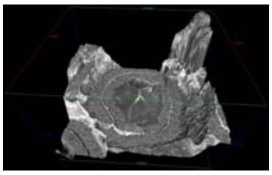
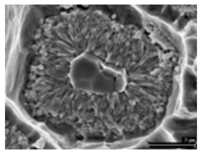
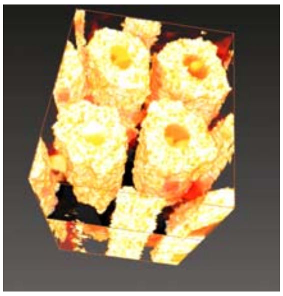
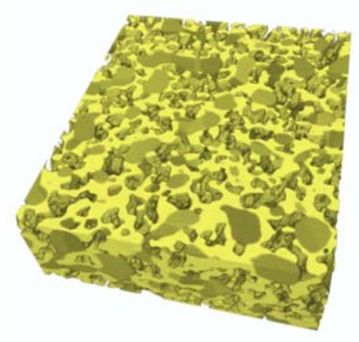
3D Microscopy and FIB Nanotomography





Marco Cantoni
CIME
mxc-133, 34816





Focused Ion Beam

a) Principles

How does it work..?

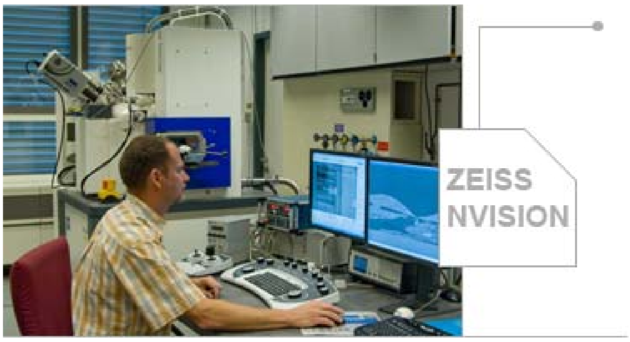
Ion source, optics, interaction with the sample

b) Basic Application

Imaging, milling, deposition, typical applications
TEM sample preparation, examples

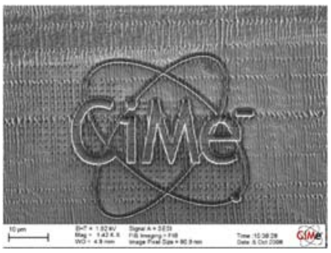
c) FIB Nanotomography, 3D microscopy

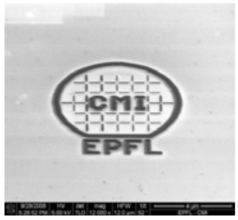
FIB @ EPFL



@CIME: since summer 2008
ZEISS NVision 40

@CMI: since 2004
FEI Nova nanolab 600
clean room installation

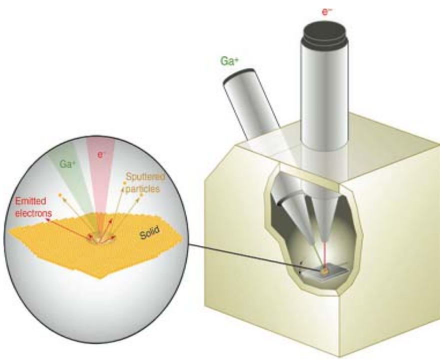




A modern FIB (Focused Ion Beam) system in a research lab (« lab » systems)

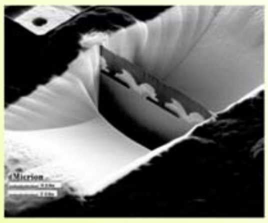
a complete state of the art (high -performance) SEM equipped with

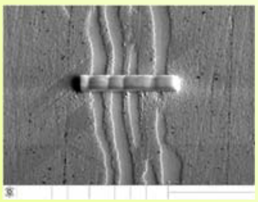
a) focused ion column
b) Gas injector system
c) micromanipulators

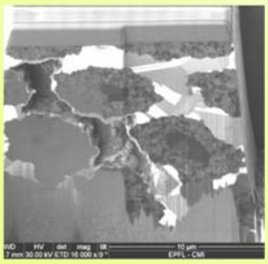


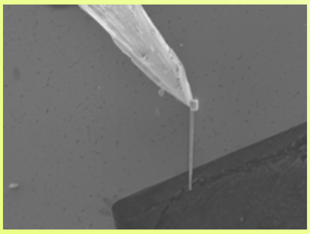
Dual beam ®, crossbeam ®

Dual Beam Nova 600 Nanolab
from FEI Company









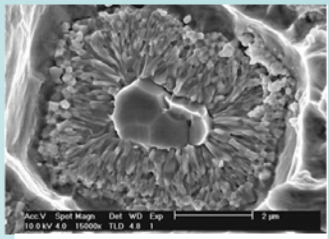
Ion Beam & Electron Beam

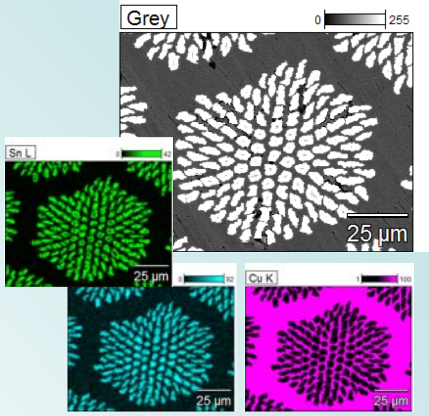
SEM: Imaging and Analysis

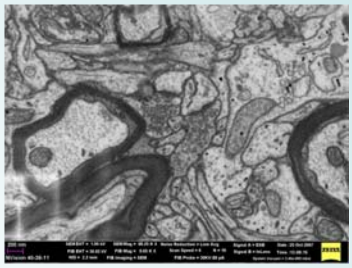
- High resolution (1-2nm) SEM
- SE, general imaging, topography contrast
- BSE, chemical (mass density contrast) contrast
- EDX microanalysis (point analysis and element mapping)
- Low voltage SE and BSE imaging (small interaction volume=high resolution), compatible with "non"-conducting and biological specimens

FIB: Nano-machining

- Machining (sputtering)
- chemically assisted deposition and etching (gas injector system)
- Ion beam induced imaging (channeling contrast), SE and SI
- Micromanipulation (multiple micromanipulators) of small objects (<10nm precision)
- ***Nano-scale "laboratory"***



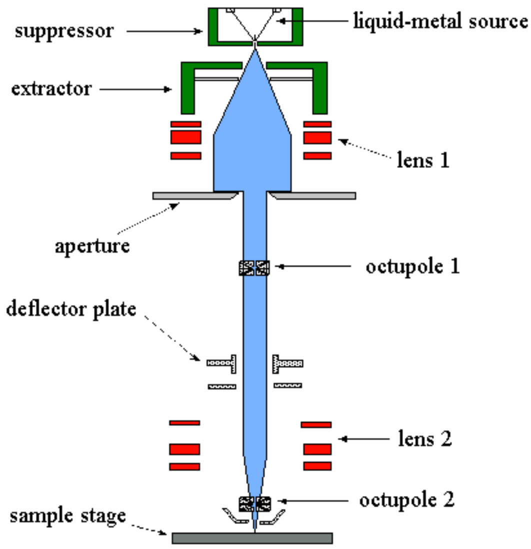




Focused Ion Beam

- 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

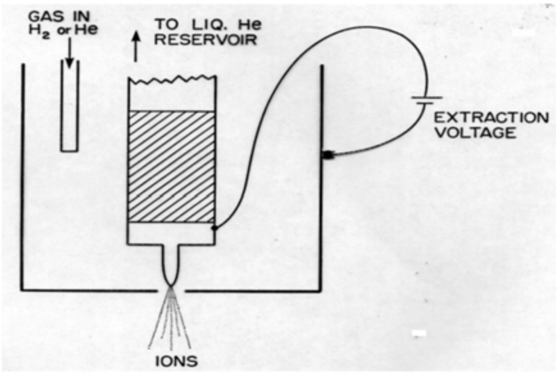


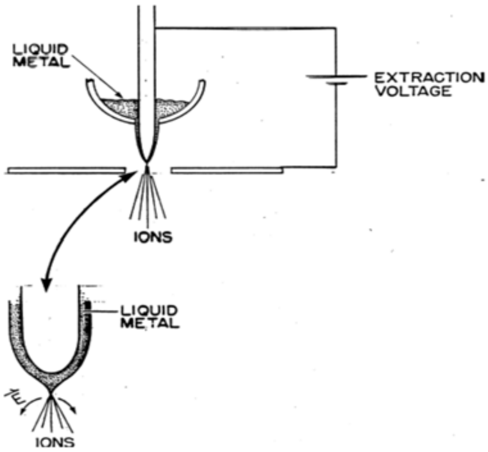
Schematic diagram of a FIB ion column
Source: IBM Almaden Research Center

Ion Sources

a) Gas Field Ion Source

b) Liquid Metal Source





Type of ion source	Ion Species	Virtual source size (nm)	Energy spread, ΔE (eV)	Unnormalized brightness, B (A/cm^2sr)	Angular brightness ($\mu A/sr$)
Liquid metal	Ga^+	50	>4	$3x10^6$	50
Gas field ion (supertip) (ref. 11)	H^+, H_2^+, He^+, Ne^+...	0.5	~1	$5x10^9$	35

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)

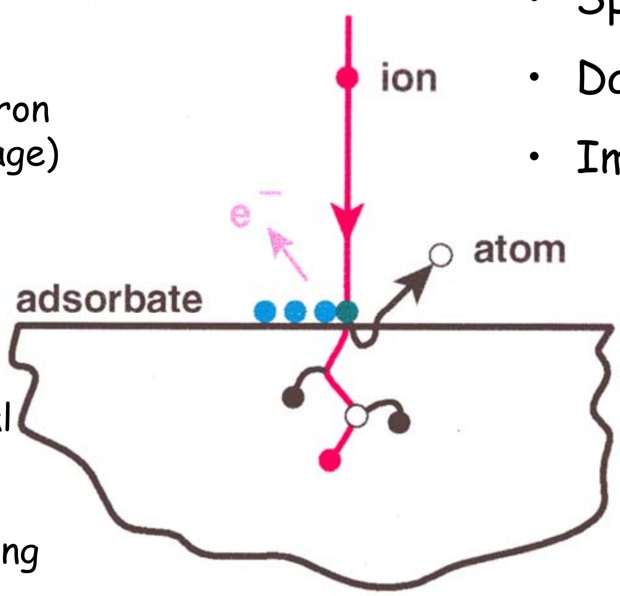
- **Ions**
- Big
  ->outer shell reactions (no x-rays)
- High interaction probability
  less penetration depth
- Ions can remain trapped -> doping
- High mass -> slow speed but high momentum
  milling !!!
- Ions are positive
- Electrostatic lenses

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

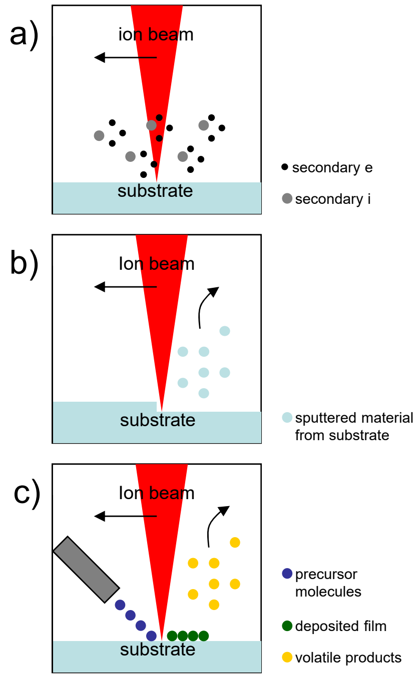
- Secondary electron emission (SE-image) 2-3 SE per Ion !
- Surface chemical reactions
  - deposition
  - enhanced etching



- Sputtering
- Damage
- Implantation

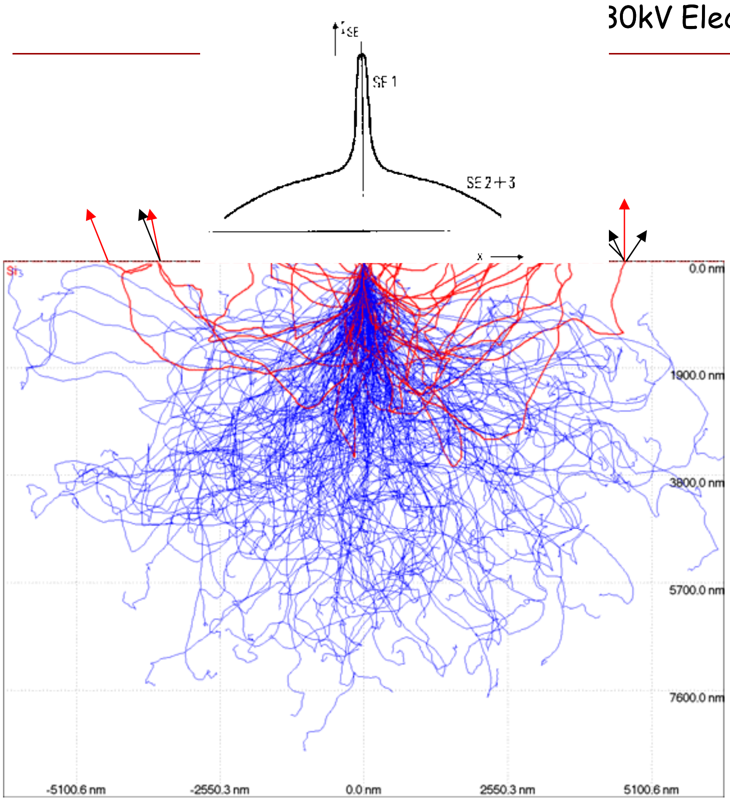
3 basic "operating modes"

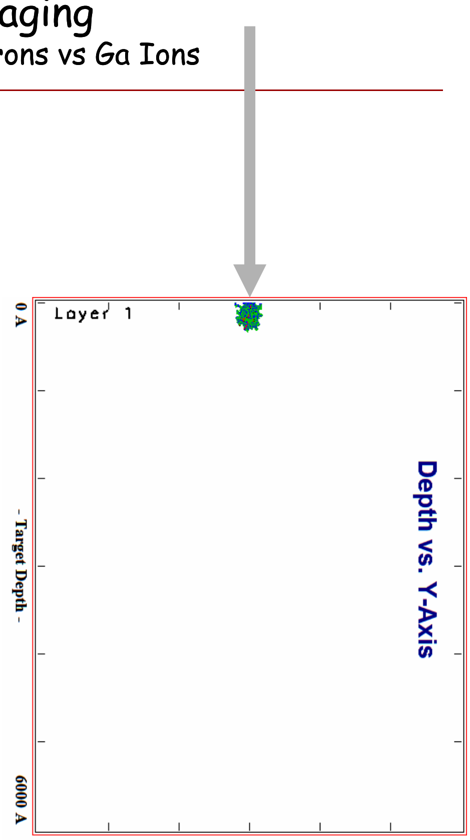
- Emission of secondary ions and electrons
  - FIB **imaging** ☛ a)
    low ion current
- Sputtering of substrate atoms
  - FIB **milling** ☛ b)
    high ion current
- Chemical interactions (gas assisted)
  - FIB **deposition**
  - Enhanced (preferrential) etching ☛ c)
- Other effects:
- Ion implantation
- Displacement of atoms in the solid
  - Induced damage
- Emission of phonons
  - Heating



Imaging

30kV Electrons vs Ga Ions

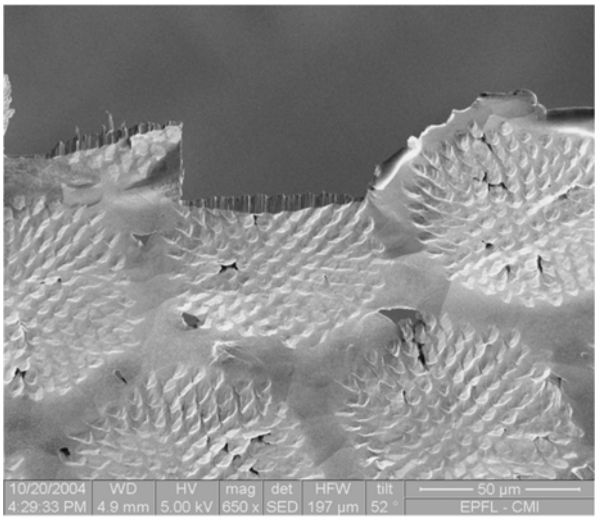




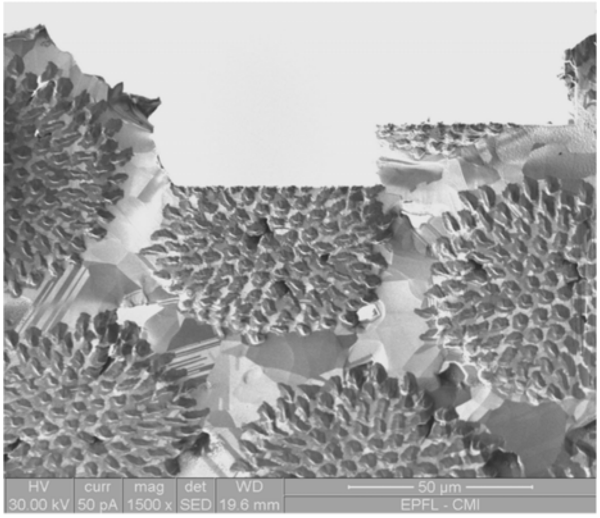
Monte-Carlo Simulation casino v2.42
http://www.gel.usherbrooke.ca/casino/download2.html

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

SE image contrast

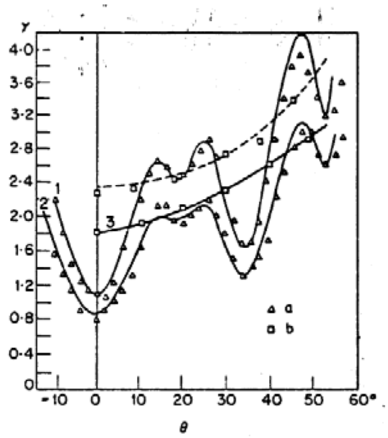


e-beam 5kV



ion-beam 30kV 50pA

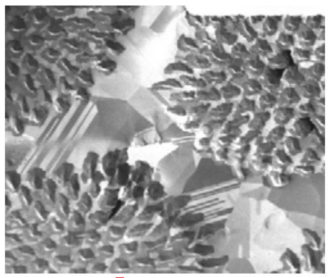
material (sputtering) contrast
orientational contrast

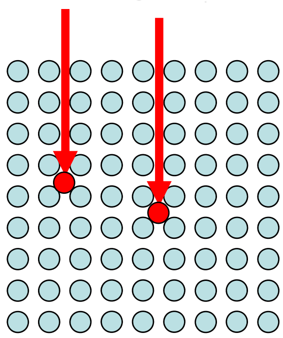


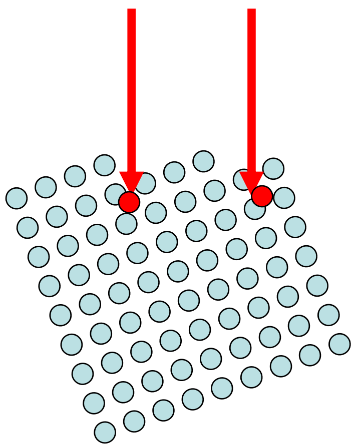
Channeling contrast

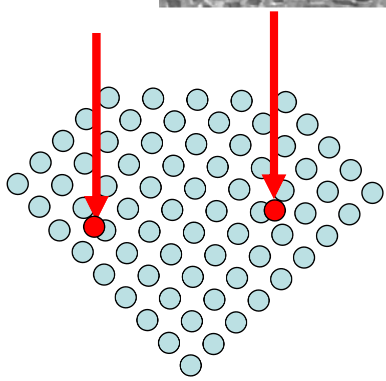
Secondary Electron Emission Coefficient vs. Angle, (100) Copper 30 keVAr Ions

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





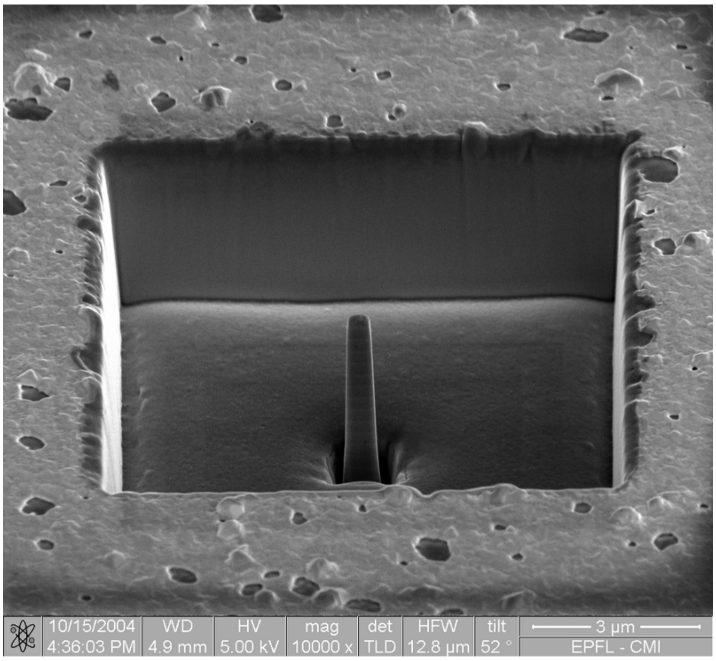




Atom columns align with the ion trajectory = higher penetration
-> less sputtering and less SE electrons

Milling

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



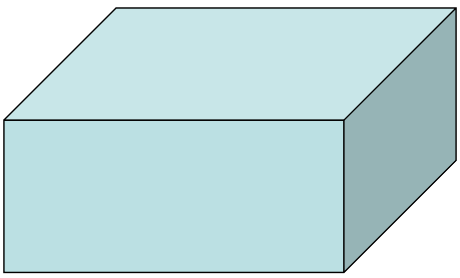
PZT-high aspect ratio „capacitor", W. Adachi (EPFL-LC)

Milling rate

- Sputter rate for a 10x10x5µm box in Cu
- Typical ion current (high) 10nA
- Sputter Yield for Cu: 0.25µm³/nC

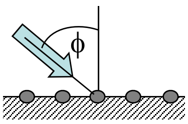
Volume: 500 um³

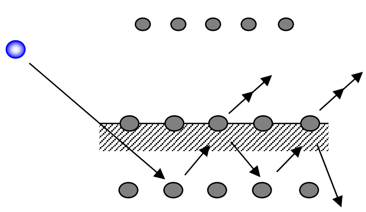
Time: Volume/sputter rate = 2000 sec. = 33 min.



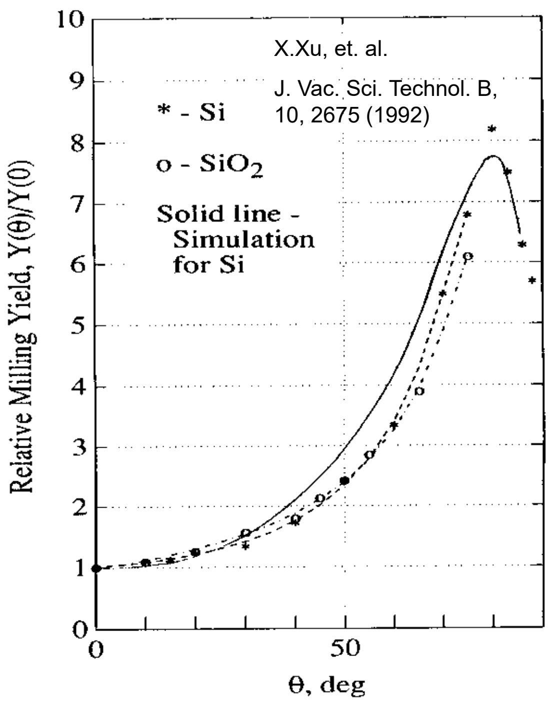
Ion-Solid interaction
Sputtering Yield

- Sputtering yield depends on incident angle ϕ

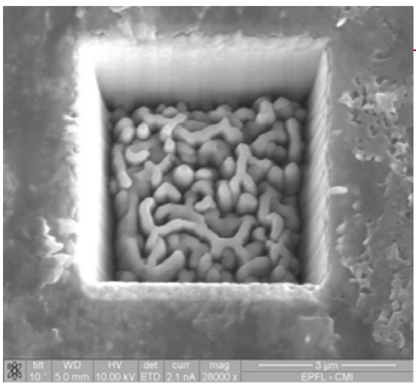




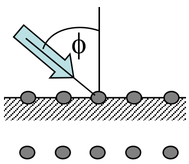
- Higher probability of collision cascades near the surface at higher ϕ
- Sputtering yield has maximum for ϕ = 75°

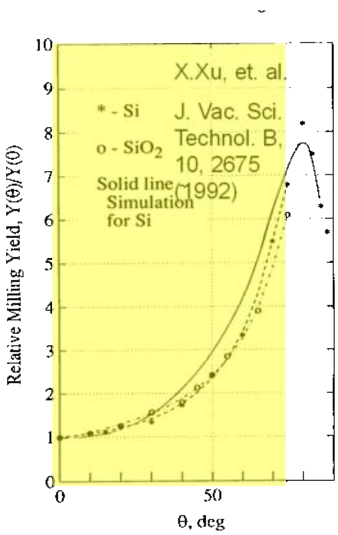


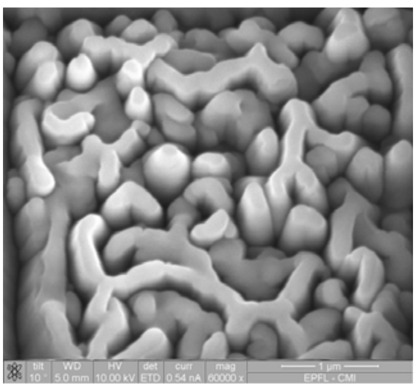
Milling

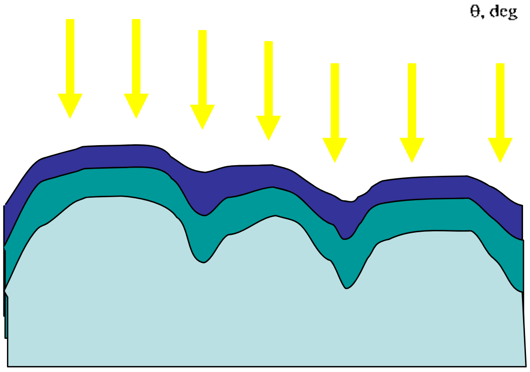


FIB milling of steel

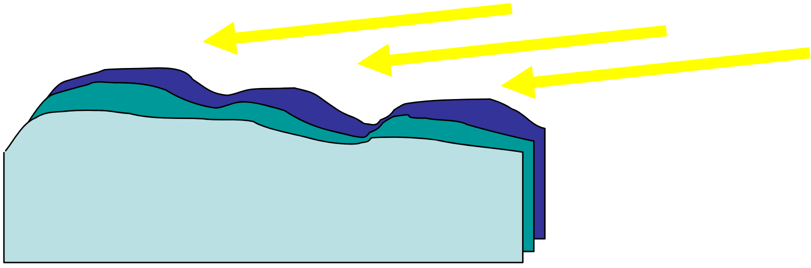


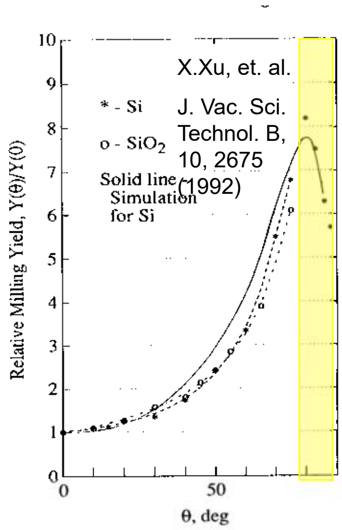


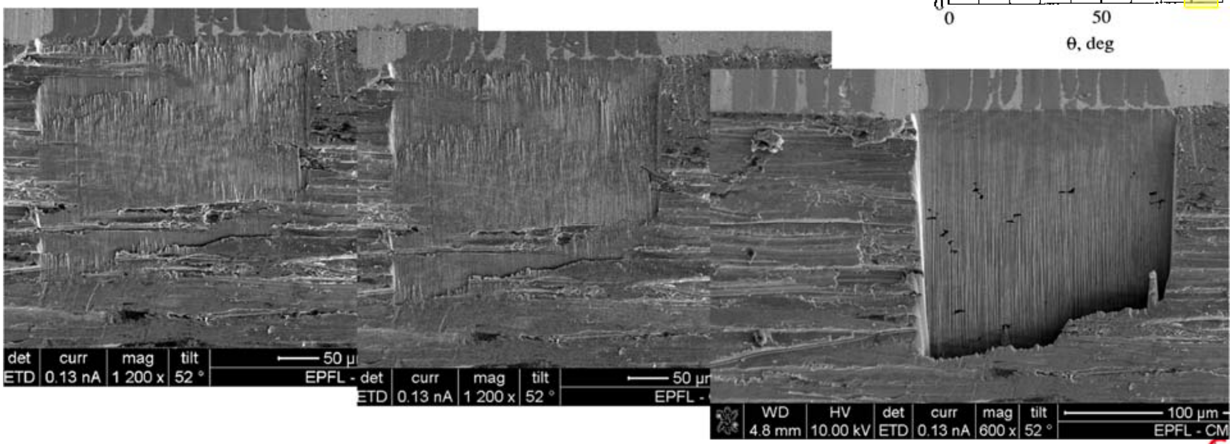




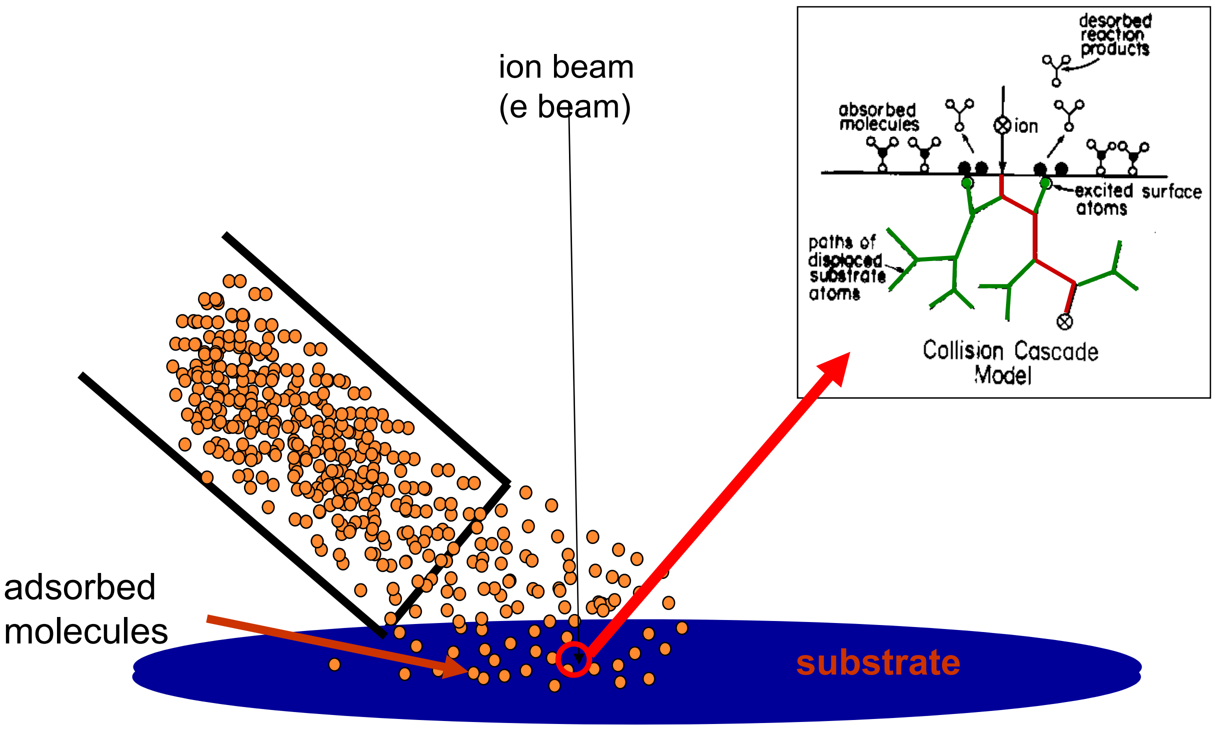
Polishing, at shallow angles



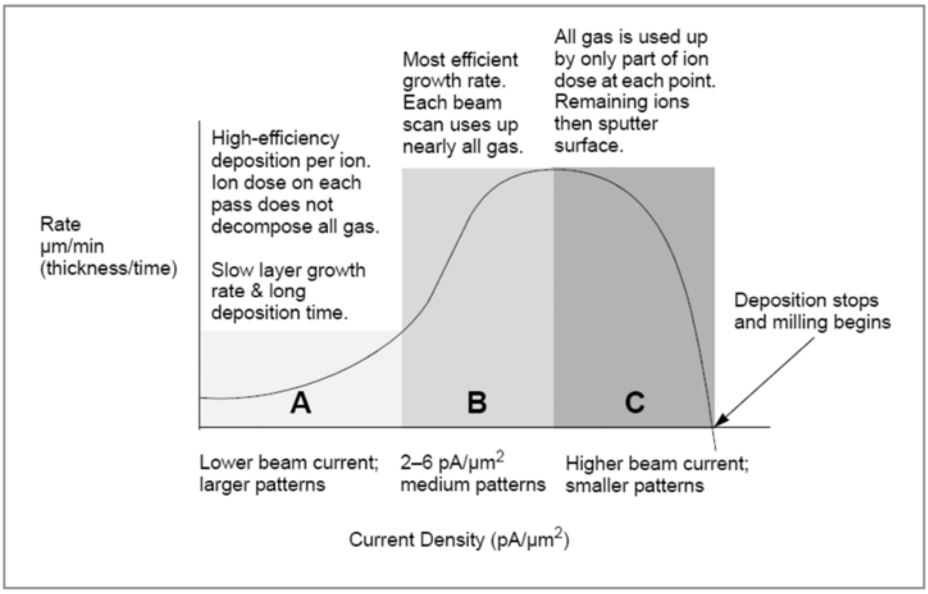




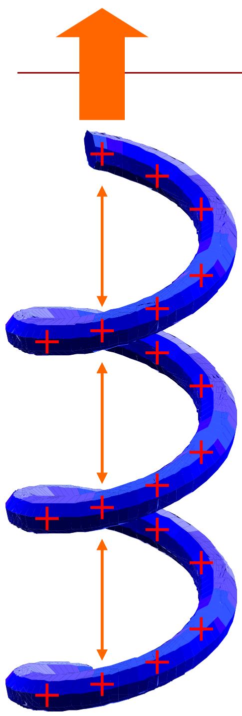
Gas assisted deposition

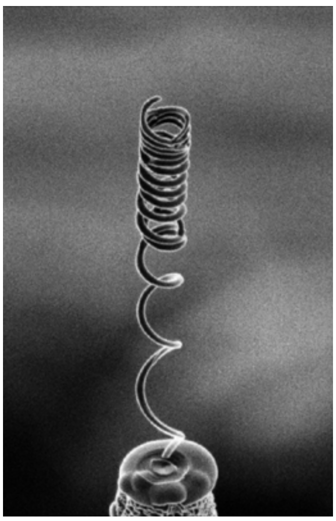


Deposition rate

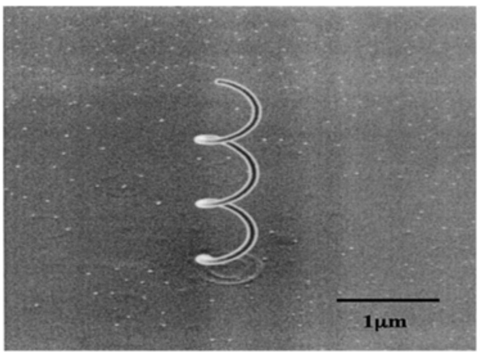


Nanofabricated structures

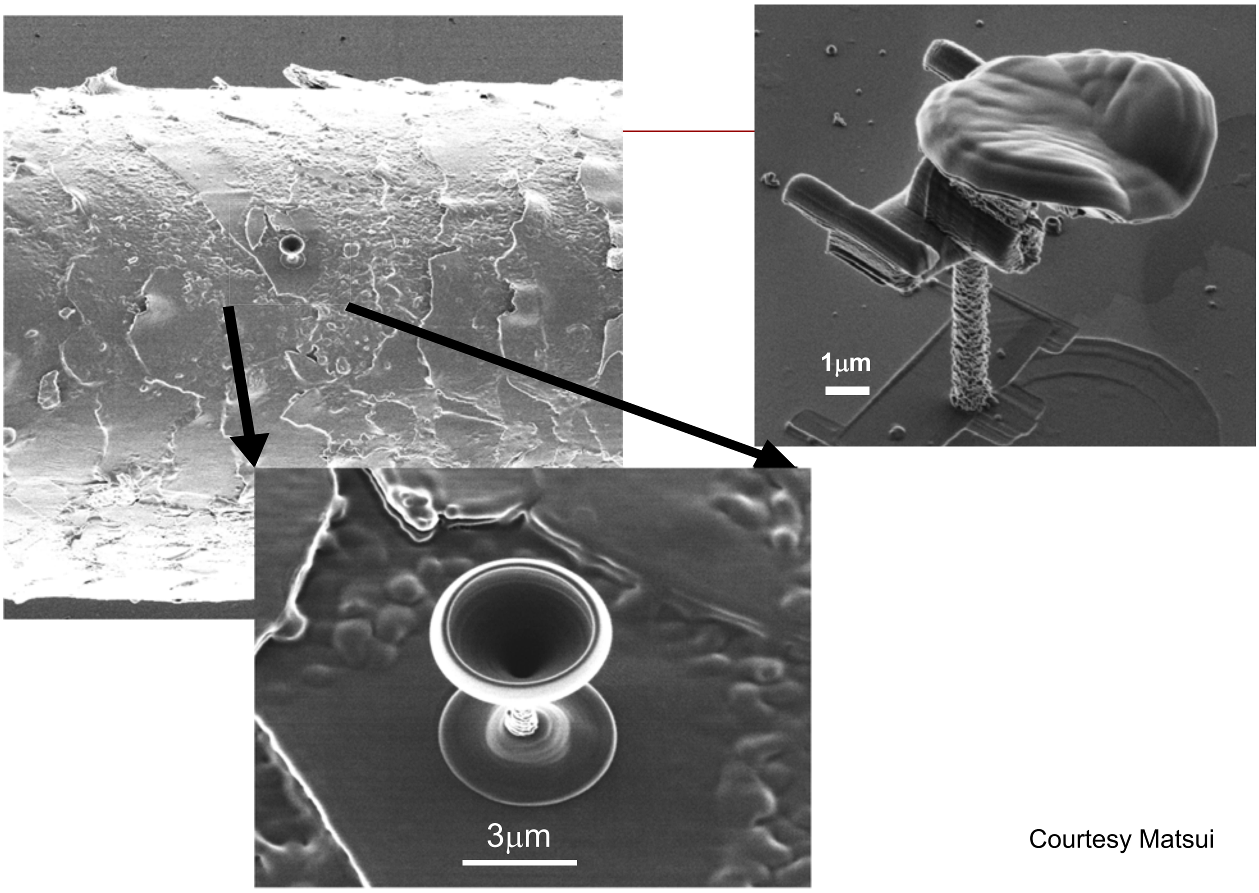




Coil 700nm pitch, 80nm line width, diamond-like amorphous carbon, FIB induced CVD



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



Courtesy Matsui

b) Basic Applications

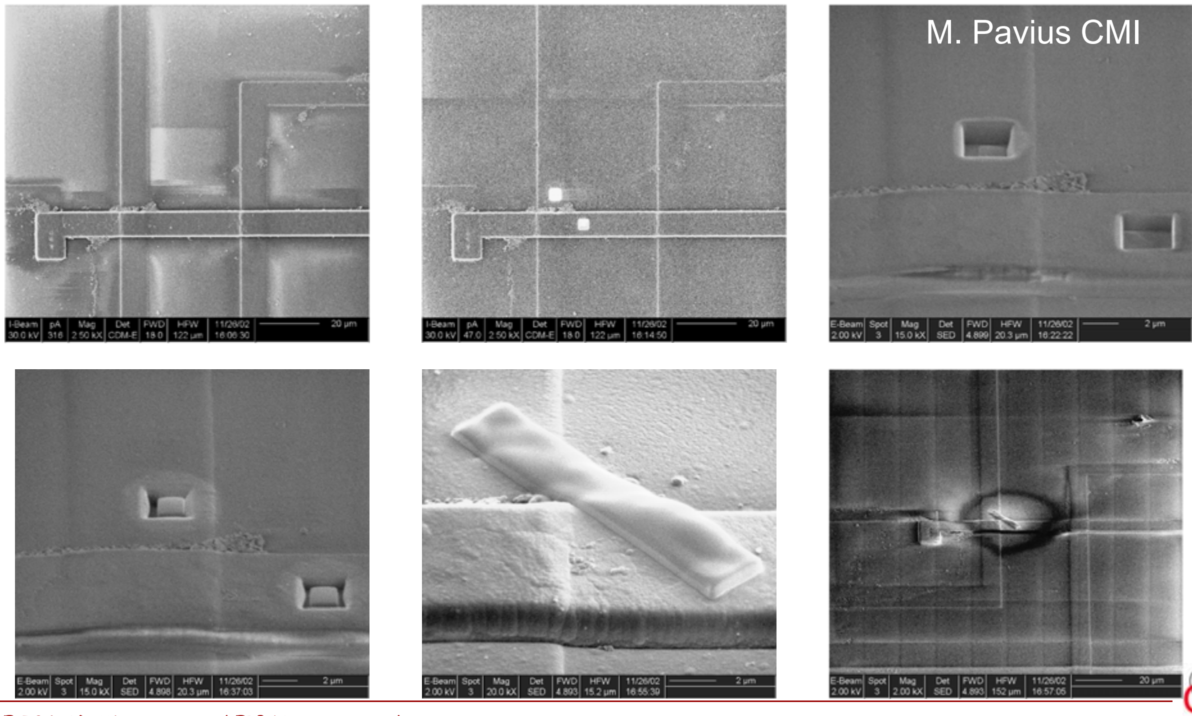
- "Industrial" applications (semiconductor industry)
- sectioning for failure analysis
- prototype circuit rewiring
- mask repair
- TEM sample preparation

- Research
- Micromachining
- Nanofabricated structures
- TEM sample preparation

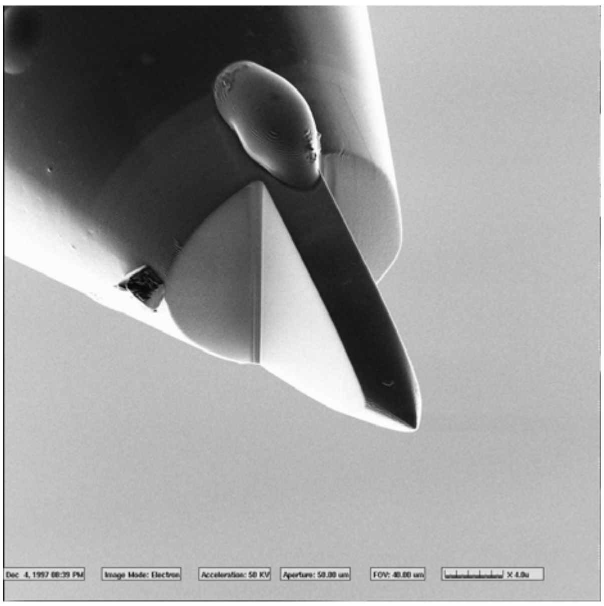
Applications   Chip Modification

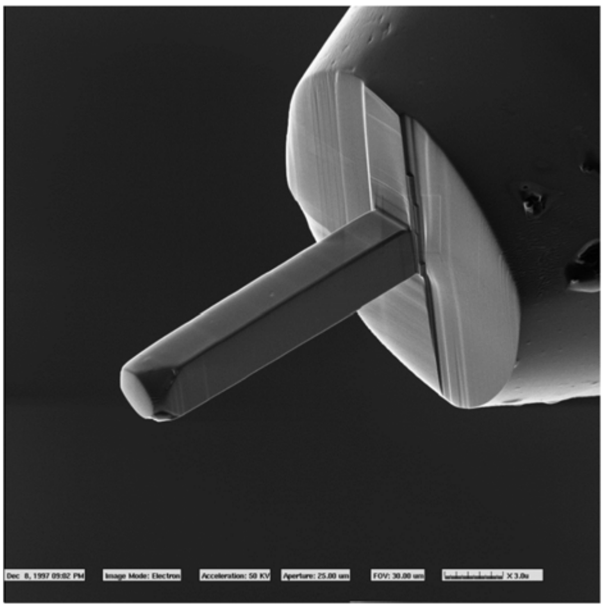
Insertion of electrical connection: 1) Removal of isolating layer (milling)

2) Pt deposition (FIB deposition)



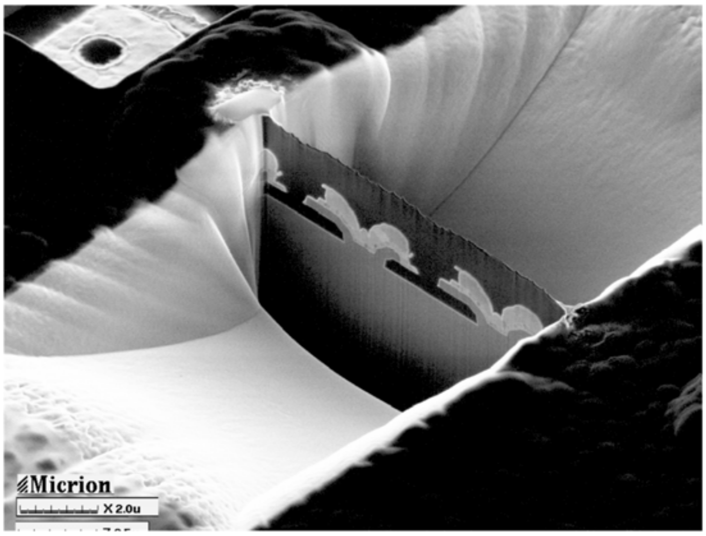
FIB-manufactured AFM-tips



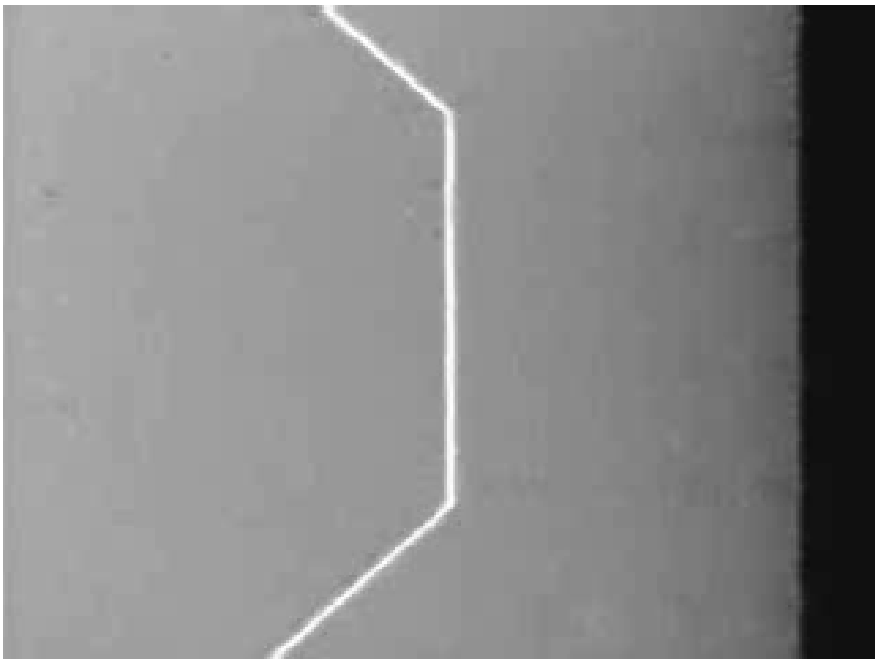


Failure analysis

FIB cross-sectioning and SEM imaging

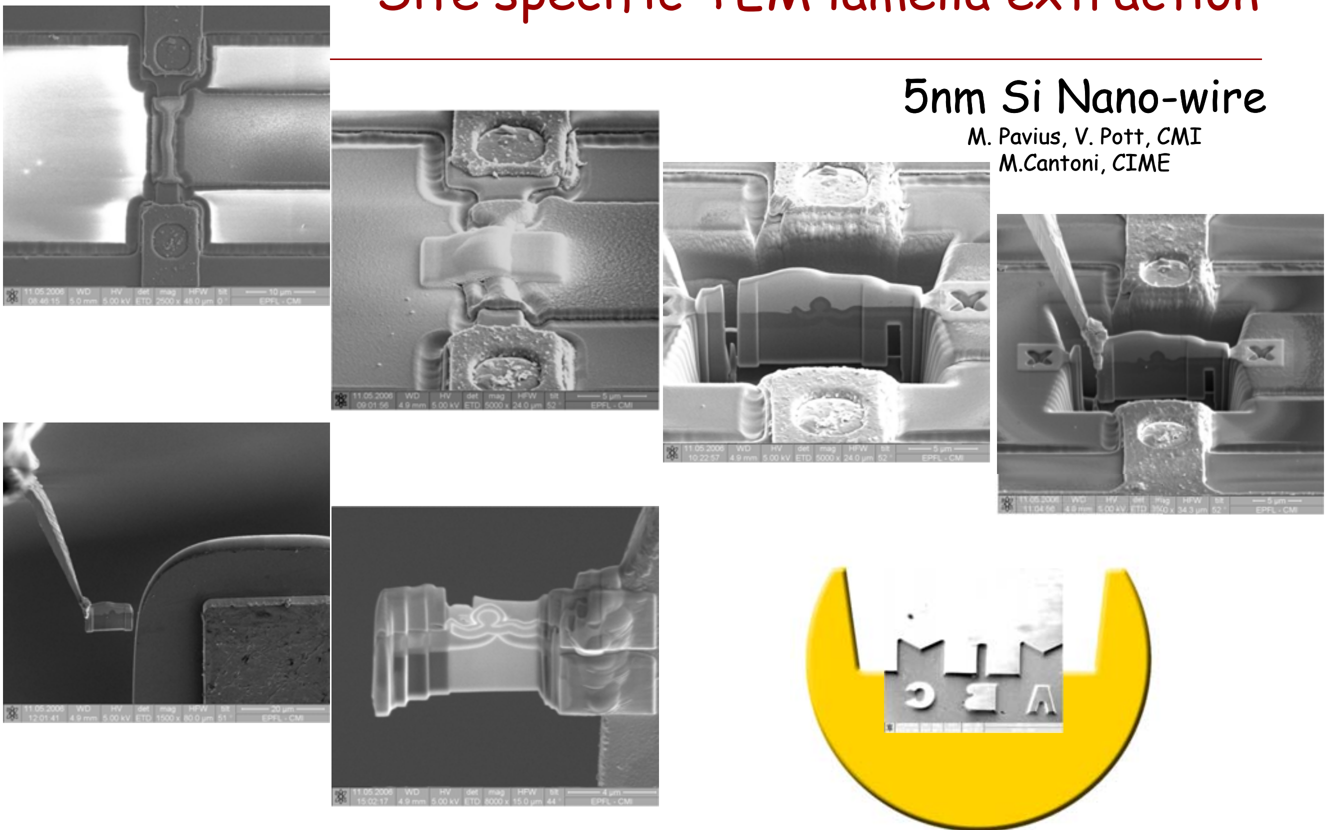


c) TEM preparation in-situ lift-out movie



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

Site specific TEM lamella extraction

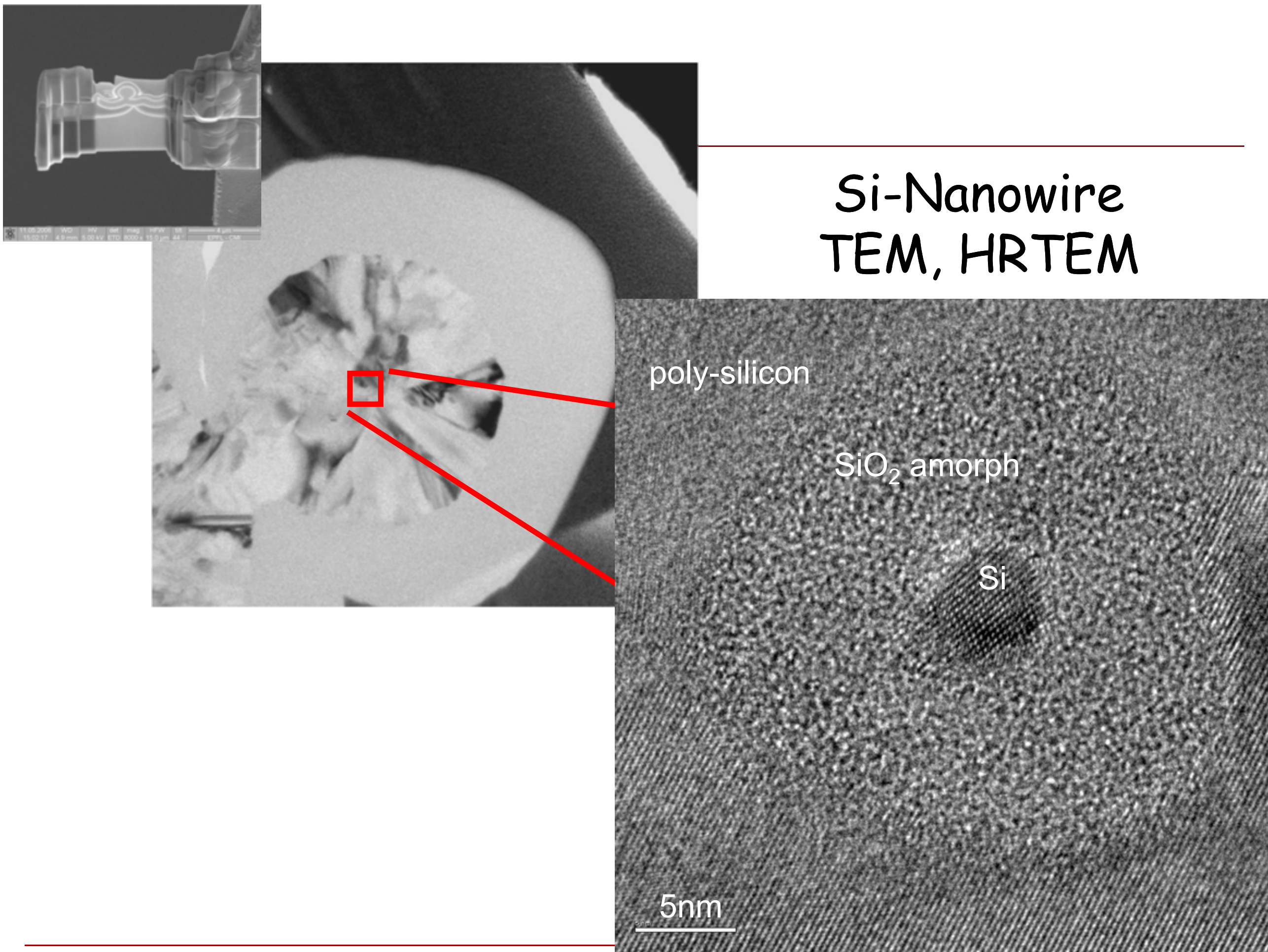


5nm Si Nano-wire

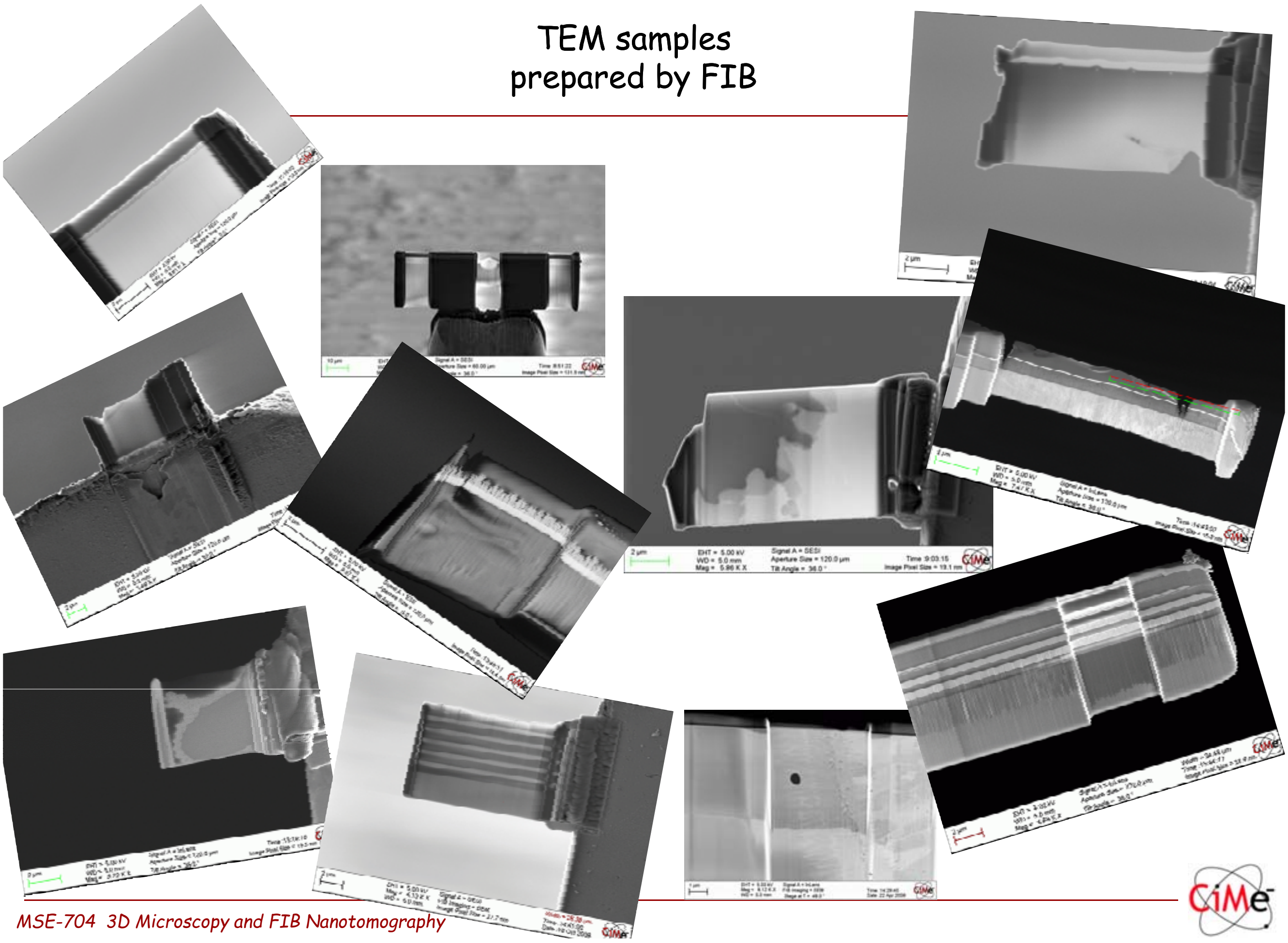
M. Pavius, V. Pott, CMI
M.Cantoni, CIME

TEM sample "grid" , diameter 3mm

Si-Nanowire TEM, HRTEM



TEM samples prepared by FIB



TEM lamellae by FIB

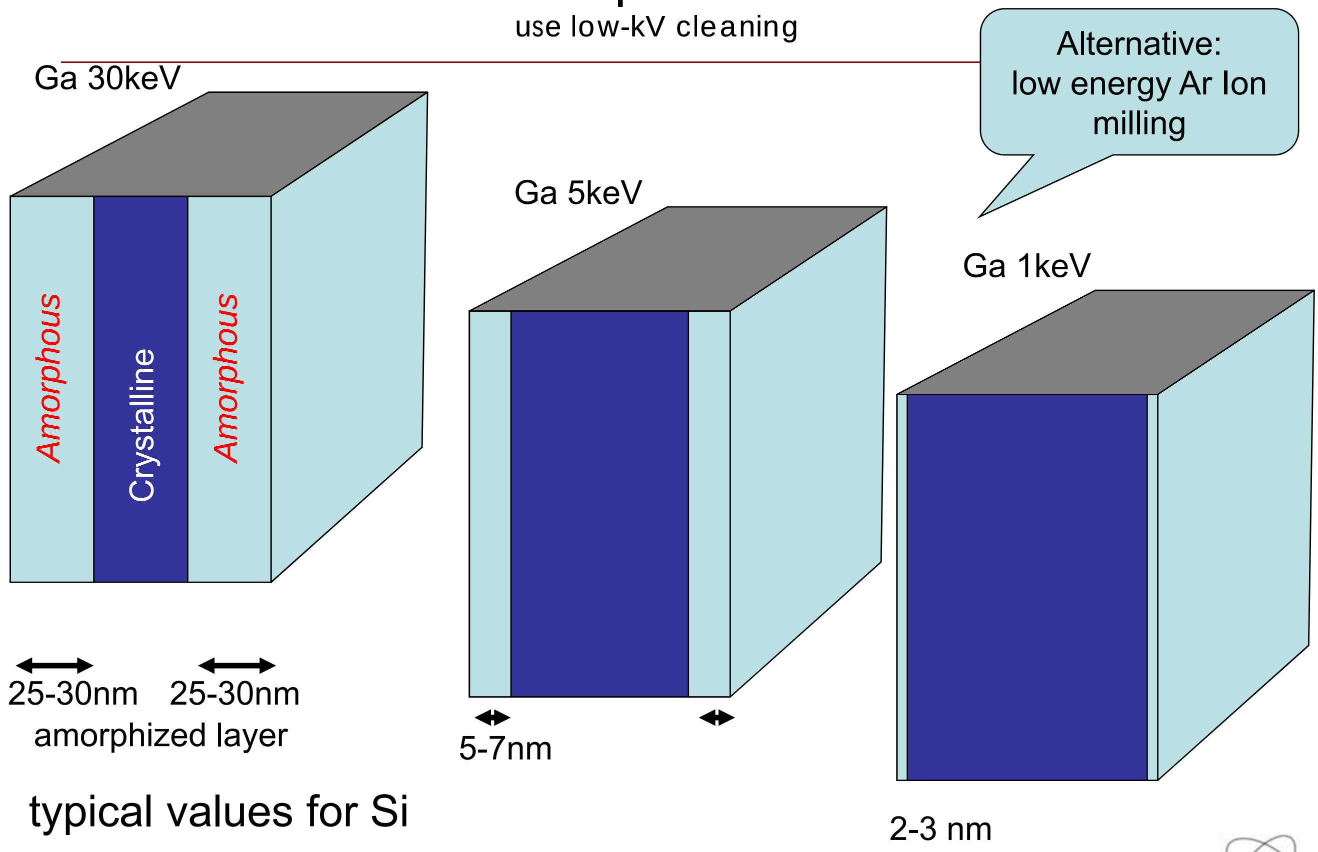
Focused Ion Beam adds a new dimension to TEM specimen preparation

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)

Amorphization

use low-kV cleaning



typical values for Si

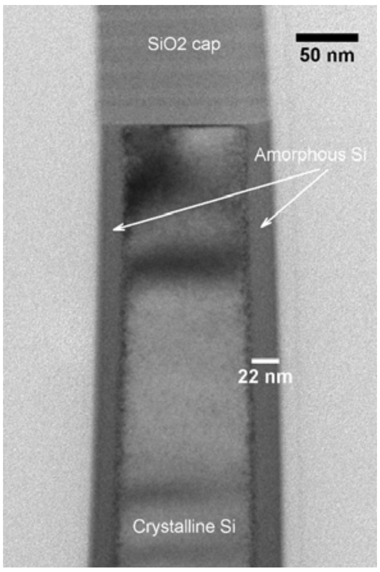
All-in-one FIB (Ga) +Ar ion milling

- TEM sample preparation by high-energy Ga FIB causes
  - Ga implantation
  - Amorphization, structural alteration

  up to 25nm underneath the surface.

- Low-energy noble gas ion polishing
  - Reduces damage layer thickness
  - Can thin the specimen further with little damage

- → Combine a low-energy noble gas ion column with a FIB/SEM system



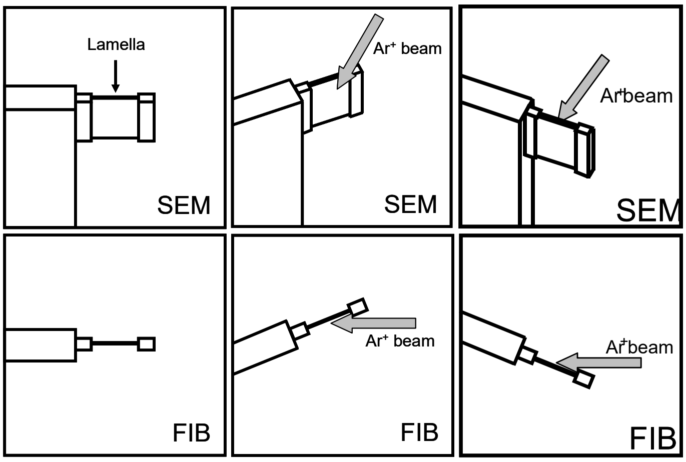
Cross-section of TEM lamella

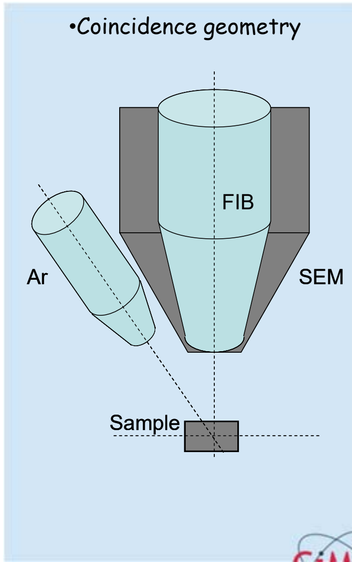
NVision 40 Argon

In-situ Low Energy Noble Gas Ion Milling in a Three Beam Instrument

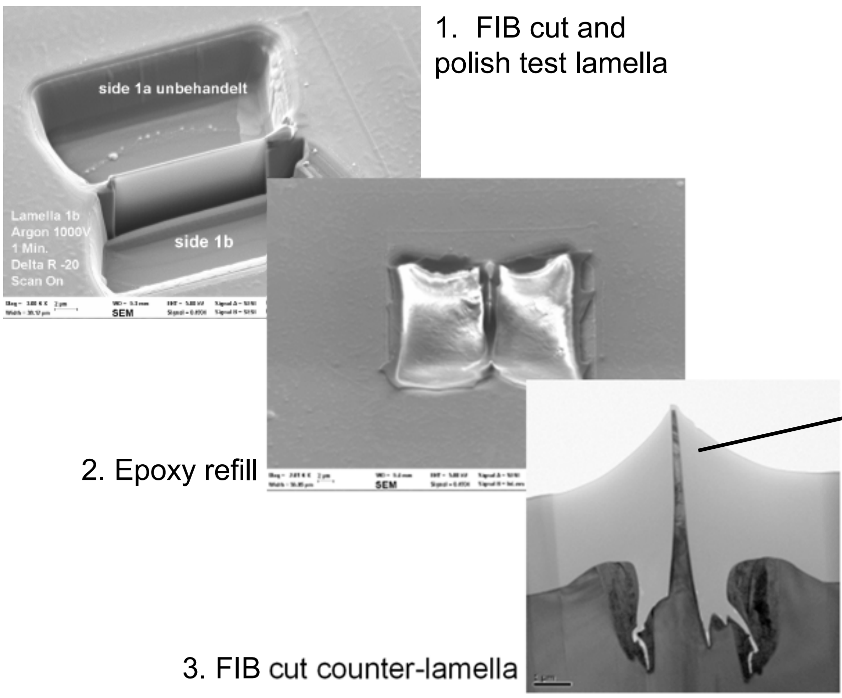
Heiko Stegmann
Carl Zeiss NTS GmbH, Oberkochen, Germany
René Hübner, Yvonne Ritz, Dirk Uteß, Beate Volkmann, Hans-Jürgen Engelmann
GLOBALFOUNDRIES Dresden LLC & Co. KG, Germany

TEM lamella after FIB | **Front side polishing** | **Back side polishing**





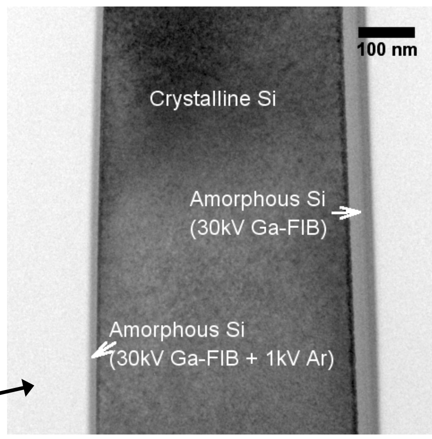
Amorphization layer thickness - Measurement method



1. FIB cut and polish test lamella

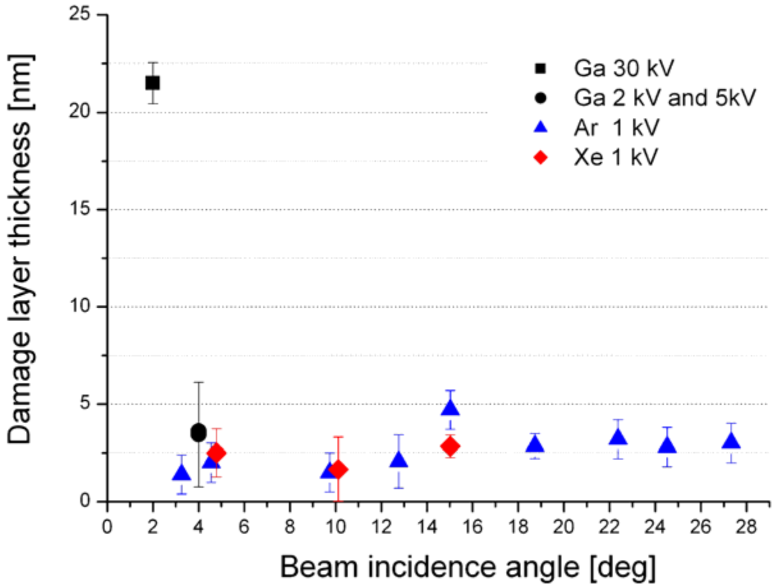
2. Epoxy refill

3. FIB cut counter-lamella



4. Measure damage layer thickness in TEM

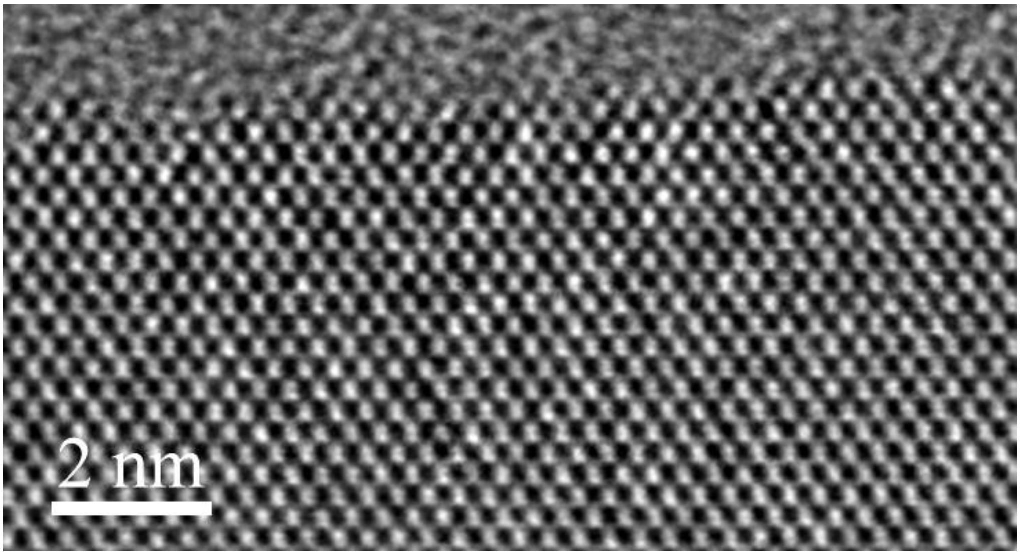
Amorphization layer thickness – Results



Cs-corrected HRTEM of Si/SiO_2 interface

SiO_2

Si cryst.

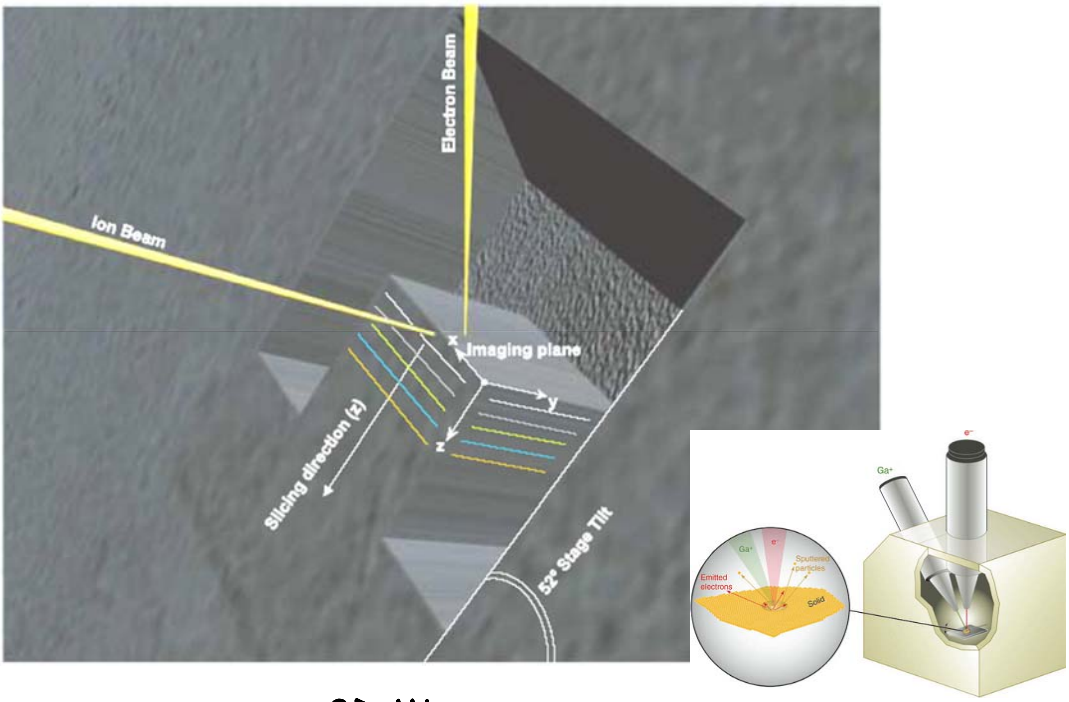


Liftout lamella 1kV Ar polished

•(Courtesy of R. Hübner/Globalfoundries Dresden LLC & Co. KG)

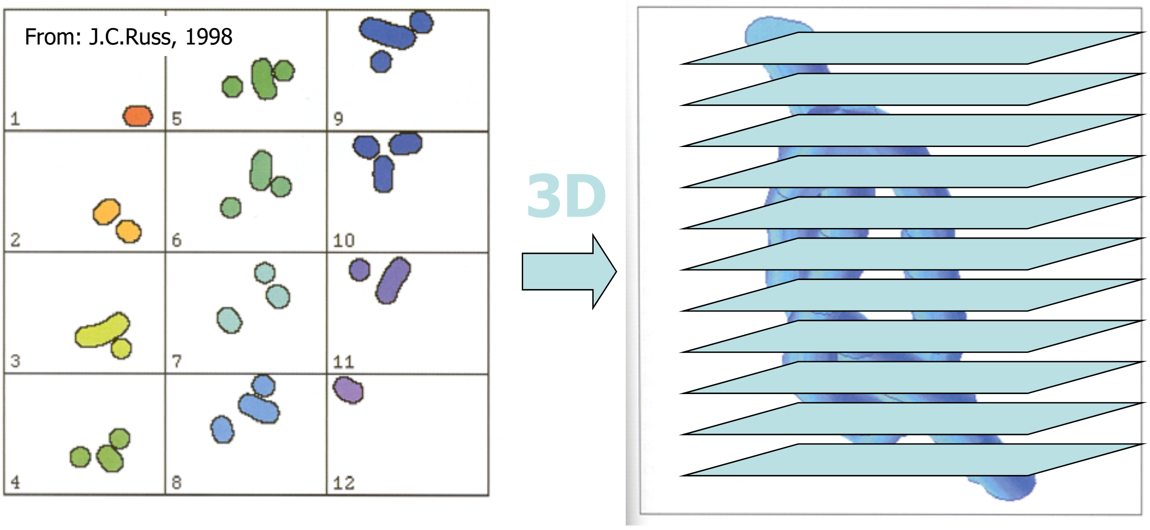
3D Microscopy

FIB Nanotomography



3D Microscopy

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

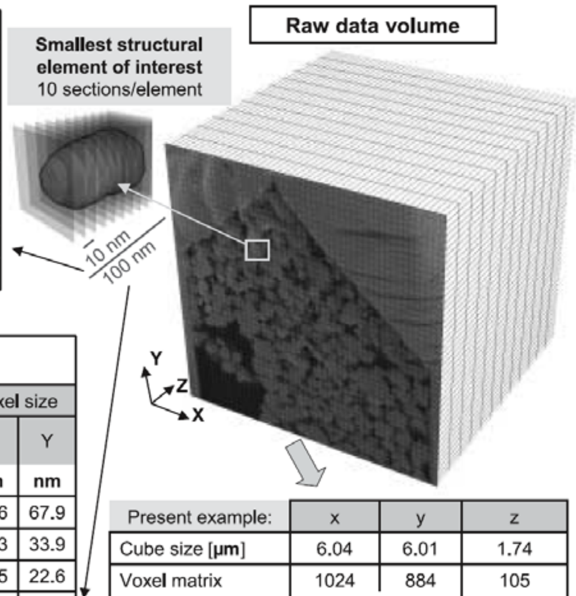


2D Volume fraction

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

Voxel, Resolution, Pixel size

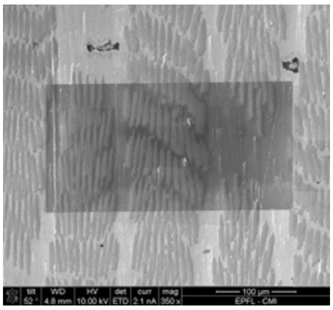
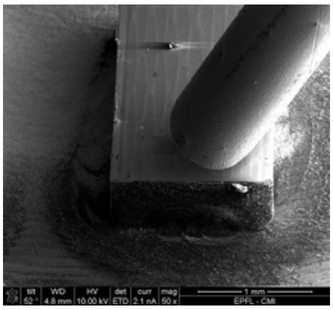
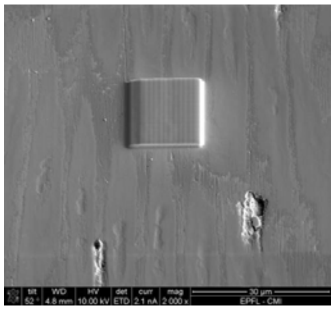
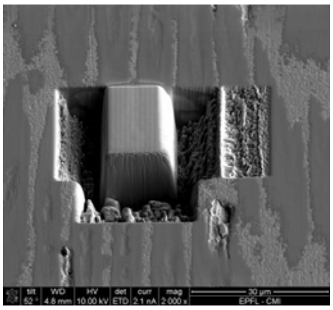
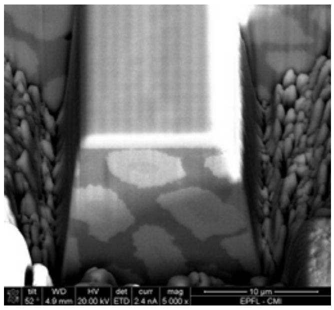
Ion-Beam			
Smallest element	Mag	Ideal z-spacing	Beam current
nm	kX	nm	pA
1000	5	100	500
500	10	50	300
300	15	30	100
200	30	20	50
100	50	10	50

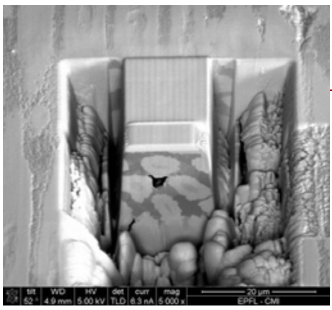


Electron-Beam					
Smallest element	Mag	Image width		Pixel size	
		1024 pxls, X	884 pxls, Y	X	Y
nm	kX	µm	µm	nm	nm
1000	5	60.0	65.73	58.6	67.9
500	10	30.0	32.86	29.3	33.9
300	15	20.0	21.91	19.5	22.6
200	30	10.0	10.95	9.8	11.3
100	50	6.0	5.18	5.9	6.8

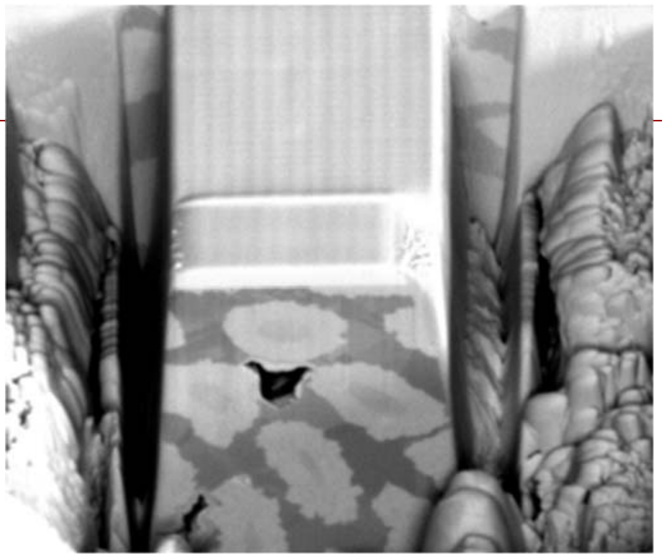
Present example:	x	y	z
Cube size [µm]	6.04	6.01	1.74
Voxel matrix	1024	884	105
	[pixels]		[sections]
Voxel size [nm]	5.9	6.8	16.6

3D slicing of multifilament Nb_3Sn superconductor

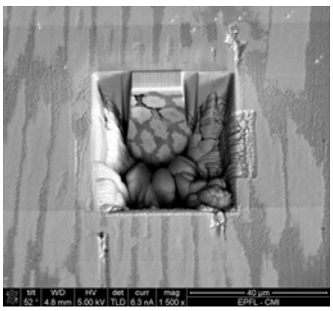
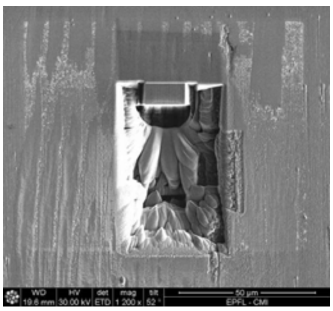


Preparing for slicing

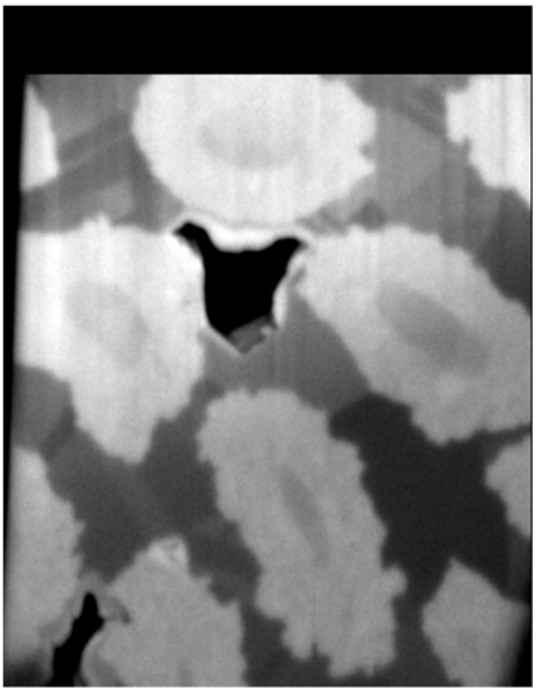


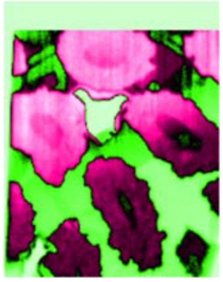
Automated milling and imaging of 170 slices (10h)

the end

align and crop

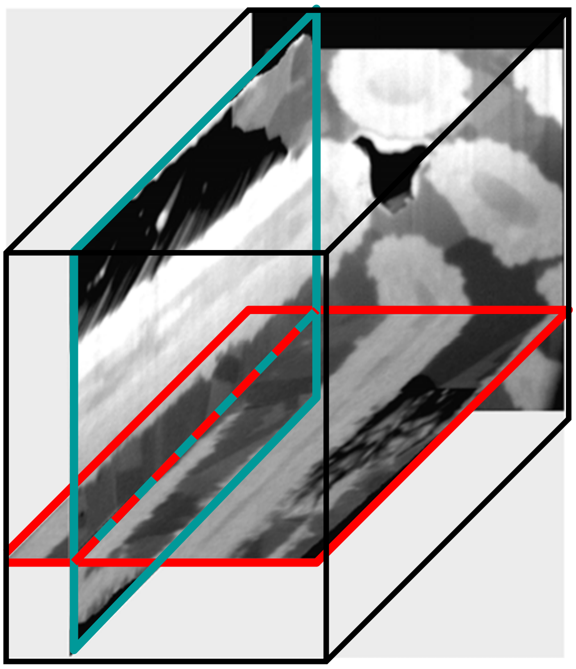


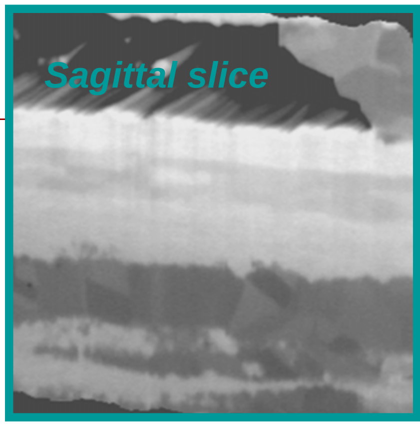


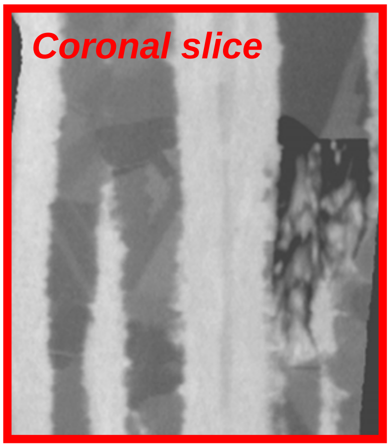
ImageJ
Image Processing and Analysis in Java

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

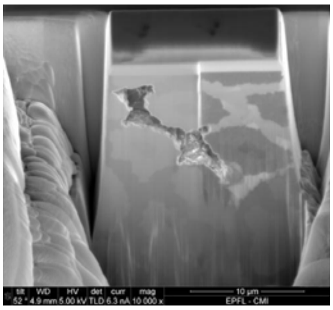
3D volume rendering, reconstruction: Orthogonal slices



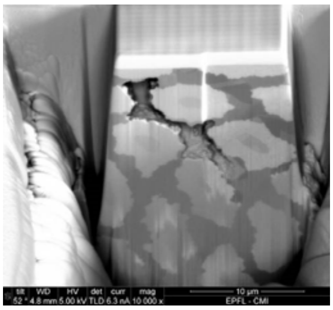




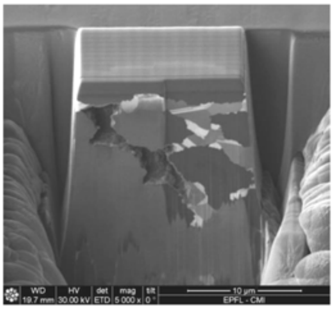
The choice of the right detector



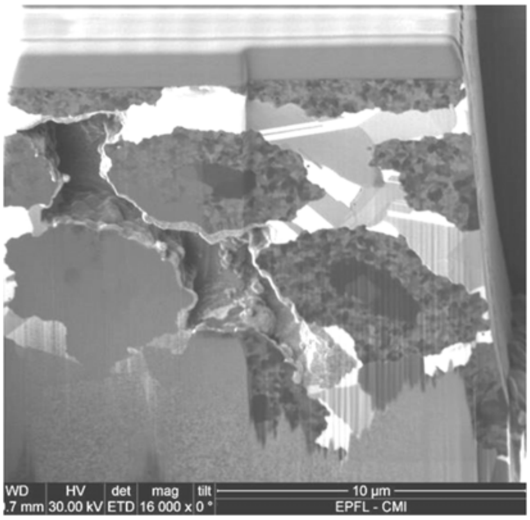
SE detector (TLD)



BSE detector (TLD)



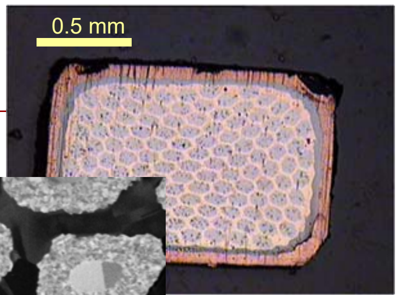
Ion beam imaging (SE)

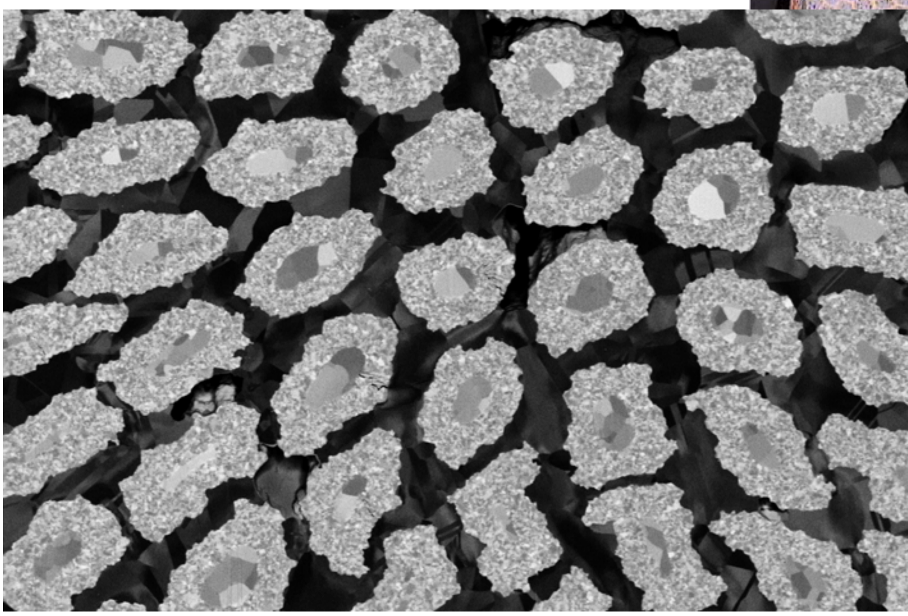


Ion beam for slicing and imaging requires stage movement…!

Nb3Sn multifilament Superconductors

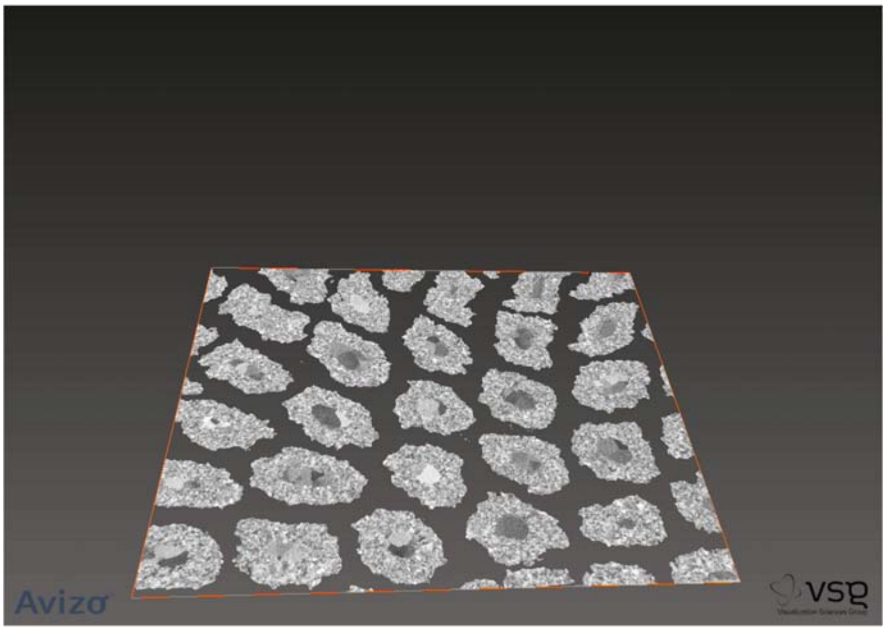
Materials & grain contrast





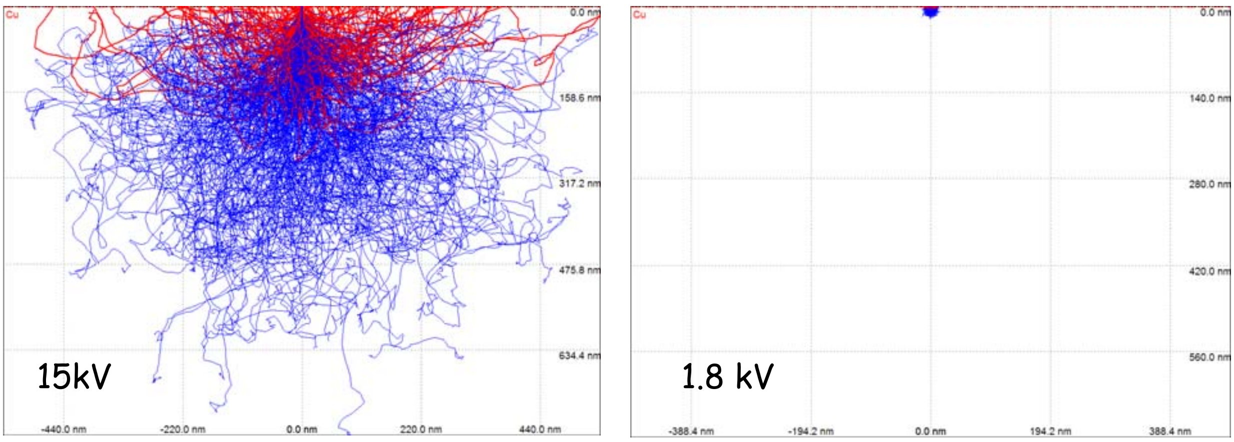
Nb_3Sn superconductor multifilament cable:
14'000 Nb_3Sn filaments (diameter ~5um) in bronze matrix
1.8kV EsB detector

Chemistry and orientation



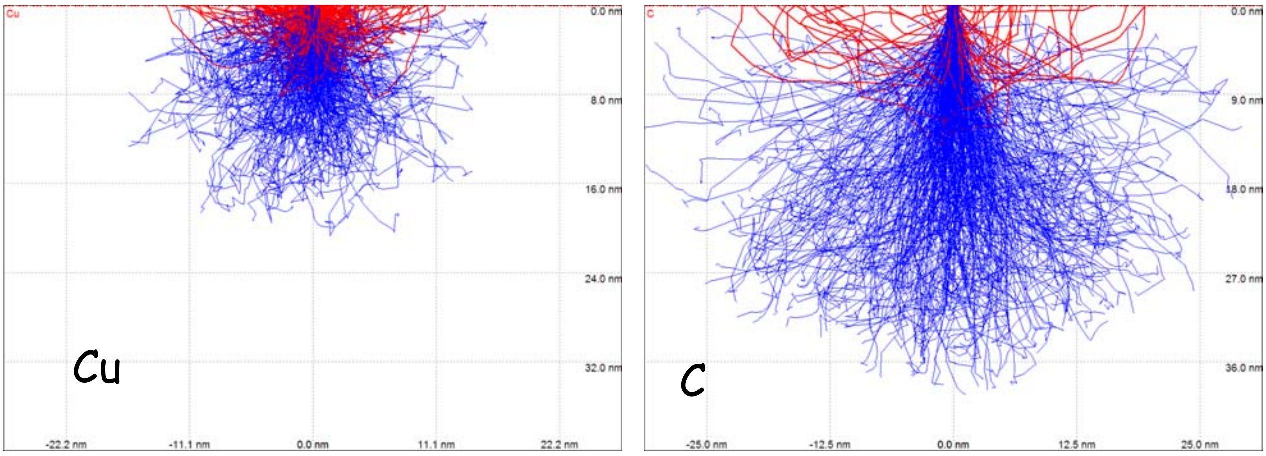
Nb_3Sn multifilament Superconductors
Materials & grain contrast
2048x1536x1700 (10x10x10nm voxel)

What is the spatial resolution in BSE imaging …?



Scatter range in Nb_3Sn:
Monte-Carlo Simulation of electron trajectories
backscattered electrons

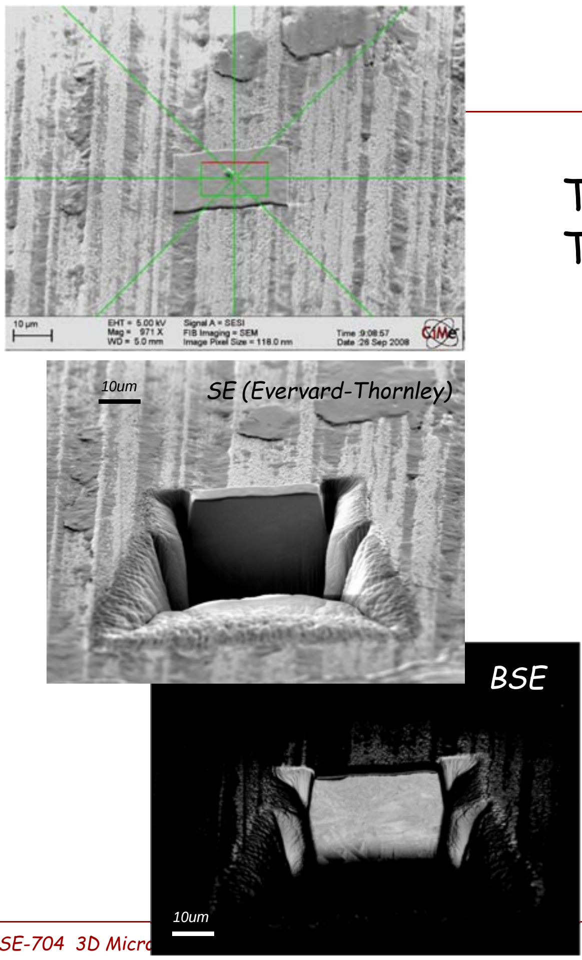
Interaction volume at 1.5kV, BSE escape depth



Monte-Carlo Simulation of electron trajectories in Cu and C
backscattered electrons

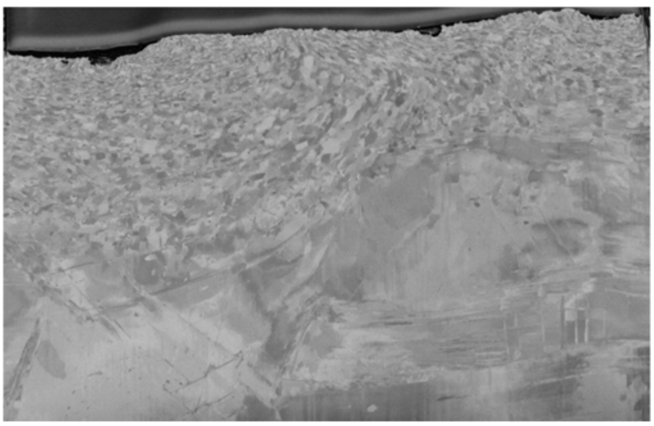
Orientation contrast

identification of grain texture

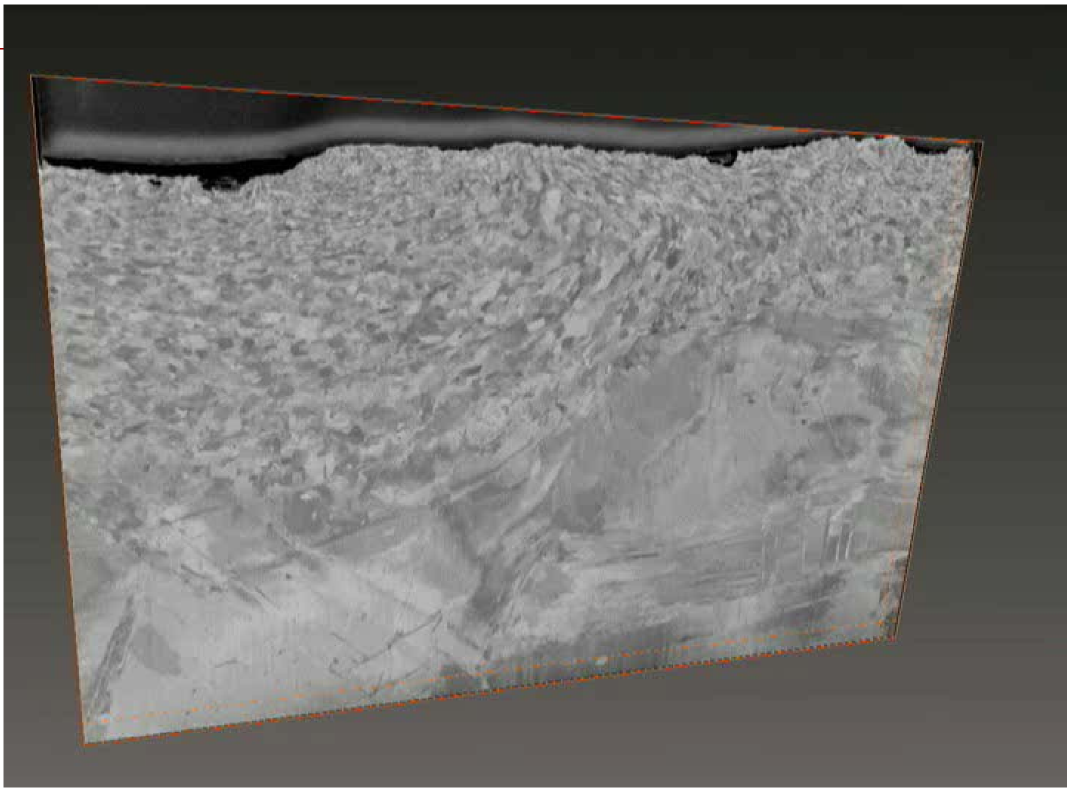


Tribology: wear trace on steel
Tribo-corrosion

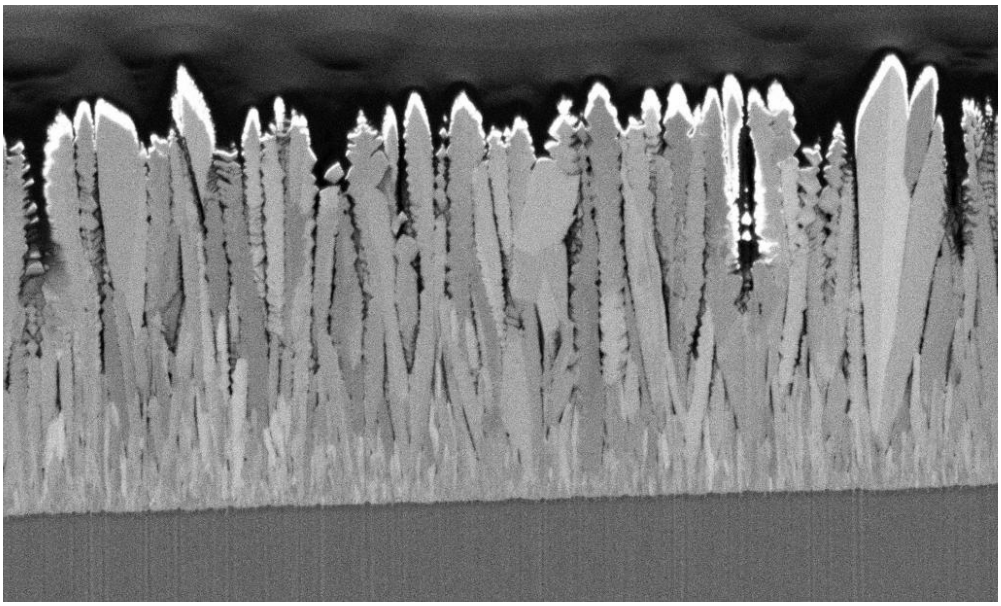
J. Perret, S.Mischler IMX-LMCH
Grain orientation contrast of small grains
(grain size < 100nm)



2048x1536x1200 volume: 20x15x12um
10x10x10nm voxel

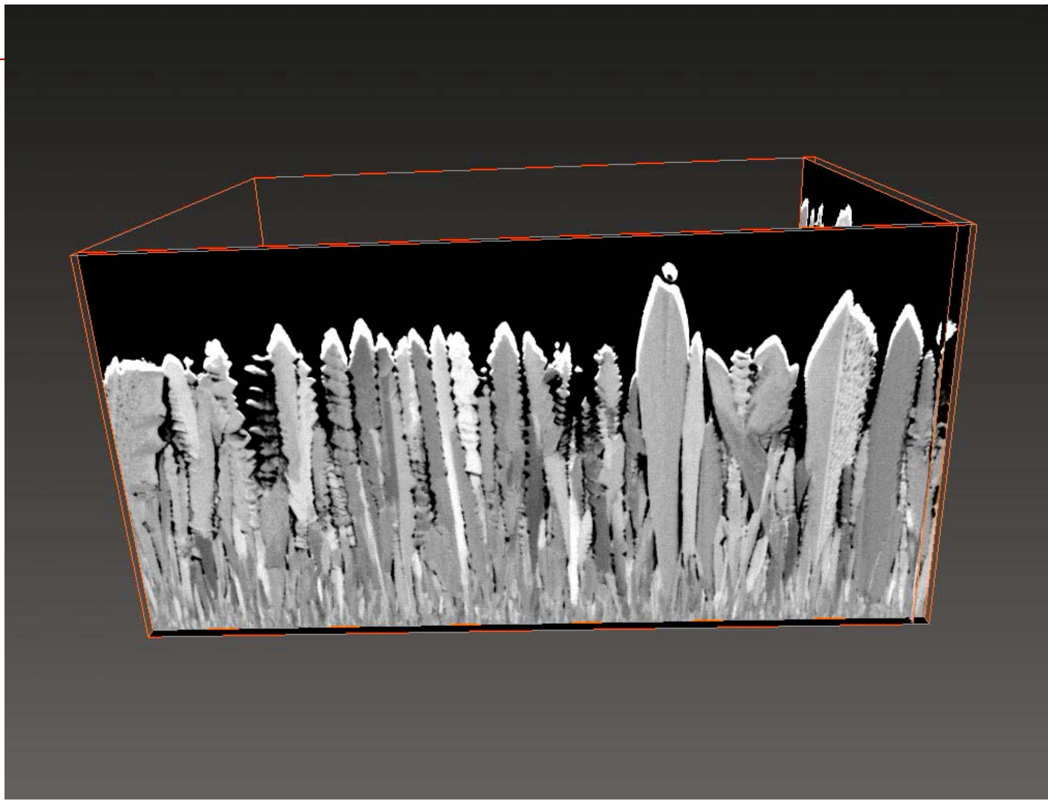


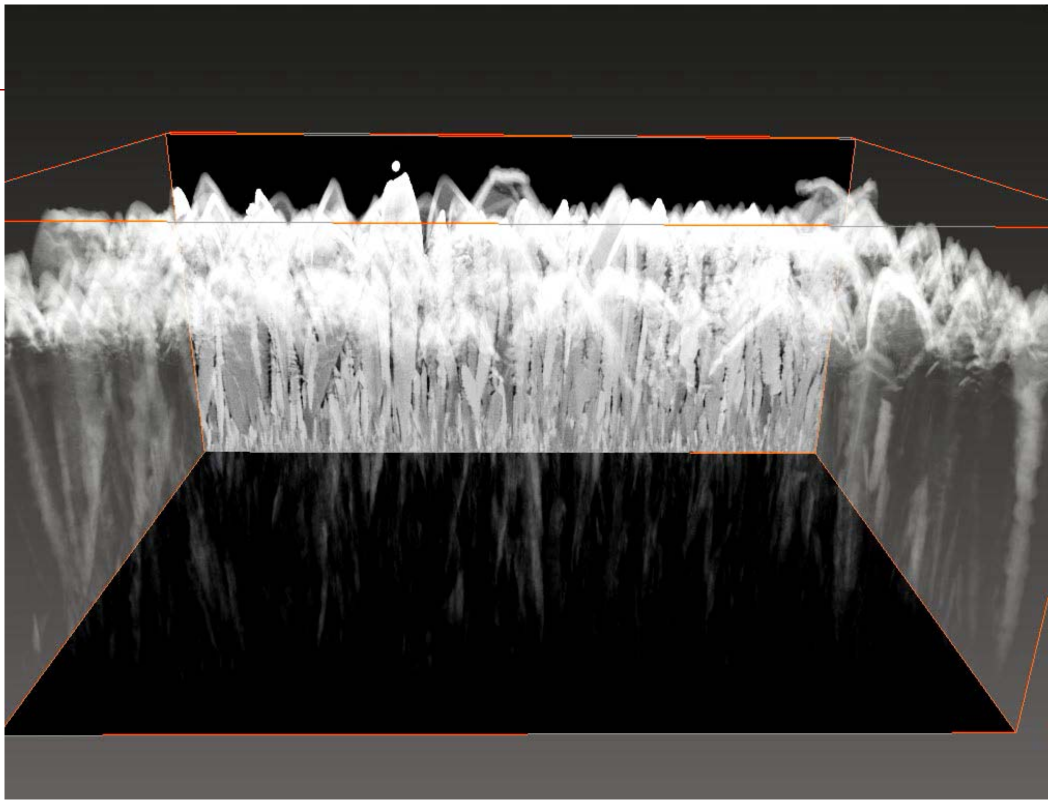
growth of ZnO films, photovoltaics

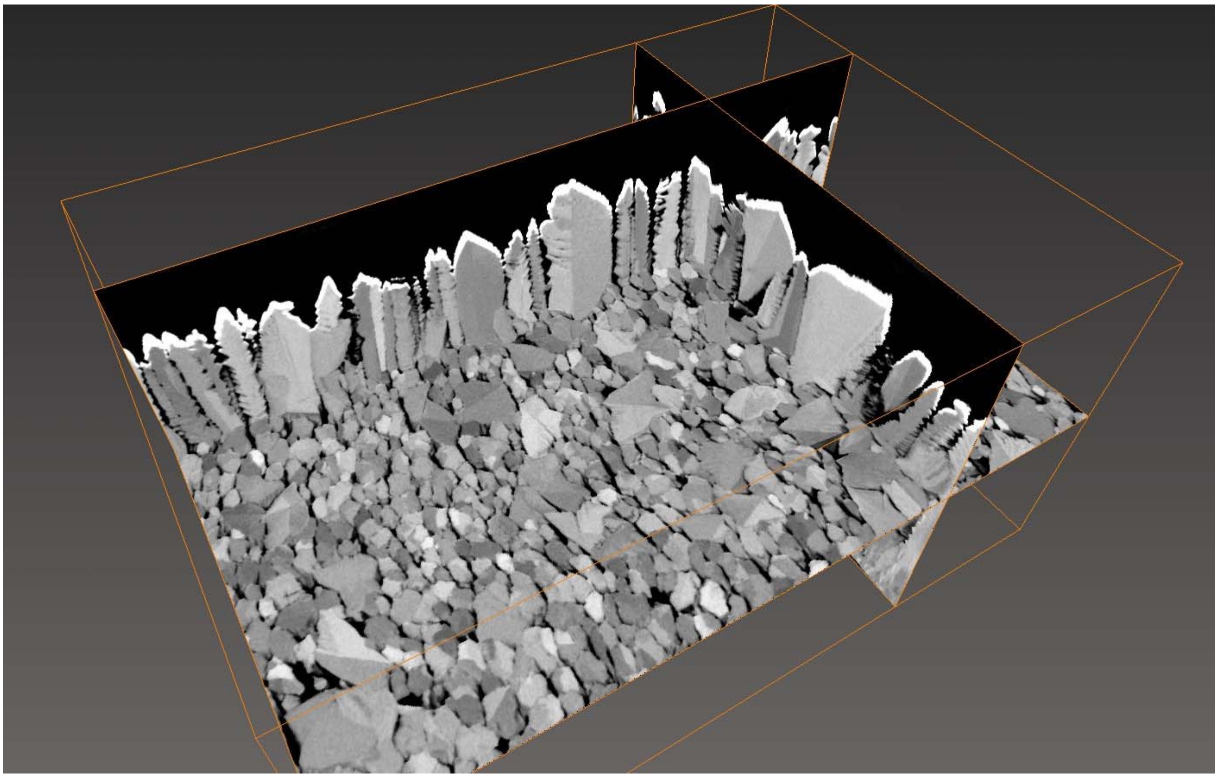


10x10x10nm voxel size, 2048x1536x2200 pixel/slices

C. Balif, S. Nicolay, D. Alexander



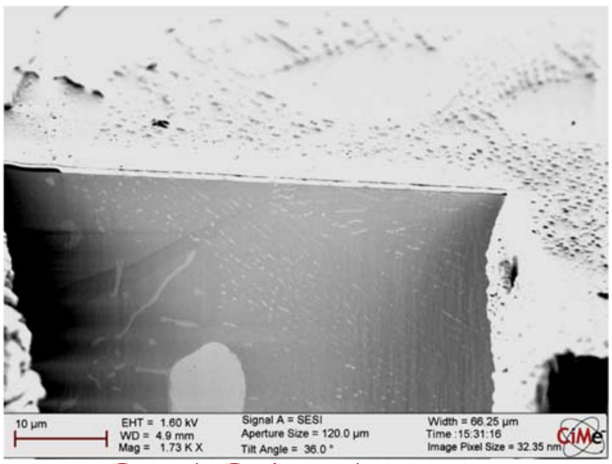




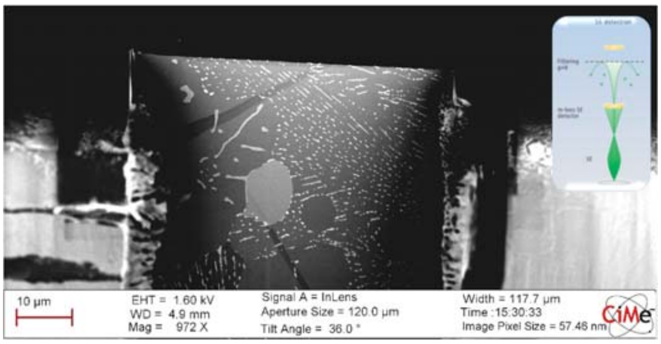
Pb-free solder:

"one detector is not enough"

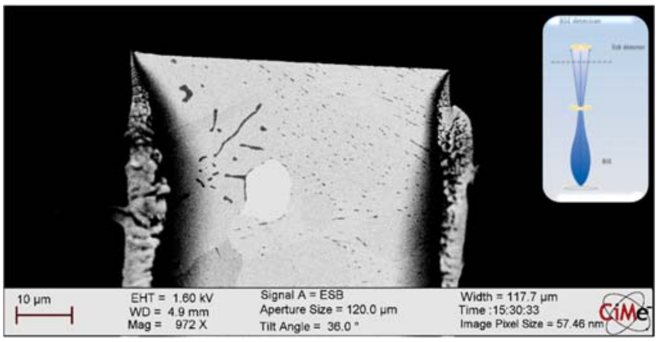
M. Maleki,
EPFL-LMAF



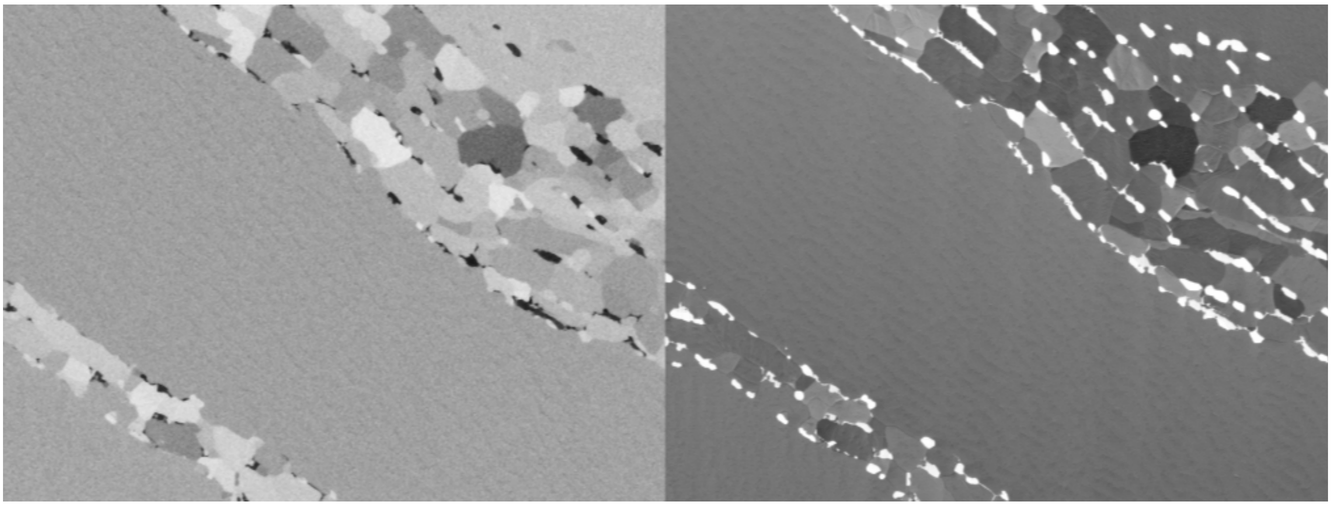
ETD (SE classic)



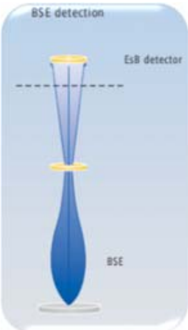
InLens: SE low energy



EsB: Energy selective Backscattered

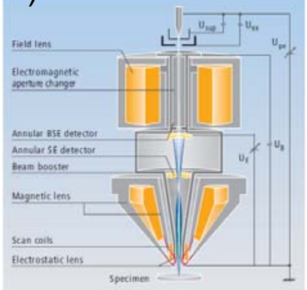


EsB

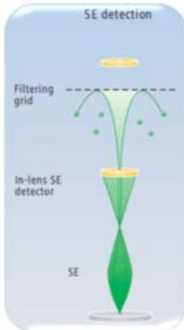


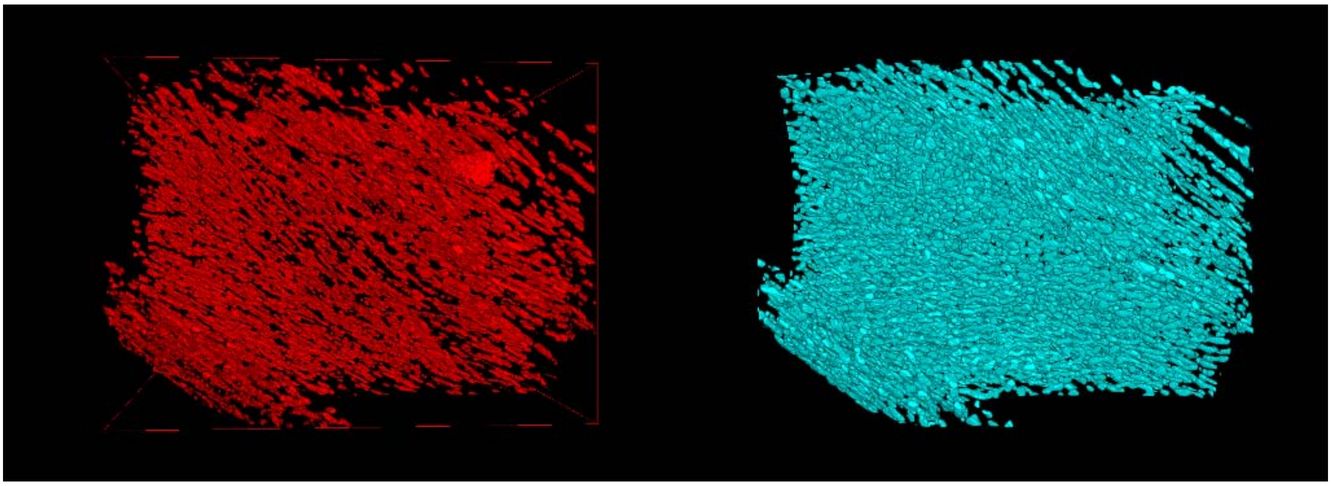
10x10x10nm voxel size, 2048x1536x2000 pixel/slices

2 images (3Mb) / slice …… 12Gb data



InLens SE

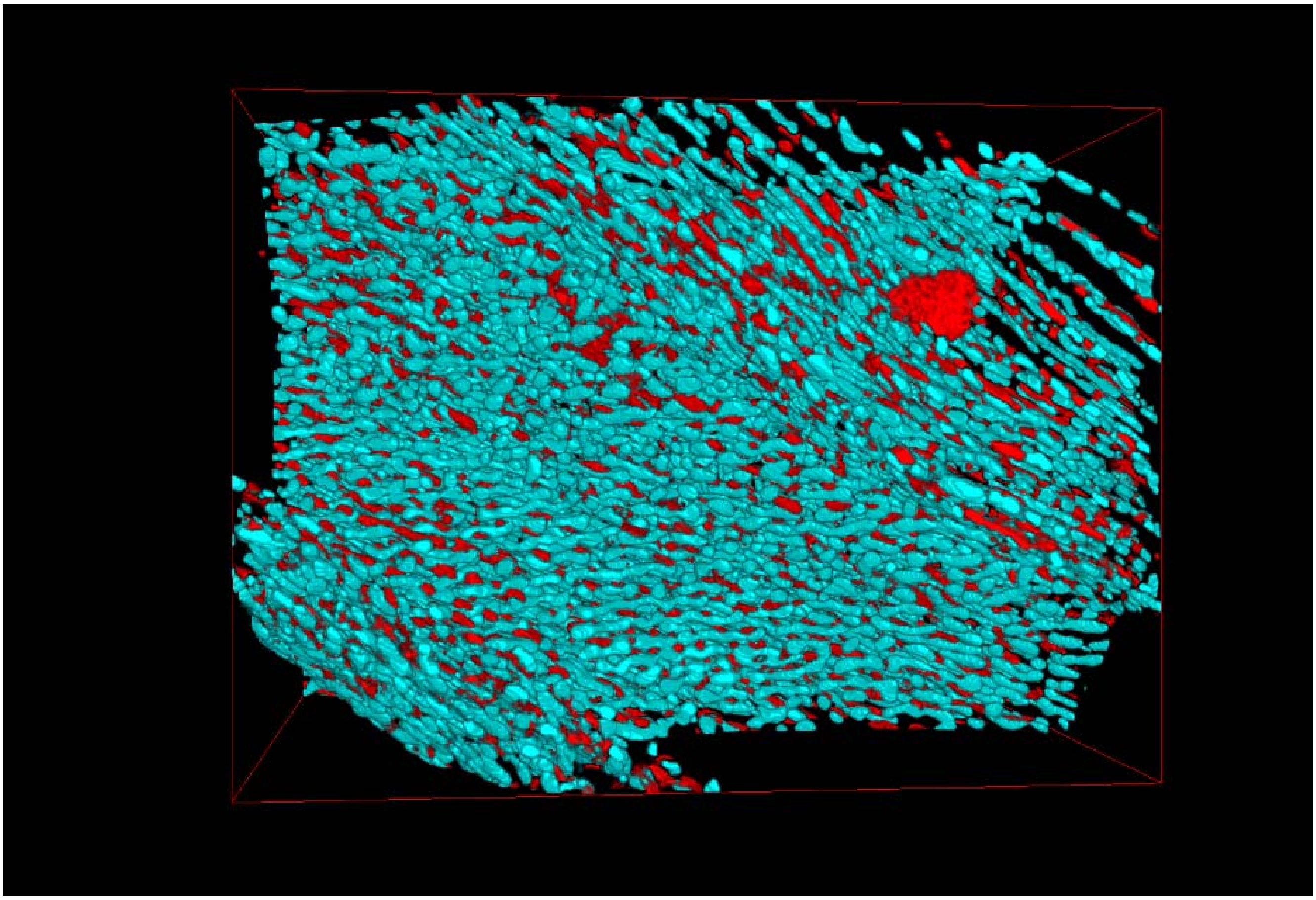


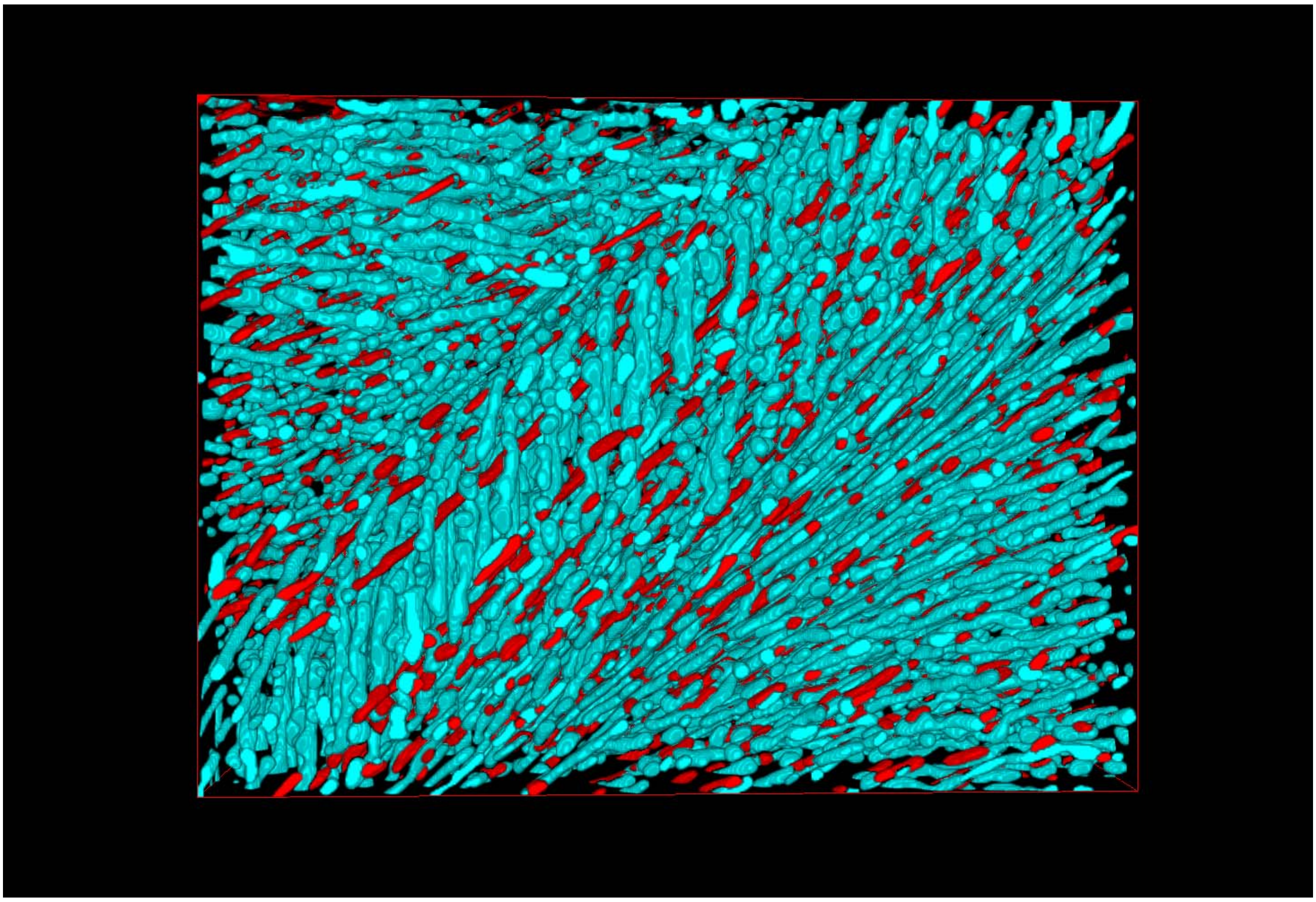


Phase 1
Dark in EsB image
White in SE-InLens

Phase 2
White in SE-InLens - Dark in EsB image

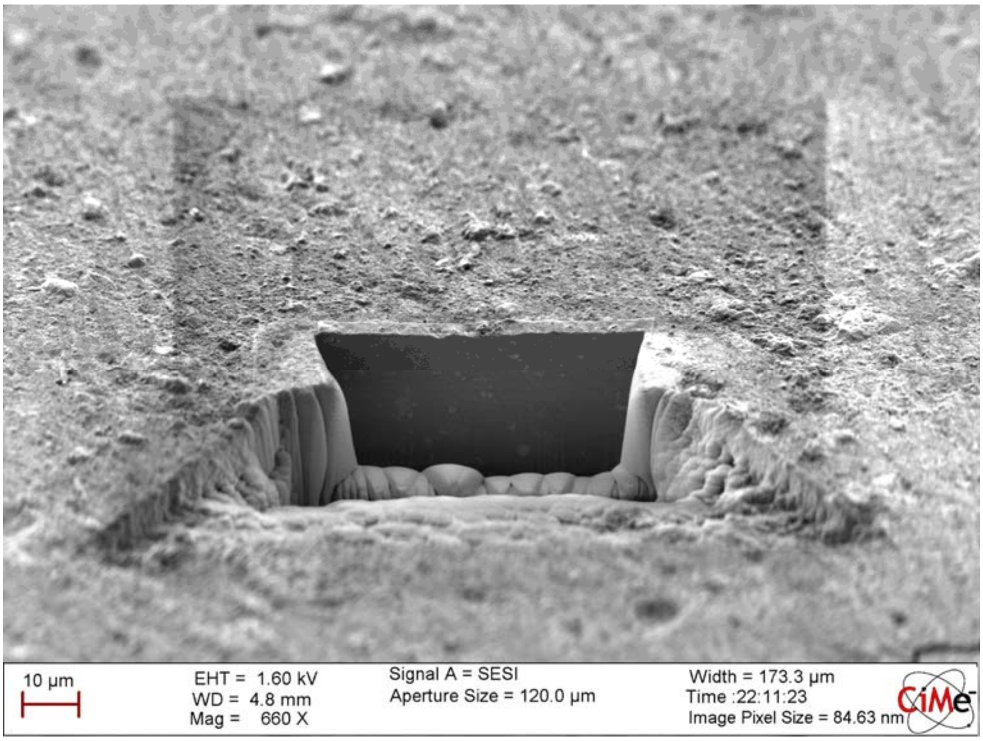
10x10x10nm voxel size, 2048x1536x2000 pixel/slices
2 images (3Mb) / slice …… 12Gb data

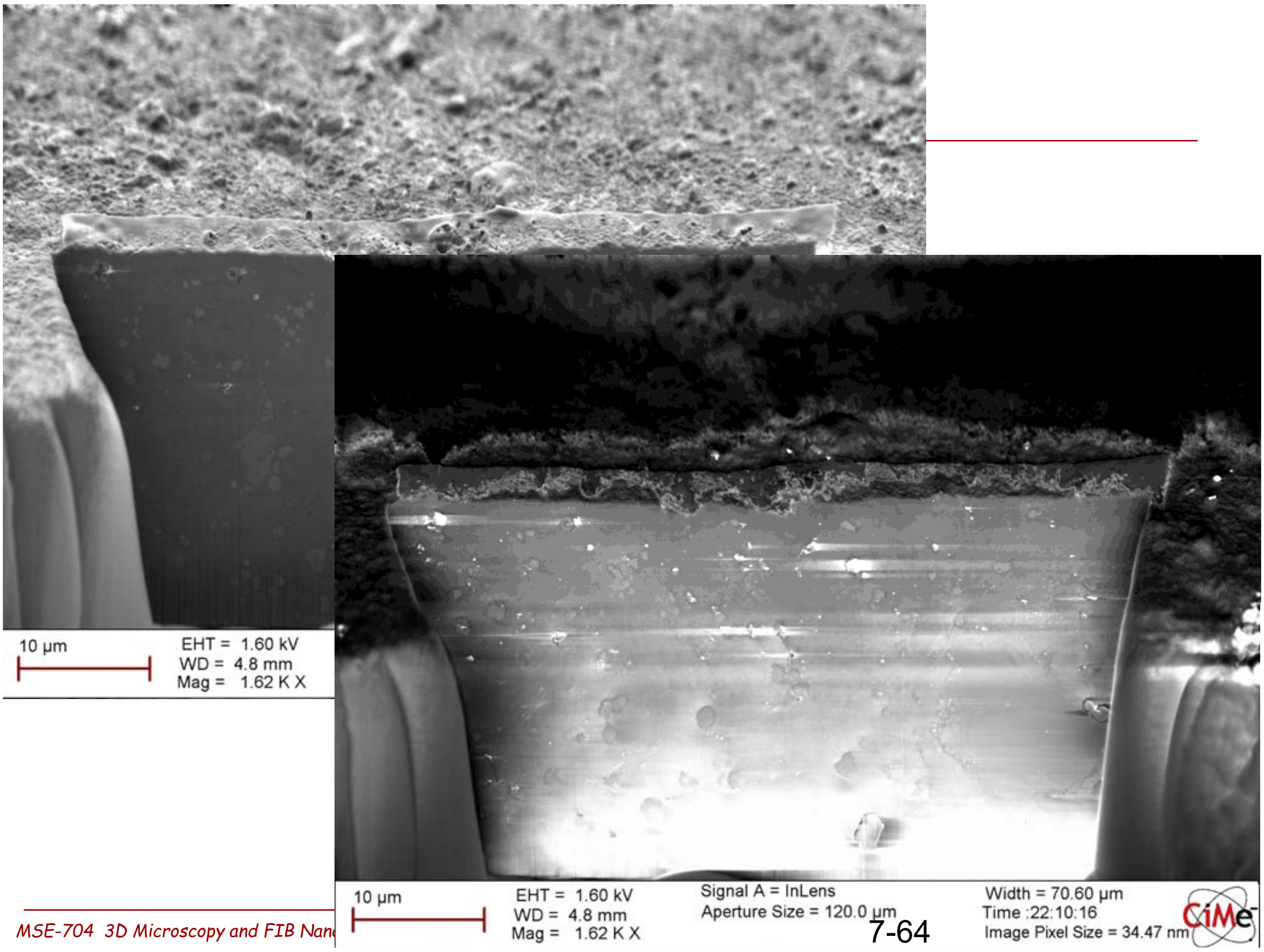


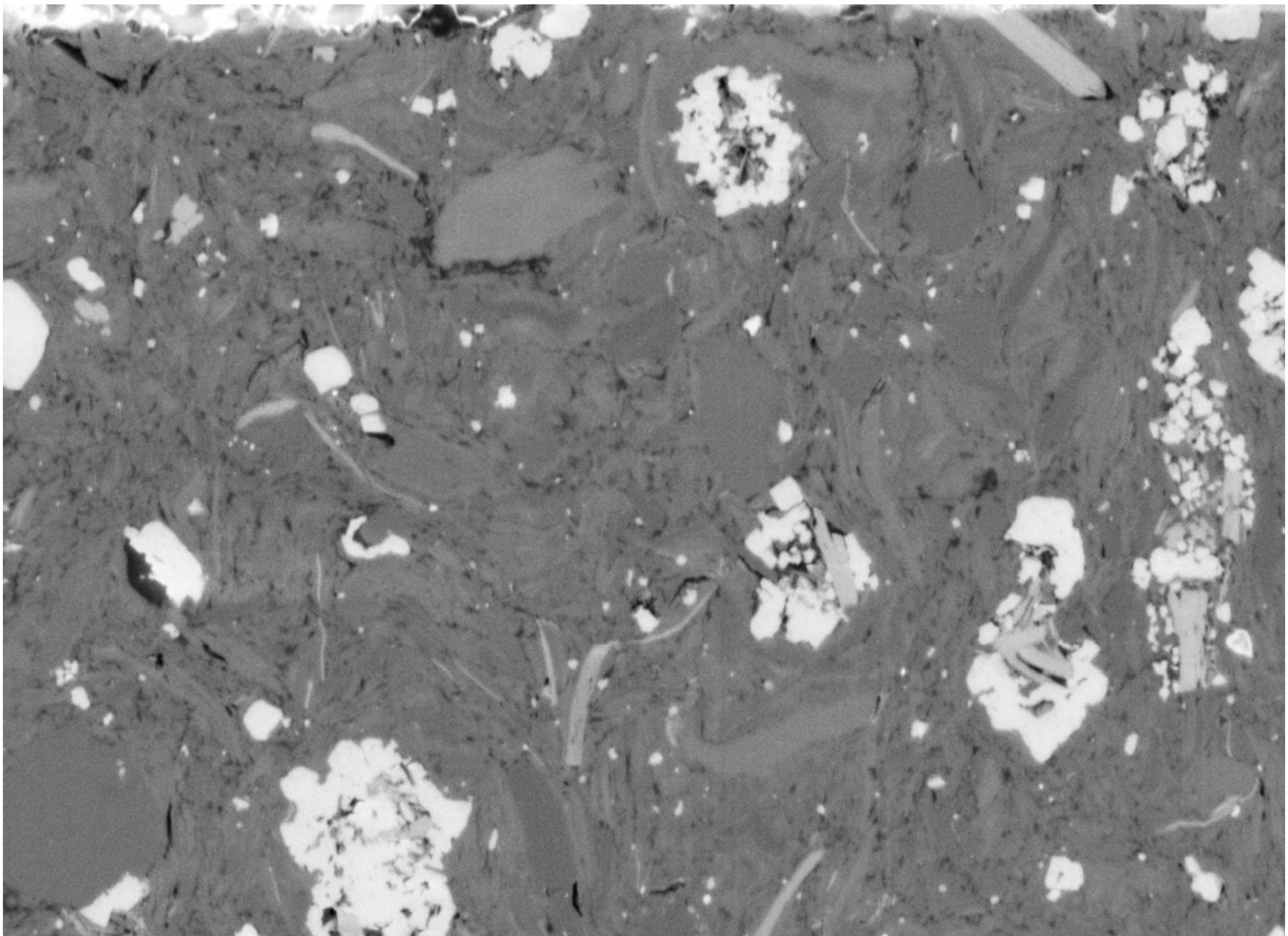


Geological samples: "rocks",clay

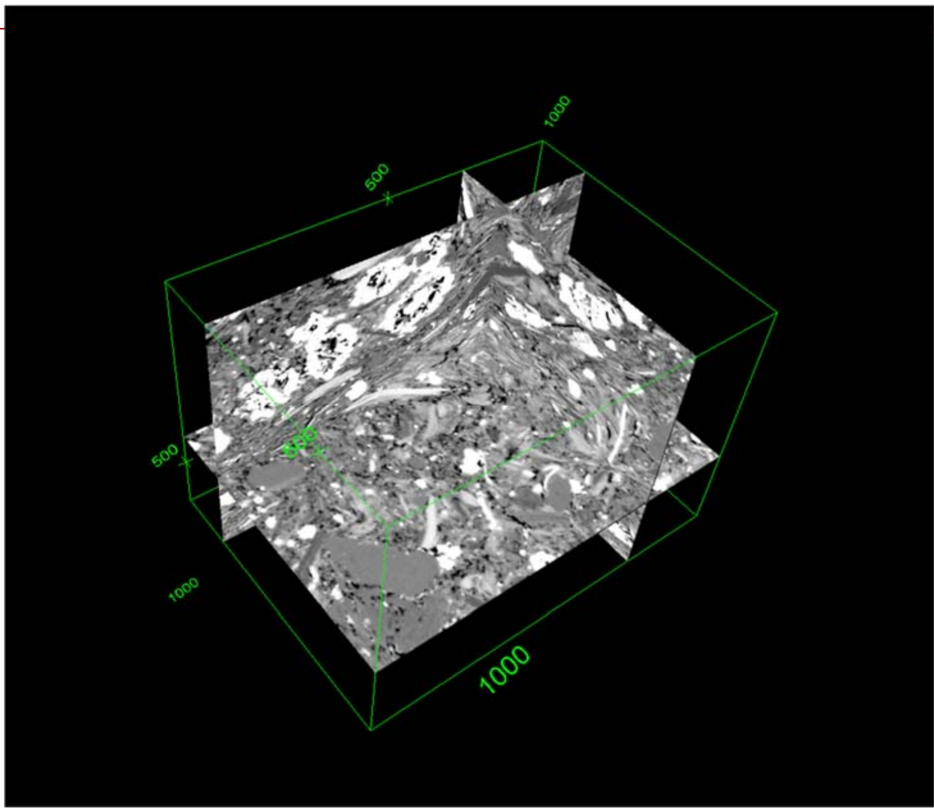
Dr. Souhail YOUSSEF, IFP

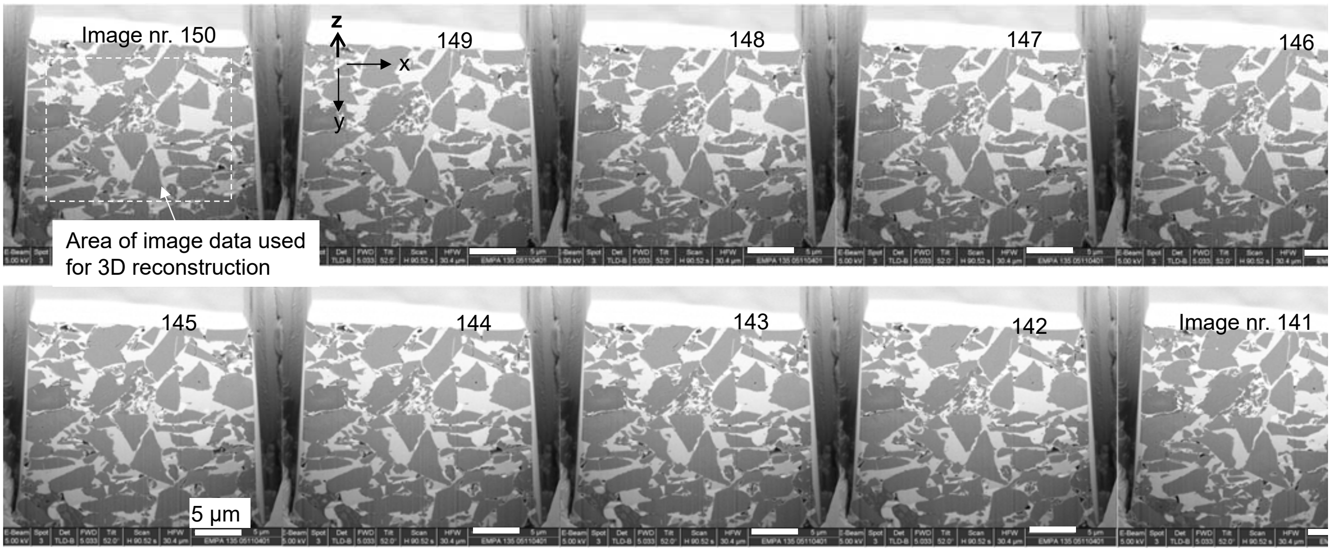






2048x1536x1510, voxel 10x10x10nm





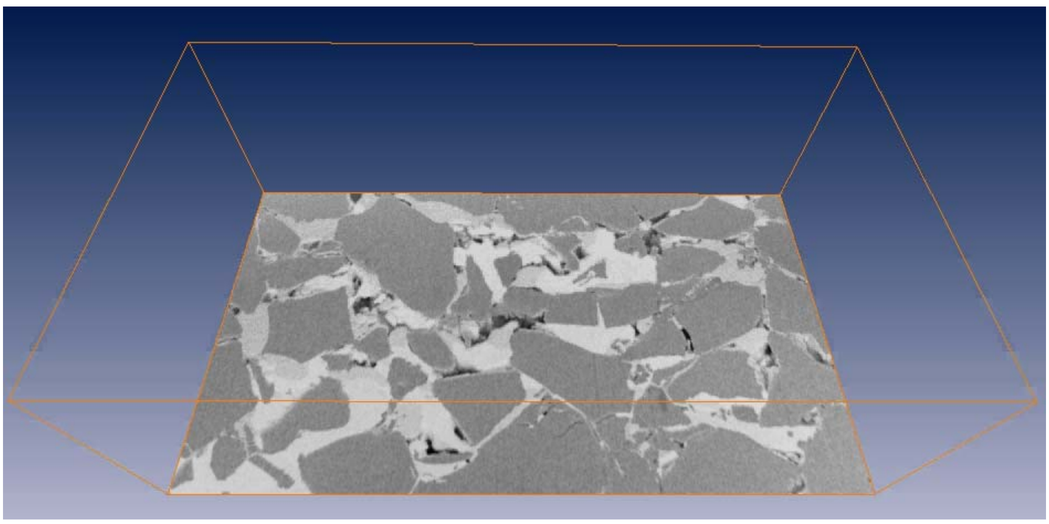
Quantitative microstructure analysis

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

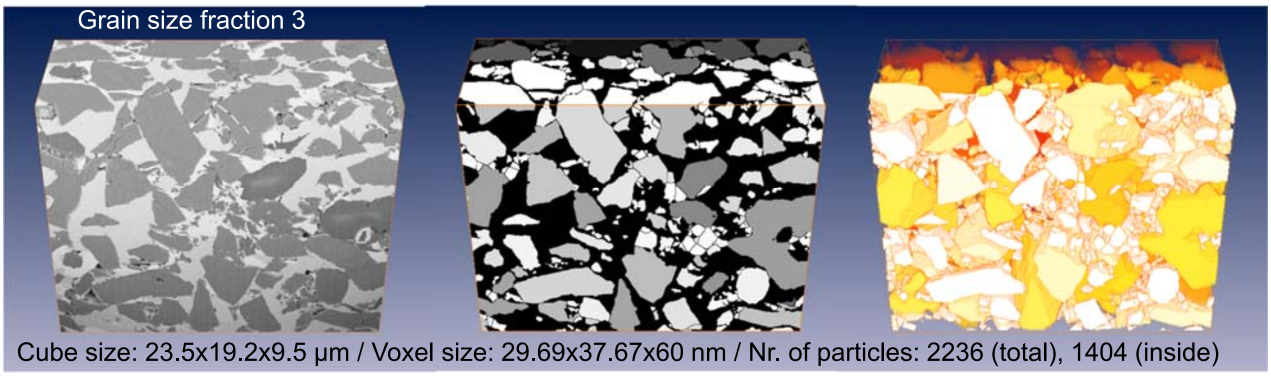
Particle recognition:Edge detection in 3D, Watershed for separation

Voxel: 75nm

Cube:
40*
20*
15 µm



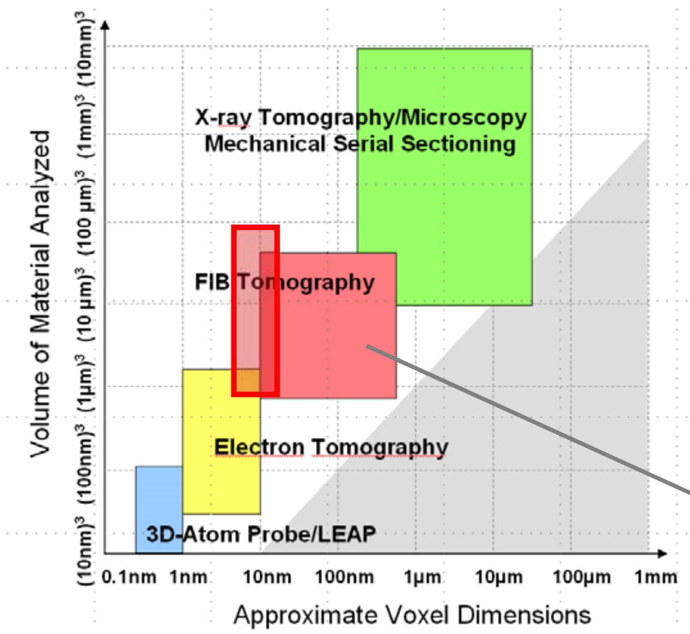
Size, 3D-shape, geometrical relationships between particles



Quantitative microstructure analysis → Algorithms

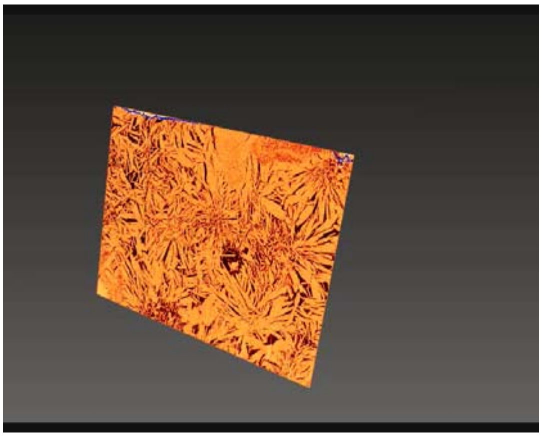
Münch and Holzer 2006
J.Amer.Ceram.Soc.
FIB-nt of particulate systems – part II:
Object recognition and
effect of boundary truncation

FIB-NT compared with other 3D-techniques



From: Uchic, Holzer and Inkson
Mat. Res. Bul.,

Ciment, A. Quennoz IMX-LMC
Materials contrast
1100 slices (20nm thickness)



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