Northern Lights on Food Masterclass

IMAGING MICROSTRUCTURES OF FOOD AND PACKAGING WITH X-RAYS AND NEUTRONS

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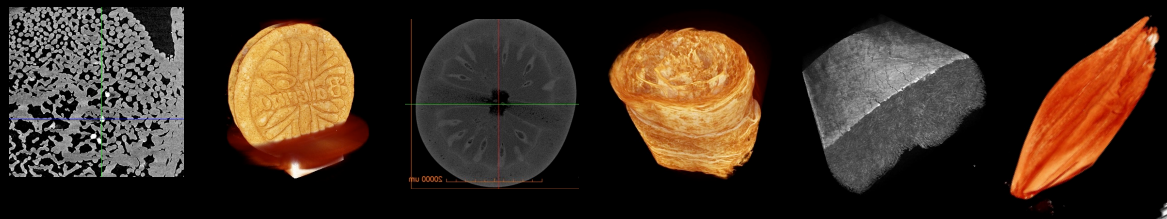
& 4D Imaging Lab @Lund University



4D IMAGING LAB

LUND INSTITUTE OF ADVANCED NEUTRON AND X-RAY SCIENCE LINXS

Why do x-ray and neutron imaging?...
what's happening inside?...

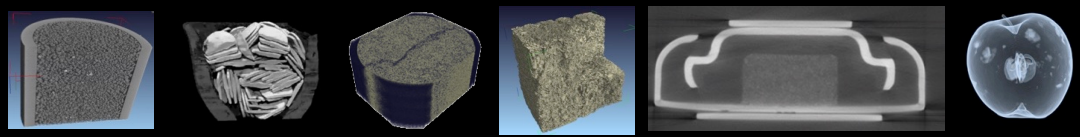
By normal visual inspection, we have no idea…

Why do x-ray and neutron imaging?...
what's happening inside?...

Dynamic neutron radiography

Objectives:

- Introduce neutron and x-ray imaging
- Describe some of the details of x-ray and neutron tomography
- Demonstrate the usage of x-ray and neutron imaging with applications to food and packaging



History

RADIOGRAPHY

- Discovery of X-rays by Wilhelm RÖntgen
- First radiographies in 1895
- First Nobel prize in Physics in 1901

Hand mit Ringen (Hand with Rings): print of Wilhelm Röntgen's first "medical" X-ray, of his wife's hand, taken on 22 December 1895 and presented to Professor Ludwig Zehnder of the Physik Institut, University of Freiburg, on 1 January 1896

(http://en.wikipedia.org/wiki/X-ray)

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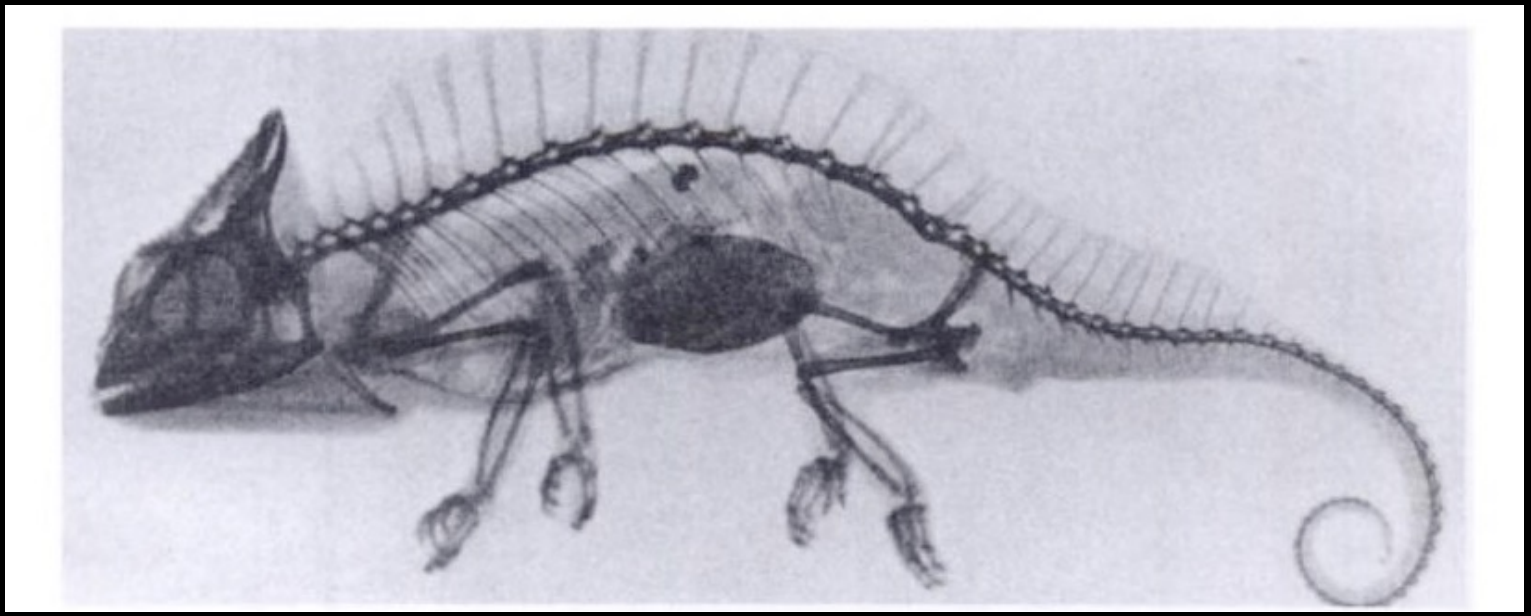


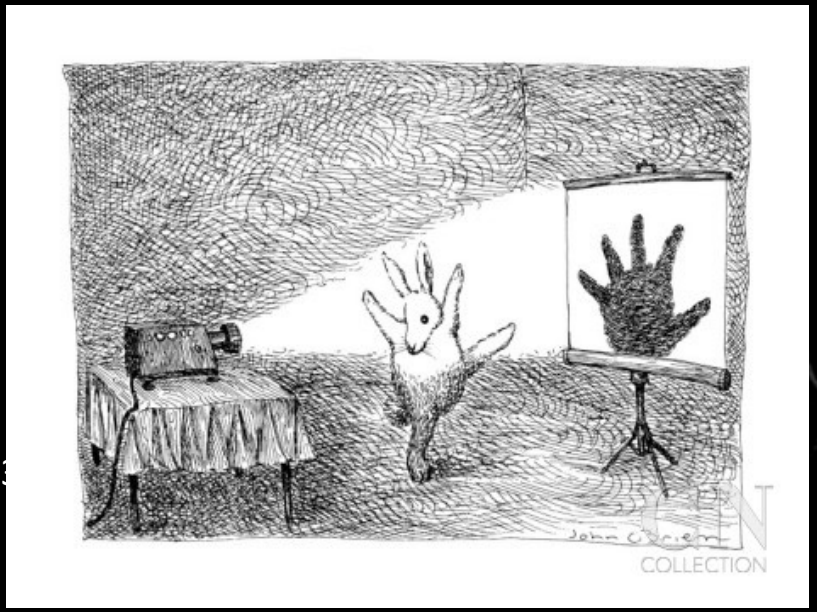
February 1896 - African chameleon

2D → 3D imaging

radiography vs. tomography

when the detail to be observed (here, a fracture) is in the plane of the radiography, this can be sufficient (for medical diagnosis in this case)

But this can be misleading…



When things must be seen in 3
radiography is not sufficient

History

TOMOGRAPHY

- Tomography = “extension” of radiography
- The original mathematical framework by Radon in 1917
- First X-ray CT scanner in 1972 by Hounsfield in collaboration with Cormack
- Nobel prize in Medicine in 1979

Godfrey N. Hounsfield with a medical “CT” scanner
(www.betterproductdesign.net)

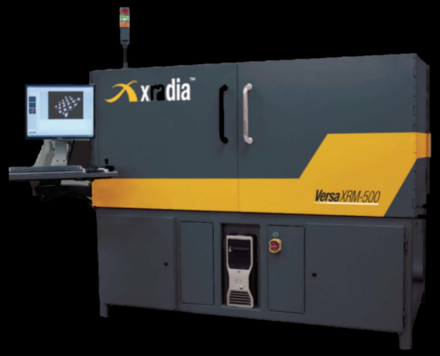
X-ray tomography machines

X-RAY SOURCE + DETECTOR + SYSTEM FOR THE MULTI-ANGLE MEASUREMENT

3 main types of machines for X-ray tomography:

Medical (“CT”), lab/industrial and synchrotron

Medical scanner
e.g., LBIC



Lab/Industrial scanner
e.g., 4D Imaging Lab

Synchrotron
e.g., MAXIV

X-RAY TUBE

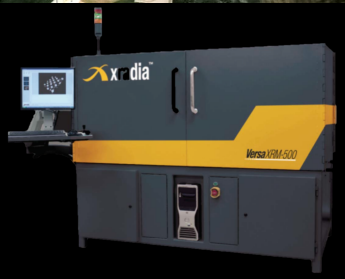
ACCELERATED ELECTRONS

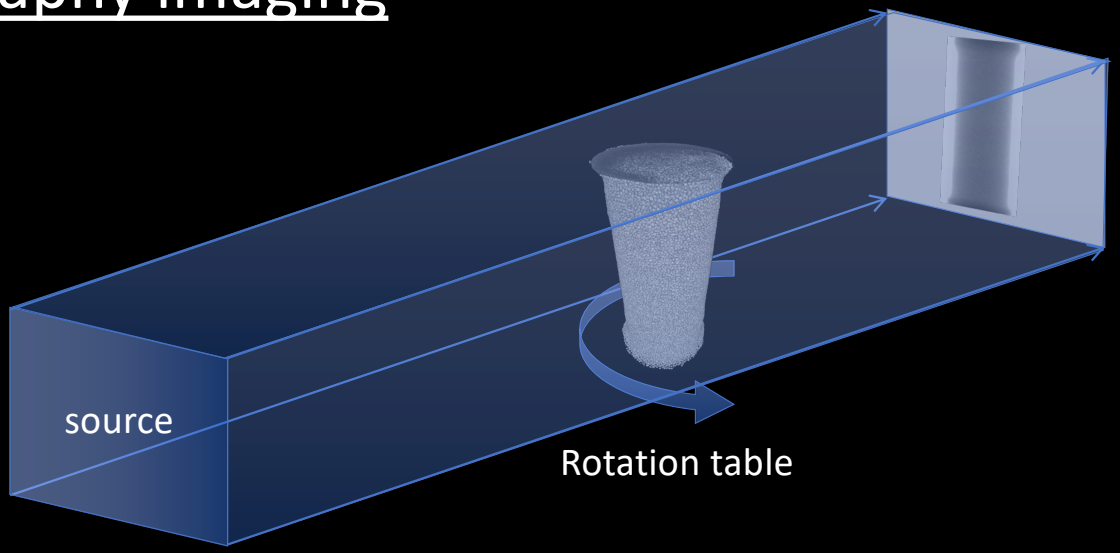
Comparison between X-ray tomography machines

Type of tomograph	Medical	Industrial/lab	Synchrotron
X-ray source	X-ray tube		Accelerator of electrons
Geometry beam	Fan or cone beam		Parallel beam
Resolution	100-500 µm	0.5-100 µm	0.1-10 µm
Object size	dm	mm-cm	µm-mm
Scanning time	sub.-sec to mins	10's mins to hrs	seconds to mins

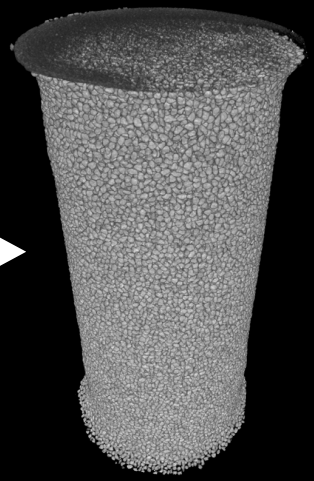
- Best resolution with industrial/lab scanners and synchrotron.
- Best scanning time with medical scanners and synchrotron.
- Synchrotron - Best conditions for slice reconstruction (i.e. parallel and monochromatic beam) BUT generally small specimen size and difficult to obtain experiment time.
- Note that rapid advances are being made (30-50 nm in one lab tomograph (Xradia) but for VERY small samples) and MAXIV, NINA-ESRF…

Full field Tomography imaging





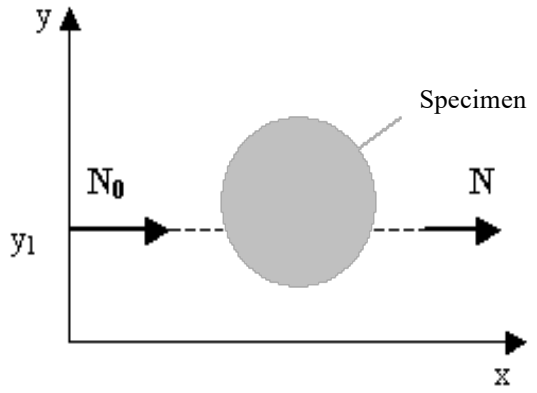
Reconstruction



Principle of transmission measurements (x-rays)

X-ray tomography uses the fact that X-rays are attenuated when they go through an object: one part is scattered, an another absorbed and the rest transmitted.

Beer-Lambert attenuation law



$$N = N_0 e^{-\left(\int_r \mu(x,y_1)dx\right)}$$

N_0 number of emitted photons
N number of recorded photons
μ linear attenuation coefficient

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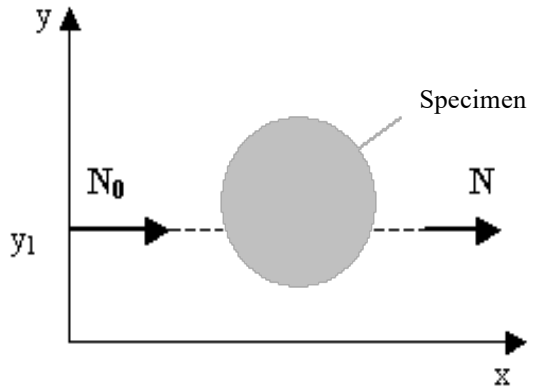

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N_0 number of emitted photons
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$$\mu = \rho\left[a\frac{Z^m}{E^n A} + b\frac{Z}{A}\right]$$

Photoelectric absorption *Compton scattering*

E : X-ray energy
ρ : mass density
Z : atomic number
A : atomic weight
m,n, a and b : constants

Principle of transmission measurements (x-rays)

Linear attenuation coefficient

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m,n, a and b : constants

$$\mu = \rho\left[a\frac{Z^{3.8}}{E^{3.2}} + b\right]$$

As Z/A is roughly constant and m and n can be determined for most of the materials and energies used

Linear attenuation coefficient

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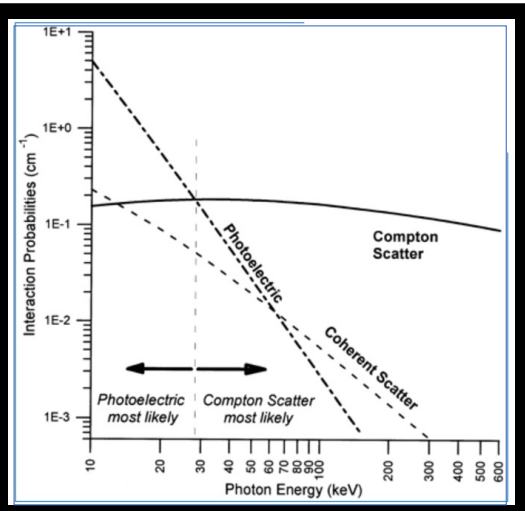
Photoelectric absorption *Compton scattering*



E < 30 keV → Photoelectric absorption is predominant so :

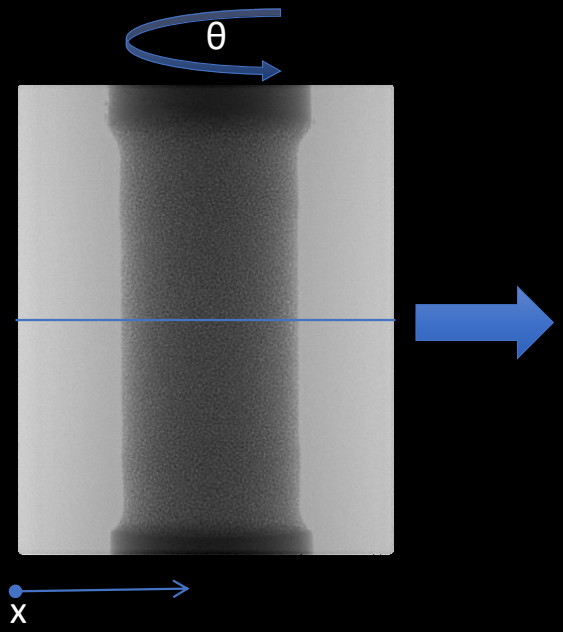
1. comparing density in two different zones in the object is only possible if the atomic number Z is constant
2. a change in density between two zones can be cancelled by a change of the atomic number in the opposite direction

E > 30 keV → Compton scattering is predominant so μ is only related to mass density BUT contrast within the image is lower:

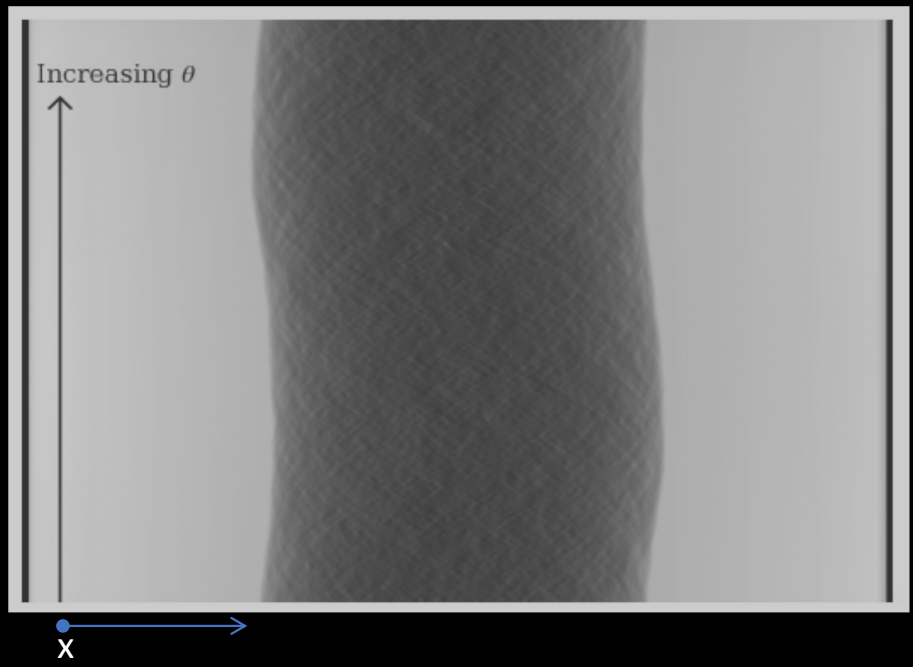
(variation with keV is material dependent)

Tomography methodology

We reconstruct the 3D image slice by slice



looking at this row of pixels as it evolves with angle gives

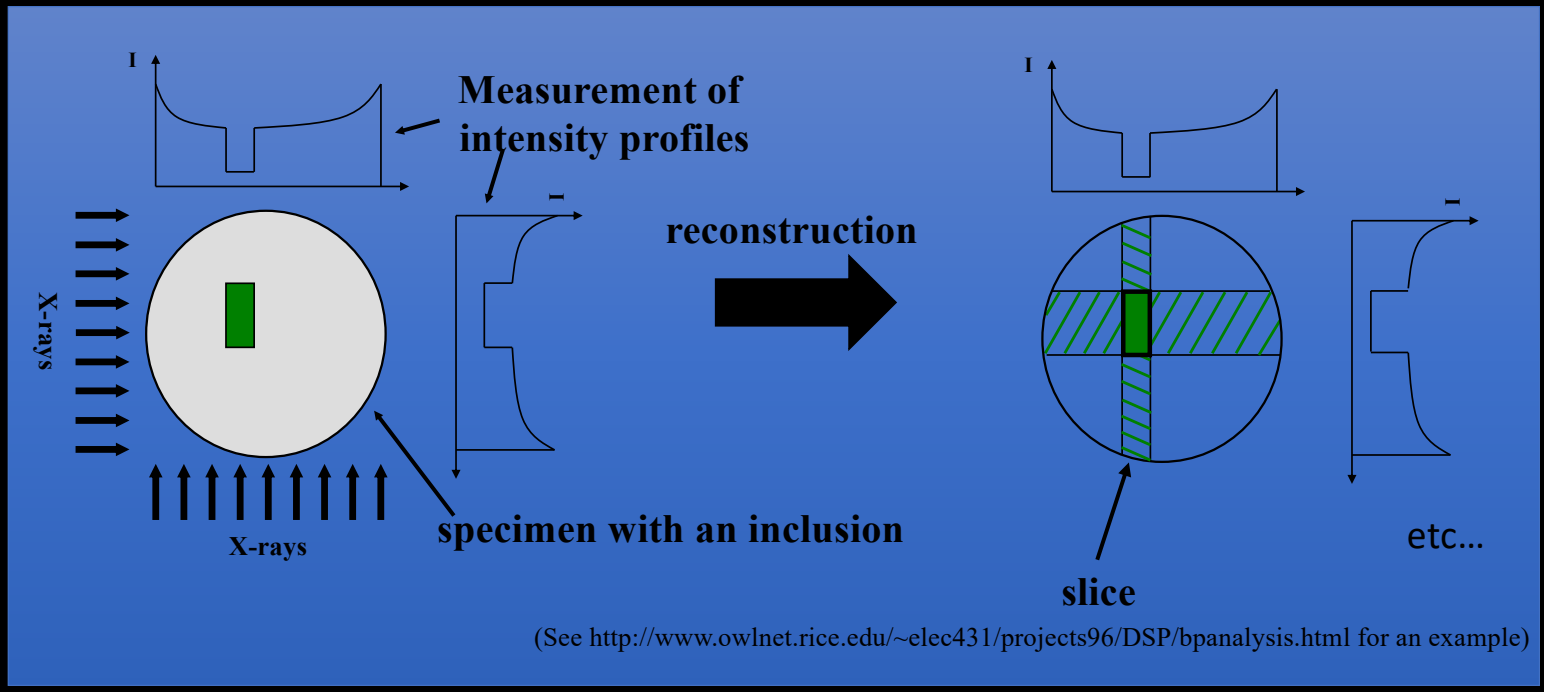


This is a sinogram - a Radon transform of the slice

Images: E. Andò
Lab 3SR, Grenoble

Principle of reconstruction

- Reconstruction of slices from different angle measurements with appropriate algorithm (analytic or algebraic methods), basic approach is back-projection:



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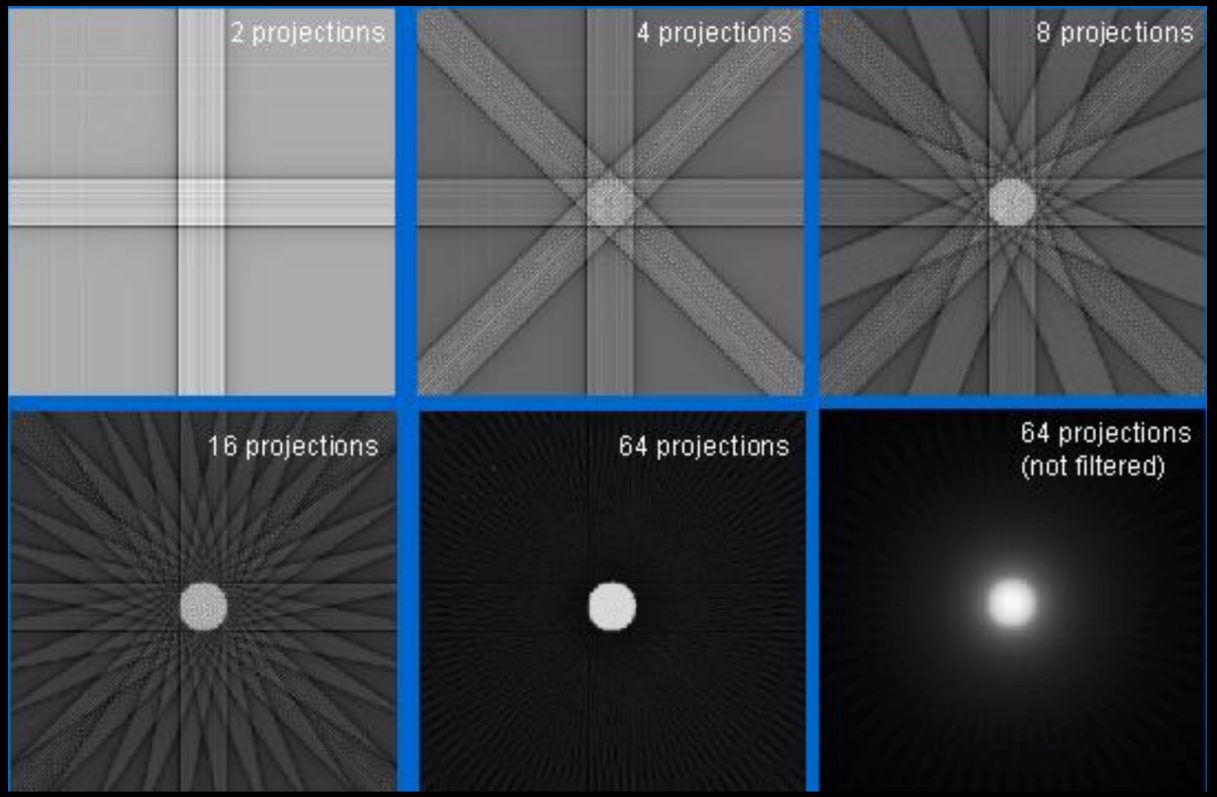
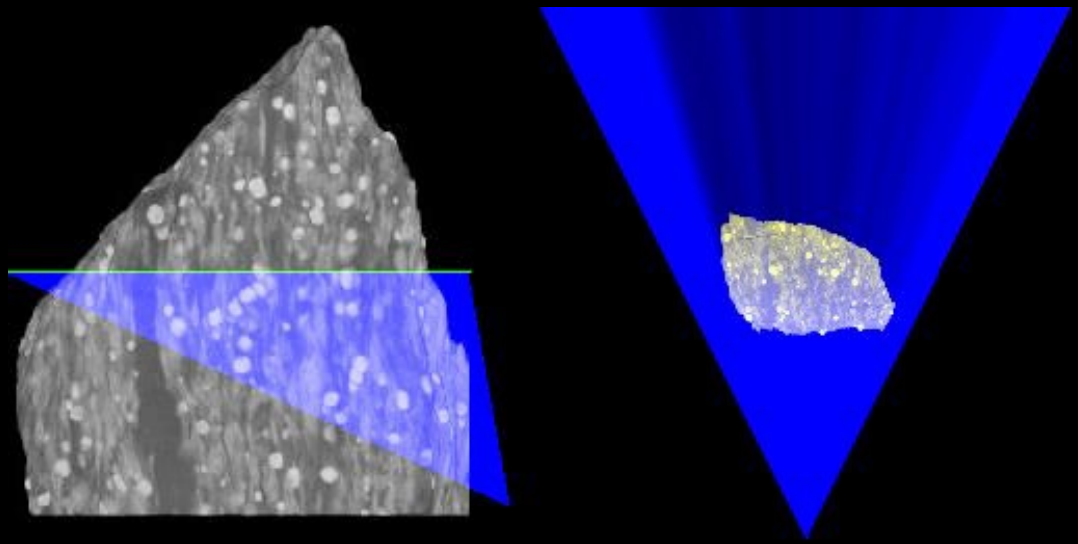


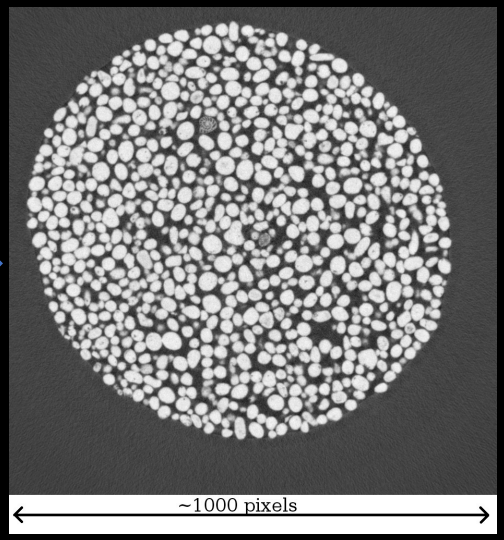
www.plant-phenotyping-network.eu/lw_resource/datapool/_items/item_161/van_loo.pdf

Principle of reconstruction

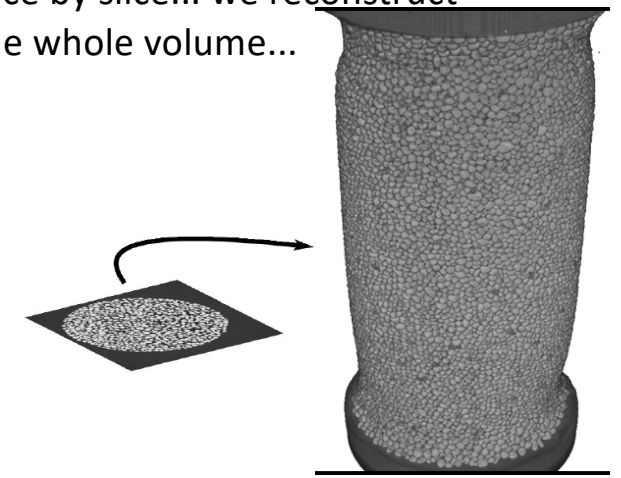


Principle of reconstruction

Knowing the centre of rotation we can solve the inverse problem (inverse radon transform), which results in a reconstruction of the slice that gave rise to the sinogram



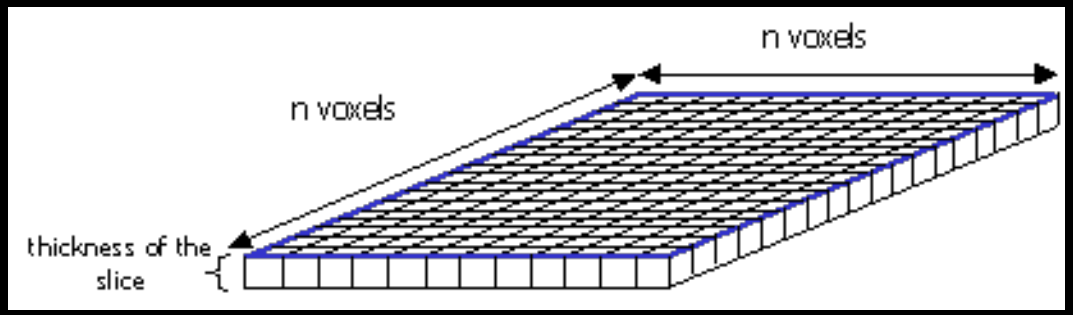
slice by slice... we reconstruct the whole volume...



Images: E. Andò
Lab 3SR, Grenoble

Voxel = pixel in 3D

Each slice represents a finite thickness of the object and is composed of voxels (extension in 3D of a pixel).



Schematic of a slice (composed of n x n voxels)

The final parameter visualised i.e., voxel values in the reconstructed slice, is linked to the mean of the linear attenuation coefficients of all the components physically presents in the voxel.

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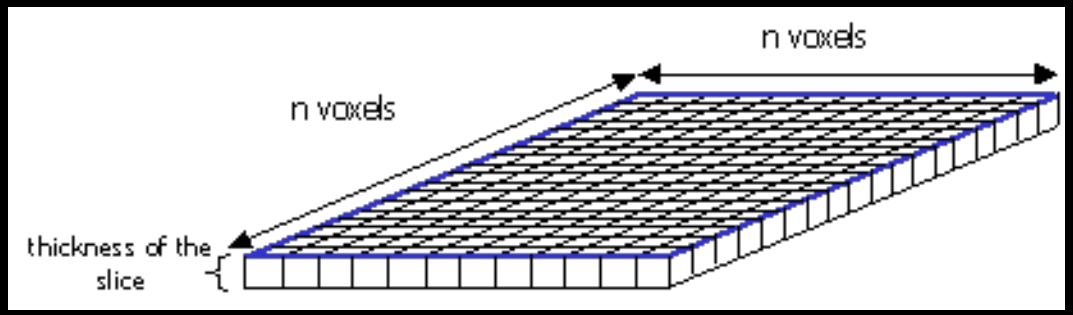

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The size of the voxel is the **image "discretisation"**…

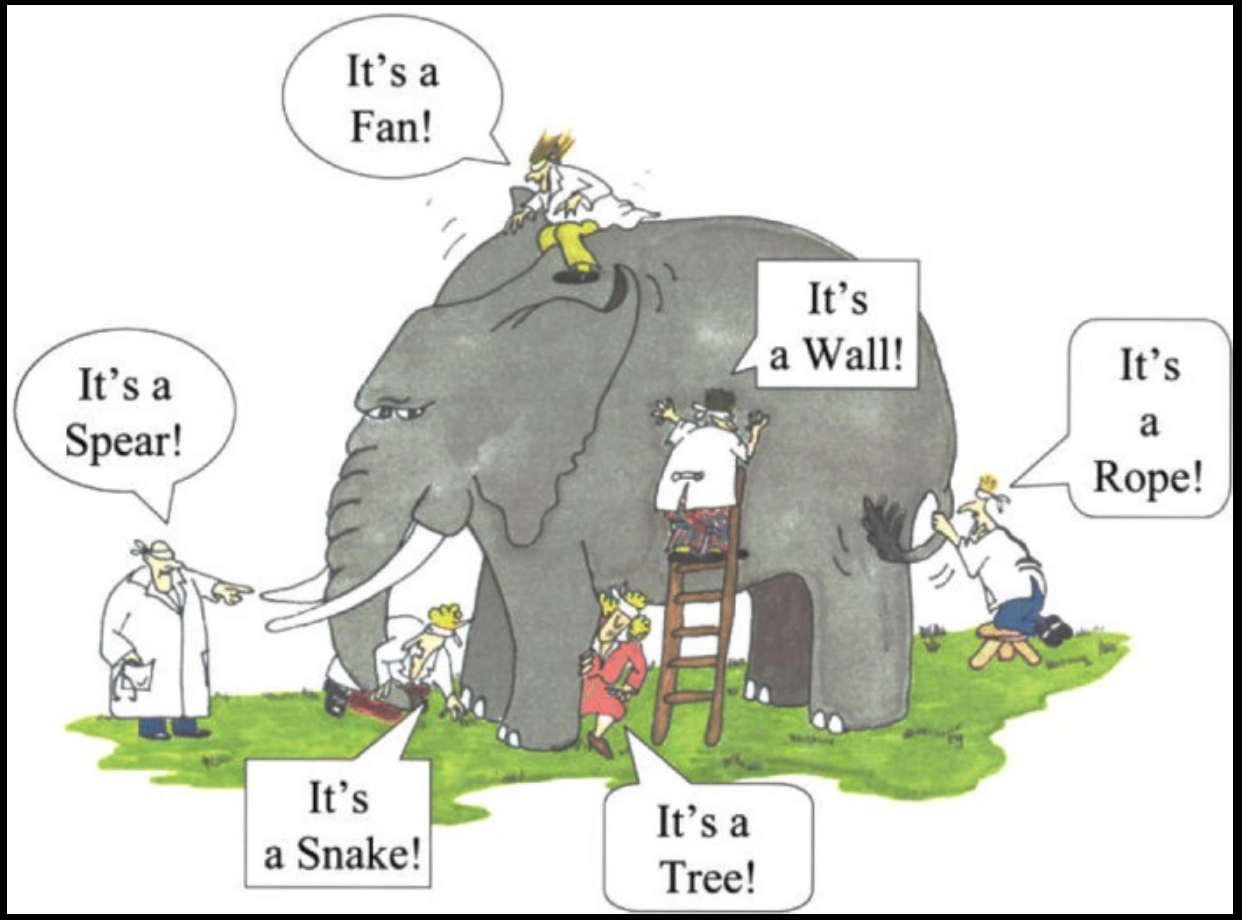
… this is not the ***spatial resolution*** as is commonly said…

… spatial resolution will be a function of the reconstruction algorithm, the detector quality and the beam divergence

… in reality the image is normally blurred between voxels

X-ray imaging or neutron imaging?

…Depends on what you want to see!



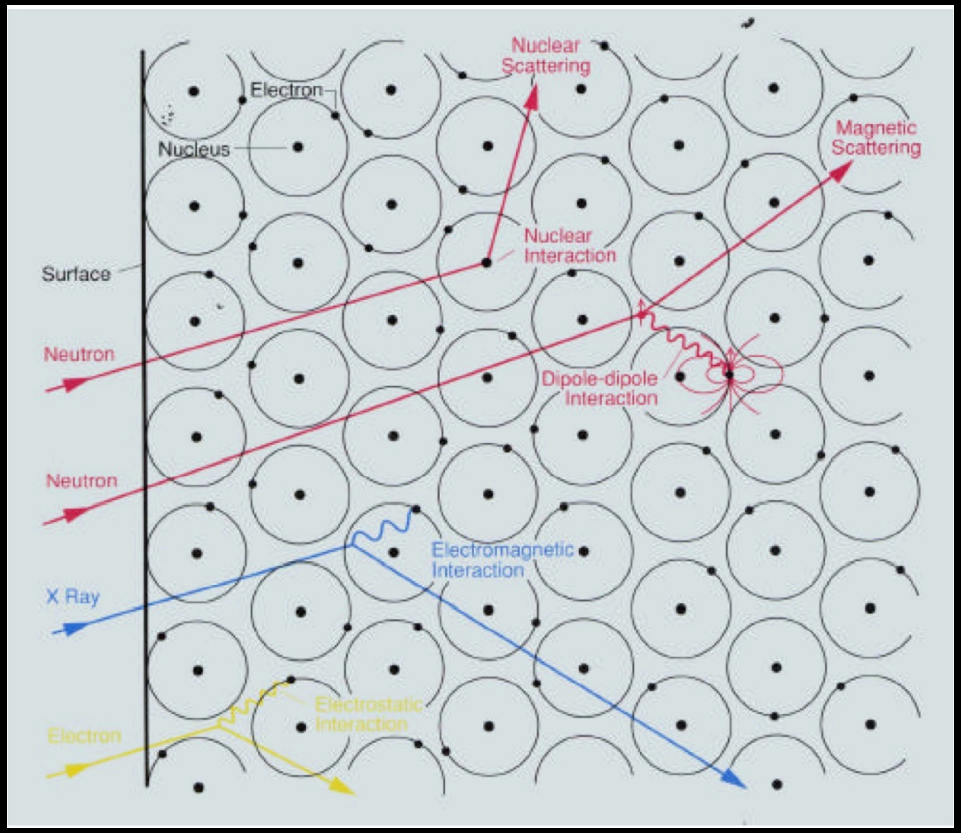
Neutron sources

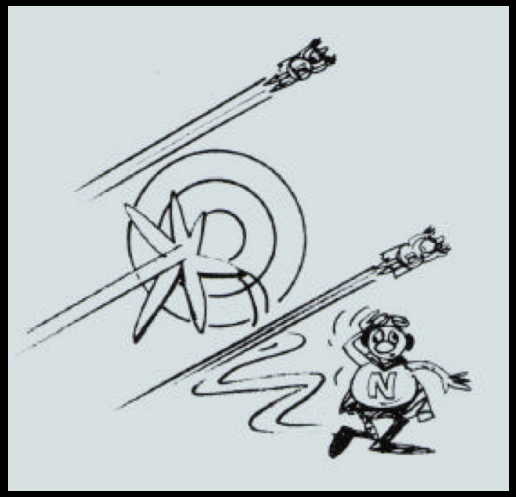
Institut Laue Langevin, Grenoble, France: Reactor source

European Spallation Source, Lund, Sweden

Neutron imaging

Neutron interaction mechanisms



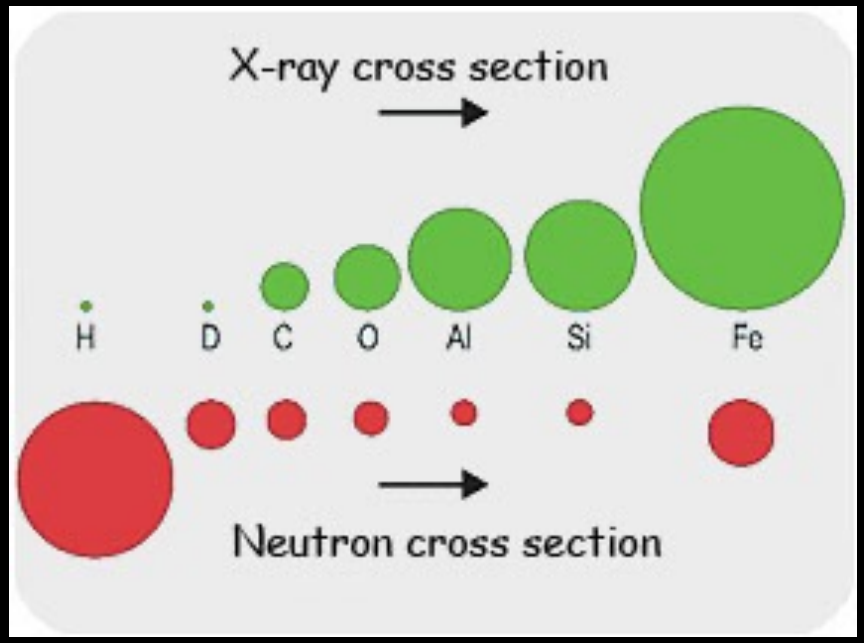


- Neutrons interact with atomic nuclei via very short range (~10^{-15}m) forces.
- Neutrons also interact with unpaired electrons and therefore “see” magnetism.

After R. Pynn (Los Alamos National Laboratory)

X-ray imaging or neutron imaging?

…Depends on what you want to see!



- Different sensitivities
- Different penetration depths for different materials
  - Can see into dense objects with neutrons

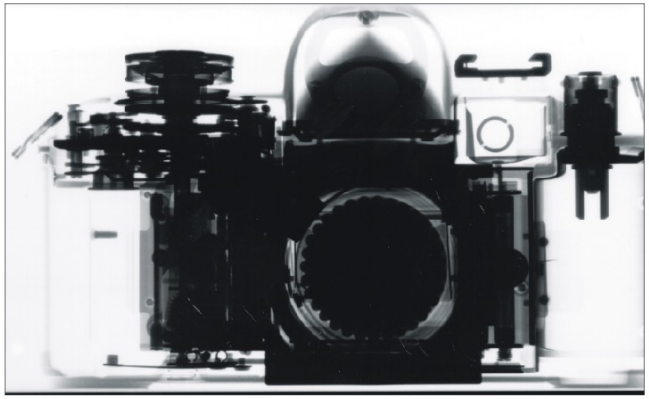


Fig. b: Radiographic image of a camera made X-rays

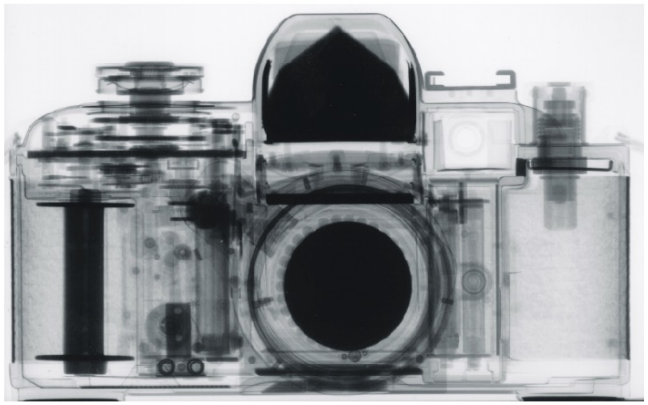


Fig. a: Neutron radiography of a camera

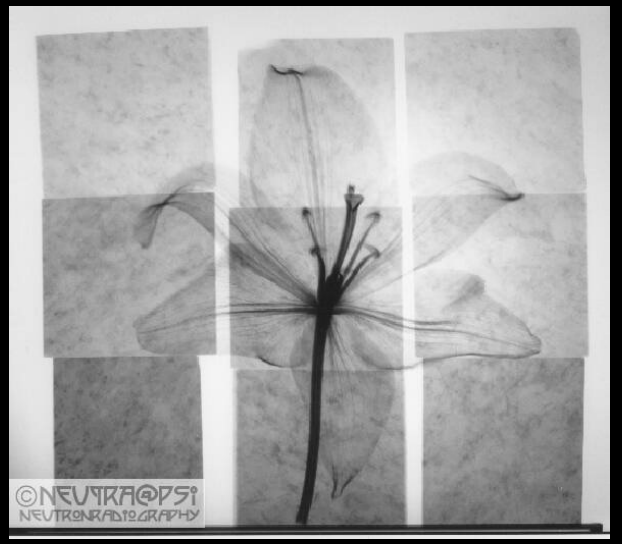
Why use neutrons

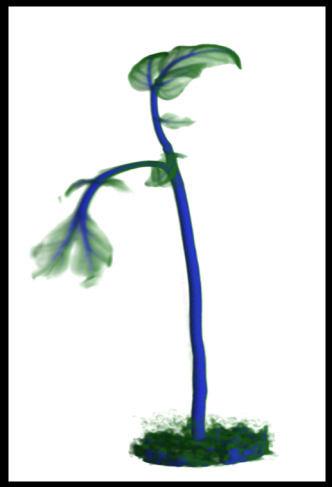
Some advantages:

High penetration power

High sensitivity to Hydrogen

Low radiation damage

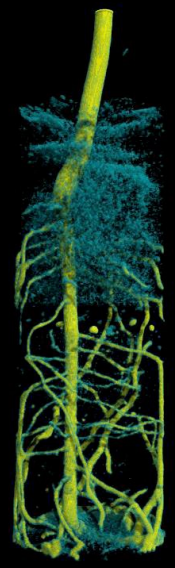


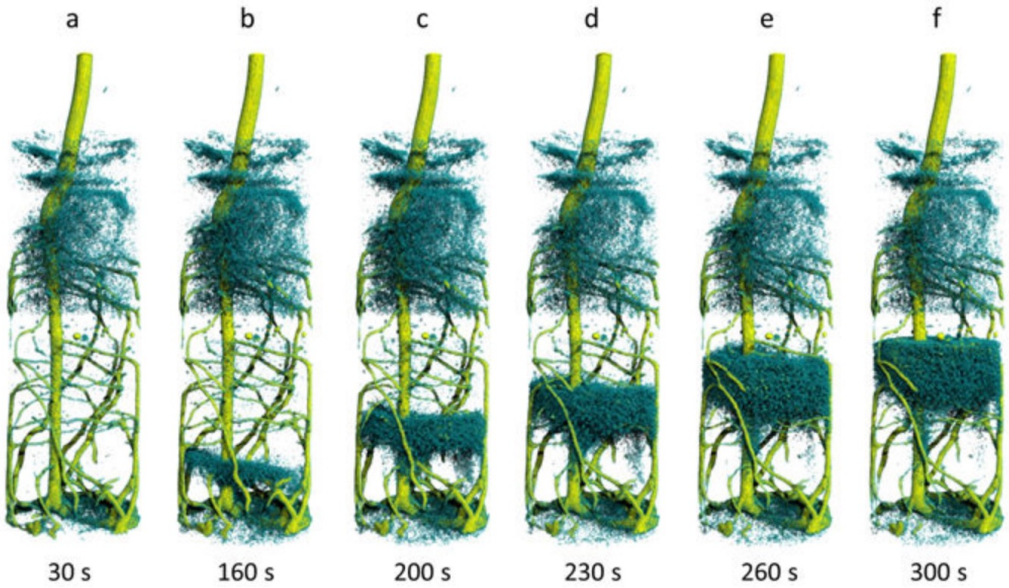


Why use neutrons

Some advantages:

Isotope sensitivity

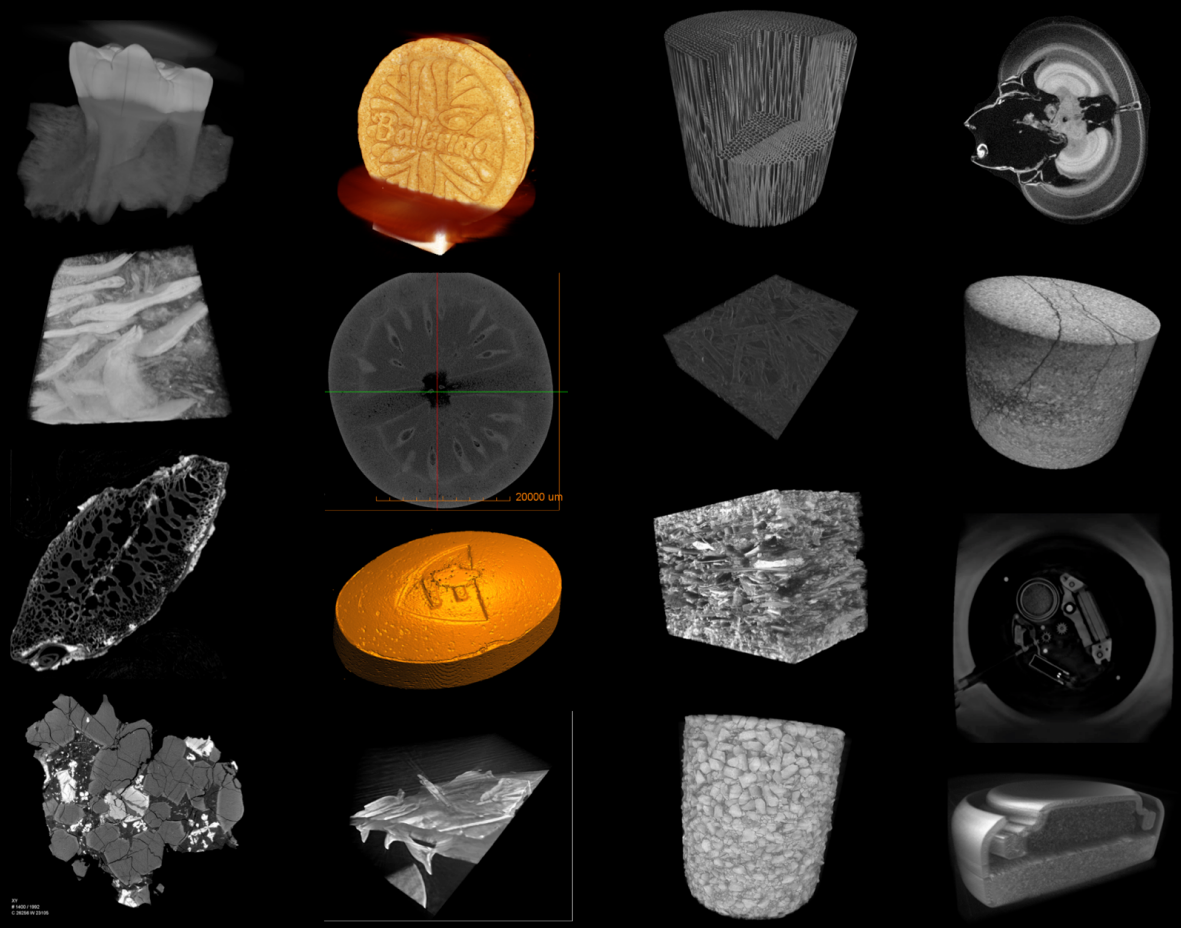




Time-resolved neutron tomography of the lupine root system after the injection of 4 ml deuterated water (D_2O) through the bottom. The time series (30 s ≤ t ≤ 300 s) shows the ascending front of water (H_2O) displaced by deuterated water moving upwards. The time resolution for each tomogram is just 10 s. A video of this sequence is provided in the supplementary information.

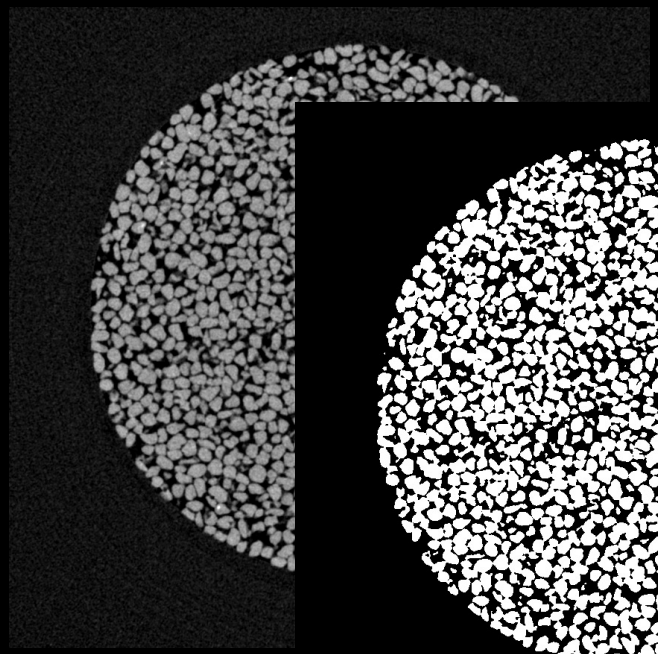
Tötzke et al., Scientific reports, 2017

Multiple applications… and many nice images…



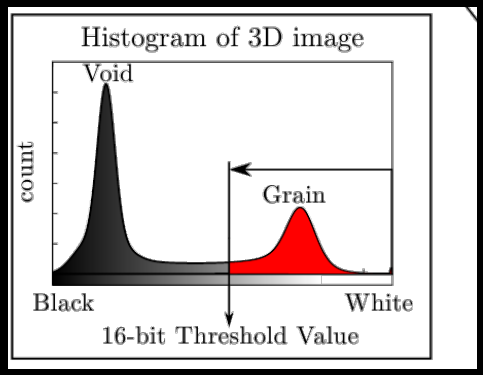
But so what…

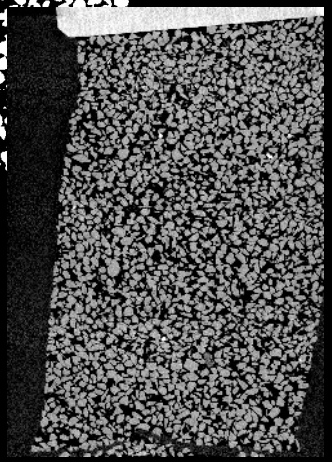
How do we see/identify and characterise microstructure?



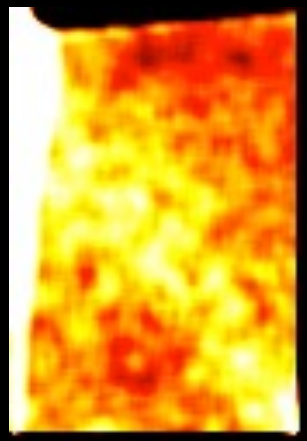
"Raw" image

Binarise
(grains and voids)

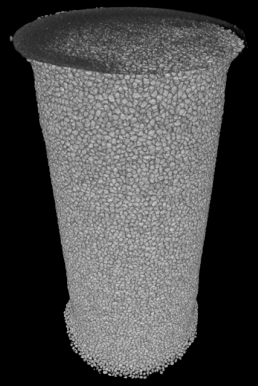




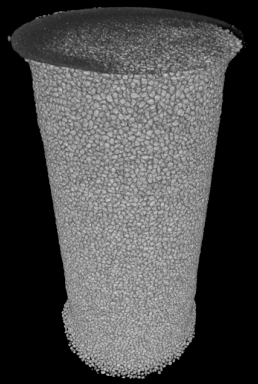
Tomo

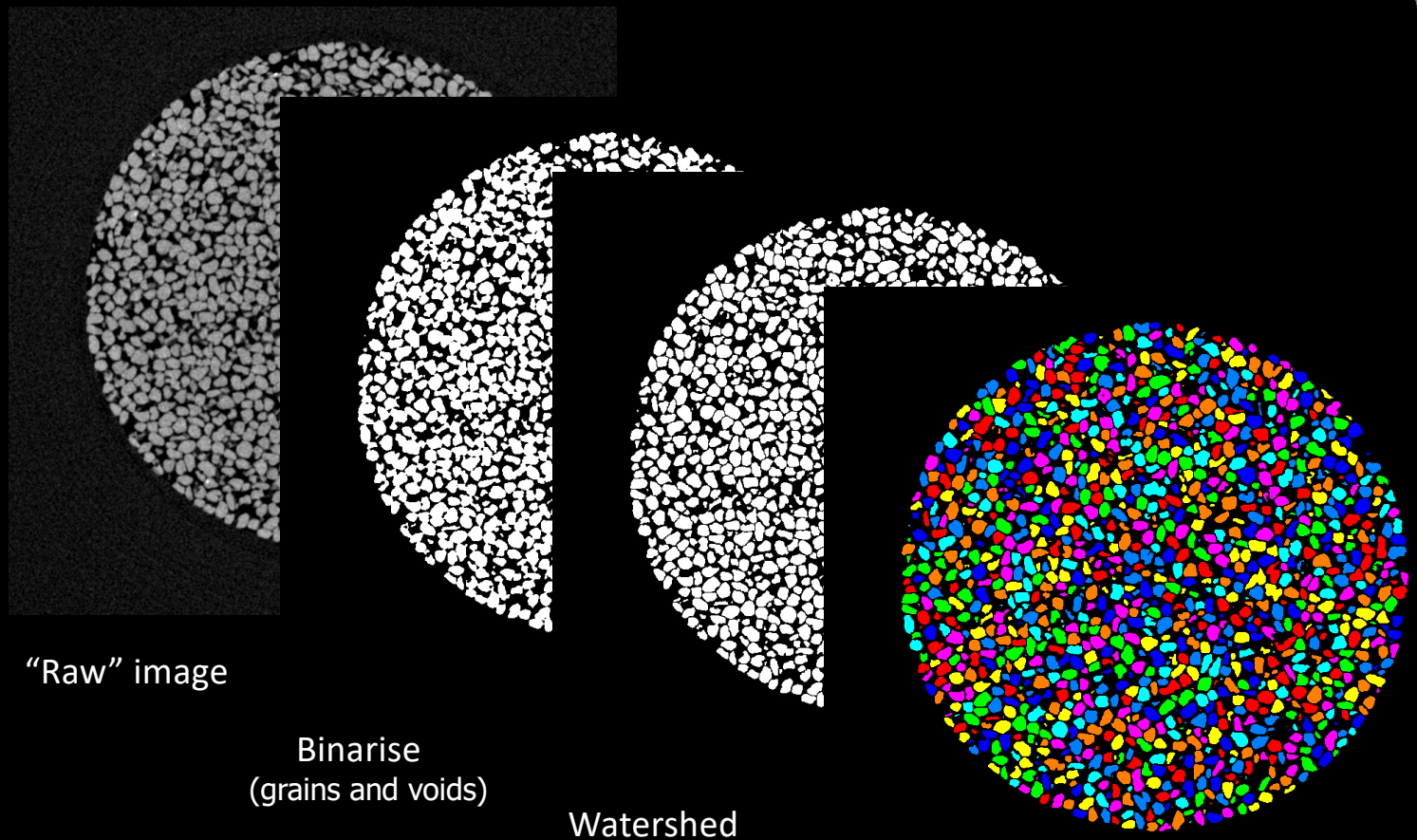


Porosity



How do we see/identify and characterise microstructure?





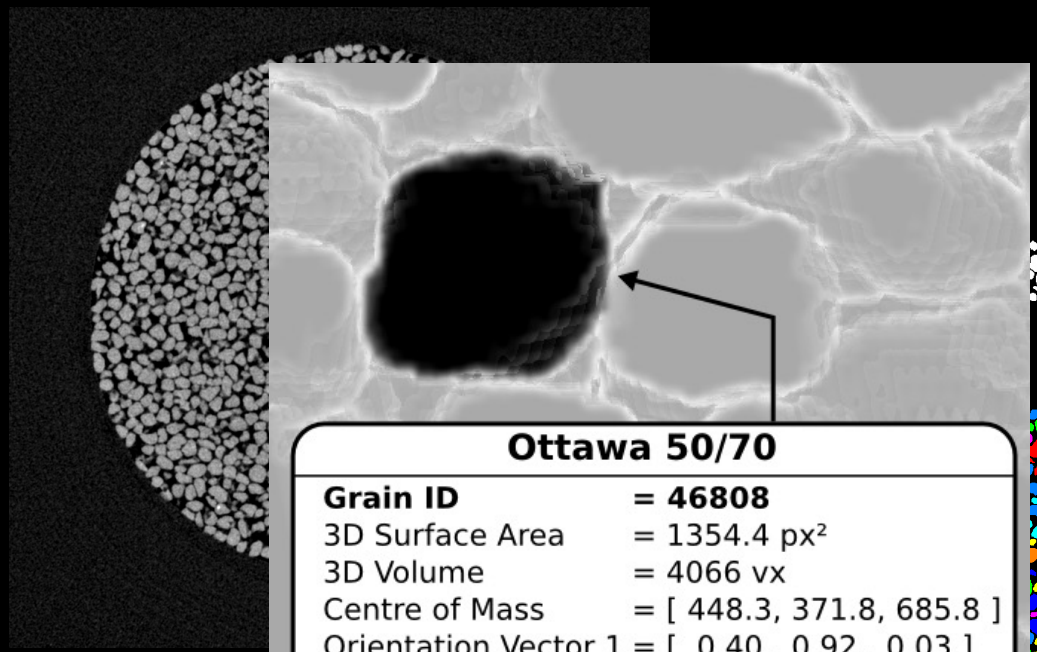
"Raw" image

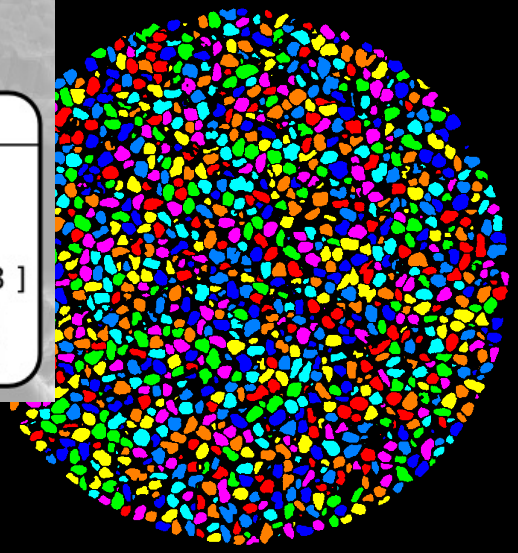
Binarise
(grains and voids)

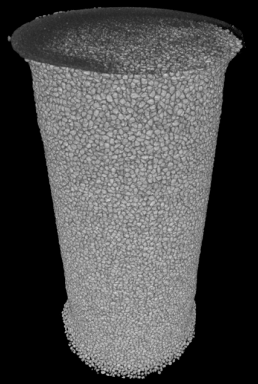
Watershed
Segmentation
(split grains apart)

Label Individual Grains

How do we see/identify and characterise microstructure?







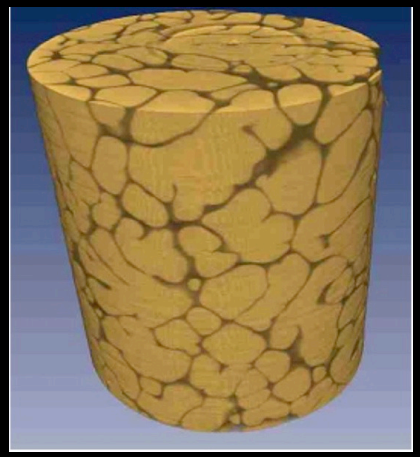
"Raw" image

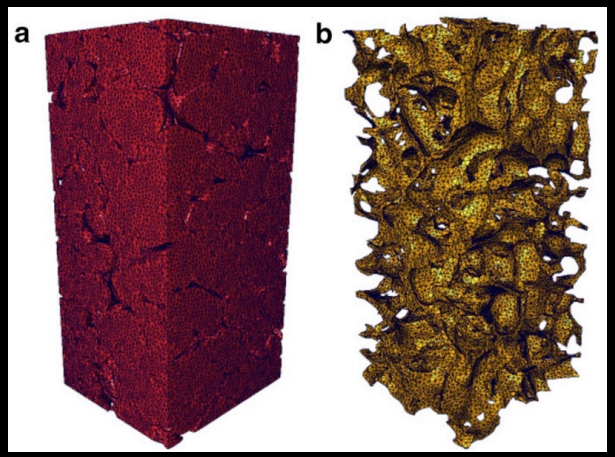
Binarise
(grains and voids)

Watershed
Segmentation
(split grains apart)

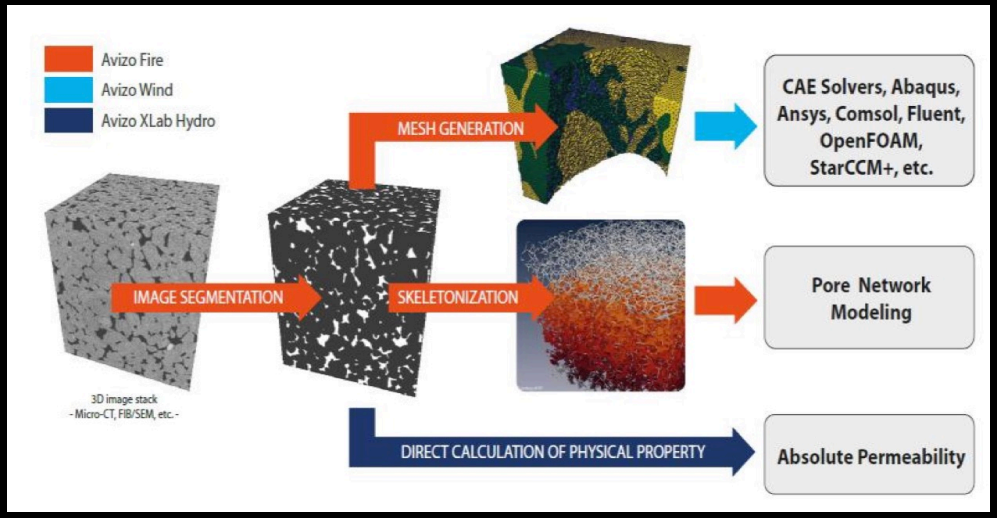
Label Individual Grains

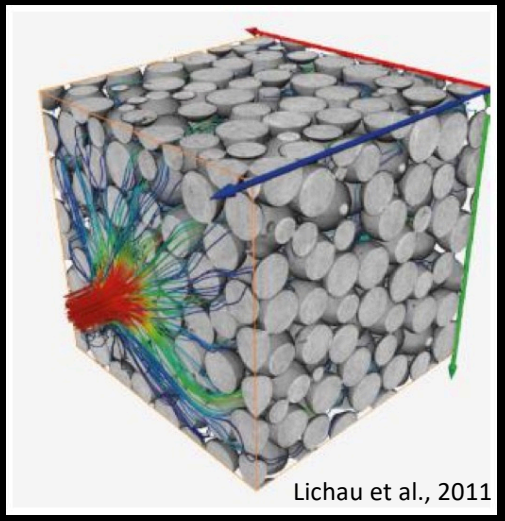
Structural imaging and characterisation → models





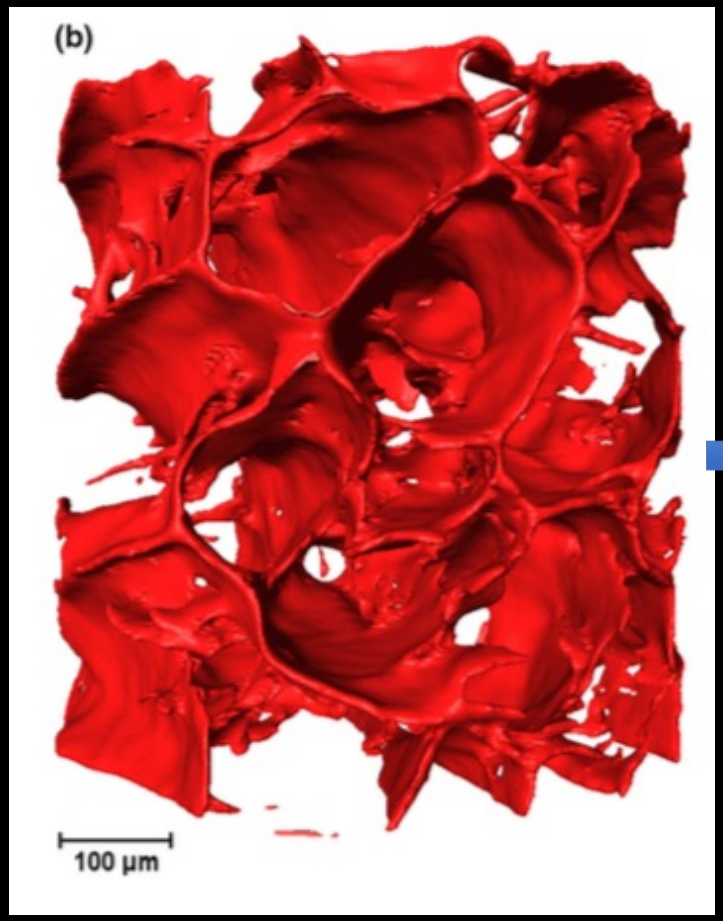
Madi et al., 2006



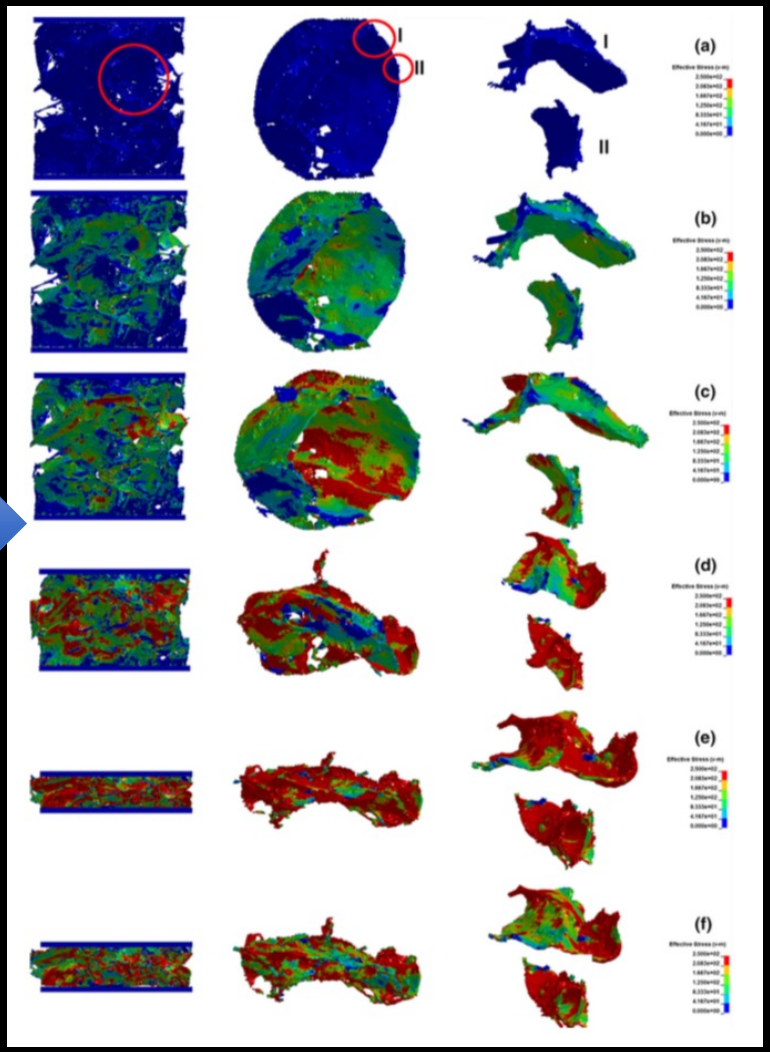


Lichau et al., 2011

Structural imaging and characterisation → models



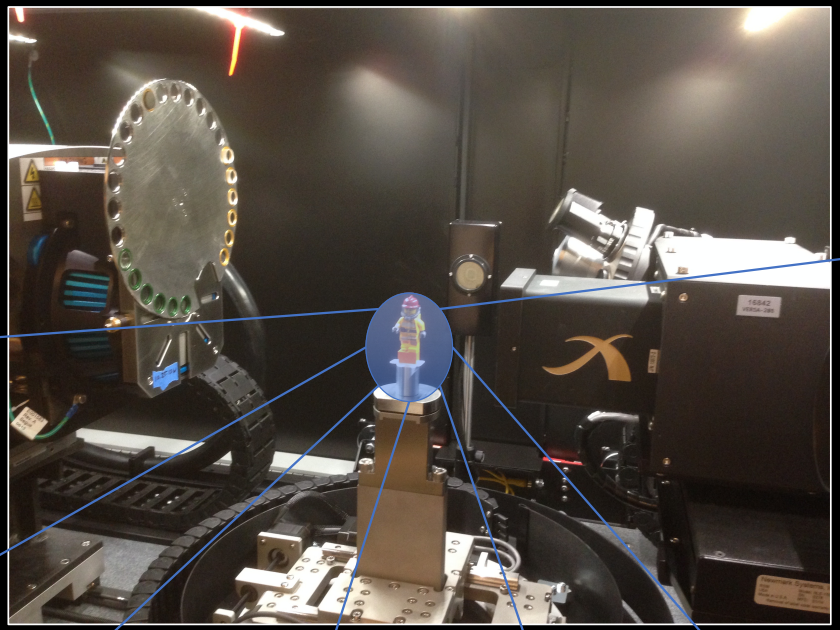
(Images from 4D Imaging Lab)

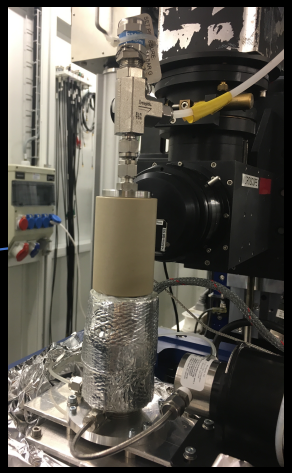


Srinivasa, Kulachenko , Karlberg, 2017, Cellulose

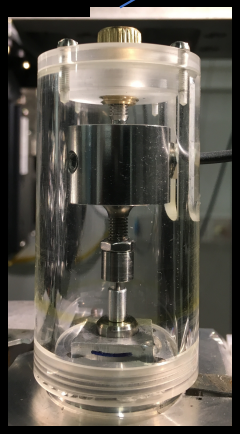
4D imaging…

- In-situ devices





Chemical treatment
(Delignification)



Compression

Tension

Peeling

Vacuum

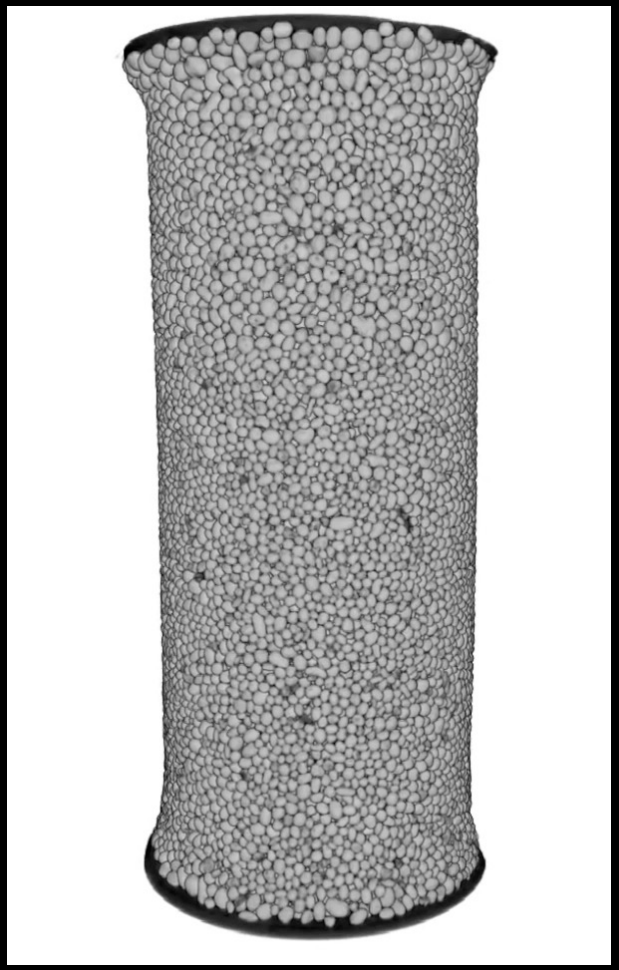
Humidity

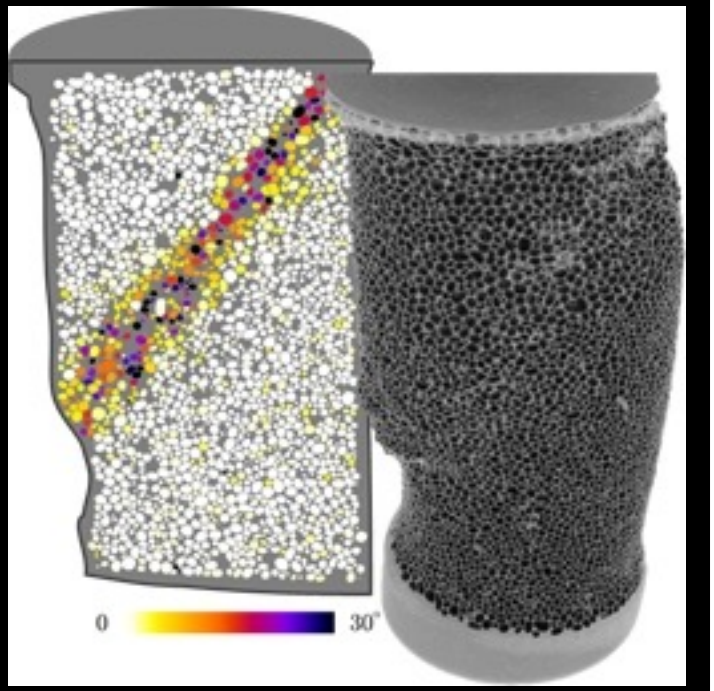
Others and Future:

- Pressure
- Fluid flow
- Electrochemical
- Heating
- Cooling (freezing)
- …

4D imaging and in-situ experiments

4D imaging of triaxial compression of a sample of (oolitic) sand (10’s thousands grains)



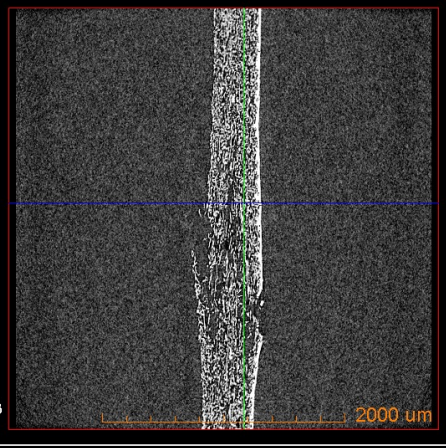


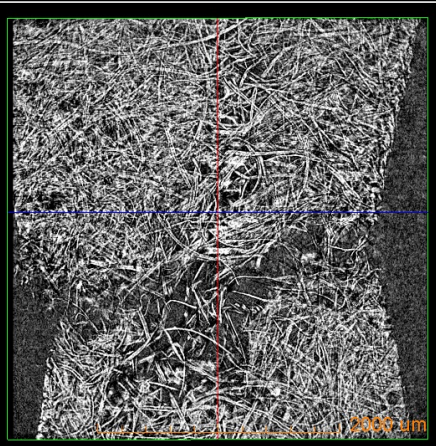
Quantification of grain kinematics

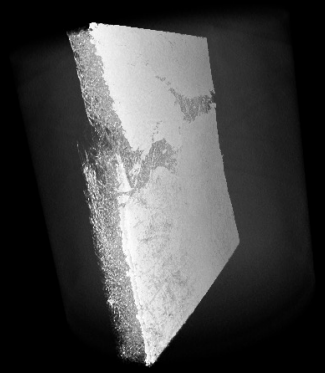
E Andò, CNRS, Grenoble, France

4D imaging and in-situ experiments

“in-situ” tensile testing of paperboard

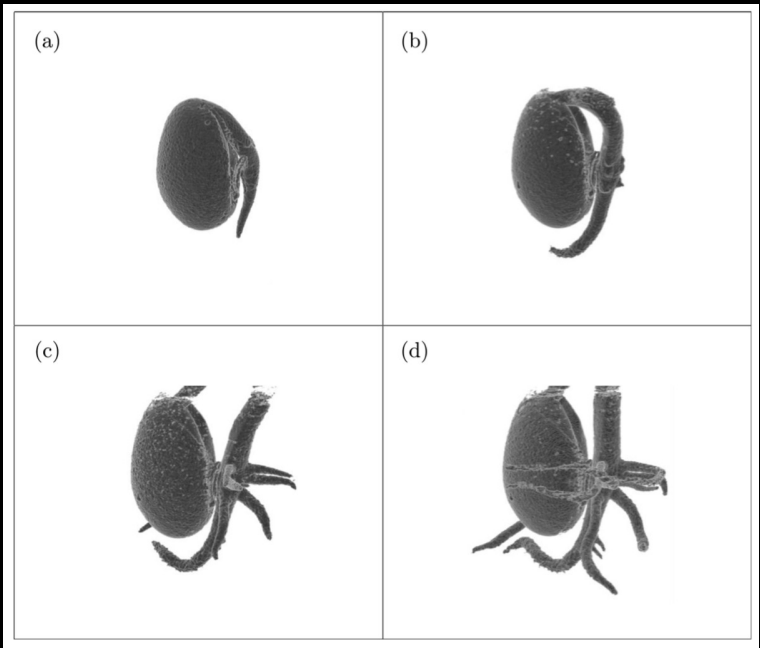




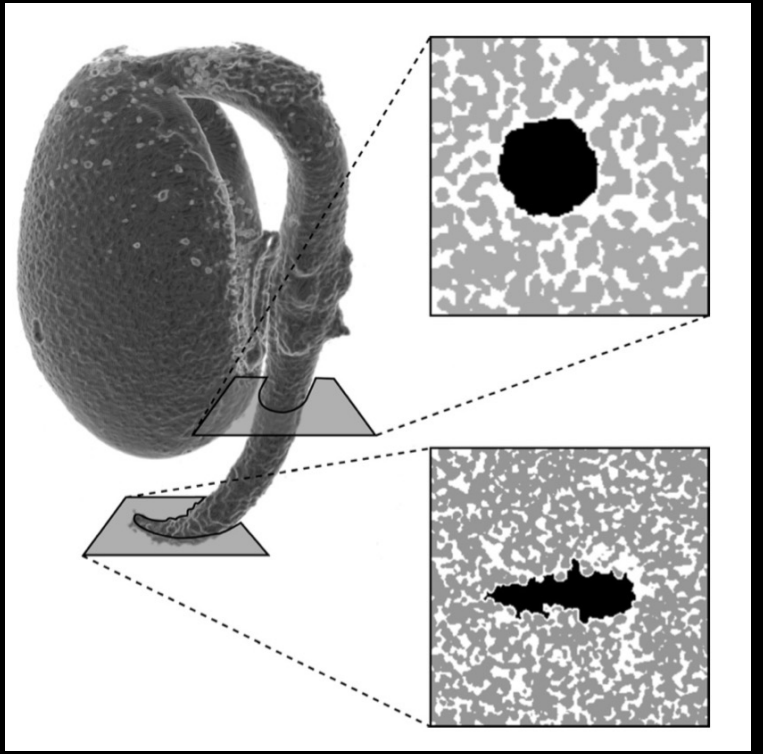


4D imaging and in-situ experiments

Growth of seeds in soil…



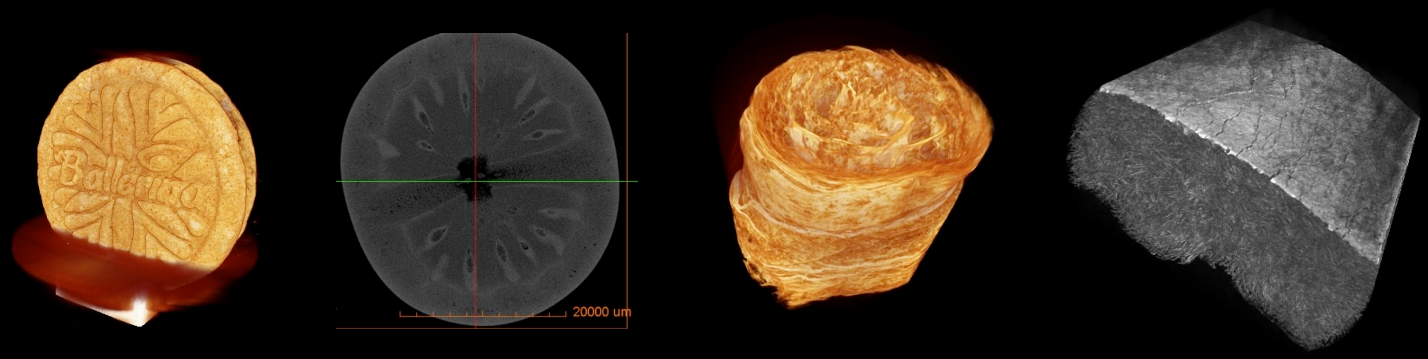
Bean-root system: evolution during germination (a) 4 days, (b) 5 days, (c) 6 days, and (d) 7 days after wetting



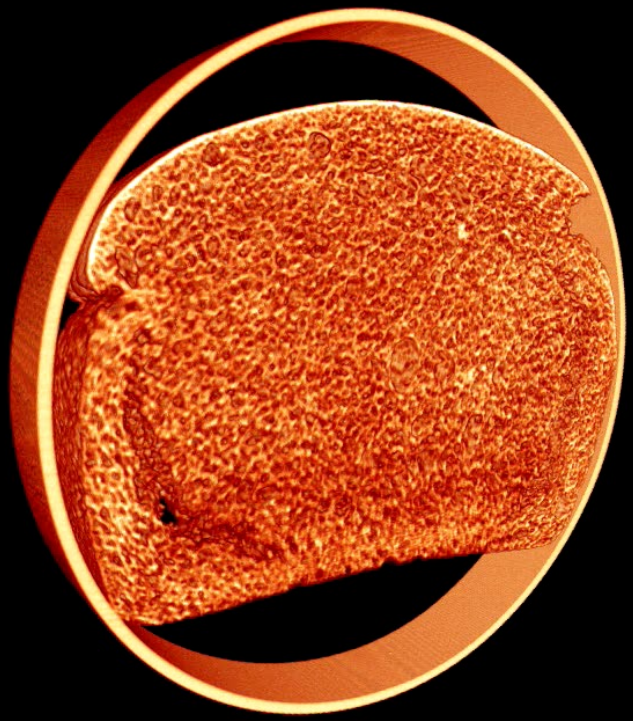
Bean-root system 5 days after wetting

Viggiani et al., 2015

Imaging Food and Packaging



Imaging internal structures: bread-products



≈ 25 mm

Collab. E. Larsson & N. Lorén, RISE

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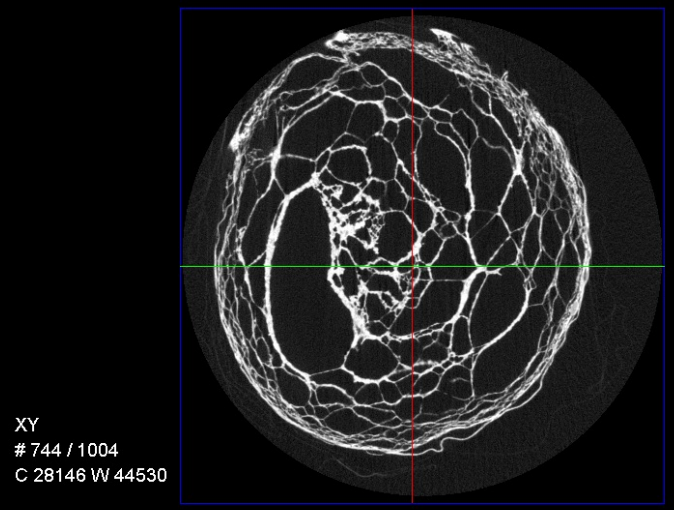
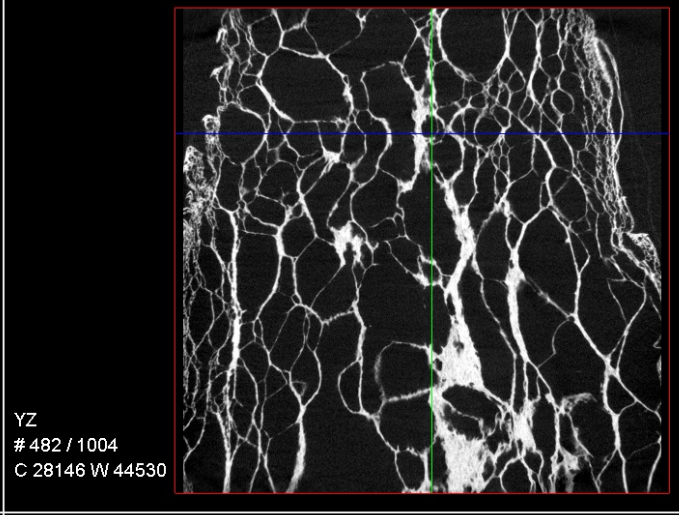
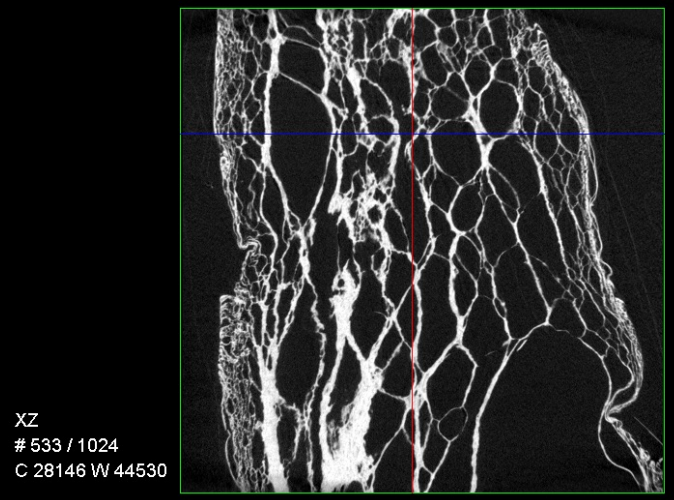
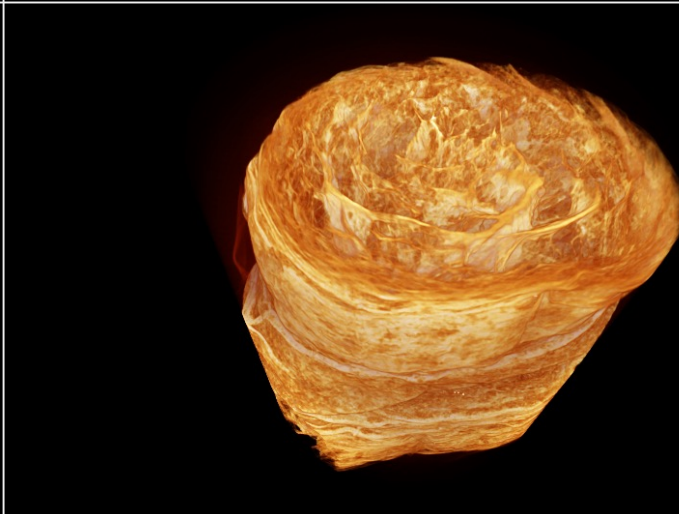
Imaging internal structures: bread-products









Collab. N. Lorén, RISE

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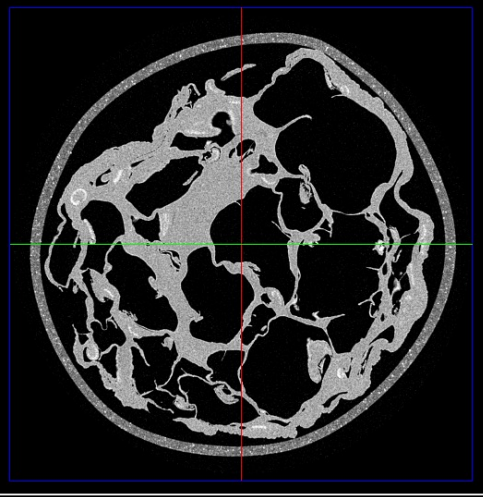
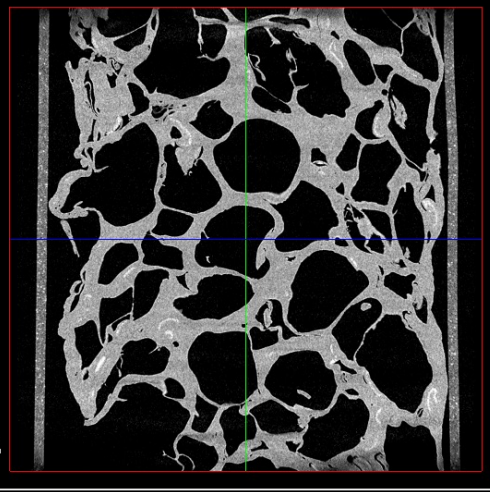
Extruded product structure



XY
503 / 1004
C 16804 W 12603



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503 / 1004
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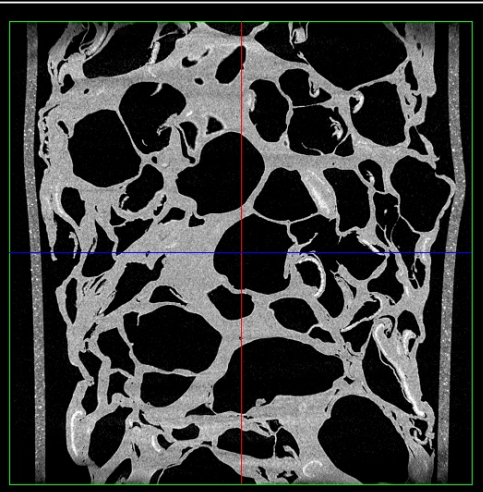

XZ
513 / 1024
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Collab. N. Lorén, RISE

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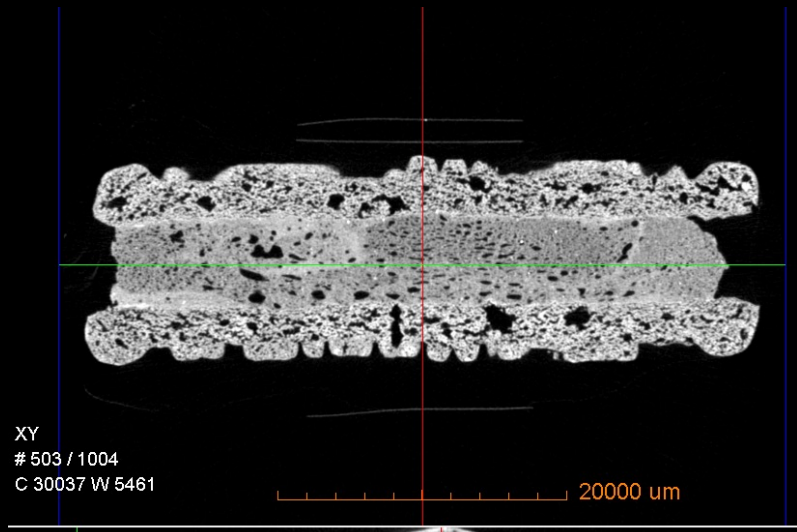
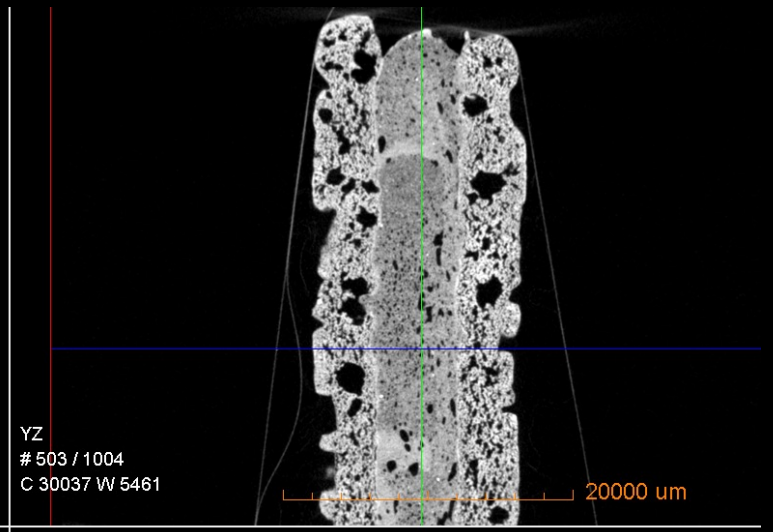
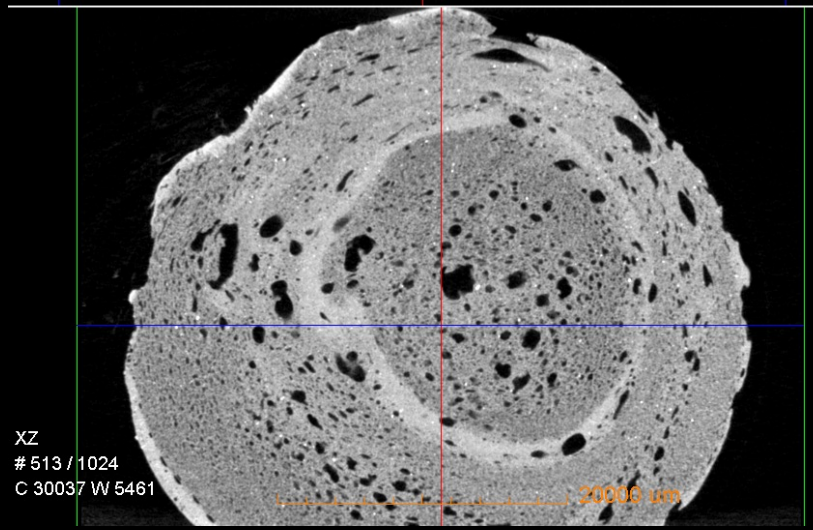
Imaging internal structures: biscuit-filling interaction







Collab. N. Lorén, RISE

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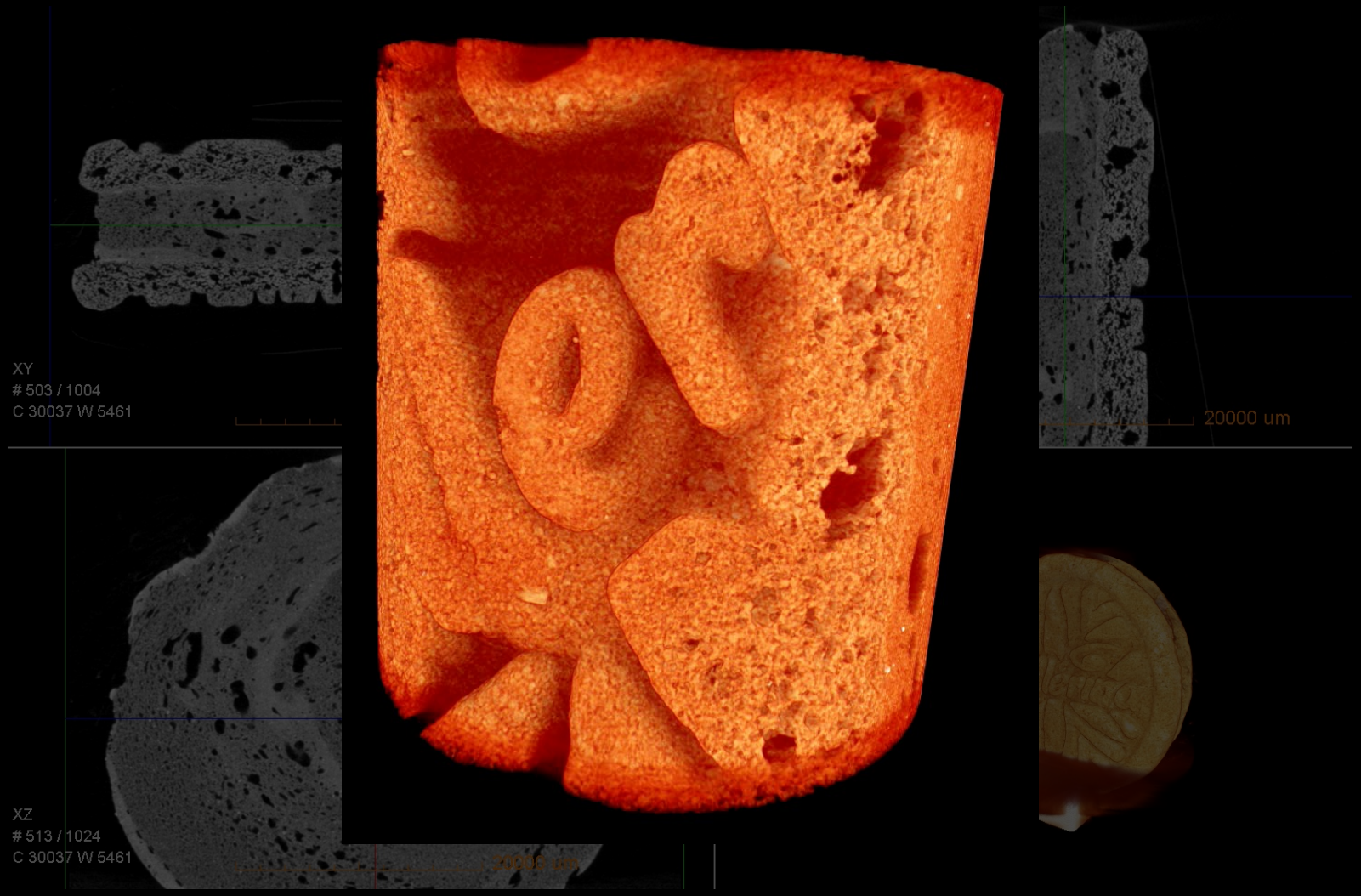
Imaging internal structures: biscuit-filling interaction



Collab. N. Lorén, RISE

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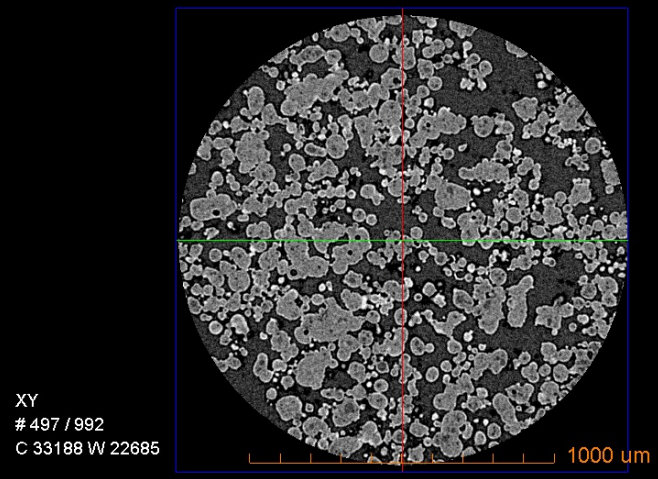
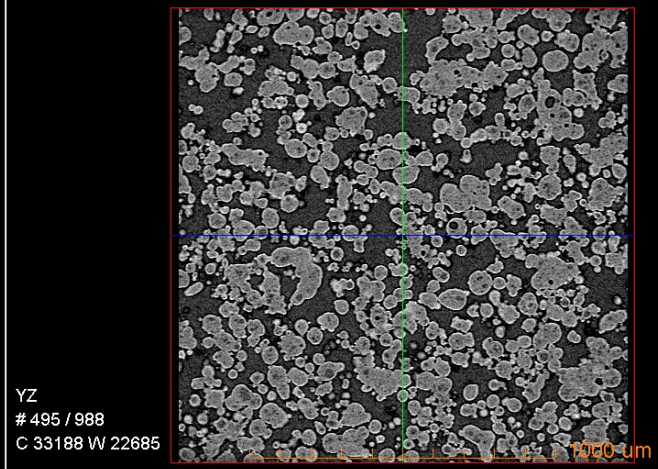
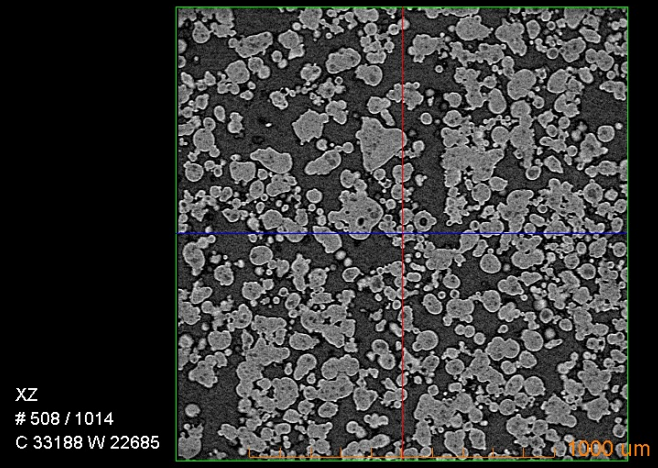
Whole-milk powder structure







Collab. B. Bergenstahl, Food Tech, LTH

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Freeze-dried milk powder structure

FoV = 324 µm

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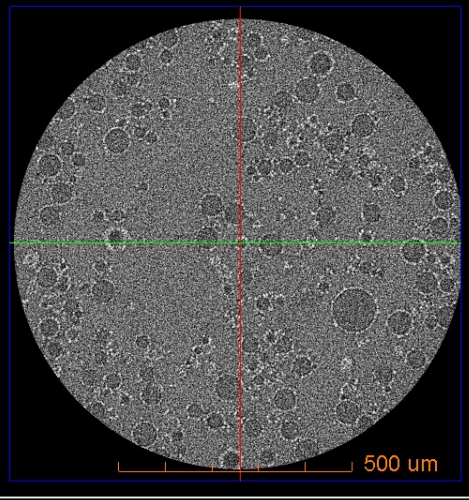
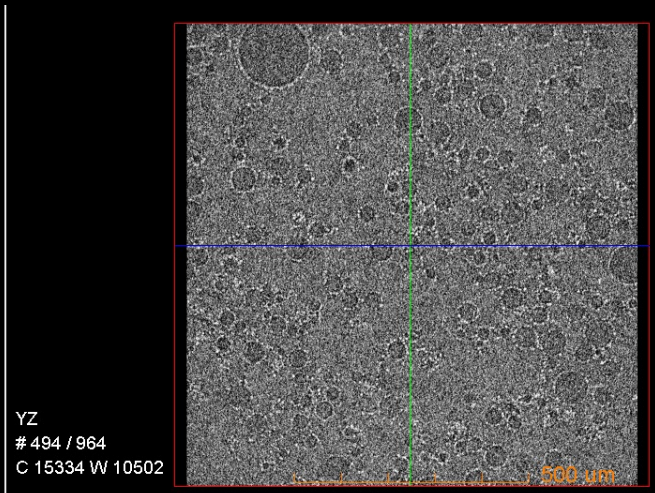
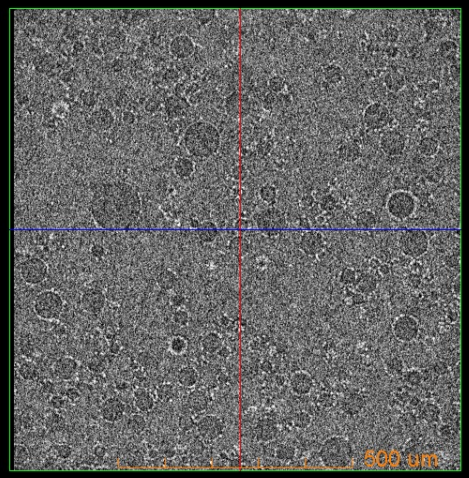
Pickering emulsions (rice based)







Collab. M. Rayner, Food Tech. LTH

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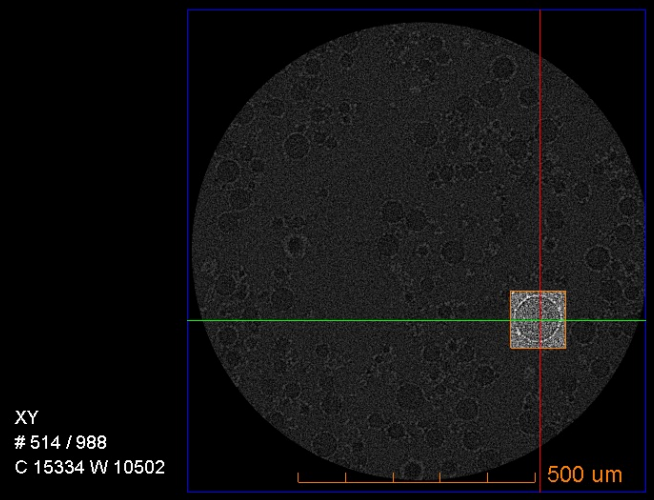
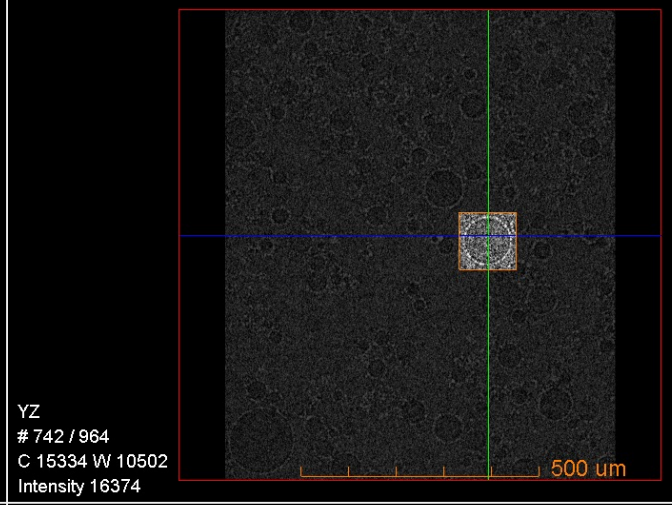
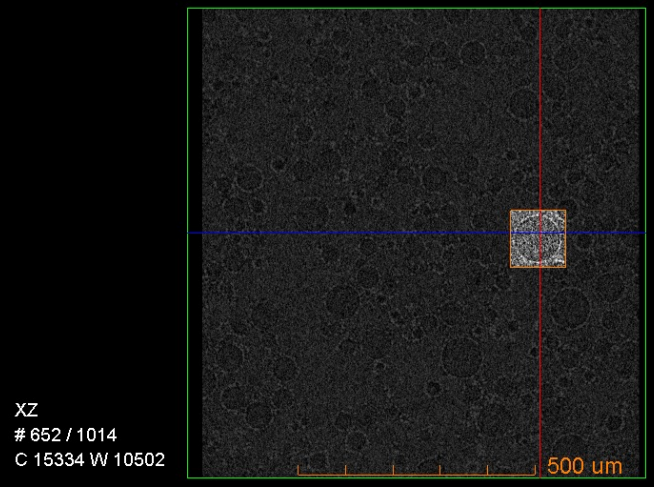
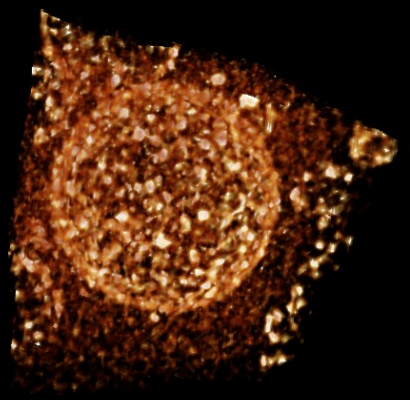
Pickering emulsions (rice based)









Collab. M. Rayner, Food Tech. LTH

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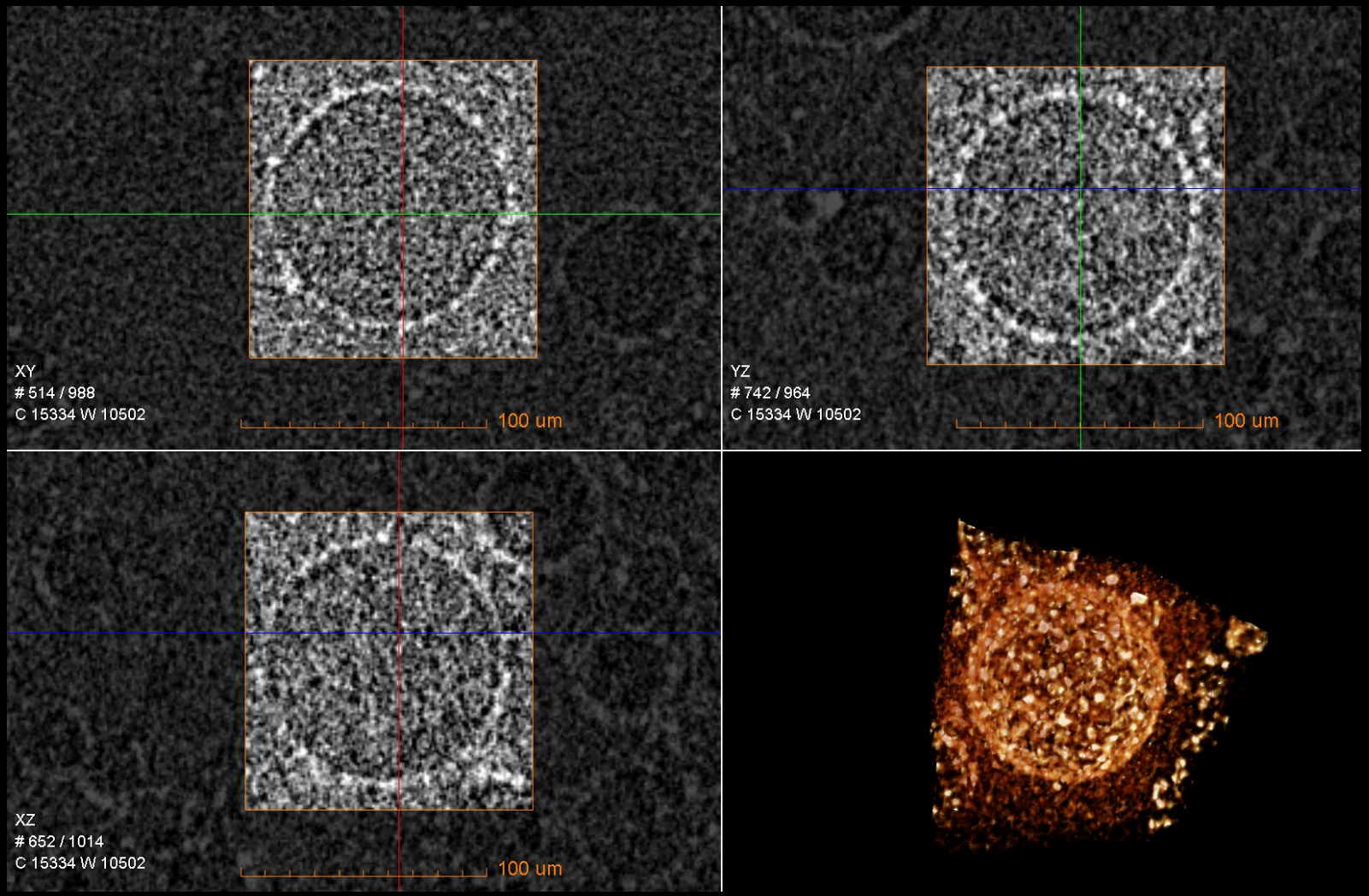
Pickering emulsions (rice based)



Collab. M. Rayner, Food Tech. LTH

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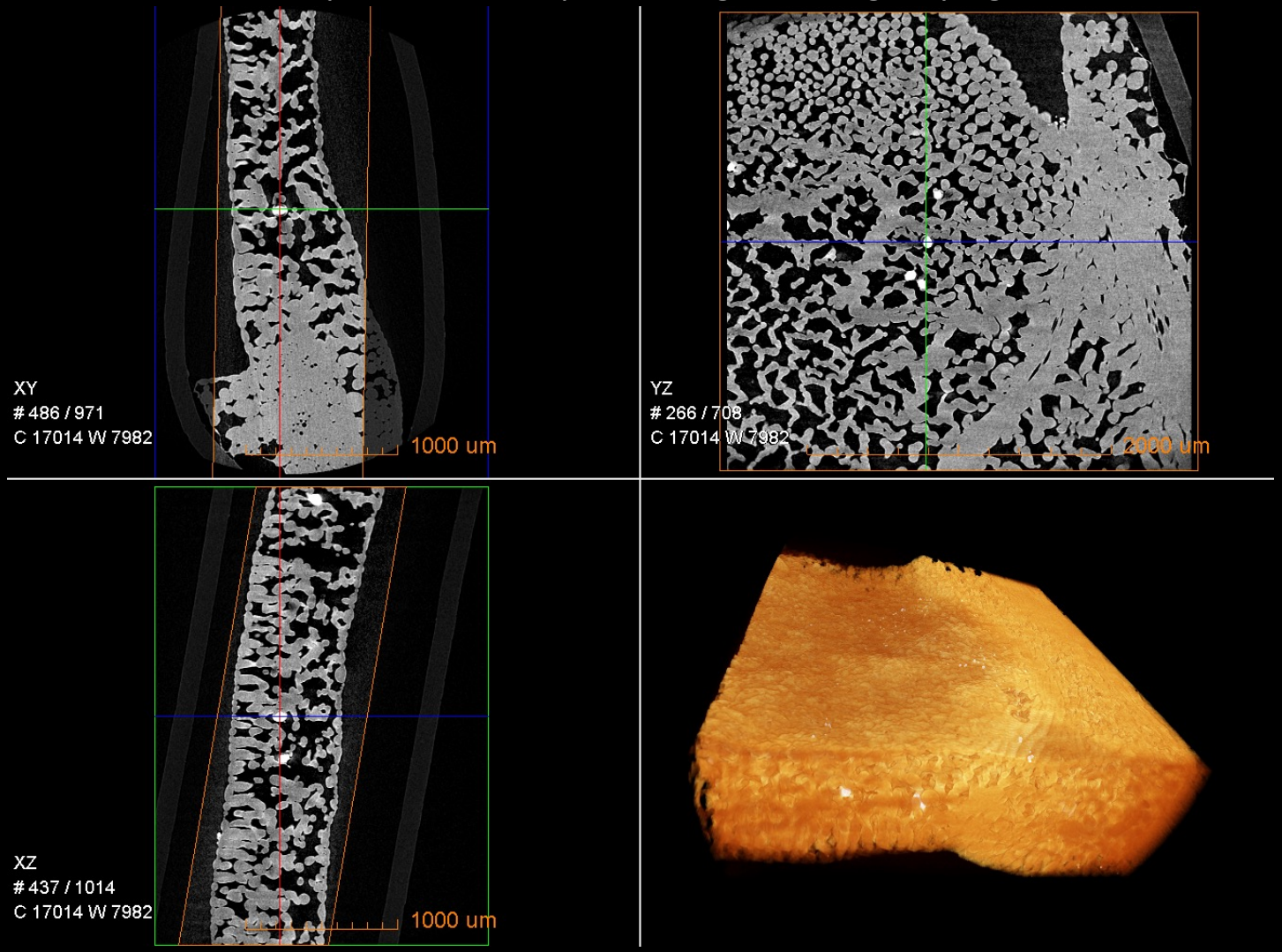
Internal structure of leaves towards optimisation of processing (freezing ,drying…)



Collab. F. Gomez & N.L. Yousef, Food Tech., LTH

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Internal structure of soft fruits/berries

Collab. N. Lorén, RISE

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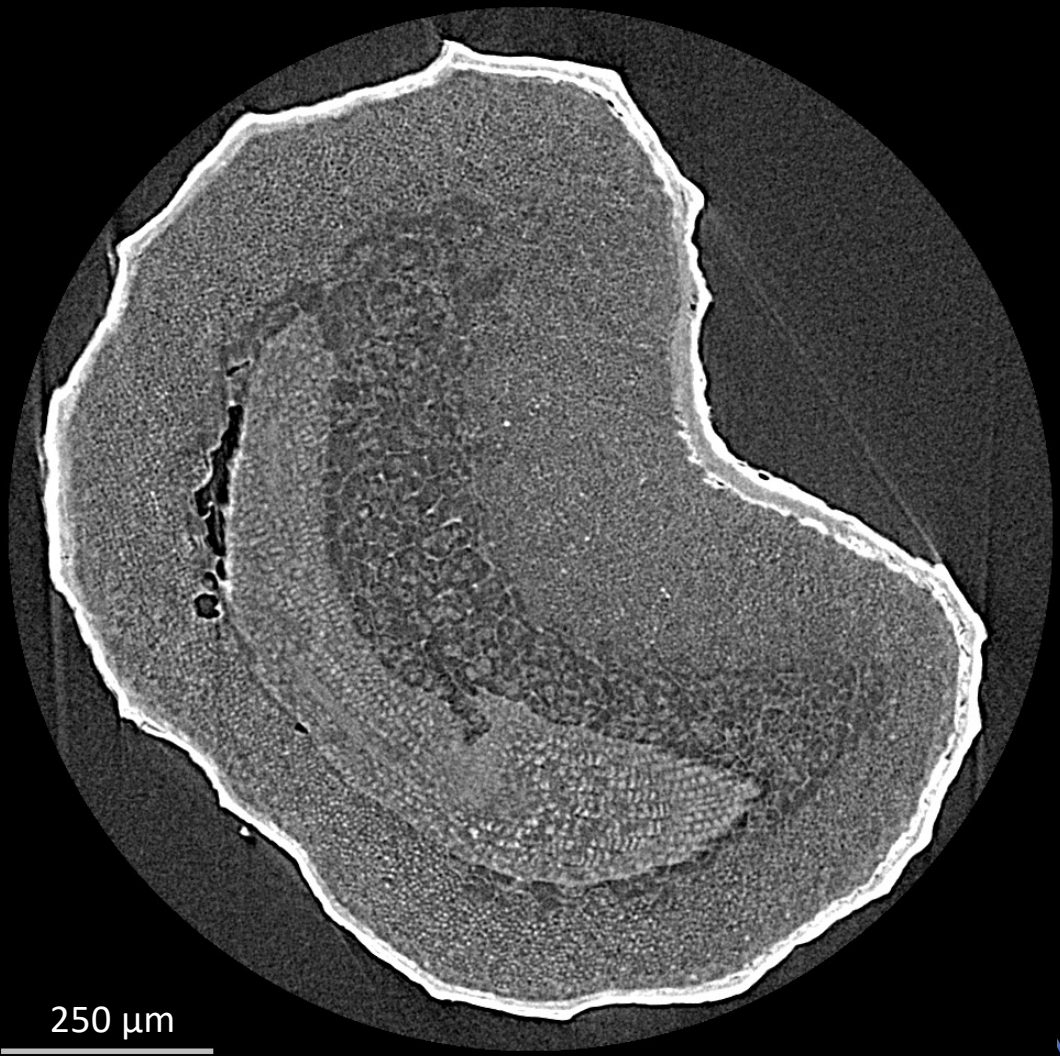
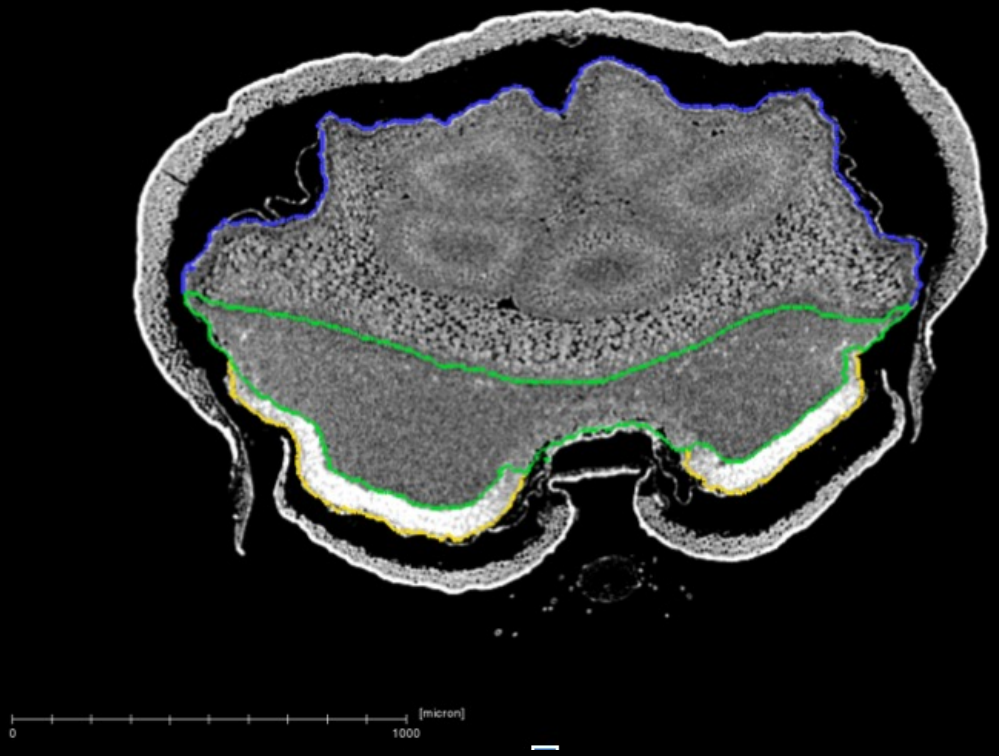
Seeds: Internal structure of a poppy seed



Seeds: Analysis of structures in barley and oat mutants

≈ 4 mm

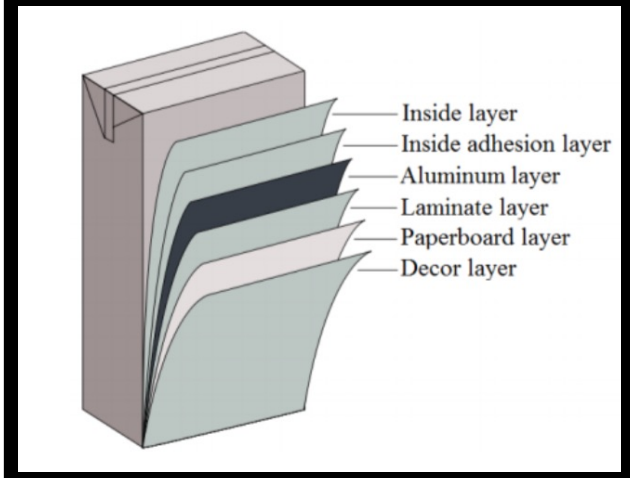


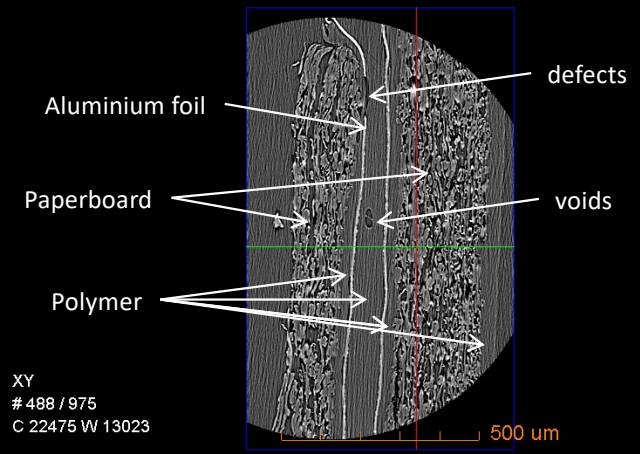
→ Quantification of differences between cereal mutations

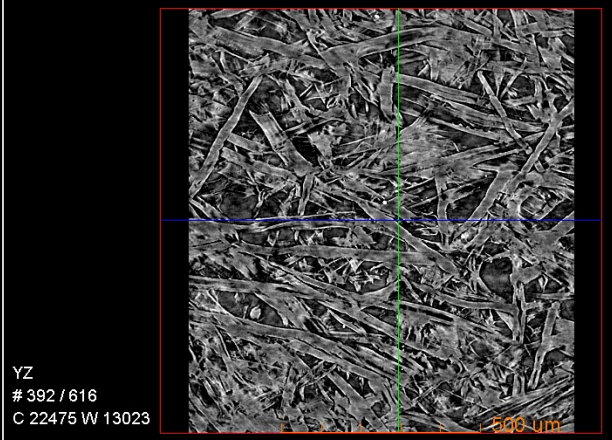
4D IMAGING LAB@LTH

Nick Sirijovski and Mats Hansson @tbiokem.lth.se

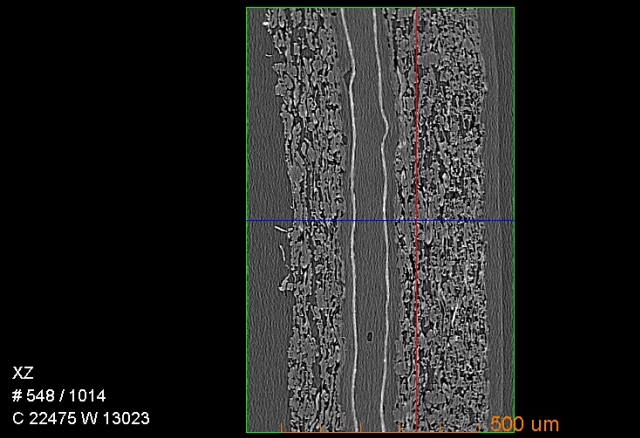
Imaging of packaging materials

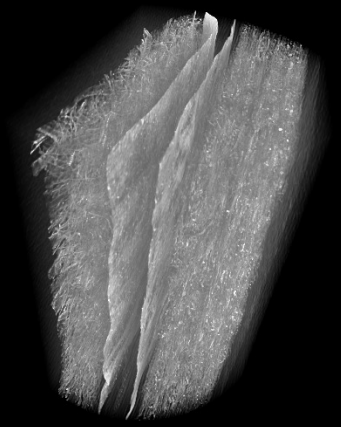






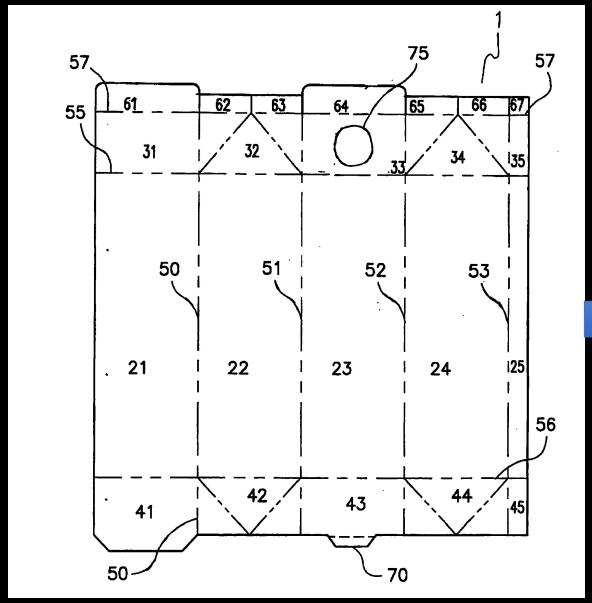
TetraPak recart

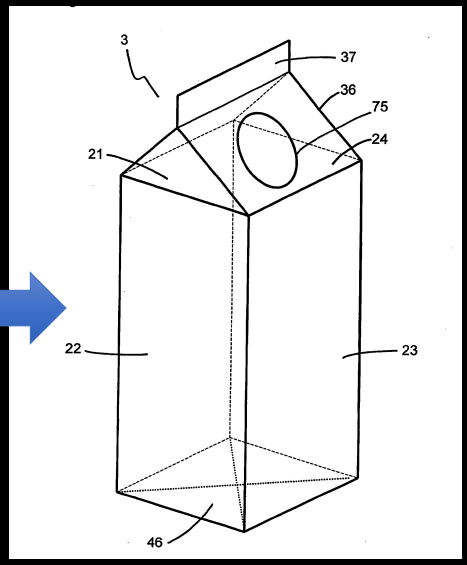




Packaging production

Paperboard package production involves a number of processing steps, including creasing and folding that produce significant deformation of the material…



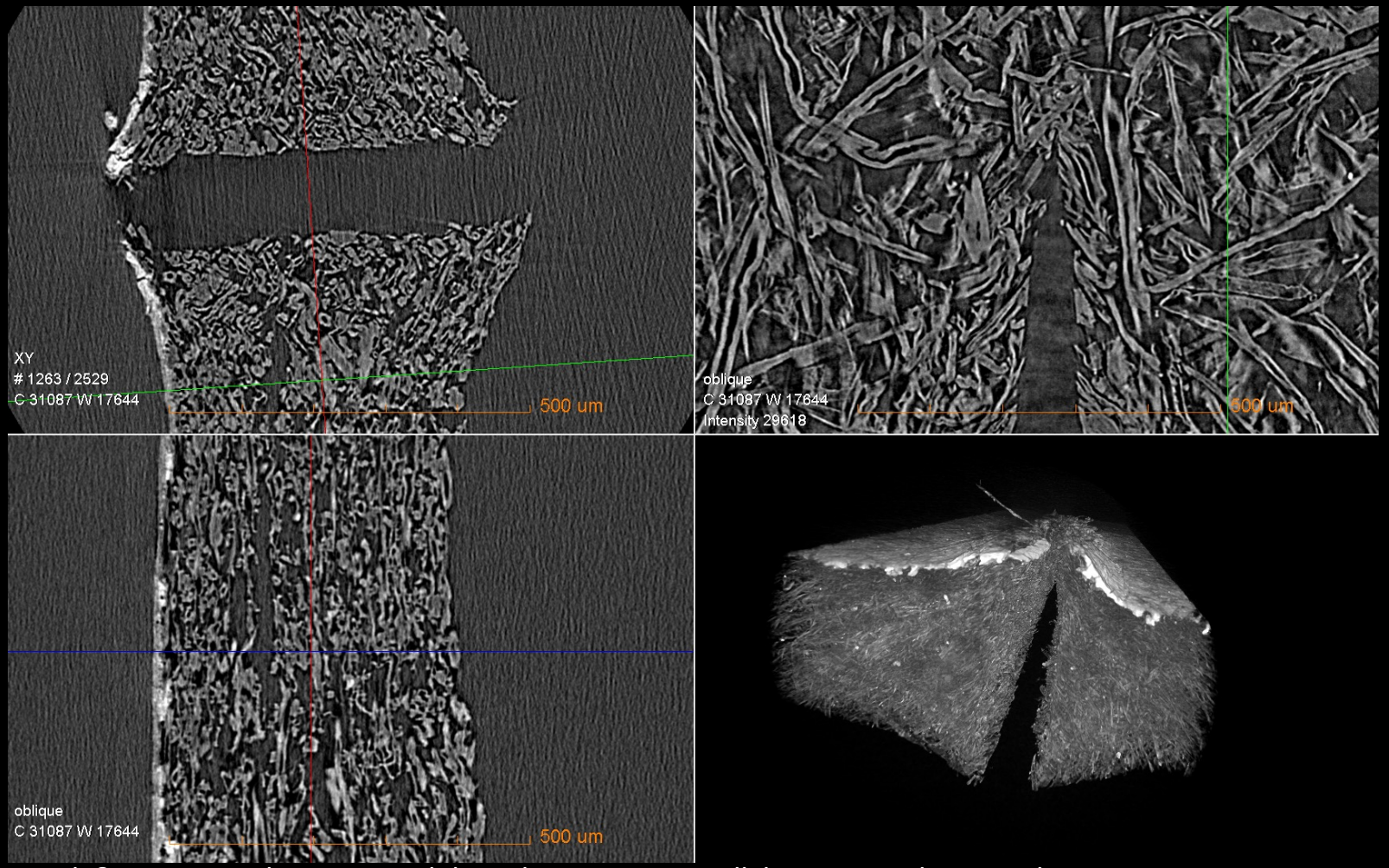


… What happens to the material(s) during these processes?
… How can the processes be optimised to mitigate product failure?

3D imaging can be used to investigate material structure, deformation and process mechanisms

Images from http://www.google.com/patents/EP2394815A1?cl=en

Sharp edge cutting through paperboard



Leek & Jern, MSc Thesis, Div. Solid Mechanics, LTH In Collaboration with TetraPak

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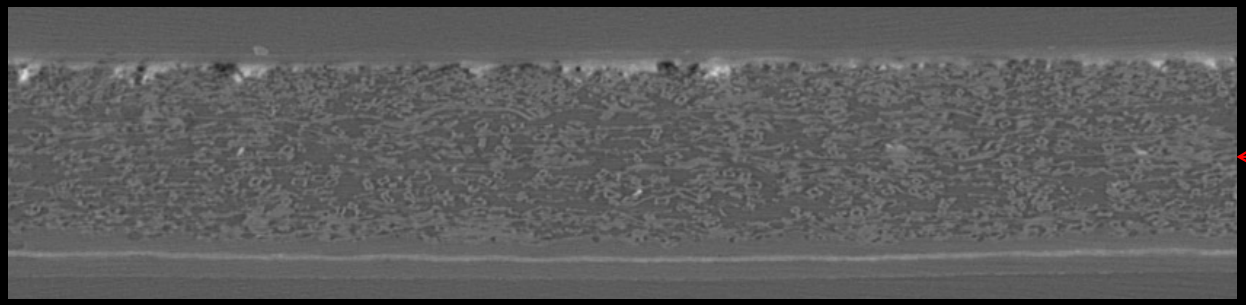
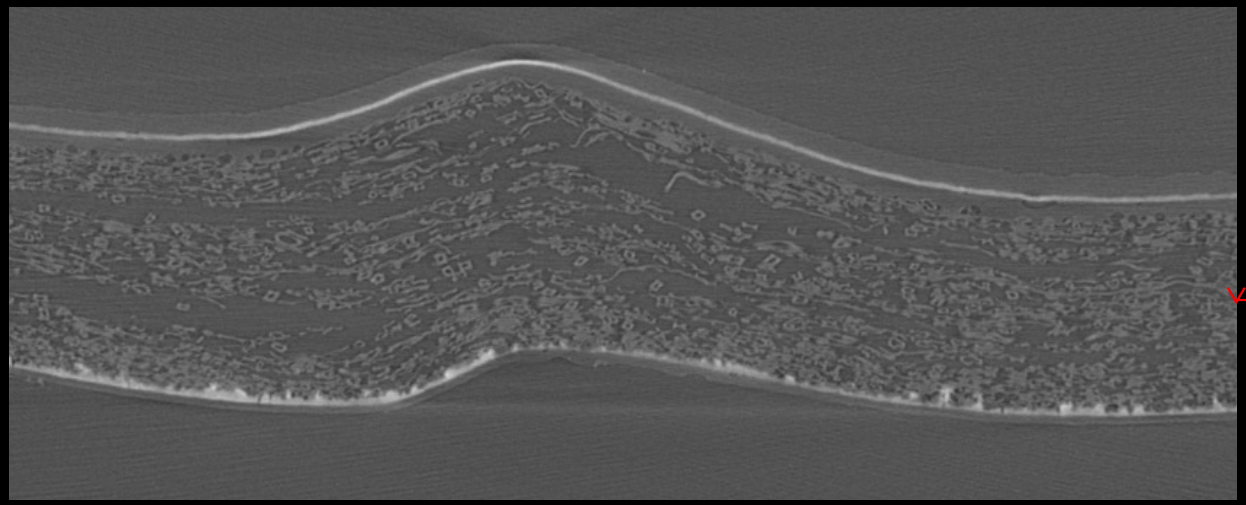
Deformation due to folding

“non-deformed” material

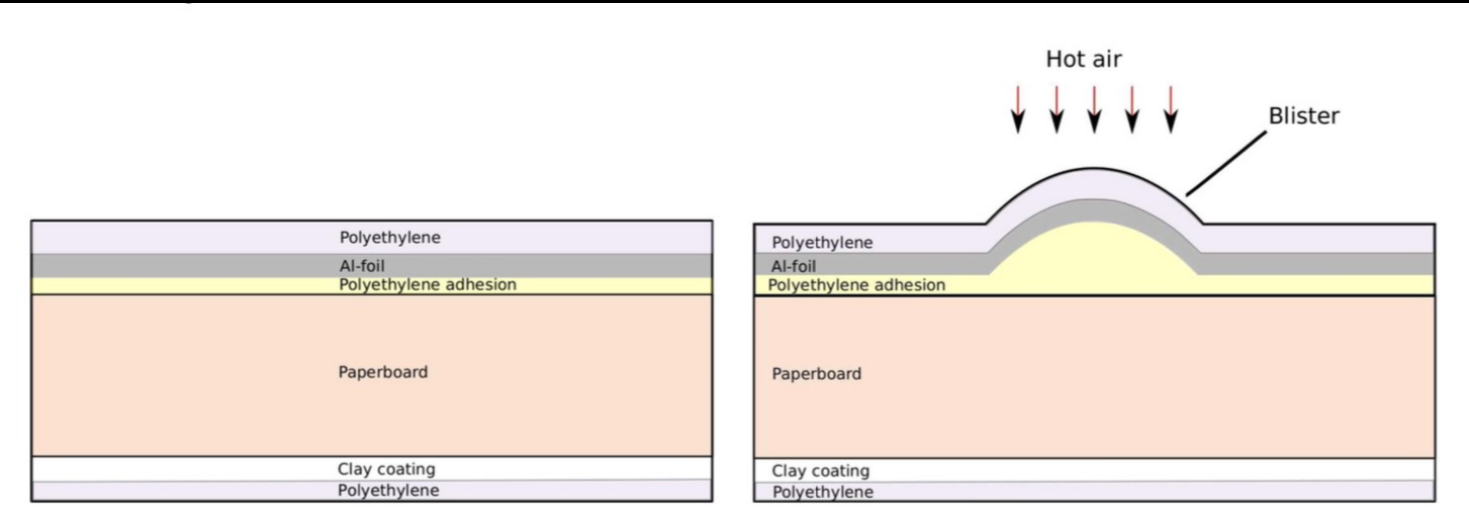


Corner fold



4D IMAGING LAB@LTH

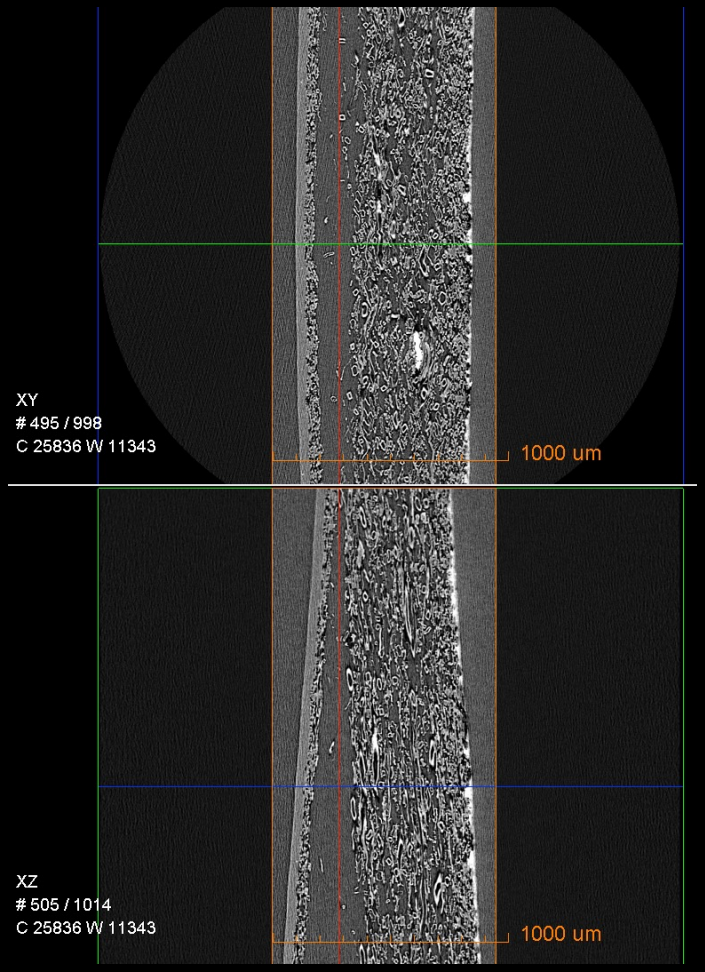
Blistering & Wrinkling in Paperboard

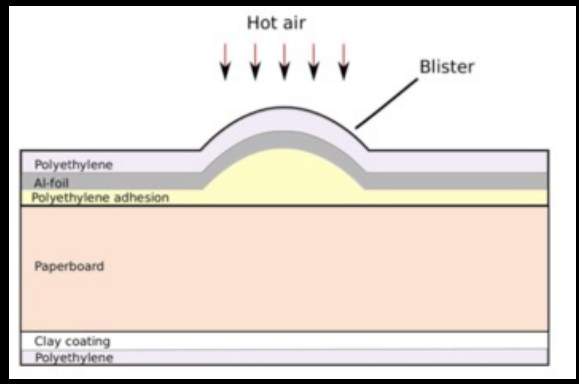


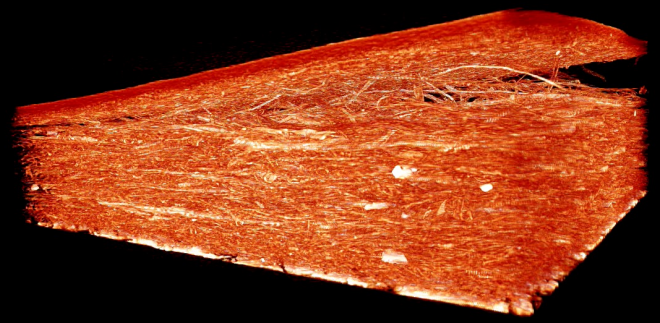
Askfelt & Ristinmaa, 2017

Image from Johansson & Dahlström,
MSc Thesis, Div. Solid Mechanics, LTH
In Collaboration with TetraPak

Blistering in Paperboard

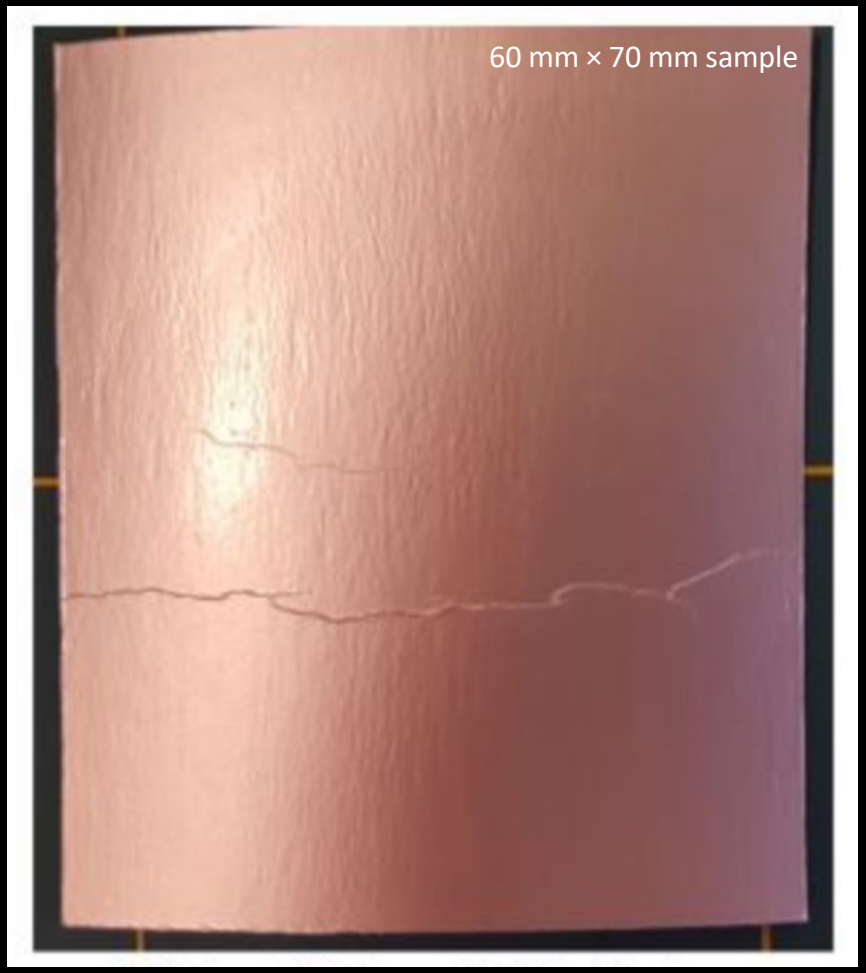


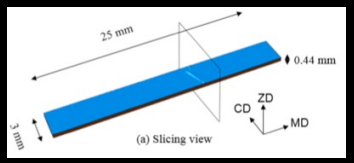


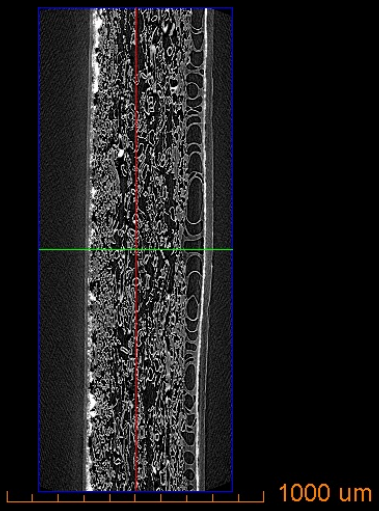


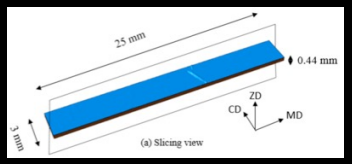
Collaboration with RISE / StoraEnso (SuMo Biomaterials)

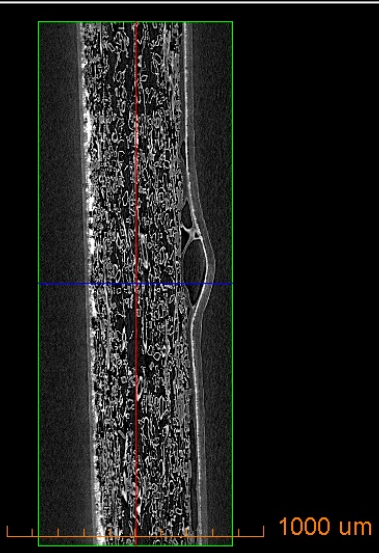
Wrinkling in Paperboard











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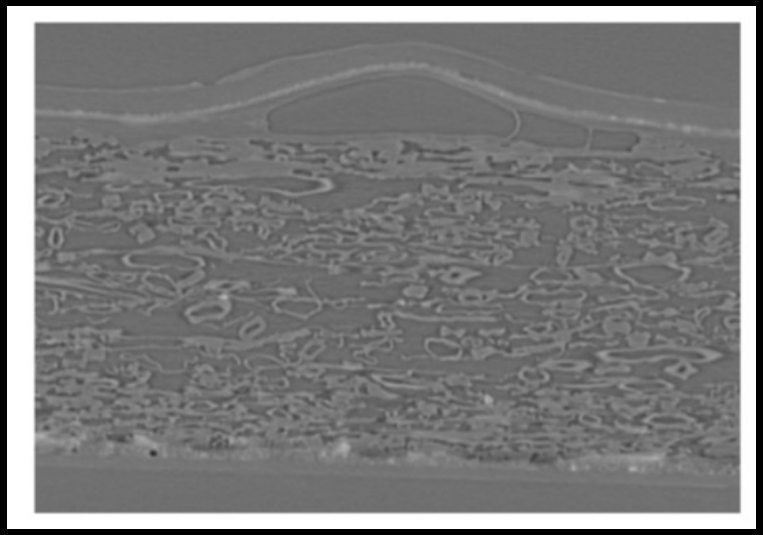
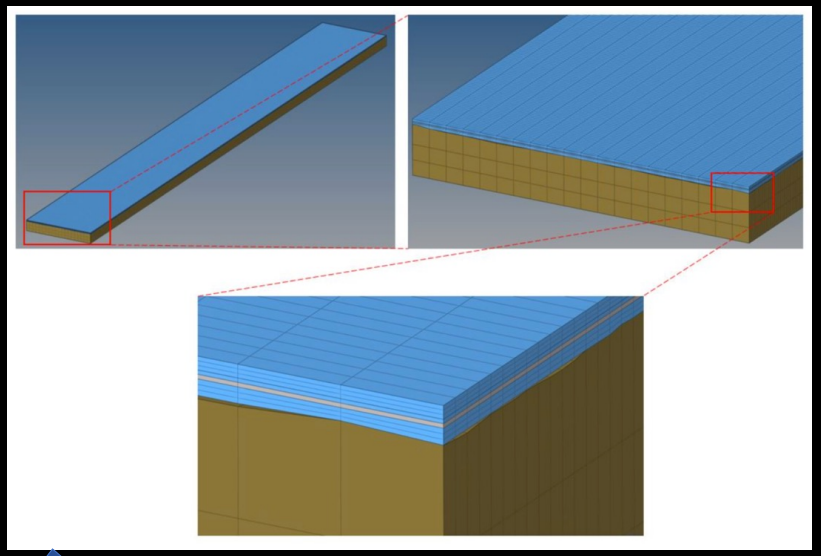
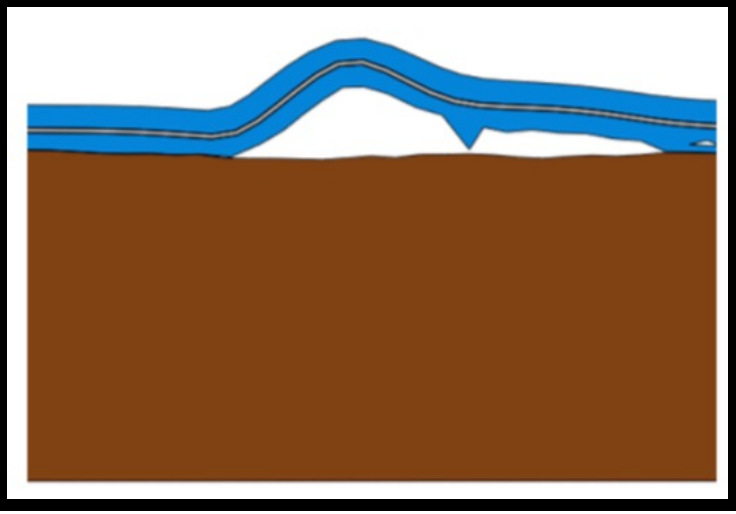
Wrinkling in Paperboard







Johansson & Dahlström, MSc Thesis,
Div. Solid Mechanics, LTH In
Collaboration with TetraPak

4D IMAGING LAB

A Lund university Infrastructure

vision of Solid Mechanics, LTH, Lund

www.solid.lth.se/resources/4d-imaging-lab/

Zeiss Xradia XRM 520

- High resolution 3D x-ray microscope
- 3D imaging resolutions down to < 700 nm (voxel sizes down to 70 nm)
- 30 – 160 kV
- Diffraction Contrast Tomography (DCT) capability

RXSolutions EasyTom150

- Large field of view (flat panel)
- 40- 150 kV
- Large working space
- Faster imaging

TREESEARCH

A Treesearch infrastructure

4D IMAGING LAB

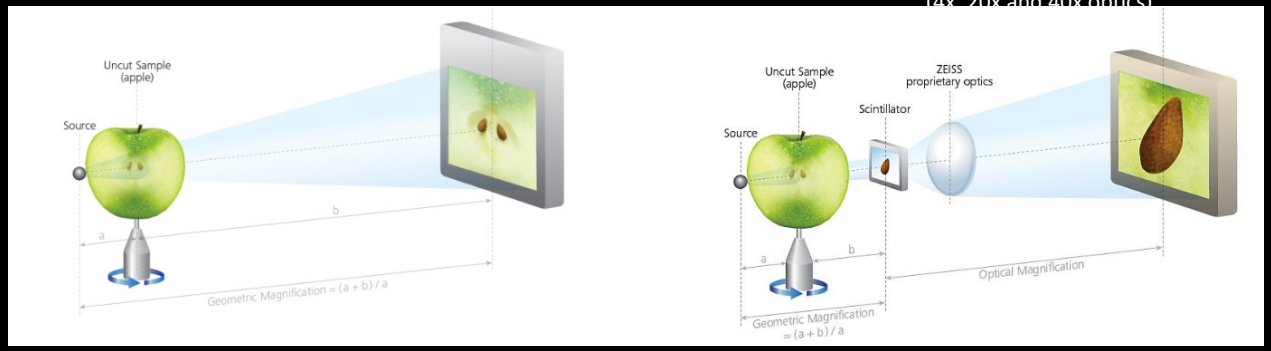
A Lund university Infrastructure

- Zeiss Xradia XRM 520 high resolution lab x-ray microscope

Geometrical magnification

Secondary optical magnification

(4x, 20x and 40x optics)



- High thermal and mechanical stability and high precision positioning
- High resolution CCD camera
- Small spot size (around 2 microns)
- 3D imaging resolutions down to < 700 nm

Images courtesy of Carl Zeiss Microscopy

4D IMAGING LAB

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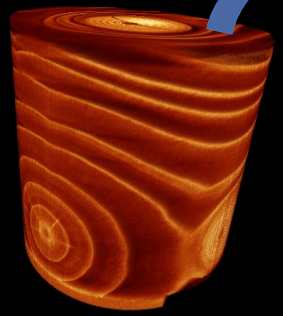
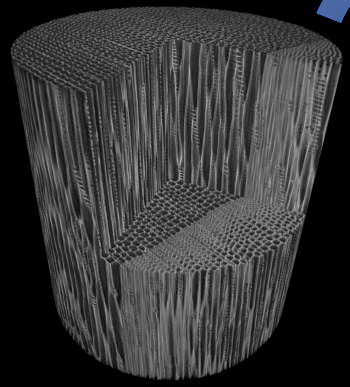
vision of Solid Mechanics, LTH, Lund

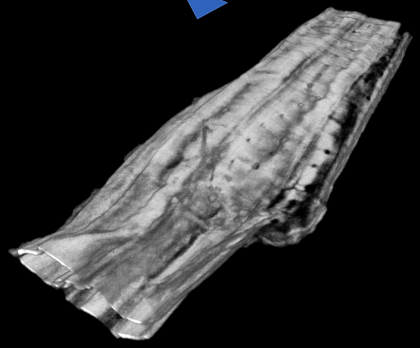
www.solid.lth.se/resources/4d-imaging-lab/



Tomographic image of
Spruce wood
FoV diameter = 49 mm



Tomographic image of a
sample of Spruce wood
FoV diameter = 1.5 mm



Tomographic image
of wood fibres
Voxel size = 300 nm
FoV = 300 microns

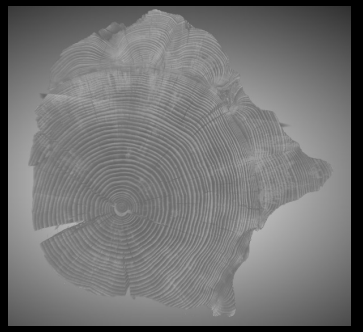
Multiscale

4D IMAGING LAB

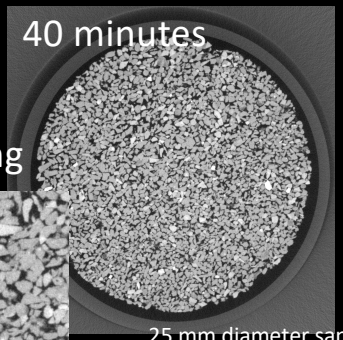
A Lund university Infrastructure

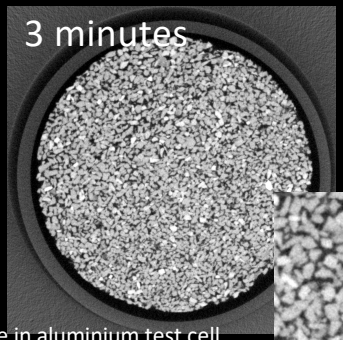
RXSolutions EasyTom150

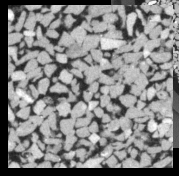
Larger samples
300 mm x 450 mm

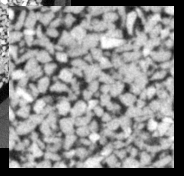


Faster imaging









25 mm diameter sandstone in aluminium test cell