



# Microscopie à balayage à double faisceaux (FIB/SEM)

*N. Blanchard<sup>1</sup>, A. Descamps<sup>2</sup>, T. Douillard<sup>3</sup> and B. Van de Moortèle<sup>4</sup>*

<sup>1</sup>ILM, Uni. C. Bernard Lyon1, Université de Lyon, France

<sup>2</sup>INL, INSA de Lyon, Université de Lyon, France

<sup>3</sup>MATEIS, INSA de Lyon, Université de Lyon, France

<sup>4</sup>LGL, ENS de Lyon, Université de Lyon, France



JEOL 2010F

1997



déc.2012

ETEM™ commercial



TOPCON



JEOL 2010



LEO-912

2000



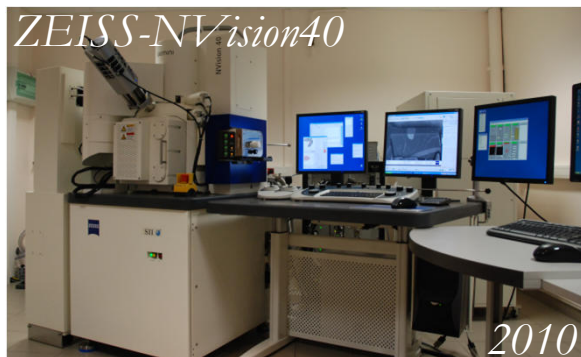
Phoenix-Varian

v|tome|x



FEI-ESEM-XL30

2002



ZEISS-NVision40

2010

instruments 100% CLYM

instruments partagés labos/CLYM



ZEISS Supra 55VP

Mateis



TESCO Mira3

INL-INSA de Lyon

instruments labos



# CaZaC



## Le bureau de CaZaC

Bertrand Van de Moortèle - ENS Lyon : Président

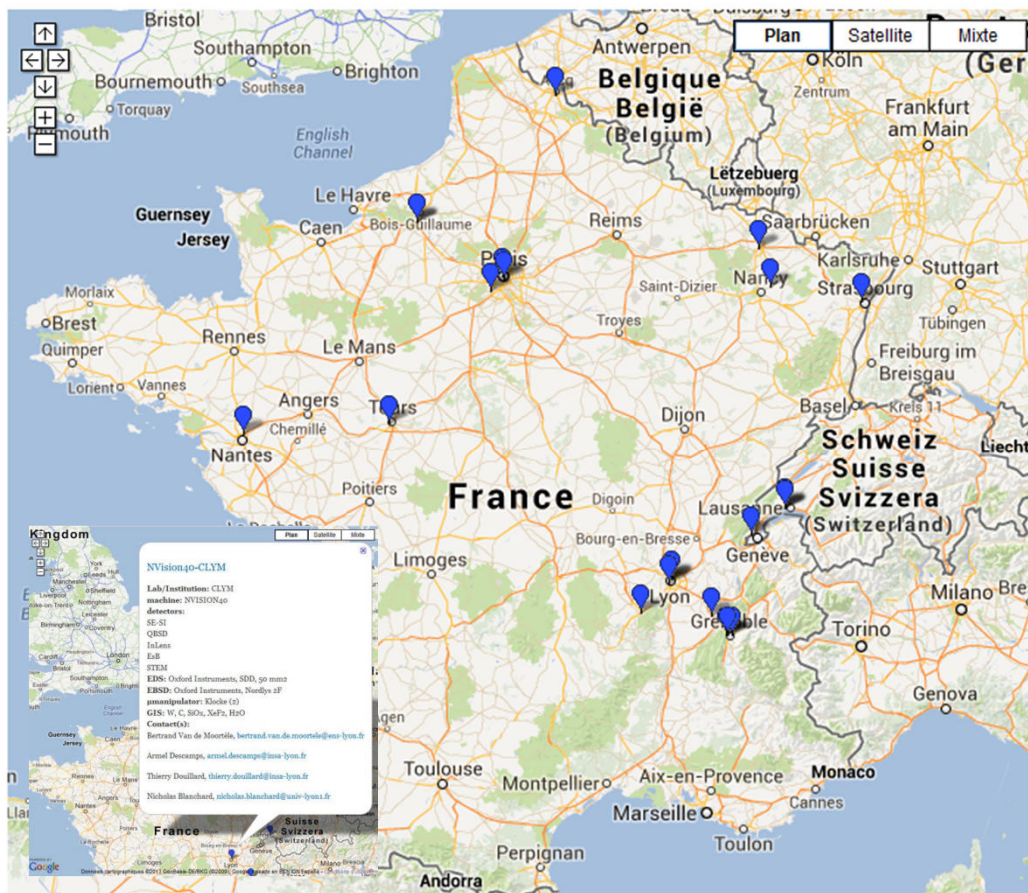
Eric Gautier - CEA Grenoble : Vice-Président

Sergio Sao-Joao - EMSE Saint Etienne : Trésorier

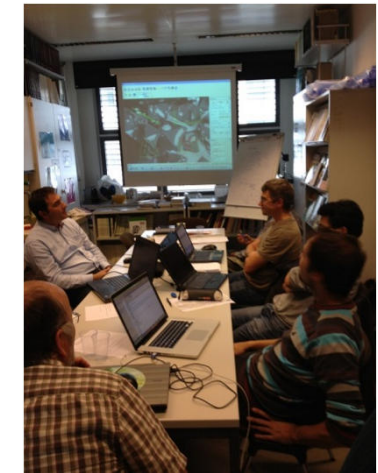
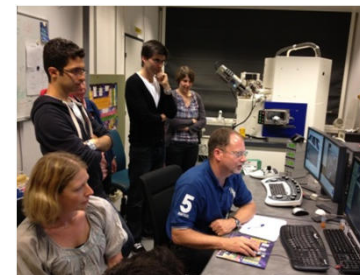
Christophe Rose - INRA de Nancy : Trésorier-adjoint

Emmanuel Cadel - Université de Rouen : Secrétaire

Michel Amez-Droz - HES : Secrétaire-adjoint



jeudi 23 Mai 2013			vendredi 24 Mai 2013		
8h30-9h00		<b>Accueil</b>	8h30-9h00		Helko Stegmann (ZEISS) : évolution du site web, quelques développements récents en API
9h00-9h30		Discours de bienvenue : P. Billardon (ZEISS), L. Lüssler (ZEISS)	9h00-9h30		P. Gnauck (ZEISS) : Orion NanoFab
9h30-10h30		J. Cazaux (Univ. De Reims) : Le Filtrage en énergie des électrons secondaires et rétrodiffusés : Principes, Intérêts, Applications	10h00-10h30		M. Cantoni (CIME-EPFL) : Merlin
10h30-11h00		pause café	10h30-11h00		pause café
11h00-12h30	11h00-12h00	P. Gnauck (ZEISS) : nouveautés chez Zeiss	11h00-12h30	12h00-12h30	D. Lethiec (INRA de Nancy) : Utilisation des techniques du froid et de la pression contrôlée pour l'observation et l'analyse 3D d'espèces biologiques
	12h00-12h30	Anna Sartori-Rupp (Institut Pasteur) : $\mu$ -corrélative		12h30-12h30	Table ronde, questions au constructeur ; contrats d'entretien, maintenance, upgrade des machines.
12h30-14h00	REPAS + photographie de groupe		12h30-14h00	REPAS (+ résultat concours photos)	
14h00-14h20		E. Robit : EDX Bruker 4 cadrans.	14h00-14h15		A.S. Gay (FPEN) : apport de la préparation par polissage ionique pour les observations MEB
14h20-14h40		PH Jouneau (CEA Grenoble) : installation des machines : clim, vibrations, etc...	14h15-14h35		J. Guyon, N. Guy (EMIS, Univ. De Metz) : prémisses de l'EBSD-3D en mode statique
14h40-15h00		C. Collet (Thales) : Structuration et réalisation de nano-objets assistés par FIB	14h35-15h00		Debriefing Workshop FIB/CS
15h00-15h30		F. Courtade (CNES) : Expertise sur matériel Spatial au laboratoire intégré du CNES	15h00-15h30		présentation libre
15h30-16h00		pause café	15h30-16h00		Frank Stietz (Zeiss) - Trends in Charged Particle
16h30-17h00		C. Boyaval (EMN, Univ. de Lille 1) : Mesures électriques in-situ			fin des journées
17h00-17h30		Nils Ampach (IME) : AFM in situ			
17h30-18h00	<b>Assemblée générale CAZAC</b>				
18h00-18h30	Soirée offerte par ZEISS				



Ateliers, CaZaC 2012, Lausanne

# CaZaC 2014

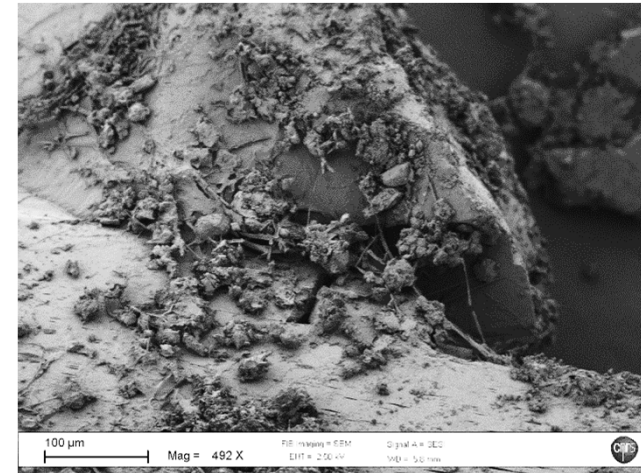
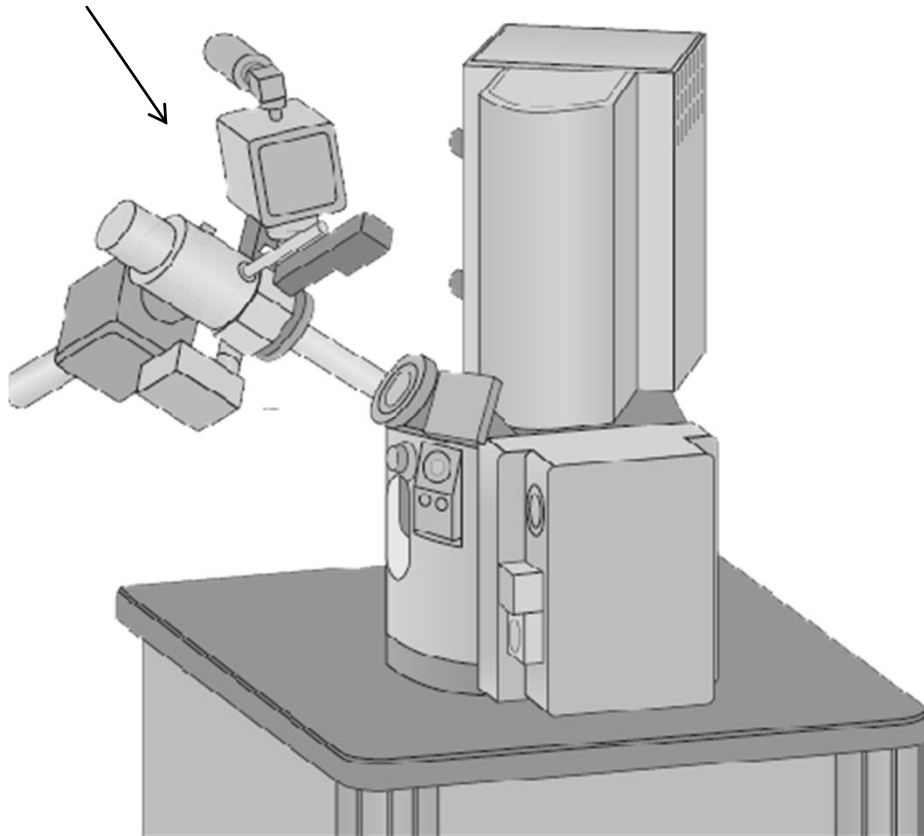
Institut Pasteur

22, 23 et 24 Mai 2014

# Qu'est-ce qu'un FIB?

FIB = Focused Ion Beam

ion column



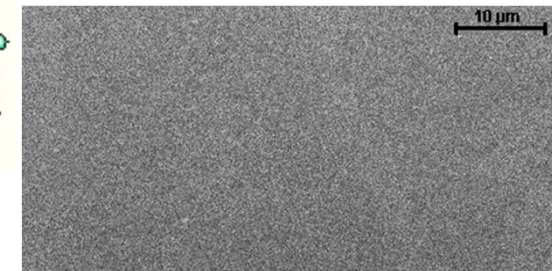
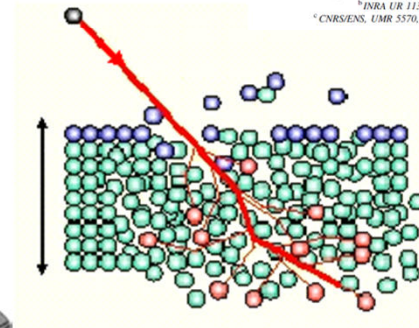
Research in Microbiology 162 (2011) 820–831

[www.elsevier.com/locate/resmic](http://www.elsevier.com/locate/resmic)

Bacterial weathering and its contribution to nutrient cycling in temperate forest ecosystems

Stéphane Uroz<sup>a,b,c</sup>, Phil Oger<sup>c</sup>, Cendrella Lepleux<sup>a,b</sup>, Christelle Collignon<sup>a,b</sup>, Pascale Frey-Klett<sup>a</sup>, Marie-Pierre Turpault<sup>b</sup>

<sup>a</sup>INRA, UMR 1136 INRA Nancy Université "Interactions Arbres Micro-organismes", Centre INRA de Nancy, 54280 Champenoux, France  
<sup>b</sup>INRA UR 1138 "Biogéochimie des Ecosystèmes Forestiers", Centre INRA de Nancy, 54280 Champenoux, France  
<sup>c</sup>CNRS/ENS, UMR 5570, Laboratoire des Sciences de la Terre Ecole Normale Supérieure 46, Allée d'Italie, 69364 Lyon Cedex, France





# Une histoire pas si récente que cela...

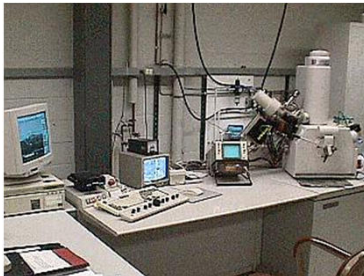
1979: R. Seigler, *Hughes Research Laboratories*, Malibu, California, USA  
“*High-resolution, ion-beam processes for microstructure fabrication*”,  
Seliger *et al*, *J. Vac. Sci. Technol.* **16** (1979) 1610

1979-1992: Focused Ion beam (single beam)

fault diagnostics, mask repair, TEM lamella, gas  
deposition, patterning, contrast orientation image,  
etc...



1993: 1<sup>st</sup> FIB dual beam, DualBeam 620 (Philips)



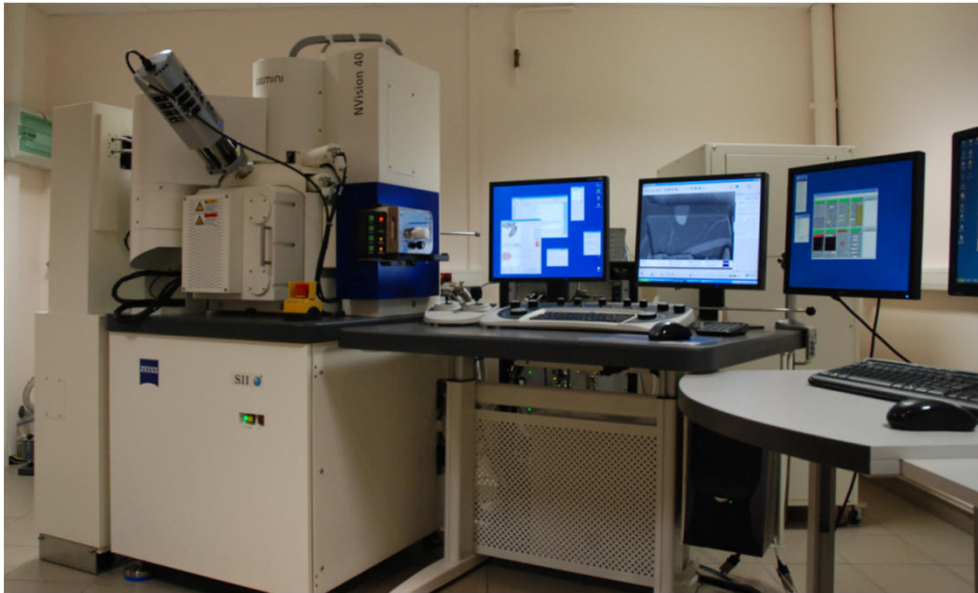
EDX  
EBSD

1998: Development of an Ion and Electron Dual Focused Beam  
Apparatus for Three-Dimensional Microanalysis  
T. Sakamoto *et al.*, *Jpn. J. Appl. Phys.* 37 (1998) pp. 2051-2056

~ 2000: in-situ  $\mu$ manipulateurs



# NVision 40, CLYM



**detectors** : SESI, BSE, InLens, EsB, STEM

**manipulators** : 2 KlockeNanotechnik

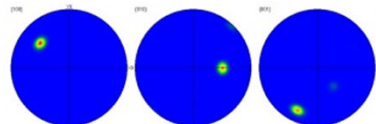
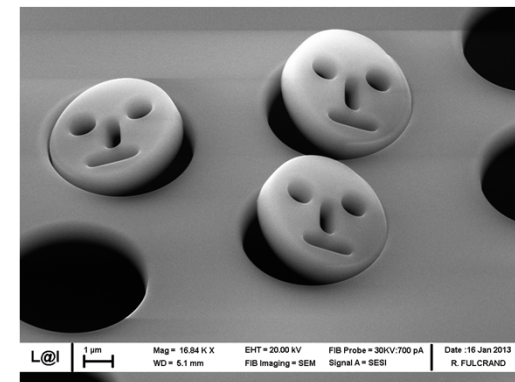
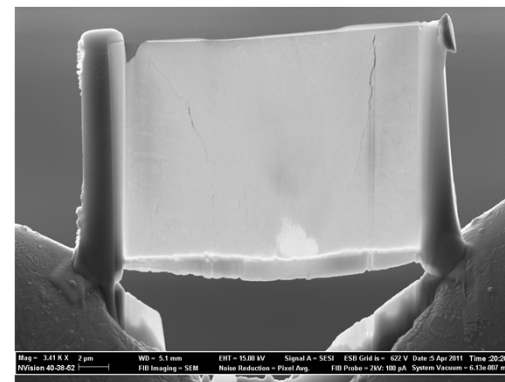
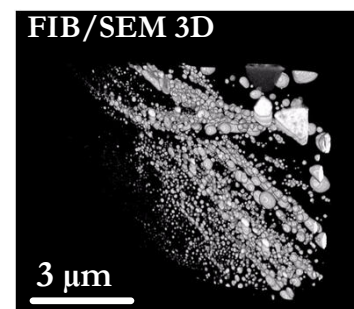
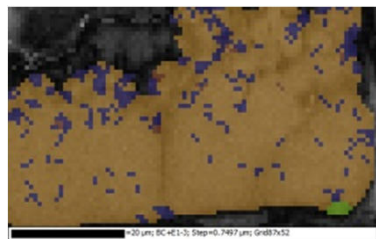
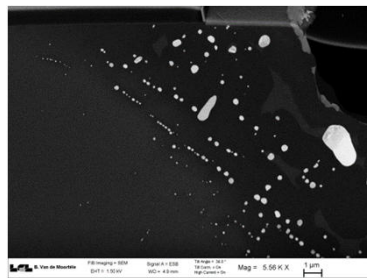
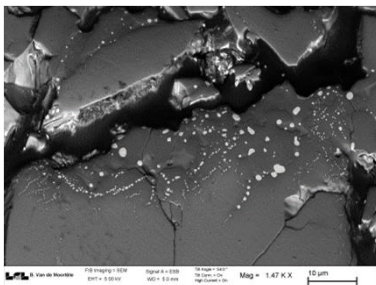
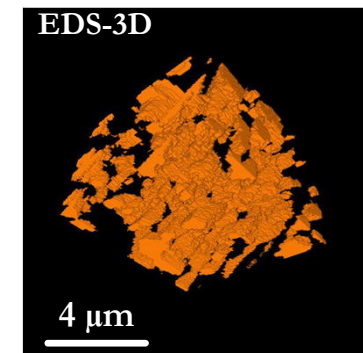
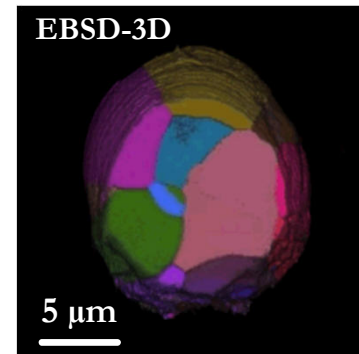
**EDX** : SDD 50 mm<sup>2</sup>, Oxf. Instr.

**EBSD** : camera Nordlys F<sup>+</sup>, Oxf. Instr.

**GIS** : W, SiO<sub>x</sub>, C, H<sub>2</sub>O, XeF<sub>2</sub>

**Flood Gun**

**External Scan generator** : FIBICS



**TEM lamella prep.**

**Nano-patterning**



# Principal Ion Column and FIB Manufacturers

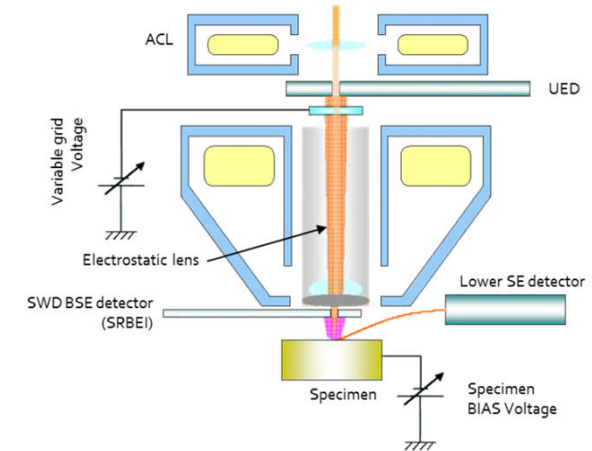
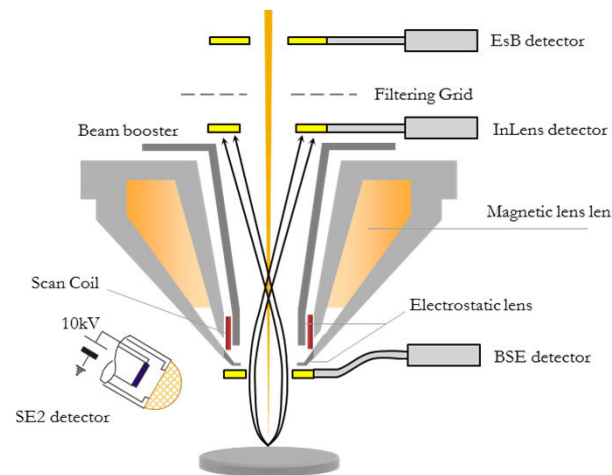
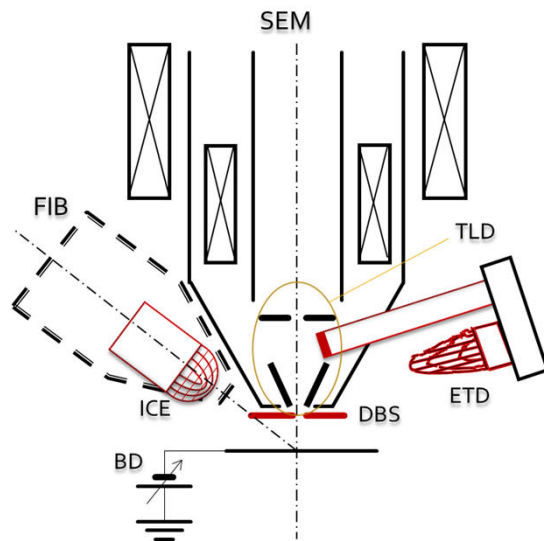
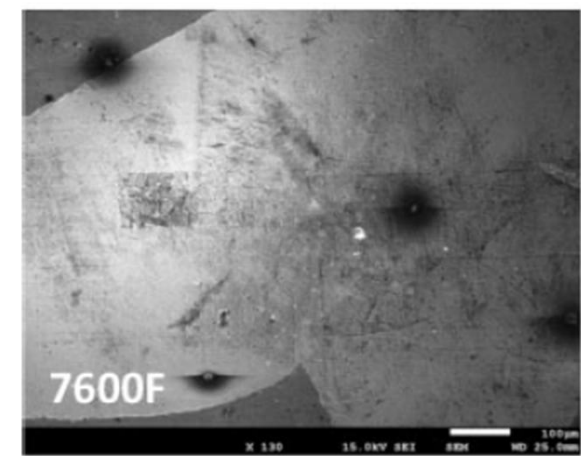
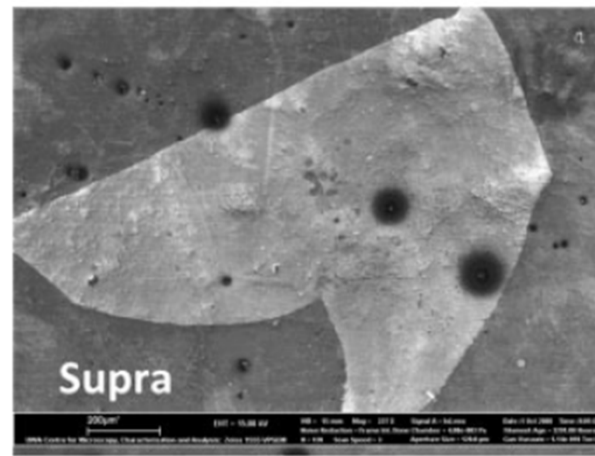
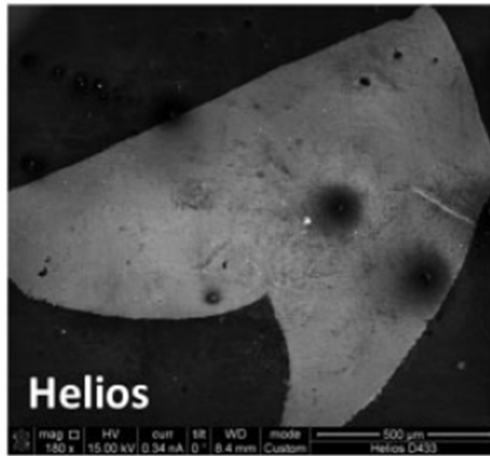


# Principal Ion Column and FIB Manufacturers

A Comparison of Conventional Everhart-Thornley Style and In-Lens Secondary Electron Detectors—A Further Variable in Scanning Electron Microscopy

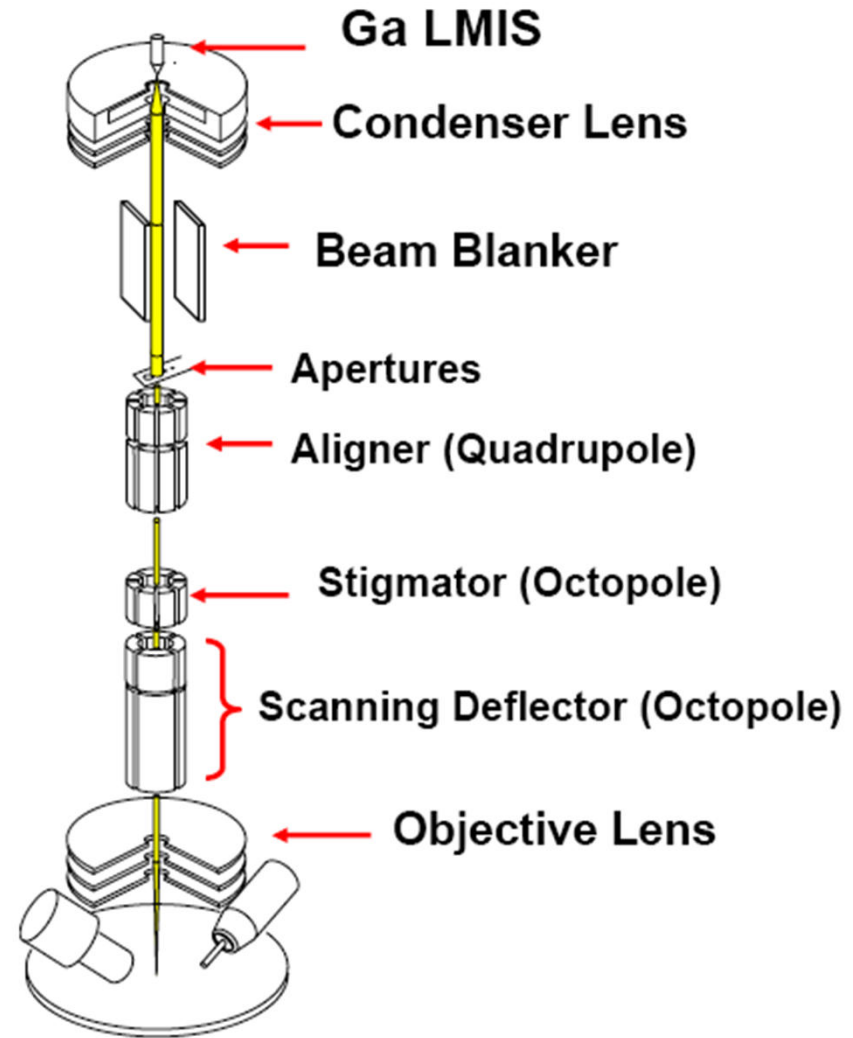
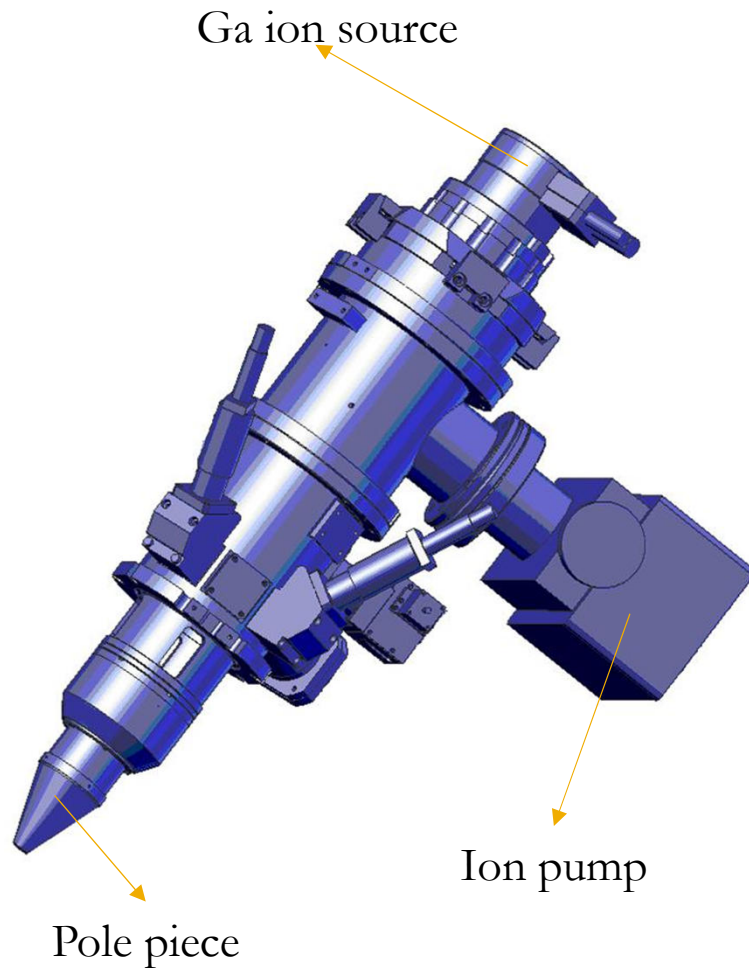
BRENDAN J. GRIFFIN

SCANNING VOL. 33, 162–173 (2011)  
© Wiley Periodicals, Inc.



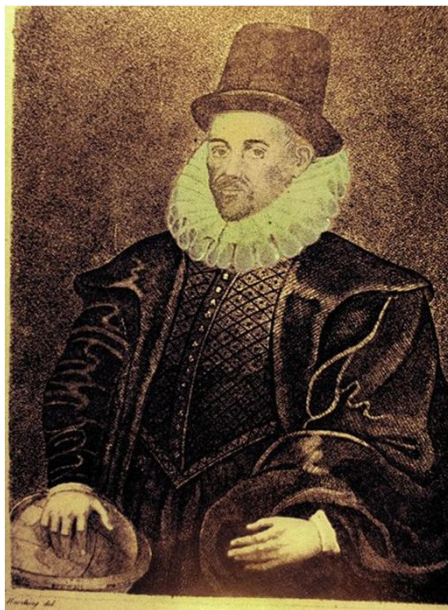


# SII Ion Column



# LMIS: history

**William GILBERT (1544-1603)**, English medical doctor and physicist. The first to observe the formation of a cone when a fluid is subjected to a high electrostatic field. This observation was described in his treatise « *De Magnete* » (1600).



*“... it is probable that amber exhales something peculiar that attracts the bodies themselves, and not air. It plainly attracts the body itself in the case of a spherical drop of water standing on a dry surface; for a piece of amber held at a suitable distance pulls towards itself the nearest particles and draws them up into a cone;...”*

“Cambridge Scientific Minds”, Edited by P. Harman & S. Mitton, Cambridge University Press, 2002



# LMIS: history



314 years later...

John ZELENY (1872-1951), American physicist.

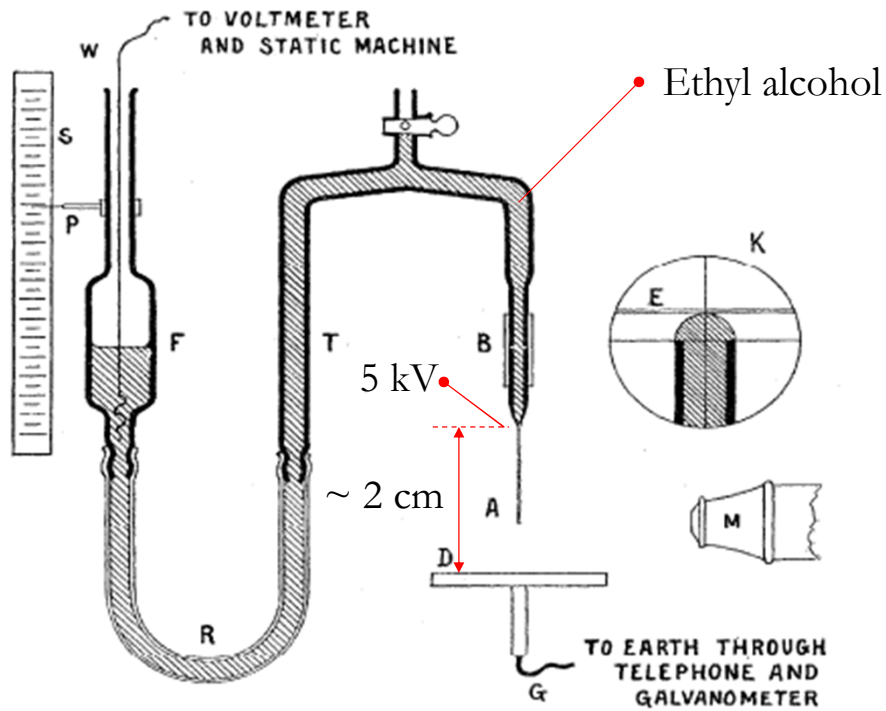
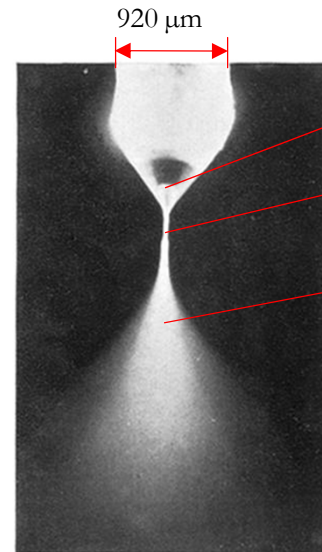
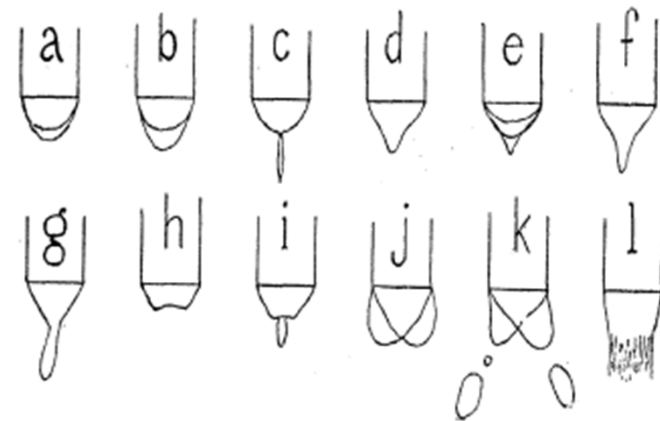


Fig. 1.  
Diagram of apparatus.



- Liquid forms a cone
- Thread dia.  $\sim 4 \mu\text{m}$
- Thread breaks up into droplets

Photograph taken with an exposure time = 30 s

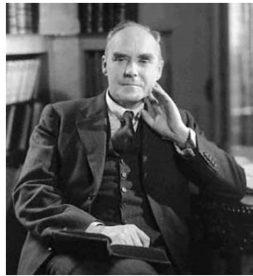


Oscillations of meniscus during intermittent discharge.

J. Zeleny, *Physical Review*, **3** (1914) 69-91

J. Zeleny, *Physical Review*, **10** (1917) 1-6

# Taylor Cone

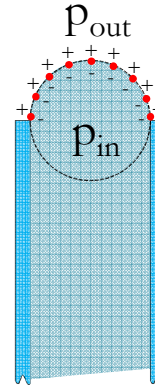


**Sir Geoffrey Ingram TAYLOR (1886-1975),**  
British physicist/mathematician,  
specialised in the field of fluid mechanics.

Taylor was interested by the behaviour of water droplets in the presence of strong electrostatic fields such as storm clouds. With this problematic in mind he found an analytic solution to the equations of electrohydrodynamics.



## Surface Pressure Jump



$$\Delta p = p_{in} - p_{out} = \gamma c - \frac{1}{2} \epsilon_0 F^2$$

Where:

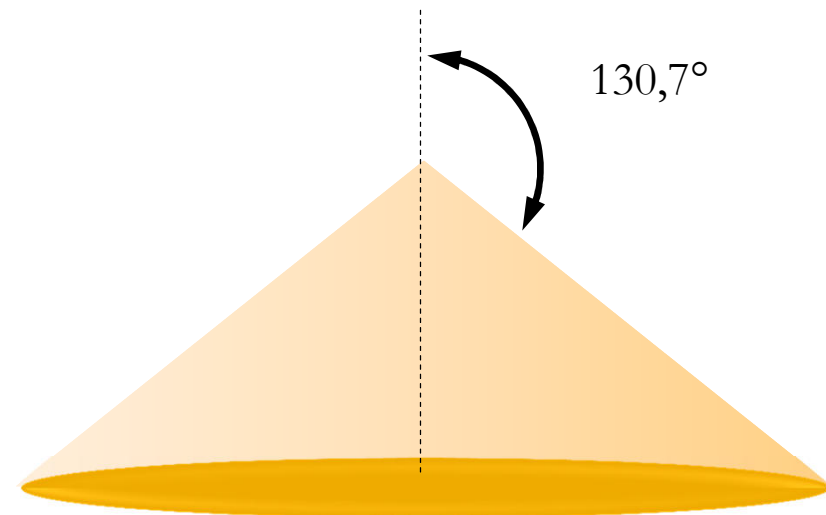
$\gamma$  = surface tension

$c$  = surface curvature

$F$  = electric field

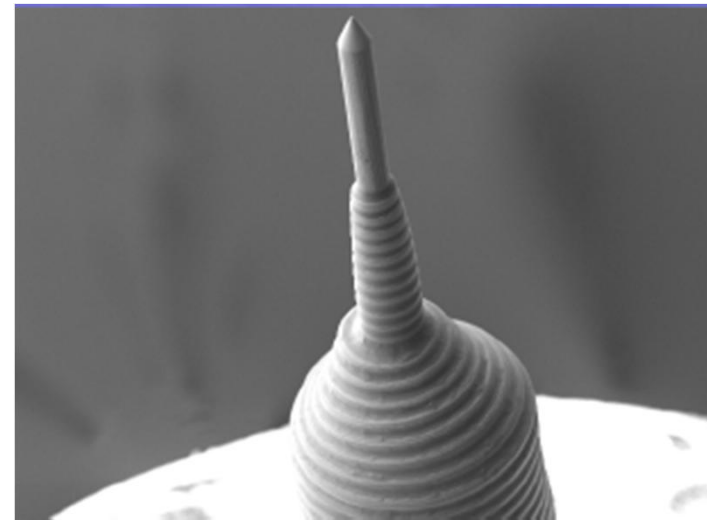
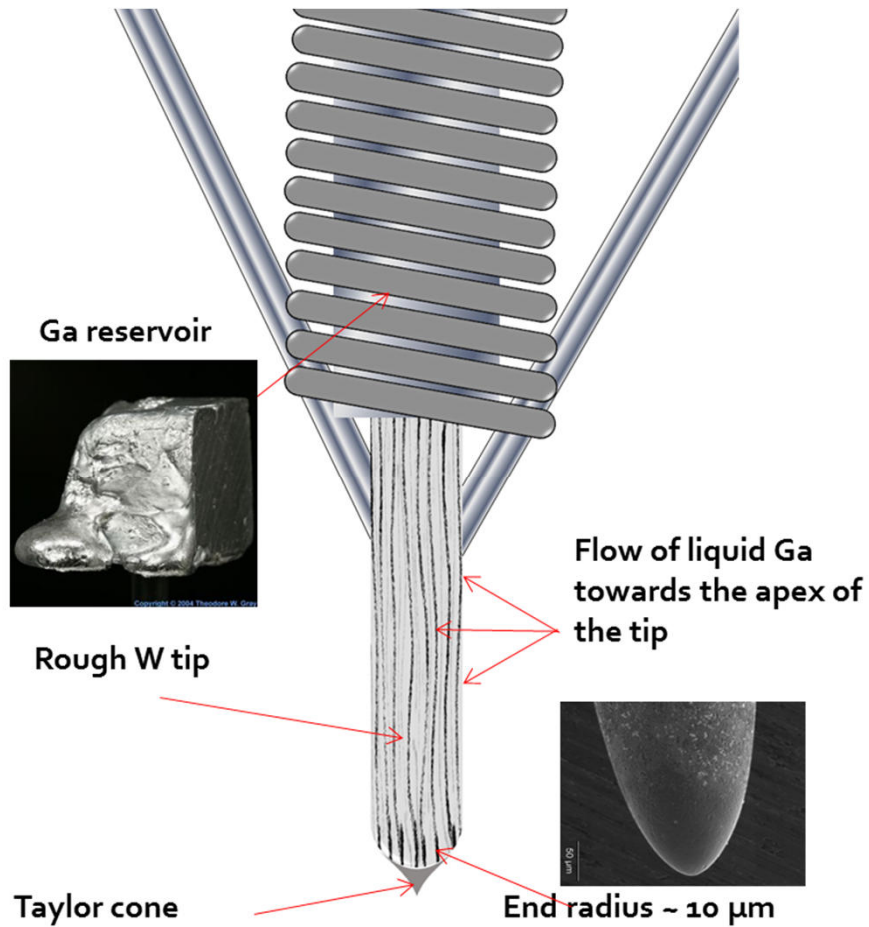
The Taylor cone is the only conical shape which satisfies the condition

$$\frac{1}{2} \epsilon_0 E^2 = 2 \gamma / r$$

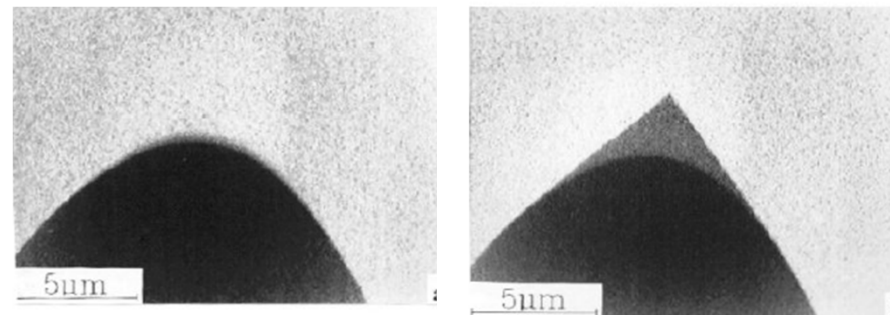




# Liquid Metal Ion Source (LMIS)

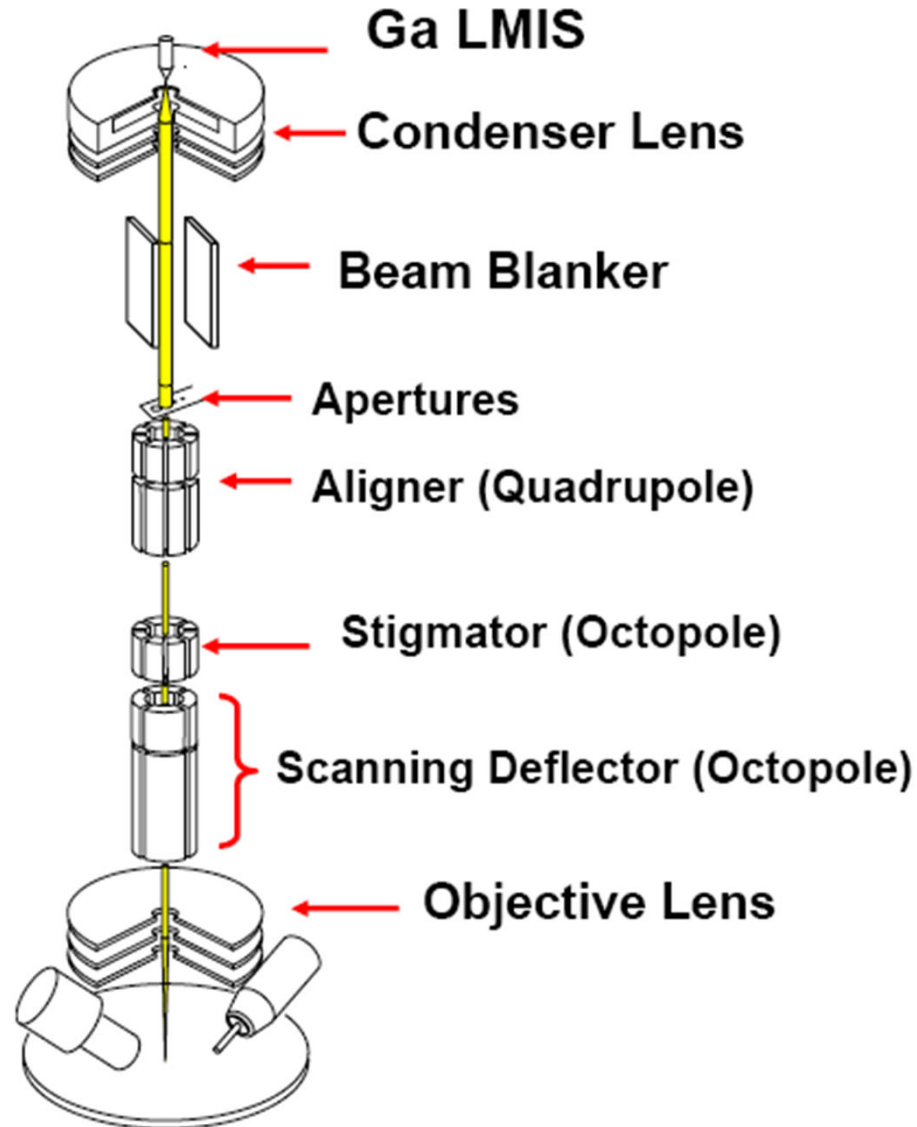


Orsay Physics Ga-LMIS



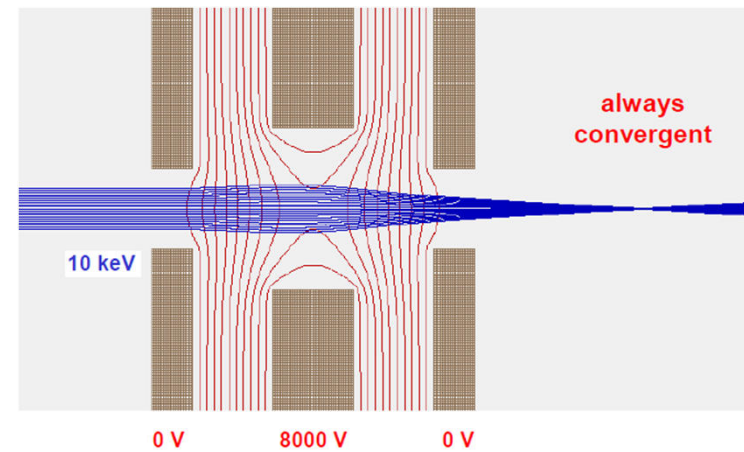
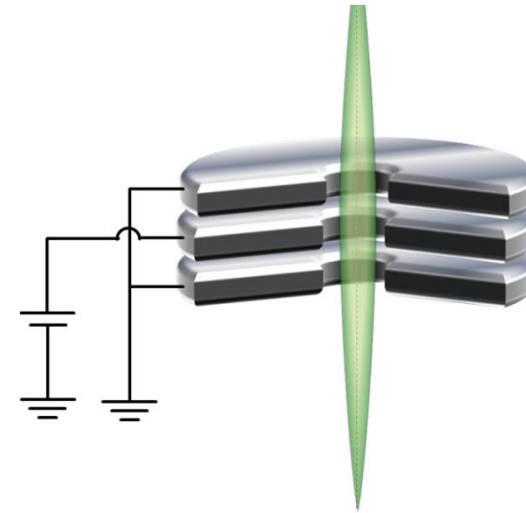
Driesel et al. – MPI Halle 1MeV TEM (1996)

# SII Ion Column



Principle of an electrostatic lens

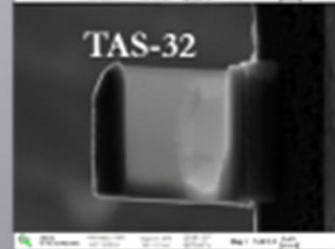
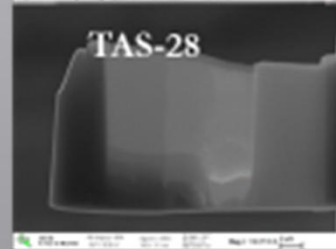
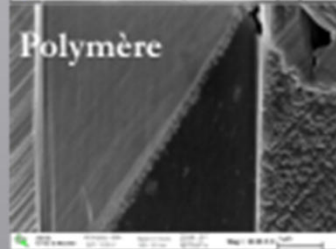
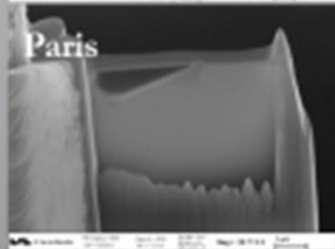
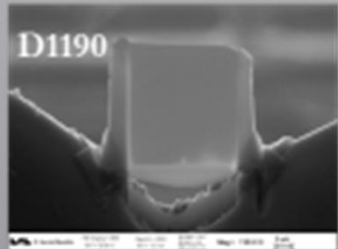
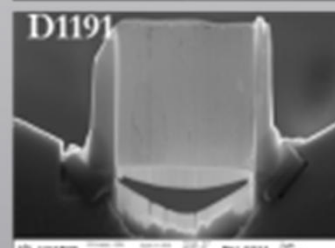
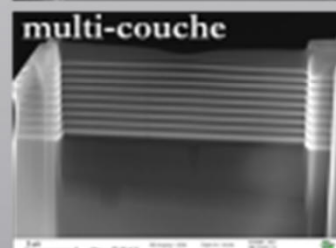
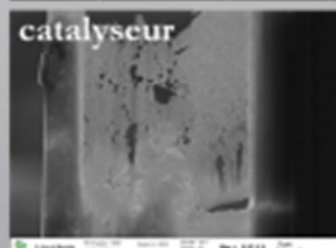
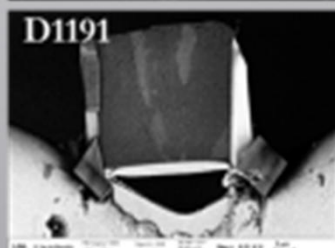
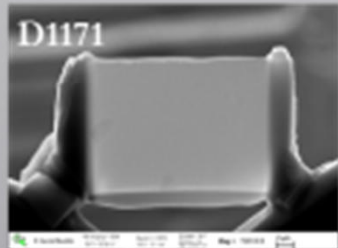
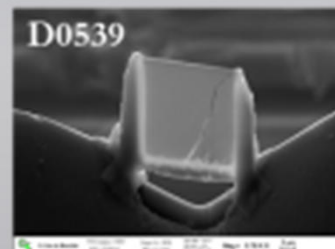
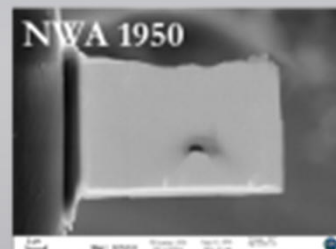
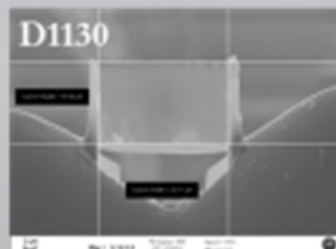
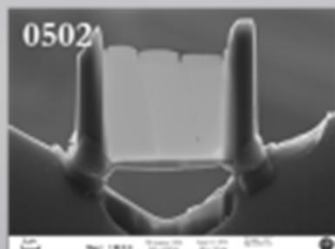
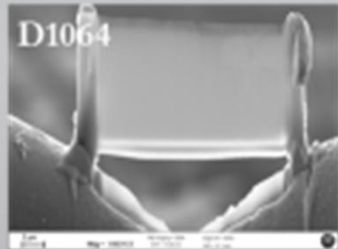
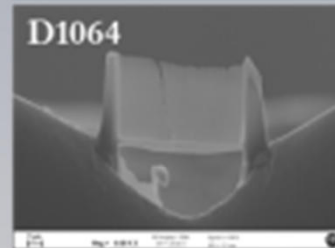
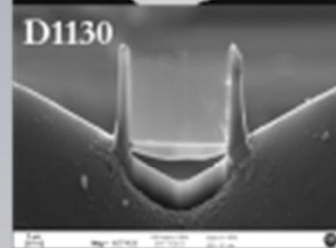
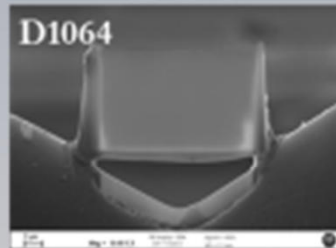
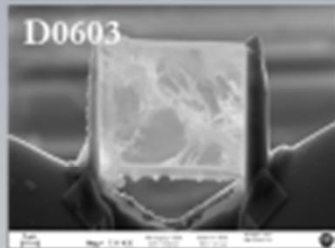
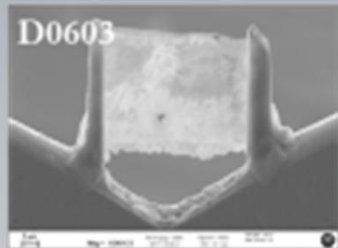
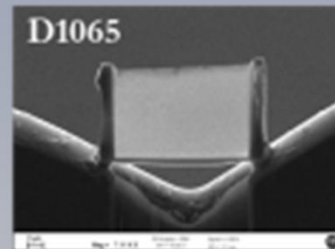
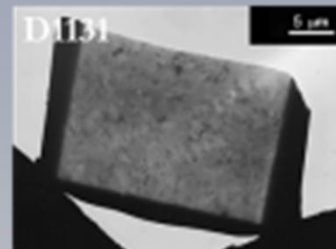
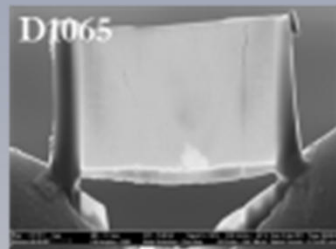
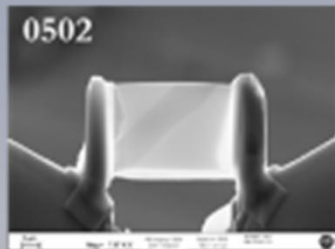
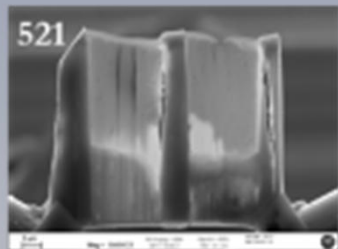
Unipotential or *Einzell* lens



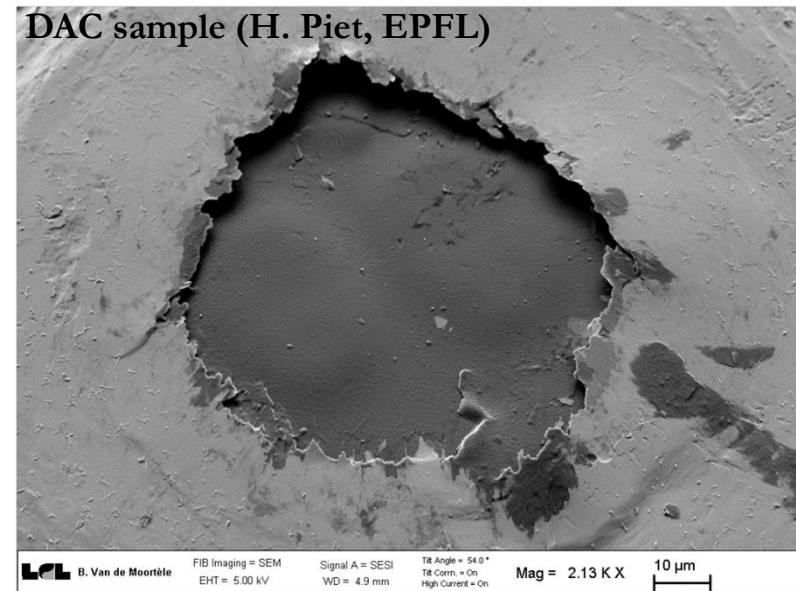
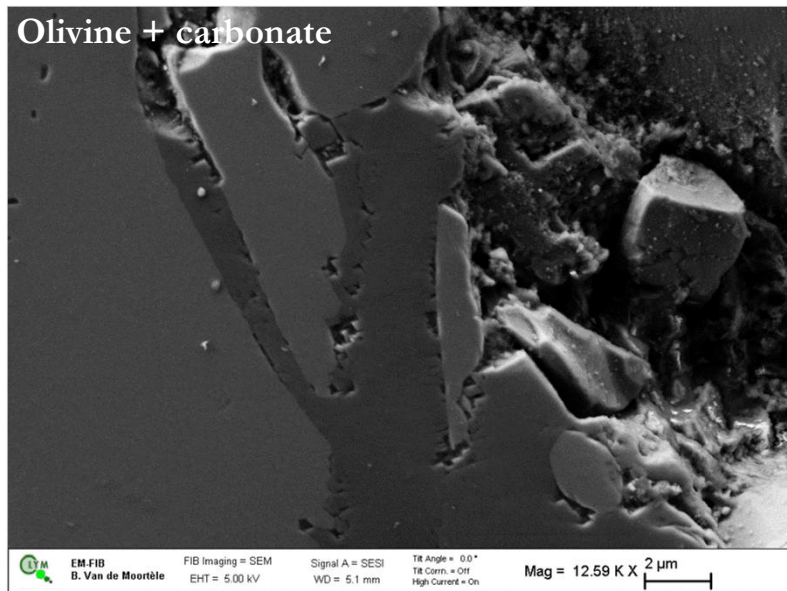
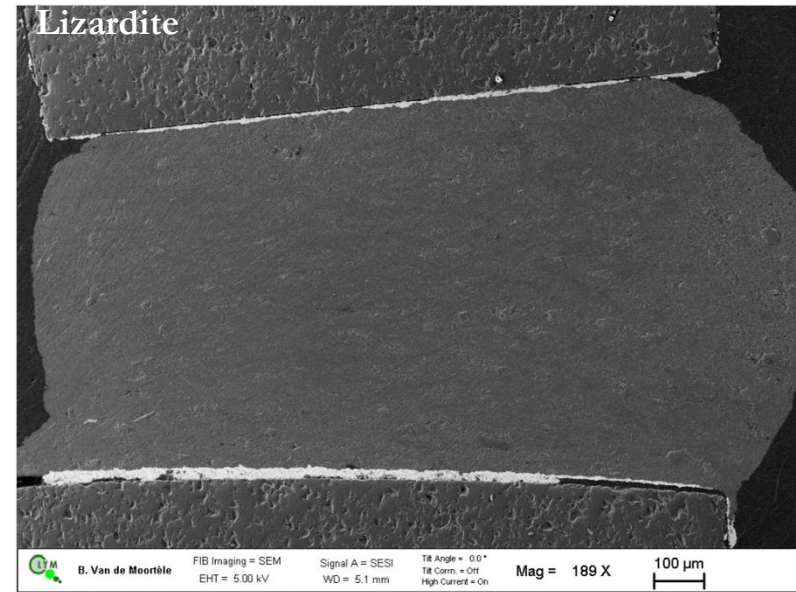
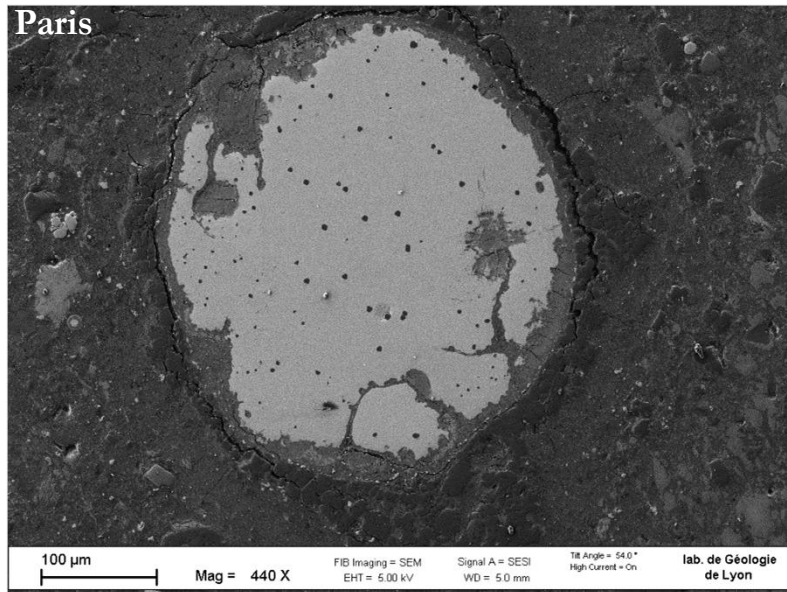
From Rasser, CNRS formation entreprises, Lyon, mars 2013



# TEM Lamella Preparation

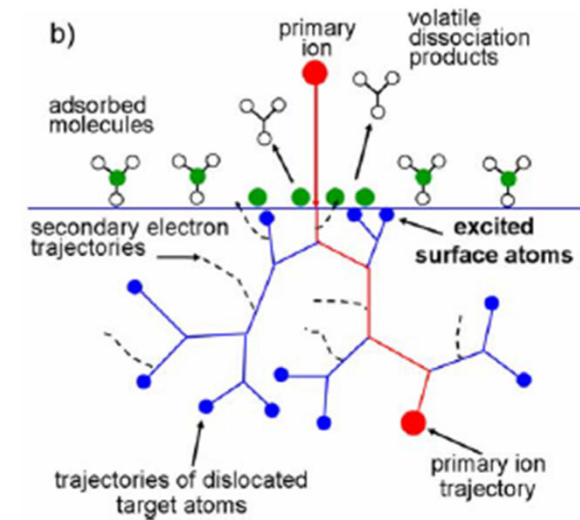
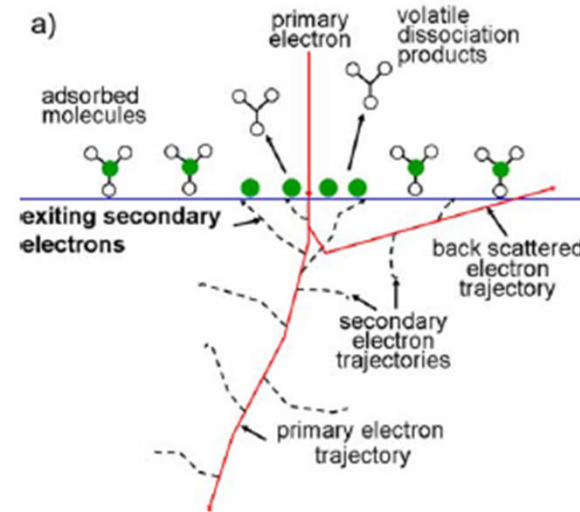
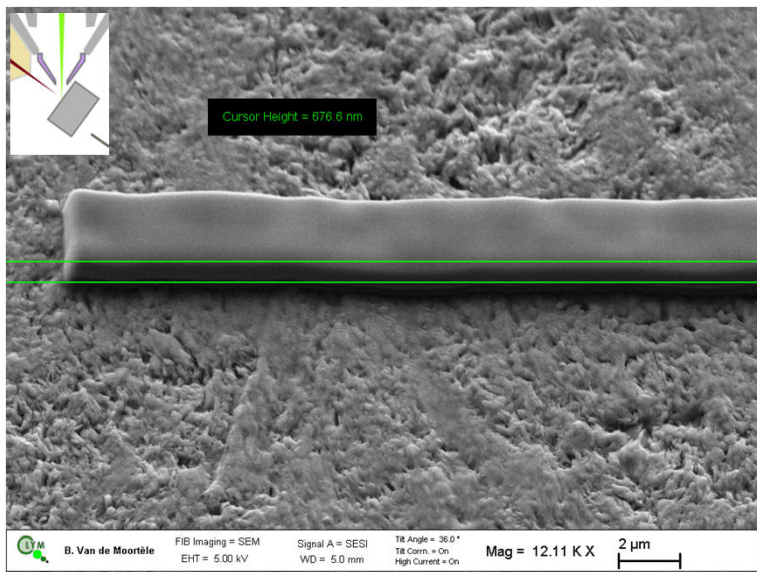
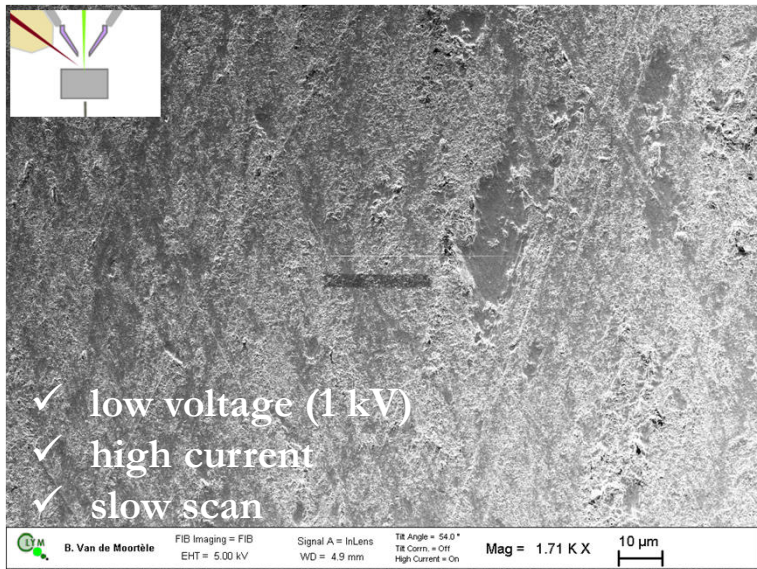


# Why using FIB for preparing TEM lamellae?





# Electron and Ion beam induced deposition



Gas-assisted focused electron beam and ion beam processing and fabrication

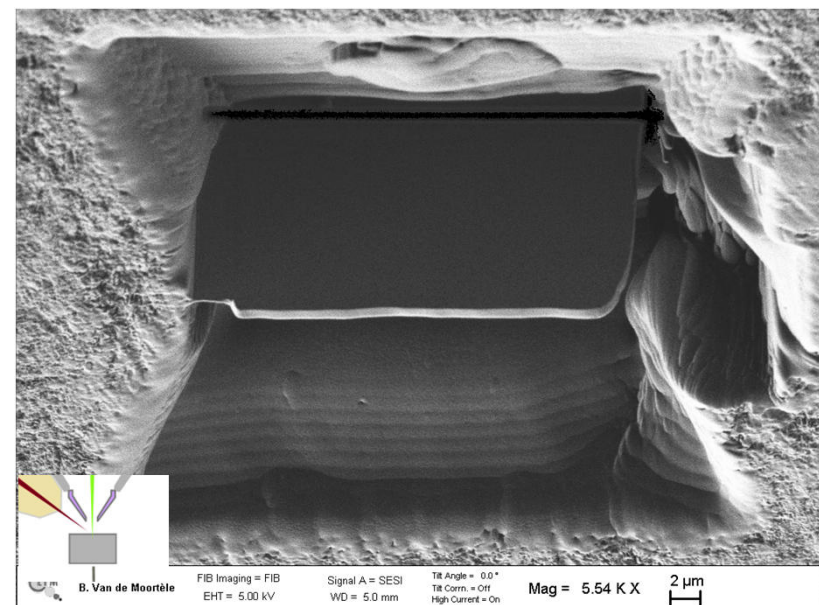
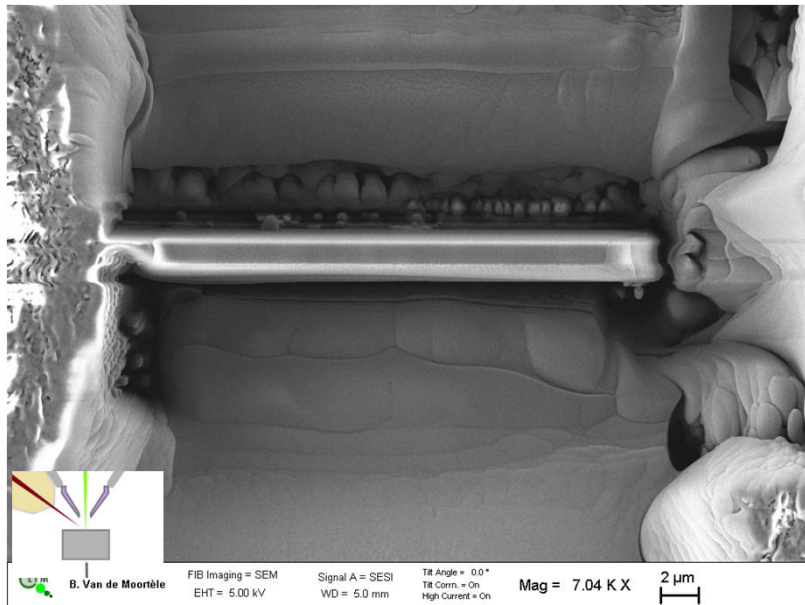
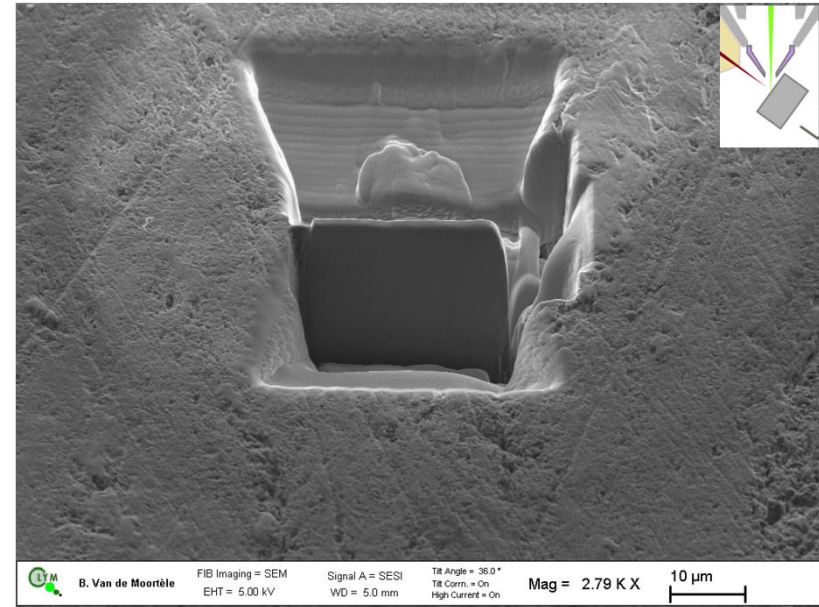
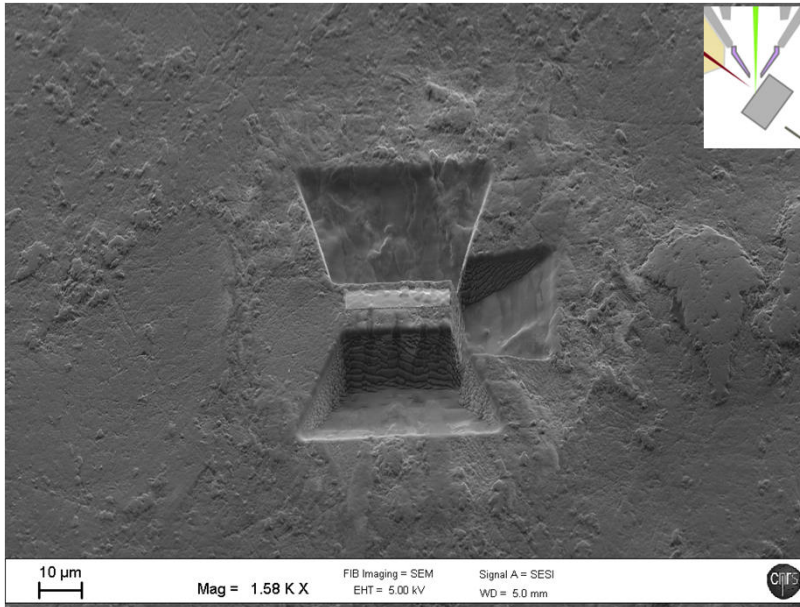
I. Utko

Journal of Vacuum Science & Technology B, Vol.26 No 4, Jul/Aug 2008

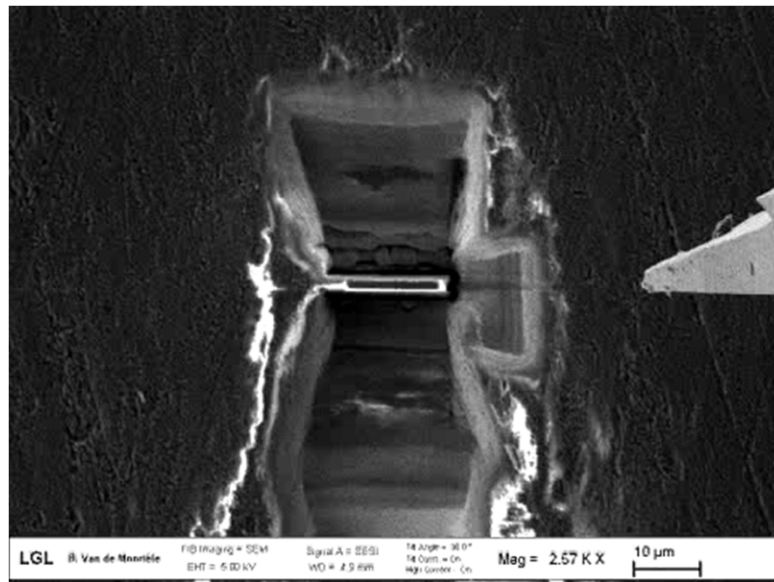
*rmui, Nancy, November 15, 2013*



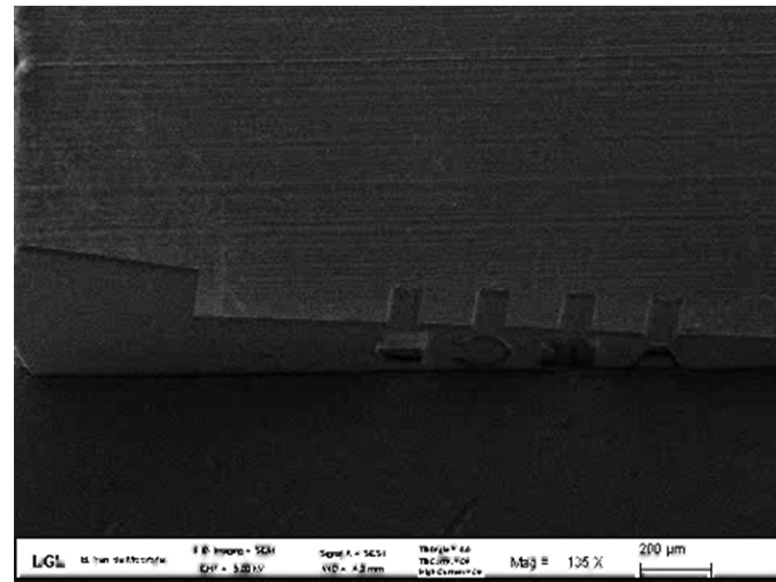
# Main steps



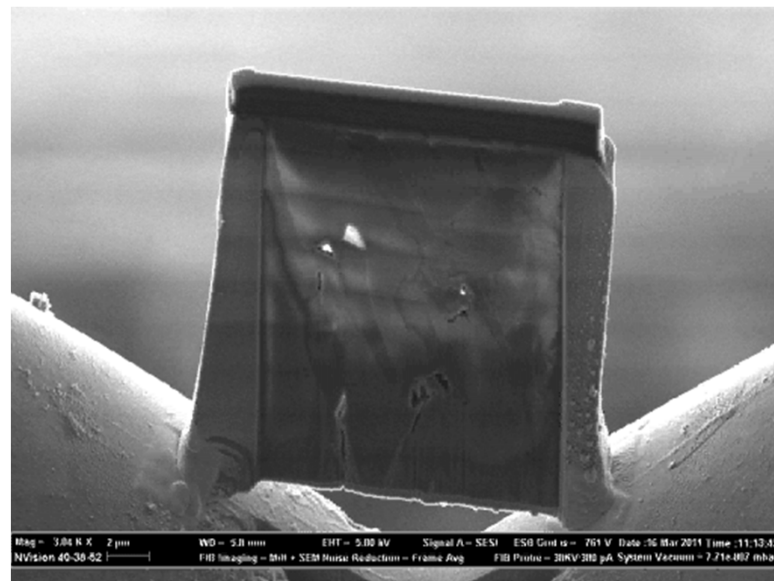
# Lift-out



Lift-out

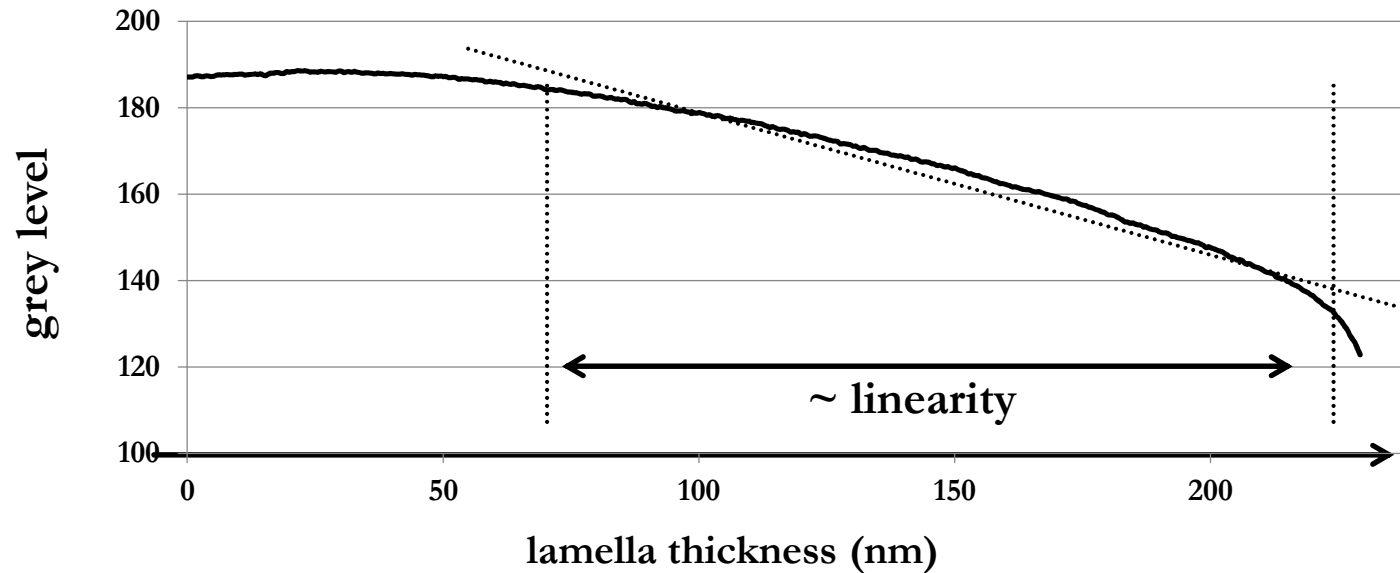
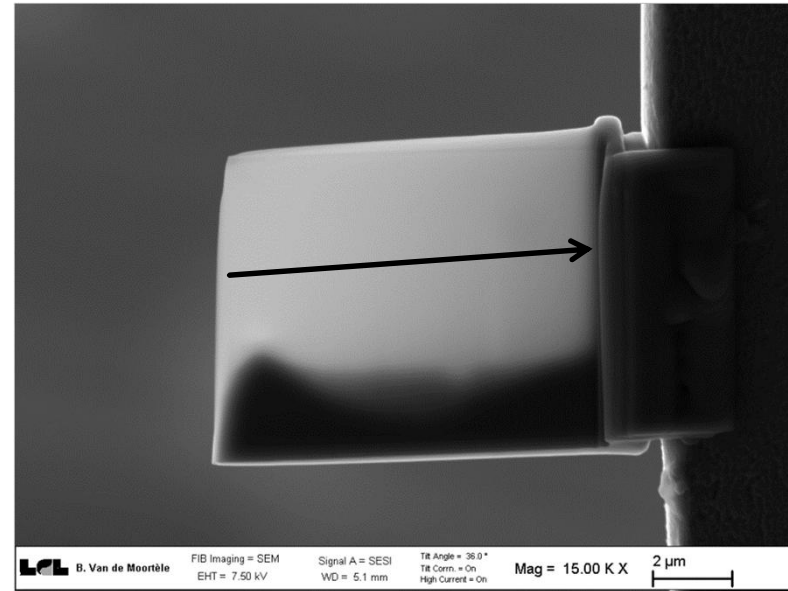
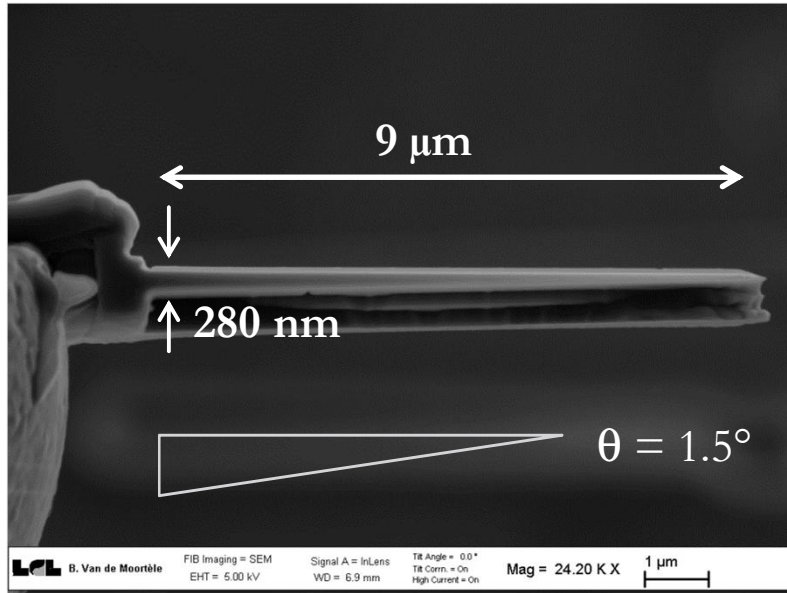


Gluing on copper grid



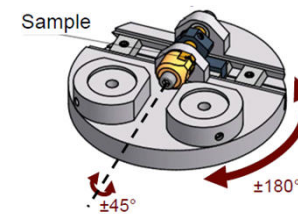
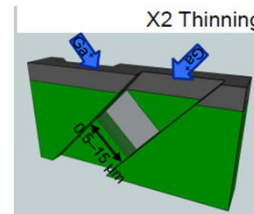
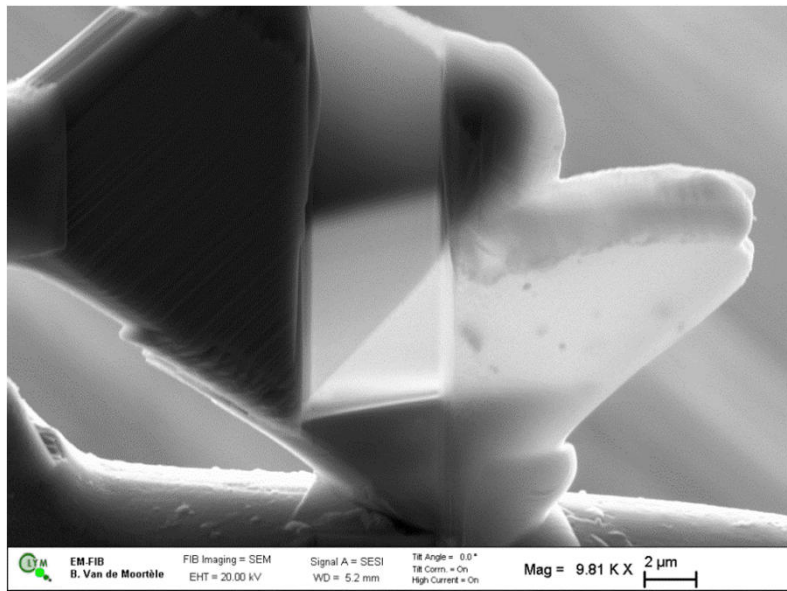
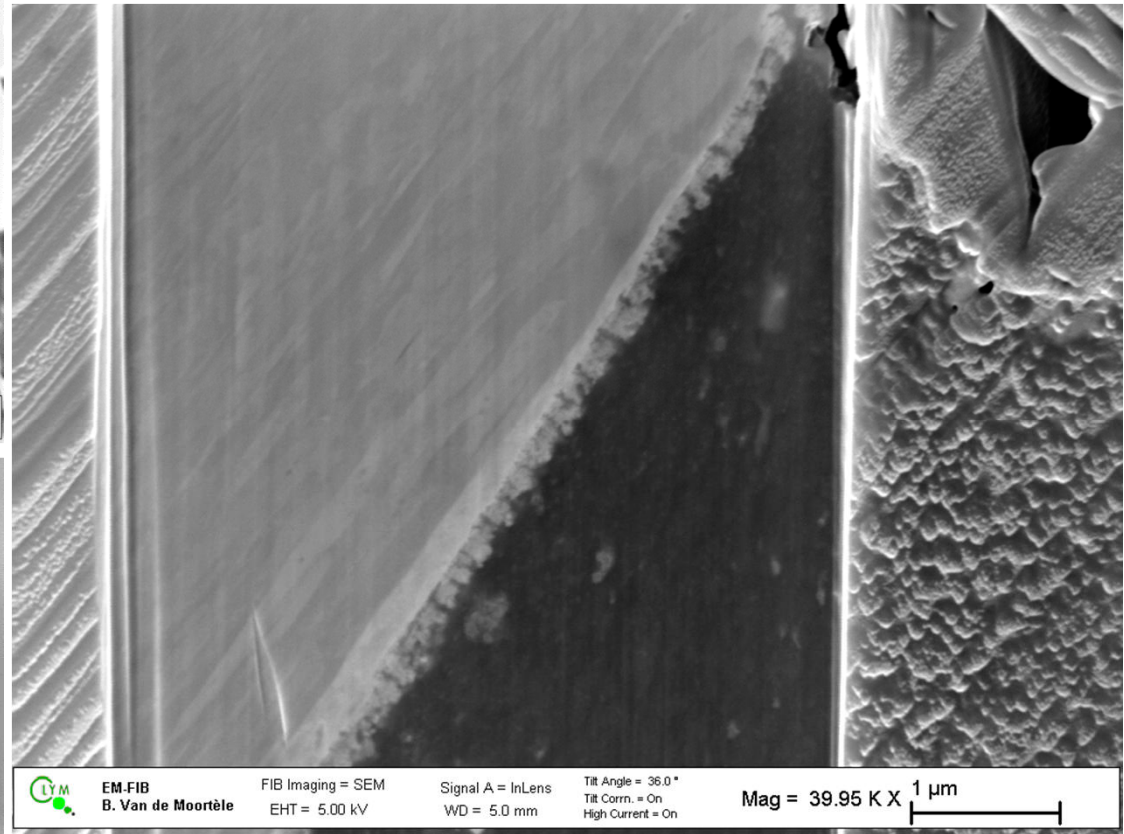
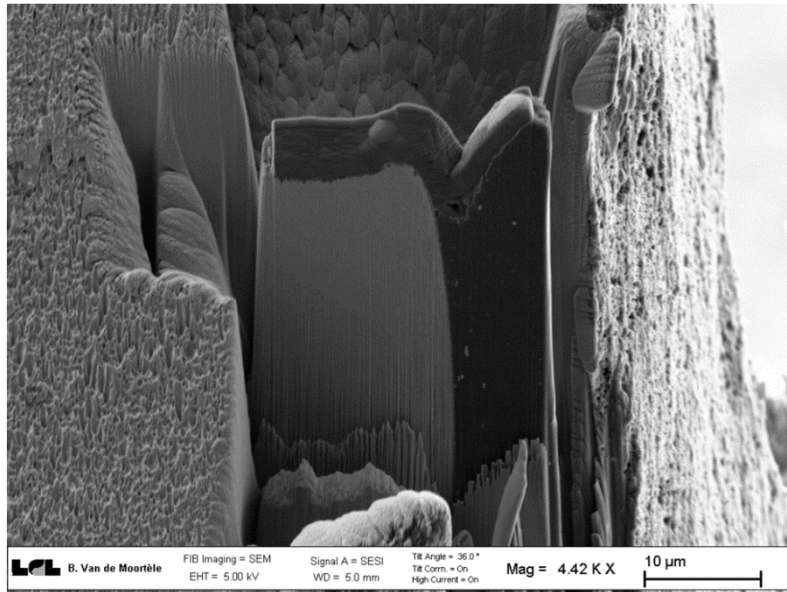
Milling

# Electron transparency





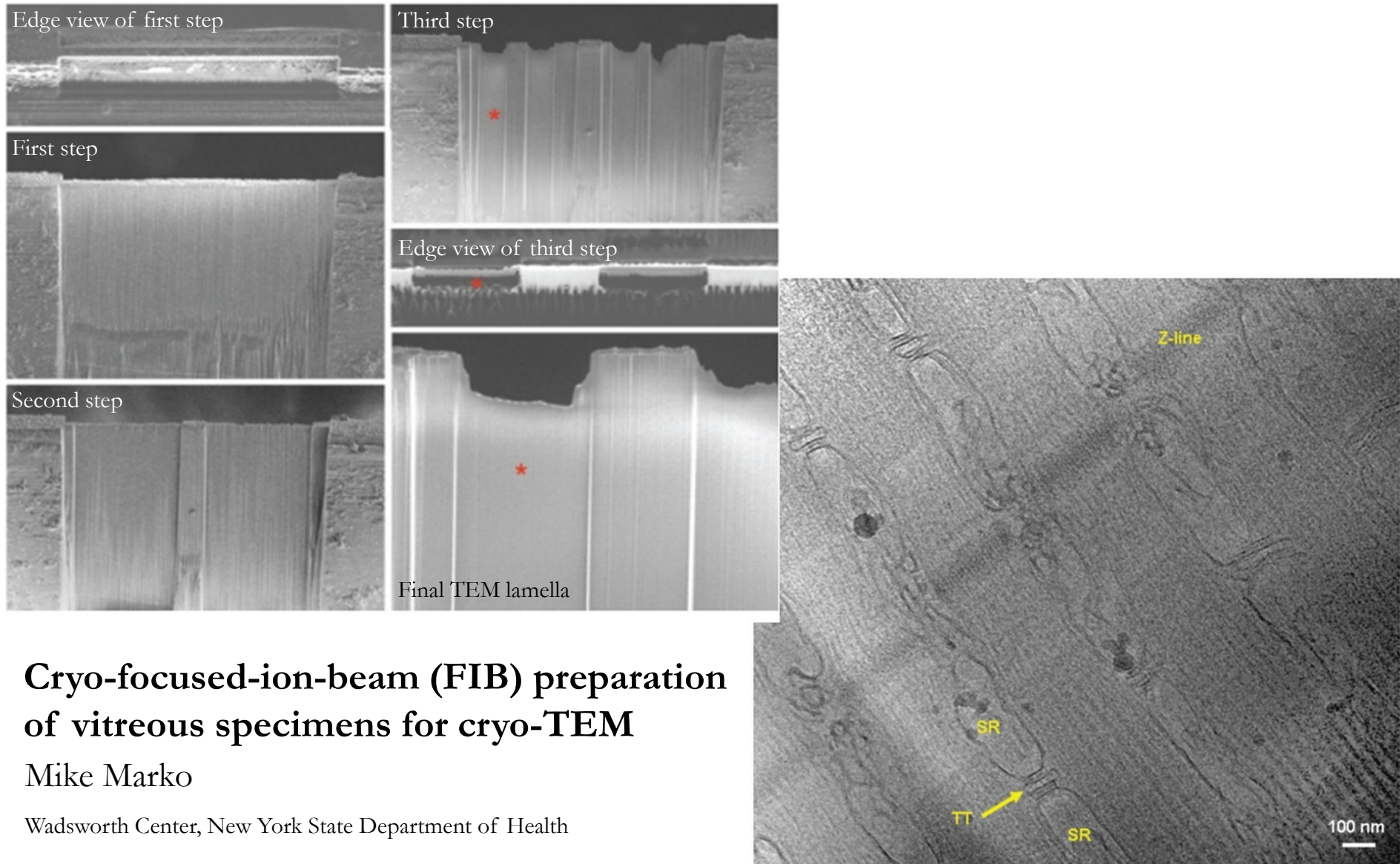
# Soft Material



S. Chalal, Zeiss France, is thanked for giving us the opportunity to use the X2 sample holder

*rmui, Nancy, November 15, 2013*

# Biological sample



## Cryo-focused-ion-beam (FIB) preparation of vitreous specimens for cryo-TEM

Mike Marko

Wadsworth Center, New York State Department of Health





emc2016  
Lyon • France  
www.emc2016.fr

The 16<sup>th</sup> European  
**MICROSCOPY CONGRESS**

Convention Center - 28<sup>th</sup> August - 2<sup>nd</sup> September

Organised by



and Under the auspices of





# FIB/SEM french training

	jour 1		jour 2		jour 3		jour 4		jour 5					
	GR1	GR2	GR1	GR2	GR1	GR2	GR1	GR2	GR1	GR2				
8h00-8h30			FIB-TP3			FIB-TP3								
8h30-9h00								fin manipe-3D						
9h00-9h30														
9h30-10h00	accueil				IF-TP4	FIB-TP4		cours lame TEM	IF-TP2	FIB-TP5				
10h00-10h30	Rappels sur le MEB		IF-TP1+TP3	FIB-TP1+TP2										
10h30-11h00						pause			FIB-TP6					
11h00-11h30							visite du CLYM	IF-TP4			questions ouvertes			
11h30-12h00											EDS-3D ou EBSD-3D			
12h00-12h30			pause déjeuner							pause déjeuner				
12h30-13h00														
13h00-13h30	pause déjeuner													
13h30-14h00			Principes généraux du FIB, B. Rasser		Principes de la 3D, applications en biologie, M. Cantoni				FIB-TP6					
14h00-14h30													EDS-3D ou EBSD-3D	
14h30-15h00	Intéactions ions-matière (+SRIM/TRIM?)													bilan des journées
15h00-15h30														
15h30-16h00														
16h00-16h30			pause											
16h30-17h00	FIB-TP1+TP2	IF-TP1+TP3												
17h00-17h30			FIB-TP4	visite du CLYM										
17h30-18h00														
18h00-18h30														

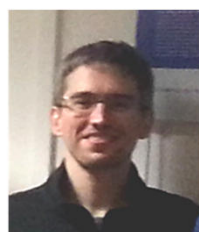
Direction de l'innovation et des relations avec les entreprises

**cnrs formation entreprises**

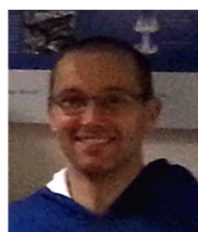


07/04/14 au 11/04/14

	TP-FIB	TP-IF
TP1	usinage (1.5h)	Fiji (2h)
TP2	dépôt (1.5h)	Fiji (2h)
TP3	μmanipulators (1h)	SRIM/TRIM; 1 h
TP4	ionique (1.5h)	ionique (1.5)
TP 5	pointe (2h)	
TP6	lame (2 ou 4h)	



N. Blanchard  
ILM  
UCBL



A. Descamps  
LPEM  
ESPCI



T. Douillard  
MATEIS,  
INSA-Lyon



C. Langlois  
MATEIS  
INSA-Lyon



B. Rasser  
Orsay Physics



E. Gautier  
Spintec  
CEA Grenoble



M. Cantoni  
CIME  
EPFL



E. Cadel  
GPM  
Univ Rouen



B. Van de Moortèle  
LGL, ENS de Lyon