

# Cross-scale 3D characterisation of complex and heterogeneous geomaterials with X-ray micro-CT

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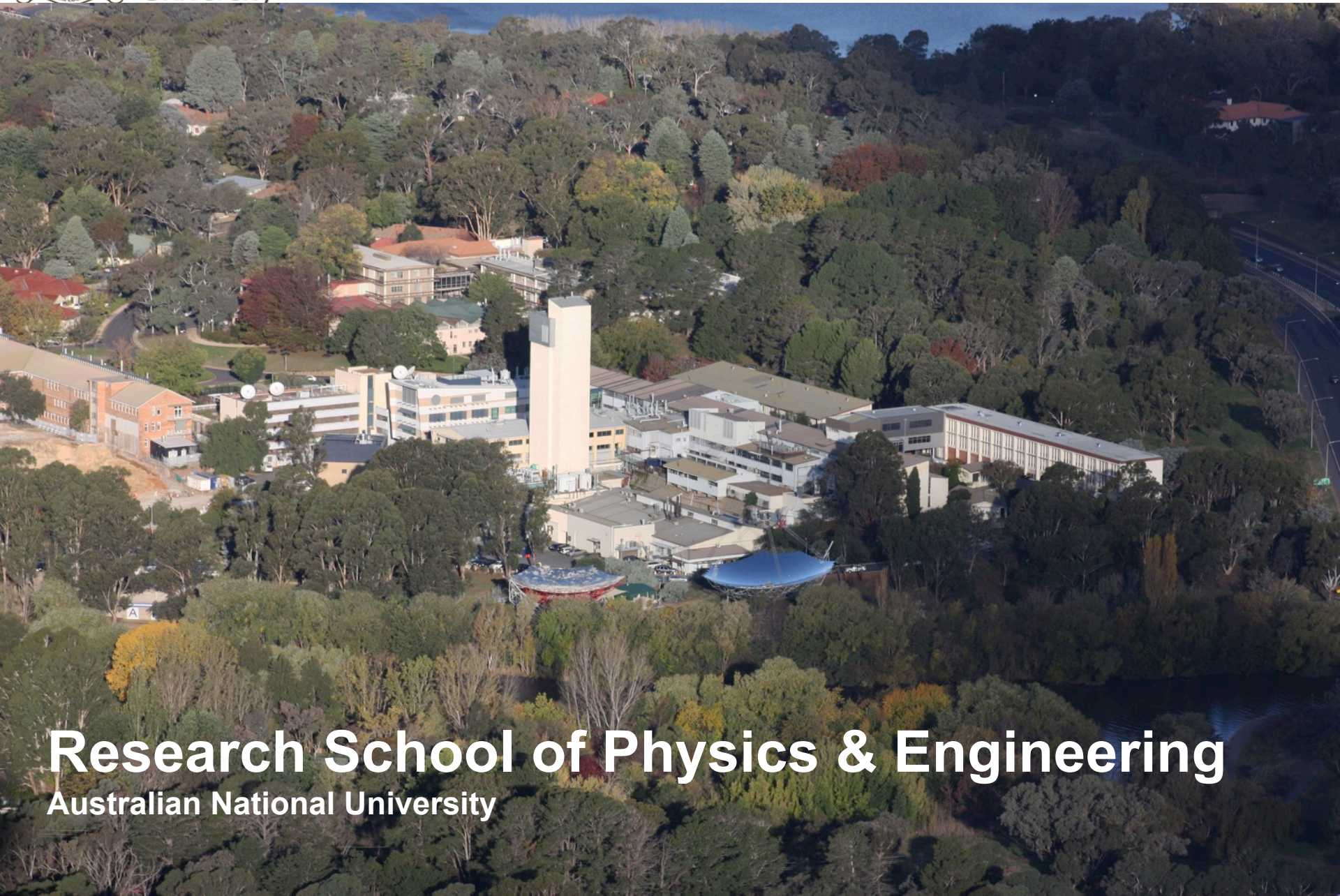
<sup>2</sup>*FEI Australia*

<sup>3</sup>*School of Petroleum Engineering, University of New South Wales, Sydney, Australia*



- **Imaging techniques**
  - Improving quality: new scanning and reconstruction techniques
  - Dynamic tomography
- **Image analysis**
  - Towards automated segmentation
  - Image registration
- **Application examples**
  - Correlating SEM-EDX mineral mapping with x-ray uCT
  - Imaging diffusion in shale
  - Iodine labelling of sub-resolution surface area
  - Cross-scale mapping of permeability in heterogeneous sandstones





# Research School of Physics & Engineering

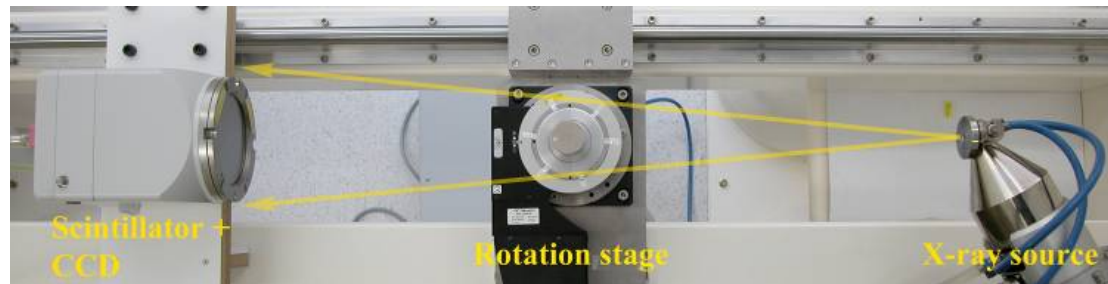
Australian National University



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# Instrument development at ANU



**2001**

- 3 $\mu$ m voxels, <60mm FOV
- CCD detector
- circular scanning



**2014:**

- 1  $\mu$ m / 2–40mm
- flat panel detector
- helical scanning
- licensed to FEI

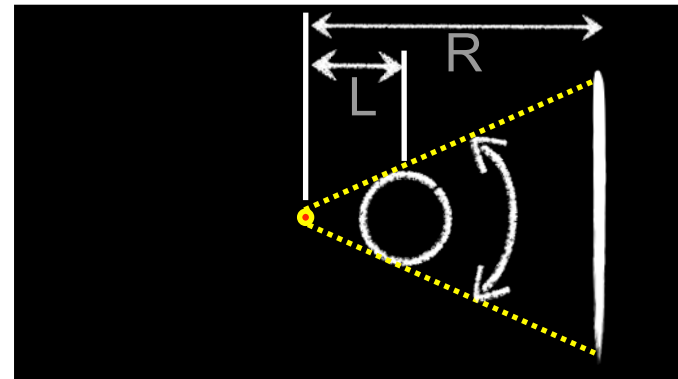
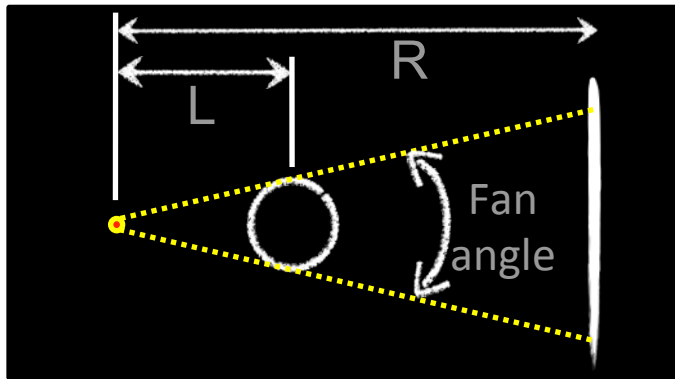
**2015:** 'nano' CT:  
300 nm / 0.7mm

**2017:** whole core  $\mu$ CT  
50  $\mu$ m / 100 mm



# Helical-scanning cone-beam micro-CT – why?

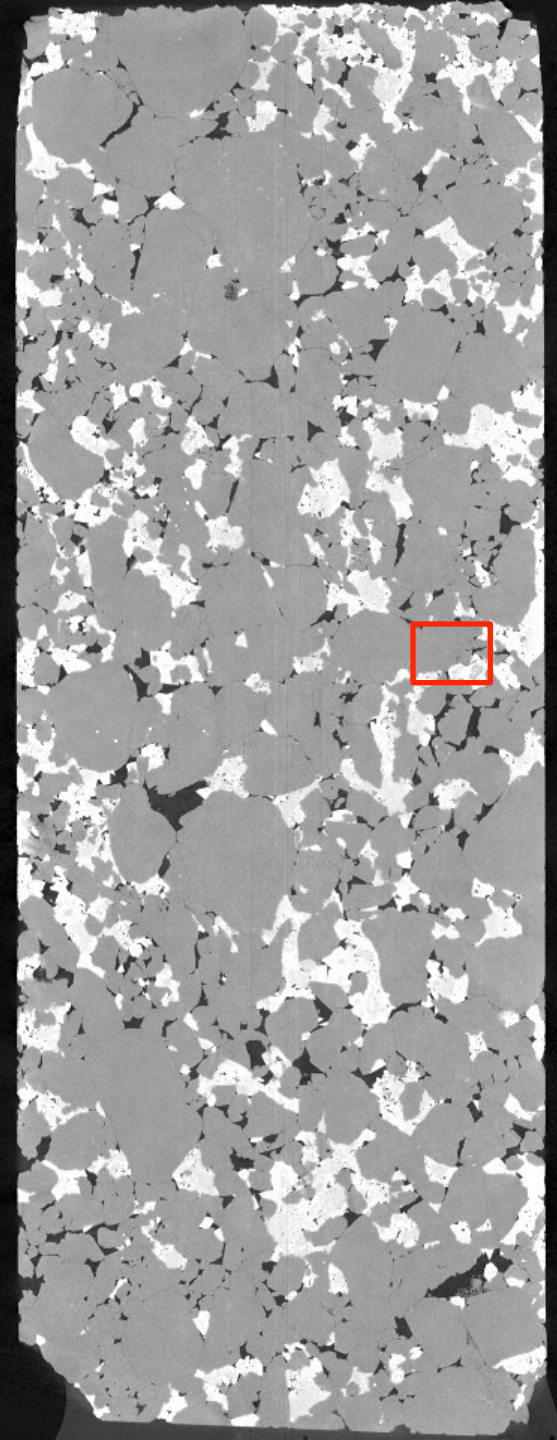
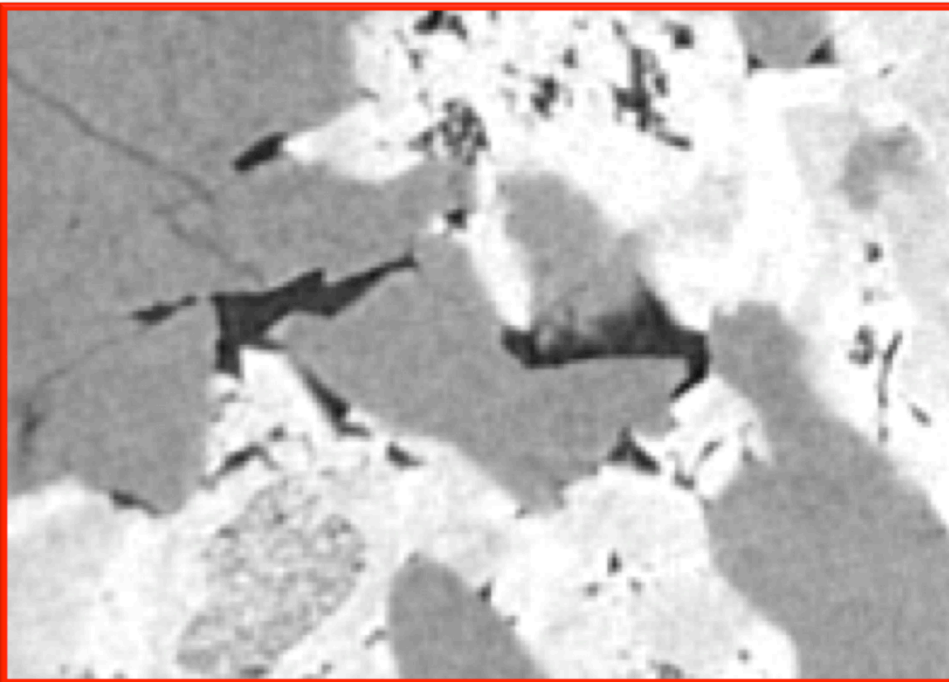
- Image noise in tomography is entirely due to finite photon numbers
- X-ray sources emit in all forward directions, can increase photon flux simply by moving sample and detector closer to the source



- Circular scanning trajectory does not provide sufficient data for high cone-angle acquisition
- Helical scanning data is “complete”, allows exact reconstruction techniques such as FBP method of Katsevich

## Helical cone-beam micro-CT

- cone angles up to  $60^\circ$
- “Autofocus” techniques critical for micron scale
- Order-of-magnitude improvement in acquisition times, for no loss of SNR
- Images up to 18000 x 3000 x 3000 (so far)





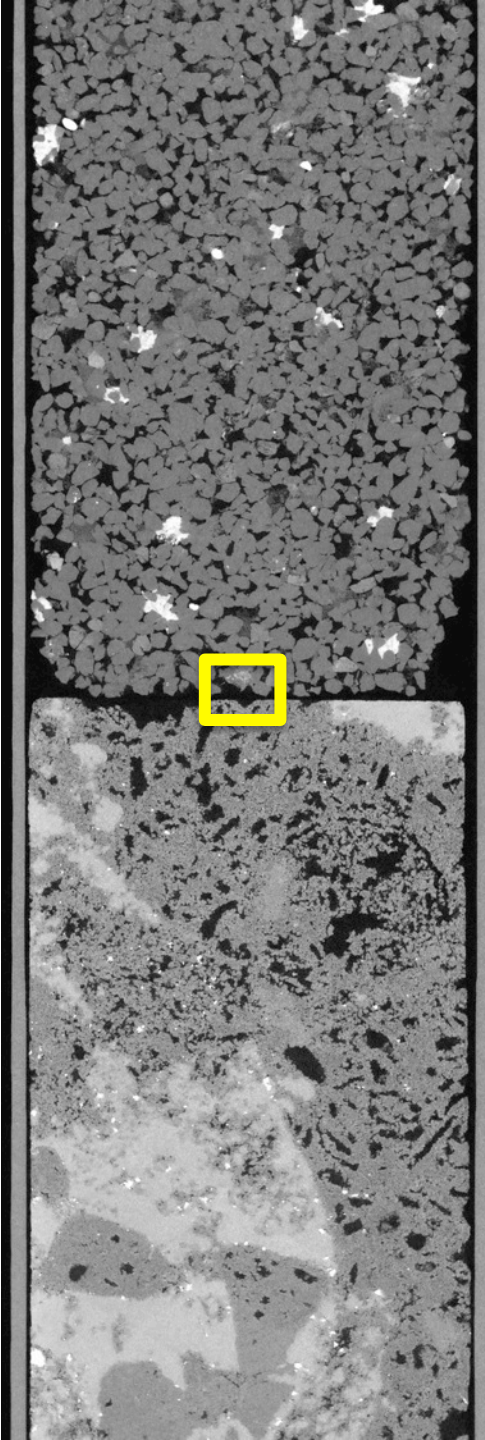
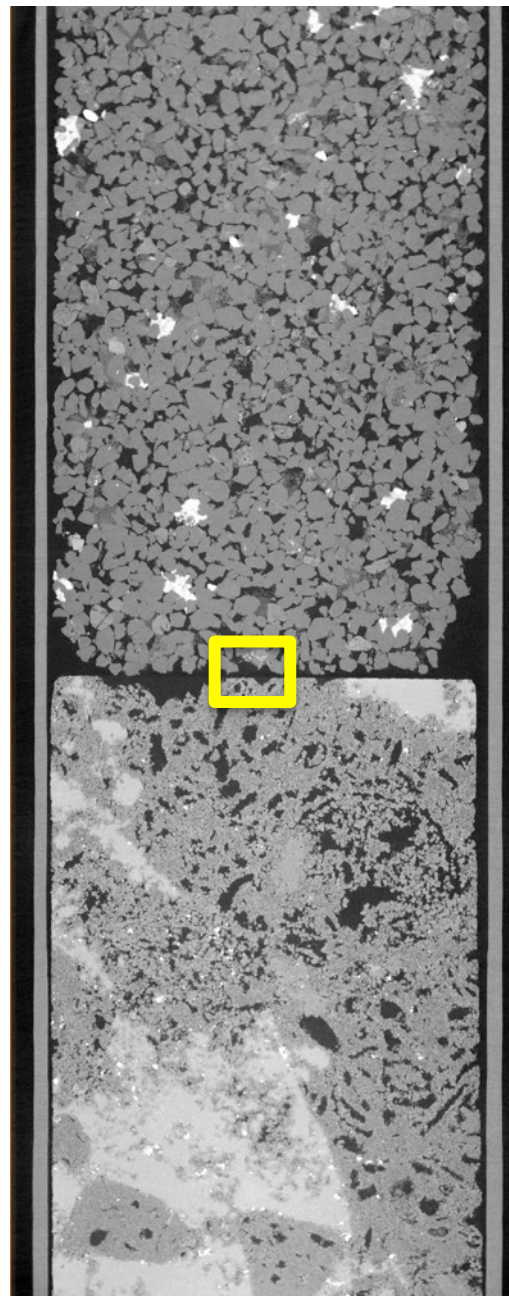
## Large-scale iterative reconstruction

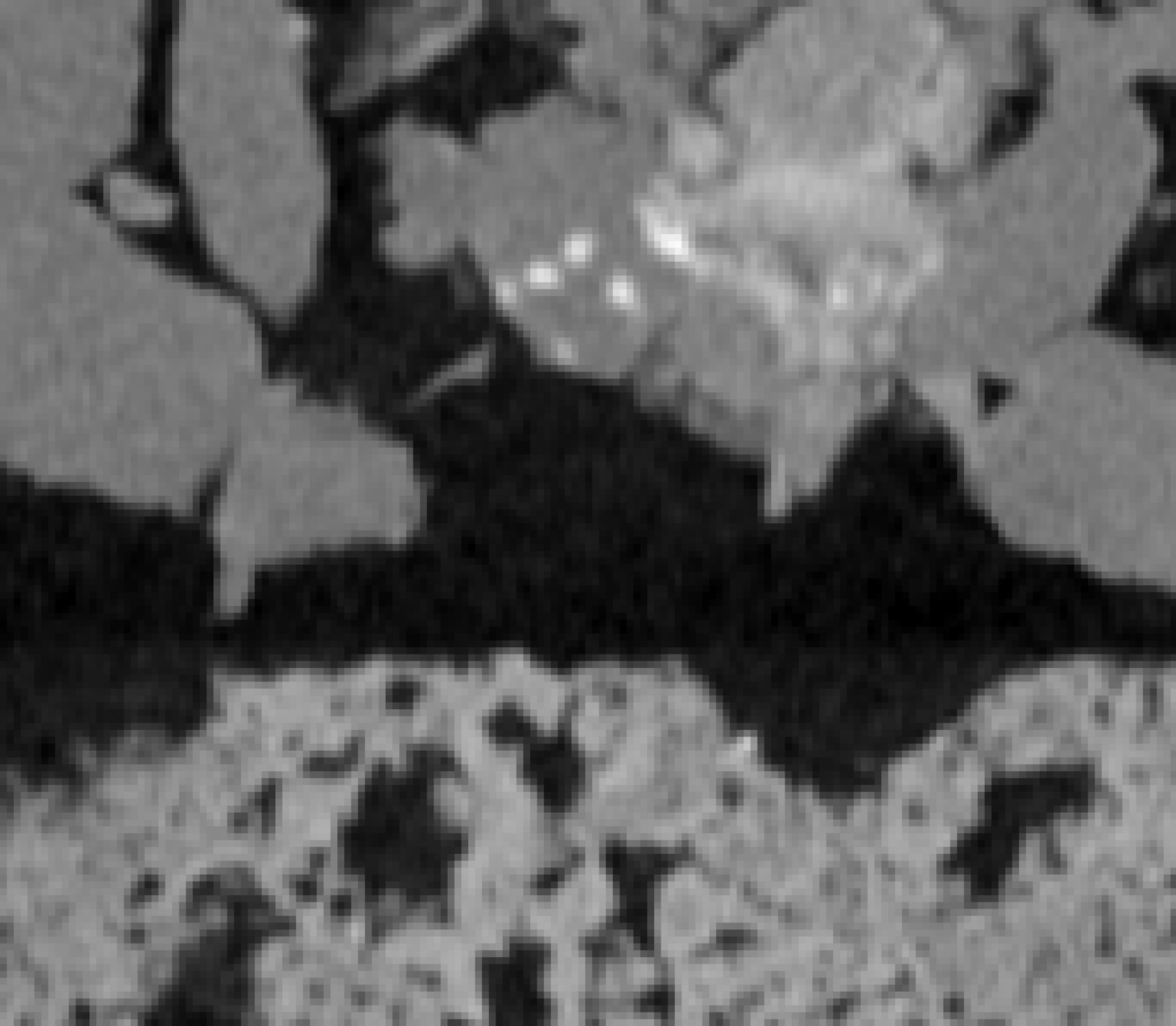
Iterative reconstruction  
(*reconstruction-by-optimisation*)  
is needed for advanced  
tomography.

**Problem:** too computationally  
demanding for big data

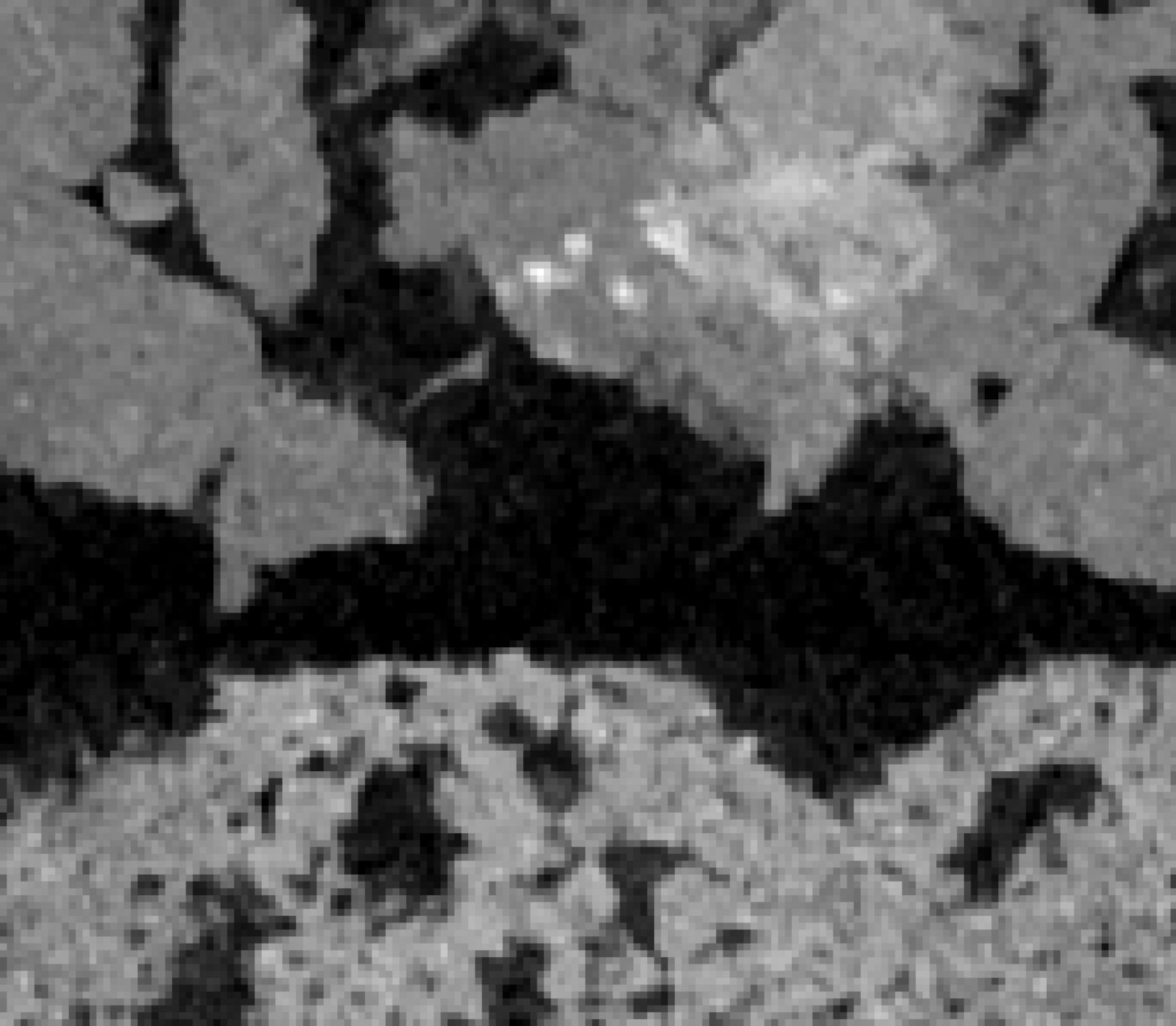
**Solution:** Multi-resolution  
iterative reconstruction;  
converges in two multi-grid  
iterations

Demonstrated on 40 Gvoxel  
images.



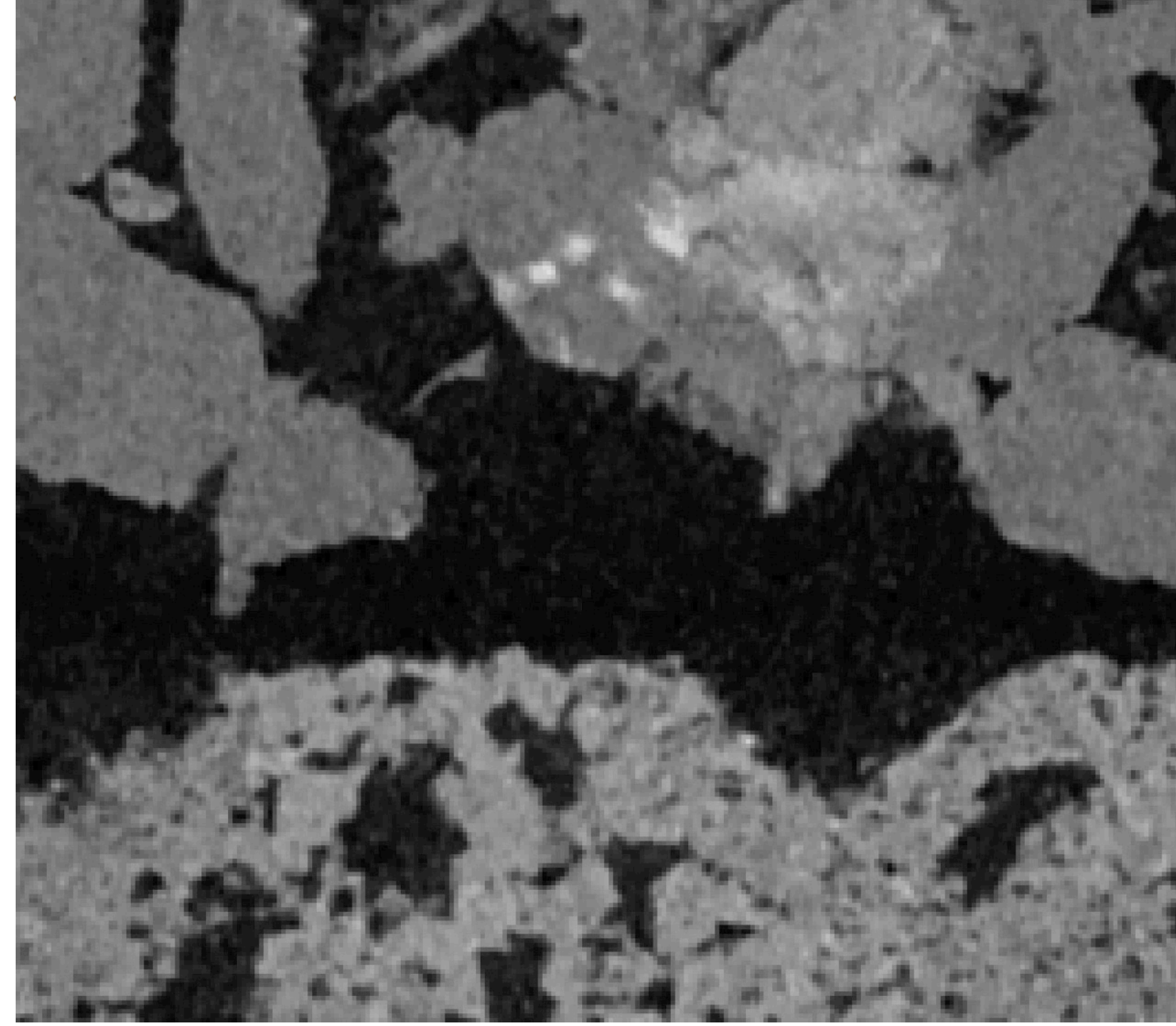


**FBP**



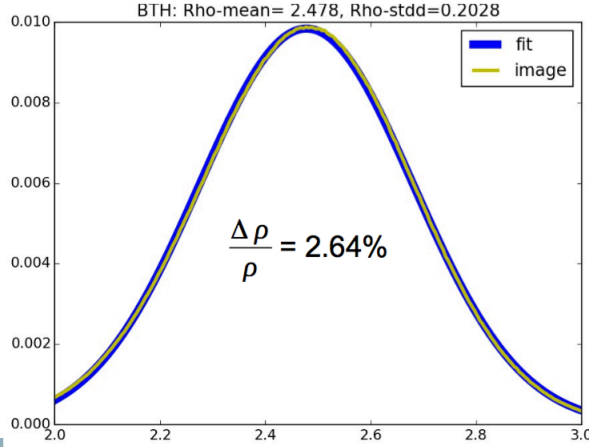
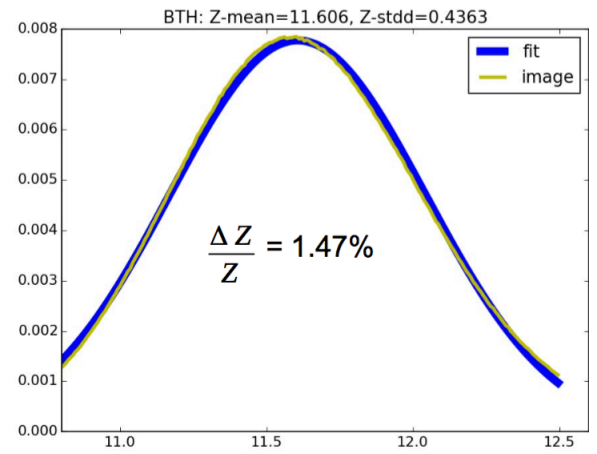
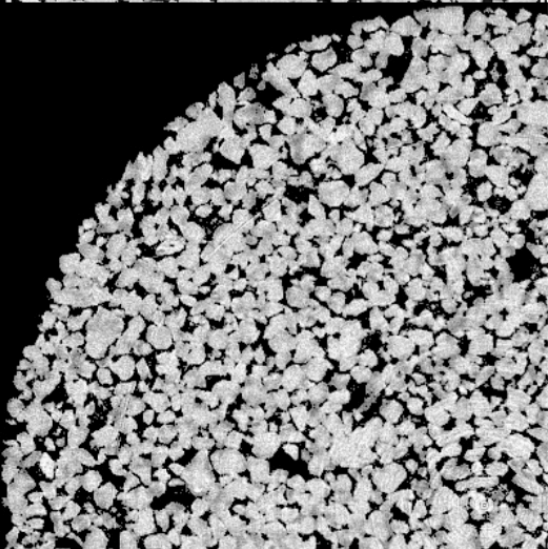
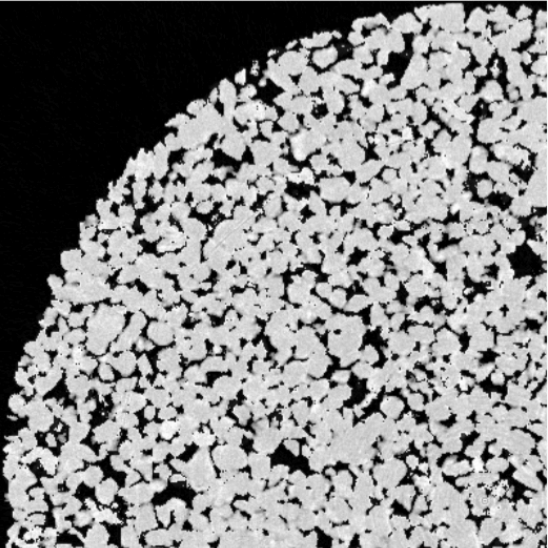
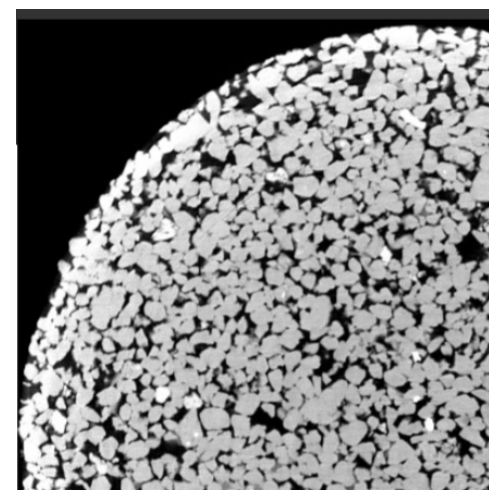
**MG**  
**Iterative**



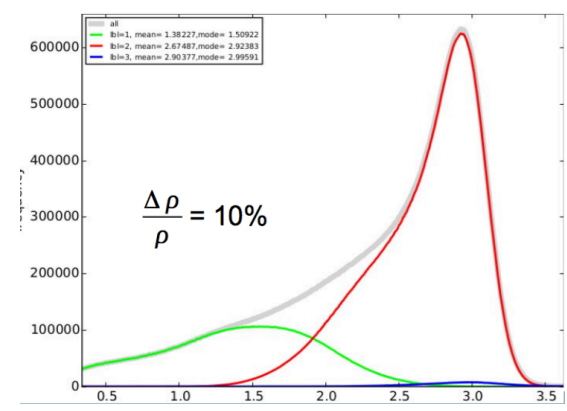
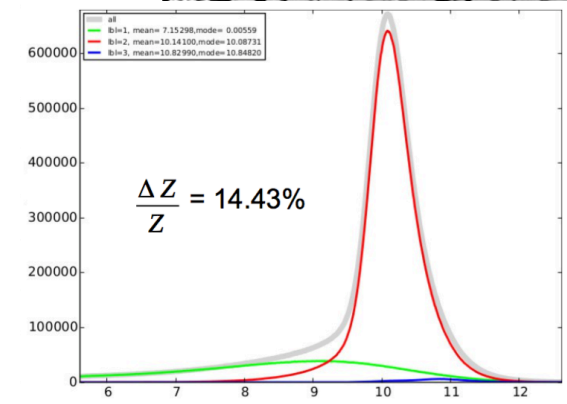


**MG**  
**Iterative**  
1.5x higher  
resolution

# Density and Atomic Number From dual energy imaging



Alvarez-Makowski



Siddiqui-Khames

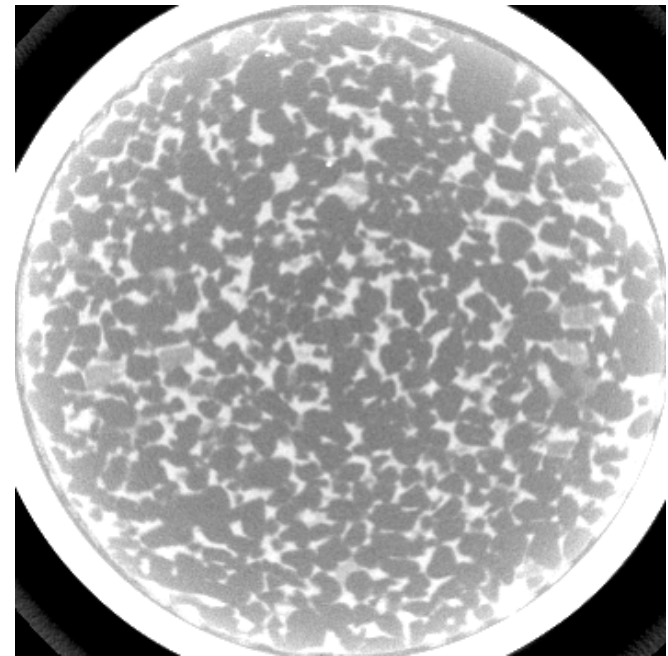


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# Dynamic Tomography

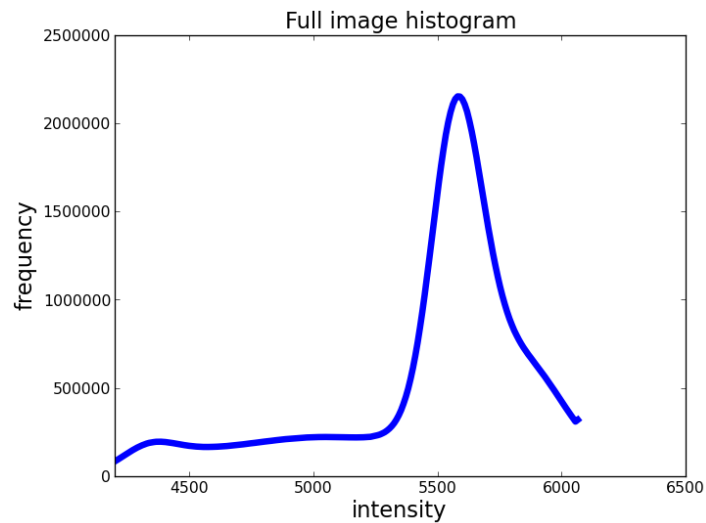
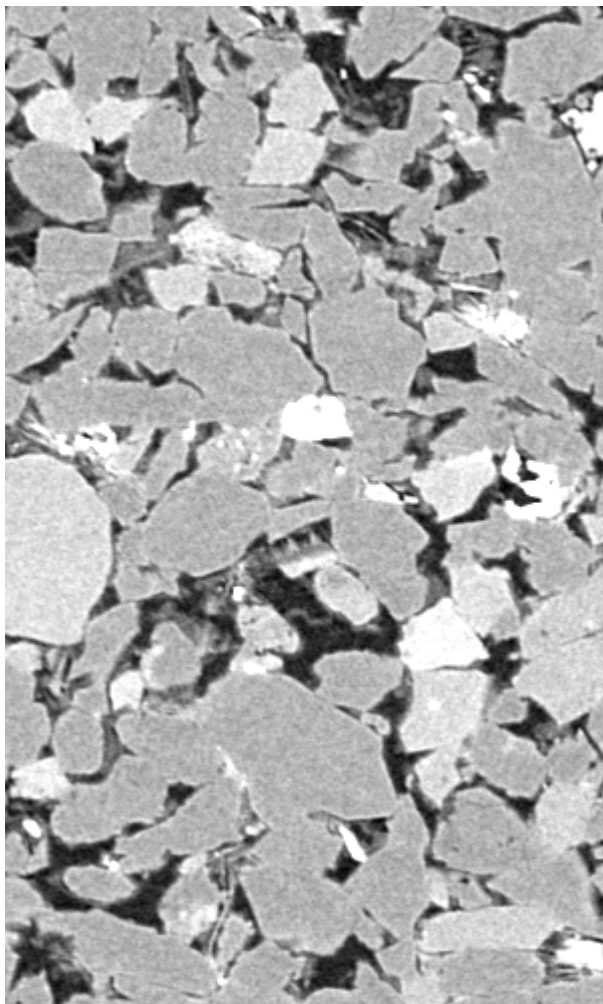
- MPEG compression greatly reduces movie file sizes: the information required to encode the *changes* from one moment to the next is much less than that required to encode each frame in isolation
- Similarly, when doing 4D tomography to capture dynamic processes, one should need far fewer projections to reconstruct just the changes between successive frames.
- Two-phase immiscible fluid flow is a good candidate for this, since it is geometrically constrained
- Can incorporate these constraints into iterative reconstruction techniques





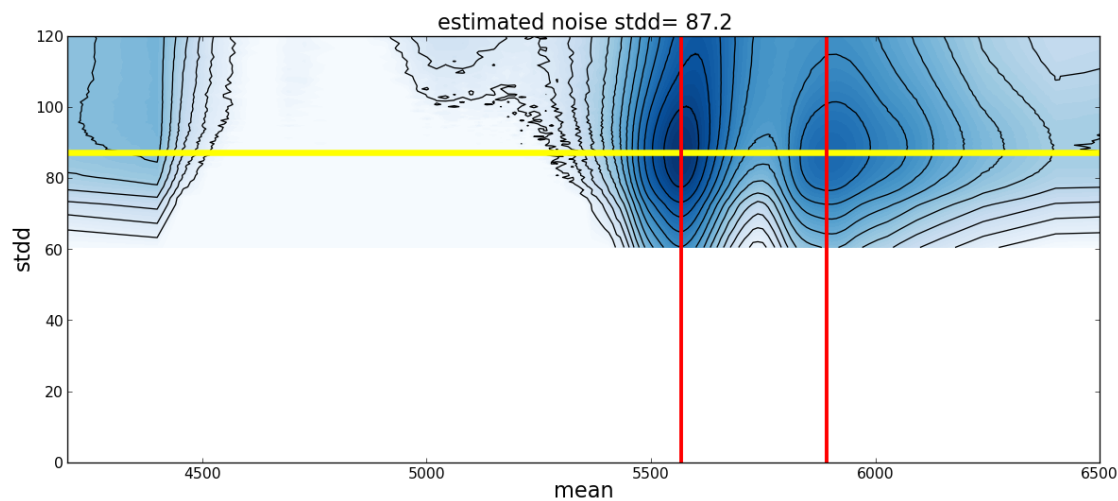
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# Bilateral Filter: Estimating Noise

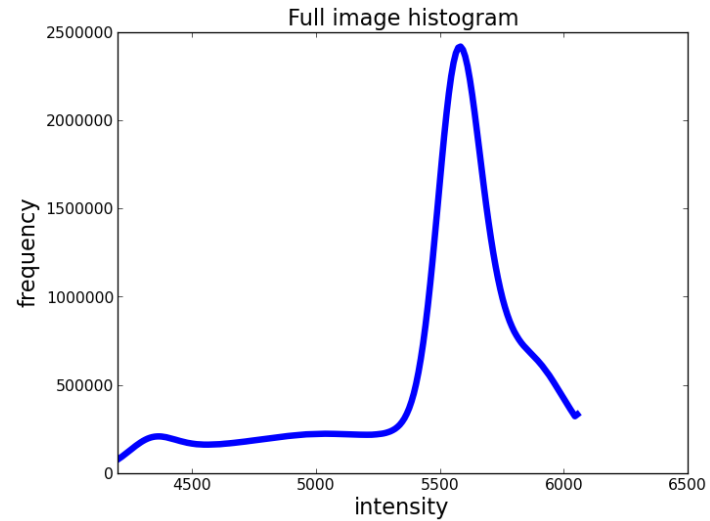
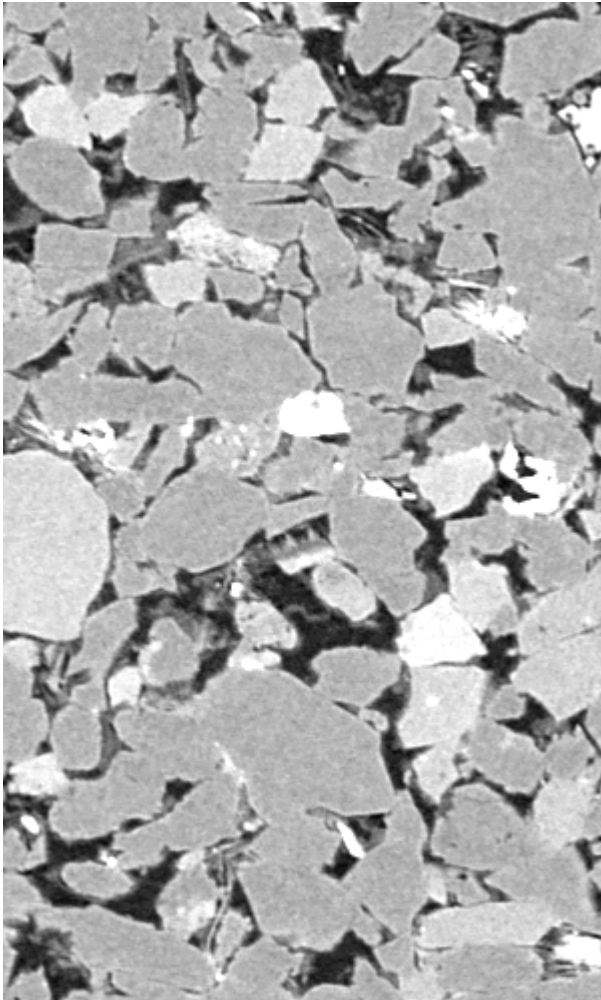


Original image

noise = 87.2

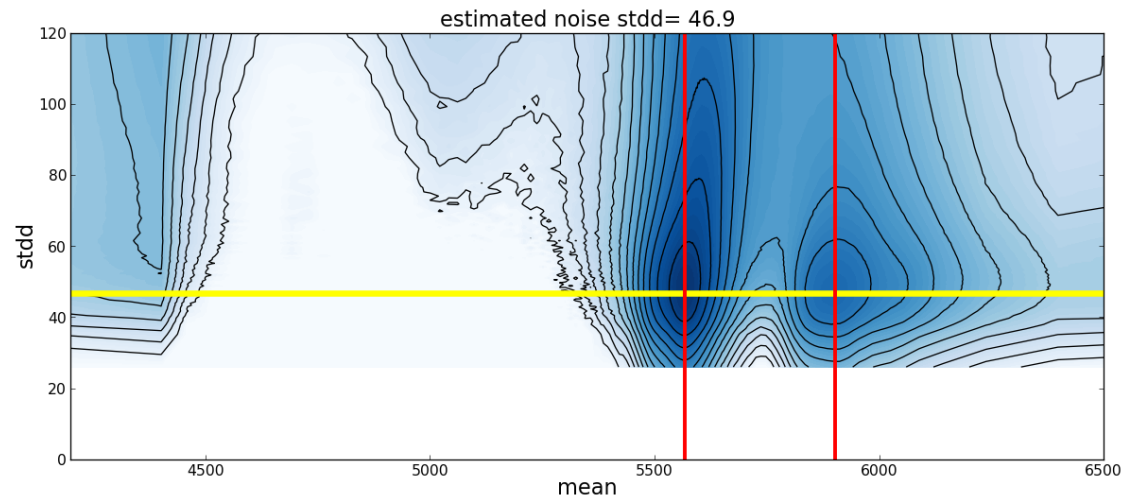


# Bilateral Filtering: using estimated noise



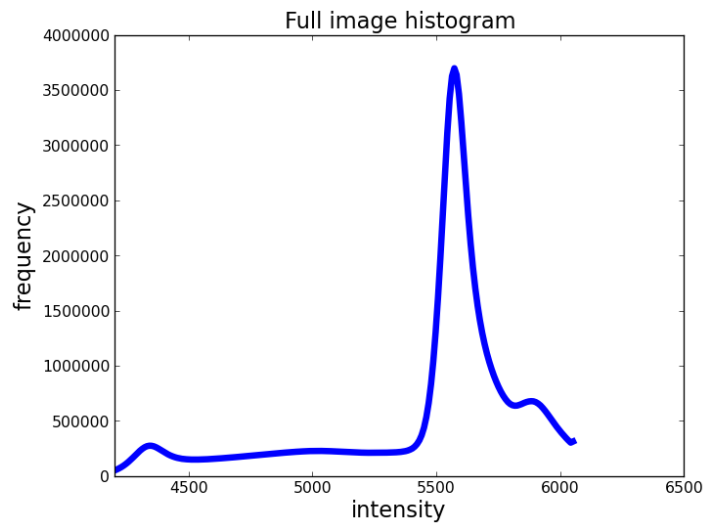
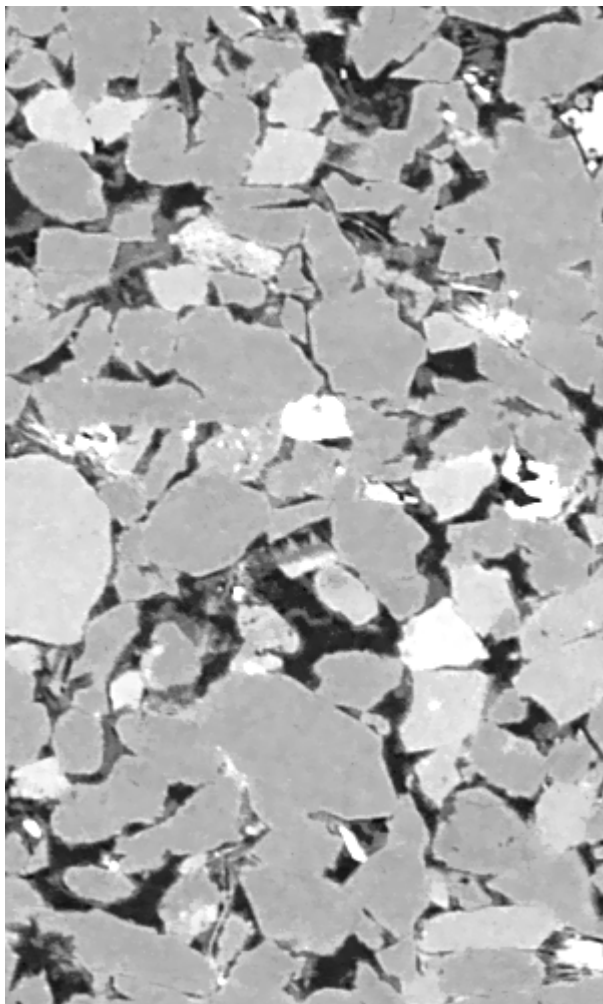
Iteration 1

noise = 46.9



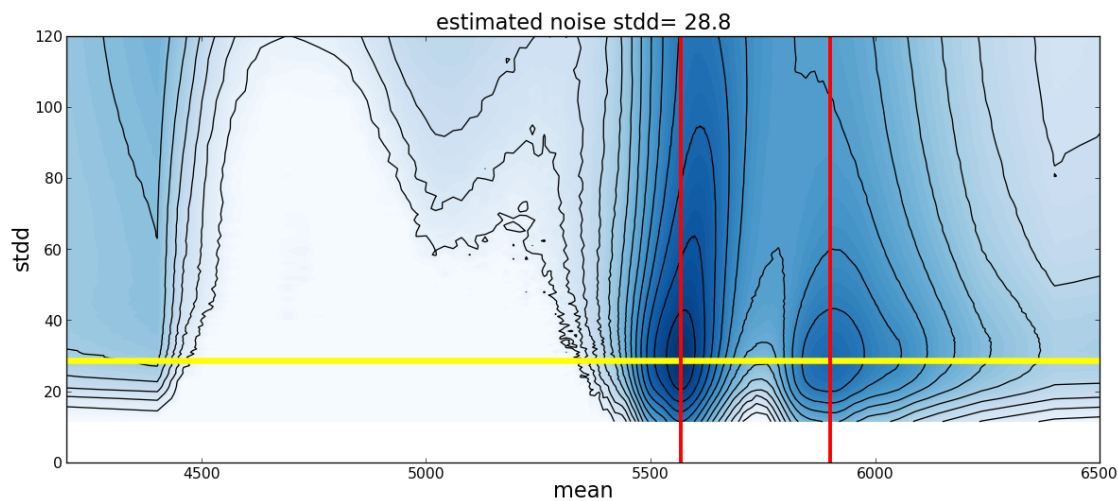


# Bilateral Filtering using estimated noise

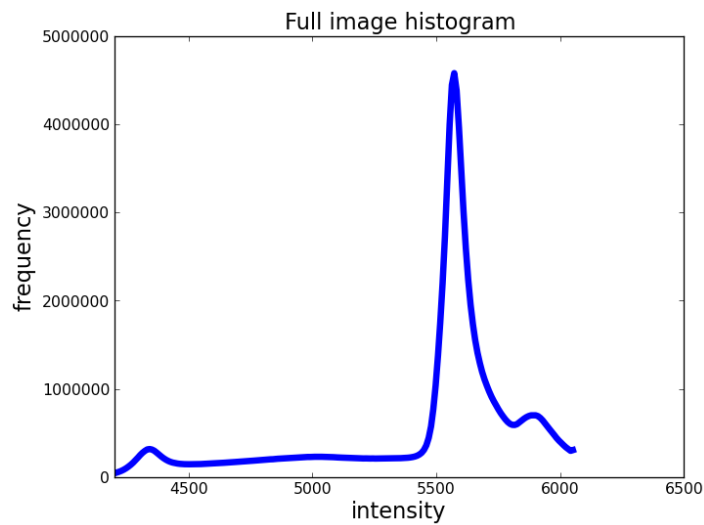
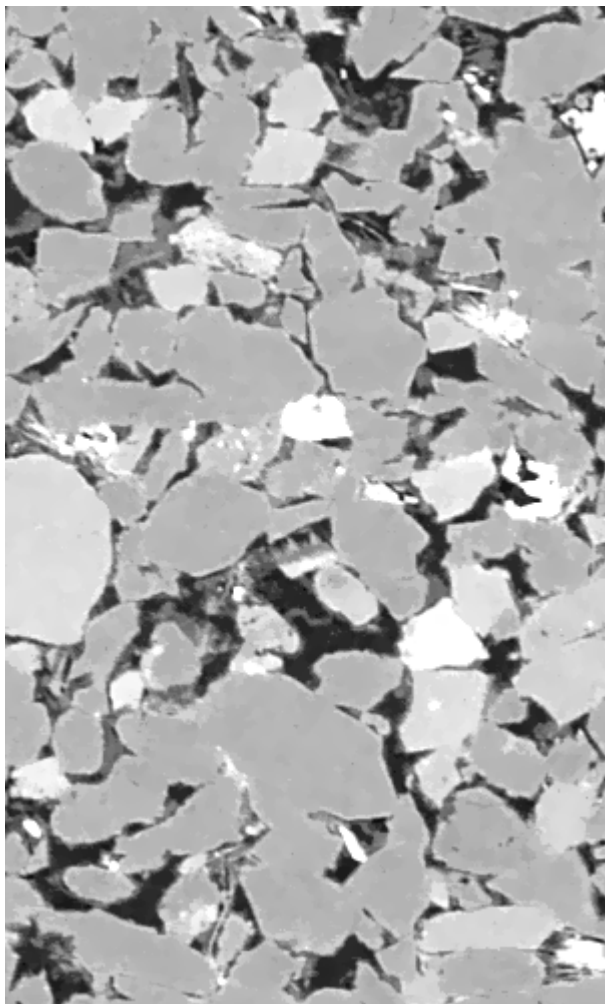


Iteration 2

noise = 28.8

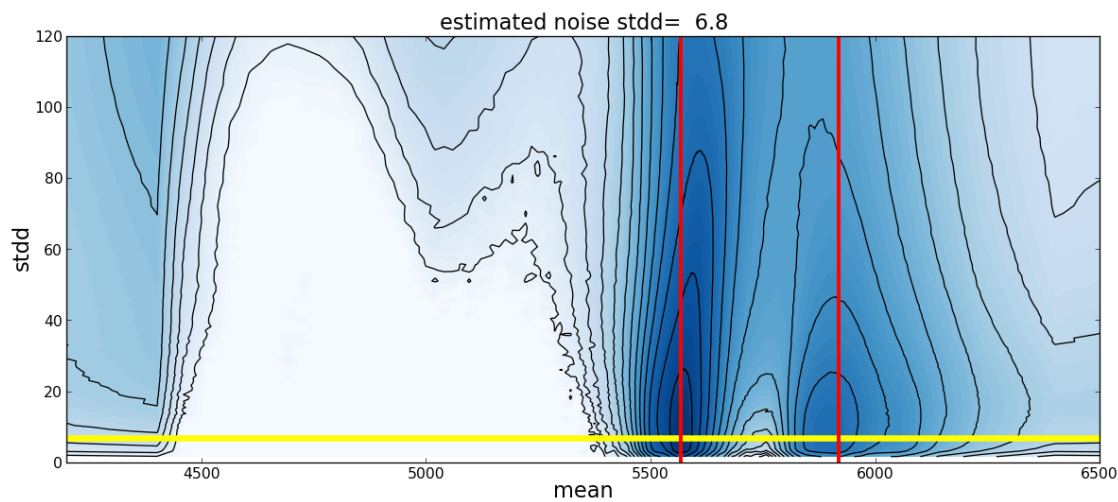


# Bilateral Filtering using estimated noise

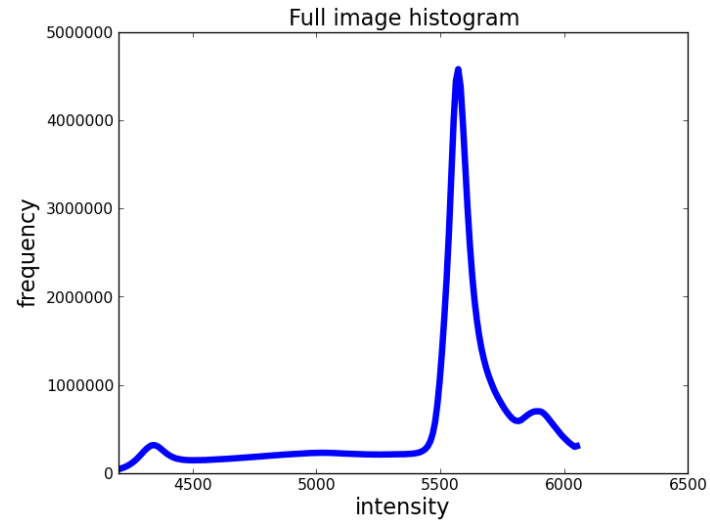
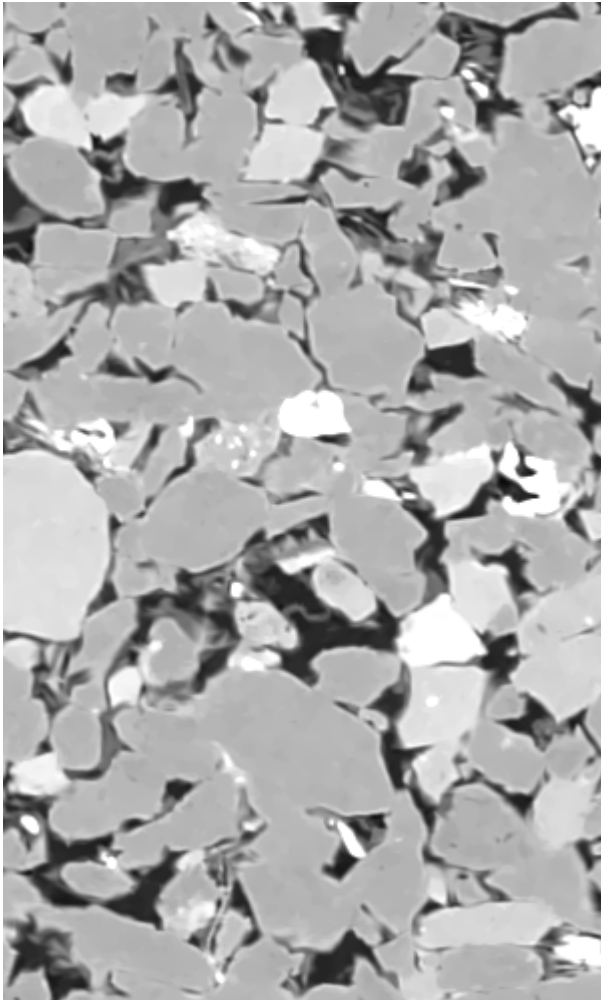


Iteration 8

noise = 8.8

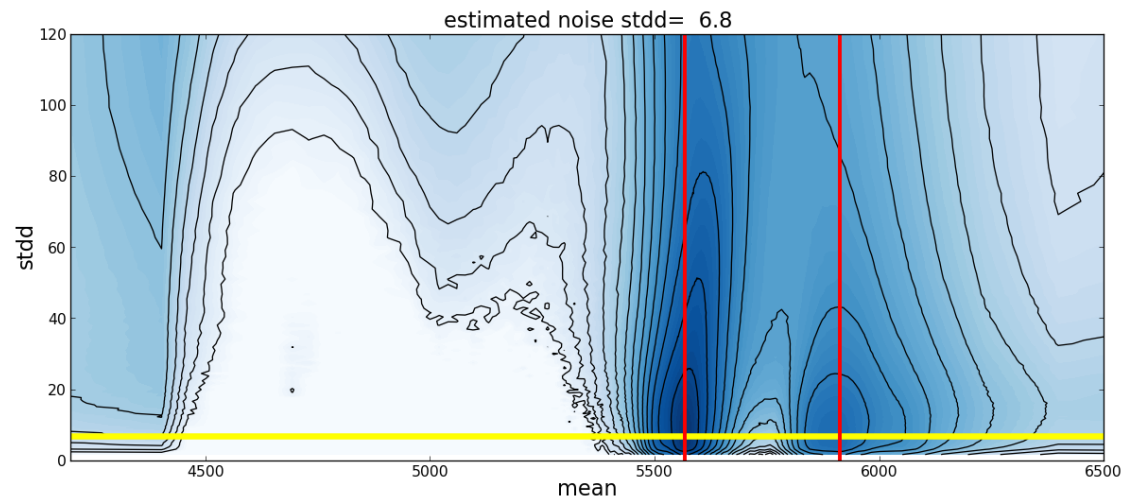


# Bilateral Filtering + Anisotropic Diffusion

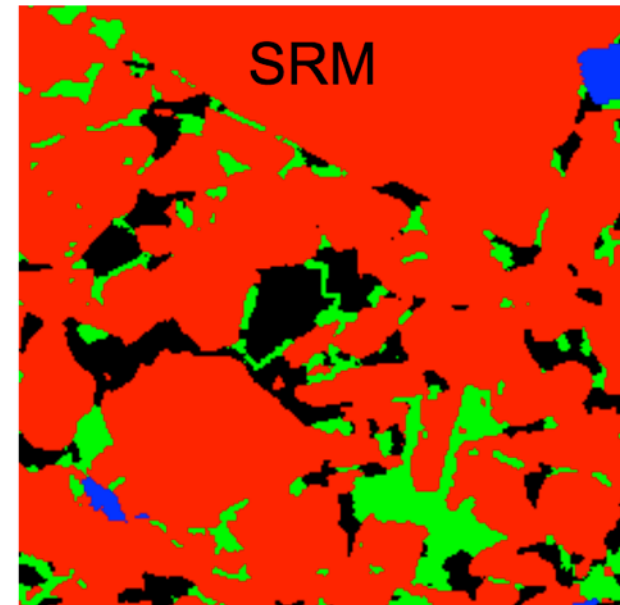
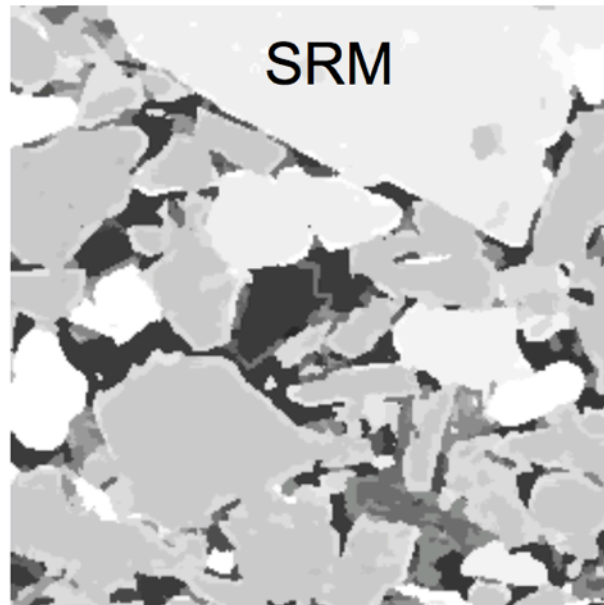
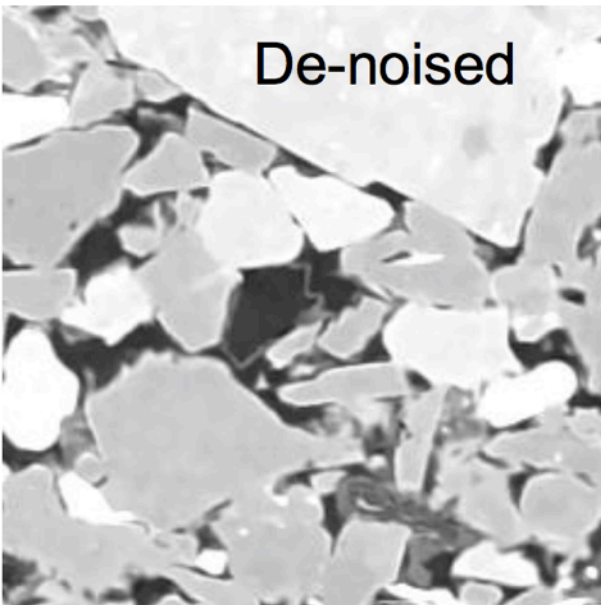
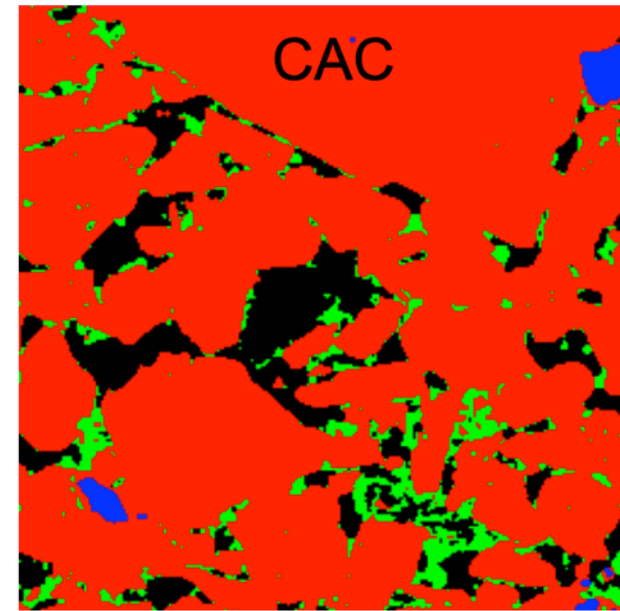
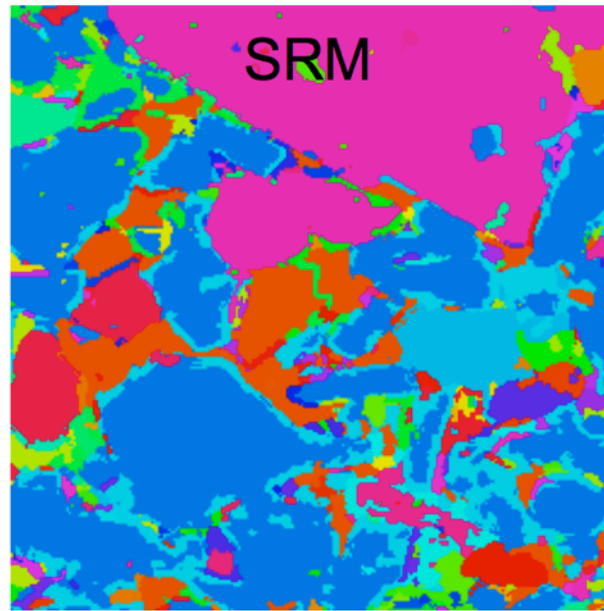
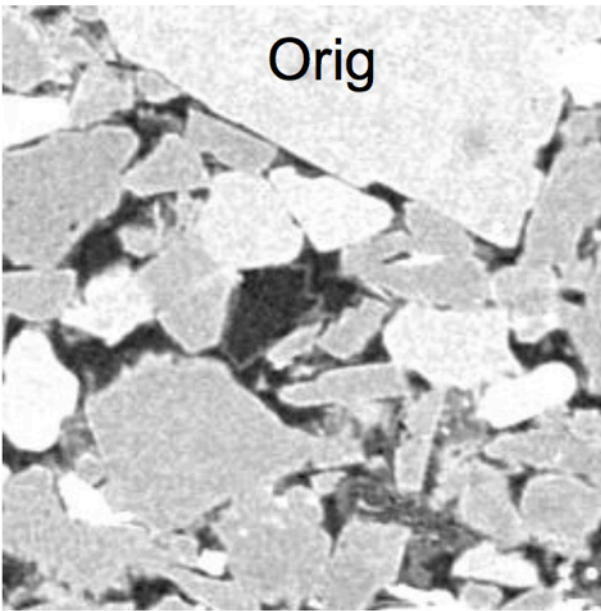


Iteration 8  
+  
Anisotropic  
Diffusion

noise = 6.8



# Statistical region merging versus manual (CAC) segmentation

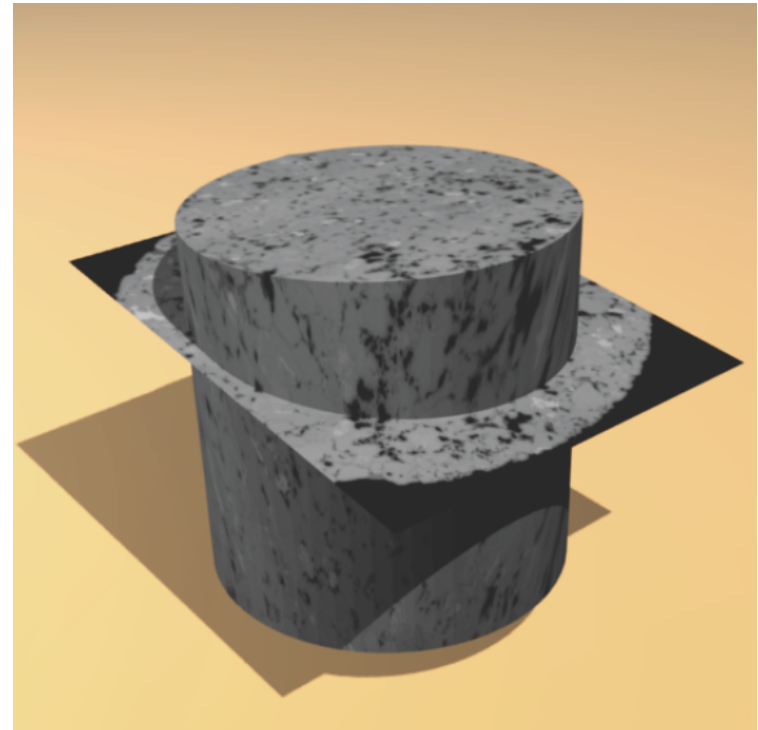




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# Image registration

- scalable, metric-based implementation
- 3D-3D:
  - Align 3D image to another, similar, 3D image
- 2D-3D:
  - after tomography, physically cut sample and make 2D image of surface within the same volume
- Image registration plays a role in most imaging studies at ANU.



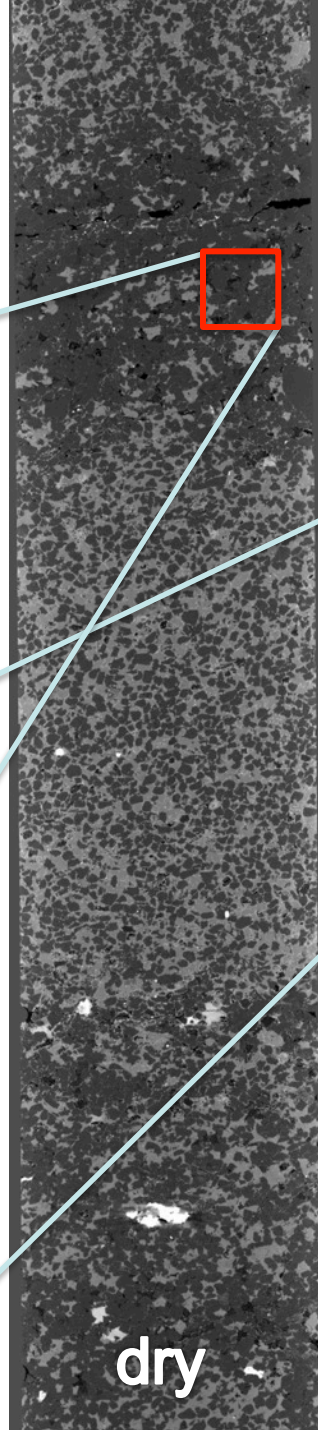
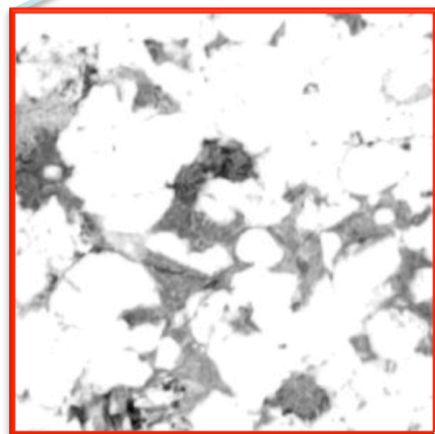
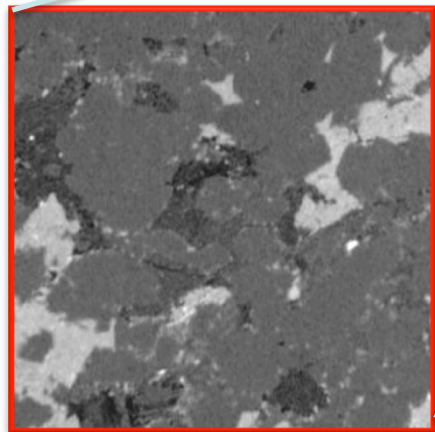


# Registration: Porosity mapping

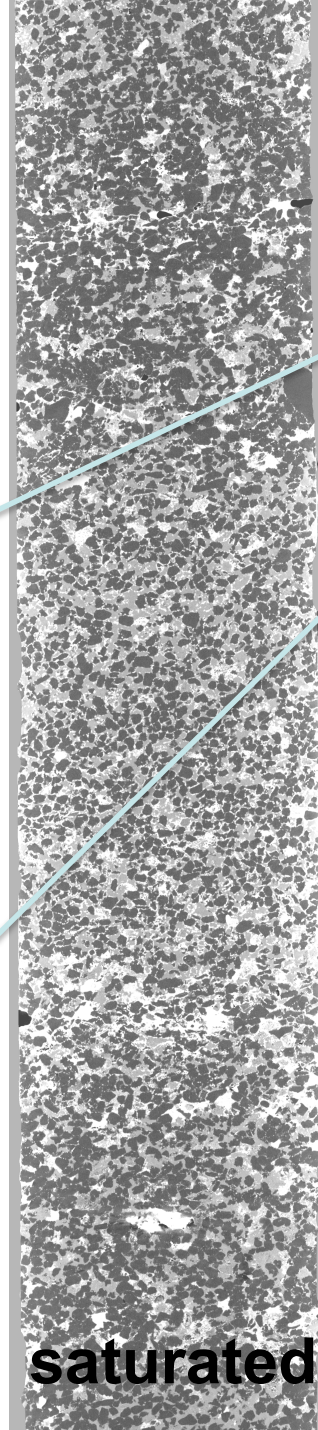
Heterogeneous  
tight sand  
36x8mm

Imaged at  
7200x1600<sup>2</sup>  
5 $\mu$ m voxels

Difference  
between  
saturated and  
dry image yields  
porosity map



**dry**



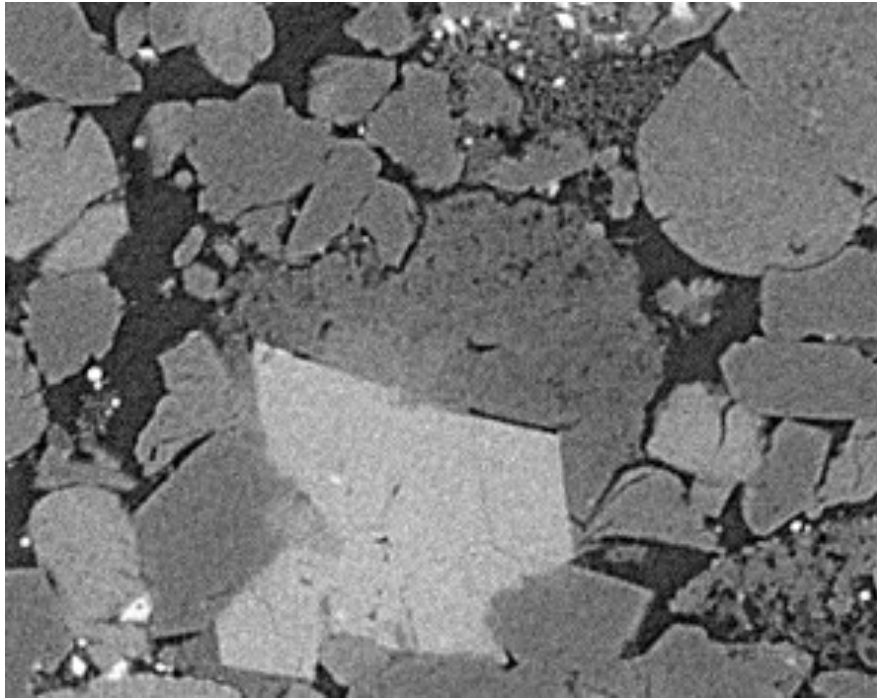
**saturated**



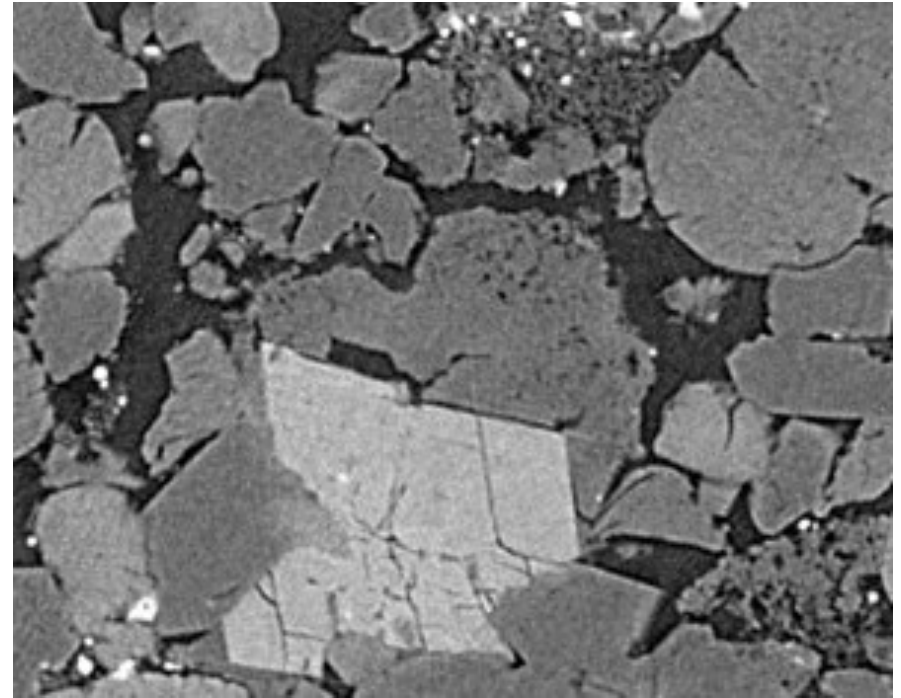
**difference**



## Imaging of CO<sub>2</sub> dissolution



before



after

*Barrow Island rock sample before and after treatment with carbonic acid for 329 hours under 1 MPa pCO<sub>2</sub> at 15-20°C. Field of view 0.8 x 0.7mm*



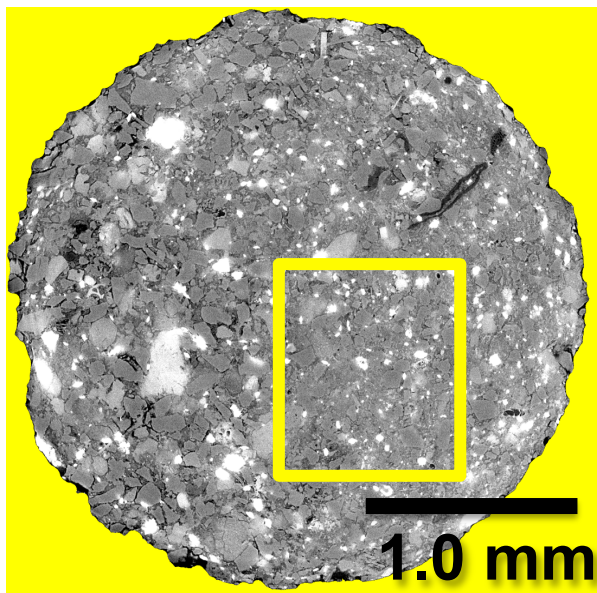
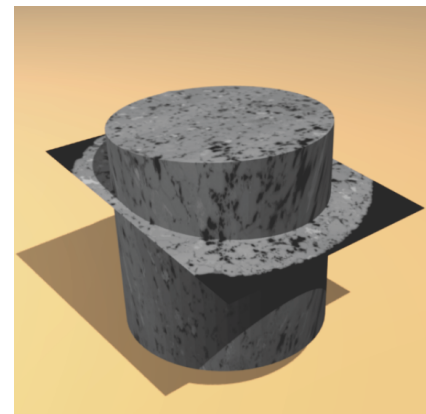


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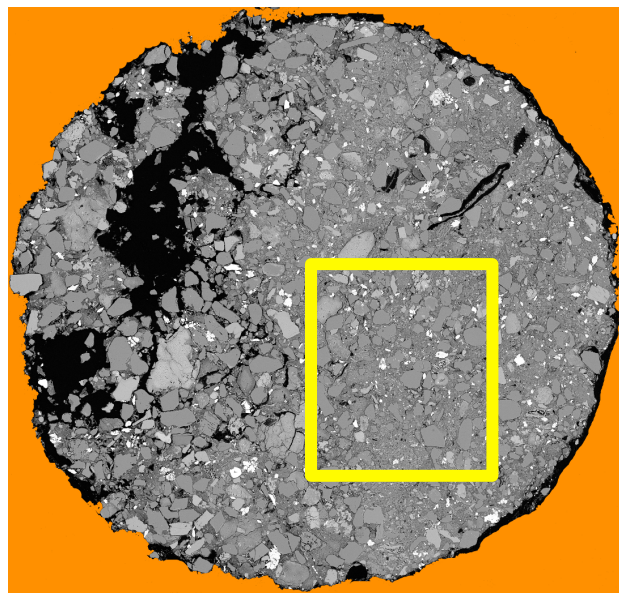


Australian  
National  
University

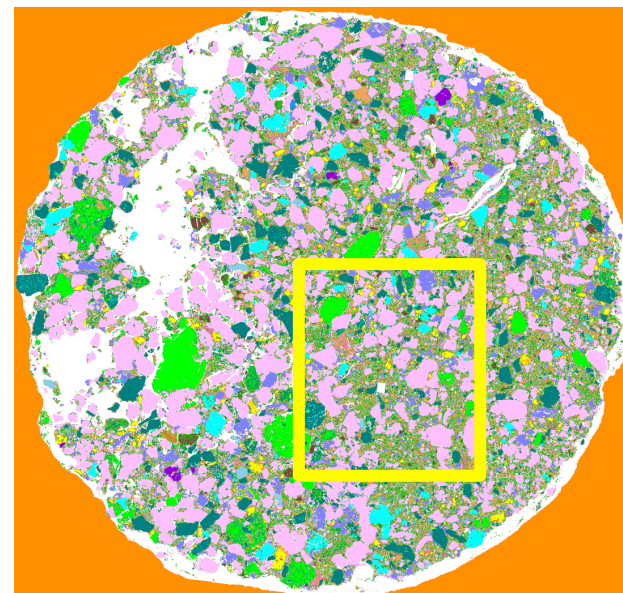
# 2D-3D Registration: X-ray MCT to SEM-EDX



X-ray microCT



SEM

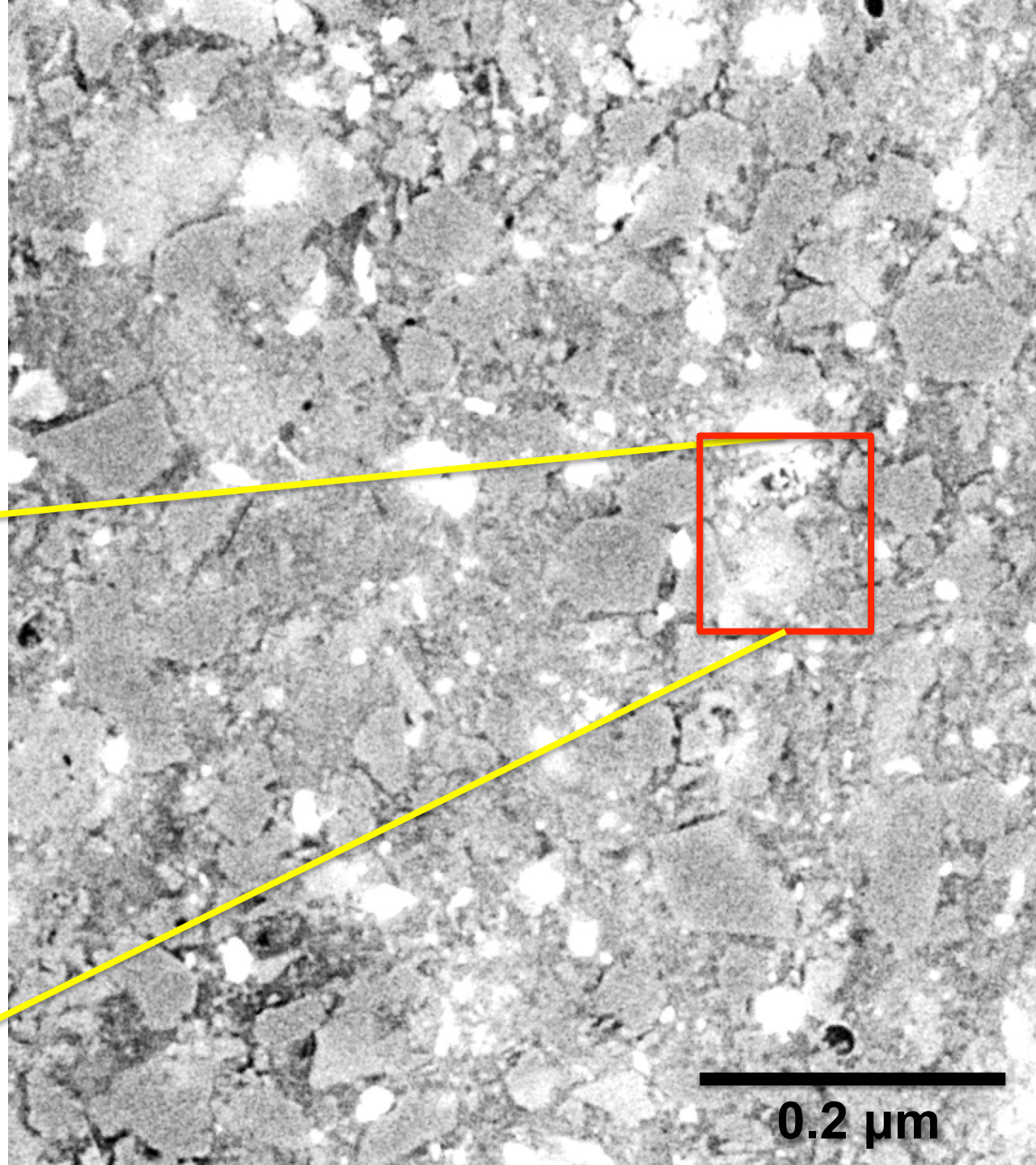


SEM-EDX  
(QEM)



# X-ray microCT

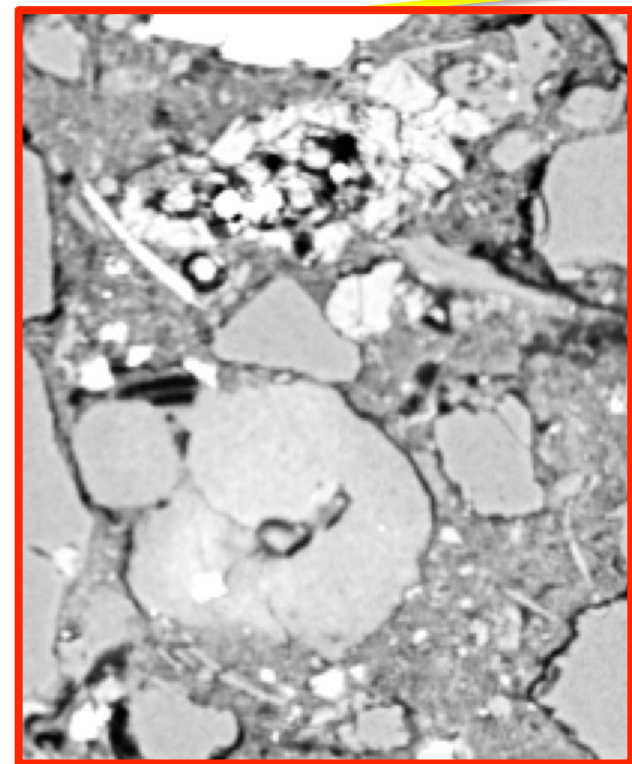
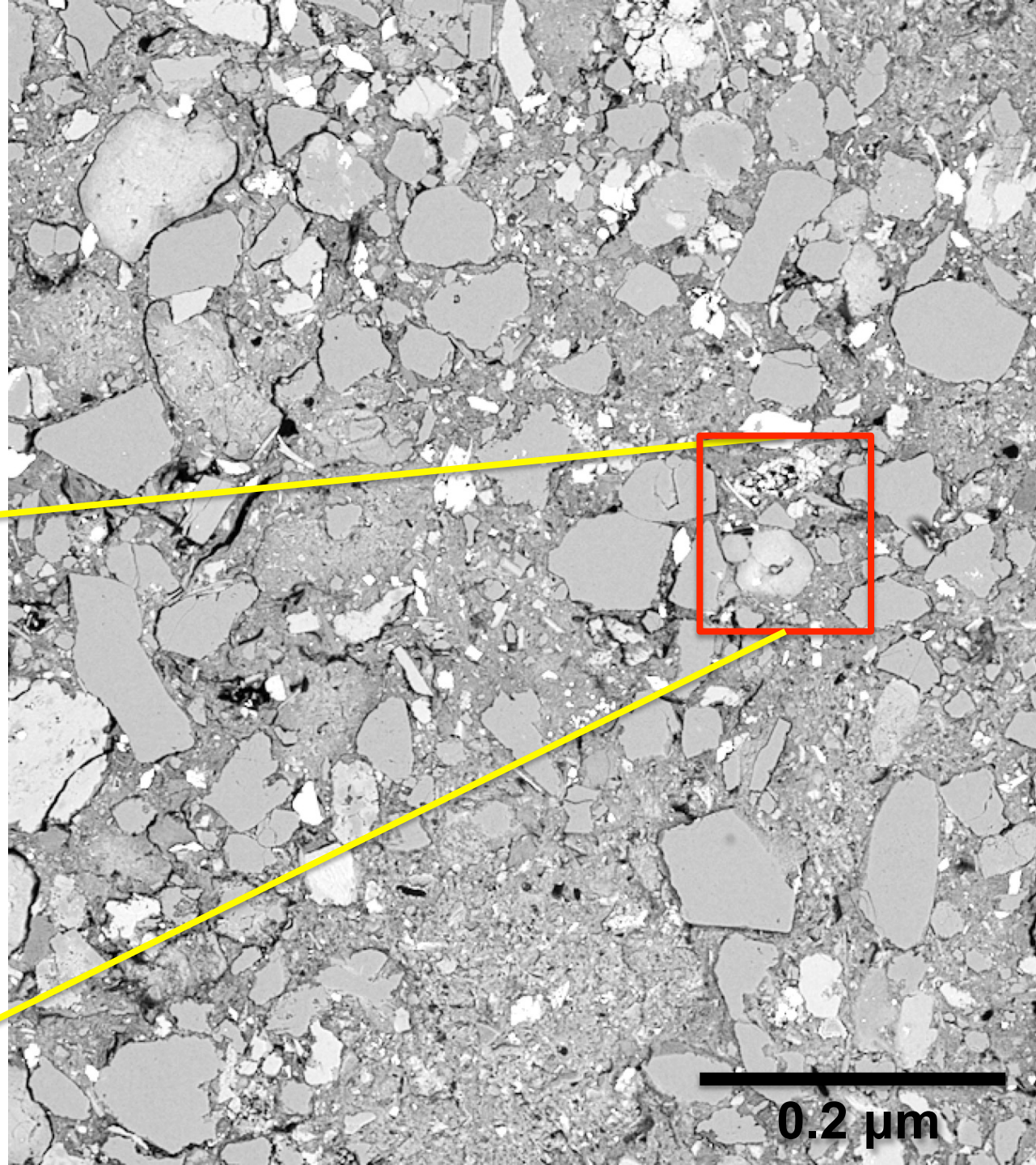
2.0  $\mu\text{m}$  voxels



0.2  $\mu\text{m}$



SEM  
(backscatter)  
0.5  $\mu\text{m}$  pixels

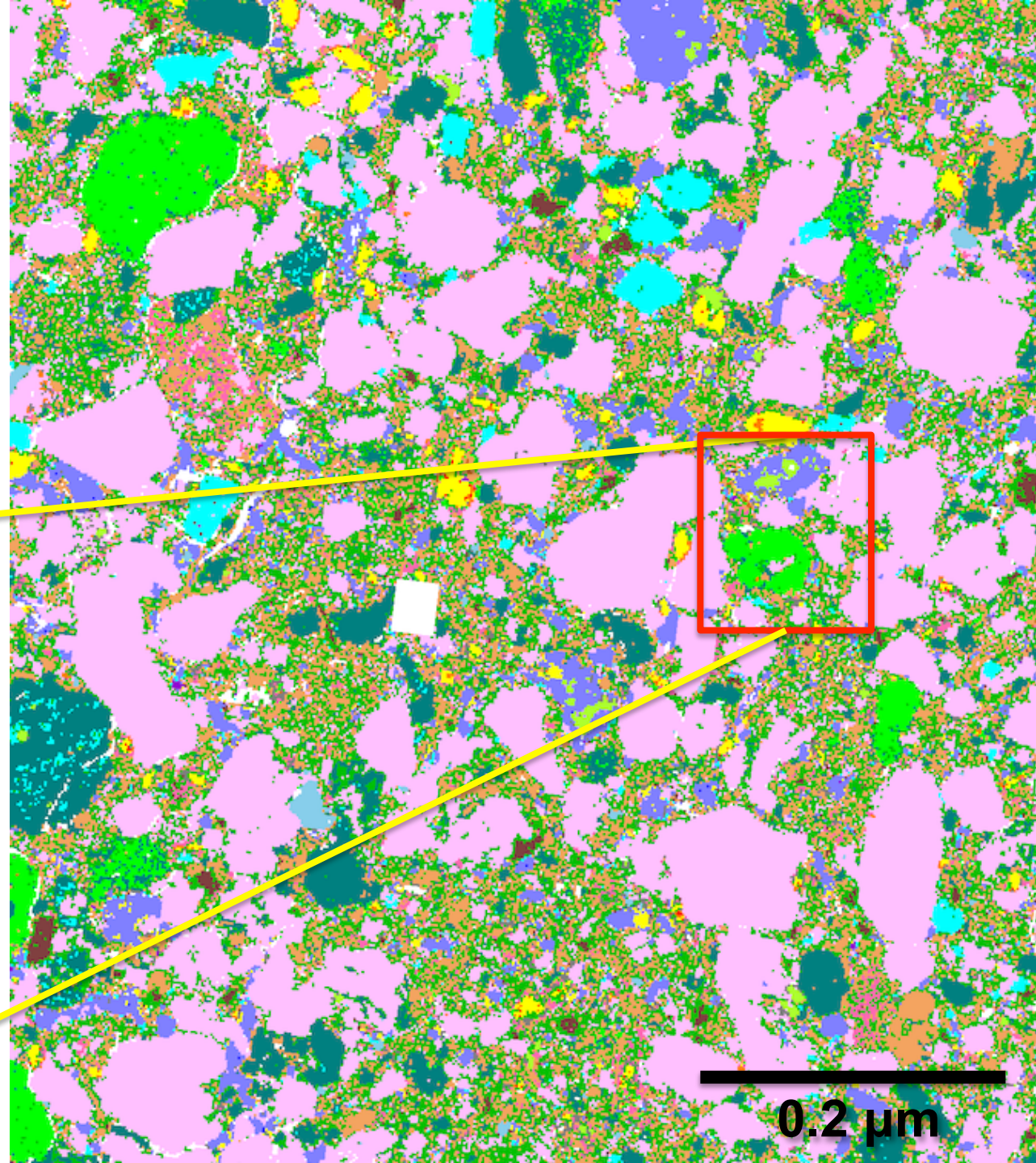
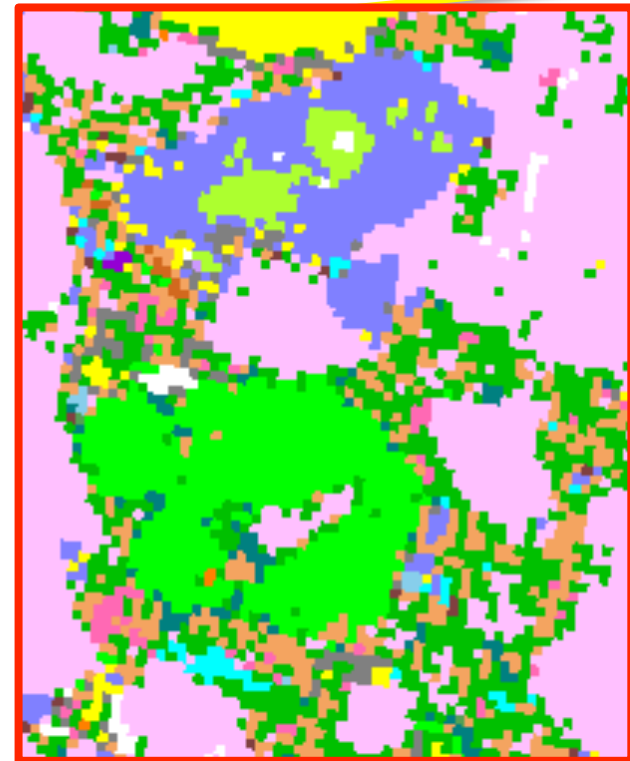


0.2  $\mu\text{m}$



# SEM-EDX (QEM)

2.0  $\mu\text{m}$  pixels



0.2  $\mu\text{m}$



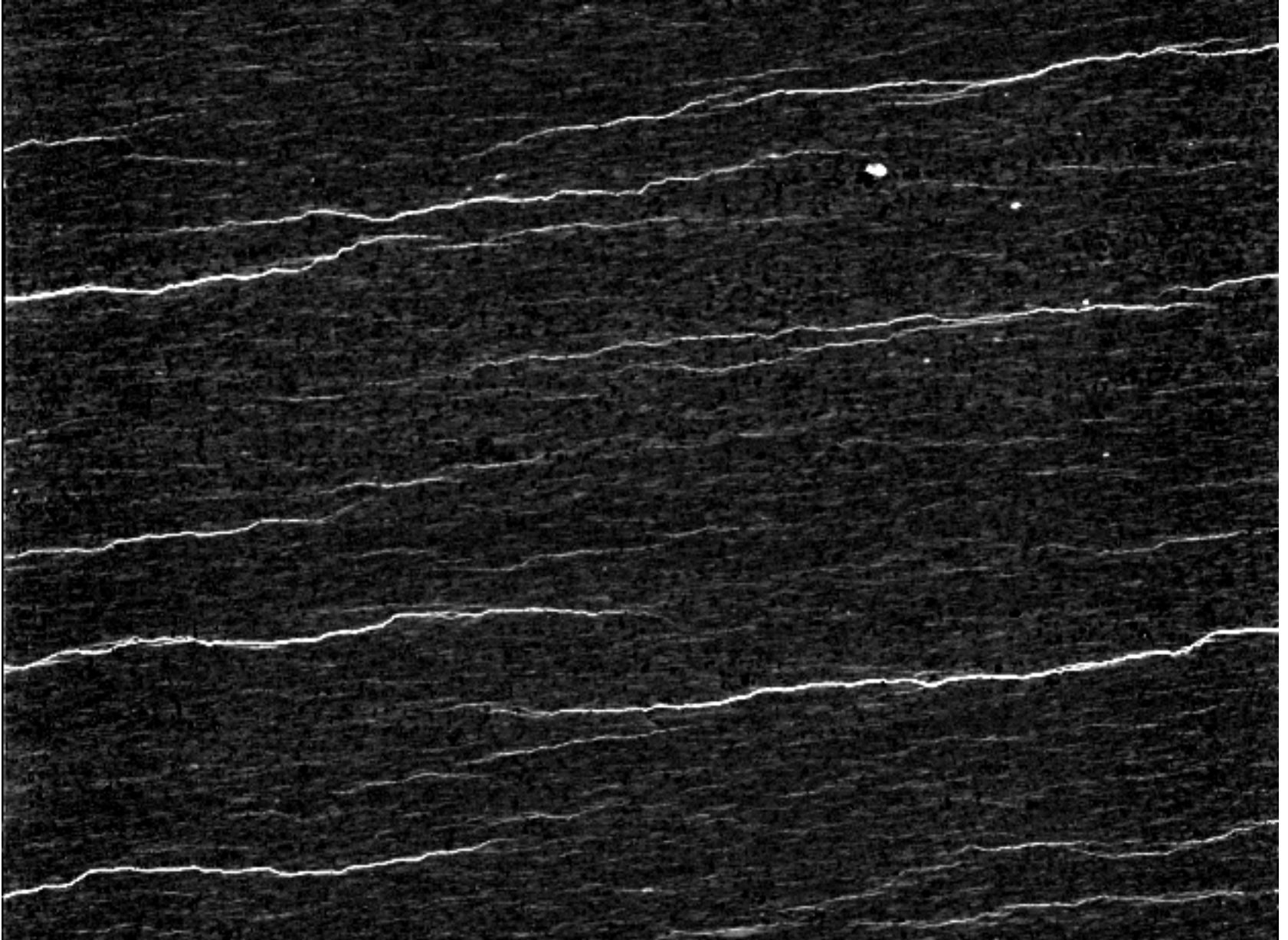
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# Studies of diffusion in shale

- $\text{CH}_2\text{I}_2$  saturated sample immersed in toluene inside micro-CT
- sequence of 80 tomograms acquired while  $\text{CH}_2\text{I}_2$  diffuses out and toluene diffuses in
- goal is to compute spatial diffusivity map, which should be closely correlated to permeability
- need concentration and mass of  $\text{CH}_2\text{I}_2$  in each voxel
- 3 samples studied at three different resolutions (3 mm, 8 mm and 12 mm)

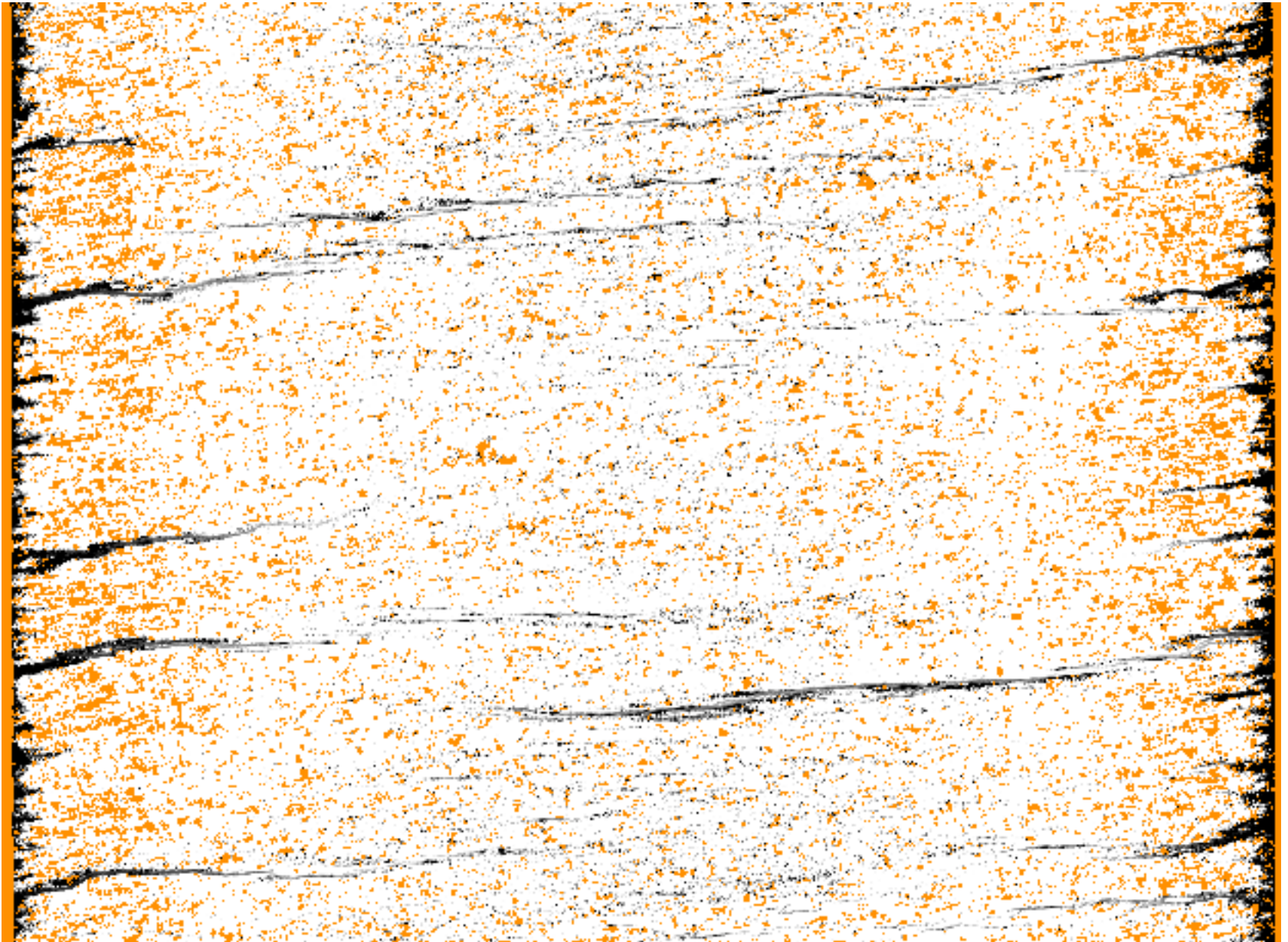


# 12mm Shale (porosity map)



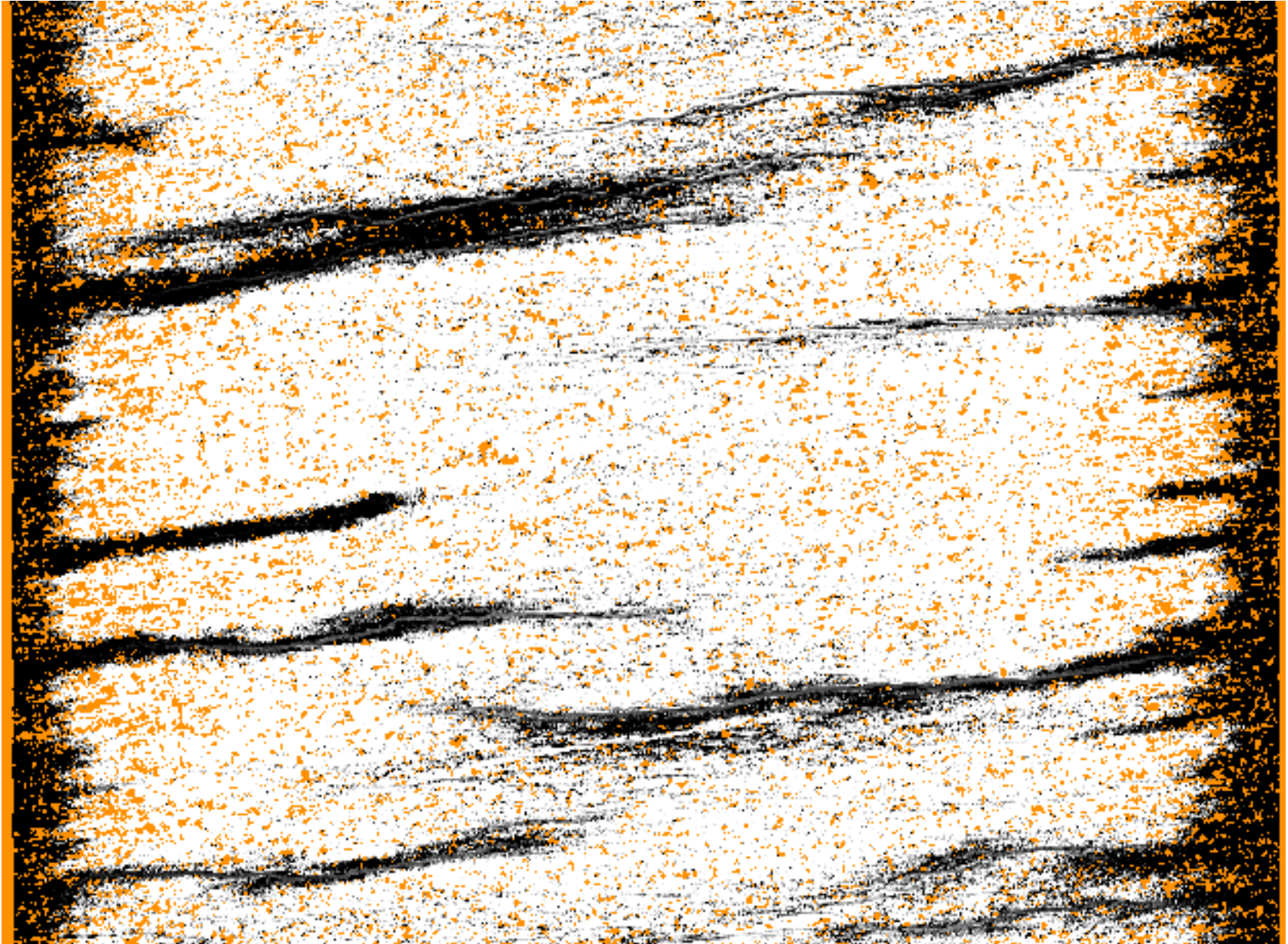


# 1<sup>st</sup> frame, map of conc( $\text{CH}_2\text{I}_2$ )



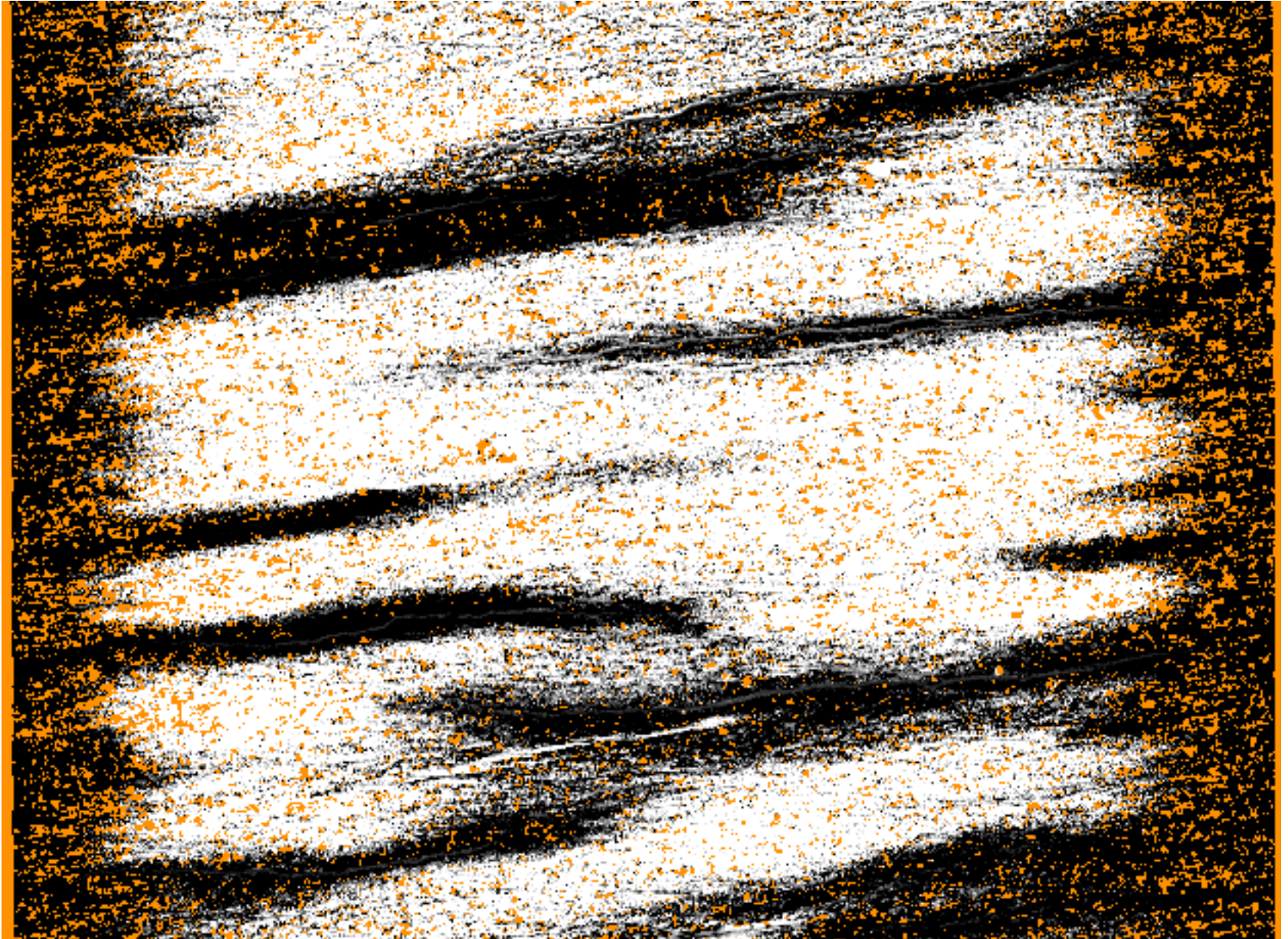


# 15<sup>th</sup> frame after tol. immersion



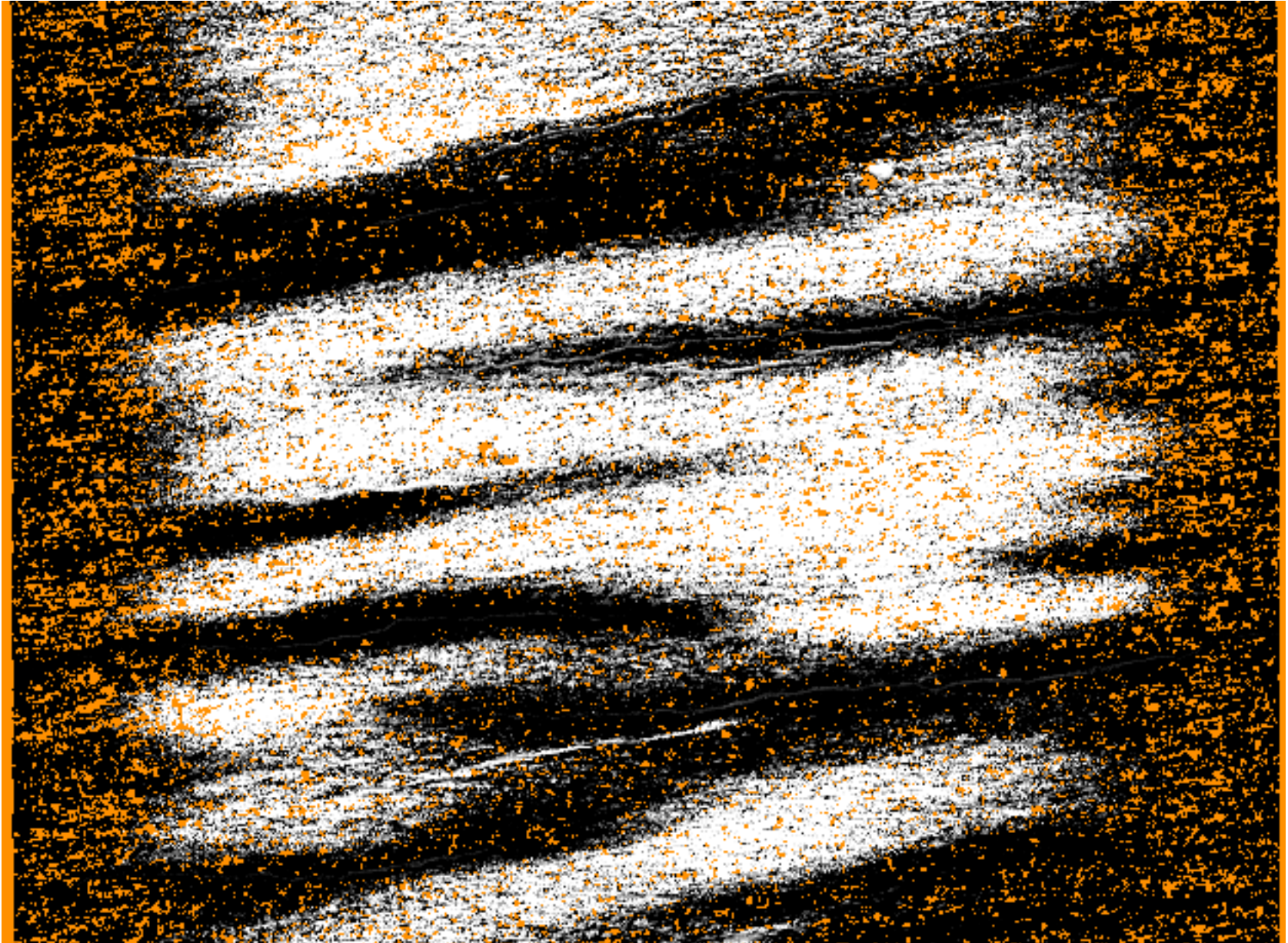


# 48<sup>th</sup> frame after tol. immersion



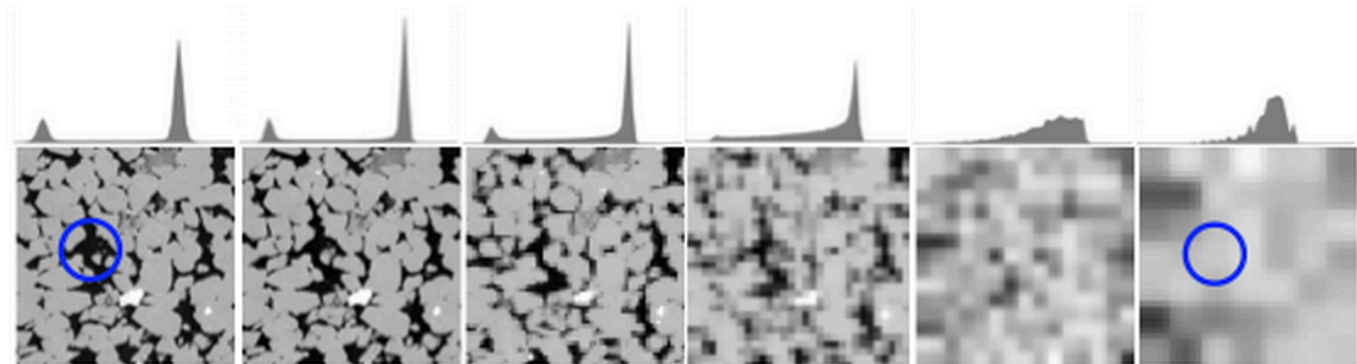


# 80<sup>th</sup> frame after tol. immersion

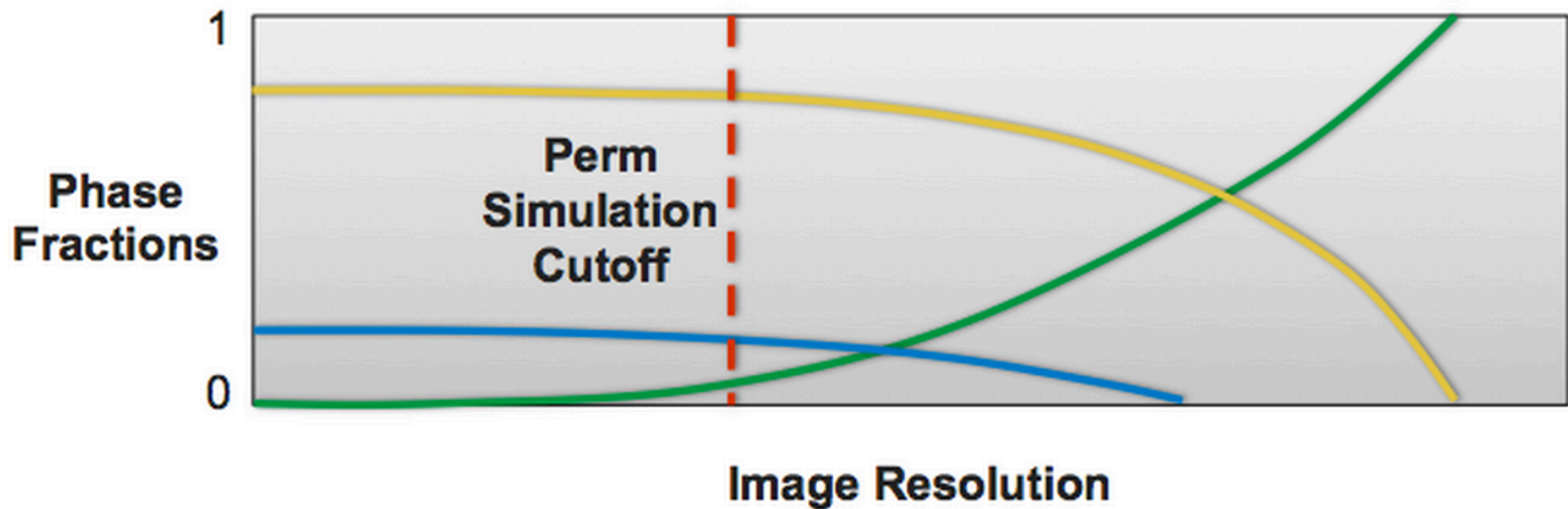


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# Effect of diminishing resolution

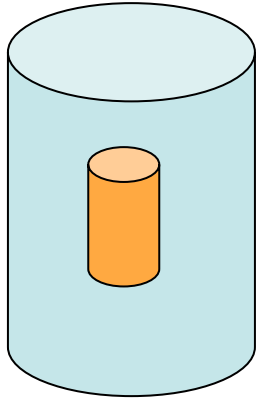


— Open Pores      — Intermediate      — Grains

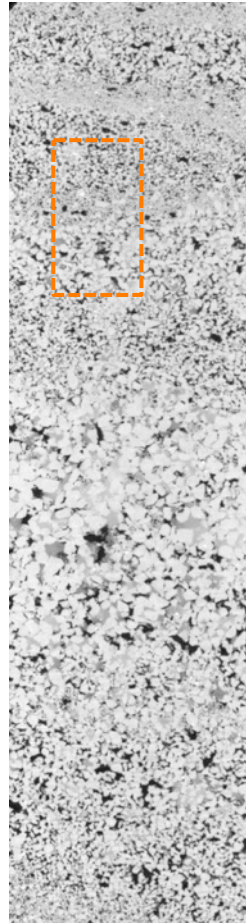




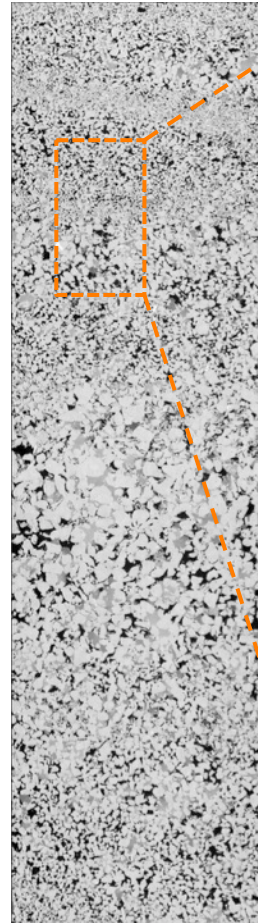
# Cross-scale imaging and registration



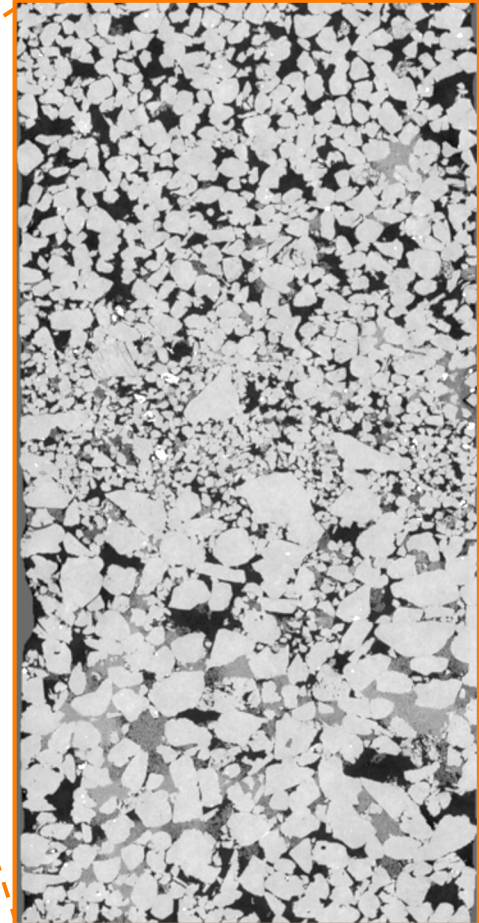
25mm Plug  
(64 $\mu\text{m}/\text{voxel}$ )



25mm Plug  
(16 $\mu\text{m}/\text{voxel}$ )



8mm Plug  
(~5 $\mu\text{m}/\text{voxel}$ )

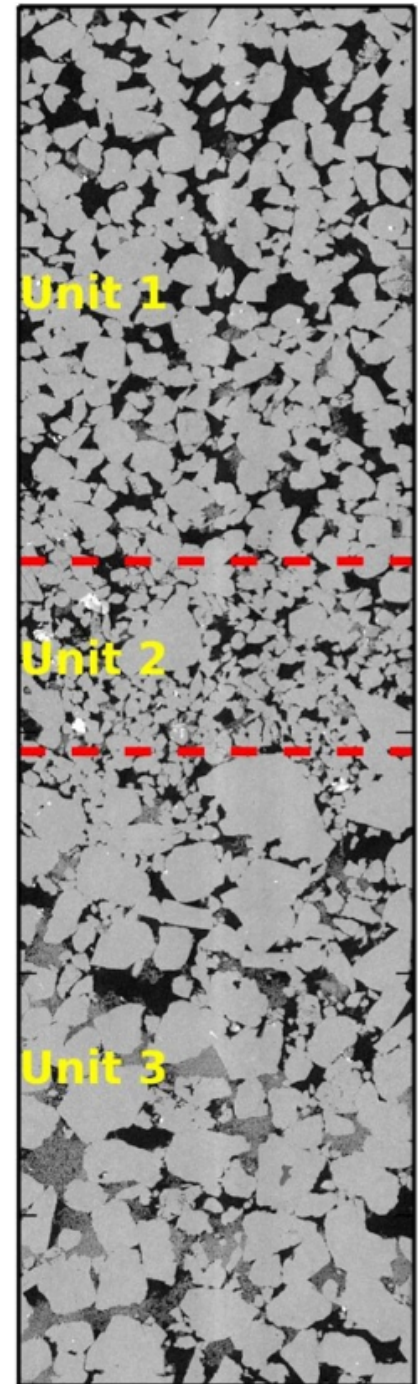
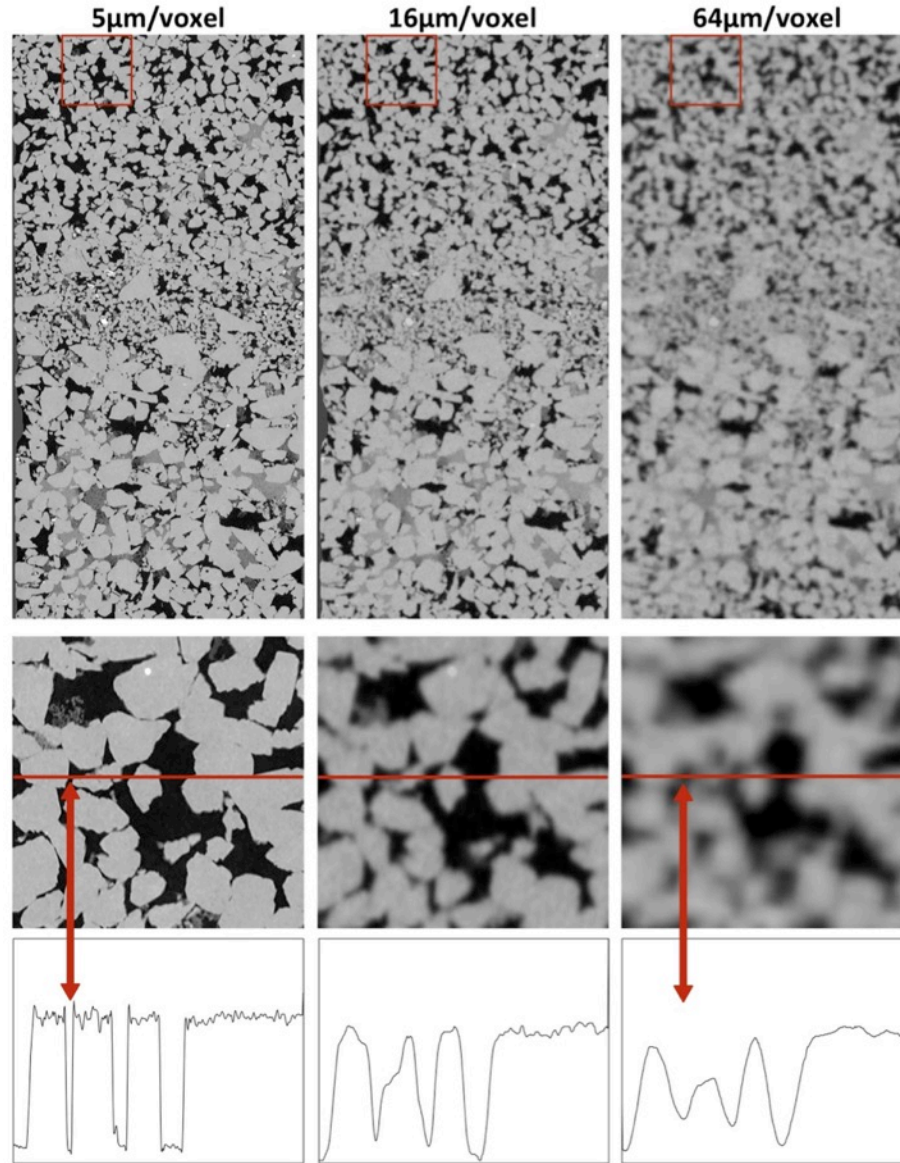


# Precipice sandstone

Imaged at 5, 16  
and 64  $\mu\text{m}$

25 mm plug, 8mm  
subplug

'Unitised'  
according to  
porosity, grain  
and pore size



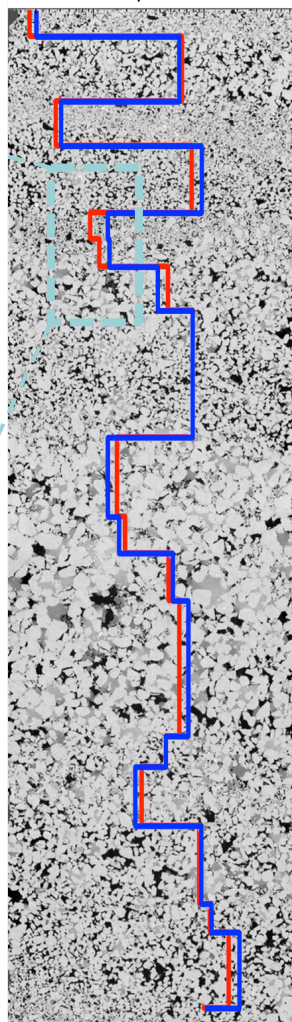
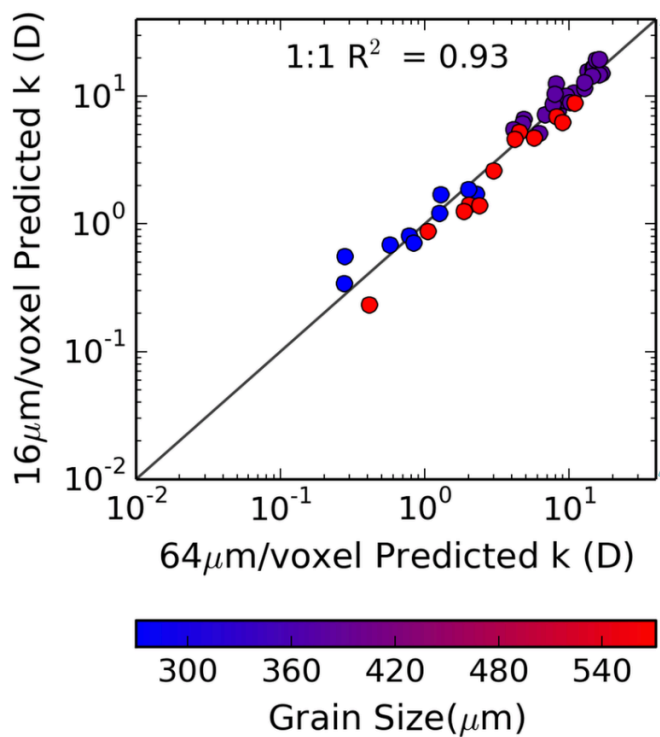




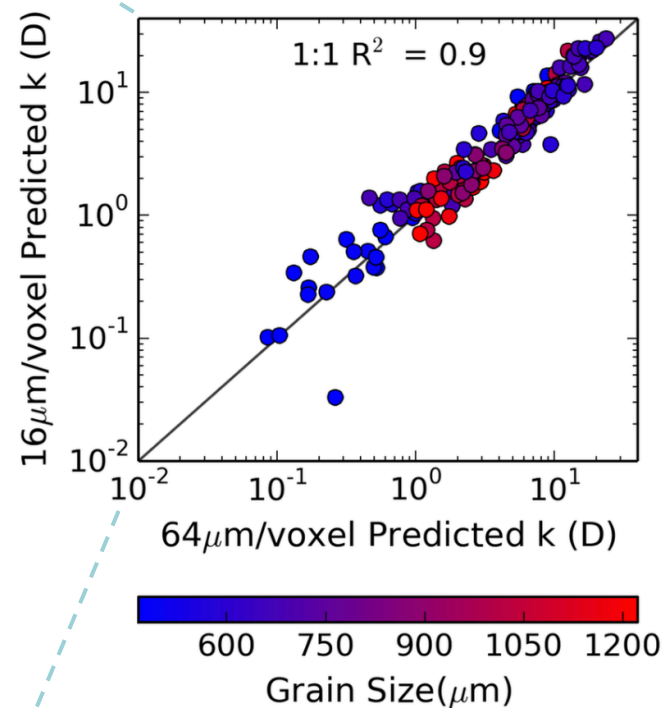
# Prediction Results

— 16 $\mu$ m Perm.  
— 64 $\mu$ m Perm.

8mm Core Plug Predicted k



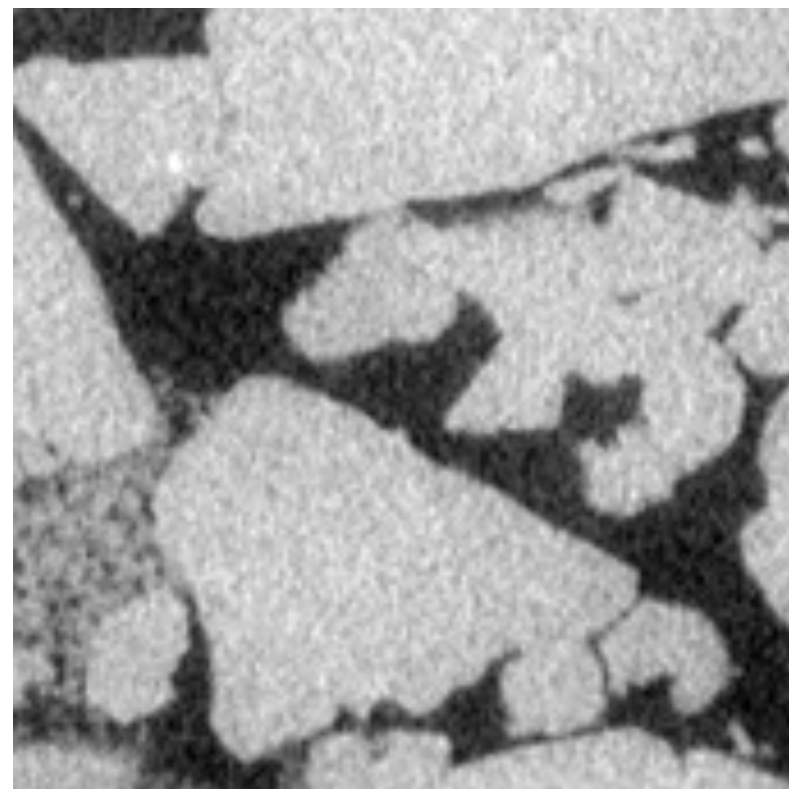
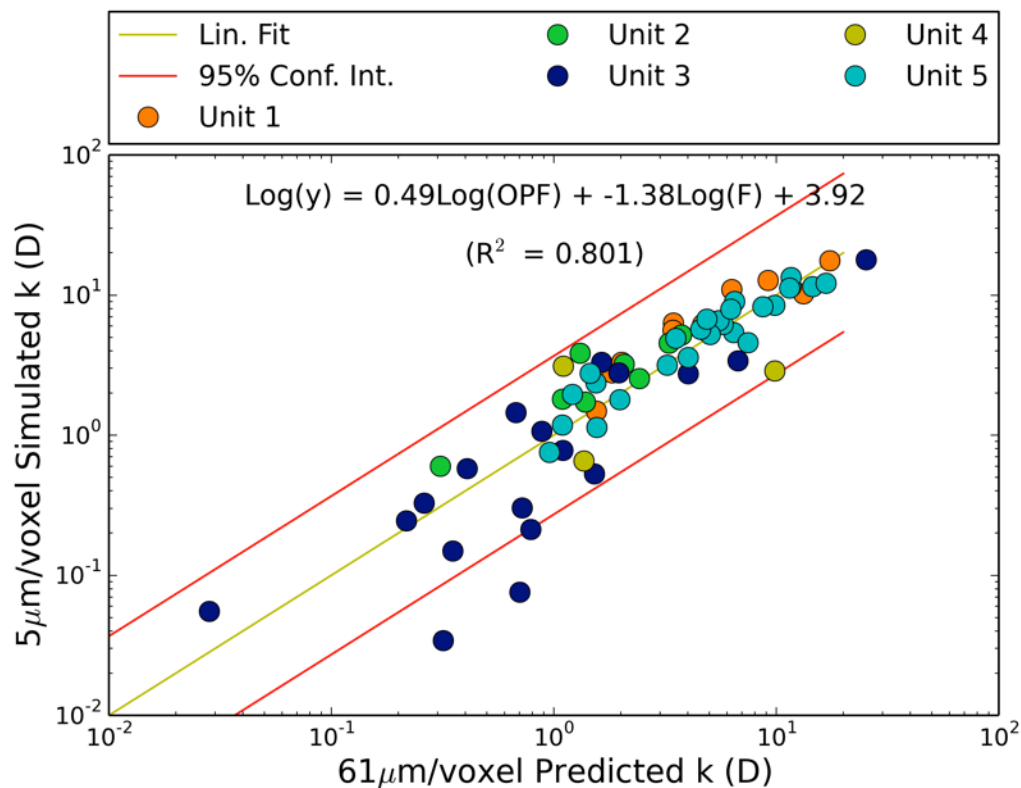
25mm Core Plug Predicted k





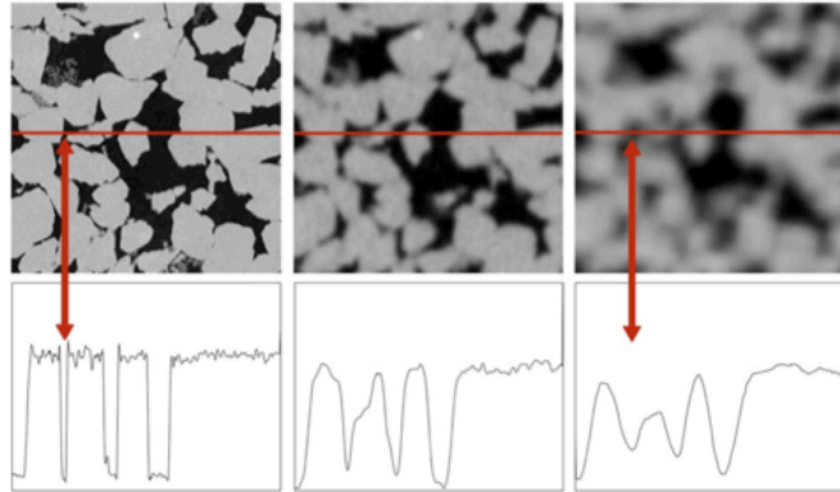
# Data Considerations

Observed vs. Predicted k



## Conclusions from cross-resolution study

- There is a range of resolution in which the sharp edges are lost, but in which the grayscale data still contains useful geometric information
- For this sandstone one can make worthwhile estimates of permeability from images at  $1/10^{\text{th}}$  of the resolution needed for Navier-Stokes solvers.
- Future work:
  - capillary pressure and other two-phase properties
  - carbonates



# Conclusions - the future?

- Quantitative X-ray tomography is possible and could hold the key for industrial applications
  - dual energy imaging and iterative reconstruction are key elements
- Unsupervised segmentation is another critical element – but what are its limits?
  - need methods that quantify their accuracy
- Multi-scale and multi-modal imaging crucial for heterogeneous media
  - need robust, fast image registration
  - often have “training” volumes, so machine learning may play a role



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