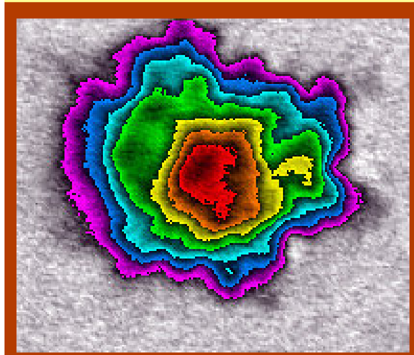
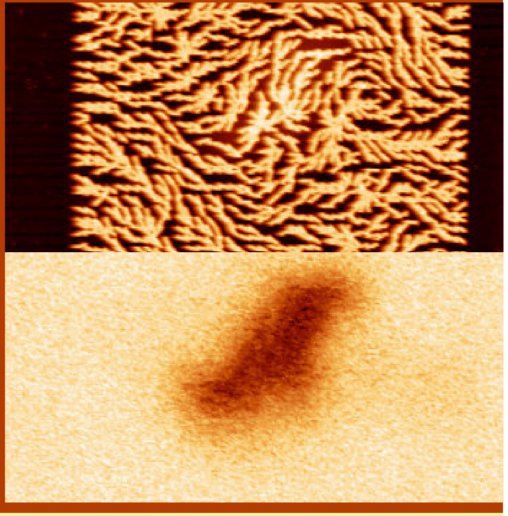
Raphaëlle Dianoux



Charge injection and detection in semiconducting nanostructures studied by Atomic Force Microscopy



CEA Grenoble / DRFMC / SP2M / SiNaPS
ESRF / Surface Science Laboratory

Outline

Electrostatic Force Microscopy in dry atmosphere

- Principles of charge injection and detection
- Minimum detectable force gradient in a Brownian motion
- Electrostatic tip-sample interaction: the plane-plane approximation
- Method of charge estimation
- Limits of this model: numerical evidence of a repulsive force

Non-linear dynamic force curves

- Coupling with the higher oscillating modes of the cantilever
- Analytical treatment of the cantilever motion
- Adding of the electrostatic interaction

Charging experiments on semiconducting nanostructures

- Charging the oxide layer
- Si nanocrystals embedded in SiO_2
- Si nanostructures made by e-beam lithography

EFM

Injection and detection

Min. force gradient

Modelling

Charge estimation

Limits

Dynamic force curves

Coupling to higher modes

Analytical treatment

+ Electrostatic interaction

Charging experiments

SiO_2 layer

Si-nanocrystals in SiO_2

Lithography Si- nanostruc.

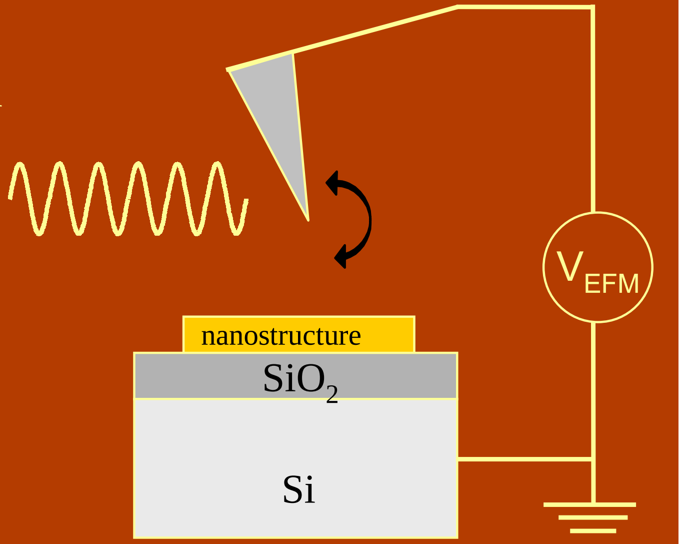
What is Electrostatic Force Microscopy?

The idea: use the AFM probe to:

Inject charges locally AND **Detect charges**

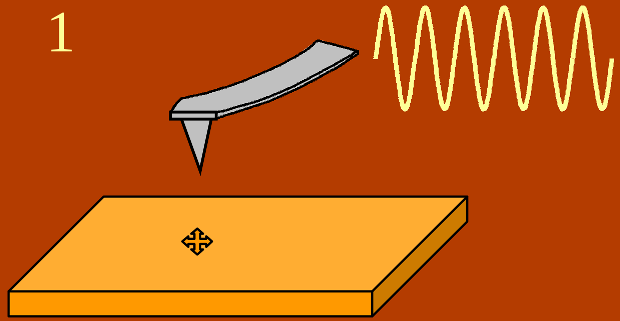
Conditions:

- the tip must be metal-coated: W_2C, PtIr
  - → Radius of curvature of the tip: ~35 nm
- the system must be electrically connected
- the tip must not touch the surface after injection
  - → oscillating mode



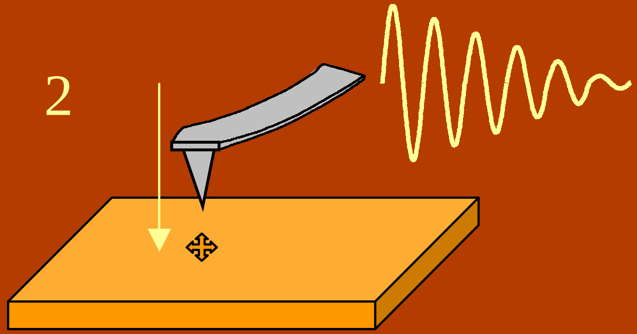
Charge injection with the tip

1



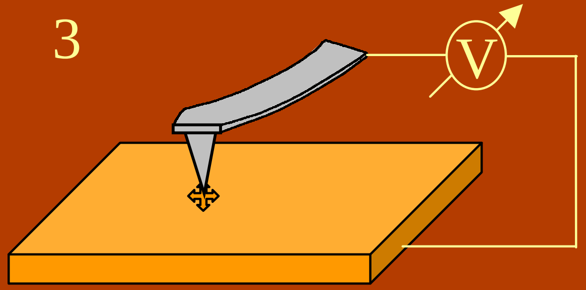
AFM in dynamic oscillating mode

2



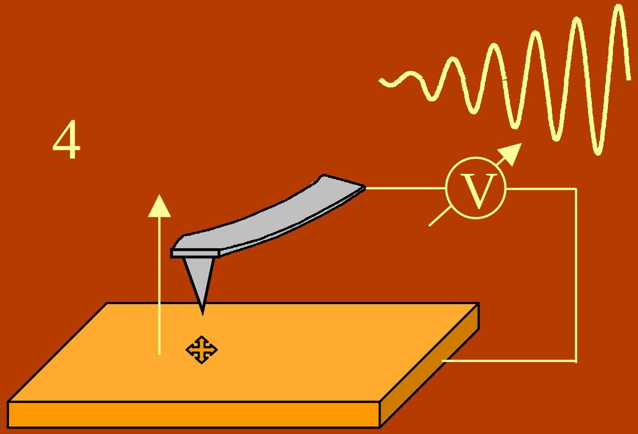
Decrease of the amplitude setpoint
to near 0: contact with the surface

3



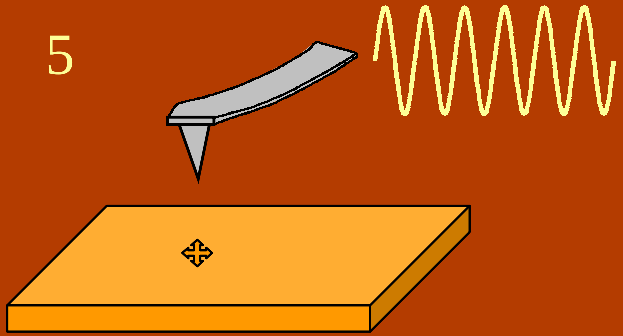
Application of a voltage (-12 to 12 V)
for 1ms to 10s

4



The setpoint is returned to
its original value

5



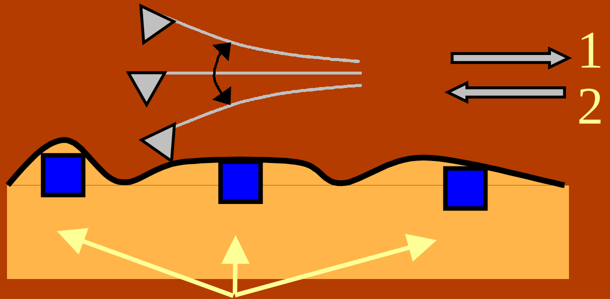
Resumes scanning

> Permanent N_2 flux

Detection of the injected charges

The double-pass method

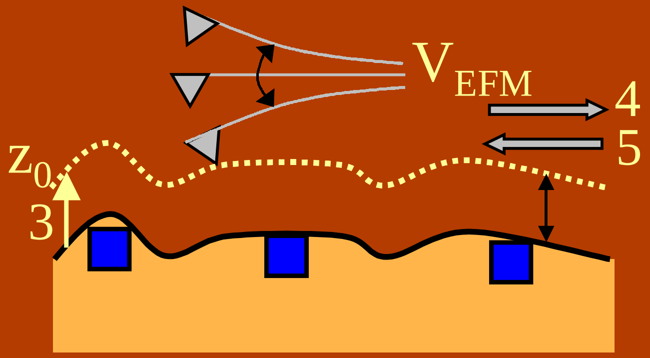
1^{st} pass: topography



Localized electric charges

1&2: Topography scan.
Feedback on the amplitude of oscillation.

2^{nd} pass: EFM signal



3: Raising of the AFM probe at a lift height z_0 of 30 to 100 nm. The feedback is cut off.

4&5: EFM scan: recording of the phase of oscillation. The tip is brought to potential V_{EFM}

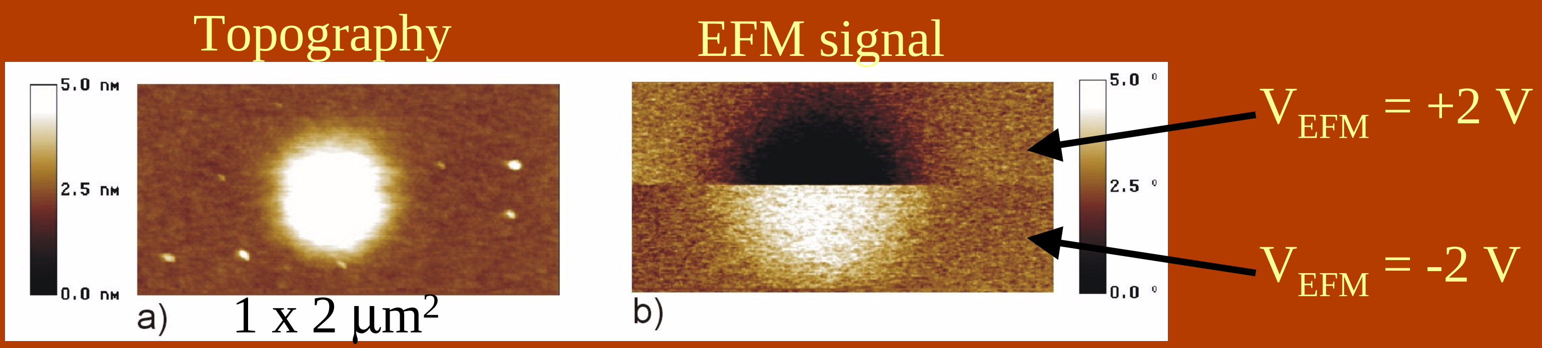
EFM signal

The EFM signal is sensitive to electrostatic force gradients.

Injection and detection of charges

Example of charge injection on 7 nm of SiO_2 on Si

Conditions: -10V/ 10s



→ EFM can distinguish the sign of the deposited charges

BUT the tip-sample force is always attractive!

EFM signal ∝ - (potential difference)^2

EFM
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Analytical treatment
+ Electrostatic interaction

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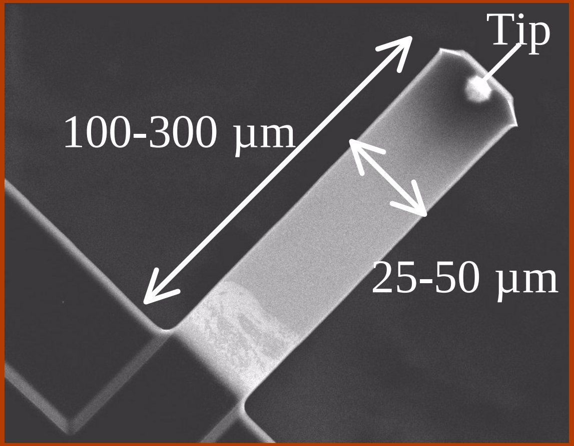
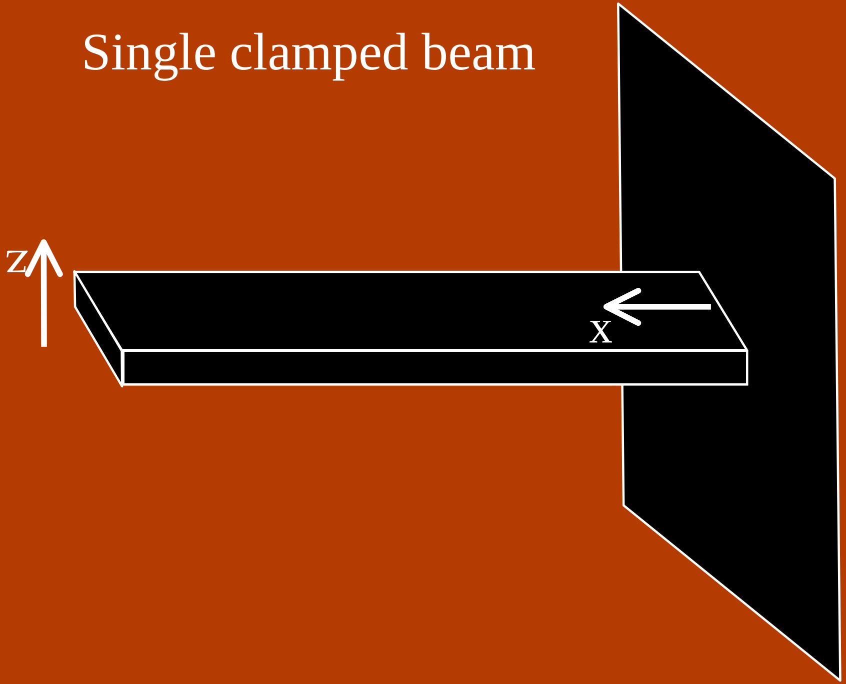
Mechanics of the cantilever



Single clamped beam

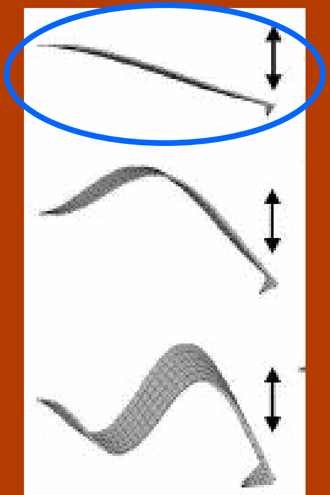


Euler-Bernouilly equation of movement:

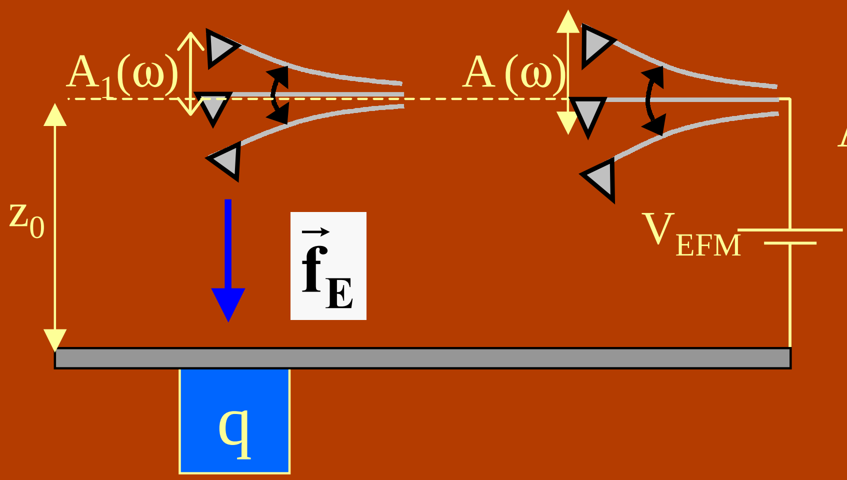
$$EI\frac{\partial^4 z}{\partial x^4}(x,t)+\rho A\frac{\partial^2 z}{\partial t^2}(x,t)=0$$

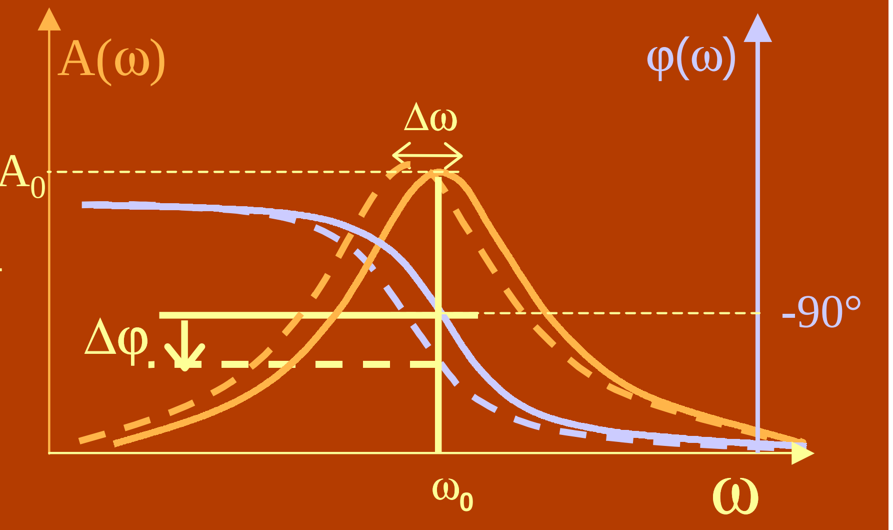
E : Young modulus
I : moment of inertia
ρ : density
A : section

Fundamental mode



Detection of a force gradient





Point-mass model:

$$\ddot{z}(t)+2\beta_0\dot{z}(t)+\omega_0^2 z(t)=\frac{F_{exc}}{m}\cos(\omega t)\quad+\frac{f(z_0+z)}{m}$$

$$f(z_0+z)\approx f(z_0)+f'(z_0)\cdot z(t)$$

$$\omega_0^2=\frac{k}{m}$$

ω_0: angular resonance frequency
k: spring constant of the cantilever
m: effective mass
β_0: friction coefficient /m

Static def

Shift of the resonance frequency

Attractive force = phase lag

$$\Delta\omega=\omega_0-\omega_1\approx\omega_0\left(\frac{1}{2k}\frac{\partial f}{\partial z}(z_0)\right)$$

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Functioning point in amplitude feedback:

$$\frac{d^2A}{d\omega^2}=0 \quad\longrightarrow\quad \omega_{s\pm}=\omega_0\left(1\pm\frac{1}{\sqrt{8}Q}\right)$$

Q: quality factor of the oscillator = 100-300

$\omega_{s\pm}\approx\omega_0$!

$$\frac{dA}{d\omega}(\omega_{s\pm})=\pm A_m\frac{4Q}{3\sqrt{3}\omega_0}$$

A_m: maximum amplitude of oscillation

$$\boxed{\Delta A=\frac{dA}{d\omega}(\omega_s)\cdot\Delta\omega=A_m\frac{2Q}{3\sqrt{3}k}\frac{\partial f}{\partial z}(z_0)}$$

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Minimum detectable force gradient in a brownian motion

Thermal noise = white-spectrum noise $\hat{R}(\omega)$
Langevin equation in Fourier space:

$$\left(-\omega^2 - i\beta_0\omega + \omega_0^2\right)\hat{Z} = \frac{\hat{R}}{m}$$

The generalized susceptibility is defined as (Landau-Lifschitz):

$$\alpha(\omega) = \frac{\hat{Z}}{\hat{R}} = \alpha'(\omega) + i\alpha''(\omega)$$

The dissipation-fluctuation theorem provides:

Spectral density
of the fluctuations

$$\left\langle \left|\hat{Z}(\omega)\right|^2 \right\rangle = \frac{k_B T}{\pi\omega}\alpha''(\omega) = \frac{k_B T Q}{\pi k \omega_0}\cdot\frac{1}{Q^2\left(1-\frac{\omega^2}{\omega_0^2}\right)^2+\frac{\omega^2}{\omega_0^2}}$$

Minimum detectable force gradient in a brownian motion

The standard deviation of movement N is:

$$N = \sqrt{4\pi B \left\langle \left| \hat{Z}(\omega) \right|^2 \right\rangle}$$

where B is the bandwidth of the system (in Hz)

<u>Simplifications:</u>

Near the resonance

$$N \approx \sqrt{\frac{4k_B TQB}{k\omega_0}}$$

Away from resonance

$$N \approx \sqrt{\frac{4k_B TB}{k\omega_0 Q}}$$

The minimum detectable force gradient is given when:
amplitude variation = standard deviation of movement

$$\Delta A = N$$

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Minimum detectable force gradient in a Brownian motion

One-dimensional, simple harmonic oscillator

Dissipation-fluctuation theorem:
Finite Q^{-1} = dissipative system = source of noise

White spectral density of the noise force *f* :

$$S_f(\omega) = \frac{4k_B T k}{Q\omega_0}$$

Units: N^2/Hz

Standard deviation of the force:

$$N \propto \sqrt{B S_f(\omega)}$$

B= bandwidth of system

where:

$$N = \sqrt{\left\langle \left(k_{eff} z\right)^2 \right\rangle} \propto \left.\frac{\partial f}{\partial z}\right|_{thermal} \cdot A_m$$

$$\boxed{\left.\frac{\partial f}{\partial z}\right|_{thermal} \propto \frac{1}{A_m}\sqrt{\frac{4k_B T k B}{Q\omega_0}}}$$

Minimum detectable force gradient in a Brownian motion

At the resonance:

$$\left.\frac{\partial f}{\partial z}\right|_{\min} = \frac{1}{A_m}\sqrt{\frac{27 k_B T B k}{\omega_0 Q}}$$

In our conditions:

- A_m = 10-20 nm
- $k_B T$ = 26 meV ambient temperature
- Q =100-300 ambient pressure
- k = 0.1-1 N/m
- ω_0 = 20 -100 kHz
- B = 500 Hz

$$\left.\frac{\partial f}{\partial z}\right|_{\min} = 3 \cdot 10^{-5}\, N.m^{-1}$$

Relation to min. detectable charge?

↓

Plane-plane approximation

Modelling of the electrostatic tip-sample interaction

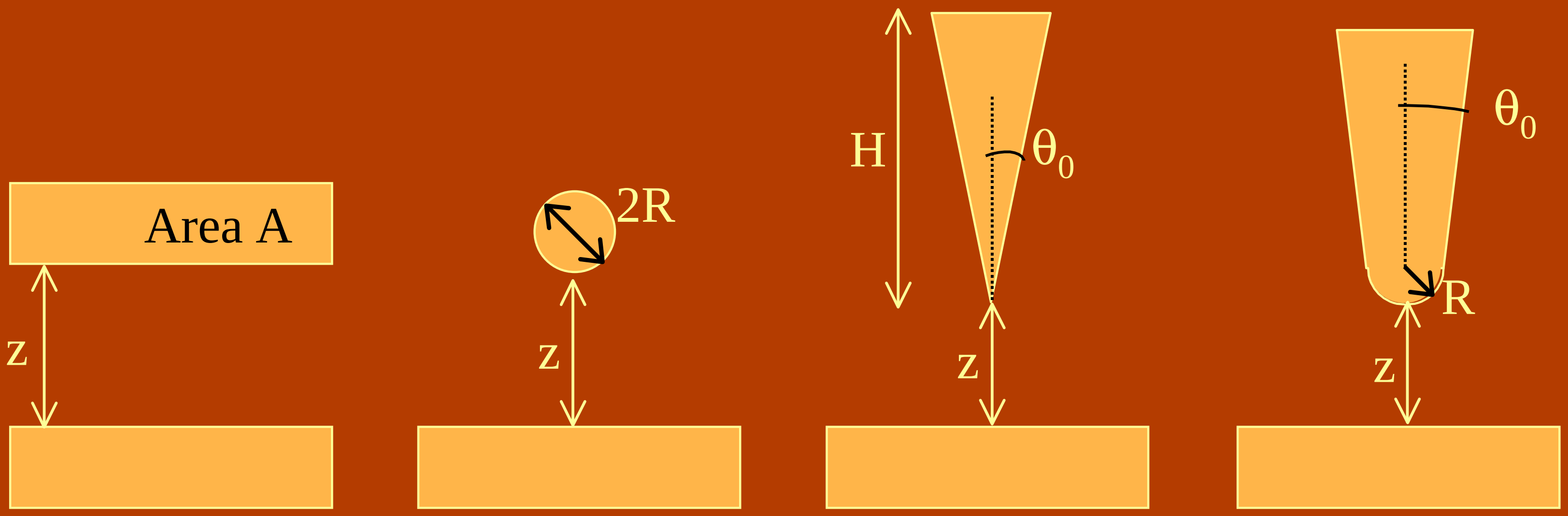
The electrostatic force is **capacitive**:

$$f(z) = \frac{1}{2}\frac{\partial C}{\partial z}(z)V^2$$

Capacitance C, C”=??

$$\frac{\partial f}{\partial z}(z) = \frac{1}{2}\frac{\partial^2 C}{\partial z^2}(z)V^2$$

Different capacitor geometries:



Plane-plane

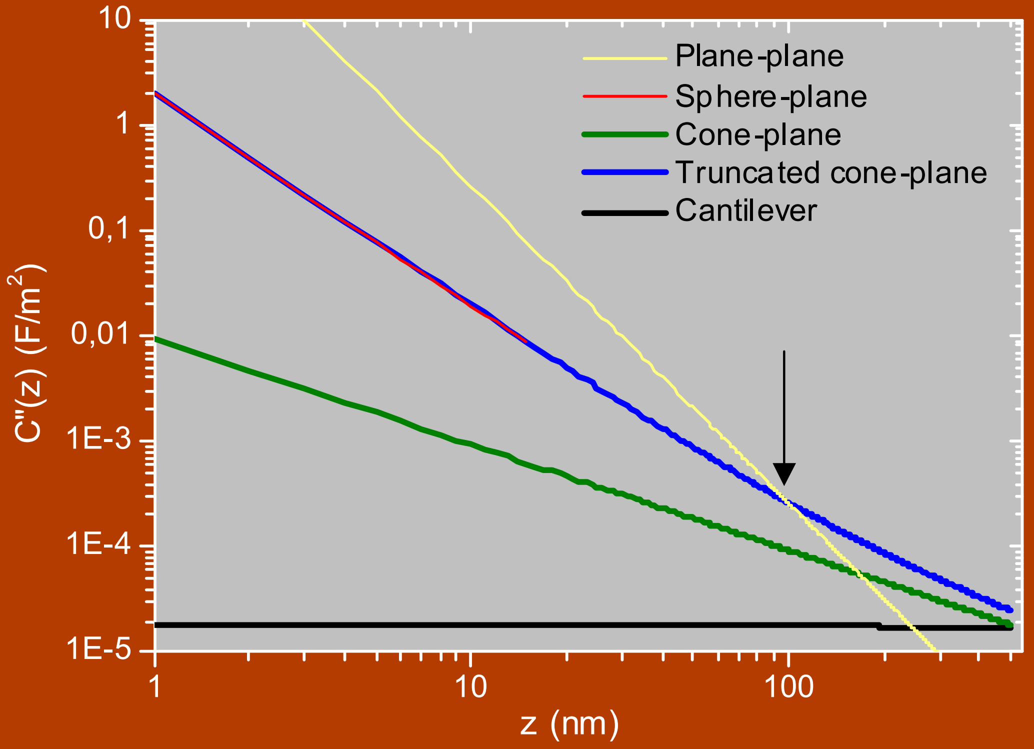
Sphere-plane

Cone-plane

Truncated cone
-plane

Modelling of the electrostatic tip-sample interaction

Plot of the 2nd derivative of capacitance vs. tip-sample distance



- Contribution of cantilever is negligible.
- Area of plane capacitor is adapted to fit C”(z) of truncated cone-plane at a lift height of 100 nm.

The simplest geometry is chosen: plane-plane capacitor

$$C''(z) = 2\varepsilon_0\varepsilon_r \frac{A}{z^3}$$

EFM

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Charging experiments

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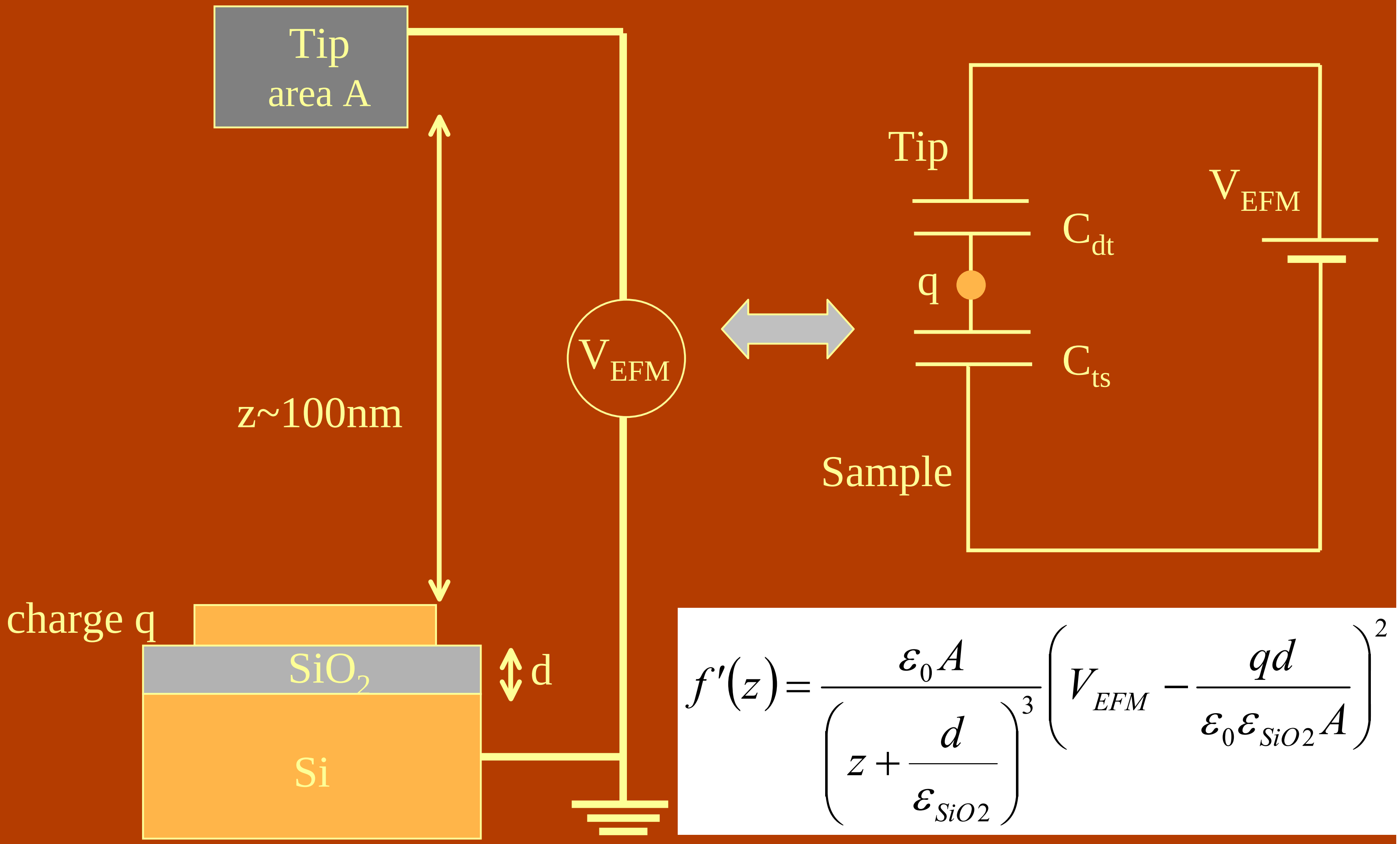
Modelling of the electrostatic tip-sample interaction

The system is modelled as 2 plane capacitors in series



$$f'(z) = \frac{\varepsilon_0 A}{\left(z + \frac{d}{\varepsilon_{SiO2}}\right)^3} \left(V_{EFM} - \frac{qd}{\varepsilon_0 \varepsilon_{SiO2} A}\right)^2$$

Minimum detectable charge at V_{EFM} =0

$$q_{\min} = \sqrt{\frac{f'_{\min}\left(z + \dfrac{d}{\varepsilon_{SiO2}}\right)^3 \varepsilon_0 \varepsilon_{SiO2}^2 A}{d^2}}$$

q_{min} dependent on:
z : lift height
d : oxide thickness
A : effective plane area

f'_{min} = 3 x 10^{-5} N.m^{-1}

z= 100 nm, A= 14700 nm^2 (disc of 140 nm in diameter)

d (nm)	7	10	25	100	400
q min (e-)	185	162	69	22	11

z= 50 nm, A= 6260 nm^2 (disc of 90 nm in diameter)

d (nm)	7	10	25	100	400
q min (e-)	54	39	18	7	5

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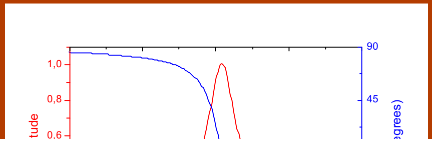
Method of charge estimation

Imaging and relating the recorded phase to a charge

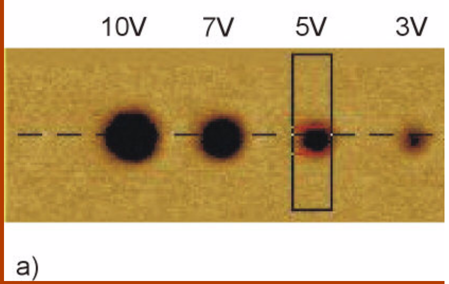
We established:

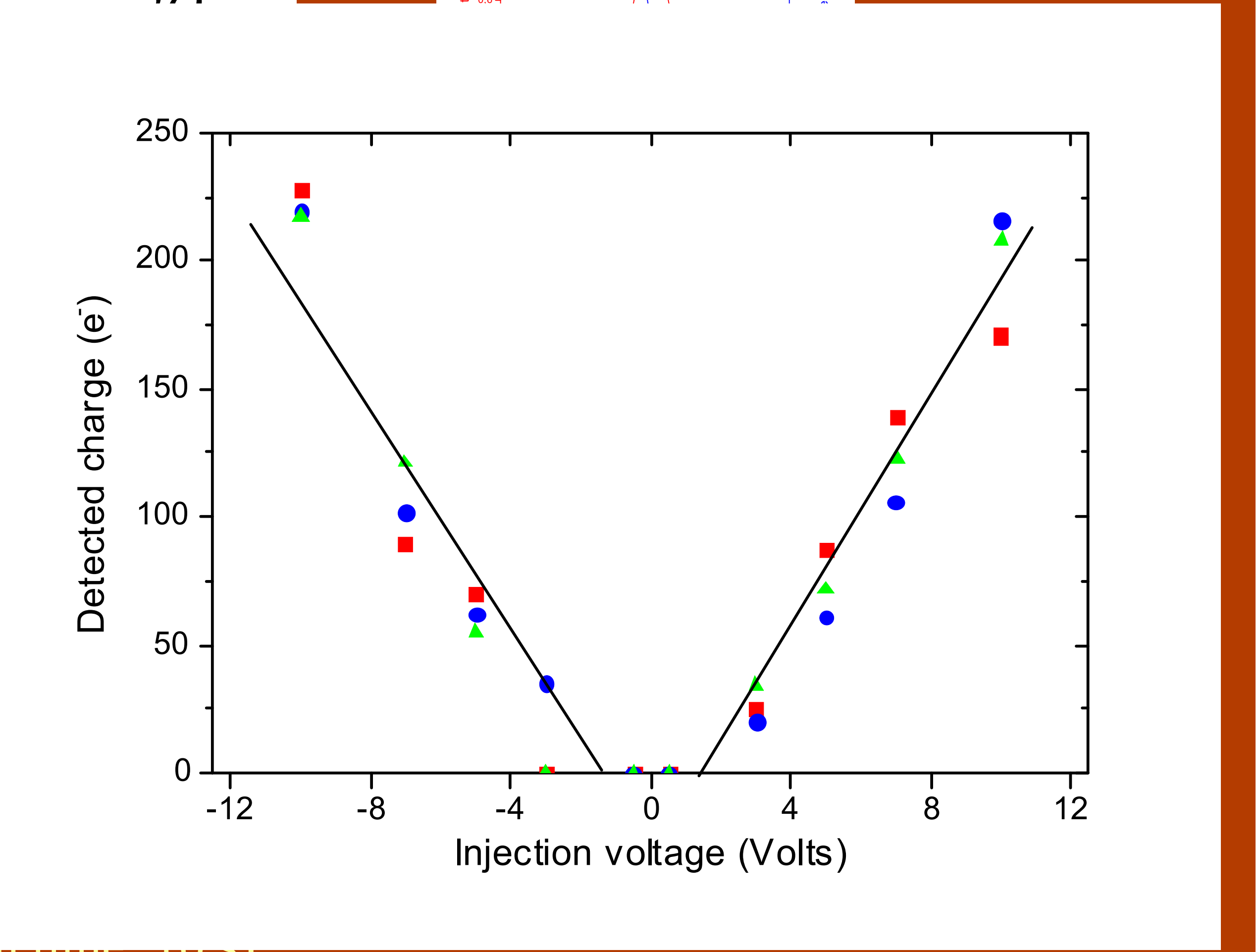
$$\Delta\omega = \frac{\omega_0}{2k}\frac{\partial f}{\partial z}(z_0)$$



Moreover:

$$q = \sqrt{\frac{\delta\phi \cdot k \left(z + \right.}{}}$$





Charging experi
(injection time: 10 s)

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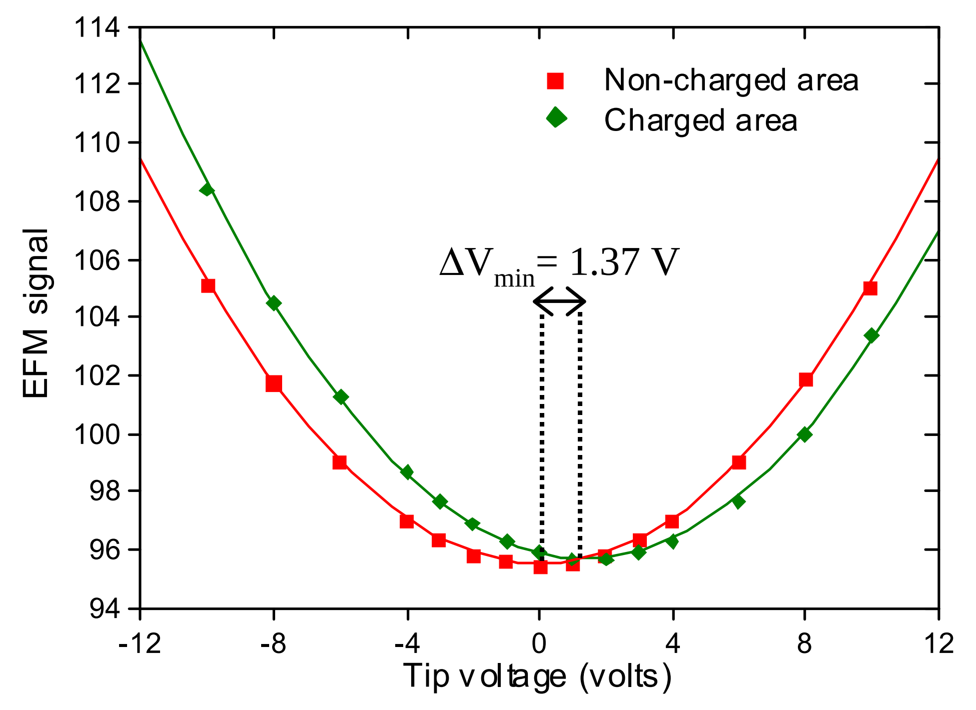
Method of charge estimation

Relate the minimum of EFM signal vs. voltage to a charge

Before and after injection, voltage V_{EFM} applied on tip is scanned

→ Minimum corresponds to $V_{EFM} = V_{surface}$



Conditions:
Injection -10V/10s
d = 25 nm
Lift height: 300 nm
A= 13 x 10^{-14} m^2
(disc 400 nm in diam.)

$$\Delta V_{\min} = \frac{qd}{\varepsilon_0 \varepsilon_{SiO2} A}$$

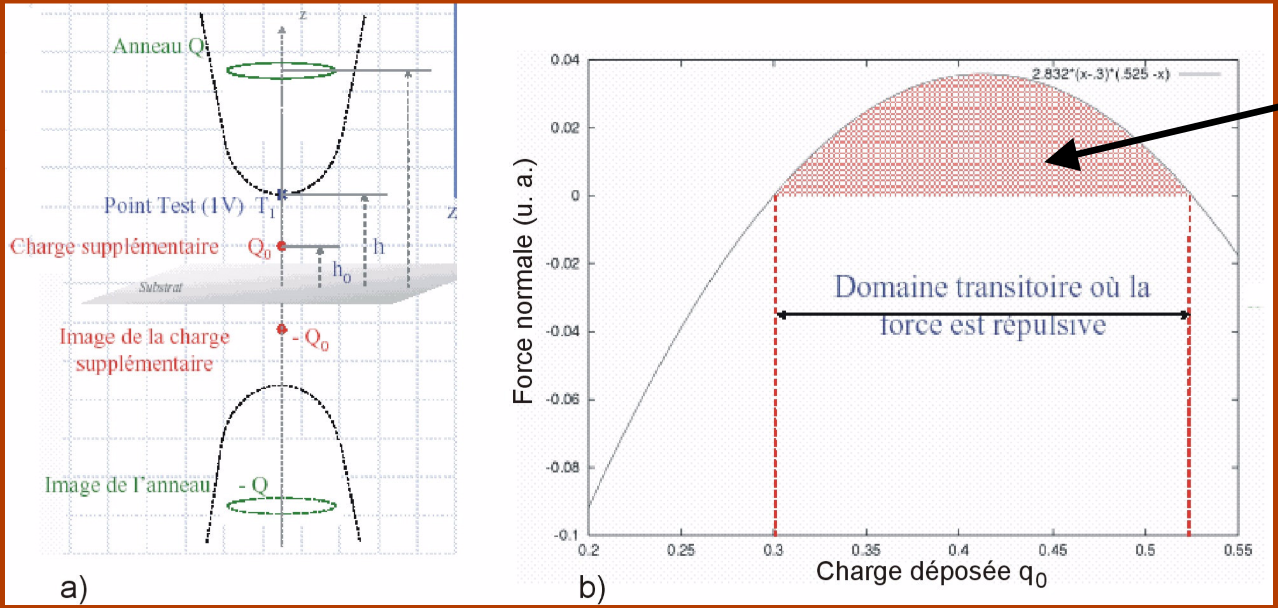
→ Here q = 1500 charges

Limits of the capacitor model

Capacitive force: always attractive

⇨ Numerical evidence of a **repulsive** interaction (J.P. Julien, CNRS)

- Distribution of equivalent charge **q** on the tip in rings
- Trapped charge $\mathbf{q_0}$ is modelled above the symmetry plane
- Charges are adjusted to have a constant potential on the tip’s surface
- Screening charges are taken into account



Domain
of repulsive
force!

⟷

16 electrons!
=barely measurable

EFM

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Non-linear dynamic force curves

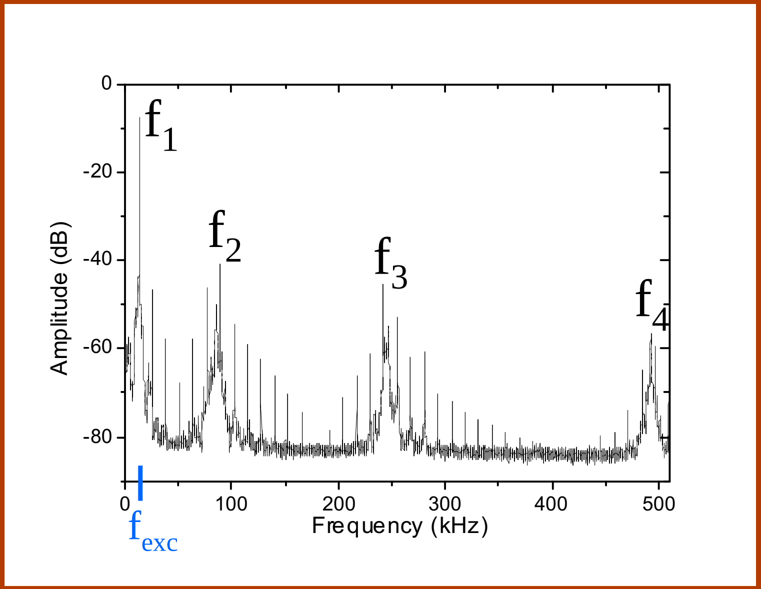
What is a force curve:

- Scanning is stopped
- Feedback on amplitude is cut off
- Cantilever is mechanically excited near resonance frequency
- Tip is approached then retracted from the surface (height ~ 200 nm)
- Amplitude and phase of oscillation are recorded

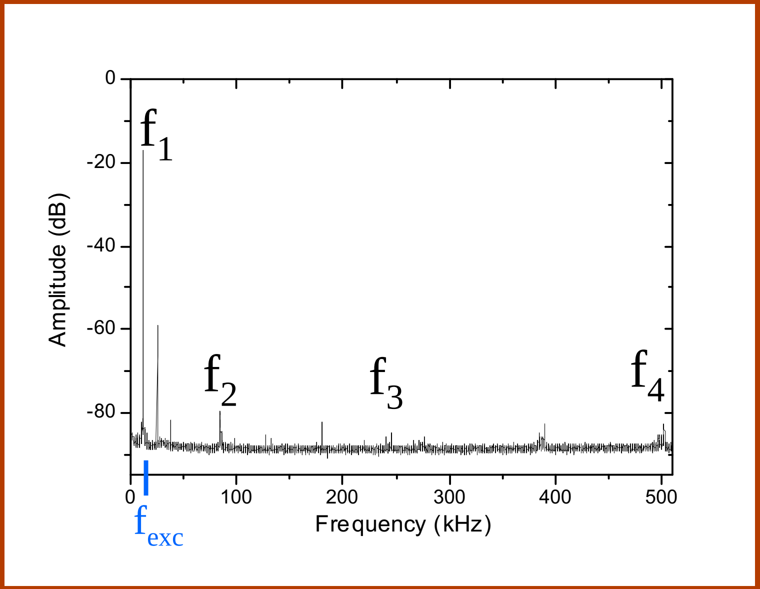
Coupling to higher oscillating modes of the cantilever

Is the movement of the cantilever still that of a harmonic oscillator?

NO! Strong excitation



YES! Normal excitation



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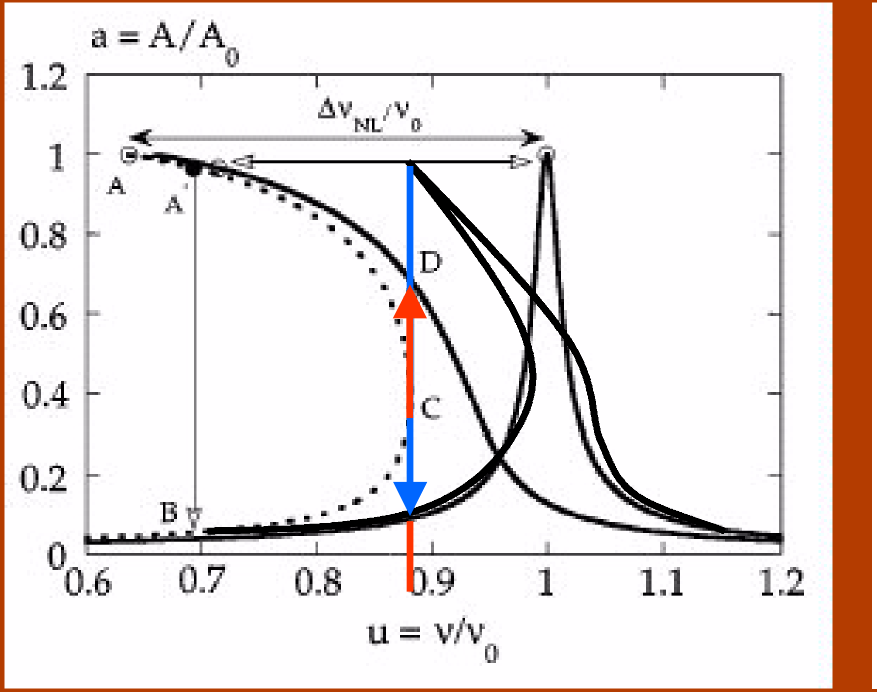
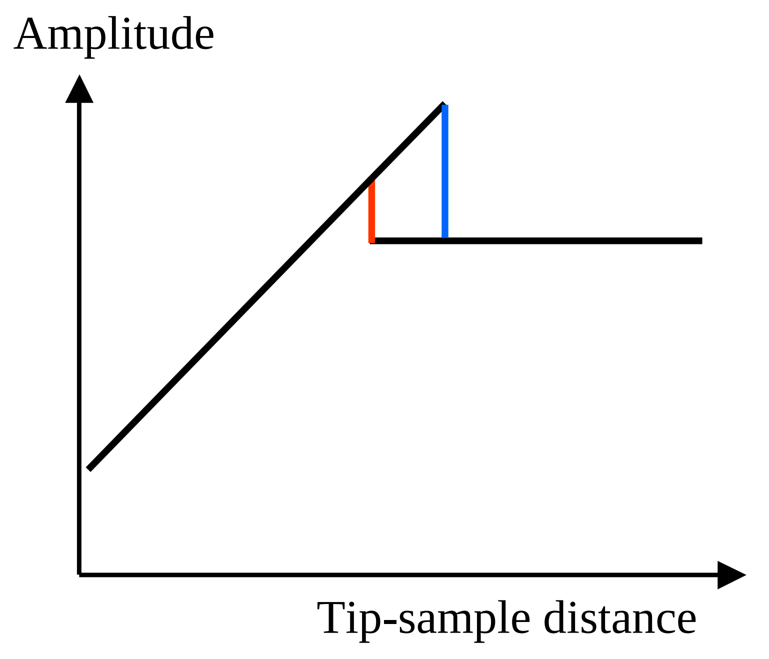
Non-linear tip-sample interaction

Deformation of the resonance curve
with increasing tip-surface interaction





Amplitude and phase of oscillation undergo hysteresis

EFM

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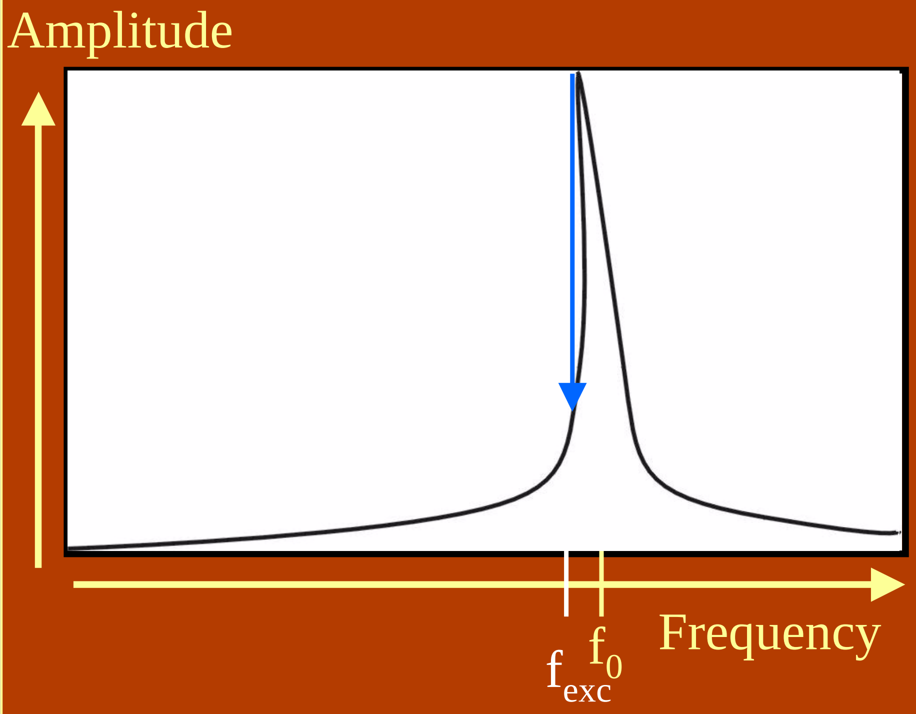

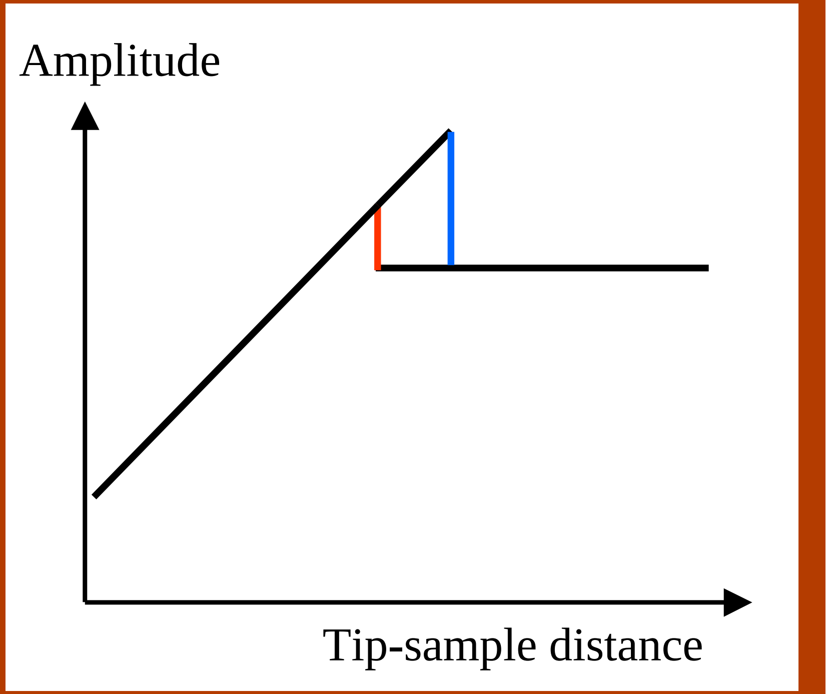

Amplitude and phase of oscillation undergo hysteresis

Analytical treatment of the movement of cantilever

Non-perturbative treatment (J.P. Aimé, CPMOH Bordeaux)

- ➢ Interaction is van der Waals : $\frac{HR}{d^2}$ (attractive force)
- ➢ Amplitude and distance are normalized to free amplitude at resonance: $a=A/A_0$, $d= z/A_0$

$$d_{A\pm} = \sqrt{a^2 + \left(\frac{k_{vdW}}{\left(u^2-1\right) \mp \frac{1}{Q}\sqrt{\frac{1}{a^2} - u^2}} \right)^{2/3}}$$

- ➢ $u= \omega / \omega_0$
- ➢ k_{vdW}: dimensionless parameter related with strength of van der Waals forces

$$\phi_{A\pm} = \arctan\left(\frac{u^2}{Q\left(u^2-1\right) + Q\frac{k_{vdW}}{\left(d_{A\pm}^2 - a^2\right)^{3/2}}} \right)$$

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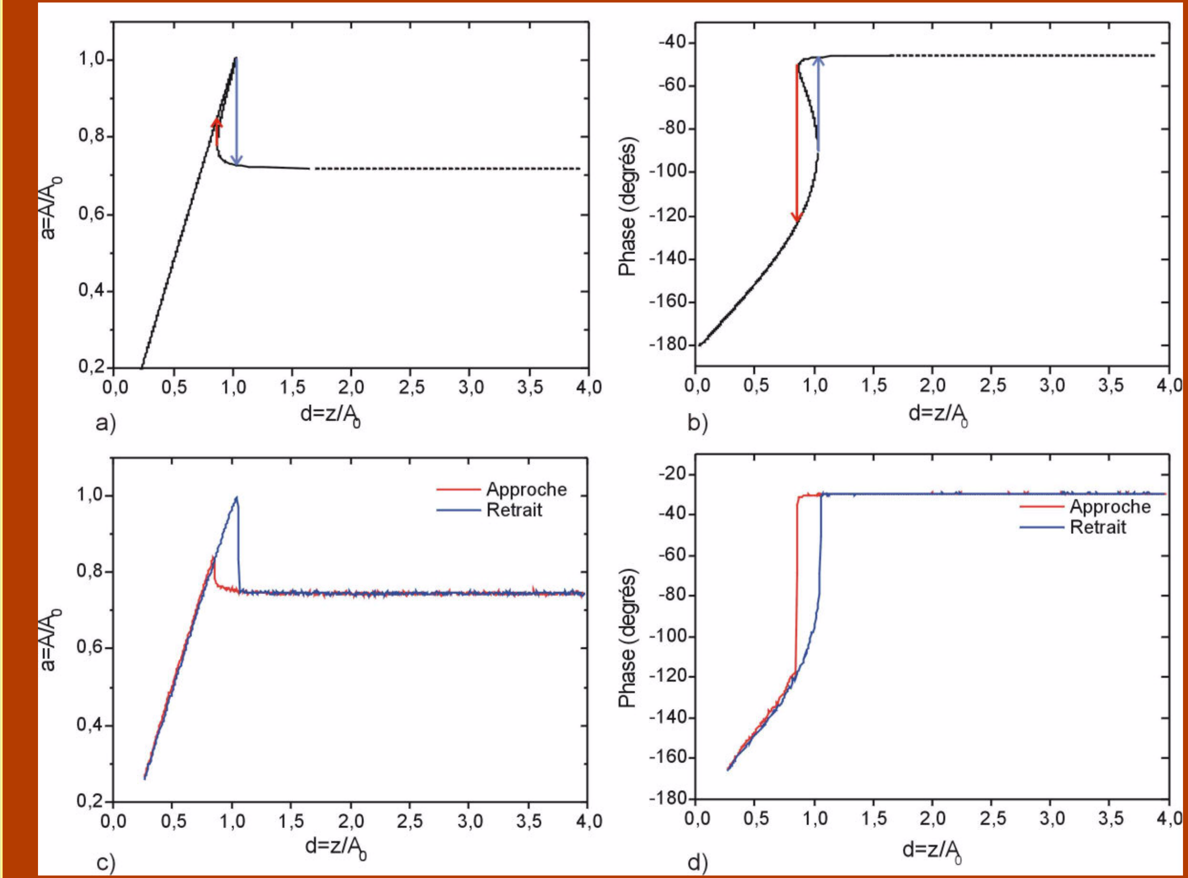
Analytical treatment of the movement of cantilever

These analytical curves explain the hysteresis observed experimentally



Analytical curves

Experimental curves

Experimental parameters used in the analytical curves:

$Q = 80$

$k = 2.3$ N/m

$\omega_0 = 57.85$ kHz

$u = 0.9939$

$A_0 = 13.5$ nm

Adding the electrostatic interaction

Capacitive tip-sample coupling taken into account

$$d_{A\pm} = \sqrt{a^2 + \left(\frac{k_{vdW} + k_{elect}V^2}{\left(u^2-1\right) \mp \frac{1}{Q}\sqrt{\frac{1}{a^2}-u^2}}\right)^{2/3}}$$

where

$$\boxed{k_{elec}V^2 = \frac{\varepsilon_0 A}{kA_0^3}V^2}$$

$$\phi_{A\pm} = \arctan\left(\frac{u^2}{Q\left(u^2-1\right)+Q\frac{k_{vdW}+k_{elect}V^2}{\left(d_{A\pm}^2-a^2\right)^{3/2}}}\right)$$

We take advantage of the fact that
the capacitive force for a plane capacitor
has the same distance-dependence d^{-2}
as the van der Waals force

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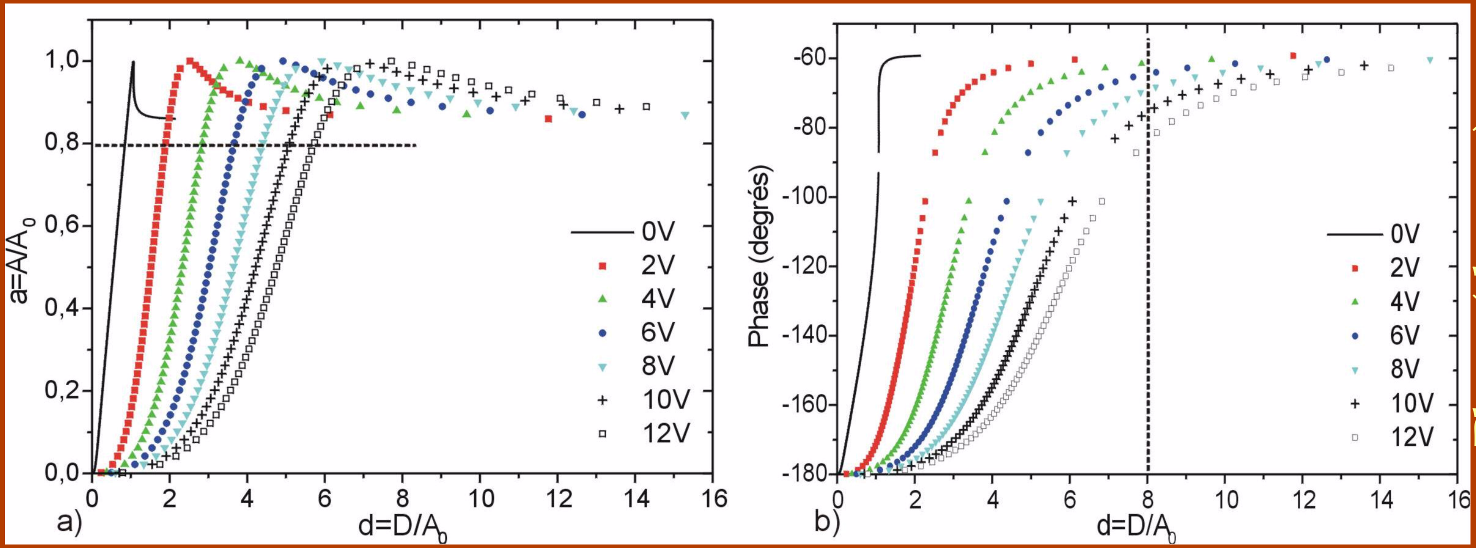
Analytical curves

Normalized amplitude



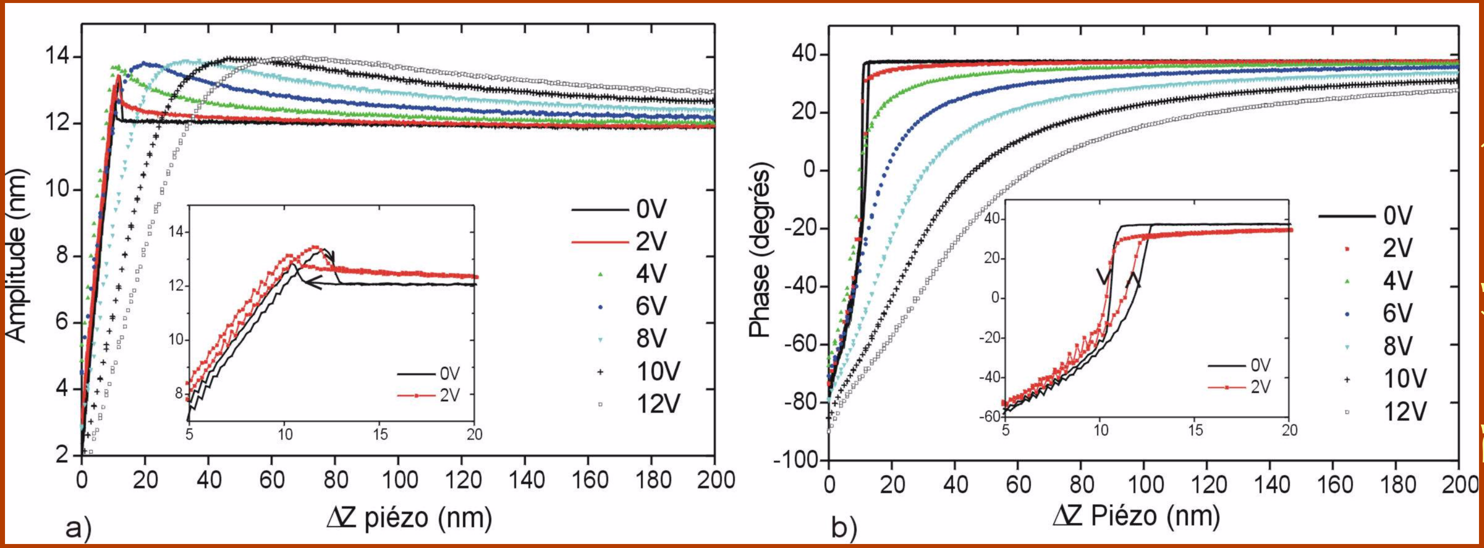
Phase (degrees)

Normalized distance

A_0= 14 nm

Experimental curves

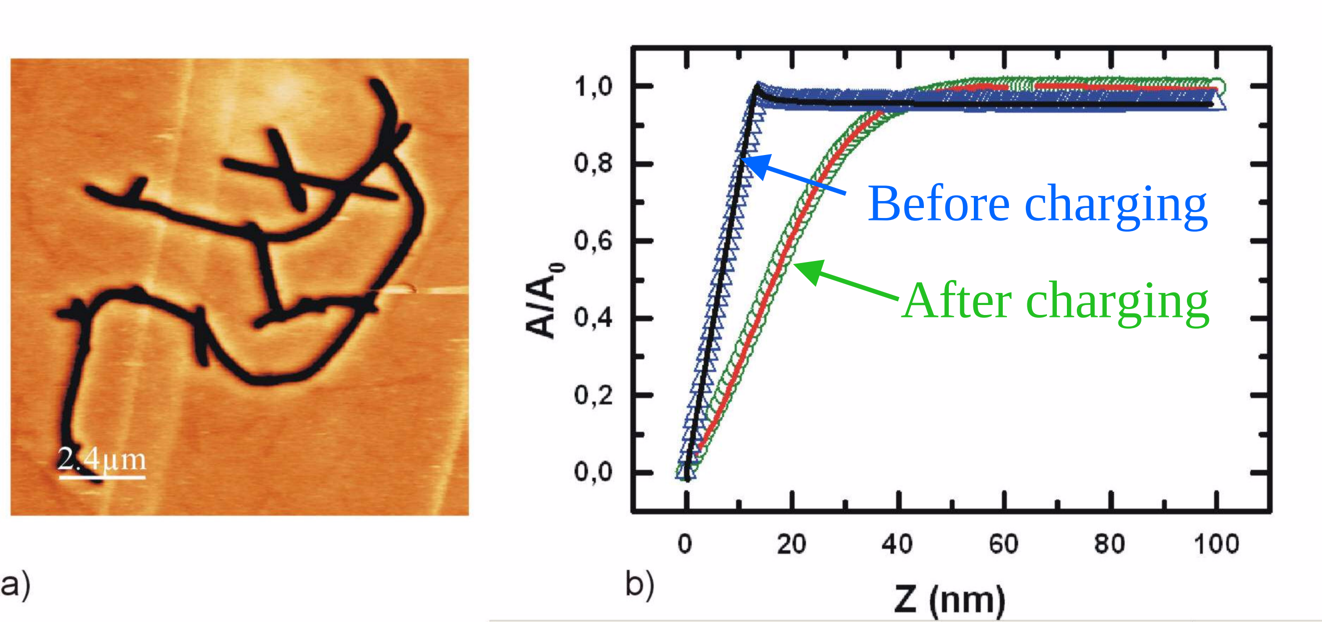
Amplitude



Phase (degrees)

Quantitative charge measurement with force curves

Application to carbon nanotubes (M. Paillet, Uni Montpellier)

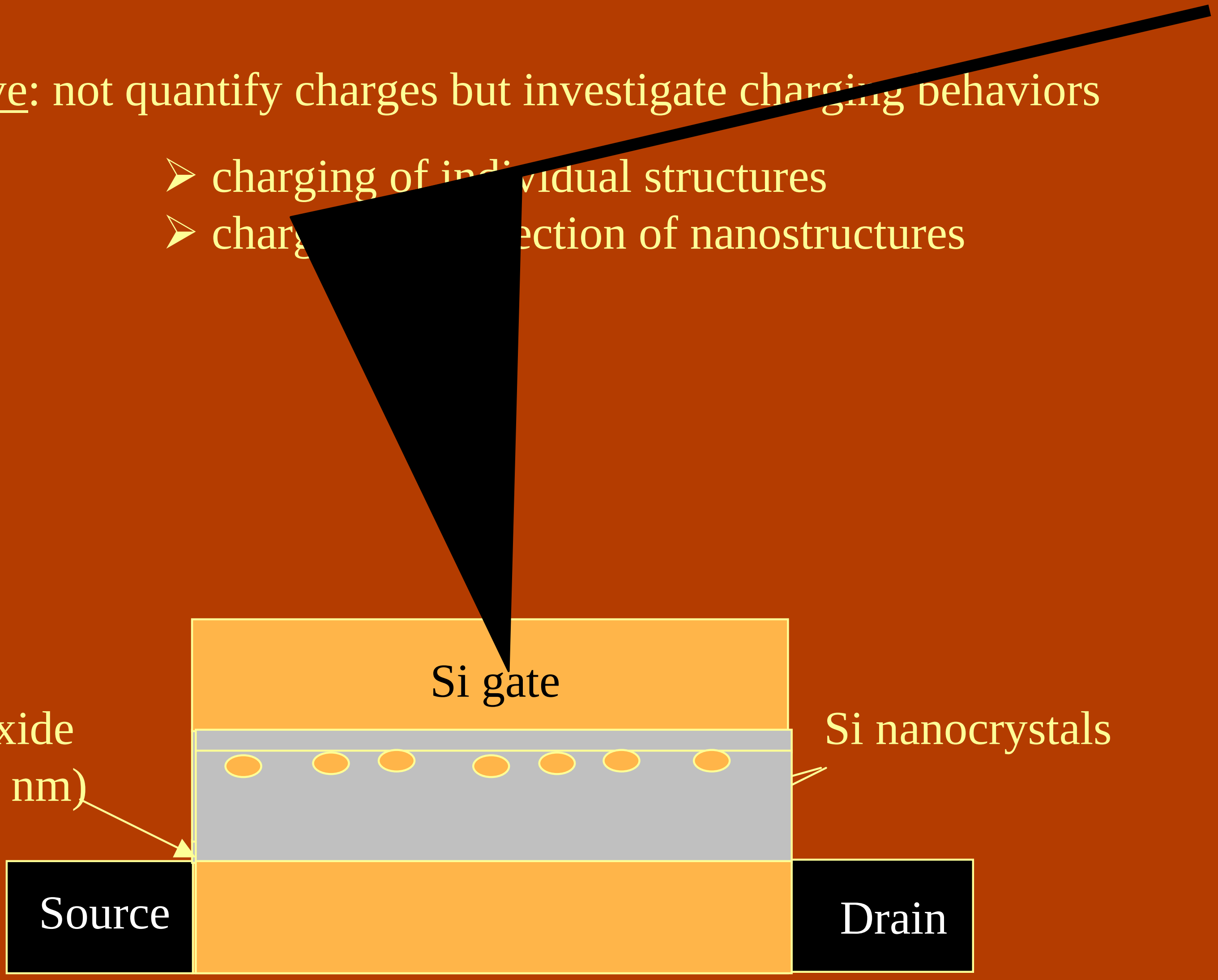


- ➢ Fitting the data before injection provides all parameters (A_0, u, U_{vdW})
- ➢ After injection, the fit provides q=10 electrons

Charging experiments on semiconducting nanostructures

Objective: not quantify charges but investigate charging behaviors

- charging of individual structures
- charg ection of nanostructures



Si gate

Tunnel oxide
SiO_2 (2,5 nm)

Si nanocrystals

Source

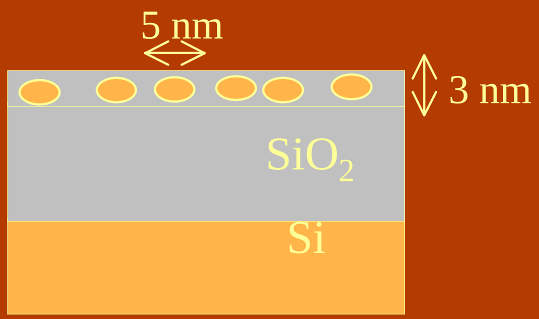
Drain

Si-nc non-volatile memory

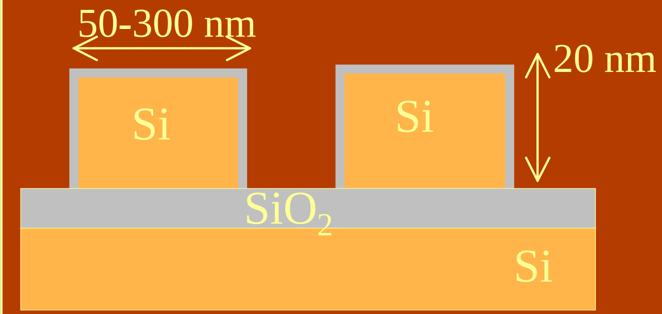
Charging experiments on semiconducting nanostructures

3 types of samples:

- ➢ Reference SiO_2 layer on Si
  - ✓charging behavior of an insulator



- ➢ Si-nanocrystals embedded in SiO_2
  - ✓very small ~5 nm in diameter
  - ✓collective behavior



- ➢ Si-nanostructures made by e-beam lithography
  - ✓well-defined, ~100 nm in dimension
  - ✓individual behavior

EFM
Injection and detection
Min. force gradient
Modelling
Charge estimation
Limits

Dynamic force curves
Coupling to higher modes
Analytical treatment
+ Electrostatic interaction

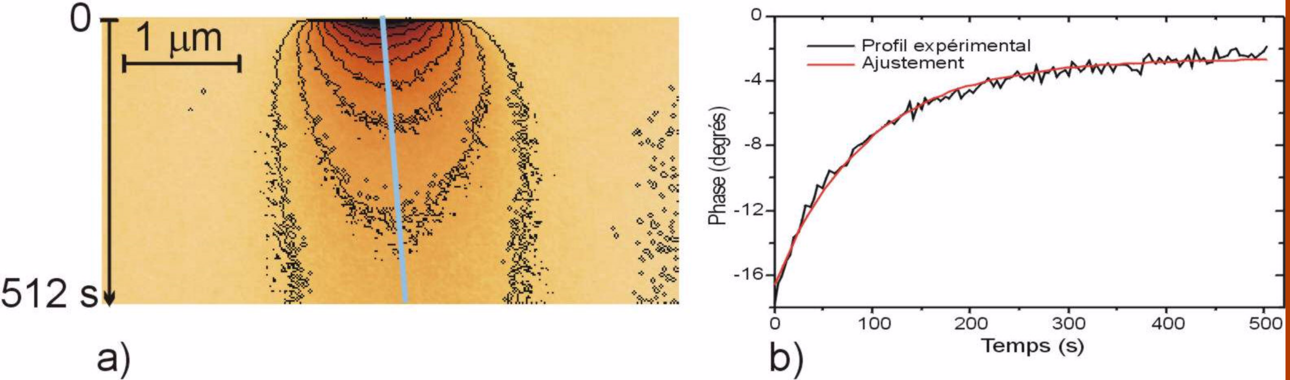
Charging experiments
SiO_2 layer
Si-nanocrystals in SiO_2
Lithography Si- nanostruc.

Charging insulators: the case of SiO_2

Large electric field (~10^8 V.m^{-1}) necessary
to deposit only a few 100 charges

Charging of 25 nm of thermal oxide, conditions: -10 V/ 10s
Recording of the EFM signal

Time



Characteristic retention time: 94 seconds
=
Low retention time

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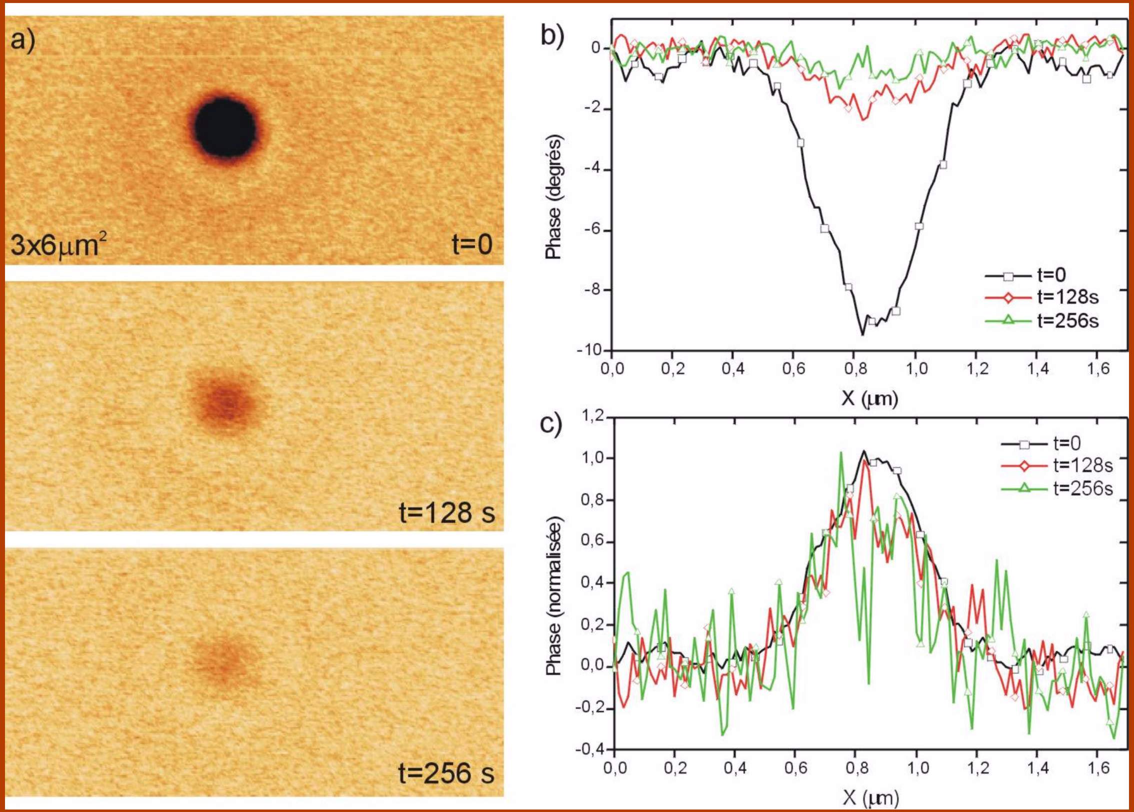
Charging insulators: the case of SiO_2

Charging of 25 nm of thermal oxide, conditions: -10 V/ 10s

Recording of the EFM signal



EFM signal

Normalized EFM signal

Absence of lateral spreading of the charges

EFM
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Limits

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Coupling to higher modes
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+ Electrostatic interaction

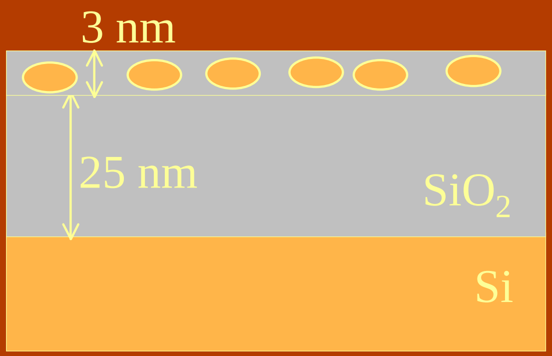
Charging experiments
SiO_2 layer
Si-nanocrystals in SiO_2
Lithography Si- nanostruc.

Silicon nanocrystals embedded in SiO_2

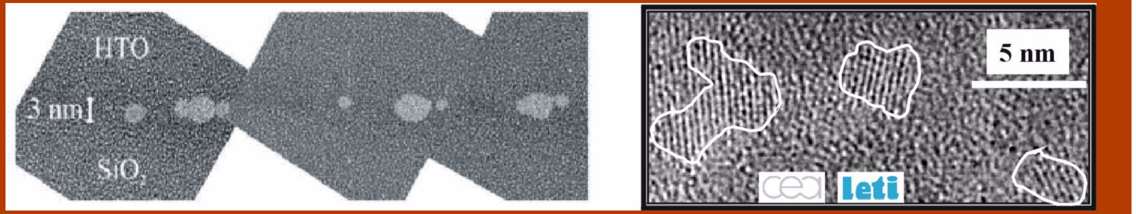
Elaboration: (CEA Grenoble/LETI)

- deposition of a SiO_x layer ($x < 2$) by LP-CVD
- annealing at 1000°C, 10 minutes

= precipitation of Si nanocrystals in SiO_2 matrix



TEM pictures



Cross-section

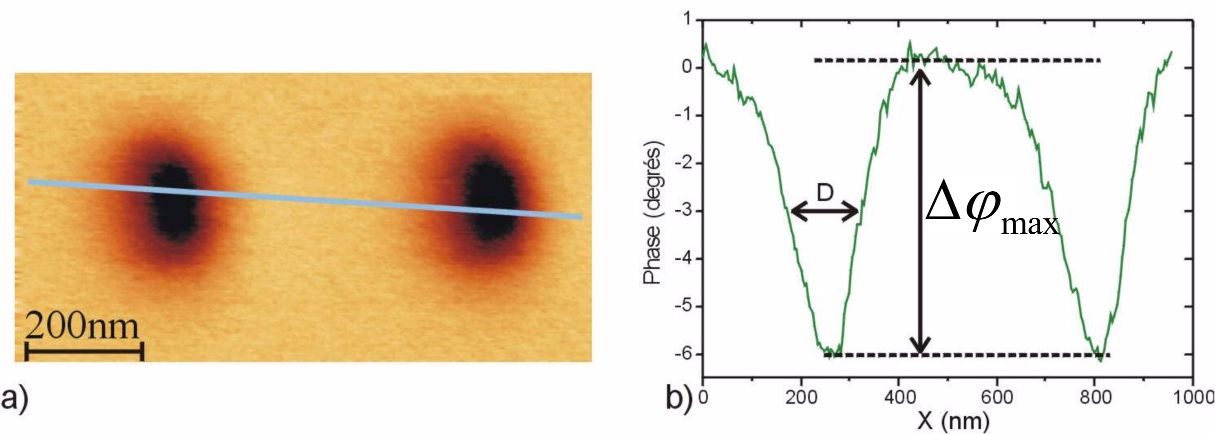
Plane-view

Typical dimension: 3 nm

Density depends on x, varies from 3×10^{11} to 10^{12} cm^{-2}

Silicon nanocrystals embedded in SiO_2

First behavior: very low Si-nc density

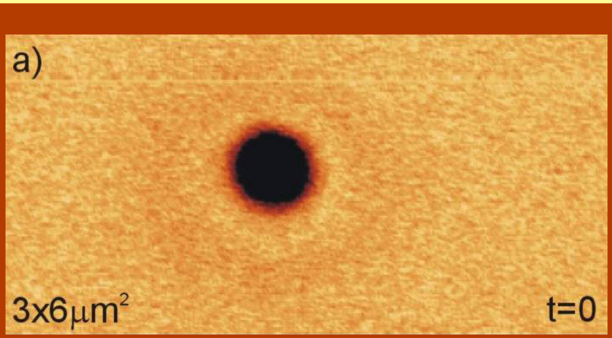


Circular shape of injected charges that does not evolve in time

Time retention: several hours

Estimation of **one electron per nanocrystal**

Any difference from reference SiO_2 sample?



EFM
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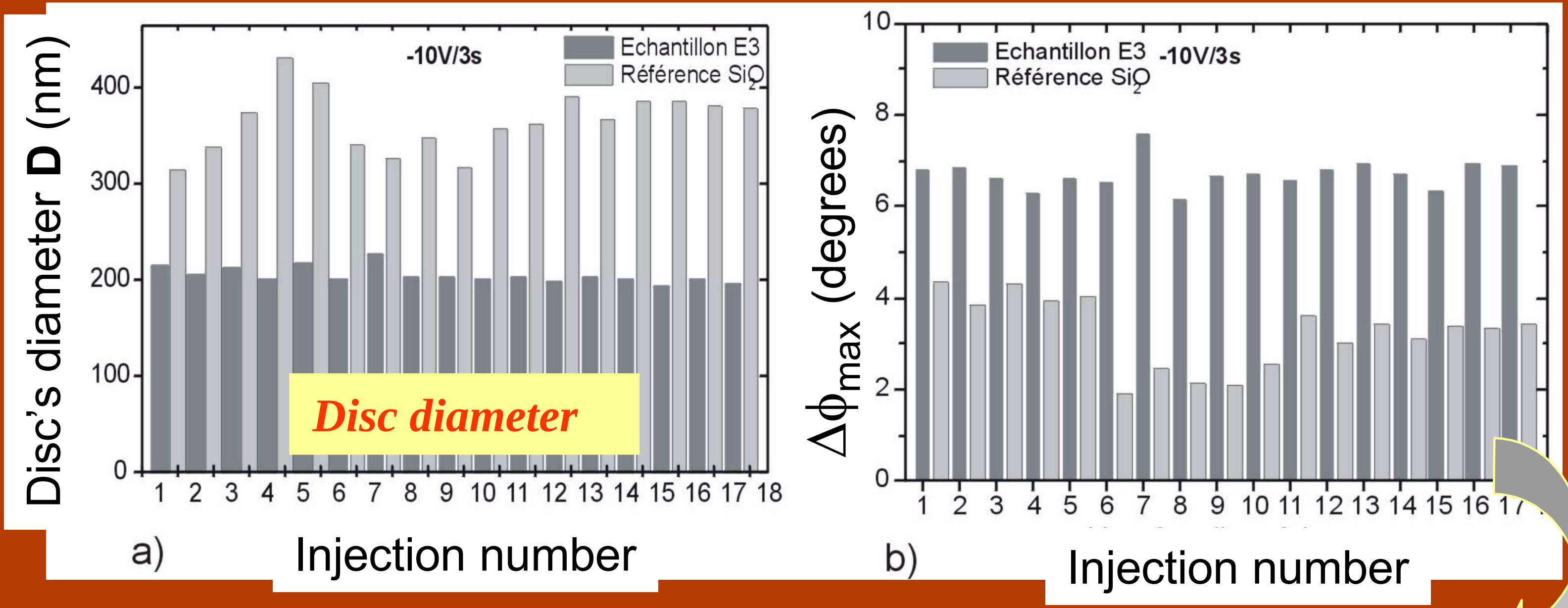
Charging experiments
SiO_2 layer
Si-nanocrystals in SiO_2
Lithography Si- nanostruc.

Low-density Si-nanocrystals embedded in SiO_2

Same charging conditions: -10 V / 3 s

■ Si-nanocrystals

□ SiO_2 reference sample



Disc diameter

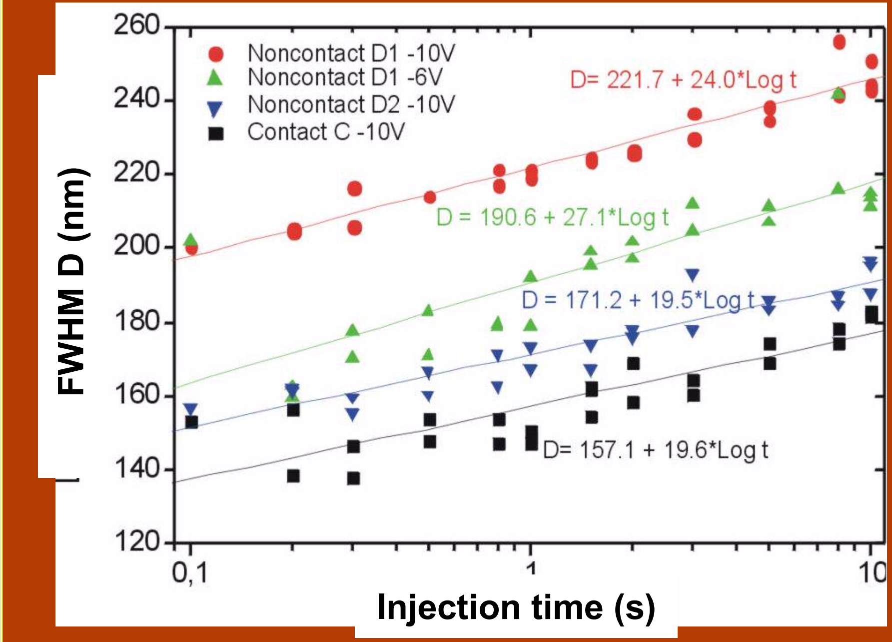
Si-nanocrystals:
D ~200 nm
$\Delta\phi_{max}$ ~6.5°

SiO_2 reference:
D ~350 nm
$\Delta\phi_{max}$ ~3 °

$\propto$ *e- density*

Smaller electron cloud
Higher surface density of electrons

Evolution of the disc with the injection time



Si-nanocrystals

Reproducible experiment with homogeneous distribution of slopes

The disc’s diameter evolves as log (injection time)

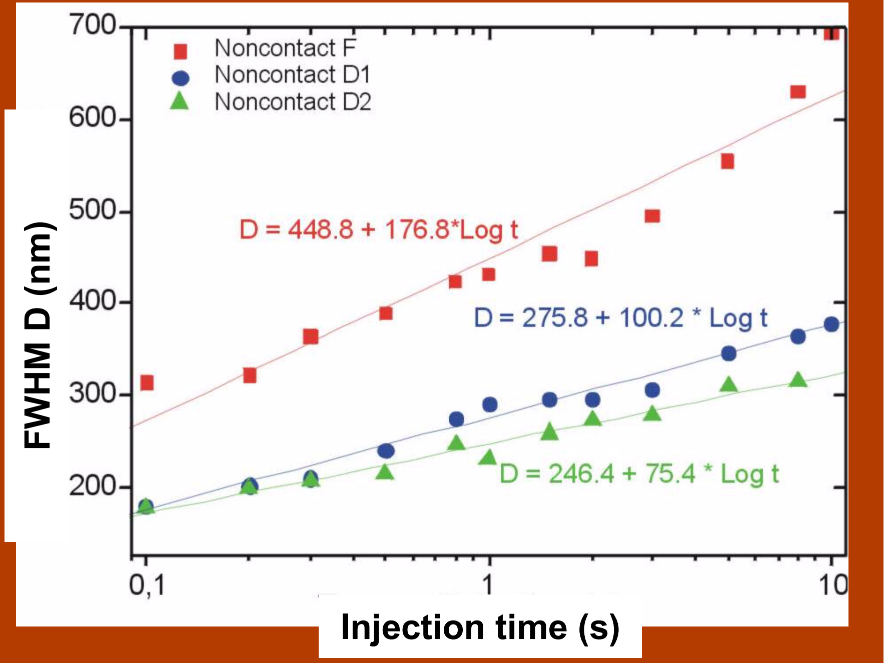
⇩

Infinitely slow saturation

Reference SiO_2

Larger disc’s diameters= easier spreading of the charges

Inhomogeneous distribution of slopes: due to flawed tip-sample contact?



Low-density Si-nanocrystals vs. SiO_2 reference sample

- Same circular shape of the electron cloud for both samples

BUT

in the same charging conditions:

- the electron cloud is **smaller** and **denser** for the Si-nanocrystal sample
- and it remains much **longer** (hours vs. minutes)

- Same logarithmic injection-time dependence

BUT

Si-nanocrystals shows **homogeneous** distribution of slopes whereas SiO_2 shows an **inhomogeneous** one

- Tip-sample contact resistance is dominant in SiO_2 sample
- Intercrystal-resistance is dominant in Si-nanocrystal sample

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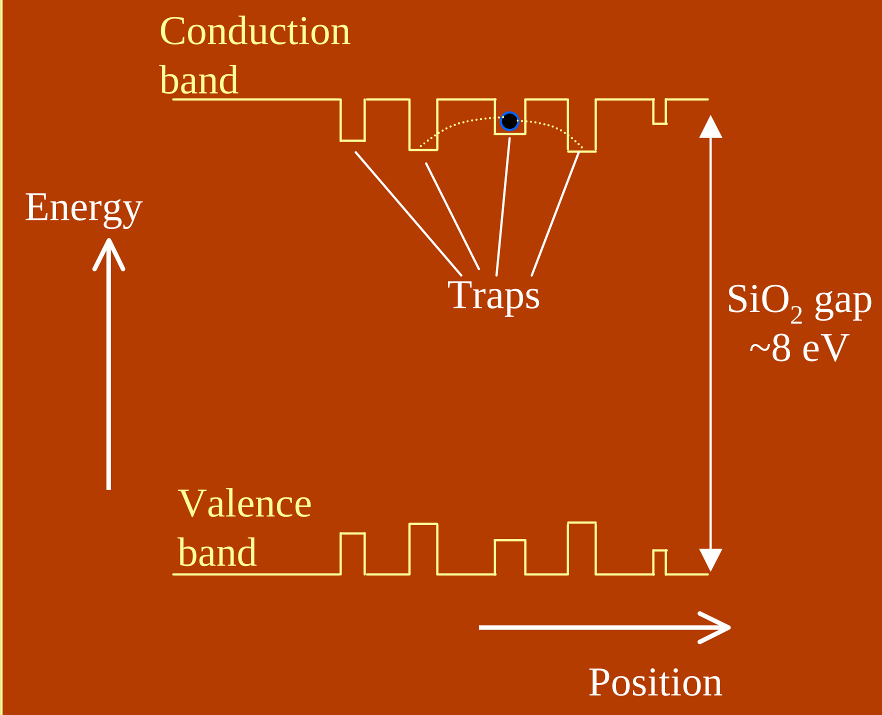
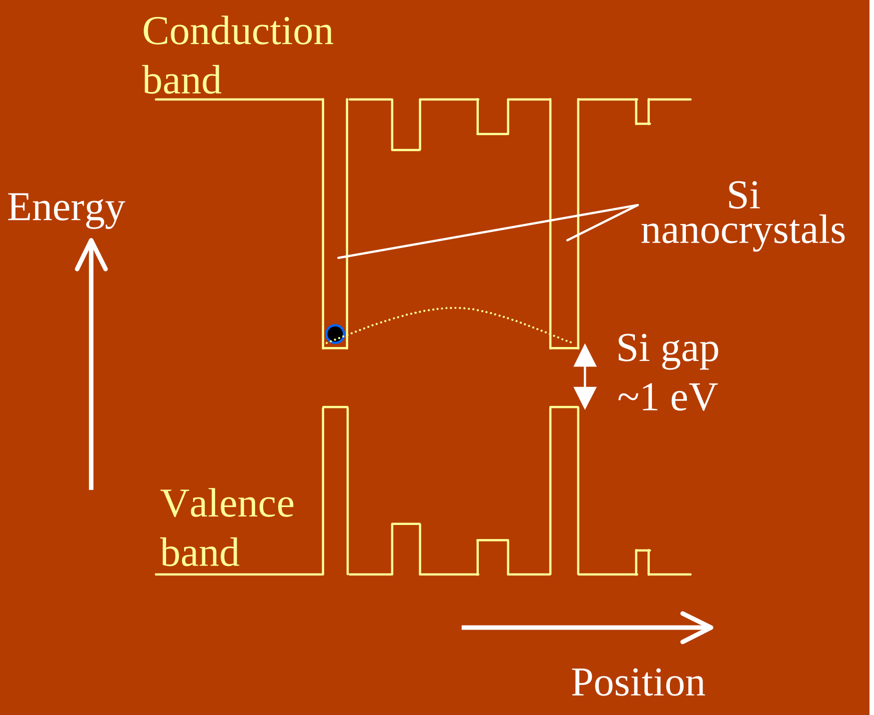
Lithography Si- nanostruc.

Tentative illustration of charge localization

Energetic diagrams

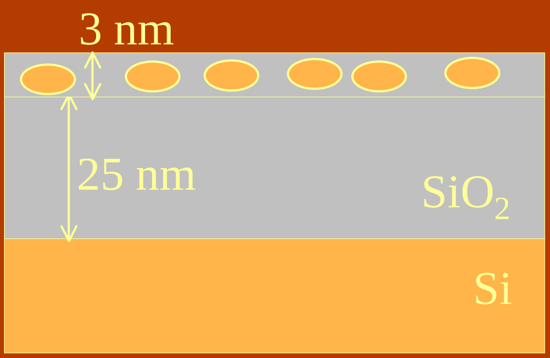


SiO_2 layer

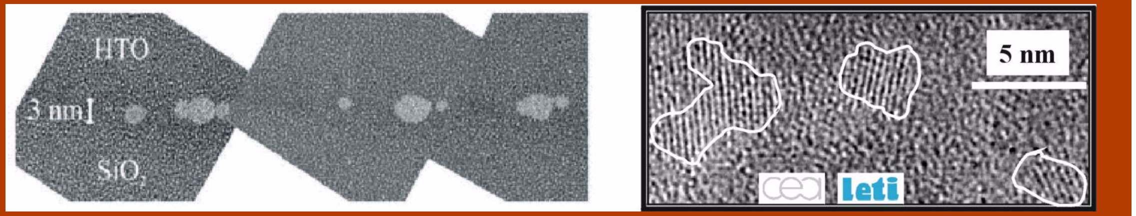


Si nanocrystals in SiO_2 layer

Silicon nanocrystals embedded in SiO_2



TEM pictures



Cross-section

Plane-view

Typical dimension: 3 nm

Density depends on x, varies from 3 x 10^{11} to 10^{12} cm^{-2}

⇨ 3 kinds of sample prepared, with varying densities

Fitting of the ellipsometric measurements provides:

Sample	Si(%)	SiO2(%)	Fraction x
E1	40	60	0,81
E2	8	92	1,67
E3	6	94	1,77

High Si-nc density

Low Si-nc density

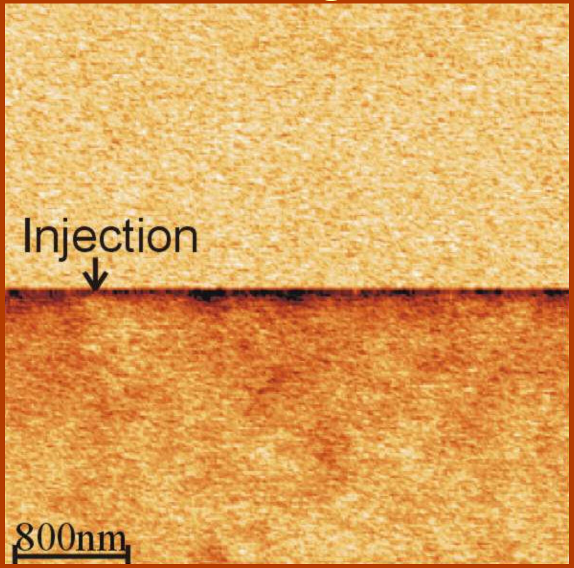
Very low Si-nc density

Silicon nanocrystals embedded in SiO_2

Sample E1: metallic behavior

Si = 40 %
SiO_2 = 60 %

EFM signal



- ➢ Charges spread away on a time scale of seconds

Si-nc touch one another, no confinement possible

EFM
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Min. force gradient
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Dynamic force curves
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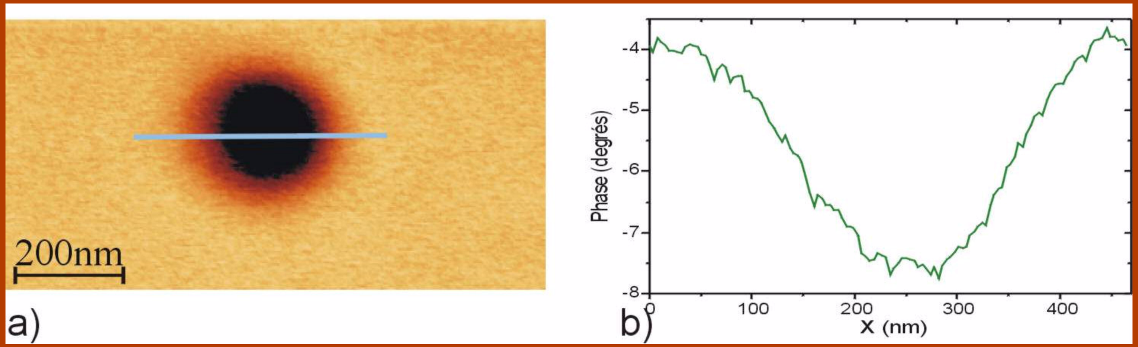
+ Electrostatic interaction

Charging experiments
SiO_2 layer
Si-nanocrystals in SiO_2
Lithography Si- nanostruc.

Silicon nanocrystals embedded in SiO_2

Sample E3: strongly confining behavior

Si = 6 %
SiO_2 = 94 % → Very low Si-nc density



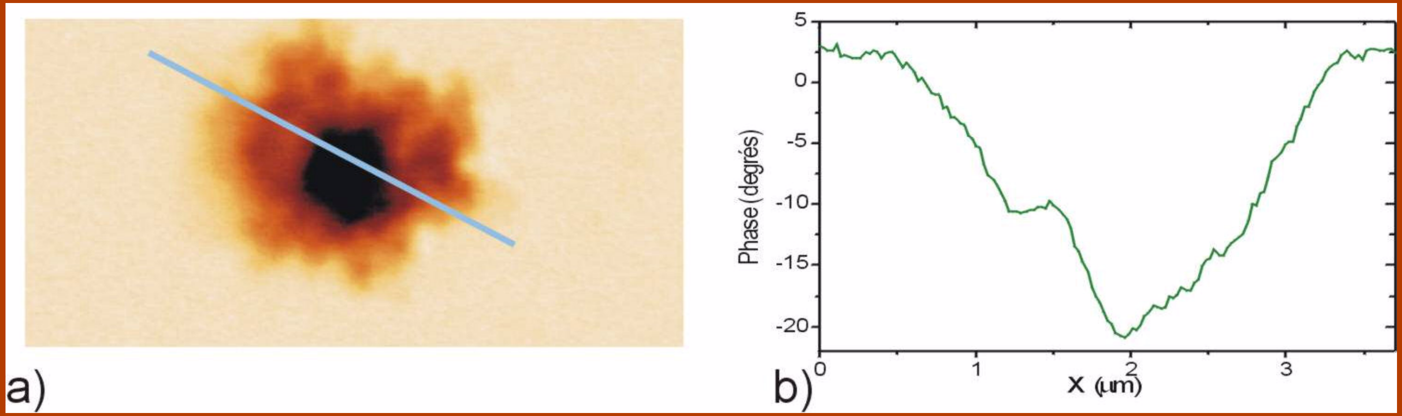
Circular shape of injected charges that does not evolve in time
Estimation of **one electron per nanocrystal**

Silicon nanocrystals embedded in SiO_2

Sample E2: partially confining behavior

Si = 8 %
SiO_2 = 92 % → Low Si-nc density

Charging conditions -10 V / 10 s



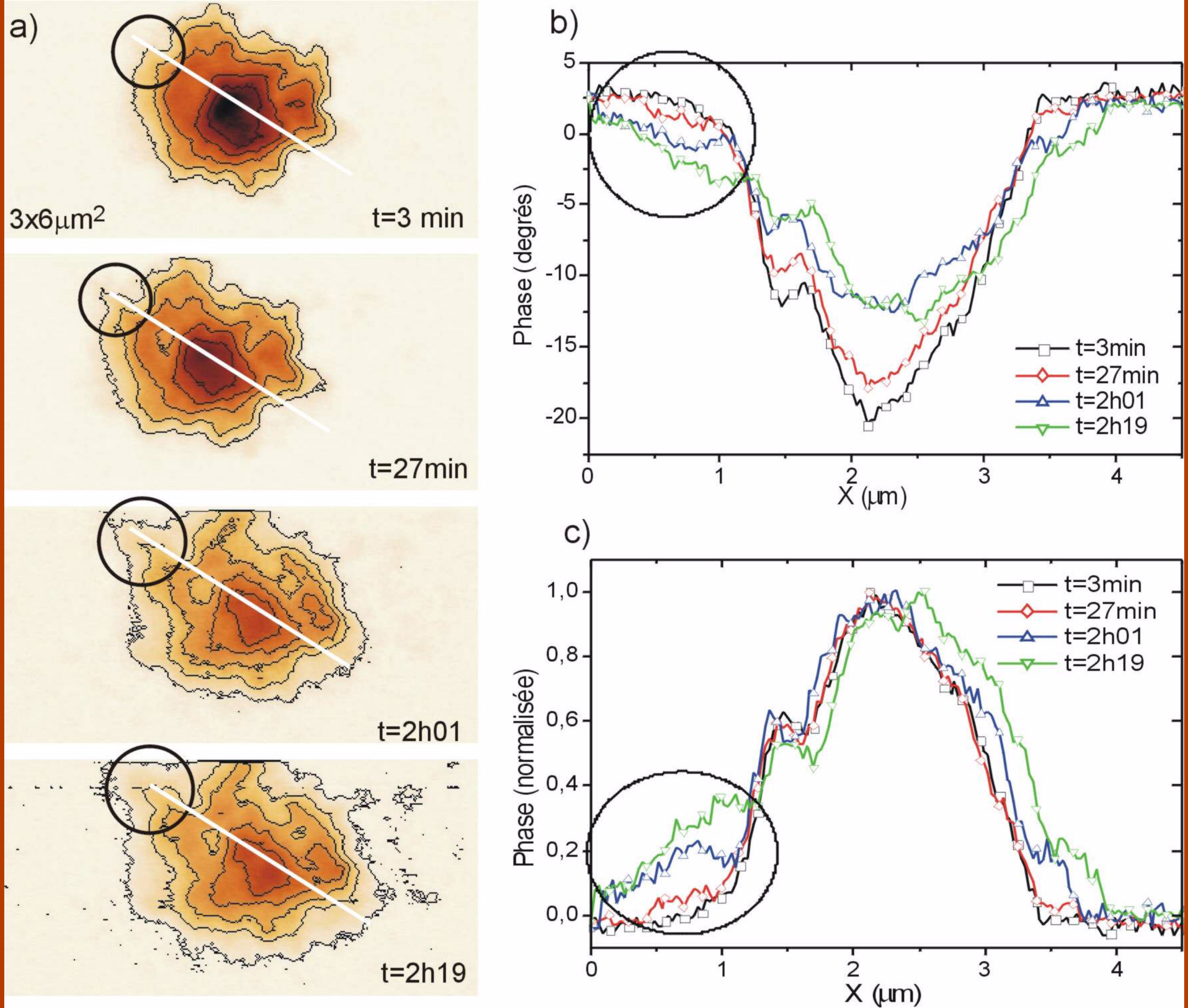
- ➢ Rough bordeline
- ➢ Inhomogeneous distribution of charges inside the electron cloud

⇓

Reflects disorder in the distribution of Si-nc at nanoscale

Sample E2: time evolution of the electron cloud

EFM images

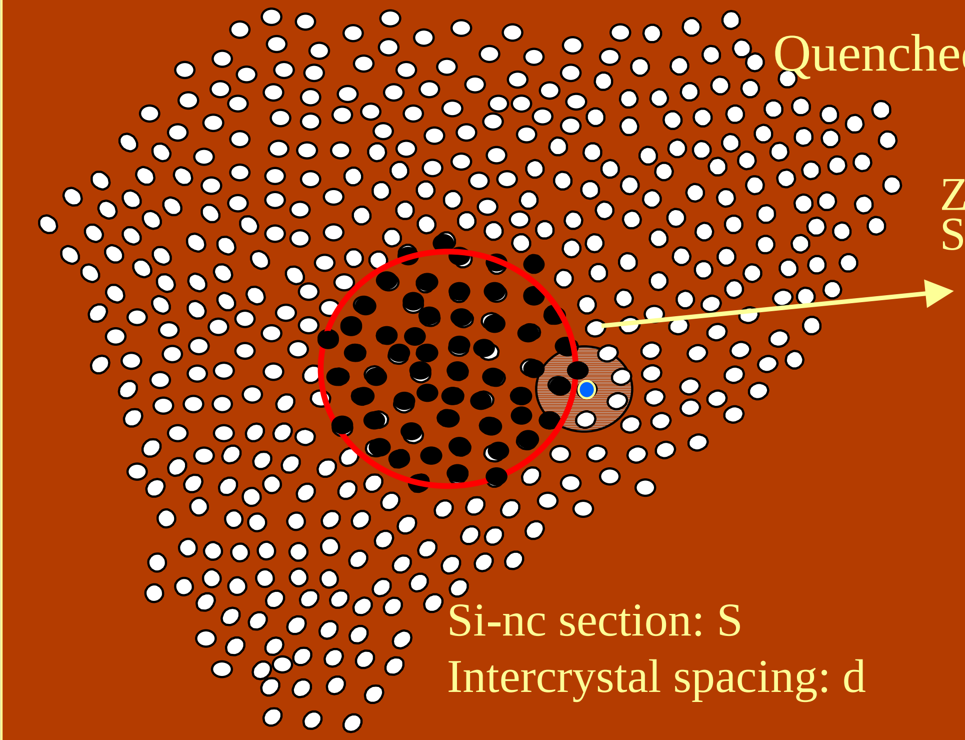


Profiles of EFM signal

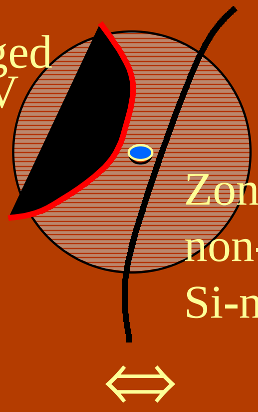
Normalized profiles

Irregular spreading of the charges, on a time scale of hours
= “kinetic roughening”

Sample E2: mechanism of charge spreading

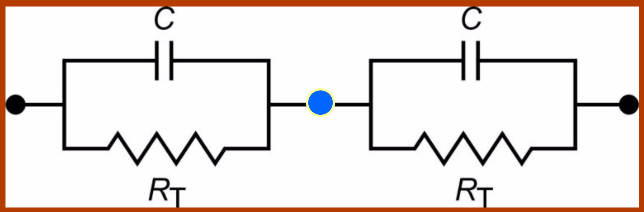

Quenched disorder

Si-nc section: S
Intercrystal spacing: d


Zone of charged Si-nc V~0.1 V

Zone of non-charged Si-nc: V=0

⇔



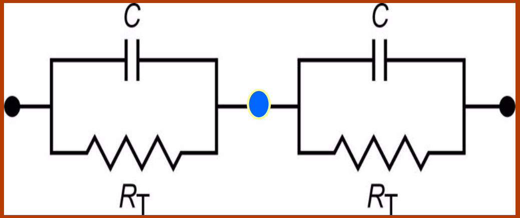
Electron transport is explained with the orthodox model of the **Single Electron Transistor** (SET) with V_{gate}=0

Passage from one Si-nc to another occurs through **tunneling**

⇓

Percolation threshold related to intercrystal distances (=density)

Sample E2: mechanism of charge spreading



$S = \pi r^2$ with $r \cong 3$ nm

$\rho_{SiO2} = 10^{14}$ to 10^{16} Ω cm

$d \cong 1$ nm

$C = \varepsilon_0 \varepsilon_{SiO2} S / d \sim 1$ aF

$R_T = \rho_{SiO2}\, d / S \sim 10^{19}\ \Omega$

Tunneling of the electrons in the frame of orthodox model

Transition rate $\Gamma = \tau^{-1}$ is:

$$\boxed{\Gamma = \frac{1}{R_T\, e^2} \frac{-\Delta F}{1 - \exp(\frac{\Delta F}{k_B T})}}$$

where:

- ΔF = f(ΔV, C) energy associated with the passage of one e^- from one Si-nc to its neighbor ~-80 meV

⇒ **$\Gamma = 5 \times 10^{-2}\ s^{-1}$ or $\boxed{\tau = 20\ s}$**

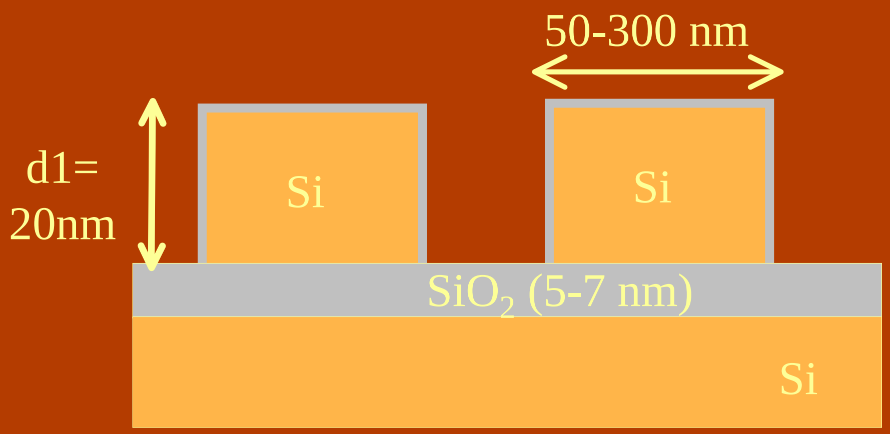
Progression of the borderline : 1 µm/hour

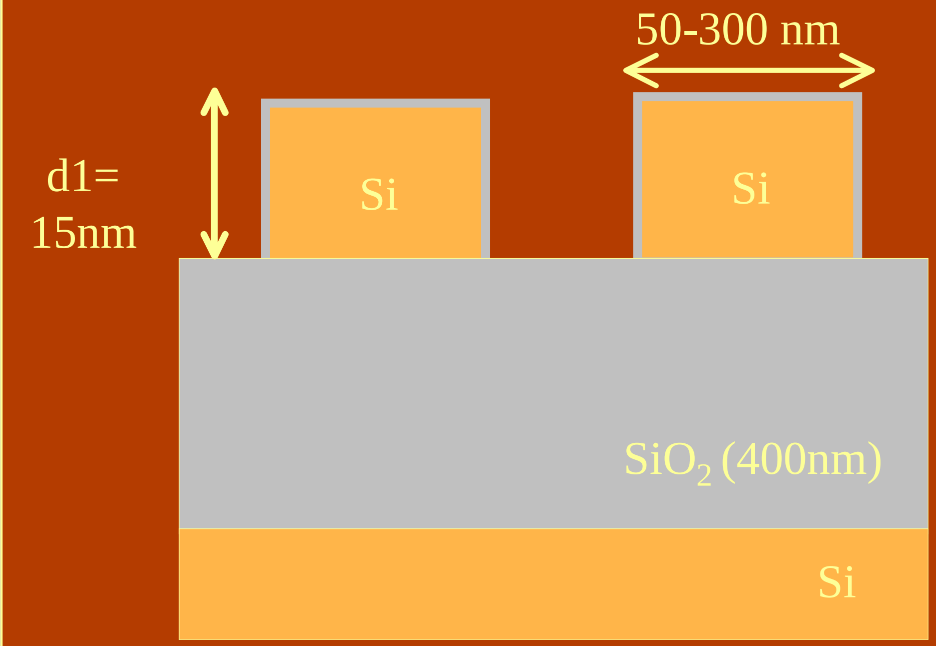
1 electron tunnels through ~200 Si-nc /hour = $\boxed{\text{1Si-nc / 20s!}}$

Paper accepted in Phys. Rev. B (Dec. 2004)

Silicon nanostructures made by e-beam lithography

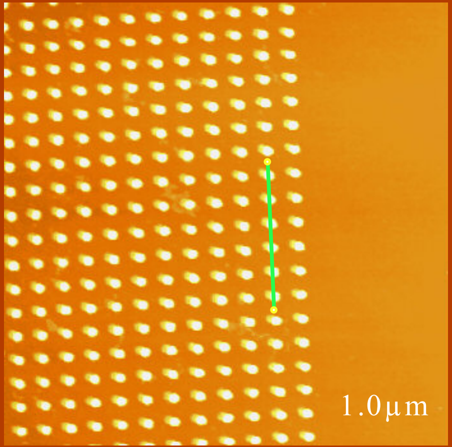
- Dots are polycrystalline Si deposited by LP-CVD
- 2 nm of SiO_2 is grown on top to protect the dots

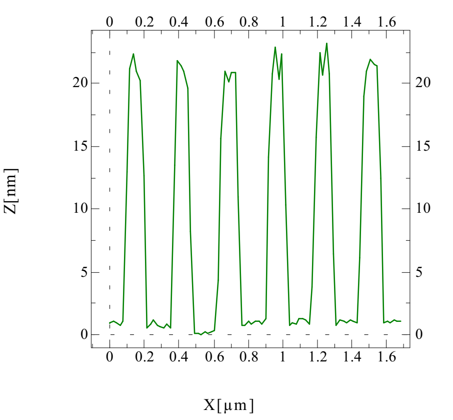




- Dots are monocrystalline Si made from SOI
- 2 nm of SiO_2 is grown on top to protect the dots

AFM characterization

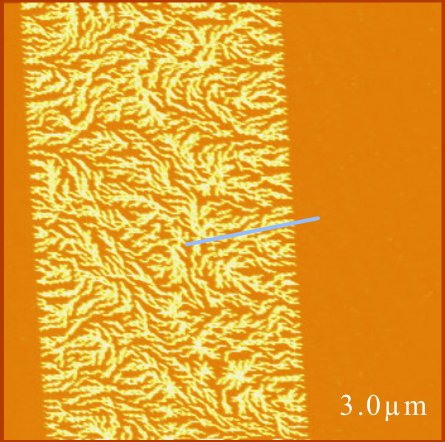


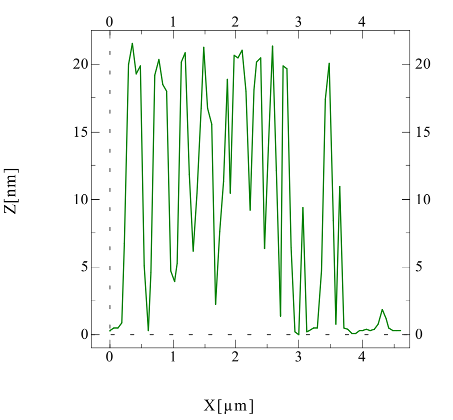


Most Si nanostructures are well-defined...

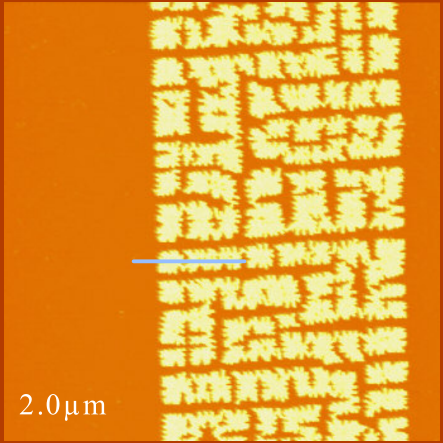
100 nm in diameter dots

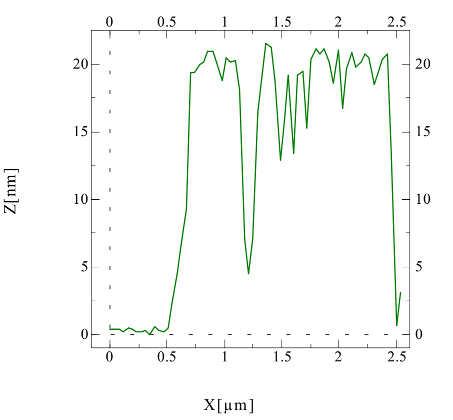
… but some are more extravagant.





50 nm in diameter dots





Influence of the oxide thickness

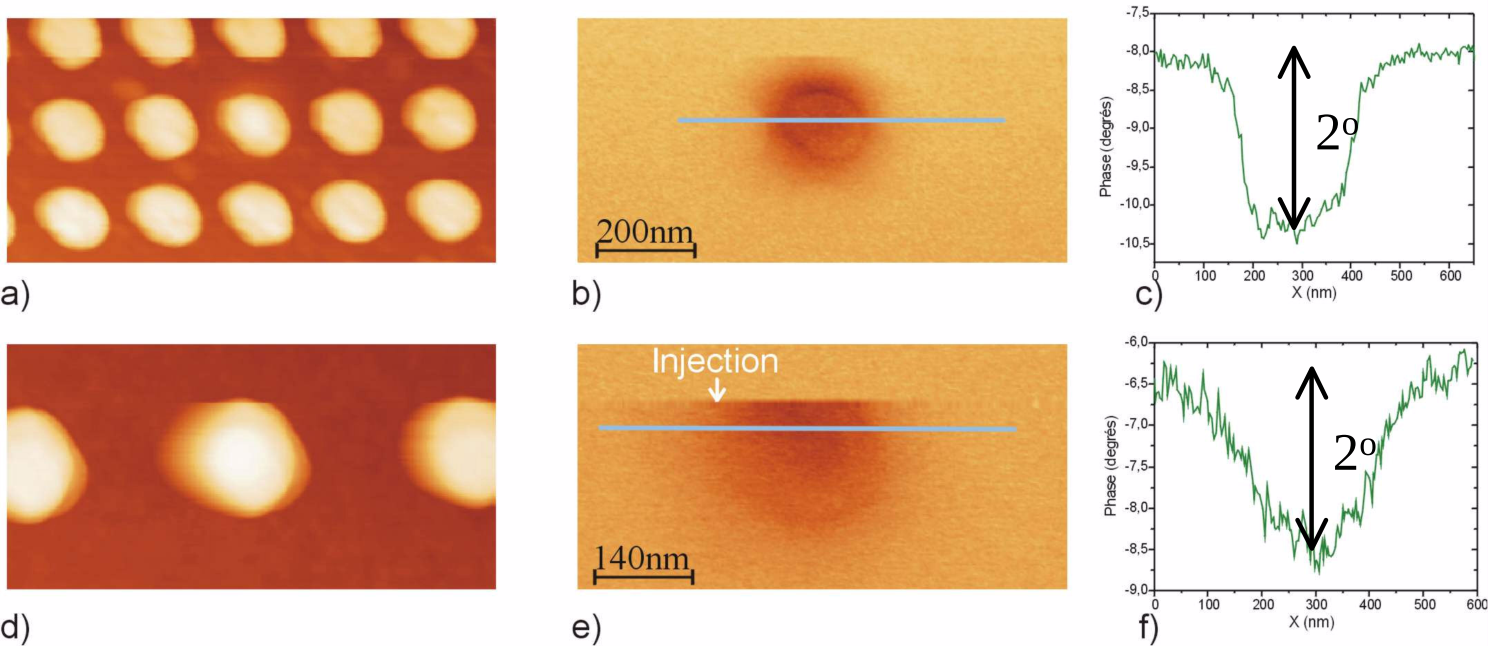
Charging conditions: -8V / 5s

Height

EFM signal

Thin oxide
(7 nm)

Thick oxide
(400 nm)



Considering the expression of charge vs. phase shift:

$$q = \sqrt{\frac{\delta\varphi \cdot k \left(z_0 + \frac{d}{\varepsilon_{SiO2}} + \frac{d1}{\varepsilon_{Si}} \right)^3 \varepsilon_0 A}{Q \left(\frac{d}{\varepsilon_{SiO2}} + \frac{d1}{\varepsilon_{Si}} \right)^2}}$$

For the same recorded phase shift,
there are **7x** less charges on thick oxide

EFM

Injection and detection

Min. force gradient

Modelling

Charge estimation

Limits

Dynamic force curves

Coupling to higher modes

Analytical treatment

+ Electrostatic interaction

Charging experiments

SiO_2 layer

Si-nanocrystals in SiO_2

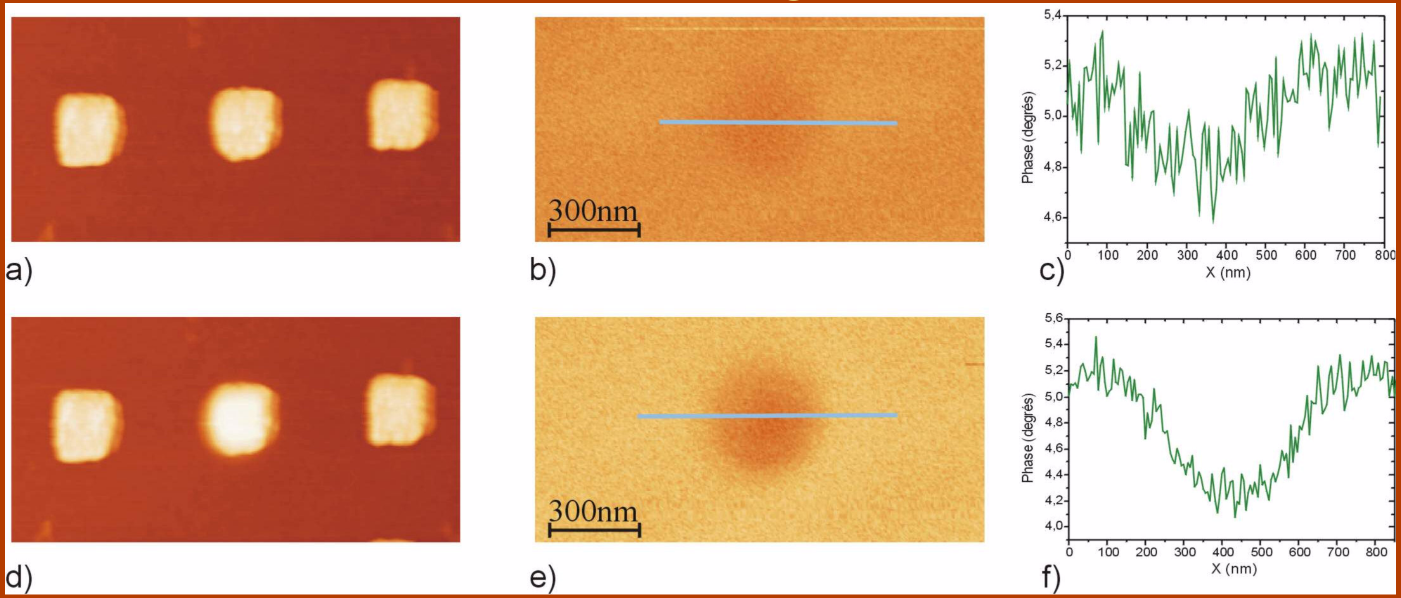
Lithography Si- nanostruc.

Existence of a voltage threshold for injection of charges

On thin-oxide sample:

Height

EFM signal



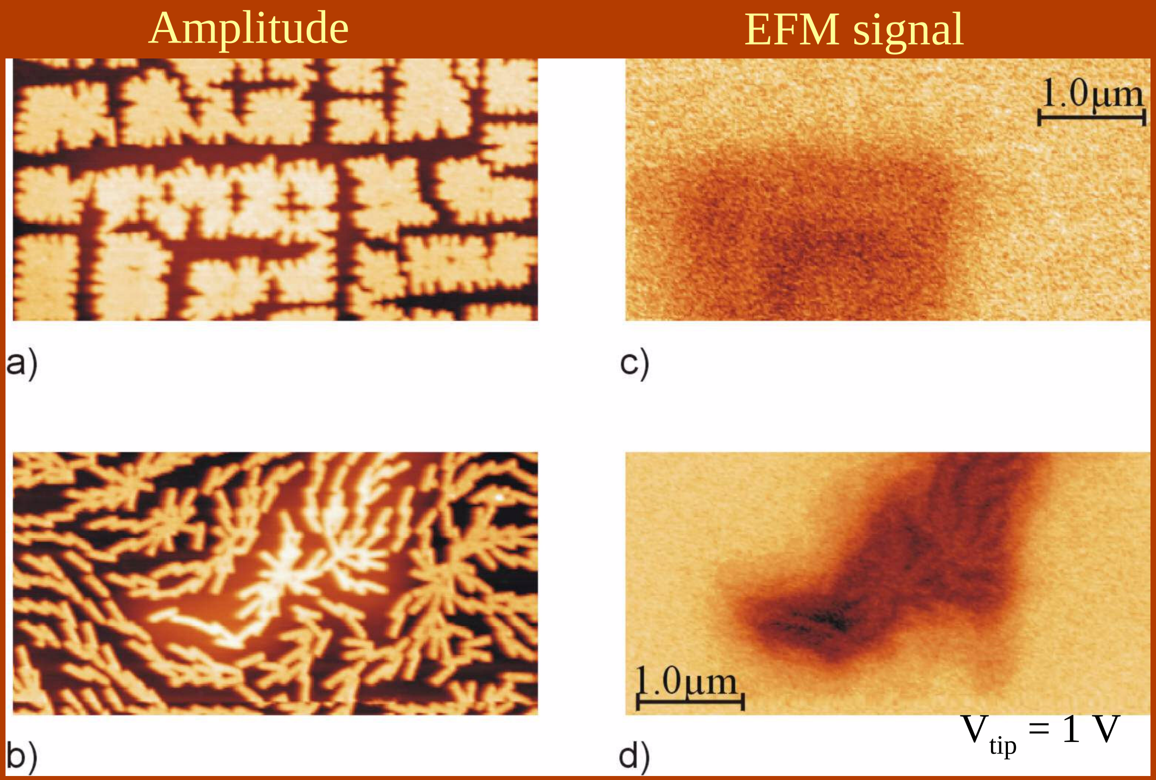
V=-5 V
t=10 s

V=-6 V
t=10 s

→ Minimum electric field of ~3 x 10^8 V.m^{-1} is required

Propagation of the charges inside a ramified structure

Thin-oxide sample
Charging conditions: -10 V / 10 s



Injection is point-like
Charges extend immediately over several microns

EFM

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Charging experiments

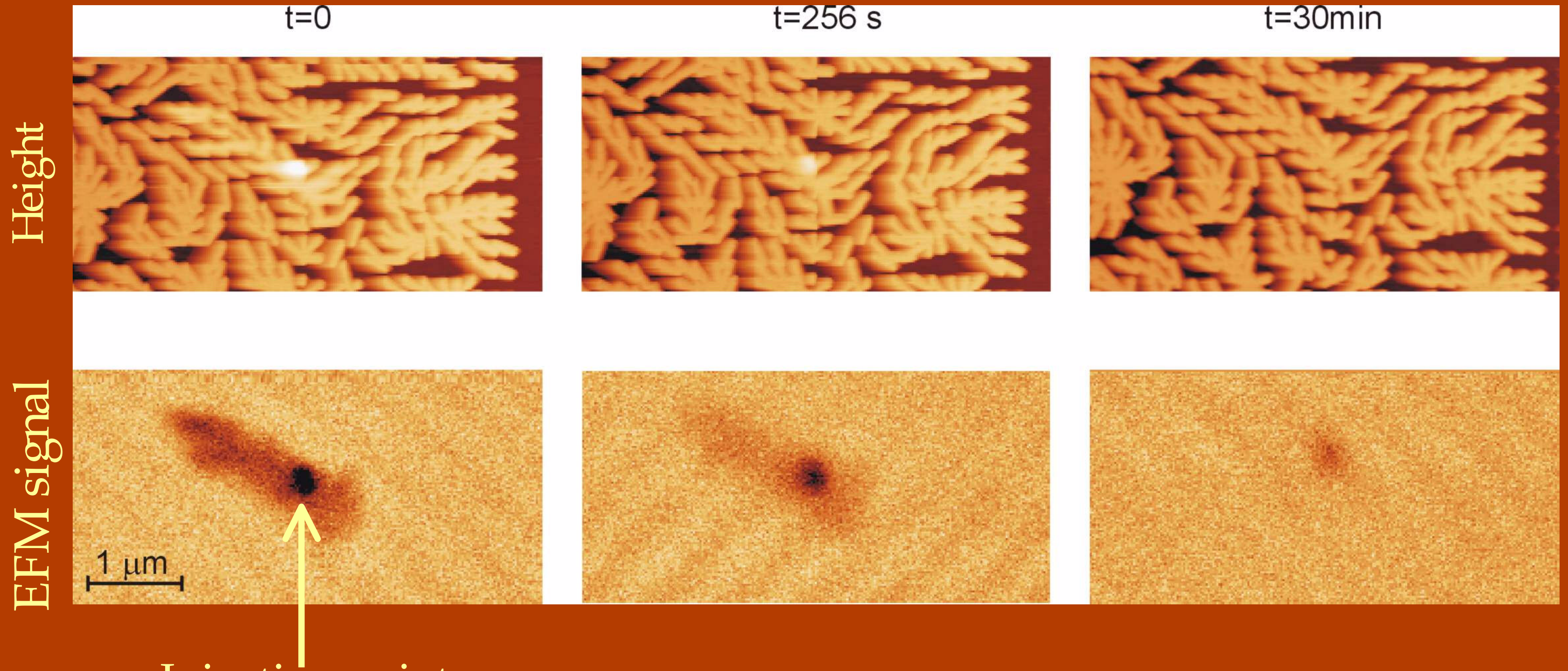
SiO_2 layer

Si-nanocrystals in SiO_2

Lithography Si- nanostruc.

Trapping of charges in the top oxide

Charging conditions: -7 V/ 10 S



Injection point

Good quality oxide (“Rapid Thermal Oxide”)

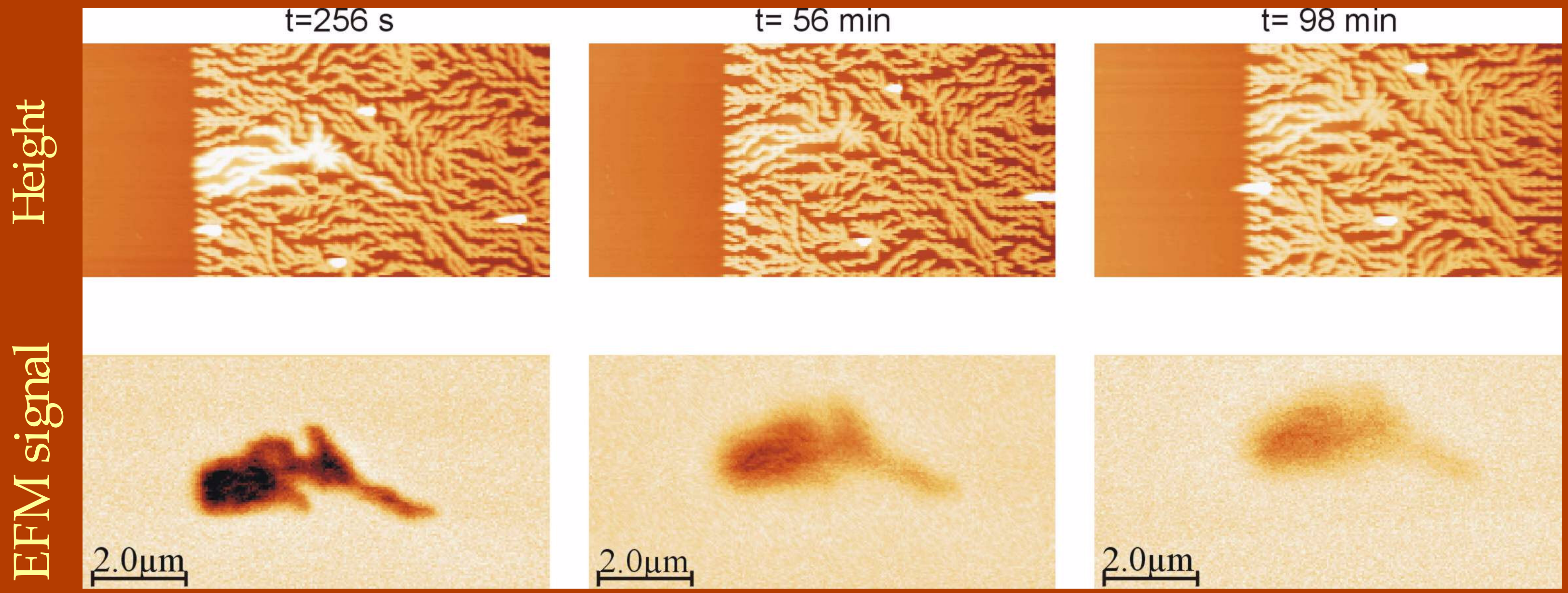
↓

traps charges for more than 30 minutes

De-charging of the ramified structures

Thin oxide (7 nm)

Charging conditions: -10 V/ 10 s



Homogeneous de-charging of the structure, although 7 nm of oxide prevent direct tunneling → De-charging mechanisms?

Strong repulsion between the electrons
(electronic density is high: ~10^{17} cm^{-3})

Existence of a Wigner crystal
=
ordering of the electrons on a regular lattice?

Silicon nanocrystals embedded in SiO_2

Sample E3: strongly confining behavior

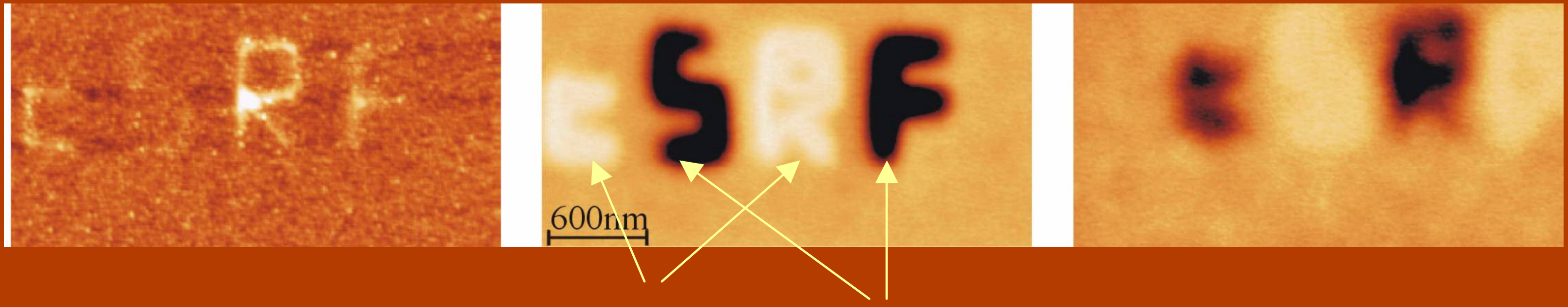
Si = 6 %
SiO_2 = 94 %

AFM image

t=0

EFM images

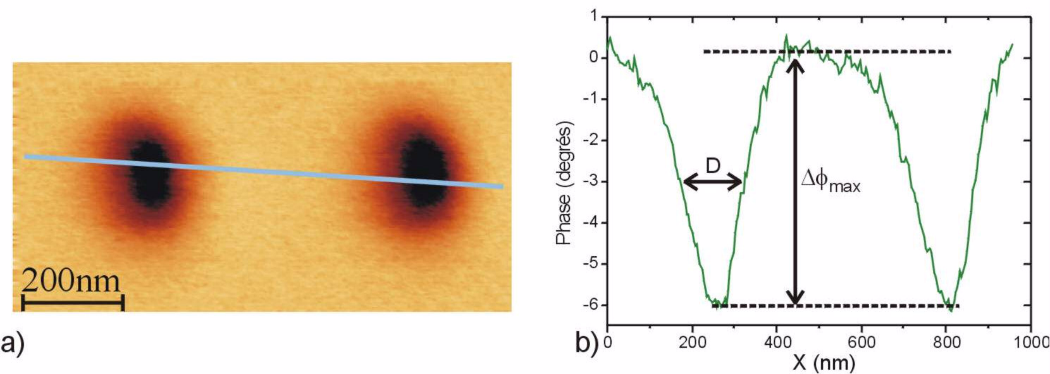
t=4 hours



-6 V

+6 V

⇒ Very long retention time



Circular shape of injected charges that does not evolve in time
Estimation of **one electron per nanocrystal**

Conclusion

Electrostastic Force Microscopy in dry atmosphere:

- ✓ Powerful method to characterize electrical properties at the nanoscale
- ✓ Charge resolution: a few tens elementary charges
- ✓ Analysis of the non-linear tip-sample interaction

Semiconducting nanostructures:

- ✓ Reference SiO_2 sample shows low charge retention and low charge density
- ✓ Collective behavior of Si-nanocrystals show 3 regimes:
  - ▪ metallic
  - ▪ intermediate: observable spreading
  - ▪ confining
- ✓ Individual behavior of Si-nanostructures

Perspectives:

Need for better resolution (charge, drift)

→ future experiments under vacuum, low temperatures
= single electron detection

Acknowledgements

For the experiments

Henk-Jan Smilde	CEA Grenoble/LETI
Martin Stark	LEPES / LSP
Julien Pascal	ESRF
Frederio Martin	ESRF
Charlène Alandi	ESRF
Emilie Dubard	ESRF
Florence Marchi	UJF / LEPES-CNRS
Fabio Comin	ESRF
André Barski	CEA Grenoble / DRFMC
Joël Chevrier	ESRF / UJF / LEPES CNRS

For the samples

Denis Mariolle	CEA Grenoble/LETI
Nicolas Buffet	CEA Grenoble/LETI
Pierre Mur	CEA Grenoble/LETI