Introduction à la Microscopie à Force Electrostatique

Action Nationale de Formation DFRT KFM | 7-8 Novembre 2017

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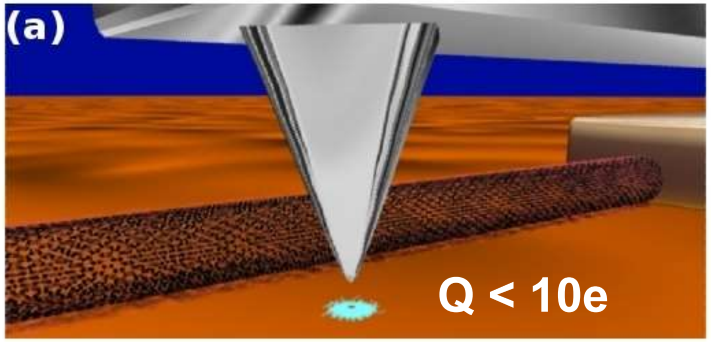
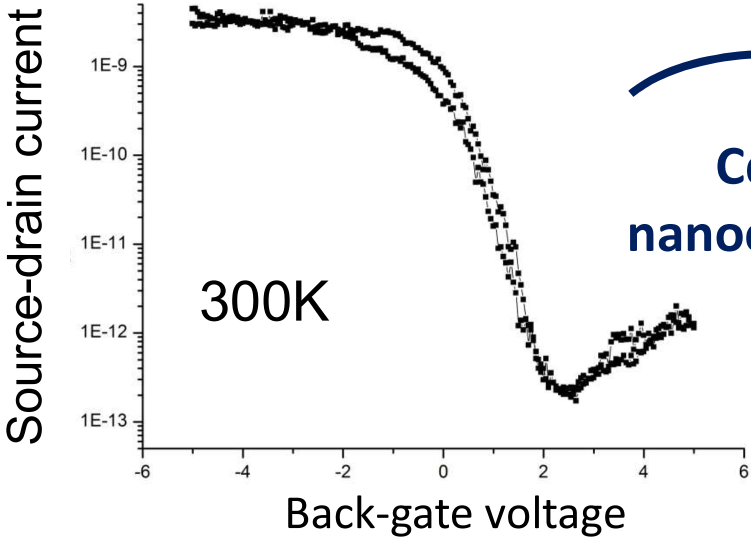
A few motivations

EX 1 : Imaging the operation of CNT-FETs as charge sensors

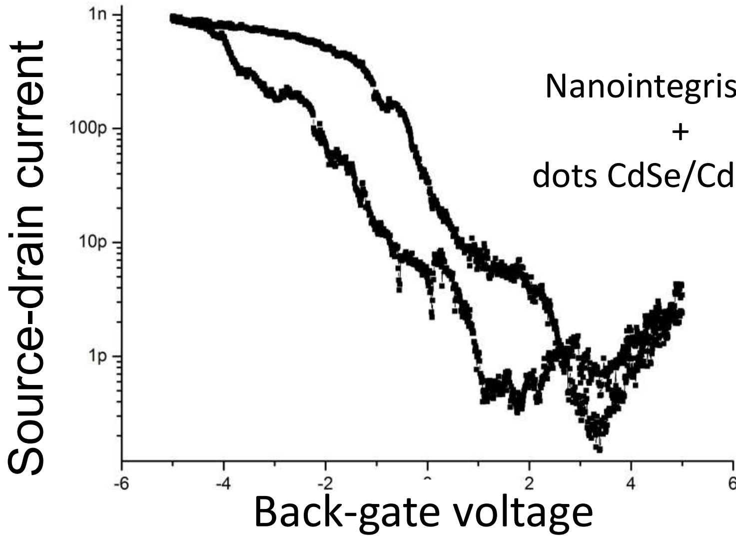
[D. Brunel et al., ACS Nano 2010]



EX 2 : coupled CNTFETs and nanocrystals



+ CdSe nanocrystals



Nanointegris tube
+
dots CdSe/CdS 5nm

un(re)solved using AFM – Nanoletters 2015

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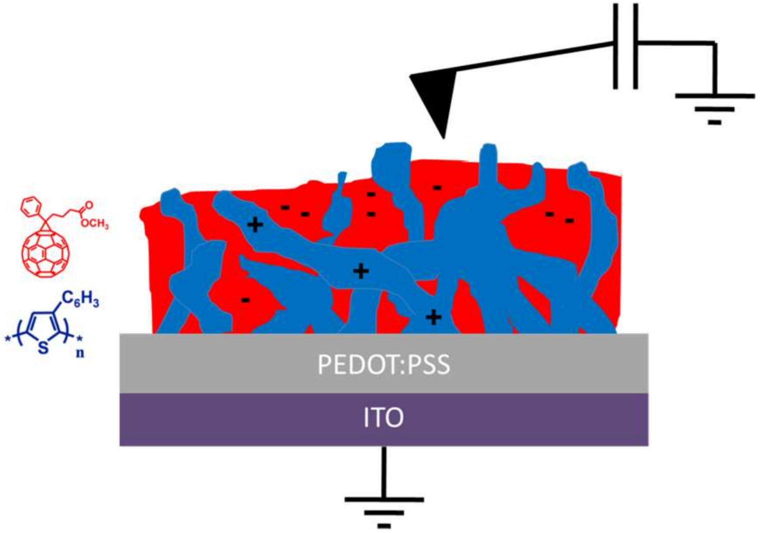
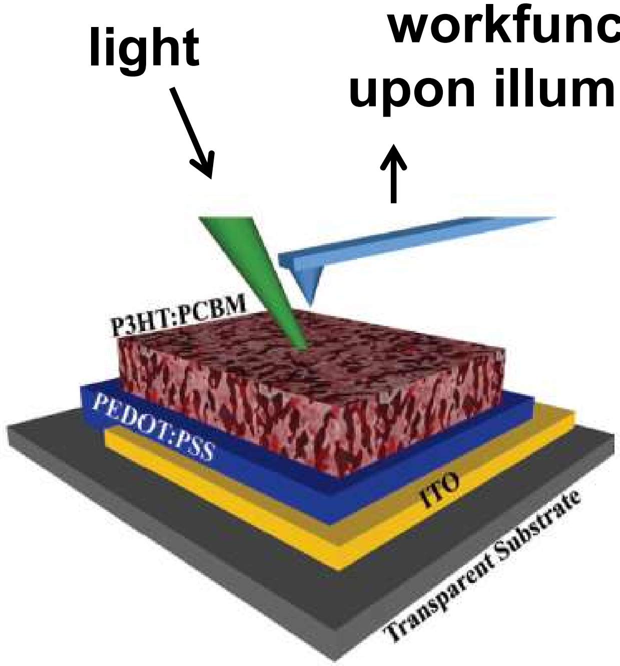
A few motivations

EX 3 : photovoltaic materials



P3HT:PCBM blend
molecular D/A junction

Change in local effective workfunctions upon illumination



cf Ł. Borowik / B. Grévin

EFM	**KPFM**
Electrostatic Force Microscopy	**Kelvin Probe Force Microscopy**
Measurement of electrostatic force gradient	*Compensation of electrostatic force (gradient)*
Units : Hz or N/m	Units : V

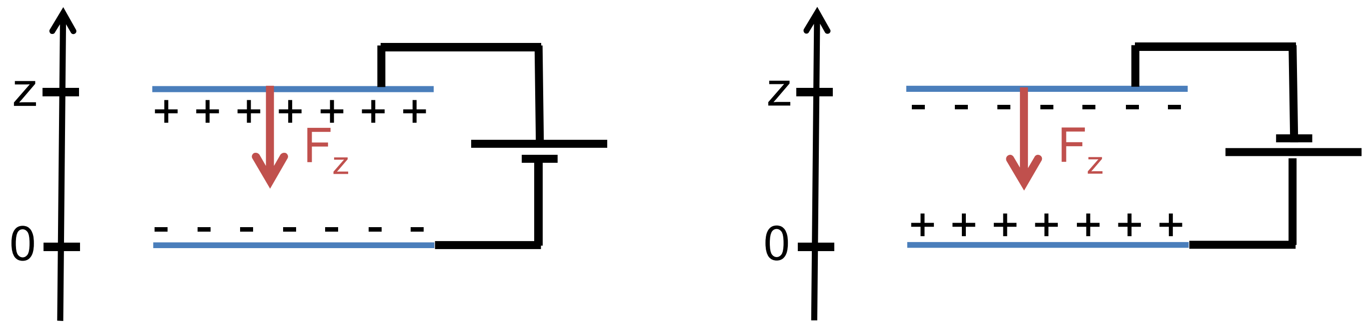
Charge detection ⟷ *Probing local surface potential*

SOMMAIRE

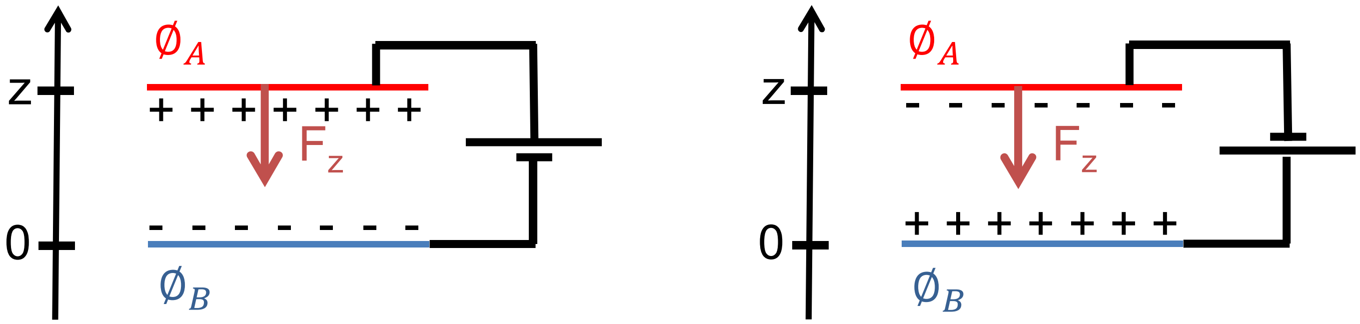
Energy stored in a capacitor ½ C V²

Attractive force between capacitor plates

$$F_z = +\frac{1}{2}\underbrace{\frac{\partial C}{\partial z}}_{<0} V^2 < 0$$



Capacitor with 2 ≠ plates



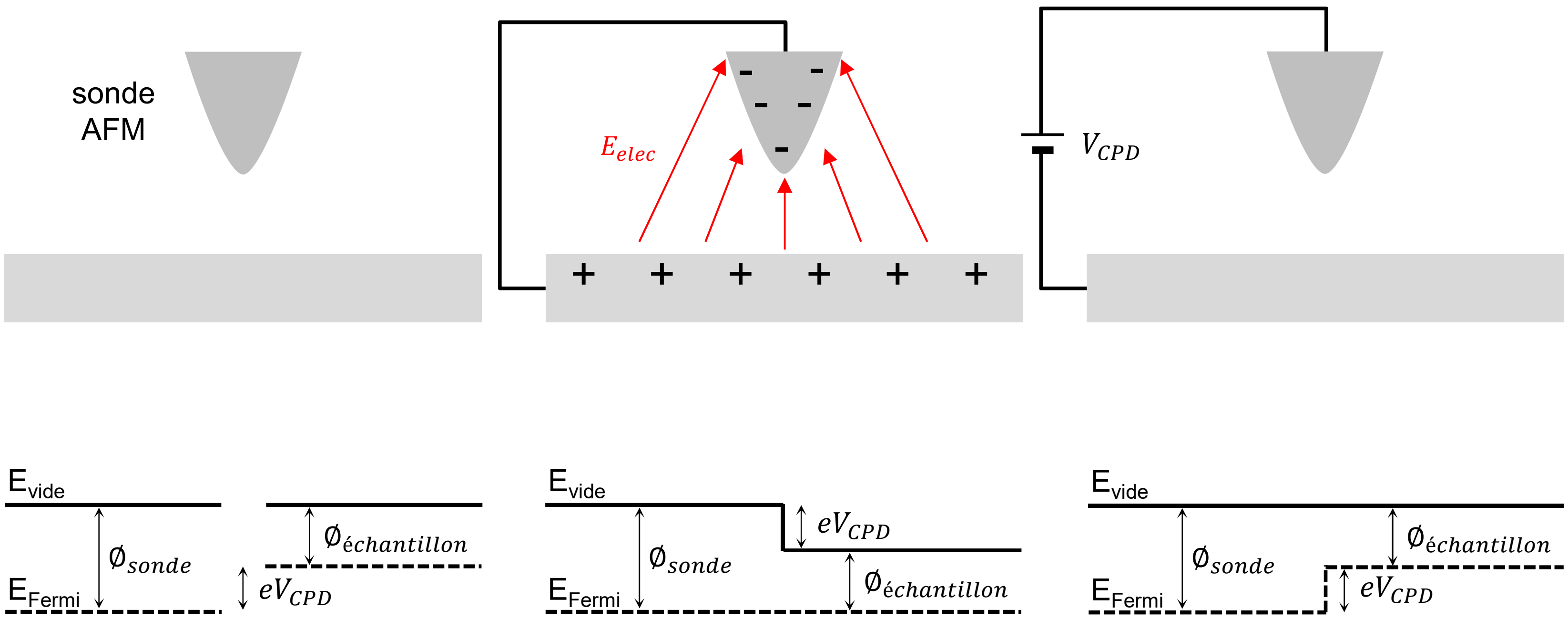
Contact potential difference $V_{CPD} = \frac{\emptyset_A - \emptyset_B}{|e|}$

$$F_z = +\frac{1}{2}\frac{\partial C}{\partial z}\left(V \pm V_{CPD}\right)^2 < 0$$

V applied to plate $A \rightarrow -$

V applied to plate $B \rightarrow +$

FORCE ÉLECTROSATIQUE (CAPACITIVE) EN AFM

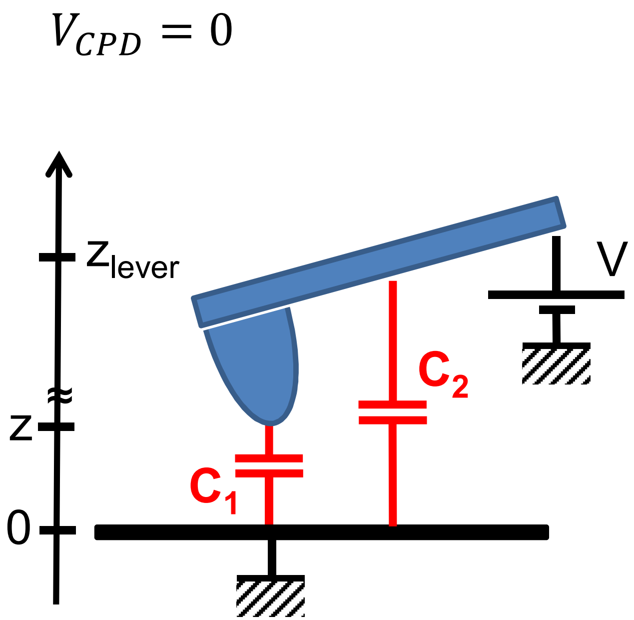


$$V_{CPD} = \frac{\emptyset_{sonde} - \emptyset_{échantillon}}{|e|}$$

Différence de potentiel de contact

SOMMAIRE

Charge {≡ capacitance}



$$Q = C.V$$

Tip apex

$C_1 \approx 4\pi\varepsilon_0 R_{apex}$

R_{apex}=20nm

$z >> R_{apex}$

↓

$Q_1 \approx 20$ e

Cantilever

$C_2 \approx \varepsilon_0 \; S_{lever} \; / \; z_{lever}$

S_{lever} = 30µm×100µm

z_{lever} =15µm

↓

$Q_2 \approx 10^4$ e

[V=1V]

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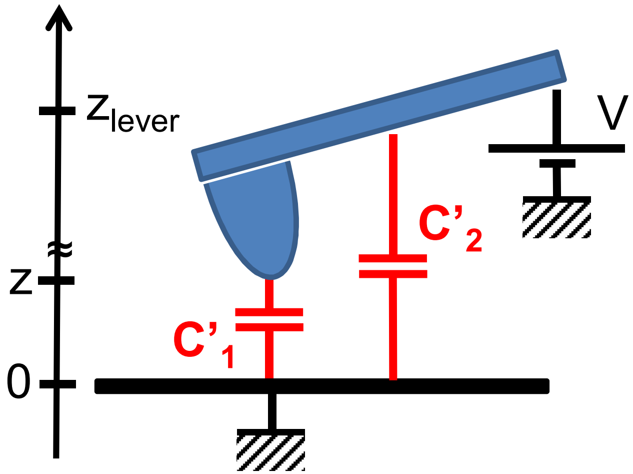
Electrostatic Force {≡ capacitance gradient}

$V_{CPD} = 0$



Attractive force between capacitor plates

$$F_z = +\frac{1}{2}\frac{\partial C}{\partial z}V^2 < 0$$

Tip apex	Cantilever
$|dC_1/dz| \approx 4\pi\varepsilon_0 R^2_{apex}/z^2$	$|dC_2/dz| \approx \varepsilon_0\ S_{lever}\ /\ z^2_{lever}$
R_{apex}=20nm z=100nm	S_{lever} = 30µm×100µm z_{lever} =15µm
$F_1 \approx$ 5 pN	$F_2 \approx$ 100 pN

[V=1V]

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Force gradient detection

$$F_z = F_z\Big|_{z_0} + (z - z_0)\,\frac{\partial F_z}{\partial z}\Big|_{z0}$$

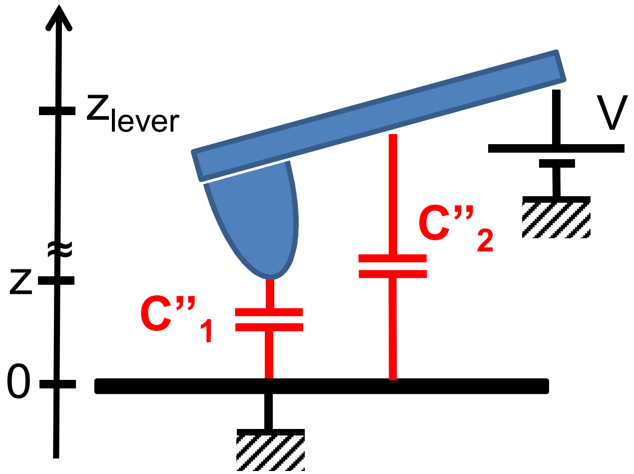
Frequency shift : $\Delta f \cong -\frac{f_\infty}{2k}\frac{\partial F_z}{\partial z}\Big|_{z0}$

- Here: long-range forces [ambient air / UHV]

- Short-range electrostatic forces disregarded here

Force gradient {≡ capacitance 2nd derivative}

$V_{CPD} = 0$



force gradient $\frac{\partial F_z}{\partial z} = +\frac{1}{2}\frac{\partial^2 C}{\partial z^2} V^2$

Tip apex

$d^2C_1/dz^2 \approx 8\pi\varepsilon_0 R^2_{apex}/z^3$

R_{apex}=20nm
z=100nm

Cantilever

$d^2C_2/dz^2 \approx 2\varepsilon_0 \; S_{lever} \; / \; z^3_{lever}$

S_{lever} = 30µm×100µm
z_{lever} =15µm

[V=1V] $dF_1/dz \approx 10^{-4}$ N/m $dF_2/dz \approx 2.10^{-5}$ N/m

dF_1/dz (apex) exceeds dF_2/dz (cantilever)

MESURE DU GRADIENT DE FORCE ÉLECTROSTATIQUE ?

- Par le décalage de phase (mécanique):

$$\Delta\varphi \cong -\frac{Q}{k}\frac{\partial F_z}{\partial z}\bigg|_{z0} = -\frac{Q}{2k}\frac{\partial^2 C}{\partial z^2}\bigg|_{d}(V \pm V_{CPD})^2$$

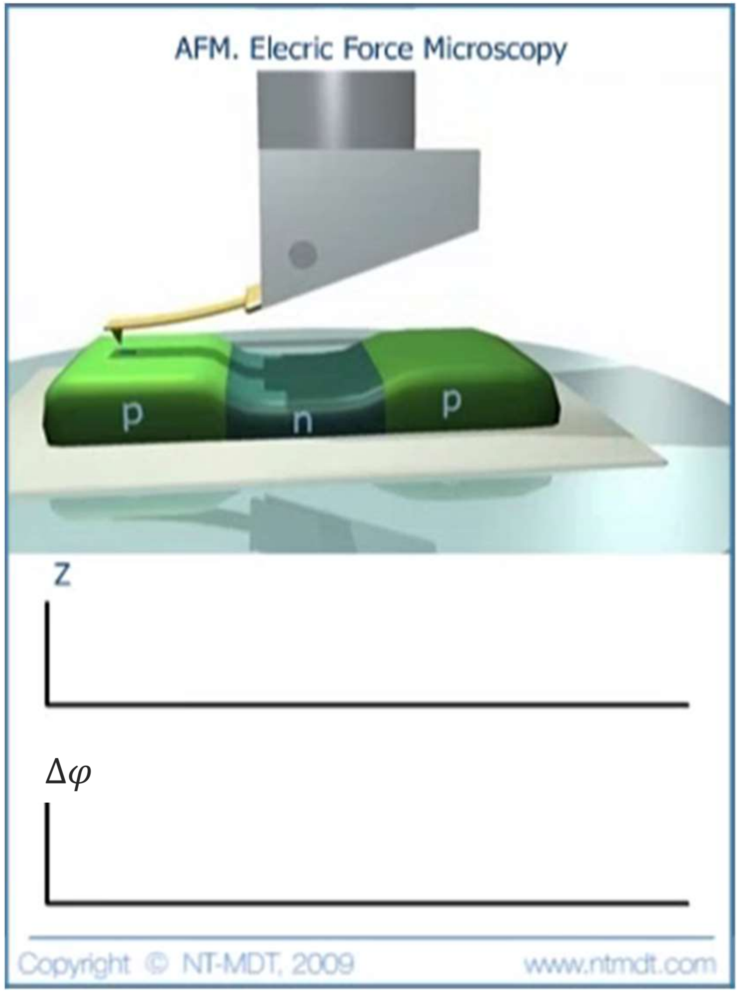
- Par le décalage de fréquence:

$$\Delta f \cong -\frac{f_\infty}{2k}\frac{\partial F_z}{\partial z}\bigg|_{z0} = -\frac{f_\infty}{2k}\frac{\partial^2 C}{\partial z^2}\bigg|_{d}(V \pm V_{CPD})^2$$

$$\frac{\Delta\varphi}{\Delta f} = \frac{2Q}{f_\infty}$$

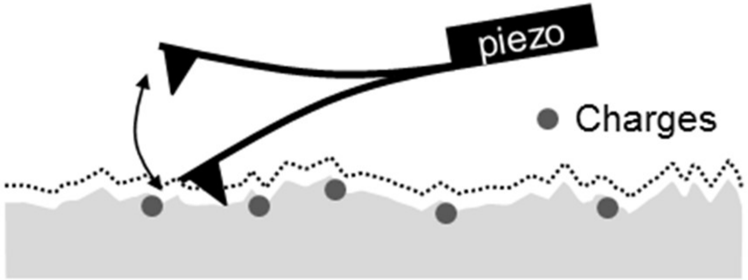
EFM ≡ mesure du décalage de fréquence ou de phase

ACQUISITION EFM



ACQUISITION EFM

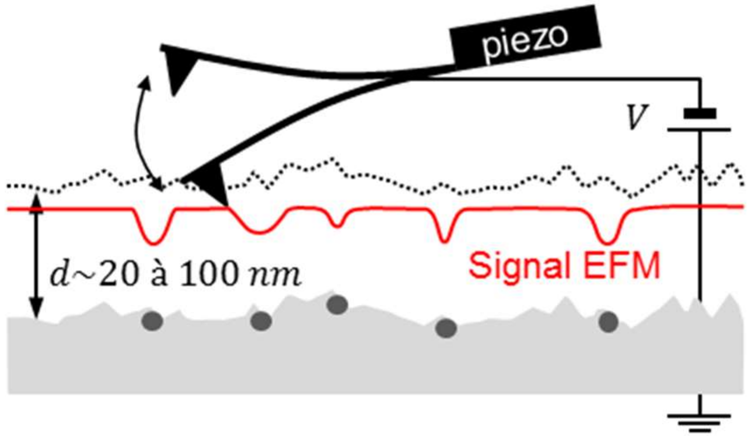
1er passage : topographie



Excitation mécanique à la résonance du levier f_0

Tapping (Air) ou nc-AFM (Vide, UHV)

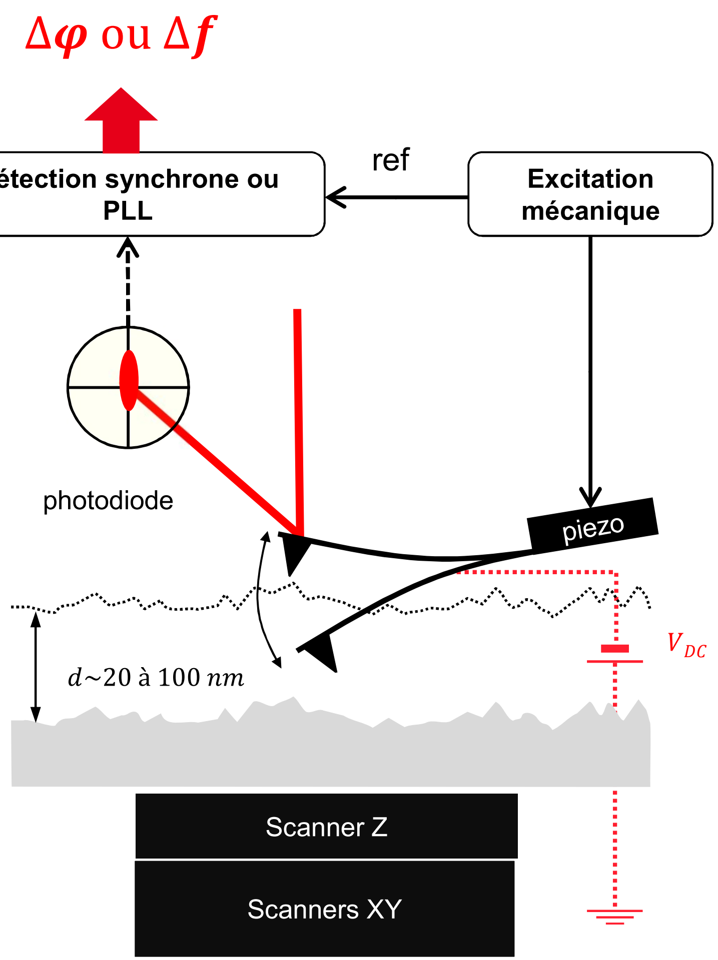
2ème passage : EFM



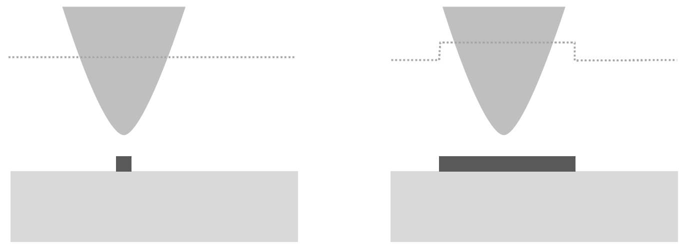
Excitation mécanique à la résonance du levier f_0 + tension continue V_{DC}

Hauteur de lift (20 à 100 nm)

EN PRATIQUE

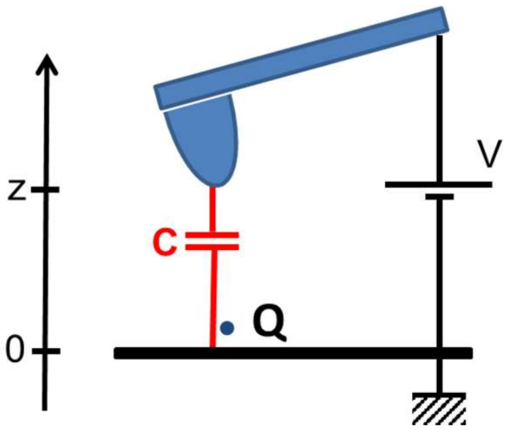


- Lors du mode lift : **excitation mécanique à la résonance du levier f_0 + tension continue V_{DC}**
- Modes : imagerie ou spectroscopie
- Distance ou altitude constante ?



SOMMAIRE

Charge in a capacitor



V_Q effective surface potential

$$\frac{\partial F_z}{\partial z} = +\frac{1}{2}\frac{\partial^2 C}{\partial z^2}\left[(V - V_{CPD})^2 - 2(V - V_{CPD})V_Q + V_Q^2 \right]$$

Capacitive force gradient $\propto V^2$

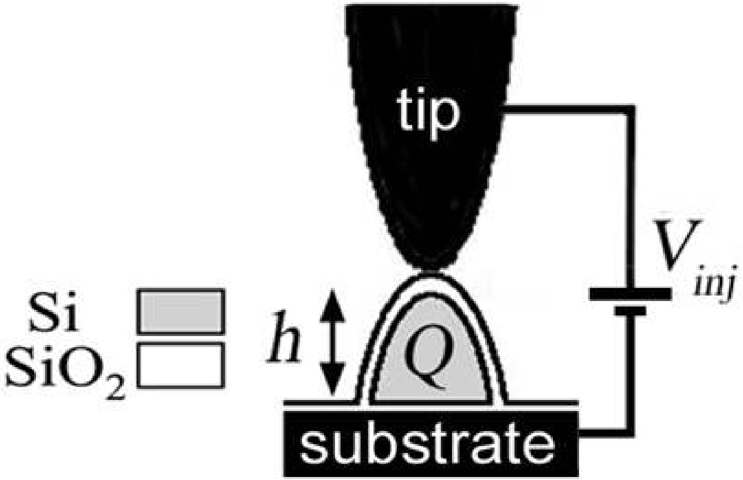
Charge interaction Q $\Leftrightarrow$ *probe charge* $\propto Q.V$

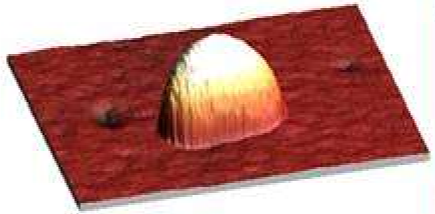
determination of the sign of the surface charge Q

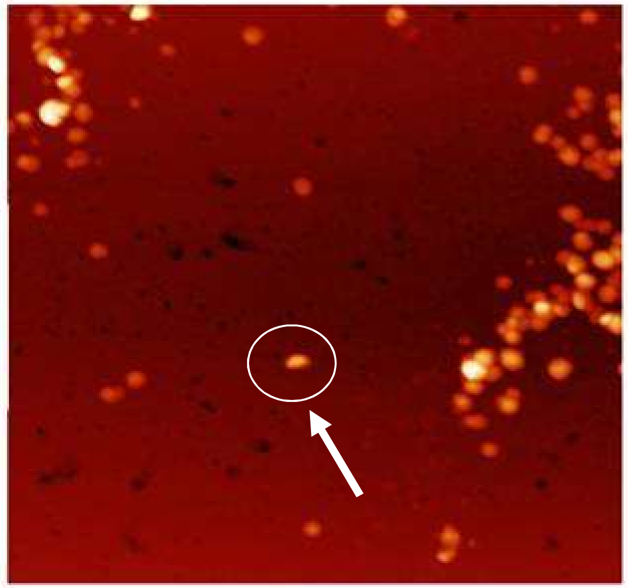
Image charge effect $\propto Q^2$

How to discriminate these electrostatic contributions ?

Example 1 : Charge manipulation







AFM Image 2500 x 2500 nm²

Contact force : a few nN

Charge retention time : of few 10 min (dry N_2)

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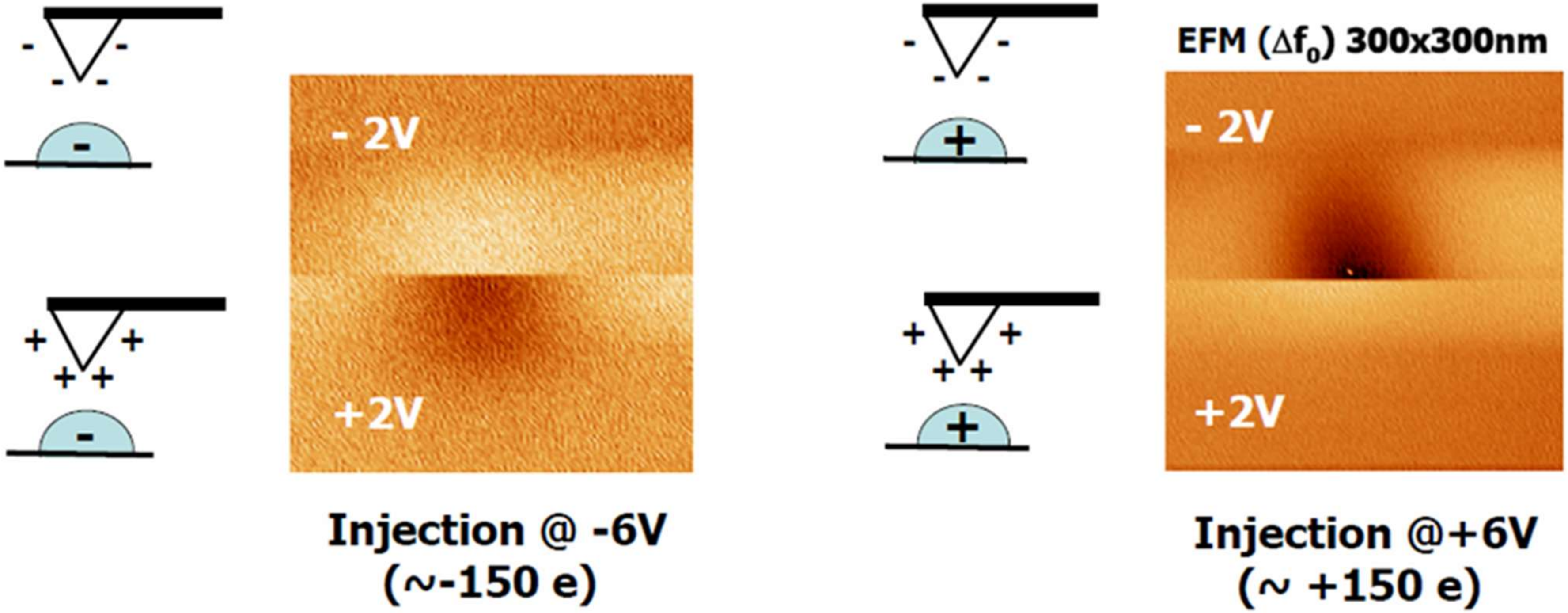
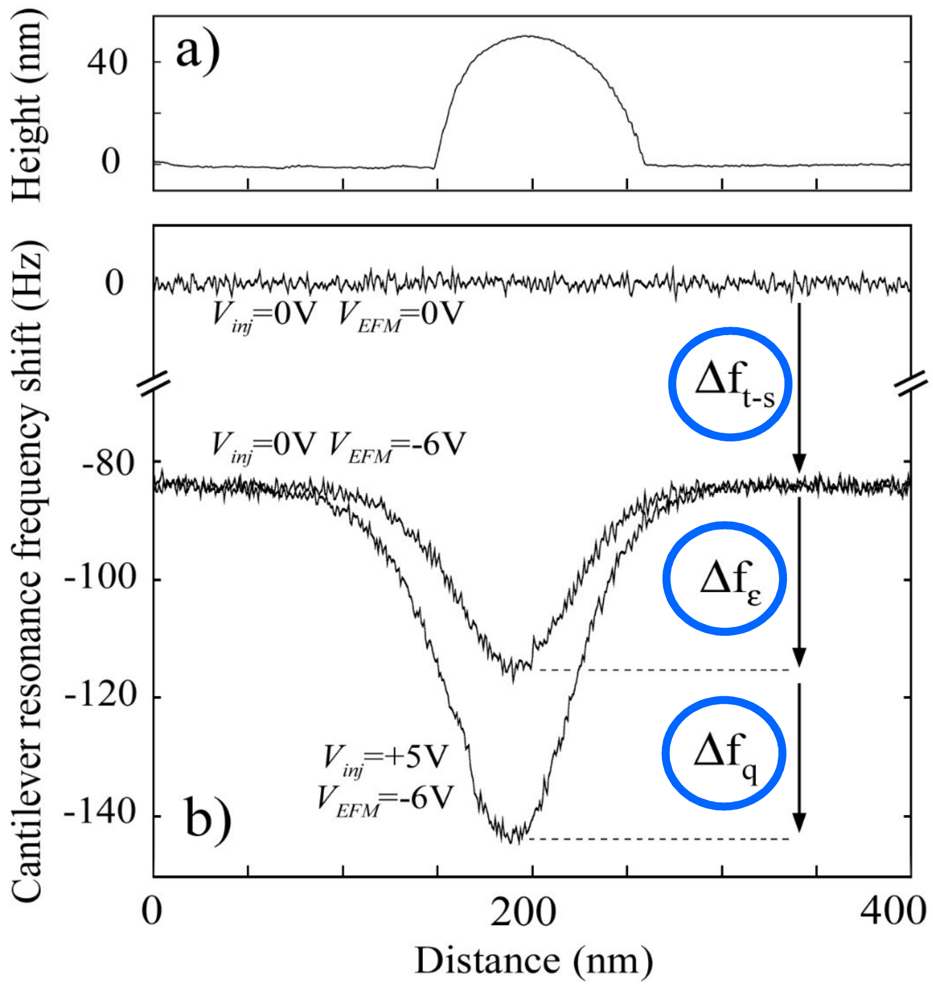
Example 1 : Imaging charged nanocrystals



Appl. Phys. Lett., 78 5054 (2002)

Example 1 : Probing a charge or a dipole ?



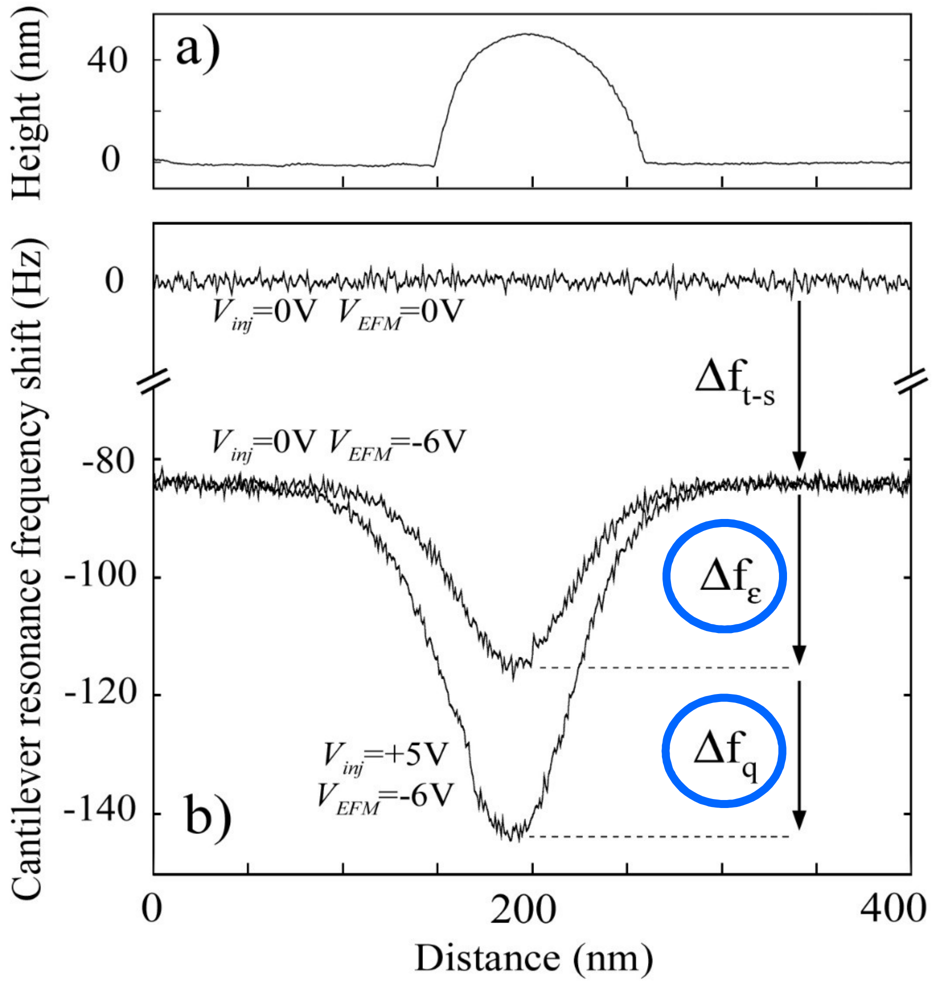
- tip-substrate capacitance
  prop. to V^2

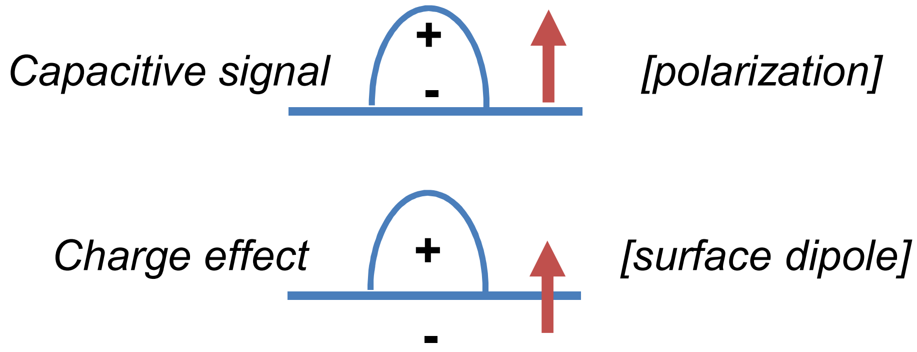
- nanoparticle capacitive effect
  prop. to V^2

- charge perturbation
  prop. to $Q.V$ **(+Q² contribution)**

T. Melin et al. , PRL 166101 (2004)

Example 1 : Probing a charge or a dipole ?





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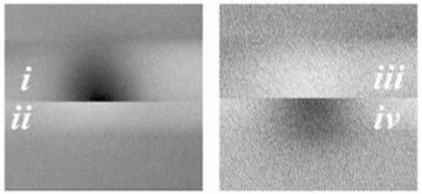
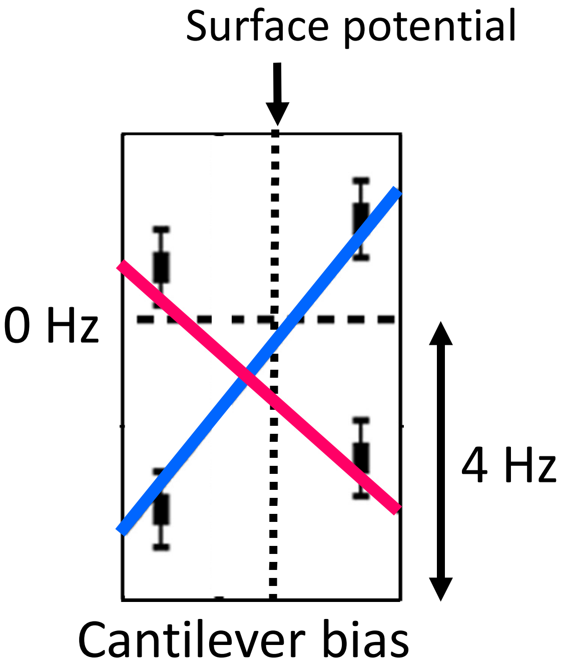
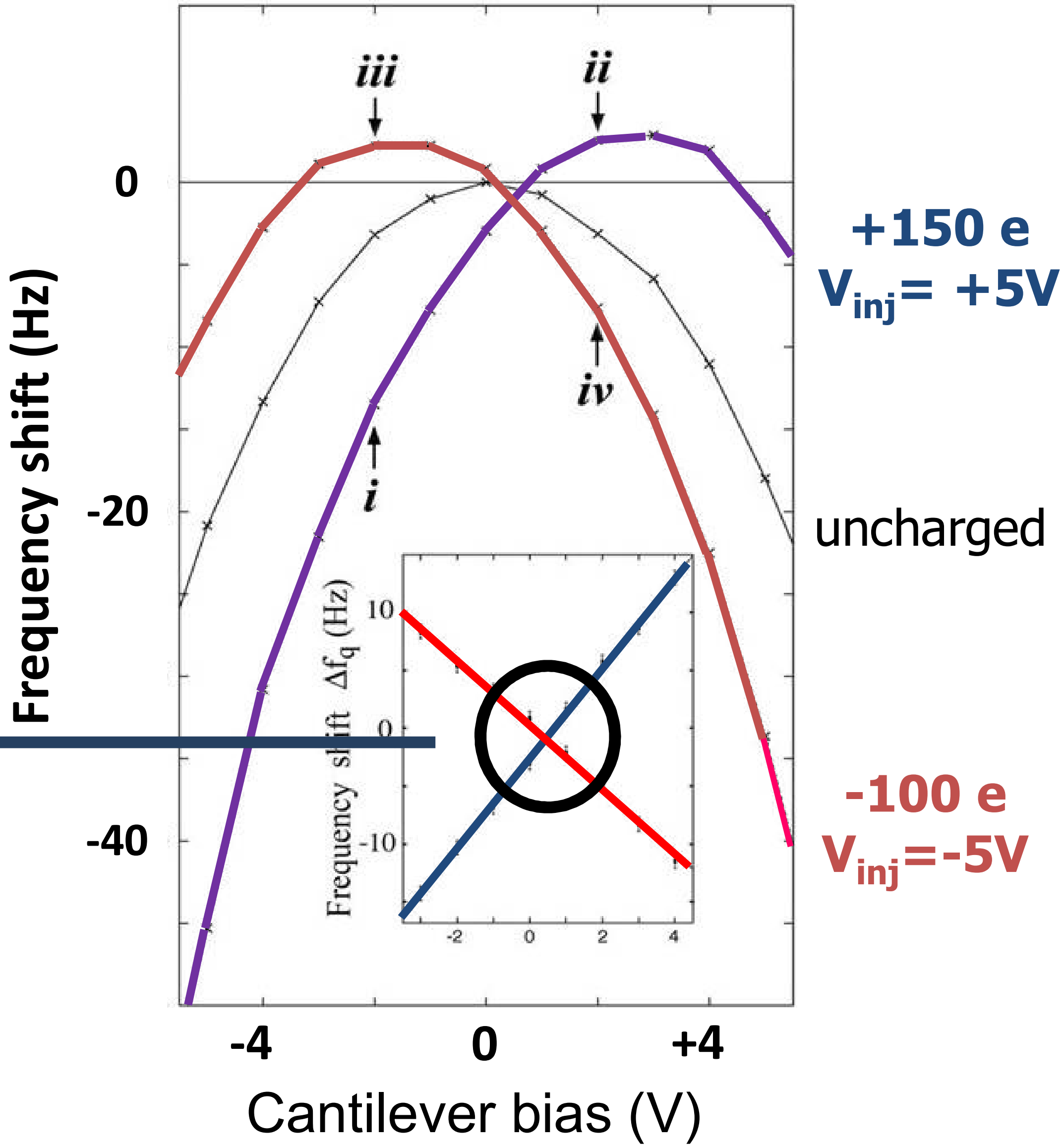
Example 1 : Spectroscopic analysis of charge signals

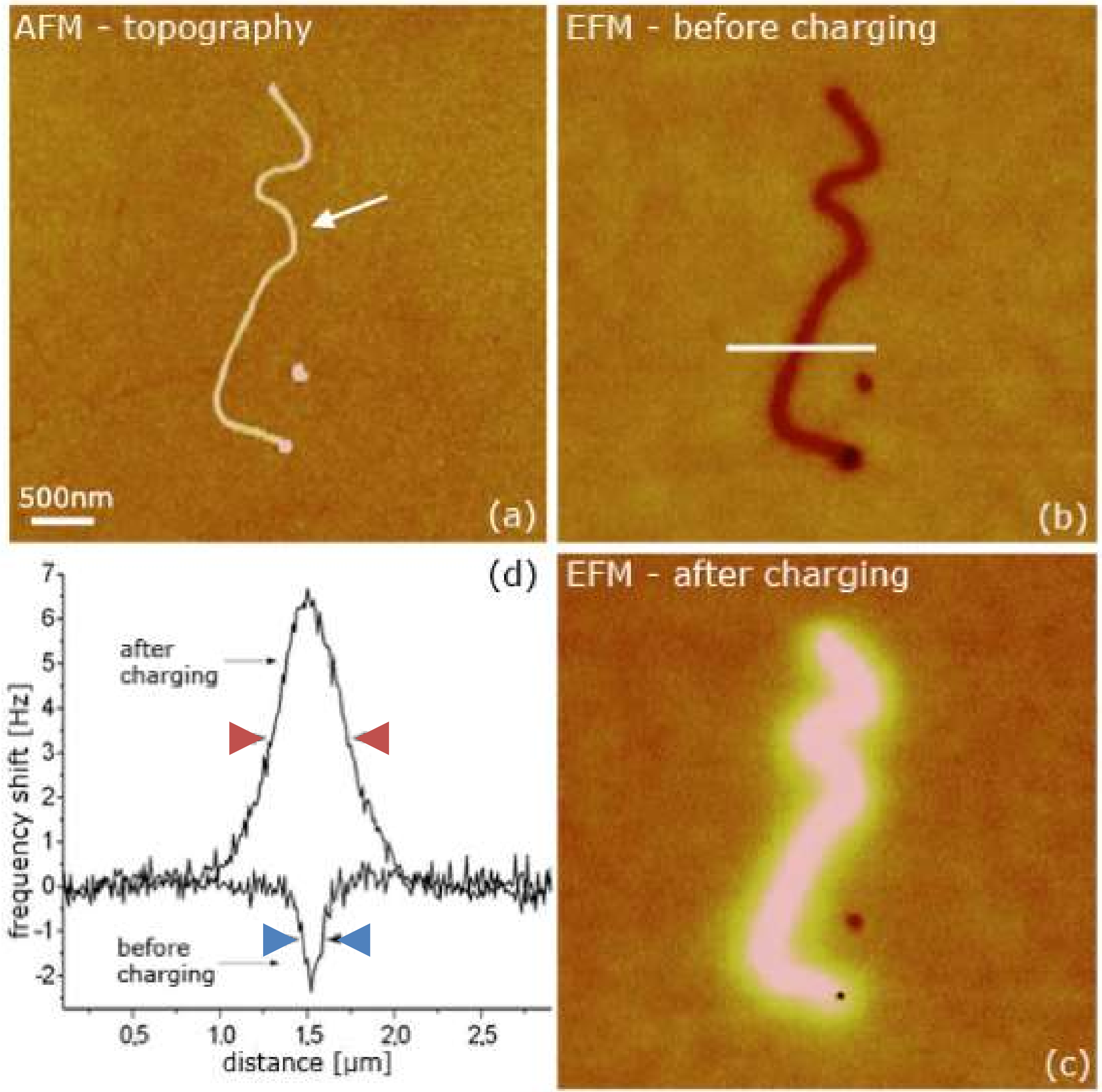


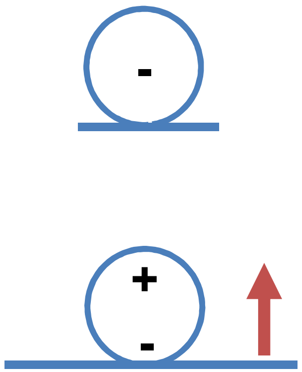




Example 2 : Probing a charge or a dipole ?

multiwalled carbon nanotube MWCNT
(~20nm diameter)
on 200nm thick SiO_2





charge signal

capacitive signal

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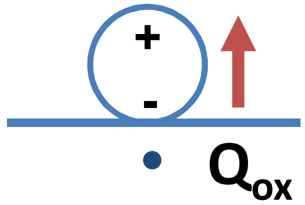
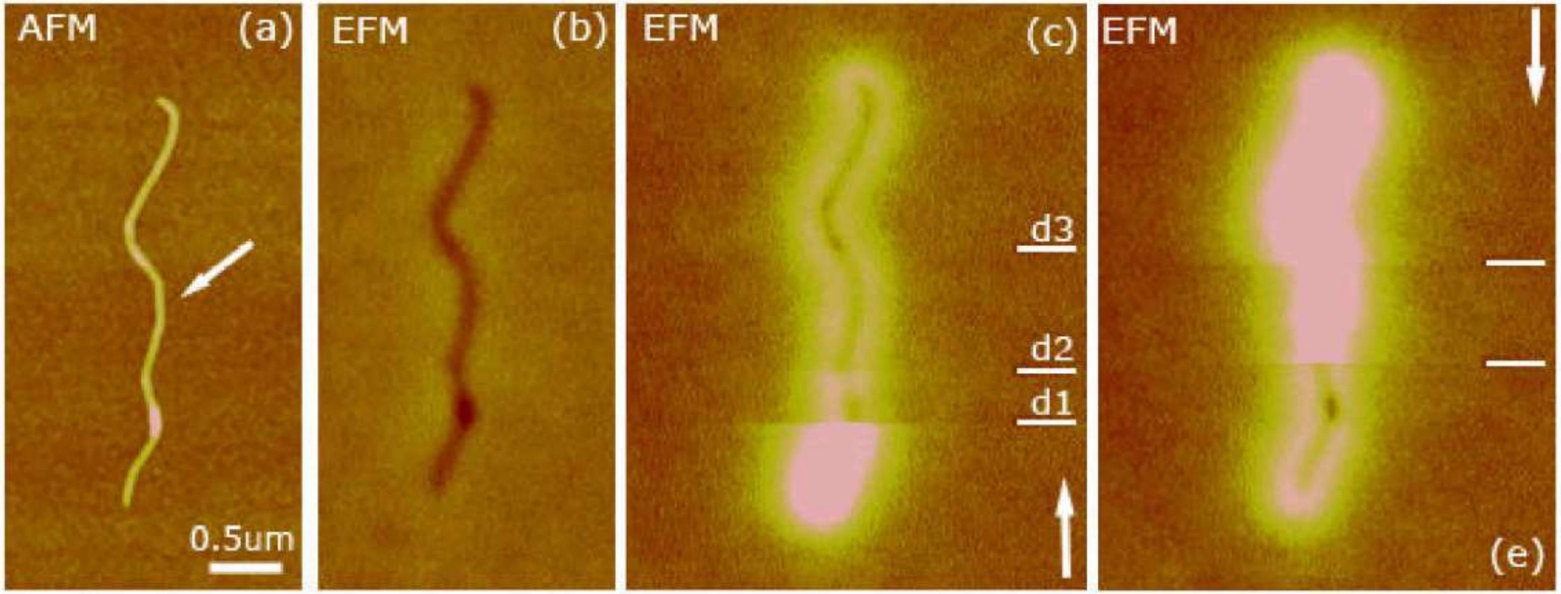
Example 2 : Probing a charge or a dipole ?

After discharge



capacitive signal

+ residual oxide charge



[MWCNT with 18 nm diameter, V_{inj}=-7V (3 min) detection V_{EFM}=-3V]

Optical beam deflection EFM
with soft cantilevers ($k=3N/m; f_0=60kHz$)

	in air	in vacuum, 300K
	limited by thermal noise	
F'_{min}	**$\sim 10^{-5}$ N/m** B=100Hz, Q=200 A=25nm	**a few 10^{-6} N/m** B=50Hz, Q=20000 A=15nm
<z>	**50-100nm**	**10-20nm**

$$F'_{min} = \sqrt{\frac{4\,k \,.\, k_B T \,.\, B}{\pi\, f_0 \,.\, A^2 \,.\, Q}}$$

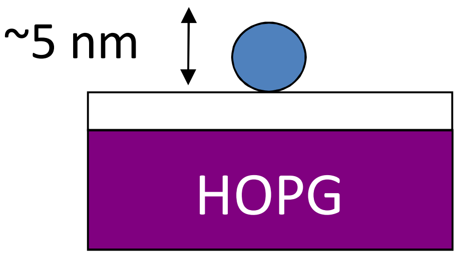
[F. Giessibl et al., Phys. Rev. B 2011 + references therein]

Sensitivity

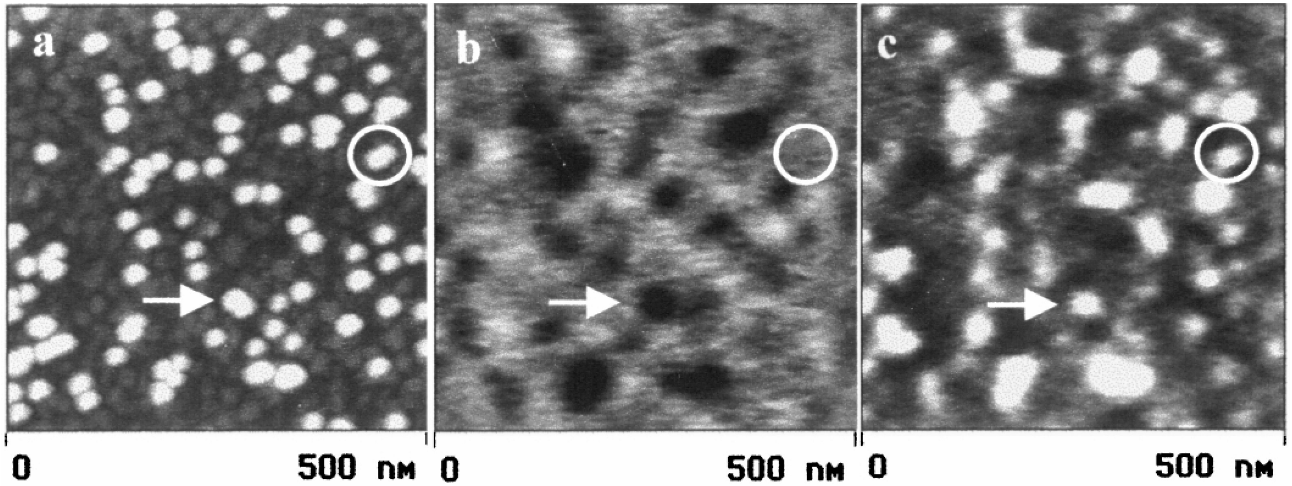
	Optical beam deflection EFM with soft cantilevers ($k=3N/m; f_0=60kHz$)		Qplus, LER
	in air	in vacuum, 300K	vacuum, 1-5 K
	limited by thermal noise		deflection noise, thermal noise, …
F'_{min}	**$\sim 10^{-5}$ N/m** B=100Hz, Q=200 A=25nm	**a few 10^{-6} N/m** B=50Hz, Q=20000 A=15nm	**$\sim 10^{-3}$ N/m** B=25Hz, Q=20000 A=200pm
<z>	**50-100nm**	**10-20nm**	**< 1 nm**
	Long-range (LR)	LR + SR	Short-range (SR)

Resolution : single charge detection in ambient air ?

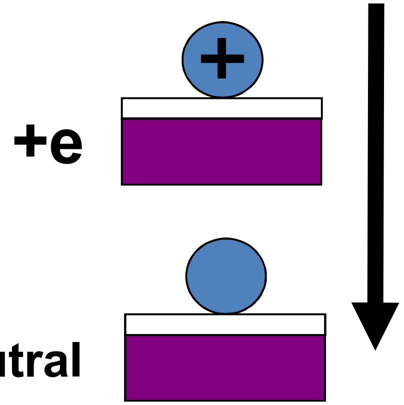
CdSe nanocrystals

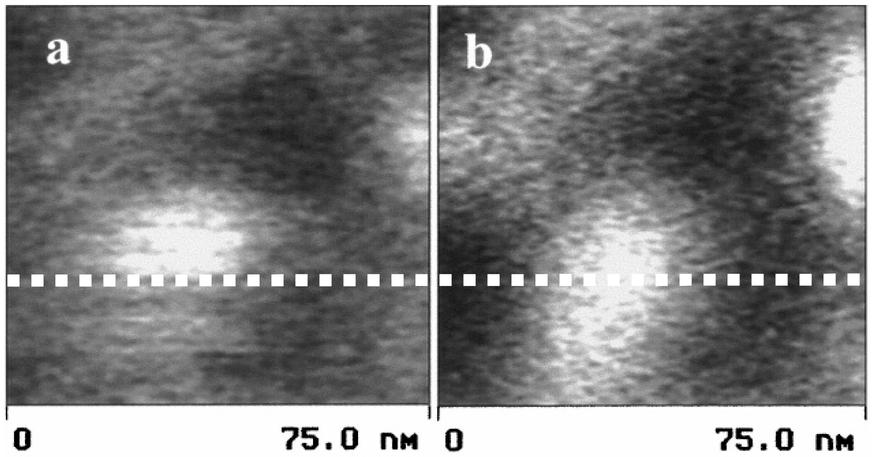


Topography | Charge image | Dielectric image



Charge image | Dielectric image





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T. Krauss & L. Brus, PRL **83** 4840 (1999)

Time resolution

- In general, limited by the phase demodulation of the cantilever oscillation

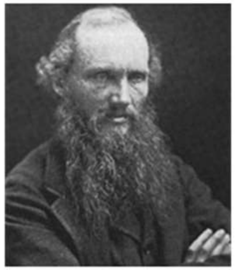
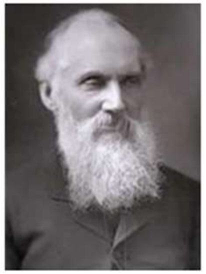
- better resolution possible :
  - fast frequency shift demodulation,
  - oscillation transients (sub-µs see D. Ginger et al. Nanoletters 2012)
  - response under modulated illumination (see Ł. Borowik)

Quantitative charge measurements ?

- in general, semi-quantitative models only
- difficult due to the large variety of dielectric environments
- numerical simulations in most situations
- single charge events as calibration

SOMMAIRE

Principle …

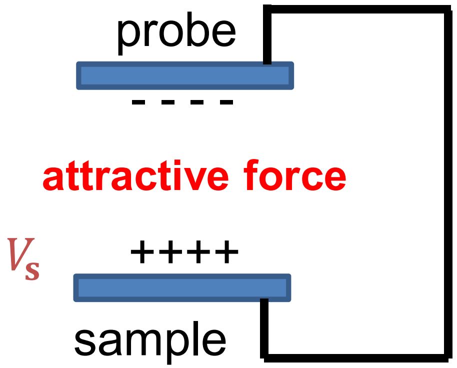



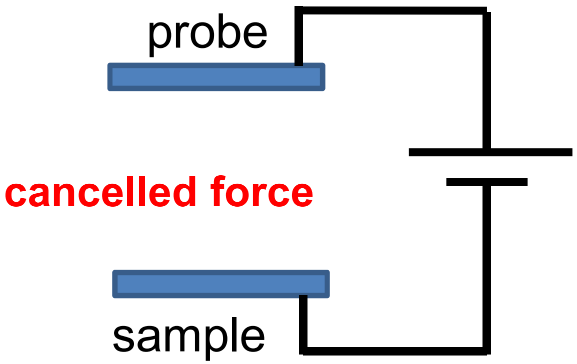

Measuring surface potentials from forces

- Lord Kelvin (1898)
- Zisman (1932) : vibrating Kelvin probe (down to mm size)
- Nonnenmacher (1991) : Kelvin probe **force** microscopy

different metals

e.g. $\phi_{probe} > \phi_{sample}$



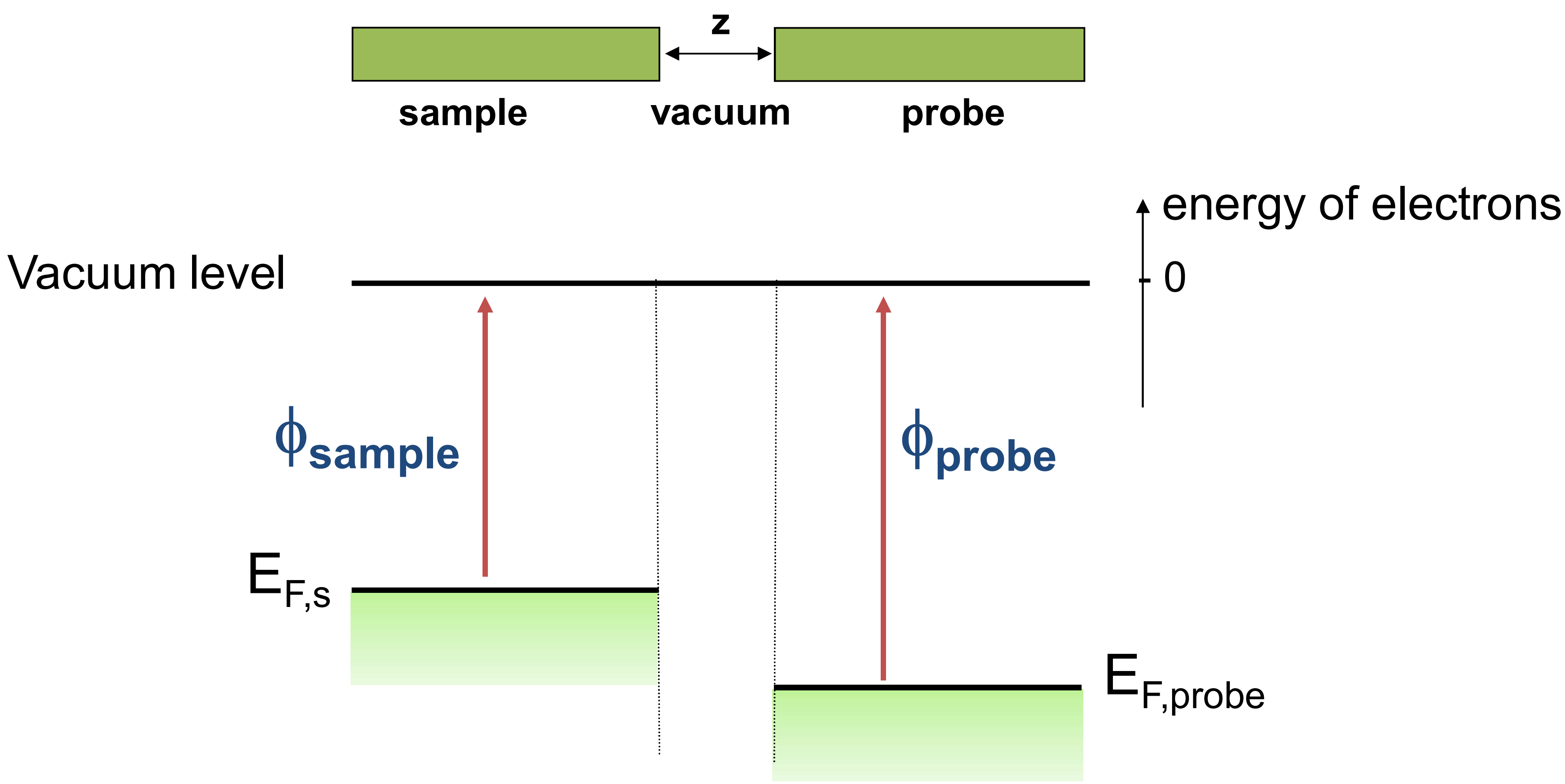


$$V_{DC} = \underbrace{\frac{\emptyset_{probe} - \emptyset_{sample}}{|e|}}$$

V_{CPD} ou V_s

Work function
measurement

Energy diagrams



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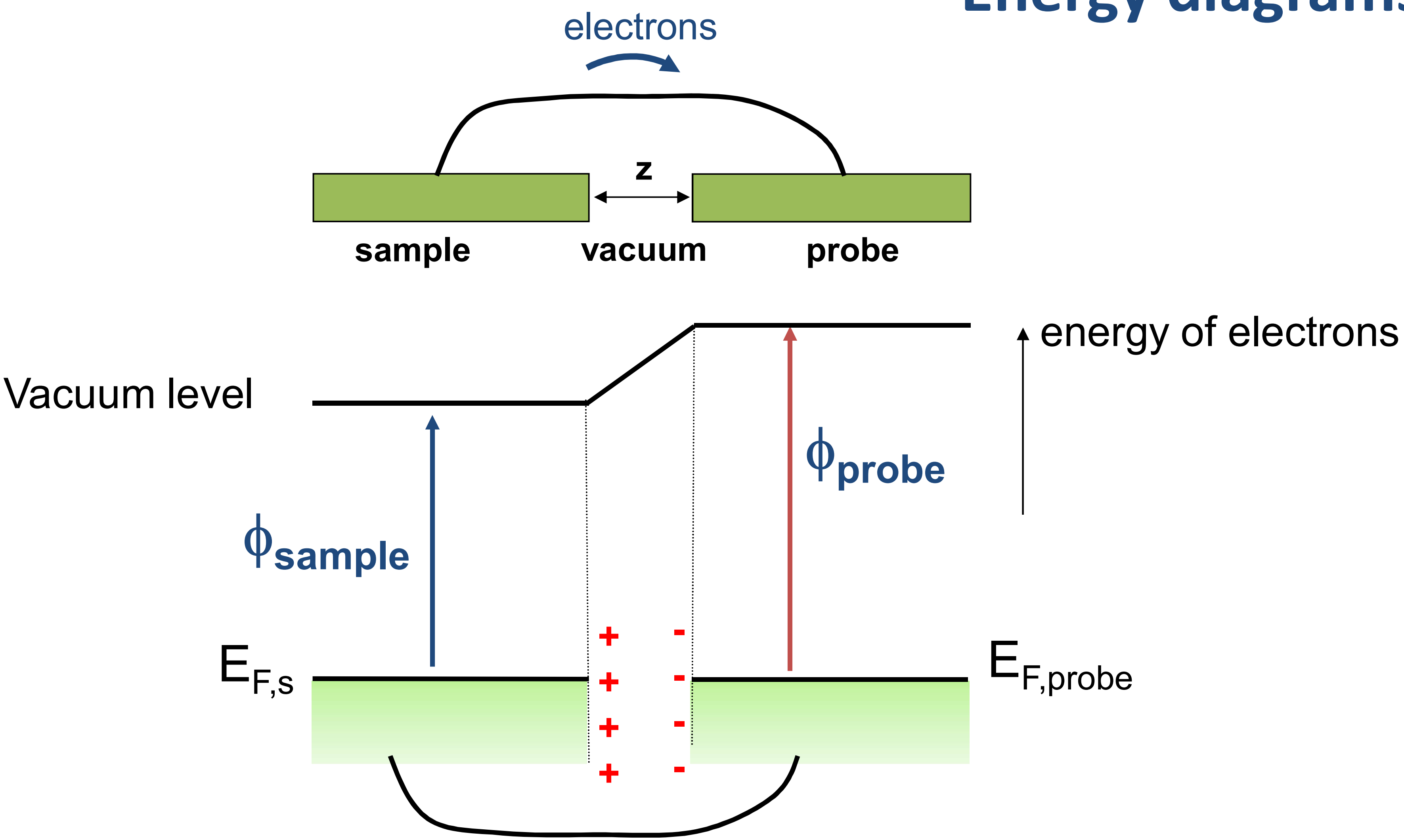
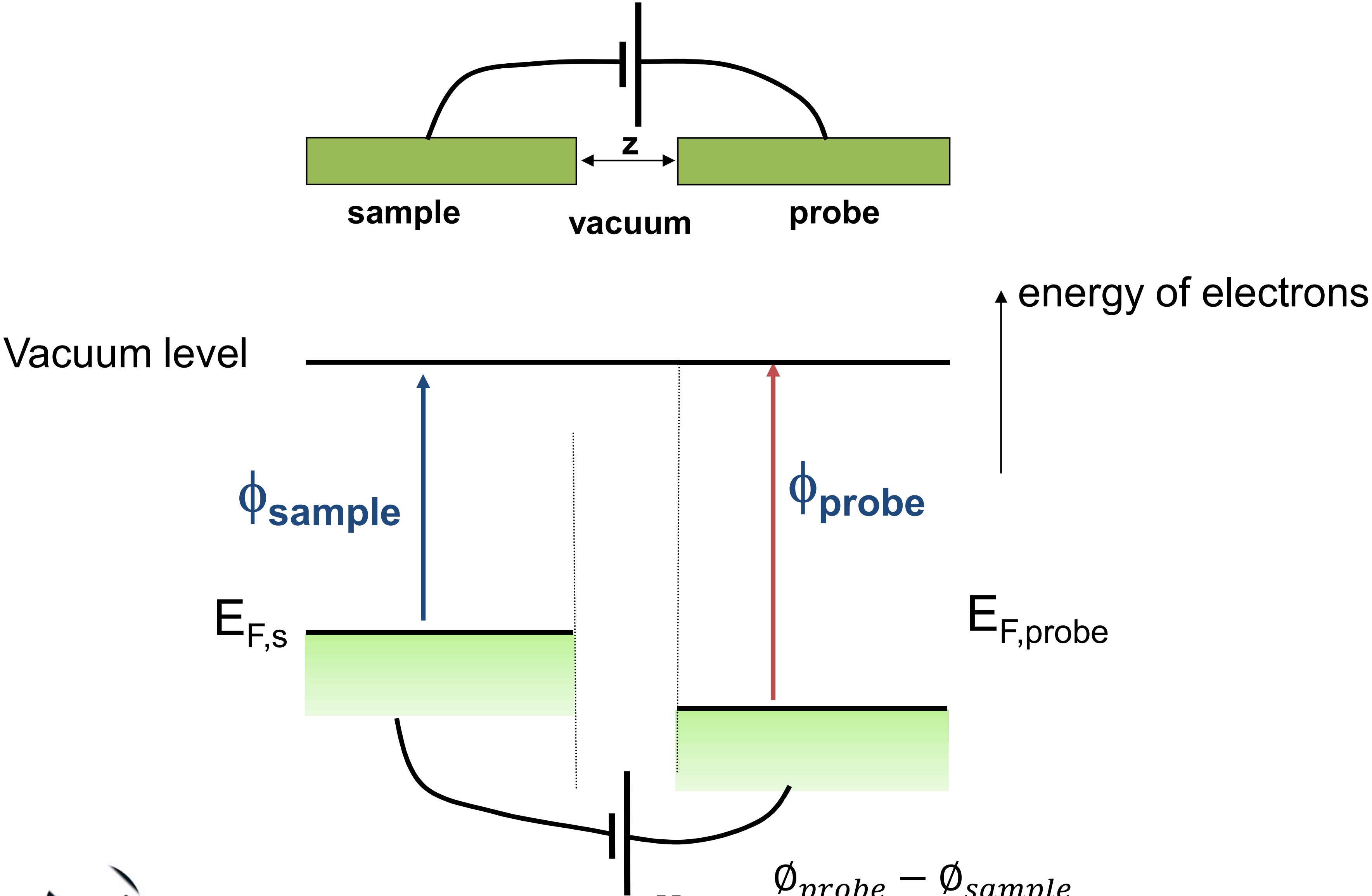


Energy diagrams



$$V_{DC} = \frac{\emptyset_{probe} - \emptyset_{sample}}{e}$$

A few remarks …

- The sign of V_{dc} is user-dependent (V_{dc} at the tip, or at the sample)

- V_{dc} at the tip (and V_s at the surface)

  ‘electrostatics-friendly’ convention :

  a positive charge or dipole (e.g. adsorbate) is ‘seen’ as a positive V_s

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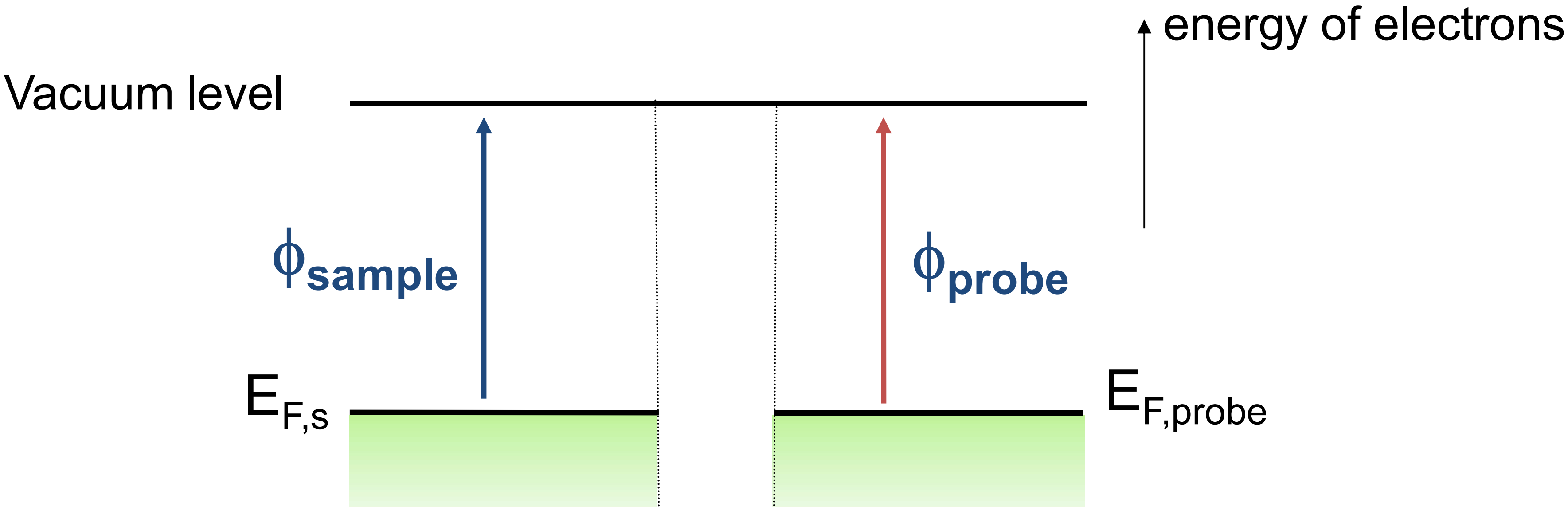

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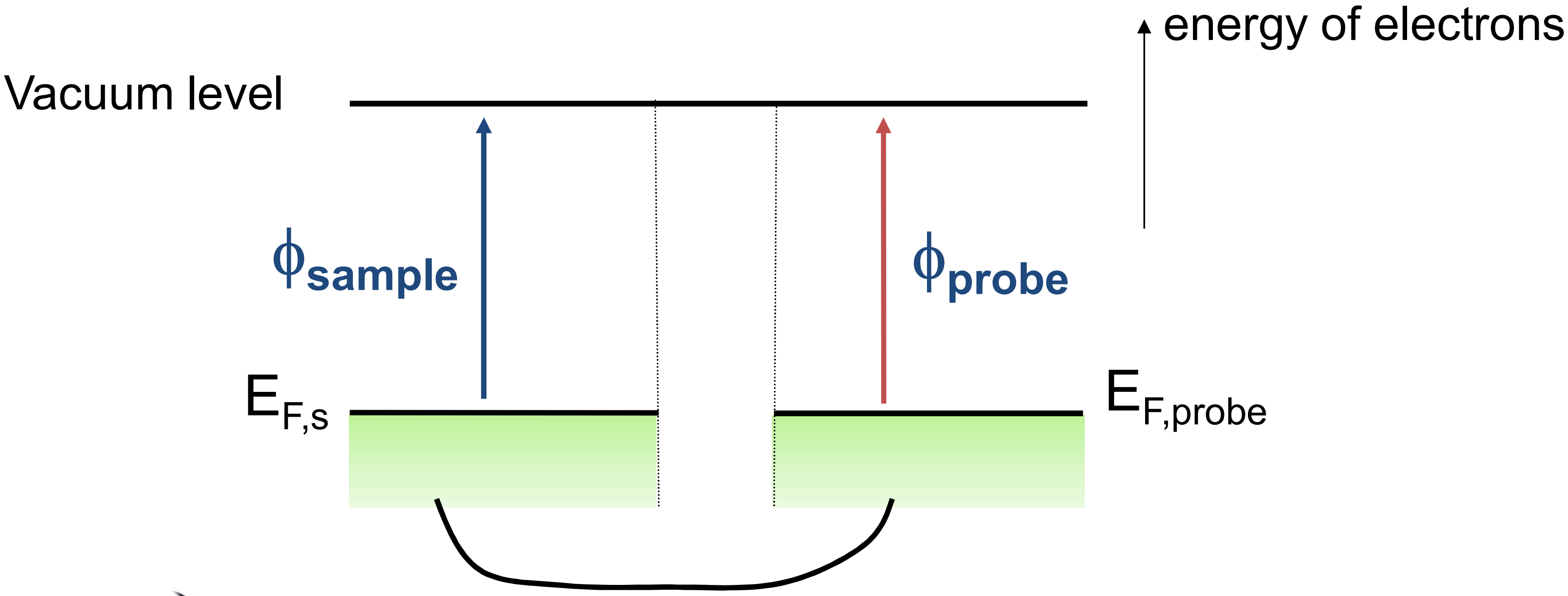

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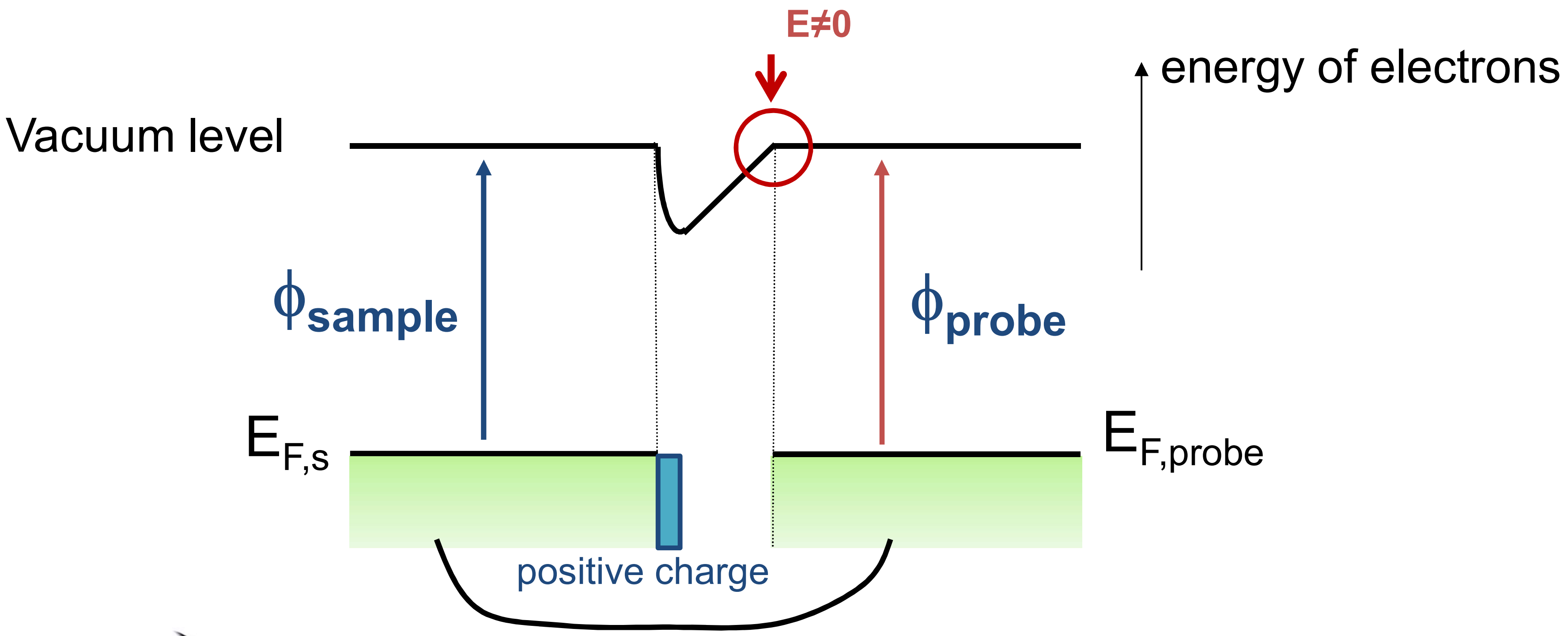

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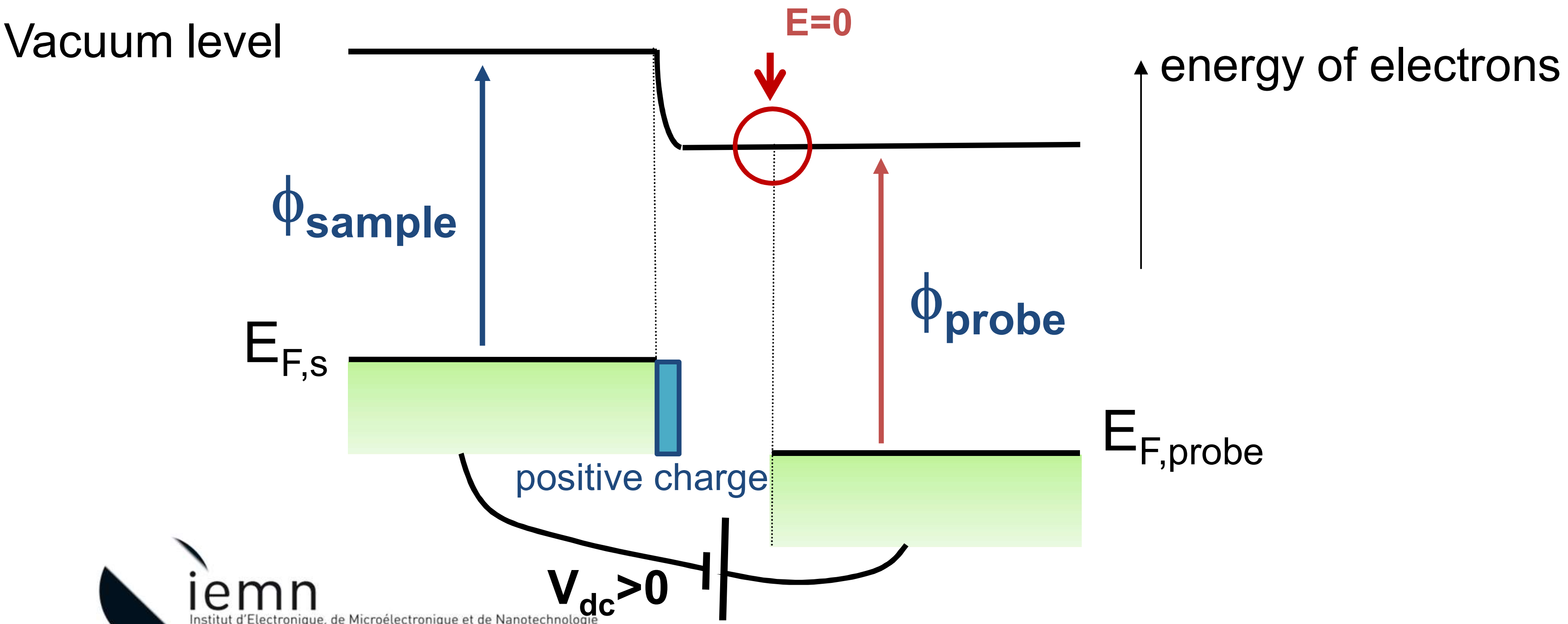
- V_{dc} at the tip (and V_s at the surface)

'electrostatics-friendly' convention :

a positive charge or dipole (e.g. adsorbate) is 'seen' as a positive V_s



A few remarks …

- **The sign of V_{dc} is user-dependent** (V_{dc} at the tip, or at the sample)

- **V_{dc} at the tip** (and V_s at the surface)

  'electrostatics friendly' convention

  a positive charge or dipole (e.g. adsorbate) is 'seen' as a positive V_s

- **V_{dc} at the sample**

  'work-function friendly' convention :

  a material with a larger work-function will be imaged as « more positive » in KPFM images

Zisman method

Rev. Sci. Instrum. **3**, 367 (1932)

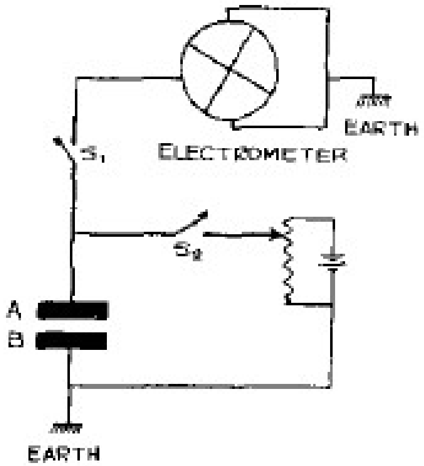
A NEW METHOD OF MEASURING CONTACT POTENTIAL DIFFERENCES IN METALS

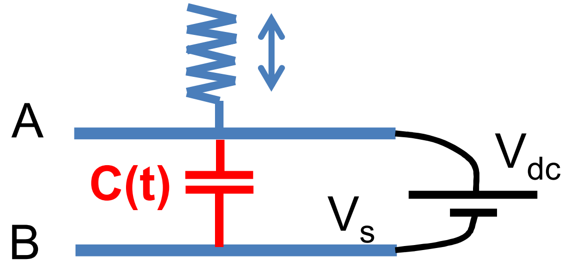
By W. A. Zisman

[Jefferson Physical Laboratory, Harvard University, Cambridge, Mass. Received March 5, 1932]

ABSTRACT

A new method is described for measuring the contact potential differences between dissimilar metals. It enables one to measure the p.d. to 1/1000 volt in a few seconds of manipulation. An apparatus is described for studying metals in air and another is described for high vacuum work.





$C = C_0 + \Delta C.\sin \omega t$

↓

$i(t) = \Delta C.\omega.[V_{dc} - V_s].\cos(\omega t)$

↓

to a loud speaker (!)
(ω in the audio range) :
zero sound for $V_{dc} = V_s$

Kelvin method

Response of the electrometer deflection as a function of V_{dc} to find the zero force

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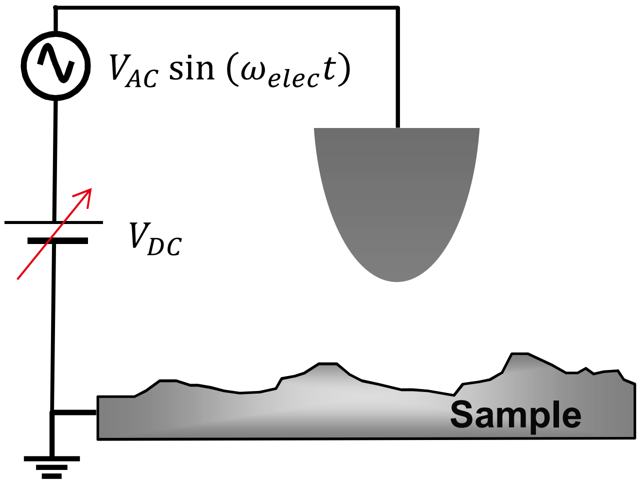
SOMMAIRE

KFM en modulation d'amplitude (AM-KFM)

$$V = V_{DC} - V_{CPD} + V_{AC}\sin(\omega_{elec}t)$$



Force électrostatique

(3 composantes)

$$F_0 = \frac{1}{2}\frac{\partial C}{\partial z}\left[(V_{DC} - V_{CPD})^2 + \frac{V_{AC}^2}{2}\right]$$

$$F_{z,\,\omega_{elec}} = \frac{\partial C}{\partial z}(V_{DC} - V_{CPD})V_{AC}$$

$$F_{z,\,2\,\omega_{elec}} = -\frac{1}{4}\frac{\partial C}{\partial z}V_{AC}^2$$

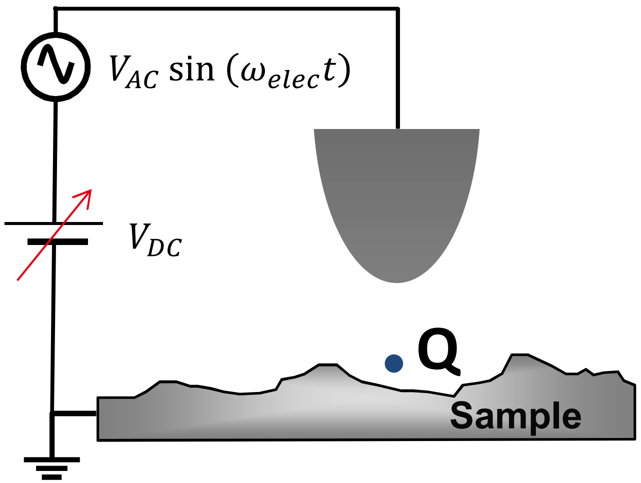
AM-KFM → Annulation de la composante à ω_{elec} de la force électrostatique

$$V_{DC} = V_{CPD}$$

M. Nonnenmacher, Appl. Phys. Lett. 58(25) 1991, p. 2921

KFM en modulation d'amplitude (AM-KFM)

$$V = V_{DC} - V_{CPD} + V_{AC}\sin(\omega_{elec} t)$$



Force électrostatique
(3 composantes)

$$F_0 = \frac{1}{2}\frac{\partial C}{\partial z}\left[(V_{DC} - V_{CPD})^2 + \frac{V_{AC}^2}{2}\right]$$ **+ contributions force image**

$$F_{z,\omega_{elec}} = \frac{\partial C}{\partial z}(V_{DC} - V_{CPD})V_{AC} + \boldsymbol{K(z)\,Q\,V_{AC}}$$

$$F_{z,2\omega_{elec}} = -\frac{1}{4}\frac{\partial C}{\partial z}V_{AC}^2$$

AM-KFM ➞ Annulation de la composante à ω_{elec} de la force électrostatique

$$V_{DC} = V_{CPD} + V_Q(z)$$

M. Nonnenmacher, Appl. Phys. Lett. 58(25) 1991, p. 2921

AM-KFM : MODES D'ACQUISITION

Simple passage

Excitation mécanique à la résonance du levier ($f_{méca} = f_0$)

Tapping (Air)
ou
nc-AFM (Vide, UHV)

+

Modulation électrique à la 1ère harmonique ($f_{élec} = f_1 \approx 6{,}3 \times f_0$)

Démodulation à $f_{élec} \rightarrow$ amplitude

Double passage

1er passage

Excitation mécanique à la résonance du levier ($f_{méca} = f_0$)

Tapping (Air)
ou
nc-AFM (Vide, UHV)

2ème passage : lift

Modulation électrique à la résonance du levier ($f_{élec} = f_0$)

Démodulation à $f_{élec} \rightarrow$ amplitude
Hauteur de lift (20 à 100 nm)

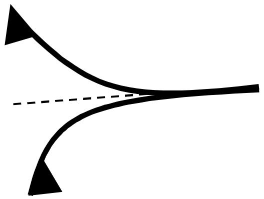
Example of a single-pass (UHV) AM-KPFM mode

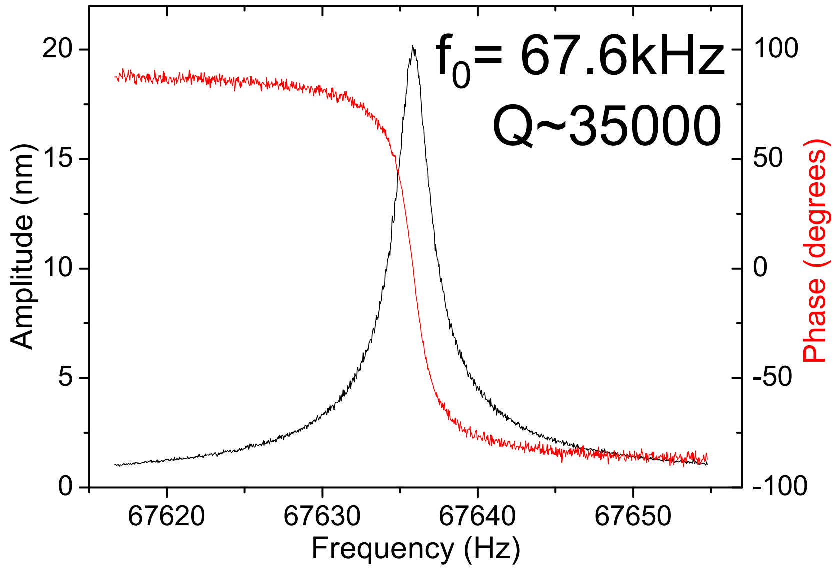
- first resonance f_0

- mechanical excitation

- **non-contact AFM**

Δf = -5Hz oscillation amplitude 15 nm
minimum tip-substrate distance ~5nm

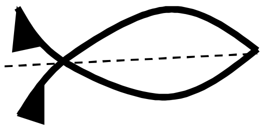


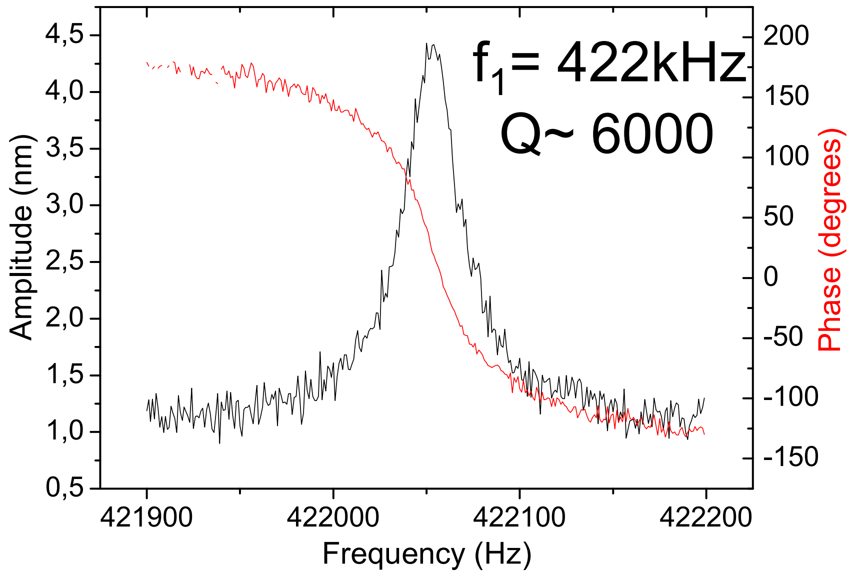


- second resonance f_1~6.2×f_0

- electrostatic excitation

- **KFM loop at f_1**





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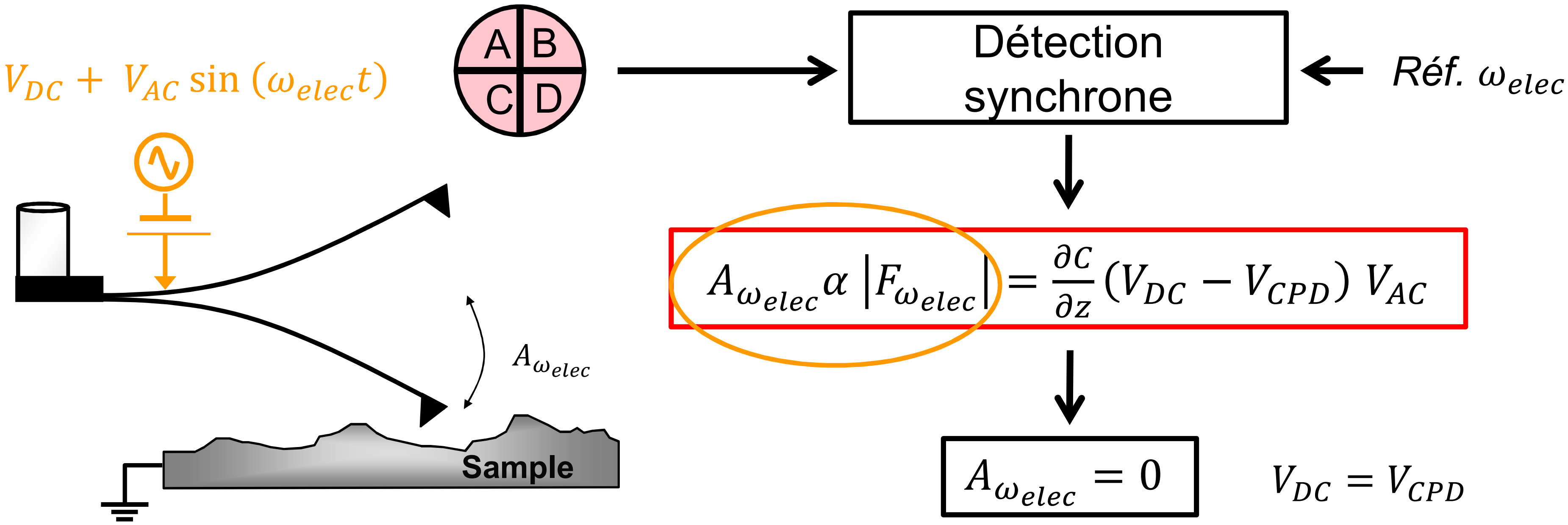
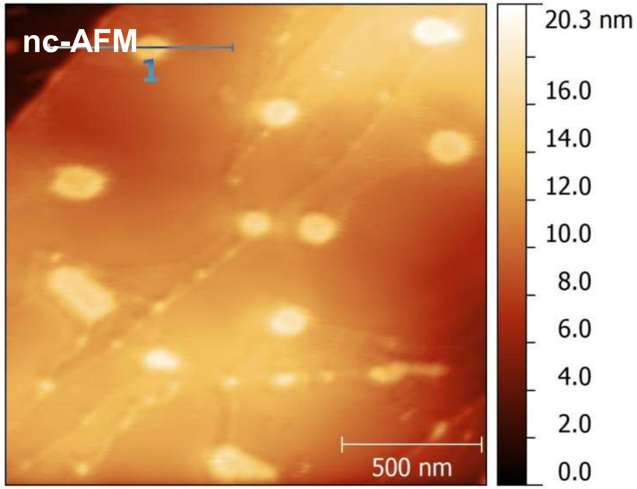
AM-KFM : EN PRATIQUE



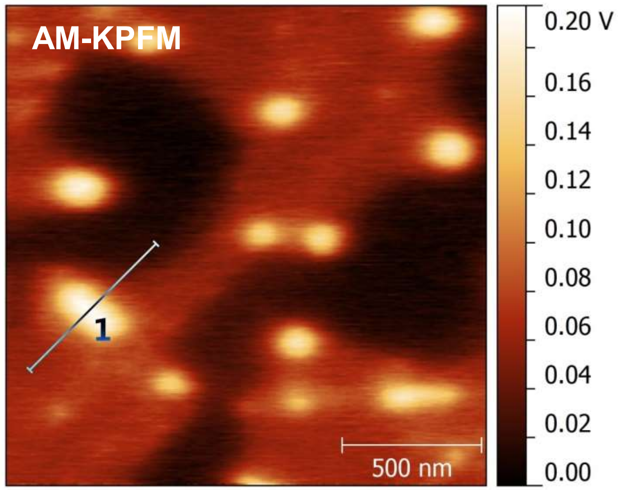
- **Une boucle de contre-réaction (PID) annule la composante $A_{\omega_{elec}}$ en ajustant la valeur du V_{DC}**

Imaging …

Doped nanocrystals inducing charge transfers to the substrate



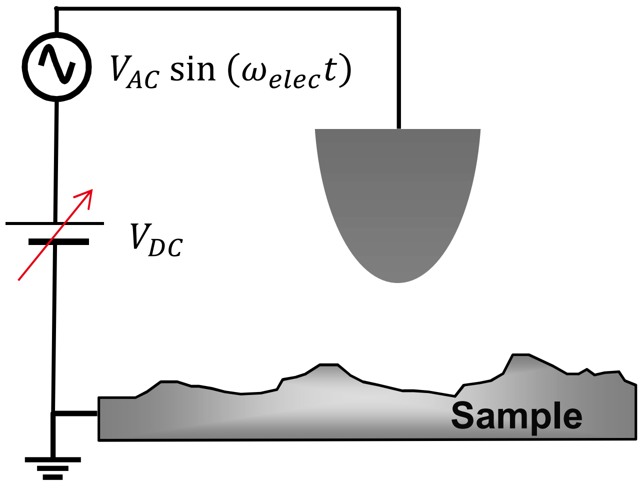
topo : $A_{pp} = 20\ nm$, Δf = - 5 Hz ;
1,7 µm * 1,7 µm; 512 *512 pixels;
tip-sample distance of 4-6 nm



AM-KPFM : V_{ac} = 200 mV;
V_{dc} = 2 V; τ = 100 µs

KFM en modulation de fréquence (FM-KFM)

$$V = V_{DC} - V_{CPD} + V_{AC}\sin(\omega_{elec} t)$$



Gradient de force électrostatique

(3 composantes)

$$\frac{\partial F_{z,0}}{\partial z} = \frac{1}{2}\frac{\partial^2 C}{\partial z^2}\left[(V_{DC} - V_{CPD})^2 + \frac{V_{AC}^2}{2}\right]$$

$$\frac{\partial F_{z,\omega_{elec}}}{\partial z} = \frac{\partial^2 C}{\partial z^2}(V_{DC} - V_{CPD})\, V_{AC}$$

$$\frac{\partial F_{z,2\omega_{elec}}}{\partial z} = -\frac{1}{4}\frac{\partial^2 C}{\partial z^2} V_{AC}^2$$

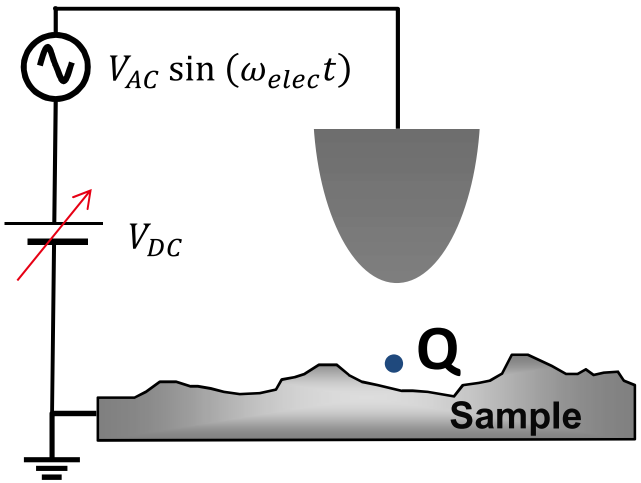
FM-KFM ➡ Annulation de la composante à ω_{elec} du gradient de la force électrostatique

$$V_{DC} = V_{CPD}$$

M. Nonnenmacher, Appl. Phys. Lett. 58(25) 1991, p. 2921

KFM en modulation de fréquence (FM-KFM)

$$V = V_{DC} - V_{CPD} + V_{AC}\sin(\omega_{elec} t)$$



Gradient de force électrostatique

(3 composantes)

$$\frac{\partial F_{z,0}}{\partial z} = \frac{1}{2}\frac{\partial^2 C}{\partial z^2}\left[(V_{DC} - V_{CPD})^2 + \frac{V_{AC}^2}{2}\right]$$ **+ contributions force image**

$$\frac{\partial F_{z,\omega_{elec}}}{\partial z} = \frac{\partial^2 C}{\partial z^2}(V_{DC} - V_{CPD})\, V_{AC} + \boldsymbol{K'(z)\, Q\, V_{AC}}$$

$$\frac{\partial F_{z,2\omega_{elec}}}{\partial z} = -\frac{1}{4}\frac{\partial^2 C}{\partial z^2} V_{AC}^2$$

FM-KFM ➡ Annulation de la composante à ω_{elec} du gradient de la la force électrostatique

$$V_{DC} = V_{CPD} + V_Q(z)$$

M. Nonnenmacher, Appl. Phys. Lett. 58(25) 1991, p. 2921

Simple passage

Excitation mécanique à la résonance du levier ($f_{méca} = f_0$)

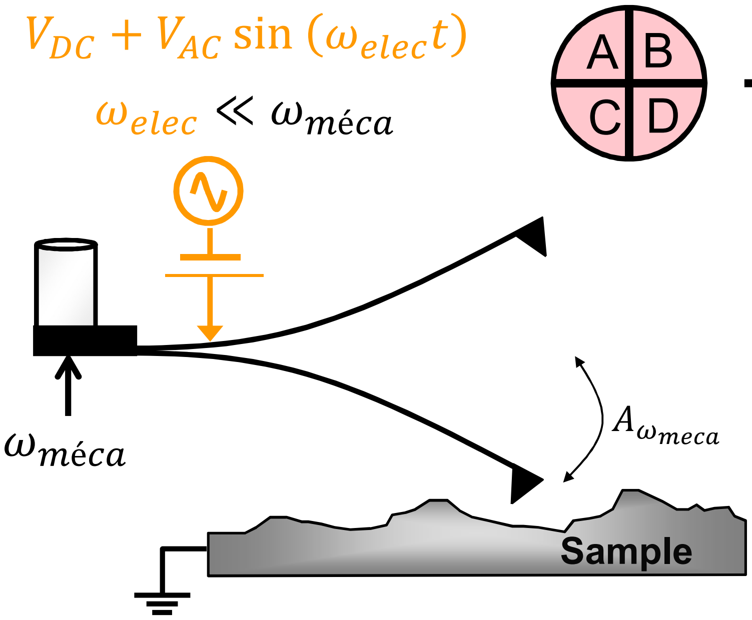
Tapping (Air)
ou
nc-AFM (Vide, UHV)

+

Modulation électrique à basse fréquence ($f_{élec} << f_0$)

Démodulation à $f_{élec}$ du signal de phase mécanique (mode Tapping) ou du décalage de fréquence (mode nc-AFM)

FM-KFM : EN MODE TAPPING (AIR)



Détection synchrone ← Réf. $\omega_{méca}$

↓

$$\Delta\varphi_{méca} = -\frac{Q}{k}\frac{\partial F}{\partial z} \propto \frac{\partial F_{elec}}{\partial z}$$

Modulation de fréquence

↓

Détection synchrone ← Réf. ω_{elec}

↓

$$\left|\frac{\partial F_{\omega_{elec}}}{\partial z}\right| = \frac{\partial^2 C}{\partial z^2}(V_{DC} - V_{CPD})\, V_{AC}$$

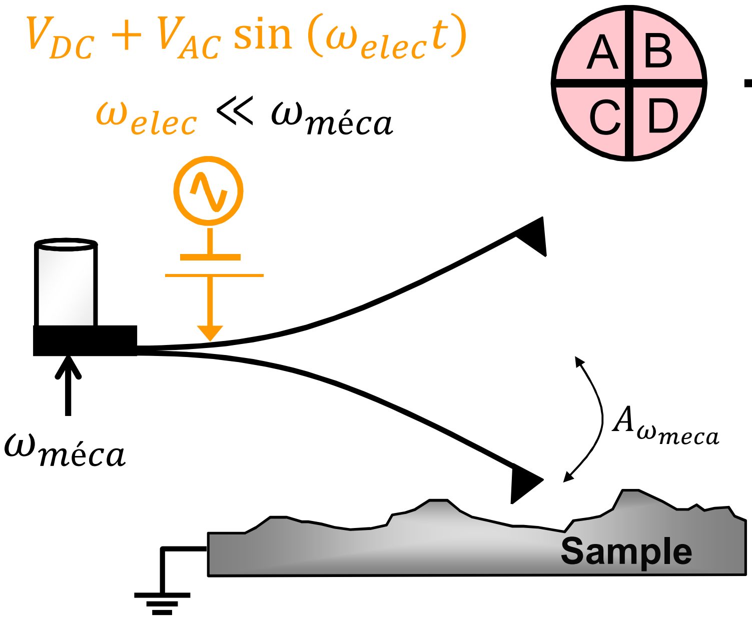
↓

$$\left|\frac{\partial F_{\omega_{elec}}}{\partial z}\right| = 0$$

$$V_{DC} = V_{CPD}$$

- **Une boucle de contre-réaction (PID) annule la composante $\frac{\partial F_{\omega_{elec}}}{\partial z}$ en ajustant la valeur du V_{DC}**

FM-KFM : EN MODE NC-AFM (UHV)



PLL

↓

$$\Delta f_{méca} = -\frac{1}{2k}\frac{\partial F}{\partial z} \propto \frac{\partial F_{elec}}{\partial z}$$

Modulation de fréquence

↓

Détection synchrone ← *Réf.* ω_{elec}

↓

$$\left|\frac{\partial F_{\omega_{elec}}}{\partial z}\right| = \frac{\partial^2 C}{\partial z^2}(V_{DC} - V_{CPD})\, V_{AC}$$

↓

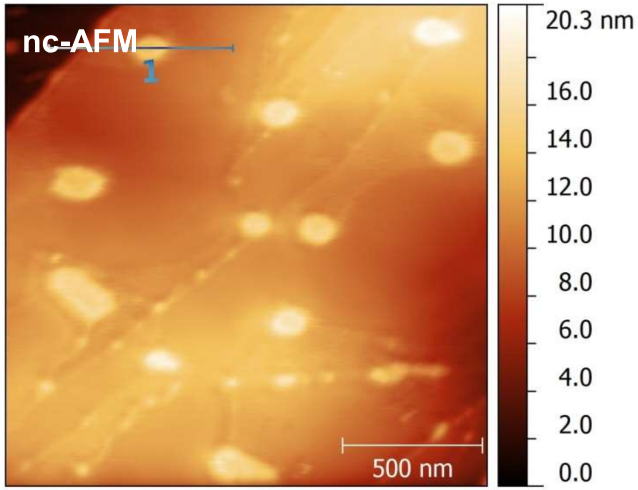
$$\left|\frac{\partial F_{\omega_{elec}}}{\partial z}\right| = 0$$

$$V_{DC} = V_{CPD}$$

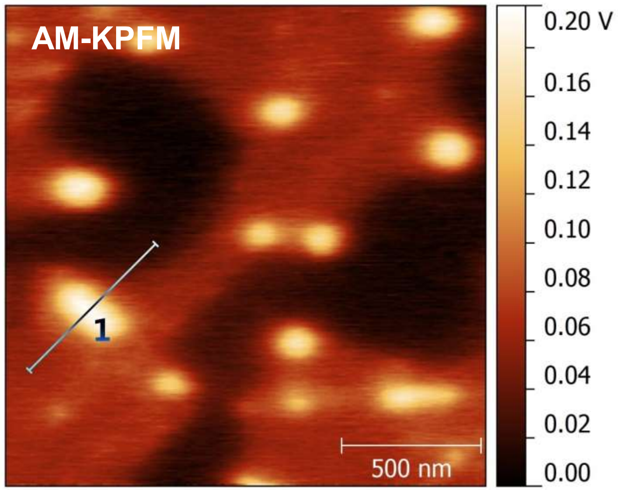
AM-KPFM versus FM-KPFM

Imaging …

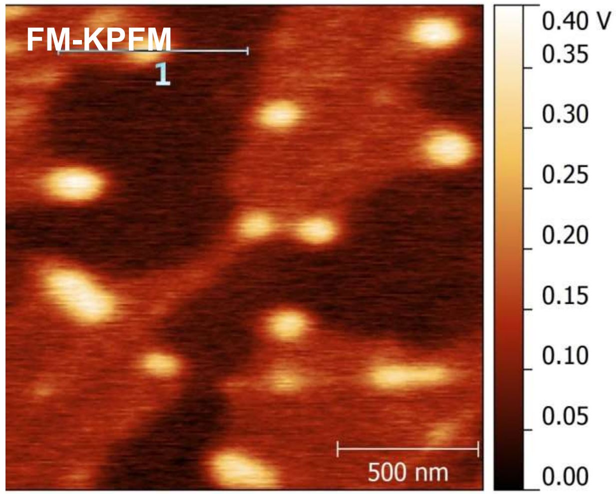
Doped nanocrystals inducing charge transfers to the substrate



topo : $A_{pp} = 20\ nm$, Δf = - 5 Hz ;
1,7 µm * 1,7 µm; 512 *512 pixels;
tip-sample distance of 4-6 nm



AM-KPFM : V_{ac} = 200 mV; V_{dc} = 2 V; τ = 100 µs

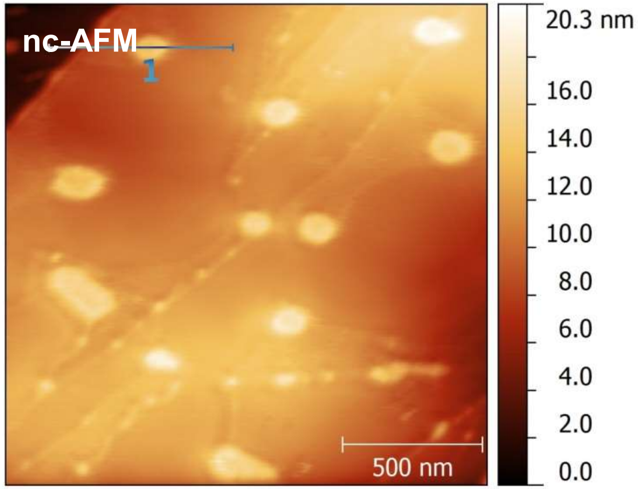


FM-KPFM : f_{ac} ~ 50Hz; V_{ac} = 200 mV

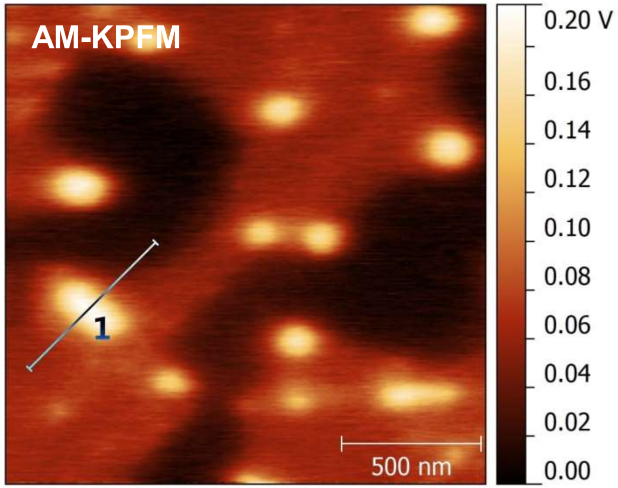
AM-KPFM versus FM-KPFM

	signal to noise	resolution
AM-KPFM	+	-
FM-KPFM	-	+

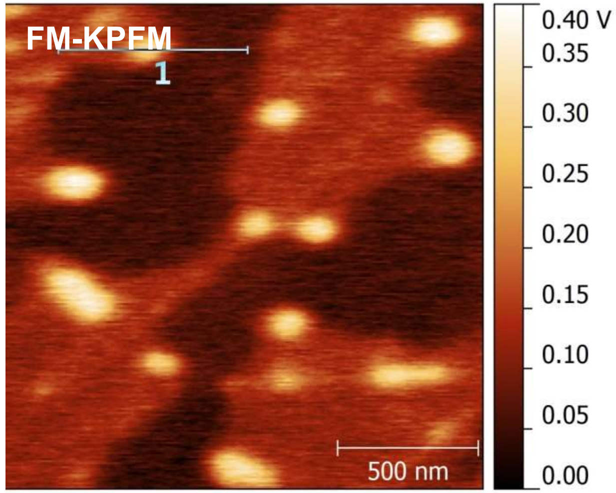
- less ac cross-talk
- no need for a 2nd resonance



topo : $A_{pp} = 20\ nm$, Δf = - 5 Hz ;
1,7 µm * 1,7 µm; 512 *512 pixels;
tip-sample distance of 4-6 nm



AM-KPFM : V_{ac} = 200 mV; V_{dc} = 2 V; τ = 100 µs



FM-KPFM : f_{ac} ~ 50Hz; V_{ac} = 200 mV

EN RÉSUMÉ : AM vs FM KFM

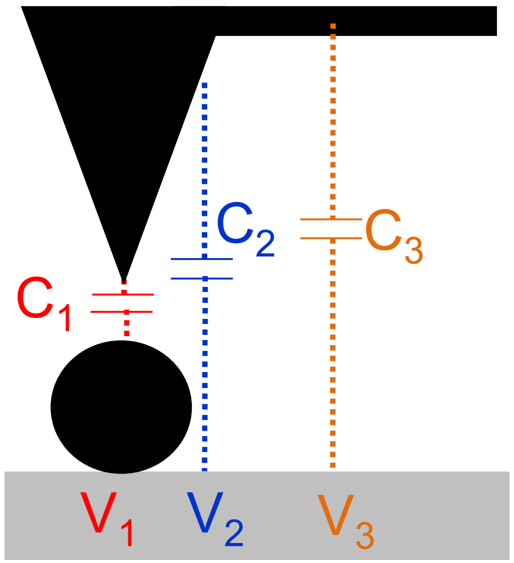
Modulation d'amplitude (AM-KFM)	Modulation de fréquence (FM-KFM)
$F_{\omega_e} = 0 \Leftrightarrow V_{DC} = \mp V_{CPD}$	$\frac{\partial F_{\omega_e}}{\partial z} = 0 \Leftrightarrow V_{DC} = \mp V_{CPD}$

Configurations

	Simple passage		Double passage
	AM-KFM	FM-KFM	AM-KFM
Topographie	Tapping (air) ou non-contact (ultra-vide) : $f_{méca} = f_0$		
Modulation électrique	$f_1 \approx 6{,}3 \times f_0$ ou hors-résonance	$f_1 \ll f_0$	f_0 ou $f_1 \approx 6{,}3 \times f_0$ ou hors-résonance

SOMMAIRE

Side-capacitance effects in AM- and FM-KPFM – 1/5



Nullification of the ω force component (AM-KPFM)

$$\frac{\partial C_1}{\partial z}(V_{DC} - V_1)V_{AC} + \frac{\partial C_2}{\partial z}(V_{DC} - V_2)V_{AC} + \frac{\partial C_3}{\partial z}(V_{DC} - V_3)V_{AC} = 0$$

$$V_{DC} = \frac{\frac{\partial C_1}{\partial z}V_1 + \frac{\partial C_2}{\partial z}V_2 + \frac{\partial C_3}{\partial z}V_3}{\frac{\partial C_1}{\partial z} + \frac{\partial C_2}{\partial z} + \frac{\partial C_3}{\partial z}}$$

KPFM : averaging technique

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H. O. Jacobs *et al.* JAP (1998)

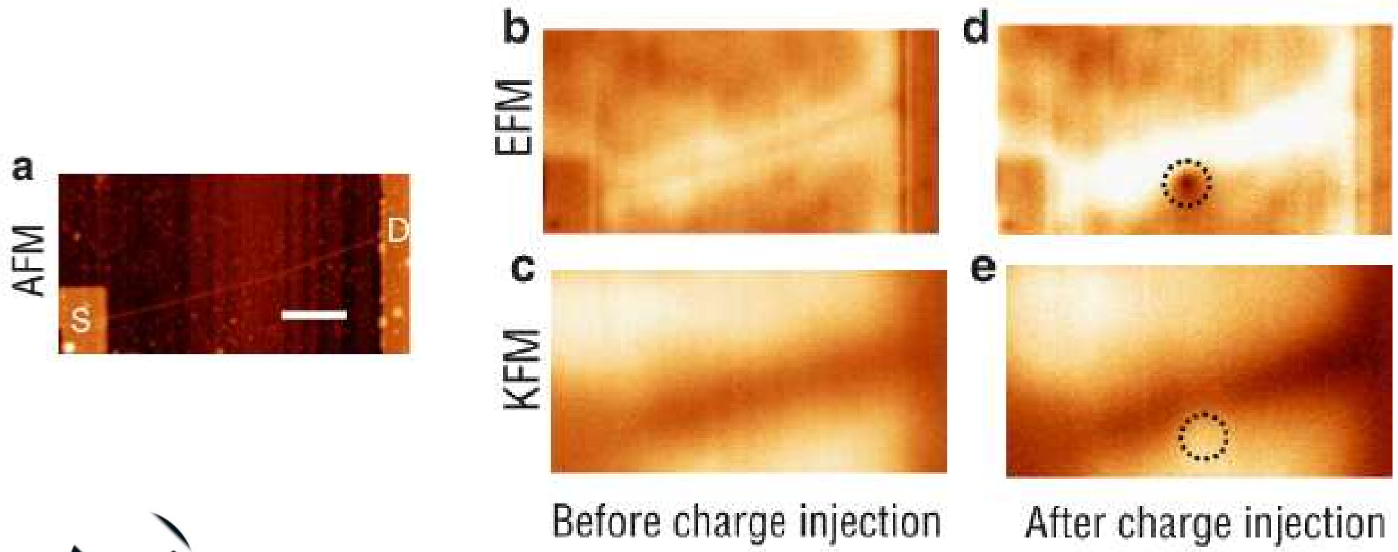
Side-capacitance effects in AM- and FM-KPFM - 2/5

generalization

AM-KPFM $$V_{DC} = \sum \frac{\partial C_i}{\partial z} V_i \Big/ \sum \frac{\partial C_i}{\partial z}$$

FM-KPFM $$V_{DC} = \sum \frac{\partial^2 C_i}{\partial z^2} V_i \Big/ \sum \frac{\partial^2 C_i}{\partial z^2}$$

- intrinsic averaging effects in AM and FM modes
- dC_i/dz less ‘peaked’ at the tip than d^2C_i/dz^2 : less resolution in AM modes

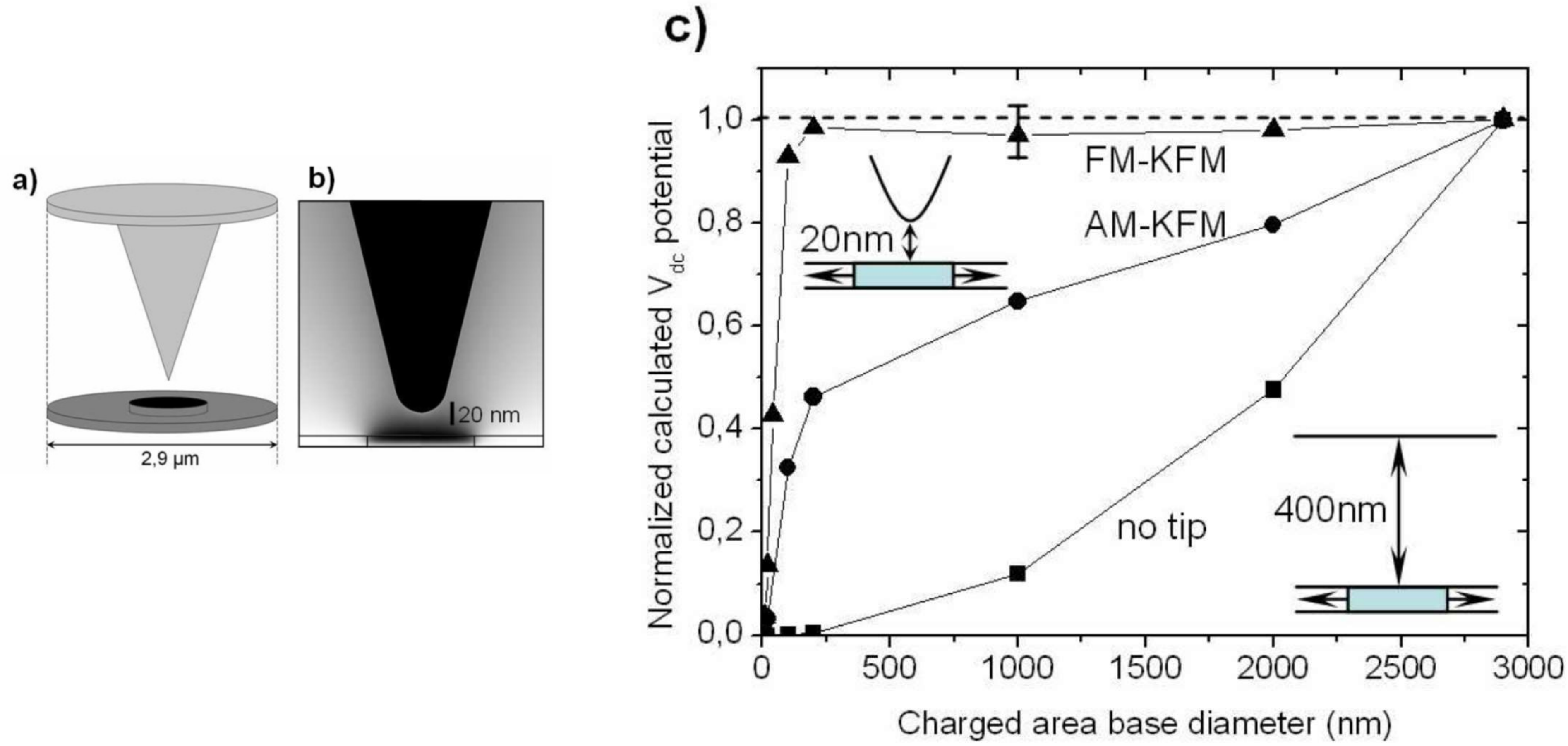


EFM≈FM-KPFM

AM-KPFM

D. Brunel et al. ACS Nano (2010) – ambient air imaging

Side-capacitance effects in AM- and FM-KPFM – 3/5



Both FM- and AM- modes are sensitive to side-capacitance effects at small size

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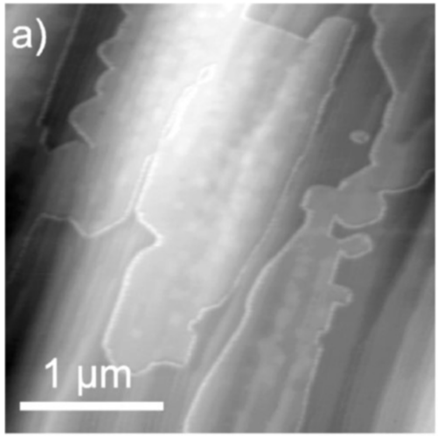
Appl. Phys. Lett. **96** 103119 (2010)

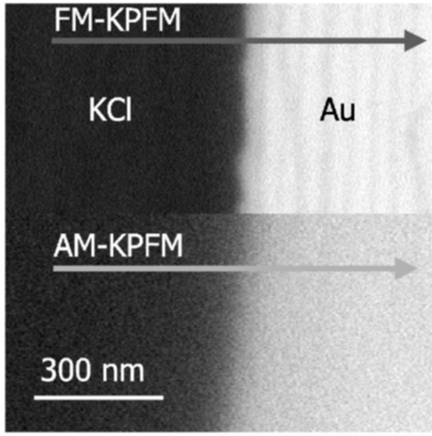
Side-capacitance effects in AM- and FM-KPFM – 4/5

	sensitivity	resolution
AM-KPFM	+	-
FM-KPFM	-	+

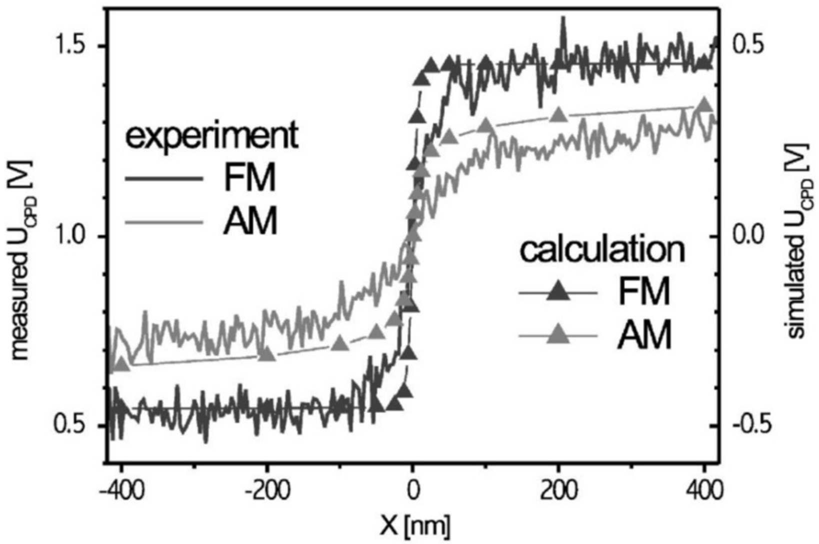
- less ac cross-talk
- no need for a 2nd resonance



KCl islands on Au 111 (topo)



KPFM accross KCl island boundaries



U. Zerweck et al., Phys Rev B 71 125424 (2005)

Side-capacitance effects in AM- and FM-KPFM – 5/5

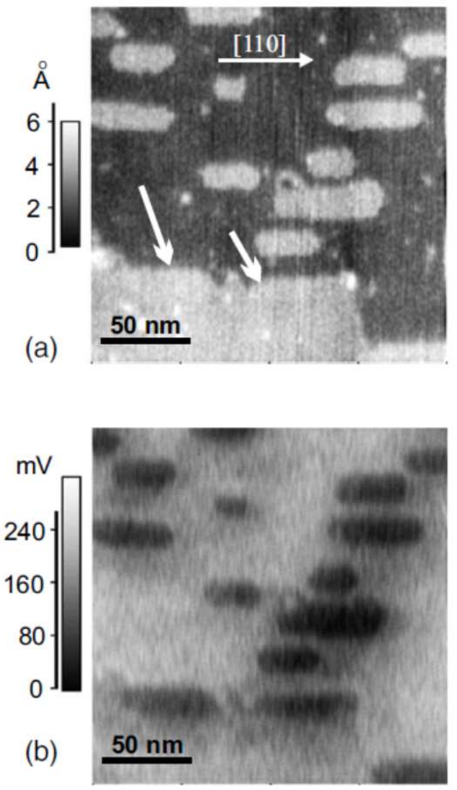
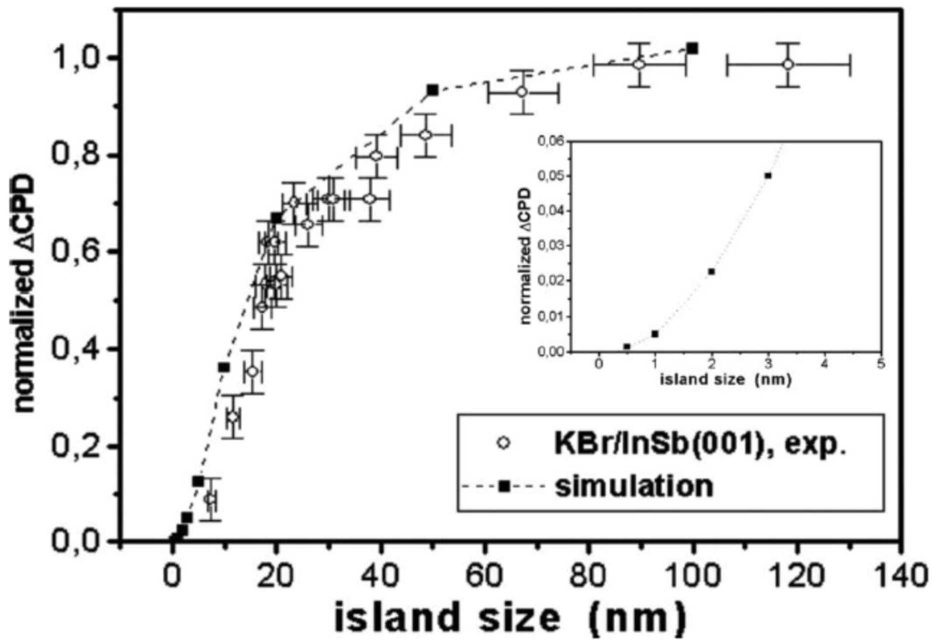


FIG. 1. (a) FM-KPFM topography and (b) ΔCPD images of KBr islands grown on InSb(001) surface (f_0=111, 1 kHz, Δf =−17 Hz). The white arrows indicate the KBr islands, which are topographically not resolved from the substrate terrace.



KBr on InSb(001)

FM-KPFM measurements

convolution in FM mode for structures with smaller size than the tip apex

F. Krok et al., Phys. Rev. B **77**, 235427 (2008)

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SOMMAIRE

Quelques rappels : force capacitive & CPD

Microscopie à force électrostatique (EFM)

Détection de charge(s) ?

Microscopie à sonde de Kelvin (KFM)

Modulation d'Amplitude (AM) vs Modulation de Fréquence (FM)

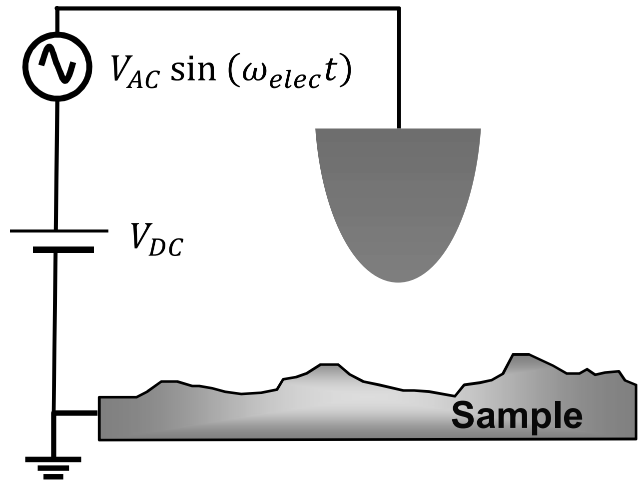
Effet des capacités latérales

Acquisition en boucle ouverte (OL-KFM)

KFM & DFRT (Dual Frequency Resonant Tracking)

Travail de sortie : mesure quantitative ?

KFM en boucle ouverte (OL-KFM)



$$V = V_{DC} - V_{CPD} + V_{AC}\sin(\omega_{elec}t)$$

$$\omega_{elec} \ll \omega_0$$

Force électrostatique

Modulation d'Amplitude → AM-KFM sans boucle PID

$$F_{\omega_{elec}} = \frac{\partial C}{\partial z}(V_{DC} - V_{CPD})\,V_{AC}$$

$$F_{2\omega_{elec}} = -\frac{1}{4}\frac{\partial C}{\partial z}V_{ac}^2$$

$$\frac{F_\omega}{F_{2\omega}} = -\frac{4}{V_{AC}}(V_{DC} - V_{CPD})$$

$$V_{CPD} = V_{DC} - \frac{{F_\omega}/{F_{2\omega}} \times V_{AC}}{4}$$

OL-KFM : MODES D'ACQUISITION

Simple passage

Excitation mécanique à la résonance du levier ($f_{méca} = f_0$)

Tapping (Air)
ou
nc-AFM (Vide, UHV)

+

Modulation électrique $f_{élec} \ll f_{méca}$

Démodulation à $f_{élec}$ et à $2 \times f_{élec} \rightarrow$ amplitudes correspondantes

Double passage

1er passage

Excitation mécanique à la résonance du levier ($f_{méca} = f_0$)

Tapping (Air)
ou
nc-AFM (Vide, UHV)

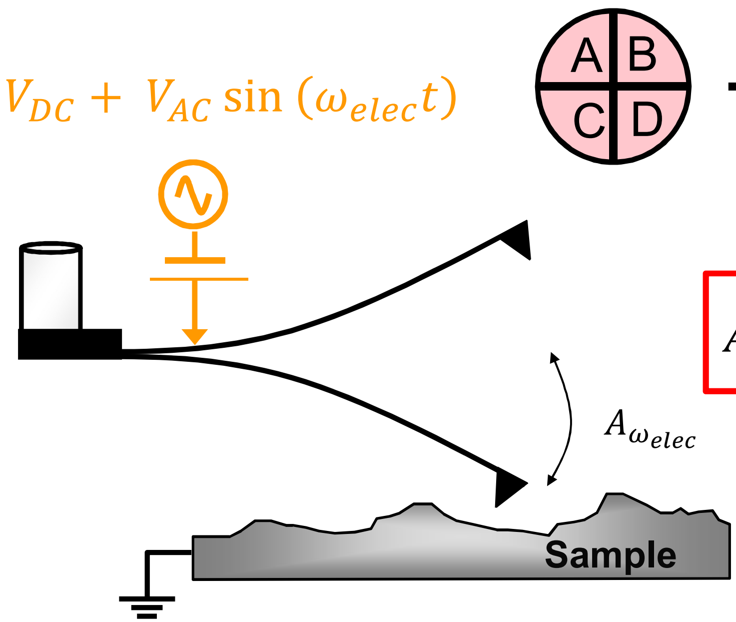
2ème passage : lift

Modulation électrique $f_{élec} \ll f_{méca}$

Démodulation à $f_{élec}$ et à $2 \times f_{élec} \rightarrow$ amplitudes

Hauteur de lift (20 à 100 nm)

AM-KFM : EN PRATIQUE



Détection synchrone ← Réf. ω_{elec}

$$A_{\omega_{elec}} = G_\omega \frac{\partial C}{\partial z}(V_{CPD} - V_{DC})V_{AC}$$

$$A_{2\,\omega_{elec}} = -G_{2\omega}\frac{\partial C}{\partial z}\frac{V_{AC}^2}{4}$$

$$V_{CPD} = V_{DC} - \frac{A_\omega}{A_{2\omega}}\frac{V_{AC}}{4} \times \frac{1}{K}$$

- **Calibration préalable de la valeur de K**
- **Calcul a posteriori du V_{CPD}**

CALIBRATION

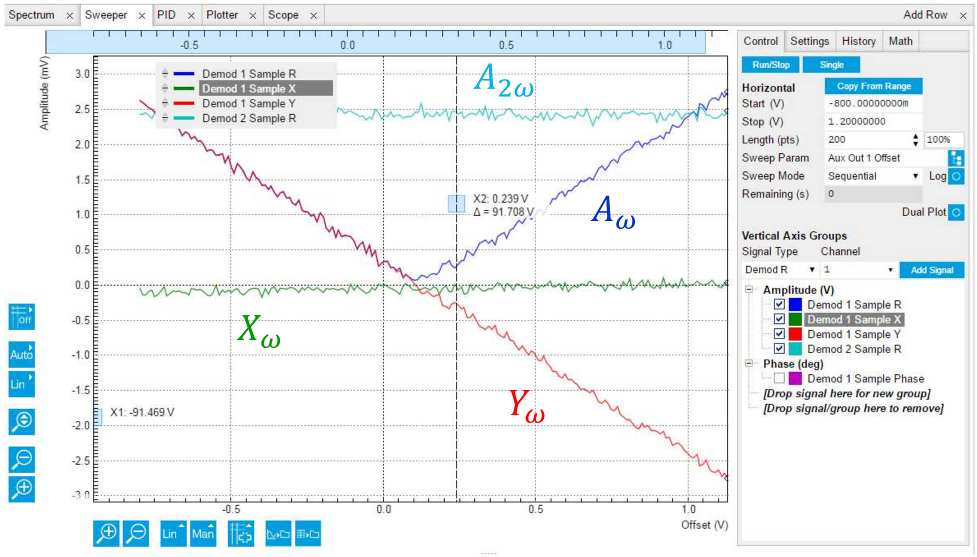
- **Theory**

$$A_{\omega} = G_{\omega}\frac{\partial C}{\partial z}(V_{CPD} - V_{DC})V_{AC} > 0$$

$$A_{2\omega} = G_{2\omega}\frac{\partial C}{\partial z}\frac{V_{AC}^2}{4} > 0$$

- **Phase adjustement**

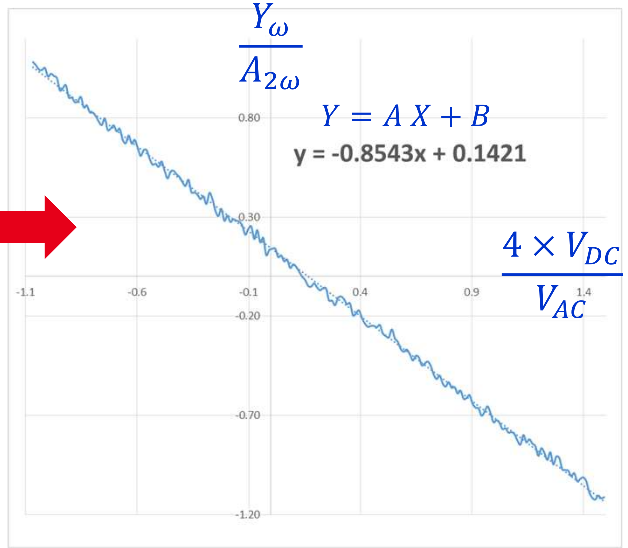
$$X_{\omega} = A_{\omega}\cos(\varphi) = 0 \qquad Y_{\omega} = A_{\omega}sin(\varphi)$$



$$\frac{Y_{\omega}}{A_{2\omega}} = \frac{G_{\omega}}{G_{2\omega}}\frac{4}{V_{AC}}(V_{DC} - V_{CPD})$$

K_{gain}

$$V_{CPD} = V_{DC} - \frac{Y_{\omega}}{A_{2\omega}} \times K_{gain}\frac{V_{AC}}{4}$$



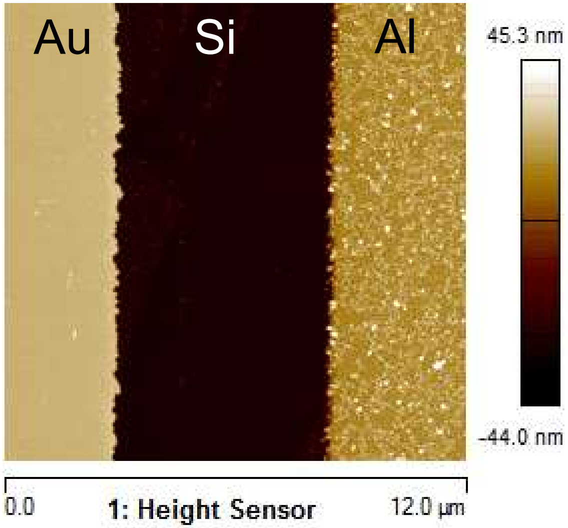
$$A = K_{gain} = -0.8543$$

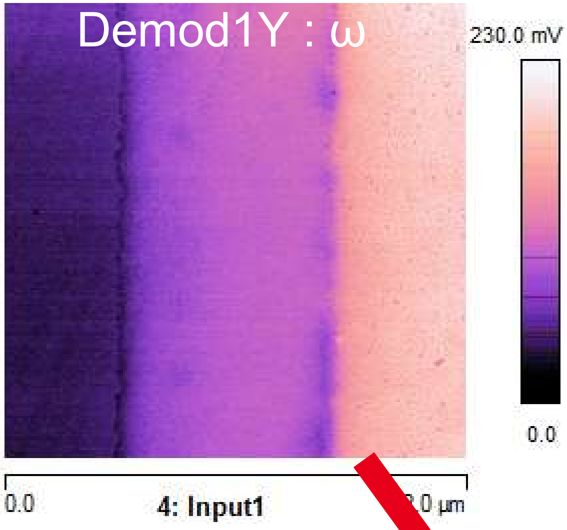
$$B = -\frac{4X_{gain}V_{CPD}}{V_{AC}} = 0.1421$$

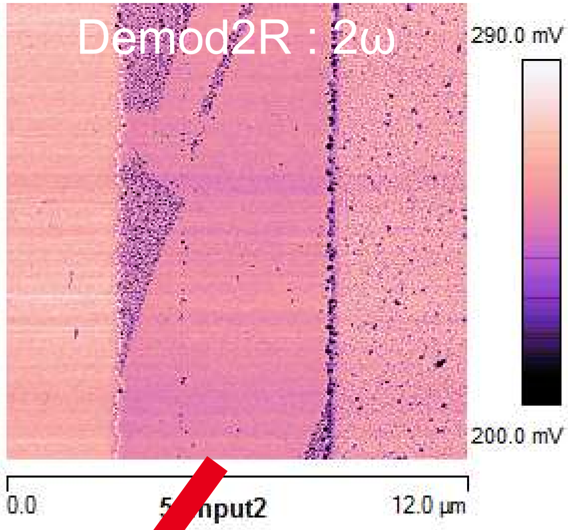
$$V_{CPD}(@0V) = 0.12\ V$$

Mesure sur une couche d'Au
V_{ac}=3 V

EXAMPLE





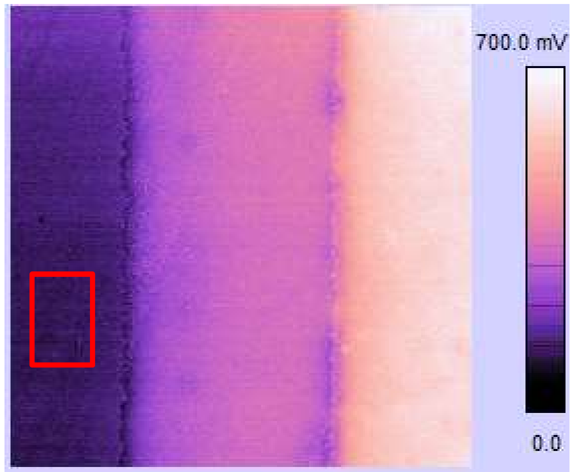


Calculated V_{CPD} (with Nanoscope Analysis)

$$V_{CPD} = -\frac{V_{ac}}{4K}\frac{Demod1Y}{Demod2R}$$

0,878

$$V_{CPD} = 0.13\ V$$



Calculated V_{CPD}

V_{ac}=3 V

X_{gain}=-0.854

OL-KFM en modulation de fréquence ?

Gradient de force électrostatique

FM-KFM sans boucle PID

$$\frac{\partial F_{z,\omega_{elec}}}{\partial z} = \frac{\partial^2 C}{\partial z^2} (V_{DC} - V_{CPD})\, V_{AC}$$

$$\frac{\partial F_{z,2\omega_{elec}}}{\partial z} = -\frac{1}{4} \frac{\partial^2 C}{\partial z^2} V_{AC}^2$$

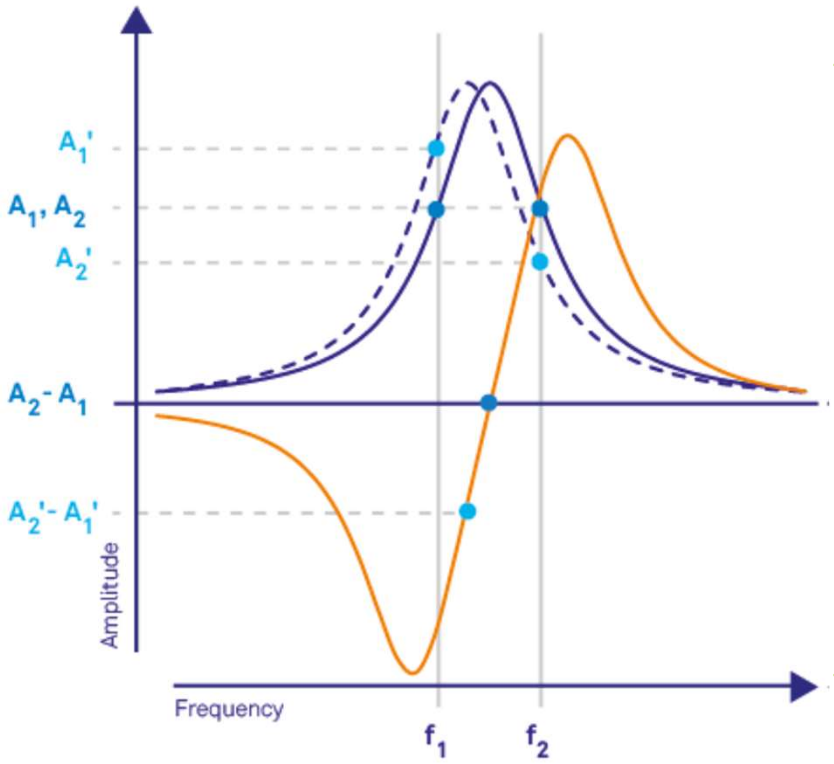
$$V_{CPD} = V_{DC} - \frac{{F'_{\omega}}/{F'_{2\omega}} \times V_{AC}}{4}$$

EFM modulé (ac-EFM)

Imagerie de charges et de prop. diélectriques

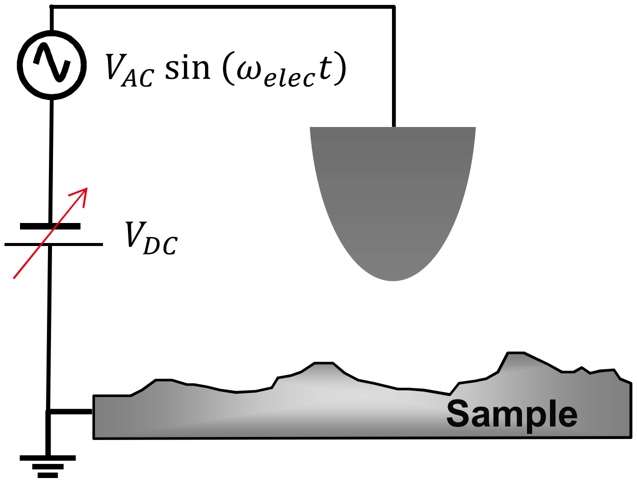
SOMMAIRE

DUAL FREQUENCY RESONANT TRACKING (DFRT)



- **Méthode de suivi de fréquence**
- **Principe**
  - Excitation bimodal autour de la fréquence de résonance $(f_0 \pm f_{mod})$
  - Mesure simultanée des amplitudes correspondantes → variation monotone de la difference (ΔA)
  - Une boucle de contre-réaction annule ΔA en ajustant la fréquence de resonance f_0
- **Applications**
  - Piezo Force Microscopy (PFM)
  - Contact resonance (CR-AFM)
  - Kelvin force microscopy (KFM)
  - EFM …

KFM en mode DFRT



- **Acquisition en mode AM-KFM → suivi de la fréquence de résonance en mode DFRT**

$$V = V_{DC} - V_{CPD} + V_{AC}\sin(\omega_{elec}t)$$

Force électrostatique

$$F_{\omega_{elec}} = \frac{\partial C}{\partial z}(V_{DC} - V_{CPD})\, V_{AC}$$

- **Annulation de la composante à ω_{elec} de la force électrostatique**
  - $V_{DC} = V_{CPD}$

OL-KFM : MODES D'ACQUISITION

Simple passage

Excitation mécanique à la résonance du levier ($f_{méca} = f_0$) + Excitation bimodal à $f_{élec} \pm f_{mo} = f_1 \pm f_m$

Tapping (Air)
ou
nc-AFM (Vide, UHV)

+

Modulation électrique $f_{élec} = f_1$

Démodulation à $f_{élec} \rightarrow$ amplitude

Double passage

1er passage

Excitation mécanique à la résonance du levier ($f_{méca} = f_0$) + Excitation bimodal à $f_{élec} \pm f_{mod} = f_1 \pm f_m$

Tapping (Air)
ou
nc-AFM (Vide, UHV)

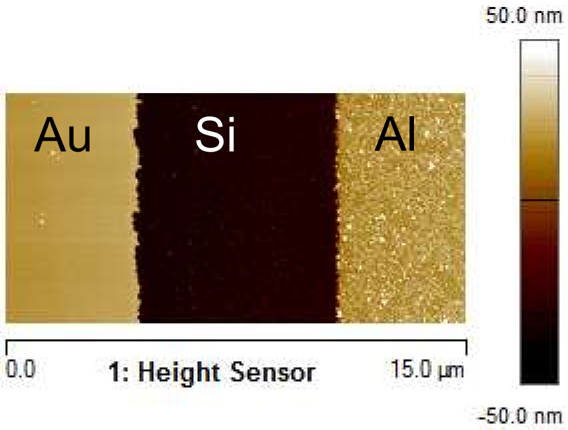
2ème passage : lift

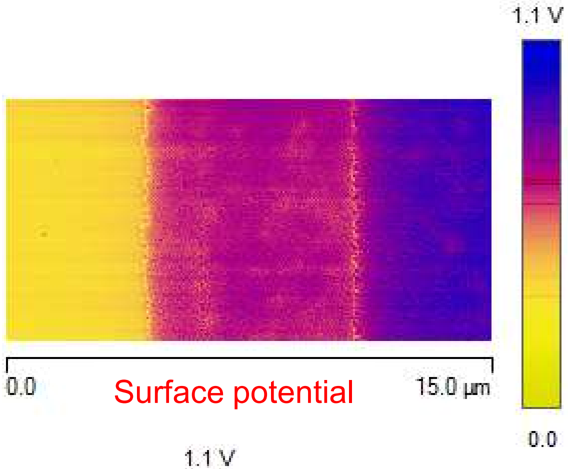
Modulation électrique $f_{élec} = f_1$

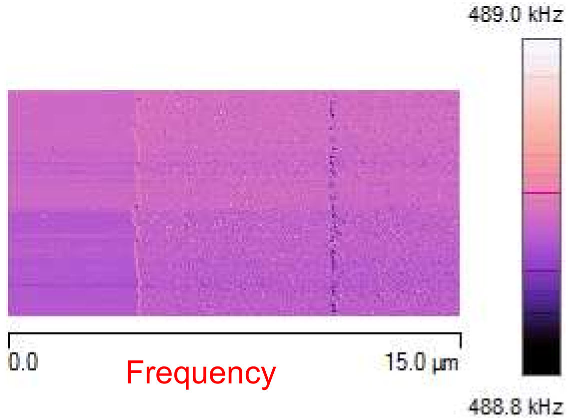
Démodulation à $f_{élec} \rightarrow$ amplitude
Hauteur de lift (20 à 100 nm)

EXEMPLE

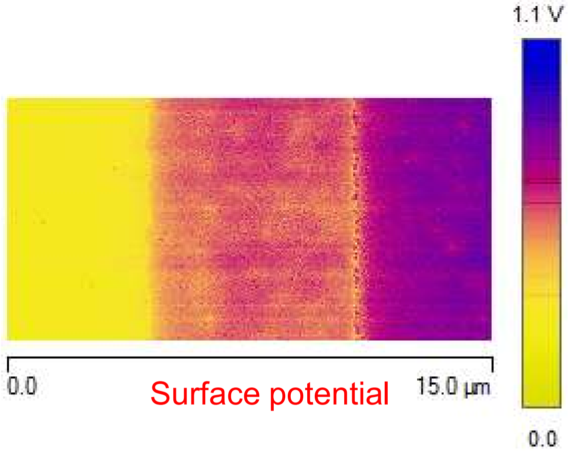
DFRT KFM : V_{ac}=500 mV, sideband: 9 mV, 1.5 KHz

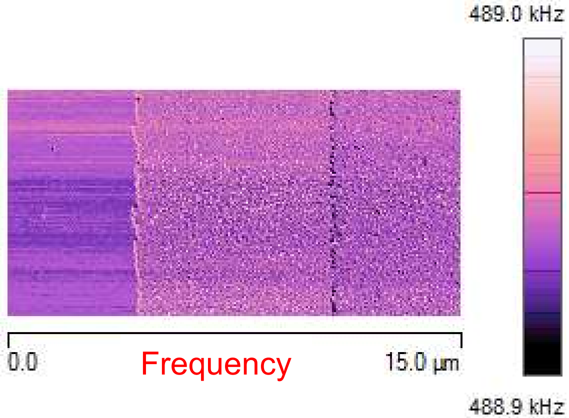






AM-KFM
V_{ac}=500 mV,
488.6 KHz

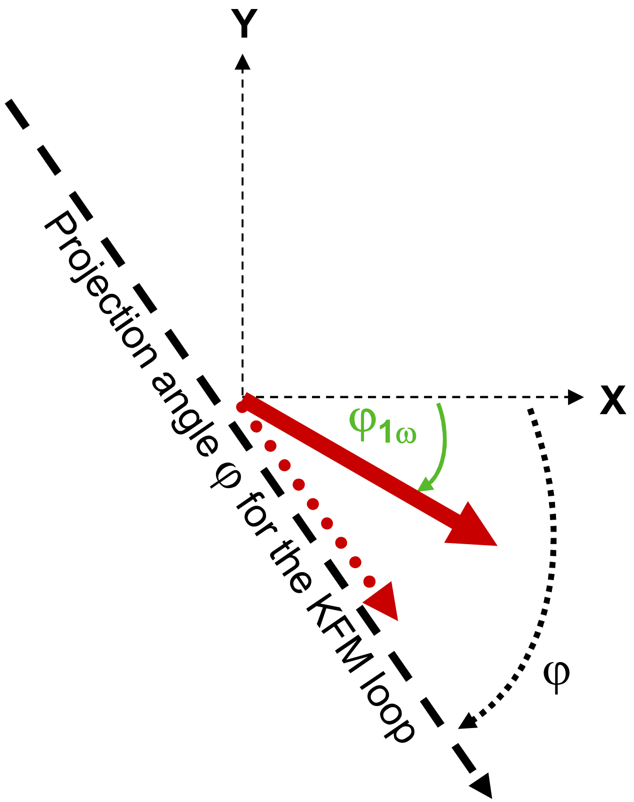


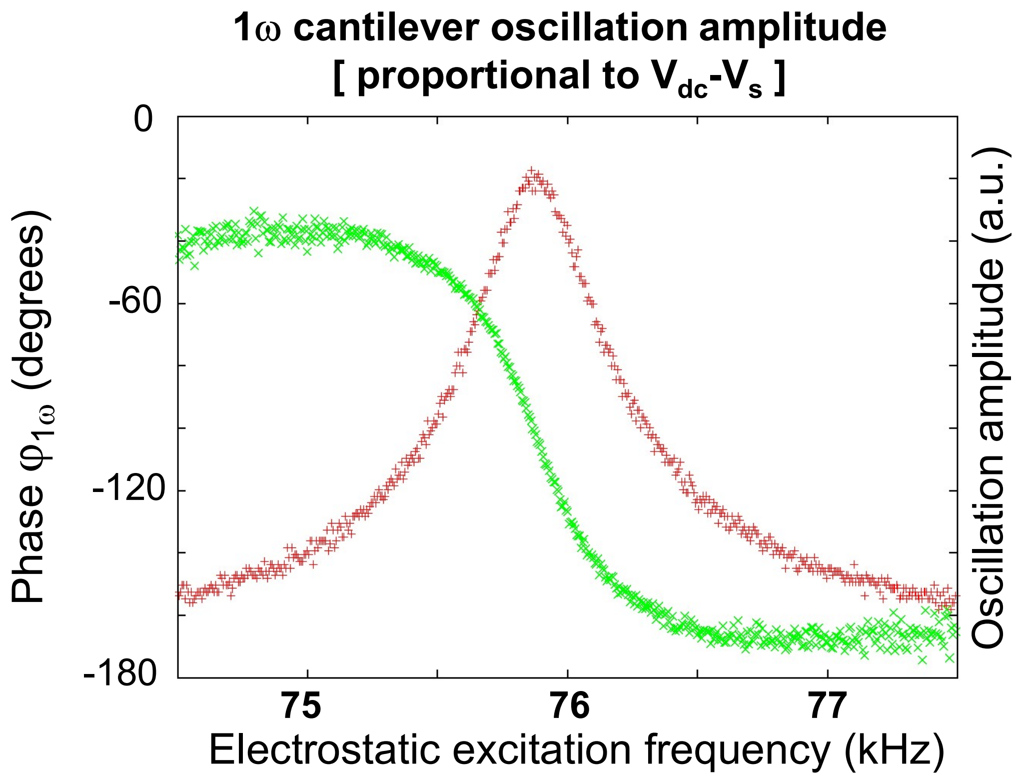


« EFM »-DFRT
Vdc=0V
sideband: 9 mV, 1.5 KHz

SOMMAIRE

Practical operation principle

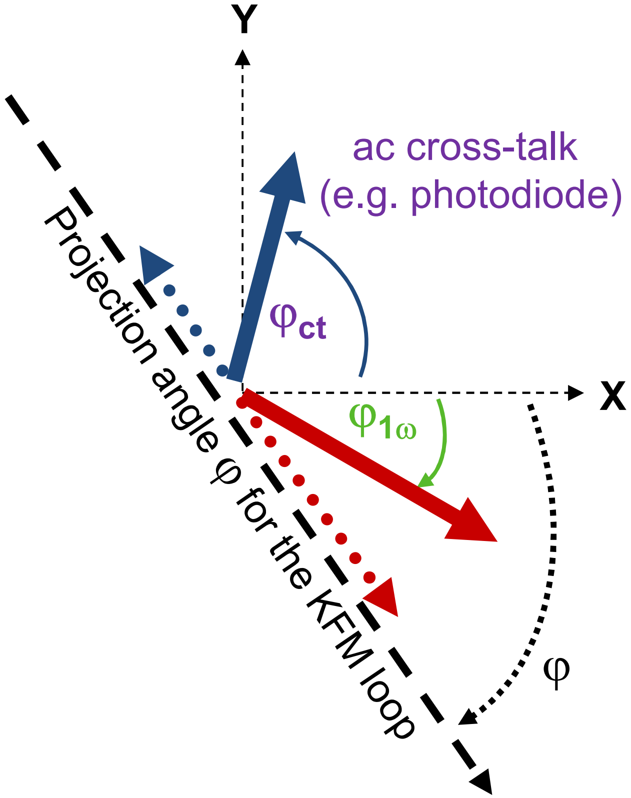




projection angle necessary for the KFM feedback loop

KFM "equation" : $dC/dz.\ (V_{dc}-V_s).V_{ac}.\ \cos(\phi_{1\omega}-\phi) = 0$

Practical operation principle … with ac cross-talks



KFM "equation"

$$dC/dz.\ (V_{dc}-V_s).V_{ac}.\ \cos(\phi_{1\omega}-\phi) + A_{ct}\ .V_{ac}\ .\ \cos(\phi_{ct}-\phi) = 0$$

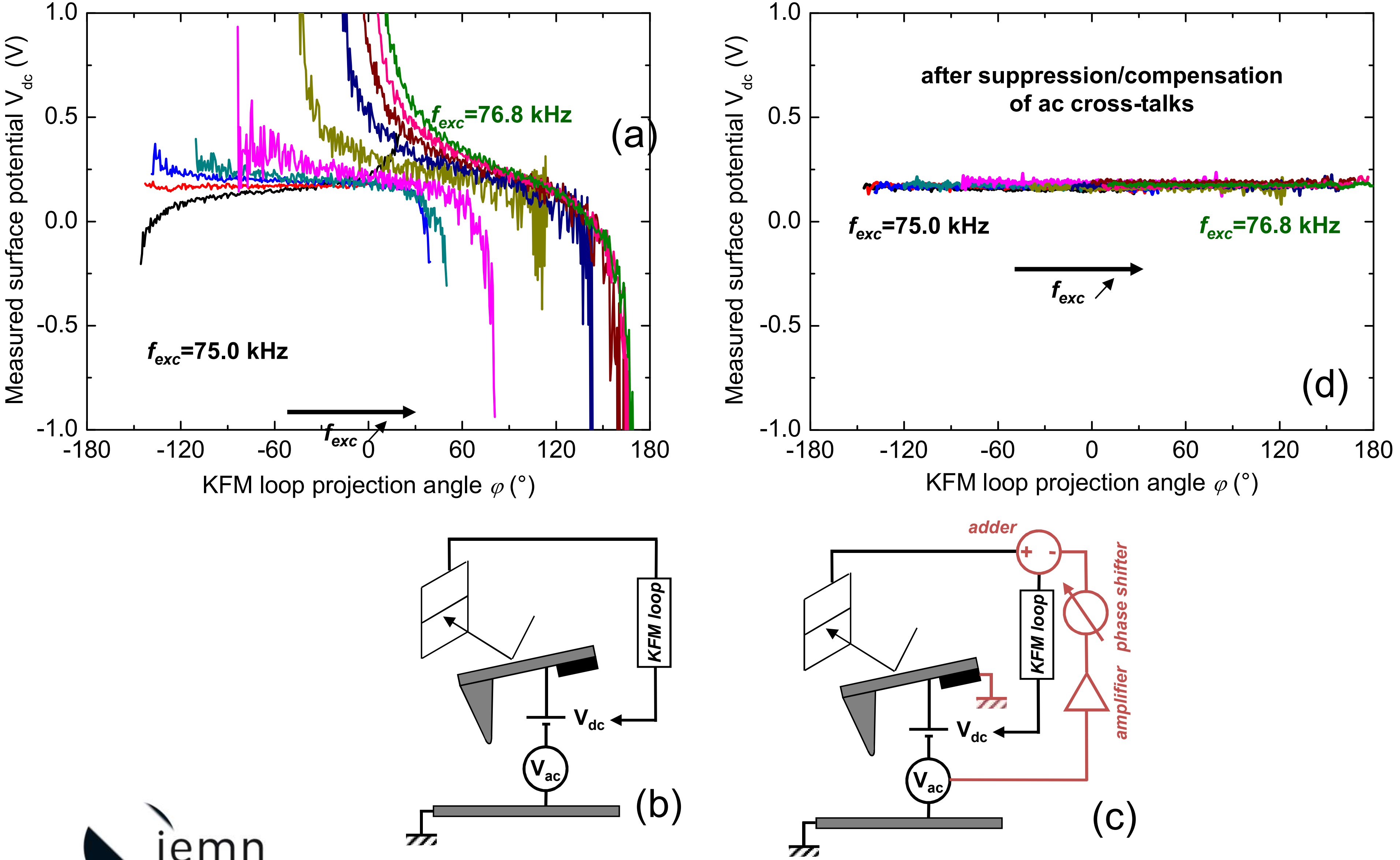
$$V_{dc}=V_s+ \underbrace{A_{ct}\ .\ \cos(\phi_{ct}-\phi)\ /\ dC/dz\ \cos(\phi_{1\omega}-\phi)}$$

This term depends ☹ ☹ ☹

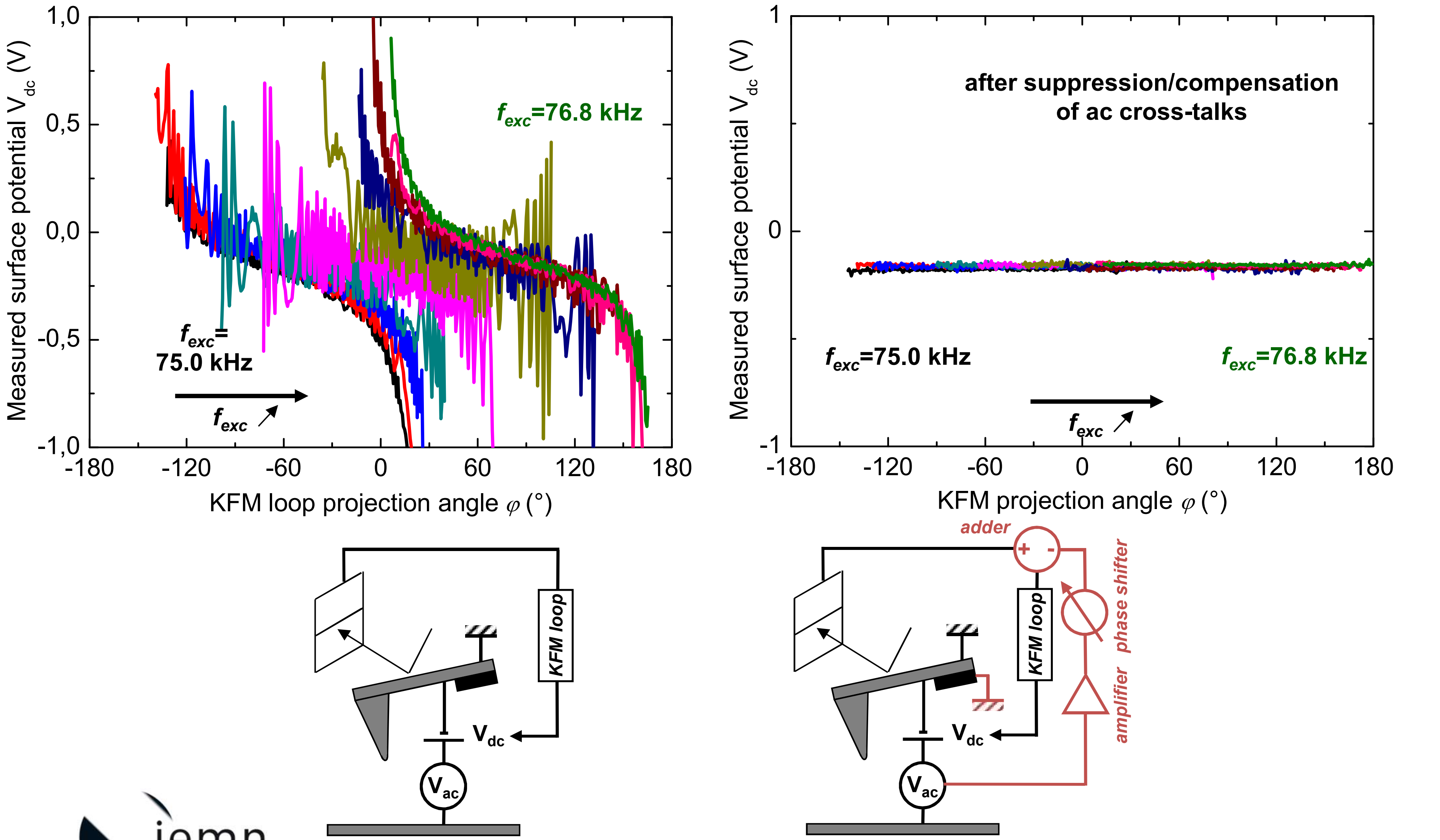
- on ϕ ("drive phase")
- on $\phi_{1\omega}$ (excitation frequency)
- on z (via dC/dz)

In practice (Brüker) : photodiode + mechanical ac-cross-talks

Cross-talk suppression/compensation



Cross-talk suppression/compensation



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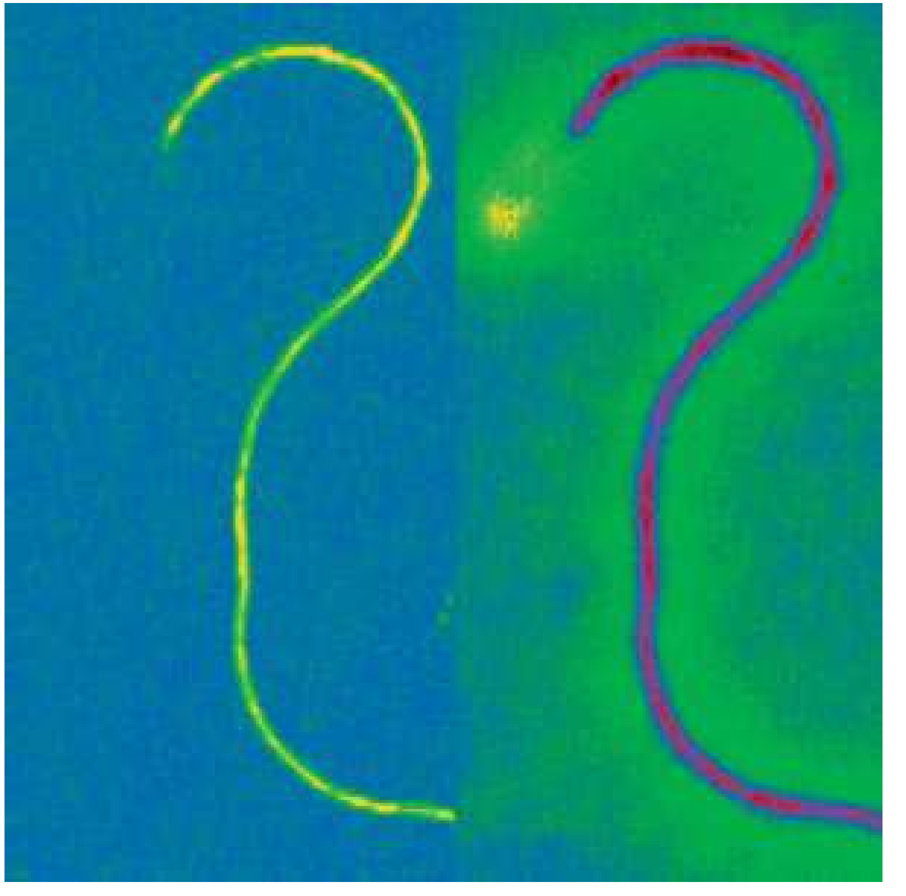
Rev. Sci. Inst. 2010

Questions ?



MICROSCOPIE EFM/KFM : CONFIGURATIONS SIMPLE PASS

	EFM	Modulation d'Amplitude			Modulation de fréquence	
		KFM	DFRT	OL-KFM	KFM	OL-KFM
Topographie	en mode Tapping (air) ou en mode non-contact (ultra-vide)					
Simple Passage	✘	✓	✓	✓	✓	✓
Excitation mécanique $f_{méca}$	✘	f_0	f_0 $f_1 \pm f_{mod}$	f_0	f_0	f_0
Modulation électrique $f_{élect}$	✘	f_1 *ou hors résonance*	f_1	*Basse fréquence* $\ll f_0$		
Démodulation	✘	Amplitude @ f_{elect}		Amplitudes @ f_{elect} et à $2f_{elect}$	Phase @ f_0 puis Amplitude @ $f_{élect}$	Phase @ f_0 puis Amplitudes @ $f_{élect}$ et à $2f_{élect}$

f_0 fréquence fondamentale du levier / f_1 1ère harmonique du levier / f_{mod} fréquence de modulation < 2 kHz

MICROSCOPIE EFM/KFM : CONFIGURATIONS DOUBLE PASS

	EFM	Modulation d'Amplitude			Modulation de fréquence	
		KFM	DFRT	OL-KFM	KFM	OL-KFM
Topographie	en mode Tapping (air) ou en mode non-contact (ultra-vide)					
Double Passage	✓	✓	✓	✓	✓	✓
Excitation mécanique $f_{méca}$ **(1er passage)**	f_0	f_0	f_0	f_0	f_0	f_0
Excitation mécanique $f_{méca}$ **(2ème passage)**	f_0	-	$f_0 \pm f_{mod}$ ou $f_1 \pm f_{mod}$	-	f_0	f_0
Modulation électrique $f_{élect}$ **(2ème passage)**	-	f_0 *ou* f_1 *ou hors résonance*	f_0 *ou* f_1	*Basse fréquence* $\ll f_0$		
Démodulation	Phase @ f_0	Amplitude @ f_{elect}		Amplitudes @ $f_{élect}$ et à $2f_{élect}$	Phase @ f_0 puis Amplitude @ $f_{élect}$	Phase @ f_0 puis Amplitudes @ $f_{élect}$ et à $2f_{élect}$

f_0 fréquence fondamentale du levier / f_1 1ère harmonique du levier / f_{mod} fréquence de modulation < 2 kHz

RÉFÉRENCES

- **S. Sadewasser and Thilo Glatzel « Kelvin probe force microscopy » Springer 2012**
- **B. Bushan « Scanning probe microscopy in nanoscience and nanotechnology » Springer 2009**
  - Chapter 4 Electrostatic Force Microscopy and Kelvin Force Microscopy as a Probe of the Electrostatic and Electronic Properties of Carbon Nanotubes