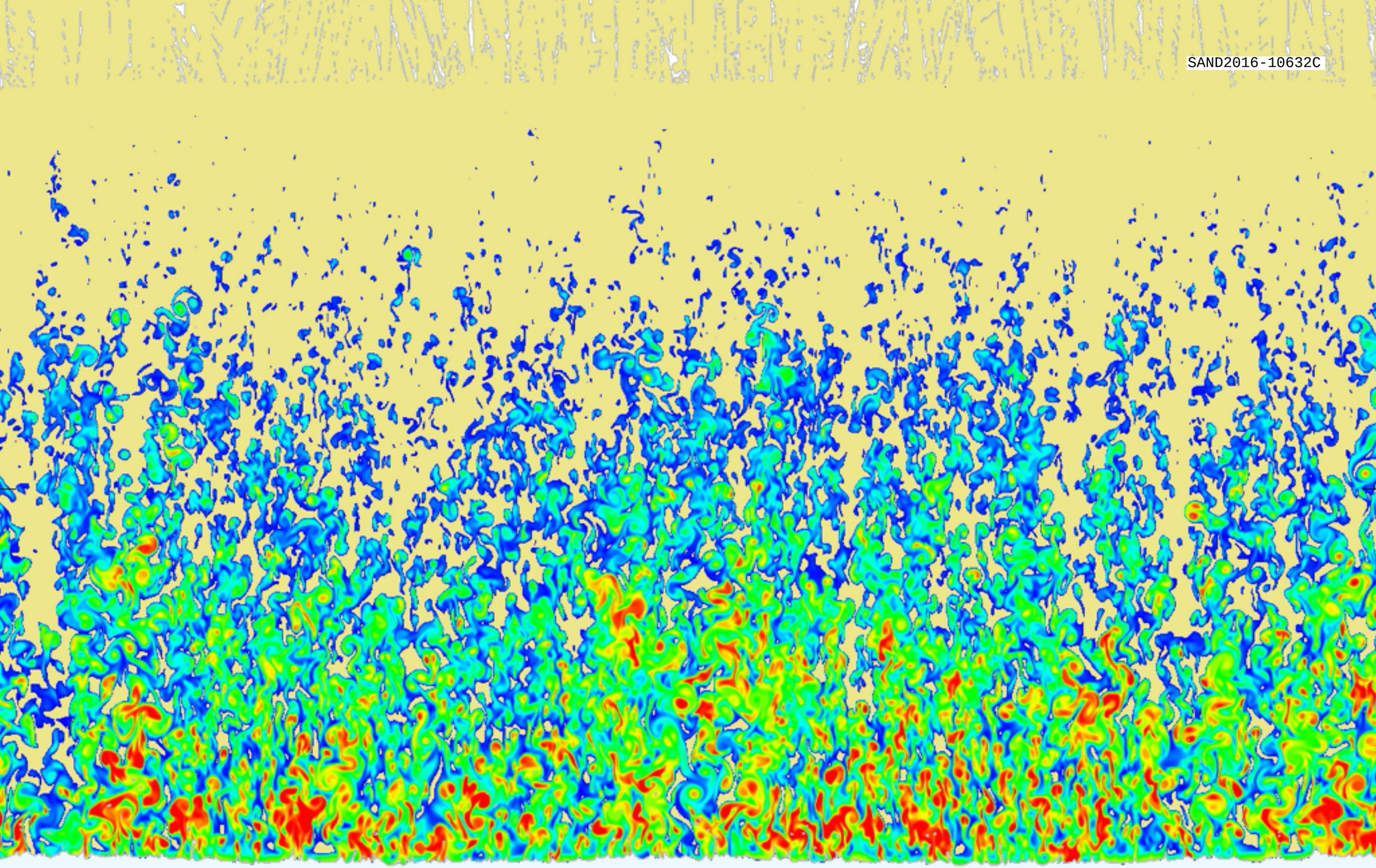
SAND2016-10632C



RYAN R. WIXOM

MICROSTRUCTURAL CHARACTERIZATION OF ENERGETIC MATERIALS

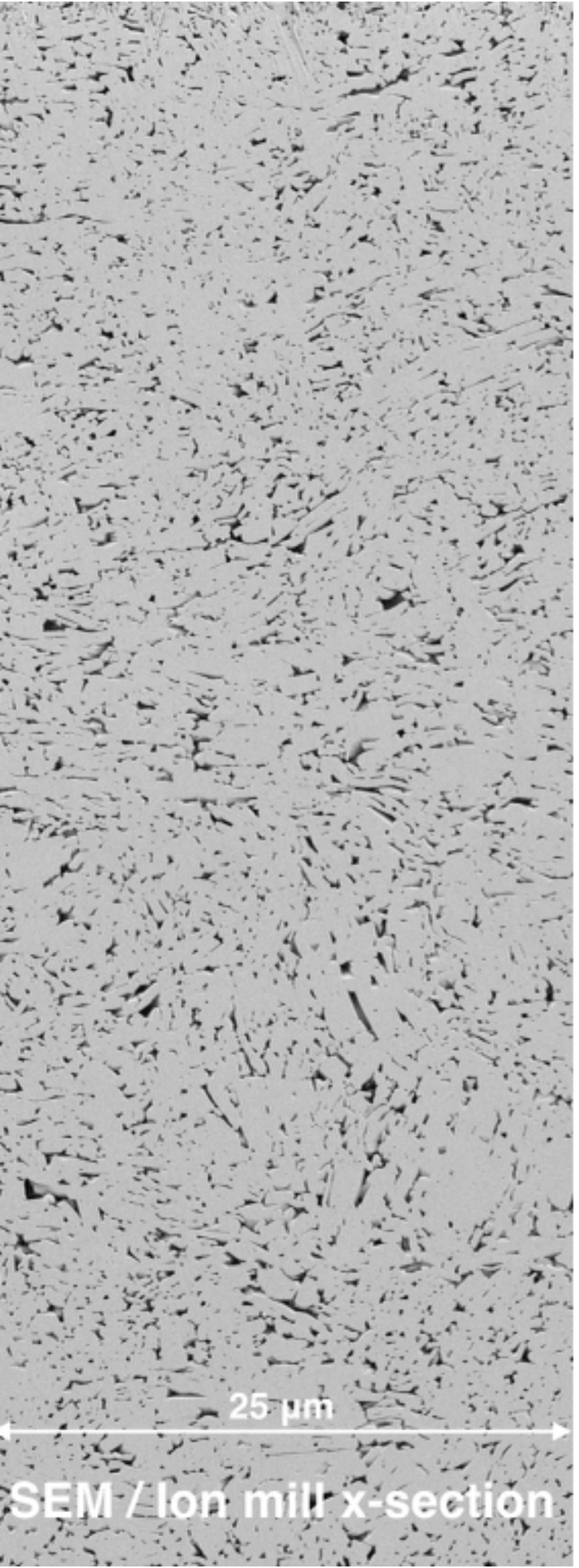
Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

PEOPLE

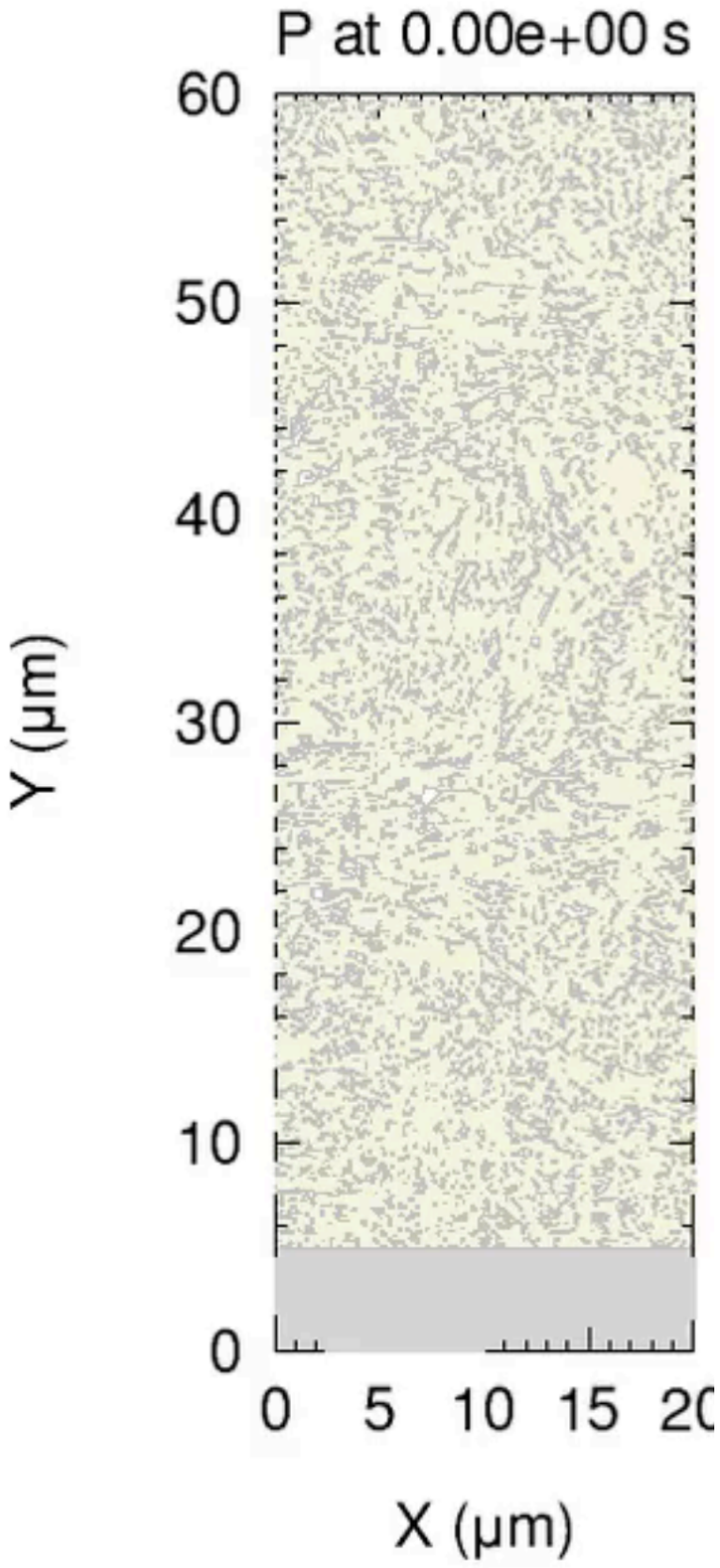
- Dave Jones, Eric Welle, Mel Baer, Alex Tappan
- Joe Michael, Micheal Rye
- **Barry Ritchey**, Cole Yarrington, Rob Knepper, David Damm
- Chris Molek (HMX and analysis)
- Todd Reedy (low density CL-20)
- Marcia Cooper, Graham Kosiba (RPI APCP characterization)

Questions / Topics of discussion:

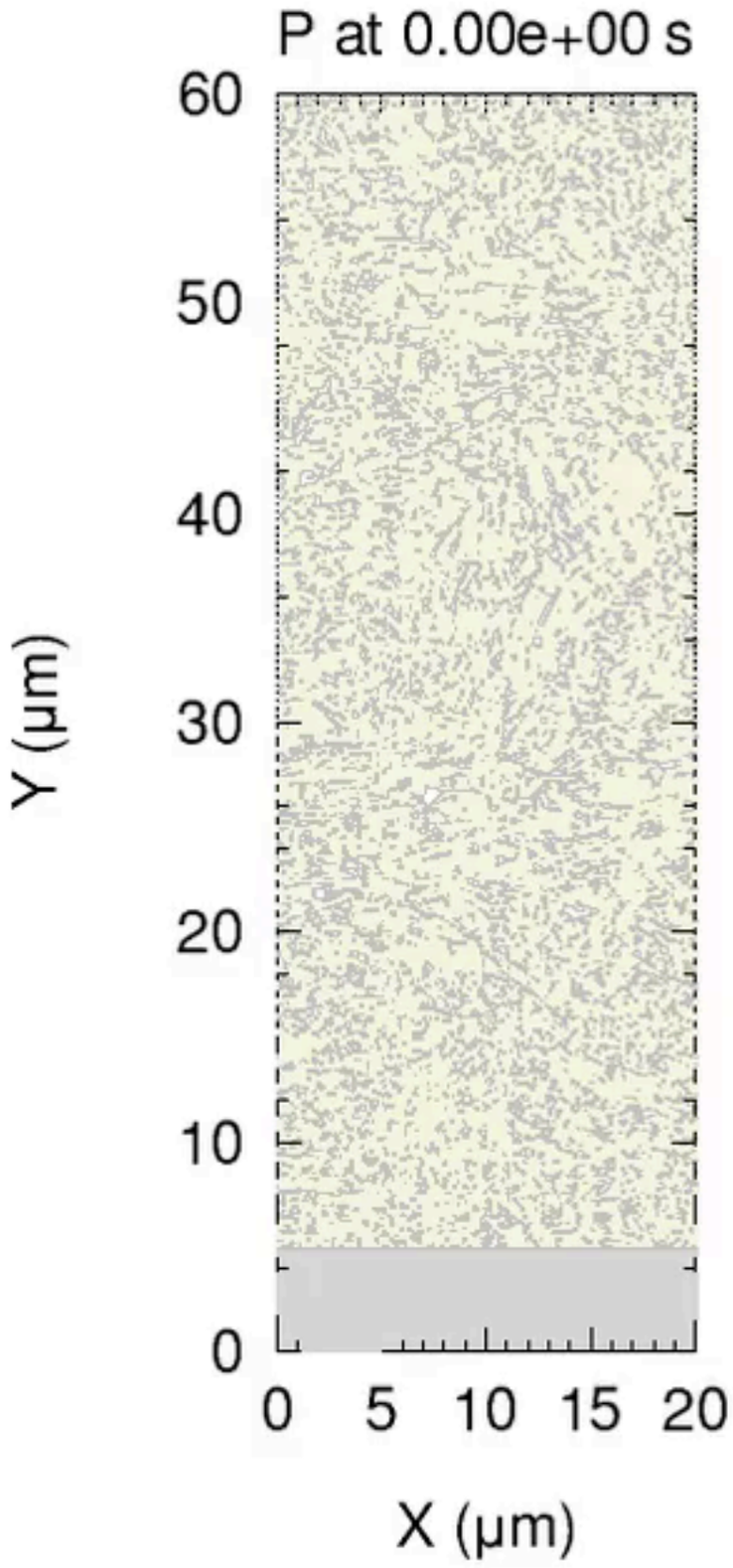
- What will the data be used for?
  - empirical correlations, simulation, parameterization, validation
- How should it be analyzed?
- When do you need 3D data? Will 2D do?
- Explicit microstructural or representative?
- What resolution and FOV is needed? What's possible?
- Which is better, Optical, SEM, FIB/SEM, xCT, AFM, XRD…

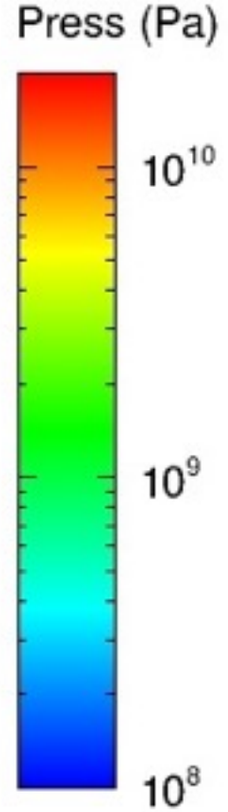


Flyer impact 1500 m/s



2500 m/s

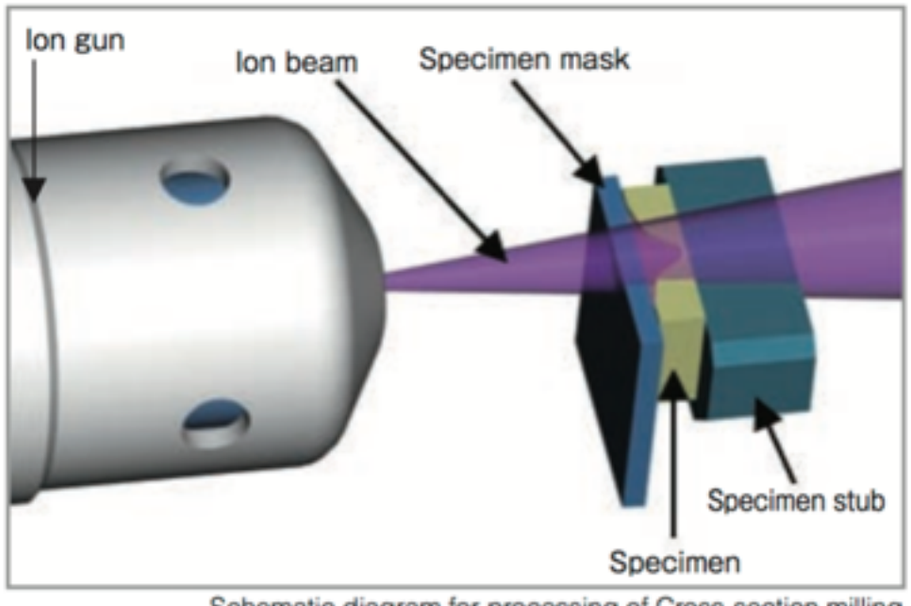




aluminum flyer impacting hexanitrostilbene

$

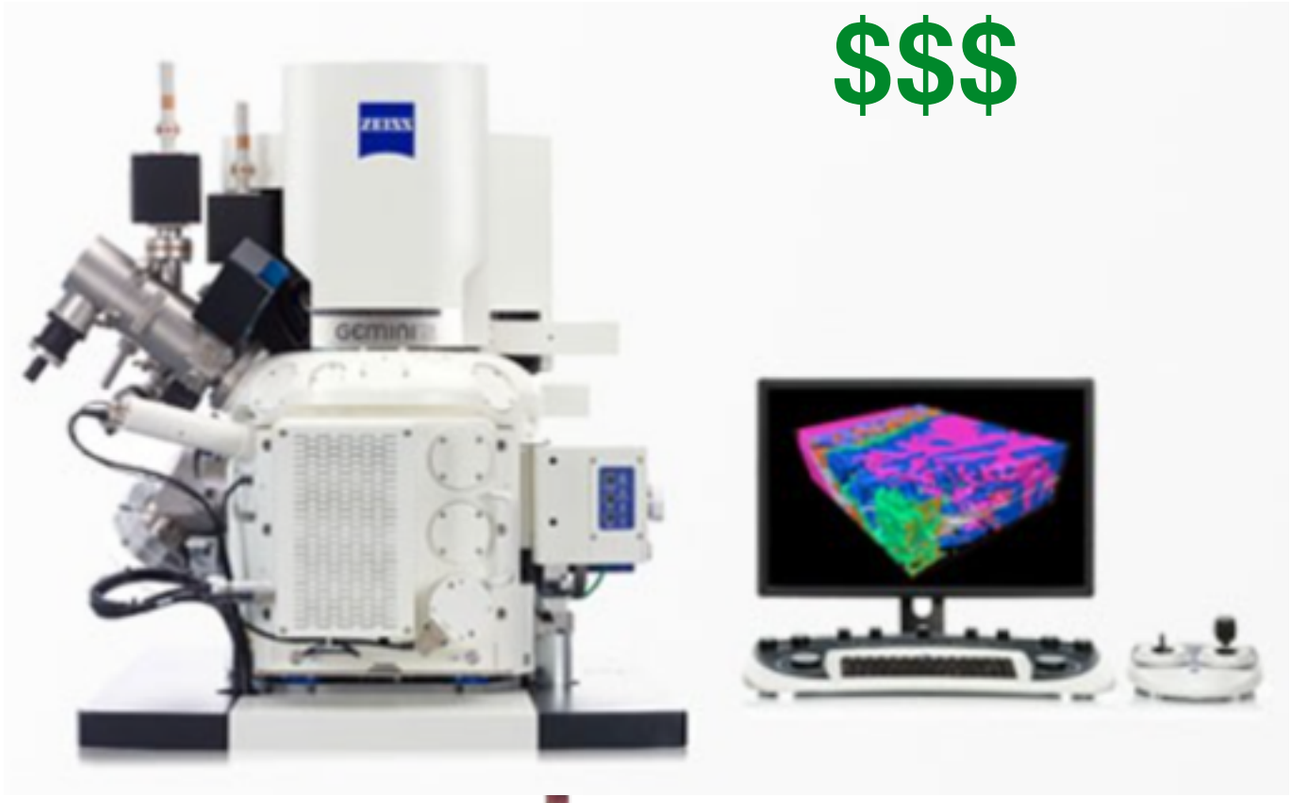
Ar ion-beam cross-sections Hitatchi IM4000



Schematic diagram for processing of Cross-section milling

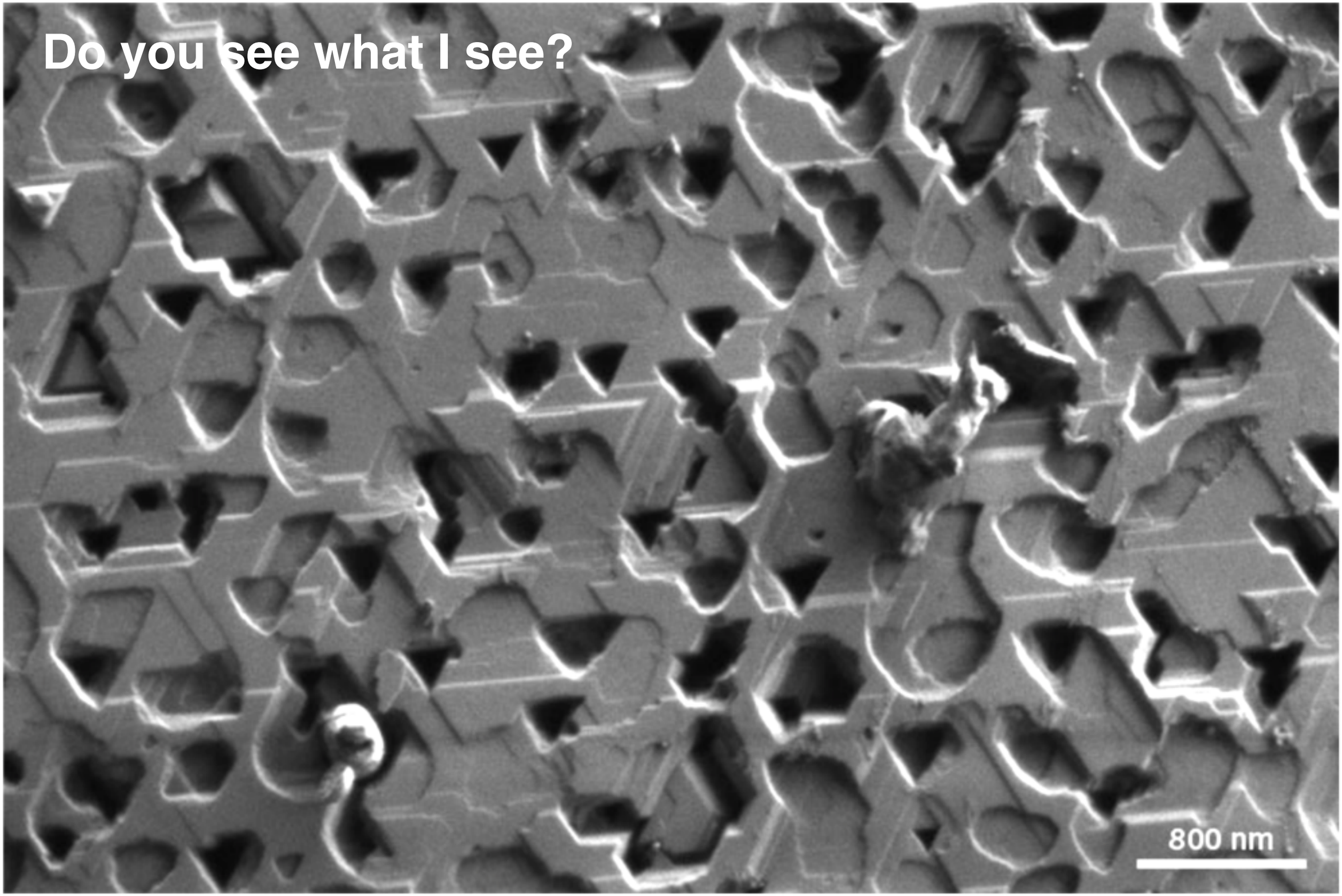
FIB/SEM Zeiss Cross-beam

$$$



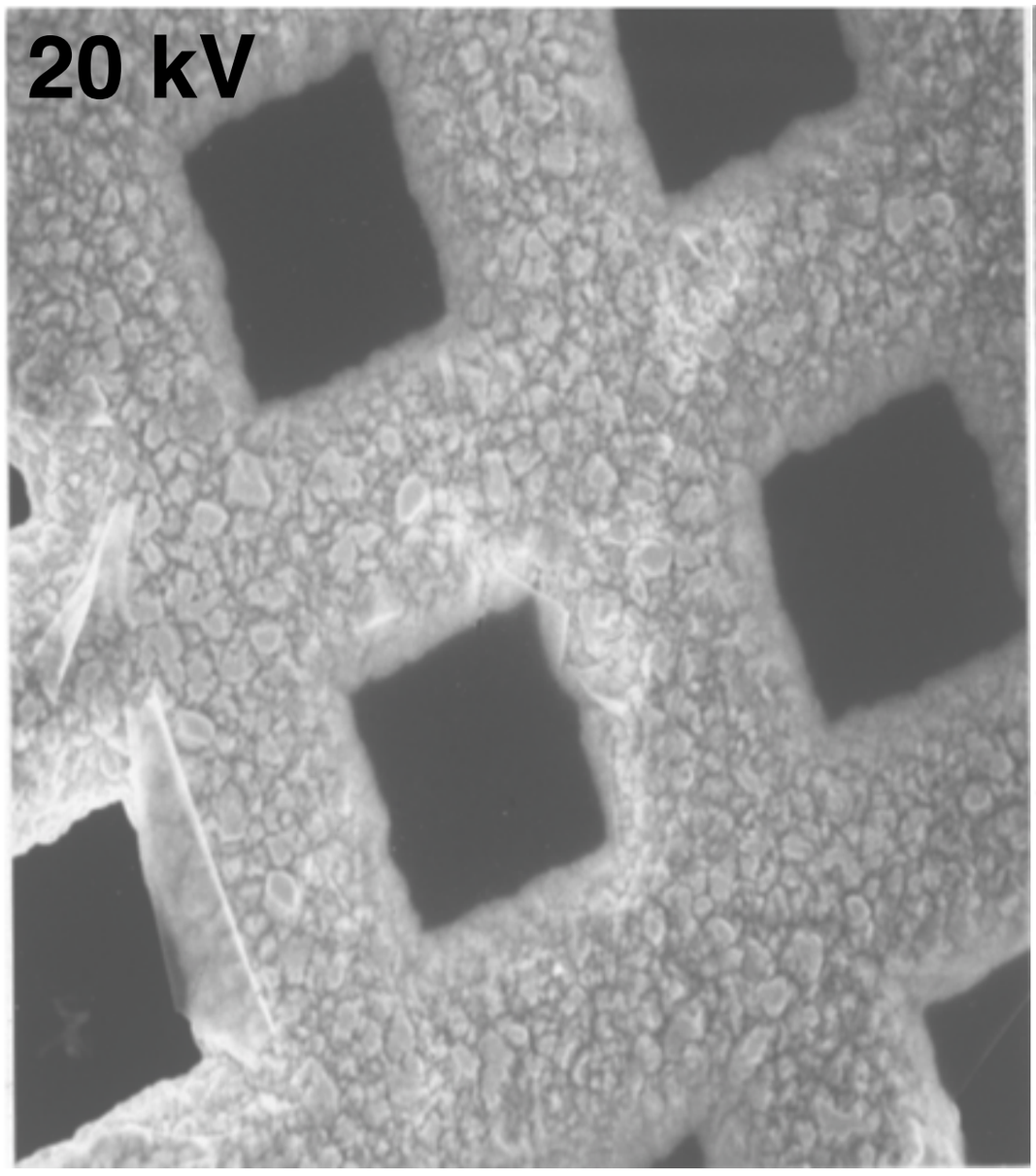
xCT Zeiss / Xradia Versa 520

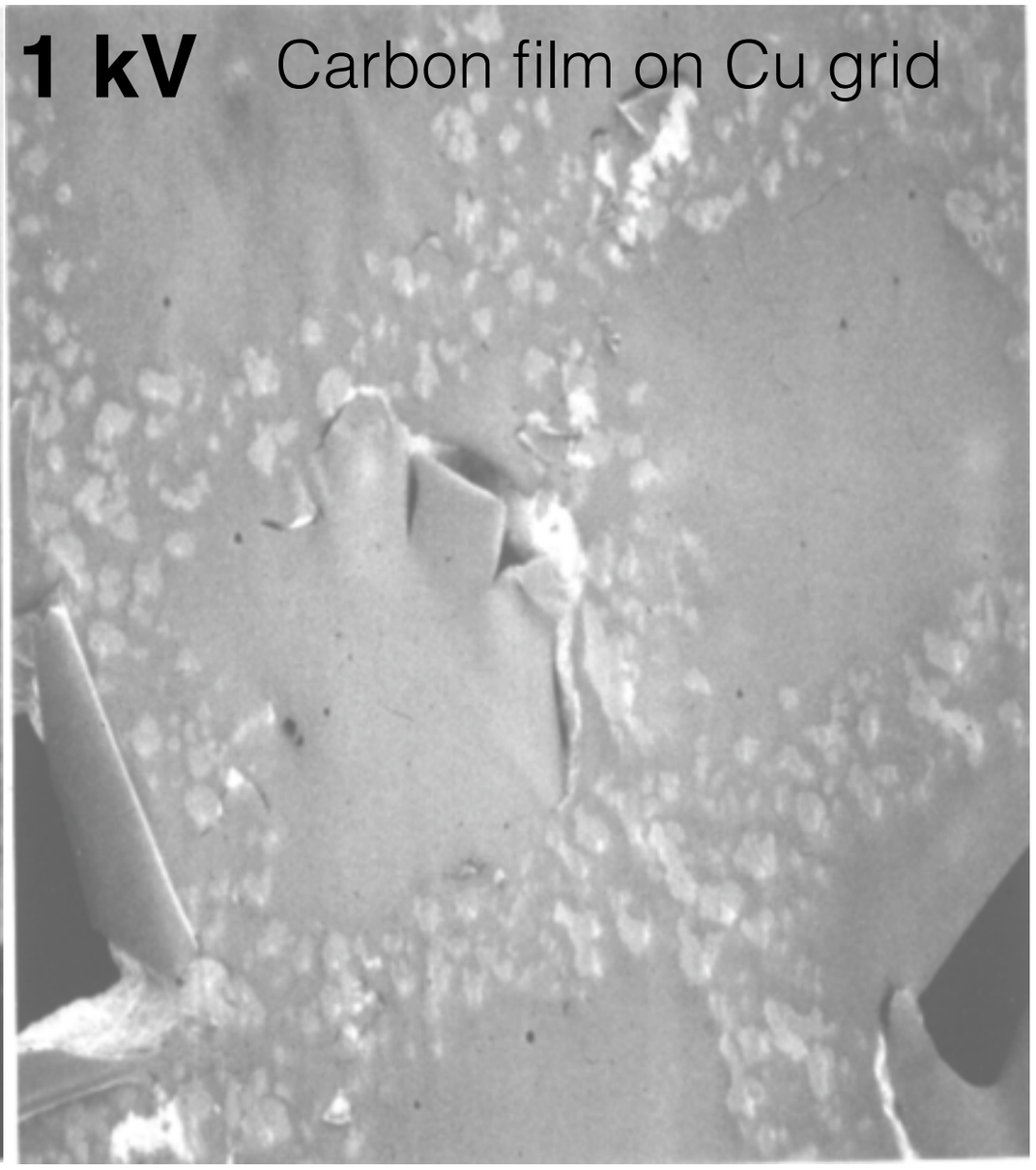
Do you see what I see?

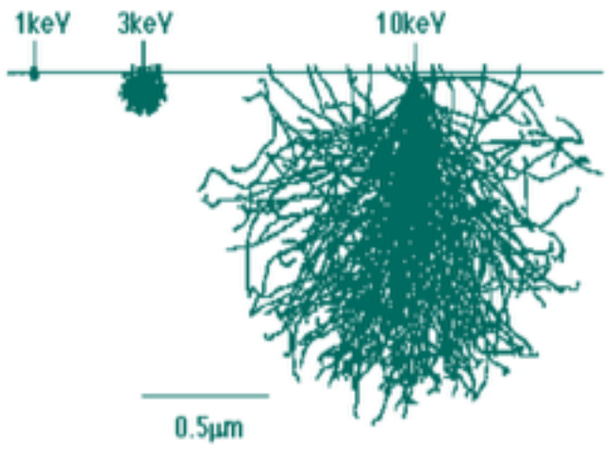


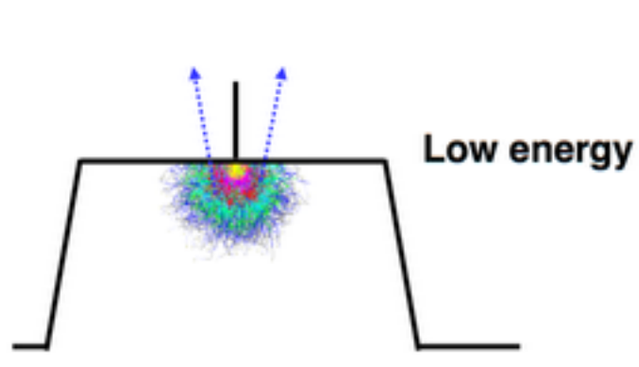
Etched silicon nanostructures at 50 V, no sample biasing. Imaged with GeminiSEM 500.
Sample: courtesy of A. Charai, Aix Marseille University, France.

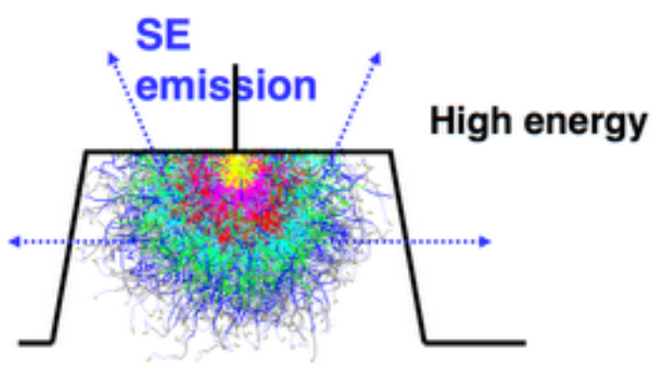
Be nice to your sample. Less voltage, please.



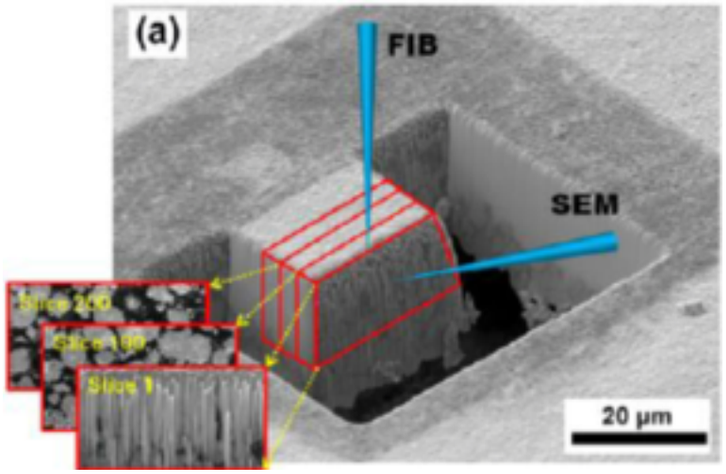


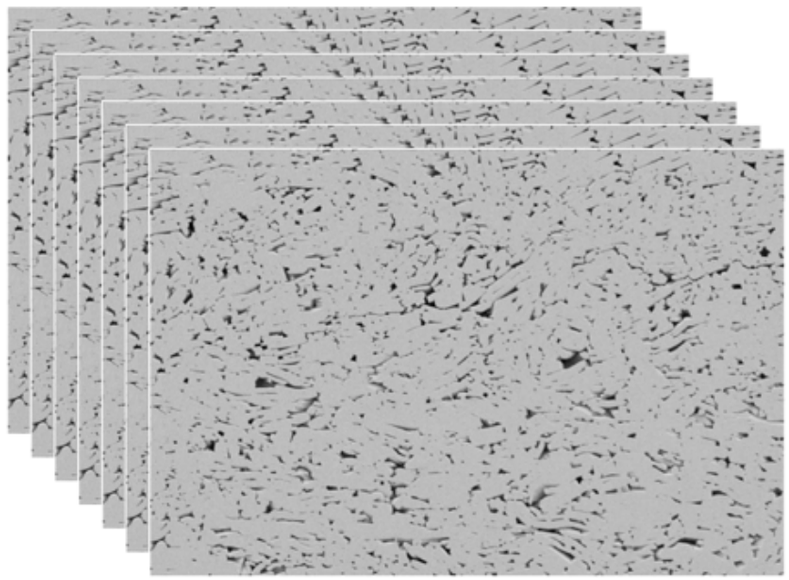


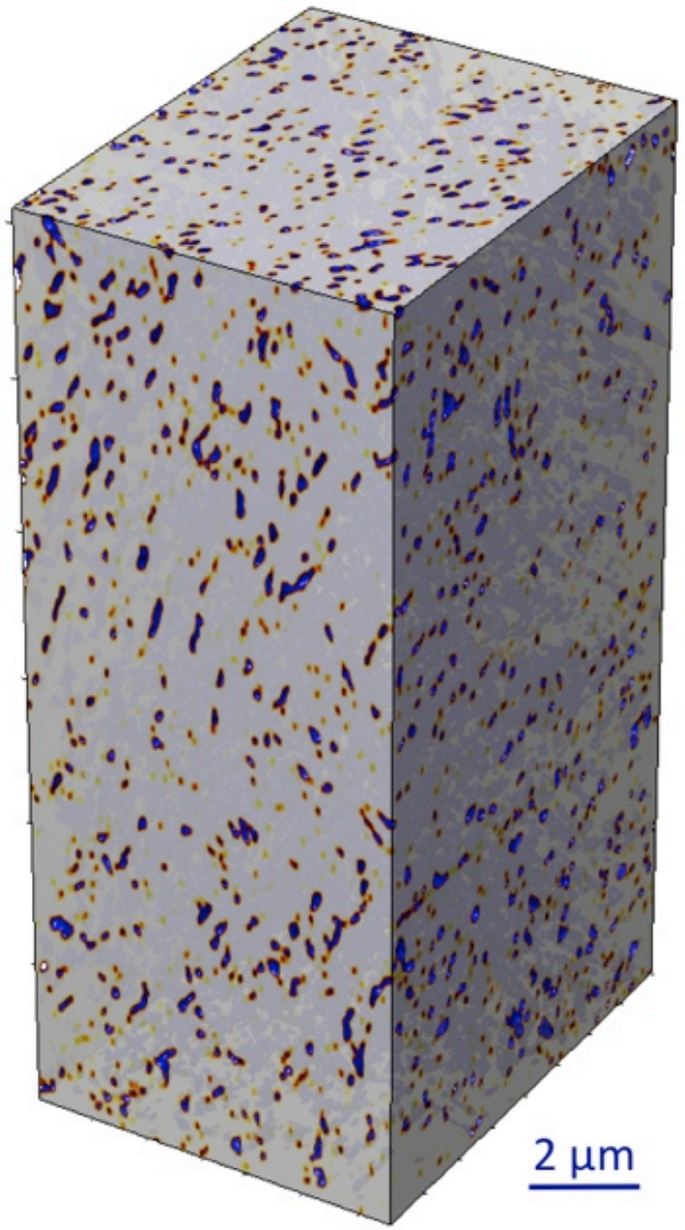


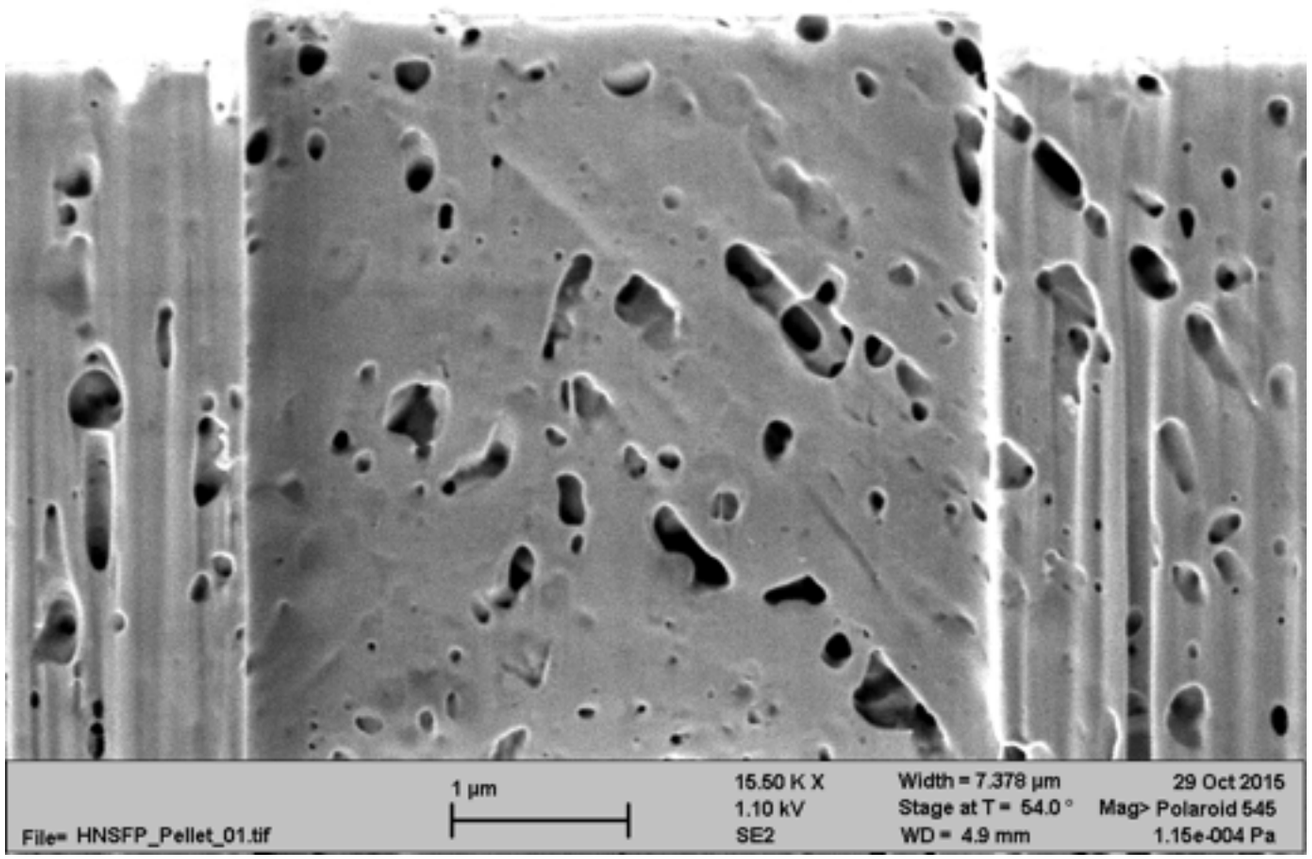


FIB/SEM Nanotomography

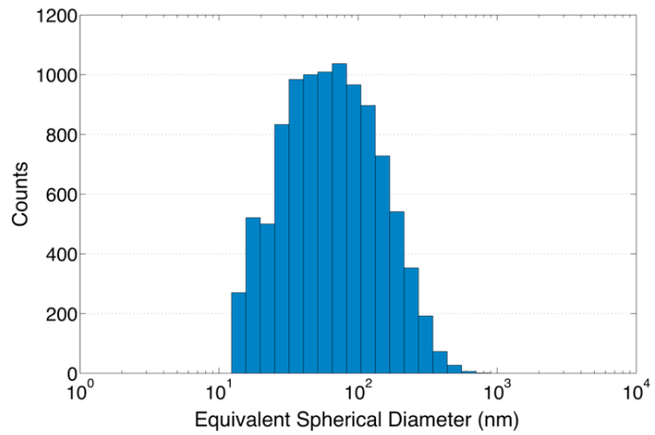








80,000 pores, mean diameter 86.3 nm



Wixom et al. J. Mater. Res. 25 1362 (2010).

Ion beam cross-sectioning

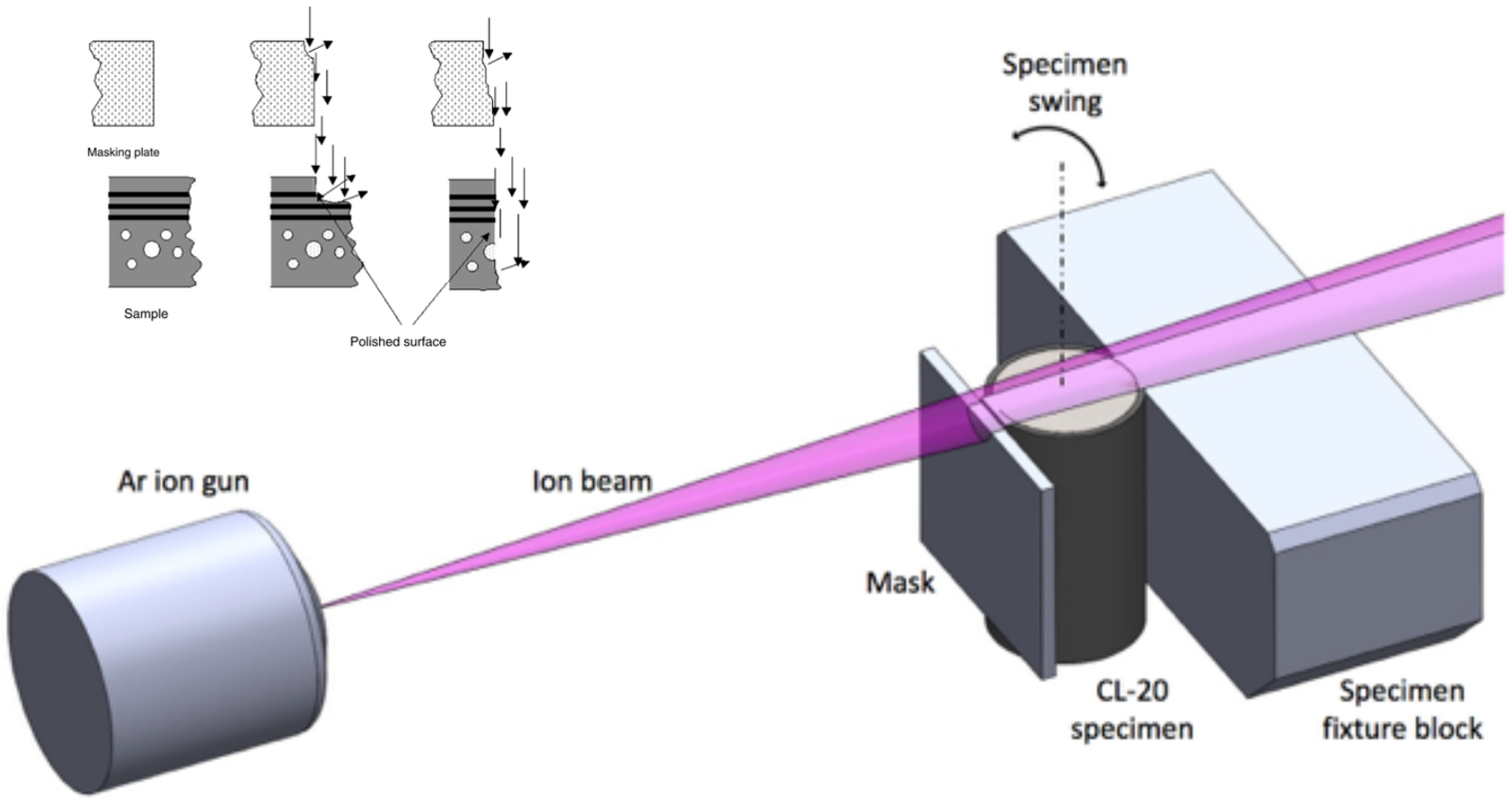


Figure 2-4. Schematic of cross-sectional ion milling.

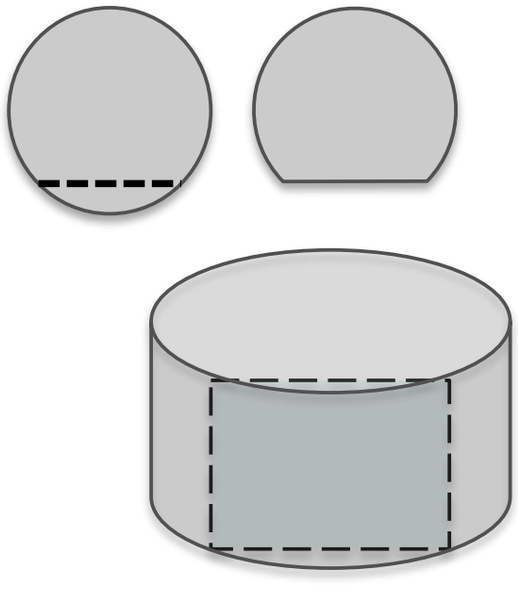
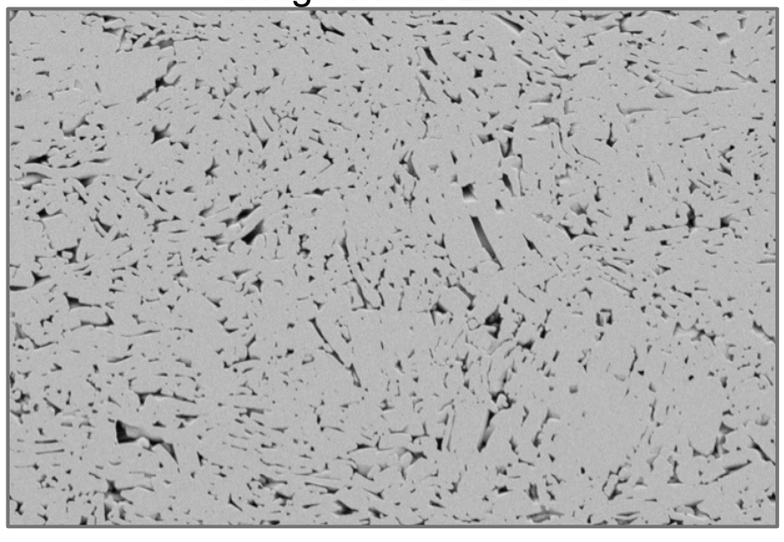


Image width: 25 um



HNS-FP, 90% TMD

CL-20: 50% 70% 90% TMD

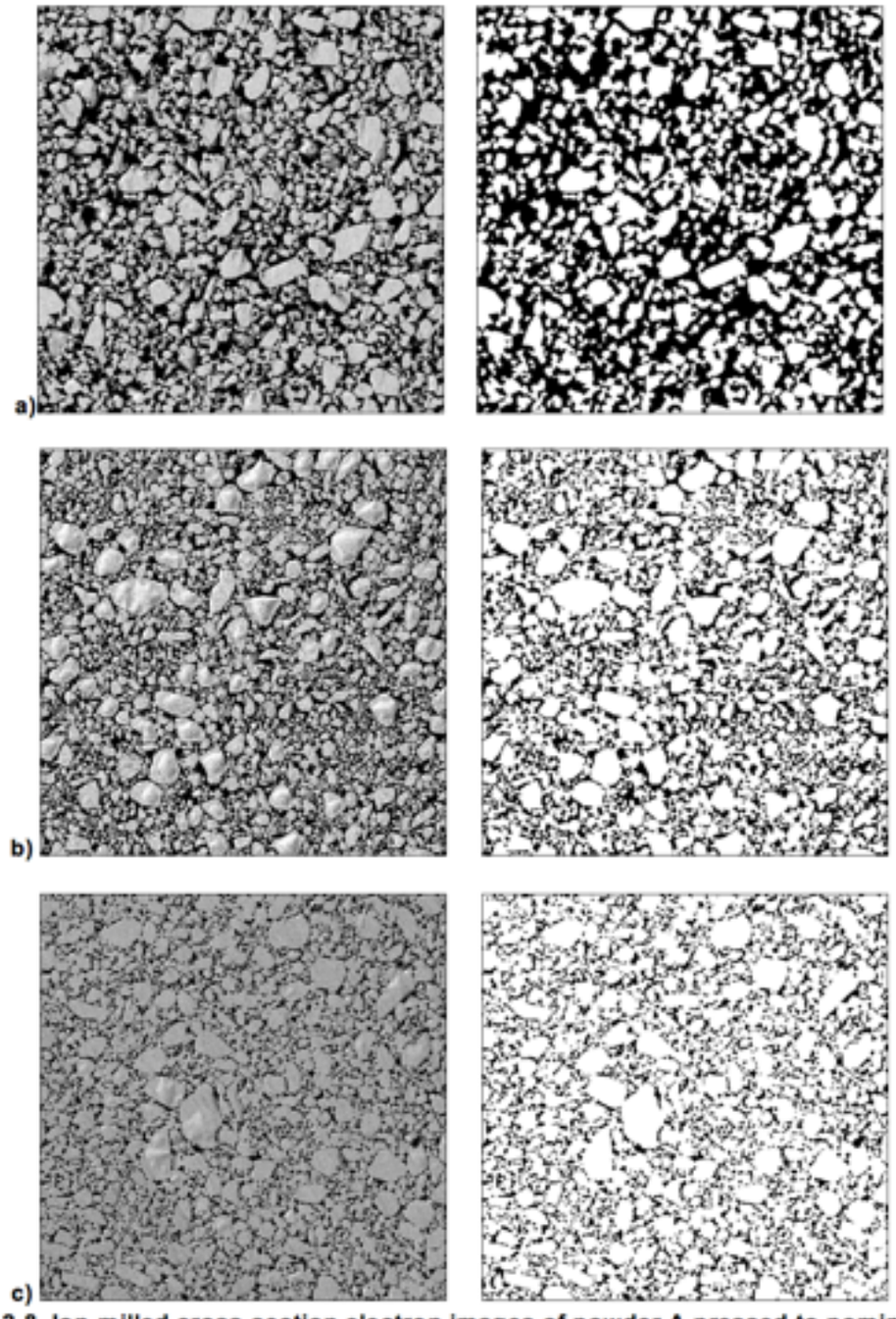
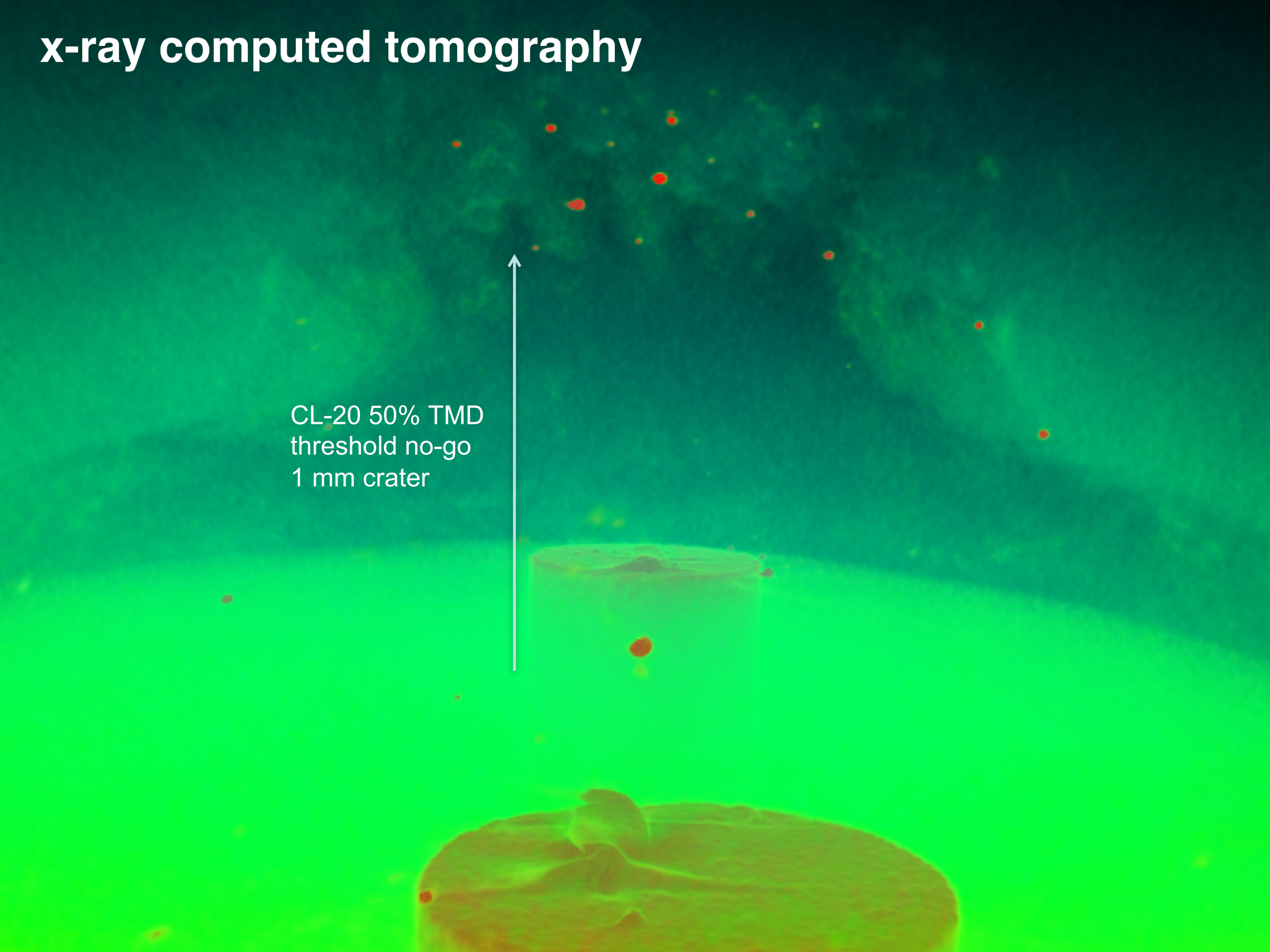
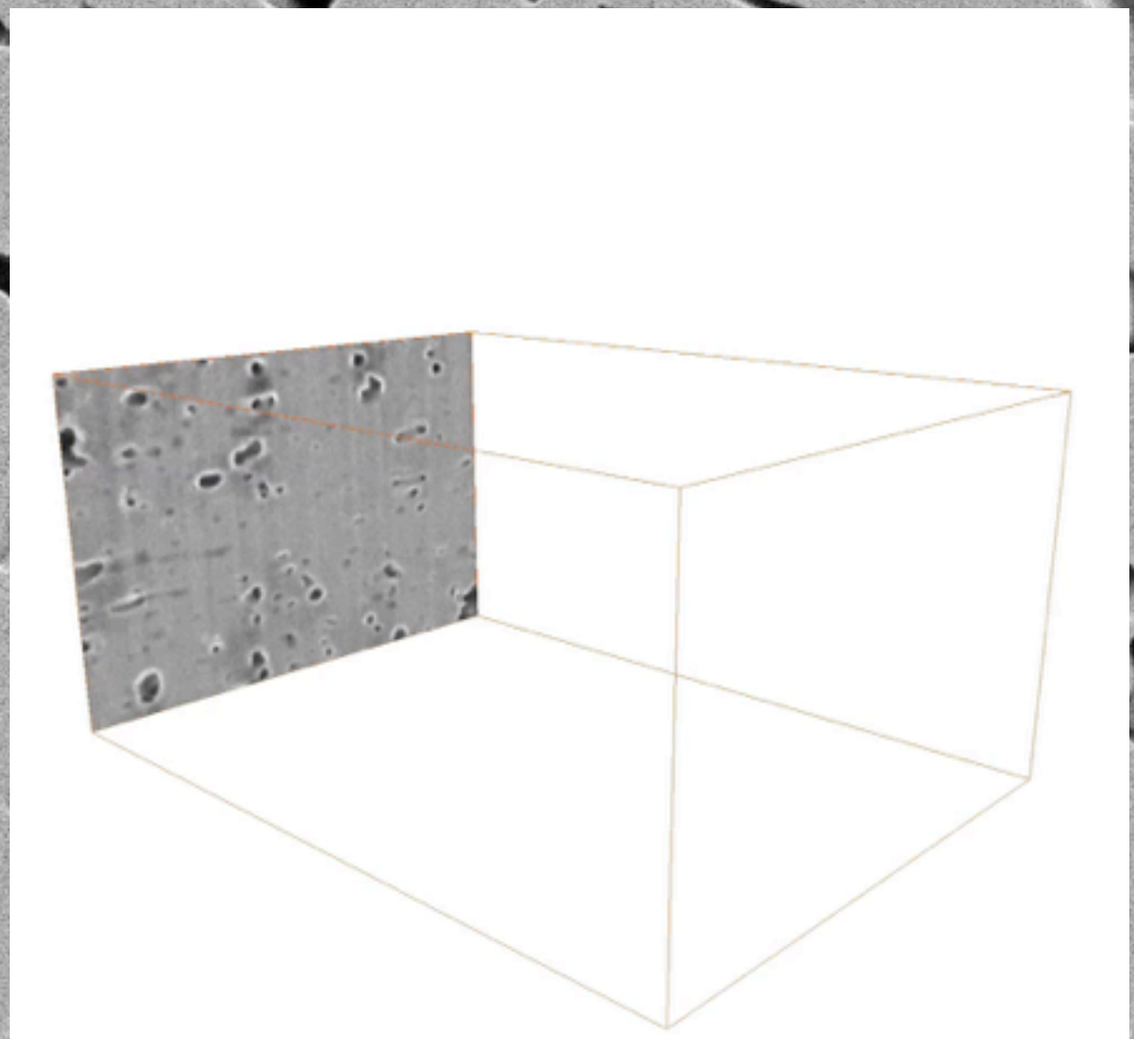


Figure 2-8. Ion-milled cross-section electron images of powder A pressed to nominally a) 50% TMD, b) 70% TMD, and c) 90% TMD. The image in right column is a segmented binary representation of the micrograph shown in the left column. Each image is 50 µm by 50 µm.

x-ray computed tomography



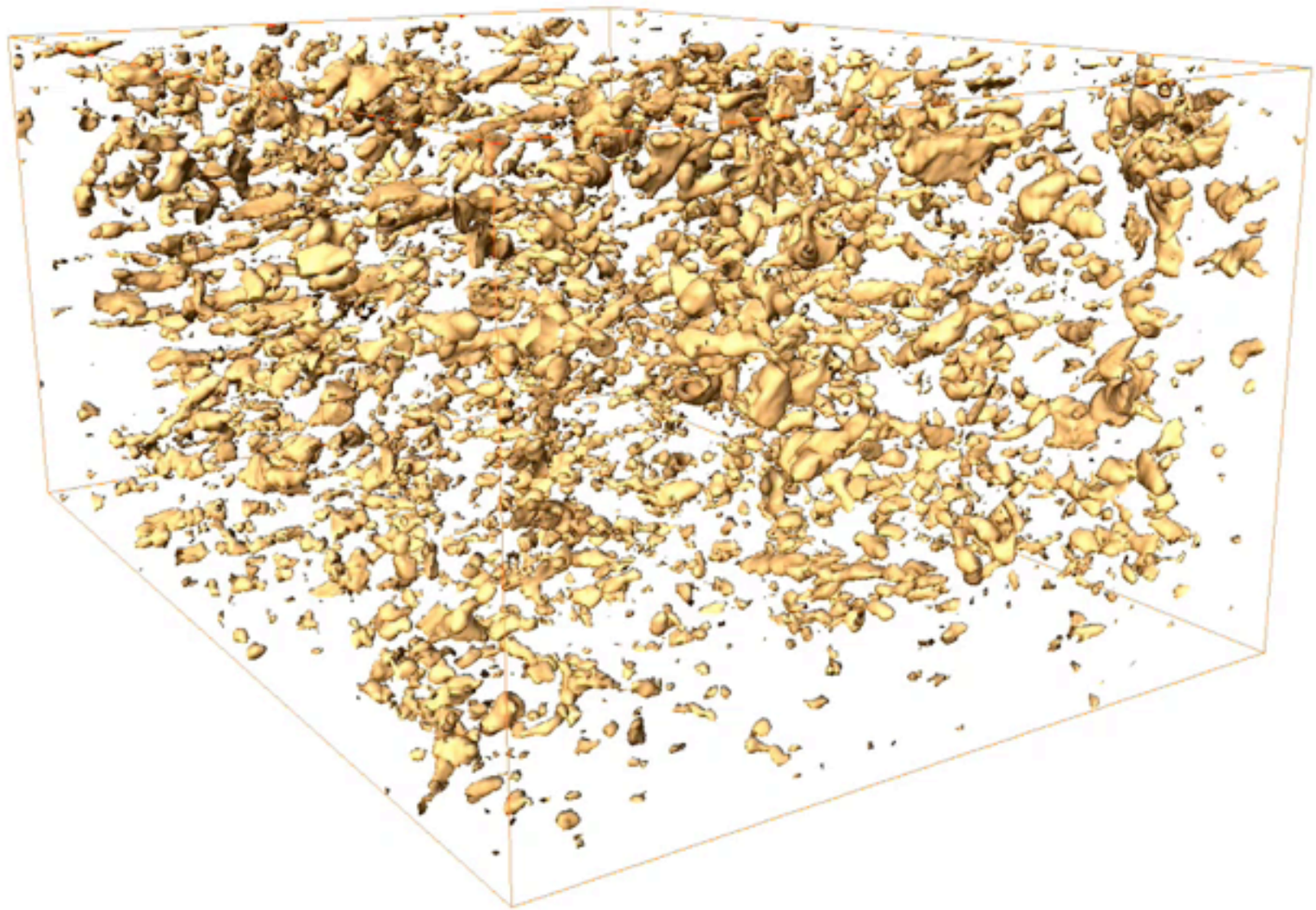


3D, but…
Not optimal imaging parameters

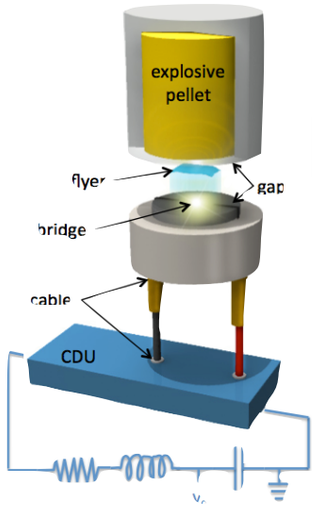
More optimal imagine parameters

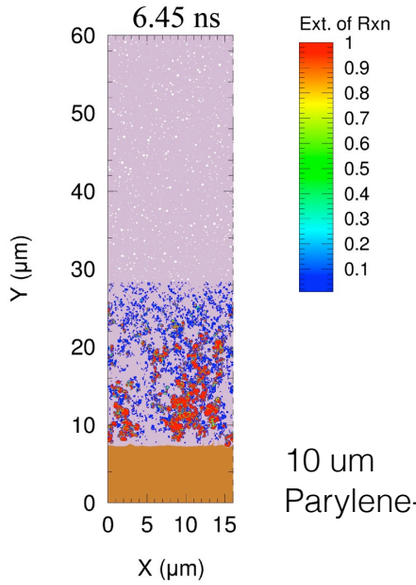
HNS-10 1 µm EHT = 1.50 kV WD = 4.3 mm Date :21 Apr 2009
Mag = 25.00 K X Signal A = SE2 Time :11:27:32

Obligatory spinning image



Mesoscale simulations with '**equivalent**' microstructure

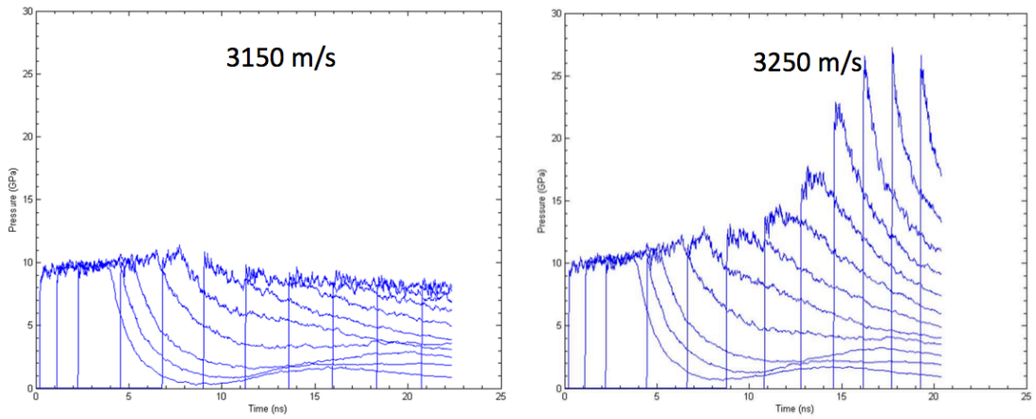




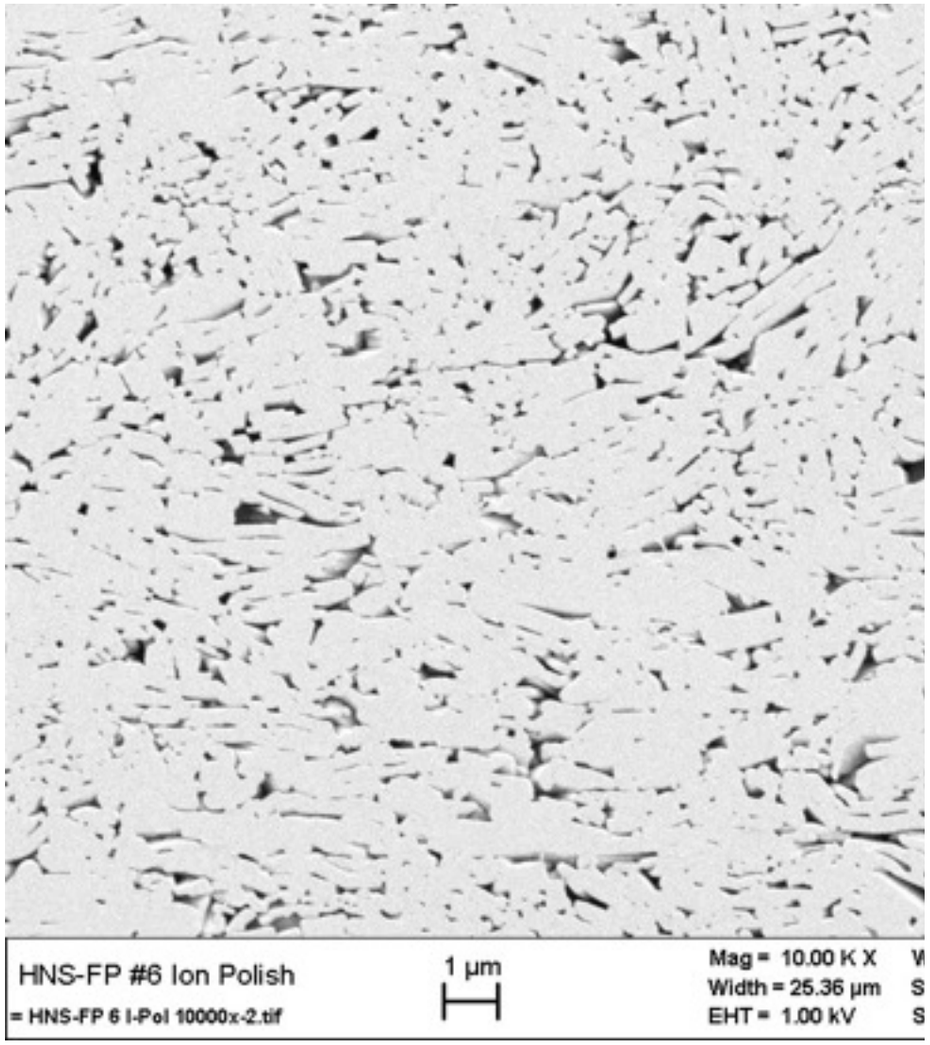
Cole Yarrington
SNL

10 um
Parylene-C flyer

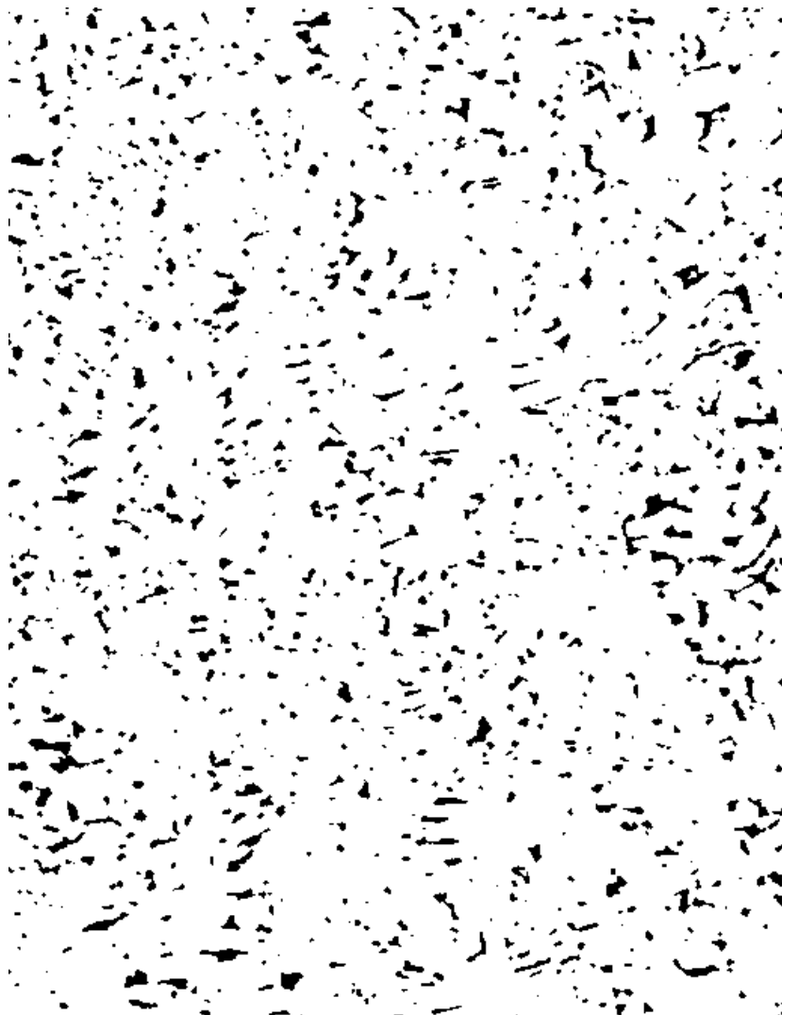
Predicts a shift in run distance with increasing pore size



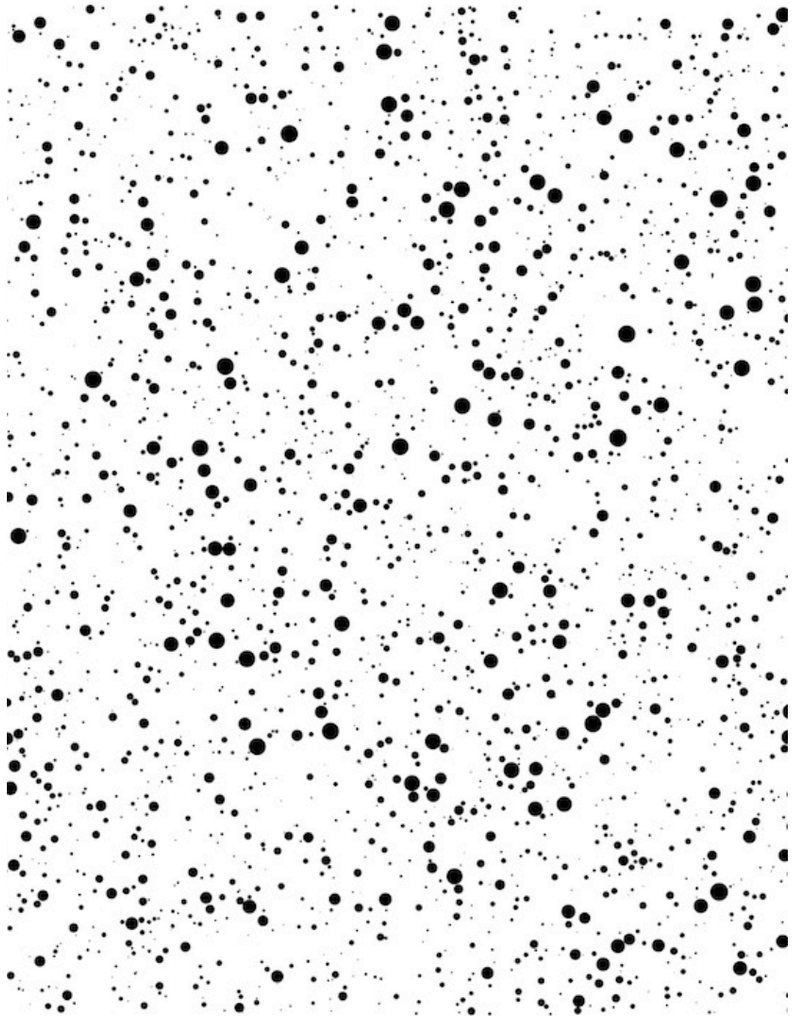
Ion mill cross section



segmented

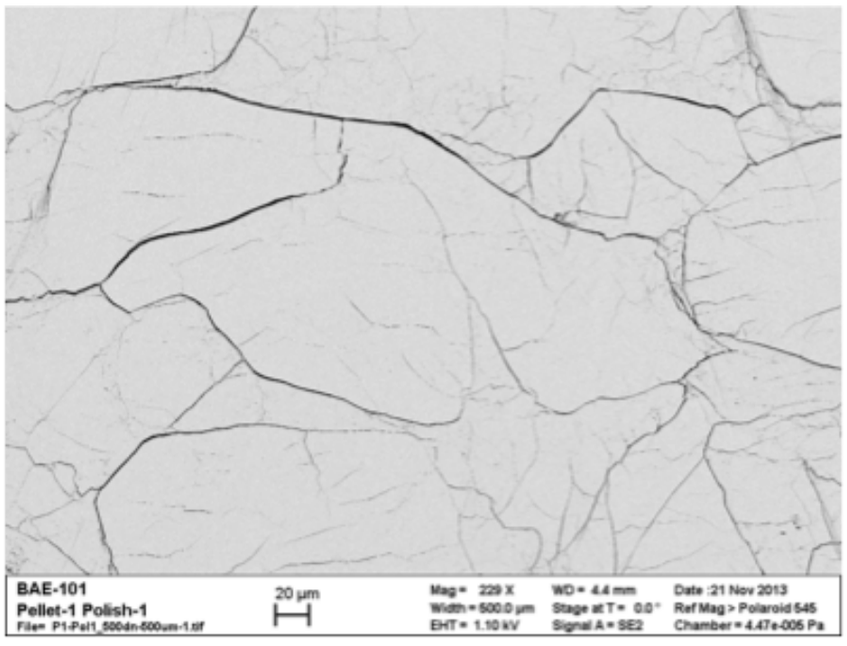


equivalent

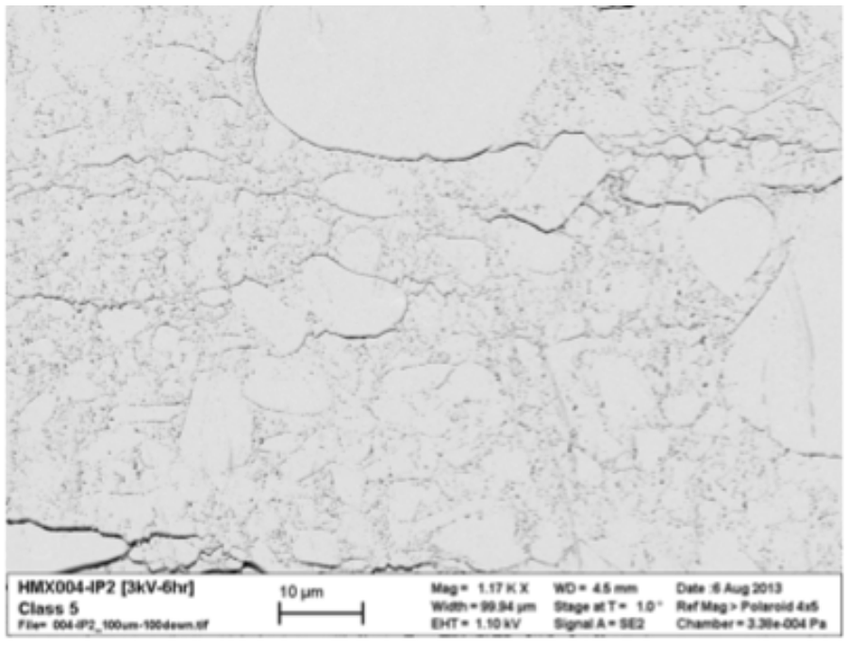


Collaboration with AFRL (Eric Welle and Chris Molek)

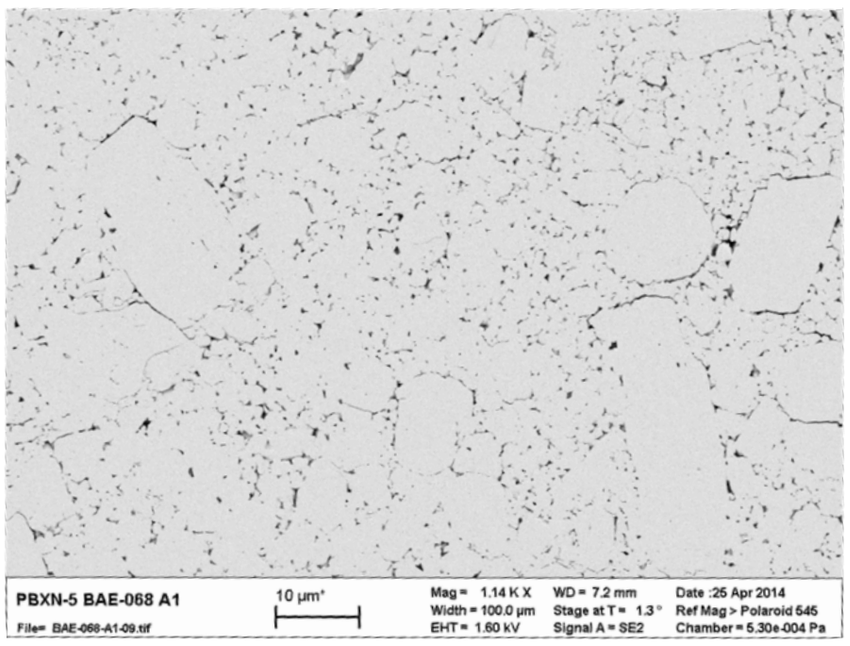
Class III

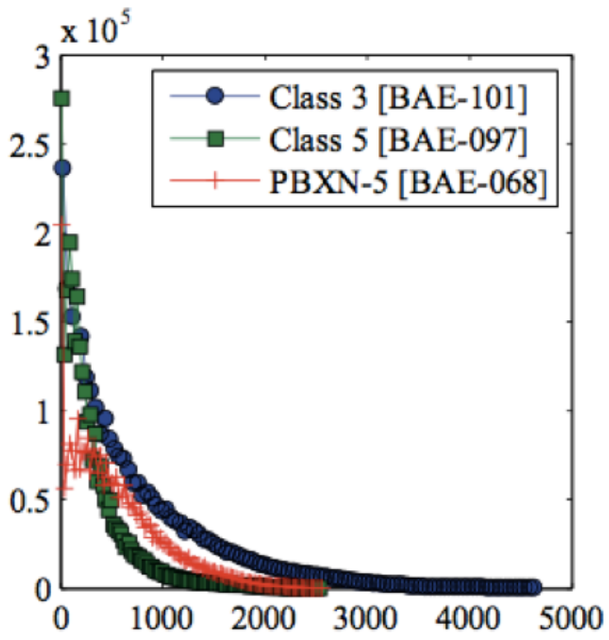


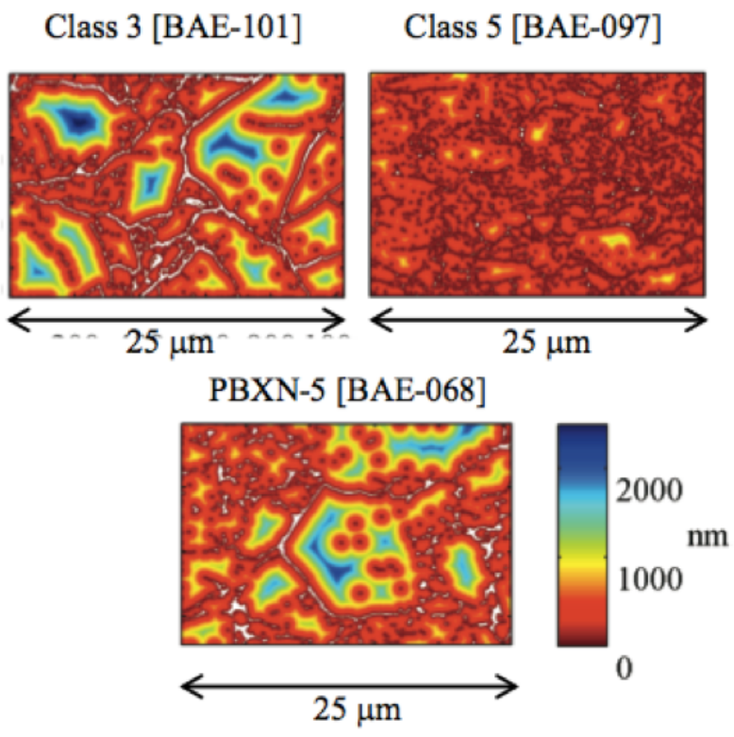
Class V



PBX N5







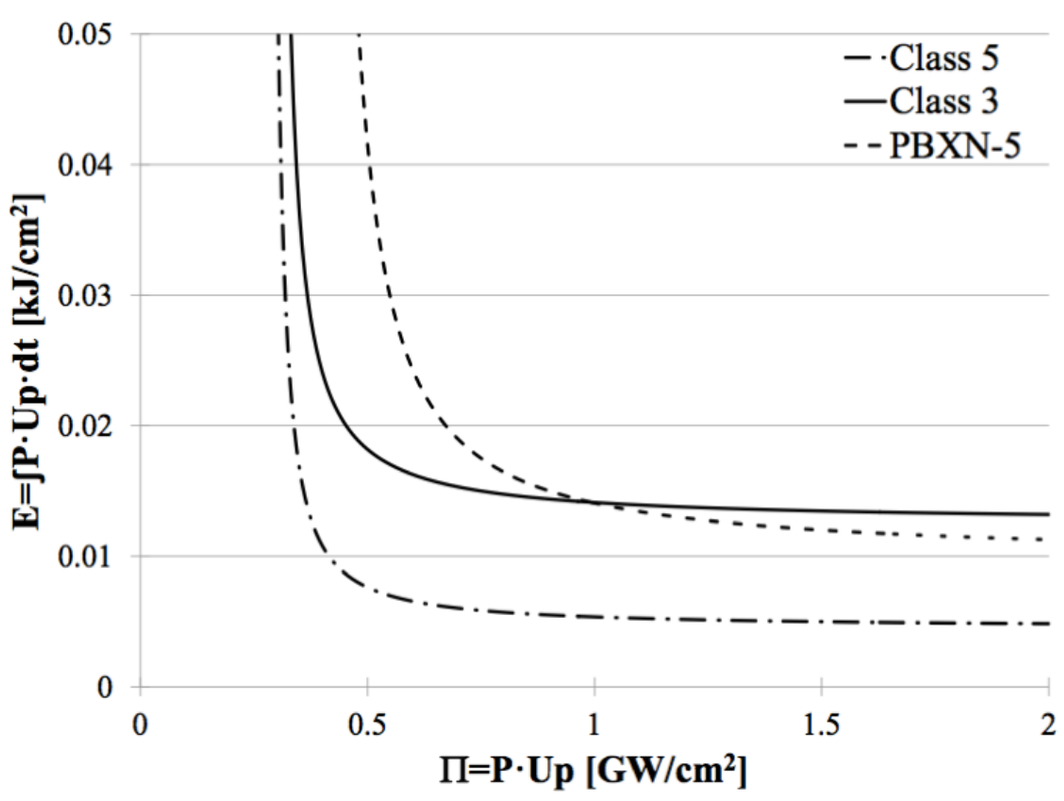


Fig. 7. Trend lines fit to initiation threshold data.

FIB/SEM, HINTS OF AMAZING THINGS TO COME

Chris Molek and Jim Vitarelli
AFRL

100 μm

30.00 μm
EHT = 3.00 kV
Mag = 39 X

Imaging Mode: SEM
Signal A = SE2
InlensDuo Grid = 0 V

30kV:65nA
WD = 5.9 mm
54.0 °

Width = 2.918 mm
Image Pixel Size = 2.849 μm

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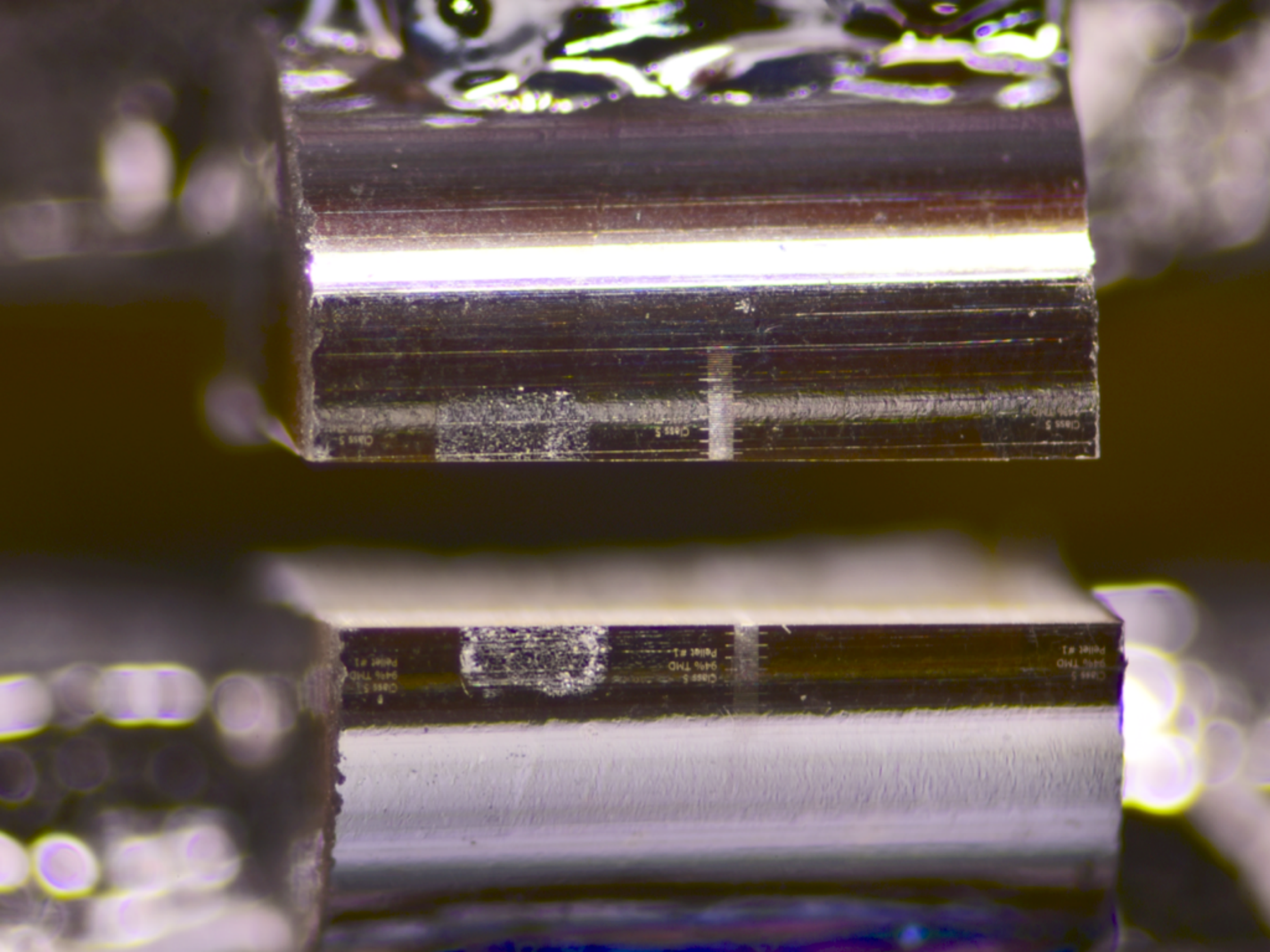
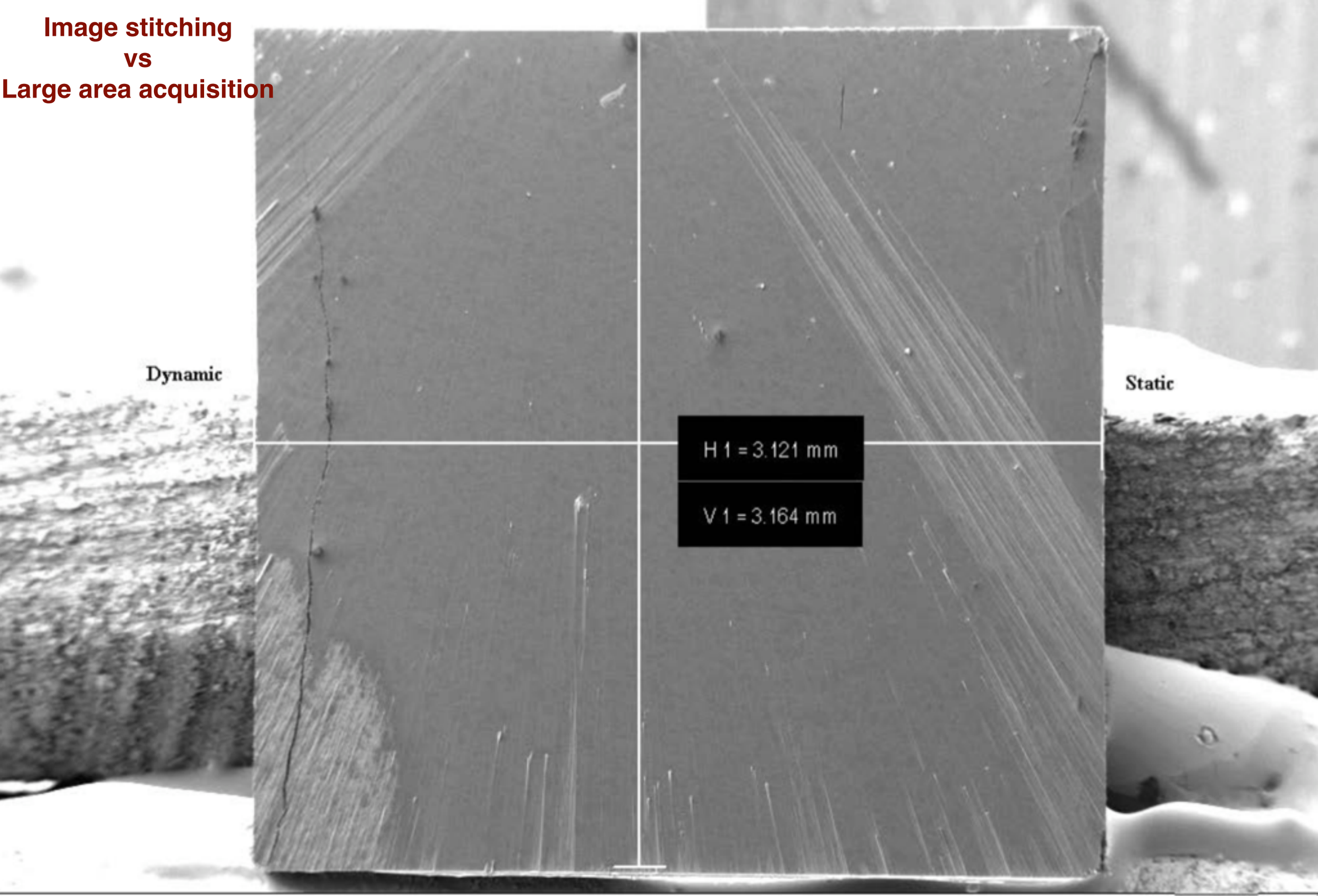

Class 5
94% THD
Pellet #1

Image stitching
vs
Large area acquisition



200 µm

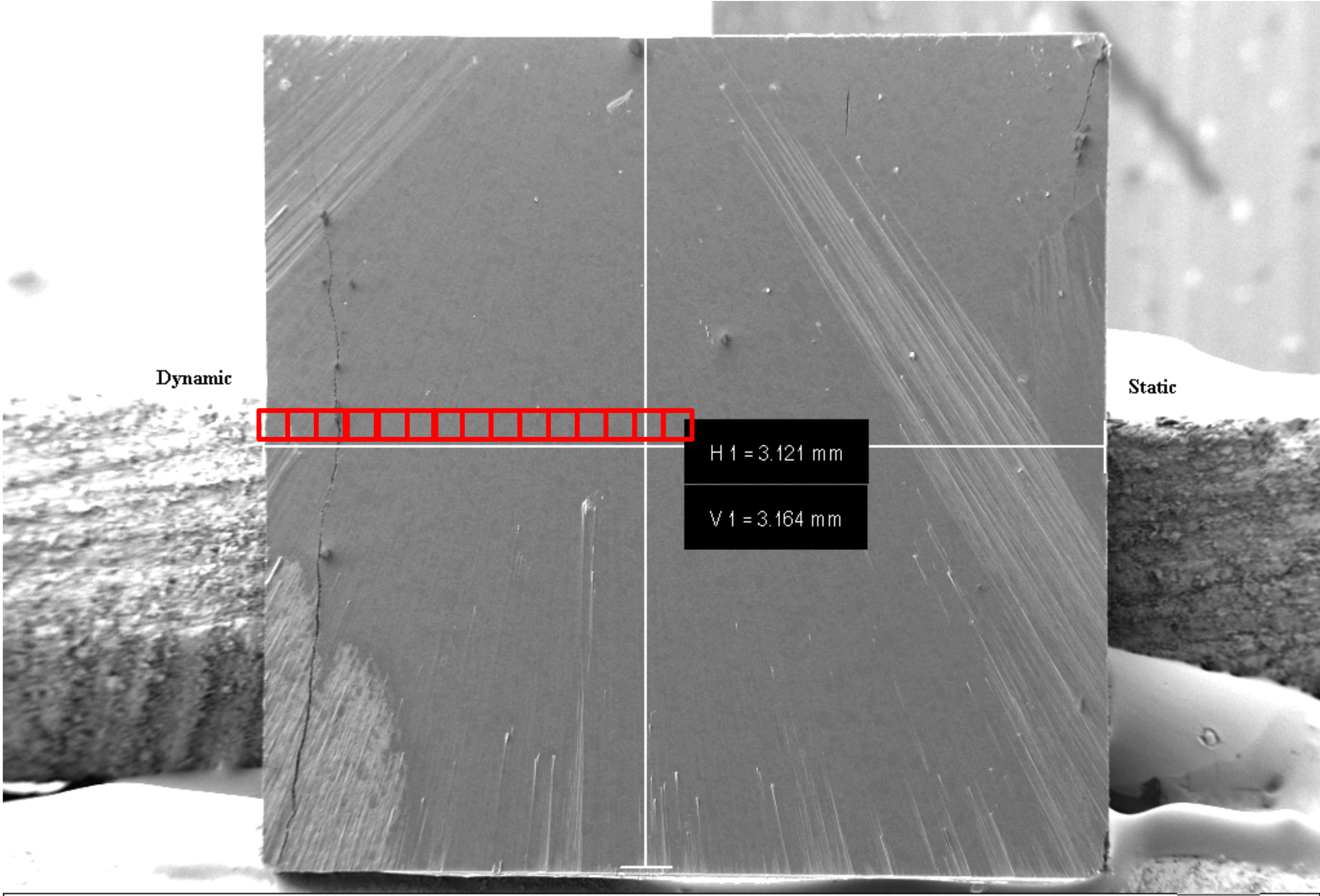
30.00 µm
EHT = 5.00 kV
Mag = 23 X

Imaging Mode: SEM
Signal A = SE2
InlensDuo Grid = 408 V

30kV:50pA ref
WD = 35.4 mm
0.0 °

Width = 4.970 mm
Image Pixel Size = 4.853 µm

UDRI
UNIVERSITY of DAYTON RESEARCH INSTITUTE



200 µm

30.00 µm
EHT = 5.00 kV
Mag = 23 X

Imaging Mode: SEM
Signal A = SE2
InlensDuo Grid = 408 V

30kV:50pA ref
WD = 35.4 mm
0.0 °

Width = 4.970 mm
Image Pixel Size = 4.853 µm

UDRI
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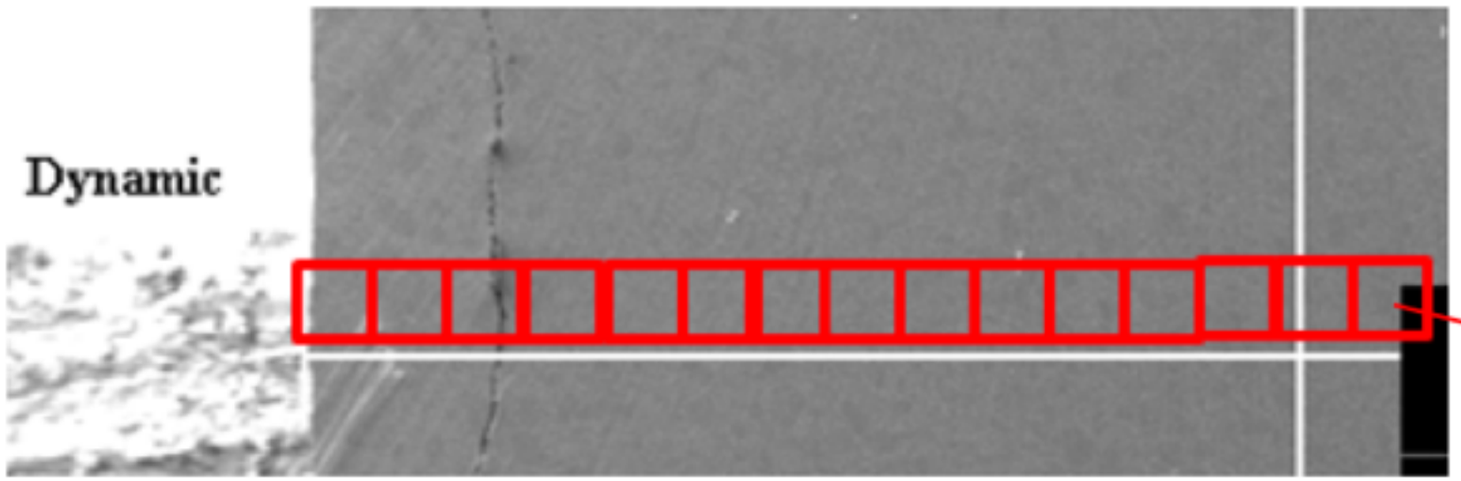
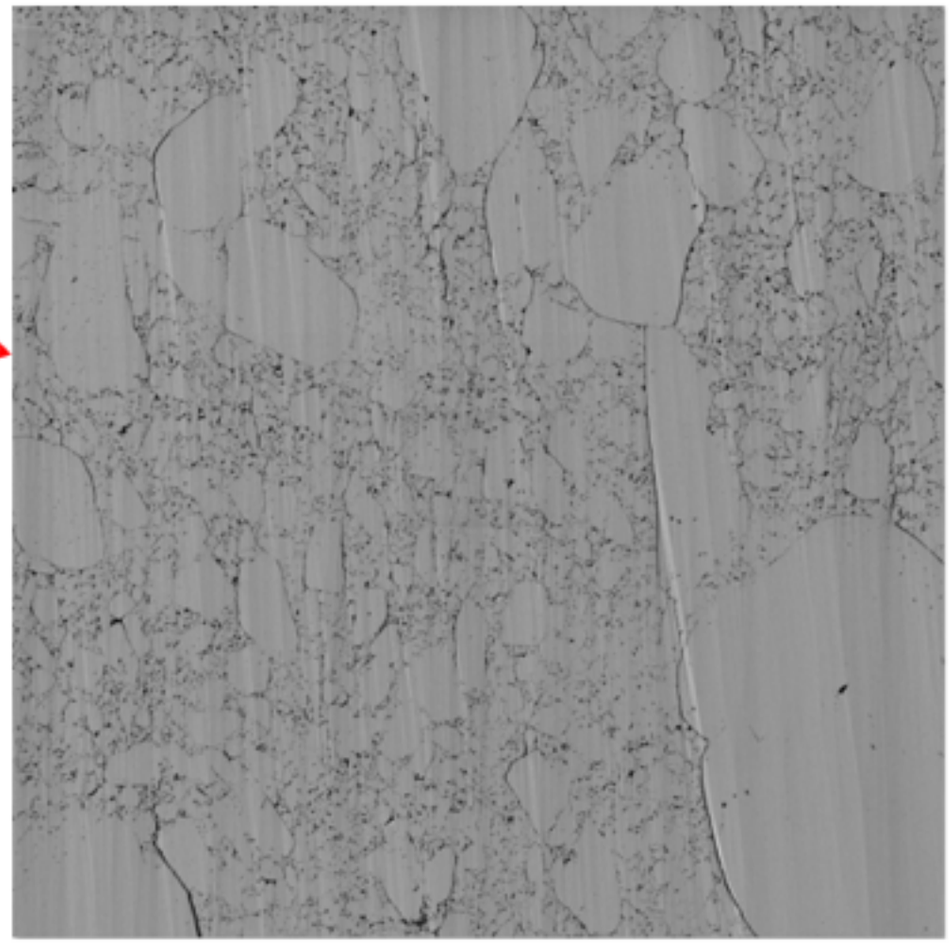
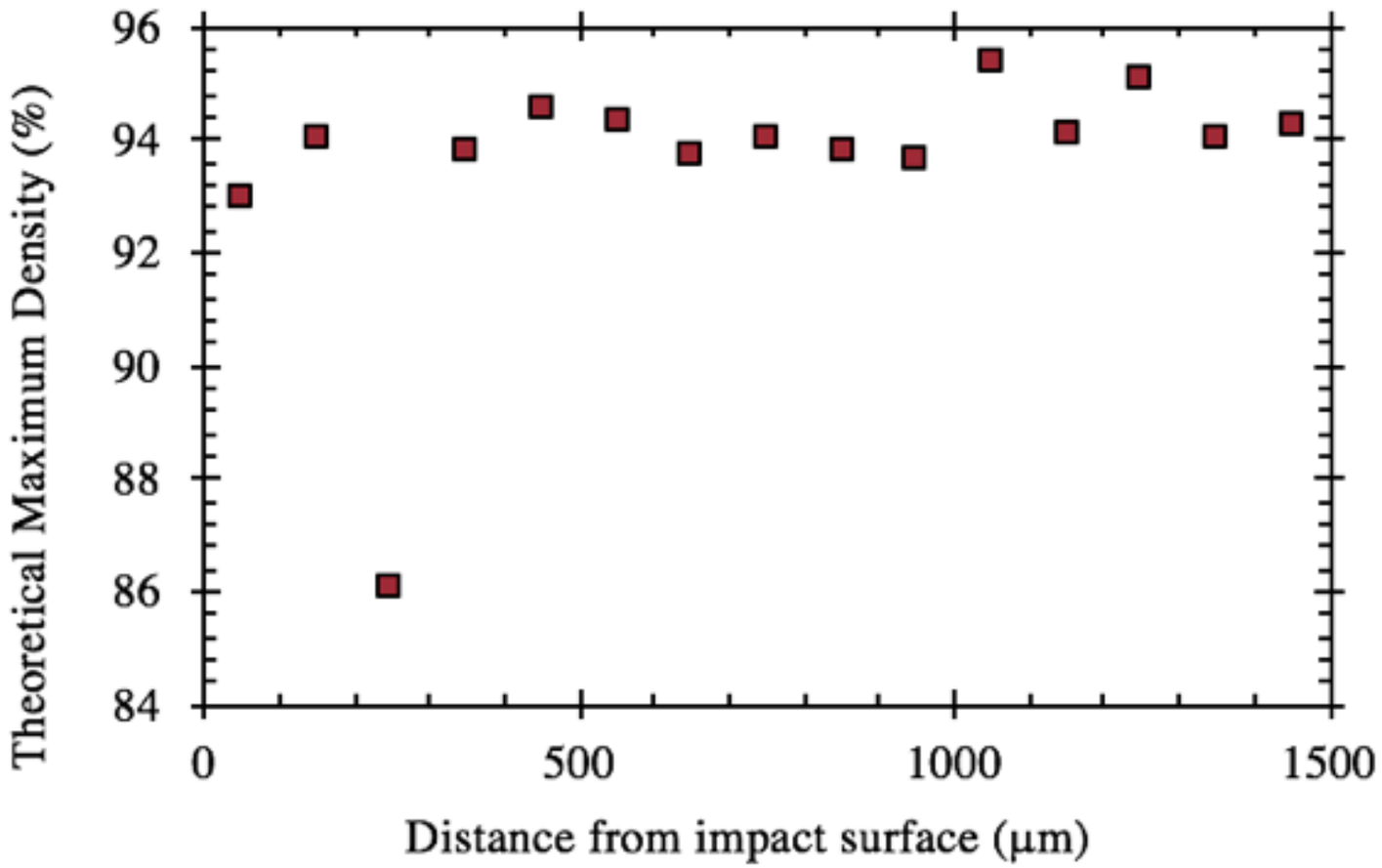
Density Variation as a Function of Depth

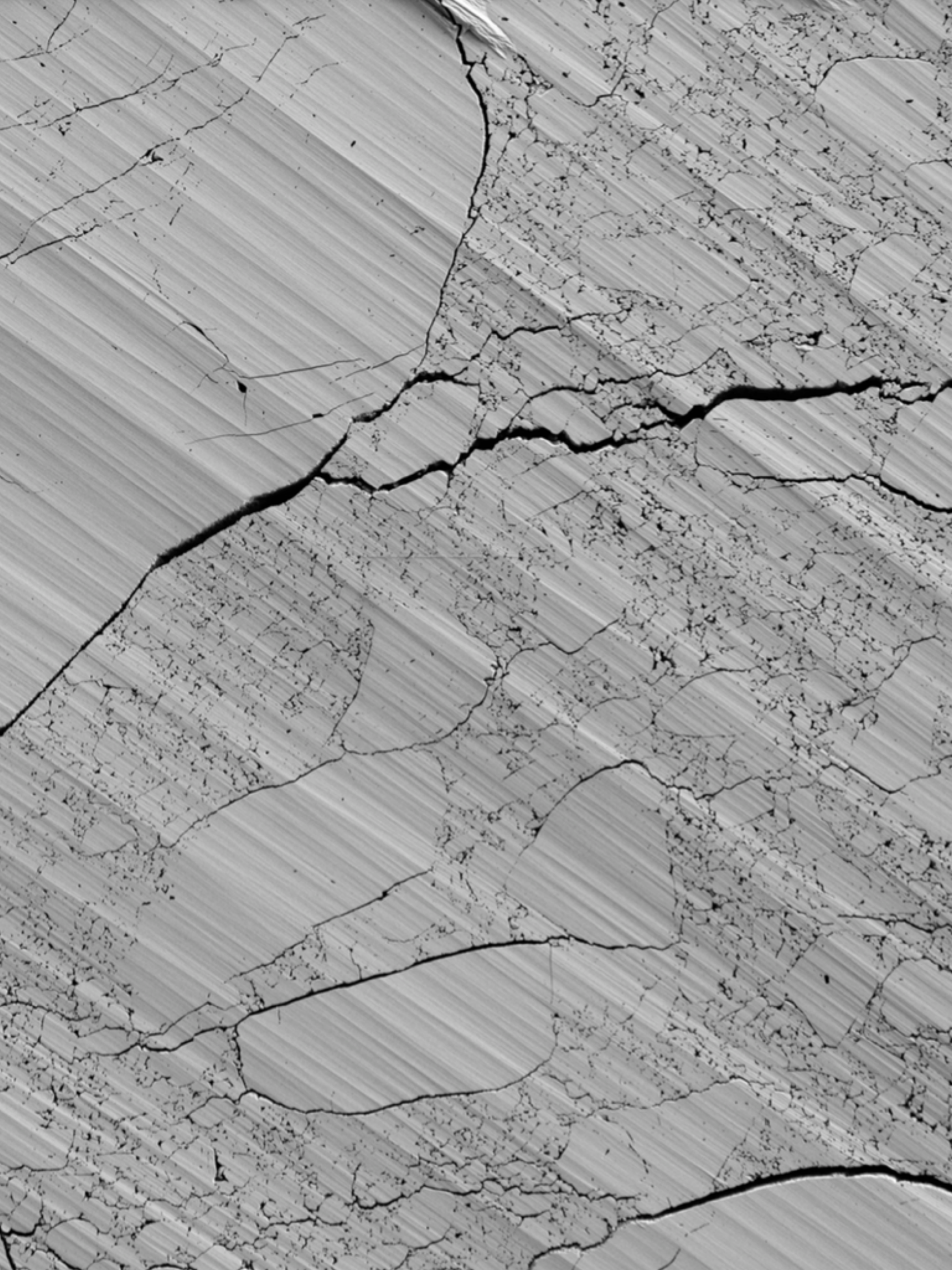


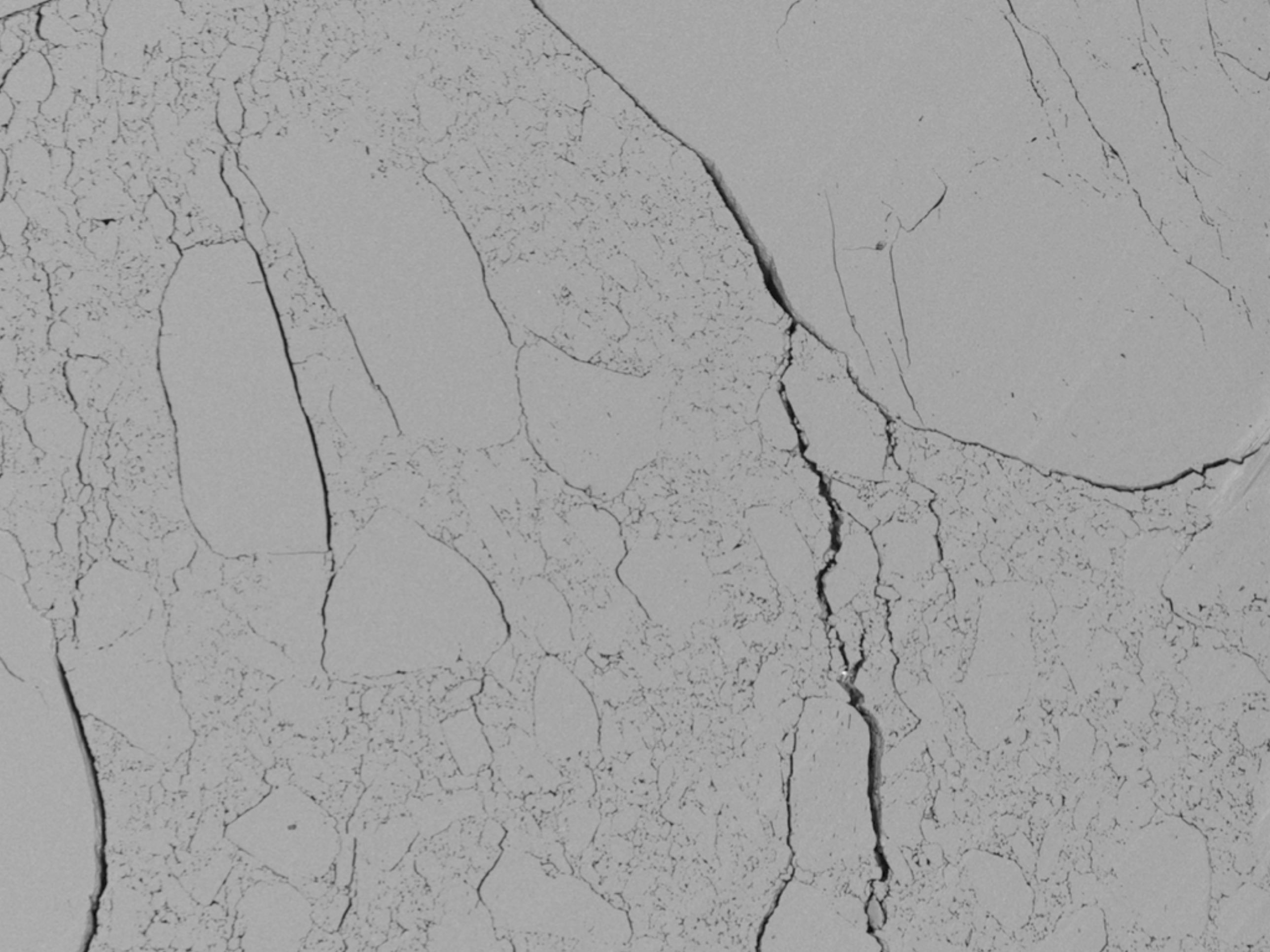


Class 5 at nominal 94% TMD

- Each image 100 μm square and 9.8 nm pixel size
- SIA is 0.0024 (0.0002) nm^2/nm^3

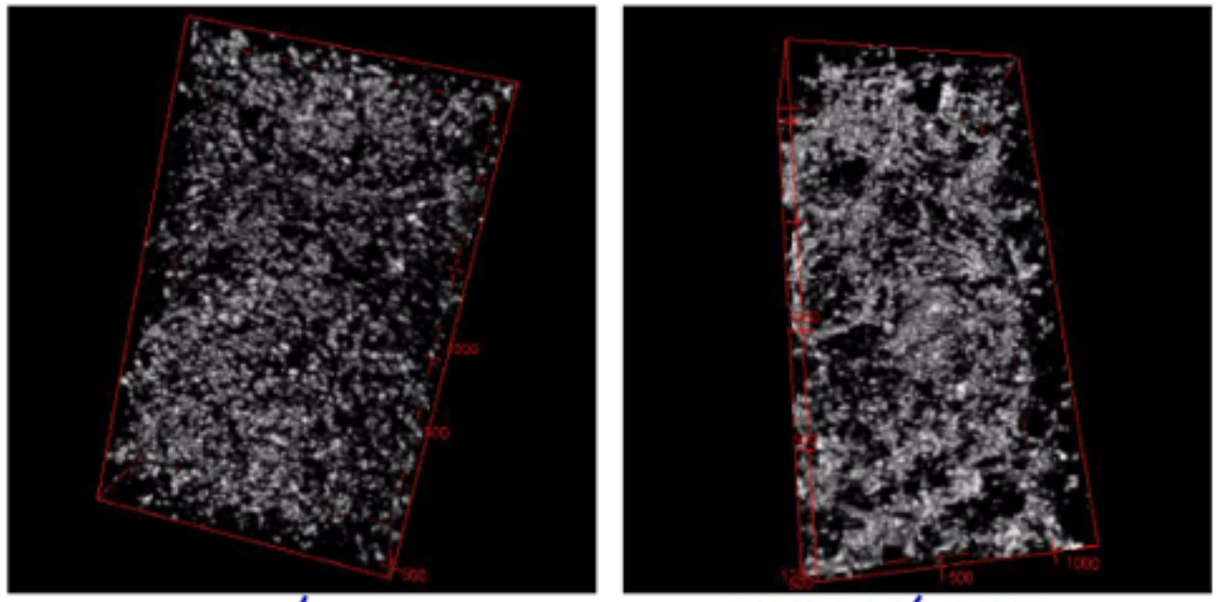




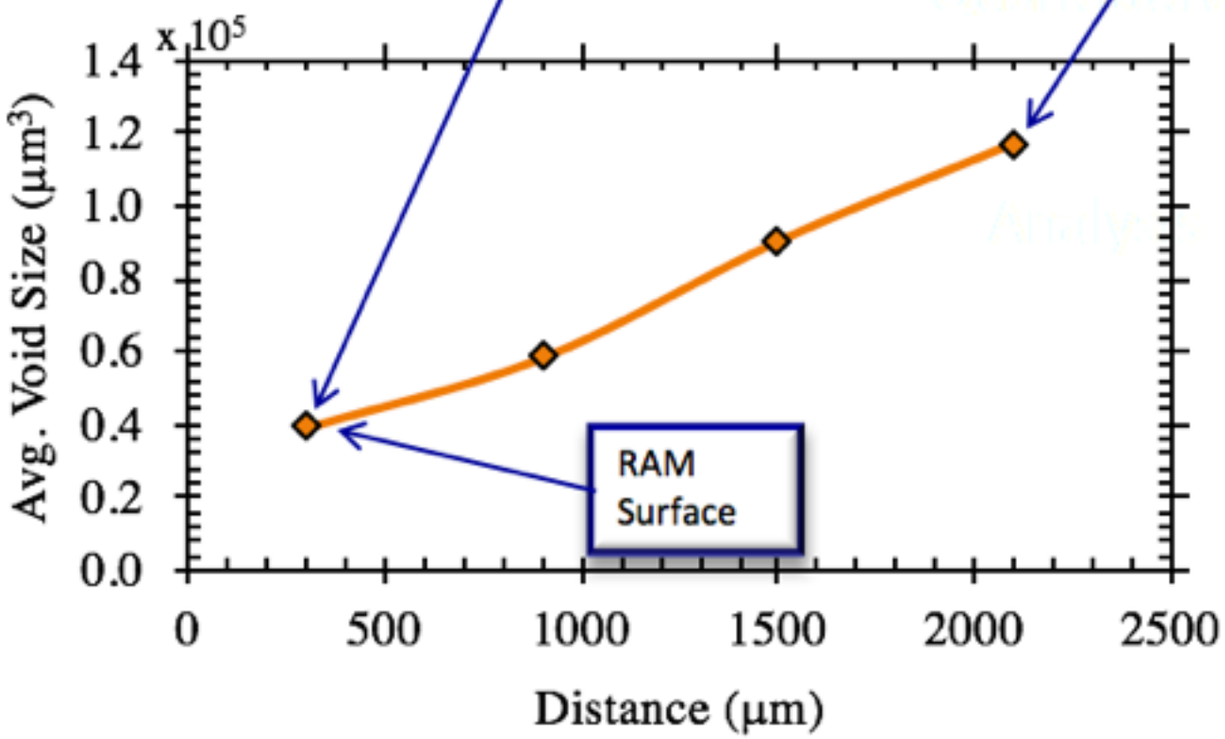


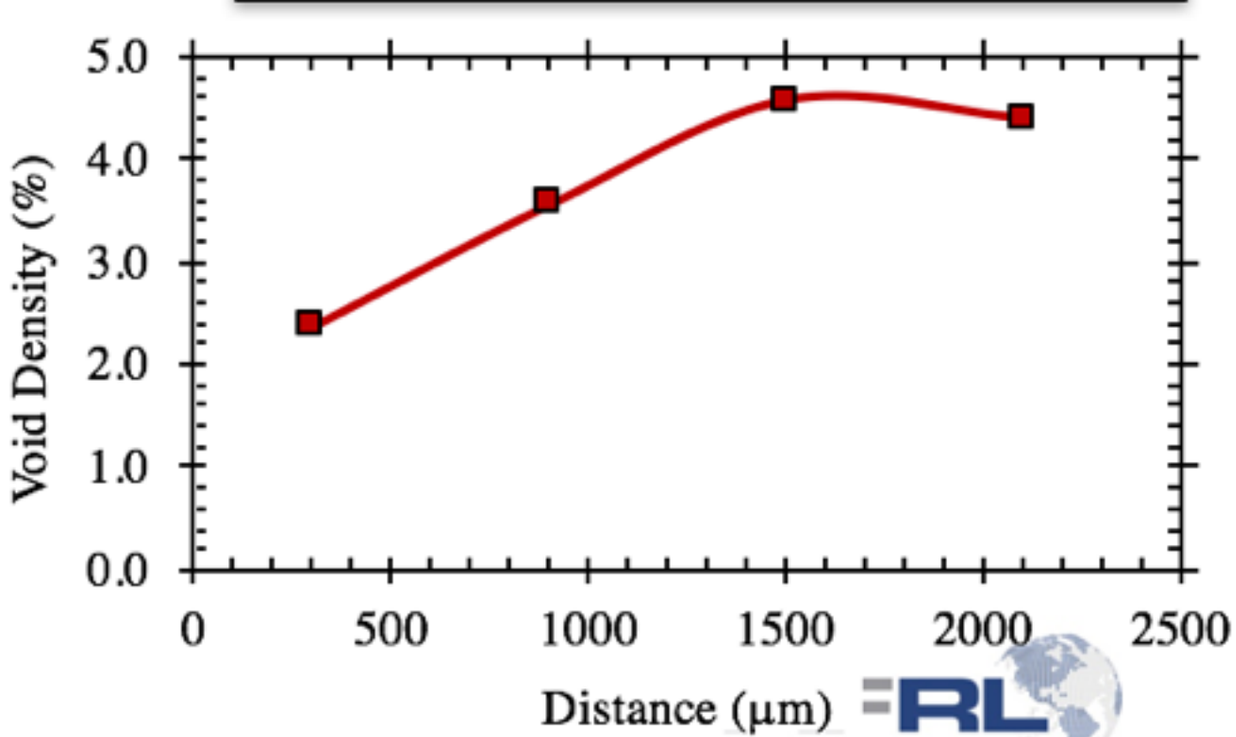
Class 1 XCMT Data

Class 1 data obtained for 3 mm pellet at 94% TMD



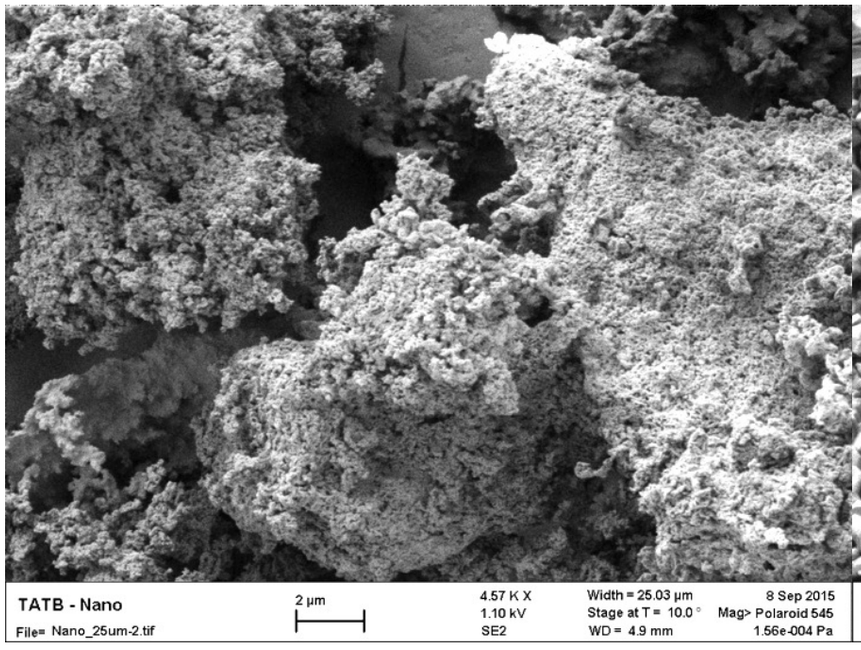
- Dynamic face of the press is correlating to higher cracking of crystals, i.e. higher observed calculated density
- Observed > 500 micron crystals with voids 20-60 μm in size in the center



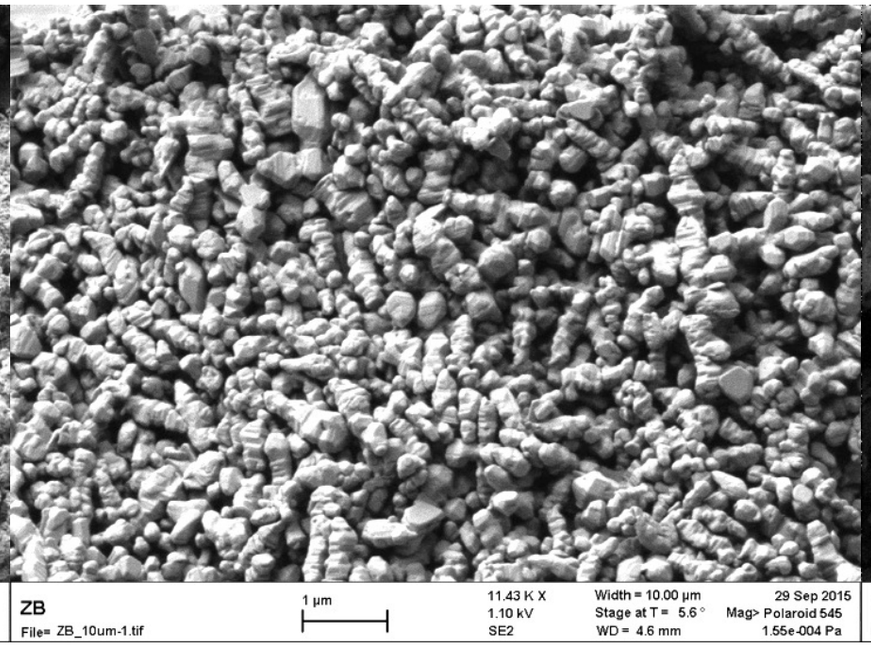


TATB STUDY: THREE ORDERS OF MAGNITUDE

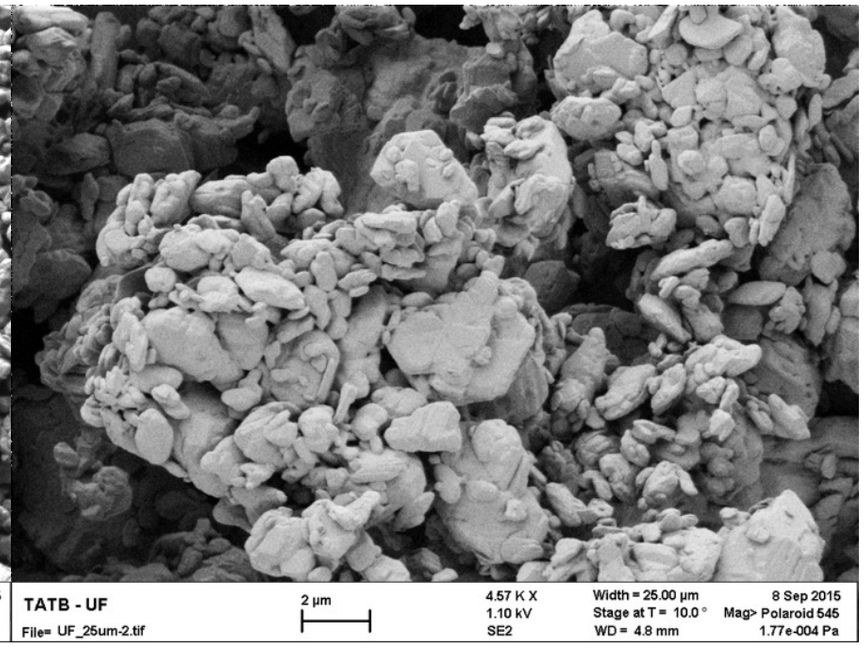
ARDEC

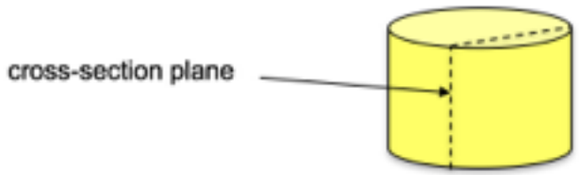


SNL

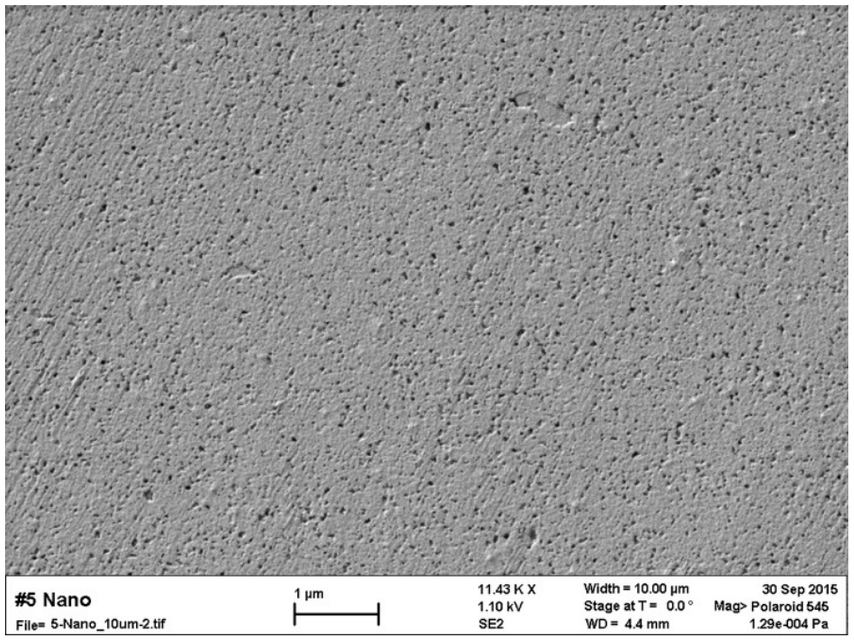


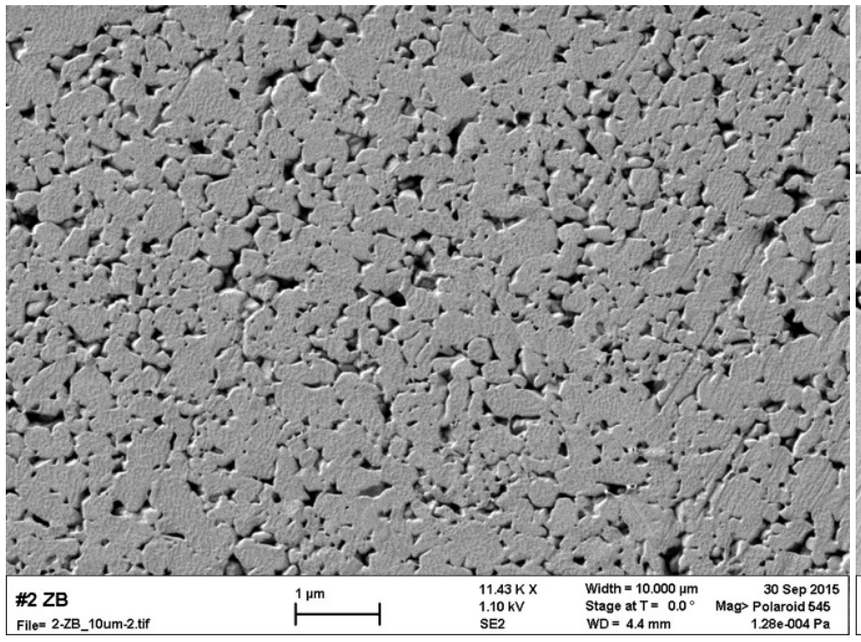
PANTEX

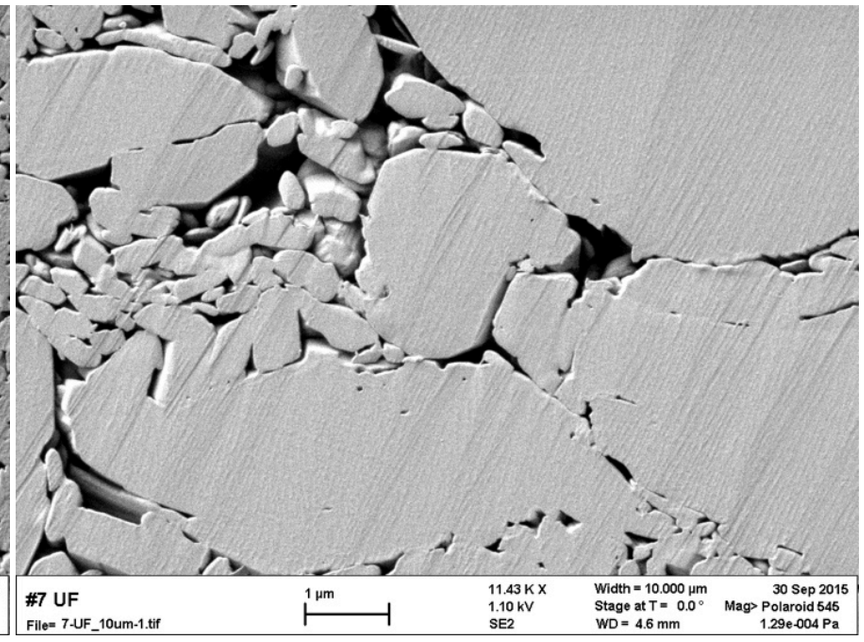


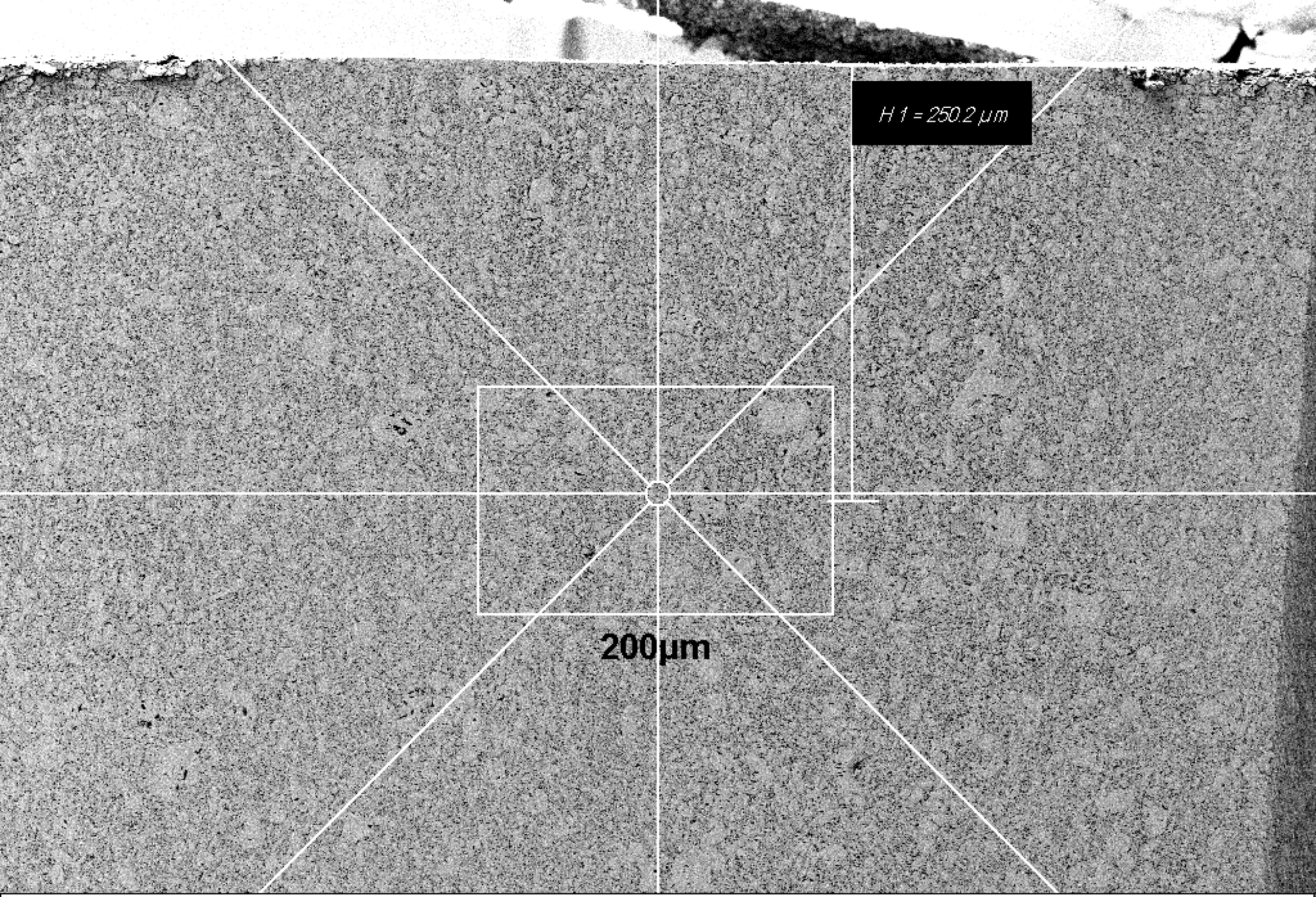


Pellets pressed to 80% TMD, ion-milled, SEM









TATB Ultra-Fine (7)

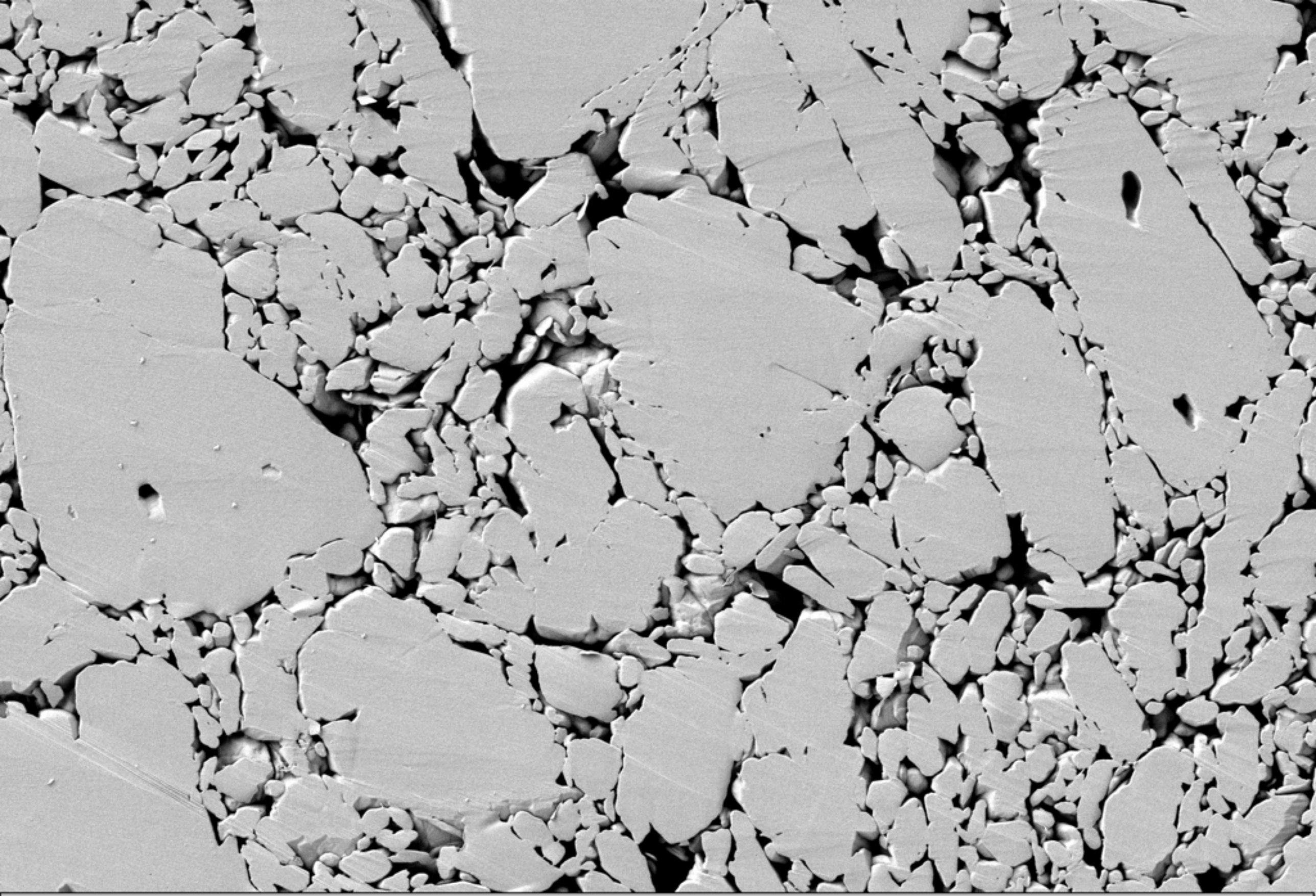
File= Ultra-Fine_IP_16K Area.tif

100 µm

151 X
1.10 kV
SE2

Width = 755.8 µm
Stage at T = 0.0 °
WD = 3.8 mm

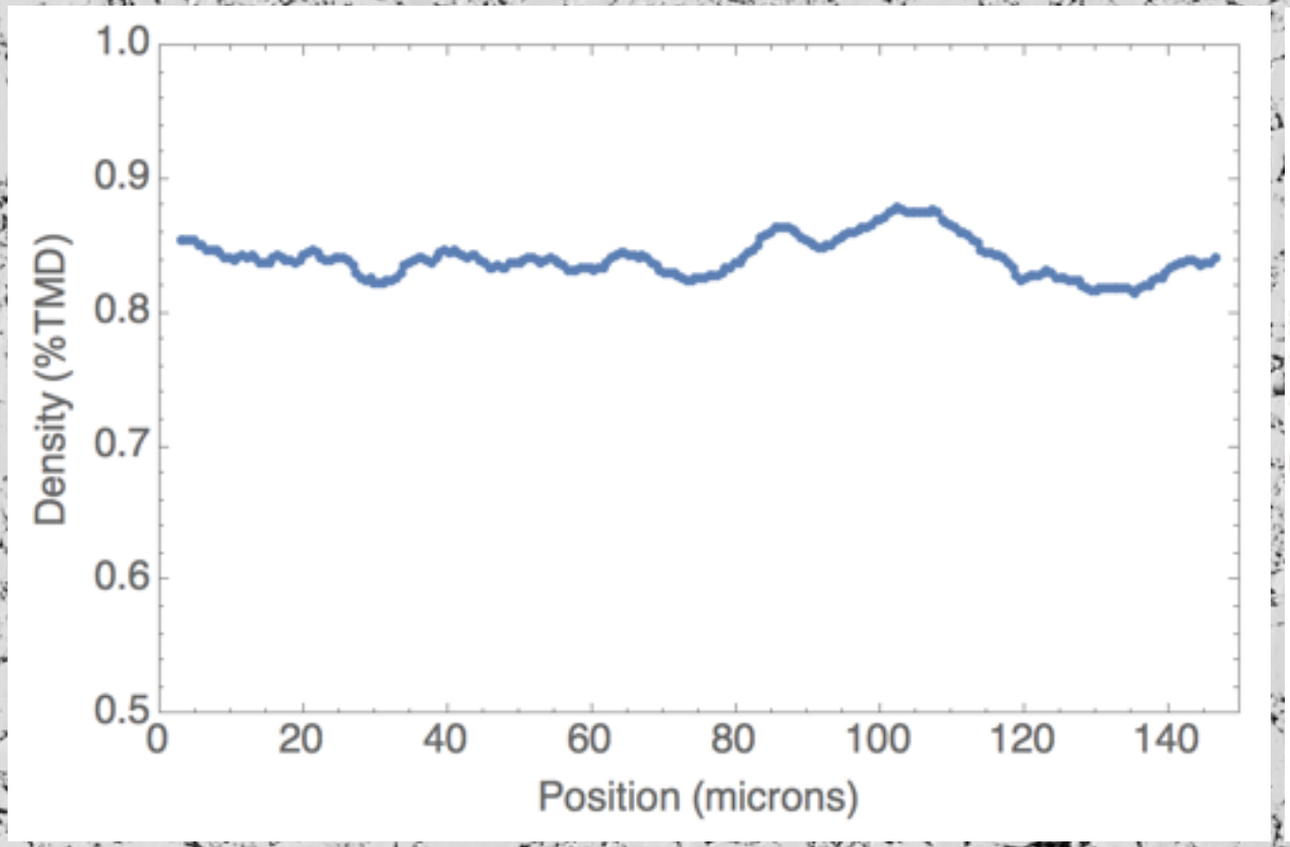
14 Jul 2016
Mag> Polaroid 545
0.00e+000 Pa

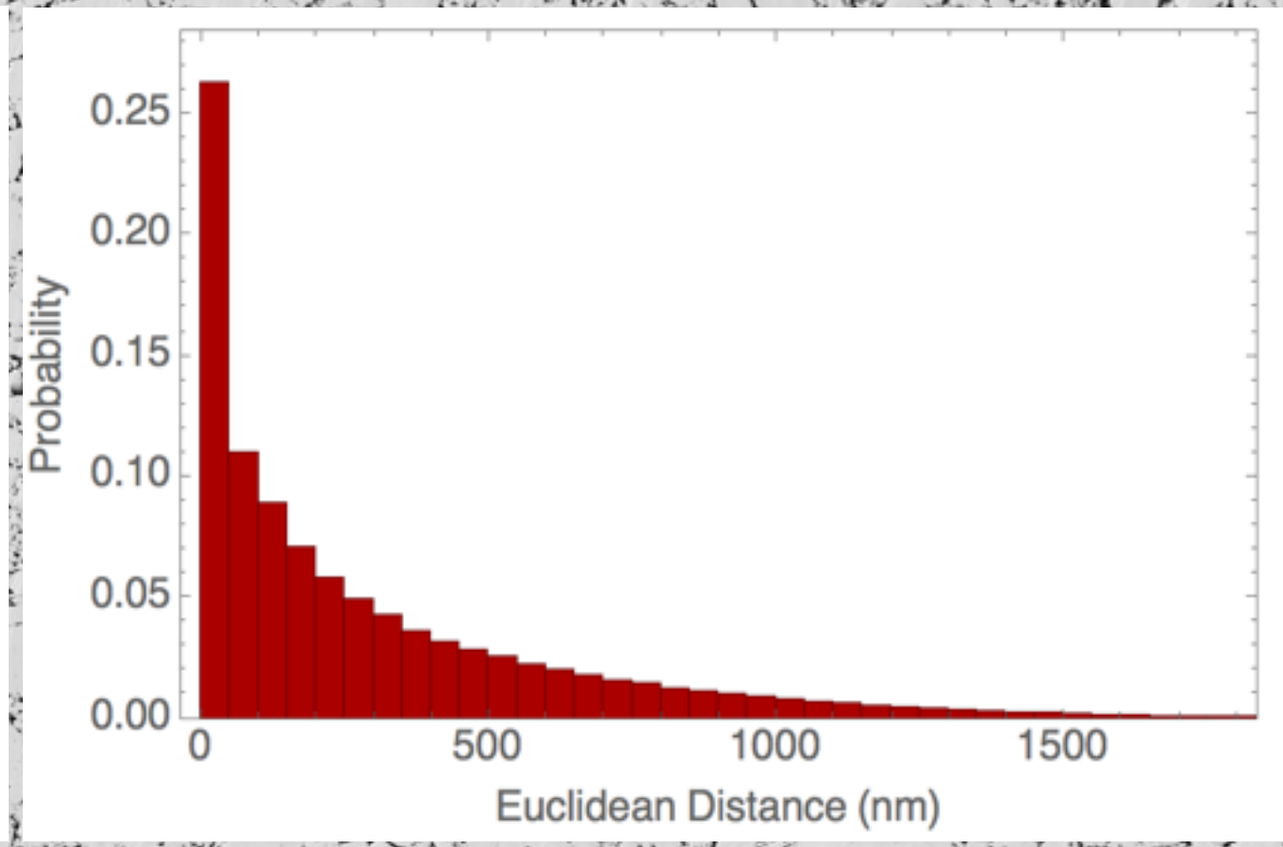

TATB Ultra-Fine (7)
File= Ultra-Fine_IP_25um-2048.tif
2 µm
4.57 K X
1.10 kV
SE2
Width = 25.00 µm
Stage at T = 0.0 °
WD = 4.5 mm
8 Jul 2016
Mag> Polaroid 545
0.00e+000 Pa

16384 x 12288 pixels / 12 nm pixel size / 200 um FOV

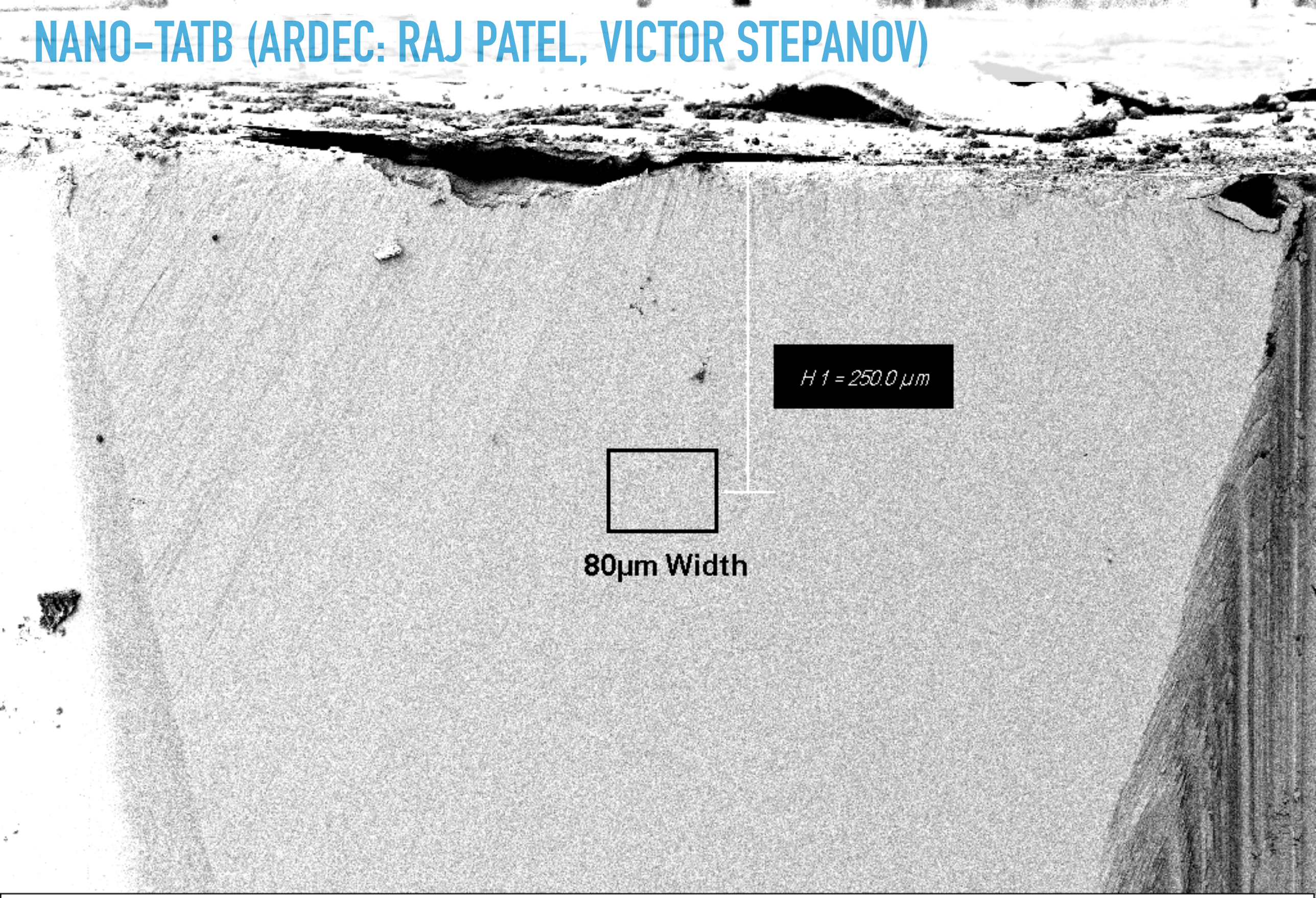
16384 x 12288 pixels / 12 nm pixel size / 200 um FOV

80% TMD
500 Bin width
50 step size



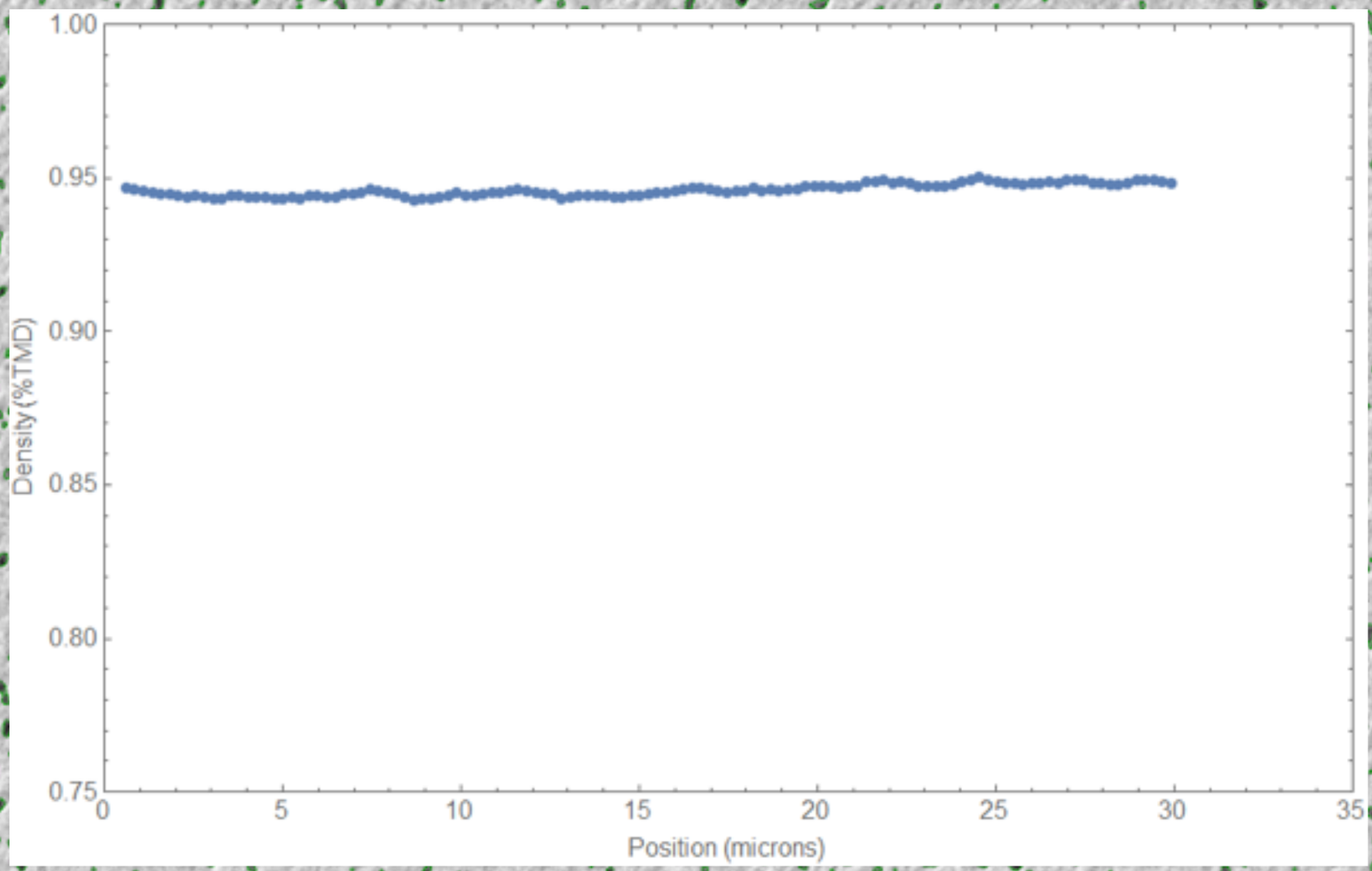


NANO-TATB (ARDEC: RAJ PATEL, VICTOR STEPANOV)

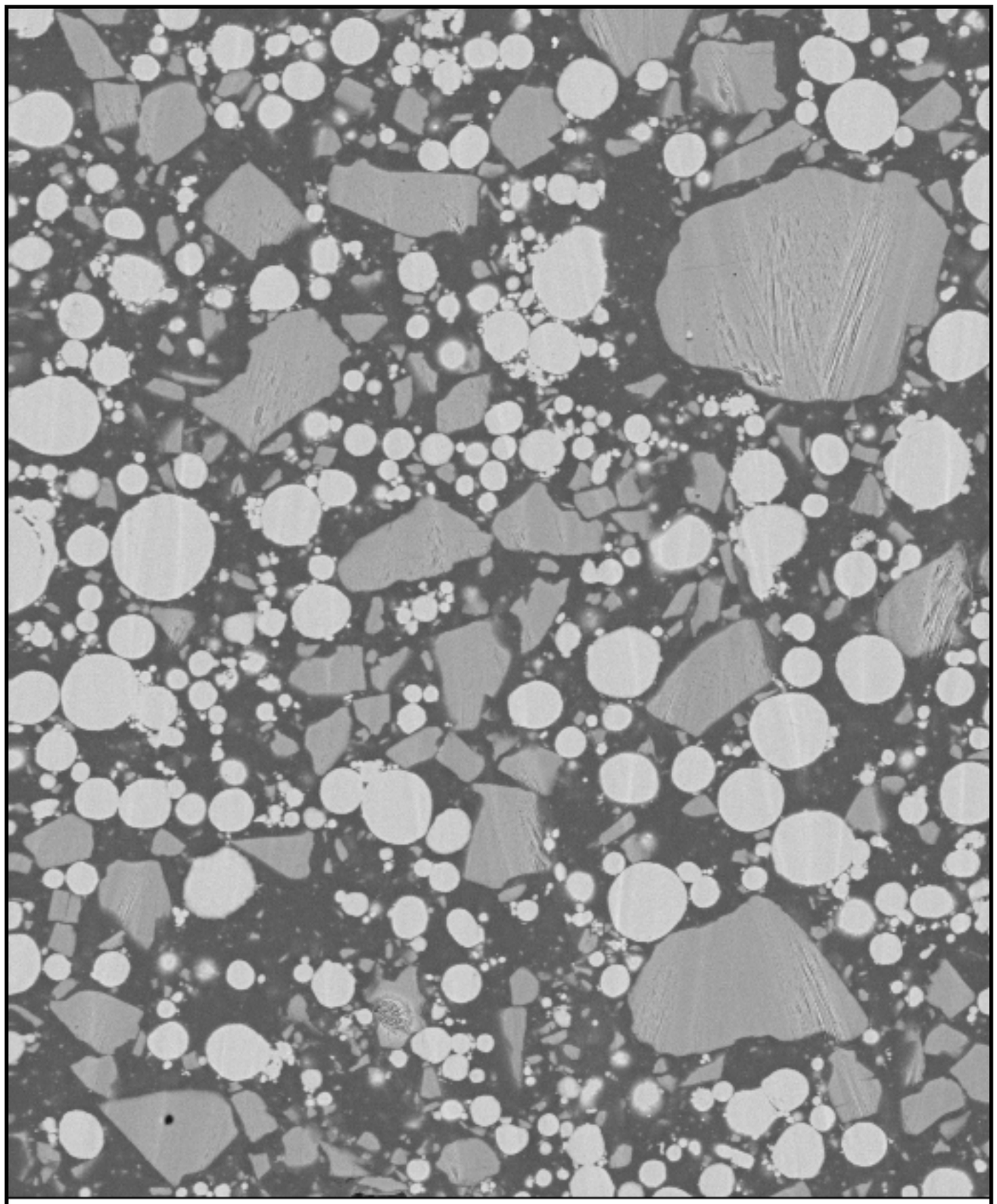


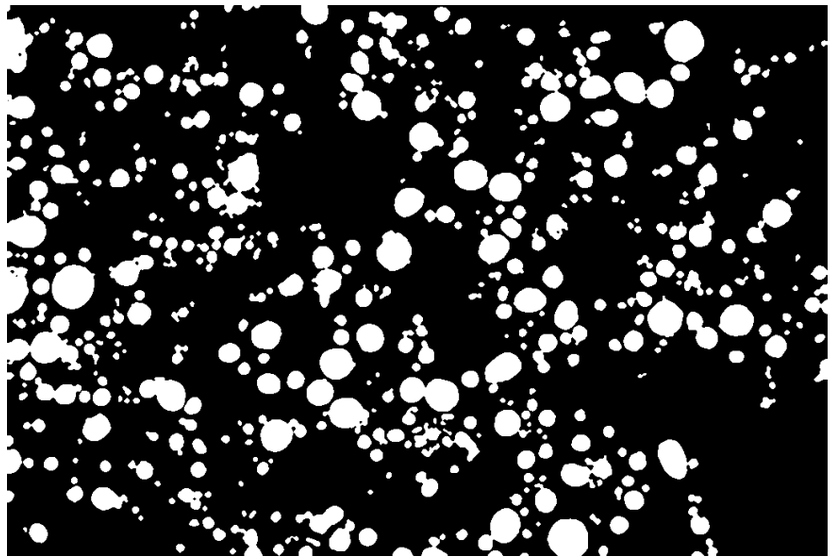

TATB Nano-Powder (5)
File= NanoPwdr_lP_10um-2048.tif
1 µm
11.43 K X
1.10 kV
SE2
Width = 10.00 µm
Stage at T = 0.0 °
WD = 4.5 mm
8 Jul 2016
Mag> Polaroid 545
0.00e+000 Pa

Density to high? Location in pellet?
Maybe even the SEM can't resolve the pores.
Perhaps AFM? FIB into center of pellet?

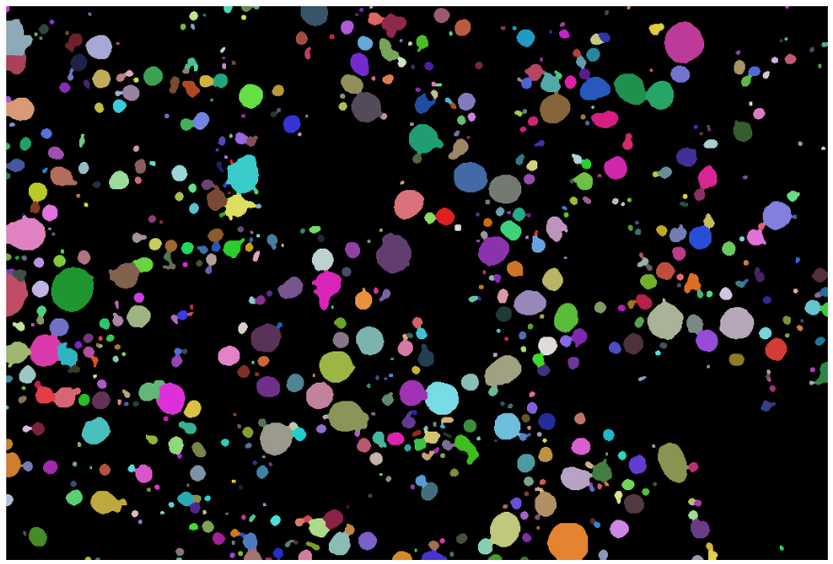


COMPOSITE PROPELLANT

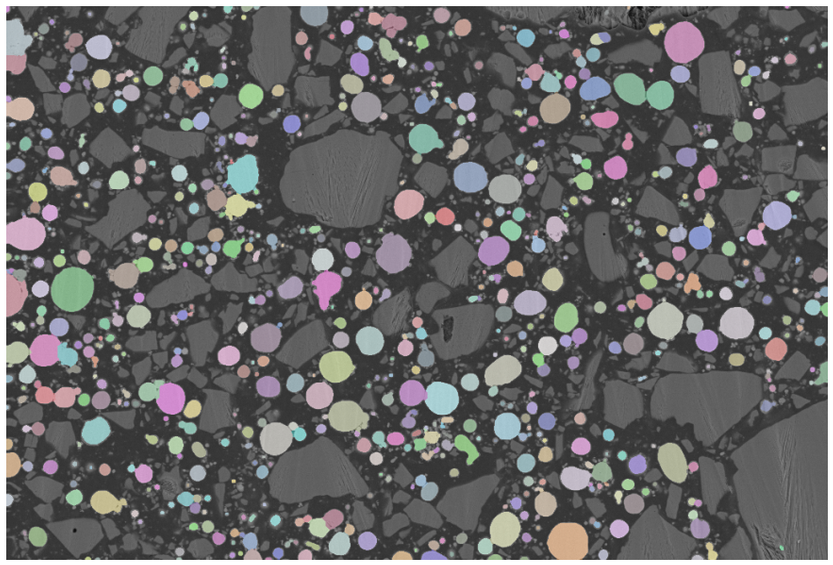




Binary image of aluminum particles

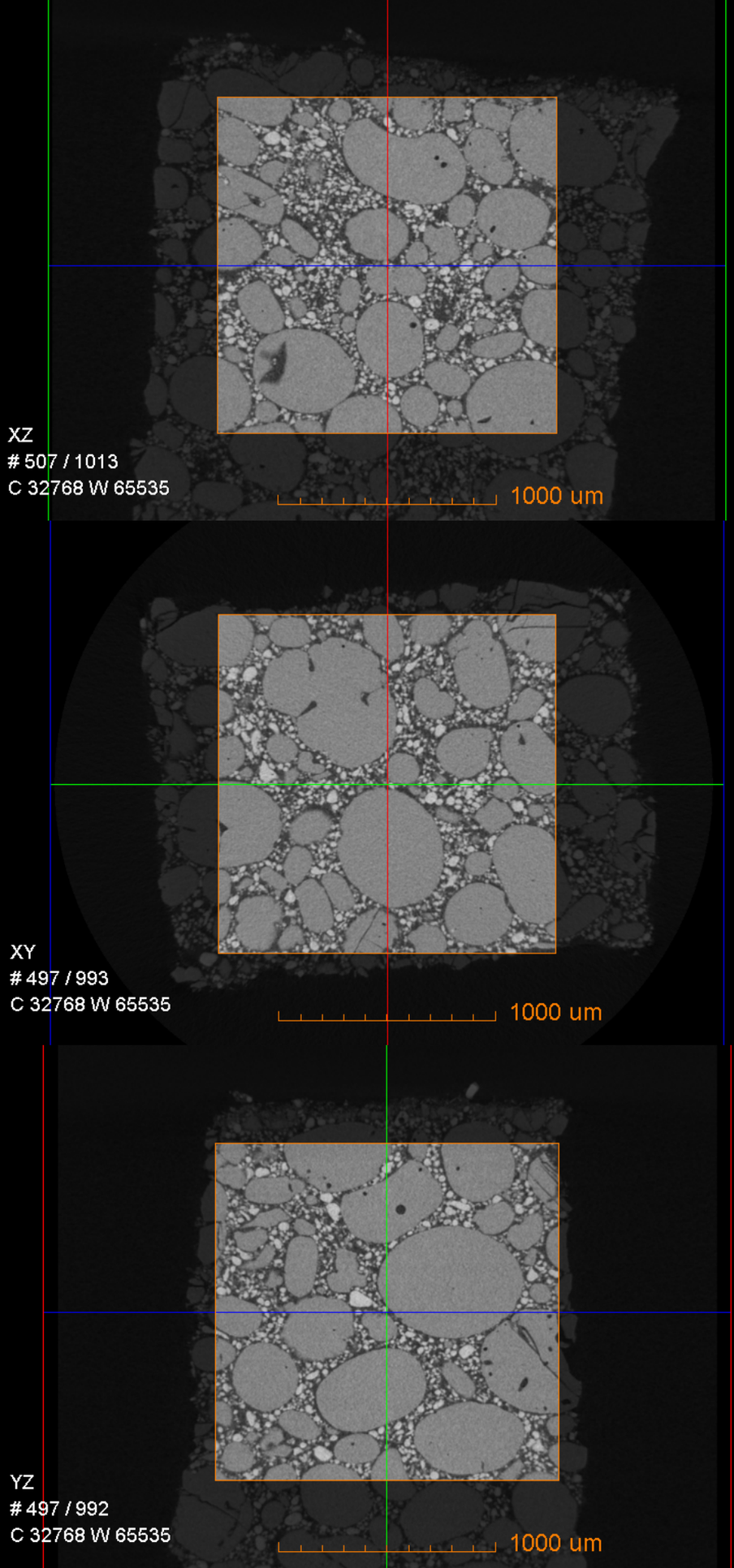


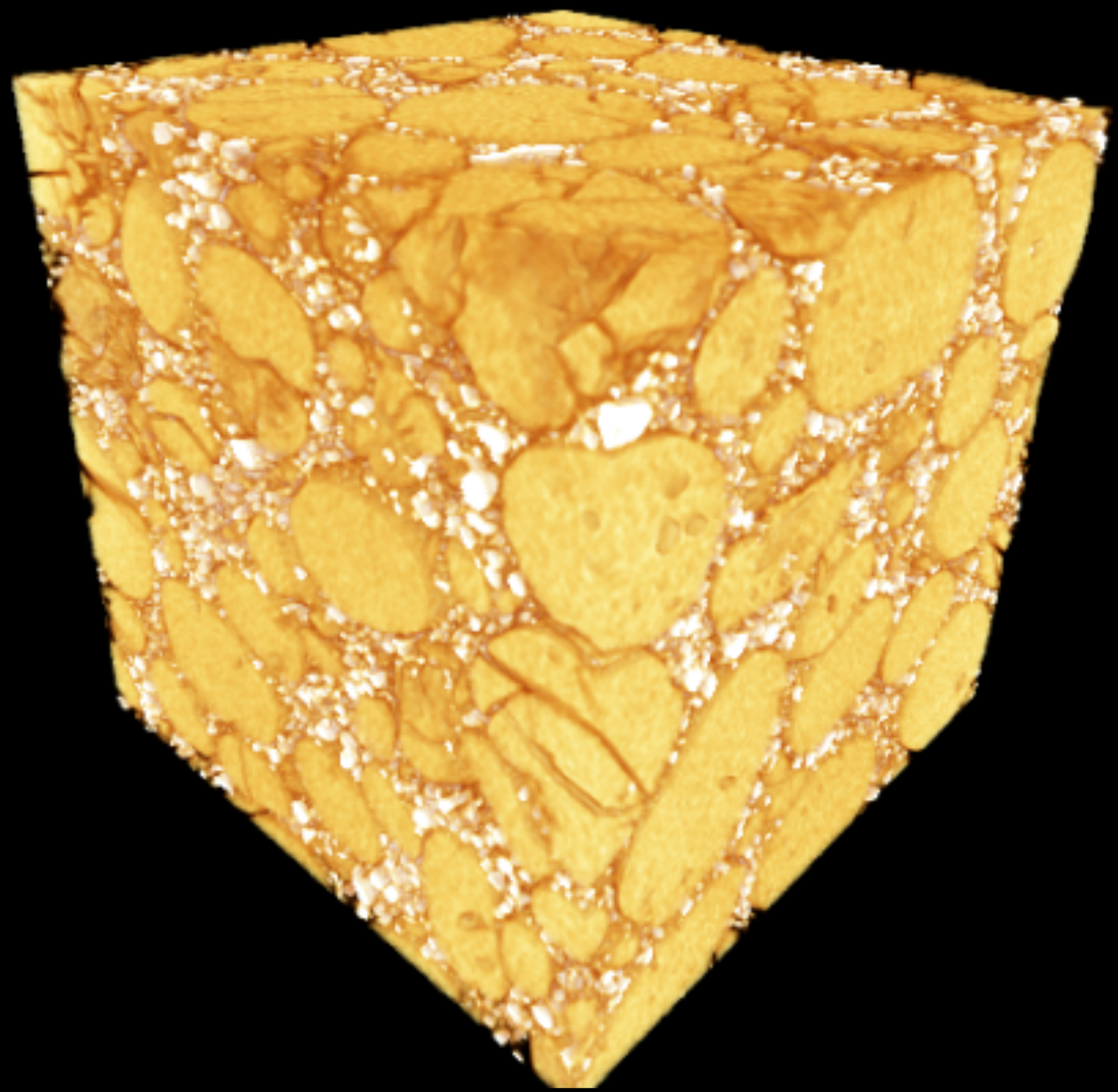
Identify and measure individual particles



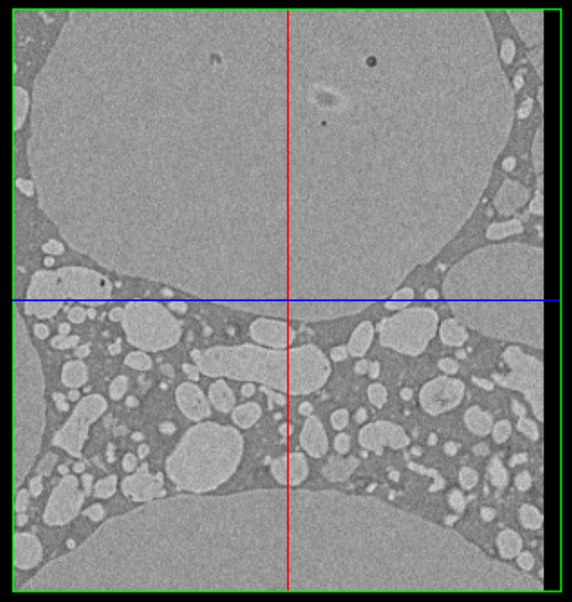
Overlay of aluminum particles onto original

APCP, 3 um pixels, 3 mm FOV

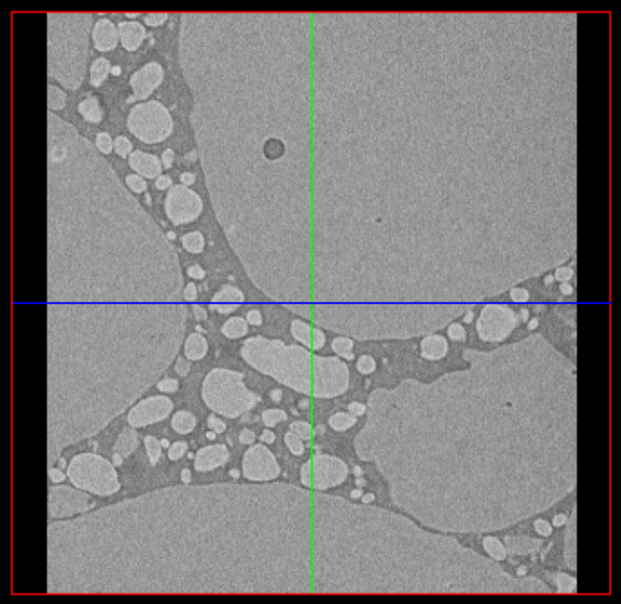




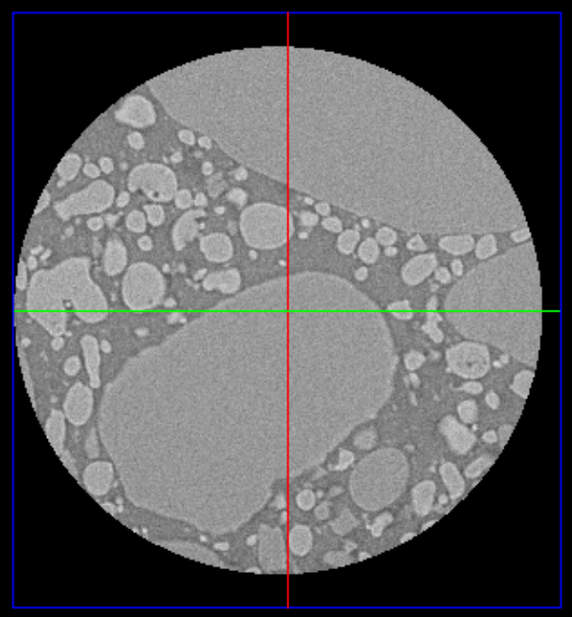
480 nm pixels, 437 um FOV



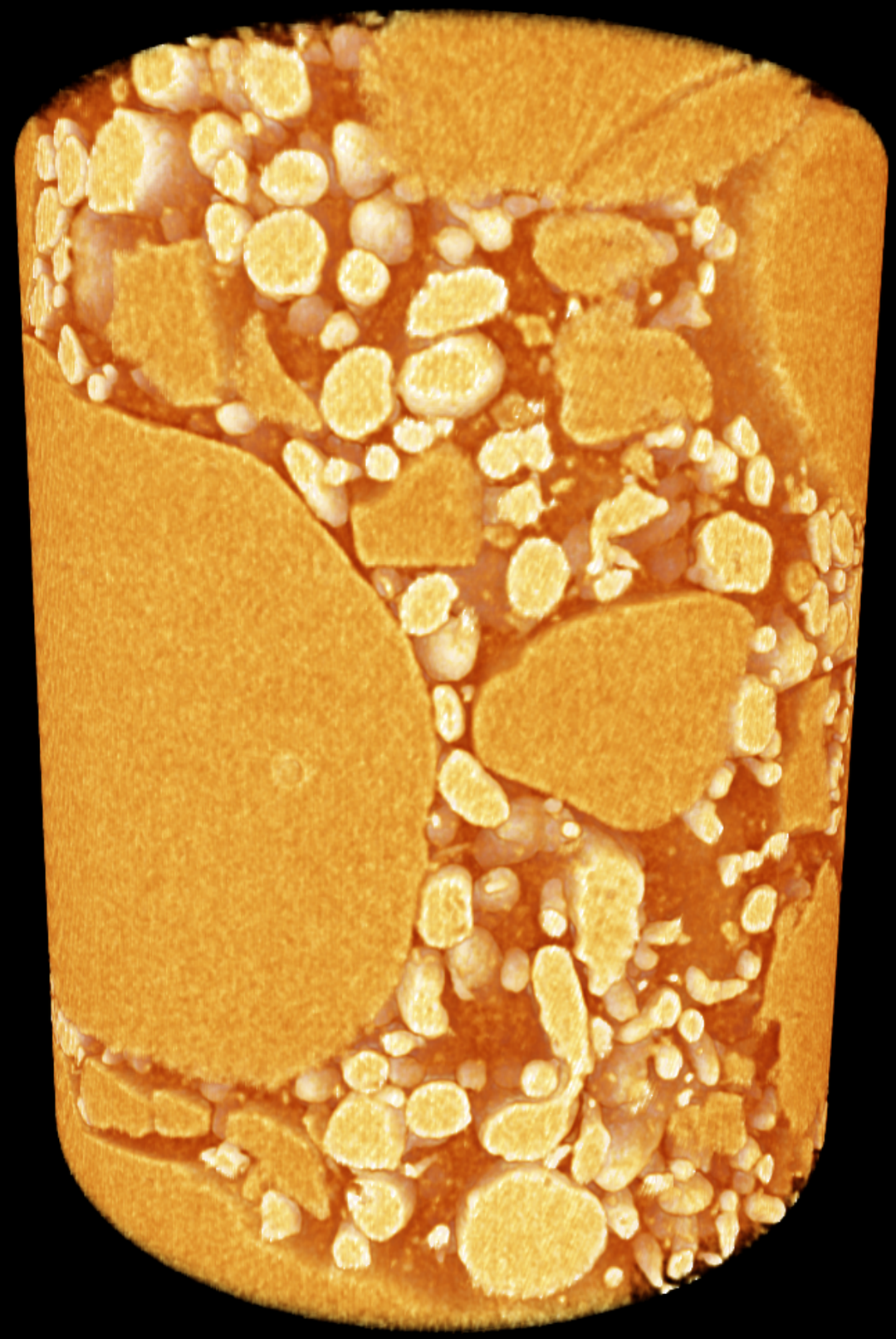
XZ
498 / 994
C 31548 W 63096



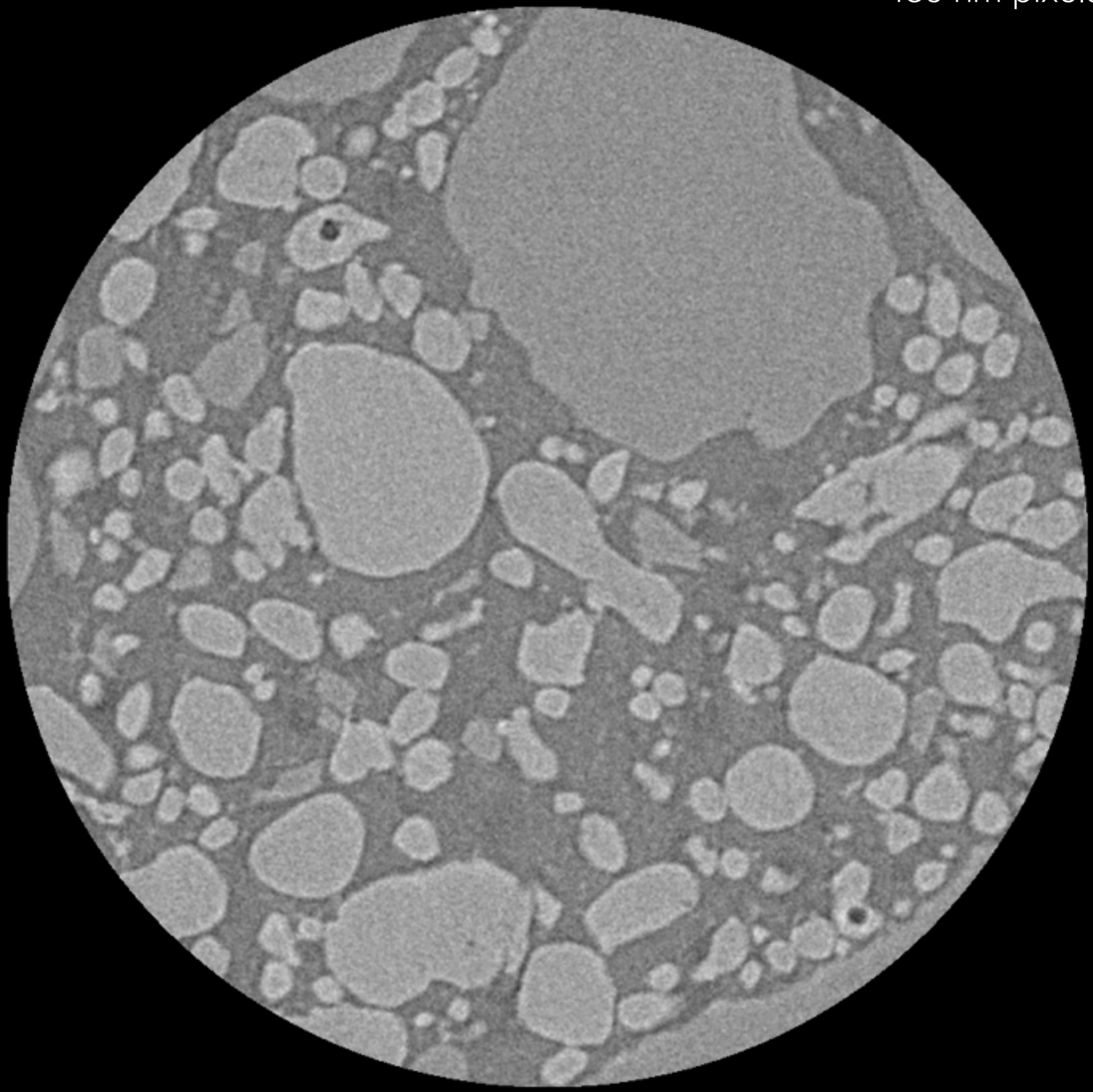
YZ
457 / 912
C 31548 W 63096



XY
481 / 960
C 31548 W 63096

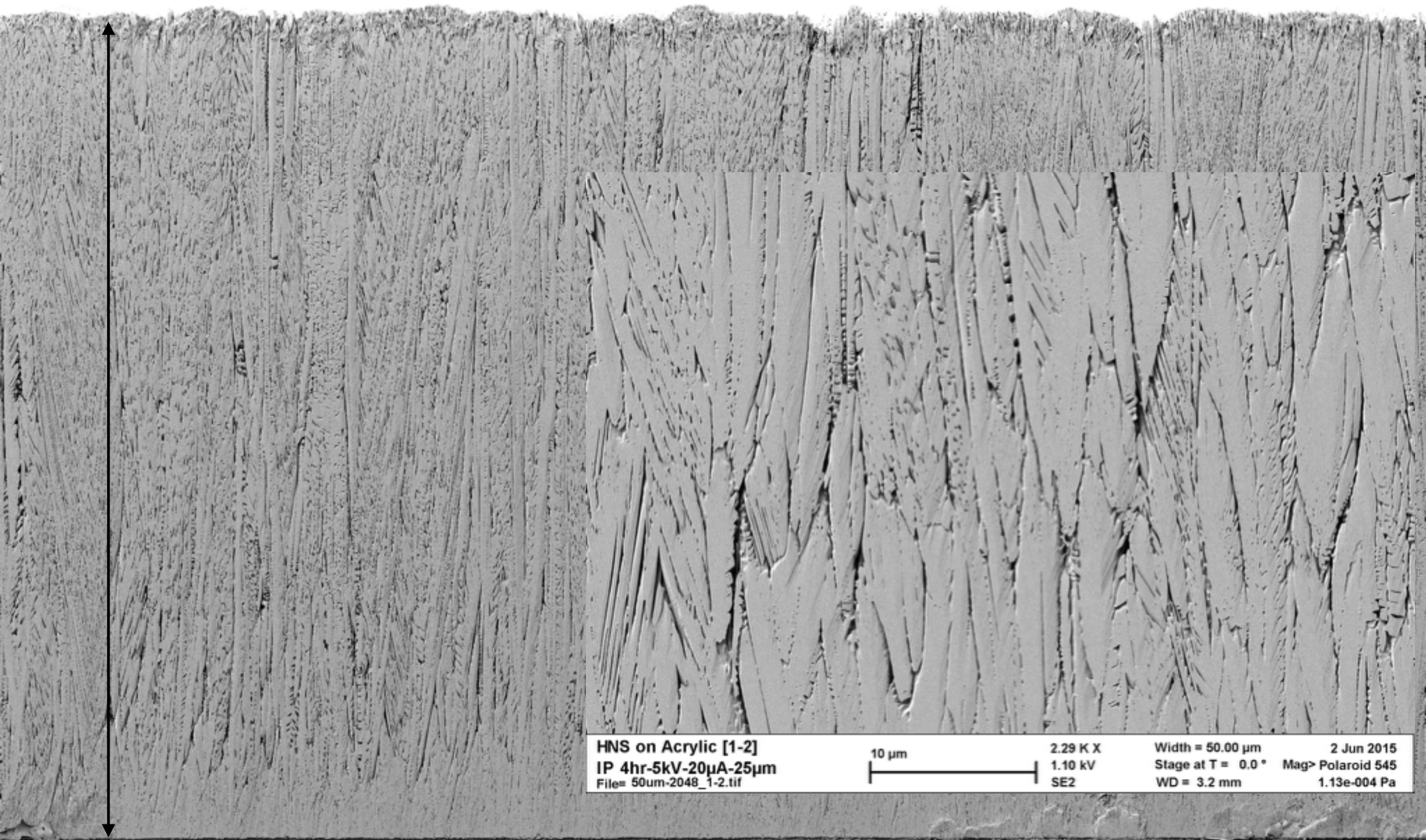


480 nm pixels, 437 um FOV



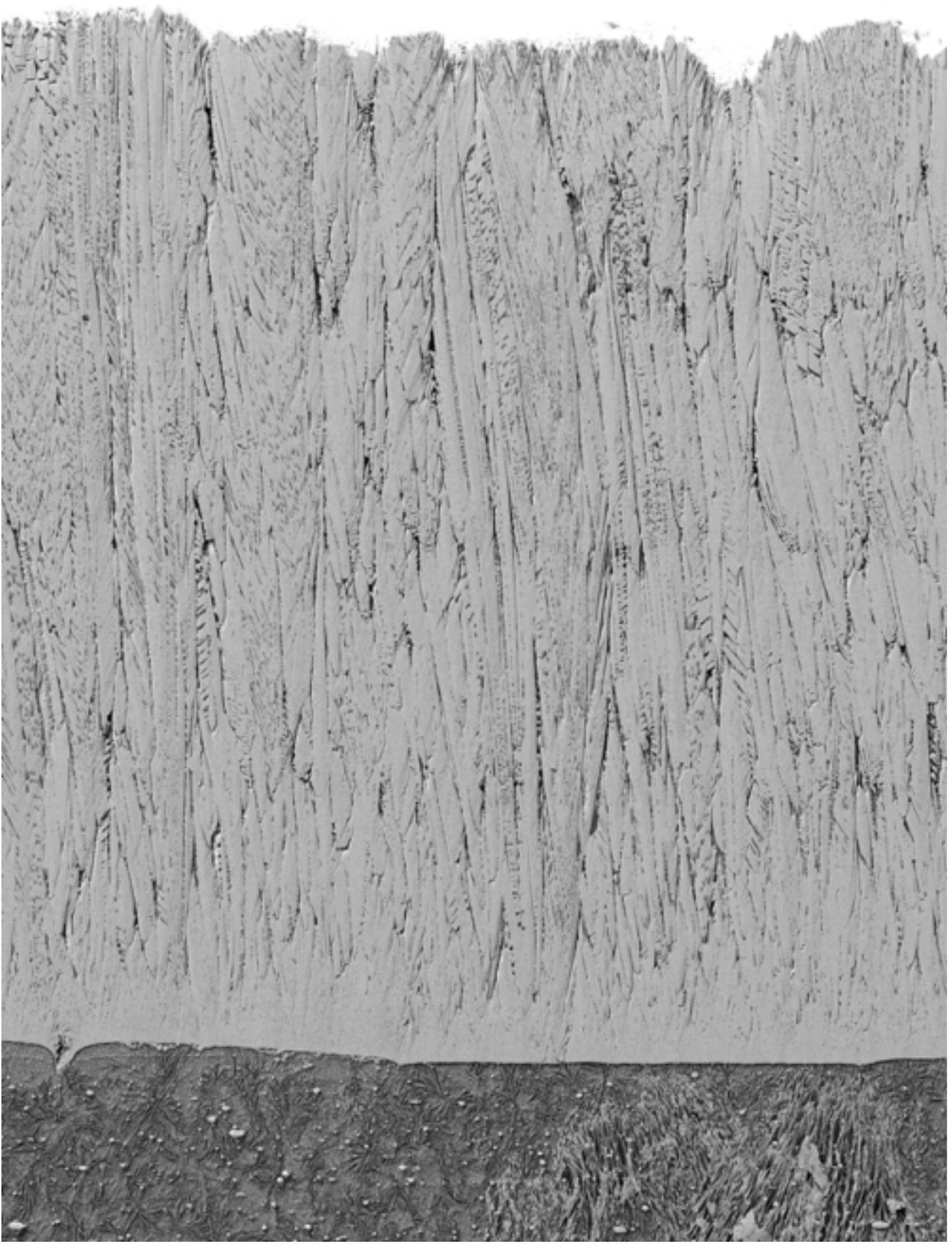
Samples: Vapor-deposited HNS (50-200um thick)

These samples are extremely cool

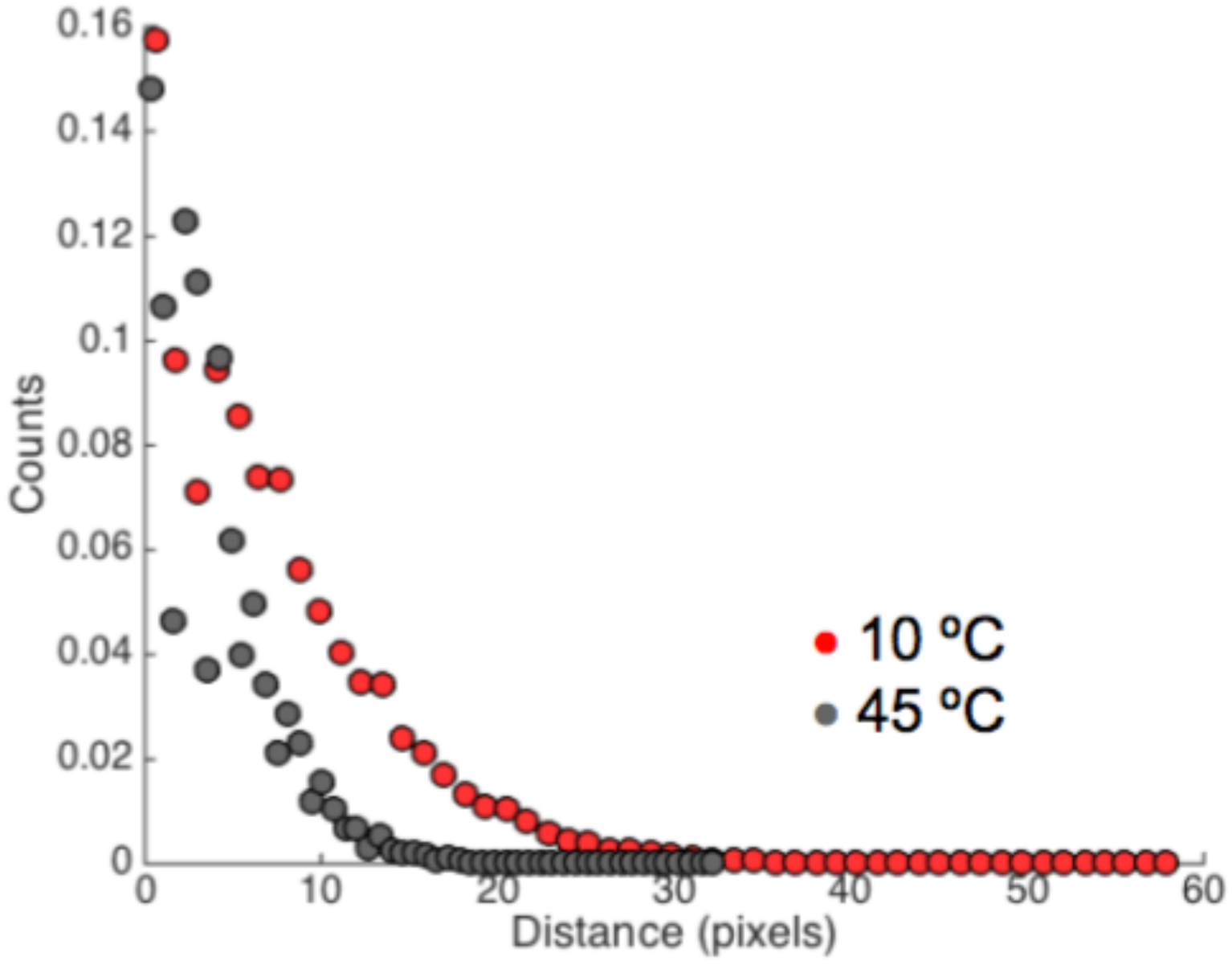


SEM of ion milled cross-section

10 C



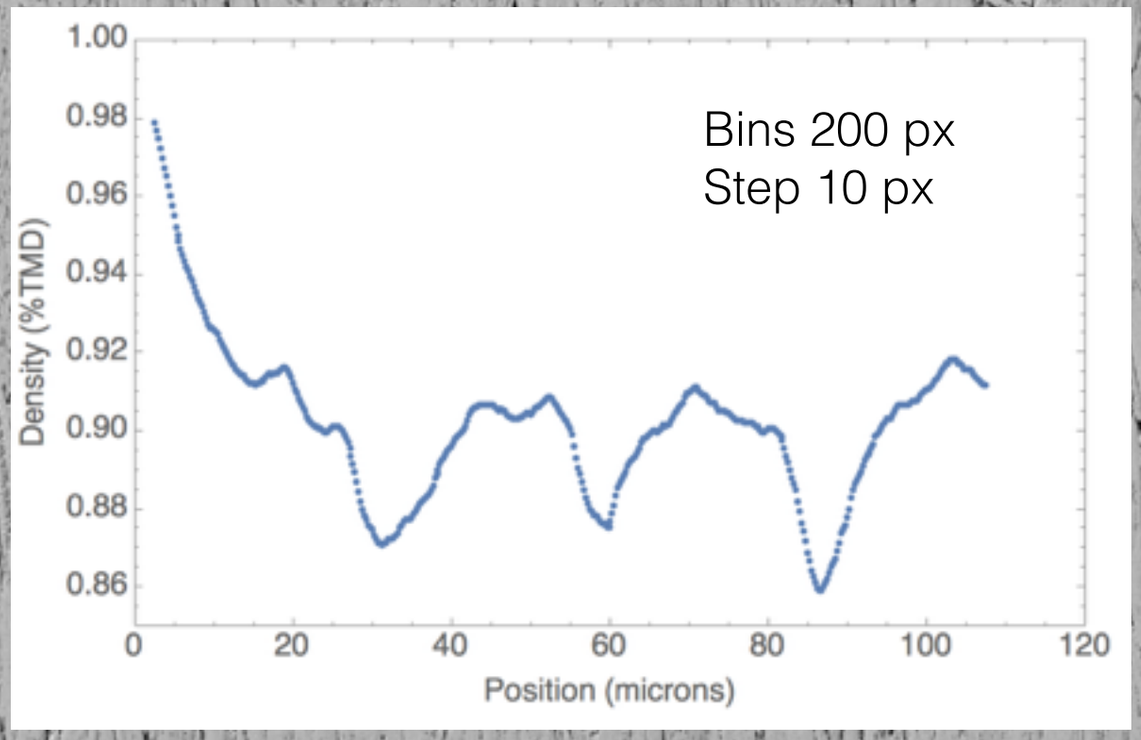
45 C



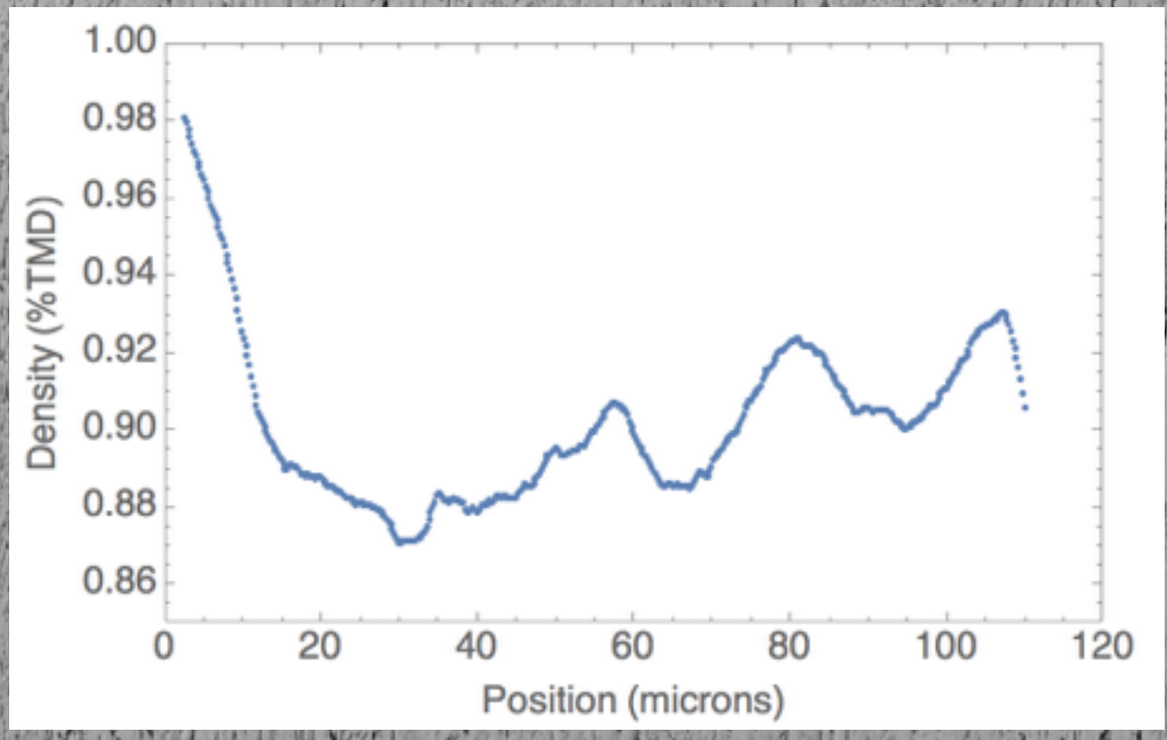
Histogram of a Euclidean distance map indicating distances from any pixel to a pore in HNS films deposited at 10 ºC and 45 ºC.

DENSITY VARIATION IN HNS FILMS

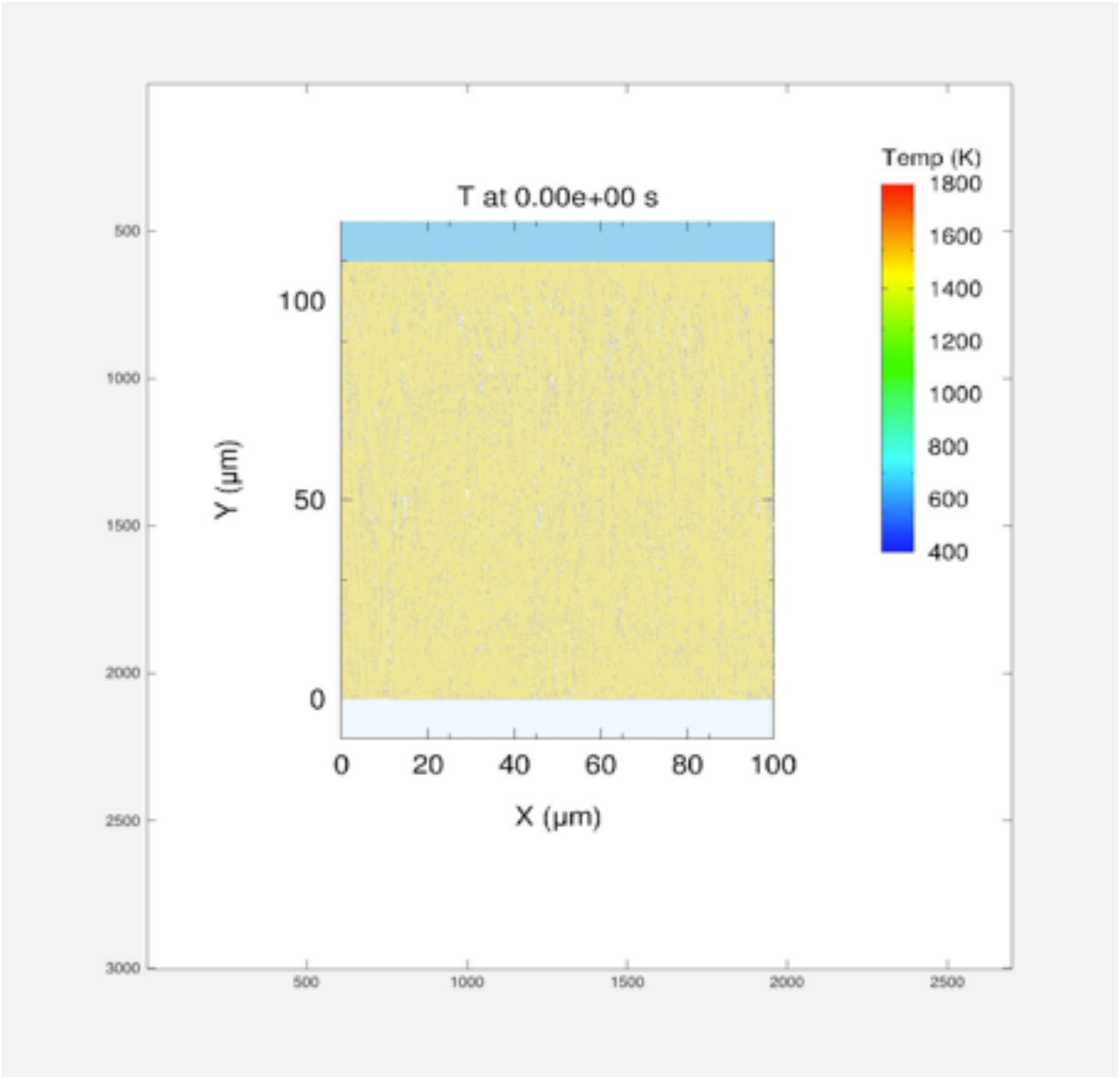
10 C

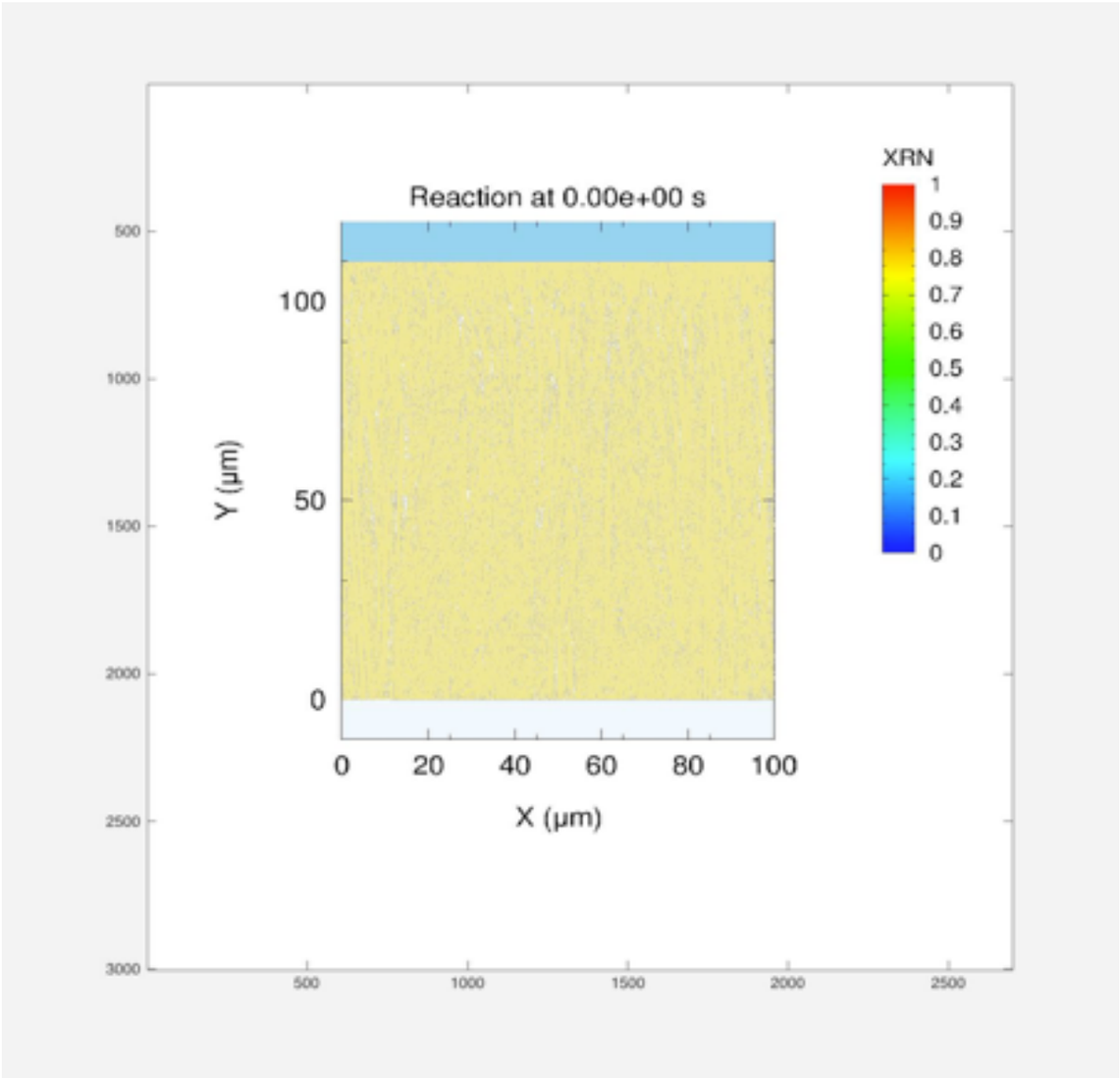


45 C



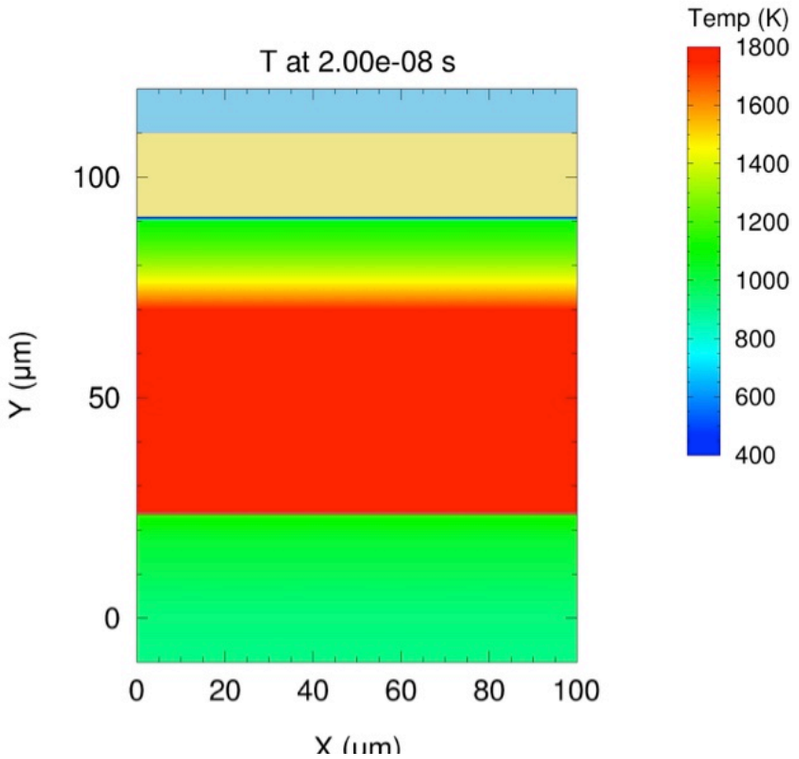
Grain-scale (mesoscale) simulation of HNS initiation

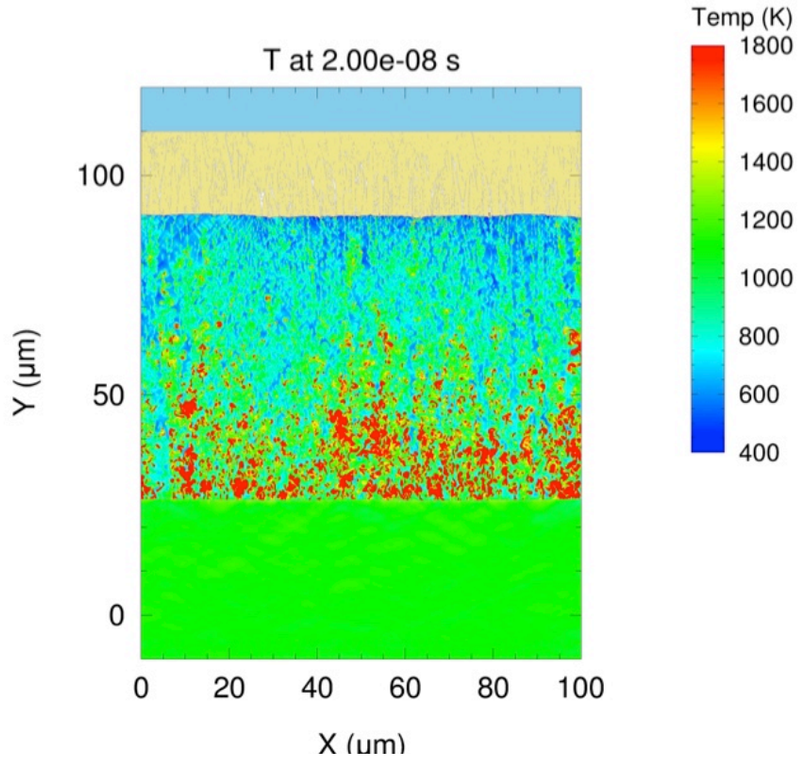




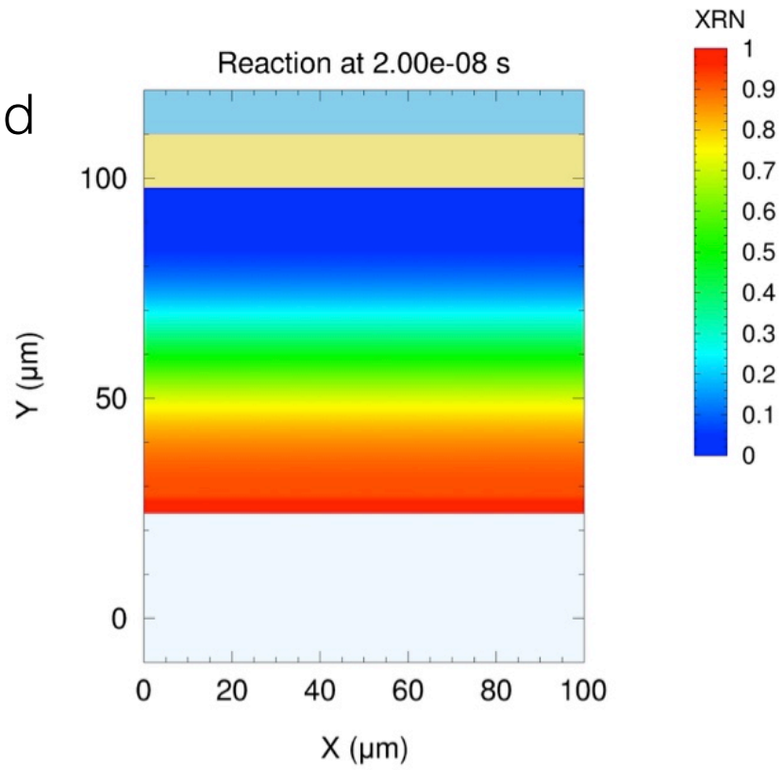
Continuum vs. mesoscale simulation, 3 km/s impact

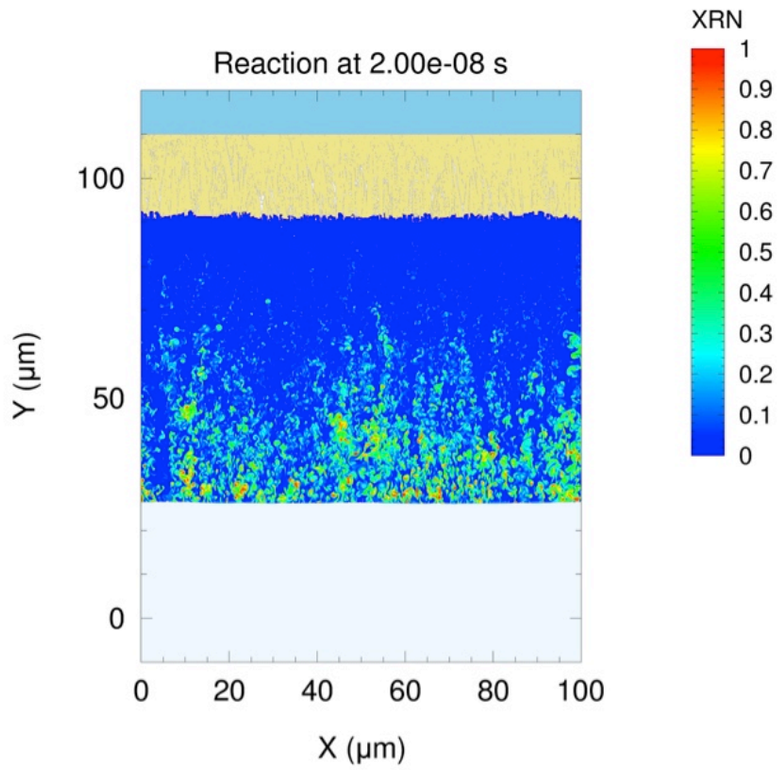
Temperature



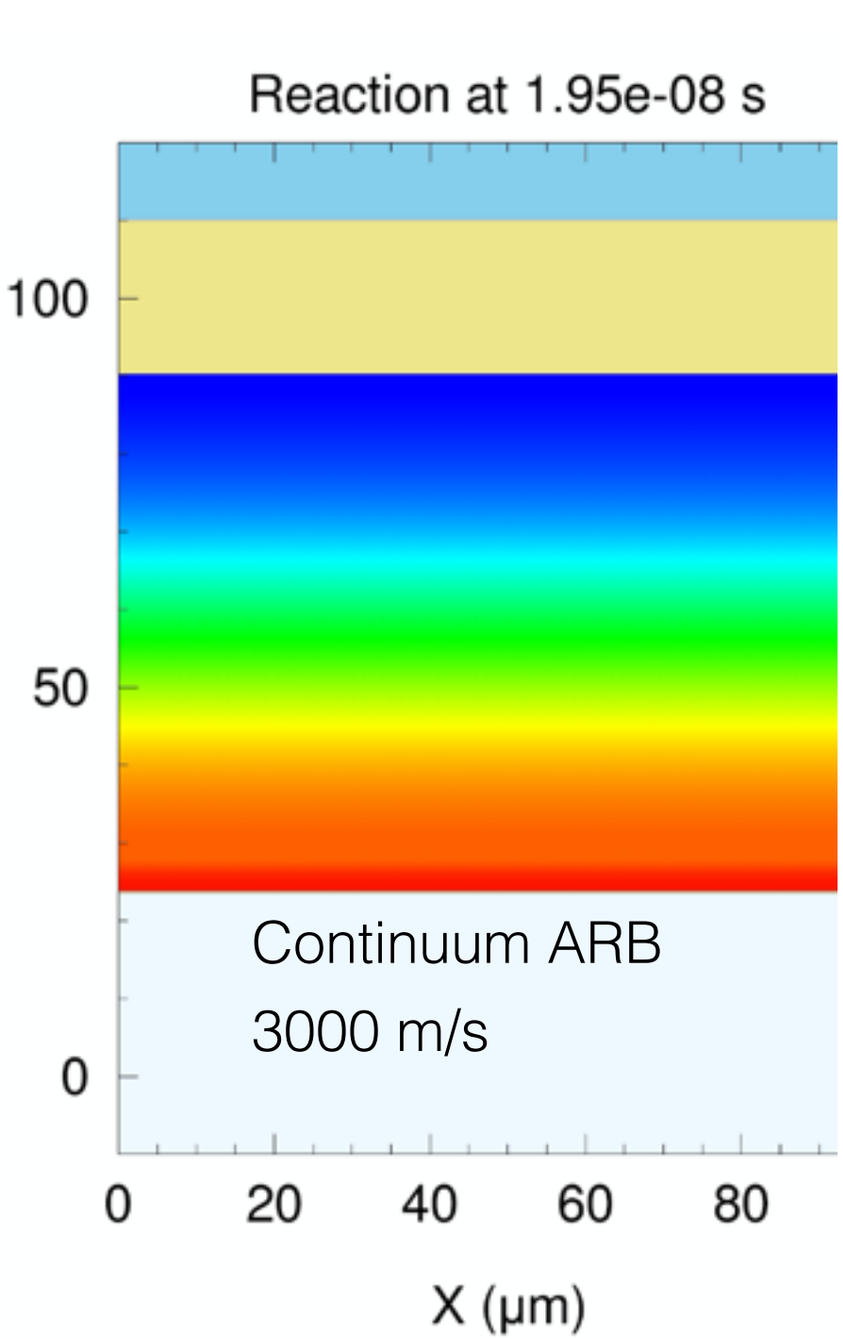


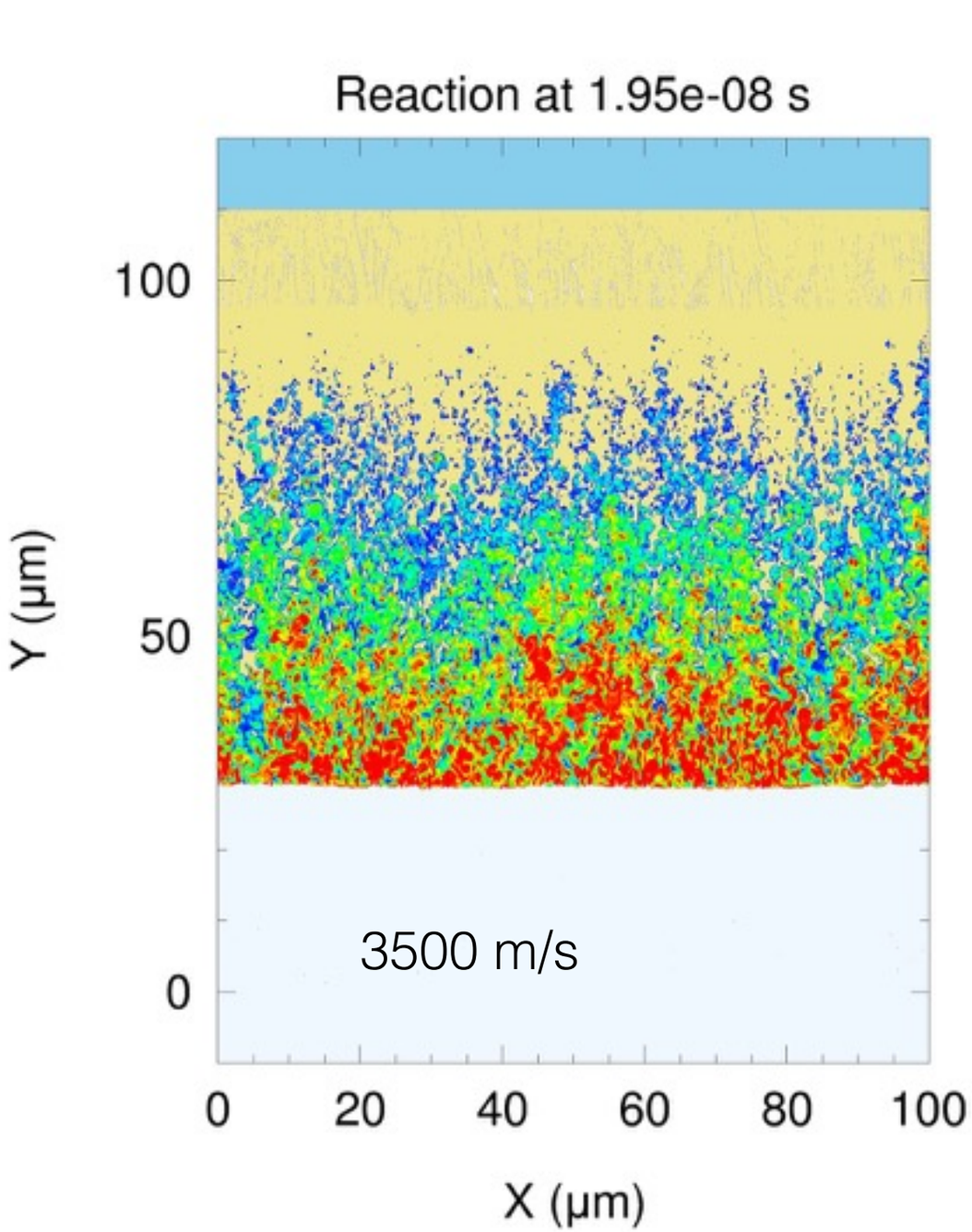
Fraction Reacted

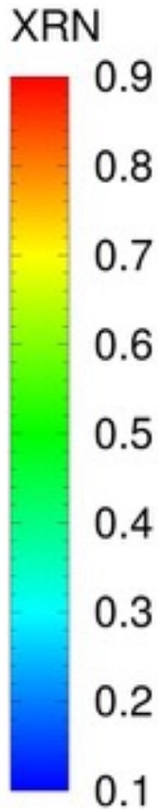




Continuum vs. Mesoscale



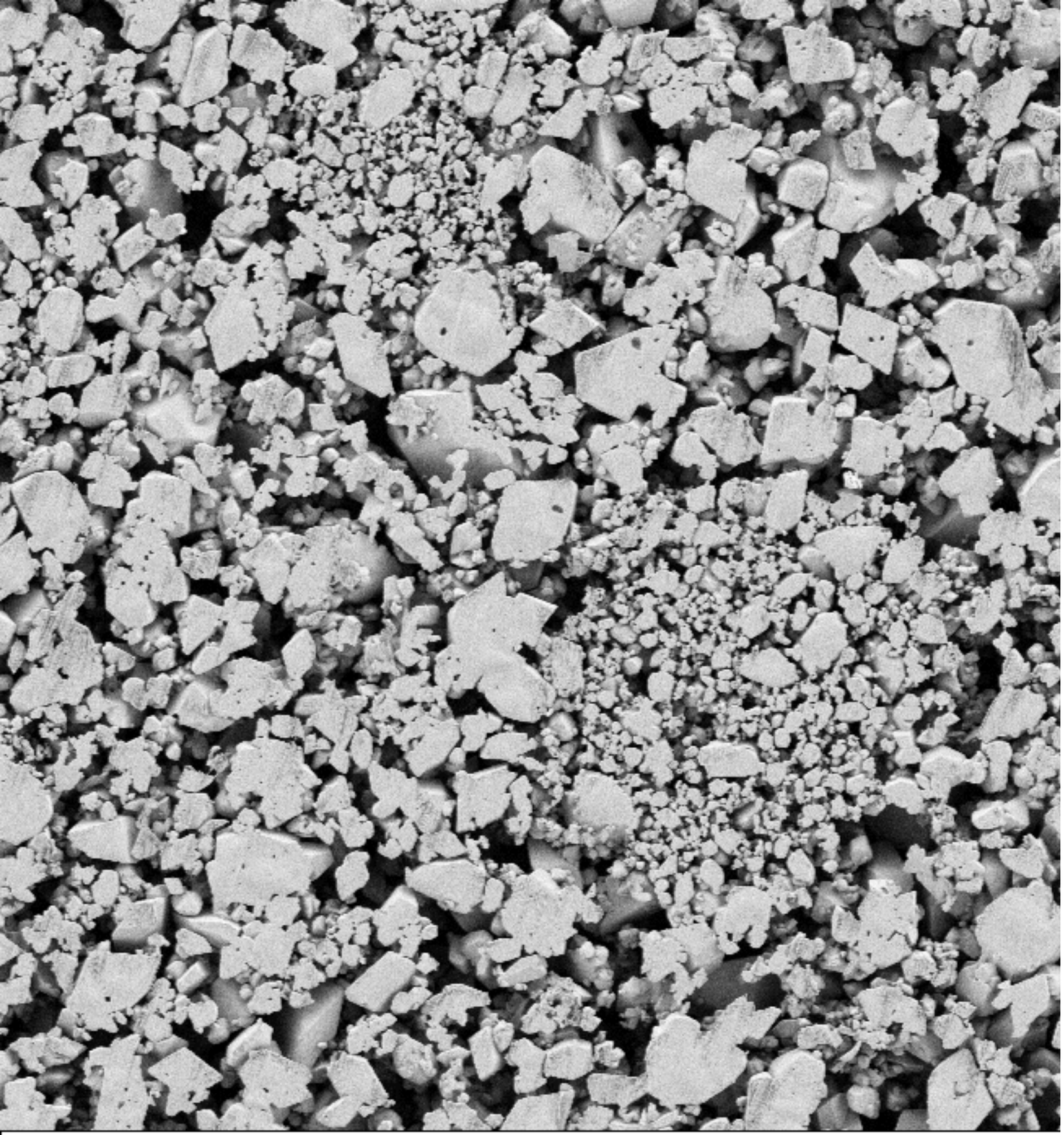




First-principles based reactants EOS (tabular)
Sesame table product EOS (historical)
Arrhenius reactive burn (ARB) with new and old parameters

Center for Integrated Nanotechnology (CINT)

www.cint.lanl.gov



Wrap-up:

- FIB/SEM, Ar Ion, xCT
- What does the modeling community need?
- What resolution and FOV?
- Resolution -> mesh
- Sample size?
- …. ?