Synchrotron Three-Dimensional X-ray Diffraction (3D-XRD)

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Canadian Powder Diffraction Workshop 18

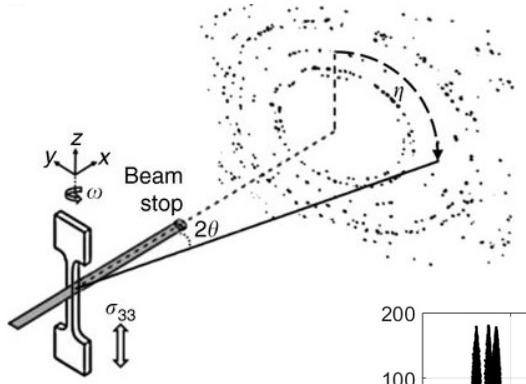
July 31st 2025

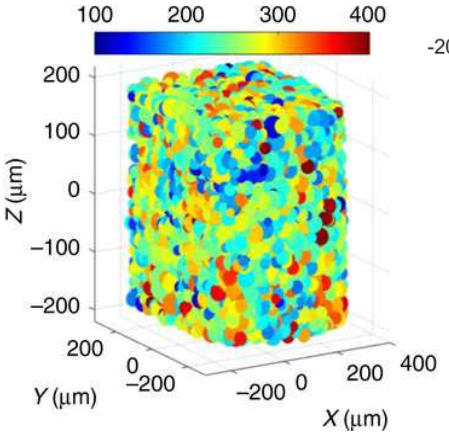
Outline

- Background
- 3D-XRD at ESRF
  - Introduction
  - Data processing
  - Results
- 3D-XRD at CHESS
  - Introduction
  - Results
- Developing 3D-XRD at CLS
  - Stress-Rig and Huber Components
  - Dataprocessing complementary tool
- Final Remarks: Where can I do 3D-XRD?

What is 3D-XRD?

- Microscopy technique for characterizing polycrystalline materials.
- Utilizes hard X-rays (30 – 100 keV) to investigate the materials at the micro- and nano- levels
- Works through Bragg condition.
- The final outputs are grain positions, orientations and elastic strains/stresses in three dimensions





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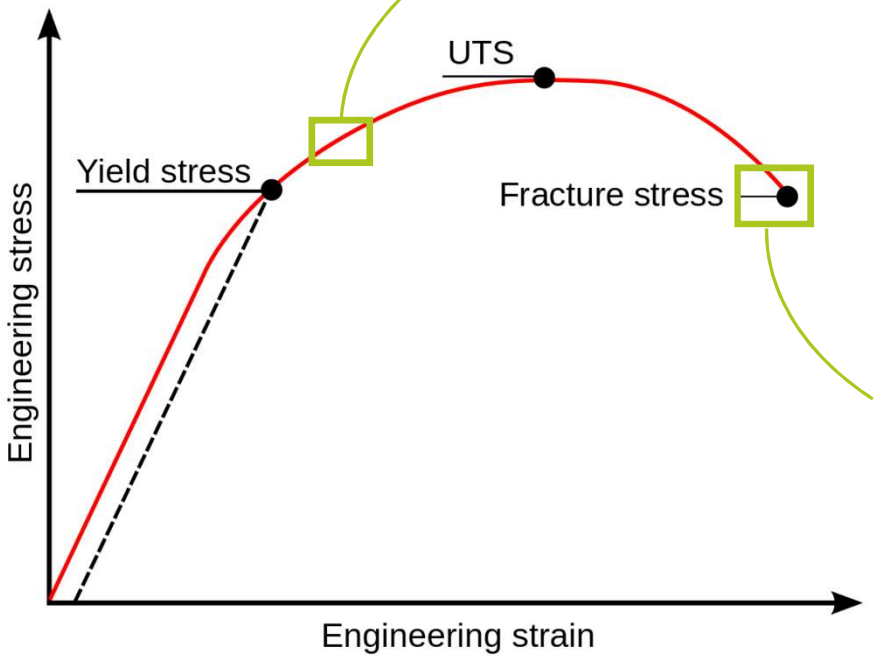
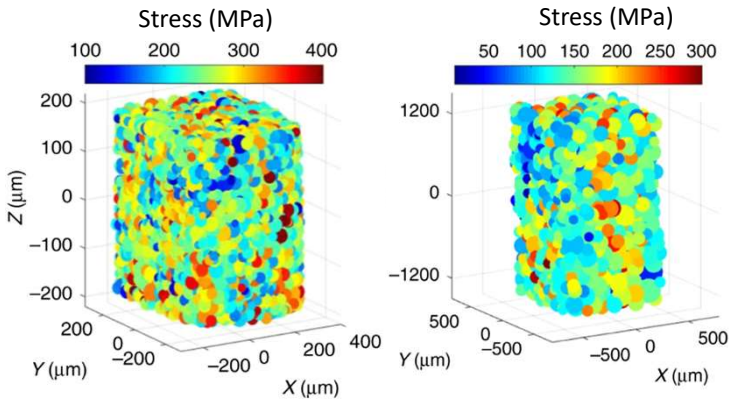
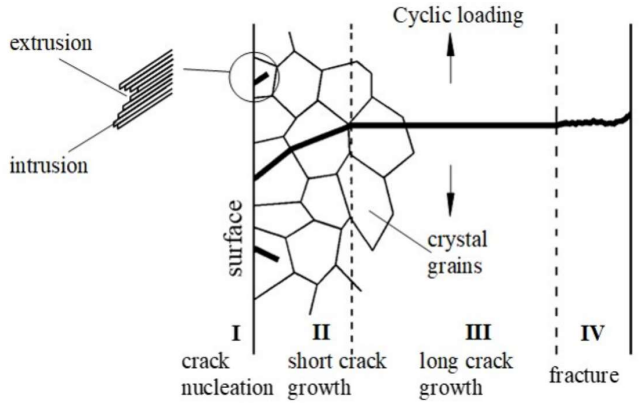
DISCOVERY AT THE SPEED OF LIGHT

Background – Polycrystalline Materials







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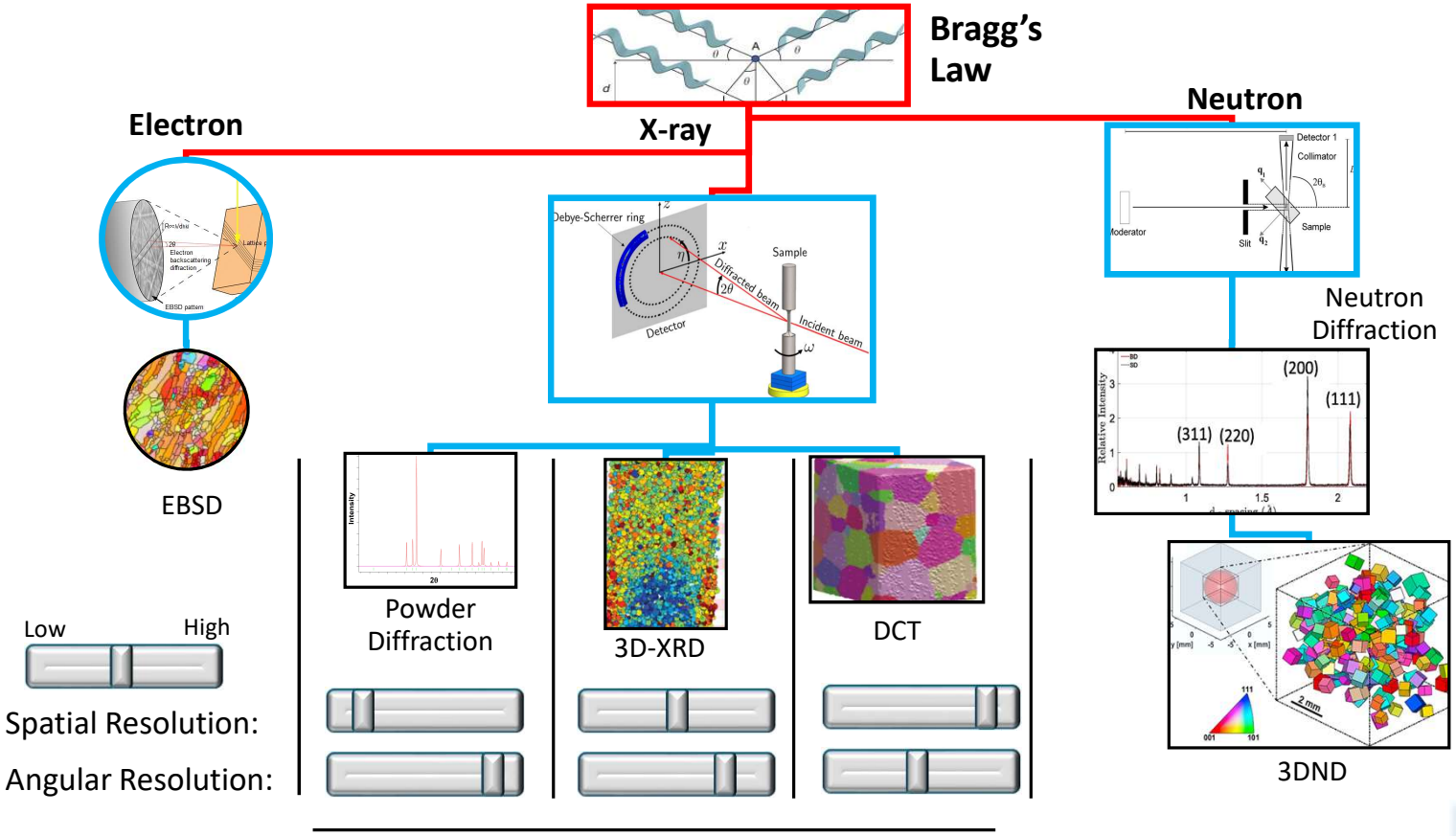
DISCOVERY AT THE SPEED OF LIGHT

Background – Diffraction Techniques



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DISCOVERY AT THE SPEED OF LIGHT

Synchrotron-1: European Synchrotron Radiation Facility (ESRF), Grenoble, France
Beamline: ID11
Topic: Deformation Twinning (Masters)

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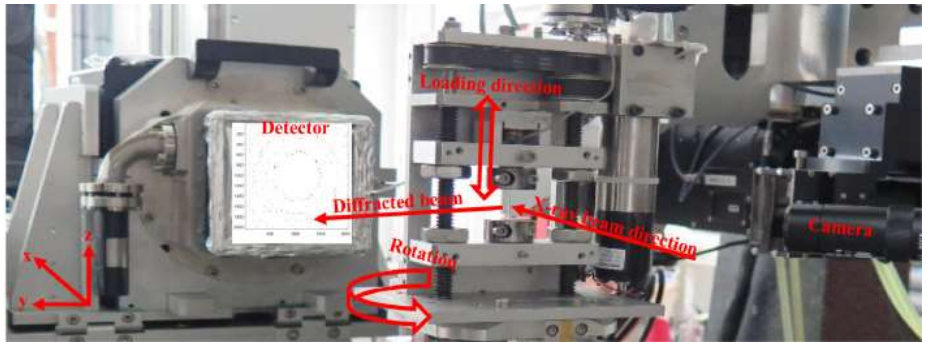
DISCOVERY AT THE SPEED OF LIGHT

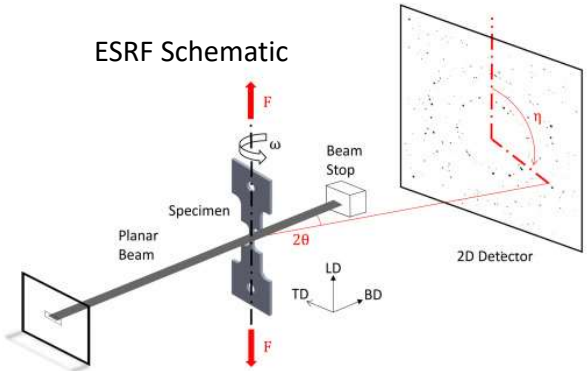
How 3D-XRD works?

- This method was developed by Henning Poulsen in 2004 [1].
- Electromagnetic radiation is generated by acceleration of charged particles (i.e. electrons)
- The x-ray beam is struck on the sample through focusing techniques
- The scattered rays are collected on a two-dimensional detector
- The sample stage is rotated to collect diffraction patterns at all angles
- <u>Objective</u>: Study deformation twinning in hexagonal close-packed (HCP) metals

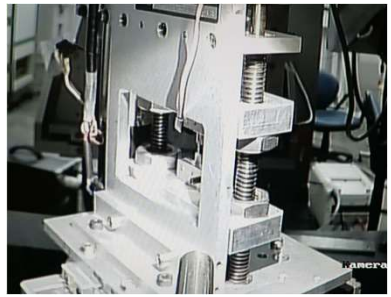
ESRF Setup



ESRF Schematic



Stage Rotation



Reference
[1] H. F. Poulsen, vol. 5. **Springer**, 2004.

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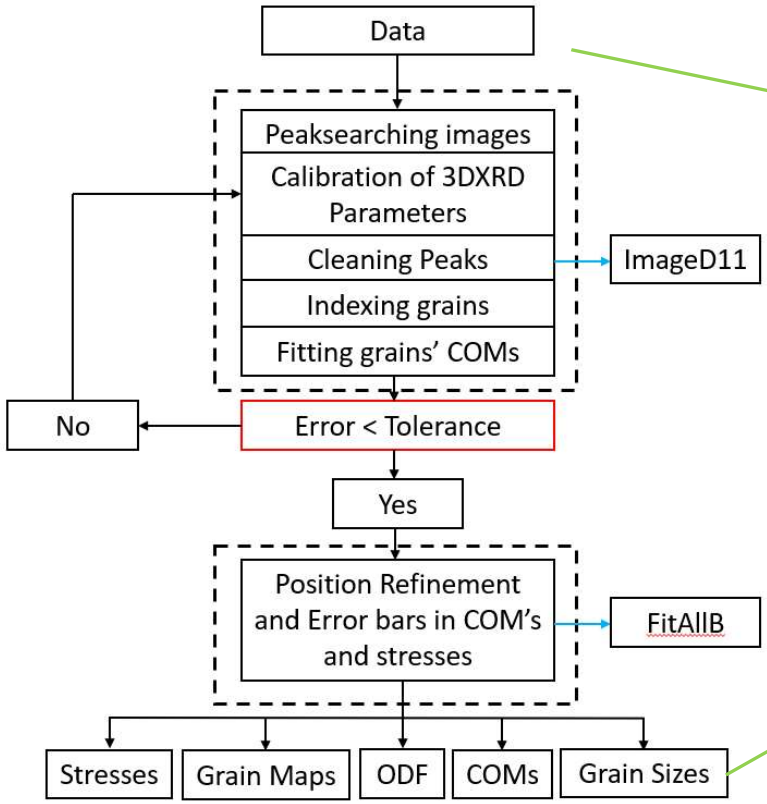
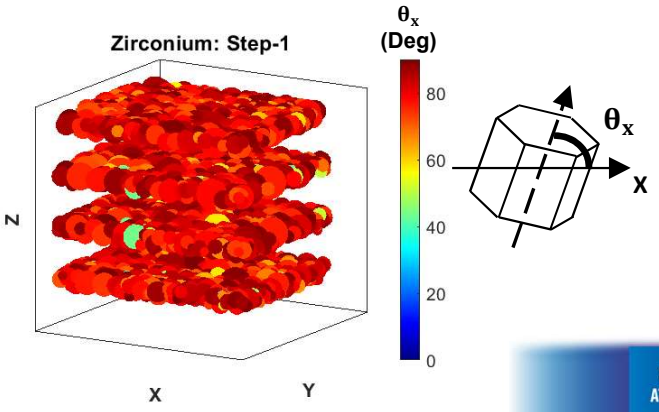
DISCOVERY AT THE SPEED OF LIGHT

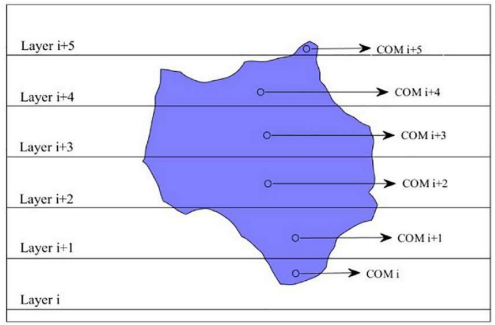
Data Processing – Indexing and Fitting



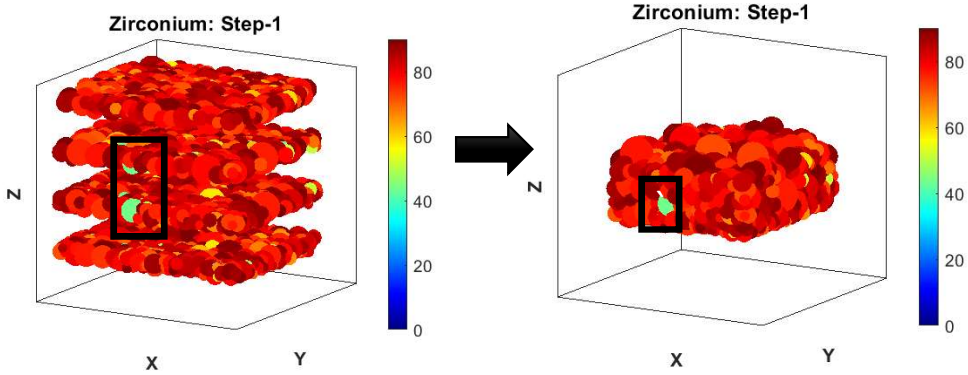


Data processing: Assembling Layers and Matching Across Loading Steps

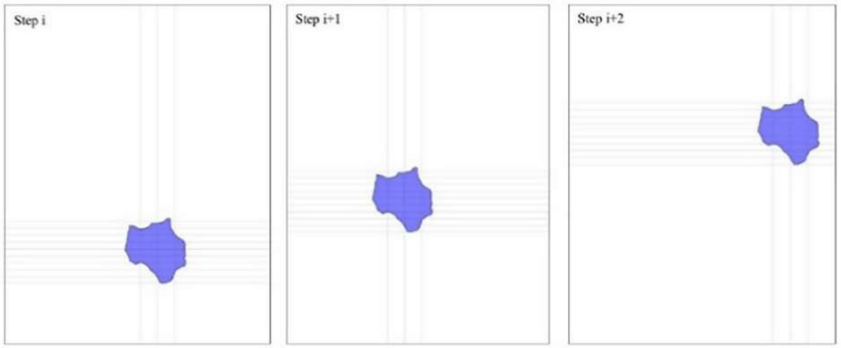
Assembling Layers

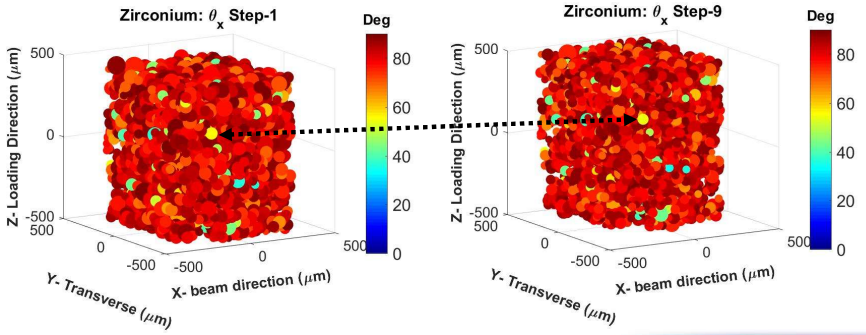


Final Maps: Before and after assembly (4 Layers demo)



Matching Across Loading Steps





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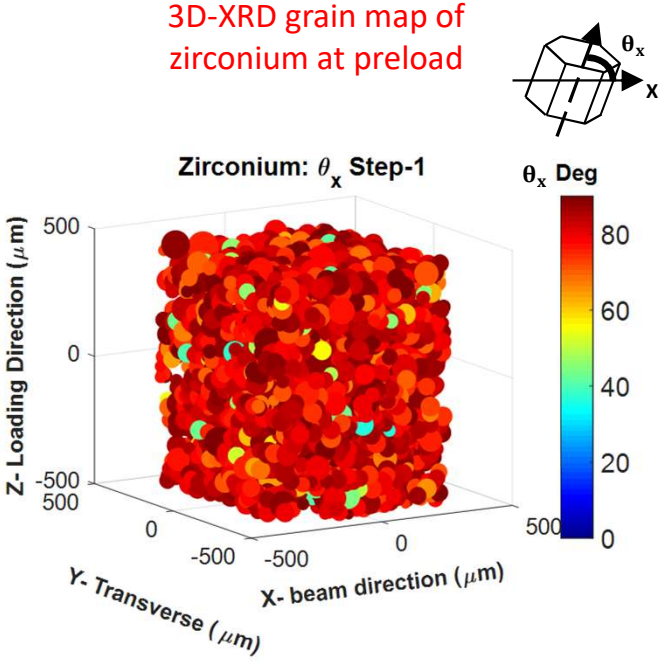
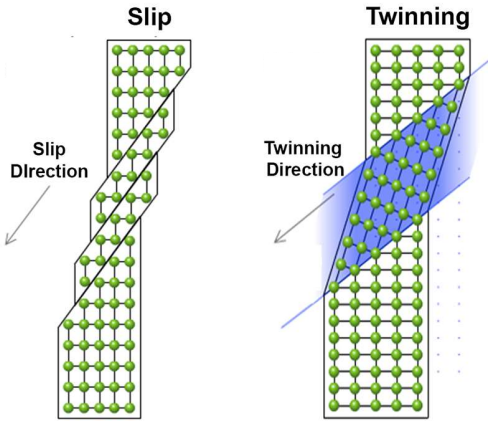
Results

3D-XRD grain map of zirconium at preload

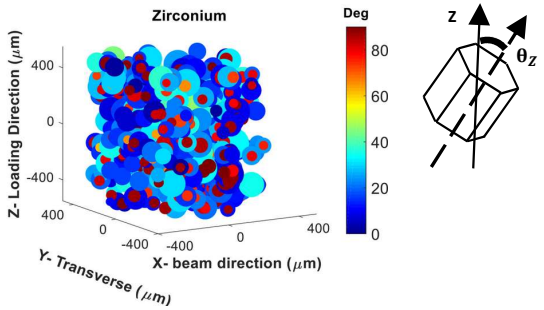


Deformation Twinning

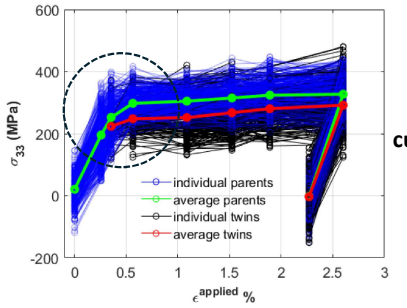


Schematic of crystal plasticity deformation mechanisms: slip and deformation twinning

Deformation Twinning in 3D-XRD



3D-XRD grain map of parent and twins



Stress-strain curves of parent and twins

Featured news and articles:

References

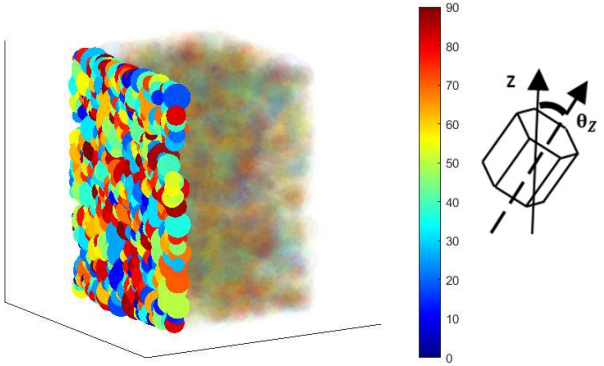
[1] H. Abdolvand, K. Louca, C. Mareau, M. Majkut, and J. Wright, ***Acta Mater.***, vol. 196, pp. 733–746, 2020

[2] K. Louca, H. Abdolvand, C. Mareau, M. Majkut, and J. Wright, **Nature Commun. Mater.**, vol. 2, no. 1, p. 9, 2021

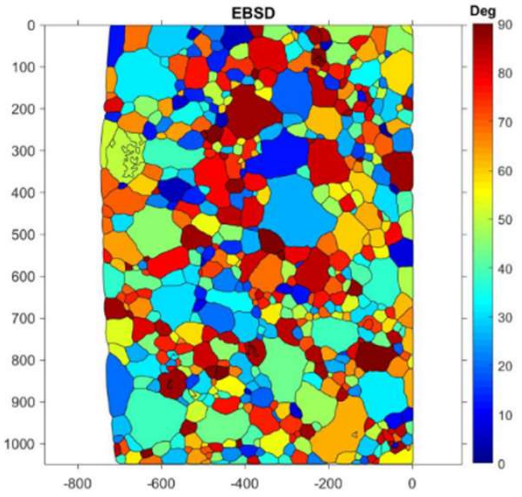
Comparison with EBSD

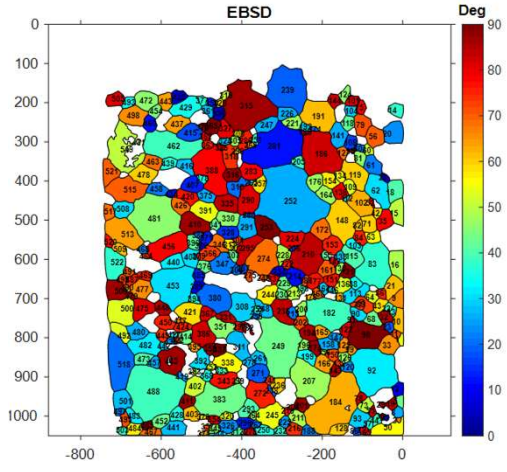
Algorithm
- Find common largest grain.
- Fix center of map.
- Apply misorientation and distance criterion.

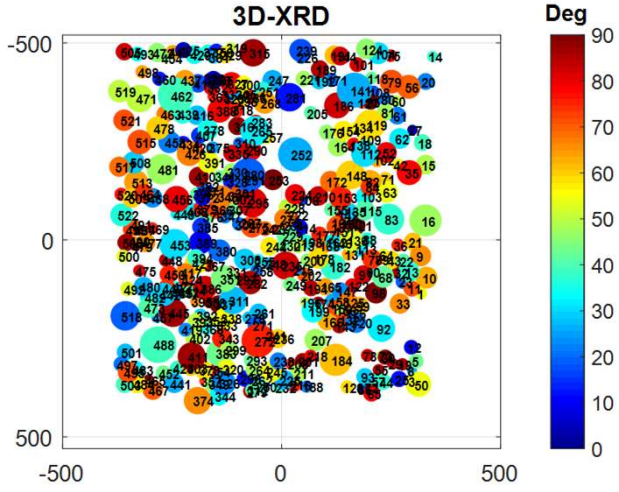
Full 3D-XRD Map



Full EBSD Map







Comparison

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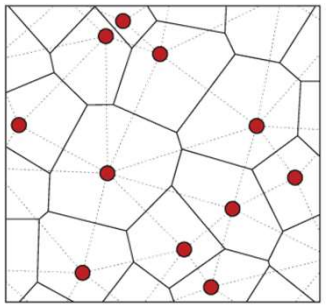
DISCOVERY AT THE SPEED OF LIGHT

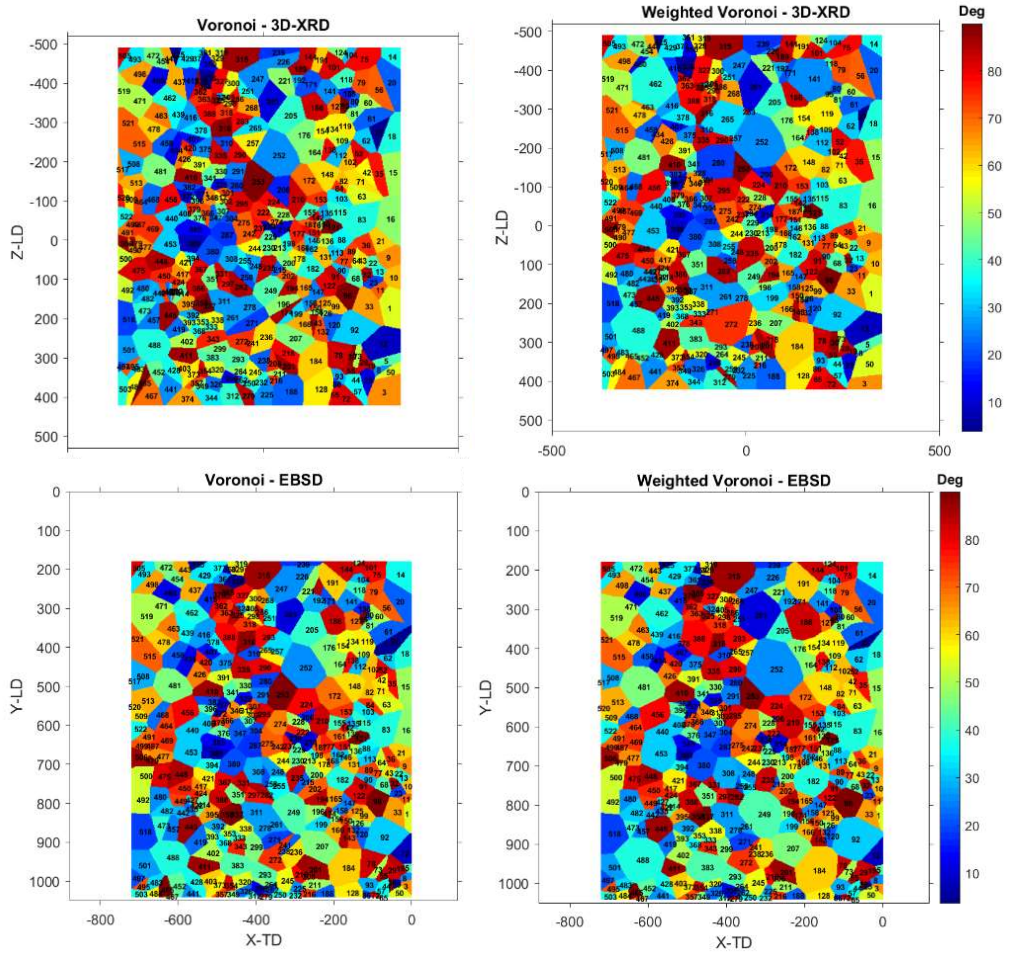
Comparison with EBSD: Voronoi Maps

- Neighborhood population of each reconstructed map is compared to EBSD,

- $$\text{MQF} = \frac{\text{Number of matched neighbors}}{\text{Total Number of neighbors measured in the EBSD grain map}} \times 100\%$$

- 94%, 90%, 81%, 80% of grain neighbors were matched for Voronoi and weighted Voronoi EBSD and 3D-XRD respectively.





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DISCOVERY AT THE SPEED OF LIGHT

Synchrotron-2: Cornell High Energy Synchrotron Source (CHESS), Cornell University, NY, US
Beamline: Forming and Shaping Technology (FAST)
Topic: Micromechanics of notched geometries (PhD)

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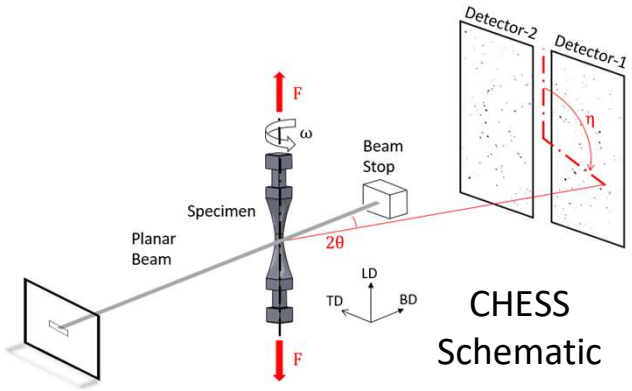
DISCOVERY AT THE SPEED OF LIGHT

Experimental Setup

Experiments

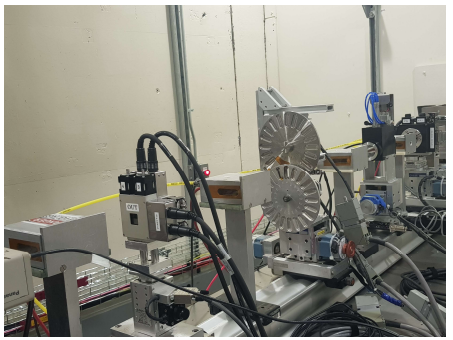
- Objective: Acquire microstress distributions in the vicinity of notches and investigate the role of microstructure.
- Study-1: 50 load-unload cycles on two pure zirconium specimens with **radial** texture with two different notch geometries.
- Study-2: 35 load-unload cycles on two pure zirconium specimens with **axial/soft** texture with two different notch geometries.

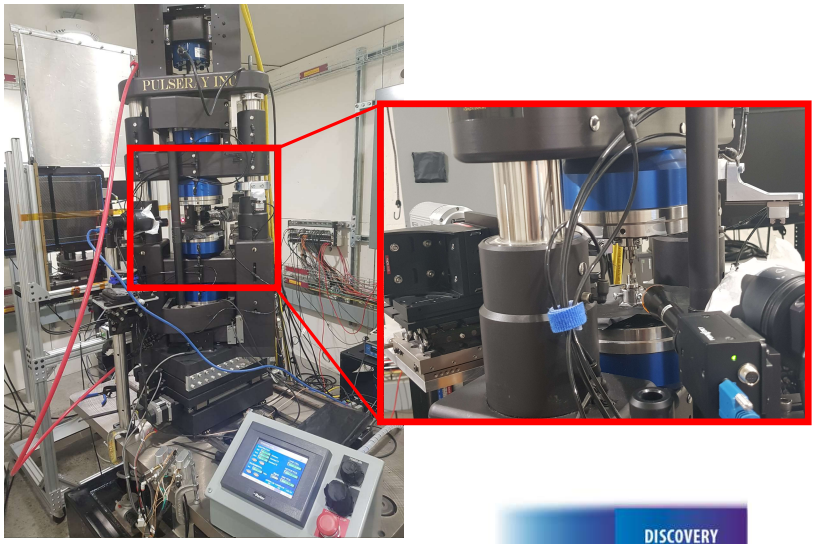
Data processing: Procedure similar to ESRF. Python library: HEXRD



CHESS Schematic

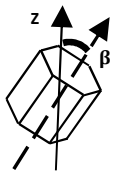
Beam Source





Tensile Rig

Results (Asymmetrical Maps)

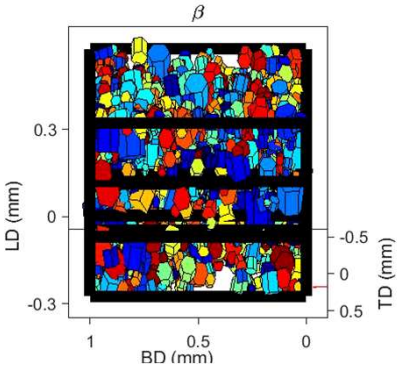


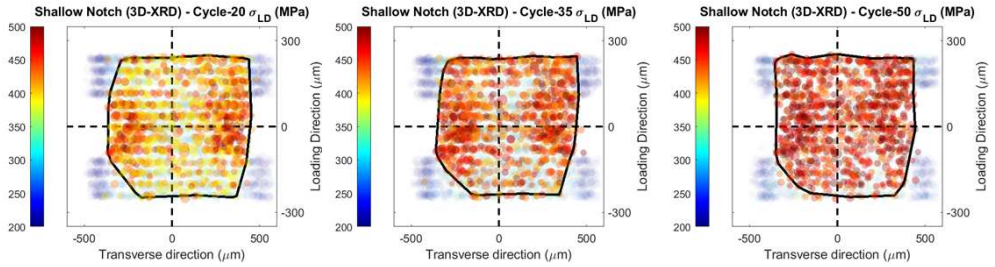
3D-XRD grain maps

Asymmetries in stressed deformation maps

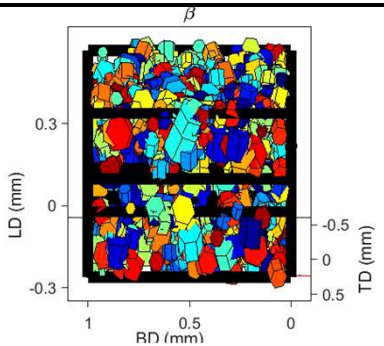
Crystal Plasticity Finite Element Modelling (CPFE)

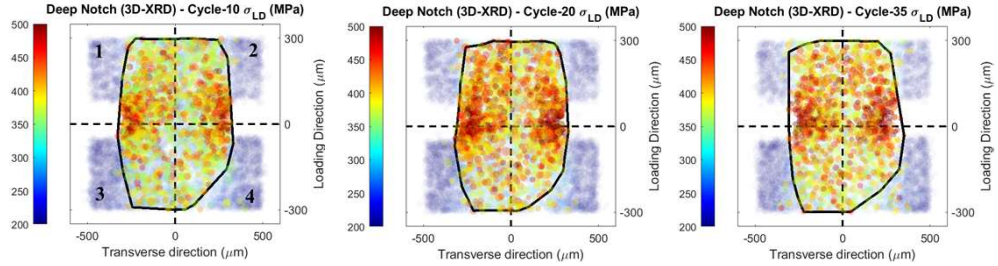
Shallow Notch

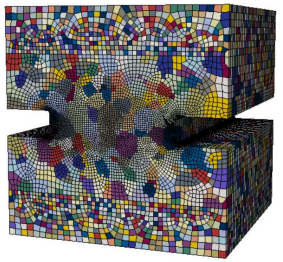




Deep Notch







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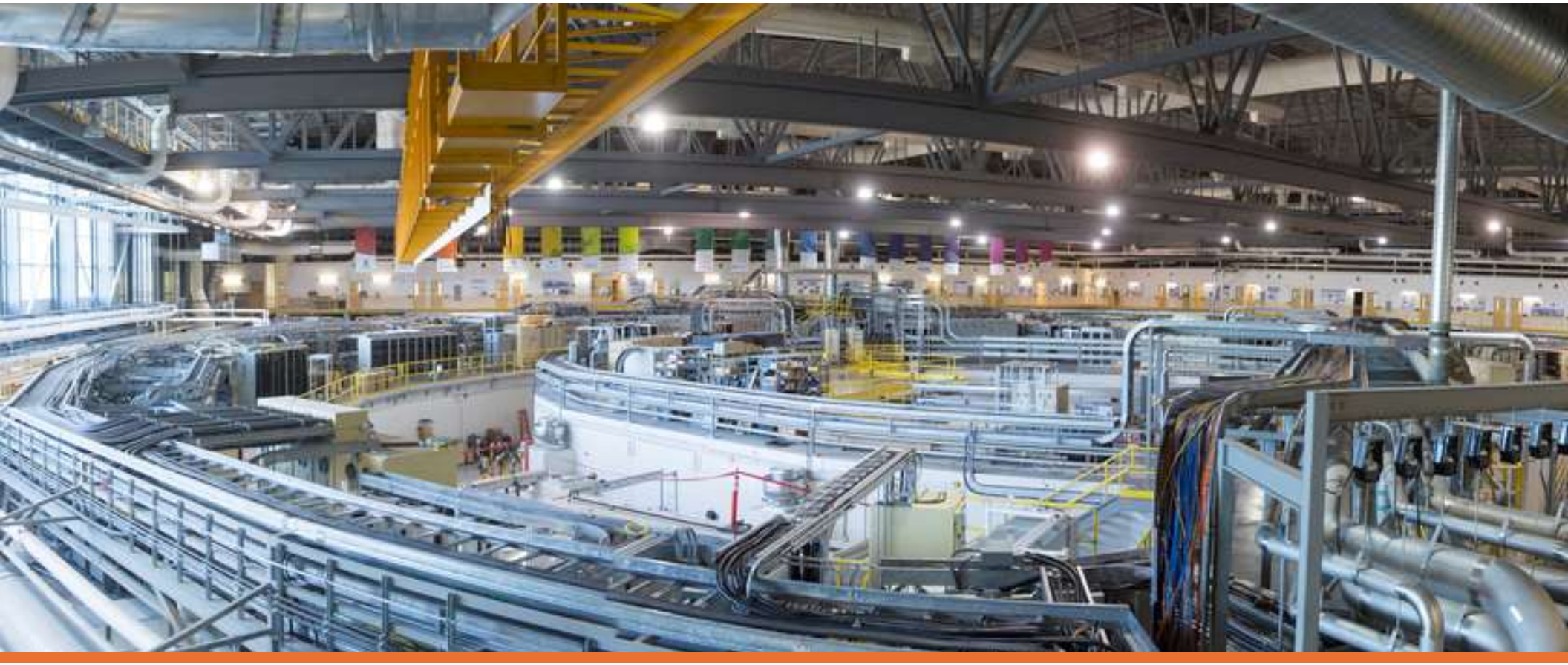
DISCOVERY AT THE SPEED OF LIGHT

Reference

[1] K. Louca, K. S. Shanks, A. Das, and H. Abdolvand, ***Acta Mater.***, vol. 264, p. 119567, 20240



Synchrotron-3: Canadian Light Source, Saskatoon, SK, CA
Beamline: Brockhouse X-ray Diffraction Sector (BXDS)
Topic: Commissioning the 3D-XRD technique

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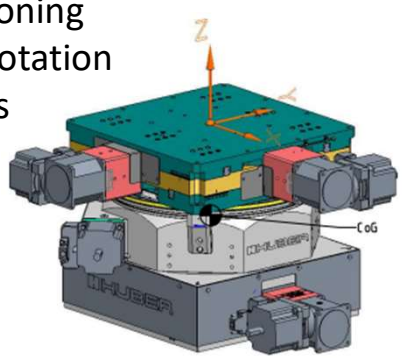
DISCOVERY AT THE SPEED OF LIGHT

Bringing 3D-XRD to the CLS

Establishing 3D-XRD

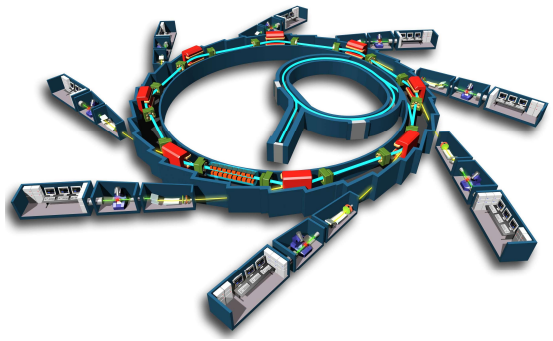
Gather Equipment → Test Measurements → Data Collection and Processing → Data Analysis

Positioning and Rotation Stages



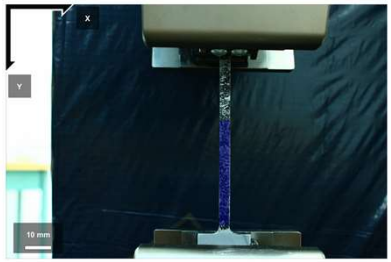
Stress Rig

Synchrotron Beam



Add-ons: Digital Image Correlation

Tensile Strain



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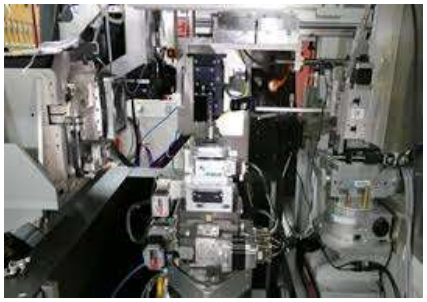
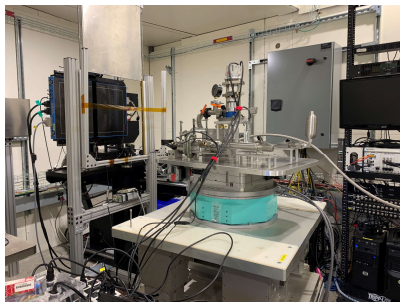
DISCOVERY AT THE SPEED OF LIGHT

Where can I do 3D-XRD?

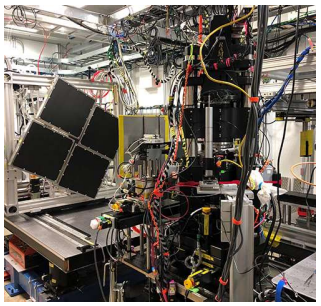
ESRF, ID11



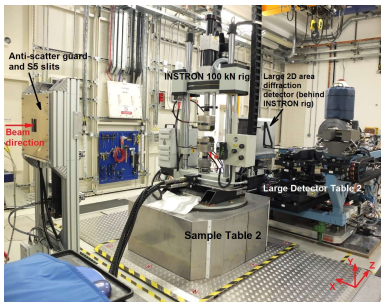
CHESS, FAST(3A)



APS, 1-ID

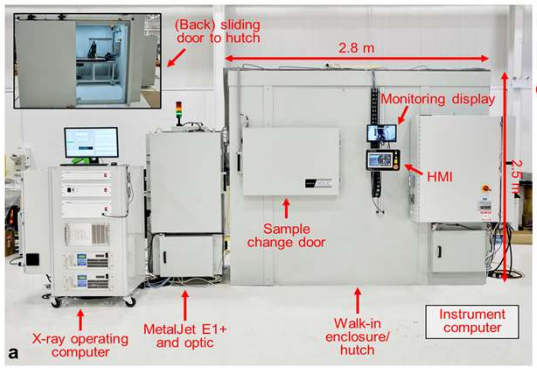


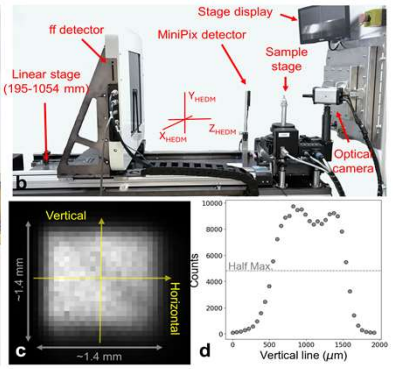
Diamond Light Source, I12 JEEP



Toyota Beamline, Spring-8

Lab at University of Michigan, Ann Arbor





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Thank You

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