Quantification of Organic Nano-Pores Using a Helium Ion Microscope*

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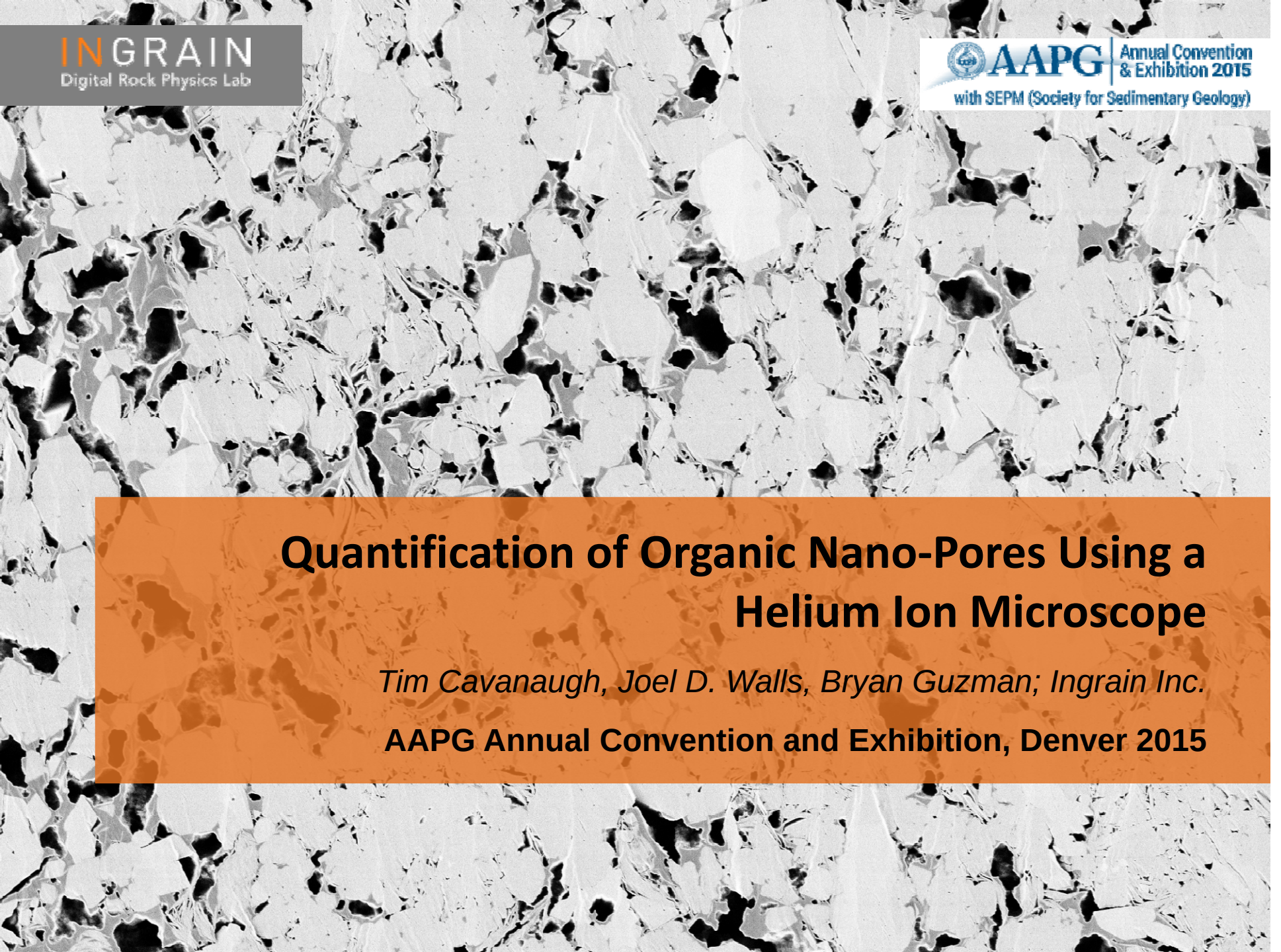
Abstract

Scanning electron microscopy of ion polished samples has become a common way to estimate porosity and organic matter content within shale resource rocks. Since quantitative SEM analysis has emerged as a means for assessing the porosity of shale rock, a common goal has been to image polished samples at extremely high resolutions. Since nano-pores are visible at pixel resolutions ranging from 5–10 nm, it is natural to consider the possibility of a pore regime below 5 nm which could contribute a significant amount to the total porosity of the system. When considering that a molecule of methane gas is on the order of 0.4 nm diameter, these 5 nm pores could be significant transport pathways in a reservoir. These nano-pores are a significant source of porosity within certain organic matter bodies, where total detectable pores using SEM (i.e., ~10 nm pore body diameter and up) can comprise up to 50 percent or more of the original volume of organic matter present. With the potential to examine the population of pores below ~10 nm in diameter using the helium ion microscope, it is possible to construct a rock model that is more representative of the varied pore size regimes present. In this study, 12 organic shale samples were selected for systematic imaging using the Carl Zeiss Orion helium ion microscope. These samples were chosen based on examination of previously completed imaging using Carl Zeiss Auriga FESEM, and were selected due to the presence of porous and non-porous organic matter. Prior to SEM imaging, the samples had been ion-polished using a Gatan argon ion polishing system. The previously completed set of SEM images were acquired with a pixel resolution of 10 nm. Samples were imaged in the helium ion system using varying parameters in order to optimize image quality. Field of view and resolution were selected and increased as appropriate, with each acquired image matching a subset of an extant SEM image to allow for a direct comparison of grayscale, resolution, and volume percentage of various materials. The smallest pixel size of these images was 0.5 nm. After careful and

consistent segmentation, it was concluded that most samples had no significant pore fraction below the detection threshold of conventional FESEM imaging. The advanced resolution capabilities of the helium ion beam provide much sharper definition of pore boundaries but the total volume of these < 10 nm diameter pores in most samples was negligible.

Reference Cited

Hill, R., J.A. Notte, and L. Scipioni, 2012, Scanning Helium Ion Microscopy, *in* P.H. Hawks (ed.), Advances in Imaging and Electron Physics, v. 170, Elsevier Inc., Amsterdam, p. 65 – 148.



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Quantification of Organic Nano-Pores Using a Helium Ion Microscope

Tim Cavanaugh, Joel D. Walls, Bryan Guzman; Ingrain Inc.

AAPG Annual Convention and Exhibition, Denver 2015

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Overview of Helium Ion Imaging

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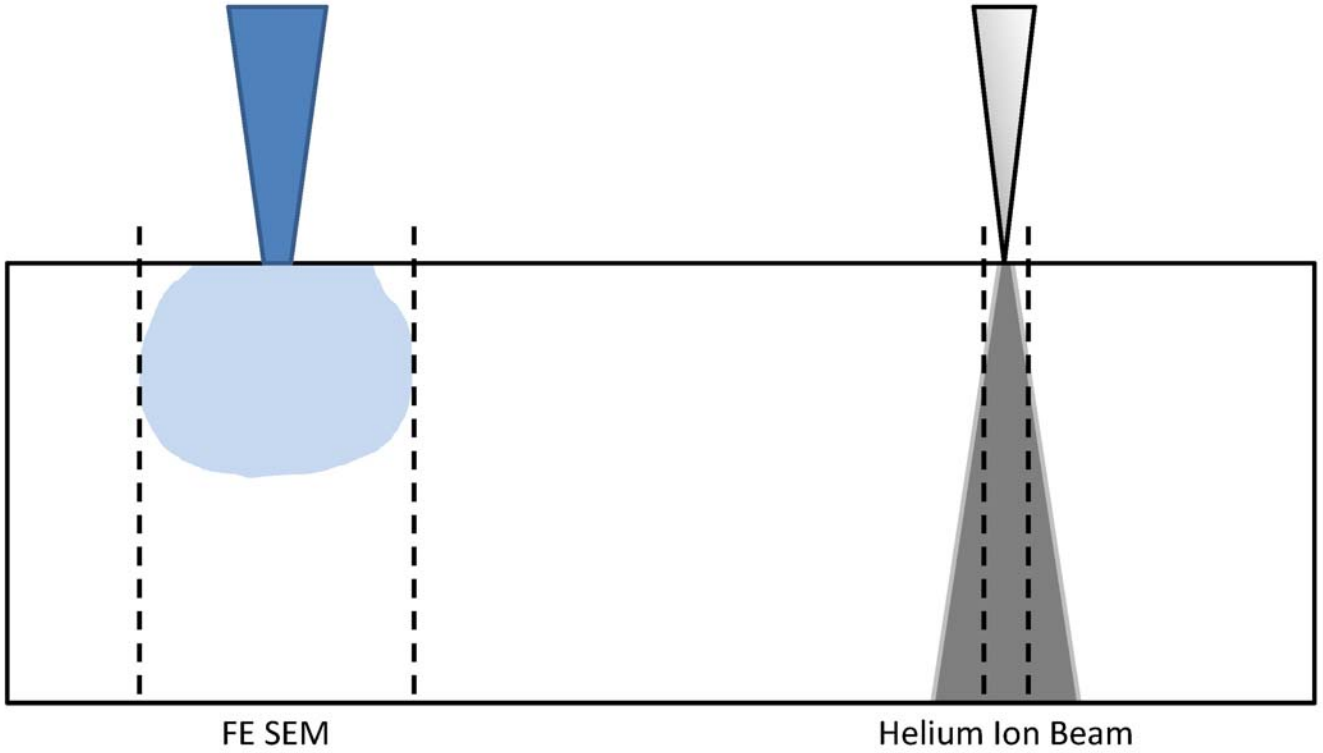
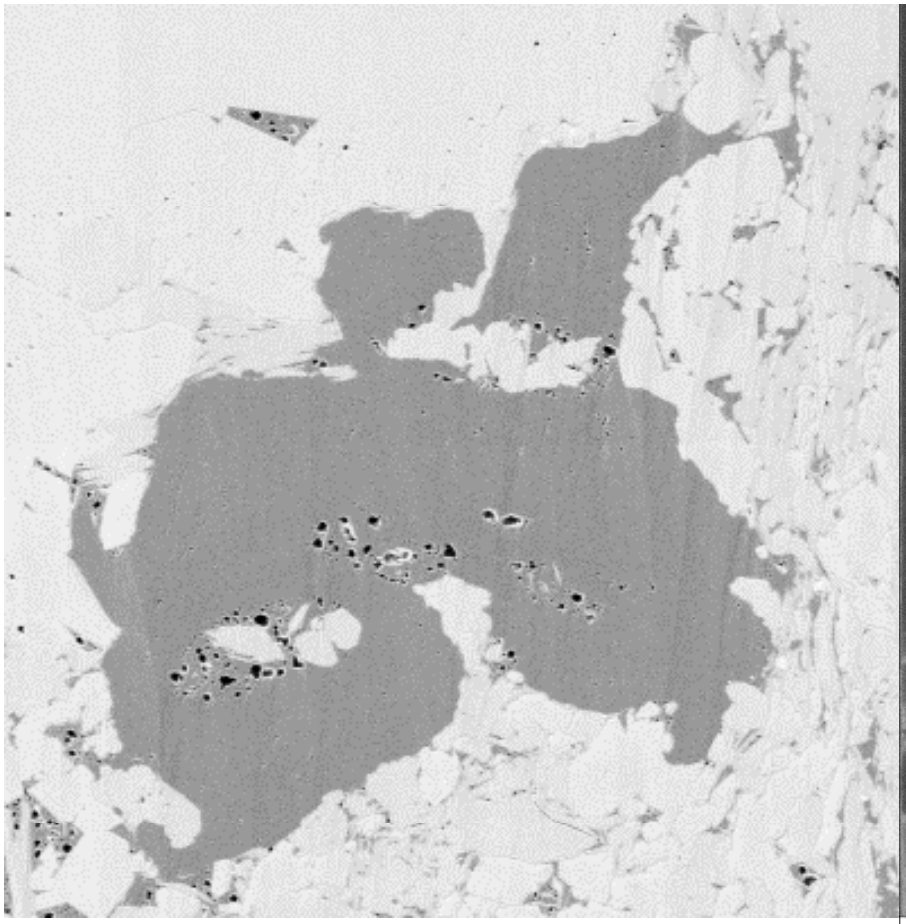
The benefit of the helium ion microscope (HIM), compared to conventional field-emission scanning electron microscopy (SEM), is the greatly reduced spot size of the ion beam providing incredible detail in X-Y resolution , and deeper information in Z direction

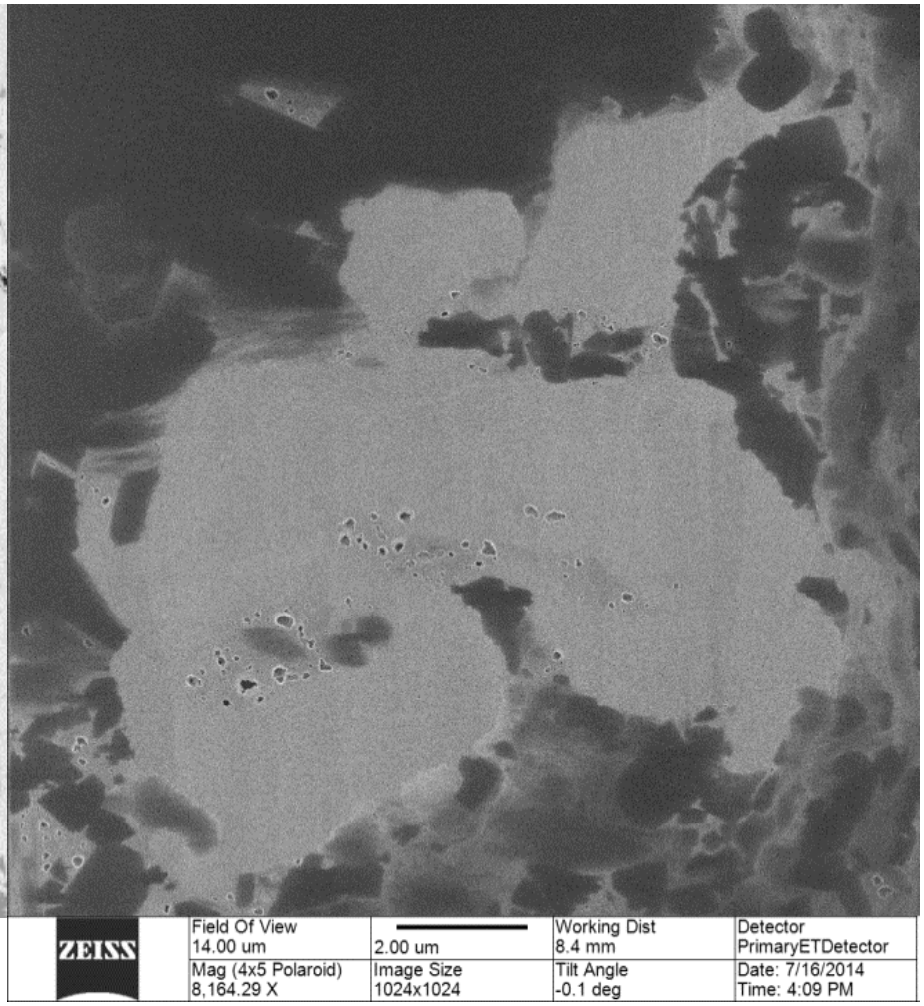


After Hill, et al, Scanning Helium Ion Microscopy, 2012

Presenter's notes: Helium ion beam secondary electron only (obviously primary beam is not electrons, can't have backscattered electrons).
Surface sensitive info- very narrow footprint- sub-nm x-y resolution.

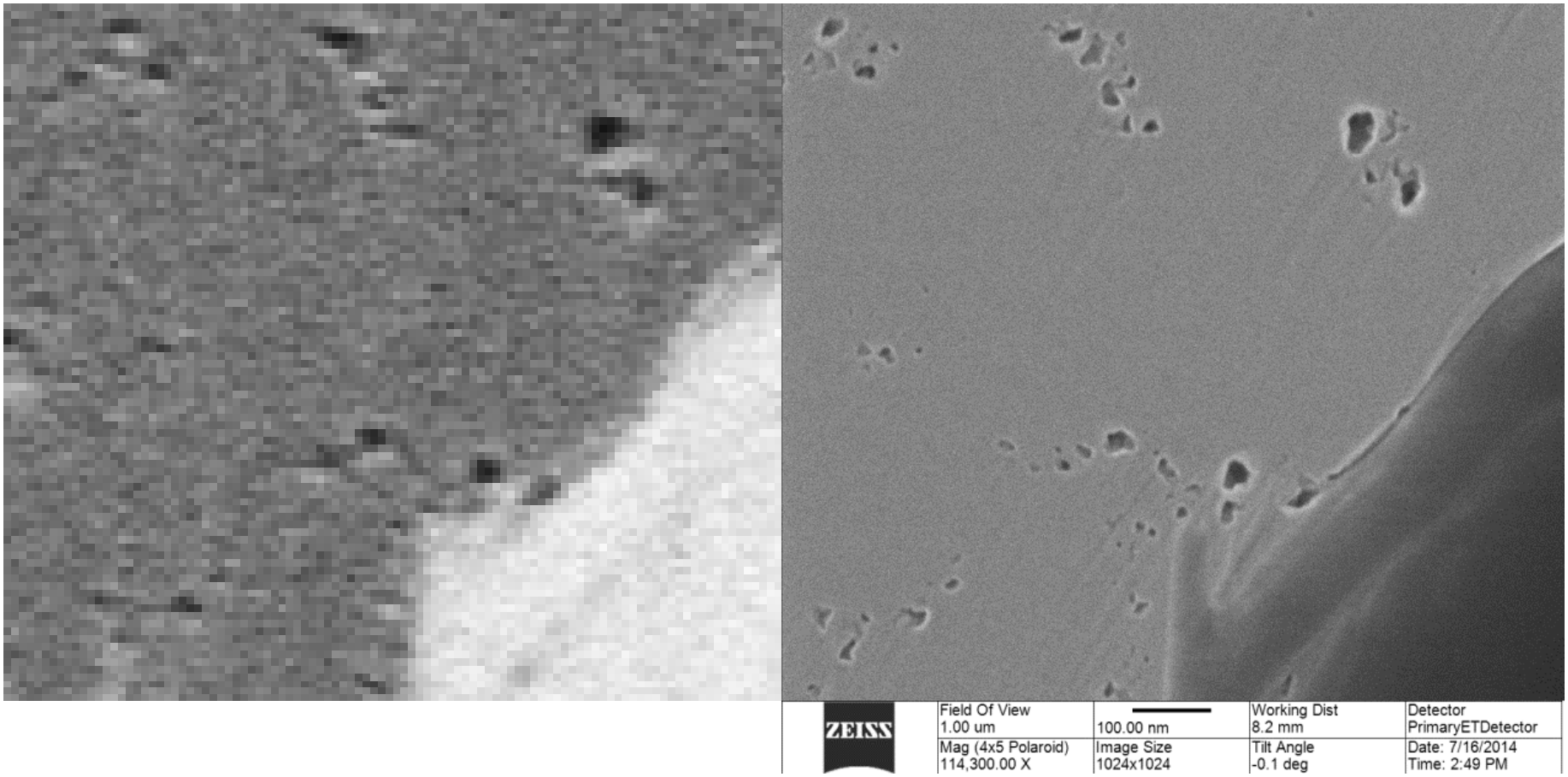
Porosity Examples: Eagle Ford





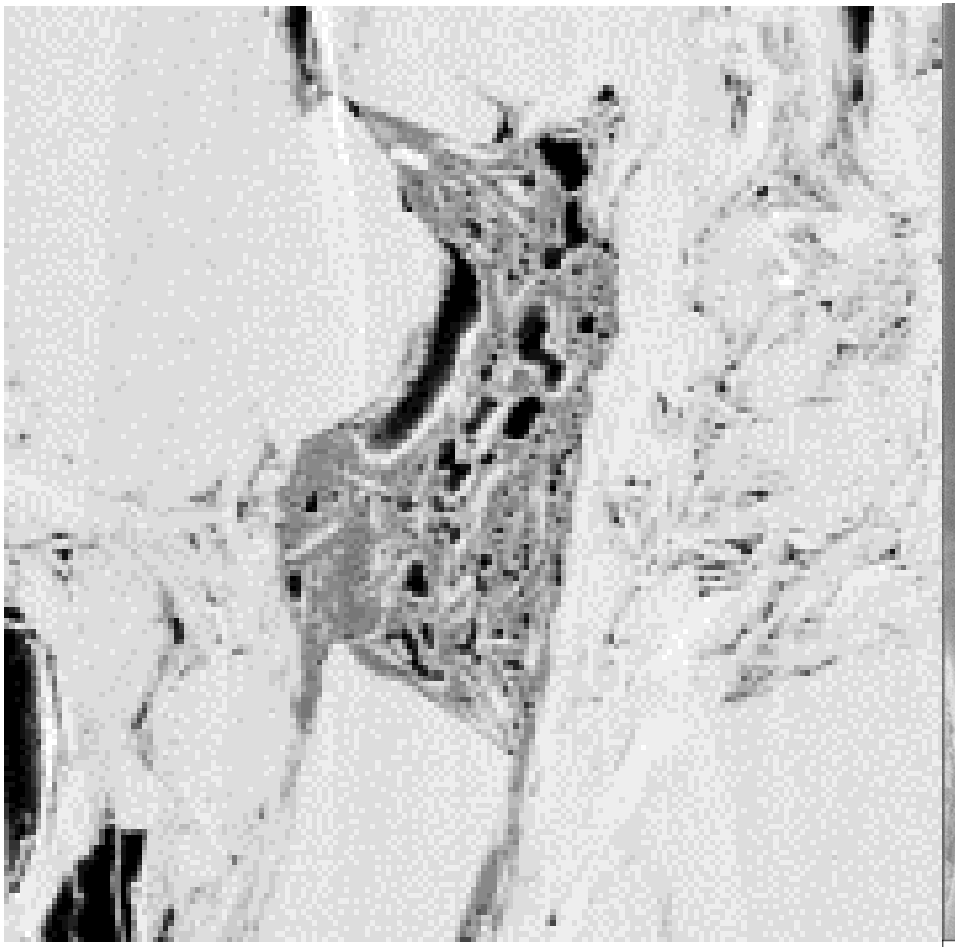
Note inverted contrast between solid grain and organic matter

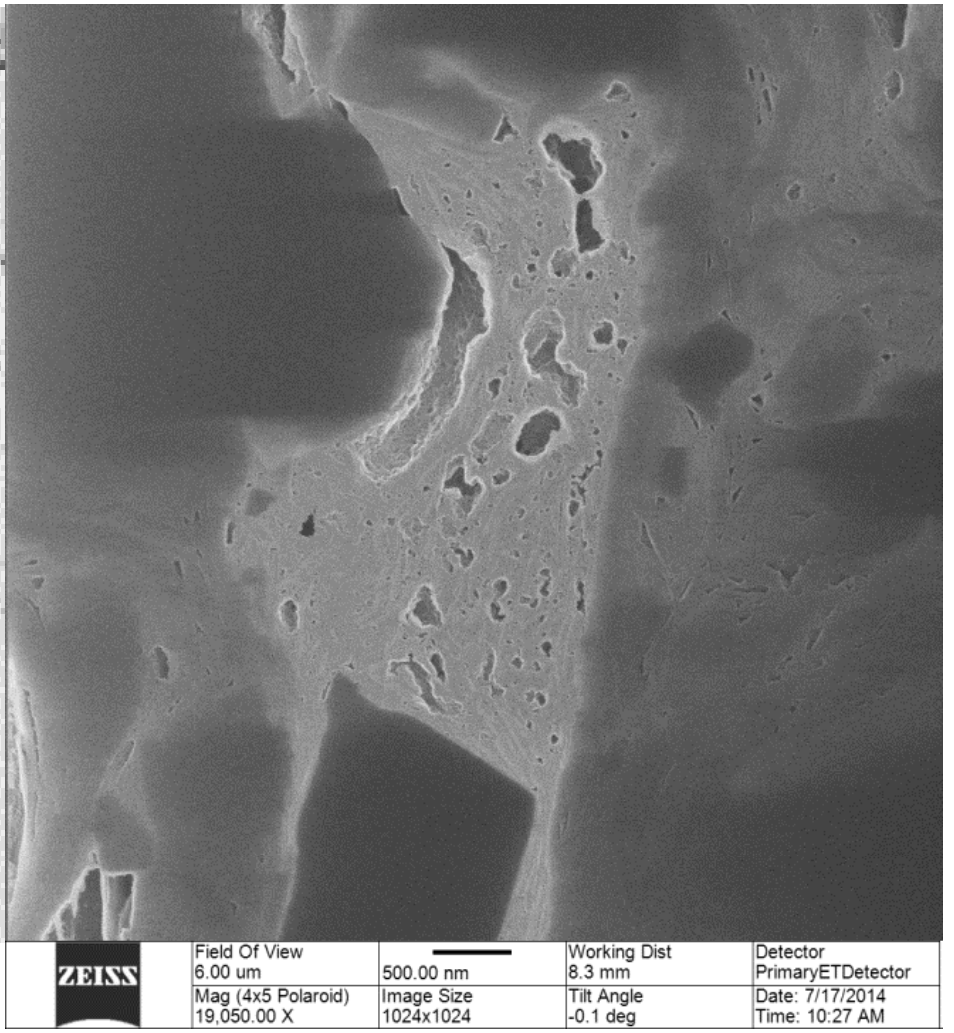
Porosity Examples: Marcellus



Much sharper definition of pore boundaries; linear pore at right is not detectable in SEM

Porosity Examples: Bossier





HIM image is effective at resolving small organic pores; less useful for imaging and calculating intergranular pores which are barely visible in image at right

Porosity Examples: Wolfcamp

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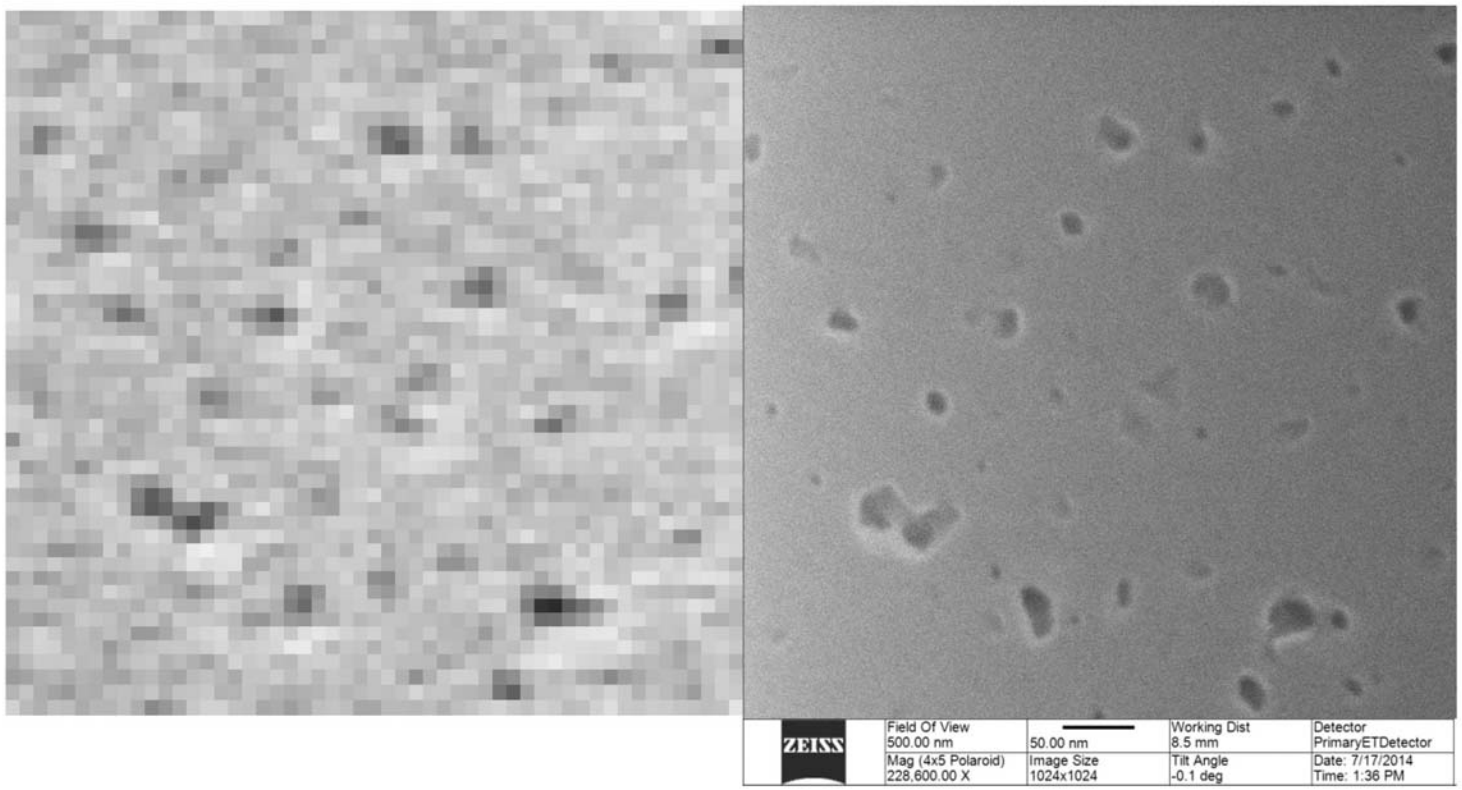

Extremely high resolution in HIM, pixel size below 0.5 nm. Smallest pore measured was 4 nm in diameter.

Presenter's notes: Super high resolution capability of helium ion, right image pixel size below 0.5 nm.

Porosity Comparison

Helium Ion vs. FE-SEM

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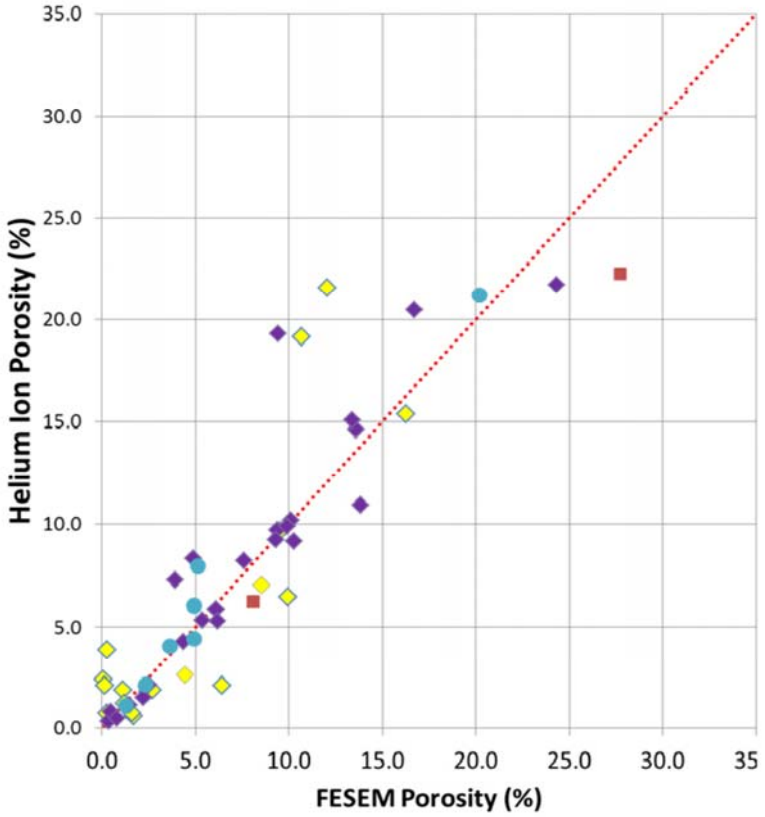
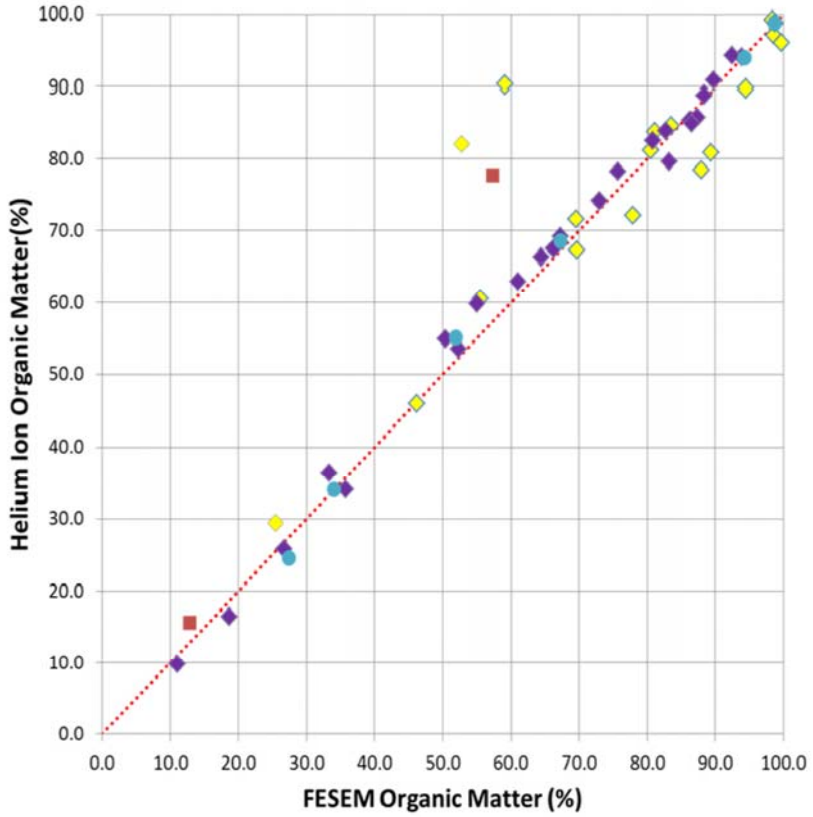
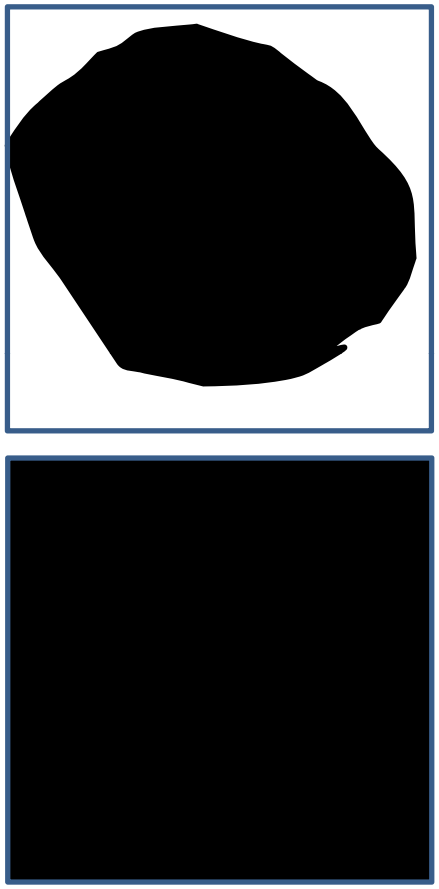


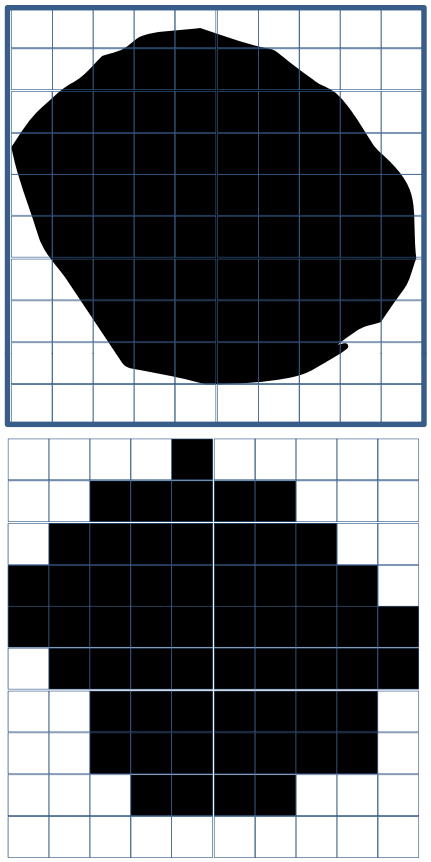


Presenter's notes: 51 subsample locations, comparison of segmented porosity and TOC at each site; a few outliers but a good relationship.

How can increasing resolution lead to decrease in calculated porosity?



Sub 10-nm pore, SEM: Entire 10nm x 10nm pixel likely classified as pore space



Sub 10-nm pore, HIM, 1 nm pixel size. 62/100 pixels classified as pore- 38% porosity reduction

Pore Size Analysis

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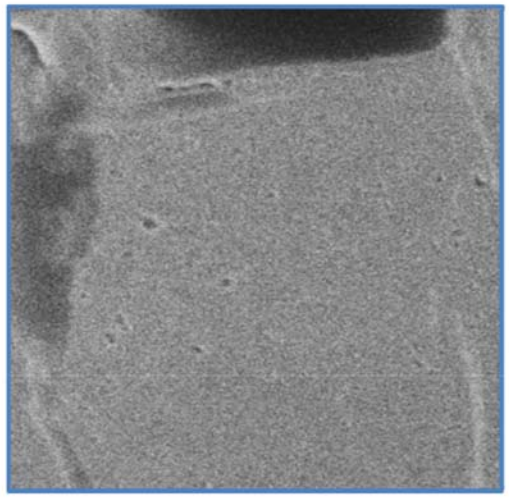
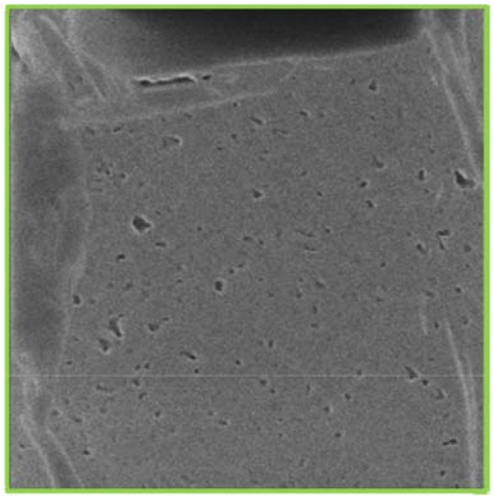
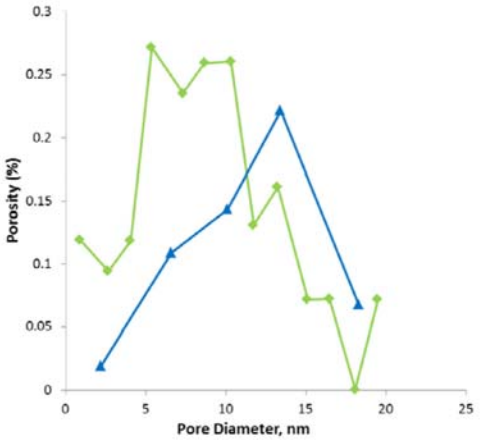
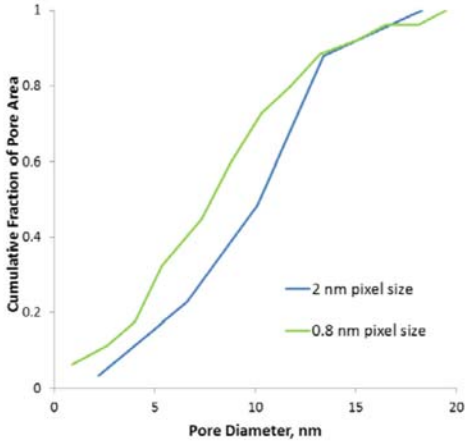


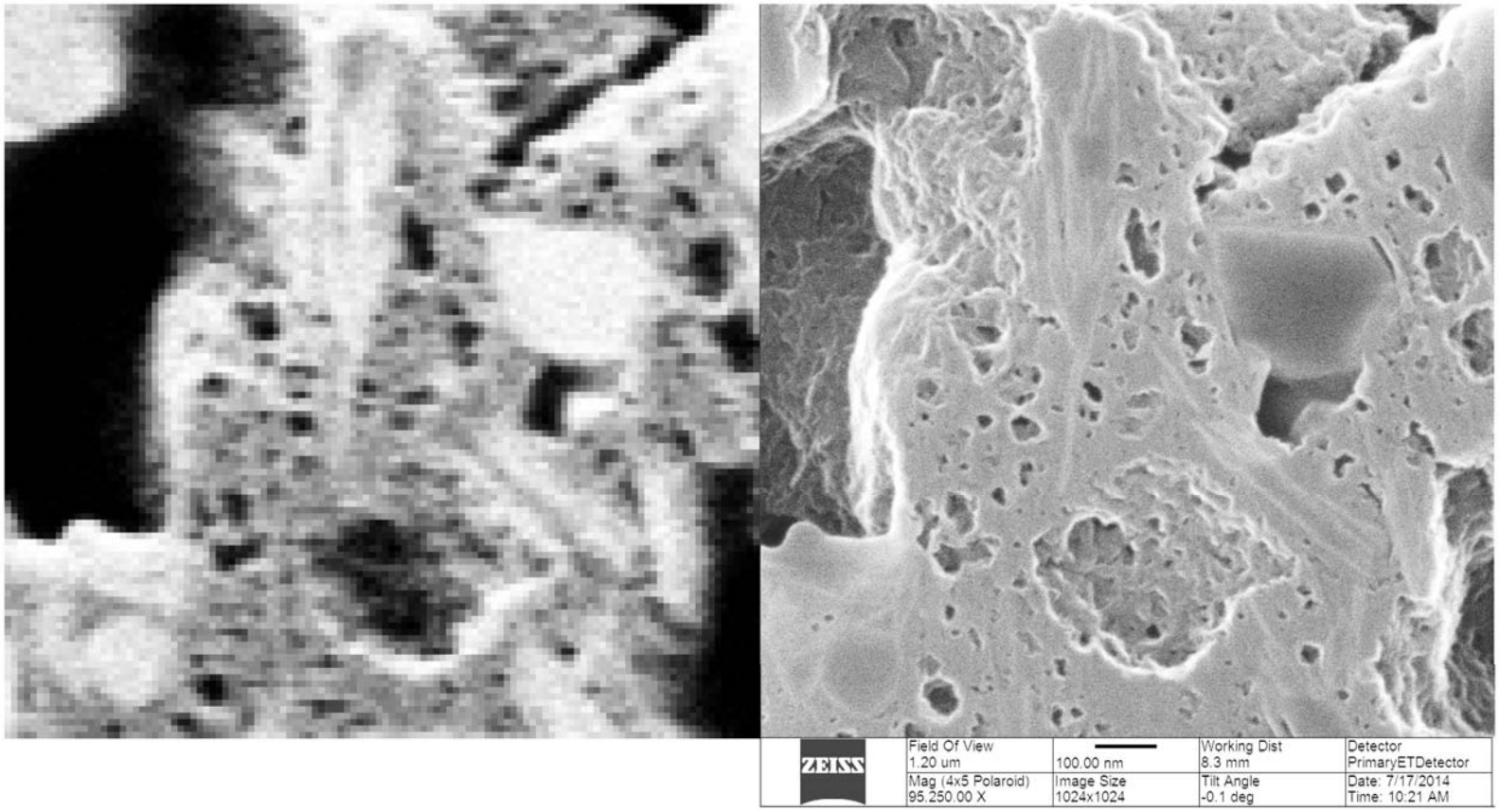






Presenter's notes: Quantifying ability to detect sub-resolution pores- left image 2 nm pixels, right image 0.8 nm pixels, pore size distribution confirms the increased segmentation of sub 10 nm diameter pores (4-5 pixels necessary for feature resolution).

Depth of Field



Presenter's notes: Higher energy of ion beam (larger particles) = ability to produce secondary electrons from surface at a different focal plane (inside pore) .

Organic Matter Imaging

Hidden Textures

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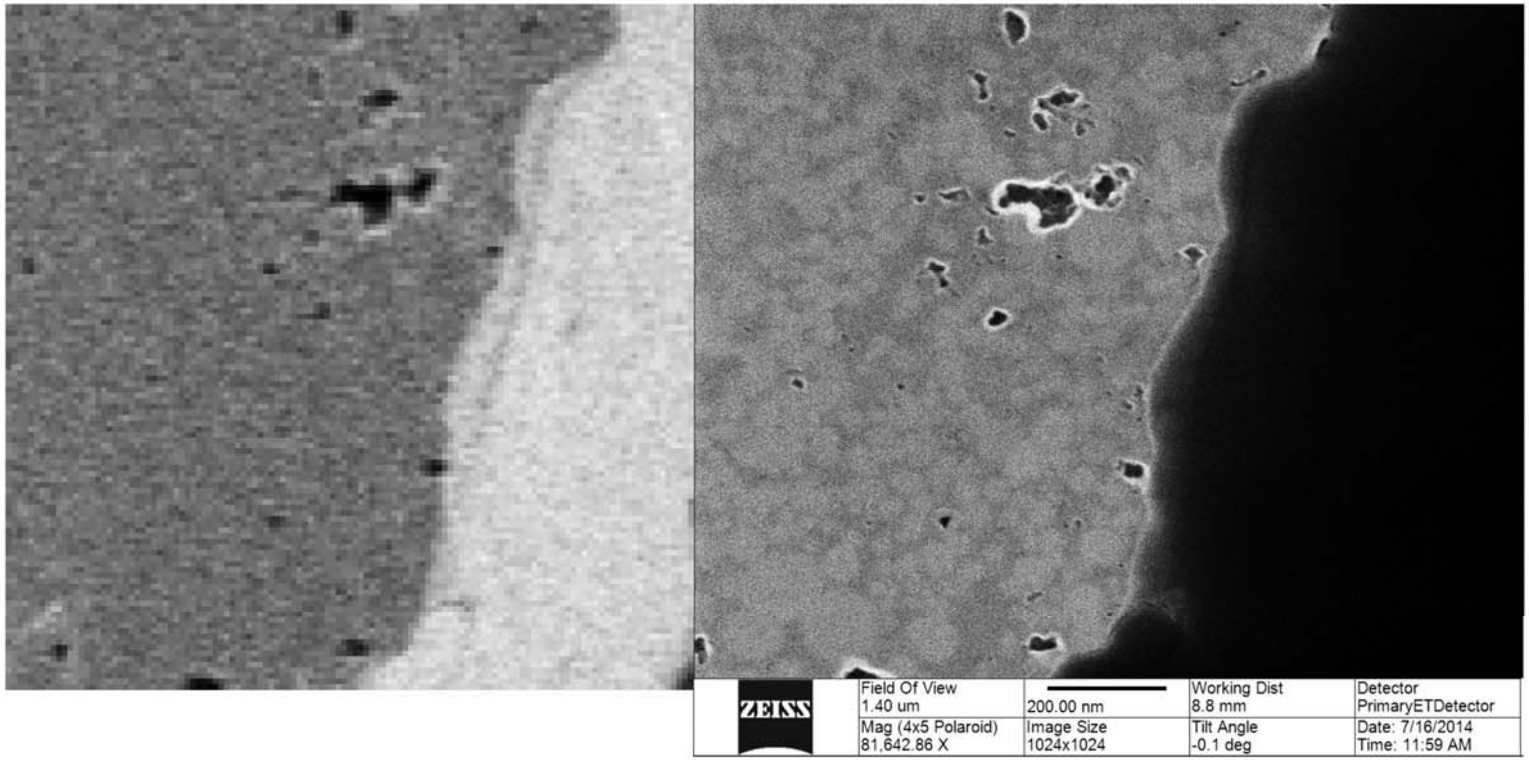

Presenter's notes: Some kind of evidence of textural variation within organic matter bodies- qualitative but seems real, not visible in SEM.

Organic Matter Imaging

Porosity as a function of texture

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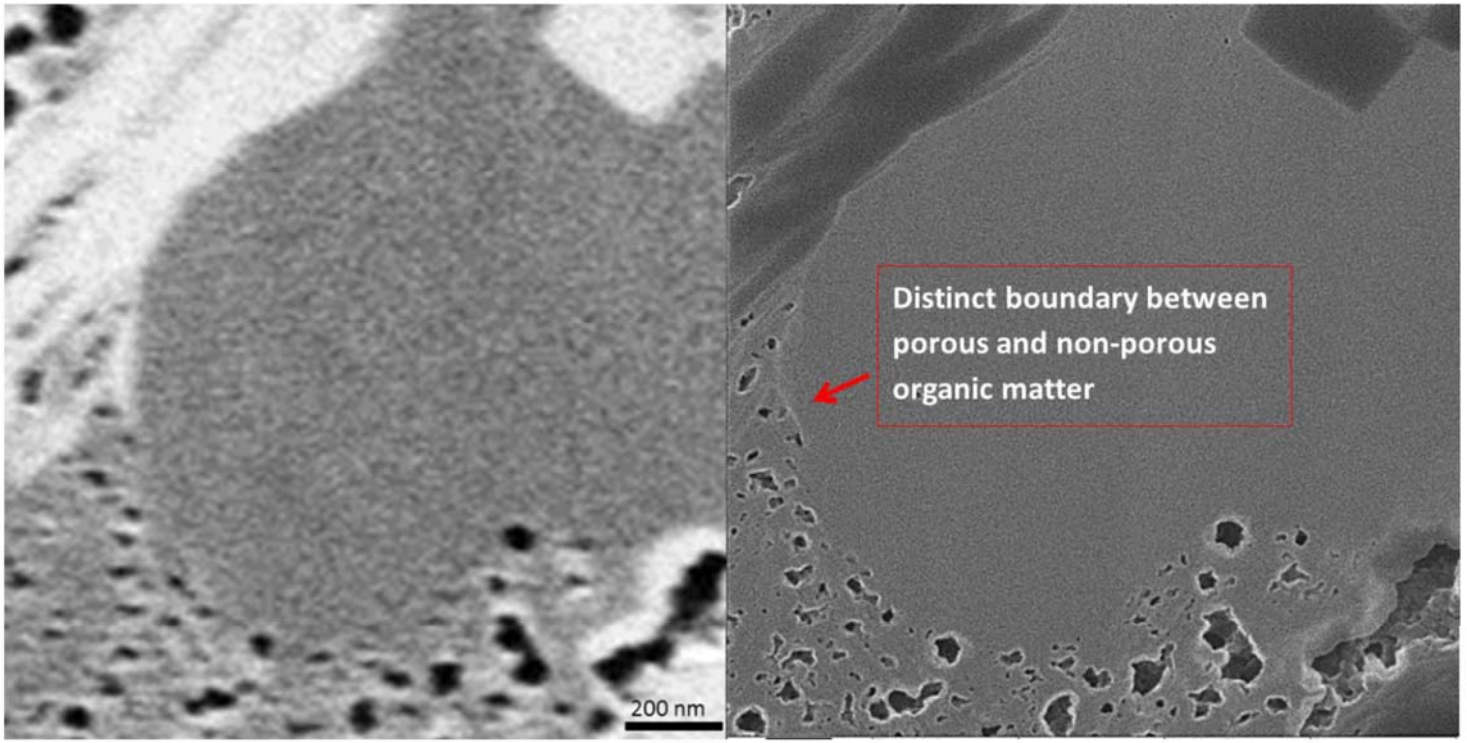

Presenter's notes: Boundary between clearly different types of organic matter, HIM confirms no sub resolution porosity from SEM.

Organic Matter Imaging

Textures Continued

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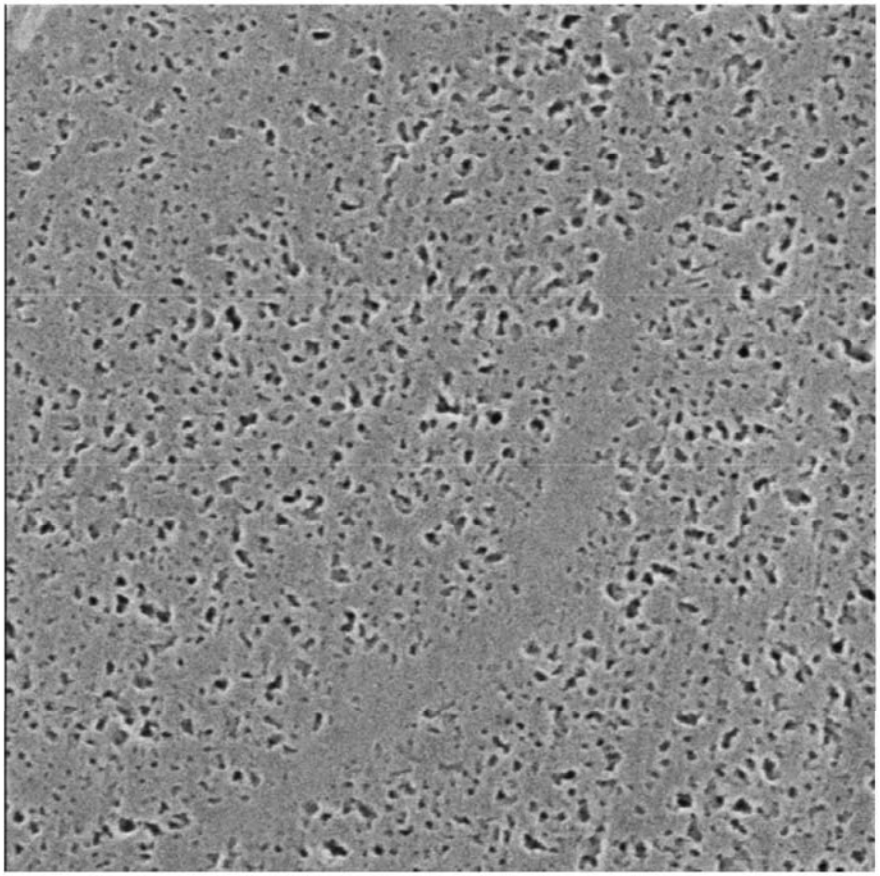

Presenter's notes: More evidence of structure, some kind of remnant structural feature inside porous OM.

Mineral Matrix Effects

Inversion of Gray Scale

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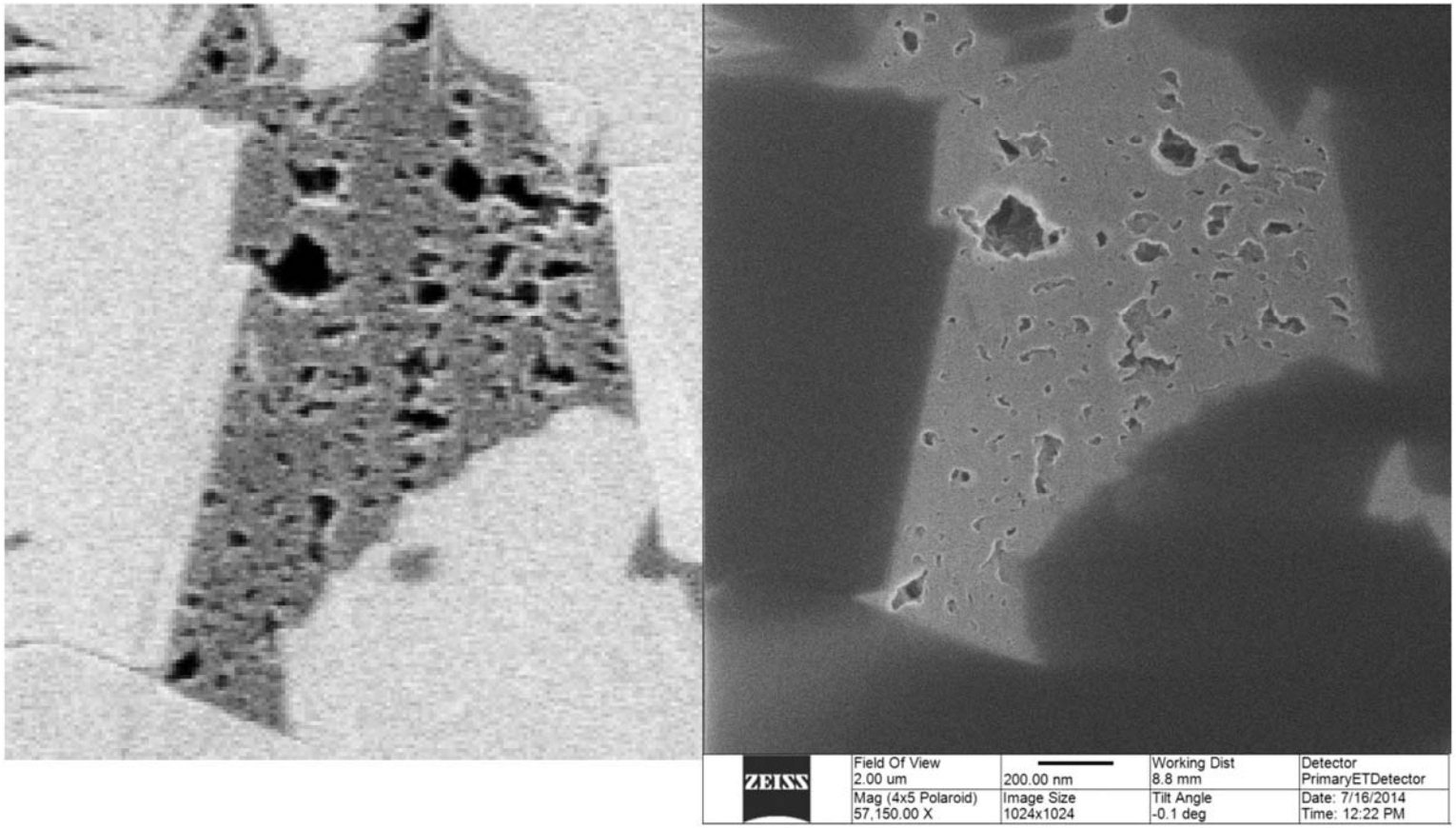

Presenter's notes: Contrast almost inverted from standard SEM results.

Mineral Matrix Effects

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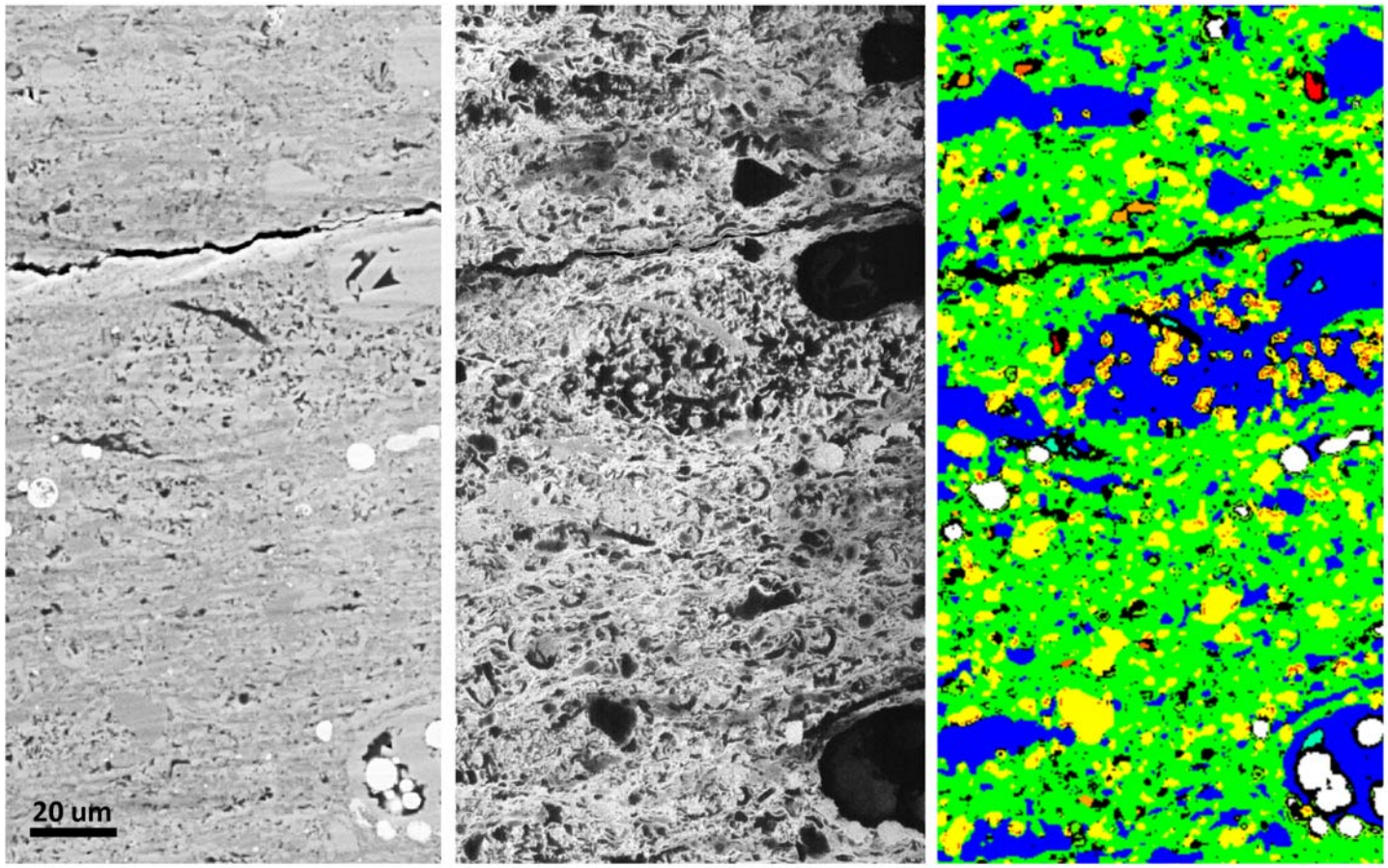

Presenter's notes: EDS Analysis of same area shows that darker regions from helium images correspond to carbonate and silicate grains, not to organic matter or clay rich regions.

Mineral Matrix Effects

Clay Imaging

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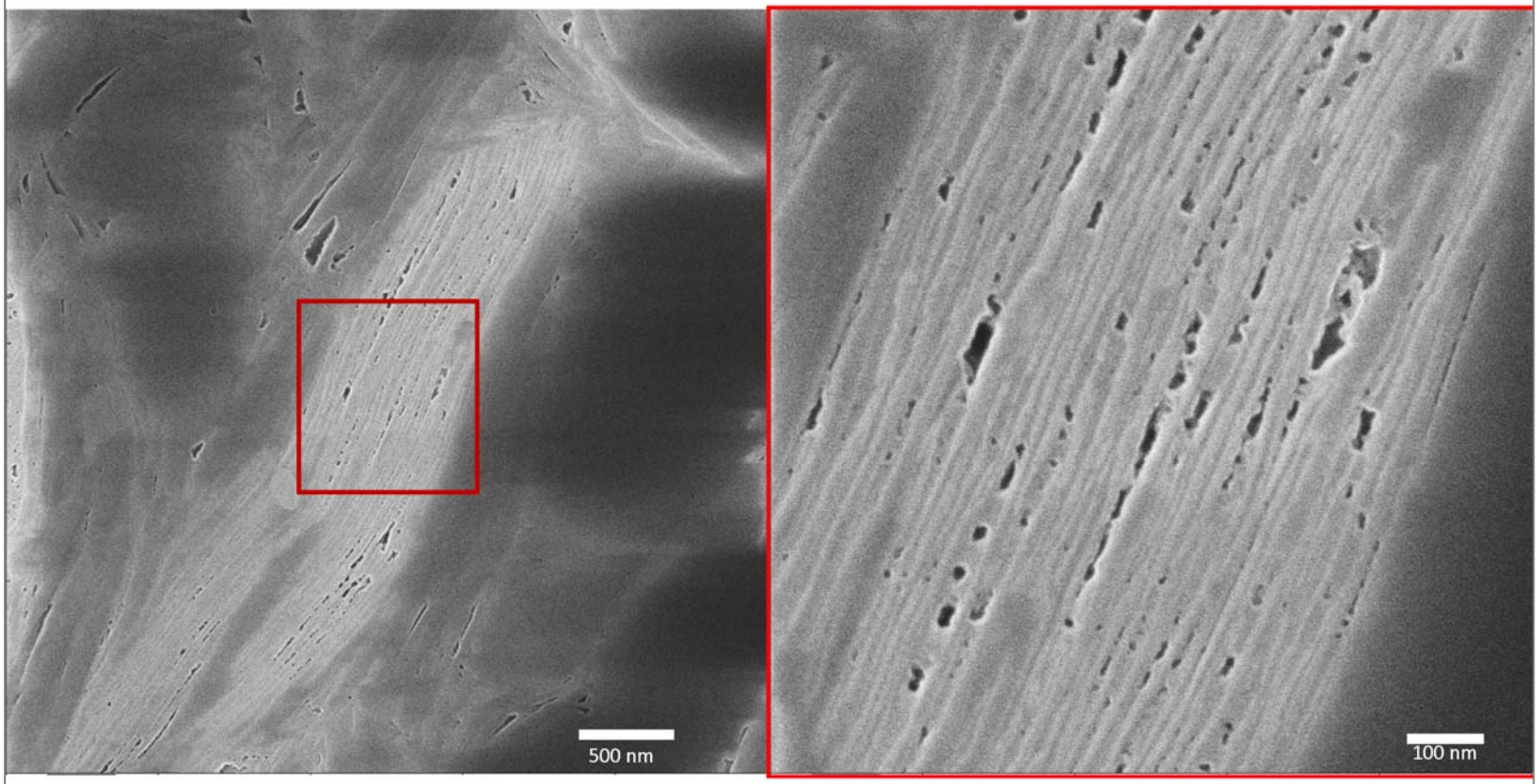
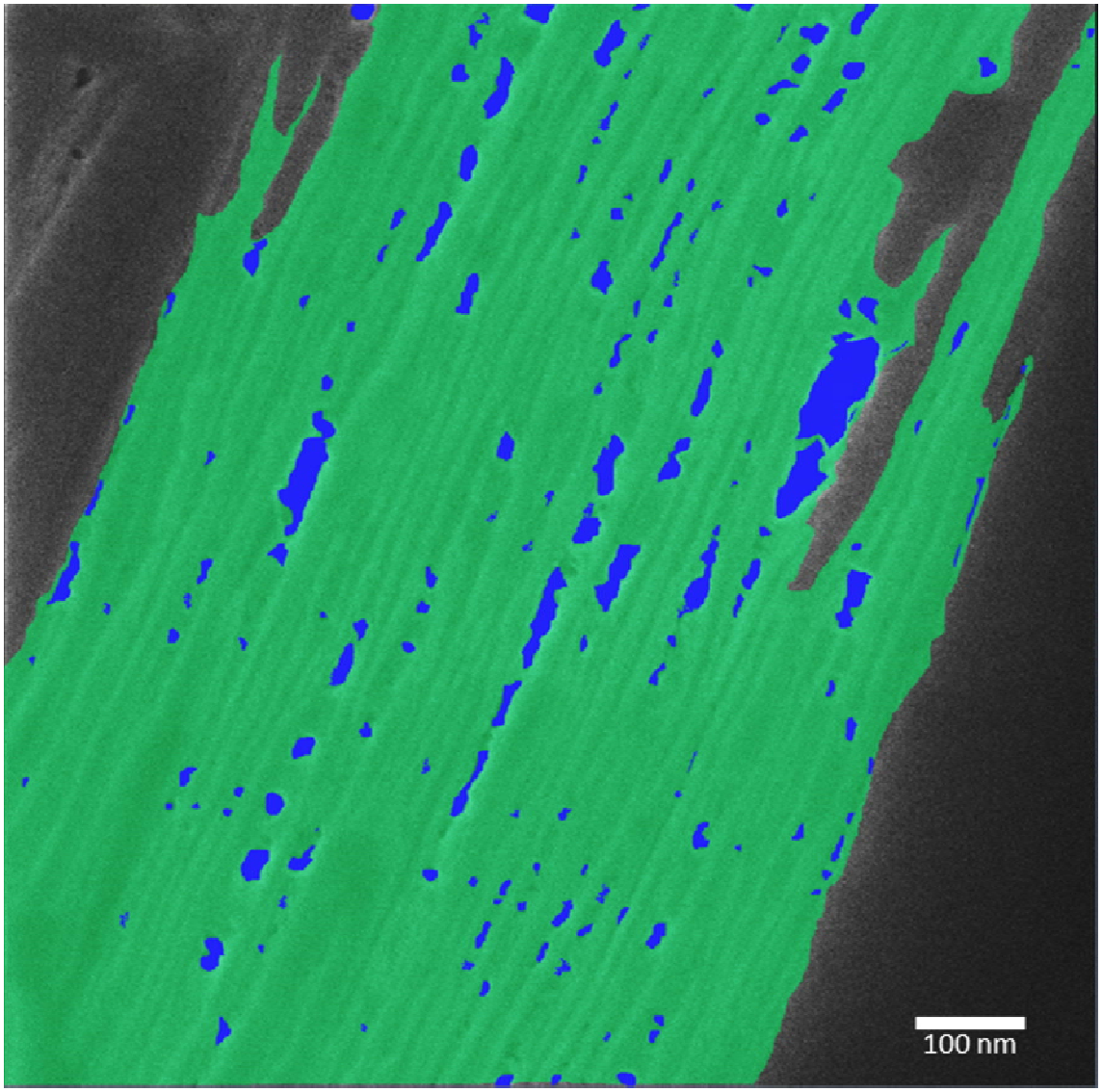


Presenter's notes: Clay (or other phyllosilicate) particle with evidence of pore and other interlayer material.



Segmentation result. Image pixel size is 0.97 nm. Green area represents boundary of clay particle; blue regions represent segmented porosity within clay particle. Total segmented porosity from this image is 4.71%.

Summary of Results

- Much better resolution on edges of organic matter-hosted pores
- Difficulty in resolving intergranular pores
- Complex mechanism of grayscale contrast formation
- Should be viewed as a complementary technique to FE-SEM, not a replacement

Thank You!