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

Alessandro Tengattini et al.





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A historical note about 3SR

Relatively new (2015) Neutron TomographBorn from the collaboration between





A historical note about 3SR

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



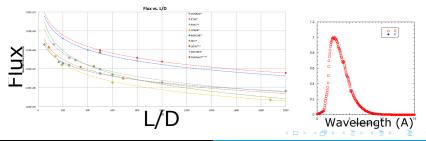


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A historical note about 3SR

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





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A historical note about 3SR

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

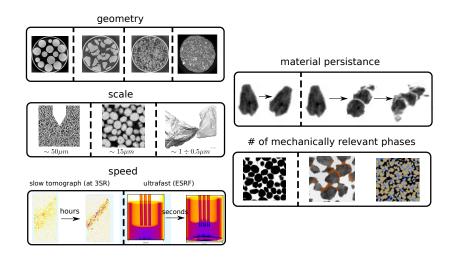


A historical note about 3SR

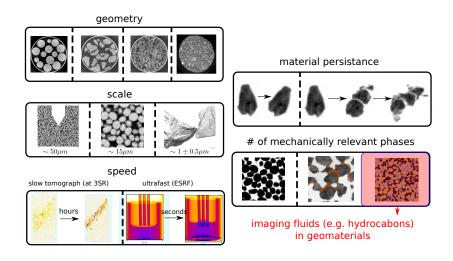
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A historical note about 3SR

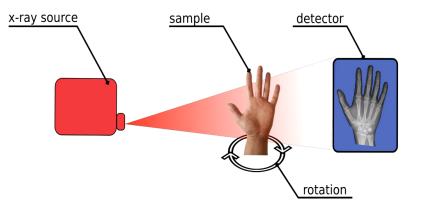


A historical note about 3SR



What is (neutron) imaging?

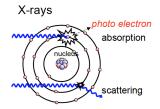
Hydro-thermo-chemo-mechanics though neutron imaging Hydro-thermo-chemo-mechanics though neutron (and x-ray!) The instrument, Conclusions and Prospectives What is tomography Neutrons and x-rays

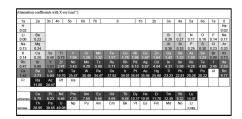


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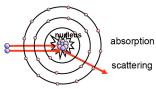
What is (neutron) imaging?

Hydro-thermo-chemo-mechanics though neutron imaging Hydro-thermo-chemo-mechanics though neutron (and x-ray!) The instrument, Conclusions and Prospectives What is tomography Neutrons and x-rays





neutrons



Attenuation coefficients with neutrons [cm?'] 3b 4b 5b 6b 7b 1b 2b 3a 4a 5a 6a 7a He 0.02 344
 B
 C
 N
 O
 F
 Ne

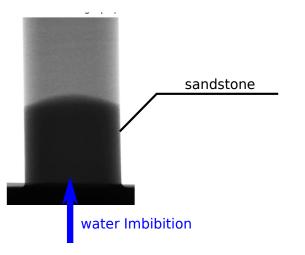
 101.60
 0.56
 0.43
 0.17
 0.20
 0.10

 Al
 Si
 P
 S
 Cl
 Ar

 0.10
 0.11
 0.12
 0.06
 1.33
 0.03
 Ca 0.08 Kr Rh 10.88 Pd Aq Cd In 0.78 4.04 115.11 7.58 0.14 Cs 0.29 Ba 0.07 Ra La Hf 0.52 4.99 Hg TI 0.47 Pb Bi R Ha Ce 0.14 Lr neut 0.69

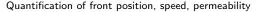
What is (neutron) imaging?

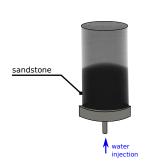
Hydro-thermo-chemo-mechanics though neutron imaging Hydro-thermo-chemo-mechanics though neutron (and x-ray!) The instrument, Conclusions and Prospectives What is tomography? Neutrons and x-rays

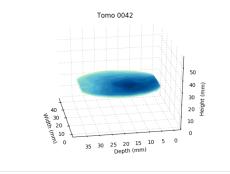


Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Fluid flow within an intact rock



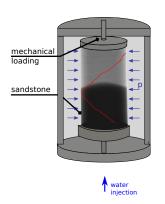




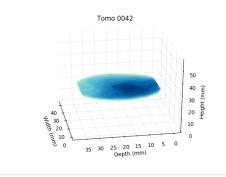
Tudisco, *et al.*, Journal of Geophysical Research: Solid Earth (2019) Extegarai, *et al.*, Journal of Imaging (2021) Viera-Lima, *et al.*, submitted (2023)

Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Fluid flow within a damaged rock



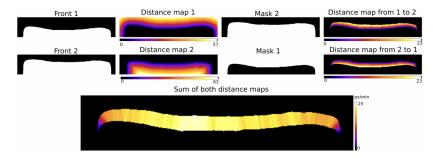
Quantification of front position, speed, permeability



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

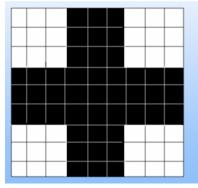
Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

An example of "Bespoke analysis" - speed maps



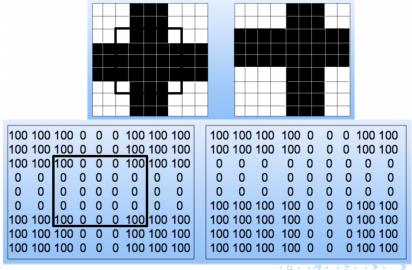
Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

A "go-to keystone Tool" Digital Volume Correlation (e.g., SPAM)

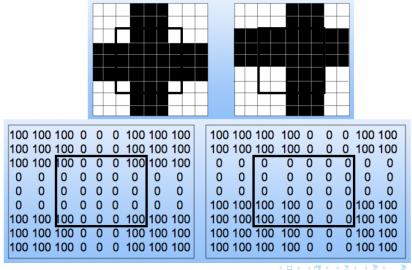


100	100	100	0	0	0	100	100	100
100	100	100	0	0	0	100	100	100
100	100	100	0	0	0	100	100	100
0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0
100	100	100	0	0	0	100	100	100
100	100	100	0	0	0	100	100	100
100	100	100	0	0	0	100	100	100

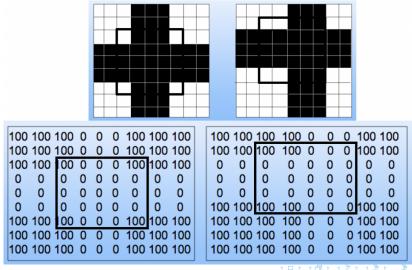




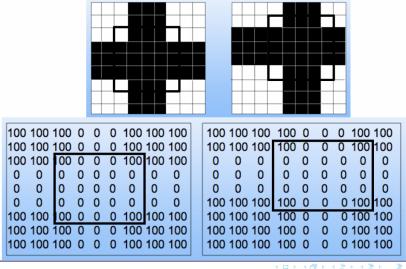








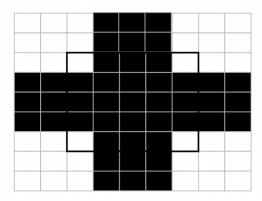


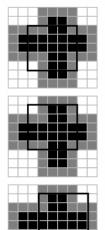


Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

A "go-to keystone Tool" Digital Volume Correlation (e.g., SPAM)

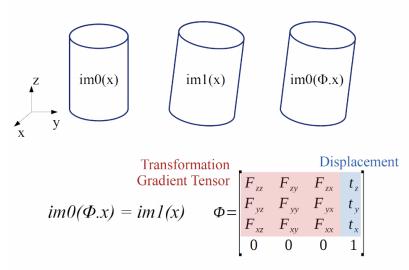
Best correlation?





Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

A "go-to keystone Tool" Digital Volume Correlation (e.g., SPAM)



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Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

A "go-to keystone Tool" Digital Volume Correlation (e.g., SPAM)

Error function:

$$\tau(\Phi) = \frac{1}{2} \sum_{x \in ROI} (im0(x) - im1(\Phi x))^2$$

Minimisation problem:

 $\nabla \tau(\Phi) = 0$

Newton's method:

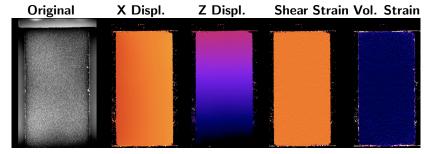
$$\nabla \tau(\Phi^n) + \nabla^2 \tau(\Phi^n) : \delta \Phi^{n+1} = 0$$

Implementation *:

$$\delta \Phi^{n+1} = (M^n)^{-1} A^n$$

Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

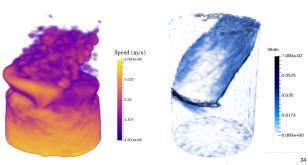
A "go-to keystone Tool" Digital Volume Correlation (e.g., SPAM)



3D SPEED FIELD

Hydro-**Hydro-mechanics** Hydro-thermo-mechanics Hydro-chemo-mechanics

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



3D DEVIATORIC STRAIN FIELD

Hydro-**Hydro-mechanics** Hydro-thermo-mechanics Hydro-chemo-mechanics

How about more complex geometries?

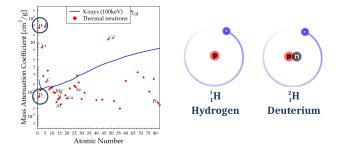


Lewis *et al.*, EGU (2017) Lewis *et al.*, Transport in Porous Media (2023)

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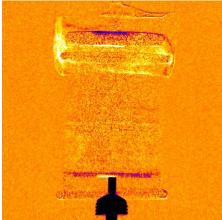
Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

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



Hydro-**Hydro-mechanics** Hydro-thermo-mechanics Hydro-chemo-mechanics

Injection in D20-saturated sample



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

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Hydro-**Hydro-mechanics** Hydro-thermo-mechanics Hydro-chemo-mechanics

and in 3D?

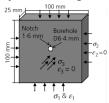


Lewis *et al.*, EGU (2017) Lewis *et al.*, Transport in Porous Media (2023)

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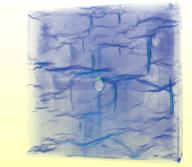
Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Hydrofracking of rocks



 $\begin{array}{l} \mbox{Marcellus shale} \\ \sigma_1 = 70 \mbox{ MPa} \\ \mbox{Flow rate } /6/12 \mbox{ ml/min} \end{array}$

Quantification of front position, speed, permeability

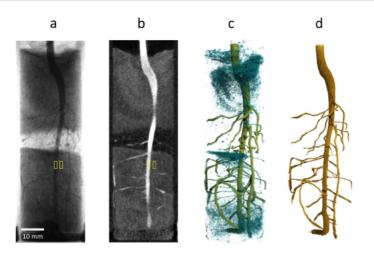


Roshankhah, et al., Géotechnique letters (2018) Roshankhah, et al., USRMS (2019)

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Hydro-**Hydro-mechanics** Hydro-thermo-mechanics Hydro-chemo-mechanics

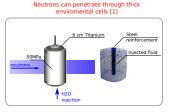
Other applications



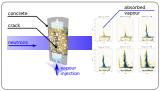
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Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

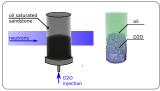
Other applications



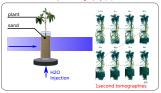
Neutrons can help track even vapour low into fractures [2]



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



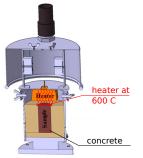
Neutrons tomographies can be acquired as fast as 1s per tomography [4]



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

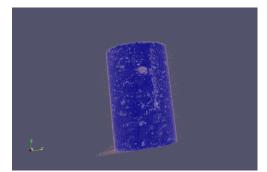
Hydro-Hydro-mechanics **Hydro-thermo-mechanics** Hydro-chemo-mechanics

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

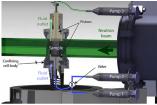


Dauti *et al.*, Cement and Concrete Research (2018) Dauti *et al.*, Transport in Porous Media (2019) Tengattini *et al.*, Strain (2020)

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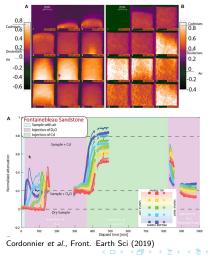
Hydro-Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Cadmium Sorption and Transport in Porous Rocks



Study of Cd concentration

$\label{eq:Quantification of Cd sorption and displacement of Contaminants$



Hydro-Hydro-mechanics Hydro-thermo-mechanics **Hydro-chemo-mechanics**

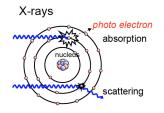
In summary

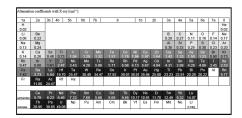
- = Neutrons have a different contrast from x-rays
- Neutrons can see light elements (such as hydrogen, lithium) and their compounds (*e.g.*, water, oil)
- Neutrons are isotope sensitive; for example can distinguish deuterated fluids as D20 vs H2O
- Neutron can penetrate through thick metal casings for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

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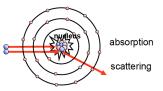
NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

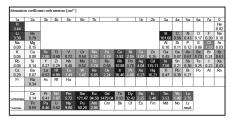
Neutrons or X-Rays?



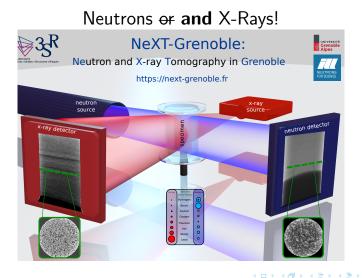


neutrons





NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics



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NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

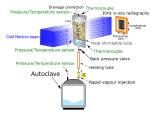


Martell et al., Science Advances, 2022

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NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Vapour Injection



Emulating Loss of Coolant Accident in Reactor

anr°

Initially saturated



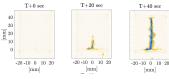




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Initially dry

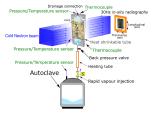




Gupta, *et al.*, Cem Concrete Research (2022) Lukich, *et al.*, Material Letters (2021)

NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Vapour Injection



Emulating Loss of Coolant Accident in Reactor

anr®

3D rendering of the superimposed segmented phases

- Voxel size = 47 µm, Image resolution: 920x920x920
- > Segmentation in spam (Stamati et al., 2020)



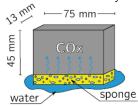




Gupta, *et al.*, Cem Concrete Research (2022) Lukich, *et al.*, Material Letters (2021)

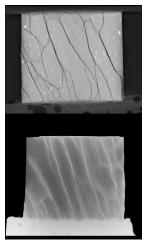
NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

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)

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What is (neutron) imaging? Hydro-thermo-chemo-mechanics though neutron (and x-ray!)

COx

00

contaminants

ANDRA

water

13 mm

mm

45

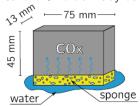
Hvdro-mechanics

Hydo-mechanical coupling of Sample Sample Sample Time since Sample Normal-N Parallel-X Normal-X first wetting Parallel-N Callovo-Oxfordian Clayrock 0-5 min -75 mm— 10-15 min 20-25 min 30-35 min sponge 40-45 min Rock used for long-term storage of radioactive 50-55 min 60-65 min 80-85 min 10 mm x-ray attenuation coefficient (µ) or neutron beam transmission (1/l_n)

> low high Stavropoulou, et al., Acta Geotechnica (2018) Stavropoulou, et al., Front. in Earth Science (2020)

NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

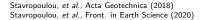
Hydo-mechanical coupling of Callovo-Oxfordian Clayrock



me change (% of initial sample volume) 10 8 Mask Volume Increase (Parallel-X) 4 Crack Volume (Parallel-X) Absorbed Water (Parallel-N) Mask Volume Increase (Normal-X) ----Crack Volume (Normal-X) ----Absorbed Water (Normal-N) ----20 40 60 80 100 Time since start of imbibition (min)

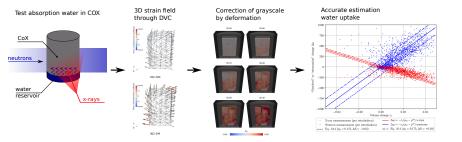
Rock used for long-term storage of radioactive contaminants





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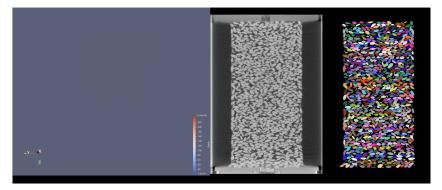
NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics



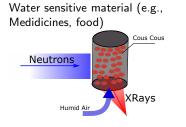
Stavropoulou, et al., Front. in Earth Science (2020)

NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

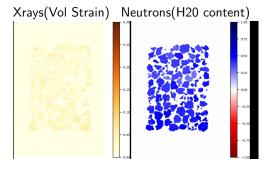
How about Granular Media - Discrete DVC!



NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

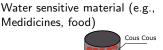


Societal problem: Caking – loss of usability of food exposed to humidity Horizon 2020 ITN CALIPER



Vego, Tengattini, *et al.*, Soft Matter (2022) Vego, Tengattini, *et al.*, Granular Matter (2023)

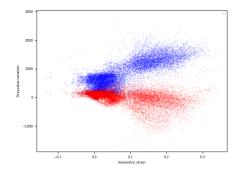
NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics



Neutrons Humid Air

Societal problem: Caking – loss of usability of food exposed to humidity Horizon 2020 ITN CALIPER

Statistical correlation of Vol. Strain to H20 content

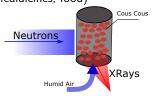


Vego, Tengattini, *et al.*, Soft Matter (2022) Vego, Tengattini, *et al.*, Granular Matter (2023)

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NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

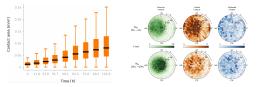
Water sensitive material (e.g., Medidicines, food)



Societal problem: Caking – loss of usability of food exposed to humidity Horizon 2020 ITN CALIPER

Contact Analysis

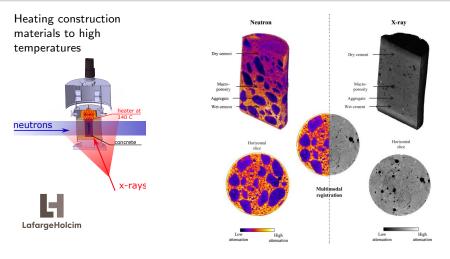




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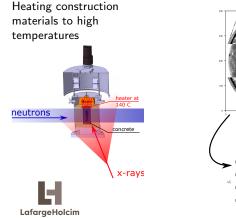
Vego, Tengattini, *et al.*, Soft Matter (2022) Vego, Tengattini, *et al.*, Granular Matter (2023)

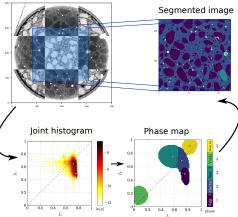
NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro chemo mechanics



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

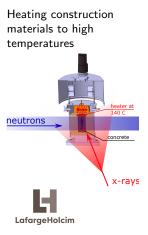
NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

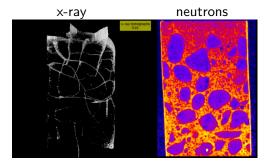




Tudisco et al., Measurement Science and Technology (2017) Roubin et al., Cement and Concrete Composites (2019) Sleiman, Cement and Concrete Research (2021)

NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

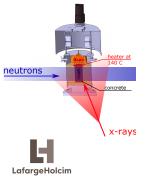


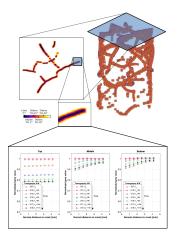


Tudisco *et al.*, Measurement Science and Technology (2017) Roubin *et al.*, Cement and Concrete Composites (2019) Sleiman, Cement and Concrete Research (2021)

NeXT-Grenoble and other instruments Hydro-mechanics **Hydro-thermo-mechanics** Hydro-chemo-mechanics

Heating construction materials to high temperatures





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

What is (neutron) imaging? Hydro-thermo-chemo-mechanics though neutron (and x-ray!)

> heater at 140 C

concrete

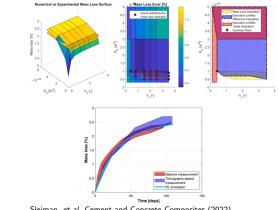
x-rays

Heating construction materials to high temperatures

neutrons

LafargeHolcim

Towards more realistic numerical models

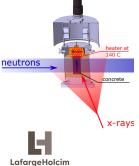




Tudisco et al., Measurement Science and Technology (2017) Roubin et al., Cement and Concrete Composites (2019)

NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Heating construction materials to high temperatures



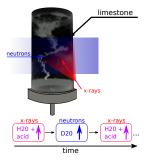
Towards more realistic numerical models



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

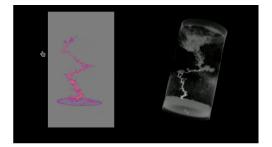
NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Wormhole Growth in Dissolving Limestones



From X-ray: wormhole development by acid dissolution

From neutron: evolution hydraulic propertie

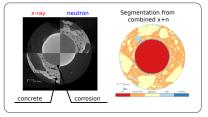


Szymczak et al., EGU (2021) Szymczak et al., AGU (2020) Cooper et al., Advances in Water Resources(2023) Cooper et al., under redaction(2023)

NeXT-Grenoble and other instruments Hydro-mechanics Hydro-thermo-mechanics Hydro-chemo-mechanics

Other applications

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



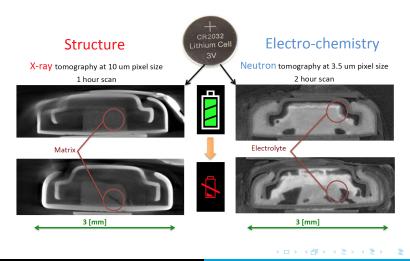
Robuschi et Al., Cement and Concrete Research (2021)
 Tudisco et Al., Measurement Science and Technology (2017)
 Roubin et Al., Cement and Concrete Composites (2019)

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



What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions

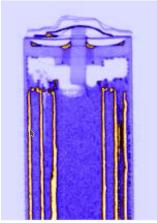
Other applications



Tengattini et al. Neutron (and X-ray) imaging for porous media 39 / 54

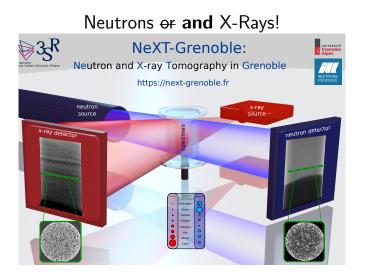
What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions

30 minute tomographies at 30um



Ziesche et Al. 2020 Nature Comm. Ziesche et Al. 2020 J. Electrochem. Soc.

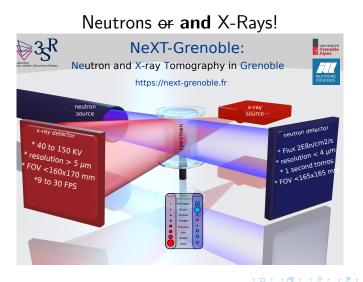
What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions



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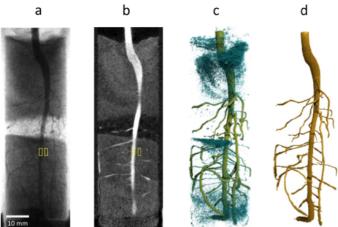
What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions



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What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions

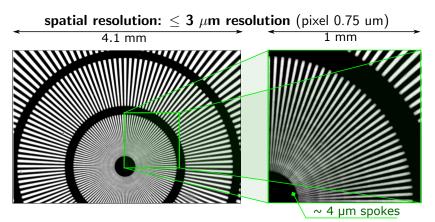
temporal resolution: 1.5s tomographies



Totzke et al., 2019 Optics Express

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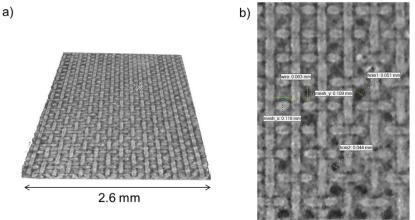
What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions



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

What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions

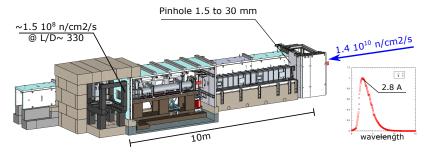
spatial resolution: \leq 3 μ m resolution (pixel size 1.5 μ m)

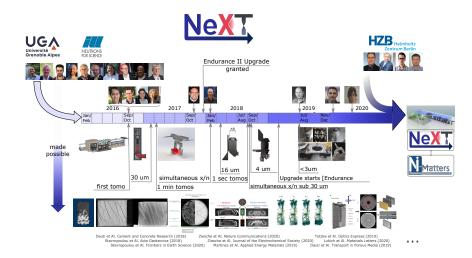


 ${\sim}10h~{\rm tomo}~{\rm Tengattini}$, et al., 2022 Optics Express

What can NeXT do? NeXT 1.0 The project A zoom on the upgrades Conclusions

The old instrument (NeXT V.1.0)/ D50Tomo





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What can NeXT do? NeXT 1.0 **The project** A zoom on the upgrades Conclusions

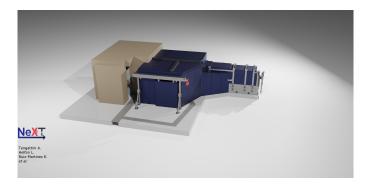
NeXT 2.0



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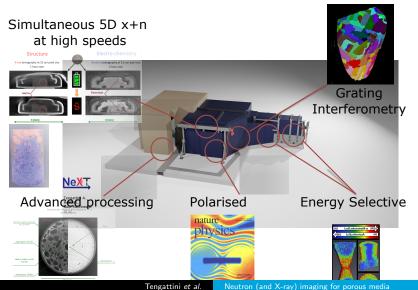
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What can NeX I do? NeXT 1.0 The project A zoom on the upgrades Conclusions



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What can NeX I do? NeXT 1.0 The project A zoom on the upgrades Conclusions



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What can NeX I do? NeXT 1.0 The project A zoom on the upgrades Conclusions

The status quo



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What can NeXT do? NeXT 1.0 The project A zoom on the upgrades **Conclusions**

- 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!

What can NeXT do? NeXT 1.0 The project A zoom on the upgrades **Conclusions**

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)
 - o contact@next-grenoble.fr
 - https://next-grenoble.fr/

Conclusions

Many many people to thank!













C. Viggiani

E. Ando'

S. Hall

L. Helfen

N. Lenoir N. Kardjilov C. Couture D. Atkins







Sleiman

P. Besuelle

S. Dal Pont H. Cheick- M. Briffaut D. Dauti



F. Dufour Ε. Stavropoulou





G. Couples M. Madankan P. Szymczak M. Cooper



E. Tudisco





E.M.

Charalampidou

I Andrade S. Roshankhah









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F. Renard B. Cordonnier D. Gregoire Α. Pluymakers





H. Lewis



Neutron (and X-ray) imaging for porous media

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