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

3D He Ion → 3D FIB-SEM → 3D nano-CT → Mineralogy / 2D SEM → 3D u-CT

1”-1 ½” Core Plug or Rock Cutting
Thin section
Slabbed core
whole core

3D X-ray Versa
3D He Ion
3D nano-CT
3D FIB-SEM
3D X-ray Ultra
2D EM
2D imaging

0.5um Sandstone
50nm Carbonate
1-2nm Shale / mudrock

Mineralogy / 2D SEM
3D u-CT

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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

<table>
<thead>
<tr>
<th>Mode</th>
<th>Mag</th>
<th>2D Res</th>
<th>Voxel</th>
<th>Field of View</th>
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<tbody>
<tr>
<td>Large Field of View</td>
<td>200X</td>
<td>150 nm</td>
<td>64 nm</td>
<td>65 μm x 65 μm</td>
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<tr>
<td>High Resolution</td>
<td>800X</td>
<td>50 nm</td>
<td>16 nm</td>
<td>16 μm x 16 μm</td>
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</tbody>
</table>

X-ray source
Condenser lens
Sample
Objective lens (Zone Plate)
Phase ring
X-ray camera

Carl Zeiss X-ray Microscopy
Ultra XRM Load Stage
Enables unique 3D imaging of nanostructure

Compression

Tension

Indentation

Top anvil

X-ray beam path

Sample

Bottom anvil

Sample rotation for tomography

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

Cracks
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

Courtesy Robert Bradley & Xuekun Lu, Univ. of Manchester
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• Prospects for DRP comparison
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
Purple = Quartz
Green = Pores/Organics
Red = Pyrites
In situ compression experiments on Shale on the Ultra load stage

Overlaid difference maps

0 mN  80 mN  200 mN  350 mN  440 mN

Sample imaged under 4 different non-zero applied stress conditions
In situ compression experiments on Shale on the Ultra load stage

Overlaid difference maps

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

0 mN

350 mN

440 mN

5 μm

Shear failure plane
**In situ** compression experiments on Shale on the Ultra load stage

Volume rendering of reconstructed data at 0 mN and overlaid distance map at 350 mN
Initial DVC effort (Digital Volume Correlation)
Strain fields in color
Initial DVC effort (Digital Volume Correlation)
Strain fields in color
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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 calculation
Effective Mechanical Properties
Linear stress-strain calculation

Calculated von mises stress
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.