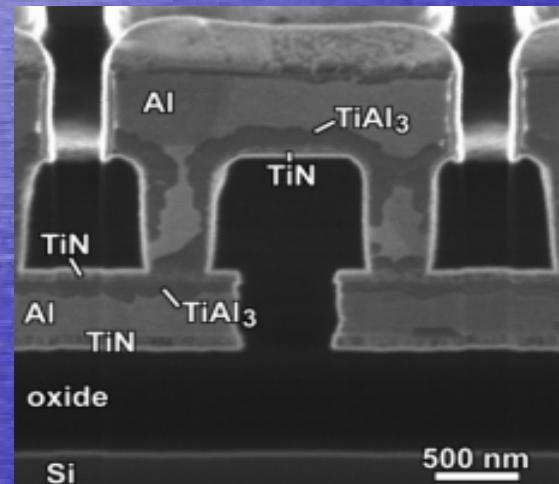
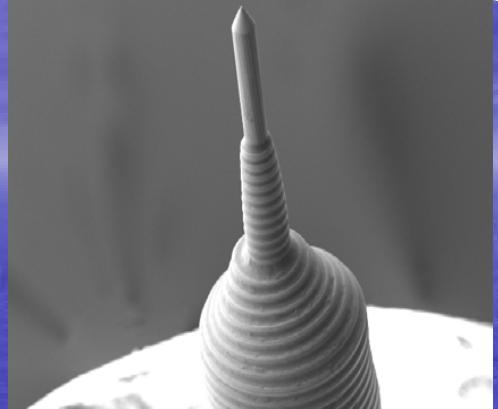


# OUTLINE

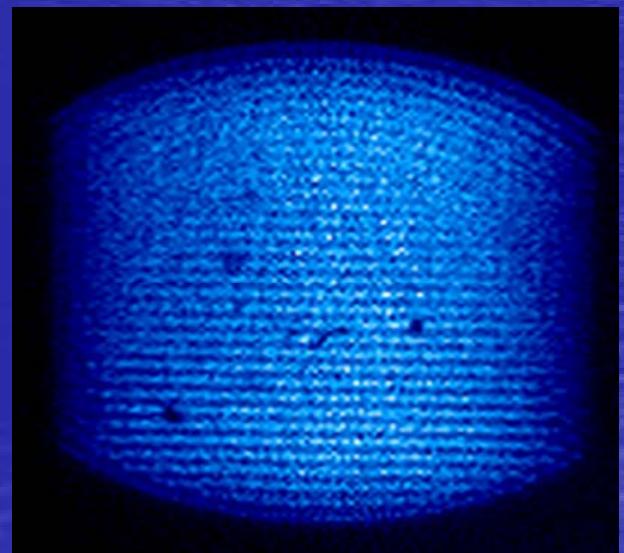


⇒ History of LMIS / FIB

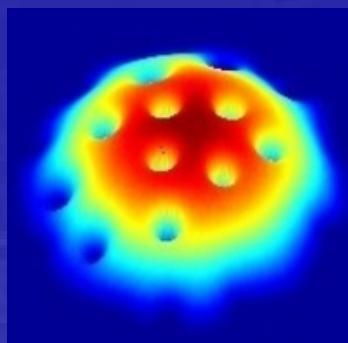
⇒ LMIS principles



⇒ F.I.B. technology



⇒ Future of FIB



# *Early observations*

## **History of Taylor cones**

Gilbert (1600)  
fluid under high tension forms a cone

Zeleny (1914)  
Observed and filmed cones and jets

Taylor (1964)  
exactly conical solution to equations of  
Electro Hydro Dynamics (EHD)



Gilbert was the scientist (and probably lover) of Queen Elisabeth I, she was fond of physical phenomena.

**From Joakim Reuteler  
ETH - DMATL**

# THE PHYSICAL REVIEW.

THE ELECTRICAL DISCHARGE FROM LIQUID POINTS, AND  
A HYDROSTATIC METHOD OF MEASURING THE  
ELECTRIC INTENSITY AT THEIR SURFACES.<sup>1</sup>

BY JOHN ZELENY.

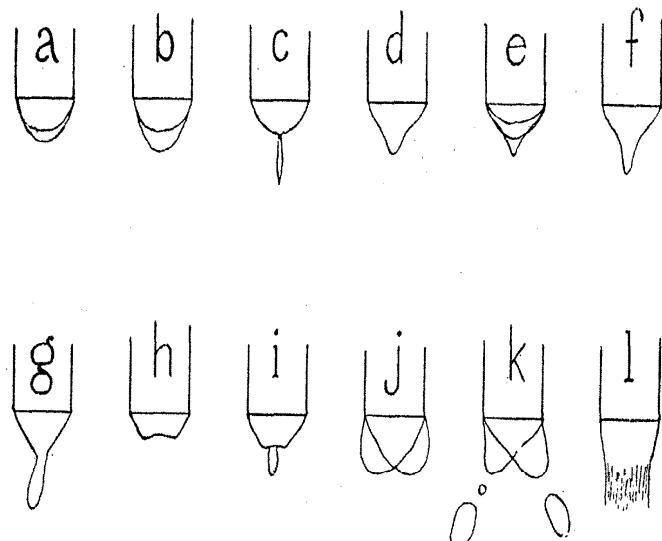


Fig. 3.

Oscillations of meniscus during intermittent discharge.

# Early work

**Zeleny 1914:** he took excellent ultra fast pictures of TC moving the photo plaque with a rubber band! Inventor of Zeleny Electrooscope (University of Minnesota)

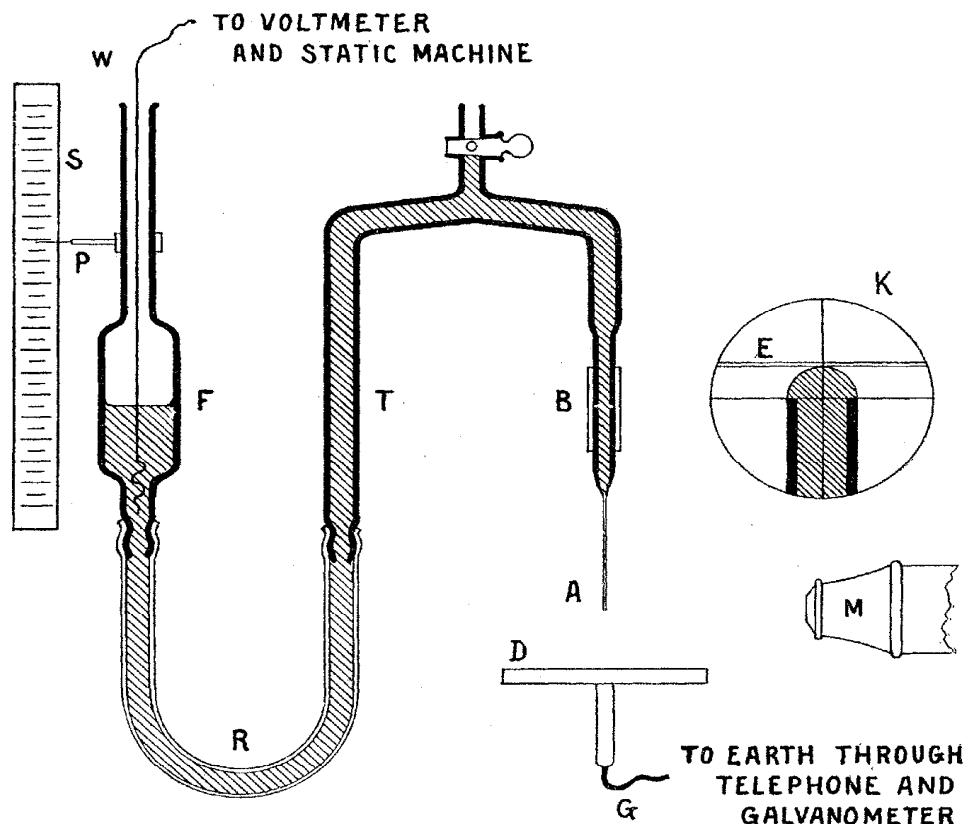
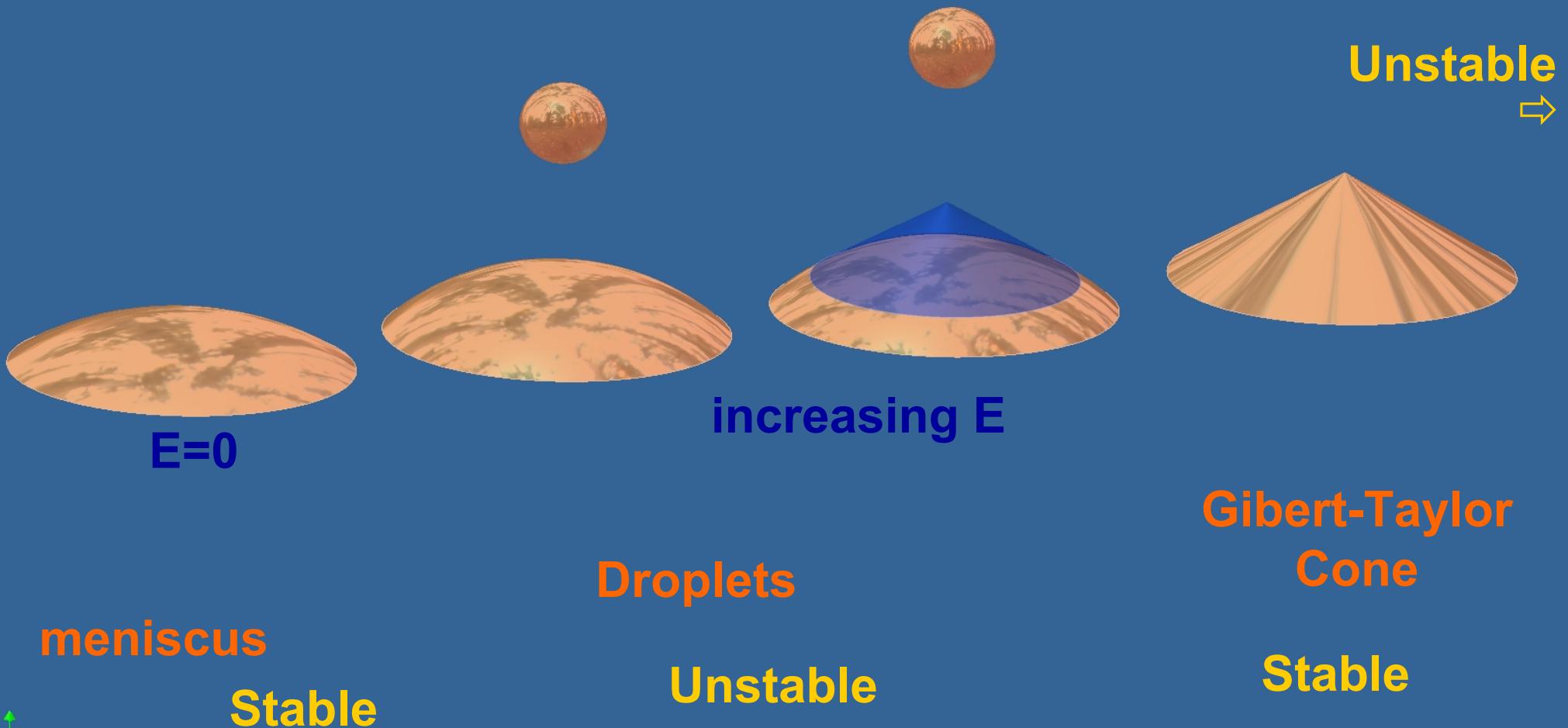


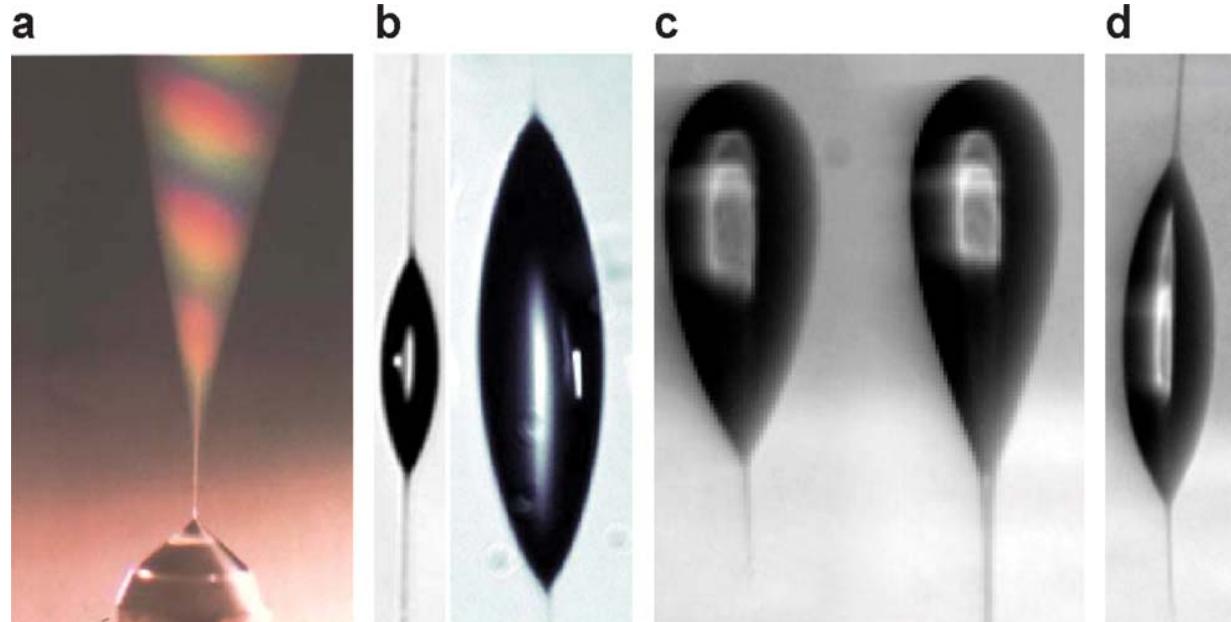
Fig. 1.  
Diagram of apparatus.

## ⇒ Interaction Electric field - Liquid surface

Electrostatic energy and surface tension energy



# History of LMIS



**AR** Fernández de la Mora J. 2007.  
Annu. Rev. Fluid Mech. 39:217–43

**a: TC on capillary**

**b: ethylene-glycol Rayleigh explosion no external field**

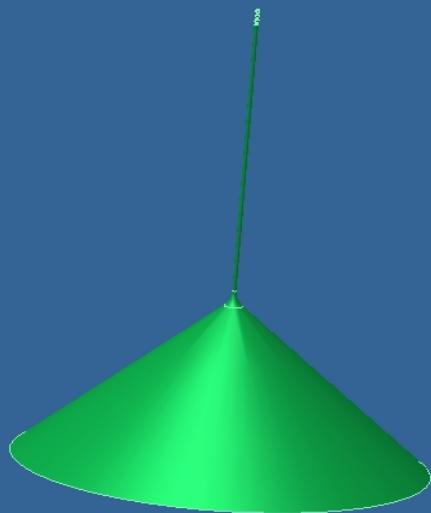
**c: idem plus external field**

**d: no charge only external field**

**Role in thunderstorms formation!**

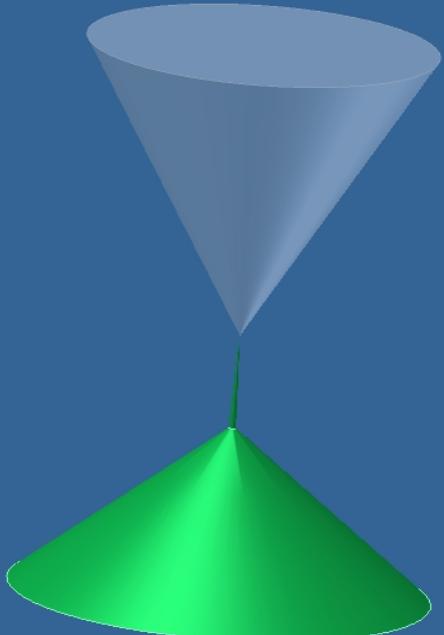
# *History of LMIS*

insulators



(Printers)

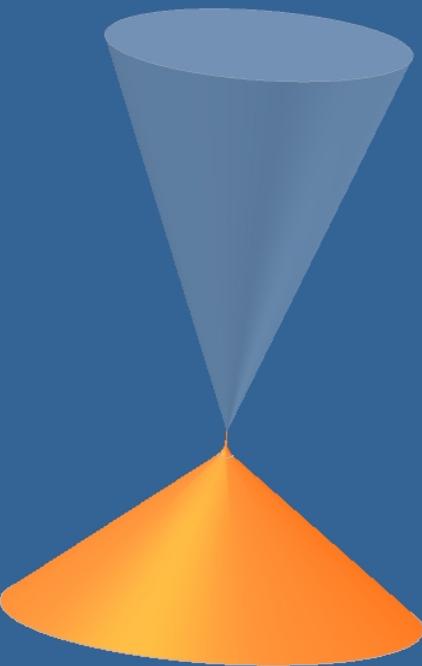
Doped insulators



Electro-spray  
MS

Metals  
electrolytes

Space  
propulsion



LMIS and FIB

## **Some pionners of LMIS & FIB**

Mahoney (1969)

Krohn Ringo (University of Chicago) 1975

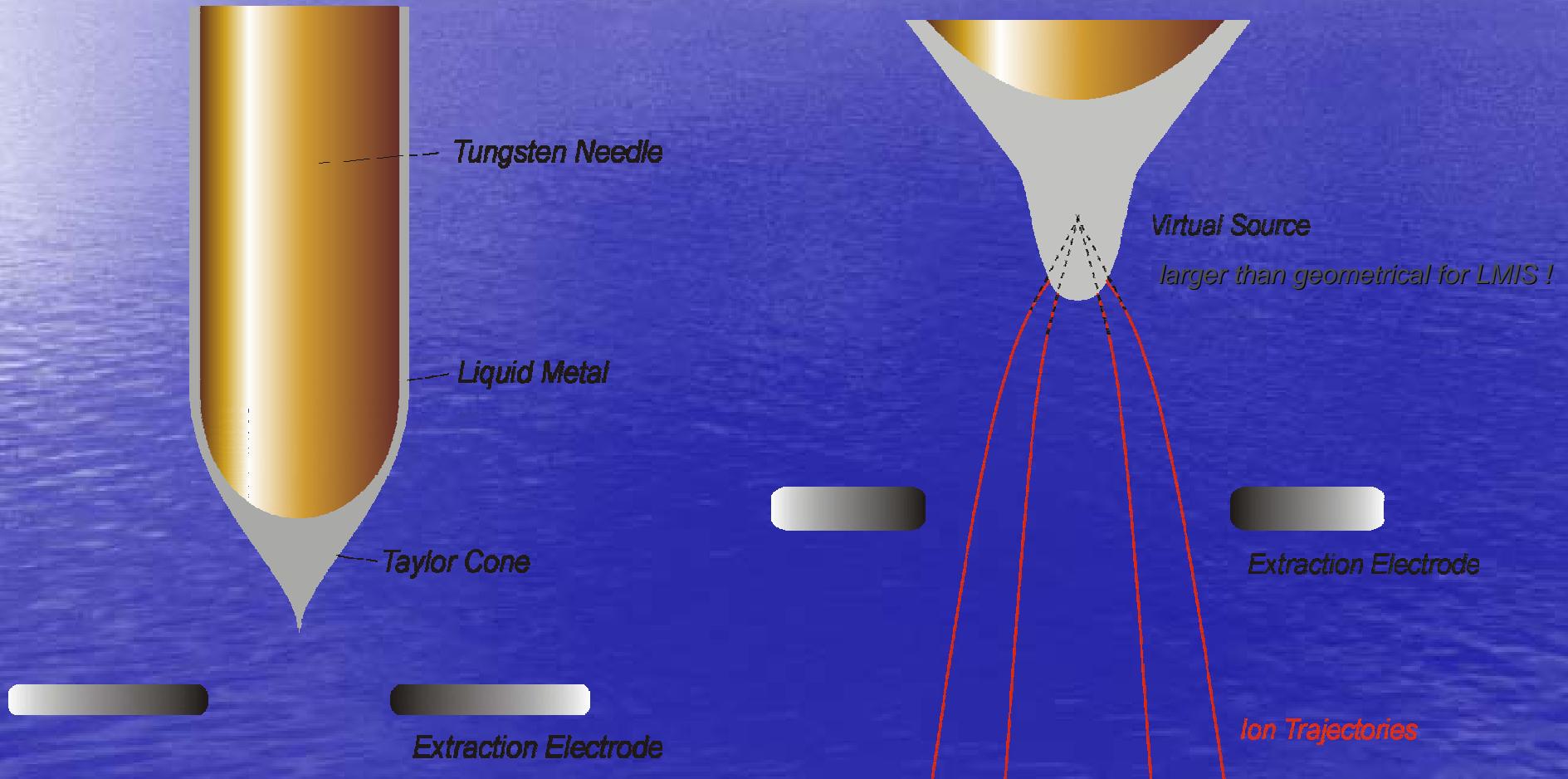
Sudraud et al Paris XI Orsay (1974)

University of Oxford Mair (1980)

Culham UK, Roy Clampitt Prewett (1980)

Oregon Graduate Center L. Swanson (1980)

## The L.M.I.S. (Liquid Metal Ion Source)

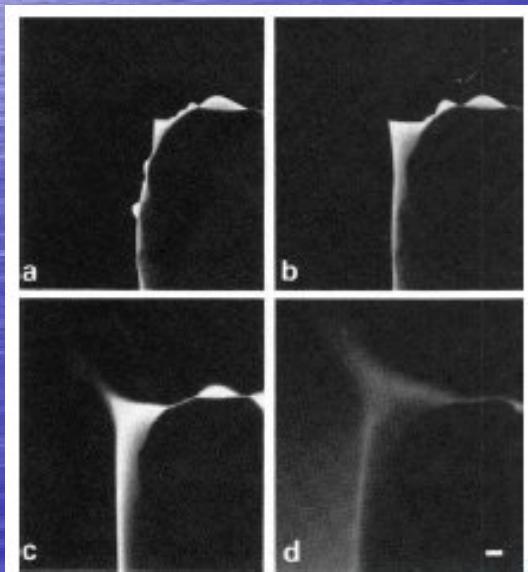
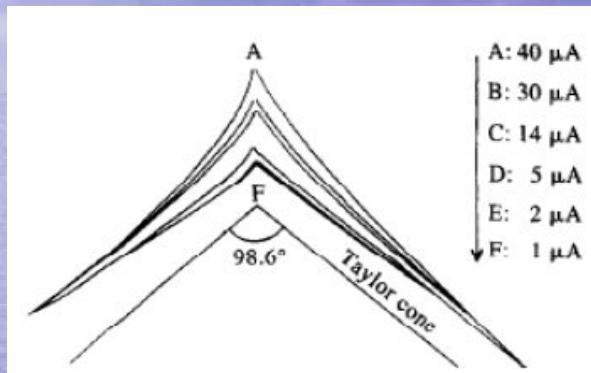


## LMIS concept

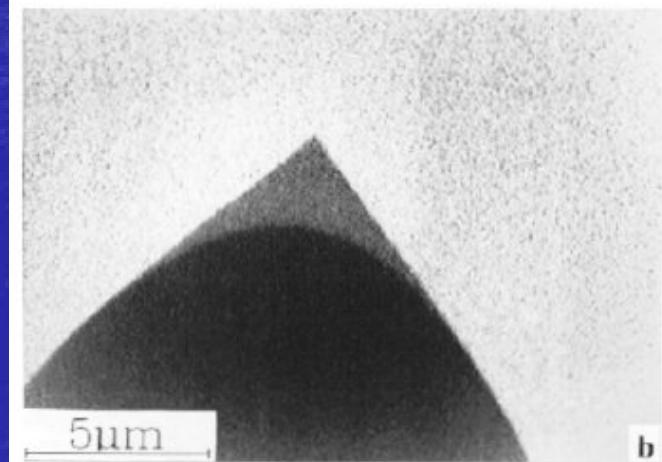
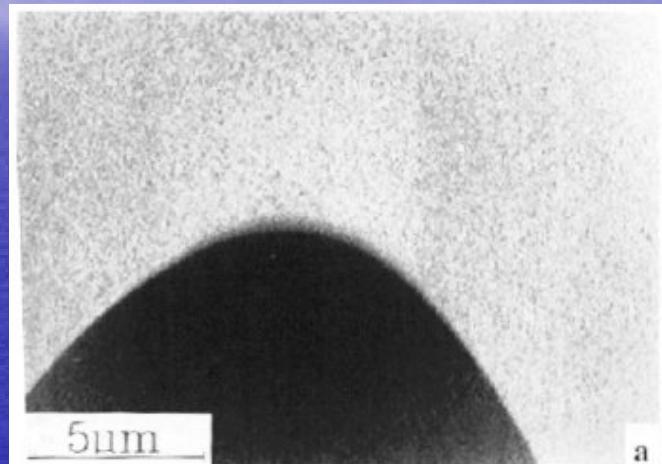
- ⇒ Substrate : Refractory, Wetting, insoluble, tip and reservoir
- ⇒ Temperature : Heater,  $T^\circ$  higher than  $M_p$
- ⇒ Liquid metal : low  $M_p$ , low vapor P, oxyde VP > metal VP
- ⇒ Tip : cone angle, apex radius
- ⇒ Liquid flow : surface compatibility
- ⇒ Reduced energy dispersion

*Gallium is a very good element !*

## L.M.I.S. (Liquid Metal Ion Source)

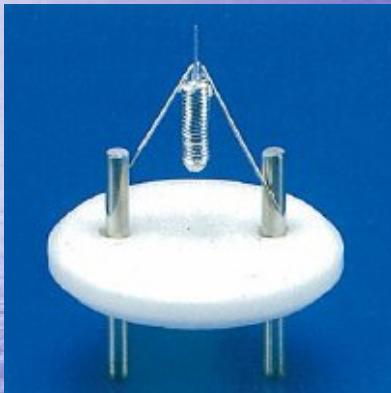


BenAssayag et al. – LPS Orsay  
3 MeV TEM (1985)



Diesel et al. – MPI Halle  
1 MeV TEM (1996)

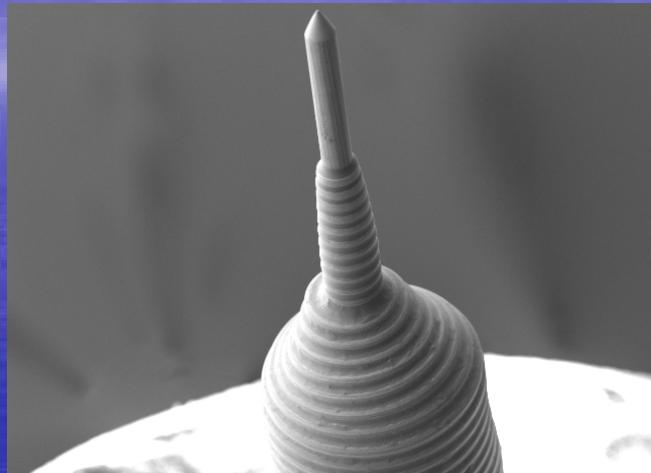
## L.M.I.S. and L.M.A.I.S.



Denka Ga LMIS

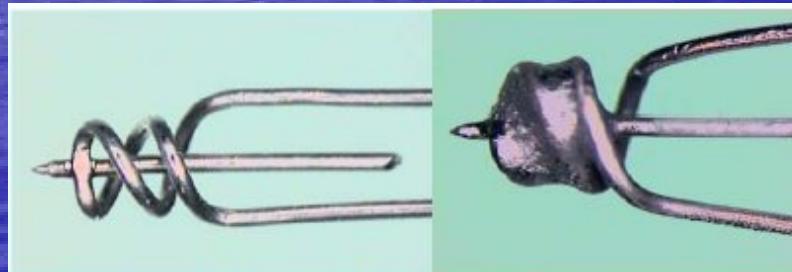


J.Gierak – LPN (F)



Orsay Physics Ga LMIS

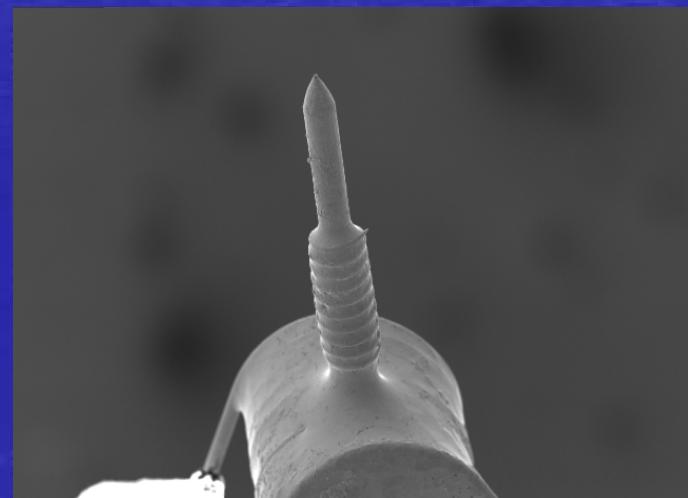
## Some geometries



L.Bischoff – FZR Dresden (D)

Alloy:  $\text{Co}_{36}\text{Nd}_{64}$

$T_m = 566^\circ\text{C}$



Orsay Physics alloy LMIS



NANOSOLUTIONS  
by  
**ORSAY PHYSICS**

# *LMIS Technology*

# Available Ion Species for Focused Ion Beam Implantation at Chair of Applied Solid State Physics of Ruhr-University of Bochum

The diagram illustrates the periodic table with a focus on Magnesium (Mg). It shows the element's symbol (Mg), atomic number (12), mass number (24.305), and its three naturally occurring isotopes:  $^{24}\text{Mg}$ ,  $^{25}\text{Mg}$ , and  $^{26}\text{Mg}$ . The natural abundance of each isotope is indicated as 78.9, 11.0, and 10.1 respectively. The atomic mass is given as 24.305. A green asterisk is placed over the Mg box.

contact:

**Prof. Dr. Andreas D. Wieck**  
Lehrstuhl für Angewandte  
Festkörperphysik  
Ruhr-Universität Bochum  
Universitätsstraße 150  
D-44780 Bochum  
Germany  
[www.rub.de/afp/](http://www.rub.de/afp/)  
Andreas.Wieck@rub.de

Lanthanides	Landolt 138.97 2 La 57	Cerium 140.12 4 Ce 58	Praseodymium 1 Pr 40.98	Nd 140.98 7 Nd 89	Promethium 15 Pm 145	Bismuth 180.00 7 Sm 103	Europium 151.96 2 Eu 83	Gadolinium 157.20 1 Gd 88	Tellurium 130.00 7 Tb 152	Dysprosium 160.00 7 Dy 100	Holmium 164.93 1 Ho 67	Erbium 167.26 2 Er 88	Thulium 169.93 1 Tm 119	Ytterbium 173.04 7 Yb 73
	150 156 2.1 9.6	159 160 162 3.3 86.4 17.1	161	142 144 145 25.1 23.8 17.2	147 152 154 16.5 26.7 32.7	151 153 16.8 32.2	156 158 160 23.9 24.8 21.9	155 19.0	162 151 154 20.0 24.9 26.2	165 17.0	165 17.0	166 167 166 22.8 23.9 27.0	168 17.0	170 179 27.0 16.1 31.8
	155	157.1	158	159	154	155	156	155	155	155	155	155	155	155
	159	157.1	158	159	154	155	156	155	155	155	155	155	155	155
Actinides	Athrium 227 2 Ac 88	Thorium 230.04 2 Th 90	Protactinium 231 2 Pa 81	Uranium 238.03 2 U 84	Nepthrium 237.08 2 Np 93	Rutherfordium 261 2 Pu 94	Amerium 243 2 Am 90	Curium 247 2 Cm 96	Berkelium 247 2 Bk 97	Cf 251 2 Cf 98	Fermium 254 2 Es 99	Promium 257 2 Fm 100	Mendelevium 256 2 Md 101	No 259 2 No 102

# LMIS Technology

## Non-exhaustive list of LMIS and LMAIS

Prof. A. Wieck - RUB

Other alloys, such as

AuSi, AuGe, GaBi, GaBiLi,

AsPdB, CoNd, MgGa,

GePd (Mühle et al, ETH)

etc...

are also available.

	Type of LMIS	Most intensive fraction of ions
1	AgGe	Ge <sup>++</sup> , Ge <sup>+</sup> , Ag <sup>+</sup>
2	AgAuGe	Ge <sup>++</sup> , Ge <sup>+</sup> , Ag <sup>+</sup> (Au <sup>++</sup> ), Au <sup>+</sup> , AuGe <sup>+</sup>
3	AuBeSi	Be <sup>++</sup> , Be <sup>+</sup> , Si <sup>++</sup> , Si <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
4	AuBGeNi	Ni <sup>++</sup> , Ni <sup>+</sup> , Ge <sup>++</sup> , Ge <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , AuGe <sup>+</sup> , Au <sub>2</sub> Ge <sup>+</sup>
5	AuCeSi	Si <sup>++</sup> , Si <sup>+</sup> , Ce <sup>++</sup> , Ce <sup>+</sup> , Ce <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
6	AuCoGe	Co <sup>++</sup> , Co <sup>+</sup> , Ge <sup>++</sup> , Ge <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , AuGe <sup>+</sup> , Au <sub>2</sub> Ge <sup>+</sup>
7	AuCrGe	Cr <sup>++</sup> , Cr <sup>+</sup> , Ge <sup>++</sup> , Ge <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , AuGe <sup>+</sup> , Au <sub>2</sub> Ge <sup>+</sup>
8	AuDyGe	Ge <sup>++</sup> , Ge <sup>+</sup> , Dy <sup>++</sup> , Dy <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , AuGe <sup>+</sup>
9	AuDySi	Si <sup>++</sup> , Si <sup>+</sup> , Dy <sup>++</sup> , Dy <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>3</sub> <sup>++</sup> , Au <sub>2</sub> <sup>+</sup>
10	AuErSi	Si <sup>++</sup> , Si <sup>+</sup> , Er <sup>++</sup> , Er <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
11	AuEuSi	Si <sup>++</sup> , Si <sup>+</sup> , Eu <sup>++</sup> , Eu <sup>+</sup> , Au <sup>++</sup> , Au <sub>2</sub> <sup>+</sup>
12	AuFeGe	Fe <sup>++</sup> , Fe <sup>+</sup> , Ge <sup>++</sup> , Ge <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , AuGe <sup>+</sup> , Au <sub>2</sub> Ge <sup>+</sup>
13	AuGdSi	Si <sup>++</sup> , Si <sup>+</sup> , Gd <sup>++</sup> , Gd <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
14	AuGeMn	Mn <sup>++</sup> , Mn <sup>+</sup> , Ge <sup>++</sup> , Ge <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , AuGe <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , Au <sub>2</sub> Ge <sup>+</sup>
15	AuGeNi	Ni <sup>++</sup> , Ni <sup>+</sup> , Ge <sup>++</sup> , Ge <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , AuGe <sup>+</sup> , Au <sub>2</sub> Ge <sup>+</sup>
16	AuGeV	V <sup>++</sup> , Ge <sup>++</sup> , Ge <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup> , AuGe <sup>+</sup> , Au <sub>2</sub> Ge <sup>+</sup>
17	AuHoSi	Si <sup>++</sup> , Si <sup>+</sup> , Ho <sup>++</sup> , Ho <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
18	AuLaSi	Si <sup>++</sup> , Si <sup>+</sup> , La <sup>++</sup> , La <sup>+</sup> , La <sup>++</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
19	AuNdSi	Si <sup>++</sup> , Si <sup>+</sup> , Nd <sup>++</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
20	AuSbSi	
21	AuSiSm	Si <sup>++</sup> , Si <sup>+</sup> , Sm <sup>++</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
22	AuSiTb	Si <sup>++</sup> , Si <sup>+</sup> , Tb <sup>++</sup> , Tb <sup>+</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
23	AuSiTm	Si <sup>++</sup> , Si <sup>+</sup> , Tm <sup>++</sup> , Au <sup>++</sup> , Au <sup>+</sup> , Au <sub>2</sub> <sup>+</sup>
24	BPt	B <sup>++</sup> , B <sup>+</sup> , Pt <sup>++</sup> , Pt <sup>+</sup>
25	Bi	Bi <sup>++</sup> , Bi <sup>+</sup> , Bi <sub>3</sub> <sup>++</sup> , Bi <sub>2</sub> <sup>+</sup> , Bi <sub>5</sub> <sup>++</sup> , Bi <sub>3</sub> <sup>+</sup> , Bi <sub>4</sub> <sup>+</sup> , Bi <sub>5</sub> <sup>+</sup>
26	BiGaIn	Ga <sup>+</sup> , Bi <sup>+</sup> , In <sup>+</sup>
27	CoDy	Co <sup>++</sup> , Co <sup>+</sup> , Dy <sup>++</sup> , Dy <sup>+</sup>
28	CuP	P <sup>+</sup> (Cu <sup>++</sup> ), Cu <sup>+</sup>
29	CuPPt	P <sup>++</sup> , P <sup>+</sup> (Cu <sup>++</sup> ), Cu <sup>+</sup> , Pt <sup>++</sup> , PtP <sup>++</sup> , PtP <sup>+</sup> , Pt <sup>+</sup>
30	DyNi	Ni <sup>++</sup> , Ni <sup>+</sup> , Dy <sup>++</sup> , Dy <sup>+</sup>
31	Ga	Ga <sup>+</sup>
32	GaIn	Ga <sup>+</sup> , In <sup>+</sup>
33	GaZn	
34	GeNiTi	Ti <sup>++</sup> , Ni <sup>++</sup> , Ge <sup>++</sup> , Ti <sup>+</sup> , Ni <sup>+</sup> , Ge <sup>+</sup>
35	HoNi	Ni <sup>++</sup> , Ni <sup>+</sup> , Ho <sup>++</sup>
36	In	In <sup>+</sup>
37	Sn	Sn <sup>++</sup> , Sn <sup>+</sup> , Sn <sub>2</sub> <sup>+</sup> , Sn <sub>3</sub> <sup>+</sup> , Sn <sub>4</sub> <sup>+</sup> , Sn <sub>5</sub> <sup>+</sup>

## Probe formation

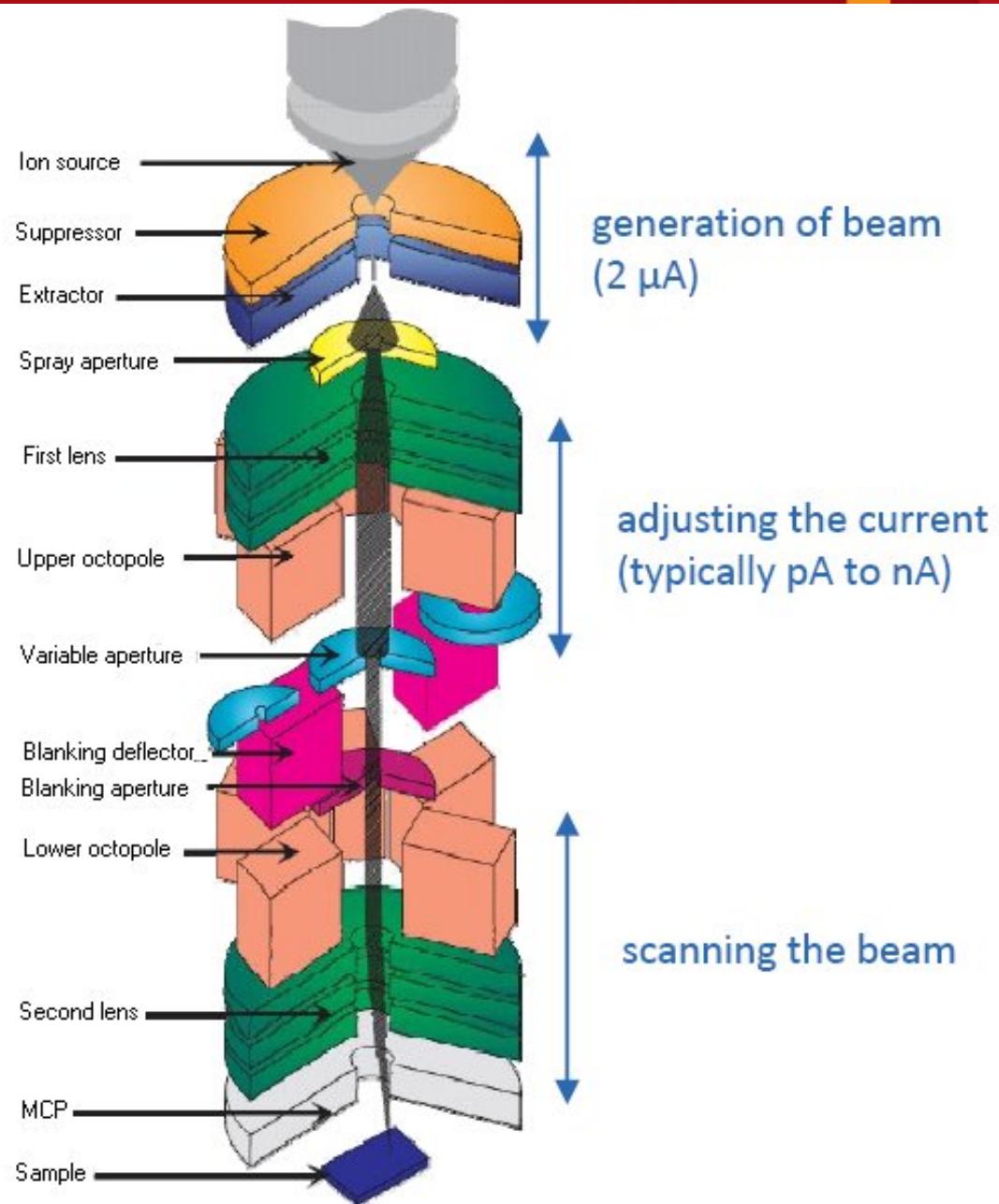
- ⇒ Minimize radius
- ⇒ Maximize current
- ⇒ Optimize I distribution
- ⇒ Beam purity

## Ion optics

- ⇒ Minimize lenses aberrations
- ⇒ Chromatic linked with source  $\Delta E$
- ⇒ Spherical (large I)
- ⇒ Coma, alignment

## Environment optimization

- ⇒ Electric and magnetic noises
- ⇒ Vibrations, acoustic noise
- ⇒ Thermal shifts (long working times)
- ⇒ Software automation



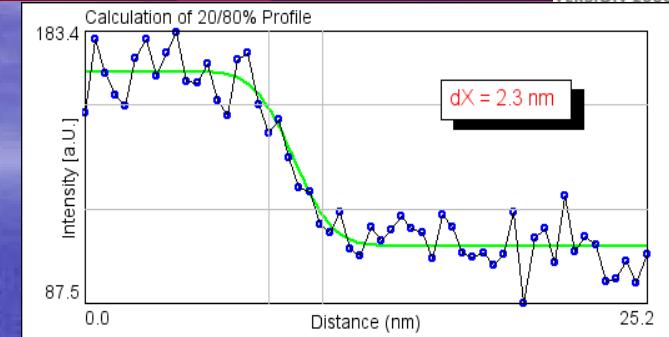
Other FIB columns :

- Sidewinder from FEI
- Cobra-FIB from OP

Typical FIB column geometry  
(SII Zeta) from J.Reuteler  
ETH Zürich

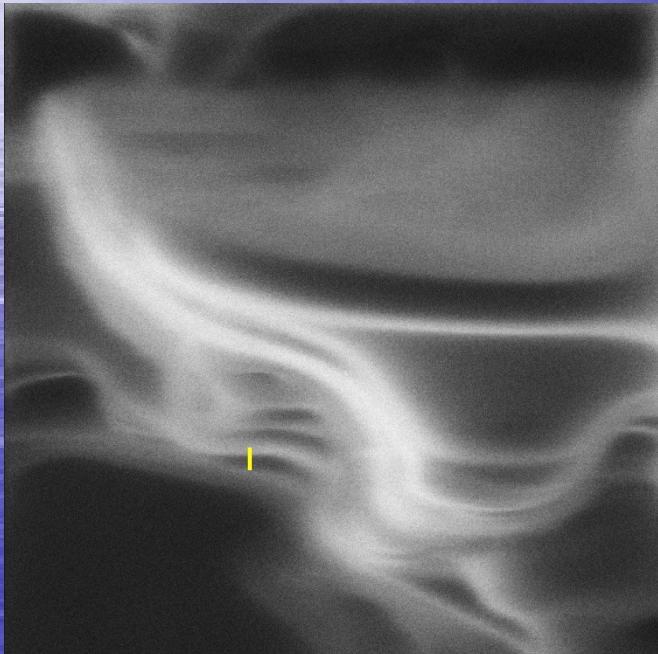
## Resolution measurement ?

- ⇒ SEM separation (subjective) of two objects
- ⇒ FIB more difficult
- ⇒ Fast scan: bad signal to noise ratio
- ⇒ Slow scan: milling while imaging
- ⇒ Measured in % of contrast:



35/65%; 16/84%; 20/80%; 25/75% or  $\Delta x_{50}$

- ⇒ E = 30 kV
- ⇒ 1 pA probe current

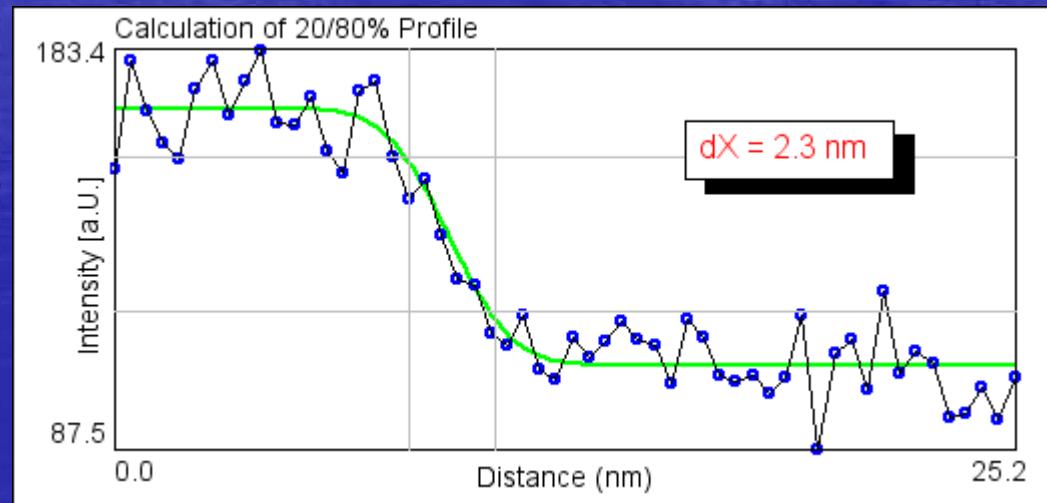


⇒ FOV = 780 nm

## New COBRA-FIB column

Dr. Anne Delobbe  
Dr. Bernard Rasser

⇒ Resolution < 2.5 nm



1,7 nm resolution has been performed at 20-80 with Cobra-FIB

## Resolution measurement

« It is interesting that you should be running into beam-specimen interactions that limit resolution. Mark Utlauf, Lyn Swanson and I, made prediction for imaging resolution for a Ga FIB based on specimen damages and S/N considerations. This issue becomes even more acute now that beam sizes decrease so much. »

*From Jon Orloff to P.Sudraud, June 26, 2008*

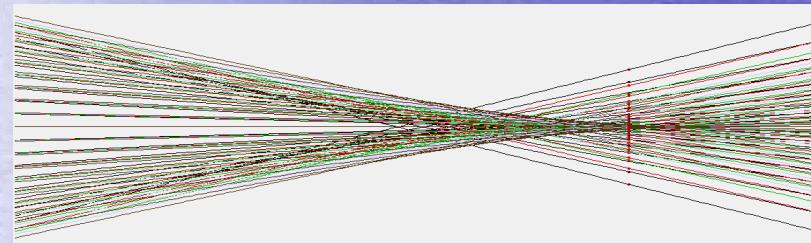
# Resolution and current with gaussian distribution

Contrast	D/sigma	I/It
10-90%	2,565	0,561
12-88%	2,355	0,5
16-84%	1,989	0,39
20-80%	1,685	0,299
25-75%	1,35	0,204
35-65%	0,771	0,072

# Large current spots

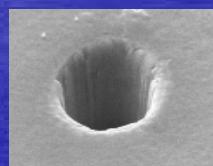
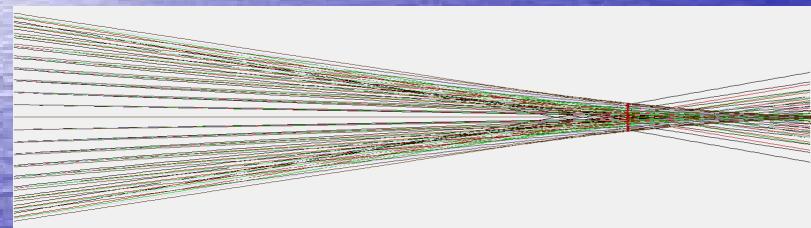
15 nA

## Focusing adjustment

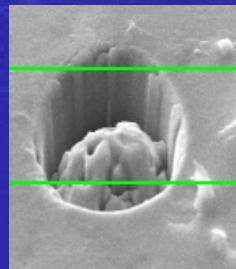
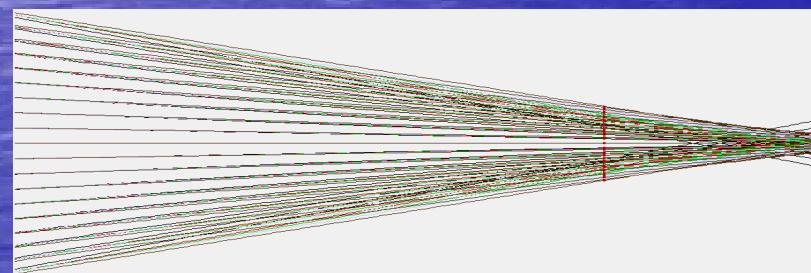


Dr.Anne Delobbe  
Dr.Bernard Rasser

a) focus



b) - 60 V



c) - 110 V

In a) imaging resolution is optimum but milling is not sharp

In b) imaging resolution is poor, but milling is optimum.

# Milling rate

Ion-solid interaction at some keV:  
It involves mostly elastic collisions.  
A very small quantity of heat is produced.

One could imagine that it is a « quiet » mechanism??



Ion-solid interaction is not so quiet !

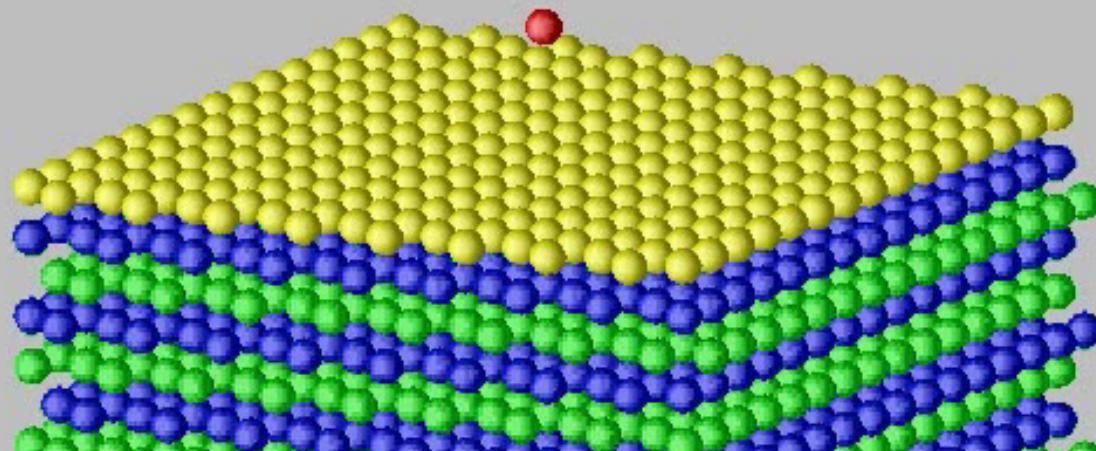


Lets see some simulations from Prof. Dr A.Wucher group  
at Duisburg Universität to be convinced !

Kate's Video Converter (Free)

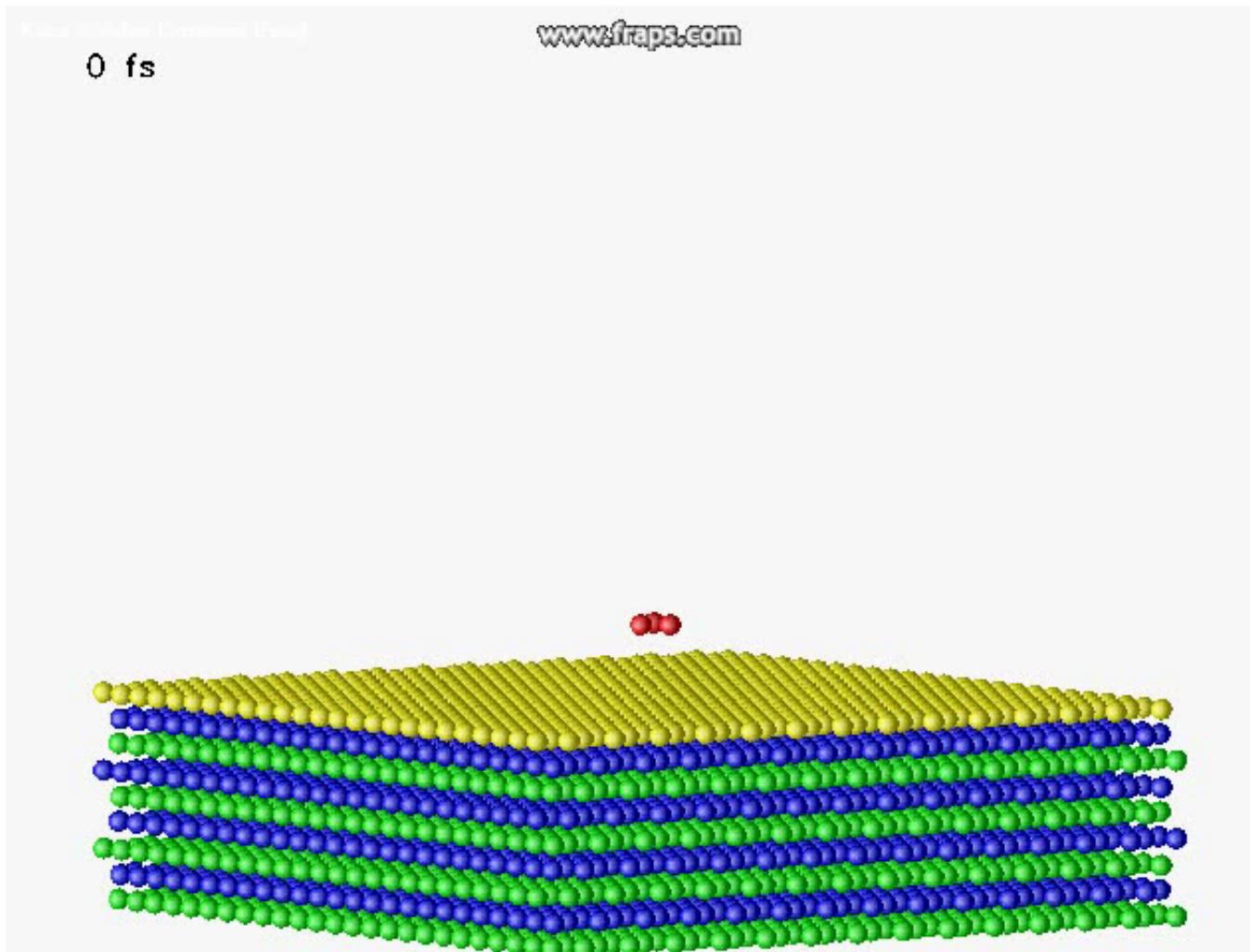
[www.fraps.com](http://www.fraps.com)

0 fs



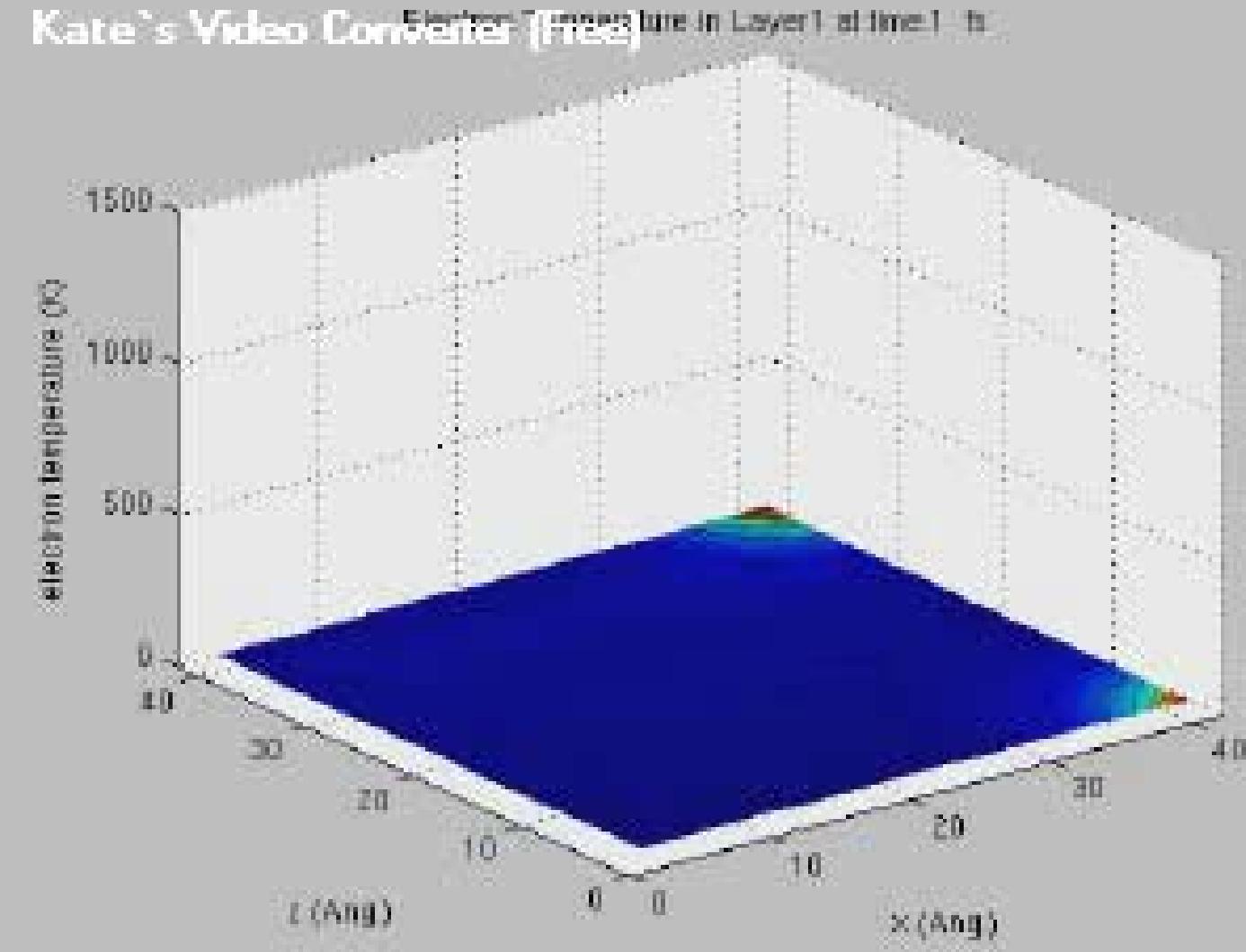
5 keV Ag on Ag (111)

Prof. Dr A.Wucher group – Duisburg Universität



6 keV  $\text{Ag}_3$  on  $\text{Ag}(111)$

Prof. Dr A.Wucher group – Duisburg Universität

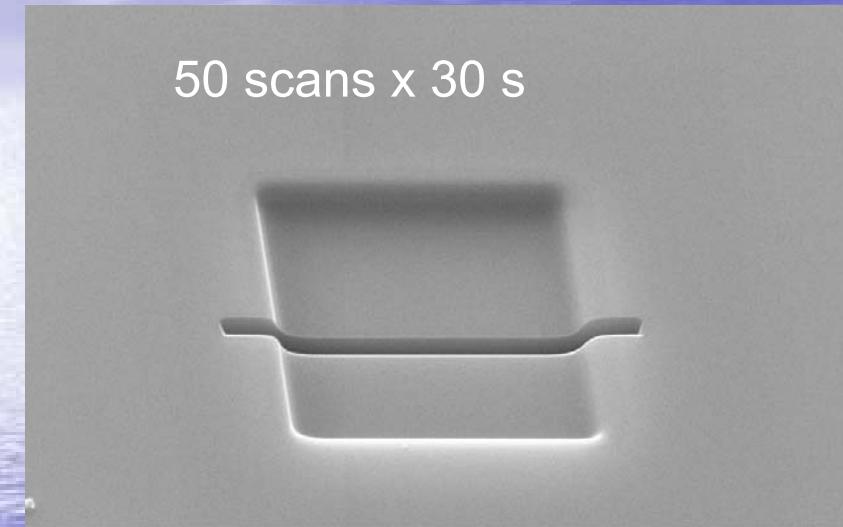


Electronic excitation

Prof. Dr A.Wucher group – Duisburg Universität

# Milling rate at 5kV

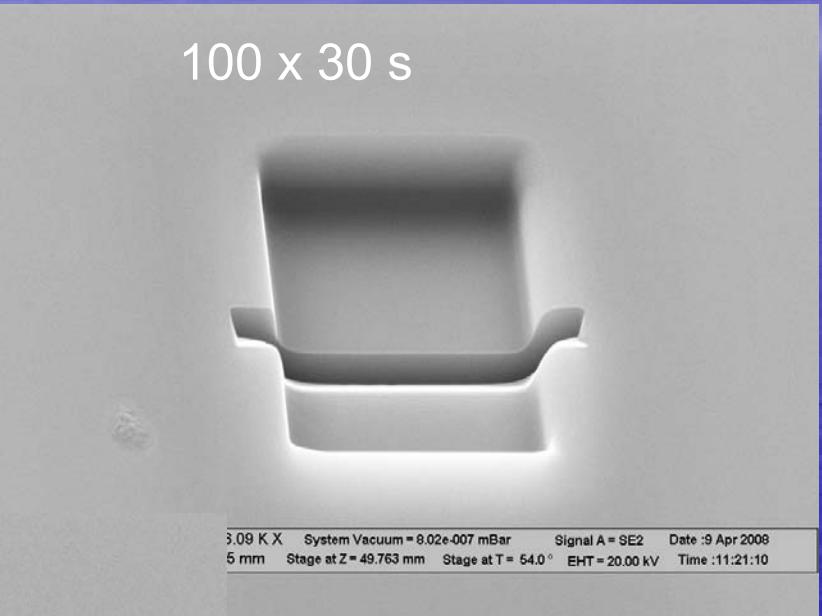
50 scans x 30 s



2μm<sup>2</sup> Mag = 43.06 K X System Vacuum = 8.39e-007 mBar Sig  
VVD = 5 mm Stage at Z = 49.763 mm Stage at T = 54.0° E

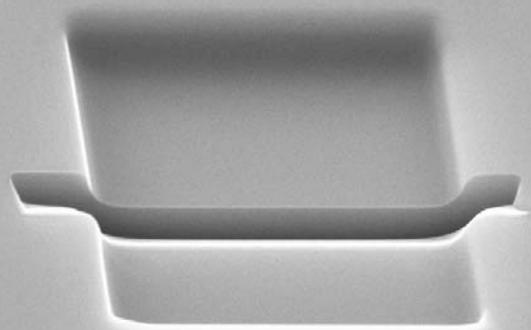
271 pA  
10 μm x 10 μm

100 x 30 s



9.09 K X System Vacuum = 8.02e-007 mBar Signal A = SE2 Date : 9 Apr 2008  
5 mm Stage at Z = 49.763 mm Stage at T = 54.0° EHT = 20.00 kV Time : 11:21:10

70 x 30 s



Sputtering rate measurement

2μm<sup>2</sup> Mag = 51.92 K X System Vacuum = 8.07e-007 mBar Signal A = SE2 Date : 9 Apr 2008  
VVD = 5 mm Stage at Z = 49.763 mm Stage at T = 54.0° EHT = 20.00 kV Time : 11:07:05

3 different times  
For linearity control

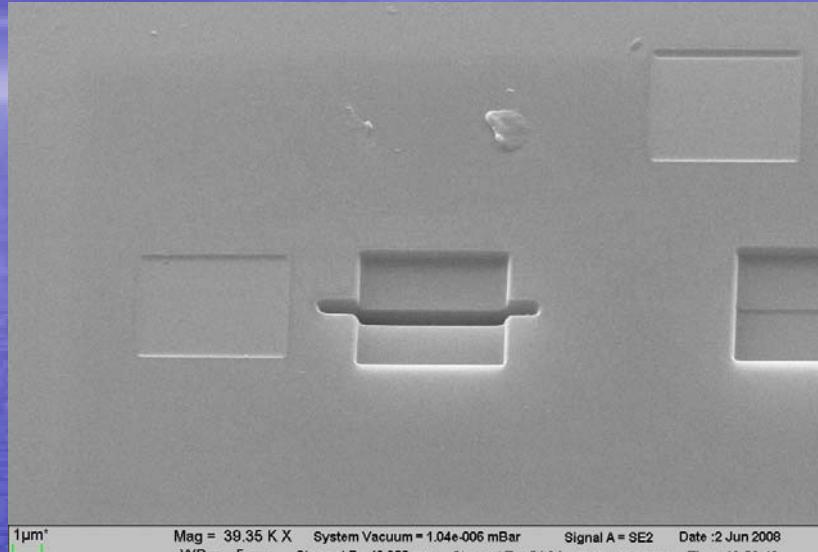
# Milling rate

⇒ Ga+ on Si

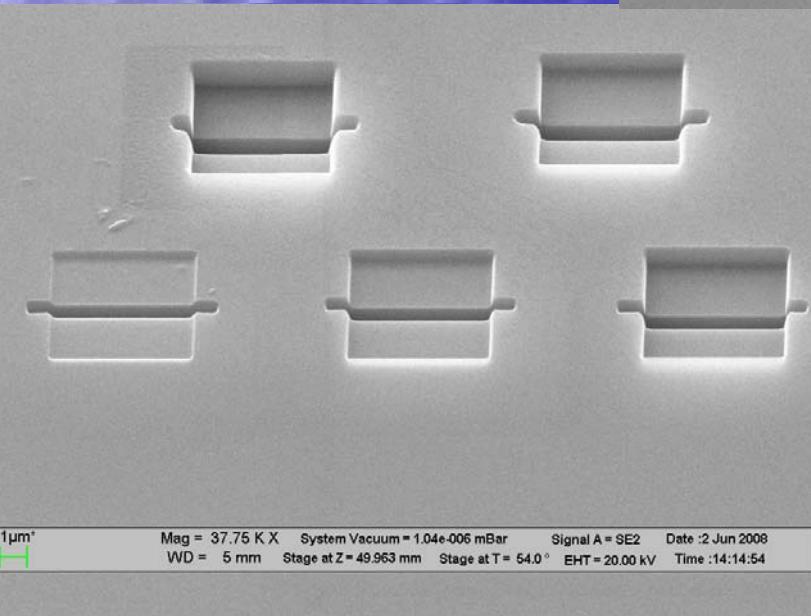
- ⇒ 30 kV : 2.4 atoms/ion
- ⇒ 15 kV : 2.0 atoms/ion
- ⇒ 5 kV : 1.4 atoms/ion

# *Au milling rate on Si*

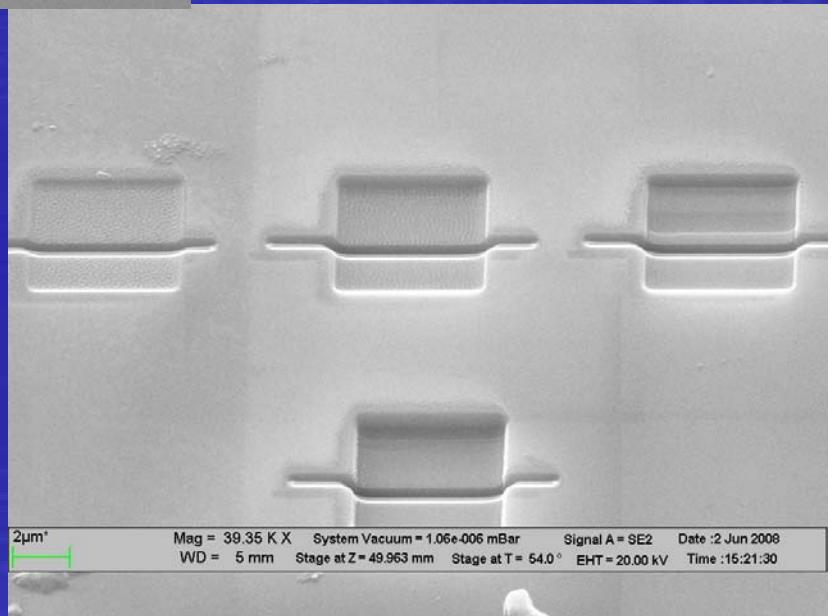
$\text{Au}^+$  sur Si



$\text{Au}^{++}$  sur Si



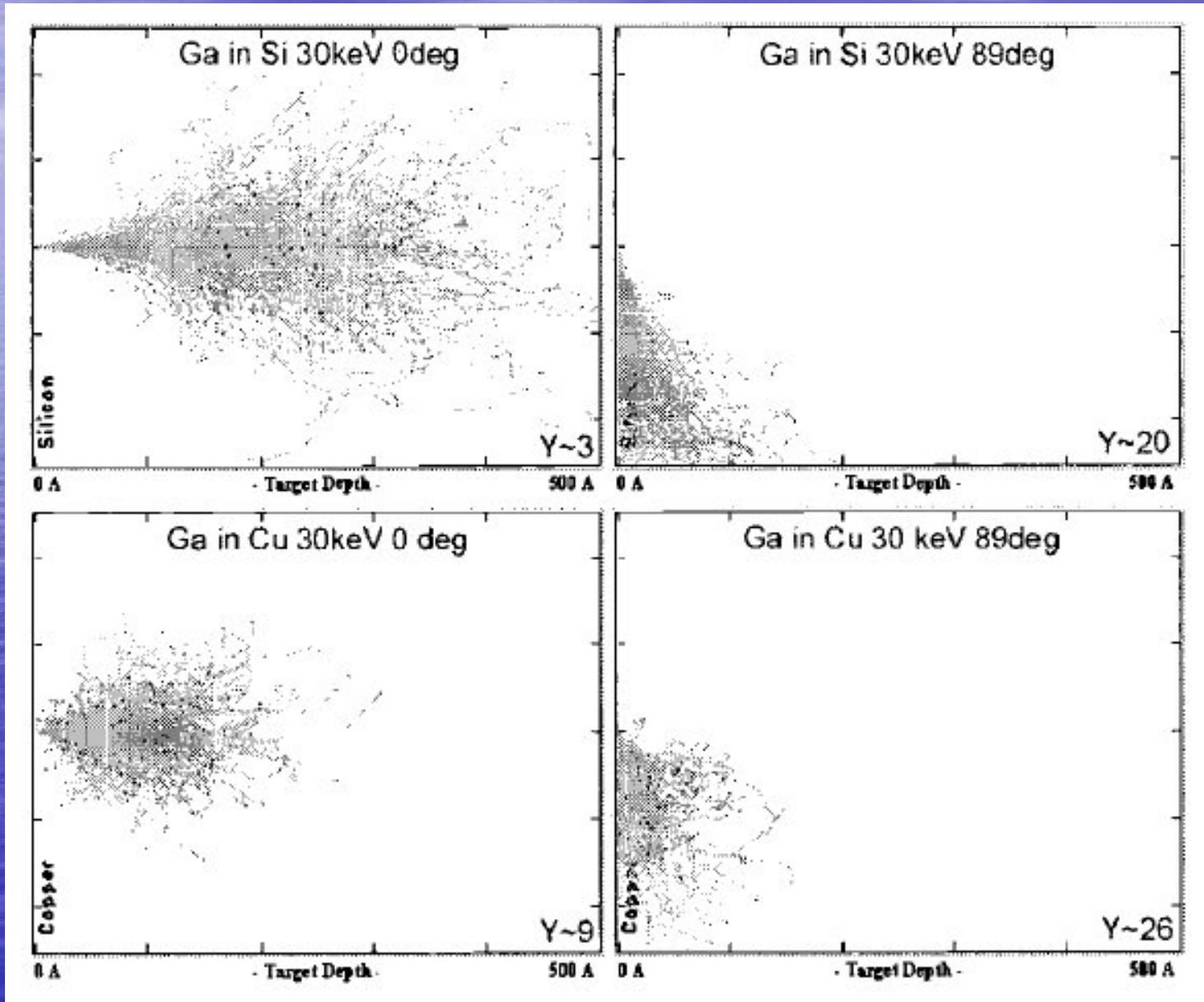
$\text{Au}_2^+$  sur Si



⇒ Au on Si  
Incidence angle 90°

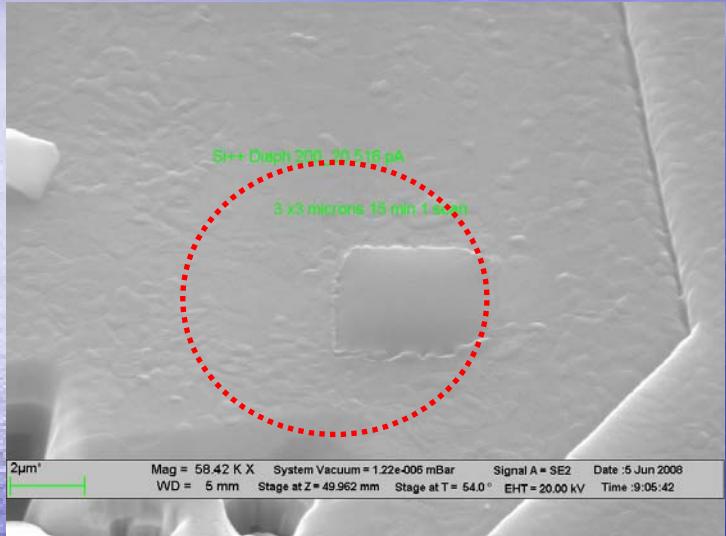
- ⇒  $\text{Au}^+$  : 5.2 atoms/ion
- ⇒  $\text{Au}^{++}$  : 5.6 atoms/ion
- ⇒  $\text{Au}_2^+$  : 9.5 atoms/ion

# Milling rate at 30 keV

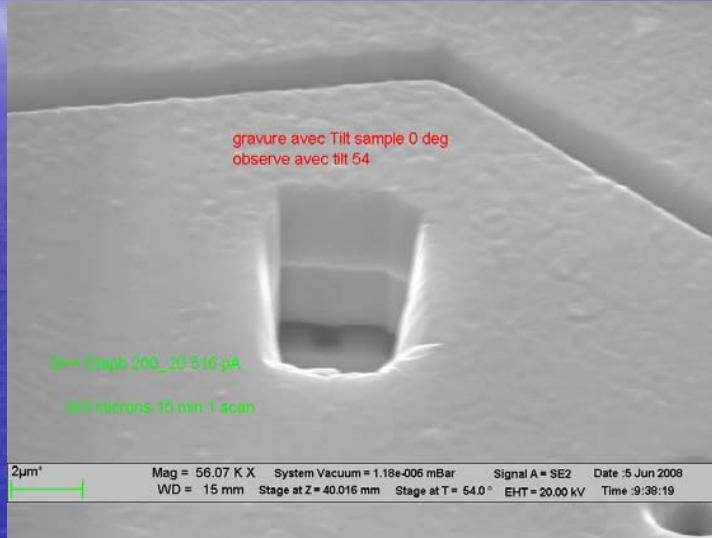


# *Si<sup>++</sup> milling on Si*

90 ° incidence



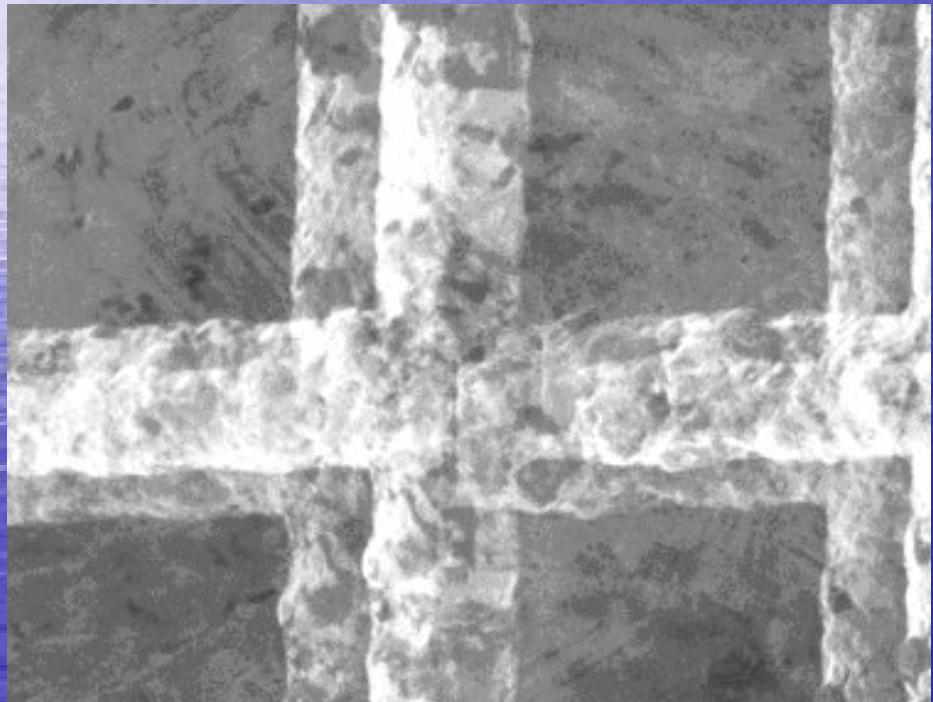
36 ° incidence



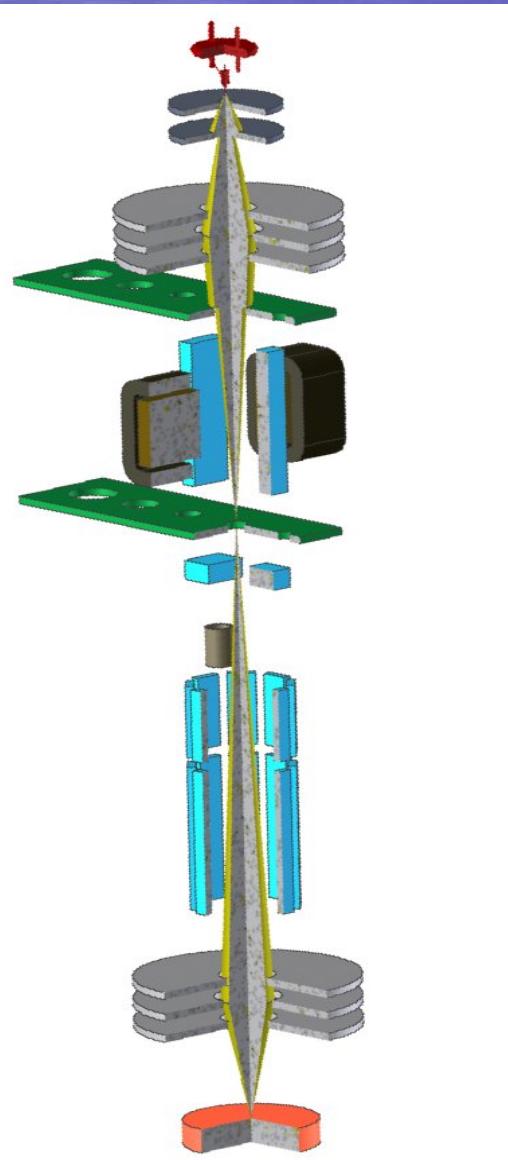
Same conditions  
at different angles

Polishing of existing  
cross section

## CANIION 31X FIB column



*Separation of the two Ga isotopes*



- Ion source
- Extraction electrodes
- Condensor lens
- Ion current selection aperture
- Wien filter
- Mass selection aperture
- Blanking
- Faraday cup
- Scanning and Stigmation octupole
- Objective lens
- Sample

## OPTIFIB column

- ✓ Patented concept
- ✓ Coaxial ion / photon beams
- ✓ Visible and I.R. imaging capabilities
  - Resolution < 0,5 µm
  - F.O.V. =  $250 \times 250 \mu\text{m}^2$
- ✓ Ion resolution < 8 nm
  - F.O.V. =  $400 \times 400 \mu\text{m}^2$
  - Beam current : 1 pA to 20 nA



# Some FIB manufacturers



**DCG**  
Systems



**ZEISS**



**FEI COMPANY™**



**Φ ULVAC**  
ULVAC-PHI, INC.



**TESCAN**  
DIGITAL MICROSCOPY IMAGING



**Raith**  
INNOVATIVE SOLUTIONS FOR NANOFABRICATION AND SEMICONDUCTOR NAVIGATION



**HITACHI**  
Inspire the Next



**SII**



**JEOL**

## **Some industrial requirements**

**Non reactive elements (in-line FIB)**

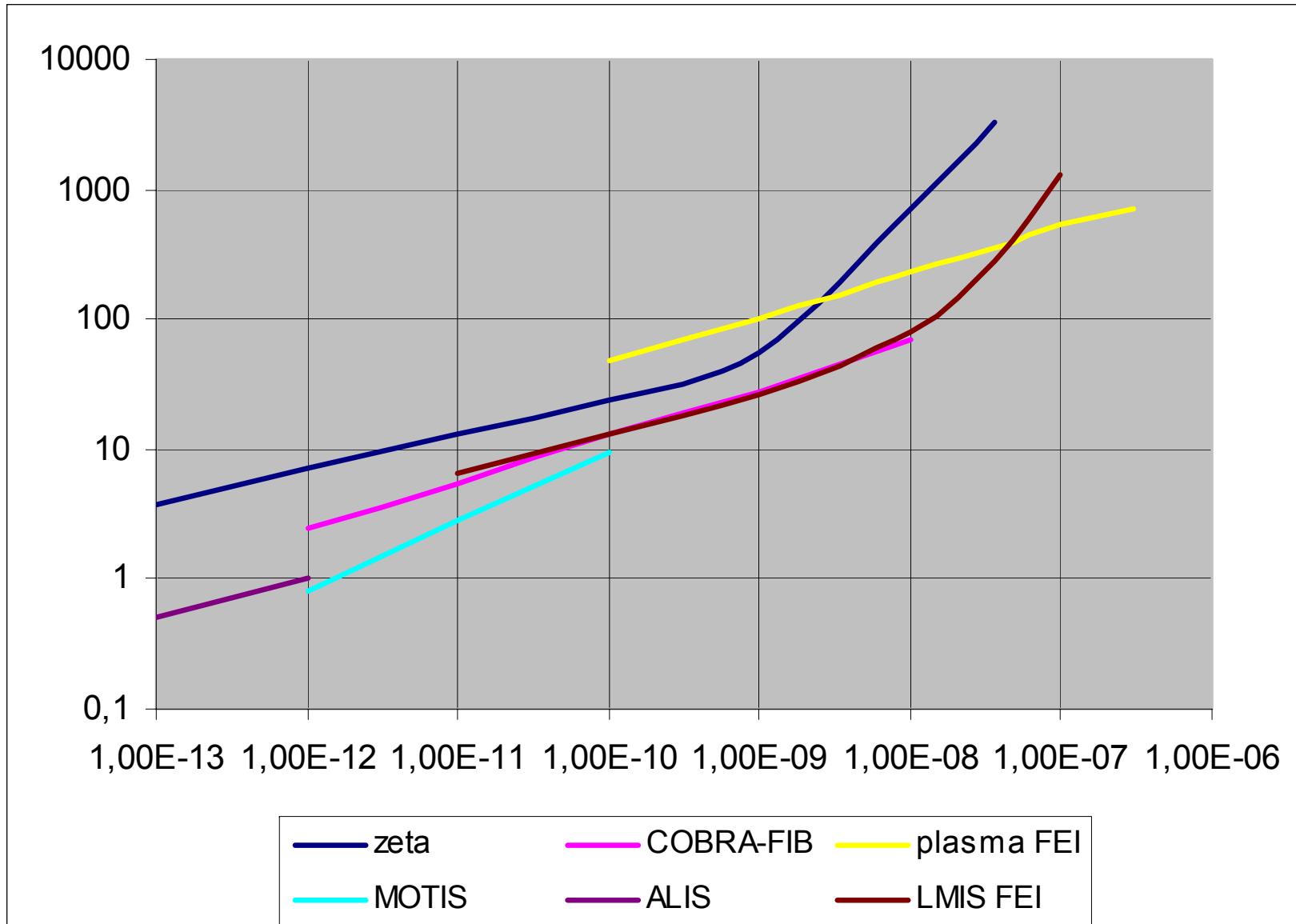
**Low energy FIB (M.Rauscher's work)**

**Nanometric and atomic (?) scale**

**Large volumes (MEMS)**

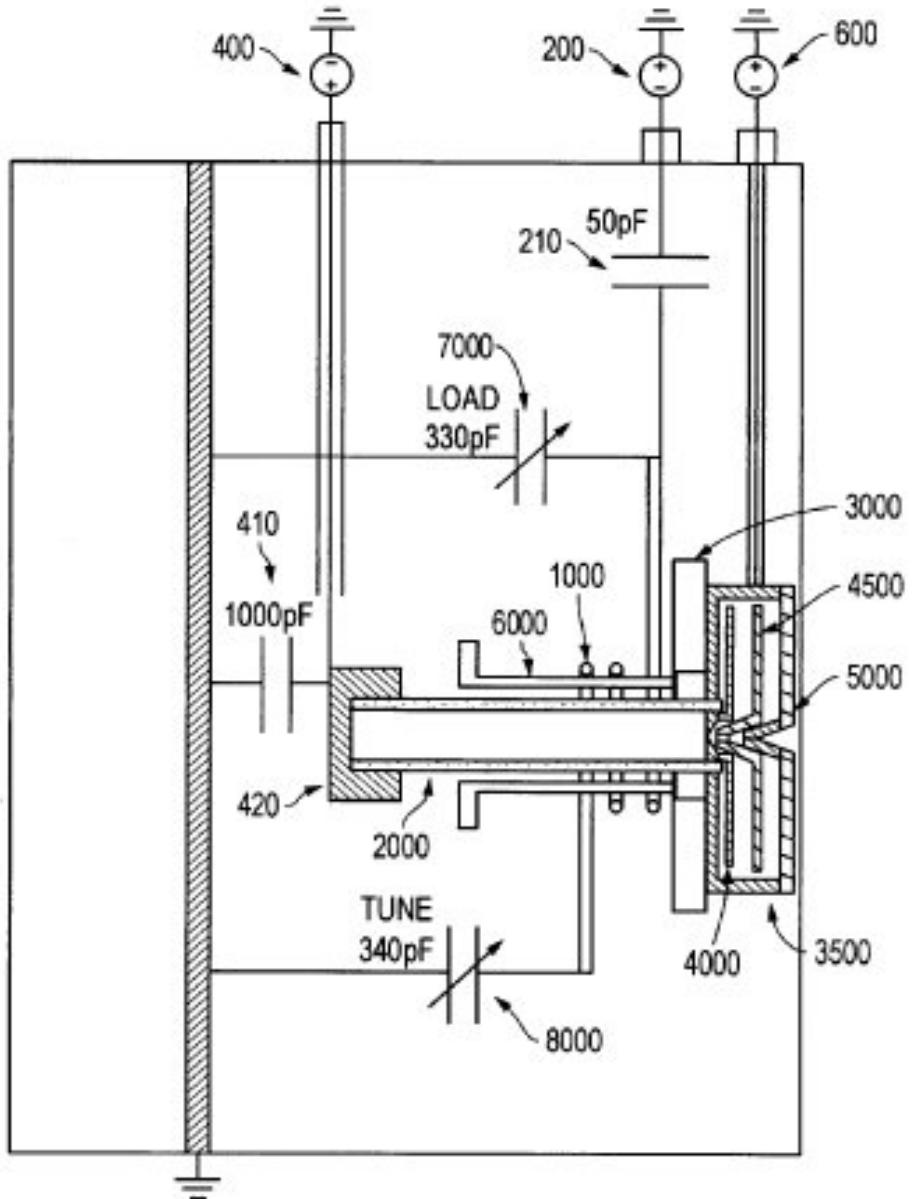
**Very reactive elements (SIMS, local chemistry)**

**Light elements (imaging, analysis)**



**Current and future FIB Technology**

# Future of FIB High currents



Patent J. Keller N. Smith et al  
(FEI)

Inductively coupled plasma

Argon or Xenon (clean FIB)

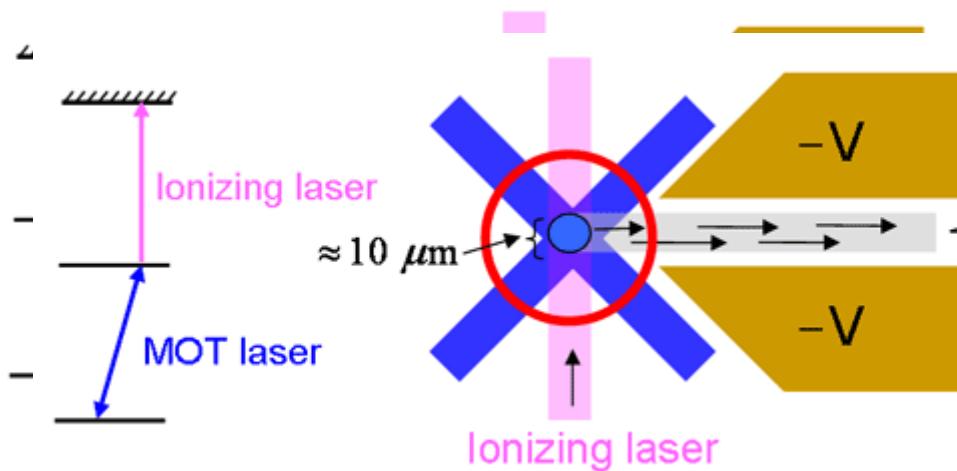
$\Delta E$  7 eV (Xenon)

Angular intensity  $\times 10^3$  LMIS

For  $I > 50\text{nA}$  better than LMIS

# Future of FIB

## Magneto optical trap ion source (motis)



Enlarges ion pannel  
(reactive & non-reactive)

Extremely low ΔE (0.1eV)

Extremely low divergence

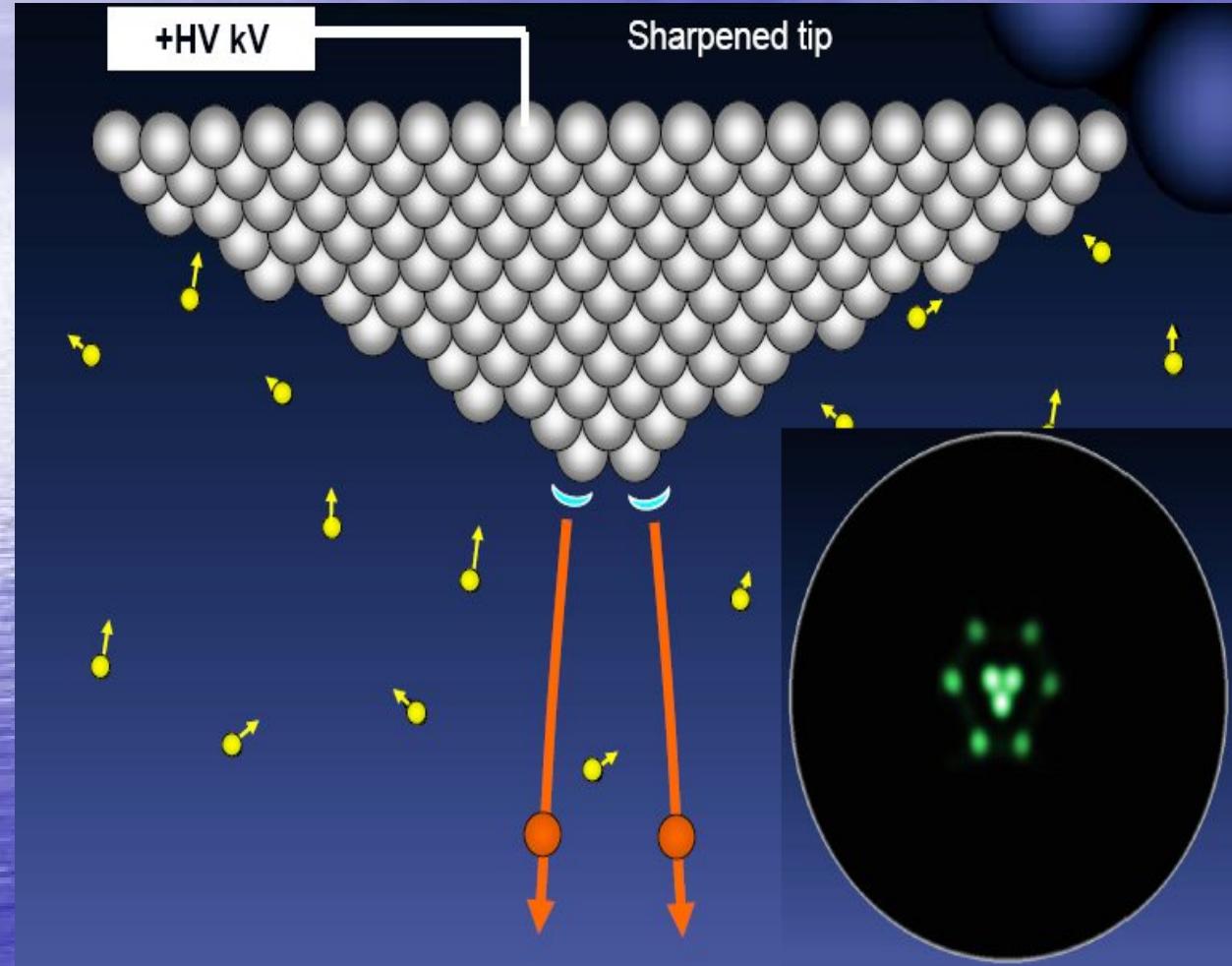
Brightness >  $10^5 \text{A m}^{-2} \text{sr}^{-1} \text{V}^{-1}$

Expensive

CNST – NIST

Orsay-Physics - Orsay Paris XI Univ.  
Dept of Applied Physics Eindhoven Univ.  
FEI

# Future of FIB GFIS Alis – Zeiss Orion



Very High Brightness

Atomic Virtual Source Size

Low energy spread

Diffraction < SEM

Sub nanometric d

Helium ions

Low current

Limited number ion species

**Patented**



# Microscopic Liquid Ionic Compound Ion Source



*MI.LICIS provides  
reactive ions*

***Both polarities***

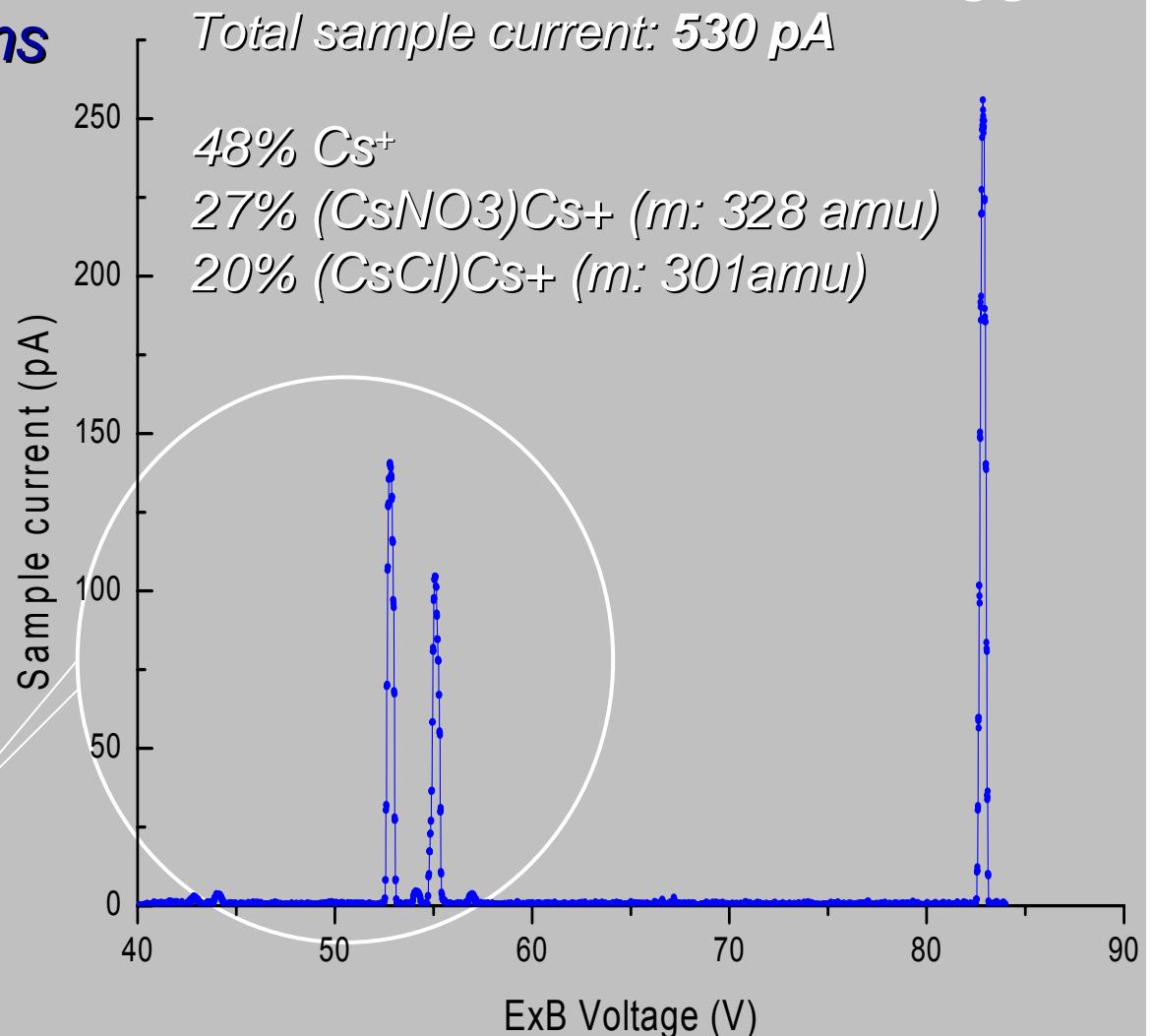
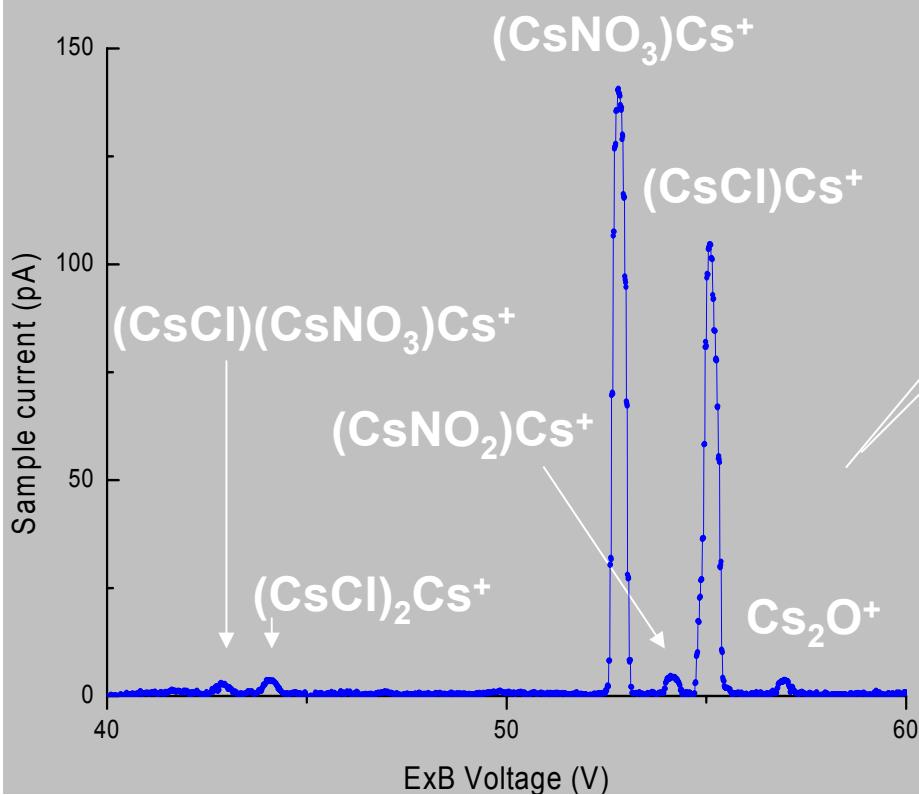


***High brightness***

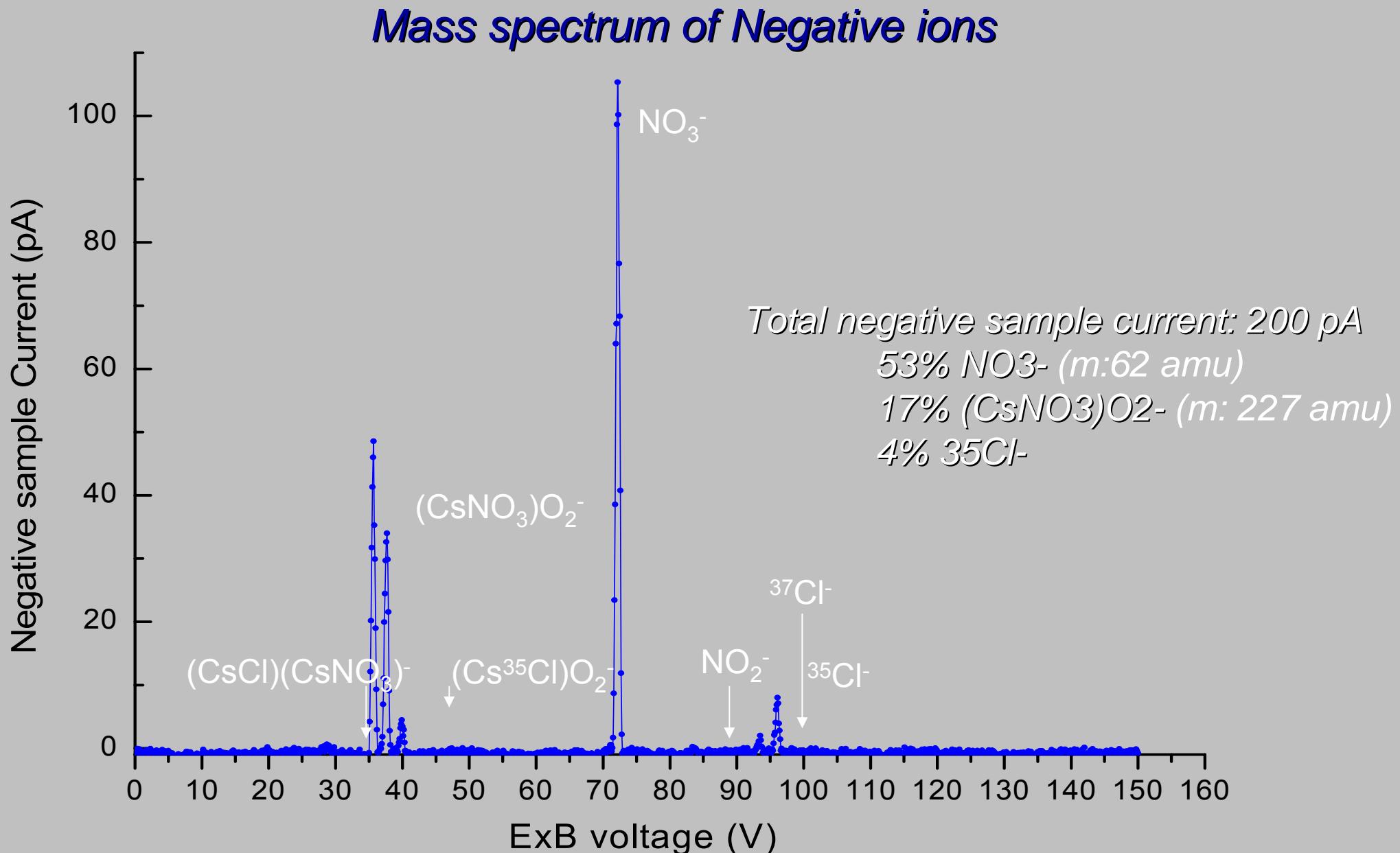
$$B \approx 10.5 \text{ A.cm}^2.\text{sr}^{-1}$$

Olivier Salord  
Dr. A. Houel  
Dr. Pierre Sudraud

## Mass spectrum of Positive ions



## M.I.L.I.C.I.S



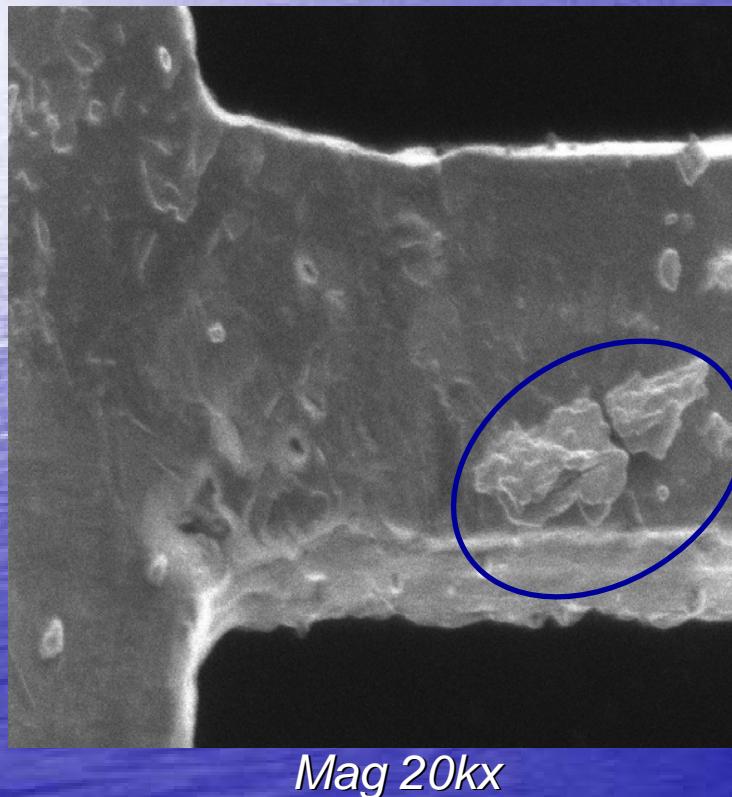
Patented

NANOSOLUTIONS  
by  
OR SAY PHYSICS

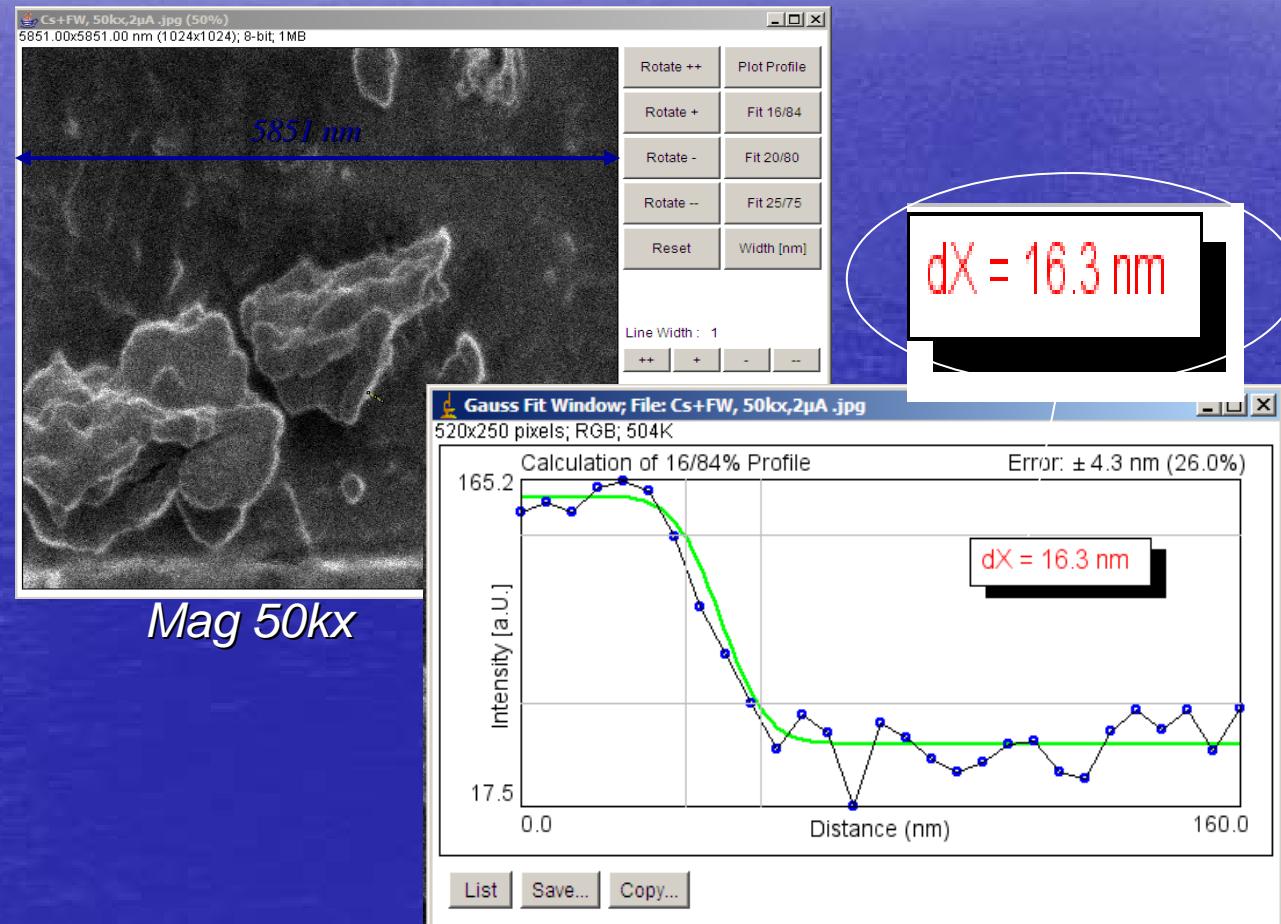


MI.L.I.C.I.S

*Resolution at 30kV obtained with experimental setup  
Positive ion beam filtered by O.P ExB Wien filter: Cs<sup>+</sup> imaging*



Mag 20kx

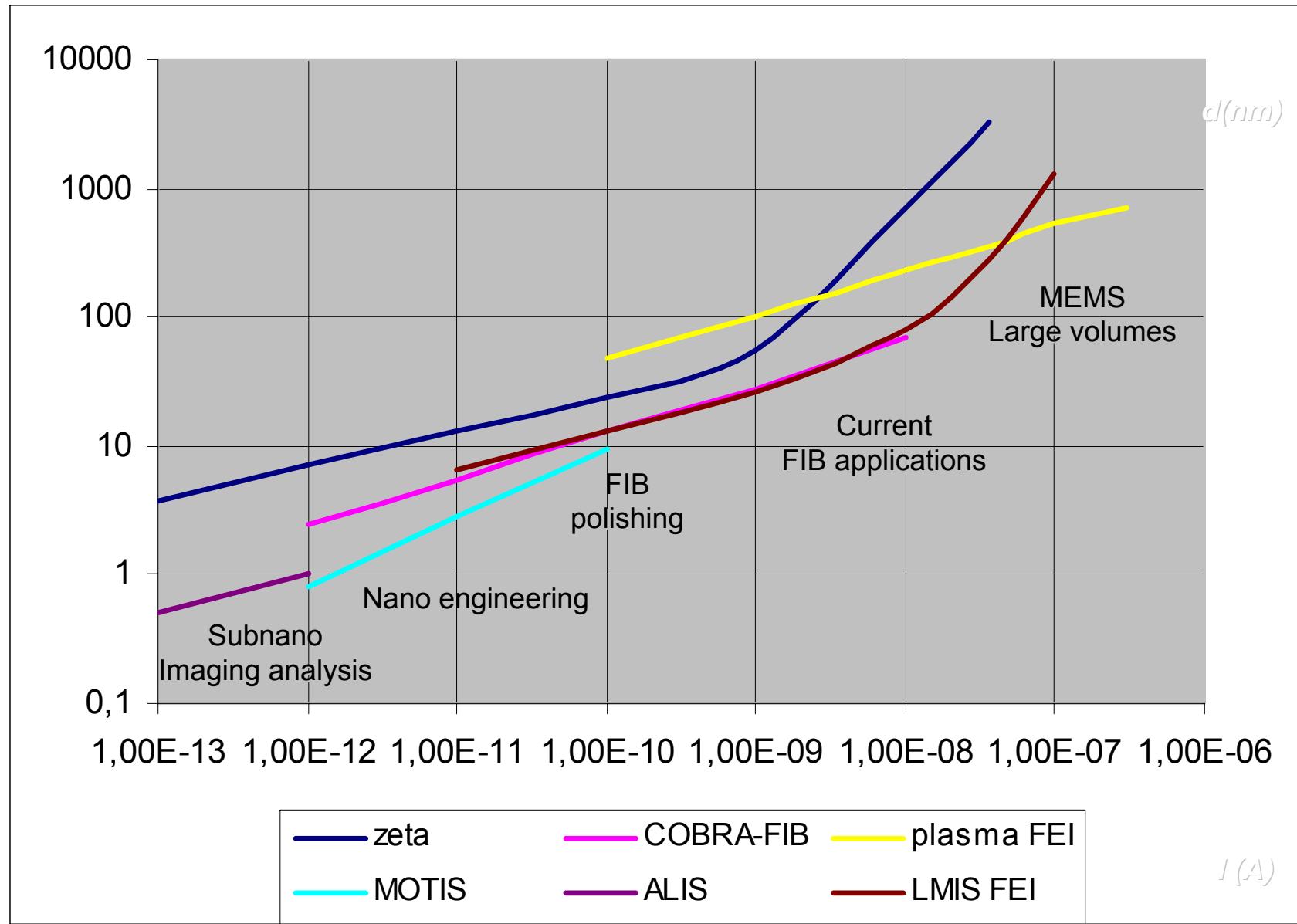


Nominal energy: 30kV

Acceptance aperture : 150μm, Mass aperture: 20 μm

$I_{sample}=10\text{pA}$ ;  $I_{em}=2\mu\text{A}$

Olivier Salord



## **Prospective of FIB: a panel of complementary technologies ?**

**ICRF plasma for clean high currents FIB**

**LMIS FIB could kept many applications**

**MOTIS for clean low energy nanometric FIB  
MOTIS and MILICIS for HR SIMS**

**GFIS FIB for imaging analysis and assisted  
processing at sub-nanometric scale**

*Thank you for your attention*