Artifacts and Data Processing in Scanning Probe Microscopy

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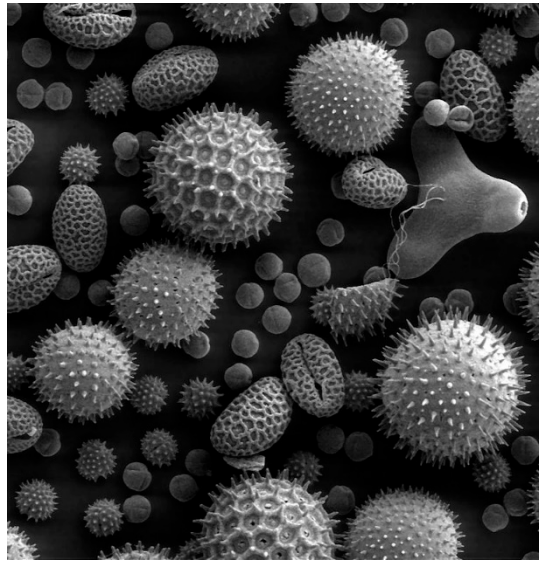
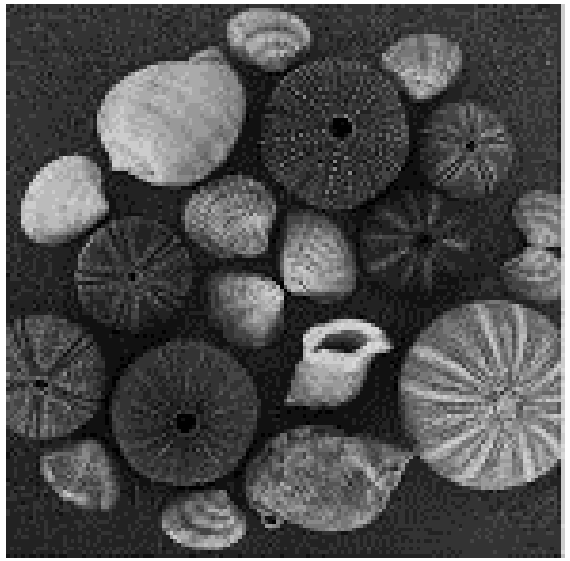
Università di Padova

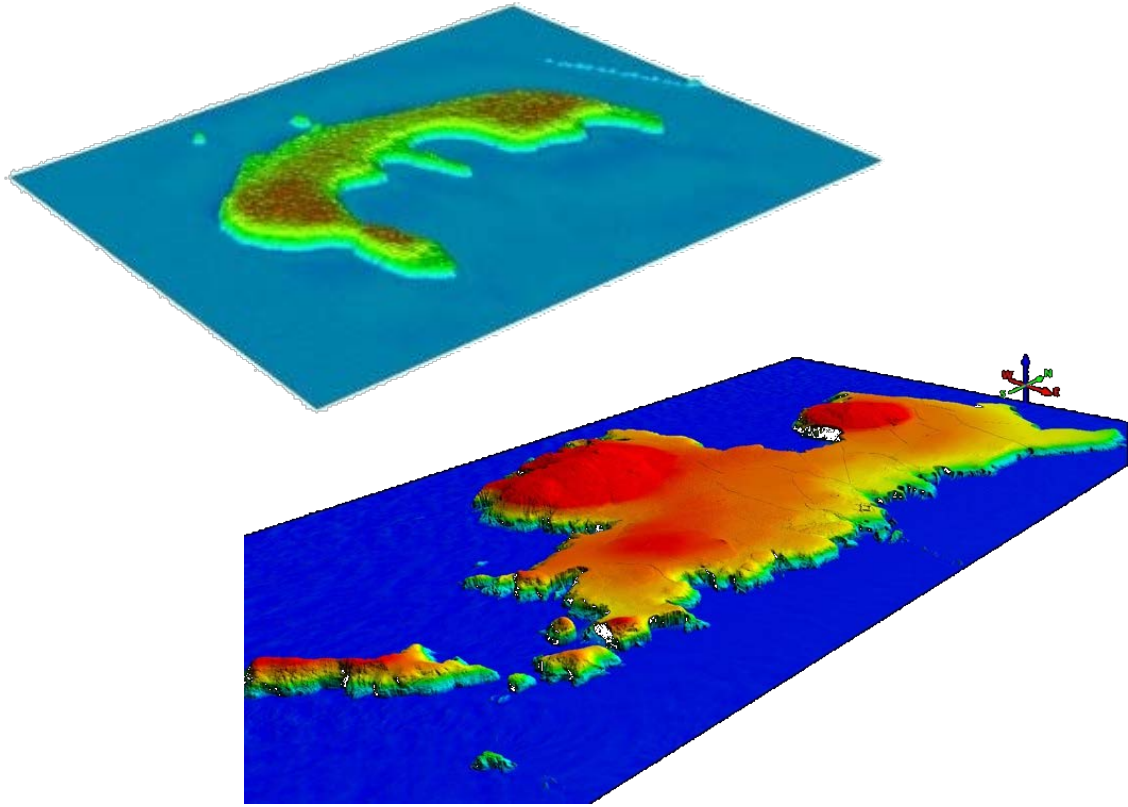
From the nanoscale to the gigascale

Mutiscale geometry is actually typical not only of natural phenomena





Pollen (50 µm)
and shells (50 mm)



Nanoparticles aggregate (10 µm)
and Ramsey Island (3 km)

From the nanoscale to the gigascale

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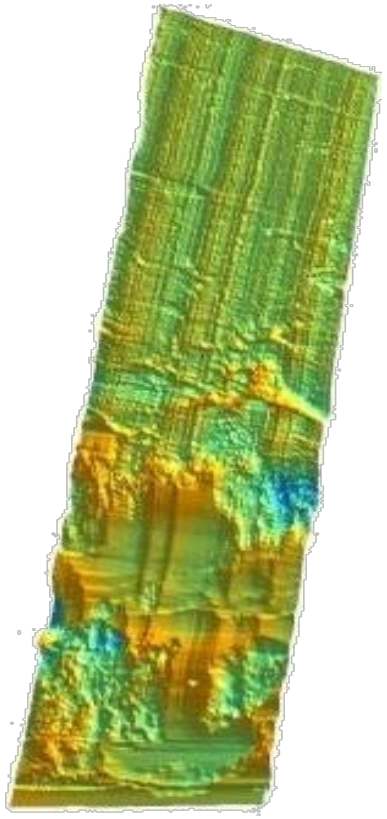
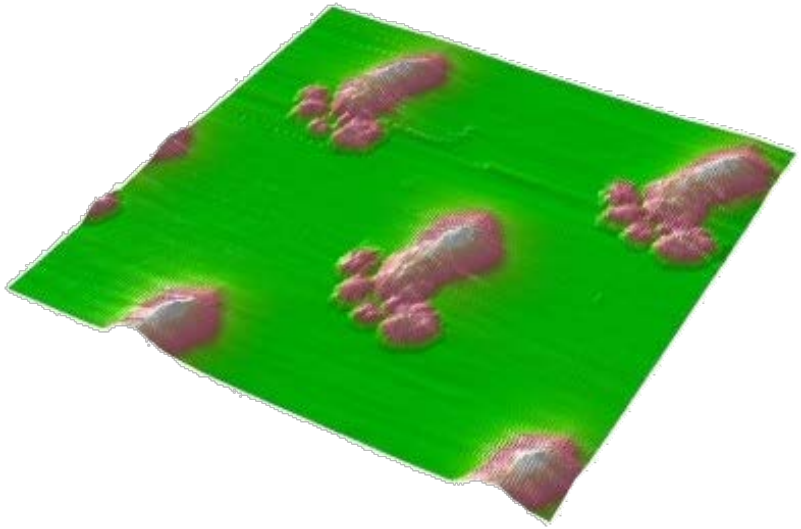


Fracture section (1 mm)
and landlside (500 m)



Nanoparticles aggregates (0,5 μm)
and grizzly bear footsteps (0,2 m)

From the nanoscale to the gigascale

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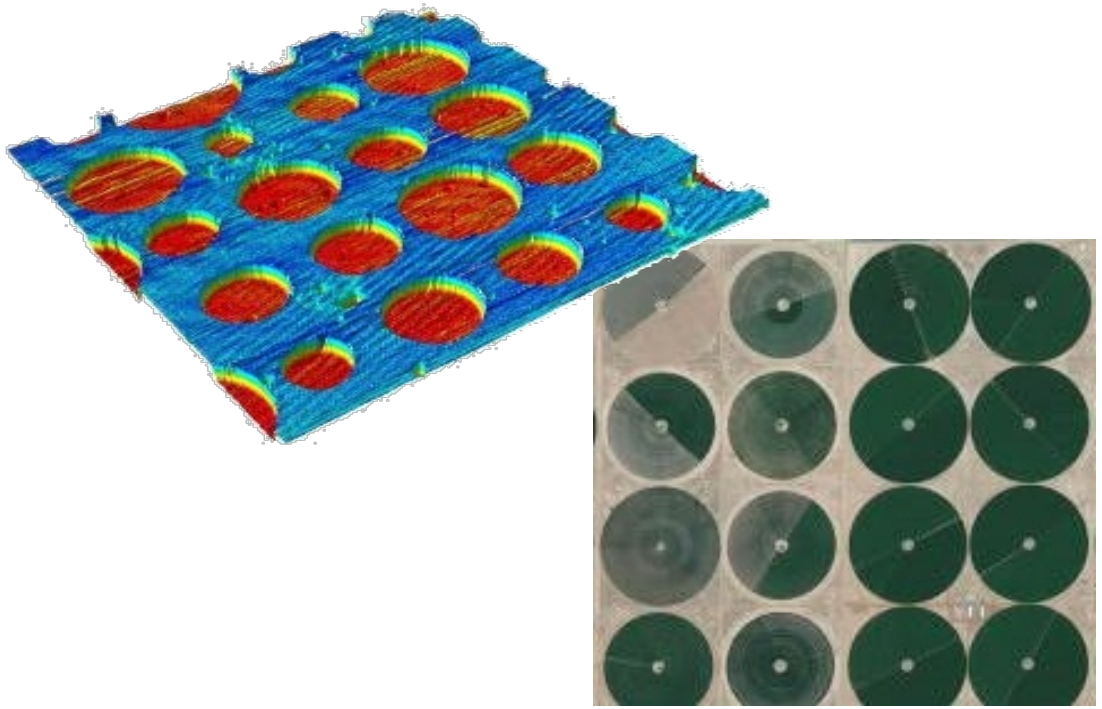
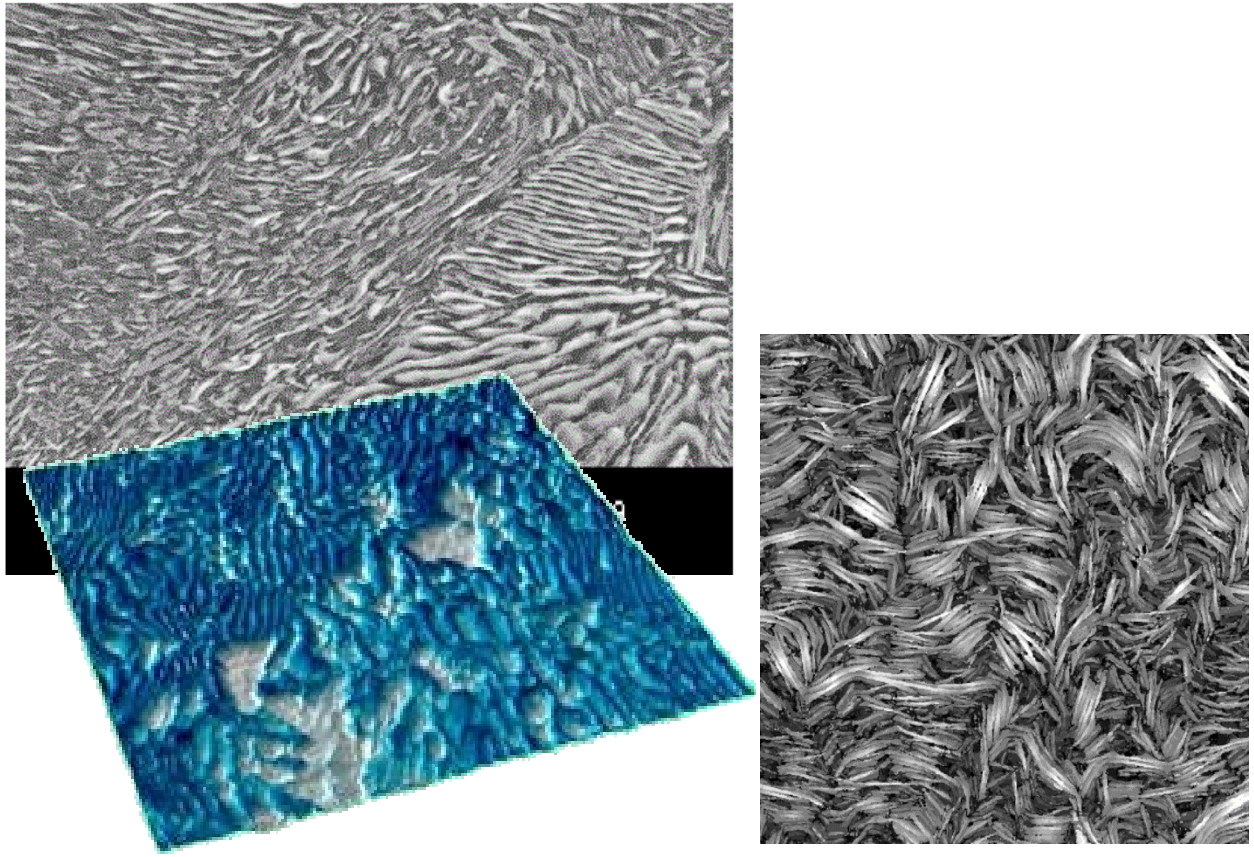


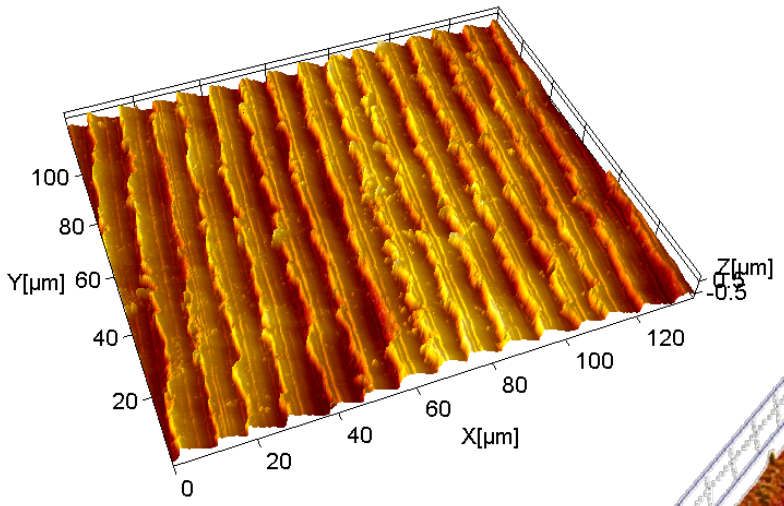
Chromium etched pattern (100 µm)
and pivot irrigated fields (2500 m)

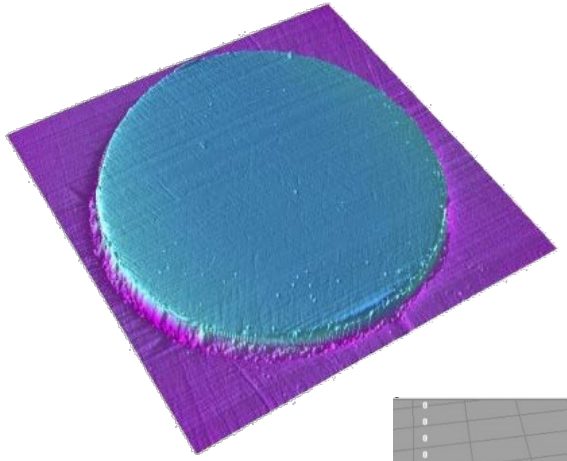


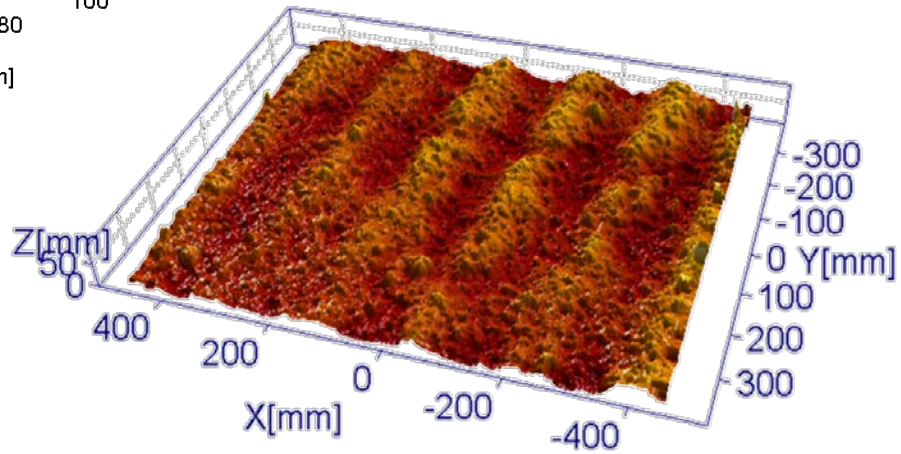
Pearlitic structure after etching (30 µm) and
SLLAC of Mediterranean land (1,5 km)

From the nanoscale to the gigascale

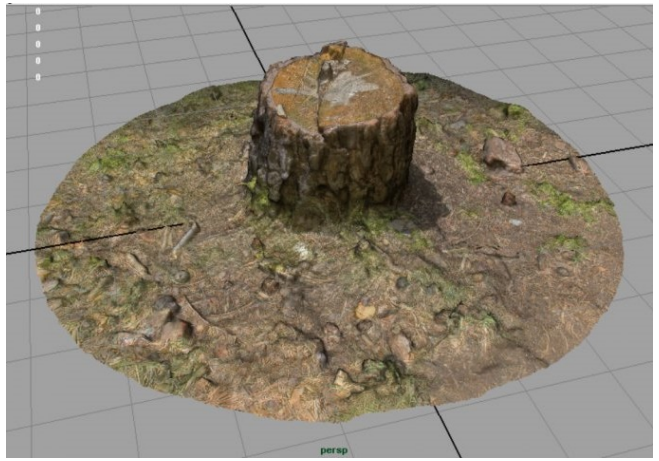
Mutiscale of shapes is actually typical of earth phenomena





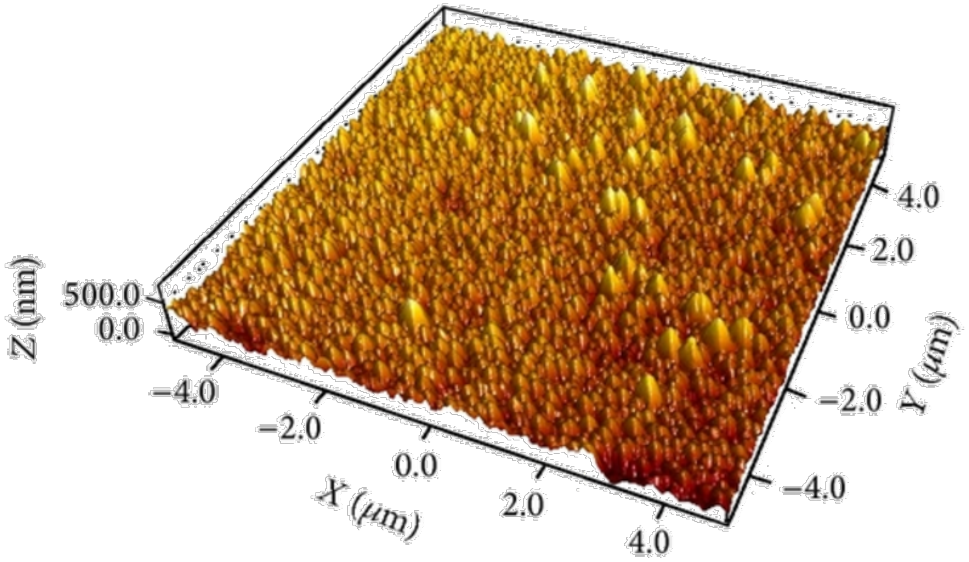


Micro-milled surface (10 µm)
and ploughed soil (1 m)

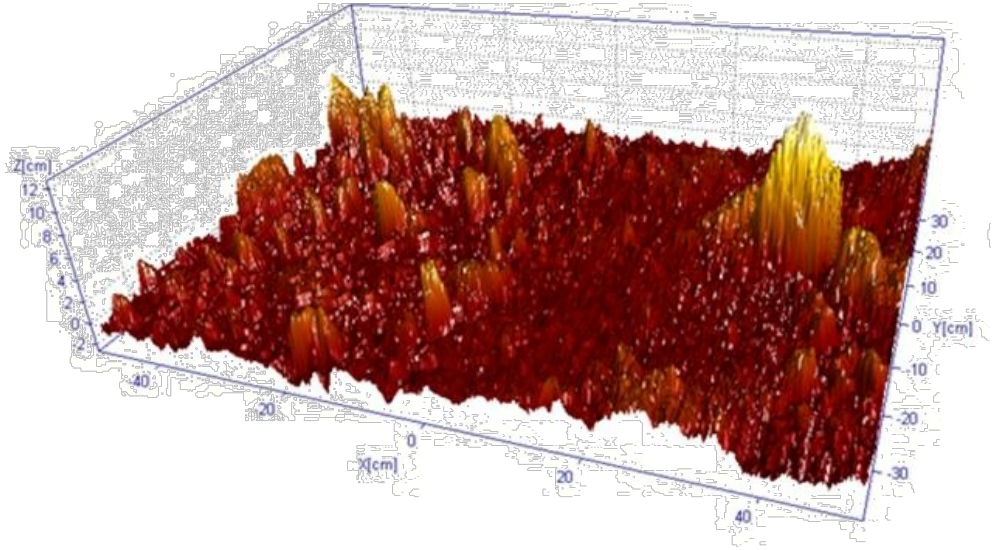


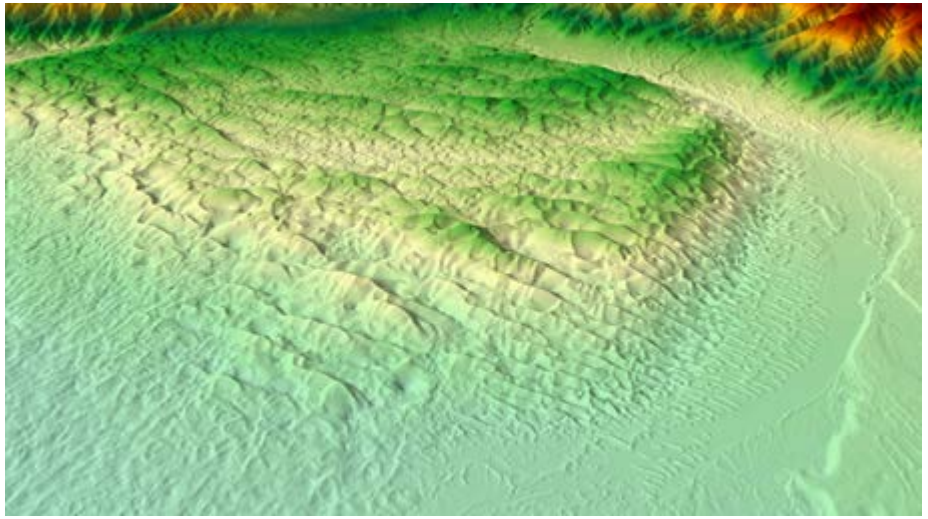
Fiberglass (125 µm)
and trunk section (0,8 m)

From the nanoscale to the gigascale



Etched glass (10 μm),
Gravel road (1 m) and
Great Sand Dunes National Park (1 km)

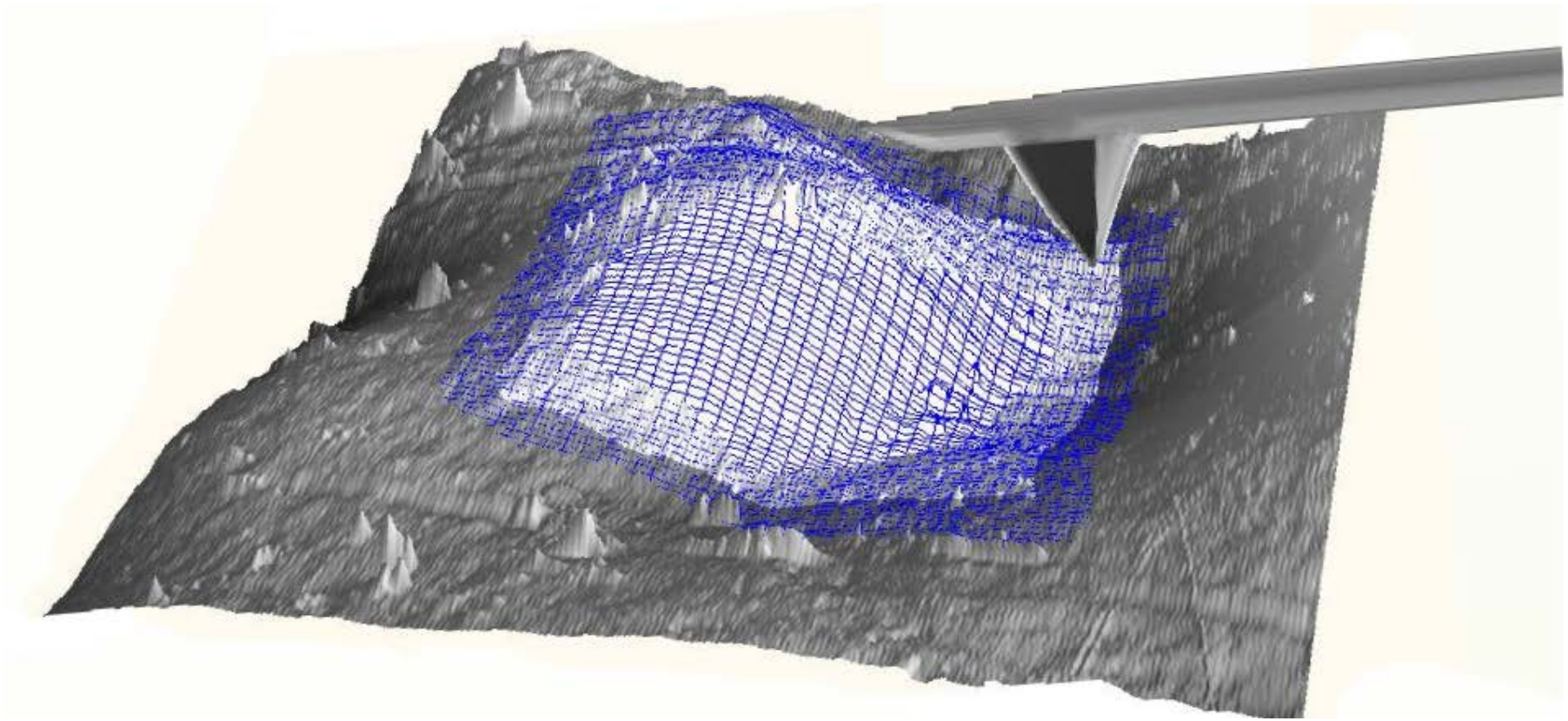




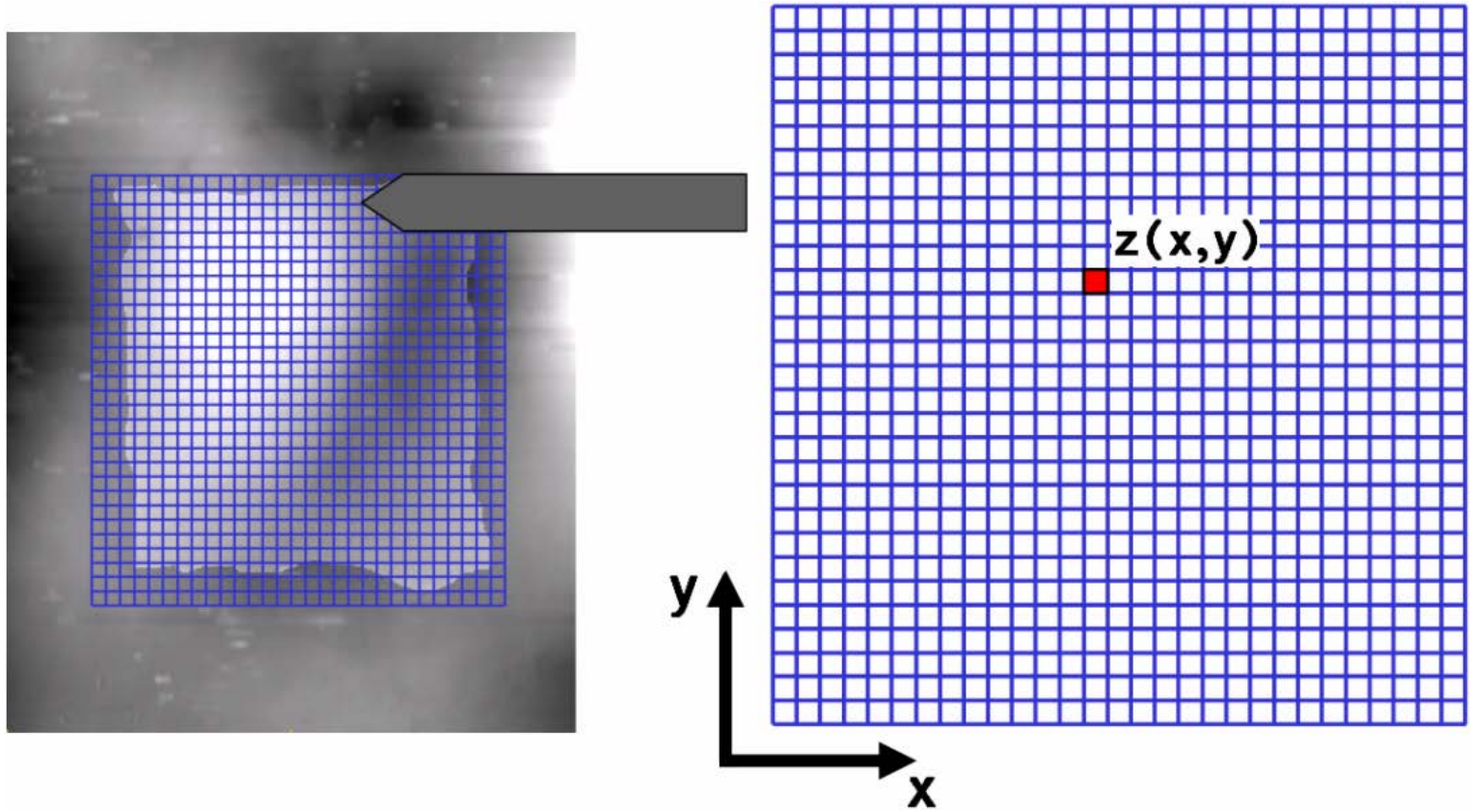
Basic definitions

What we commonly have in common is a 2D array of pixels.

Whenever a measurement is performed and a topography is mapped, a point cloud is aquired by the instrument.



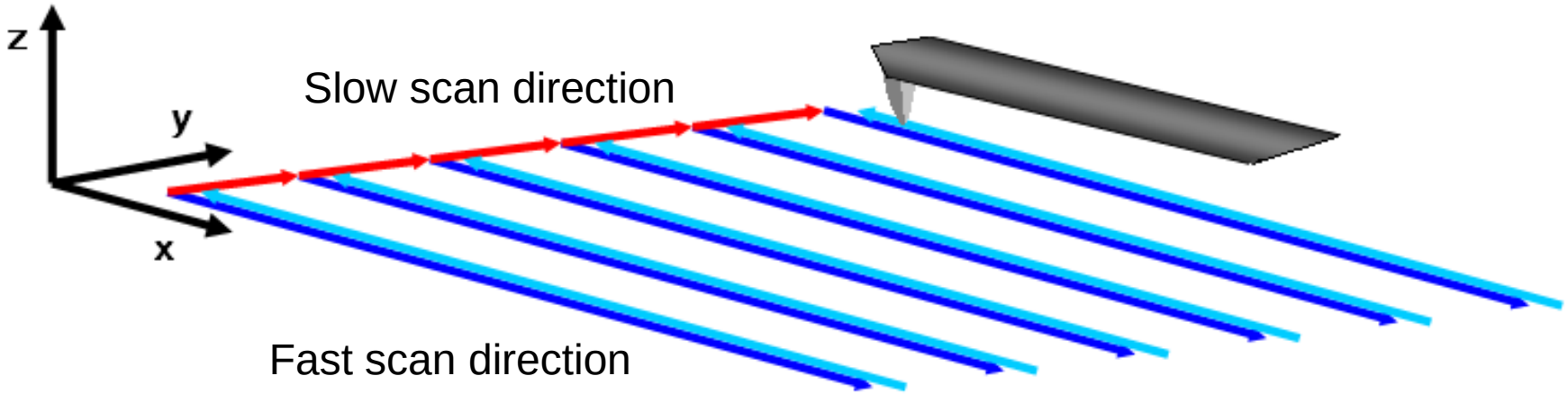
In the case of Scanning Probe Microscopes, such point cloud is a 2D array of points *x*, *y*, *z*(*x*,*y*), where *z* is the "information" revealed by the instrument: height, angle, transmitted light, conductivity, frequency, local contact stiffness,... depending on the implemented scanning technique.



Basic definitions

Two main scanning direction can be identified: the fast scan direction (the direction of the scanned profiles, either perpendicular or parallel to the cantilever) and the slow scan direction (perpendicular to the fast scan direction).

Each profile is normally scanned twice: one forward and one back. The two movements are normally indicated as “scan” or “trace” and “back-scan” or “retrace”.



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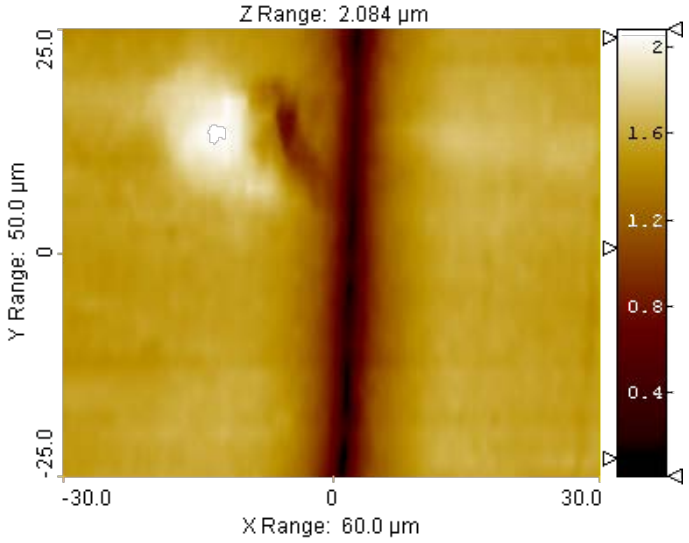
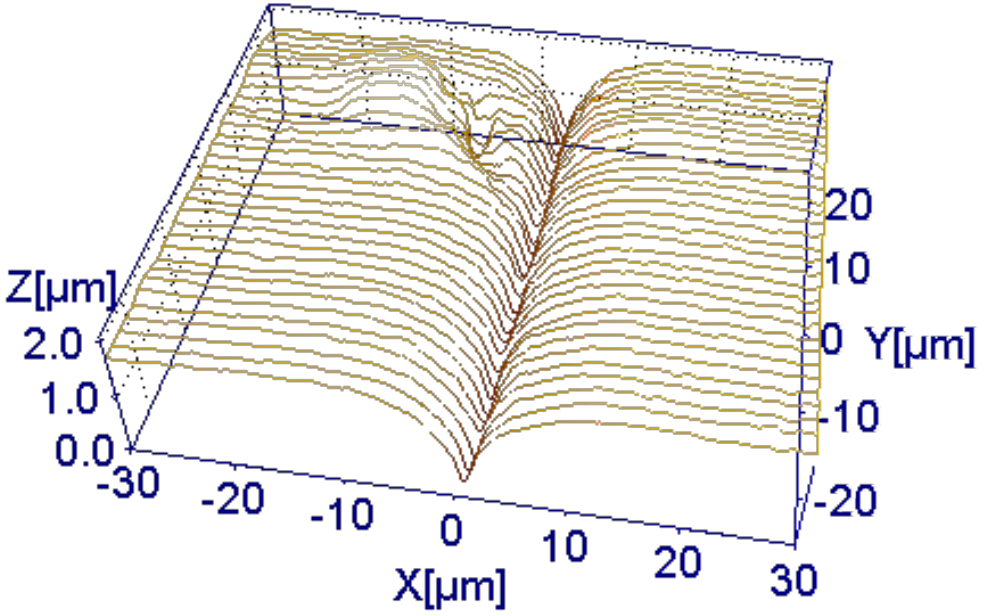
For proper data processing, SPM maps should be in general regarded as collection of profiles rather than single data sets.





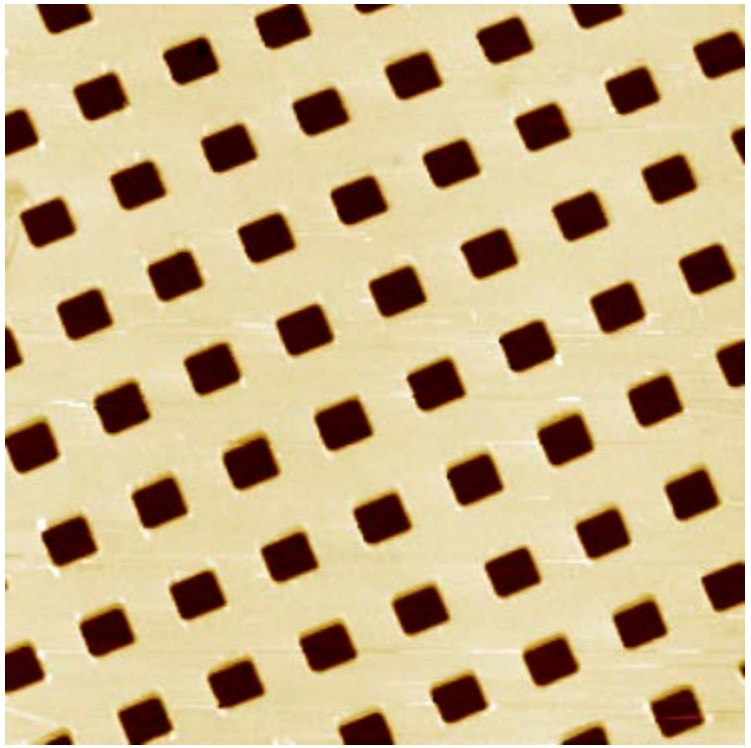
ARTIFACTS

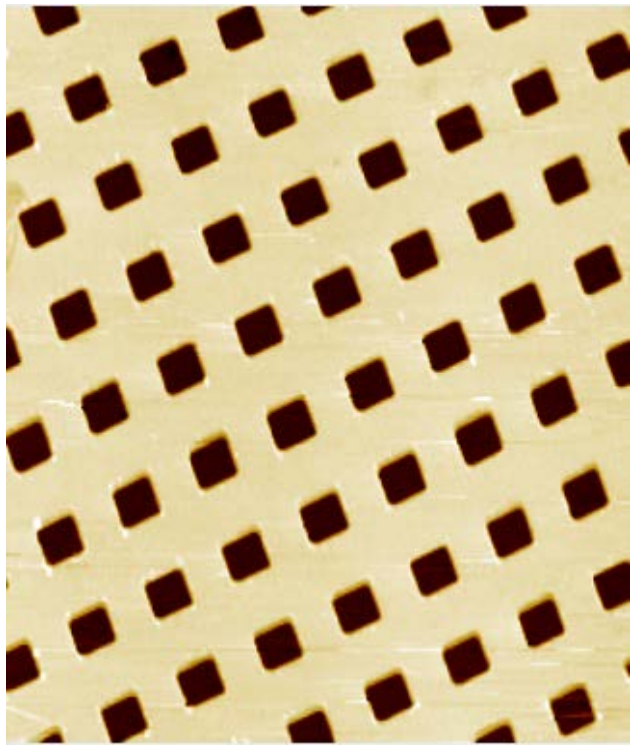
Artifacts

SOURCE	DISTORTION
scanning system	scaling, non-linearity, hysteresis, aging, creep, crosstalk, bow
tip-surface interaction	overshoots, mode-switching, convolution, unsampled parts, tip artefacts
environment	drift, noise
data-processing	filtering, levelling

Artifacts: linear and non linear (scanner) distortions

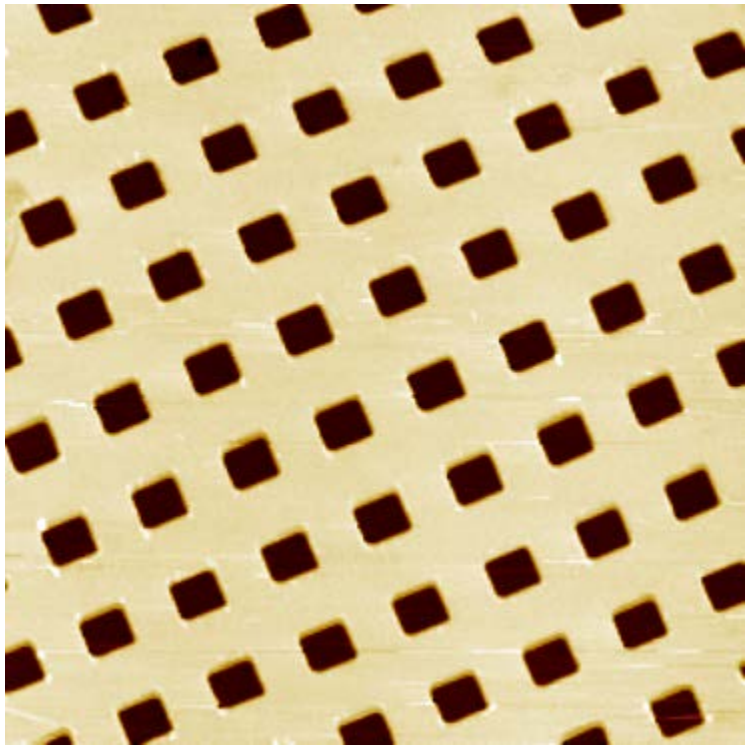
During scanning distortions occur: linear distortions

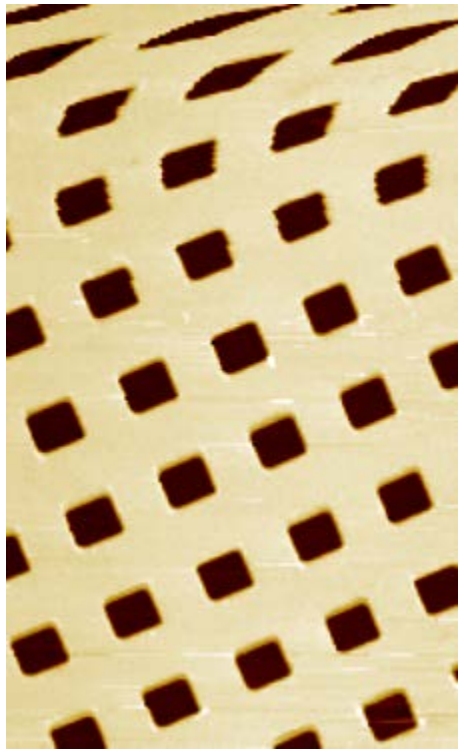




Artifacts: linear and non linear (scanner) distortions

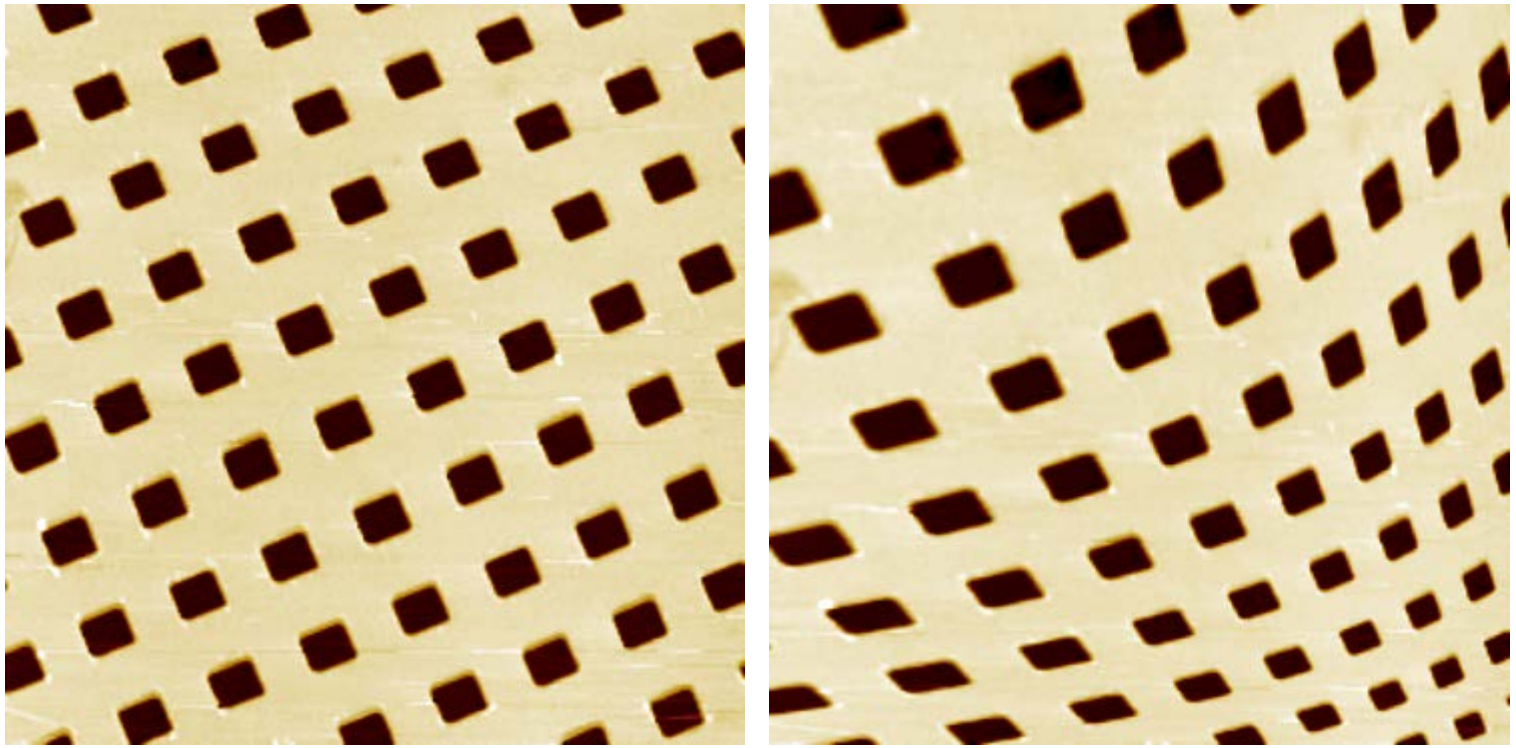
During scanning distortions occur: non linearities





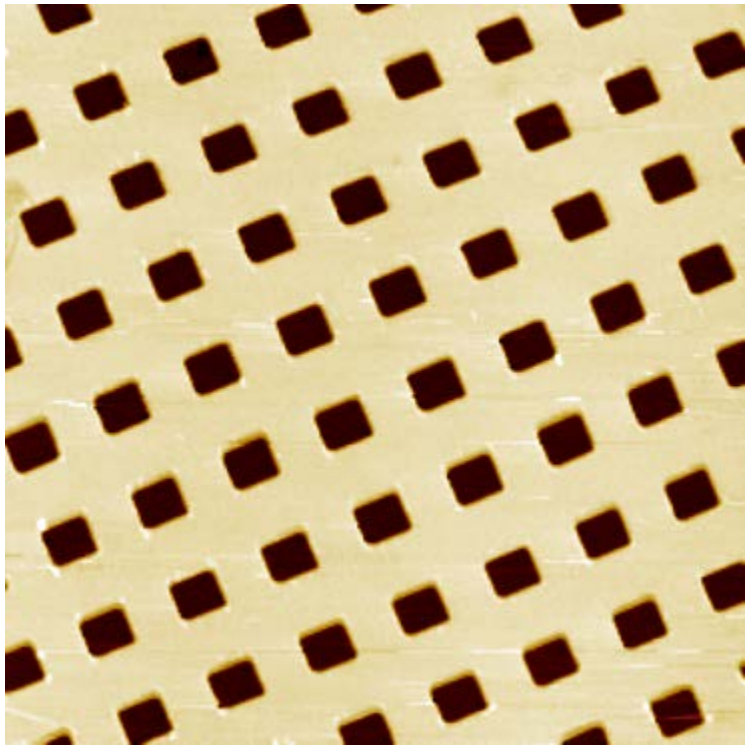
Artifacts: linear and non linear (scanner) distortions

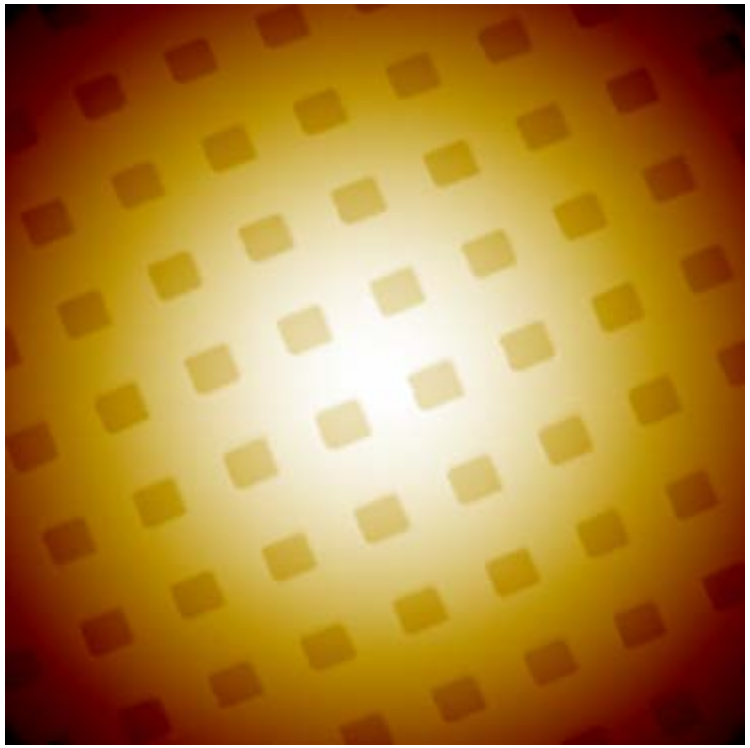
During scanning distortions occur: hysteresis



Artifacts: linear and non linear (scanner) distortions

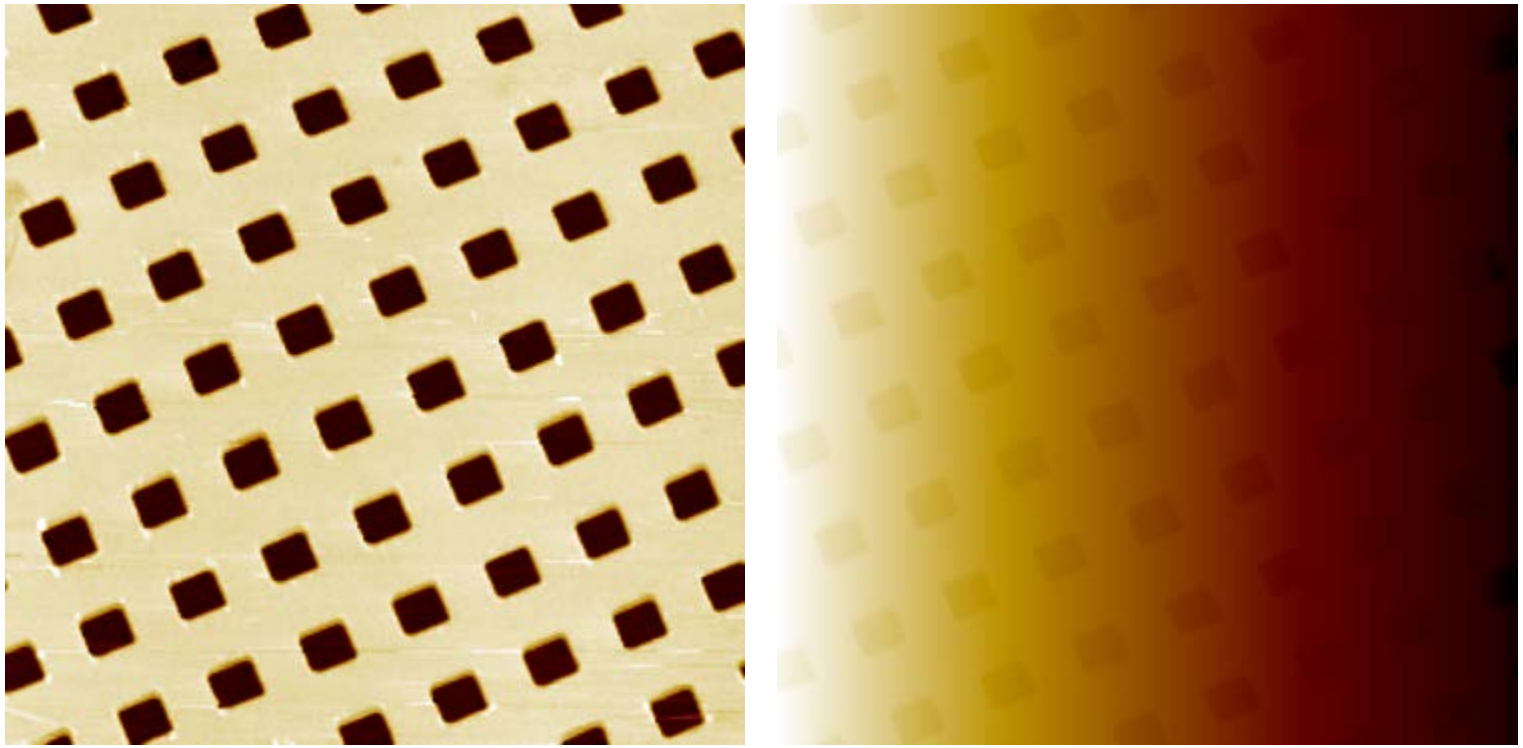
During scanning distortions occur: bow



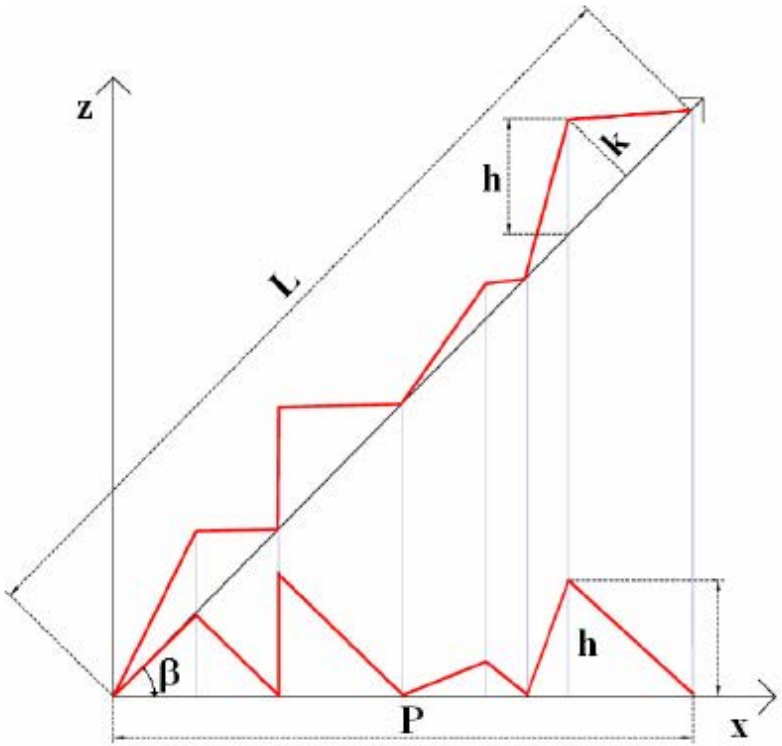


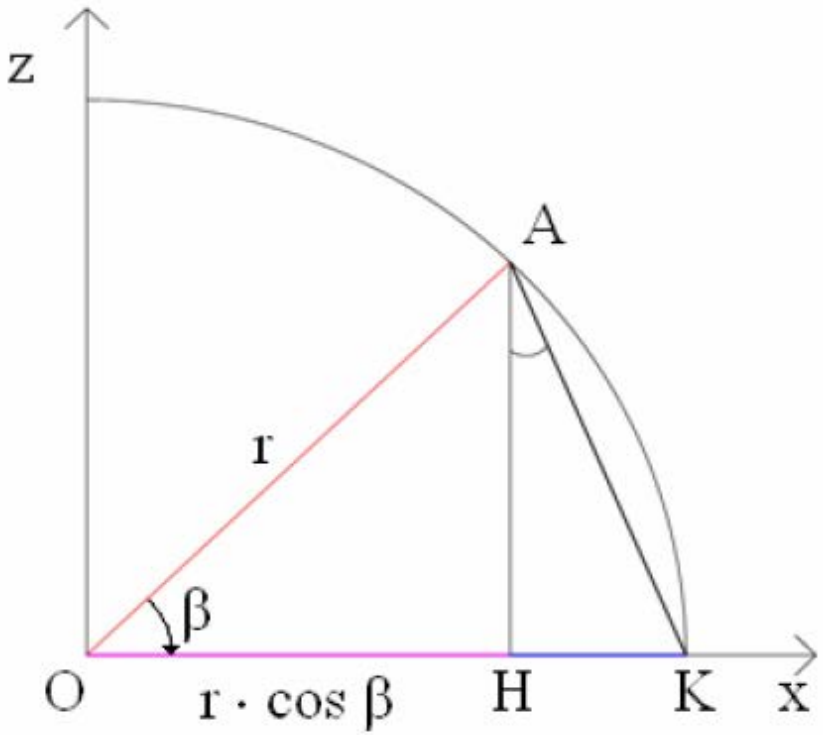
Artifacts: linear and non linear (scanner) distortions

During scanning distortions occur: installation slope



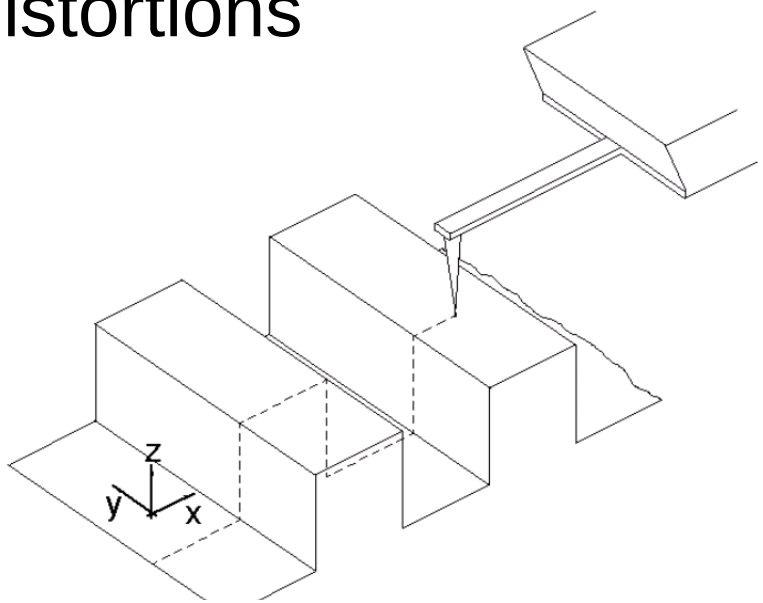
Post processing distortions: installation slope





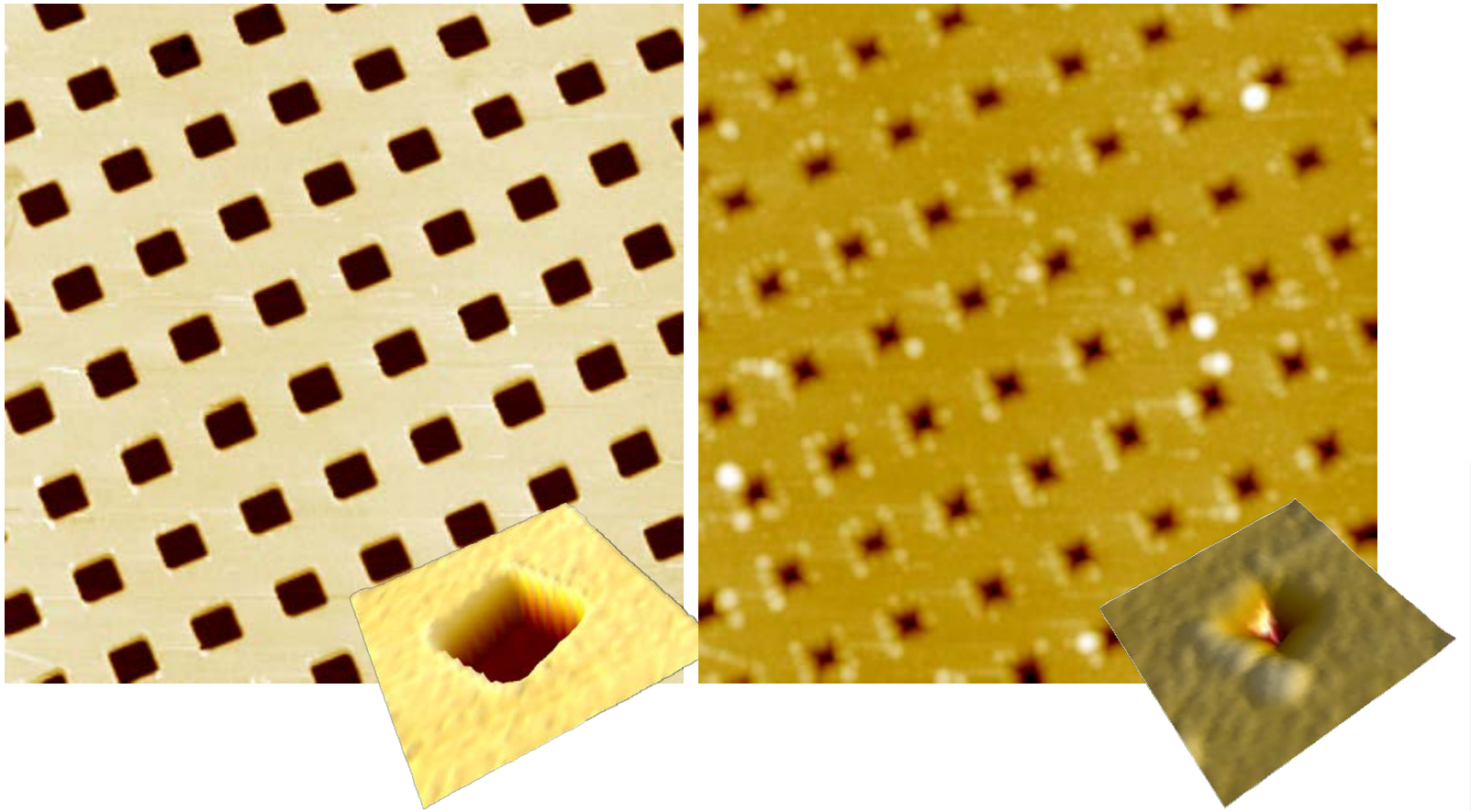
Post processing compensation: linear distortions

SPM type	Position control	Linearity
Metrological	Integrated interferometers (traceable by the laser wavelength)	~0.01%
Closed-loop	Integrated position sensors (capacitive, inductive, encoders, . . .)	<1%
Open-loop	Open loop (positioning based on the applied voltage)	<10%

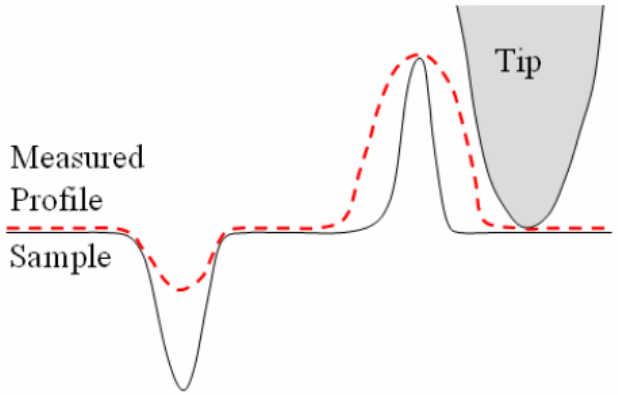


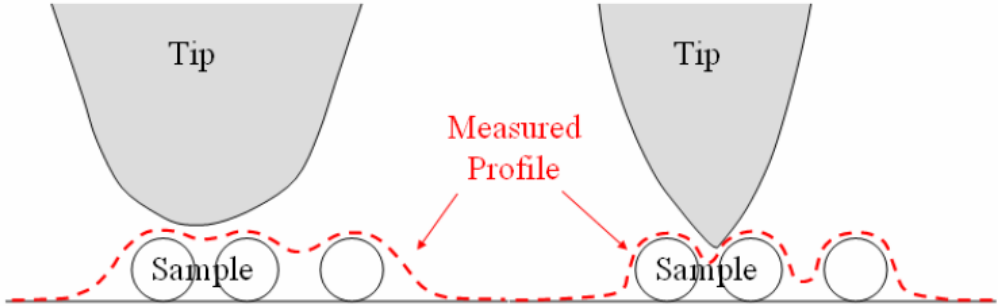
$$\begin{bmatrix} x \\ y \\ z \end{bmatrix} = \begin{bmatrix} c_{xx'} & c_{xy'} & c_{xz'} \\ c_{yx'} & c_{yy'} & c_{yz'} \\ c_{zx'} & c_{zy'} & c_{zz'} \end{bmatrix} \cdot \begin{bmatrix} x' \\ y' \\ z' \end{bmatrix} + \begin{bmatrix} c_{xx'^2} & c_{xy'^2} & c_{xz'^2} & c_{xx'y'} & c_{xx'z'} & c_{xy'z'} \\ c_{yx'^2} & c_{yy'^2} & c_{yz'^2} & c_{yx'y'} & c_{yx'z'} & c_{yy'z'} \\ c_{zx'^2} & c_{zy'^2} & c_{zz'^2} & c_{zx'y'} & c_{zx'z'} & c_{zy'z'} \end{bmatrix} \cdot \begin{bmatrix} x'^2 \\ y'^2 \\ z'^2 \\ x'y' \\ x'z' \\ y'z' \end{bmatrix} + \begin{bmatrix} c_{xx'^3} & c_{xy'^3} & c_{xz'^3} & c_{xx'^2y'} & c_{xx'^2z'} & c_{xx'y'^2} & c_{xx'z'^2} & c_{xy'^2z'} & c_{xy'z'^2} & c_{xx'y'z'} \\ c_{yx'^3} & c_{yy'^3} & c_{yz'^3} & c_{yx'^2y'} & c_{yx'^2z'} & c_{yx'y'^2} & c_{yx'z'^2} & c_{yy'^2z'} & c_{yy'z'^2} & c_{yx'y'z'} \\ c_{zx'^3} & c_{zy'^3} & c_{zz'^3} & c_{zx'^2y'} & c_{zx'^2z'} & c_{zx'y'^2} & c_{zx'z'^2} & c_{zy'^2z'} & c_{zy'z'^2} & c_{zx'y'z'} \end{bmatrix} \cdot \begin{bmatrix} x'^3 \\ y'^3 \\ z'^3 \\ x'^2y' \\ x'^2z' \\ x'y'^2 \\ x'z'^2 \\ y'^2z' \\ y'z'^2 \\ x'y'z' \end{bmatrix} + \sigma \begin{bmatrix} x'^3 \\ y'^3 \\ z'^3 \\ x'^2y' \\ x'^2z' \\ x'y'^2 \\ x'z'^2 \\ y'^2z' \\ y'z'^2 \\ x'y'z' \end{bmatrix}$$

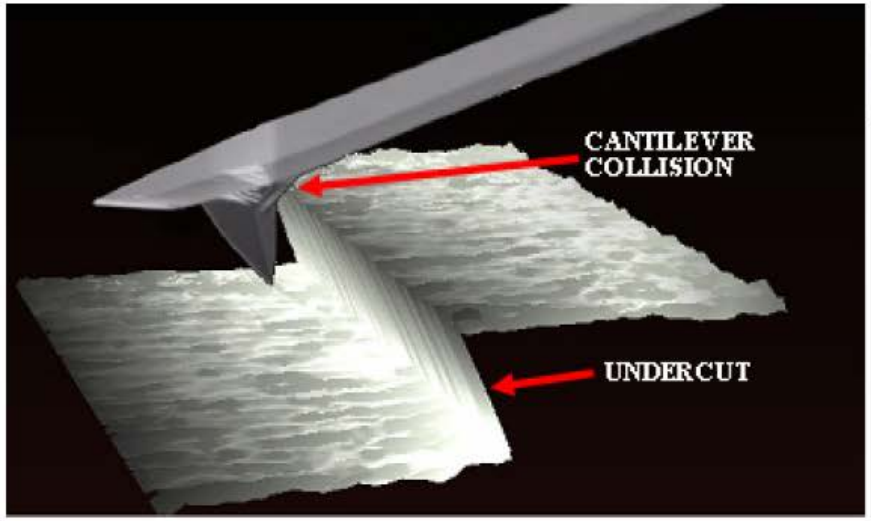
During scanning distortions occur: tip convolution

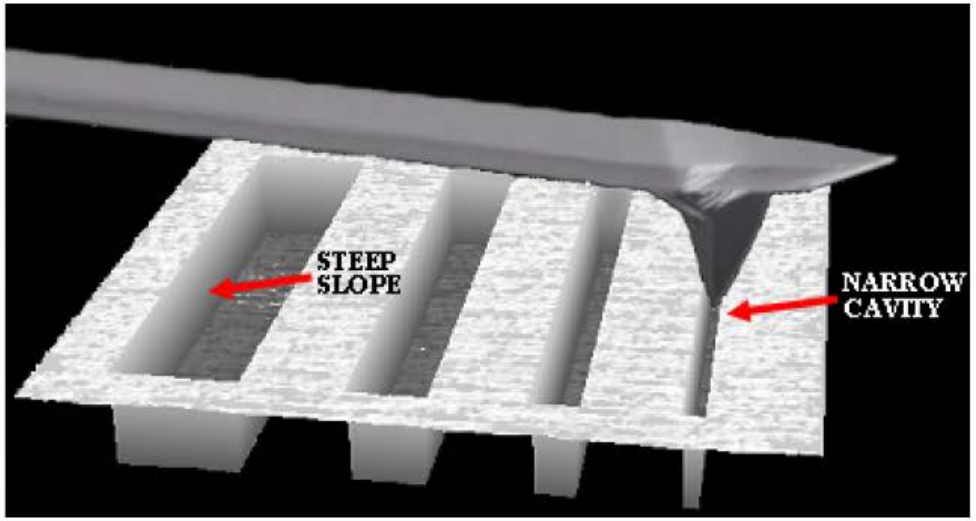


During scanning distortions occur: tip convolution

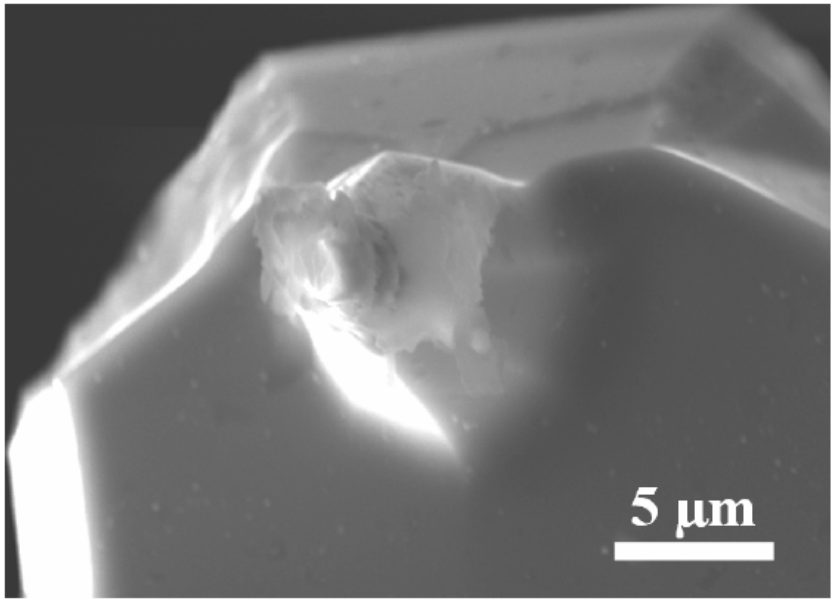


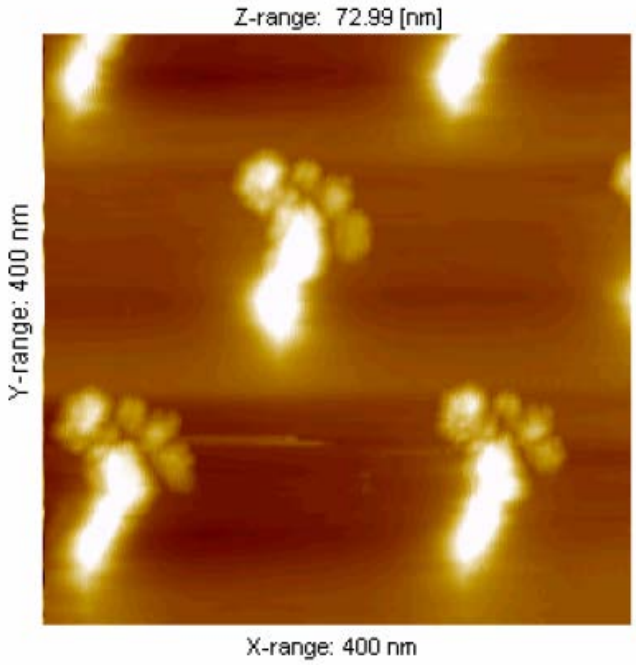






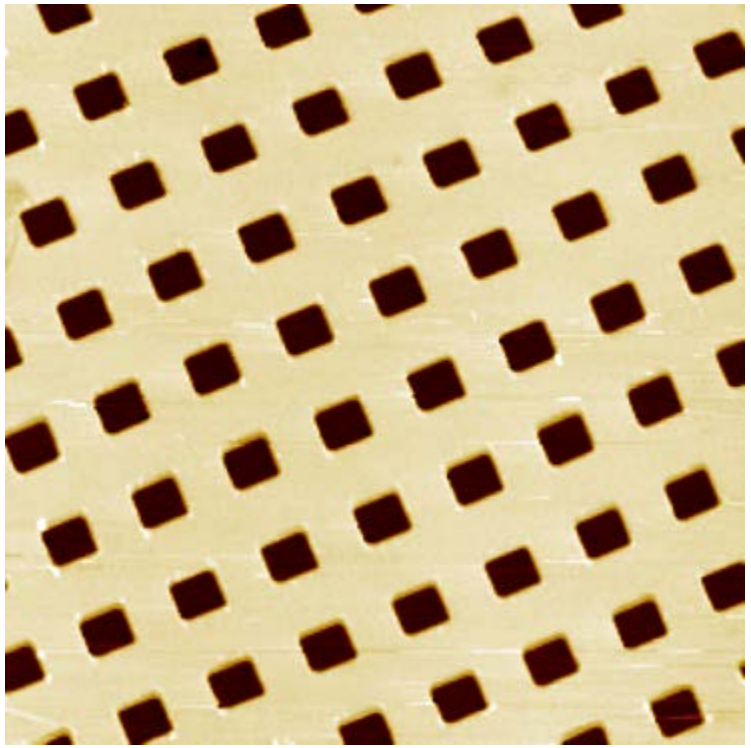
During scanning distortions occur: tip convolution

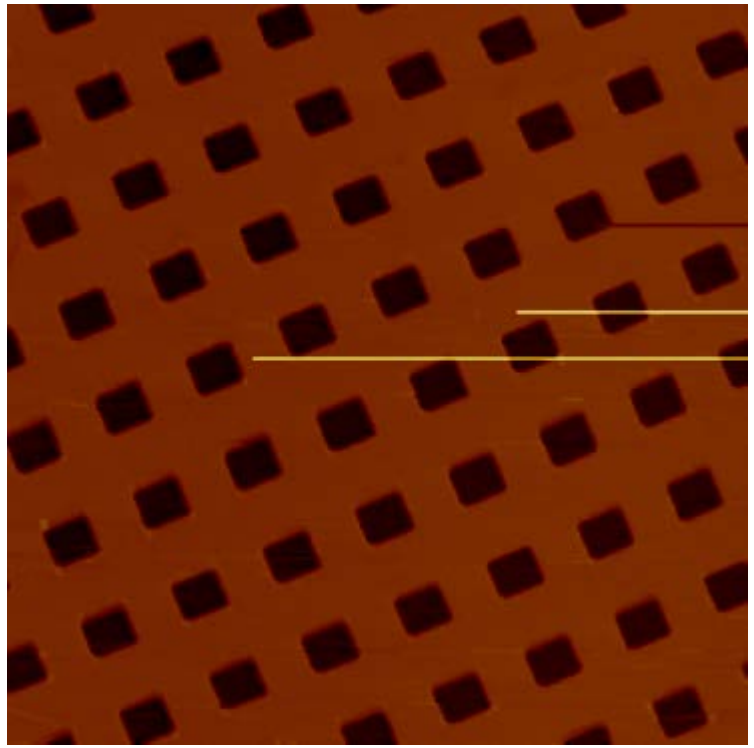




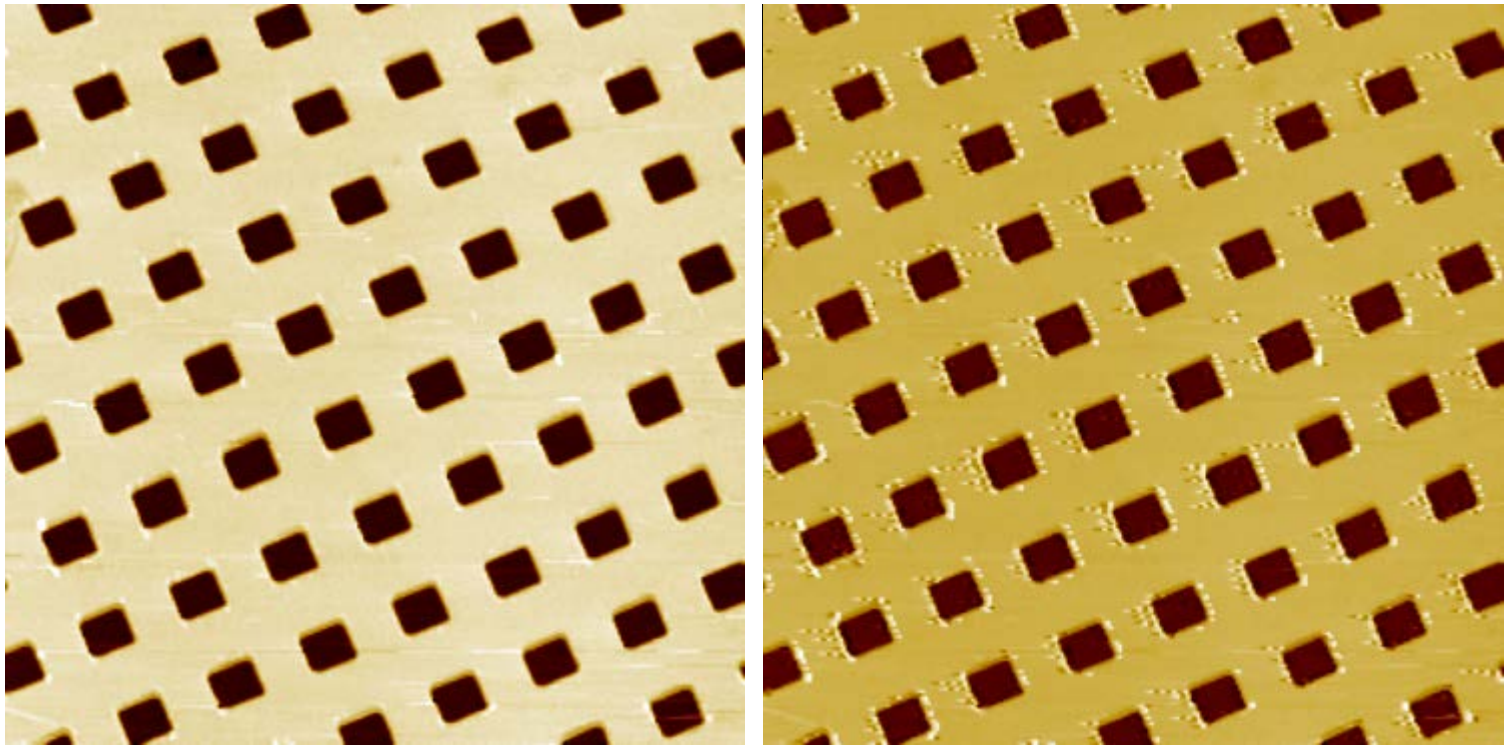
Artifacts: probe distortions

During scanning distortions occur: dust pick up



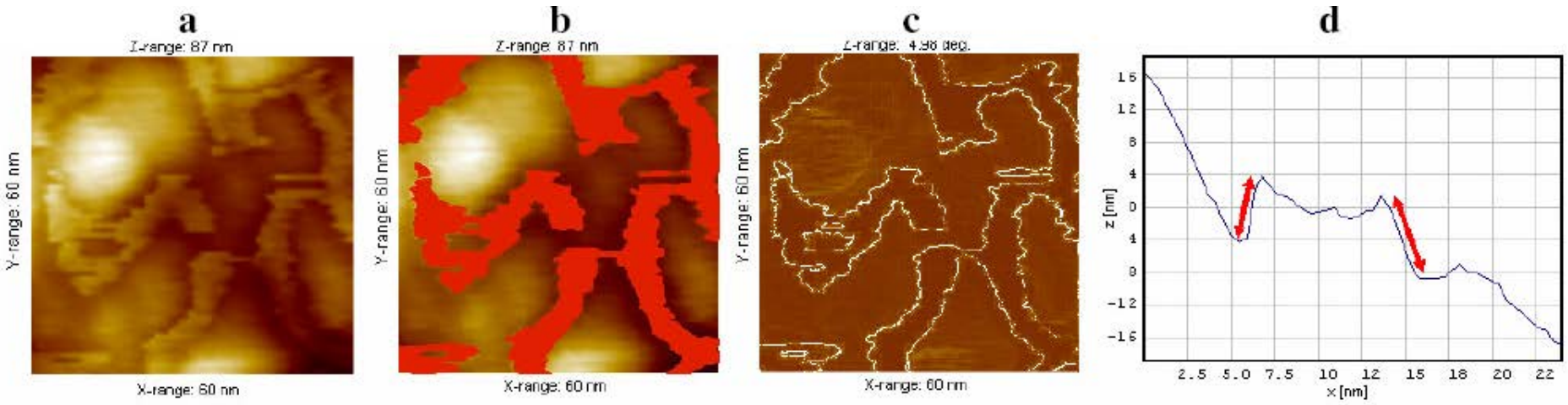


During scanning distortions occur: feedback



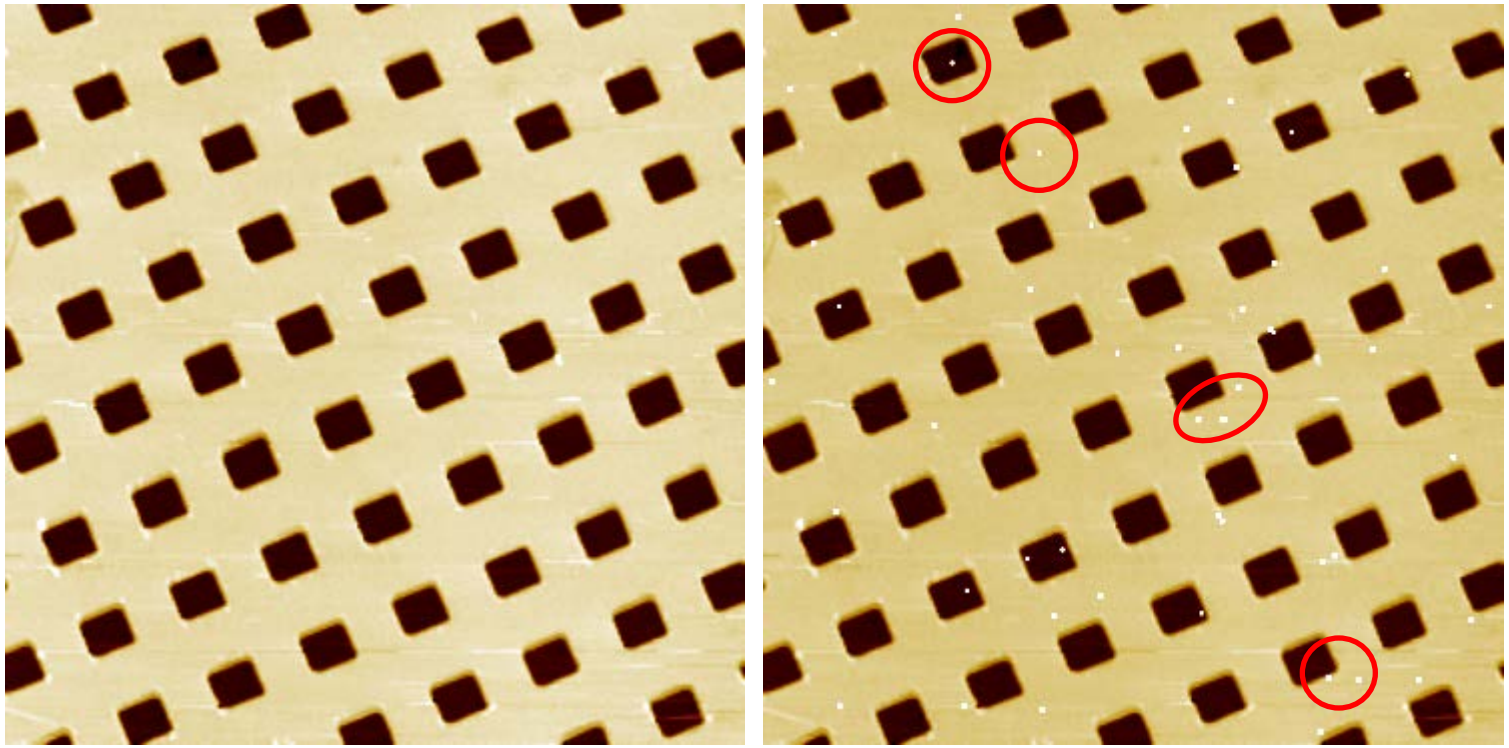
Typical edge artifacts: a) Overshoots, related to bad compensation of creep and hysteresis of the vertical servo control; b) feedback instability due to excessive gain and (c) smooth edge, the two terraces are far apart from each other.

During scanning distortions occur: mode switching

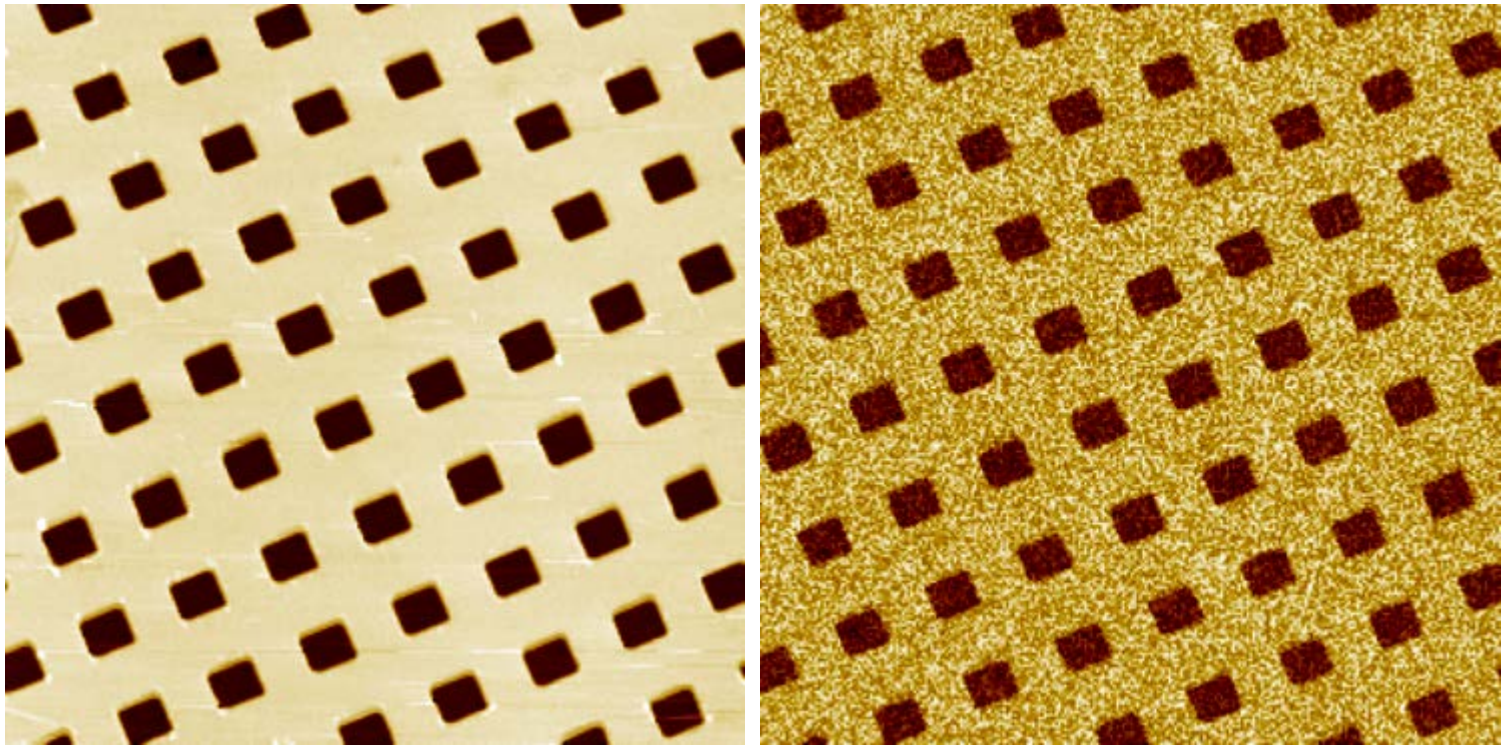


a) Fringes around NiO structures due to mode switching; b) and c) segmentation and isolation of jumps; d) profile evidencing an apparent height level shift.

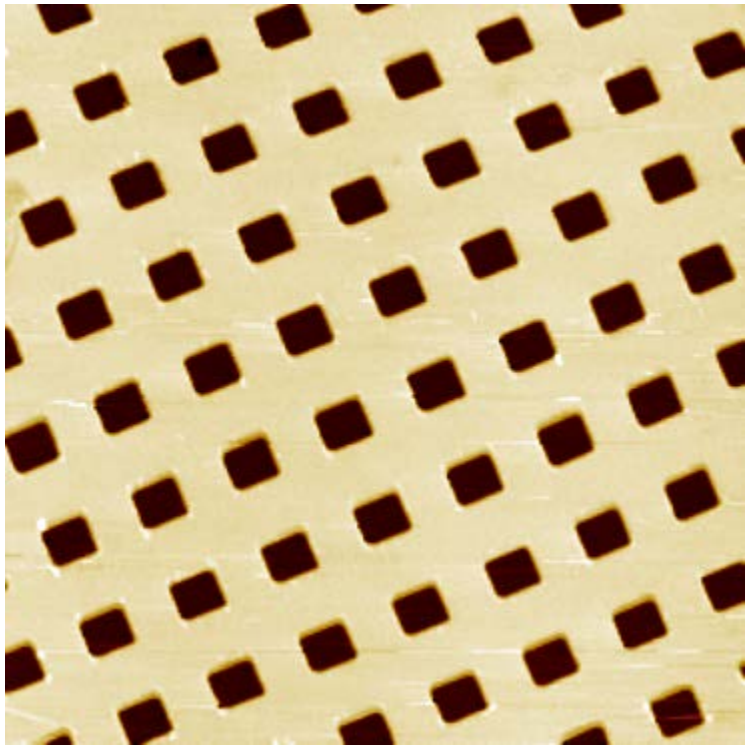
During scanning distortions occur: spikes

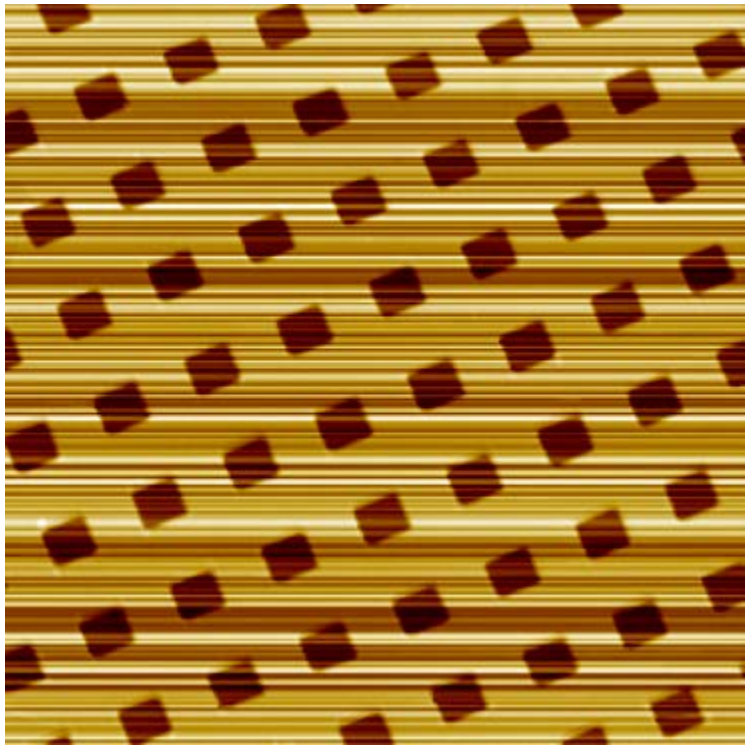


During scanning distortions occur: noise

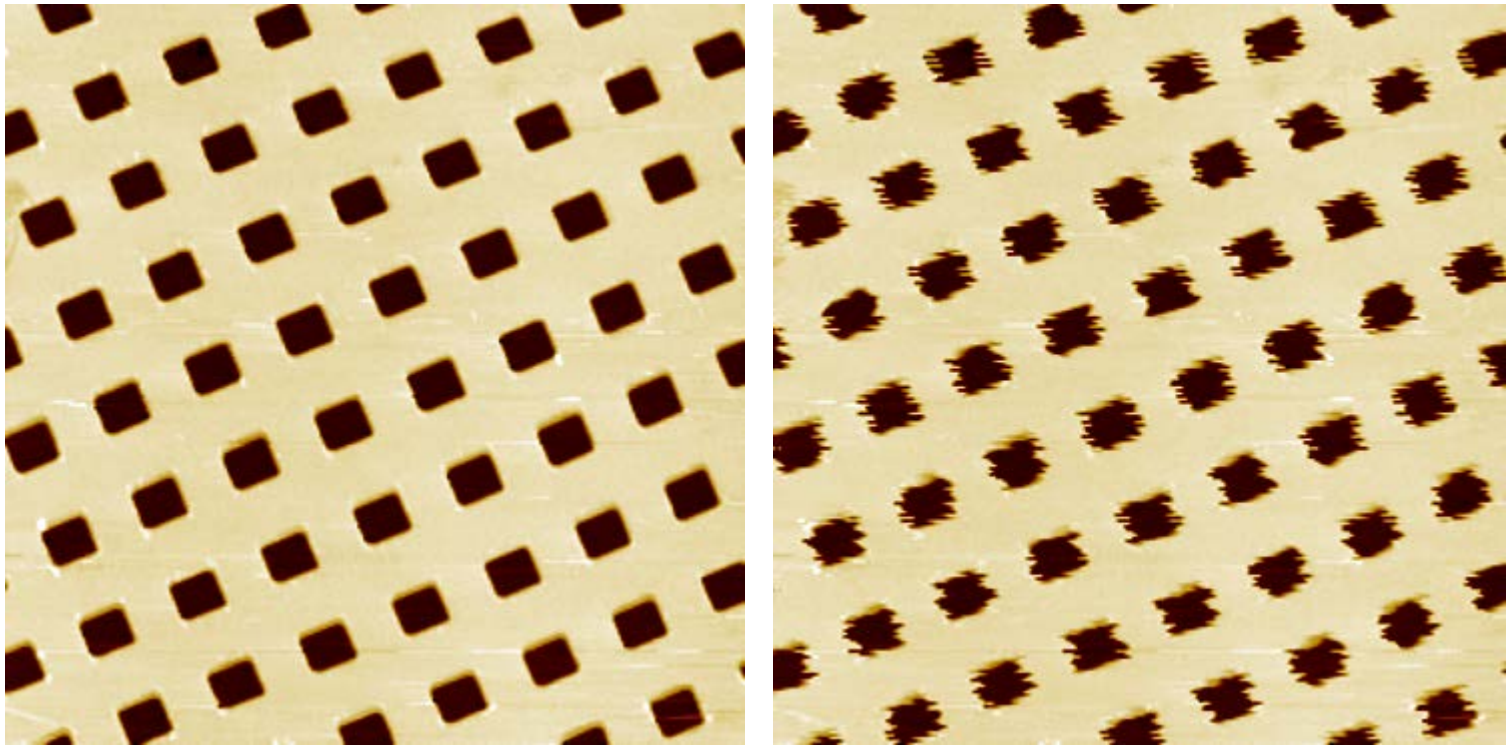


During scanning distortions occur: vertical drift

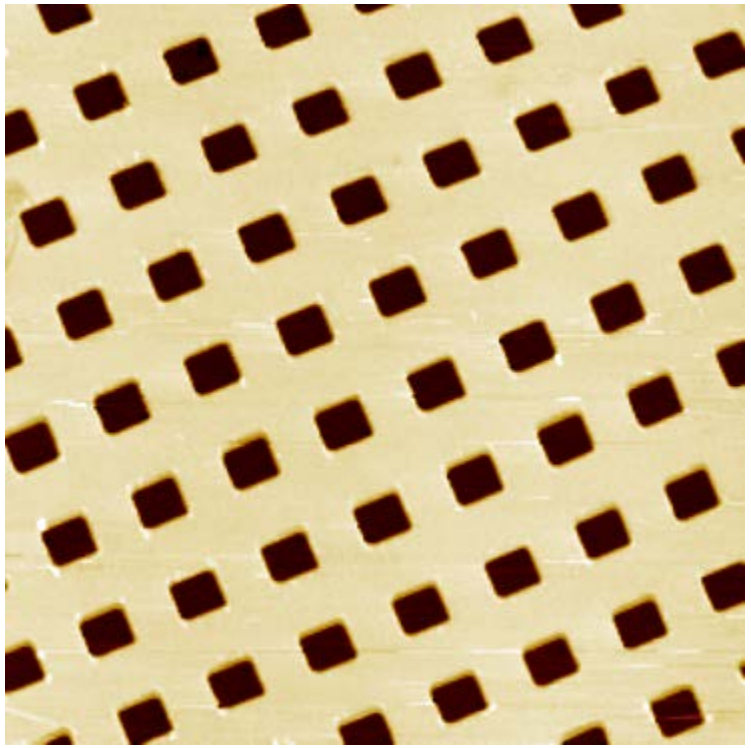


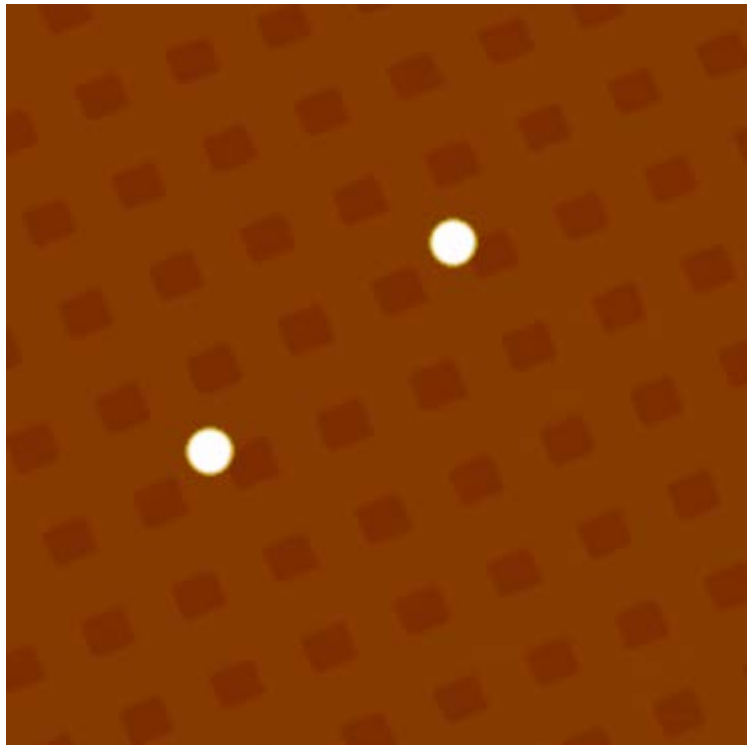


During scanning distortions occur: horizontal drift

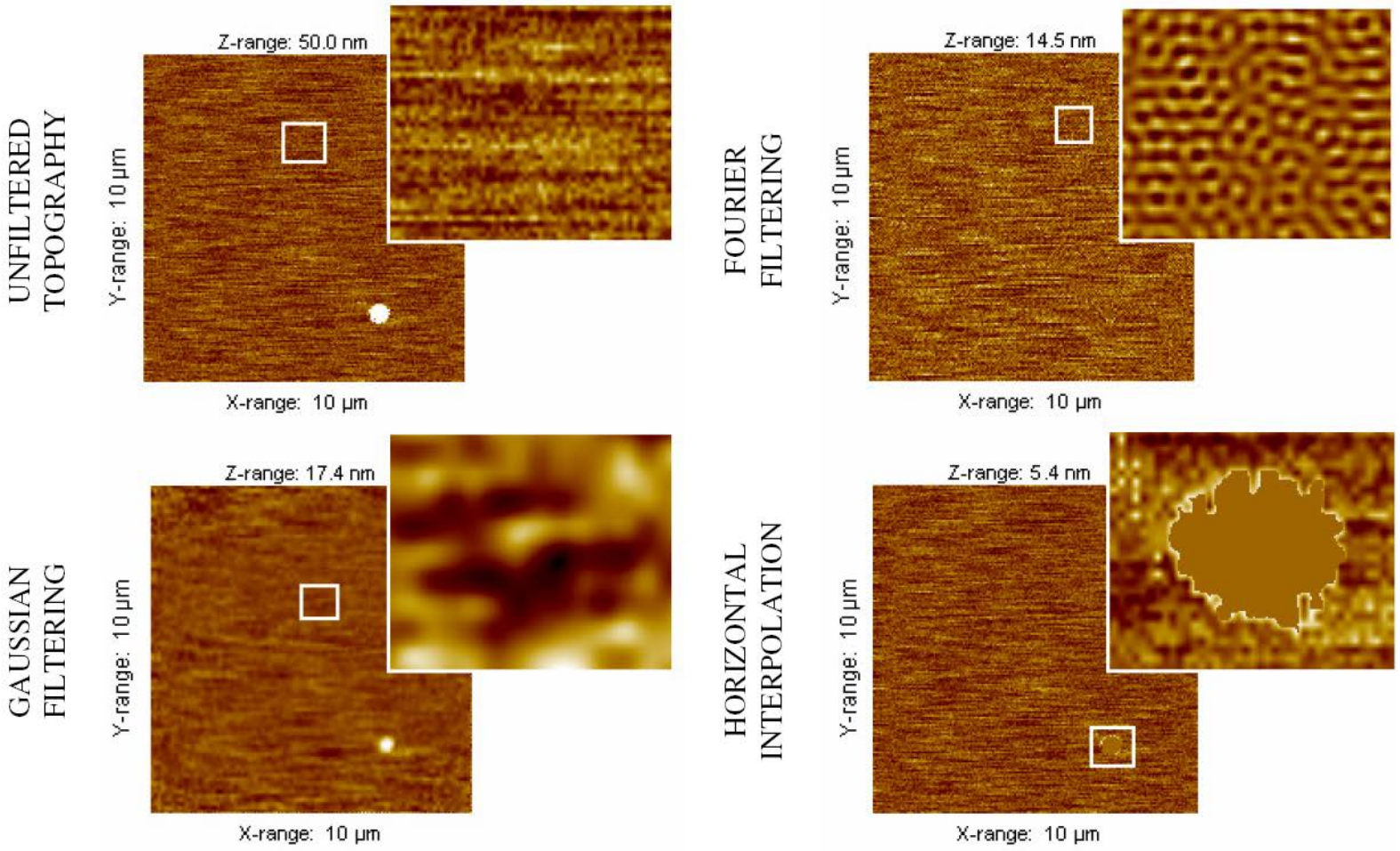


During scanning distortions occur: contaminations

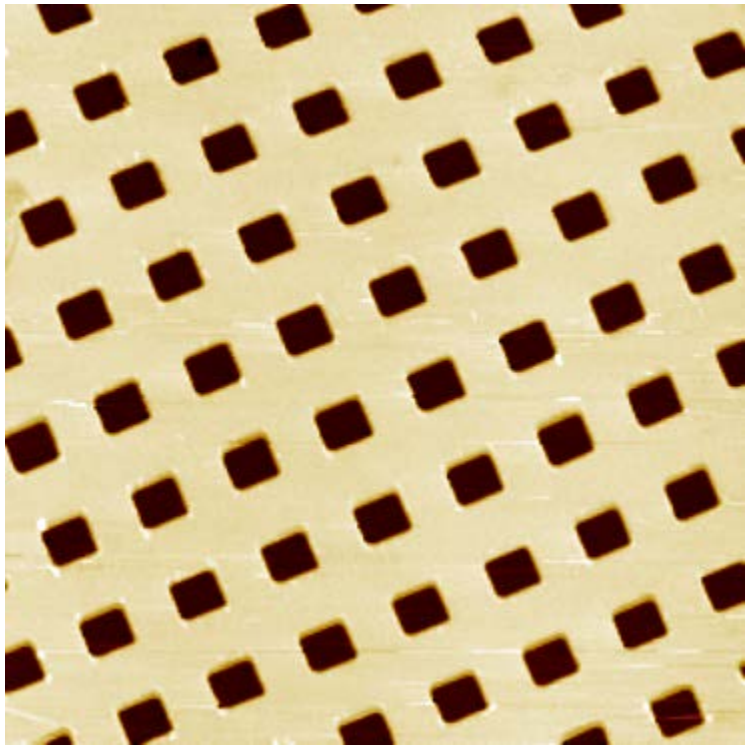


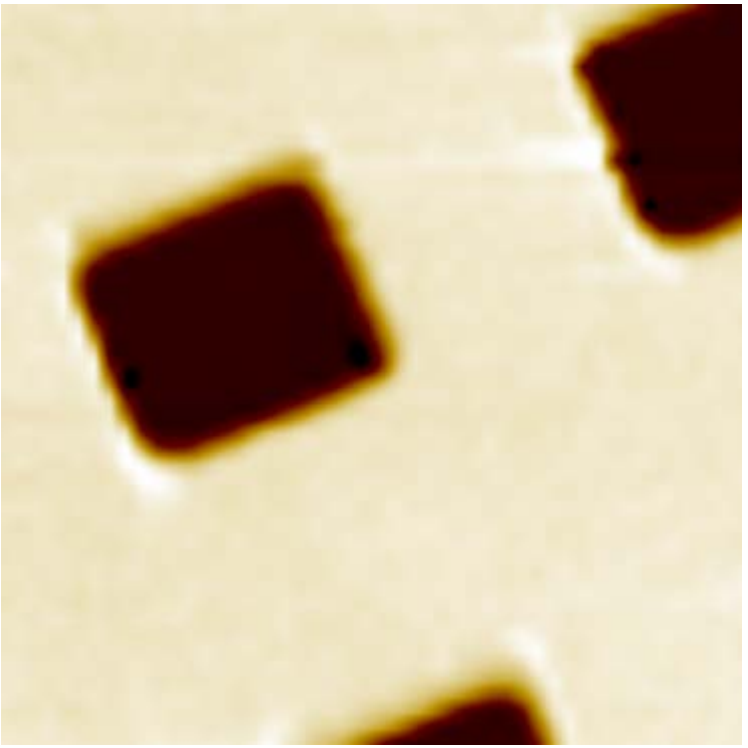


Post processing distortions: filtering

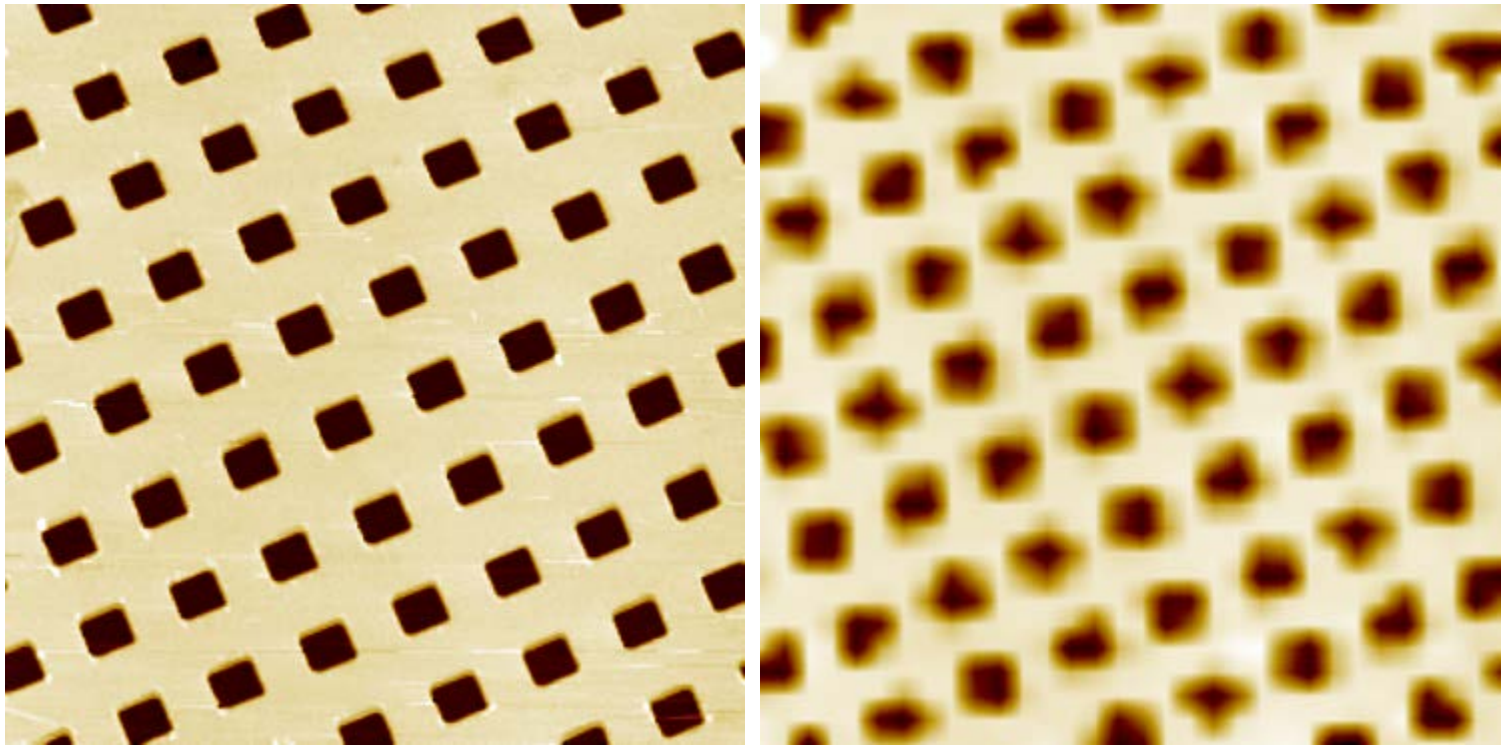


During scanning distortions occur: bad range definition

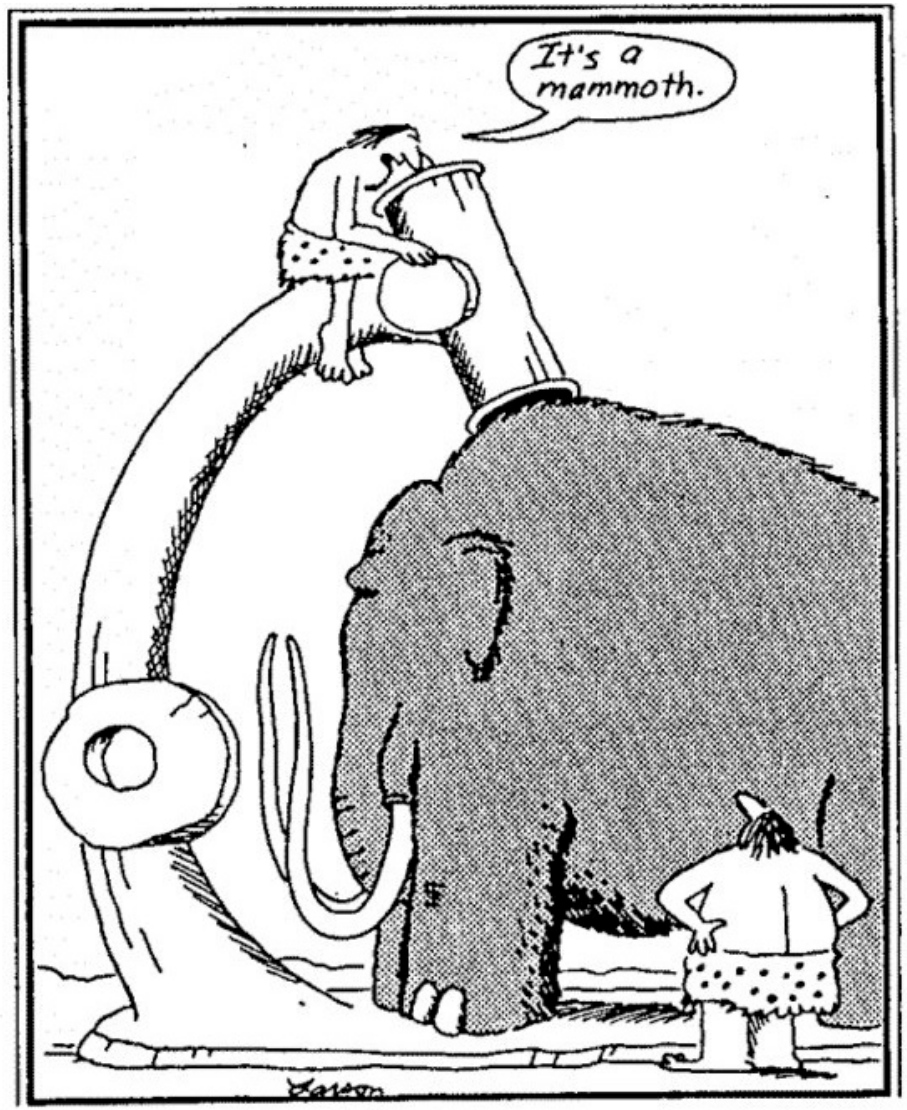




During scanning distortions occur: low resolution



APPROACH



Early microscope

SPM analyses approach

There are different approaches to SPM measurements:

- Top down: «I don't know anything about the surface so let's start from a 50×50µm fast scan and let's see»

- Bottom up/bottom around «I don't know anything about the surface but I know there is something, so let's start from a 0,5×0,5 µm slow scan high resolution and let's see»

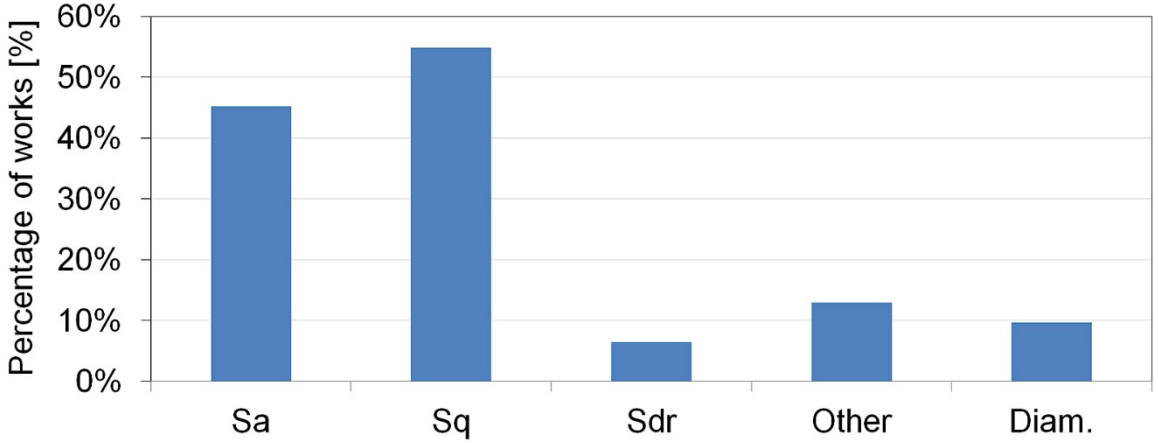
- Focused scan «I perfectly know my surface so let's do 5 measurements 2×2µm slow/high resolution set of scans»

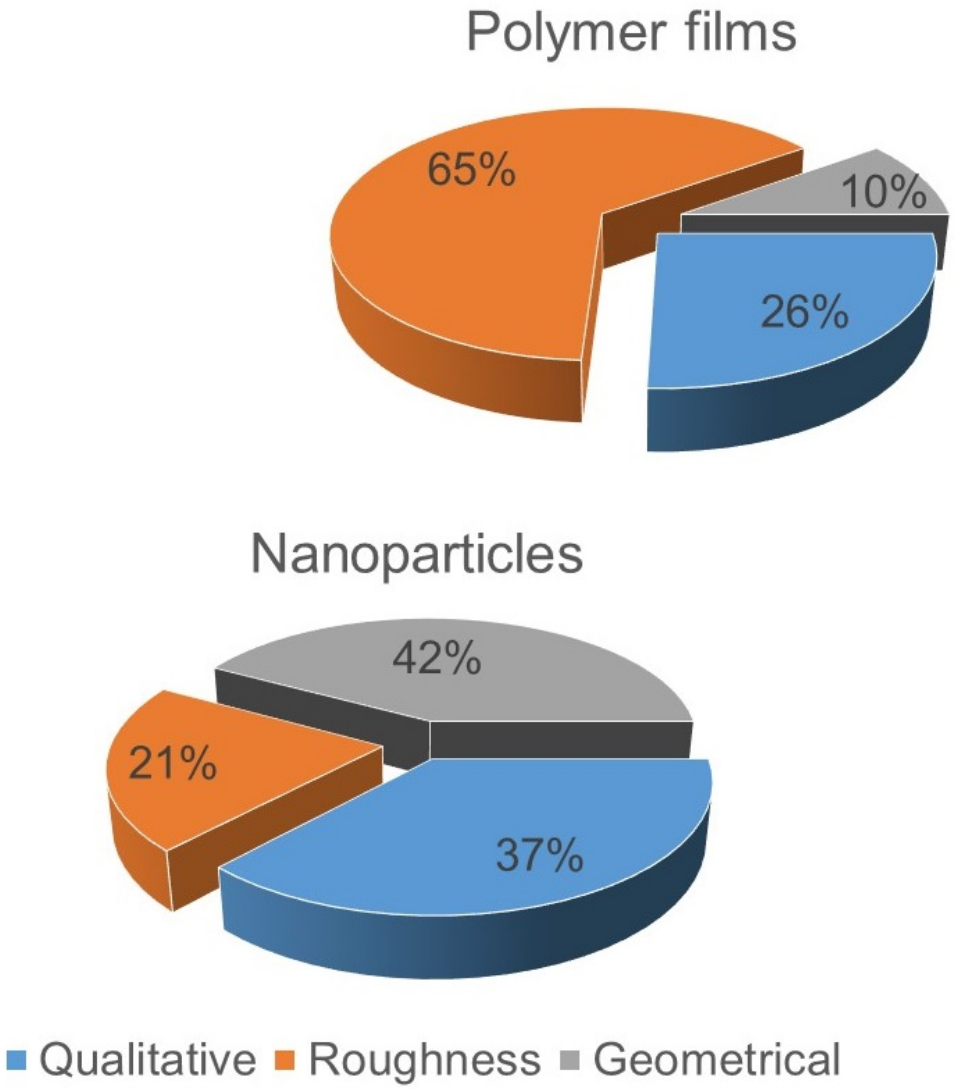
Often our approach to the measurement is not considering what is coming next and in particular:

- The need for calibration
- The need for extraction of quantitative parameters

SPM analyses approach

Looking at published papers, different trends are recognizable among different research field (such as food packaging films, or nanoparticles), however some consideration can be done:





SPM analyses approach

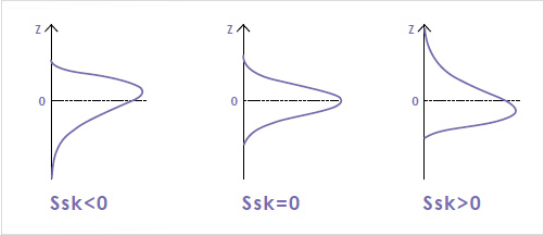
Different groups of roughness parameters, including: height parameters (root mean square roughness, kurtosis, skewness,…), function related parameters (material ratio, volume,…), hybrid parameters (interfacial area ratio, root mean square gradient,…), spatial parameters (autocorrelation functions, texture direction,…)

$$Sa = \frac{1}{A}\iint_A |z(x,y)|dxdy$$

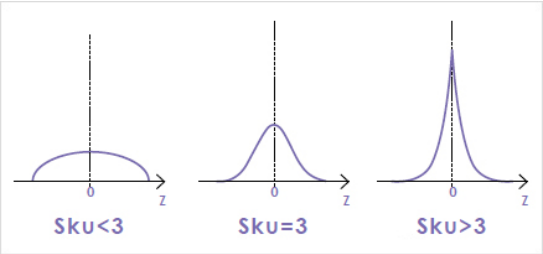
$$Sq = \sqrt{\frac{1}{A}\iint_A [z(x,y)-\bar{z}]^2 dxdy}$$

$$Sdr = \frac{1}{A}\iint_A \sqrt{\left[1+\left(\frac{\partial z(x,y)}{\partial x}\right)^2+\left(\frac{\partial z(x,y)}{\partial y}\right)^2\right]}-1\,dxdy$$

$$Ssk = \frac{1}{Sq^3}\frac{1}{A}\iint_A z^3(x,y)dxdy$$

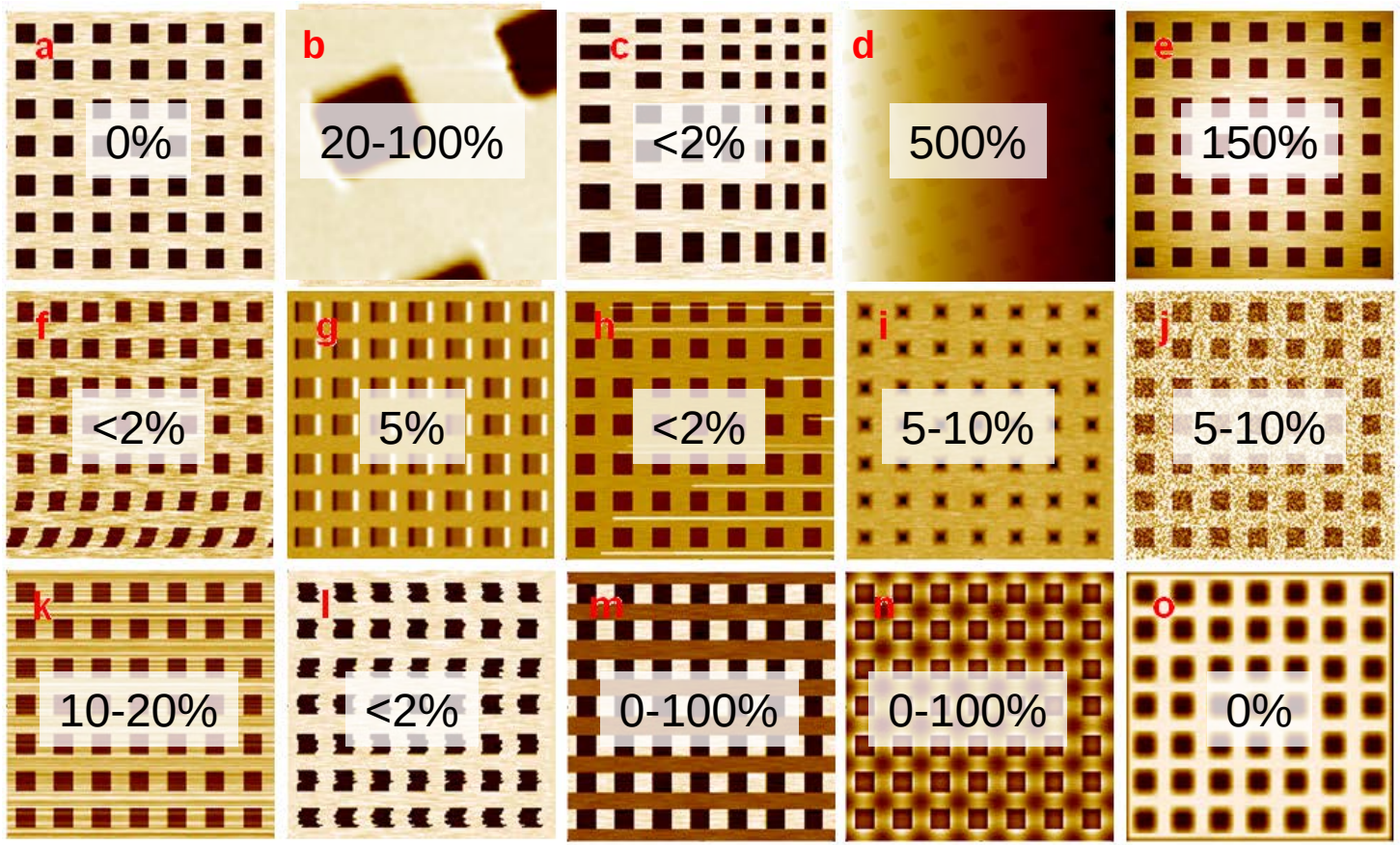


$$Sku = \frac{1}{Sq^4}\frac{1}{A}\iint_A z^4(x,y)dxdy$$



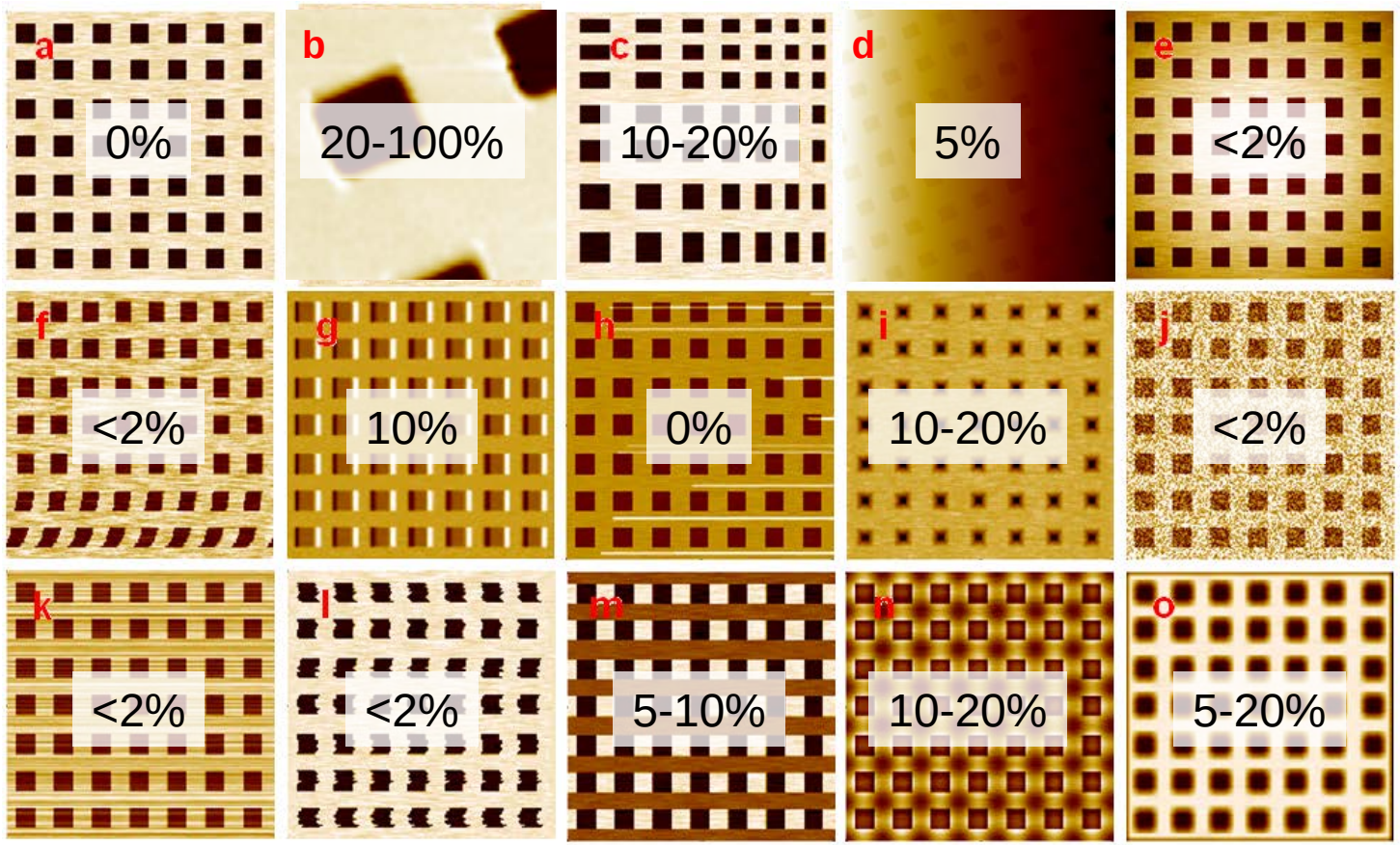
However all of these parameters are heavily affected by SPM artifacts

$$Sa = \frac{1}{A}\iint_A |z(x,y)|dxdy$$



However all of these parameters are heavily affected by SPM artifacts

Nanoparticles diameter:



SPM analyses approach

A reasonable approach should start from 3 questions:

- Measurement: «What is the ideal trade off between the size of the features of interest, the parameters I will calculate and the time needed for the scan?»

- Calibration: «Is there any possibility of calibrating at that scanning conditions?»

- Parameters estimation: «Is there a standard method for data processing (filtering and parameter calculation)?»

SPM analyses approach

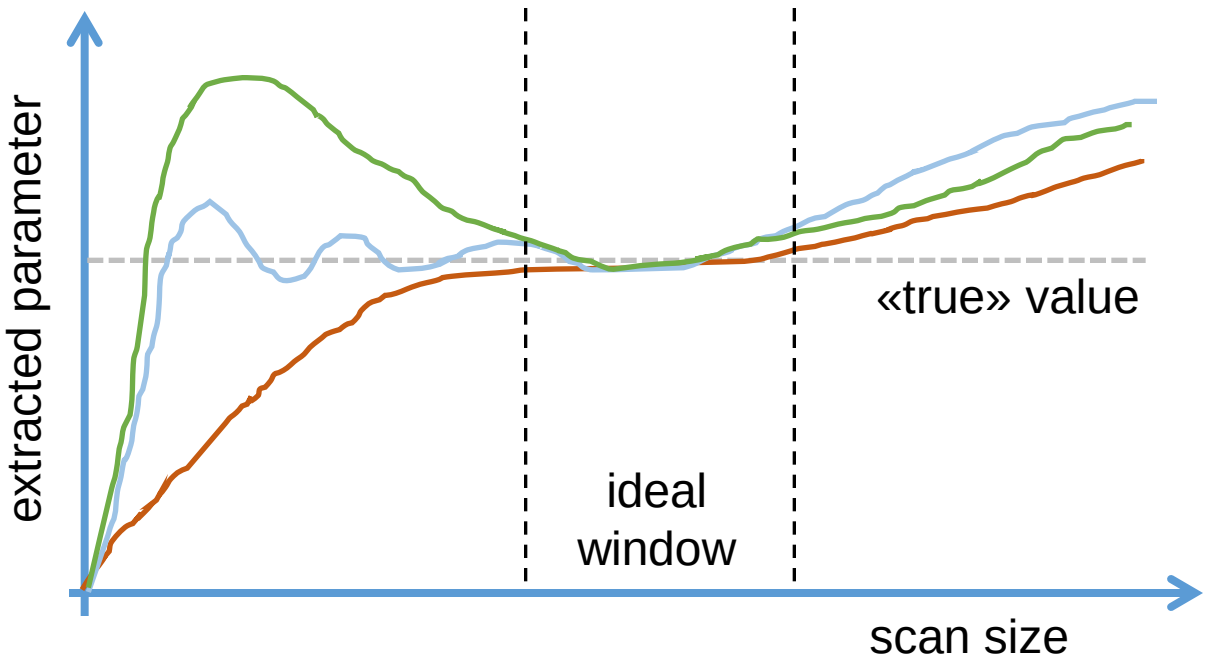
A reasonable approach should start from 3 questions:

- Measurement: «What is the ideal trade off between the size of the features of interest, the parameters I will calculate and the time needed for the scan?»

- an old rule of metrology says that at least 5 units of the feature of ineterest have to be present in the analysed area

- the parameter to be calculated should have reached a convergence

- the longer is the time, the higher is the possibility of distortions entering the masurement

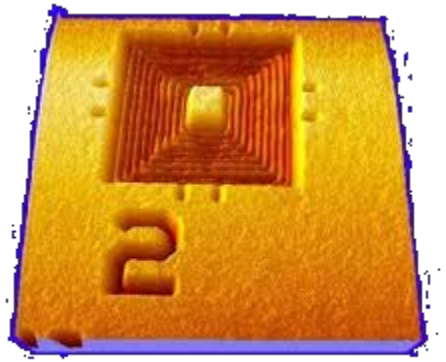


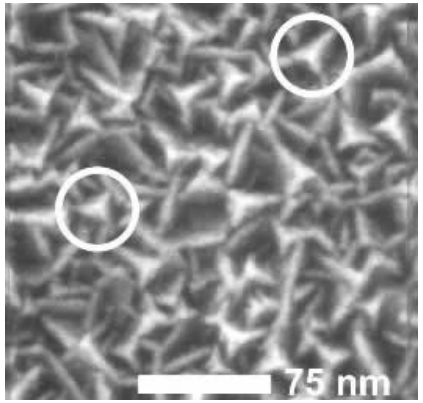
SPM analyses approach

A reasonable approach should start from 3 questions:

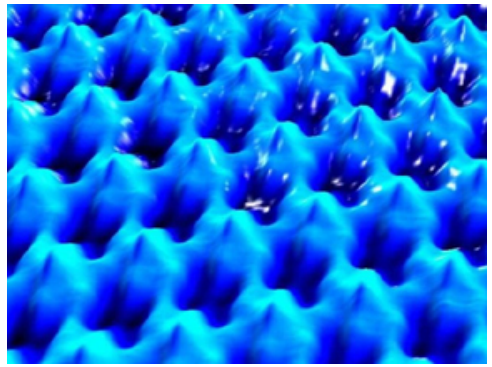
- Calibration: «Is there any possibility of calibrating at that scanning conditions?»

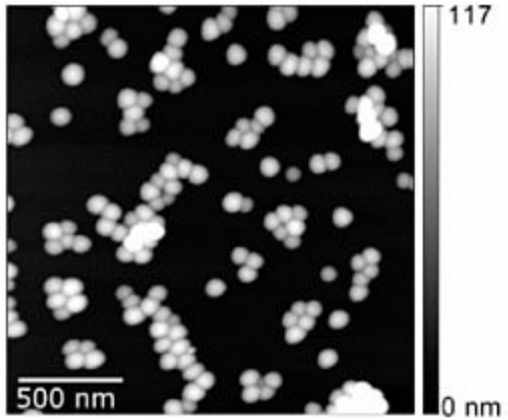
- the calibration standard and scanning procedure should be as close as possible to the measurement to be done (substitution method)





- multiple calibration artifacts are needed resembling standard scanning conditions (generic calibration)



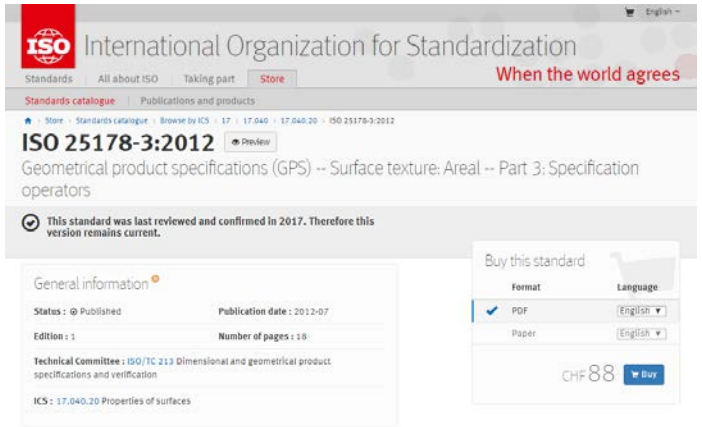


- calibration is part of the emasurement, and can introduce uncertainty (e.g. tip wear)

SPM analyses approach

A reasonable approach should start from 3 questions:

- Parameters estimation: «Is there a standard method for data processing (filtering and parameter calculation)?»



- if yes, that's good

- if not, I need to pay attention in order to make the post processing operation as much repeatable as possible, but in this case another dilemma arises
  - Higher filtering=less noise
  - Lower filtering=higher repeatability

To conclude

The measurement is not the end of the story… it is just the begin

Standard procedures are needed for different SPM tasks

Scientific research should also consider clear reporting on

- Measuring procedures
- Post processing procedures
- Parameters estimation
- Uncertainty estimation

THANK YOU

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References:

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