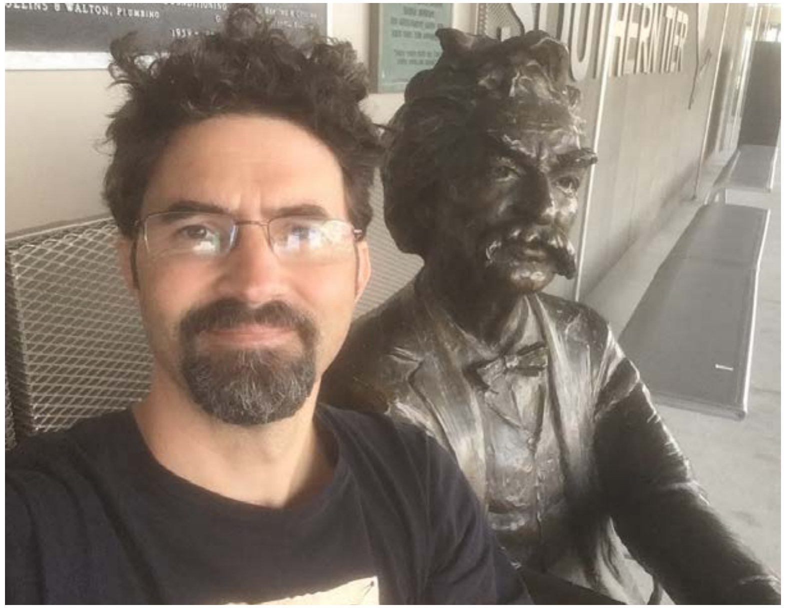


HEXT 2020: X-ray Imaging

Arthur Woll

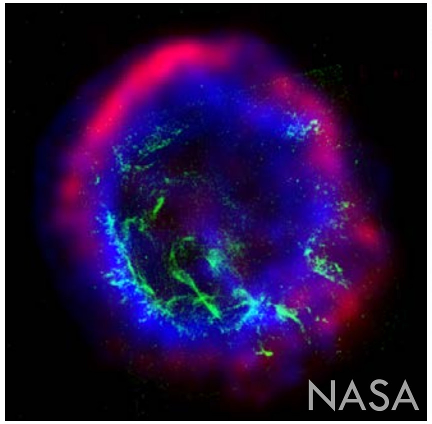
Director, The Materials Solutions Network at CHESS (MSN-C)

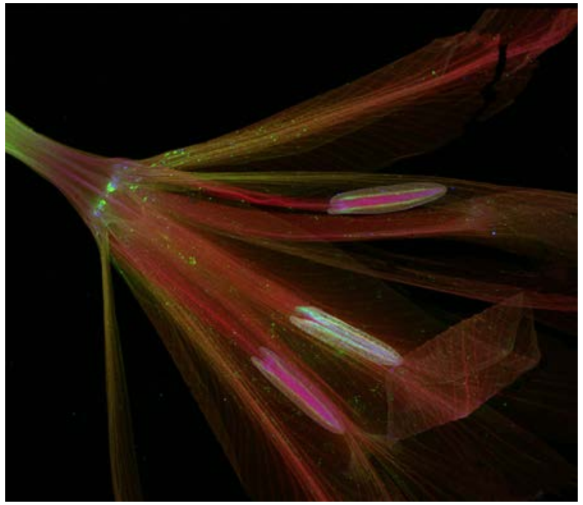
aw30@cornell.edu

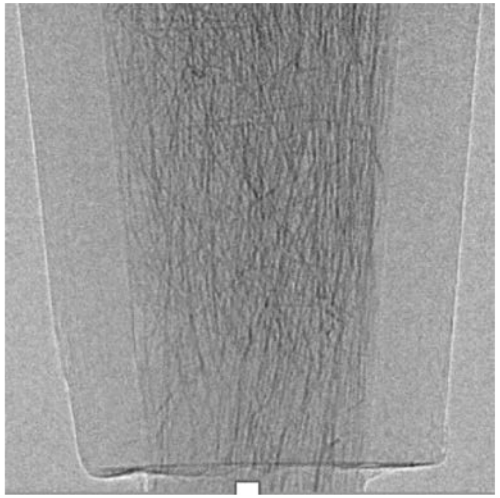
With thanks to: Louisa Smieska and Stan Stoupin (CHESS), and Edward Trigg, Brendan Croom, and Hilmar Koerner (AFRL).

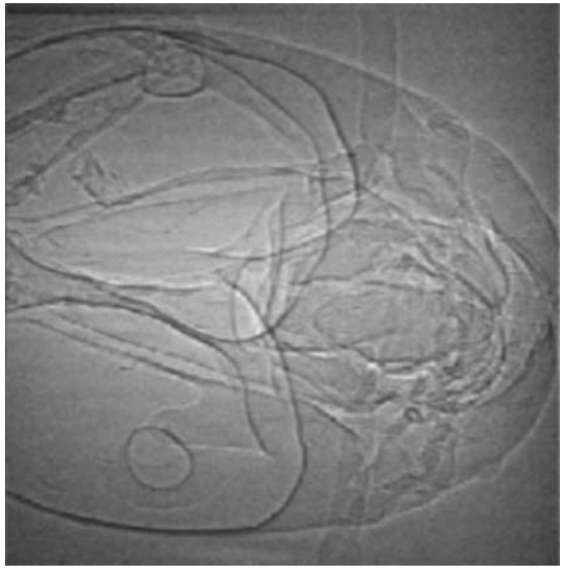
High Energy X-ray Techniques Workshop, June 11th 2020

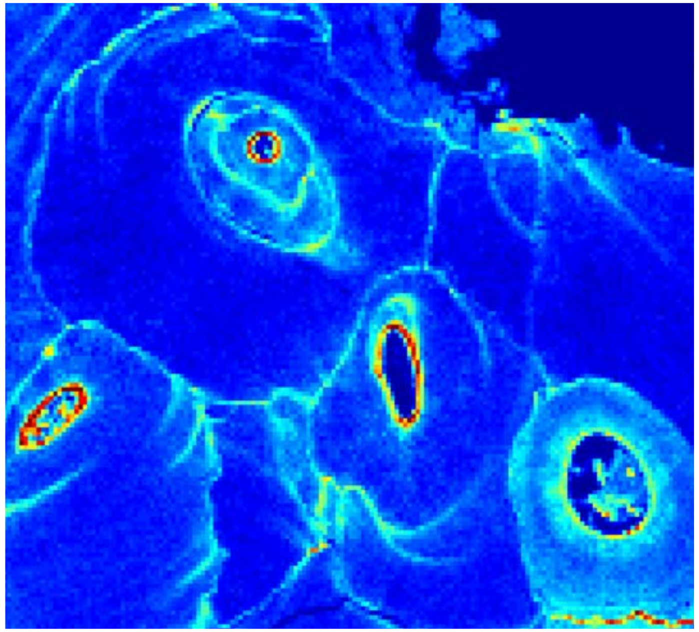
Motivation: “X-ray Imaging” means many things

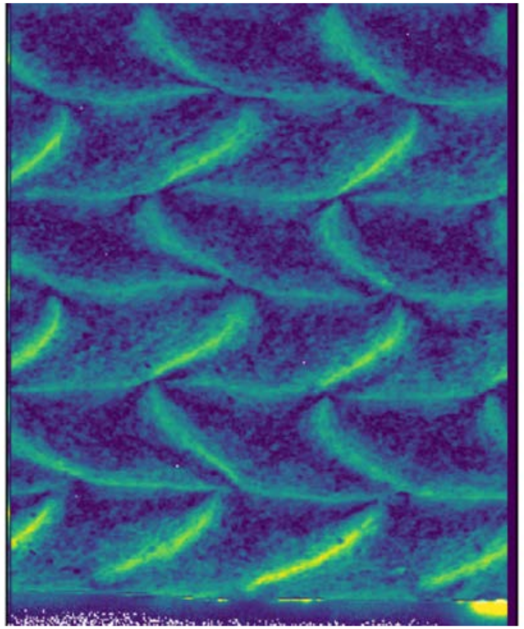




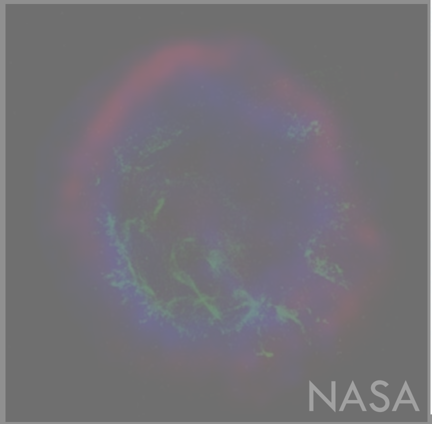


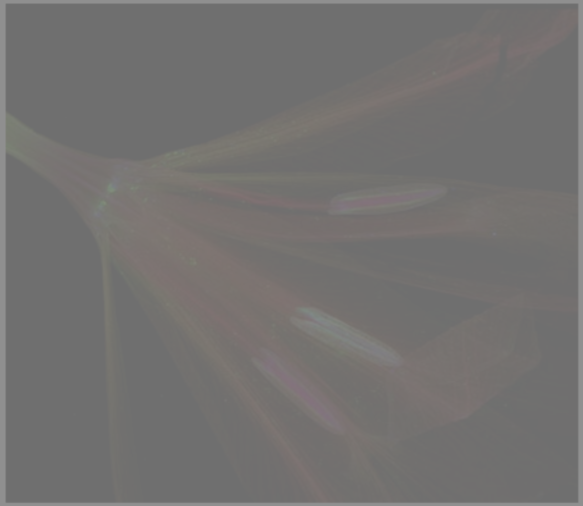


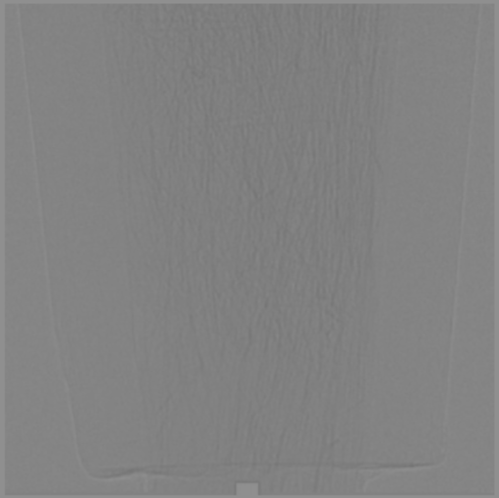


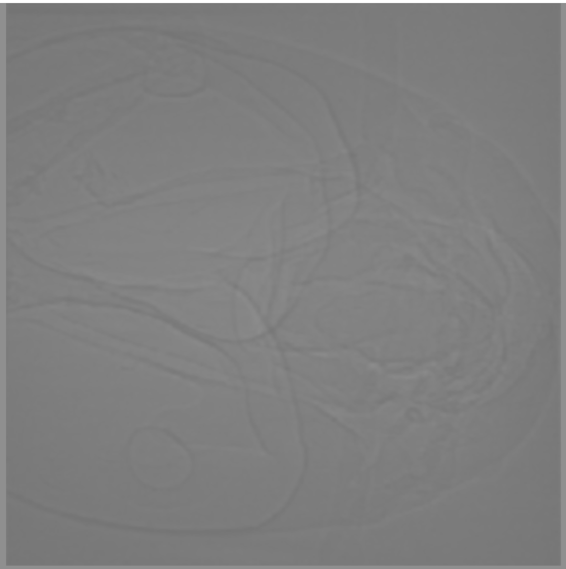


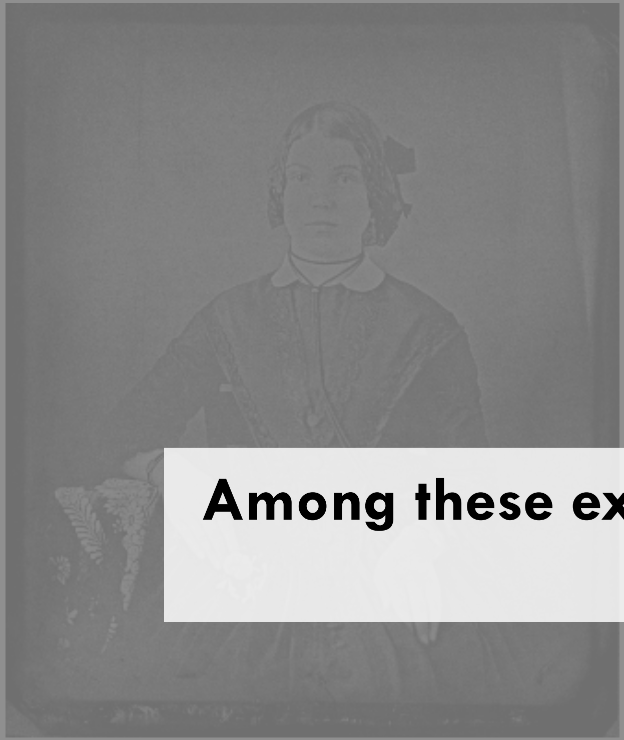
Motivation: “X-ray Imaging” means many things

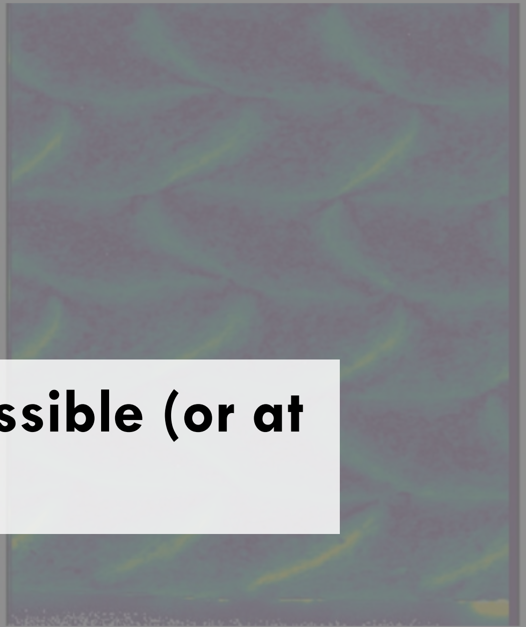






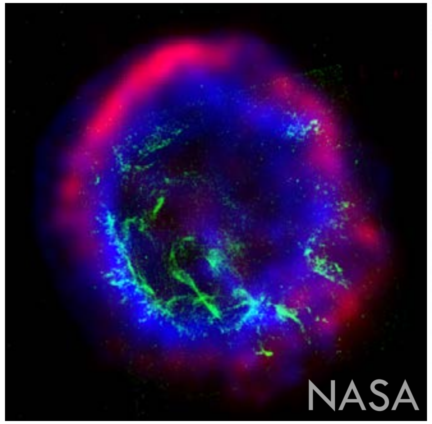


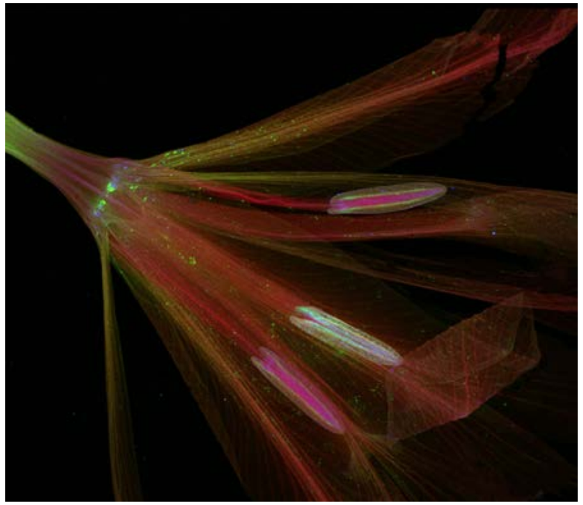
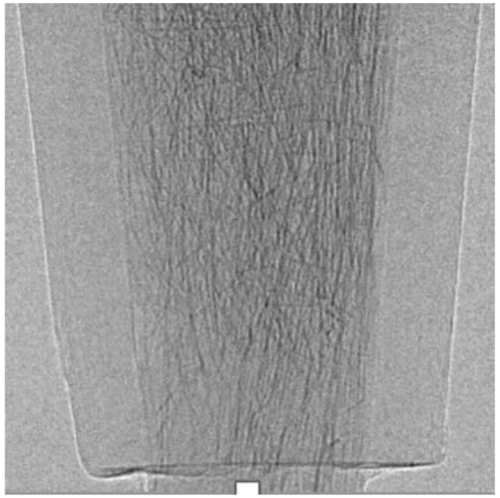
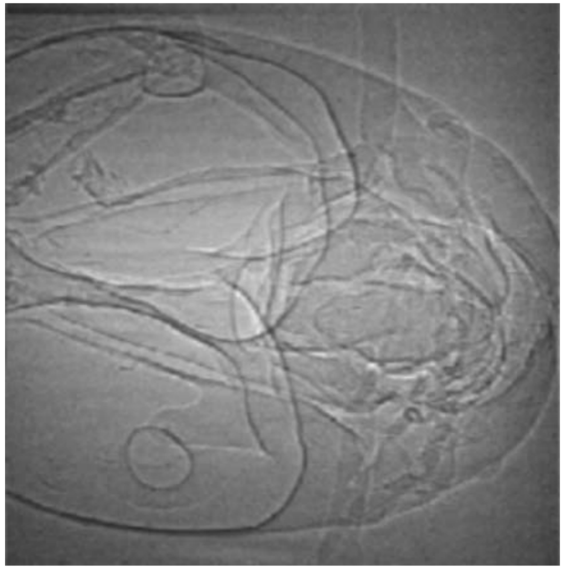


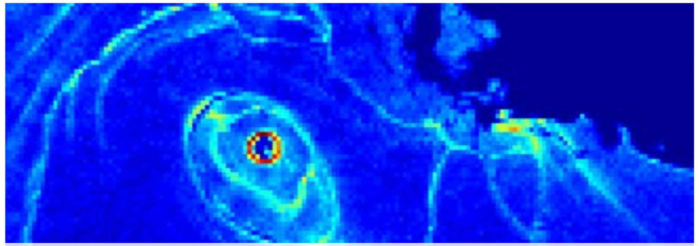
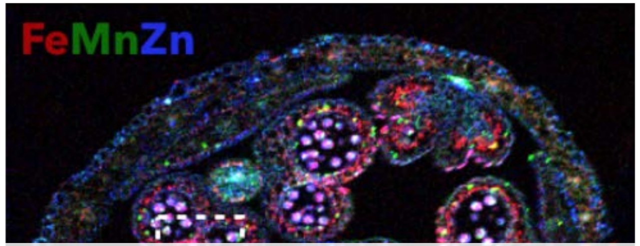


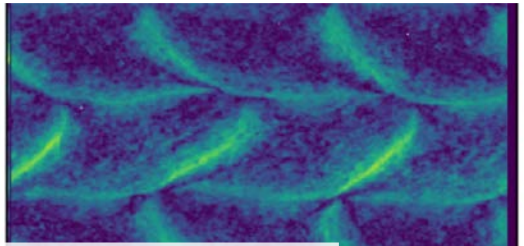
Among these examples, this is the only one that would’ve been possible (or at least practical) 20 years ago.

Motivation: “X-ray Imaging” means many things





1. Resolution: What sets the size and resolution of each image?
2. Contrast: What information does color or brightness represent?

One goal of this workshop is to provide information and intuition for the wide breadth of different answers to these questions for images formed with x-rays.

But first: who is here?

Are you: an undergraduate, graduate student, post-doc, or other?

Have you taken part in a synchrotron experiment?

Do you have a specific x-ray imaging experiment you would like to perform at CHESS?

Topics & Estimated Schedule

General, Introductory

Specific, Advanced

9:00 - 9:45: Introduction to X-ray Imaging

1. **What is X-ray Imaging?** *Definition, Examples, and Key Strengths*
2. **Categorization of different x-ray imaging techniques:**
   1. *Contrast mechanisms:* what determines contrast, brightness & color?
   2. *Image Formation: Full-field* vs. *scan-probe* imaging
3. **Common topics:** Image Quality, Computed Tomography, Focusing

10:00-10:45: The Functional Materials Beamline at CHESS & First Results

1. Absorption contrast imaging during 3D printing
2. SAXS imaging of a 3D-printed epoxy / fiber composite

11:00 – 11:15: Demo of SAXS/WAXS Image Viewing Software & a Jupyter Notebook example of simple image manipulation

“X-ray Imaging” means many things

1. Radiography
2. Computed tomography (xCT)
3. TXM with magnification via:
    a. Fresnel Zone Plates
    b. Refractive Lenses
4. Topography
5. STXM (*usually with soft x-rays*)
6. Phase Contrast Imaging
    a. by free propagation
    b. by diffraction-enhancement
    c. Zernike phase-contrast
    d. Talbot Interferometry
7. K-edge subtraction imaging
8. X-ray holography
9. Coherent Diffraction Imaging & variants: Ptychography, Bragg CDI
10. Nano-, micro-, macroscopic scan-probe: µXRF, µXRD, µSAXS
11. High Energy diffraction-based approaches: EDD, powder diffraction, HEDM.
12. *High Energy* Compton Scatter imaging
13. Other 3D variants:
    a. Confocal XRF
    b. Plenoptic XRM
    c. CT extensions of XRF, SAXS, powder diffraction...

A useful (but not universal!) definition:

x-ray imaging is any technique intended to create a real-space, 2D image of an inhomogeneous sample

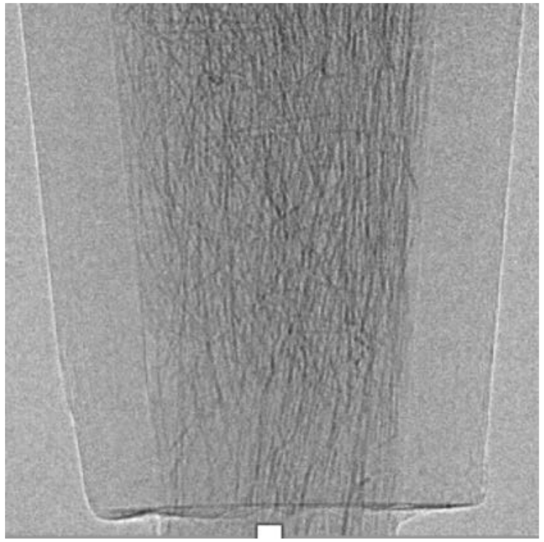
Fundamental Advantages of x-rays:

- High penetration through air, liquids, solids (compared to electrons or light)
- Does not require vacuum
- Wide range of length scales – *Angstroms to meters*
- Many contrast modes – *density, index of refraction, elemental composition, speciation, long & short-range order*
- Many sample geometries
- Speed

A useful (but not universal!) definition:

x-ray imaging is any technique intended to create a real-space, 2D image of an inhomogeneous sample

radiography or other full-field imaging

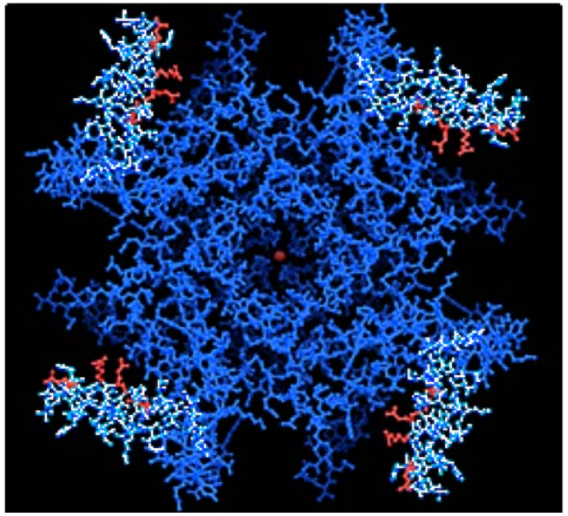


Imaging

XRF or other scan-probe imaging

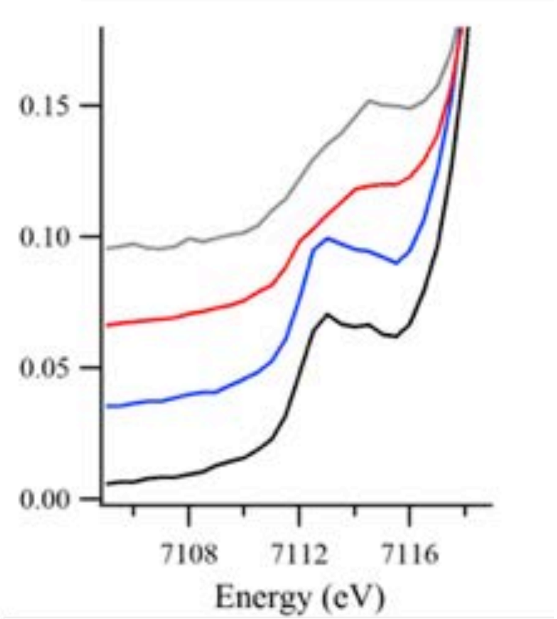
Imaging

crystalography



not Imaging

spectroscopy



not Imaging

X-ray Imaging: Organizational Schemes

1. **Historical:** Radiography (1895), Lens-less Microscopy (1913), FZP-based TXM (1940s), Tomography (1970s), Practical TXMs (1970s); Scan-probe methods (STXM, uXRF, uXRD, 1980s), Coherent Diffraction Imaging (1999), Phase-Contrast Imaging (1965, 1995, 2006), Holography (2004), ...
2. **Applications:** Biology, Chemistry, Geology, Physics, Materials Science,...
3. **Critical Physics:**
   a. Optics (wavelength, index of refraction, numerical aperture)
   b. Contrast Mechanisms (*Diffraction, Absorption, Fluorescence*)
   c. X-ray detection.
4. **Image Formation:** Full Field vs. Scan-Probe Imaging
5. **Instrumentation:** Beam & source characteristics, sample size, optics, speed, detection methods.

X-ray Imaging: Organizational Schemes

1. **Historical:** Radiography (1895), Lens-less Microscopy (1913), FZP-based TXM (1940s), Tomography (1970s), Practical TXMs (1970s); Scan-probe methods (STXM, uXRF, uXRD, 1980s), Coherent Diffraction Imaging (1999), Phase-Contrast Imaging (1965, 1995, 2006), Holography (2004), ...
2. **Applications:** Biology, Chemistry, Geology, Physics, Materials Science,...
3. **Critical Physics:**
   a. Optics (wavelength, index of refraction, numerical aperture)
   b. **Contrast Mechanisms (Diffraction, Absorption, Fluorescence)**
   c. X-ray detection.
4. **Image Formation: Full Field vs. Scan-Probe Imaging**
5. **Instrumentation:** Beam & source characteristics, sample size, optics, speed, detection methods.

Claim: These are the two most informative facts about an imaging technique:

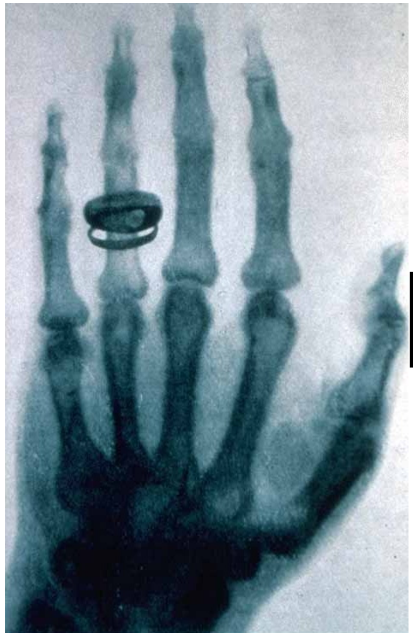
1. **What is the Contrast Mechanism?** ***(Diffraction, Absorption, Fluorescence)***
2. **How are images formed?** ***Full Field*** **vs.** ***Scan-Probe*** **Imaging**

Why? Because most other questions will be determined in part by these two.

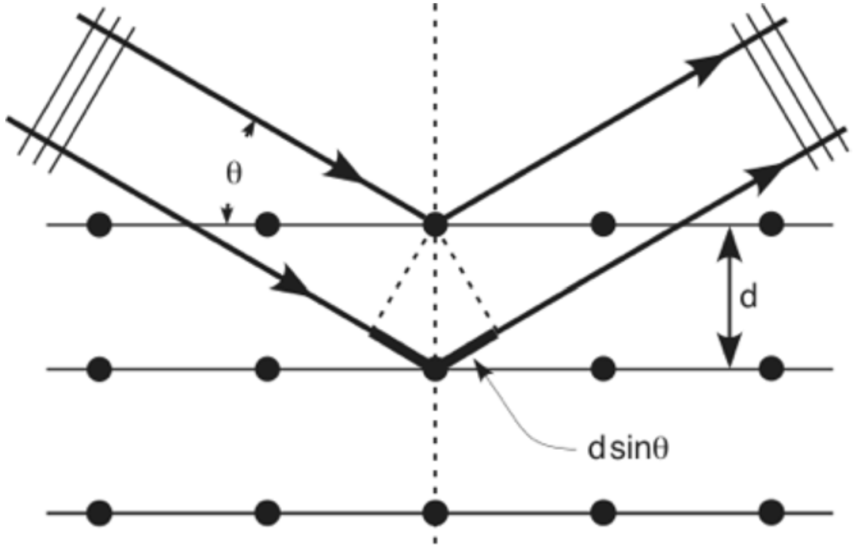
1. What can I learn about my sample?
2. What spatial resolution is possible?
3. What *time resolution* is possible?
4. What limits sample geometry?
5. How quantitative is the information?
6. How sensitive is the technique?

Contrast Mechanisms

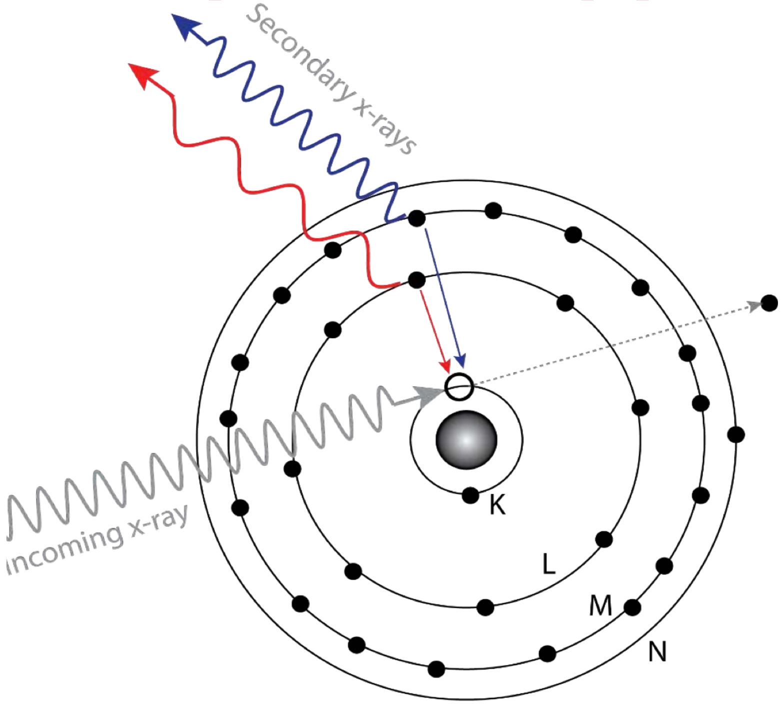
Absorption / Refraction



Scattering / Diffraction



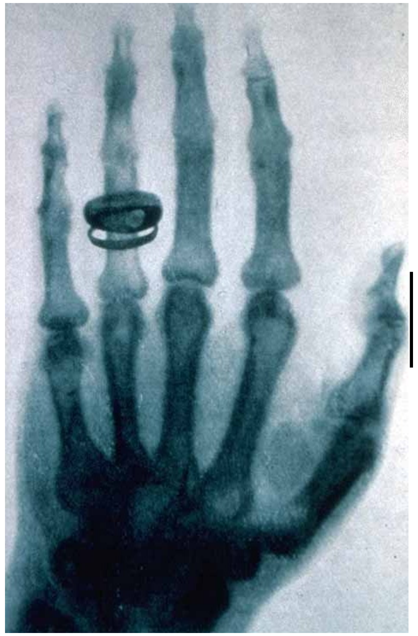
Fluorescence / Spectroscopy



Question: What do these interactions reveal about a sample?

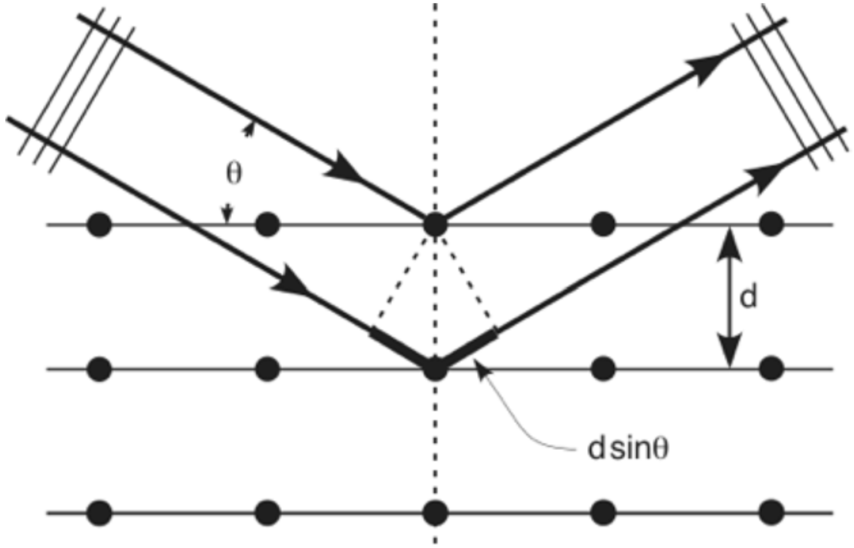
Question: What do these contrast mechanisms reveal about a sample?

Absorption / Refraction



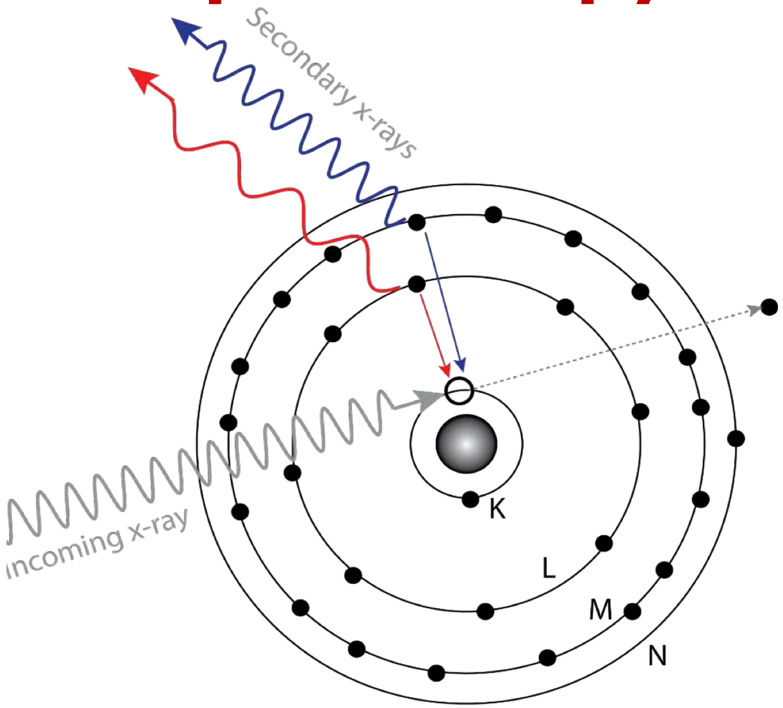
Density

Scattering / Diffraction



Molecular/Atomic Scale Order

Fluorescence / Spectroscopy



Chemical Composition / Speciation

Claim: These are the two most informative facts about an imaging technique:

1. **What is the Contrast Mechanism? *(Diffraction, Absorption, Fluorescence)***
2. **How are images formed? *Full Field* vs. *Scan-Probe* Imaging**

Why? Because most other questions will be determined in part by these two.

1. What can I learn about my sample?
2. What spatial resolution is possible?
3. What *time resolution* is possible?
4. What limits sample geometry?
5. How quantitative is the information?
6. How sensitive is the technique?

Image Formation: ”Full-field” vs. “Scan-probe” Imaging

• Full Field Imaging

- Resolution determined by detector(~1 µm) or lens (10 nm).
- Frame Rate: **Hz to MHz**
- Contrast: Absorption, Phase, Diffraction*, Compton*.

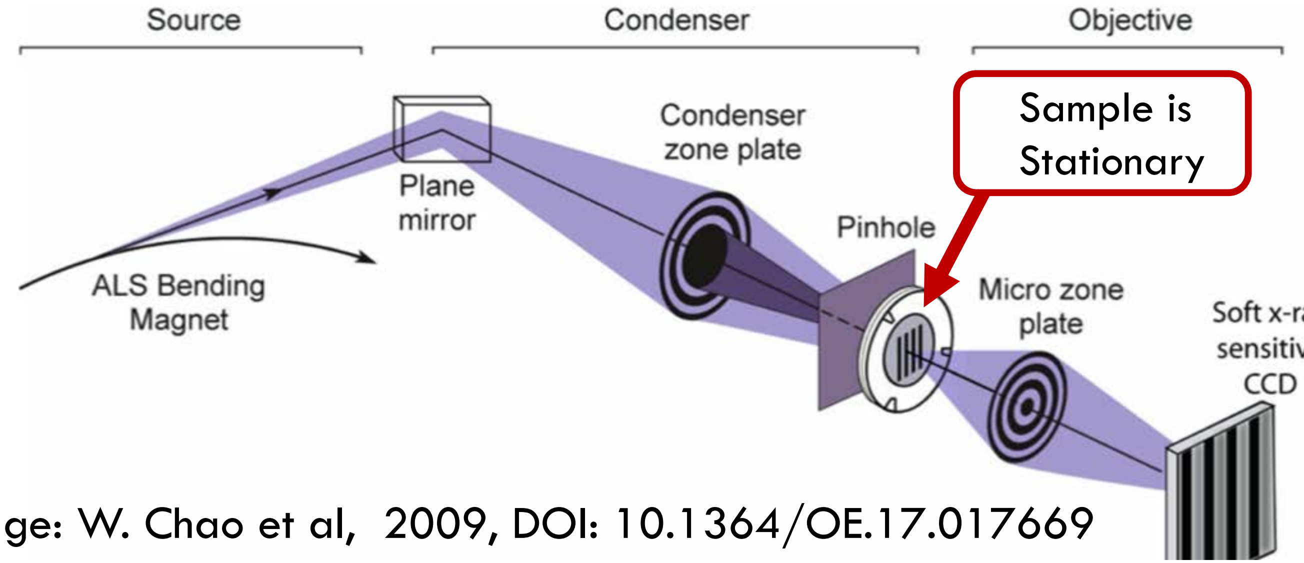


Image: W. Chao et al, 2009, DOI: 10.1364/OE.17.017669

• Scan Probe Imaging

- Resolution determined by incident beam-size – reaching ~10 nm
- Time/frame: **Minutes to hours.**
- Contrast: Absorption, Phase, Diffraction, Compton, SAXS, fluorescence, XANES, EXAFS,...

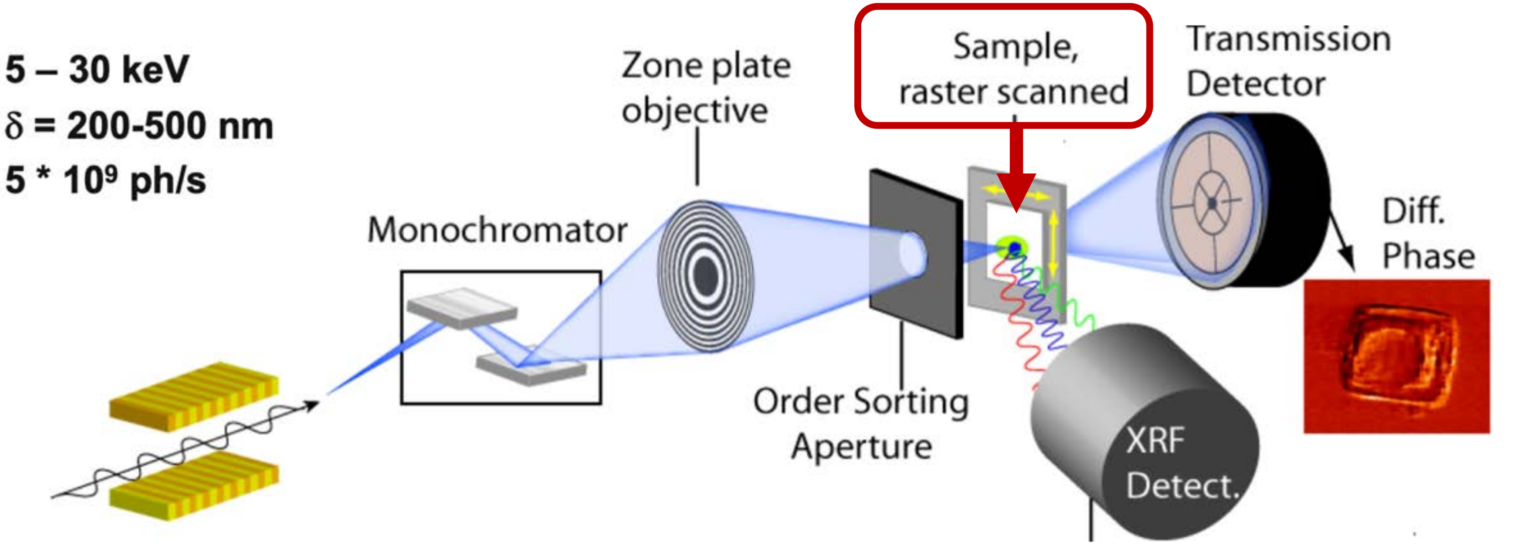


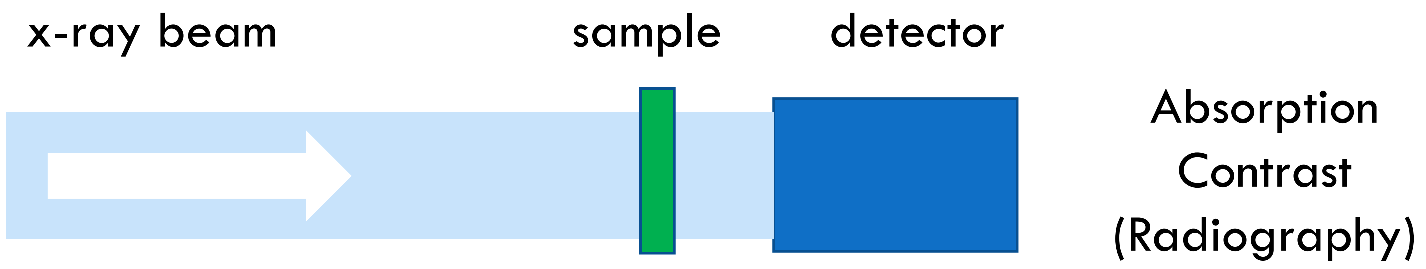
Image: Stephen Vogt (2016)

“X-ray Imaging” means many things

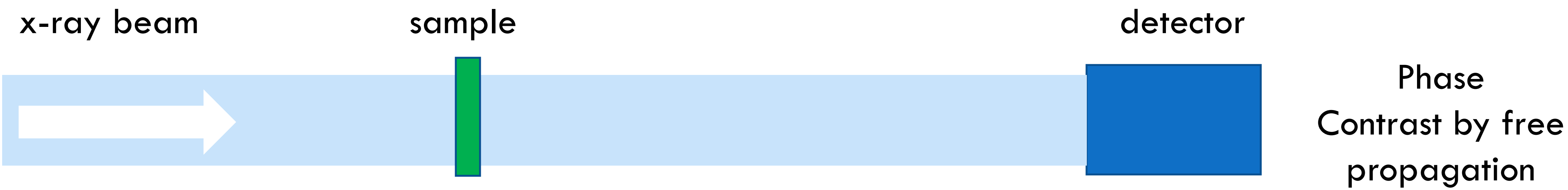
1. Radiography
2. Computed tomography (xCT)*
3. TXM with magnification via:
   a. Fresnel Zone Plates
   b. Refractive Lenses
4. Topography
5. STXM (*usually with soft x-rays*)
6. Phase Contrast Imaging
   a. by free propagation
   b. by diffraction-enhancement
   c. Zernike phase-contrast
   d. Talbot Interferometry
7. K-edge subtraction imaging
8. X-ray holography
9. Coherent Diffraction Imaging & variants: *Ptychography*, Bragg CDI
10. Nano-, micro-, macroscopic scan-probe: μXRF, μXRD, μSAXS
11. High Energy diffraction-based approaches: EDD, powder diffraction, *HEDM*
12. (High Energy) Compton Scatter imaging
13. Other 3D variants:
    a. Confocal XRF
    b. Plenoptic XRM
    c. CT extensions of XRF, SAXS, powder diffraction...

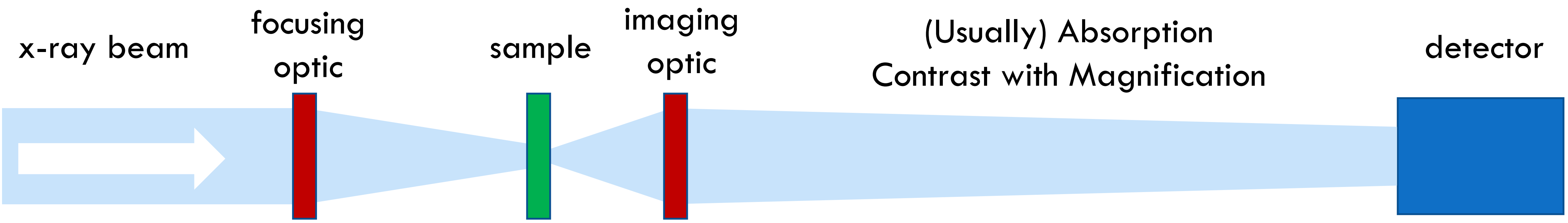
Full-Field, Scan-Probe

Full-field Image Formation: Example configurations



Spatial resolution determined by detector pixel size, beam divergence, or the magnification of the imaging lens





Full-field Imaging examples: Radiography

APPLIED PHYSICS LETTERS VOLUME 83, NUMBER 8 25 AUGUST 2003

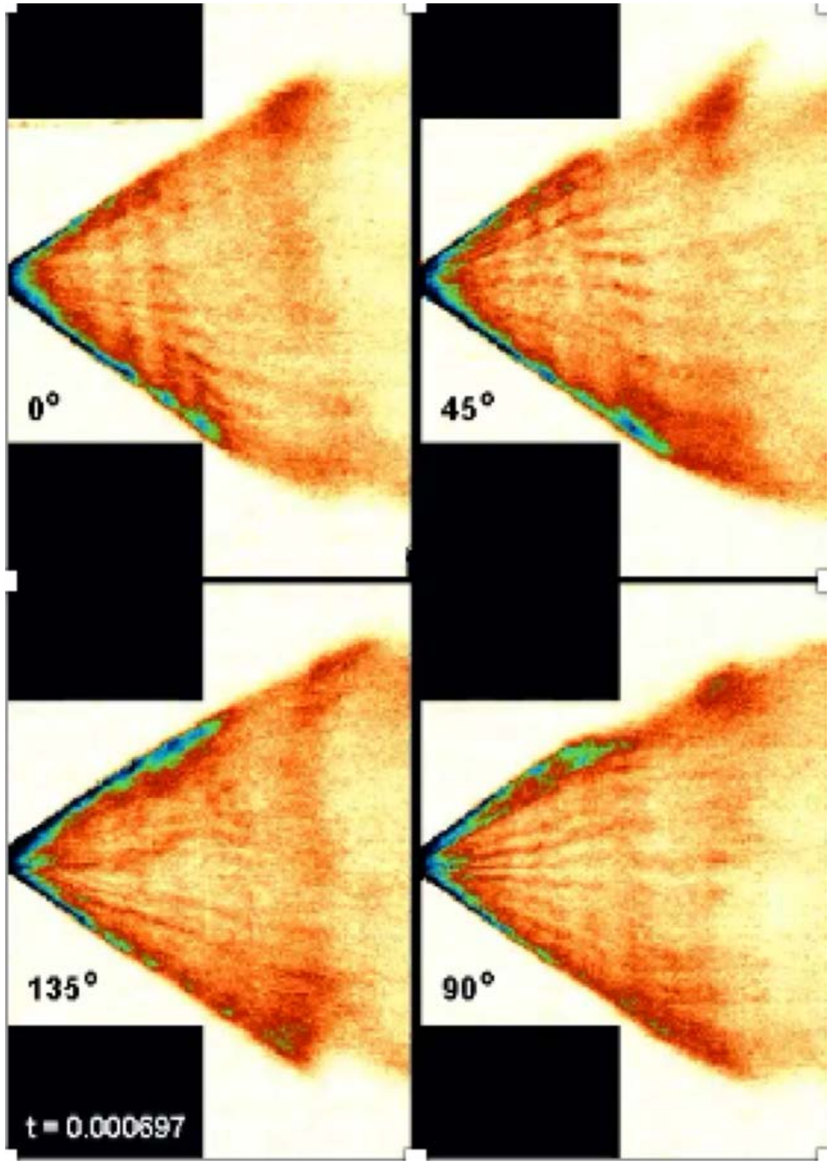
Quantitative analysis of highly transient fuel sprays by time-resolved x-radiography

Wenyi Cai,[a),b)] Christopher F. Powell,[a),c)] Yong Yue,[c)] Suresh Narayanan,[a)] and Jin Wang[a),d)]
Argonne National Laboratory, Argonne, Illinois 60439

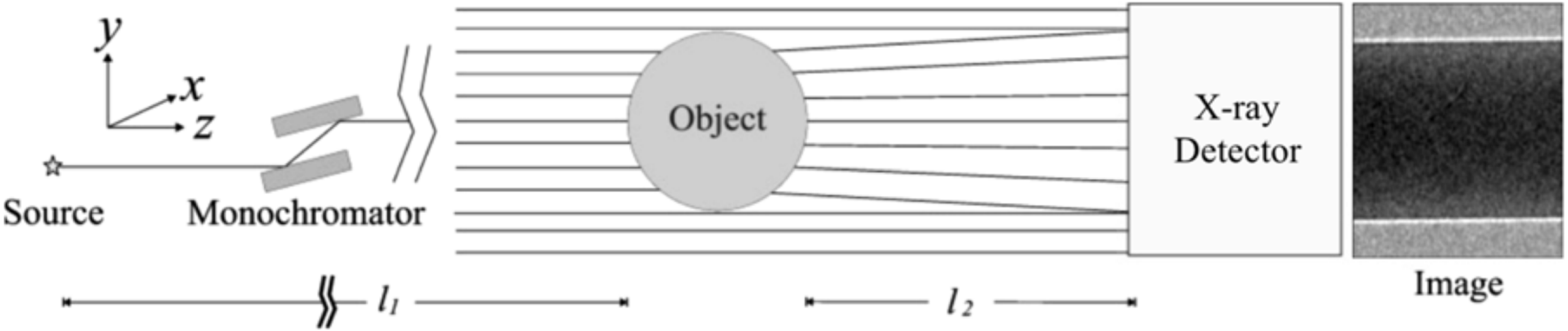
Mark W. Tate,[e)] Matthew J. Renzi,[e)] Alper Ercan,[e)] Ernest Fontes,[f)] and Sol M. Gruner[e),f)]
Cornell University, Ithaca, New York 14853

(Received 25 March 2003; accepted 6 June 2003)

Microsecond time-resolved synchrotron x-radiography has been used to elucidate the structure and dynamics of optically turbid, multiphase, direct-injection gasoline fuel sprays. The combination of an ultrafast x-ray framing detector and tomographic analysis allowed three-dimensional reconstruction of the dynamics of the entire 1-ms-long injection cycle. Striking, detailed features were observed, including complex traveling density waves, and unexpected axially asymmetric flows. These results will facilitate realistic computational fluid dynamic simulations of high-pressure sprays and combustion. [DOI: 10.1063/1.1604161]

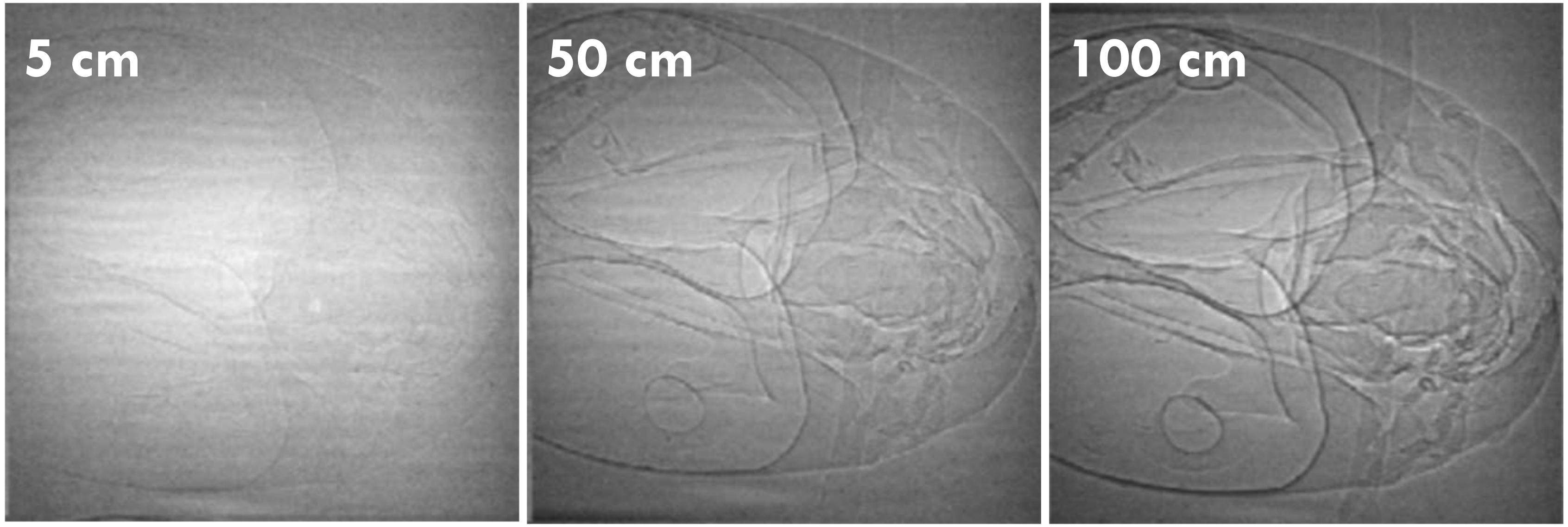


Full-field Imaging example: Phase-Contrast Imaging



Croton et al, *Scientific Reports* **8** 11412 (2018)

Example: PCI of an ant head, illustrating *increasing* contrast with sample-to-detector distance.



Socha et al, BMC Biology **5**(6) 2007, doi:10.1186/1741-7007-5-6

Scan-field Image Formation: Example configurations

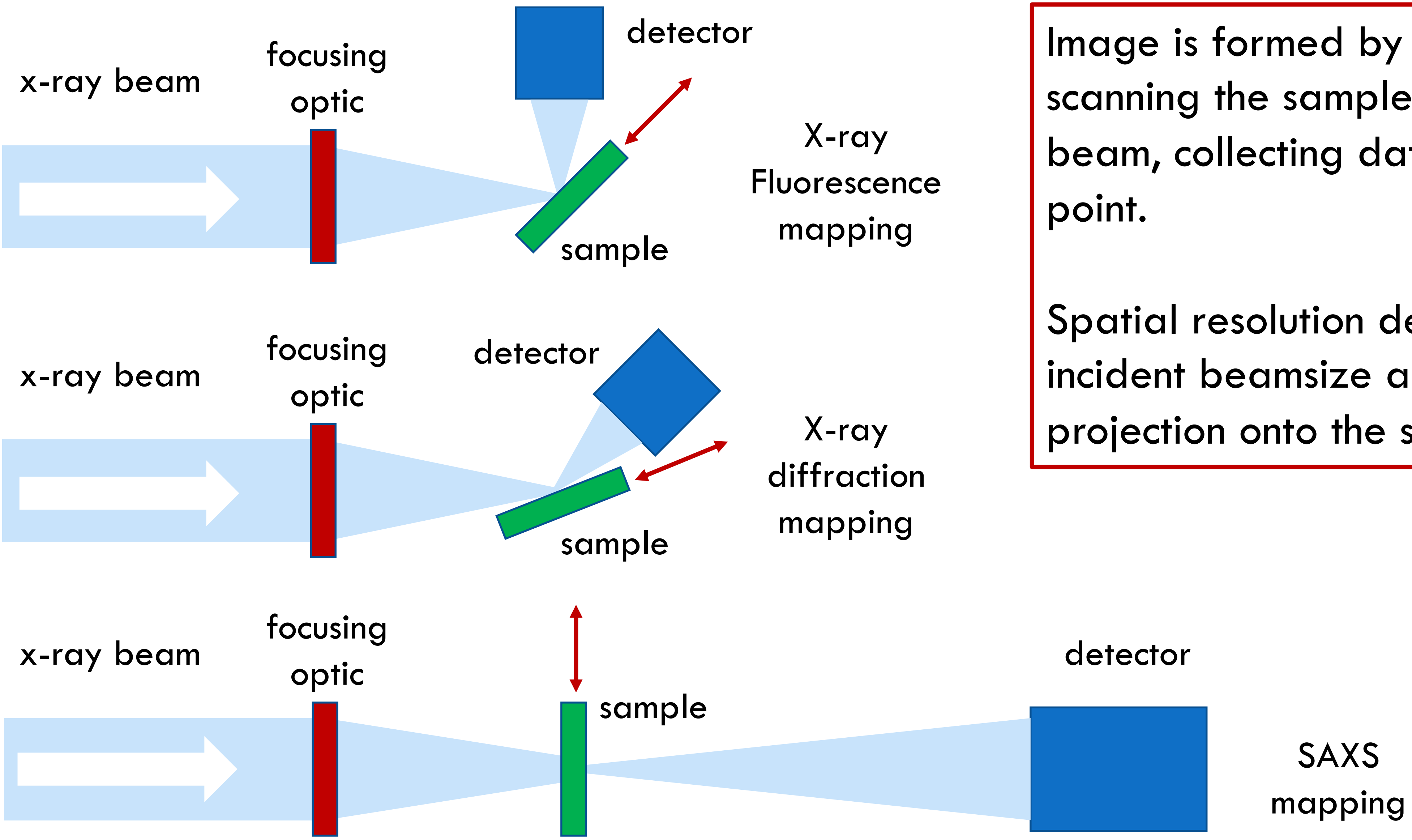
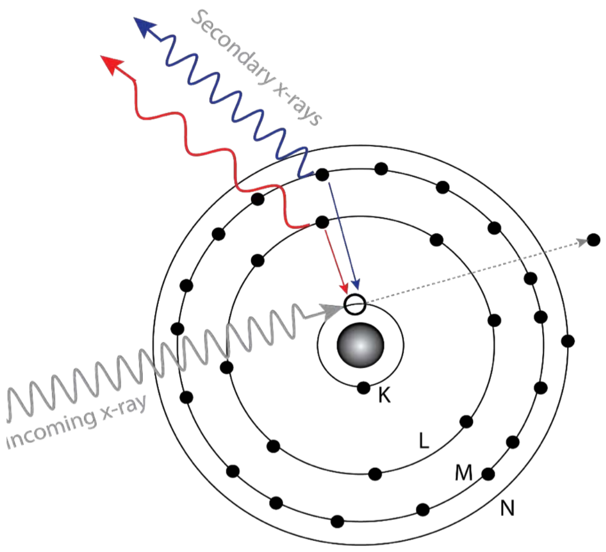


Image is formed by raster-scanning the sample through the beam, collecting data at each point.

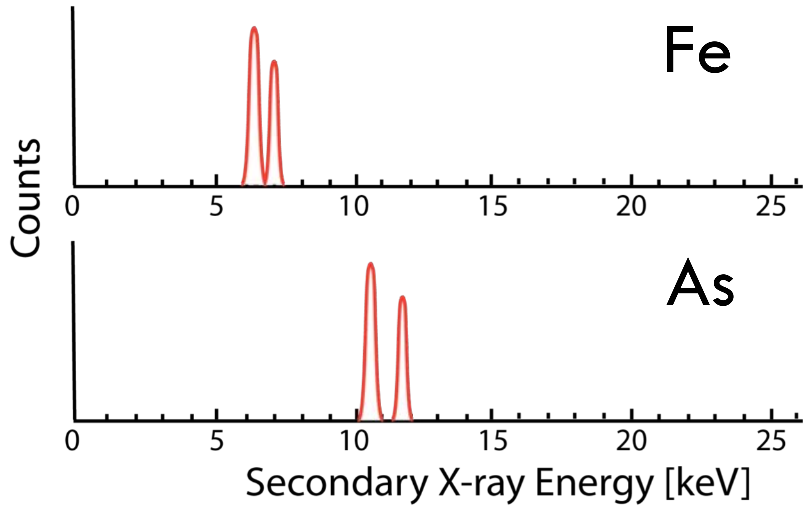
Spatial resolution determined by incident beamsize and its projection onto the sample

Scan-probe Imaging Example: µXRF

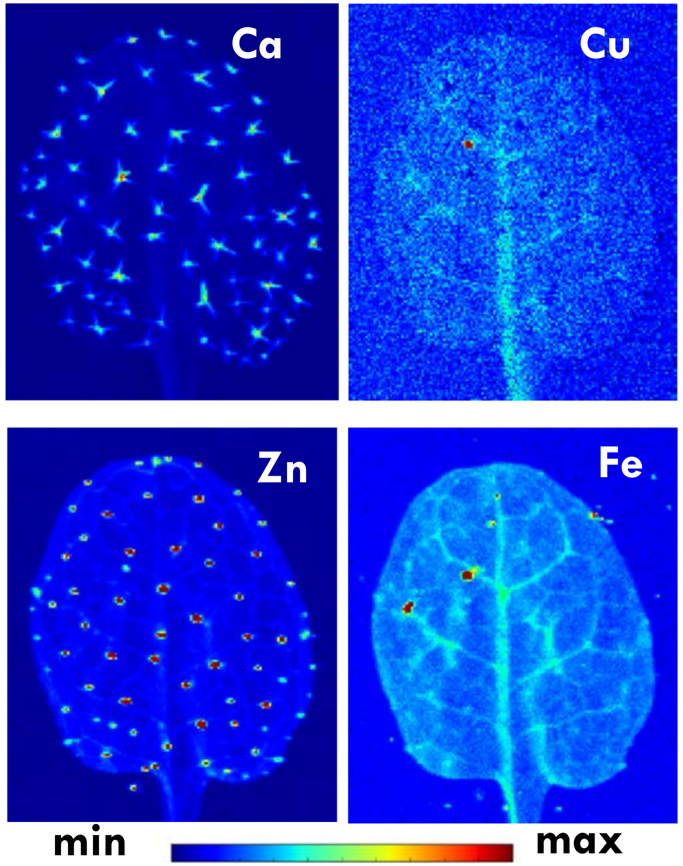
X-ray absorption by an atom results in the re-emission of lower energy X-rays (fluorescence)



- Each element of the sample has a unique energy (fluorescence) fingerprint.
- Spectra are collected while sample is rastered through beam



Peak areas are extracted at each point, forming one image per element



Kumar et al,, **2017**, ***Plant Phys***, *175(3):1254-1268*

Scan-probe Imaging Example: ”Macro”-XRF

Visible Light

Raster scan, ~10 hours

1 cm

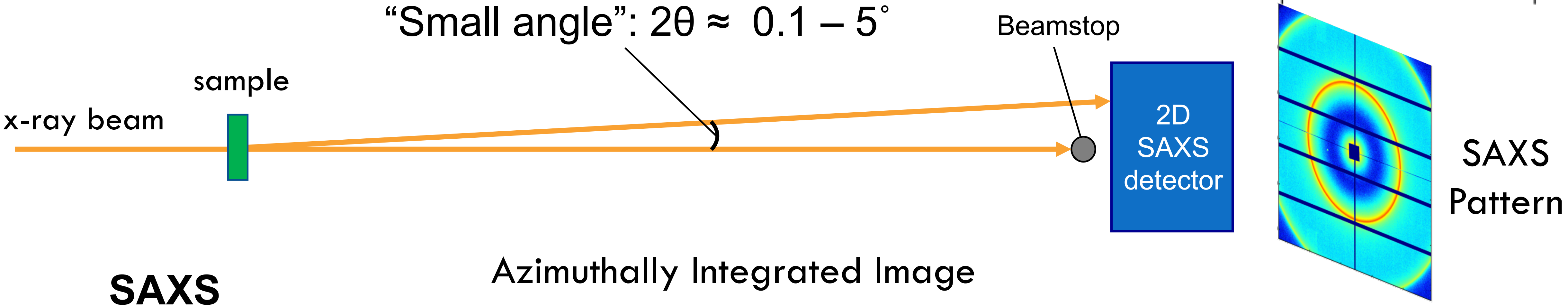
Mercury Distribution

Gold Distribution

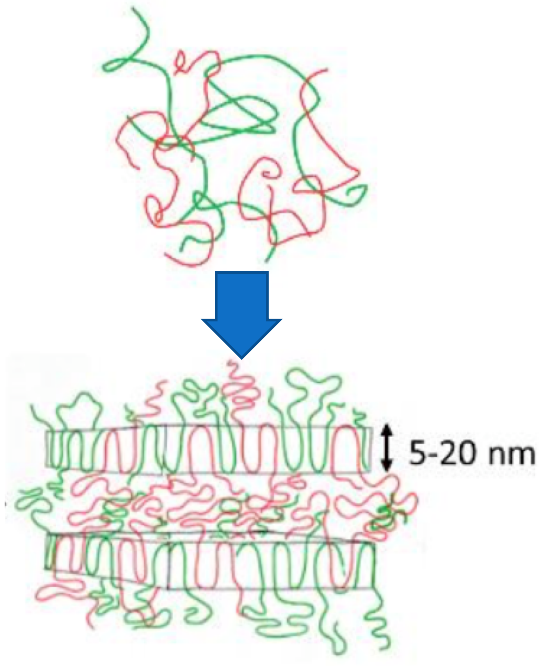
Kozachuk, M.S.,..., Smieska, L.; Woll, A.R. *Heritage* **2019**, 2, 568-586.; doi.org/10.3390/heritage2010037

Scan-probe Imaging Example: SAXS

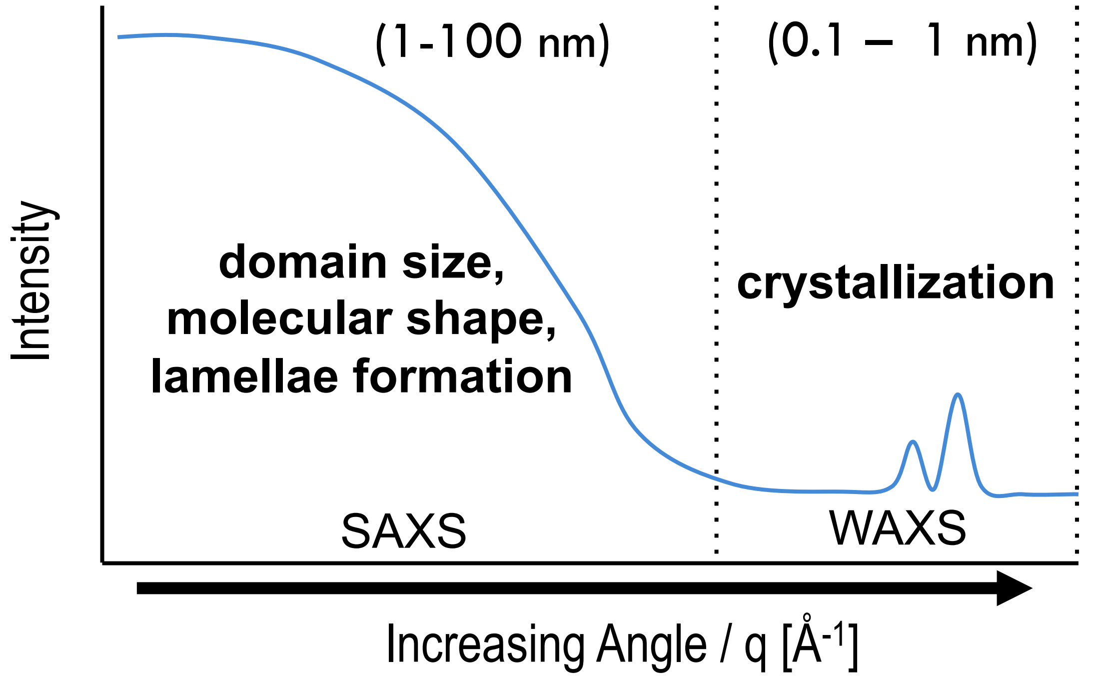
$$I(\vec{q}) \propto \left| \int \rho(\vec{r}) e^{-i\vec{q}\cdot\vec{r}} d\vec{r} \right|^2$$



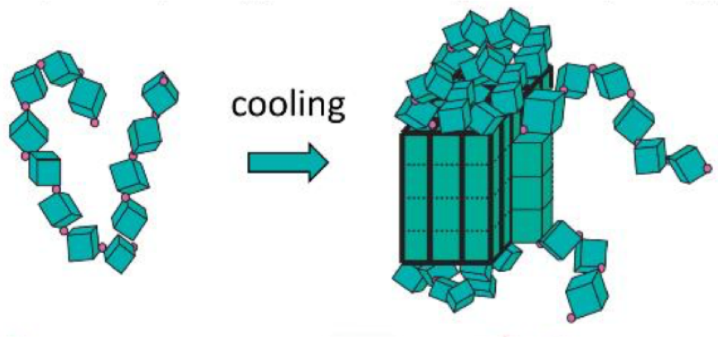
SAXS



Azimuthally Integrated Image

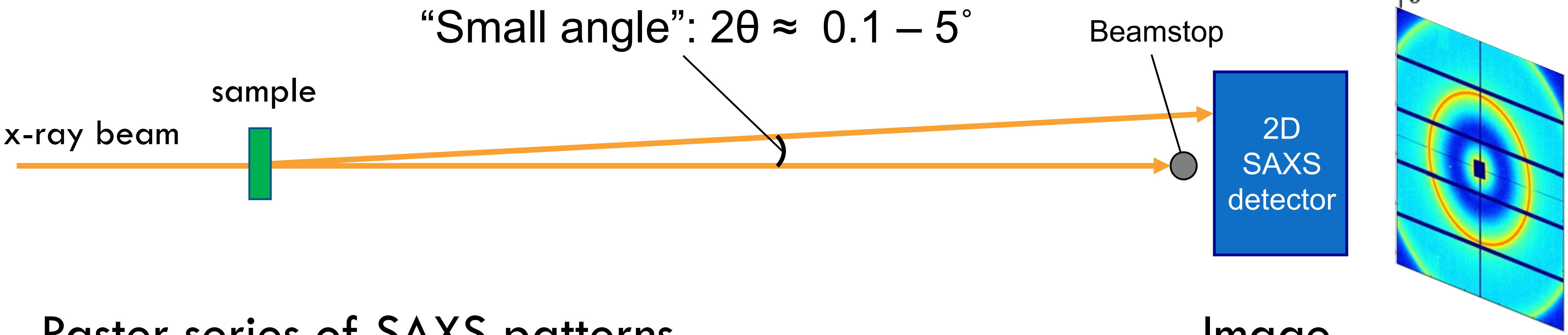


WAXS



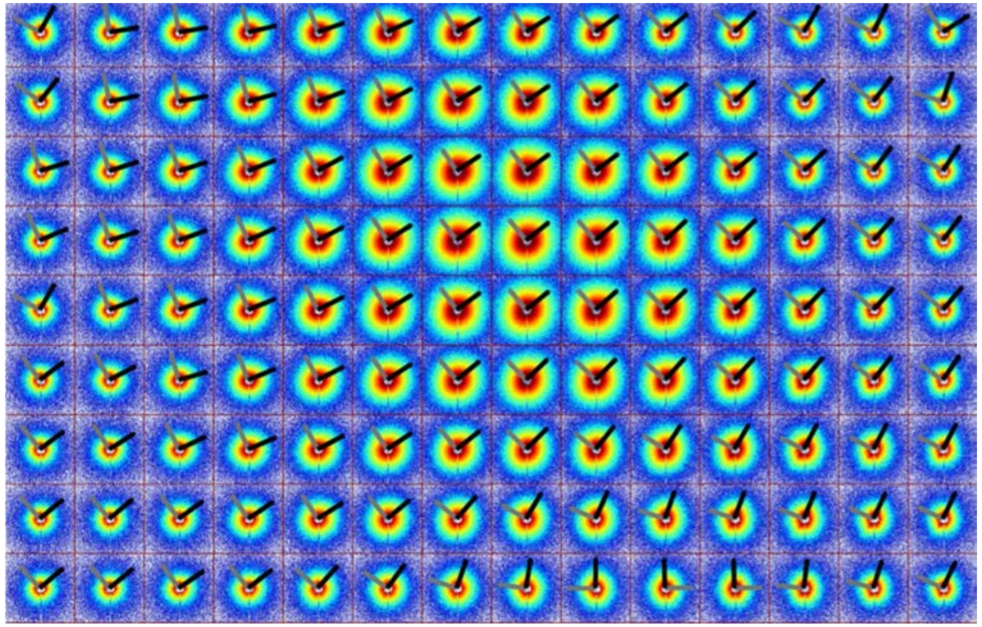
Scan-probe Imaging Example: SAXS

$$I(\vec{q}) \propto \left| \int \rho(\vec{r}) e^{-i\vec{q}\cdot\vec{r}} d\vec{r} \right|^2$$

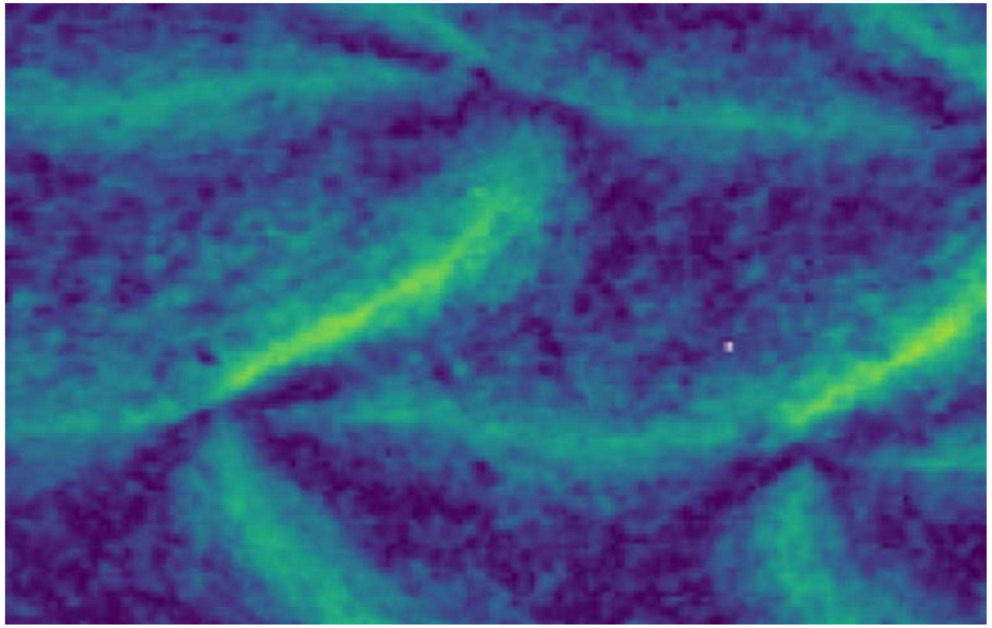


SAXS Pattern

Raster series of SAXS patterns



Image



More Examples of Full-field vs. Scan probe Imaging

Satellite View

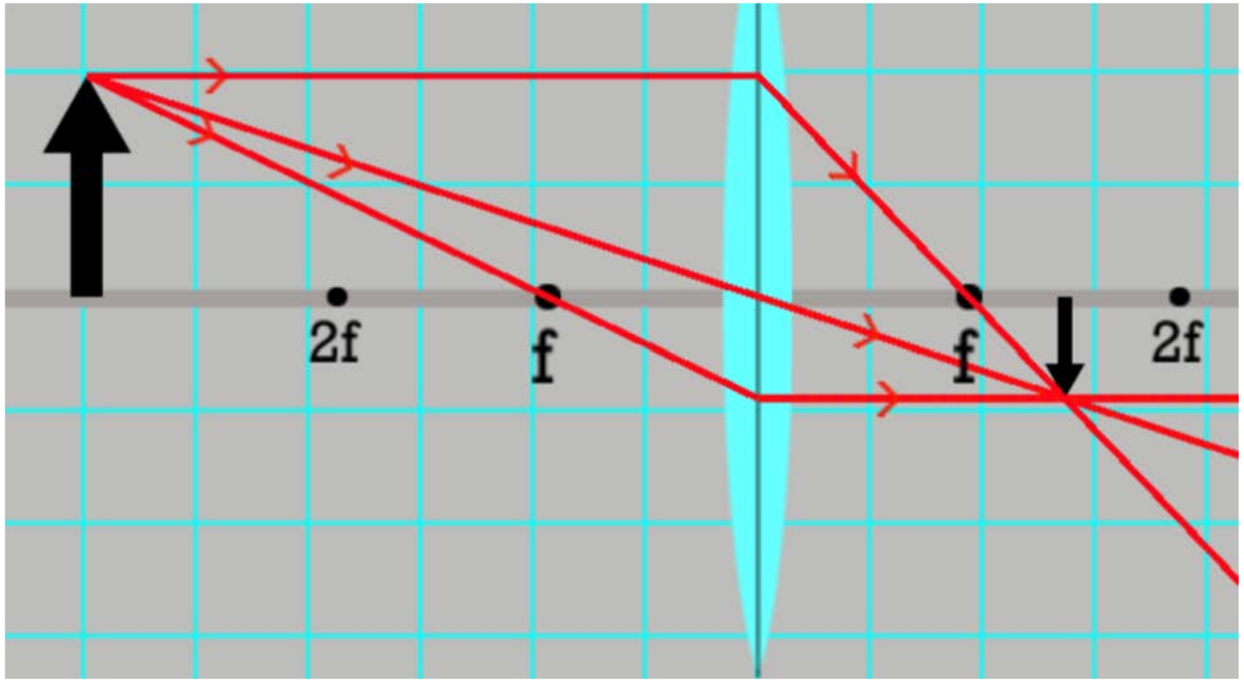
Street View

The Full-Field/Scan-probe distinction clarifies:

1. What contrast mechanisms are possible.
2. What determines the spatial resolution (e.g. incident beamsize or detector pixel size)?
3. How fast can I acquire an image / what is the frame rate?
4. How to optimize a beamline: Monochromator selection, Front-end and in-hutch focusing, etc.
5. Effect of source, optics, and detectors on performance
6. Comparisons to non-x-ray based microscopies.

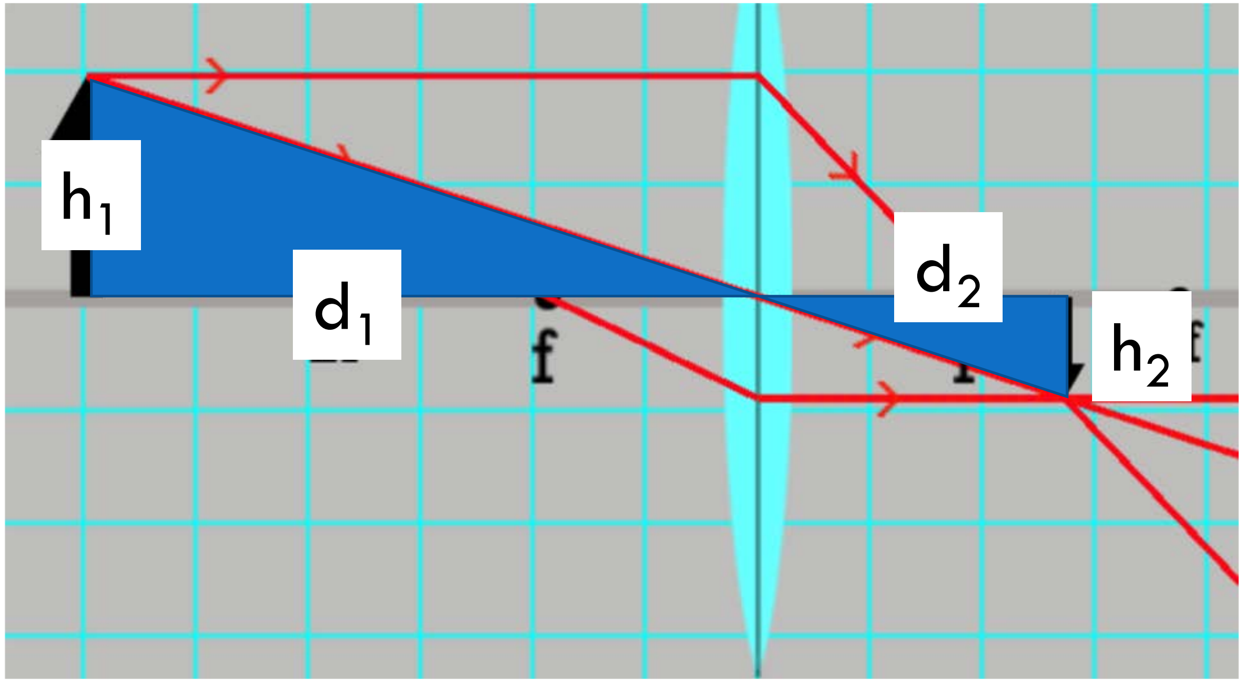
A note on focusing optics & geometry

Relationships between *beamline length*, *spot-size*, *lens focal distance*, and *space available at the sample*, for a single-lens system, are determined by a simple rules for magnification



A note on focusing optics & geometry

Relationships between *beamline length*, *spot-size*, *lens focal distance*, and *space available at the sample*, for a single-lens system, are determined by a simple rules for magnification



Magnification, h_1/h_2,
Equals the ratio of distances, d_1/d_2

Example:

- A typical CHESS source size is 1mm wide by 0.1 mm tall.
- If the total length of a beamline is 25 meters, and an available optic focuses this source 25 cm from the optic, what horizontal beam size can be achieved?

Answer: Magnification = 25 m / 25 cm = 100, so the HZ beamsize is ≥10 µm

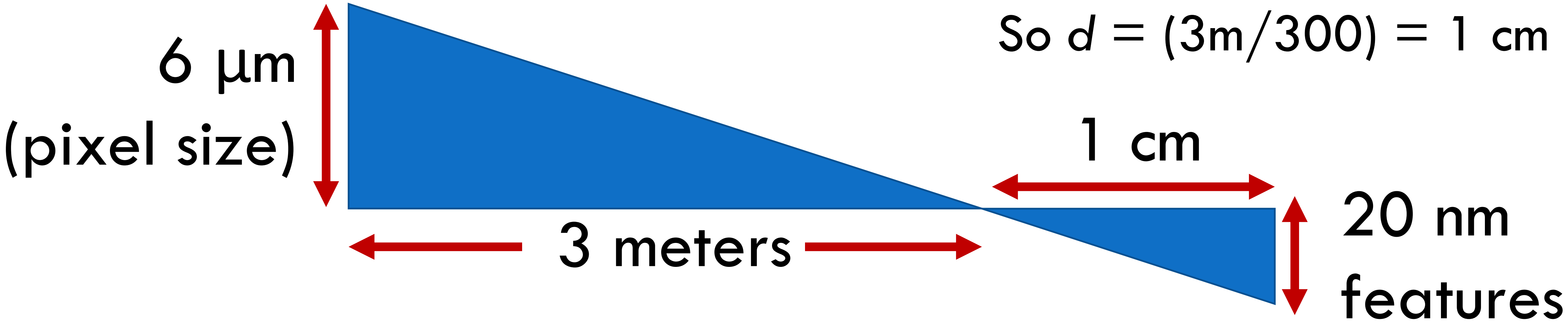
Focusing optics & geometry – 2nd example

- A (full-field) x-ray transmission microscope imaging beamline has:
  - a zone plate for its imaging optic
  - 3 meters between the sample and detector,
  - a detector with 6 µm pixels,
  - and can achieve 20 nm resolution.

How much space is available for the sample – i.e. what is the distance *d* between the sample and zone plate?

$$M = (6\ \mu m / 20\ nm) = 300;$$

So $d = (3m/300) = 1$ cm

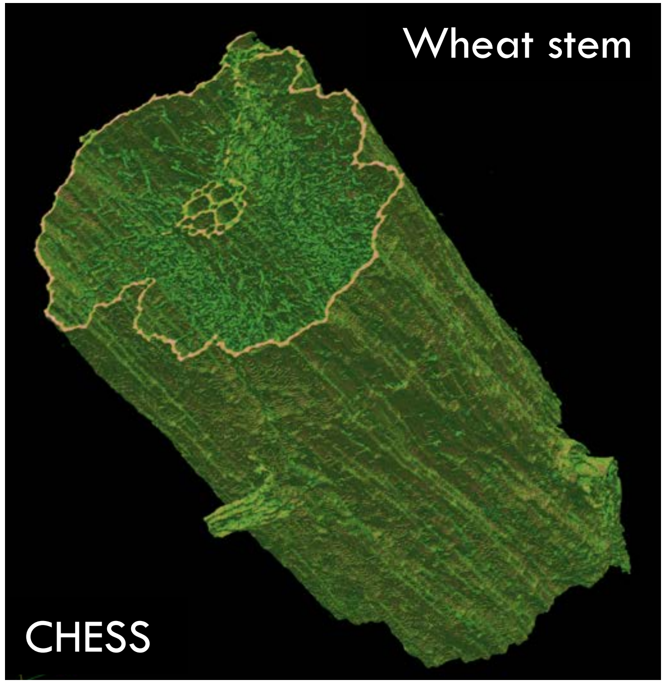


A note on computed tomography

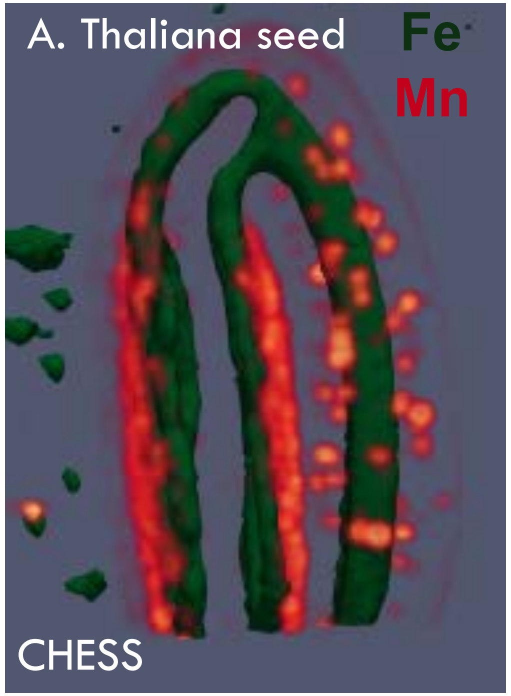
Computed Tomography (CT) refers to closely-related mathematical techniques to **convert a series 2D images** obtained at different sample angles **into a 3D reconstruction**.

With some caveats, CT can be applied to ***any such series***, regardless of how the images are formed -- for instance via full-field or scan-probe techniques.

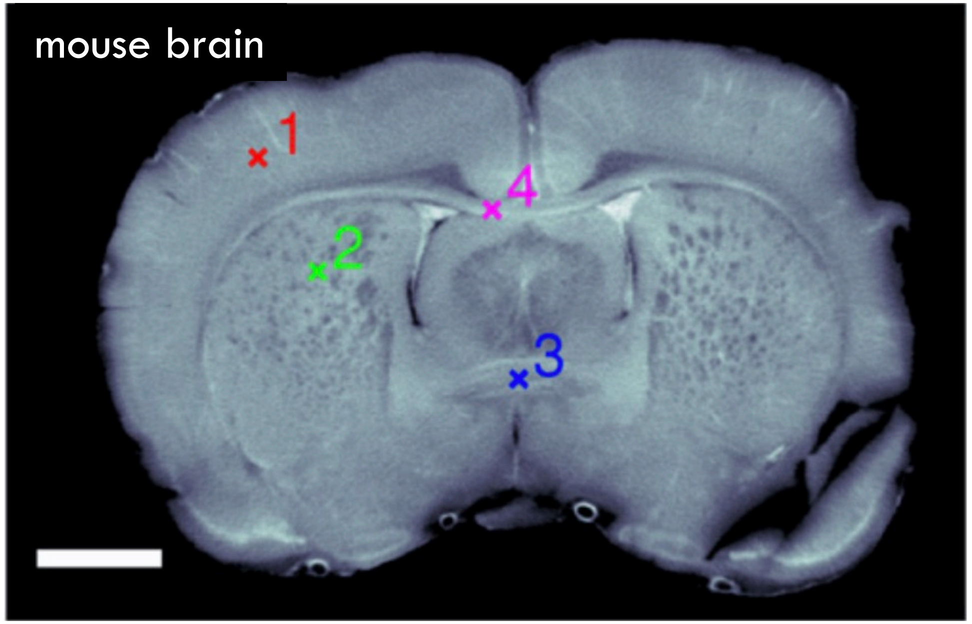
Phase-Contrast, Full-field CT



XRF, scan probe CT



SAXS, scan probe CT

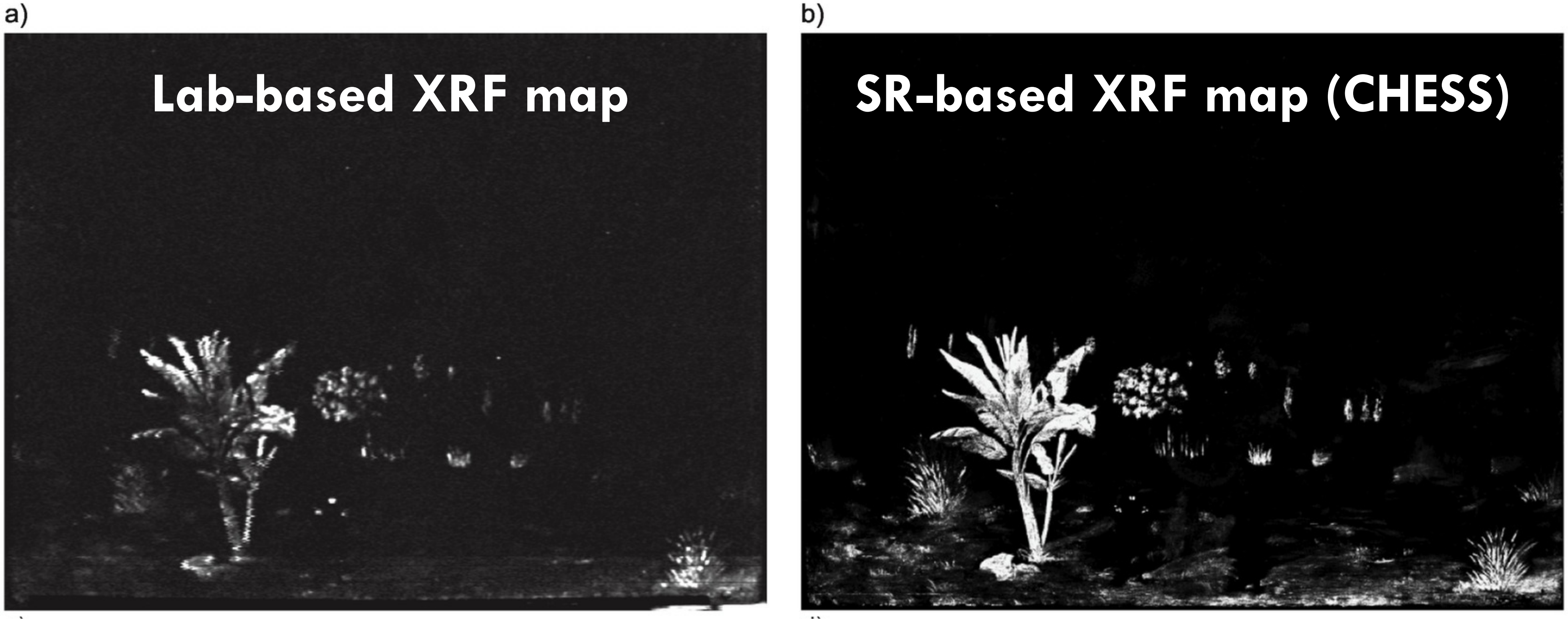


Torben H Jensen *et al* 2011 *Phys. Med. Biol.* **56** 1717

A note on image quality / signal to noise ratio

Regardless of how images are formed, a critical measure of their quality is signal-to-noise ratio, which determines the minimum level of contrast required for a feature to be observed.

Fig. 1. *Exit from the Theater*, attributed to Honoré Daumier (French, 1808–1879). 19th century, oil on panel. Unframed dimensions: 12 13/16 × 16 1/8 in. (32.6 × 41.0 cm). Nelson-Atkins Museum of Art 32-31.

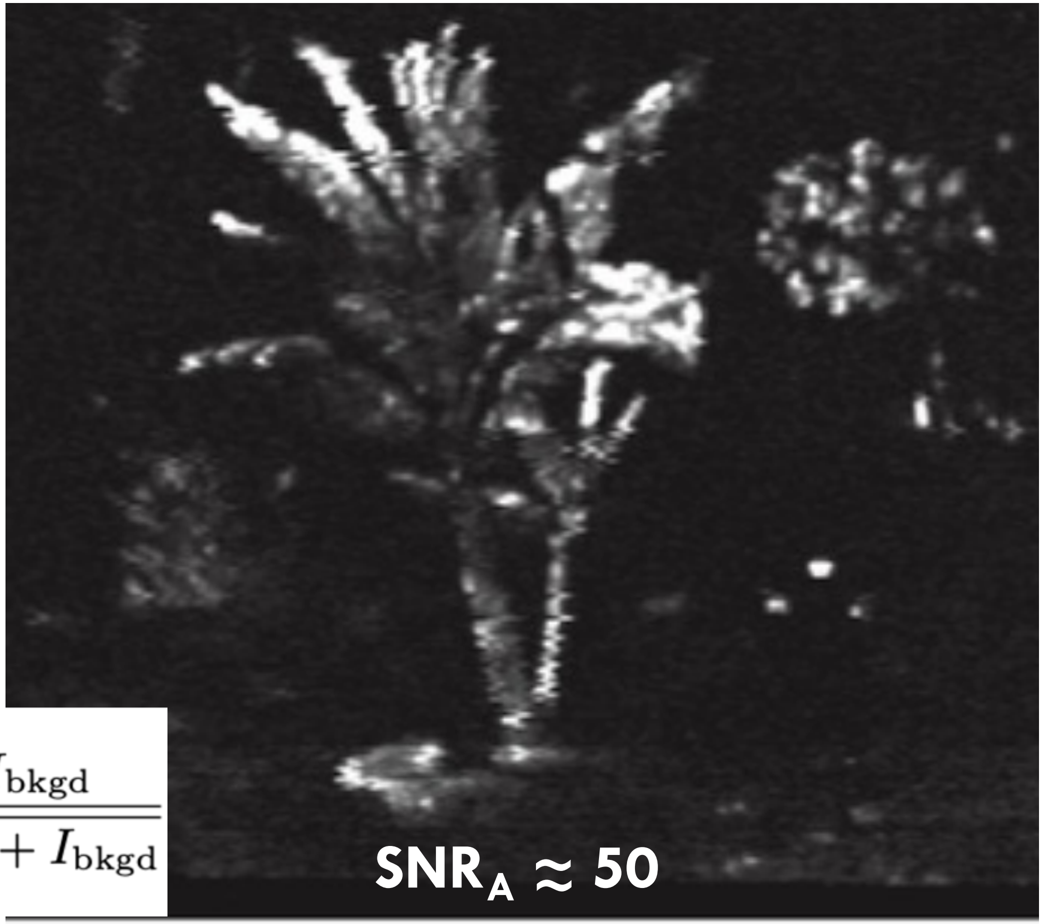


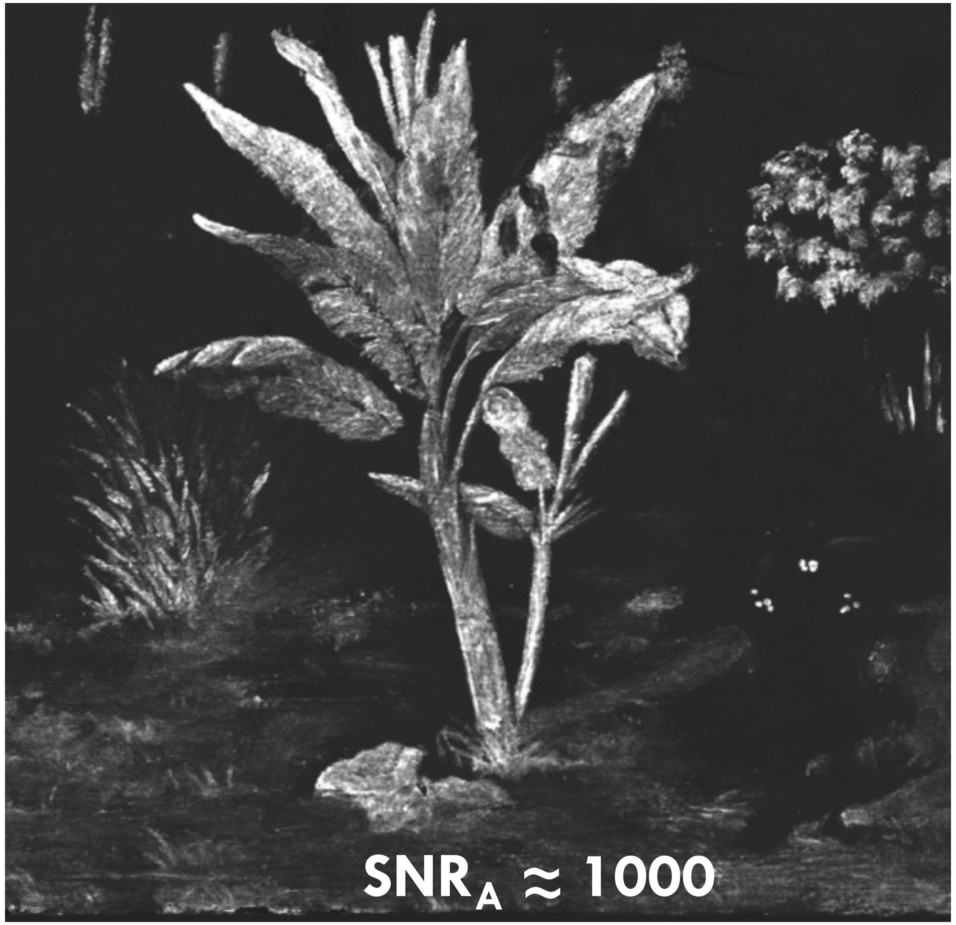
Smieska et al, 2019, DOI 10.1016/j.microc.2019.01.058

A note on image quality / signal to noise ratio

Regardless of how images are formed, a critical measure of their quality is signal-to-noise ratio, which determines the minimum level of contrast required for a feature to be observed.

Fig. 1. *Exit from the Theater*, attributed to Honoré Daumier (French, 1808–1879). 19th century, oil on panel. Unframed dimensions: 12 13/16 × 16 1/8 in. (32.6 × 41.0 cm). Nelson-Atkins Museum of Art 32-31.





$$\mathrm{SNR}_A = \frac{I_{\mathrm{max}} - I_{\mathrm{bkgd}}}{\sqrt{A}\sqrt{I_{\mathrm{max}} + I_{\mathrm{bkgd}}}}$$

Smieska et al, 2019, DOI 10.1016/j.microc.2019.01.058

Image Formation: ”Full-field” vs. “Scan-probe” Imaging

- **Full Field Imaging**
  - Resolution determined by detector(~1 µm) or lens (10 nm).
  - Frame Rate: **Hz to MHz**
  - Contrast: Absorption, Phase, Diffraction*, Compton*.

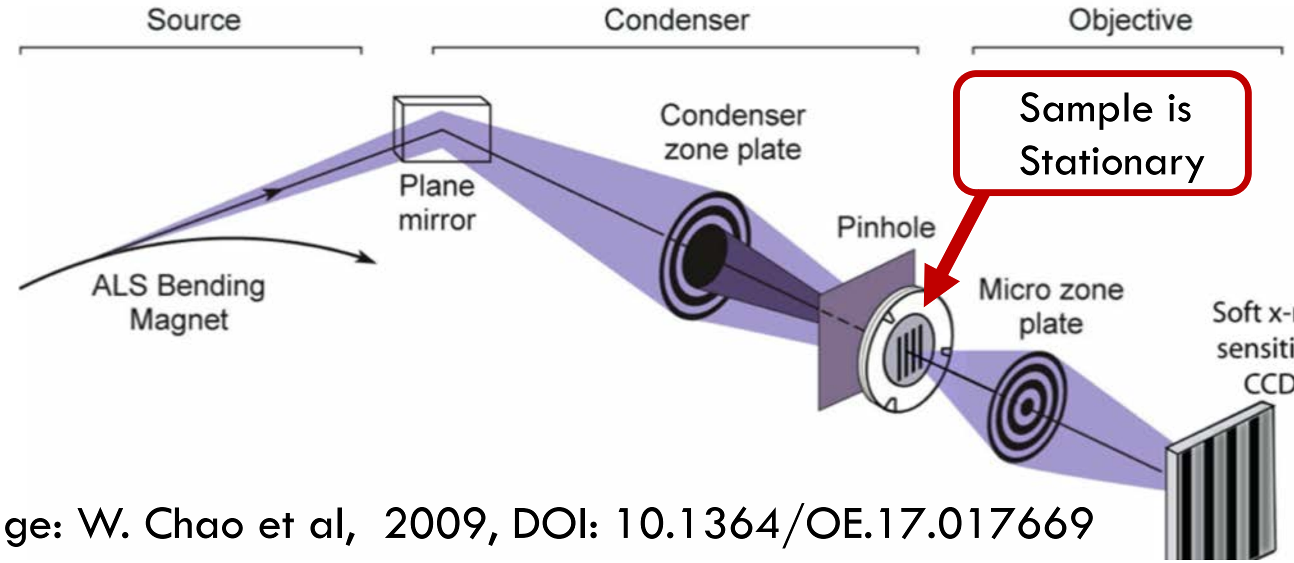


Image: W. Chao et al, 2009, DOI: 10.1364/OE.17.017669

- **Scan Probe Imaging**
  - Resolution determined by incident beam-size – reaching ~10 nm
  - Time/frame: **Minutes to hours.**
  - Contrast: Absorption, Phase, Diffraction, Compton, SAXS, fluorescence, XANES, EXAFS,...

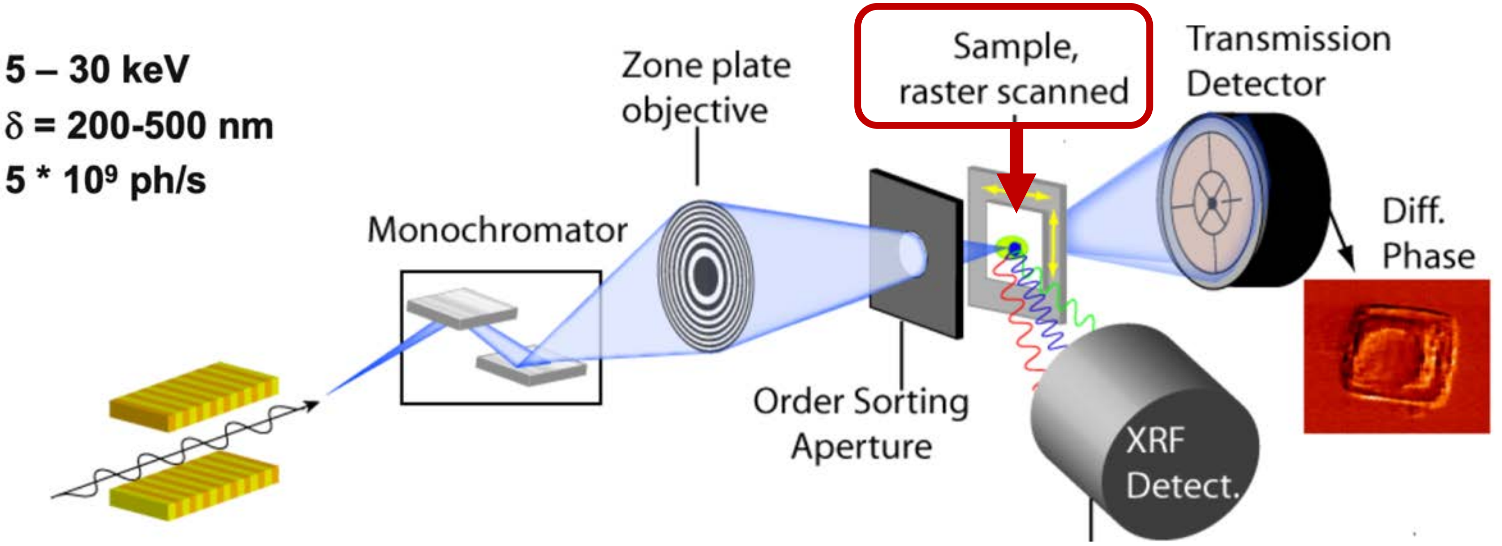
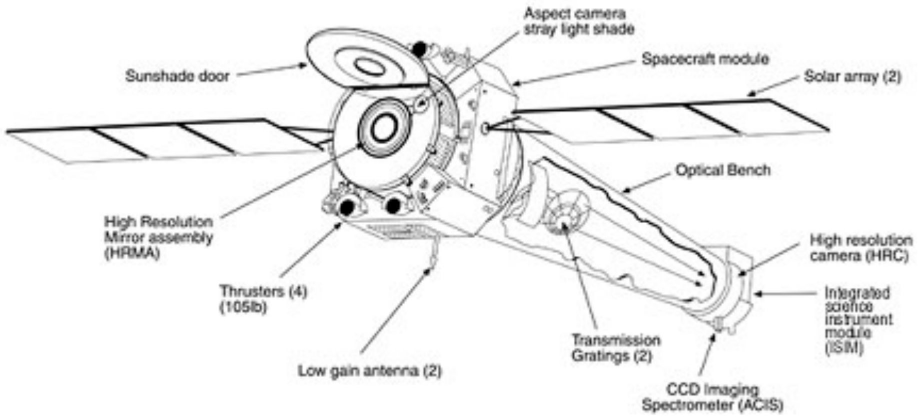


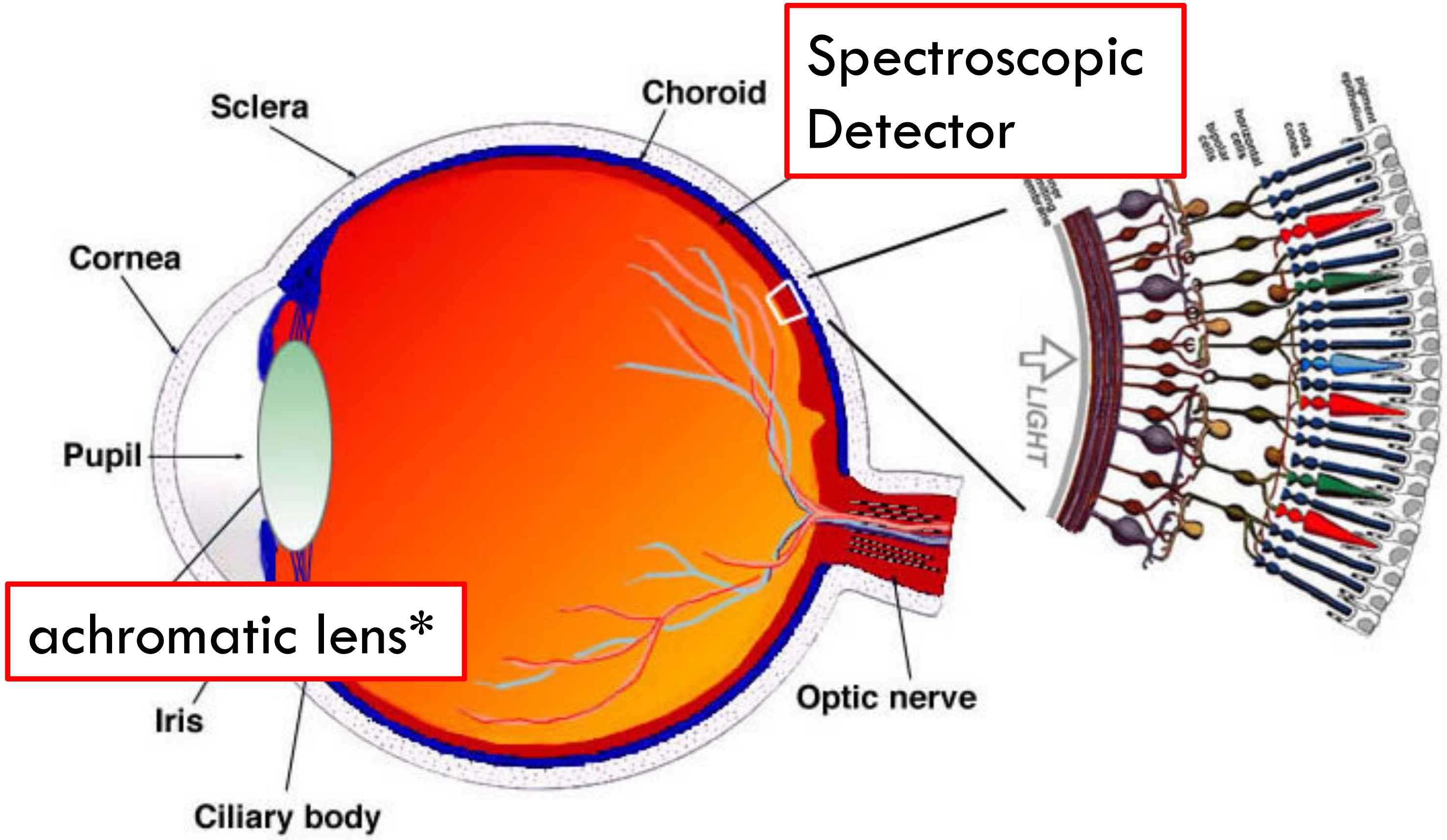
Image: Stephen Vogt (2016)

Can we combine the benefits of scan-probe with the speed of full-field?

Yes and no. Achromatic imaging lenses (esp. Wolter mirrors) and spectroscopic imaging detectors both exist for x-rays, but not with quite the right properties to make this a competitive approach…

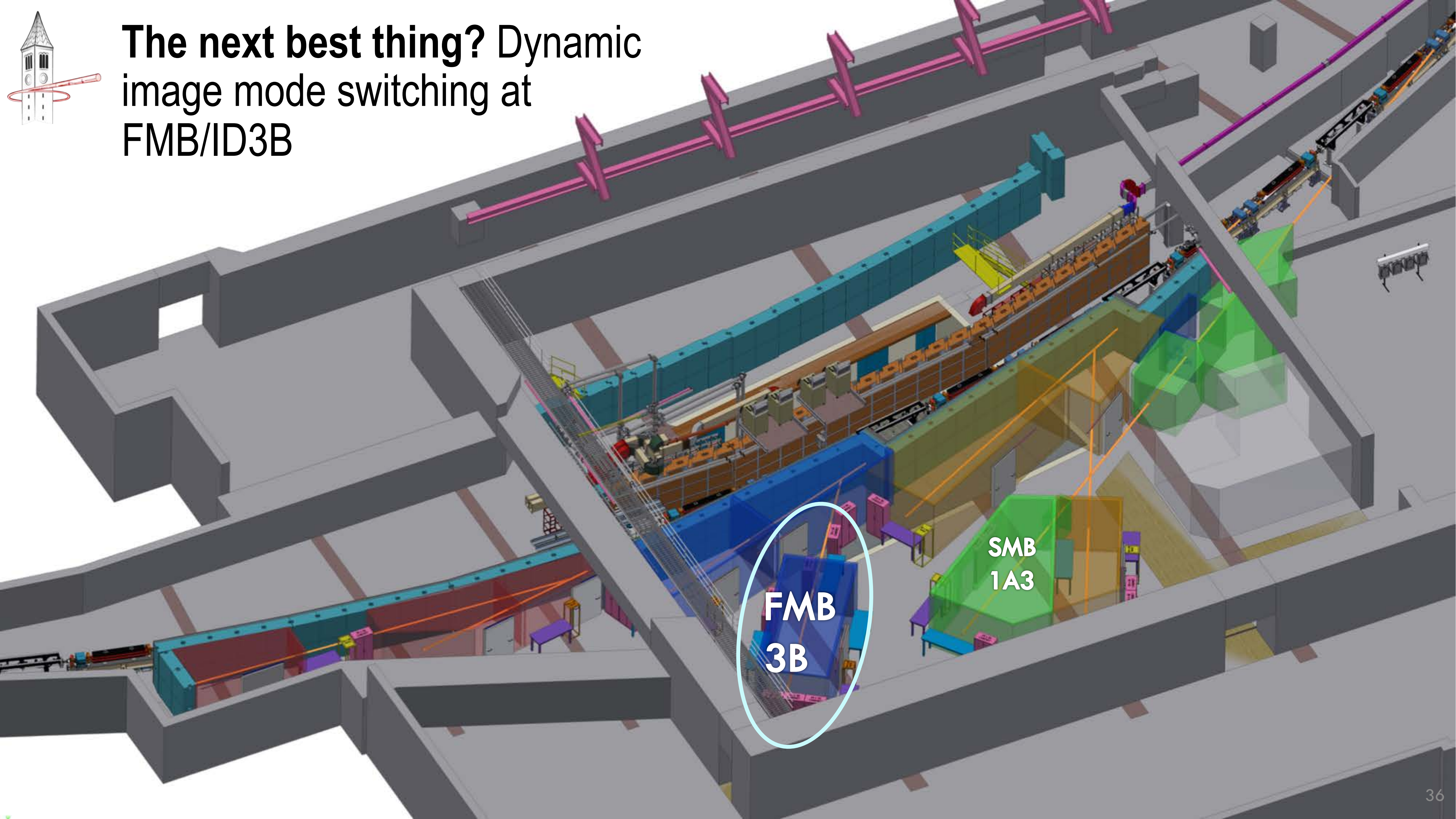


Chandra X-ray Observatory



Also, there are important but specialized methods (HEDM) that combine full-field imaging with diffraction…

The next best thing? Dynamic image mode switching at FMB/ID3B



BREAK

Up Next: Examples from the Functional Materials Beamline

https://imgs.xkcd.com/comics/coronavirus_polling.png

CHESS ID3B: Functional Materials Beamline

FMB provides four discrete energies for monochromatic X-ray experiments:

"SIDE BOUNCE" MONOCHROMATIC BEAM :

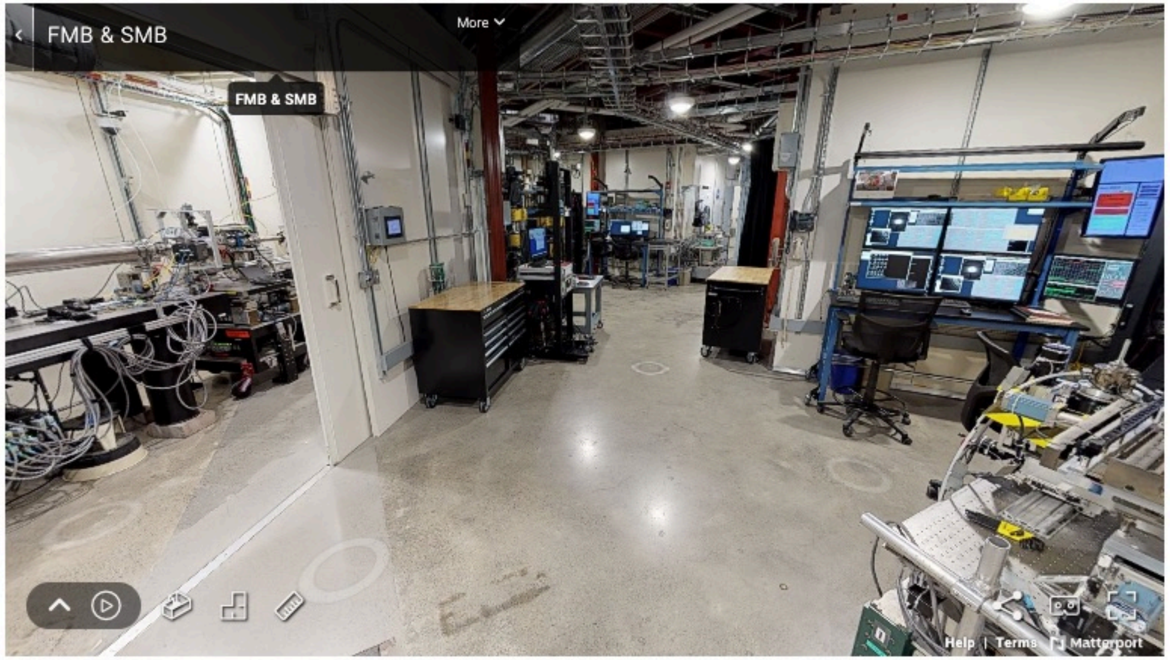
- Energy is selected by translating one of four diamond crystals into the beam path
  - Flux at all energies > 10^{12} ph/s/mm^2
- Mirror for harmonic rejection >25 keV
- Distances:
  - Undulator to side bounce mono: 16.8 m
  - Side bounce mono to upstream hutch wall: 7.4 m

Reflection	Energy
Diamond(111) – Bragg	9.73 keV
Diamond(220) – Laue	15.9 keV
Diamond(131) – Laue	18.65 keV
Diamond(004) – Laue	22.5 keV

CHESS ID3B: Functional Materials Beamline – Virtual Reality Tour

Link: https://www.chess.cornell.edu/partners/msn-c

MSN-C VIRTUAL TOUR:



MSN-C Functional Materials Beamline / ID3B

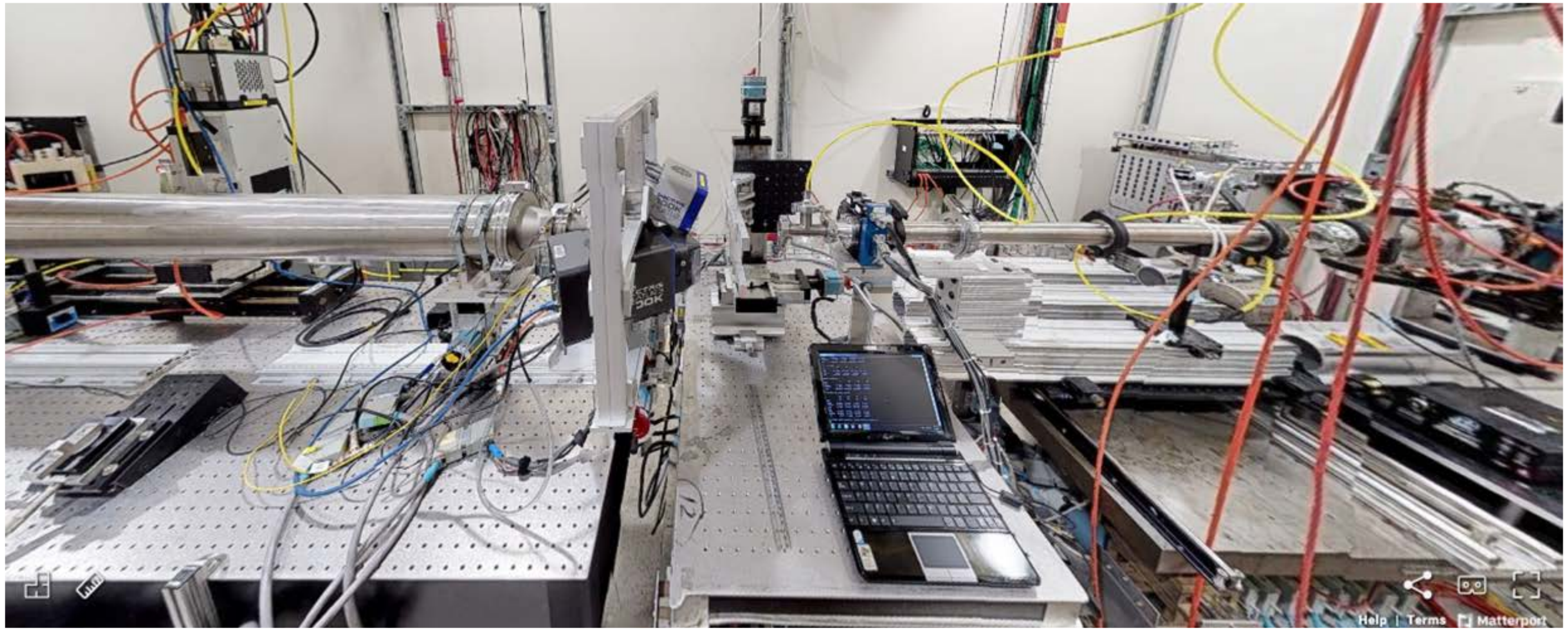
The Functional Materials Beamline is designed to provide **flexible, modular, micron-scale x-ray characterization** of *in-situ* materials processing and *ex-situ* samples specialized for soft materials and composites

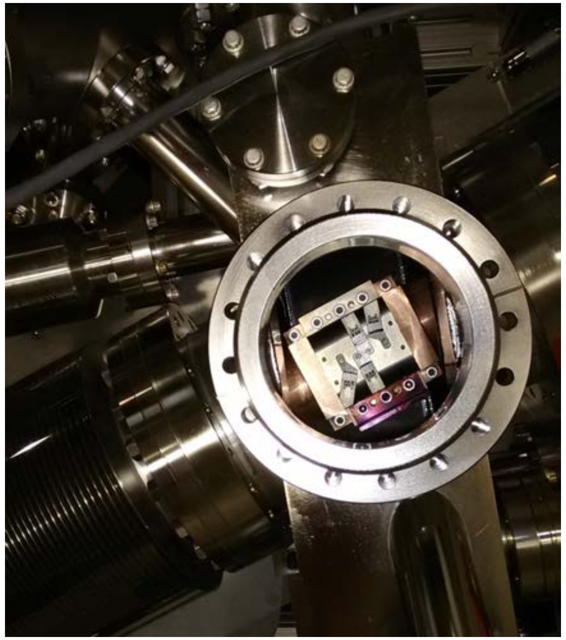
Detectors
Scattering or Imaging

Sample environment

Upstream optics
μ-Focusing
or
Full field

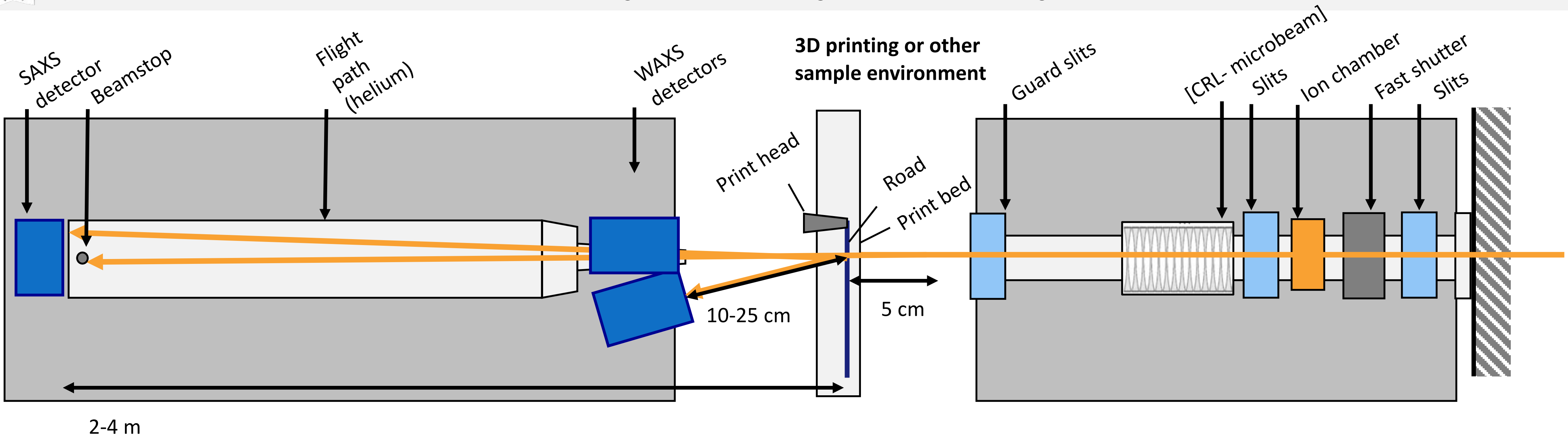
Side-bounce mono
4 discrete energies

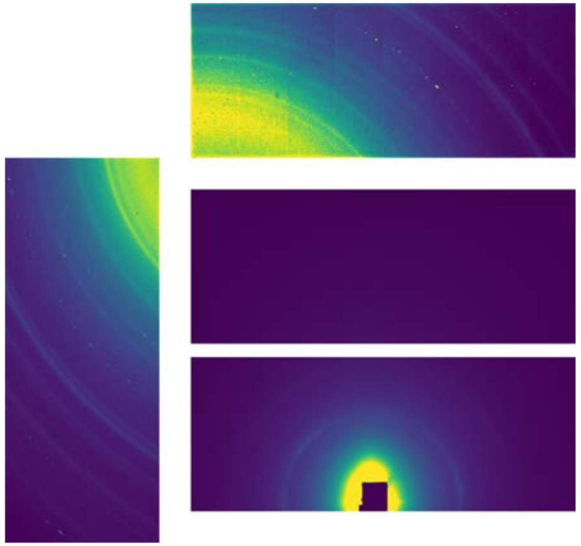




Core techniques: simultaneous SAXS/WAXS

Assess materials properties at multiple length scales during in-situ processing

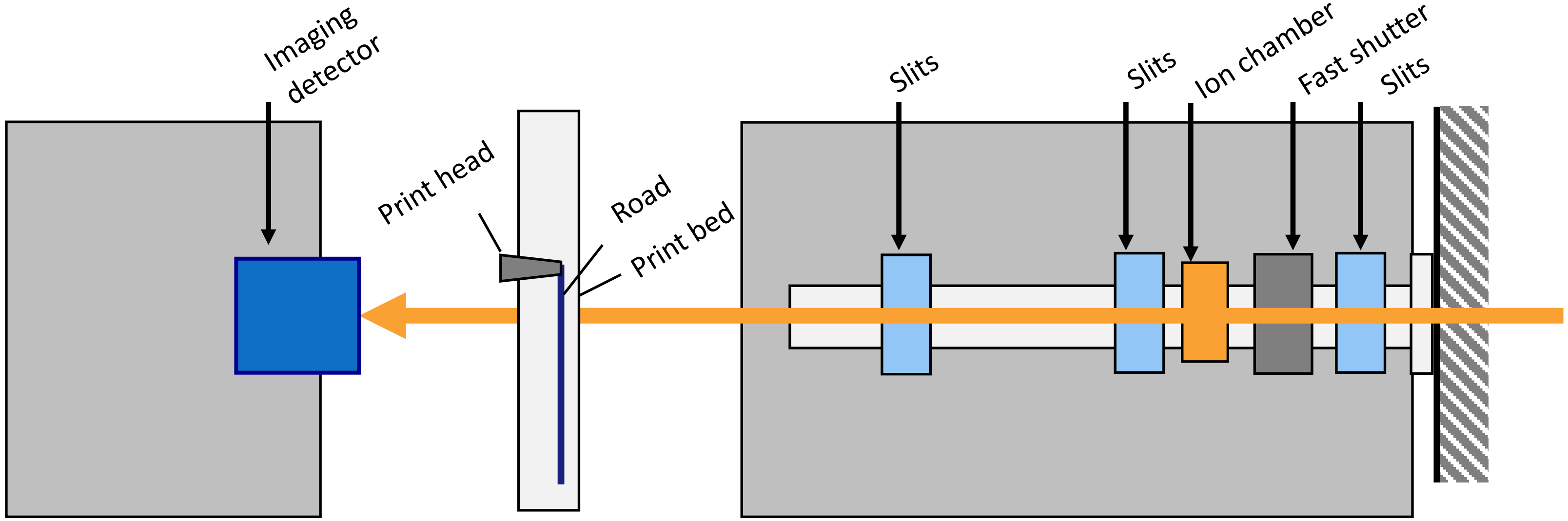


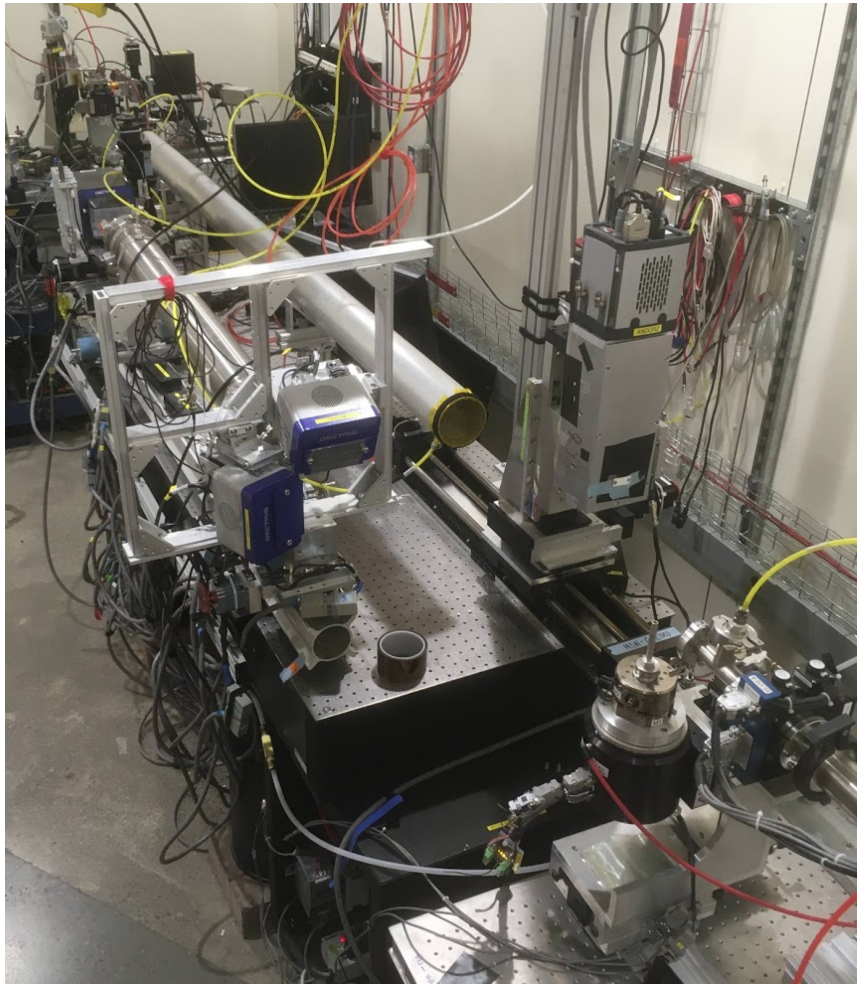


- Simultaneous SAXS/WAXS data collection
- 2D SAXS/WAXS mapping possible with or without focusing
- Sample environments: 3D printing, Linkam heating stage, diamond anvil cell

Core techniques: Absorption and phase contrast imaging

In-situ movies and ex-situ tomography



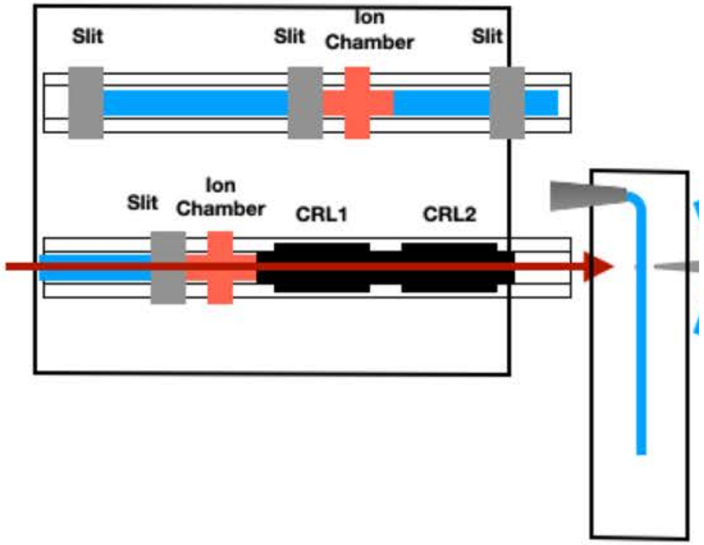


- Maximum frame rate ~50 Hz
- Maximum field of view roughly 2x2 cm

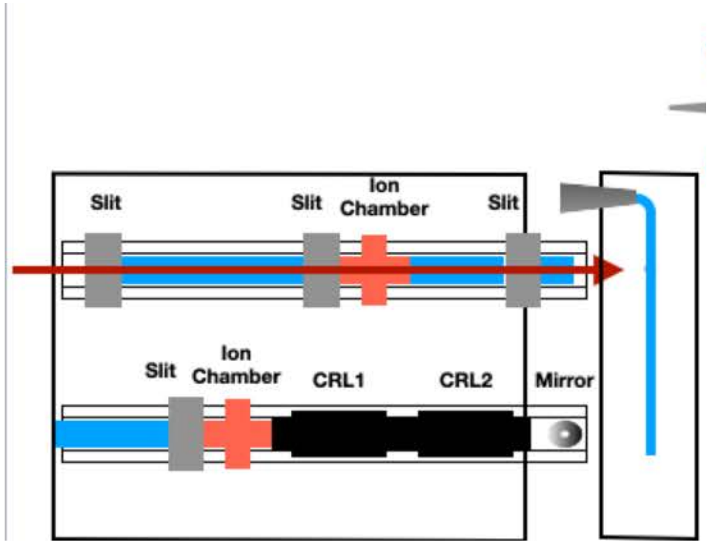
Freeze-dried wheat stem, 15.8 keV

FMB Summer 2020: Dynamic Switching b/w full-field and scan-probe

Scan-probe mode

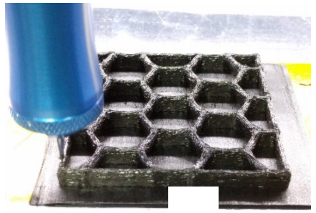


Full-Field mode



FMB highlights: Air Force Research Lab Focus on Composites

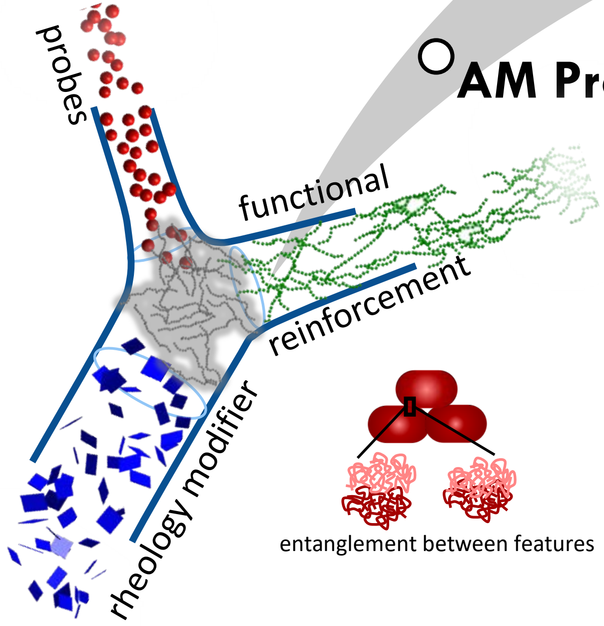
AFRL goal: thermoset additive manufacturing for aerospace structures and agile processing

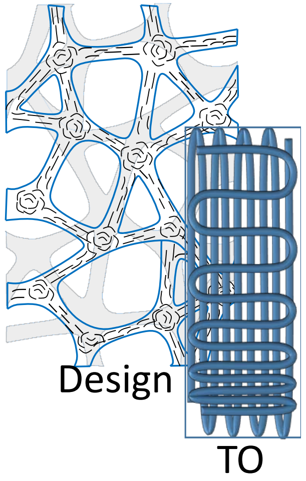


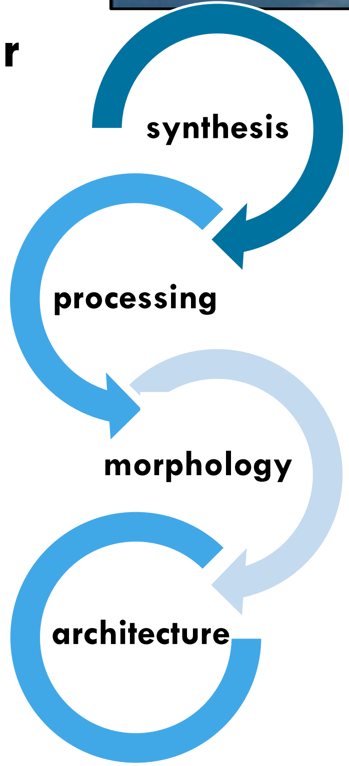
AM Processing

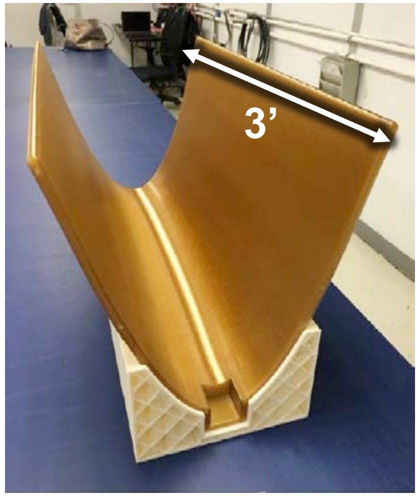
Complex Structures

Aerospace Tooling, Structures and Repair





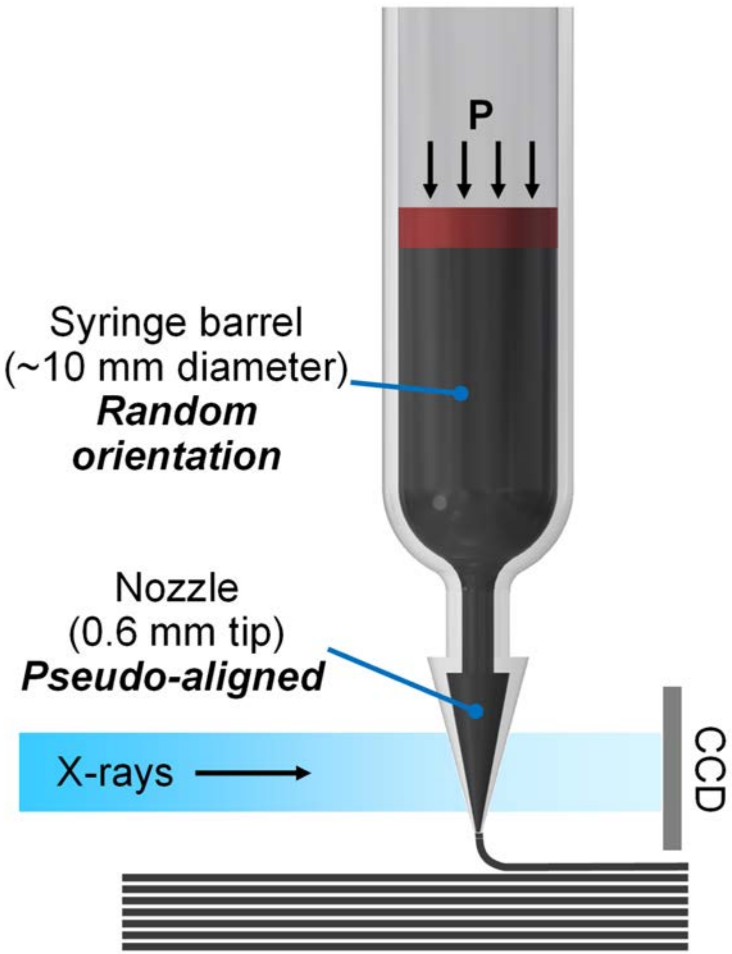


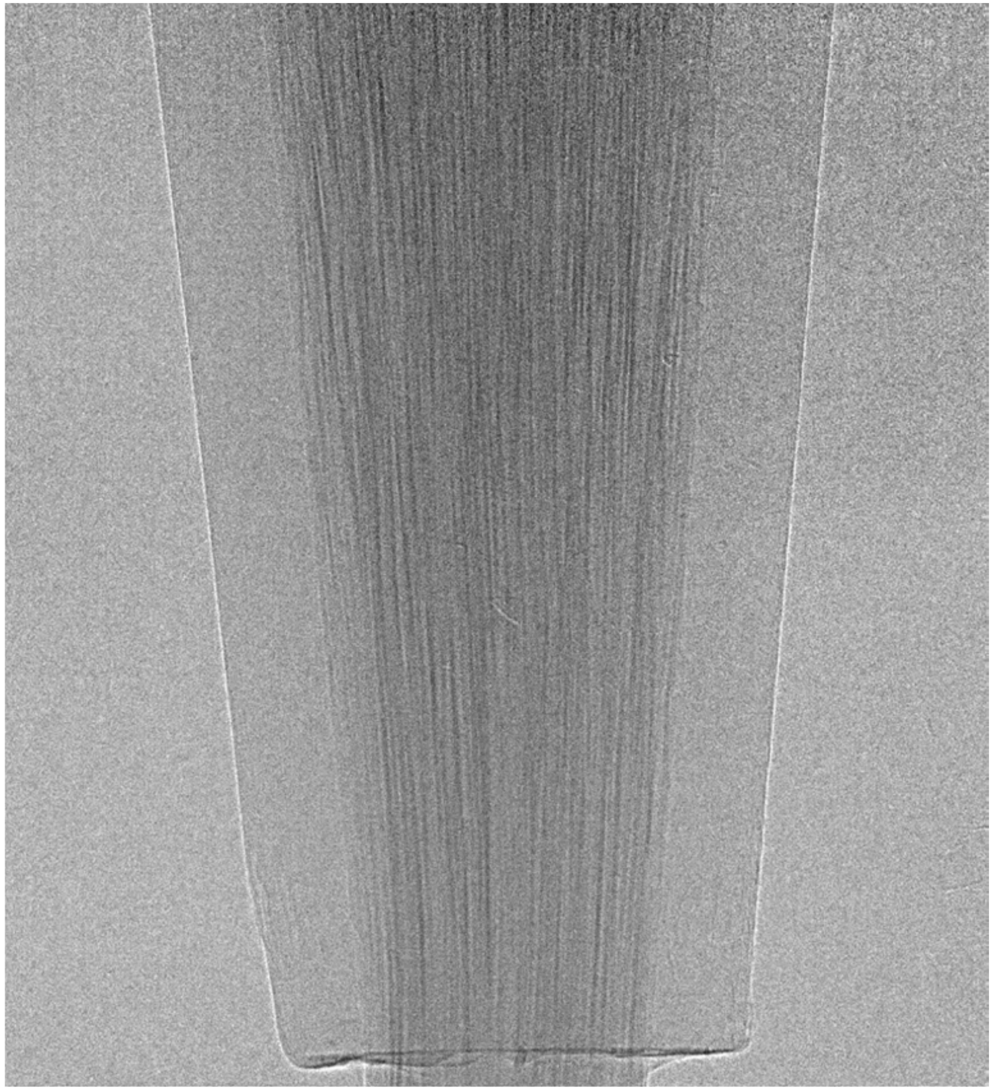


THE AIR FORCE RESEARCH LABORATORY
LEAD | DISCOVER | DEVELOP | DELIVER

Example: Full-Field Imaging

Measuring dynamics of fiber alignment during 3D printing of polymer-carbon fiber composites





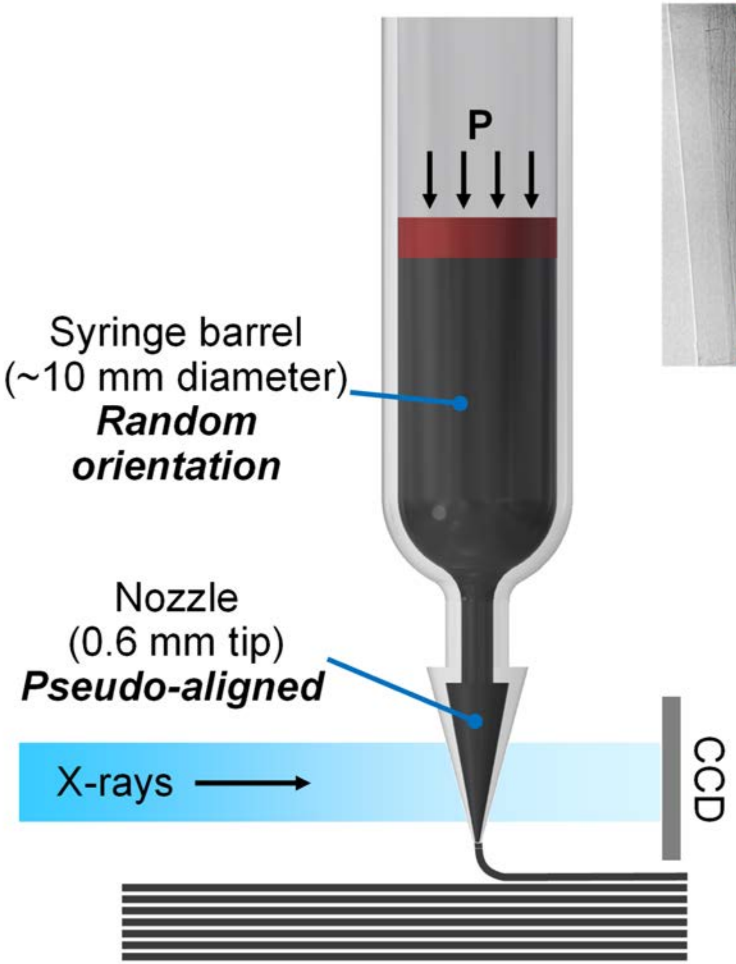
- Mechanical properties governed by alignment of fibers
- Quantify dynamics of fiber alignment as a function of velocity history, ink rheology and nozzle geometry.
- High X-ray flux at MSN-C enables imaging at 50 frames per second. Laboratory experiments are limited to 1 frame every 10 seconds
- First direct measurements of fiber alignment process.

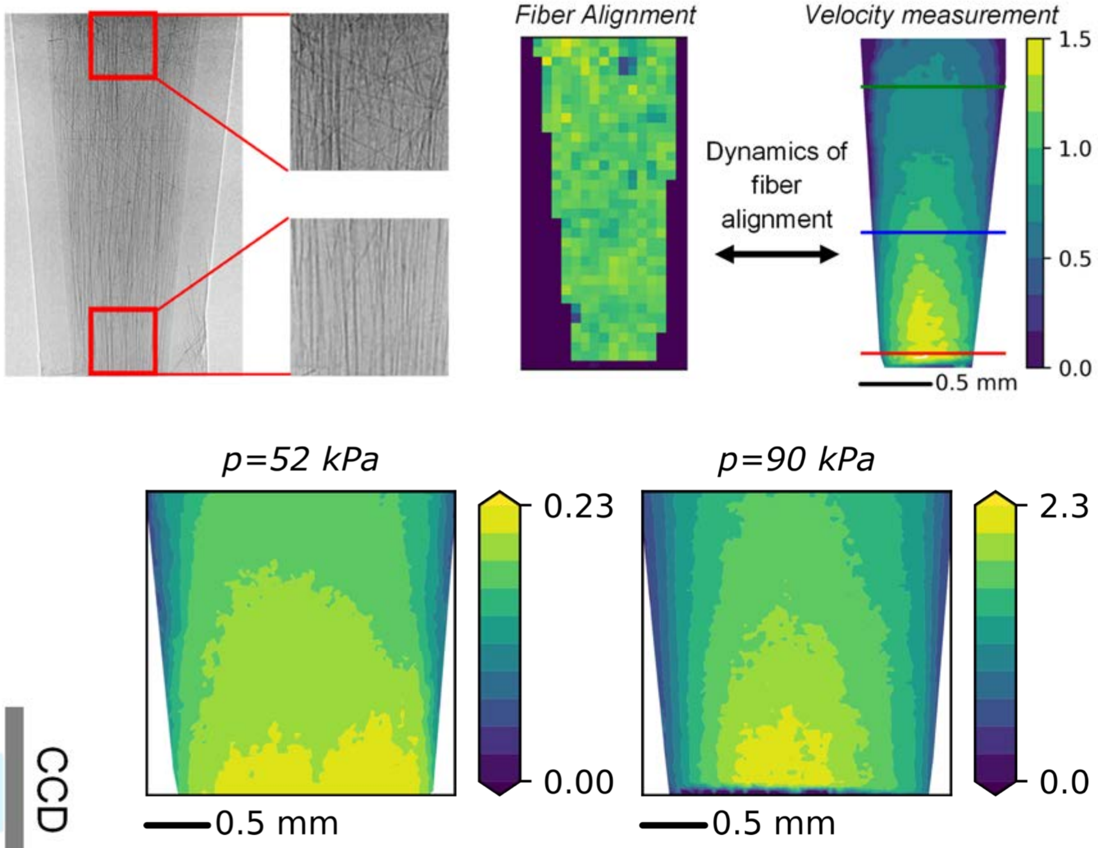
High-speed X-ray phase contrast. Local fiber alignment is related to the fluid velocity history, ink rheology and nozzle geometry.

Brendan Croom, AFRL, in preparation

Imaging highlight

Measuring dynamics of fiber alignment during 3D printing of polymer-carbon fiber composites





"Plug flow" to "pseudo-Newtonian" @ increasing velocity

- Mechanical properties governed by alignment of fibers
- Quantify dynamics of fiber alignment as a function of velocity history, ink rheology and nozzle geometry.
- High X-ray flux at MSN-C enables imaging at 50 frames per second. Laboratory experiments are limited to 1 frame every 10 seconds
- First direct measurements of fiber alignment process.

High-speed X-ray phase contrast. Local fiber alignment is related to the fluid velocity history, ink rheology and nozzle geometry.

Brendan Croom, AFRL, in preparation

Example: Full-Field Imaging (Simple Image Processing in Python)

Measuring dynamics of fiber alignment during 3D printing of polymer-carbon fiber composites

Flat-field correction tutorial

Developer: Brendan Croom (brendan.croom.ctr@us.af.mil)

Date: May 26, 2020

In this tutorial, we will go over the following:

- Loading and exploring HDF data using h5py
- Plotting radiographs using Matplotlib / Pyplot
- Flat-field correction of radiographs
- Conveting a series of images to a video (MP4 format)

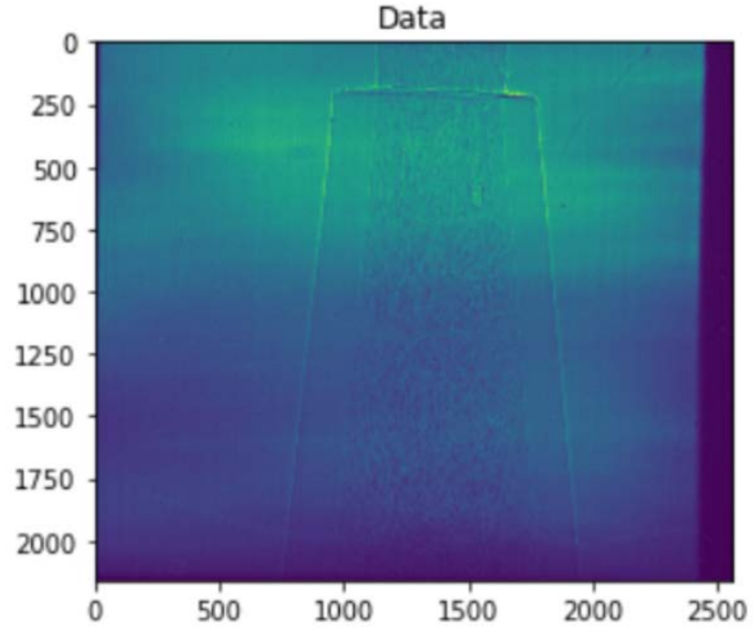
To begin, we need to load a few libraries and define where the data is saved.

In [1]:
```
# Load the appropriate libraries:
import os
from matplotlib import pyplot as plt
import numpy as np
import h5py
import h5py_radio_processing as radio

# Where is the data saved?
data_folder = 'xray_data'
dark_file = 'cf10to1_darks_ANDOR2_001_0000.hdf'  # contains a sequence of imag
es
white_file = 'cf10to1_whites_ANDOR2_001_0000.hdf'  # also contains a sequence
 of images
data_file = 'cf10to1_mosaic_p16.5_1_ANDOR2_001_0000.hdf'  # contains only a si
ngle image
```

Brendan Croom, AFRL, in preparation

Plot radiographs using Pyplot

In [4]:
```
data_image = hdf_data[DATA_LOCATION]

f, ax = plt.subplots()
ax.imshow(data_image)
ax.set_title('Data')
plt.show()
```



Revealing Filler Morphology in 3D-Printed Thermoset Nanocomposites by Scanning Microbeam SAXS and WAXS

Edward Trigg, Hilmar Koerner - *Air Force Research Laboratory*

Louisa Smieska, Arthur Woll - *Cornell High Energy Synchrotron Source*

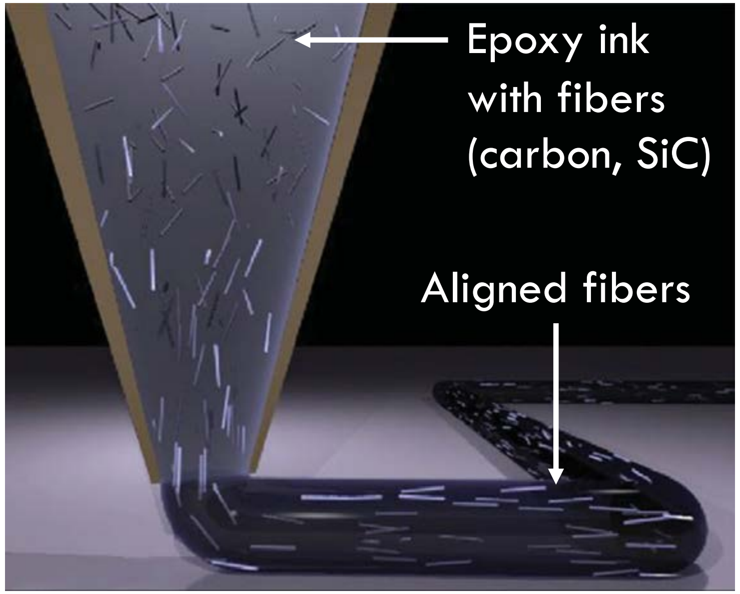
Nadim Hmeidat, Brett Compton - *Univ. of Tennessee, Knoxville*

April 24, 2020

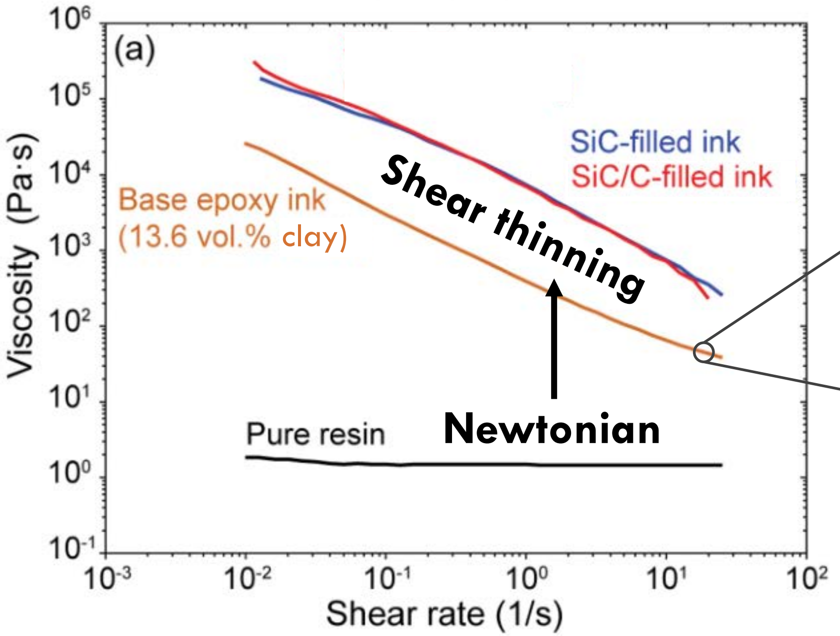
Submitted, 04/17/2020

Promising method for printing of epoxy + reinforcing filler developed in 2014

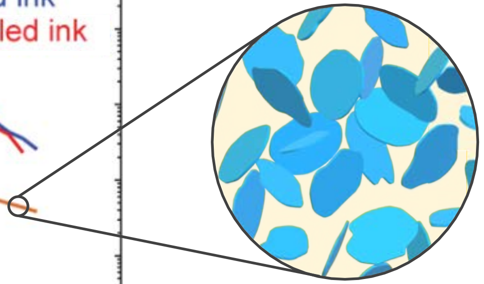
3D printing concept



Addition of nanoclay makes epoxy "printable"



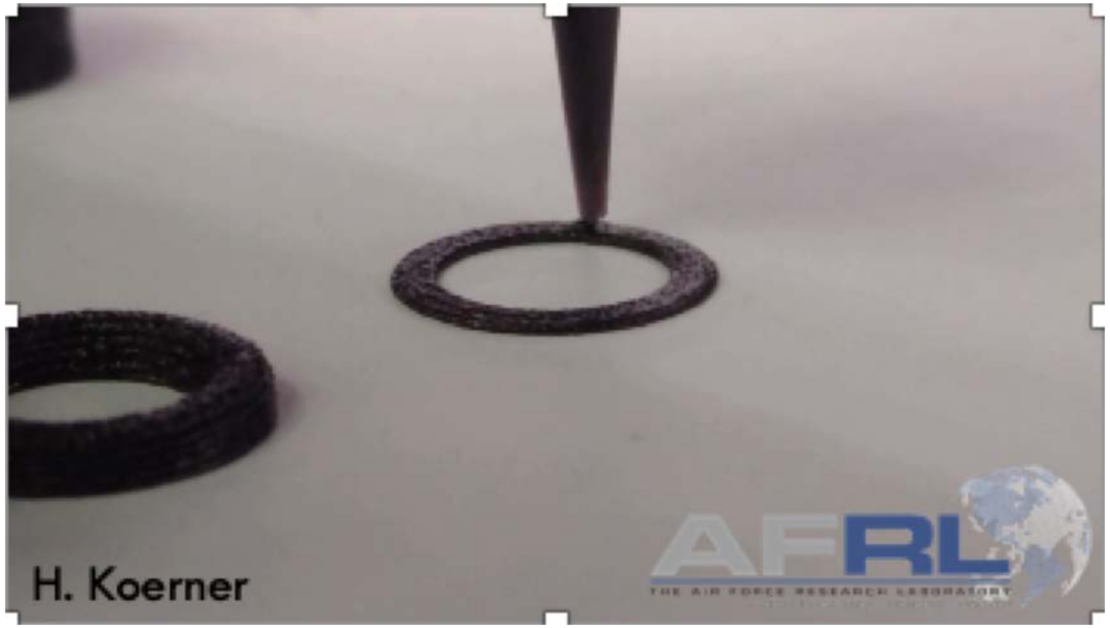
Nanoclay platelets



- Print "unmanufacturable" structures
- Parts show high modulus and strength
- Tunable fiber alignment – tunable mechanical properties

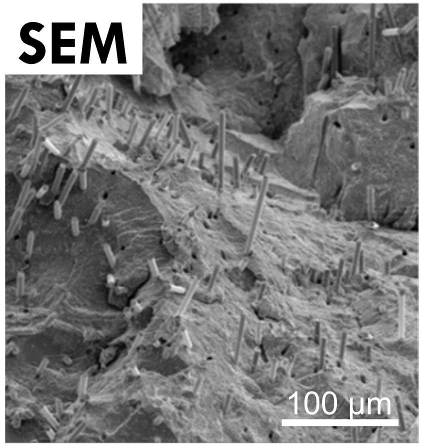
Compton, B. G.; Lewis, J. A. *Adv. Mater.* **2014**, *26* (34), 5930.

Increasing interest, but insufficient characterization to date

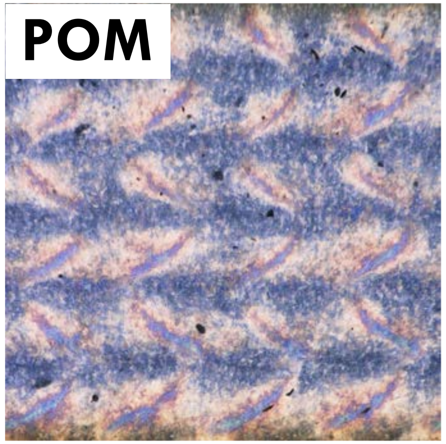


Recent work on this system:

(1) Johnson et al. *Langmuir* **2019**, 35, 8758–8768.
(2) Pierson et al. *Exp. Mech.* **2019**, 59 (6), 843–857.
(3) Hmeidat et al. *Compos. Sci. Technol.* **2018**, 160, 9–20.
(4) Hmeidat et al. *Submitted* **2020**.
(5) Abbott et al. In *SAMPE Conference and Exhibition*, **2019**, Charlotte, NC.



AFRL, unpublished results



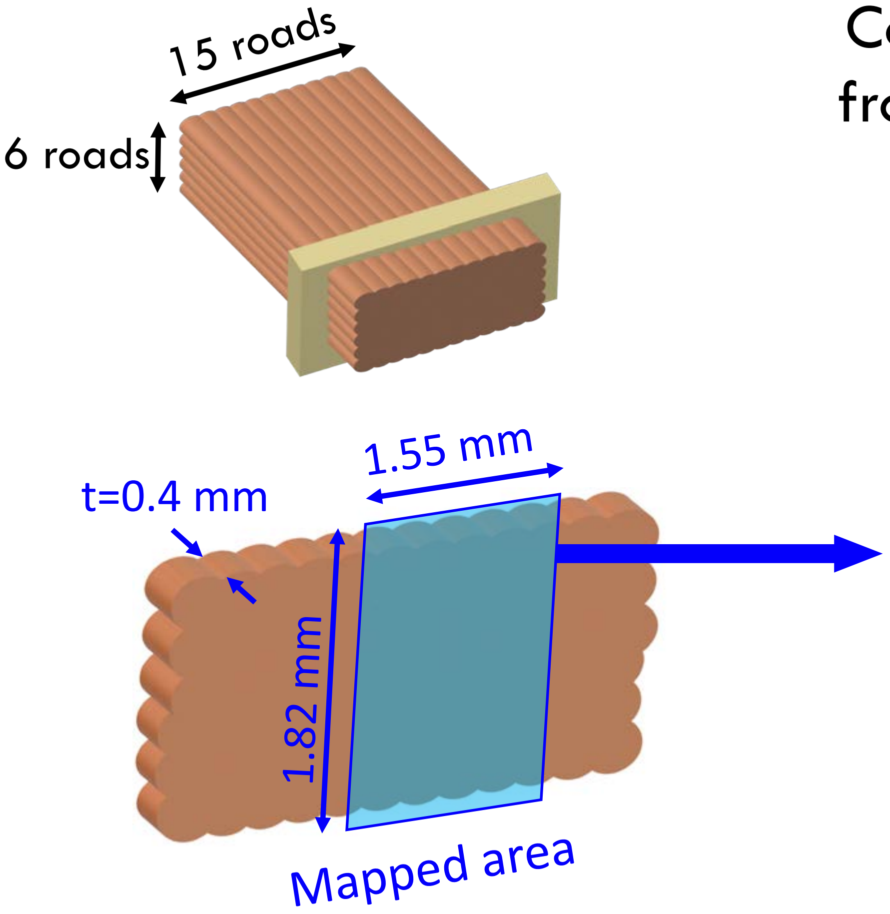
This work

<u>Our approach</u>:
Scanning microbeam SAXS/WAXS on cross-sections

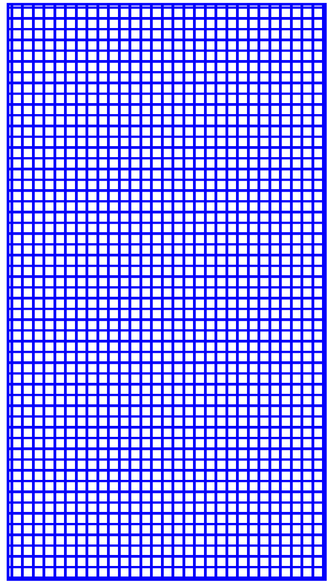
Experiments

Composition	Sample 1	Sample 2
Epoxy resin [vol. %]	92.5	91.8
Nanoclay [vol. %]	7.5	7.5
Carbon fiber [vol. %]	0	0.7

Cross-section cut from printed sample, and polished

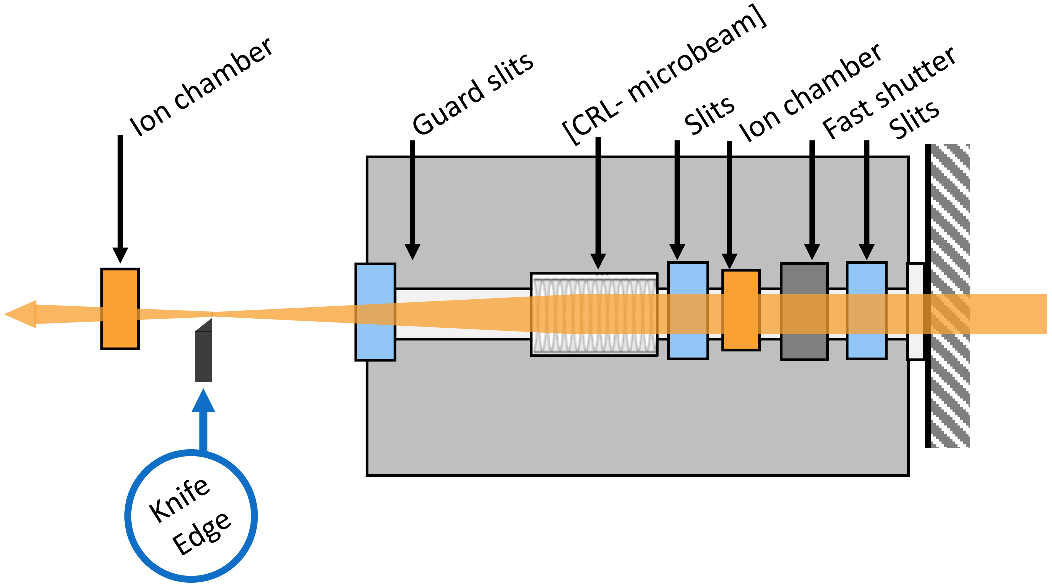


Collect SAXS+WAXS from every point on a 5 x 5 µm grid



225,000 tiffs per sample

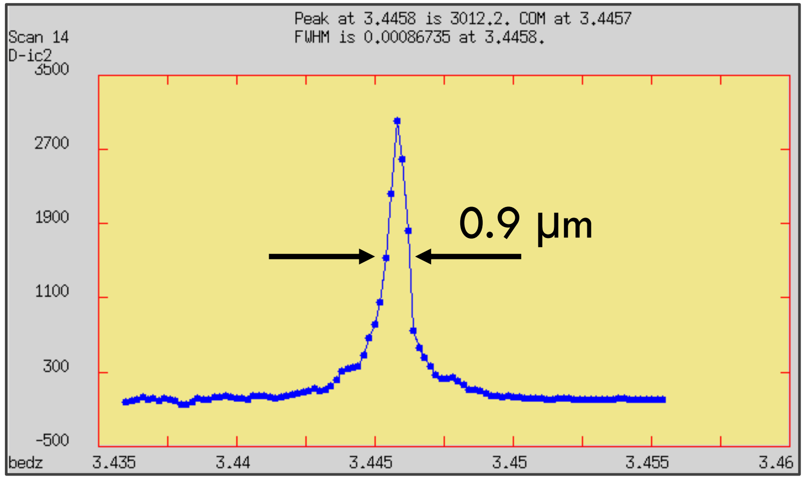
Characterization of CRL focusing



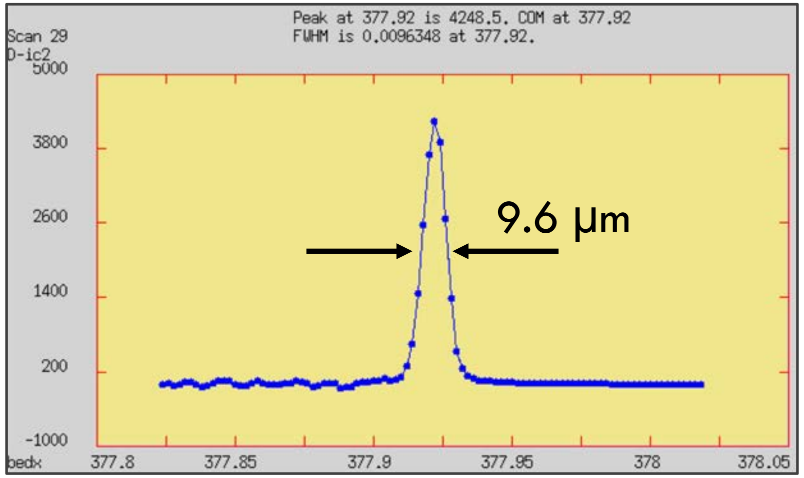
Characteristics:

X-ray energy	9.7366 keV
Secondary source aperture (s0h)	0.3 mm
Photons/sec	7.5×10^9
VT	1.1 µm
HZ	9.6 µm

Vertical beam size:

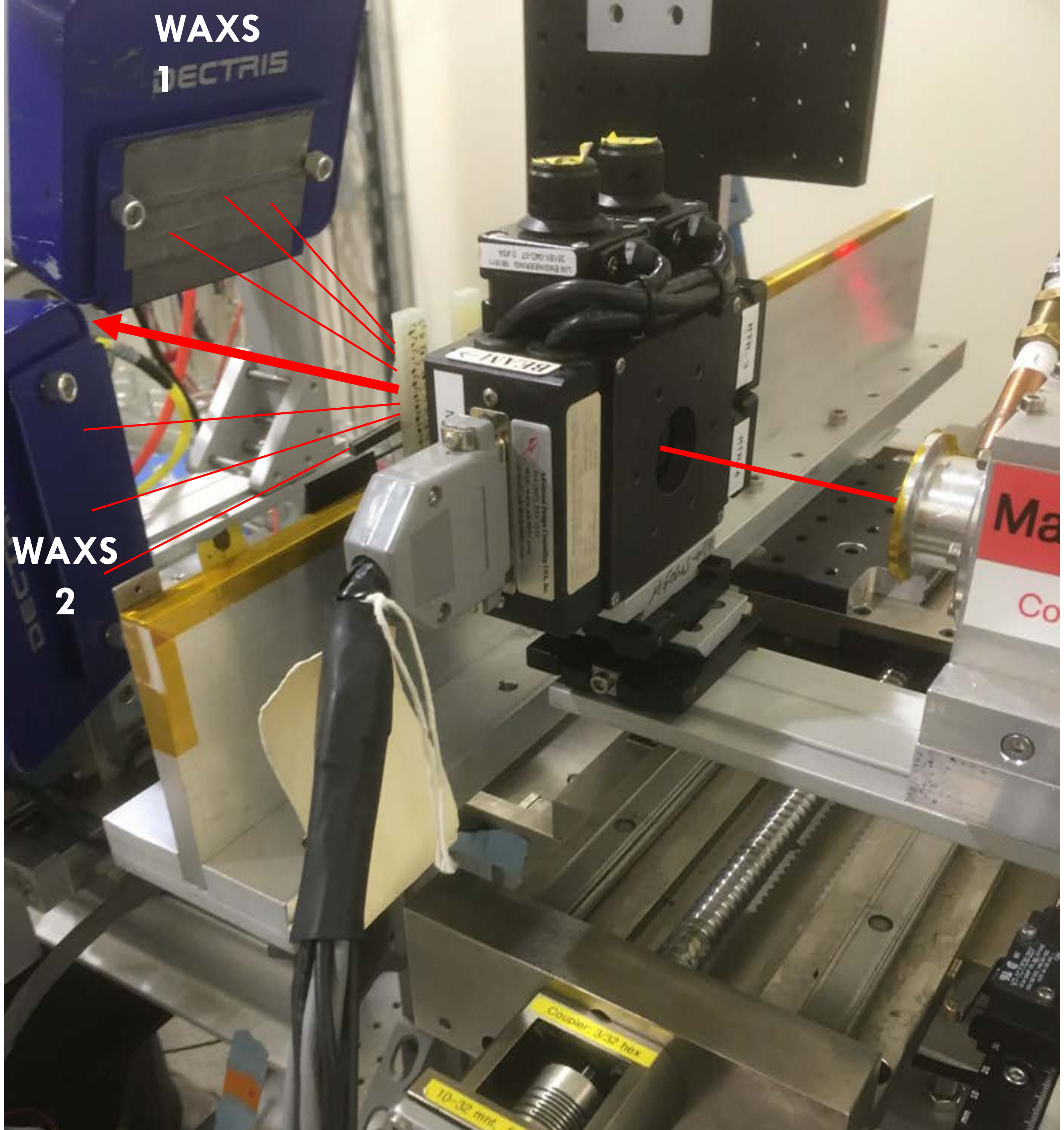


Horizontal beam size:

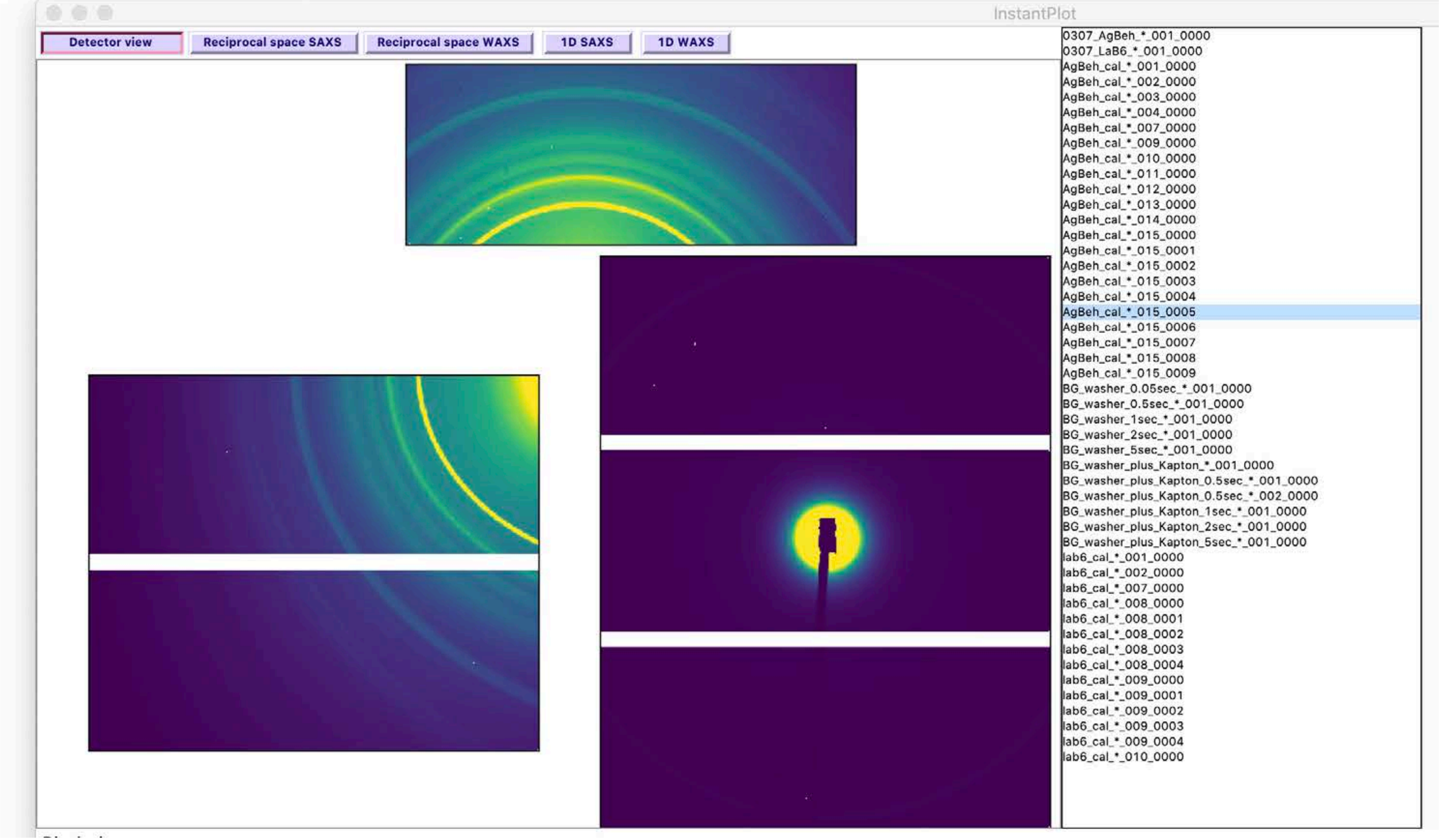


Detector setup

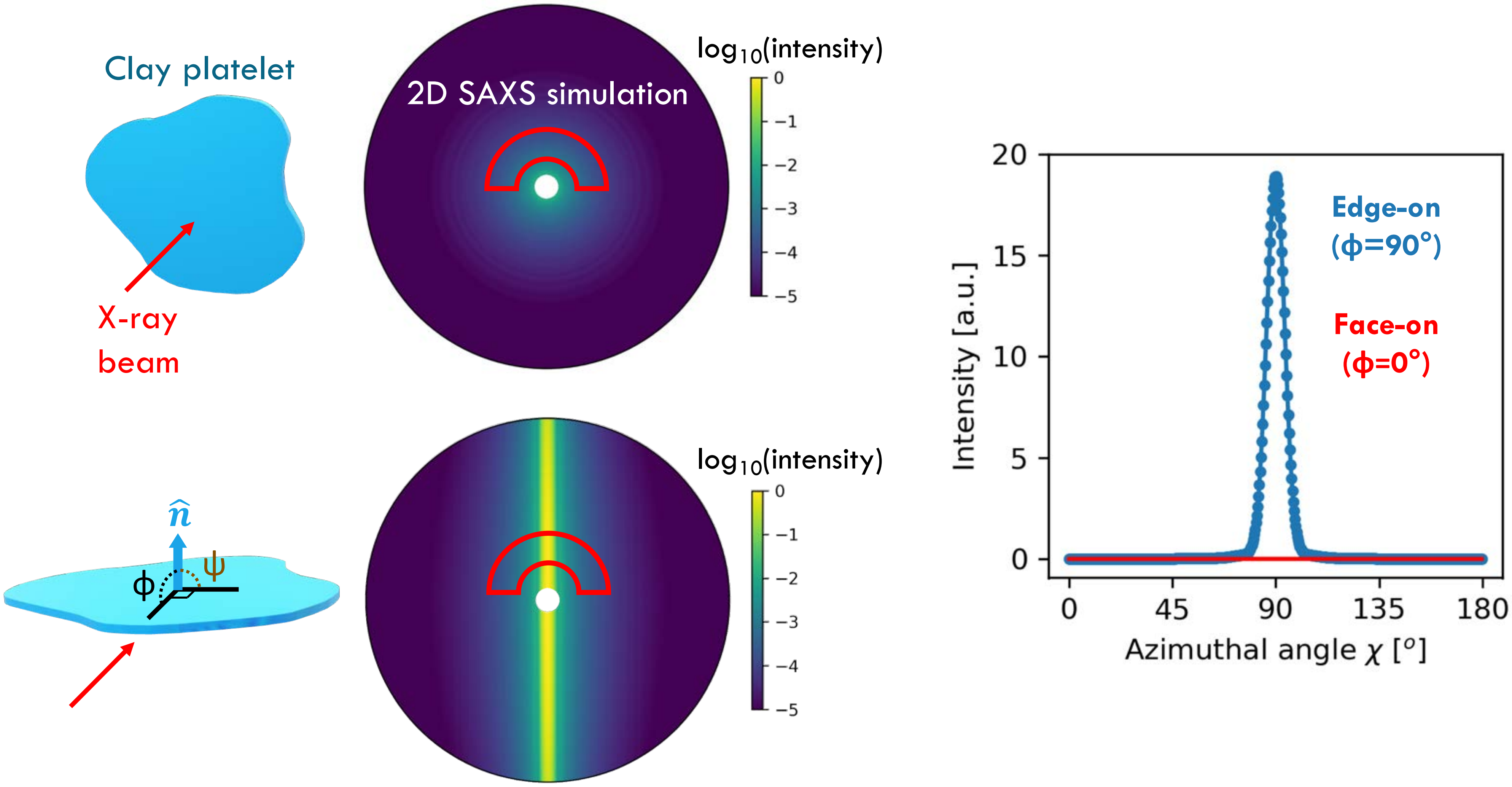
	SAXS	WAXS
Detectors	Pilatus 200k (1)	Pilatus 100k (2)
Sample-detector distance [m]	0.99	0.14
q range [Å^{-1}]	0.02 – 0.2	1.25 – 3.3
Exposure time [s]	0.1	0.1



Detector output via “InstantPlot_v1.py”, E. Trigg

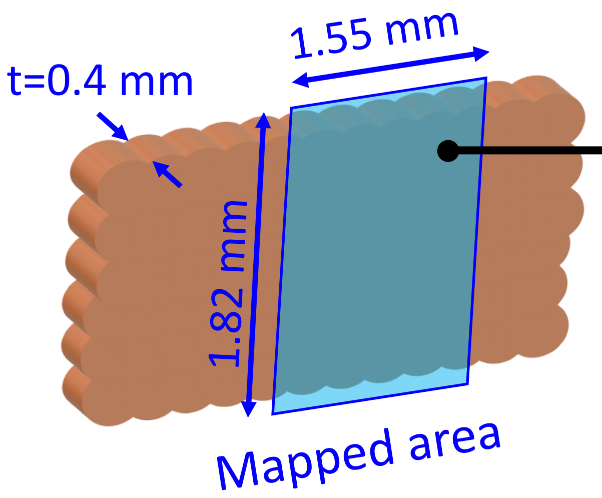


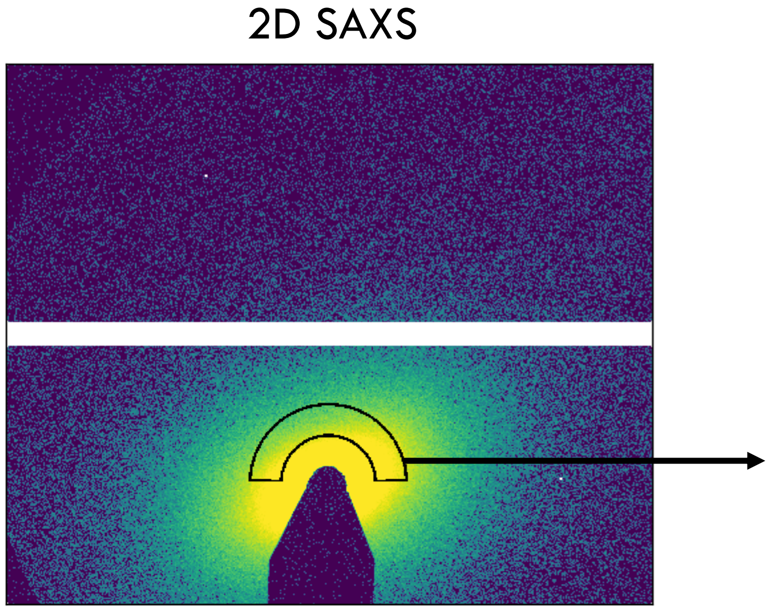
Simulated 2D SAXS of nanoclay platelets

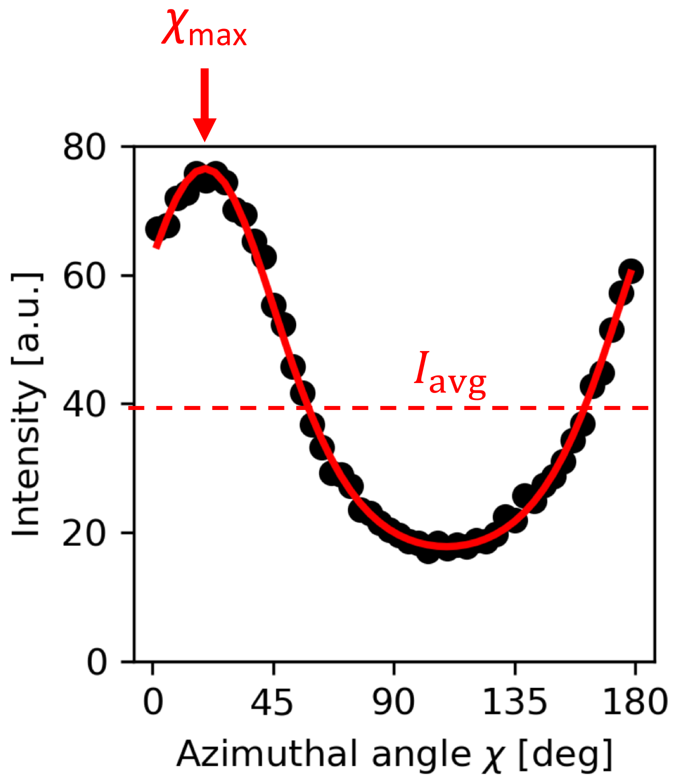


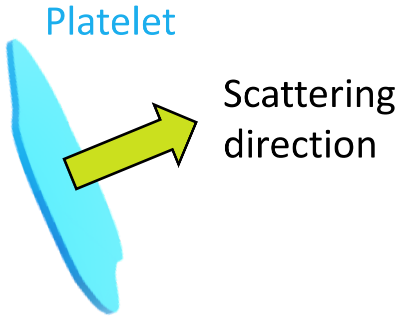
Using equations from Bihannic, I.; Baravian, C.; Duval, J. F. L.; Paineau, E.; Meneau, F.; Levitz, P.; De Silva, J. P.; Davidson, P.; Michot, L. J. *J. Phys. Chem. B* **2010**, *114* (49), 16347–16355.

Microbeam SAXS from one sample location





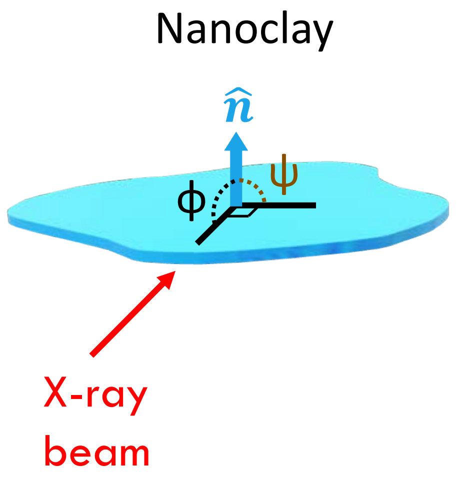


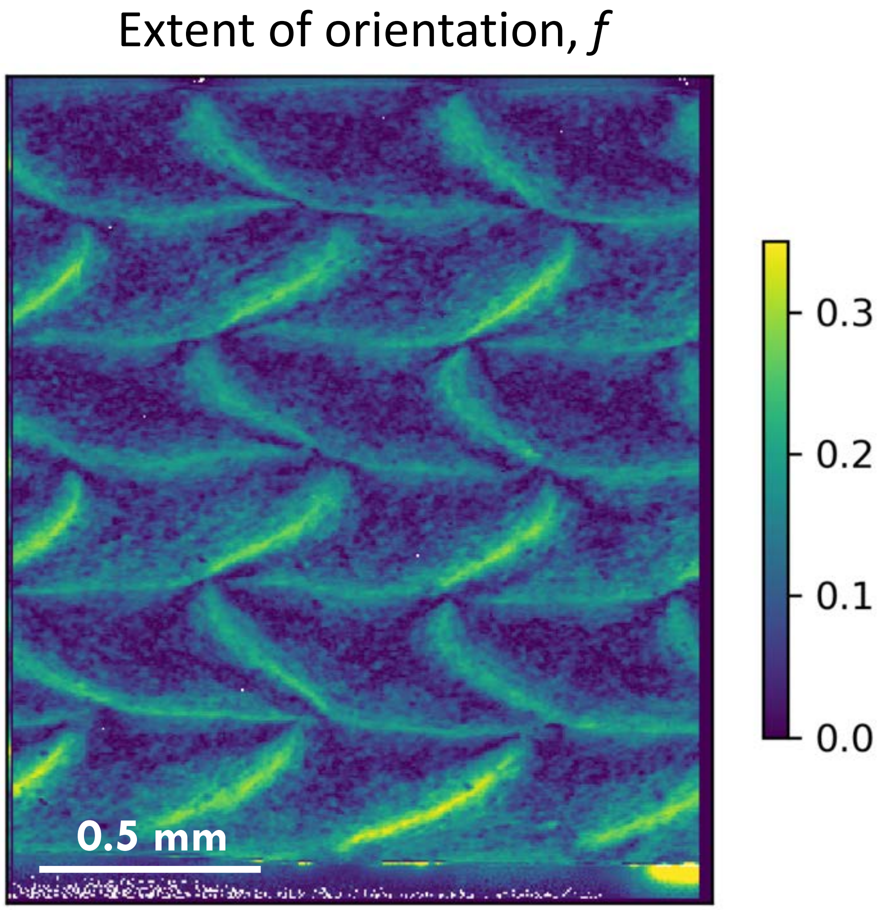


- Fit to Maier-Saupe orientation distribution function
- Calculate extent of orientation:

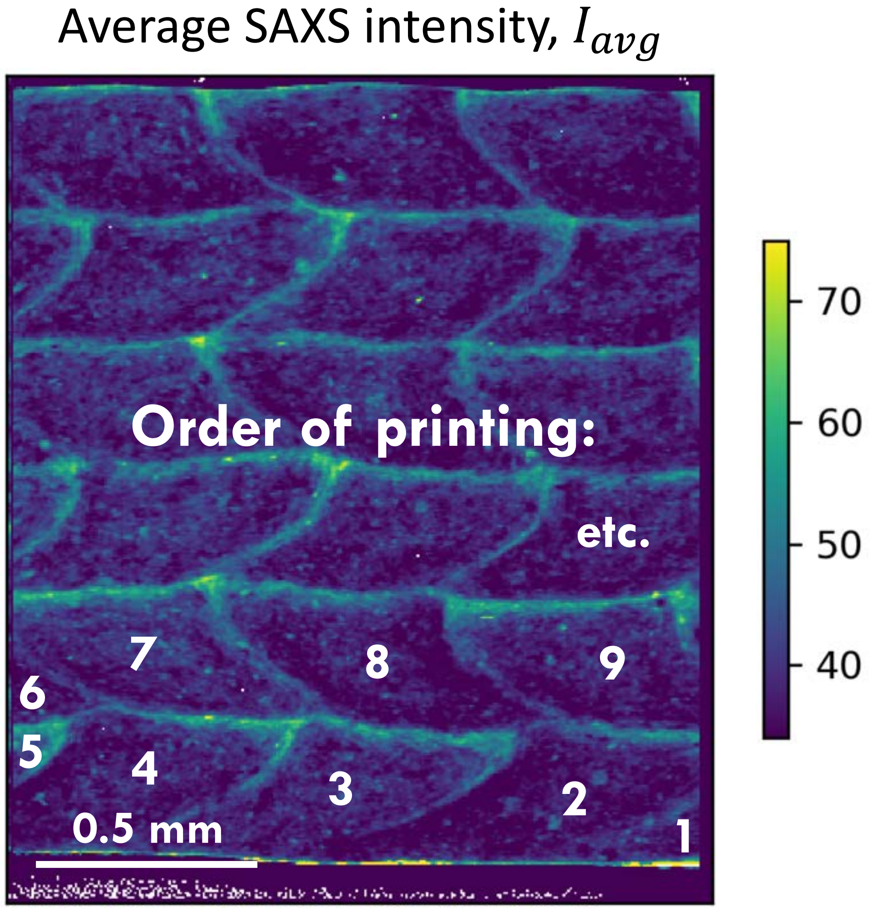
$$f = \frac{3}{2}\langle \cos^2(\chi - \chi_{max}) \rangle - \frac{1}{2}$$

SAXS reveals road structure and nanoclay orientation



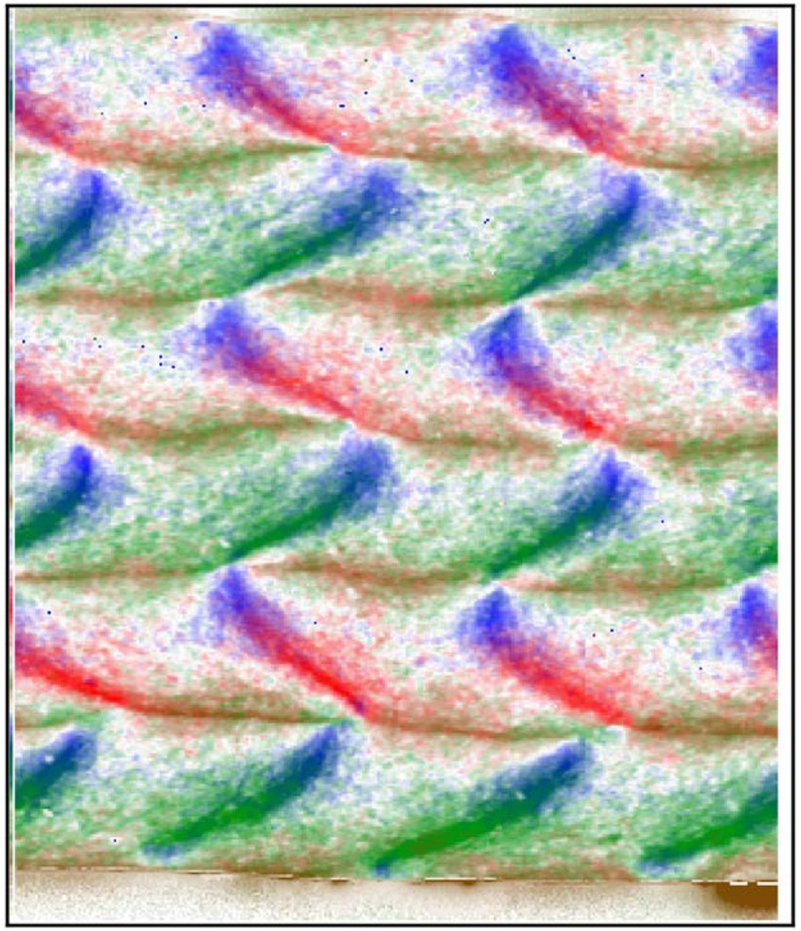


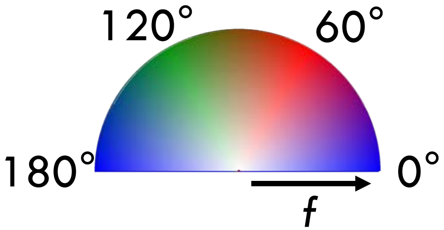
Related to ψ

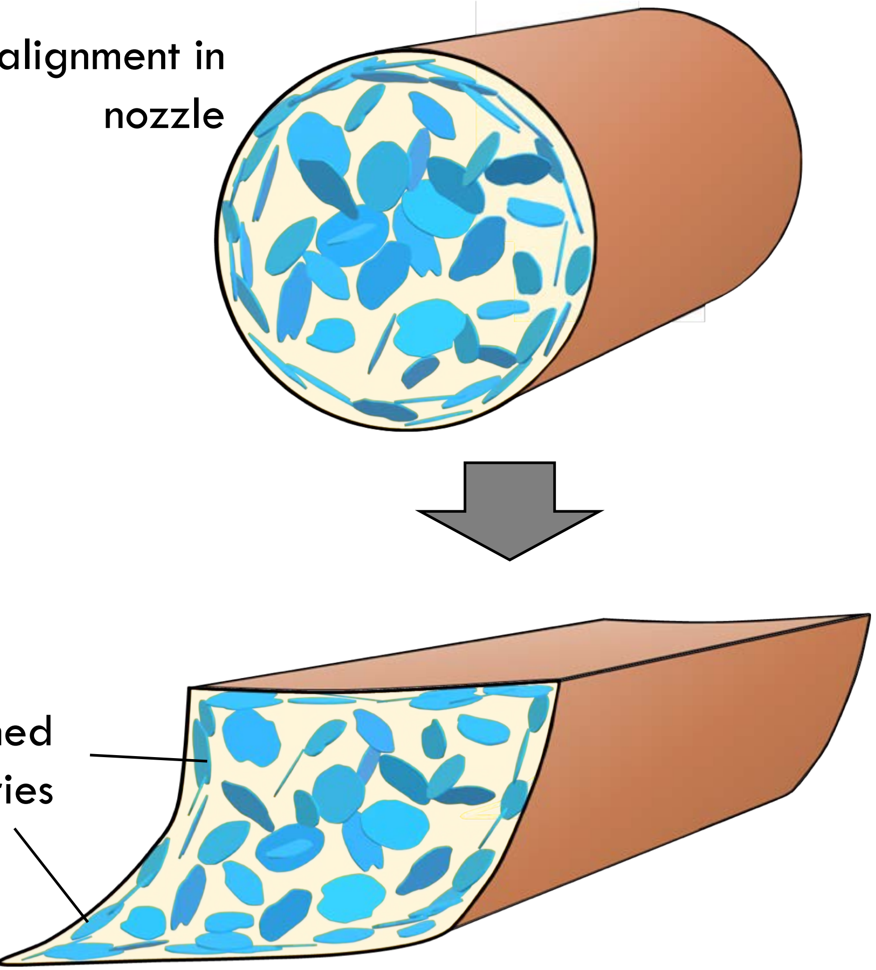


Related to ϕ

SAXS: mapping the *direction* of orientation, χ_{max}

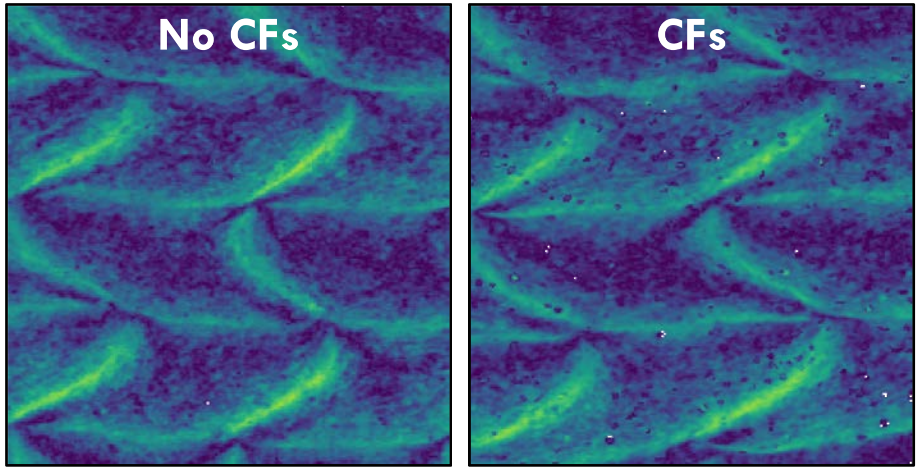




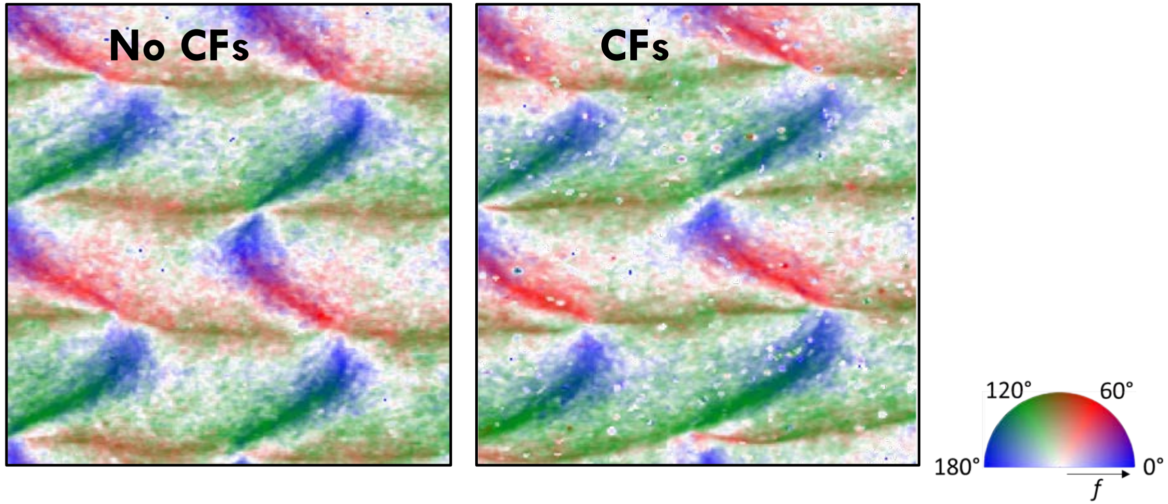


Adding carbon fibers does not change the nanoclay morphology

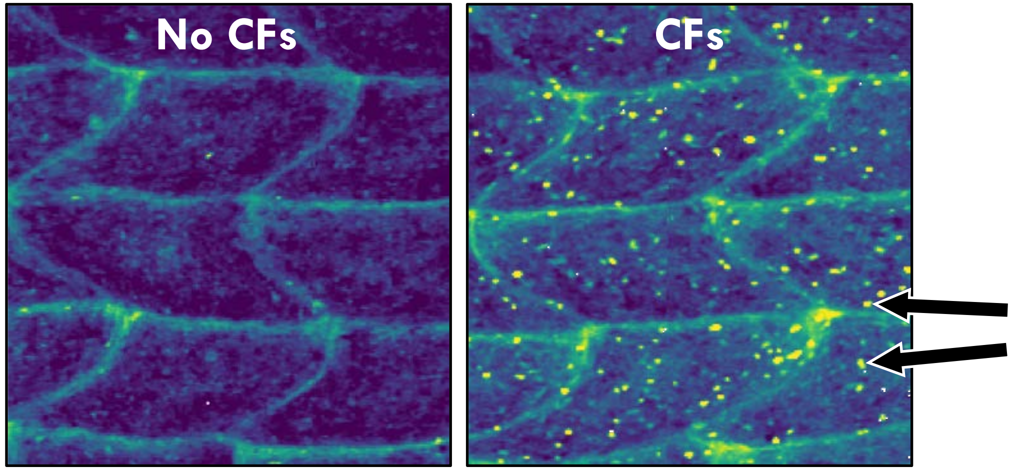
Extent of orientation, f



Direction of orientation, χ_{max}



Average SAXS intensity, I_{avg}

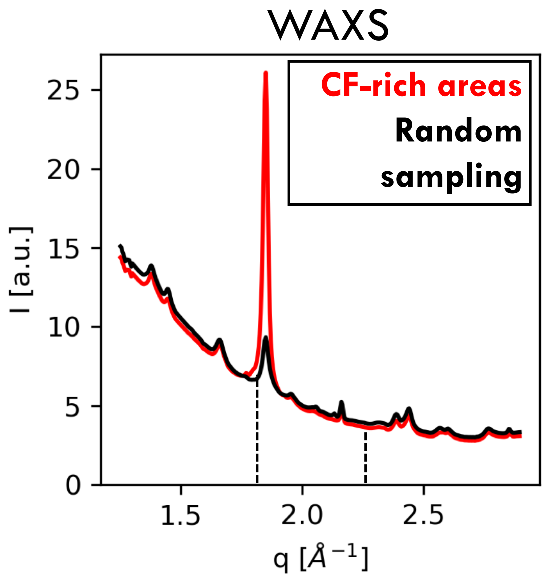


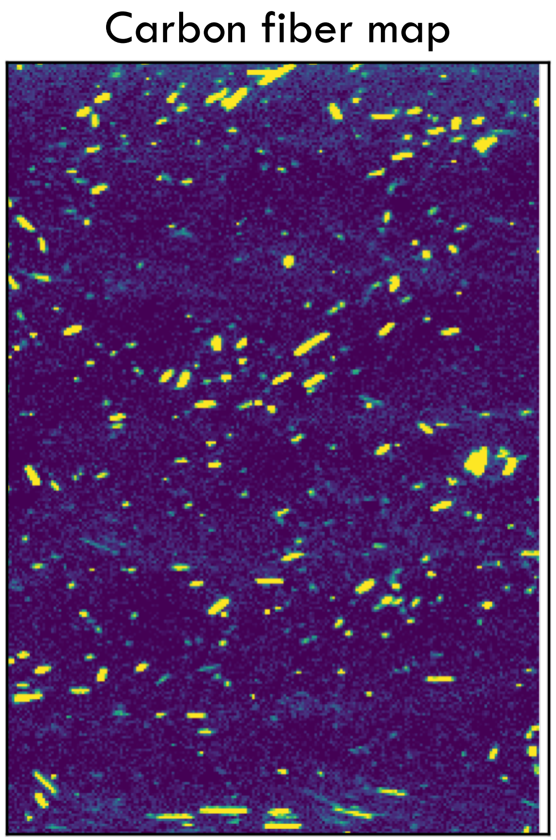
Are these the carbon fibers?

<u>Carbon fibers:</u>

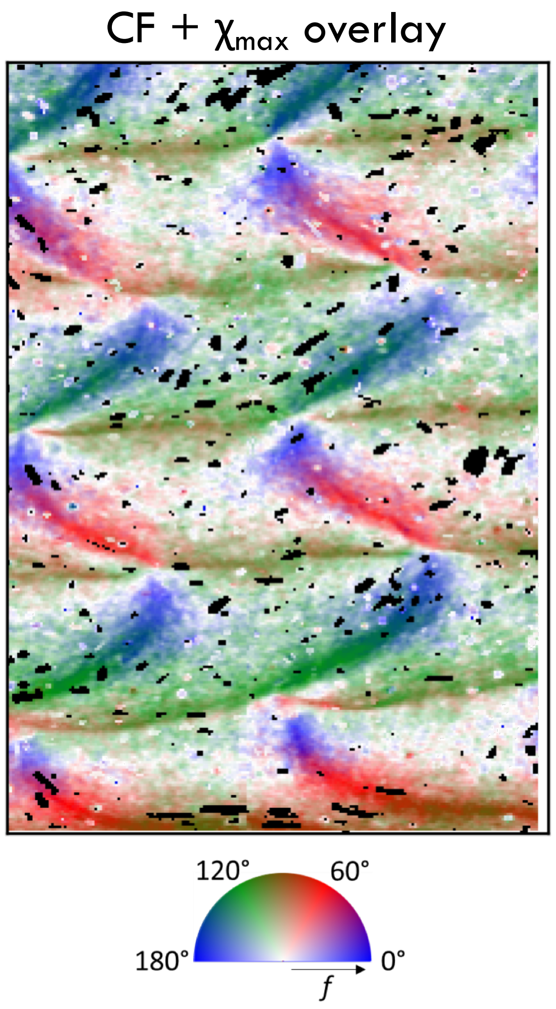
- Dimensions ≈ 150 μm x 10 μm
- Loading = 0.7% by volume

Mapping the carbon fibers with WAXS





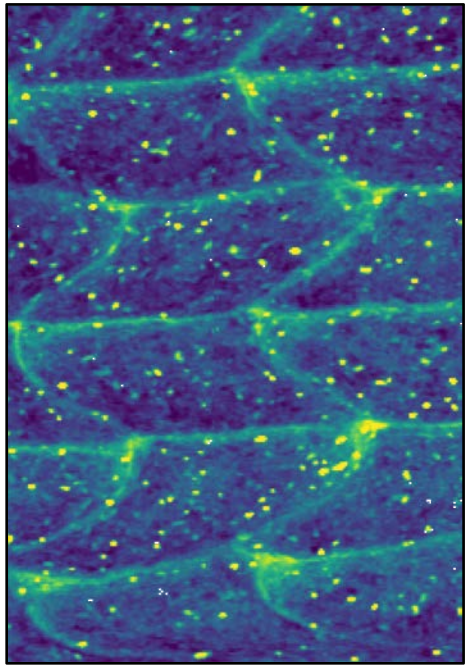
$$\frac{I(q = 1.77)}{I(q = 2.24)}$$



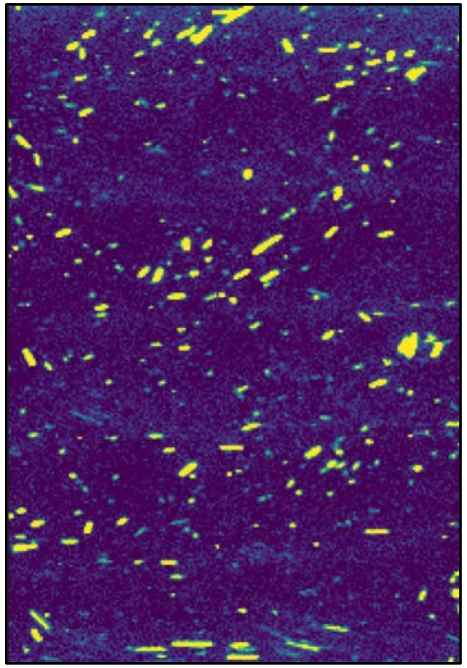
- Non-uniform dispersion
- Coplanar with clay

The high-intensity SAXS features are NOT the fibers

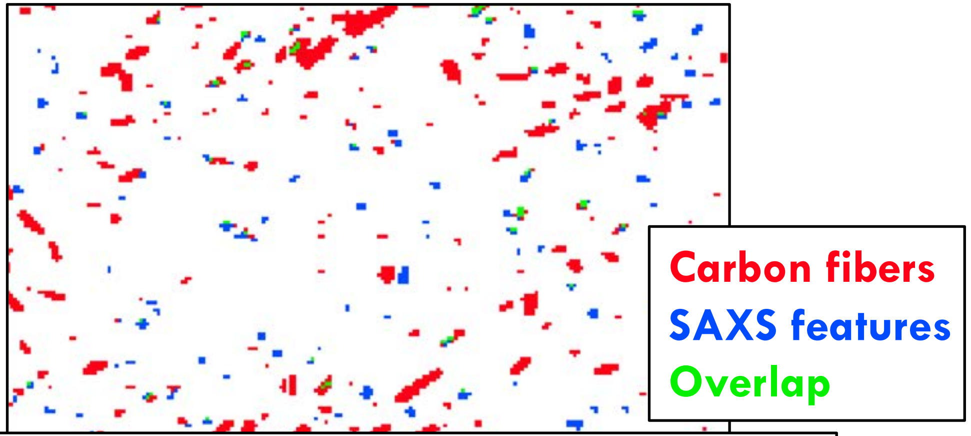
I_{avg} (SAXS)

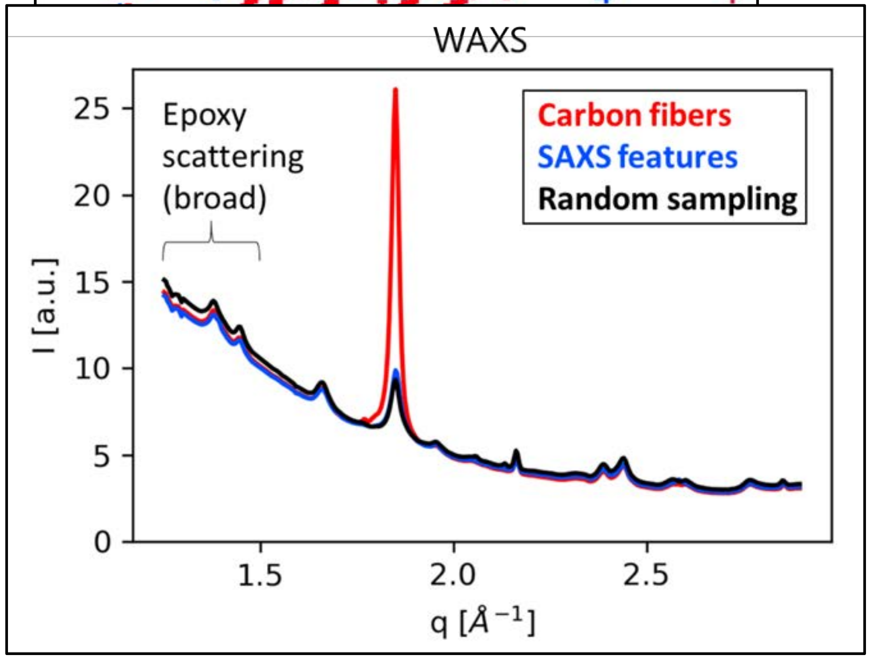


CF map (WAXS)



Overlay of binary images





Simulating polarized optical microscopy

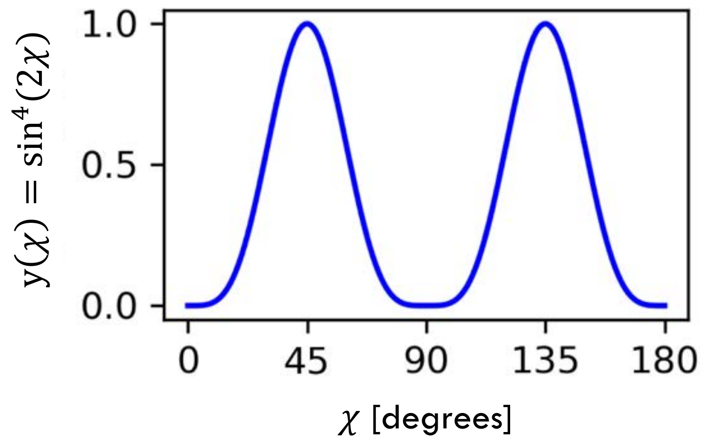
$$B = f * \sin^4(2\chi_{\max})$$

B: brightness of the pixel

f: Extent of orientation

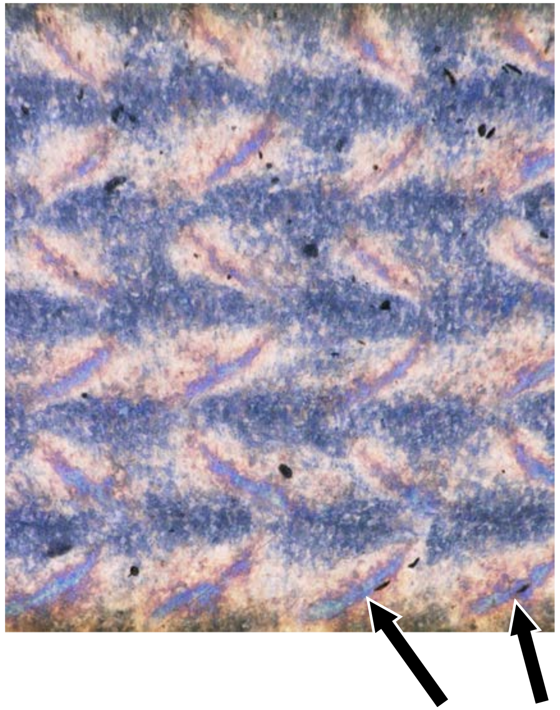
$\chi_{\max}$: Direction of orientation

$$y(\chi) = \sin^4(2\chi)$$

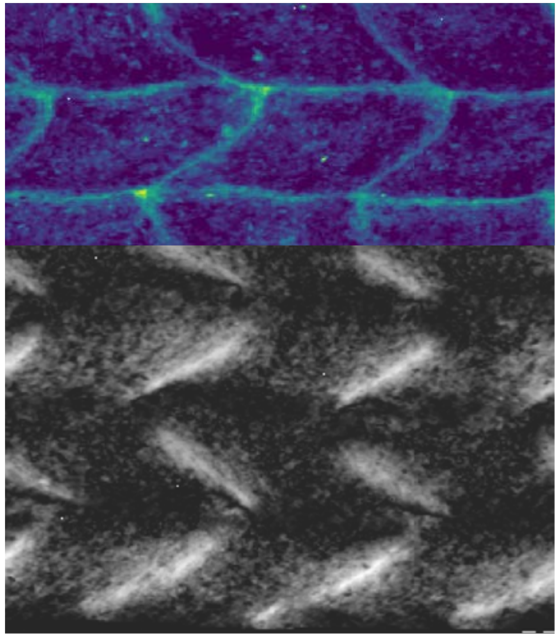


Used this function because POM brightness maxima occur at ϕ=45°, 135°, …

Optical microscopy

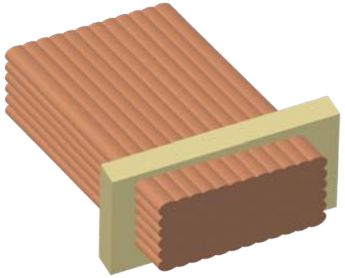
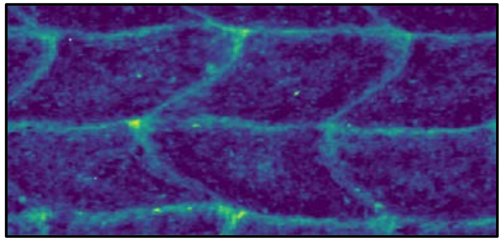


Simulated from SAXS

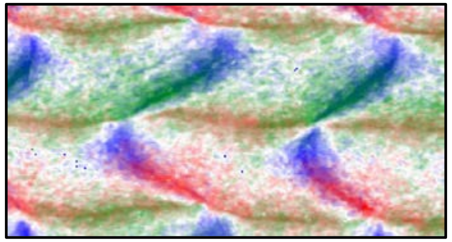
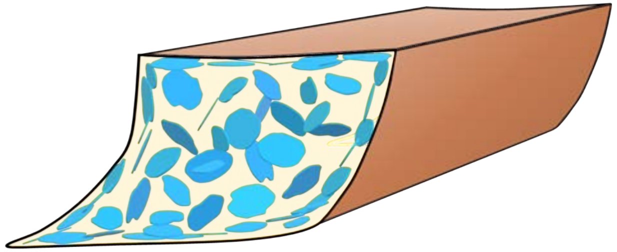


Road boundaries!

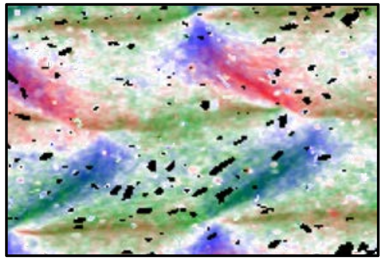
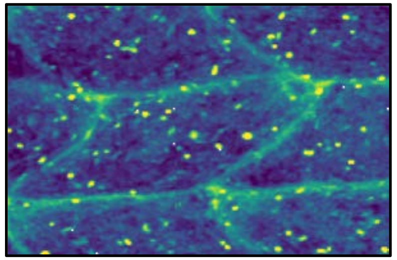
Conclusions

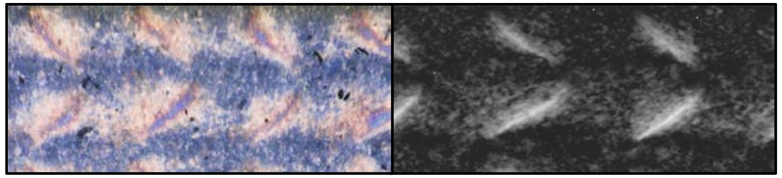
- Visualized road structure in two 3D-printed samples

- Mapped heterogeneous shear-induced nanoclay orientation
- Mapped carbon fiber onto road structure

- Observed possible voids in carbon fiber sample (previously undetected)
- Found that optical microscopy visualizes road boundaries (enabled by nanoclay alignment)

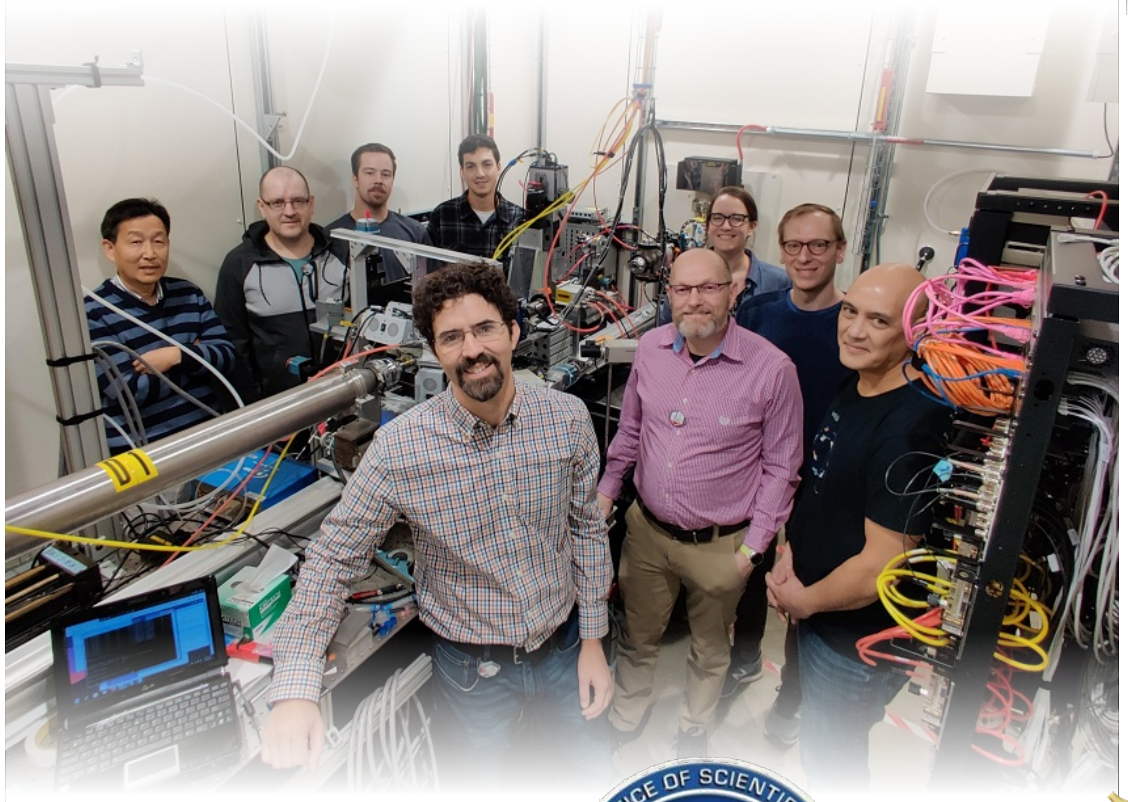


Acknowledgments:

CHESS
CORNELL HIGH ENERGY SYNCHROTRON SOURCE

AFRL
THE AIR FORCE RESEARCH LABORATORY
LEAD | DISCOVER | DEVELOP | DELIVER

MSN-C
FMB



Funding

NAS (NRC Fellowship)
AFOSR (17RXCOR436)
AFRL (FA8650-19-2-5220)
NSF (CMMI-1825815)
Honeywell FM&T (DE-NA0002839)

The National Academies of
SCIENCES · ENGINEERING · MEDICINE

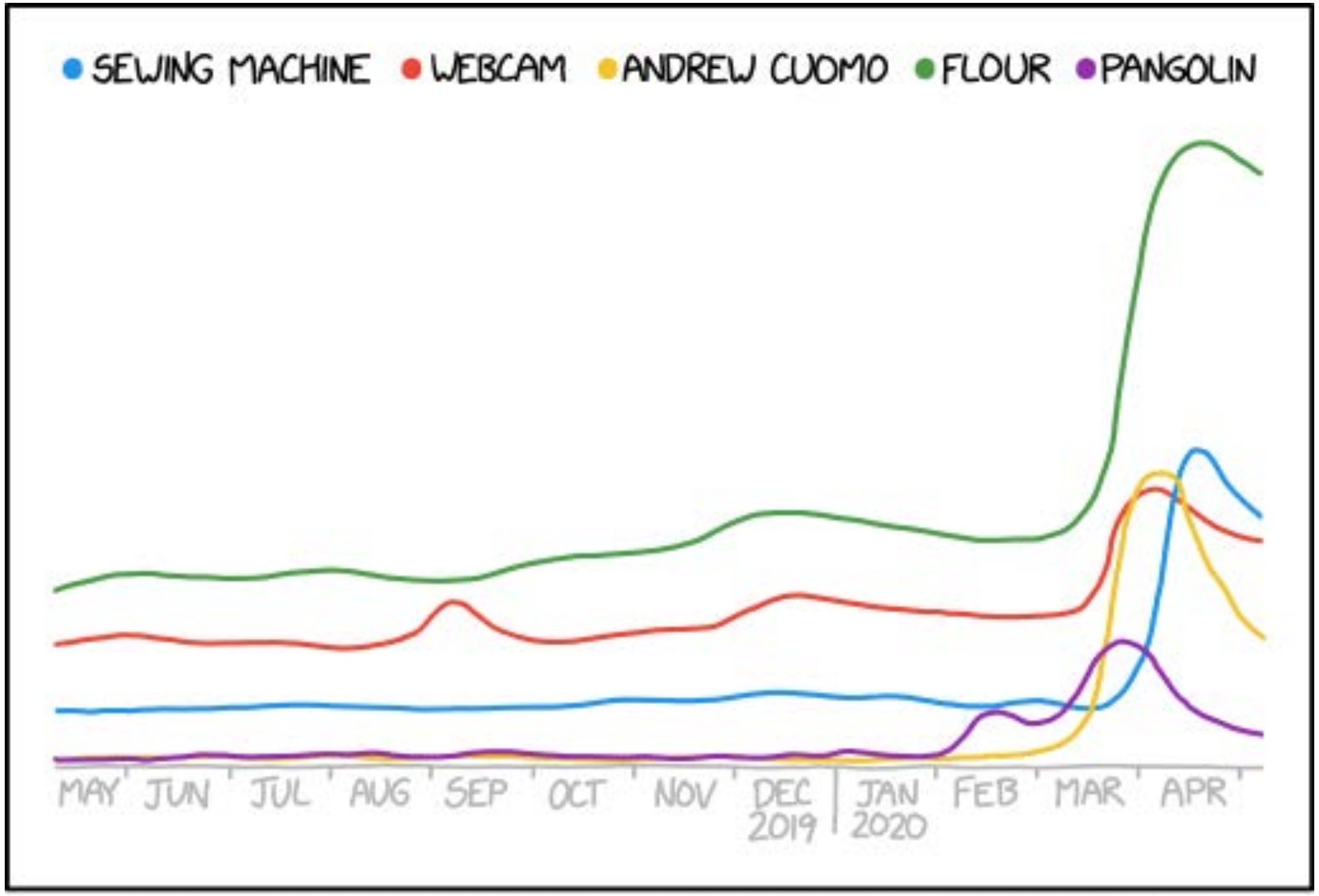
AIR FORCE OFFICE OF SCIENTIFIC RESEARCH
UNITED STATES AIR FORCE

NSF

Honeywell

BREAK

Up Next: Examples of SAXS/WAXS viewing at the beamline and/or Jupyter-based image processing.



I WANT TO SHOW SOMEONE FROM 2019 THIS GOOGLE TRENDS GRAPH AND WATCH THEM TRY TO GUESS WHAT HAPPENED IN 2020.

https://imgs.xkcd.com/comics/2020_google_trends.png