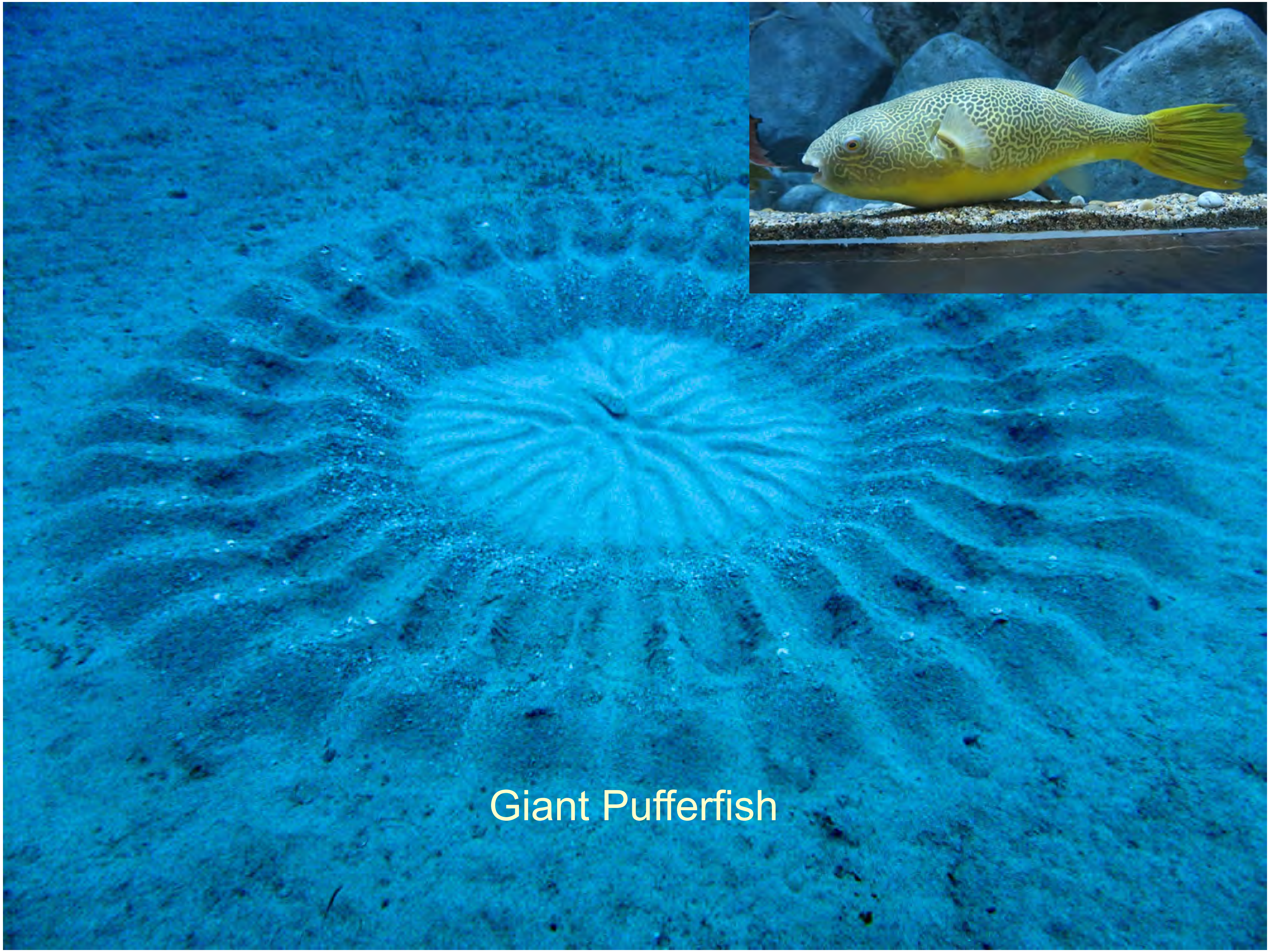
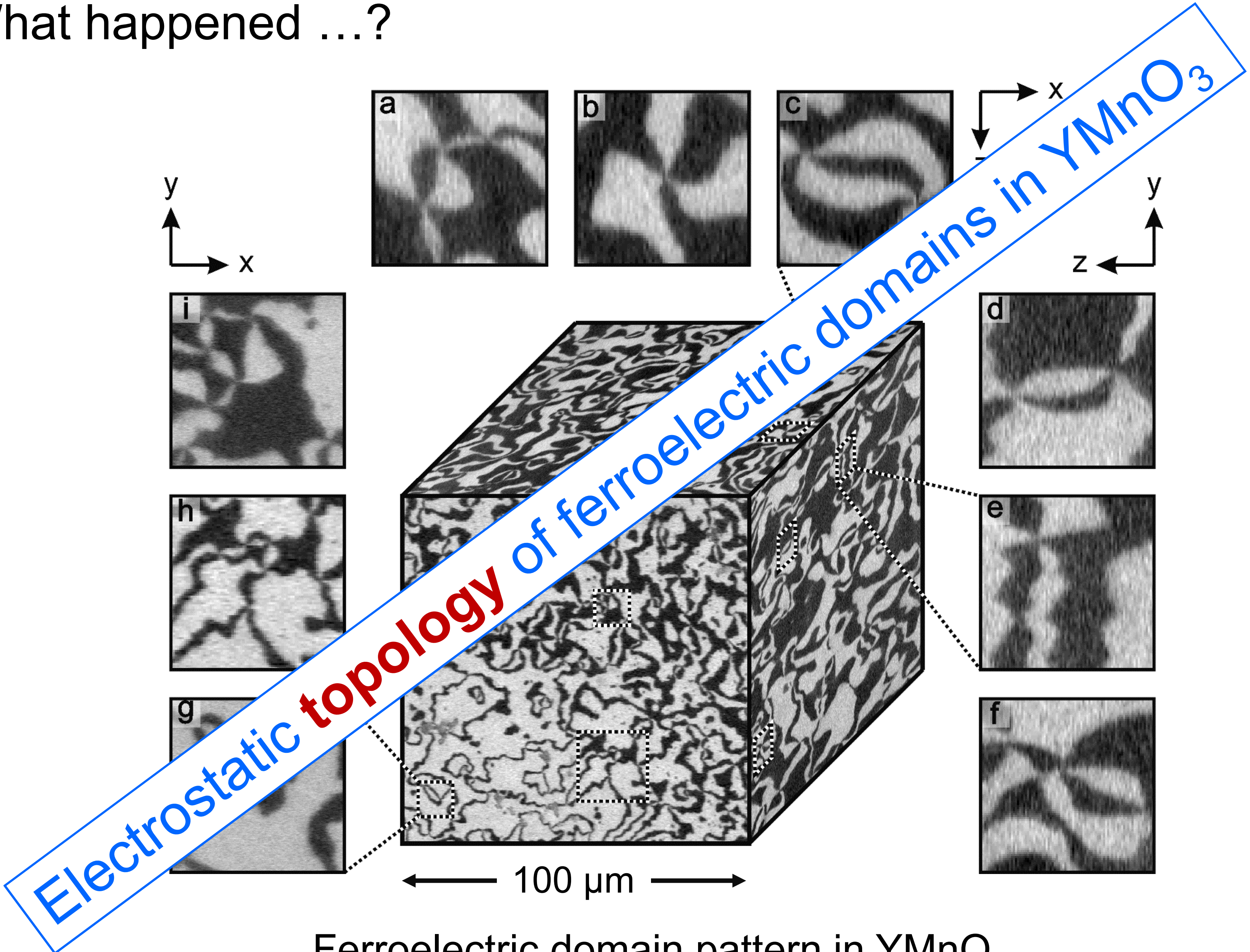

Giant Pufferfish

What happened …?



Ferroelectric domain pattern in $YMnO_3$

APL 97, 012904 (2010)

What to do?

From "plug & play"
towards a better understanding
of scanning probe microscopy imaging

Elisabeth Soergel

University of Bonn

… something else …

27. July 2017

Kunsthistorisches Museum Wien.

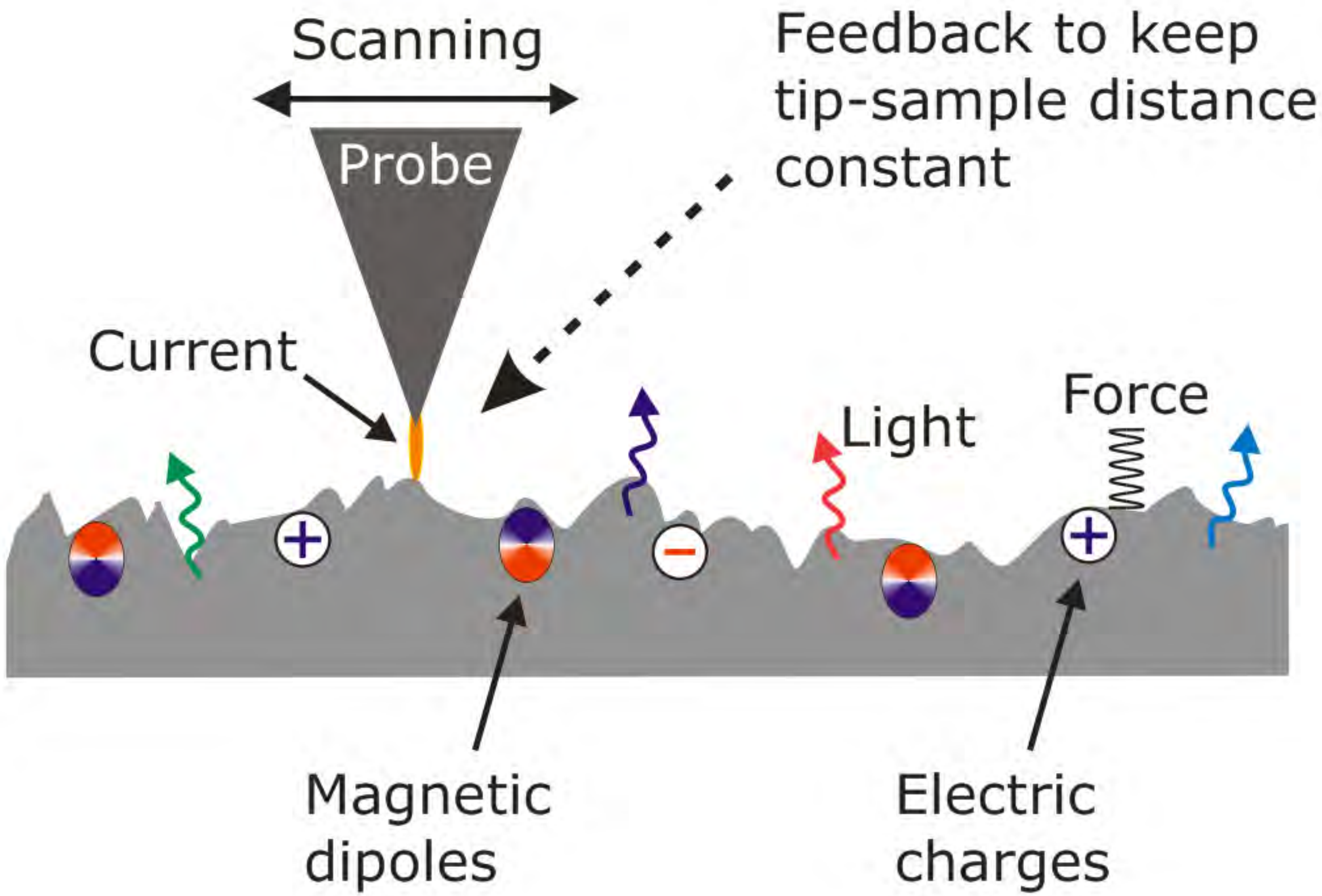
Jan Brueghel the Elder (1568 - 1625**)**
Flowers in a Wooden Vessel, 1603

Scanning Probe Microscopy
a bouquet of possibilities
for investigating samples

Lithium Niobate
a rich playground
for fascinating science

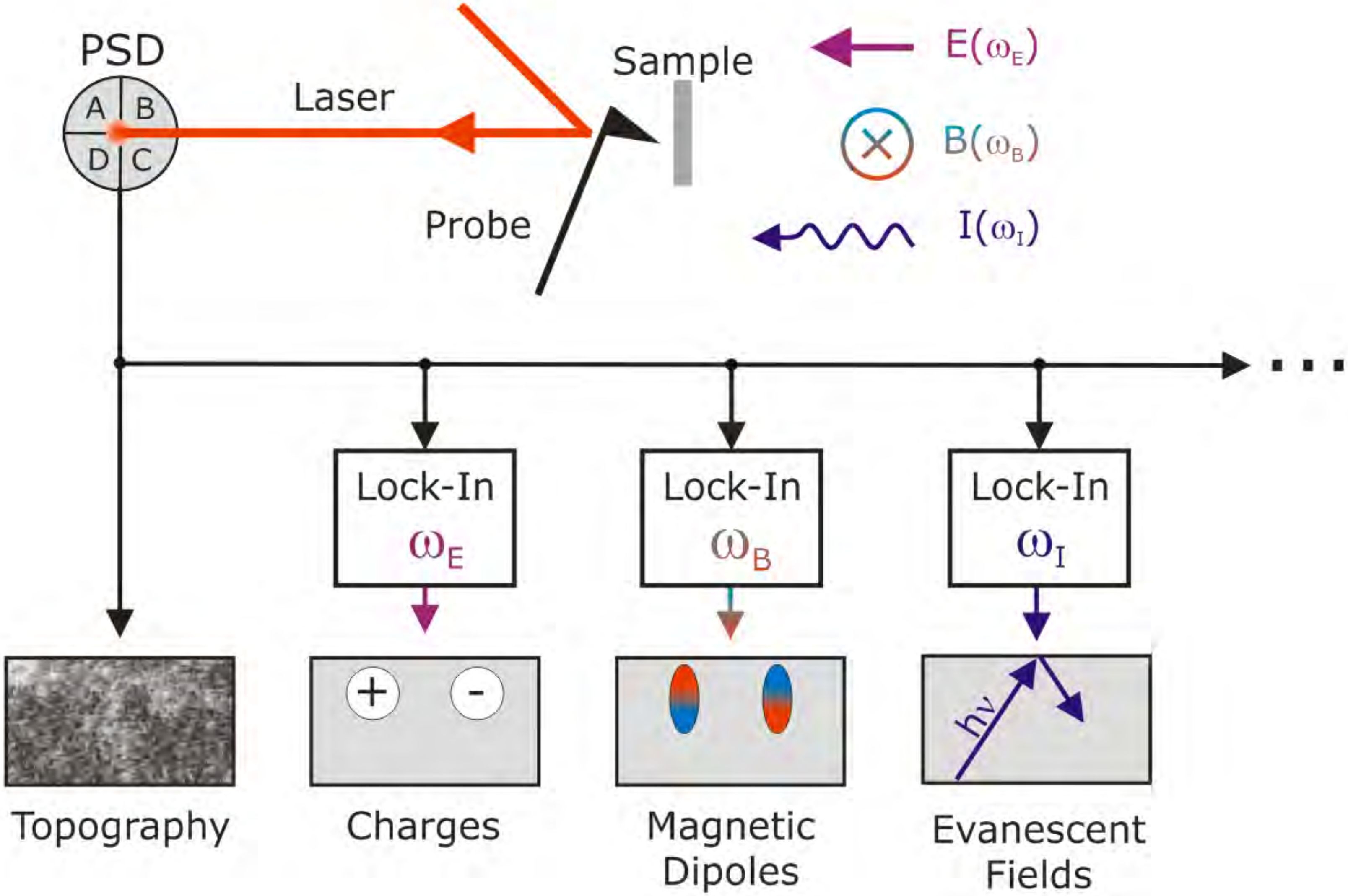
Scanning Probe Microscopy (SFM)

A bouquet of possibilties for investigating samples



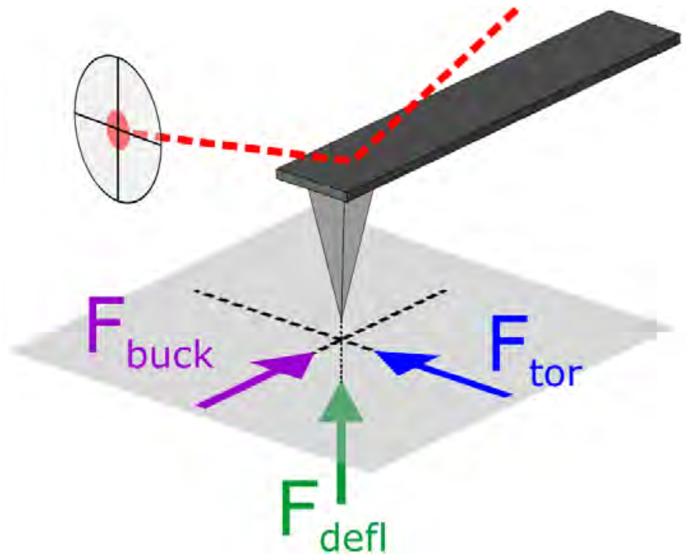
Scanning Force Microscopy (SFM)

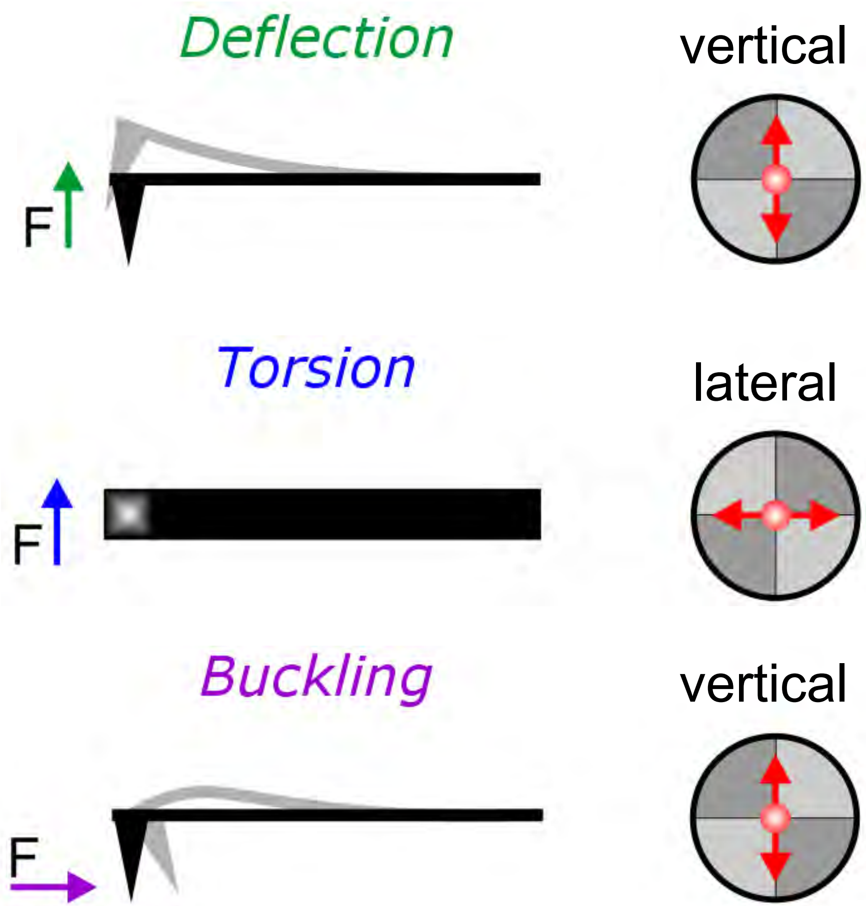
Basics



Scanning Force Microscopy (SFM)

Cantilever movement readout





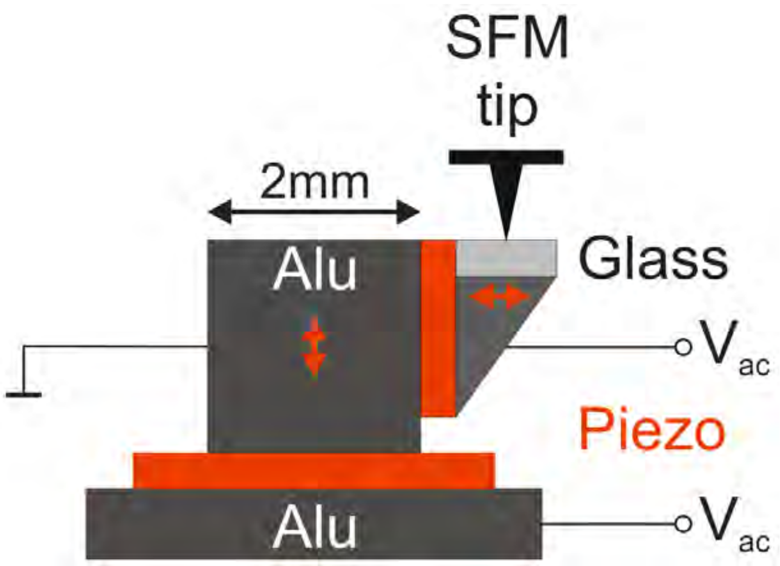
Vertical read-out channel:
Deflection & Buckling

Lateral read-out channel:
Torsion

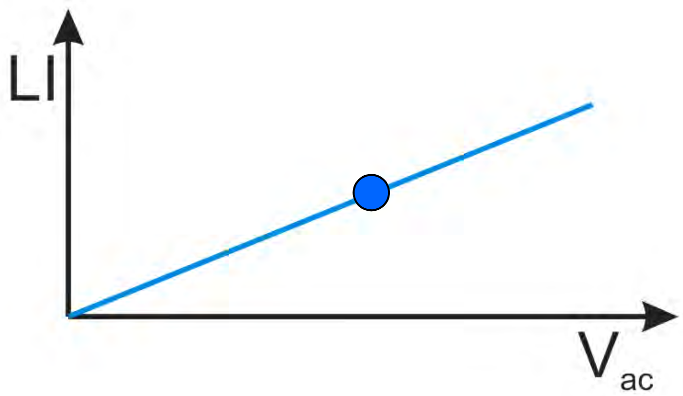
Torsion ←→ Bucking:
Rotation of the sample by 90°

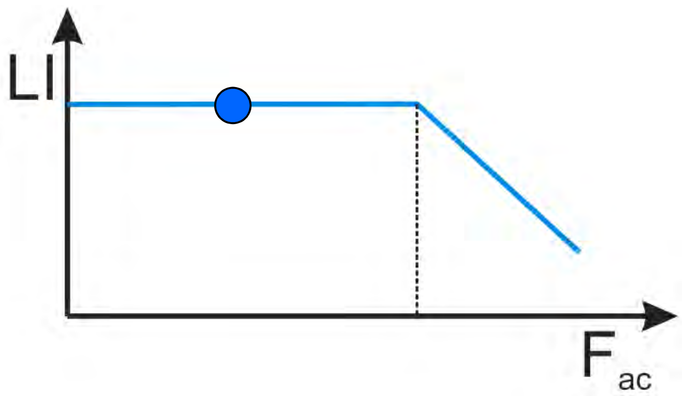
Scanning Force Microscopy (SFM)

Calibration of in-plane displacements (torsion & buckling)



- Use two identical piezoplates
- Use calibrated height scanner to calibrate the piezoplate
- Find appropriate amplitude & frequency ($2V_{pp}$ / 10kHz)

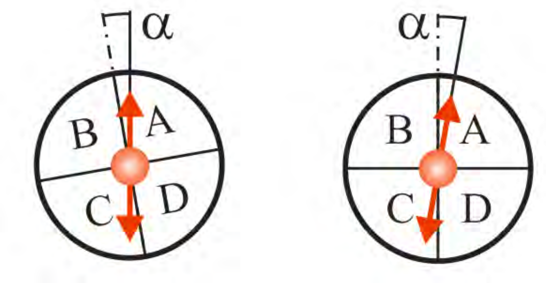




Scanning Force Microscopy (SFM)

Crosstalk between the signals?

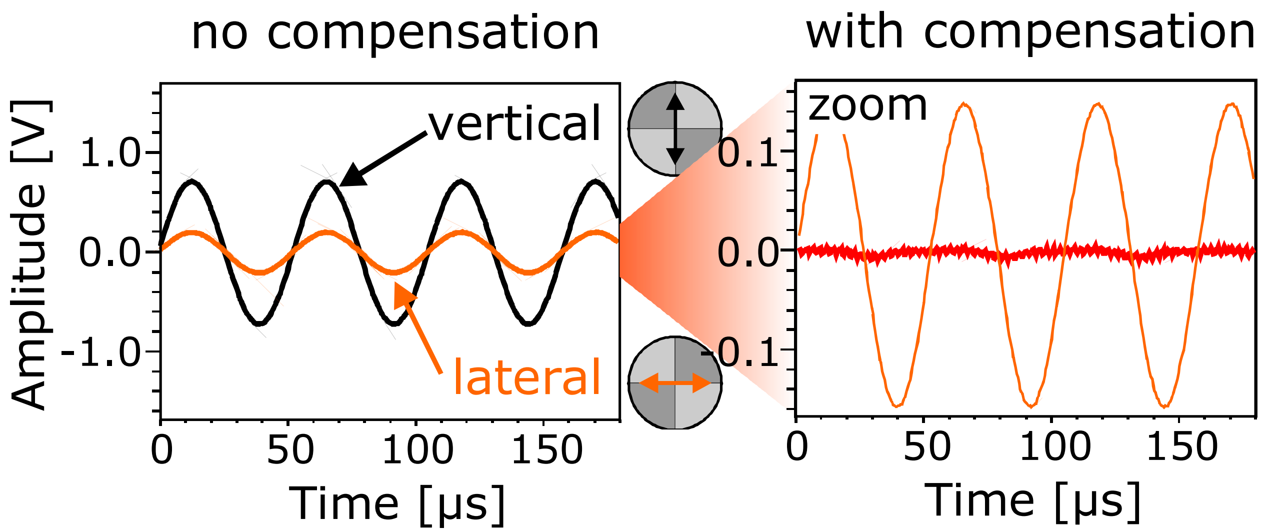
Misalignment
→ Crosstalk-compensator



Mechanical (sample topography)
→ Slow scanning

Electronical (manufacturer's fault)
→ Better shielding

Excite the cantilever at its resonance frequency

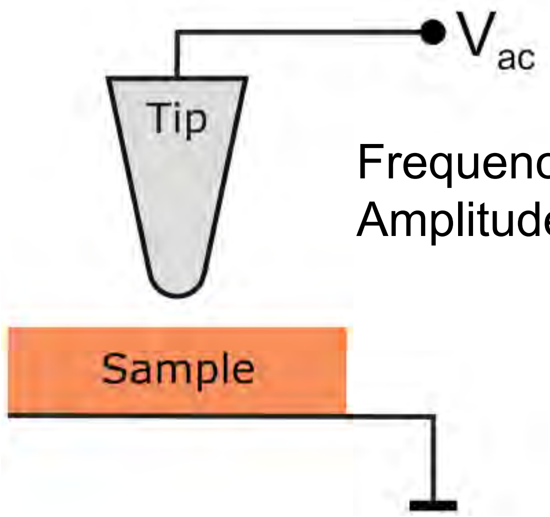


"phantom"
lateral signal
< 1%

*RSI **78**, 016101 2007*

Scanning Force Microscopy (SFM)

Electrostatic Force Microscopy (EFM)



Frequency: few 10 kHz
Amplitude: few V

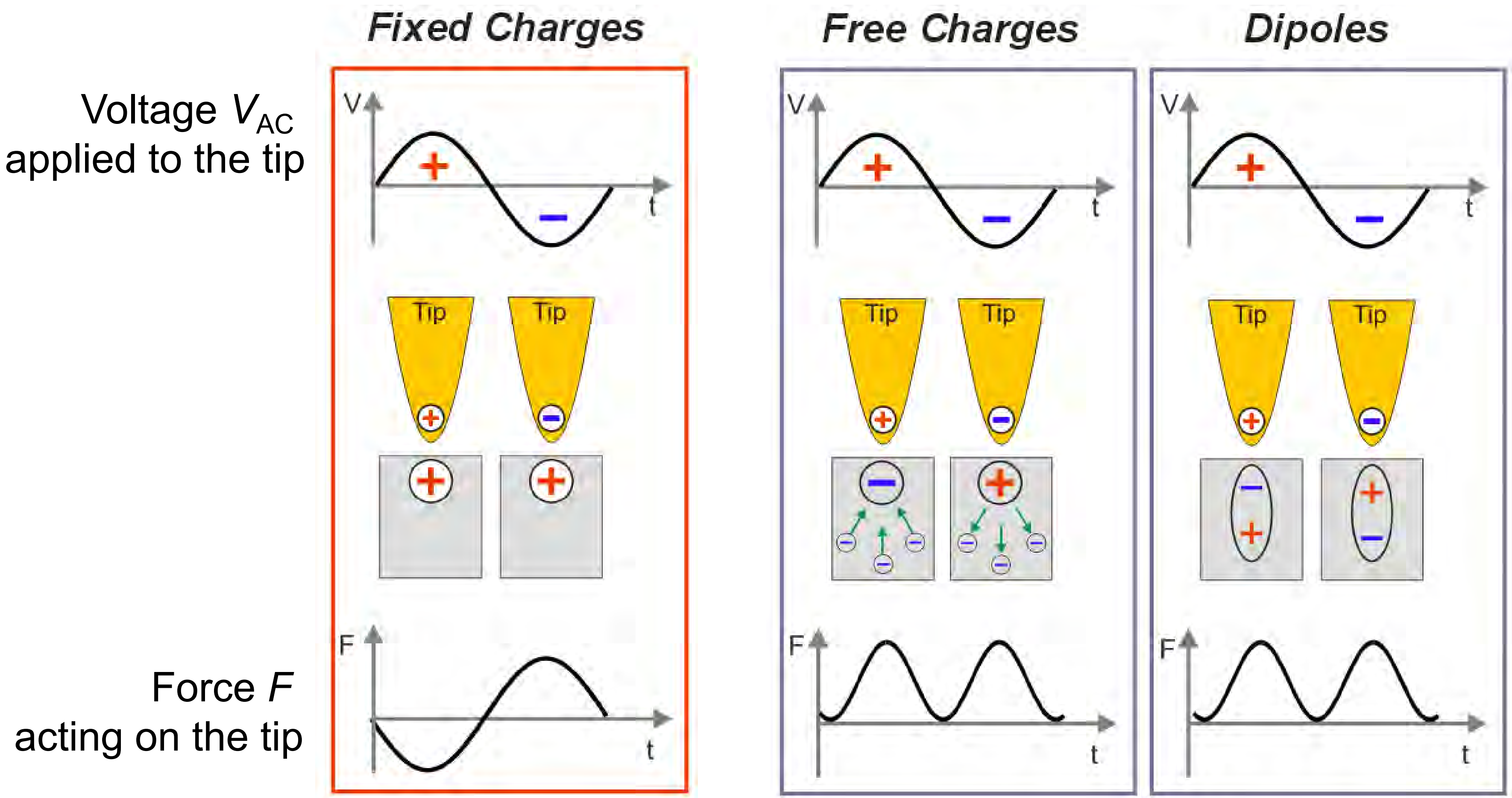
SFM in non-contact mode:
tip-sample distance z few nm

Lateral resolution $\propto r$ and z

Sensitivity:
Single(!) charge detection (fN)

Scanning Force Microscopy (SFM)

Electrostatic Force Microscopy (EFM)



$F = F(\omega)$

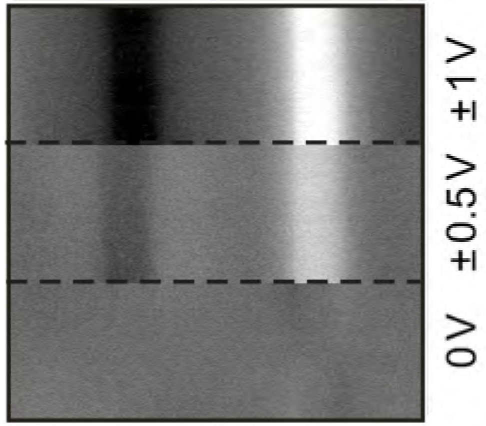
$$= \frac{q\,C\,V_{AC}}{4\pi\varepsilon_0 z^2}\sin\omega t$$

$F = F(2\omega)$

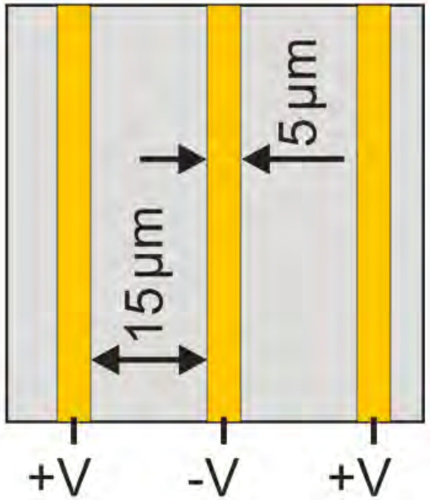
$$= \frac{1}{2}\frac{dC}{dz}V_{AC}^2\cos(2\omega t)$$

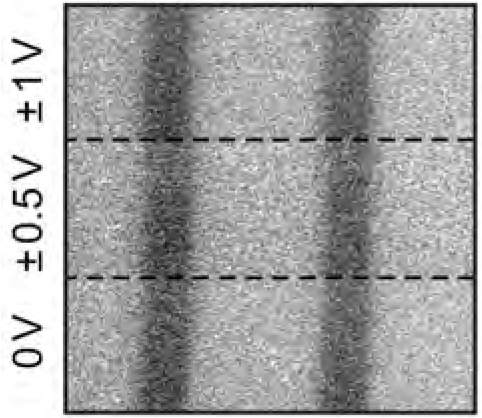
Scanning Force Microscopy (SFM)

Electrostatic Force Microscopy (EFM)

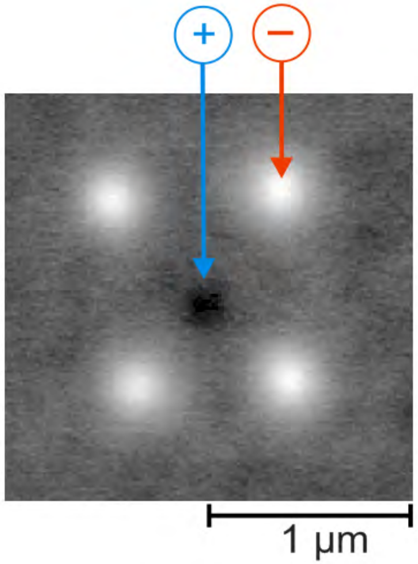


ω-signal



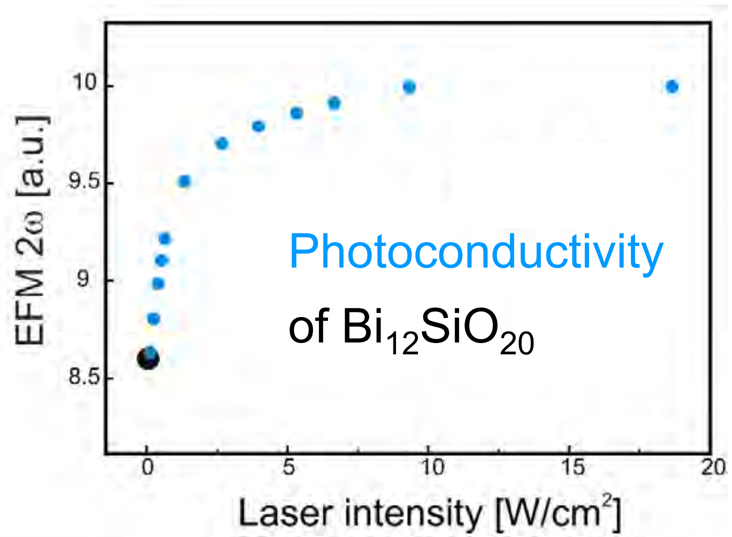


2ω-signal



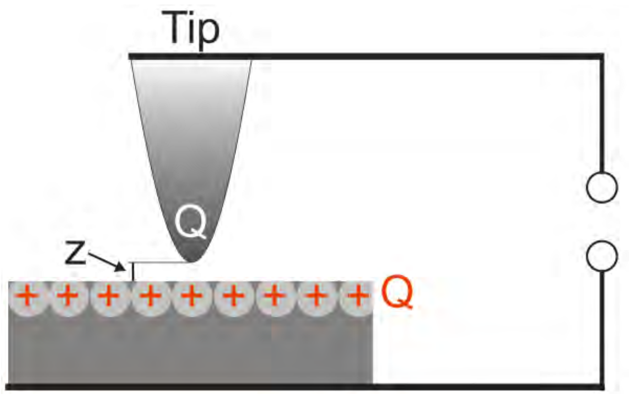
Corona discharges
(± 10 V, 10 ms)
on $Bi_{12}SiO_{20}$

500 nm tick
dielectric layer



Surface Charge Density

Determination of the Surface Charge Density (SCM)



Probe can no longer be approximated by a sphere

→ Cone, cantilever, …
have to be taken into account

Need of a calibration sample

= a sample with known surface charge density
→ impossible

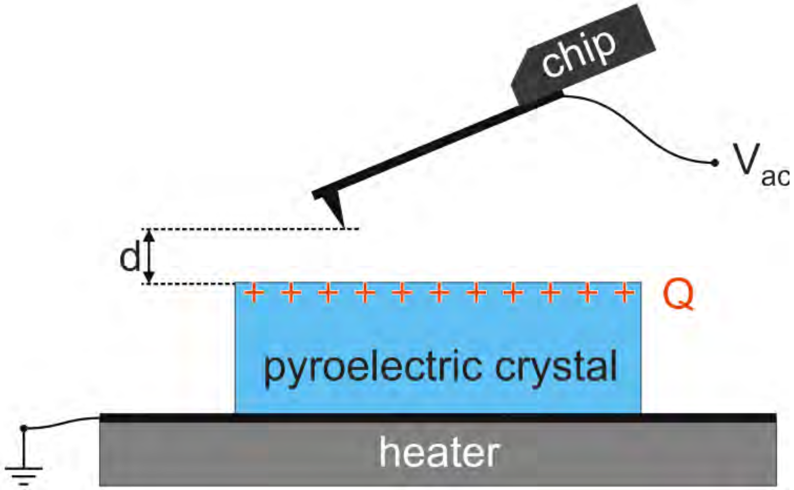
= a sample with known, controllable change of the surface charge density
→ pyroelectric crystal

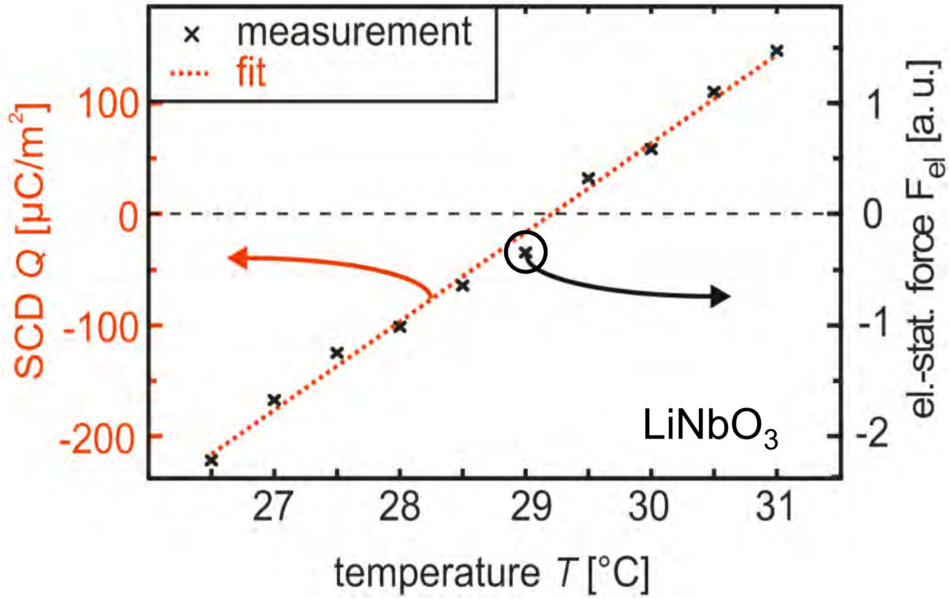
With a known pyroelectric coefficient dP_s/dT

a controlled change ΔT leads to a known $\Delta Q_{surface}$

Surface Charge Density

Calibration & Results for the SCD on $LiNbO_3$





$LiNbO_3$: dP_s/dT = 80 x 10^6 C/Km²

$LiTaO_3$: dP_s/dT = 190 x 10^6 C/Km²

Temp. steps ΔT: 0.5 to 2.5 K

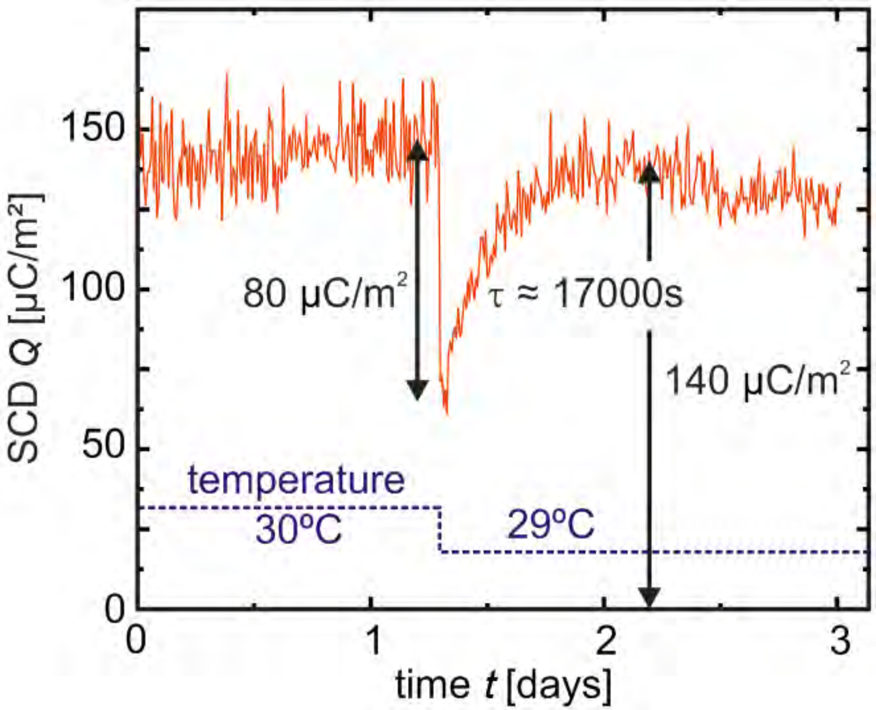
stabilized Peltier [ΔT in min]
heating resistor [ΔT in s]

[ΔT in min] or [ΔT in s] yielded the same results.

Measurements with $LiTaO_3$ were consistent.

Surface Charge Density

Long-Term Measurement of the SCD on $LiNbO_3$



- Surface charge recovers
- Recovering takes half a day
  [τ = 1.7 x 10^4 s]
- 0.5%Fe:$LiNbO_3$ shows fast recovery
  [σ = 10^{-9} $(\Omega cm)^{-1}$]
- Dry atmosphere
  did not alter the result

Maxwell relaxation time: $$\tau = \frac{\varepsilon \cdot \varepsilon_0}{\sigma} \rightarrow \tau = 10^3 - 10^5 \text{ s}$$

$$\varepsilon = 28$$
$$\sigma = 10^{-16} - 10^{-18} (\Omega \text{ cm})^{-1}$$

*APL **95**, 232906 (2009)*

Ferroelectric Workhorse

Lithium Niobate ($LiNbO_3$)

Ferro-electric

- Surface charge density: σ = 0.7 C/m²
- Coercive field: E_c = 20 kV/mm
- 180°-domains

Pyro-electric

ΔP_s = 80 µC/m²K

Piezo-electric

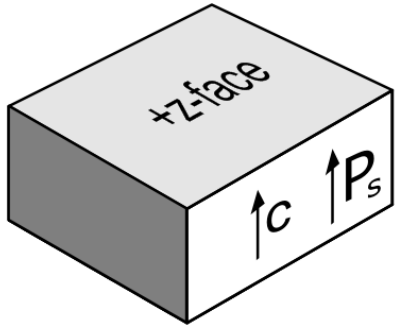
d_{11} = 0, d_{22} = 21, d_{33} = 7, d_{15} = 69, d_{31} = -1 } pm/V

Electro-optic

$\Delta n = -\frac{1}{2} n_0^3 r_{13} E$

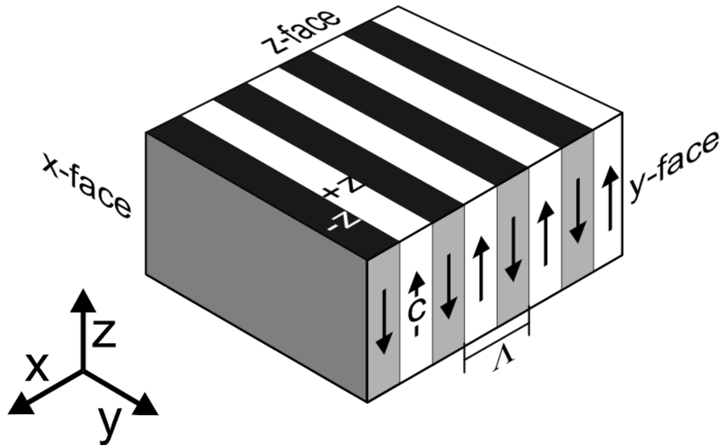
r_{13} = 11.9 pm/V

⋮ …



"z-cut"-crystal

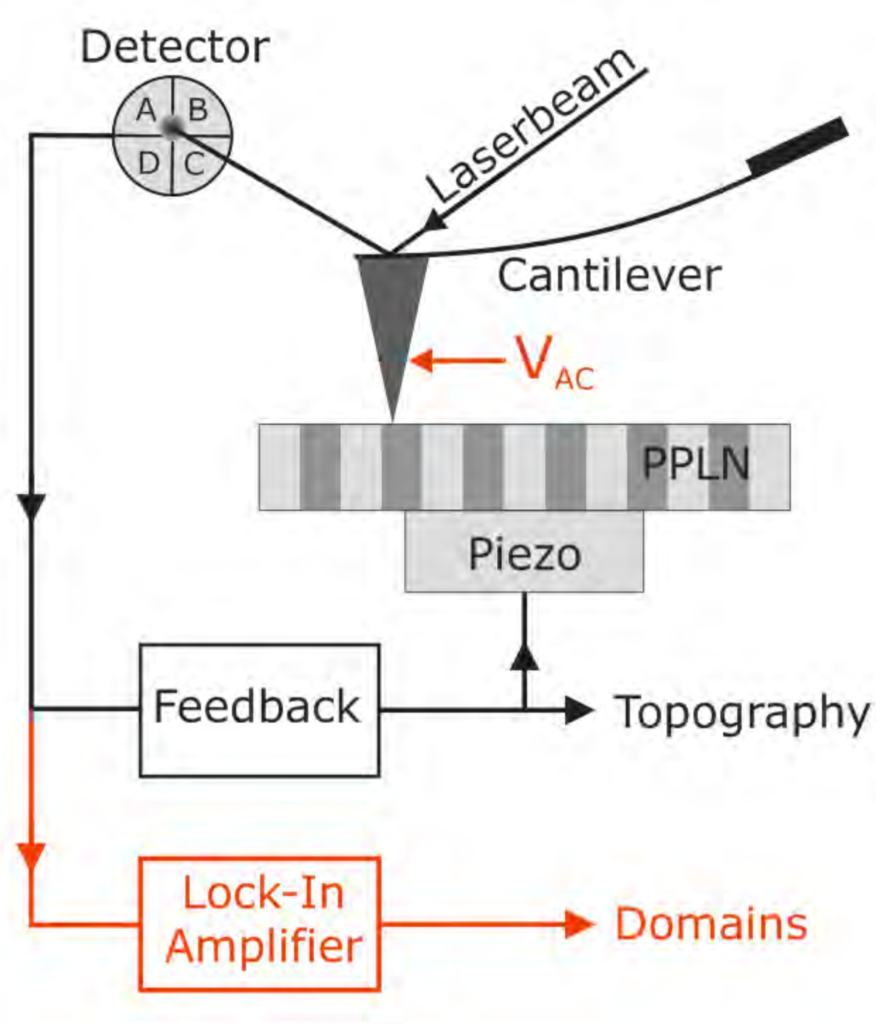
Periodically poled $LiNbO_3$ (PPLN)



PPLN (Λ: 8–30 µm)
Thickness: 500 µm

Piezoresponse Force Microscopy (PFM)

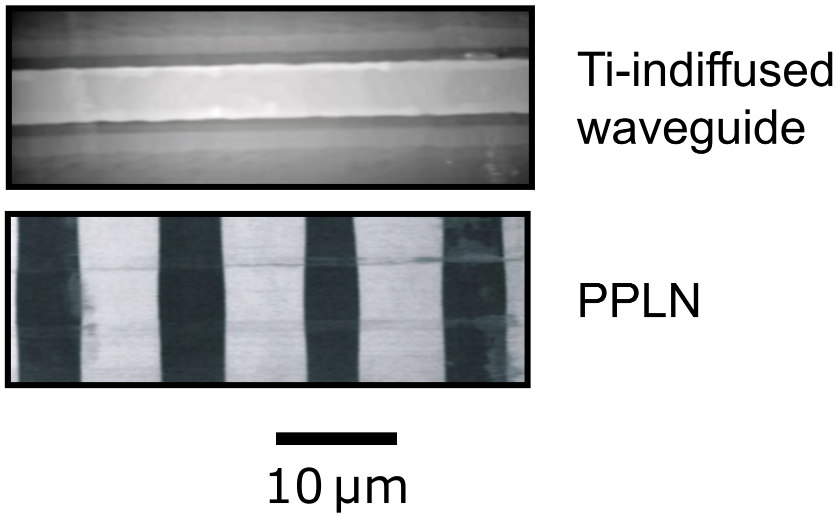
Basics



Topography and piezoresponse are readout simultaneously and independently

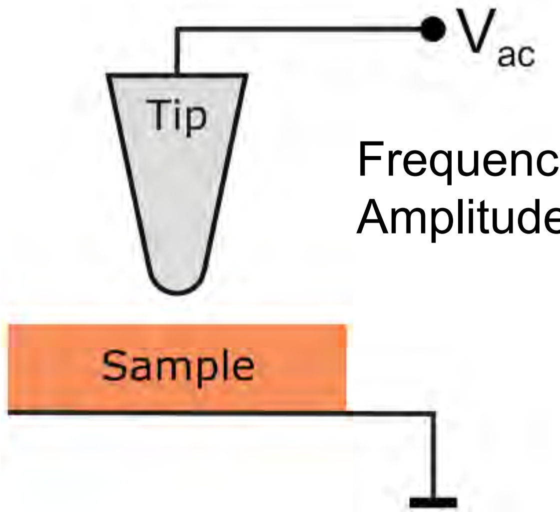
Lateral resolution ∝ tip radius
→ typically 20 nm

Sensitivity of PFM: < 0.1 pm/V



Scanning Force Microscopy (SFM)

EFM - Electrostatic Force Microscopy



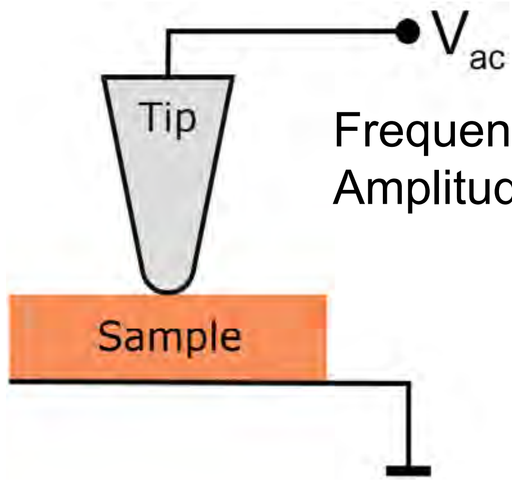
Frequency: few 10 kHz
Amplitude: few V

SFM in non-contact mode:
tip-sample distance z few nm

Lateral resolution $\propto r$ and z

Sensitivity:
Single(!) charge detection (fN)

PFM - Piezoresponse Force Microscopy



Frequency: few 10 kHz
Amplitude: few V

SFM in contact mode:
tip-sample distance $z = 0$

Lateral resolution $\propto r$

Sensitivity:
Piezoresponse of < 0.1 pm/V

PFM versus EFM

Electrostatic contribution to PFM?

- Surface charge density:

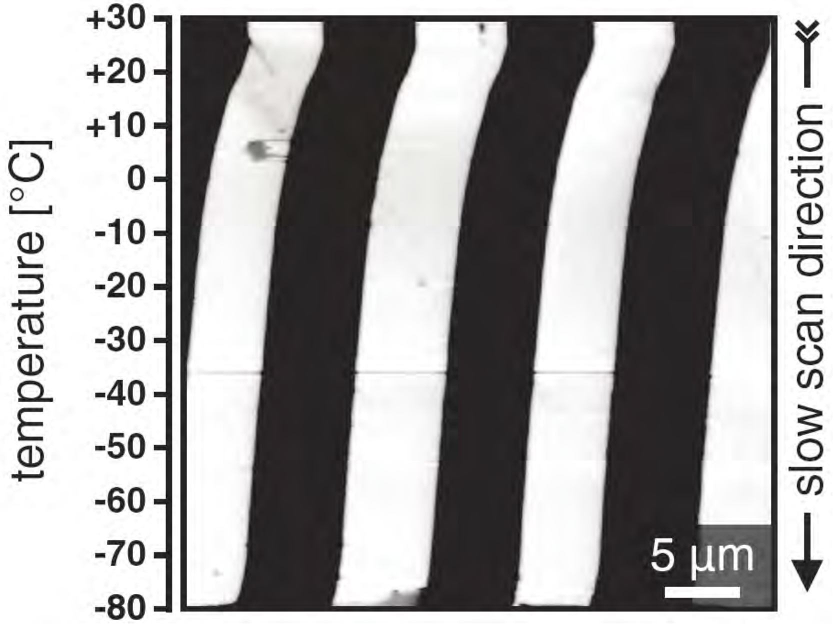
  σ = 140 µC/m²

  (experimentally determined)

- Pyroelectric coefficient:

  ΔP_s= 80 µC/m^{2}K

  (data sheet)



No change of the contrast upon cooling the crystal

→ No effect of the surface charging on the PFM signal

RSI ***84***, 043703 (2013)

PFM – Temperature-Dependent Measurements

Domain evolution in barium titanate ($BaTiO_3$)

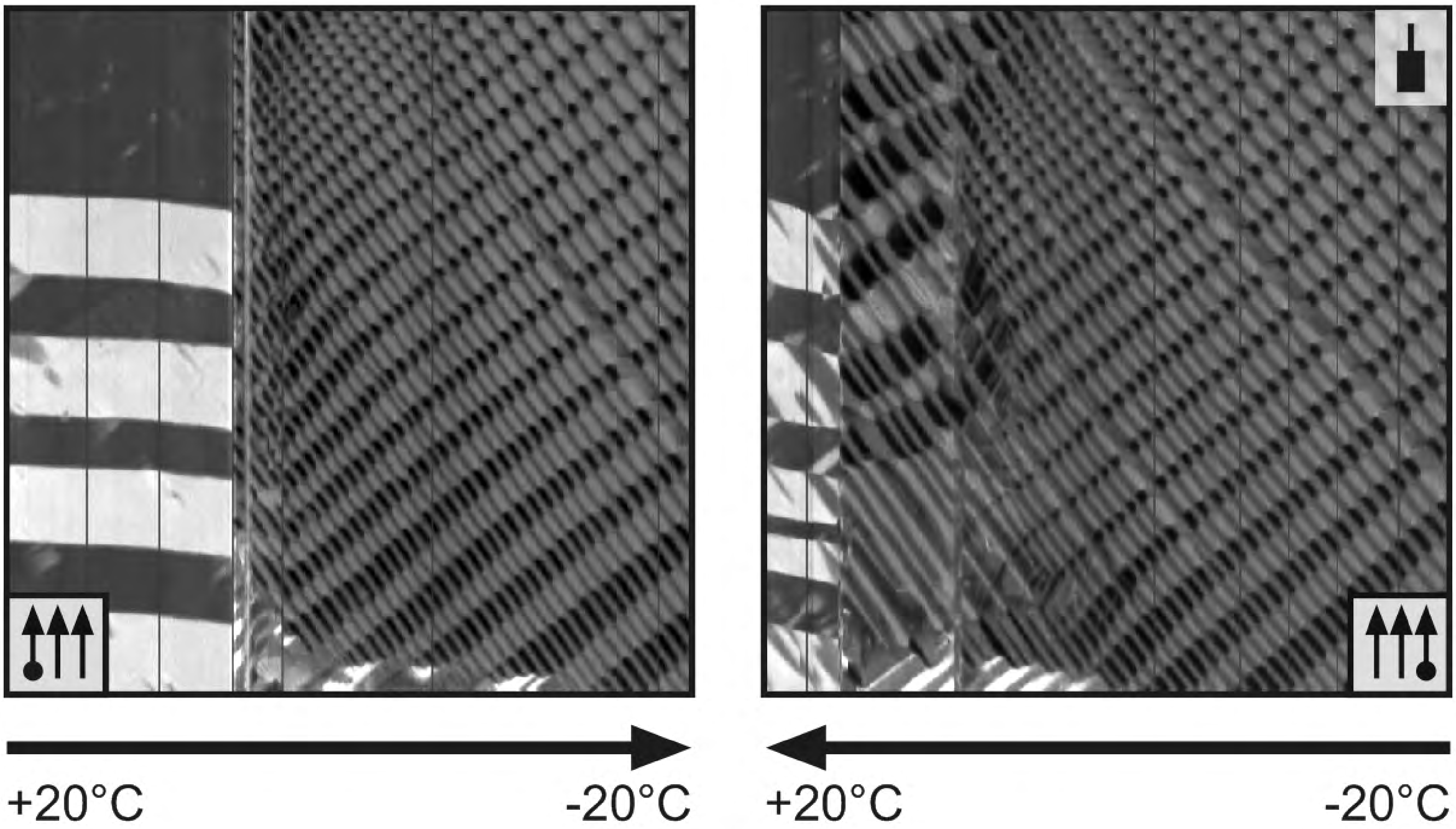
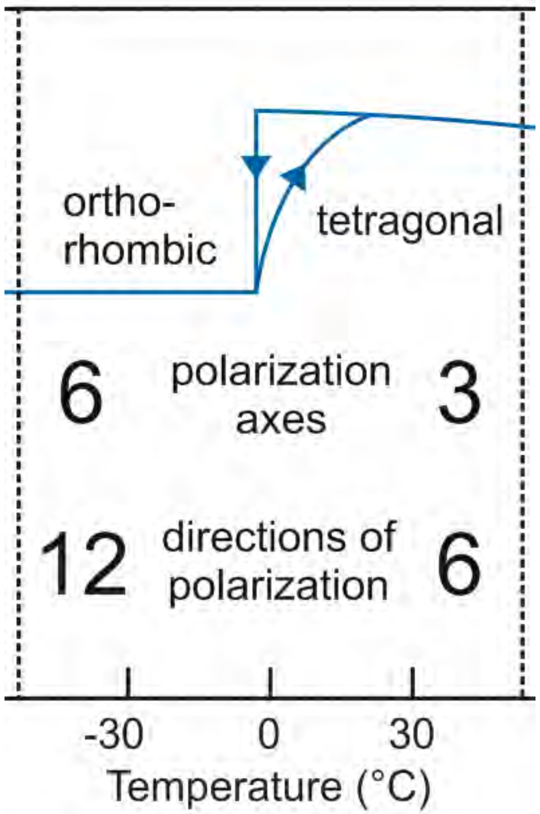


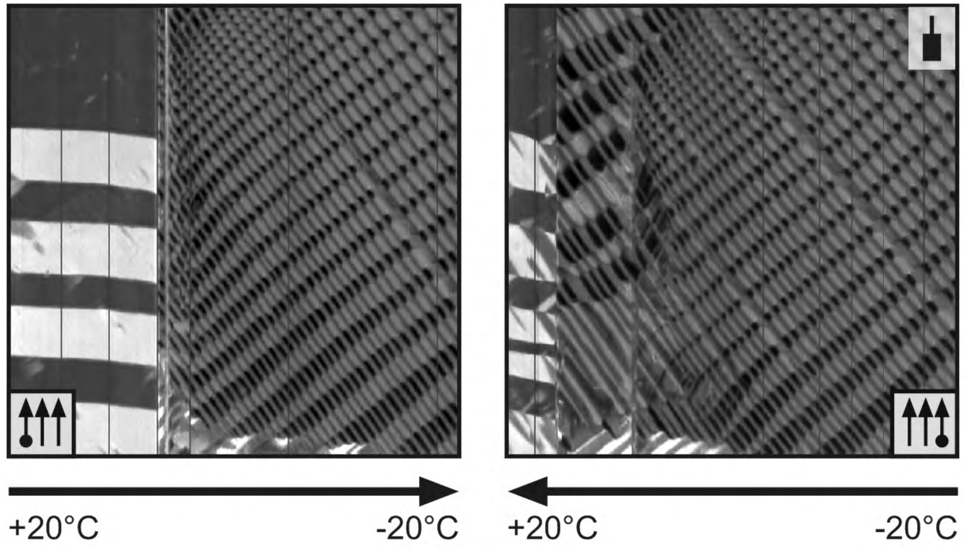
image size 20 x 20 µm²

*APL **105**, 152901 (2014)*

PFM – Temperature-Dependent Measurements

Domain evolution in barium titanate ($BaTiO_3$)





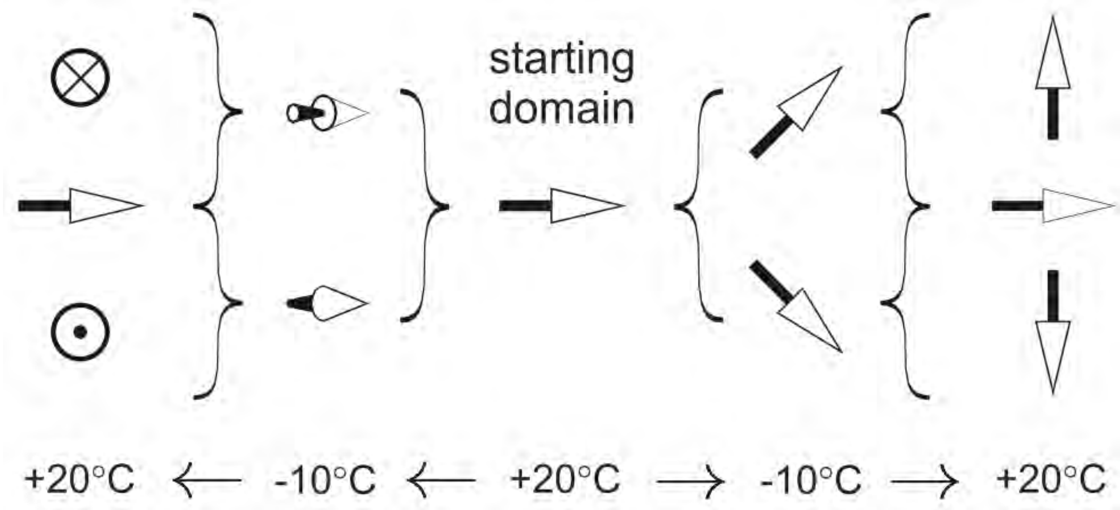
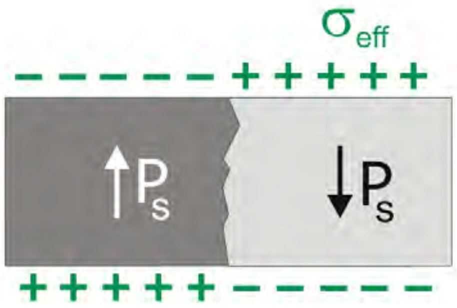


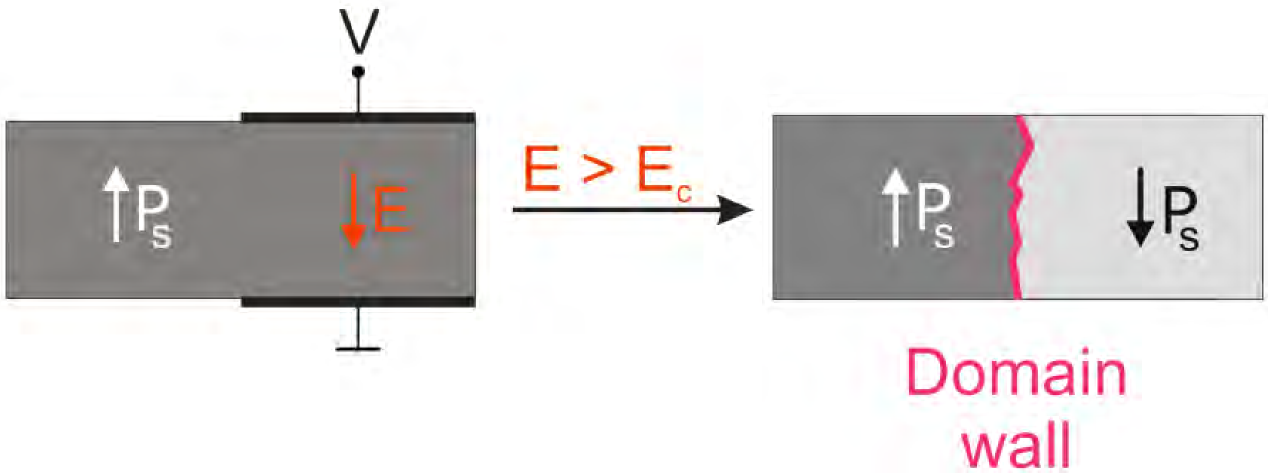
image size 20 x 20 µm²

Ferroelectrics & Scanning Force Microscopy

… using PFM and EFM

- Visualization of domain patterns
- Evolution of domain patterns across phase transitions
- Determination of the effective surface charge density





Properties of ferroelectric domain walls

Width? Few nm

And? … they are conductive!

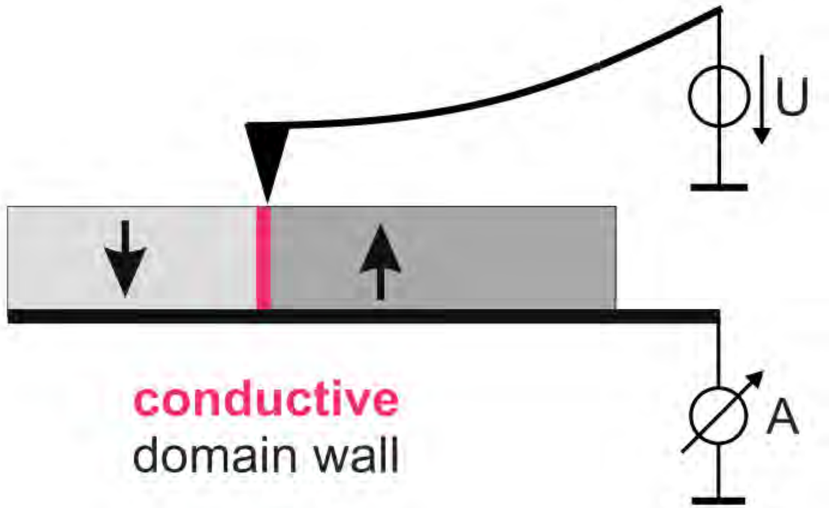
Domain Wall Conductivity

Status

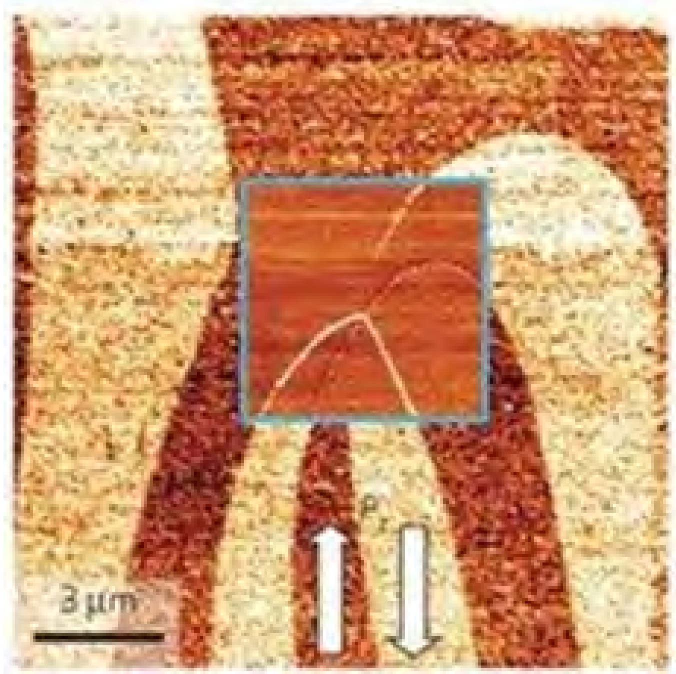
DWC has first been observed in 2009
in thin film BFO

Since then: exponential increase of research
(and publications)

Standard experimental setup: c-AFM



$ErMnO_3$

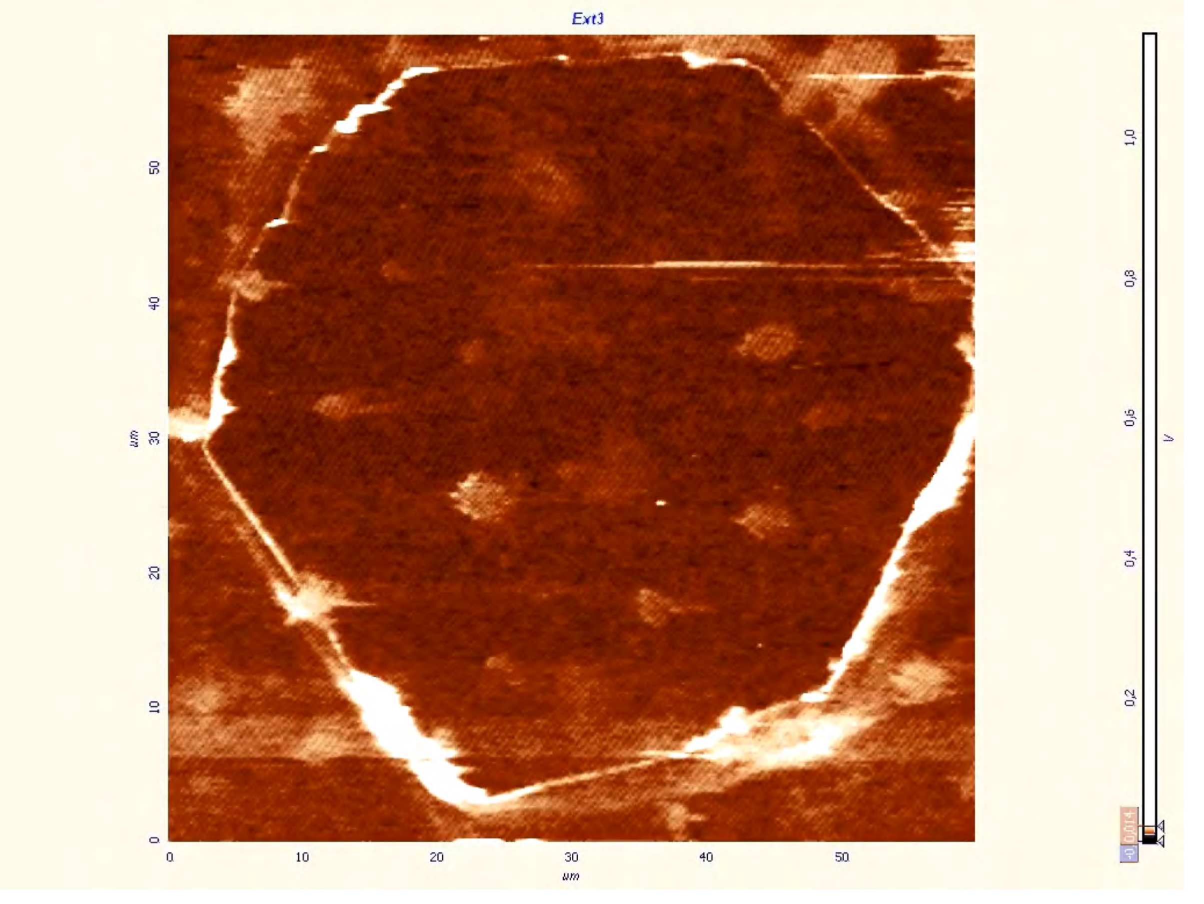


D. Meier et al.
Nature Materials 11, 284 (2012)

… conductive
= less insulating …

Domain Wall Conductivity in Lithium Niobate

Temporal evolution over 1.5 days in a 30 µm thin sample



60 x 60 µm²

Domain Wall Conductivity in Lithium Niobate

Quantitative estimates

Dark conductivity of Mg:$LiNbO_3$: $\sigma = 10^{-15}(\Omega cm)^{-1}$

Conductivity of the 1° inclined domain walls: $\sigma = 0.03\ (\Omega cm)^{-1}$
(assuming a wall width of w = 10 nm)

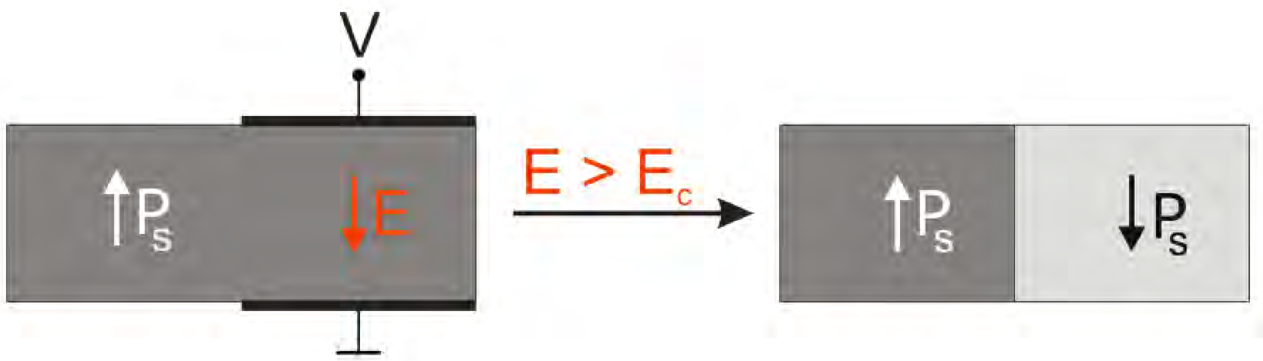
Conductivity of something real (copper): $\sigma = 10^{9}\ (\Omega cm)^{-1}$

A conductive DW is just
less insulating than its surroundings …

… but it's non the less fun!

→ Further investigations …

From ferroelectric domain patterning …



… towards nanostructuring

Domain Selective Etching

in 48% HF @RT

+z does not etch

–z etches 800 nm/h

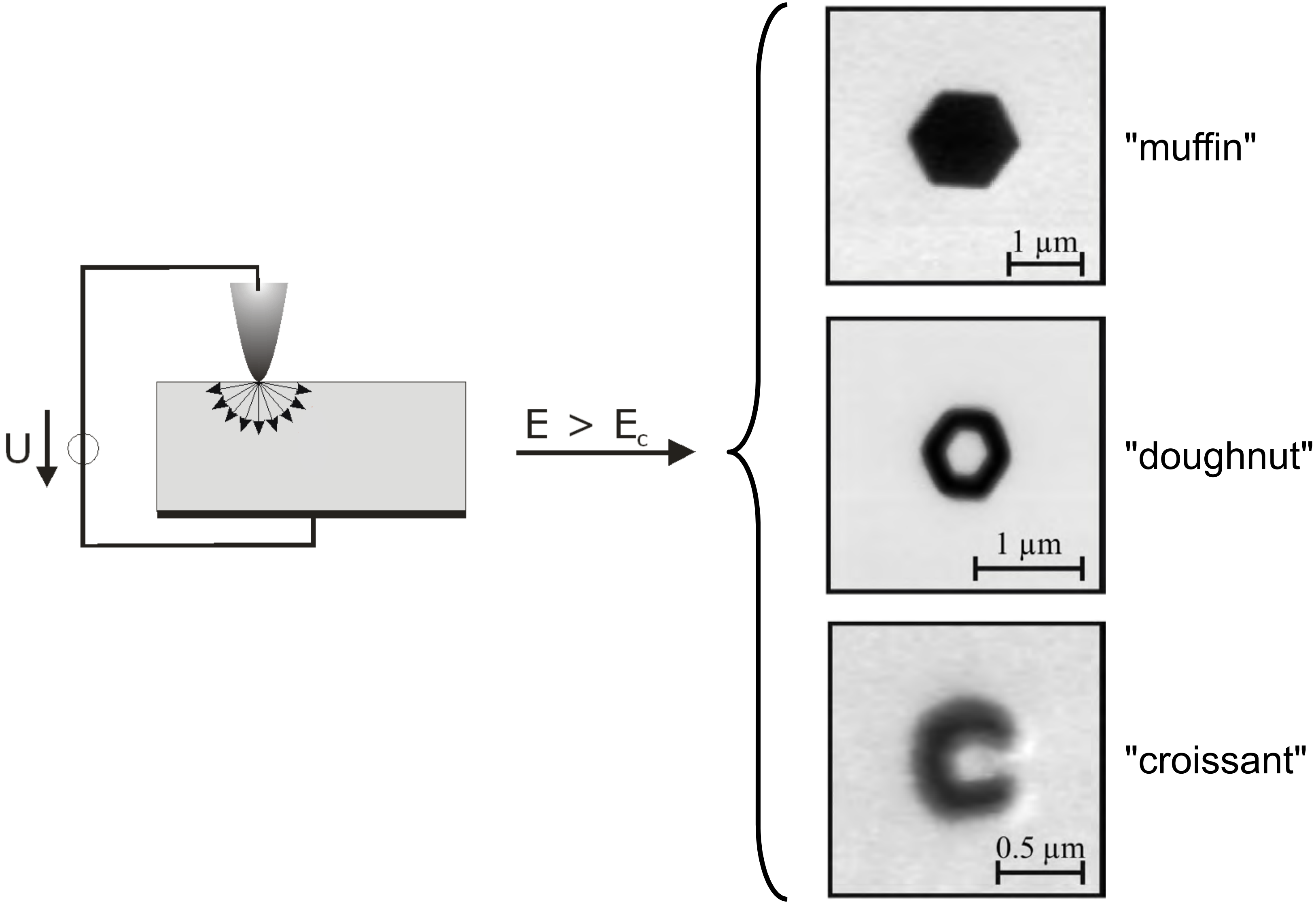
Annealing at T < Tc

→ Keeps the ferroelectric domains

→ Smoothens the surface

Poling of Domains with an SFM Tip

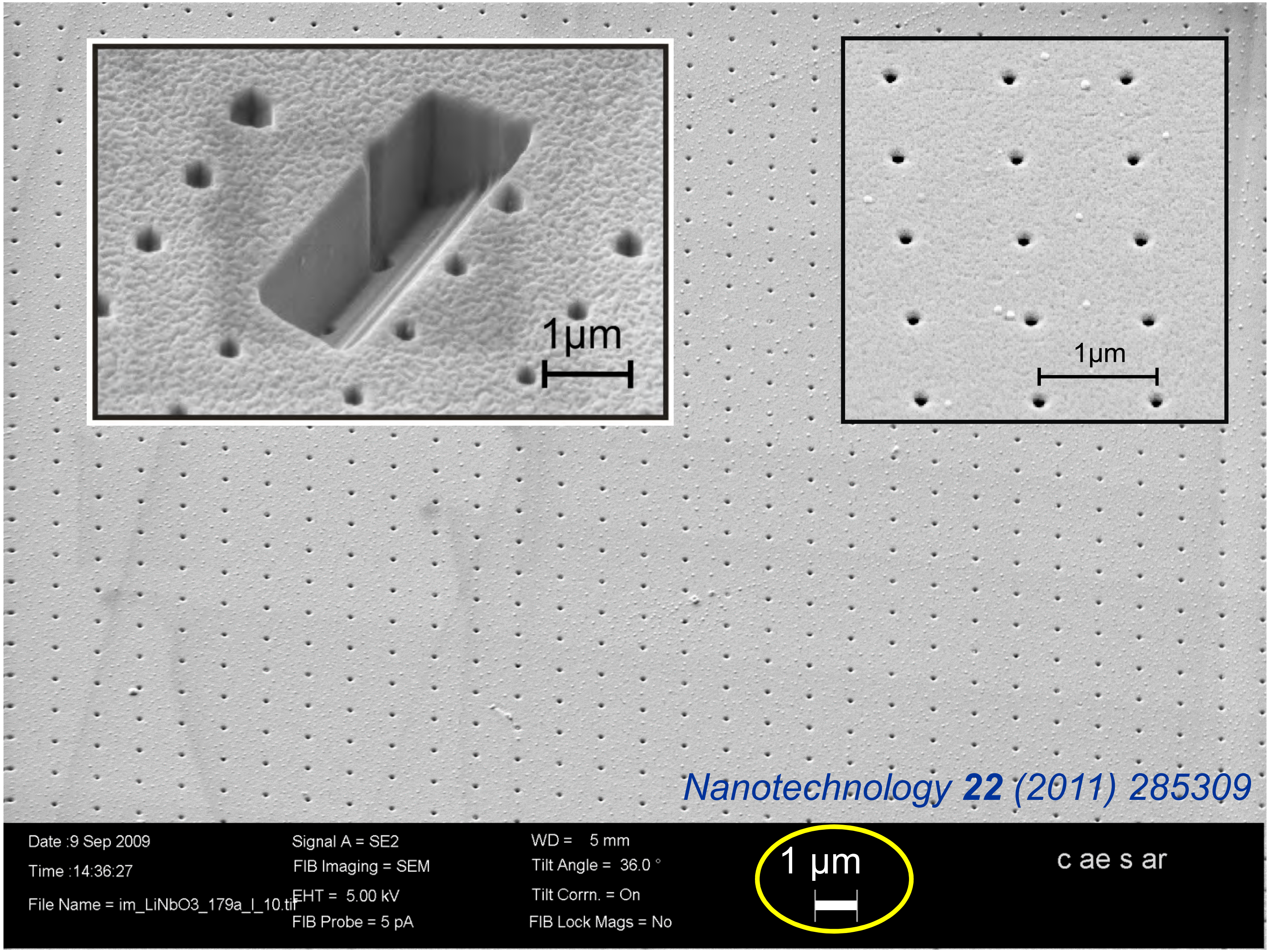
Basic principle



*JAP **110**, 052018 (2011)*

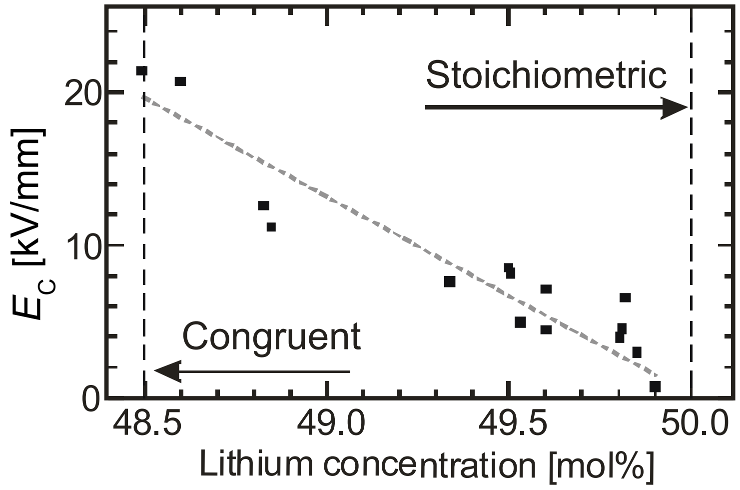
Domains in He-Implanted $LiNbO_3$ Samples

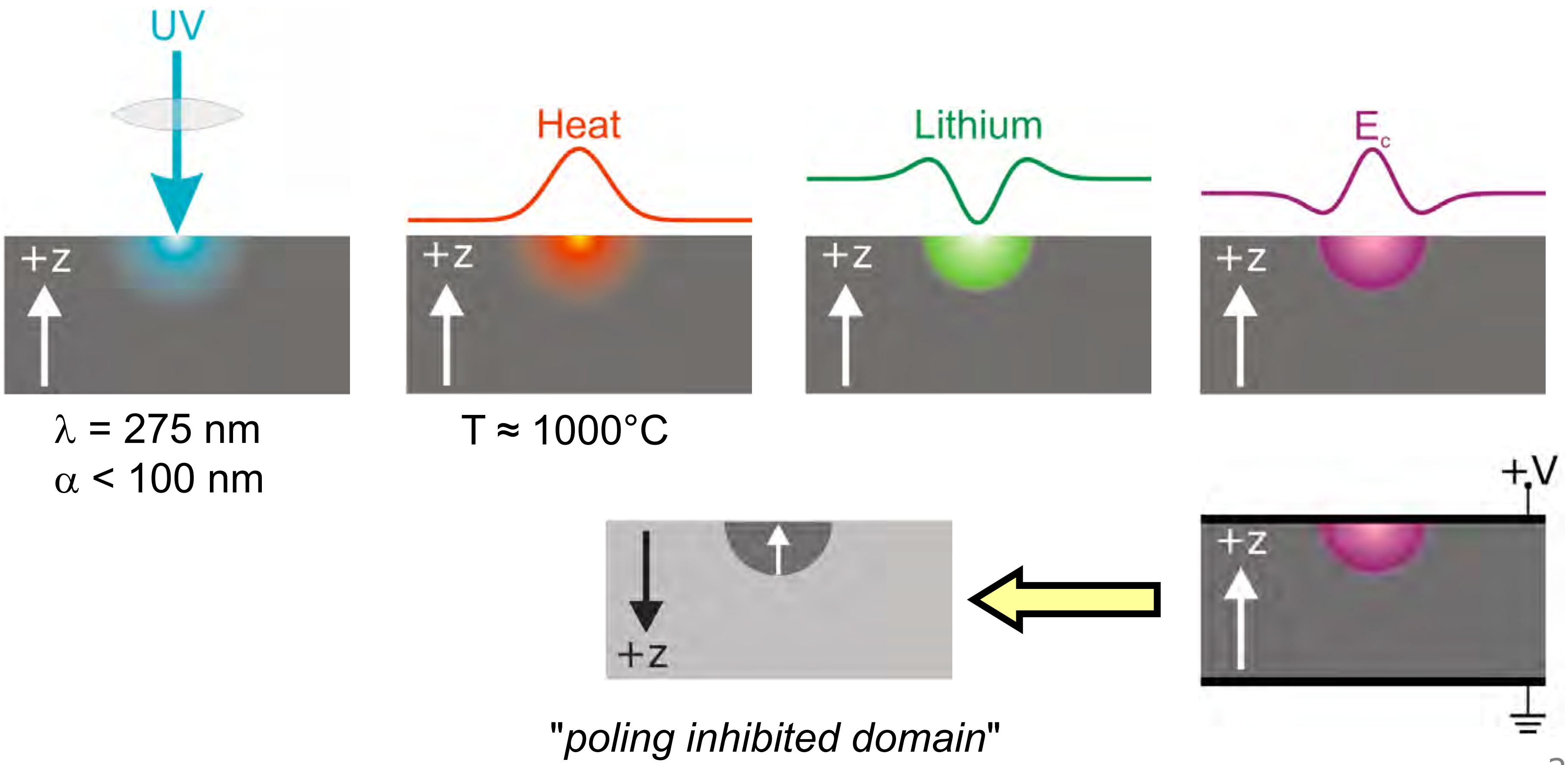
SFM writing & Domain selective etching



Poling of Domains by UV-Light

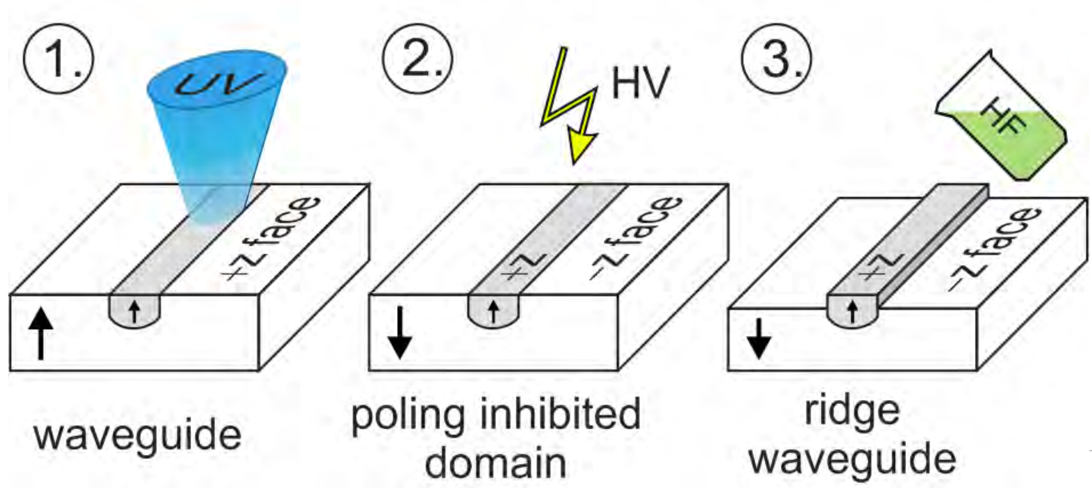
Poling inhibition

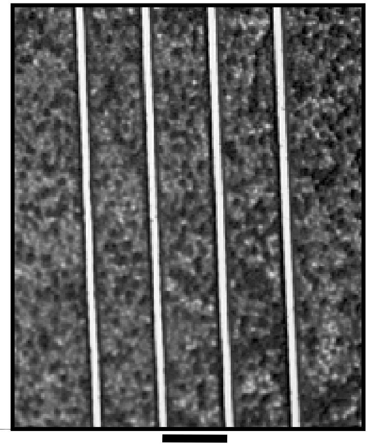


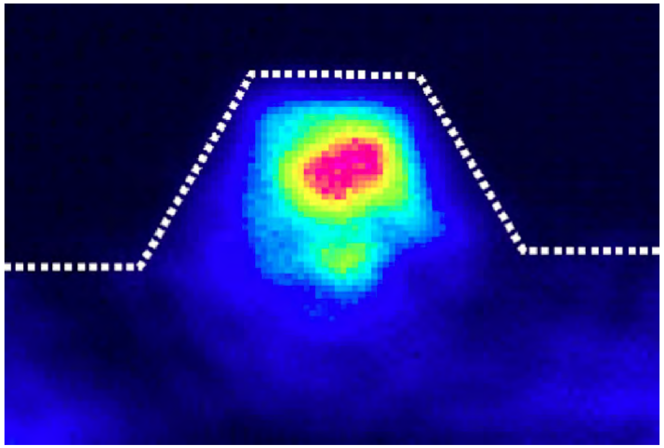


Fabrication of Waveguides

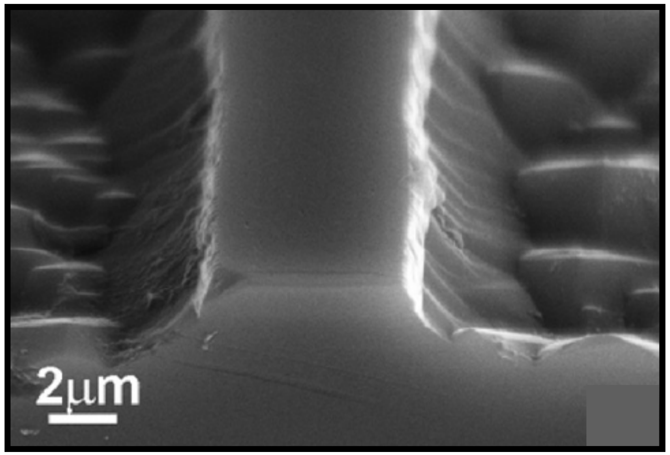
UV-light induced domains & Domain selective etching







near-field profile @ 633 nm

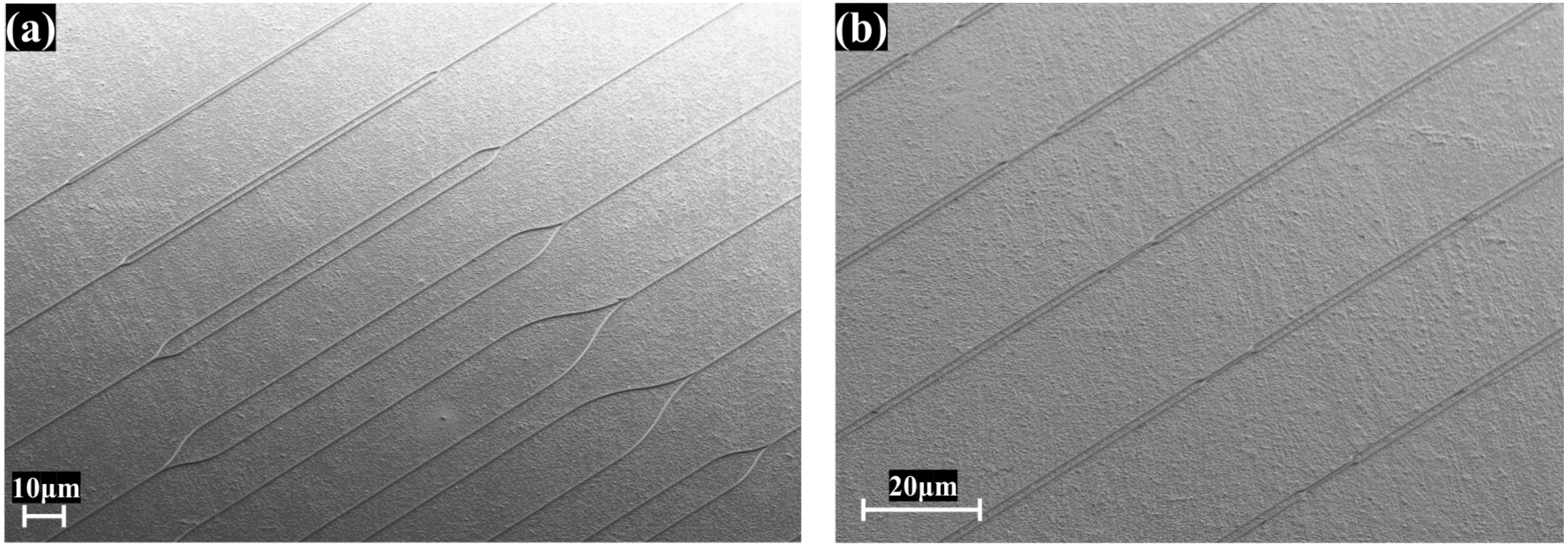


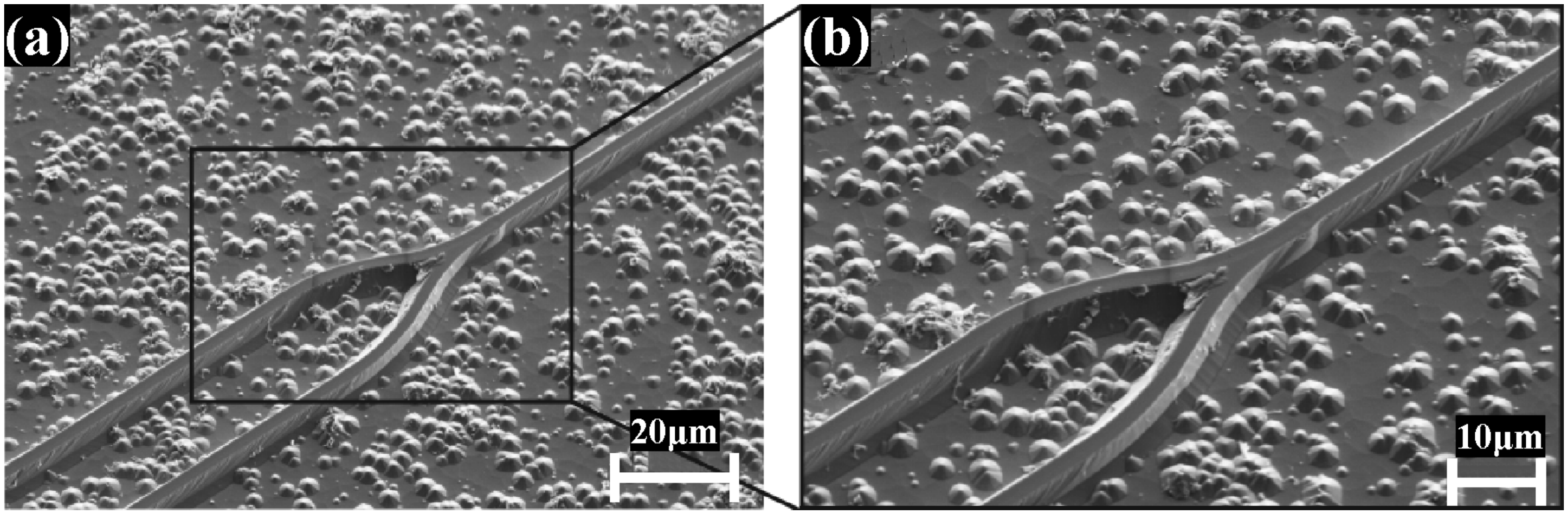
SEM image of a ridge waveuide

*APL **97**, 151112 (2010)*

Fabrication of Waveguides

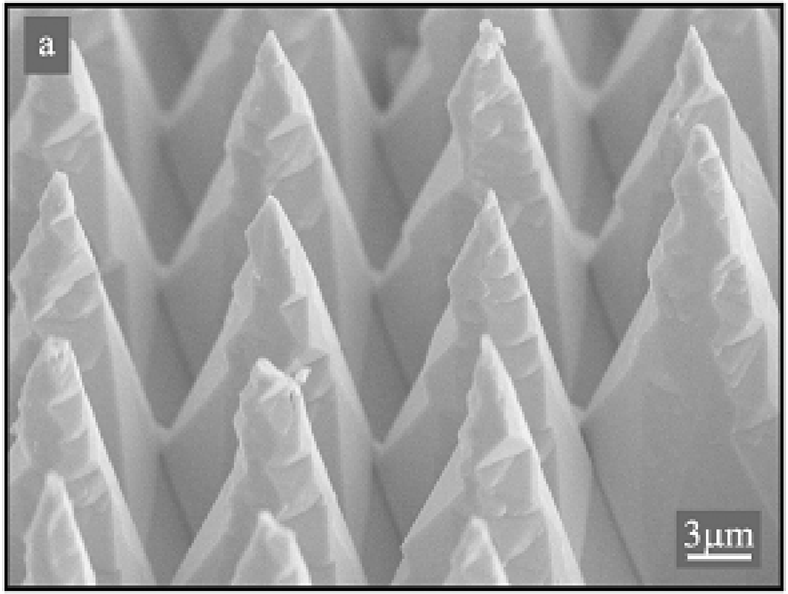
UV-light induced domains & Domain selective etching

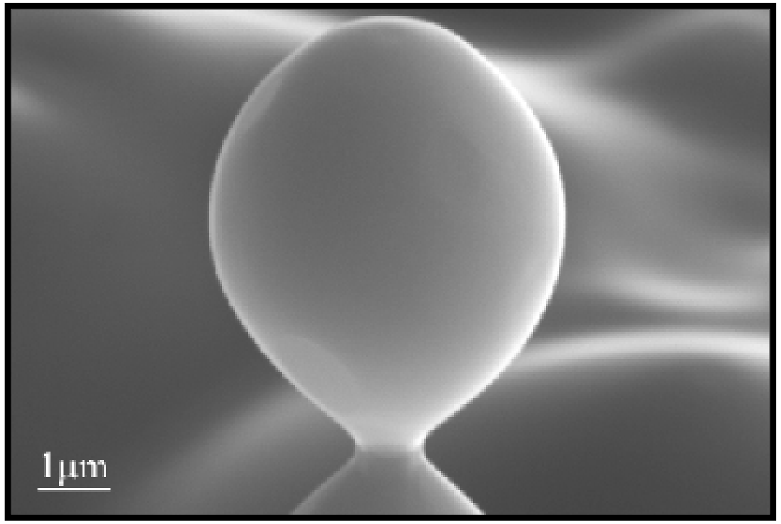


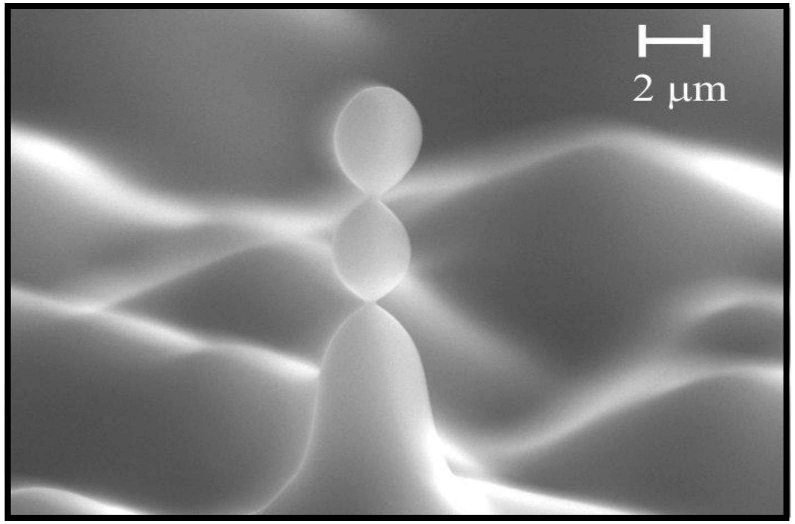


Fabrication of Photonic Micro-Structures

UV-light induced domains & Domain selective etching & Annealing







*Opt. Express **18**, 11508-11513 (2010)*

Physikalisches Institut
AFM Group

Many thanks to the Bonn crew

COLUMBIA UNIVERSITY
IN THE CITY OF NEW YORK

A. Ofan, R. Osgood, Jr.

UNIVERSITY OF
Southampton
Optoelectronics
Research Centre

Y. Jing, S. Mailis

Kunsthistorisches Museum Wien.

Pieter Brueghel the Elder (1530 – 1569**)**

Children's Games 1560

The Brueghel Dynasty

Genealogy