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## Introduction

Characterizing heterogeneity from pore (nanometer) to larger length scales is a major challenge to understanding coupled processes in shale. To address this challenge, we present application of high throughput multi-beam scanning electron microscopy (mSEM) to shale characterization. The mSEM collects ~4 nm resolution SEM images at the scale of several square millimeters in a short period of time. We analyze mSEM images for pore size and shape characteristics including spatial correlation and structure. Nano-indentation, micropillar compression, and axisymmetric testing at multiple length scales allows for examining the influence of sampling size on mechanical response. The combined data set is used to: investigate representative elementary volume (and areas for the 2D images) for shale samples; determine if scale separation occurs; and determine if transport and mechanical properties at a given length scale can be statistically defined.

## Research Questions and Methods

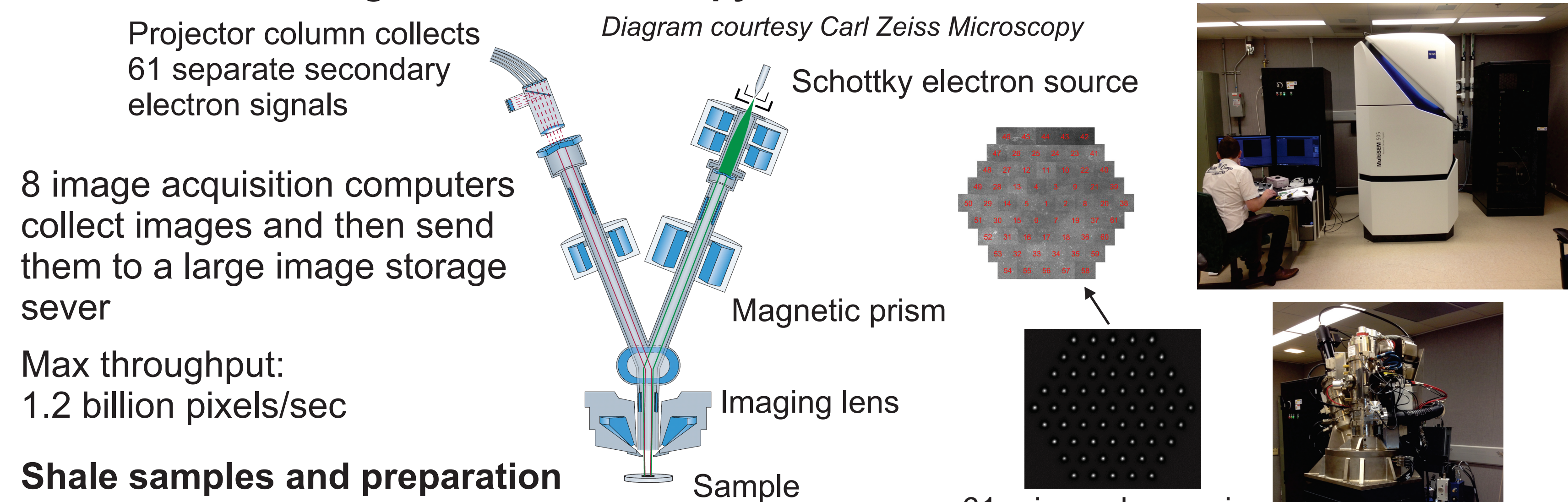
### Research questions

- Does "scale separation" occur between the microscale pore structure and macroscale response for mechanical and flow and transport processes in shale?
- What are the representative elementary volumes for different types of shale?
- What is the multiscale nature of porosity in shale?

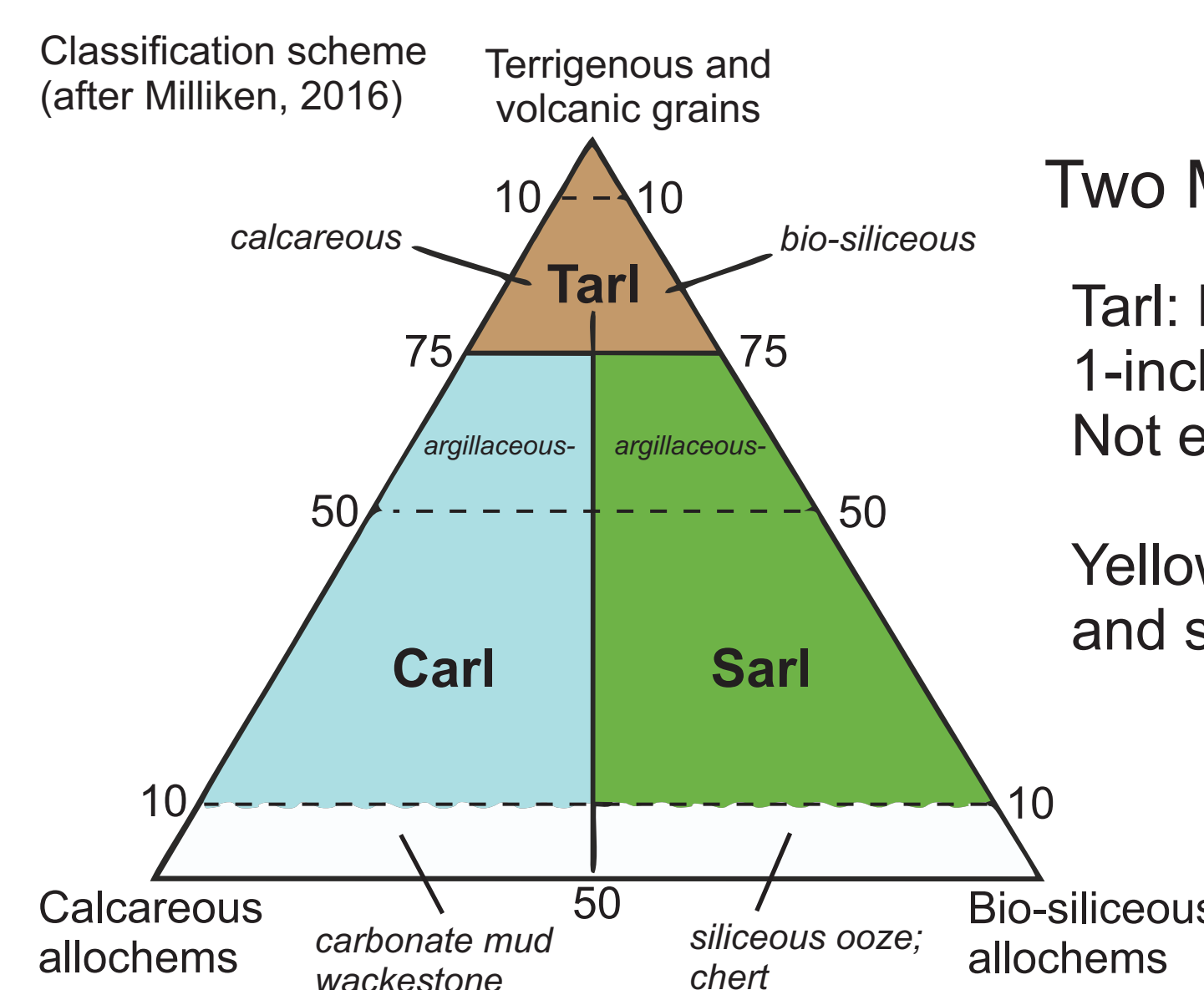
### Approach

- Apply high-throughput mSEM imaging to examine nano-scale pores over areas up to square millimeters.
- Test mSEM on multiple types of shale to evaluate usefulness of mSEM for shale studies.
- Extract pore characteristics from mSEM images and analyze for representative elementary area.
- Combine mSEM-based knowledge on shale pores with petrographic and other data types.

### Multi-beam scanning electron microscopy



### Shale samples and preparation



### Two Mesozoic Tarl and Sarl shale samples

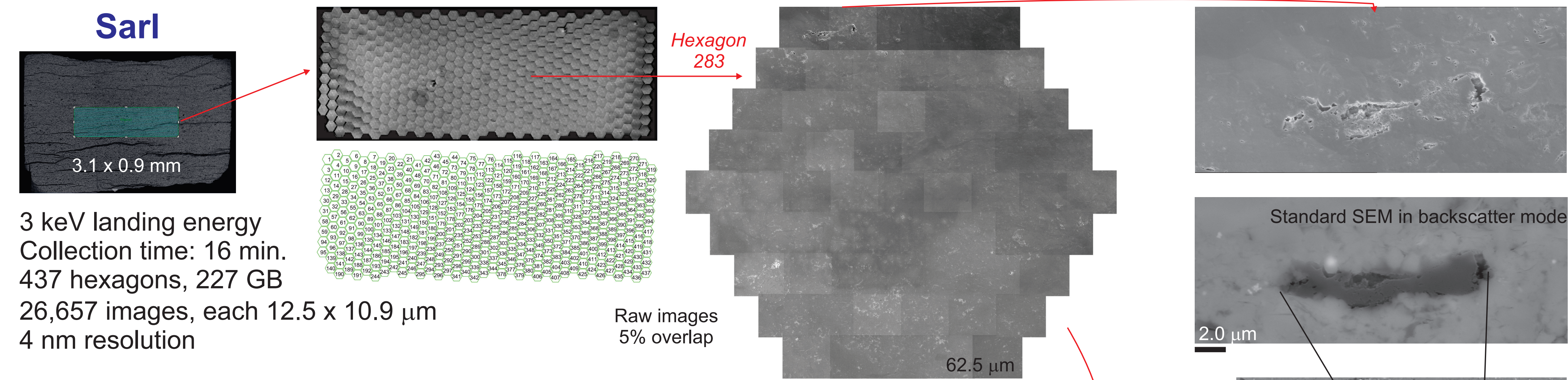
Tarl: Mancos Shale  
1-inch plug  
Not epoxied

Yellow: region of EDS mapping and standard backscatter SEM

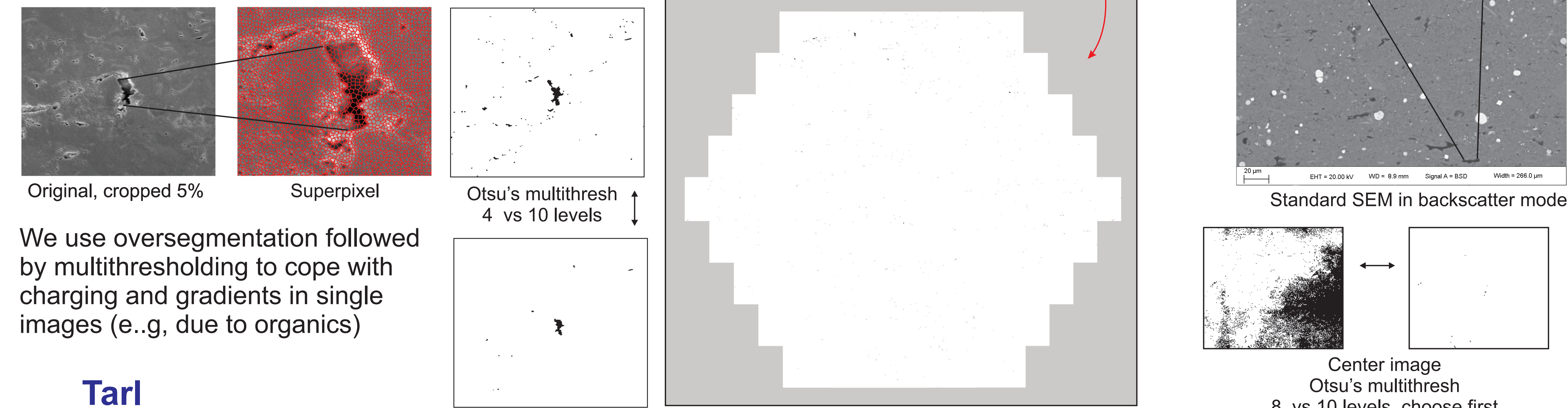
Broad ion-beam (BIB) polishing was performed on the Tarl and Sarl samples for nm-flat samples

Sarl: A non-epoxied Mesozoic siliceous shale sample was provided by Kitty Milliken, Bureau of Economic Geology, UT-Austin

## mSEM Results, Pore Identification, and Mechanics

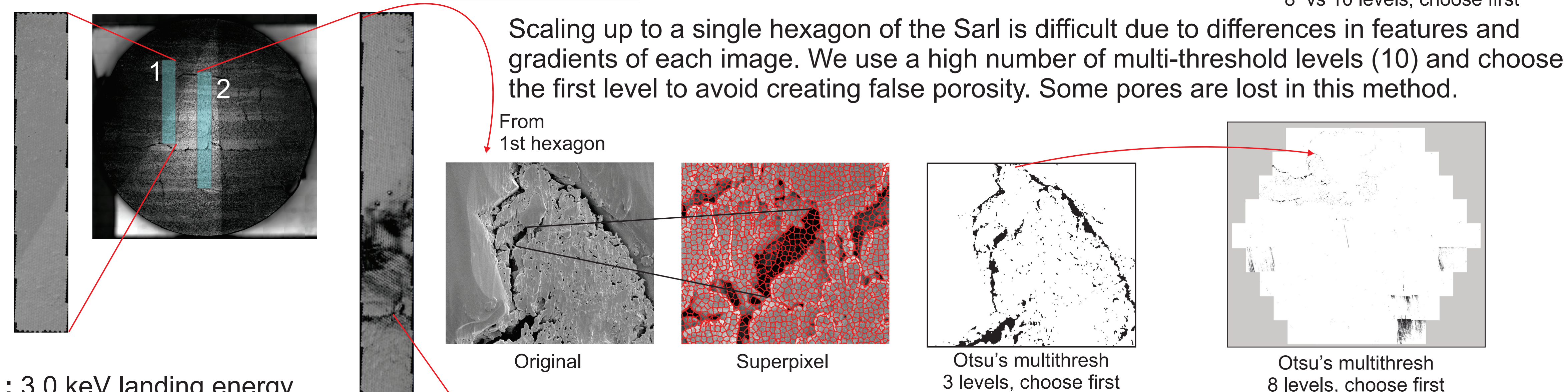


### Automatic pore identification: filter then segment



We use oversegmentation followed by multithresholding to cope with charging and gradients in single images (e.g., due to organics)

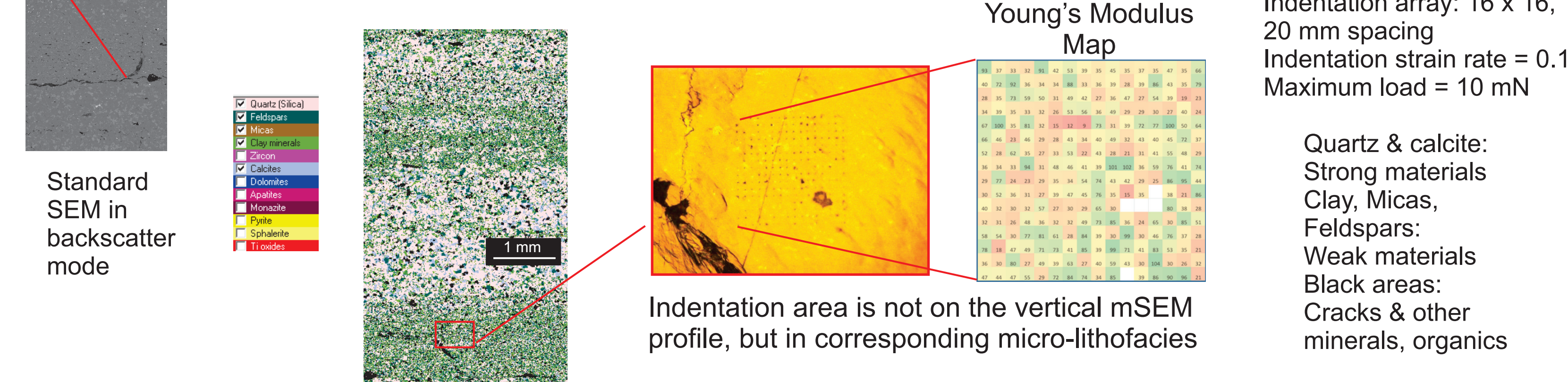
### Tarl



1: 3.0 keV landing energy  
1.5 x 10 mm  
Collection time: 104 min.  
2,296 hexagons, 140,056 images  
1.08 TB, 12.5 x 10.9 mm

2: 1.5 keV landing energy  
1.5 x 14 mm  
Collection time: 115 min.  
3,122 hexagons, 1.47 TB  
190,442 images at 12.5 x 10.9 mm

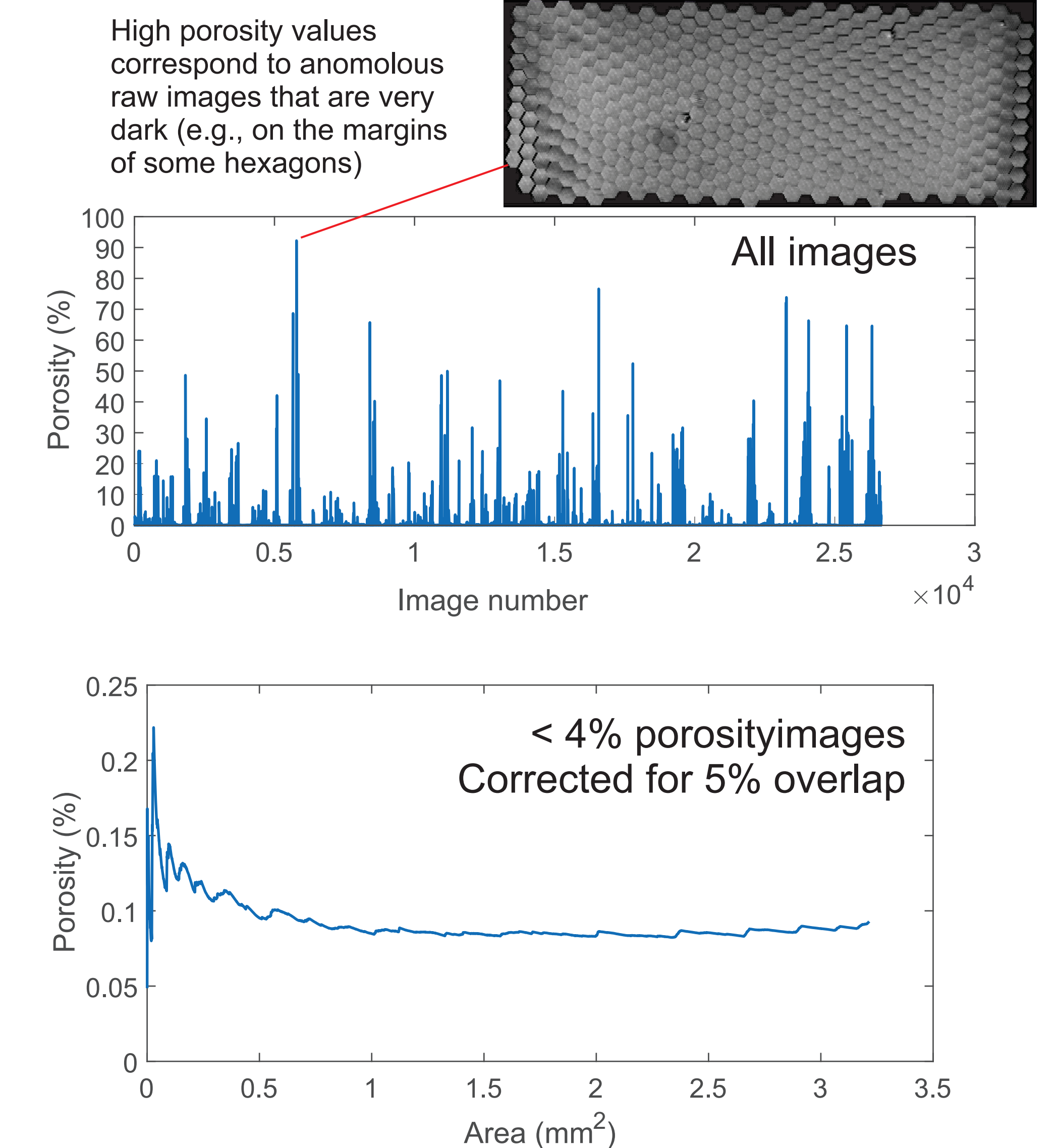
### Nano-indentation



## Multiscale Porosity

### Preliminary imaging processing scale-up on 200 cores

- Using superpixel/multithreshold method with 10 levels, selecting the first level
- All 437 hexagons of Sarl images are processed
- Using the segmented images, the number of black pixels over total pixels for a given area is the porosity



## Conclusions & Future Work

- For the Sarl, mSEM image analysis suggests that a stable representative elementary area (surrogate for REV) occurs.
- Based on the porosity REV, nano-indentation may be below the mechanical REV of the Sarl (yet to be tested).
- Multi-beam SEM collects many useful images, but adaptive algorithms are needed to properly cope with gradients in images and other feature variability.
- Future work will include analyzing representative elementary areas for the micro-lithofacies and synthesizing with the mechanical data. We will further validate full-scale processing methods.

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