



# advanced image analysis to elucidate coupled problems in porous media

work performed at the DMEX Center for X-ray Imaging

by Syrine Ben Elhadj Hamida, Hannelore Derluyn, Jelle Dhaene, Peter Moonen and co-workers

**Prof. Peter MOONEN**

UAR 3360 - Développement de Methodologies Expérimentales (DMEX)  
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# real applications are complex

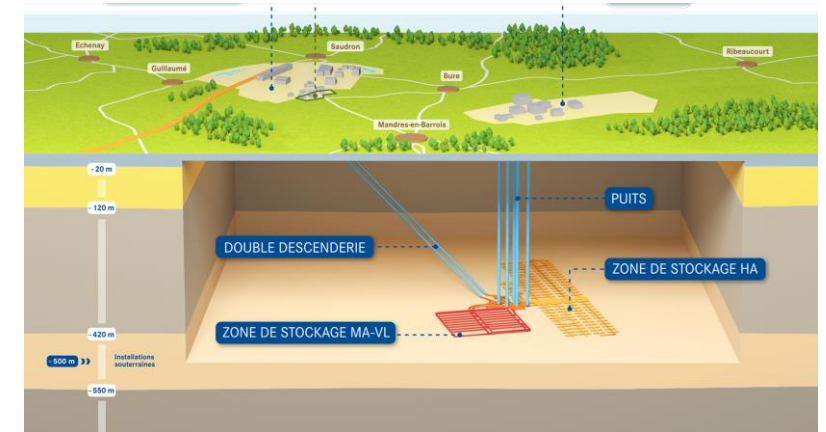
## cultural heritage preservation



© AdobeStock

Villa Belza, Biarritz, France

## nuclear waste storage



© ANDRA

Meuse, France

## natural hydrogen production



Sao Francisco Basin, Brazil

- coupled
- cracking
  - heat
  - moisture
  - reactions
  - biology
  - ...

# the study of coupled processes



© Bruno LECLERC

- ▶ is challenging
- ▶ requires a combination of experimental and numerical research

our group uses X-ray (& neutron) imaging

- non-invasive
- three-dimensional + time

we develop acquisition and analysis protocols with focus on in situ/operando studies



# tomographs @ DMEX (UPPA, Pau, France)



## high resolution



source : 30-160 keV, 10W  
CCD : 4 MPx; 0.61 fps

sample size : mm-range  
voxel size :  $\geq 0.3 \mu\text{m}$

low blur

## high contrast



20-180keV, 300W  
8 MPx; 100 fps

cm-range  
75-3  $\mu\text{m}$

spectral ability

## high-speed

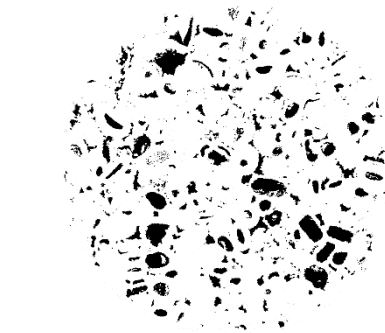
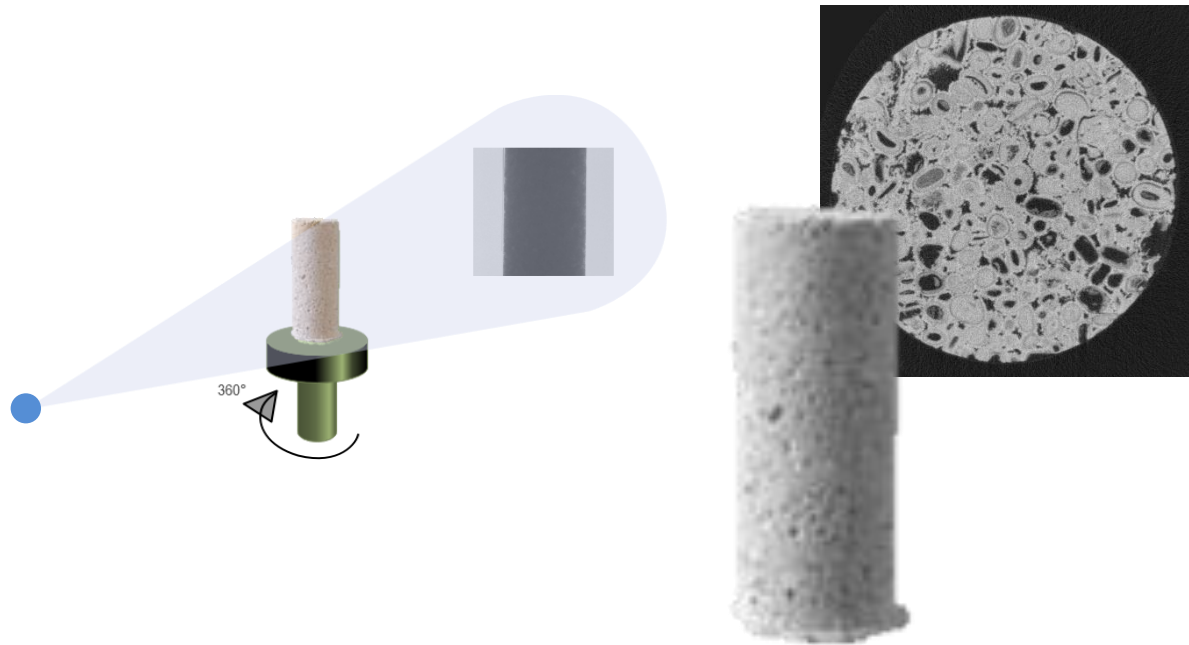


40-130keV, 39W  
2.8 MPx;  $>100$  fps

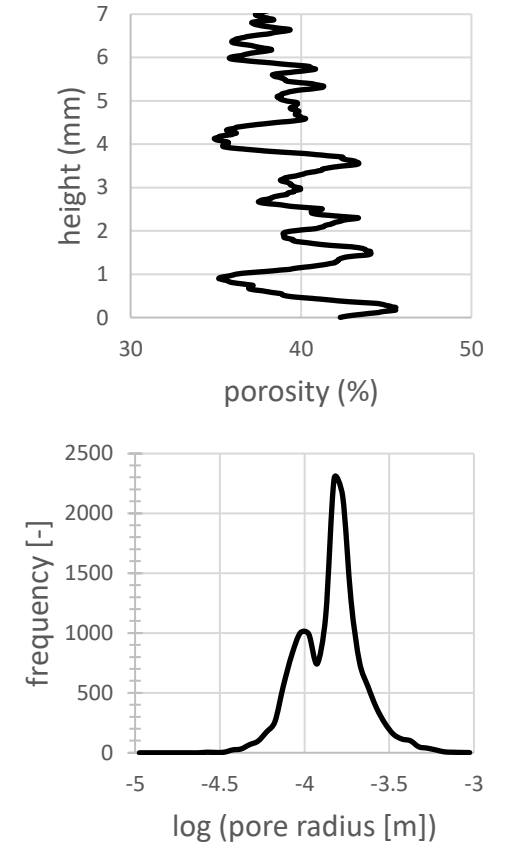
cm-range  
37.5-5  $\mu\text{m}$

no sample motion

# conventional workflow



- filtering
- segmentation
- ...



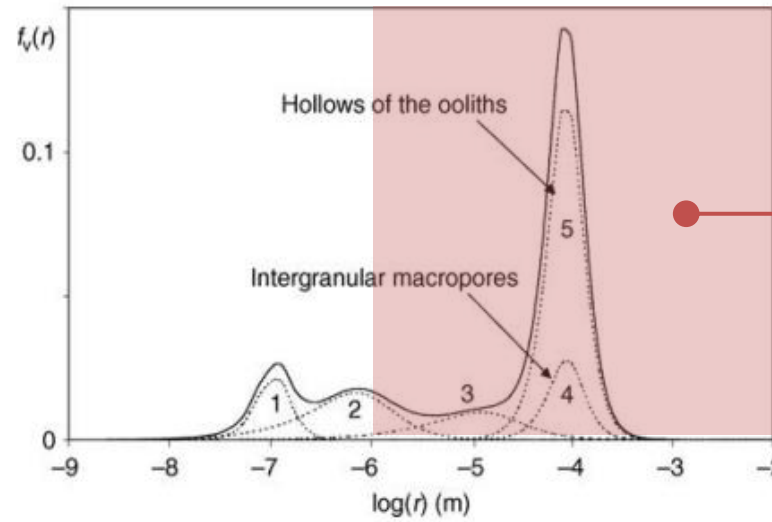
**material & process**

# LIMITS OF THE CONVENTIONAL WORKFLOW

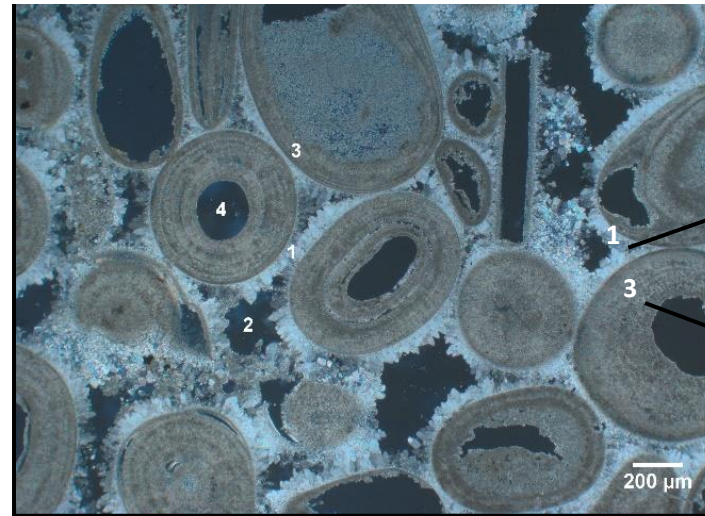
# multiple scales



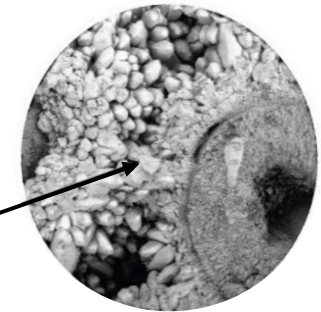
© Peter Haas



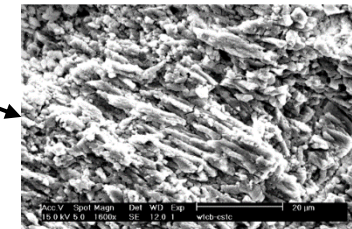
direct observation by lab-based X-ray tomography



© Hannelore Derluyn

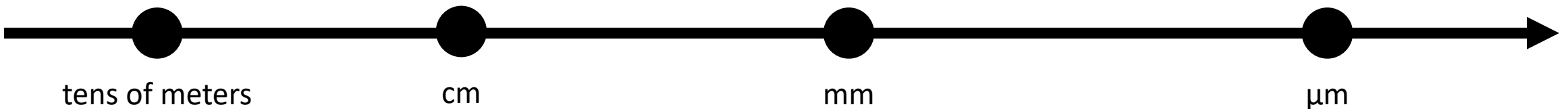


50μm

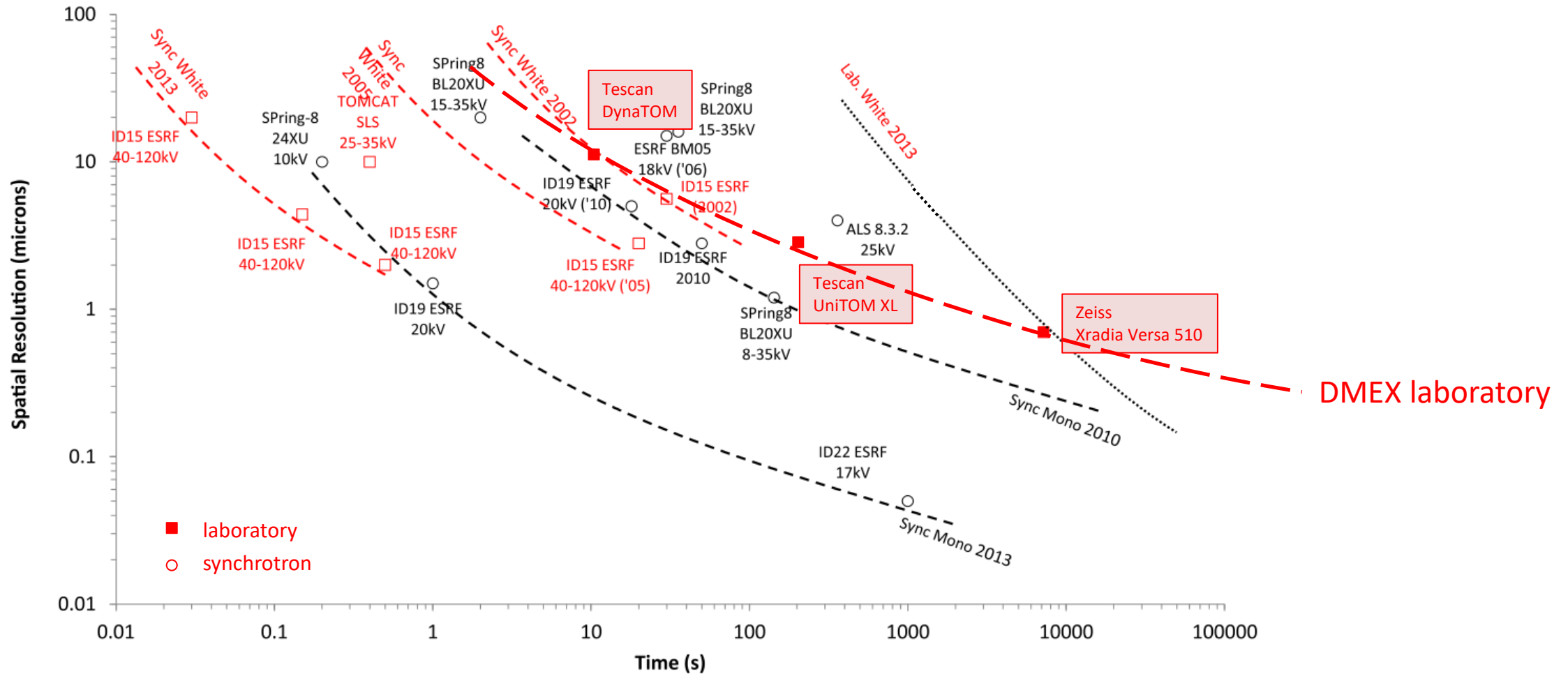


20μm

© Staf Roels



# spatio-temporal instrumental constraints



adapted from Bultreys et al. 2016

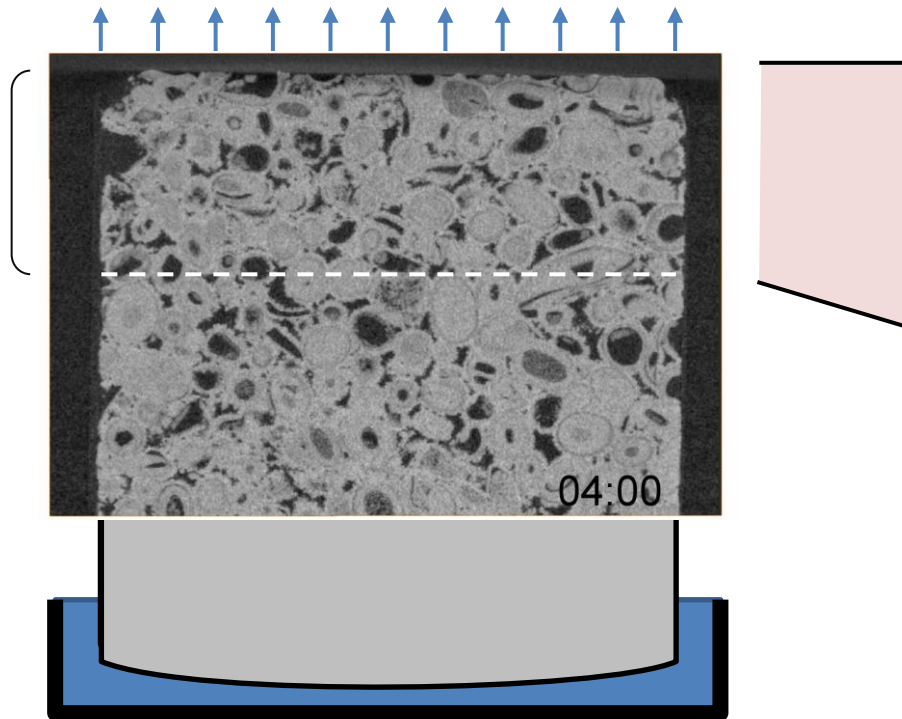


# coupled processes

Savonnières sample ( $\phi=8.4\text{mm}$ ,  $H=9\text{mm}$ )

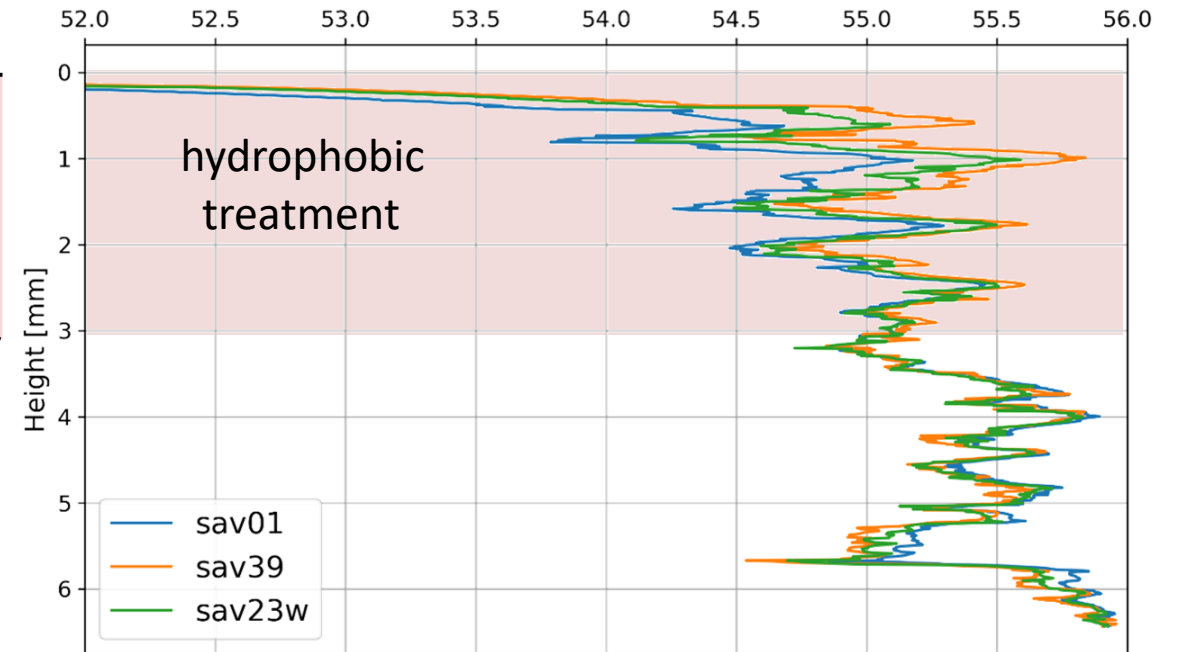
evaporation

hydrophobic treatment



uptake with 6 molal NaCl solution

cross-sectional area over time  
[mm]



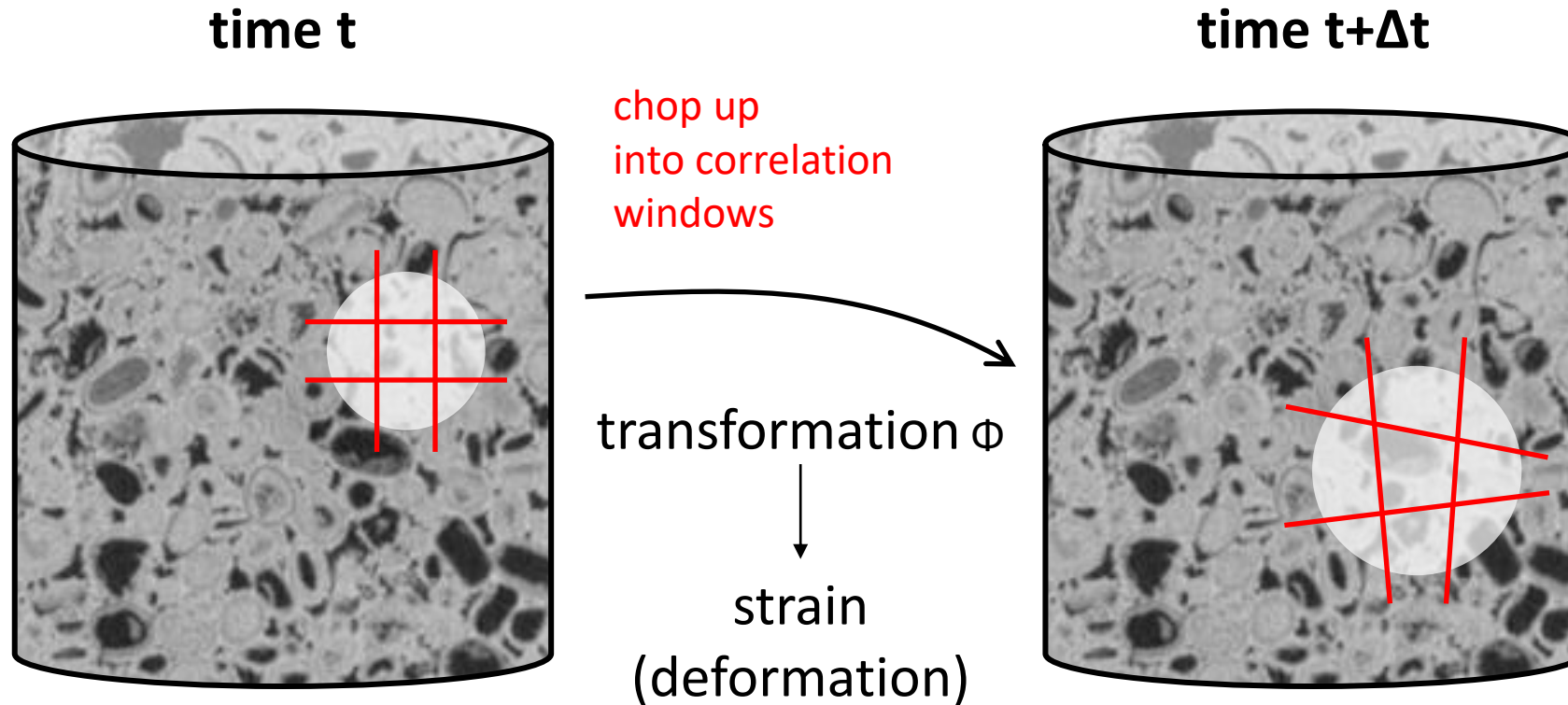
dimensional changes

→ direct comparison different time points difficult

accounting for deformation

# DIGITAL VOLUME CORRELATION (DVC)

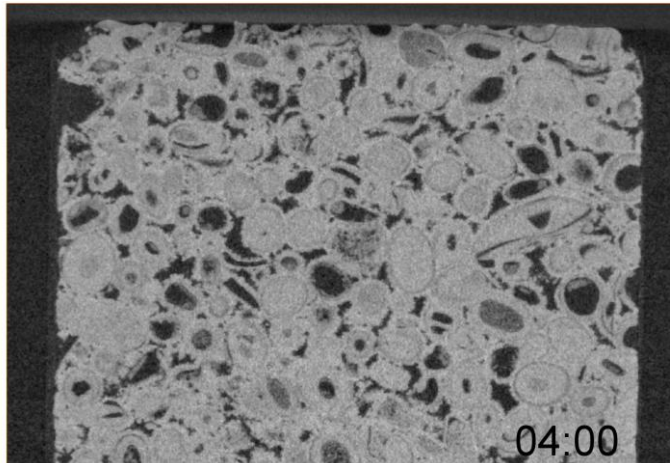
# principle of (local) DVC



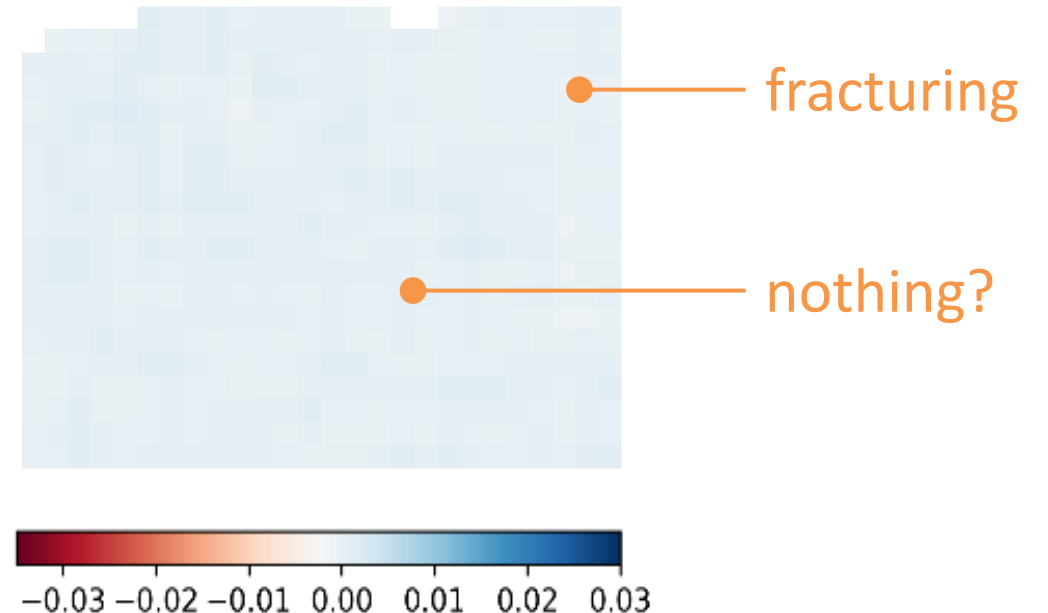
→ implemented in tools such as SPAM ([spam-project.dev](http://spam-project.dev), Stamati et al. 2020)

# DVC applied to a drying sample (1/2)

raw data



volumetric strain [-]



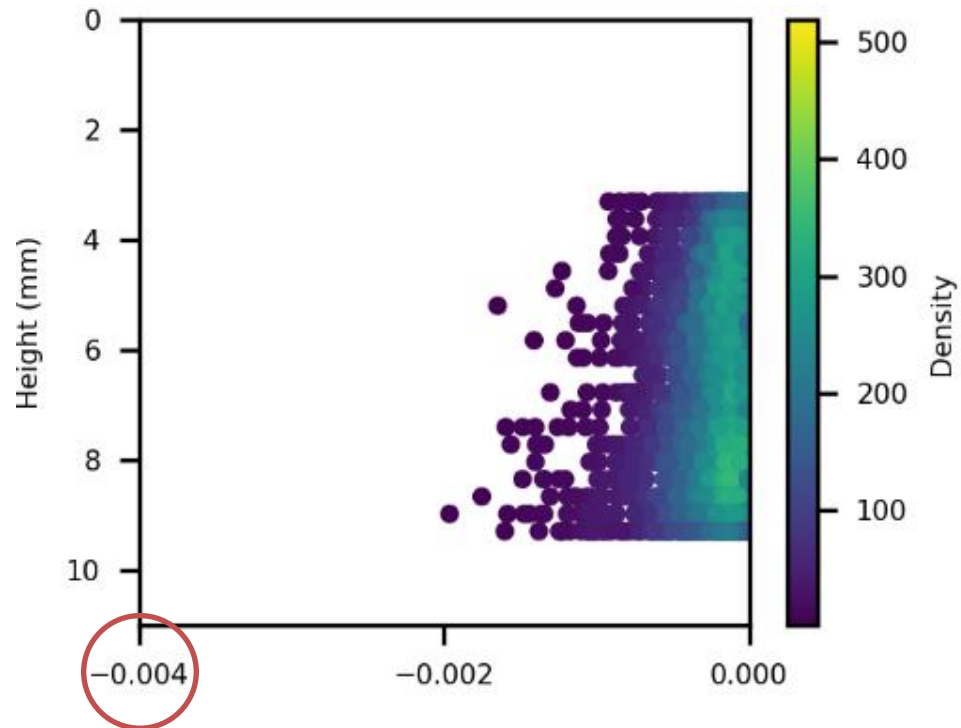
- high positive volumetric strain = fractures
- bulk of sample: small volumetric strain



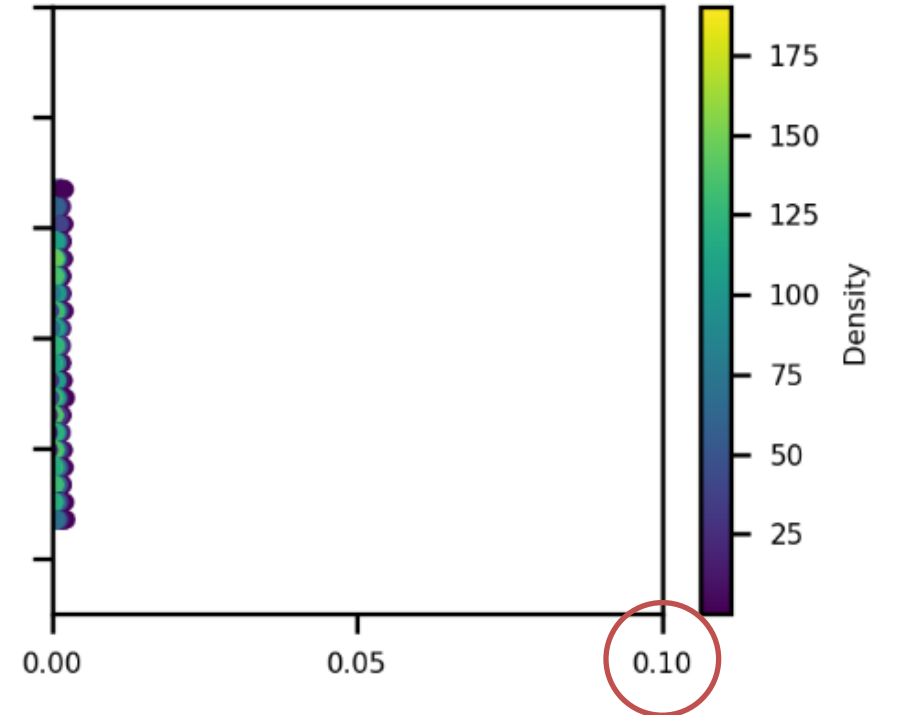
# DVC applied to a drying sample (2/2)

90 min  
drying

**negative** volumetric  
strain distribution



**positive** volumetric  
strain distribution

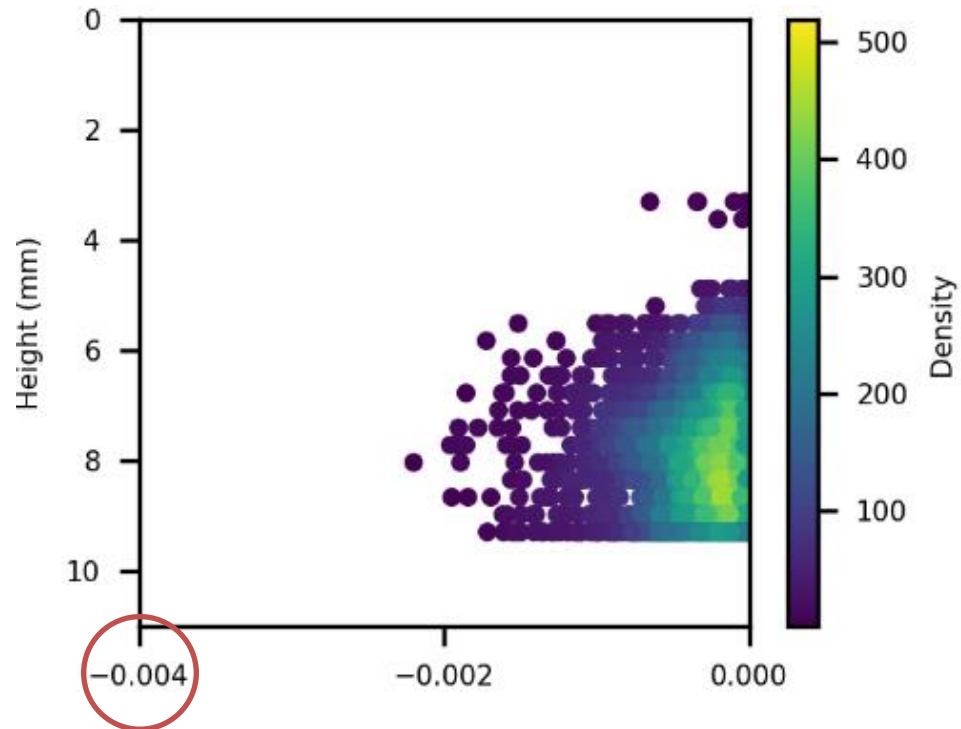


note scale difference

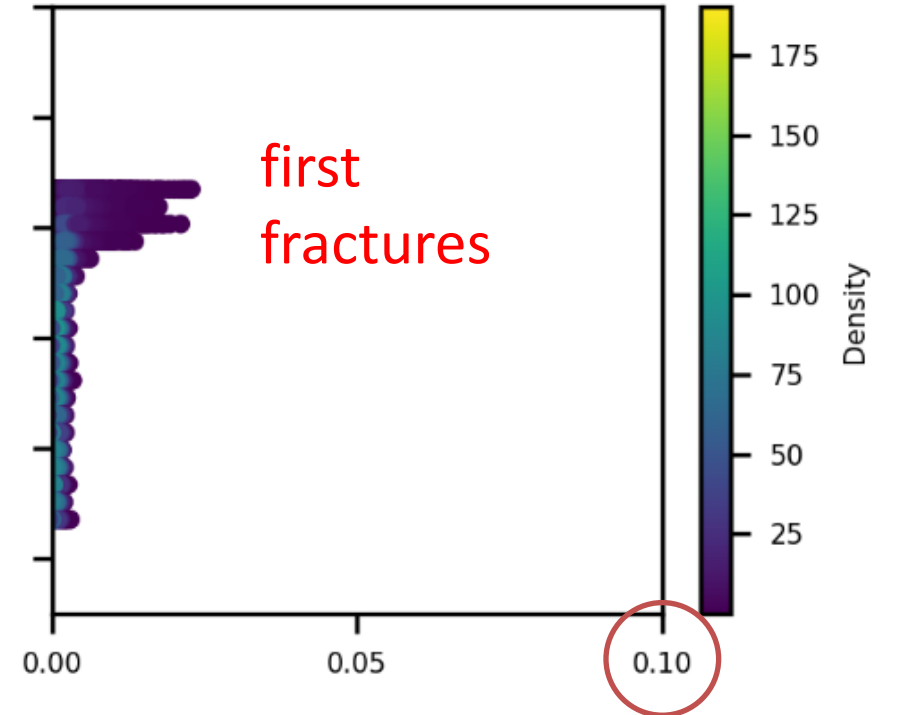
# DVC applied to a drying sample (2/2)

270 min  
drying

**negative** volumetric  
strain distribution



**positive** volumetric  
strain distribution

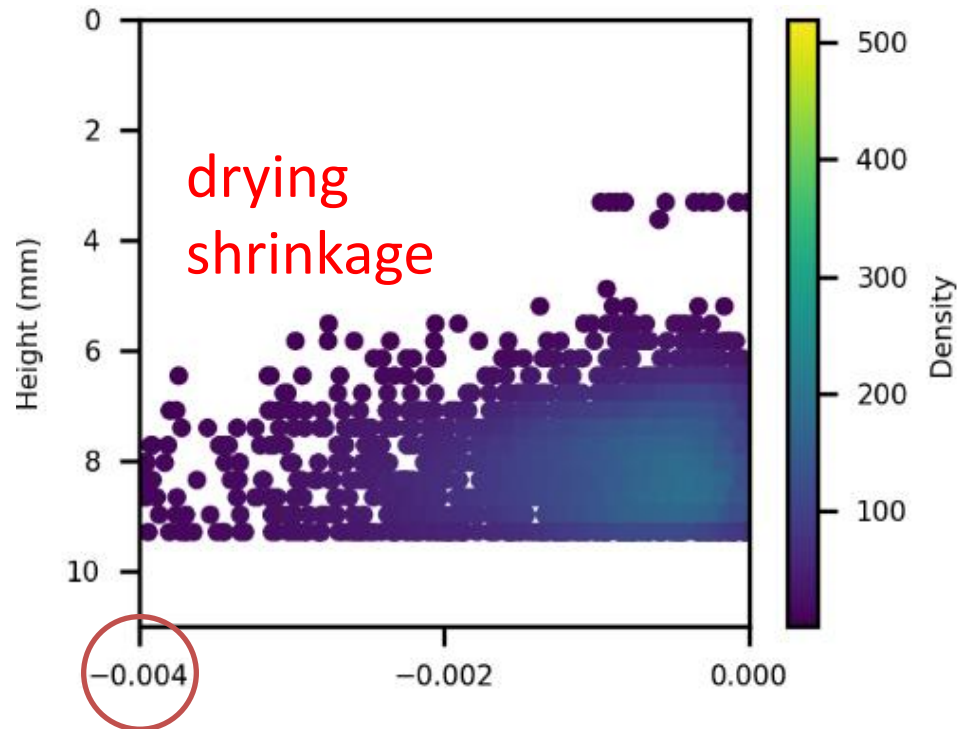


note scale difference

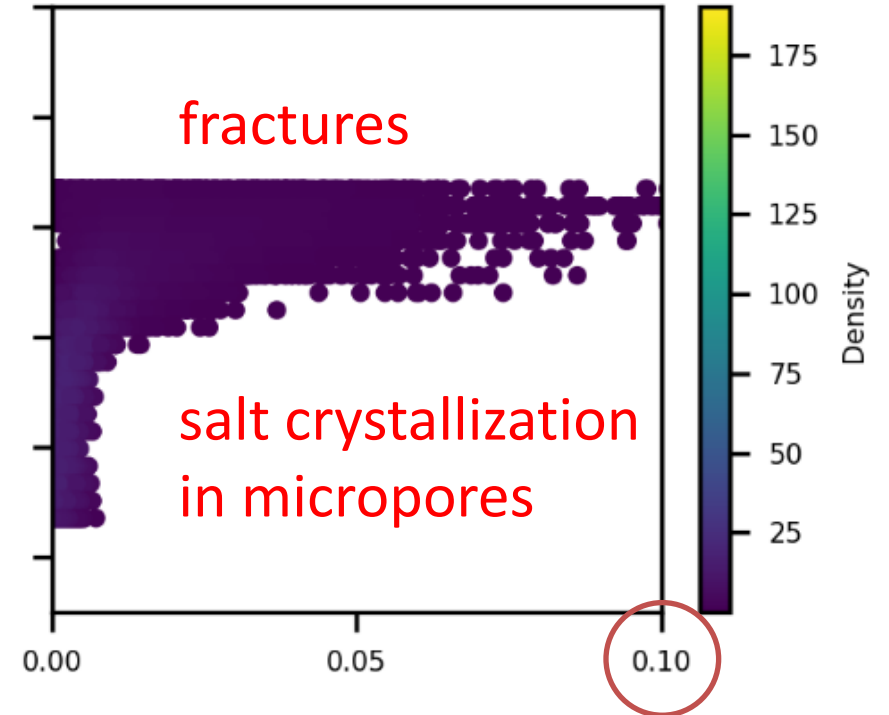
# DVC applied to a drying sample (2/2)

1200 min  
drying

**negative** volumetric  
strain distribution



**positive** volumetric  
strain distribution



note scale difference

**extracting fluid saturation**

# **DVC-CORRECTED DIFFERENTIAL IMAGING**

Ben Elhadj Hamida et al. (in preparation)



# motivation

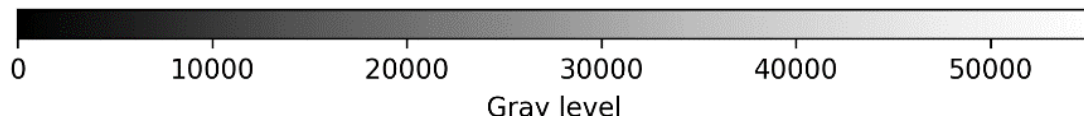
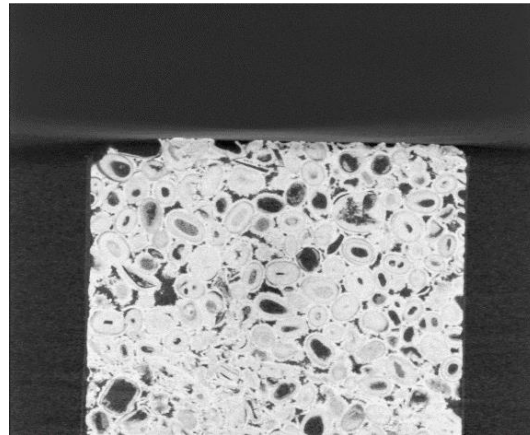
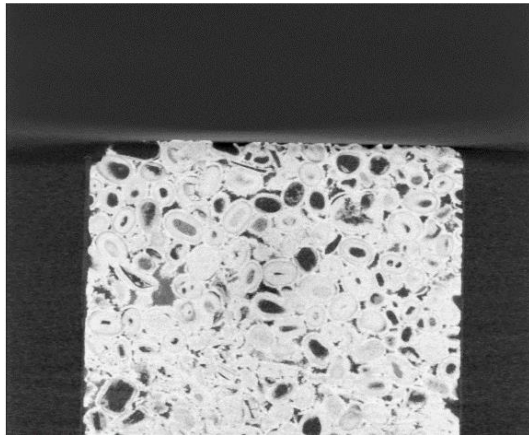


European Research Council  
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PRD-Trigger Grant n°850853

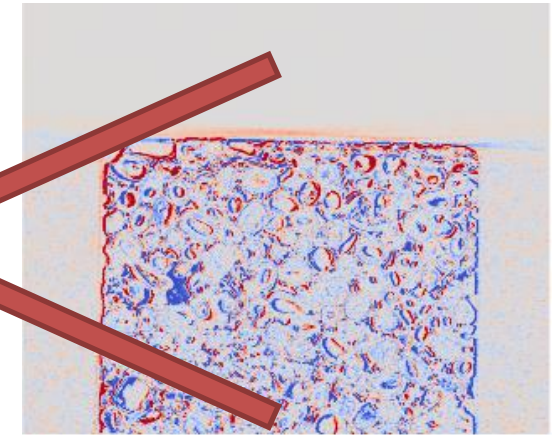
time  $t$

time  $t+\Delta t$

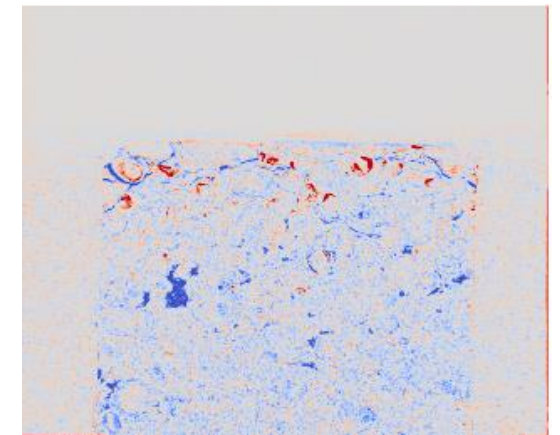


residual field

~~$IM(t+\Delta t) - IM(t)$~~



$IM(t+\Delta t) - IM(t, \Phi(x))$



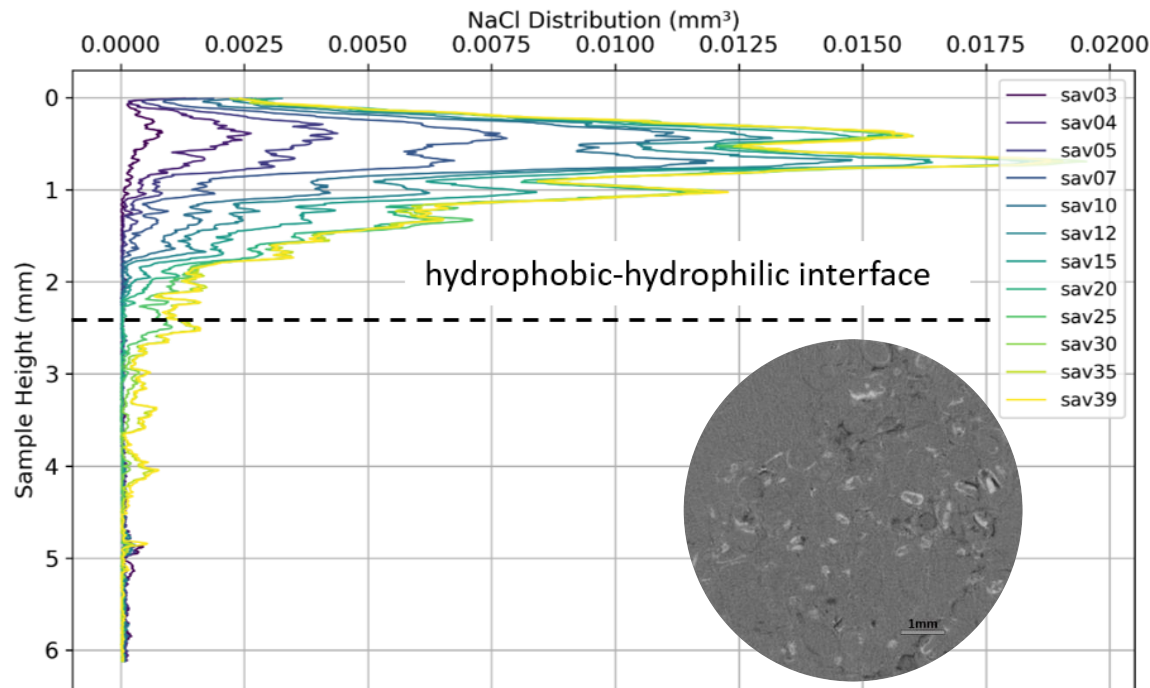
# positive differences = salt crystallization



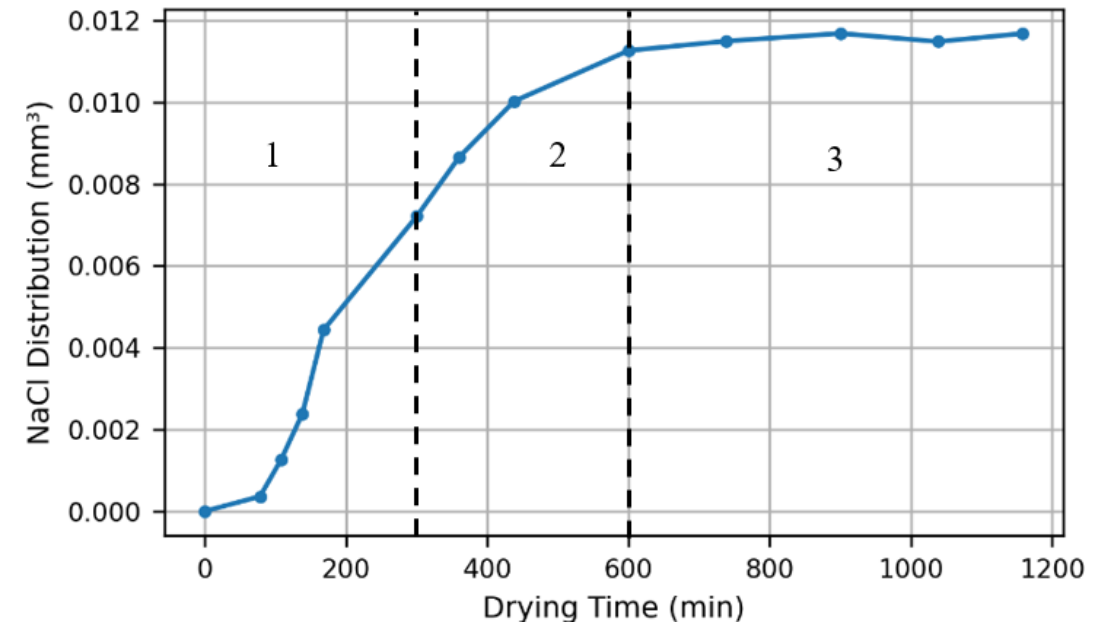
European Research Council  
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## crystal volume over height



## cumulative salt volume



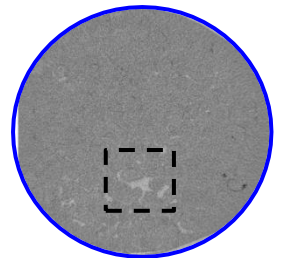
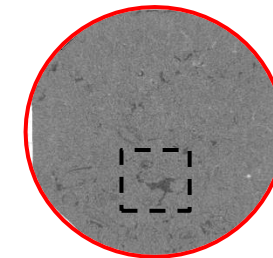
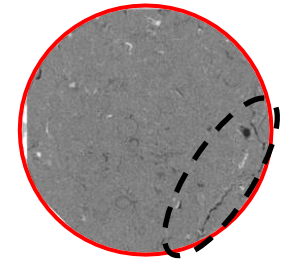
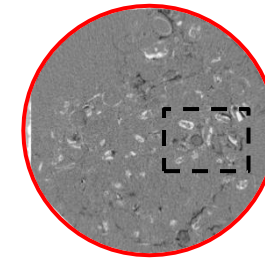
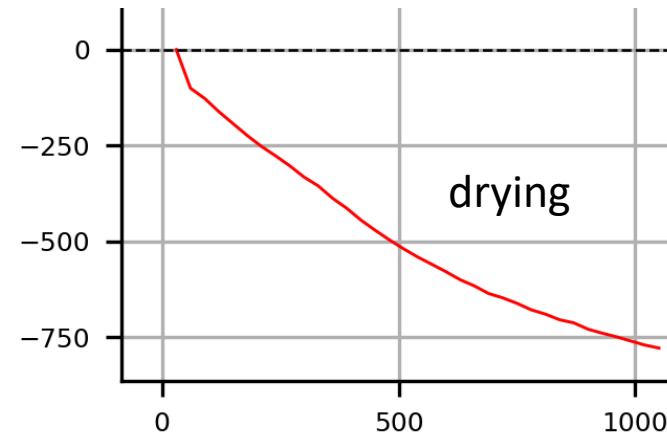
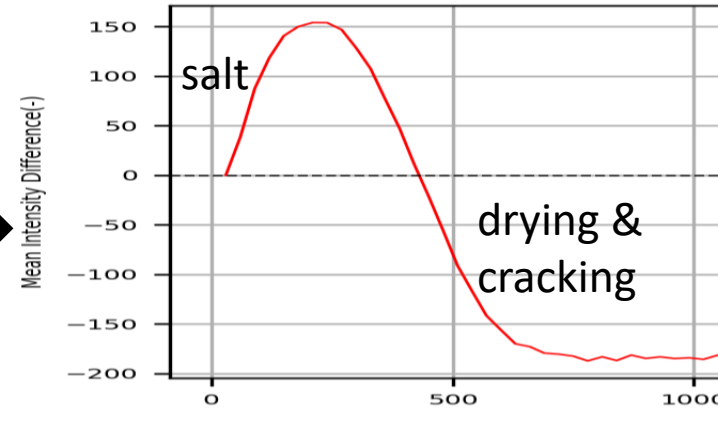
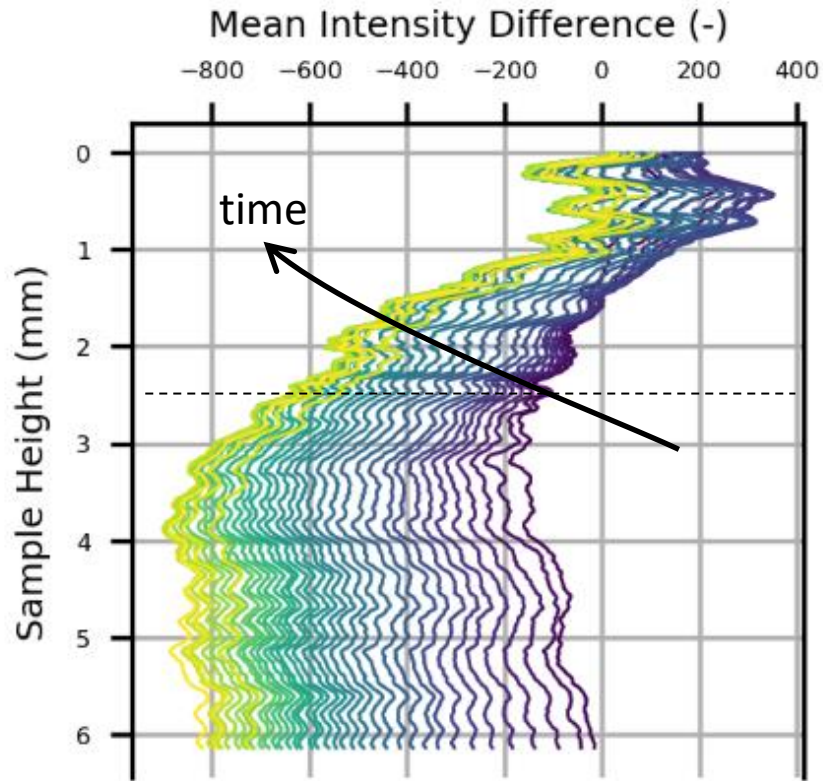
→ enables studying crystallization kinetics and localization

# negative differences = decrease in saturation



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→ enables studying macro-scale transport kinetics

towards a meso-scale analysis

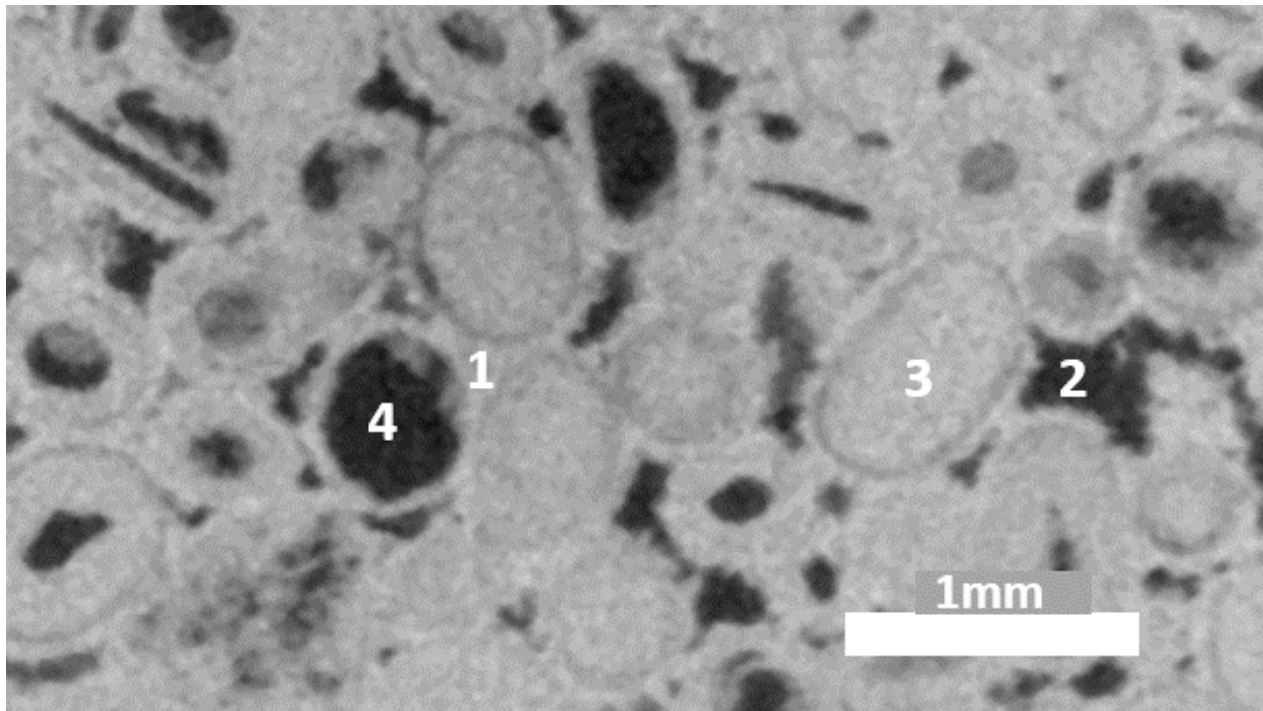
# MACHINE LEARNING

Ben Elhadj Hamida et al. (in preparation)

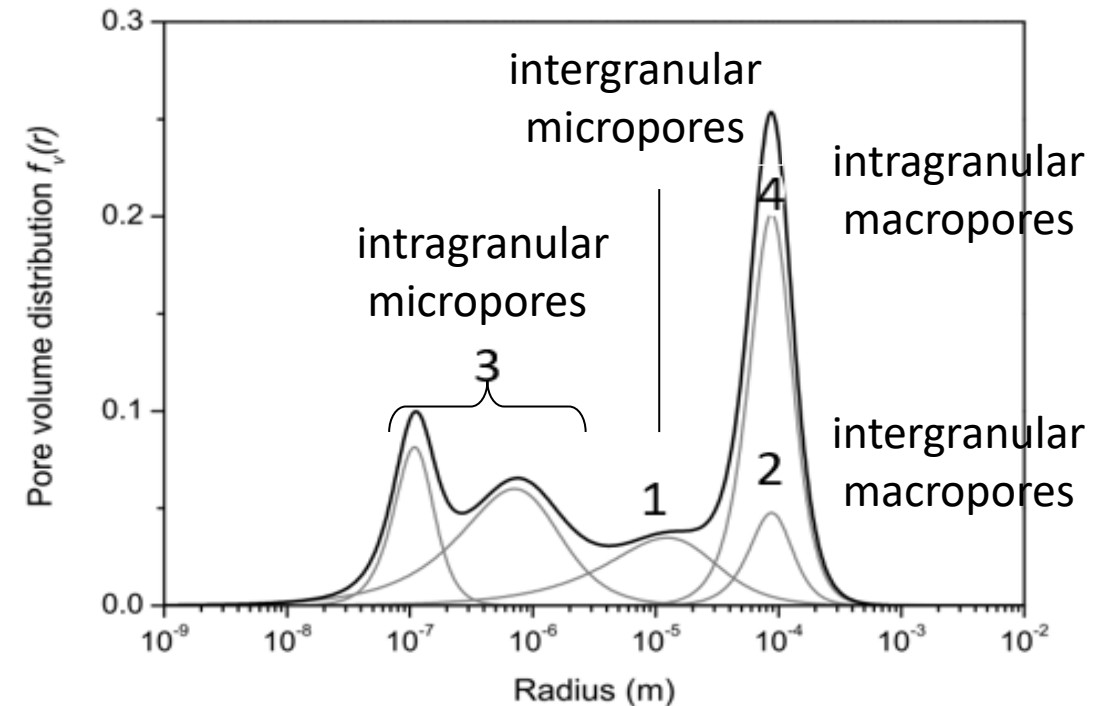


# role of each pore family

representative x-ray slice



real pore size distribution



→ how can we distinguish between 1 and 3 (both unresolved)

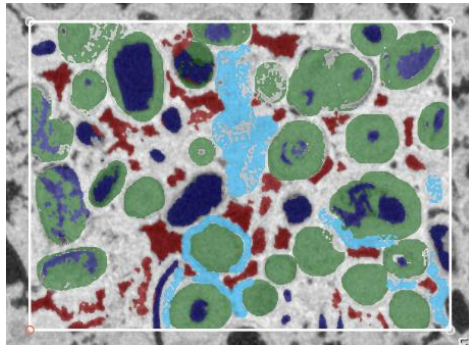
# deep learning segmentation: preparation



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step 1:  
manual labelling

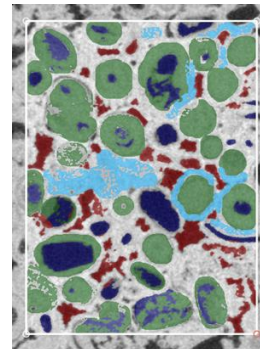


- Intragranular macropores*
- Intergranular macropores*
- Intragranular micropores*
- Intergranular micropores*

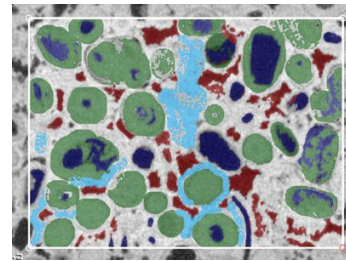
10 slices

step 2:  
data augmentation

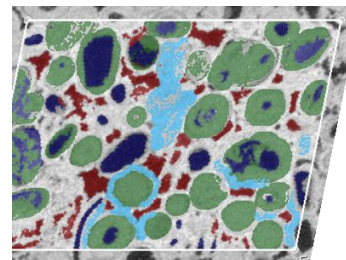
rotate



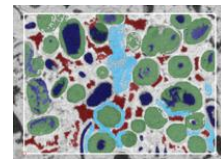
flipping



shearing



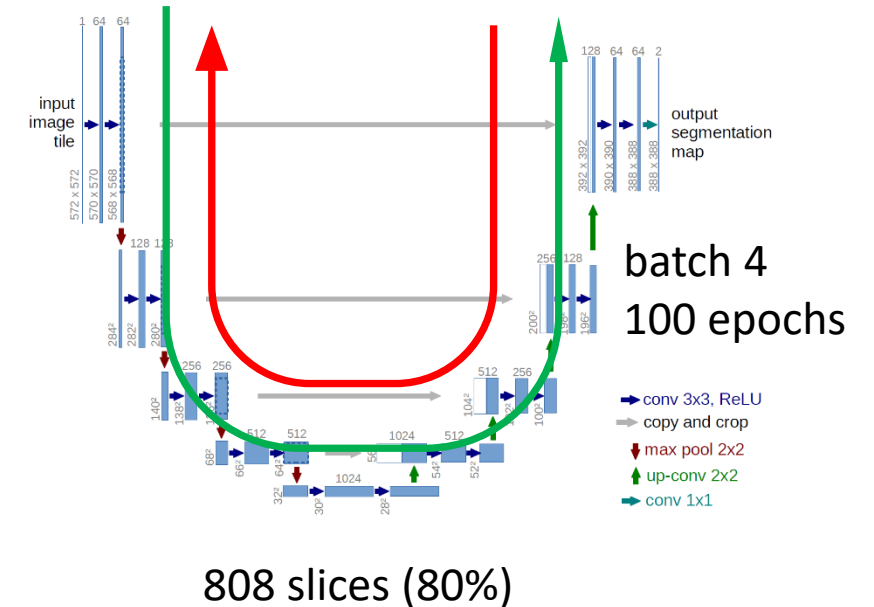
scaling



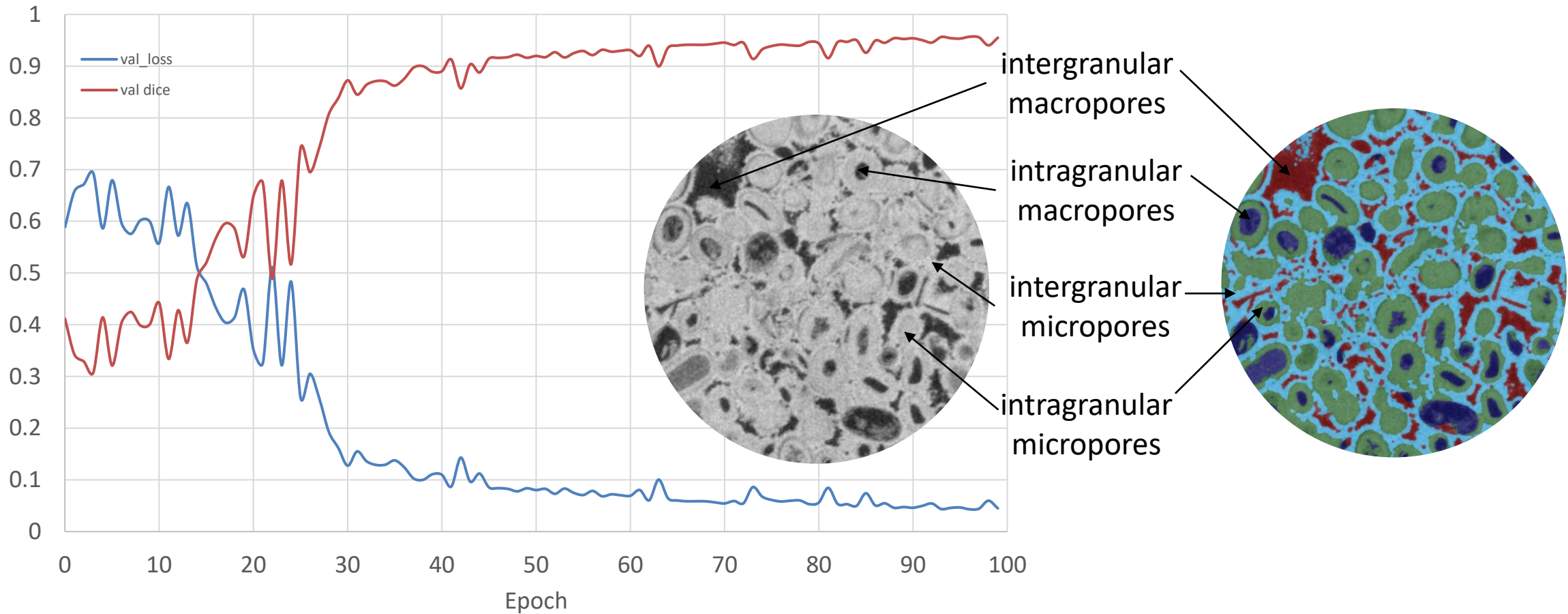
1000 slices

step 3:  
model training

a) forward pass  
b) backpropagation



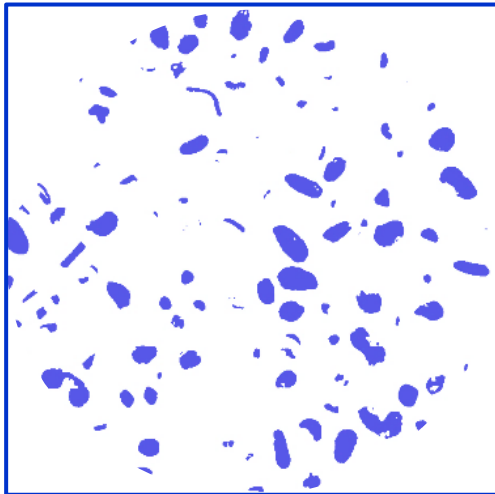
# deep learning segmentation: validation



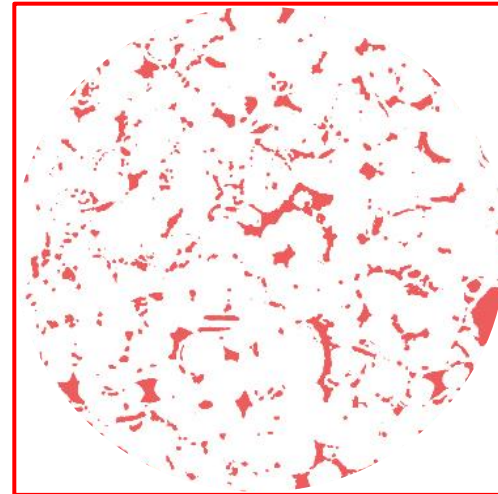


# deep learning segmentation: result

intra-granular  
macropores



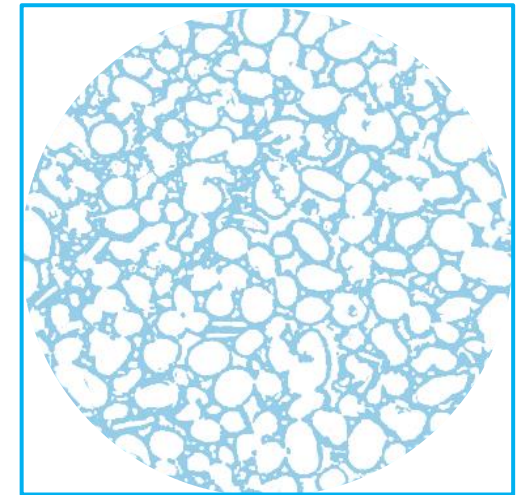
inter-granular  
macropores



intra-granular  
micropores



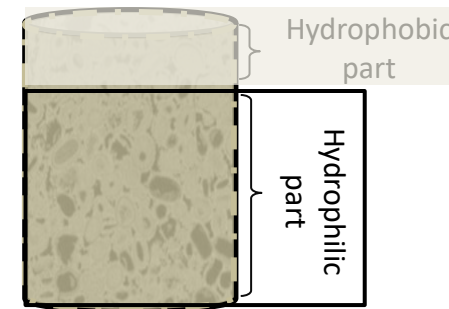
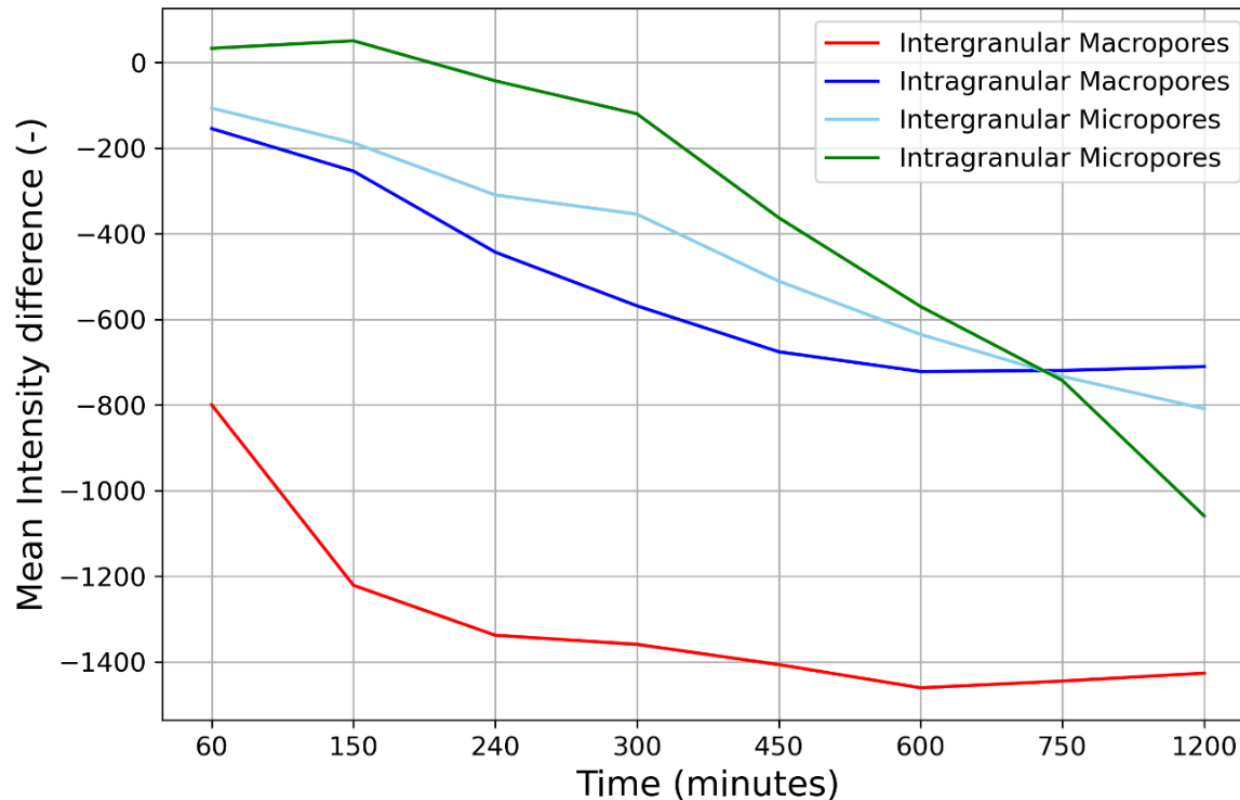
inter-granular  
micropores



- applied on DVC-corrected reference images
- masks for any time step



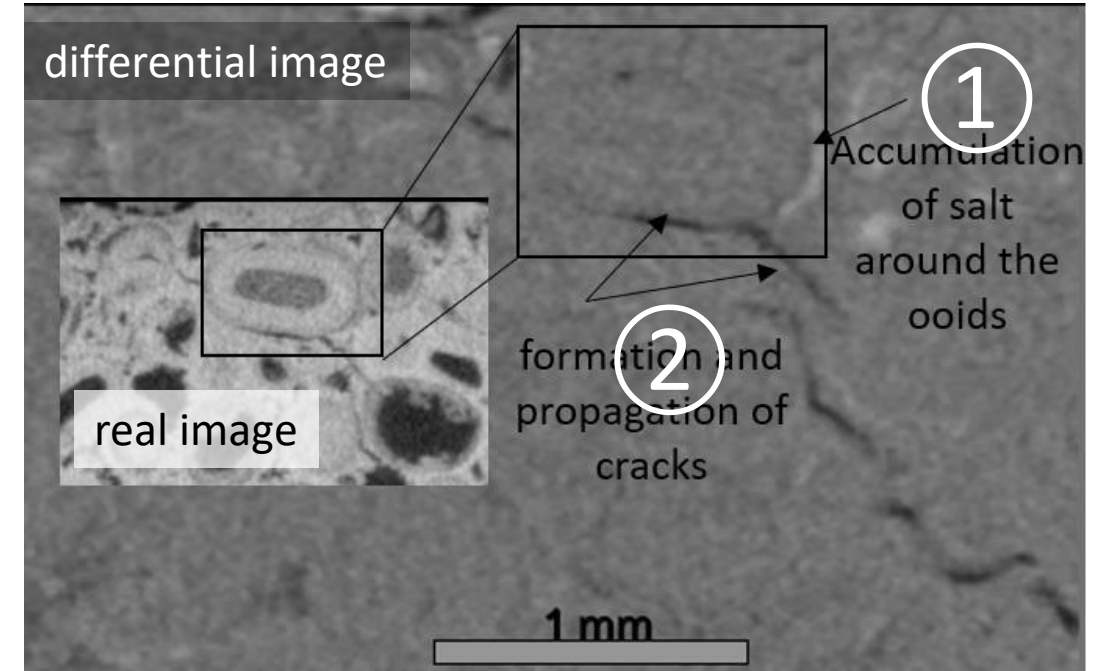
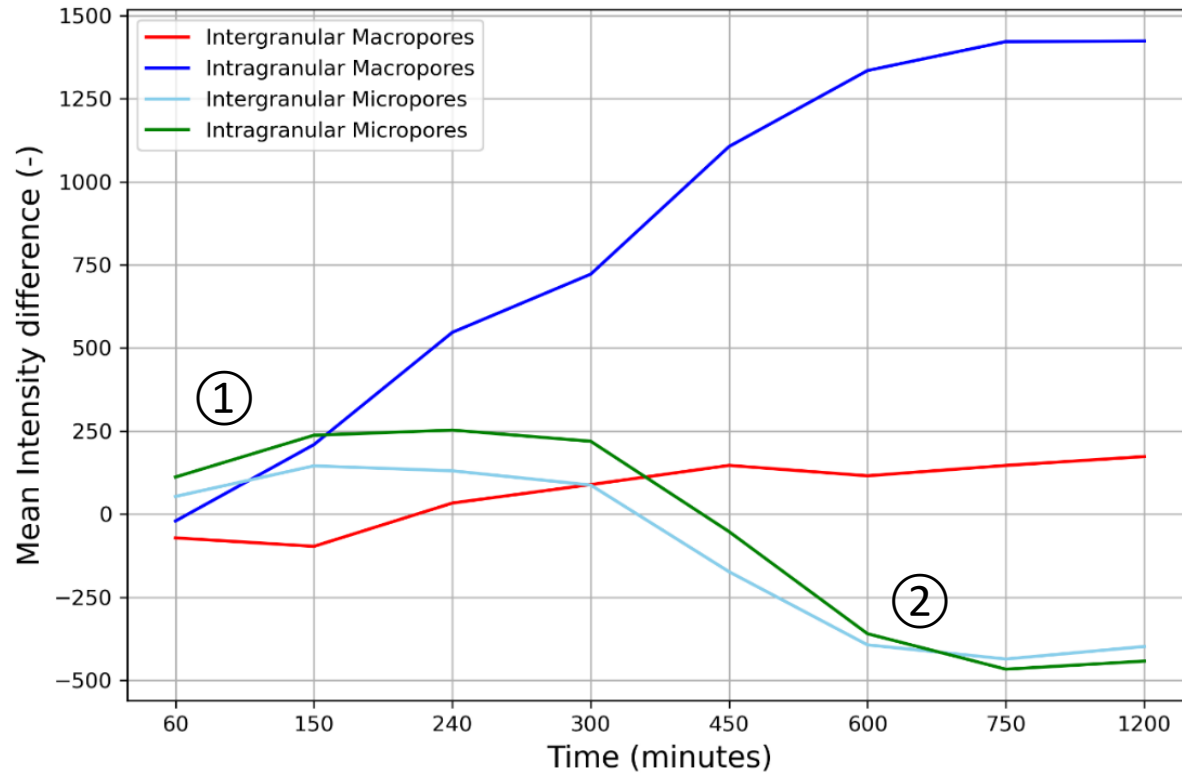
# drying kinetics



drying sequence:  
inter, intra macropores  
inter, intra micropores

→ enables studying transport kinetics *for each pore family*

# salt crystallization



- ① salt deposition at interface  
intra/intergranular micropores
- ② crack initiation around ooids,  
propagating into matrix (intergran. micropores)

calibration-free quantification of grey levels and their changes

# PHYSICS-BASED IMPROVEMENTS

step 1: initial geometry

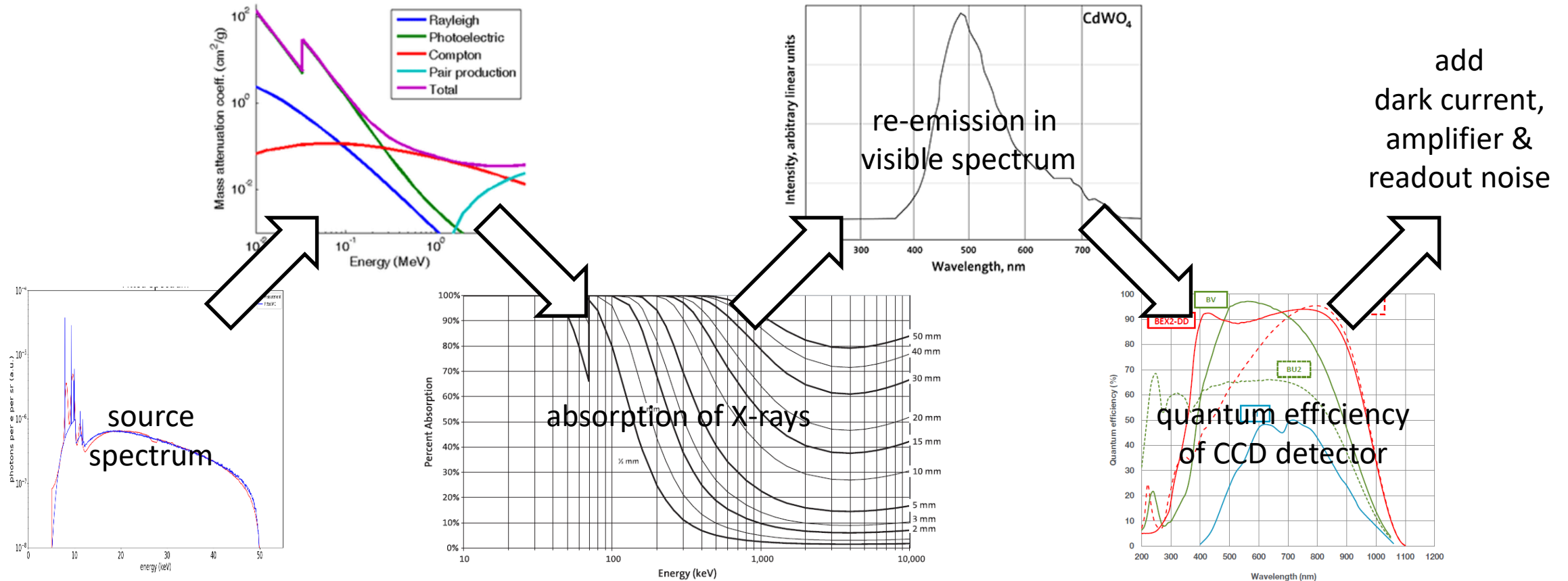
# physics-based forward projector

source

sample

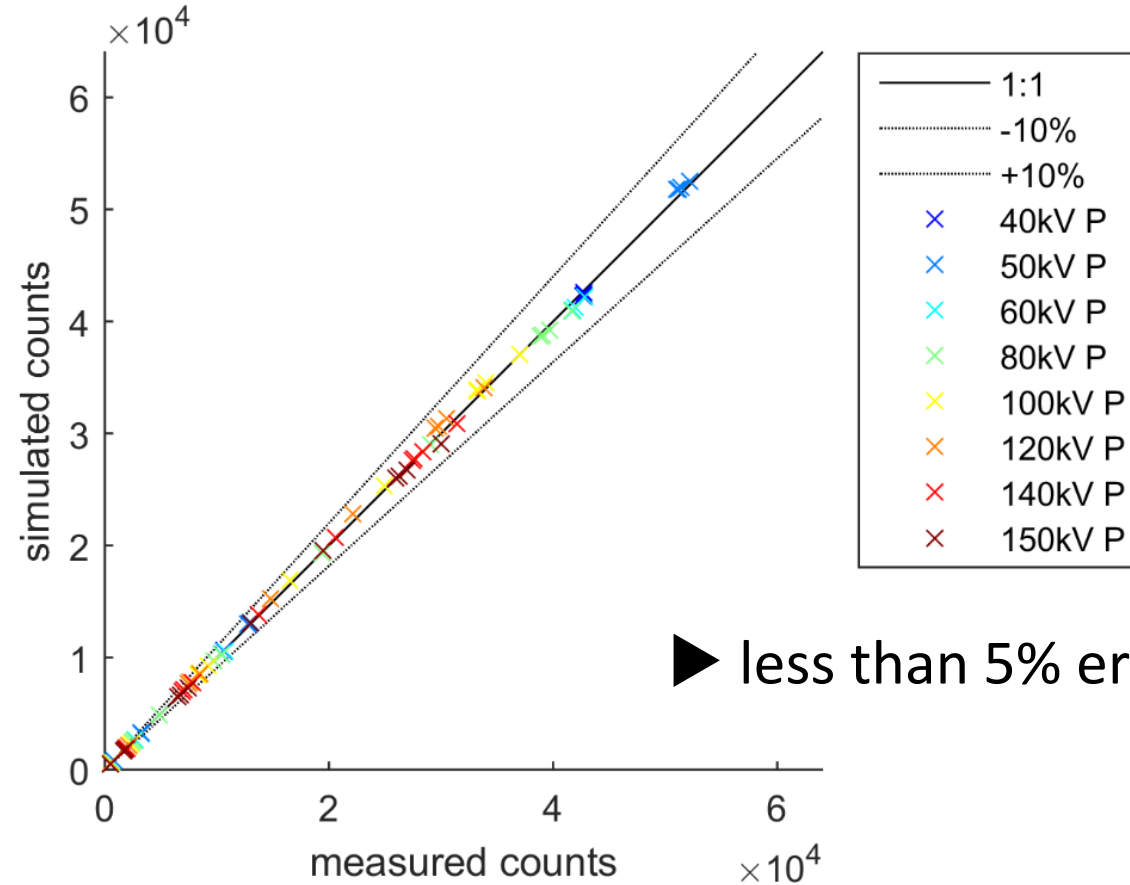
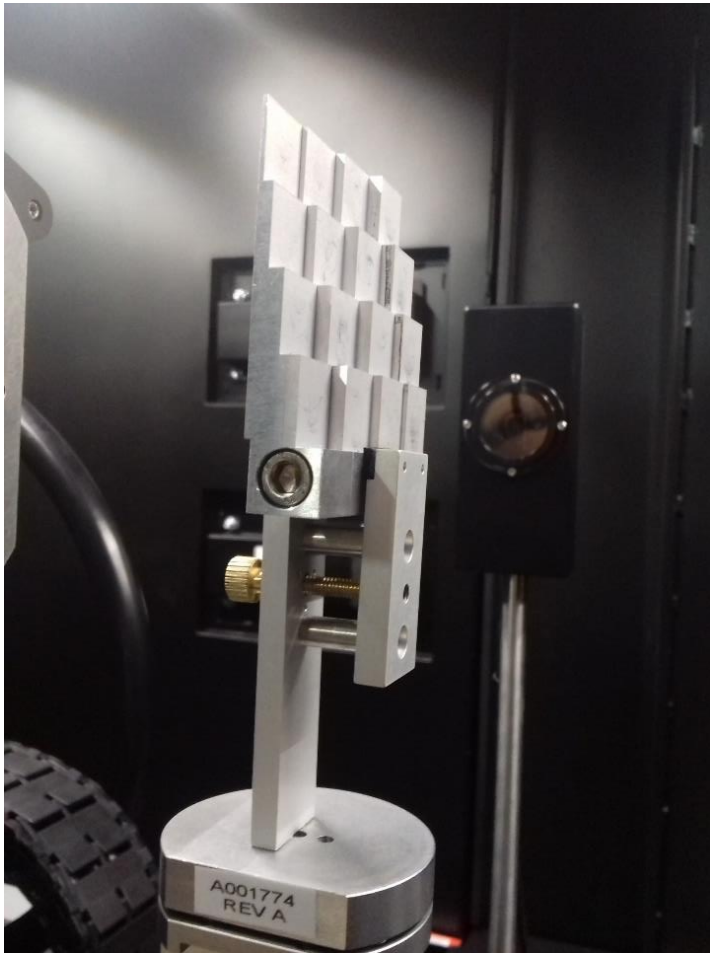
scintillator

CCD camera



# physics-based forward projector

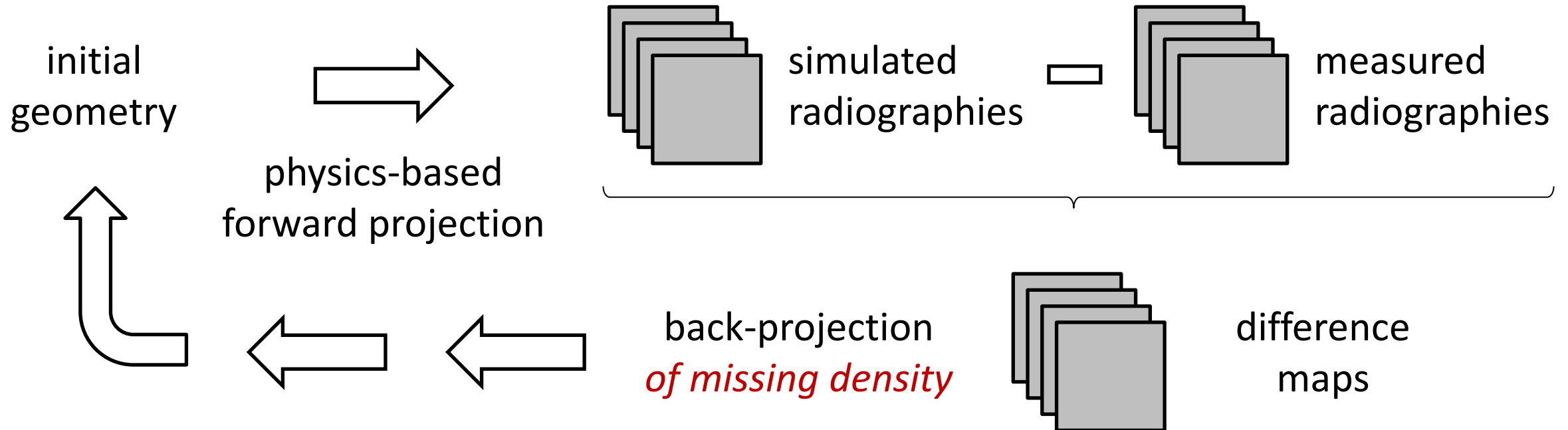
s



► less than 5% error on full range

# iterative reconstruction framework

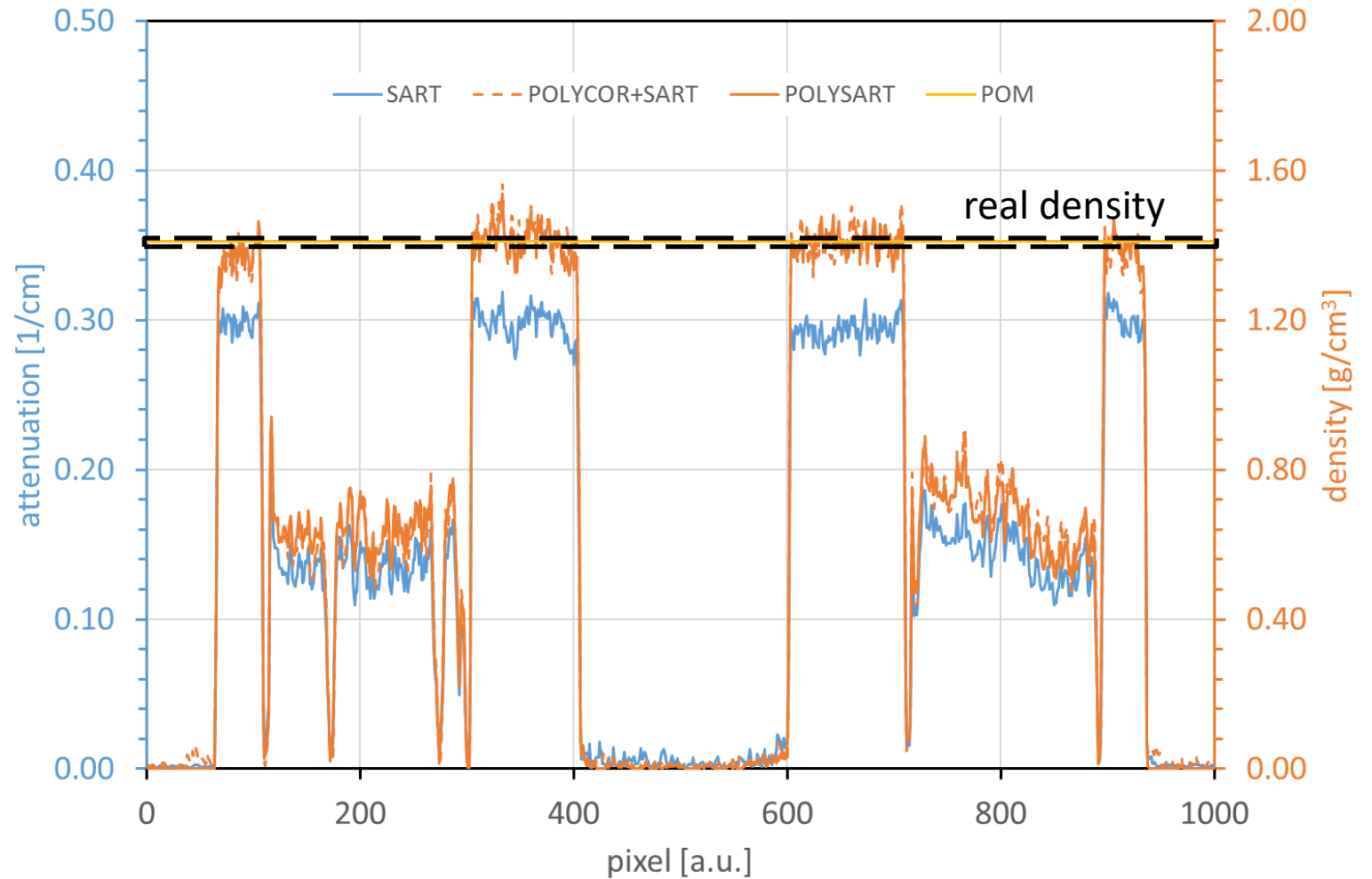
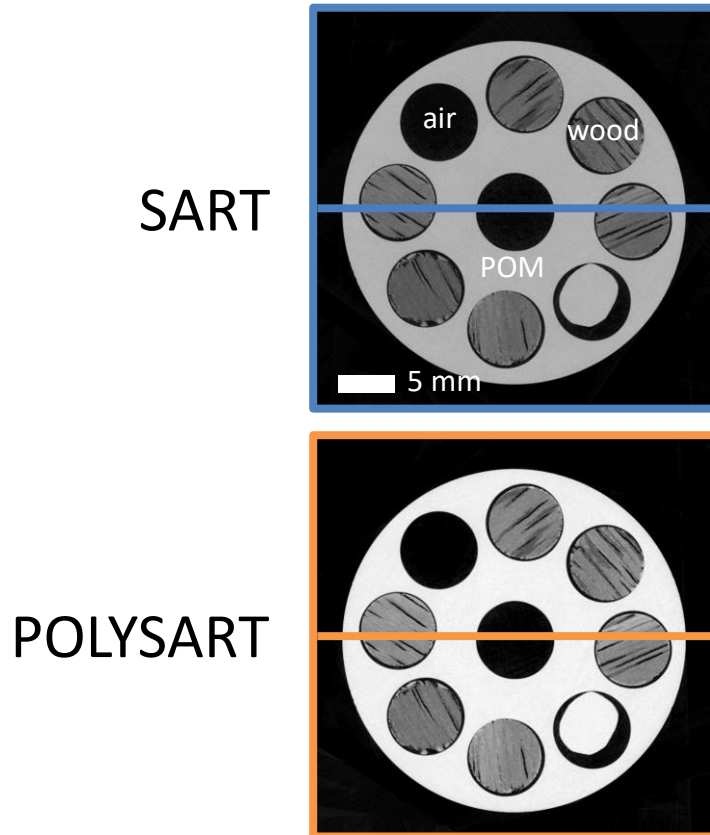
## ► POLYSART (POLYchromatic Simultaneous Algebraic Reconstruction Technique)



Moonen et al. 2021

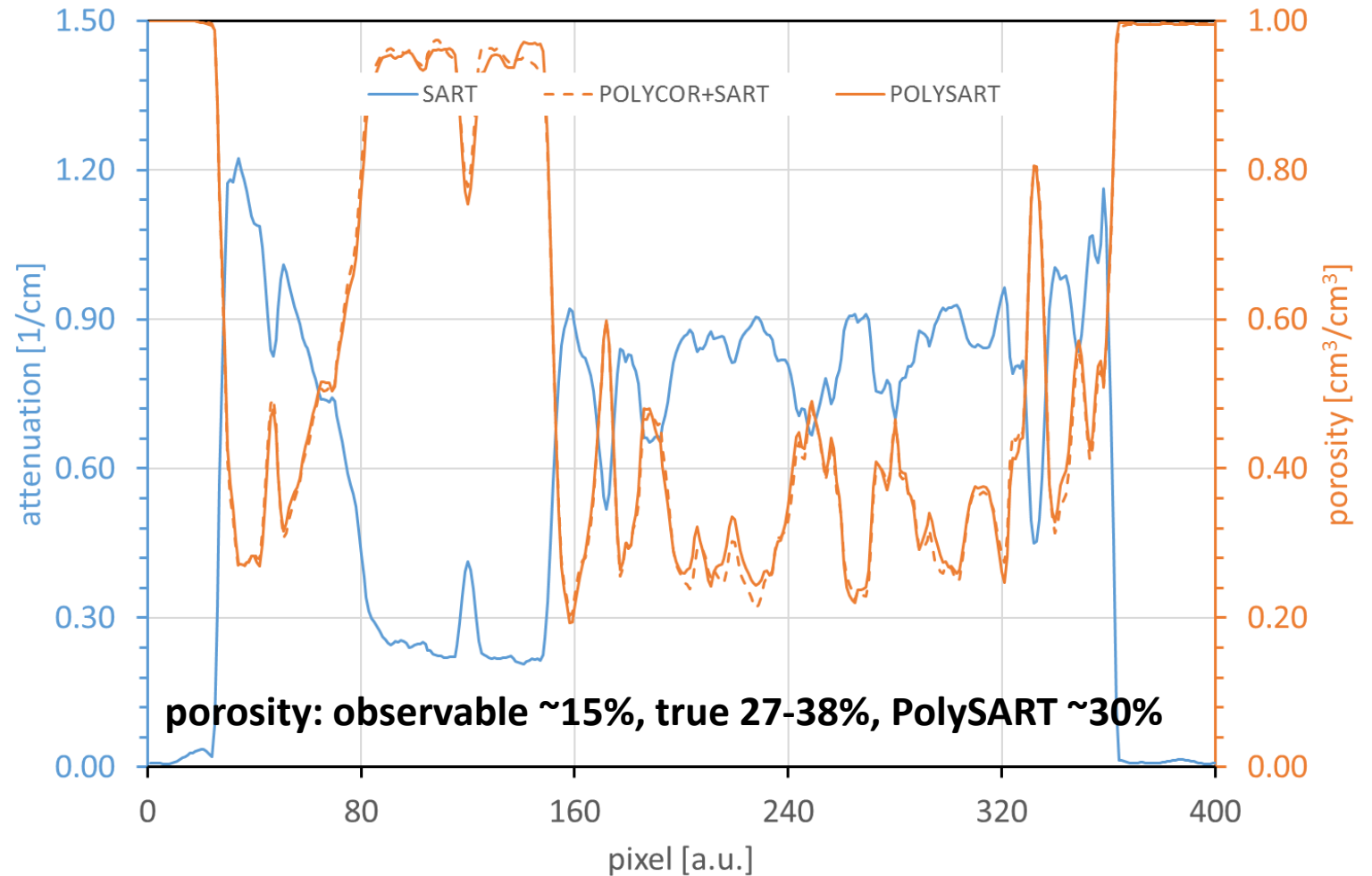
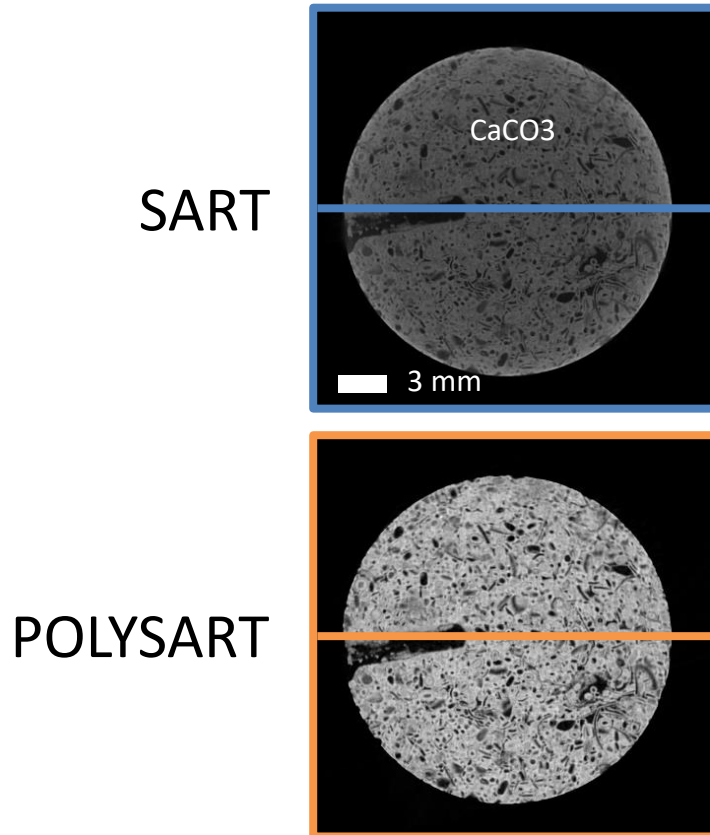


# application 1: densitometry



Moonen et al. 2021

# application 2: sub-resolution porosity



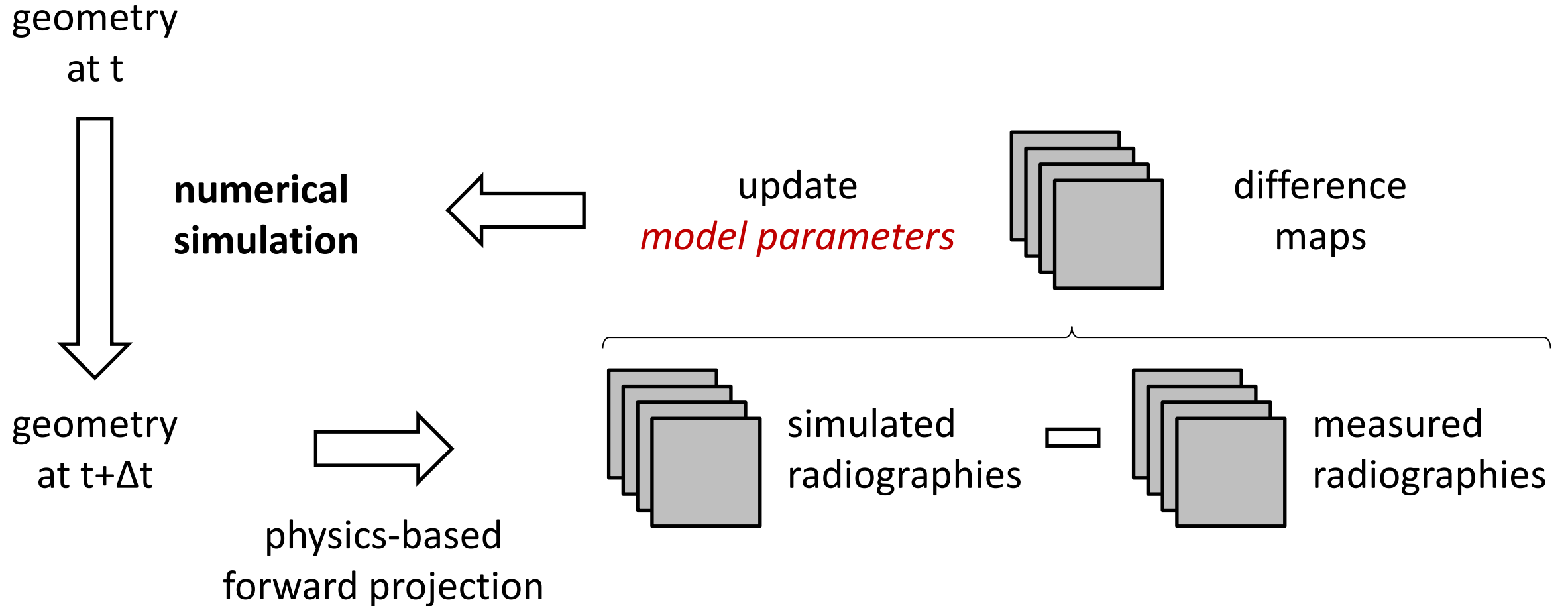
Moonen et al. 2021

**calibration-free quantification of grey levels and their changes**

# **PHYSICS-BASED IMPROVEMENTS**

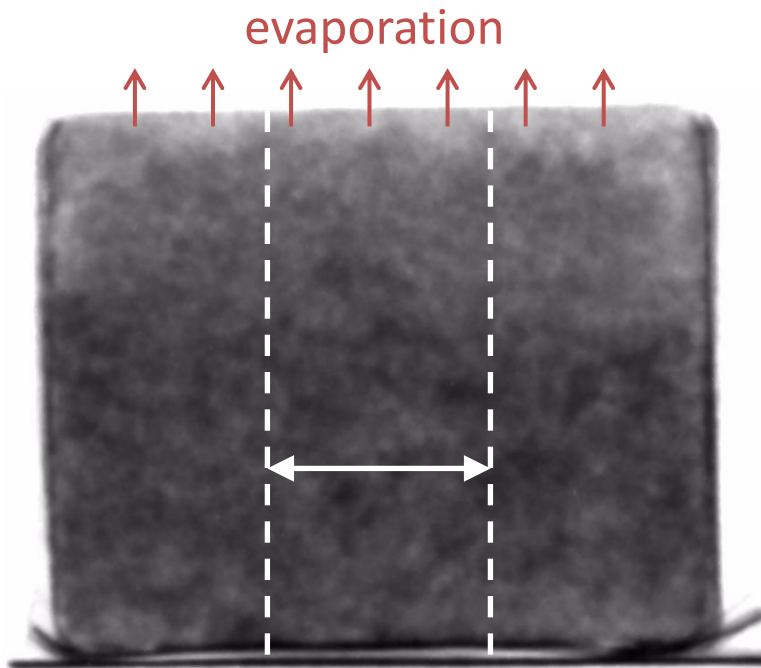
**step 2: temporal evolution**

# physics-based registration

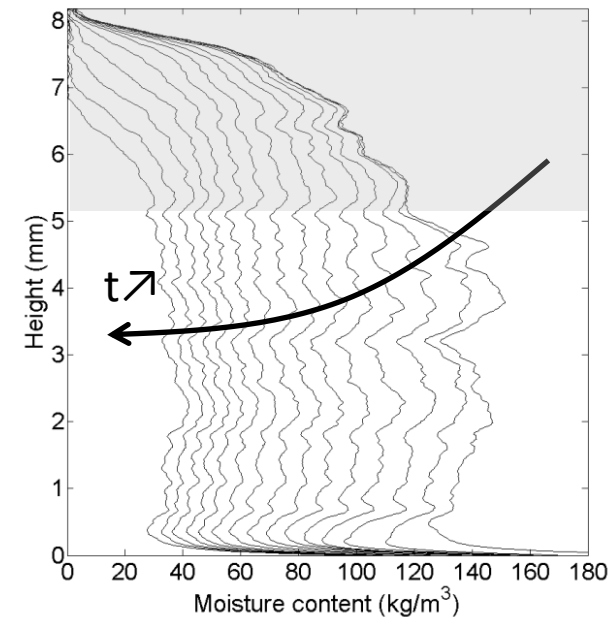


# (A) experiment: evaporative drying

neutron radiography @ SINQ

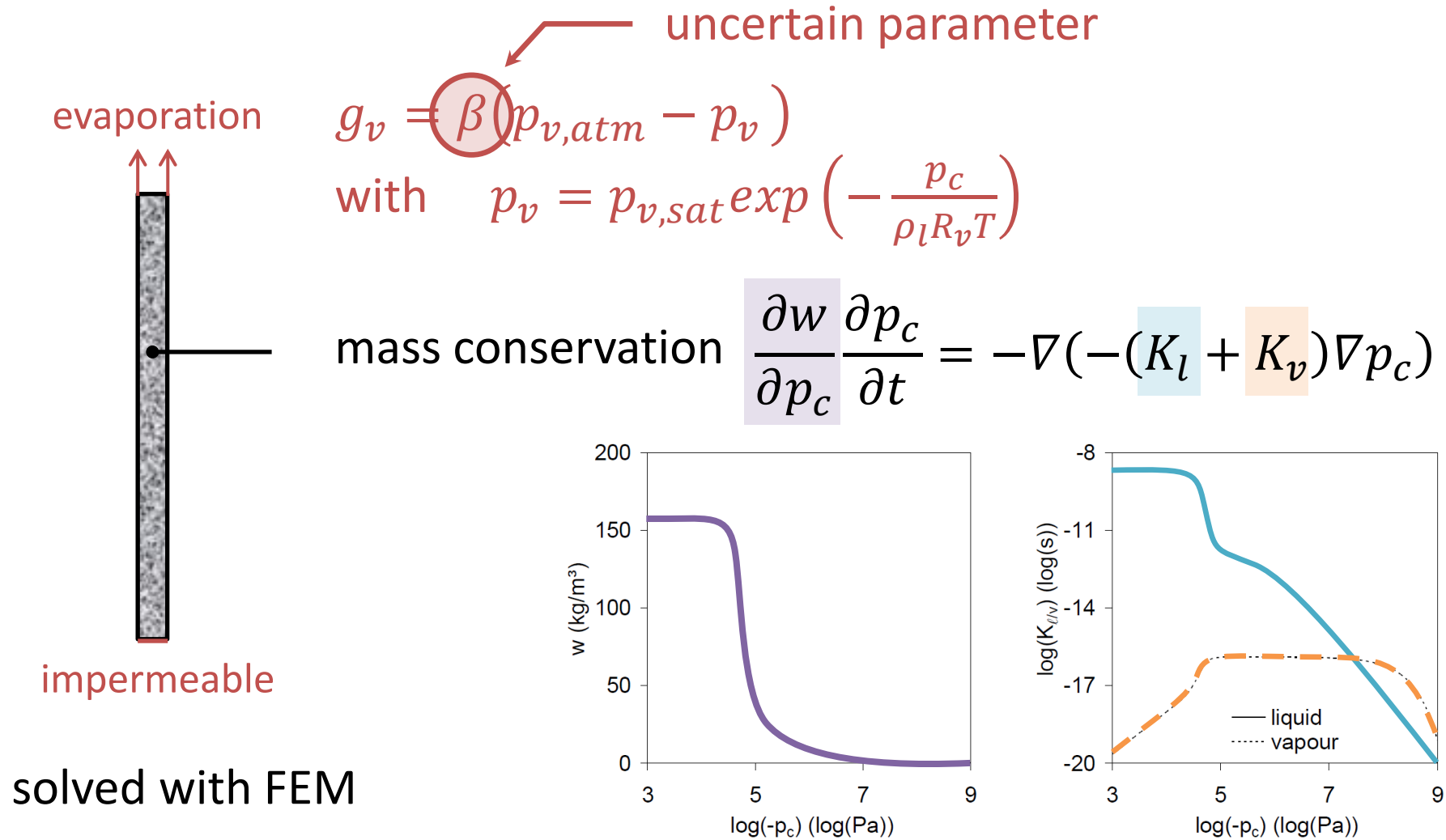


post-processing with QNI



experimental details in Derluyn 2012

# (B) simulation (toy-)model





# (C) « force to match »

## trial-and-error

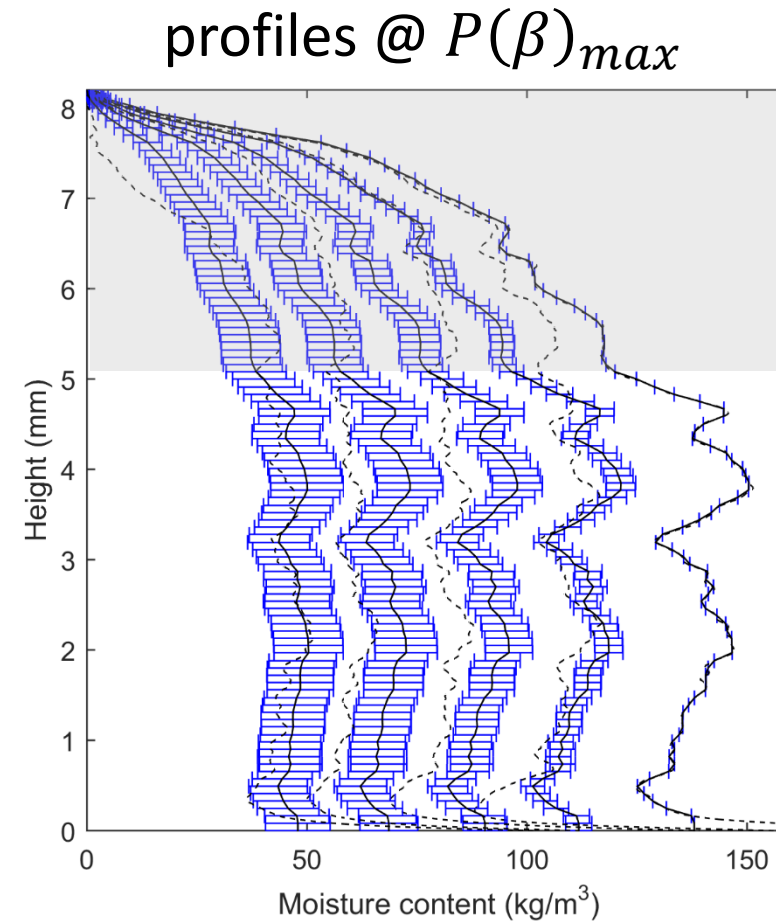
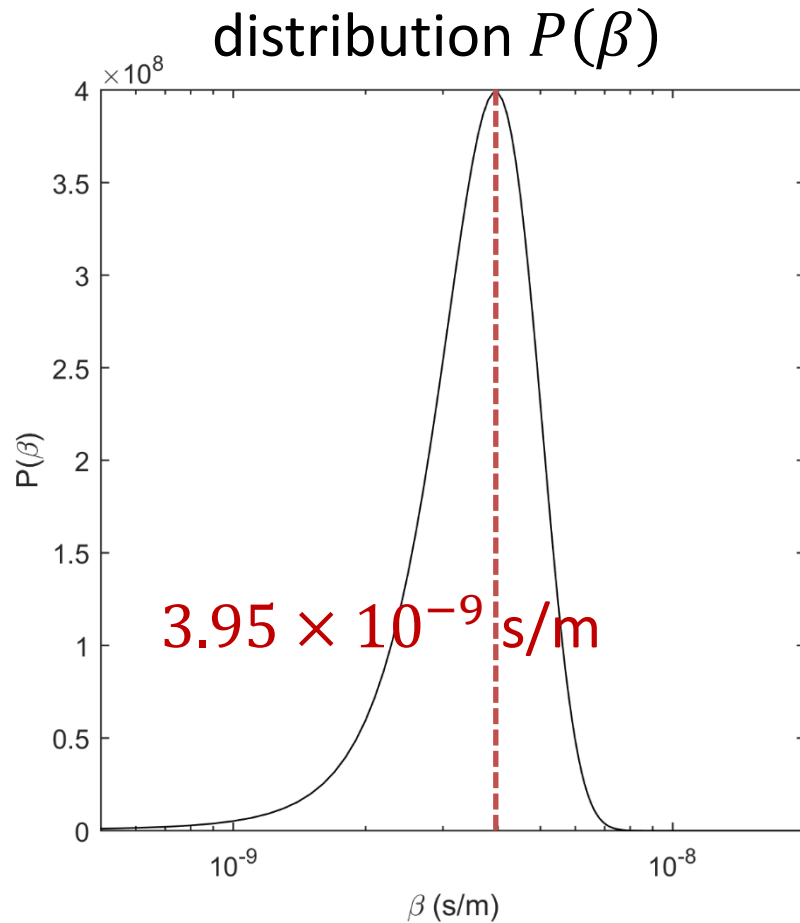
- ▶ run model for various values  $\beta$  until a “good” value is found
  - result:  $\beta$
  - manual or automatic (e.g. Levenberg-Marquardt)
  - cost  $\sim$  number of tested values

## Bayesian calibration

- ▶ run model for  $m$  values of  $\beta$ . Calibration yields distribution
  - result:  $P(\beta)$
  - automatic (e.g. MCMC by Metropolis-Hastings)
  - cost =  $m$  runs

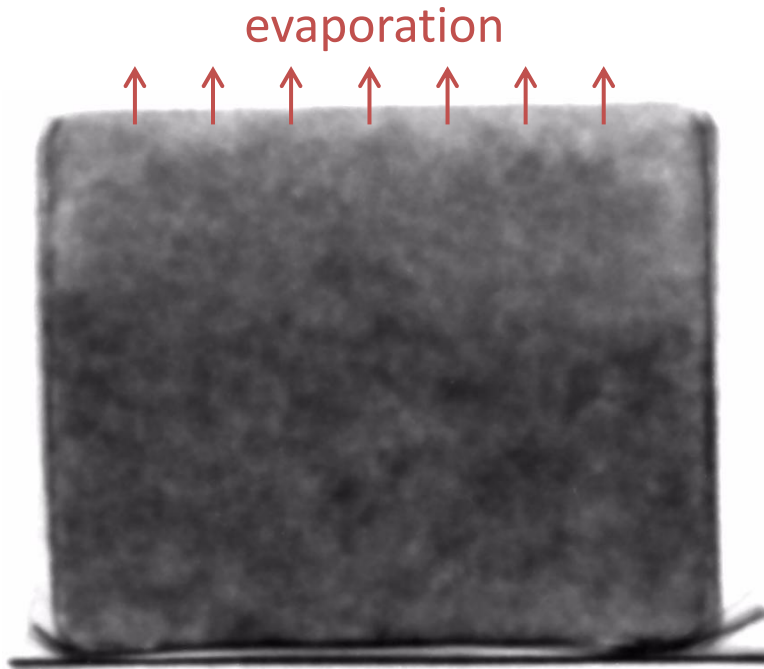
+ accounts for noise and bias

# results: unknown transfer coefficient $\beta$

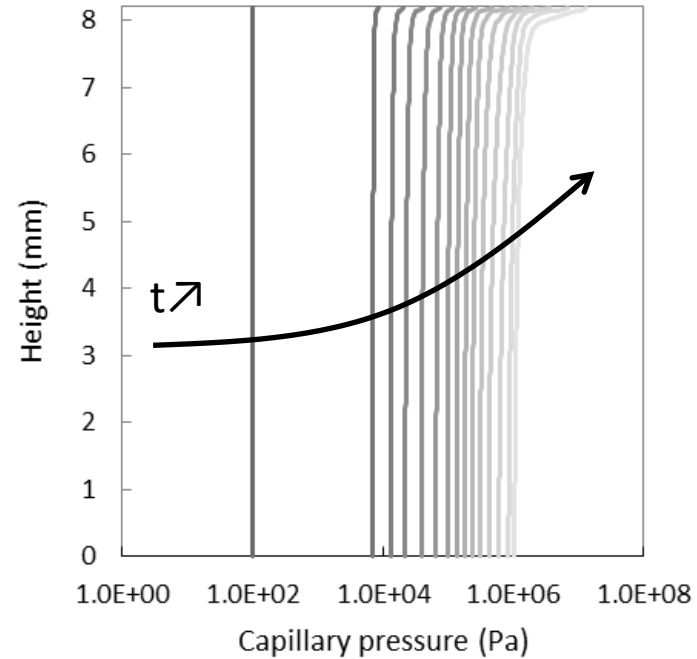


# results: pressure profiles

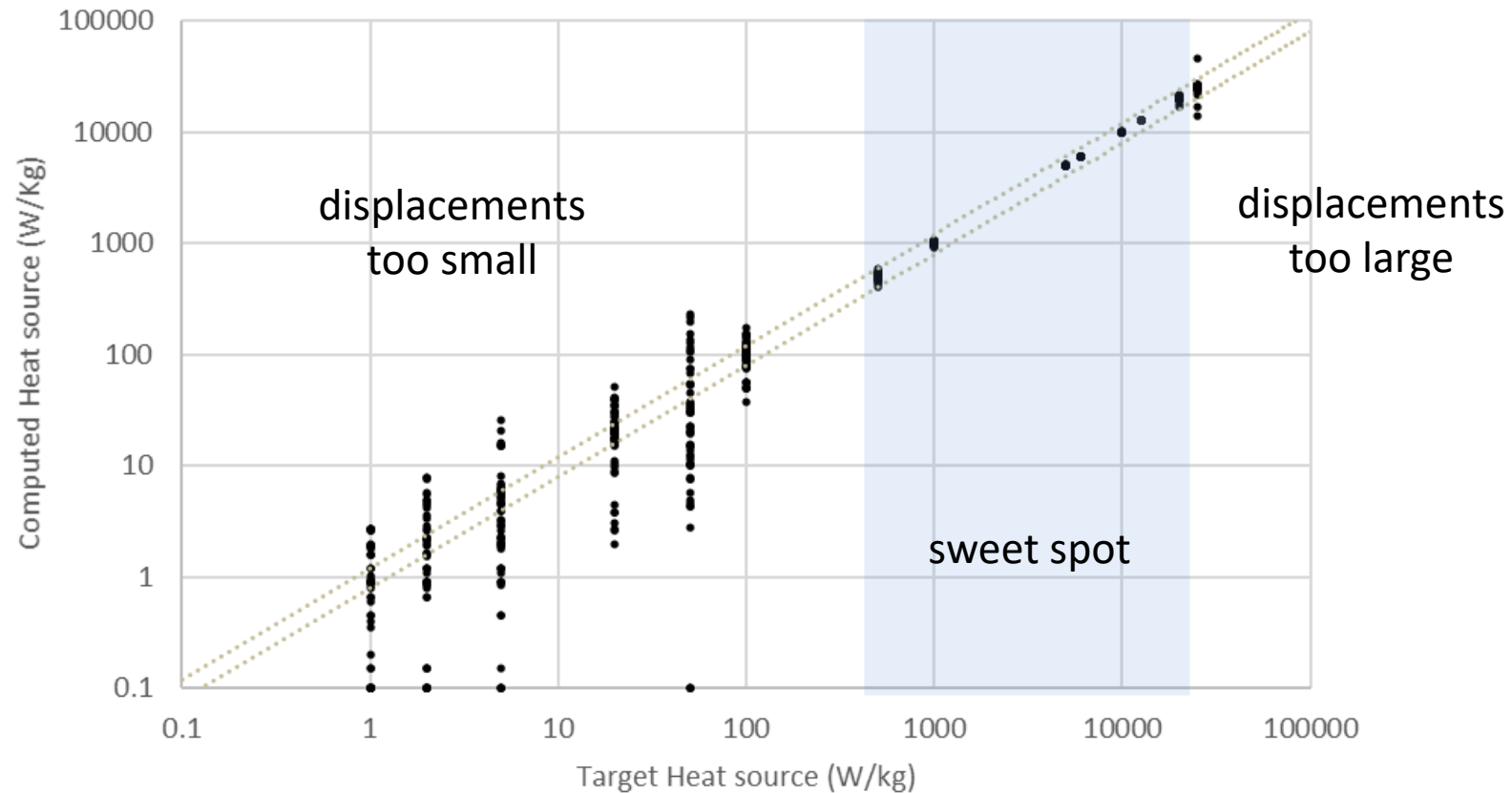
neutron radiography @ SINQ



most likely pressure profiles



# similar approach used to estimate reaction heat



Ait Hamouda et al. (in preparation)

# CONCLUDING REMARKS



# toolbox for image analysis (1/2)

- ▶ Digital Volume Correlation (DVC)  
strains, regions with cracking
- ▶ DVC-corrected differential imaging  
macro-scale kinetics of crystallization and transport
- ▶ Machine Learning segmentation  
meso-scale kinetics of crystallization and transport



# toolbox for image analysis (1/2)

- ▶ Polychromatic reconstruction (PolySART)  
calibration-free density and porosity estimates at sub-voxel scale
  
- ▶ Physics-based registration
  - identification of uncertain model parameters
  - identification of missing physics (via bias term)
  - yields invisible parameters (pressure, temperature, stress, ...)

→ enables true analysis of coupled processes in porous media

# acknowledgements

- ▶ **Syrine Ben Elhadj Hamida**

currently postdoc at UPPA, performed DVC, DVC-corrected differential imaging and ML-based meso-scale analysis

- ▶ **Hannelore Derluyn**

acquired the time lapse Savonnières datasets with X-rays and neutrons  
main supervisor of Syrine Ben Elhadj Hamida

- ▶ **Jelle Dhaene**

former postdoc at UPPA, programmed and validated PolySART

# thank you for your attention

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Director of the Institute of Pluridisciplinary Research and its Applications (IPRA)

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<http://imagingcentre.univ-pau.fr/>