Atom Probe Tomography and Semiconductor Nanostructures: Principles, Applications, and Correlative Techniques.

Groupe de Physique des Matériaux

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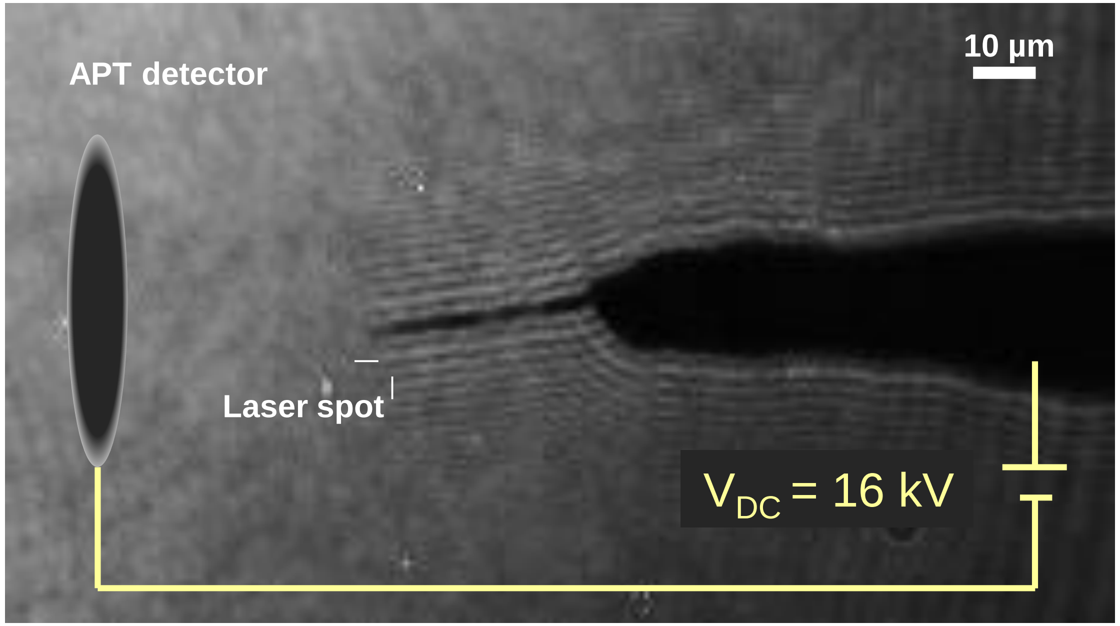
GDR PULSE Summer School, Porquerolles, 3-8/7/2021

2-8 july 2021
PULSE school
Epitaxy bases and promises

Groupe de Physique des Matériaux, Rouen

A nanometric tip in a high electric field

$$\sigma = \frac{1}{2} \varepsilon_0 F^2$$



Diamond Micro-Nano-needle glued on a tungsten tip

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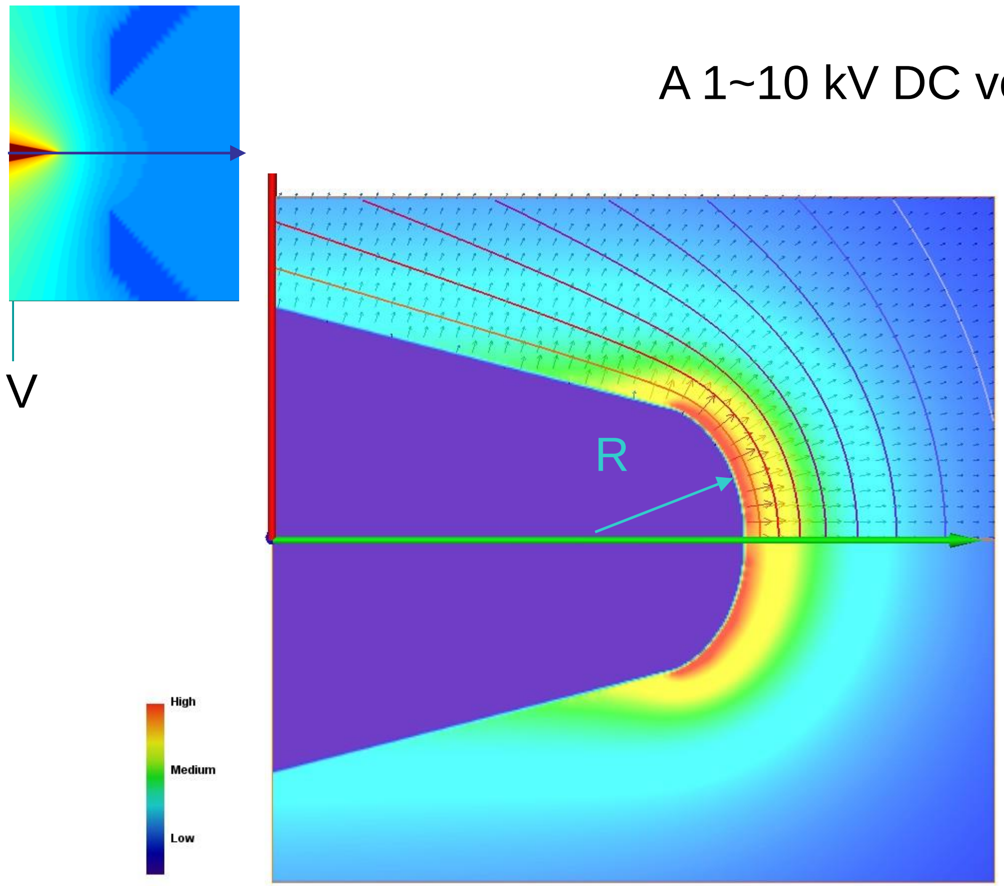
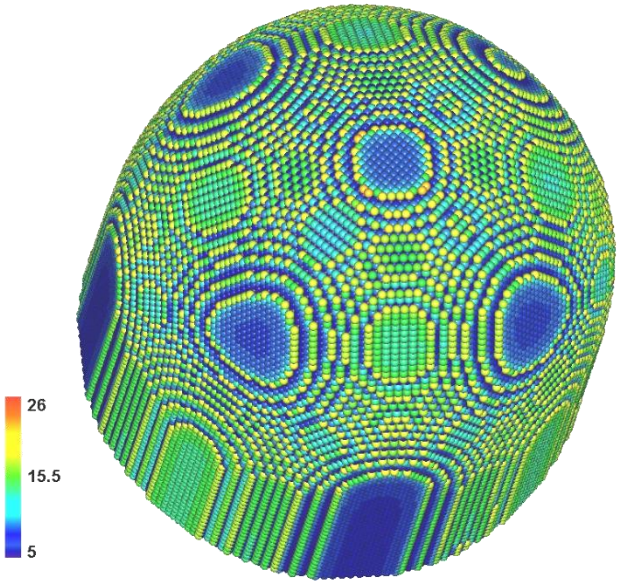
A nanometric tip in a high electric field

A 1~10 kV DC voltage applied to a specimen tip



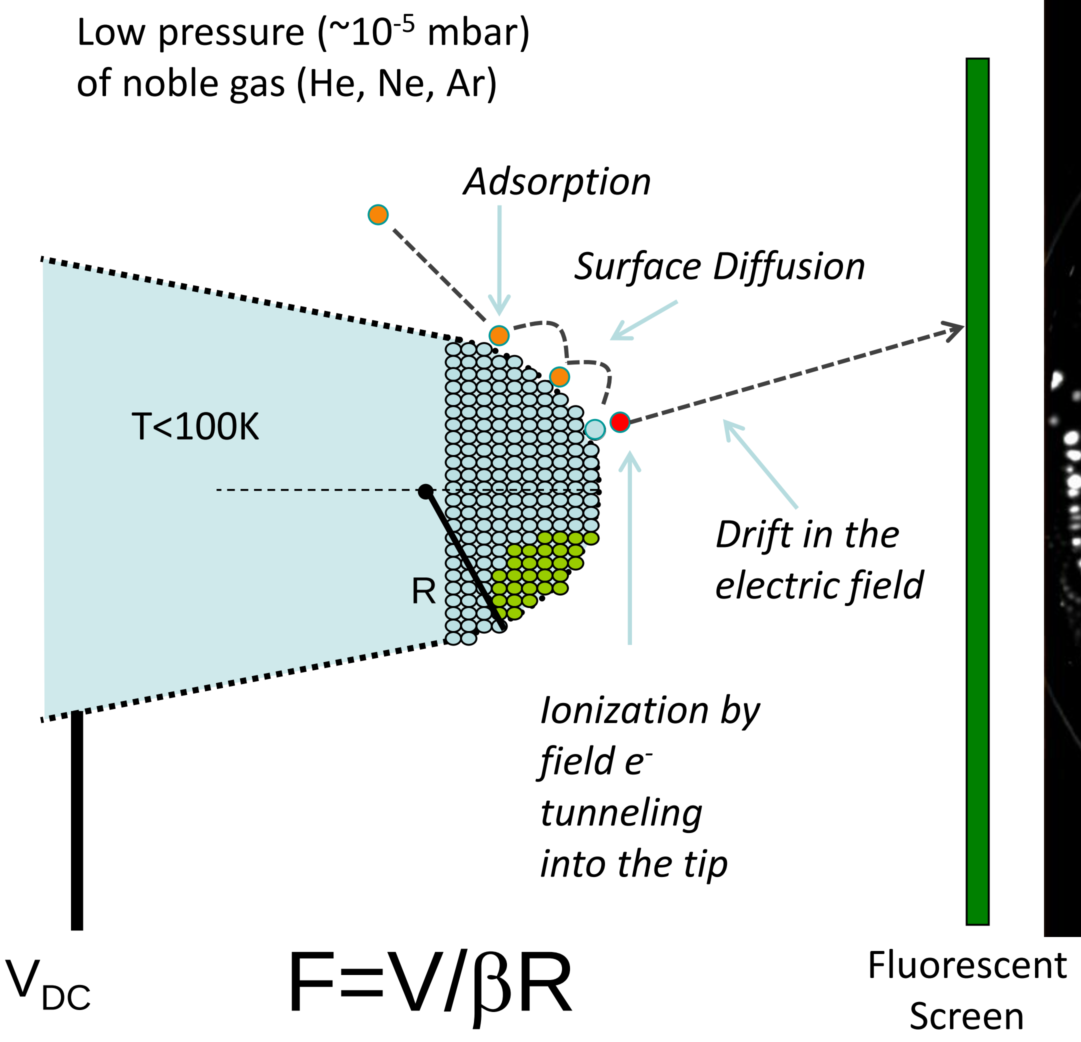


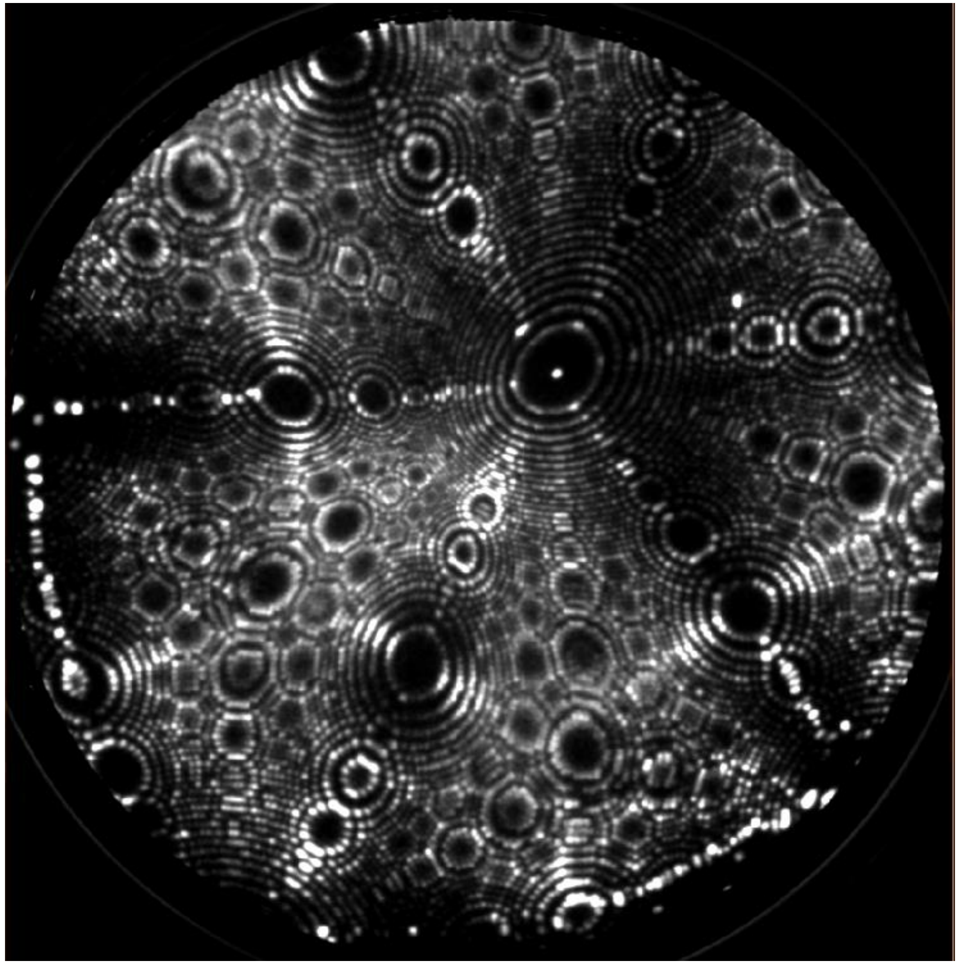
Surface electric field (V/nm)

$F = V/\beta R$

$\sigma = \frac{1}{2} \varepsilon_0 F^2$

Field ion microscopy



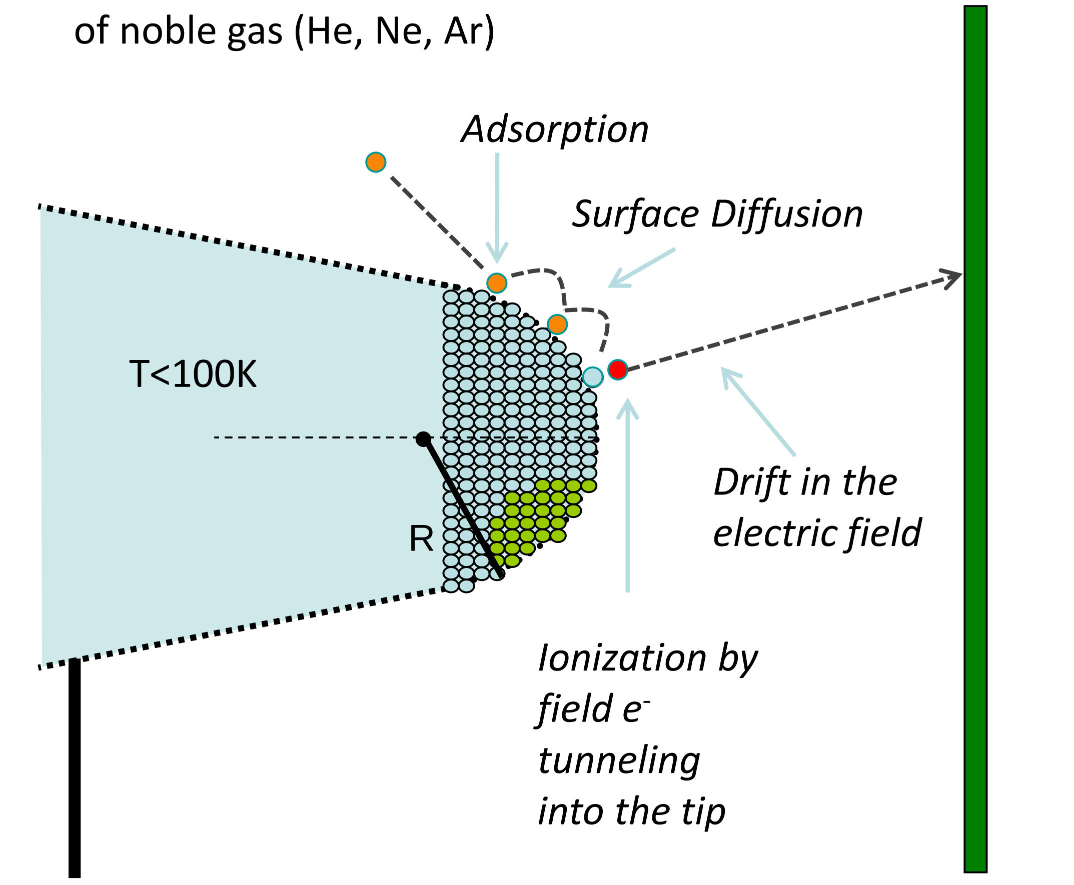


FIM of Au tip (F. Vurpillot, F. Danoix)

E. Müller, 1951

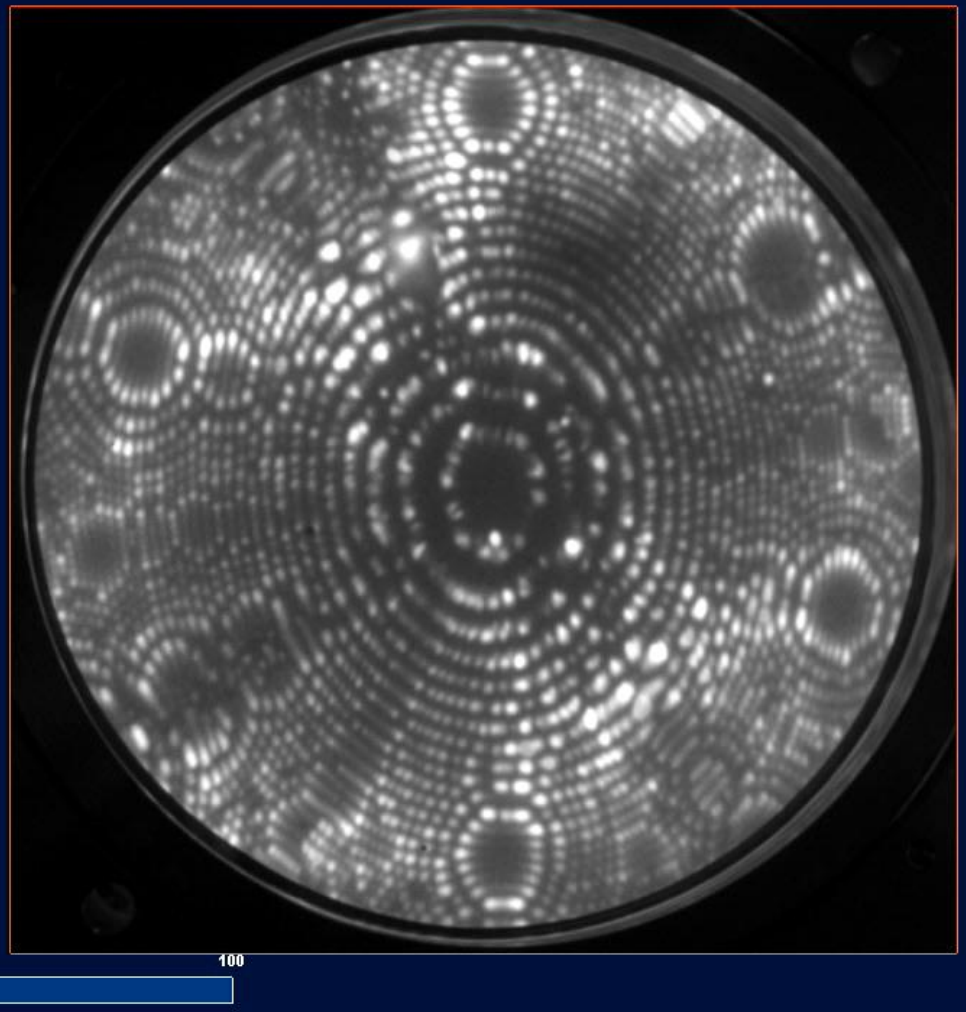
First technique to allow for the observation of atoms in the direct space

Field ion evaporation

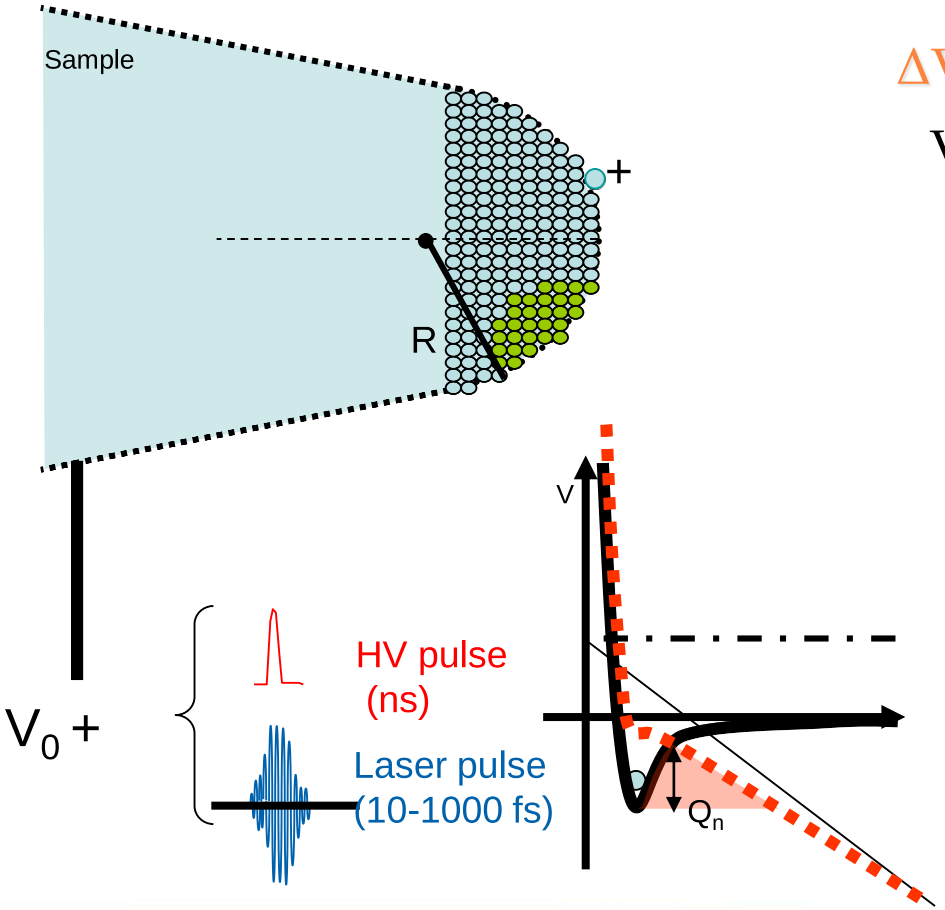


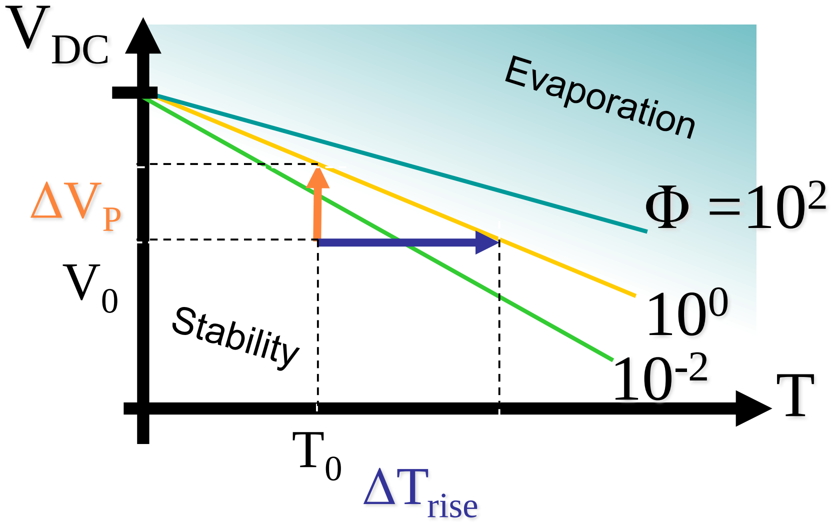
$$F = V/\beta R$$

FIM of evaporating steel tip (F. Danoix)



Controlling Field Ion Evaporation in Time





Intrinsic evaporation time ~1ps

$$K_e = \nu_0 \exp\left(-\frac{Q_n(F)}{k_B T_0}\right)$$

$$Q_n(F) \approx Q_0'\left(1 - \frac{F}{F_{evap}}\right)$$

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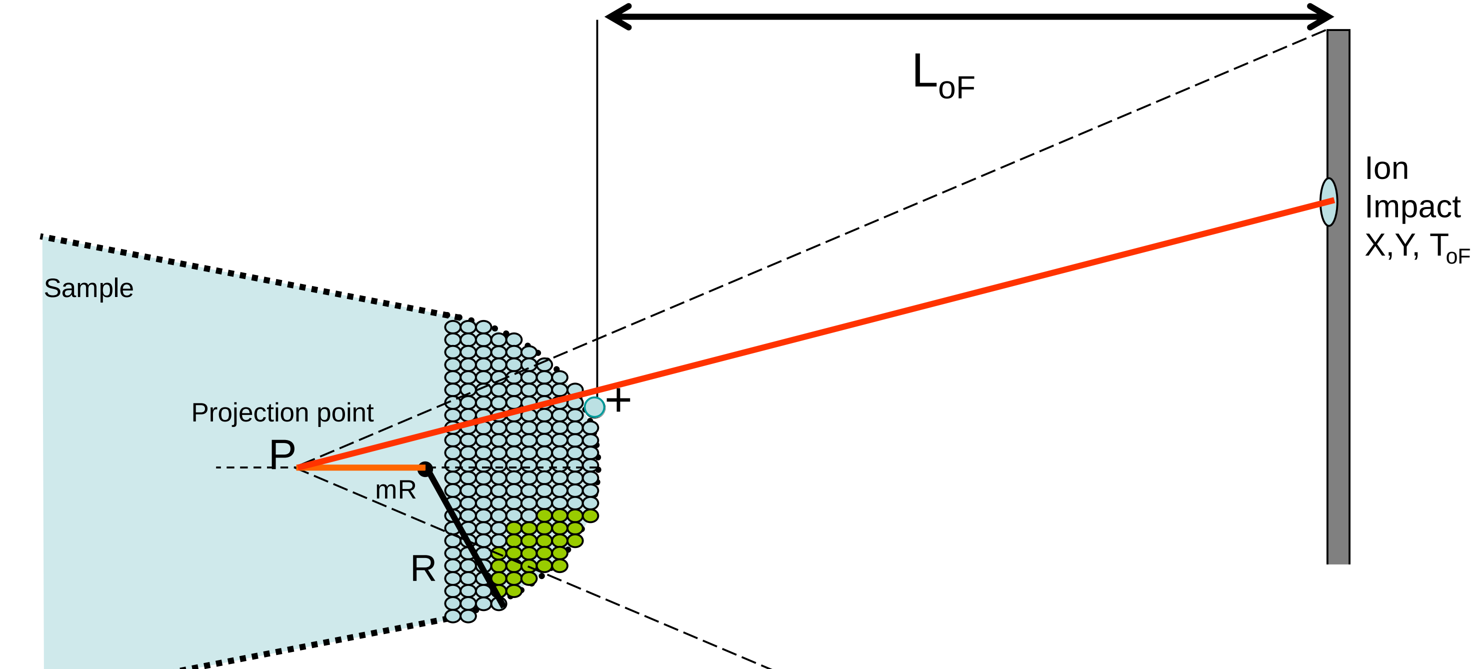
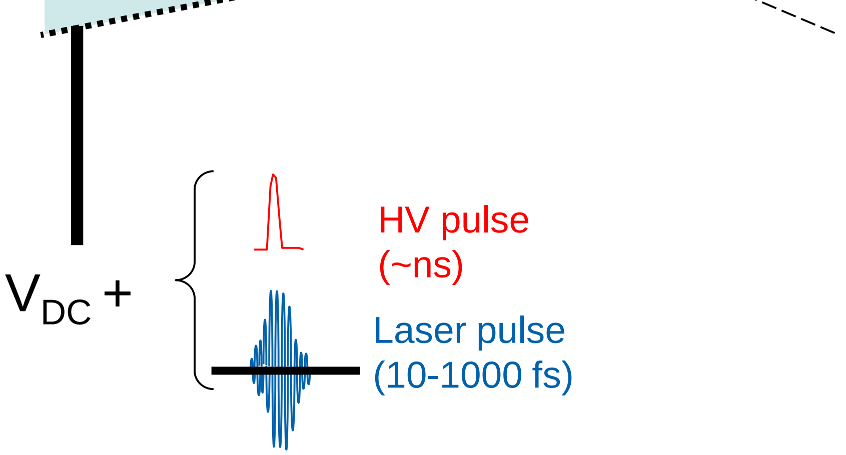
Time-of-Flight Spectrometry





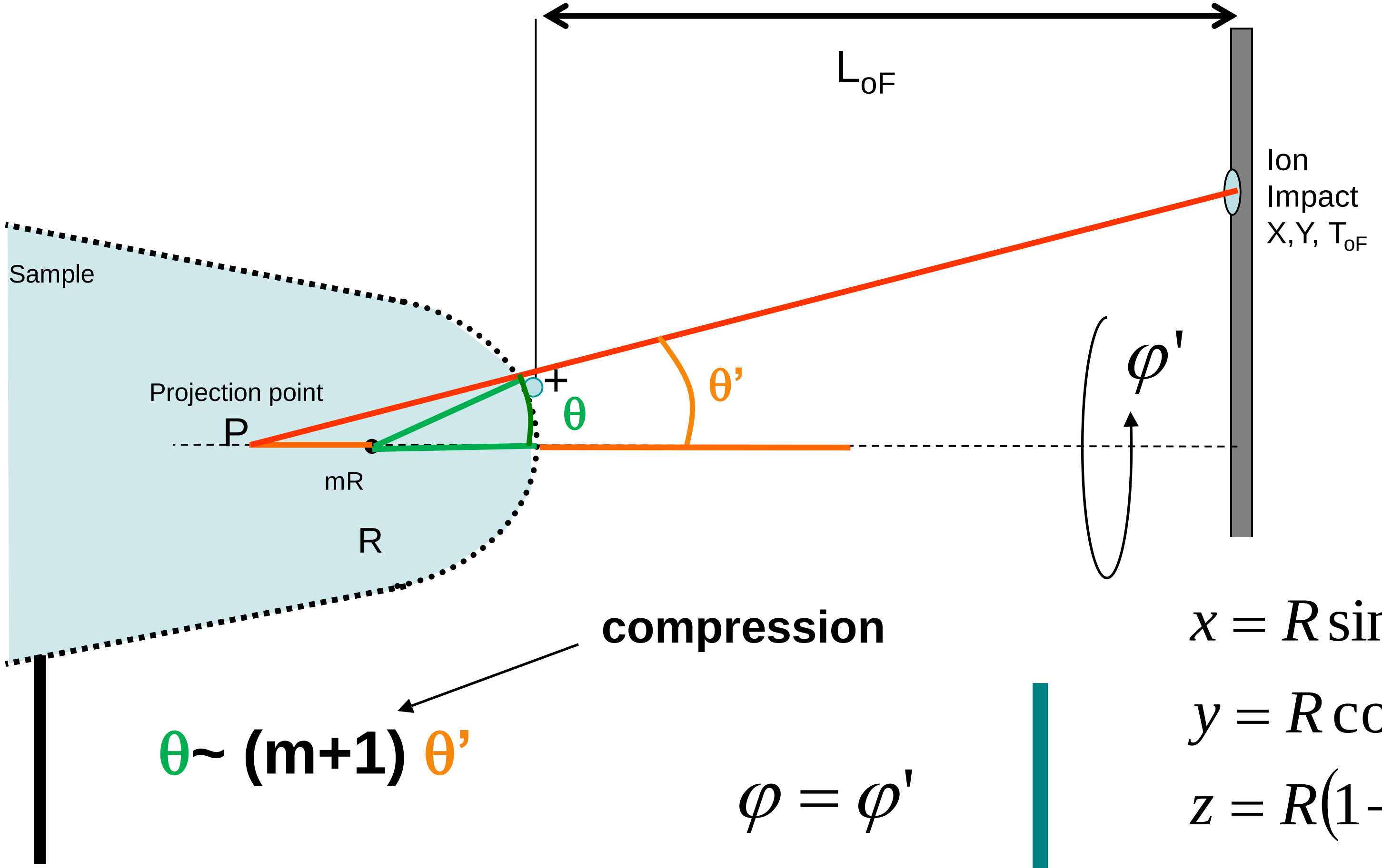
Chemical identification (mass-over-charge ratio) by time-of-flight spectrometry

$$\frac{m}{n} = 2eV \times \frac{t_{oF}{}^{2}}{L_{oF}{}^{2}}$$

t_{oF}: 10-1000 ns

L_{oF}: 10-100 cm

Position-Sensitive Detection and back-Projection



compression

$\theta \sim (m+1)\,\theta'$

$$\theta' = a\tan\left(\sqrt{\frac{X^2+Y^2}{L^2}}\right)$$

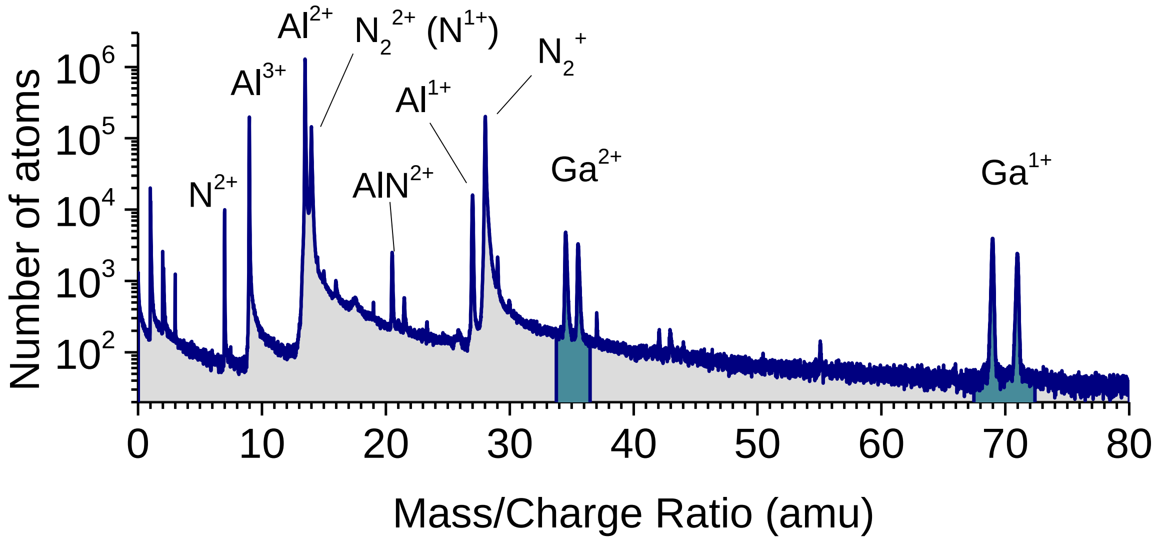
$$\varphi = \varphi'$$

$$x = R\sin(\varphi)\sin(\theta)$$
$$y = R\cos(\varphi)\sin(\theta)$$
$$z = R(1-\cos(\theta))$$

$$\delta z_{atome} = \frac{\Omega}{\xi S_{analyse}}$$

F. Vurpillot et al. Utramicroscopy (2013)
P. Bas et al, Appl. Surf. Sci. (1995)

Atom Probe Tomography: basic information



Chemical identification by time-of-flight

$$\frac{m}{n} = 2eV \times \frac{t_{oF}{}^{2}}{L_{oF}{}^{2}}$$

Charge state ratios: indicators of surface electric field

$N(Ga^{2+})/N(Ga^{+})$

3D reconstruction

Position on the detector → Position on the tip (different back-projection algorithms)

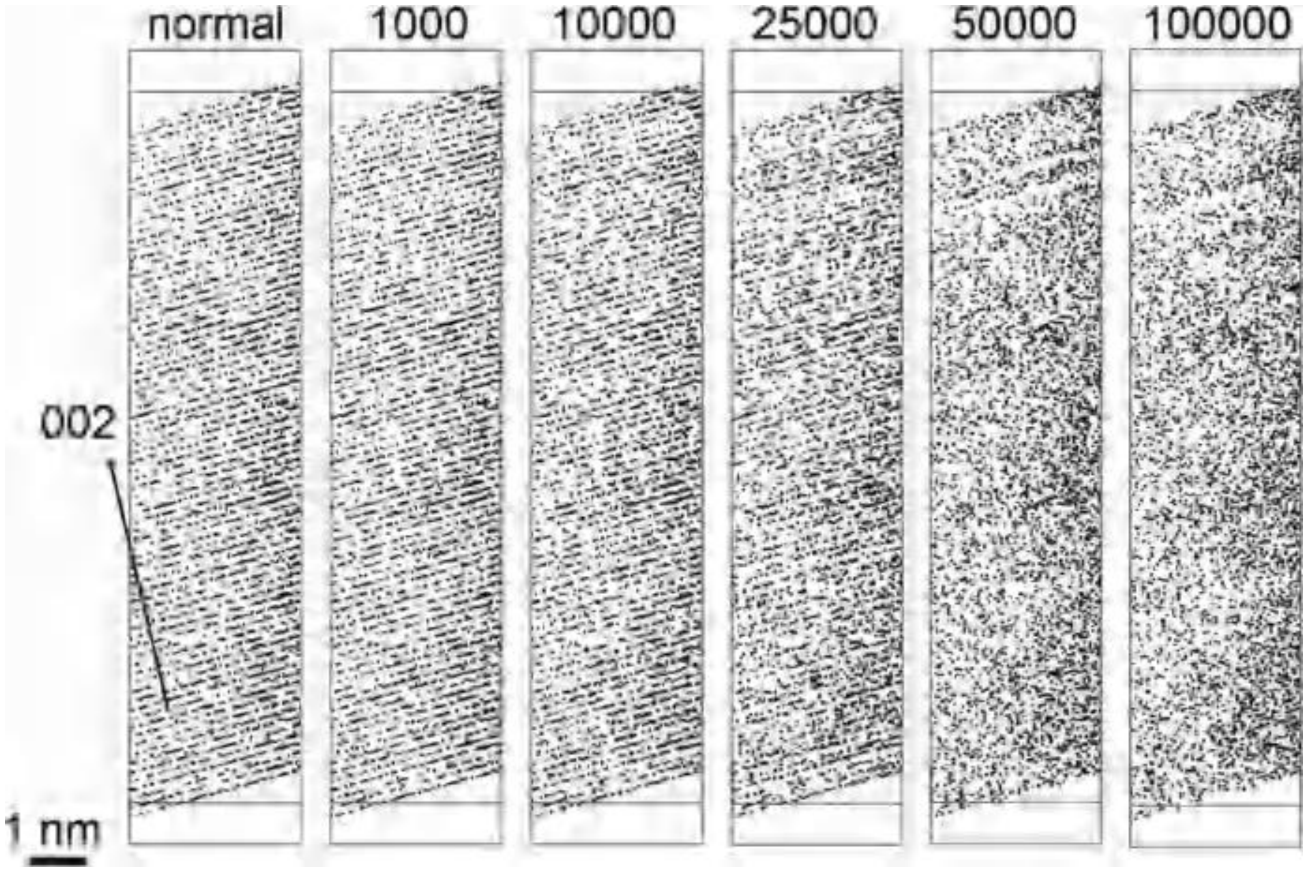
Ga atoms

Interfaces, impurity density, clustering, alloy distribution…

D. Blavette et al. Nature (1993)
D. R. Kingham Surf Sci. (1982)
D. Blavette et al. RSI (1993)
B. Gault et al. RSI (2006)
F. Vurpillot et al. Utramicroscopy (2013)

Spatial resolution of Atom Probe Tomography – In depth

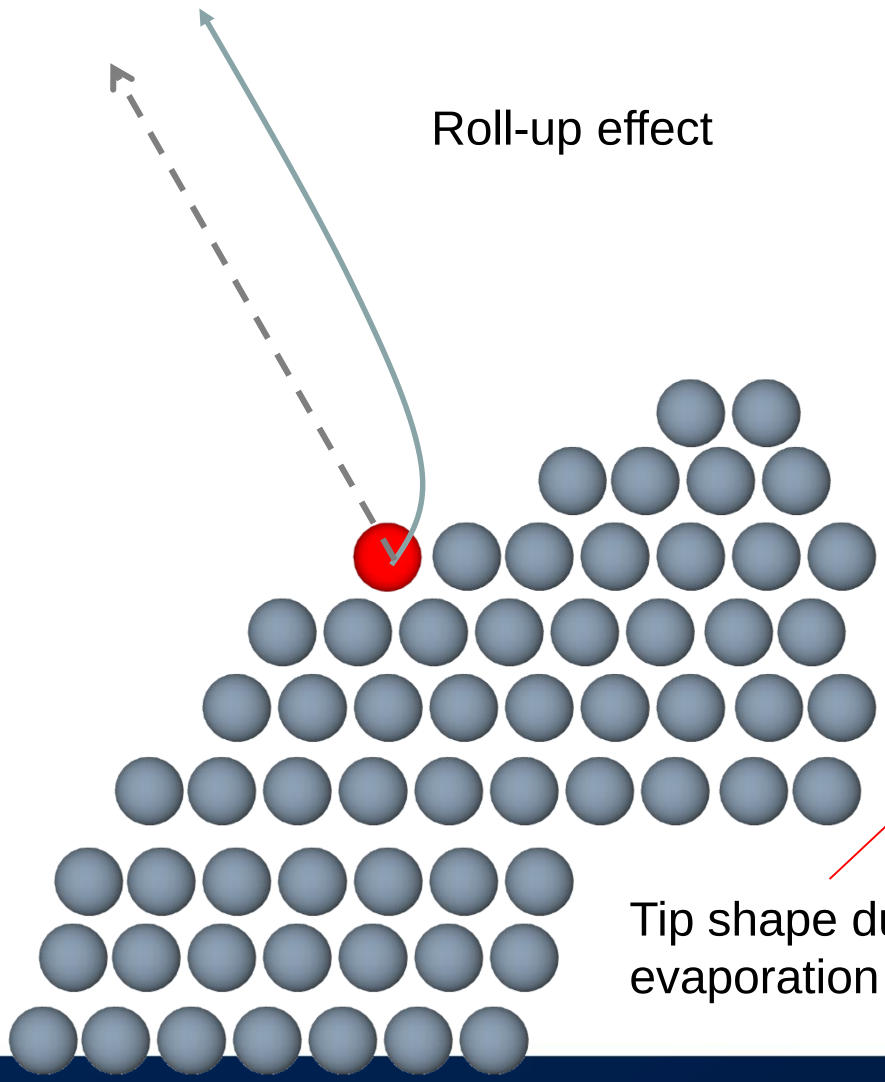
Is it possible to resolve the crystal structure?

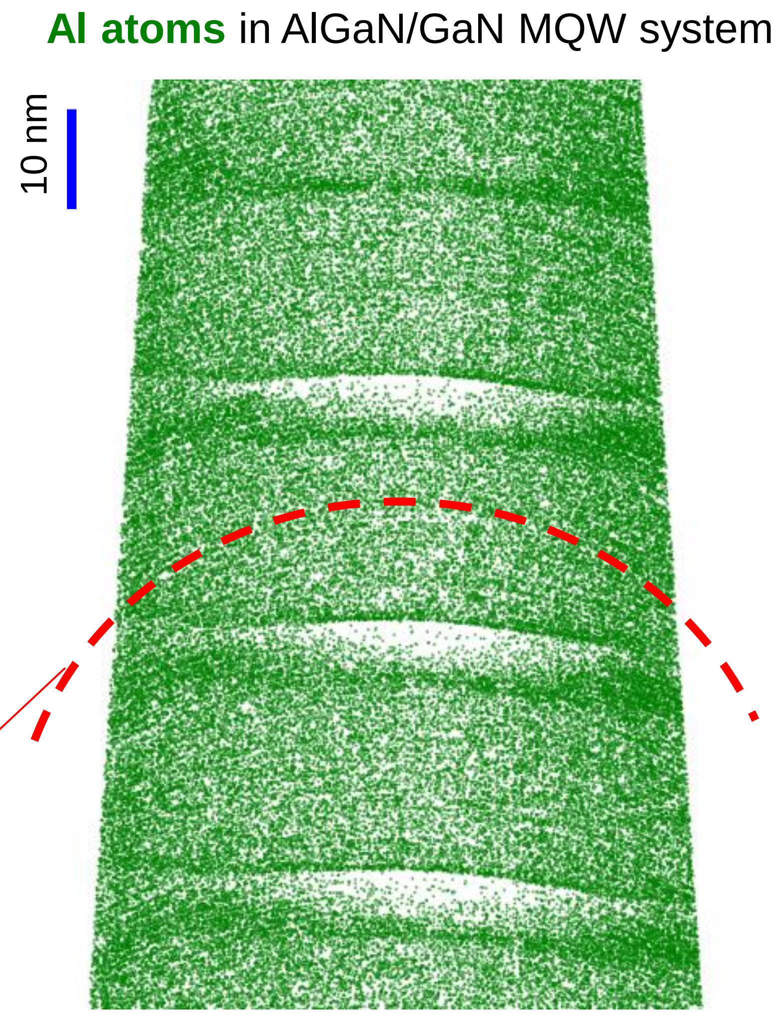


Aluminum - True sequence → Randomized sequence

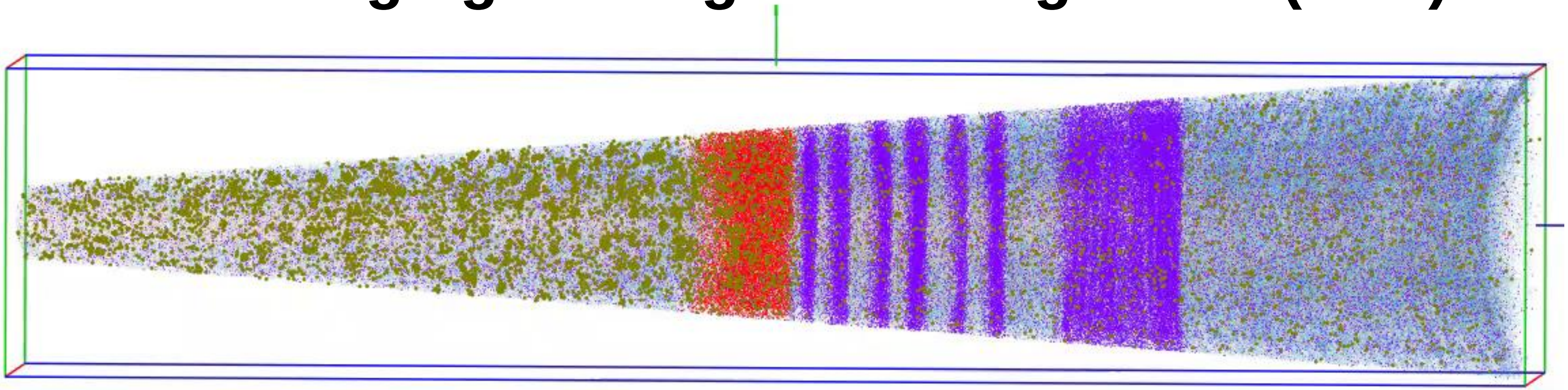
Gault, Moody, Cairney, Ringer, Atom Probe Microscopy (Springer)

Spatial resolution of Atom Probe Tomography - Lateral





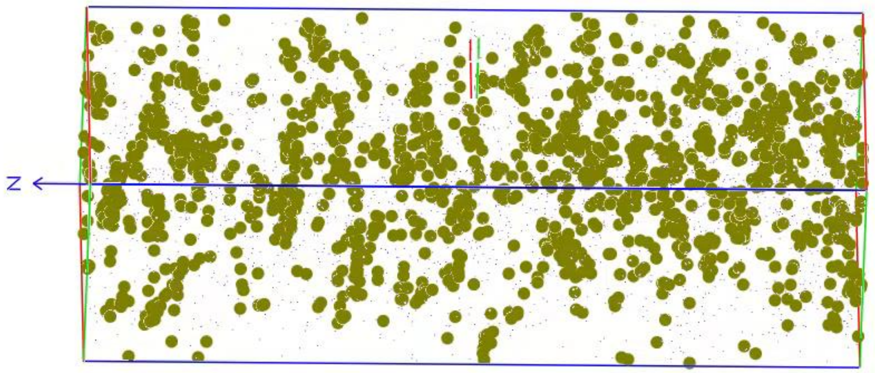
APT imaging of a Light-Emitting Diode (LED)



19.9 nm

Mg Al In Ga N

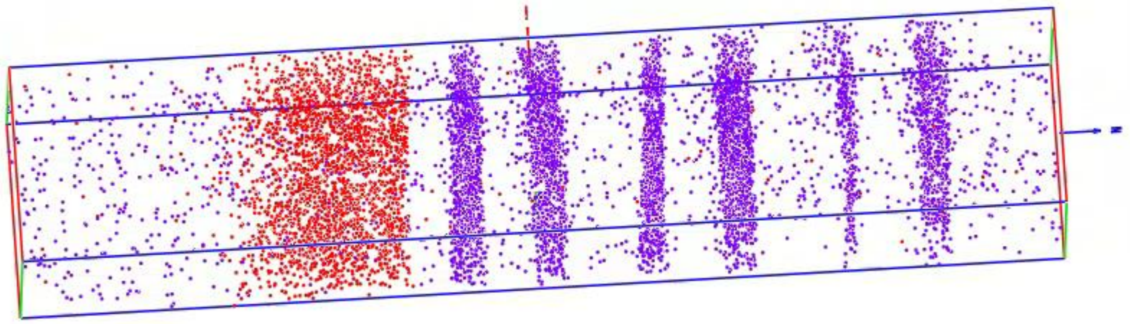
Dopage p



Mg

5.3 nm

Région active (EBL, QWs)



Al In

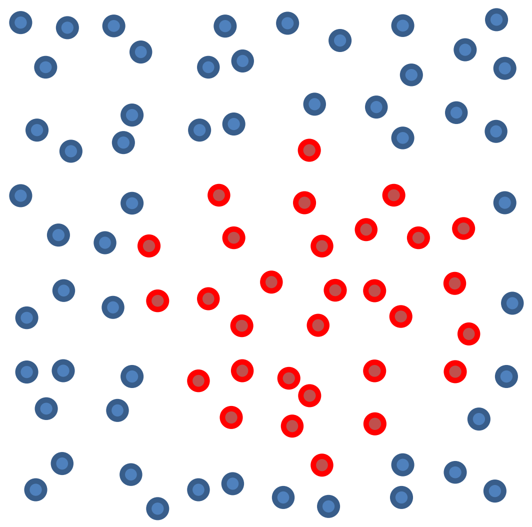
8.5 nm

Spatially resolved composition

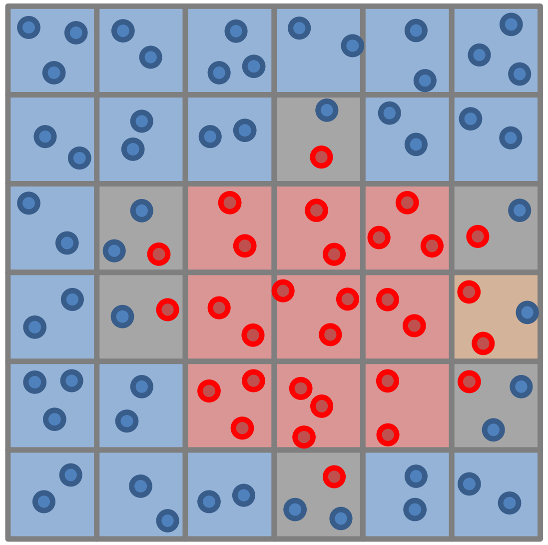
Example:

Binary compound AB: ● A atoms ● B atoms

APT data



APT reconstructed space mesh (3D)



Atomic fraction $X_i = \frac{n_i}{\sum_j n_j}$

Site alloys:

Site fraction $A_y B_{(1-y)} C$ $y = \frac{n_A}{n_A + n_B}$

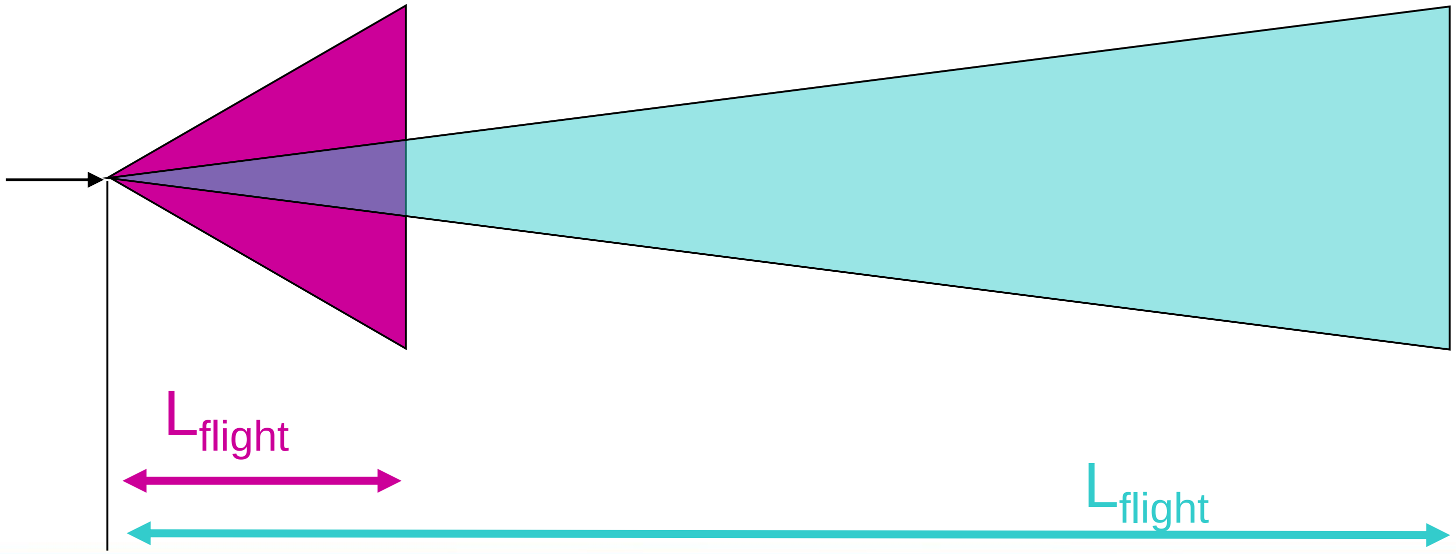
Optics and mass spectrometry

Compromise angular field of view / mass resolution

$$\frac{m}{n} = 2eV \times \frac{t_{vol}^{2}}{L_{vol}^{2}} \quad \Rightarrow \quad \frac{\Delta m}{m} \approx \frac{\Delta V}{V} + 2\frac{\Delta t_{vol}}{t_{vol}} + 2\frac{\Delta L_{vol}}{L_{vol}}$$

Large FOV
Large Δt/t

Small FOV
Small Δt/t

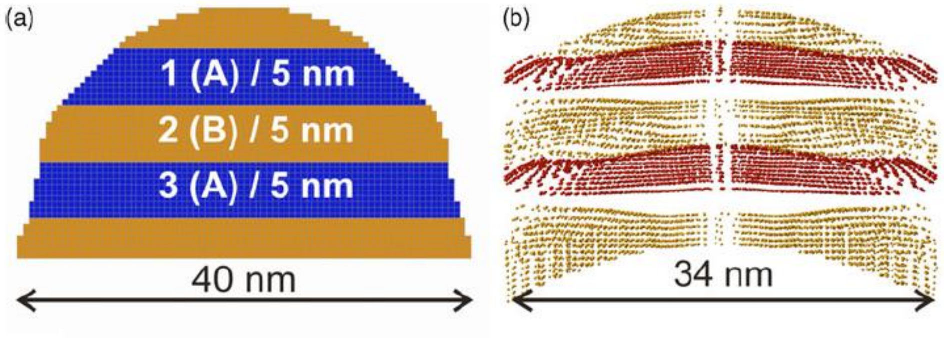


Atom Probe Tomography: metrology issues

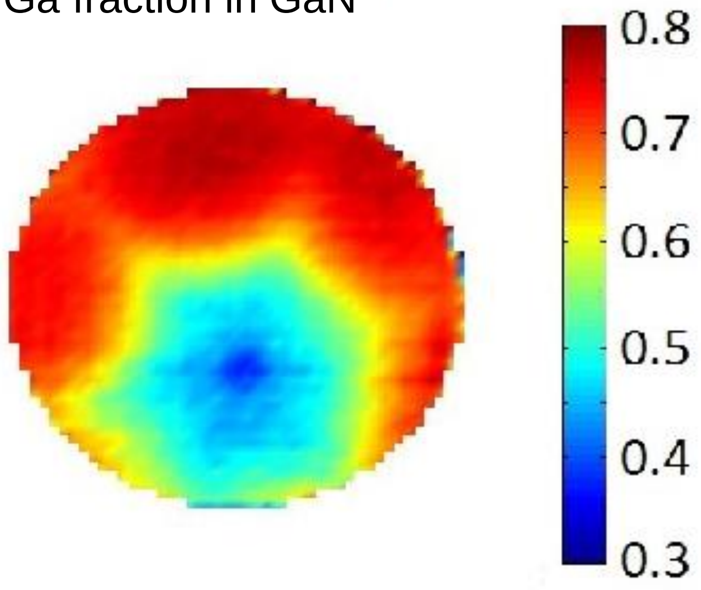
Reconstruction issues:
- The sample *is* part of the ion optics. Inhomogeneous samples induce trajectory aberrations.

Detection efficiency issues:
- Intrinsic detector performance (60-80%)
- Specific losses (neutral production, preferential evaporation)

A – low field phase
B – high field phase



Ga fraction in GaN



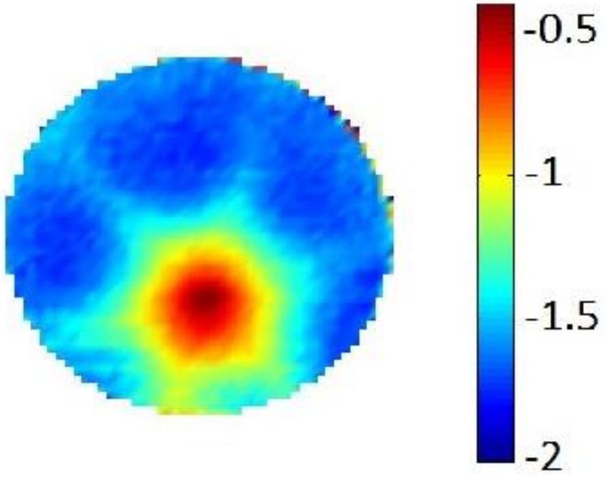
→ Distorted reconstructions
Degradation of spatial resolution, mixing of separate phases

→ Compositional inaccuracies

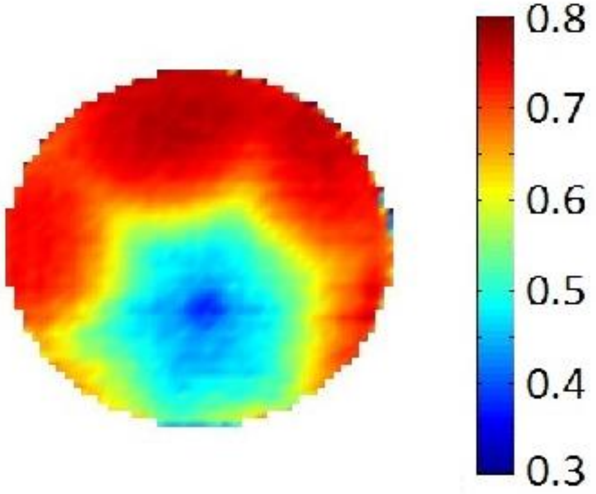
F. Vurpillot et al. Utramicroscopy (2013)
Oberdorfer and Schmitz, Microsc. Microanal. (2011)
L. Mancini et al. J. Phys. Chem. C (2014)
E. Di Russo et al. Microsc. Microanal. (2017)

Compositional biases in GaN

Log ($[Ga^{2+}]/[Ga^{+}]$)
~ surface electric field



Ga atomic fraction



- Loss of detection efficiency
- Non-uniform composition meausured on the tip surface
- Significant problem for the correct assessment of **doping concentration and alloy fractions**

L. Mancini et al. J. Phys. Chem C (2014)
J. Riley et al. ACS Nano (2012)
Agarwal et al. J. Phys. Chem. C (2011)
Karakha et al. Appl. Phys. Lett. (2015)
Xia et al. J. Appl. Phys (2015)
B. Gault et al. New J. Phys. (2016)

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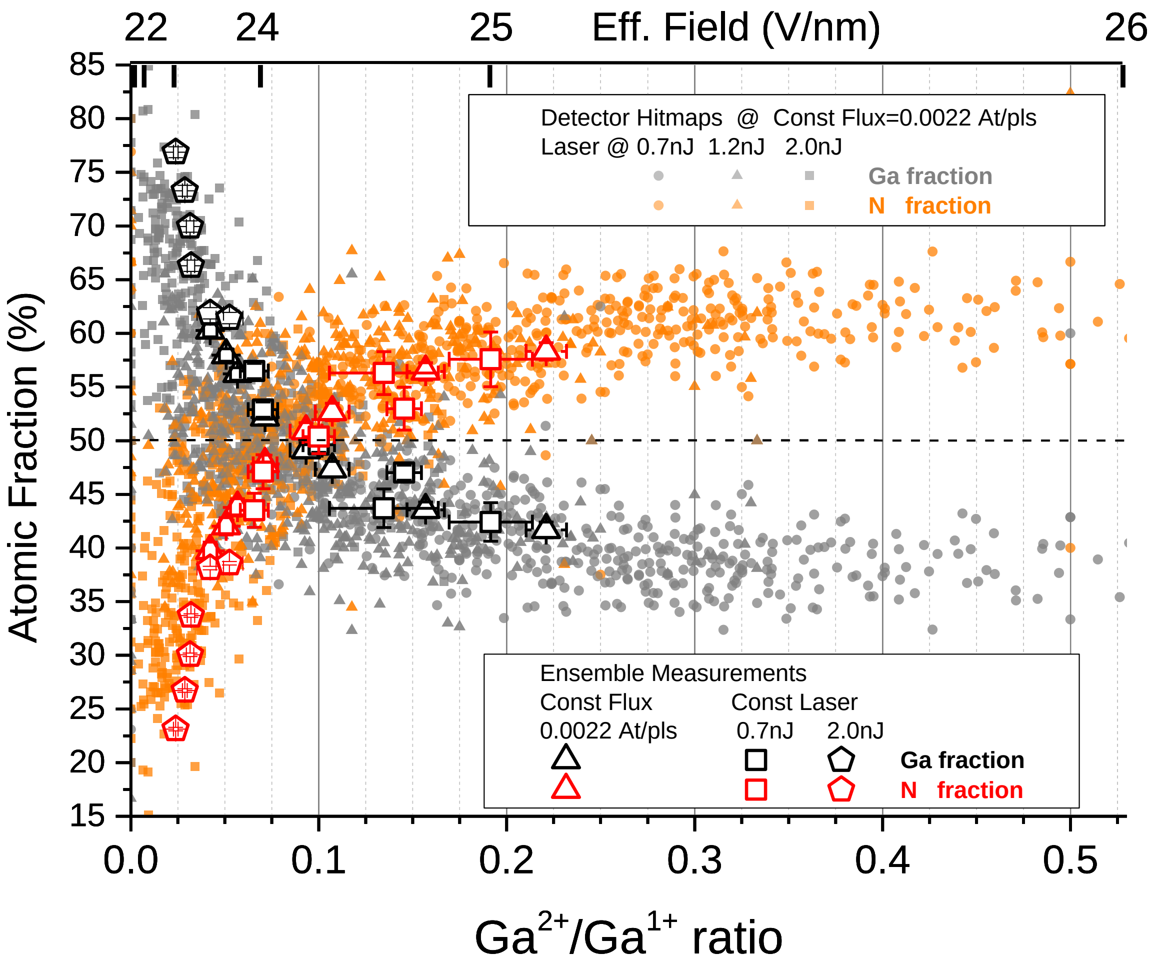
Composition vs Field

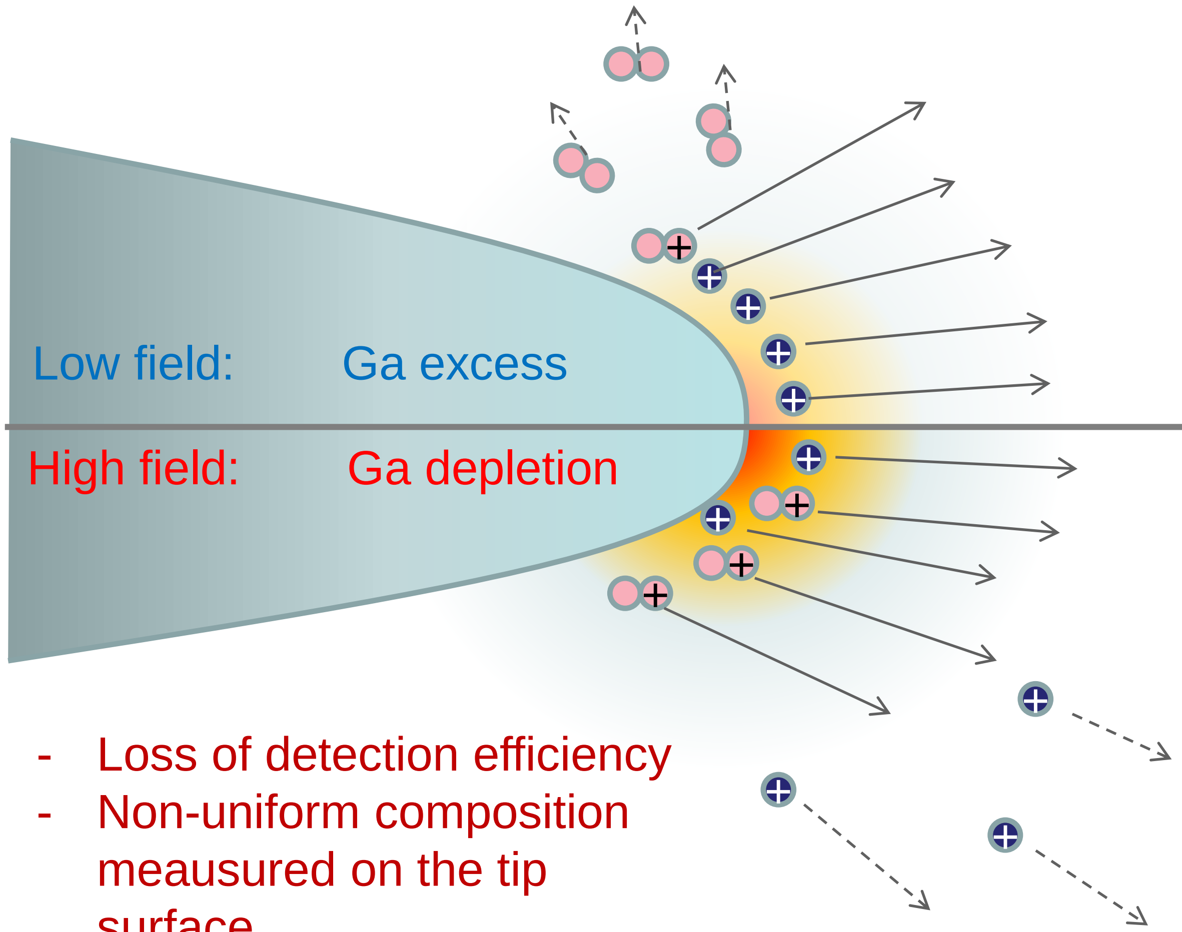
Low field

High field



L. Mancini et al. J. Phys. Chem C (2014)

Proposed Mechanism (GaN)



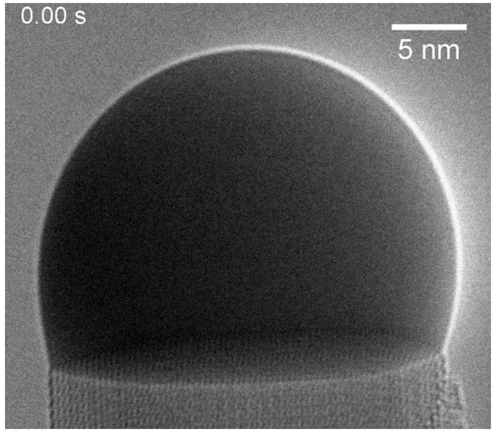
- N forms molecular N_2 on the surface evaporating as a neutral species.
- Low post-ionization of N_2 molecules: neutral N_2 is undetected
- Ga evaporates as Ga+ and is detected

- High post-ionization of N_2 molecules, increase of N detection efficiency
- Continuous evaporation of Ga^+ and Ga^{2+}, uncorrelated with laser pulse and undetected

- Loss of detection efficiency
- Non-uniform composition meausured on the tip surface

L. Mancini et al. J. Phys. Chem C (2014)
J. Riley et al. ACS Nano (2012)
Agarwal et al. J. Phys. Chem. C (2011)

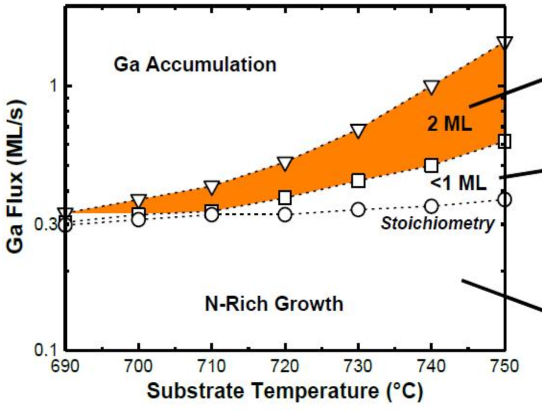
Surface Physics and Chemistry: Epitaxy vs field evaporation

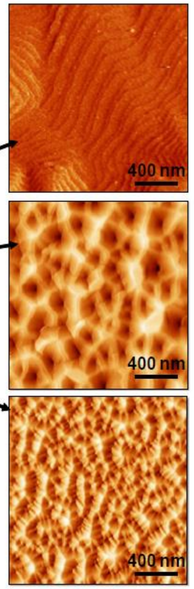


J.-C. Harmand, et al. Physical Review Letters, 121, 166101 (2018)

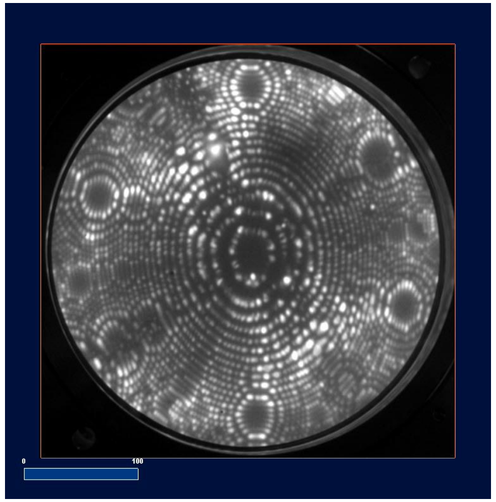
Epitaxy

- Ordered addition
- Flat surfaces (except 3D growth)
- Neutral species
- No field





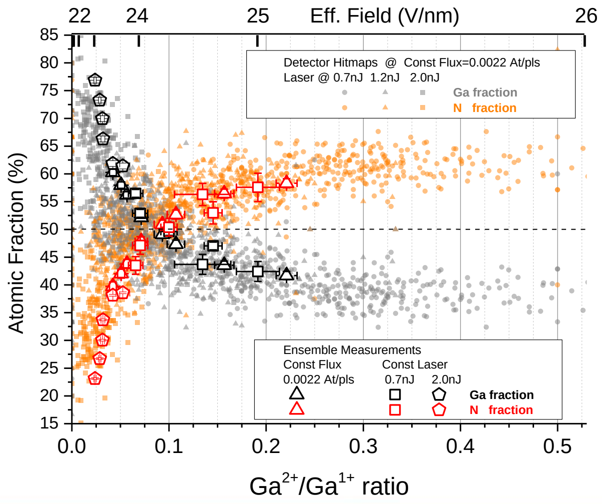
Adelmann, H. C., PhD Thesis, 2002



Field ion evaporation

- Ordered subtraction (reverse time scale)
- Curved surfaces
- Neutral to ionized species
- High field

"Apotaxy" ???



FIM of evaporating steel tip (F. Danoix)

L. Mancini et al. J. Phys. Chem C (2014)

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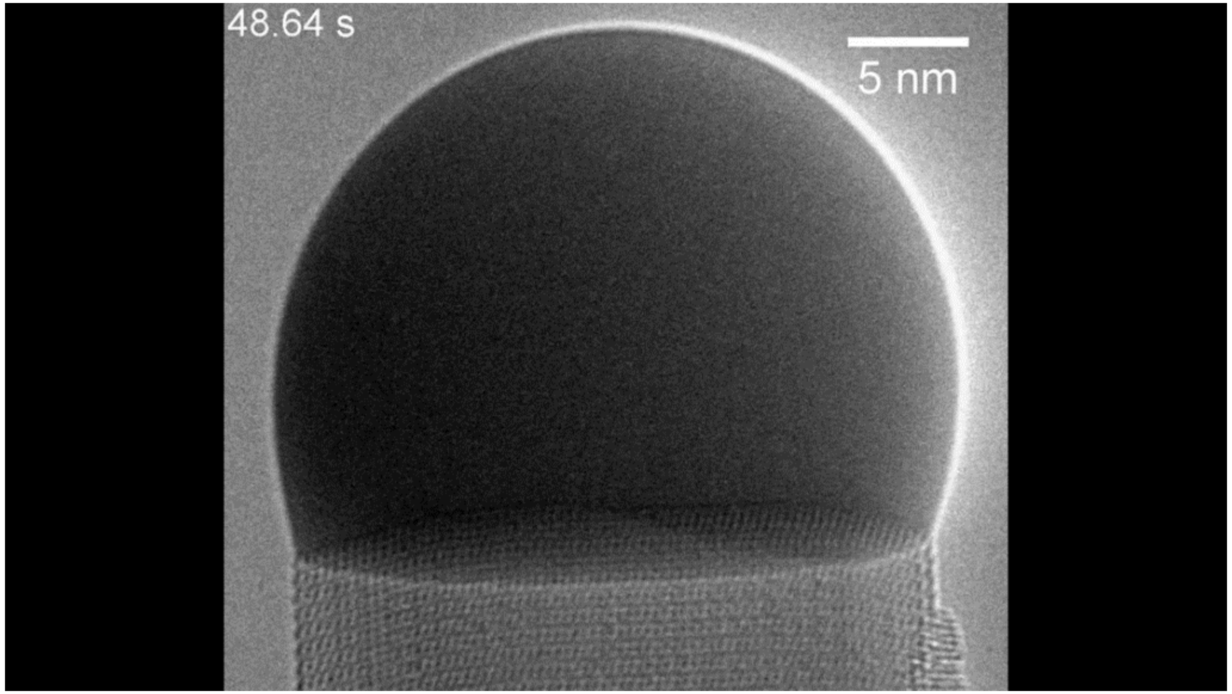
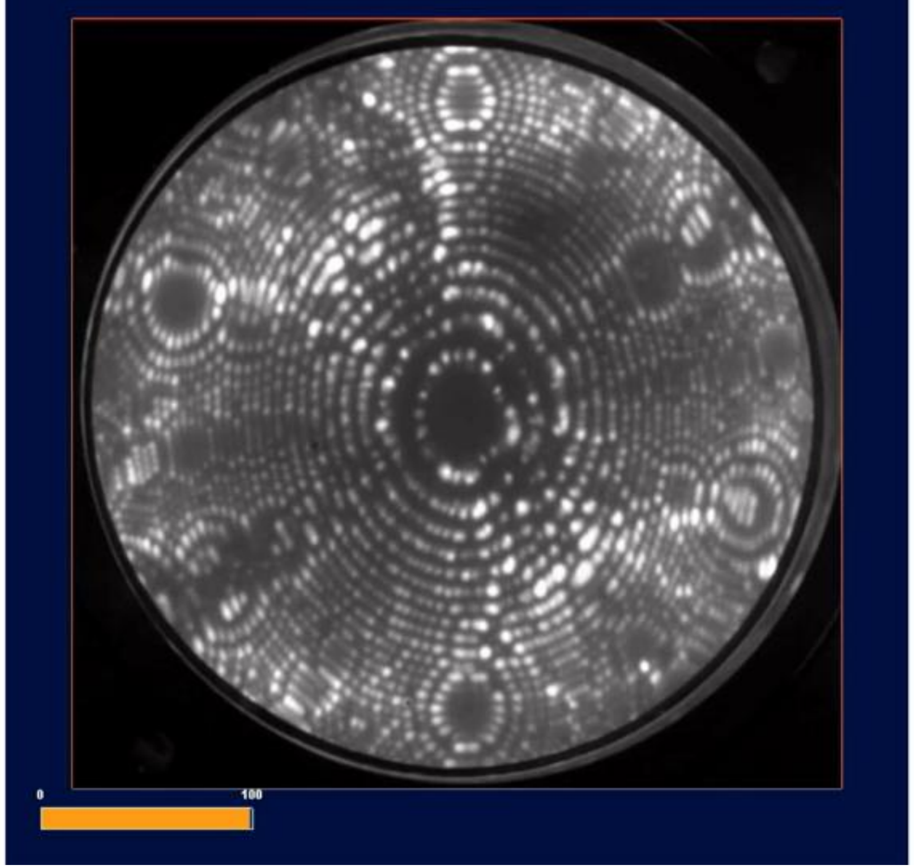
Indeed you just reverse the time…

J.-C. Harmand, et al. Physical Review Letters, 121, 166101 (2018)

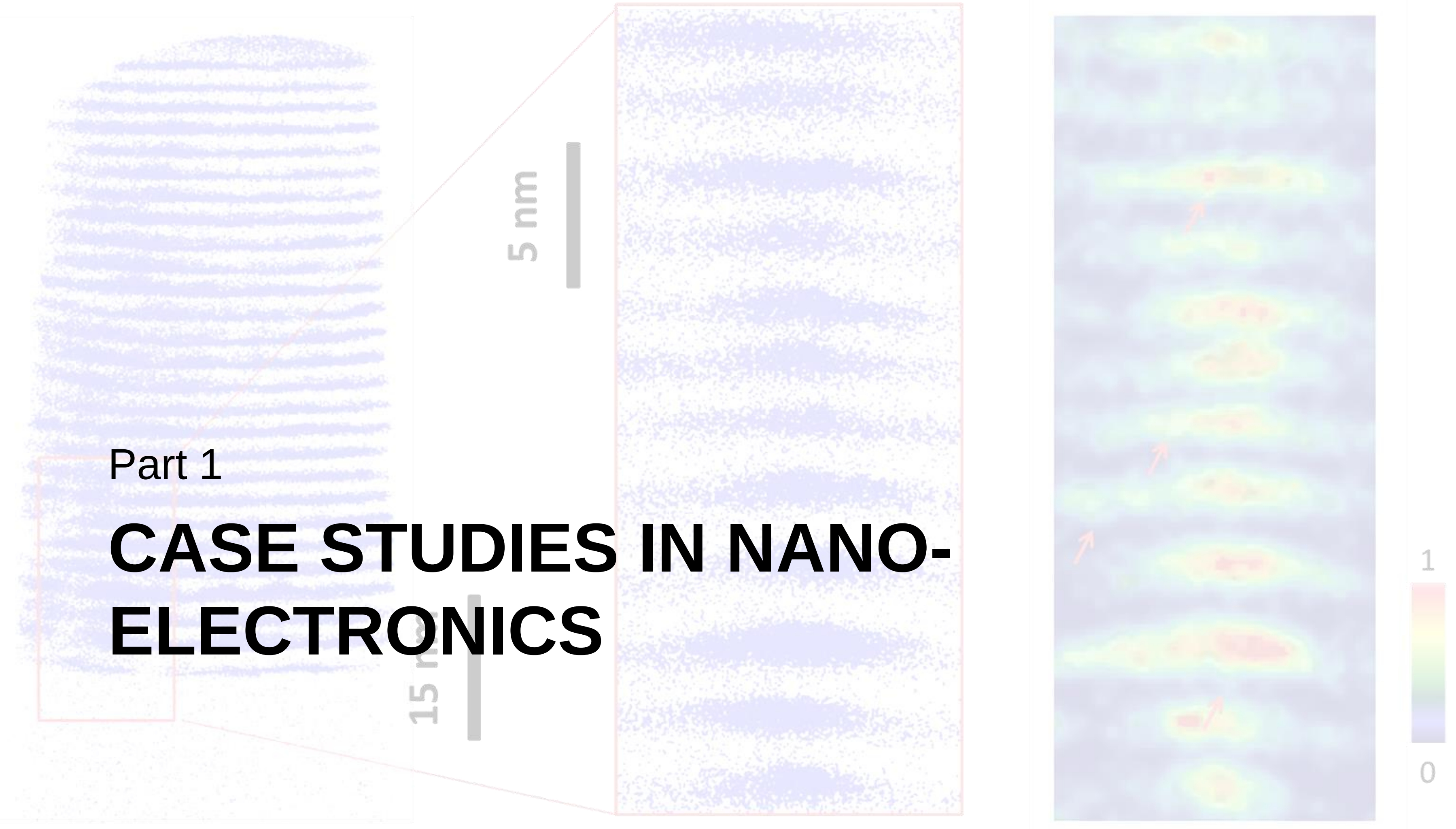


The nanowire evaporates

FIM of evaporating steel tip (F. Danoix)



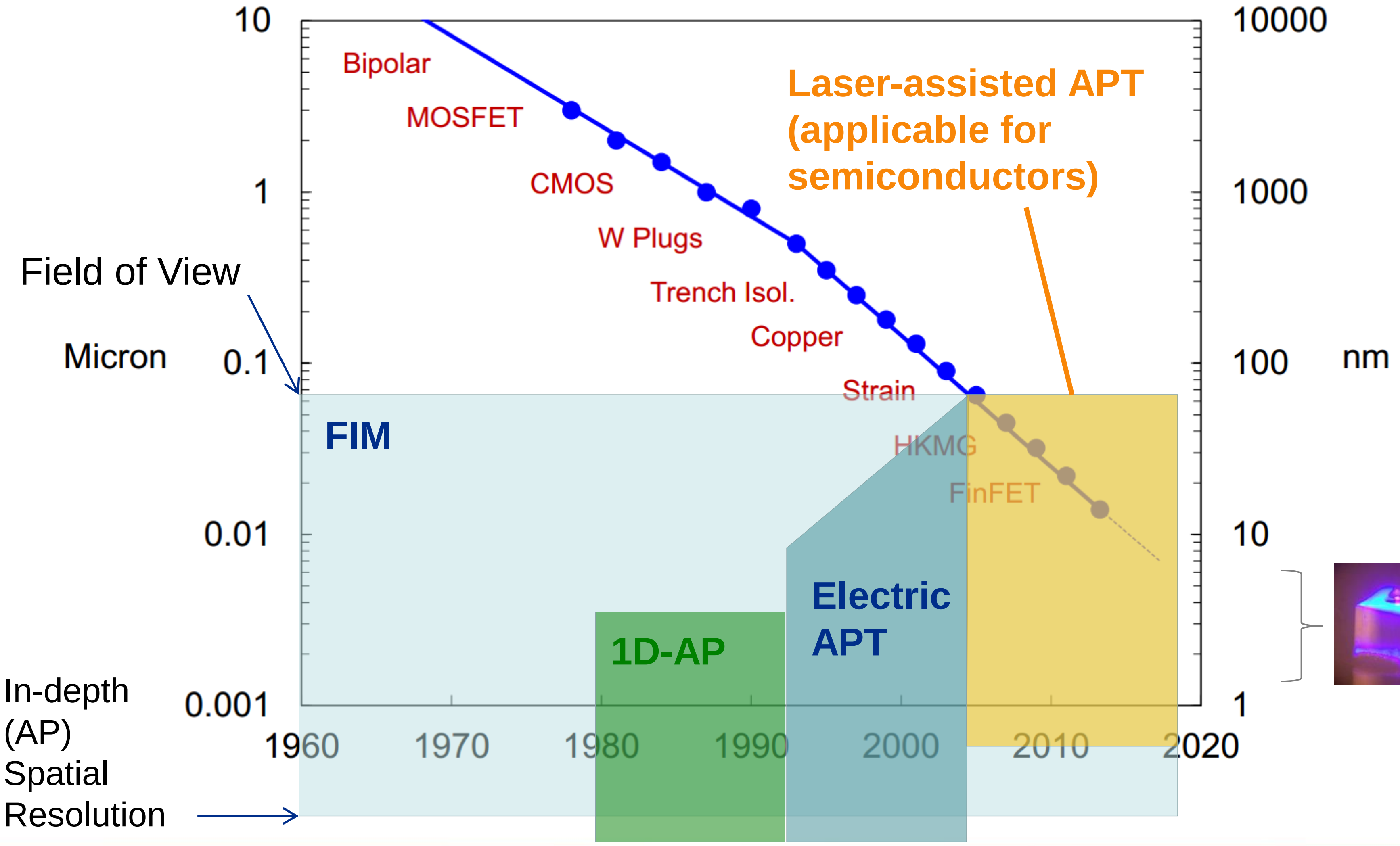
The steel tip grows

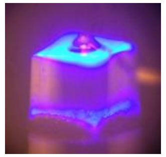


Part 1

CASE STUDIES IN NANO-ELECTRONICS

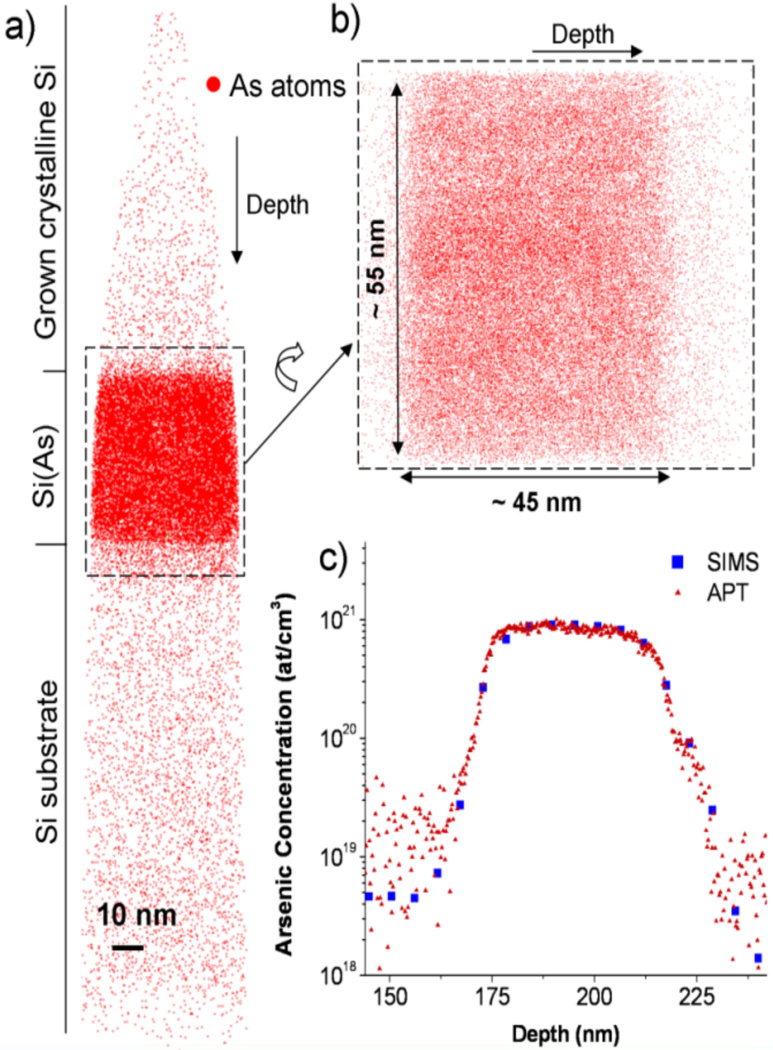
APT in electronics – a matter of scaling and timing

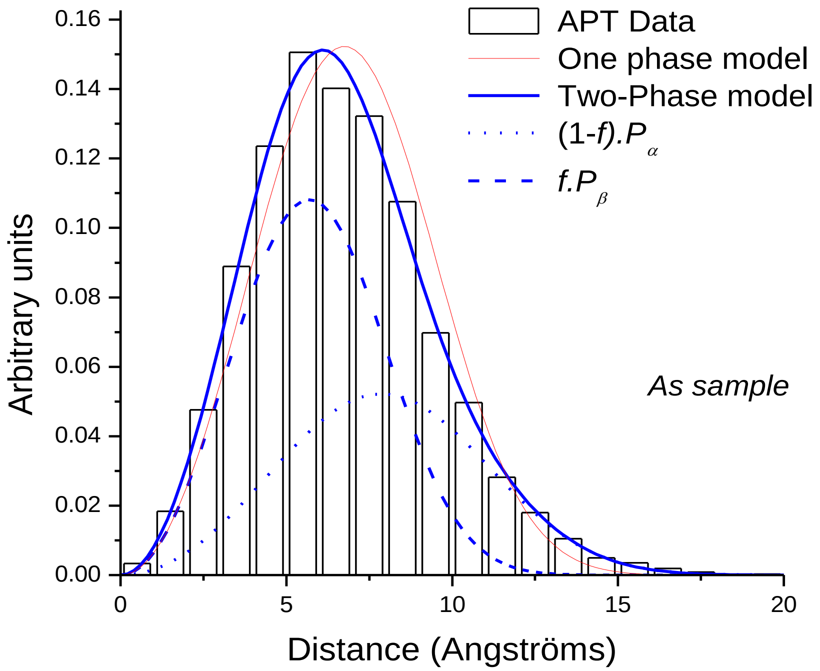




Device Scale Evolution Graph, Source: Intel

Studies of doping by APT

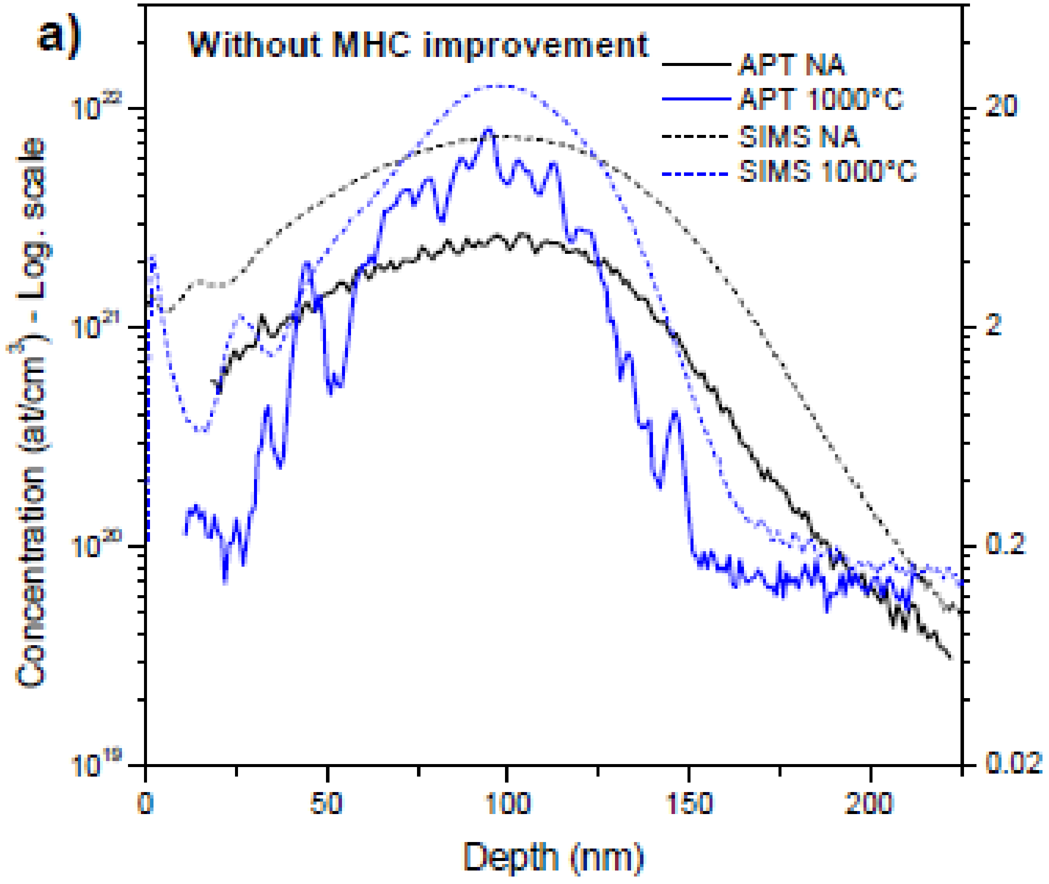




- Distribution of As atoms in Si seems homogeneous but high electrical deactivation (80%)
- However, the distance between first neighbor distribution indicates an inhomogeneous distribution
- Evidence of the presence of a short range ordering accordingly to literature

S. Duguay, et al, J. of Appl. Phys, 2009

Case study : Boron in Si



Large discrepancy between APT and SIMS data

Explanation: B undergoes preferential retention: Si evaporates until B is in high surface concentration; It then evaporate as a multi-ion burst and many ions are not detected

Improvement of detection system needed

Dubois C., Prudon G., Gautier B., Dupuy J.C., "Quantitative SIMS measurement of high concentration of boron in silicon (up to 20 at.%) using an isotopic comparative method", Applied Surface Science 255, 1377–1380, (2008).

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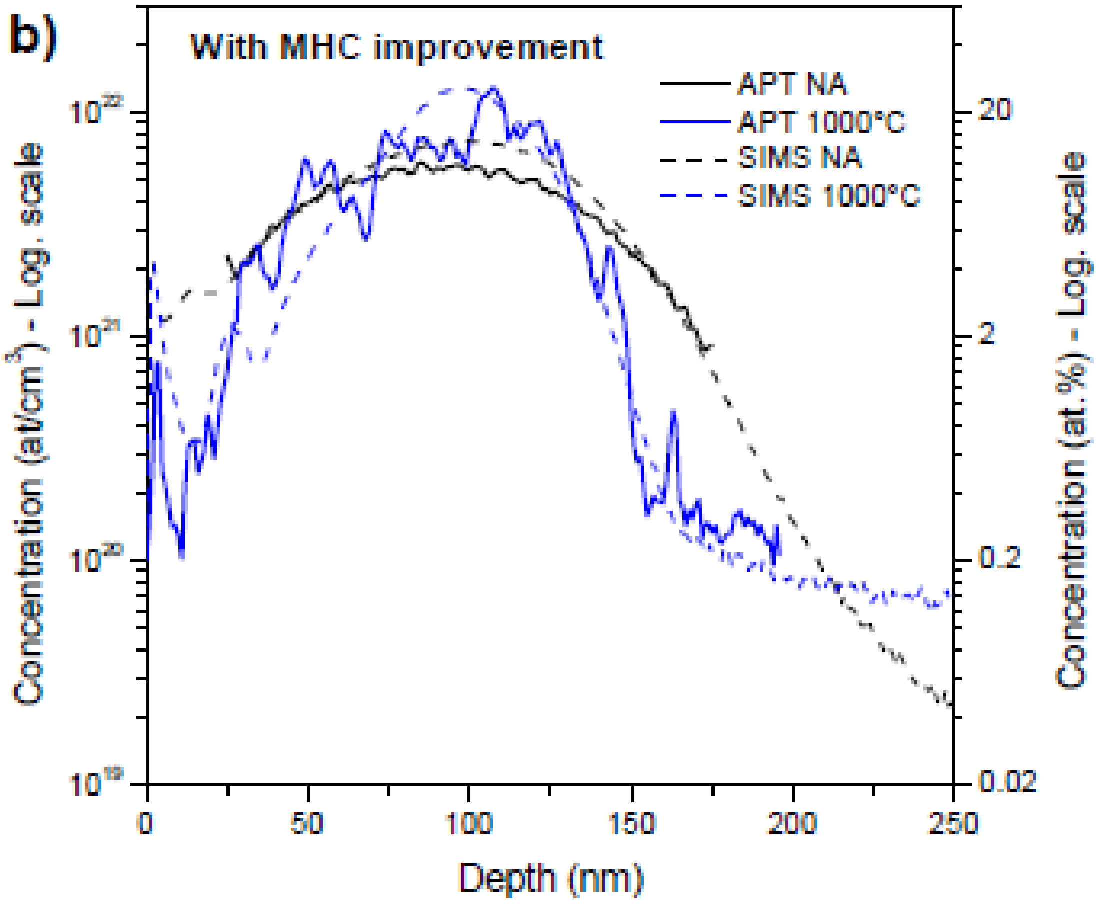
Case study : Boron in Si

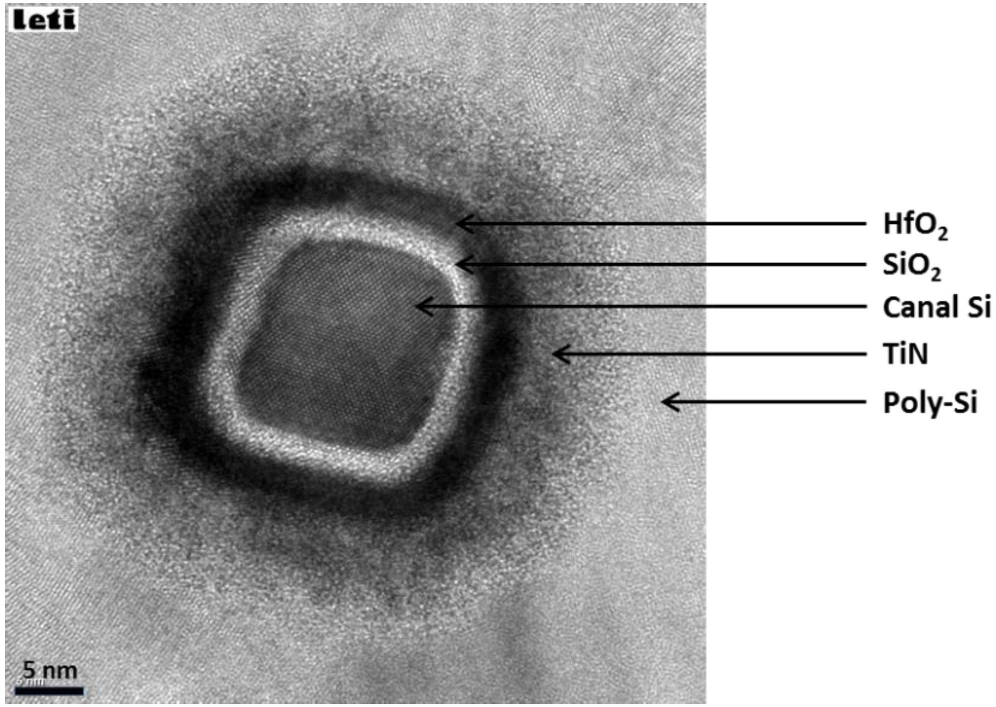


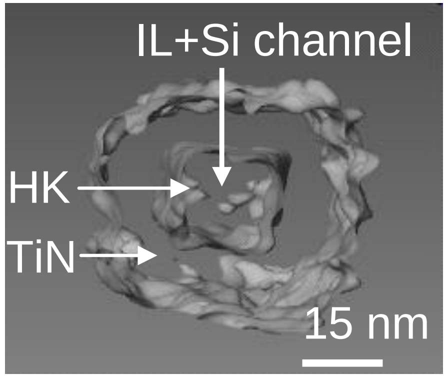
Development of an advanced delay line detector with pulse deconvolution and capability to discriminate up to 30 impacts per pulse

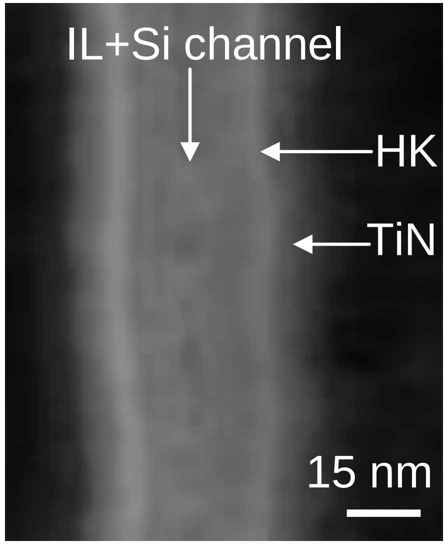
APT and SIMS are now fairly consistent.

G. Da Costa, H. Wang, S. Duguay et al., *Review of Scientific Instruments* 83, (Dec, 2012).

Case study: field effect transistors





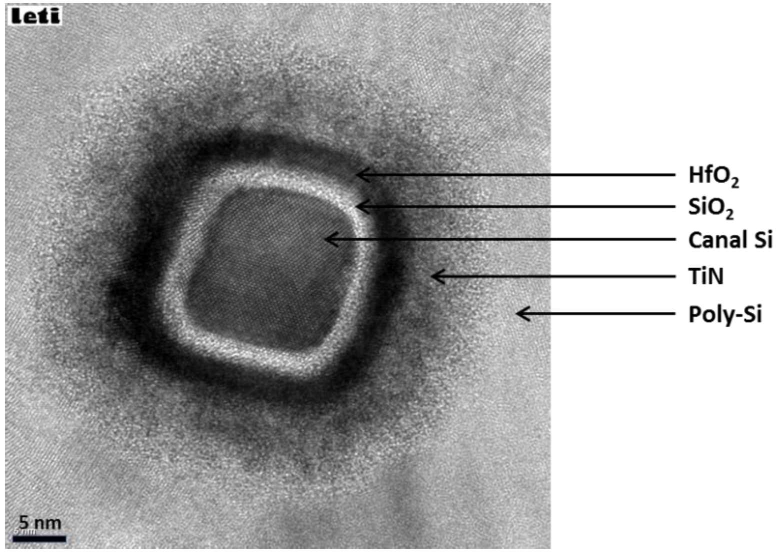


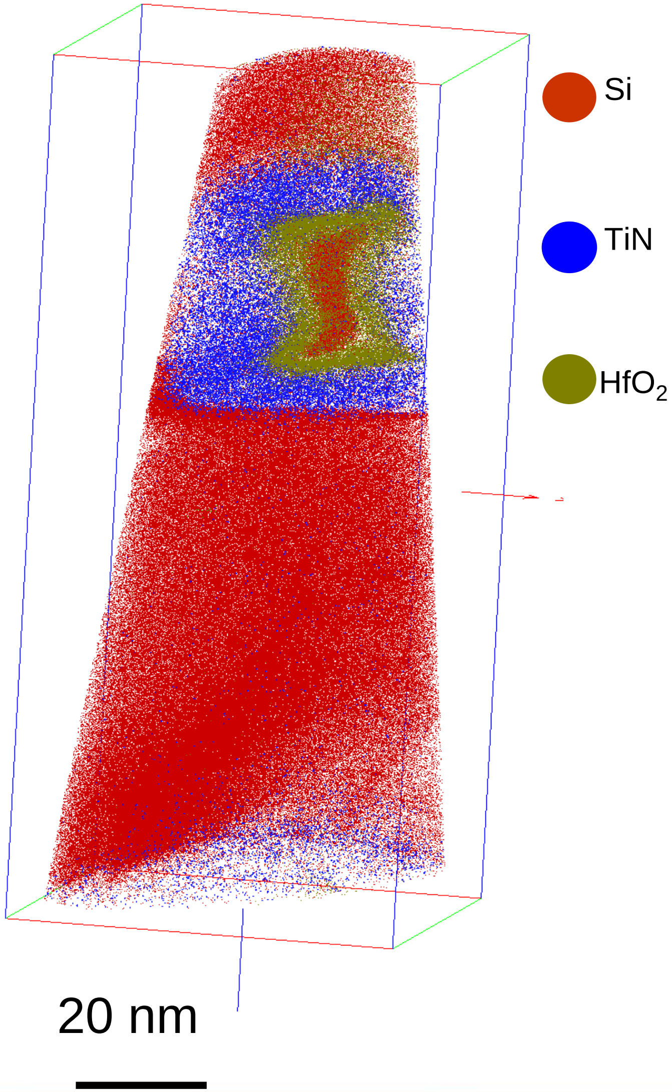
- Gate-all-around transistors: promising candidates for future CMOS devices because of low off-state leakage current and reduced short-channel effects
- High performances for sub-22 nm technologies

Collaboration GPM/STMicrolectronics / CEA-LETI – Courtesy of Sébastien Duguay

Case study: field effect transistors

APT – reconstruction biases





- Reconstruction with the standard model (cone angle) gives a wrong representation of the device.

- Can such a shape be reproduced by simulations?

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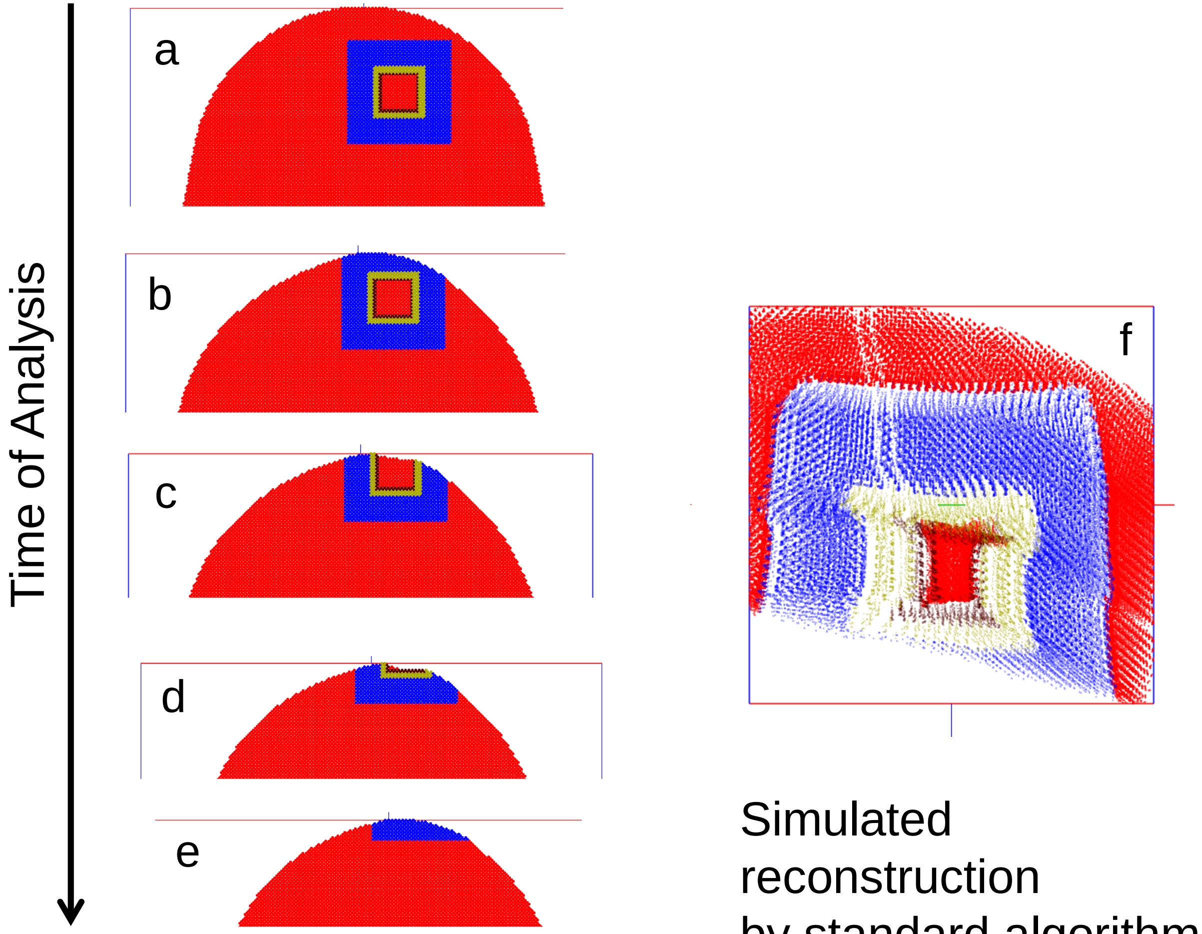
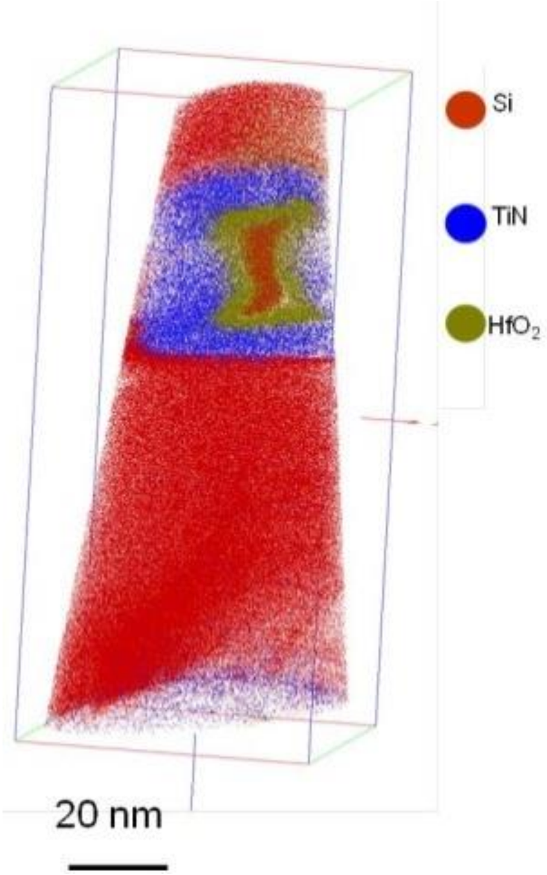
Case study: field effect transistors

APT simulation – explanation of reconstruction biases



Simulated
reconstruction
by standard algorithm



Reconstruction
of experimental data
by standard algorithm

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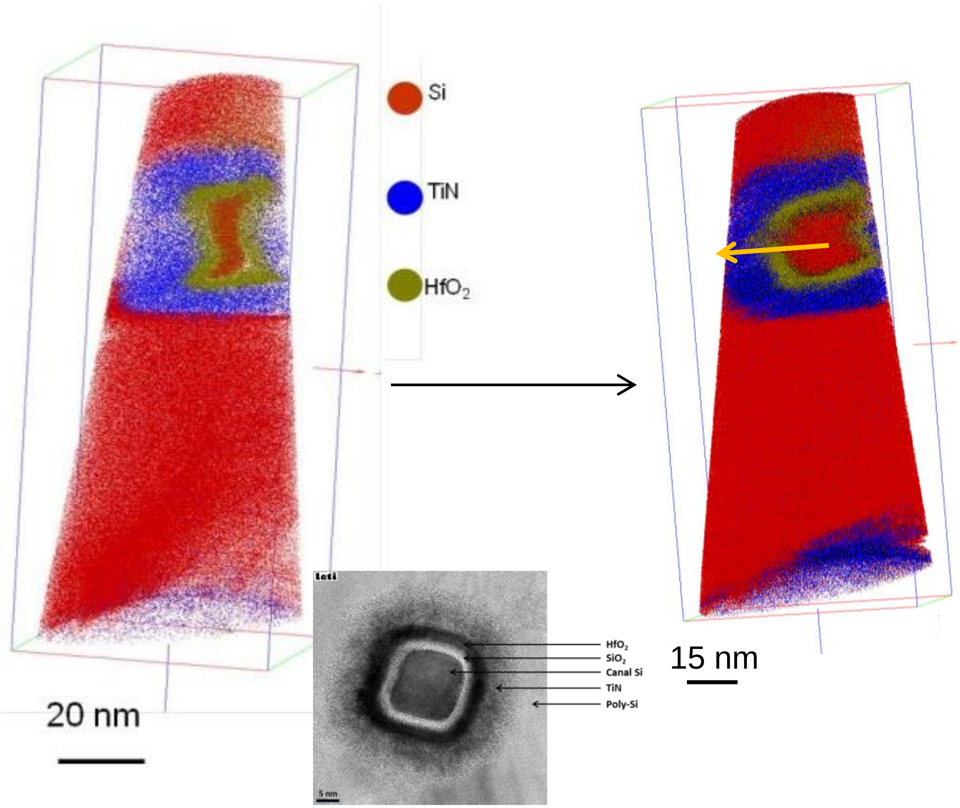
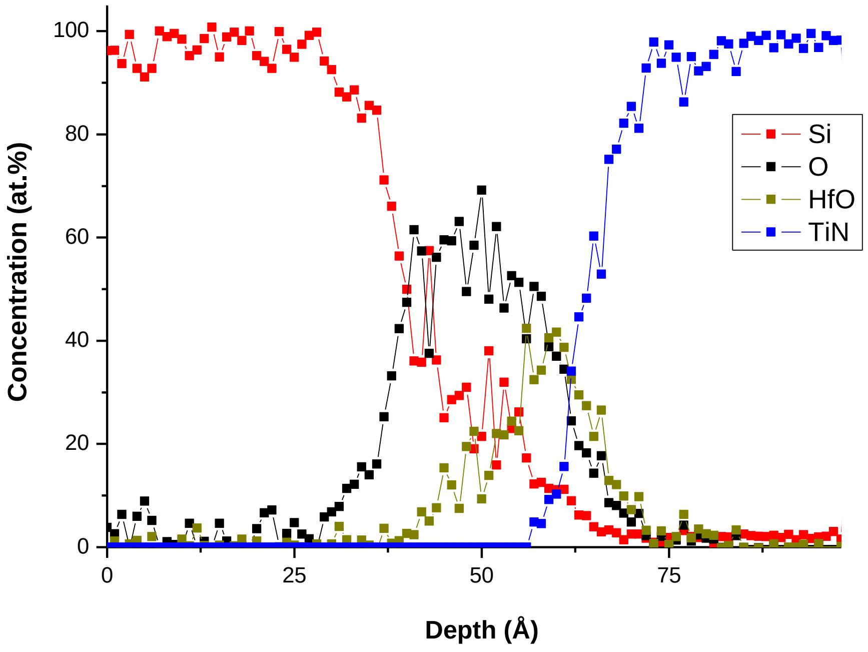
Case study: field effect transistors

Correction of reconstruction biases



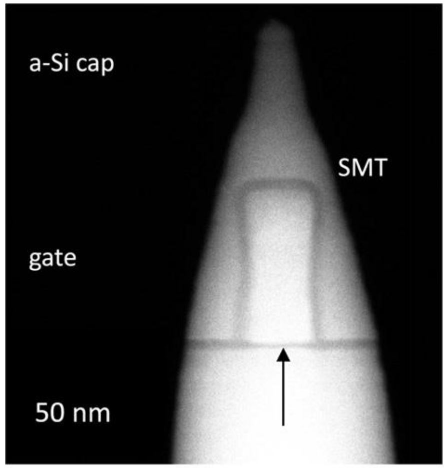


• Advanced reconstruction based on density correction permits to get a better image of the analyzed tip but there is a large room for improvement.

Collaboration GPM/STMicrolectronics / CEA-LETI – Courtesy of Sébastien Duguay

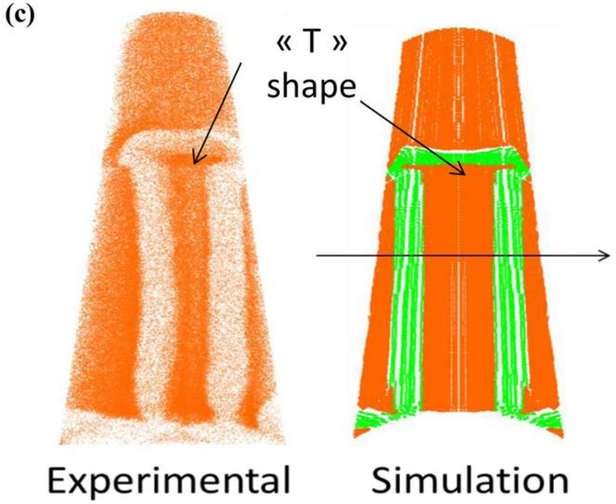
A. Grenier et al. Ultramicroscopy (2014) A. Grenier et al. APL (2015)

Towards a new paradigm: Correlated Tomographies



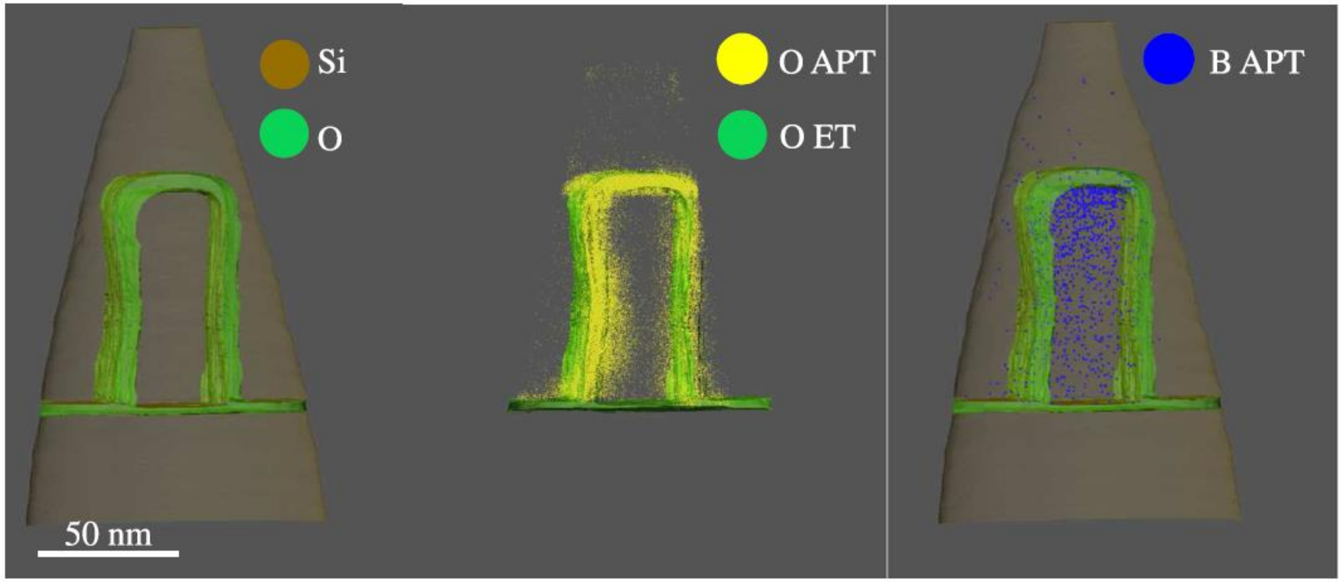
Field-Evaporation Simulation

- Explanation of origin and features of artifacts
- Input for APT and artifact correction



Electron Tomography

- 3D morphologic information
- Benchmark for APT reconstruction and artifact correction



Collaboration GPM/STMicrolectronics / CEA-LETI – Courtesy of Sébastien Duguay

A. Grenier et al. Ultramicroscopy (2014) A. Grenier et al. APL (2015)

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Part 1

CORRELATION BETWEEN EMISSION SPECTROSCOPY AND ATOM PROBE TOMOGRAPHY

APPLICATION TO SEMICONDUCTOR HETEROSTRUCTURES

Radiative phenomena in semiconductors

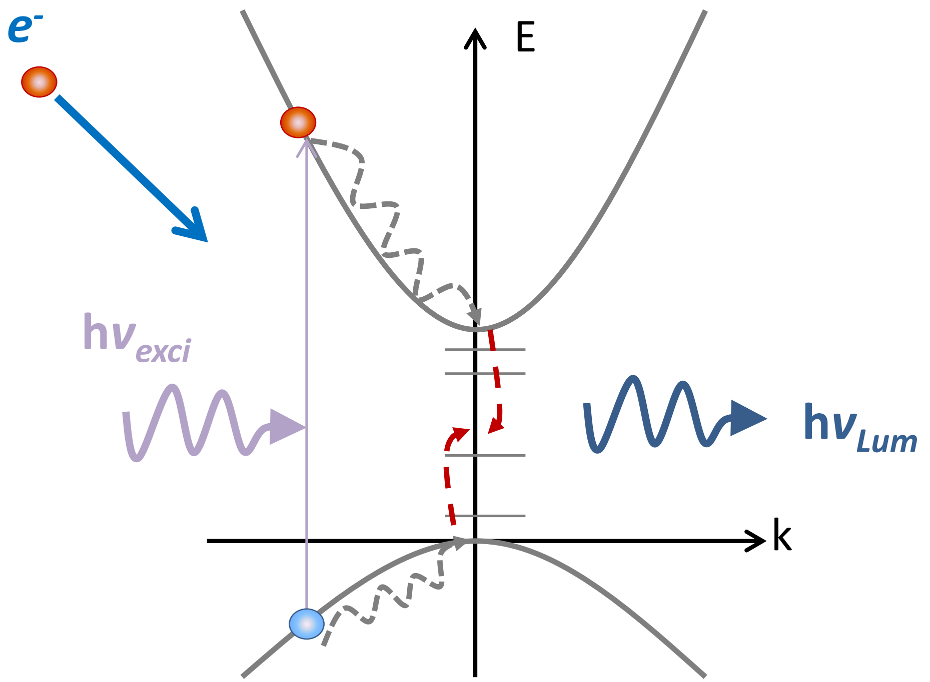
Physical phenomena giving rise to the emission of photons: electron-hole recombination in semiconductors (not exhaustive)

In semiconductors the optical transition properties depend on chemical and structural factors

- **Constituent atoms**
- **Crystal symmetry**
- Impurity atoms
- Structural defects
- Presence of surfaces, surface states

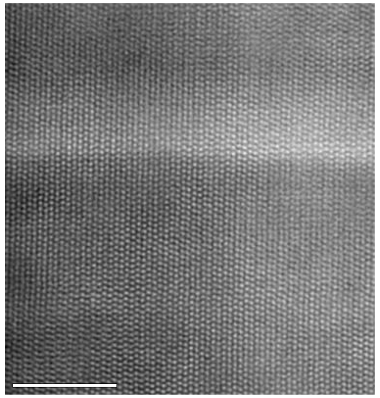
As well as on environmental parameters

- Temperature
- **Stress / Strain**
- External fields (Electric, Magnetic)
- ...

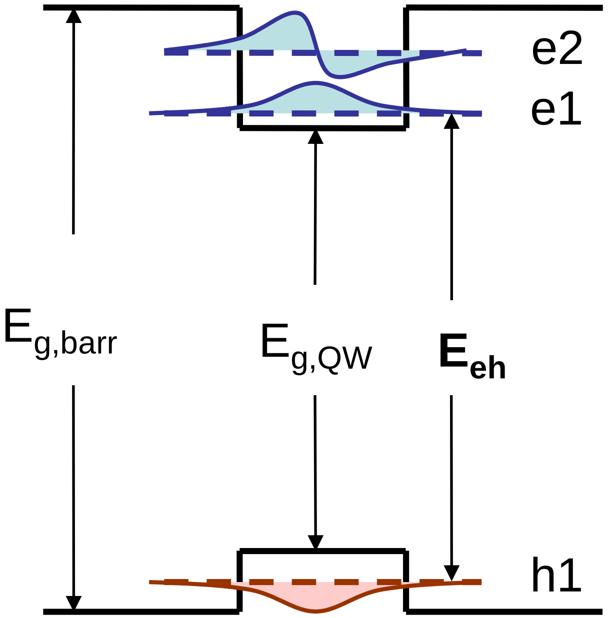


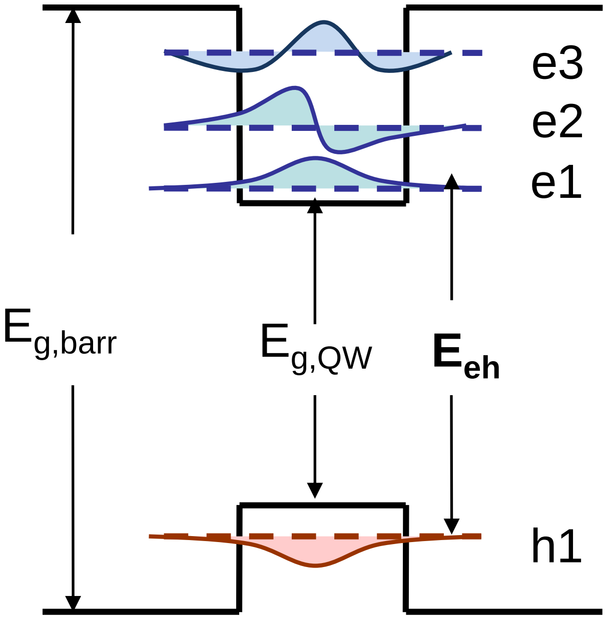
Excitation of radiative transitions: photons, electrons, electrical injection…

Band engineering: quantum-confined systems



Band engineering: optical transition energy may be tuned by changing the alloy composition of one/more of the layers …

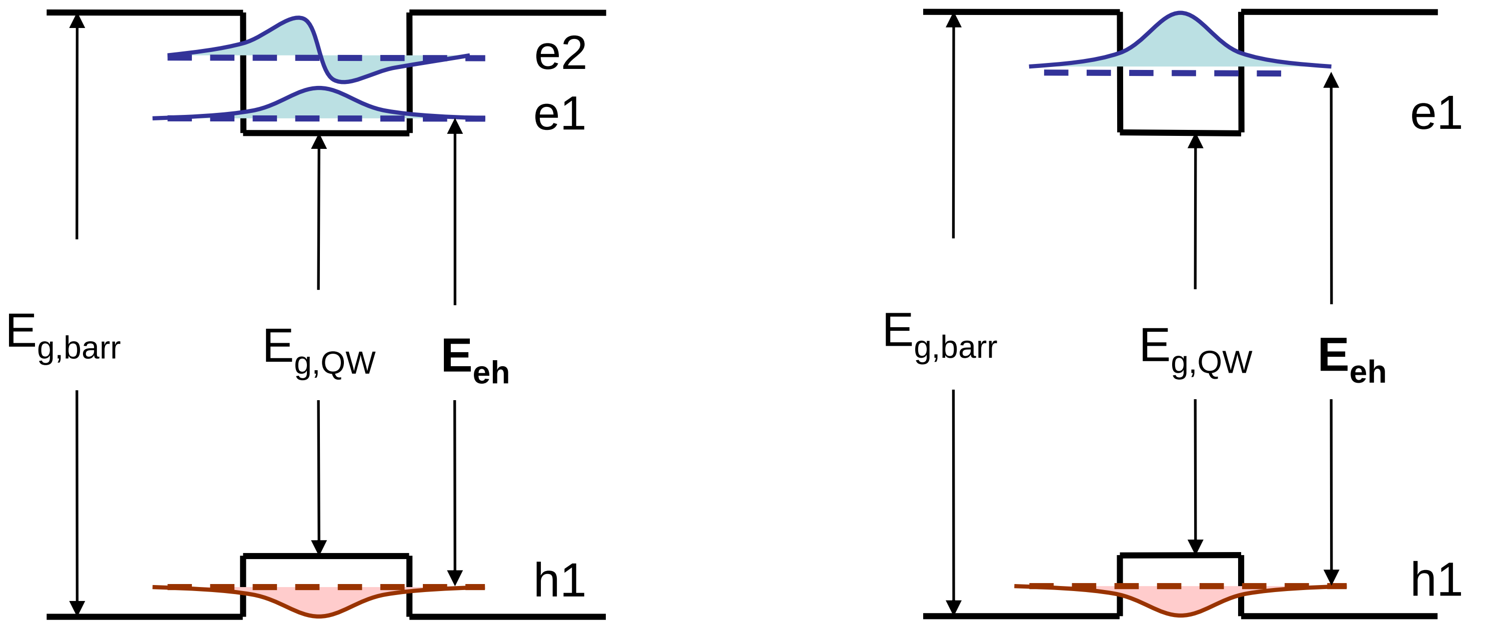




Band engineering: quantum-confined systems

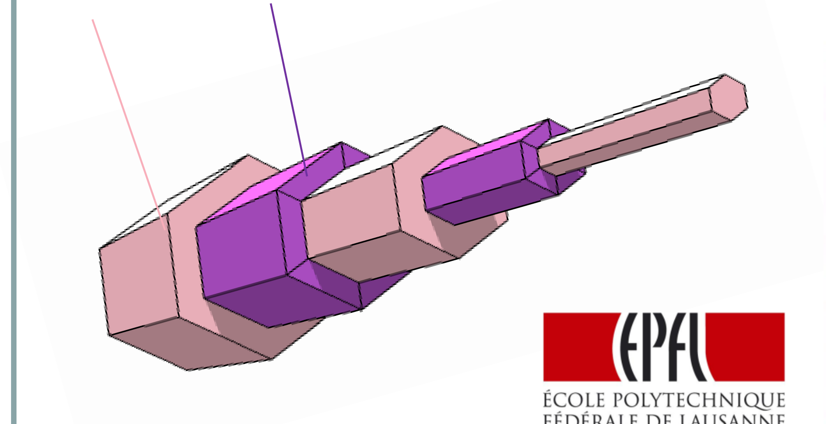
Band engineering:

… or the layer thickness /dot size



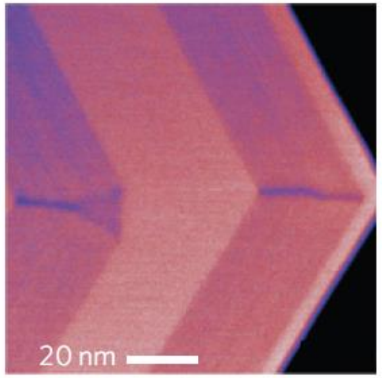
Statistical Correlation

GaAs / AlGaAs core-shell nanowires containing single-photon emitters

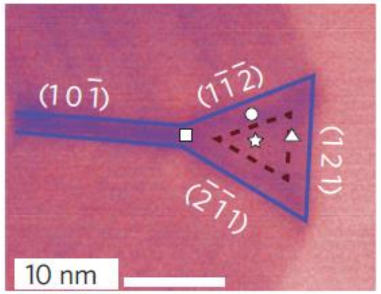


MBE A. Fontcuberta i Morral

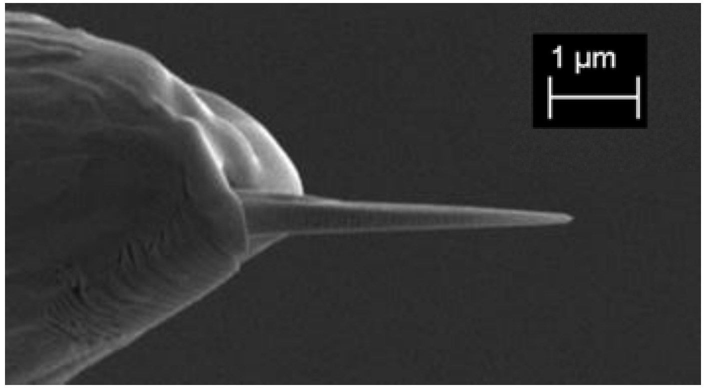
ÉCOLE POLYTECHNIQUE FÉDÉRALE DE LAUSANNE



Heiss et al. Nature Mater. (2013)



Large diameter(~400 nm) → FIB Preparation necessary (ion beam annular milling)



L. Mancini et al. Appl. Phys. Lett. (2014)

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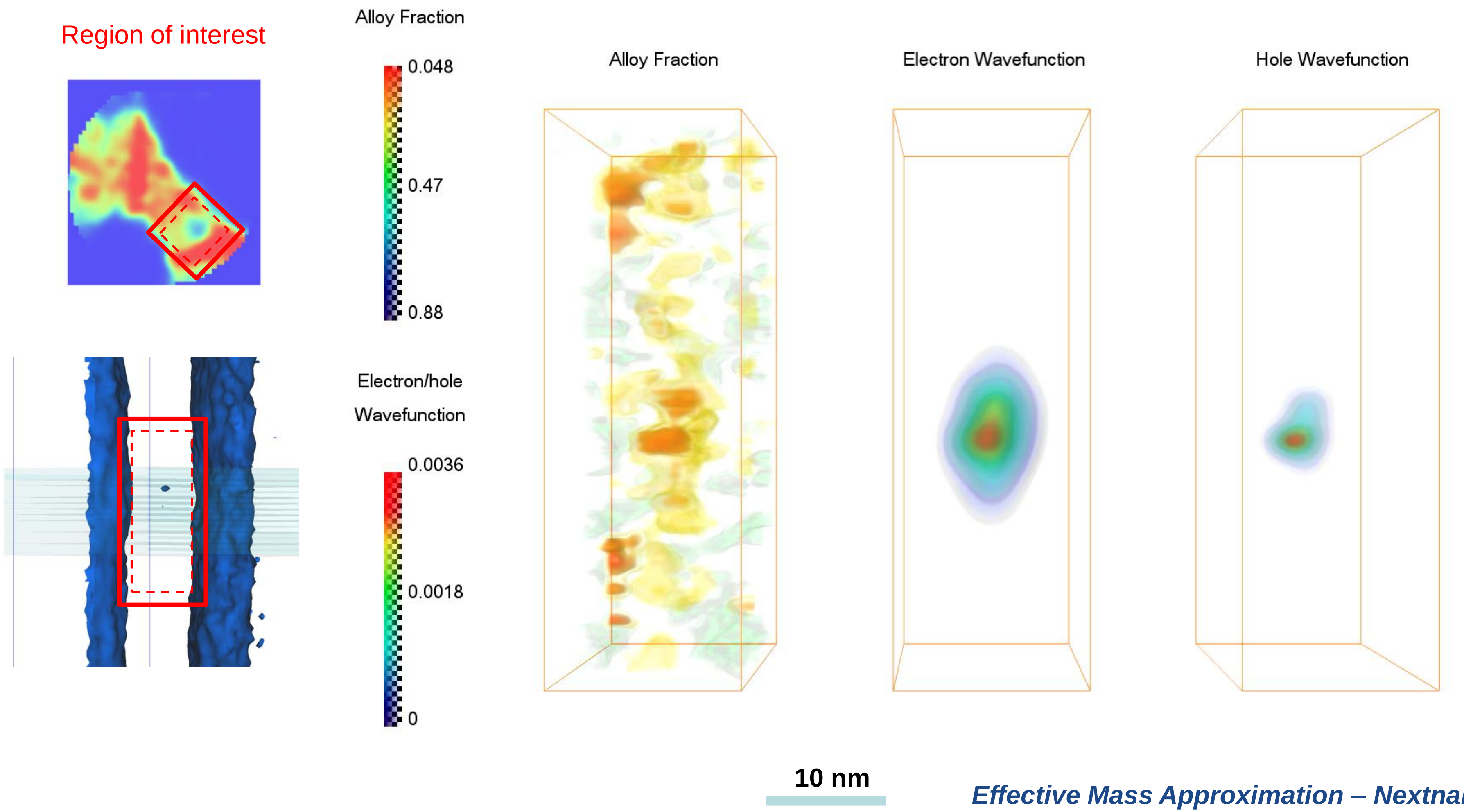
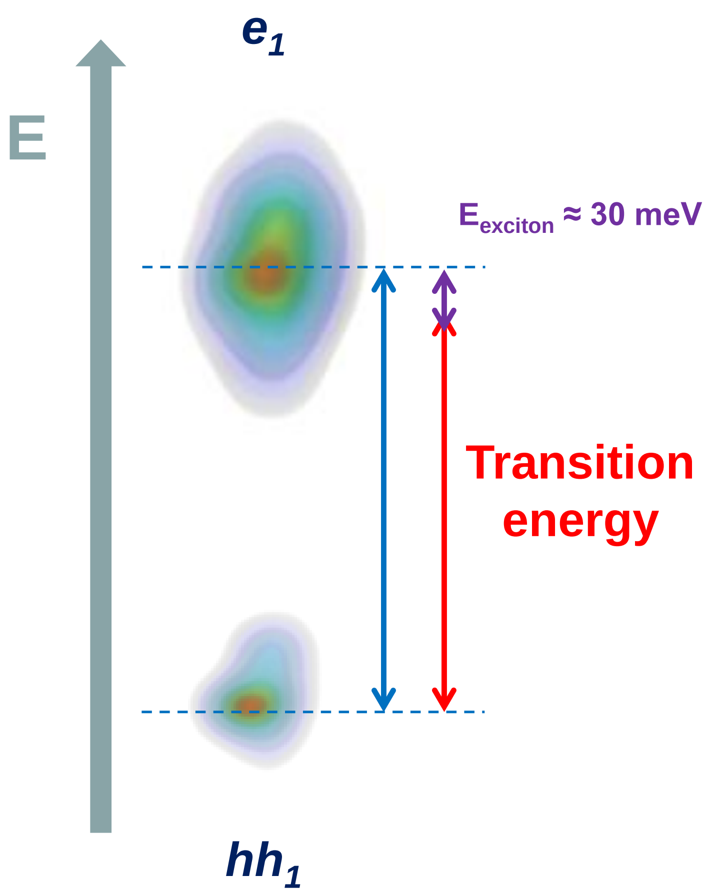
Statistical Correlation

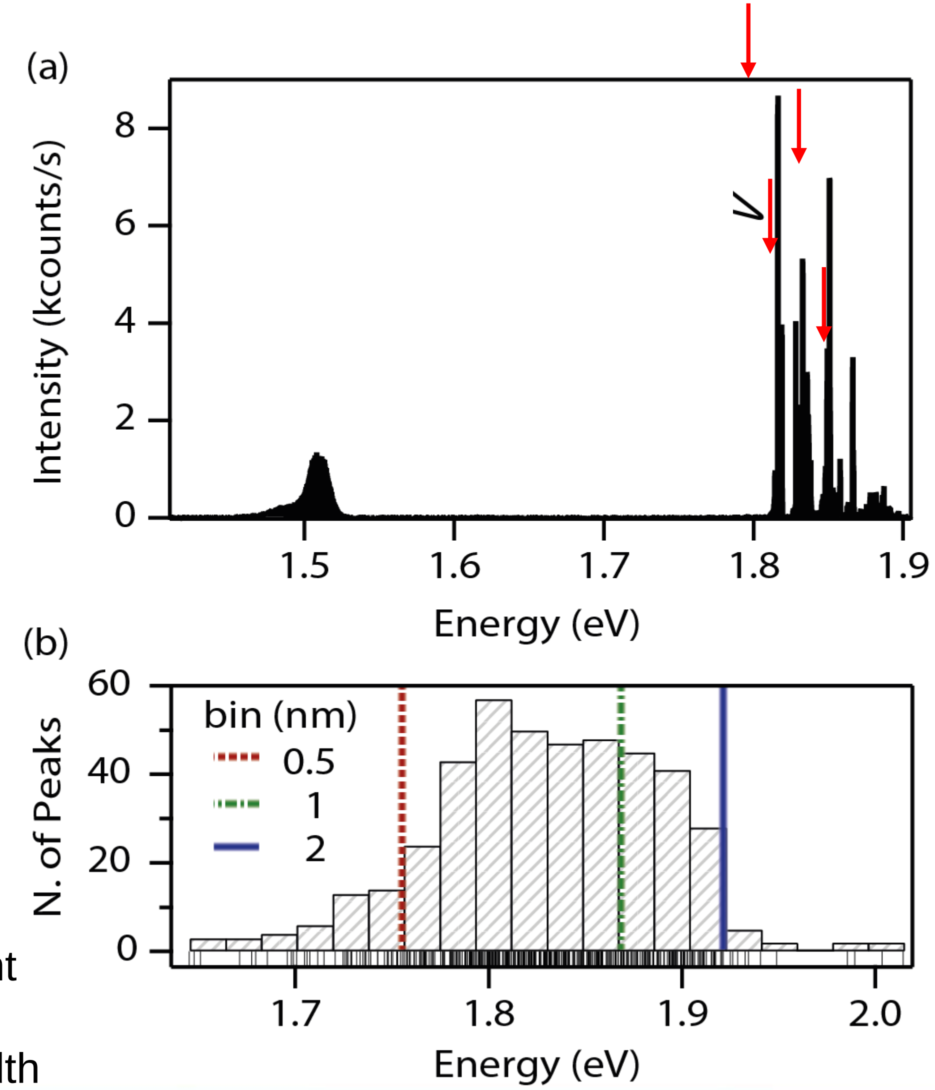


Effective Mass Approximation – Nextnano^3

L. Mancini et al. Appl. Phys. Lett. (2014)

Statistical Correlation



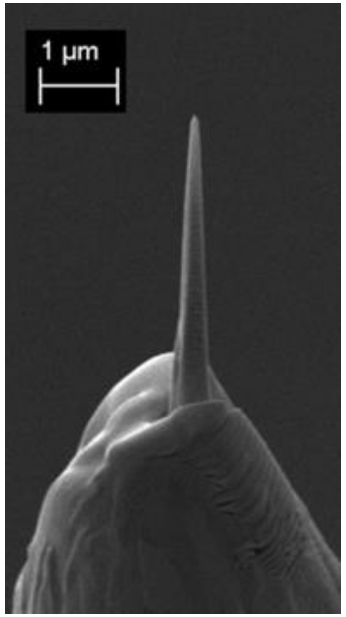


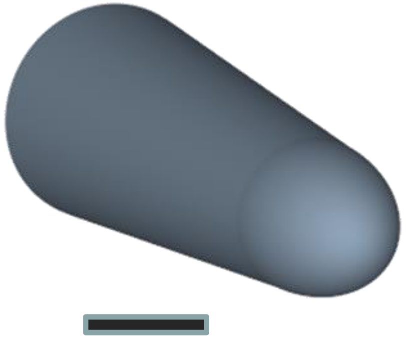
Emission energies in reasonable agreement with experimental PL spectra

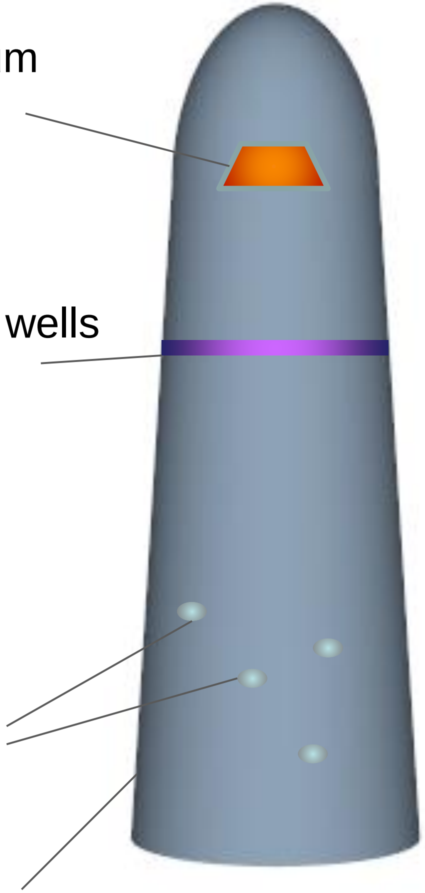
BUT significant dependence on binning width

Optically active nanoscale objects

Aim: correlation of optical and structural properties at the nanoscale







Critical role of surfaces: damaged if specimen prepared by FIB

L. Rigutti et al. Ultramicroscopy (2013)
L. Rigutti et al. Nano Letters (2014)
L. Mancini et al. Appl. Phys. Lett. (2016)
L. Mancini et al. Nano Letters (2017)

E. Di Russo et al. Appl. Phys. Lett. (2017)

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A hopefully virtuous circle

Structural properties

Atom Probe Tomography

Physics of field evaporation

MODEL 1
MODEL 2
….

Functional properties

Optical spectroscopy

Optical signatures

TEM is also very useful, as well as other complementary techniques

L. Rigutti et al. J. Appl. Phys (2016)
L. Rigutti et al. Semicond. Sci. Tech (2016)
L. Rigutti, Acta Physics Polonica (2016)

L. Mancini et al. Appl. Phys. Lett. (2014)
L. Mancini et al. J. Phys. Chem. C (2014)
L. Mancini et al. Appl. Phys. Lett. (2016)
L. Rigutti et al. Scripta Mater. (2017)

Instrumental development (GPM):
- Jonathan Houard
- Antoine Normand
- Gérald Da Costa
- Fabien Delaroche

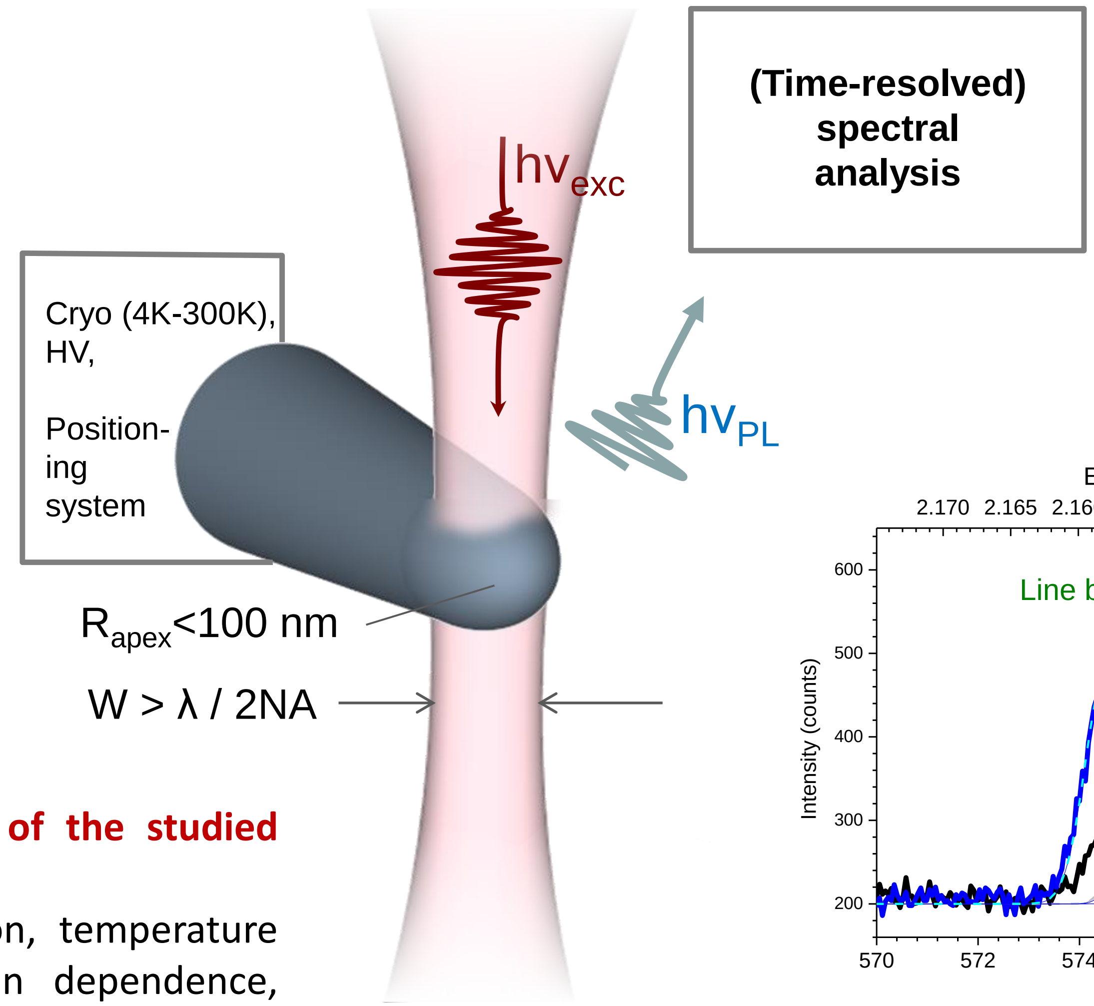
PhD, Post Doc (GPM):
- Enrico Di Russo
- Linda Venturi
- Pradip Dalapati

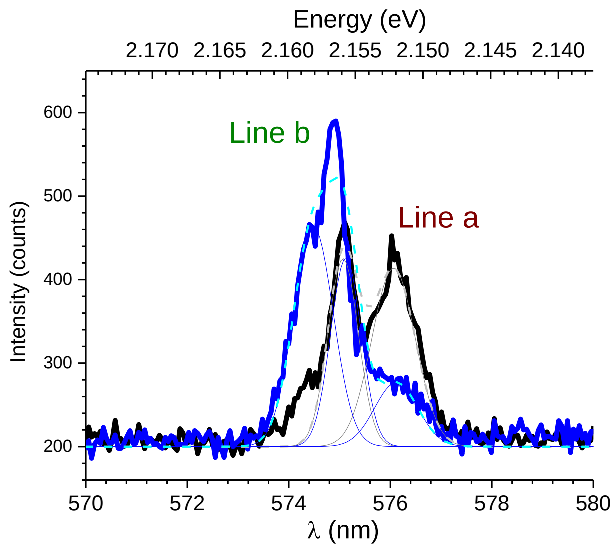
Growth & Synthesis:
- J.M. Chauveau (CRHEA)
- M. Hugues (CRHEA)
- A. Obraztsov (Moscow U.)

Electron Microscopy (GPM):
- Simona Moldovan
- Williams Lefebvre

IN-SITU CORRELATION OF µPL AND APT

Micro-Photoluminescence





Optical signature of the studied system

energy, polarization, temperature dependence, strain dependence, carrier relaxation…

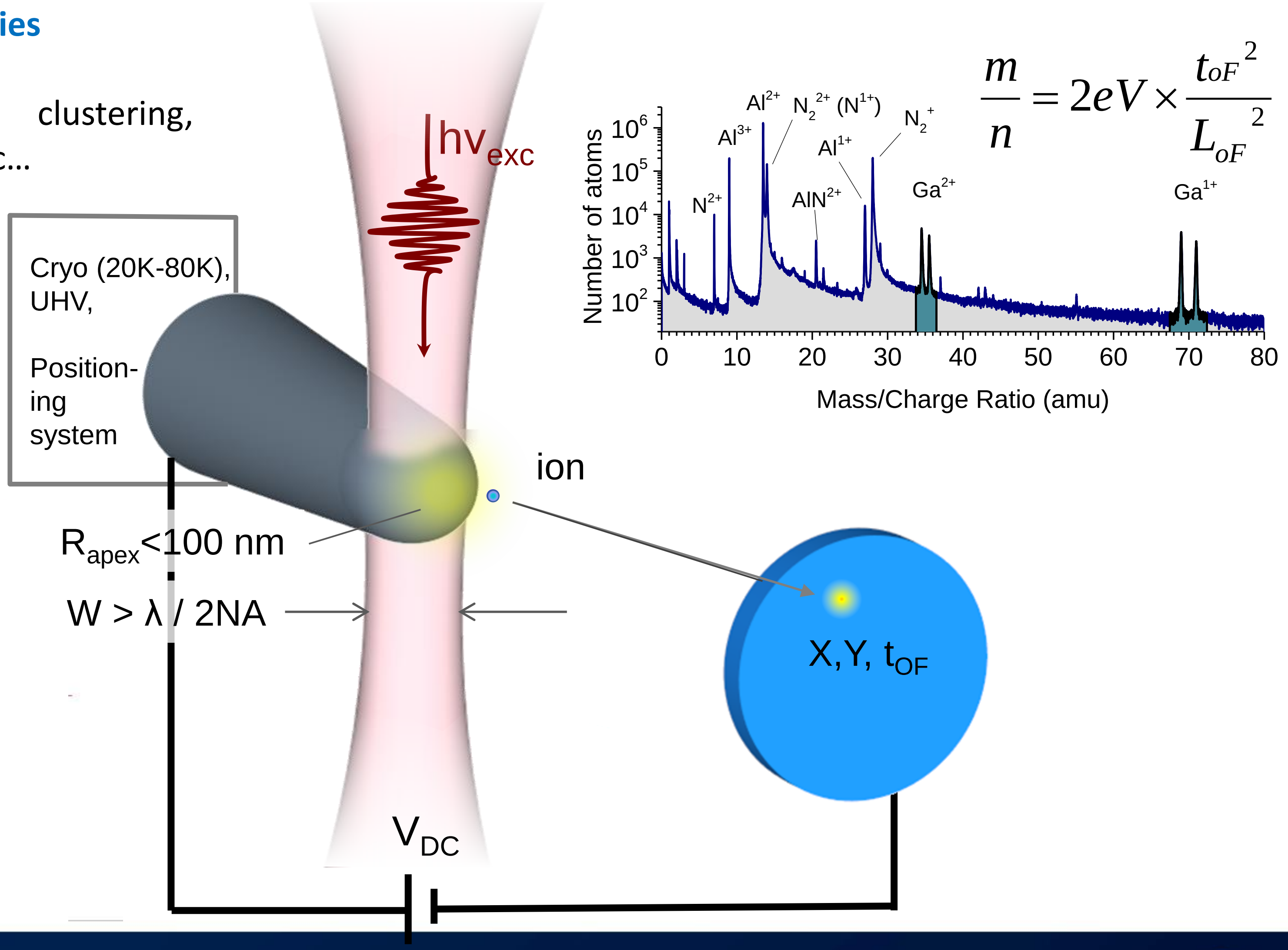
Atom Probe Tomography

Structural properties

Homogeneity, clustering, interfaces, size, etc…

Crystal symmetry?

10 nm

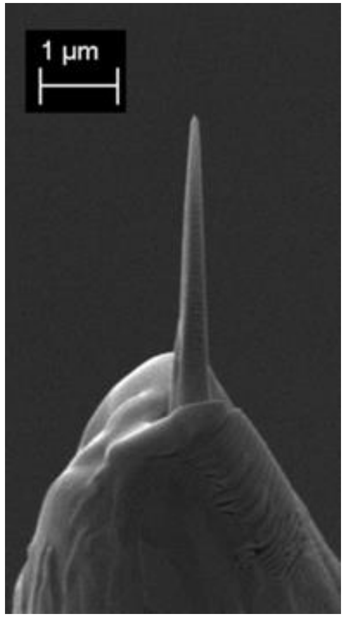


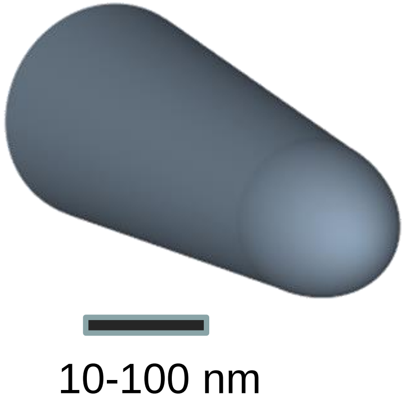
$$\frac{m}{n} = 2eV \times \frac{t_{oF}^{\;2}}{L_{oF}^{\;2}}$$

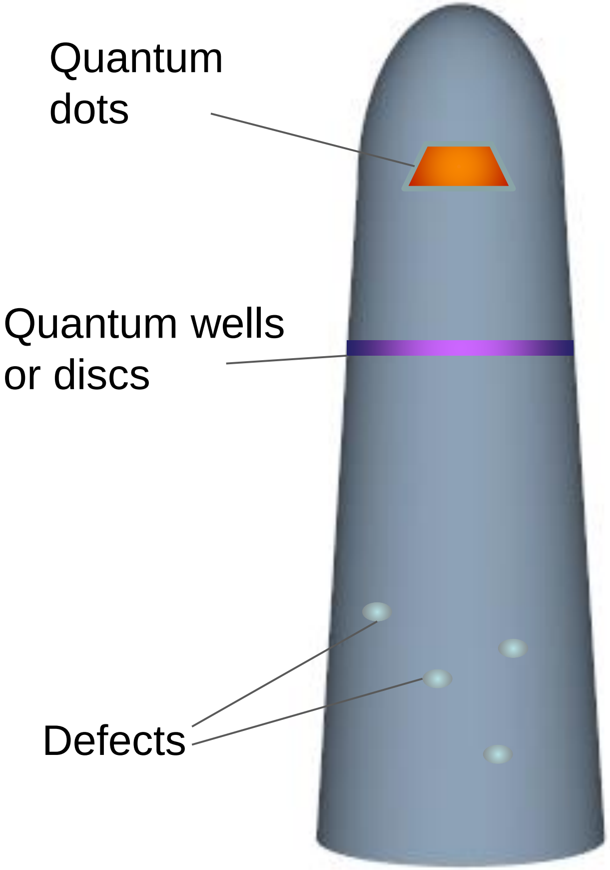
D. Blavette et al. Nature (1993)
D. R. Kingham Surf Sci. (1982)
D. Blavette et al. RSI (1993)
B. Gault et al. RSI (2006)
F. Vurpillot et al. Utramicroscopy (2013)

Correlative studies of individual nanoscale objects

Aim: understanding the optical signature (Energy, Polarization, etc.) of a nanoscale system based on its structural properties.



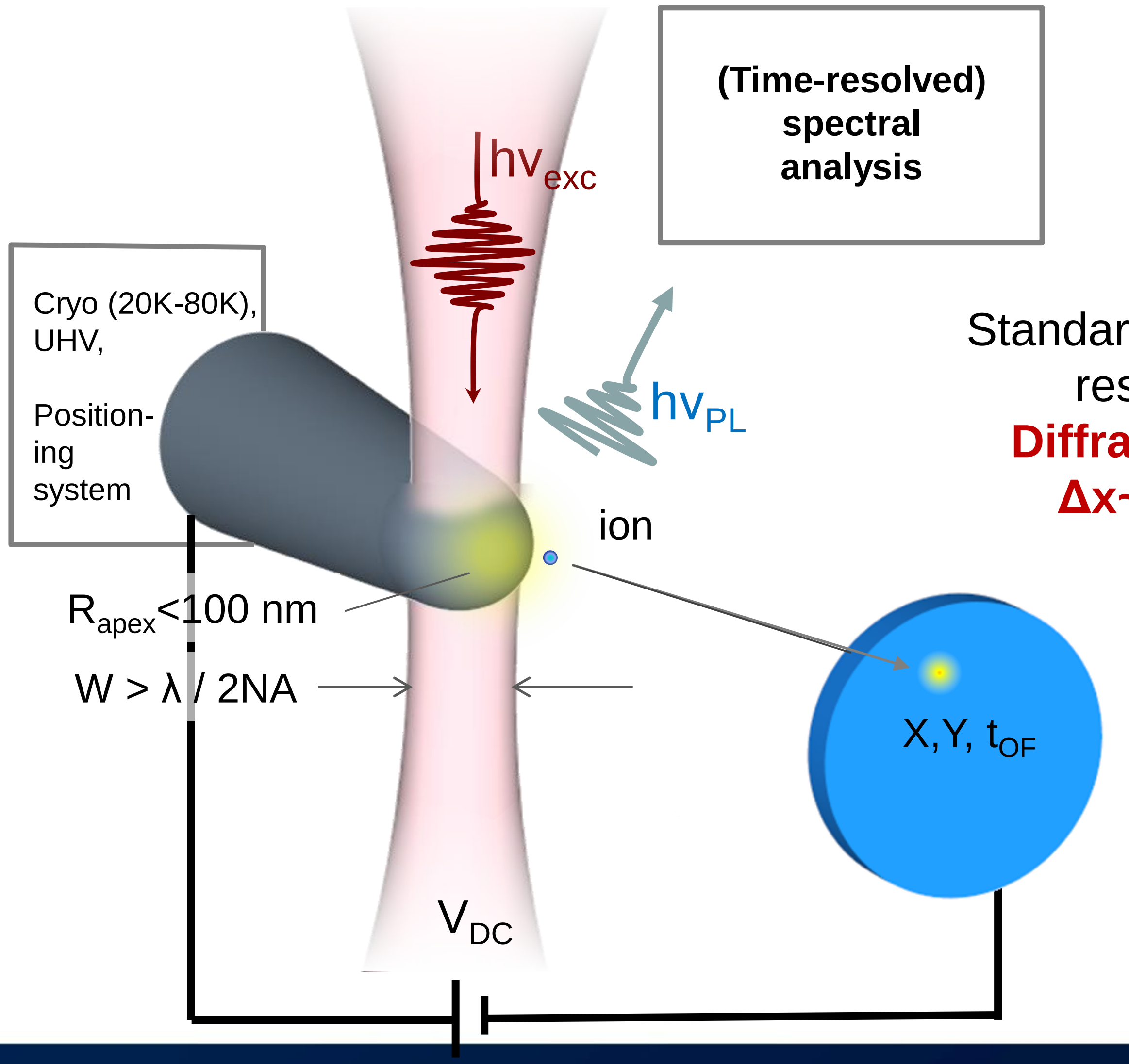




L. Rigutti et al. Ultramicroscopy (2013)
L. Rigutti et al. Nano Letters (2014)
L. Mancini et al. Appl. Phys. Lett. (2016)
L. Mancini et al. Nano Letters (2017)

E. Di Russo et al. Appl. Phys. Lett. (2017)

In-situ µPL + APT



Standard µPL spatial resolution:
Diffraction limit
Δx~λ/(2NA)

In-situ µPL + APT

Chief operator
& actual
µPL-APT interface

APT chamber

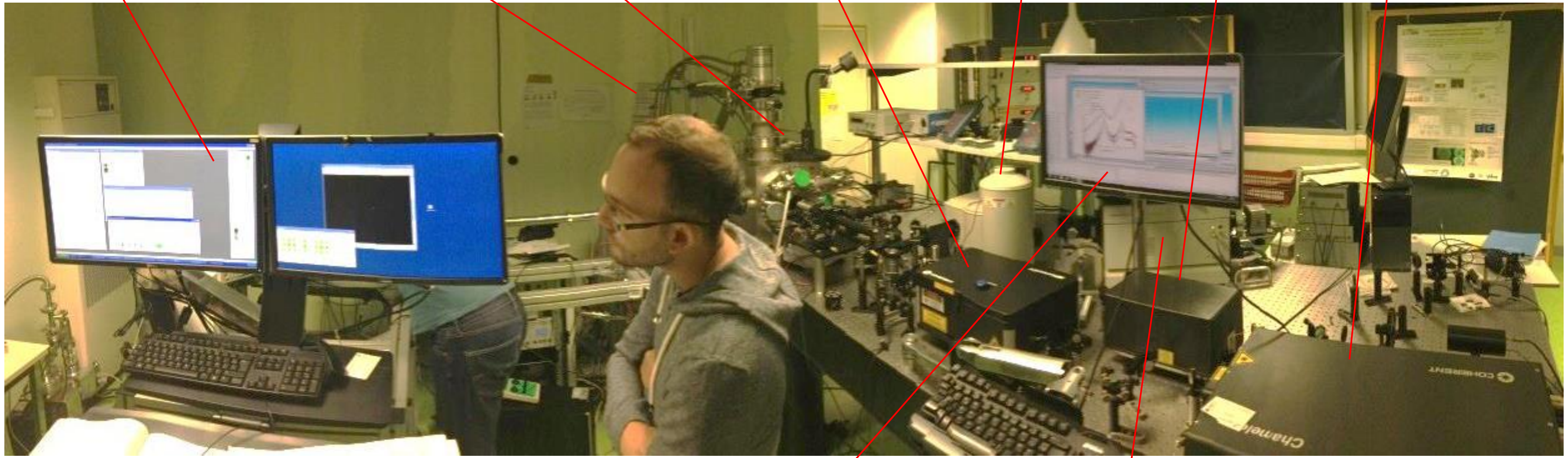
SHG-THG
generation

Spectrometer
+ CCD

Ti-Sa fs laser
(680-1100 nm)
80 MHz

APT control

Pulse picker



Spectrometer
Control

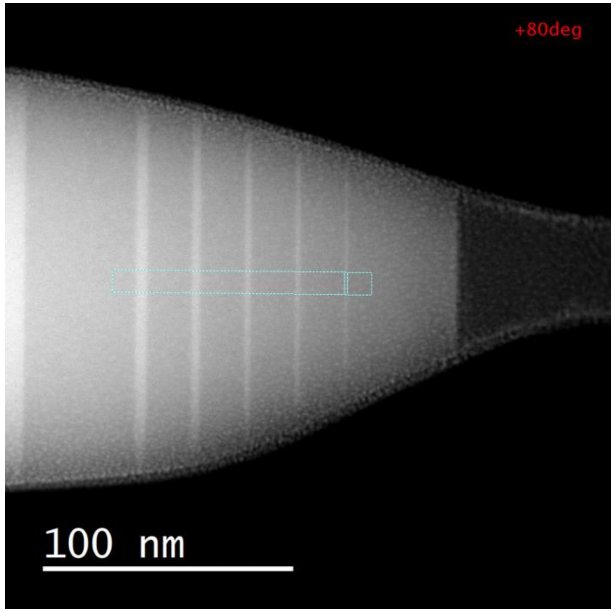
Streak Camera

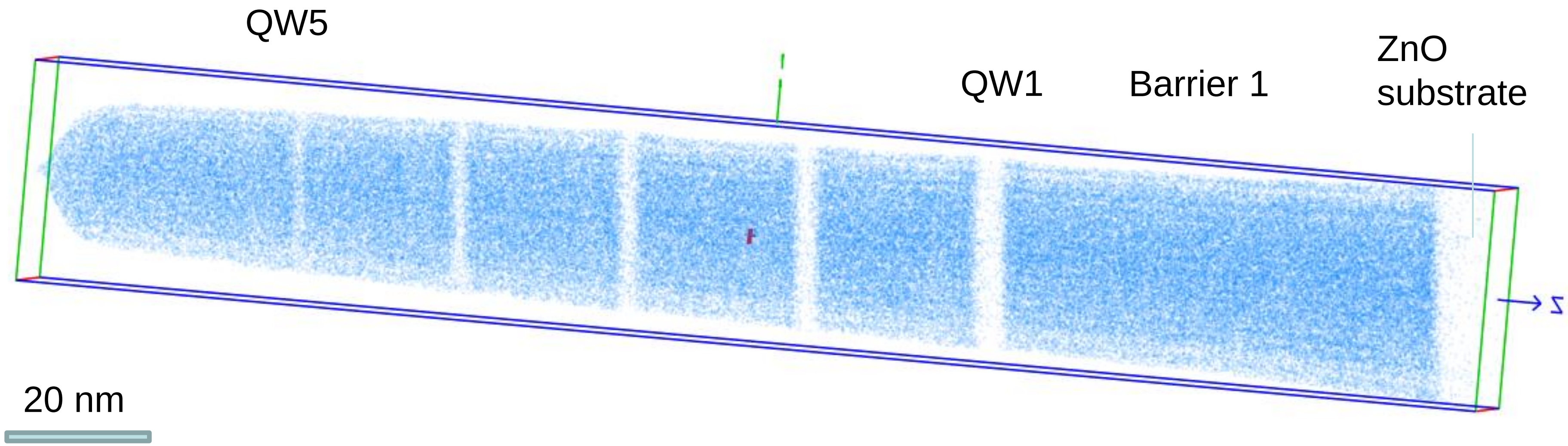
Semipolar QWs

5 ZnO/$Mg_{0.15}Zn_{0.85}O$
r-plane QWs with
decreasing thickness
Interwell distance ~20 nm

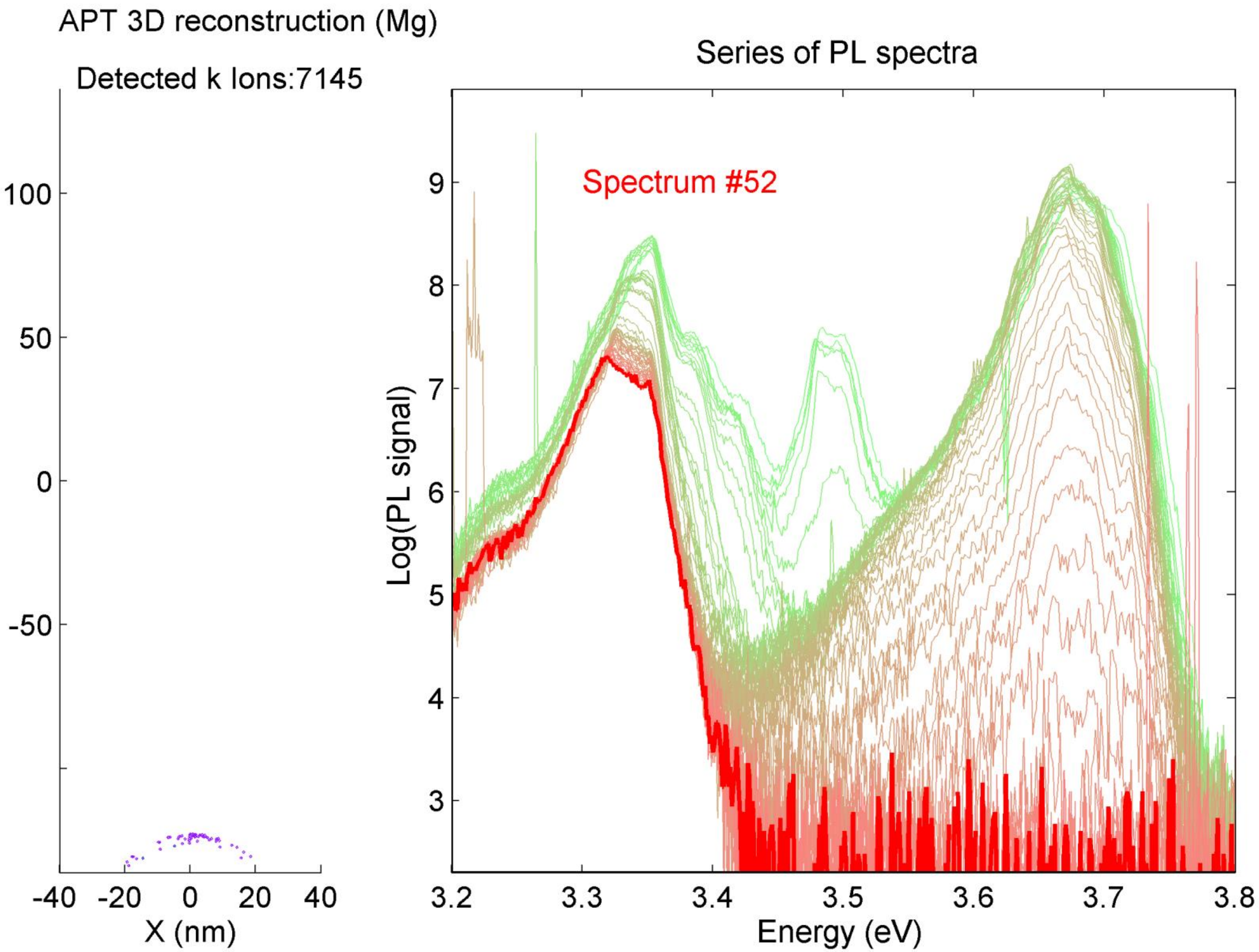
Mg II-site fraction y=0.148 ± 0.02

QW	Thickness (nm)
5	0.5 ± 0.1
4	1.0 ± 0.2
3	1.9 ± 0.2
2	3.2 ± 0.2
1	4.1 ± 0.2





In-situ µPL-APT



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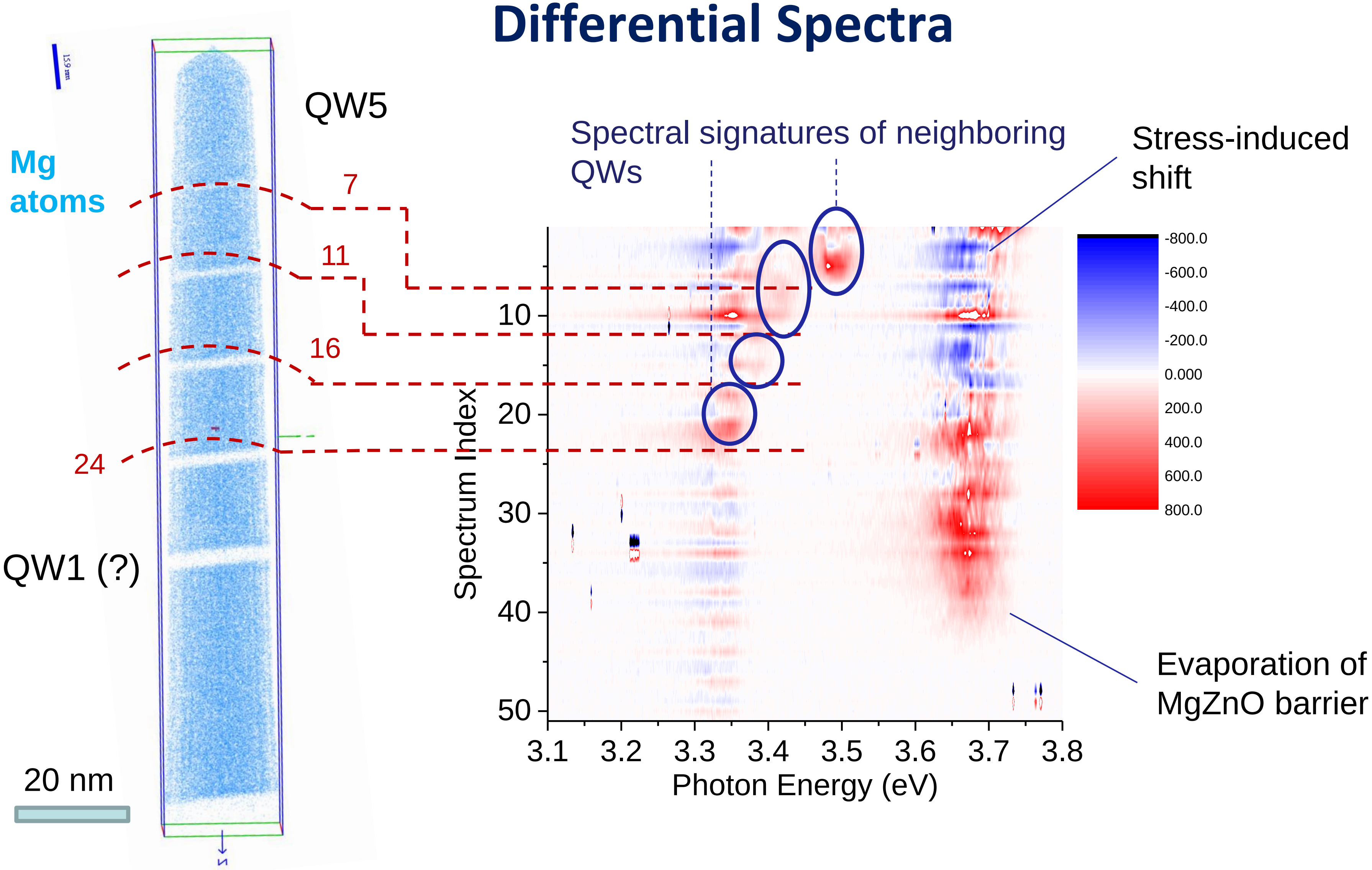
Differential Spectra

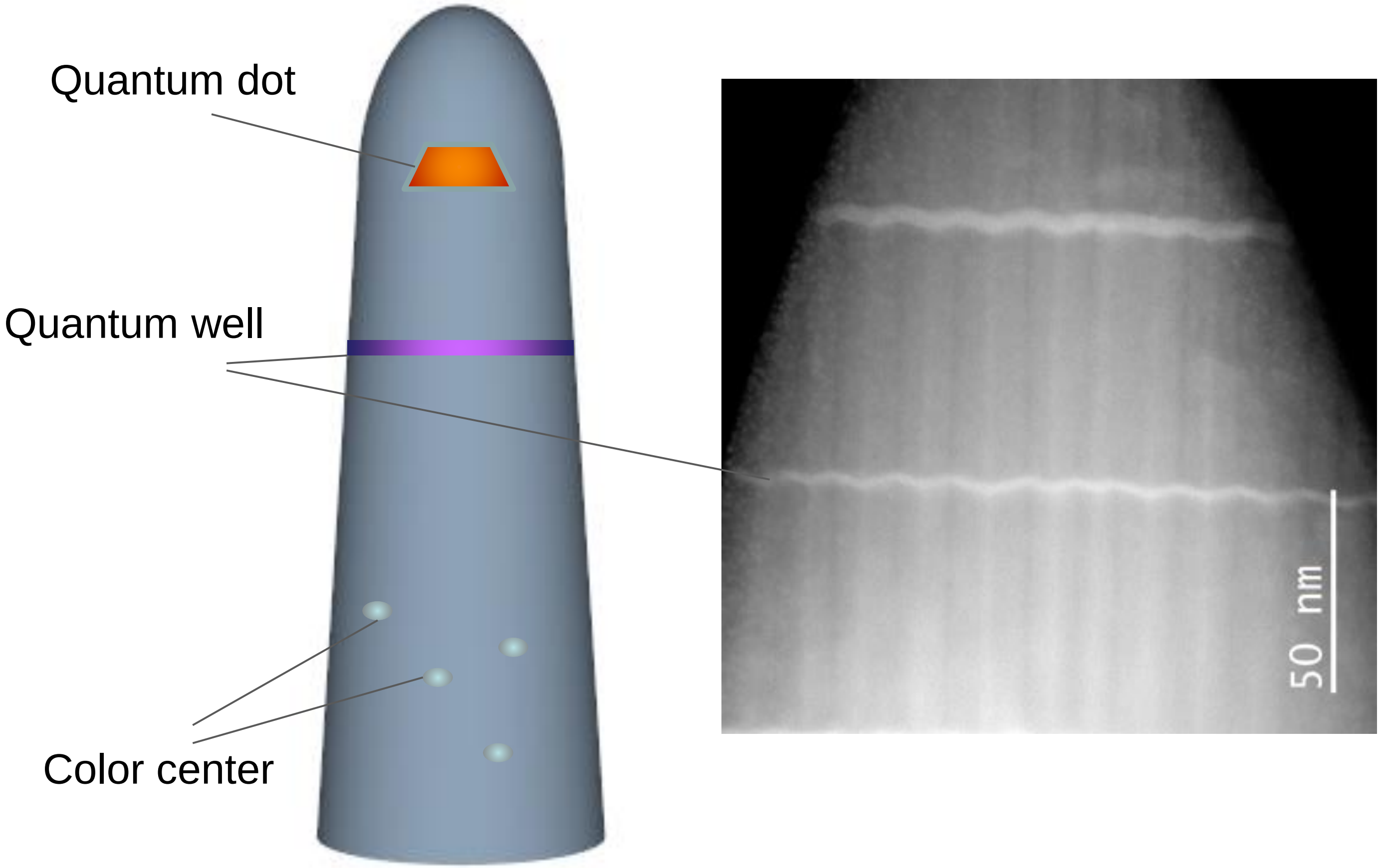


Single-emitter spectroscopy

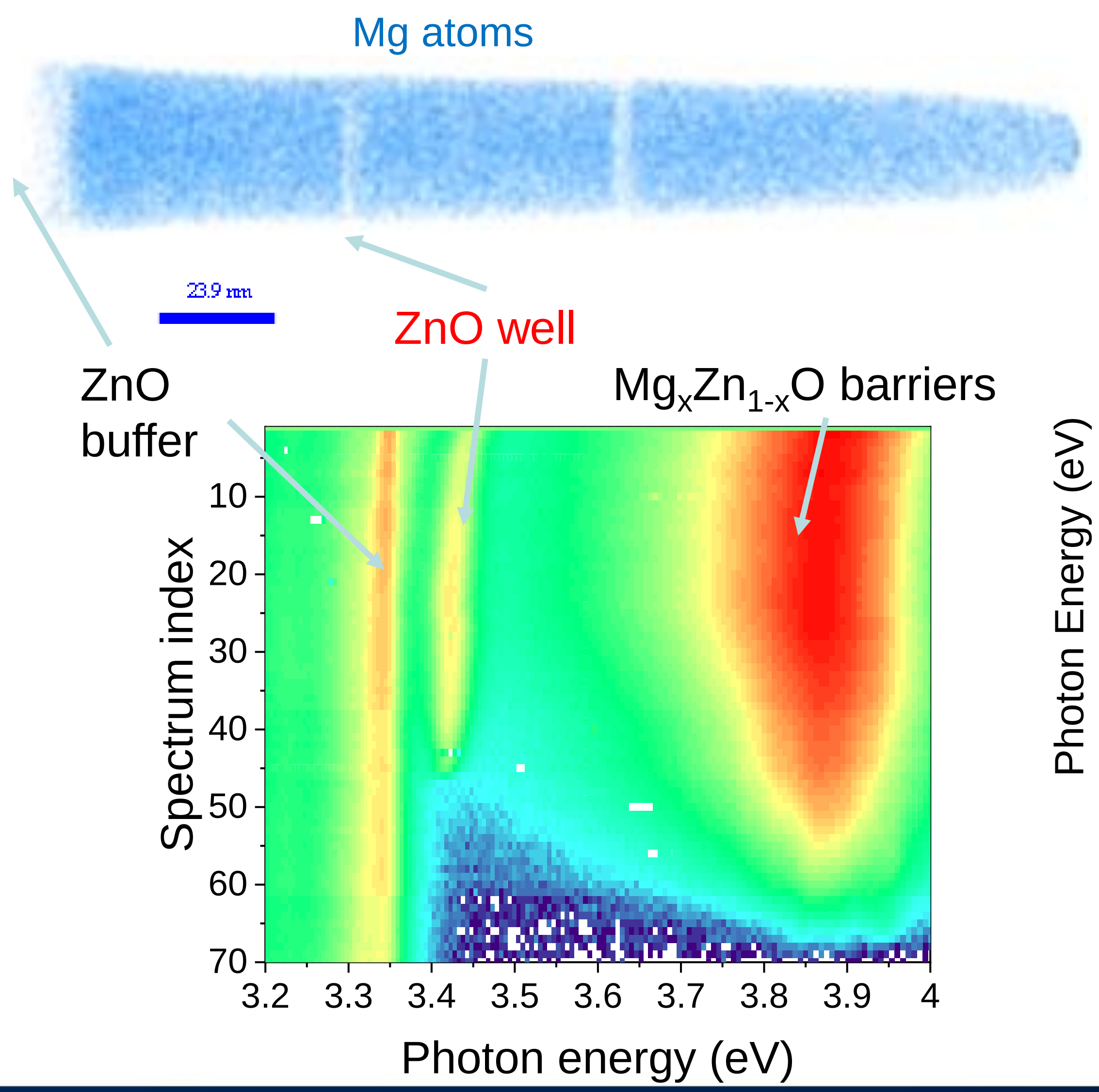
Physics of field evaporation
Single probe of stress state and optical field inside a tip specimen.

Materials Science
Correlation between structural and optical properties of a single quantum dot (ex-situ also).

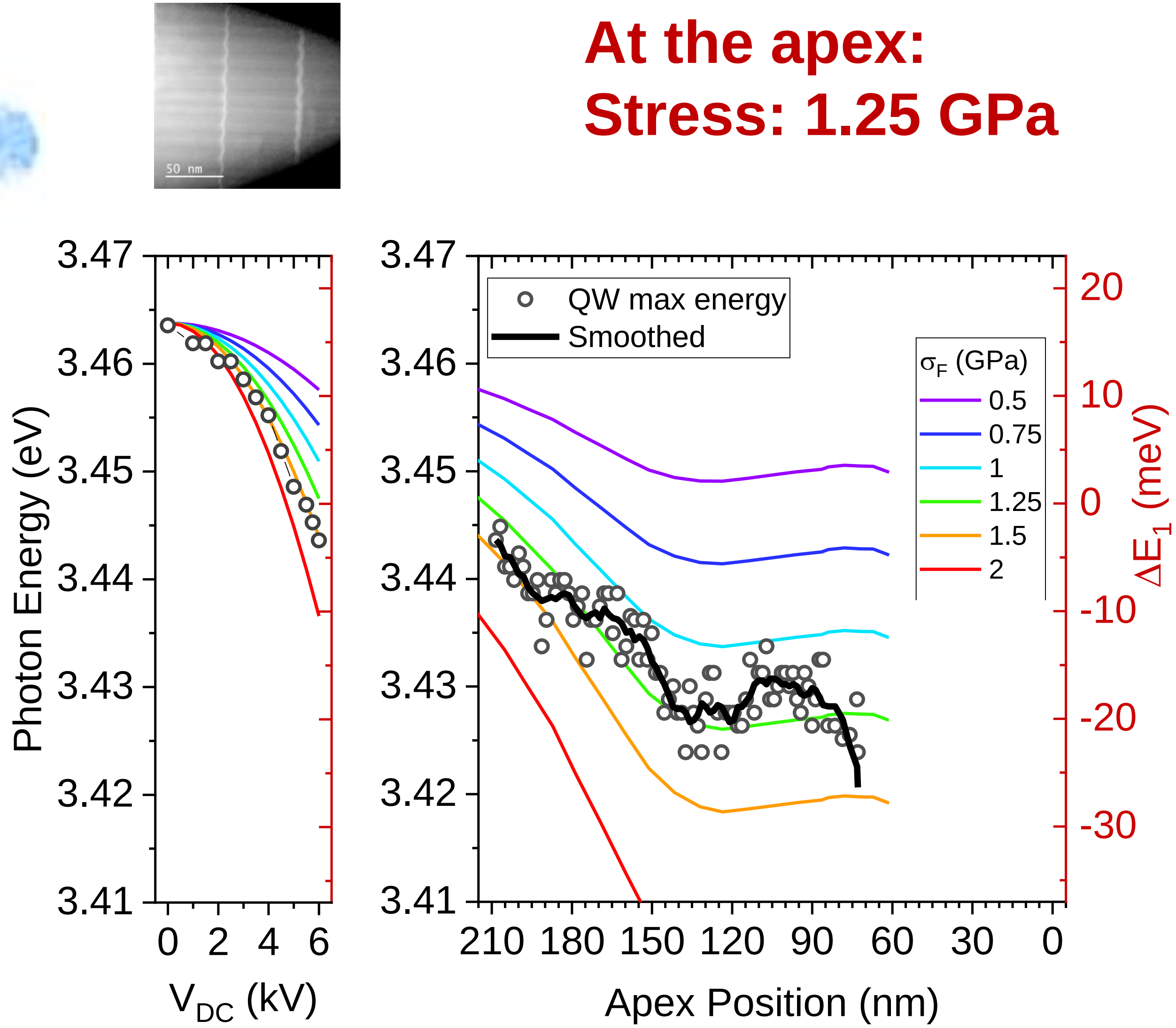
Nanophotonics
Interaction between localzed emitter and changing environment (waveguiding, scattering)



Spectral evolution of a single emitter



At the apex:
Stress: 1.25 GPa



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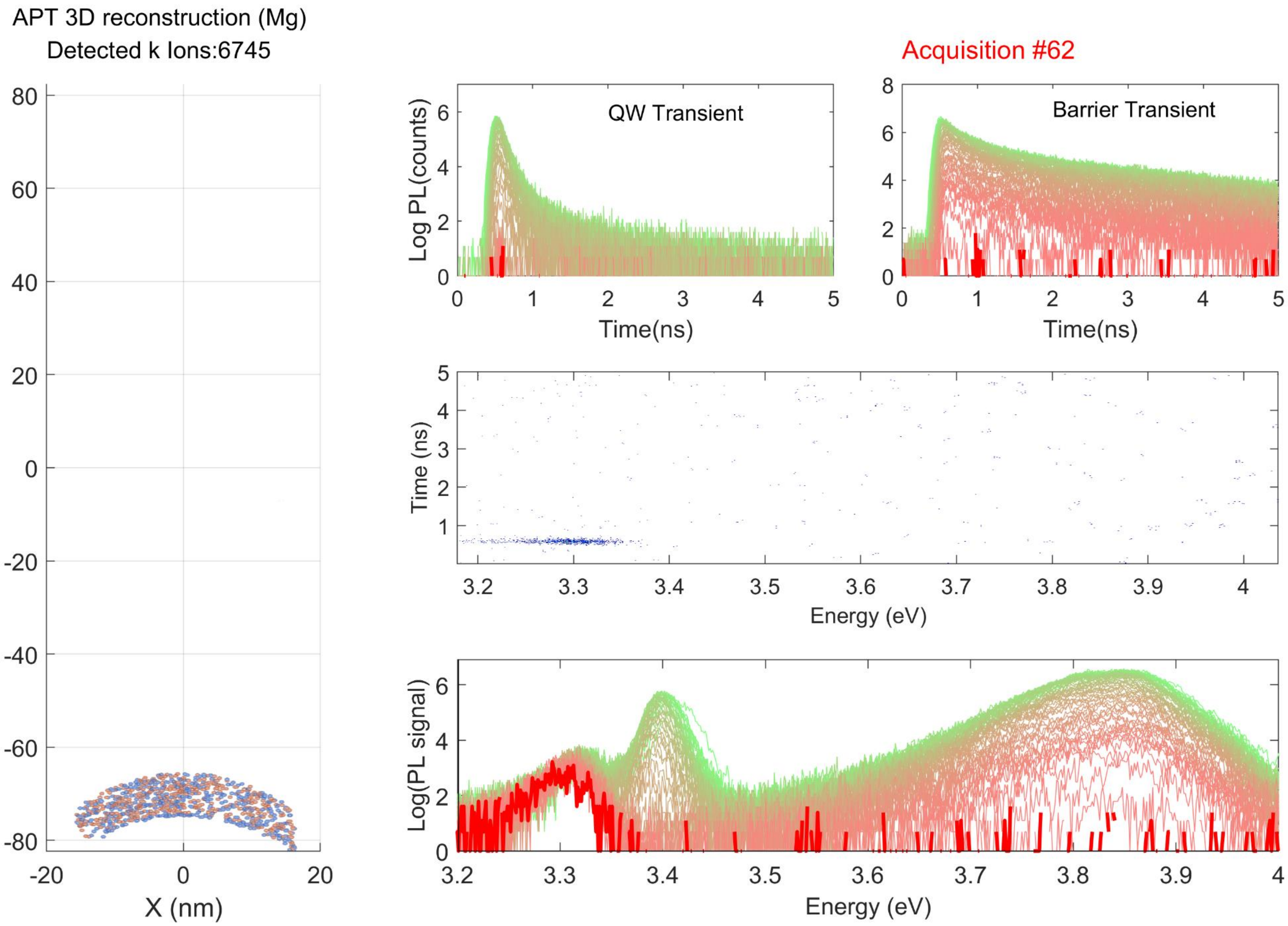
Time-resolved PL during APT



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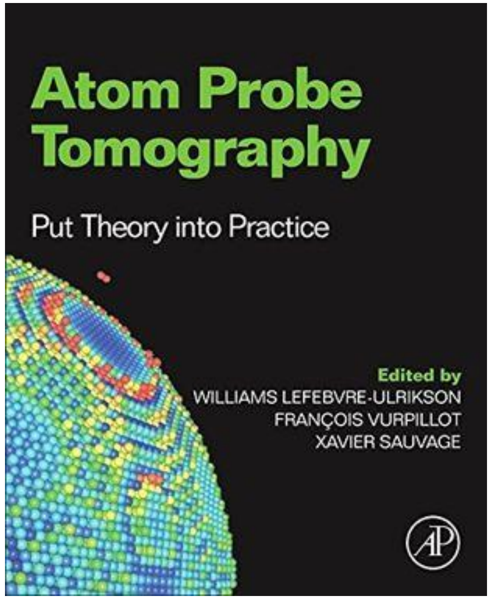
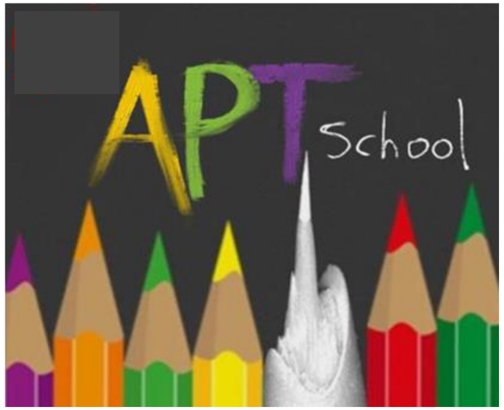
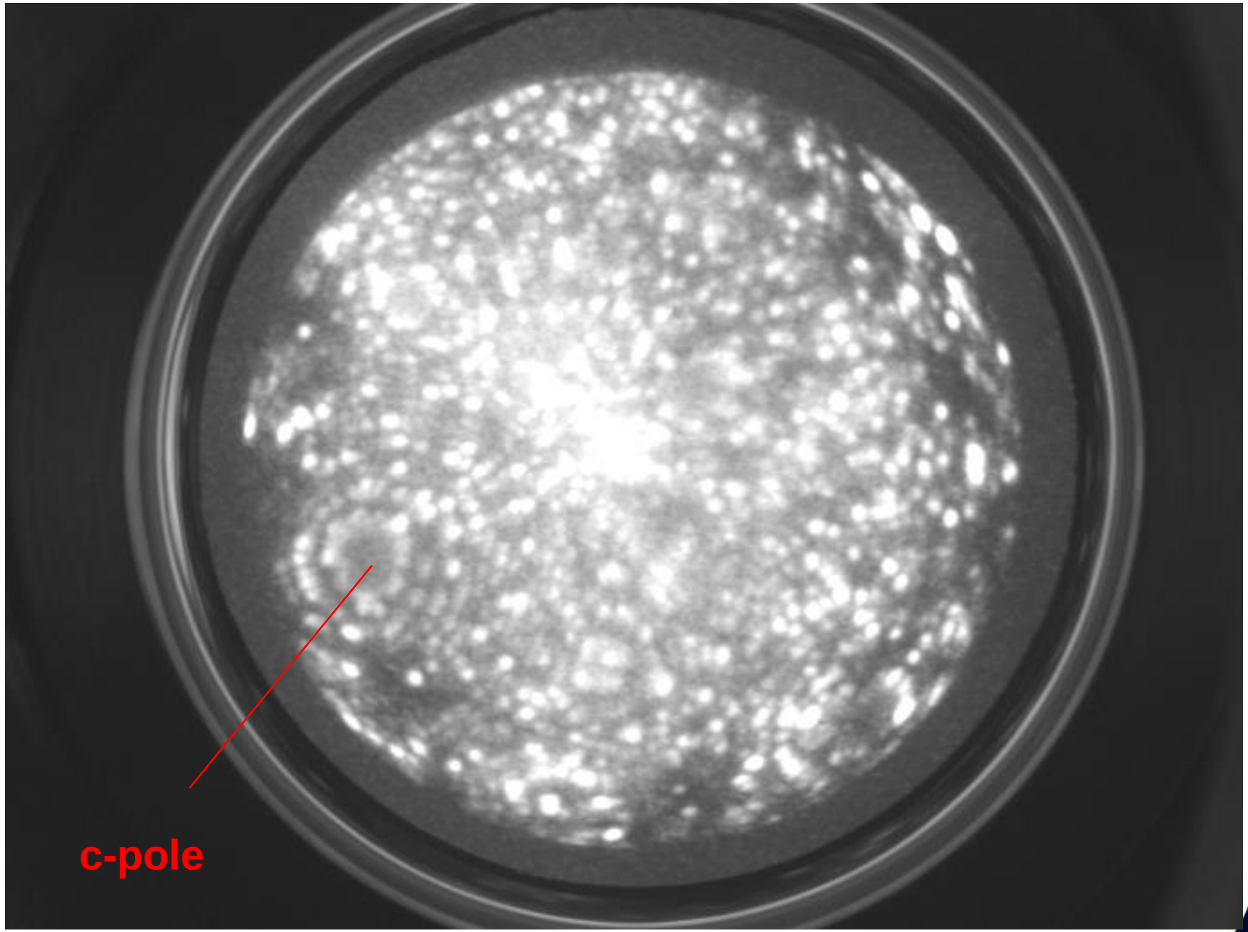
Conclusions



International APT School,
Rouen, ~fall 2021



FIM of evaporating GaN



Acknowledg€ments

GPM Rouen
M. Gilbert, J. Houard, D. Shinde, I. Blum, A. Normand, D. Hernandez-Maldonado, A. Vella, F. Vurpillot, B. Deconihout, W. Lefebvre, S. Duguay, D. Blavette

C2N Université Paris Saclay
M. Tchernycheva, F. Julien, L. Mancini
J.C. Harmand, L. Largeau, N. Gogneau

CEA-CNRS-U-Grenoble Alpes-Inst. Néel
C. Durand, J. Eymery, R. Songmuang, A. Das, E. Monroy

EPFL Lausanne
A. Fontcuberta i Morral, Y. Fontana, E. Russo Averchi, M. Heiss, L. Francaviglia
G. Jacopin, R. Butté, J.F. Carlin, N. Grandjean

Lomonosov University Moscow
A. Obraztsov, E. Obraztsova, S. Malykhin

CRHEA Sophia-Antipolis
J.M. Chauveau, M. Hugues, N. Le Biavan

SIMI 10 JCJC project "TAPOTER "

Labex EMC3 project « ASAP »

CPER-FEDER Cathy-2, Bridge, iMust

Institut Carnot ESP projects "NanoT-AP", "TeraSAT"

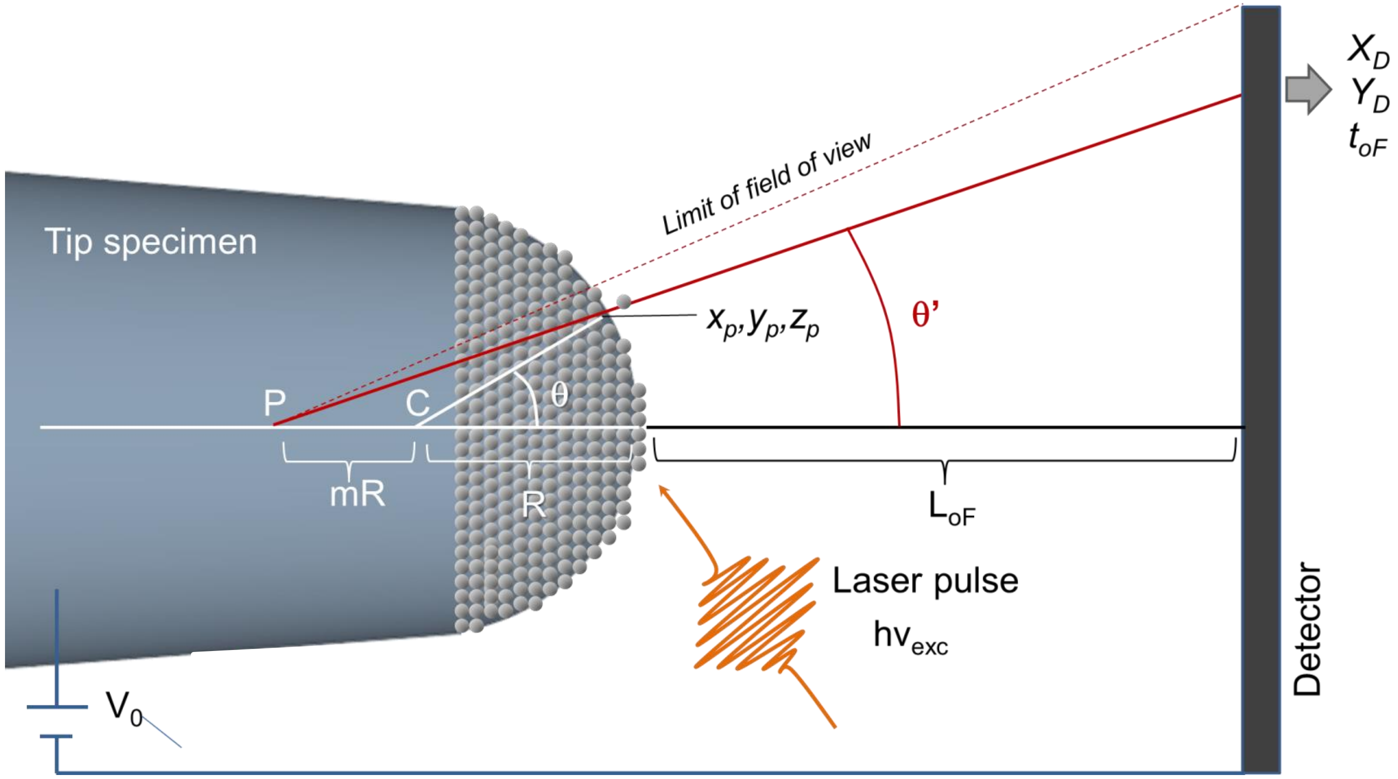
Remerciements

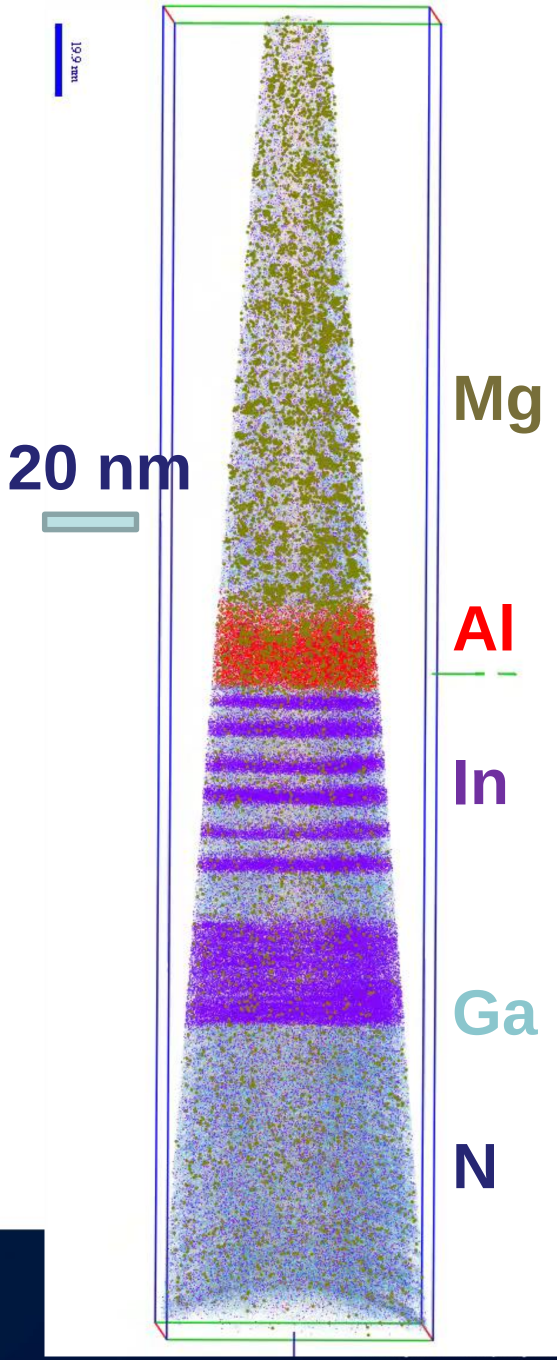
Labex EMC3 projet « ASAP »

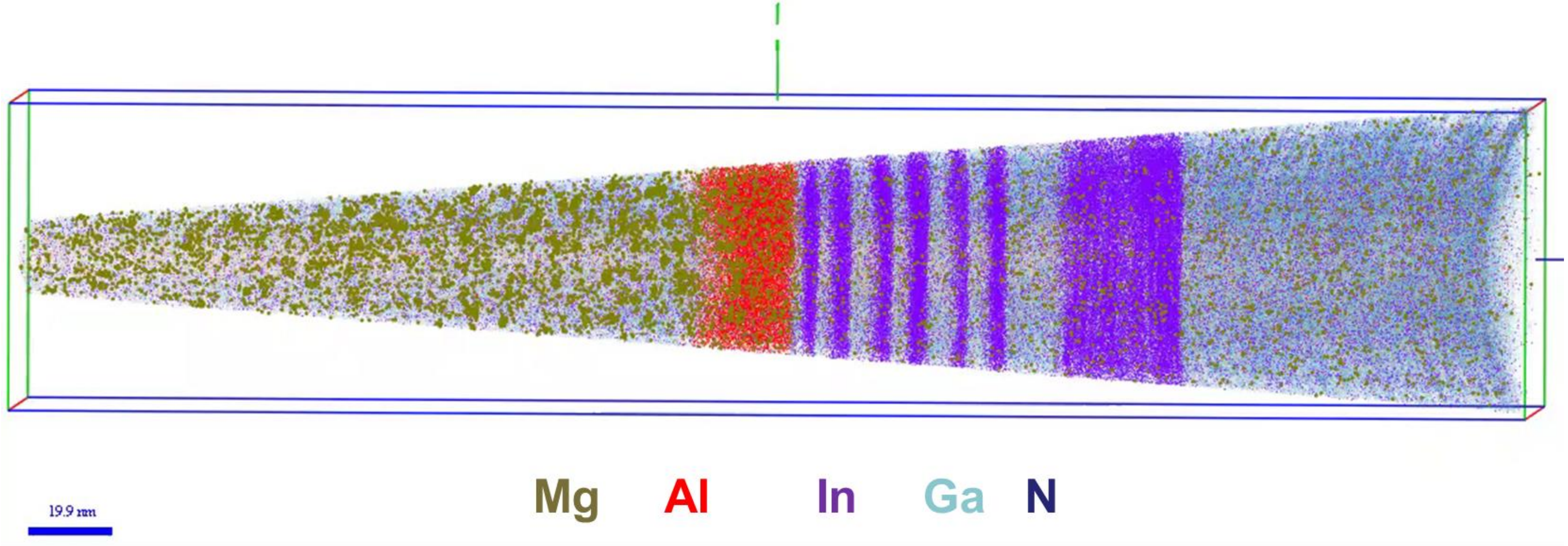
JCJC projet « TAPOTER »

GRR électronique MIST

Institut Carnot ESP projet NanoT-AP


Limit of field of view
Tip specimen
x_p, y_p, z_p
θ'
X_D
Y_D
t_{oF}
P
C
θ
mR
R
L_{oF}
Laser pulse
$h\nu_{exc}$
Detector
V_0


20 nm
Mg
Al
In
Ga
N



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I. Blum

Collaboration GPM Rouen – CIMAP Caen

B. Gervais, D. Zanuttini, E. Jacquet. J. Douady, P.M. Anglade

Projet Labex EMC3 « AQURATE »

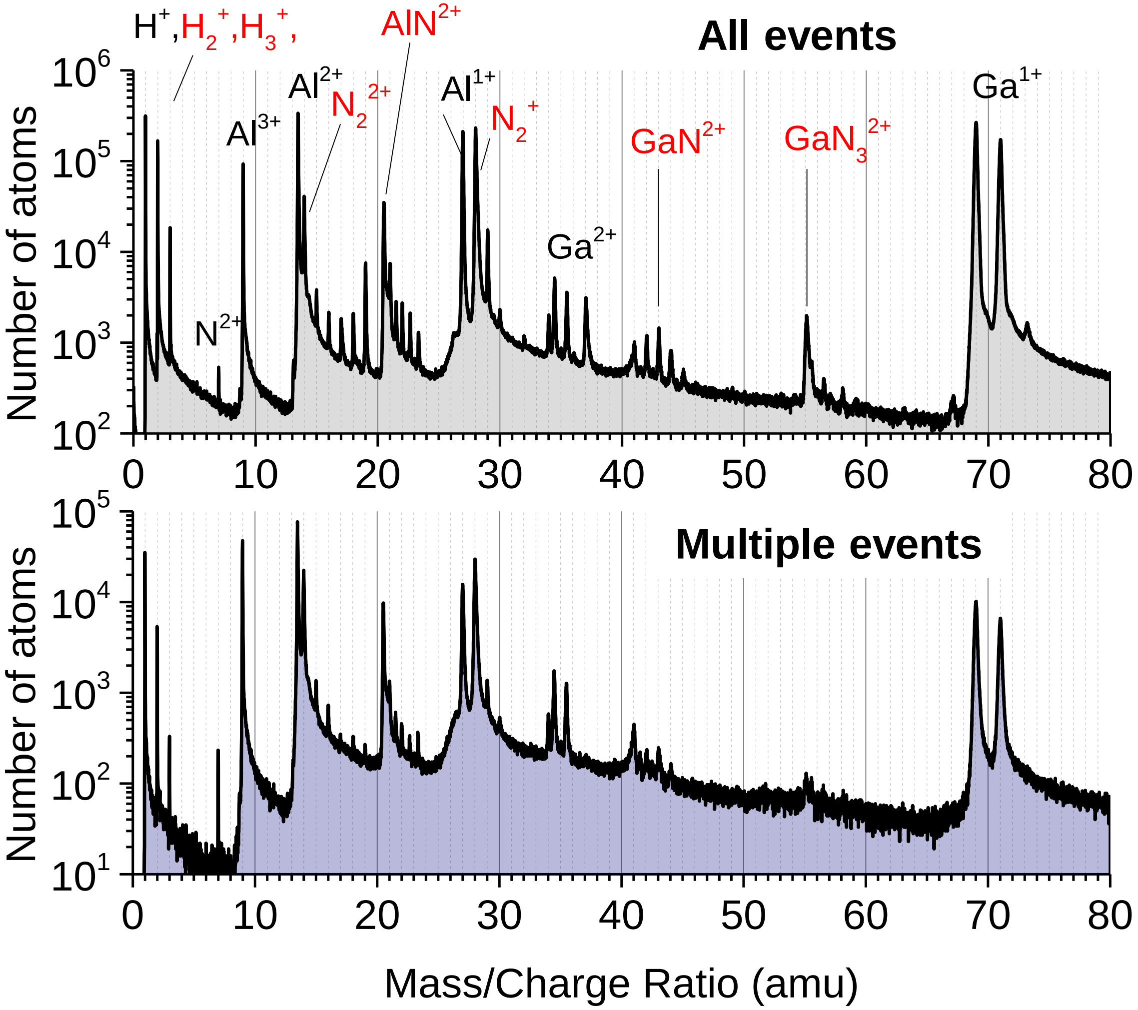
E. Di Russo

Entracte

A REACTION MICROSCOPE

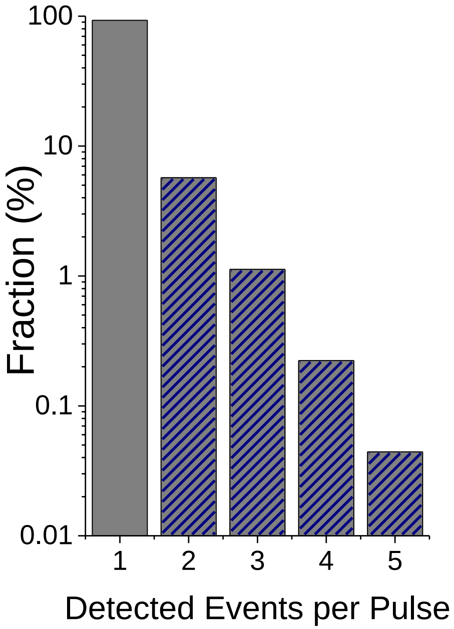
STUDY OF MOLECULAR DISSOCIATION REACTIONS IN ATOM PROBE

Mass spectrum of AlGaN/GaN heterostructure



Molecular ions are present

~10% of the detected events are multiple occurring after a single laser pulse



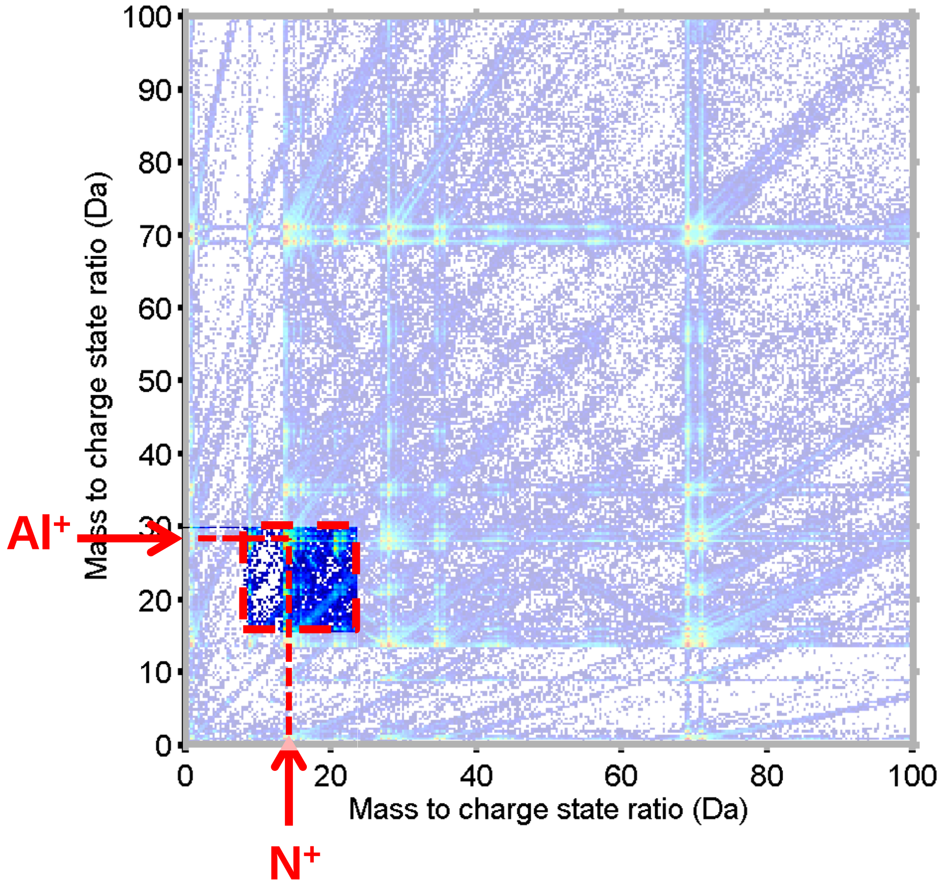
Correlated evaporation events

- 1 pulse → n events ≥ 2
- Ions being detected together are chemically correlated

→ Can plot each pair in a 2D histogram

Contains information both on:

- correlated evaporation
- dissociation events

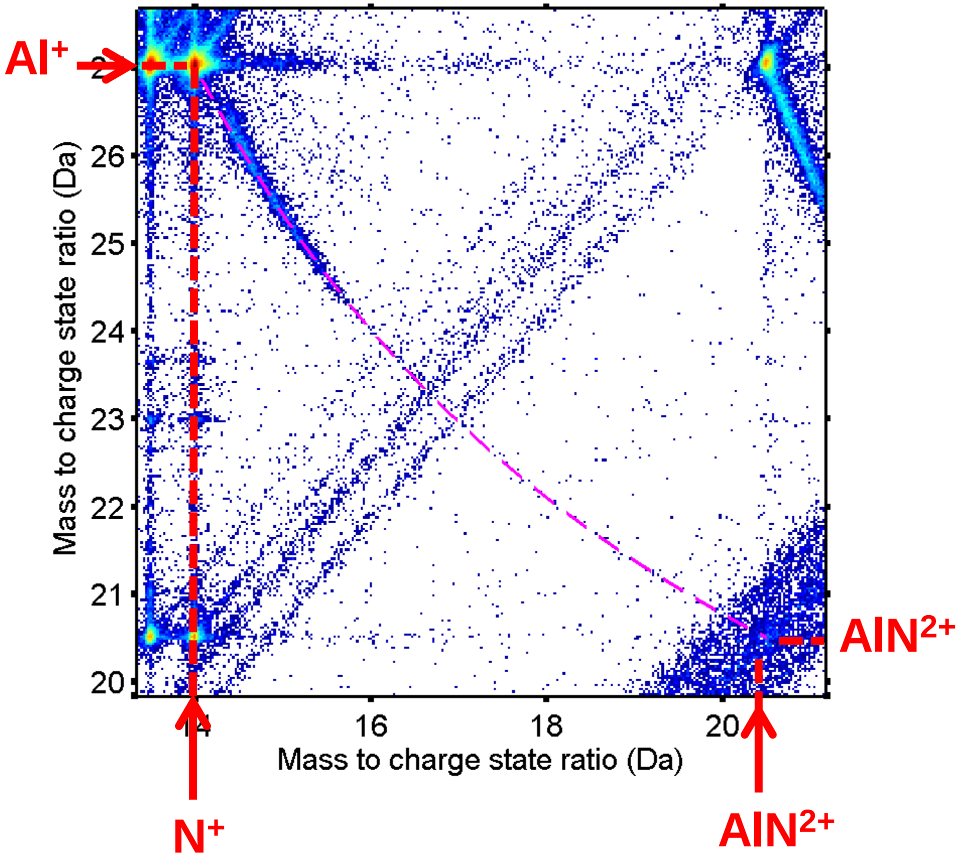


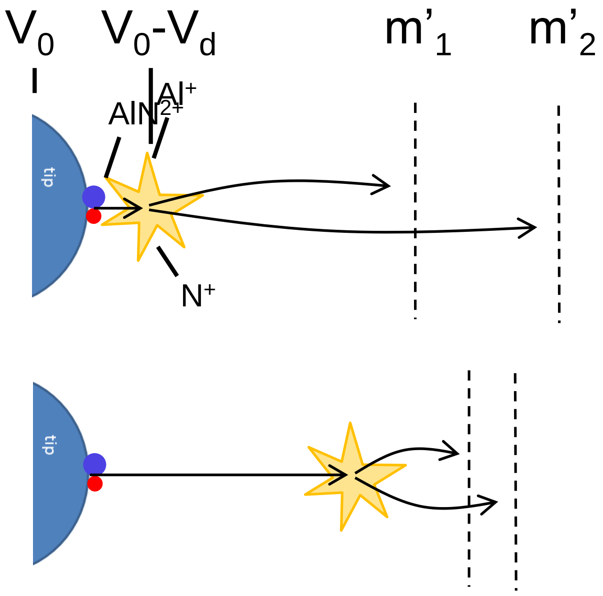
Dissociation of AlN^{2+} ions

Saxey, D. W. (2011). *Ultramicroscopy*, *111*(6), 473–479

Dissociation of molecular ions

Dissociation track : $AlN^{2+} \rightarrow Al^{+} + N^{+}$





$$m_1' = m_1\left[1 - \frac{V_d}{V_{DC}}\left(1 - \frac{m_1}{m_p}\right)\right]^{-1}$$ [1]

- Ion times of flight can be used to measure the dissociation potential

[1] Saxey, D. W. (2011). *Ultramicroscopy*, *111*(6), 473–479

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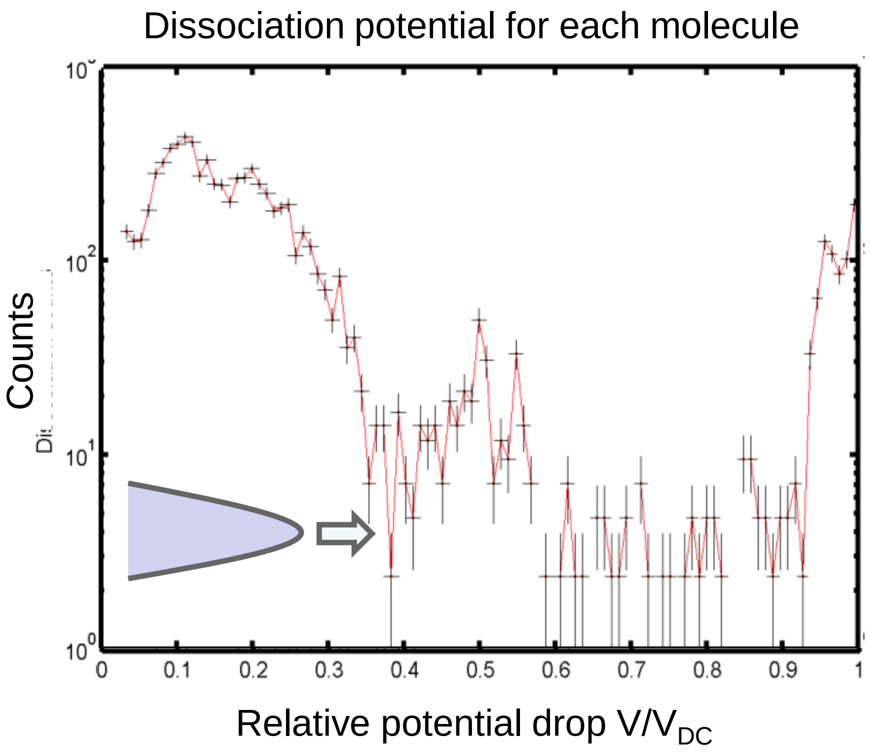
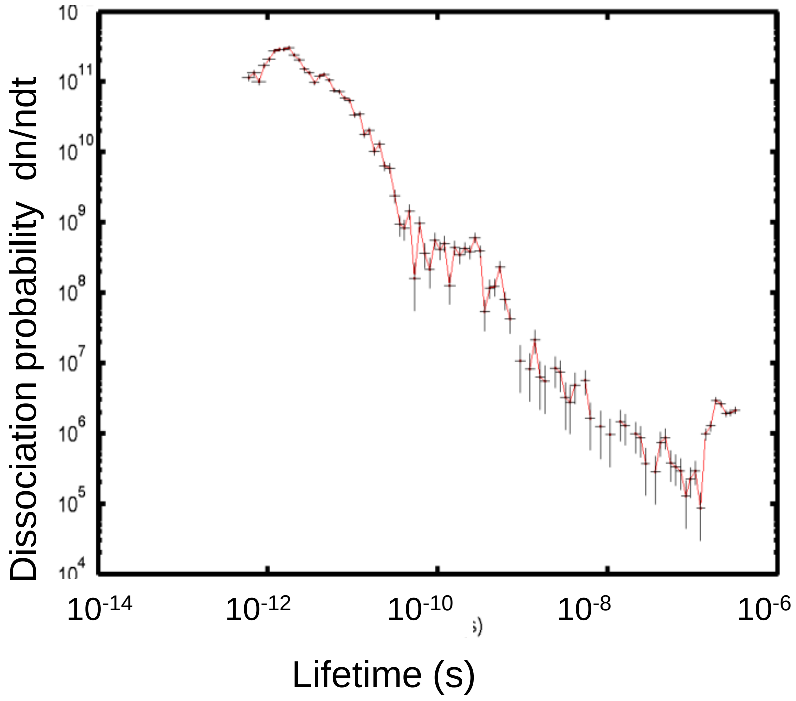
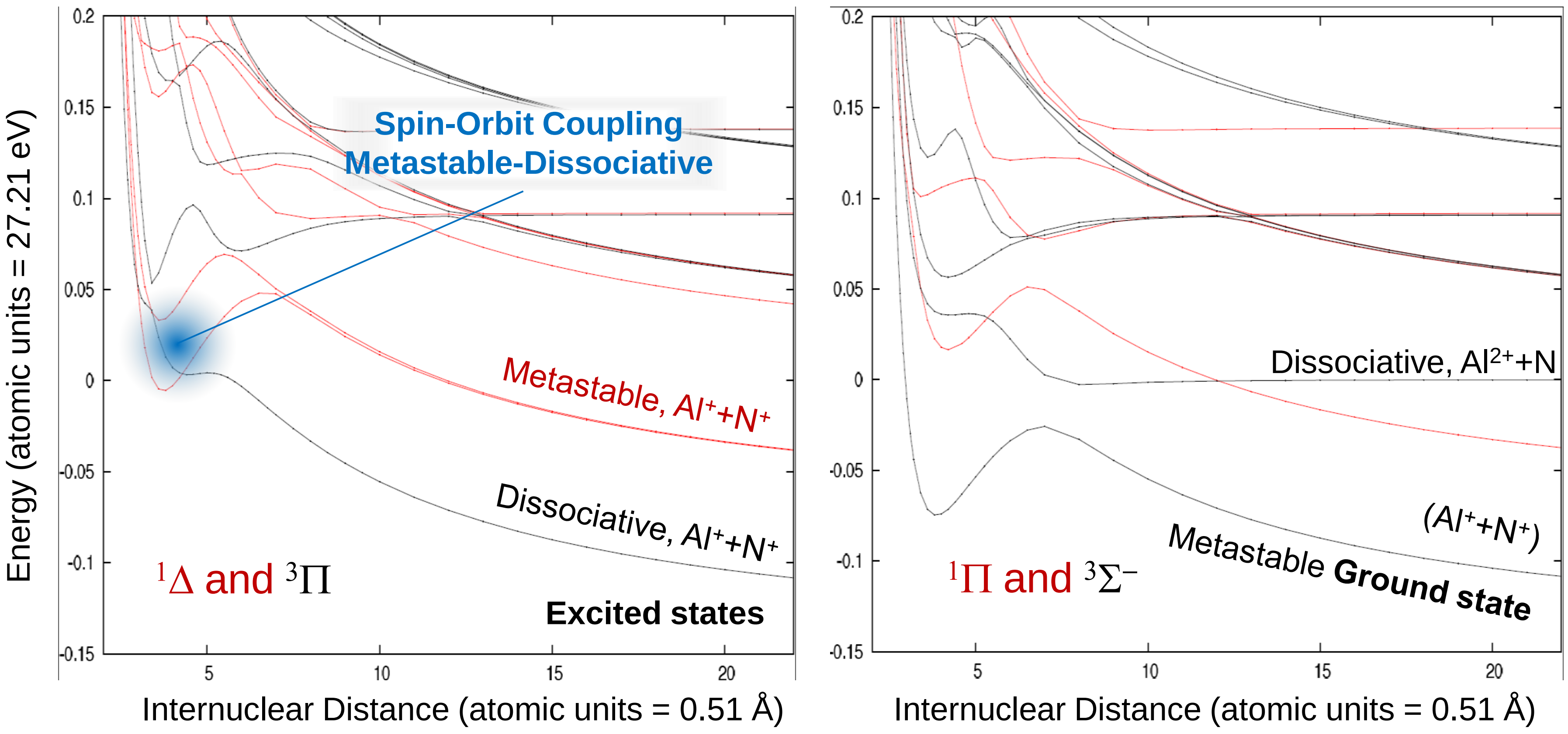
Access to molecule lifetime





- Analytical equation for potential distribution (Paraboloidal model)
- Ion trajectory simulation gives **<u>molecules lifetime</u>**

Calculation of potential energy surfaces [1]



The observed dissociative process $AlN^{2+} \rightarrow Al^{+} + N^{+}$ is possible only if the molecule evaporates in an **excited state**

[1] D. Zanuttini et al. Phys. Rev. A 95, 061401(R) (2017)

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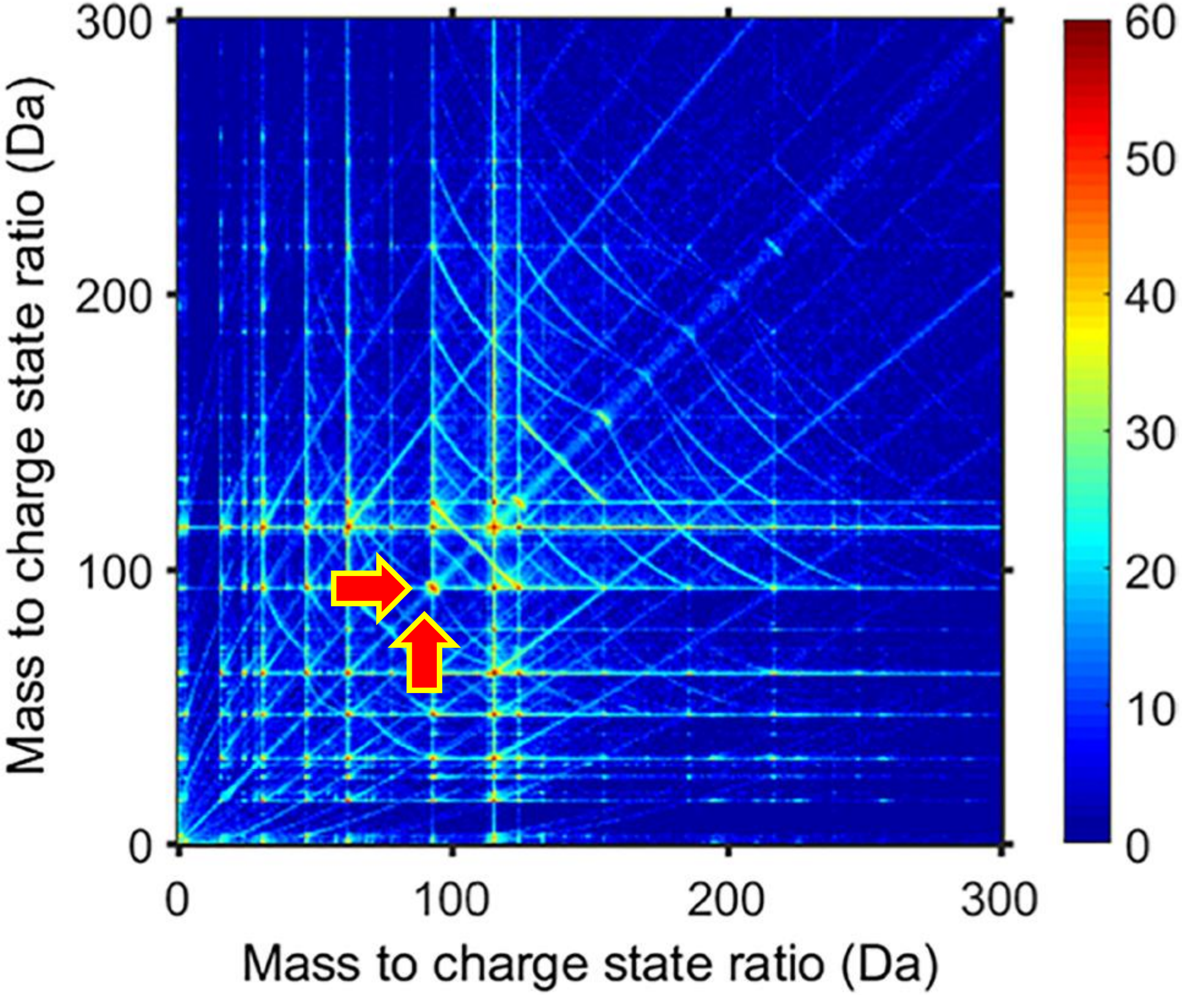
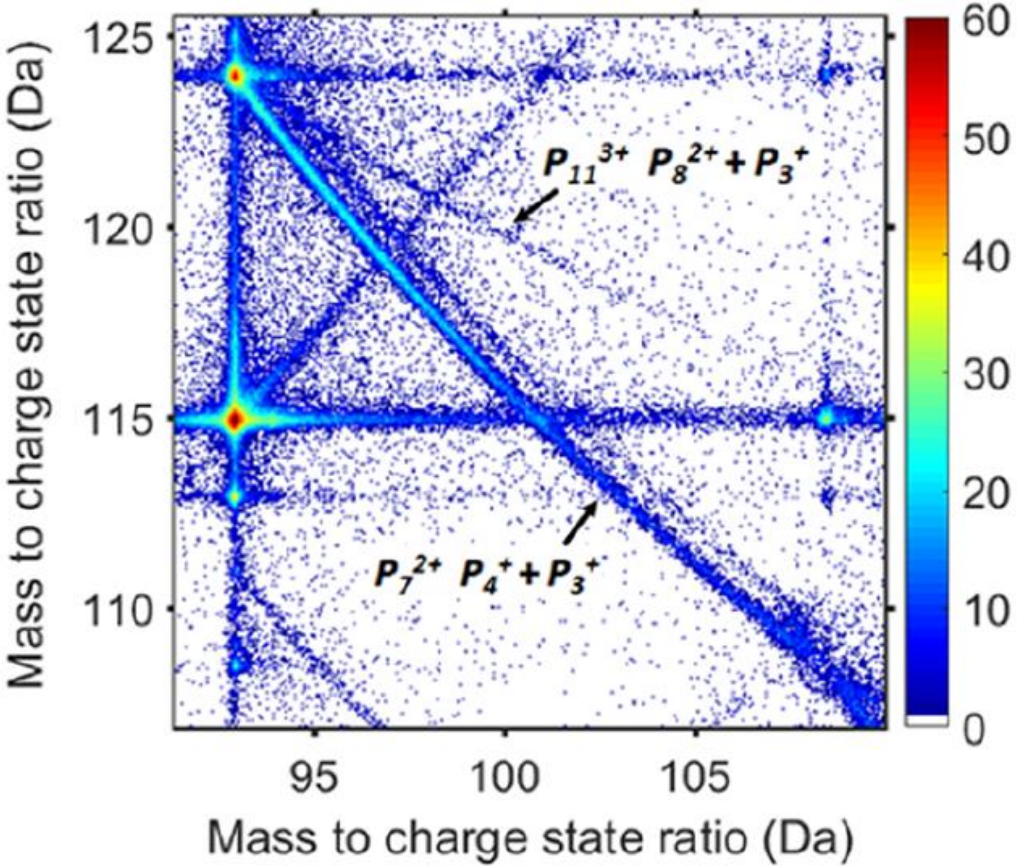
Dissociation "fireworks" in InP





E. DI Russo et al. J. Phys. Chem. A 124, 10977−10988 (2020)

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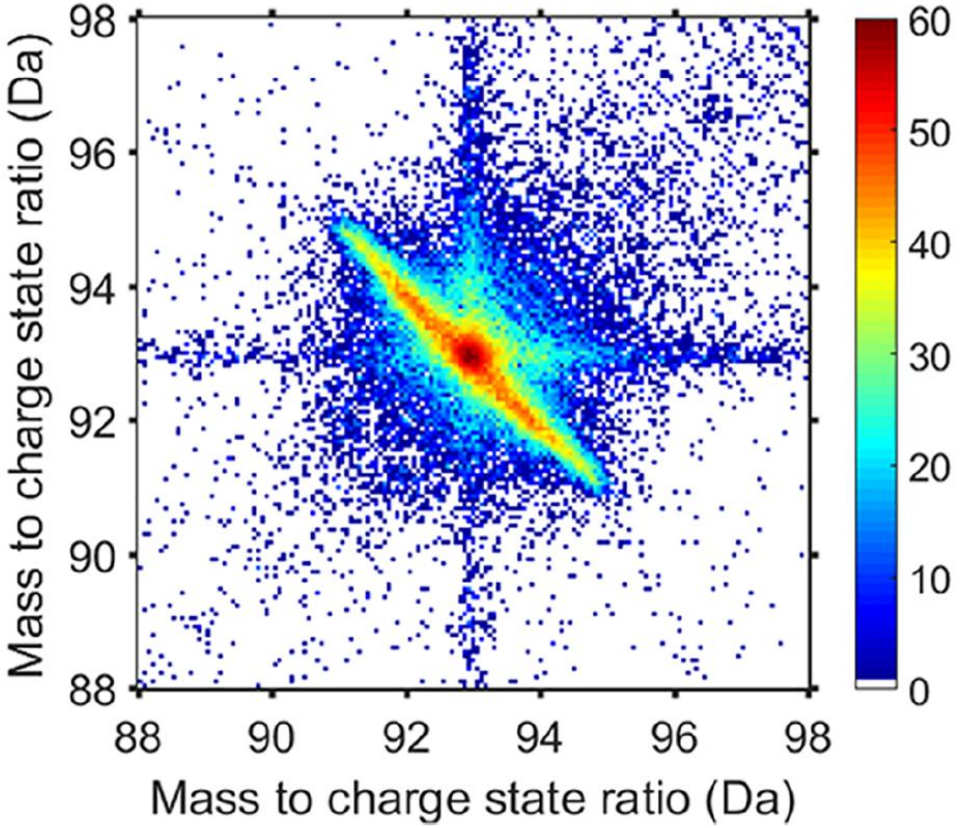
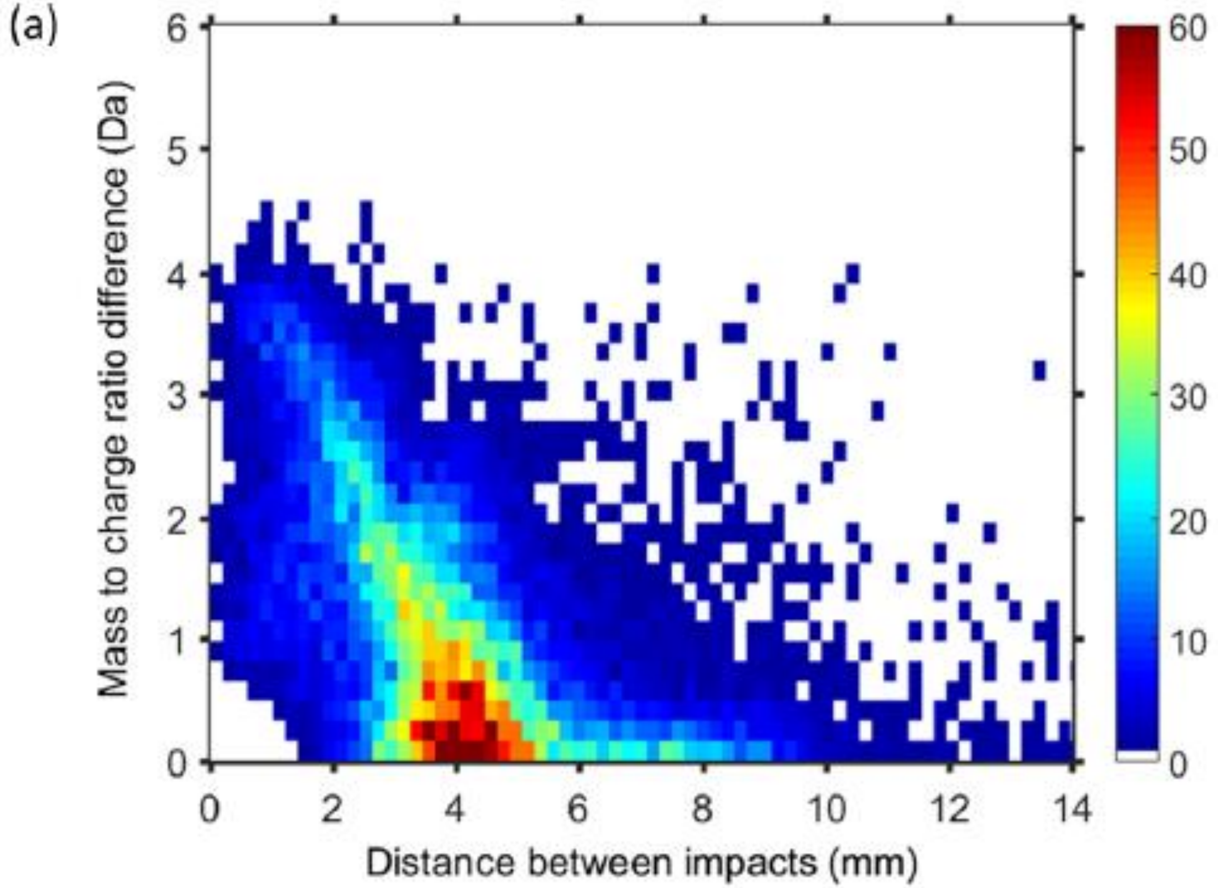
Focus on a "homolytic reaction" $P_6^{2+} \rightarrow P_3^+ + P_3^+$





E. DI Russo et al. J. Phys. Chem. A 124, 10977−10988 (2020)

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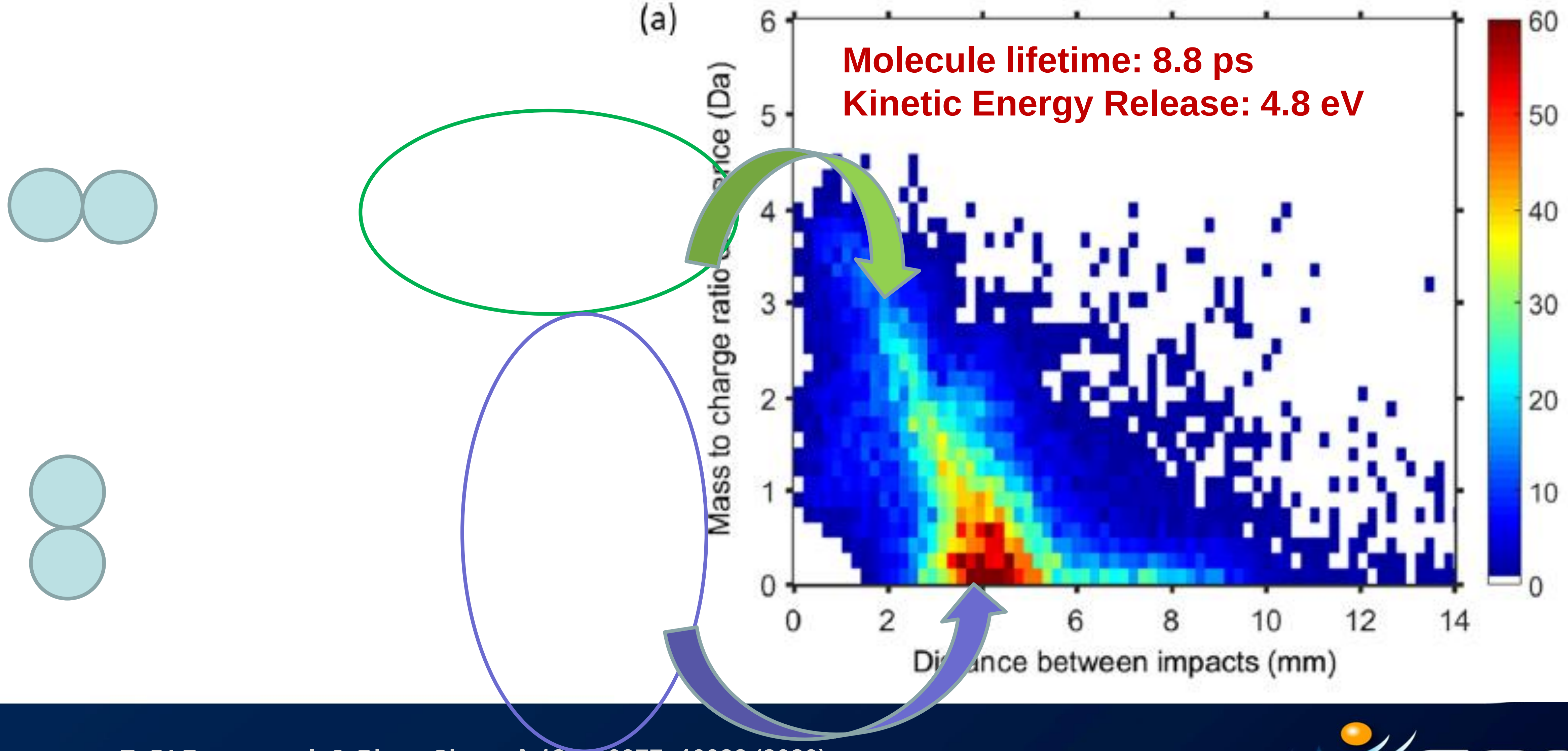

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GPM Groupe de Physique des Matériaux

Towards the strictly correlative study

(sorry I have to choose)

Case 1
InGaN/GaN Quantum Wells

Discover how electrons and holes are trapped at alloy fluctuations and stacking faults… and then produce light

Case 2
GaN/AlN Quantum Dots

Enjoy a 3D trip full of metrological implications into interface fluctuations of nanoscale emitters with ultrapure optical properties

Case 3
ZnO/MgZnO Quantum Wells

One of the morphologically and compositionally weirdest semiconductor heterostructure systems ever grown

Rigutti, Nano Letters (2014)
Mancini, Appl.Phys.Lett. (2016)

Mancini, Nano Letters (2017)
Mancini, Appl. Phys. Lett. (2017)

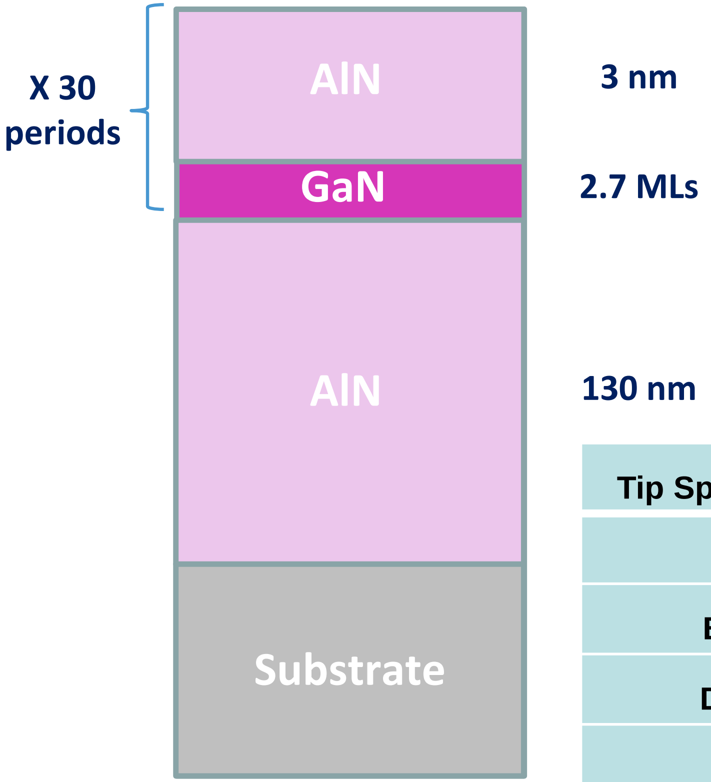
Di Russo, Appl.Phys.Lett. (2017)

Case 2

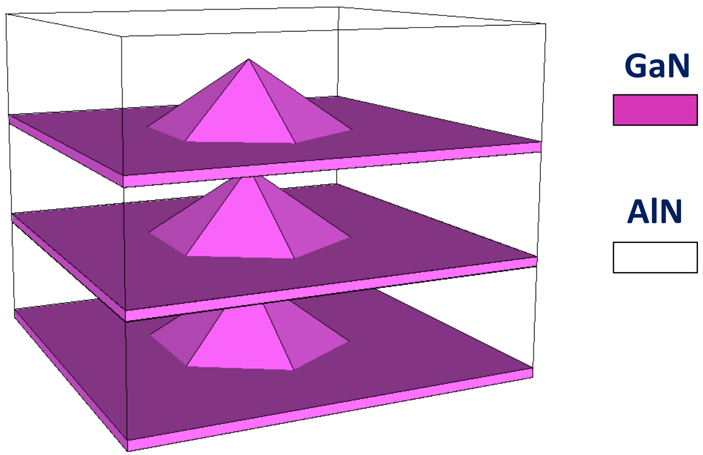
GaN/AlN QUANTUM DOTS

GaN/AlN Quantum Dots

PAMBE grown structure



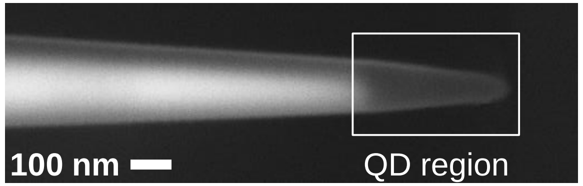
Stranski-Krastanov GaN/AlN QDs [1]

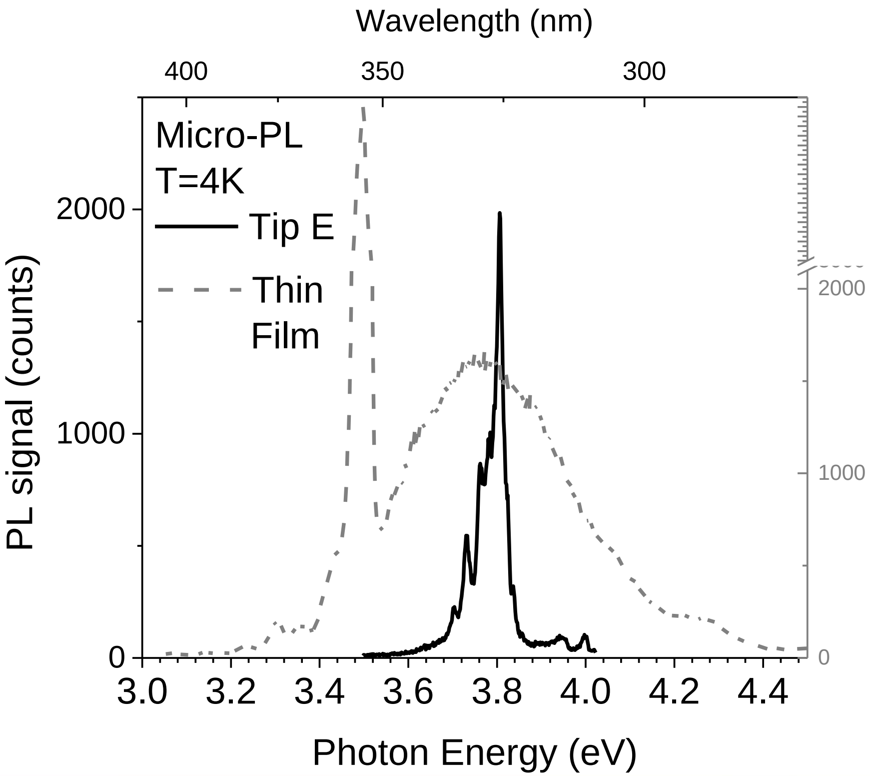


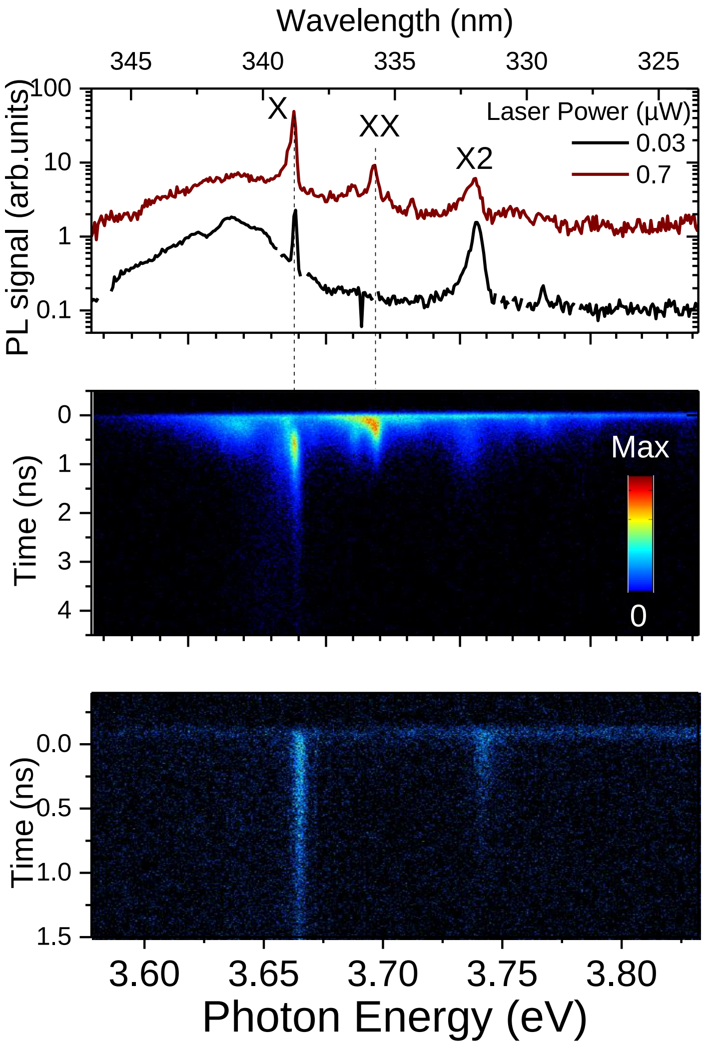
Tip Specimens	µPL	STEM (ET)	APT
A	X		X
B,C			X
D, F	X	X	
E	X	X	X
G-Q (11 tips)	X		

[1] C. Leclere et al, JAP 113 034311 (2013)
[2] B. Daudin et al, PRB 56, (1997)
[3] N. Gogneau, J. Appl. Phys. 94, 2254 (2003);

Optical properties: thin film vs APT tip specimens



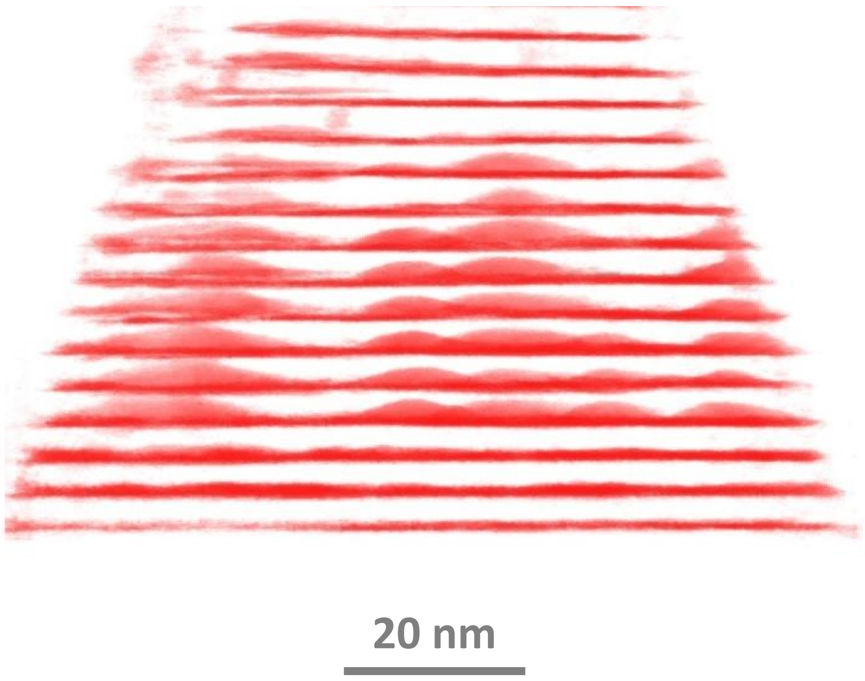


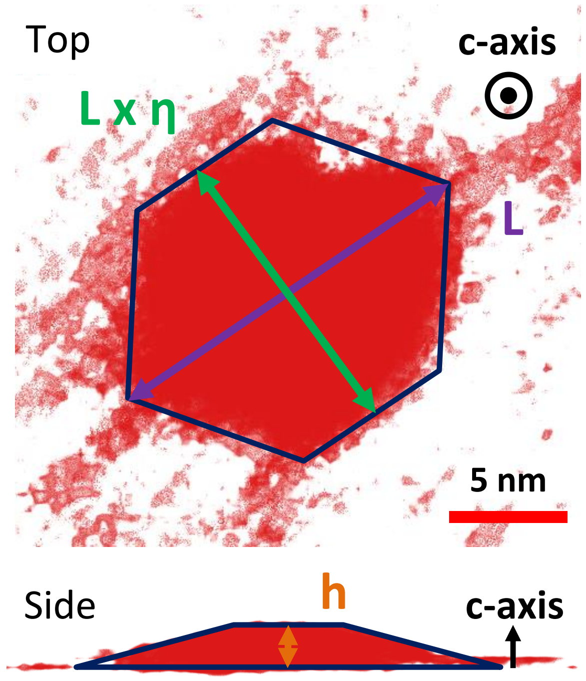


L. Mancini, APL (2017)

Electron Tomography

Tomography from STEM images – Tip E

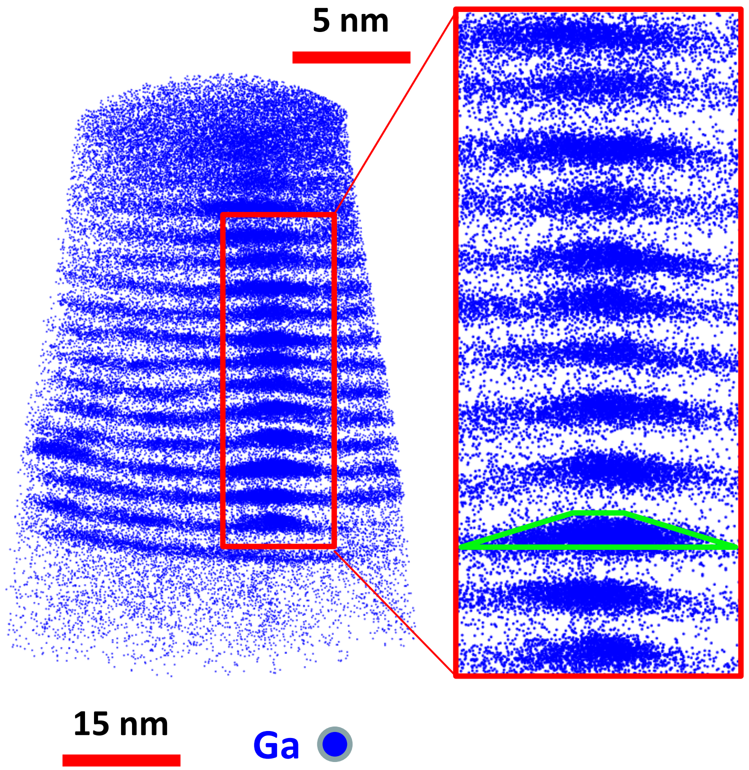




3D size and shape of QDs assessed by fit with hexagonal pyramids

3 geometrical parameters (L,η,h)

Atom Probe Tomography

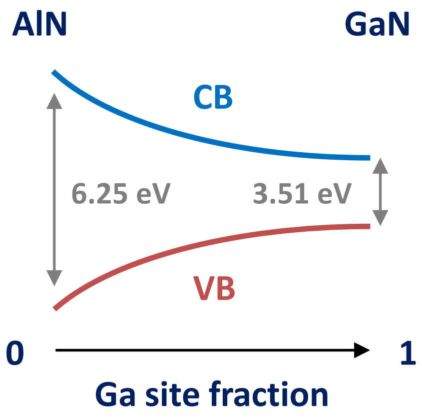


Composition

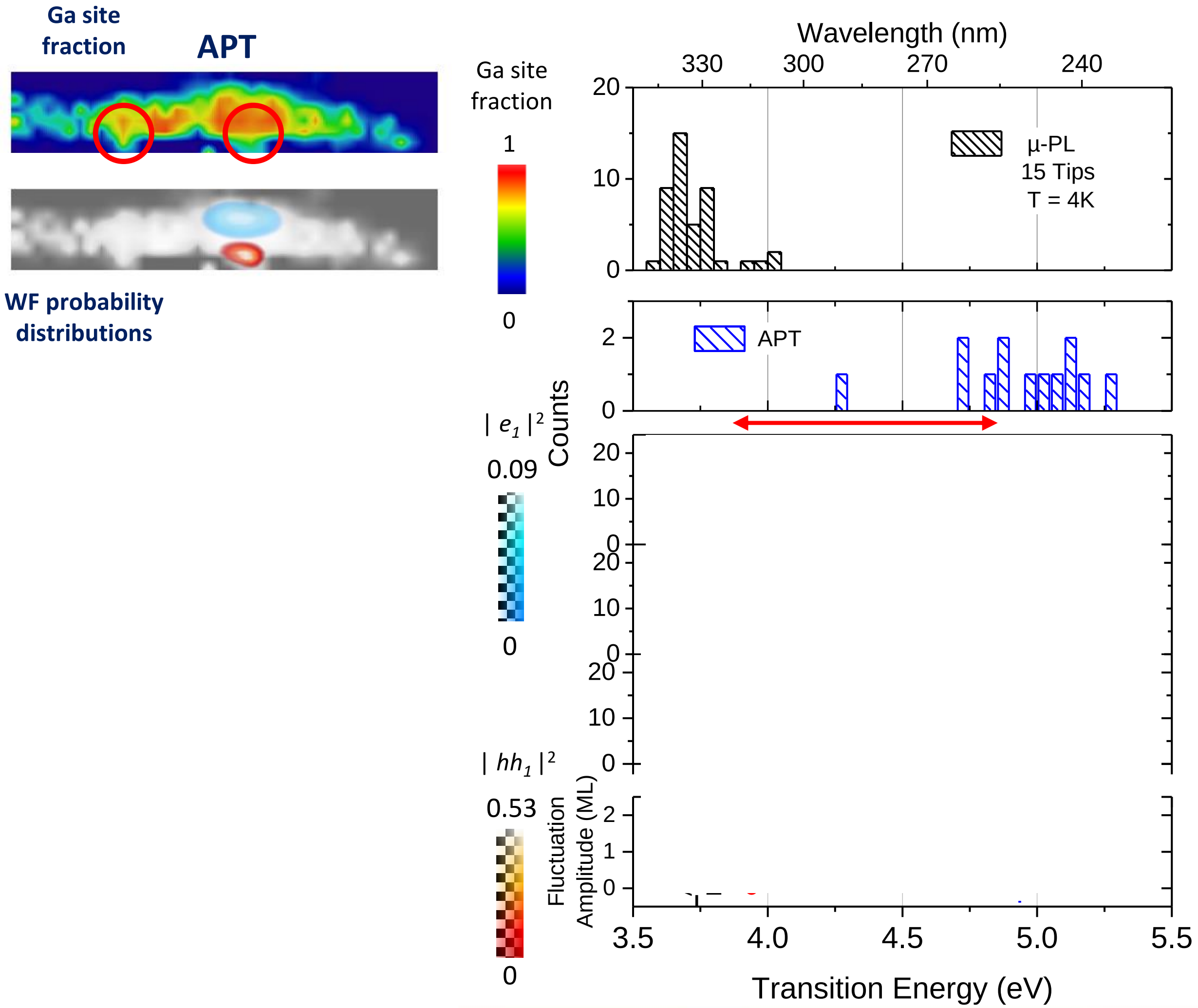
3D Ga site fraction map

AlGaN system:

$$X_{ga} = \frac{N_{Ga}}{N_{Ga} + N_{Al}}$$



Correlation optics-structure (k.p 6x6 model)



Photoluminescence energy computed as:

$$E_{PL} = E_e - E_{hh} - E_{xc} \ [1,2]$$

Energies calculated from APT data are overestimated

↓

Limited APT lateral resolution leads to biased QDs composition

APT reconstructed QDs show monolayer interface fluctuations

↓

Best agreement with PL obtained considering e-tomography reconstructed QDs with added fluctuations

L. Mancini, Nanoletters (2017) (EMS Outstanding paper)