ILL–ESS User Meeting 2022

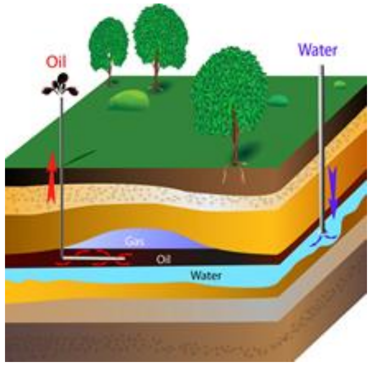
Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography

Fernando Vieira Lima[a], Stephen Hall[a], Jonas Engqvist[a], Erika Tudisco[a],
Robin Worcek[b], Alessandro Tengattini[c], Cyrille Couture[c]

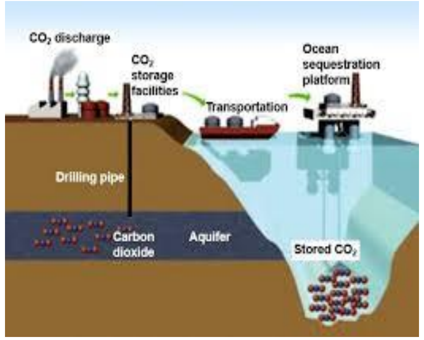
[a] Lund University
[b] European Spallation Source (ESS)
[C] Institute Laue-Langevin (ILL) and Université Grenoble Alpes

1. INTRODUCTION

Localized Deformations → Effects on Permeability of Rocks



Oil Extraction



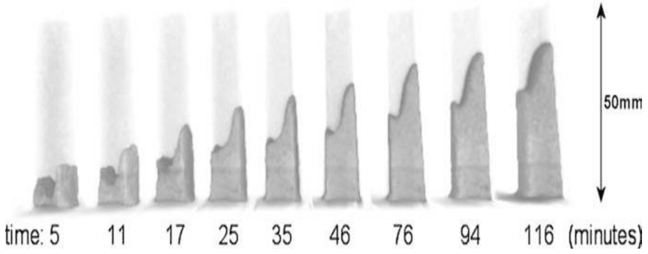
CO2 Storage

Laboratory

Traditional Tests
(bulk measurements)

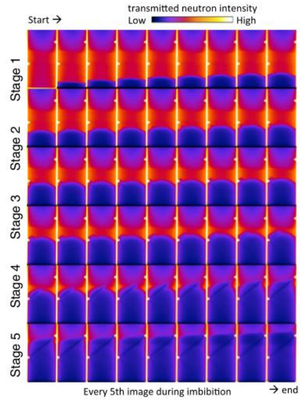
Advanced Tests
(full-field measurements)

Tracking the fluid movement in unsaturated rock using 3D neutron tomography



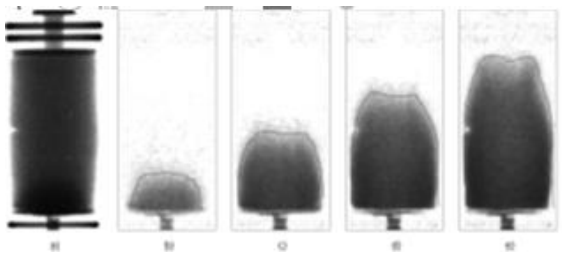
Masschaele *et al.* (2004)

Radiographies in Deformed Samples



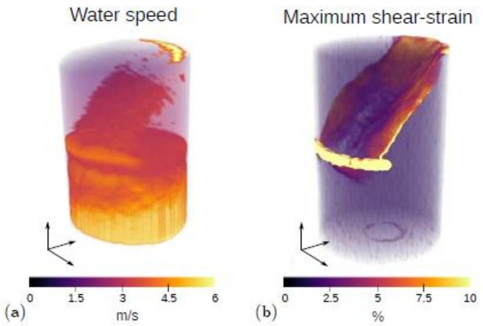
Hall (2013)

In-situ Deformation + Flow Radiographies



Tudisco et al. (2017)

High-speed neutron tomography



Tudisco et al. (2019)

1. INTRODUCTION

X-RAY AND NEUTRON IMAGING

Woracek (2021)

Attenuation coefficients with X-ray [$cm^{?1}$]

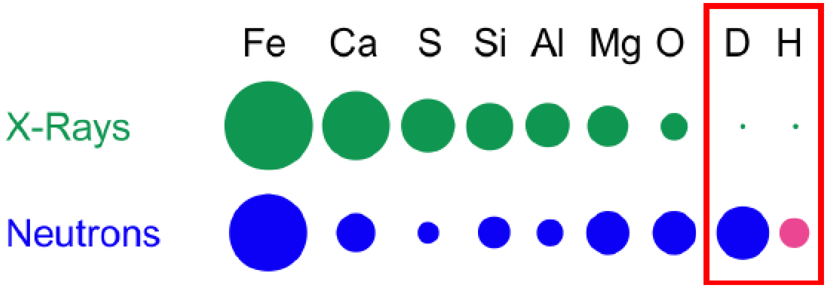
For X-rays = proportional increase!

1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 0.02																	He 0.02
Li 0.06	Be 0.22											B 0.28	C 0.27	N 0.11	O 0.16	F 0.14	Ne 0.17
Na 0.13	Mg 0.24											Al 0.38	Si 0.33	P 0.25	S 0.30	Cl 0.23	Ar 0.20
K 0.14	Ca 0.26	Sc 0.48	Ti 0.73	V 1.04	Cr 1.29	Mn 1.32	Fe 1.57	Co 1.78	Ni 1.96	Cu 1.97	Zn 1.64	Ga 1.42	Ge 1.33	As 1.50	Se 1.23	Br 0.90	Kr 0.73
Rb 0.47	Sr 0.86	Y 1.61	Zr 2.47	Nb 3.43	Mo 4.29	Tc 5.06	Ru 5.71	Rh 6.08	Pd 6.13	Ag 5.67	Cd 4.84	In 4.31	Sn 3.98	Sb 4.28	Te 4.06	I 3.45	Xe 2.53
Cs 1.42	Ba 2.73	La 5.04	Hf 19.70	Ta 25.47	W 30.49	Re 34.47	Os 37.92	Ir 39.01	Pt 38.61	Au 35.94	Hg 25.88	Tl 23.23	Pb 22.81	Bi 20.28	Po 20.22	At	Rn 9.77
Fr	Ra 11.80	Ac 24.47	Rf	Ha													
*Lanthanides	Ce 5.79	Pr 6.23	Nd 6.46	Pm 7.33	Sm 7.68	Eu 5.66	Gd 8.69	Tb 9.46	Dy 10.17	Ho 10.91	Er 11.70	Tm 12.49	Yb 9.32	Lu 14.07			
**Actinides	Th 28.95	Pa 39.65	U 49.08	Np	Pu	Am	Cm	Bk	Vf	Es	Fm	Md	No	Lr x-ray			

Attenuation coefficients with neutrons [$cm^{?1}$]

For neutrons = completely unsystematic!

1a	2a	3b	4b	5b	6b	7b	8			1b	2b	3a	4a	5a	6a	7a	0
H 3.44																	He 0.02
Li 3.30	Be 0.79											B 101.60	C 0.56	N 0.43	O 0.17	F 0.20	Ne 0.10
Na 0.09	Mg 0.15											Al 0.10	Si 0.11	P 0.12	S 0.06	Cl 1.33	Ar 0.03
K 0.06	Ca 0.08	Sc 2.00	Ti 0.60	V 0.72	Cr 0.54	Mn 1.21	Fe 1.19	Co 3.92	Ni 2.05	Cu 1.07	Zn 0.35	Ga 0.49	Ge 0.47	As 0.67	Se 0.73	Br 0.24	Kr 0.61
Rb 0.08	Sr 0.14	Y 0.27	Zr 0.29	Nb 0.40	Mo 0.52	Tc 1.76	Ru 0.58	Rh 10.88	Pd 0.78	Ag 4.04	Cd 115.11	In 7.58	Sn 0.21	Sb 0.30	Te 0.25	I 0.23	Xe 0.43
Cs 0.29	Ba 0.07	La 0.52	Hf 4.99	Ta 1.49	W 1.47	Re 6.85	Os 2.24	Ir 30.46	Pt 1.46	Au 6.23	Hg 16.21	Tl 0.47	Pb 0.38	Bi 0.27	Po	At	Rn
Fr	Ra 0.34	Ac	Rf	Ha													
*Lanthanides	Ce 0.14	Pr 0.41	Nd 1.87	Pm 5.72	Sm 171.47	Eu 94.58	Gd 1479.04	Tb 0.93	Dy 32.42	Ho 2.25	Er 5.48	Tm 3.53	Yb 1.40	Lu 2.75			
**Actinides	Th 0.59	Pa 8.46	U 0.82	Np 9.80	Pu 50.20	Am 2.86	Cm	Bk	Cf	Es	Fm	Md	No	Lr neut.			



...even for different isotopes of the same element!

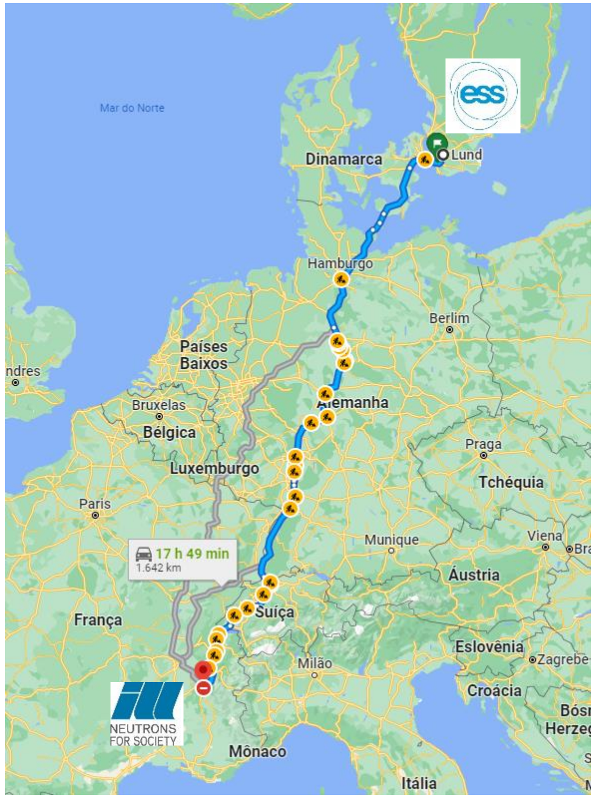
Element/ Isotope	Coh. Scattering (barn)	Inch. Scattering (barn)	Absorption (barn)
H	1.757	80.26	0.555
D	5.592	2.050	0.001

This contrast makes possible fluid front tracking with close to a ”single phase” condition

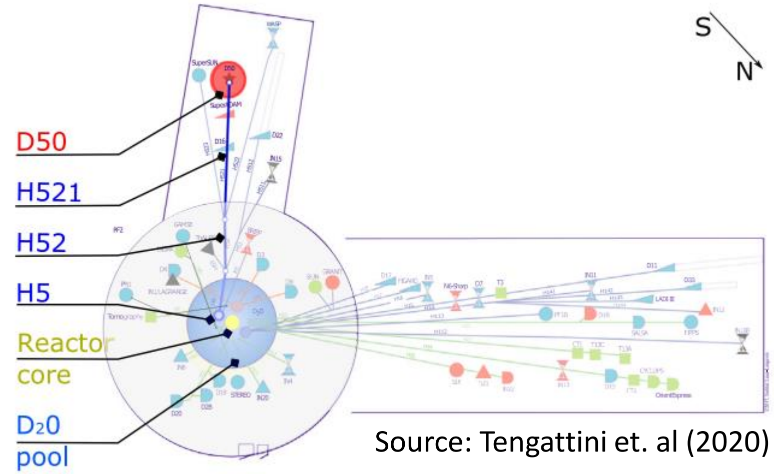
Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

1. INTRODUCTION

Neutron EuroTrip (June 2021)

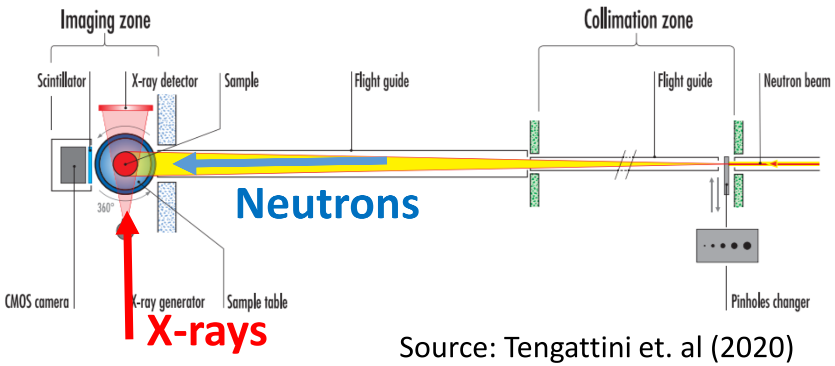


NEUTRONS FOR SOCIETY



Source: Tengattini et. al (2020)

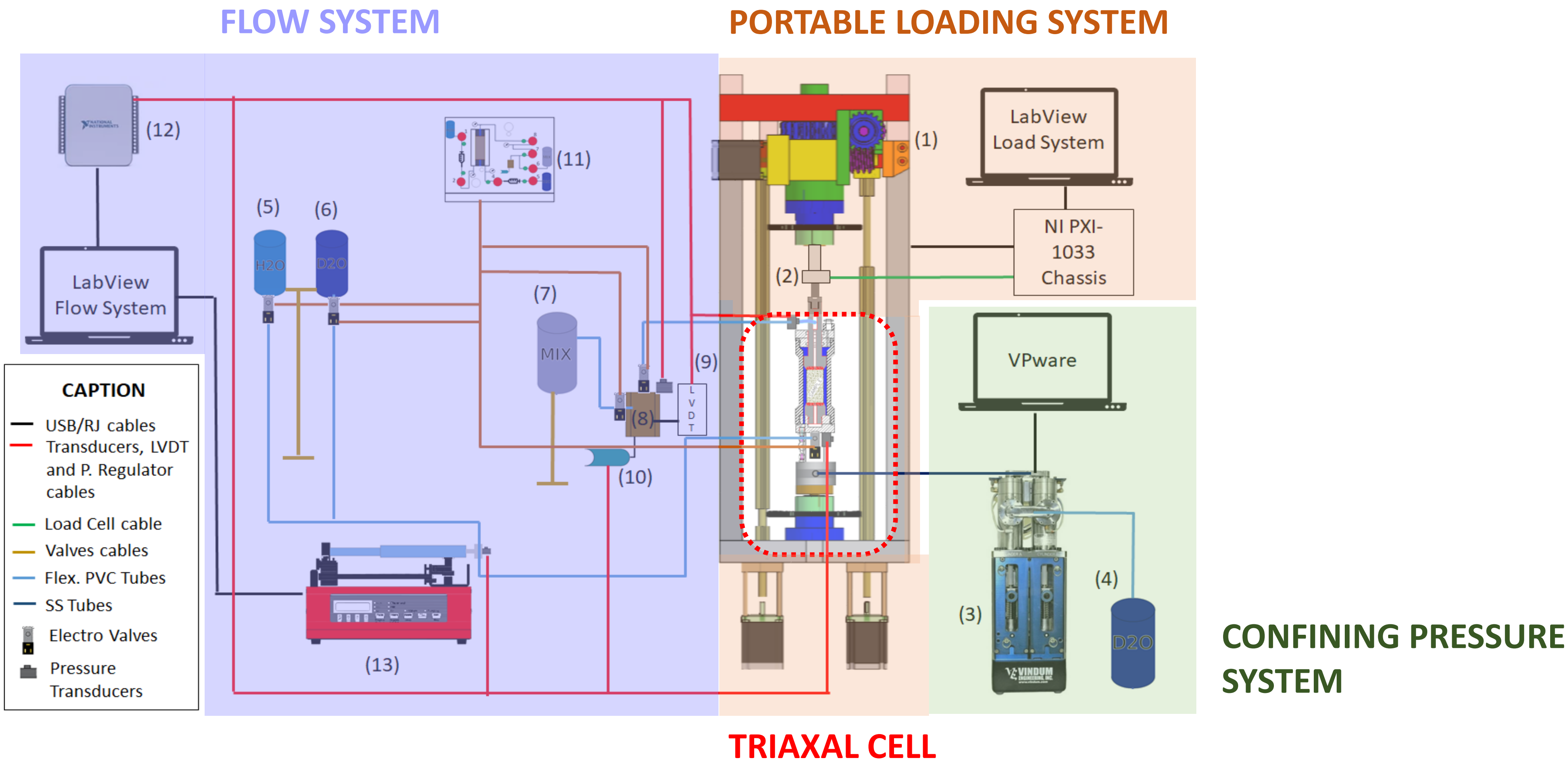
NeXT



Source: Tengattini et. al (2020)

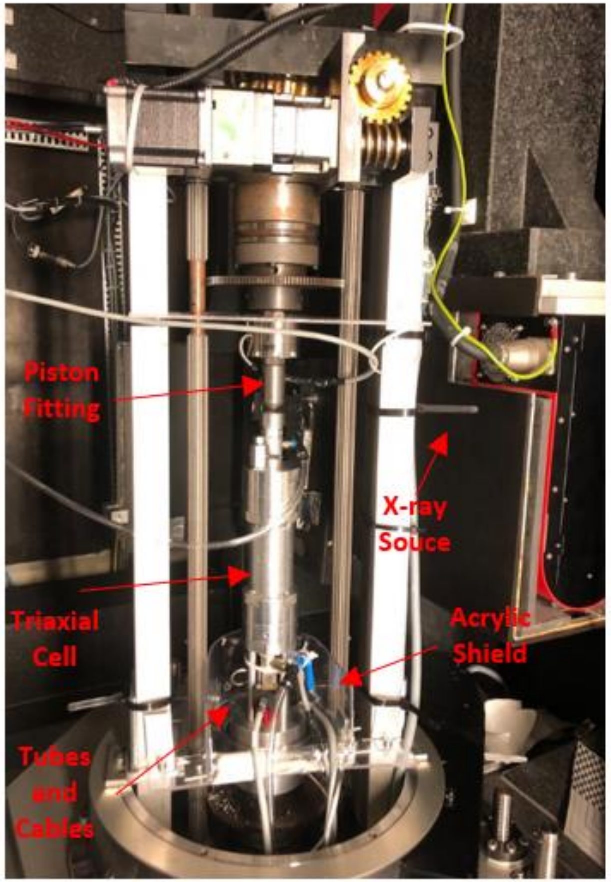
1. INTRODUCTION

EXPERIMENTAL SETUP

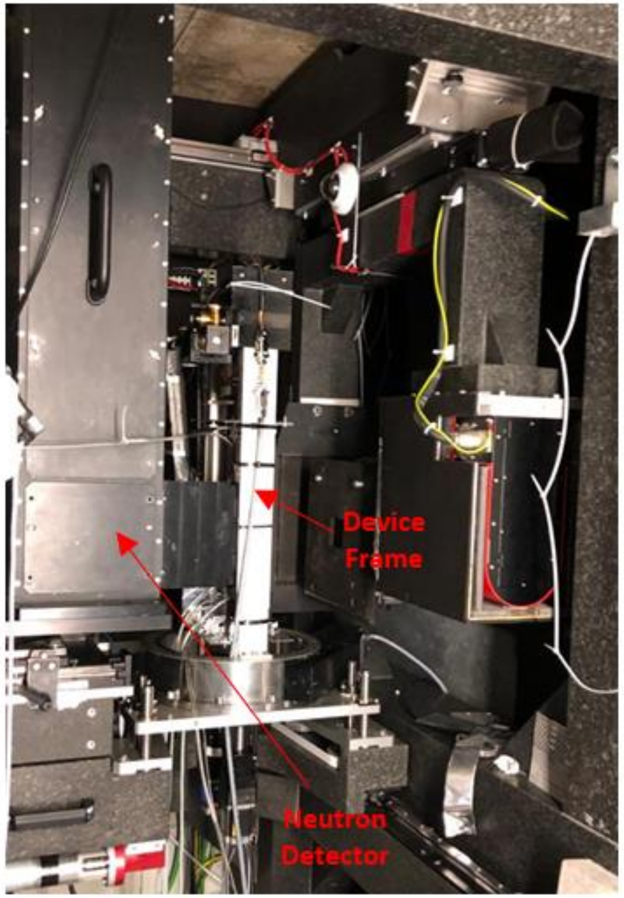


1. INTRODUCTION

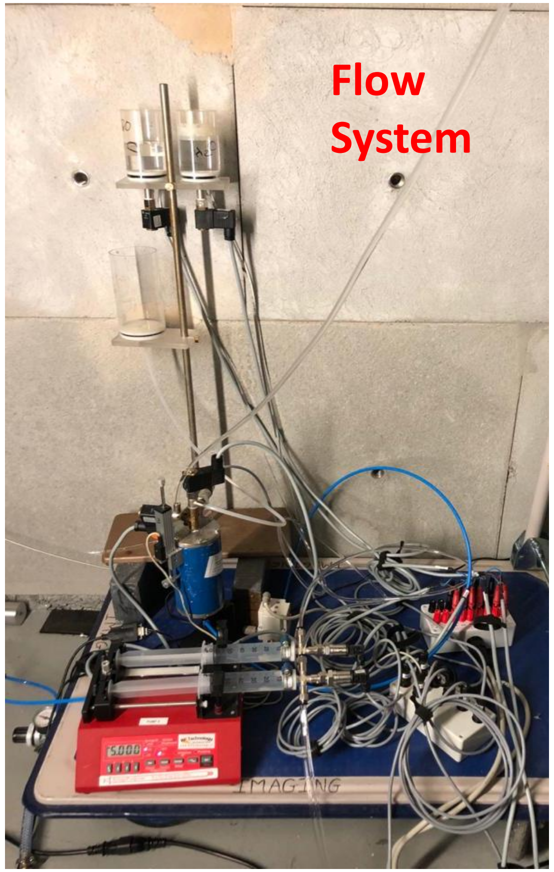
EXPERIMENTAL SETUP

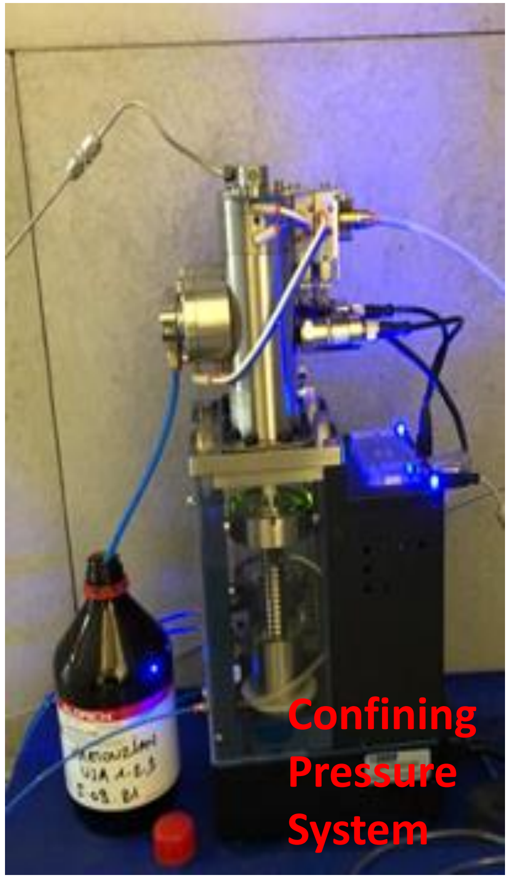


X-RAY SCAN POSITION



NEUTRON SCAN POSITION

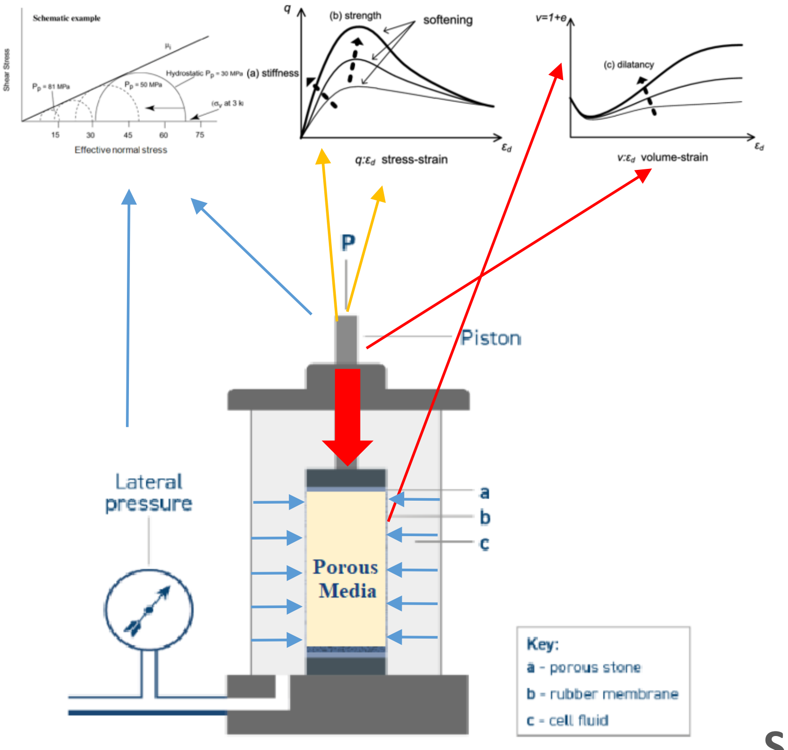




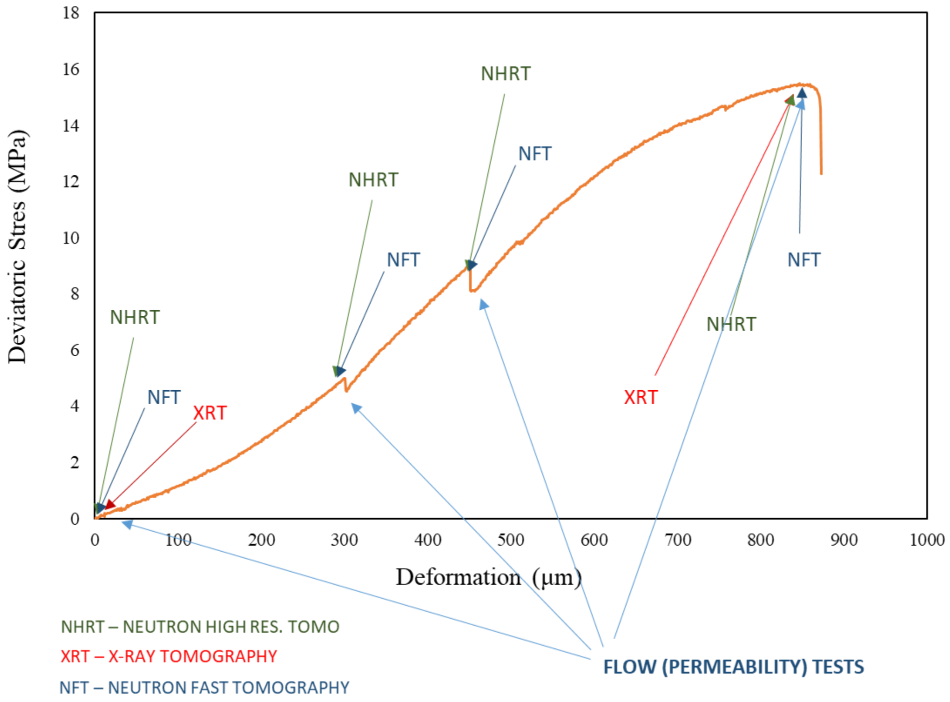
Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

2. METHODOLOGY

EXPERIMENT OUTLINE



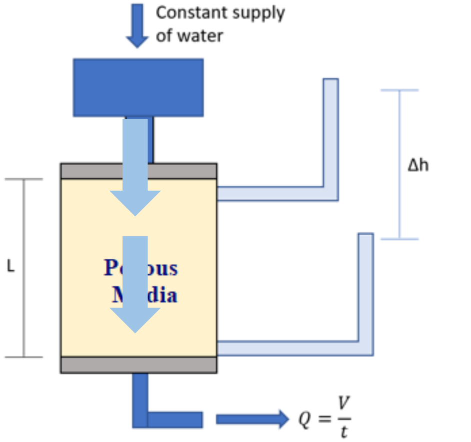
Conventional Triaxial Test Scheme



H_2O into the D_2O saturated sample

Flow rate = **0.07 ml/min**

Maximum displacement of **1 pixel per scan**



Conventional Permeability Test Scheme

SAMPLE: IDAHO GRAY SANDSTONE (d = 25 mm and h = 50 mm)

Confining Pressure = **1 MPa**

Axial compression in **3 steps (Sample 1) and 2 Steps (Sample 2)**

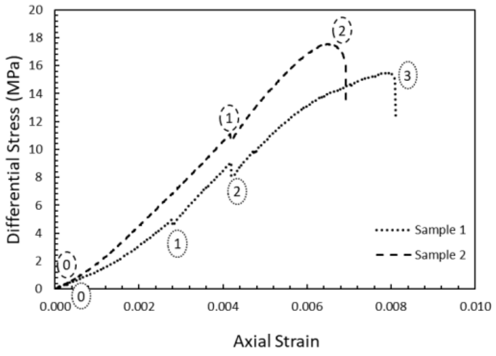
Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

1. INTRODUCTION

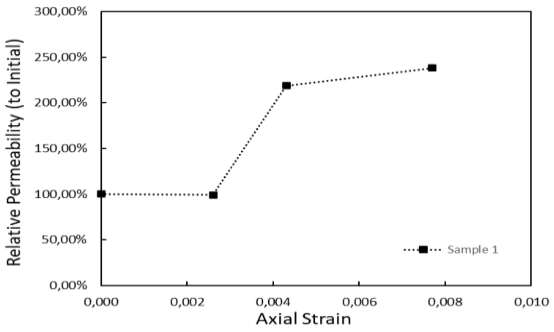
What can we get from the in-situ experiments?

FROM BULK MEASUREMENTS (TRADITIONAL)

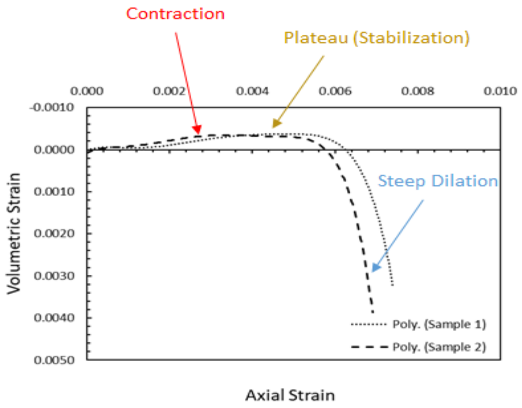
Stress-Strain Curves



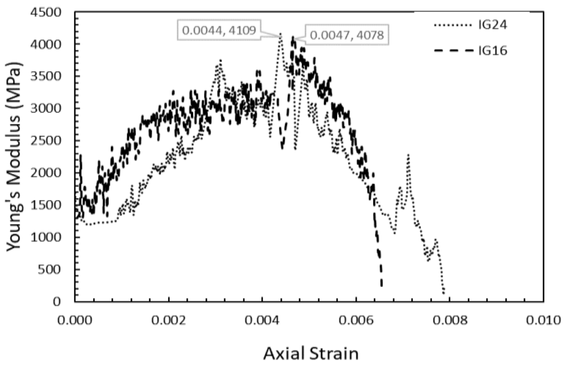
Permeability-Axial Strain Curves



Volumetric-Axial Strain Curves



Young's Module-Axial Strain Curves

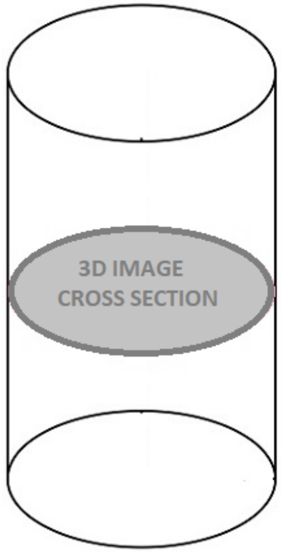


Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

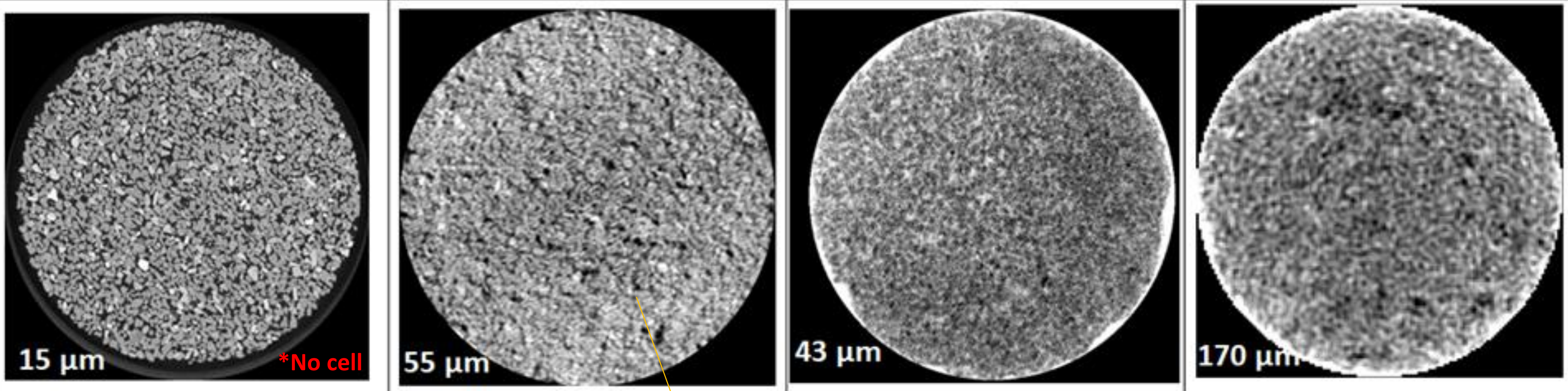
1. INTRODUCTION

What can we get from the in-situ experiments?

FROM FULL-FIELD MEASUREMENTS (ADVANCED)



X-ray Tomography of the sample (at 4D Image Lab, Lund)	In-situ X-ray Tomography	In-situ High-Resolution Neutron Tomography	In-situ Fast Neutron Tomography (H2O into D2O)
30 minutes scan	28 minutes scan	30 minutes scan	1 minute scan



Instability in the device's rotation table + conic beam

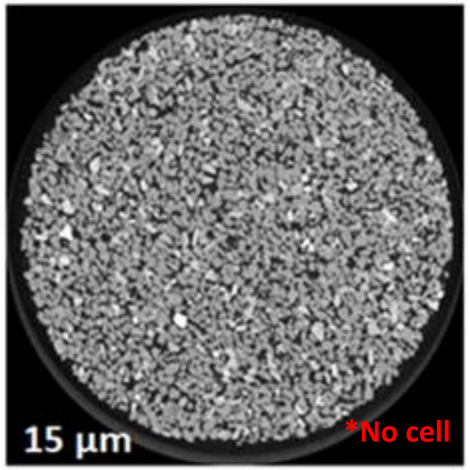
*not the same slices

Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

What can we get from the in-situ experiments?

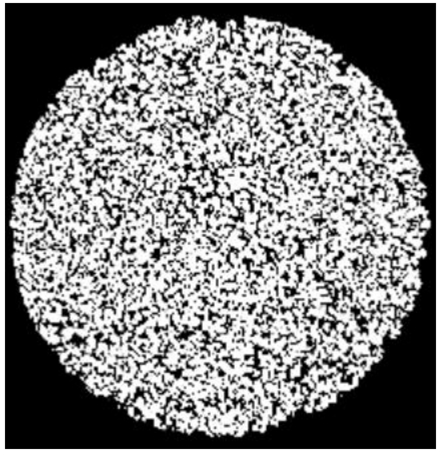
FROM FULL-FIELD MEASUREMENTS (ADVANCED)

X-ray Tomography of the sample (at 4D Image Lab, Lund)

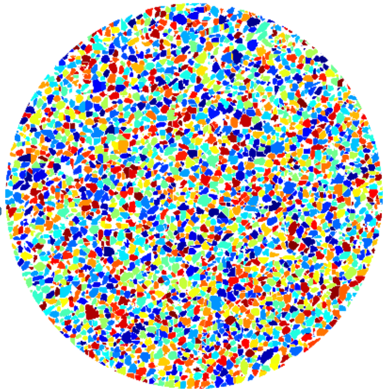


30 minutes scan

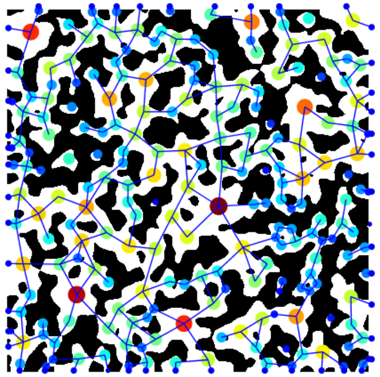
Binarized Images

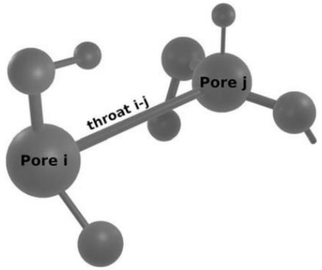


Grain Segmentation

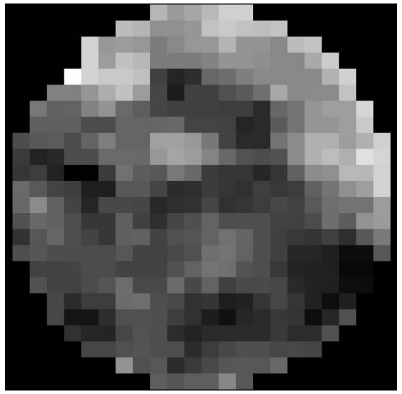


Pore Network Models





Porosity Maps (3D)



Voxel Size (15µm) ⟷ 20x ⟷ Grain Size (300µm)

Grain Resolution

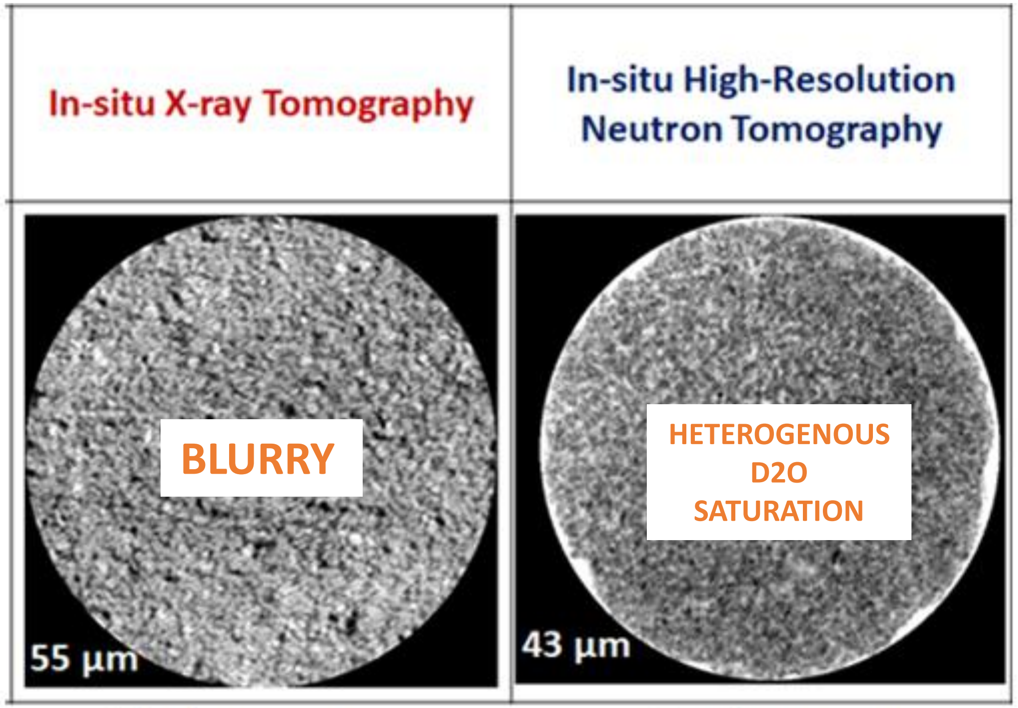
Source: Boccardo et. al (2020)

Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

1. INTRODUCTION

What can we get from the in-situ experiments?

FROM FULL-FIELD MEASUREMENTS (ADVANCED)

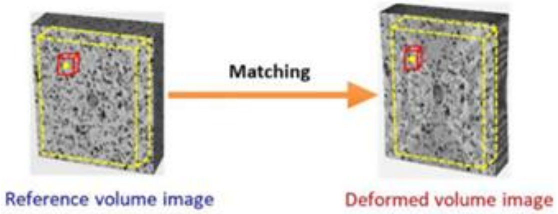


28 minutes scan | 30 minutes scan

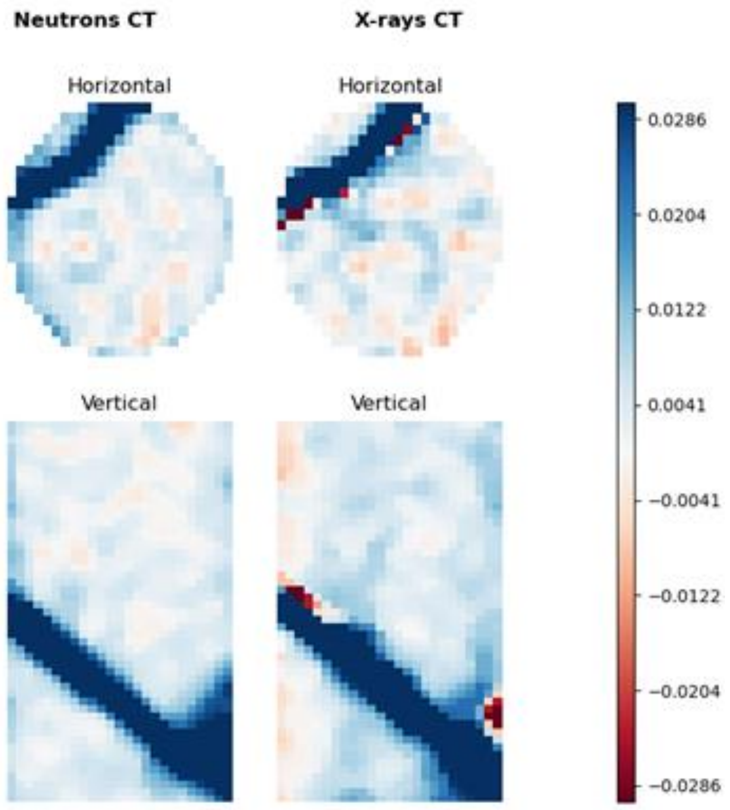
Voxel Size (~50µm) ⟷ 6x ⟷ Grain Size (300µm)

"Texture" Resolution

DIGITAL VOLUME CORRELATION



Accumulated Volumetric Strain Fields

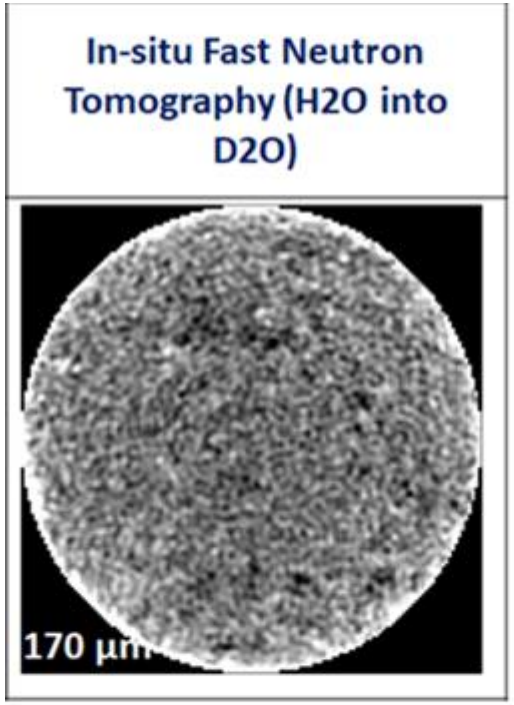


Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

1. INTRODUCTION

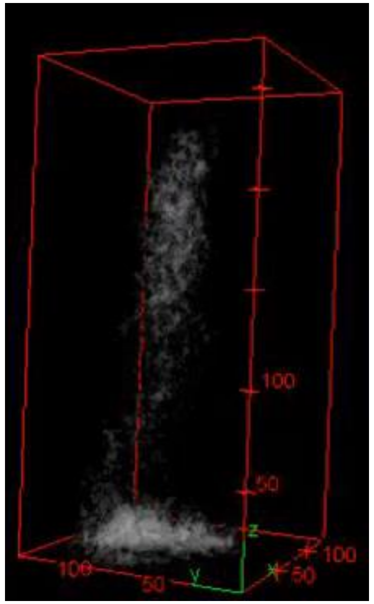
What can we get from the in-situ experiments?

FROM FULL-FIELD MEASUREMENTS (ADVANCED)

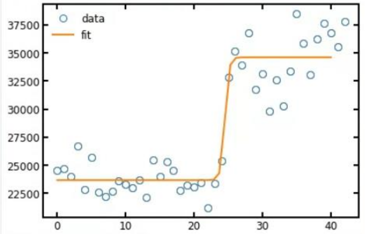


1 minute scan

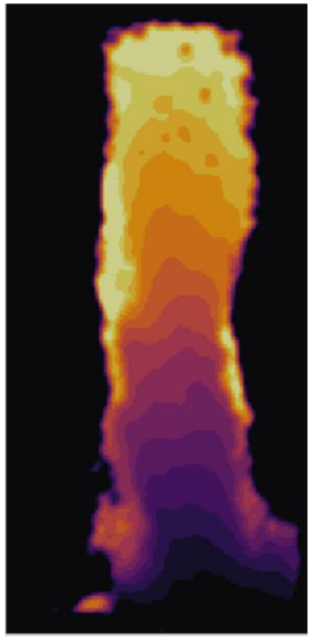
Time series of tomograms (3D)



Front Advance Field (3D)

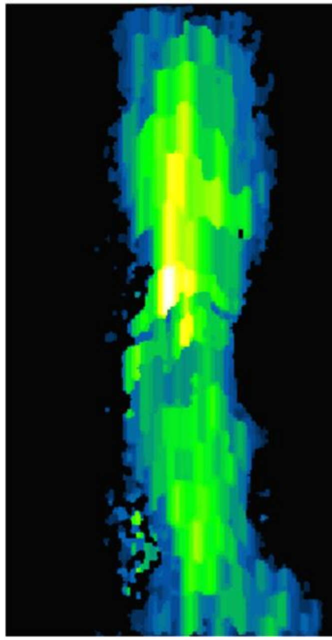


Voxel saturation time (fitting sigmoid curve)



Flow Speed Field (3D)

From:
- Front advance binarization
- Distance maps (Euclidian Distance) / Acquisition Time
- Filtering



Voxel Size (170µm) ⇔ 2x ⇔ Grain Size (300µm)

Only Fluid flow tracking

Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

2. RESEARCH QUESTIONS

Q1 - HOW CAN SAMPLE HETEROGENEITY AFFECT THE FLUID FLOW (UNDEFORMED STATE)?

Q2 - HOW CAN SAMPLE HETEROGENEITY INFLUENCE THE DISTRIBUTION OF DEFORMATION THROUGHOUT DEVIATORIC COMPRESSION?

Q3 - HOW DOES FLUID FLOW EVOLVE ALONG DEVIATORIC COMPRESSION?

Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

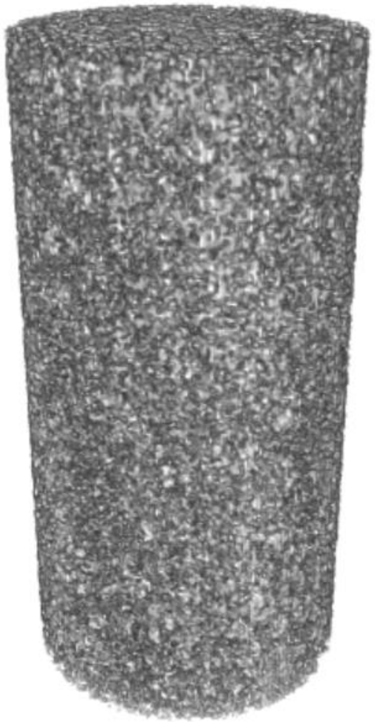
3. RESULTS

Q1 - HETEROGENEITY VS FLUID FLOW

PORE NETWORK MODEL (PNM) SIMULATION

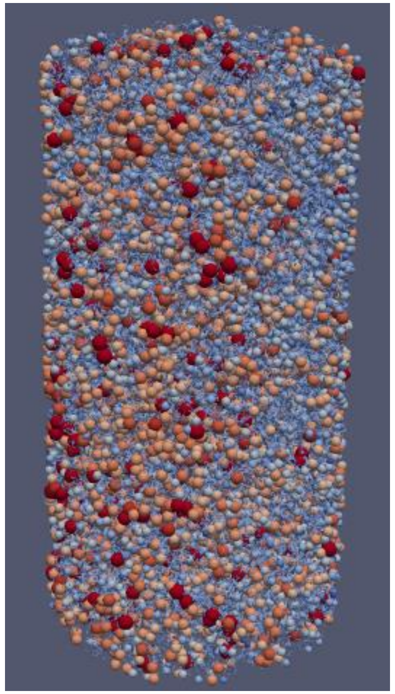
Sample 1

Binarized image from X-ray tomo



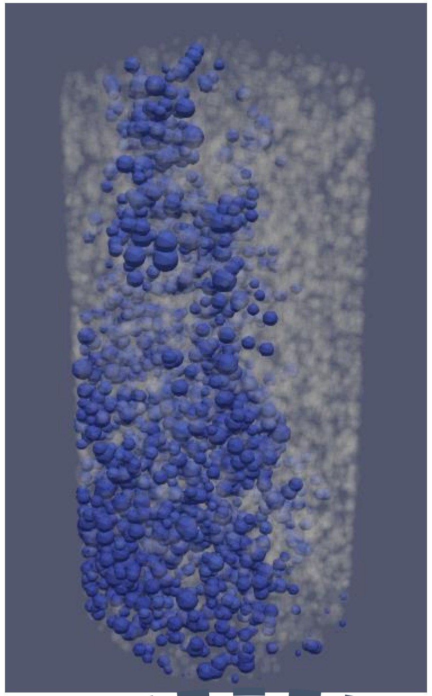
Gostick, J et al., (2016)

PNM

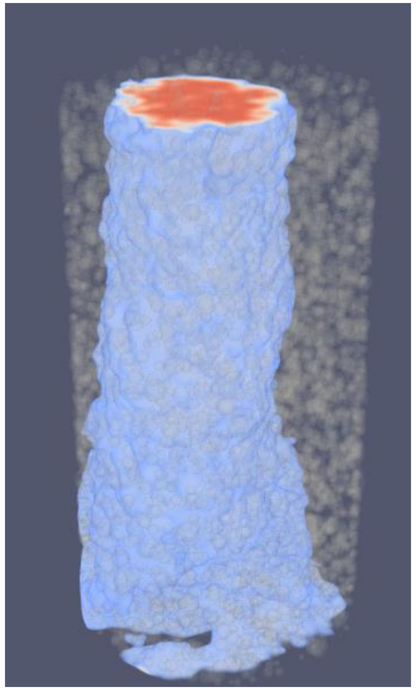


Gostick, J et al., (2016)

OpenPNM Injection Simulation showing saturated regions



Thresholded image from final tomography showing saturated regions



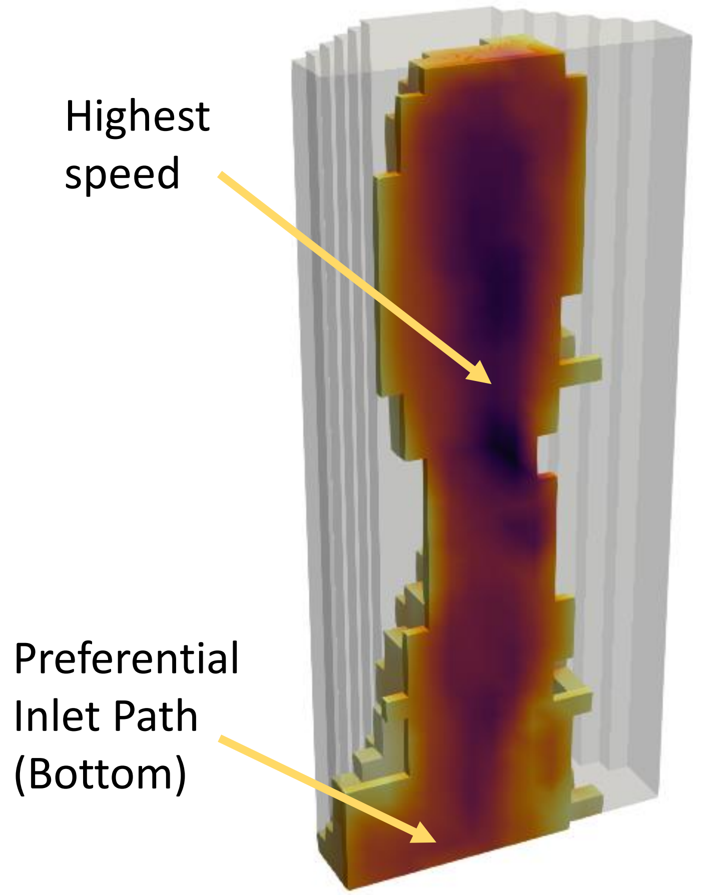
SIMPLE PHYSICS

Pore-throat sorting
Vs
Entry Pressure

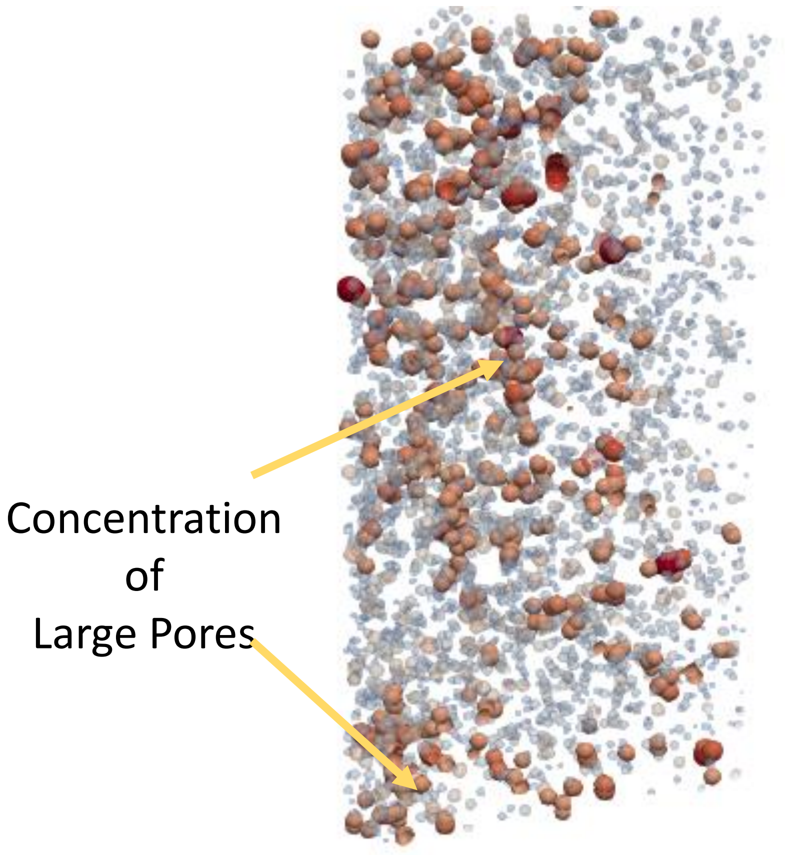
Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

3. RESULTS

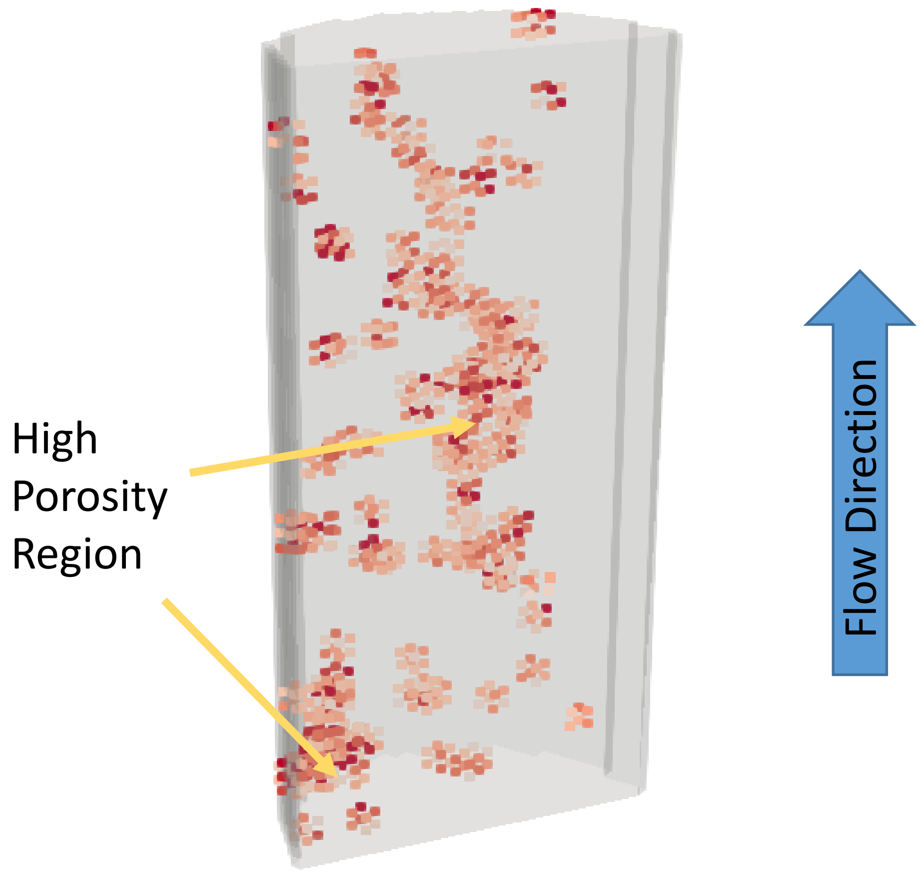
Q1 - HETEROGENEITY VS FLUID FLOW



Flow Speed Field (3D)



Pore Network Model



Thresholded Porosity Map

Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

2. RESEARCH QUESTIONS

Q1 - HOW CAN SAMPLE HETEROGENEITY AFFECT THE FLUID FLOW (UNDEFORMED STATE)? ✓

Q2 - HOW CAN SAMPLE HETEROGENEITY INFLUENCE THE DISTRIBUTION OF DEFORMATION THROUGHOUT DEVIATORIC COMPRESSION?

Q3 - HOW DOES FLUID FLOW EVOLVE ALONG DEVIATORIC COMPRESSION?

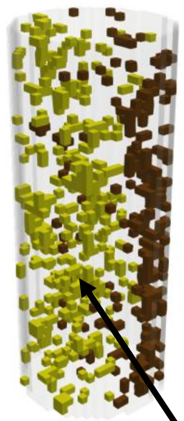
3. RESULTS

Q2 – HETEROGENEITY VS DEVIATORIC DEFORMATION DISTRIBUTION

IG24

HIGHEST POROSITY VOXELS (FROM POROSITY MAPS)

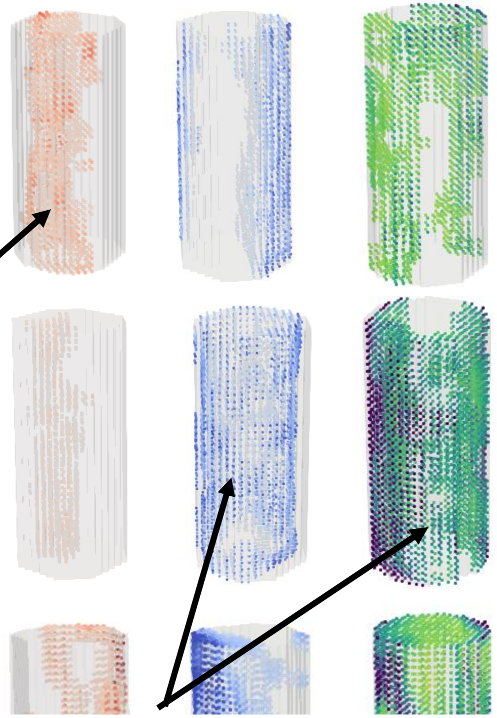
Porosity Map

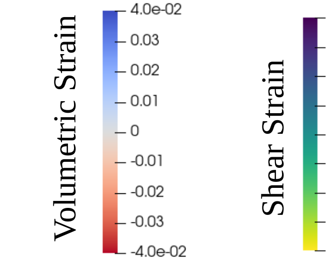


Contractive Strain | Dilative Strain | Shear Strain

$\frac{\varepsilon_{zz}}{\varepsilon_{zzf}} = 0.34$

$\frac{\varepsilon_{zz}}{\varepsilon_{zzf}} = 0.51$





Contraction occurs mostly in the high-porosity zone (one side of the sample)

Dilation starts to dominate, and shear increases

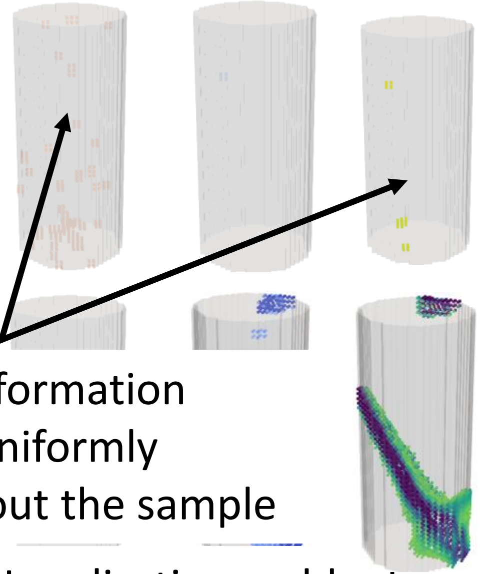
Localization occurs 

IG16

Porosity Map

Contractive Strain | Dilative Strain | Shear Strain

$\frac{\varepsilon_{zz}}{\varepsilon_{zzf}} = 0.61$



Small deformation spread uniformly throughout the sample

Localization suddenly occurs

Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

LUND UNIVERSITY | LTH FACULTY OF ENGINEERING

3. RESULTS

Q2 – HETEROGENEITY VS DEVIATORIC DEFORMATION DISTRIBUTION

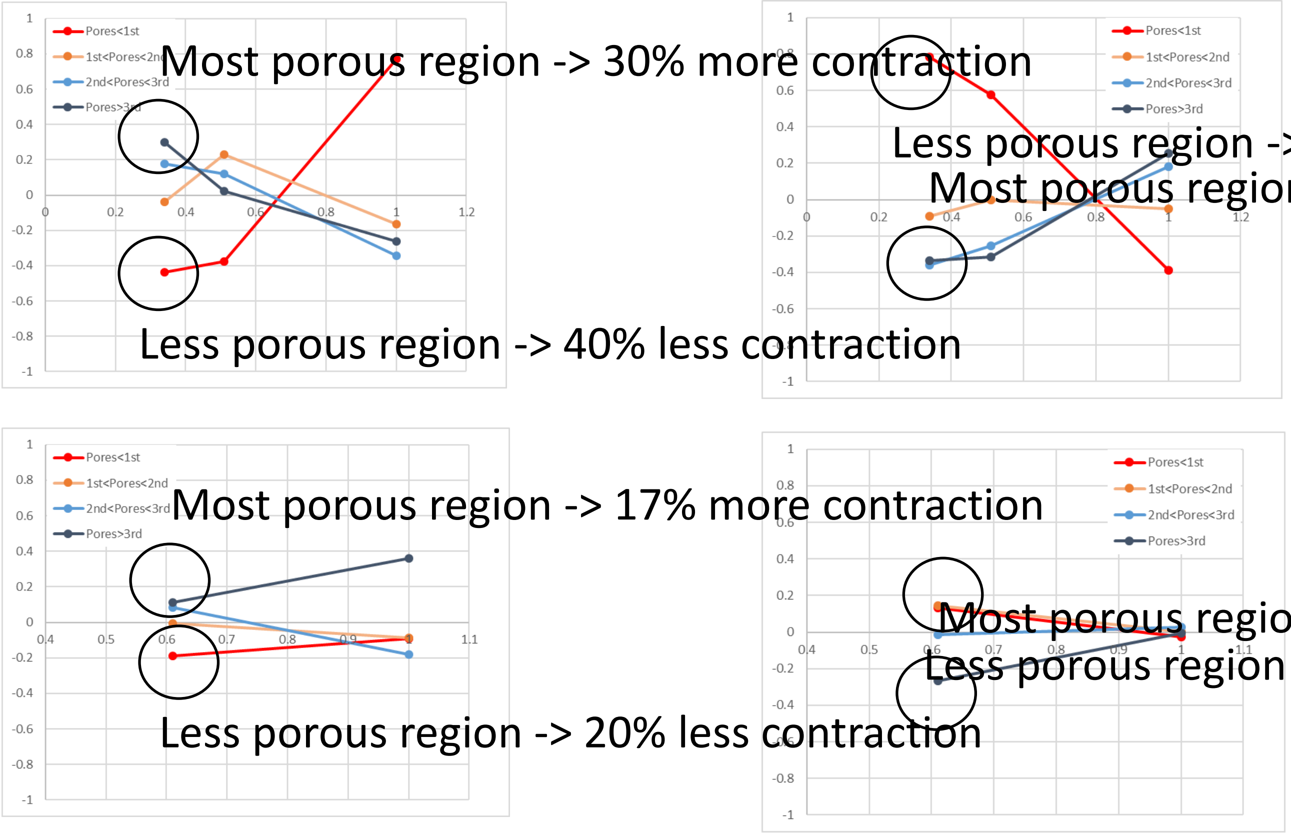
STATISTICS - FOUR GROUPS OF POROSITY: THE STRAIN EVOLUTION COMPARED TO THE SAMPLE'S AVERAGE

Contractive Strain

Dilative Strain

IG24

IG16



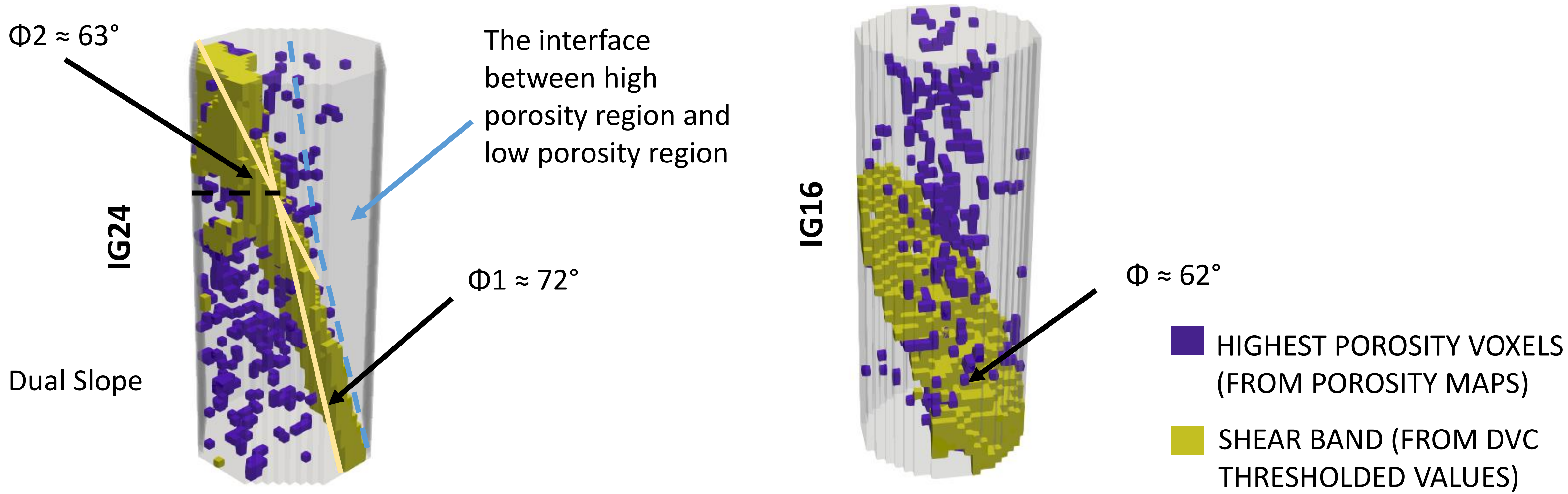
Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

LUND UNIVERSITY | LTH FACULTY OF ENGINEERING

Q2 – HETEROGENEITY VS DEVIATORIC DEFORMATION DISTRIBUTION

DIGITAL VOLUME CORRELATION VS POROSITY MAPS FROM HIGH-RESOLUTION X-RAY IMAGES

SHEAR BAND VS HIGH-POROSITY CONCENTRATION



Vieira Lima et al (2022). Characterization of triaxial deformation and hydraulic behavior of porous sandstones through in-situ testing with X-ray and neutron tomography. ILL-ESS User Meeting 2022.

2. RESEARCH QUESTIONS

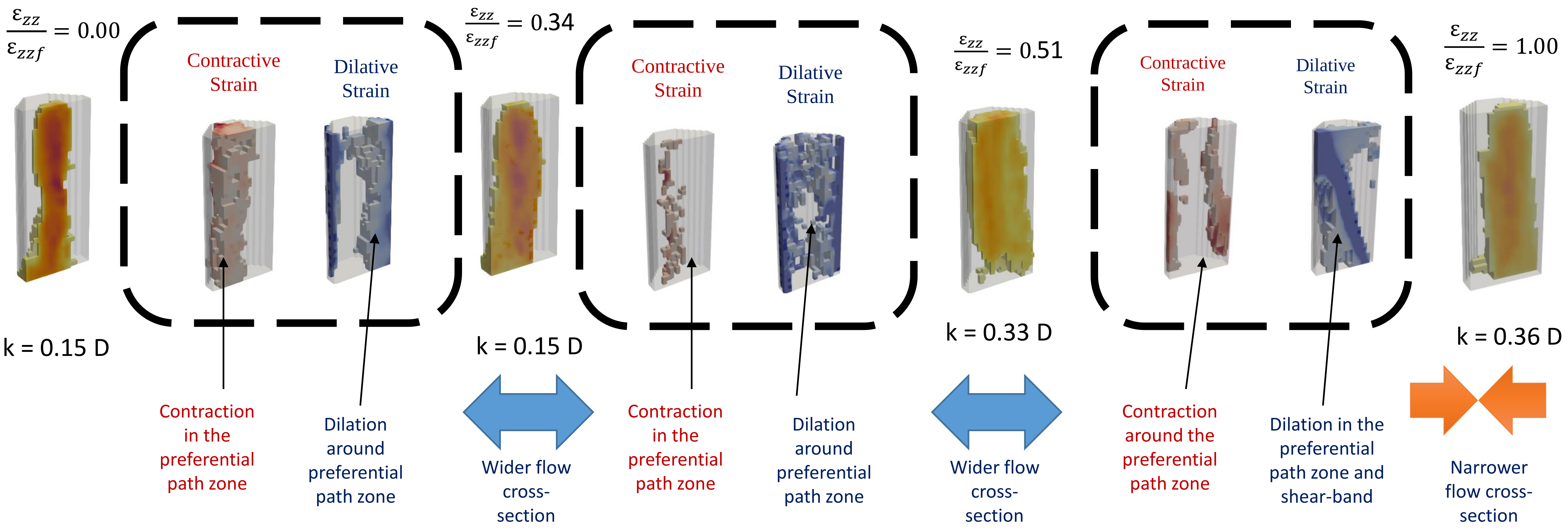
Q1 - HOW CAN SAMPLE HETEROGENEITY AFFECT THE FLUID FLOW (UNDEFORMED STATE)? ✓

Q2 - HOW CAN SAMPLE HETEROGENEITY INFLUENCE THE DISTRIBUTION OF DEFORMATION THROUGHOUT DEVIATORIC COMPRESSION? ✓

Q3 - HOW DOES FLUID FLOW EVOLVE ALONG DEVIATORIC COMPRESSION?

Q3 – FLUID FLOW EVOLUTION ALONG THE DEVIATORIC COMPRESISION

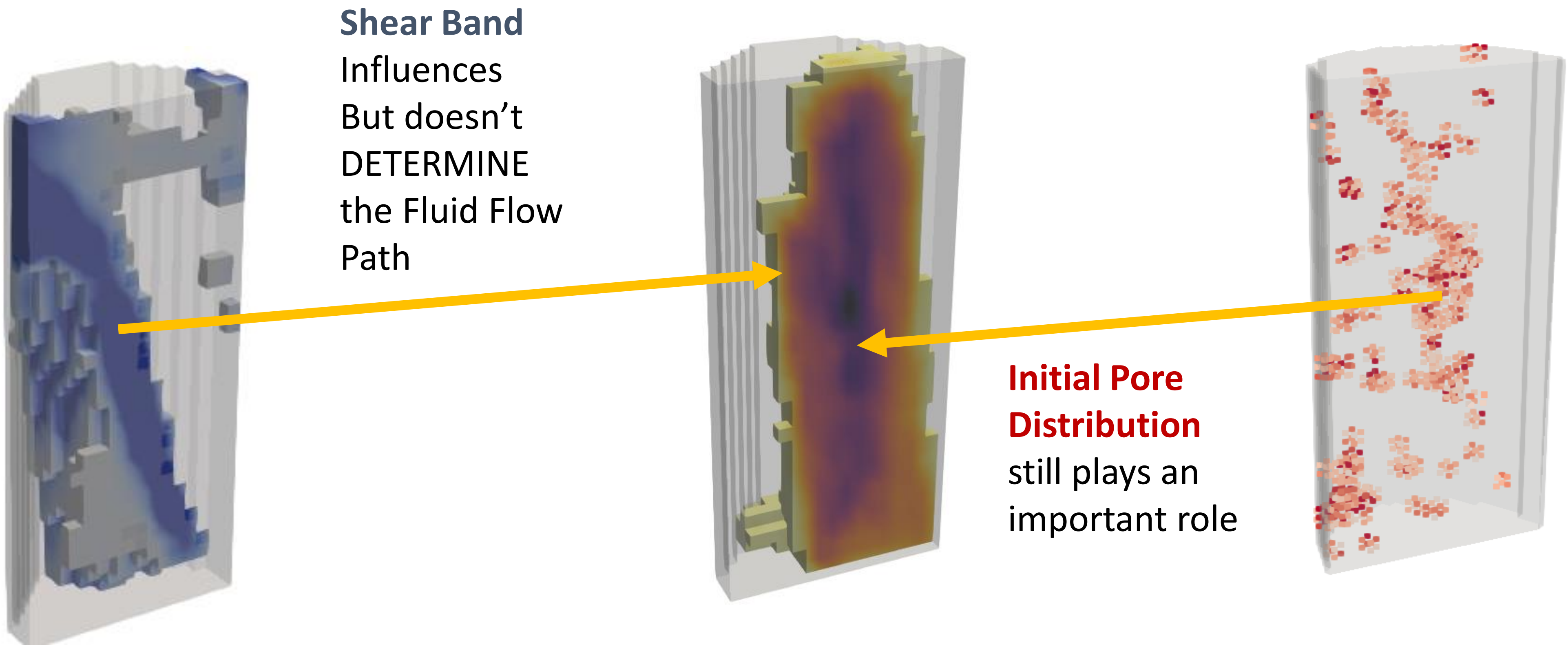
FLOW SPEED FIELDS VS CROSS-SECTION VOLUMETRIC STRAINS



Vieira Lima et al (2022). Understanding the influence of triaxial deformation and heterogeneity on the hydraulic behavior of porous sandstones from in-situ testing with x-ray and neutron tomography. Alert Geomaterials Workshop, 2022.

3. RESULTS

Q3 – FLUID FLOW EVOLUTION ALONG THE DEVIATORIC COMPRESISION



Vieira Lima et al (2022). Understanding the influence of triaxial deformation and heterogeneity on the hydraulic behavior of porous sandstones from in-situ testing with x-ray and neutron tomography. Alert Geomaterials Workshop, 2022.

5. CONCLUSIONS AND FURTHER DEVELPMENTS

- **A novel testing system** for rock's hydromechanical investigation
    - **4D imaging of flow and deformation** under a triaxial state of stress .

- Bulk measurements
- Porosity maps
- DVCs
    - Compaction and Dilation (all steps)
    - Strain localization
- Fluid front tracking and z-speed field

Q1 – FLOW PATH AND SPEED DEPEND ON PORE SIZE AND CONNECTIVITY

Q2 - HETEROGENEITY INFLUENCES THE DEFORMATION PATTERN AND SHEAR BAND GEOMETRY

Q3 - FLUID FLOW PATH AND SPEED DEPEND ON DEFORMATION AND ON INITIAL PORE DISTRIBUTION

Help to understand the hydromechanics of the samples

Vieira Lima et al (2022). Understanding the influence of triaxial deformation and heterogeneity on the hydraulic behavior of porous sandstones from in-situ testing with x-ray and neutron tomography. Alert Geomaterials Workshop, 2022.

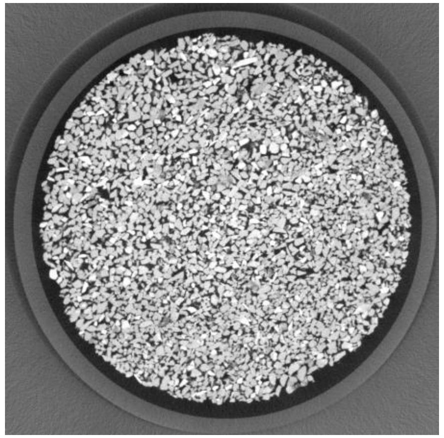
WE CAN DO IT BETTER!

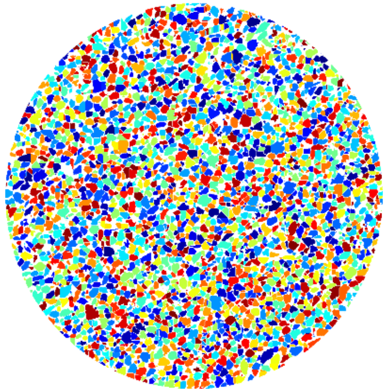
X-ray High-Resolution Scan with a Laboratory Tomography

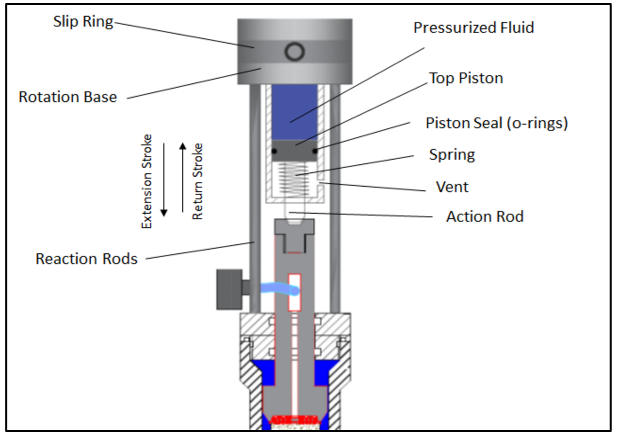
Acquisition time = 3x15 mins

14 µm

Inside the cell







GRAINS TRACKING?

CRACKS TRACKING?

DAMAGE TRACKING?

PNM INJECTION SIMULATION FOR EVERY LOAD STEP?

Vieira Lima et al (2022). Understanding the influence of triaxial deformation and heterogeneity on the hydraulic behavior of porous sandstones from in-situ testing with x-ray and neutron tomography. Alert Geomaterials Workshop, 2022.

5. REFERENCES

- Masschaele, B., et al. (2004), "The use of neutrons and monochromatic X-rays for non-destructive testing in geological materials", in: Environmental Geology, Vol. 46, No. 3-4, pp. 486–492.
- Hall, S. A. (2013), "Characterization of fluid flow in a shear band in porous rock using neutron radiography", in: Geophysical Research Letters, Vol. 40, No. 11, pp. 2613–2618.
- Tudisco, E., Hall, S. A., Athanasopoulos, S., and Hovind, J. (2017), "Neutron imaging and digital volume correlation to analyse the coupled hydro-mechanics of geomaterials", in: Rivista Italiana di Geotecnica, Vol. 51, No. 4, pp. 60–68.
- Tudisco, E. et al. (2019), "Fast 4-D Imaging of Fluid Flow in Rock by High-Speed Neutron Tomography," J. Geophys. Res. Solid Earth, vol. 124, no. 4, pp. 3557–3569, doi: 10.1029/2018JB016522.
- Woracek, R. (2021), LINXS Neutron Course Notes
- Etxegarai, M. (2019), "Coupled hydro- mechanics of reservoir rocks studied by quantitative in-situ neutron imaging,".
- Woracek, R. (2015), "Energy selective neutron imaging for the characterization of polycrystalline materials".
- Etxegarai, M., et. al. (2021). Characterisation of single-phase fluid-flow heterogeneity due to localised deformation in a porous rock using rapid neutron tomography. Journal of Imaging, 7(12), 275.
- Stamati et al., (2020). spam: Software for Practical Analysis of Materials. Journal of Open Source Software, 5(51), 2286, https://doi.org/10.21105/joss.02286
- Gostick, J et al., (2016) OpenPNM: a pore network modeling package. Computing in Science & Engineering. doi:10.1109/MCSE.2016.49.
- Boccardo, G et al. (2020) Nanomaterials for the Detection and Removal of Wastewater Pollutants. https://doi.org/10.1016/B978-0-12-818489-9.00013-X

THANK YOU
ANY QUESTIONS?

fernando.vieira_lima@solid.lth.se

ACKNOWLEDGMENT

LUND UNIVERSITY

LTH FACULTY OF ENGINEERING


Stephen Hall


Jonas Engqvist


Erika Tudisco

ess EUROPEAN SPALLATION SOURCE


Robin Woracek

UGA Université Grenoble Alpes

ILL NEUTRONS FOR SOCIETY


Alessandro Tengattini


Cyrille Couture