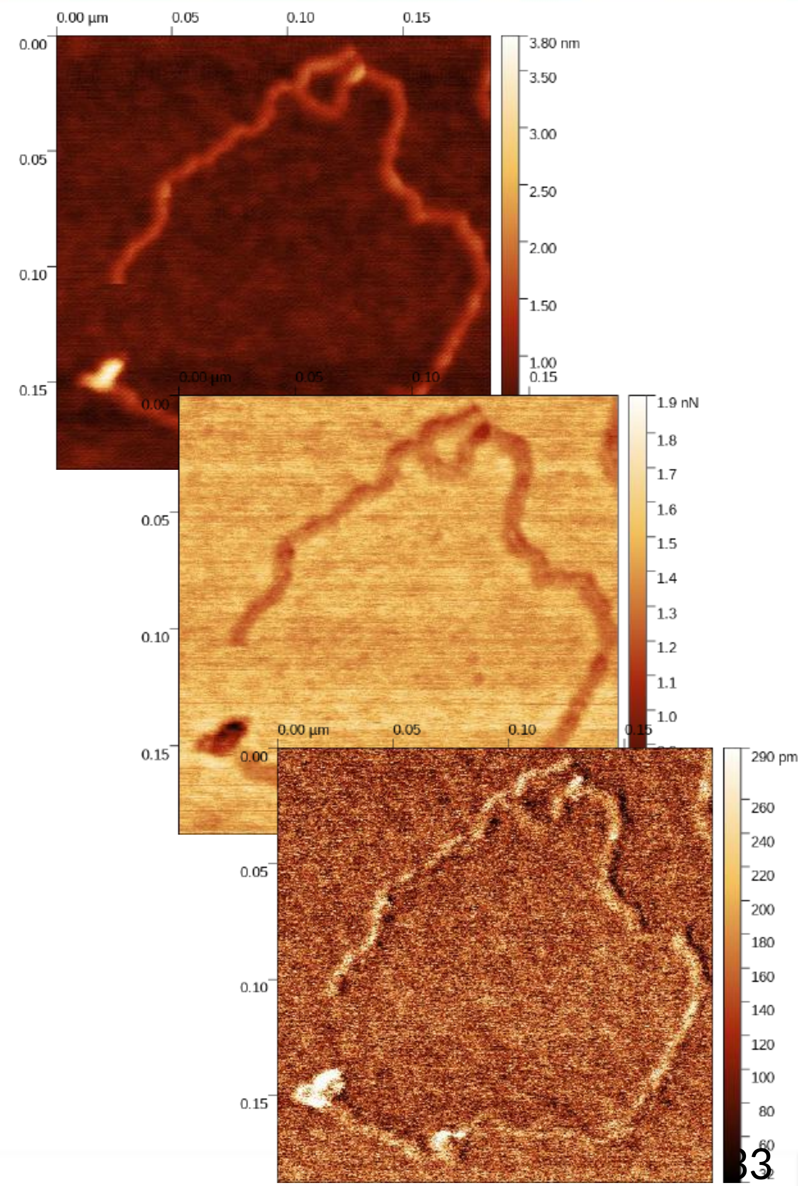
Applications:

Many impressive results on biological samples, like cells or tissues, on polymers, single molecules and molecular films, graphene and other 2D materials, thin films, nanocomposites.





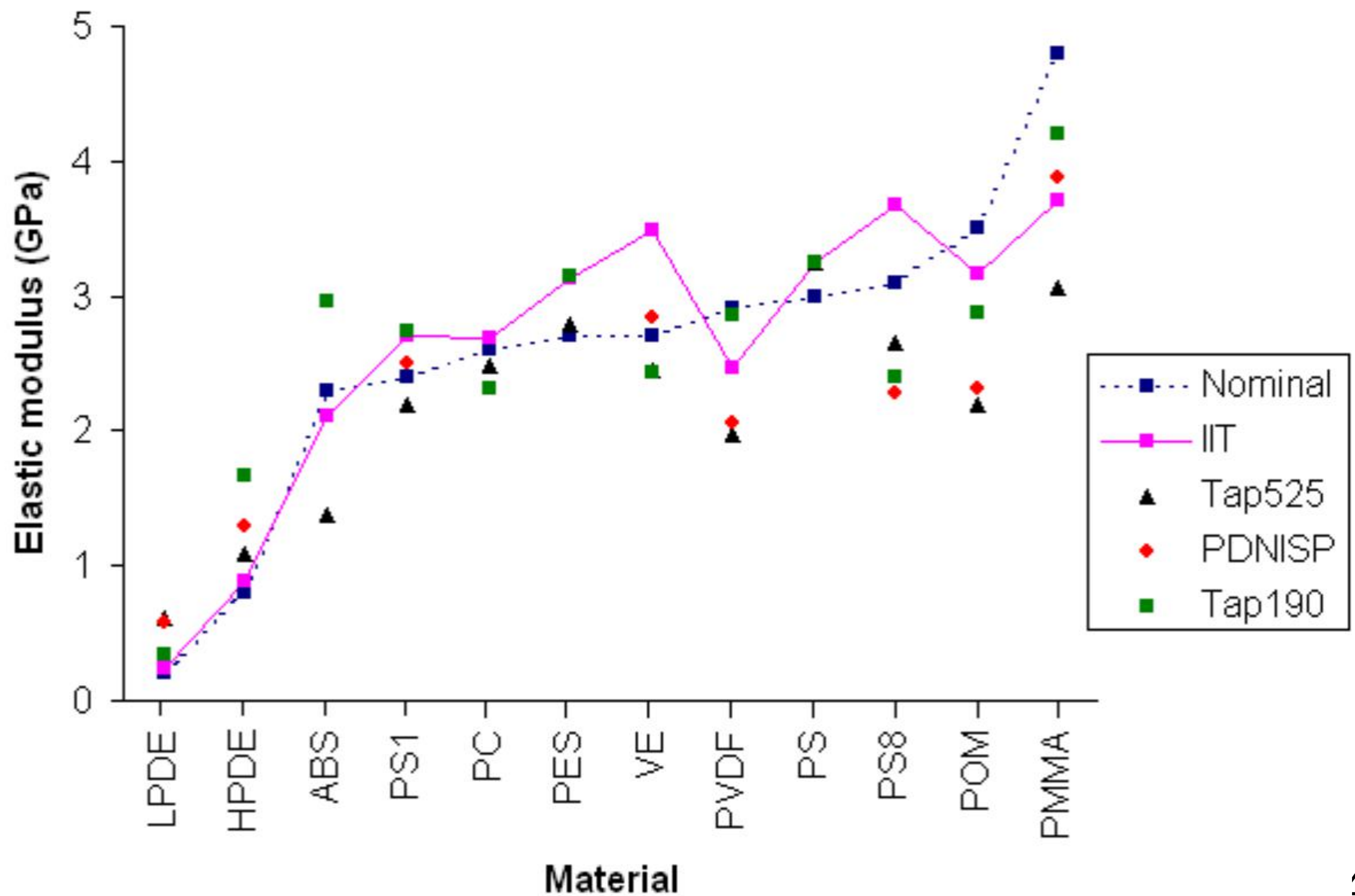
Sample courtesy:
Petr Marek



Accuracy and traceability?

There are only few studies on method accuracy in the literature.

http://epubs.surrey.ac.uk/722268/3/Young_et_al%2C_Peak_Force_QNM.pdf

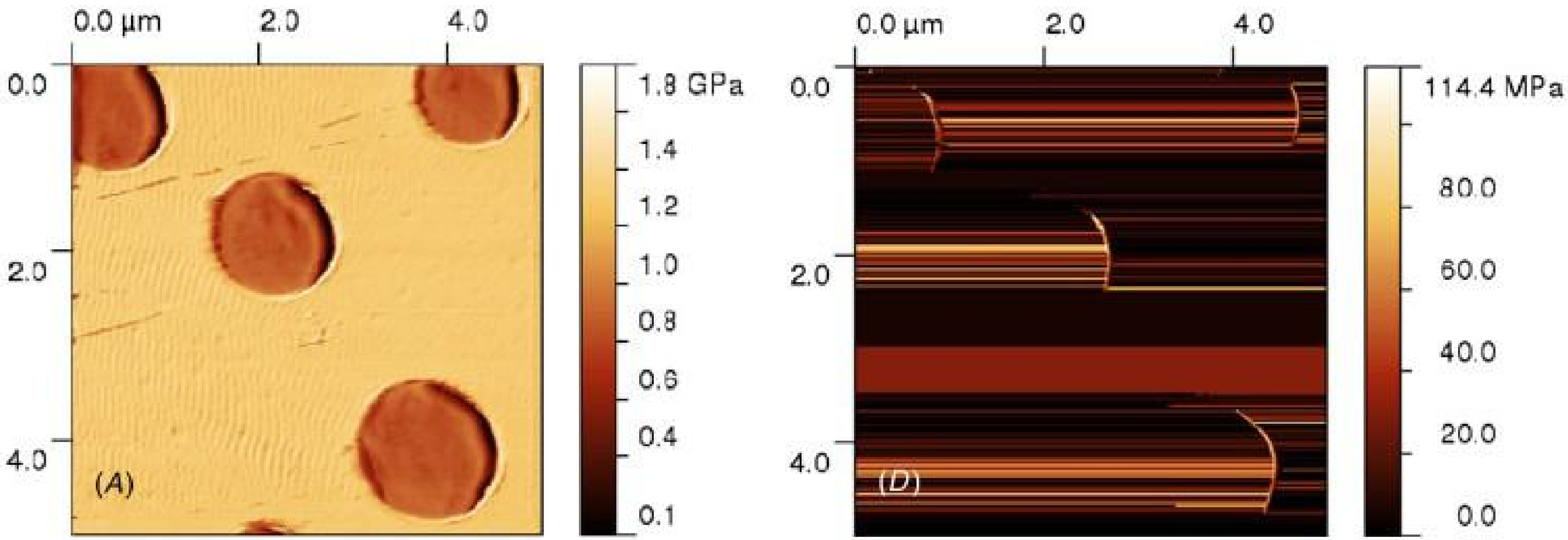


Accuracy and traceability?

Measurement protocol needs to be very carefully followed to get anything quantitative.
Even after that, manufacturer's calibration routines have limited accuracy.

Wide range of potential results depending on settings, e.g. tip radius.
Real time data analysis does not work always, which can be easily unnoticed.

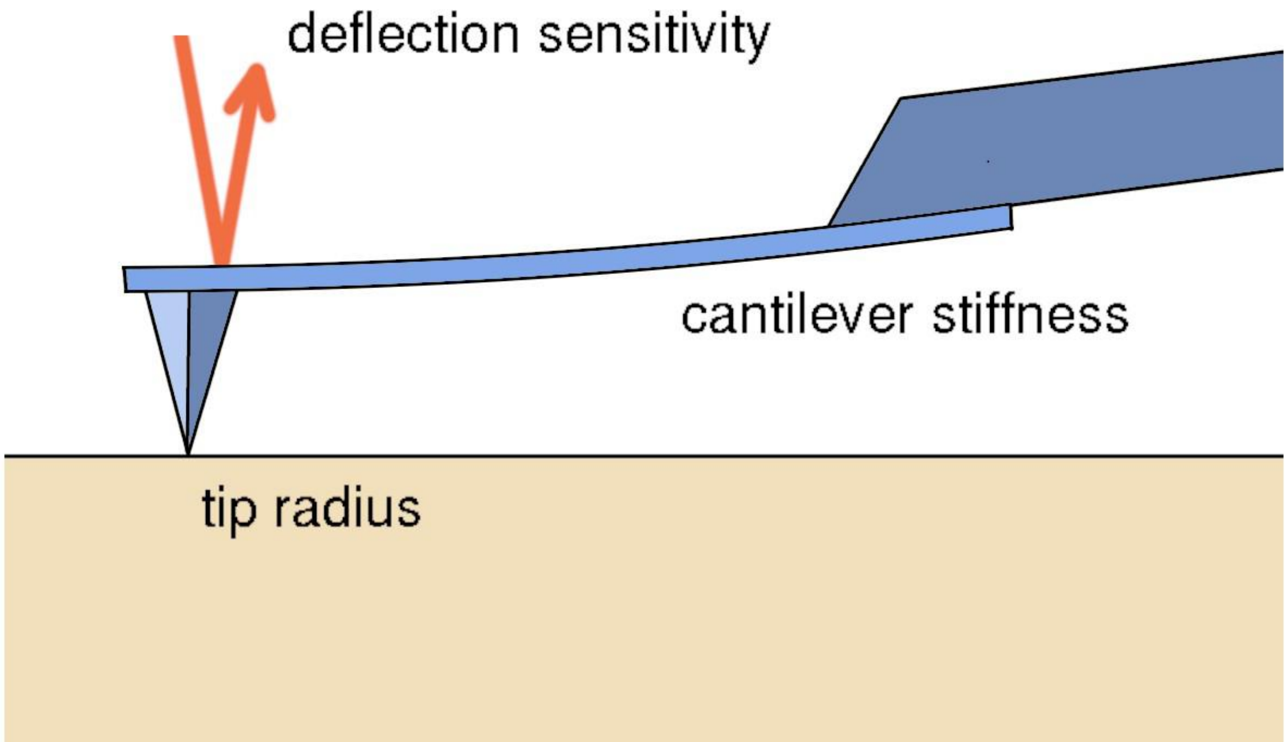
It is assumed that results can be about 10 percent accurate. How often this happens?



Methodology, traceability and uncertainties

The key elements in SPM are the probe/cantilever assembly and the optical pickup.

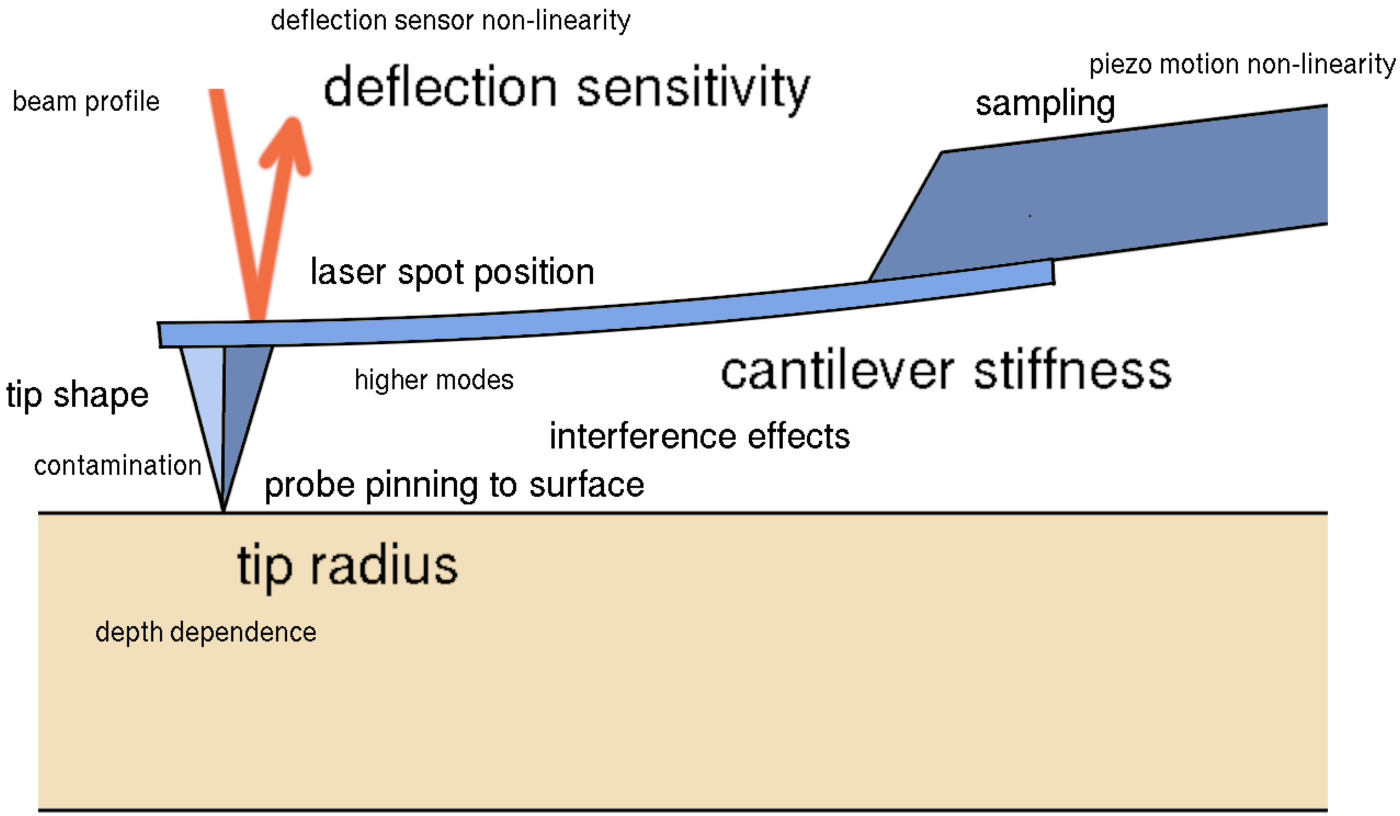
Parameters most affecting the measurement:



Methodology, traceability and uncertainties

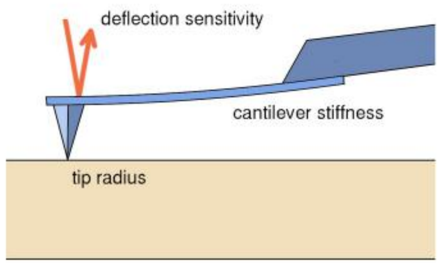
The key elements in SPM are the probe/cantilever assembly and the optical pickup.

Reality is even worse:



Deflection sensitivity

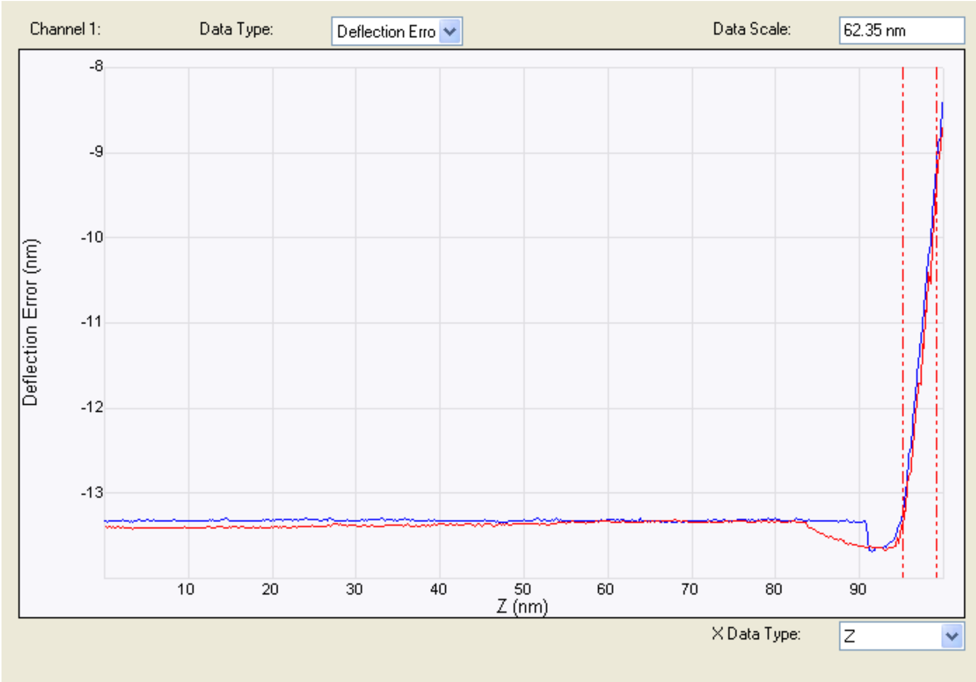
This includes calibration of the whole sensing element of the microscope, including the laser alignment, position sensitive detector settings and electronics readout.



It needs to be done for each probe, everytime it is mounted and it is done via pressing the cantilever towards hard surface (e.g. sapphire).

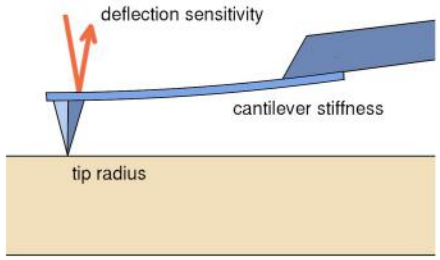
Unless we want to measure simliarly hard surfaces, it works fine.

Variance of the results is in percents.

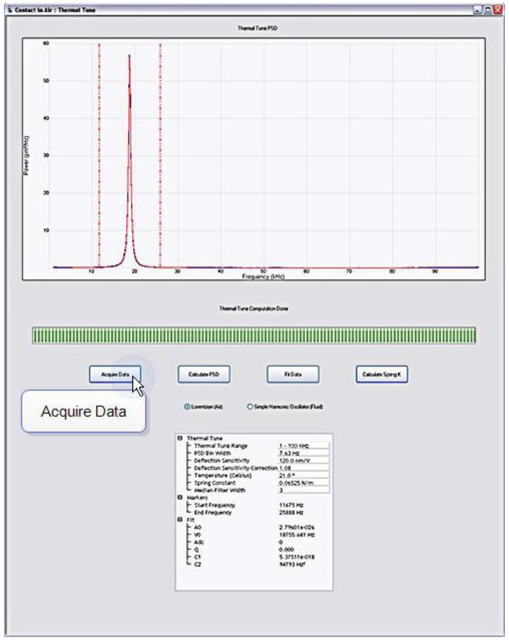


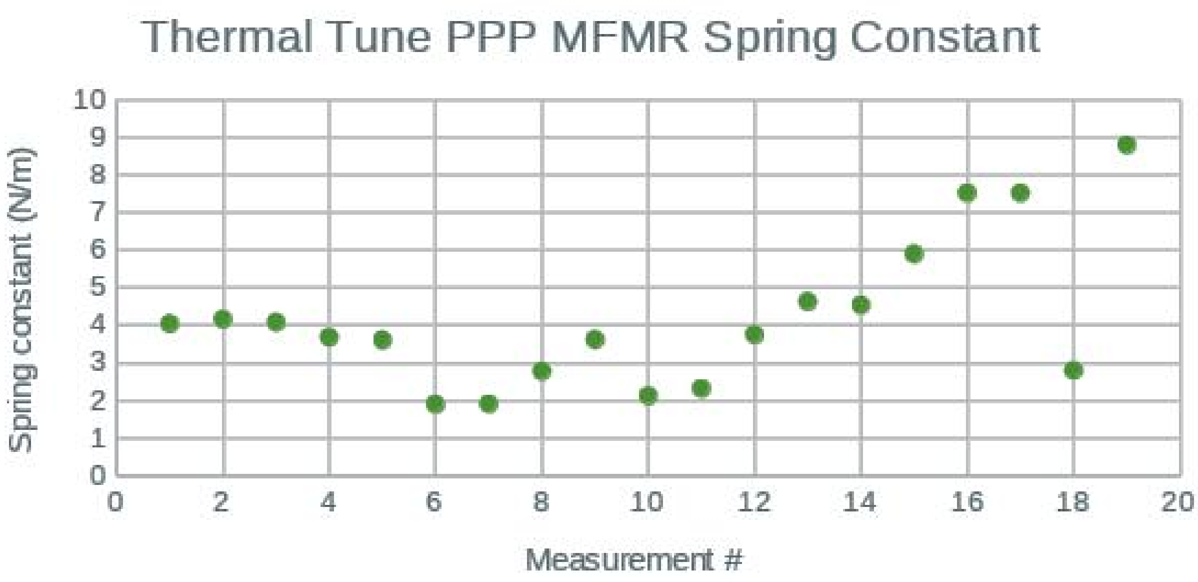
Cantilever stiffness

Our sensor measures deflection, not force.
Stiffness calibration needs to be done.



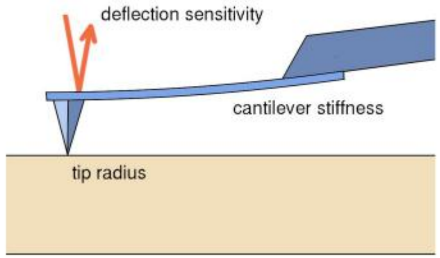
The most common and built-in methods are based on thermal fluctuations, which can be done up to about 10-15 N/m cantilever stiffness.



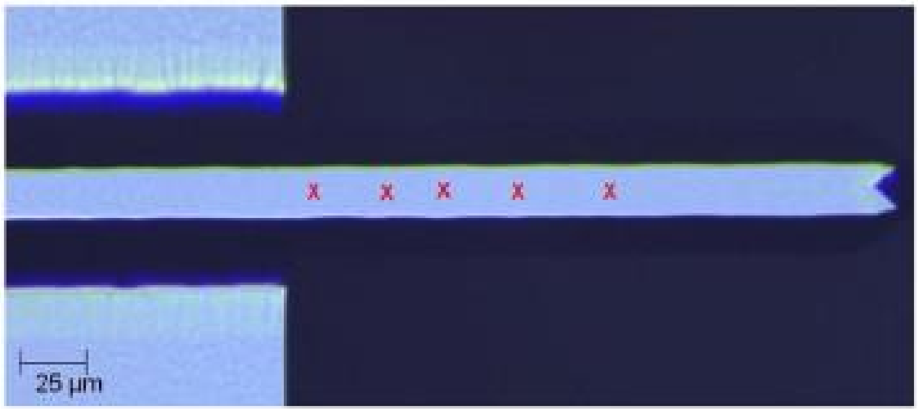


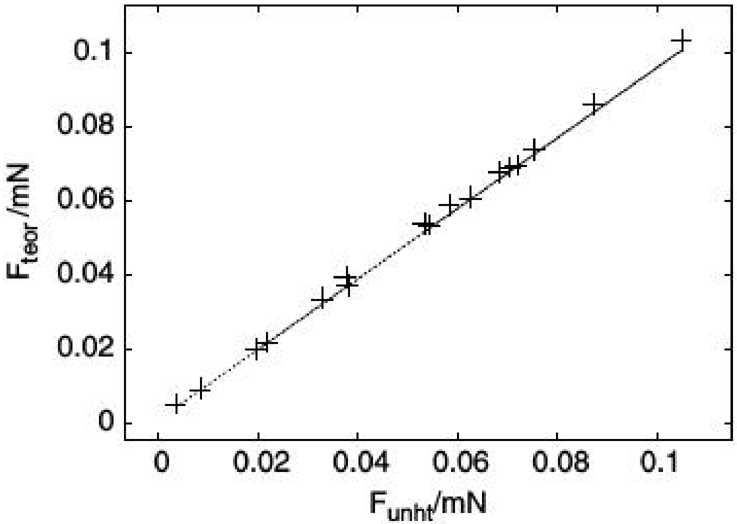
Cantilever stiffness

There are many more elaborated methods, some of them having uncertainties below 10 percent.



One of them is to use the instrumented indentation and measure the dependence of force and displacement on the cantilever. In principle even a single measurement should work.



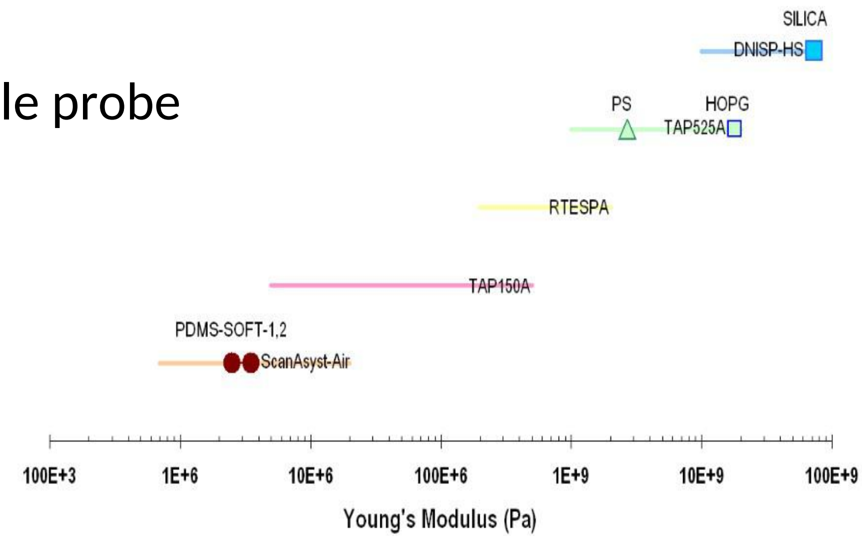


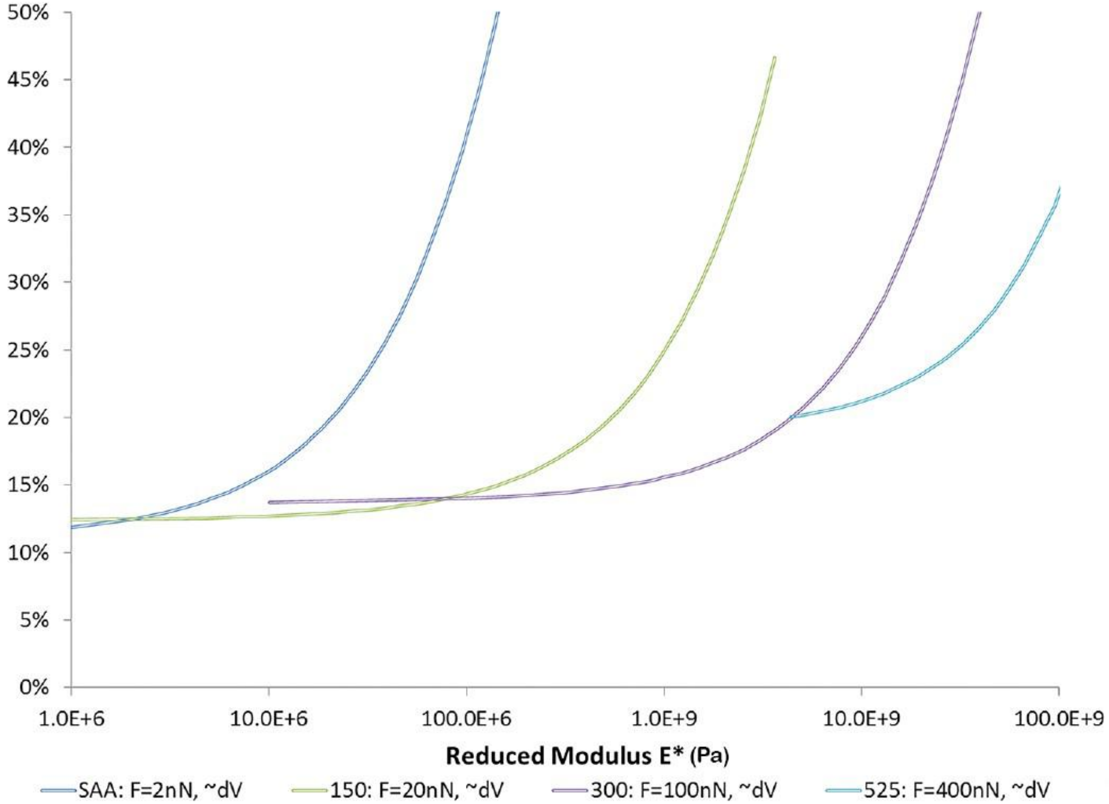
A Campbellová *et al* Meas. Sci. Technol. **22** (2011) 094007

Cantilever selection

Not every probe is suitable for every measurement

The dynamic range of AFM is quite low and a suitable probe needs to be taken for every sample type.



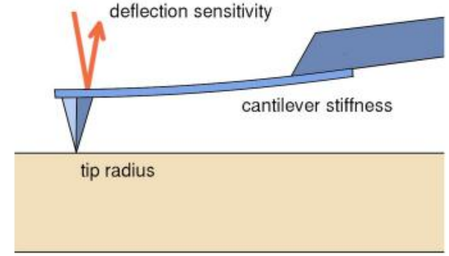


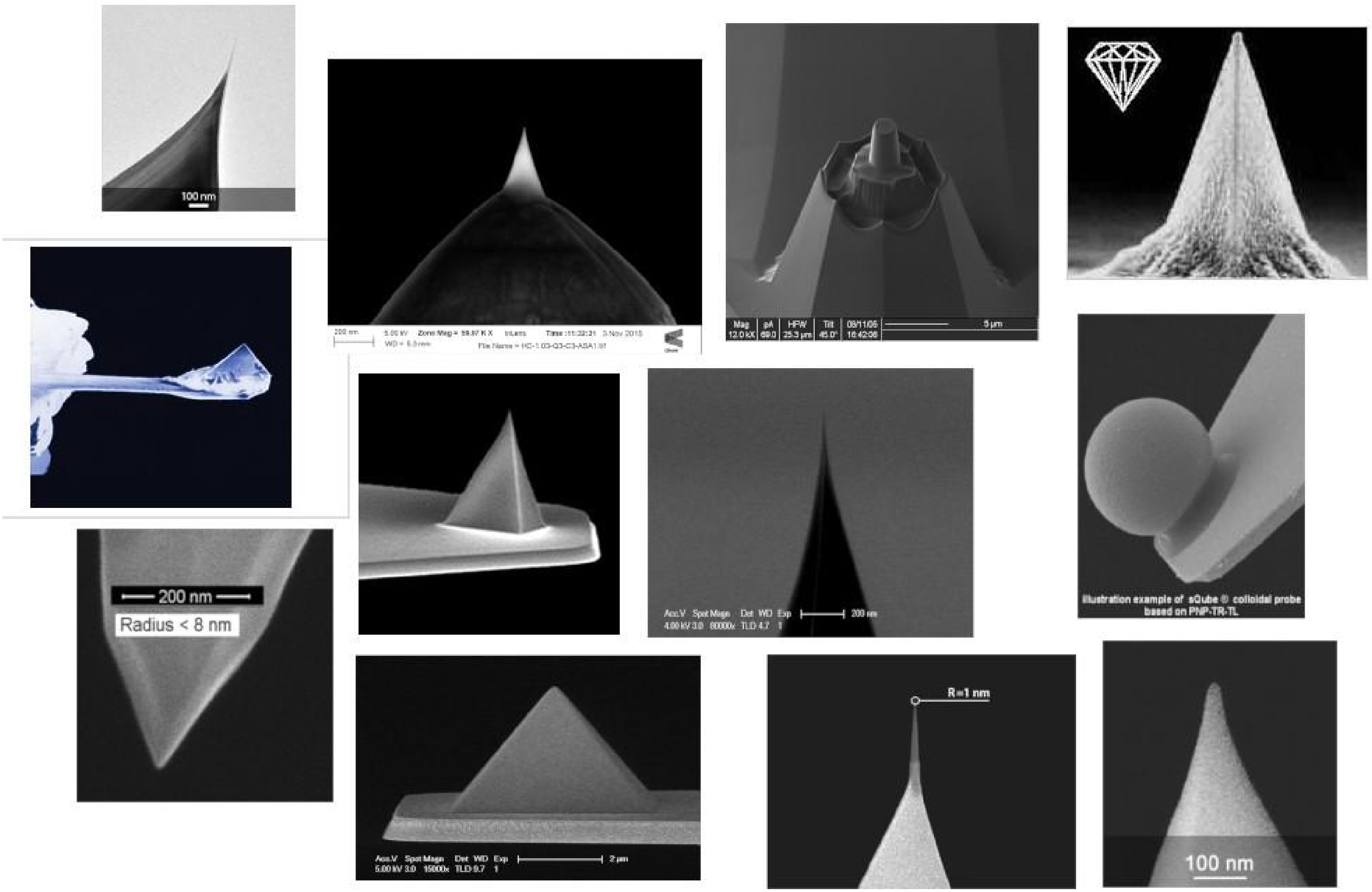
Probe	Radius (nm)	kc(N/m)	Min. E (MPa)	Max. E (MPa)
SAA-HPI-30	33	0.25	0	15
RTESPA150-30	33	5	5	500
RTESPA300-30	33	40	200	8,000
RTESPA525-30	33	200	1,000	50,000
DNISP-HS	40	450	10,000	100,000

Bruker App note DOI: 10.13140/RG.2.2.15272.67844

Tip radius estimation

Tip radius needs to be determined and **constant**.

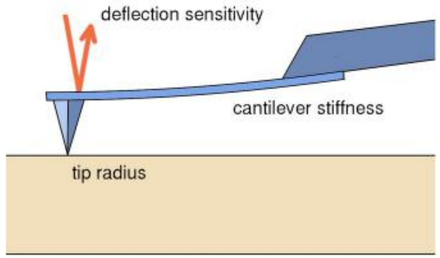


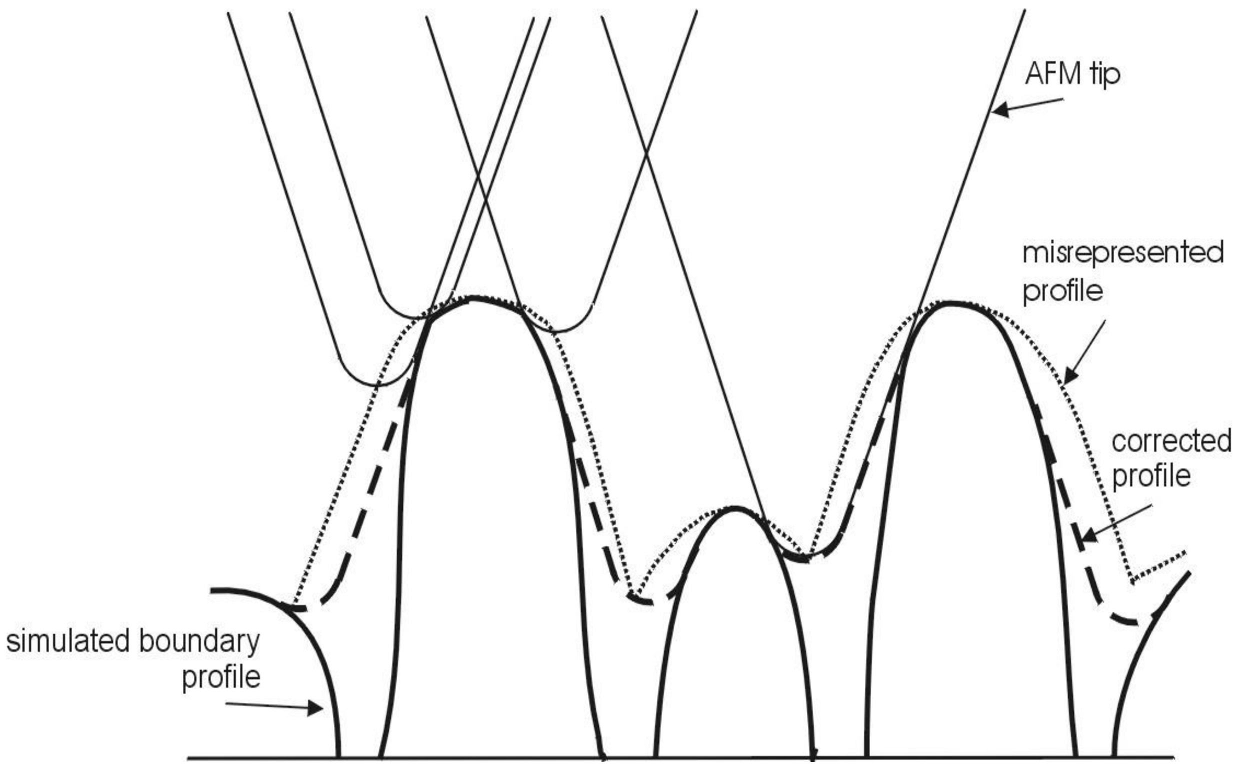


Tip radius estimation

Tip radius needs to be determined and **constant**.

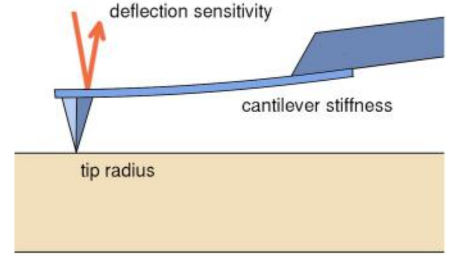
We use tip-sample convolution to determine it, scanning a known surface.



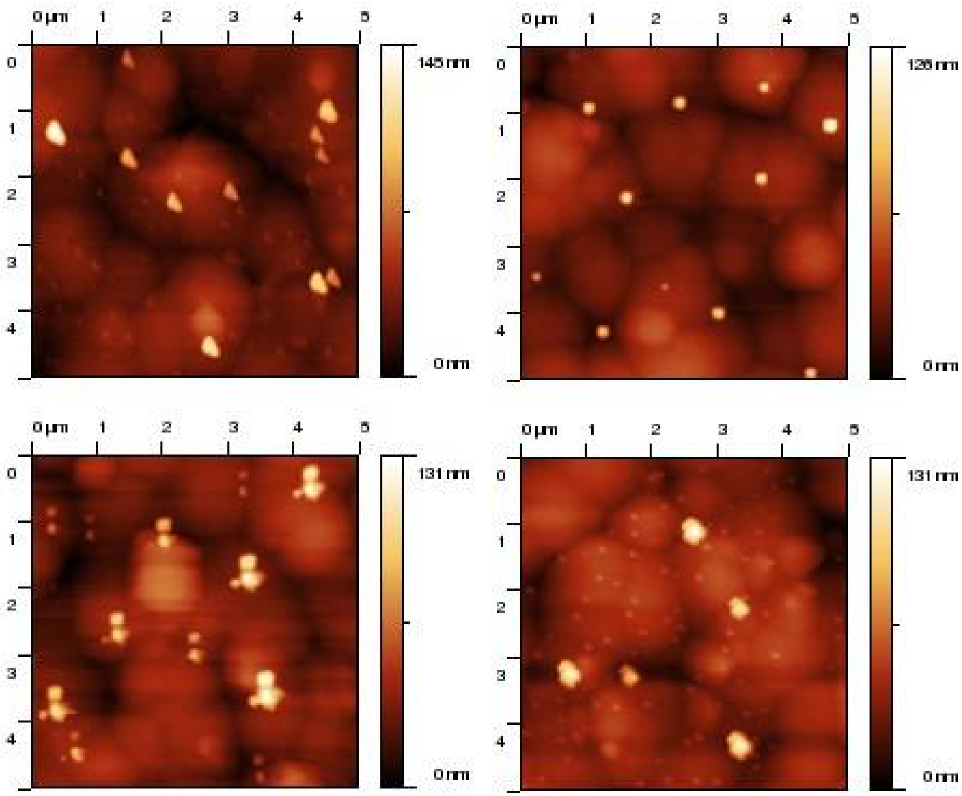


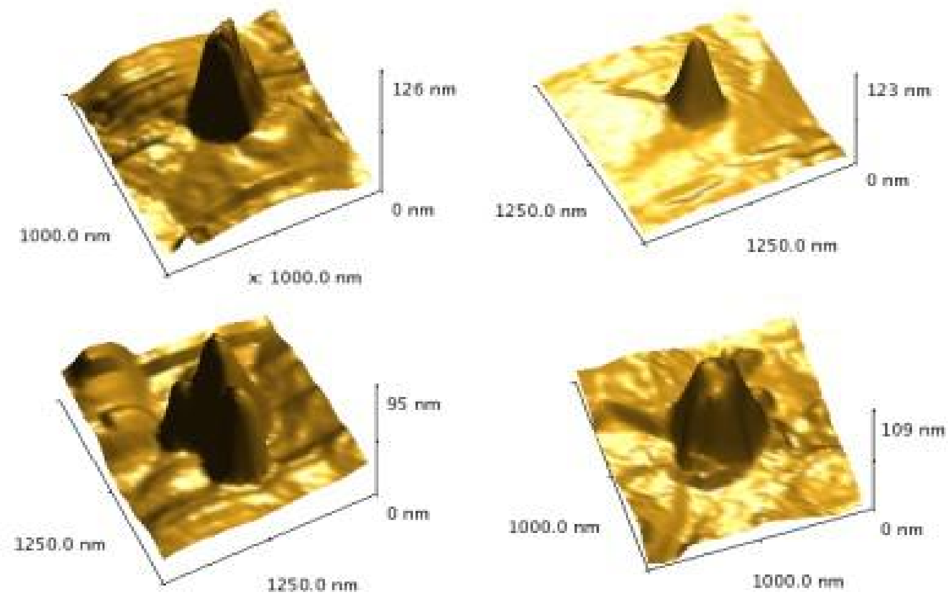
Tip radius estimation

Tip radius needs to be determined and **constant**.



We use tip-sample convolution to determine it, scanning a known surface.





Tip radius estimation

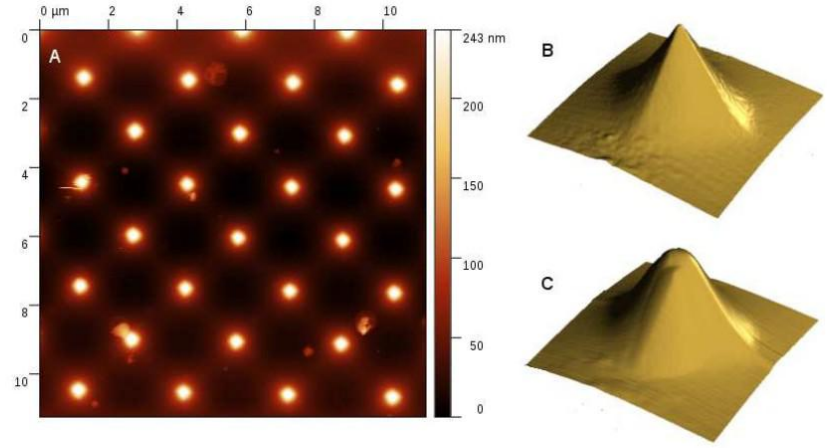
Reference samples for radius determination are usually part of the calibration set for nanomechanical mapping

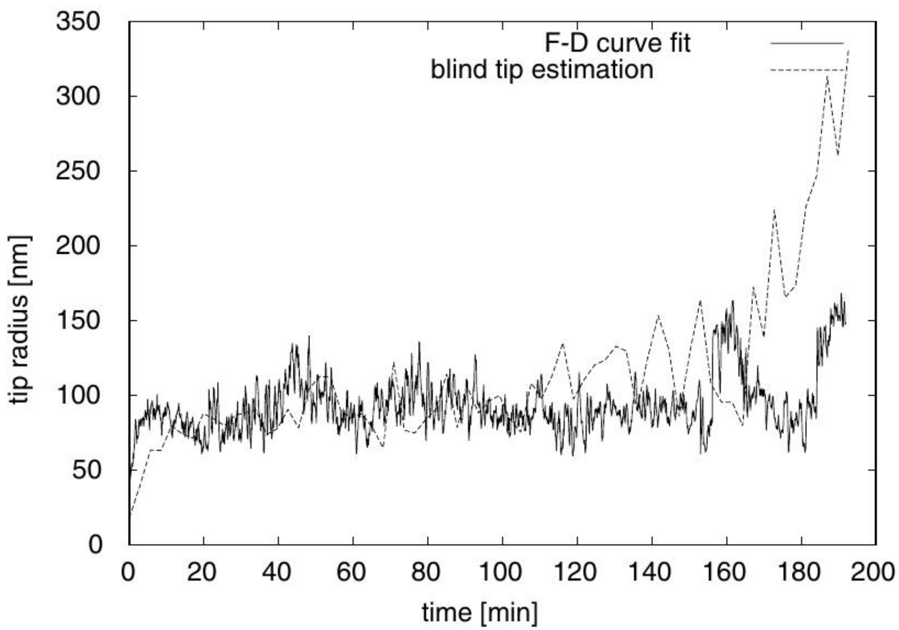
However, radius can change (and often changes) while scanning.

Also while scanning the reference samples.

It is therefore very likely that our tip radius might be wrong by tens of percents.

P Klapetek and D Nečas
Meas. Sci. Technol. **25** (2014) 044009





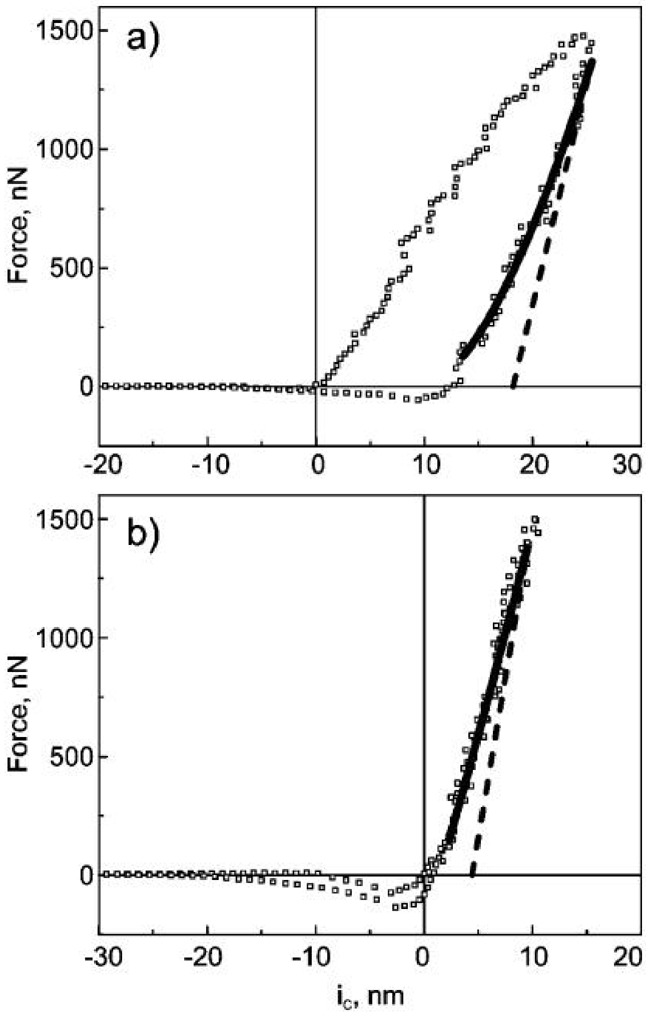
Tip radius selection

Sharp probe is however always **not ideal** tool for nanomechanical measurements.

It can lead to plastic deformation of the sample (or probe) and if high resolution is not needed, results obtained with somewhat blut probe can be more reliable.

See e.g. images on left from: Dokukin, M.; Sokolov, I. Macromolecules 2012, 45, 4277−4288.

Some manufacturers even sell spherical probes with large radius (e.g. 200 nm), but usually the sphere material is not very hard.



Methodology and measurement protocol

Two potential approaches are used for getting the measurements traceable

Absolute method: probe radius is determined on a tip check sample

Benefits: good for understanding what happens
Drawbacks: limited applicability

Relative method: one or two reference samples are used and probe radius is matched to get the correct results

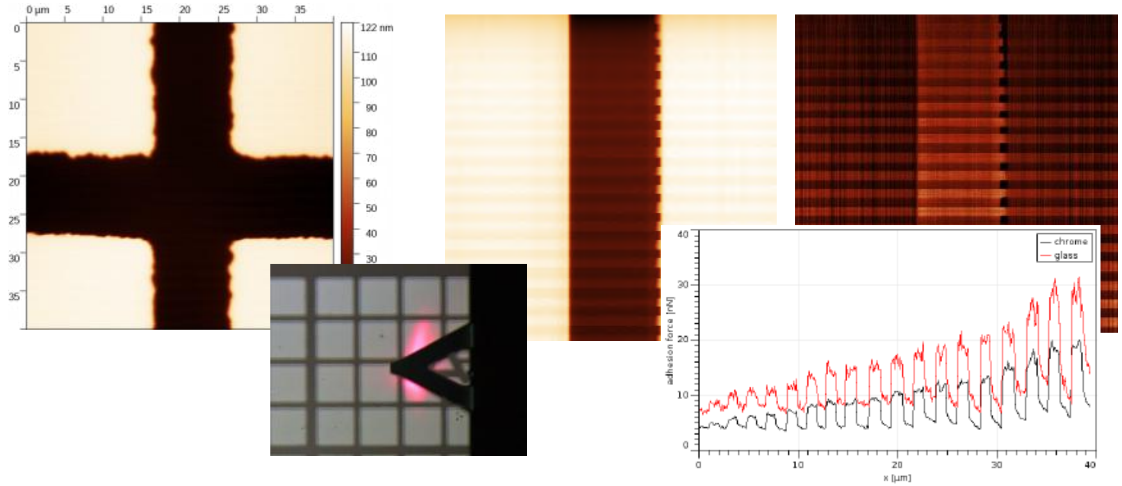
Benefits: many systematic errors can be hidden into it
Drawbacks: relies on reference samples, measurement on unknown sample should be similar.

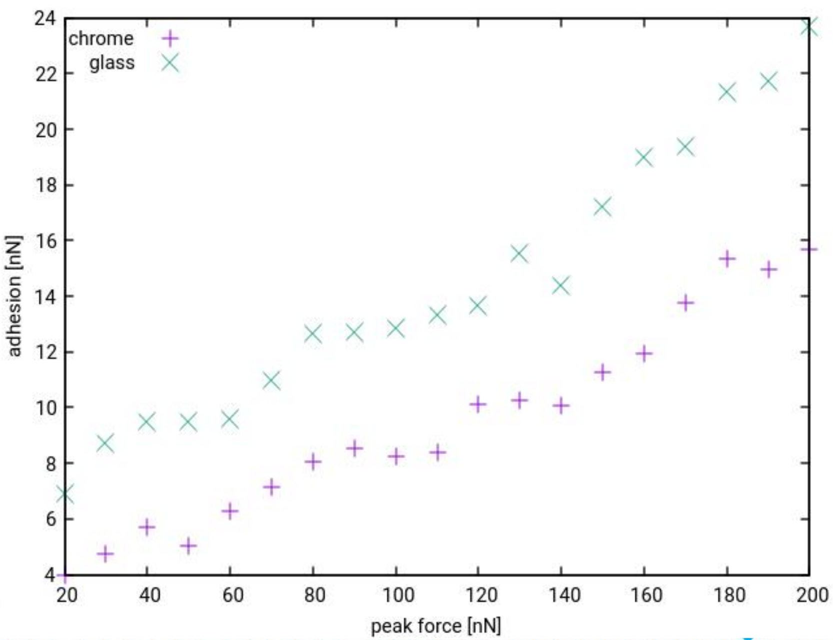
Methodology and measurement protocol

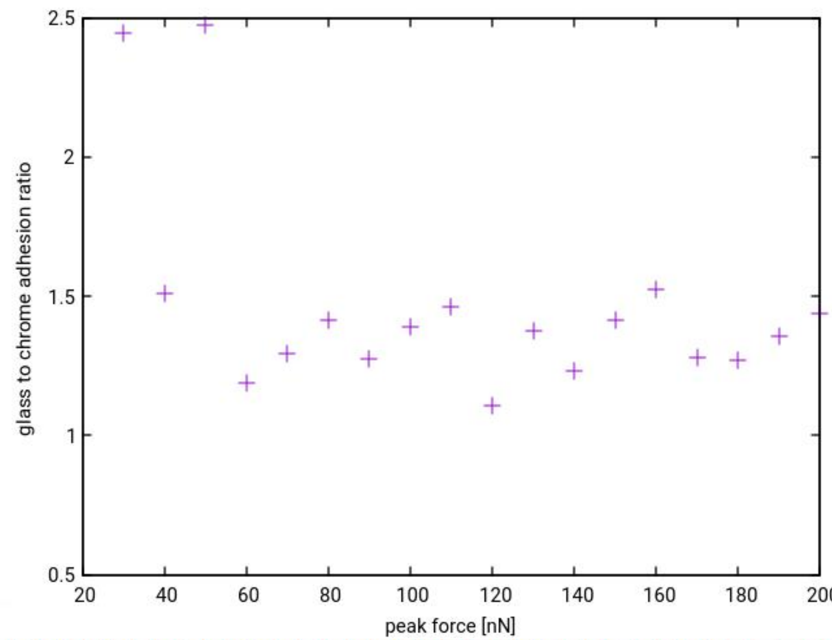
Dependence on load

Chrome on glass sample

Adhesion channel evolution for different peak forces.





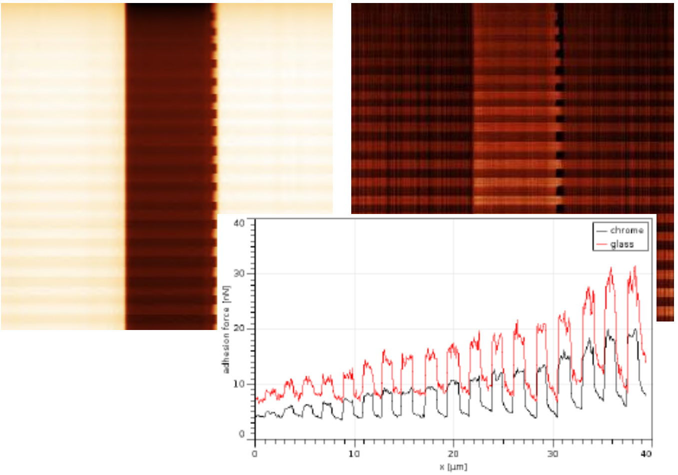


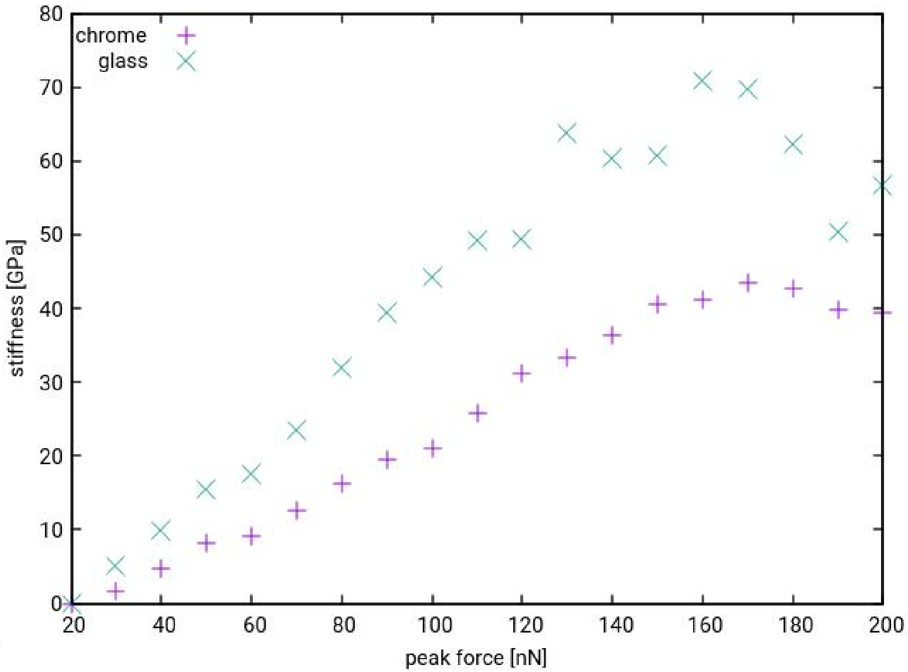
Methodology and measurement protocol

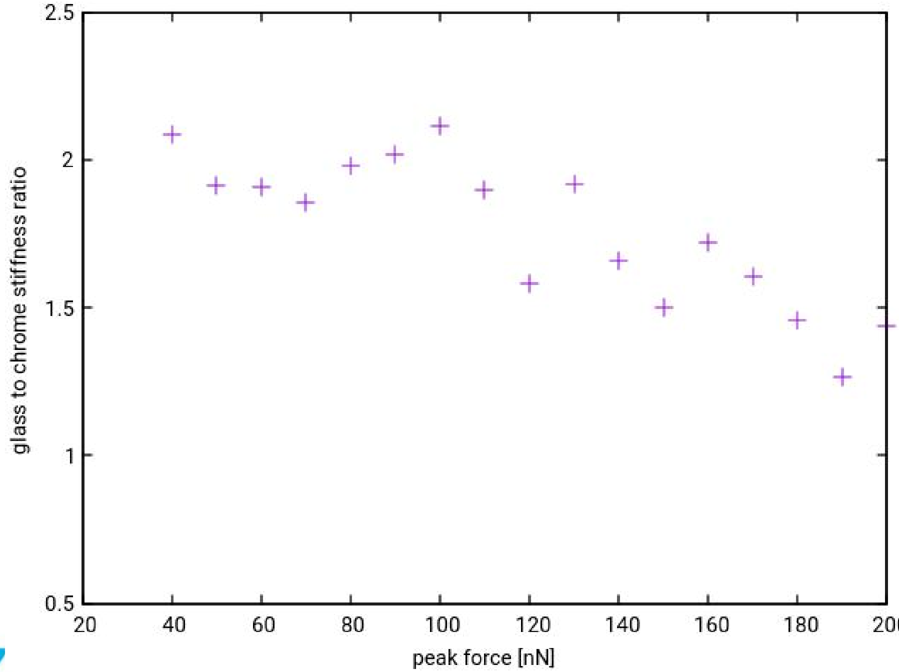
Dependence on load

Chrome on glass sample

DMT modulus channel evolution for different peak forces.

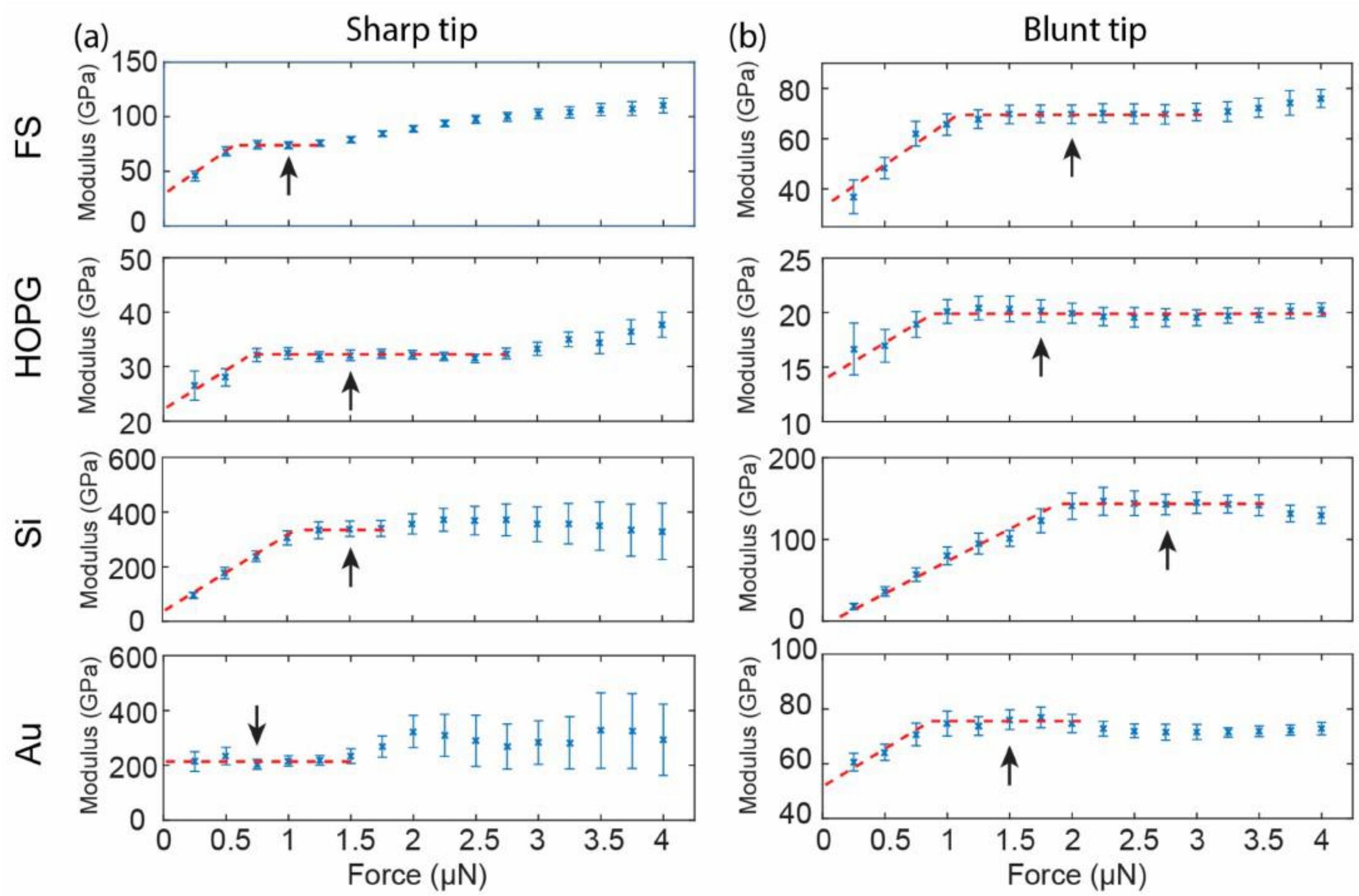






Methodology and measurement protocol

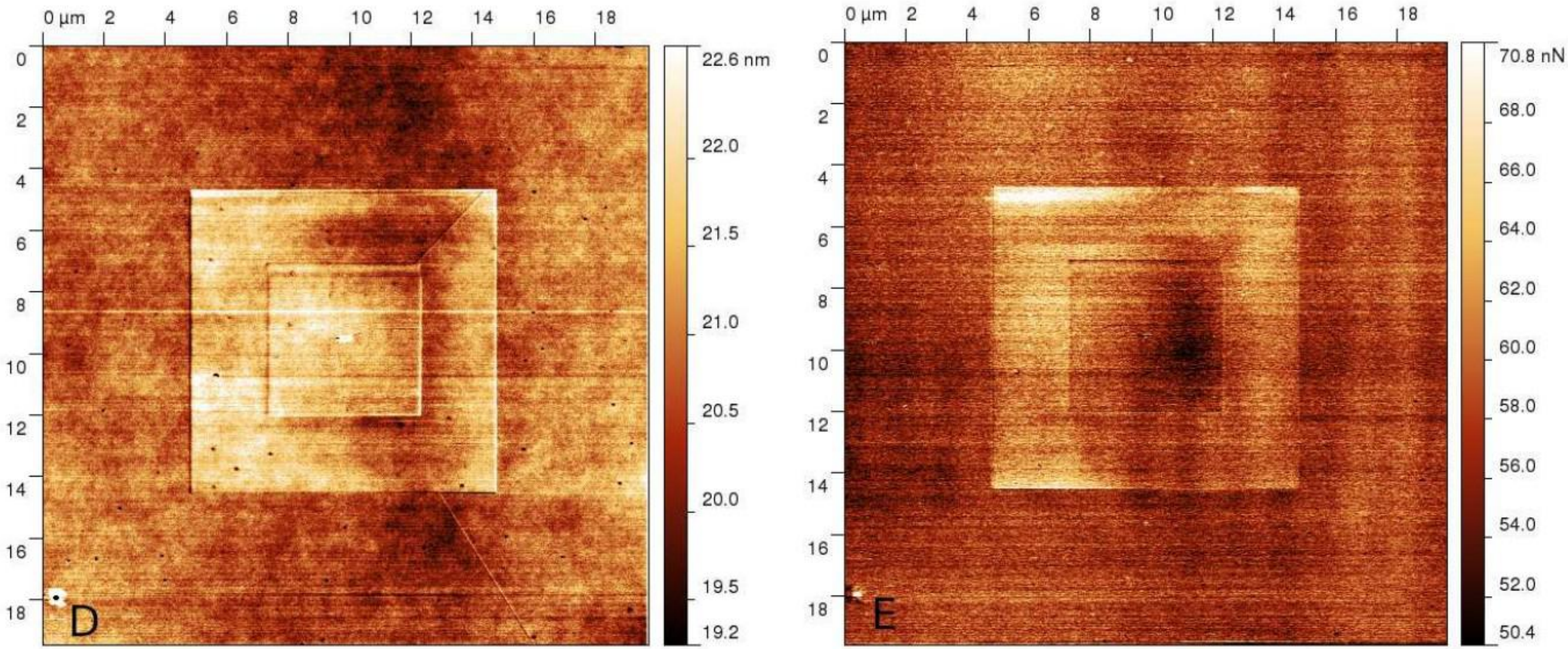
Dependence on load was observed also by other authors



Nanomaterials **2018**, *8*, 616; doi:10.3390/nano8080616

Still non-destructive technique?

Residuals on silicon surface after repeated PeakForceQNM measurement (topography and adhesion channel)

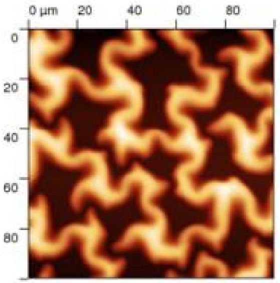


Mechanical wrap up

Mechanical measurements guidelines:

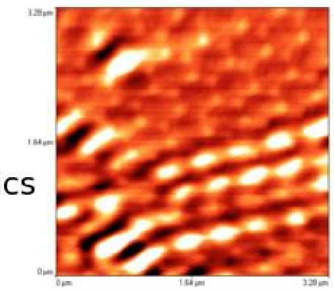
- calibrate your probe (deflection sensitivity and stiffness), do not believe default values
- determine your probe radius after measurement
- use some test sample to check that everything works
- store data for off-line processing

SPM – scanning probe microscopy

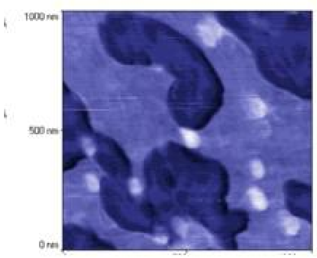


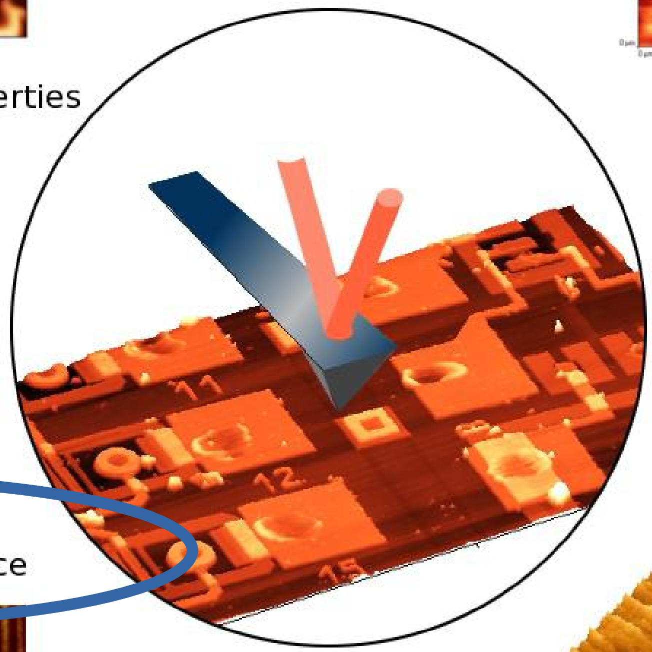
Morphology from microscale
to nanoscale

Optics and plasmonics

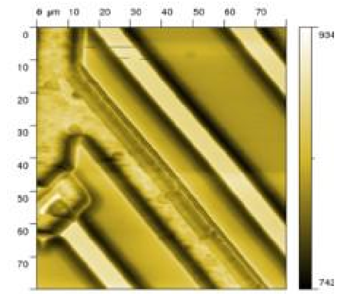


Local mechanical properties

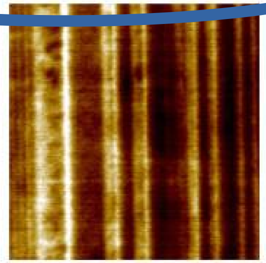




Temperature and
thermal conductivity



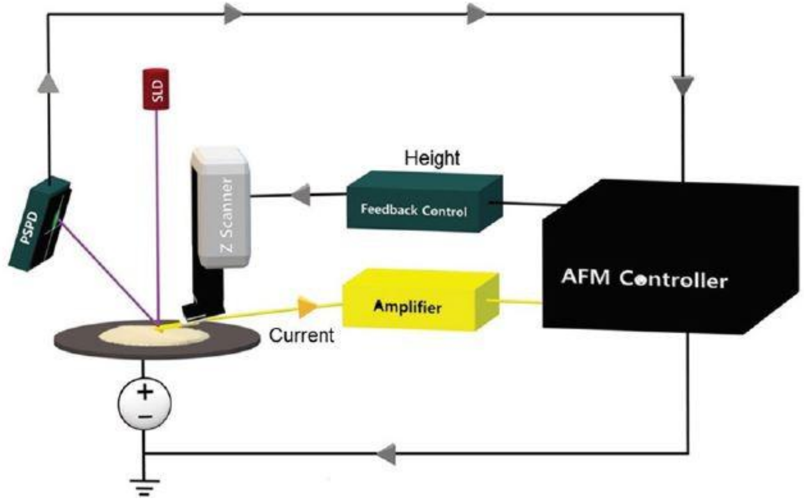
Electrical properties,
forces and capacitance



Magnetic properties

Conductive Atomic Force Microscopy

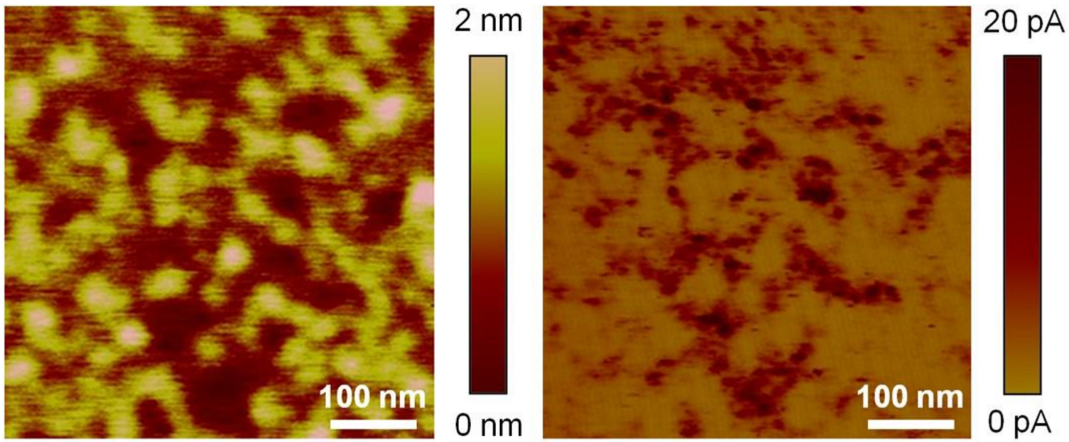
Use of electrically conducting probe connected to a transimpedance amplifier.



Applications:

Semiconductors, solar cells, 1D and 2D materials

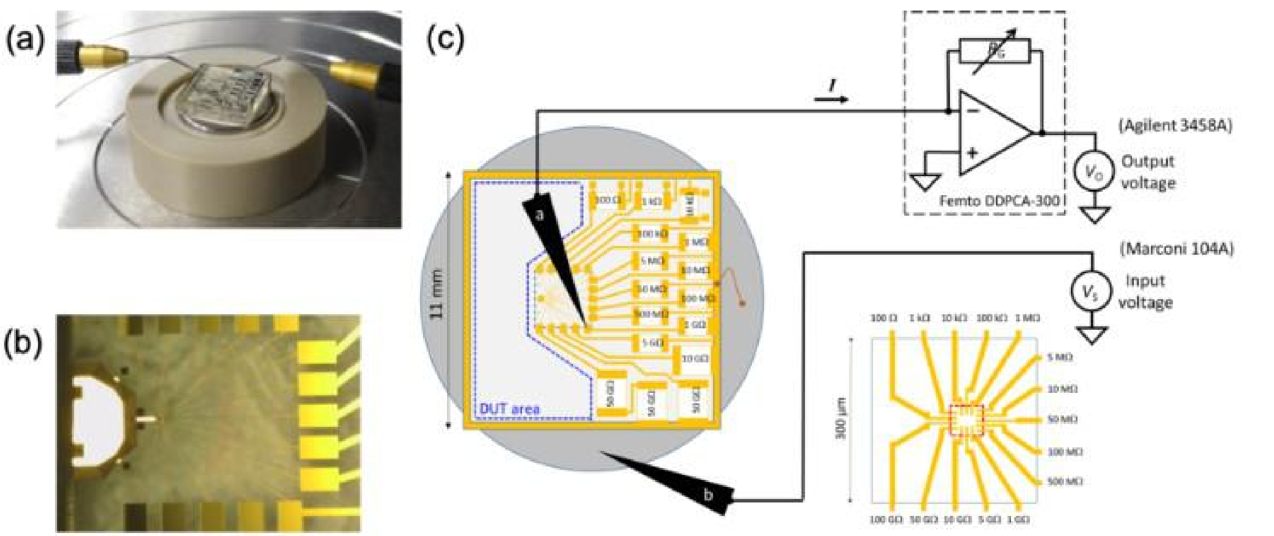
Image source: Park Systems, Wikipedia, Nanosurf

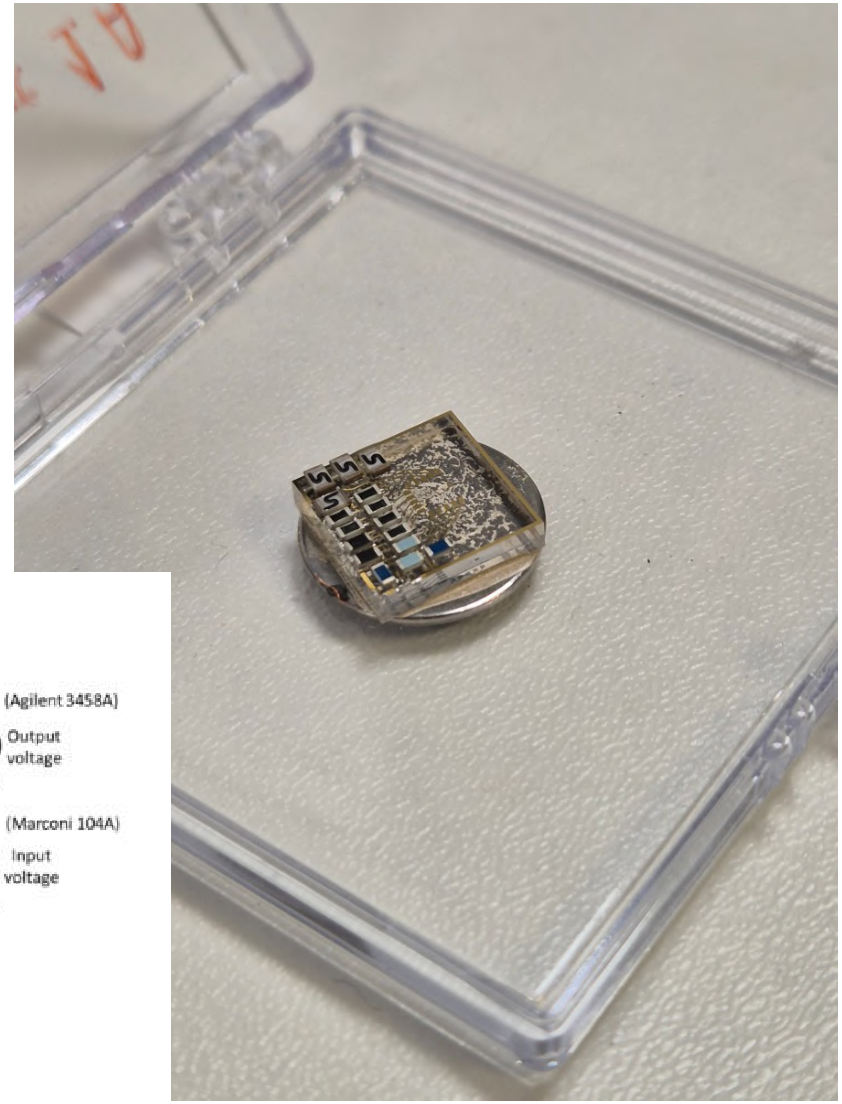


C-AFM reference samples?

“Multi-resistance standard” developed by French metrology institute LNE, within project EMPIR ELENA.

Set of SMD resistors with many decades of resistance, mounted on a glass block, with pads leading to the central part measurable using C-AFM.



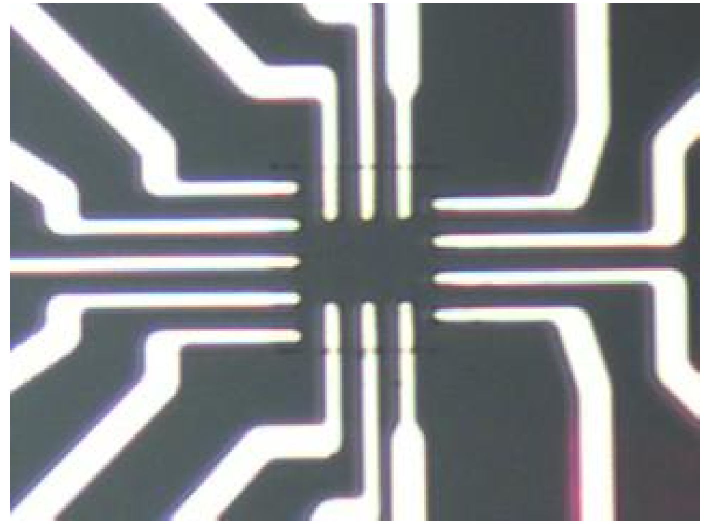


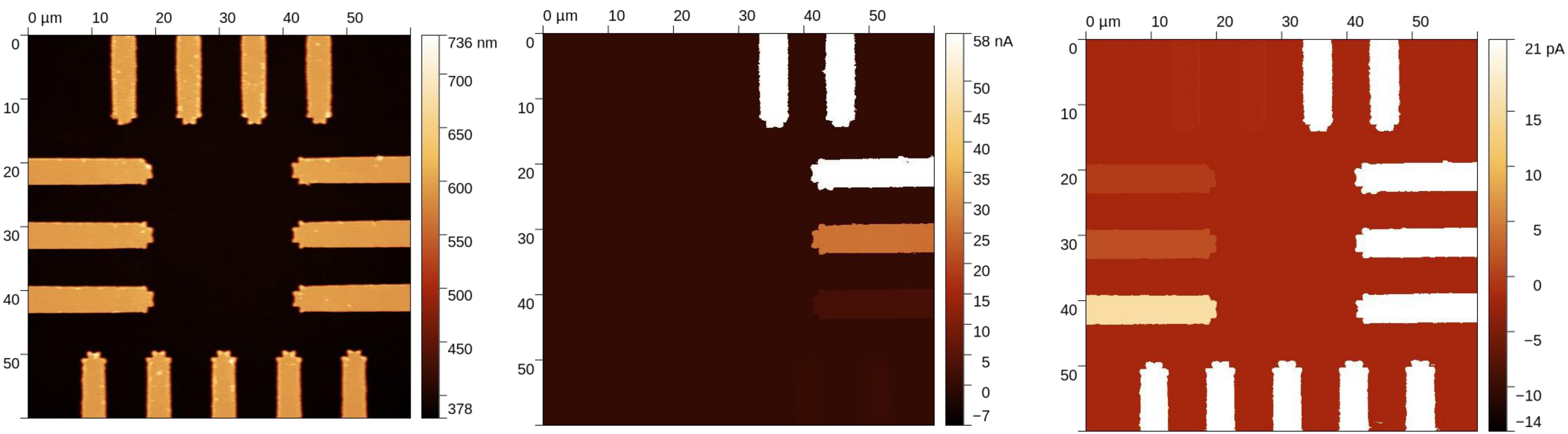
C-AFM reference samples

How to use the calibration sample?

Mapping the sample conductivity with some probe-sample bias.

Limits of the transimpedance amplifier range – with single settings we can get reasonable signal on few pads only. Logarithmic amplifier, as used in SSRM would be better.

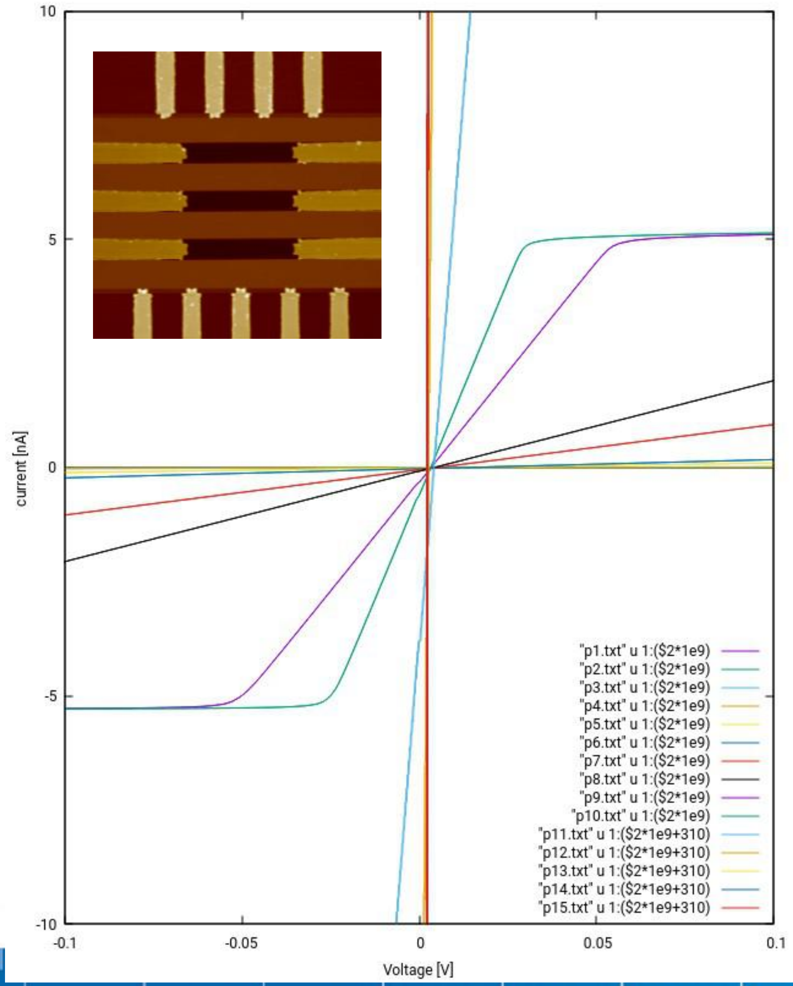


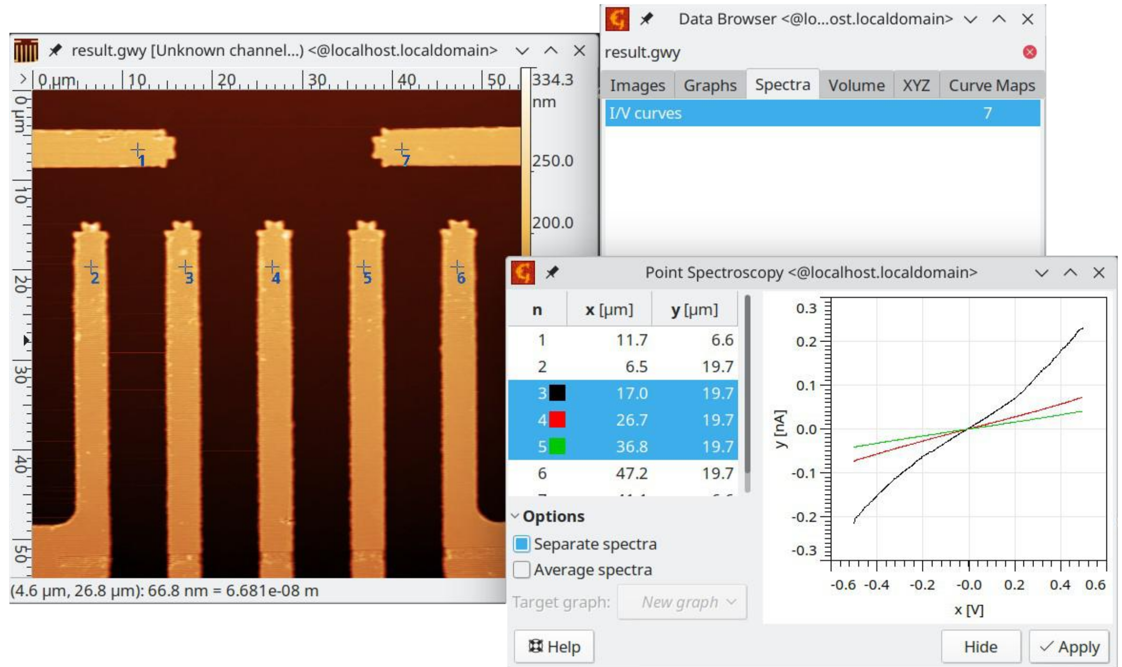


C-AFM reference samples

Measurement of I/V curves

Potential problems with parasitic capacitance when not connected properly.





Conductive wrap up

Conductive AFM measurements guidelines:

- calibrate your transimpedance amplifier (at least once)
- use solid body probes as coated ones can wear
- larger force is usually better
- do not believe in data obtained on rough samples

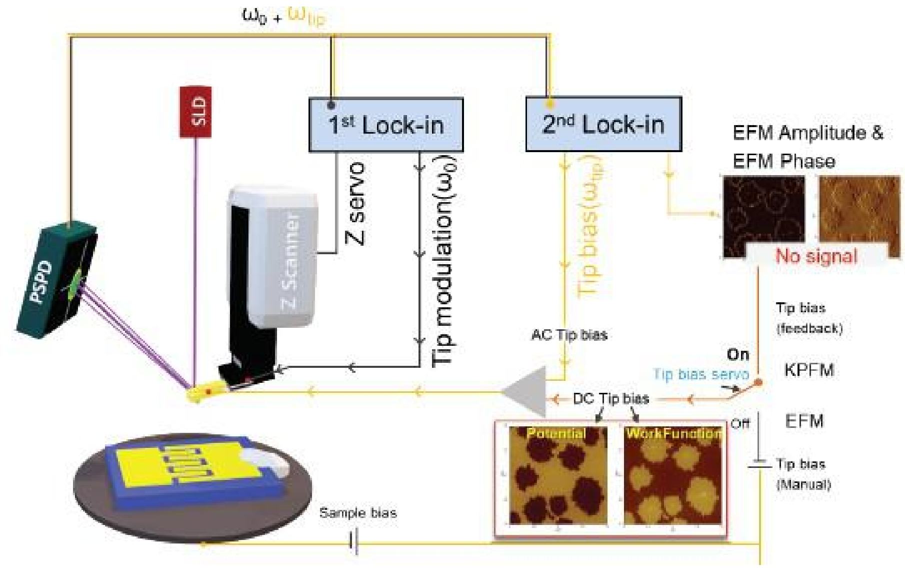
KPFM

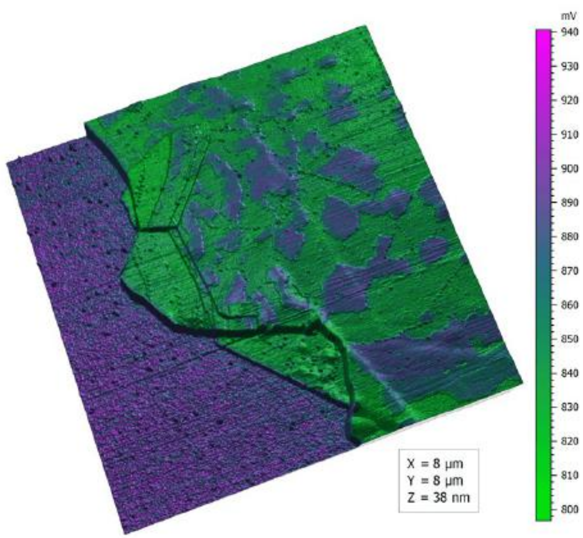
Kelvin Probe Force Microscopy

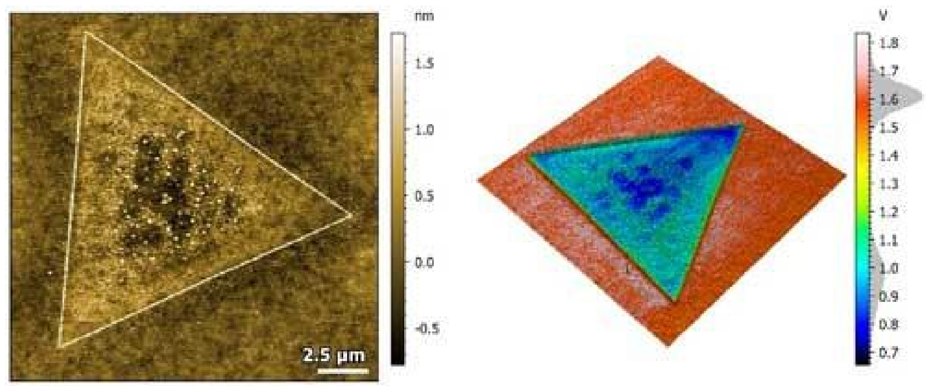
Monitoring electrostatic force between probe and sample and adjusting probe bias to minimize it … measurement of contact potential difference.

Applications:

Semiconductors, 1D and 2D materials.







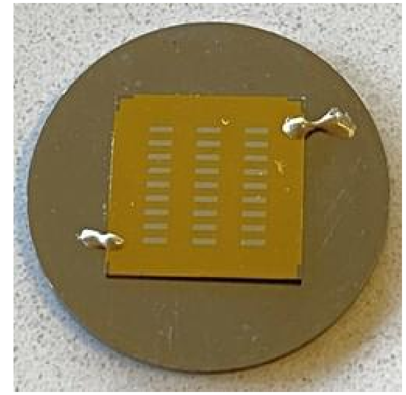
2D materials KPFM, Image sources: Park systems, Nanosurf

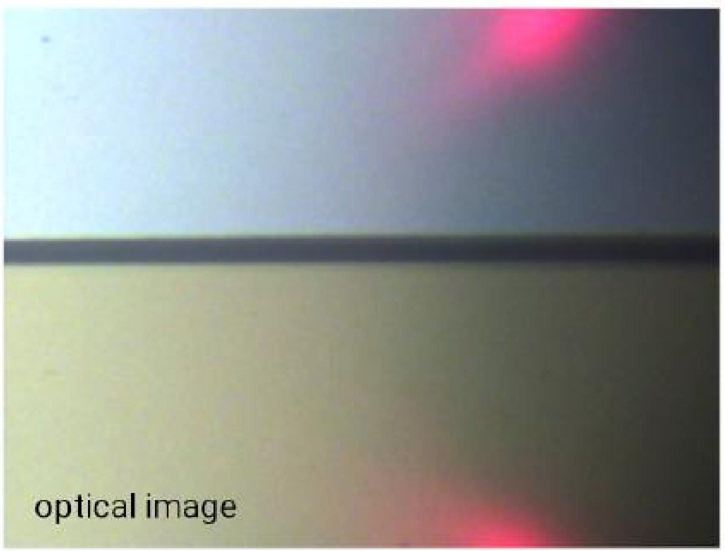
KPFM traceability and test samples

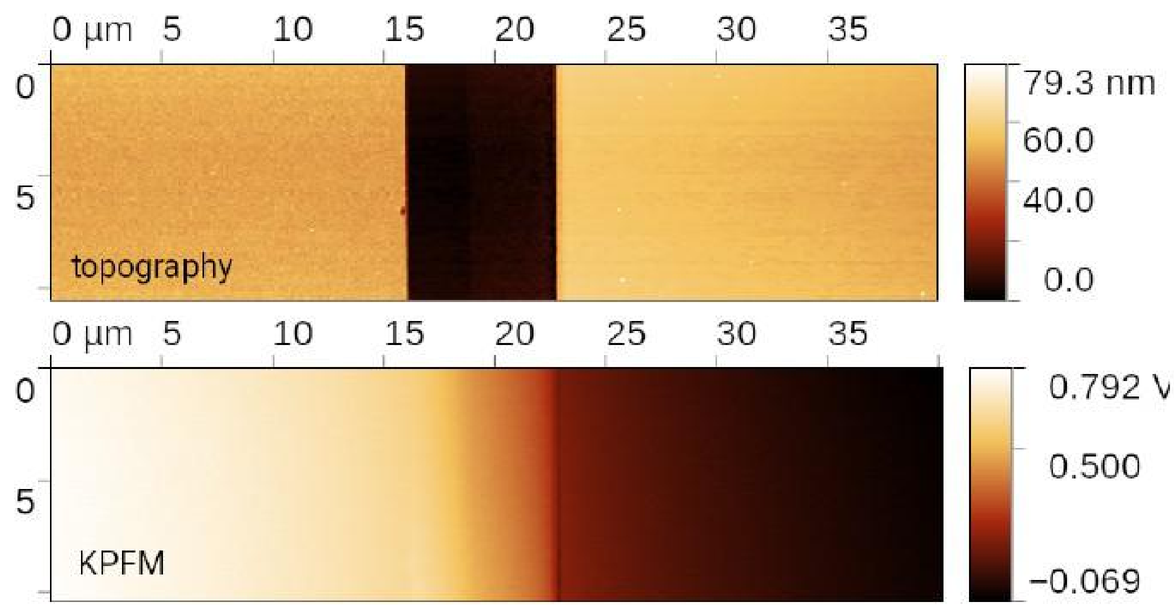
Bruker reference sample: aluminium and gold on silicon

Benefits: cheap, widely used.

Drawbacks: on one transition can be measured in a single image, gap is large.





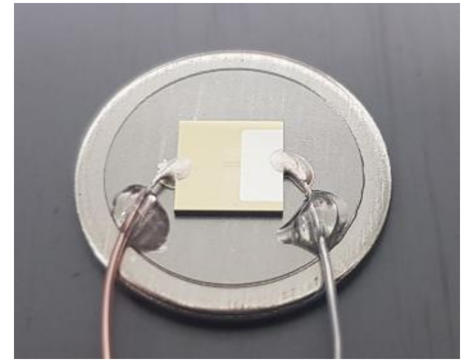


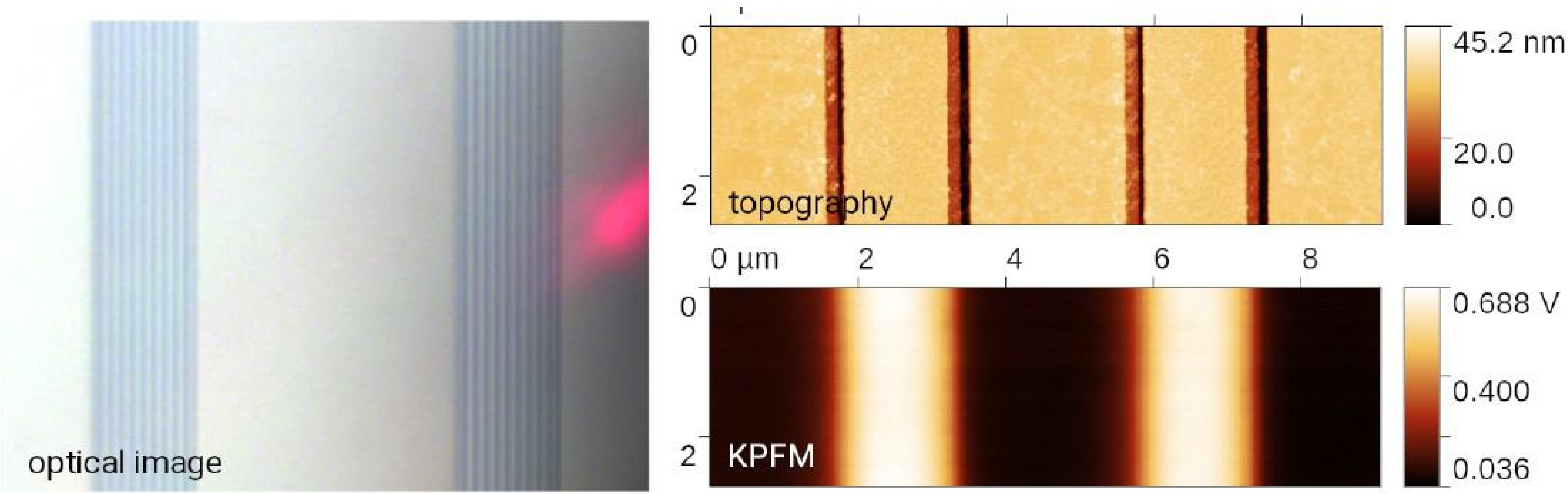
KPFM traceability and test samples

BudgetSensors: interleaved electrode arrays

Benefits: external voltage can be set on the electrodes, determining the KPFM scale and linearity.

Drawbacks: smallest electrodes are micrometer sized.



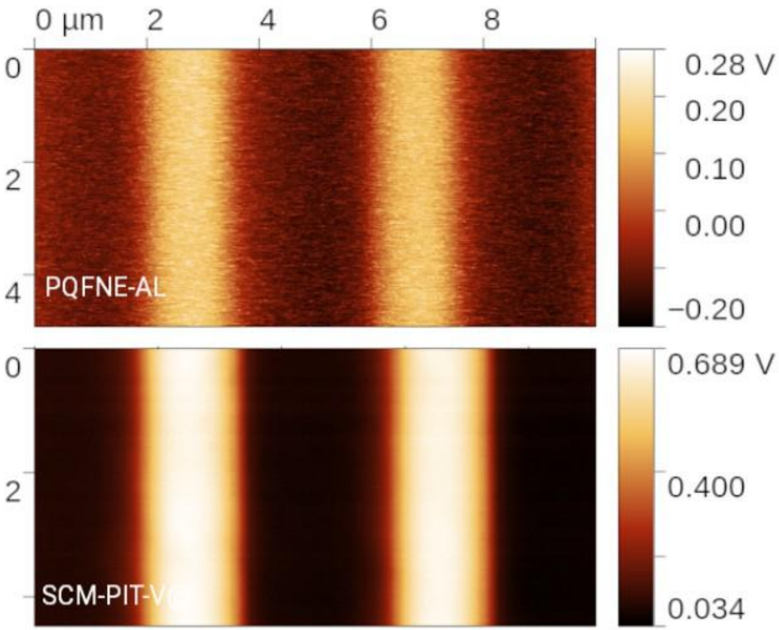


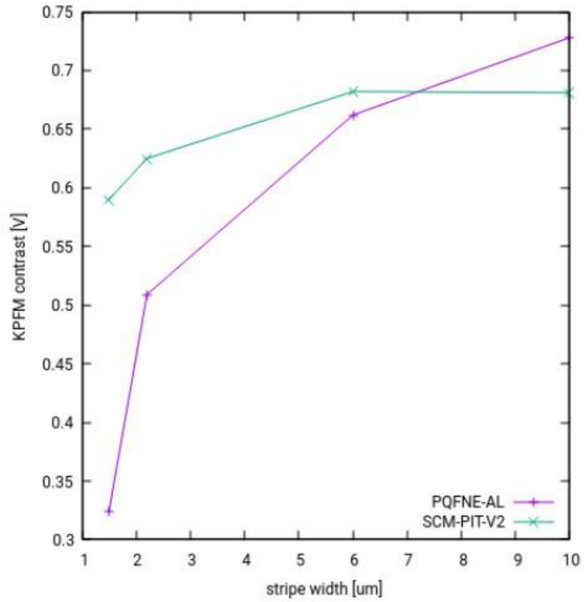
Spatial resolution vs. accuracy

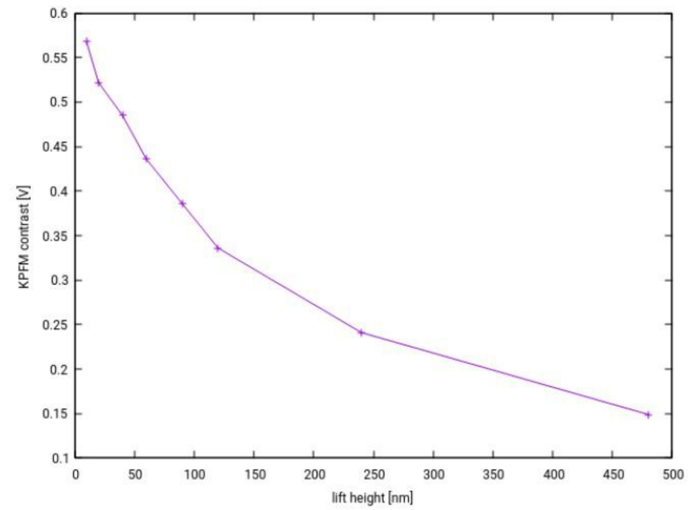
How accurately we can get V_{CPD} depending on the feature size?

When using AM-KPFM, a reasonable result could be obtained only on quite large features, in micrometer scale.

It is important to measure in as low lift height as possible, when using two-pass techniques.



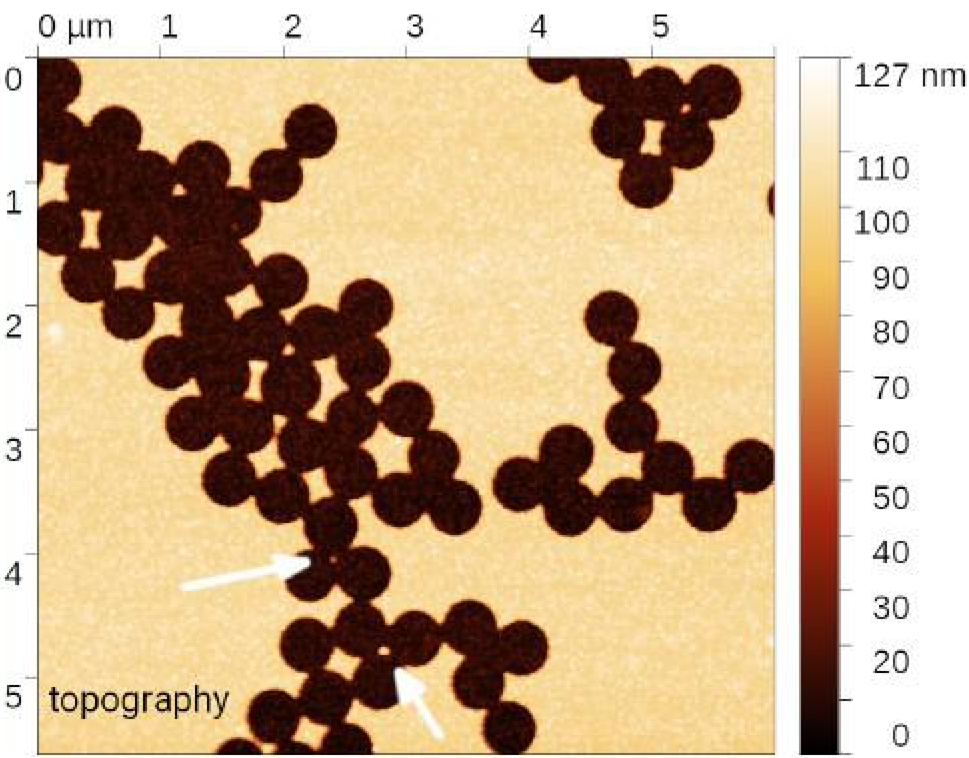


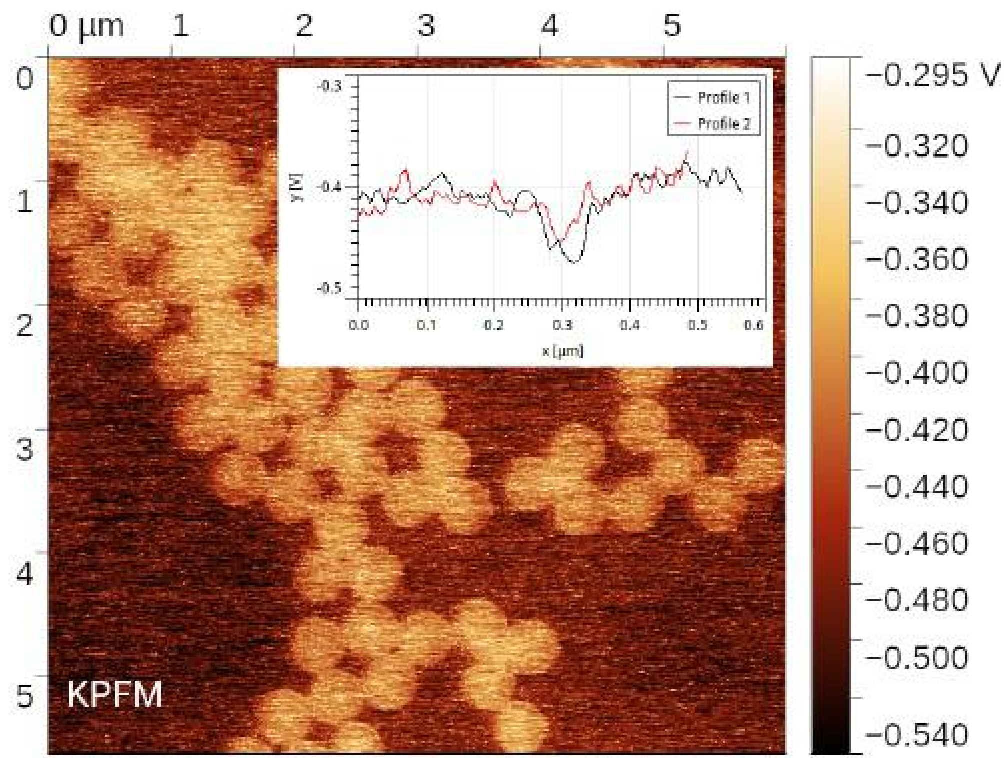


Spatial resolution vs. accuracy

Monolithic PQFNE-AL probe was best in term of resolution in our studies.

How to determine resolution?
One option is to search for smallest resolvable islands.

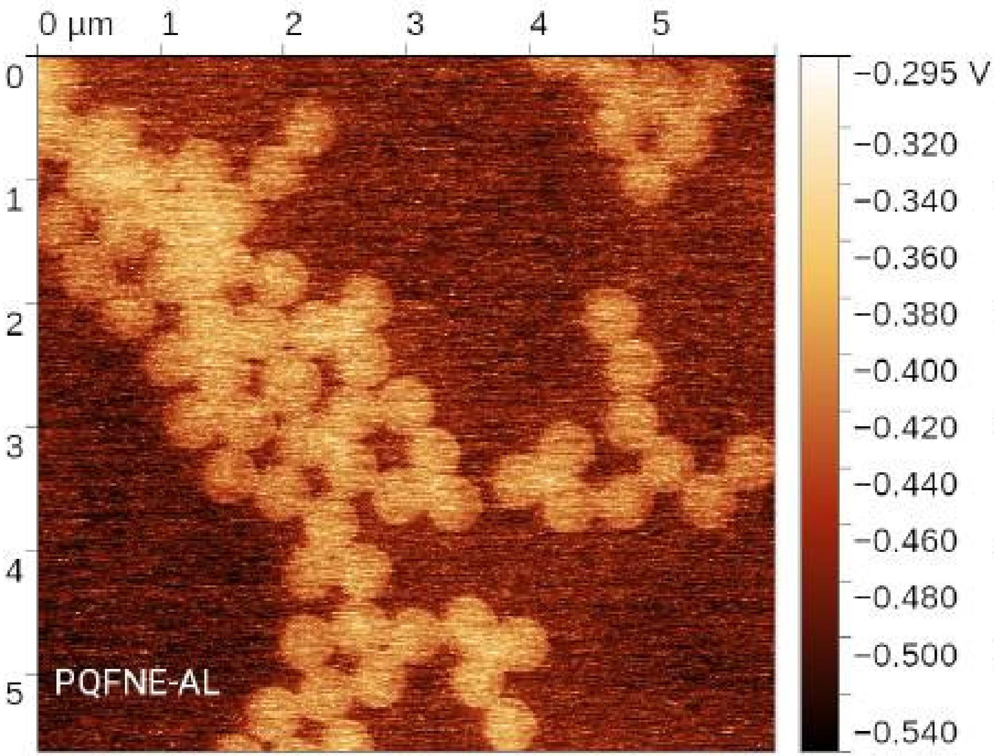


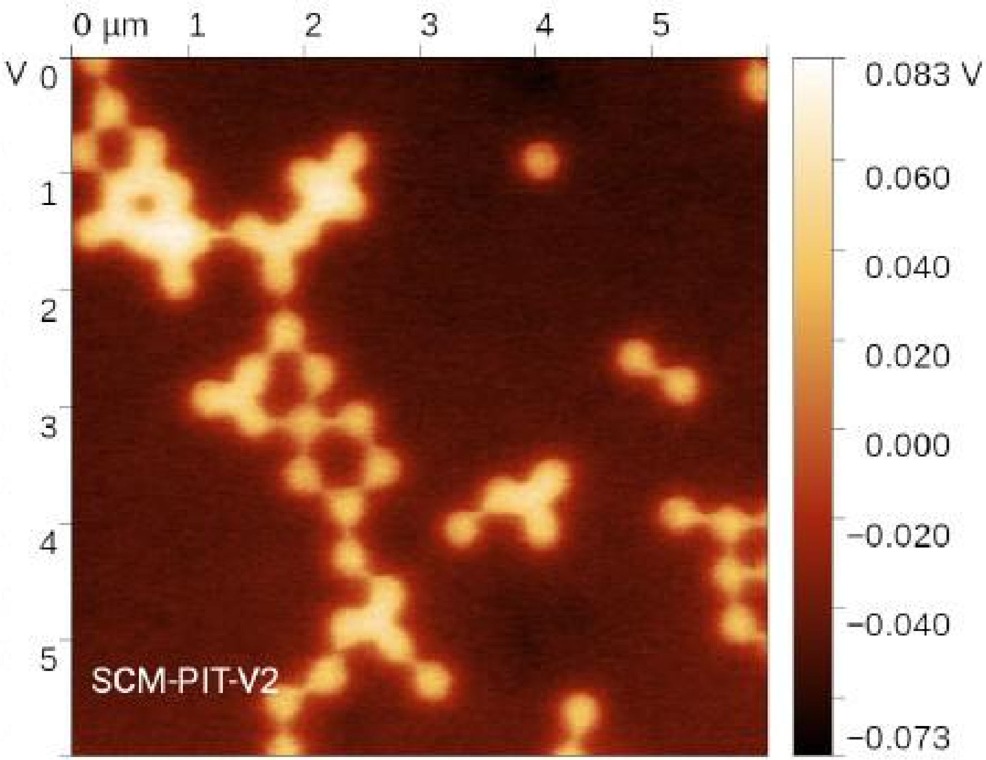


Spatial resolution vs. accuracy

How to determine resolution?
Second option is to search for shape of signal on an edge.

- resolution with PQFNE-AL probe: 15 nm,
- resolution with SCM-PIT-V2 probe: 100 nm.



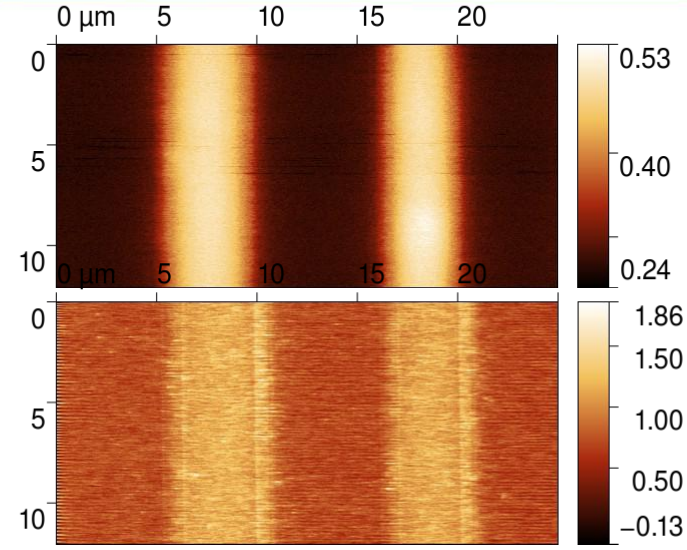


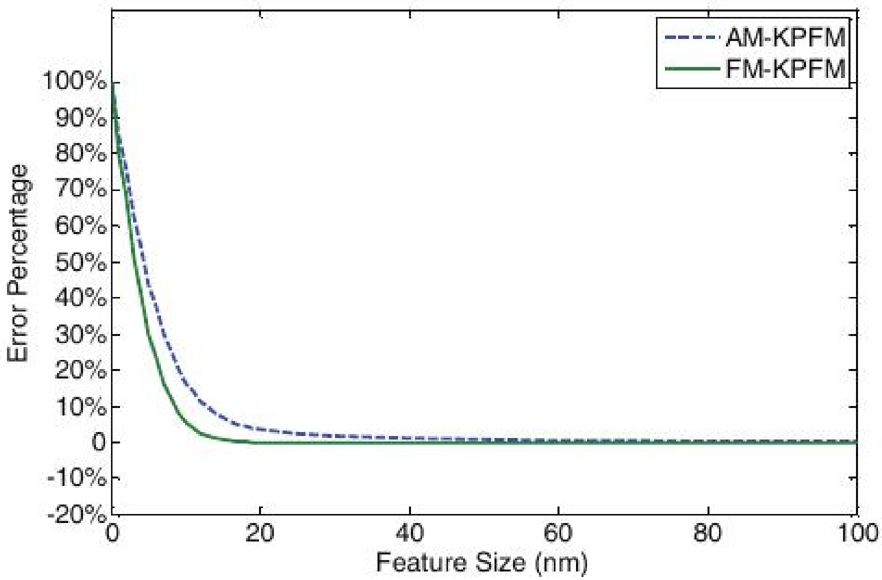
Spatial resolution vs. accuracy

How to increase the resolution?

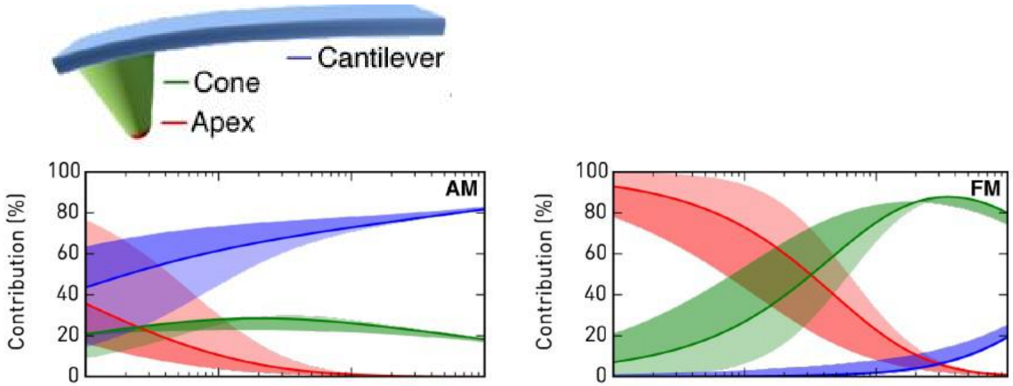
Use FM-KPFM which uses more localized interaction, force gradient instead of its value.

It is less sensitive to the impact of cantilever and other long range force sources, but can be more noisy.





Li *et al.* Rev. Sci. Instrum. **83**, 113701 (2012)



T. Wagner et al.

Beilstein J. Nanotechnol. **2015,** *6,* 2193–2206.

doi:10.3762/bjnano.6.225

Kelvin probe wrap up

Kelvin probe measurements guidelines:

- use FM mode if you want to get better resolution
- use specialized probes for KPFM if you have them
- use some test sample before measurements to check that everything works
- note that every probe produces different CPD, calibrate if, e.g. on HOPG

Scanning Microwave Microscopy

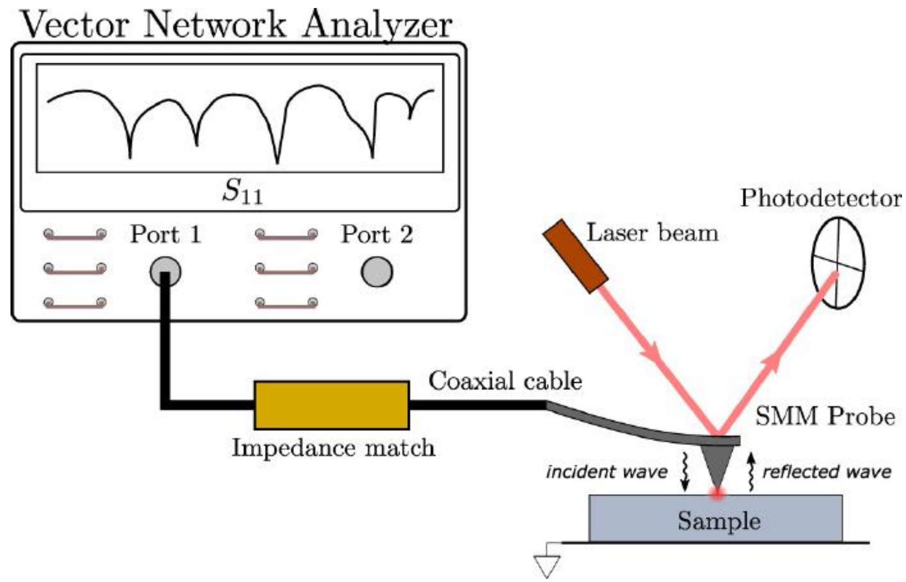
Conductive probe + Vector network analyzer

Frequency 1-20 GHz, rarely even up to 100 GHz

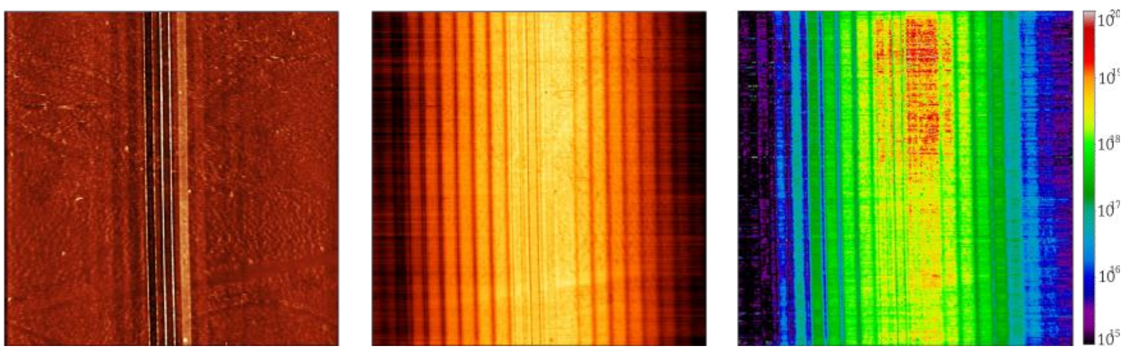
Impedance matching element can be complicated, frequency dependent and expensive.

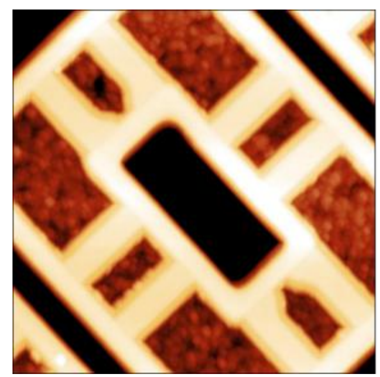
As an output, VNA provides reflection coefficient, a complex number, called S_{11}

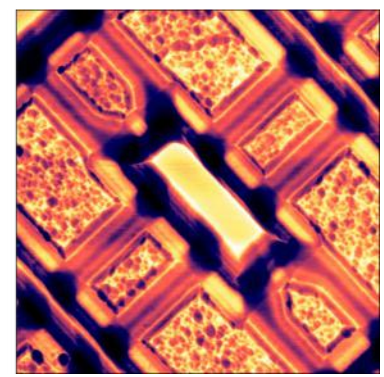
This can be used to address sample dielectric properties.



Sample results from Nanosurf (dopants, SRAM)







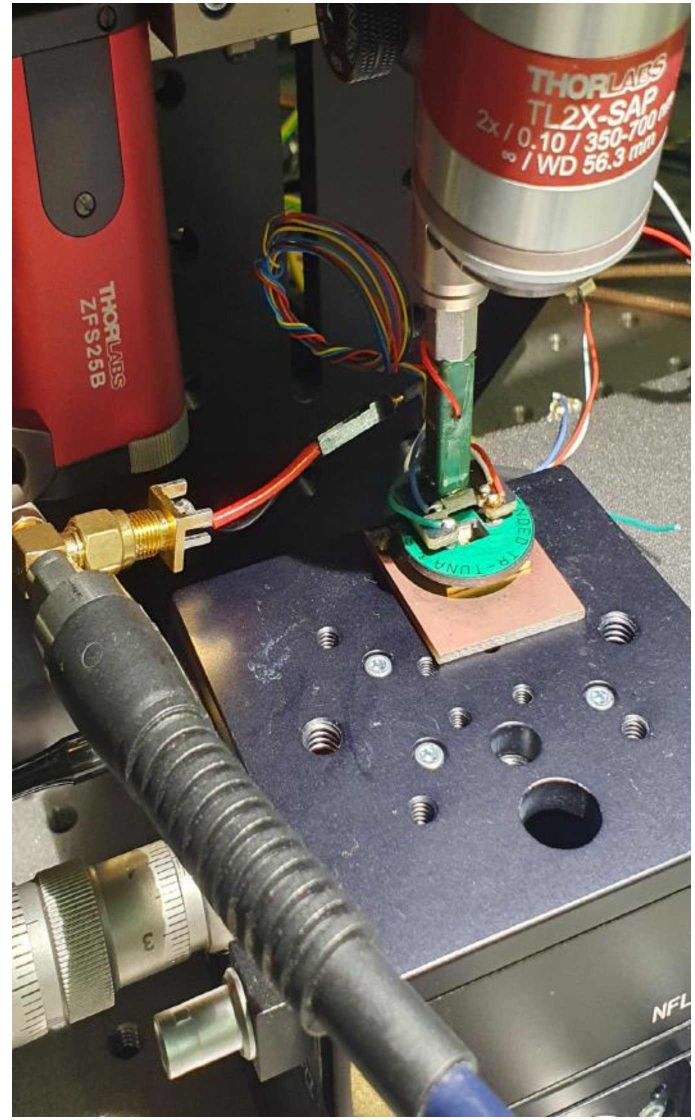
Scanning Microwave Microscopy

Only few options how to buy it.
Can it be done as custom built instrument?

Keysight PNA (> 30 kEuro), 128 dB range

PicoVNA (~ kEuro), 118 dB range

LibreVNA (~ 600 Euro), 100 dB range

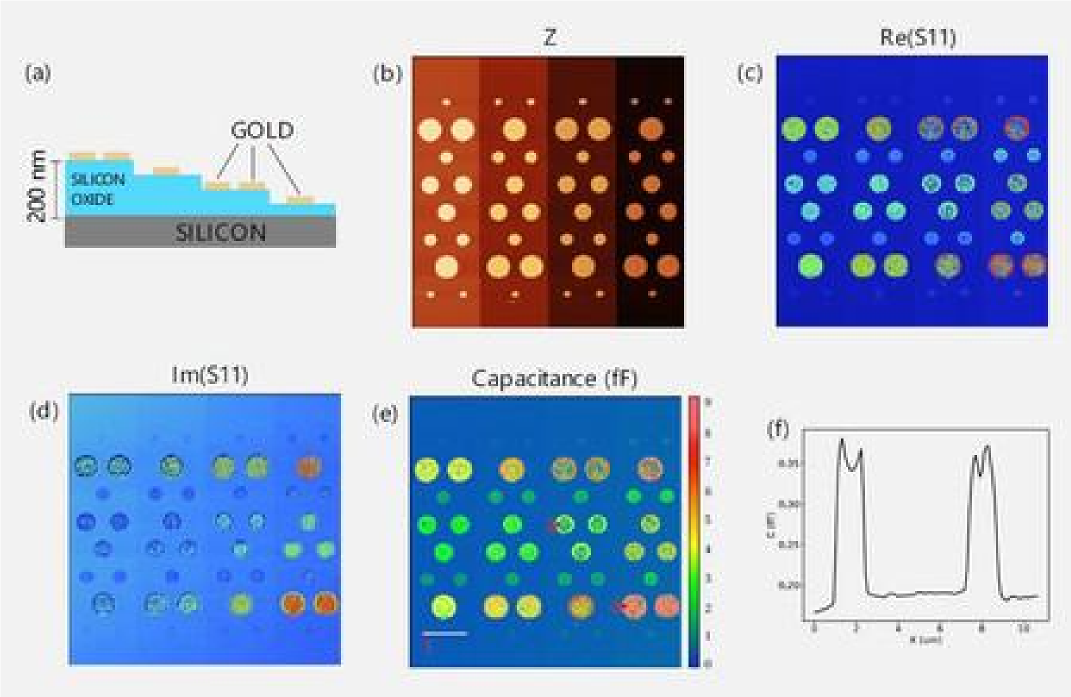


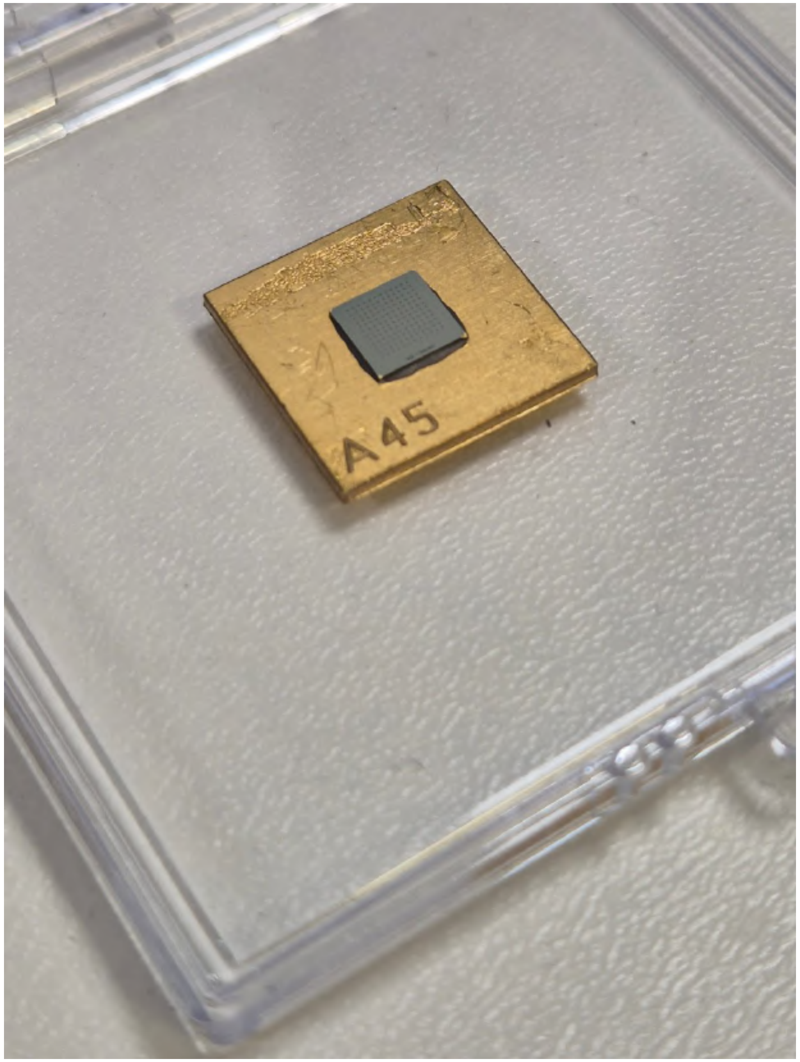
SMM reference samples

SMM reference samples

Small capacitors developed by MC2 company.

Gold pads on silicon dioxide with varying thickness and pad size. Very wide range of capacitances in fF range.



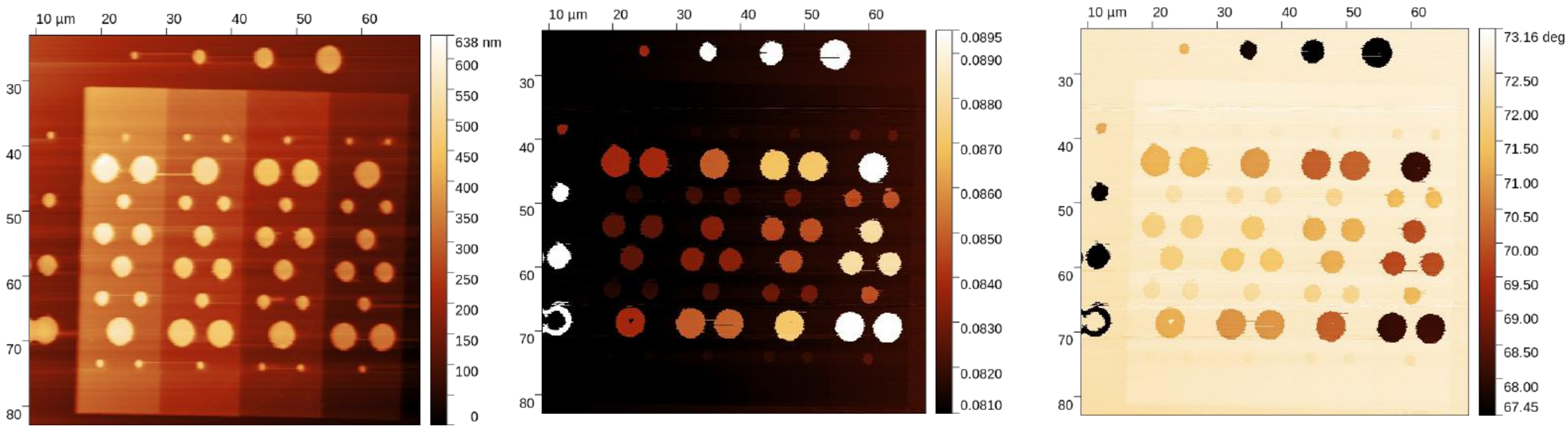


SMM reference samples

Testing a simple setup based on PicoVNA on MC2 calibration sample.

Probes are crucial – here we use full platinum Rocky Mountains probe

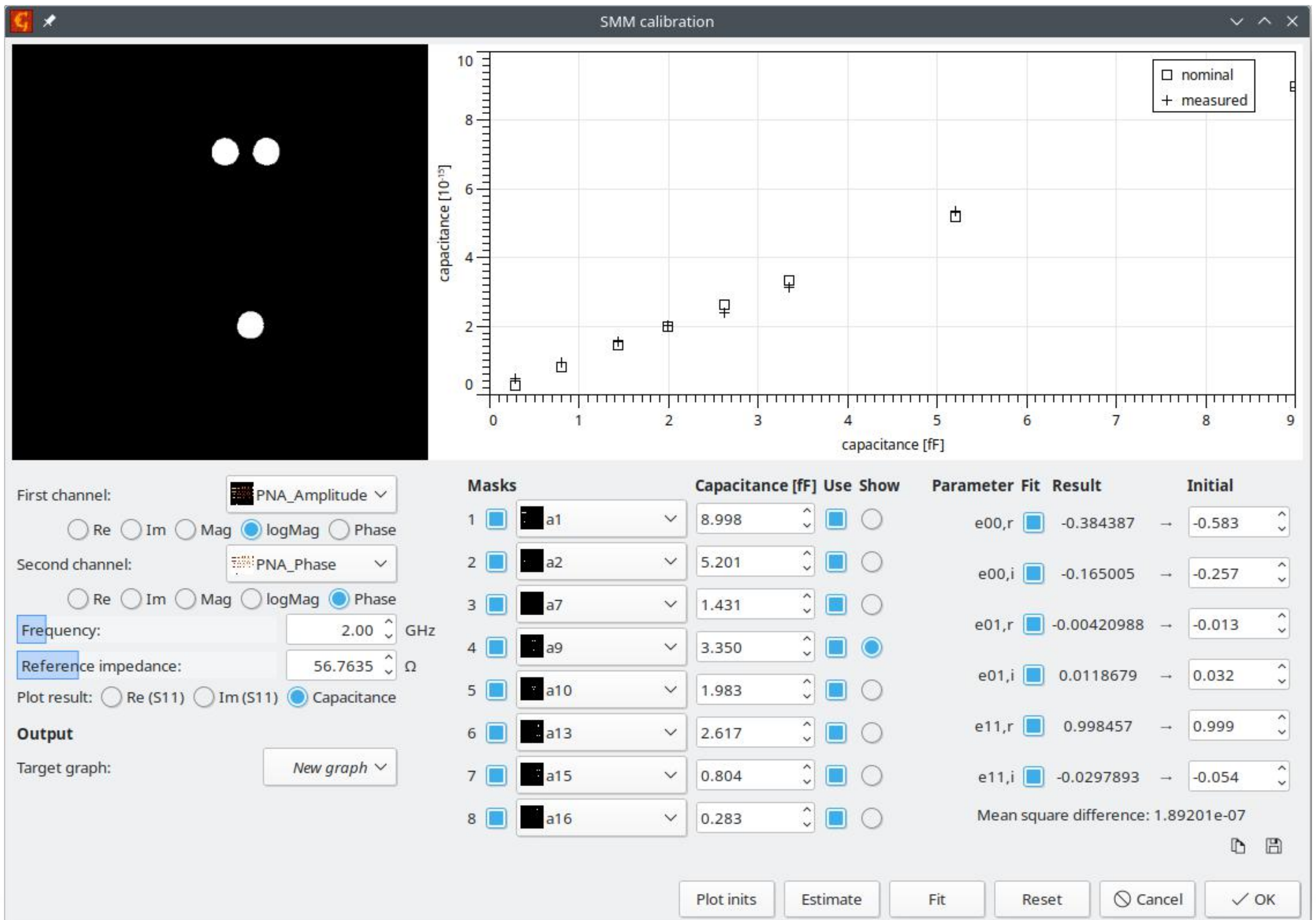
Using VNA we get log(magnitude) and phase signal in every pixel of the image.

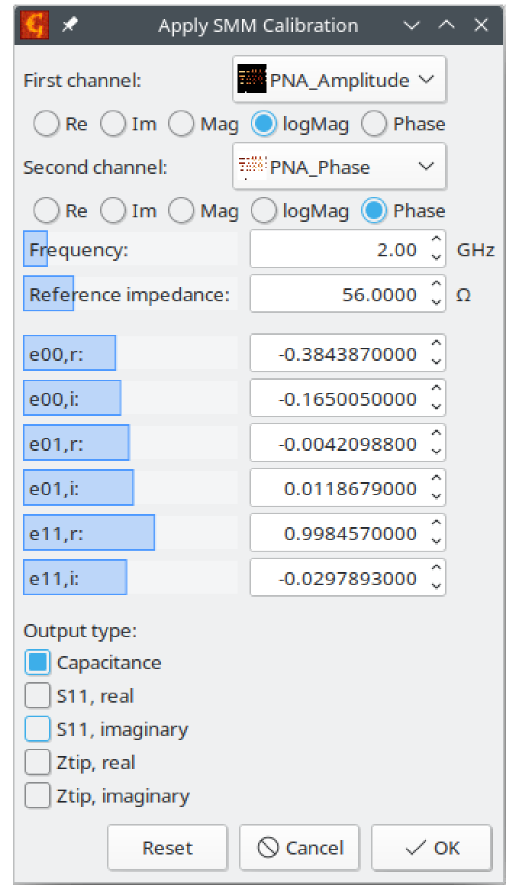


SMM calibration

We can calibrate our setup by using known capacitances and determining three complex parameters of the microwave circuit, using modified SOL calibration methodology.

See more in: Nanomaterials 2021, 11(3), 820; https://doi.org/10.3390/nano11030820



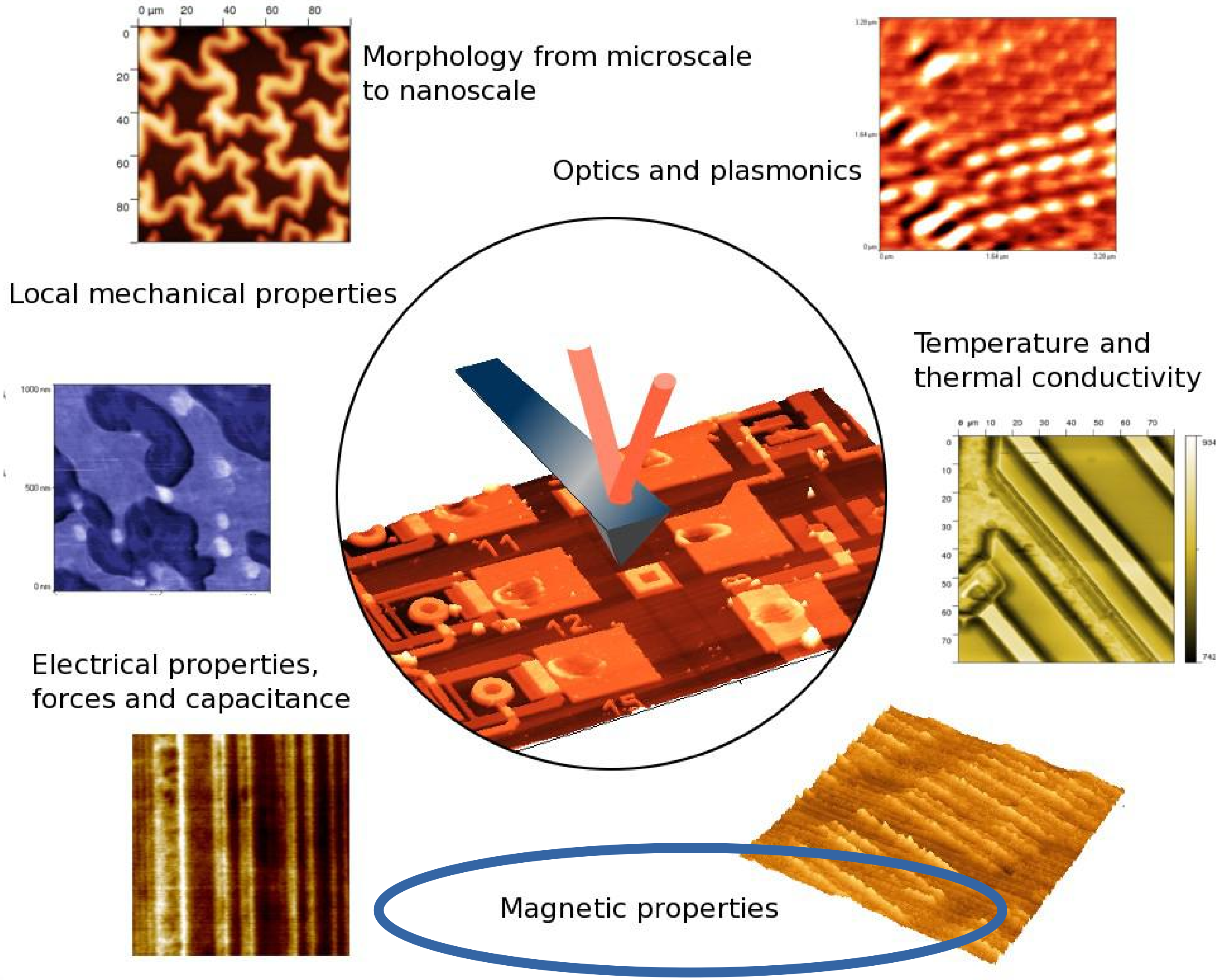


SMM wrap up

SMM measurements guidelines:

- know your setup, including impedance matching circuitry
- calibrate the response on known samples
- solid large probes work better

SPM – scanning probe microscopy



Magnetic force Microscopy

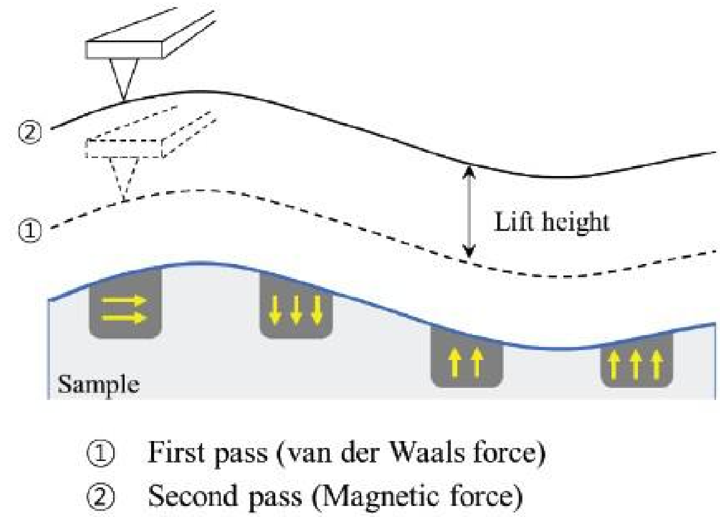
Use of a magnetically coated probe to address the stray field above the sample.

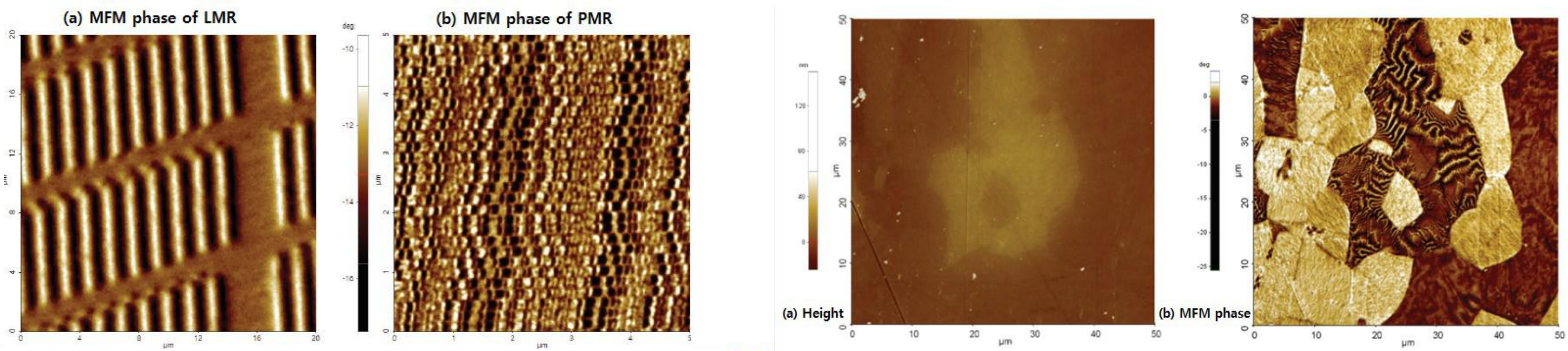
Two pass measurement or force-volume data acquisition.

Applications:

Data storage, materials, nano-magnetism

Image source: Park Systems (HDD, steel)





MFM traceability

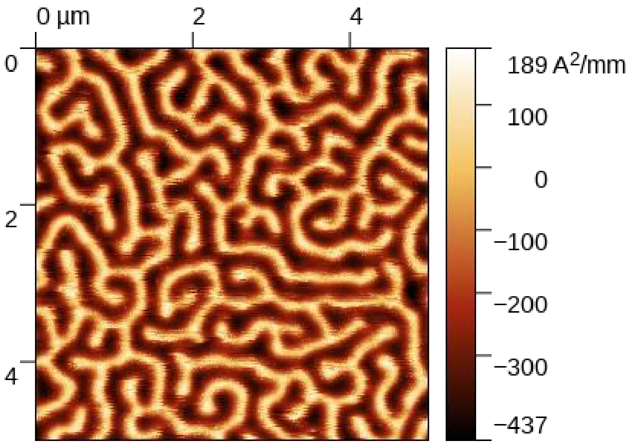
Quantitative MFM:

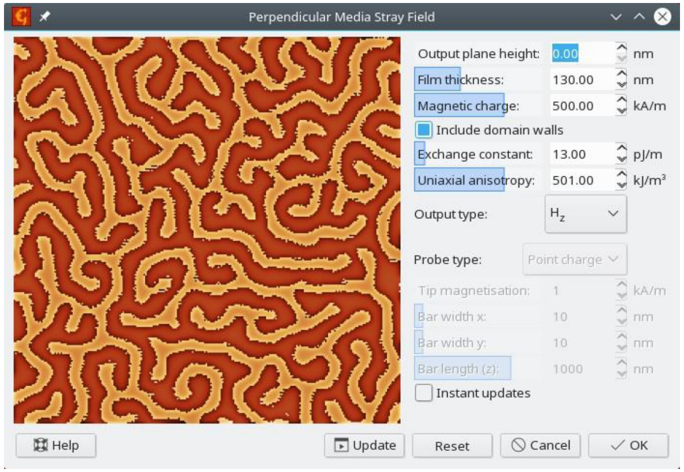
- Use a sample with calculable stray field (e.g. perpendicularly magnetised pattern in a multilayer).

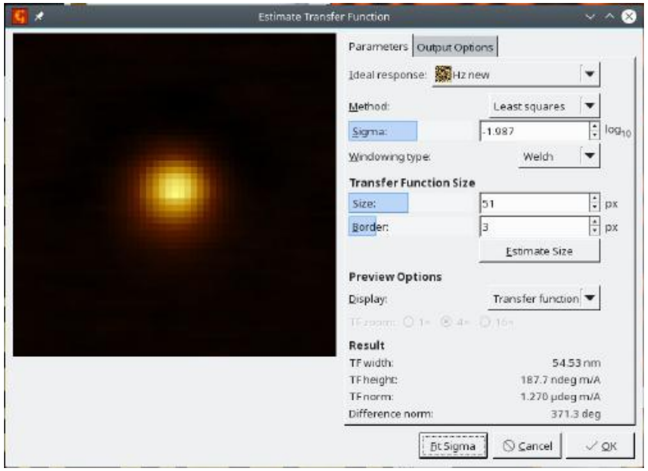
- characterize the probe with it, obtaining a tip transfer function.

- deconvolve the TTF from measurements on the unknown sample.

See more in Nečas et al, https://doi.org/10.1038/s41598-019-40477-x



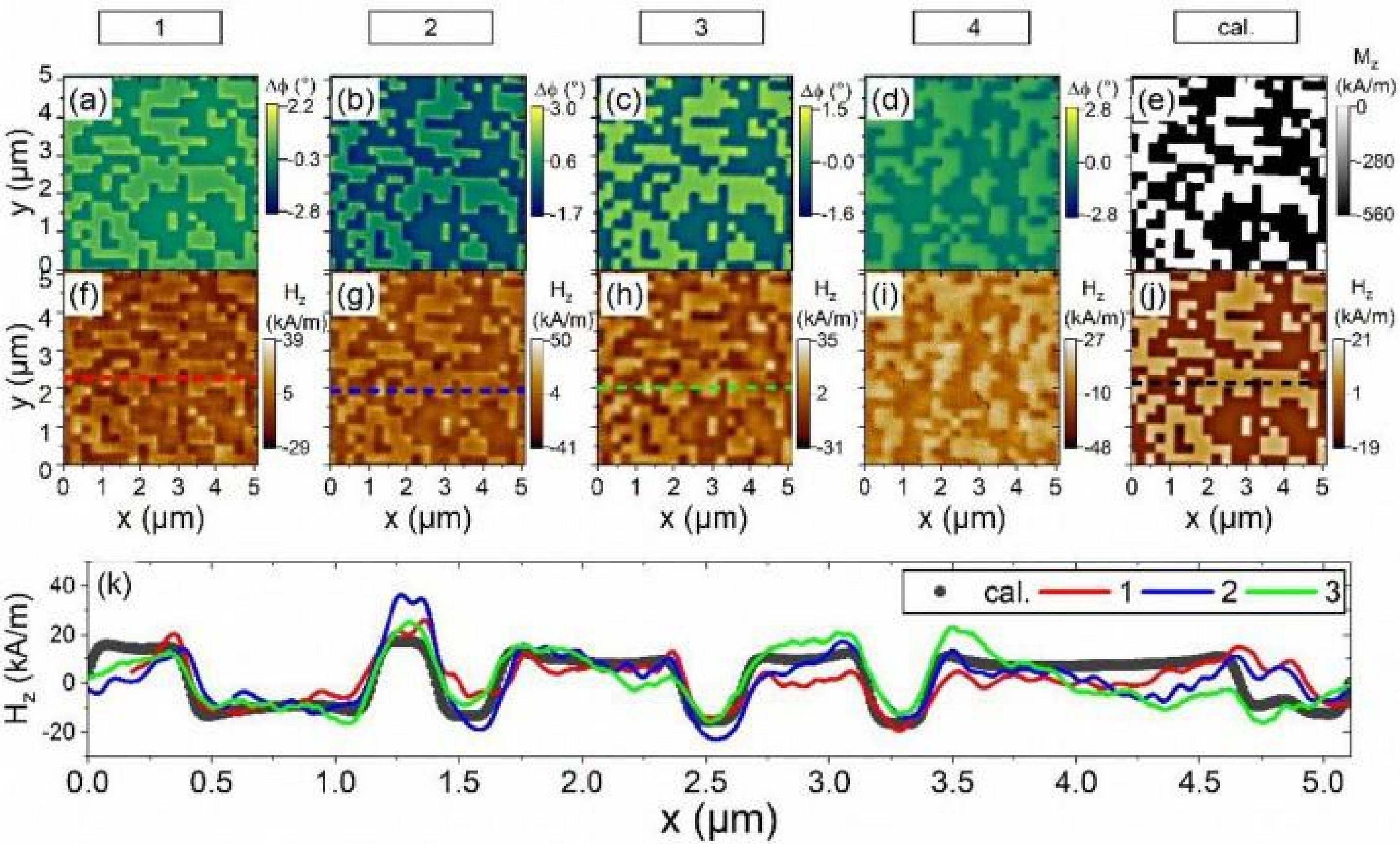




MFM traceability

EMPIR Nanomag project comparison using the quantitative MFM methodology:

See more in: X. Hu et al, https://doi.org/10.1016/j.jmmm.2020.166947

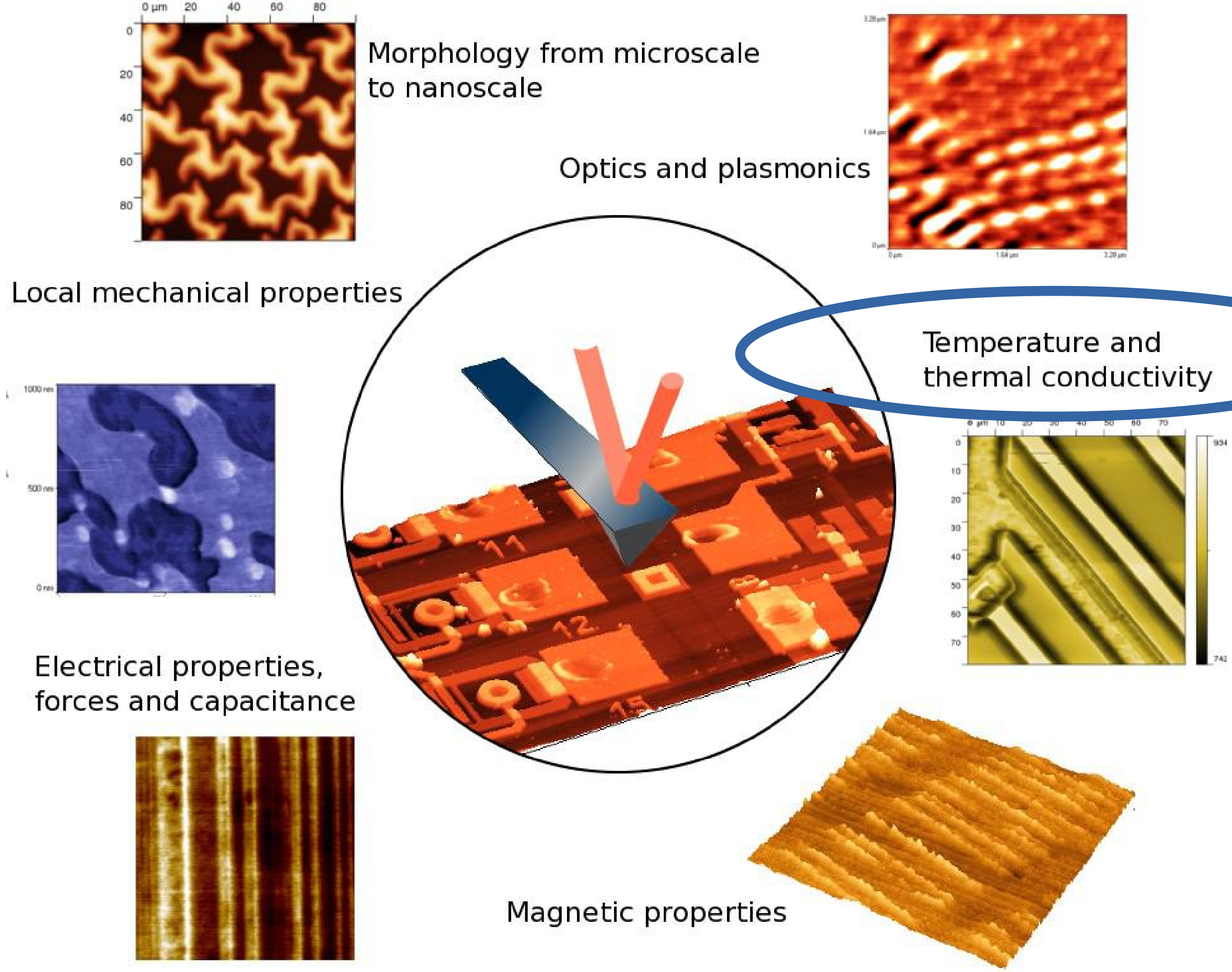


Magnetic wrap up

MFM measurements guidelines:

- get some reference samples
- calibrate your probe and use the whole quantitative MFM procedure
- scan in more lift heights to get rid of van der Waals forces
- be cautious if your probe has low magnetic moment and sample high

SPM – scanning probe microscopy



Scanning Thermal Microscopy

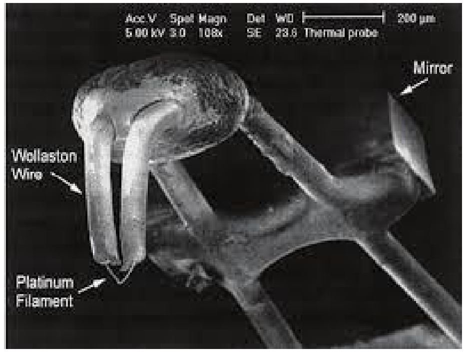
Scanning Thermal Microscopy (SThM)

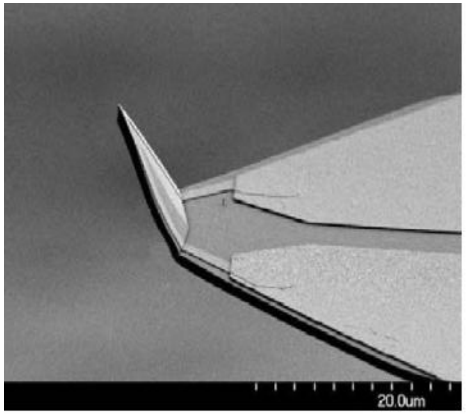
- use of a special probe that can generate heat and sense temperature.

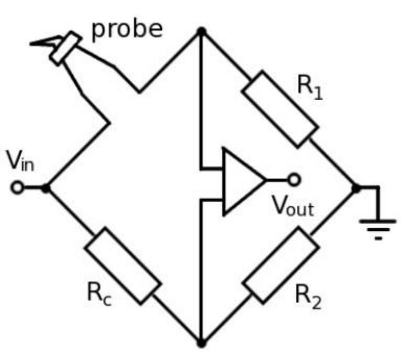
- **temperature** measurements: minimize probe self-heating, measure the probe resistance only with minimum current.

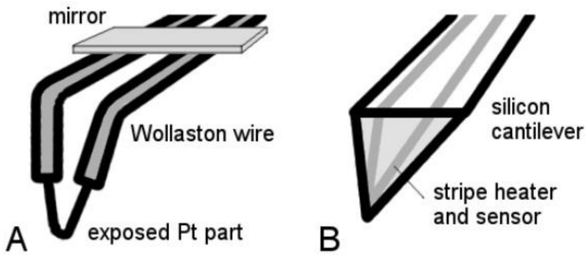
- **thermal conductivity** measurements: heat the sample using probe and monitor thermal losses.

- nanoscal thermal analysis: sample **transition temperature** by ramping the probe temperature and monitoring the mechanical response.









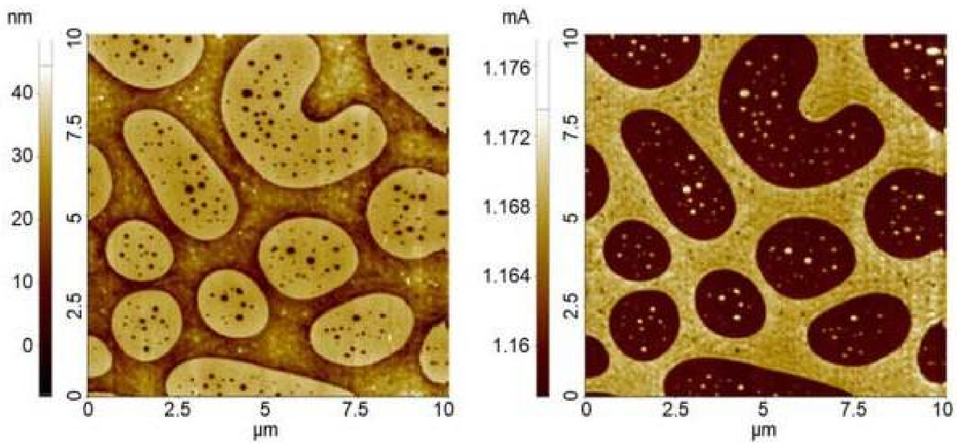
Scanning Thermal Microscopy

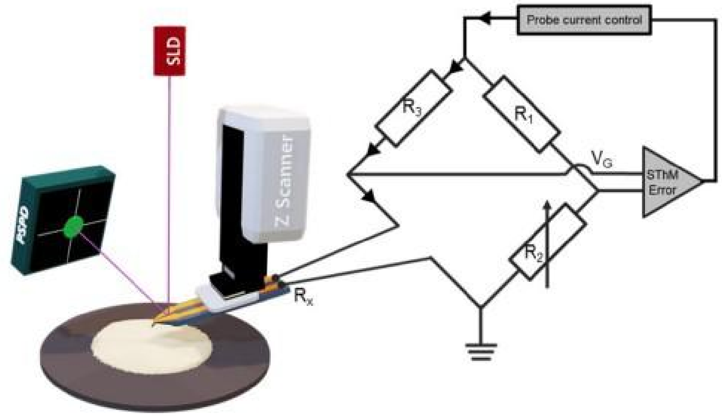
Temperature applications:
high power transistors, microelectronics, optoelectronic devices.

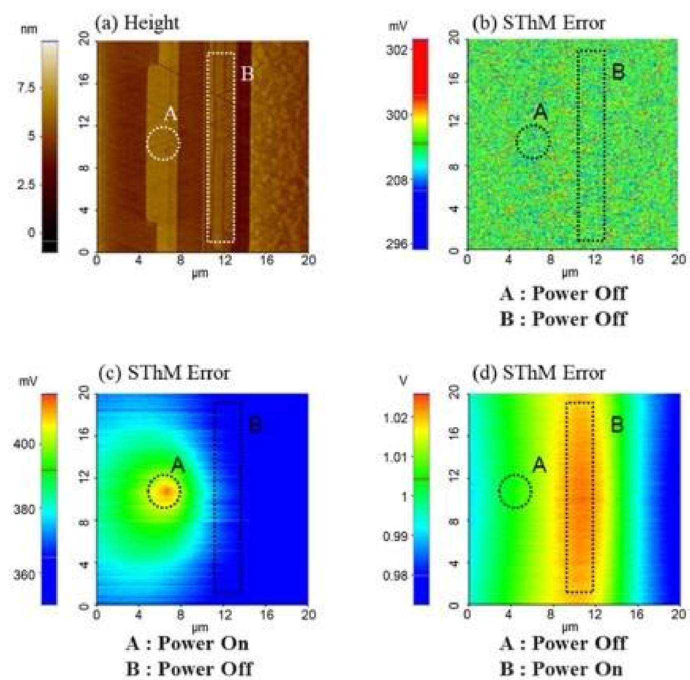
Thermal conductivity applications:
heat management materials, nanocomposites, 1D and 2D materials.

Nano thermal analysis applications:
polymers

Image source: Park Systems (polymer blend, HDD)



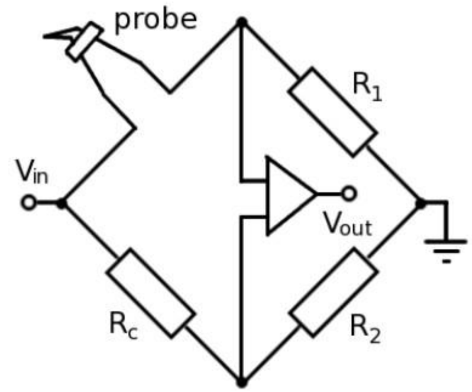




Thermal conductivity traceability

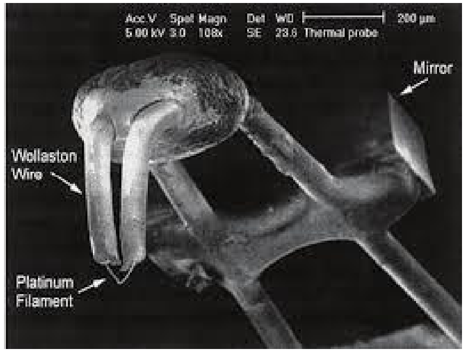
Local thermal conductivity

Our goal is to heat the sample with the probe (by passing current through it) and to use it as a sensor of the local temperature at the same time.



Measurement methodology

- in contrast to temperature measurements, do not minimize self heating
- use value far from the sample as a reference
- use set of calibration samples for traceability



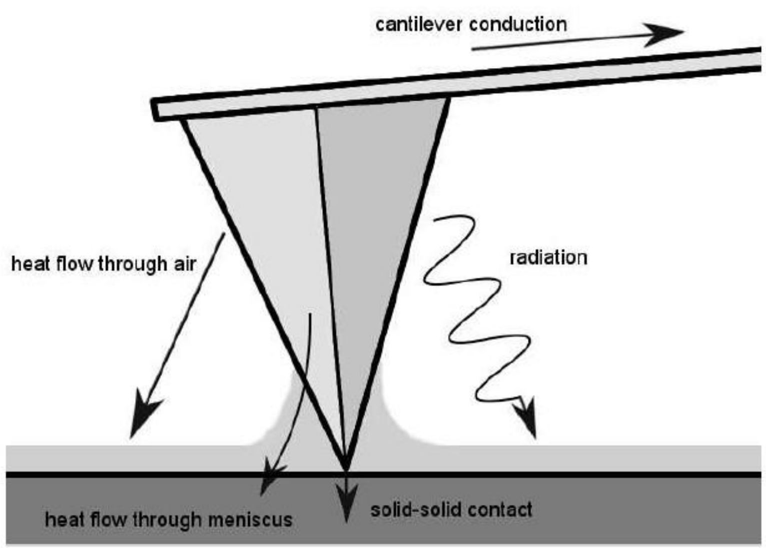
Potential calibration strategies:
A) measurement of the energy flow in the system
B) calibration on known reference samples

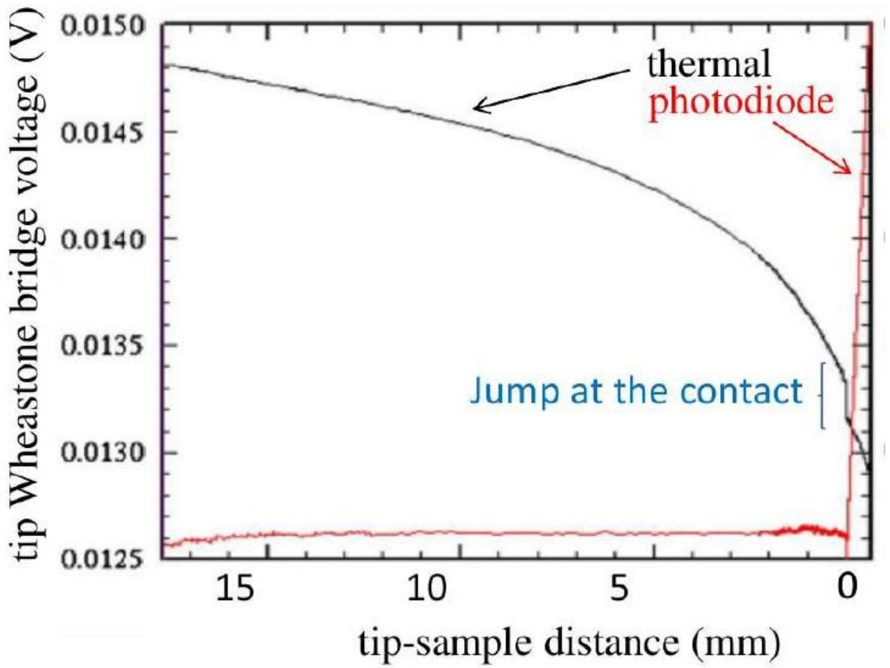
Thermal conductivity traceability

A) Calibration of thermal conductivity based on energy balance in the system

- problems with too many unknown heat transfer paths
- in most cases the solid-solid heat flow is only very small contribution

… really not an option in the present state of knowledge

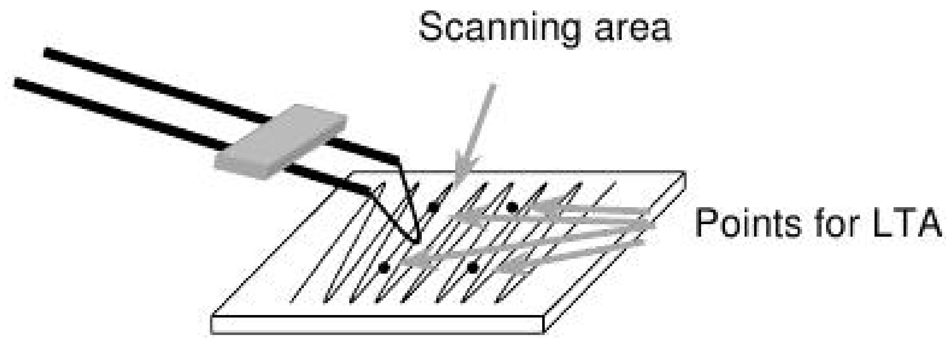




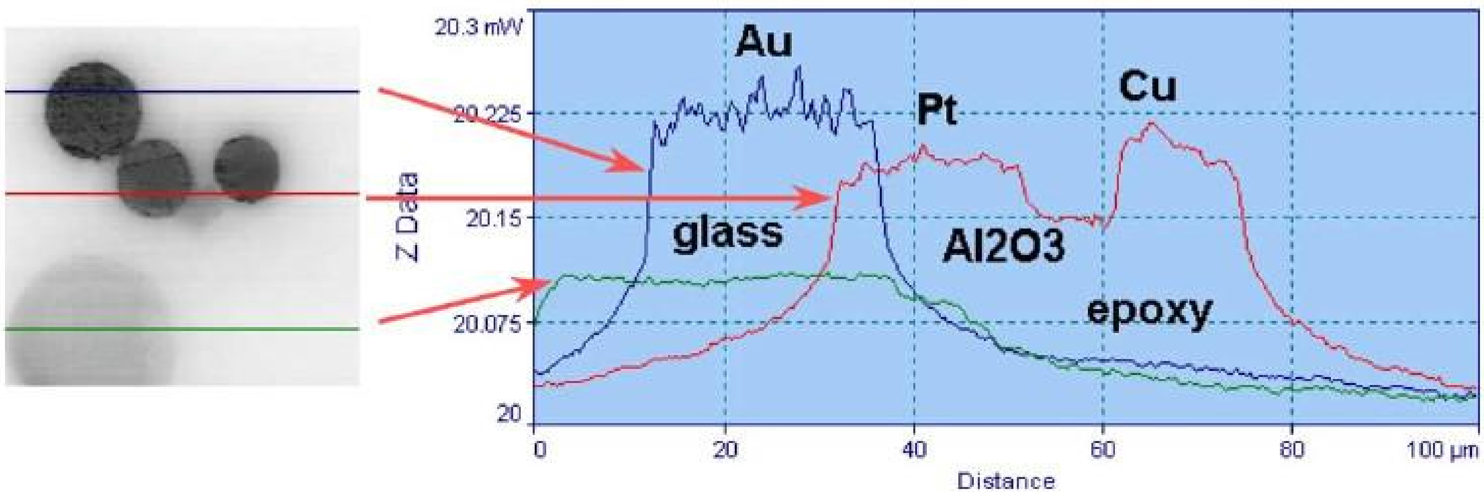
B) Calibration based on samples

Method proposed by Fischer, at present mostly used approach.

Ideally, we want to use sample with multiple known materials within single scan range (e.g. 100x100 micrometers).



H. Fischer / Thermochimica Acta 425 (2005) 69–74



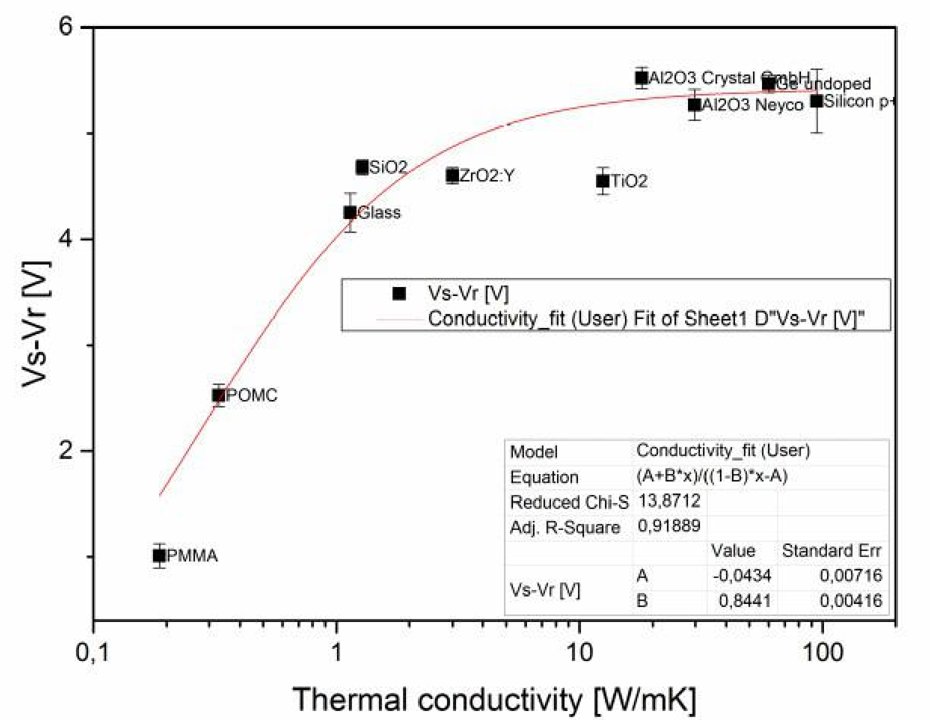
Thermal conductivity traceability

Bulk based calibration samples

Set of bulk samples prepared during Quantiheat project, measured by laser flash method.

Benefits: bulk samples, known thermal conductivity
Drawbacks: impact of roughness, different types of conductors

Sample	Th. C. [W/mK]	Vs-Vr[V]	Err.
PMMA	0.187	1.01	0.06
POMC	0.329	2.52	0.05
Glass	1.14	4.25	0.09
TiO_2	12.52	4.55	0.06
ZrO_2:Y	3	4.60	0.04
SiO_2	1.28	4.68	0.03
Al_2O_3 Neyco	29.8	5.27	0.07
Silicon p++	94.3	5.30	0.15
Ge undoped	60	5.46	0.04
Al_2O_3 Crystal GmbH	18	5.52	0.05



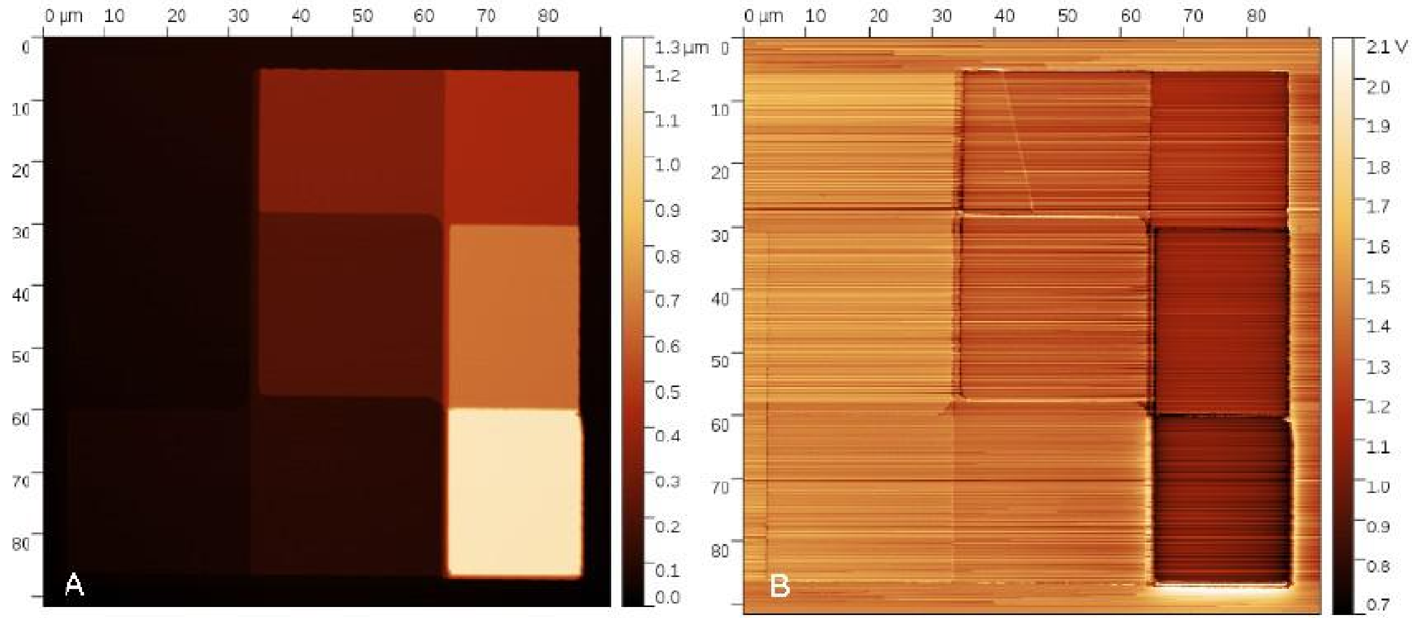
Thermal conductivity traceability

Thin film based calibration samples

Alternative sample from Glasgow university: silicon dioxide films with different thickness.

Benefits: smooth surface, similar on all parts of the sample.
Drawbacks: limited conductivity range, missing reference value.

topography and thermal signal on the Glasgow Quantiheat sample

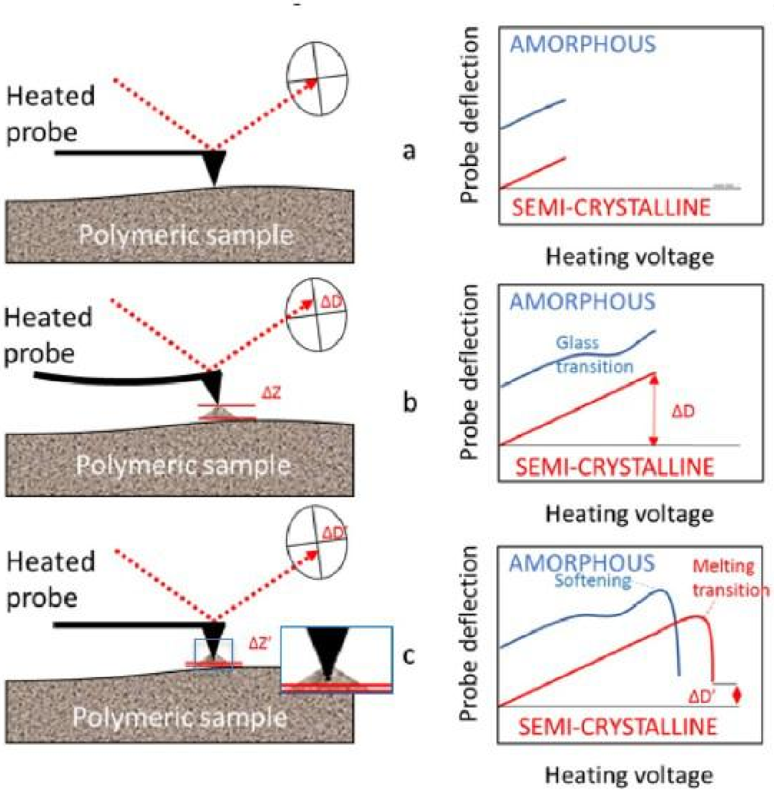


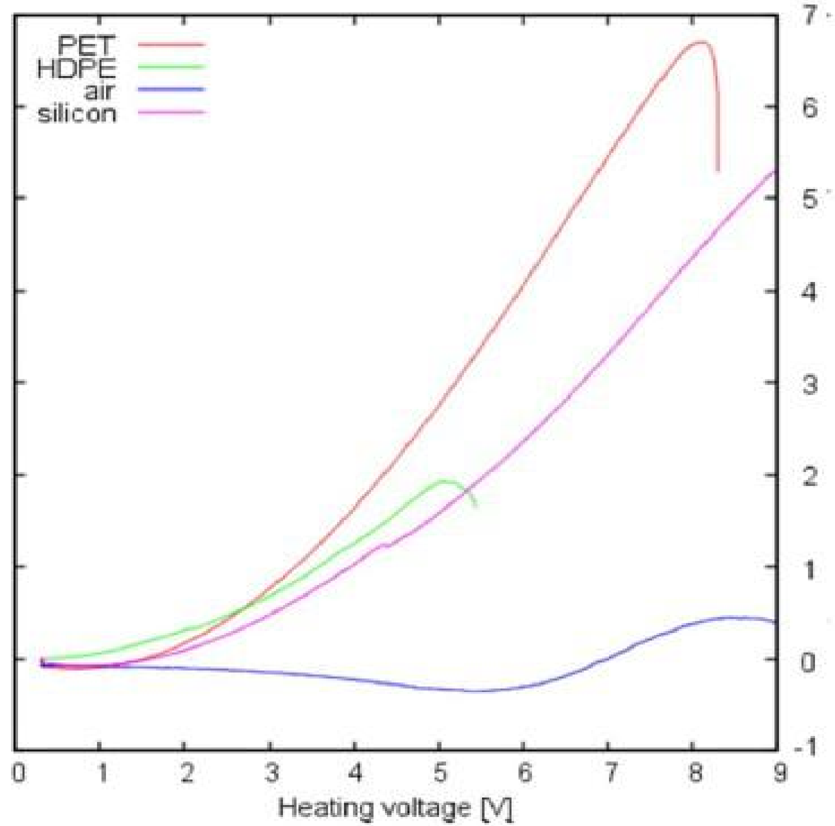
Thermal conductivity wrap up

SThM thermal conductivity measurements guidelines:

- calibrate your setup on known bulk samples
- test your bridge time stability
- do whole experiment at once, without stopping
- do not believe in data obtained on rough samples

To measure thermomechanical properties, we ramp the temperature of the probe, while staying in contact to the surface. When probe reaches glass transition temperature or melting point, it penetrates the sample, which can be monitored in the probe-sample force signal.





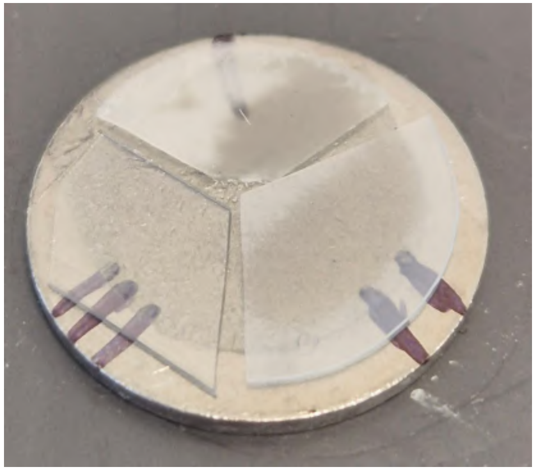
Thermomechanical traceability

Metrological traceability

Differential Scanning Calorimetry: monitoring heat capacity changes with temperature.

Set of polymer samples with known transition temperatures developed in the Quantiheat FP7 project.

Bruker nano-thermal analysis test sample:
PCL, HDPE, PET, 55-235 °C.

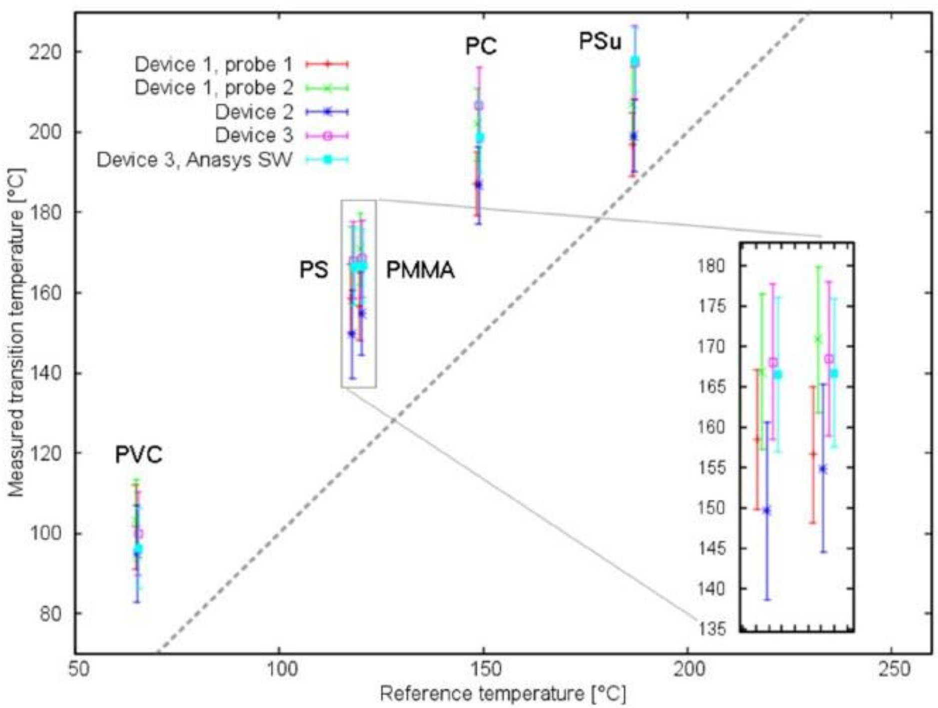


Thermomechanical measurements

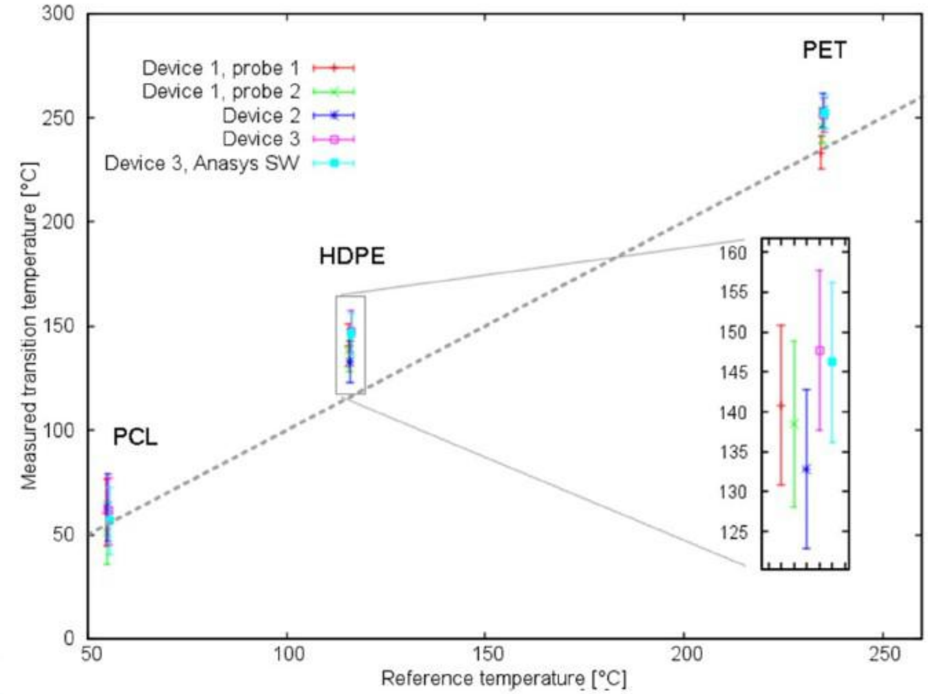
An interlaboratory comparison was run by ČMI, NPL and INSA (CNRS), using different probes, electronics and microscopes.

The results match within the measurement uncertainty (~10 K), however are shifted systematically when compared to DSC data. This can be related to different probing volume when comparing bulk and local measurements.

Quantiheat "unknown" samples



Anasys test samples



Thermomechanical wrap up

Thermomechanical SThM measurements guidelines:

- get some reference samples
- calibrate your probe
- test the probe response in air and on solid sample (e.g. silicon)
- use same ramp rate when doing the calibration and measurement
- avoid sinking into the samples and probe contamination

Conclusions

General recommendations to keep your measurement quantitative:

- don't believe every promise

- get some known samples – ideally traceable ones

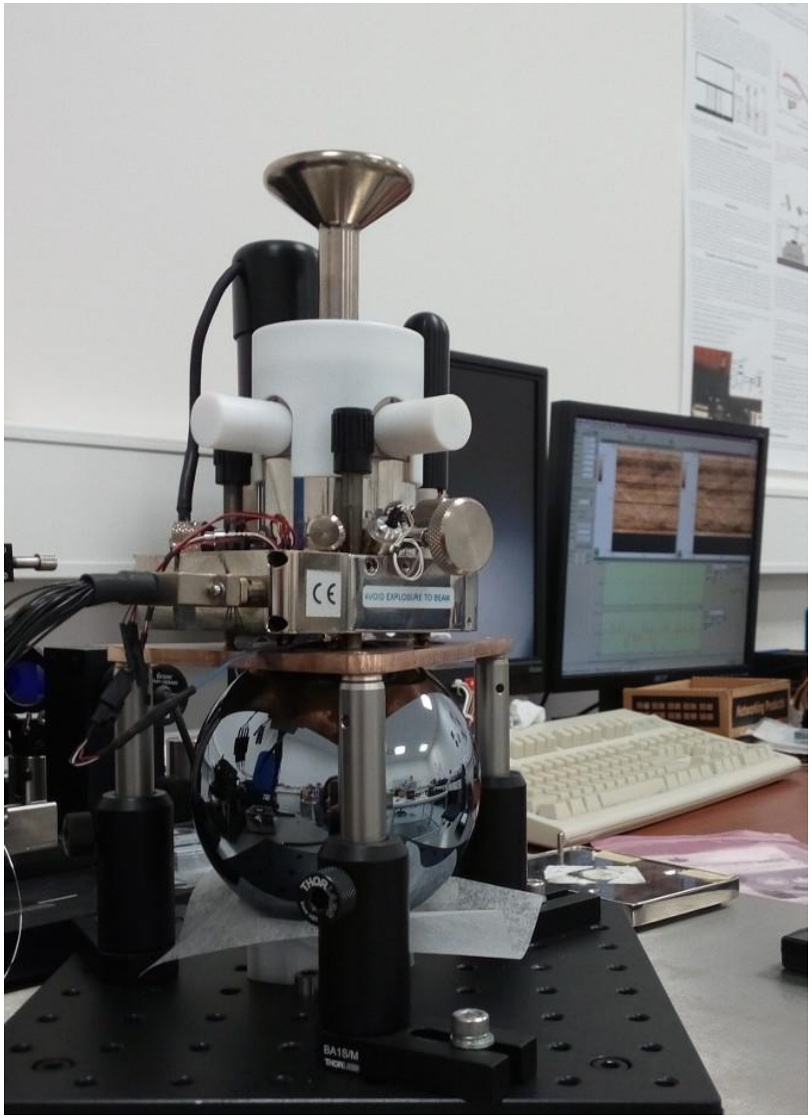
- know your uncertainty sources

- process your data only minimally

- try to use different probes, voltages, amplitudes, etc. to verify that everything works correctly

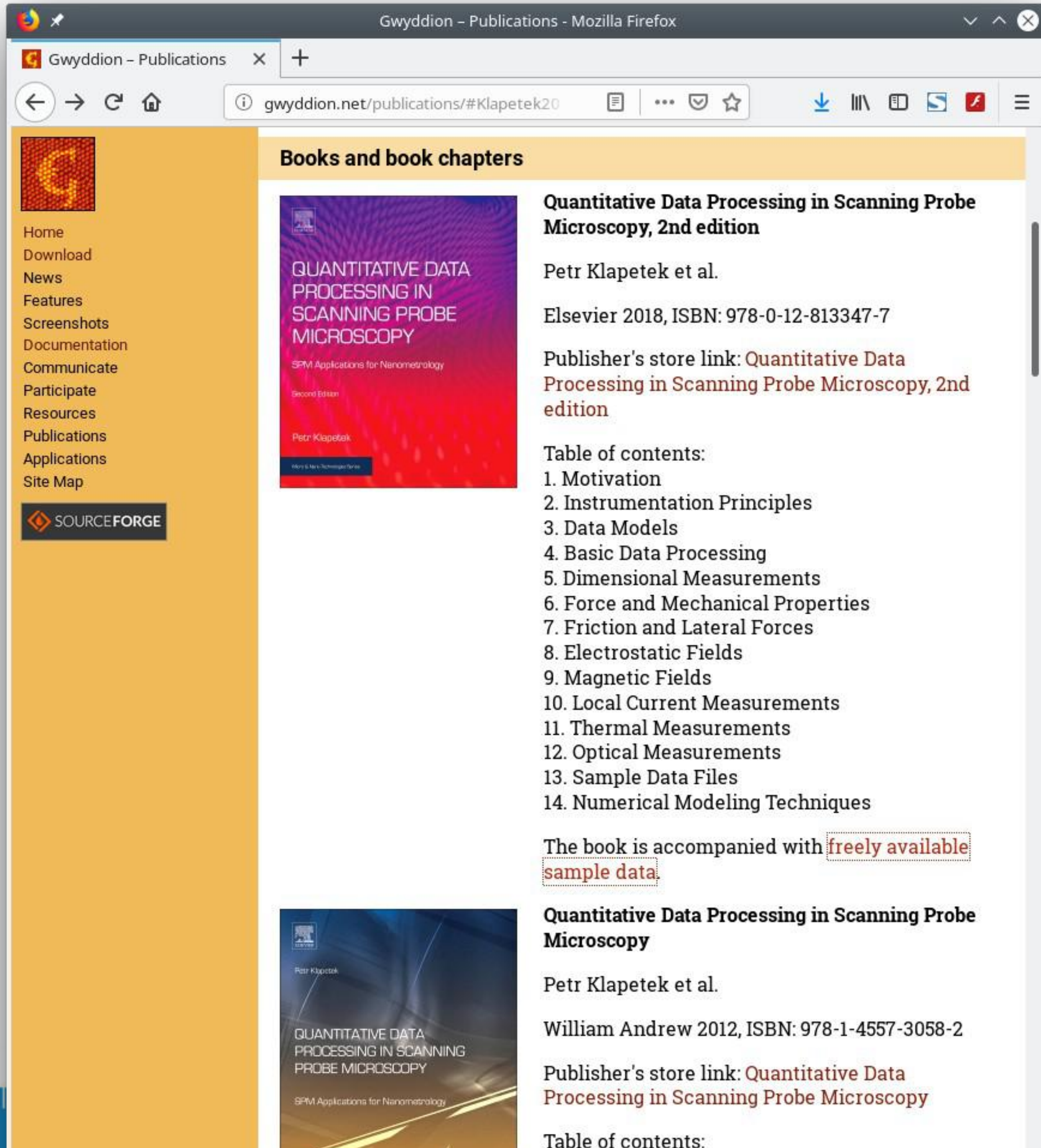
For dimensional measurements 1% uncertainty should be reachable.

For other properties, be happy for 10%.



Gwyddion basics: resources

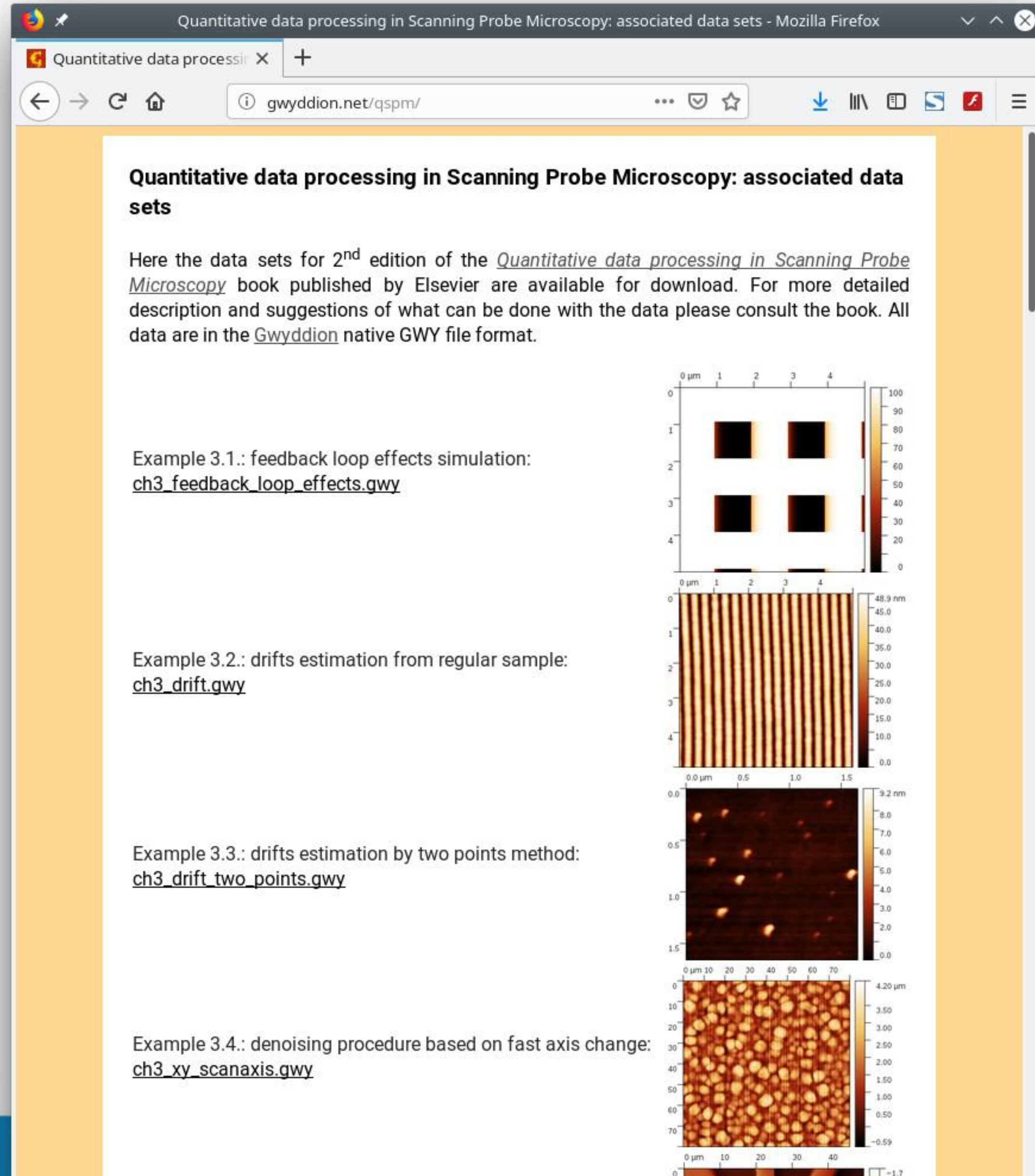
More details about the data processing for different SPM measurement methods: book published by Elsevier

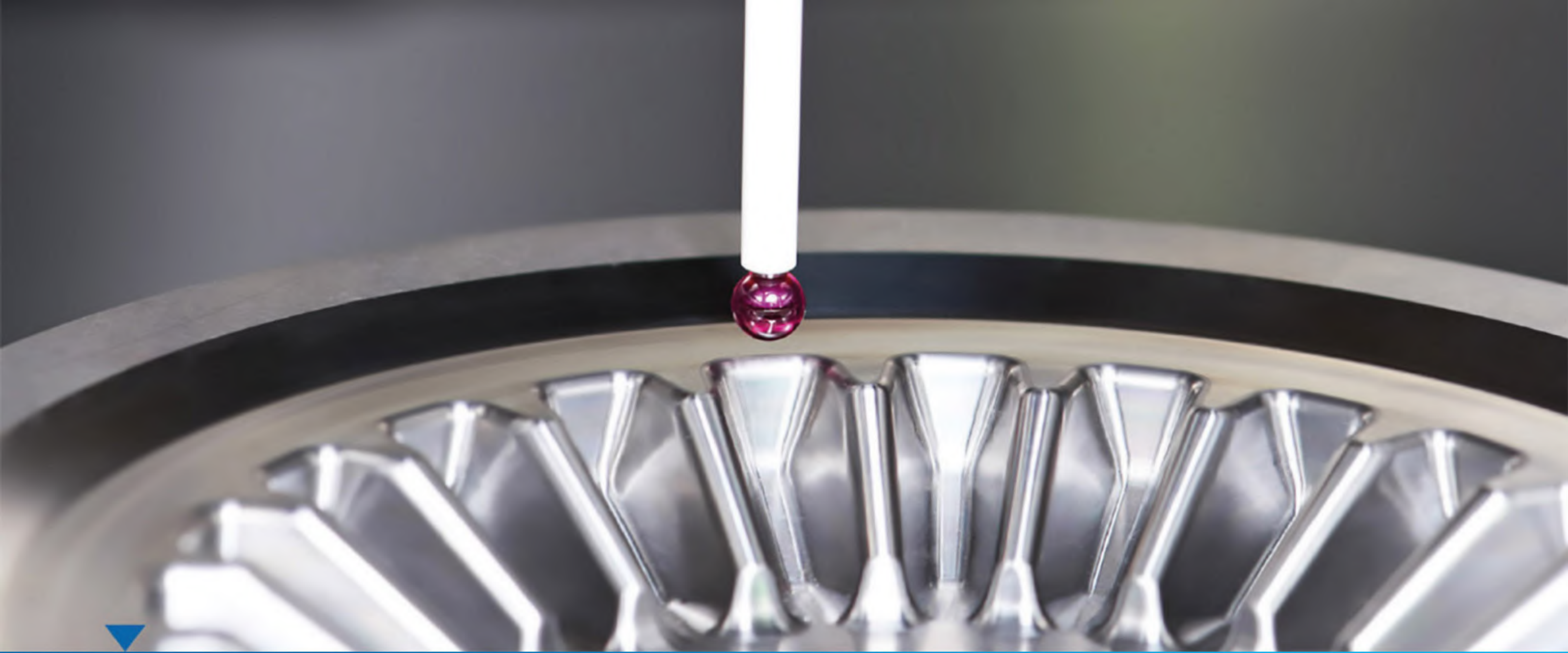


Gwyddion basics: resources

Large set of sample data related to the book:

http://gwyddion.net/qspm/





Thank you for your attention

CZECH METROLOGY INSTITUTE

Okružní 31, 638 00 Brno, Czech Republic, + 420 545 555 337, www.cmi.cz