FIB

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Focused Ion Beam

a) Principles

How does it work..?

Ion source, optics, interaction with the sample

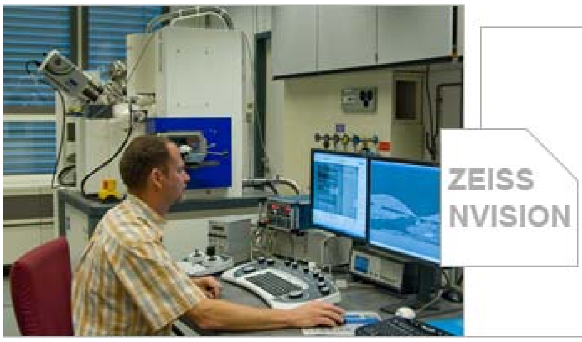
b) Basic Application

Imaging, milling, deposition, typical applications

c) TEM sample preparation, examples

d) FIB Nanotomography, 3D microscopy

FIB @ CIME



Since July 2008

NVISION 40 from CARL ZEISS

Dual Beam = SEM + FIB

SEM: Schottky thermal field emitter

FIB: Ga LMIS

4 Gas Injector Systems
- Pt deposition (C9H16Pt)
- C deposition
- SiO2 deposition (TEOS)
- Insulator Enhanced Etch (XeF2)
- Selective Carbon Mill (MgSO4)

2 Kleindiek Micromanipulator (in situ TEM lamella lift out)

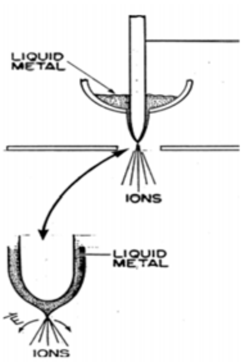
Focused Ion Beam

Mainly developed in 1970's and 80's (Escovitz, Levi-Setti, Orloff, Swanson…)

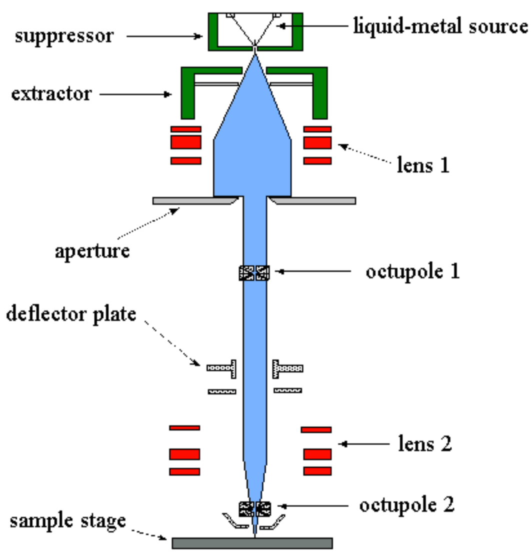
Ion column structure similar to that of SEM

Source: Liquid Metal Ion Source (LMIS).
Ex: Ga, Au, Be, Si, Pd, B, P, As, Ni, Sb, alloys …

Principle:
A strong electromagnetic field causes the emission of positively charged ions



SIM = Scanning Ion Microscope



Schematic diagram of a FIB ion column
Source: IBM Almaden Research Center

Why use ions instead of electrons?

Electrons

are very small
inner shell reactions
High penetration depth
Low mass -> higher speed for given energy
Electrons are negative
Magnetic lens (Lorentz force)

Ions

Big
->outer shell reactions (no x-rays)
High interaction probability
less penetration depth
Ions can remain trapped -> doping
High mass -> slow speed but high momentum
milling !!!
Ions are positive
Electrostatic lenses

Why Gallium ?

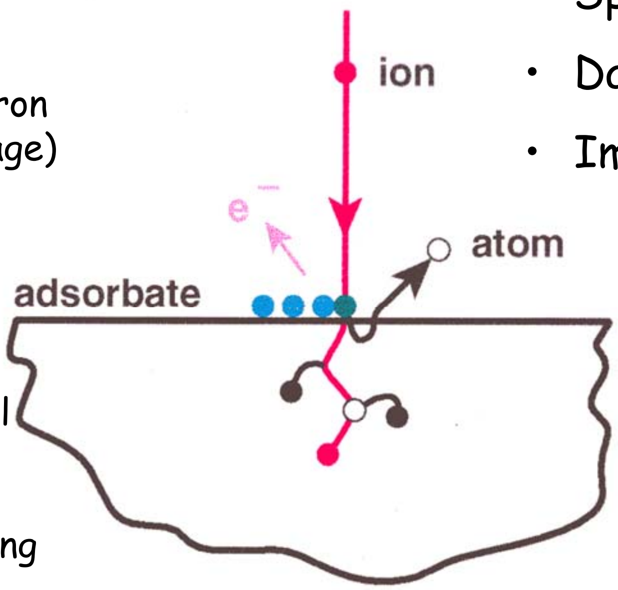
Ga is metallic, low melting point, in the middle of the periodic table, no overlap with other elements in EDX

comparison

		FIB	SEM	Ratio
Particle	type	Ga+ ion	electron	
	elementary charge	+1	-1	
	particle size	0.2 nm	0.00001 nm	20’000
	mass	1.2 .10-25 kg	9.1.10-31 kg	130’000
	velocity at 30 kV	2.8.105 m/s	1.0 108 m/s	0.0028
	velocity at 2 kV	7.3.104 m/s	2.6.107 m/s	0.0028
	momentum at 30 kV	3.4.10-20 kgm/s	9.1.10-23 kgm/s	370
	momentum at 2 kV	8.8.10-21 kgm/s	2.4.10-23 kgm/s	370
Beam	size	nm range	nm range	
	energy	up to 30 kV	up to 30 kV	
	current	pA to nA range	pA to uA range	
Penetration depth	In polymer at 30 kV	60 nm	12000 nm	
	In polymer at 2 kV	12 nm	100 nm	
	In iron at 30 kV	20 nm	1800 nm	
	In iron at 2 kV	4 nm	25 nm	
Average electrons signal per 100 particles at 20 kV	**secondary electrons**	**100 - 200**	**50 - 75**	
	back scattered electron	**0**	**30 - 50**	
	substrate atom	**500**	**0**	
	secondary ion	**30**	**0**	
	x-ray	**0**	**0.7**	

Ion Solid interaction

- Secondary electron emission (SE-image) 2-3 SE per Ion !
- Surface chemical reactions
  - deposition
  - enhanced etching



- Sputtering
- Damage
- Implantation

3 basic "operating modes"

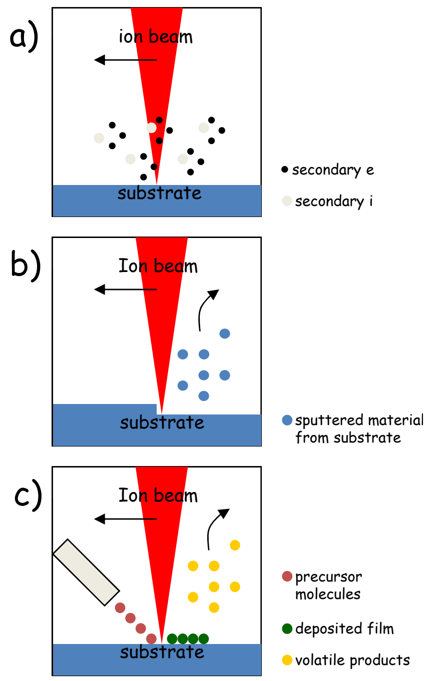
Emission of secondary ions and electrons
- FIB **imaging** ☛ a)
  low ion current

Sputtering of substrate atoms
- FIB **milling** ☛ b)
  high ion current

Chemical interactions (gas assisted)
- FIB **deposition**
- Enhanced (preferrential) etching ☛ c)

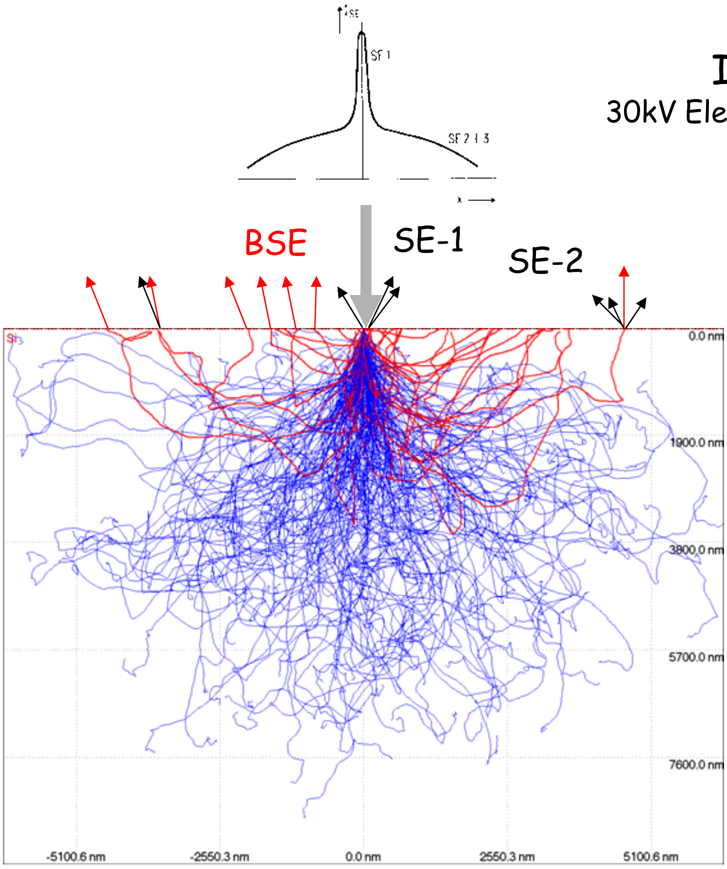
Other effects:
Ion implantation
Displacement of atoms in the solid
- Induced damage

Emission of phonons
- Heating

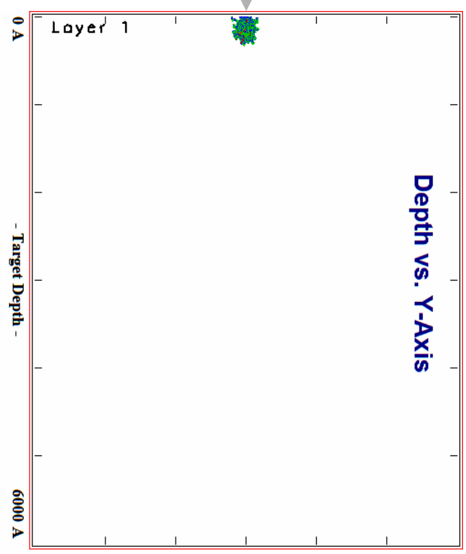


Imaging

30kV Electrons vs Ga Ions

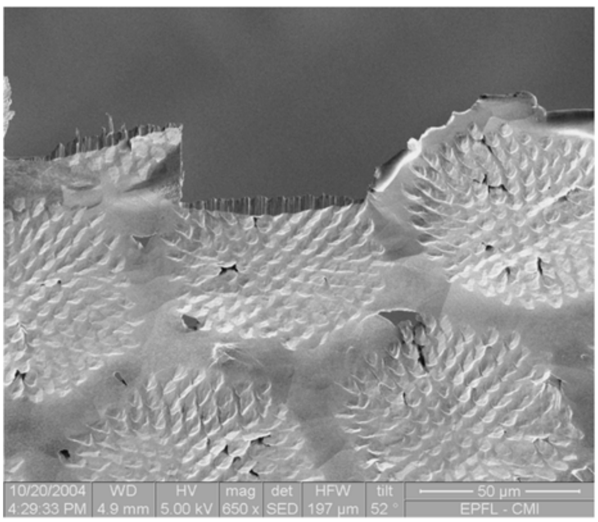


Monte-Carlo Simulation casino v2.42
http://www.gel.usherbrooke.ca/casino/download2.html

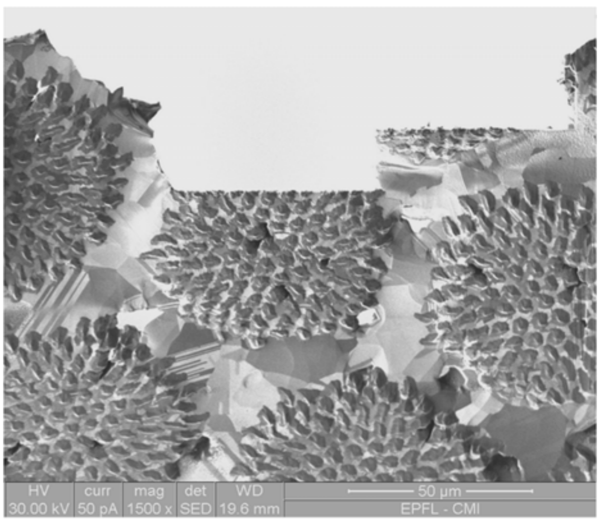


SRIM 2006
http://www.srim.org/

SE image contrast

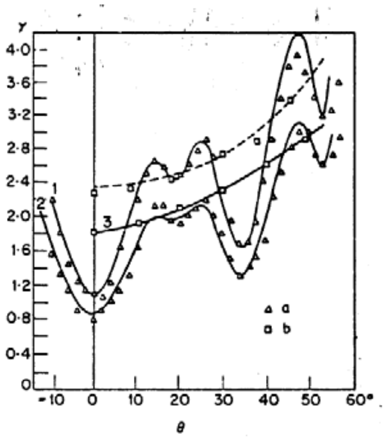


e-beam 5kV



ion-beam 30kV 50pA

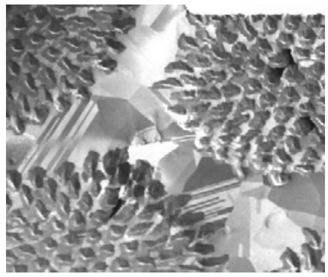
material (sputtering) contrast
orientational contrast

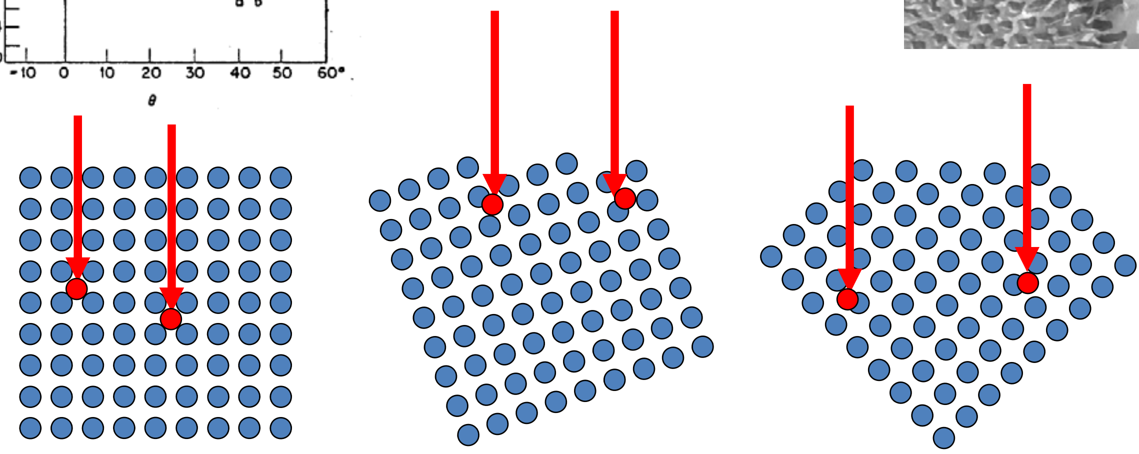


Channeling contrast

Secondary Electron Emission Coefficient vs. Angle, (100) Copper 30 keVAr Ions

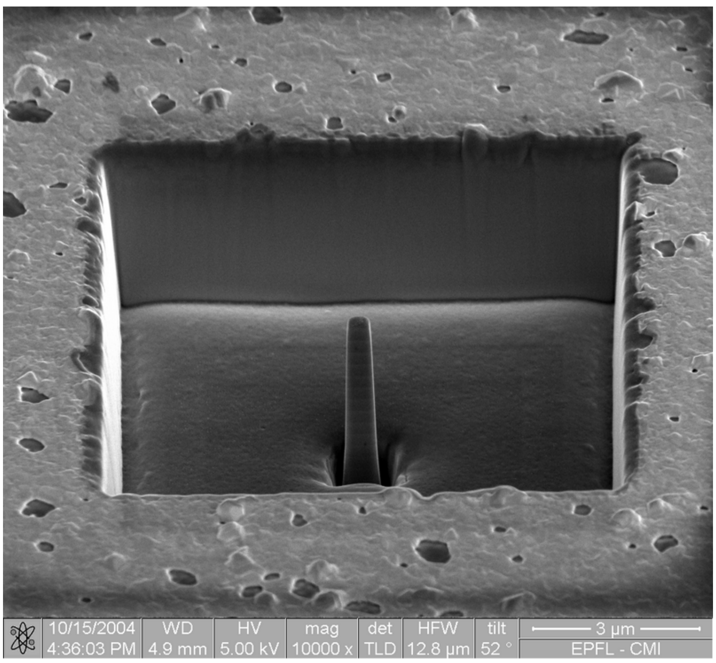
G. Carter and J.S. Colligan, *Ion Bombardment of Solids*, (Elsevier 1968)





Atom columns align with the ion trajectory = higher penetration
-> less sputtering and less SE electrons

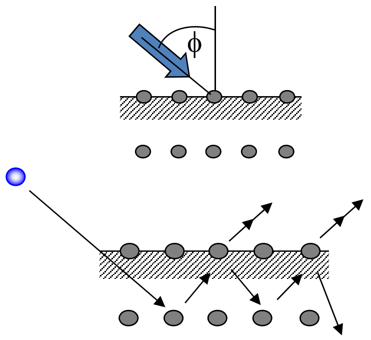
Milling



PZT-high aspect ratio „capacitor", W. Adachi (EPFL-LC)

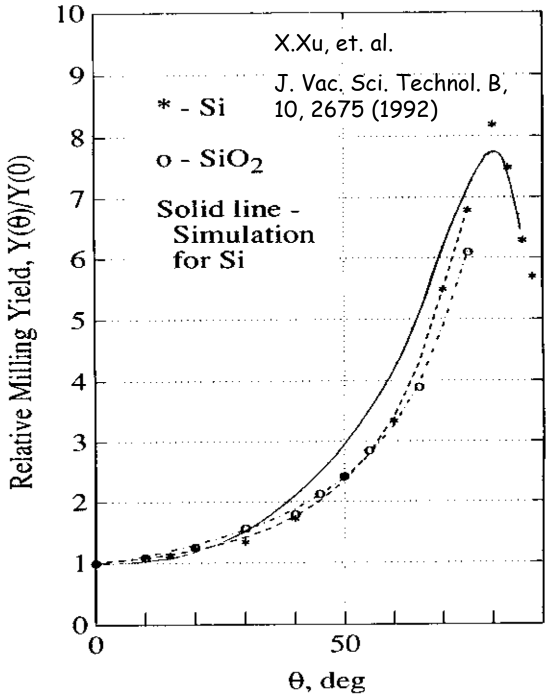
Ion-Solid interaction
Sputtering Yield

Sputtering yield depends on incident angle ϕ

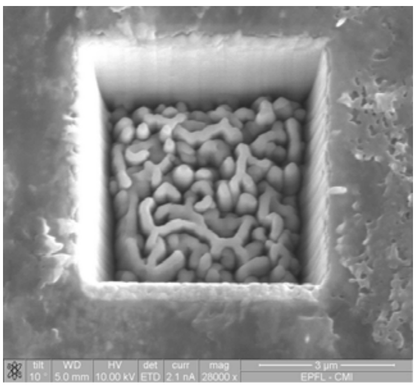


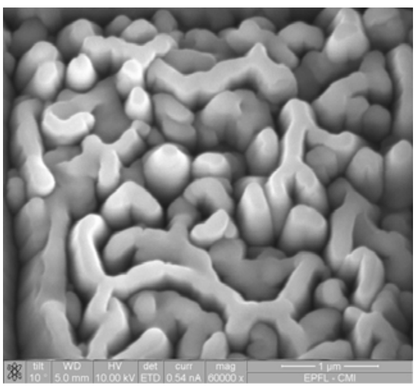
Higher probability of collision cascades near the surface at higher ϕ

Sputtering yield has maximum for ϕ = 75°

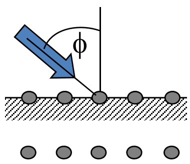


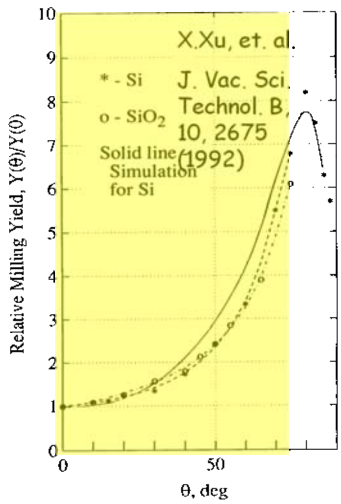
Milling

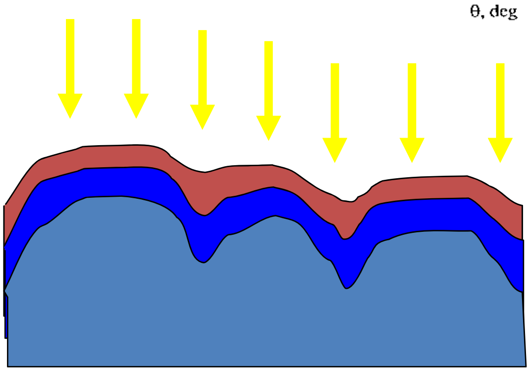




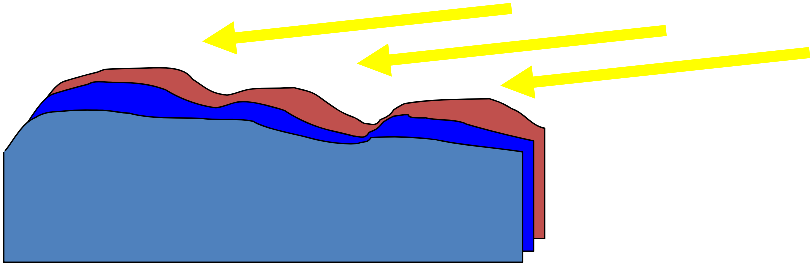
FIB milling of steel

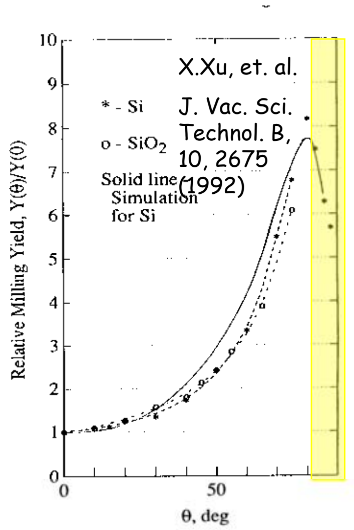


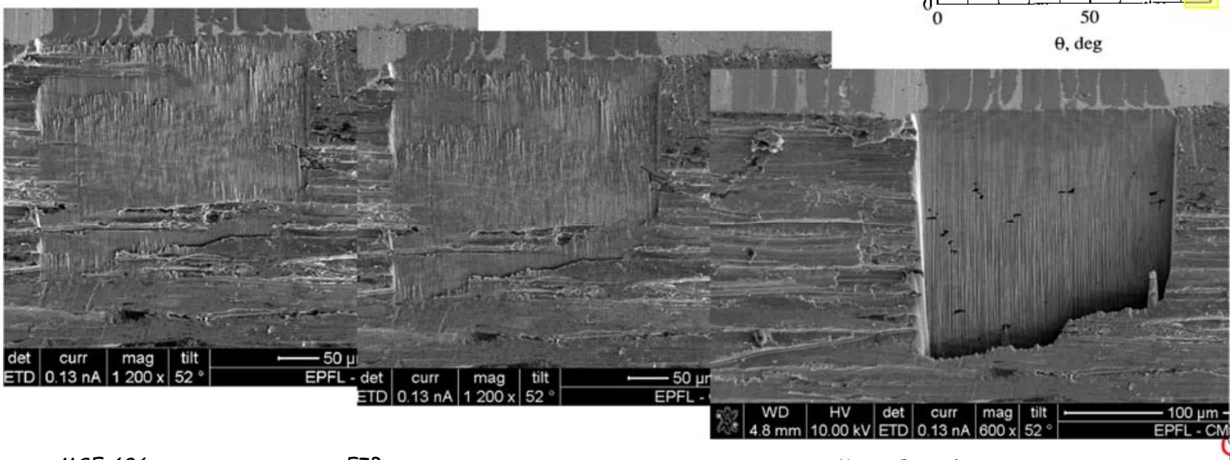




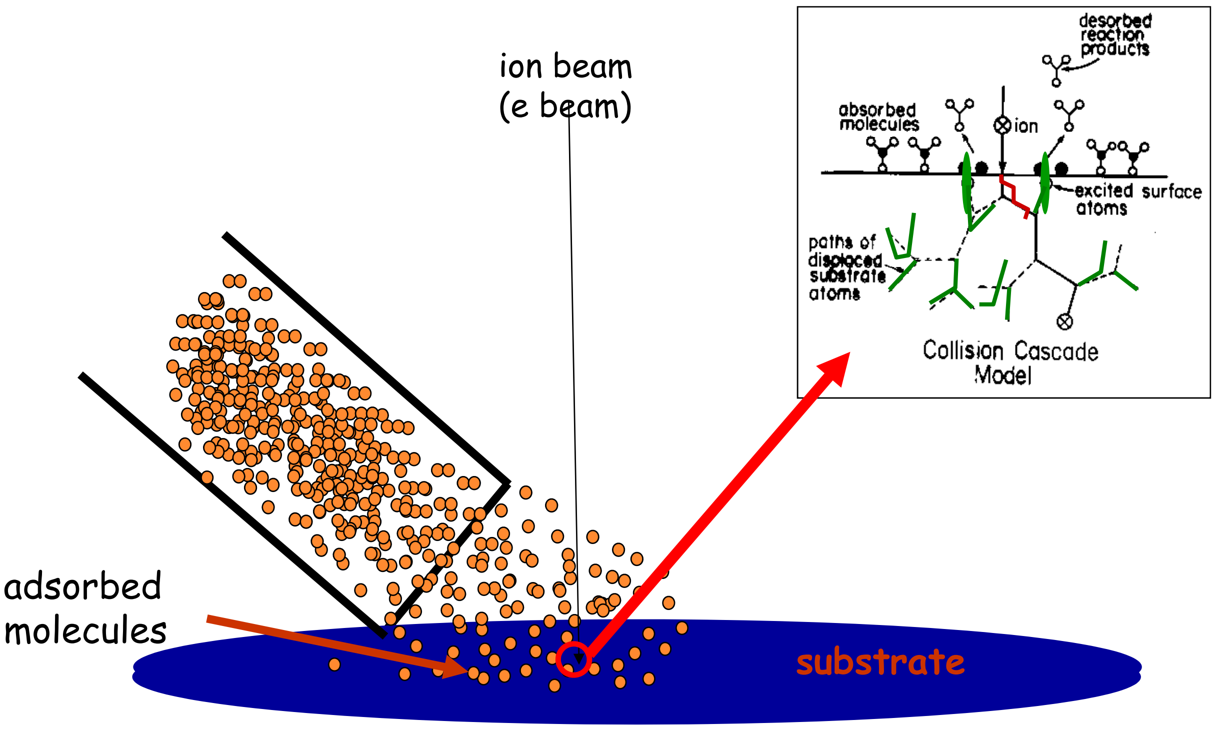
Polishing,
at shallow angles



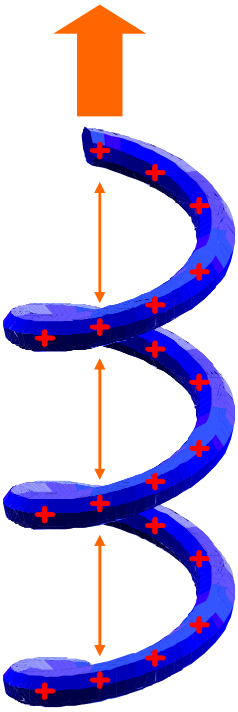


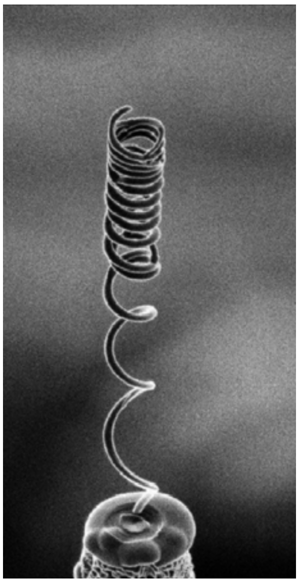


Gas assisted deposition

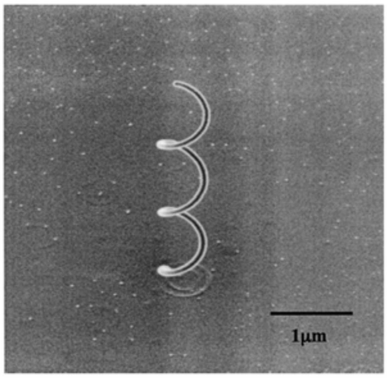


Nanofabricated structures

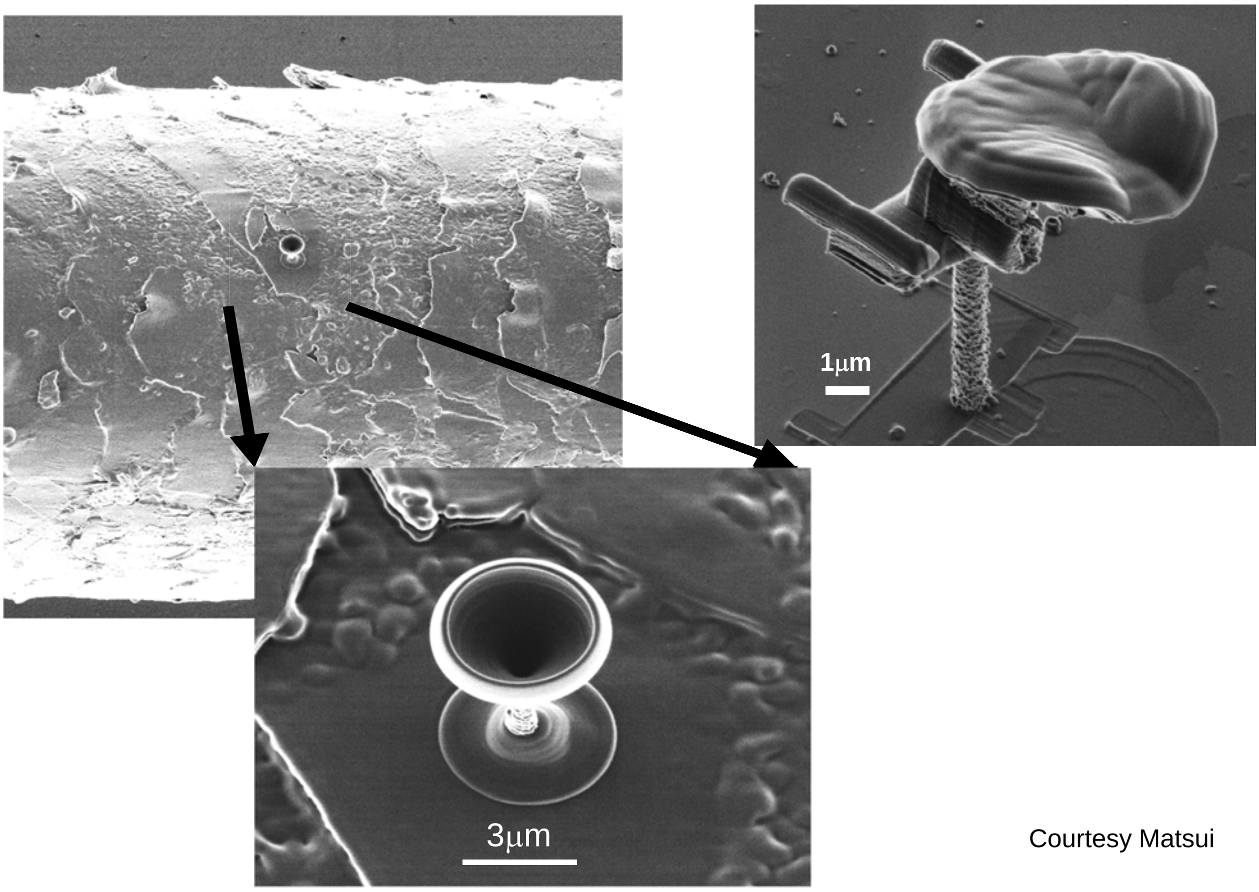




Coil 700nm pitch, 80nm line width, diamond-like amorphous carbon, FIB induced CVD



Shinji Matsui, et.al.
J. Vac. Sci. TechnolB18, 3181
(Nov/Dec, 2000)
(HimejiInstitute of Technology,Hyogo, Japan)



Courtesy Matsui

b) Basic Applications

"Industrial" applications (semiconductor industry)

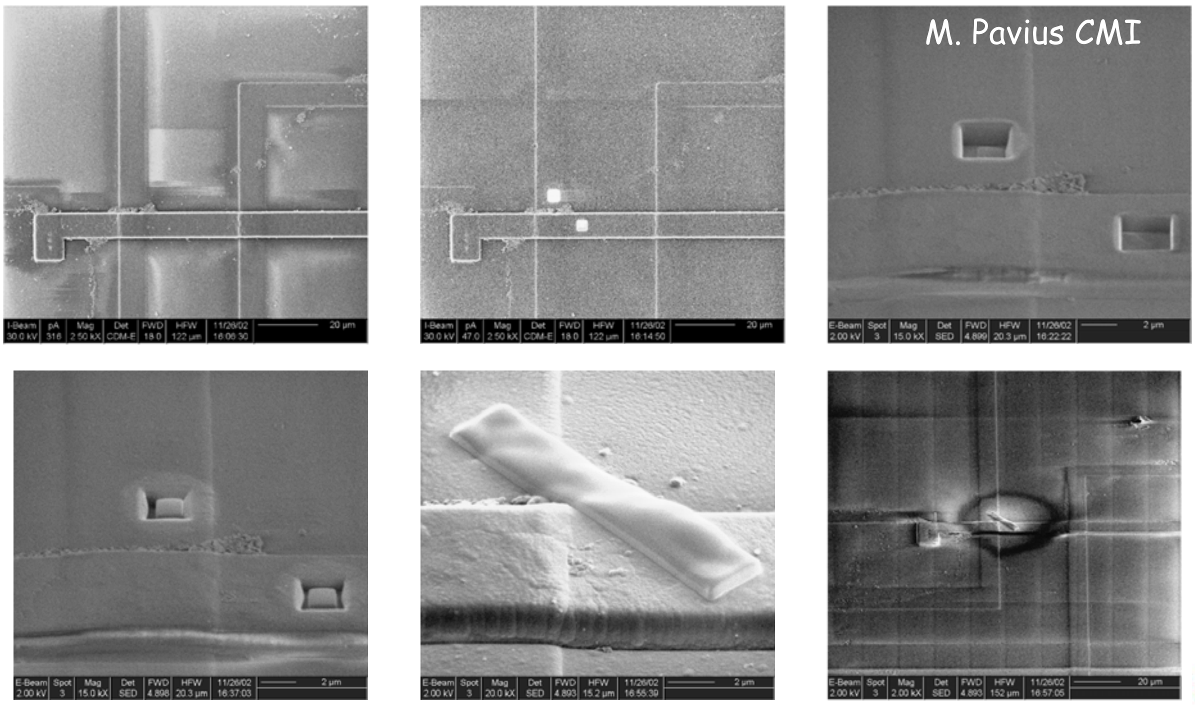
sectioning for failure analysis
prototype circuit rewiring
mask repair
TEM sample preparation

Research

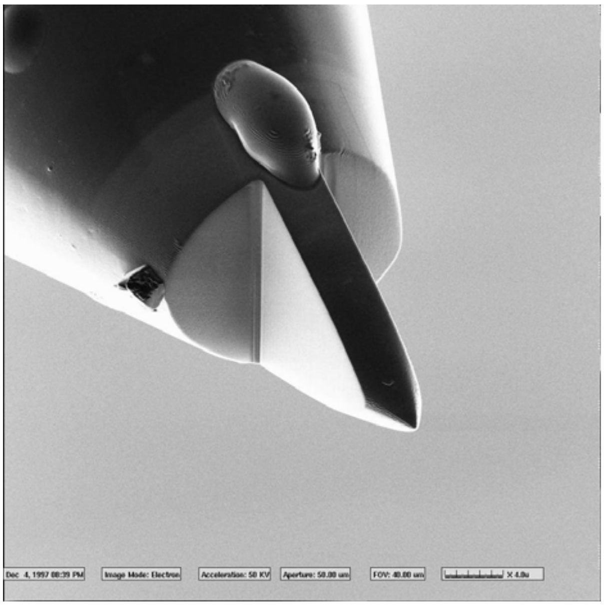
Micromachining
Nanofabricated structures
TEM sample preparation

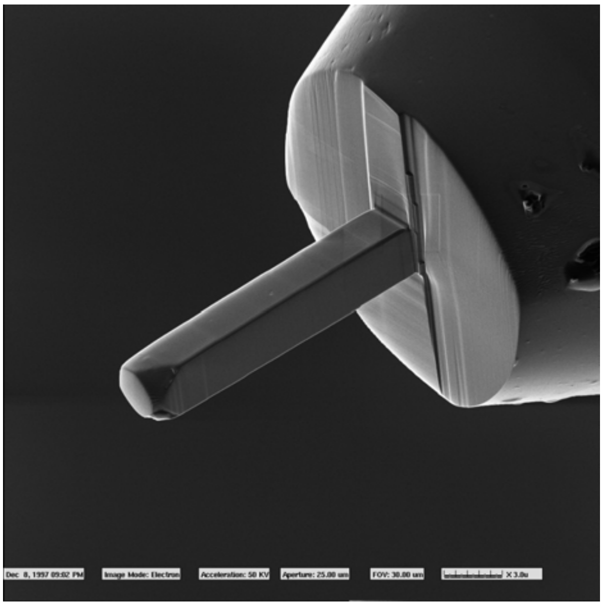
Applications Chip Modification

Insertion of electrical connection: 1) Removal of isolating layer (milling)
2) Pt deposition (FIB deposition)



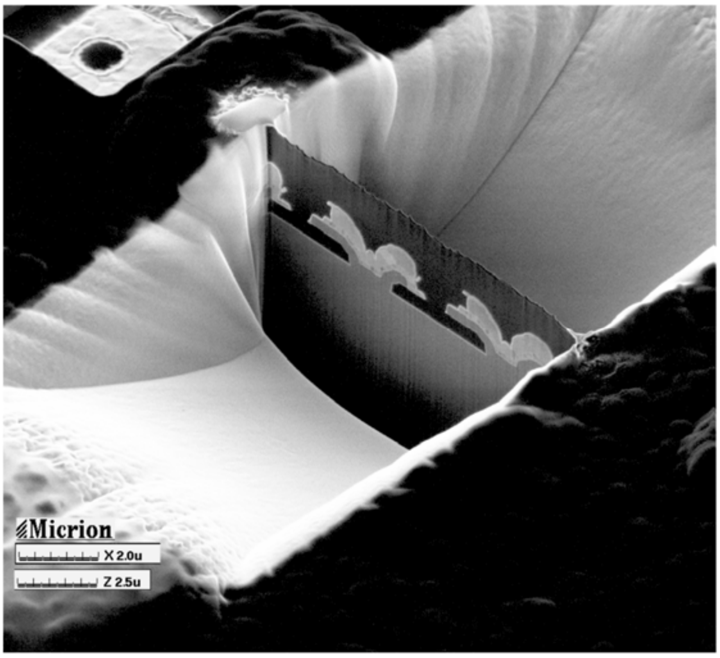
FIB-manufactured AFM-tips



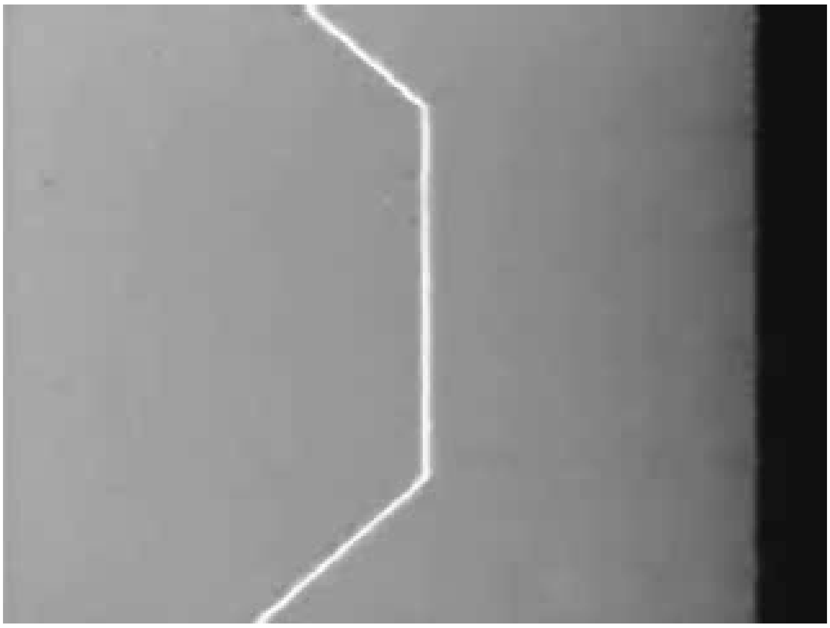


Failure analysis

FIB cross-sectioning
and SEM imaging



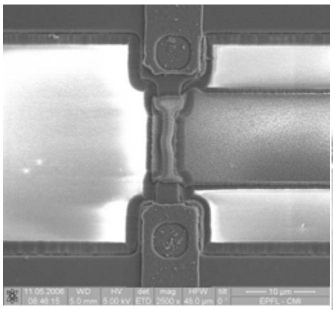
c) TEM preparation
in-situ lift-out movie

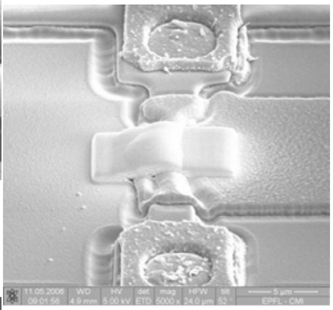


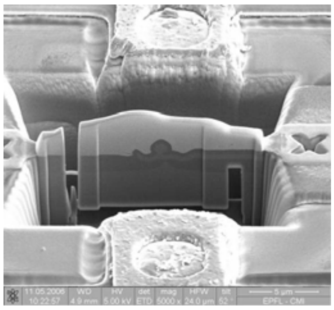
(downloaded from http://www.feicompany.com/

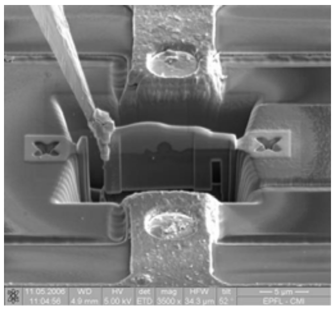
Si nano-wire

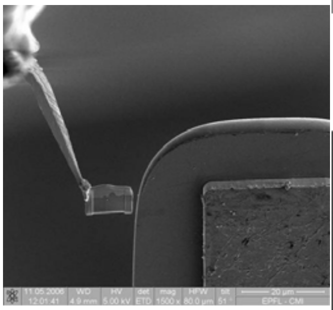
M. Pavius, V. Pott, CMI

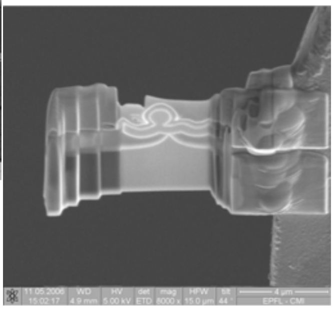


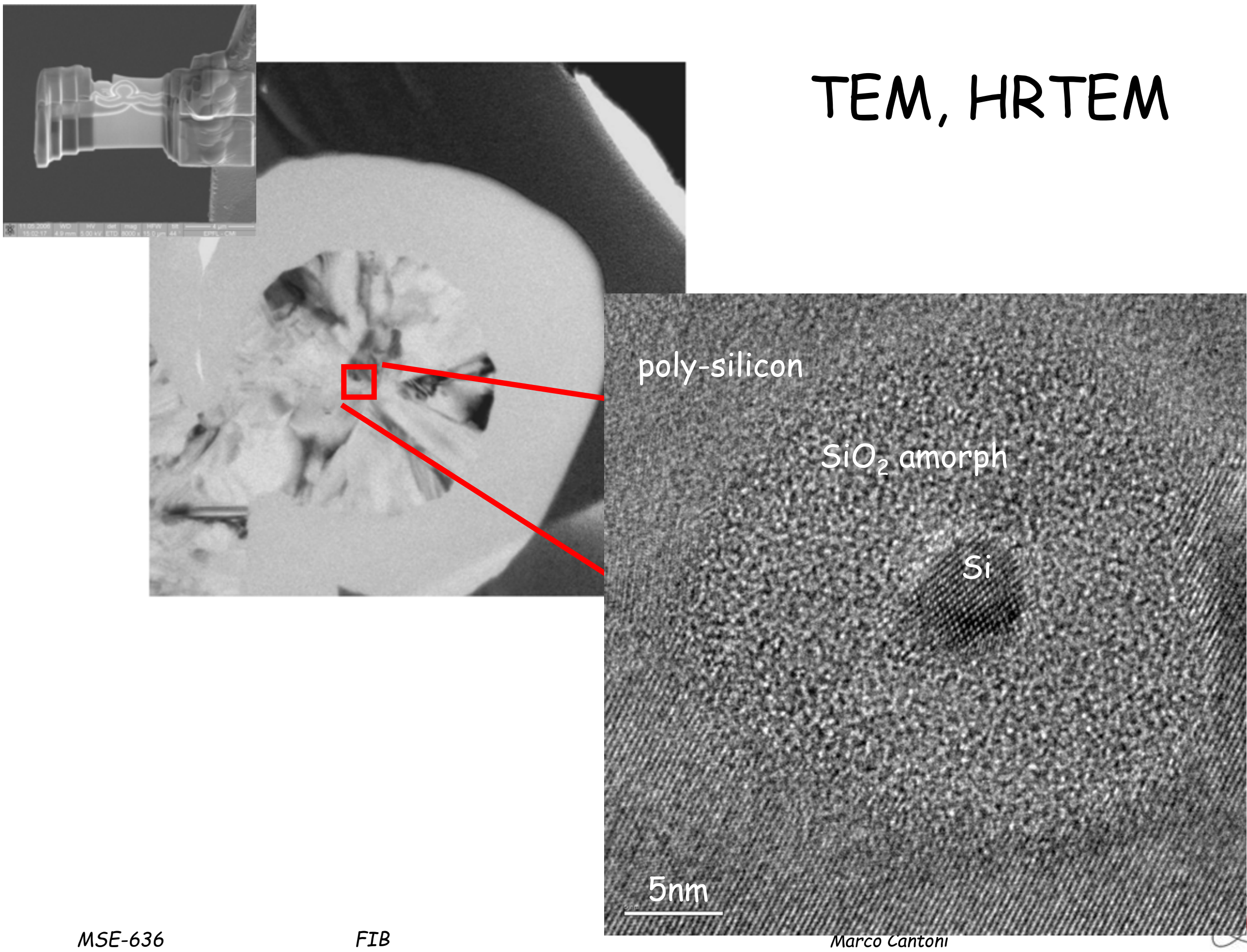












TEM, HRTEM

TEM lamellae by FIB

Focused Ion Beam adds a new dimension to TEM specimen preparation

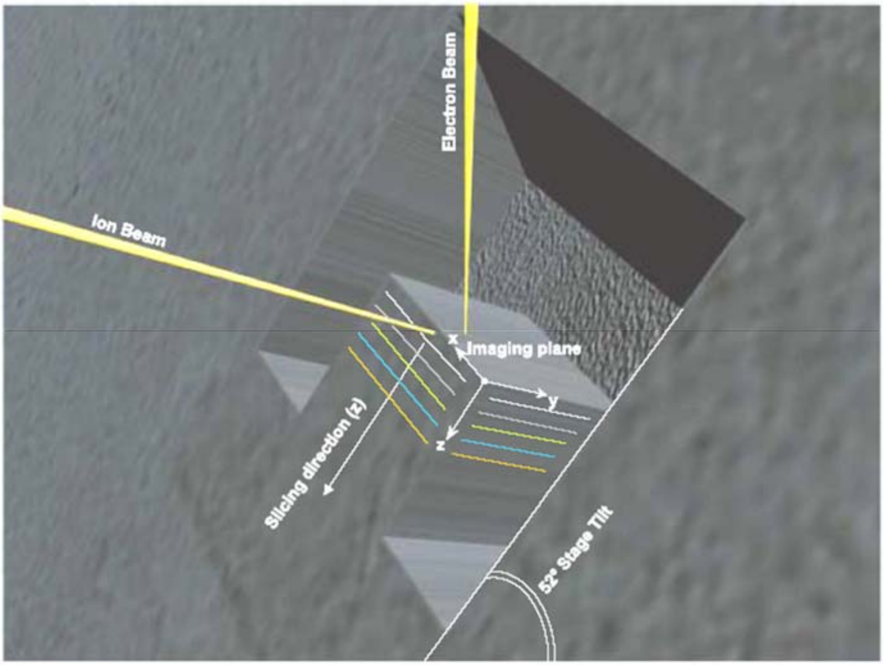
Take care of artifacts !!!

Large (10x5um) flat areas with uniform thickness (50-80 nm)

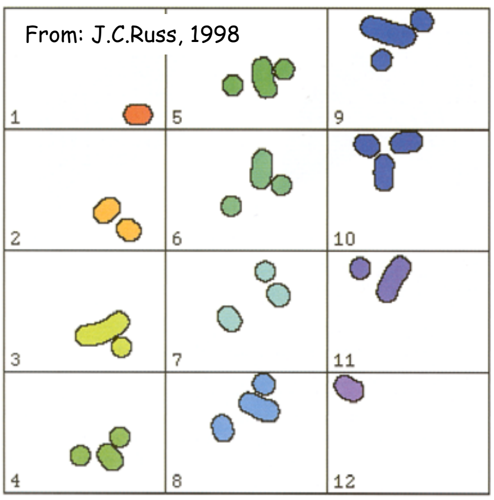
Preparation of heterogeneous samples with "difficult" material combinations becomes possible

Precise selection of the lamella position possible (devices)

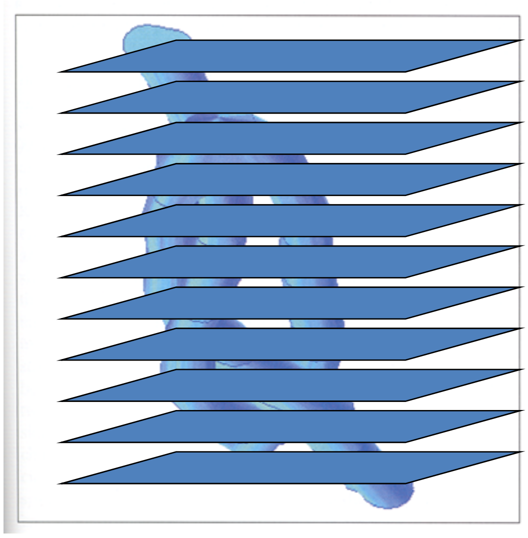
FIB Nanotomography
3D Microscopy



Problem of serial sectioning:
3D-reconstruction of disordered microstructures



3D

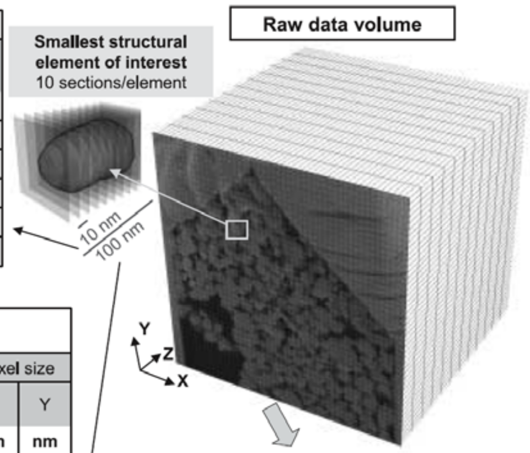


2D Volume fraction

?? Nr of particles ??
?? Shape ??

Voxel, Resolution, Pixel size

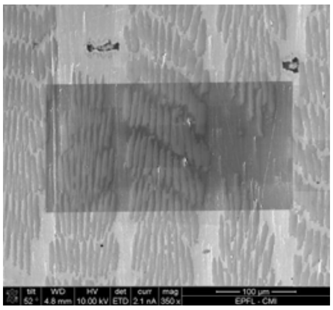
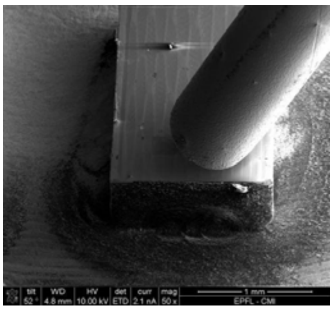
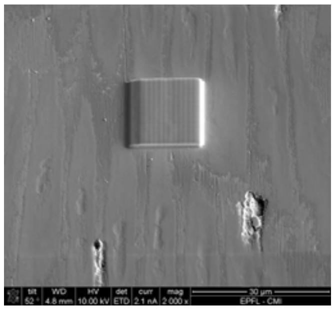
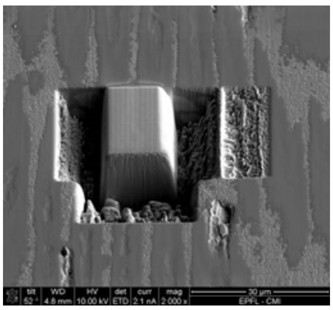
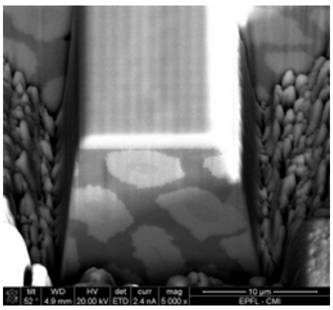
Ion-Beam			
Smallest element	Mag	Ideal z-spacing	Beam current
nm	**kX**	**nm**	**pA**
1000	5	100	500
500	10	50	300
300	15	30	100
200	30	20	50
100	50	10	50

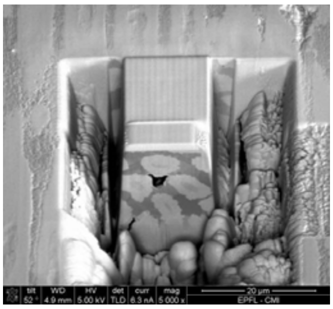


Electron-Beam					
Smallest element	Mag	Image width		Pixel size	
		1024 pxls, X	884 pxls, Y	X	Y
nm	**kX**	**µm**	**µm**	**nm**	**nm**
1000	5	60.0	65.73	58.6	67.9
500	10	30.0	32.86	29.3	33.9
300	15	20.0	21.91	19.5	22.6
200	30	10.0	10.95	9.8	11.3
100	50	6.0	5.18	5.9	6.8

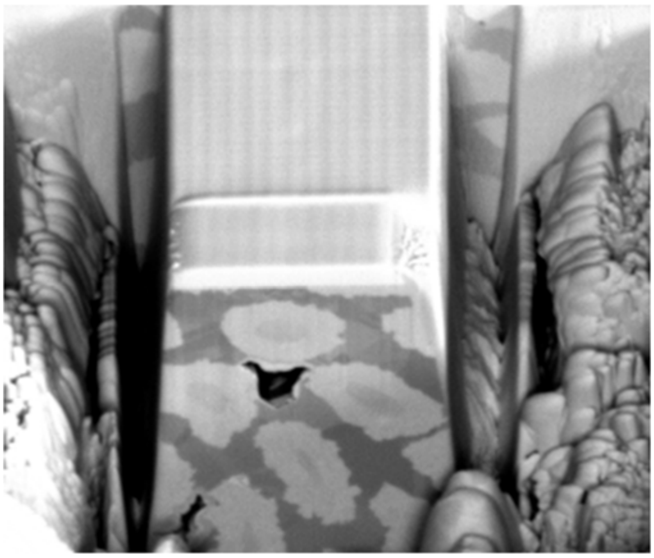
Present example:	x	y	z
Cube size [µm]	6.04	6.01	1.74
Voxel matrix	1024	884	105
	[pixels]		[sections]
Voxel size [nm]	5.9	6.8	16.6

3D slicing of multifilament Nb_3Sn superconductor

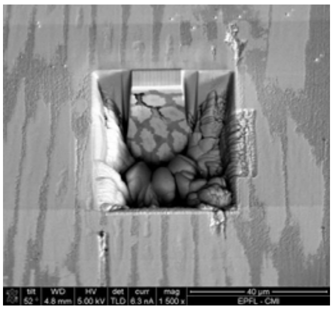


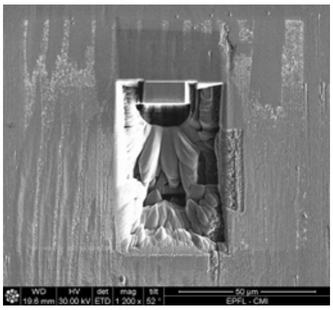
Preparing for slicing



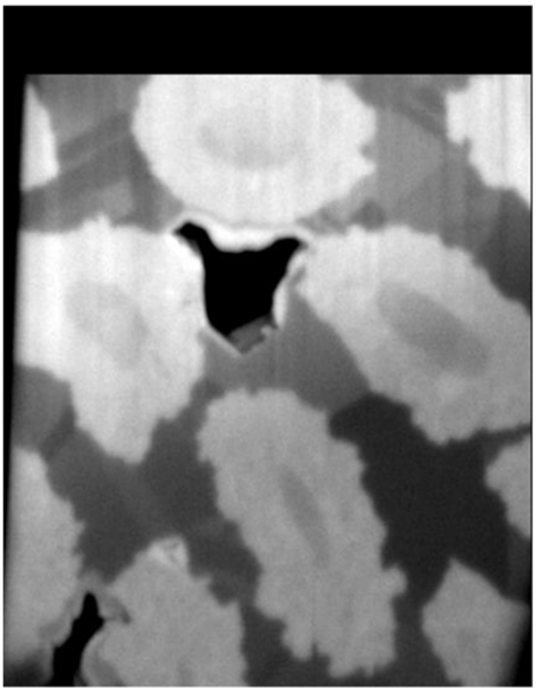
Automated milling and imaging of 170 slices (10h)

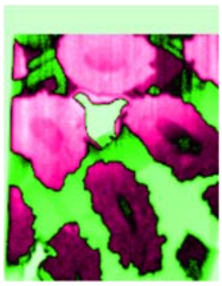
the end





align and crop



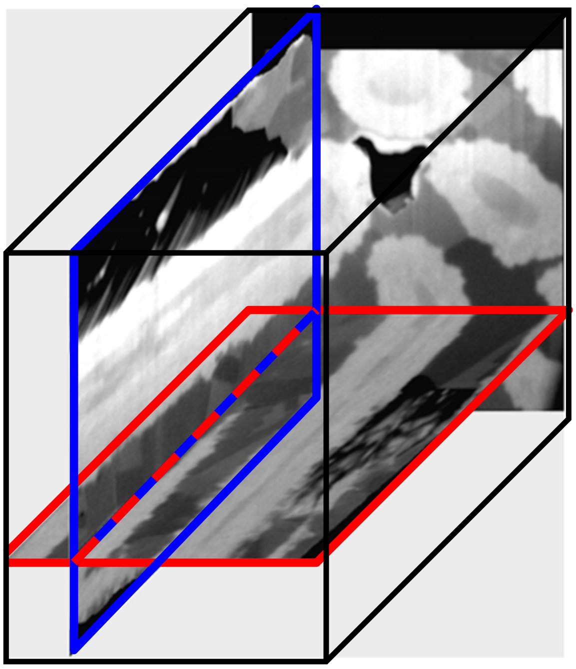


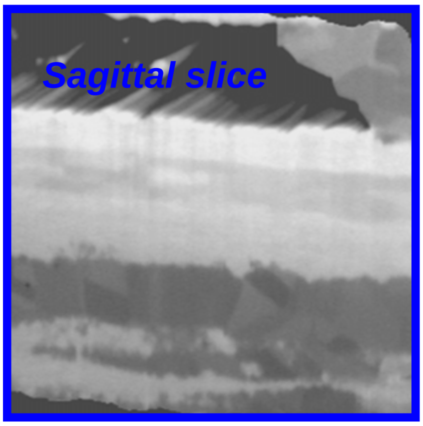
ImageJ

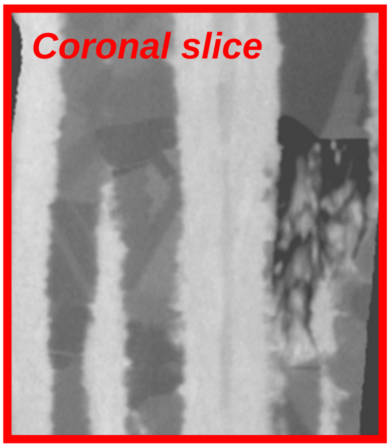
Image Processing and Analysis in Java

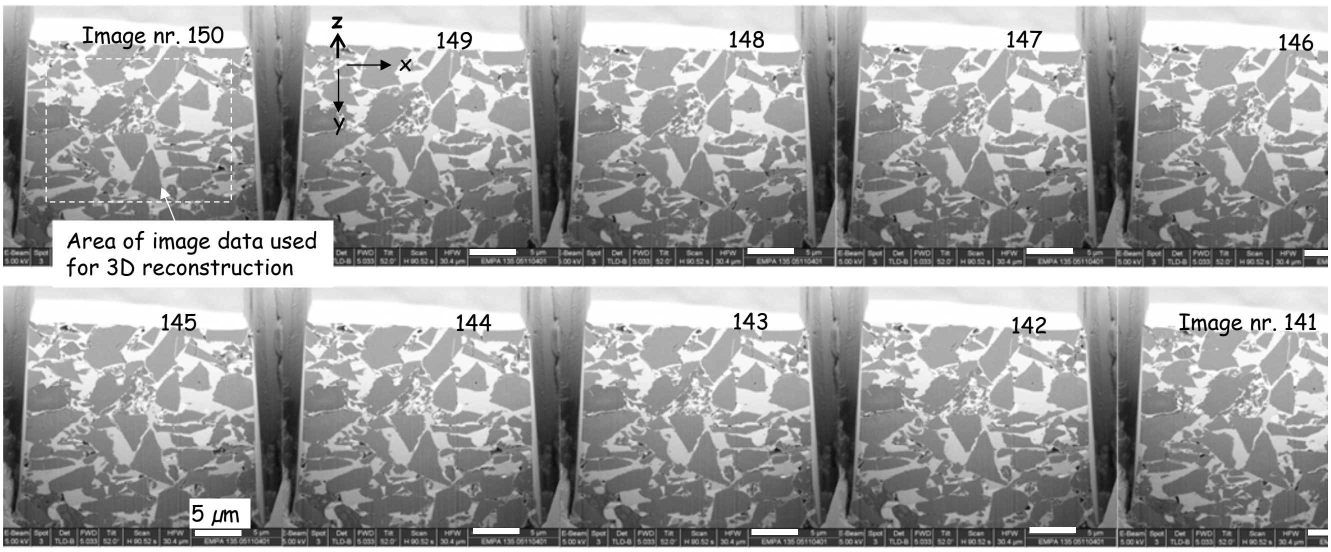
http://rsb.info.nih.gov/ij/index.html

3D volume rendering, reconstruction:
Orthogonal slices







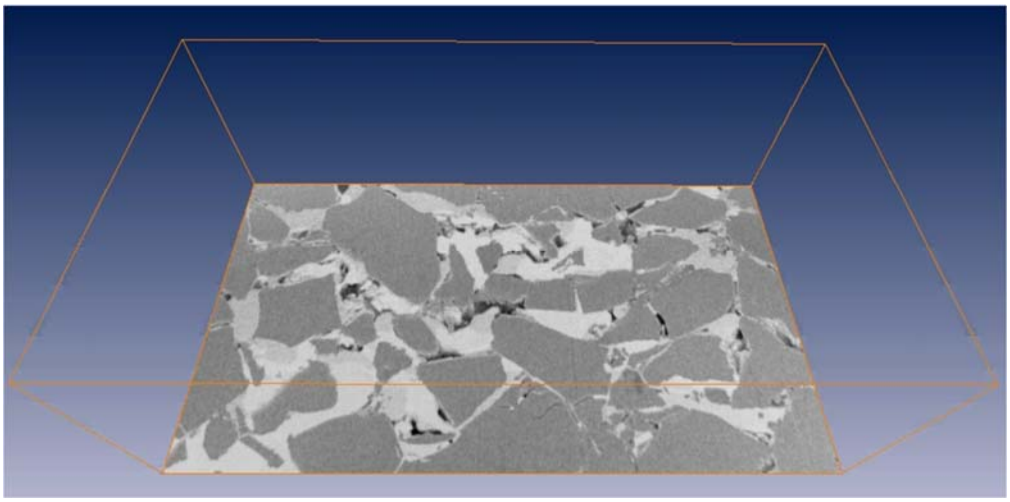


Quantitative microstructure analysis
→ Algorithms
→ object recognition
→ stereological correction of boundary truncation
→ extraction of statistical data (particle shape and size distribution)

Particle recognition:
Edge detection in 3D,
Watershed for separation

Voxel:
75nm

Cube:
40*
20*
15 µm



Size, 3D-shape, geometrical relationships between particles

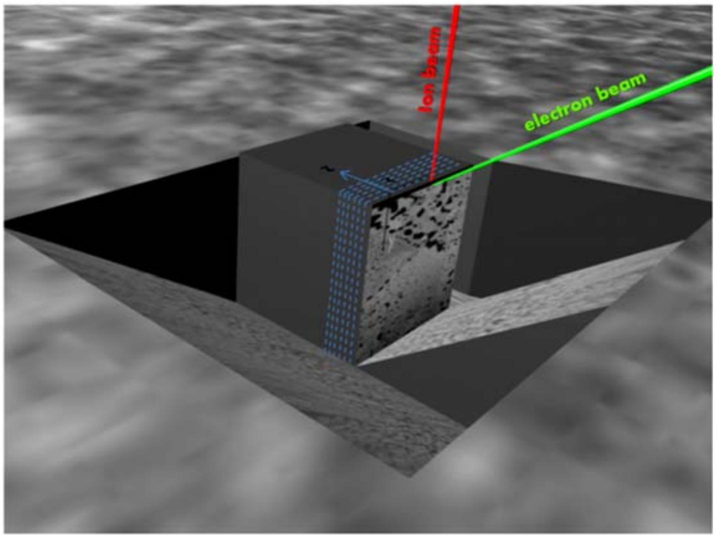
Grain size fraction 3

Cube size: 23.5x19.2x9.5 µm / Voxel size: 29.69x37.67x60 nm / Nr. of particles: 2236 (total), 1404 (inside)

Quantitative microstructure analysis → Algorithms

Münch and Holzer 2006
J.Amer.Ceram.Soc.
IB-nt of particulate systems – part I
Object recognition and
effect of boundary truncation

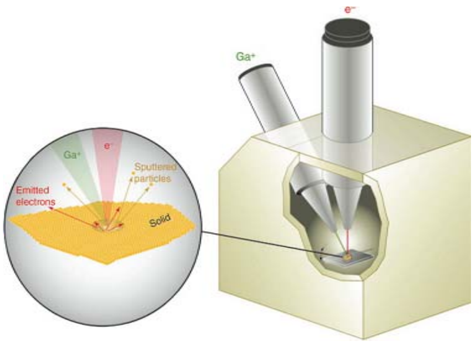
3D FIB/SEM: volume reconstruction



Three-dimensional analysis of porous $BaTiO_3$ ceramics using FIB nanotomography

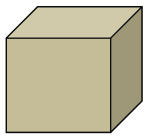
L. HOLZER, F. INDUTNYI, PH. GASSER, B. MÜNCH & M. WEGMANN
EMPA, Swiss Federal Laboratories for Materials Testing and Research, Ueberlandstrasse 129, 8600 Dübendorf, Switzerland

Journal of Microscopy, Vol. 216, Pt 1 October 2004, pp. 84–95



3D FIB/SEM: volume reconstruction

"Leitmotiv"
Isometric voxel size x = y = z



Slice thickness (z) = image pixel size (x,y)
Z dimension ~ X or Y, typical: 10nm, possible 5nm (3nm)

Image dimensions / data size (8-bit grey level tiff):
- 1024 x 786: 800 slices -> 640 Mb
- 2048 x 1572: 1600 slices -> 5 Gb
- 3096 x 2358: 3000 slices -> 21 Gb

Acquisition time ~1min / slice
(40-60 slices / hour)
-> high S/N ratio, beam current (1-1.5nA), detector efficiency

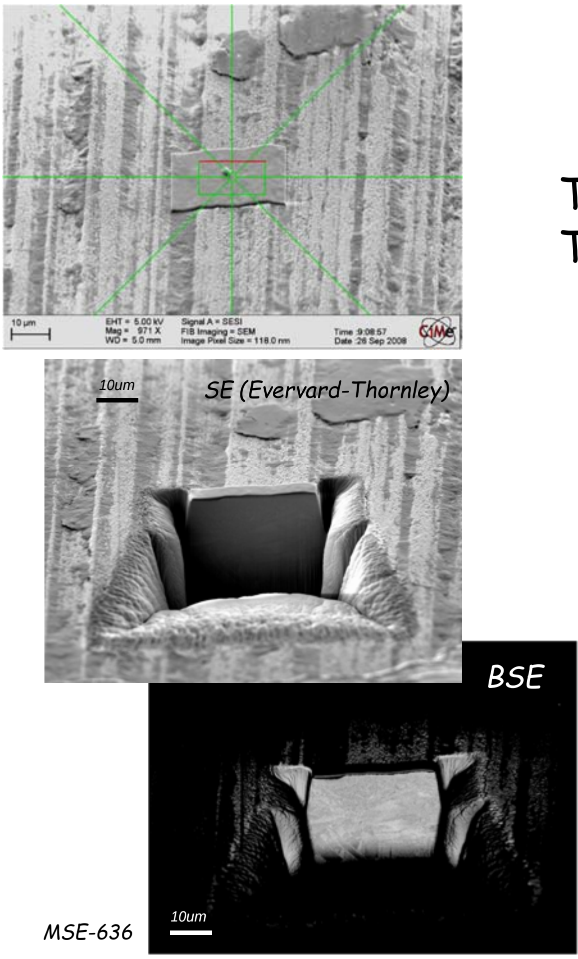
Dwell times/pixel 5- 15µsec. (detector signal -> 256 grey levels)

High throughput: minimise overhead, no tilting, rotating, drift correction

Z- Resolution: low kV !!!

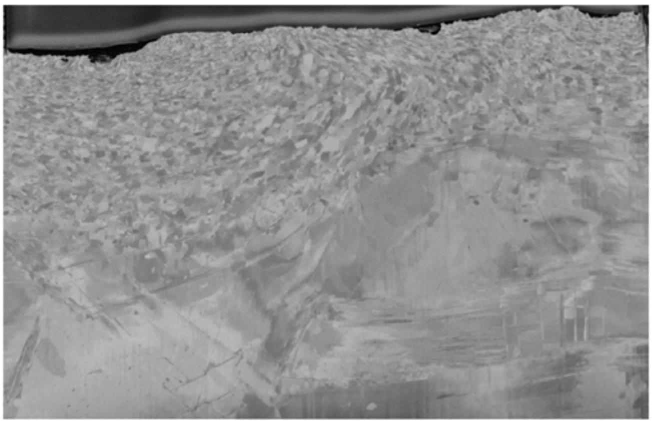
Orientation contrast

identification of grain texture

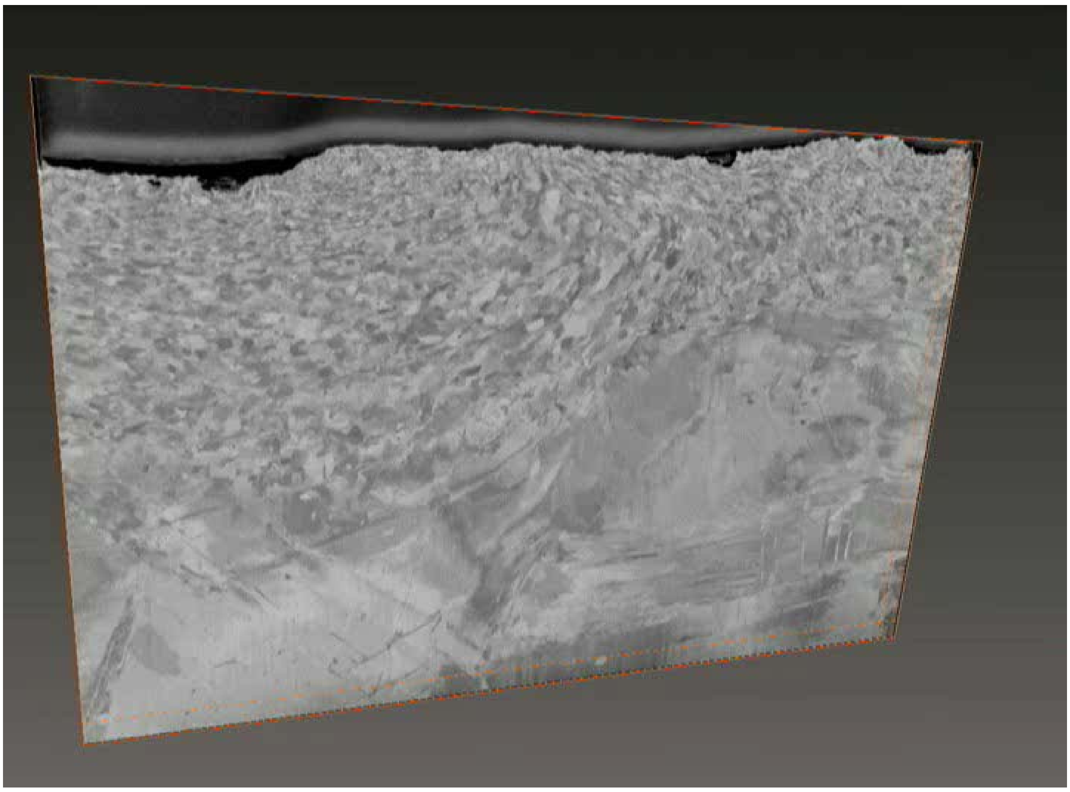


Tribology: wear trace on steel
Tribo-corrosion

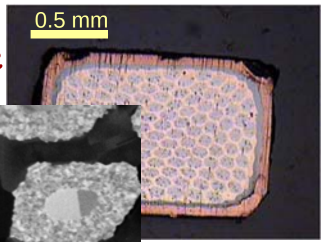
J. Perret, S.Mischler IMX-LMCH
Grain orientation contrast of small grains
(grain size < 100nm)

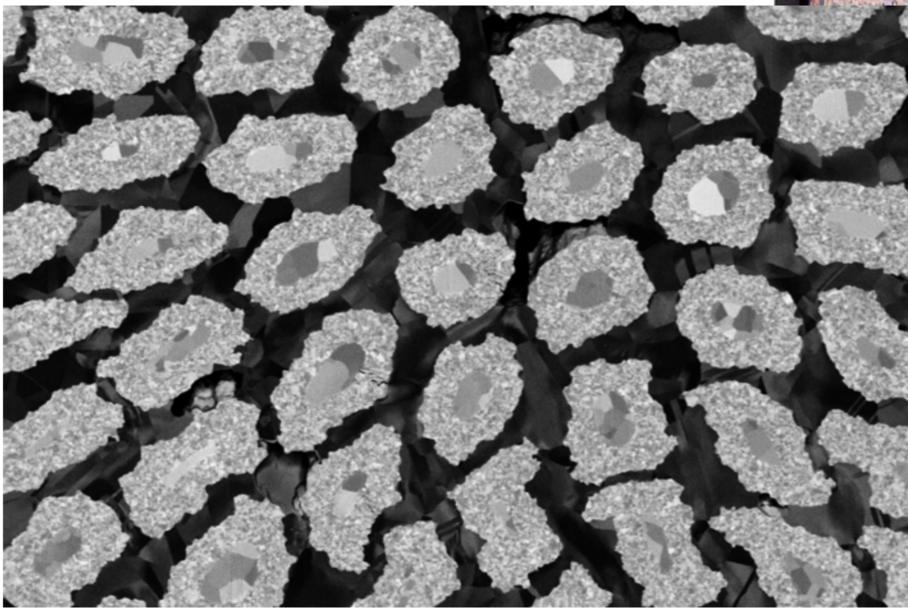


2048x1536x1200 volume: 20x15x12um
10x10x10nm voxel



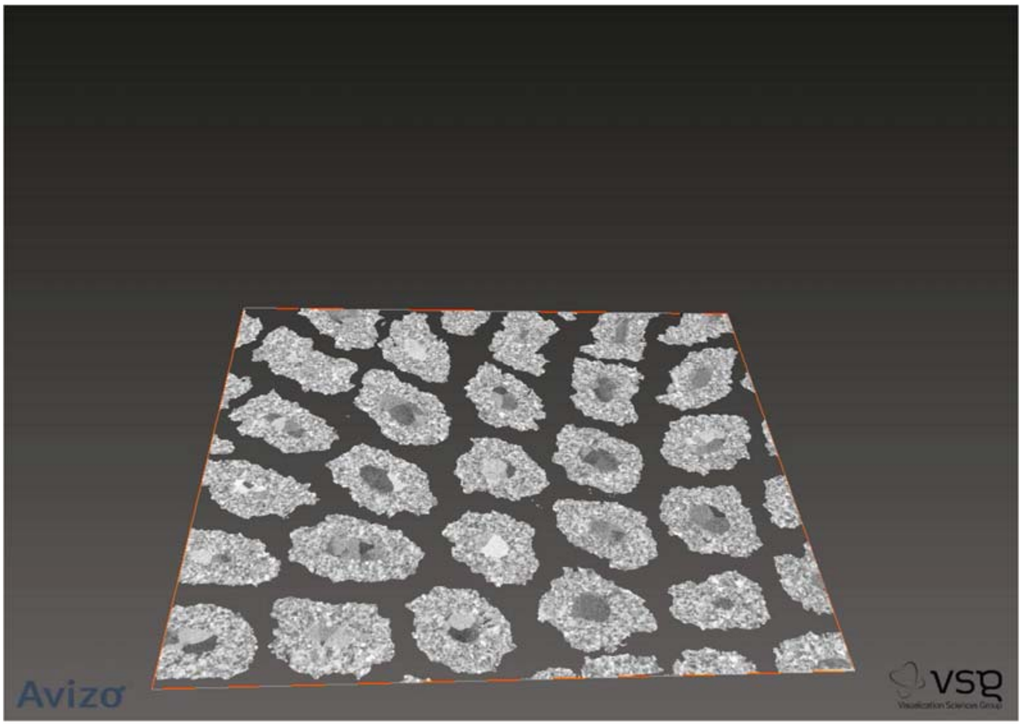
Nb_3Sn multifilament superconducting cable





Nb_3Sn superconductor multifilament cable:
14’000 Nb_3Sn filaments (diameter ~5um) in bronze matrix

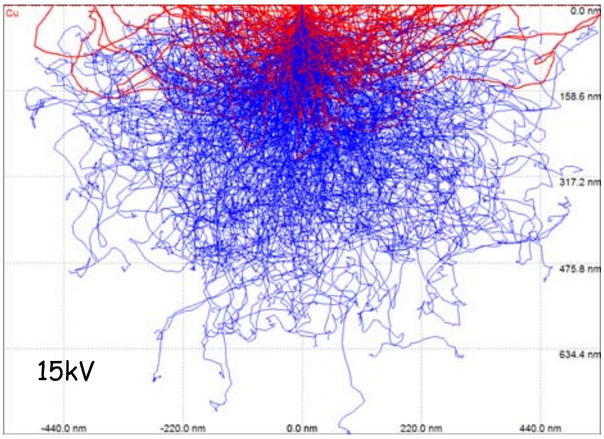
1.8kV EsB detector: Materials & orientation contrast



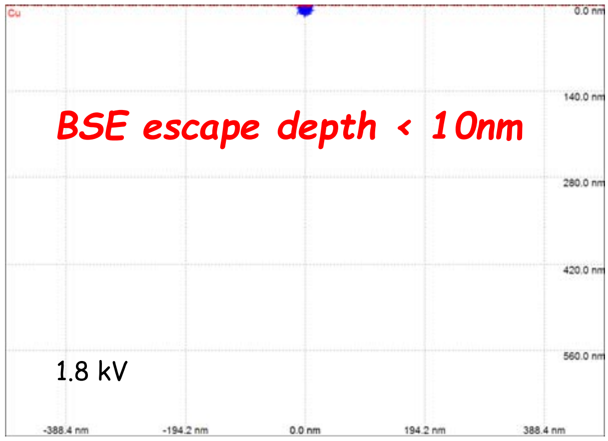
Materials & grain contrast
2048x1536x1700, (10x10x10nm voxel), 28hours

What is the spatial resolution ...?

"Classic"



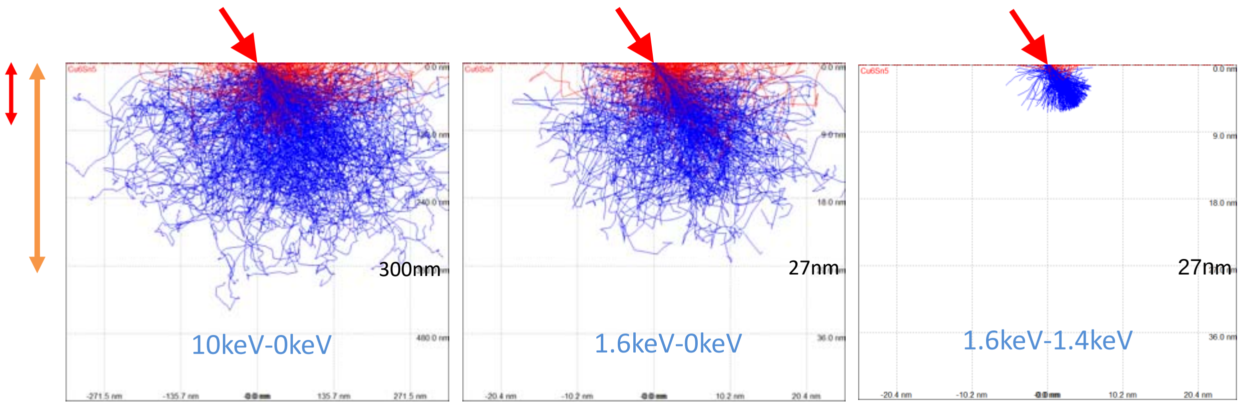
Low kV



Scatter range in Nb_3Sn:
Monte-Carlo Simulation (CASINO 2.42) of electron trajectories
backscattered electrons

What is the spatial resolution of BSE electrons ?

Scatter range in Nb_3Sn:



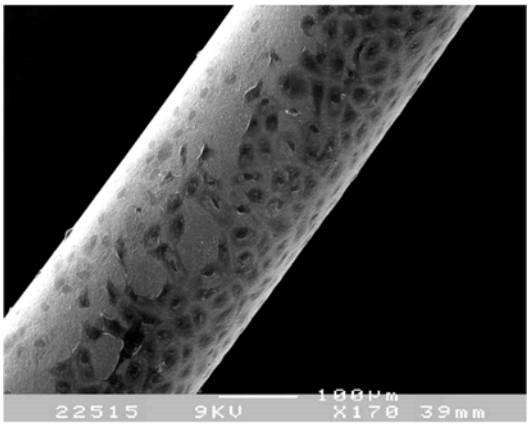
HT	10keV	1.6keV	1.6keV (low loss, EsB grid at 1.4kV)
BSE esc. depth	100nm	10nm	2-3nm
penetration	300nm	20nm	(20nm)

Energy selective BS

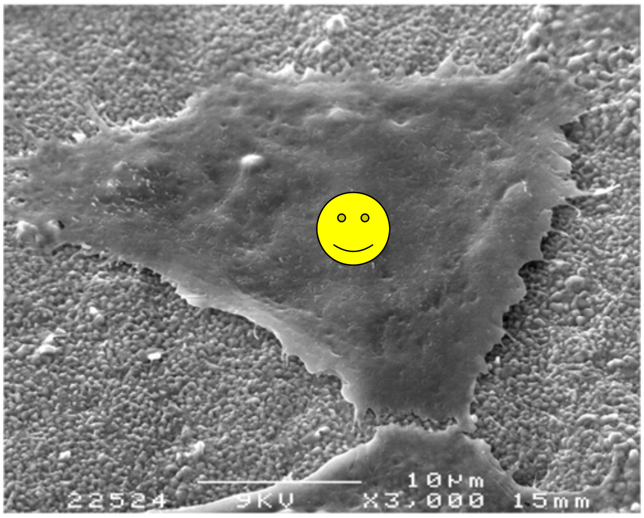
FIB Nanotomography of biocompatible materials

K. Dittmar, A. Tourvielle, H. Hofmann EPFL-IMX-LTP
M.Cantoni EPFL-CIME

Biocompatibility of implants (ceramic coatings)
Drug delivery from implants

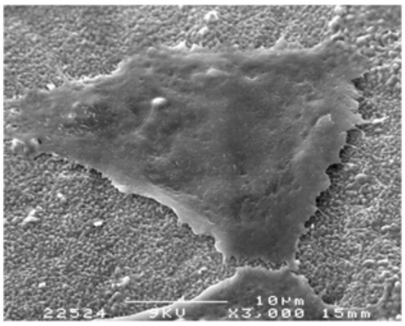


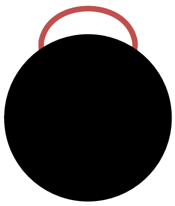
SEM: critical point drying, metal coating

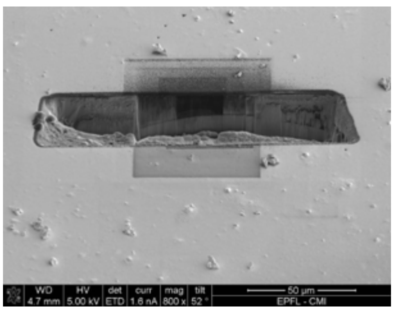


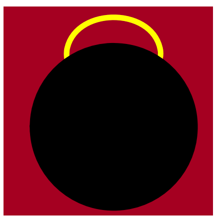
How do cells attach to a surface..?

FIB Cross-section of a fixed, epoxy-embedded and stained sample



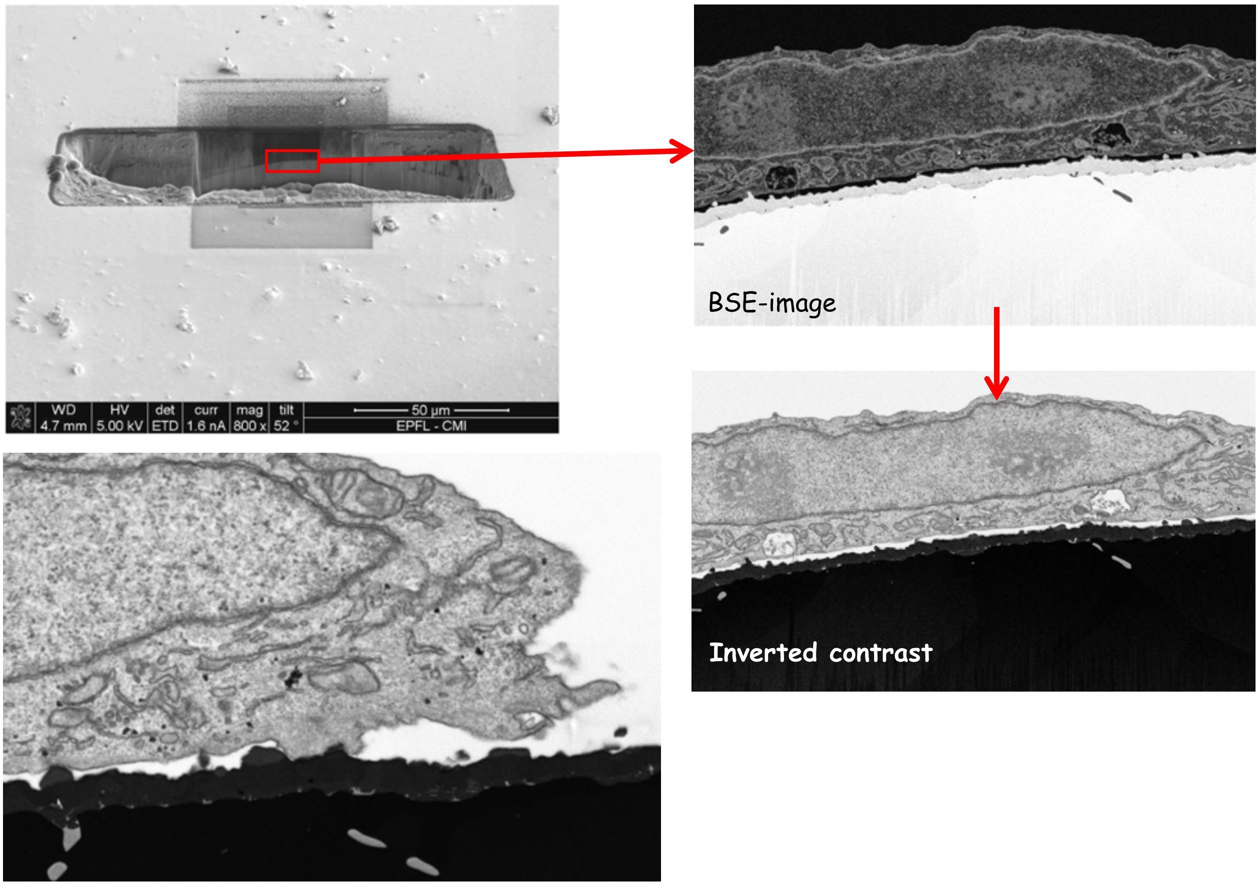


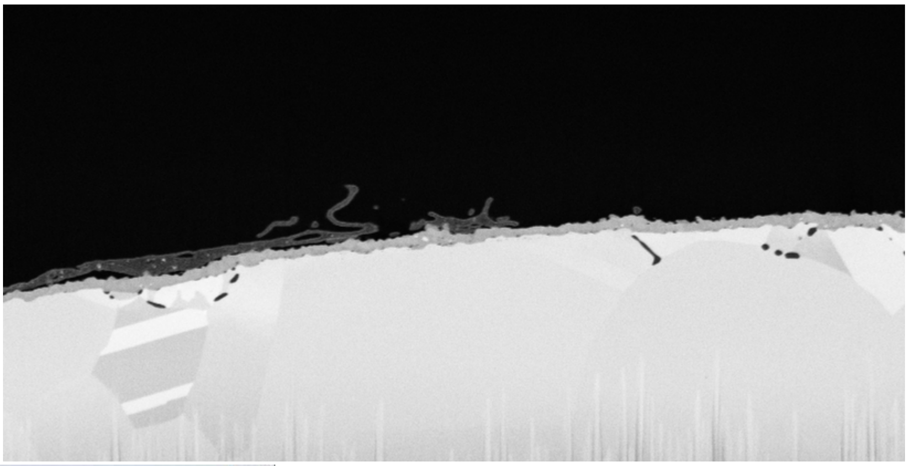




FIB milling of
"hollow" structure
versus
FIB milling of
massive "homogenous block"

Does this cell like the coating…?





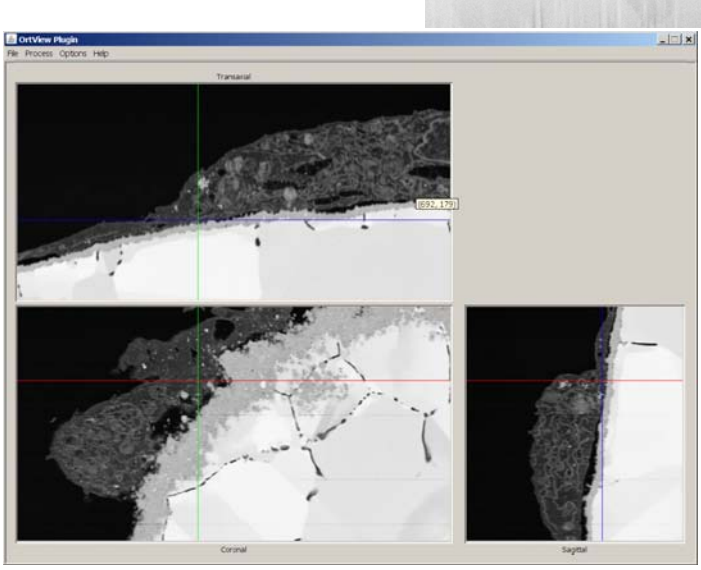


Image stack

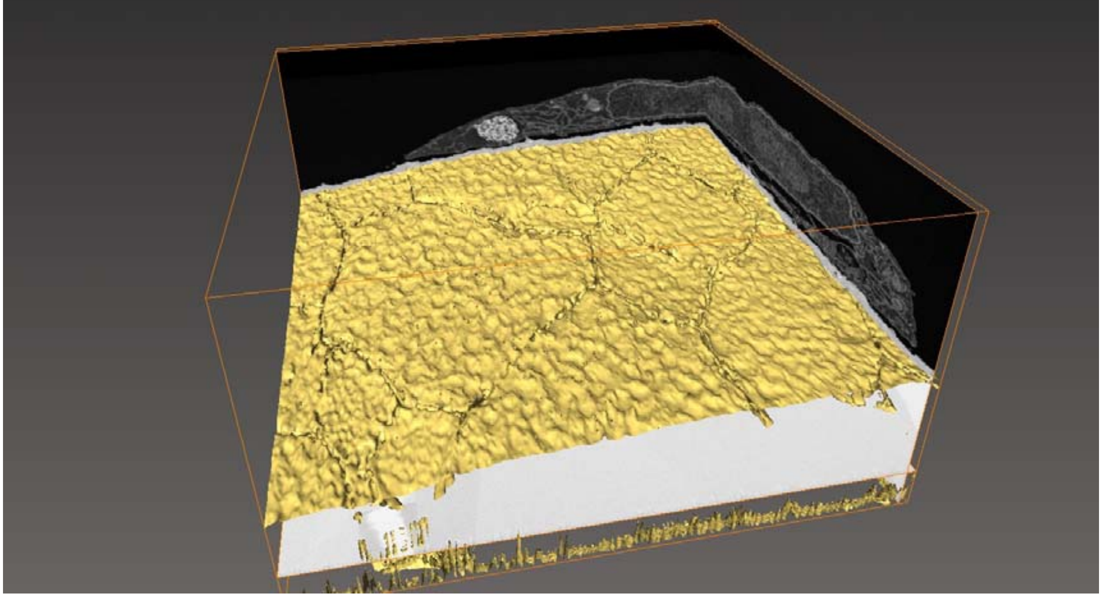
1024x786 pixel:
(10nm image pixel size)

2kV, 60um Aperture, high current,
EsB detector (grid 1.5kV)

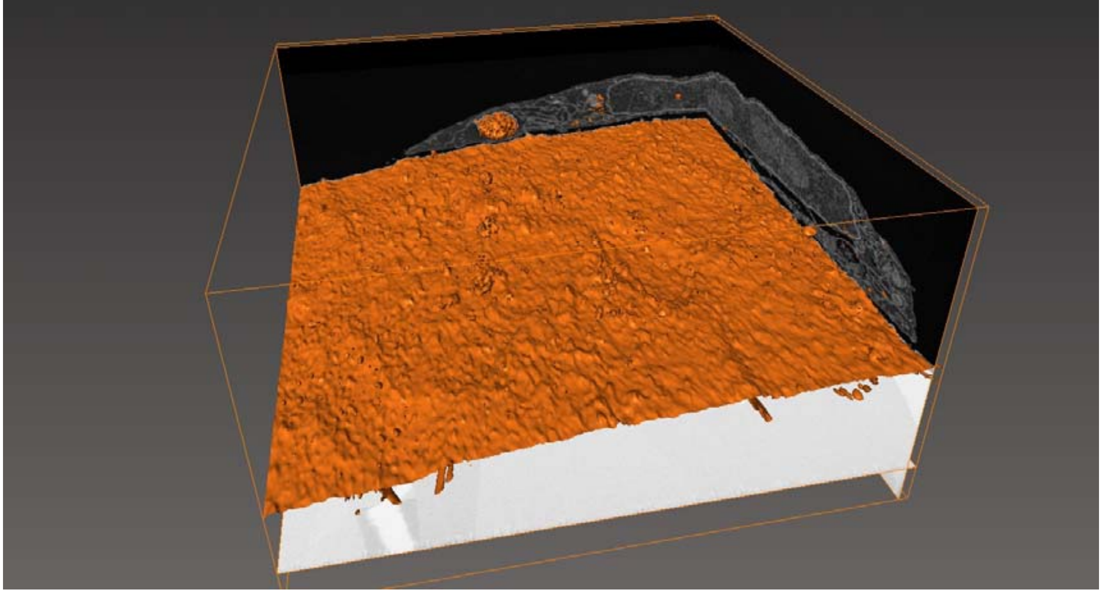
600 slices, 20nm thickness

Milling current 700pA

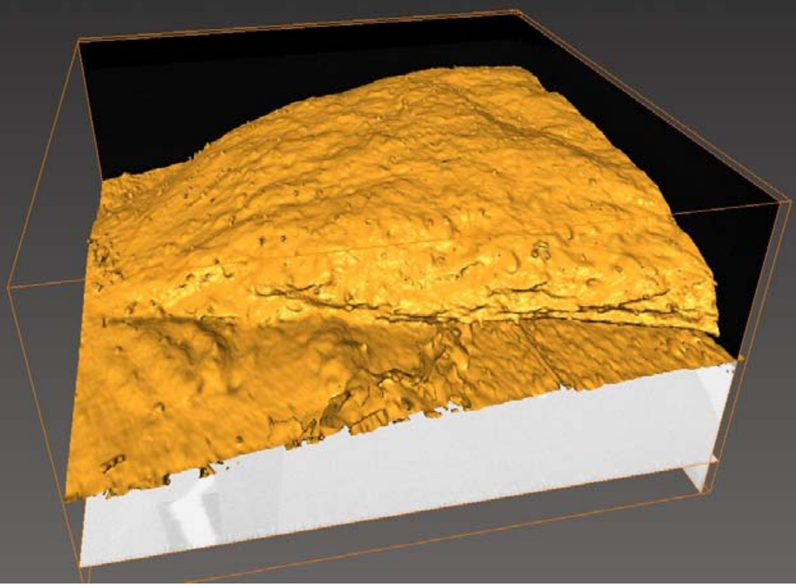
Rendering of isosurfaces: steel

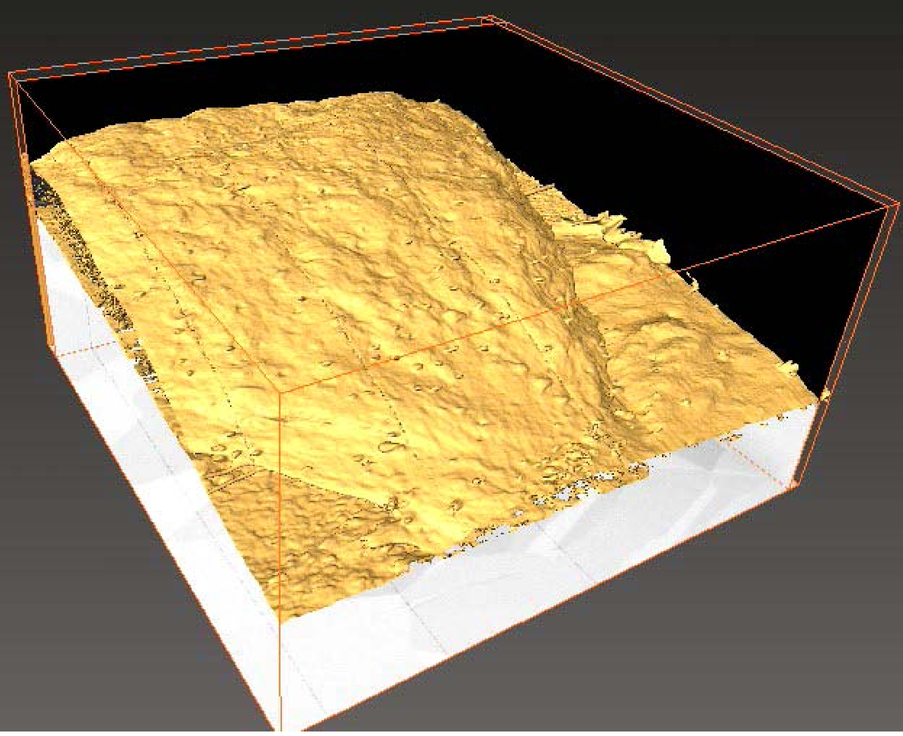


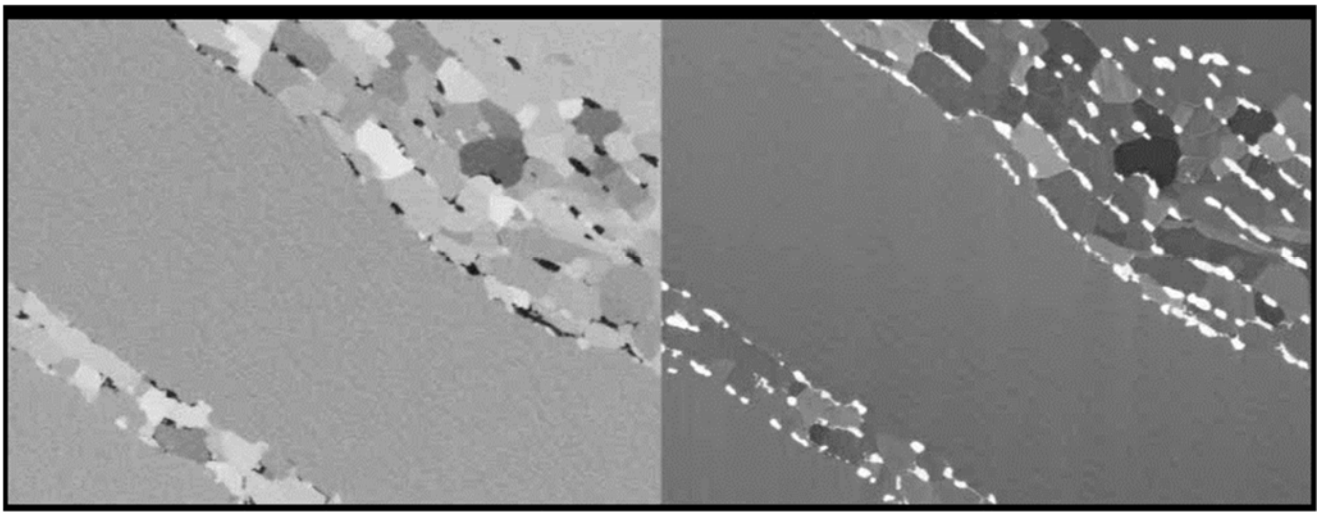
Rendering of isosurfaces: TiO_2 coating



Rendering of isosurfaces: cell surface

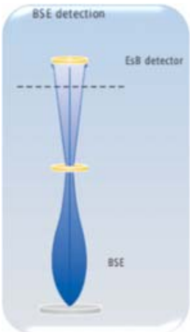




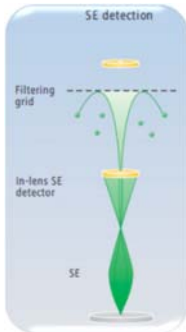


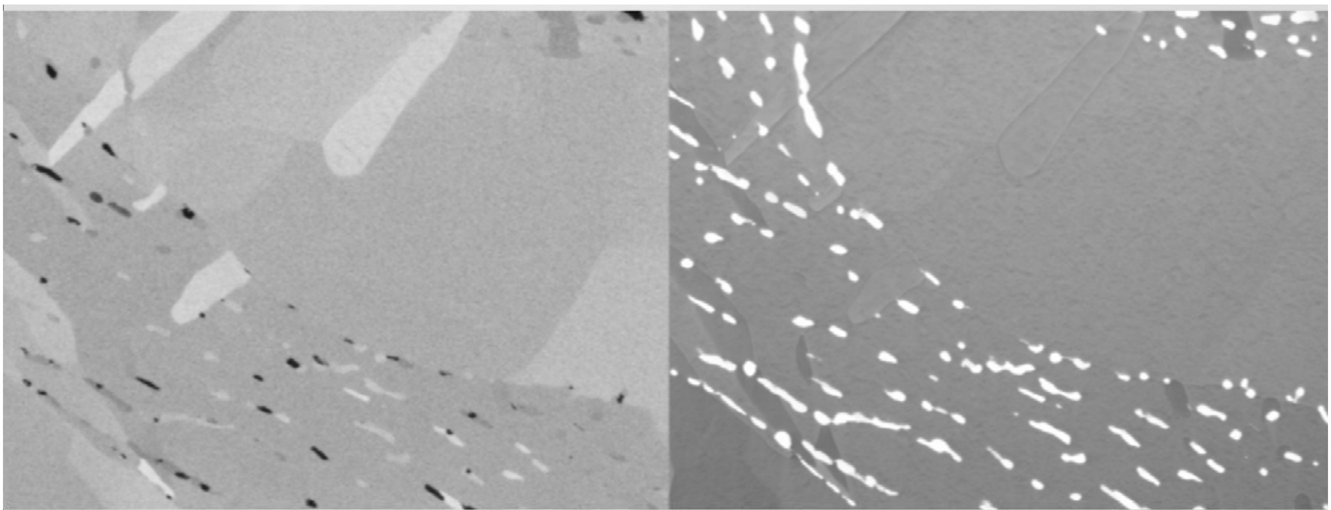
EsB

InLens SE



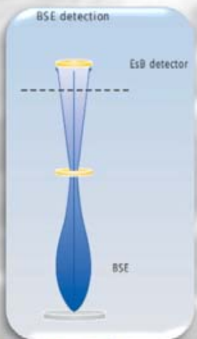
10x10x10nm voxel size, 2048x1536x2000
2 images (2x3Mb) / slice ...! (DUAL Channel !)
12Gb data



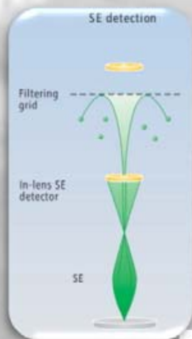


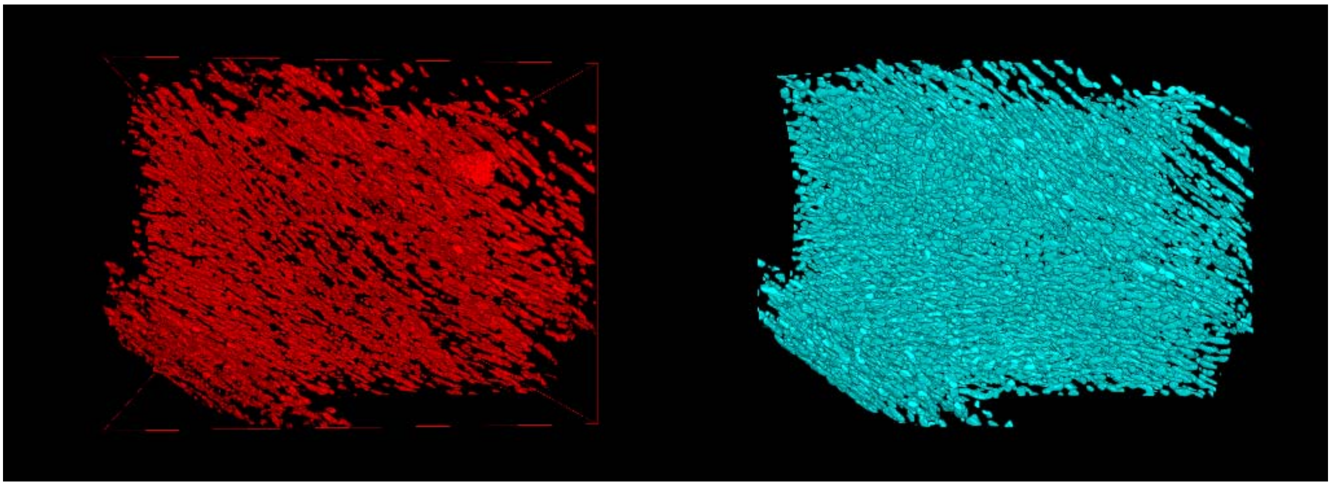
EsB

InLens SE



10x10x10nm voxel size, 2048x1536x2000
2 images (2x3Mb) / slice ...! (DUAL Channel !)
12Gb data

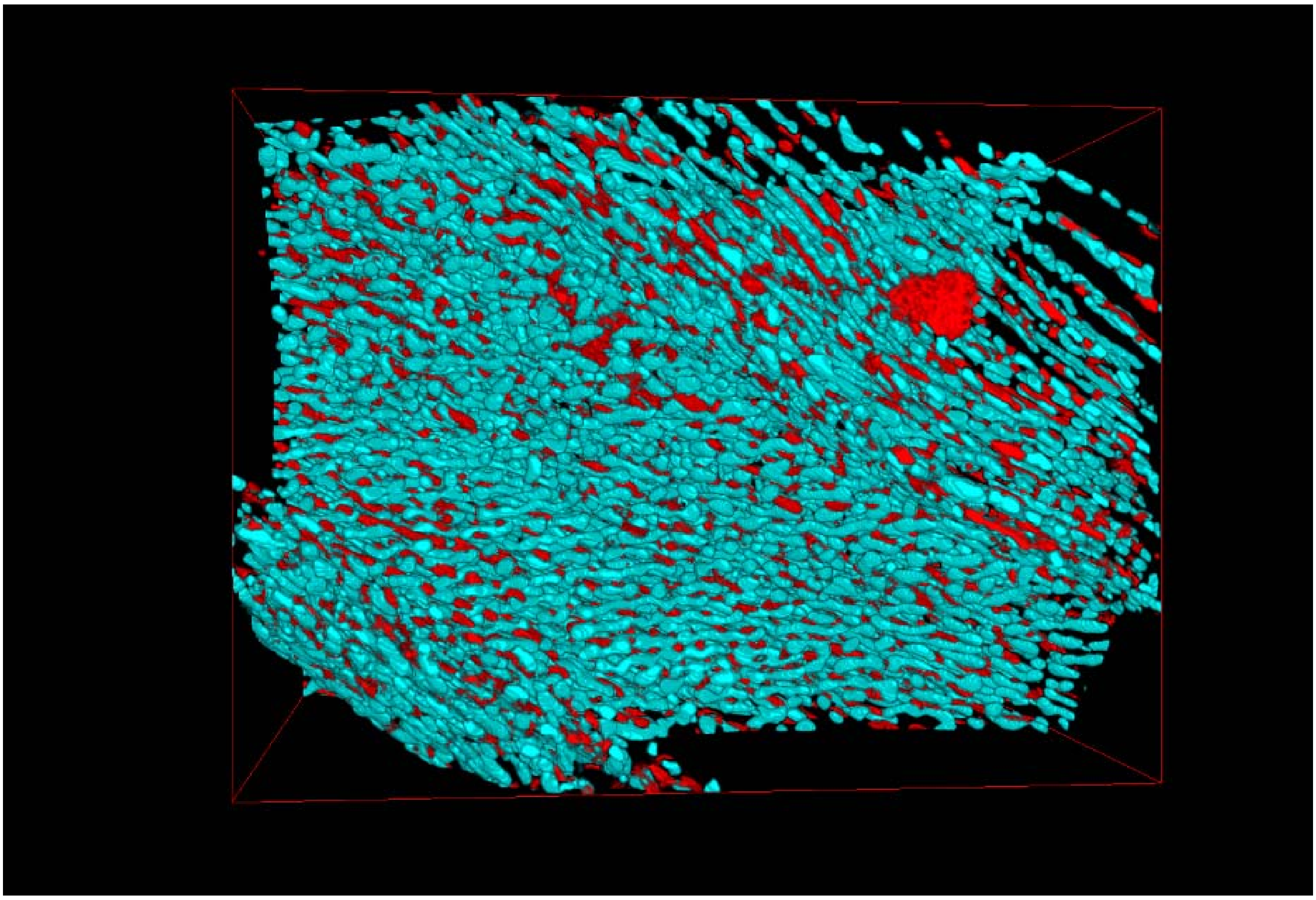


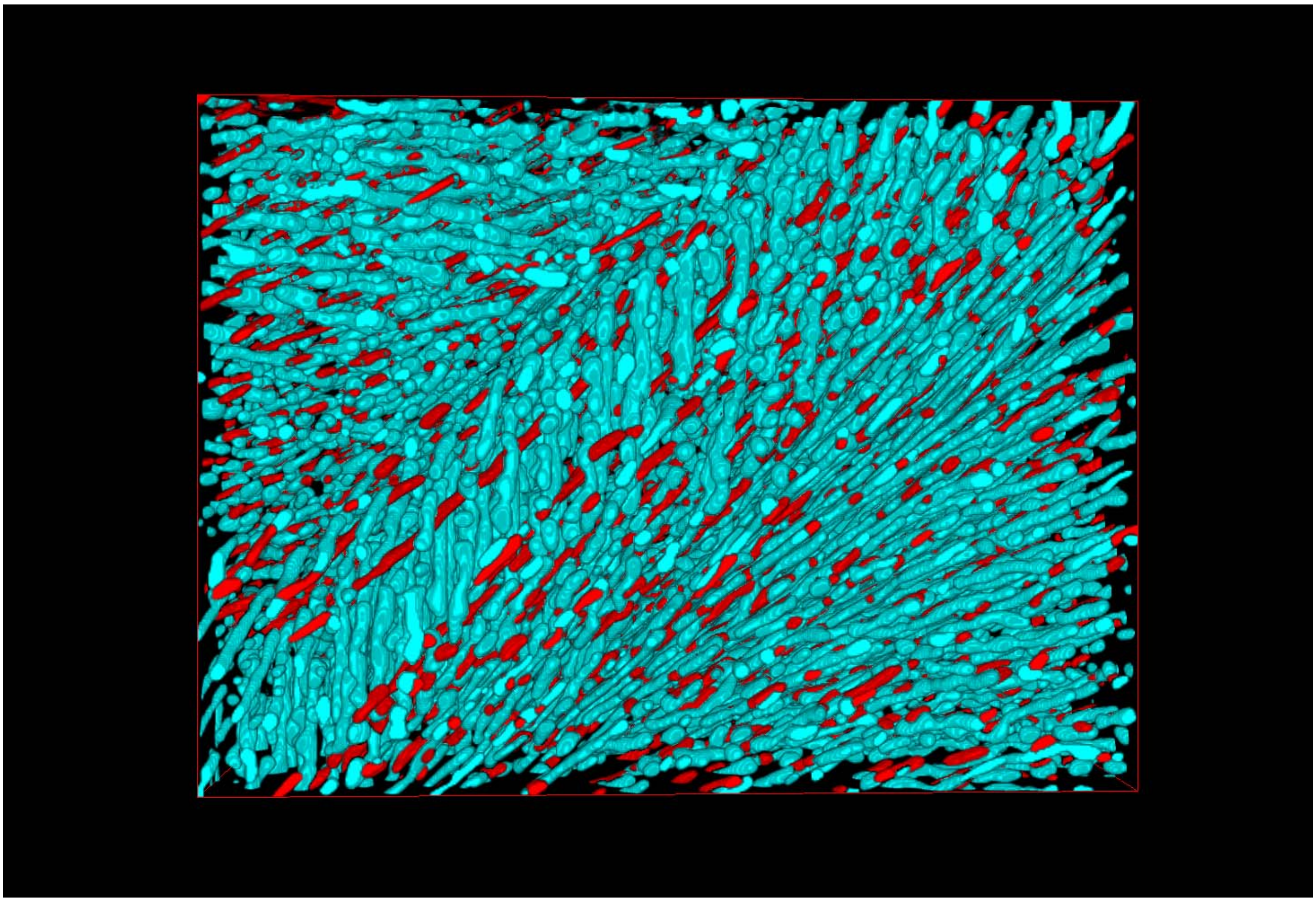


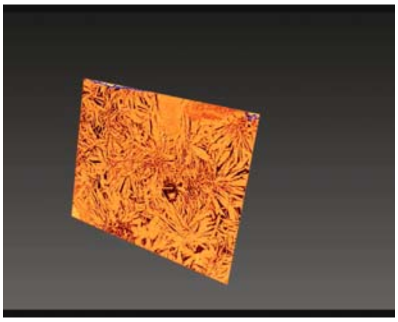
Phase 1: **Cu_6Sn_5**
Dark in EsB image
White in SE-InLens

Phase 2: **Ag_3Sn**
White in SE-InLens - Dark in EsB image

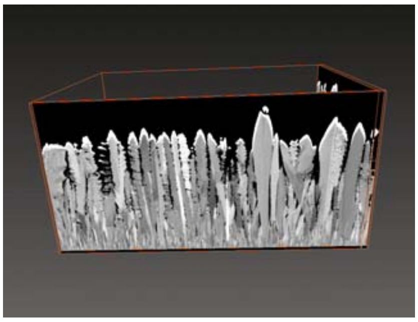
10x10x10nm voxel size, 2048x1536x2000 pixel/slices
2 images (3Mb) / slice …… 12Gb data
32 hours



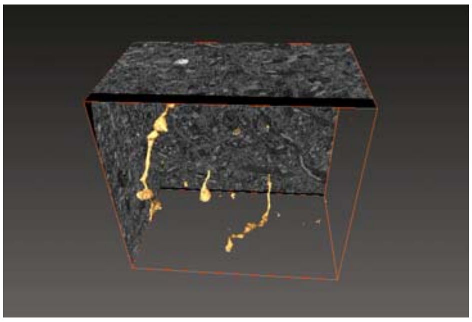




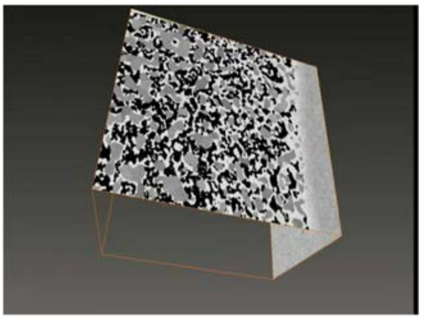
Cement, (10nm)^3 voxel



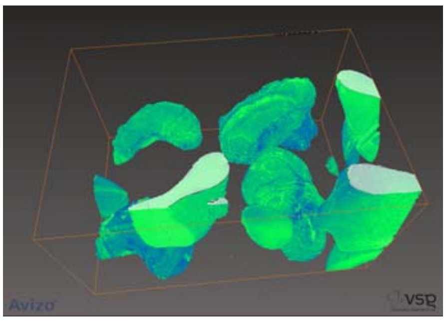
Solar cell: ZnO, (10nm)^3 voxel



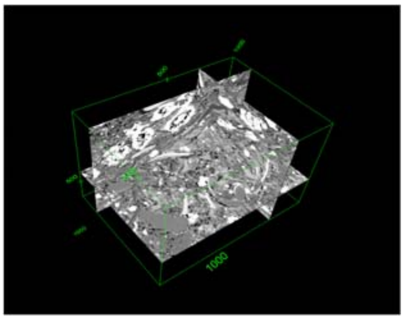
Rat brain, (5nm)^3 voxel



SOFC, (10nm)^3 voxel



Malaria parasite, (8nm)^3 voxel

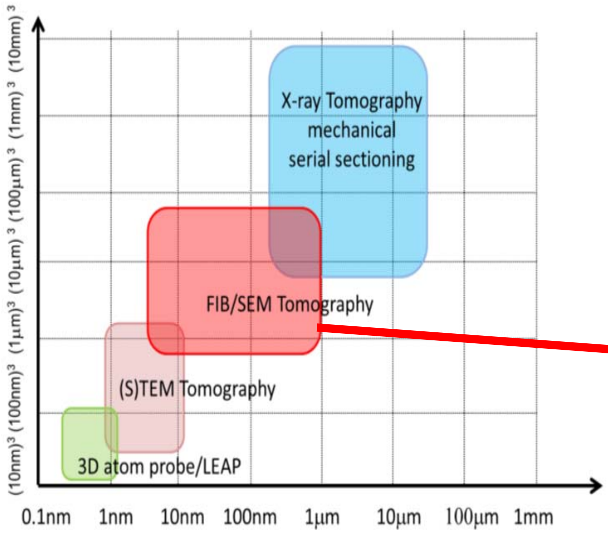


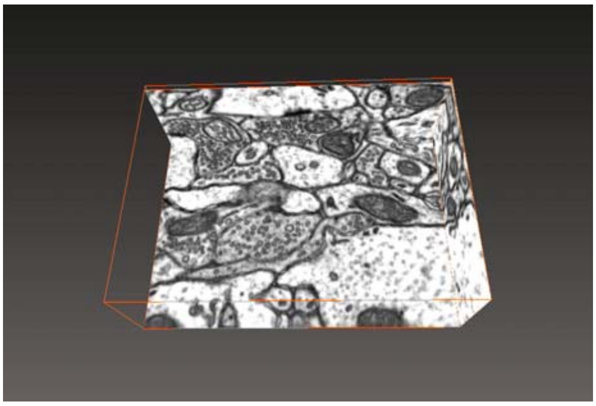
clay, (10nm)^3 voxel

FIB/SEM Nanotomography, volume reconstruction
Typical voxel sizes

FIB-NT compared with other 3D-techniques

- Voxel size ~5-10nm
- Dwell time ~10µsec.
- 1 slice, image / min.
- HT: 1-2kV
- Escape depth of signal (BSE) ≤ 5nm





3x3x3 nm voxel brain tissue

New possibilities in 3D-microscopy: Combination with quantitative analytical SEM techniques: EBSD, **EDX**

Focused Ion Beam
adds a new dimension to electron microscopy

SEM goes 3D
- from 2D characterization and topography imaging to 3D volume analysis

TEM preparation
- « impossible » samples can now be prepared (heterogeneous samples)
- location of the transparent area can be selected with nm precision
- Parallel surfaces (uniform thickness) ideally for AEM