Oak Ridge National Laboratory

Imaging with Neutrons

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August 9, 2023

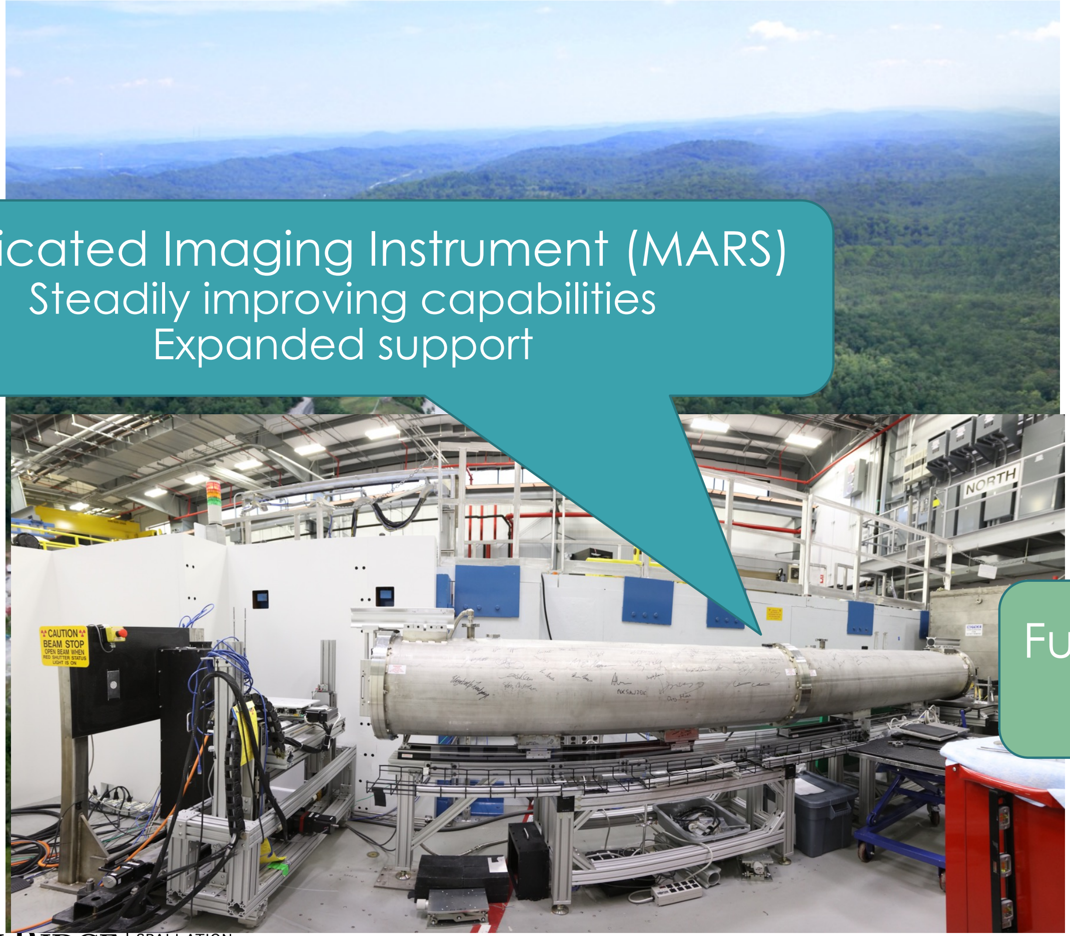
NXS

Neutron Imaging
Oak Ridge National Laboratory

U.S. Department of Energy

Imaging is a Growing Part of the ORNL Neutron Sciences Program

High Flux Isotope Reactor (HFIR)
Intense steady-state neutron flux
and a high-brightness cold neutron source



Dedicated Imaging Instrument (MARS)
Steadily improving capabilities
Expanded support

Spallation Neutron Source (SNS)
World's most powerful accelerator-based neutron source

VENUS is under construction (start of commissioning in July 2024)

Future CUPI^{2}D beamline at STS (Bragg edge and grating interferometry)

The Neutron Imaging Team


Yuxuan Zhang,
HFIR MARS Scientist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Venkatakrishnan, Adv.
Reconstruction
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Shimin Tang, Artificial
Intelligence, Machine
Learning, Hyperspectral
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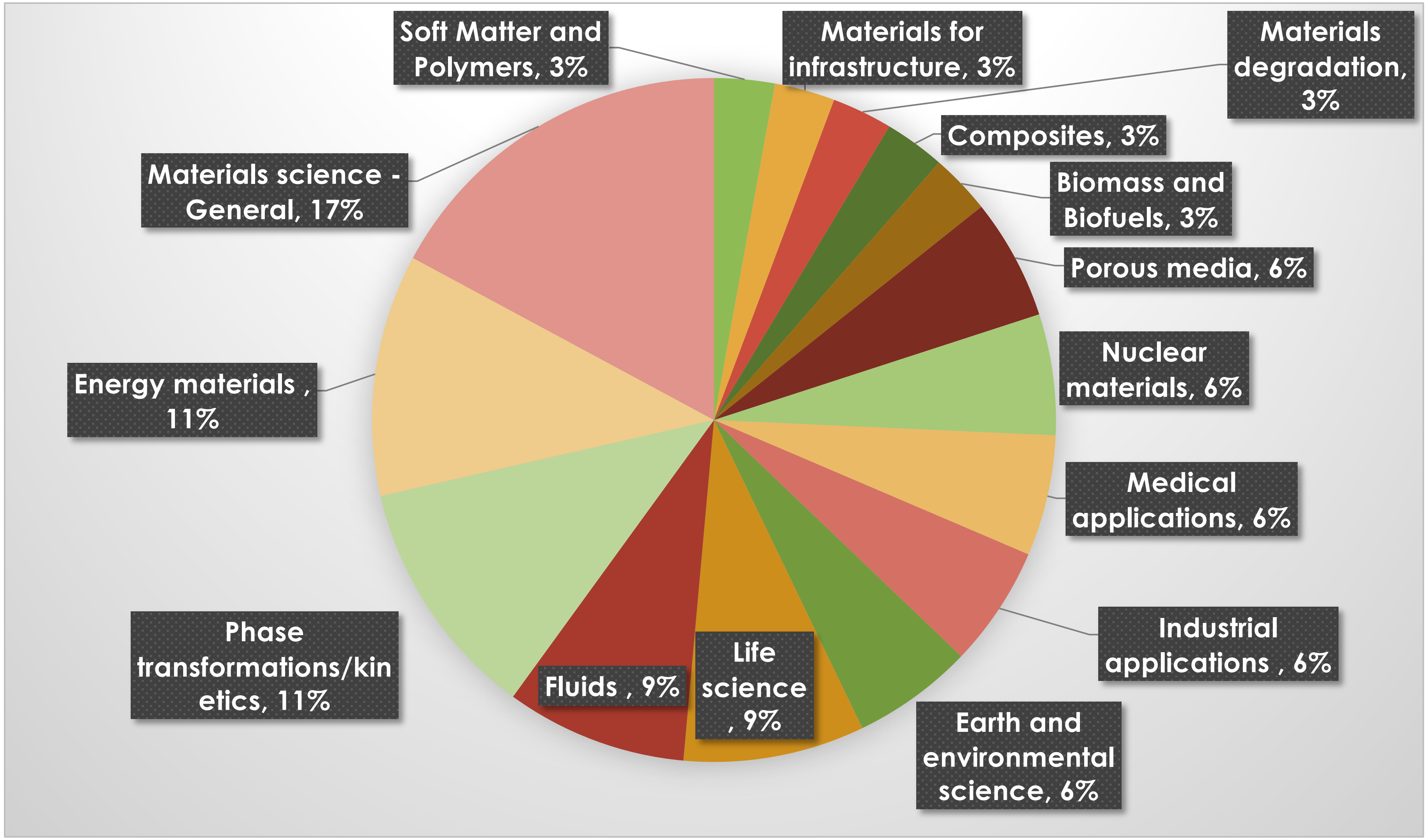
Mary-Ellen Donnelly,
Imaging SA


Harley Skorpenske,
SNS Group Leader


Jamie Molaison,
SNS SNAP SA


Hassina Bilheux,
SNS VENUS Scientist

HFIR imaging has a broad scientific portfolio



Outline

- Imaging at the High Flux Isotope Reactor MARS beamline:
  - Principle of neutron radiography and computed tomography at a continuous source
  - The CG-1D imaging beamline
  - Examples

Yuxuan

- Imaging at the Spallation Neutron Source:
  - Principle of neutron radiography at a pulsed source
  - Examples
  - The VENUS construction project

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- Software tools for imaging

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Outline

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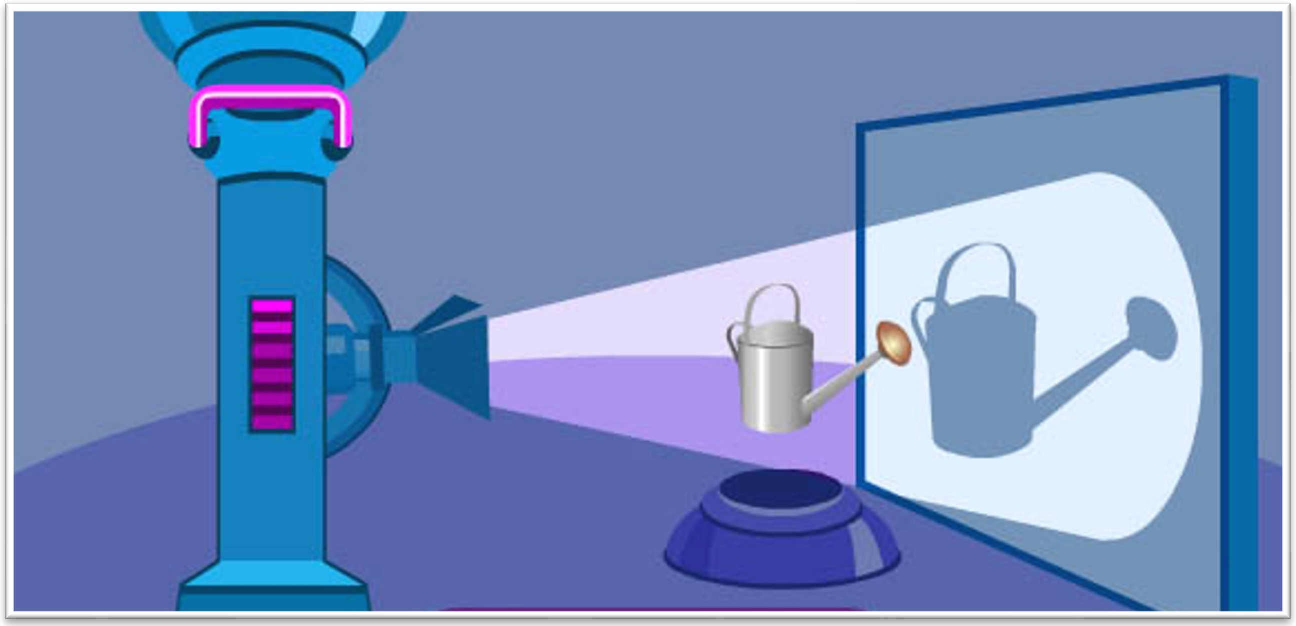
What is Neutron Imaging?

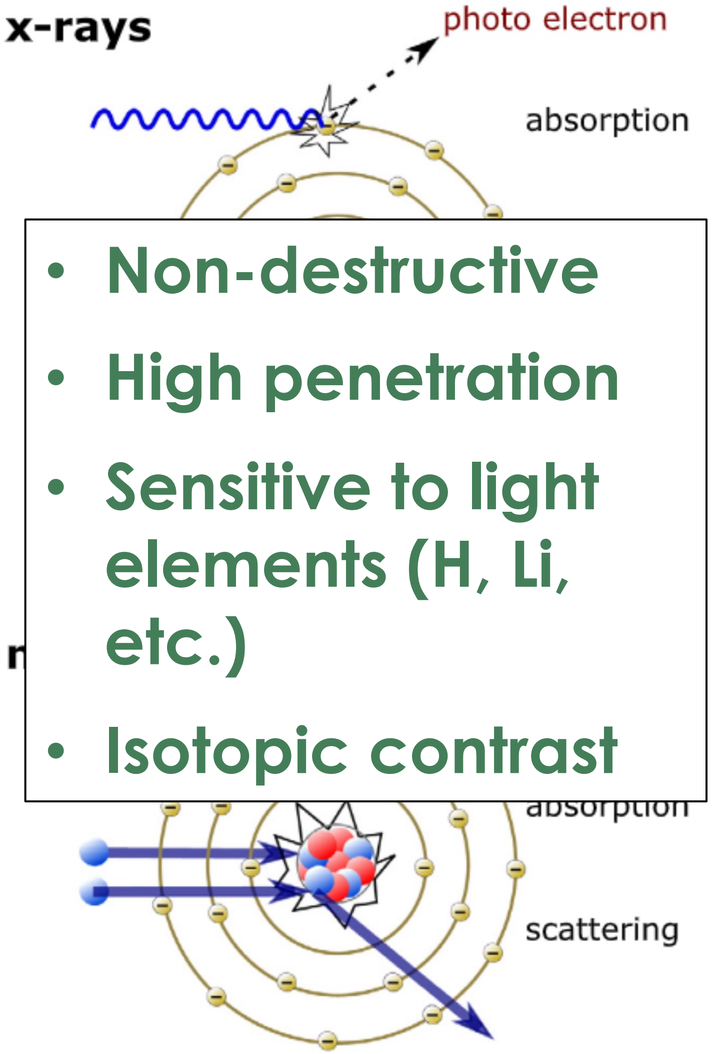
- Similar to X-ray Imaging, Neutron Imaging is a **non-destructive** technique that can **spatially resolve** the structure of a sample



Source **Object** **Detector**

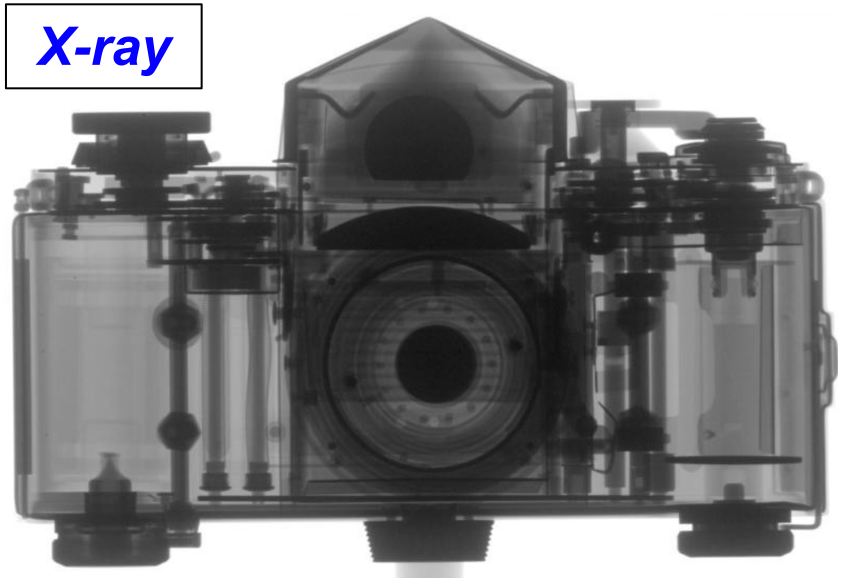
(Figure source: aven.amritalearning.com,. (2013). Shadows and Pin Hole Camera.)

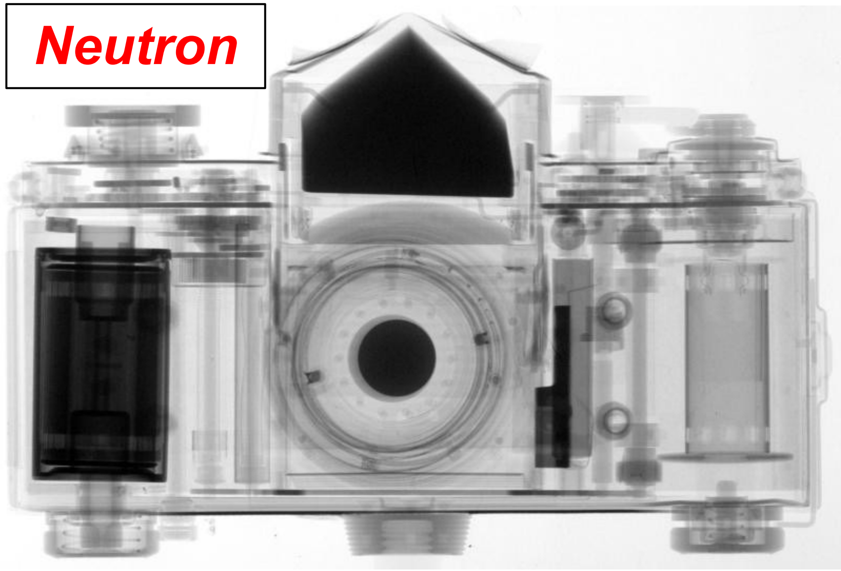
Neutrons interact uniquely with matter (cont'd)



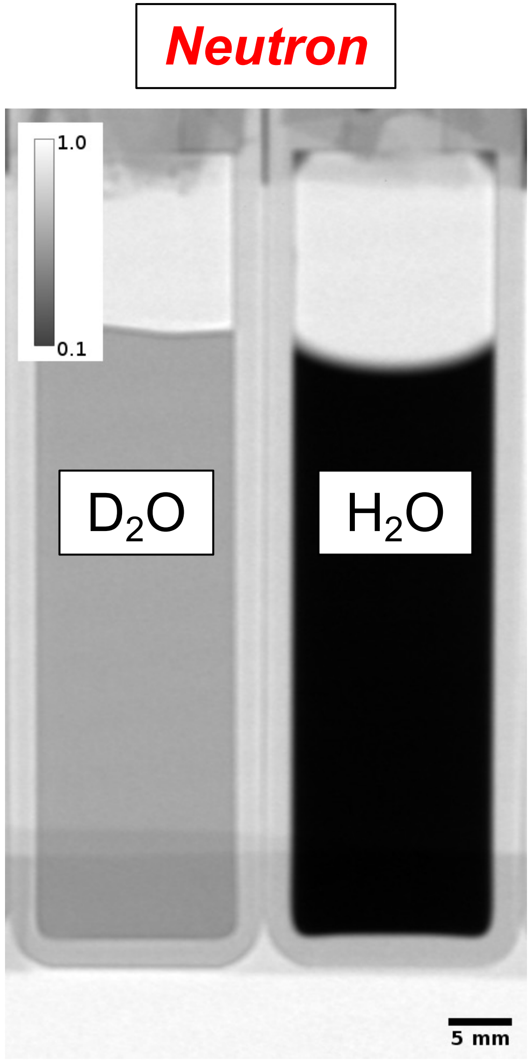
- Non-destructive
- High penetration
- Sensitive to light elements (H, Li, etc.)
- Isotopic contrast

A. Tengattini, et al. Geomechanics for Energy and the Environment 27 (2021)



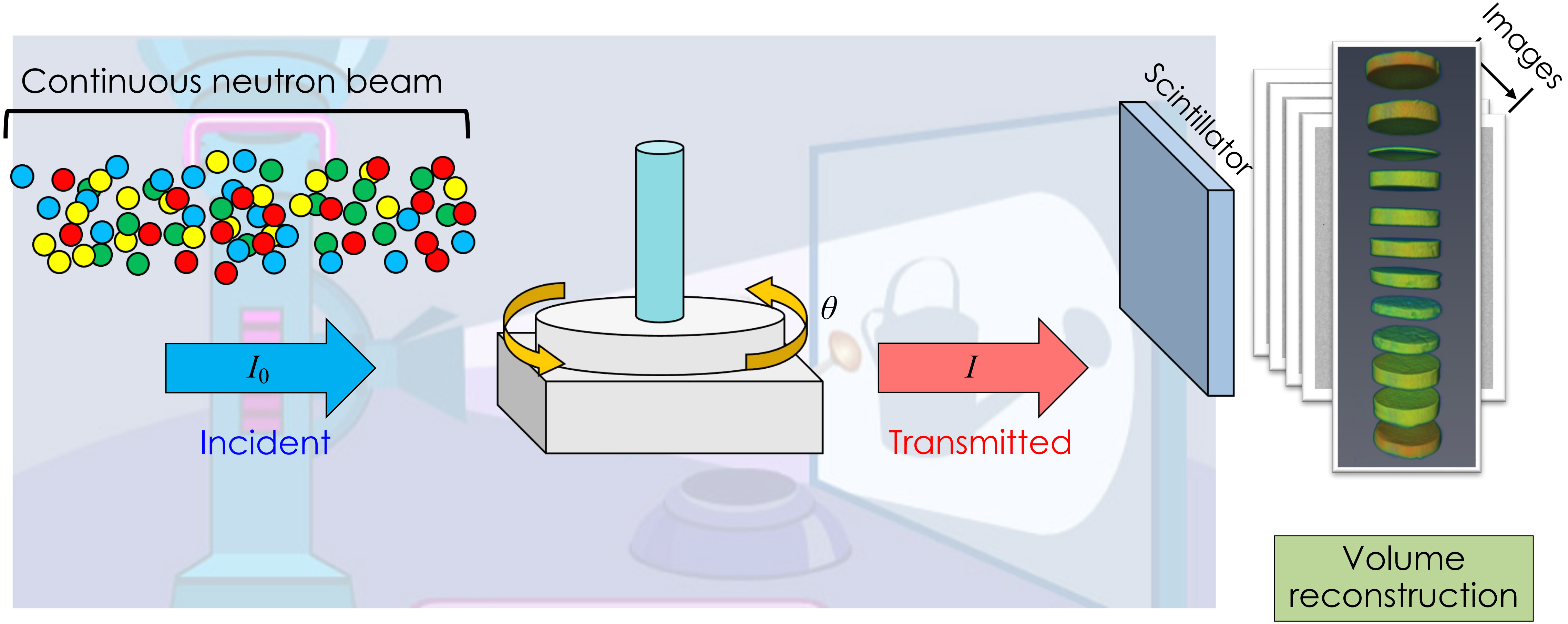


https://www.psi.ch/en/niag/what-is-neutron-imaging

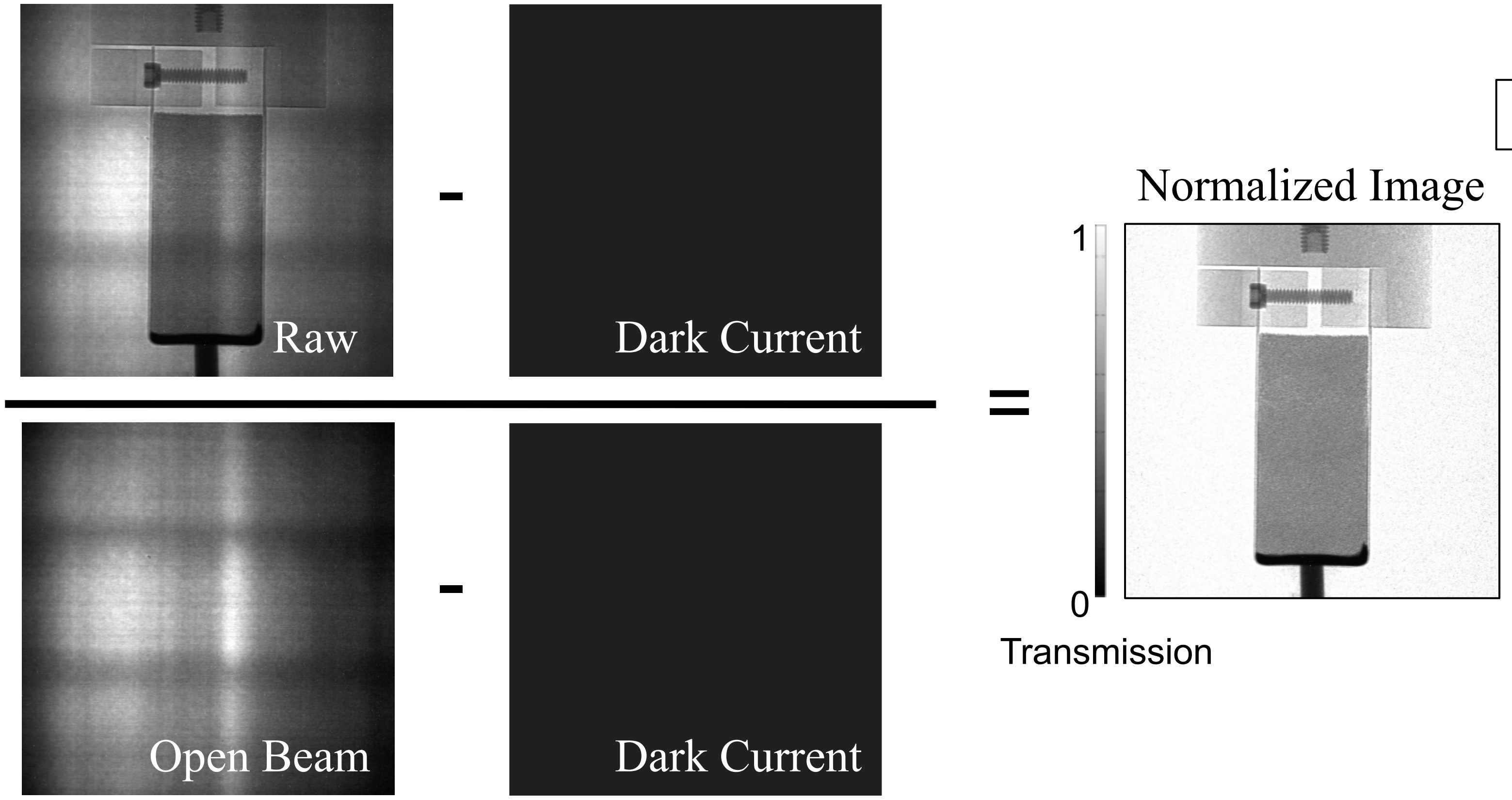


IPTS-26032, Y. Zhang, 2022

Transmitted neutrons recorded as image



From raw image to normalized image



$$\frac{\boldsymbol{Raw}(x,y) - \boldsymbol{DC}(x,y)}{\boldsymbol{OB}(x,y) - \boldsymbol{DC}(x,y)} = \textbf{Normalized Image}$$

Lambert-Beer Law

Transmission

Sample thickness

$$T = \frac{I}{I_0} = e^{-\mu x}$$

Attenuation co.

Avogadro constant

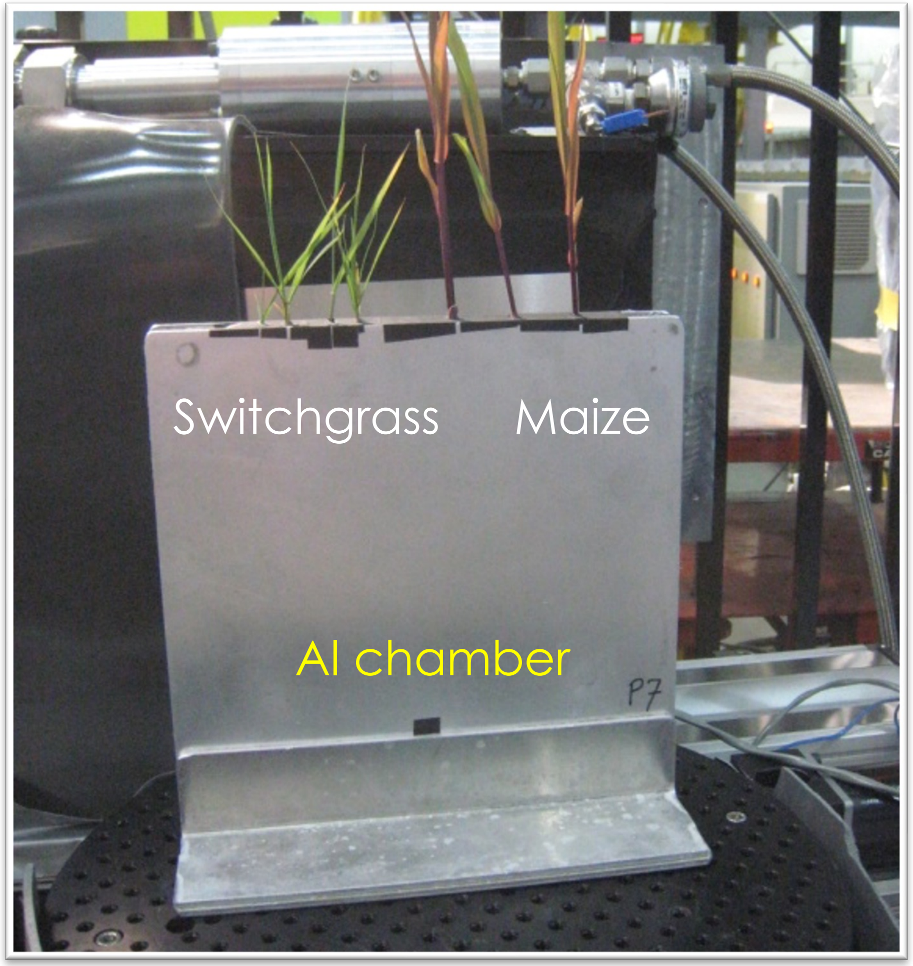
Density

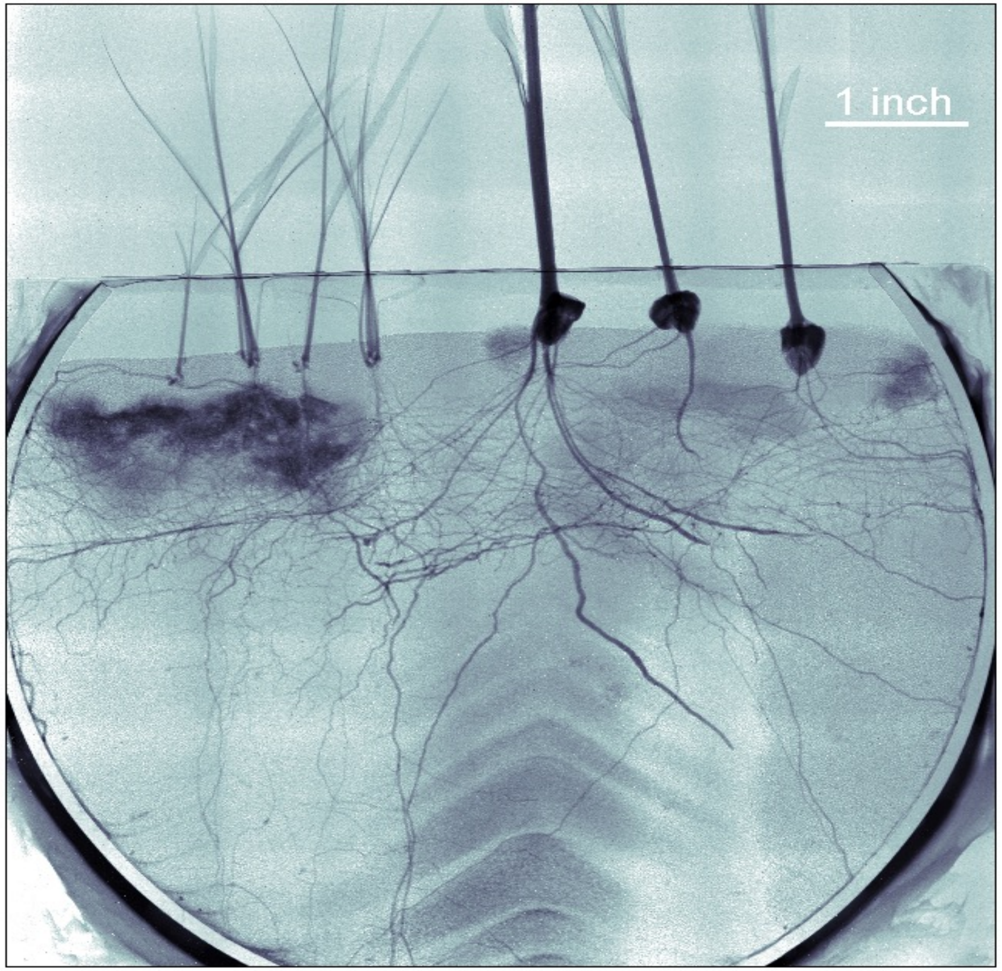
$$\mu = \sigma_{tot} \frac{\rho N_A}{M}$$

Total cross-section

Molar mass

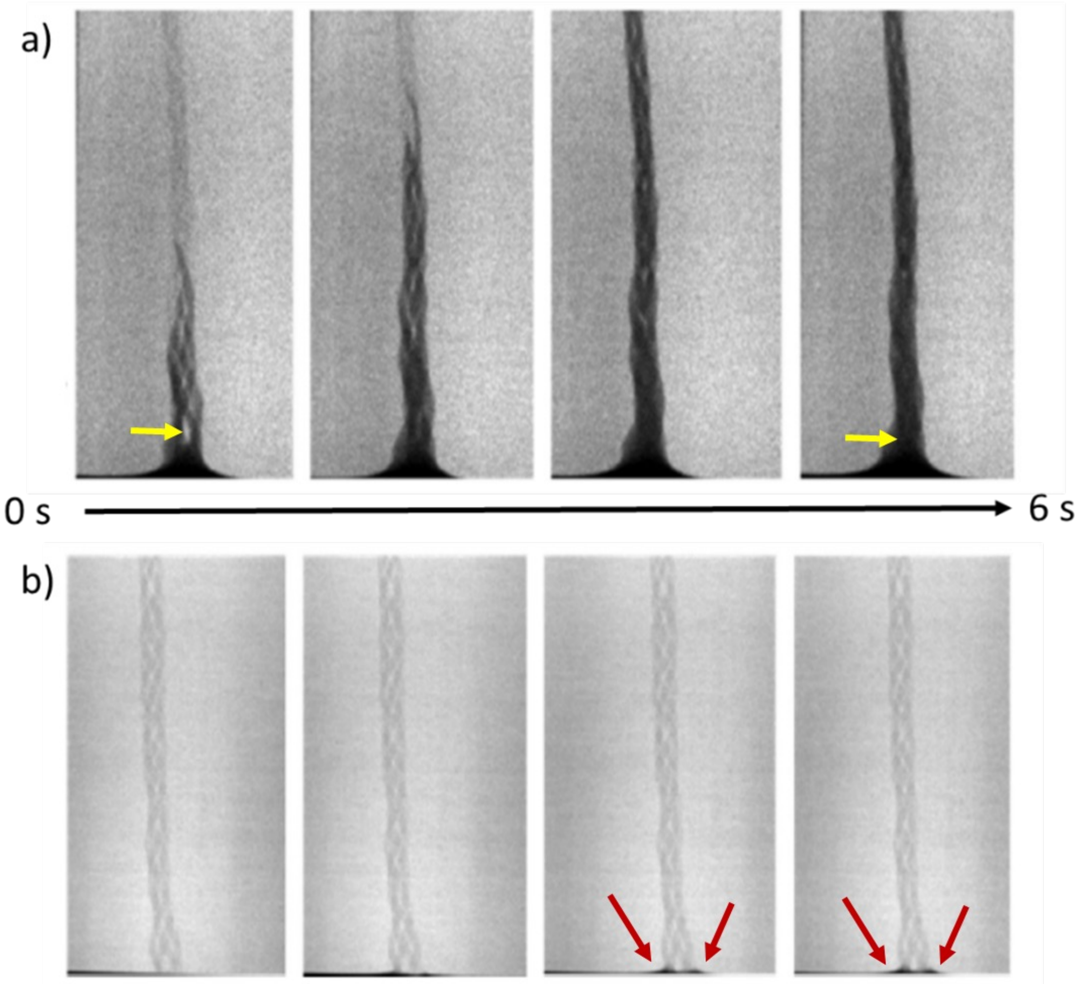
Example: visualize live root system

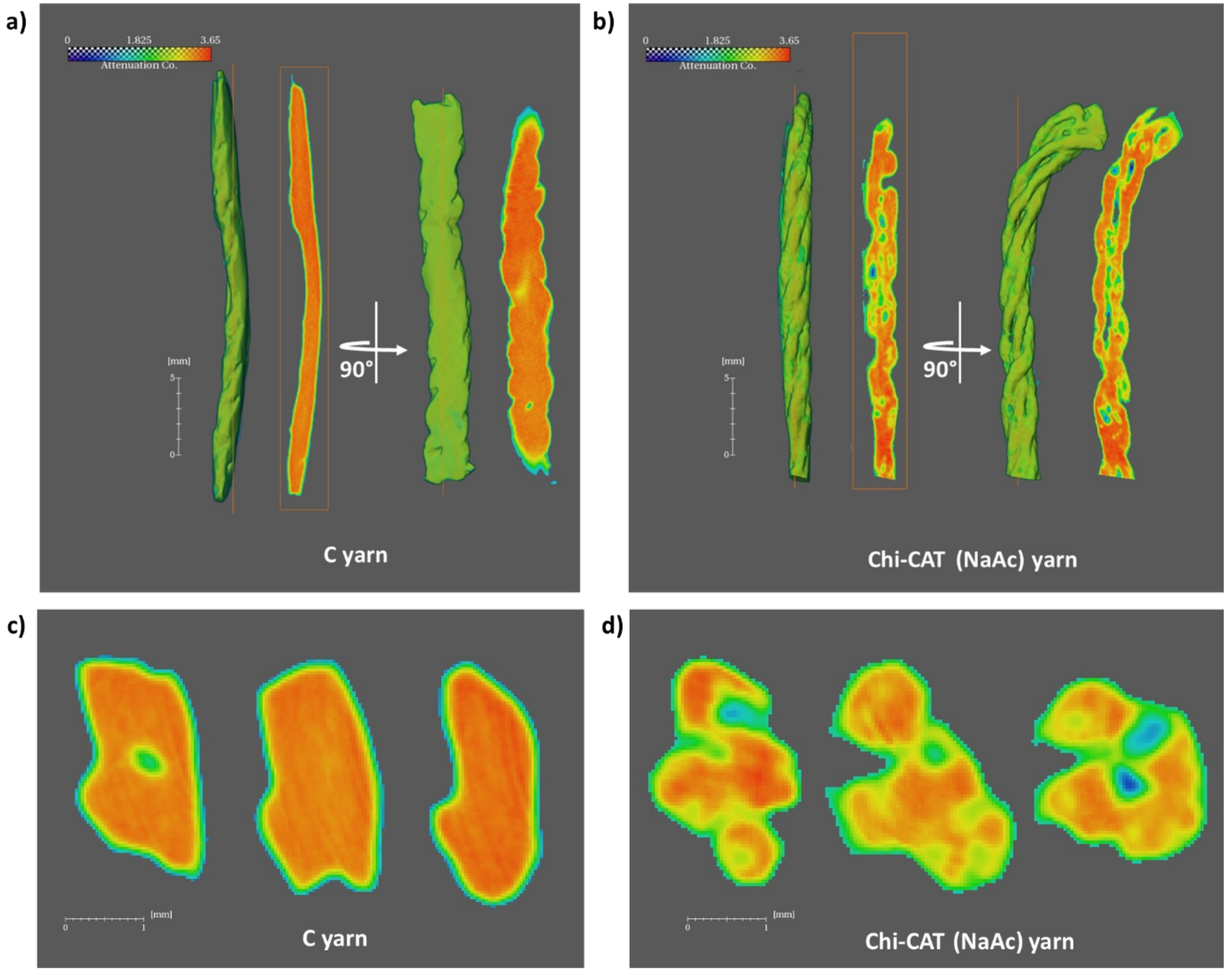




(J. Warren (PI), H, Bilheux, M. Kang, S. Voisin, C. Cheng, J. Horita, E. Perfect, Plant Soil, 2013)

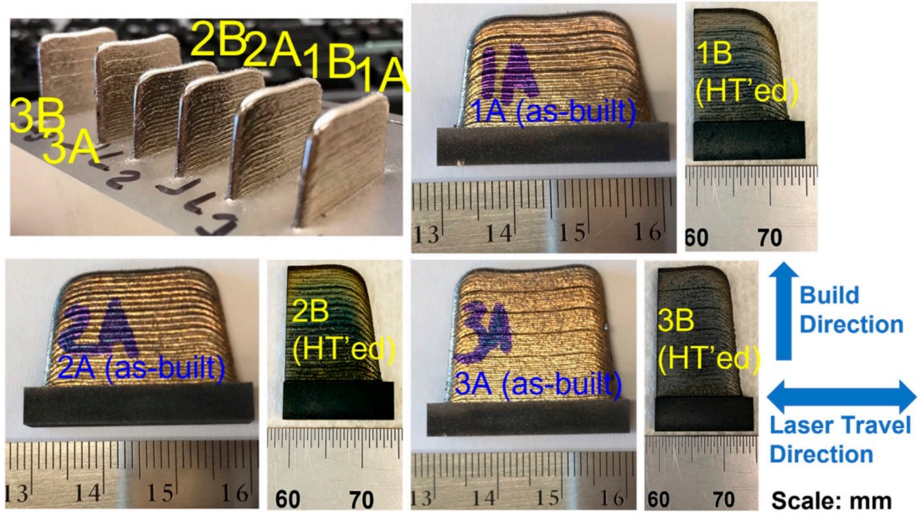
Wicking exp. with yarn

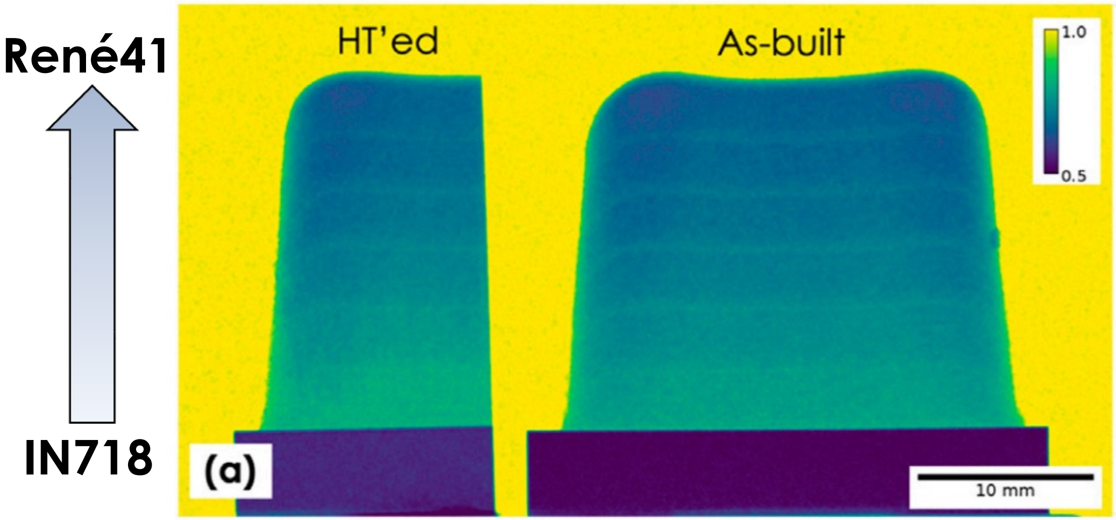


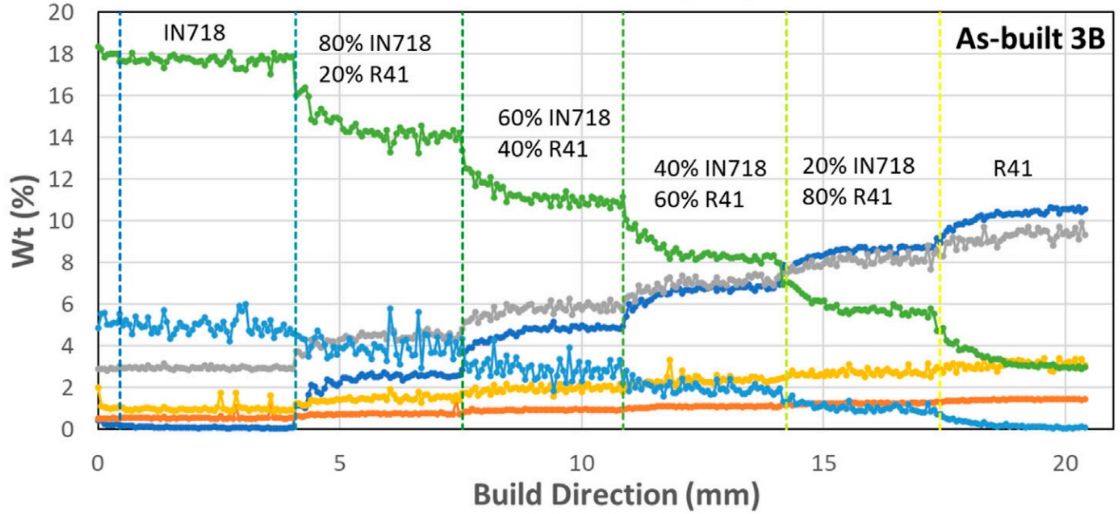


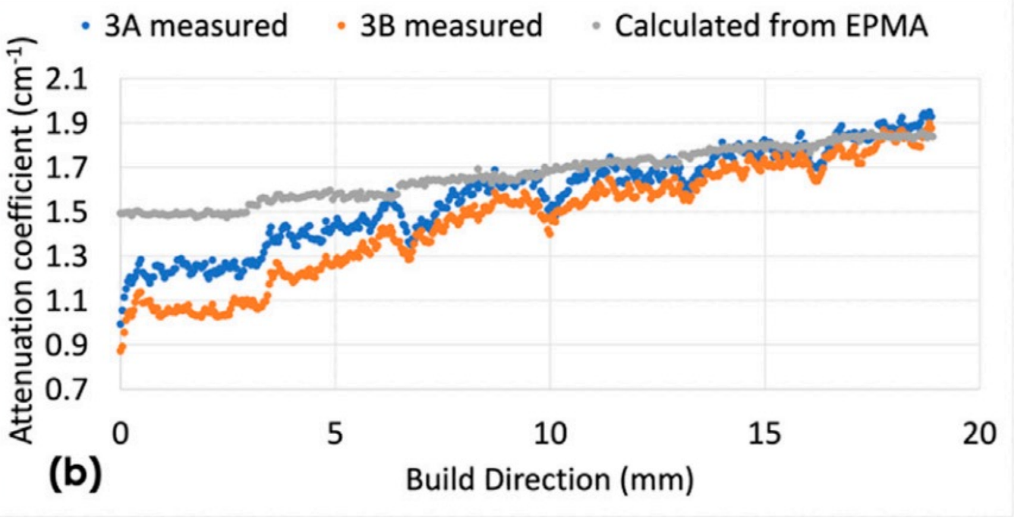
Y. Yuan, Y. Zhang, H. Bilheux, and S. Salmon. "Biocatalytic yarn for peroxide decomposition with controlled liquid transport," *Advanced Materials Interfaces*, 2021, DOI: 10.1002/admi.202002104

Measures the composition gradient in graded superalloy



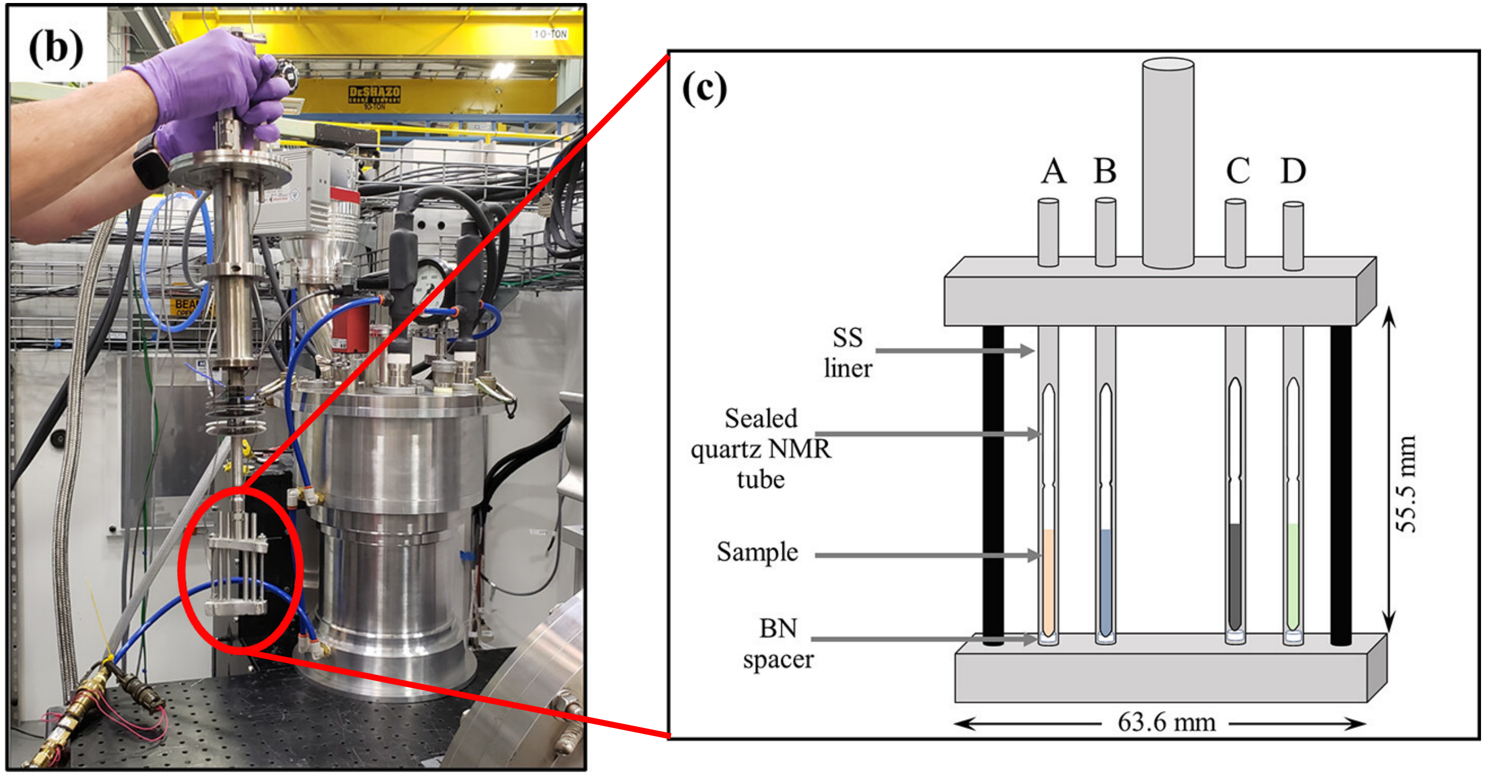


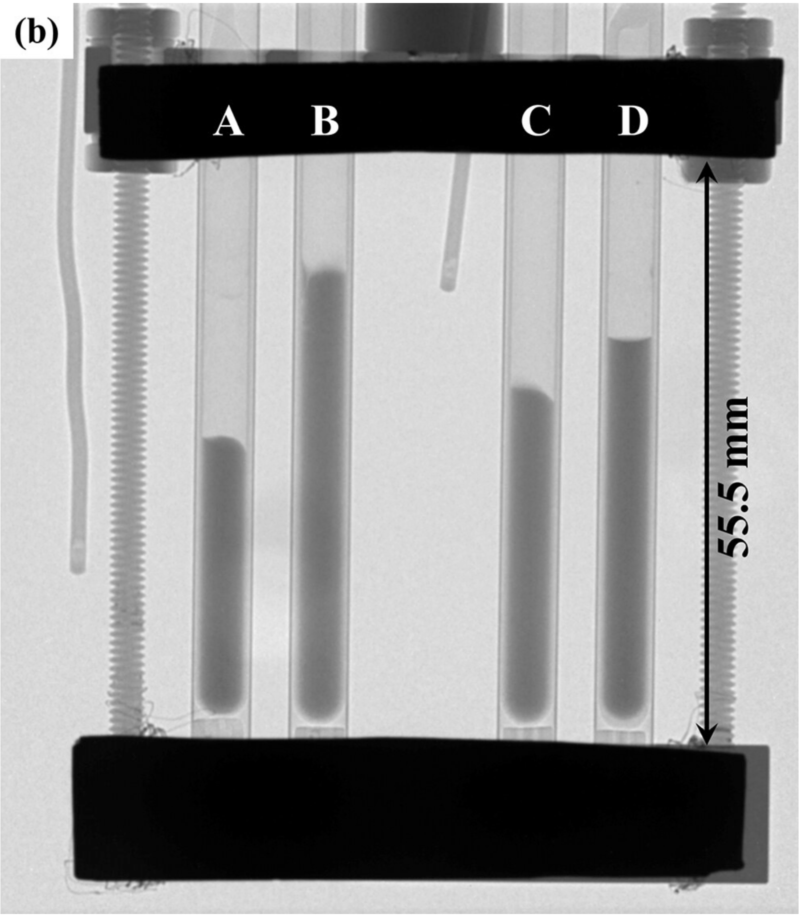




Huang S., Shen C., An K., Zhang Y., Spinelli I., Brennan M., Yu D., ***Frontiers in Metals and Alloys***, 1, 1070562 (**2022**)

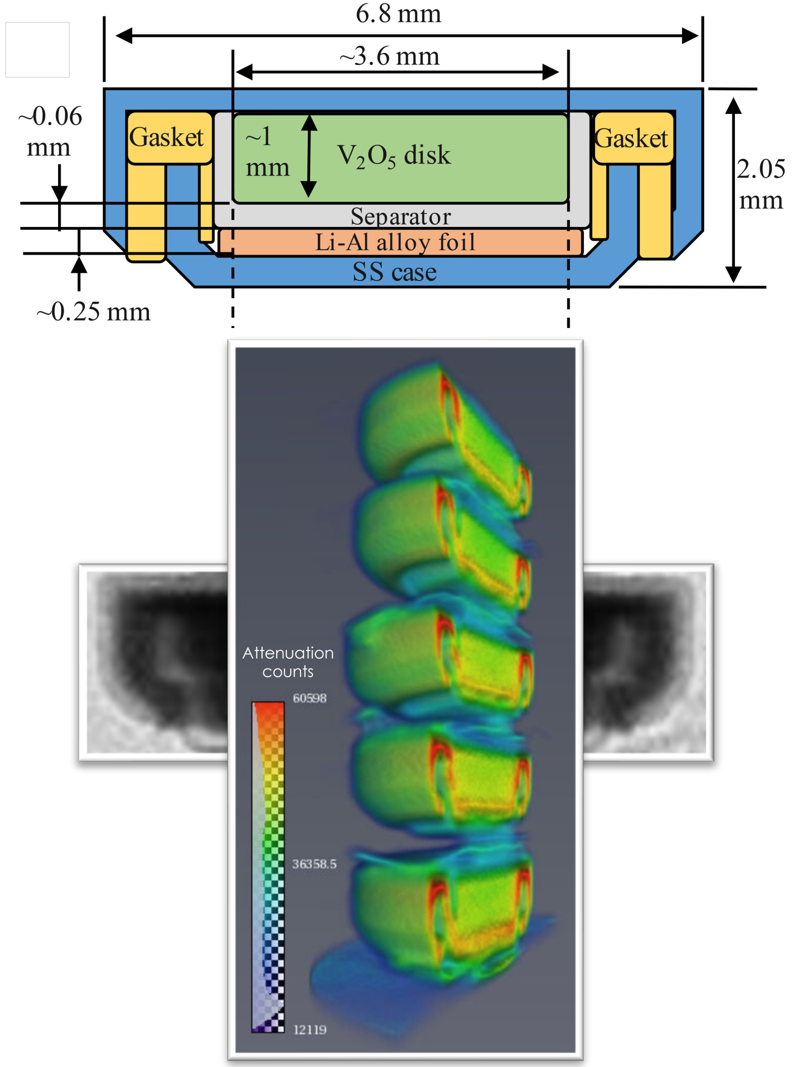
Measures molten salt densities at MSR operating temperatures



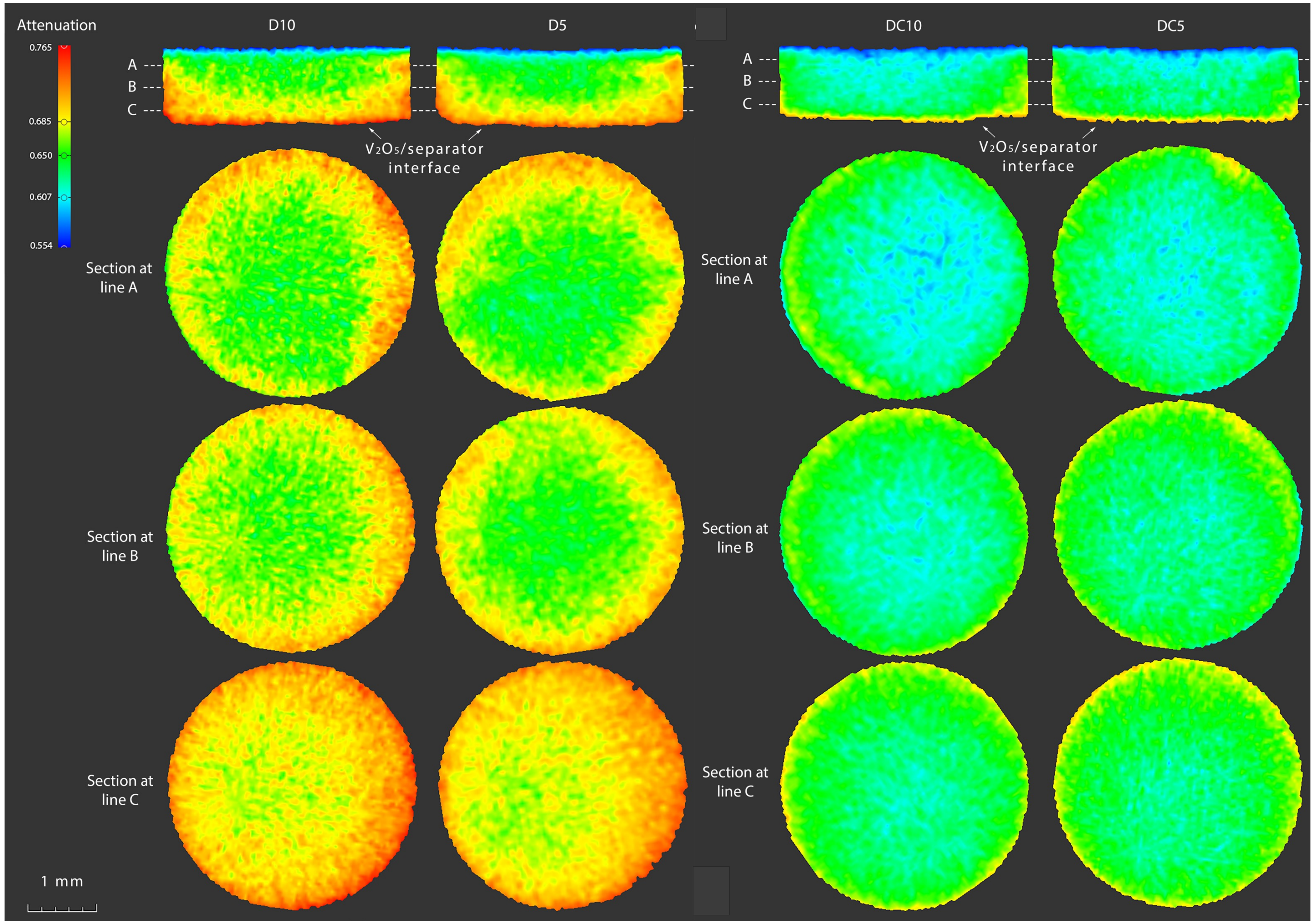


Moon J., Andrews H.B., Agca C., Bilheux J., Braatz A.D., McAlister A., McFarlane J., McMurray J.W., Robb K.R., Zhang Y., ***Industrial & Engineering Chemistry Research***, 61, 17665-17673 (**2022**)

Li distribution is heterogenous and faster C-rates lead to more non-uniformity after lithiation



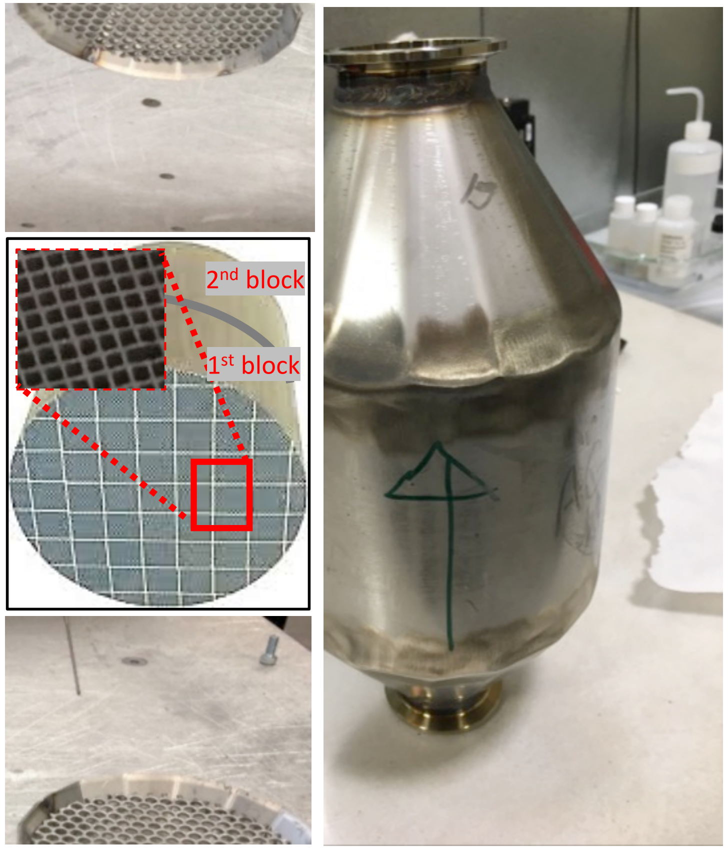
Slow Fast Slow Fast

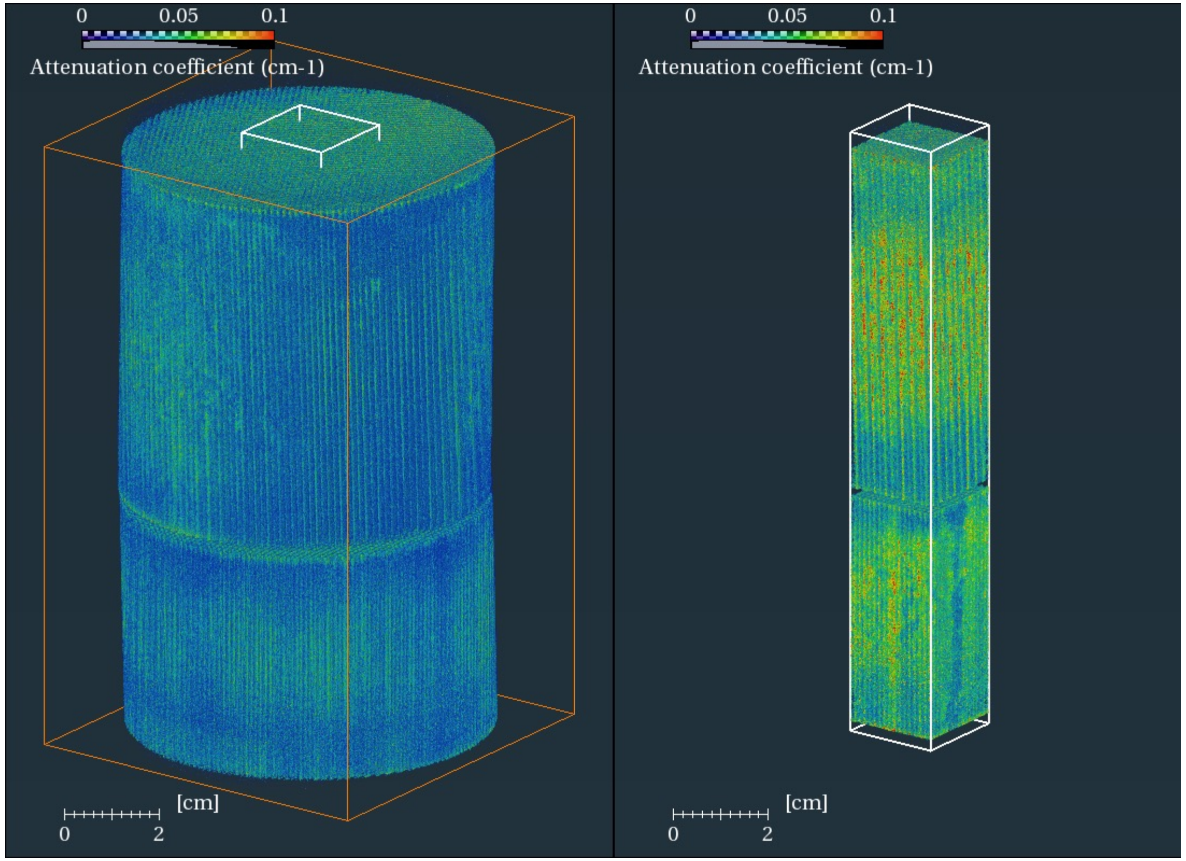


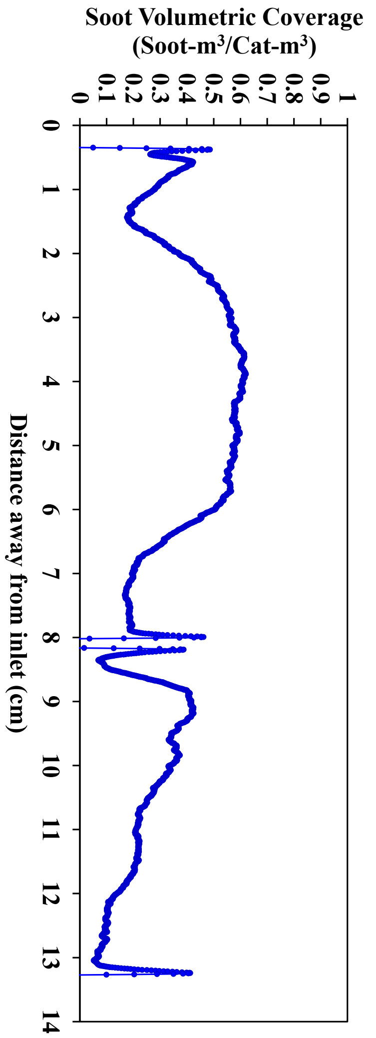
Lithiation Delithiation

Y. ZHANG et al.,, Journal of Power Sources, vol. 376, pp. 125–130, Feb. 2018.

Measures soot distribution in Acidic Gas Reduction device

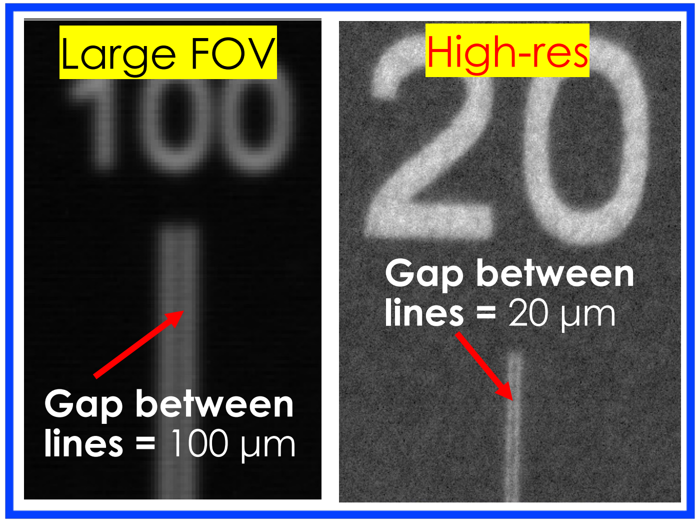


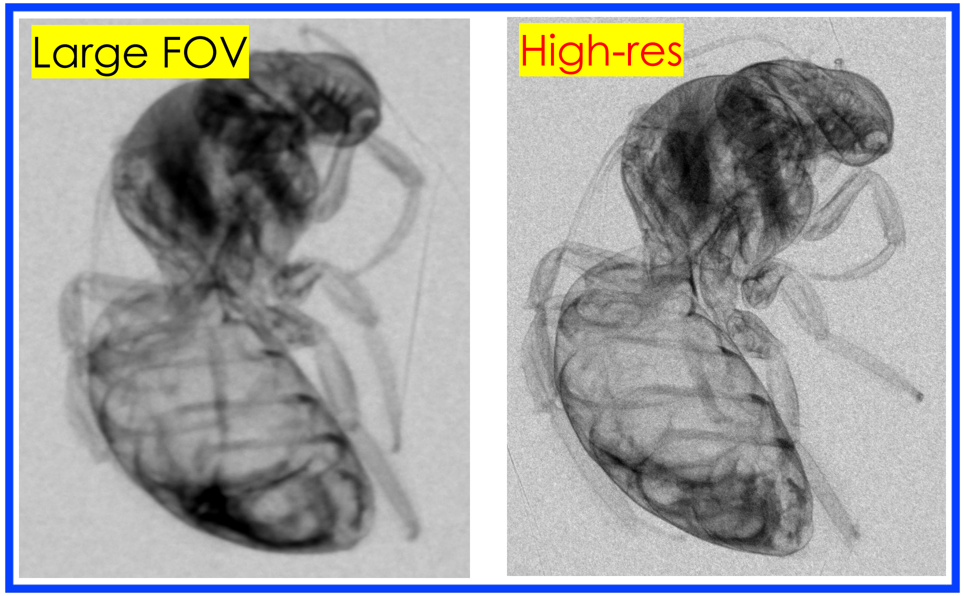


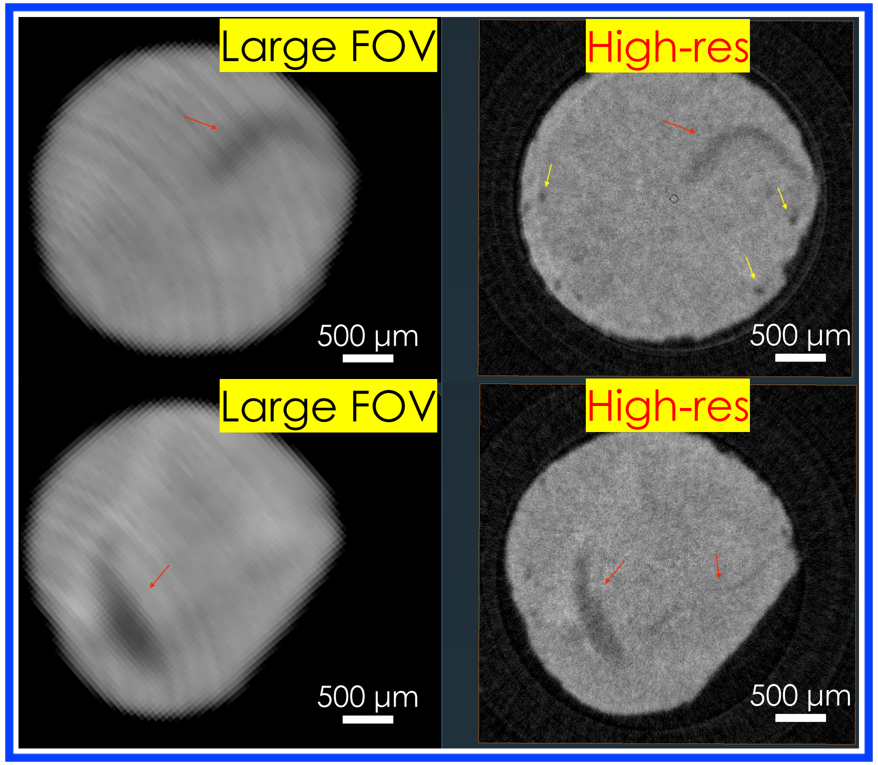


Gao Z., Zhang Y., Qian S., Yang W., Wu Z., Gluesenkamp K.R., Nawaz K., Gehl A., ***Chemical Engineering Journal***, 454, 140099 (**2023**)

Recently improved spatial resolution for finer details





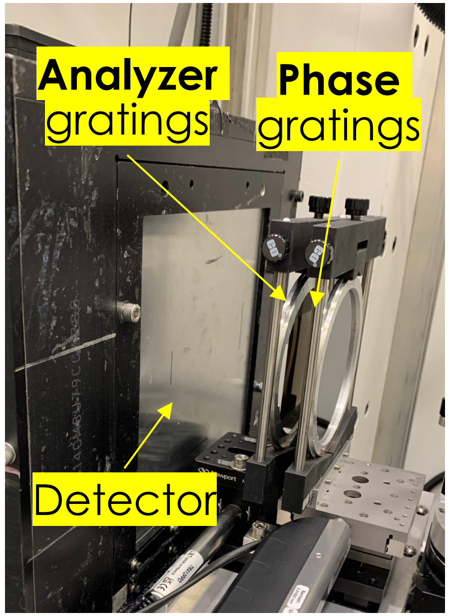


Setup	Max. FOV	CT scan time	Spatial resolution
Large FOV	80 x 80 mm^2	≥7 hours	75-100 µm
High-res	**50 x 50 mm^2**	**≥70 hours**	**20-25 µm**

IPTS-27734, Y. Zhang, 2022

Implementation of nGI to probe smaller length scale

Neutron Grating Interferometry (**nGI**)



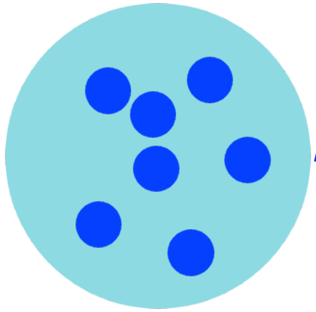
nGI setup at the downstream

nGI setup at the upstream

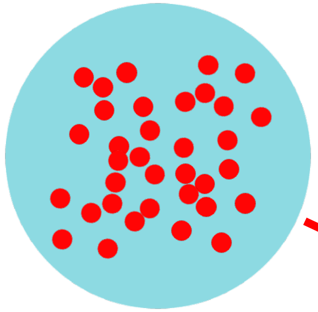
- Spatial resolution stay unchanged
- But sensitive to much smaller length scales (~40 nm – 2.2 μm) ***selectively***

Sensitive to nm or μm size feature ≠ **Spatial resolution** of nm or μm

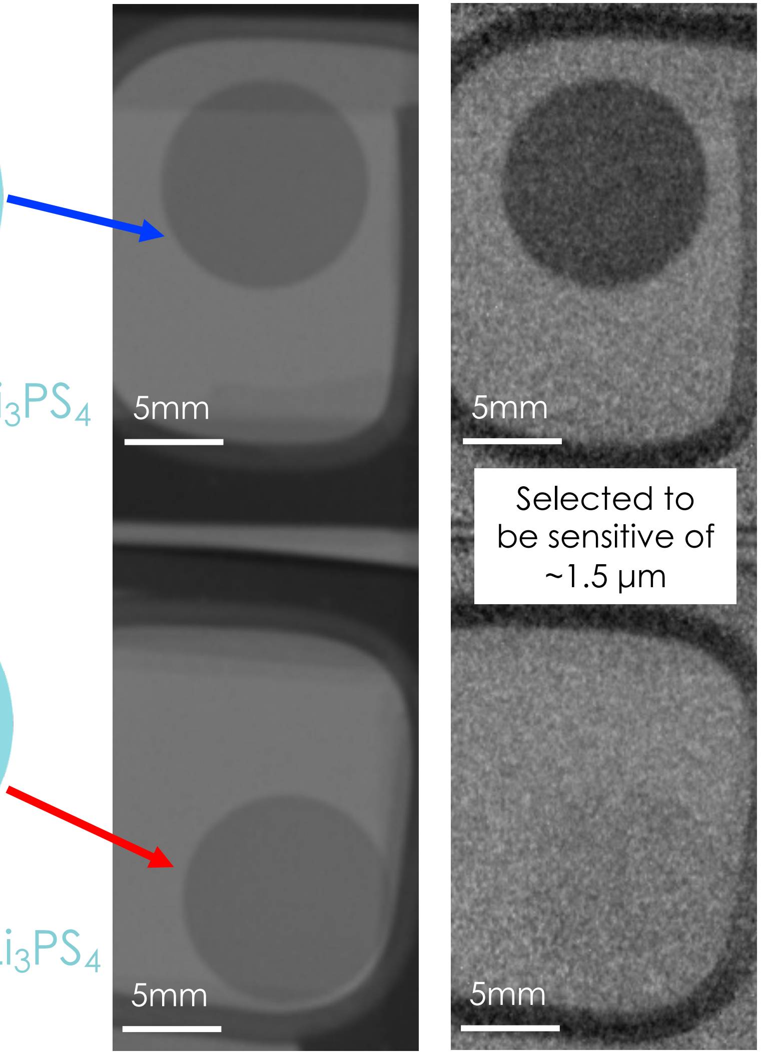
Neutron as **particle** | Neutron as **wave**



μSi (1-5 μm) in Li_3PS_4 30 wt.%



nSi (<50 nm) in Li_3PS_4 30 wt.%

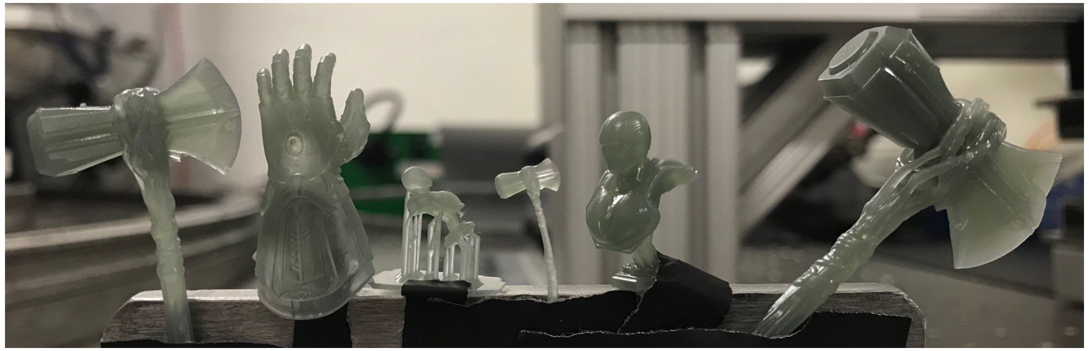


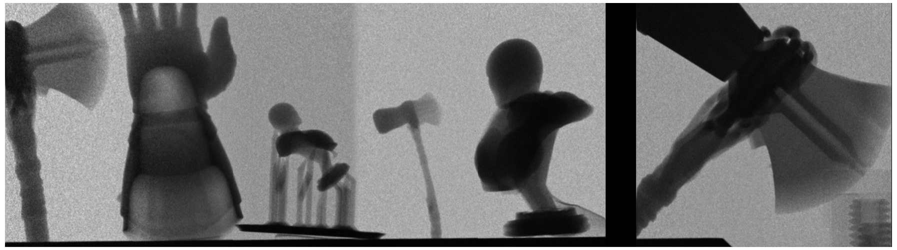
IPTS-26647, Y. Zhang, 2022

(Battery electrodes, courtesy of Fudong Han)

Neutron Imaging Capabilities at Steady-State (HFIR) Sources

- Radiography
- Tomography
- Grating Interferometry
- Polarized Neutron Imaging
- Monochromatic Imaging





(Sample credit: George Williams)

Type of detector	Field-of-view (FOV)	Pixel size (µm)	Highest spatial resolution (µm)	Typical acquisition time of 1 radiograph	Maximum speed @16 bit
High-res	50 x 49 mm^2	7.63	20-25	360 s	1 image/second
High-speed	88 x 88 mm^2	43	~100	—	74 image/second
Balanced	88 x 88 mm^2	16	~50	30-90 s	1 image/second

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Yuxuan

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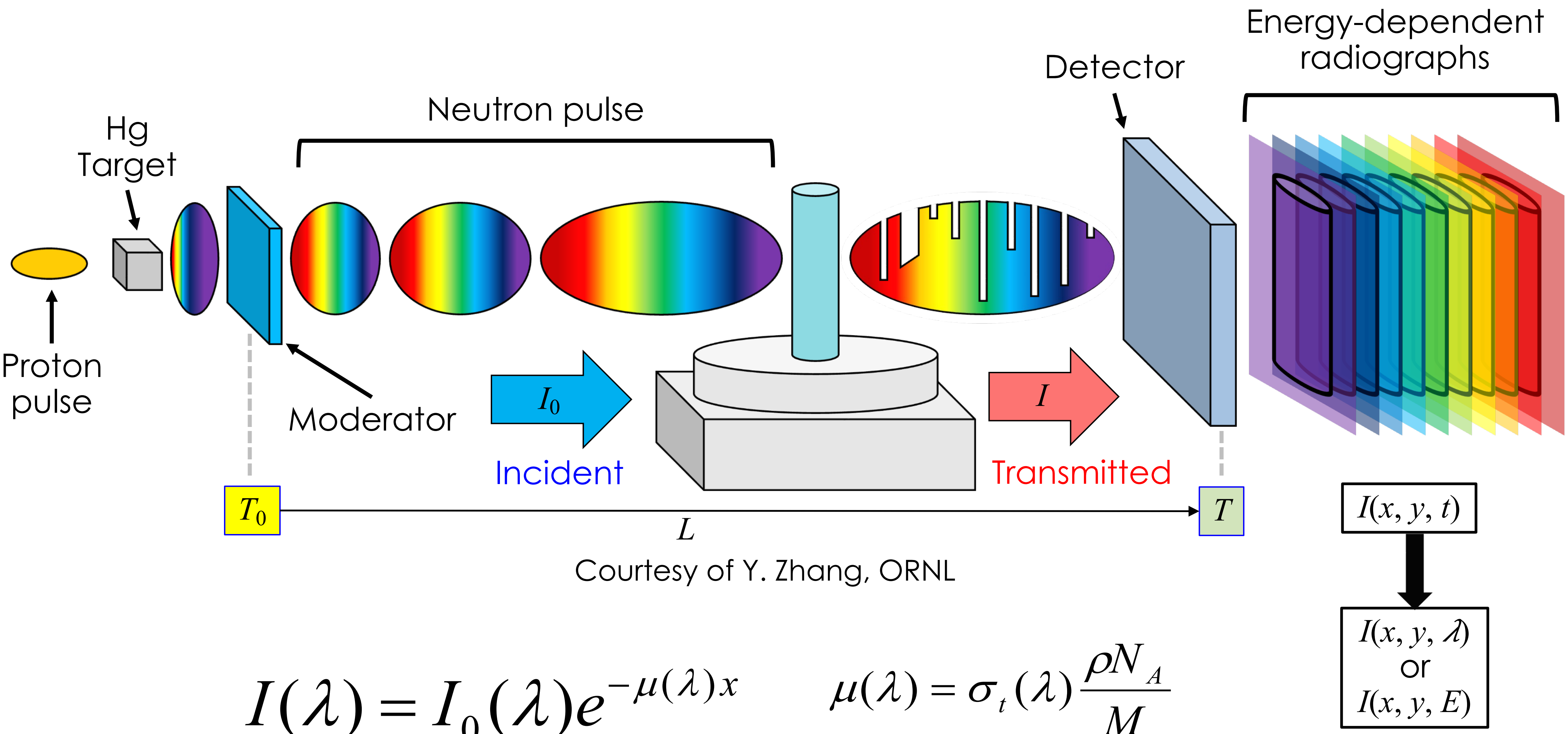
  - **Principle of neutron radiography at a pulsed source**
  - **Examples**
  - **The VENUS construction project**

Hassina

- Software tools for imaging

Jean

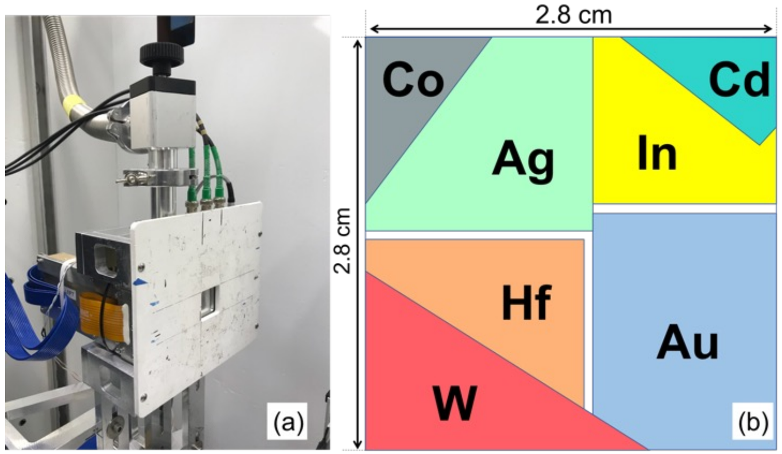
Imaging at a pulsed source (SNS)

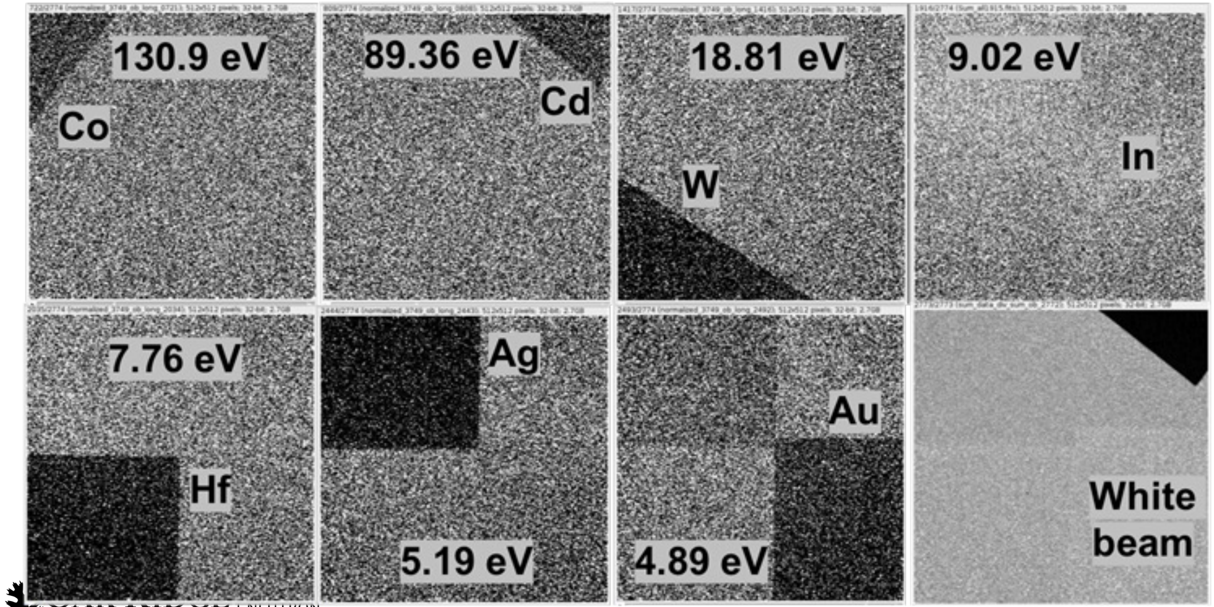


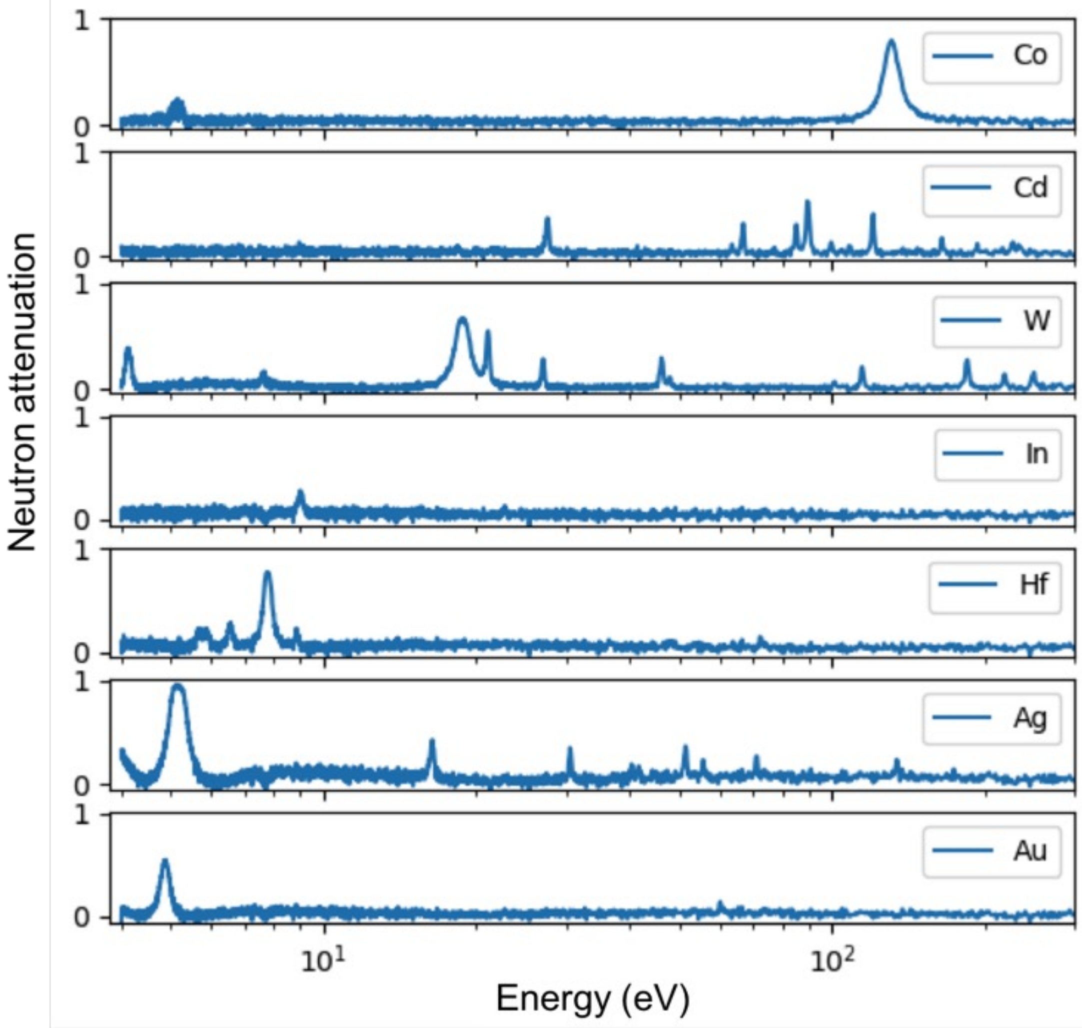
Courtesy of Y. Zhang, ORNL

$$I(\lambda) = I_0(\lambda) e^{-\mu(\lambda) x} \qquad \mu(\lambda) = \sigma_t(\lambda) \frac{\rho N_A}{M}$$

Higher energy neutrons can also be used for imaging (neutrons of energies higher than 1 eV): Resonance Imaging



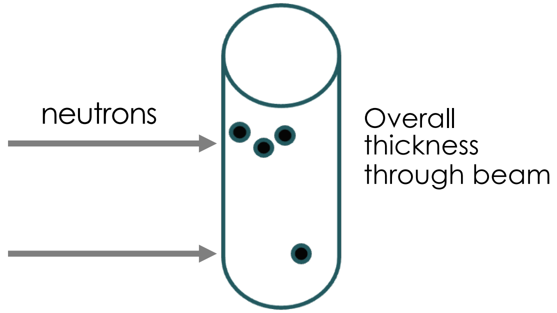




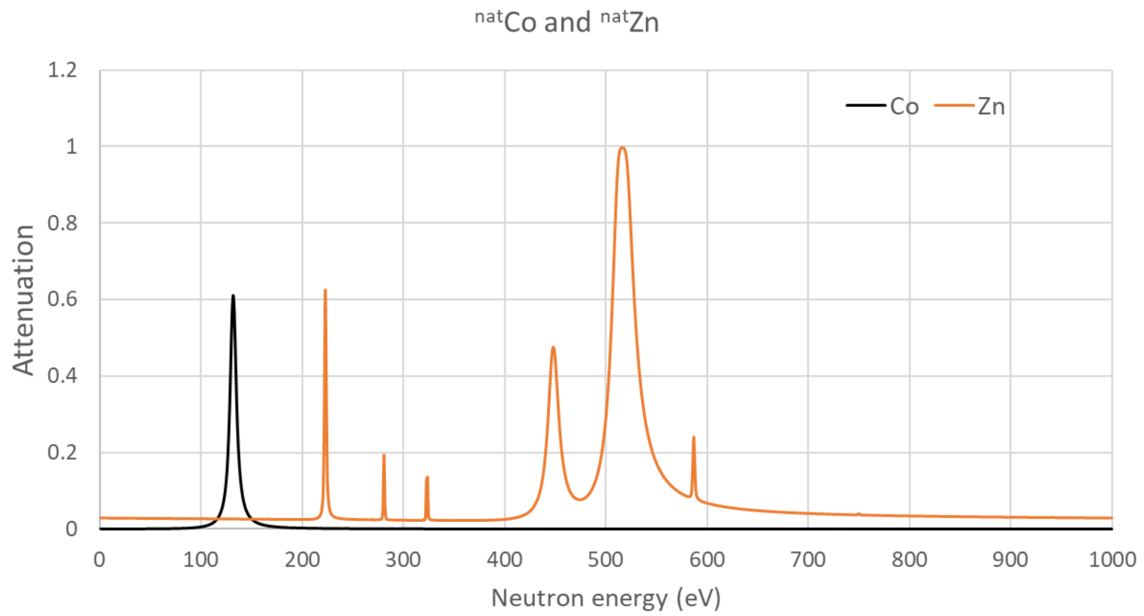
Zhang Y., Myhre K.G., Bilheux H.Z., Tremsin A.S., Johnson J.A., Bilheux J., Miskowiec A., Hunt R.D., Santodonato L., Molaison J.J., "Neutron Resonance Radiography and Application to Nuclear Fuel Materials", *Transactions of the American Nuclear Society*, (2018).

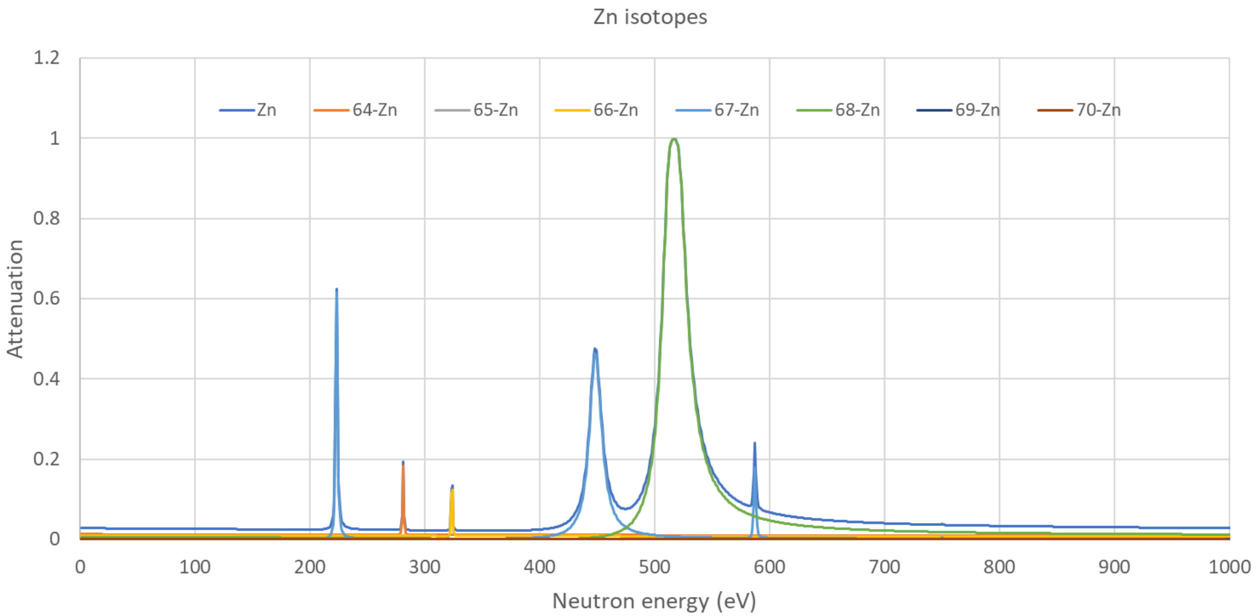
Resonance imaging: preparing your experiment

- Soil surveys, contaminants in soil, etc.:
  - transmission through 0.01 mm thickness of ^{nat}Co (between 1 and 5 Å) = 99.5 %
  - transmission through 1 mm thickness of ^{nat}Zn (between 1 and 5 Å) = 96.4 %



Simulated resonance for elements of interest(*)

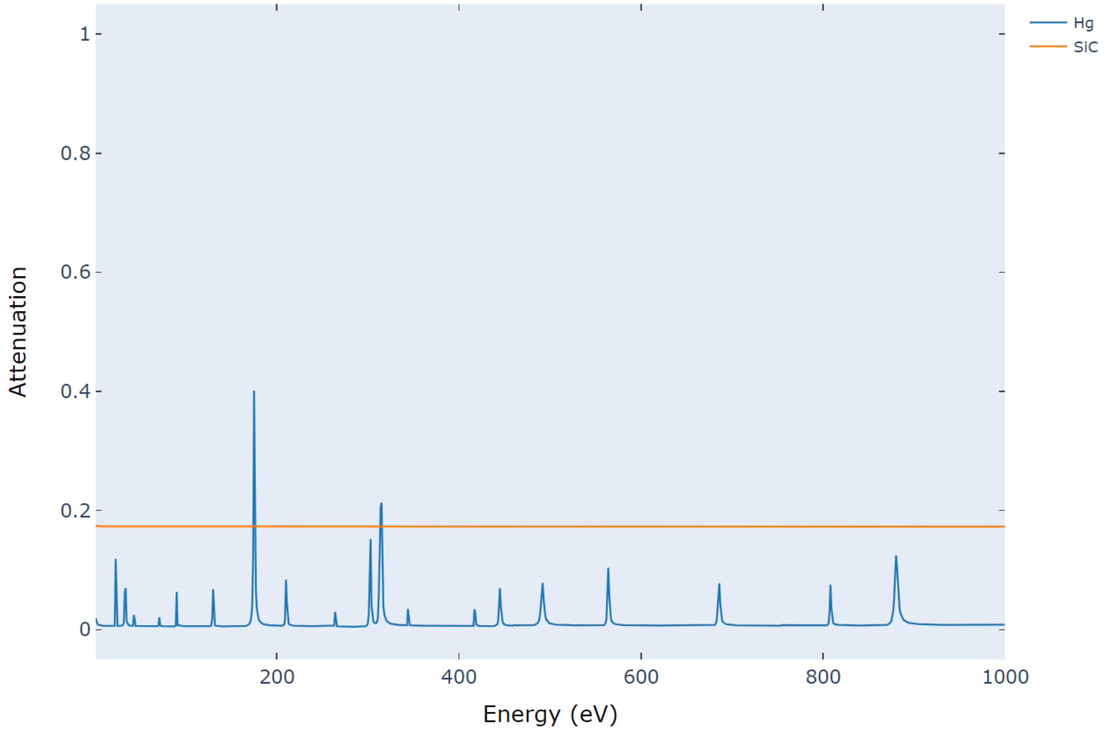




(*)Soil surveys in UK (1978-1983, 2000, 2011-2012)

Resonance imaging: preparing your experiment (cont'd)

- Hg contamination in soil
  - Assumptions: 0.1 mm Hg (13.6 g/cm^3) + 12.5 mm SiC (with 1.5 g/cm^3)
    - Transmission (1 and 5 Å) = 66.4 %



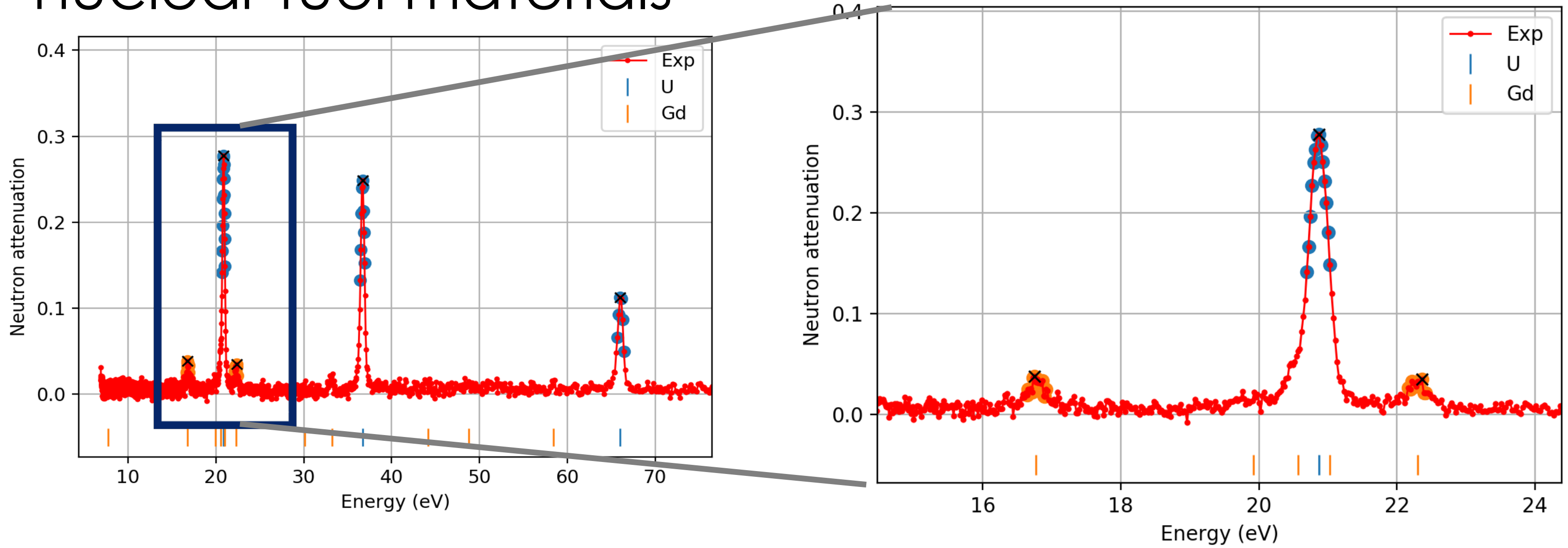
Resonance peaks plotted with:

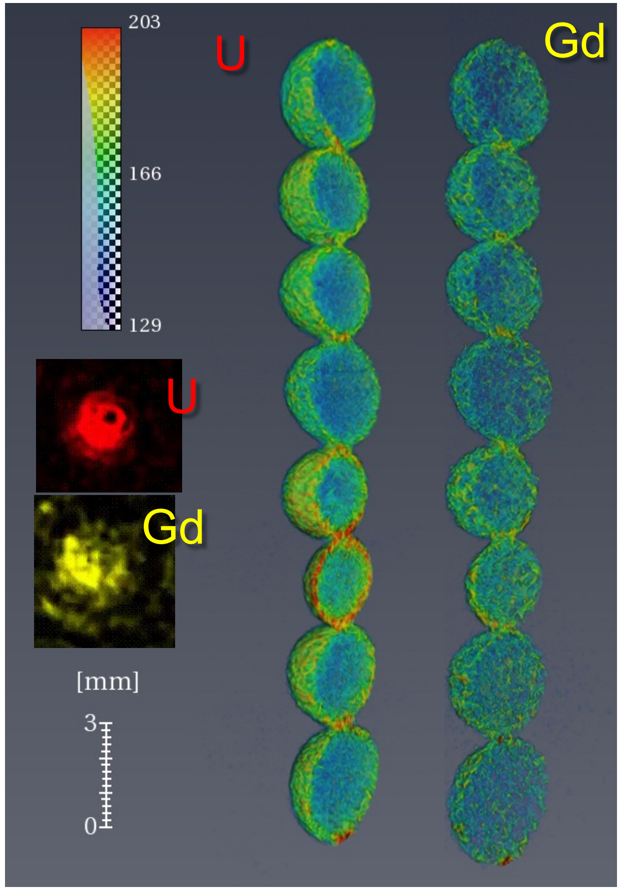
iNEUIT ("*I knew it*")

iNEUtron Imaging Toolbox

Using epithermal neutrons (energy > 1 eV), resonance imaging can map the isotopic content in advanced nuclear fuel materials in 3D

- Distribution of elements drive the performance of the novel advanced nuclear fuel materials





- **Quantitative analysis is being developed using in-house open source Python package (ResoFit)**

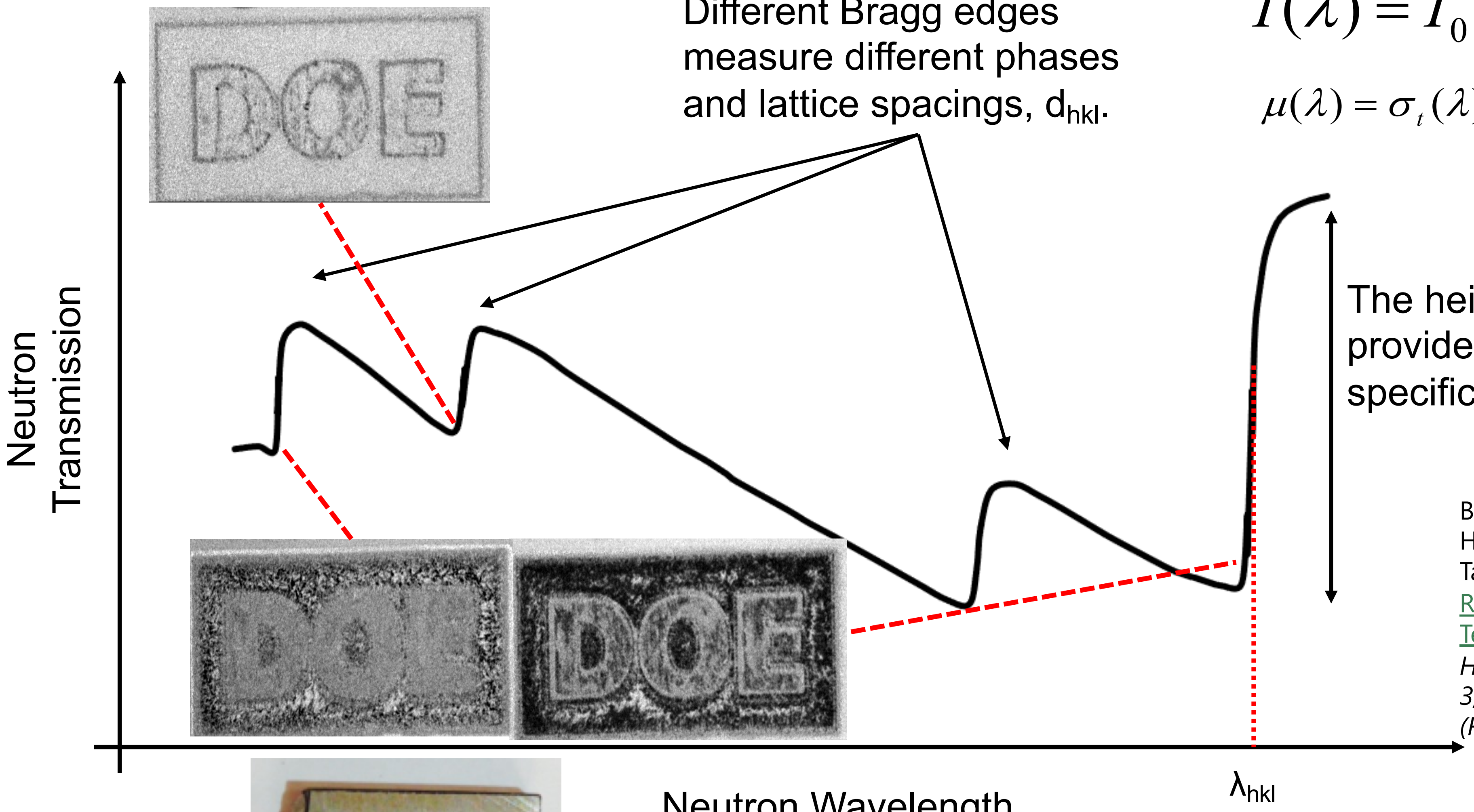
Myhre K.G., Zhang Y., Bilheux H.Z., Johnson J.A., Bilheux J., Miskowiec A., Hunt R.D., "Nondestructive Tomographic Mapping of Uranium and Gadolinium Using Energy-Resolved Neutron Imaging", *Transactions of the American Nuclear Society,* (2018).

Bragg edge imaging: how does it work?

$$I(\lambda) = I_0(\lambda)e^{-\mu(\lambda)x}$$

$$\mu(\lambda) = \sigma_t(\lambda)\frac{\rho N_A}{M}$$

Different Bragg edges measure different phases and lattice spacings, d_{hkl}.



The height of Bragg edge provides the *amount* of a specific phase.

Barton J.P., Bilheux H.Z., Bossi R., Herwig K.W., Santodonato L., Taylor M., "Chapter 12: Neutron Radiography for Nondestructive Testing", *Nondestructive Testing Handbook, Fourth Edition: Volume 3, Radiographic Testing (RT)* (2019).

3D printed Inconel 718

1 cm

The position of the Bragg edge, $\lambda_{hkl} = 2\,d_{hkl}$, is a measure of the *strain* in the sample

Principle of Bragg edge Transmission

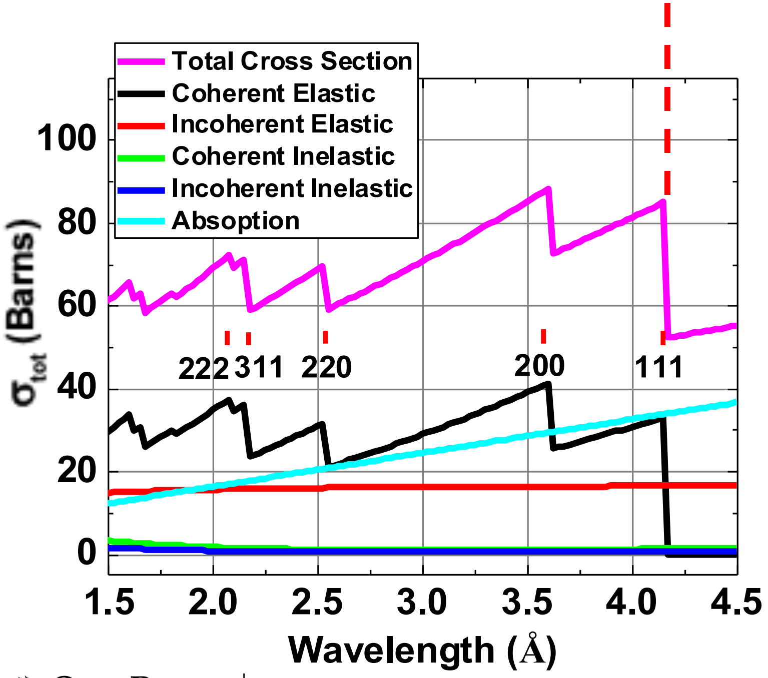
- ✓ Utilizes thermal and cold neutrons (approximately between 1 and 10 Å)
- ✓ Obeys Bragg's Law $\lambda_{hkl} = 2d_{hkl} \sin \theta_{hkl}$ simplifies: $\lambda_{hkl} = 2d_{hkl}$

March-Dollase model | Sabine's primary extinction model

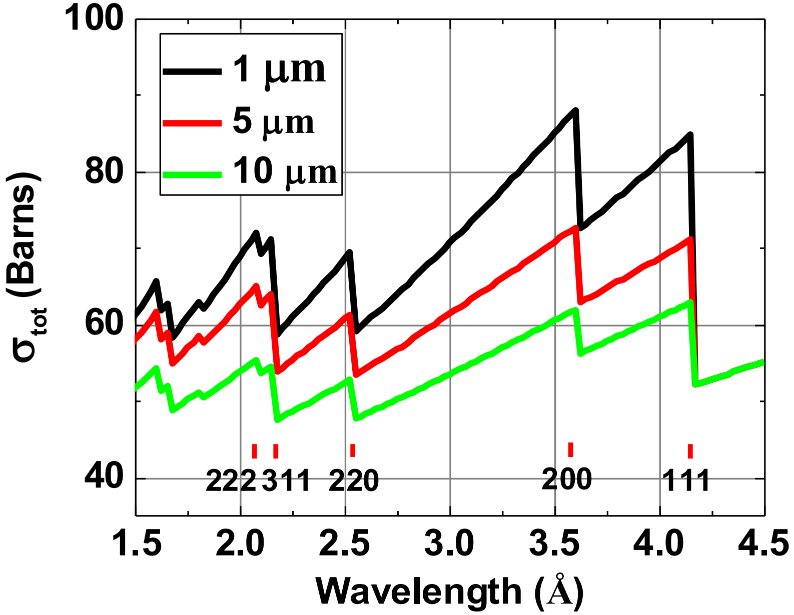
$$\sigma_{Bragg}(\lambda) = \frac{\lambda^2}{2V_0} \sum_{hkl}^{2d_{hkl}>\lambda} |F_{hkl}|^2 d_{hkl} \, P\left(\alpha_{\vec{h}}(\lambda)\right) E_{hkl}(\lambda, F_{hkl})$$

V_0: volume of unit cell
F_{hkl}: Structure factor including Debye-Waller factor

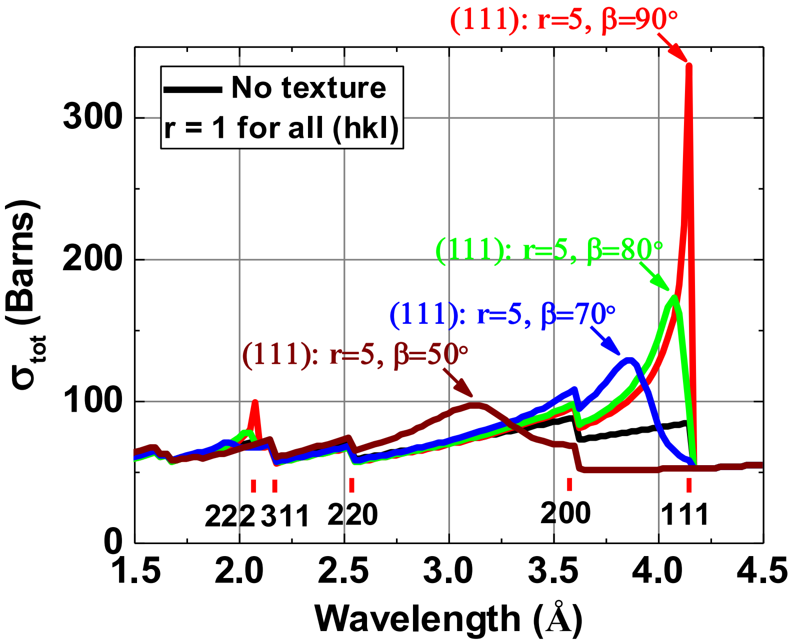
Total cross section for Inconel 718



Crystallite size effect (E_{hkl})

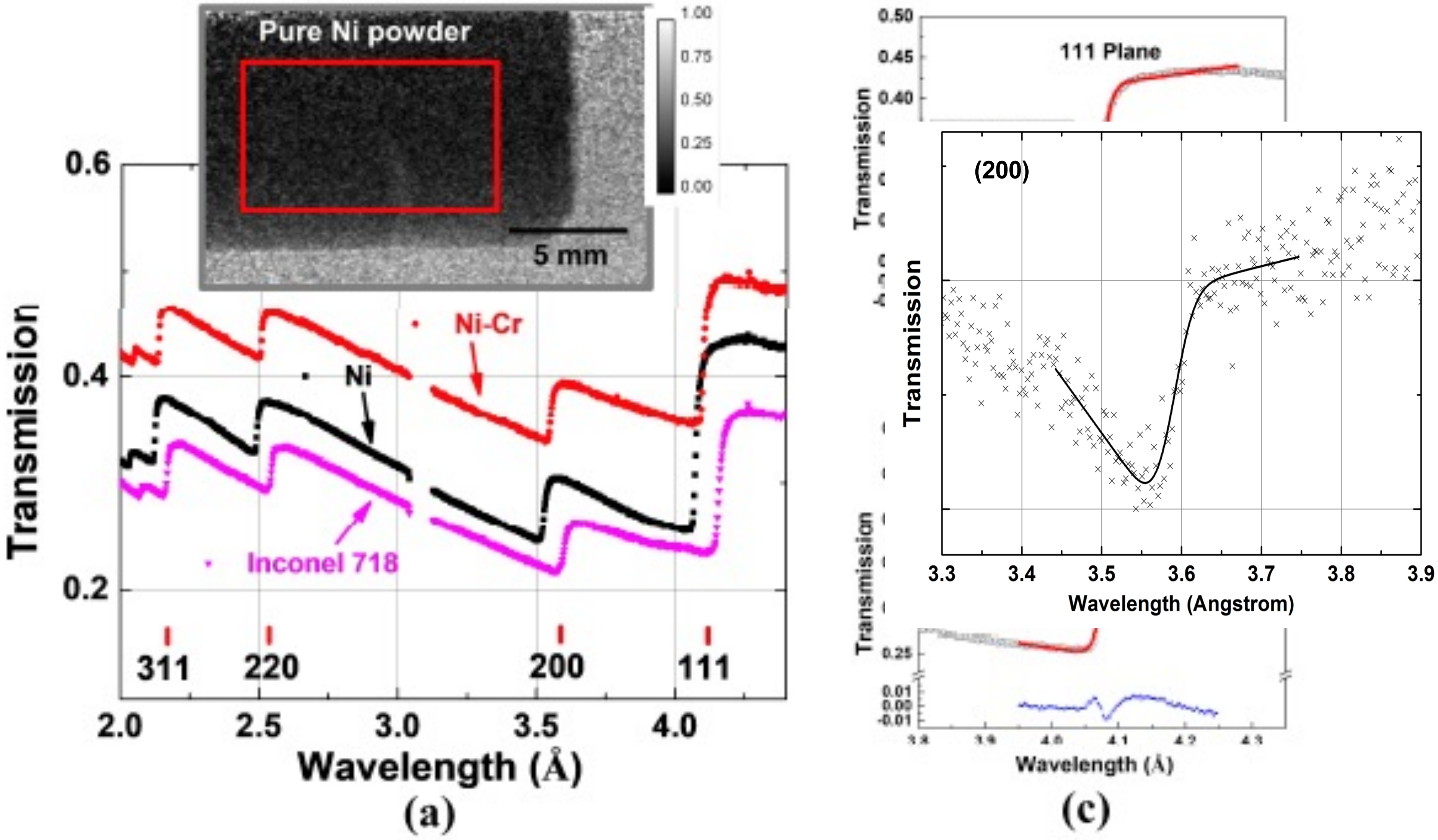


Crystallite orientation effect ($P\left(\alpha_{\vec{h}}(\lambda)\right)$: r and β)



G. Song et al., Journal of Imaging, 3, 65 (2017).

The perfect case study: powders



Song G., Lin J.Y., Bilheux J., Xie Q., Santodonato L., Molaison J.J., Skorpenske H.D., dos Santos A.M., Tulk C.A., An K., Stoica A.D., Kirka M.M., Dehoff R.R., Tremsin A.S., Bunn J.R., Sochalski-Kolbus L.M., Bilheux H.Z., "Characterization of Crystallographic Structures Using Bragg-Edge Neutron Imaging at the Spallation Neutron Source", *Journal of Imaging*, **3,** 4, 65 (2017).

Materials Behavior: Monitoring residual strain relaxation and preferred grain orientation of additively manufactured Inconel 625 by in-situ neutron imaging

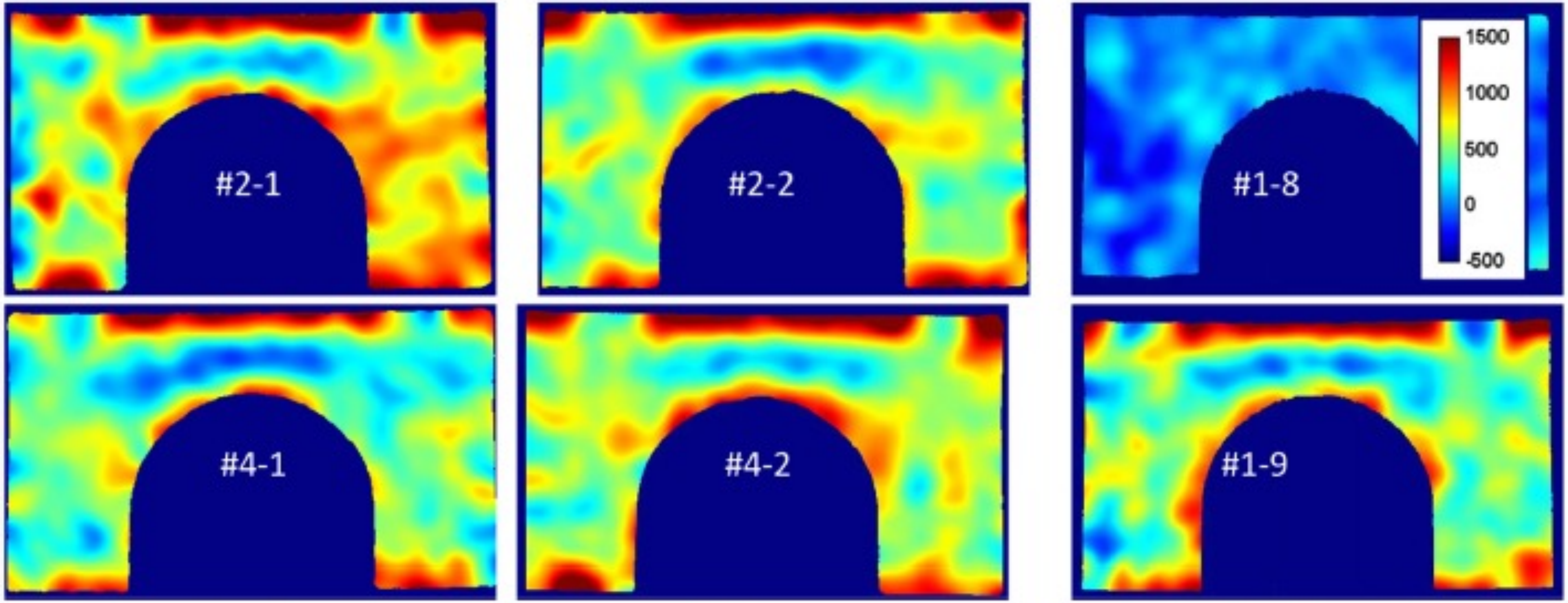
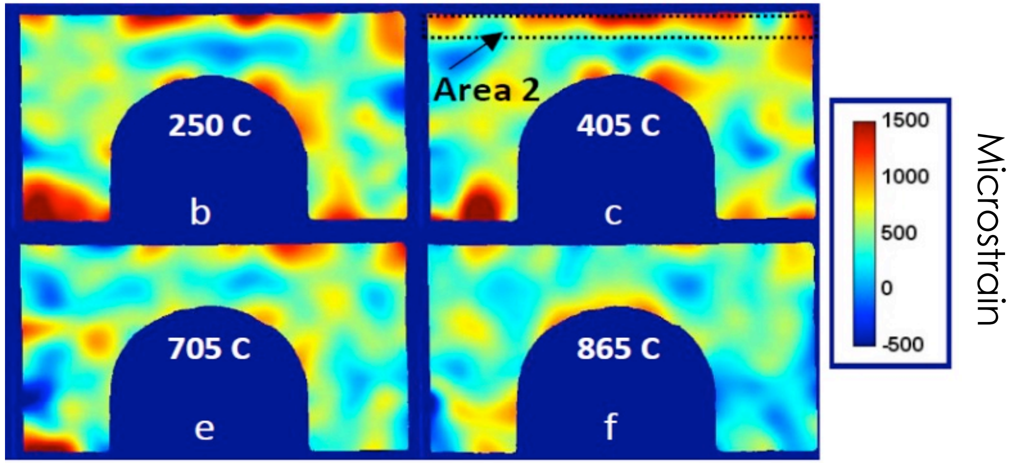
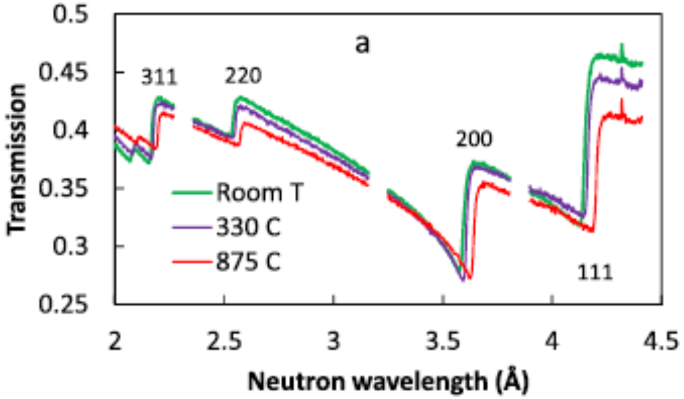


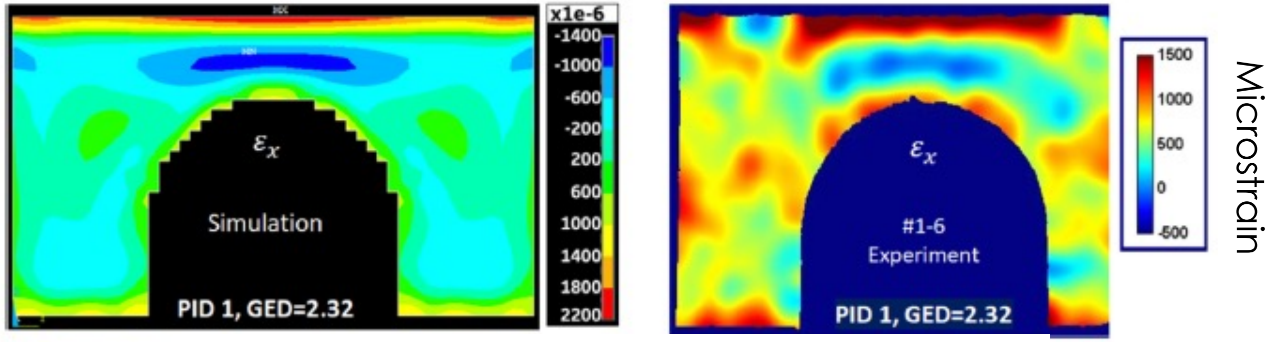
Fig. 8. Strain distribution (in microstrain) at the (111) Bragg edge measured at room temperature along the sample thickness direction X. SNAP beamline. The image integration time was about 2 h at SNAP. λ_0 value is taken from the annealed sample #1–8 (average across the entire sample). The legend indicates the strain values in microstrain.

Engineered Materials: Monitoring residual strain relaxation and preferred grain orientation of additively manufactured Inconel 625 by in-situ neutron imaging (10 min measurements)



AM Inconel 625 strain evolution as a function of temperature





Modeled and experimental results.

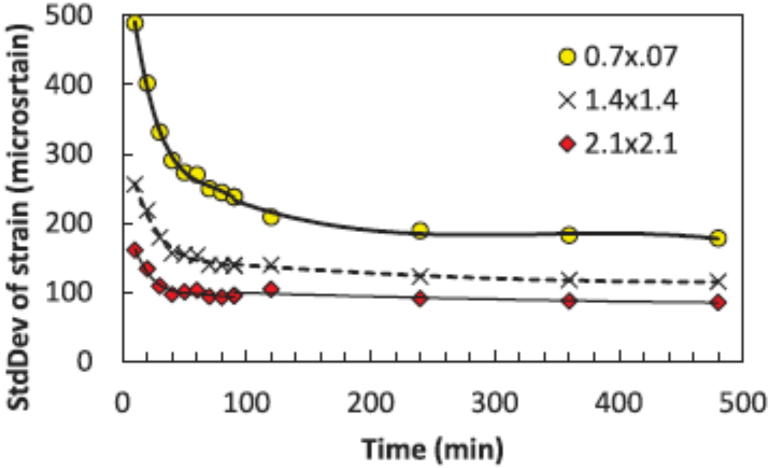
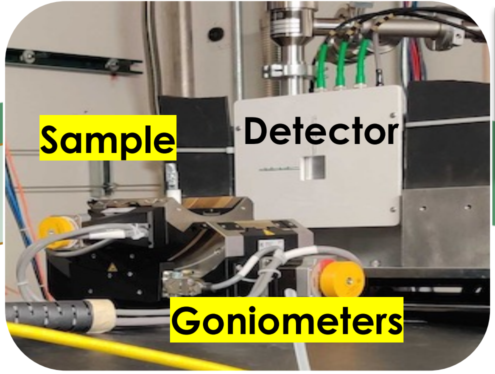


Fig. 16. Variation of the standard deviation of the reconstructed strain as a function of image integration time. Three different sizes of the area used for pixel grouping are used for strain reconstruction, as indicated by the legend (in mm²).

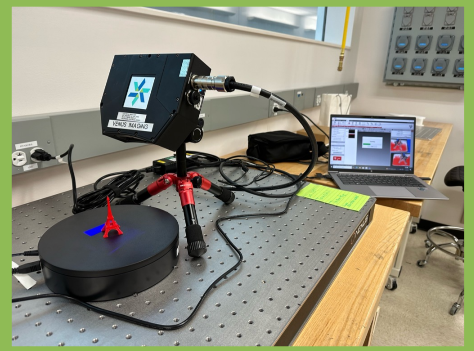
Tremsin et al, Additive Manufacturing, 2021.

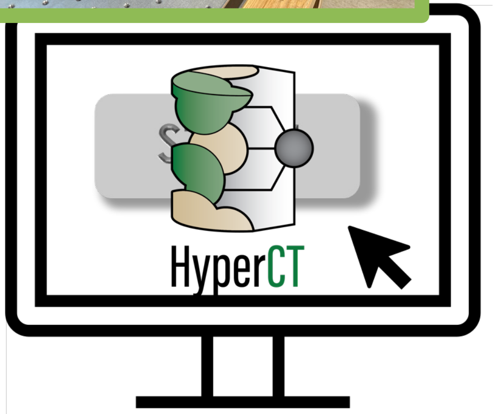
Tremsin et al, Nuc. Instr. Methods in Phys. Res. A, 2021.

Autonomous Hyperspectral Neutron CT Experiment at ORNL



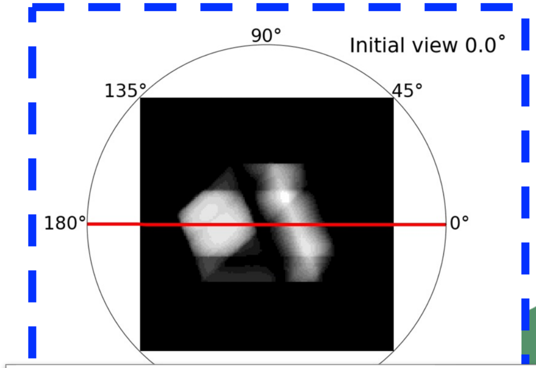
Light scan and preselection of projection angles



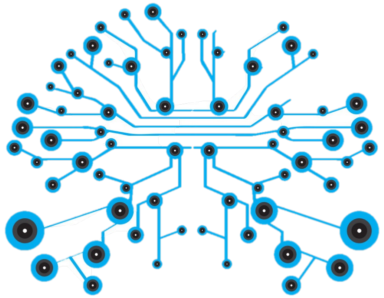


Up to factor 5 improvement in time

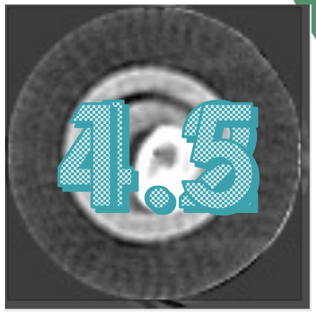
- Optimization of the scan based on the unique sample geometry
- Ability to provide real-time reconstructed data using advanced iterative reconstruction methods



Sample Adaptive Scanning Angles (active learning)



AI Quality Evaluation



Autonomous Decision

Stop

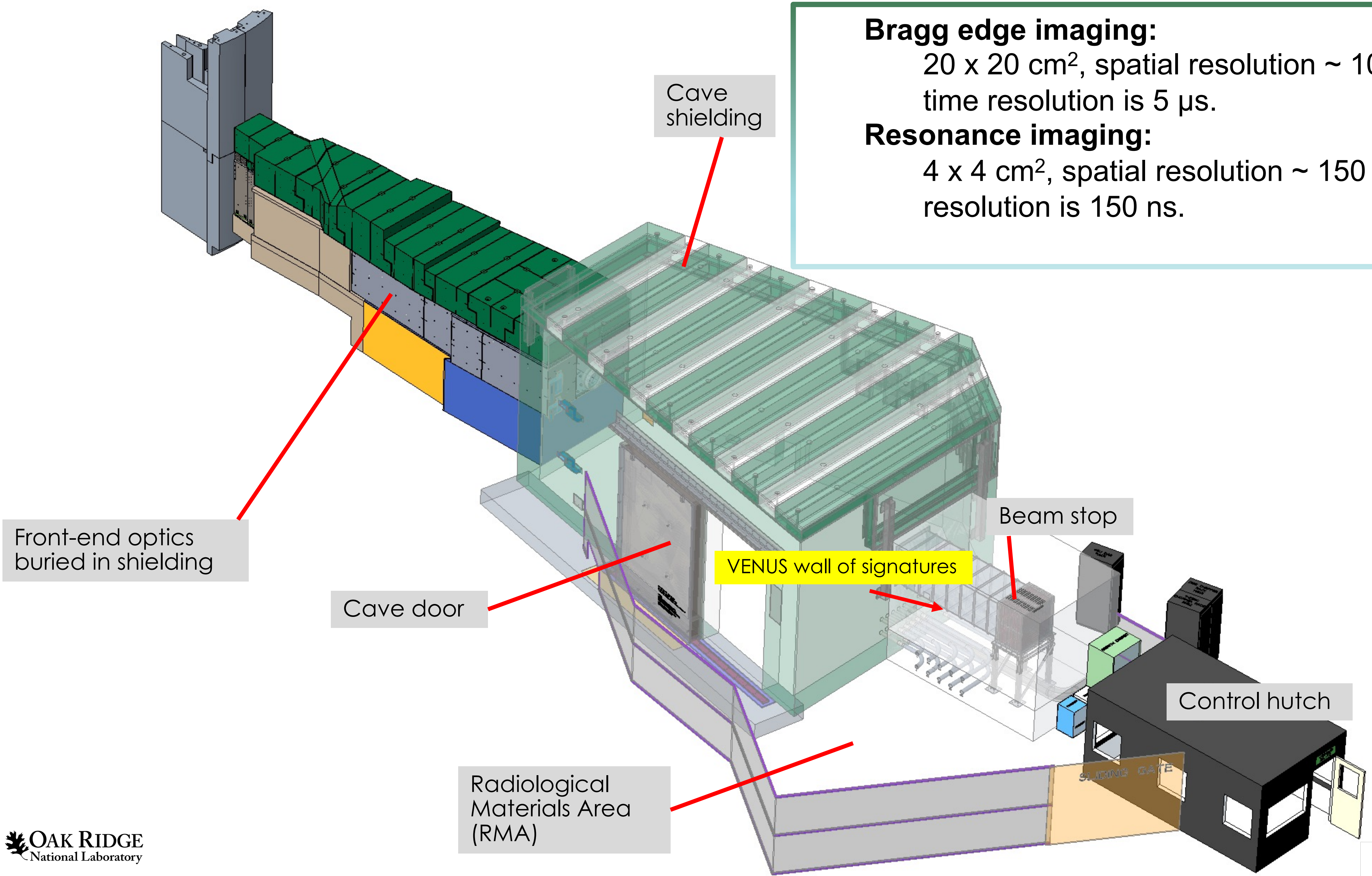
Continue

Bragg edge imaging:

20 x 20 cm^2, spatial resolution ~ 100 μm, time resolution is 5 μs.

Resonance imaging:

4 x 4 cm^2, spatial resolution ~ 150 μm, time resolution is 150 ns.



February 2020 to now

Outline

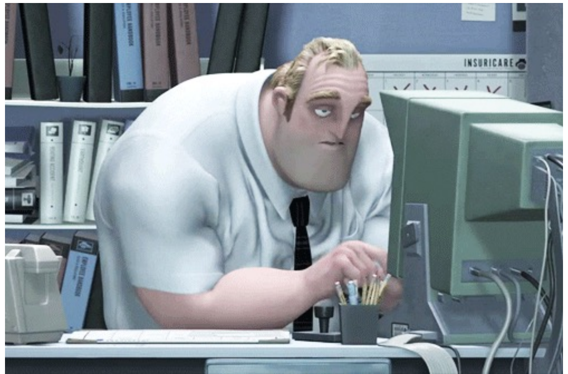
- Imaging at the High Flux Isotope Reactor MARS beamline:
  - Principle of neutron radiography and computed tomography at a continuous source
  - The CG-1D imaging beamline
  - Examples

Yuxuan

- Imaging at the Spallation Neutron Source:
  - Principle of neutron radiography at a pulsed source
  - Examples
  - The VENUS construction project

Hassina

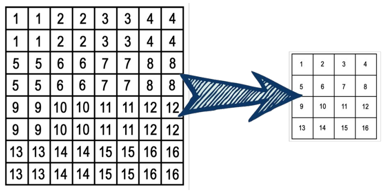
- **Software tools for imaging**



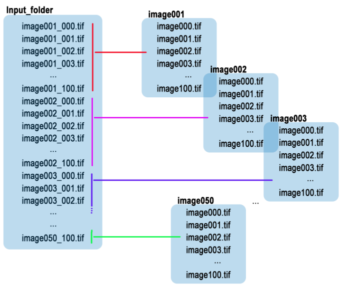
Jean

Simple tasks that could be showstoppers because of the amount of files

- Renaming thousand of files (keep/don't keep part of the initial file name)
- Binning pixels



- Combining images (tiff, fits) 2 by 2, 3 by 3, etc. using different algorithms
- Combining folders

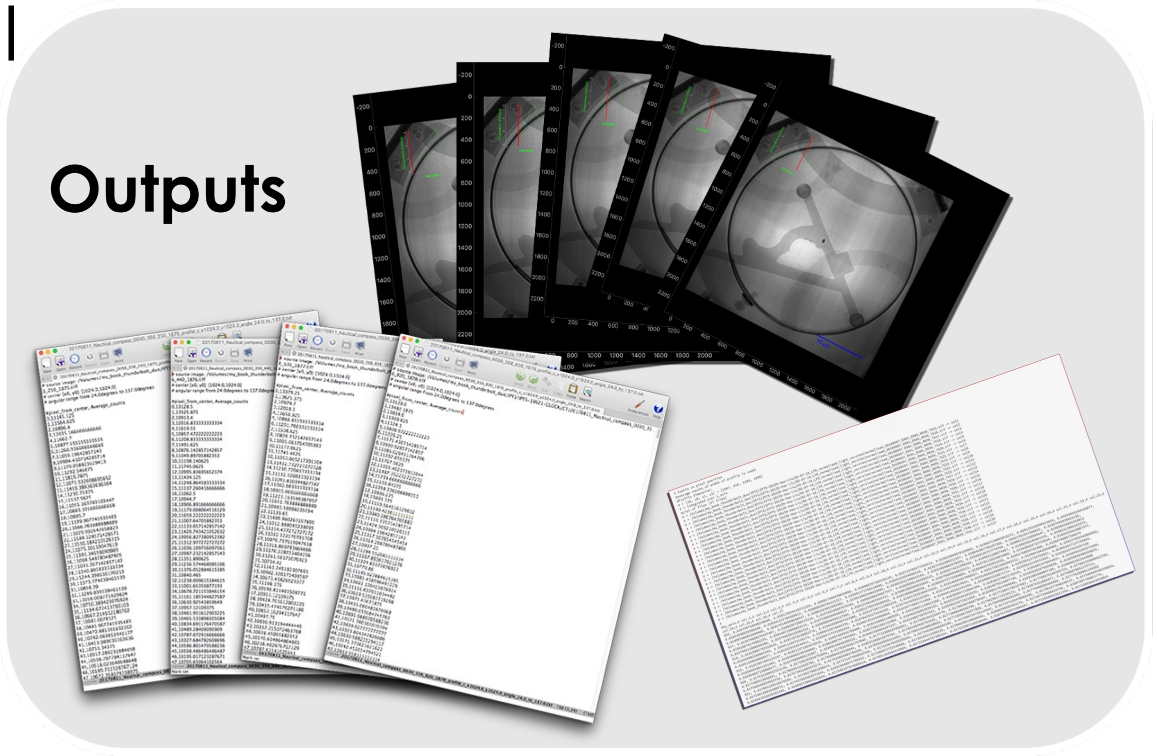


- Dealing images
- Extract evenly spaced files

Imagine yourself as a user who does not program, how would you do those?

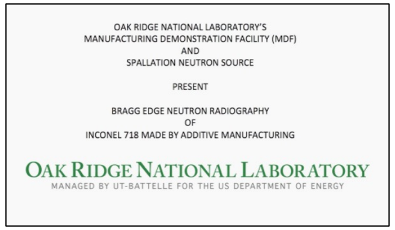
More complicated tasks

- Create a list of file name vs time stamp
- Display and export images with metadata/time stamp
- Display the change of a given metadata over time on top of images
- Gamma filtering optimization tool
- Profiles
  - Linear
  - Radial
- Calibrate transmission
- Normalization



Advanced tasks

Bragg Edge

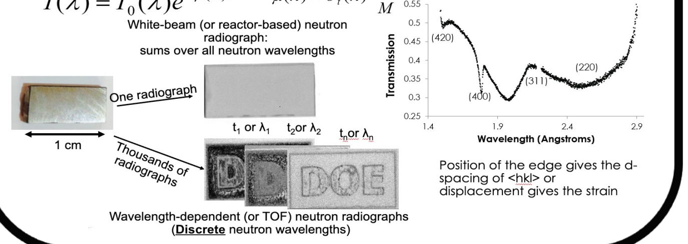

OAK RIDGE NATIONAL LABORATORY'S
MANUFACTURING DEMONSTRATION FACILITY (MDF)
AND
SPALLATION NEUTRON SOURCE

PRESENT

BRAGG EDGE NEUTRON RADIOGRAPHY
OF
INCONEL 718 MADE BY ADDITIVE MANUFACTURING

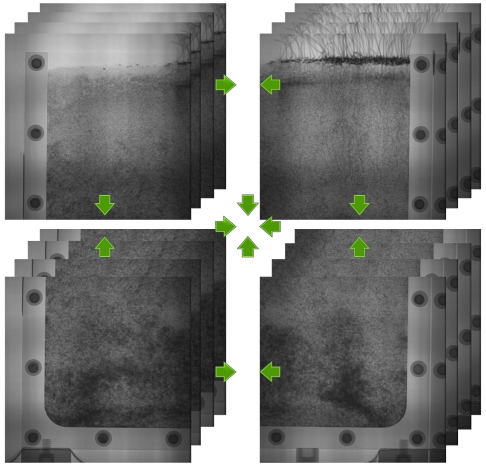
OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

$$I(\lambda) = I_0(\lambda)e^{-\mu(\lambda)x} \qquad \mu(\lambda) = \sigma_t(\lambda)\frac{\rho N_A}{M}$$



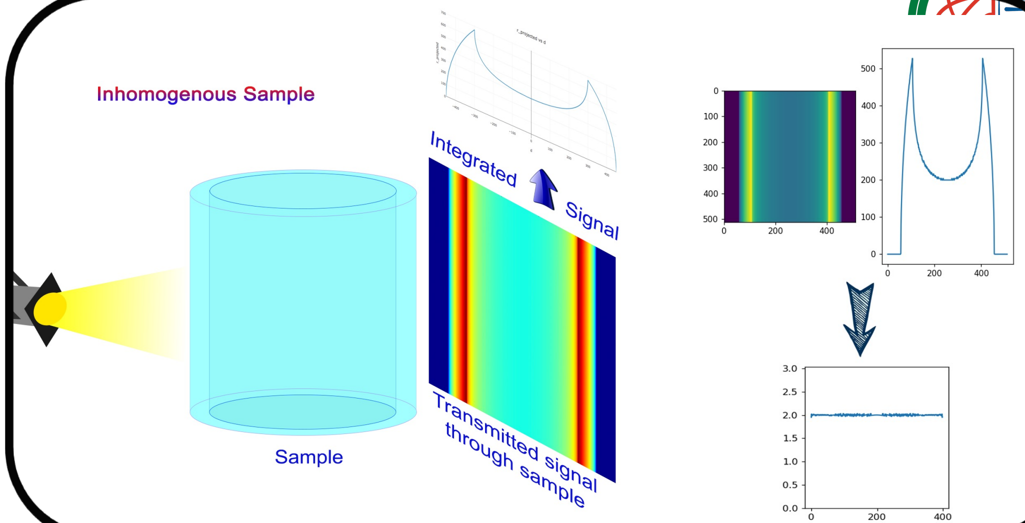
Position of the edge gives the d-spacing of <hkl> or displacement gives the strain

Panoramic Stitching

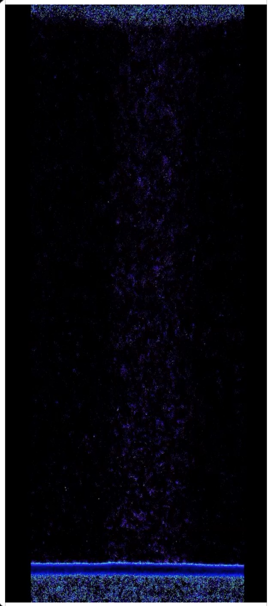


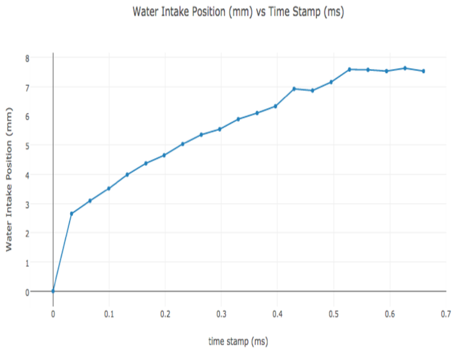
Impact of plant roots and mycorrhizal fungal hyphae on soil water retention parameters. Ed Perfect et al.

Cylindrical Geometry Correction



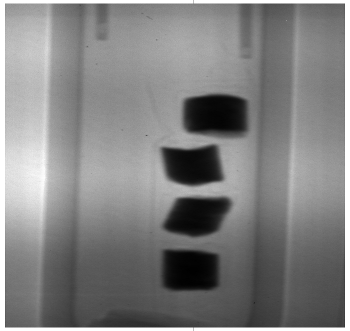
Water-intake Profile Calculator





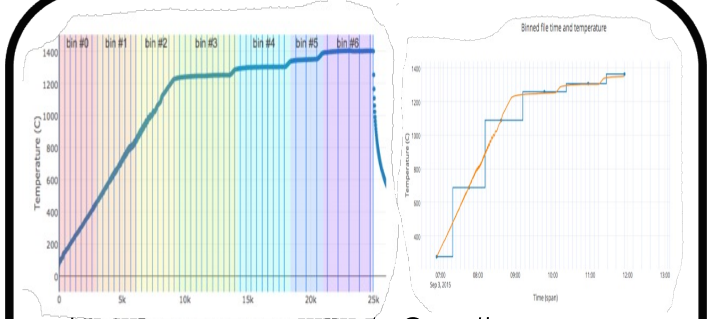
Acoustic facturing in shale samples. Richard Hale et al.

Registration



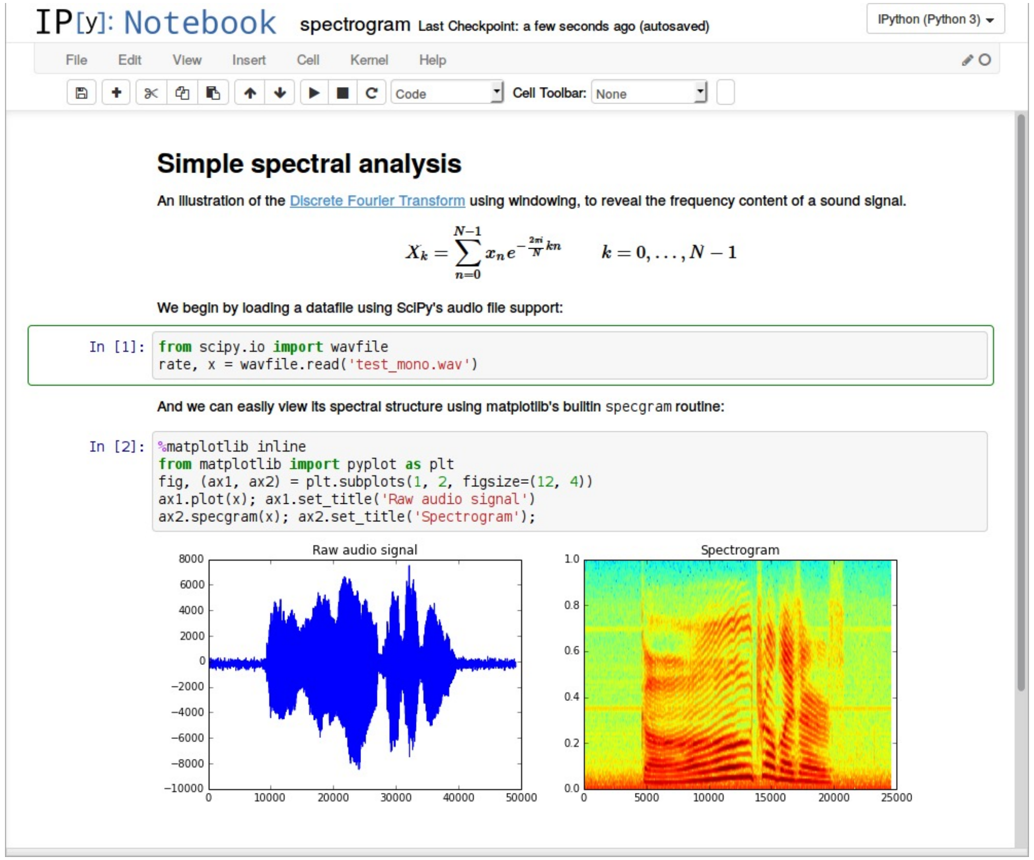
Characterization of different stages of hydrogen loss in biomass under pyrolysis by radiography and tomography. Frederik Ossler et al.

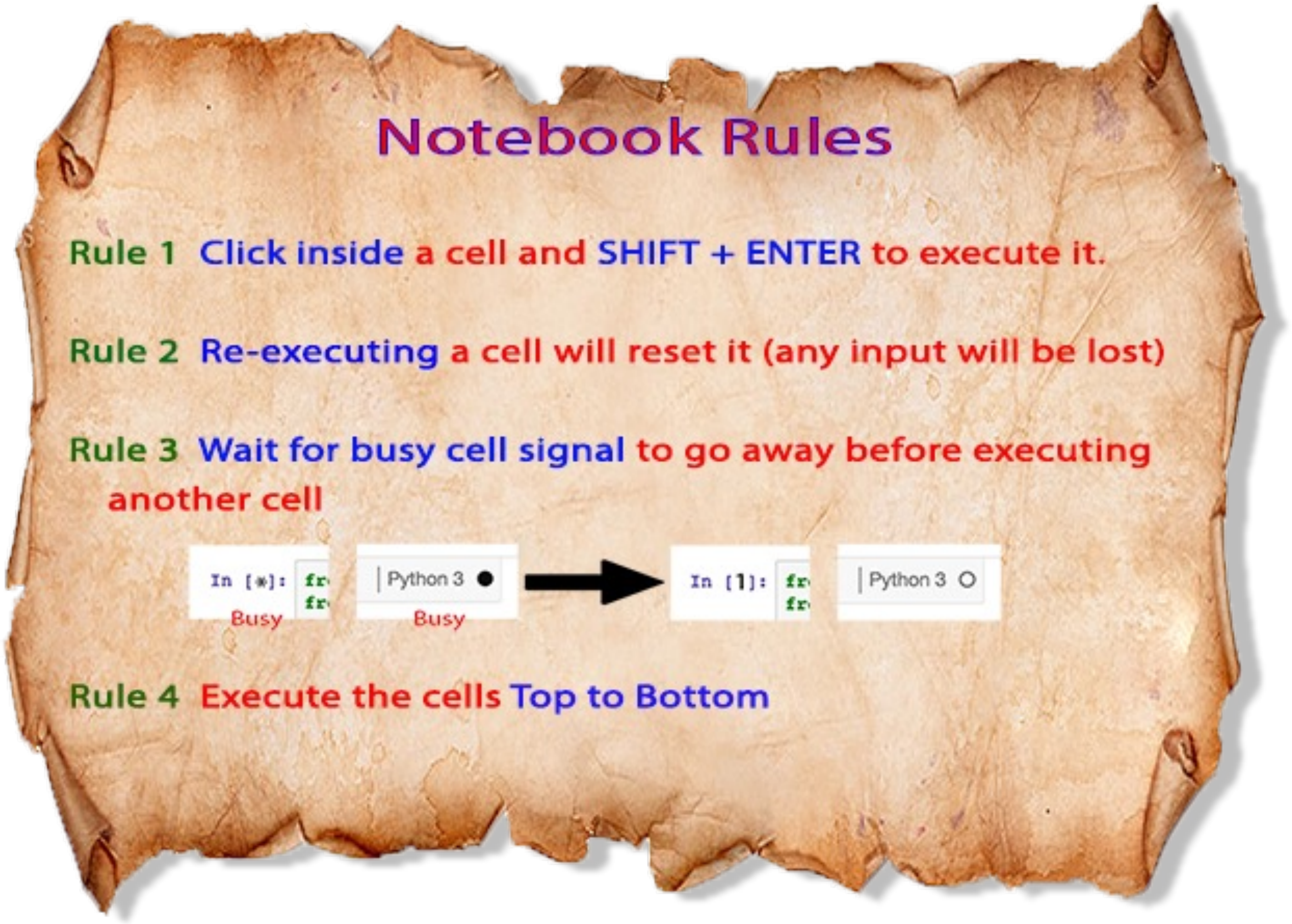
Images and Metadata Extrapolation Matcher



High Temperature Grain Growth Characterization of FeCrAl alloys by Time of Flight Neutron Radiography. Sebastien Dryepondt et al.

Solution: Jupyter notebooks

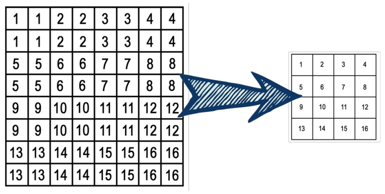




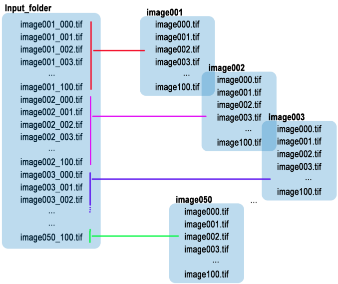
Simple tasks that could be showstoppers

Simple tasks for the notebooks

- Renaming thousand of files (keeping or not part of the initial file name)
- Binning pixels



- Combining images (tiff, fits) 2 by 2, 3 by 3, etc. using different algorithms
- Combining folders

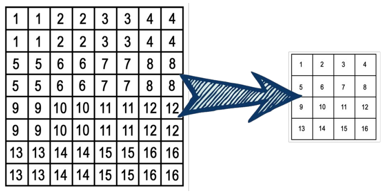


- Dealing images
- Extract evenly spaced files

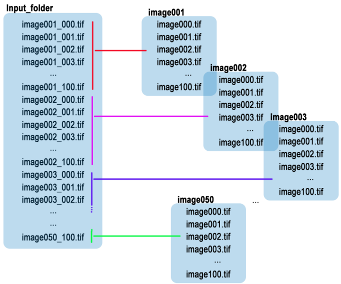
Imaging yourself as a user who does not program, how would you do those?

Simple tasks for the notebooks

- Renaming thousand of files (keeping or not part of the initial file name)
- Binning pixels

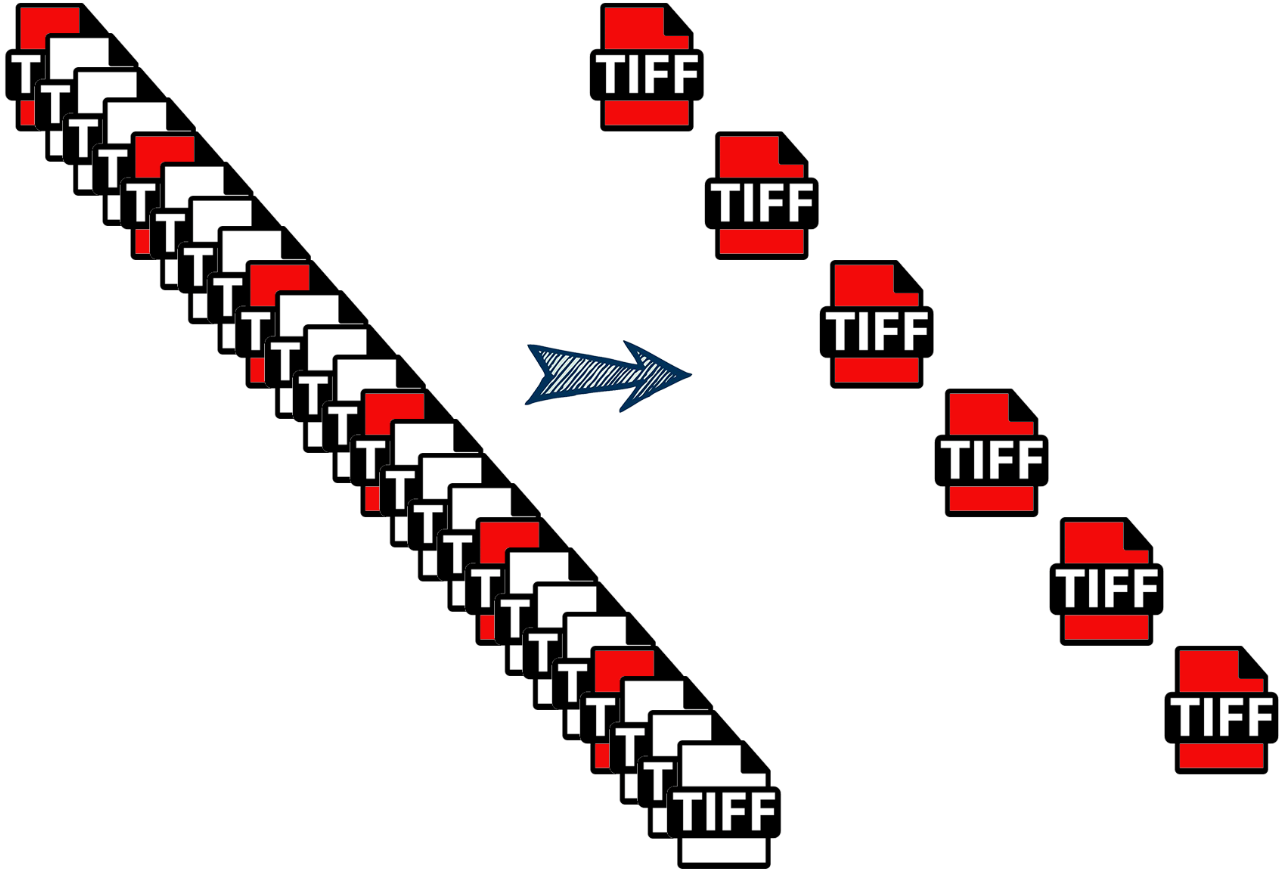


- Combining images (tiff, fits) 2 by 2, 3 by 3, etc. using different algorithms
- Combining folders



- Dealing images
- **Extract evenly spaced files**

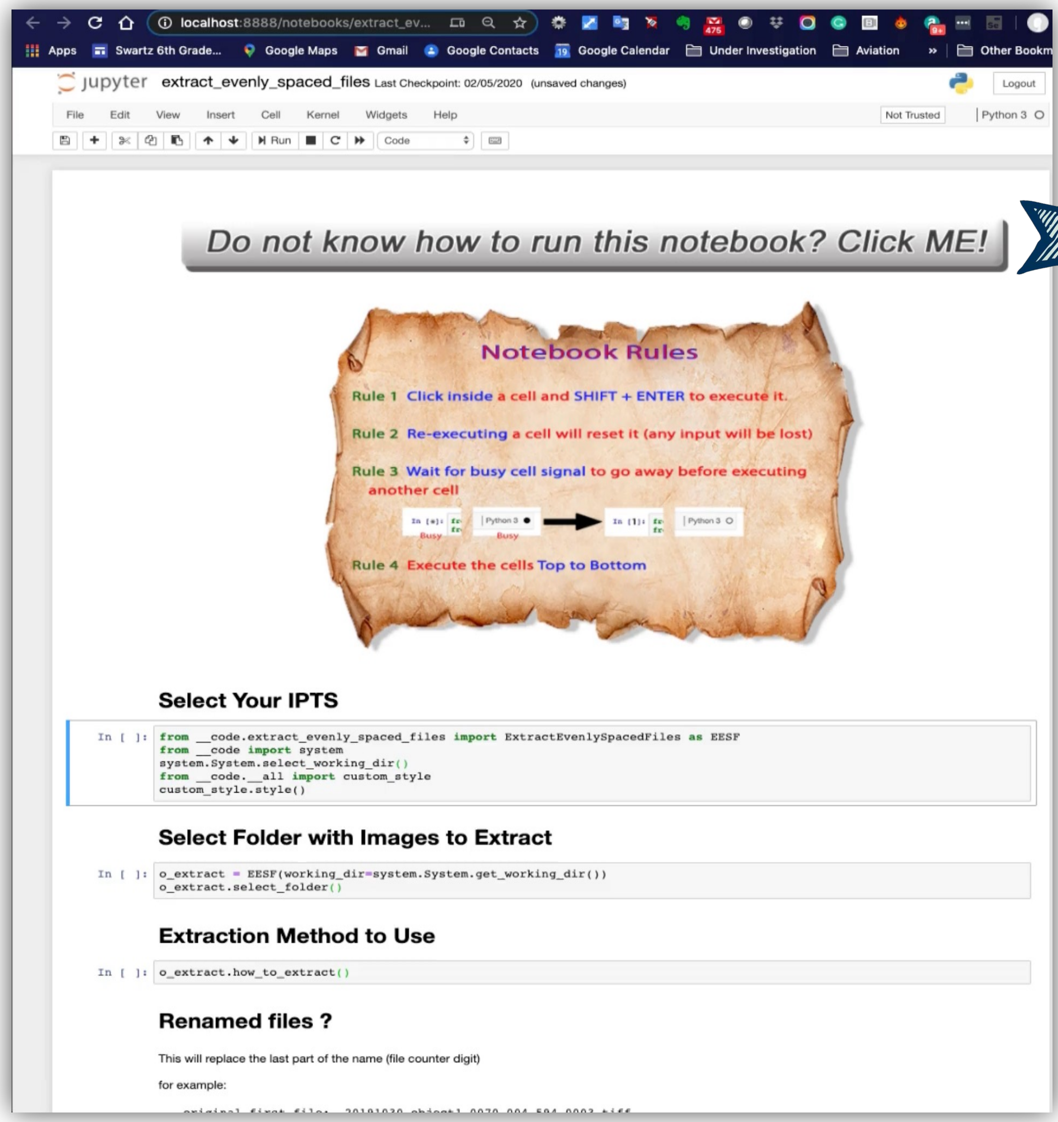
Simple tasks for the notebooks

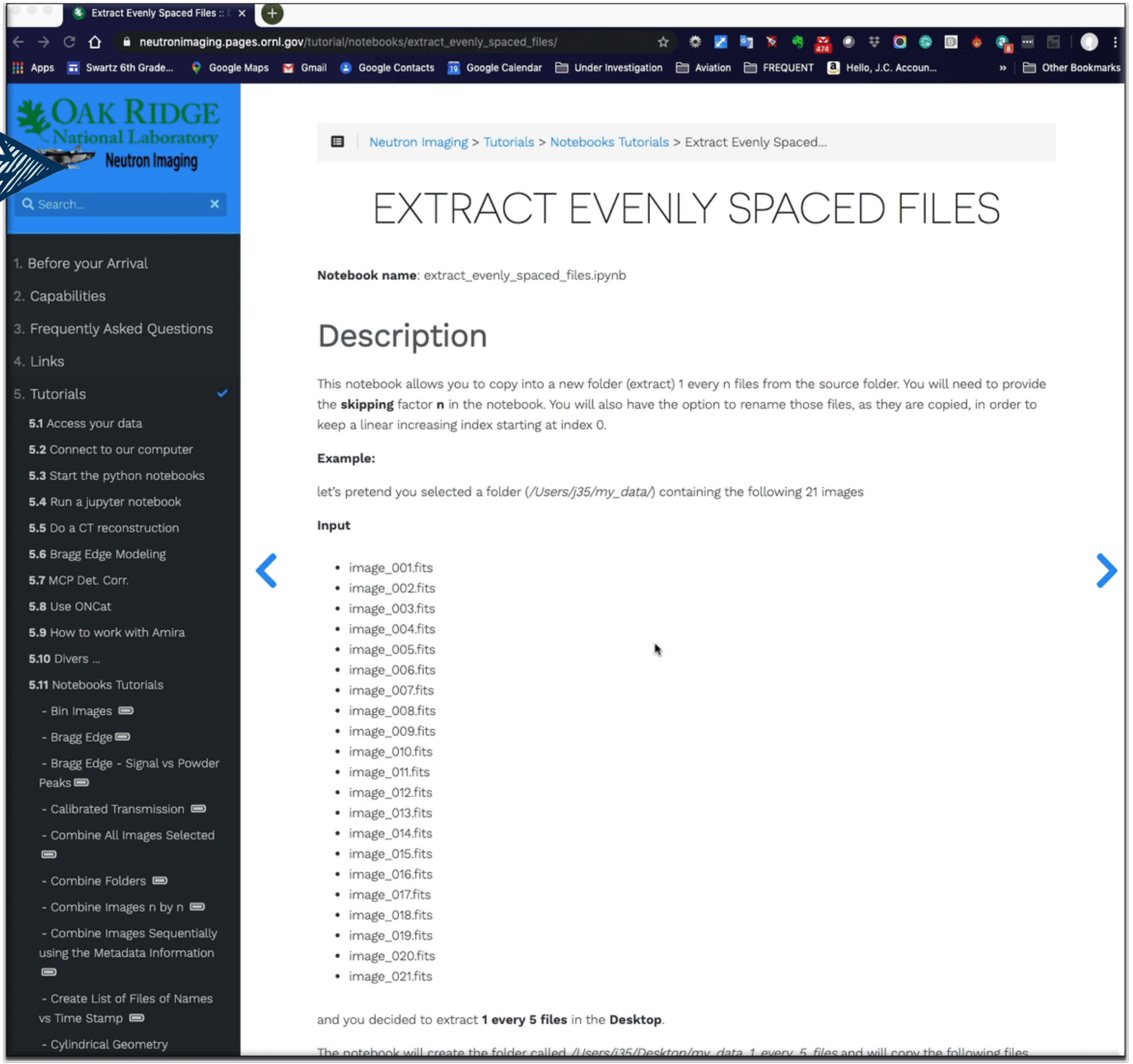


Extract from list of files every n spaced files

Simple tasks for the notebooks

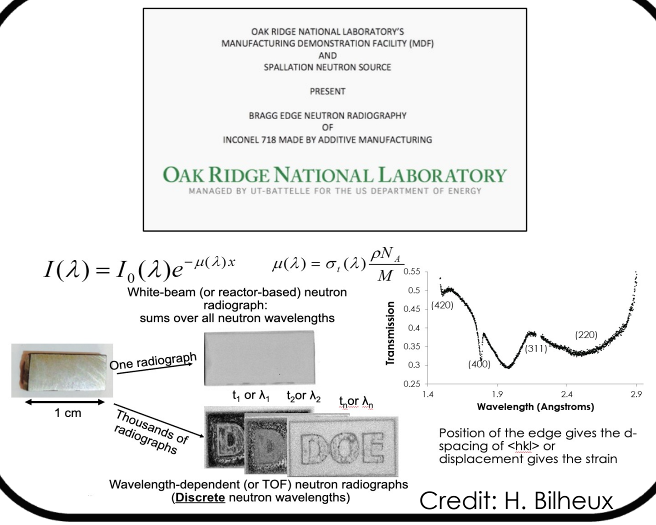
Extract evenly spaced files





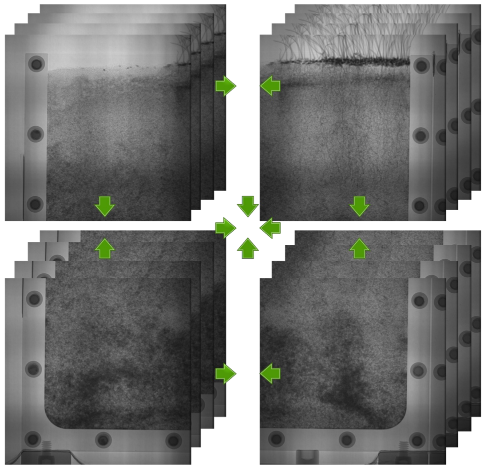
Advanced tasks

Bragg Edge



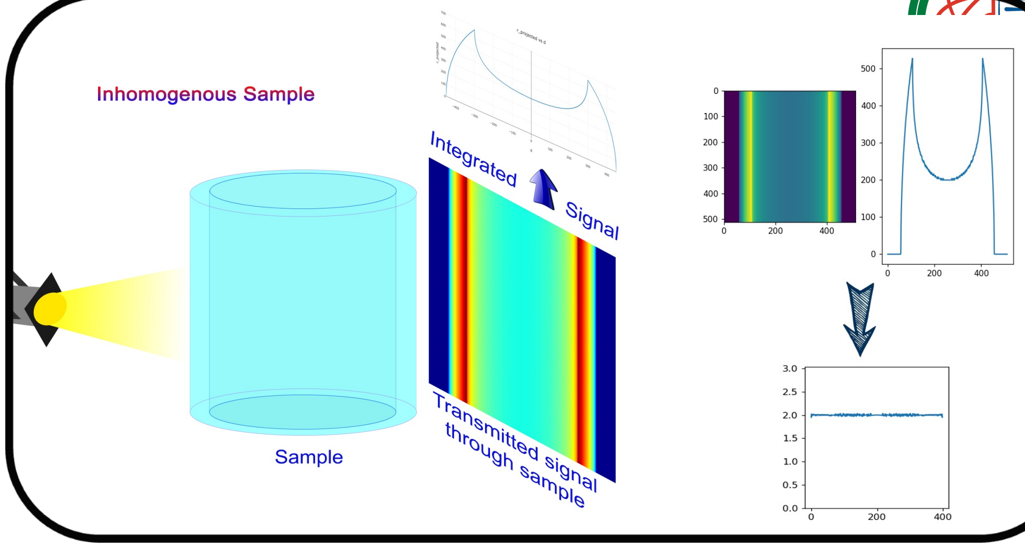
Credit: H. Bilheux

Panoramic Stitching

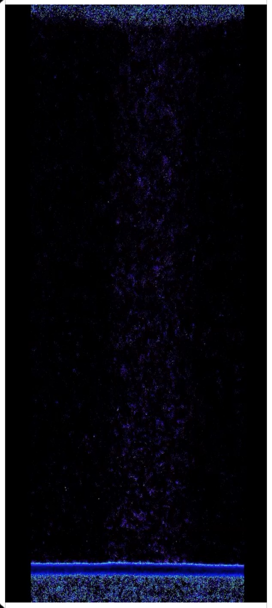


Impact of plant roots and mycorrhizal fungal hyphae on soil water retention parameters. Ed Perfect et al.

Cylindrical Geometry Correction



Water-intake Profile Calculator



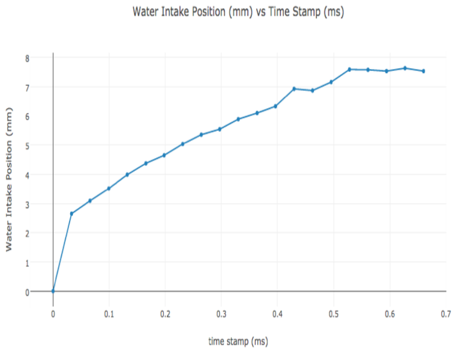
Eagle Ford shale
(1.25cm diameter)
under acoustic excitation
with an acoustic array
(frequencies from 100 to 50,000Hz)

Duration: 37 minutes

Research Team

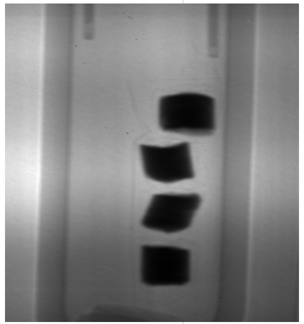
Richard Hale
Jean-Christophe Bilheux
Bruce Patton
Joanna McFarlane
Ayyoub Momen
Paris Cornwell
Hassina Bilheux
Steve Oliver

The CG-1D beamline at the High Flux Isotope Reactor of Oak Ridge National Laboratory



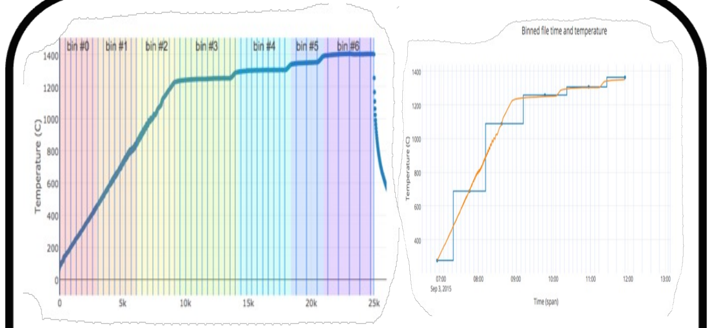
Acoustic facturing in shale samples. Richard Hale et al.

Registration



Characterization of different stages of hydrogen loss in biomass under pyrolysis by radiography and tomography. Frederik Ossler et al.

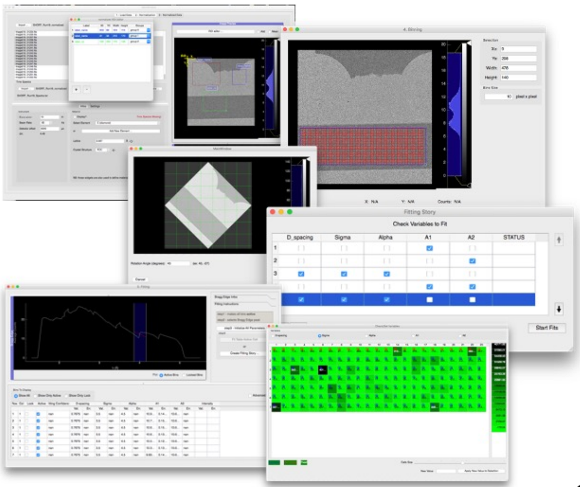
Images and Metadata Extrapolation Matcher



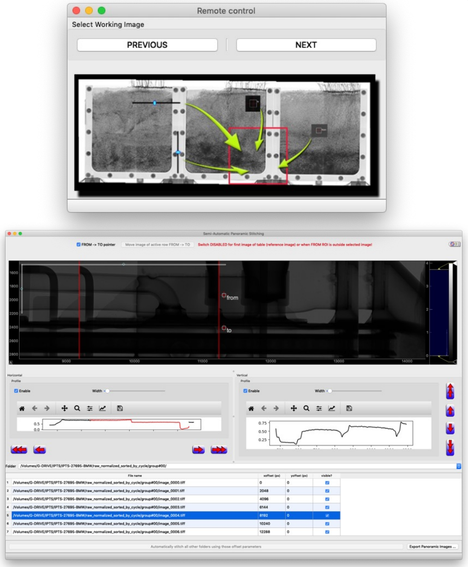
High Temperature Grain Growth Characterization of FeCrAl alloys by Time of Flight Neutron Radiography. Sebastien Dryepondt et al.

Advanced tasks

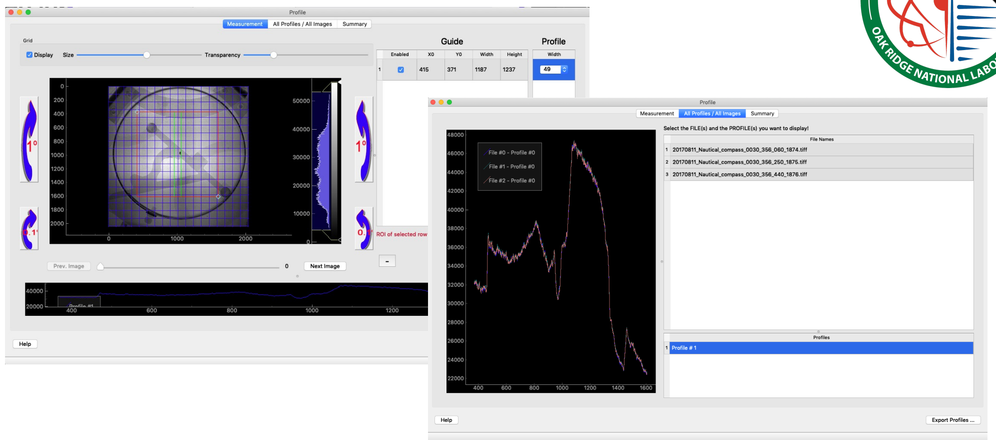
Bragg Edge



Panoramic Stitching

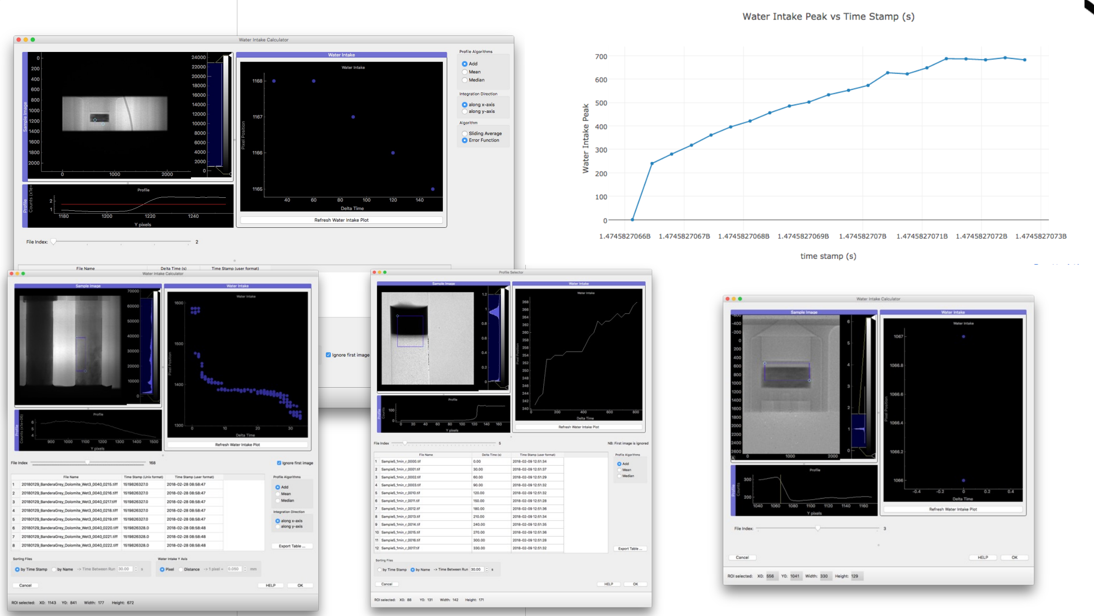


Cylindrical Geometry Correction

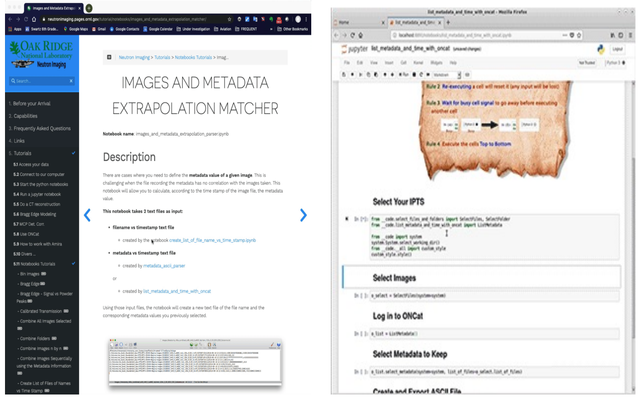


GitHub https://github.com/ornlneutronimaging/CylindricalGeometryCorrection

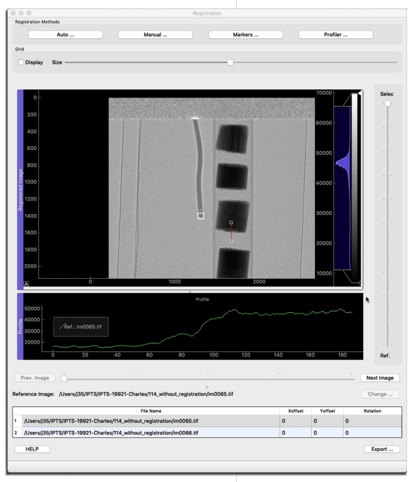
Water-intake Profile Calculator



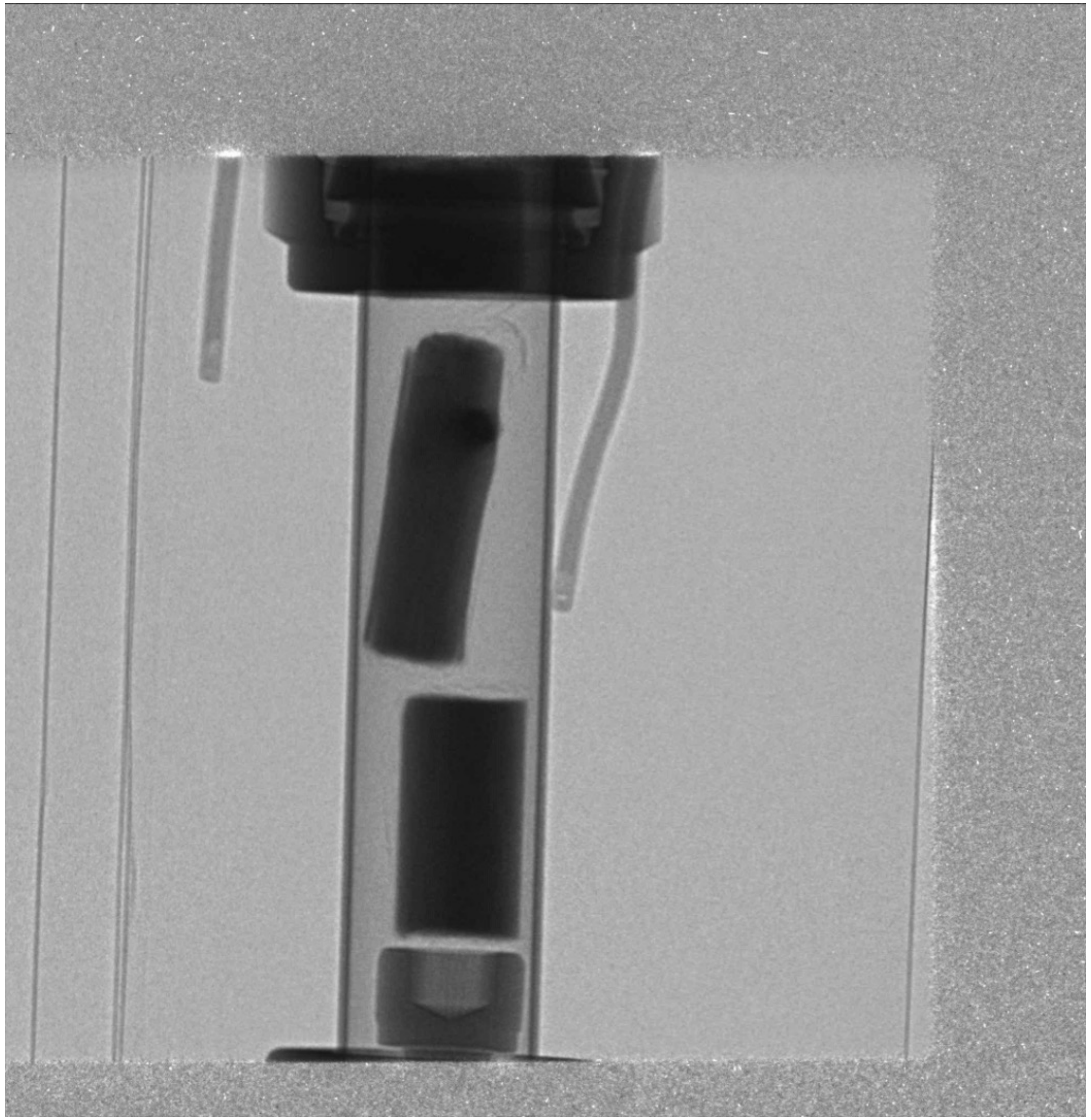
Images and Metadata Extrapolation Matcher



Registration

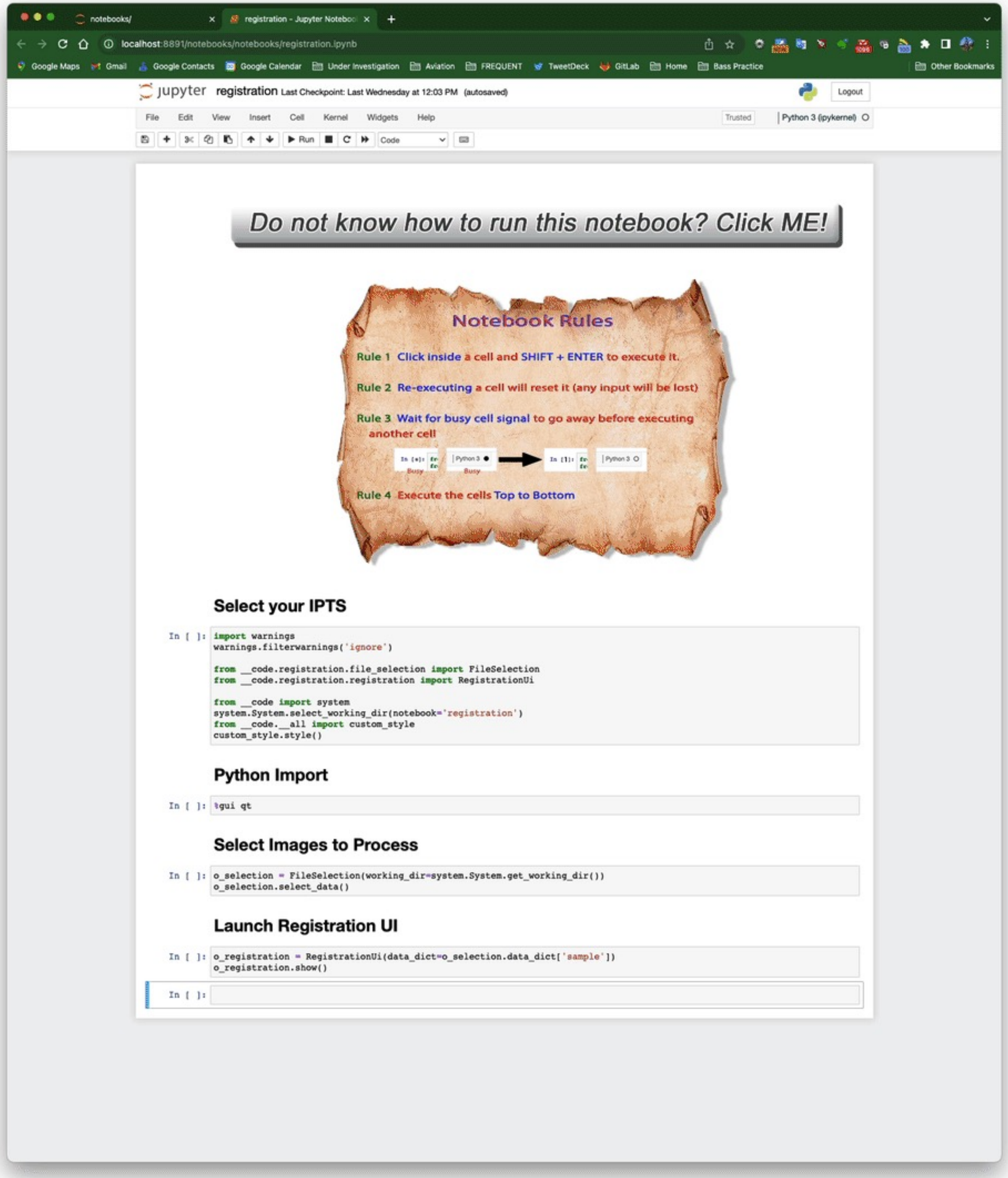


Registration



Courtesy of Charles Finney and Frederik Ossler.

Registration



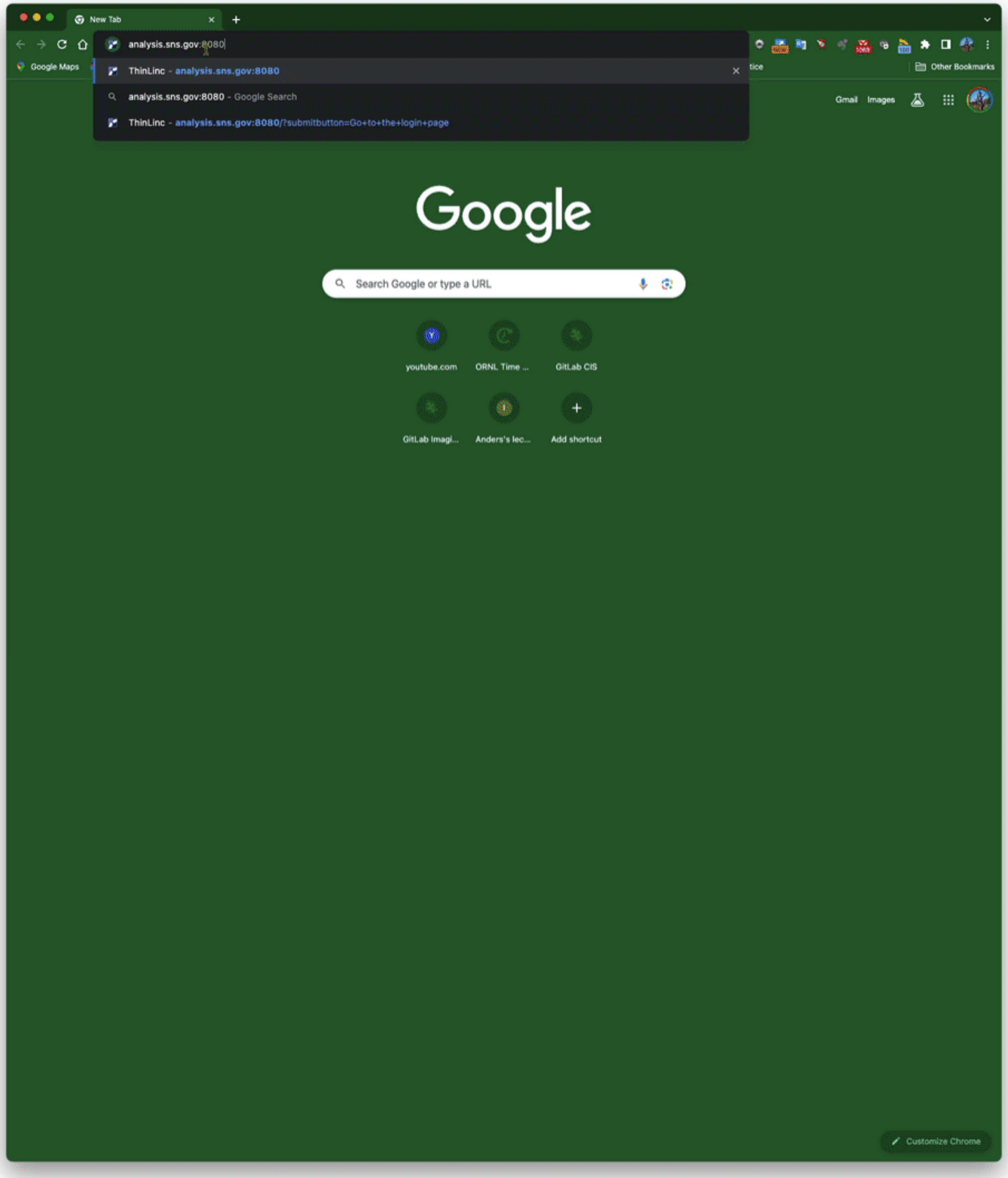
How to access all those tools?

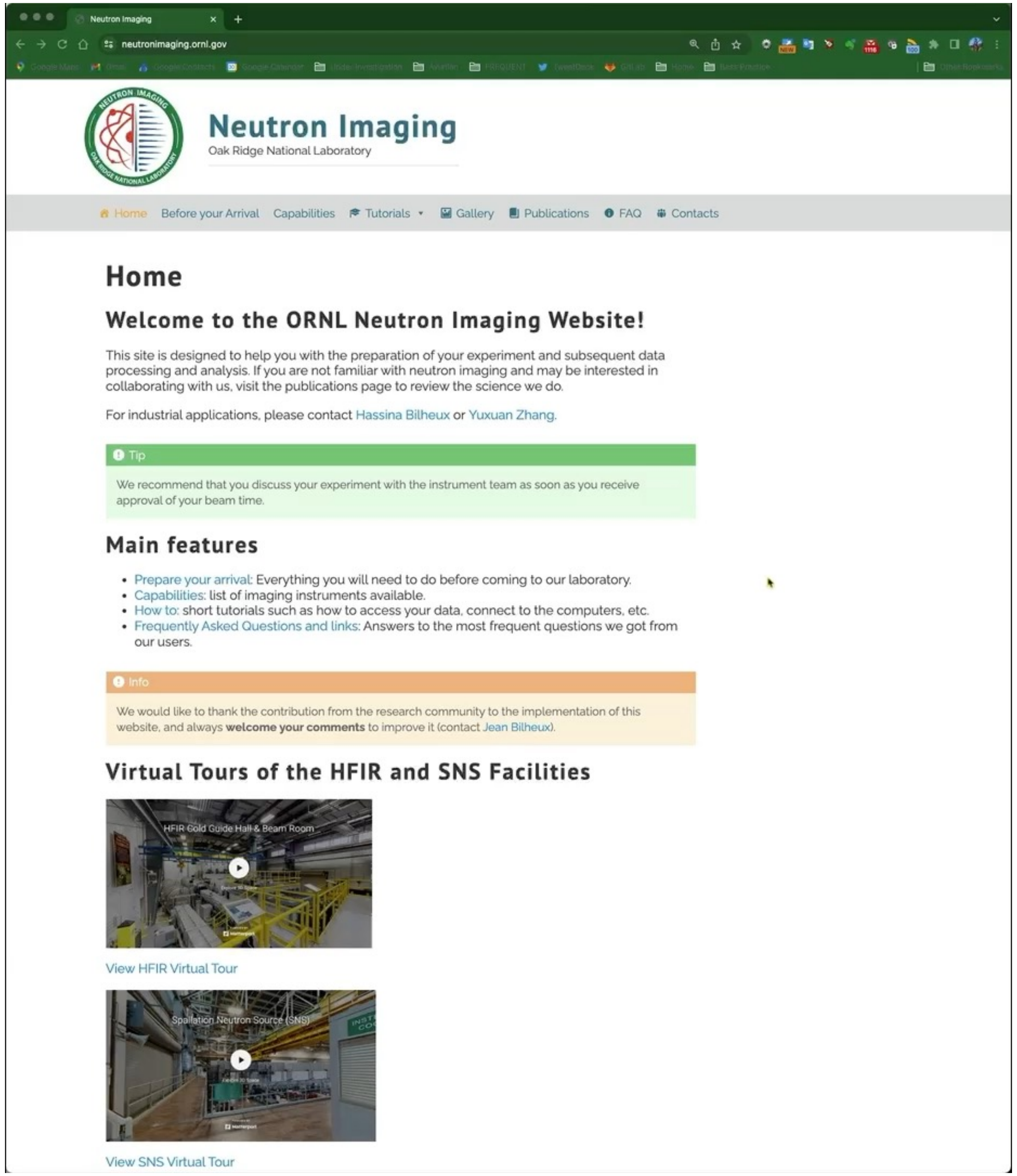
- Go to **analysis.sns.gov**
- Select **Applications > Analysis-Imaging**

PROS
- Nothing to install
- You always get the latest up-to-date version of the software
- No need to move the data (fast access)
- Availability of many other tools (Matlab, IDL, ImageJ, …)

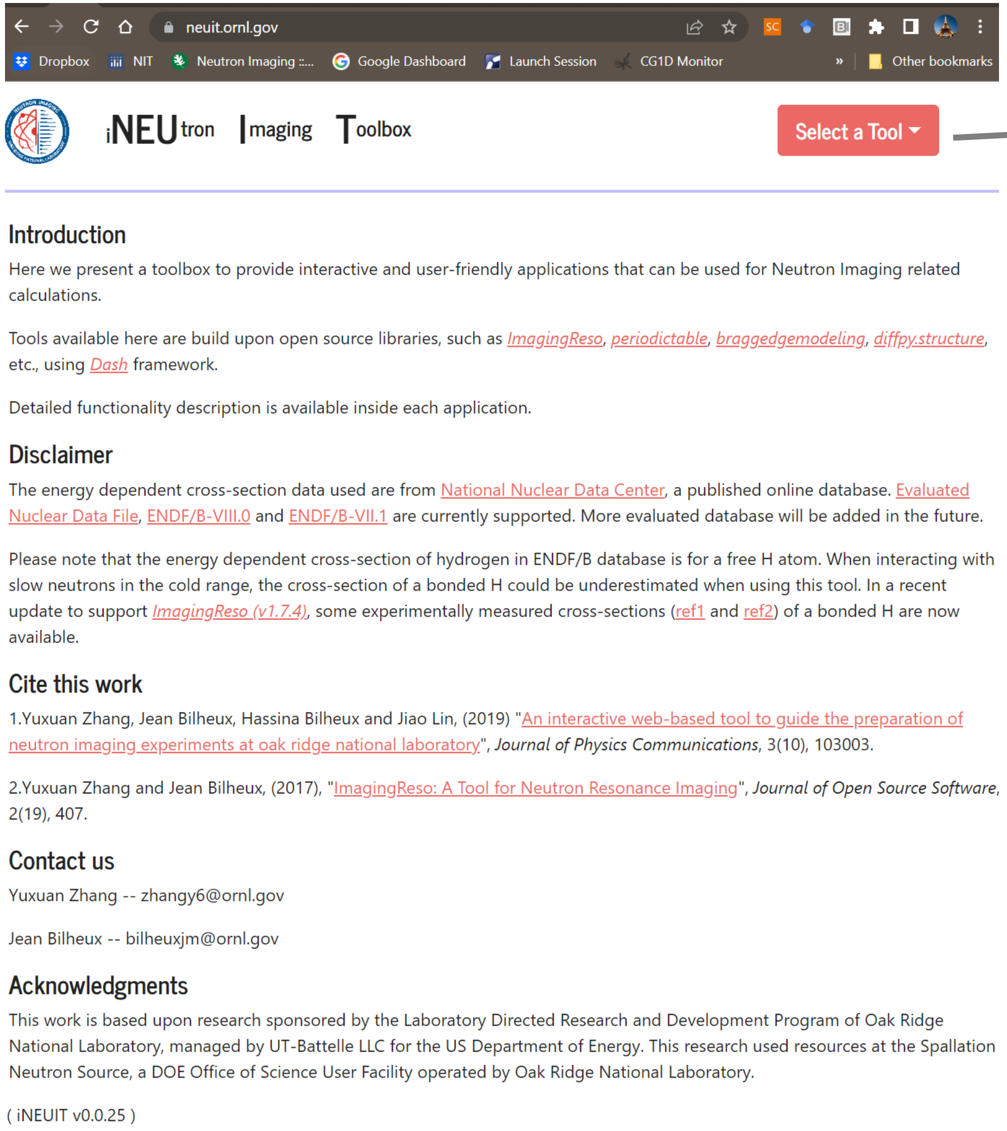
How to access all those tools?

neutronimaging.ornl.gov





Experiment planning tools: *iNEUIT* (i**NEU**tron **I**maging **T**oolbox)



neuit.ornl.gov

Dropbox | NIT | Neutron Imaging :... | Google Dashboard | Launch Session | CG1D Monitor | Other bookmarks

iNEUtron Imaging Toolbox

Select a Tool

Introduction

Here we present a toolbox to provide interactive and user-friendly applications that can be used for Neutron Imaging related calculations.

Tools available here are build upon open source libraries, such as ImagingReso, periodictable, braggedgemodeling, diffpy.structure, etc., using Dash framework.

Detailed functionality description is available inside each application.

Disclaimer

The energy dependent cross-section data used are from National Nuclear Data Center, a published online database. Evaluated Nuclear Data File, ENDF/B-VIII.0 and ENDF/B-VII.1 are currently supported. More evaluated database will be added in the future.

Please note that the energy dependent cross-section of hydrogen in ENDF/B database is for a free H atom. When interacting with slow neutrons in the cold range, the cross-section of a bonded H could be underestimated when using this tool. In a recent update to support *ImagingReso (v1.7.4)*, some experimentally measured cross-sections (ref1 and ref2) of a bonded H are now available.

Cite this work

1.Yuxuan Zhang, Jean Bilheux, Hassina Bilheux and Jiao Lin, (2019) "An interactive web-based tool to guide the preparation of neutron imaging experiments at oak ridge national laboratory", *Journal of Physics Communications*, 3(10), 103003.

2.Yuxuan Zhang and Jean Bilheux, (2017), "ImagingReso: A Tool for Neutron Resonance Imaging", *Journal of Open Source Software*, 2(19), 407.

Contact us

Yuxuan Zhang -- zhangy6@ornl.gov

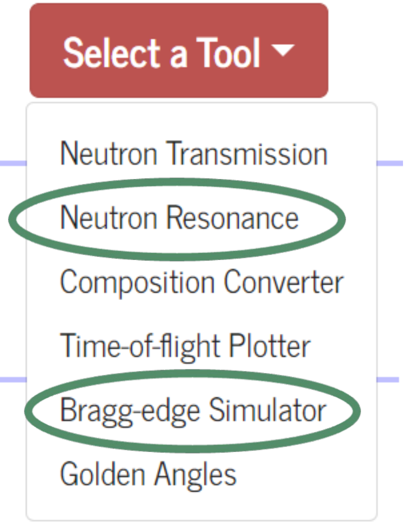
Jean Bilheux -- bilheuxjm@ornl.gov

Acknowledgments

This work is based upon research sponsored by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle LLC for the US Department of Energy. This research used resources at the Spallation Neutron Source, a DOE Office of Science User Facility operated by Oak Ridge National Laboratory.

(iNEUIT v0.0.25)

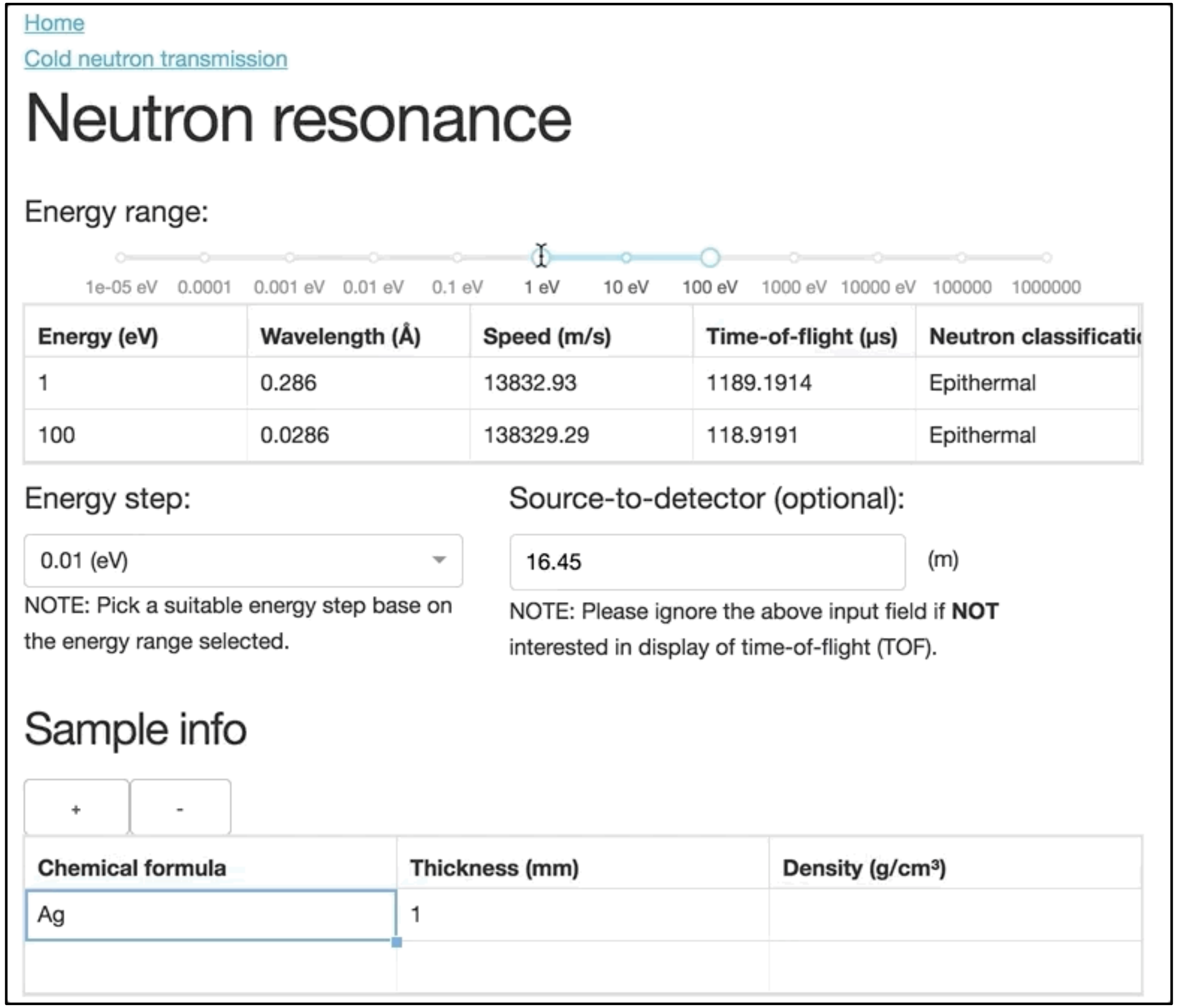
- Available tools:



- Nuclear database supported
  - ENDF/B-VIII.0 (BNL)
  - ENDF/B-VII.1 (BNL)
- Elemental/isotopic info
  - PeriodicTable 1.5.0 (NIST)

iNEUtron Imaging Toolbox (iNEUIT)

https://neuit.ornl.gov



Resonance

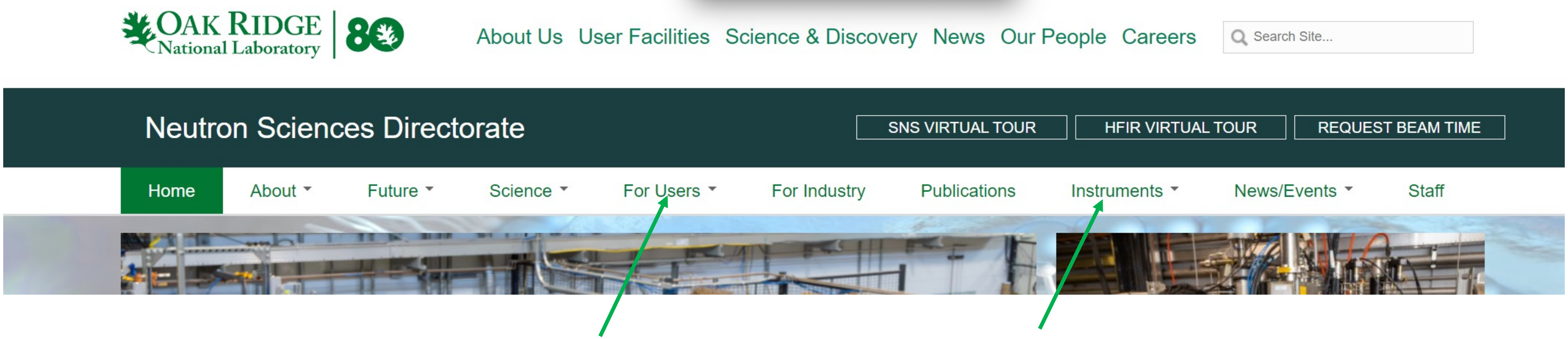
Bragg edge simulator

Thank you

neutronimaging.ornl.gov

The portion of this research used resources at the SNS and HFIR, DOE Office of Science User Facilities operated by the Oak Ridge National Laboratory.

neutrons.ornl.gov



How to collaborate with us

Our beamlines and contact information