Neutron (and X-ray) imaging for coupled processes in porous media

Alessandro Tengattini *et al.*

Laboratoire
Sols Solides Structures Risques

NEUTRONS
FOR SCIENCE

- *Relatively* new (2015) Neutron Tomograph
- Born from the collaboration between

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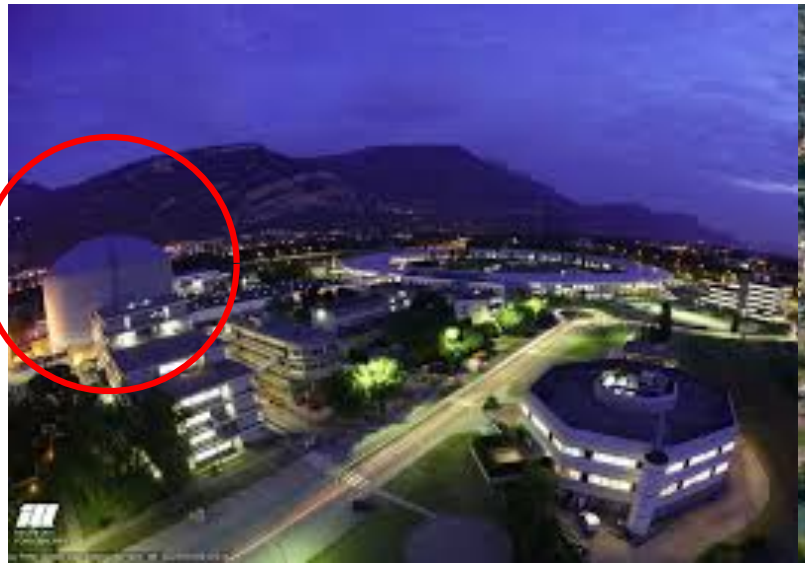

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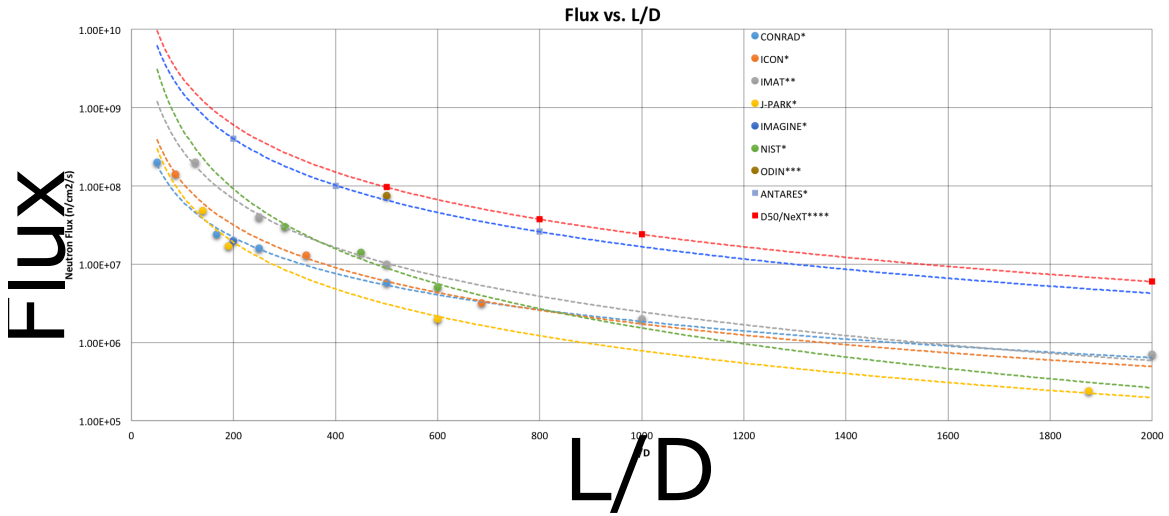

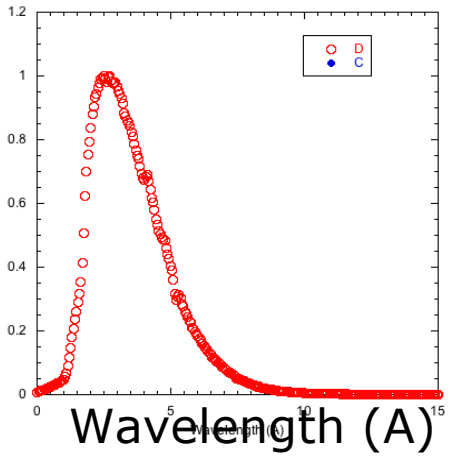

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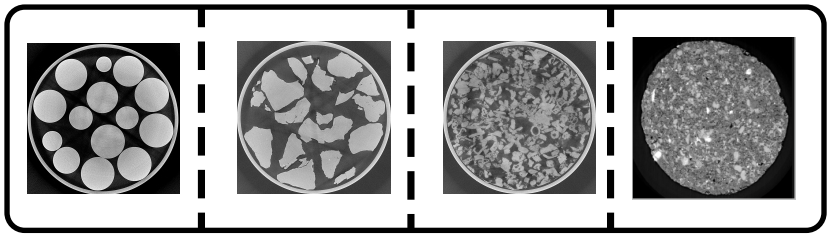
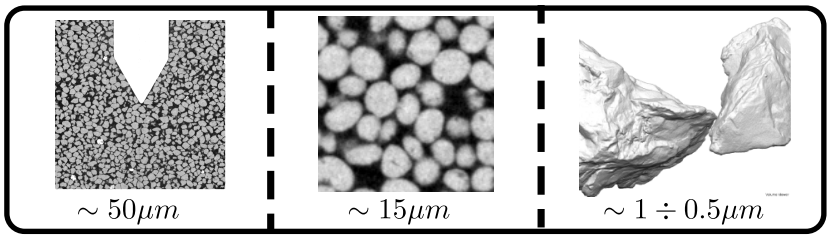
geometry

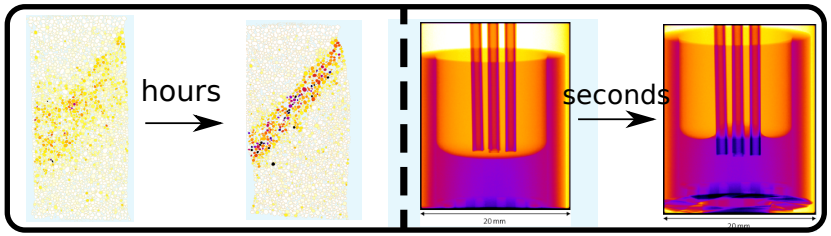


scale

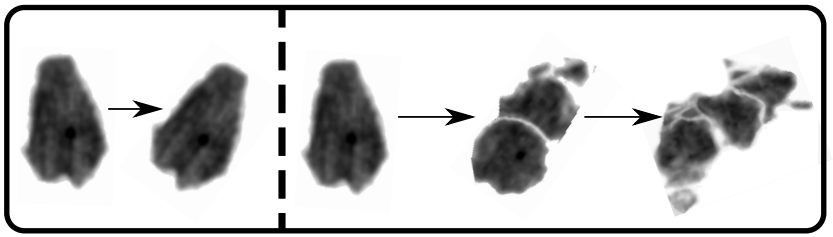


speed

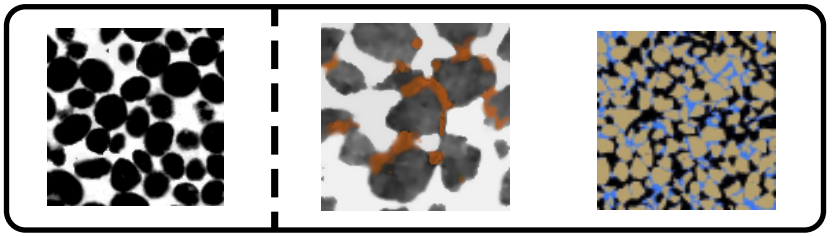
slow tomograph (at 3SR) ultrafast (ESRF)



material persistance



# of mechanically relevant phases



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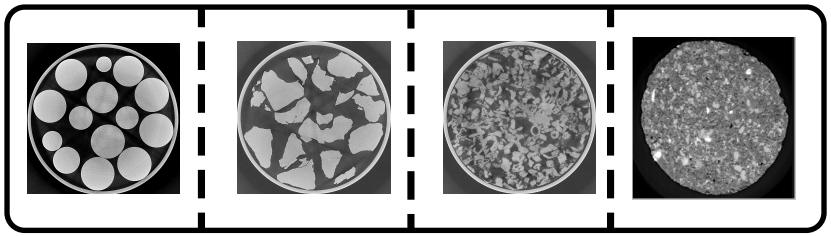

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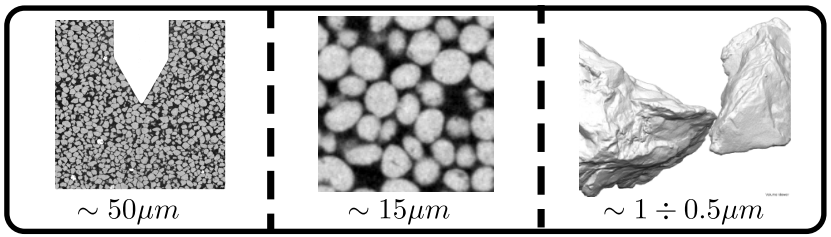

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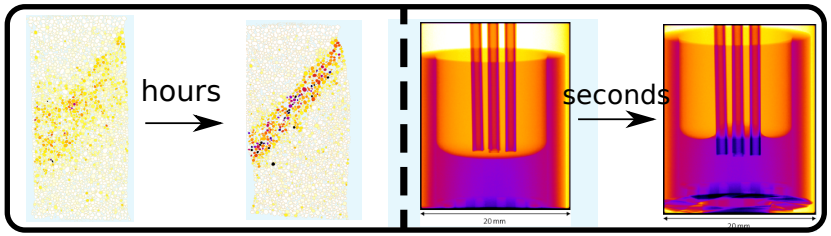

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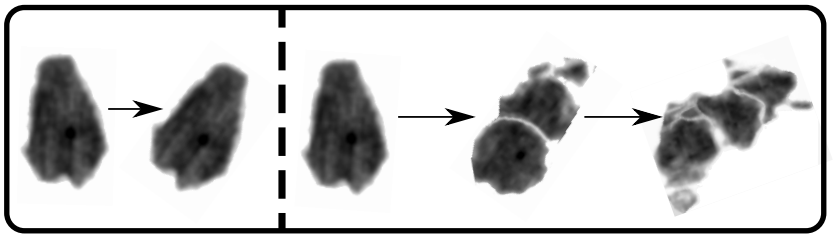

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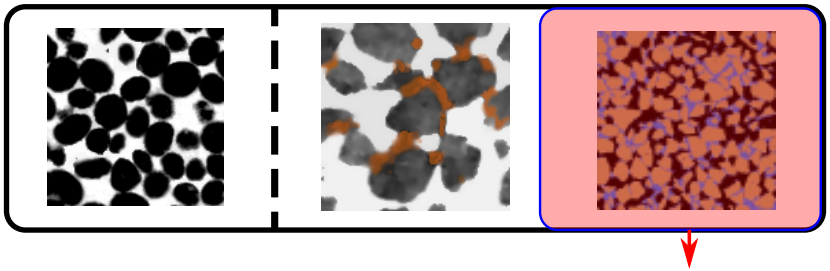
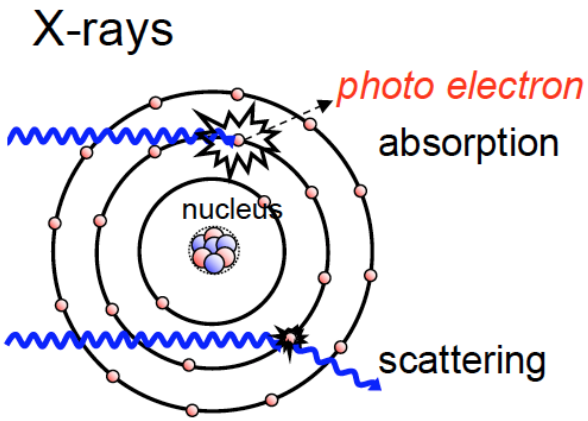


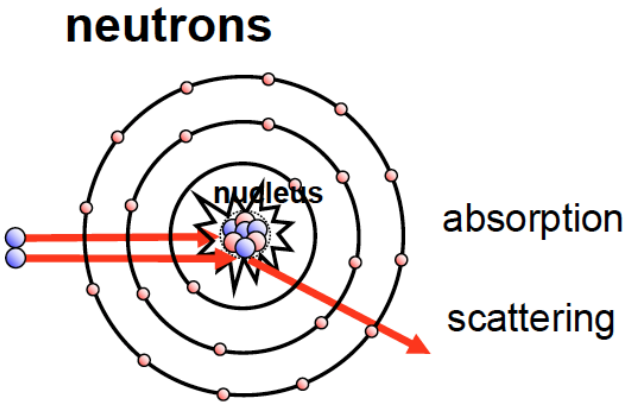
imaging fluids (e.g. hydrocabons) in geomaterials



Attenuation coefficients with X-ray [cm²¹]

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													

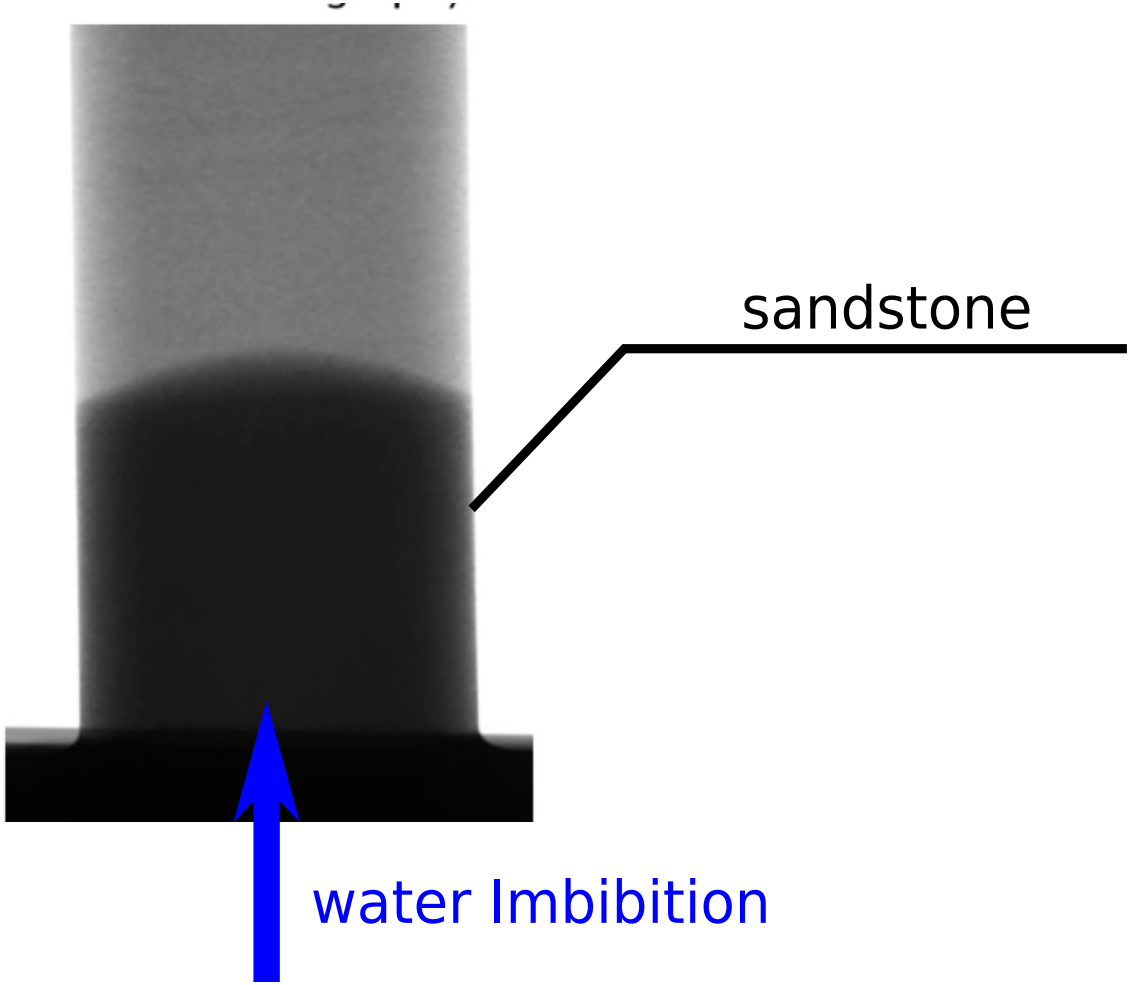
*Lanthanides	Ce 5.79	Pr 6.23	Nd 6.46	Pm 7.33	Sm 7.68	Eu 5.66	Gd 8.69	Tb 9.46	Dy 10.17	Ho 10.91	Er 11.70	Tm 12.49	Yb 9.32	Lu 14.07
**Actinides	Th 28.95	Pa 39.65	U 49.08	Np	Pu	Am	Cm	Bk	Vf	Es	Fm	Md	No	Lr x-ray



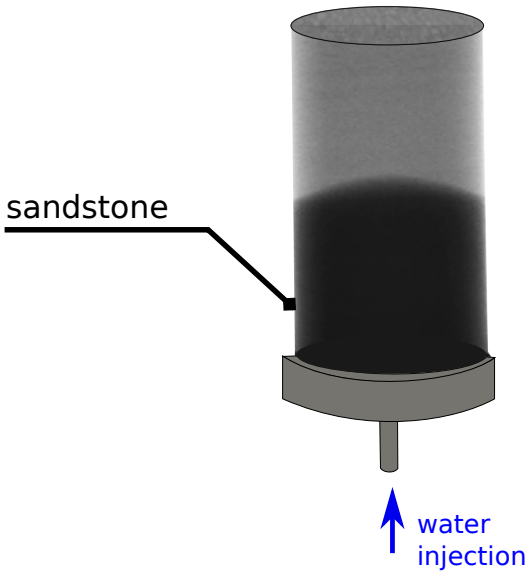
Attenuation coefficients with neutrons [cm²¹]

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													

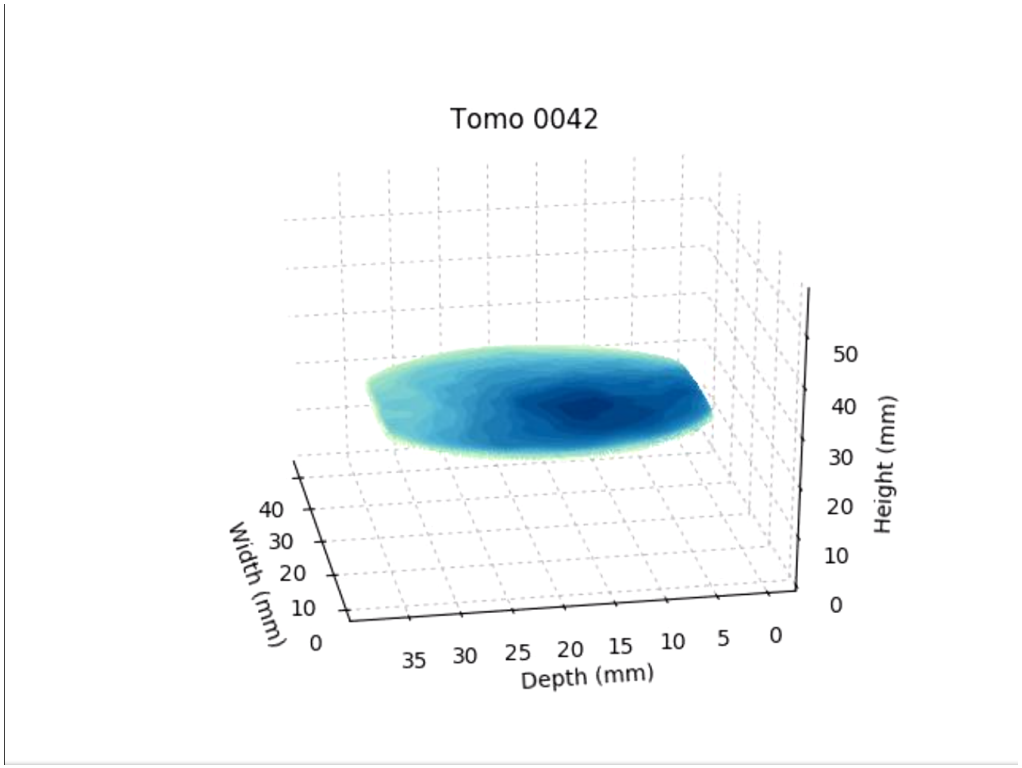
*Lanthanides	Ce 0.14	Pr 0.41	Nd 1.87	Pm 5.72	Sm 171.47	Eu 94.58	Gd 1479.04	Tb 0.93	Dy 32.42	Ho 2.25	Er 5.48	Tm 3.53	Yb 1.40	Lu 2.75
**Actinides	Th 0.59	Pa 8.46	U 0.82	Np 9.80	Pu 50.20	Am 2.86	Cm	Bk	Cf	Es	Fm	Md	No	Lr neut.


sandstone
water Imbibition

Fluid flow within an intact rock

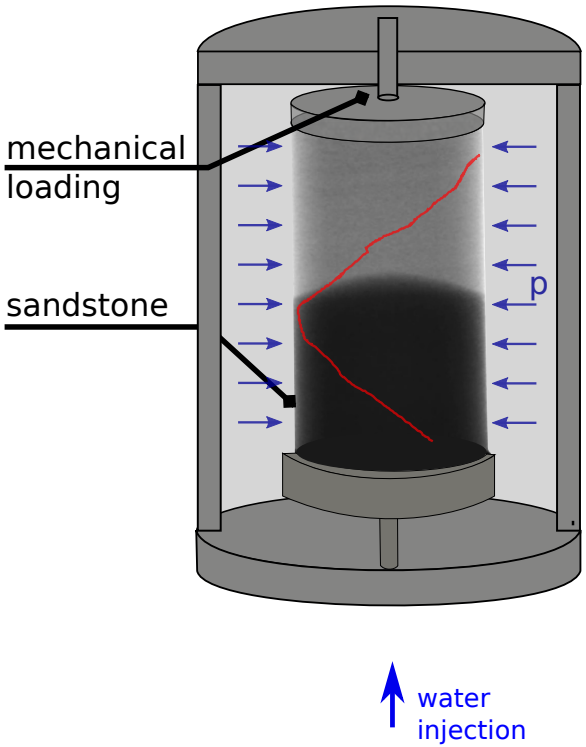


Quantification of front position, speed, permeability



Tudisco, *et al.*, Journal of Geophysical Research: Solid Earth (2019)
Extegarai, *et al.*, Journal of Imaging (2021)
Viera-Lima, *et al.*, submitted (2023)

Fluid flow within a damaged rock



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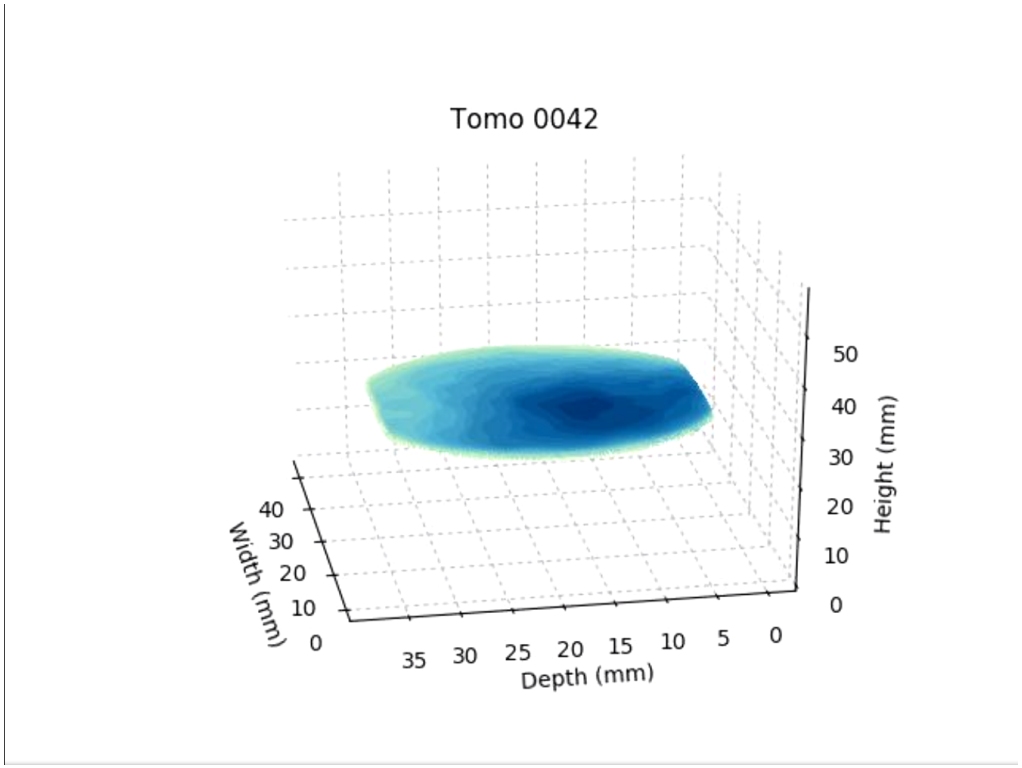
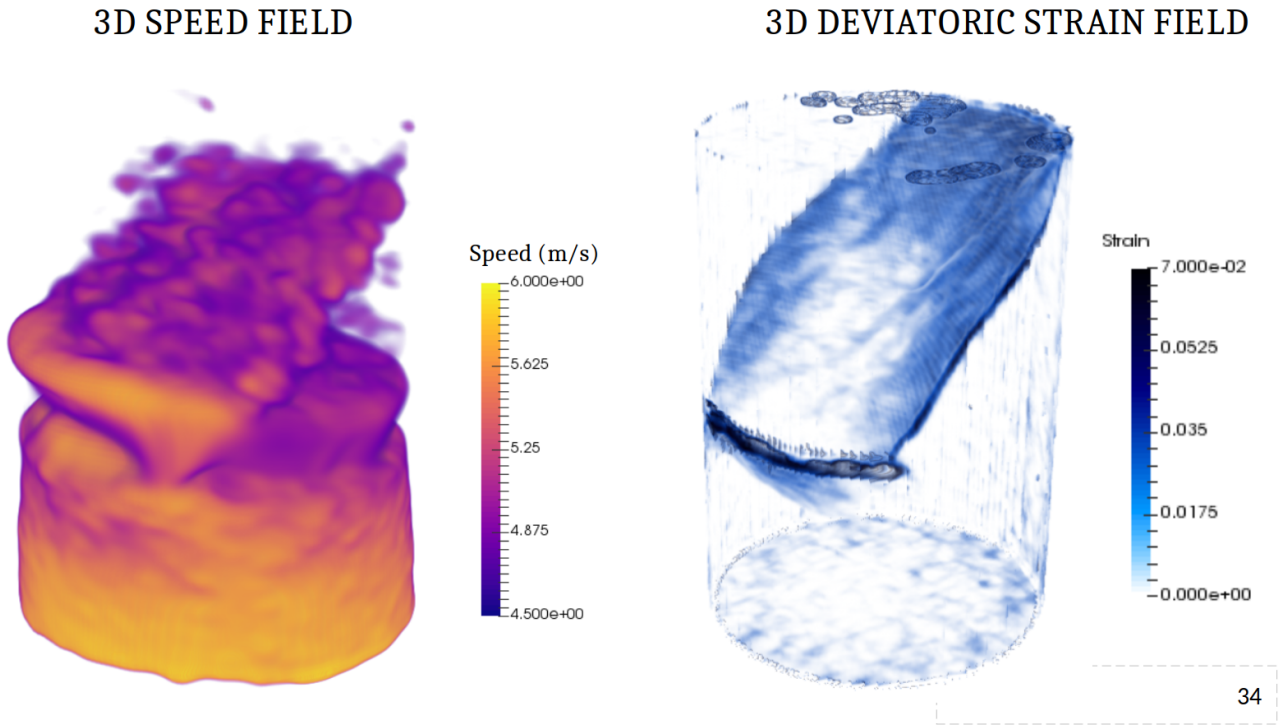
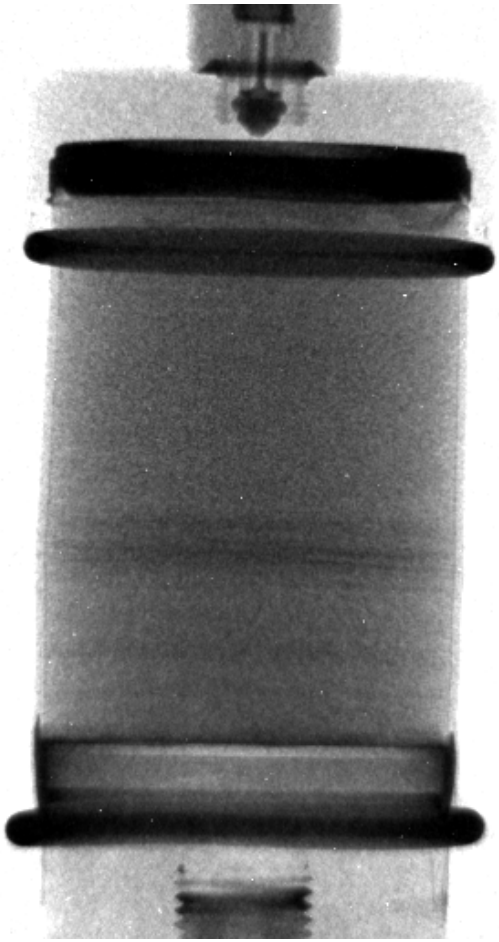


Tudisco, *et al.*, Journal of Geophysical Research: Solid Earth (2019)
Extegarai, *et al.*, Journal of Imaging (2021)
Viera-Lima, *et al.*, submitted (2023)

Quantification of incremental strain through Digital Volume correlation of the map of fluid Speed

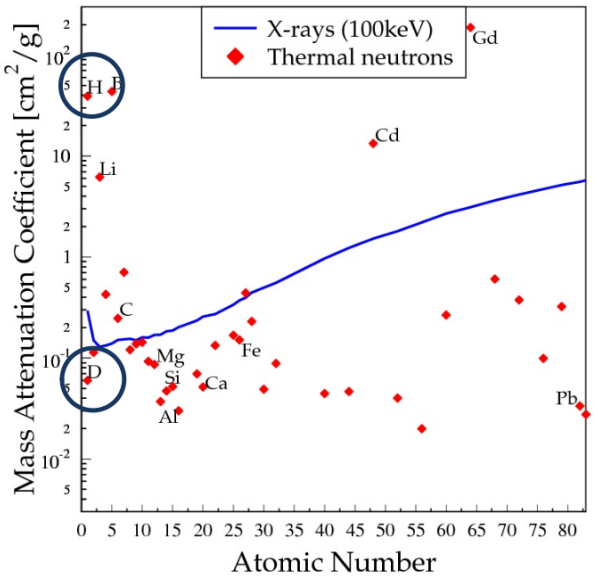


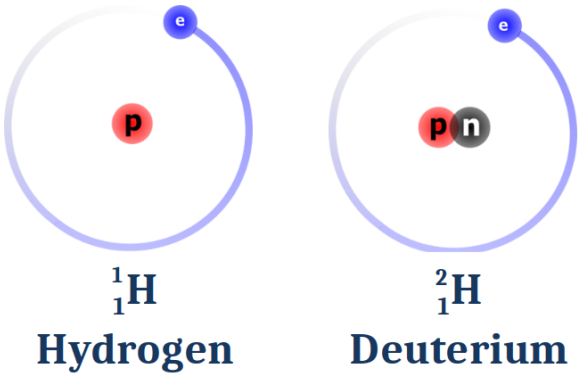
How about more complex geometries?



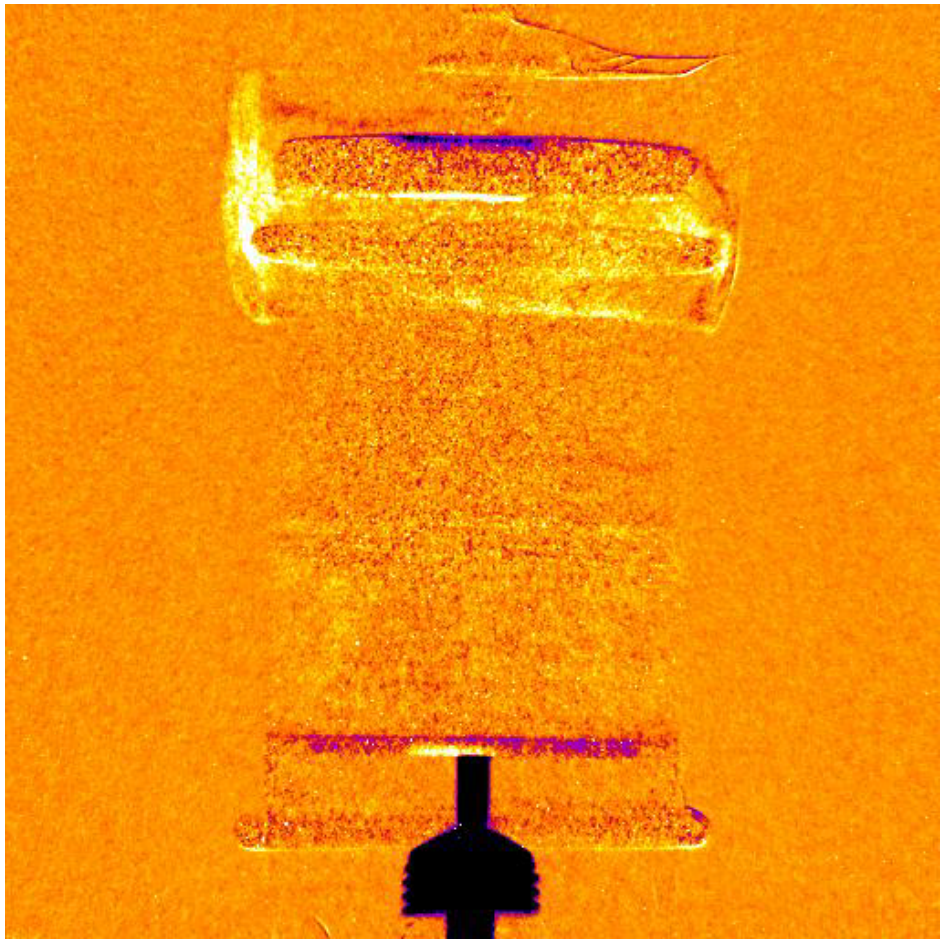
Lewis *et al.*, EGU (2017)
Lewis *et al.*, Transport in Porous Media (2023)

Neutron are isotope sensitive, "**normal water**" and **heavy water** have an order of magnitude difference in opacity
Can be used to **track fluid in an already saturated sample**



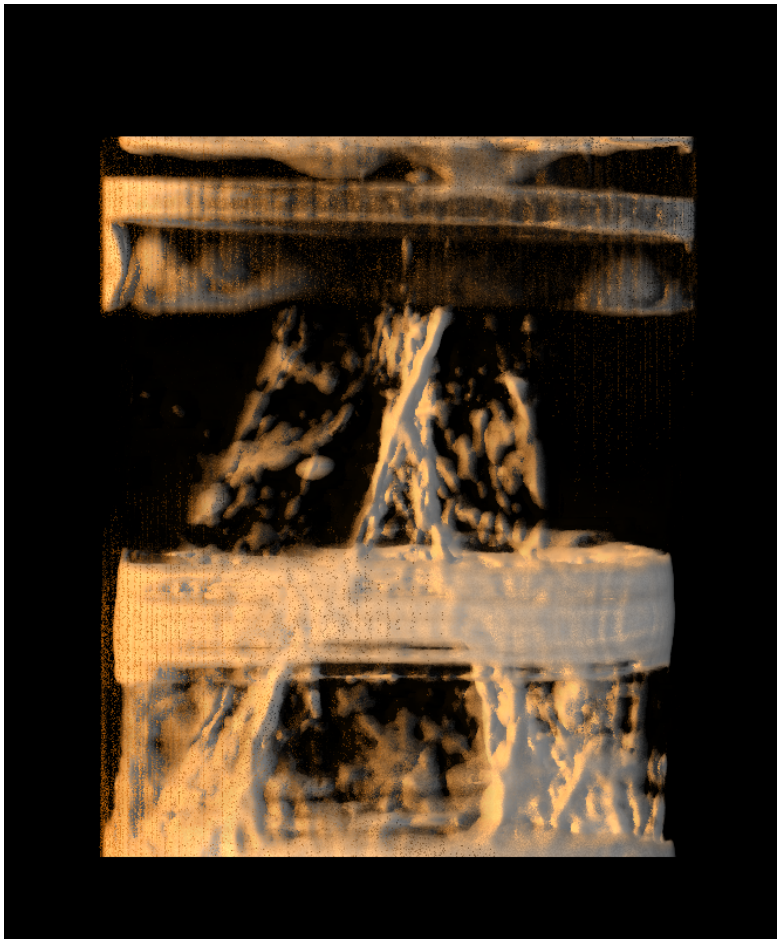


Injection in D20-saturated sample



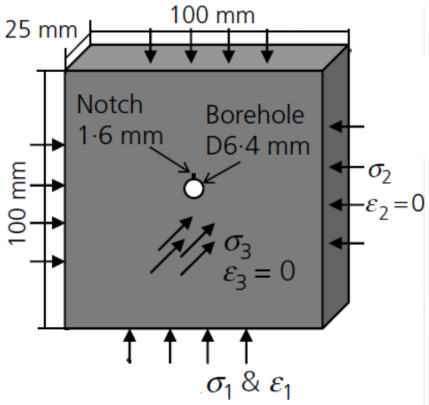
Lewis *et al.*, EGU (2017)
Lewis *et al.*, Transport in Porous Media (2023)

and in 3D?



Lewis *et al.*, EGU (2017)
Lewis *et al.*, Transport in Porous Media (2023)

Hydrofracking of rocks

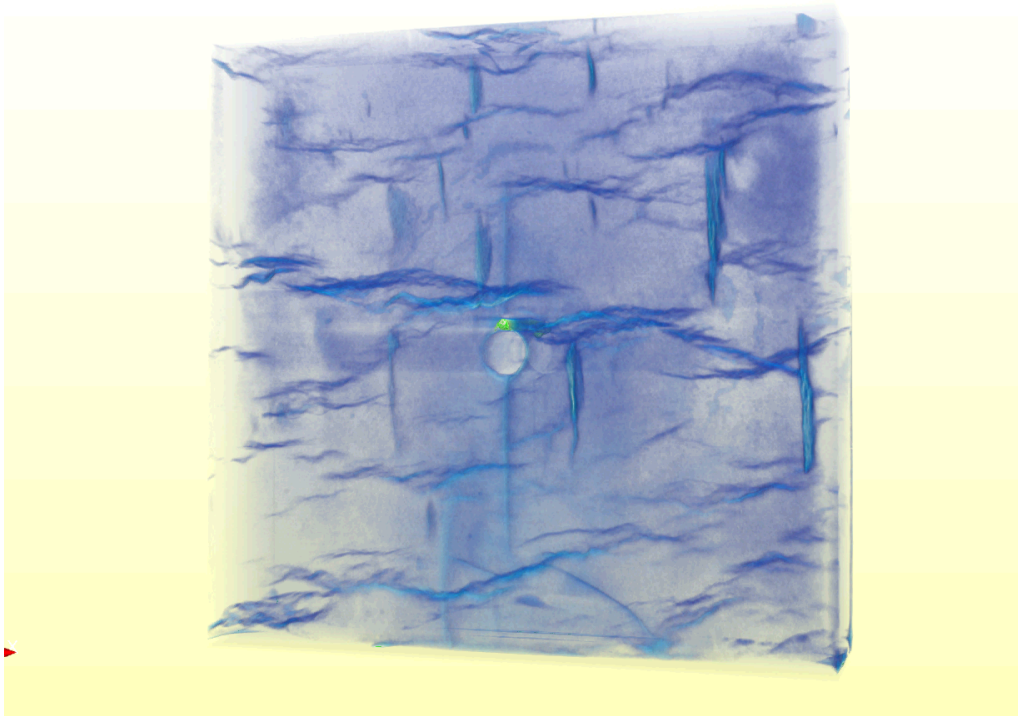


Marcellus shale

$\sigma_1 = 70$ MPa

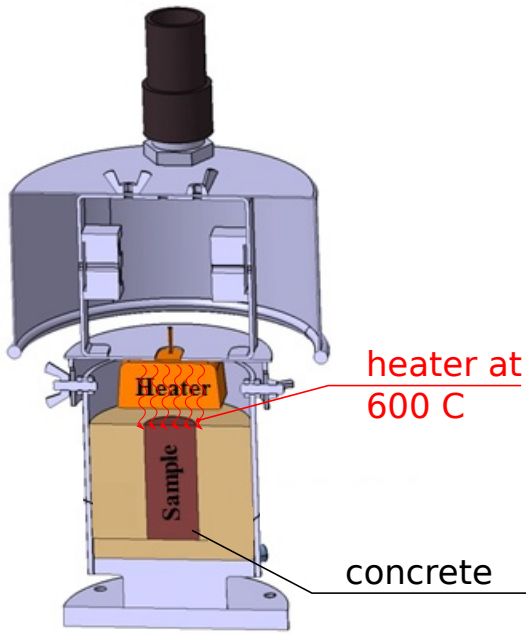
Flow rate /6/12 ml/min

Quantification of front position, speed, permeability



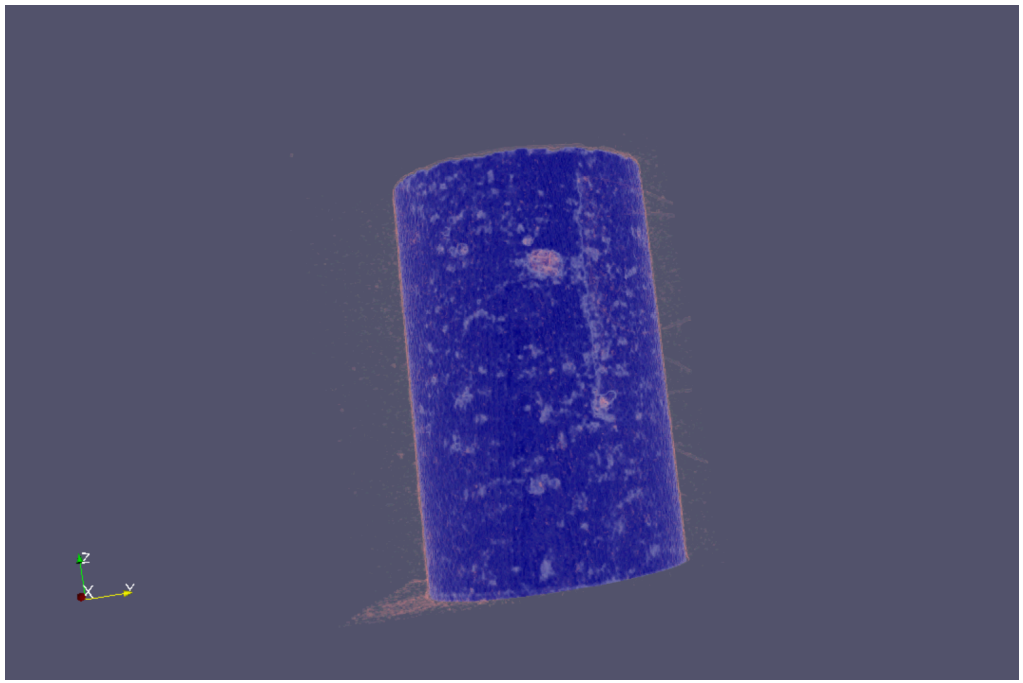
Roshankhah, *et al.*, Géotechnique letters (2018)
Roshankhah, *et al.*, USRMS (2019)

Heating construction materials to fire-like conditions



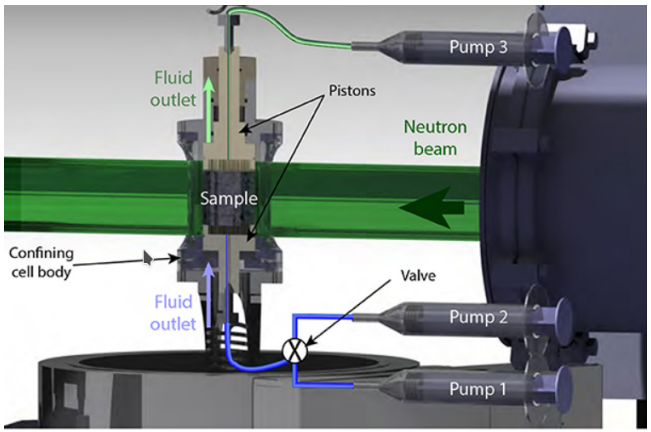
Understanding of explosive spalling for fire safety of buildings

Quantification of moisture migration, accumulation for different materials, 20 second tomographies



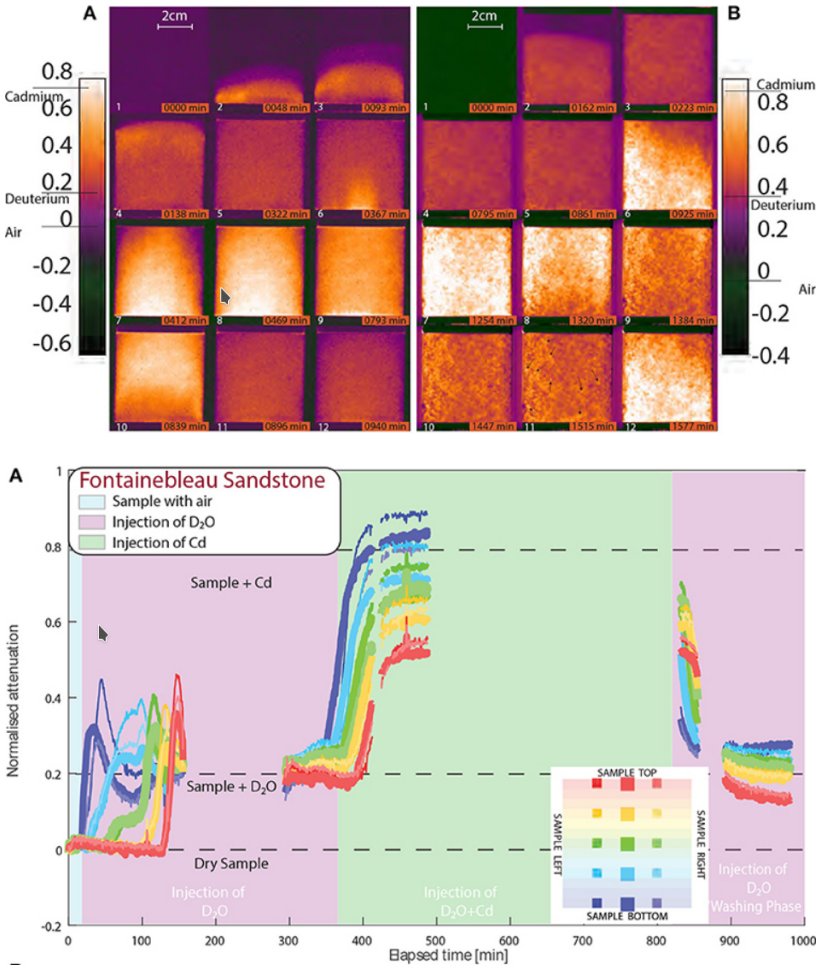
Dauti *et al.*,, Cement and Concrete Research (2018)
Dauti *et al.*, Transport in Porous Media (2019)
Tengattini *et al.*, Strain (2020)

Cadmium Sorption and Transport in Porous Rocks



Study of Cd concentration

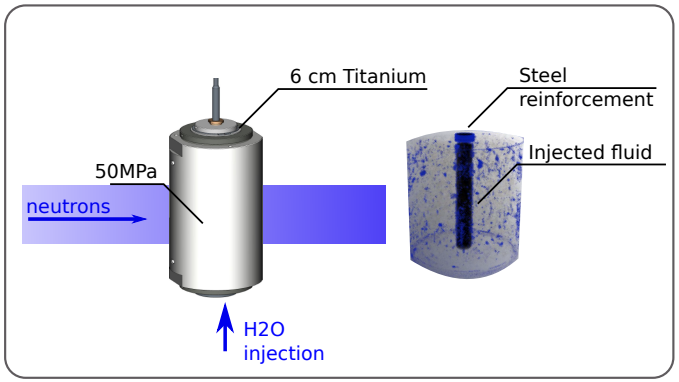
Quantification of Cd sorption and displacement of Contaminants



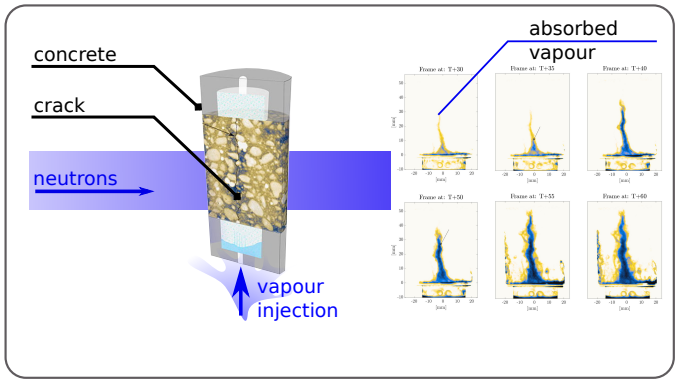
Cordonnier *et al.*, Front. Earth Sci (2019)

Other applications

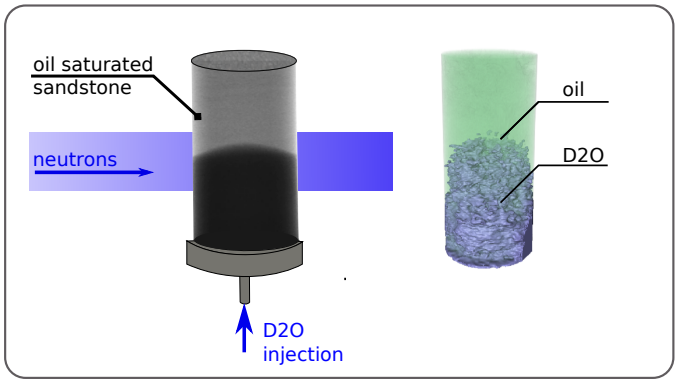
Neutrons can penetrate through thick enviromental cells [1]



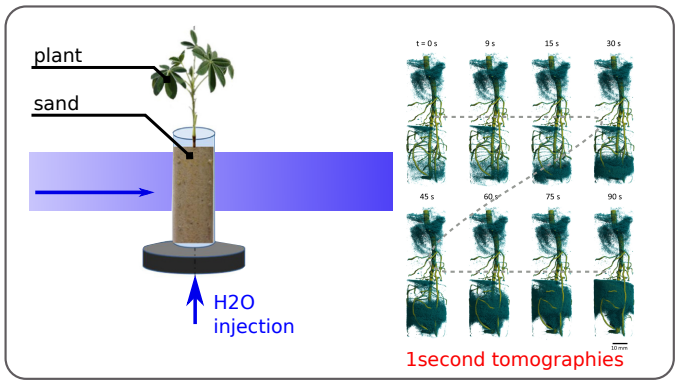
Neutrons can help track even vapour low into fractures [2]



Neutrons can help track flow into immiscible fluids (e.g., oil) [3]



Neutrons tomographies can be acquired as fast as 1s per tomography [4]



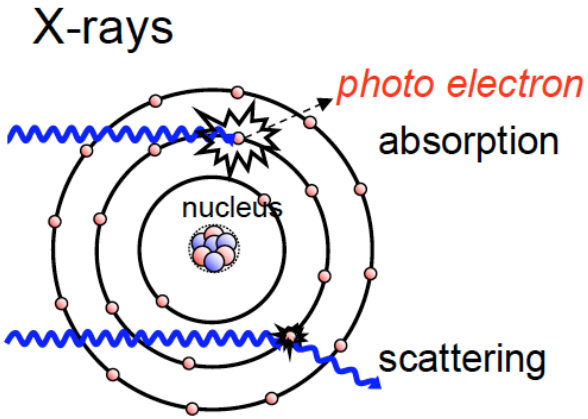
[1] Yehya *et Al,*. Nucl. Inst. Met. Phys. Res. A (2018)
[3] Madankan *et al.*, Ready for submission (2021)

[2] Lukich *et al.*, Mat. Letters (2021)
[4] Totzke *et al.*, Optics Express (2019)

In summary

- Neutrons have a different contrast from x-rays
- Neutrons can see light elements (such as hydrogen, lithium) and their compounds (*e.g.*, water, oil)
- Neutrons are isotope sensitive; for example can distinguish deuterated fluids as D20 vs H2O
- Neutron can penetrate through thick metal casings for extreme conditions (pressure, temperature, acids)

Neutrons or X-Rays?



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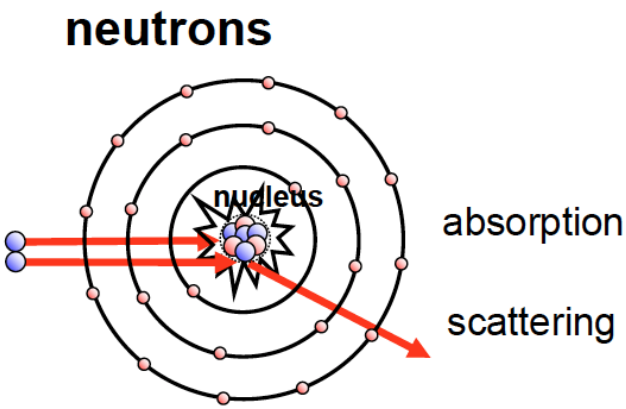

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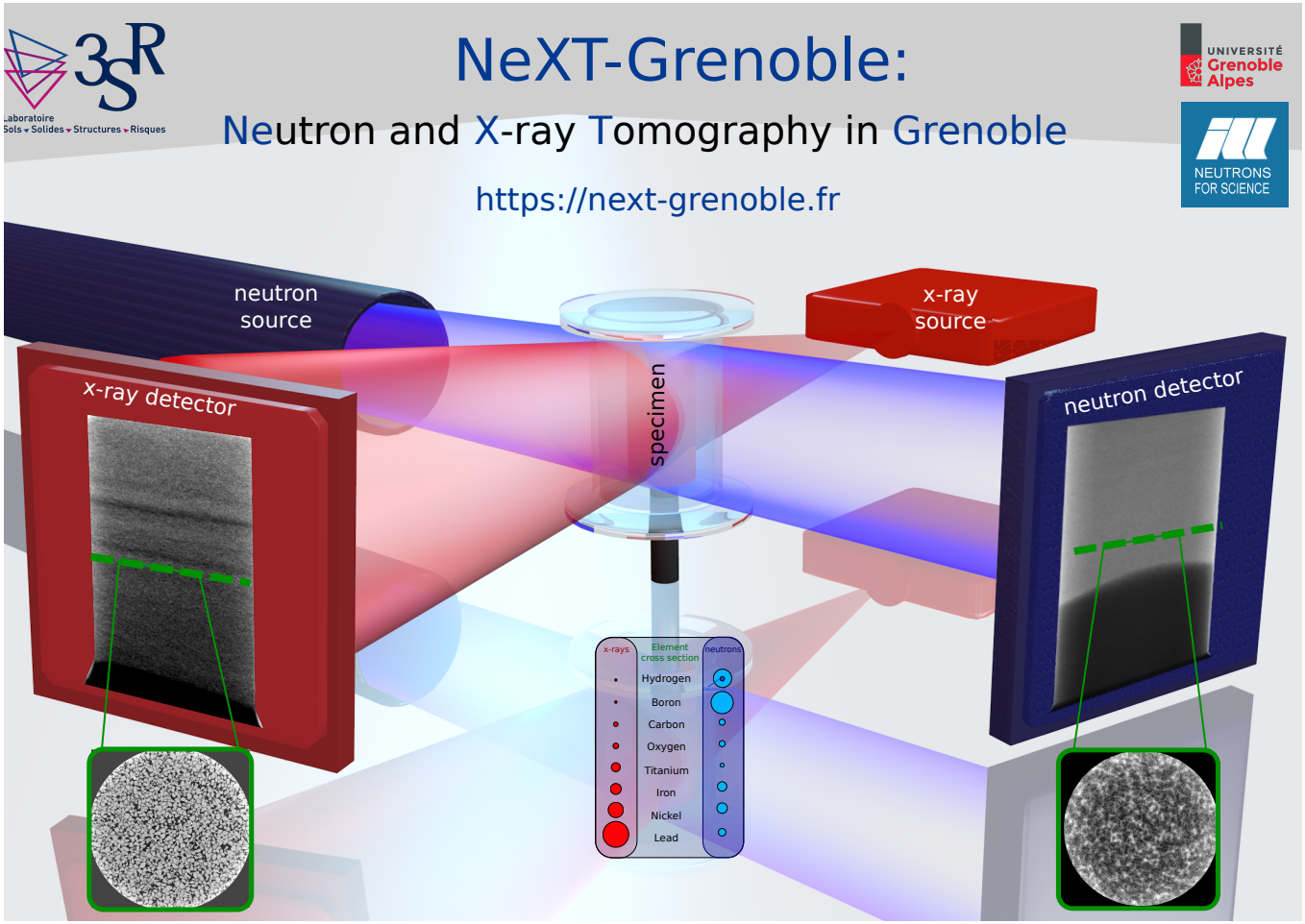
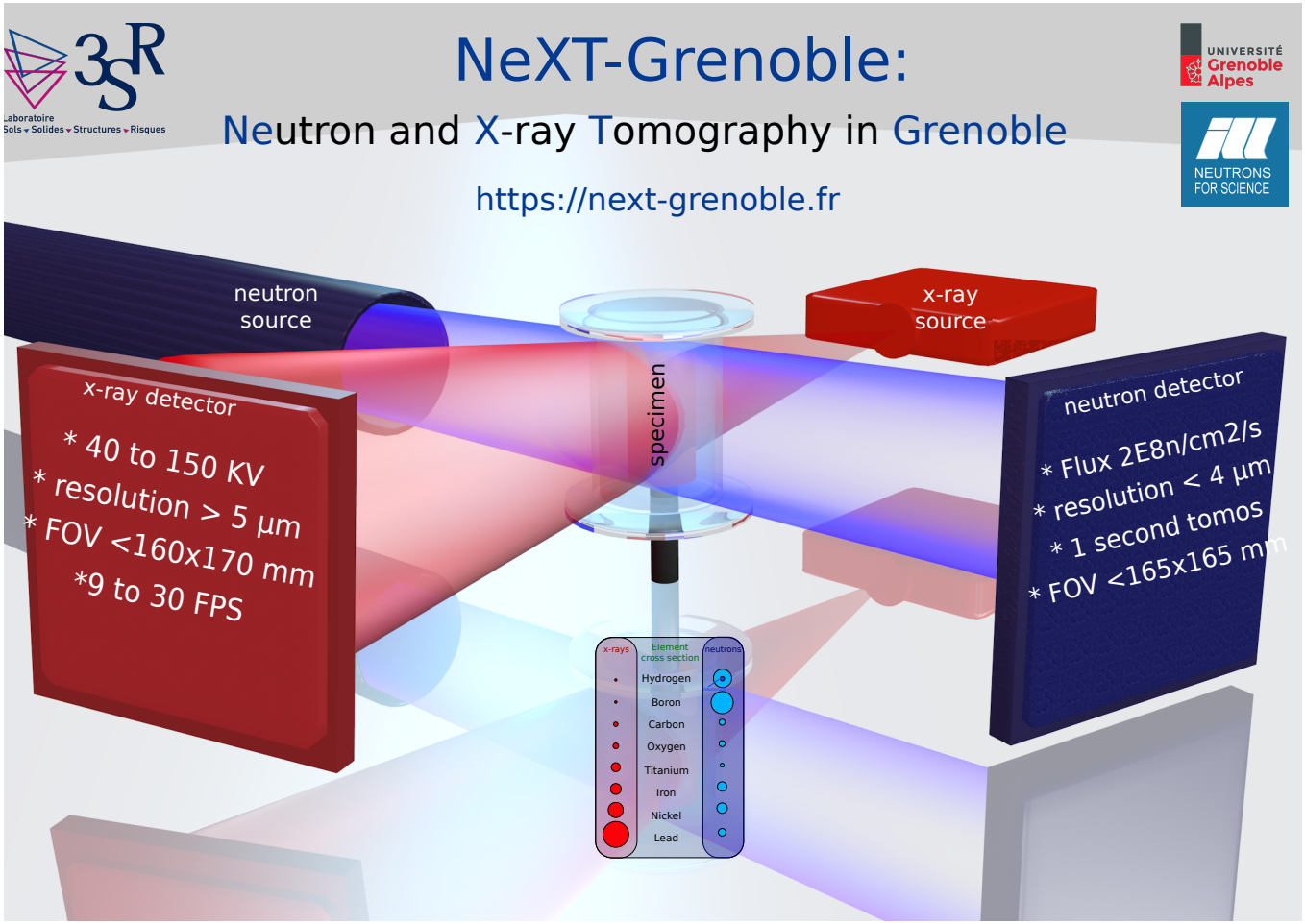
K 0.06	Ca 0.08	Sc 2.00	Ti 0.60	V 0.72	Cr 0.54	Mn 1.21	Fe 1.19	Co 3.92	Ni 2.05	Cu 1.07	Zn 0.35	Ga 0.49	Ge 0.47	As 0.67	Se 0.73	Br 0.24	Kr 0.61
Rb 0.08	Sr 0.14	Y 0.27	Zr 0.29	Nb 0.40	Mo 0.52	Tc 1.76	Ru 0.58	Rh 10.88	Pd 0.78	Ag 4.04	Cd 115.11	In 7.58	Sn 0.21	Sb 0.30	Te 0.25	I 0.23	Xe 0.43
Cs 0.29	Ba 0.07	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.

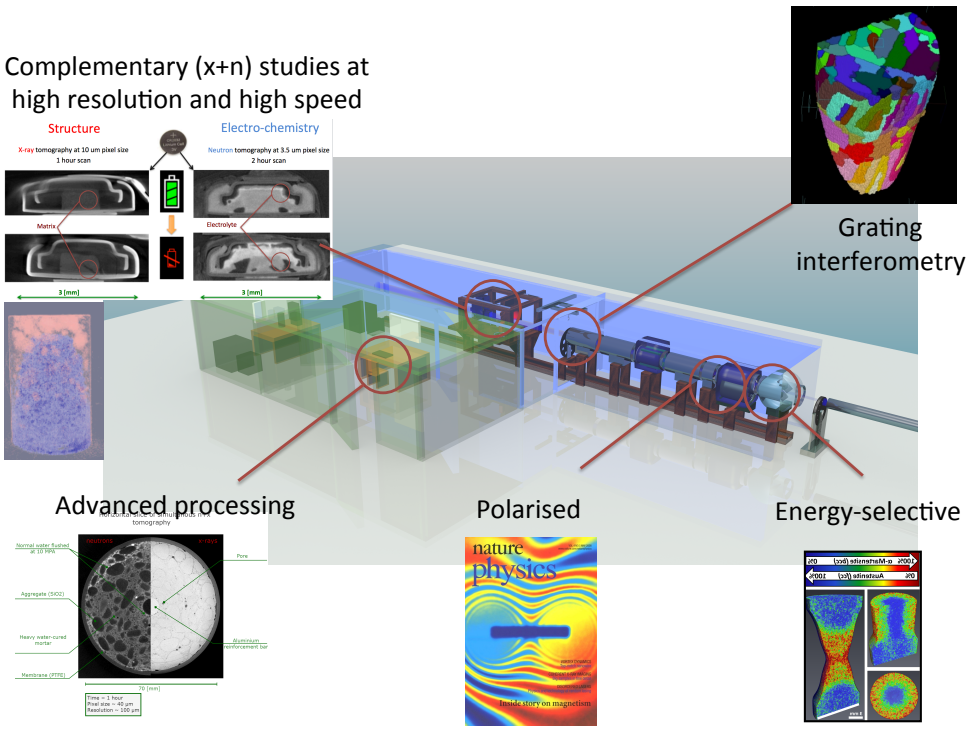
Neutrons ~~or~~ **and** X-Rays!



Neutrons ~~or~~ **and** X-Rays!



NeXT V1.0 since 2015: NeXT V 2.0 upgrade in 2022/2023



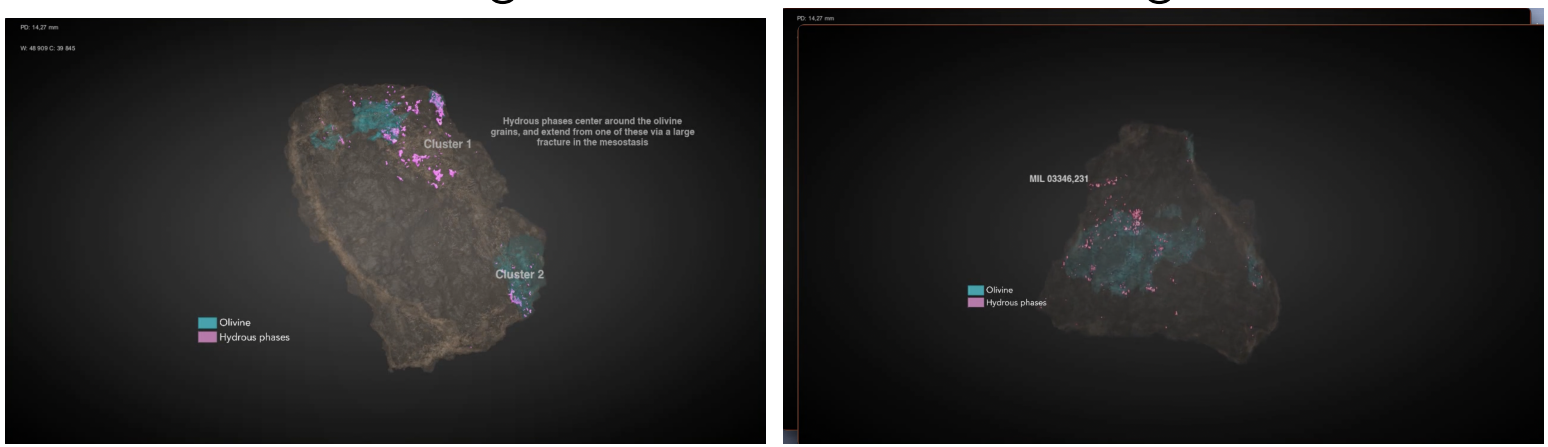
Facilities with simultaneous Neutron and X-ray Imaging:

- NeXT-Grenoble, ILL, France
- Icon beamline, PSI, Switzerland
- BT-2 beamline, (NEXT-NIST), U.S.A.

Multi-modal imaging for **segmentation**

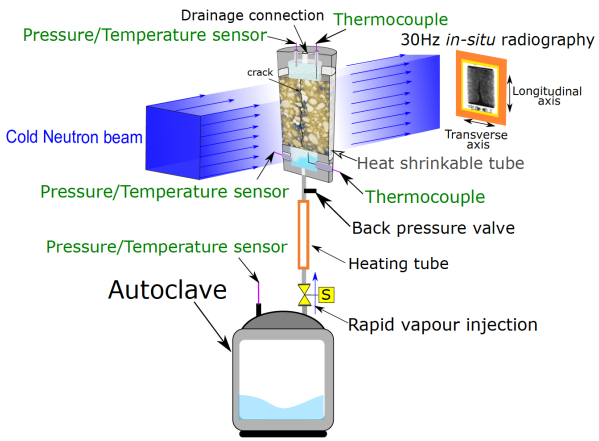
Martian Meteorite

Miller Range 03346 Mill.Range 230



Martell *et al.*, Science Advances, 2022

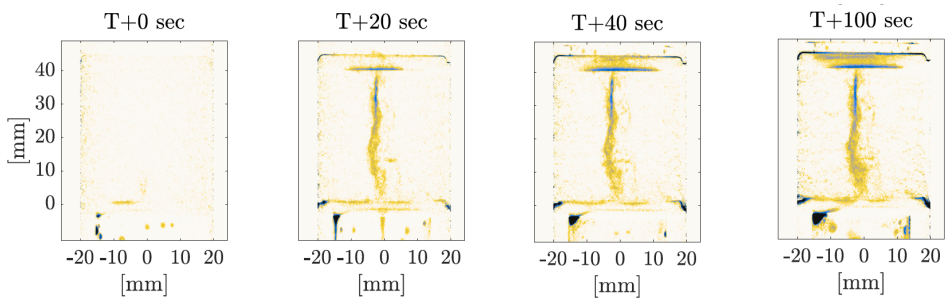
Vapour Injection



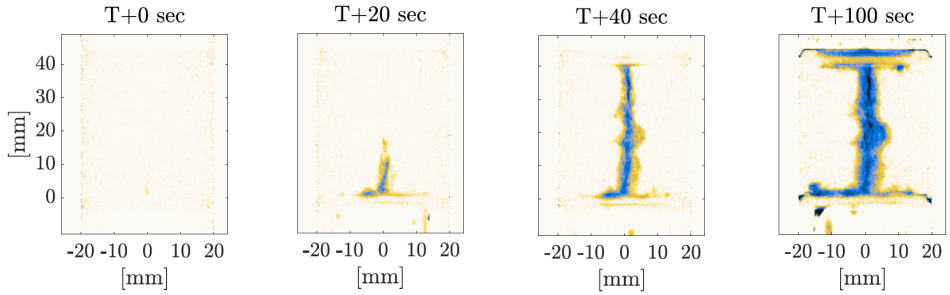
Emulating Loss of Coolant Accident in Reactor

anr

Initially saturated

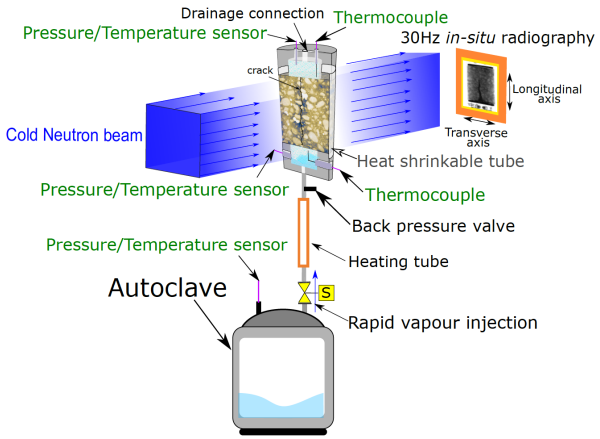


Initially dry

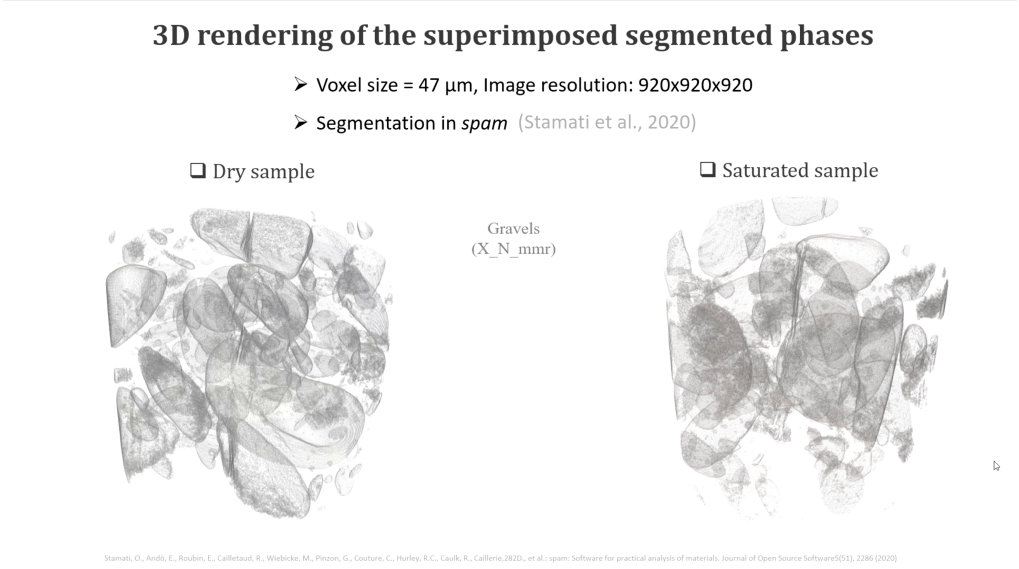


Gupta, *et al.*, Cem Concrete Research (2022)
Lukich, *et al.*, Material Letters (2021)
Nemati, *et al.*, Transport in Porous Media, (2023)

Vapour Injection

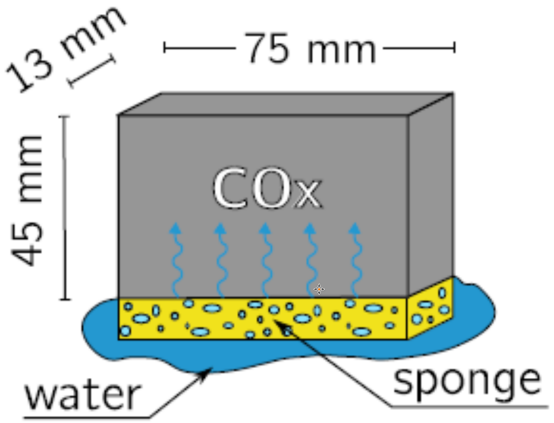


Emulating Loss of Coolant Accident in Reactor

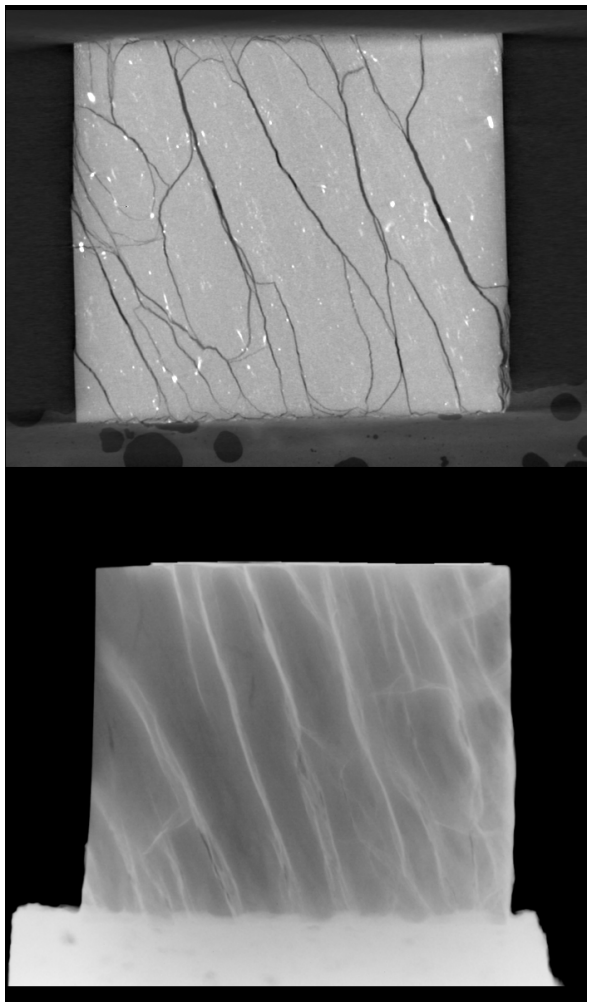


Gupta, *et al.*, Cem Concrete Research (2022)
Lukich, *et al.*, Material Letters (2021)
Nemati, *et al.*, Transport in Porous Media, (2023)

Hydo-mechanical coupling of Callovo-Oxfordian Clayrock



Rock used for long-term storage of radioactive contaminants

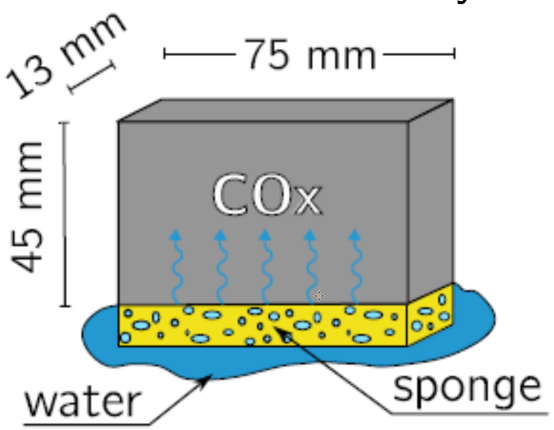


x-rays (structure)

neutrons (fluids)

Stavropoulou, *et al.*, Acta Geotechnica (2018)
Stavropoulou, *et al.*, Front. in Earth Science (2020)

Hydo-mechanical coupling of Callovo-Oxfordian Clayrock



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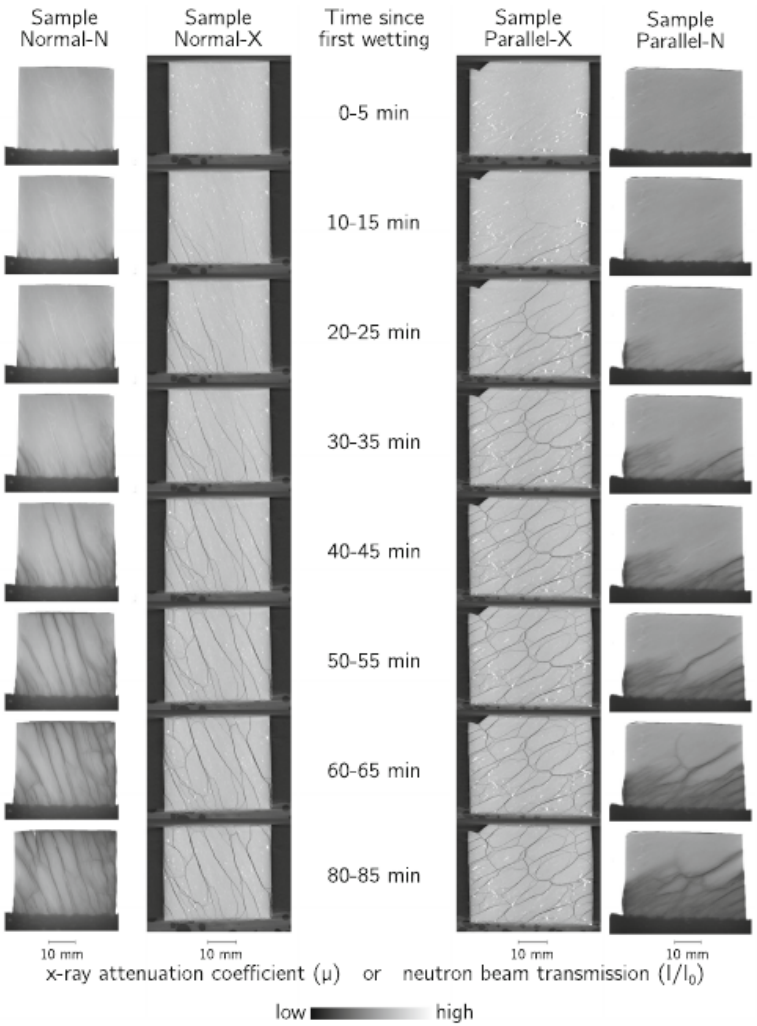
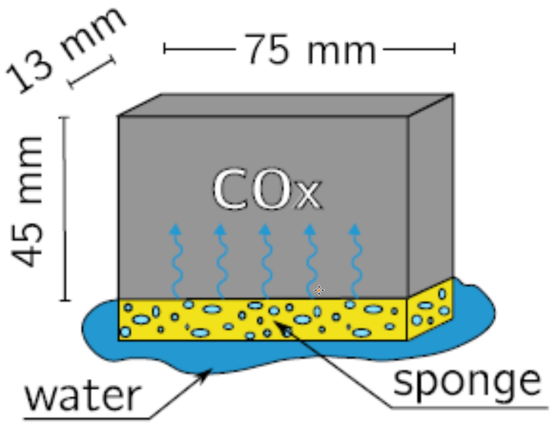


Stavropoulou, *et al.*, Acta Geotechnica (2018)
Stavropoulou, *et al.*, Front. in Earth Science (2020)

Hydo-mechanical coupling of Callovo-Oxfordian Clayrock



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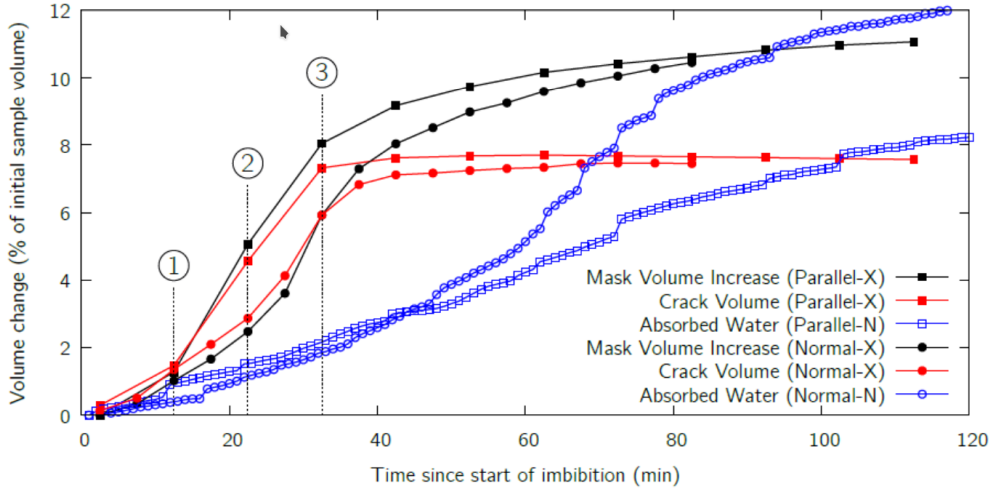
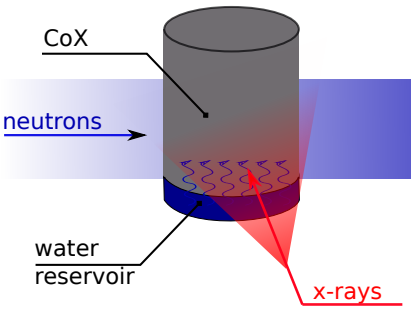
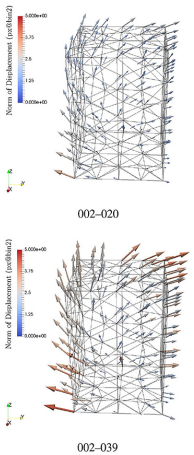


Stavropoulou, *et al.*, Acta Geotechnica (2018)
Stavropoulou, *et al.*, Front. in Earth Science (2020)

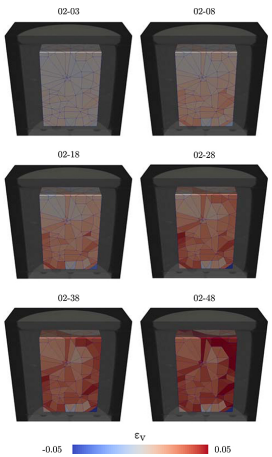
Test absorption water in COX



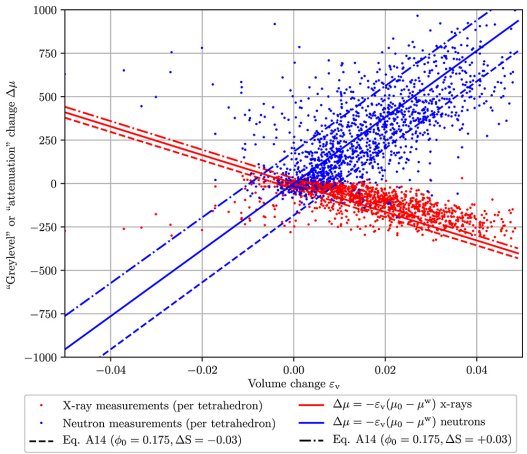
3D strain field through DVC



Correction of grayscale by deformation



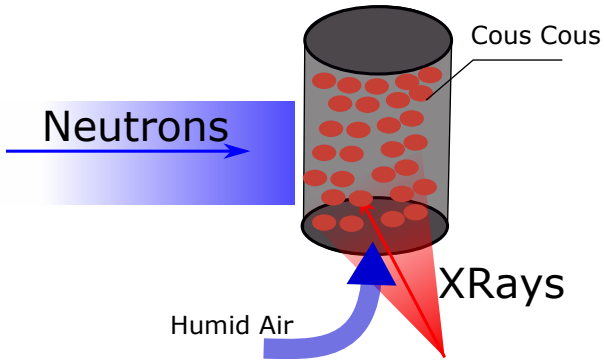
Accurate estimation water uptake



Stavropoulou, *et al.*, Front. in Earth Science (2020)

Porous media are not just geomaterials!

Water sensitive material (e.g., Medidicines, food)

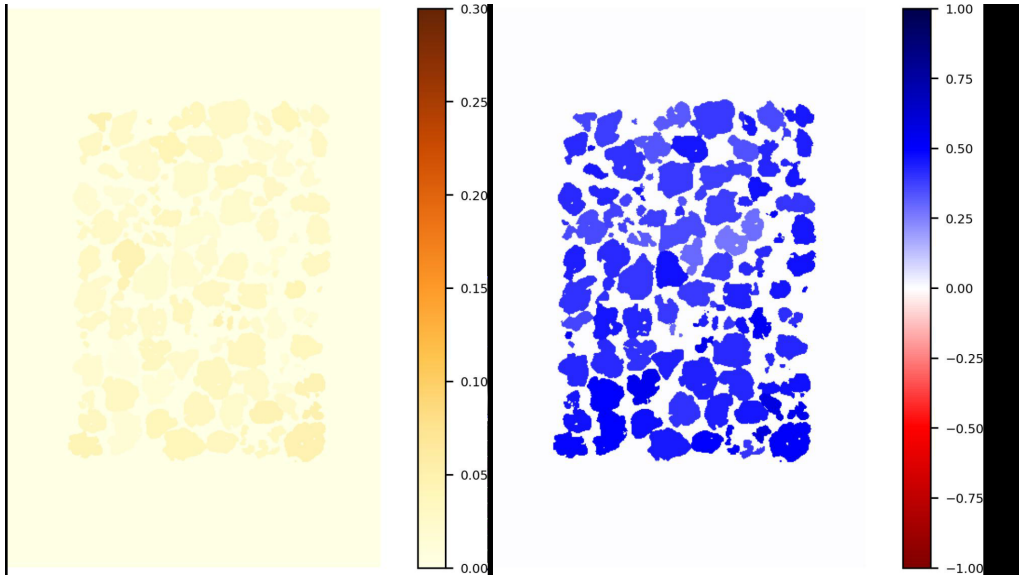


Societal problem: Caking – loss of usability of food exposed to humidity

Horizon 2020 ITN

CALIPER Unilever

Xrays(Vol Strain) Neutrons(H20 content)



Vego, Tengattini, *et al.*, Soft Matter (2022)

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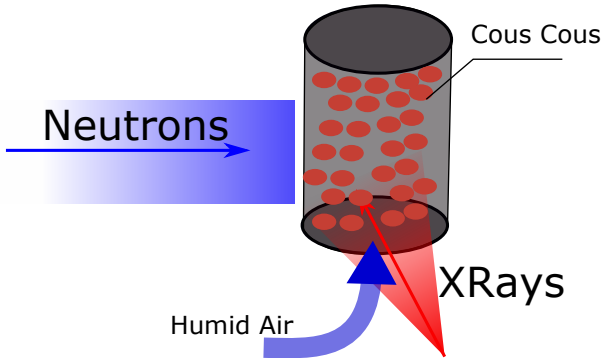
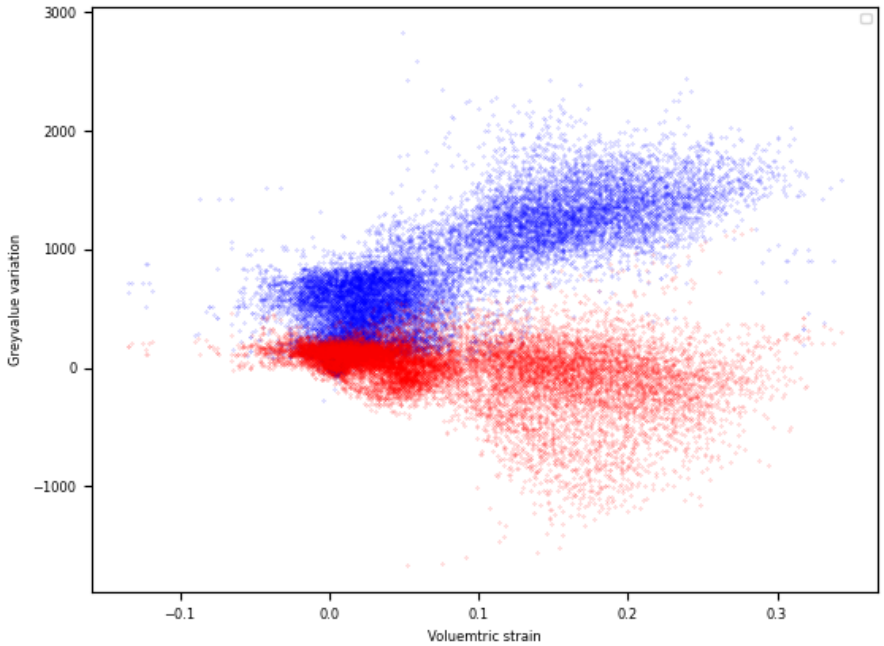


Societal problem: Caking – loss of usability of food exposed to humidity

Horizon 2020 ITN

CALIPER Unilever

Statistical correlation of Vol. Strain to H20 content



Vego, Tengattini, *et al.*, Soft Matter (2022)

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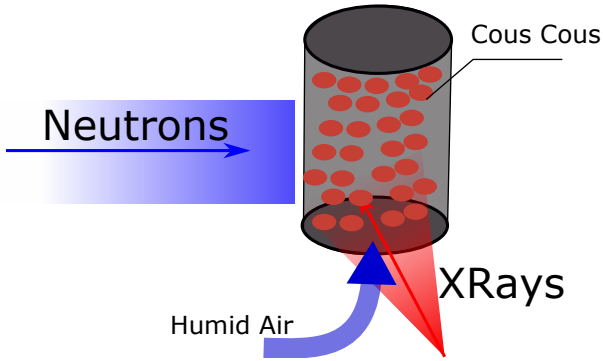
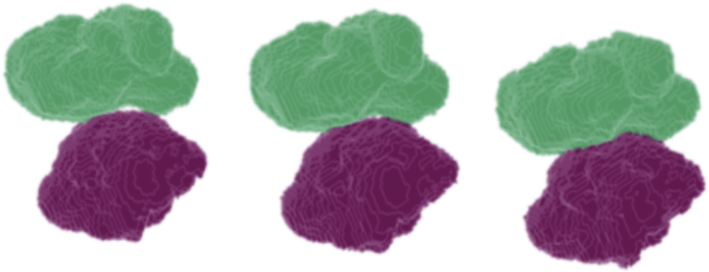


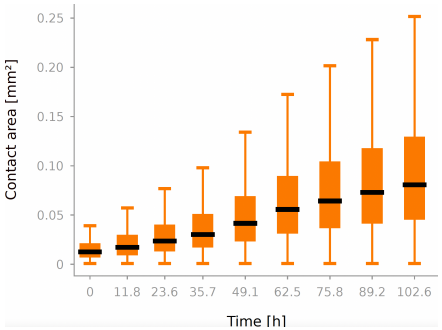
Societal problem: Caking – loss of usability of food exposed to humidity

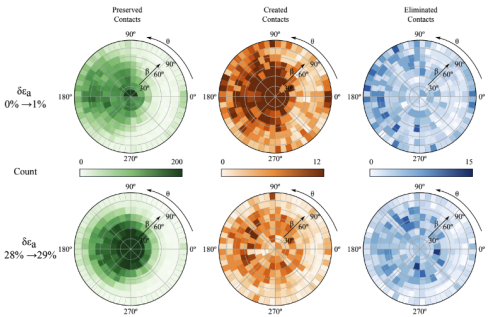
Horizon 2020 ITN

CALIPER Unilever

Contact Analysis

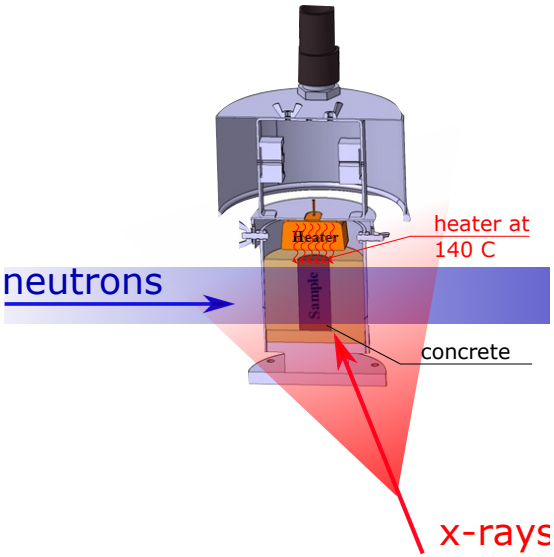




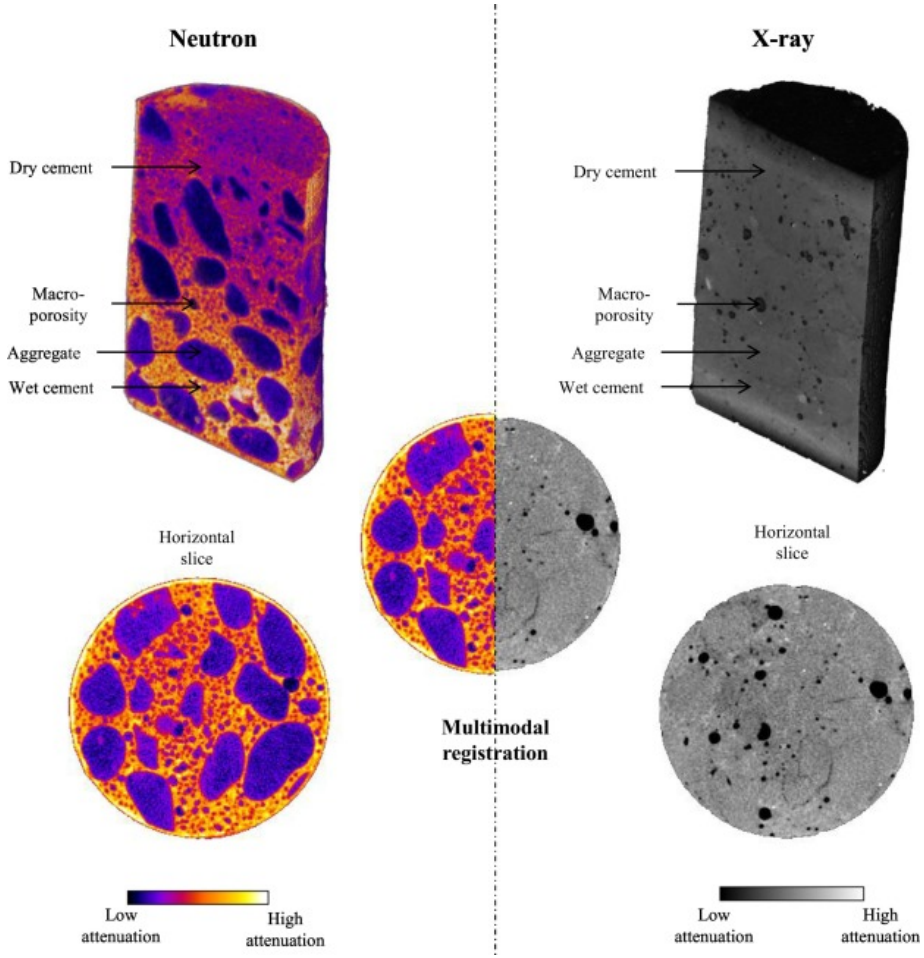


Vego, Tengattini, *et al.*, Soft Matter (2022)

Heating construction materials to high temperatures



LafargeHolcim



Tudisco *et al.*, Measurement Science and Technology (2017)
Roubin *et al.*, Cement and Concrete Composites (2019)
Sleiman, Cement and Concrete Research (2021)

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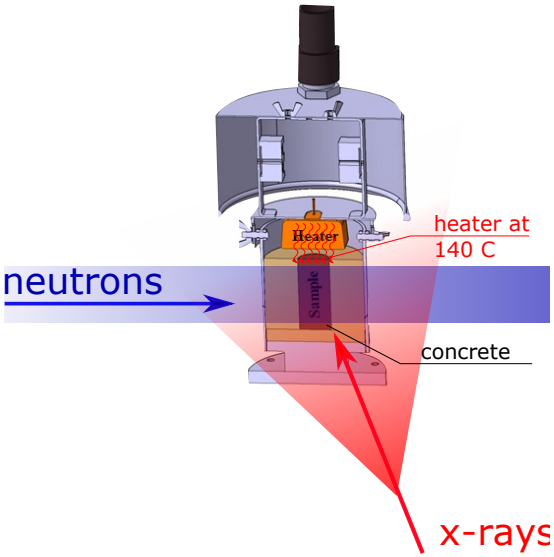
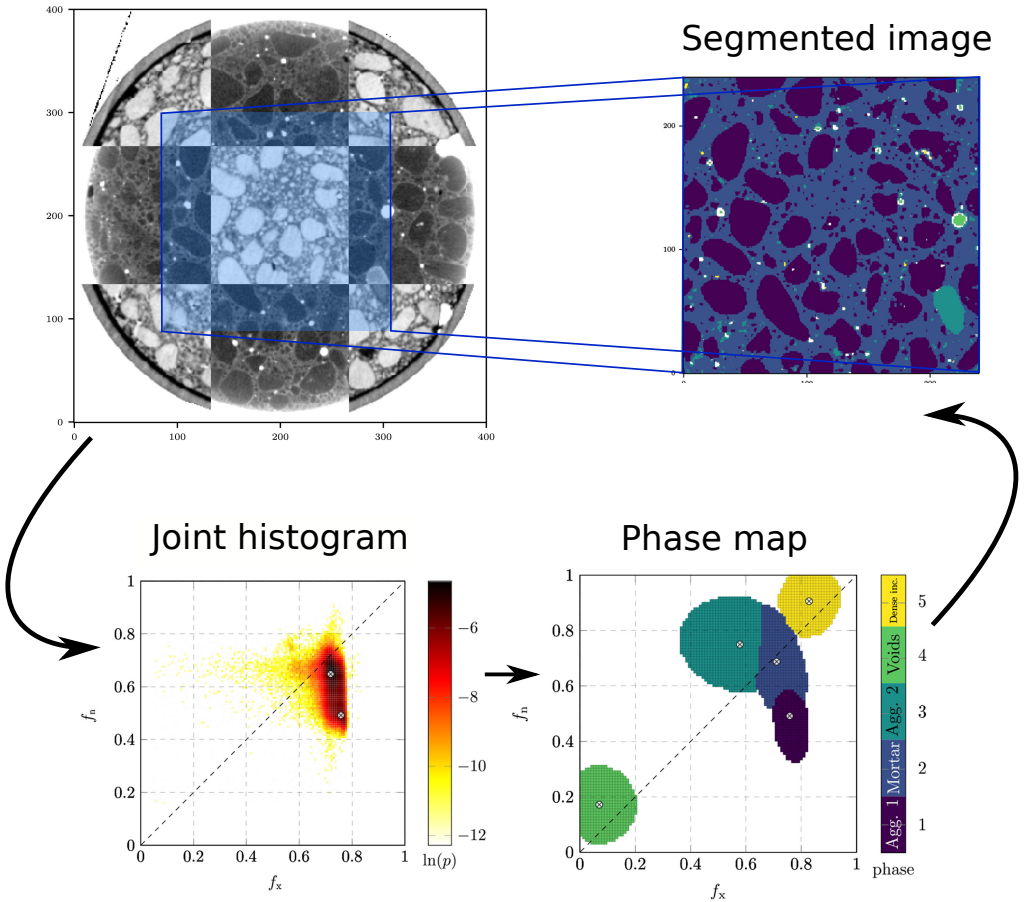
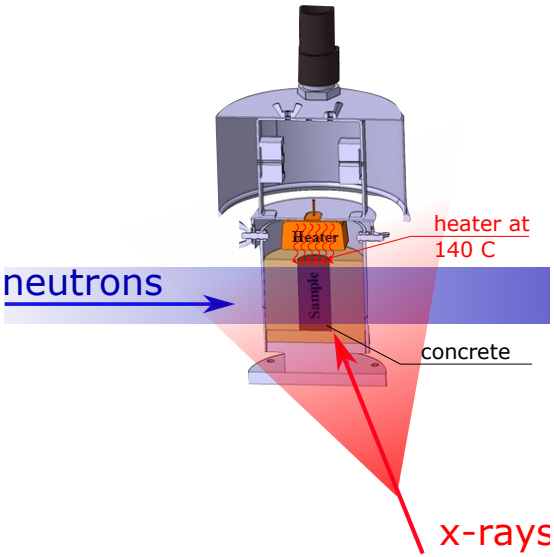


LafargeHolcim



Tudisco *et al.*, Measurement Science and Technology (2017)
Roubin *et al.*, Cement and Concrete Composites (2019)
Sleiman, Cement and Concrete Research (2021)

Heating construction materials to high temperatures



LafargeHolcim

spam
ractical analysis

Stamati *et al.*, 2020

Tudisco *et al.*, Measurement Science and Technology (2017)
Roubin *et al.*, Cement and Concrete Composites (2019)
Sleiman, Cement and Concrete Research (2021)

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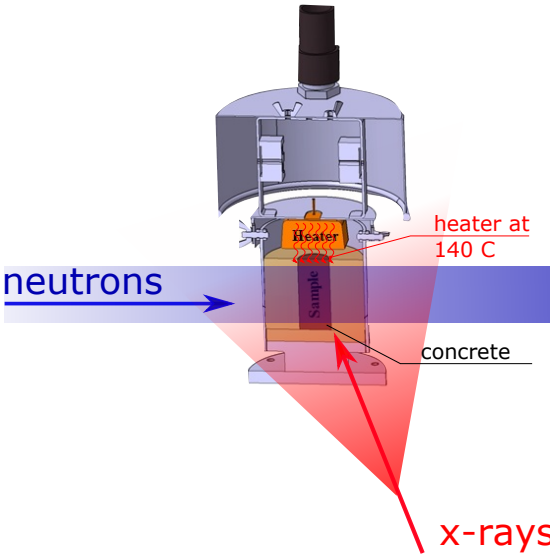
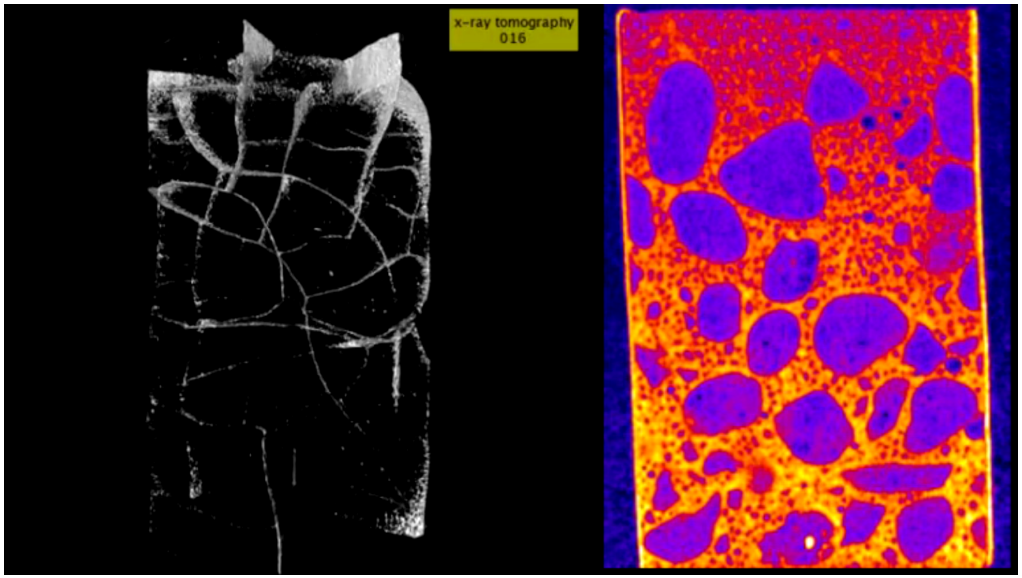


LafargeHolcim

x-ray neutrons



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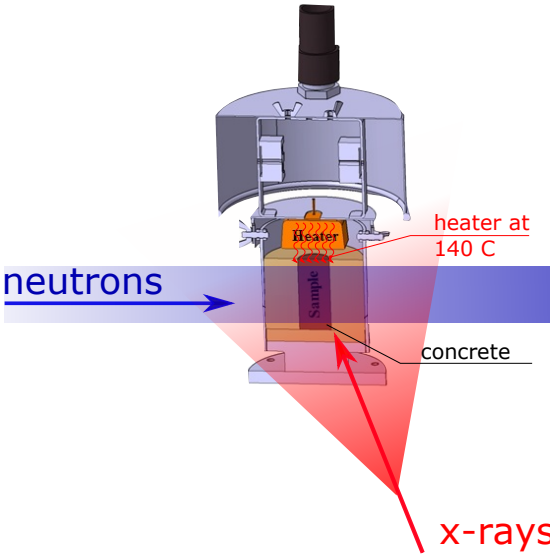
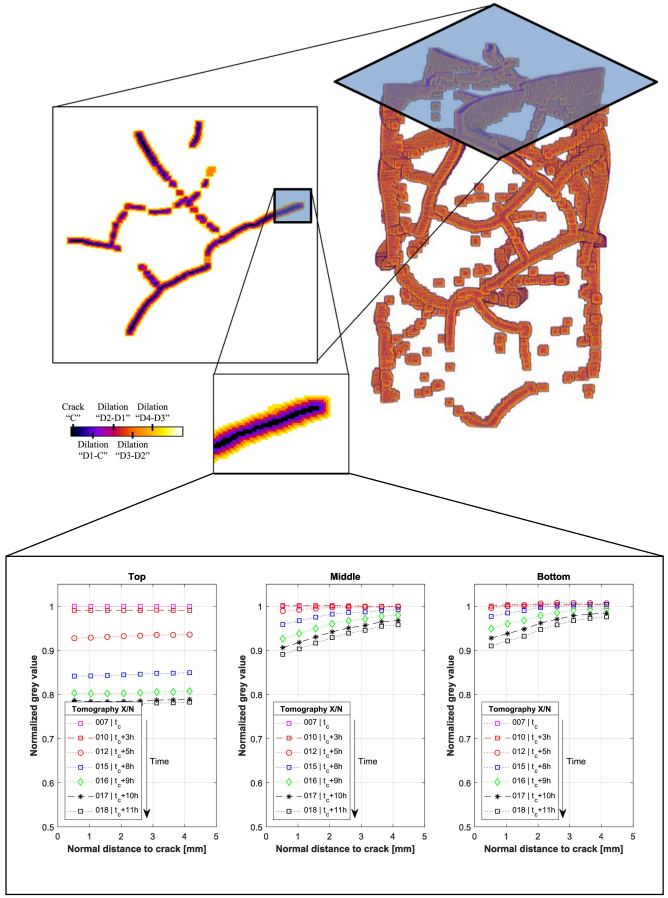
Heating construction materials to high temperatures

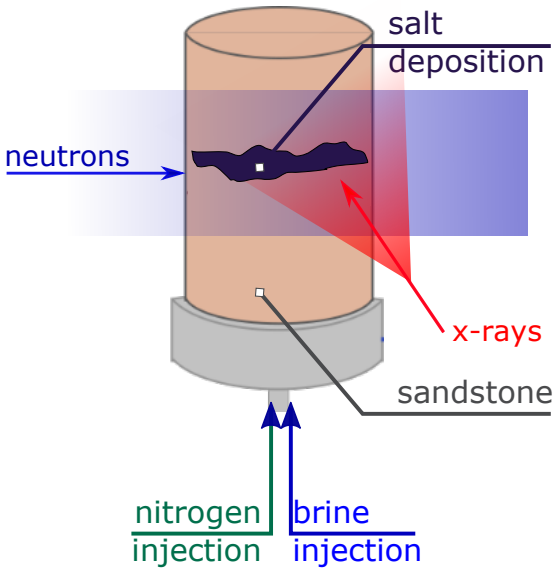


LafargeHolcim



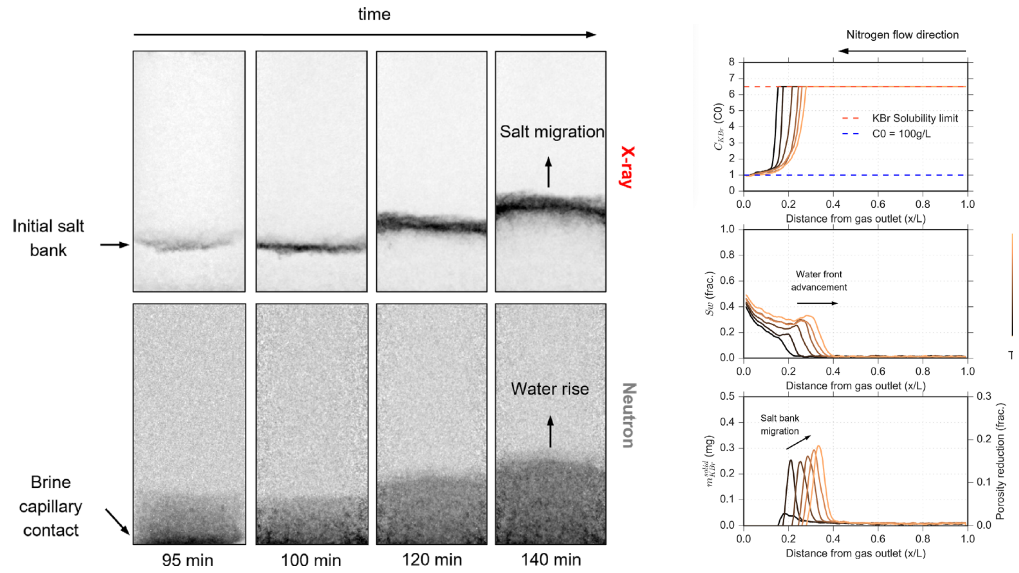
Tudisco *et al.*, Measurement Science and Technology (2017)
Roubin *et al.*, Cement and Concrete Composites (2019)
Sleiman, Cement and Concrete Research (2021)

Salt Precipitation



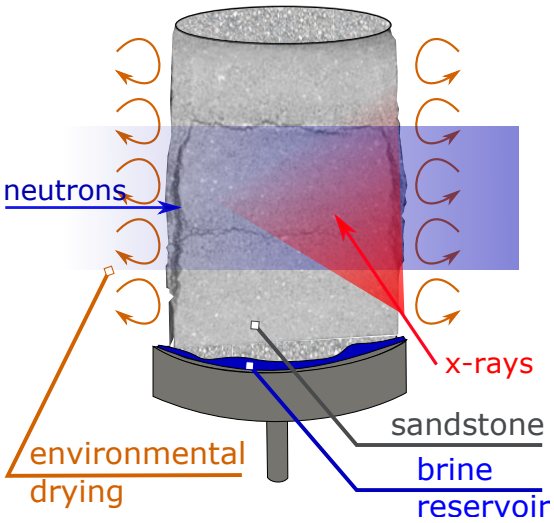
From X-ray: Salt accumulation

From neutron: evolution hydraulic properties



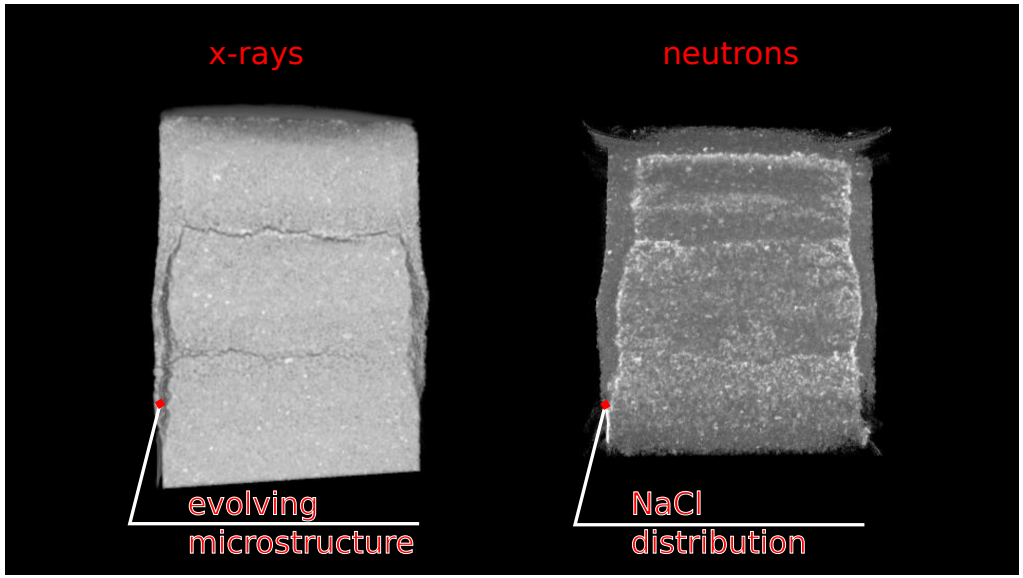
Mascle *et Al.*, Science and Technology for Energy Transition (2023)

Haloclasty: weathering by growth of salt crystals



Neutrons see Salts (Cl in NaCl highly visible)

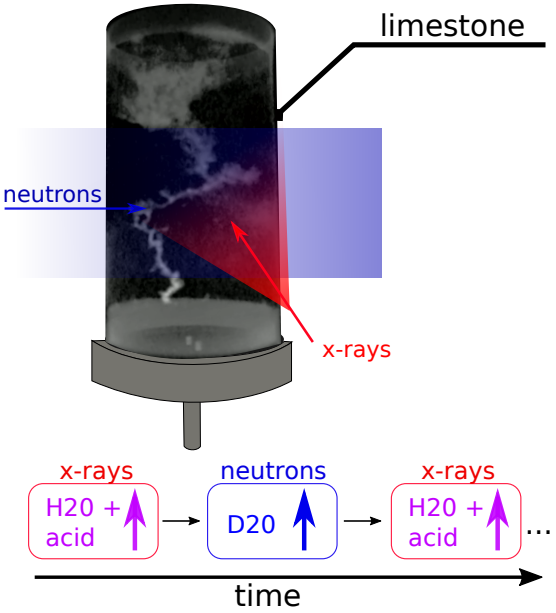
X-rays see microstructure



Okumiko V., Gregoire, D. *et al.*, Ezponda, "Feder funds" Manuscript under redaction

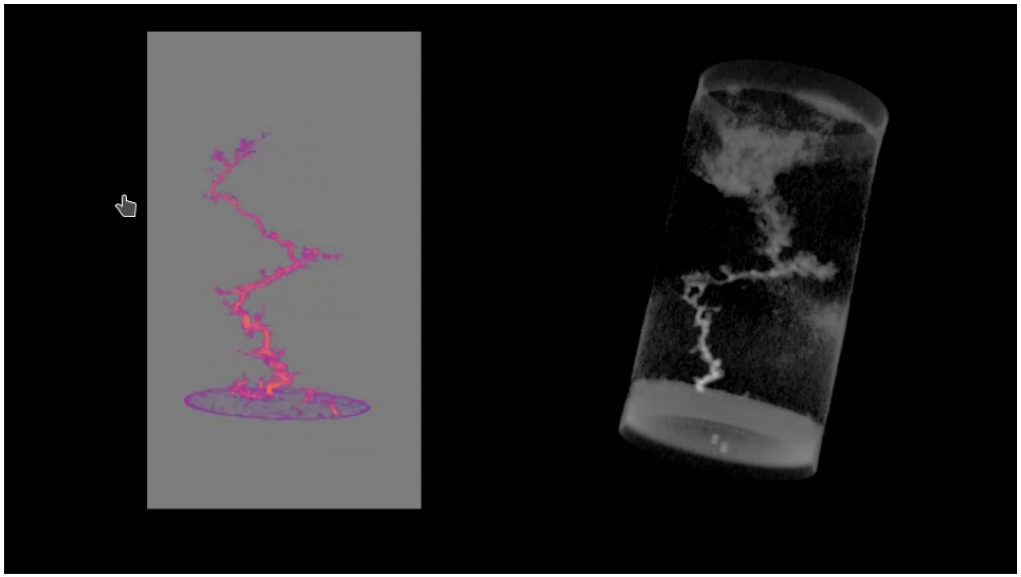
Ezponda

Wormhole Growth in Dissolving Limestones



From X-ray: wormhole development by acid dissolution

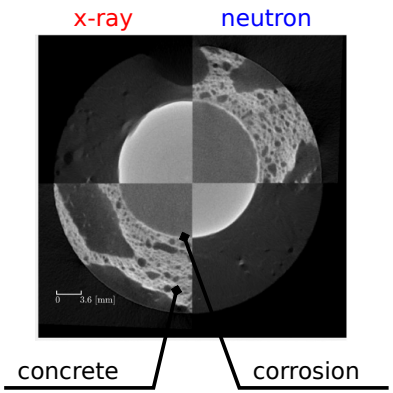
From neutron: evolution hydraulic propertie

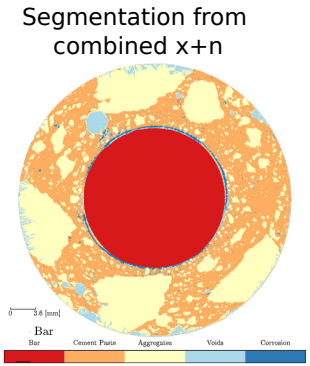


Szymczak *et Al.*, EGU (2021)
Szymczak *et Al.*, AGU (2020)
Cooper *et Al.*, Advances in Water Resources (2023)

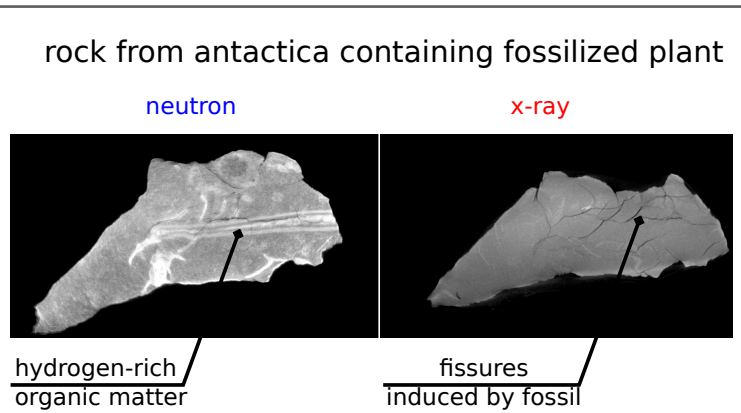
Other applications

The combination of neutron and x-rays allow for advanced segmentation and image processing [1-3]



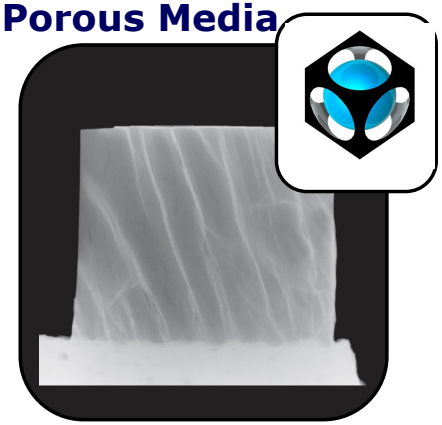


Neutron and x-ray imaging are highlty complementary *e.g.*, to identify organic material



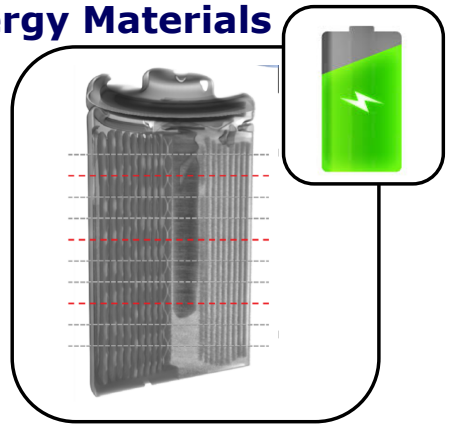
[1]Robuschi et *Al.*, Cement and Concrete Research (2021)
[2]Tudisco et *Al.*, Measurement Science and Technology (2017)
[3]Roubin et *Al.*, Cement and Concrete Composites (2019)

Porous Media



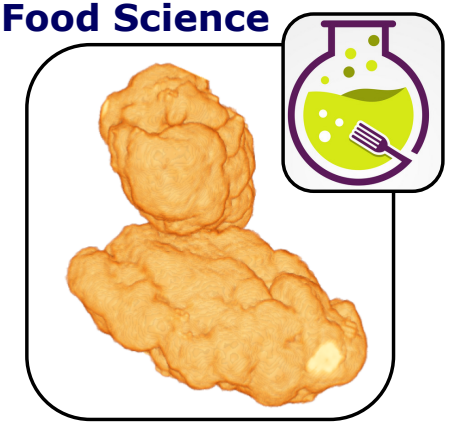
Lewis, et al., Transport in Porous Media (2023)
Gupta et al., Cement and Concrete Research (2022)
Sleiman et al., Cement and Concrete Research (2022)
...

Energy Materials



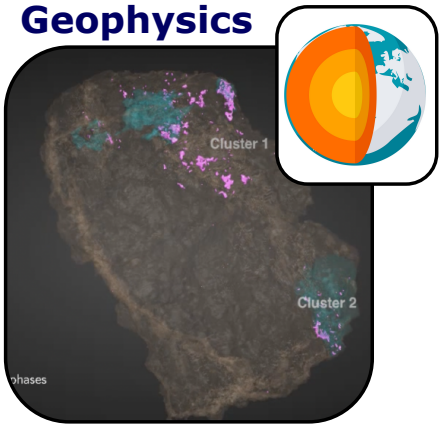
Bradbury et al., Advanced Functional Materials (2023)
Magnier, et al. Frontiers in Energy Research (2022)
Ziesche, et al., Nat Comm (2020)
...

Food Science



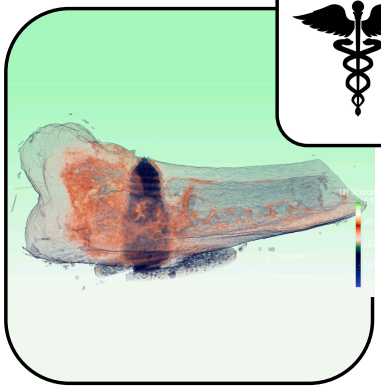
Vego, et al., Soft Matter (2022)
Vego, et al., Food Structure (2023)
Vego, et al., Granular Matter (2023)
...

Geophysics



Martell, et al., Science Advances (2022)
Cordonnier et al, Frontiers in earth science (2020)
...

Medical sciences



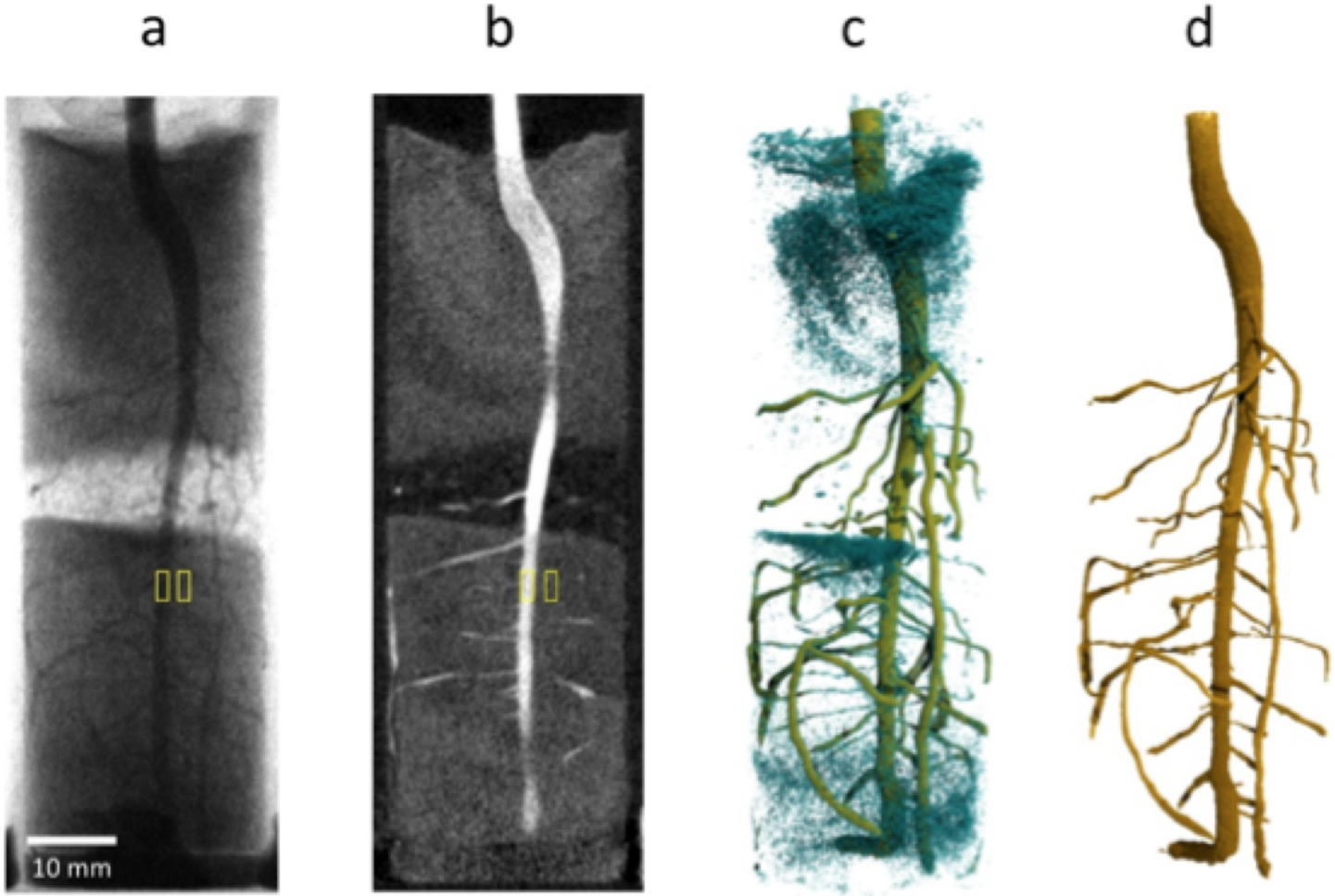
Tornquist, et al., Fontiers in bioengineering (2022)
Lecann, et al. Physics in Medicine & Biology (2021)

...

Archeology

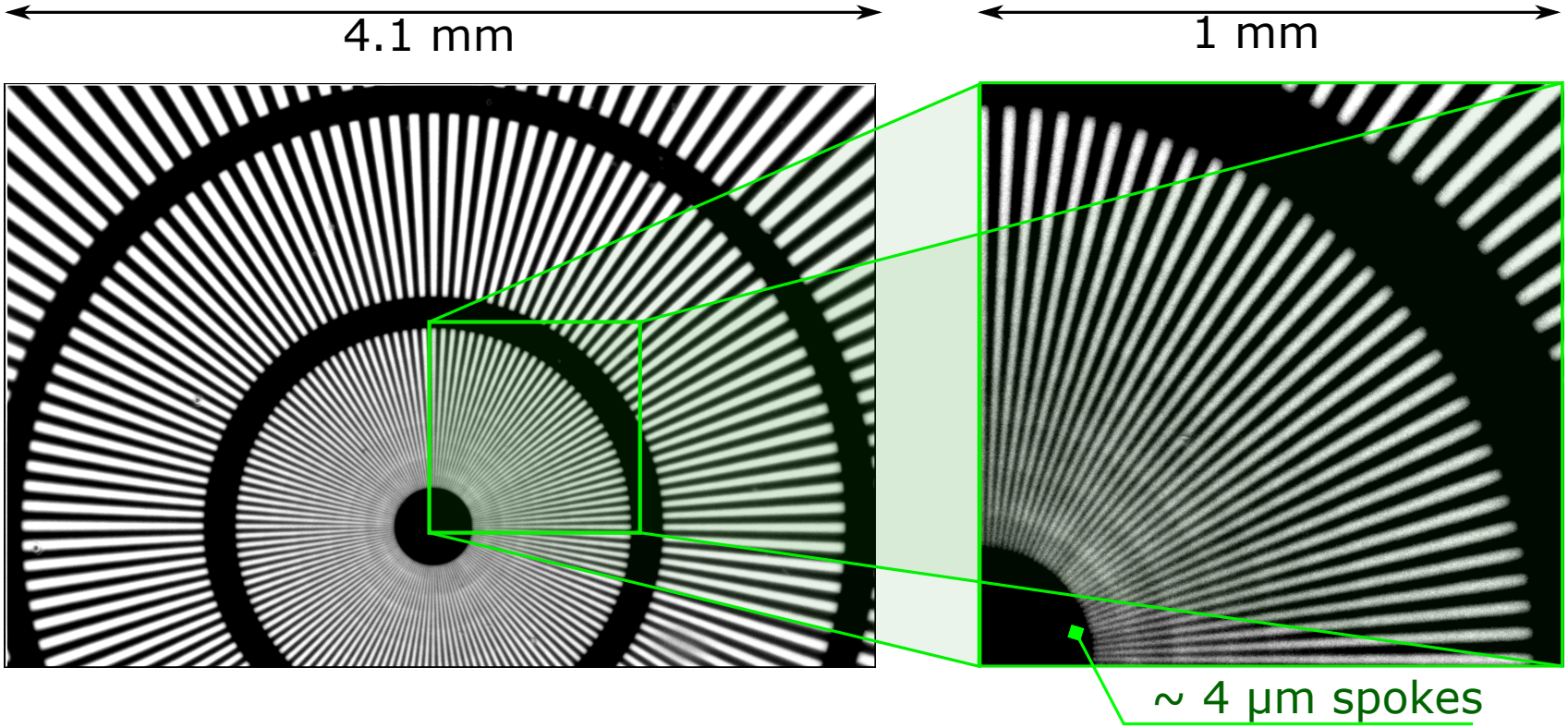
And much more!

temporal resolution: 1.5s tomographies



Totzke *et al.*, 2019 Optics Express

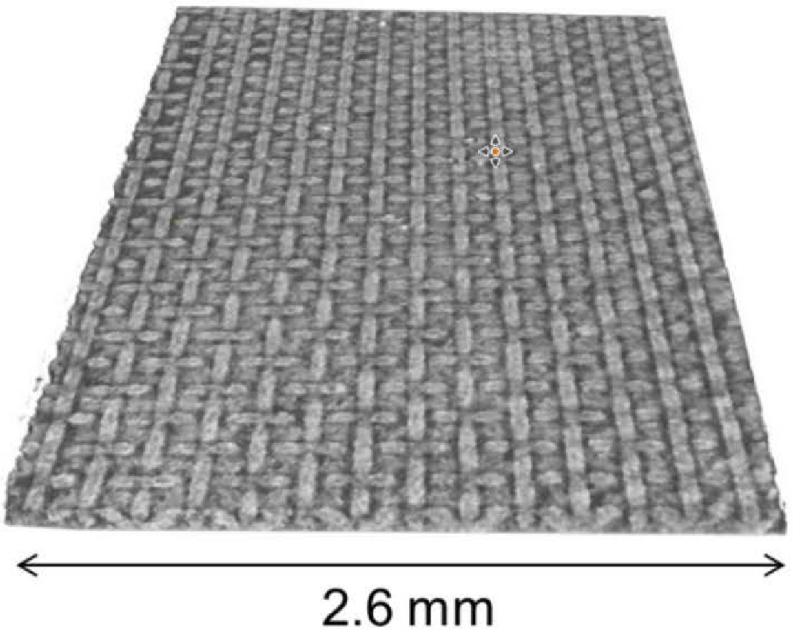
spatial resolution: $\leq$ 3 μm resolution (pixel 0.75 um)



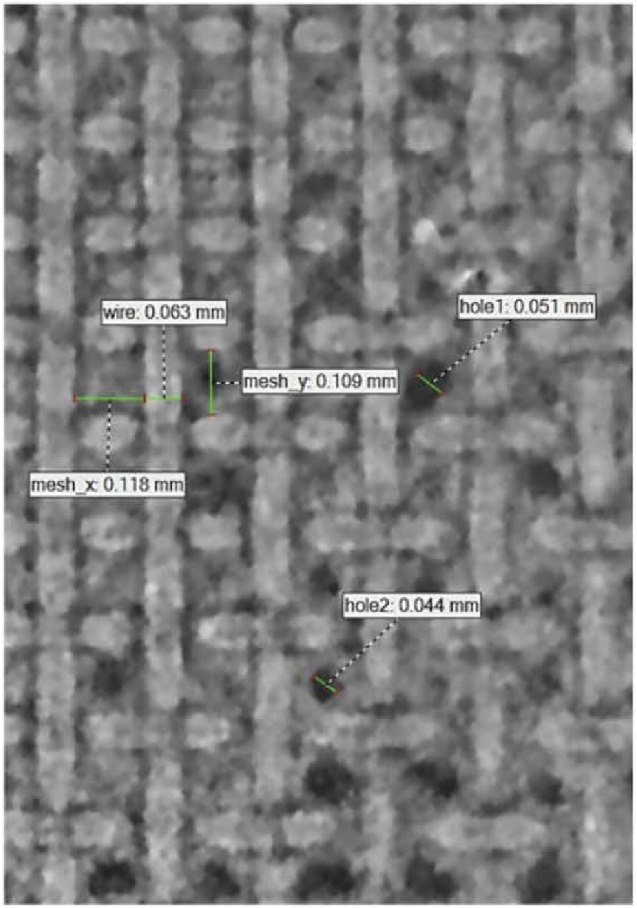
Tengattini, Kardjilov, Helfen *et al.*, 2022 Optics Express

spatial resolution: $\leq$ 3 μm resolution (pixel size 1.5μm)

a)

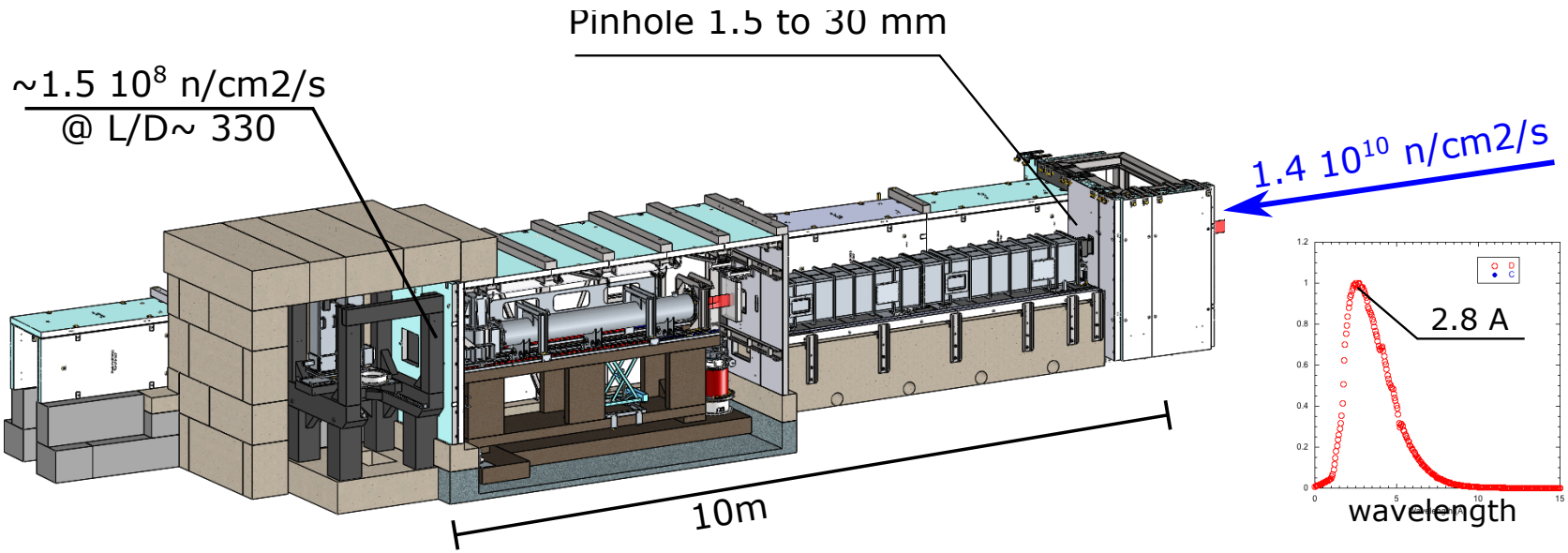


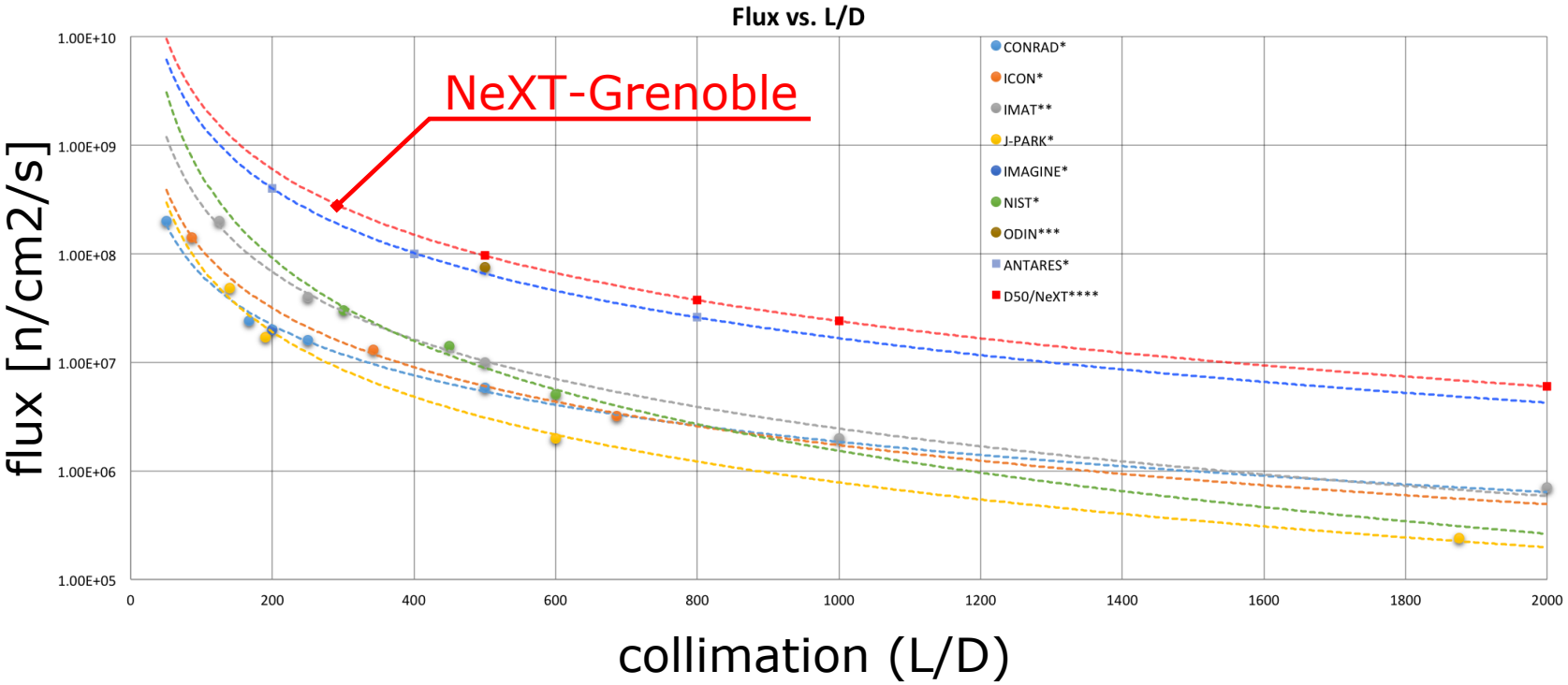
b)

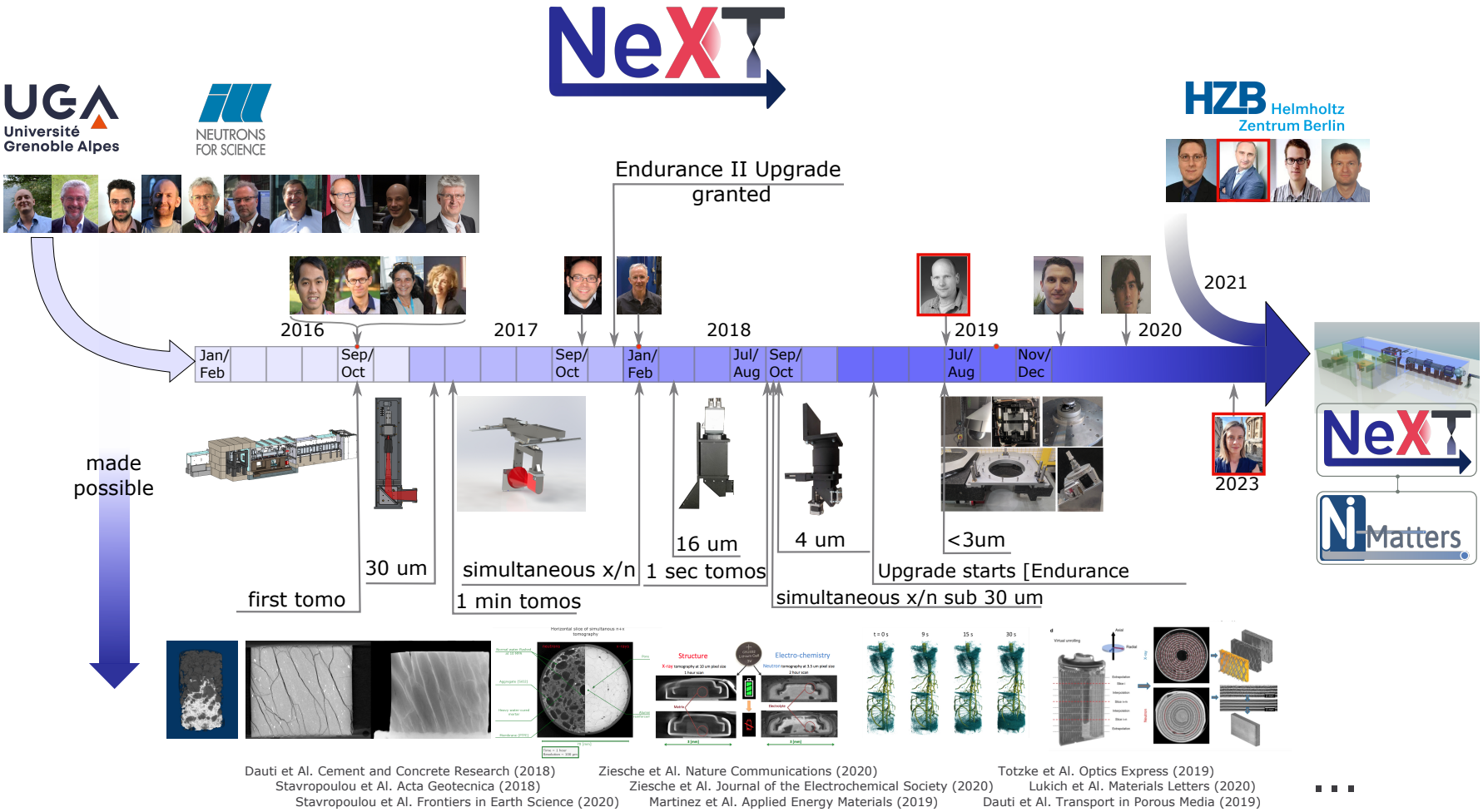


$\sim$10h tomo Tengattini, *et al.*, 2022 Optics Express

The old instrument (NeXT V.1.0)/ D50Tomo




Flux vs. L/D
NeXT-Grenoble
flux [n/cm2/s]
1.00E+10
1.00E+09
1.00E+08
1.00E+07
1.00E+06
1.00E+05
0
200
400
600
800
1000
1200
1400
1600
1800
2000
collimation (L/D)
CONRAD*
ICON*
IMAT**
J-PARK*
IMAGINE*
NIST*
ODIN***
ANTARES*
D50/NeXT****



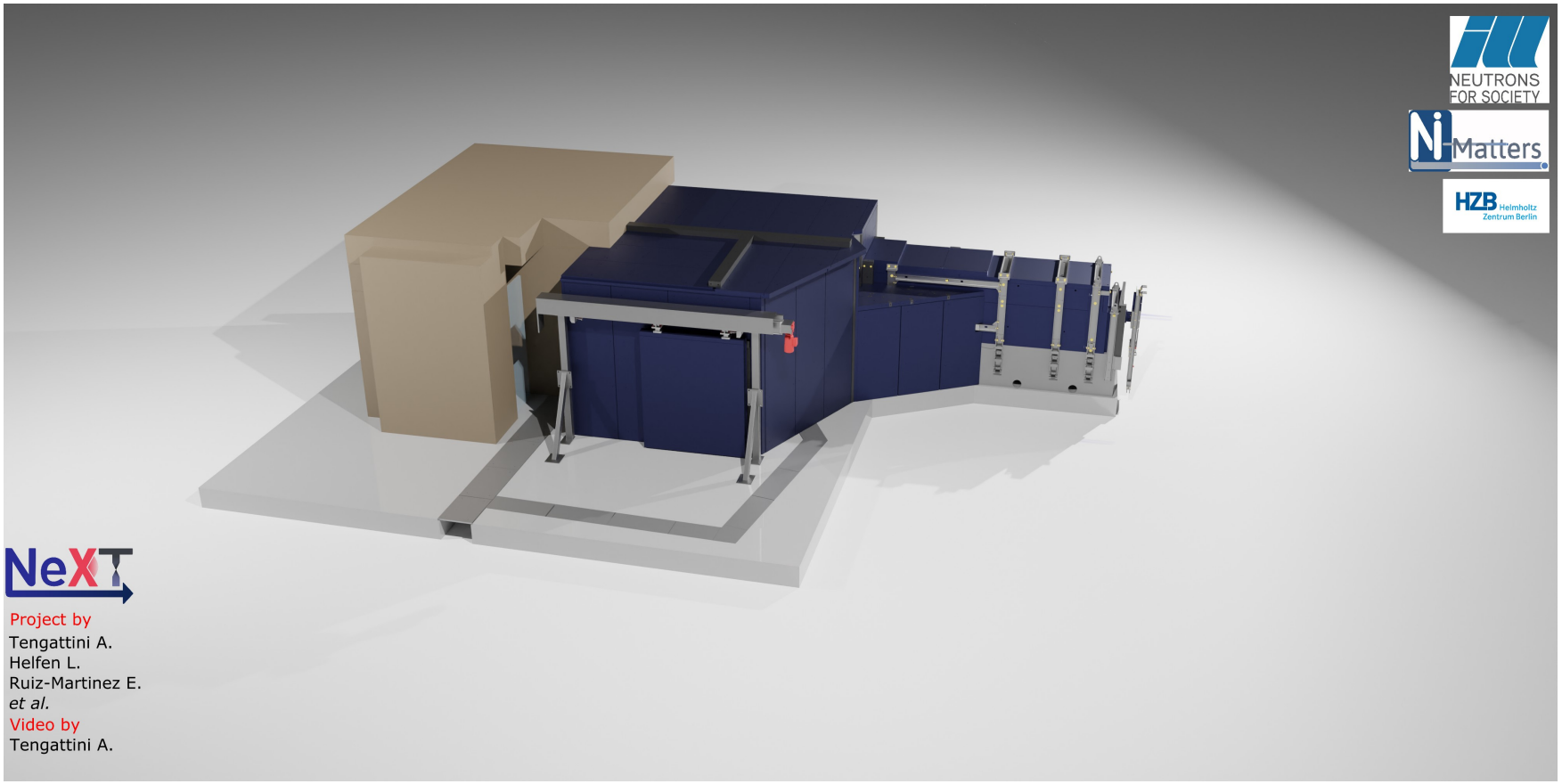
Dauti et Al. Cement and Concrete Research (2018)
Stavropoulou et Al. Acta Geotecnica (2018)
Stavropoulou et Al. Frontiers in Earth Science (2020)

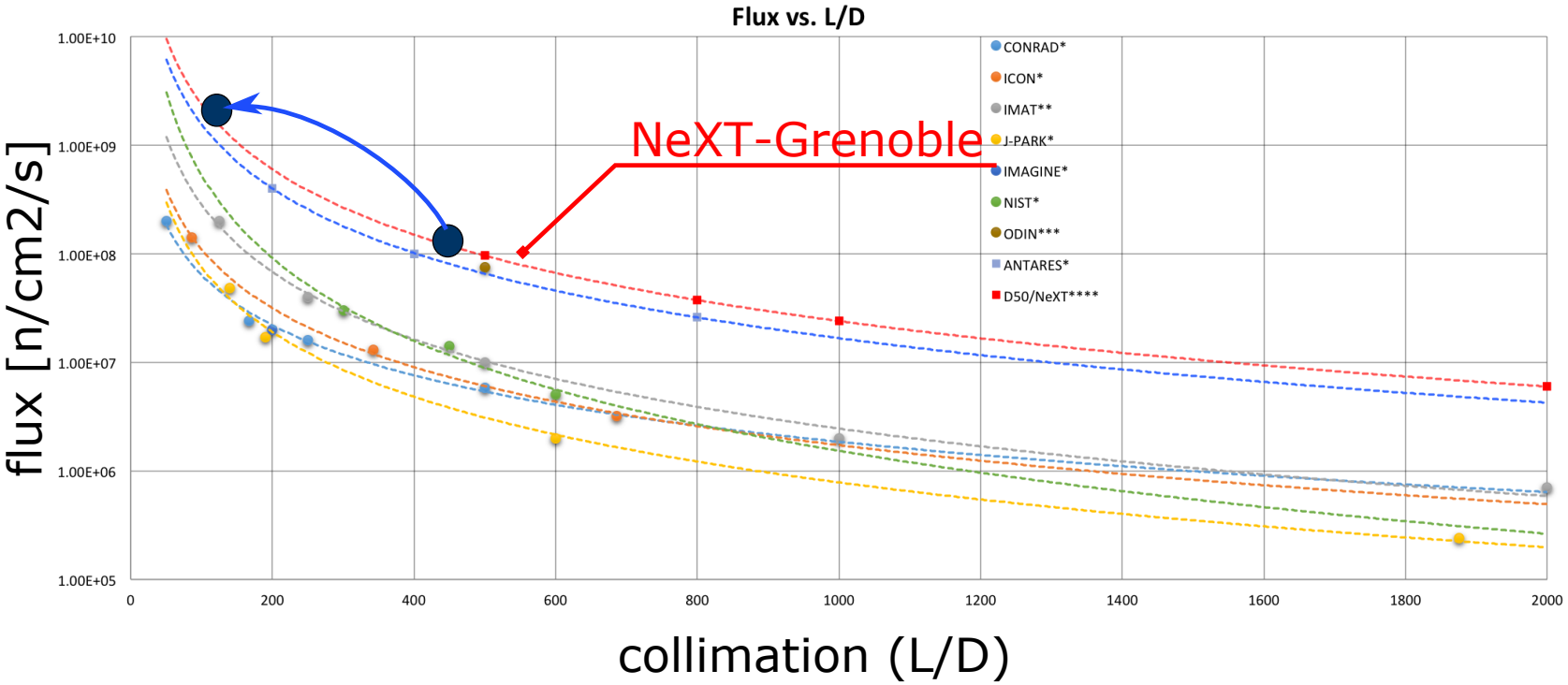
Ziesche et Al. Nature Communications (2020)
Ziesche et Al. Journal of the Electrochemical Society (2020)
Martinez et Al. Applied Energy Materials (2019)

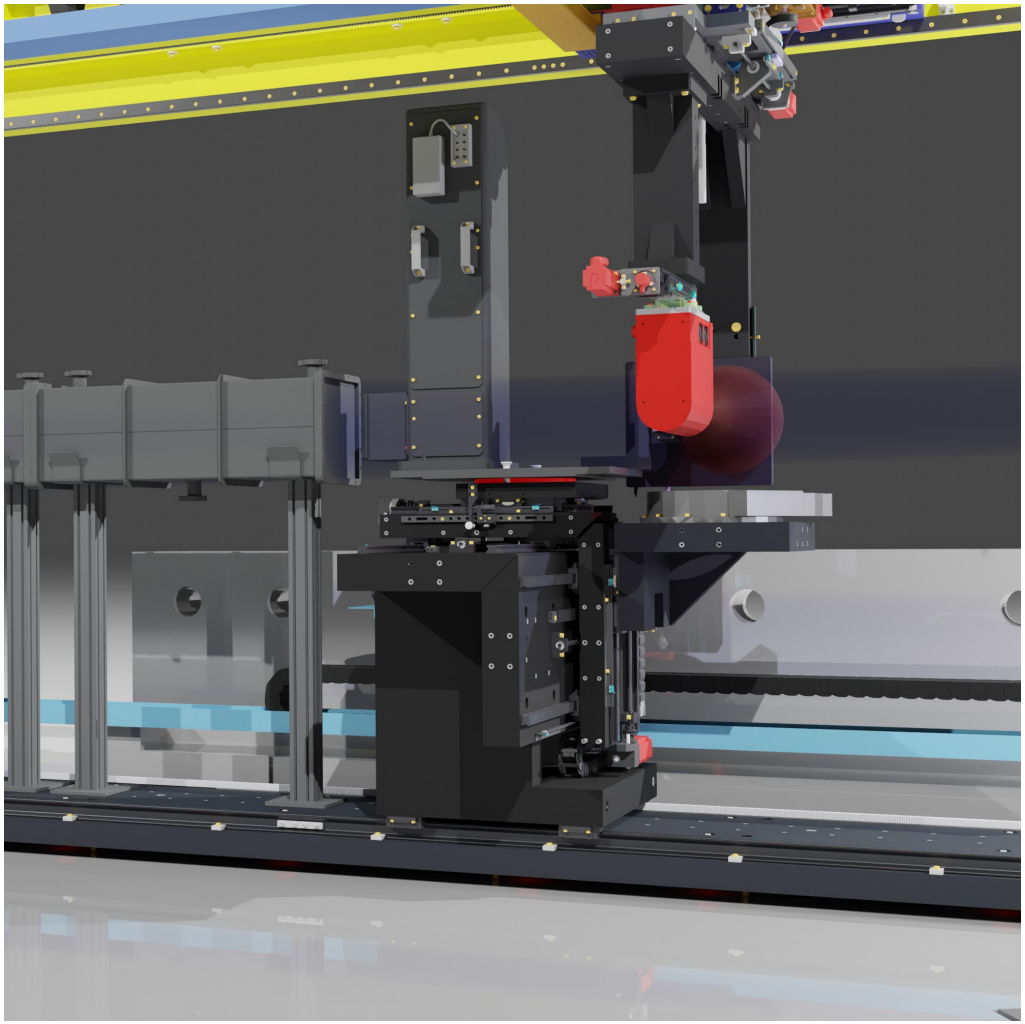
Totzke et Al. Optics Express (2019)
Lukich et Al. Materials Letters (2020)
Dauti et Al. Transport in Porous Media (2019)

...

NeXT 2.0


NEUTRONS FOR SOCIETY
Ni-Matters
HZB Helmholtz Zentrum Berlin
NeXT
Project by
Tengattini A.
Helfen L.
Ruiz-Martinez E.
et al.
Video by
Tengattini A.

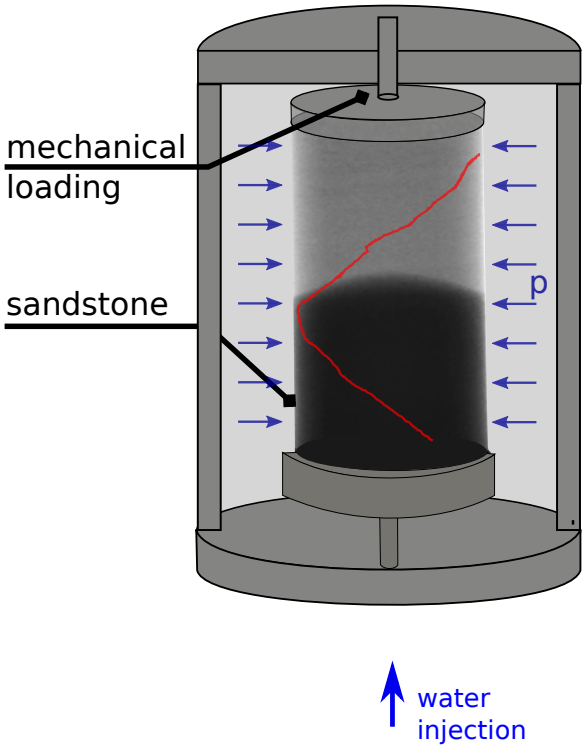

Flux vs. L/D
flux [n/cm2/s]
1.00E+10
1.00E+09
1.00E+08
1.00E+07
1.00E+06
1.00E+05
0
200
400
600
800
1000
1200
1400
1600
1800
2000
collimation (L/D)
NeXT-Grenoble
CONRAD*
ICON*
IMAT**
J-PARK*
IMAGINE*
NIST*
ODIN***
ANTARES*
D50/NeXT****



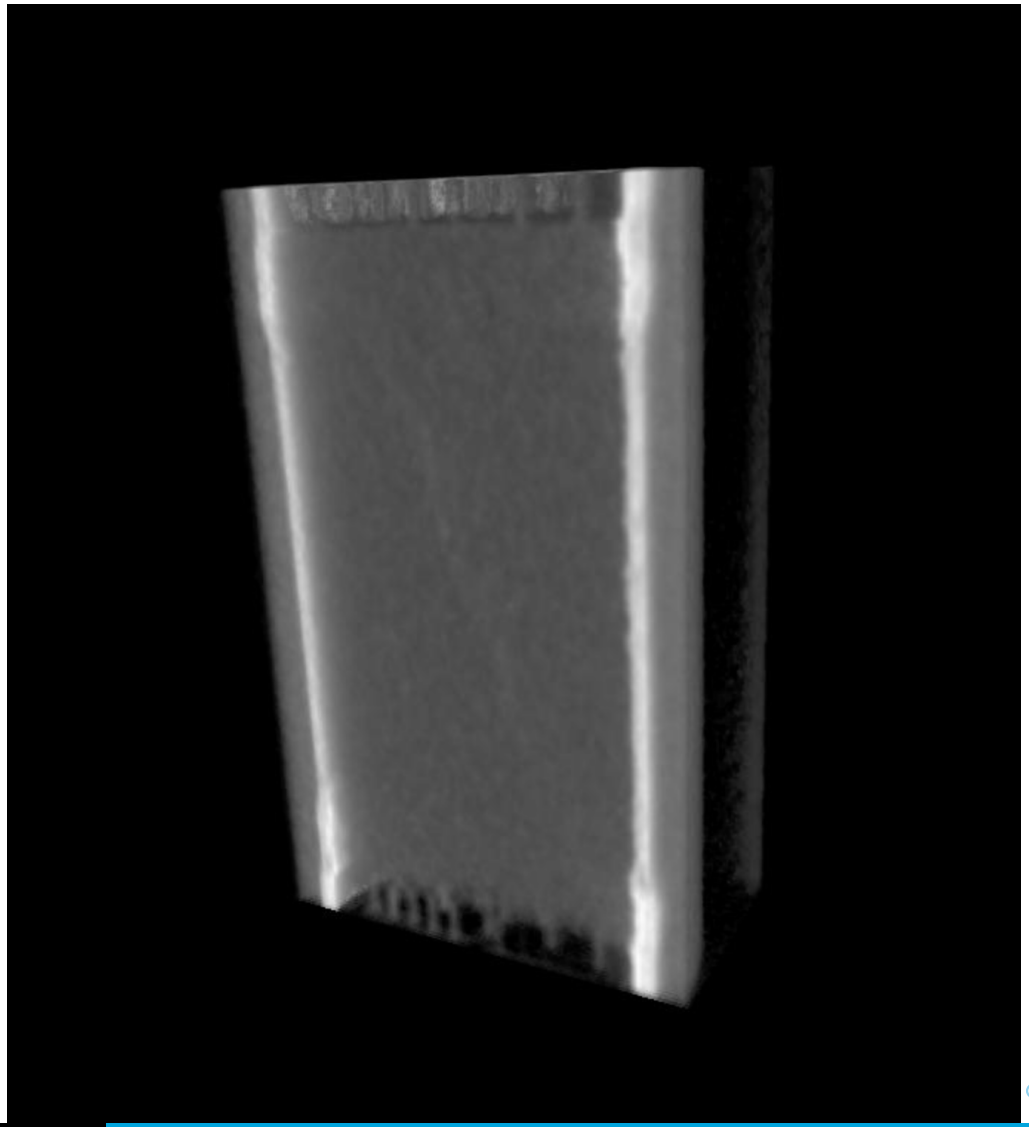
Improved tomographic station

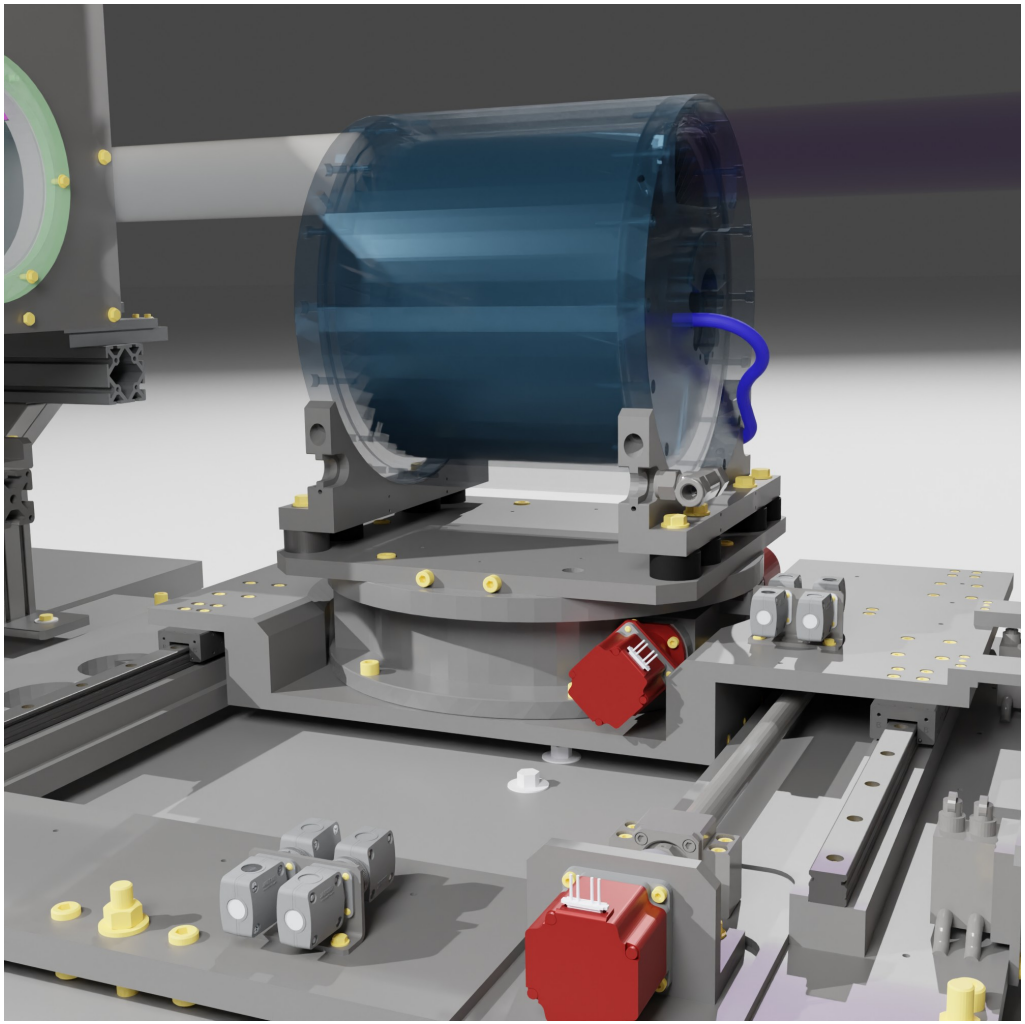
Adjustable collimation (L/D),
Higher flux
More x-ray options, 150kV $\rightarrow$ 300kV
More space for operando test
Expanded detector suite
...

Fluid flow within a damaged rock

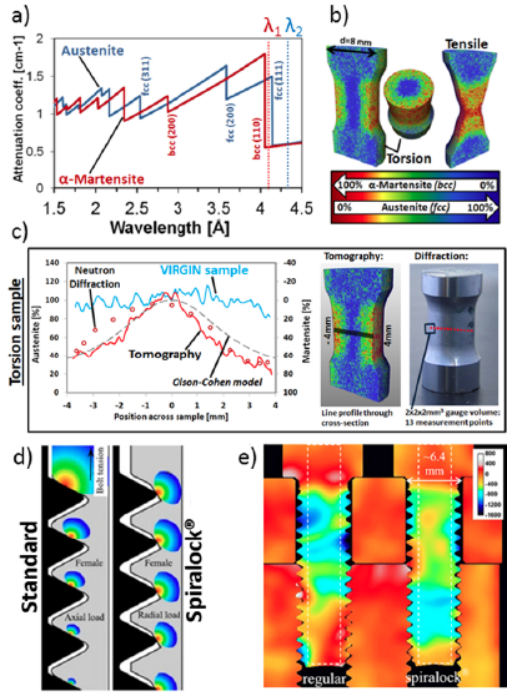


Tests in June 2023, 30 second tomographies

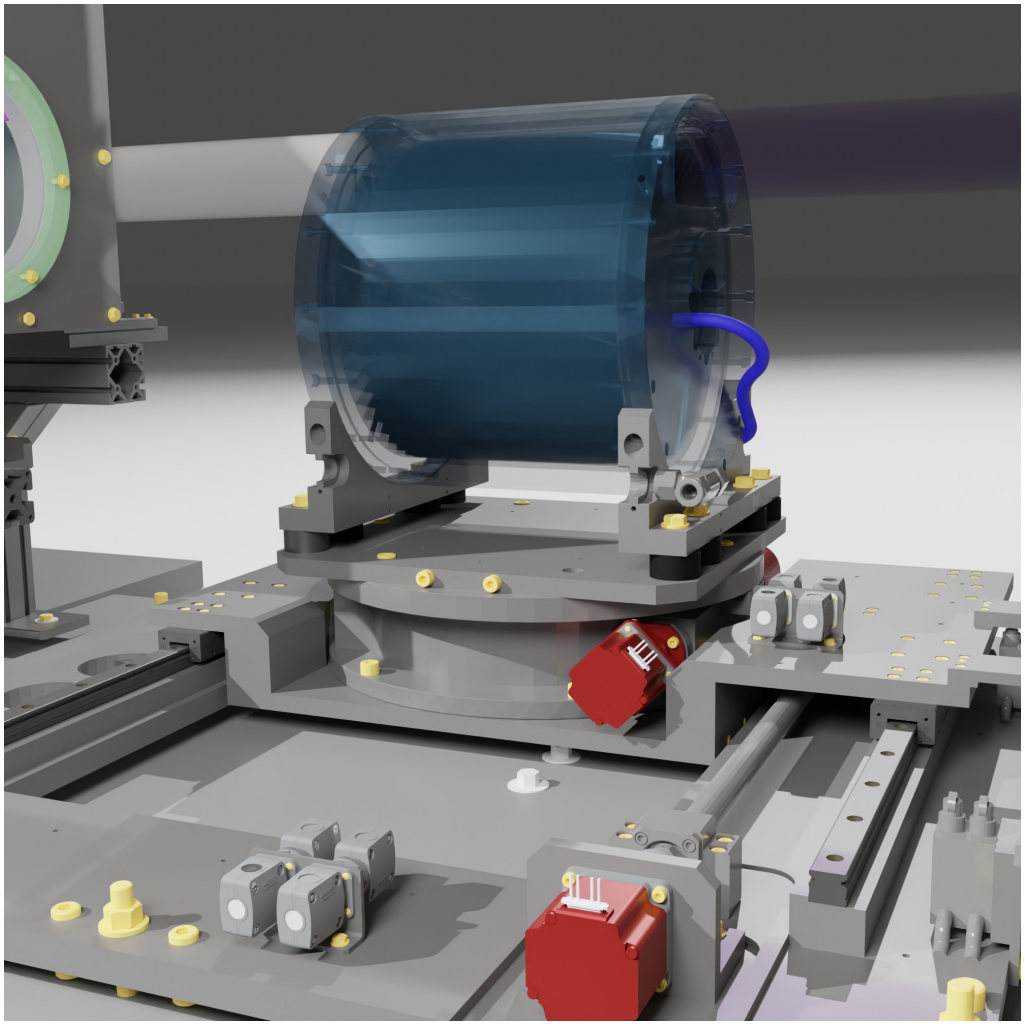




Monochromatic imaging options



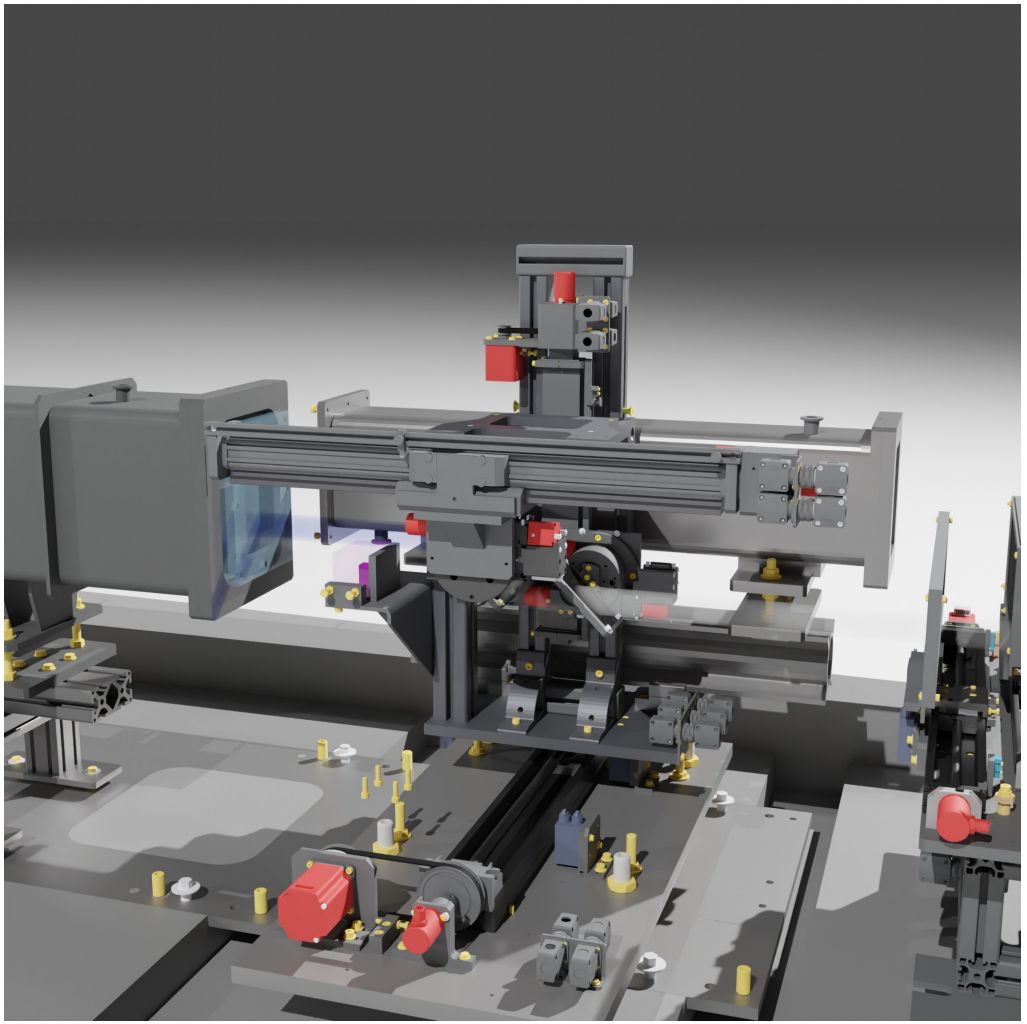
Dabah, E., et al., Journal of Materials Science (2016)



Monochromatic imaging options

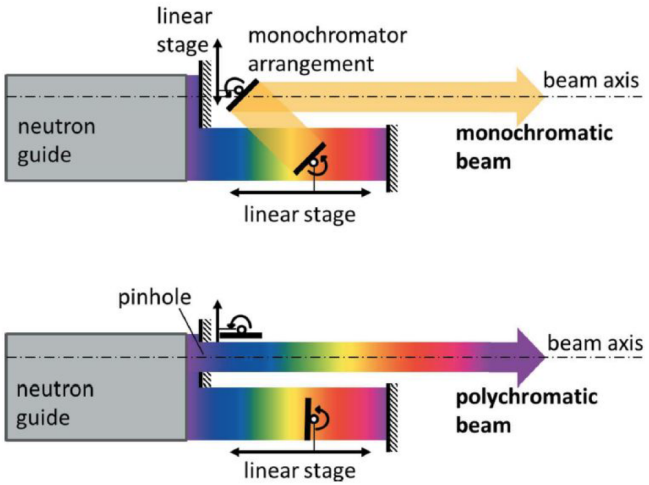
Velocity selector

- Wider wavelength $\Delta\lambda/\lambda \sim 15 - 20\%$
- Standard Astrium (now Airbus), 2.5 to 40 $\AA$

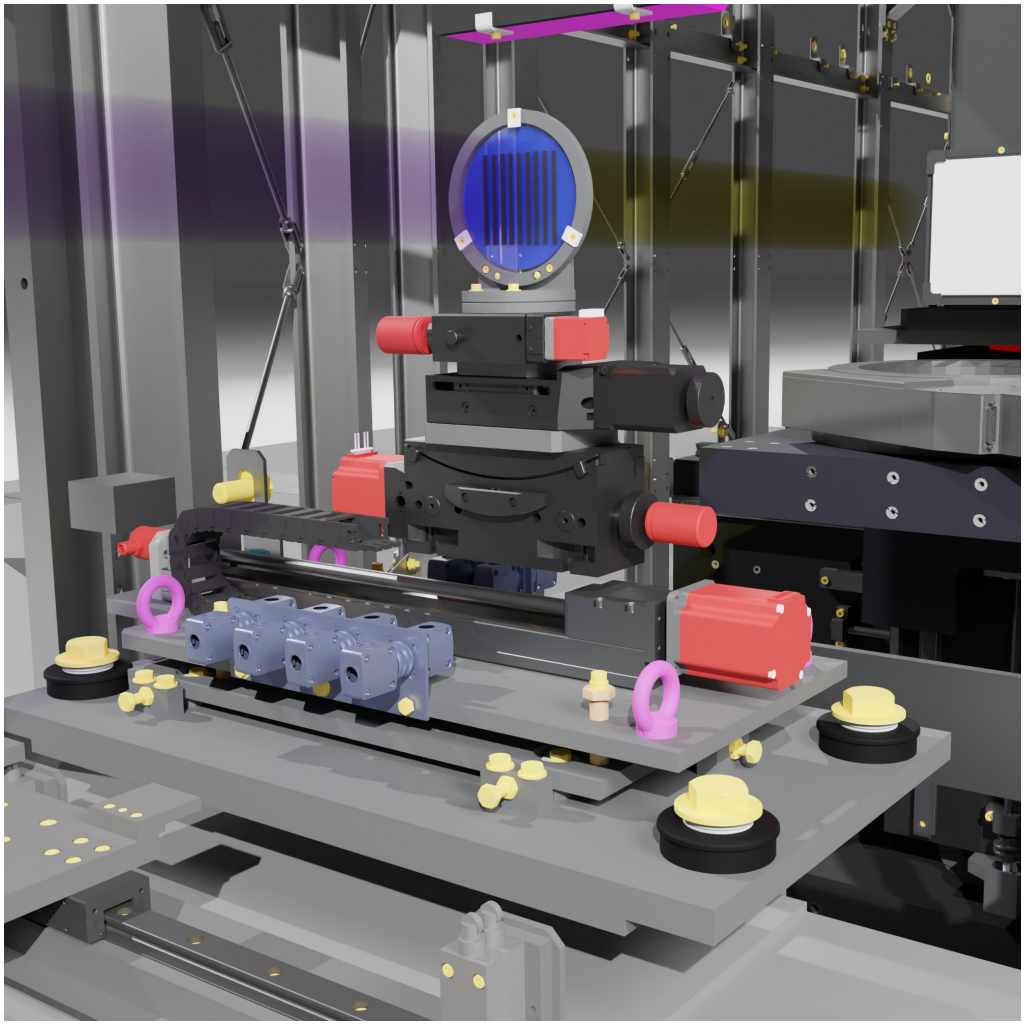


Monochromatic imaging options

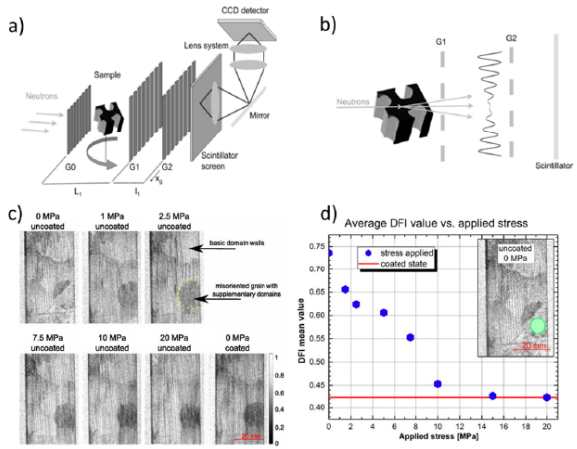
Double-crystal monoch.



Narrower wavelength $\Delta\lambda/\lambda \sim 5\%$



Grating Interferometry

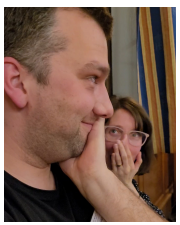


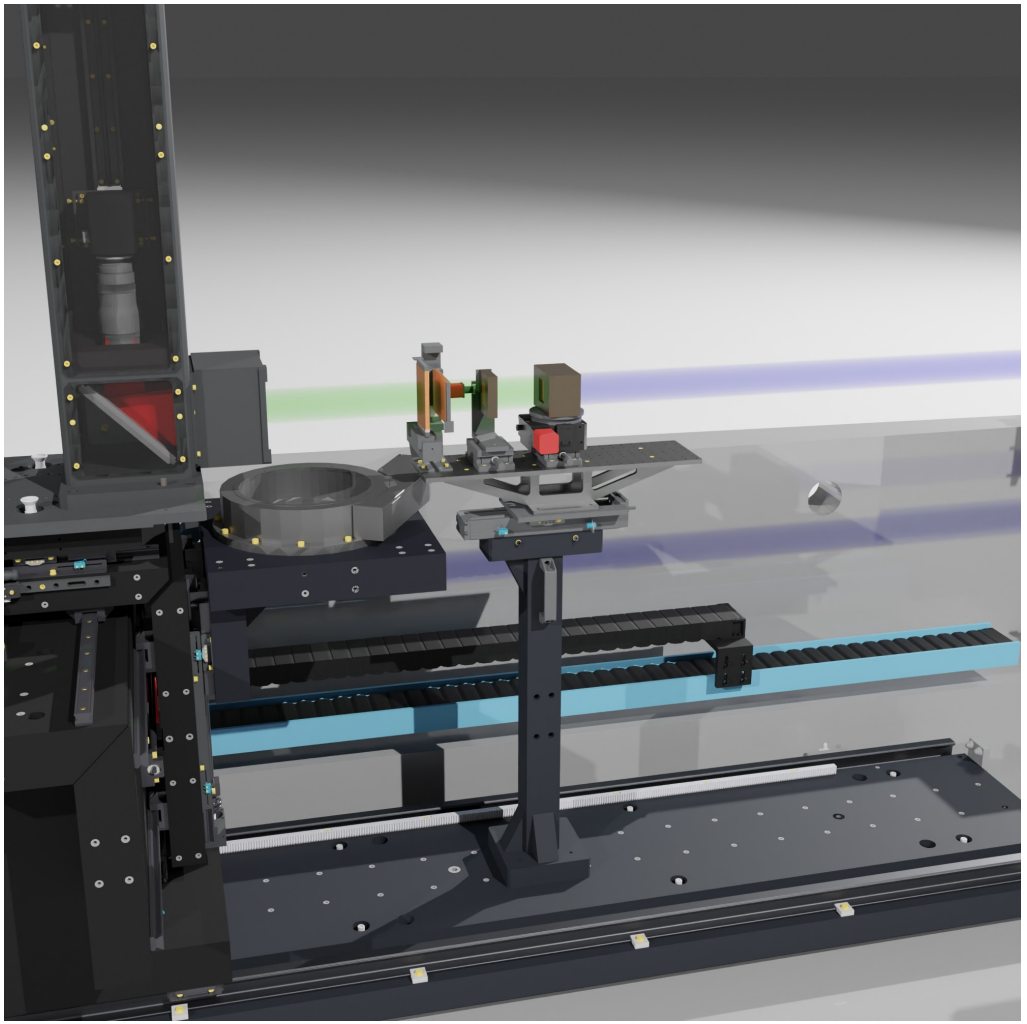
Reimann, T., et al., Journal of Applied Crystallography

In collaboration with TUM

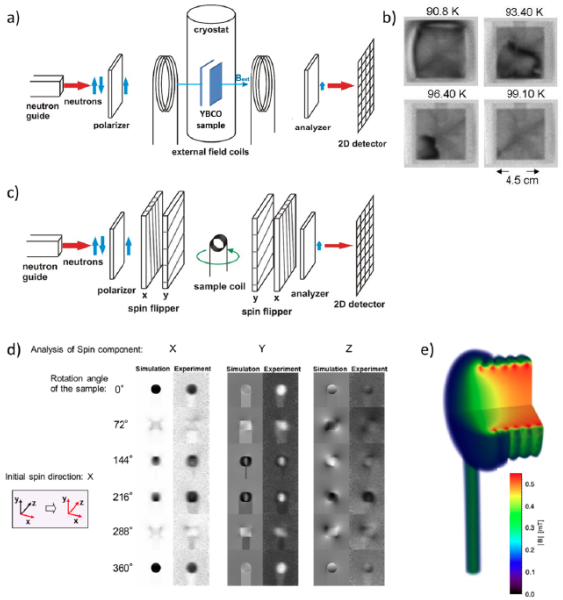

M. Schultz


T. Neuwirth


S. Sebold

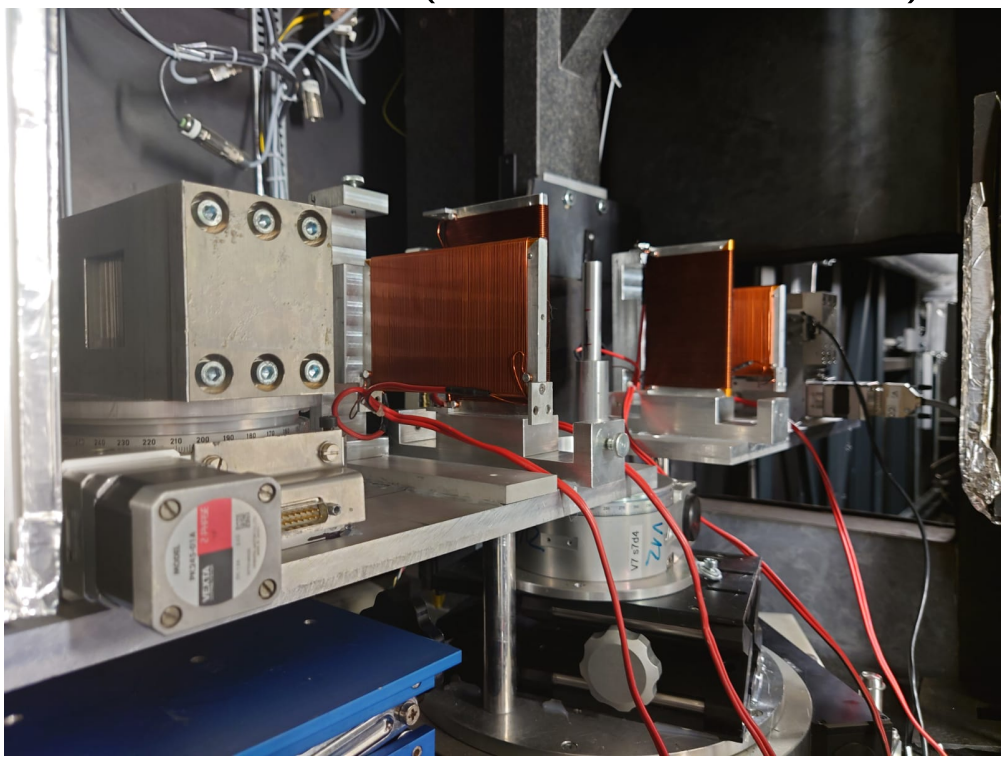


Polariser

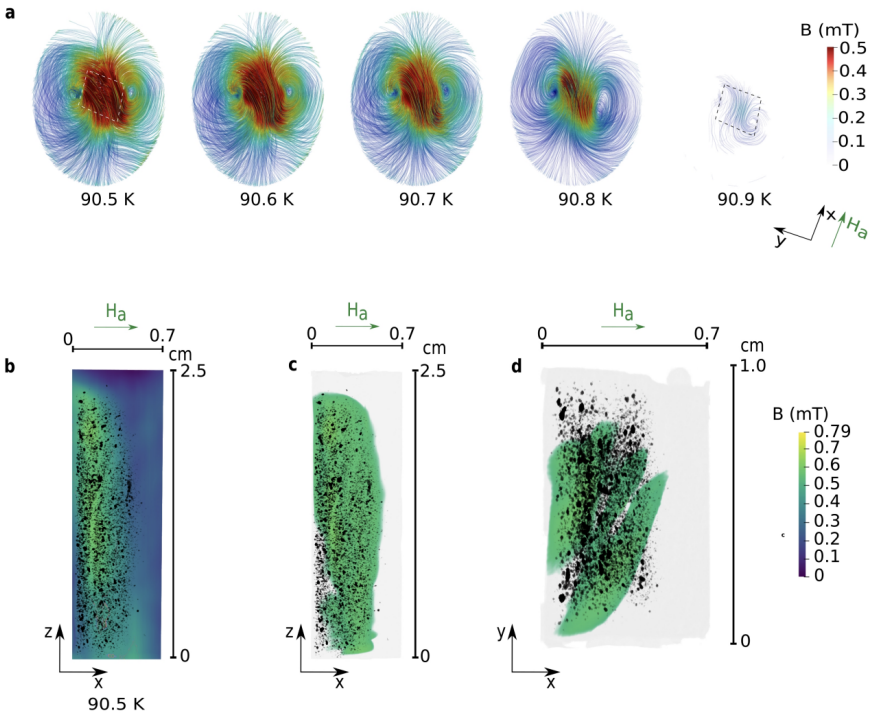


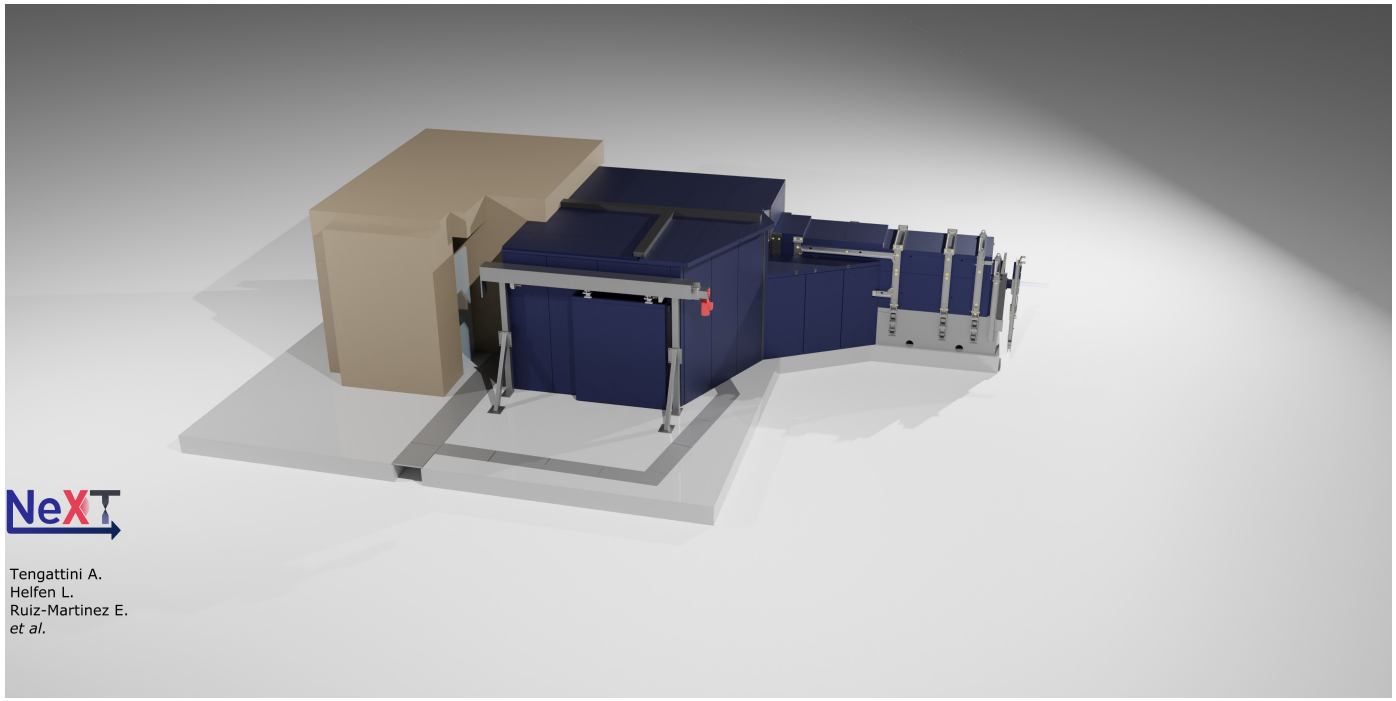
Kardjilov, N., et al., Nuclear Instruments and Methods in Physics Research Section A (2009) 605 (1-2),13

In NeXT 1.0, 2021 with solid state bender, more tests in 2 weeks at NeXT 2.0 (also with ^{3}He cells)

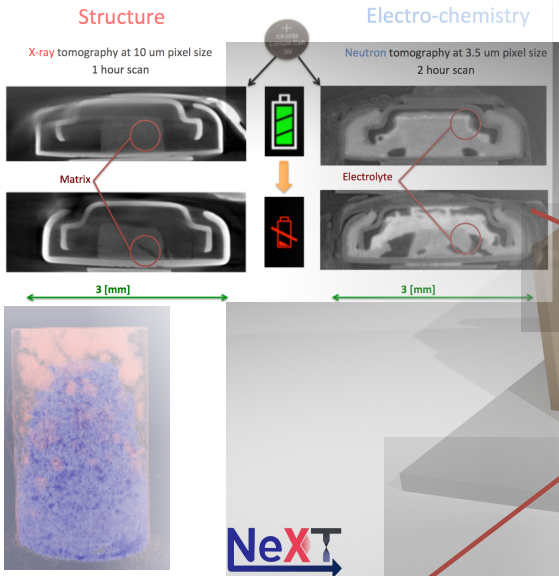


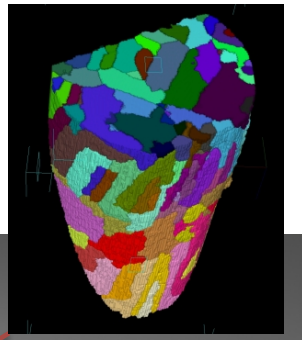
In NeXT 1.0, 2021 with solid state bender
Thesis of Oji Ugwumsinachi with N. Kardjilov



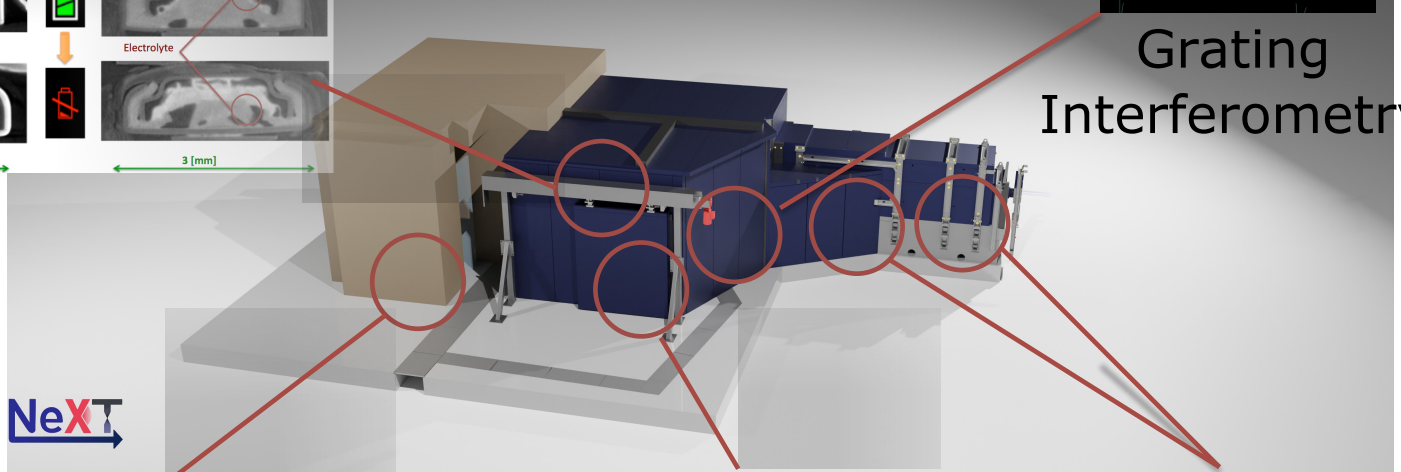

NeXT
Tengattini A.
Helfen L.
Ruiz-Martinez E.
et al.

Simultaneous 5D x+n at high speeds





Grating Interferometry

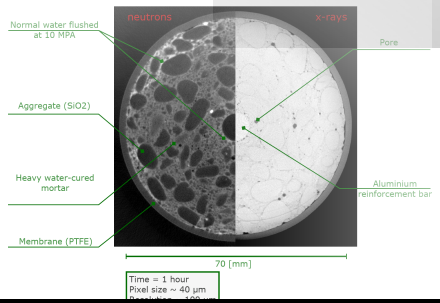


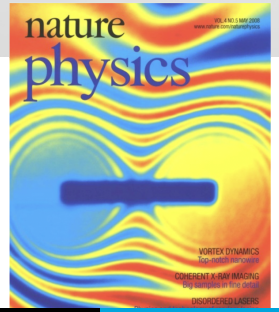
NeXT

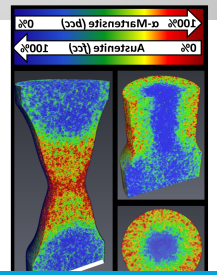
Advanced processing

Polarised

Energy Selective







- Temendous possibilities from Neutron and x-ray imaging
- Neutron Imaging now down to $\leq 4\mu$m resolution and up to 1s tomographies (at much coarser resolutions)
- These images are *quantitative*, *i.e.*, they are **data**
- Plenty of things to fix, plenty of things to develop, in particular in the joint use of x-rays and neutrons

there is still plenty of work (and fun) ahead!

Want to know more?

- About neutron (plus x-ray) imaging review papers:
  - *For Geomechanics:* Tengattini *et al.*, Geom. Ener. Envir. (2021)
  - *For Material science :* Kardjilov *et al.*, Materials today (2018)
  - *For Fluid Flow:* Perfect *et al.*, Earth Science Review (2014)
- about NeXT-Grenoble:
  - Tengattini *et al.*, Nucl. Inst. Met. Phys. Res. A (2020)
  - `contact@next-grenoble.fr`
  - `https://next-grenoble.fr/`

(Too) many people to thank! (*in no particular order)

On the instrument...


L. Helfen

A. Fedrigo

E. Ruiz-Mar.

K. Buckley

N. Kardjilov

N. Lenoir

B. Giroud

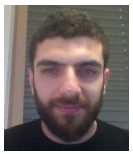
C. Couture

...for the science...


E. Andò

S. Hall

S. Roux

S. Dal Pont

H. Cheick-Sleiman

O. Stamati

Bl Lukich

R. Woracek

P. Moonen

E. Stavrop.

H Lewis

... and for everything!


C. Viggiani

G. Fragneto

H. Schober

M. Johnson

R. Schweins

A. Meyer

J. Jestin

P. Langan

J. Estrade

H. Courtois

Y. Lakhnech

... and many others

Baptiste Amoudrouz ,Luc Didier and the design office, Mark Jacques and the D22 team, Olivier Aguettaz, Alex Quirk, Bob Cubitt and Charles Dewhurst, R.Schweins Thierry Mazilli and Hall D'Essai,Erik Lampasona and Benoit Jarry, Emmanuel Courraud, Nahuel Vega, Fabrice Rencurel, Ian Sives and Sebastien Grimaud and the RP service, Isabelle Perbet and Marius Vuillet, Ali Elaazzouzi, Paolo Mutti and the SCI group, Simon Baudoin, Pierre Courtois, Patrice Cogo, all the the support groups!