6. FIB

Focused Ion Beam

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Focused Ion Beam

a) Principles

How does it work..?

Ion sources, optics, interaction with the sample

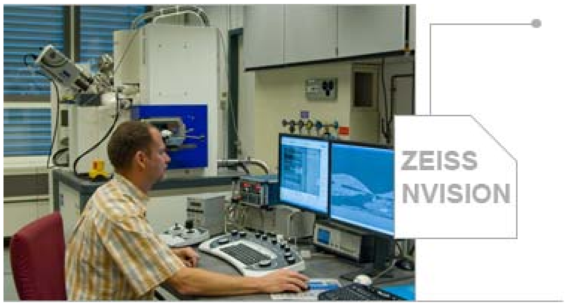
b) Basic Application

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)

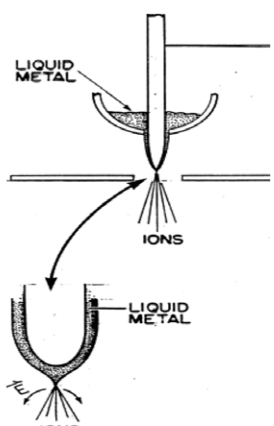
2 Kleindiek Micromanipulator (in situ TEM lamella lift out)

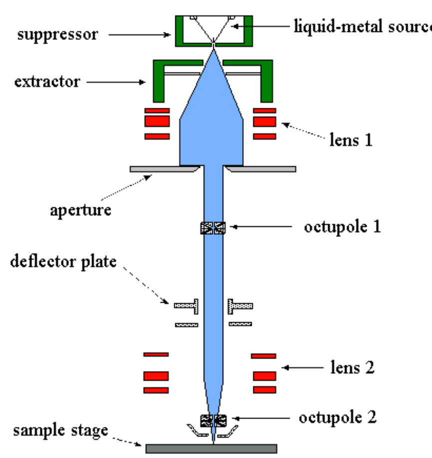
Focused Ion Beam

Mainly developed in 1970's and 80's
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





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

SIM = Scanning Ion Microscope

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

Advantage of Gallium:

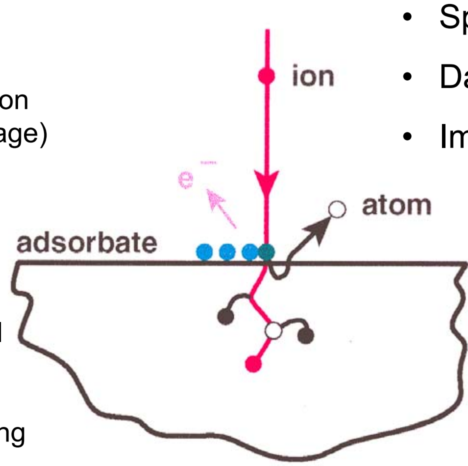
Ga is metallic, low melting point, in the middle of the periodic table, no overlap with other elements in EDX

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”

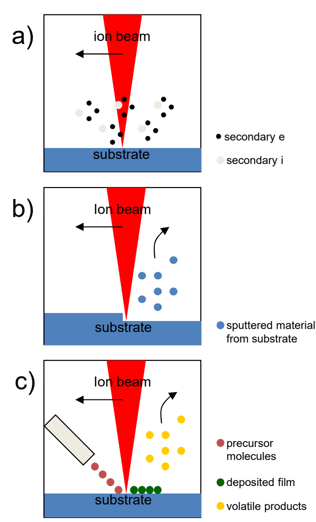
Emission of secondary ions and electrons
- FIB **imaging** ☛ a)
  low ion current

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

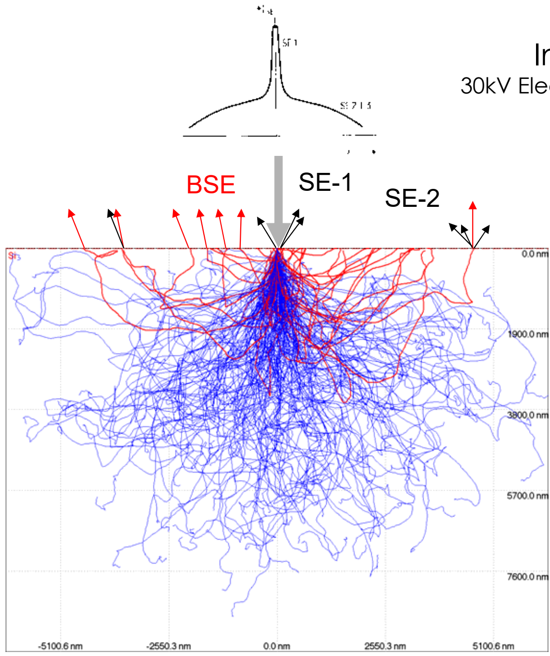
Chemical interactions (gas assisted)
- FIB **deposition**
- Enhanced (preferrential) etching ☛ c)

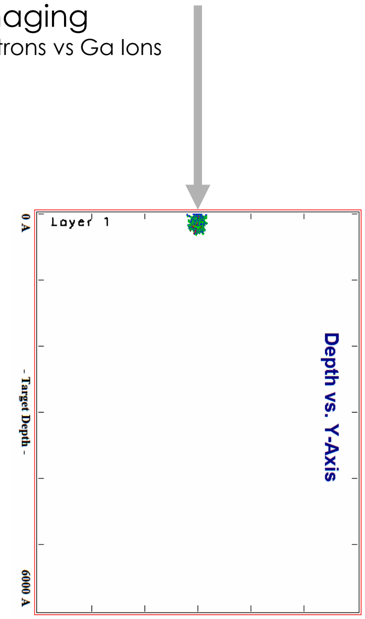
Other effects:
Ion implantation
Displacement of atoms in the solid
- Induced damage

Emission of phonons
- Heating



Imaging
30kV Electrons vs Ga Ions

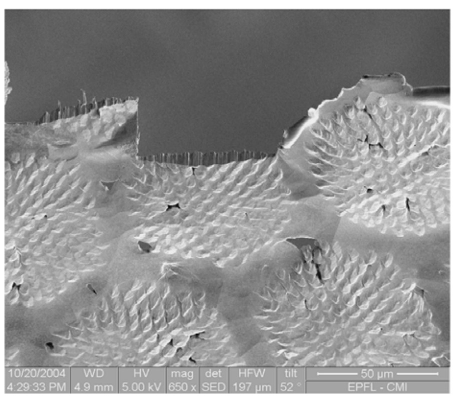




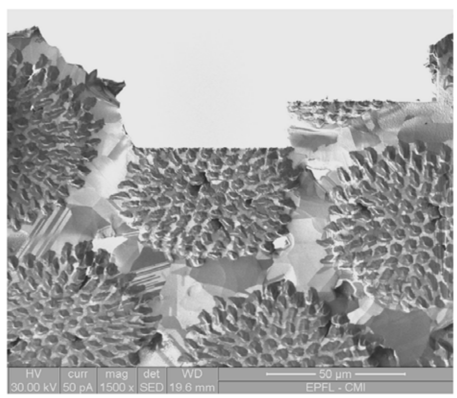
Monte-Carlo Simulation casino v2.42
http://www.gel.usherbrooke.ca/casino/download2.html

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

SE image contrast

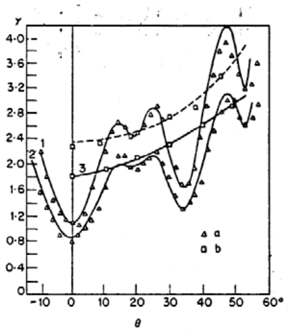


e-beam 5kV



ion-beam 30kV 50pA

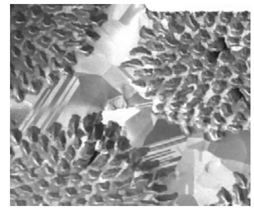
material (sputtering) contrast
orientational contrast

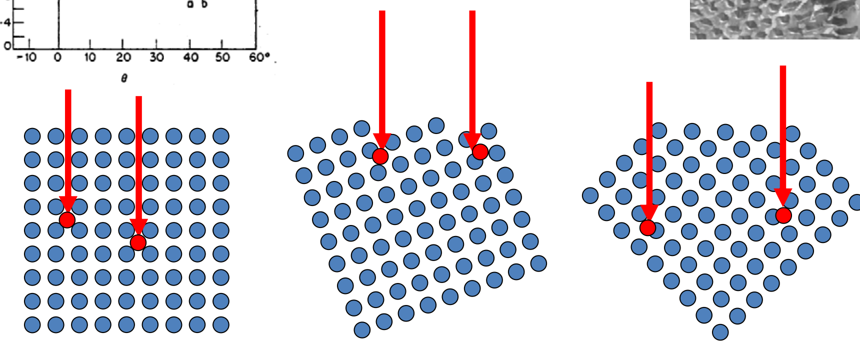


Channeling contrast

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

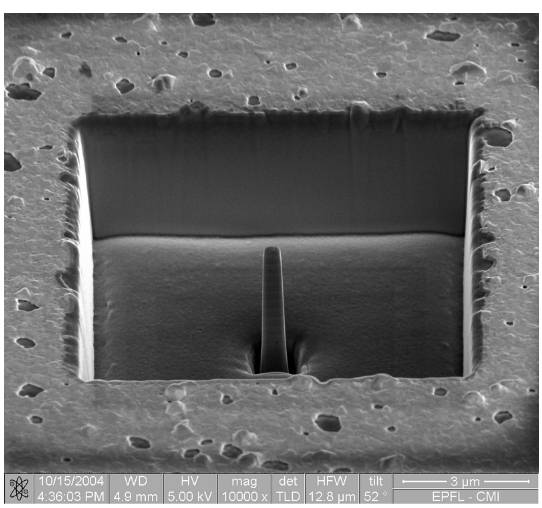
G. Carter and J.S. Colligan, *Ion Bombardment of Solids,* (Elsevier 1968)





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

Milling

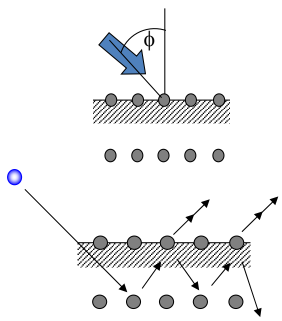


PZT-high aspect ratio „capacitor“, W. Adachi (EPFL-LC)

Material	Sputterrate [µm³/nC]
Si	0.27
Thermal Oxide	0.24
TEOS	0.24
Al	0.3
Al2O3	0.08
GaAs	0.61
InP	1.2
Au	1.5
TiN	0.15
Si3N4	0.2
C	0.18
Ti	0.37
Cr	0.1
Fe	0.29
Ni	0.14
Cu	0.25
Mo	0.12
Ta	0.32
W	0.12
MgO	0.15
TiO	0.15
Fe2O3	0.25
Pt	0.23
PMMA	0.4

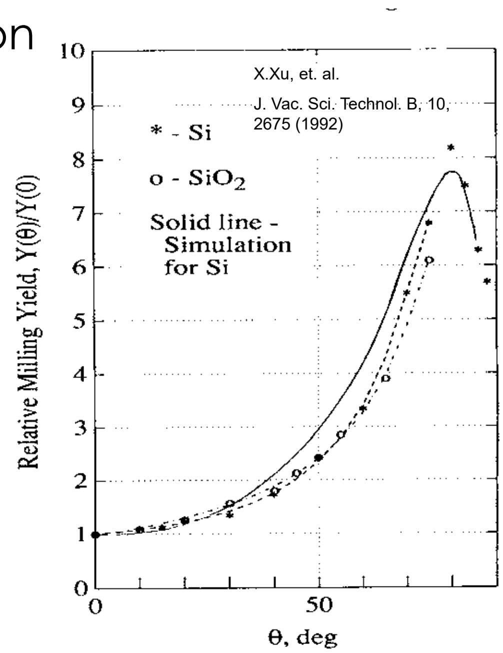
Ion-Solid interaction
Sputtering Yield

Sputtering yield depends on incident angle ϕ

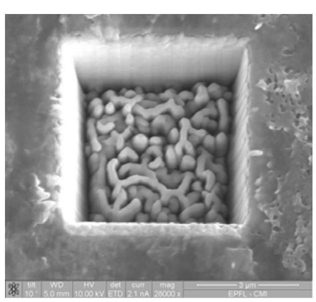


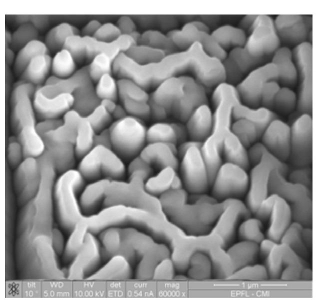
Higher probability of collision cascades near the surface at higher ϕ

Sputtering yield has maximum for ϕ = 75˚

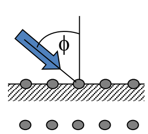


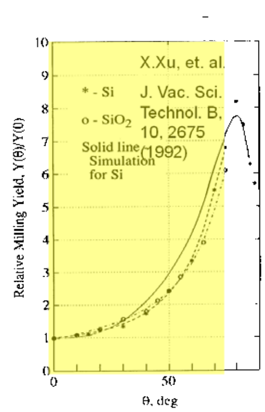
Milling

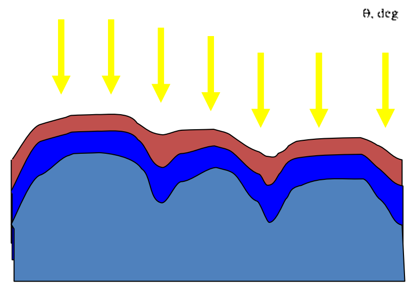




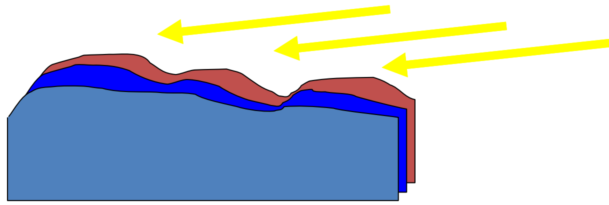
FIB milling of steel

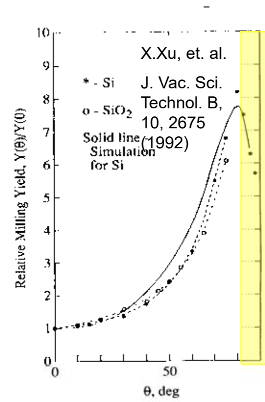


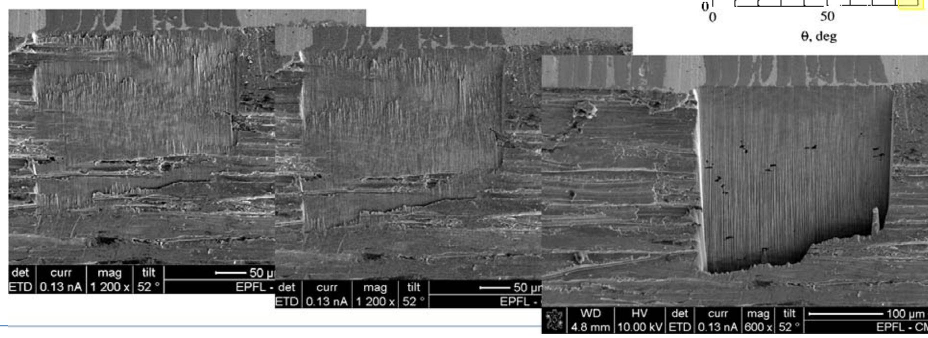




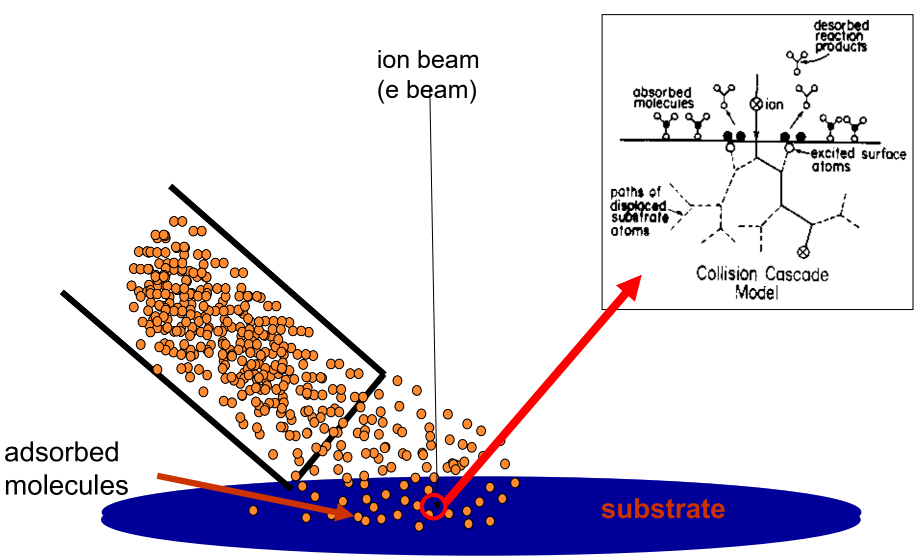
Polishing,
at shallow angles



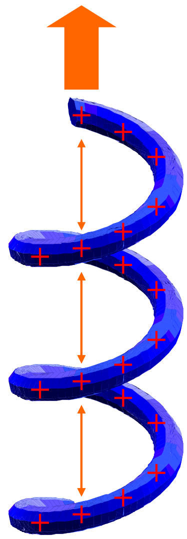


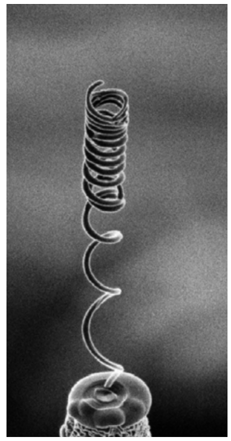


Gas assisted deposition

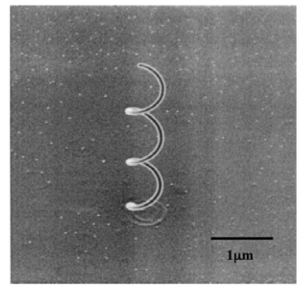


Nanofabricated structures

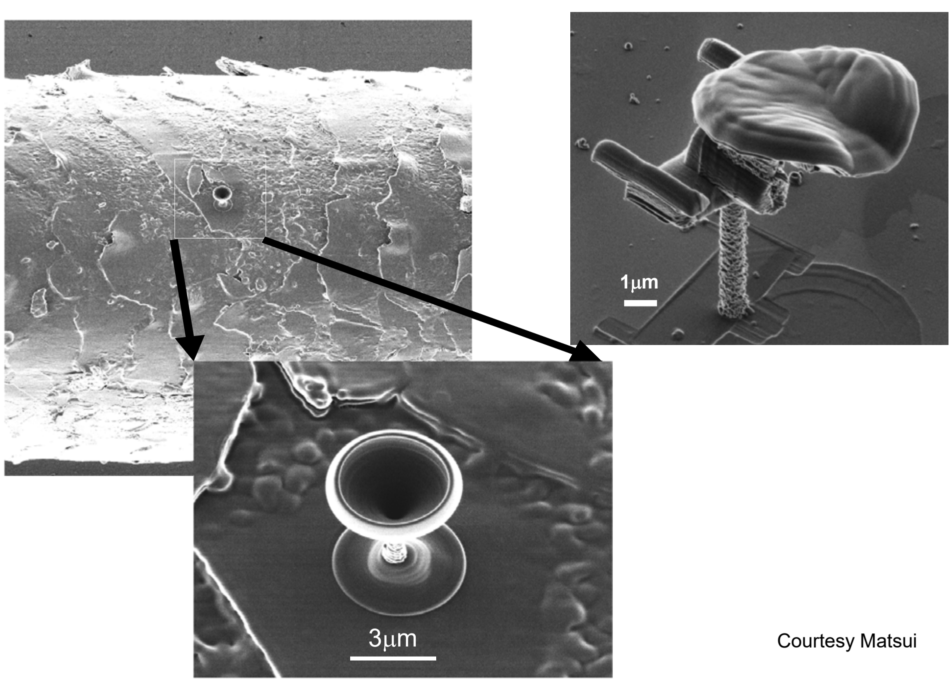




Coil 700nm pitch, 80nm line width,
diamond-like amorphous carbon,
FIB induced CVD



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



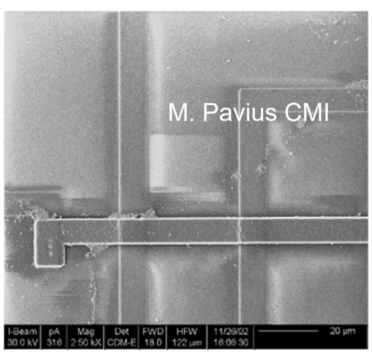
Courtesy Matsui

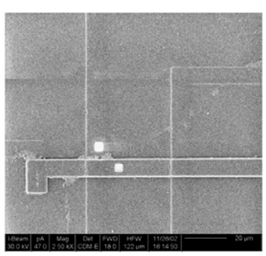
b) Basic Applications

“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

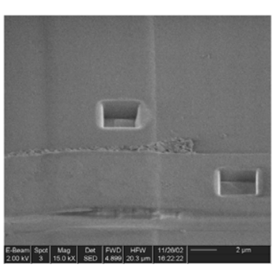


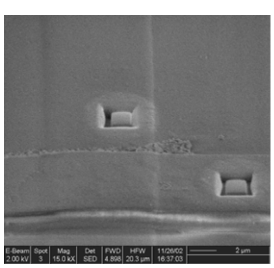


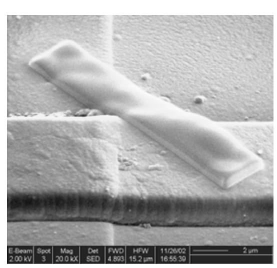
Insertion of electrical connection:

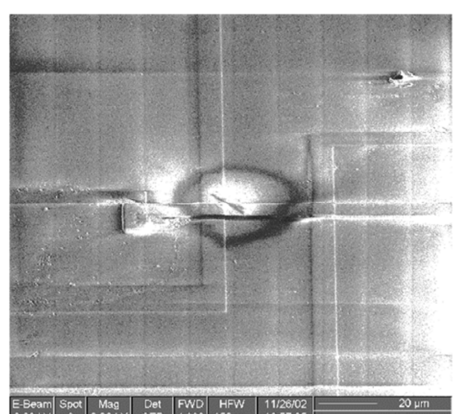
1) Removal of isolating layer (milling)

2) Pt deposition (FIB deposition)

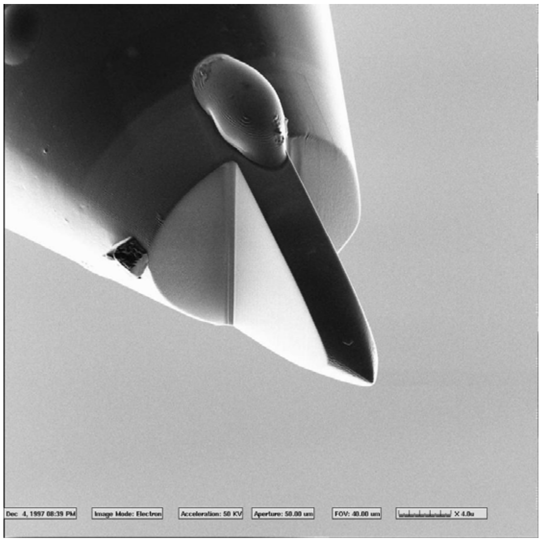






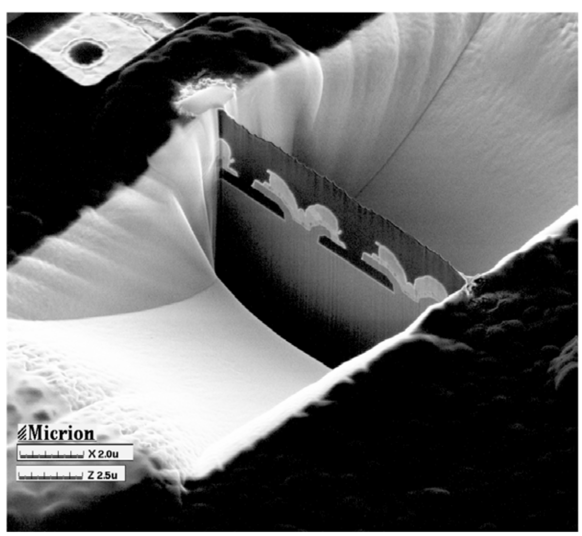


FIB-manufactured AFM-tips

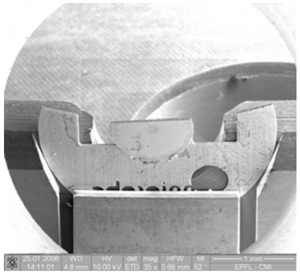


Failure analysis

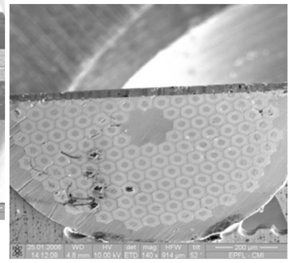
FIB cross-sectioning and
SEM imaging

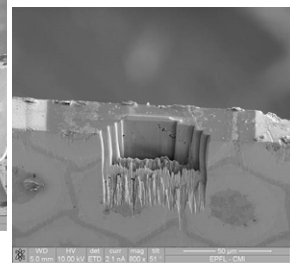


Pre-thinned (H-bar)

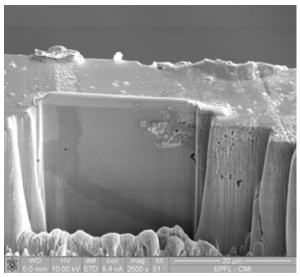


Prethinned sample on TEM-«grid »

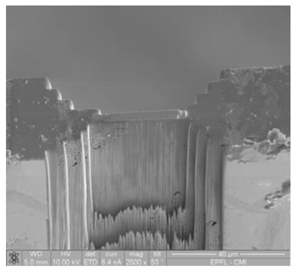




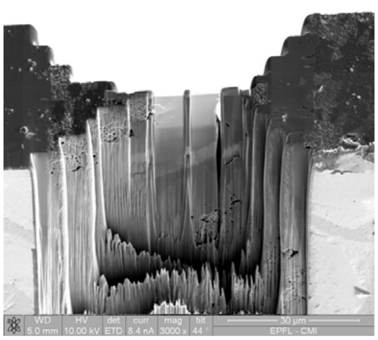
Rough milling at high currents

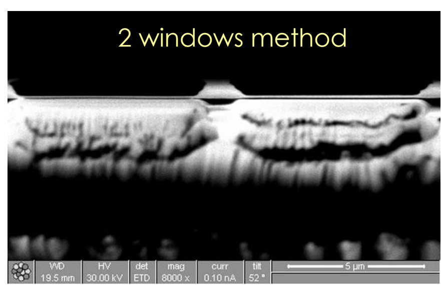


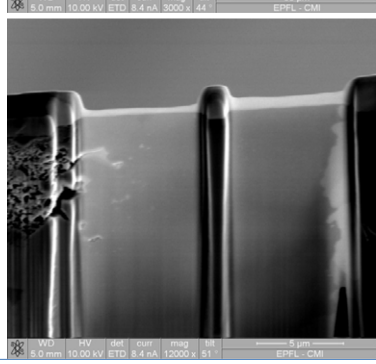
filling of voids

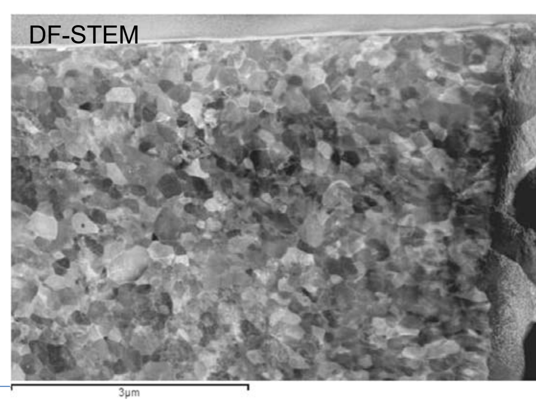


Nb_3Sn superconductor
P-Y. Pfyrter (diploma work)

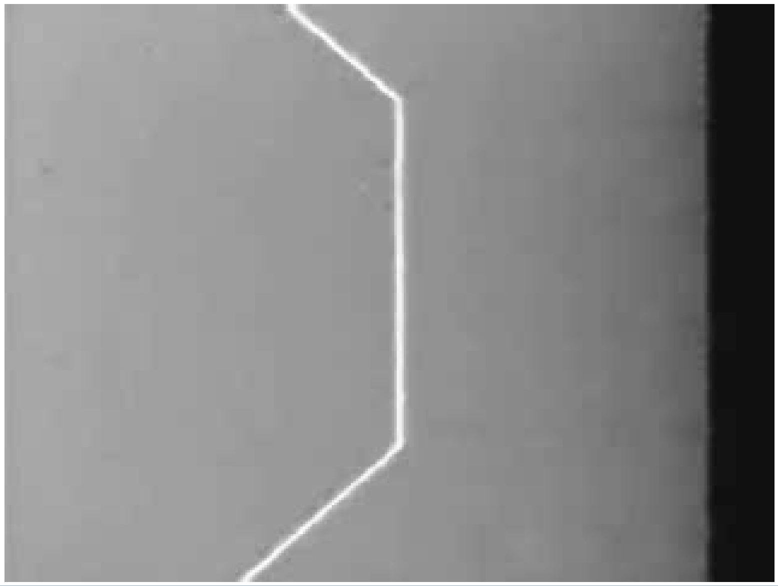






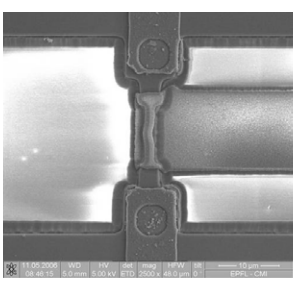


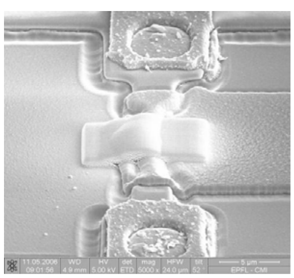
c) TEM preparation
in-situ lift-out movie

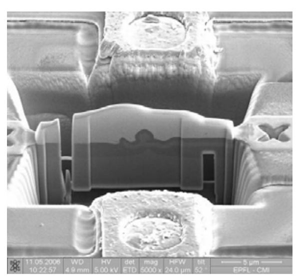


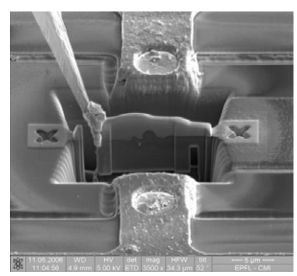
Si nano-wire

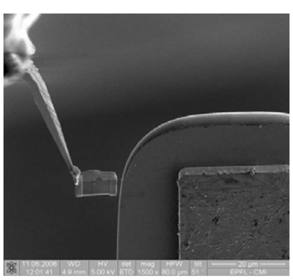
M. Pavius, V. Pott, CMI

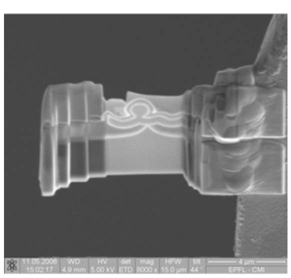






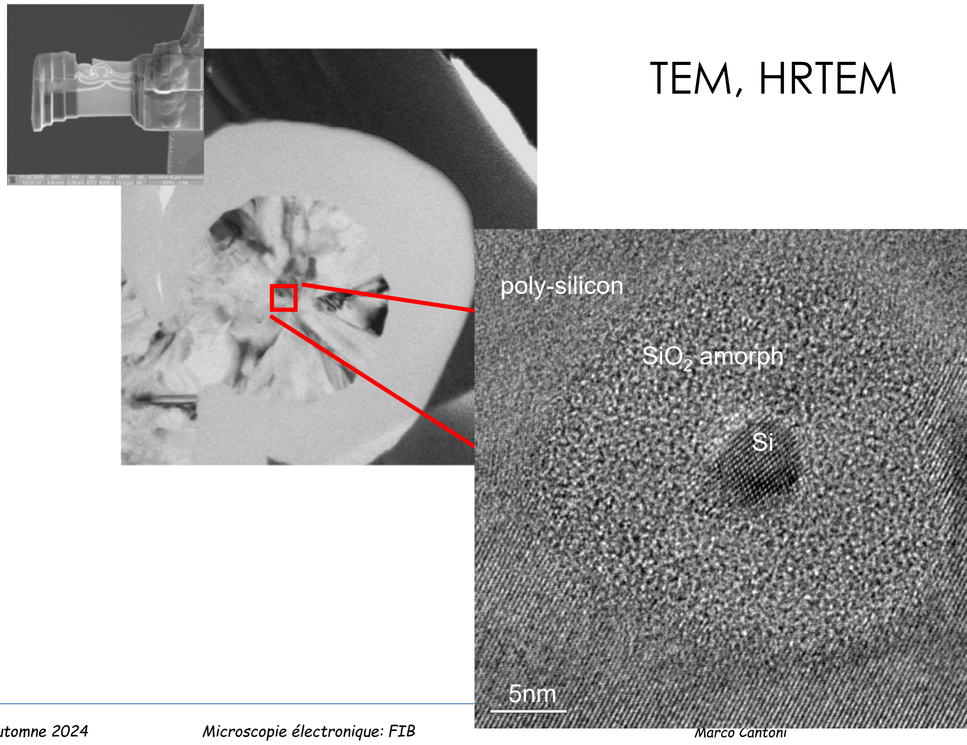






TEM sample “grid” , diameter 3mm

TEM, HRTEM



TEM lamellae by FIB

Focused Ion Beam adds a new dimension to TEM specimen preparation

Take care of artifacts !!!

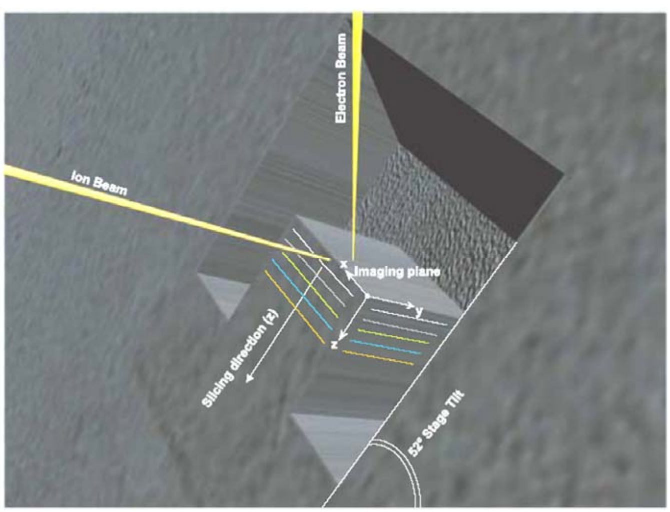
Large (10x5um) flat areas with uniform thickness (50-80 nm)

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

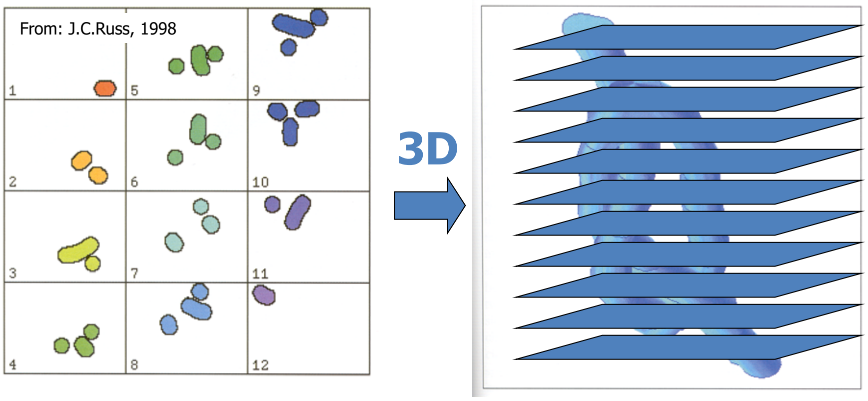
Precise selection of the lamella position possible (devices)

3D Microscopy

d) FIB Nanotomography
3D Microscopy



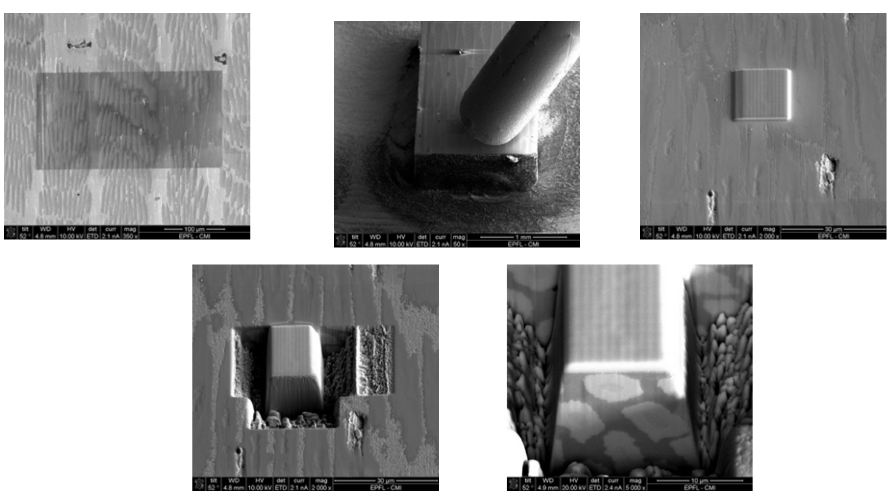
Problem of serial sectioning:
3D-reconstruction of disordered microstructures

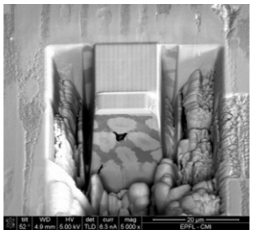


2D Volume fraction

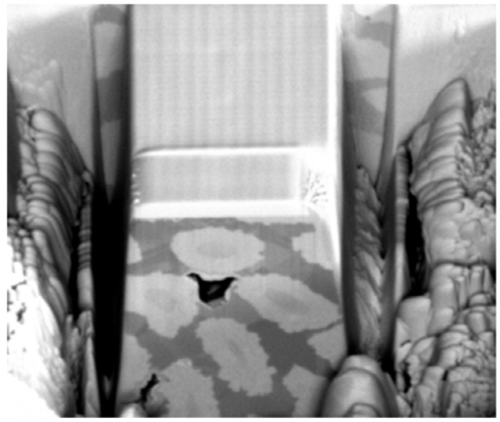
?? Nr of particles ??
?? Shape ??

3D slicing of multifilament
Nb_3Sn superconductor



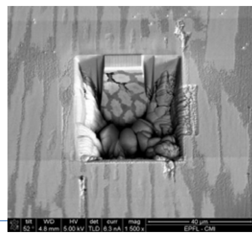


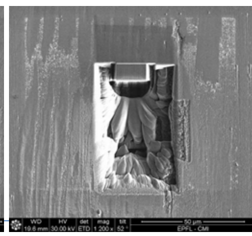
Preparing for slicing



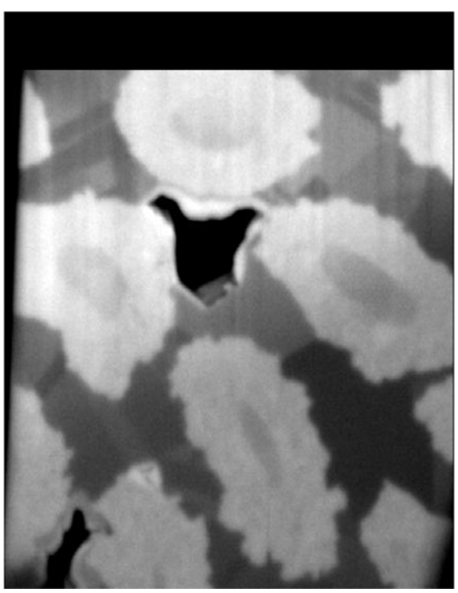
Automated milling and imaging of 170 slices (10h)

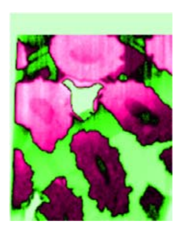
the end





align and crop



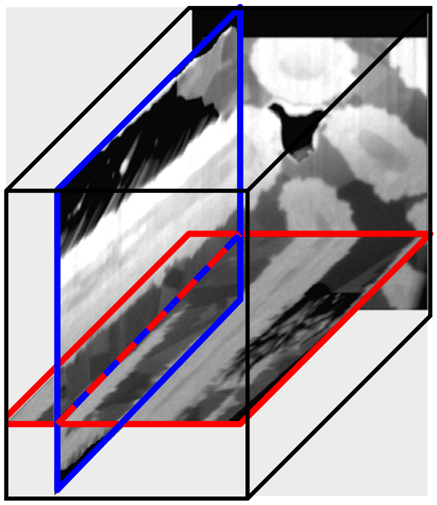


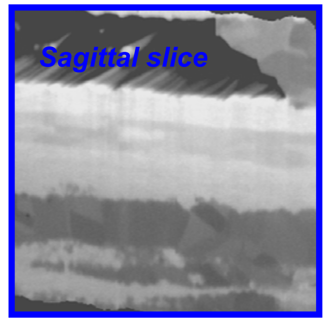
ImageJ

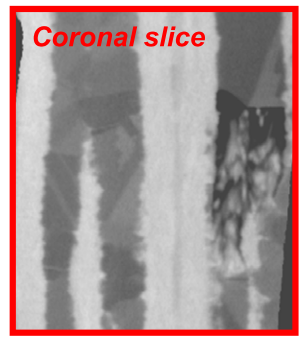
Image Processing and Analysis in Java

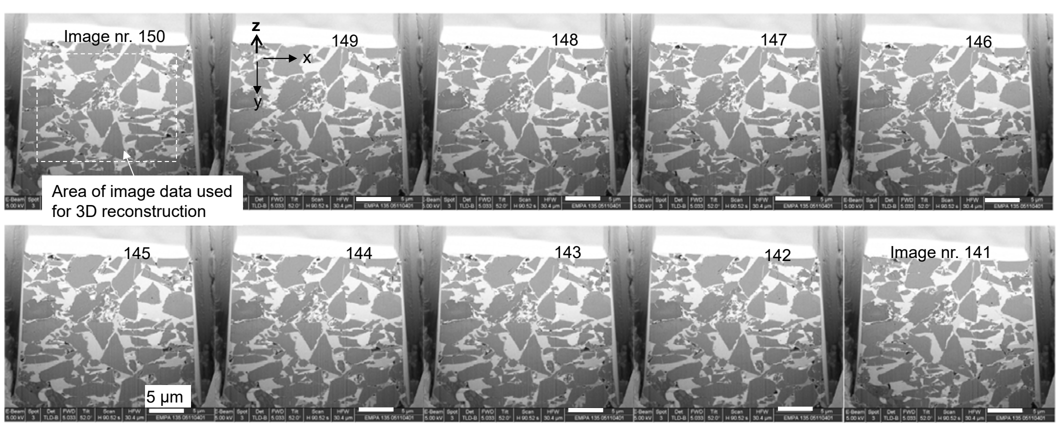
http://rsb.info.nih.gov/ij/index.html

3D volume rendering, reconstruction:
Orthogonal slices







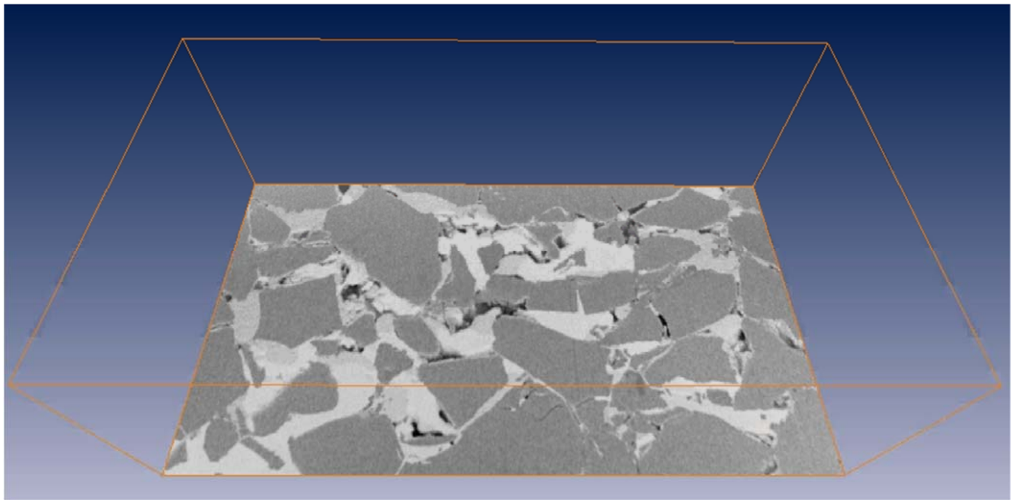


Quantitative microstructure analysis
→ Algorithms
→ object recognition
→ stereological correction of boundary truncation
→ extraction of statistical data (particle shape and size distribution)

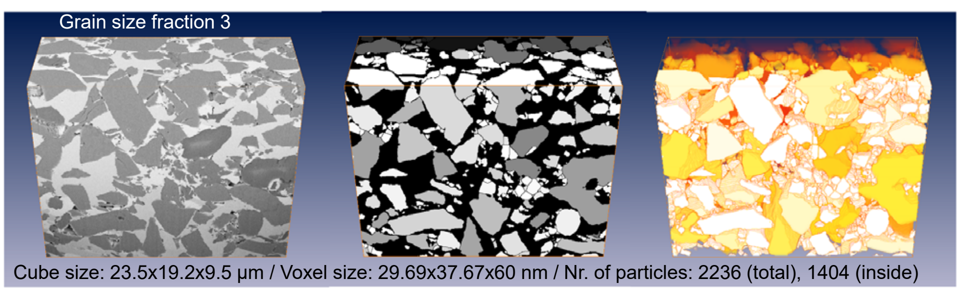
Particle recognition:
Edge detection in 3D,
Watershed for separation

Voxel:
75nm

Cube:
40 * 20 * 15 µm



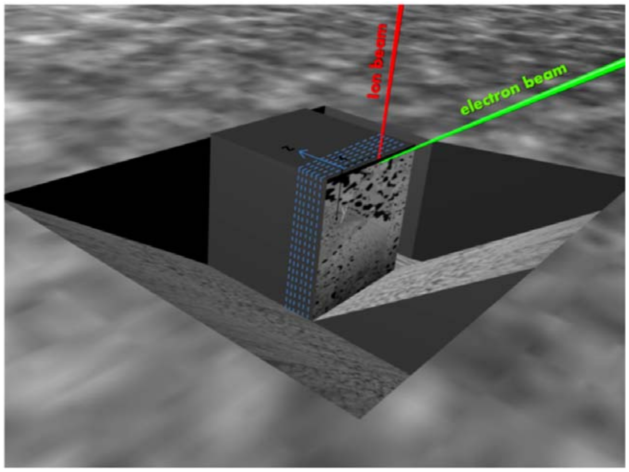
Size, 3D-shape, geometrical relationships between particles



Quantitative microstructure analysis → Algorithms

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

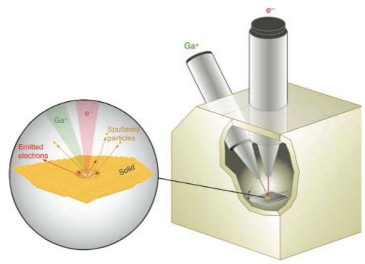
3D FIB/SEM: volume reconstruction

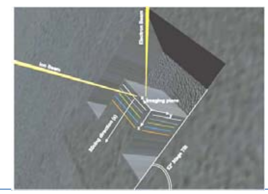


Three-dimensional analysis of porous $BaTiO_3$ ceramics using FIB nanotomography

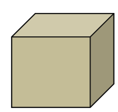
L. HOLZER, F. INDUTNYI, PH. GASSER, B. MÜNCH & M. WEGMANN
EMPA, Swiss Federal Laboratories for Materials Testing and Research, Ueberlandstrasse 129, 8600 Dübendorf, Switzerland

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





“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

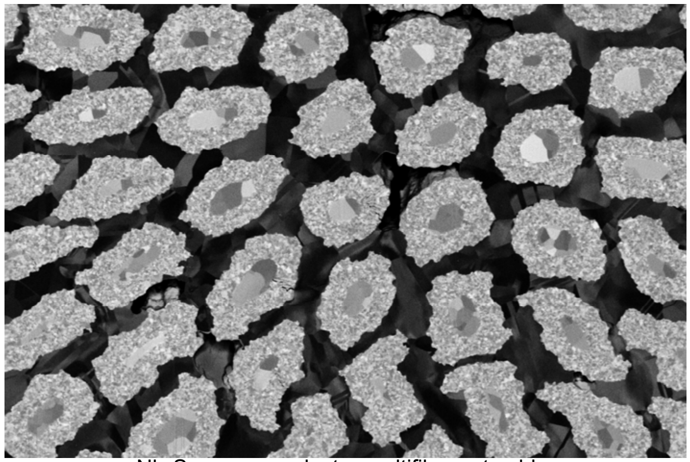
Dwell times/pixel 5- 15µsec. (detector signal -> 256 grey levels)

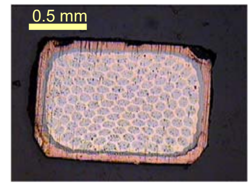
High throughput: minimise overhead, no tilting, rotating, drift correction

Z- Resolution: low kV !!!

3D FIB/SEM: volume reconstruction

Nb_3Sn multifilament superconducting cable

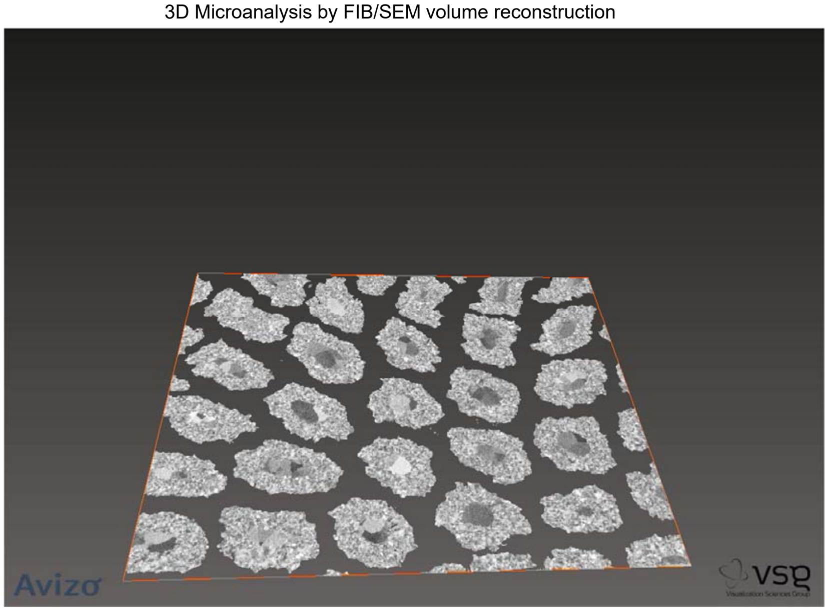




Nb_3Sn superconductor multifilament cable:
14'000 Nb_3Sn filaments (diameter ~5um) in bronze matrix

1.8kV EsB detector: Materials & orientation contrast

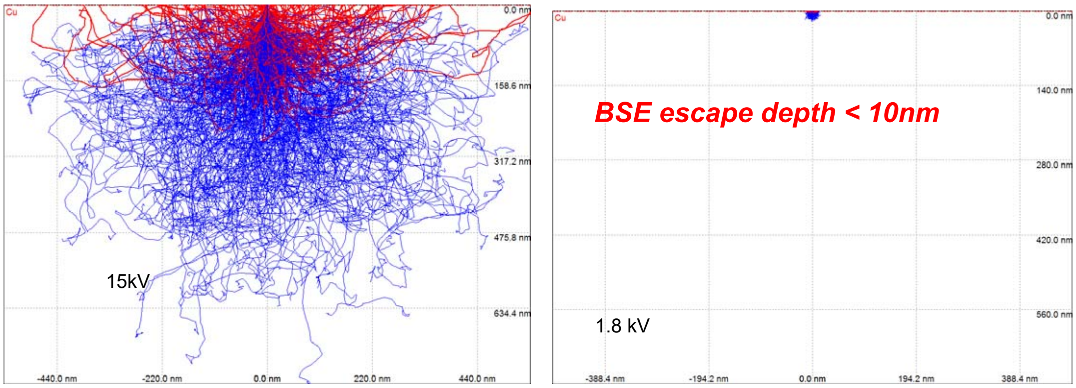
3D Microanalysis by FIB/SEM volume reconstruction



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

What is the spatial resolution …?

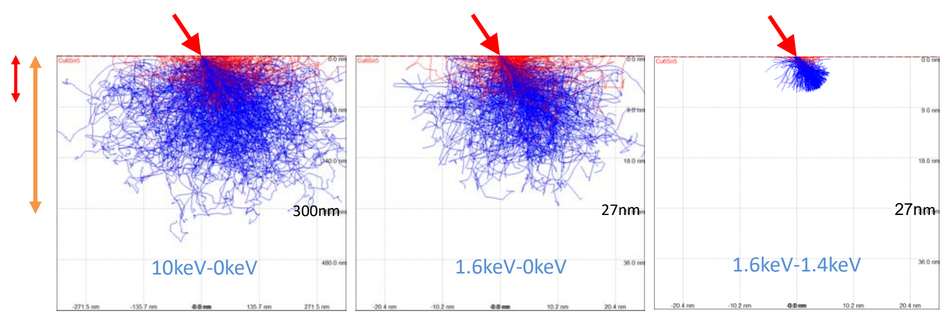
"Classic" *Low kV*



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)

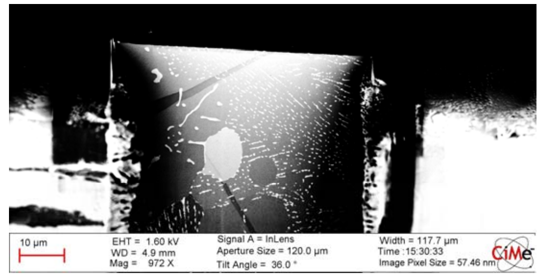
Energy selective BS

3D FIB/SEM: volume reconstruction

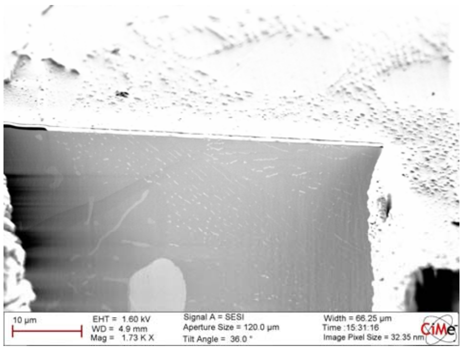
Pb-free solder SnAgCu:

"one detector is not enough"

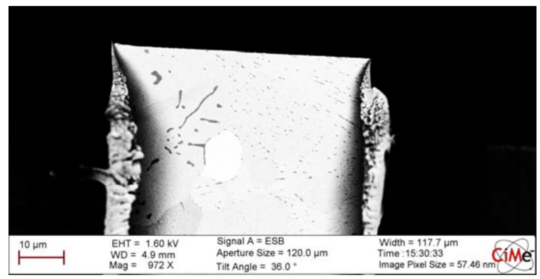
M. Maleki, EPFL-LMAF



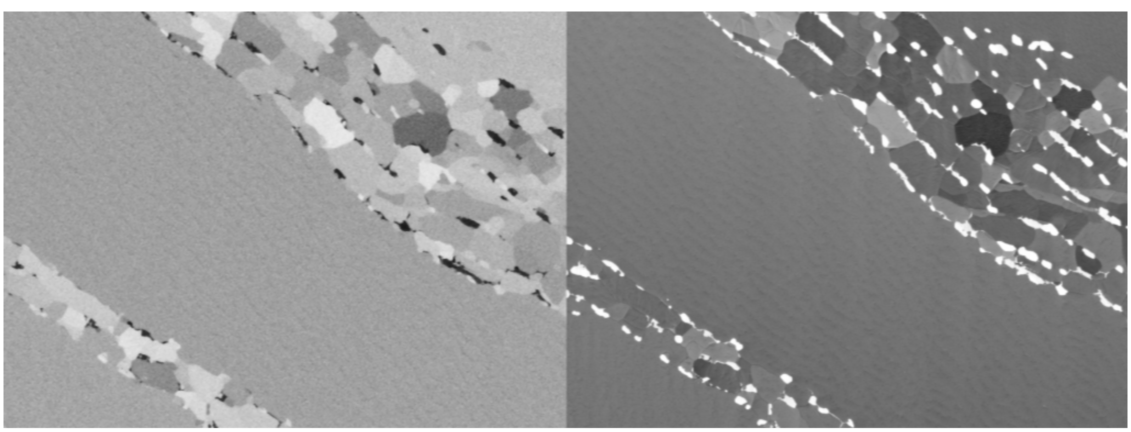
InLens: SE low energy

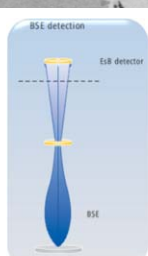


ETD (SE classic)



EsB: Energy selective Backscattered



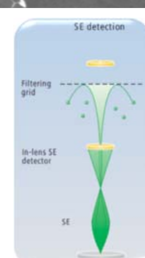


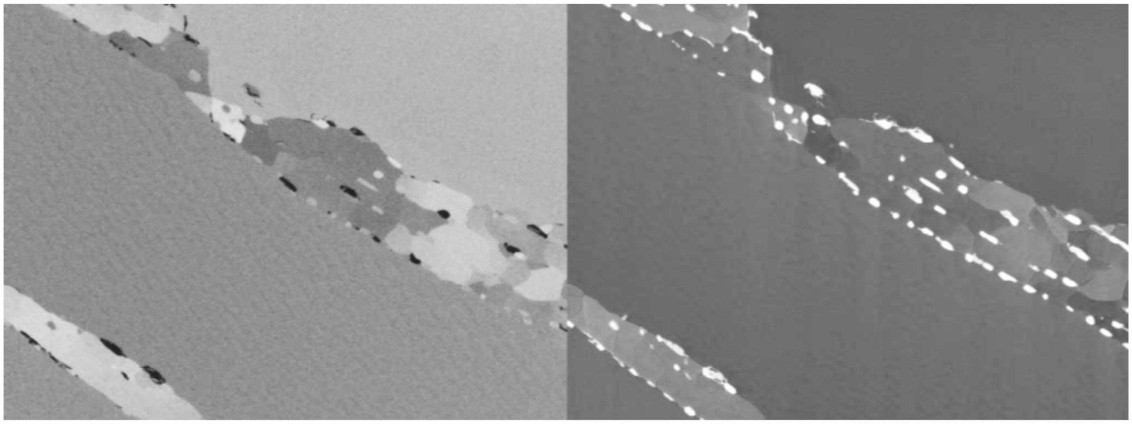
10x10x10nm voxel size, 2048x1536x2000

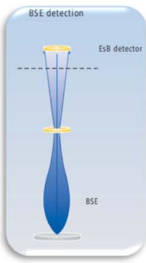
2 images (2x3Mb) / slice ...! (DUAL Channel !)

In parallel: image signal from in-lens SE and also BSE detector

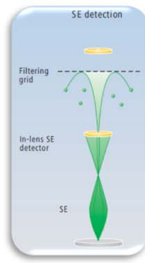
12Gb data



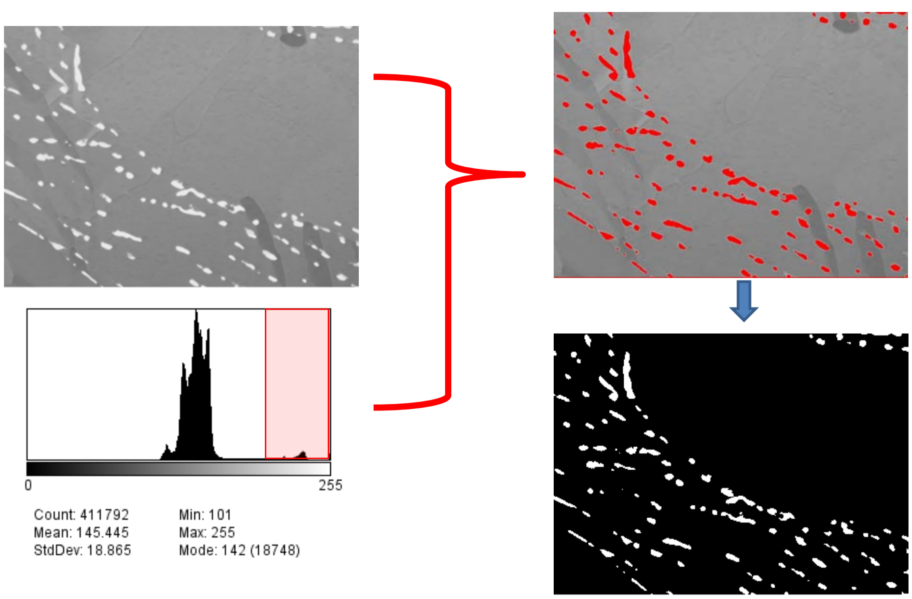


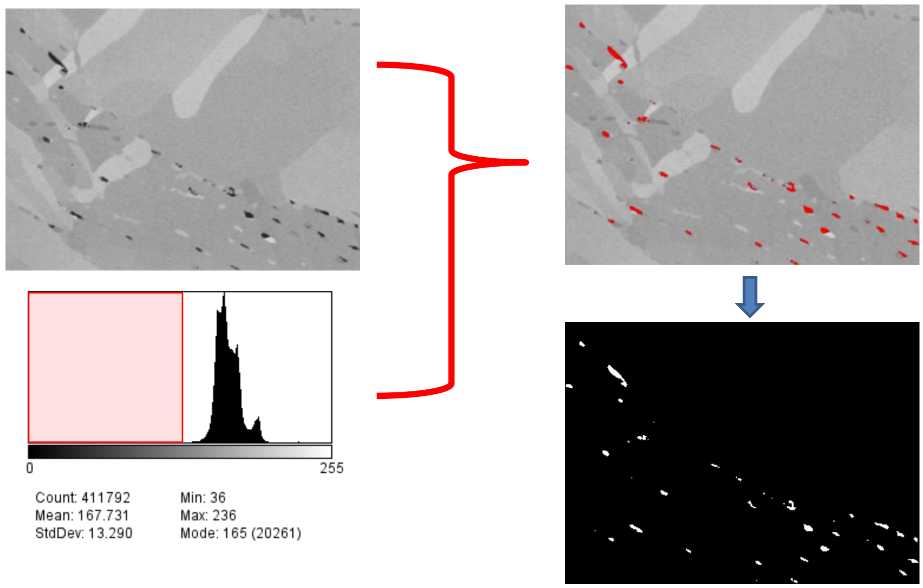


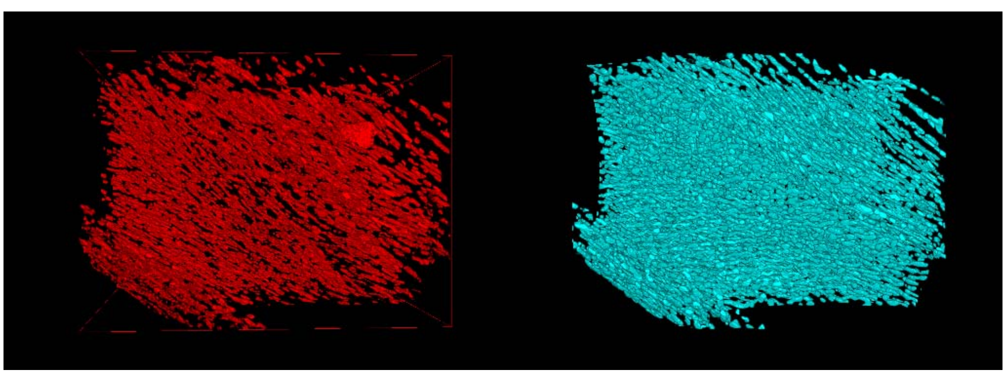
10x10x10nm voxel size, 2048x1536x2000
2 images (2x3Mb) / slice ...! (DUAL Channel !)
12Gb data



segmentation = identification of phases



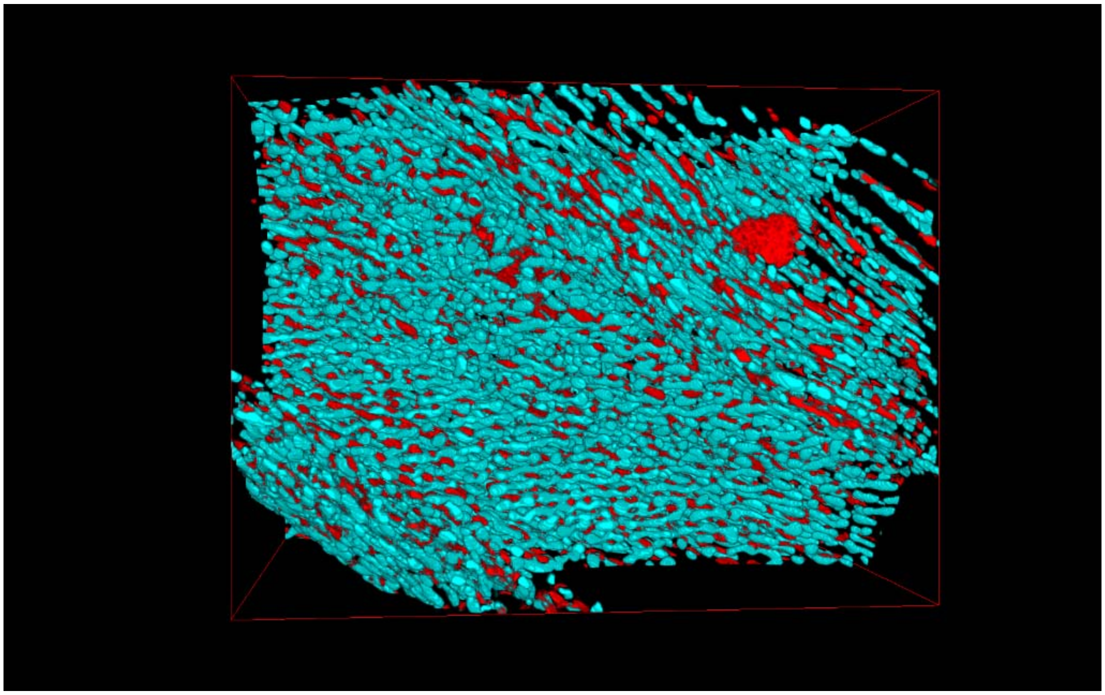


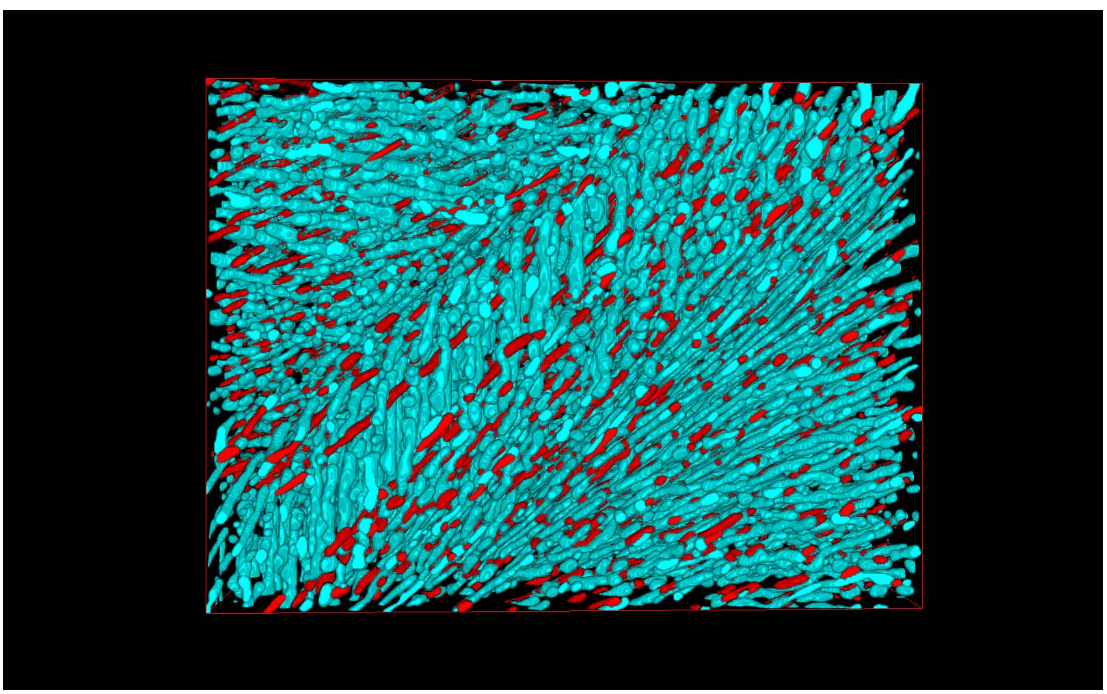


Phase 1: **Cu_6Sn_5**
Dark in EsB image
White in SE-InLens

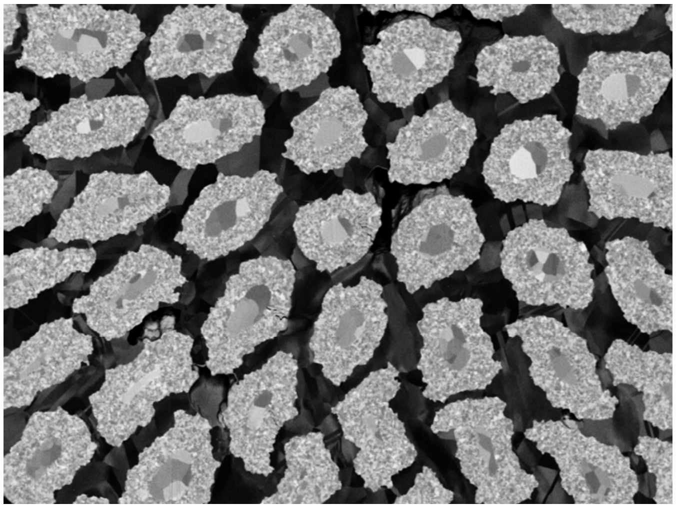
Phase 2: **Ag_3Sn**
White in SE-InLens - Dark in EsB image

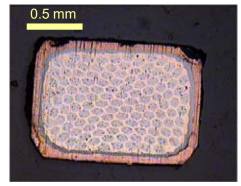
10x10x10nm voxel size, 2048x1536x2000 pixel/slices
2 images (3Mb) / slice …… 12Gb data
32 hours





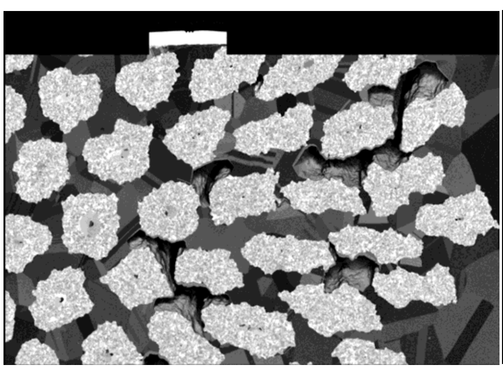
Nb_3Sn multifilament superconducting cable



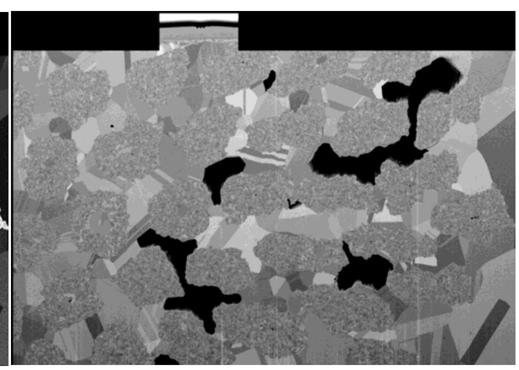


Nb_3Sn superconductor multifilament cable:
14'000 Nb_3Sn filaments (diameter ~5um) in Cu matrix

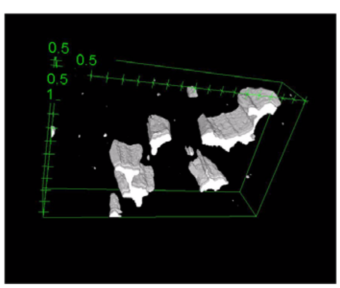
1.8kV EsB detector: Materials & orientation contrast

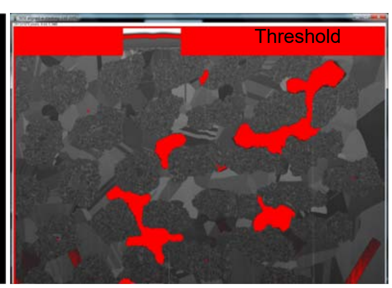


EsB



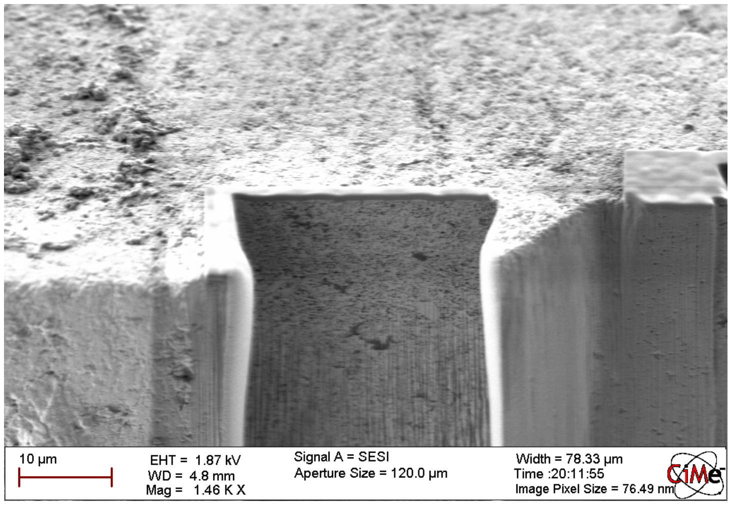
SE





Solid Oxide Fuel Cell cathode

P. Tanasini, LENI



Porous, non-conducting:
Resin infiltration, gold coating

The right conditions

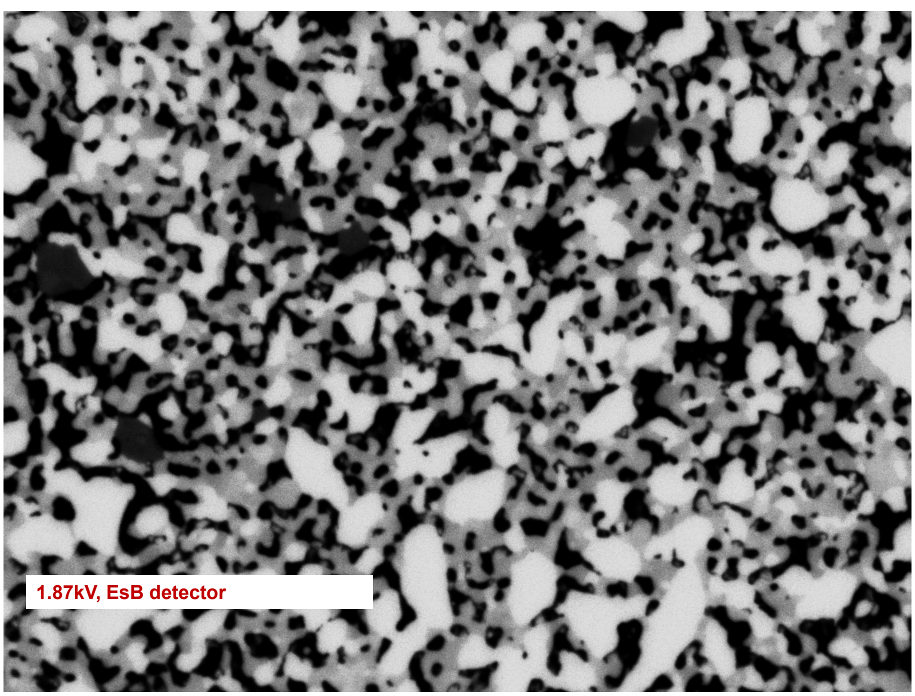
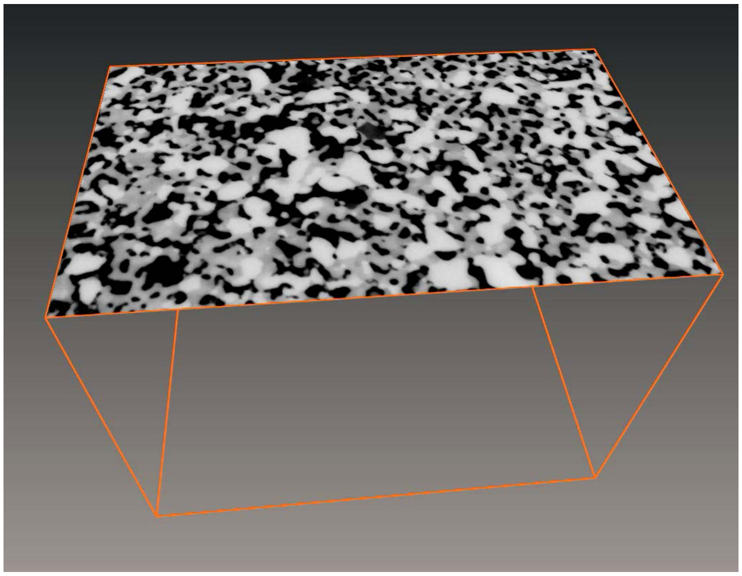
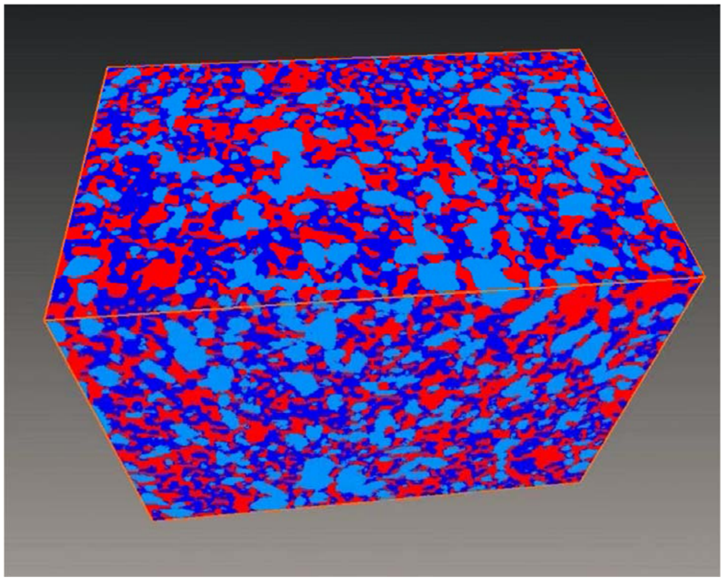


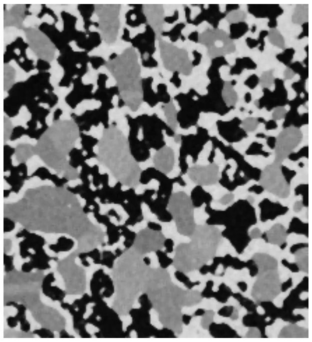
Image:
2048x1536
10nm pixel size

2200 images
36hours

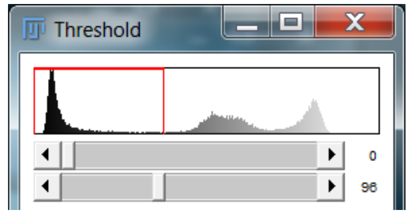


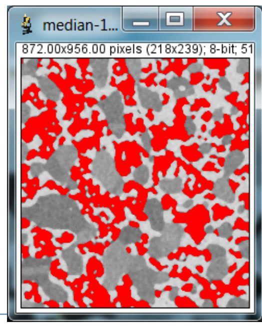


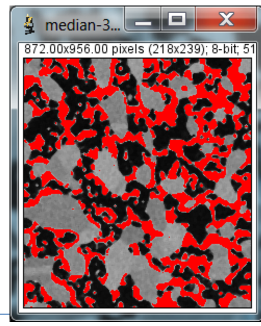
Segmentation and analysis

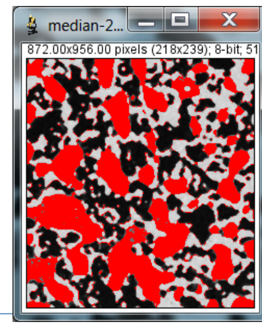


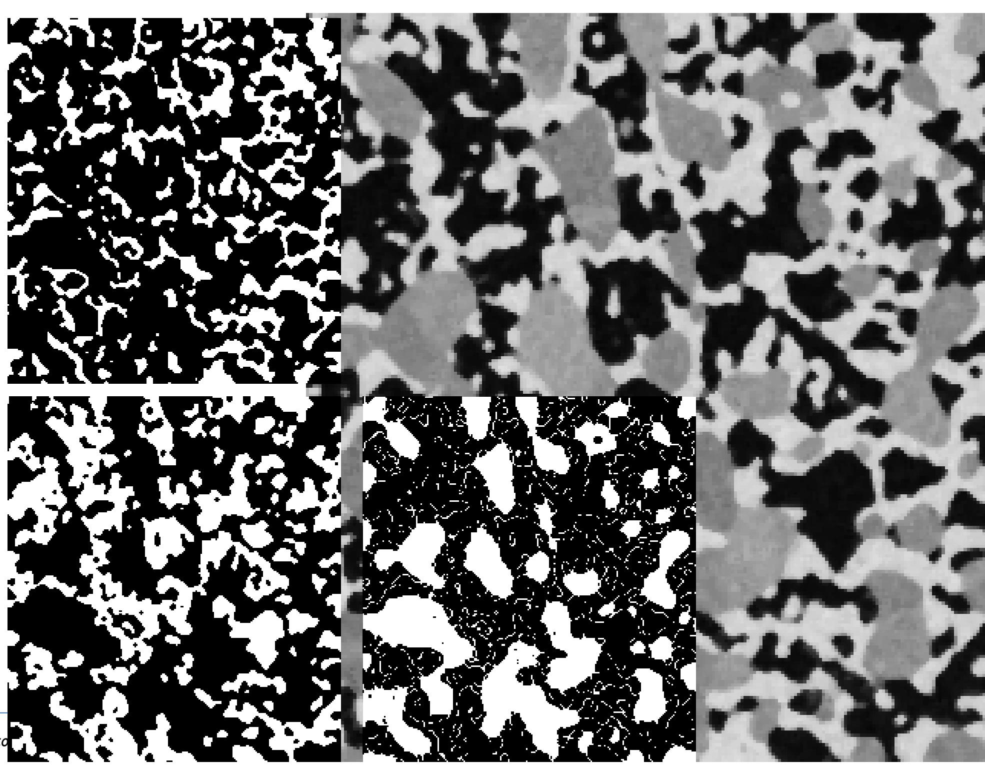
thresholding



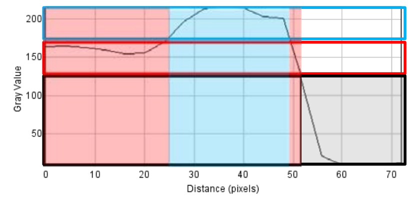


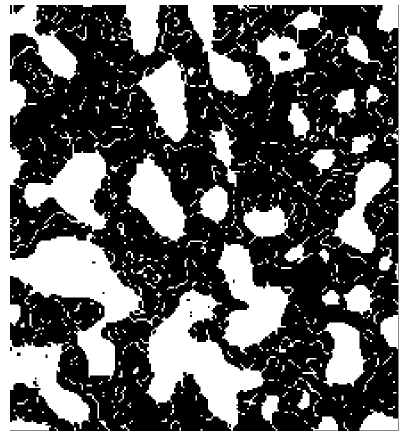


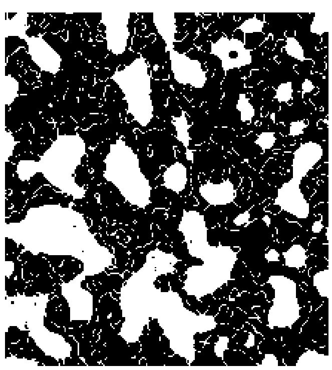




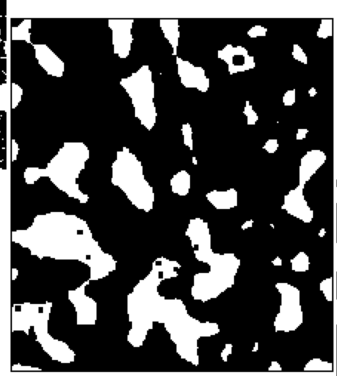
The gradient problem…



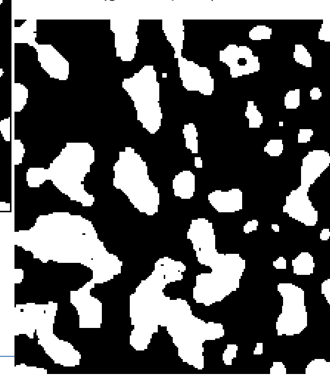


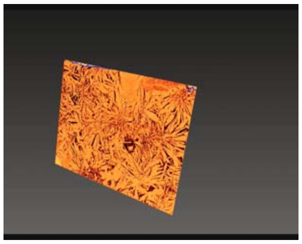


Erode
(shrink 1 pixel)

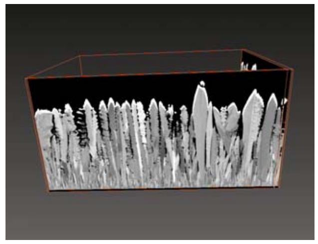


Dilate
(grow 1 pixel)

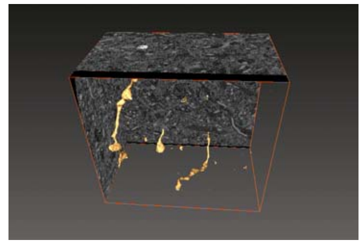




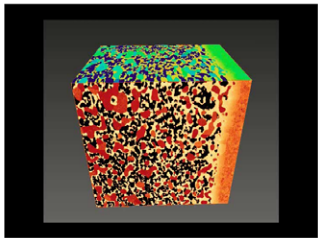
Cement, $(10nm)^3$ voxel



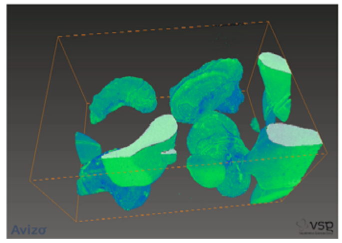
Solar cell: ZnO, $(10nm)^3$ voxel



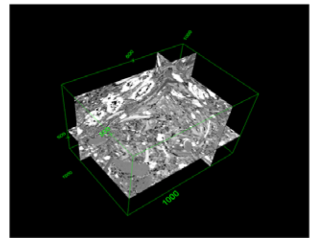
Rat brain, $(5nm)^3$ voxel



SOFC, $(10nm)^3$ voxel



Malaria parasite, $(8nm)^3$ voxel

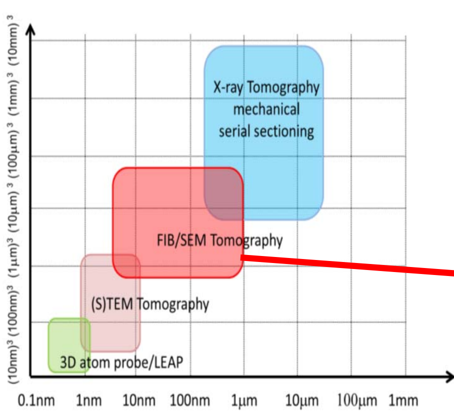


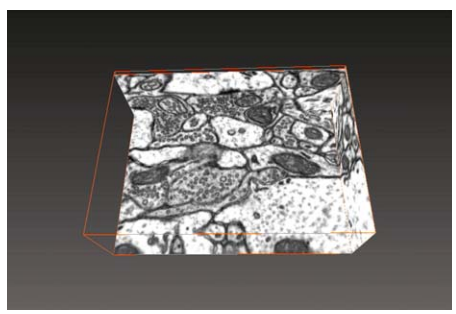
clay, $(10nm)^3$ voxel

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