On the prospects for **nanometer-scale *in situ* geomechanics of shale** with laboratory X-ray microscopy and comparison with DRP

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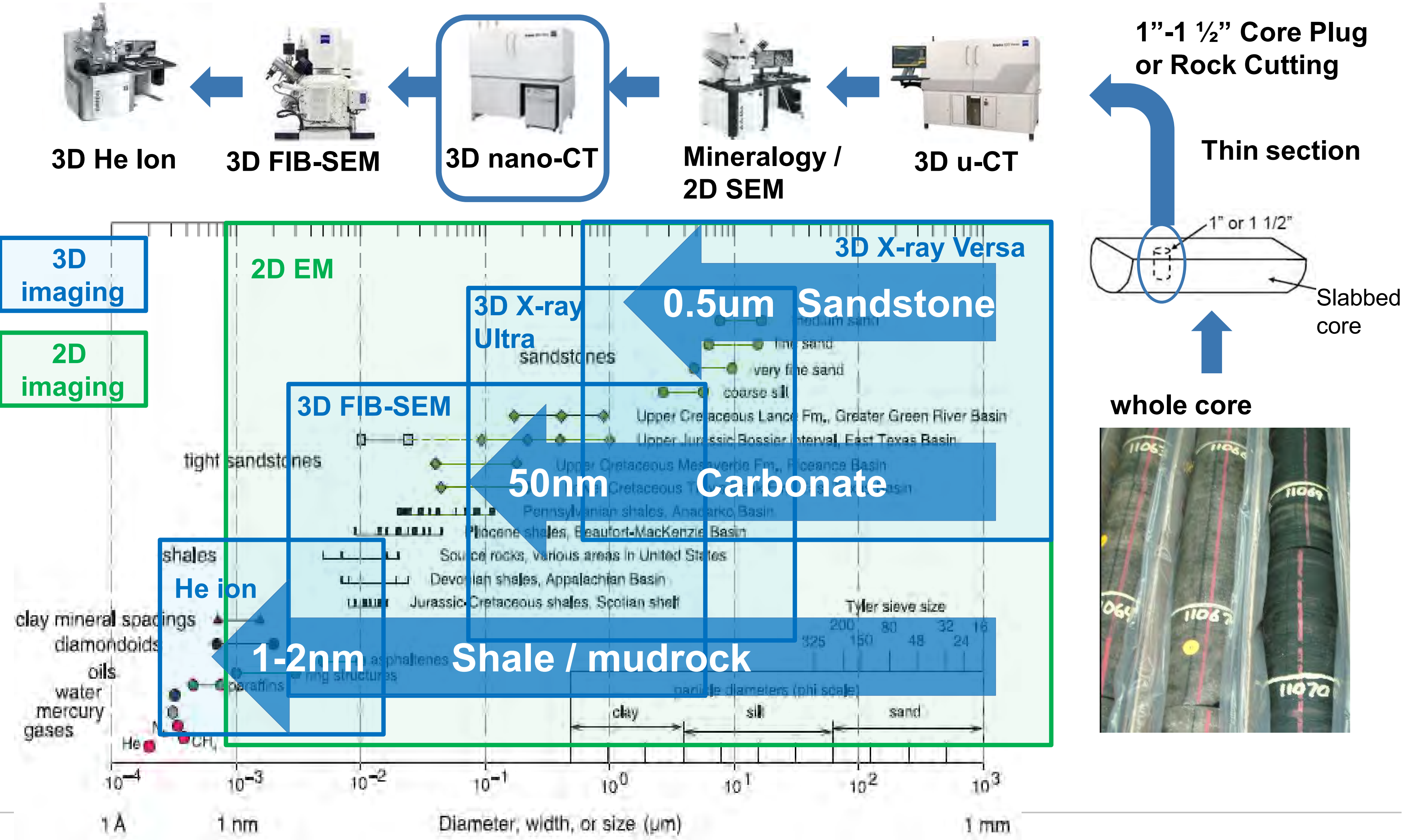
Carl Zeiss X-ray Microscopy

AGENDA

- **Introduction**
  - Context & experimental apparatus
- **In situ experiment on shale**
- **Prospects for DRP comparison**

The multiscale challenge in reservoir rock

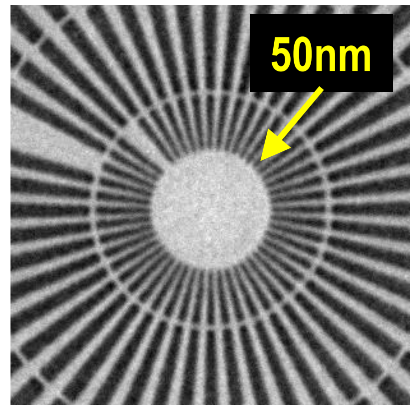
Getting answers across ≥ 8 orders of magnitude



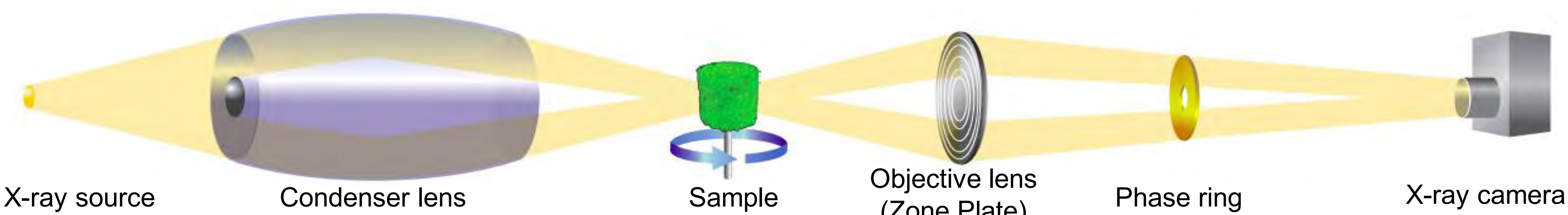
Ultra X-Ray Microscope (XRM)
3D X-ray Nanotomography down to 50 nm Resolution

The only non-destructive, laboratory based 3D imaging solution with resolution down to 50 nm: Ideal for 4D and *in situ* studies

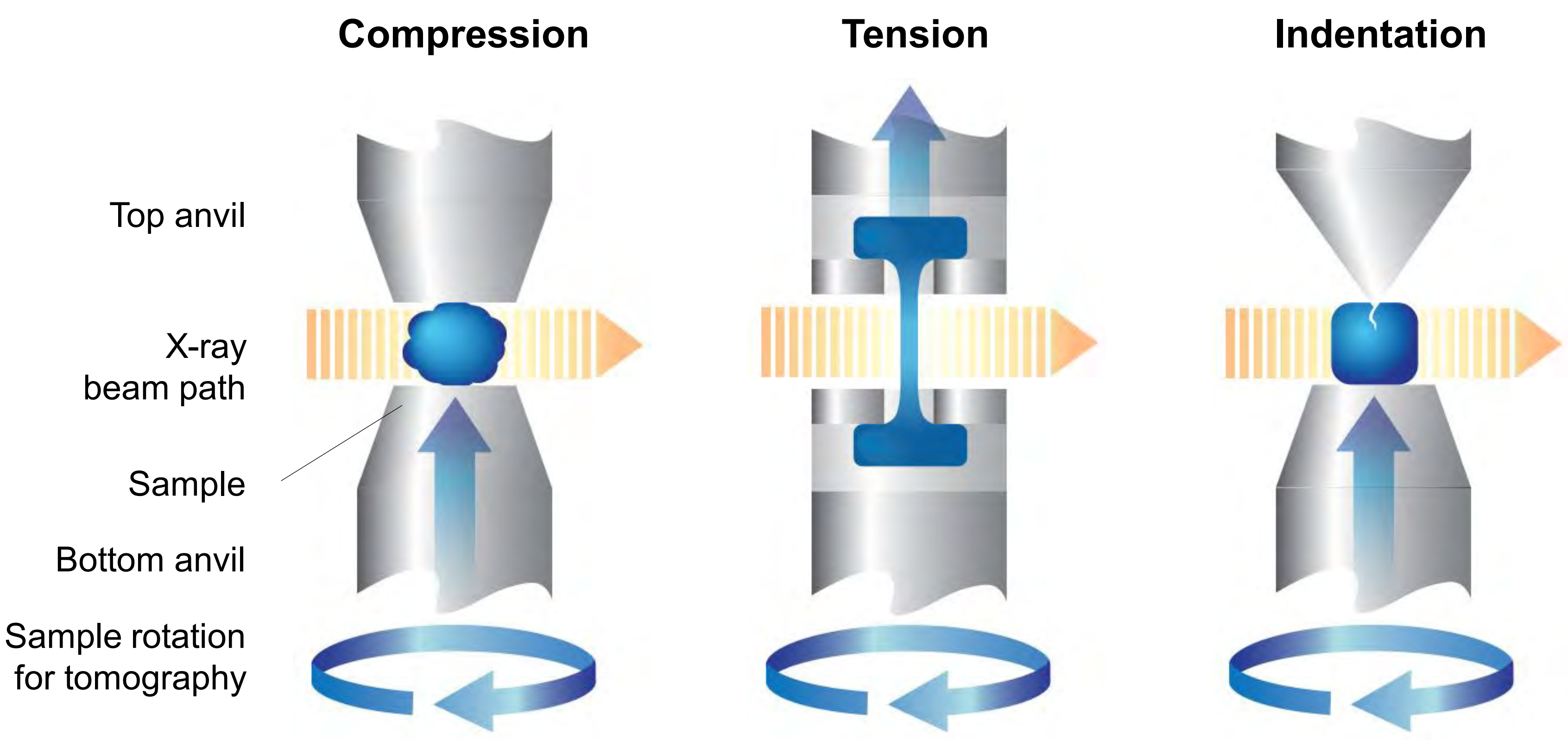
- High brightness X-ray source
  - **810 Ultra: 5.4 keV**
  - **800 Ultra: 8.0 keV**
- 50 nm spatial (16 nm voxel) resolution
- Advanced X-ray optics
- Absorption and Zernike phase contrast



Mode	Mag	2D Res	Voxel	Field of View
Large Field of View	200X	150 nm	64 nm	65 µm x 65 µm
High Resolution	800X	50 nm	16 nm	16 µm x 16 µm



Ultra XRM Load Stage
Enables unique 3D imaging of nanostructure



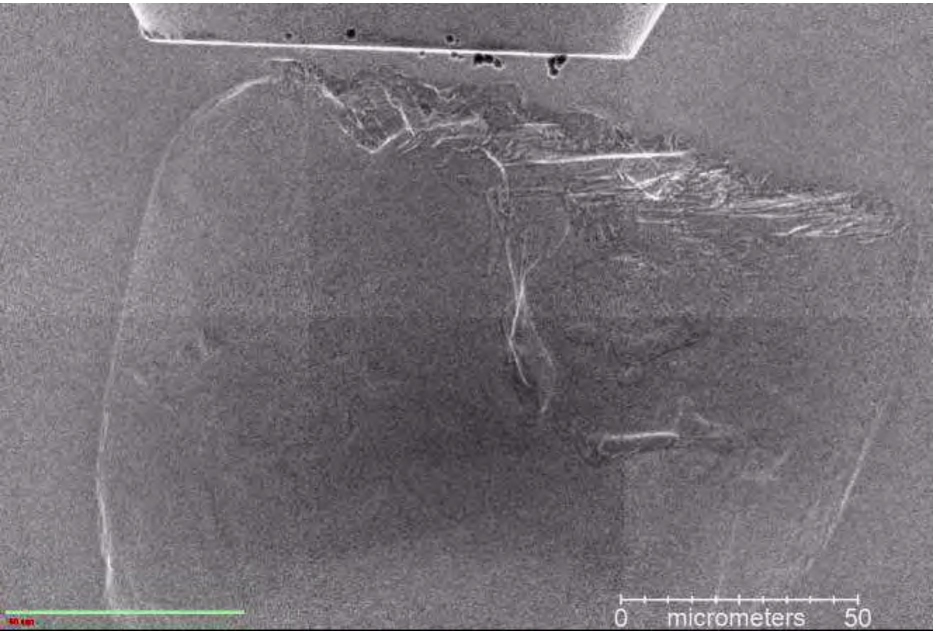
Deformation and failure under uniaxial compressive load

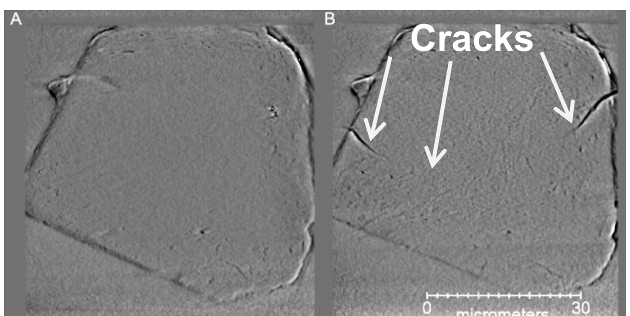
Deformation and failure under uniaxial tensile load

Isolated deformation and failure surrounding the indentation site

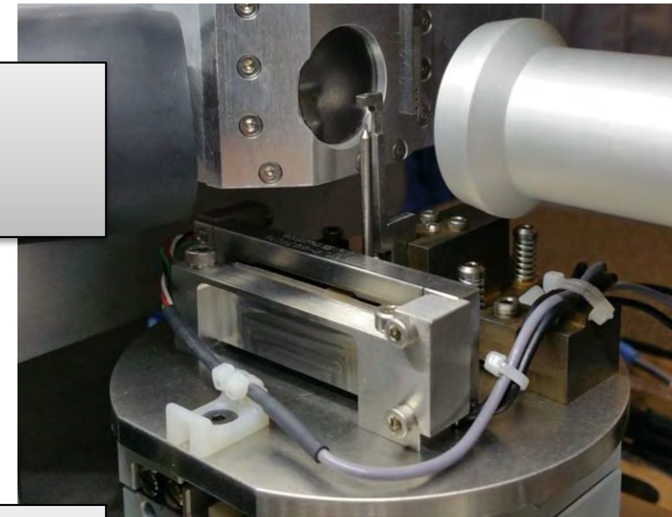
Ultra Load Stage

In Situ Nanomechanical Test Stage for Xradia Ultra

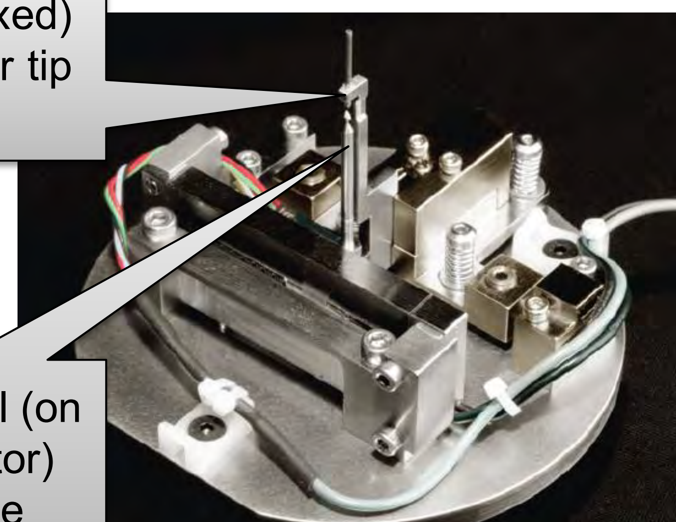




Load Stage installed in 810 Ultra

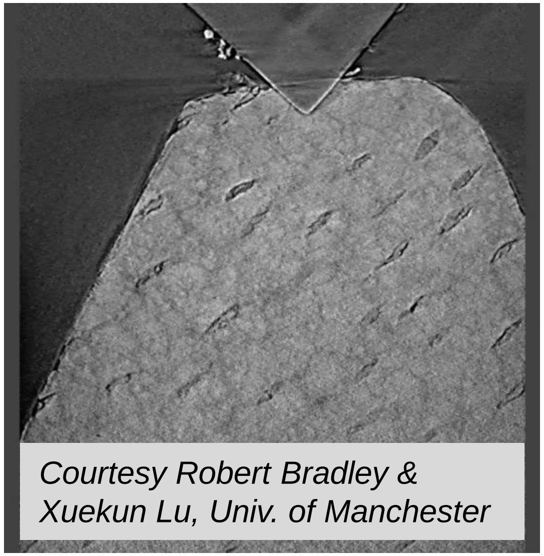


Top anvil (fixed) with indenter tip or flat anvil

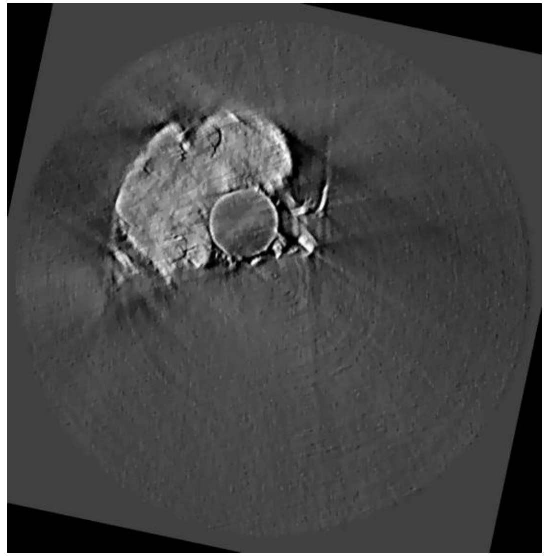


Bottom anvil (on piezo actuator) holds sample

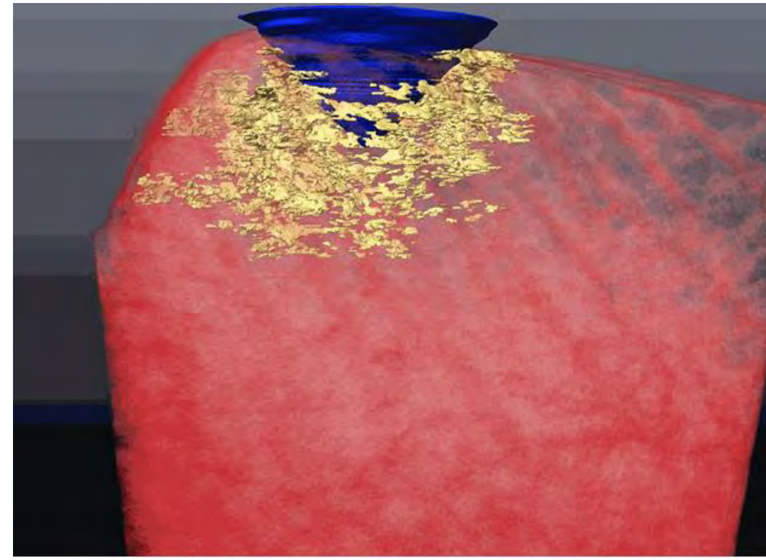
Ultra Load Stage – Indentation:
Crack generation and propagation in dentin



Virtual cross section (vertical)



Virtual cross section (horizontal)



3D rendering

- Goal to study anisotropic fracture toughness and crack tip shielding in dentin
- Helps development of biomimetic restorative materials and oral treatments
- ***In situ* nanoscale XRM helps understand anisotropic fracture behavior relative to orientation of tubules**

Elephant dentin sample mounted in Load Stage, with diamond indenter mounted above

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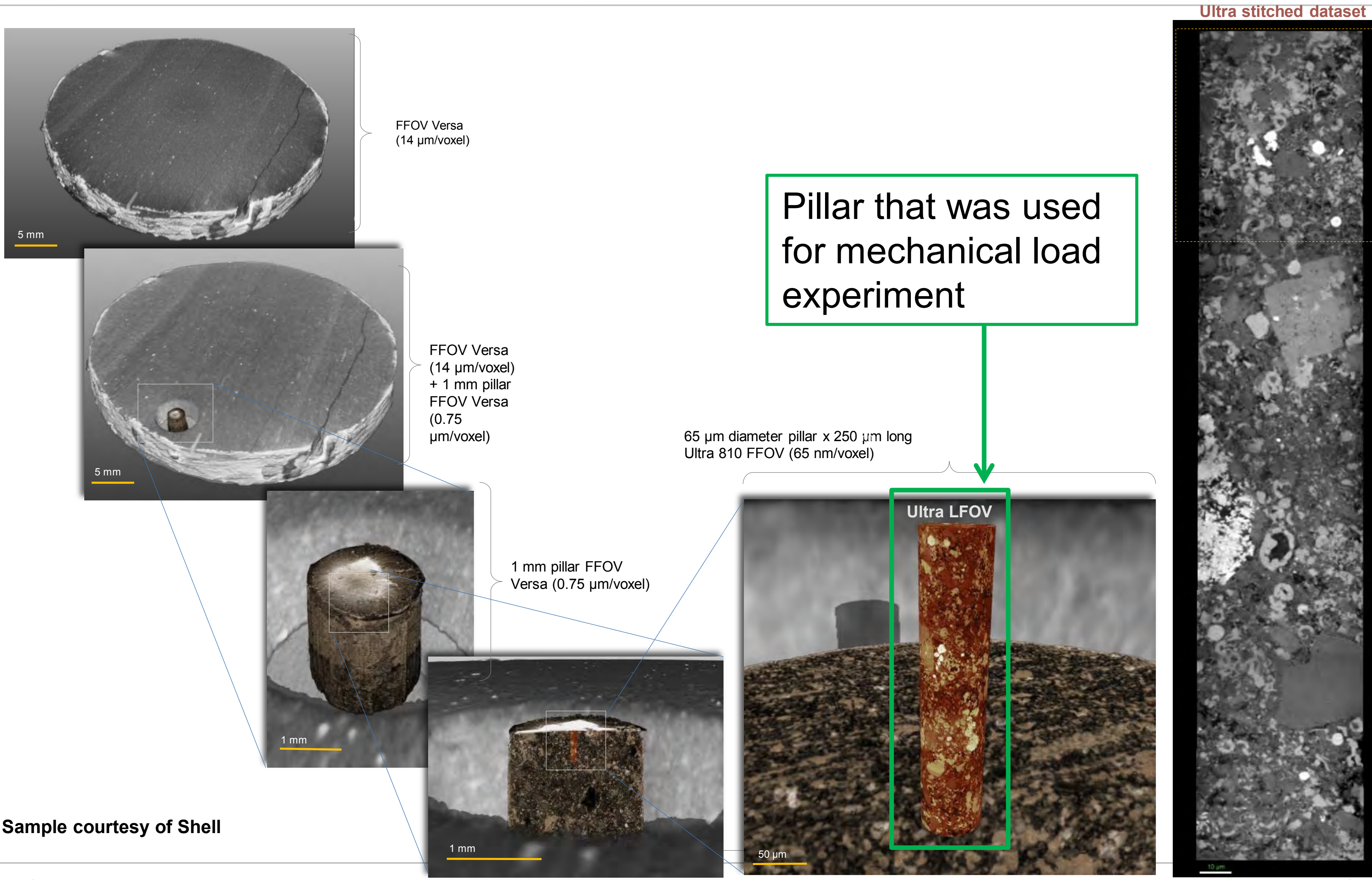
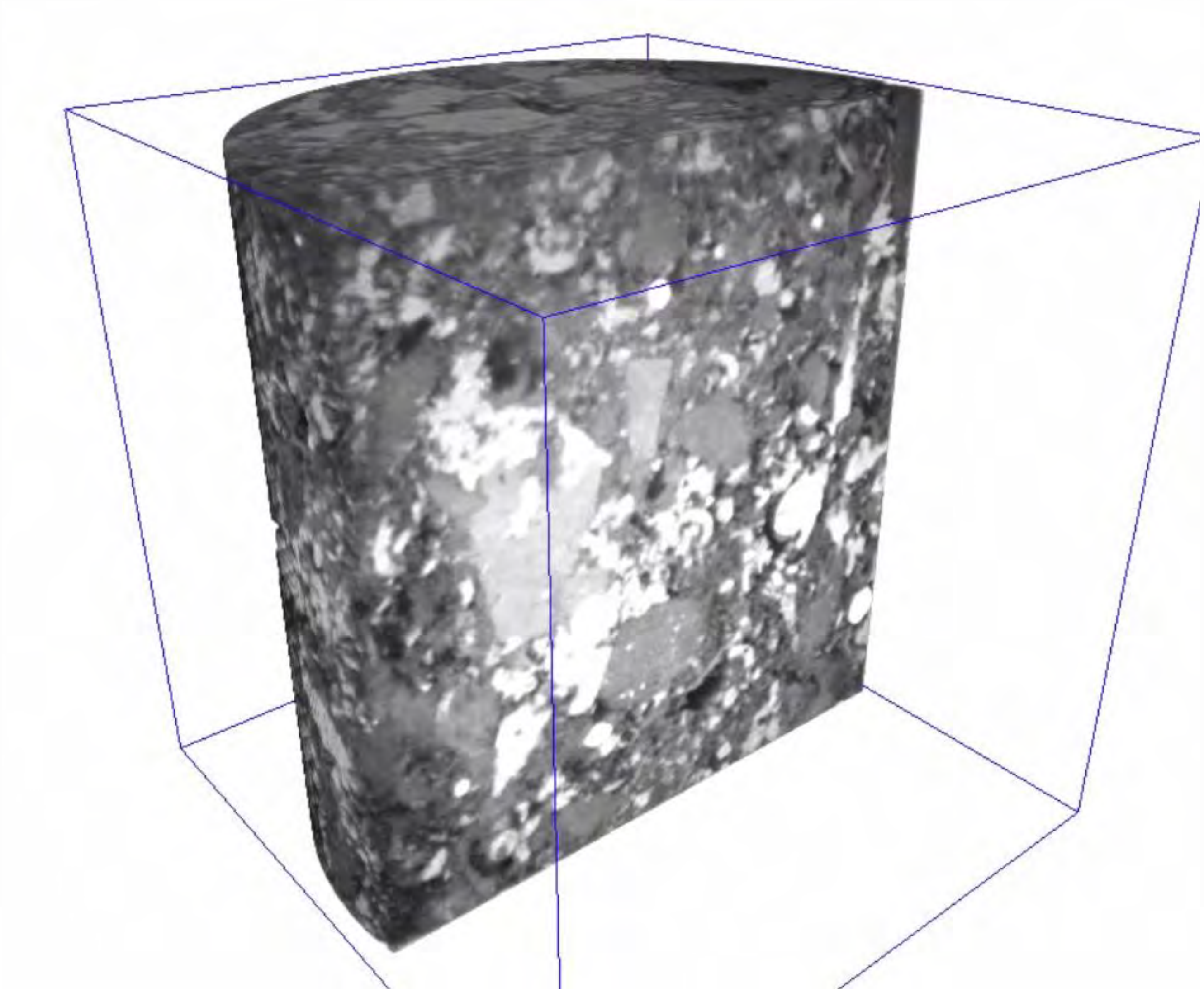
Shale 1” end trim
Multi-scale imaging & sample prep

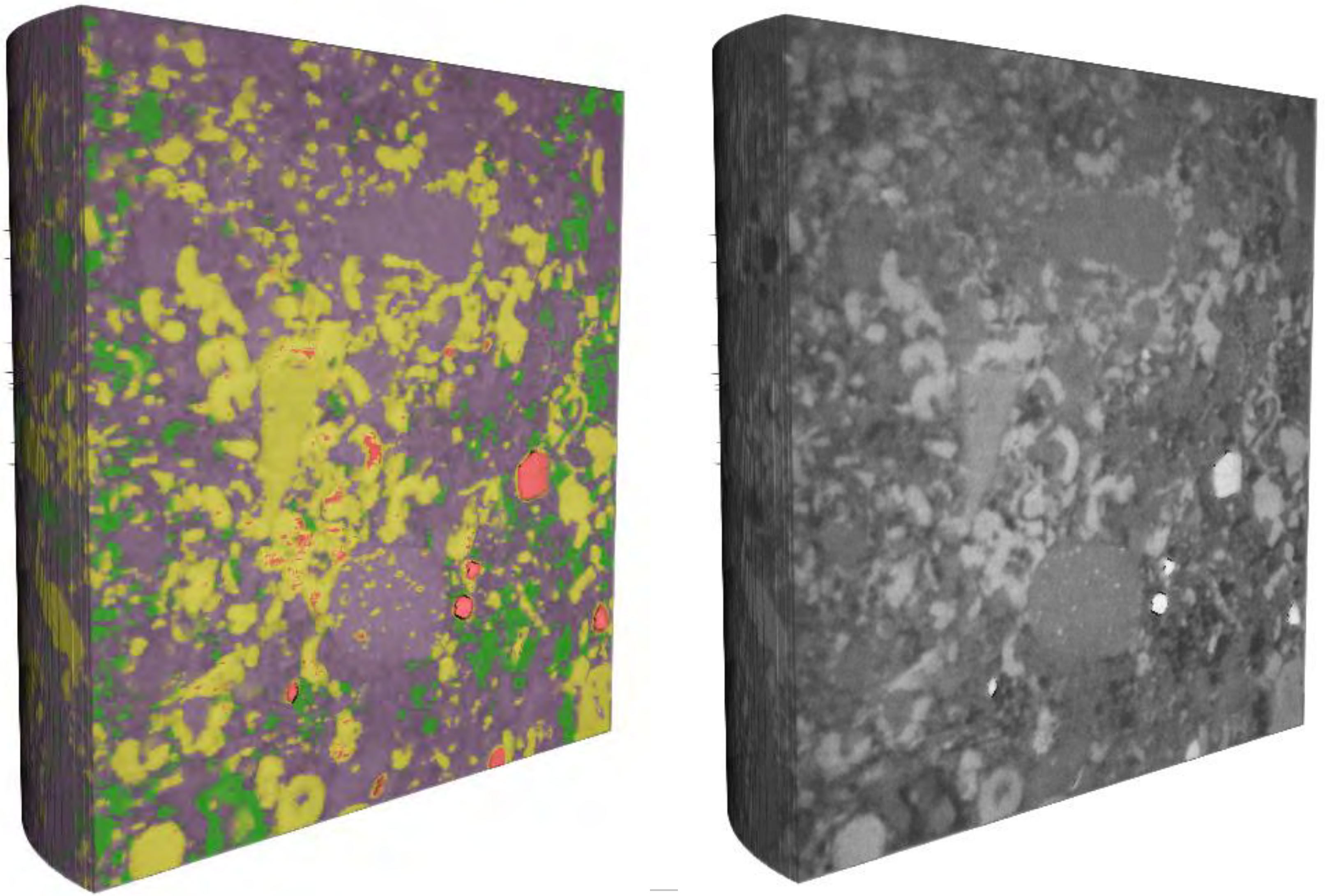


Sample courtesy of Shell

Typical measured shale sample
Laser-prepared ~65 um x 250 um pillar

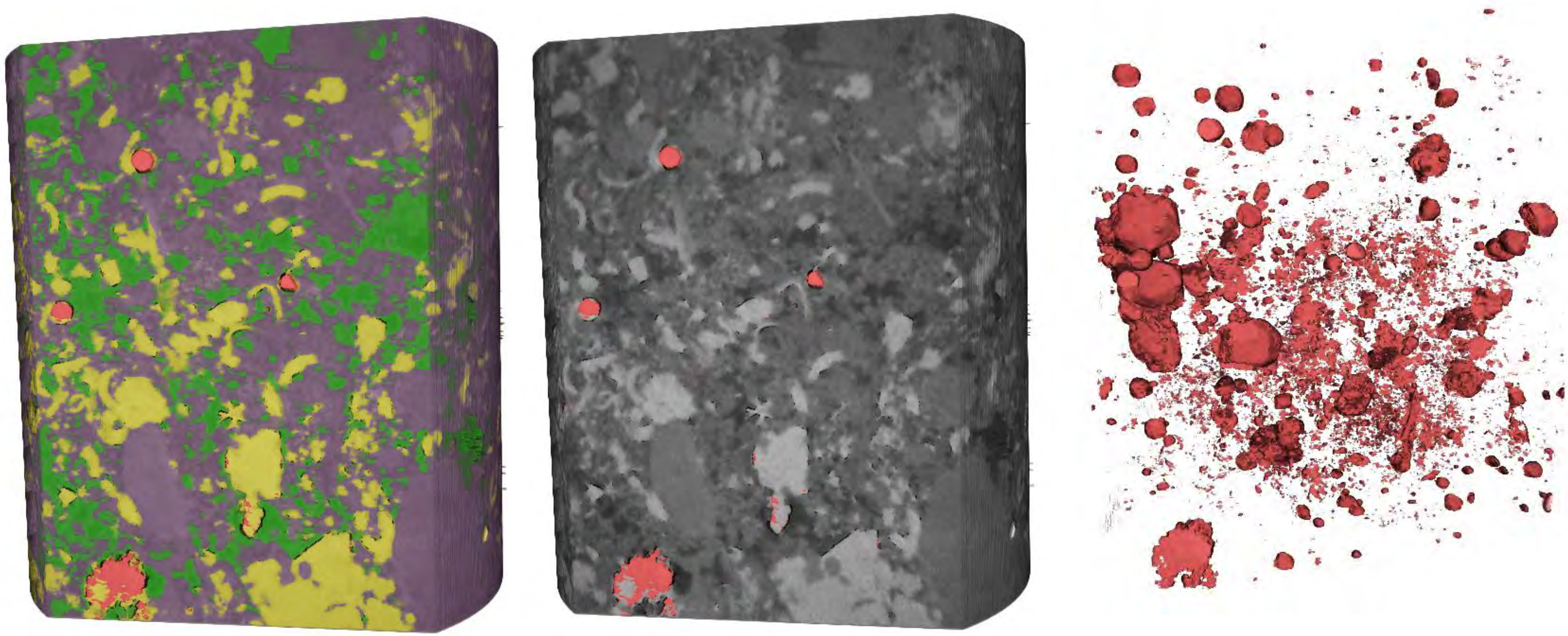


Sections of shale sample
Segmented and raw data



Yellow = Carbonate, Purple = Quartz, Green = Pores/Organics, Red =Pyrites

Sections of shale sample
Segmented and raw data



Yellow = Carbonate
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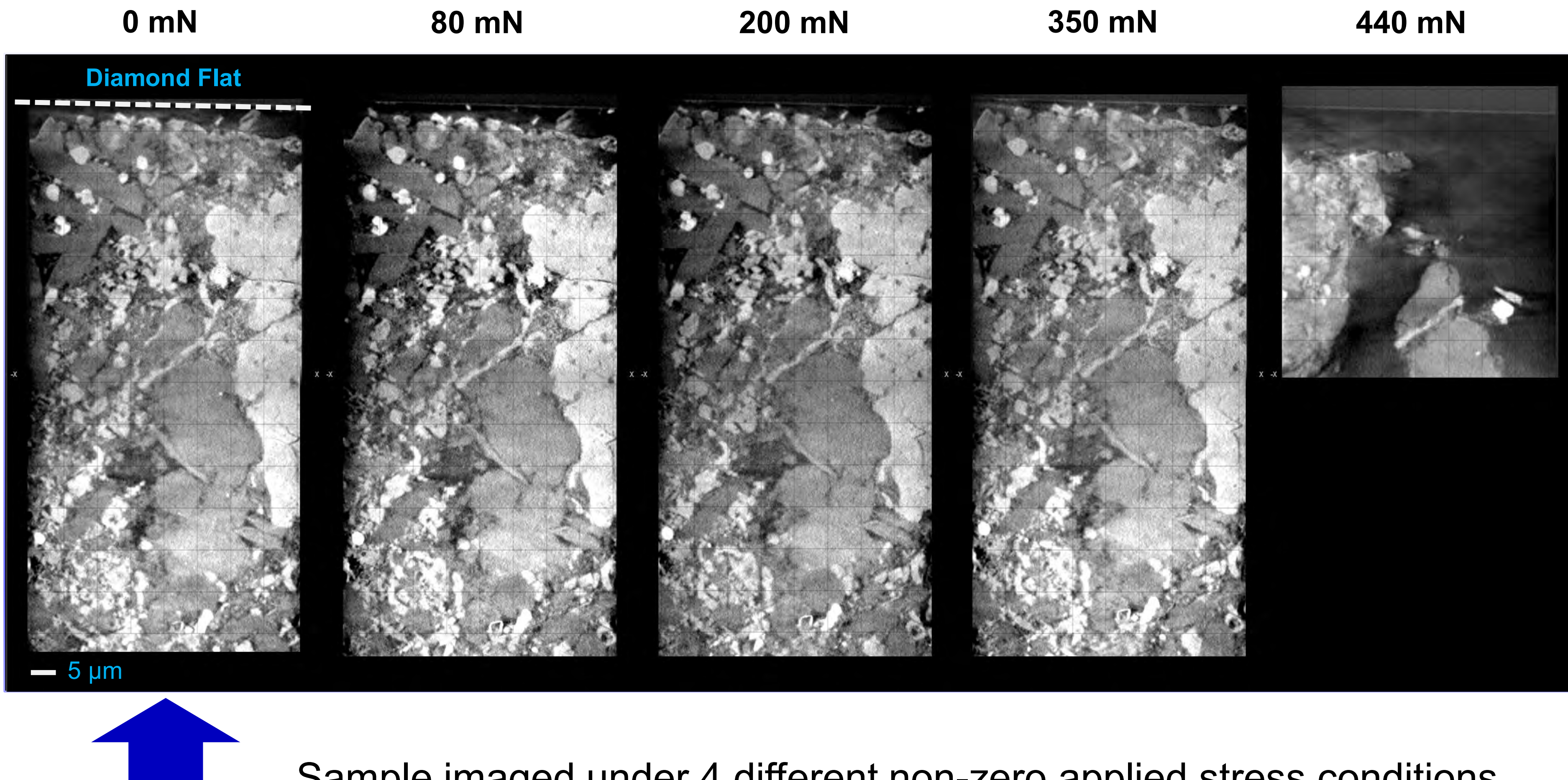
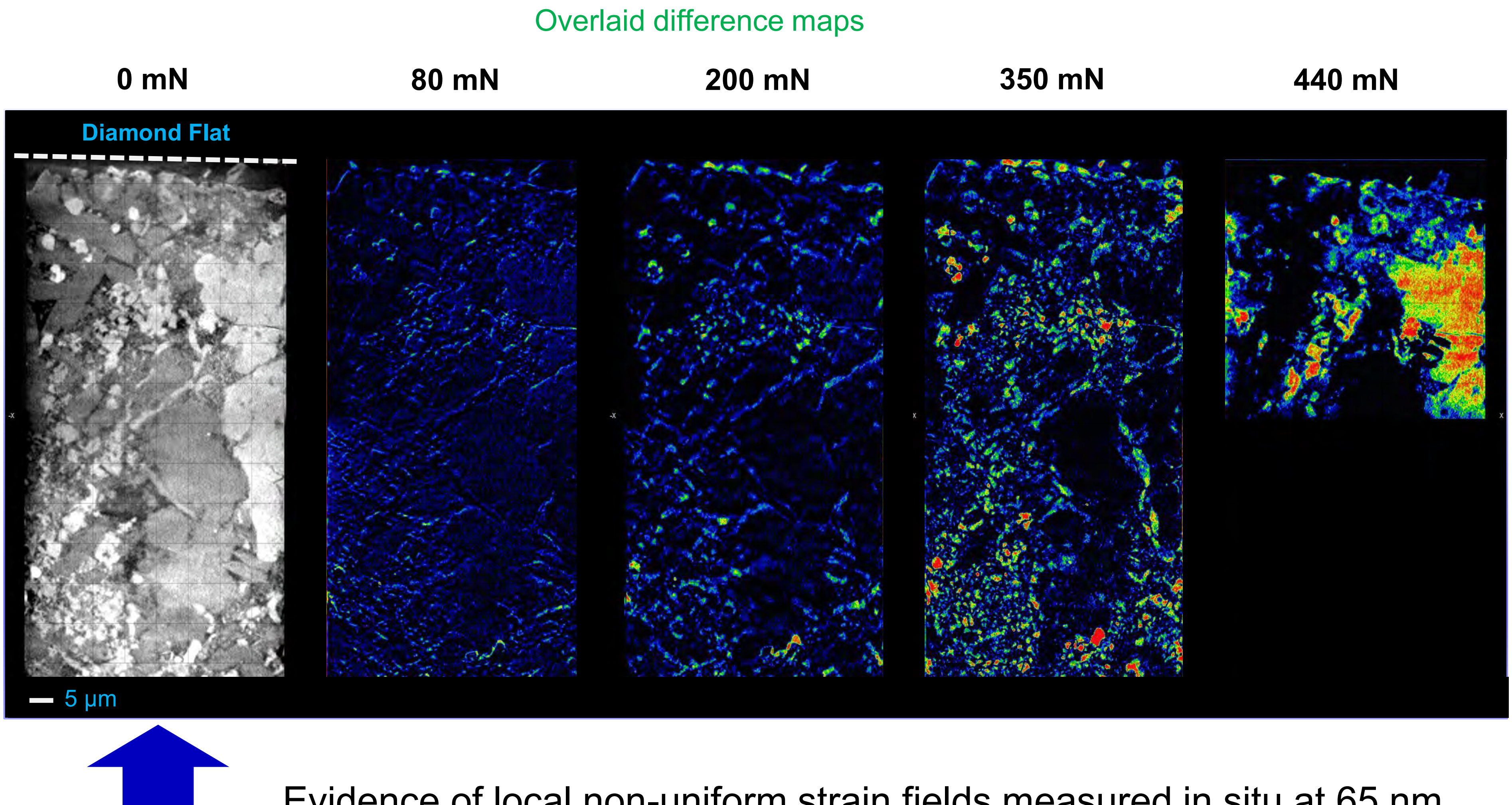
In situ compression experiments on Shale on the Ultra load stage

Overlaid difference maps



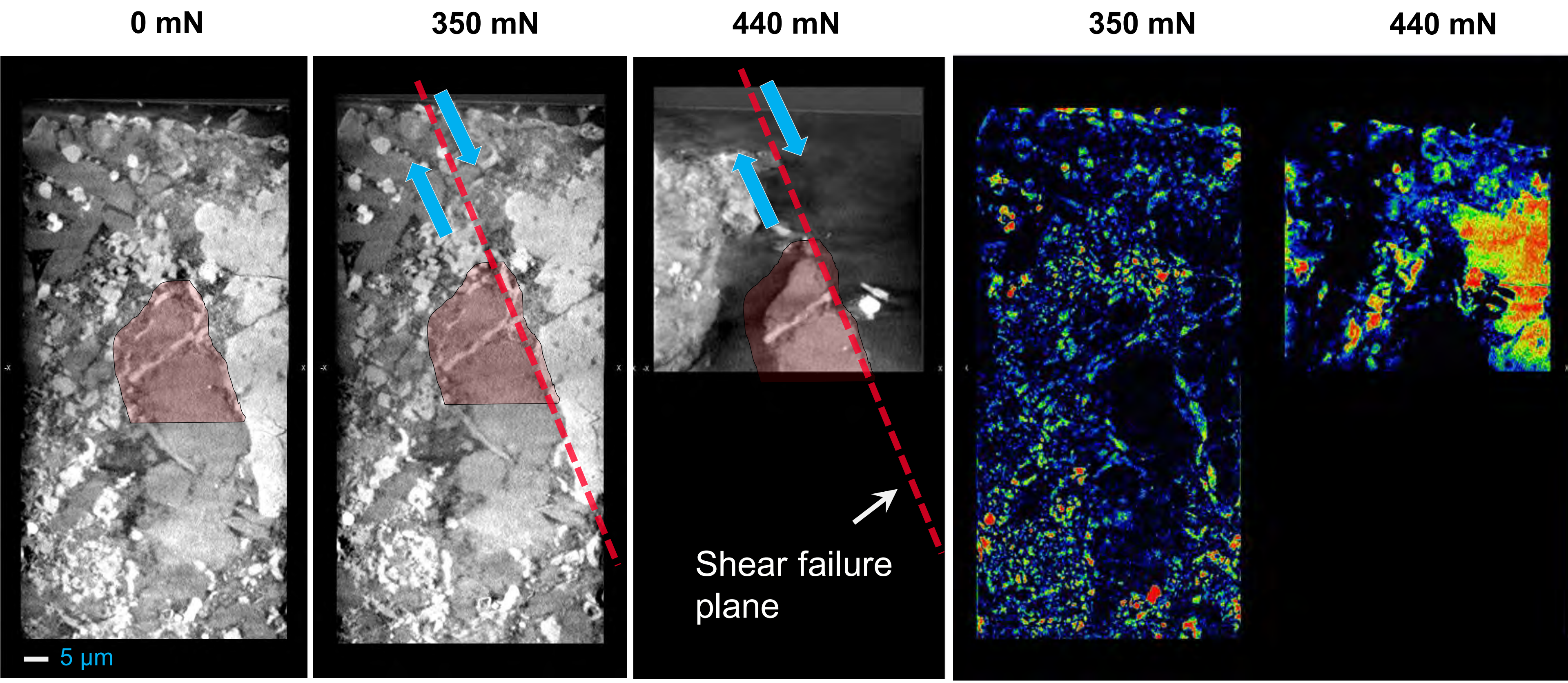
Sample imaged under 4 different non-zero applied stress conditions

In situ compression experiments on Shale on the Ultra load stage



Evidence of local non-uniform strain fields measured in situ at 65 nm voxel size using Ultra nano-CT/XRM

In situ compression experiments on Shale on the Ultra load stage

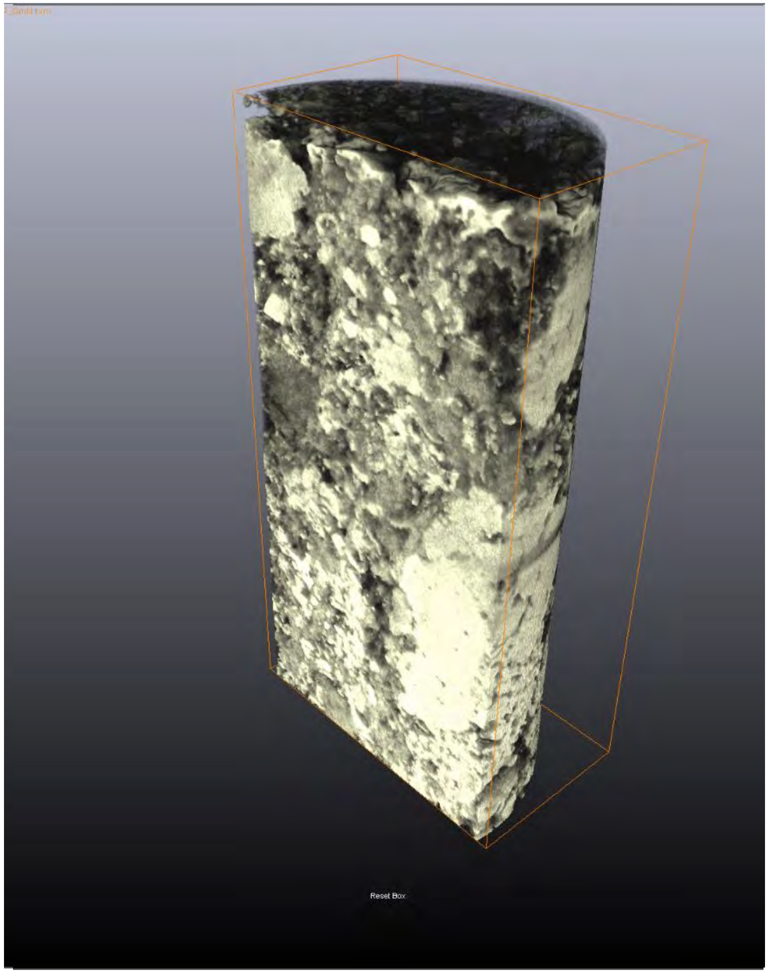


Reconstructed slices

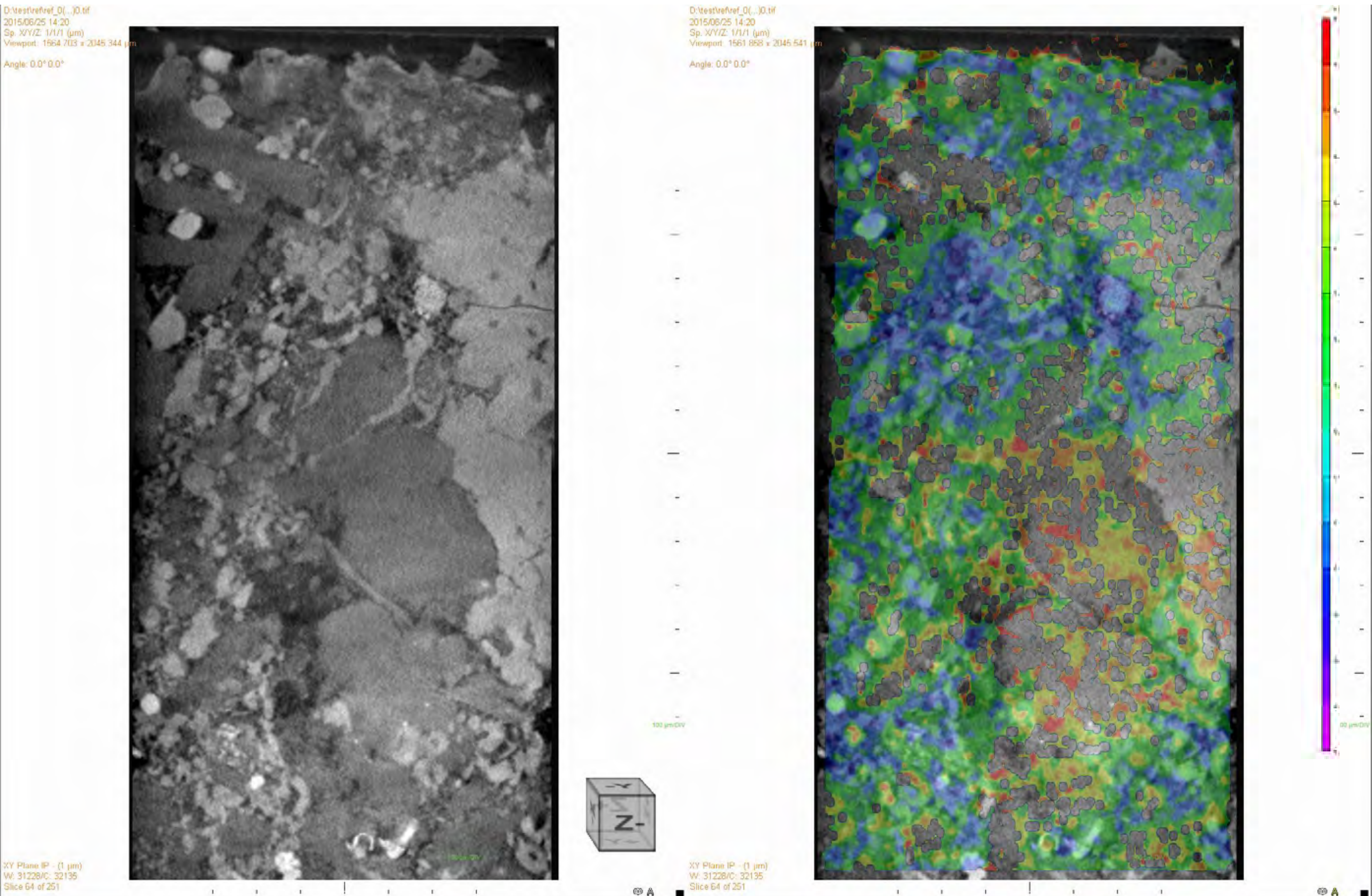
Difference maps

In situ compression experiments on Shale on the Ultra load stage

Volume rendering of reconstructed data at 0 mN and overlaid distance map at 350mN



Initial DVC effort (Digital Volume Correlation)
Strain fields in color



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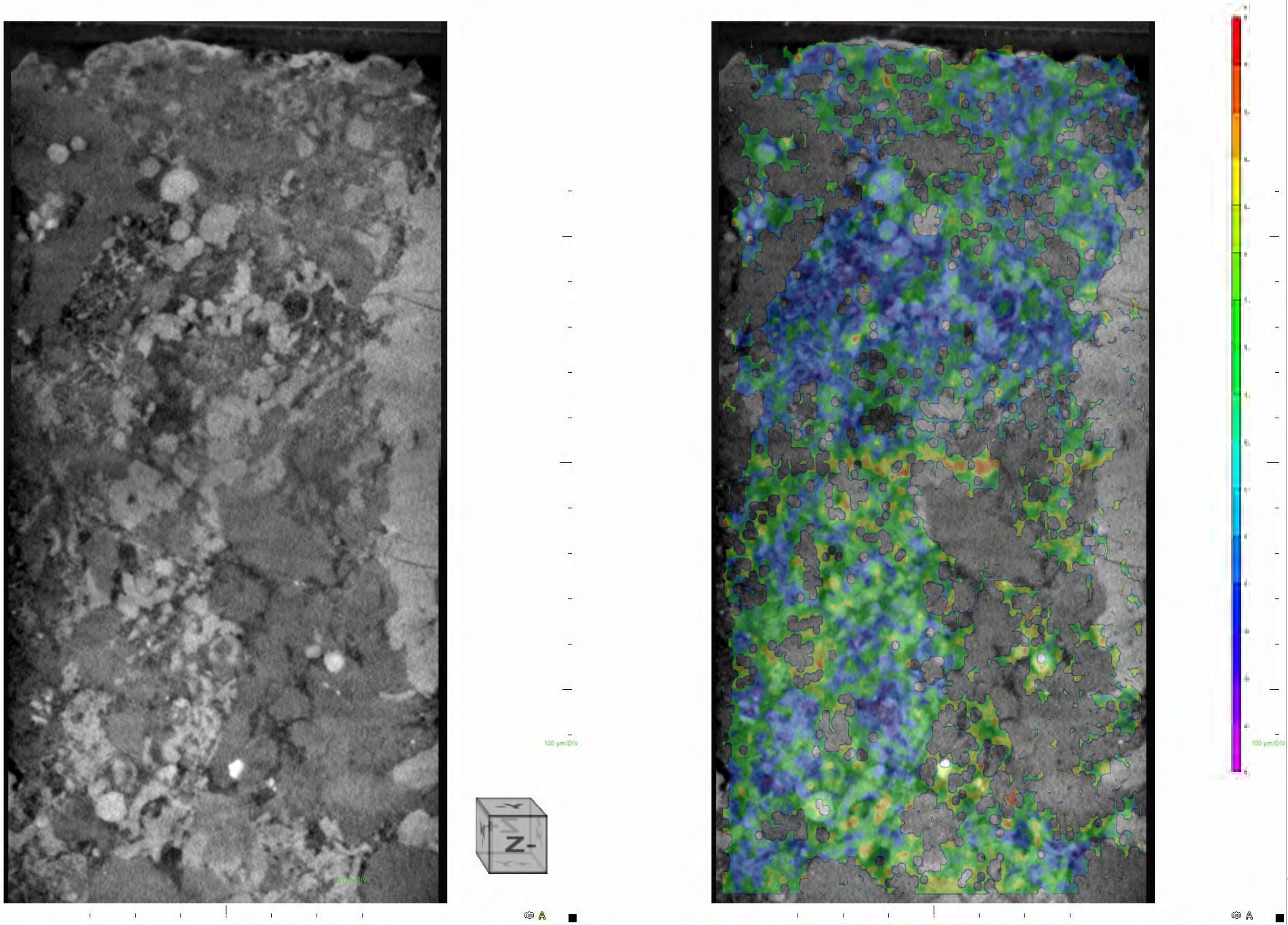

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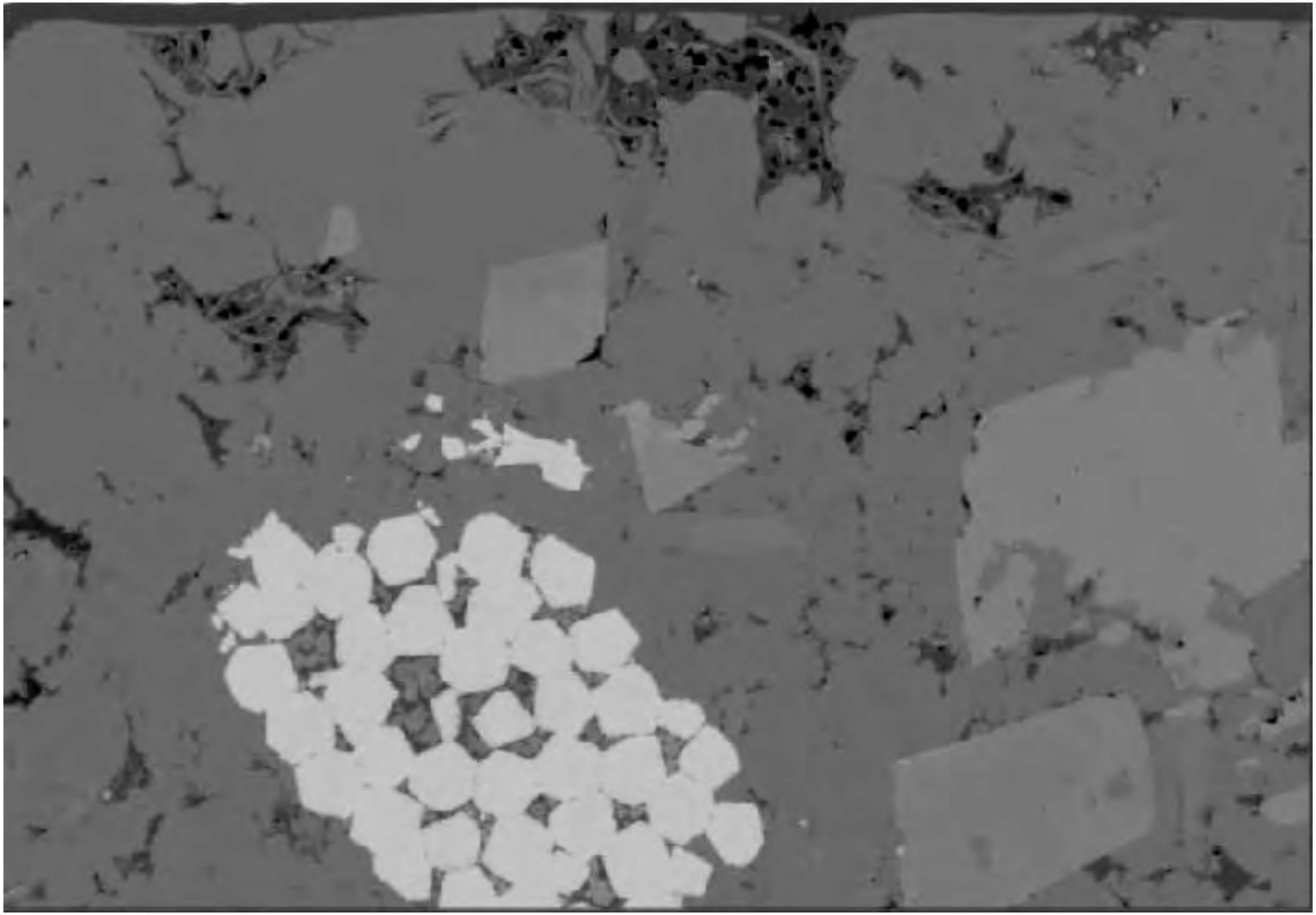
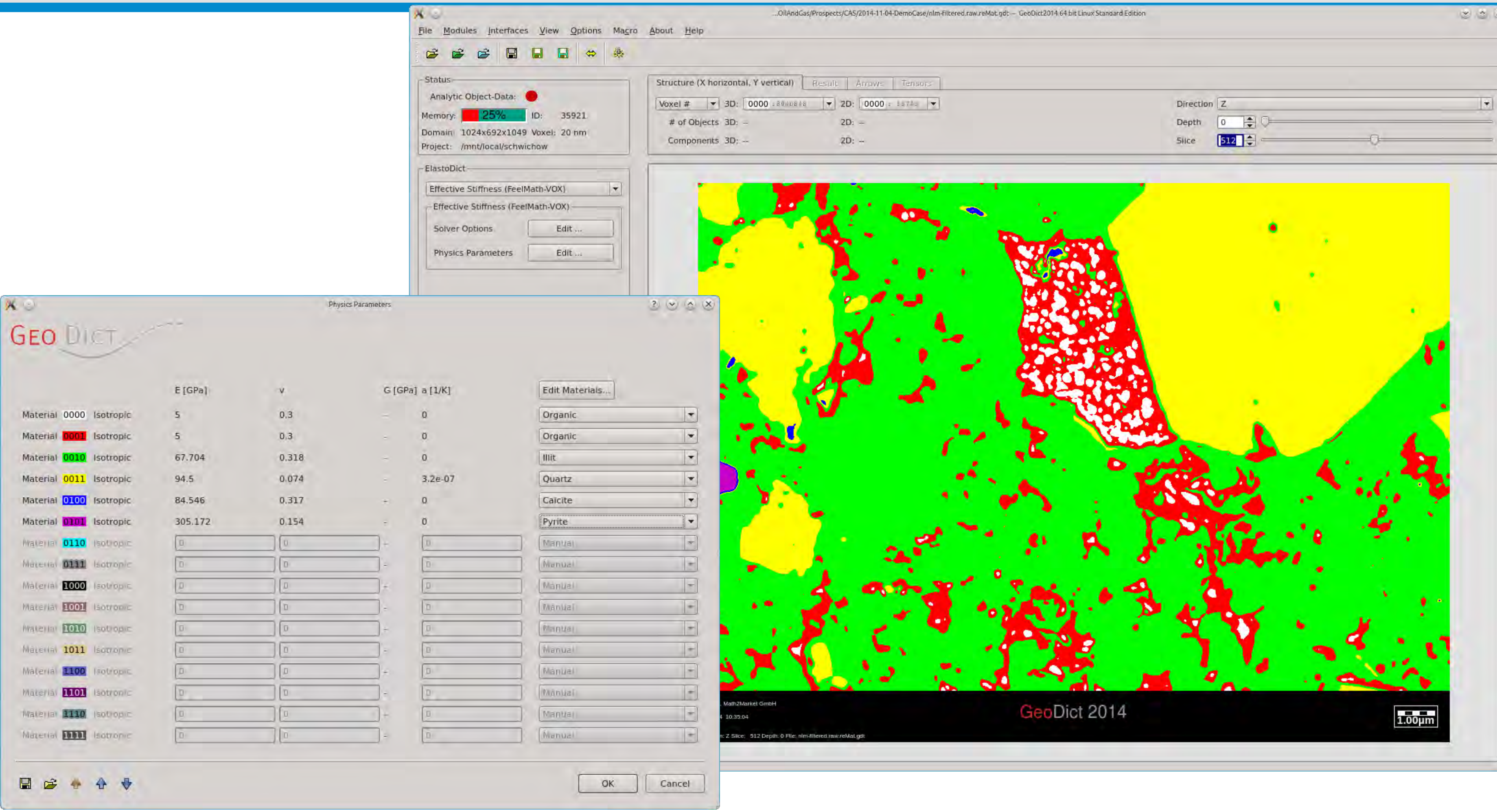
Raw and segmented FIB-SEM data (30x15x15um @10nm) for calculation of linear elastic stress-strain properties by DRP



Effective Mechanical Properties

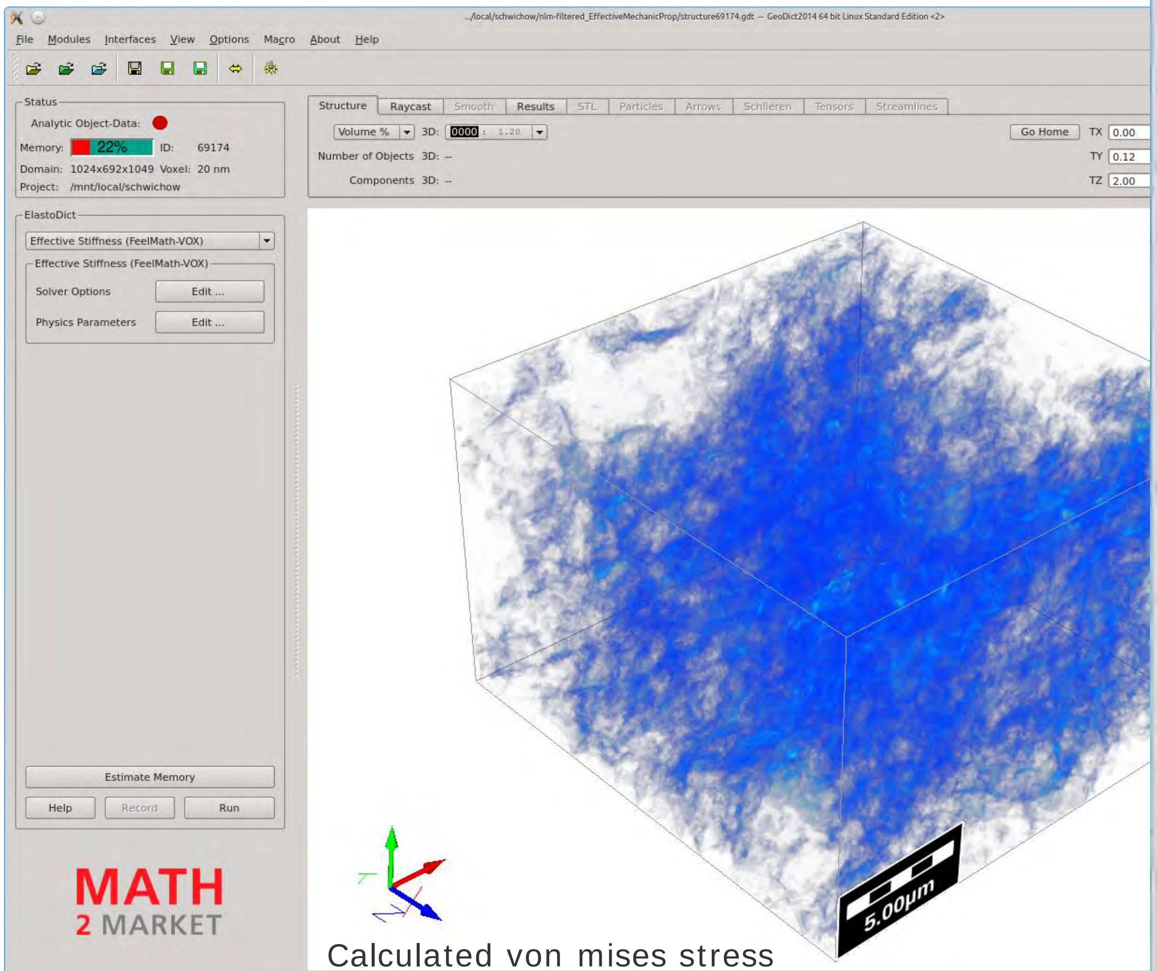
Linear stress-strain caculation



Effective Mechanical Properties

Linear stress-strain caculation

ElastoDict



Calculated von mises stress

Module: ElastoDict (2014 Build 5523)
Command: SolveFeelMathVOX
Wed Nov 5 2014

Domain: 1024 x 692 x 1049 Voxel: 20 nm Load

Input Map | Results Map | Results Info | Visualization

---------------- Engineering Parameters ----------------

(green): error <= 1 %; (yellow): 1 % < error <= 10 %; (red): error > 10 %.

Isotropic Approximation

	Strain Equivalence	Energy Equivalence	Mean Value
Young's Modulus E [GPa]	49.7315	-	-
Poisson Ratio ν	0.2736	-	-
Shear Modulus G [GPa]	19.5243	-	-
Lame Modulus λ [GPa]	23.5916	-	-
Bulk Modulus K [GPa]	36.6078	-	-

-------- Stiffness Formulation for Strain Equivalence --------

Anisotropic Elasticity Tensor [GPa]

unknown	**unknown**	**23.623**	unknown	unknown	unknown
unknown	**unknown**	**23.561**	unknown	unknown	unknown
unknown	**unknown**	**62.64**	unknown	unknown	unknown
unknown	unknown	0.1868	**unknown**	unknown	unknown
unknown	unknown	0.028876	unknown	**unknown**	unknown
unknown	unknown	0.044769	unknown	unknown	**unknown**

-------- Stiffness Formulation for Energy Equivalence --------

Anisotropic Elasticity Tensor [GPa]

unknown	**unknown**	**unknown**	unknown	unknown	unknown
unknown	**unknown**	**unknown**	unknown	unknown	unknown
unknown	**unknown**	**62.638**	unknown	unknown	unknown
unknown	unknown	unknown	**unknown**	unknown	unknown
unknown	unknown	unknown	unknown	**unknown**	unknown
unknown	unknown	unknown	unknown	unknown	**unknown**

Pack ... | Load Input Map | Store As Html | Close

MATH 2 MARKET

ZEISS

Summary / conclusions

- We have successfully measured the strain response at the 65 nm voxel (150 nm resolution) level using a laboratory X-ray Microscope via a series of applied, increasing in situ stresses causing plastic deformation and fracture (at the final load condition)
- Although more thorough data analysis with DVC (Digital Volume Correlation) algorithms is required, it is clear that this method should be able to extract the actual experimental strain fields of a shale at the 150 nm resolution level, which is sufficient resolution to distinguish the grain structure in detail for many shales
- It should be possible to understand the nano-scale root cause of the stress-strain curve based on physical phenomenon including, for example, grain boundary slip, mineral compression, microfractures, etc…
- With the nano-indentation mode of this in situ cell, it may be feasible to understand the smallest microfractures and their patterns induced by hydraulic fracturing
- Using DRP modelling software, it may be feasible to compare simulation and experimental stress-strain data to calibrate simulation models and to get a better holistic understanding of the shale properties by using both DRP and in situ experiments combined