Combined Atomic, Microwave and Electron Microscope: A tool for Hybrid Characterization of Nanomaterials

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RF measurements at the nanoscale : why?

- Electrical properties investigation at the microwave of:
  - Carbon NanoTubes, Graphene, Self-Assembled Monolayers,
  - Liquids, Biological samples
  - Etc…

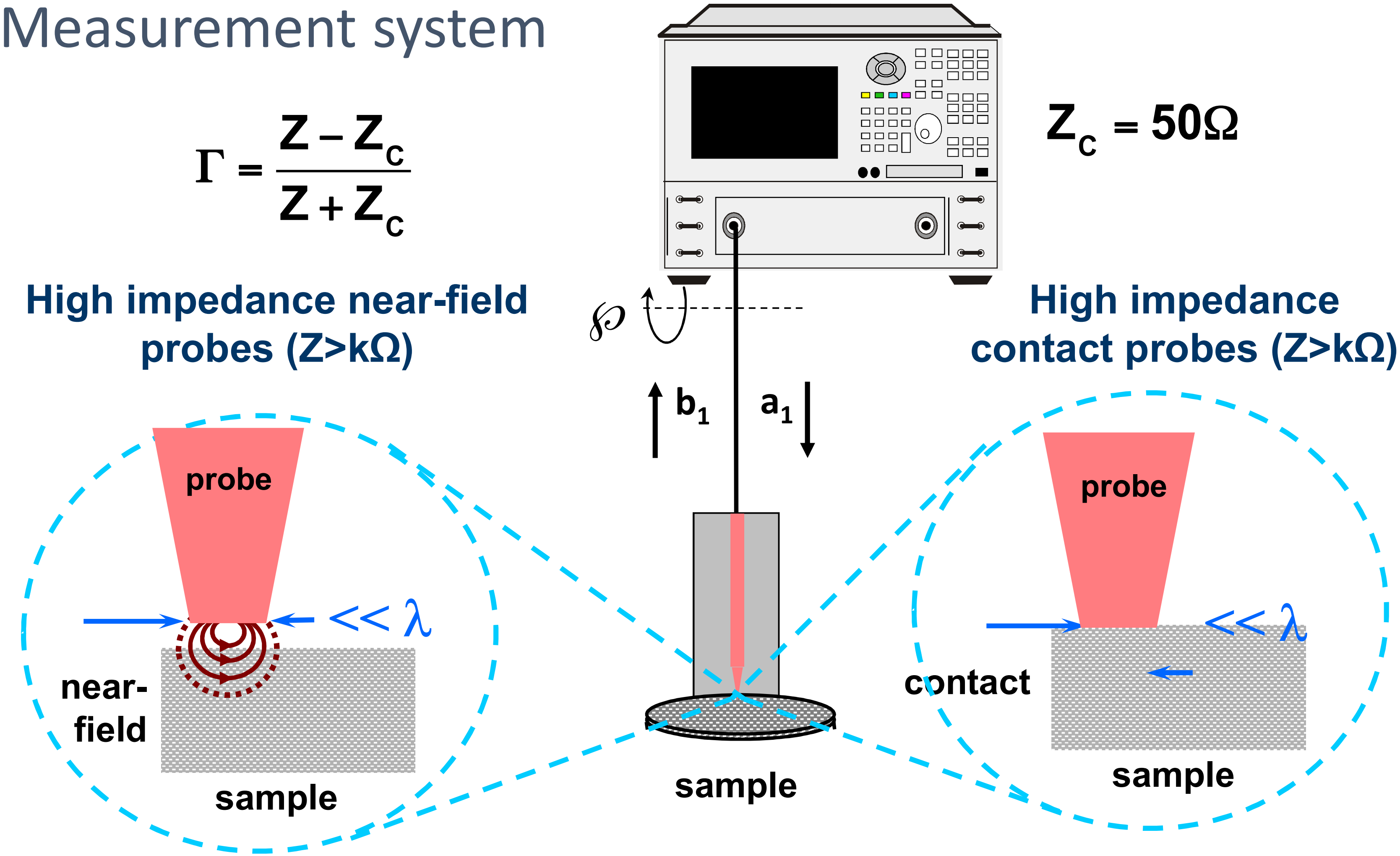
- 3 main difficulties:
  - Nanoobjects present very high impedances at microwave frequency and conventional vector network analysers are optimized for 50 Ω.
  - Contacting nanodevices and supplying microwave signal to nanodevices and nanoobjects is a problem => AFM is a possible approach.
  - Quantitative measurements require calibration samples. CO, CC, 50 Ω are far from high impedances. There is no dedicated calibration for high impedances

Generic principle for a solution

- Measurement system

$$\Gamma = \frac{Z - Z_C}{Z + Z_C}$$

$Z_C = 50\Omega$



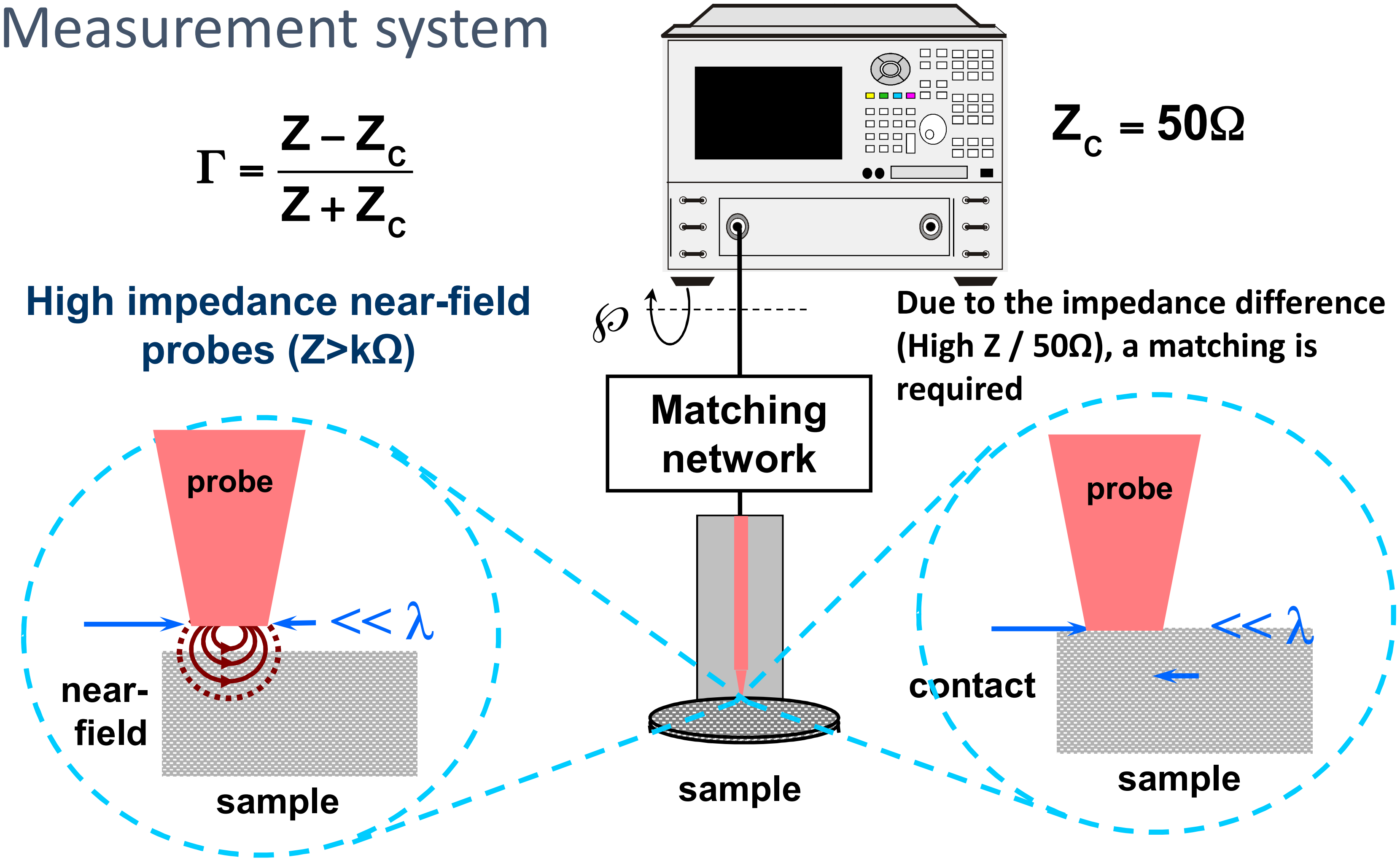
High impedance near-field probes (Z>kΩ)

High impedance contact probes (Z>kΩ)

Generic principle for a solution

- Measurement system

$$\Gamma = \frac{Z - Z_C}{Z + Z_C}$$



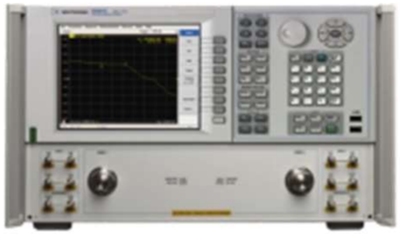
$Z_C = 50\Omega$

High impedance near-field probes (Z>kΩ)

Due to the impedance difference (High Z / 50Ω), a matching is required

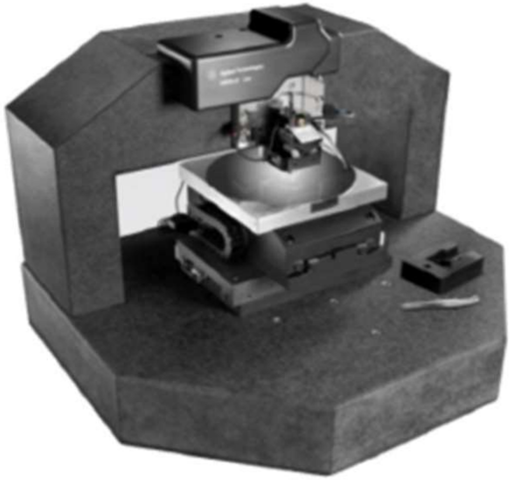
Solution Keysight^{TM} : Scanning Microwave Microscope (2008 -)

Atomic force microscope (AFM) interfaced with a vector network analyzer



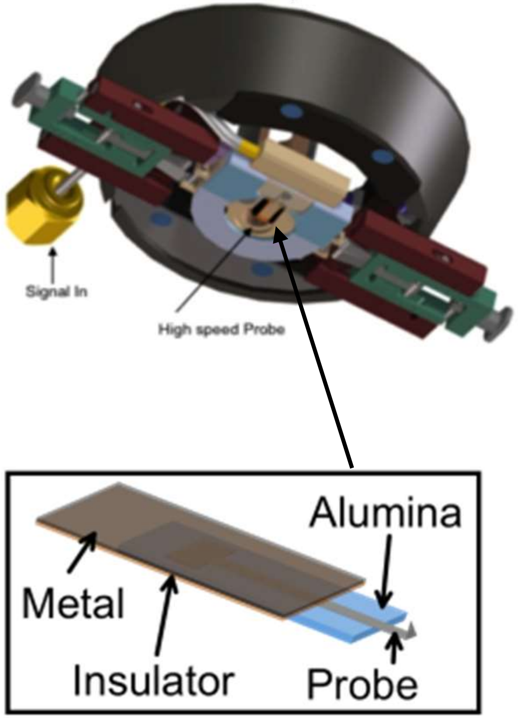
VNA (ex: E8363)

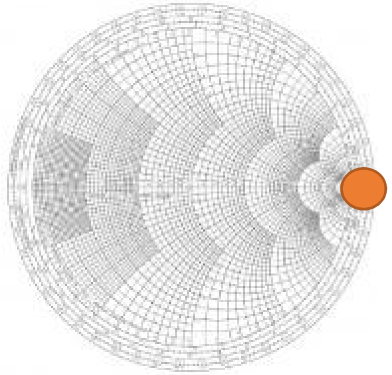
+



AFM 5600LS

+





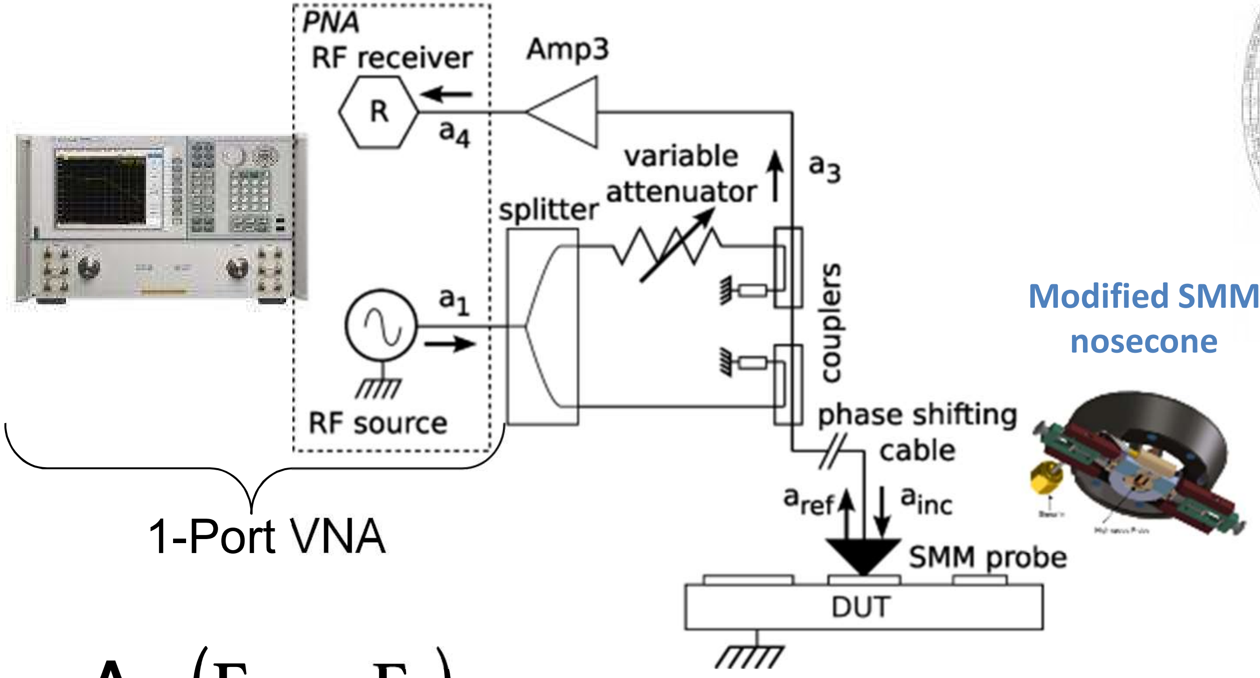
Point on a Smith chart

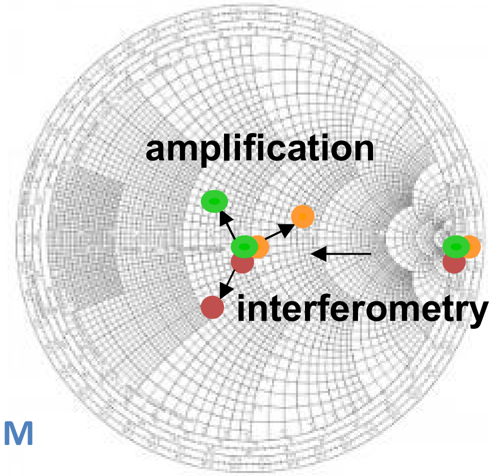
$|\Gamma|$#0.9999987…

Difference between 2 high impedances

Impedance matching: Interferometric set-up

- “Mach Zehnder” interferometer
- Concept *Move and Zoom*





« Move and zoom »

|*Γ*1|#0.001…
|*Γ*2|#0.0012…

VNA precision

$$T_{ITF} = A_{ITF}\left(\Gamma_{DUT} - \Gamma_0\right)$$

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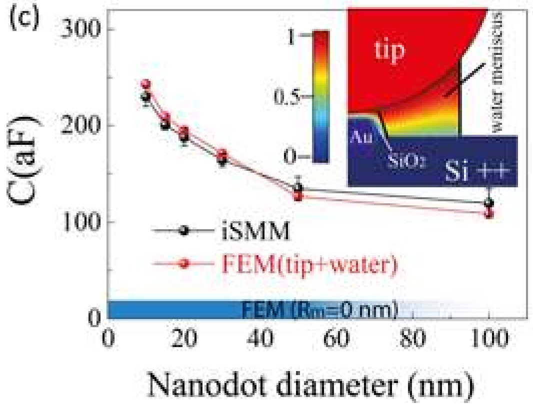
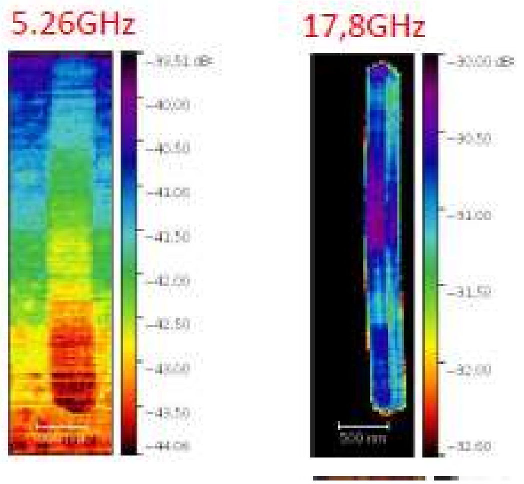
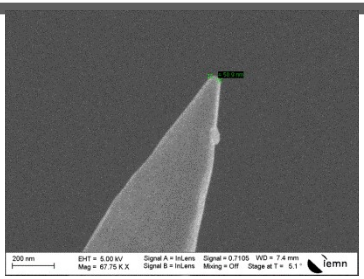
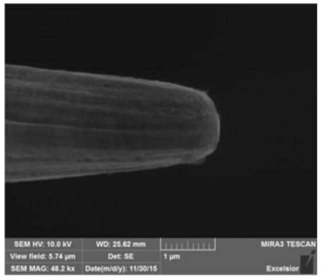
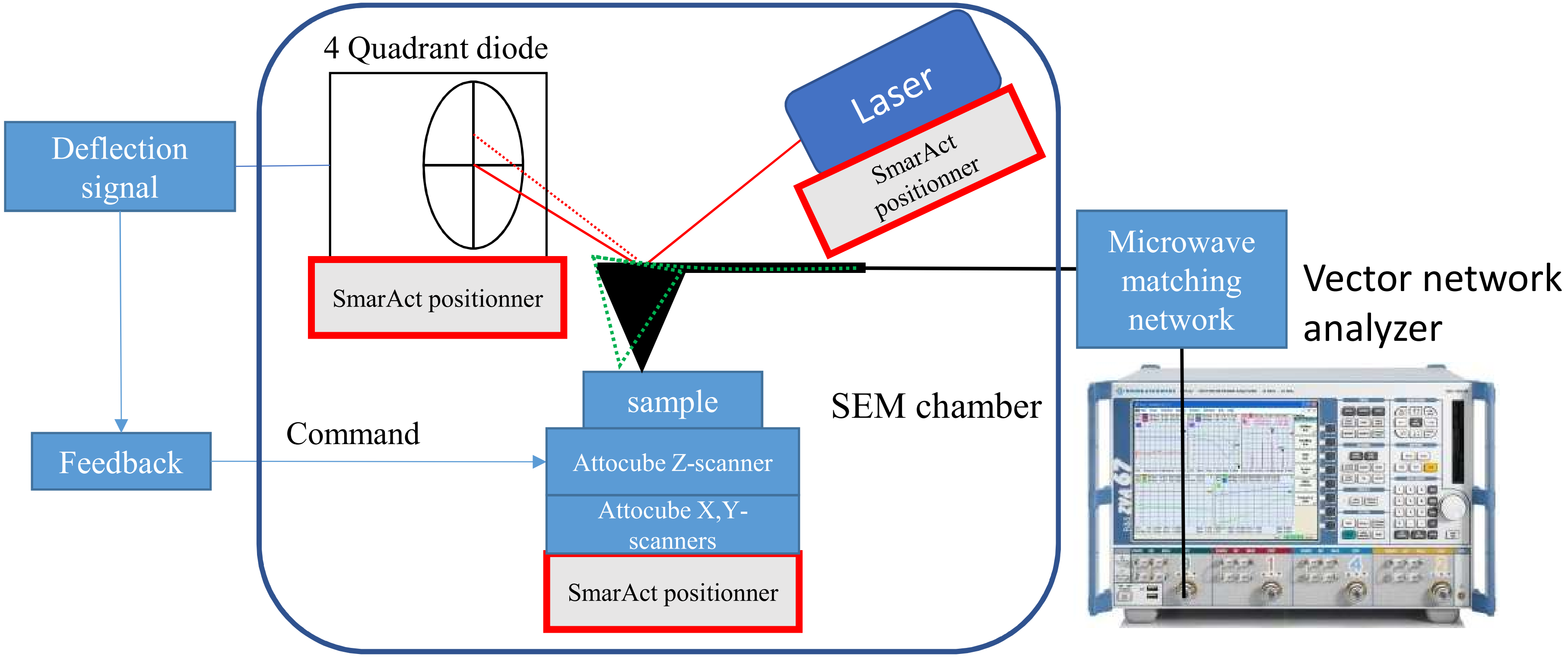
2 close impedances can be distinguished on the Smith chart

Issues to address nm resolution

Water meniscus	Resolution vs Wavelength	Probe life/Modeling
Sub-100 nm diam. capacitor  	GaAs Nanowire (400 nm)  	 
Increase the parasitic capacitance	Lateral resolution is frequency dependent	From 50 nm to 500 nm after a few scans

Nanotechnology 25 , 40 (2014) 405703

Scanning Microwave Microscopy in Scanning Electron Microscope



4 Quadrant diode

Laser

SmarAct positionner

Deflection signal

SmarAct positionner

Microwave matching network

Vector network analyzer

sample

SEM chamber

Feedback

Command

Attocube Z-scanner

Attocube X,Y-scanners

SmarAct positionner

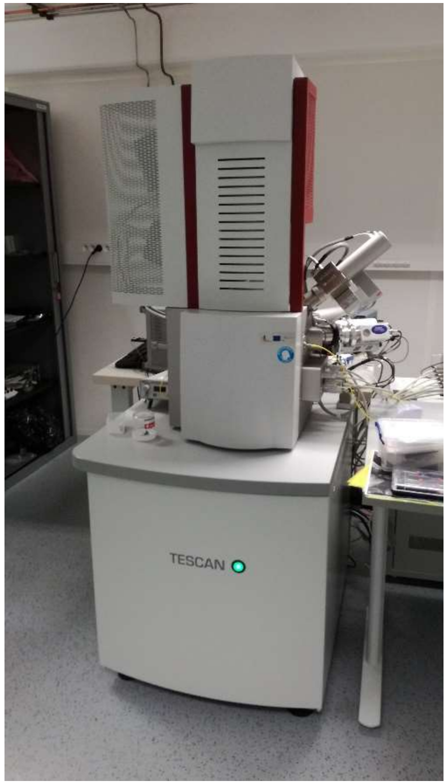
Issues and solutions:

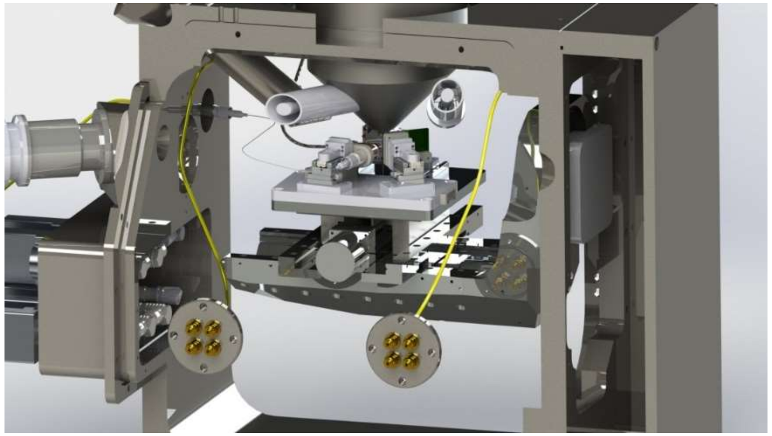
- Water meniscus => vacuum + heating
- Wavelength => probe and waveguide design
- Probe life => Scanning Electron Microscopy images

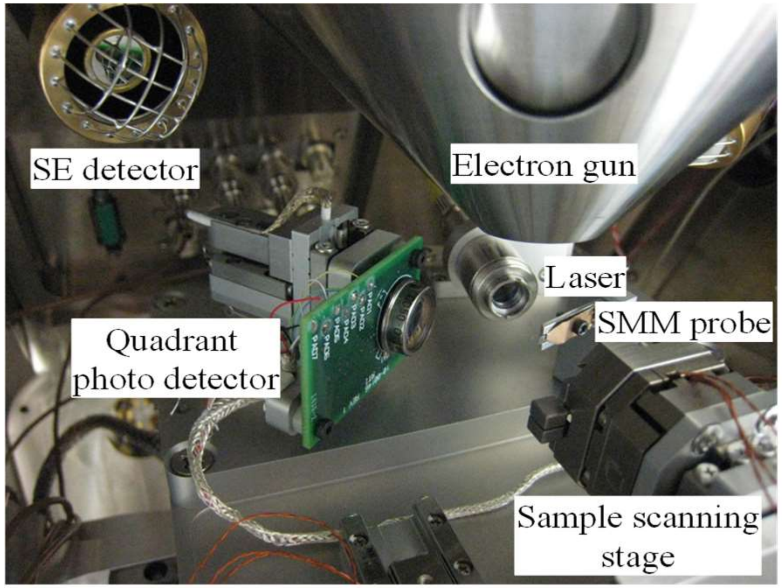
The validation of new set-up requires several experiments:

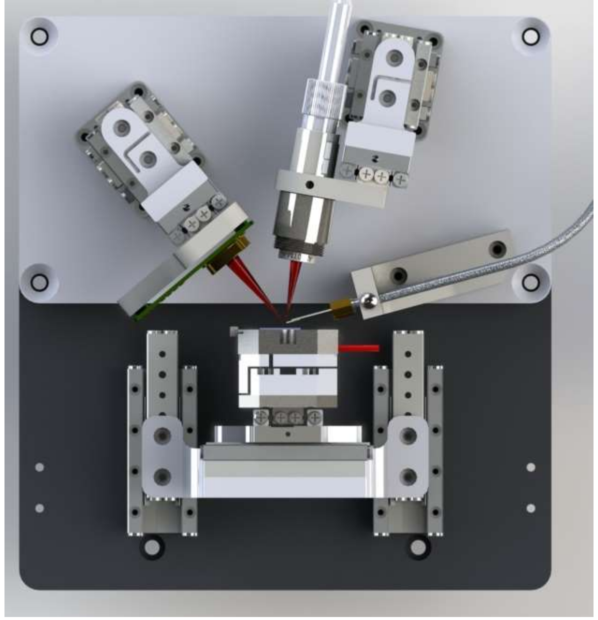
- The impact of the drift of SmarAct positioners
- Impact of thermomechanical noise – is the precision enough?
- Quality of feedback adjustment

Scanning Microwave Microscopy in Scanning Electron Microscope



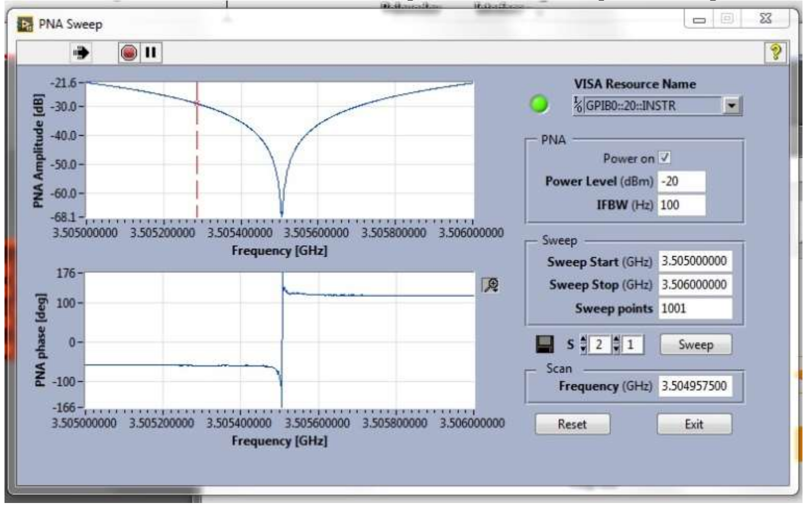






LabVIEW for control and data acquisition

Interface for the sweep and frequency choice



VISA Resource Name GPIB0::20::INSTR

PNA

Power on ☑

Power Level (dBm) -20

IFBW (Hz) 100

Sweep

Sweep Start (GHz) 3.505000000

Sweep Stop (GHz) 3.506000000

Sweep points 1001

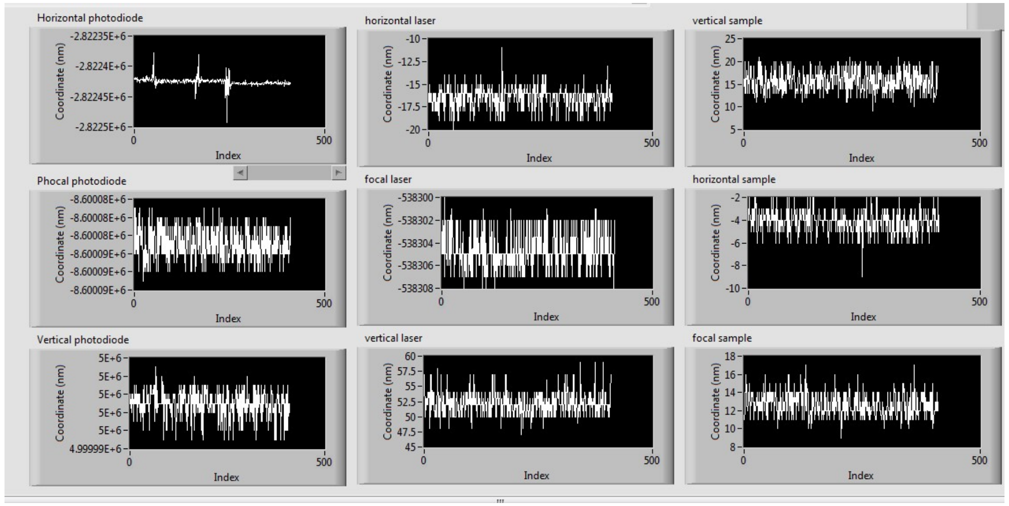
S 2 1 Sweep

Scan

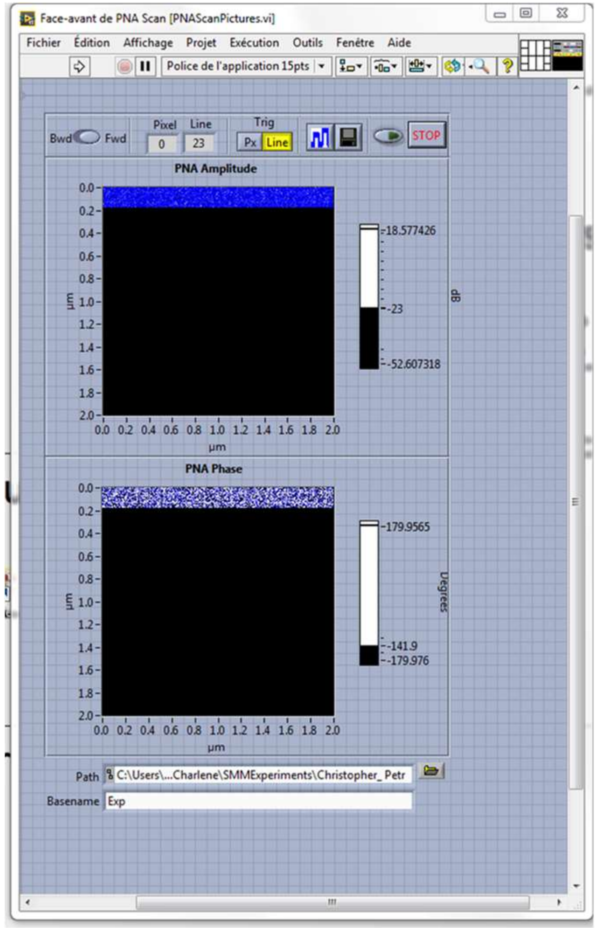
Frequency (GHz) 3.504957500

Reset Exit

SmarAct measurements



Interface for the acquisition of RF scans



Pixel 0

Line 23

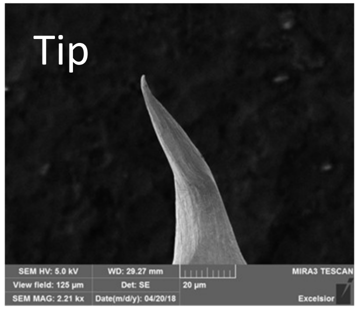
Path C:\Users\...Charlene\SMMExperiments\Christopher_Petr

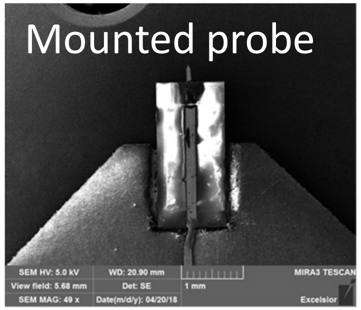
Basename Exp

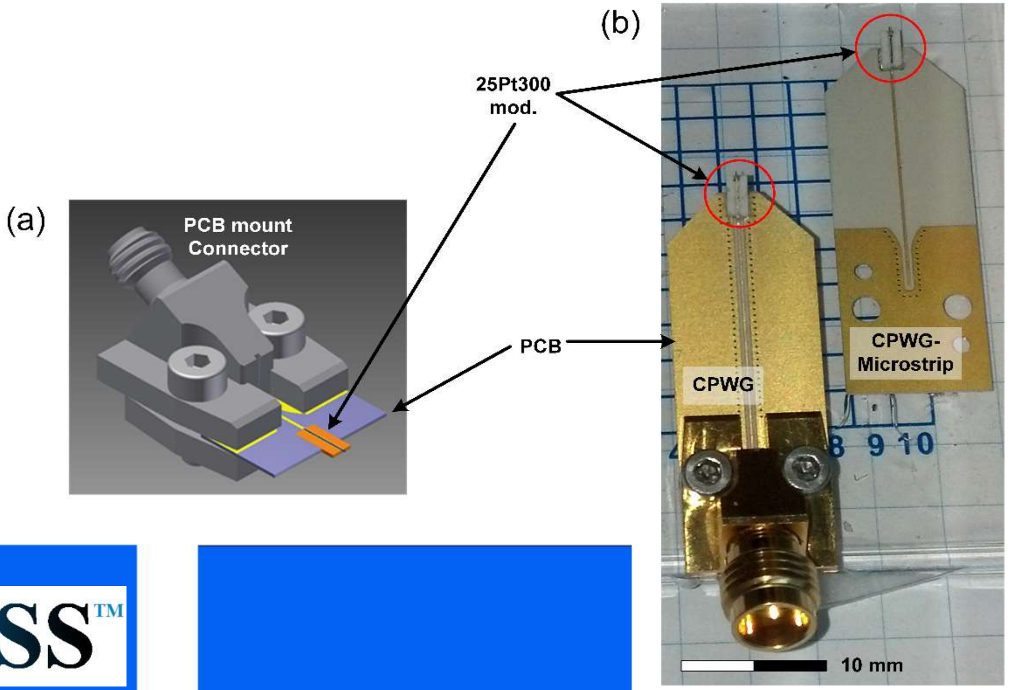
Proposed 1-110 GHz probe (resolution vs wavelength issue)

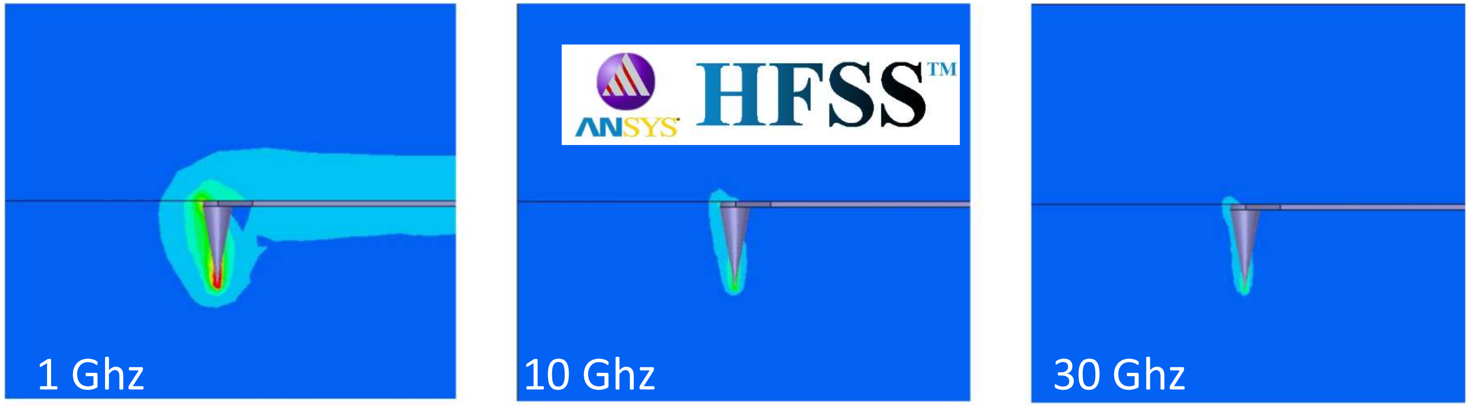
SMM Keysight is limited to 24 GHz

Rocky Mountain Nanotechnology™



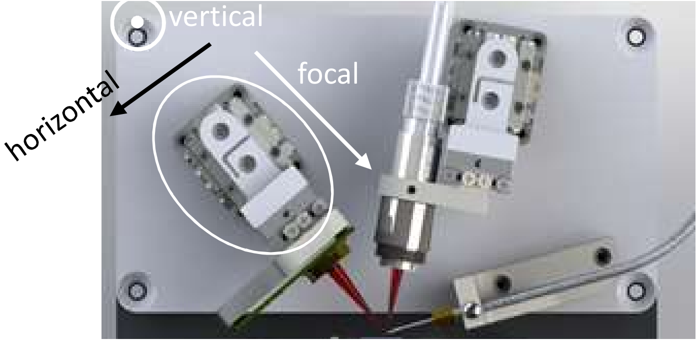




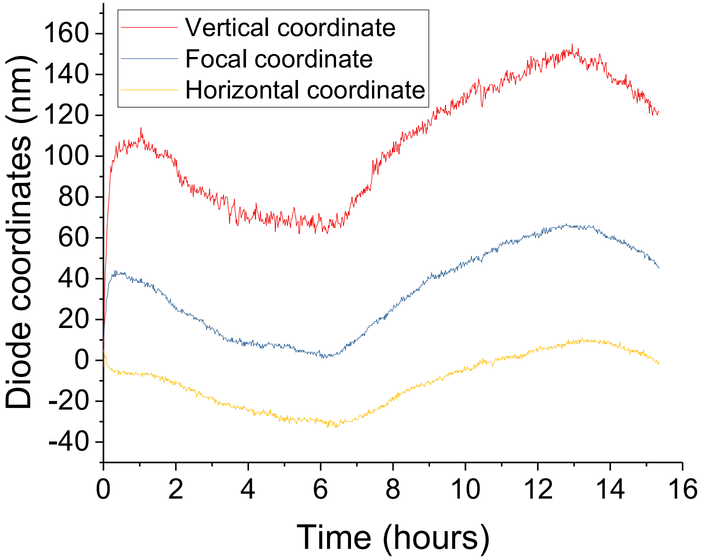


SmarAct positioners drift measurement

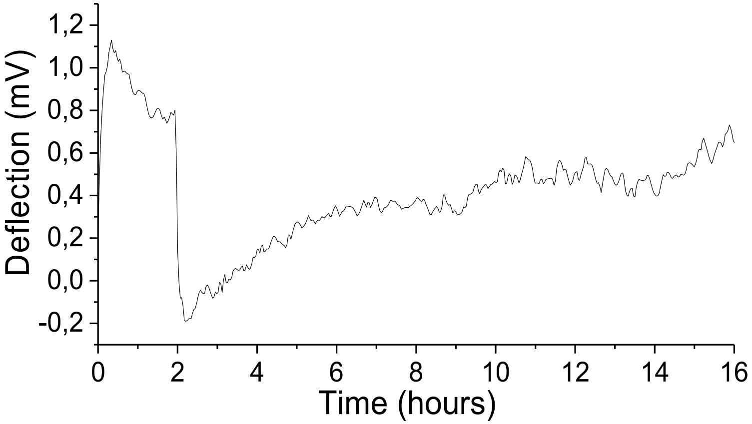
The drift of SmarAct positioners is present during the experiment. That could be determining for the SMM measurements



Measurement of SmarAct positioner by an optical sensor



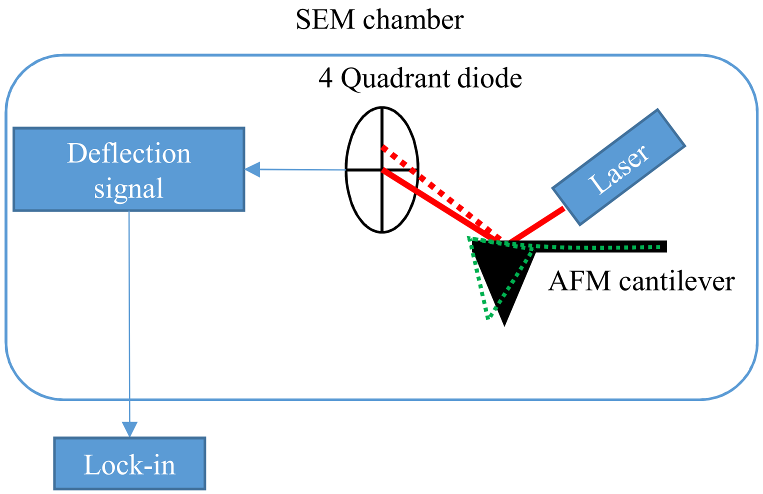
Simultaneous photodiode measurements are performed

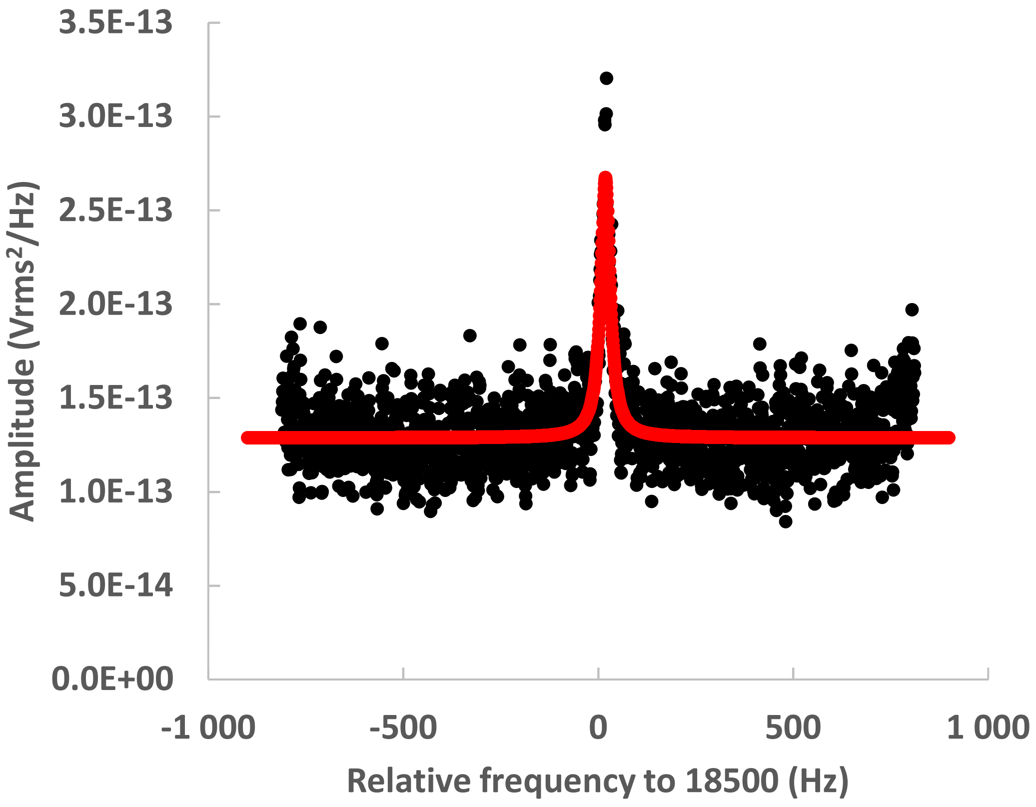


- 4Q diode position stable within a 100 nm range for 16 hours => much lower than the spot size (~ a few µm) and position on the cantilever.
- Deflection signal stability of 1 mV <=> 10 nm fluctuation.

Thermomechanical noise. Precision

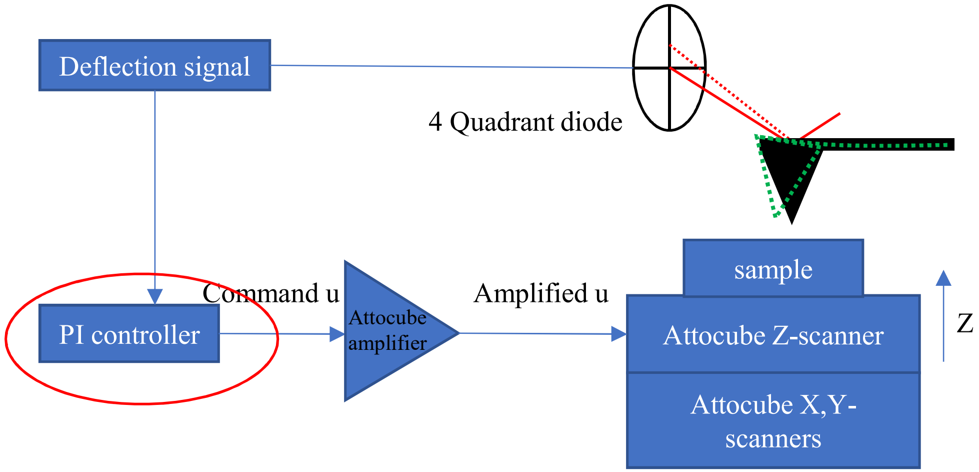
Simplified AFM/SEM noise detection.
No scanning. No contact between the tip and the sample





Resonator quality factor is f/Δf = 640
From the integral of spectral density the amplitude is 15 pm

Feedback adjustment



Deflection error

$$e(t) = D(t) - D_{setpoint}$$

PI controller output:

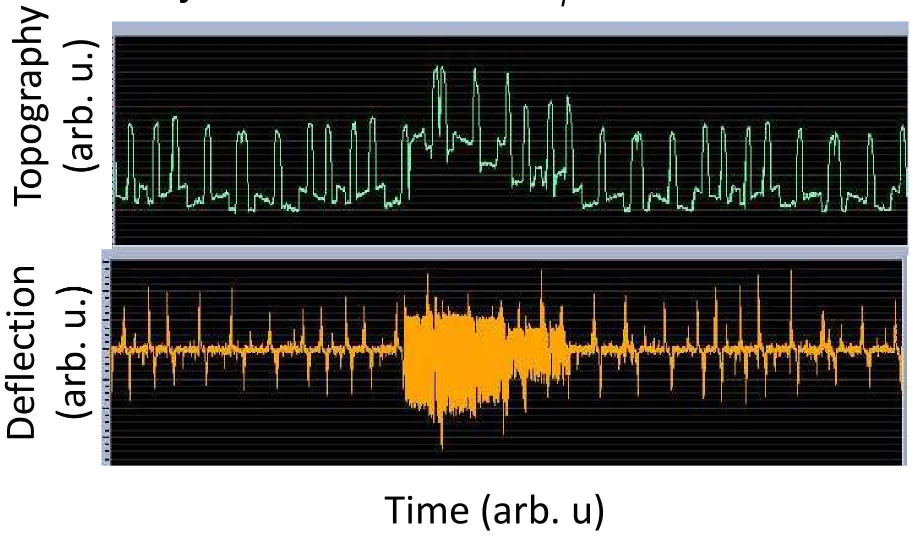
$$u(t) = Pe(t) + \frac{P}{T_i}\int_0^t e(\tau)d\tau$$

From the regulation

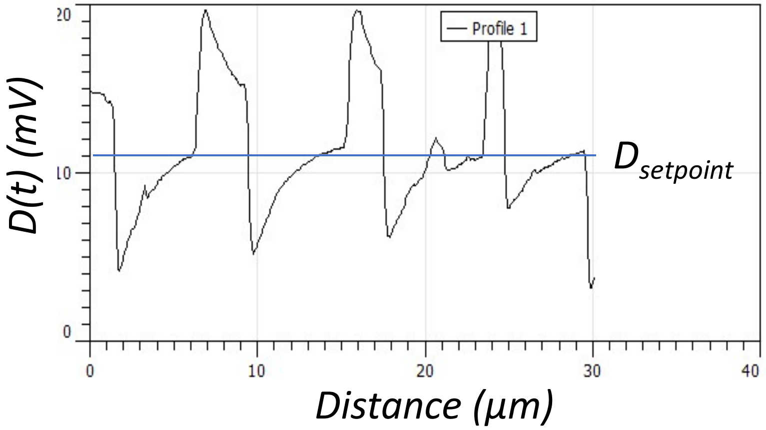
$T_i \approx 100$ µs is set to piezo scanner cut-off time.

$P \approx 10$ nm/mV from the approach-retract curve

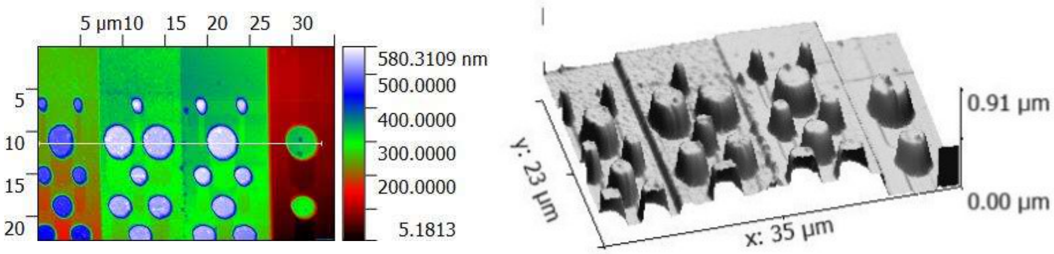
Adjustment of P and T_i while a scan:



Example of scan while the T_i is big:



AFM Keysight and IEMN comparison



2D and 3D topography representations.

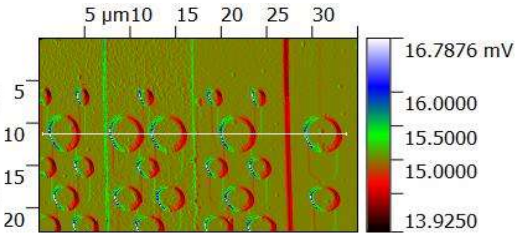
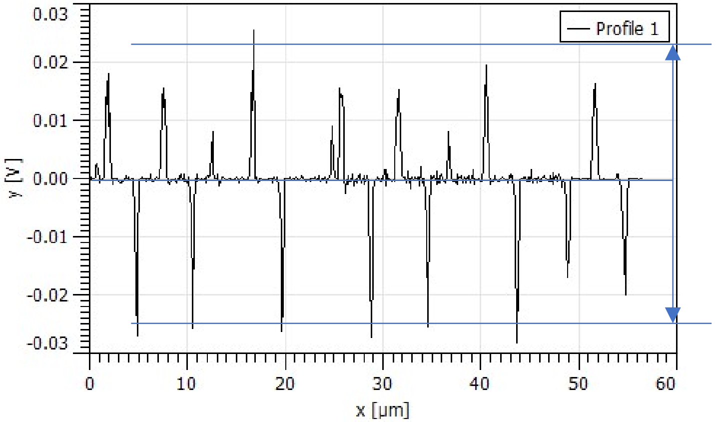


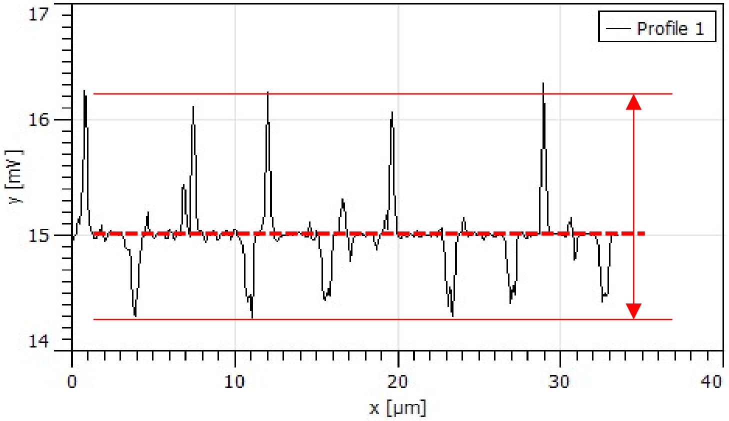
Fig. Deflection signal image. The Deflection is measured simultaneously with topography. There is change in deflection while passing the edges of µ-plots and the SiO_2 steps.

AFM Keysight deflection error



The profile from the deflection image. The relative change of deflection is ±4%.

AFM IEMN deflection error

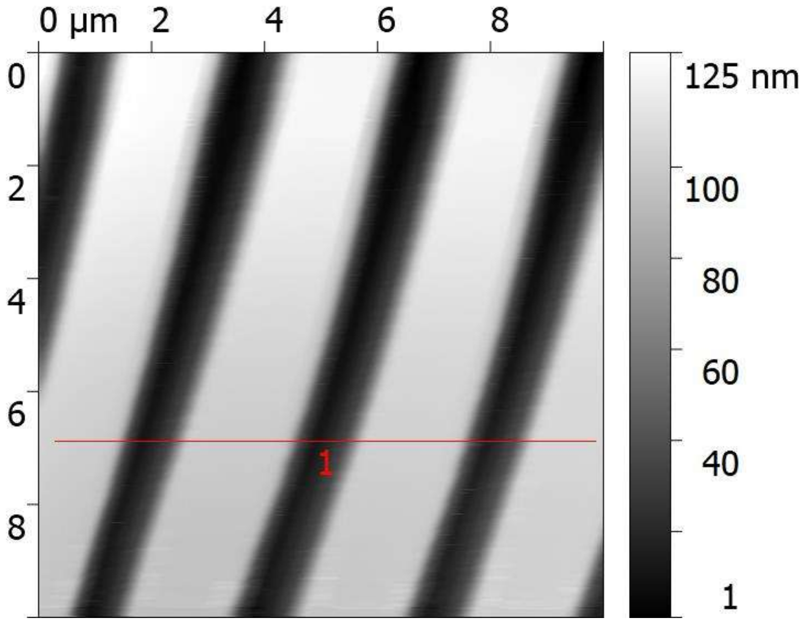


The profile from the deflection image. The relative change of deflection is ± 6%.

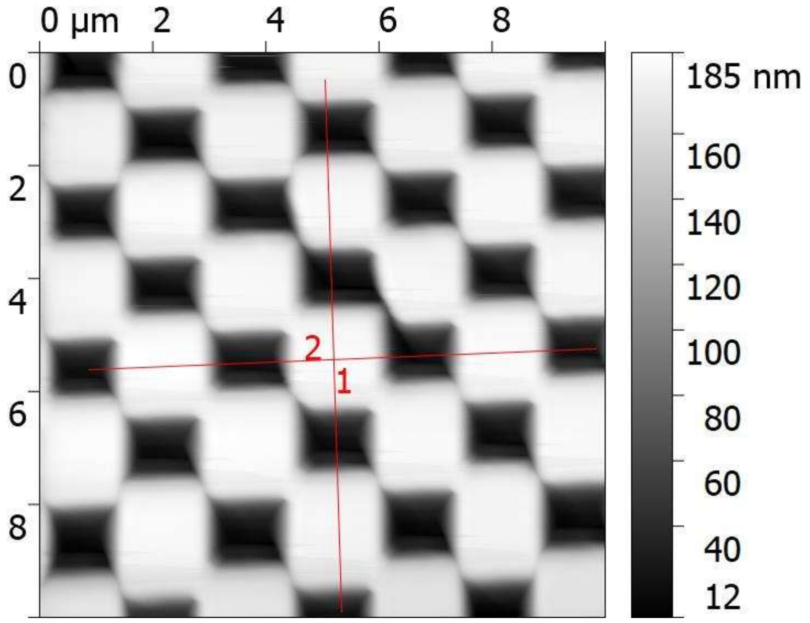
The deflection errors are comparable. The PI controller mode is functional

AFM calibration

After addressing AFM technical issues the piezo scanner calibration is performed:



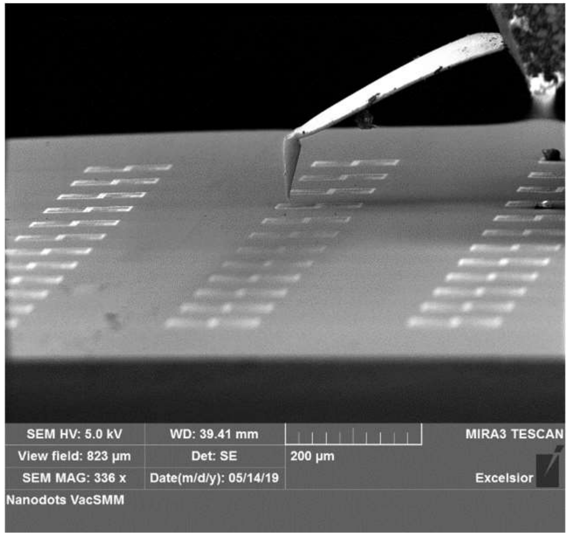
Calibration sample Z

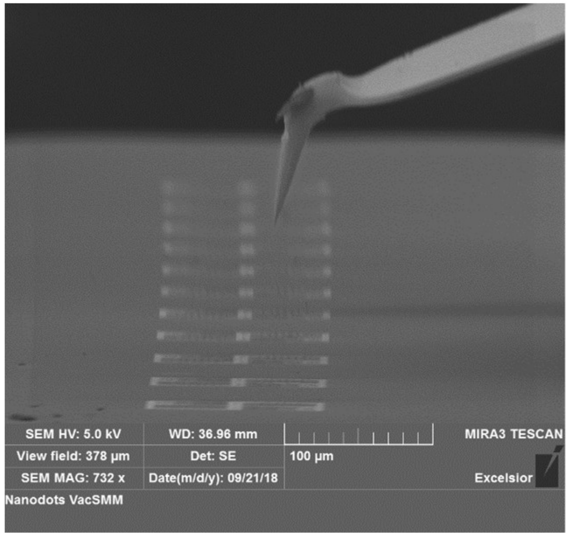


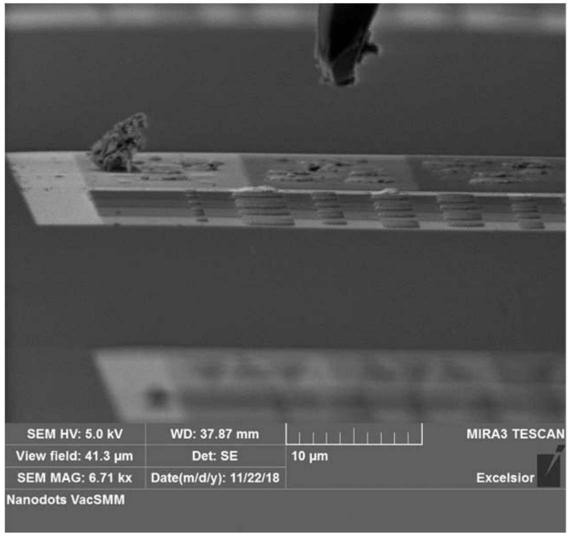
Calibration sample XY

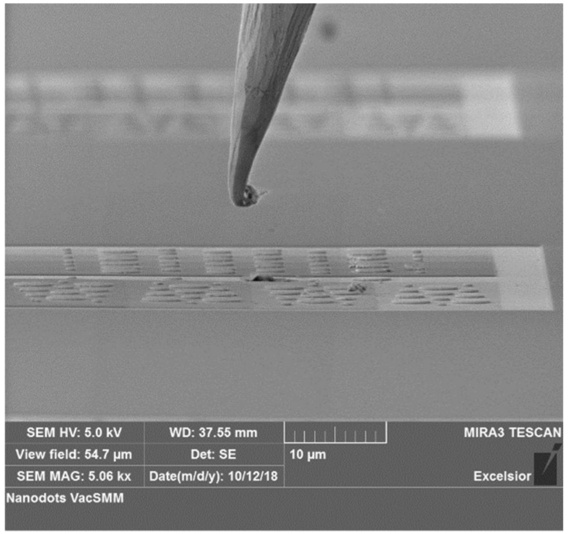
Conclusion: AFM is functional

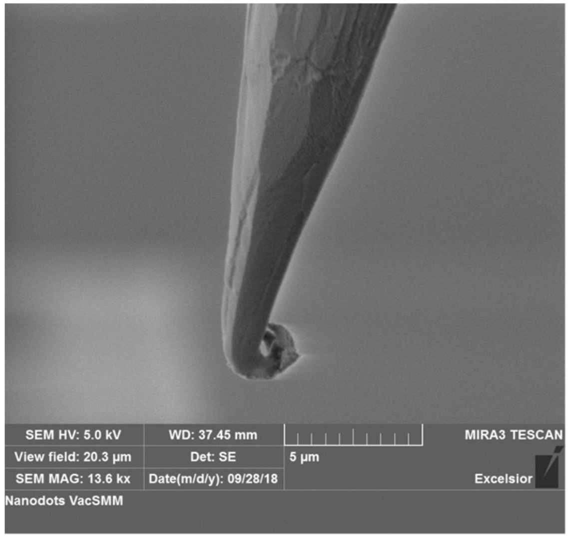
The advantage of using SEM

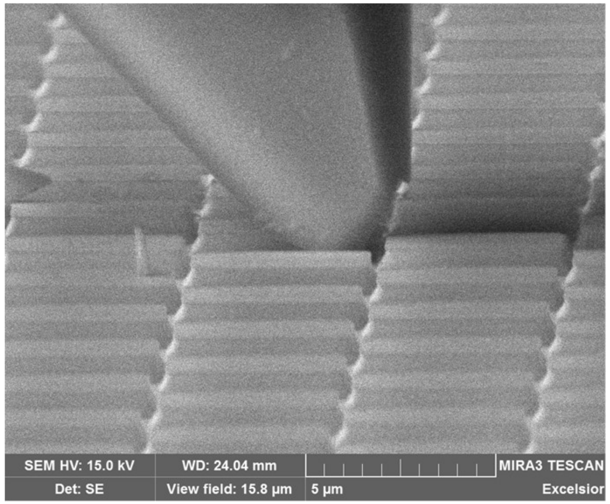




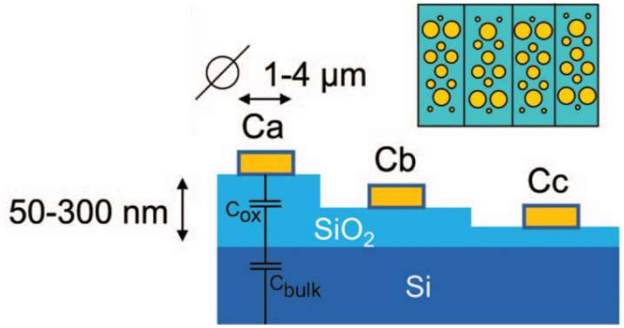






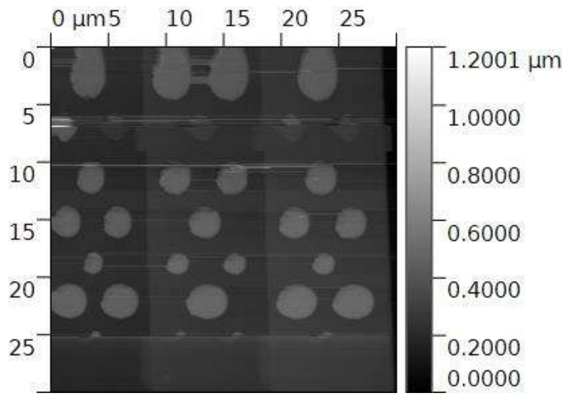


SMM data for calibration

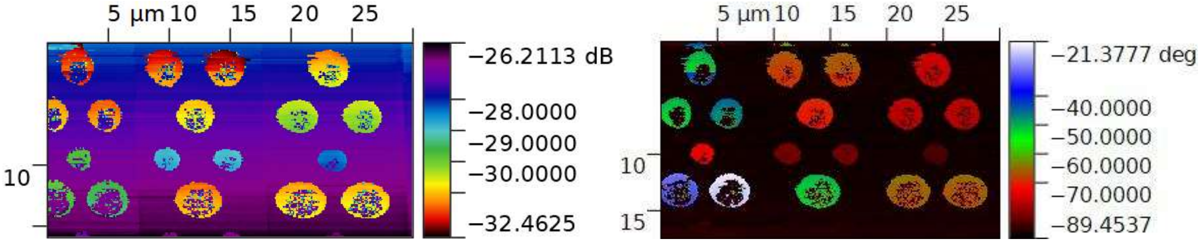


Silicon (1-0-0) P doped type
thickness: 325-375µm
Resistivity: 1-3 Ohm.cm

Frequency = 7.59 GHz
Microwave signal power = -5 dBm
Demodulation IF Bandwidth = 100 Hz
Interferometric set-up + LNA (30 dB gain)



AFM topographic image of capacitors

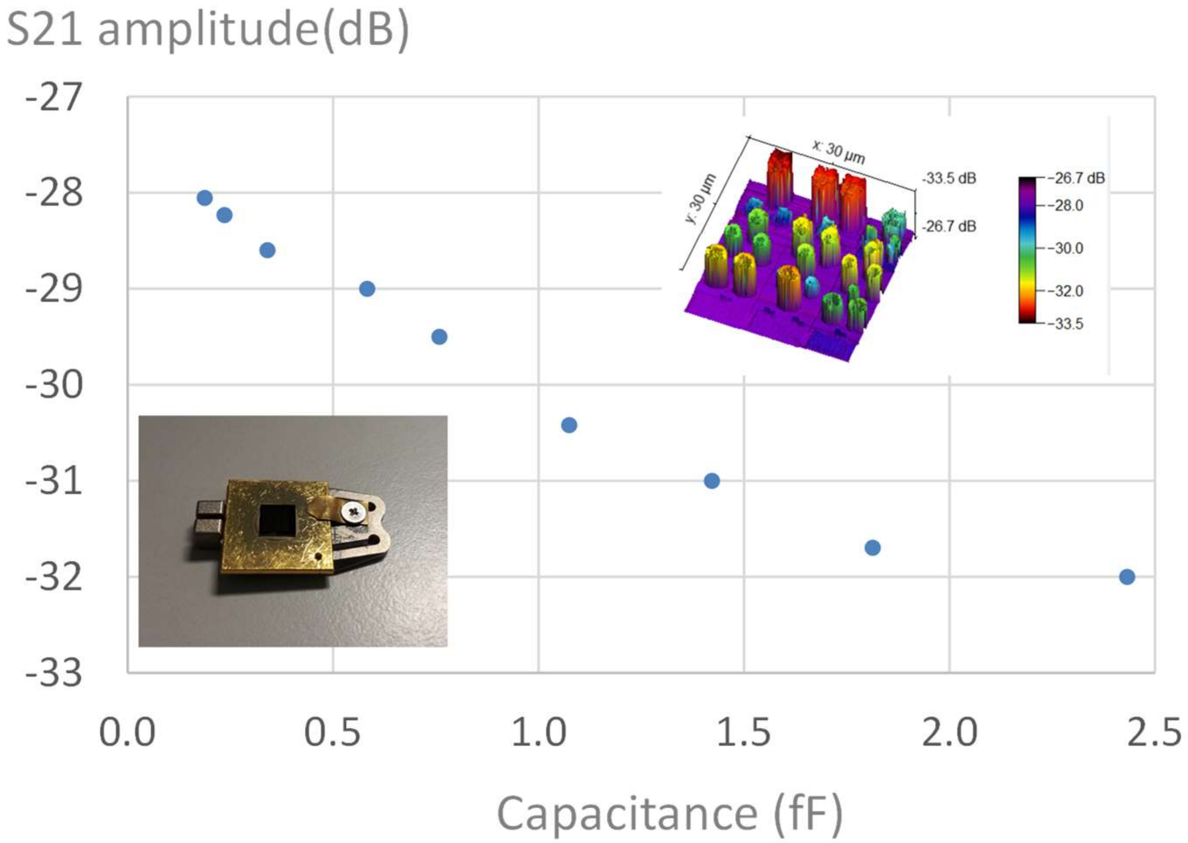


Amplitude and phase of the microwave reflection coefficient (S parameter)

Dependence of the S parameter with the capacitance value => possible calibration of the microwave signal for further impedance measurement.

Calibration data

The measured S parameter values could be used for the calibration



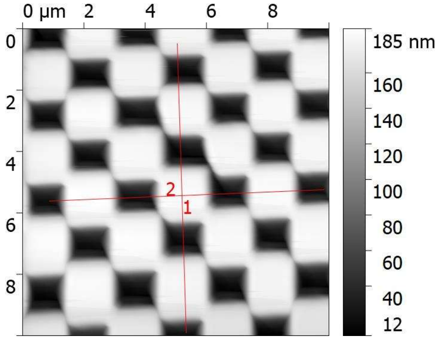
=> calibration is possible

Conclusions

SMM in SEM is implemented

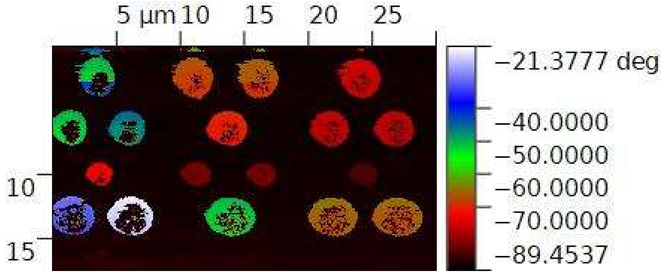
AFM:

- The preliminary tests are performed. The home-made AFM is operational.

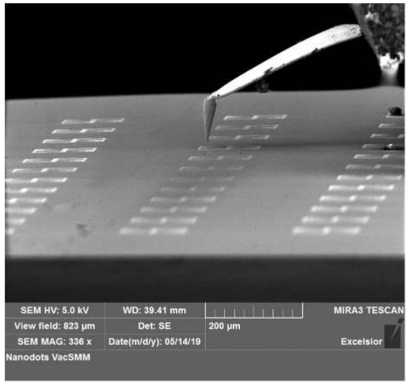


SMM measurements:

- The calibration SMM experiments could be performed in the new SMM/SEM with the frequency of 7,59 GHz



SEM images are possible



Thank you for your attention

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