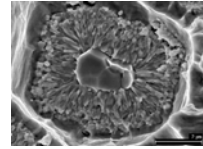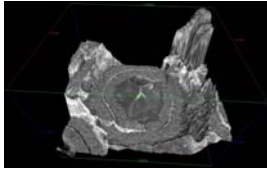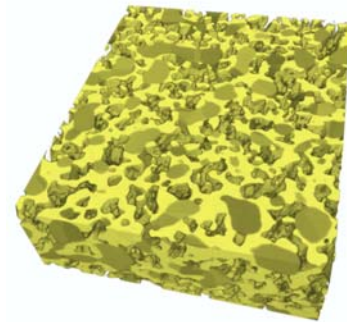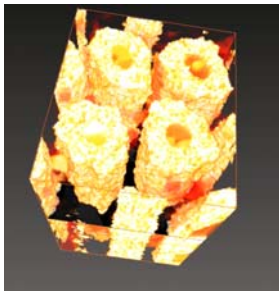# 3D Microscopy and FIB Nanotomography

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"*
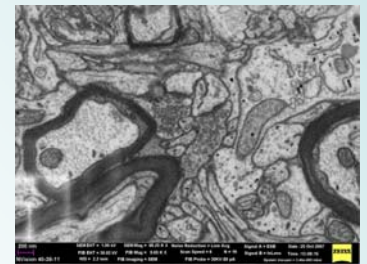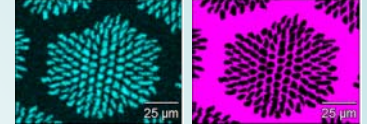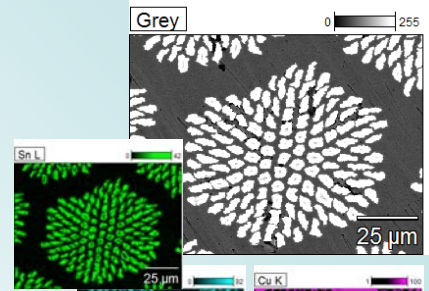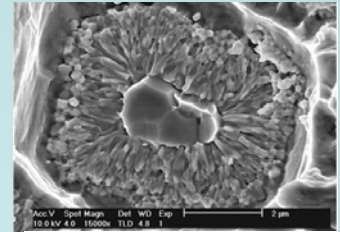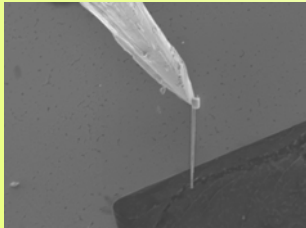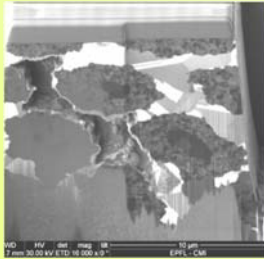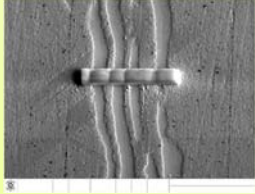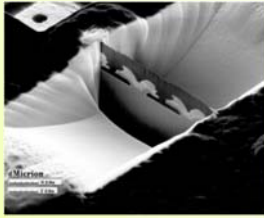
Focused Ion Beam

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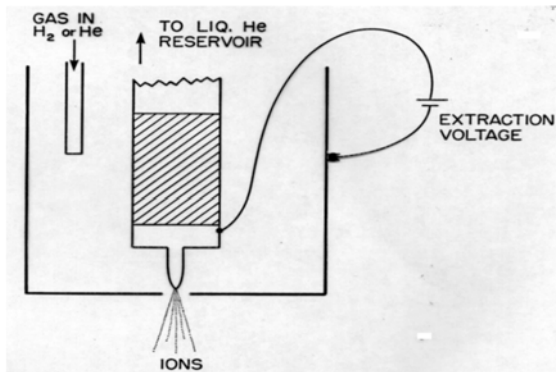 A strong electromagnetic field causes the emission of positively charged ions

SIM = Scanning Ion Microscope
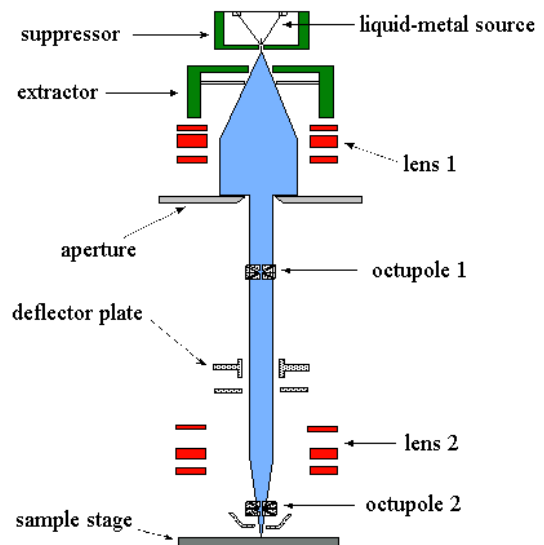
Schematic diagram of a FIB ion column
Source: IBM Almaden Research Center

Ion Sources

a) Gas Field Ion Source

b) Liquid Metal Source
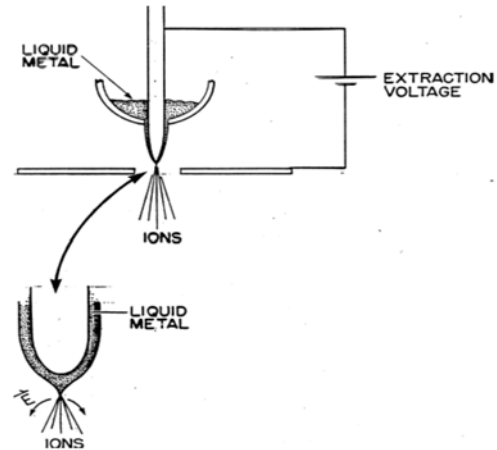
Type of ion source	Ion Species	Virtual source size (nm)	Energy spread, ΔE (eV)	Unnormalized brightness, B (A/cm^{2}sr)	Angular brightness (μA/sr)
Liquid metal	Ga$^+$	50	>4	3×10^6	50
Gas field ion (supertip) (ref. 11)	H$^+$, H$_2^+$, He$^+$, Ne$^+$...	0.5	~1	5×10^9	35

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
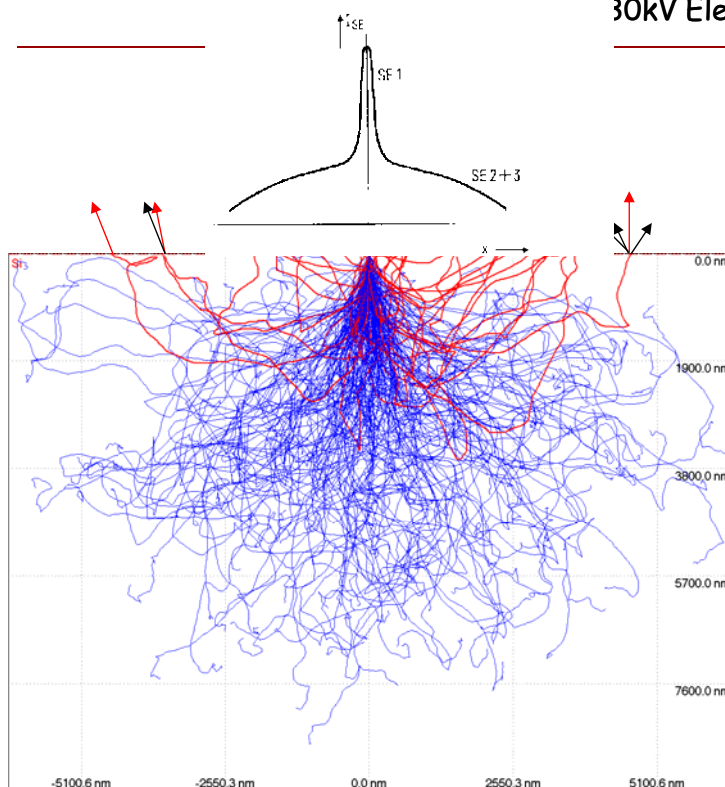

- Sputtering
- Damage
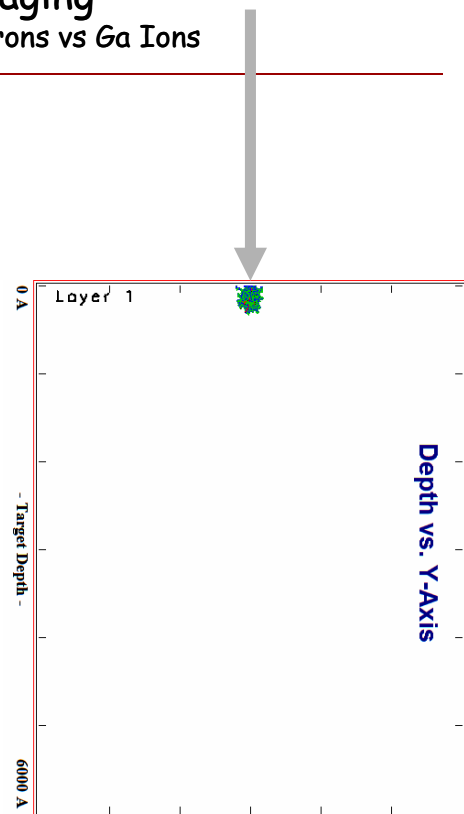- Implantation

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
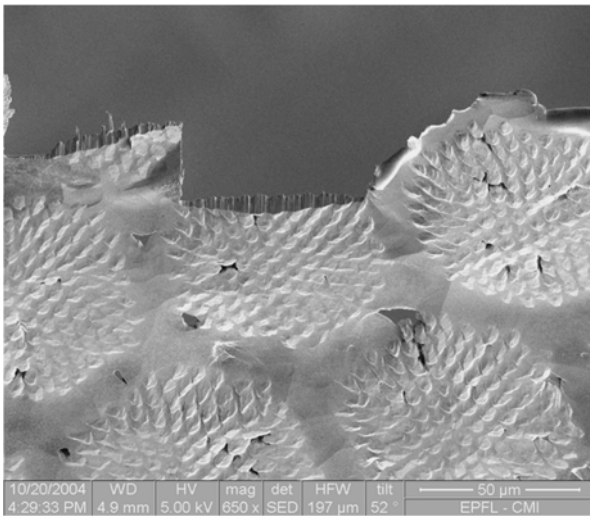 - Induced damage
- Emission of phonons
  - Heating

a)
ion beam
substrate
- secondary e
- secondary i

b)
Ion beam
substrate
- sputtered material from substrate

c)
Ion beam
substrate
- precursor molecules
- deposited film
- volatile products
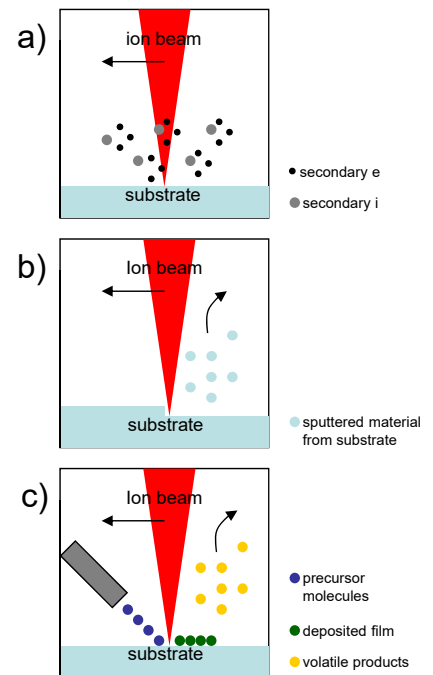
Imaging
30kV Electrons vs Ga Ions

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
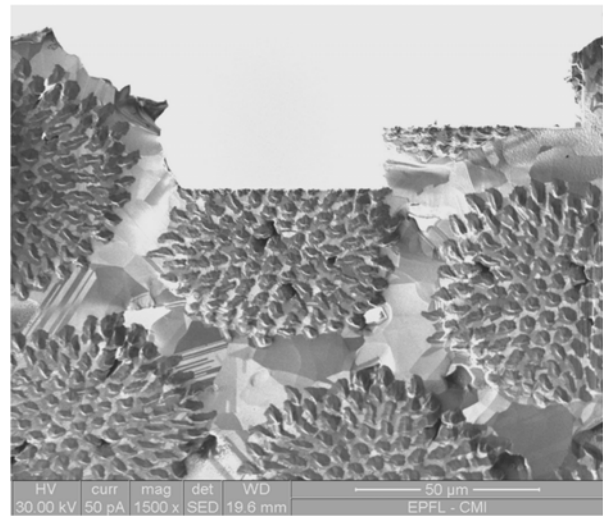
ion-beam 30kV 50pA

material (sputtering) contrast
orientational contrast
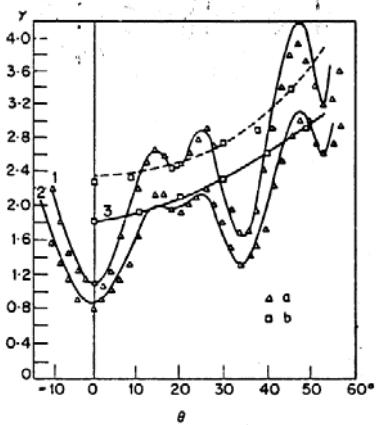
Channeling contrast

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

G. Carter and J.S. Colligan, *Ion Bombardment of Solids,* (Elsevier 1968)
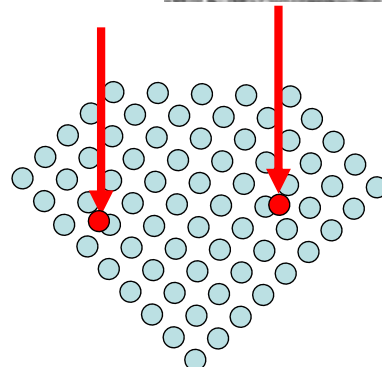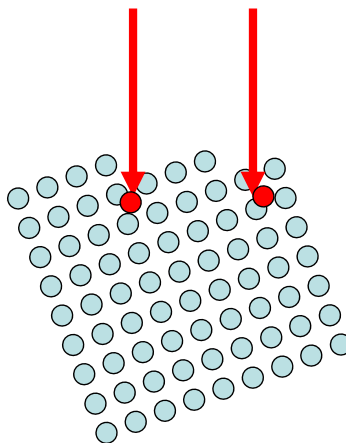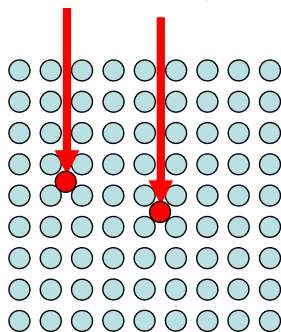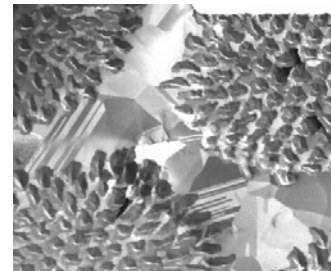
Atom columns align with the ion trajectory = higher penetration
-> less sputtering and less SE electrons

Milling
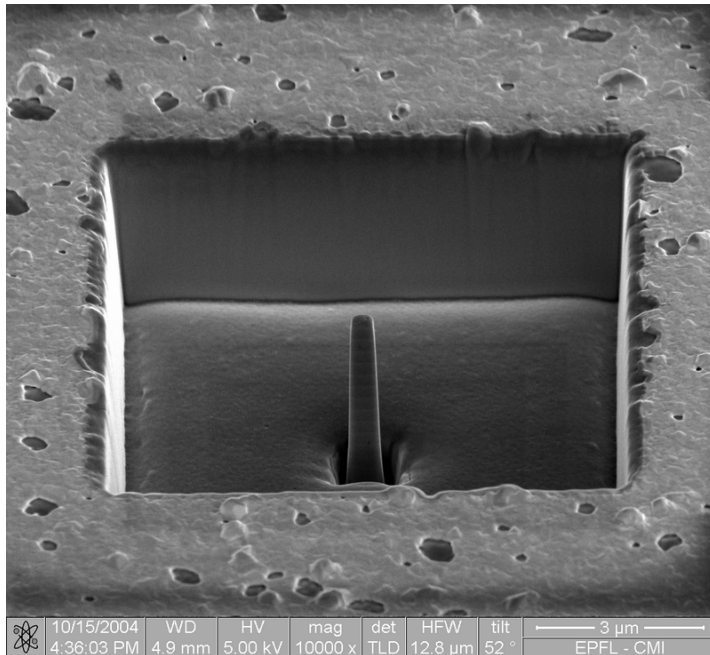

PZT-high aspect ratio „capacitor", W. Adachi (EPFL-LC)

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

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.
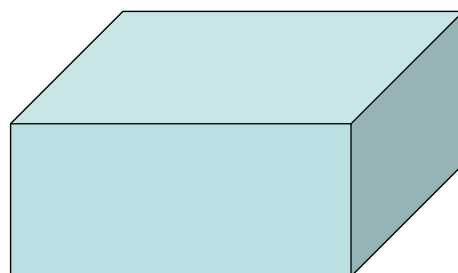
Ion-Solid interaction
Sputtering Yield

- Sputtering yield depends on incident angle ϕ
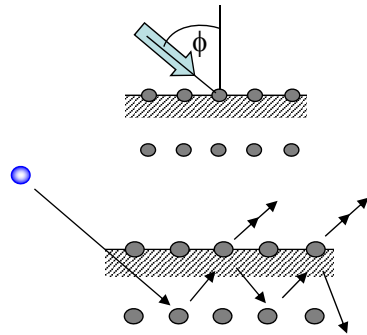
- Higher probability of collision cascades near the surface at higher ϕ
- Sputtering yield has maximum for $\phi = 75°$

X.Xu, et. al.
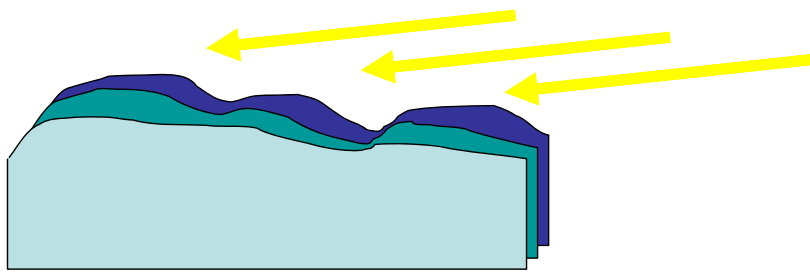J. Vac. Sci. Technol. B, 10, 2675 (1992)

* - Si

o - SiO$_2$

Solid line - Simulation for Si
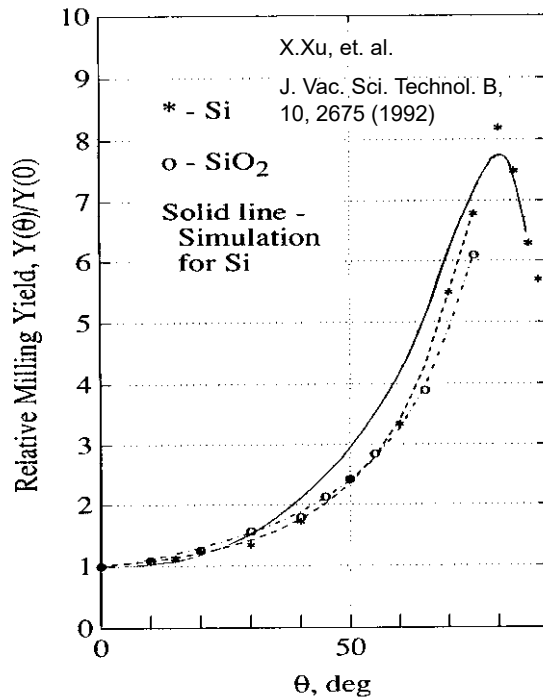
Milling

FIB milling of steel

X.Xu, et. al.
* - Si   J. Vac. Sci. Technol. B, 10, 2675
o - SiO$_2$ (1992)
Solid line Simulation for Si
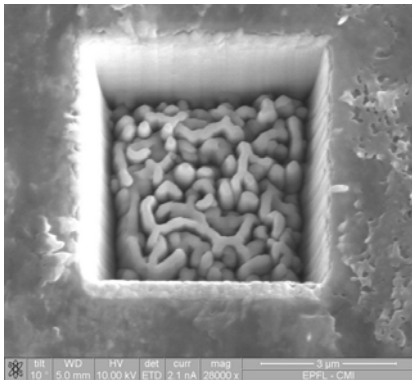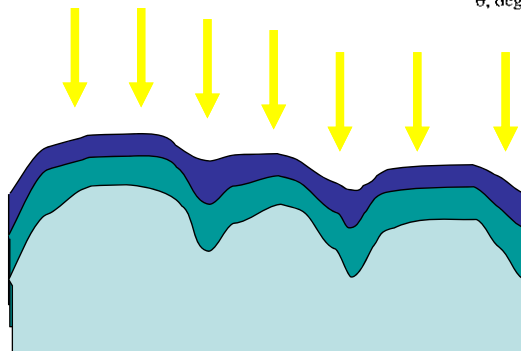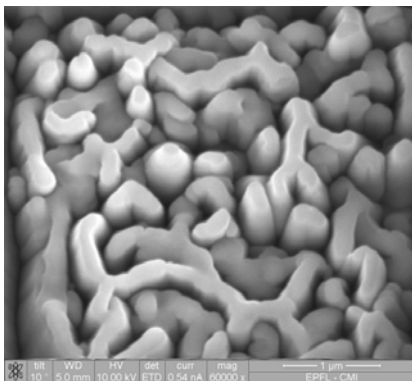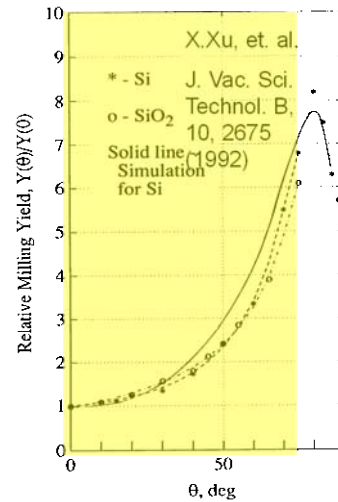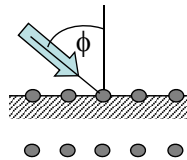
Polishing,
at shallow angles

X.Xu, et. al.

* - Si   J. Vac. Sci.
o - SiO$_2$ Technol. B,
    10, 2675
Solid line (1992)
Simulation
for Si

Gas assisted deposition

ion beam
(e beam)

desorbed
reaction
products

absorbed
molecules

⊗ion

excited surface
atoms

paths of
displaced
substrate
atoms

Collision Cascade
Model

adsorbed
molecules

substrate

Deposition rate

Rate μm/min (thickness/time)

A — High-efficiency deposition per ion. Ion dose on each pass does not decompose all gas. Slow layer growth rate & long deposition time.

B — Most efficient growth rate. Each beam scan uses up nearly all gas.

C — All gas is used up by only part of ion dose at each point. Remaining ions then sputter surface.

Deposition stops and milling begins

Lower beam current; larger patterns

$2–6$ pA/μm^2 medium patterns

Higher beam current; smaller patterns

Current Density (pA/μm^2)

Nanofabricated structures

Coil 700nm pitch, 80nm line width, diamond-like amorphous carbon, FIB induced CVD
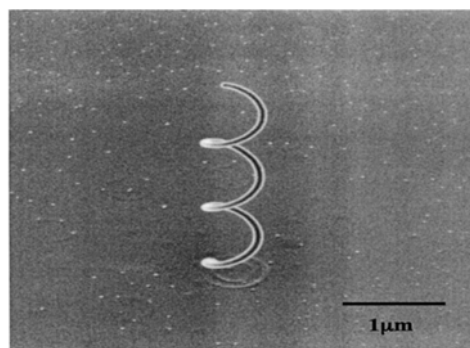
Shinji Matsui, et.al.
J. Vac. Sci. TechnolB18, 3181
(Nov/Dec, 2000)
(HimejiInstitute of Technology,Hyogo, Japan)

1μm

1μm

3μm
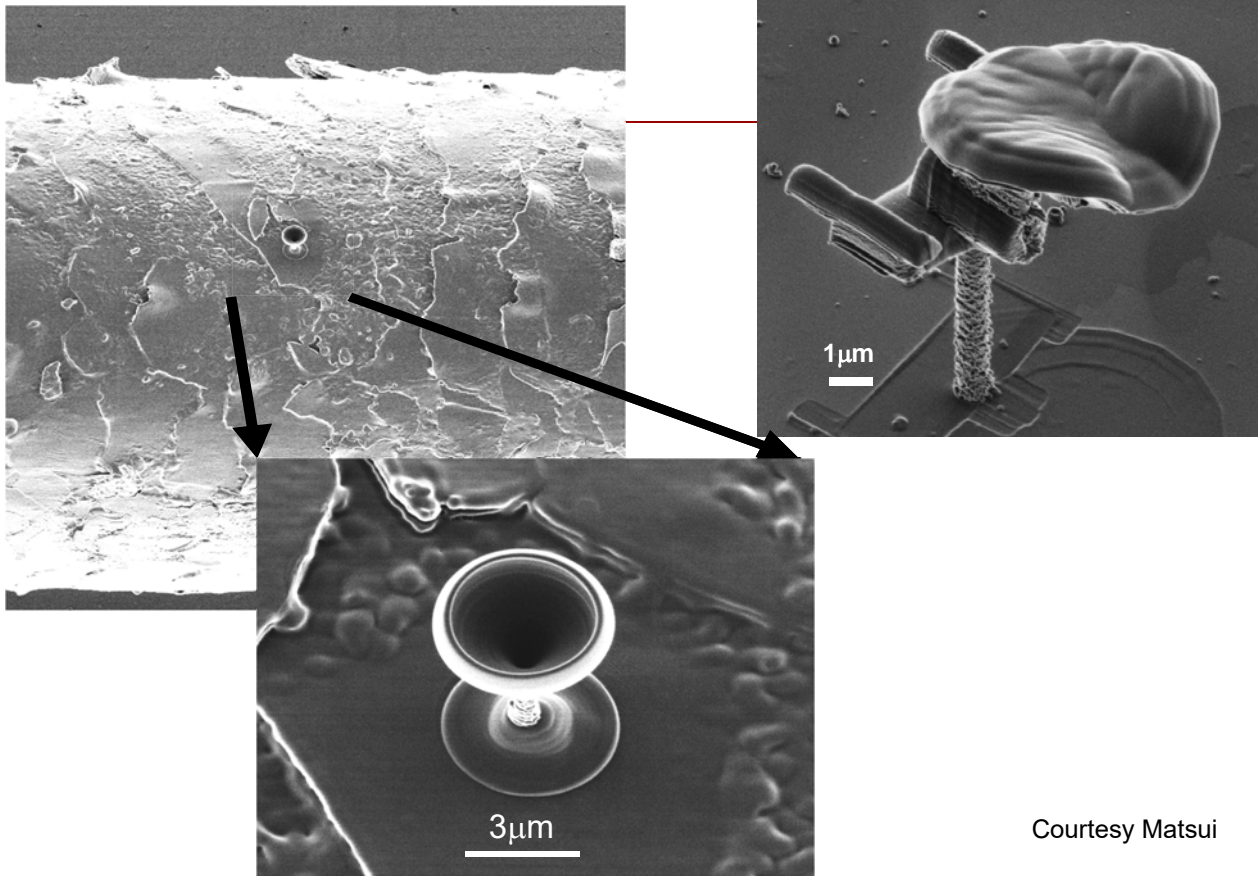
Courtesy Matsui

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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)

M. Pavius CMI
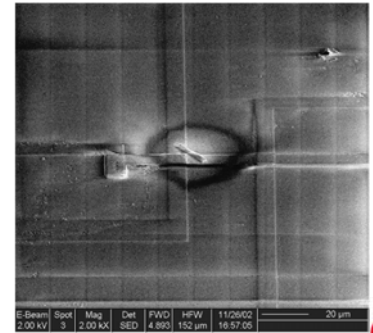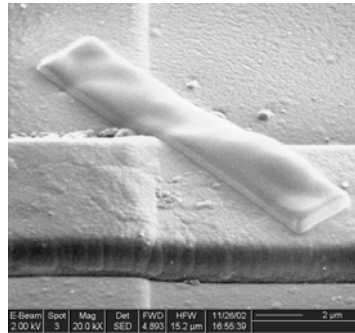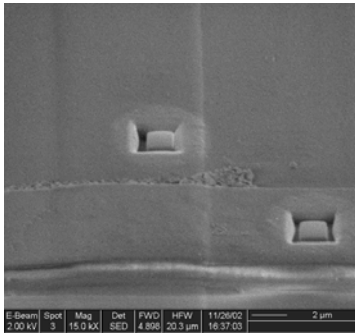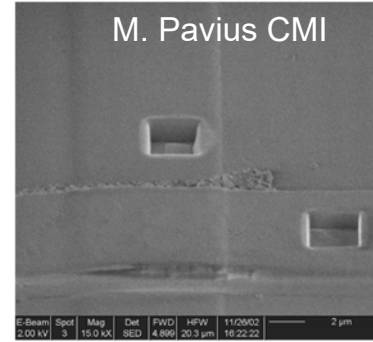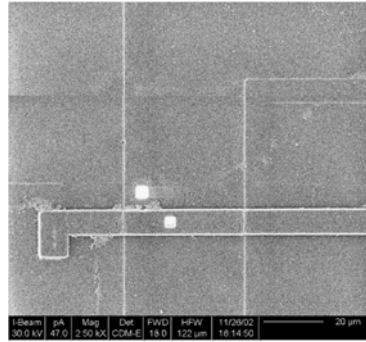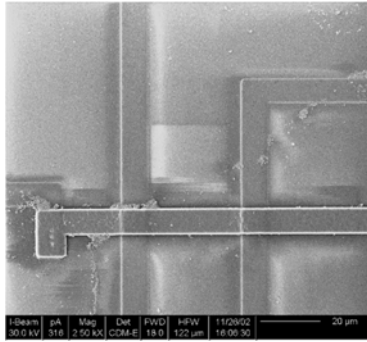
FIB-manufactured AFM-tips
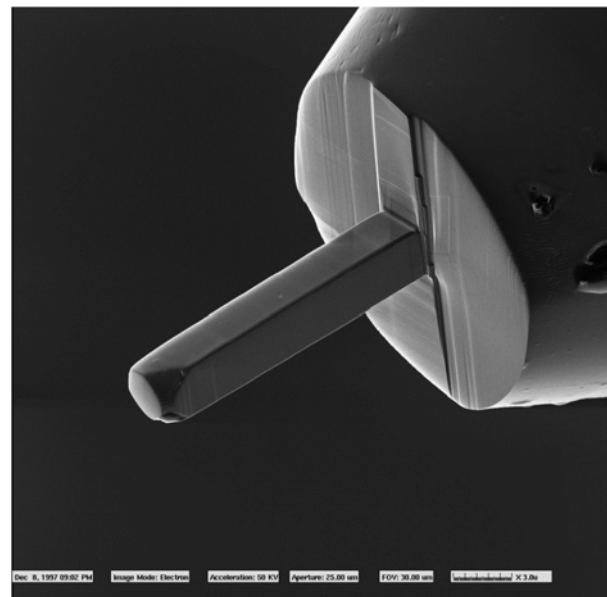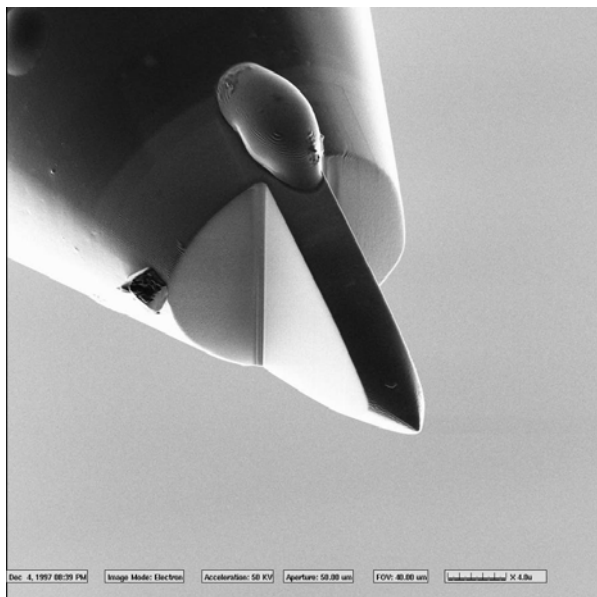
Failure analysis

FIB cross-sectioning and
SEM imaging
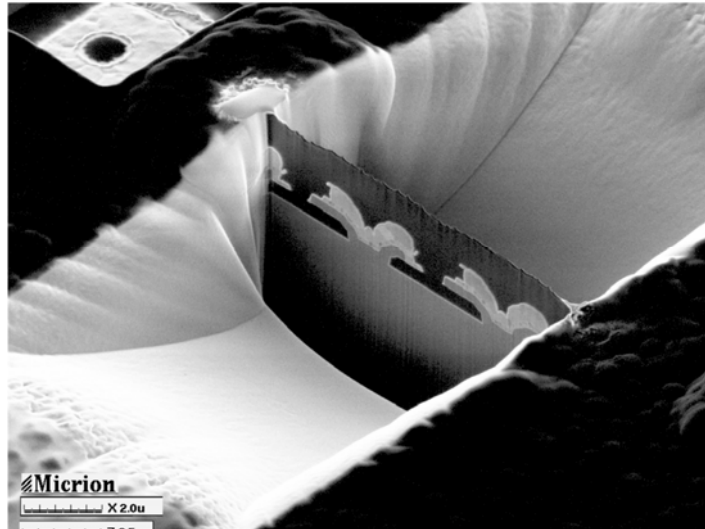
c) TEM preparation
in-situ lift-out movie
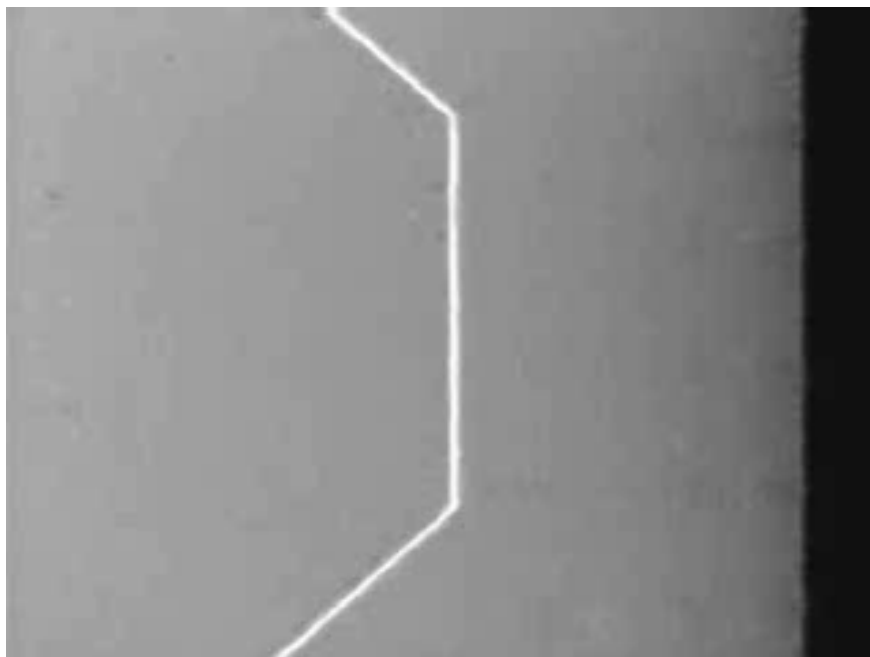
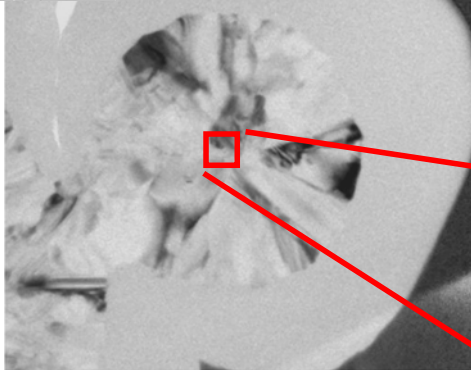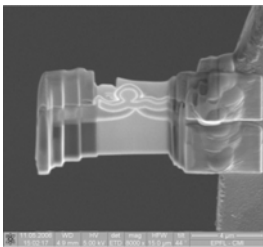(downloaded from http://www.feicompany.com/

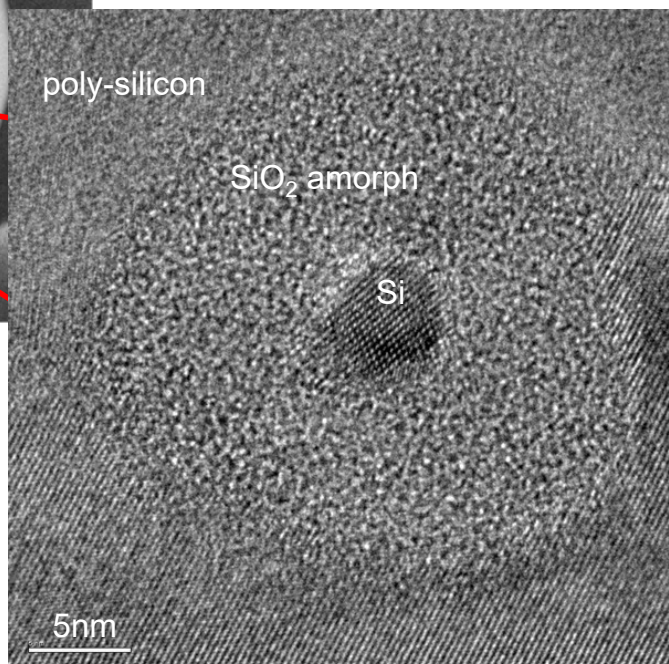# Site specific TEM lamella extraction
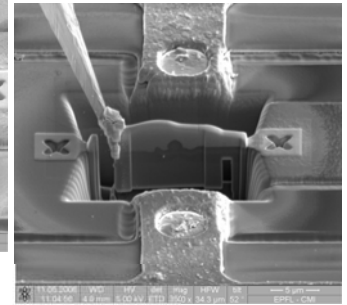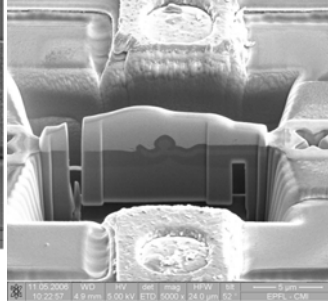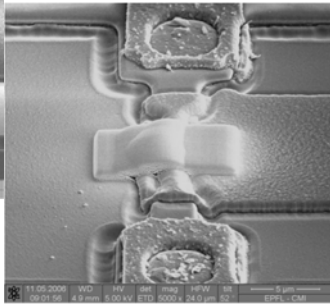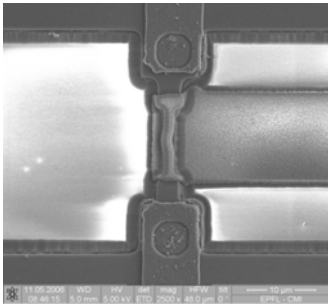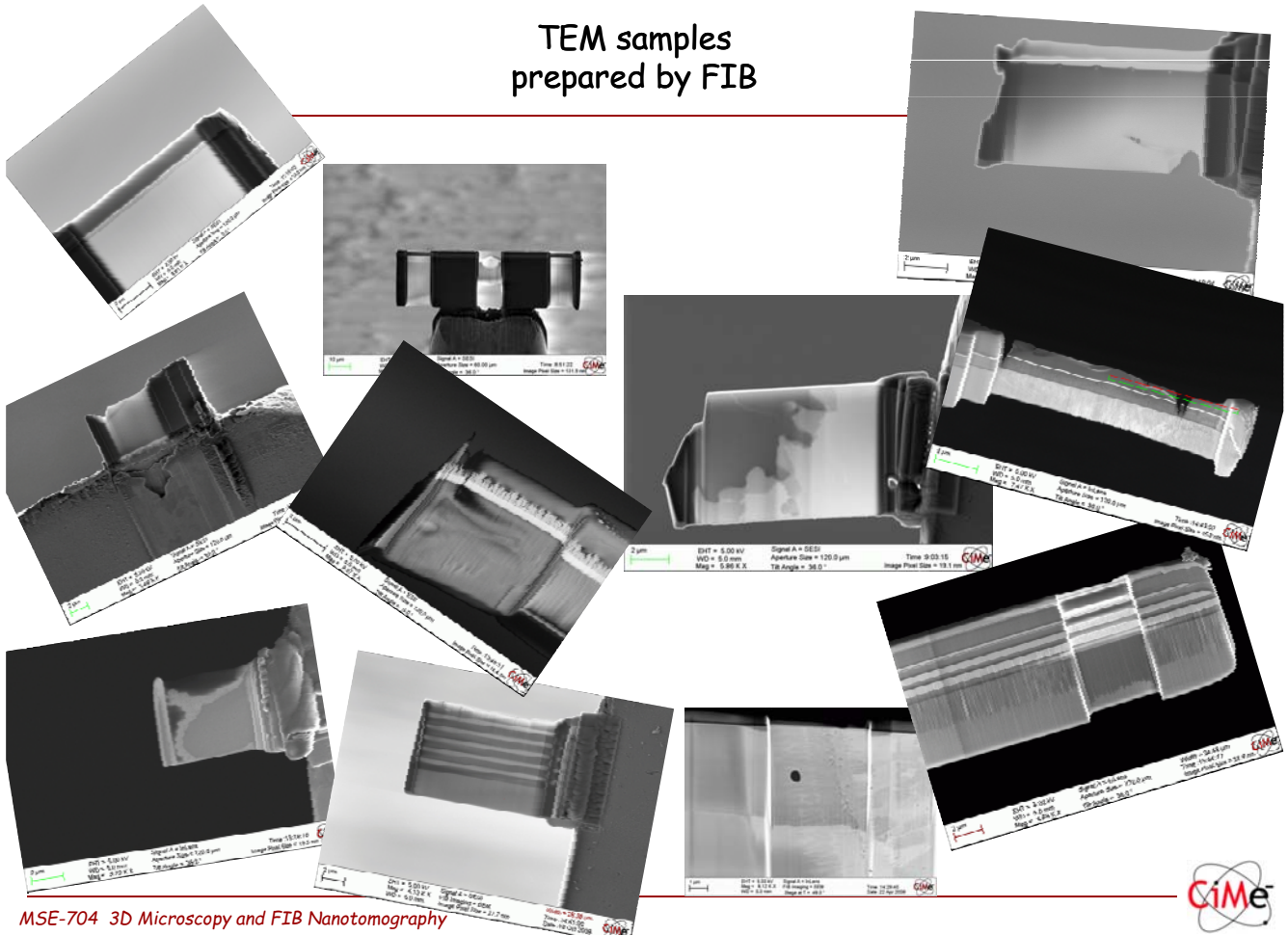
5nm Si Nano-wire

M. Pavius, V. Pott, CMI
M.Cantoni, CIME
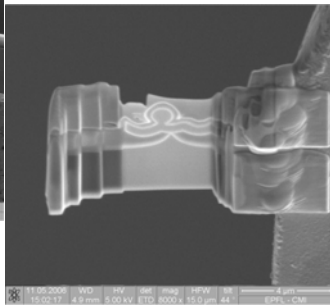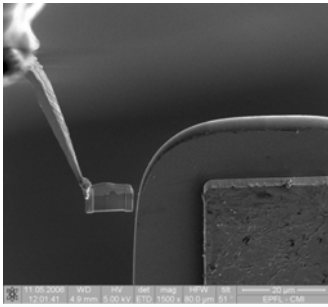
TEM sample "grid" , diameter 3mm

Si-Nanowire
TEM, HRTEM

poly-silicon

SiO_2 amorph

Si

5nm
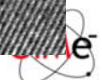
TEM samples
prepared by FIB

TEM lamellae by FIB
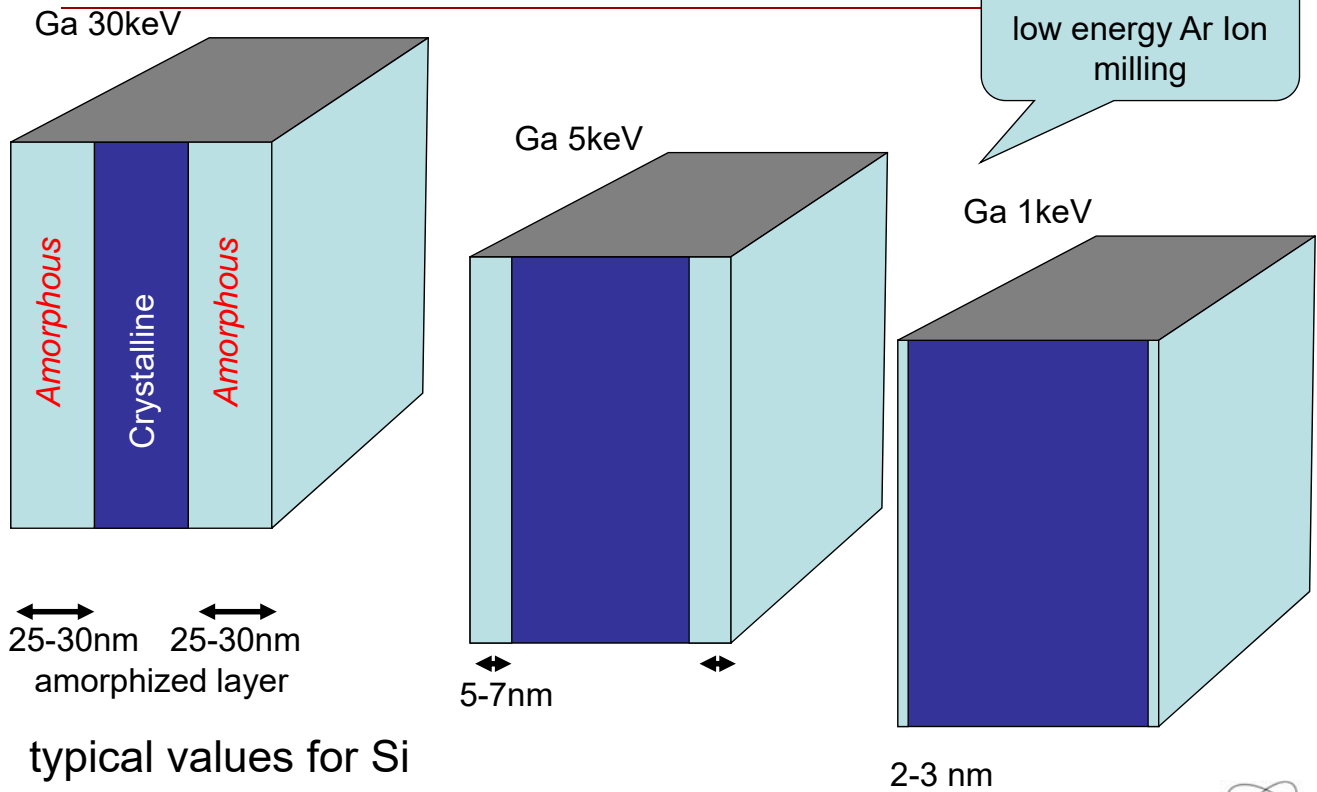
Focused Ion Beam adds a new dimension
to TEM specimen preparation

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

Ga 30keV

Amorphous | Crystalline | Amorphous

Ga 5keV

Alternative:
low energy Ar Ion milling

Ga 1keV

25-30nm   25-30nm
amorphized layer

5-7nm

typical values for Si

2-3 nm

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

SiO2 cap

50 nm

Amorphous Si

22 nm

Crystalline Si
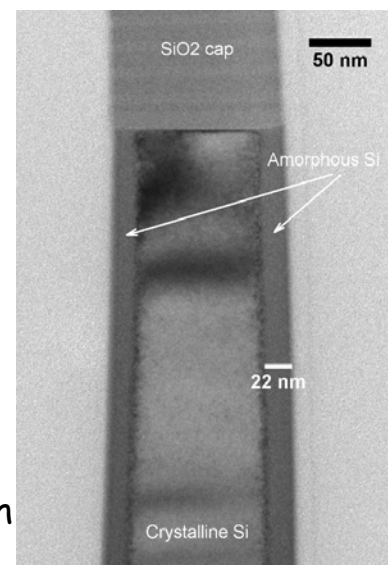
Cross-section of TEM lamella

NVision 40 Argon

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

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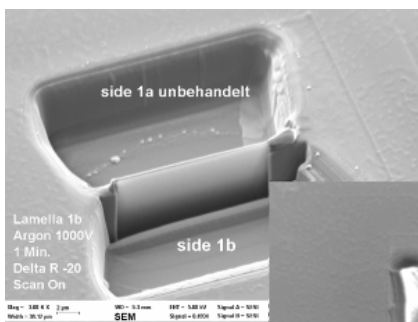**René Hübner, Yvonne Ritz, Dirk Uteß, Beate Volkmann, Hans-Jürgen Engelmann**
GLOBALFOUNDRIES Dresden LLC & Co. KG, Germany

TEM lamella after FIB	Front side polishing	Back side polishing
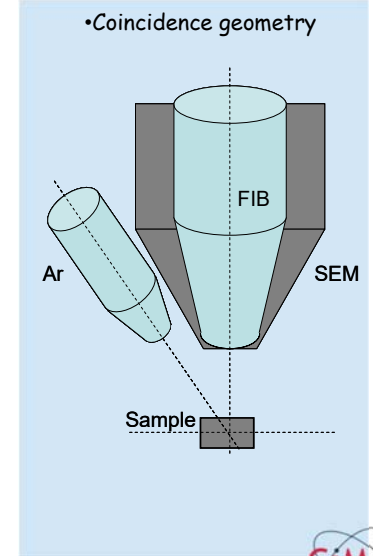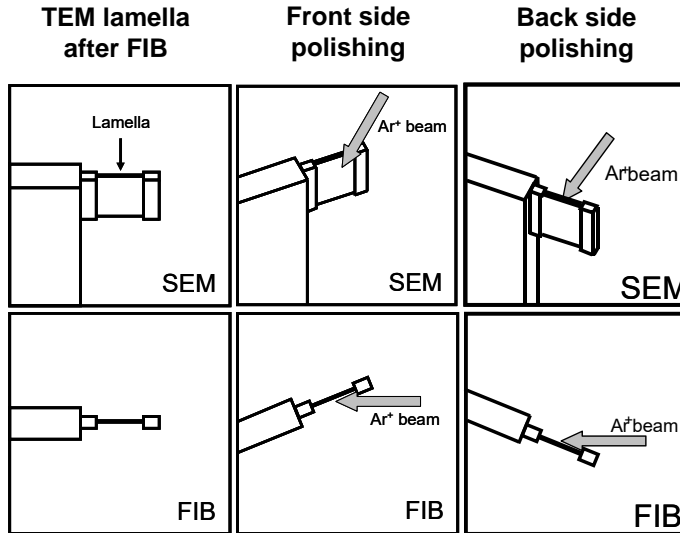
• Coincidence geometry

Amorphization layer thickness - Measurement method

1. FIB cut and polish test lamella

2. Epoxy refill
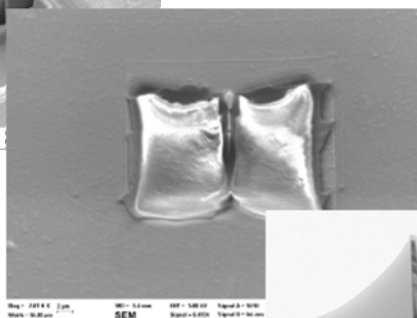
3. FIB cut counter-lamella
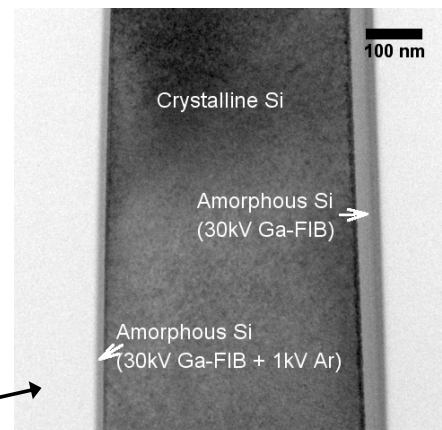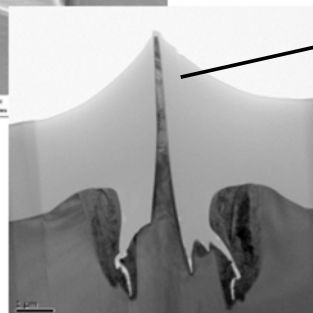
4. Measure damage layer thickness in TEM

Crystalline Si

Amorphous Si (30kV Ga-FIB)

Amorphous Si (30kV Ga-FIB + 1kV Ar)

100 nm

Amorphization layer thickness – Results
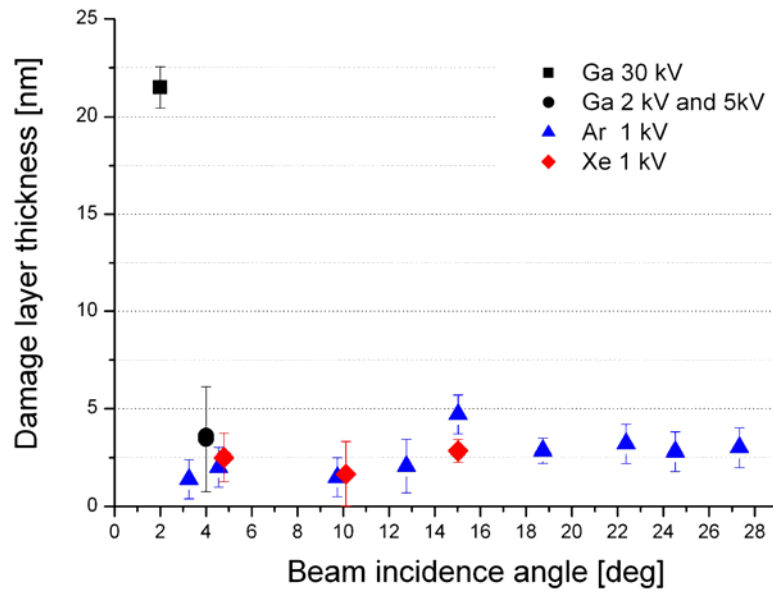
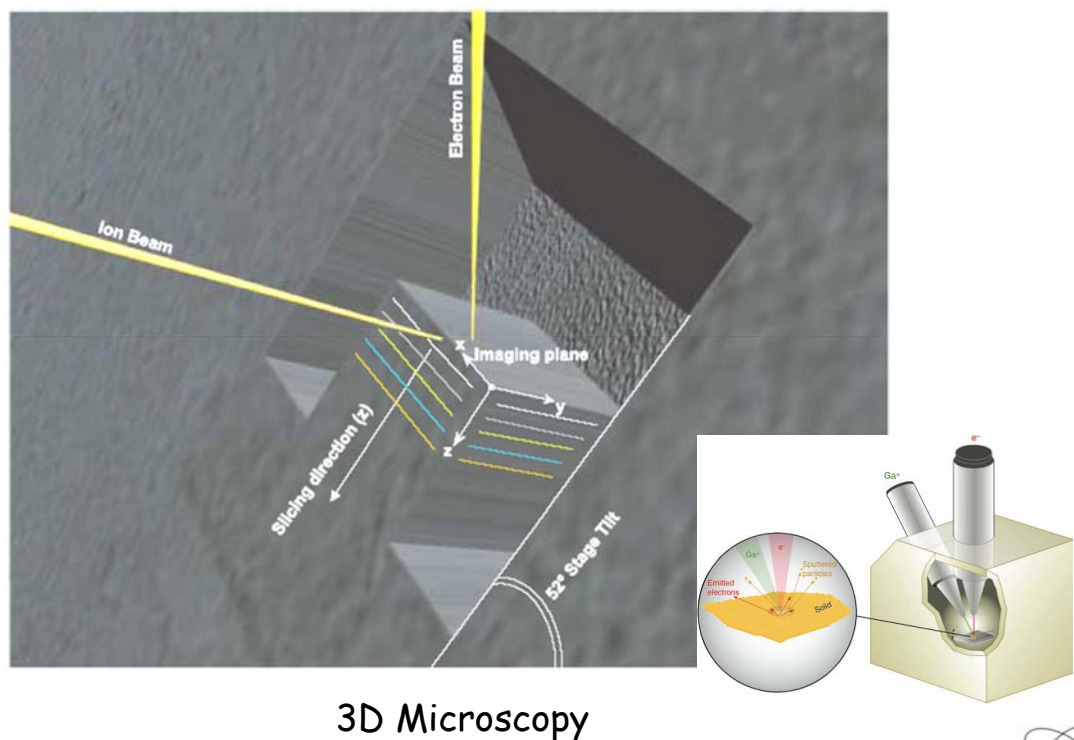# Cs-corrected HRTEM of Si/SiO$_2$ interface
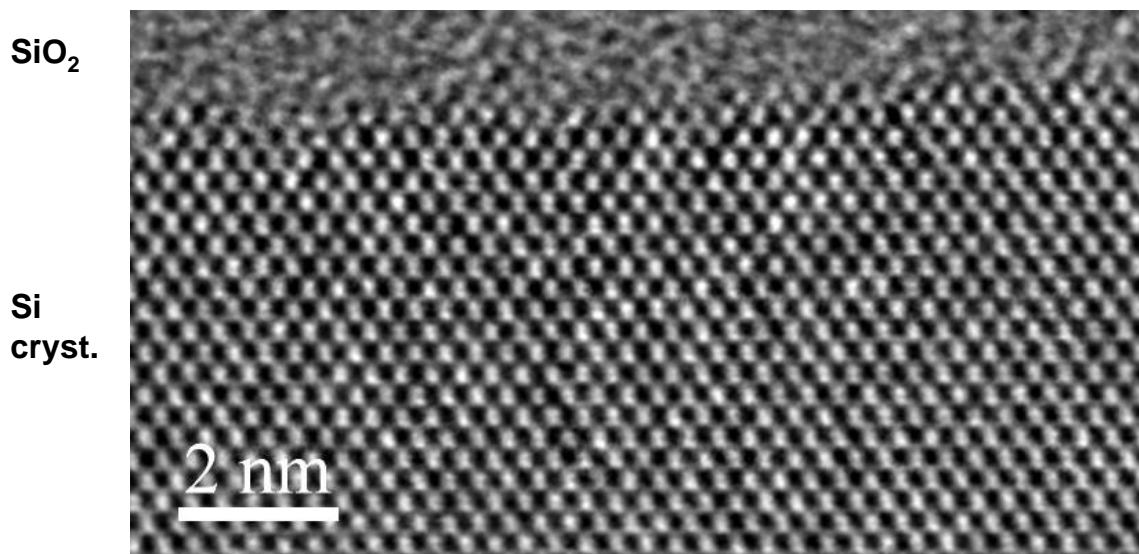
Liftout lamella 1kV Ar polished

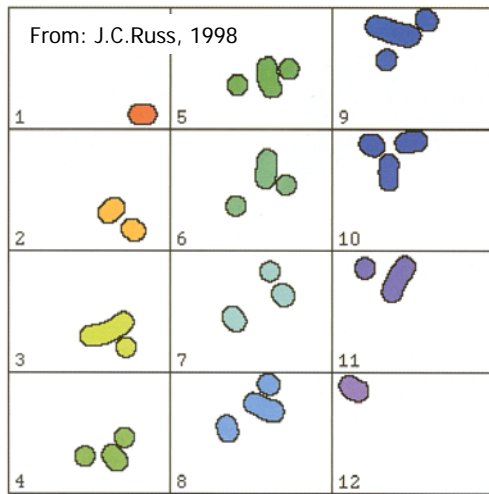·(Courtesy of R. Hübner/Globalfoundries Dresden LLC & Co. KG)
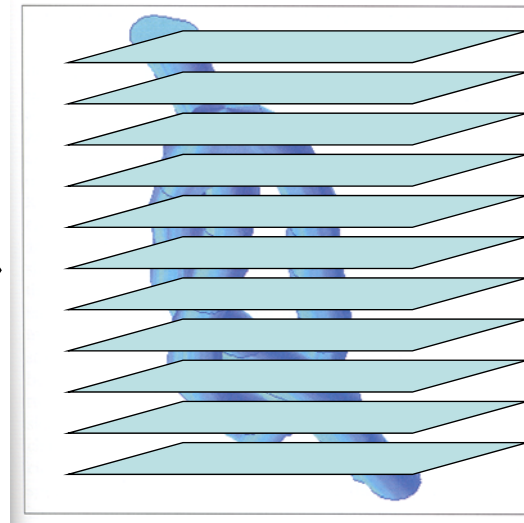
3D Microscopy

FIB Nanotomography

3D Microscopy

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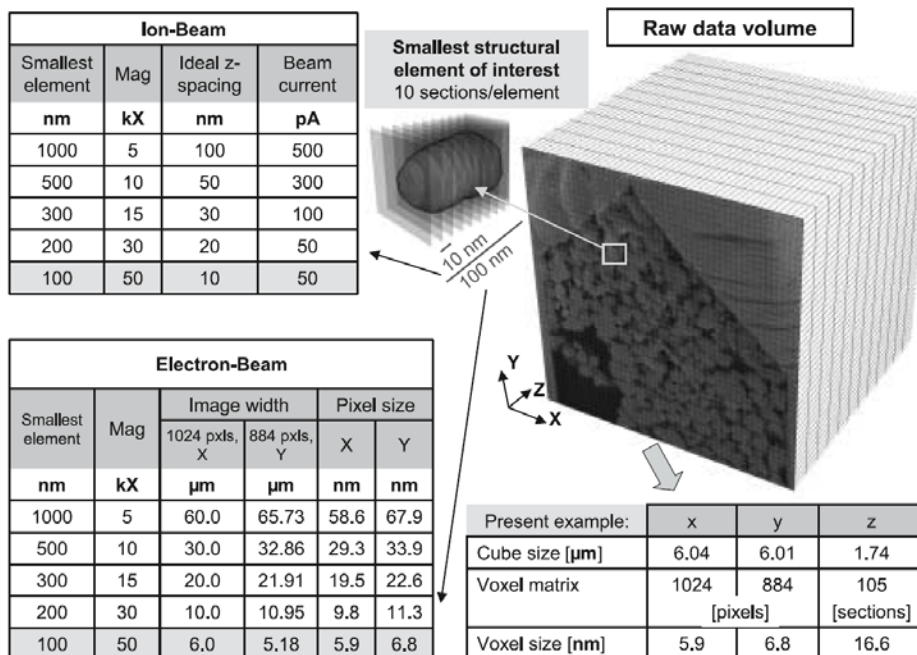## 3D-reconstruction of disordered microstructures

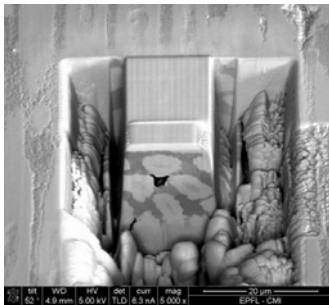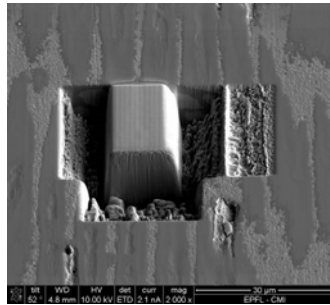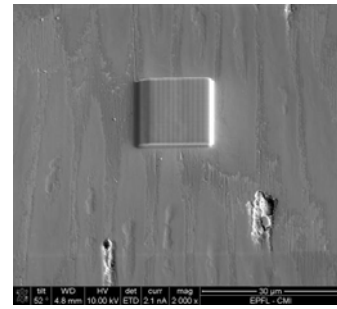From: J.C.Russ, 1998

2D Volume fraction

3D

?? 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

Smallest structural element of interest
10 sections/element

10 nm
100 nm

Raw data volume

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

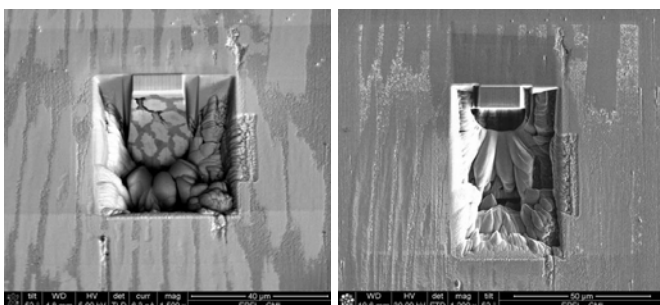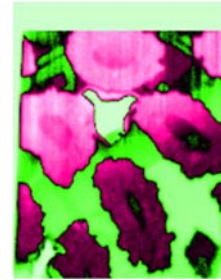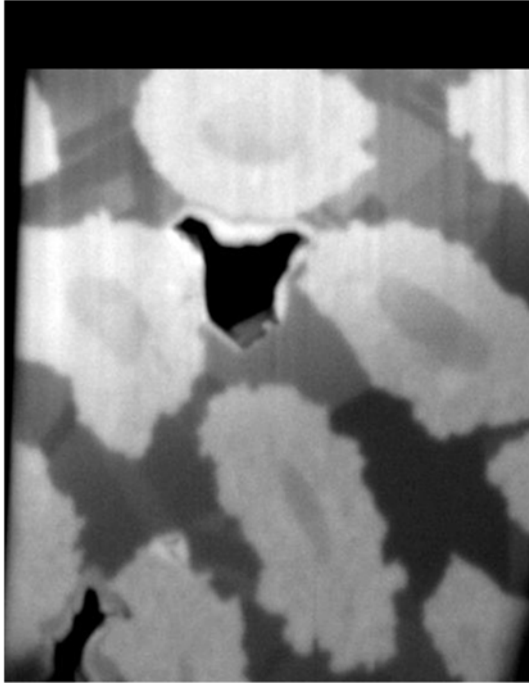# 3D slicing of multifilament Nb₃Sn superconductor
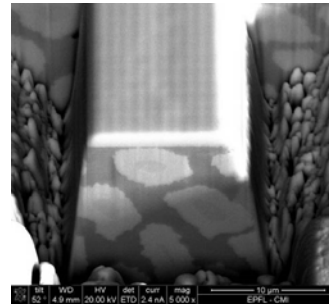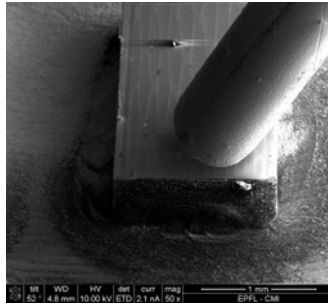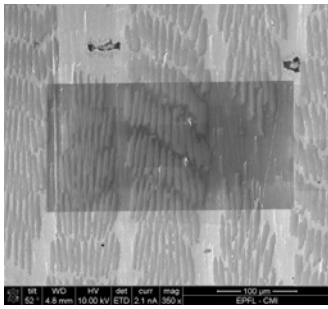
Preparing for slicing
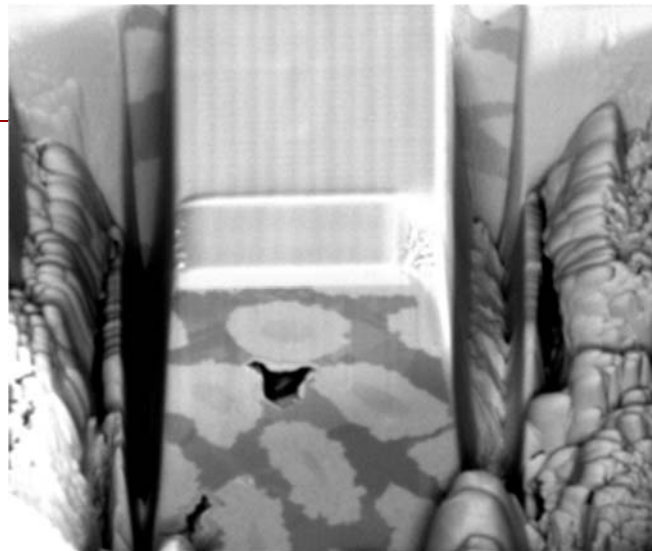
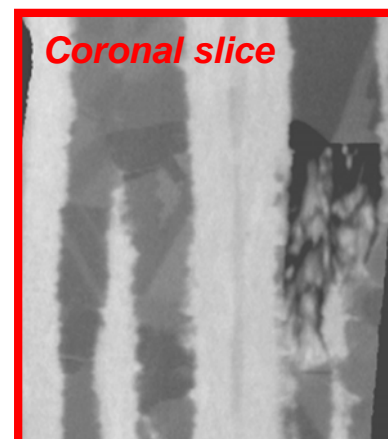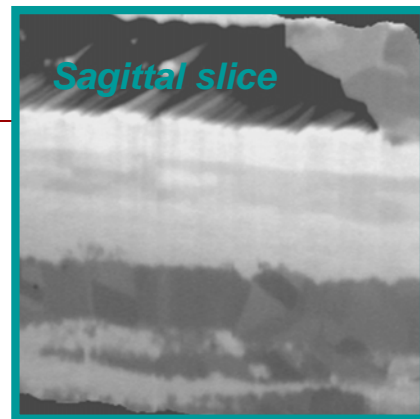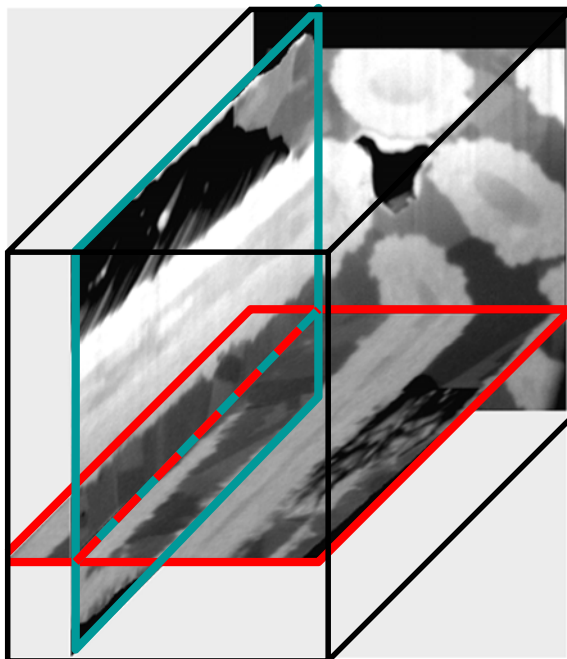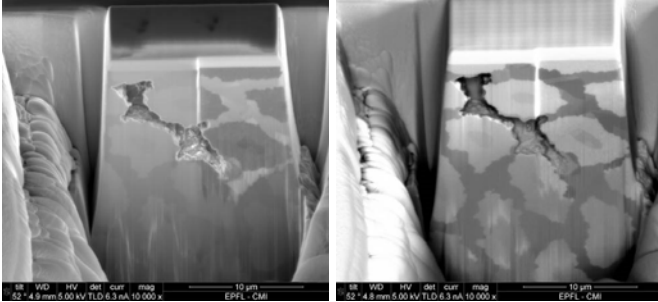Automated milling and imaging of 170 slices (10h)

the end

align and crop

ImageJ
Image Processing and Analysis in Java

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

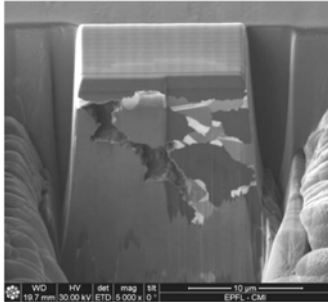**3D volume rendering, reconstruction:**

Orthogonal slices

Sagittal slice

Coronal slice

The choice of the right detector

SE detector (TLD)   BSE detector (TLD)

Ion beam imaging (SE)
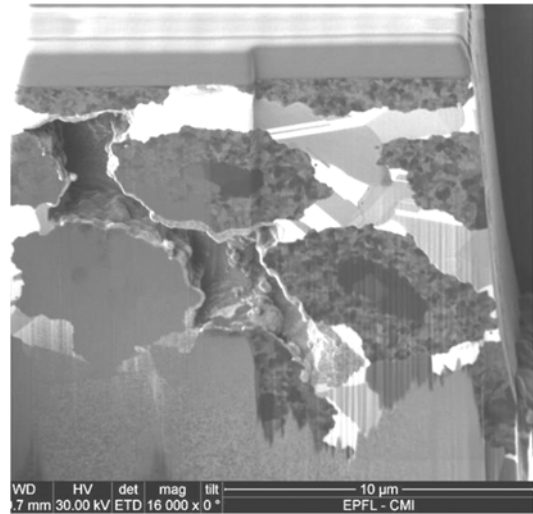
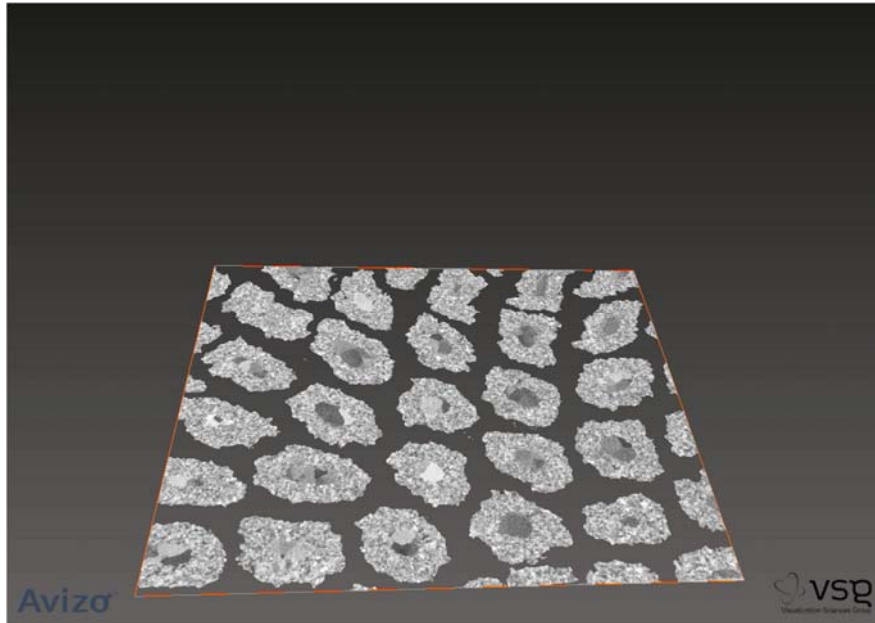
Ion beam for slicing and imaging requires stage movement…!

Nb3Sn multifilament Superconductors
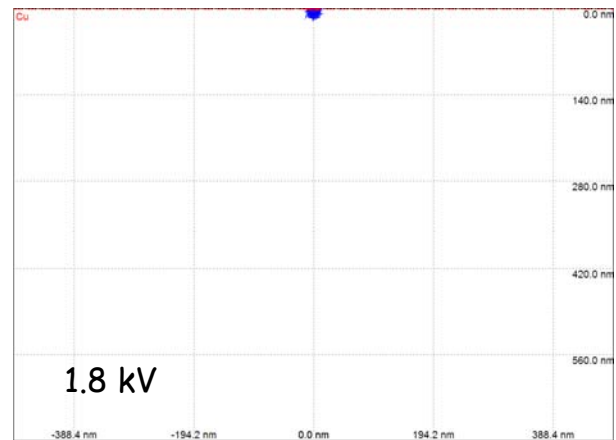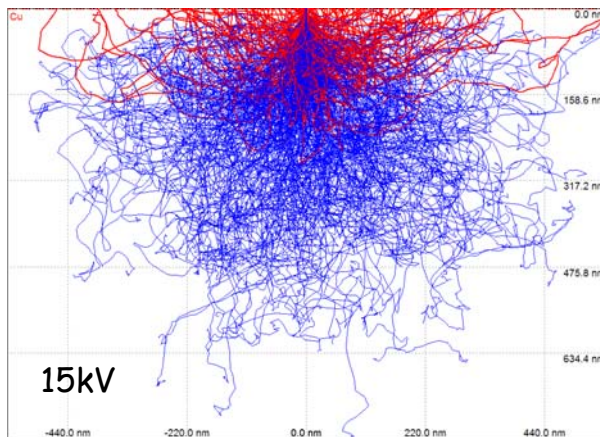
Materials & grain contrast

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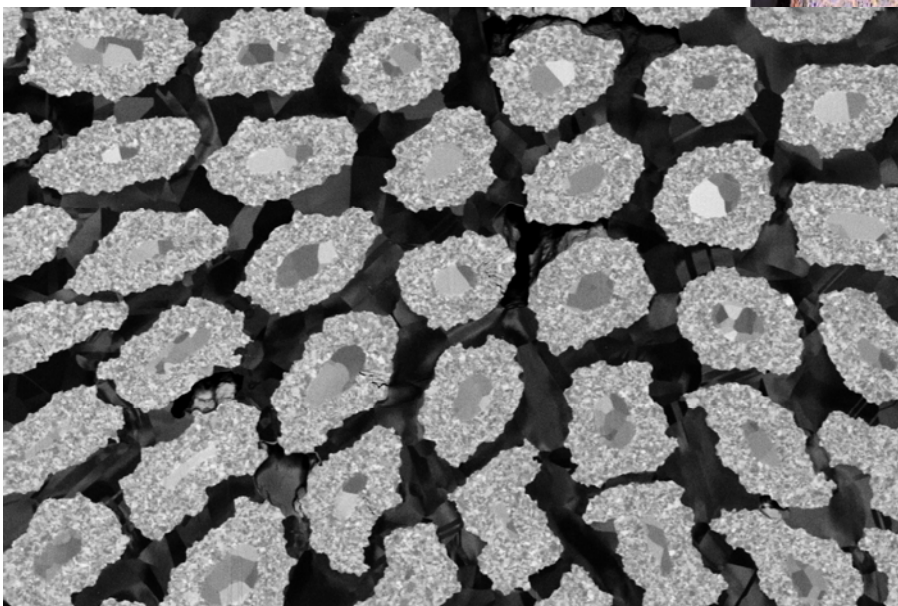
Nb_3Sn superconductor multifilament cable:
14'000 Nb_3Sn filaments (diameter ~5um) in bronze matrix
1.8kV EsB detector

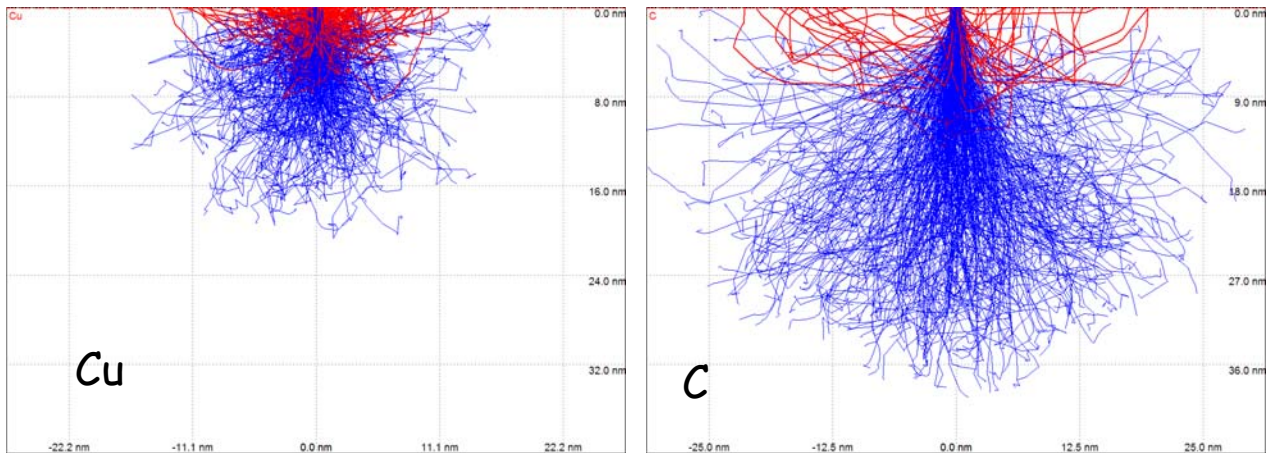# Chemistry and orientation

Nb$_3$Sn multifilament Superconductors
Materials & grain contrast
2048x1536x1700 (10x10x10nm voxel)

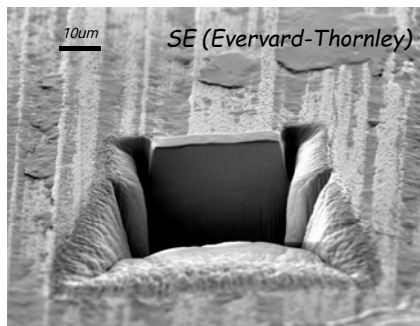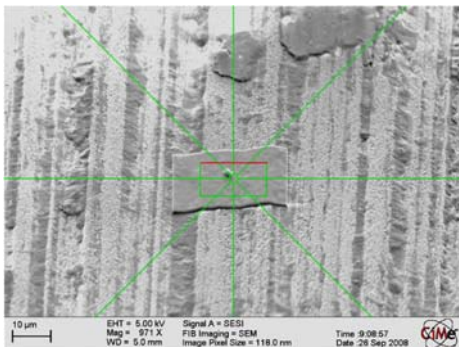# What is the spatial resolution in BSE imaging ...?

Scatter range in Nb$_3$Sn:
Monte-Carlo Simulation of electron trajectories
backscattered electrons
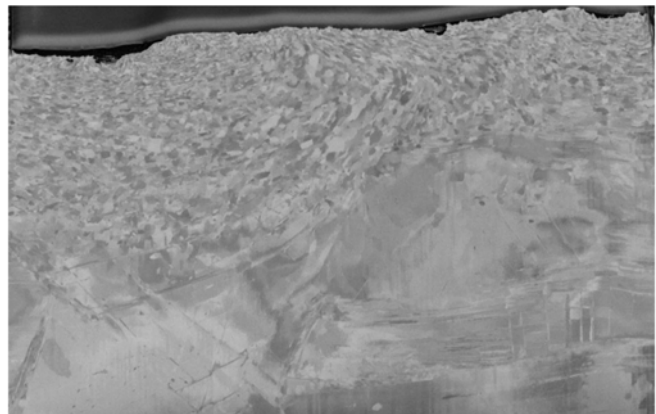
Interaction volume at 1.5kV, BSE escape depth

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

Orientation contrast
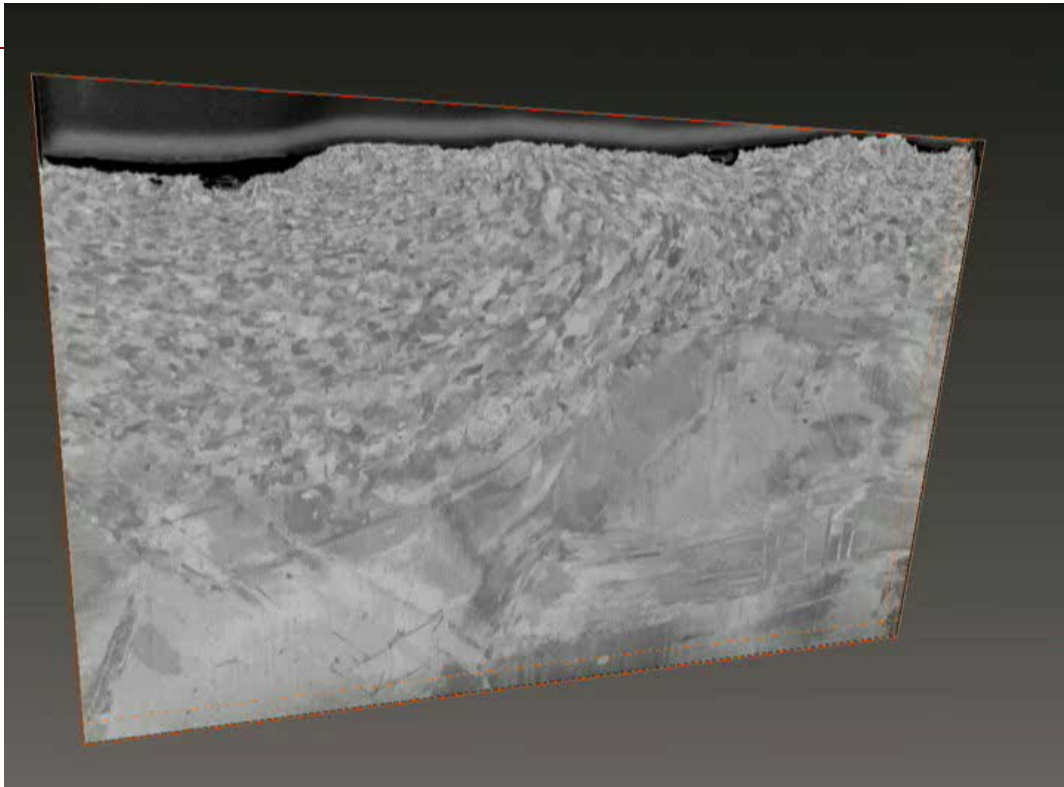### *identification of grain texture*

Tribology: wear trace on steel
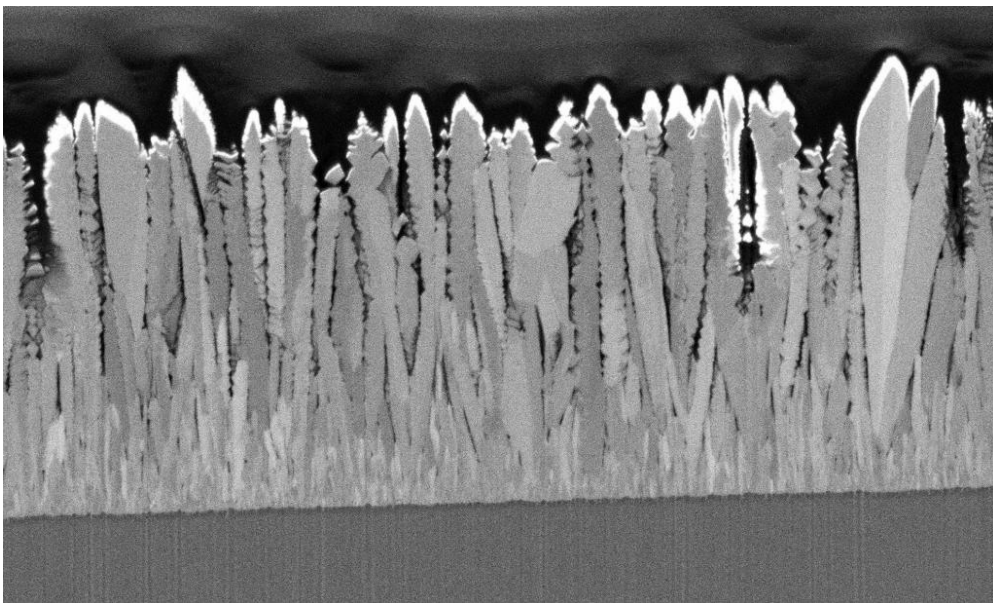## Tribo-corrosion

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

SE (Evervard-Thornley)

BSE

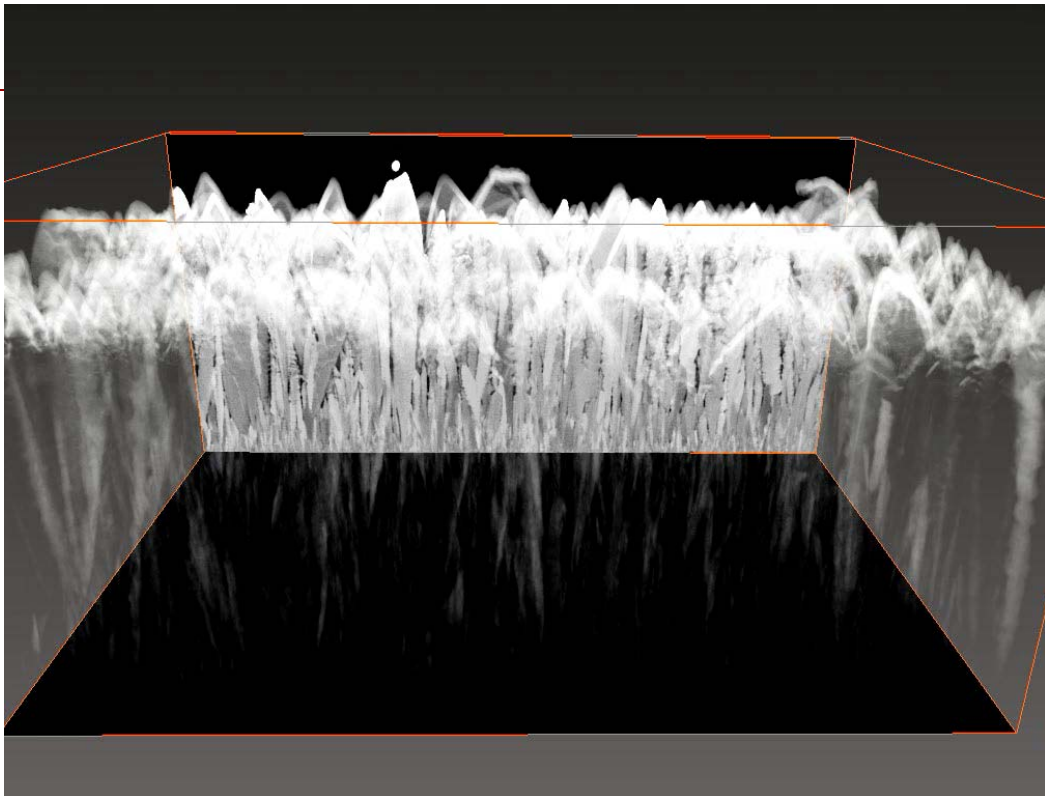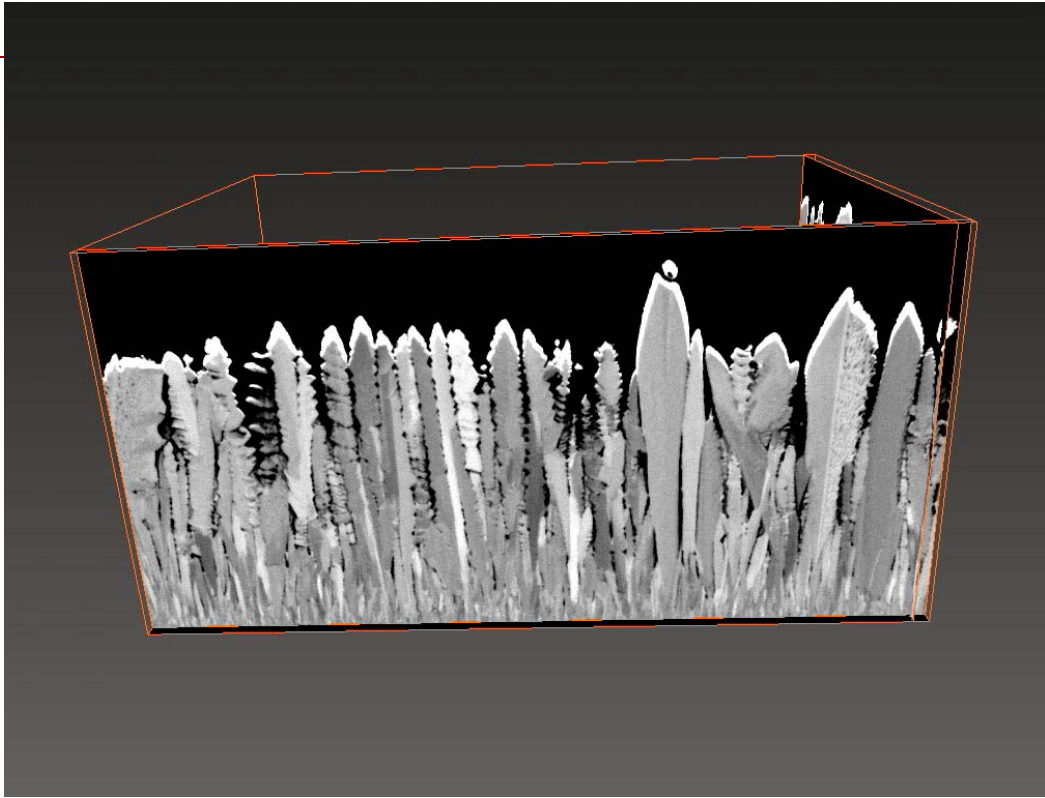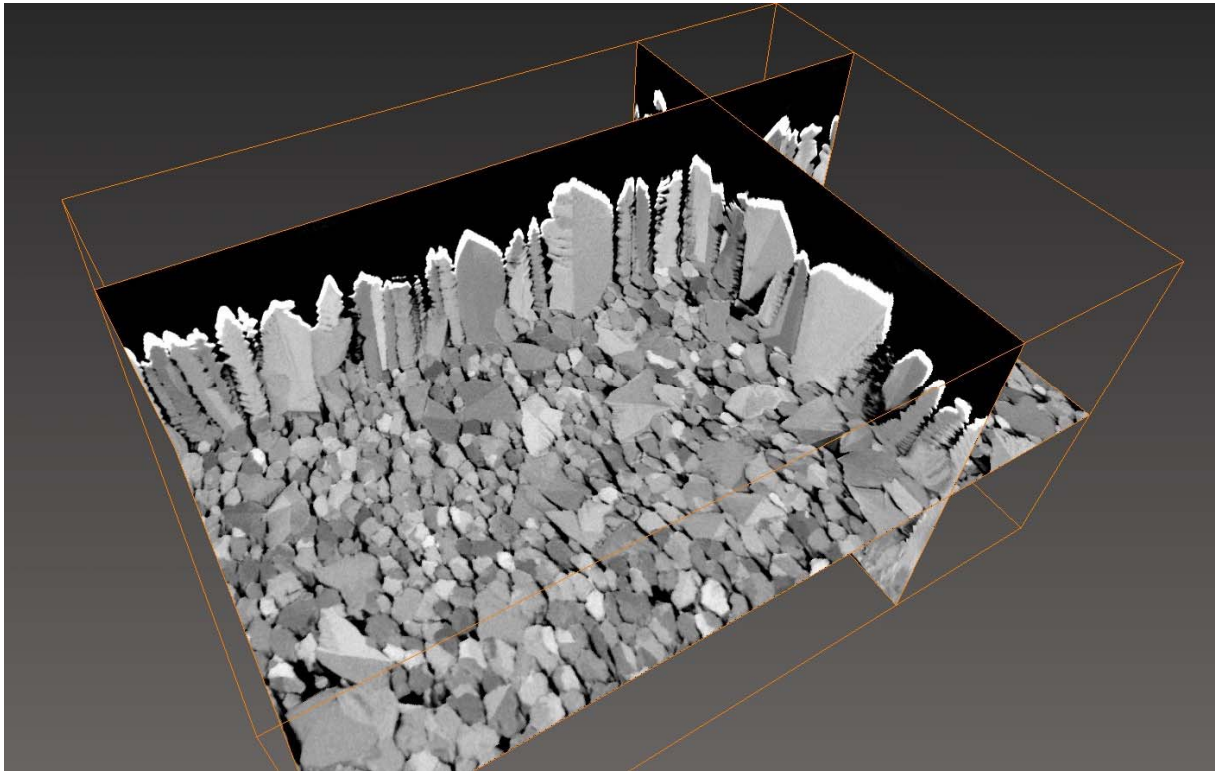2048x1536x1200 volume: 20x15x12um
10x10x10nm voxel

growth of ZnO films, photovoltaics

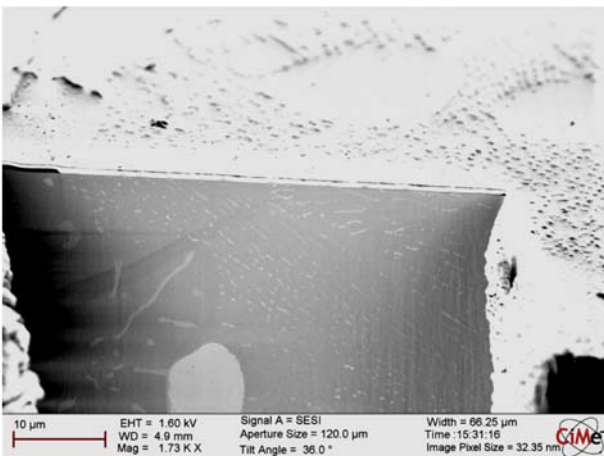*10x10x10nm voxel size, 2048x1536x2200 pixel/slices*
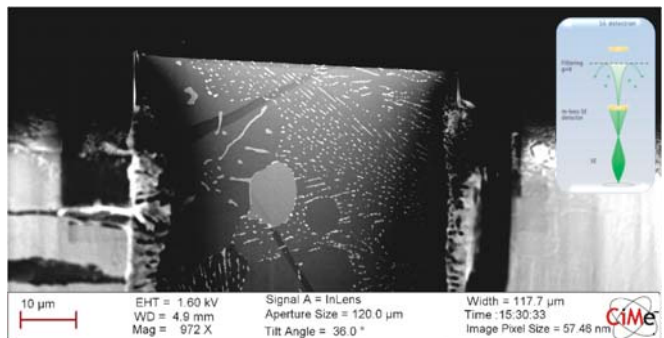
C. Balif, S. Nicolay, D. Alexander
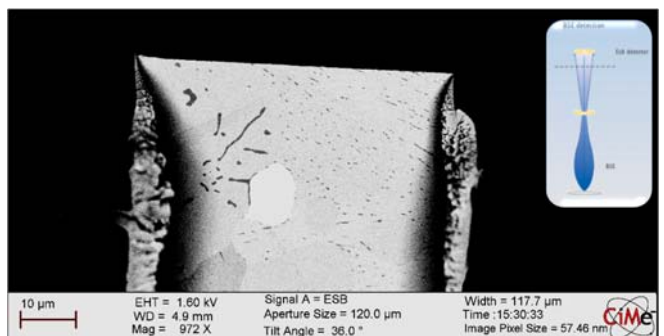
Pb-free solder:

"one detector is not enough"

M. Maleki,
EPFL-LMAF

InLens: SE low energy

ETD (SE classic)
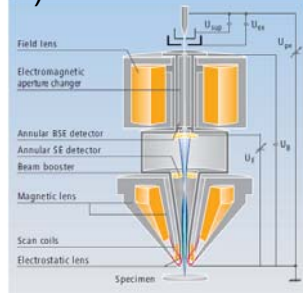
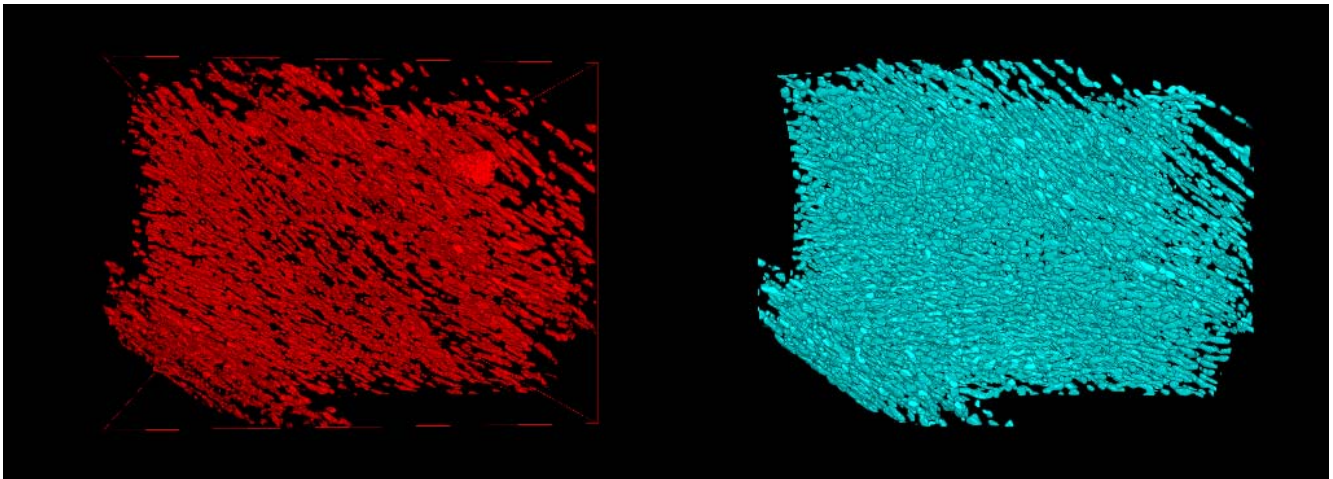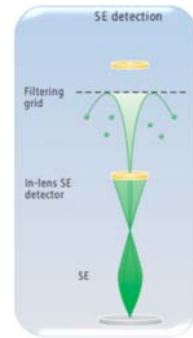

EsB: Energy selective
Backscattered
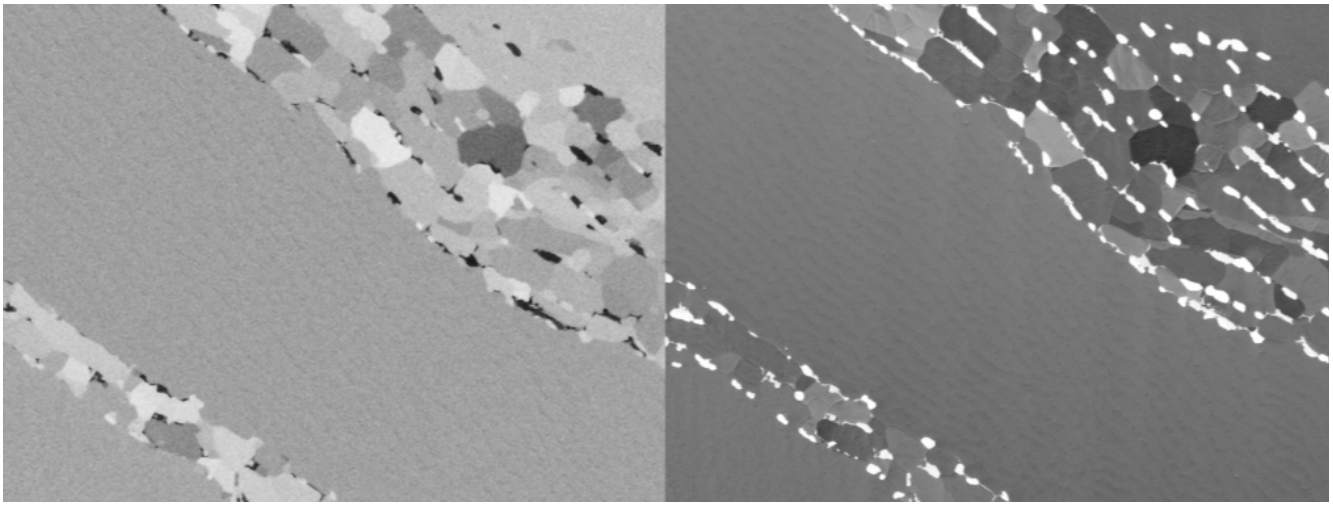
EsB
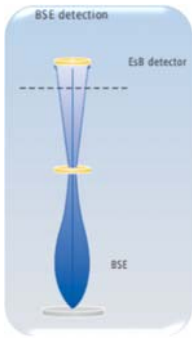
10x10x10nm voxel size, 2048x1536x2000 pixel/slices

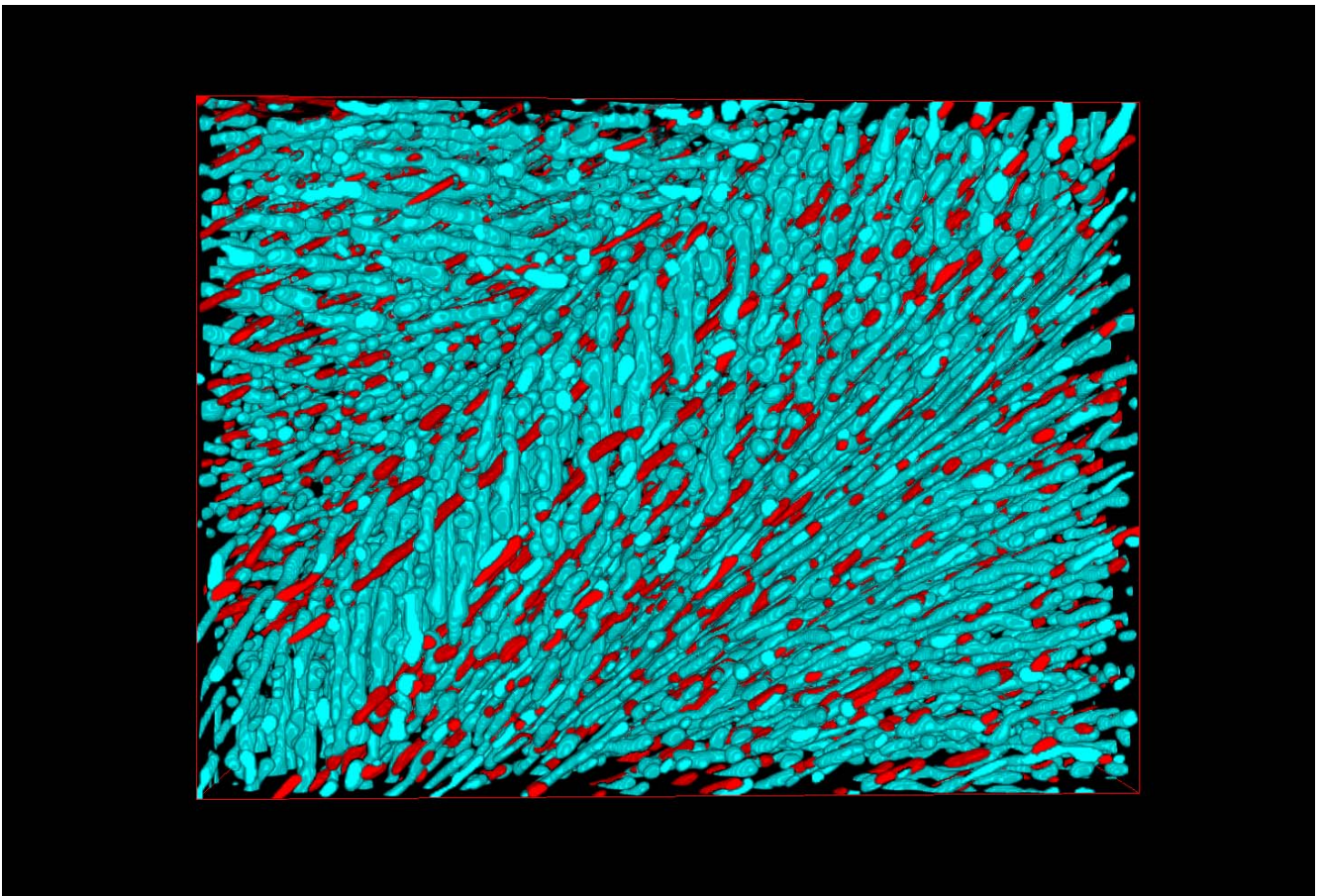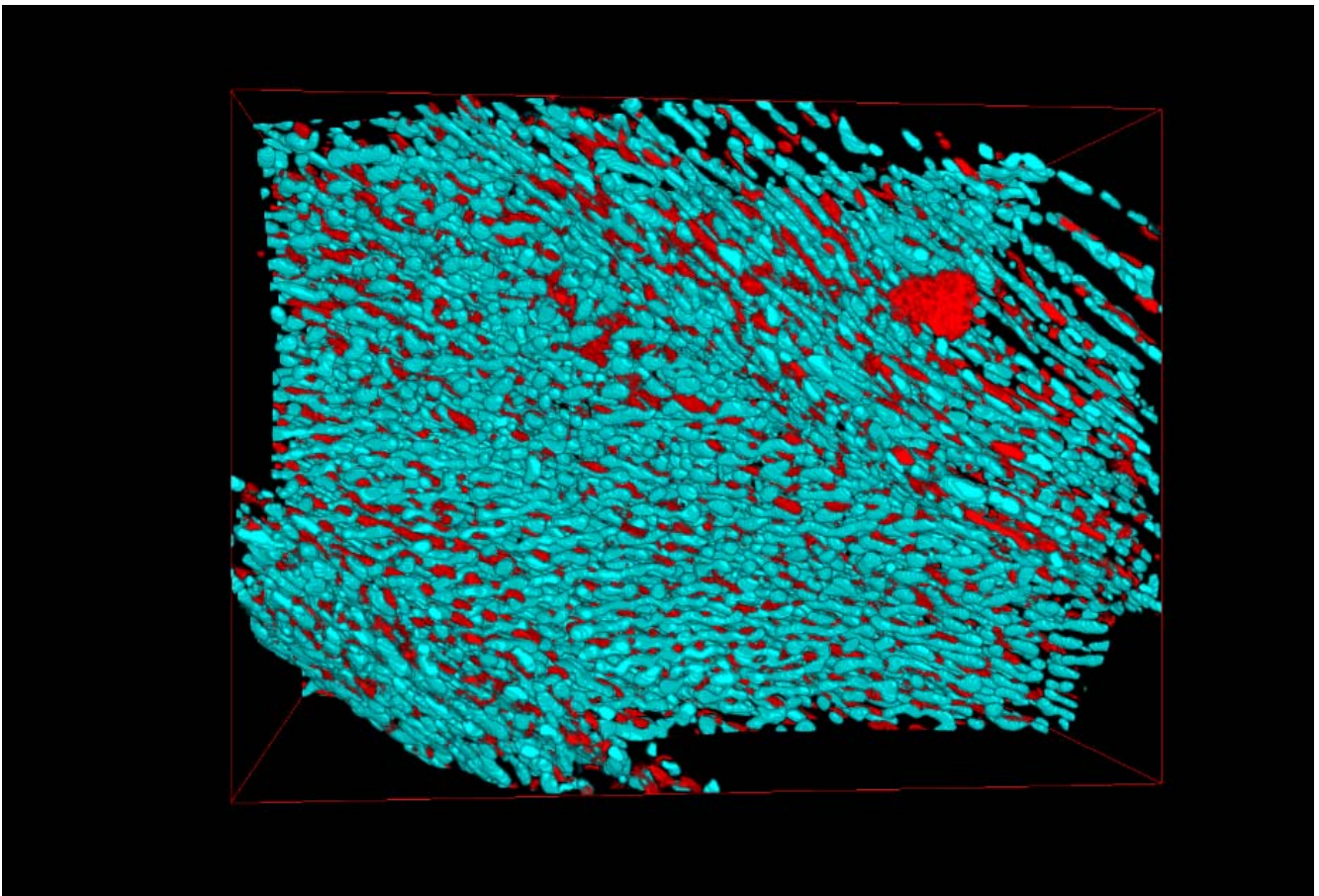2 images (3Mb) / slice ……   12Gb data

InLens SE

Phase 1
Dark in EsB image
White in SE-InLens

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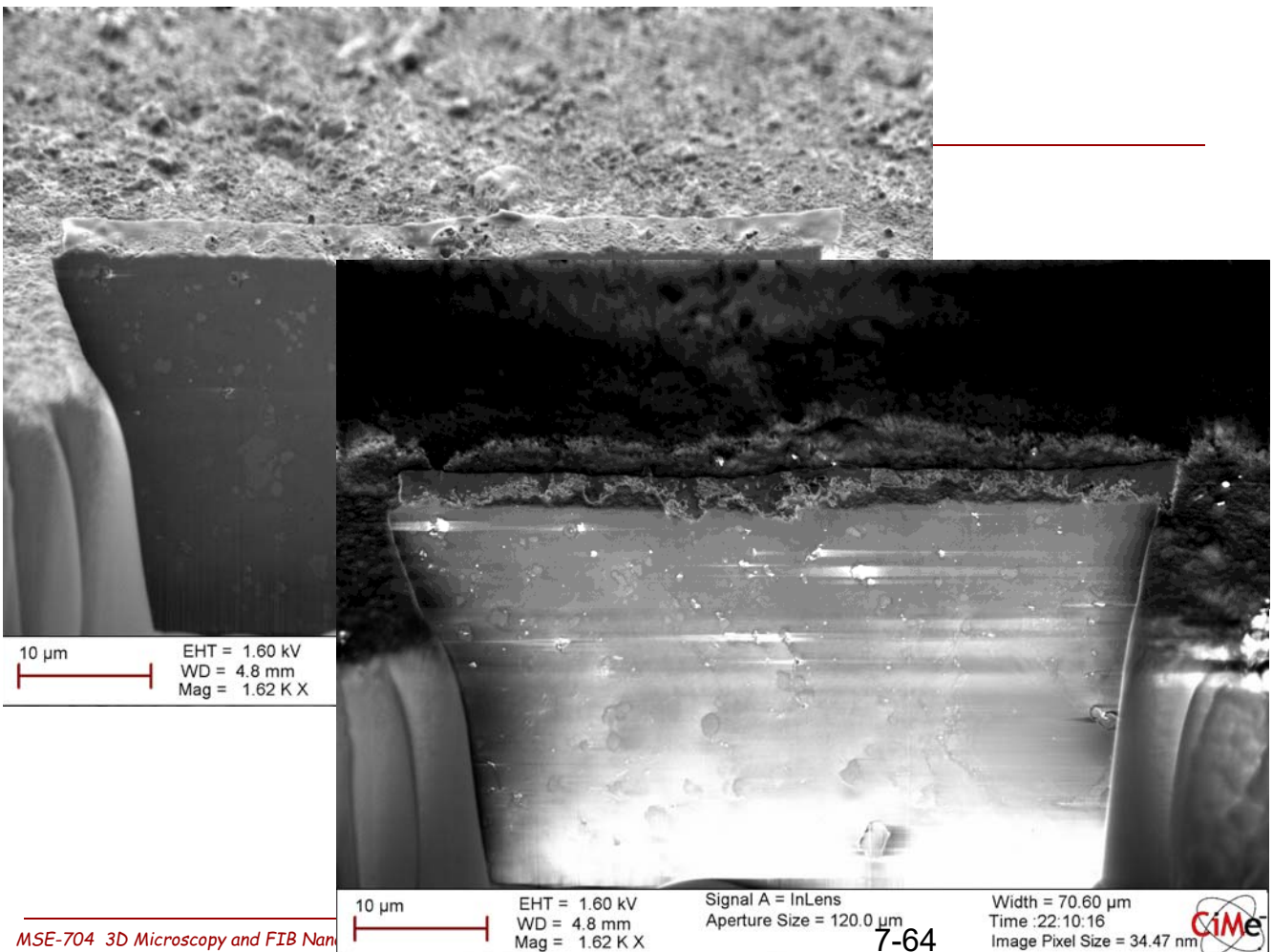White in SE-InLens - Dark in EsB image

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

10 µm	EHT = 1.60 kV	Signal A = SESI	Width = 173.3 µm	
	WD = 4.8 mm	Aperture Size = 120.0 µm	Time :22:11:23	
	Mag = 660 X		Image Pixel Size = 84.63 nm	

10 µm	EHT = 1.60 kV
	WD = 4.8 mm
	Mag = 1.62 K X

10 µm	EHT = 1.60 kV	Signal A = InLens	Width = 70.60 µm	
	WD = 4.8 mm	Aperture Size = 120.0 µm	Time :22:10:16	
	Mag = 1.62 K X		Image Pixel Size = 34.47 nm	
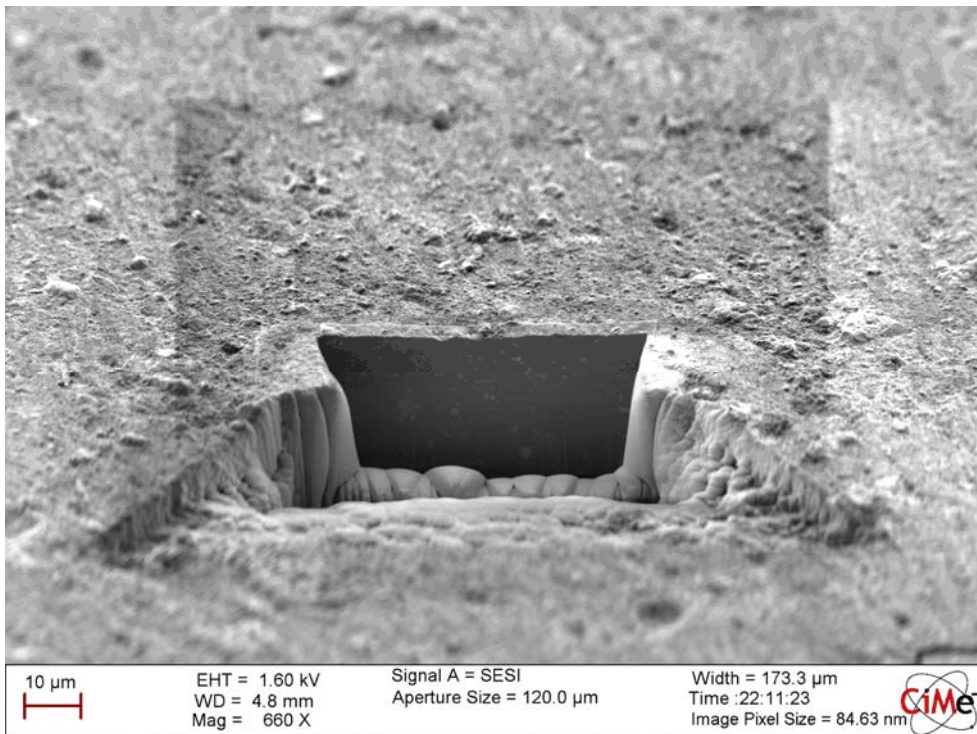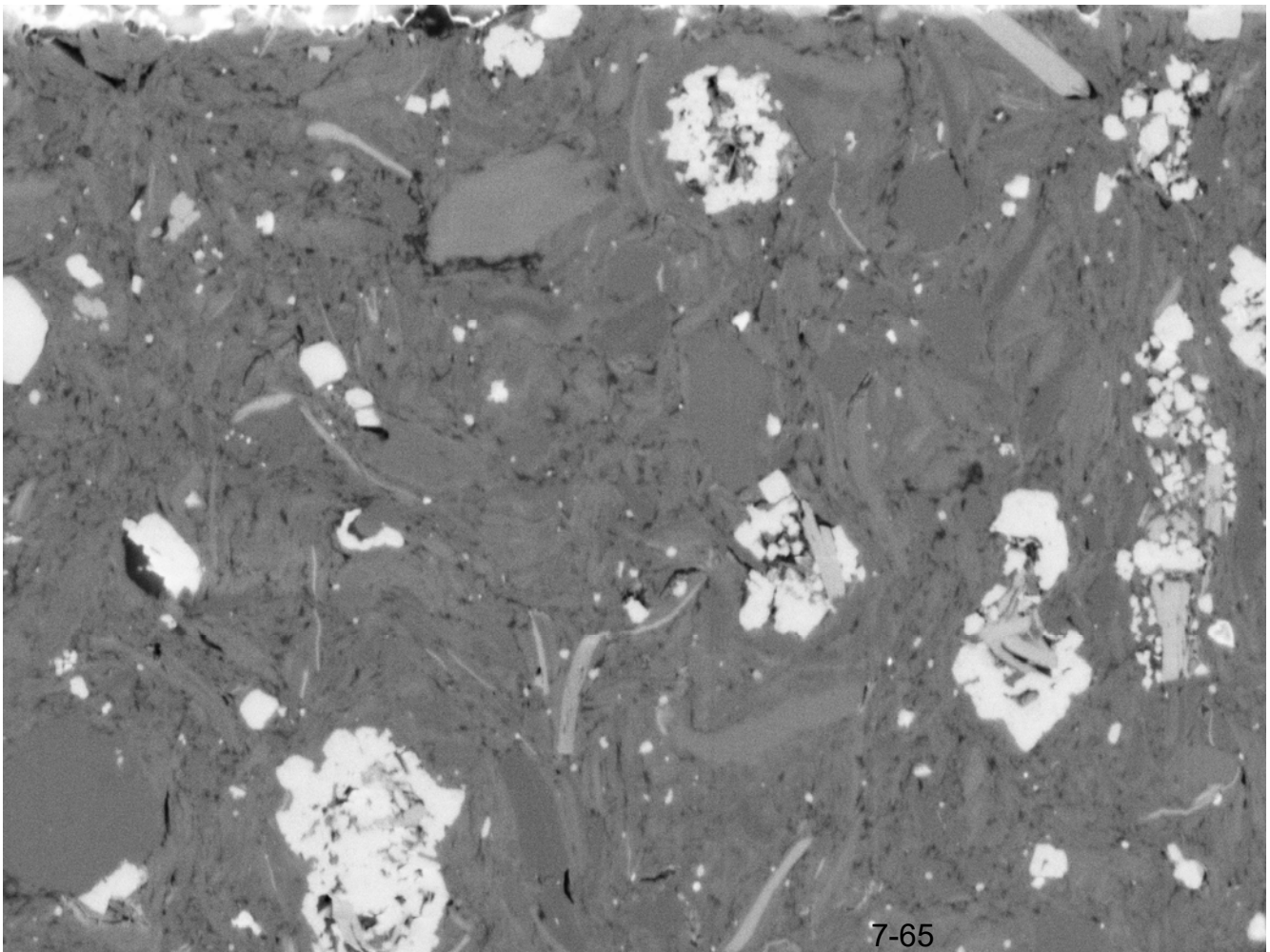
2048x1536x1510, voxel 10x10x10nm

Area of image data used for 3D reconstruction
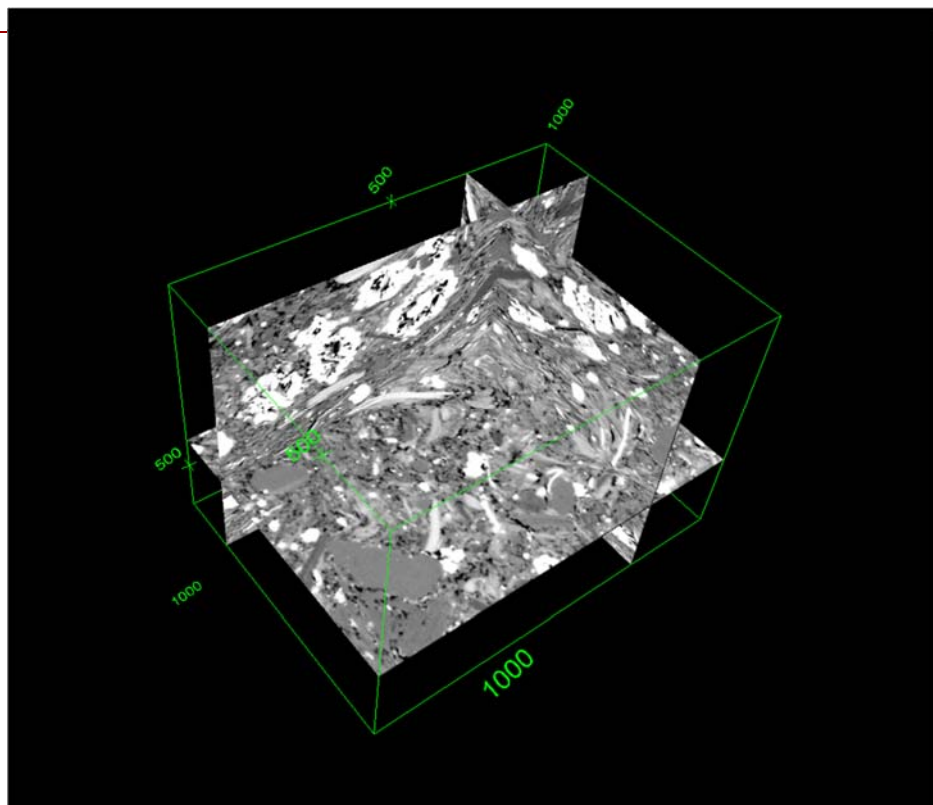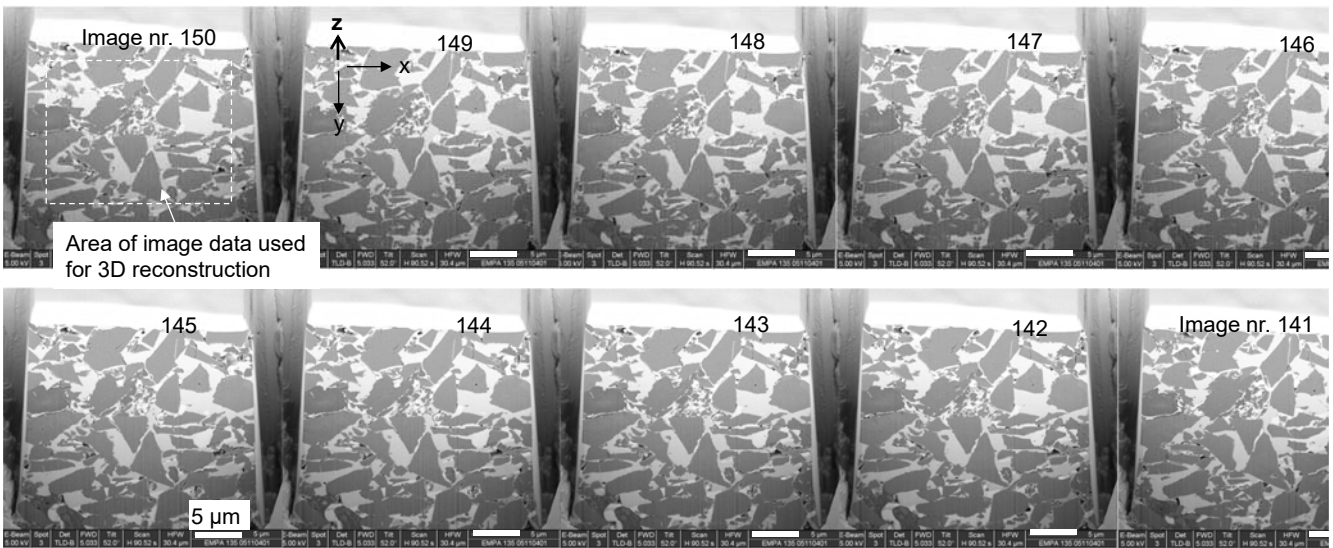
Quantitative microstructure analysis
→ Algorithms
→ object recognition
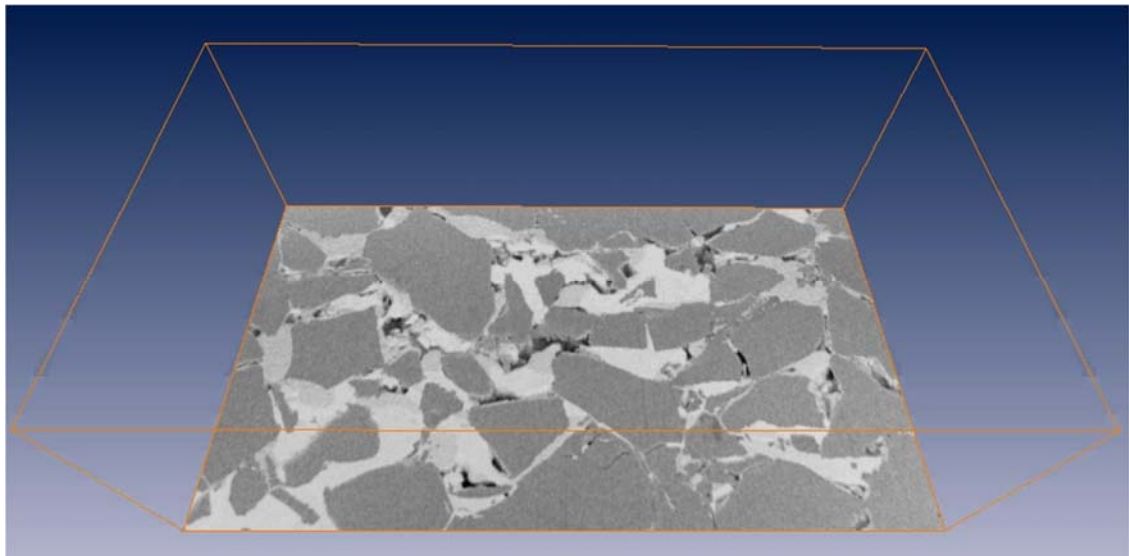→ stereological correction of boundary truncation
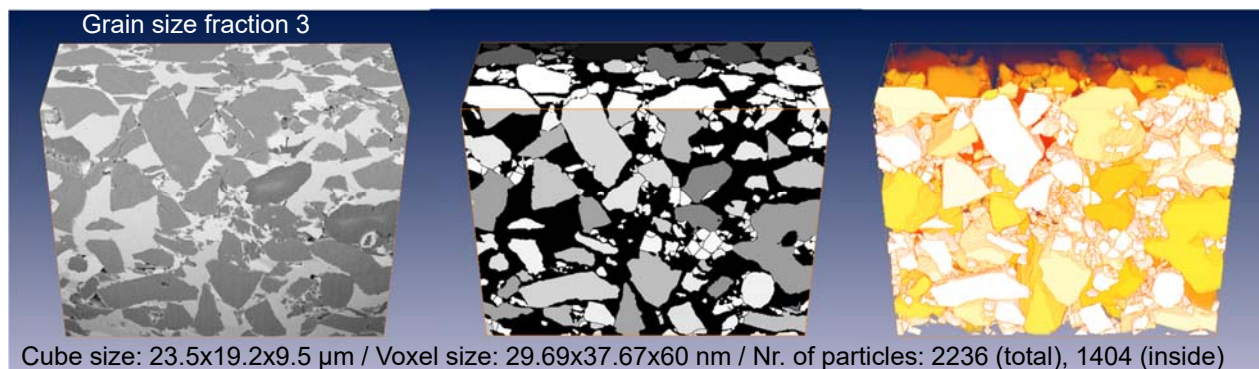→ extraction of statistical data (particle shape and size distribution)

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)
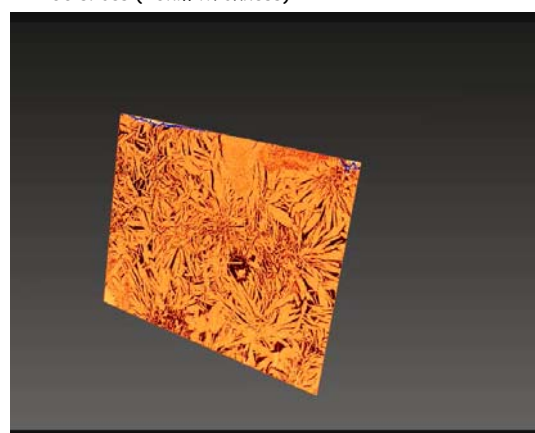
Quantitative microstructure analysis → Algorithms

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

CiMe

FIB-NT compared with other 3D-techniques
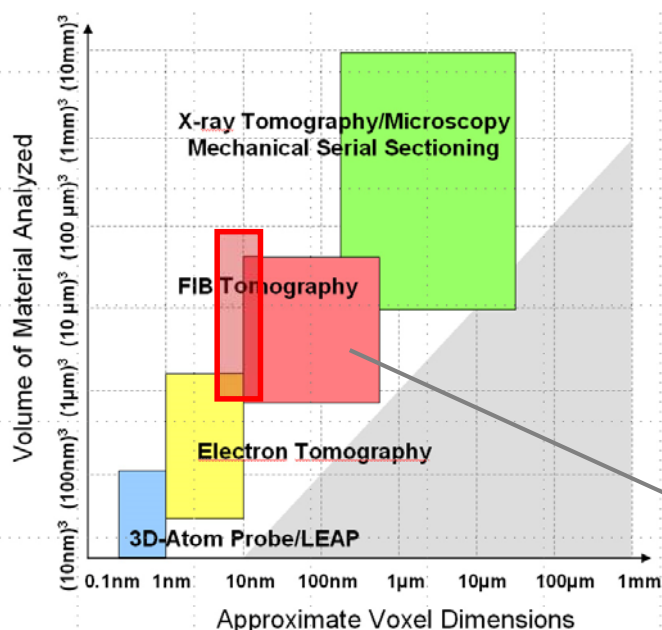
Ciment, A. Quennoz IMX-LMC
Materials contrast
1100 slices (20nm thickness)

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

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

CiMe