Polycrystalline Diffraction

Matt Miller

Sibley School of Mechanical and Aerospace Engineering

Cornell High Energy Synchrotron Source (CHESS)

Cornell University

CHESS

Cornell High Energy
Synchrotron Source

Cornell University
College of Engineering

Where's Cornell?

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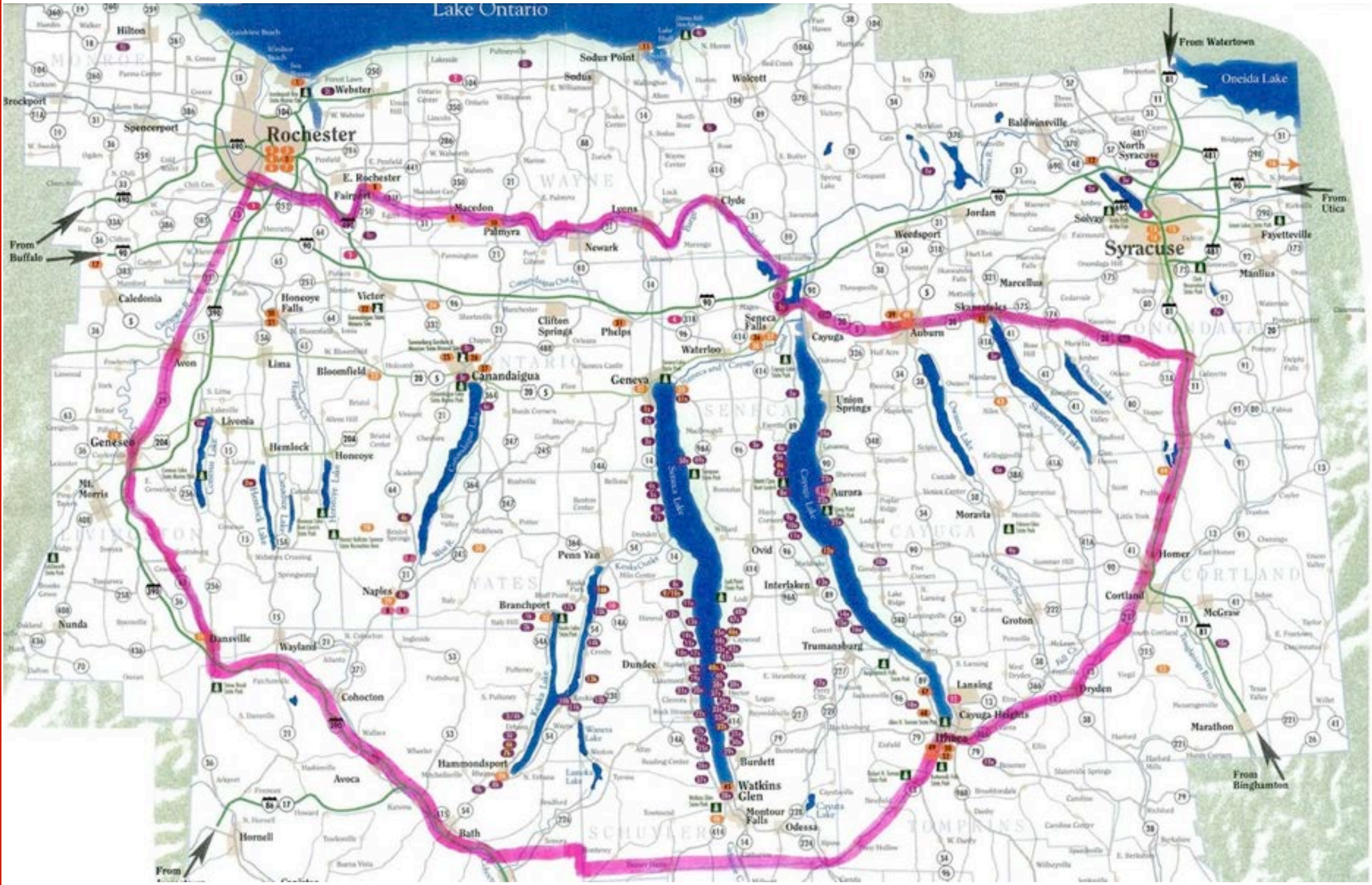
Fingerlakes AVA Wine Region (American Viticultural Area)



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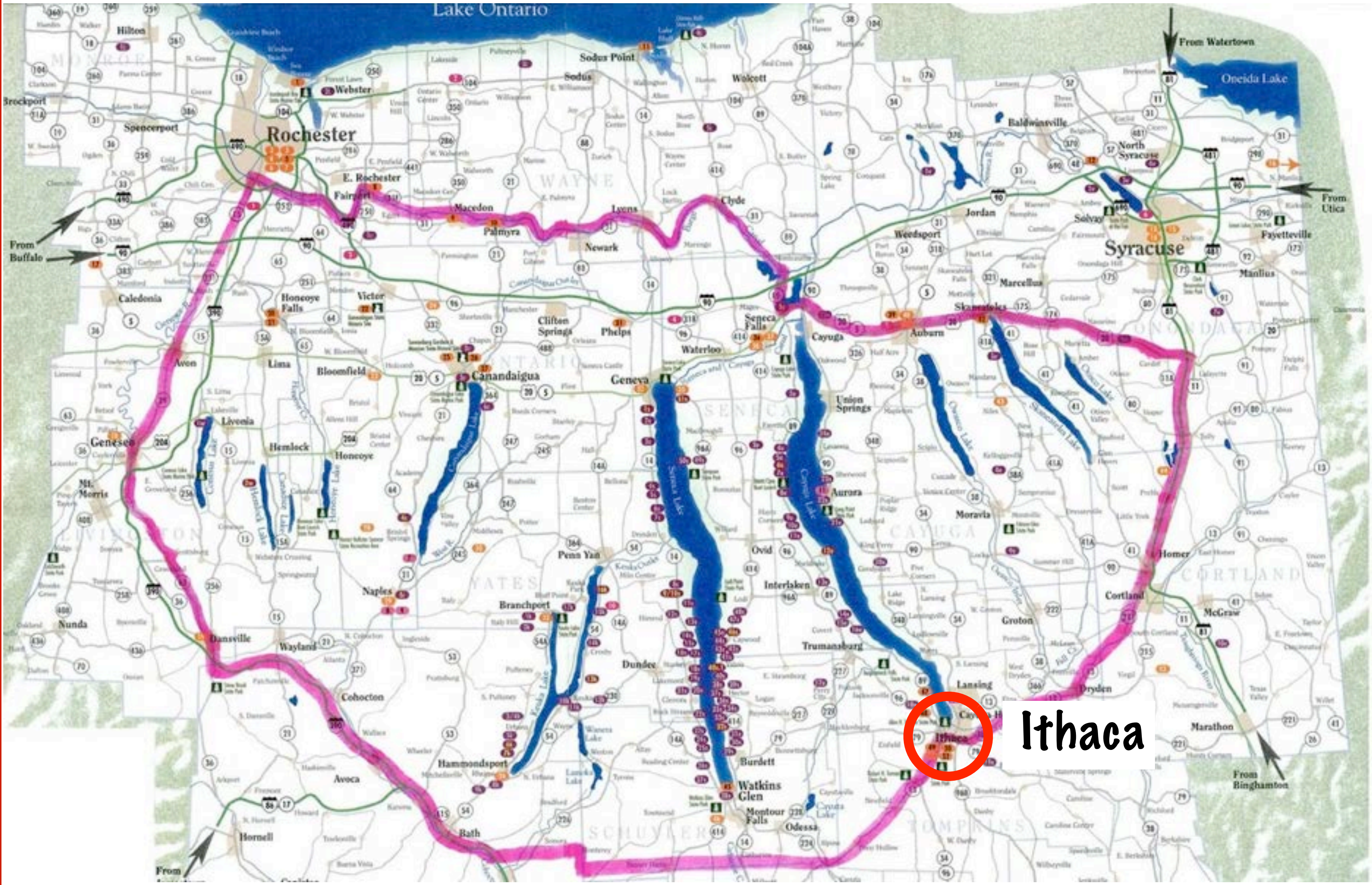

Ithaca is Gorges

Ithaca Falls

Taughannock Falls

Cornell Campus - Ithaca

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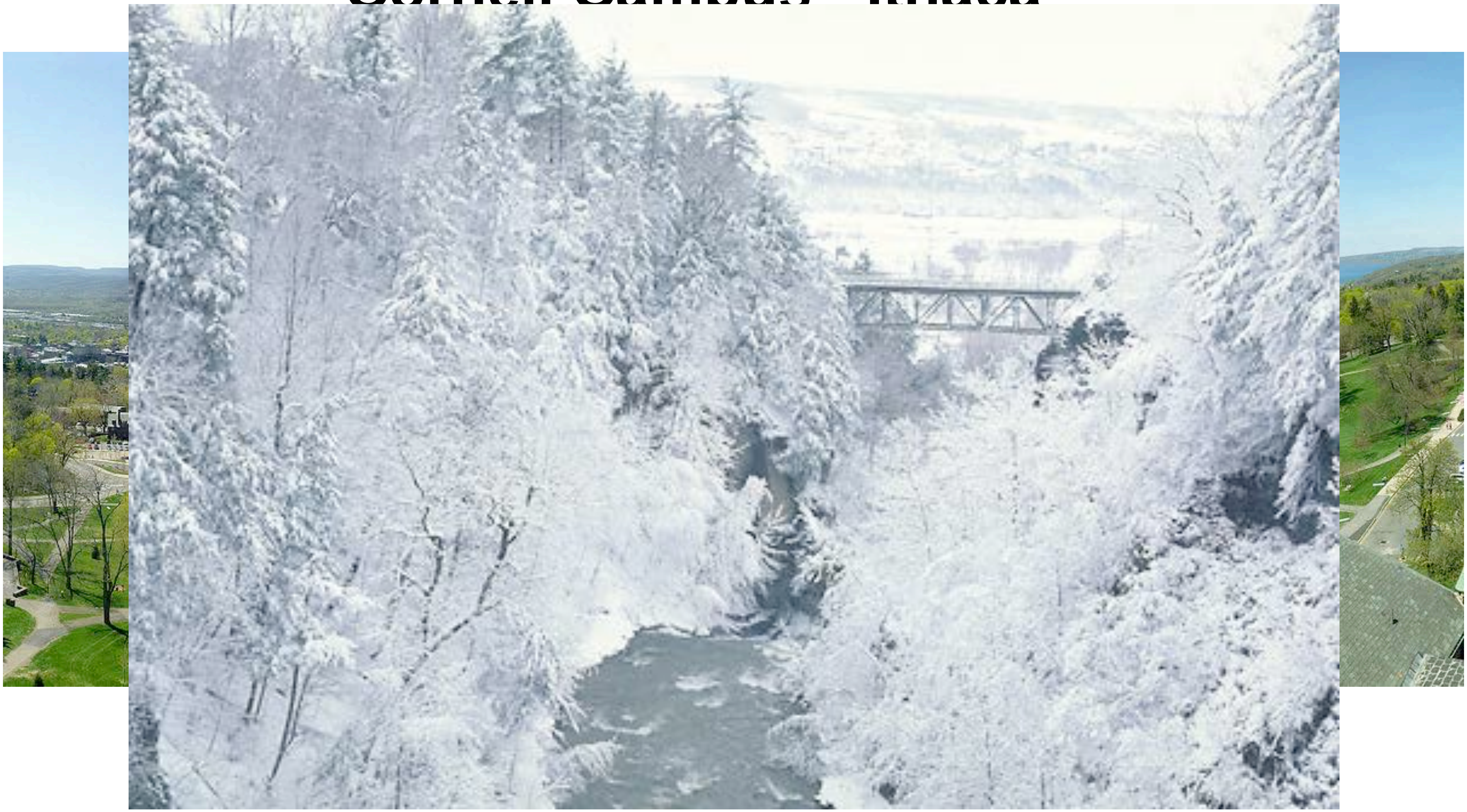

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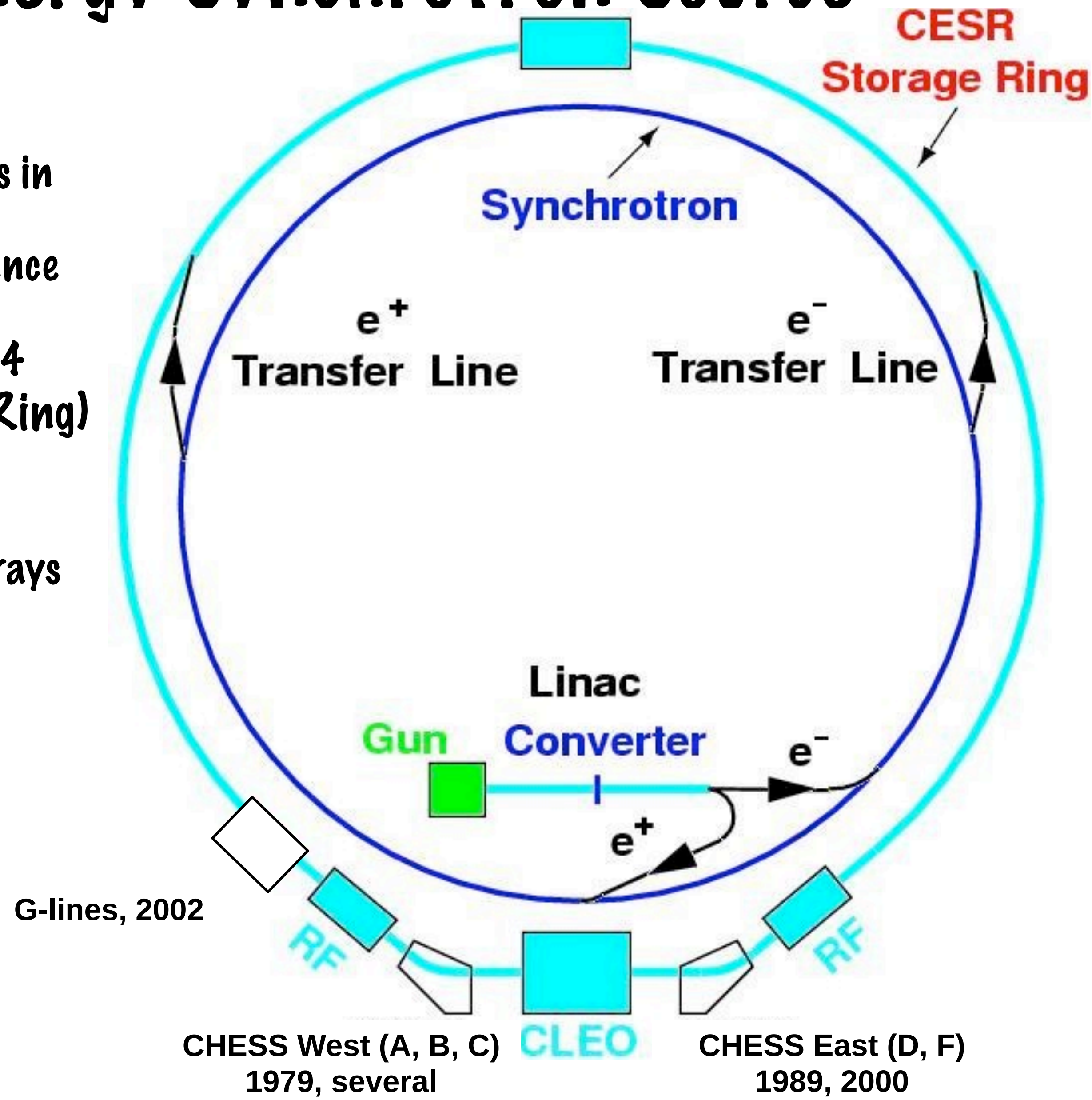

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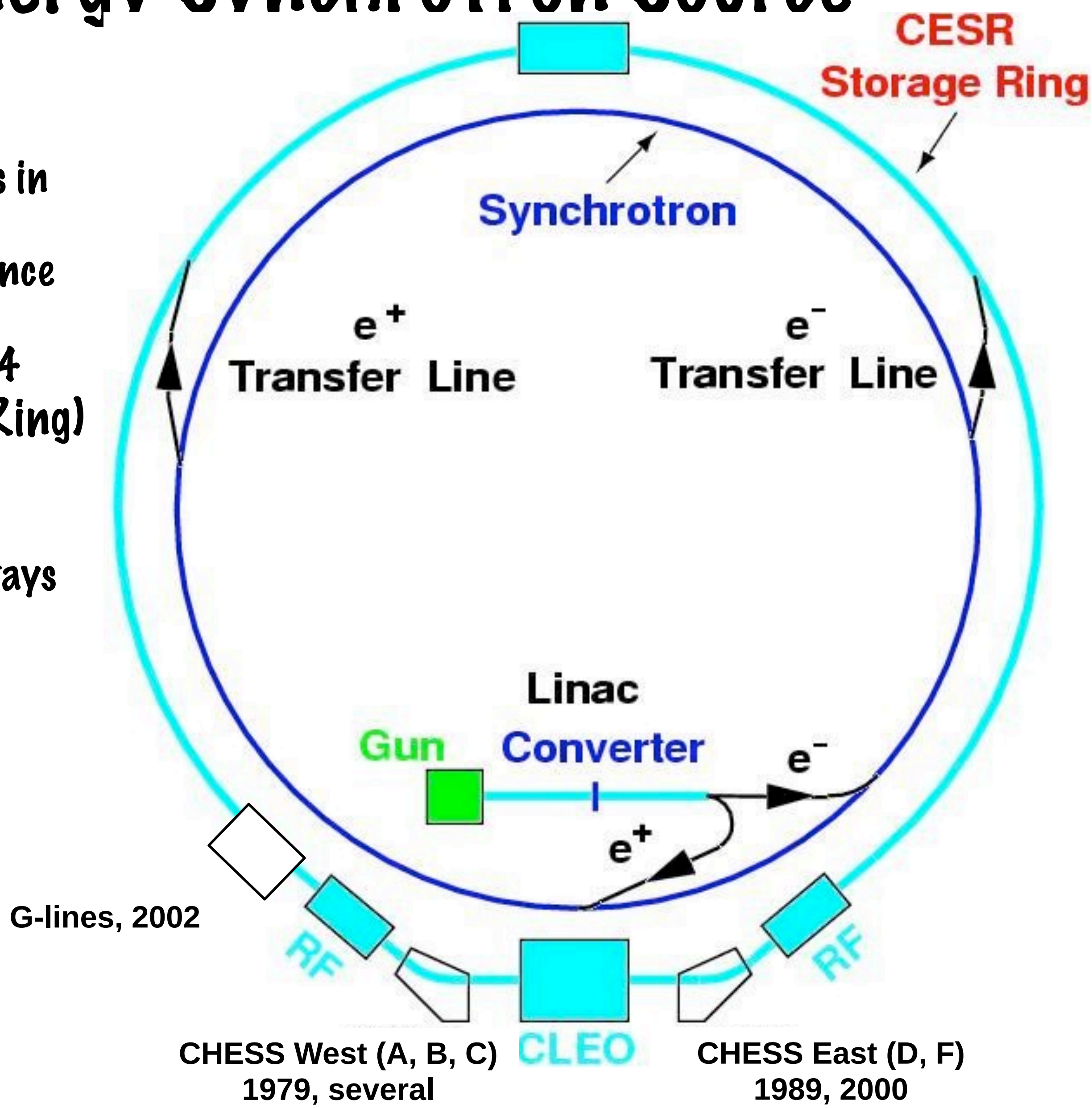

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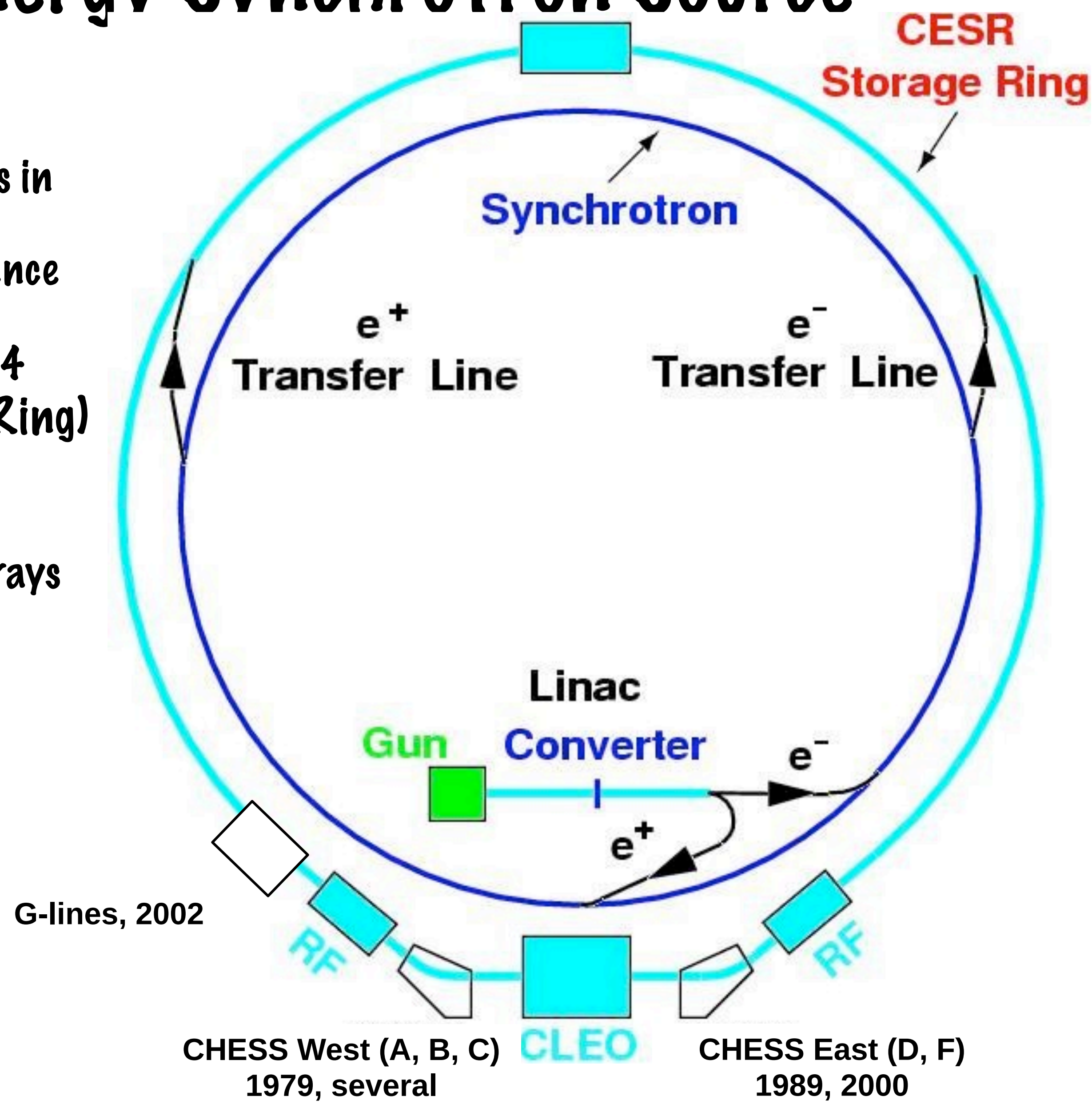

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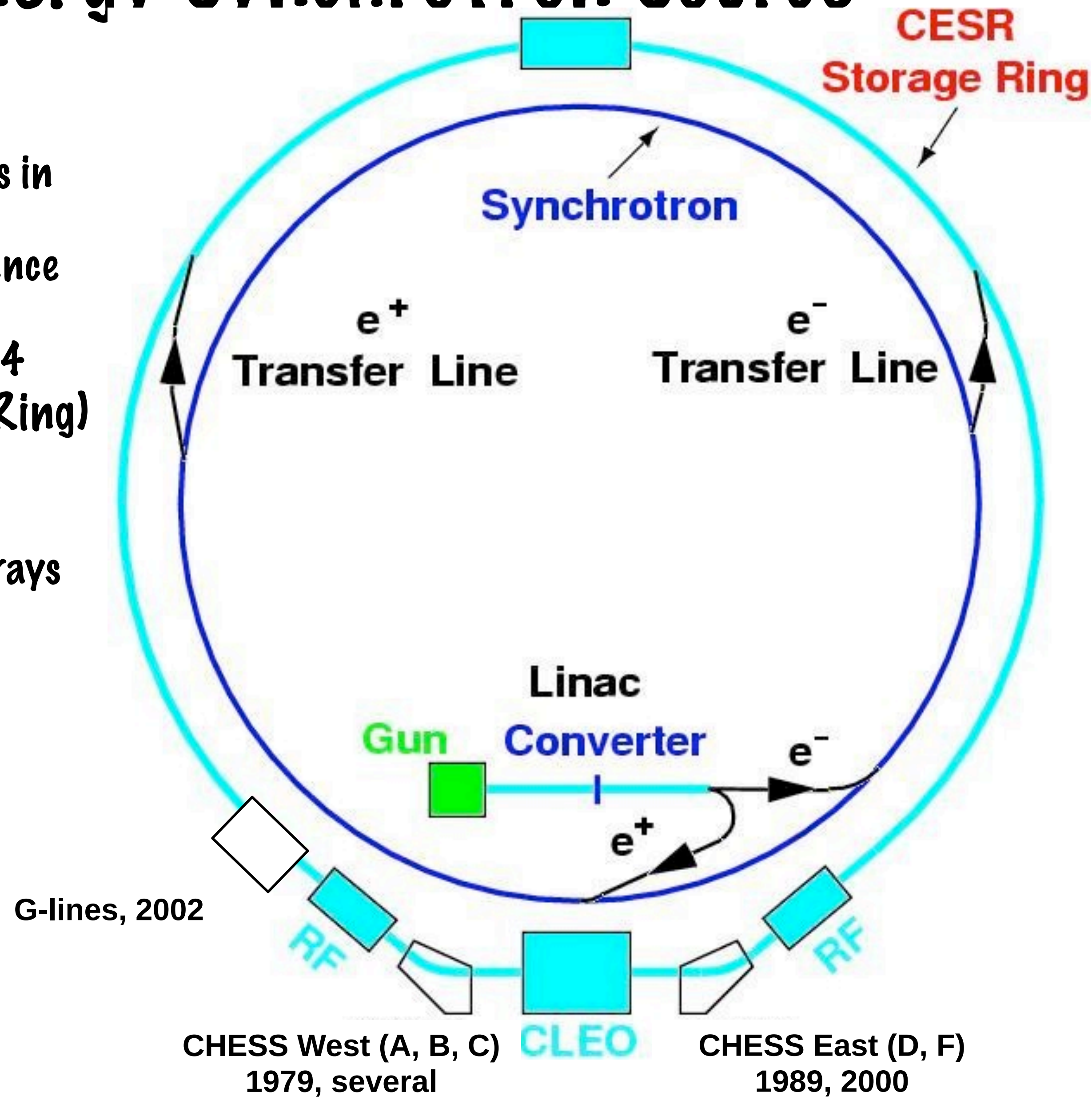

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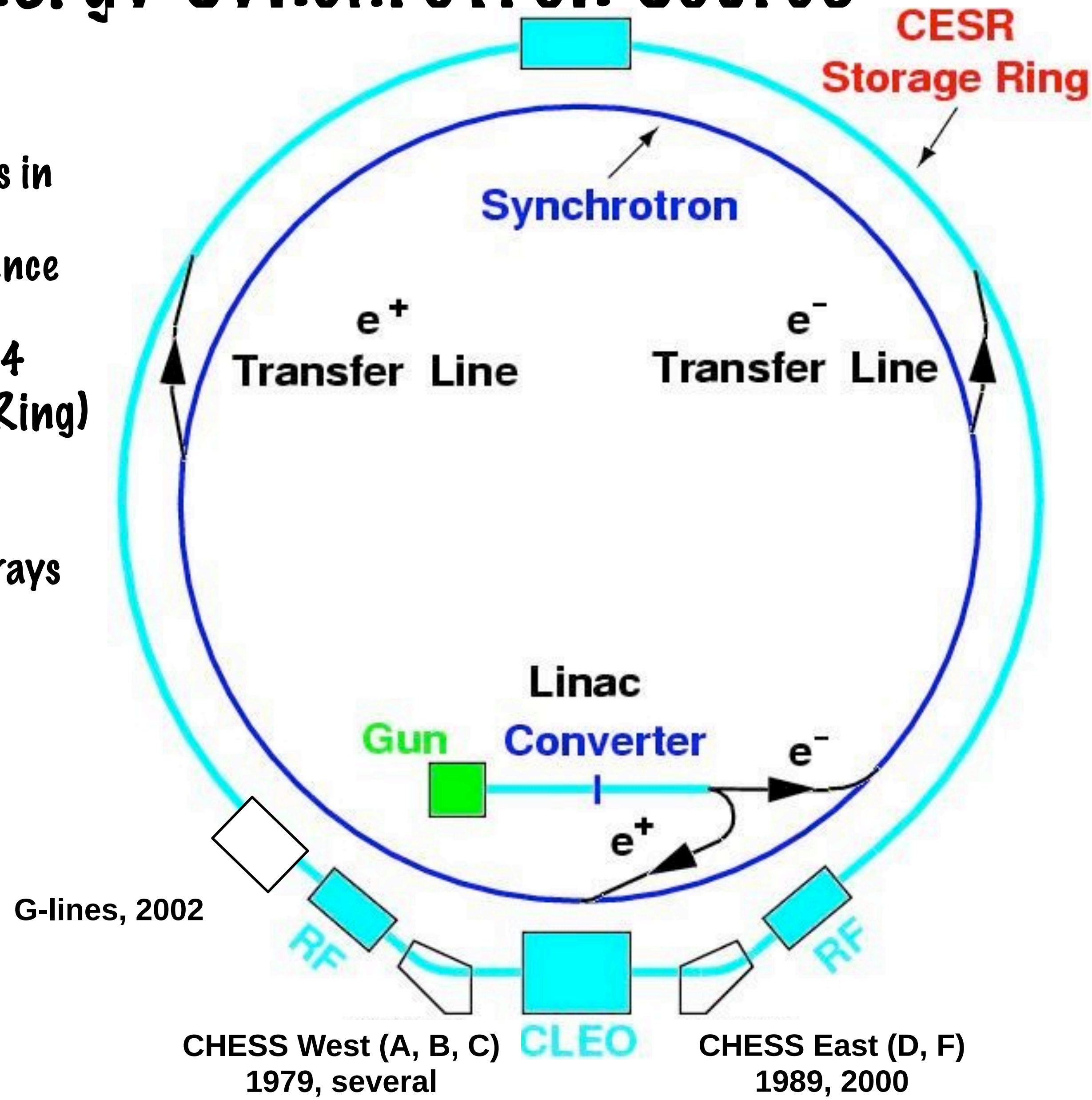

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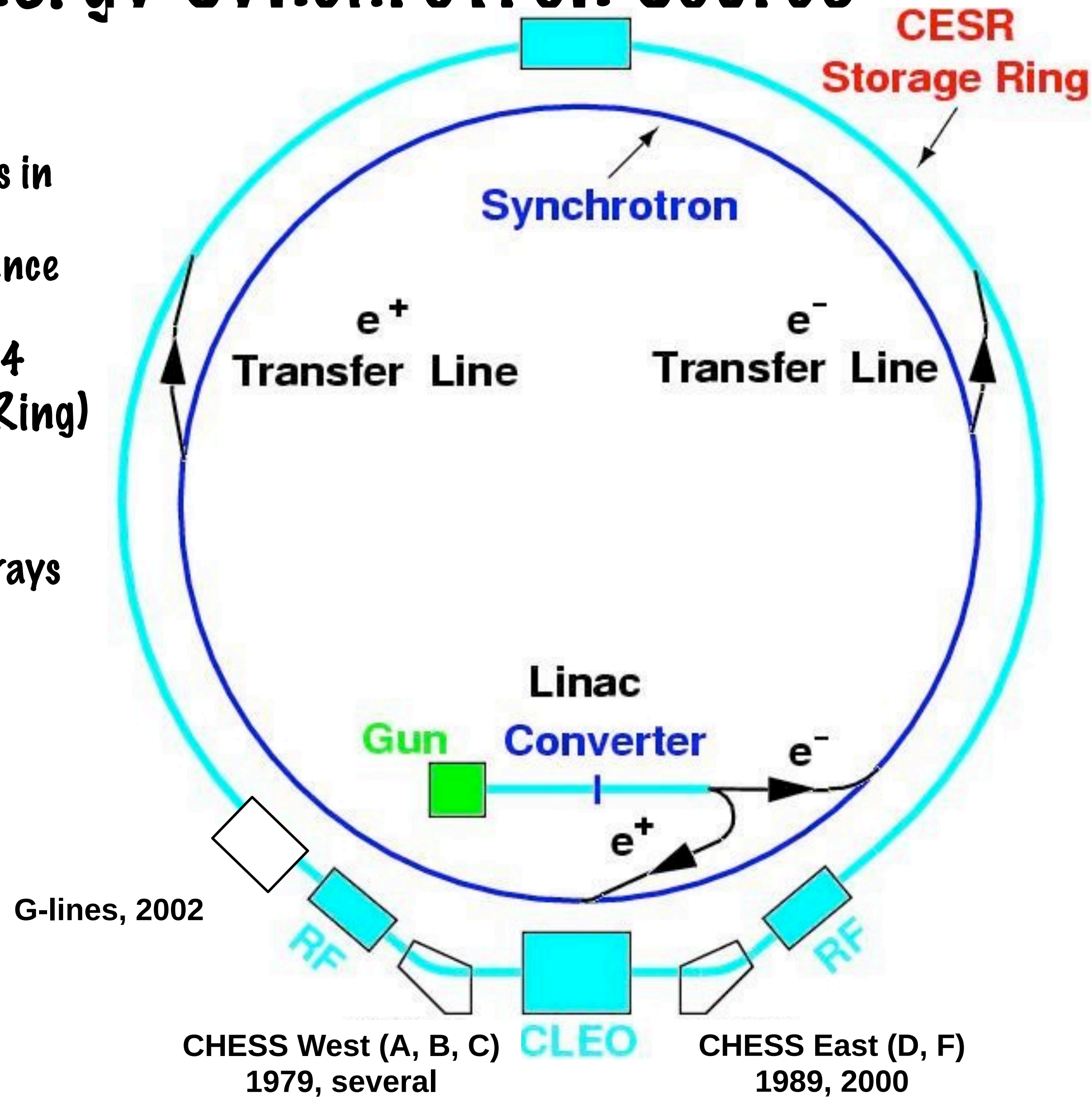

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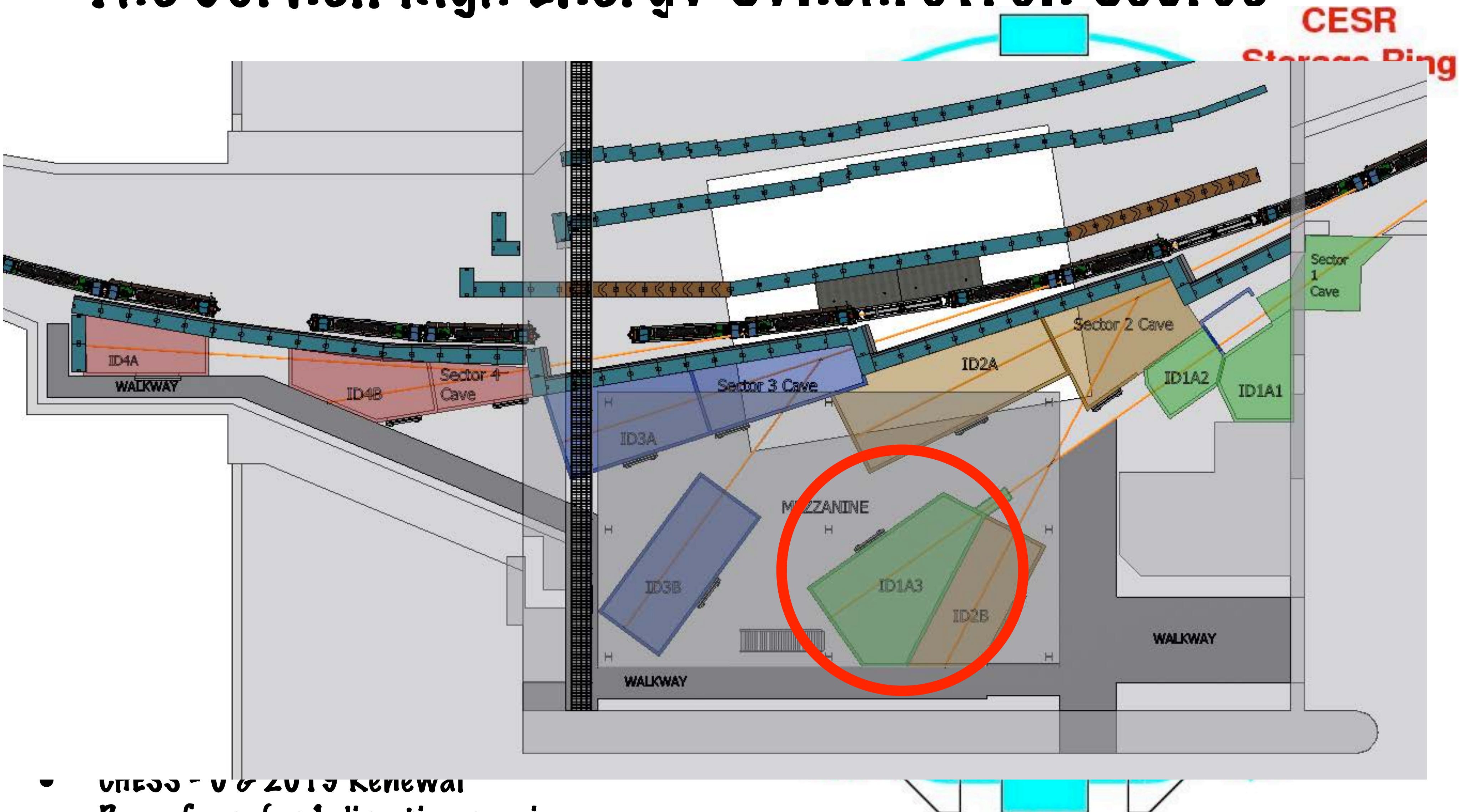

CLEO

CHESS West (A, B, C)
1979, several

CHESS East (D, F)
1989, 2000

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The World’s High Energy Synchrotrons

APS - Chicago

http://www.anl.gov

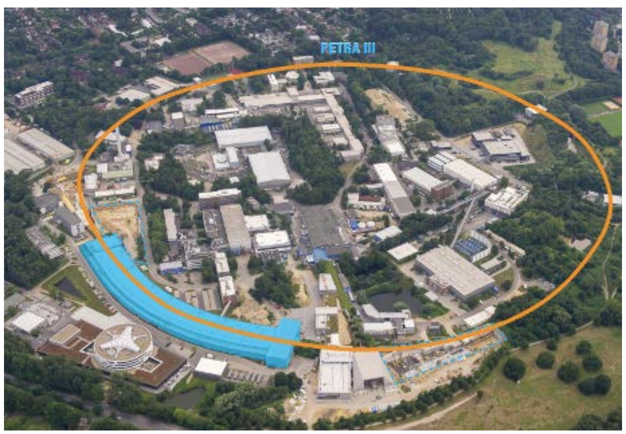
SPring-8 - Japan

http://www.riken.jp

ESRF - Grenoble

http://www.esrf.eu

Petra-III - Hamburg



http://http://photon-science.desy.de

CHESS

Outline

- Our Motivation
- Quick diffraction primer
- Examples
- A New CHESS
- Directions
- Tips

Our Motivation for Exploring High Energy X-ray Diffraction

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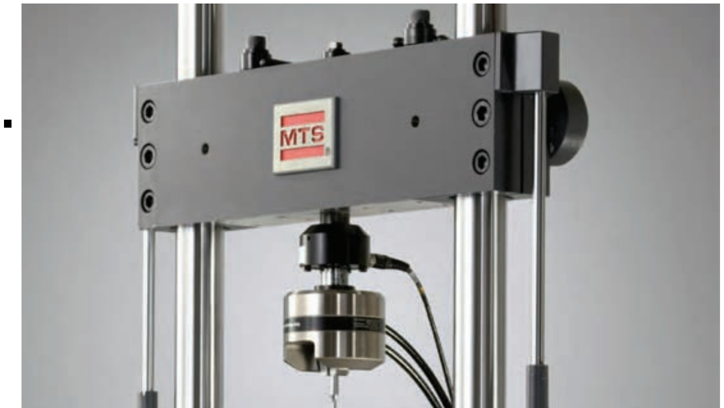

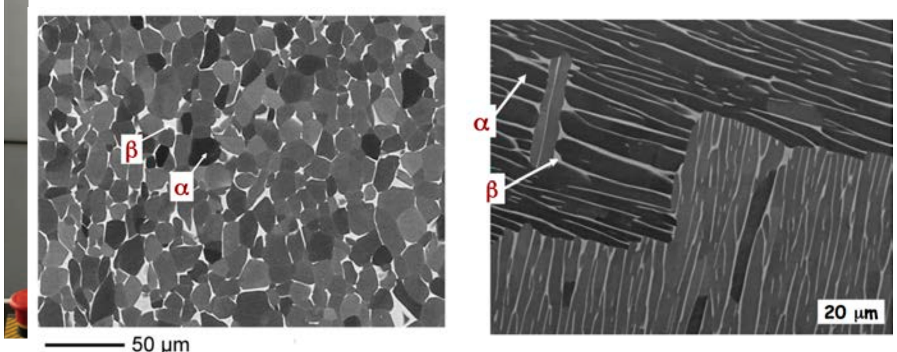

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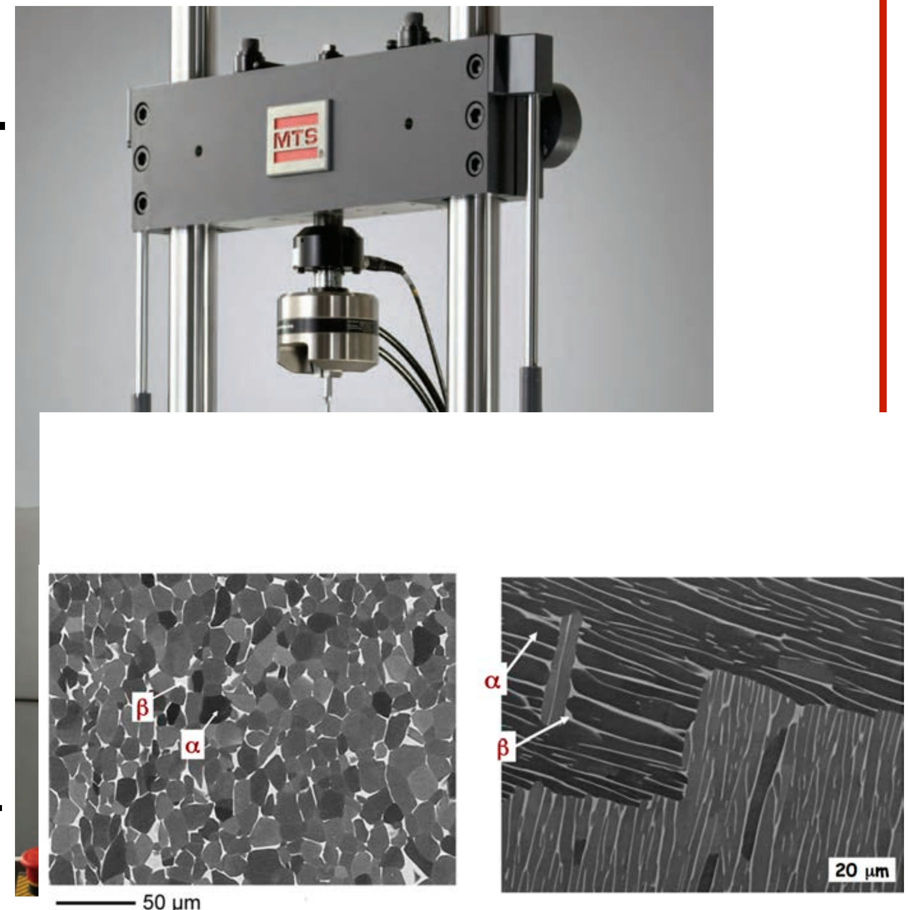

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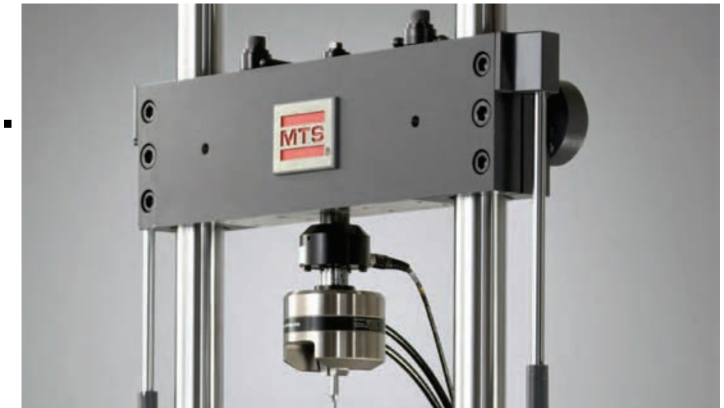

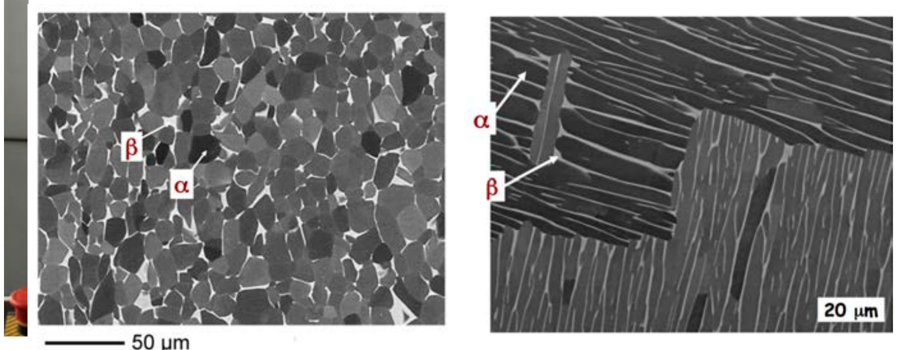

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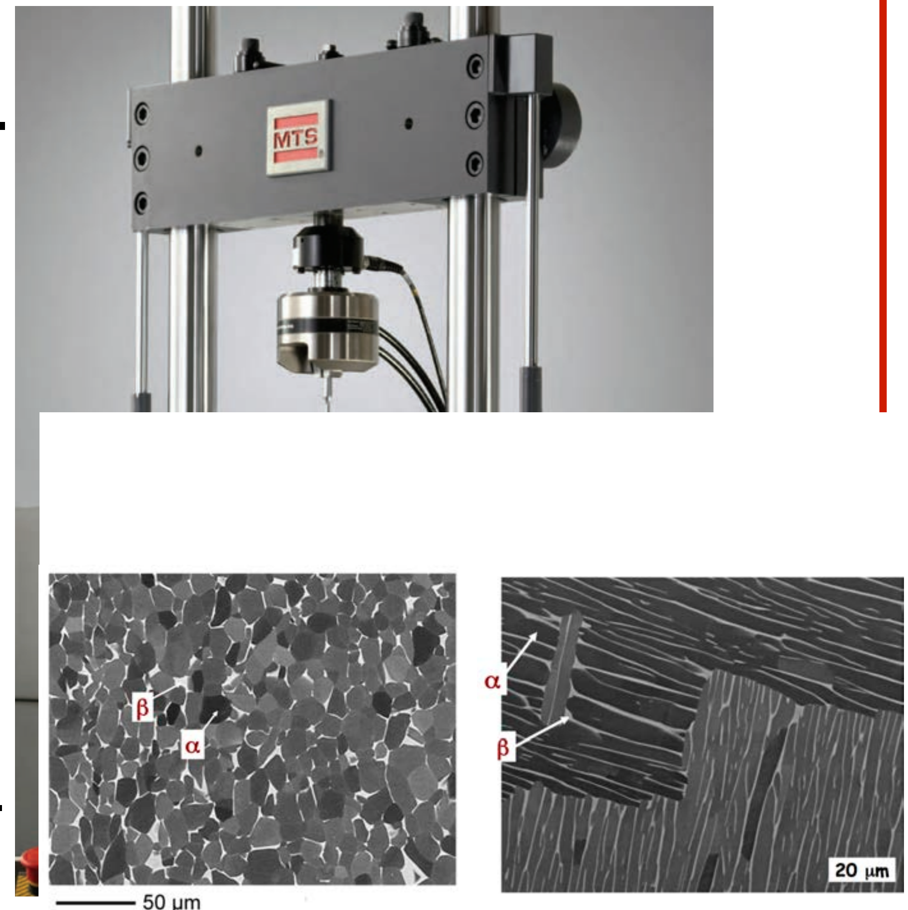

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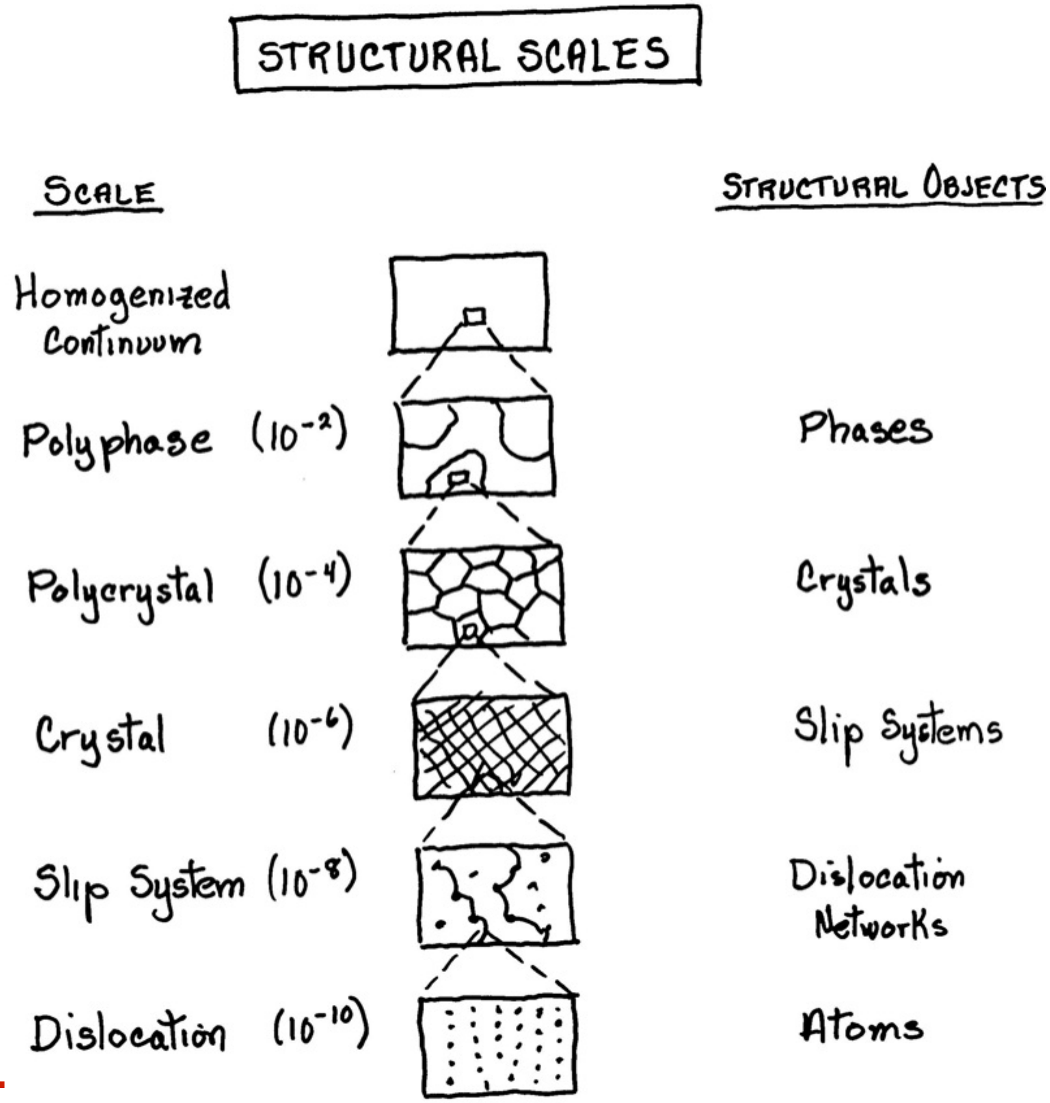
Hope: Microstructure-Based Modeling

Owen Richmond (US Steel, Alcoa), 1985



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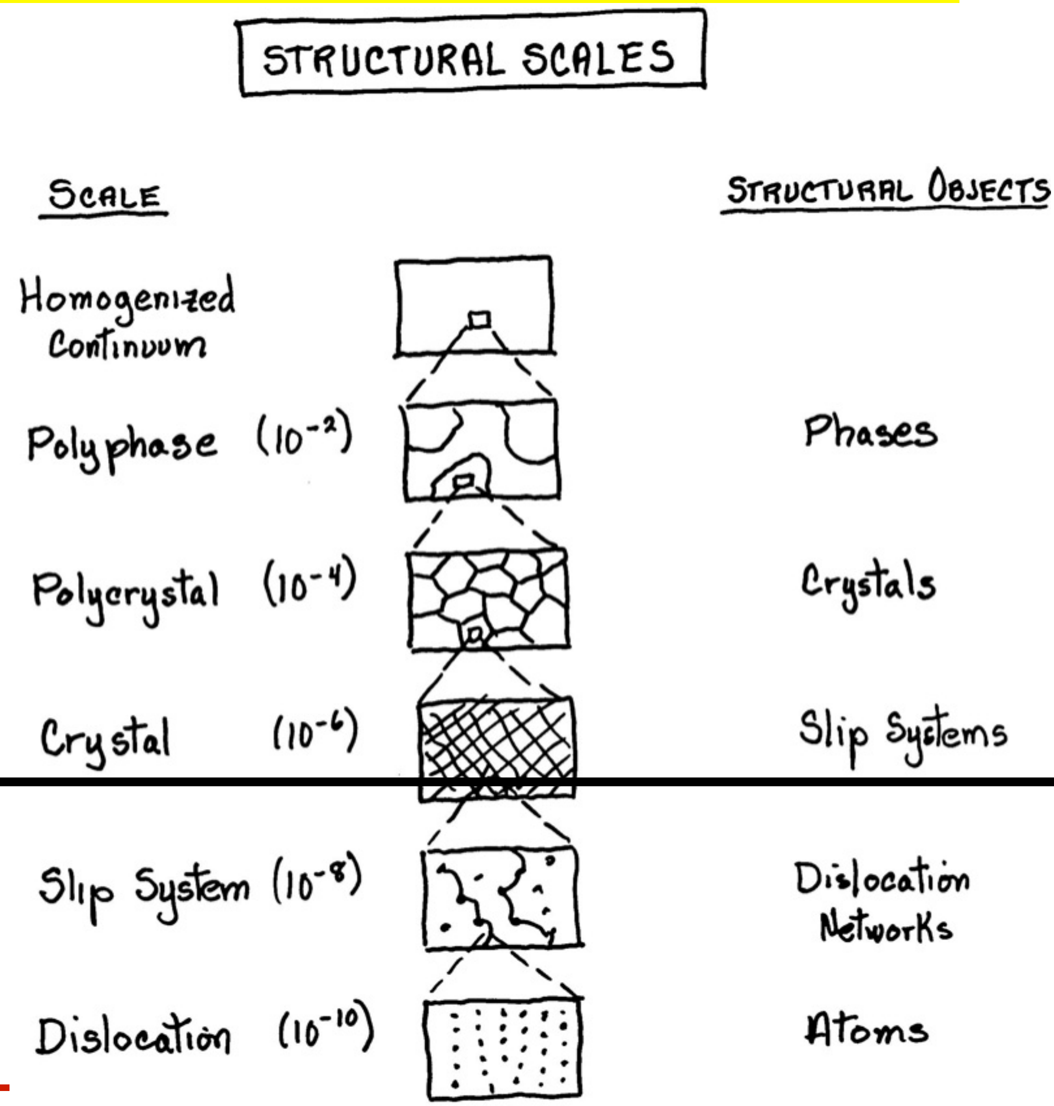

"Polycrystal" Models

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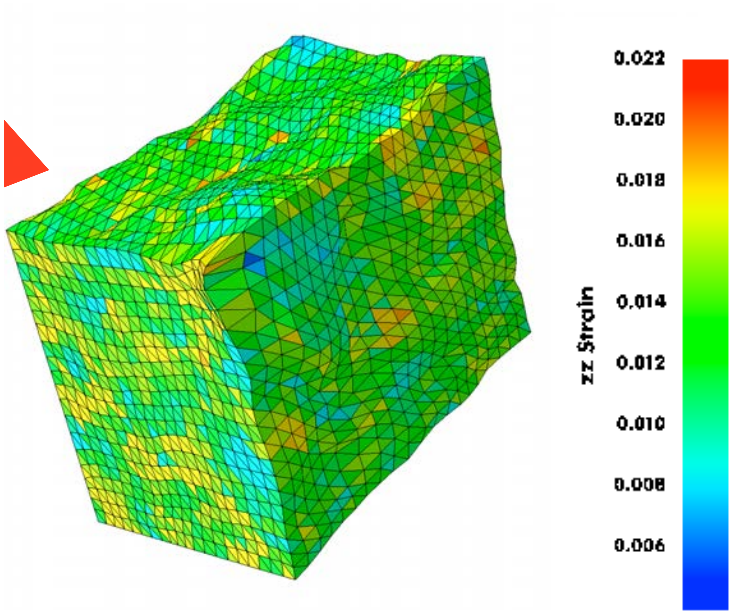

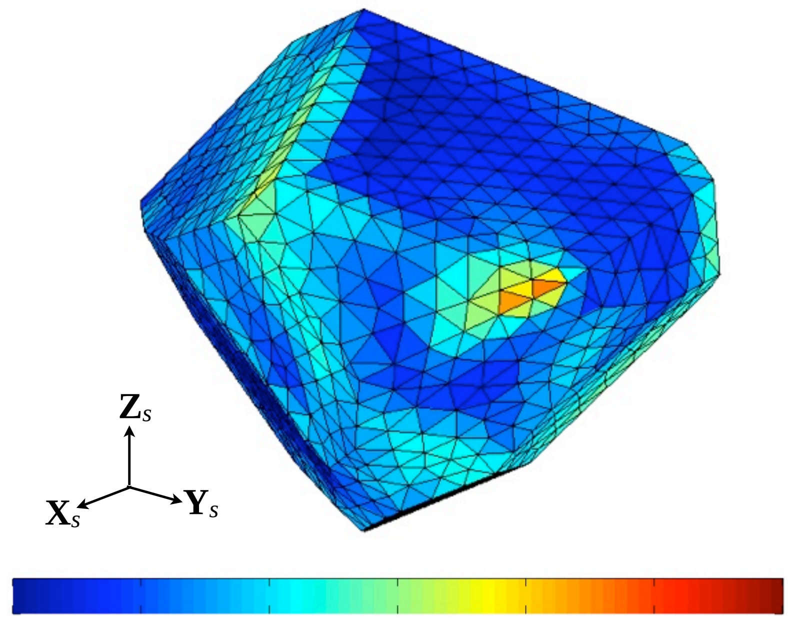

Crystal Scale Finite Elements - FEpX
Paul Dawson

Hope: Microstructure-Based Modeling

- Multiscale modeling research is "rampant" - very little crystal scale mechanical testing data, however
- Overarching idea: use High Energy X-ray Diffraction (HEXD) data and in situ loading with FEM representation of microstructure to understand crystal scale material behavior
- Processing and Performance - induced CHANGES in unit cell to understand material response
- Merge model with diffraction data:

M.P. Miller and P.R. Dawson, Current Opinion in Solid State & Materials Science, 18, 286-299, 2014.

Instructional Videos From CHESS

- **Google: Chess x-ray micromechanics**
- **https://www.youtube.com/watch?v=kYEboNz423A&t=9s**

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- Obtaining in situ (real time) information over an entire polycrystalline aggregate is the main advantage of doing diffraction at a light source
- Be careful, it is easy to get "hooked" on diffraction and light sources and late nights and multiple days at the beam line and the tool becomes your science... it's actually pretty great!

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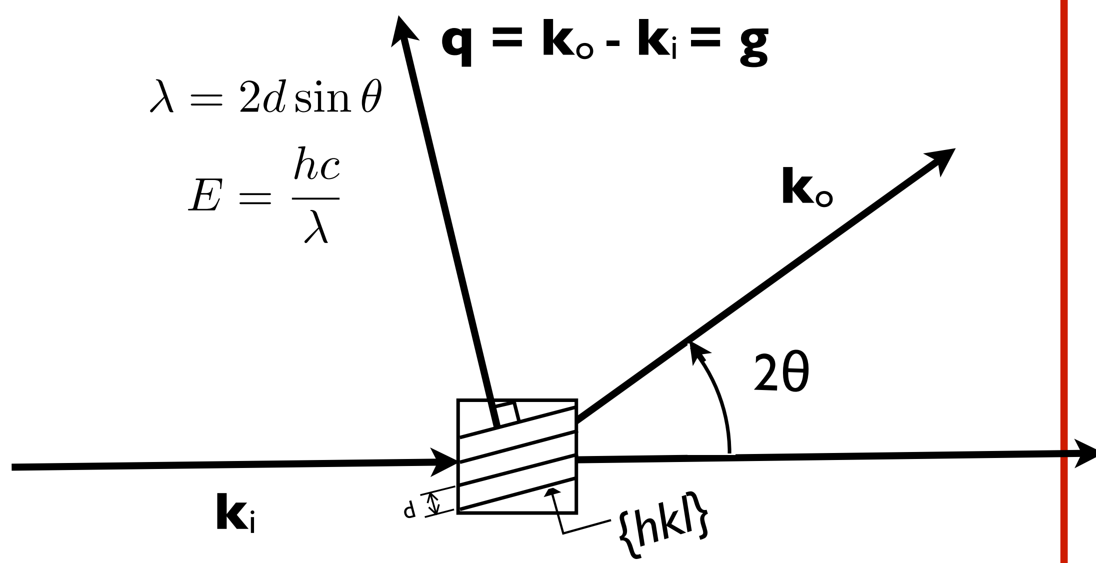

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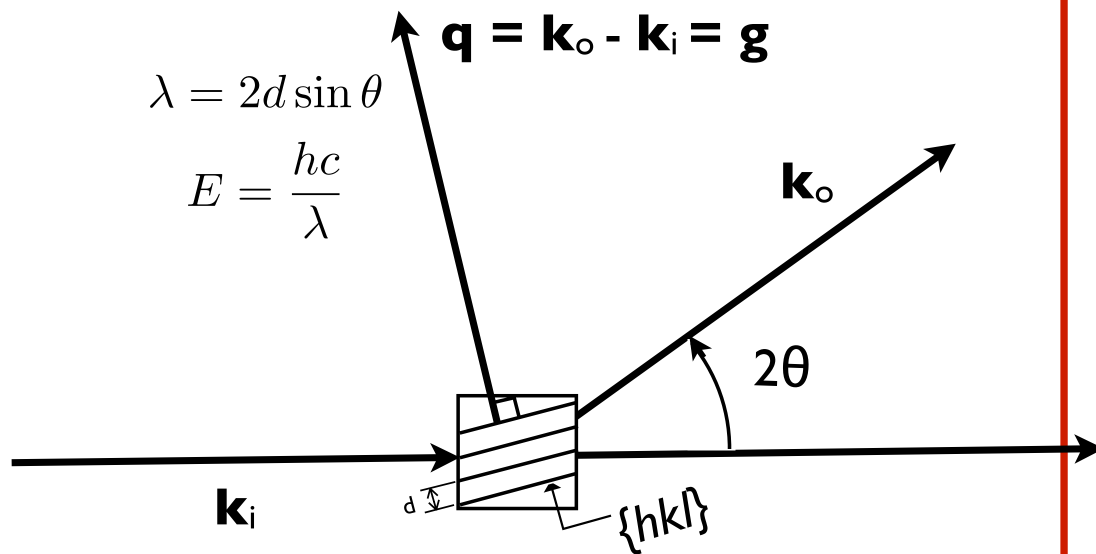

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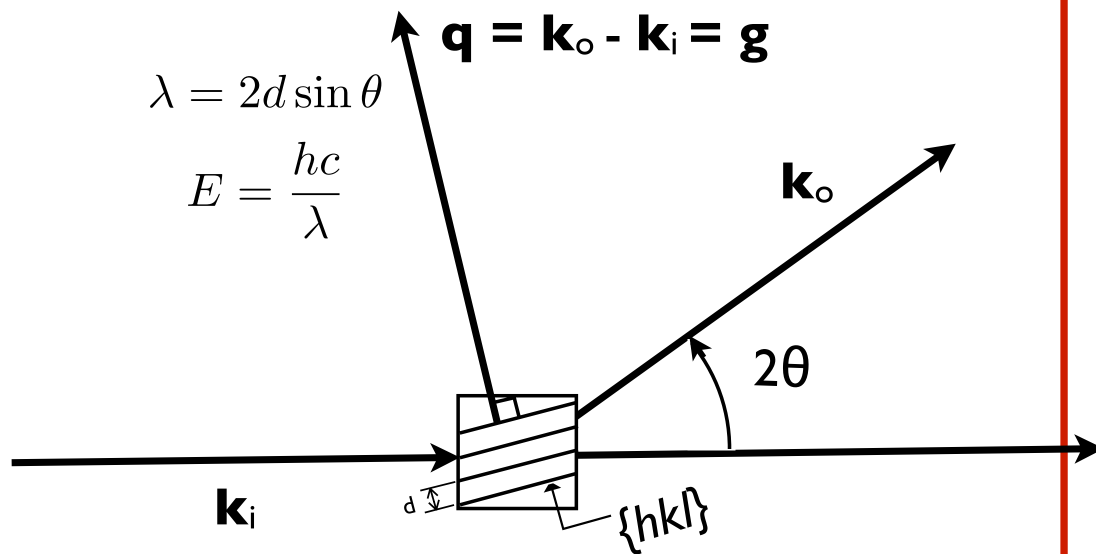

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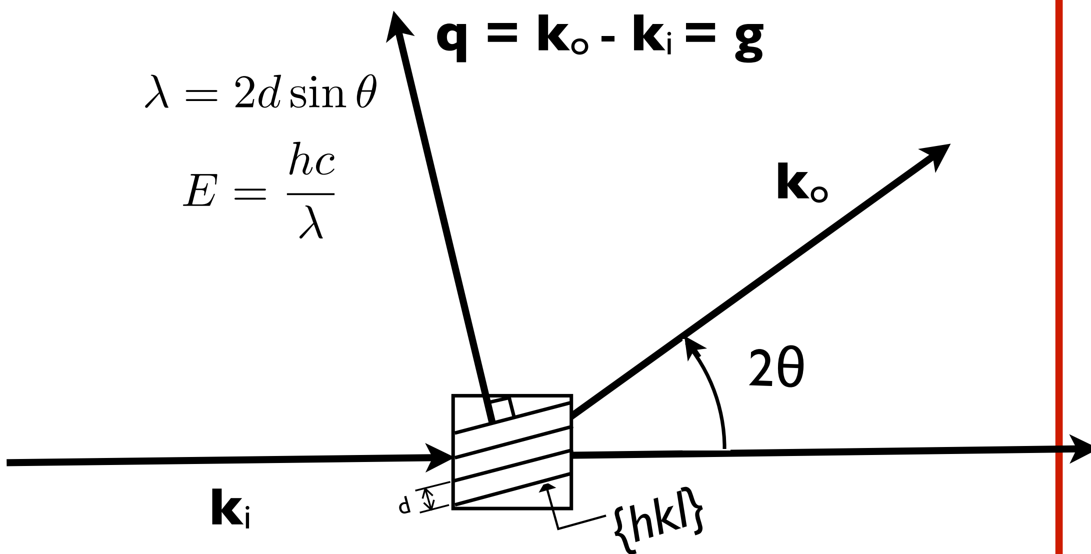

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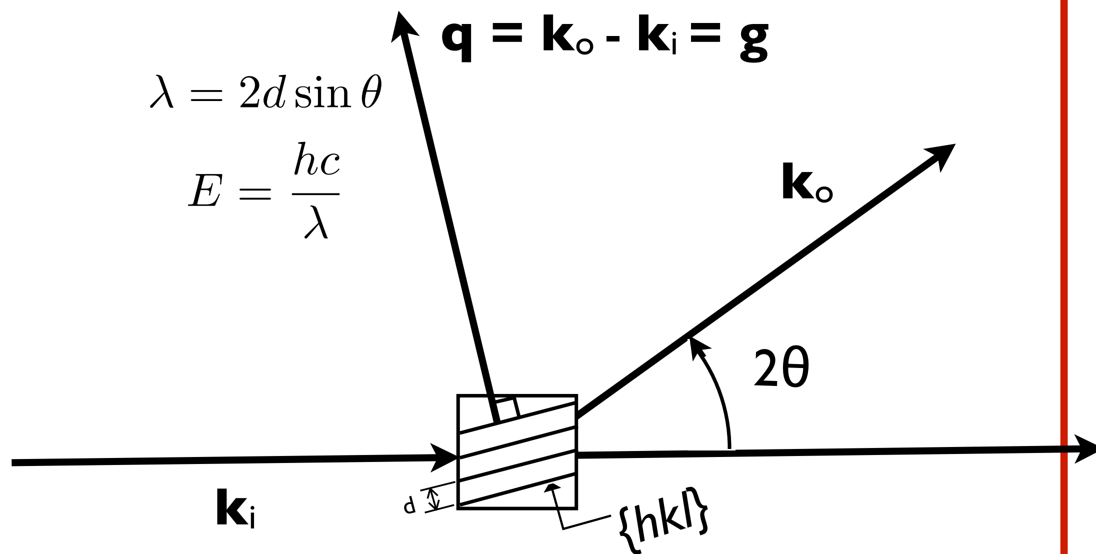

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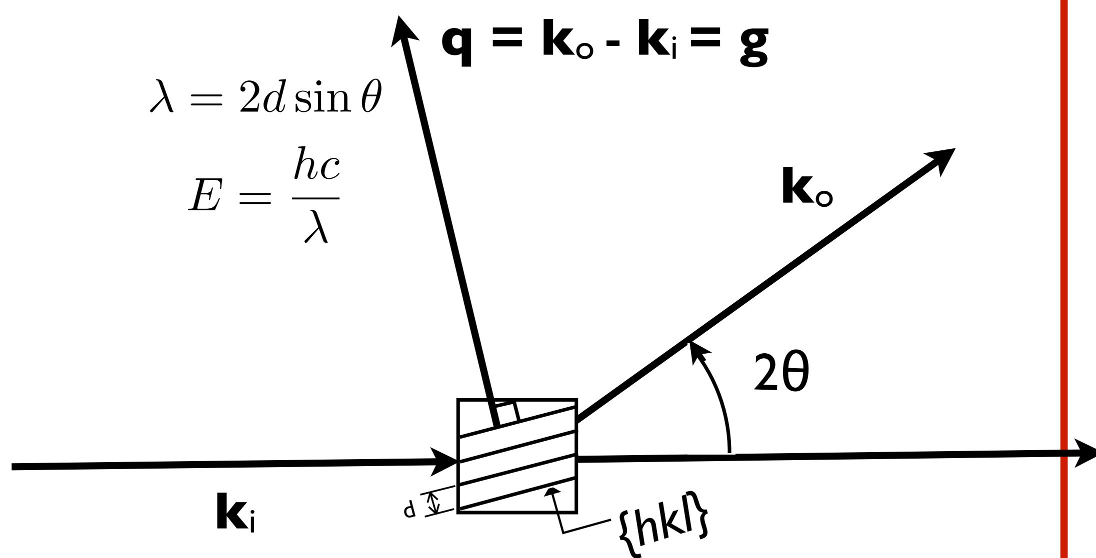

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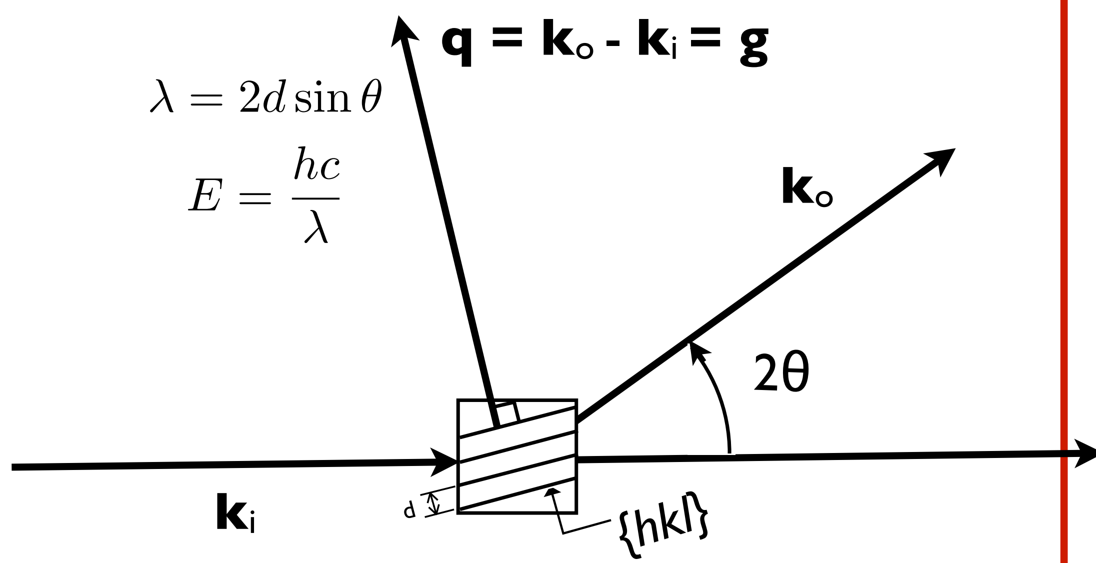

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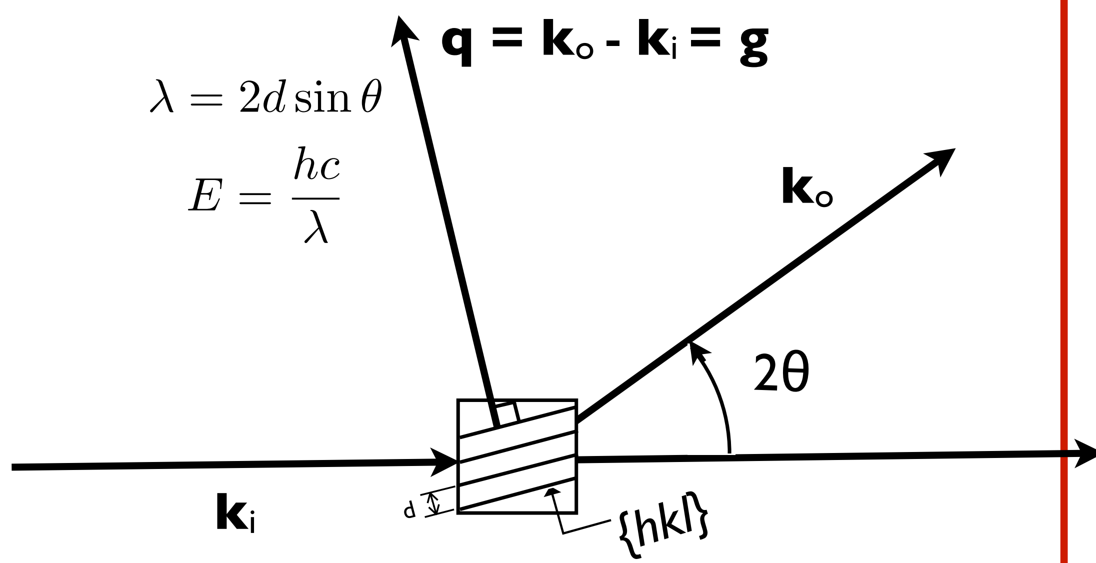

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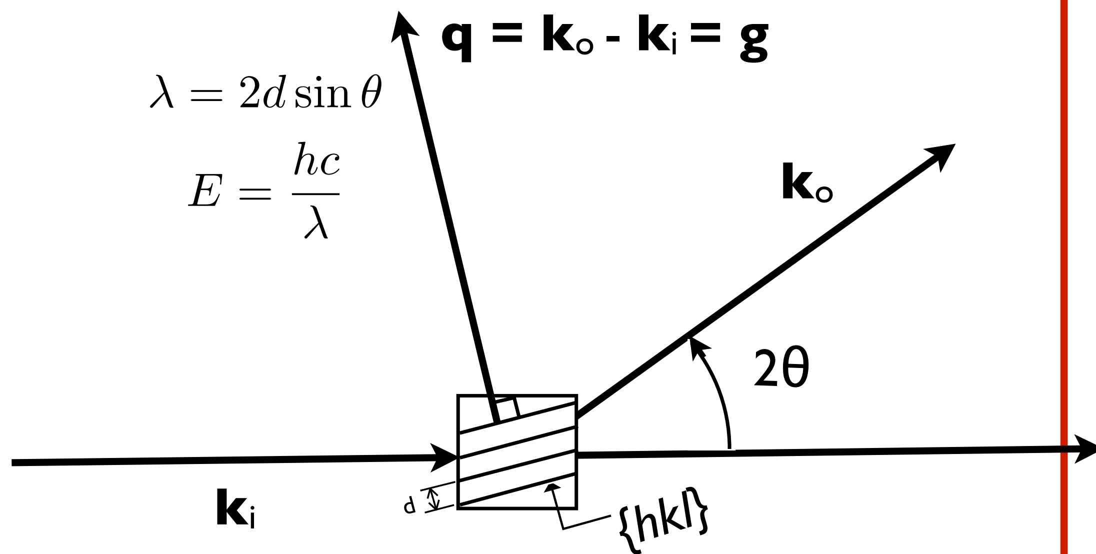

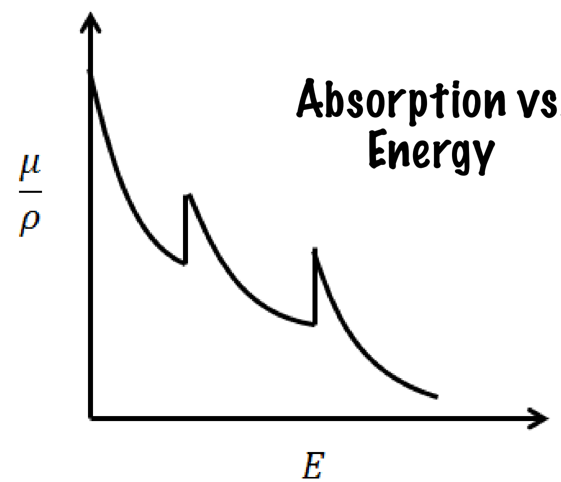

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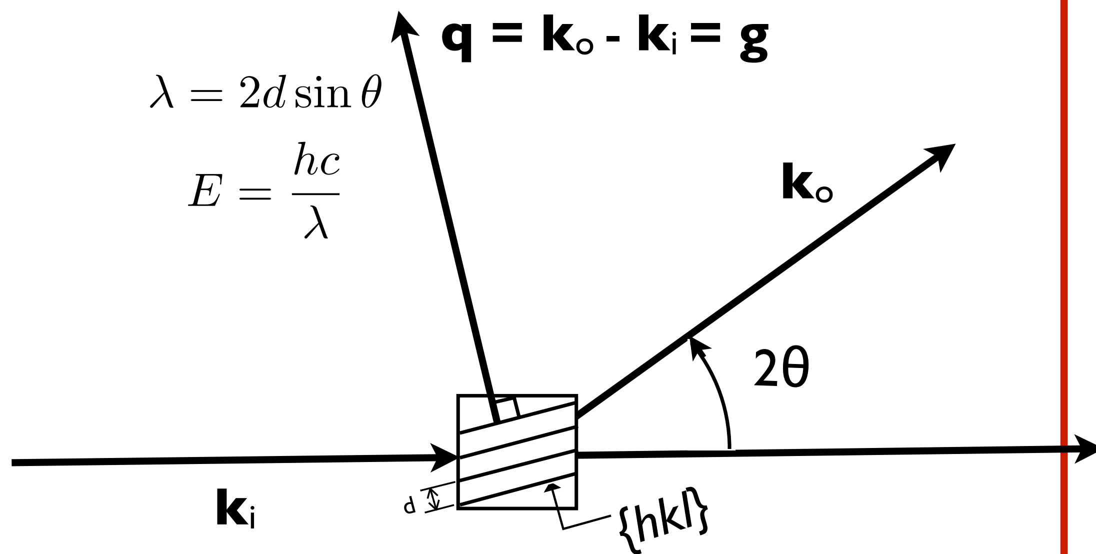

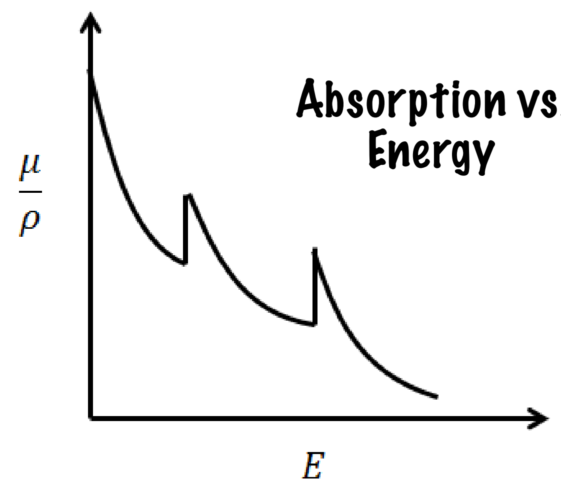

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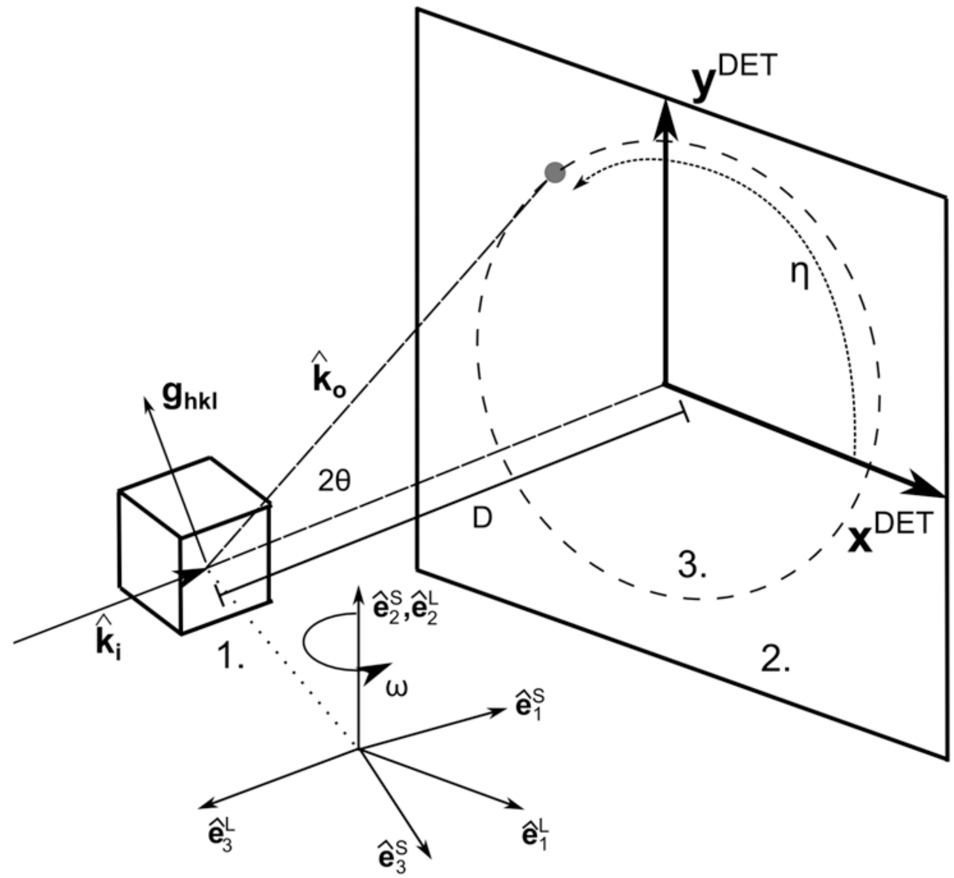

1. Diffracting Crystal
2. Area Detector
3. Debye-Scherrer Ring

E

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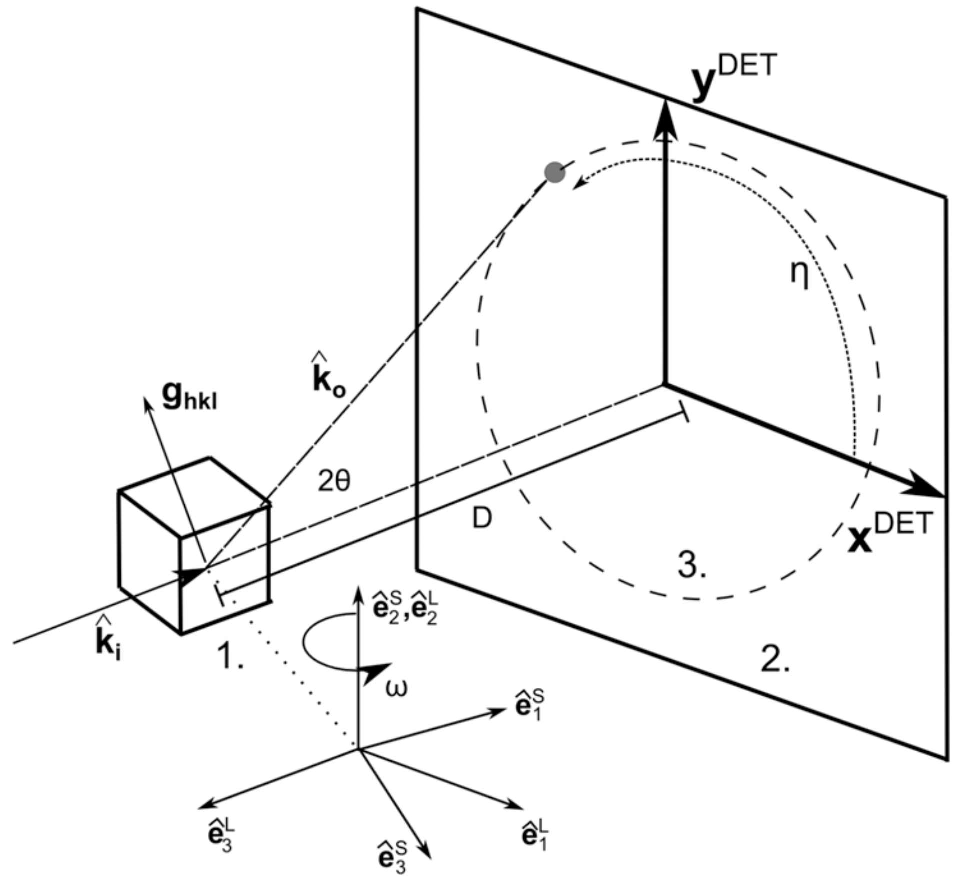

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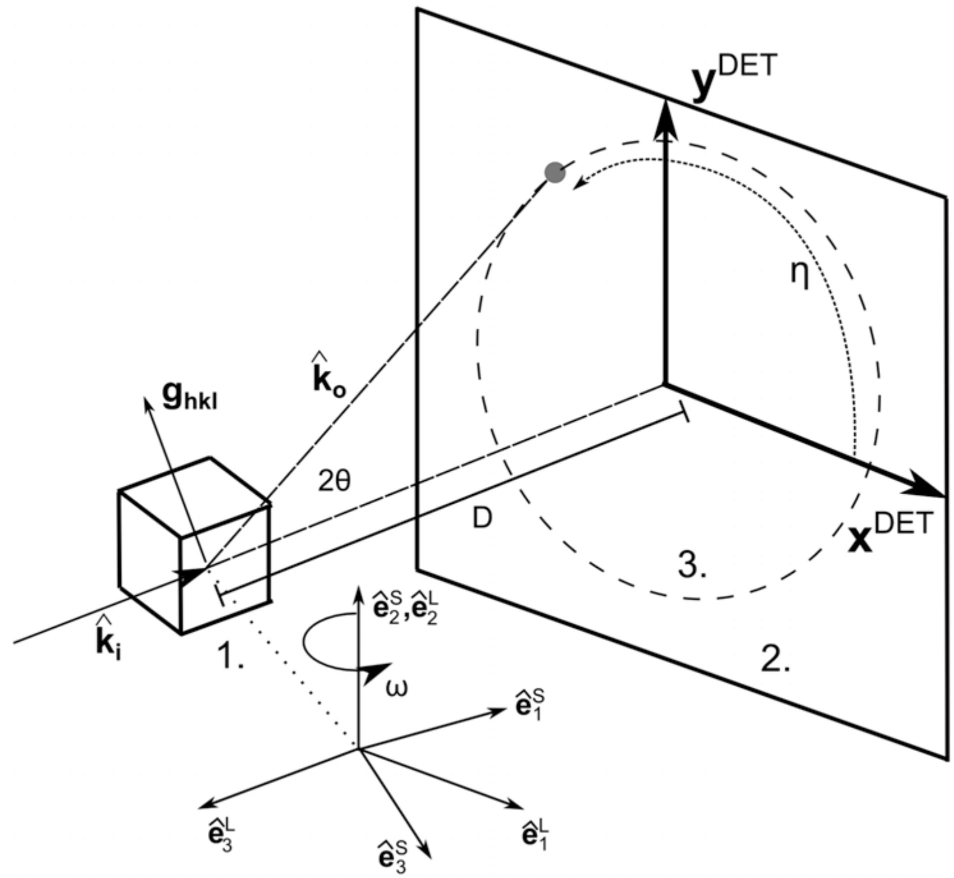

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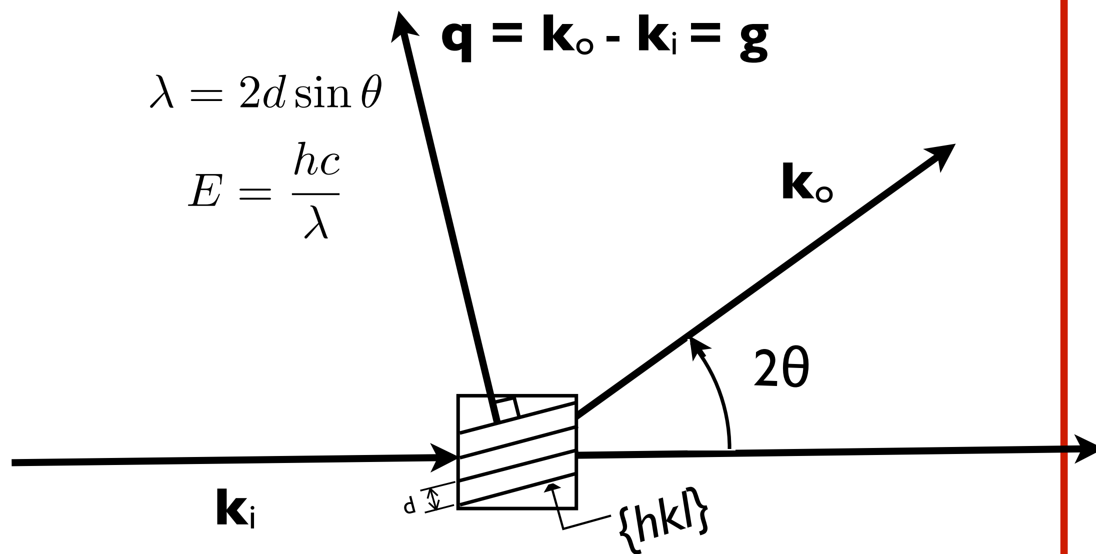

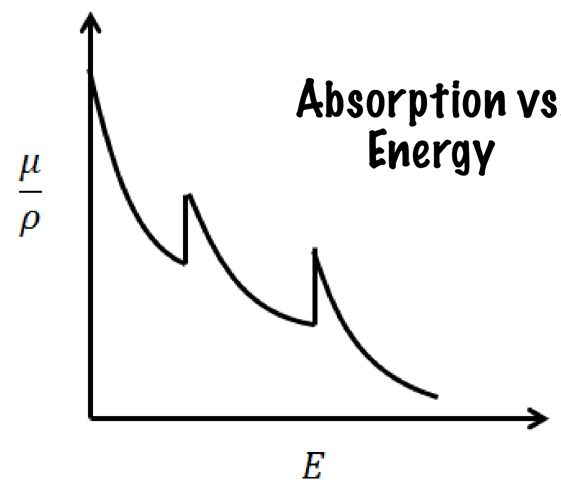

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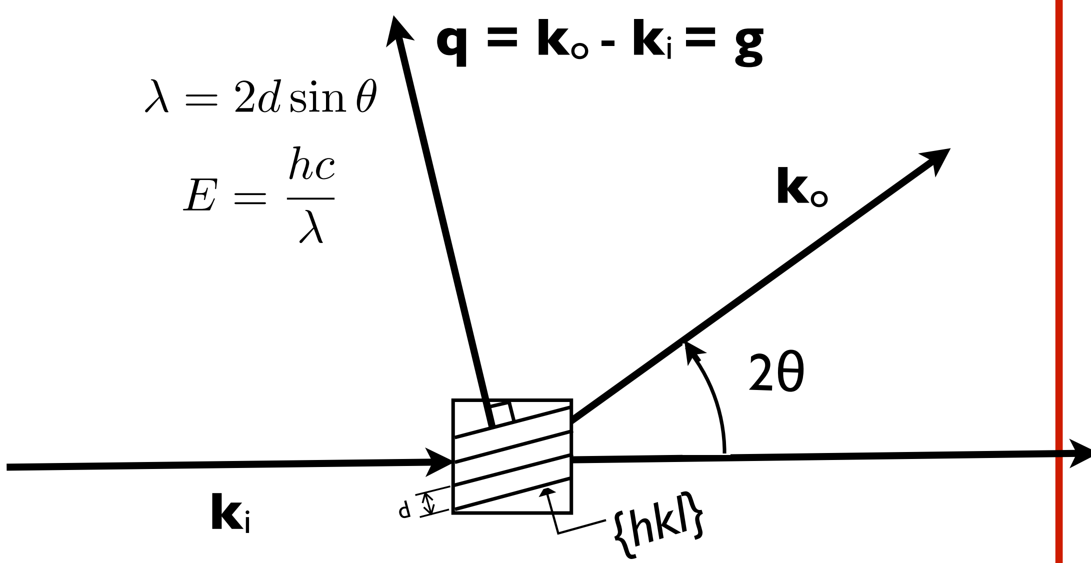

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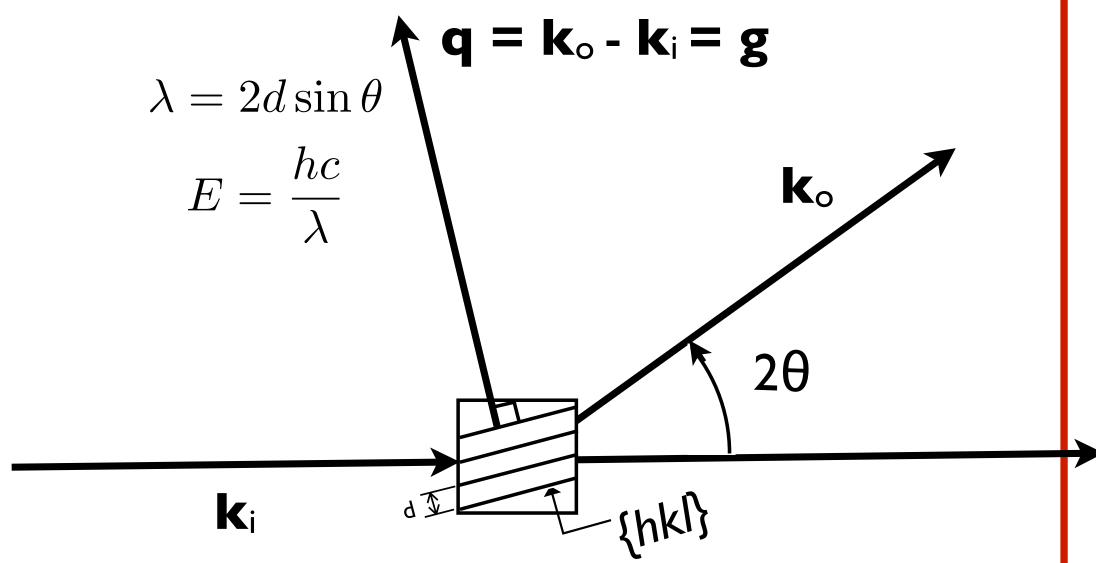

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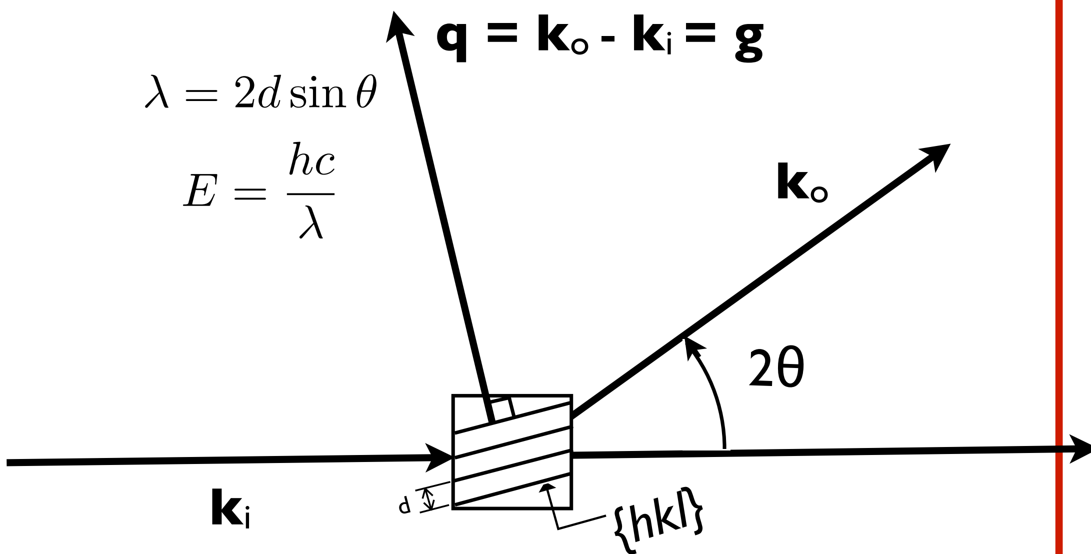

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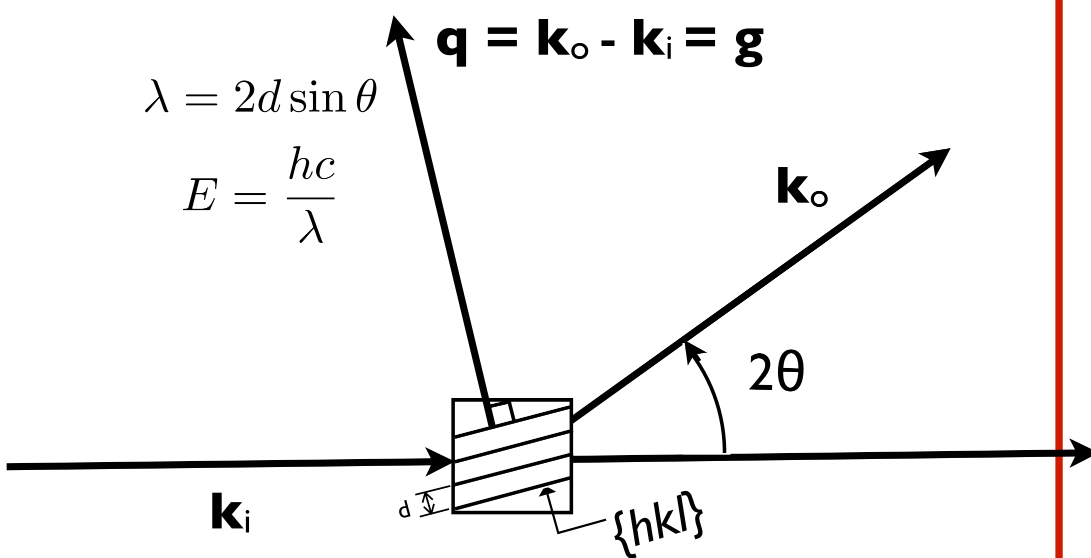

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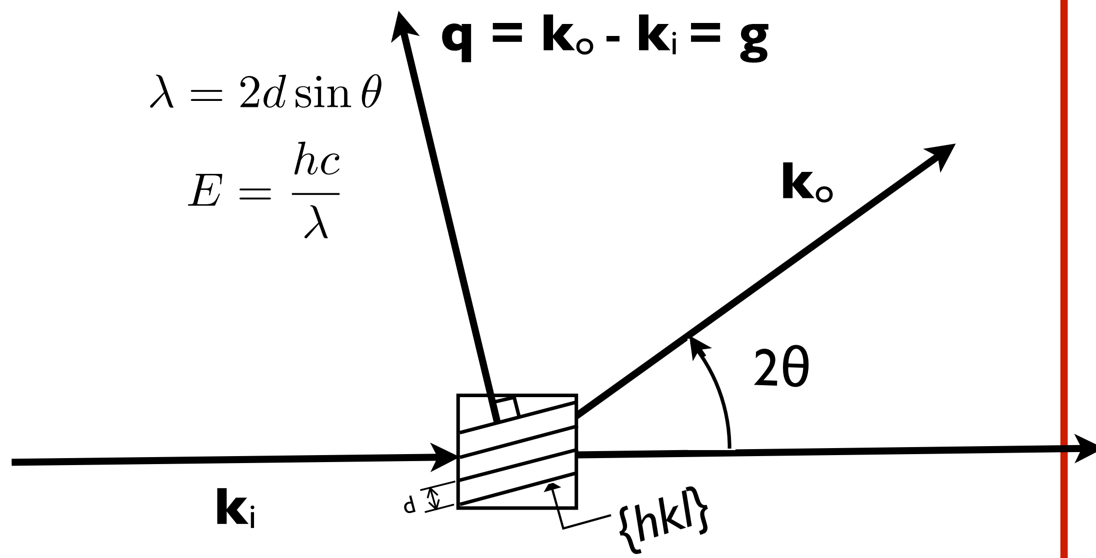

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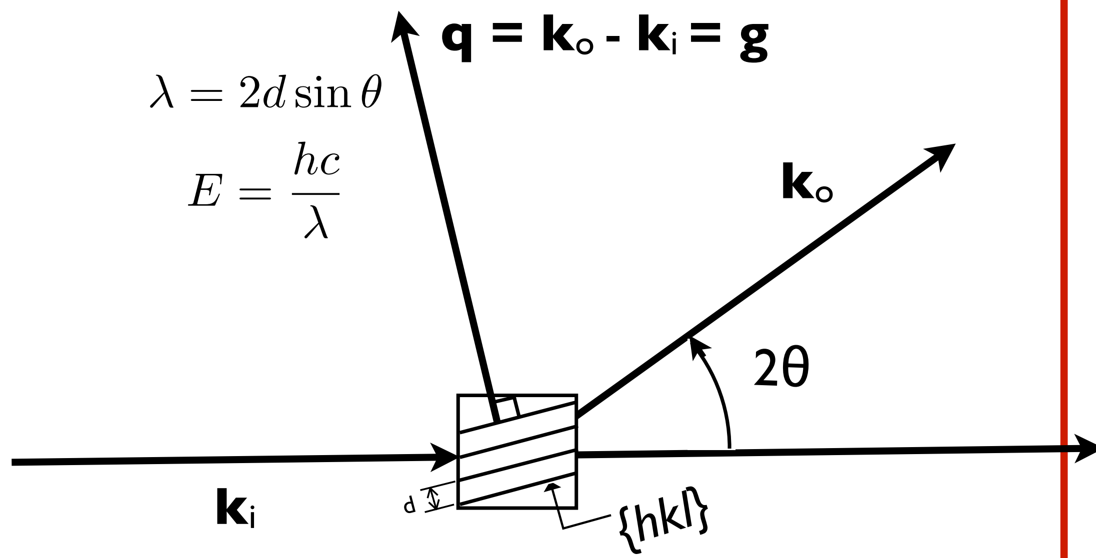

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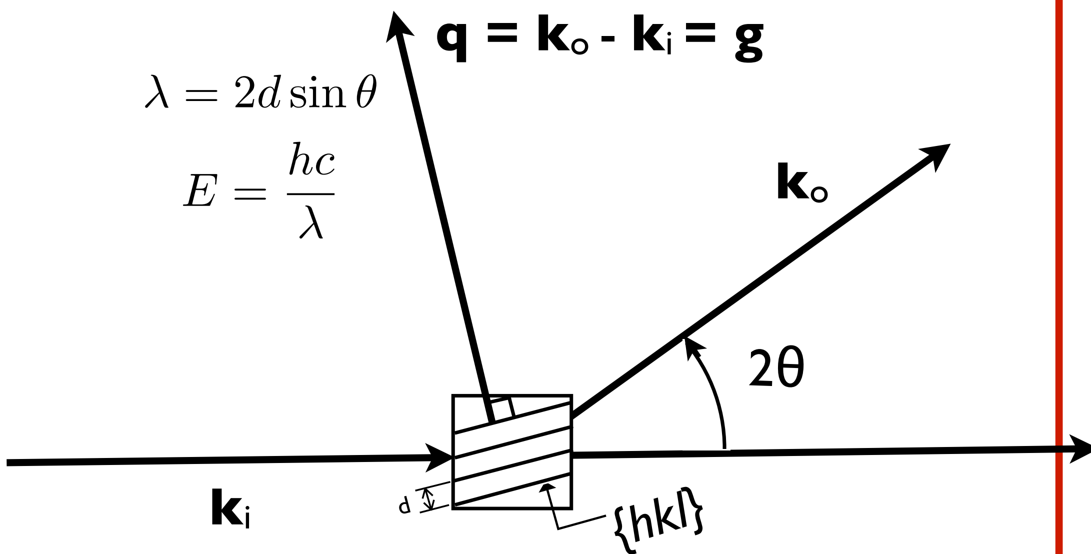

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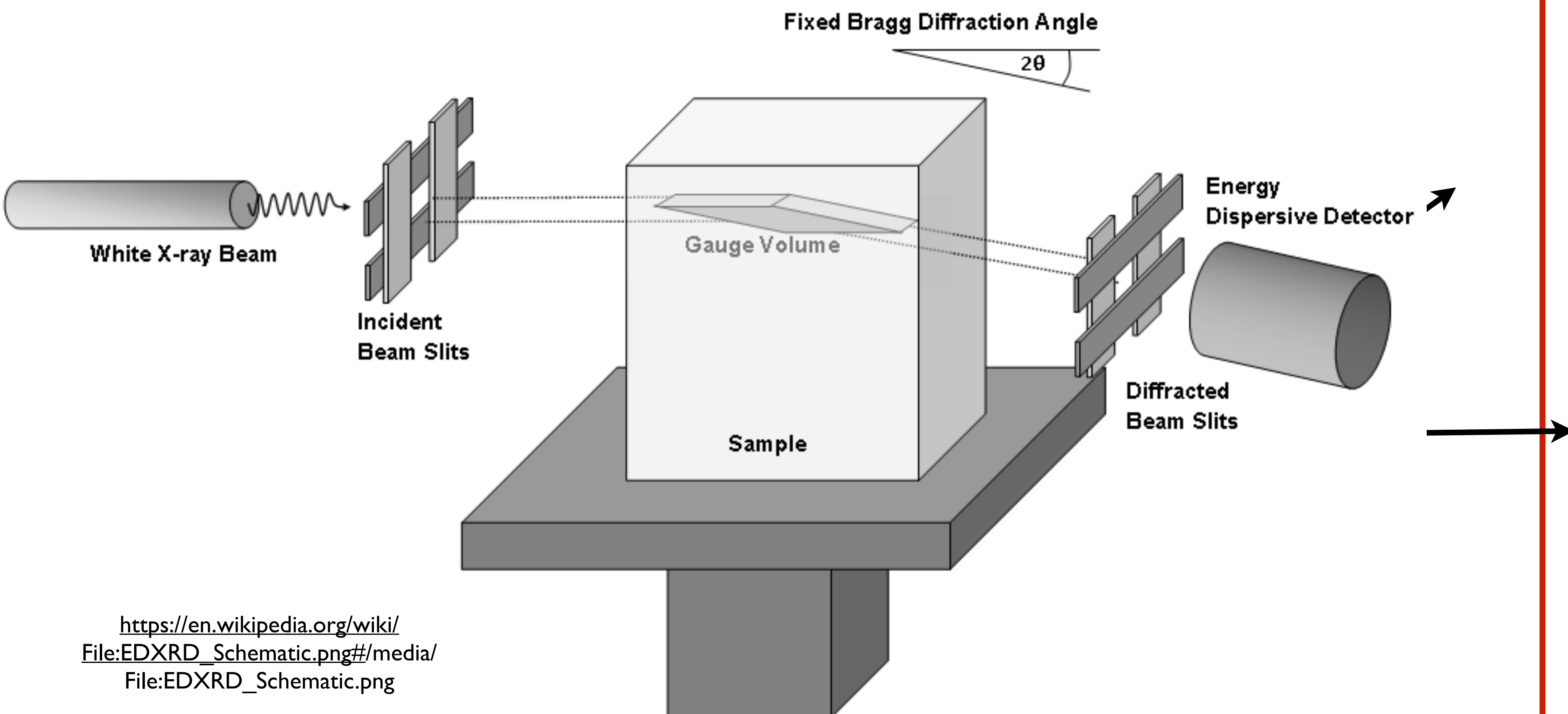

https://en.wikipedia.org/wiki/File:EDXRD_Schematic.png#/media/File:EDXRD_Schematic.png

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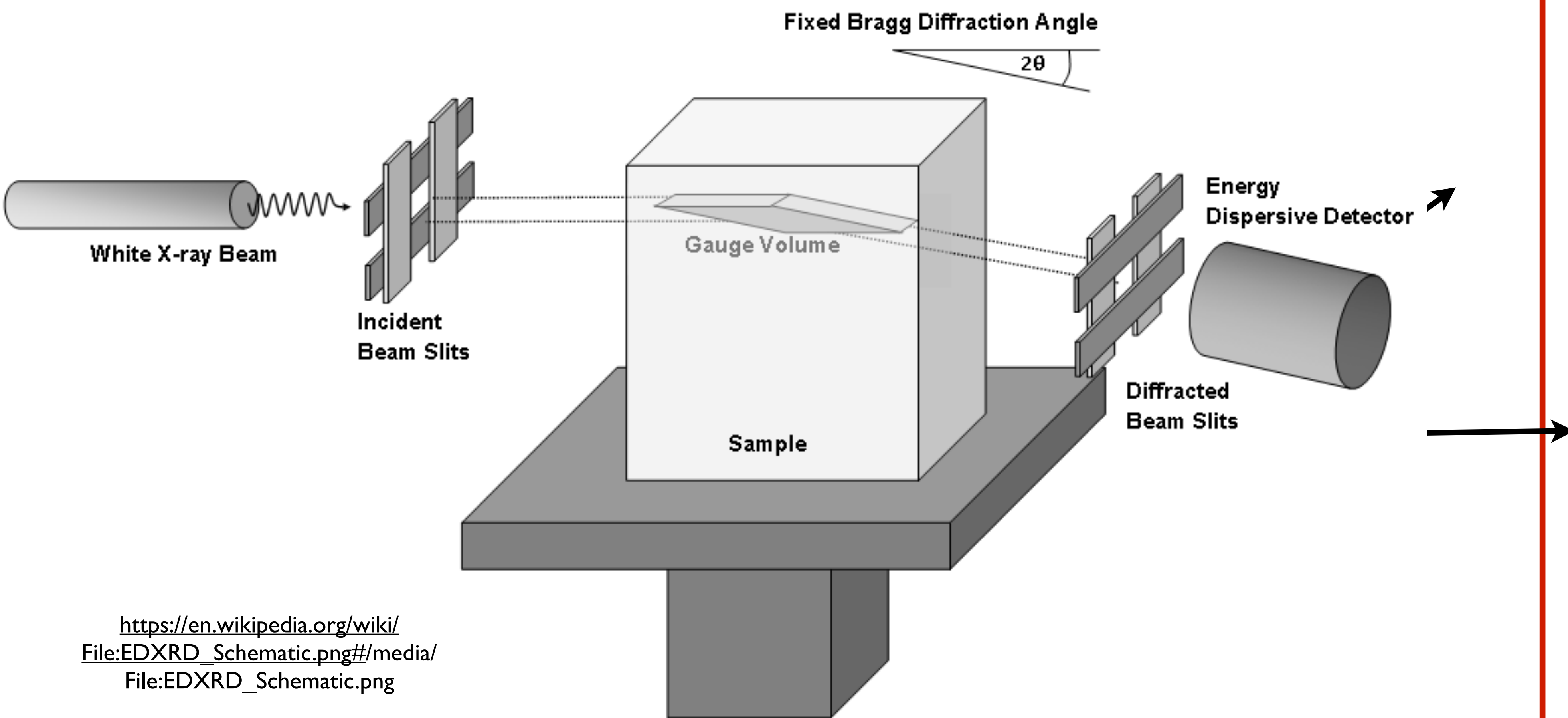

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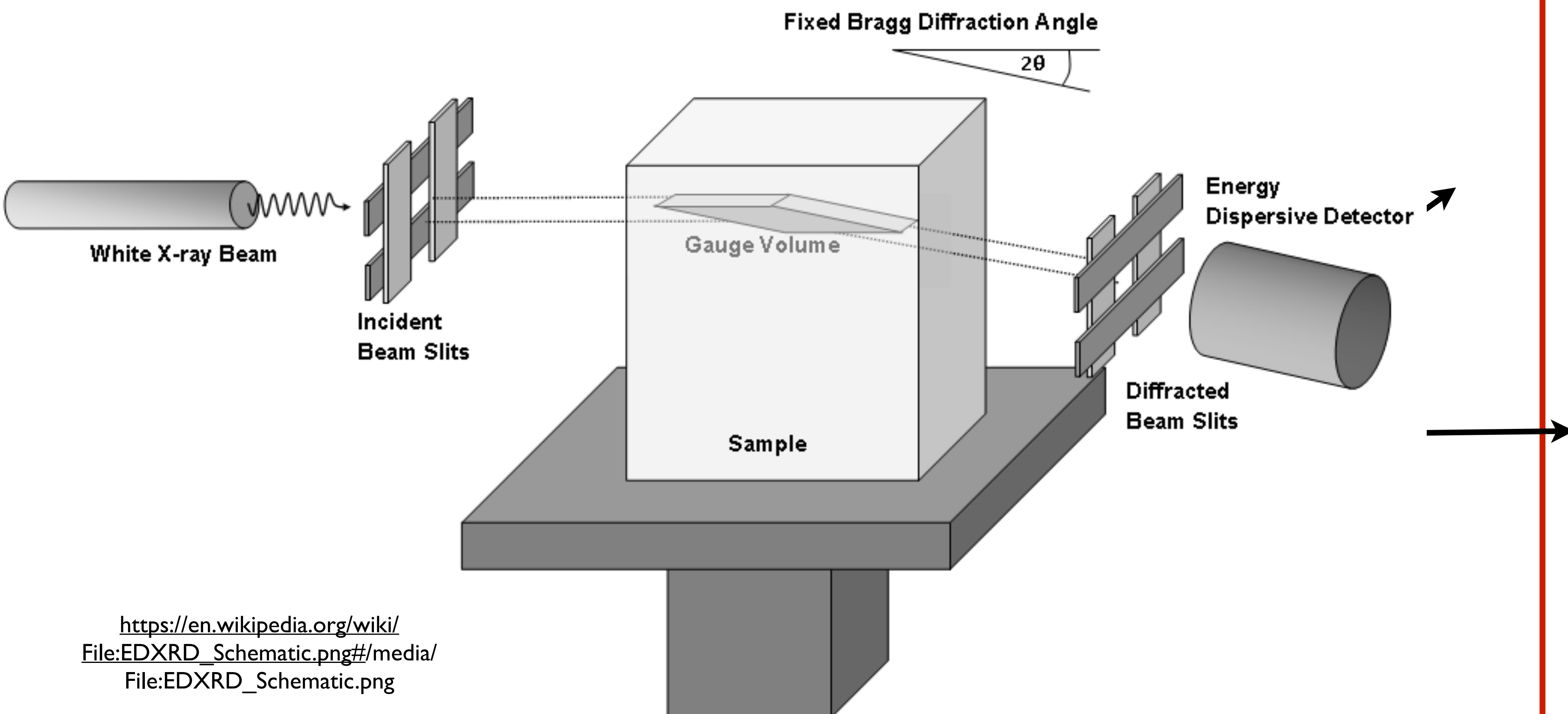
– Translate sample and/or detector
  - Collect a field of data
  - Slits enable stepping through T

High Energy Diffraction Basics

- X
- S
- M



- F

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  - Translate sample and/or detector
    - Collect a field of data
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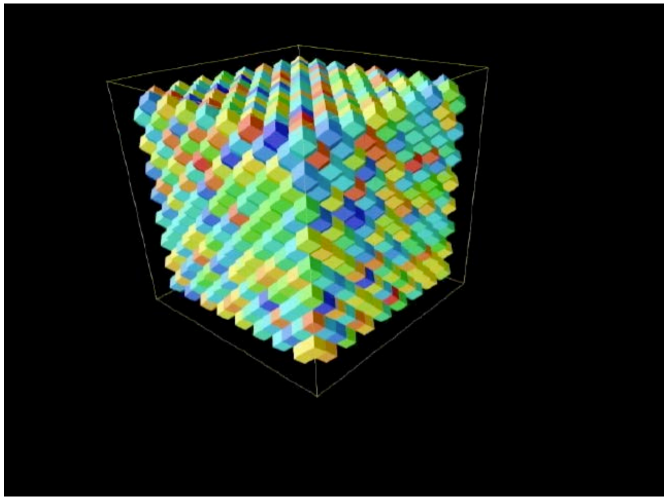
  - **SERIOUS fast alternative to neutron diffraction**

Beam size / grain size: Powder or Single Crystal



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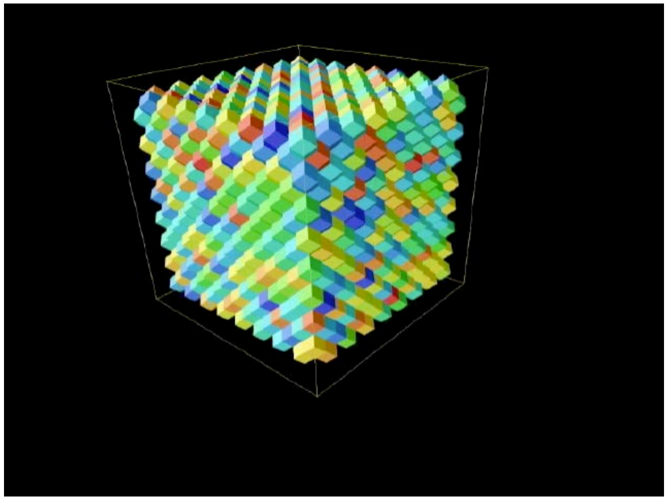

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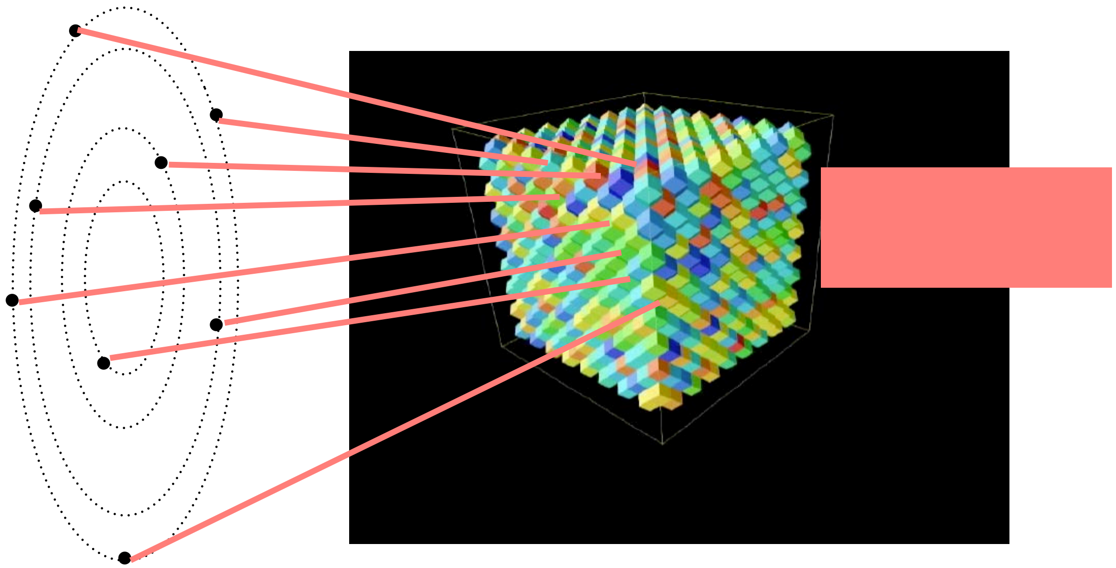

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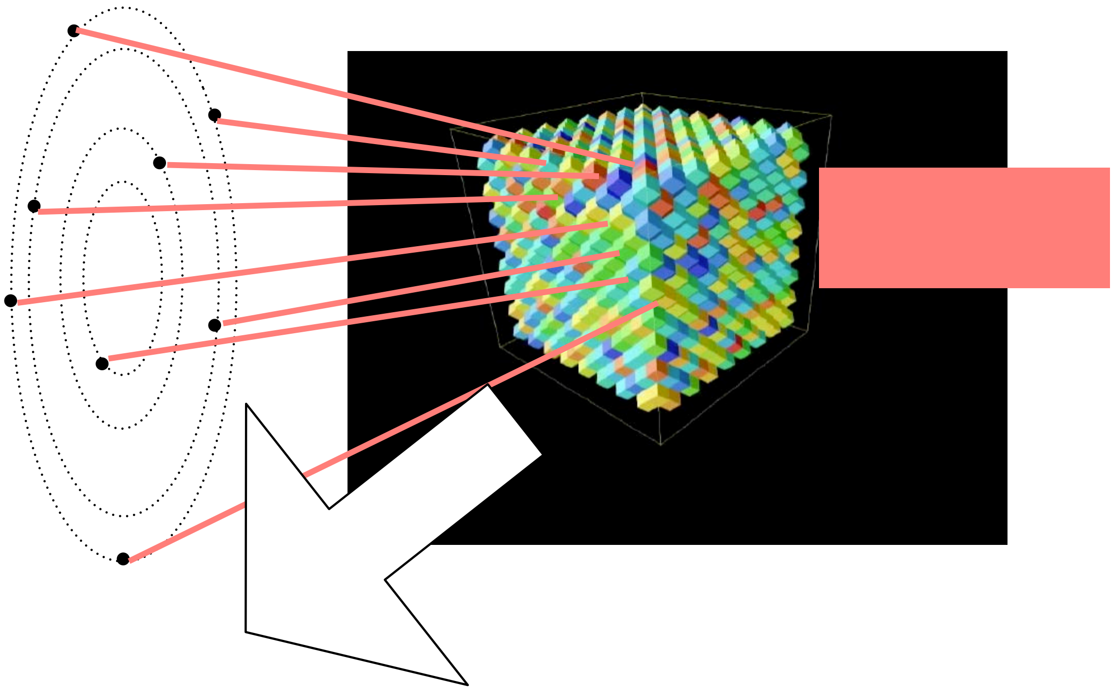

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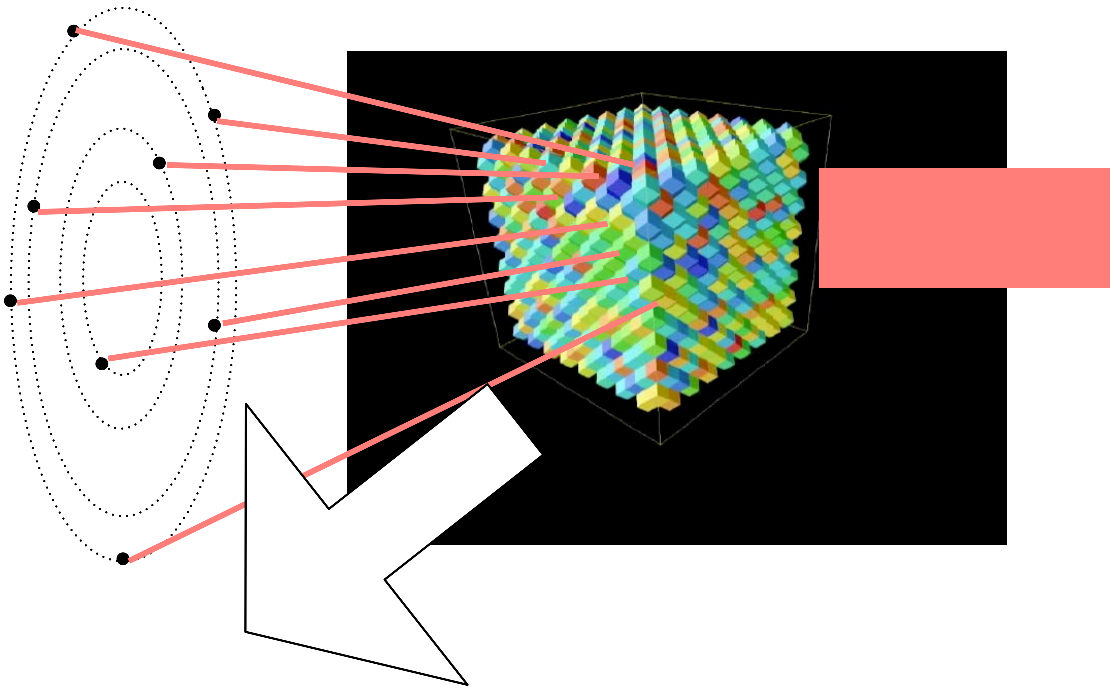

- **MultiGrain Experiments (Spots)**
  - **Collect diffracted intensity in each grain**
  - **100s to 2000 grains**
  - **Detector distance**
    - Near field - orientation map of polycrystal
    - Fare field - strains and evolution with in-situ loading

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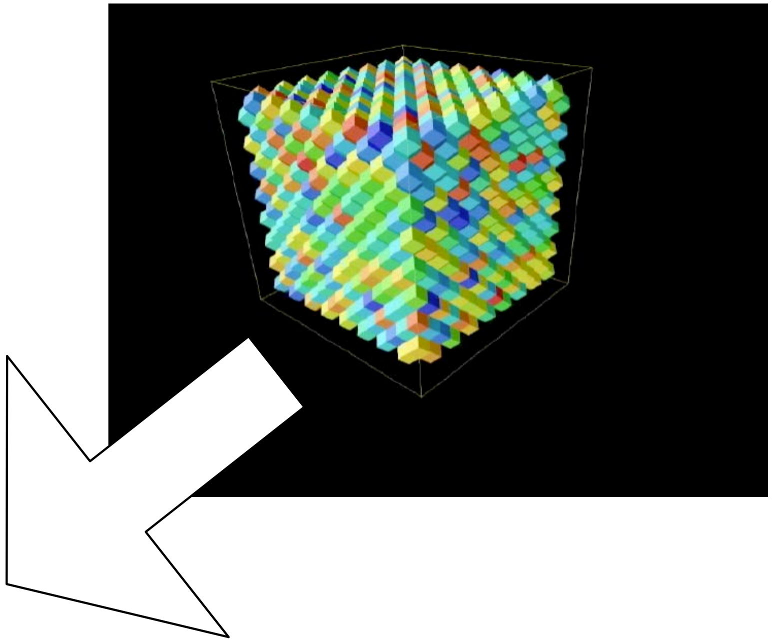

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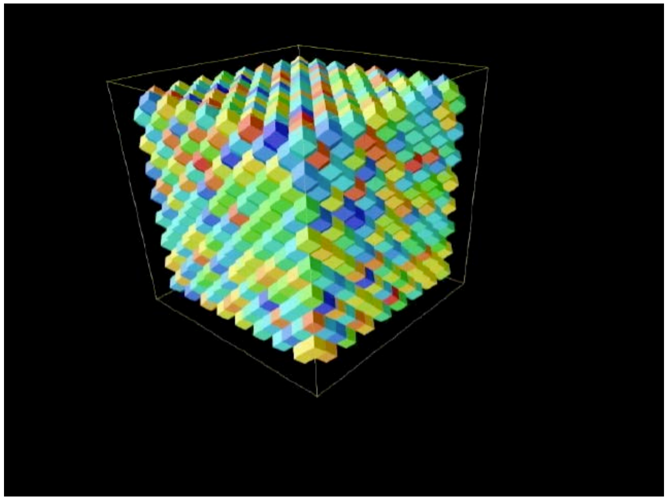

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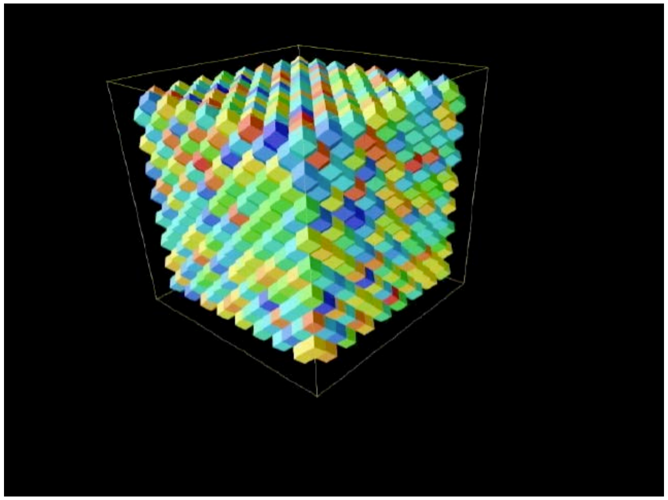

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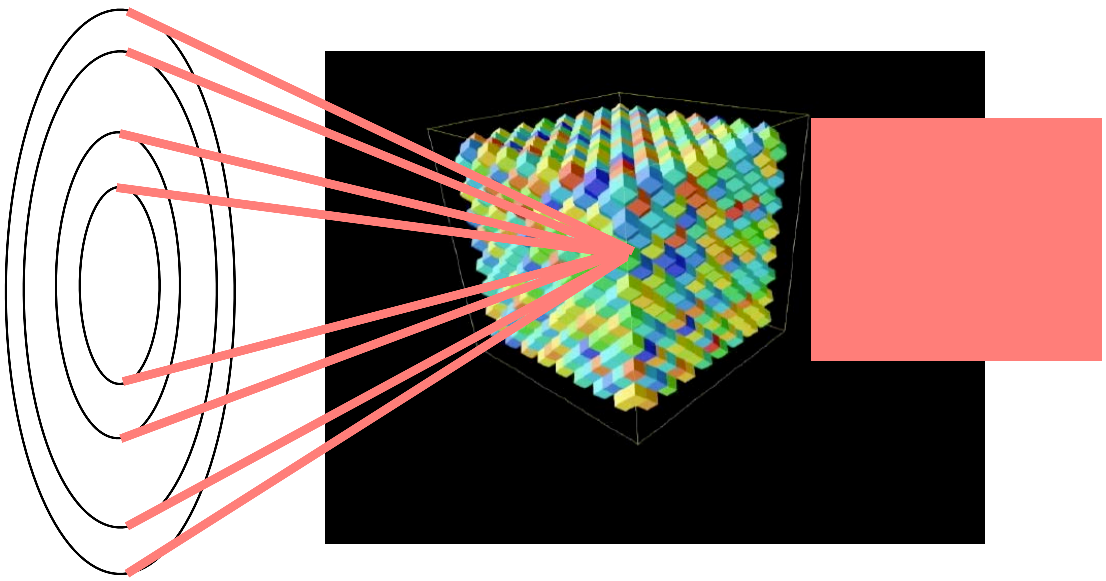

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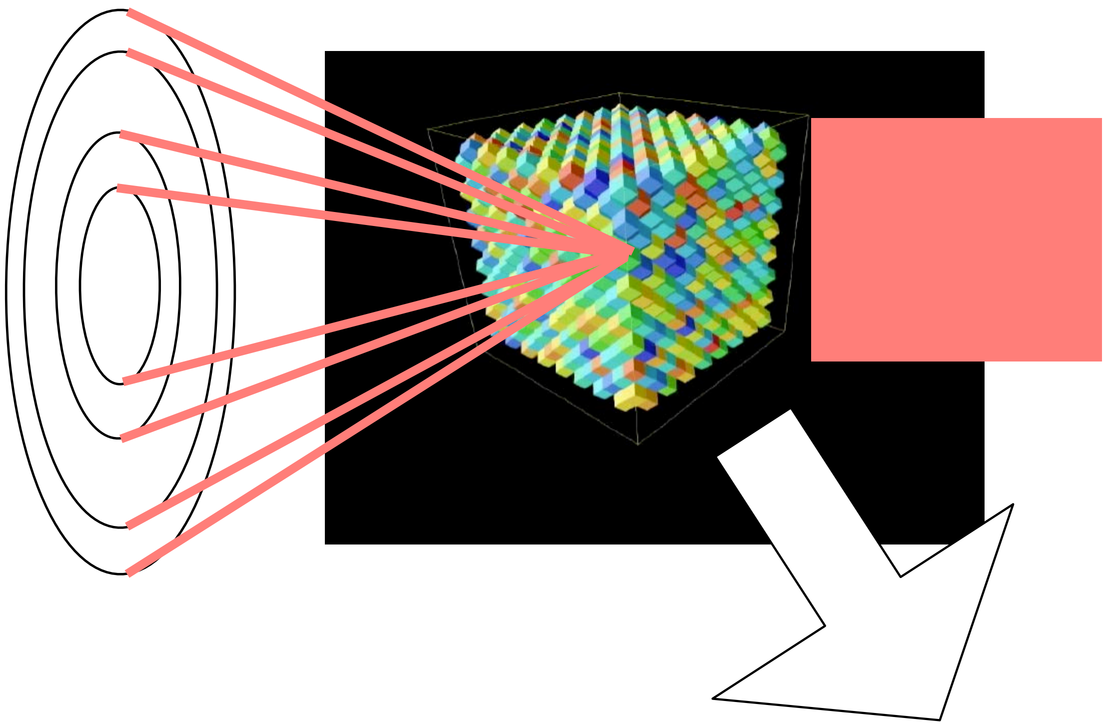

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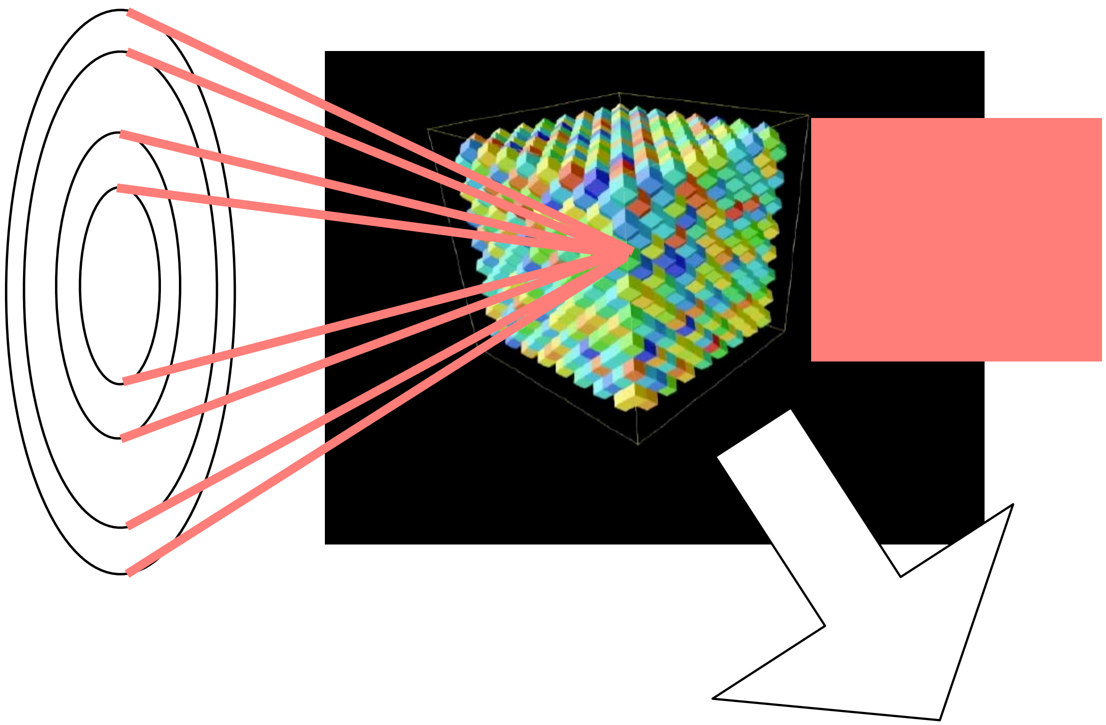


- **“Powder” Experiments**
  - **5,000-10,000 grains**
  - **Distribution information**
  - **In situ loading or residual stress**

High Energy X-ray diffraction (HEXD) Detector Distances

Near Field
Grain Maps

Far Field
Lattice Strain (Tensor)
and Orientations
of each crystal

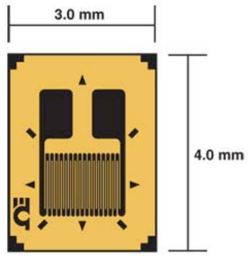
Very Far Field
High Resolution
Strain and
Orientation

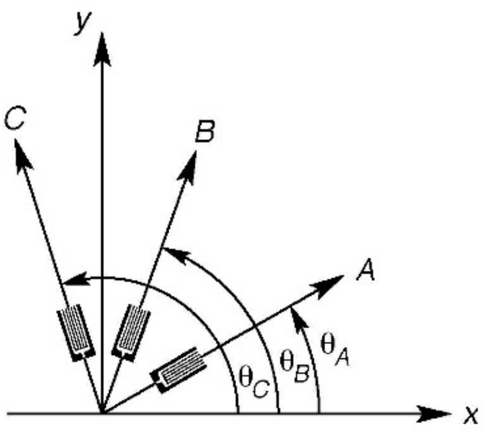
Real Space Resolution

Reciprocal Space Resolution

Better Reciprocal Space Resolution

Lattice Strains - Link to Stress





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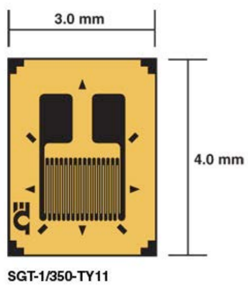
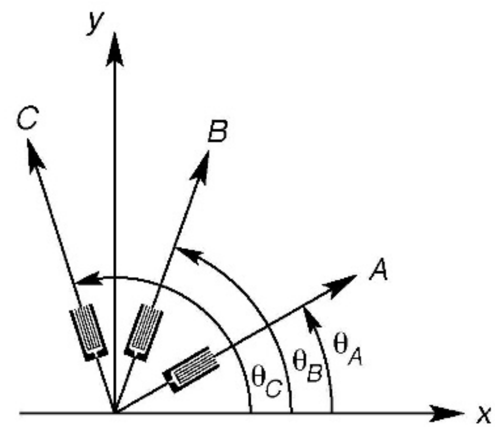
- **Start with the idea of stress analysis using resistance strain gages**





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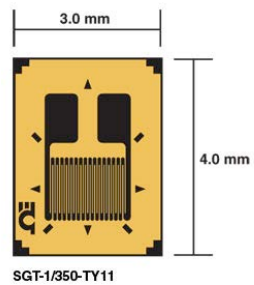
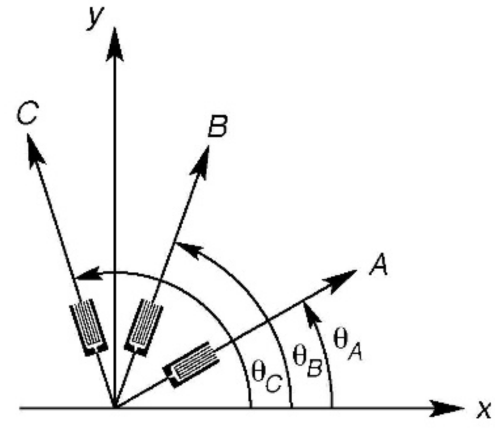
  - **Plane Stress - 3 Strains**





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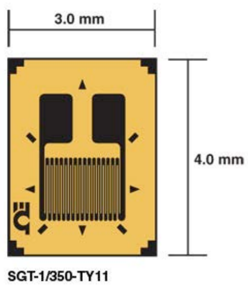

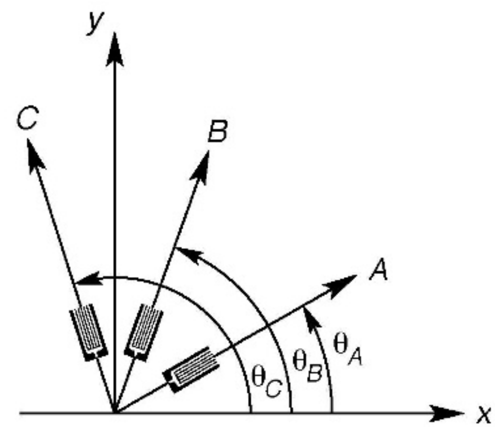

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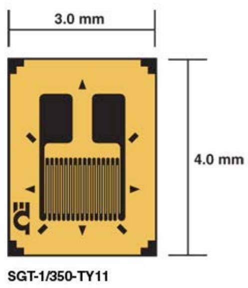

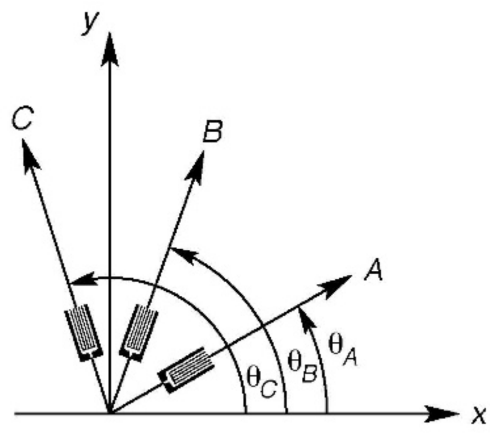

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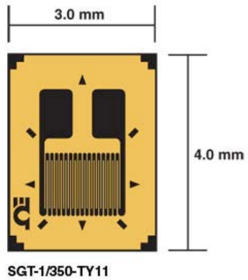
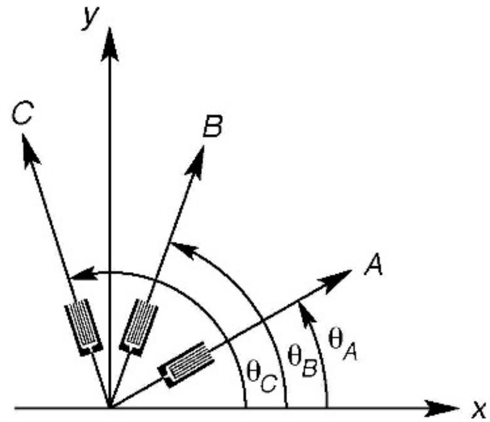
  - Change in 2θ produces a peak shift = normal strain





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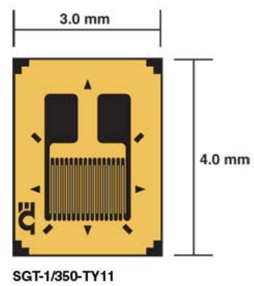
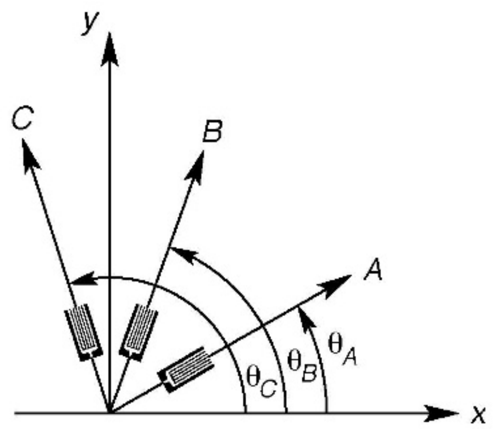
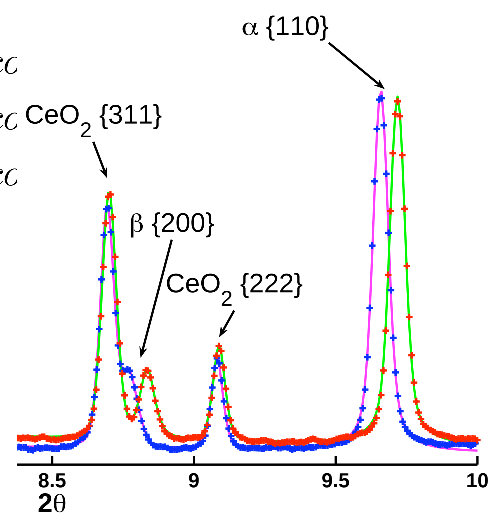




$\epsilon_A(\theta_A) = cc$ … $\imath(\theta_A)cos(\theta_A)\epsilon_{12}$

$\epsilon_B(\theta_B) = cc$ … $\imath(\theta_B)cos(\theta_B)\epsilon_{12}$

$\epsilon_C(\theta_C) = cc$ … $\imath(\theta_C)cos(\theta_C)\epsilon_{12}$



Lattice Strains - Link to Stress

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  - **Plane Stress - 3 Strains**
  - **Rosette Equations for the strain tensor**
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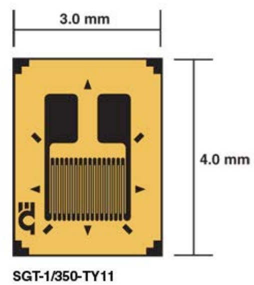
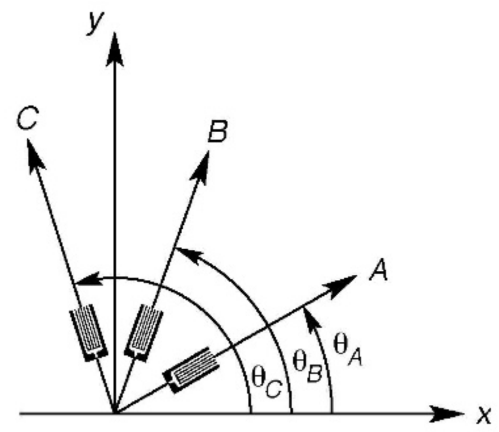
  - **Scattering vector is strain "direction"**

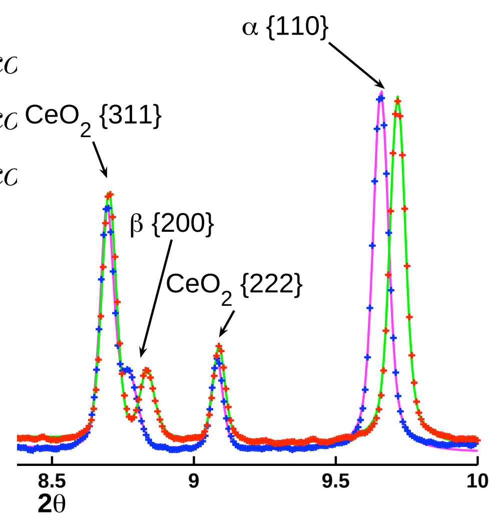




$\epsilon_A(\theta_A) = co$ … $n(\theta_A)cos(\theta_A)\epsilon_{12}$

$\epsilon_B(\theta_B) = co$ … $n(\theta_B)cos(\theta_B)\epsilon_{12}$

$\epsilon_C(\theta_C) = co$ … $n(\theta_C)cos(\theta_C)\epsilon_{12}$



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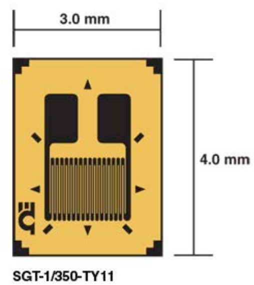

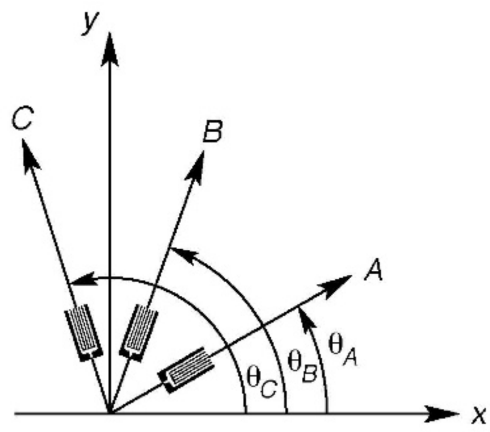

$$\epsilon_A(\theta_A) = cc \quad \imath(\theta_A)cos(\theta_A)\epsilon_{12}$$
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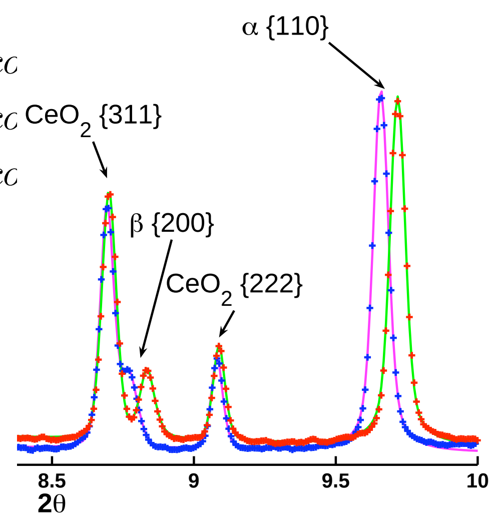

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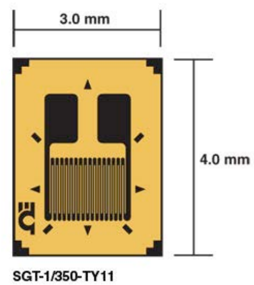
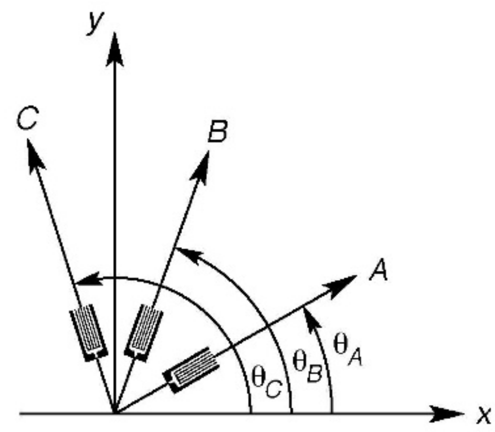
- **Collect enough strains to build lattice (elastic) strain tensor using rosette equations**
  - **Mechanics: equilibrium and 6 strains**





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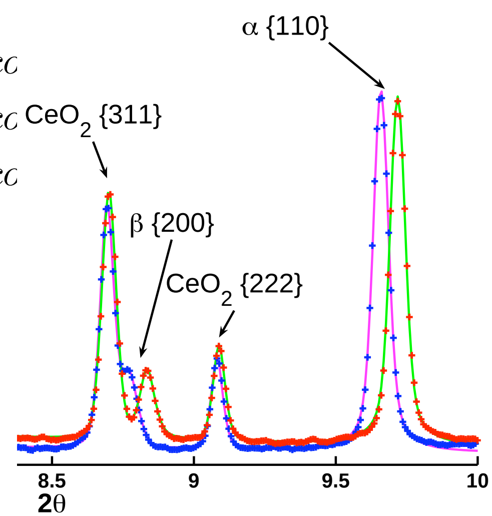
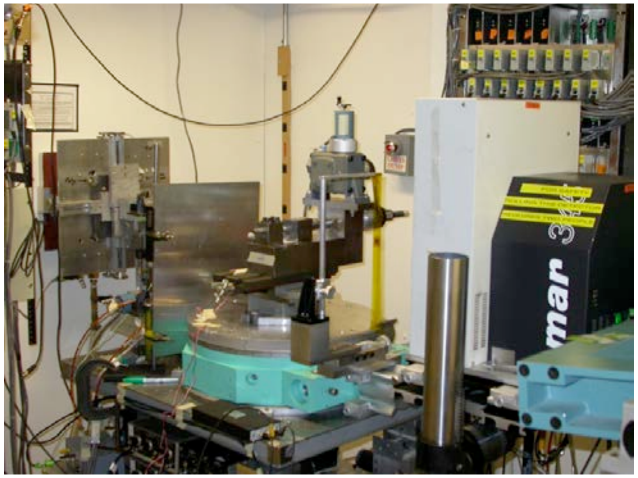


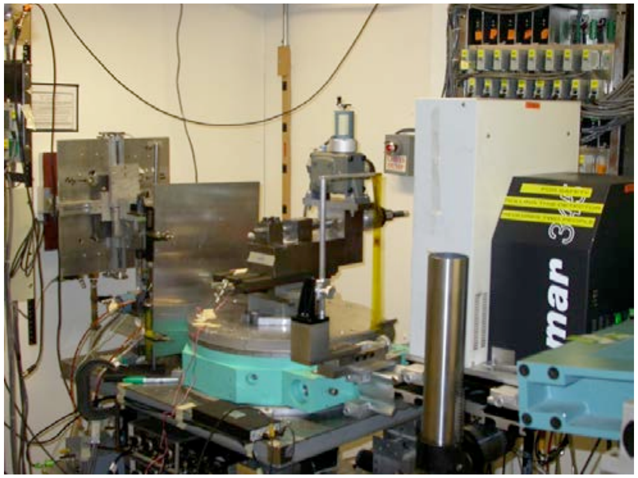
High Energy X-ray Diffraction at CHESS & APS sector 1

High Energy X-ray Diffraction at CHESS & APS sector 1



CHESS A2 - 2003

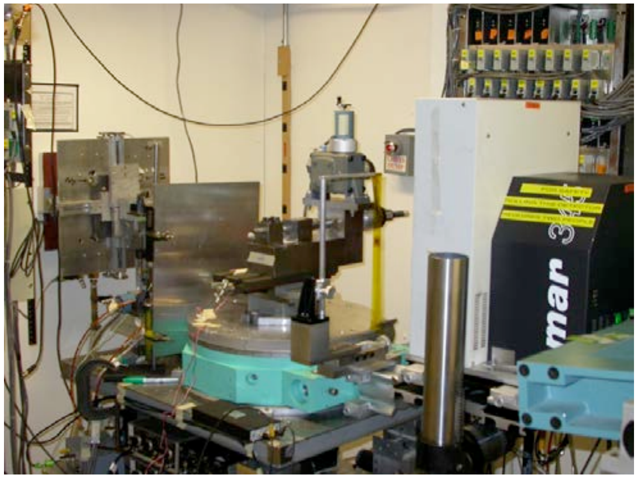
High Energy X-ray Diffraction at CHESS & APS sector 1



CHESS A2 - 2003

CHESS A2 - 2005

High Energy X-ray Diffraction at CHESS & APS sector 1

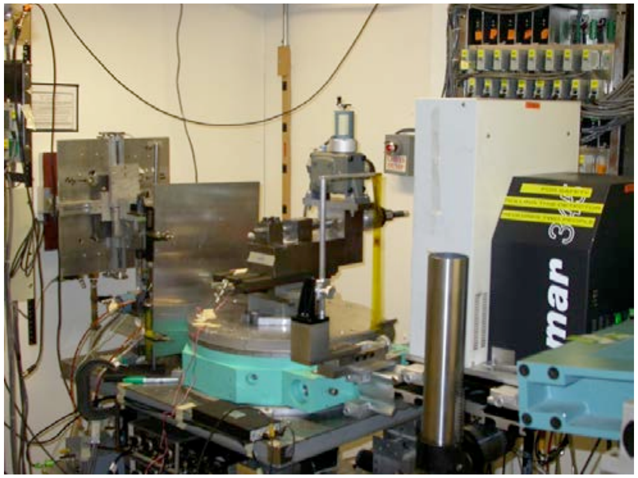


CHESS A2 - 2003

CHESS A2 - 2005

A2 - 2010

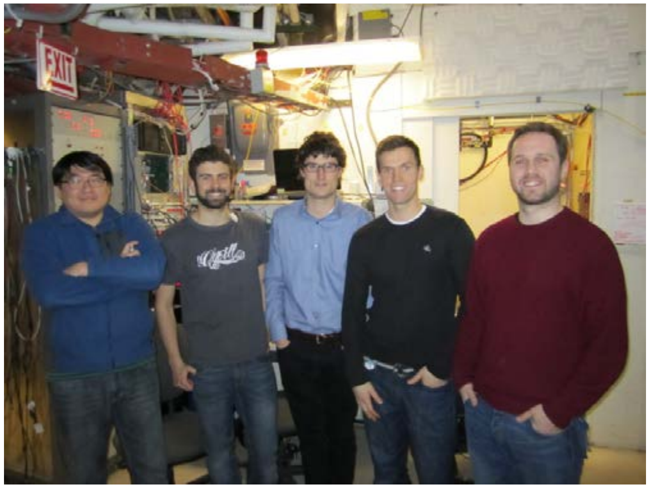
High Energy X-ray Diffraction at CHESS & APS sector 1



CHESS A2 - 2003

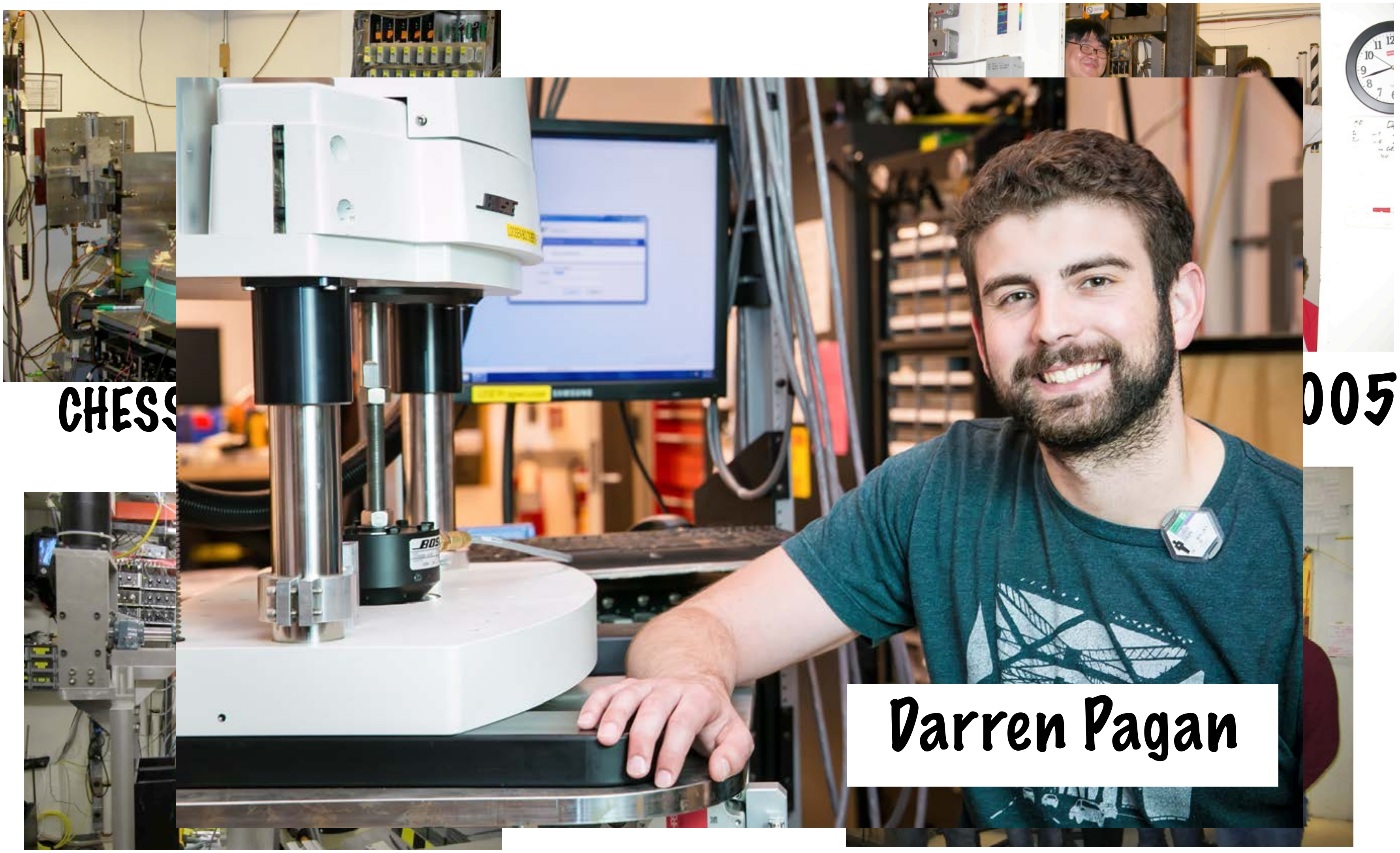
CHESS A2 - 2005

A2 - 2010



A2 - 2013

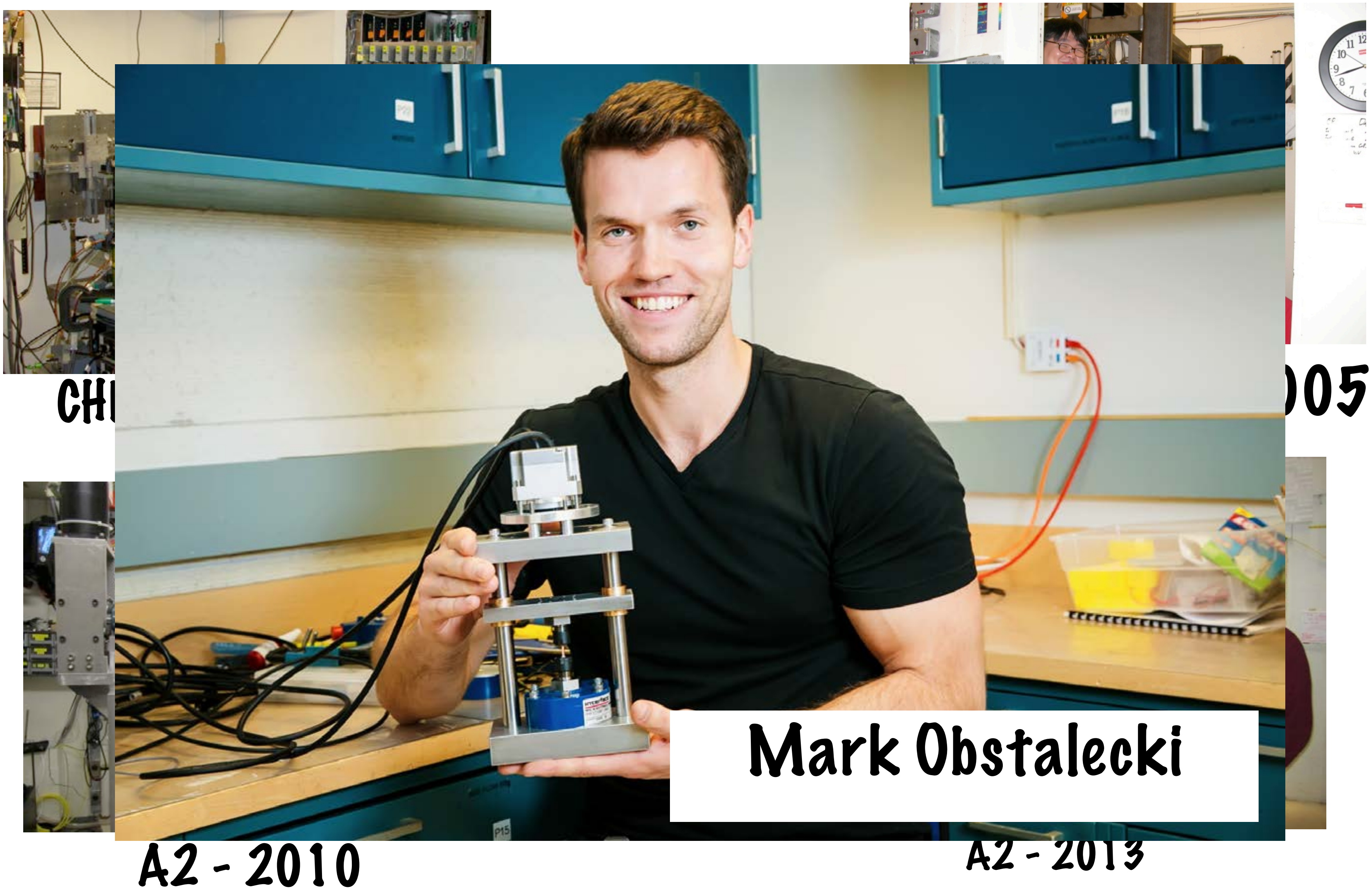
High Energy X-ray Diffraction at CHESS & APS sector 1



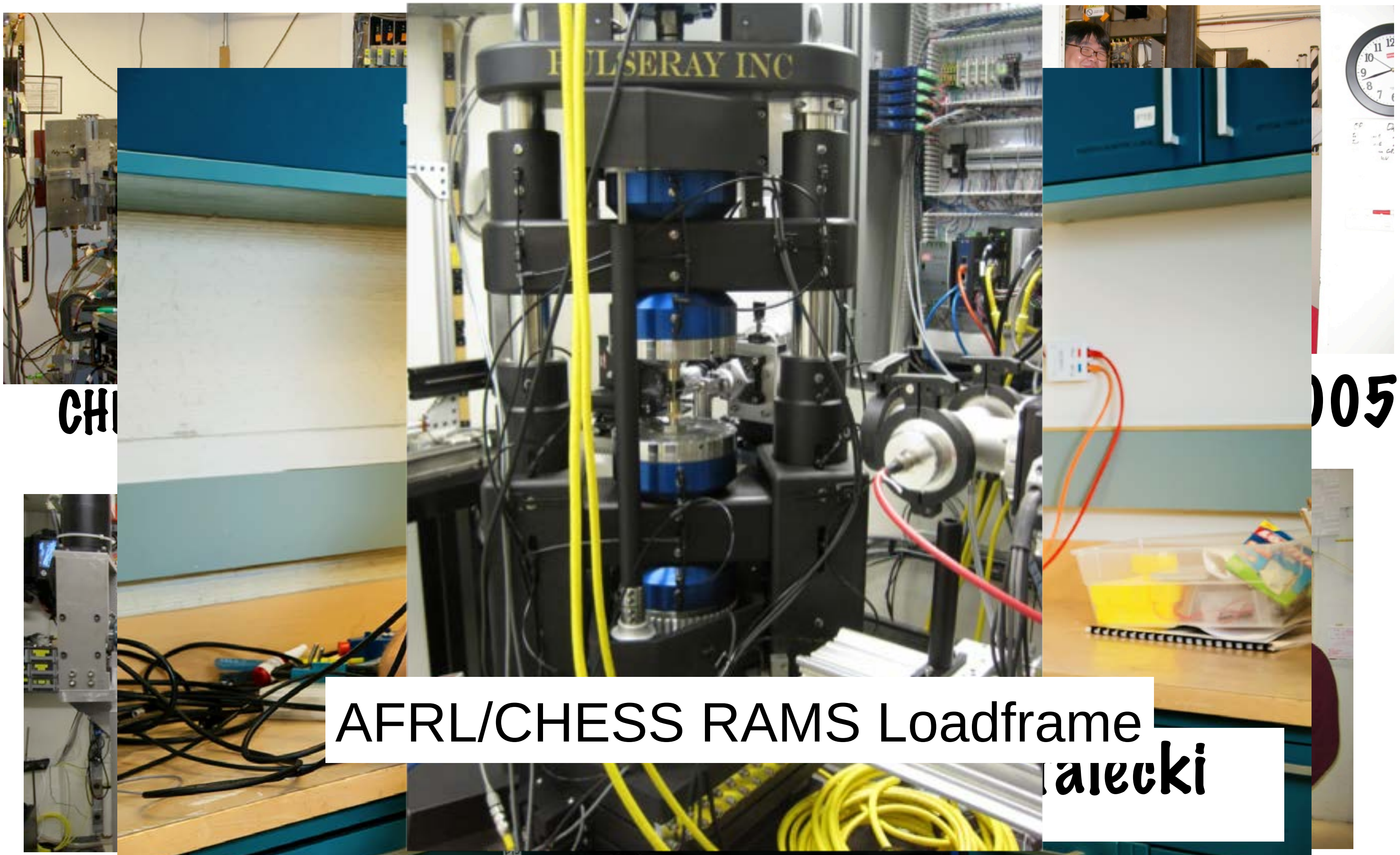
A2 - 2010

A2 - 2013

High Energy X-ray Diffraction at CHESS & APS sector 1



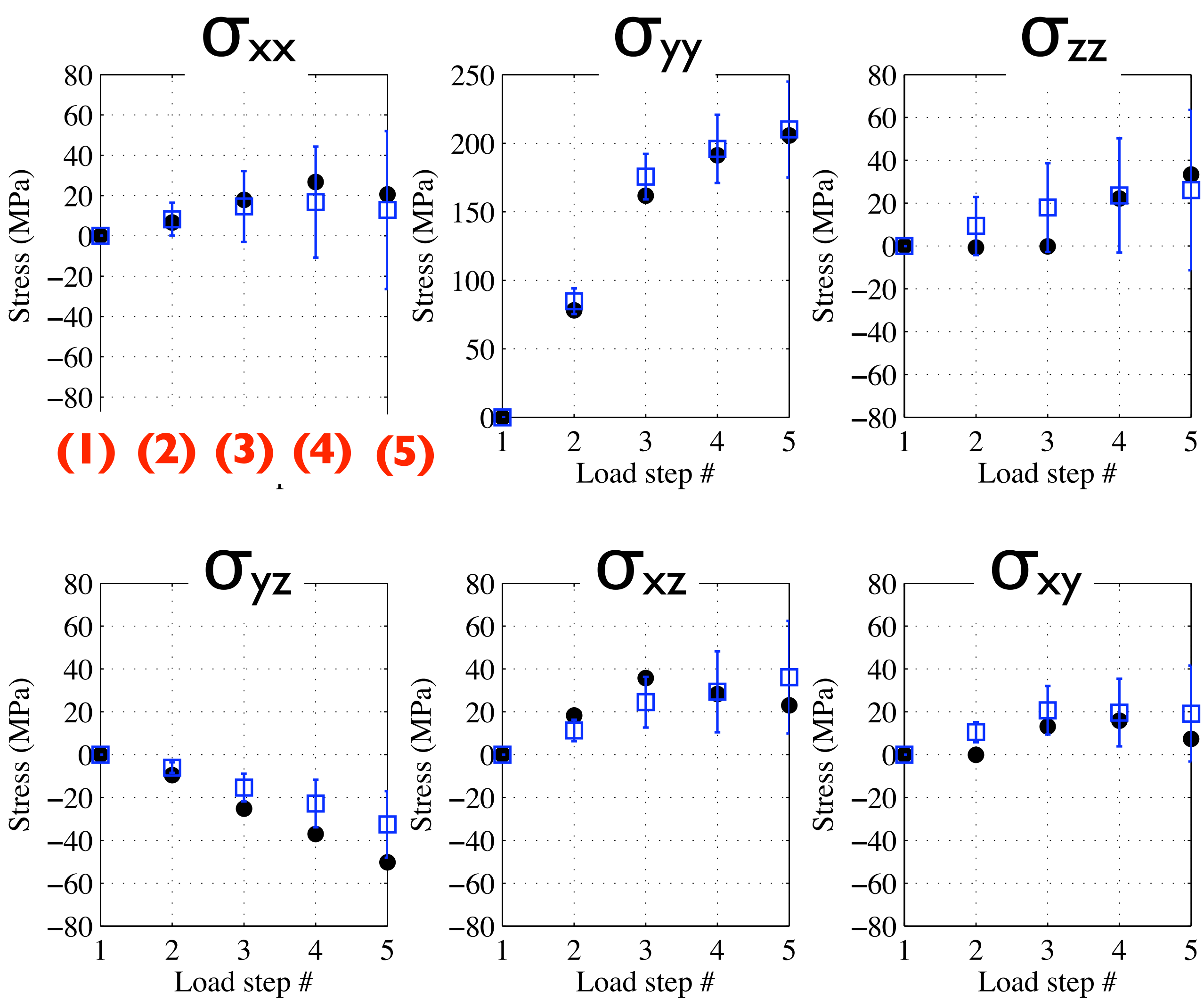
High Energy X-ray Diffraction at CHESS & APS sector 1

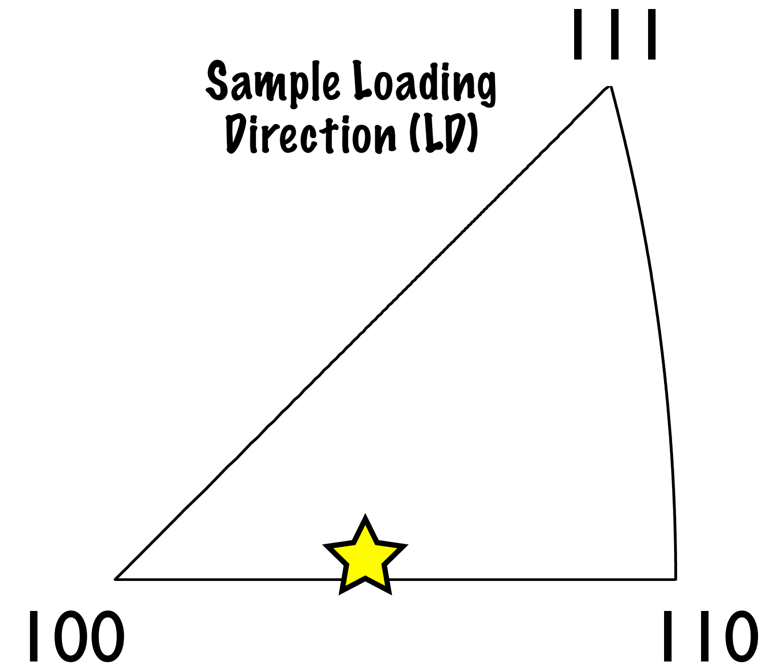


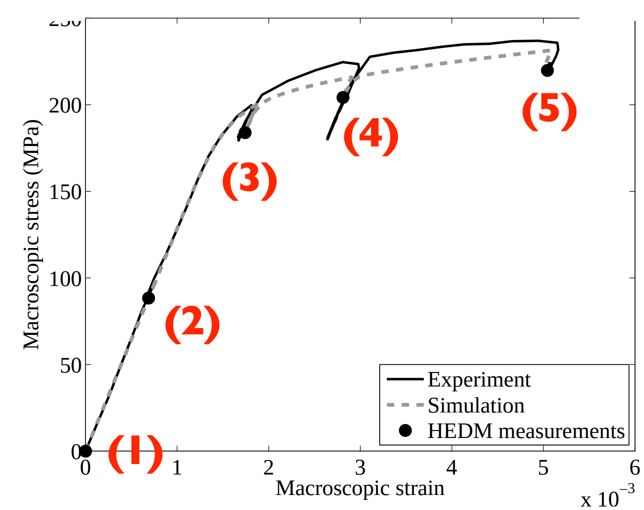
A2 - 2010

A2 - 2013

Tensile Response of One Crystal - Far Field







Wong et. al, 2013, Comp. Mat. Sci., 77, 456-466.

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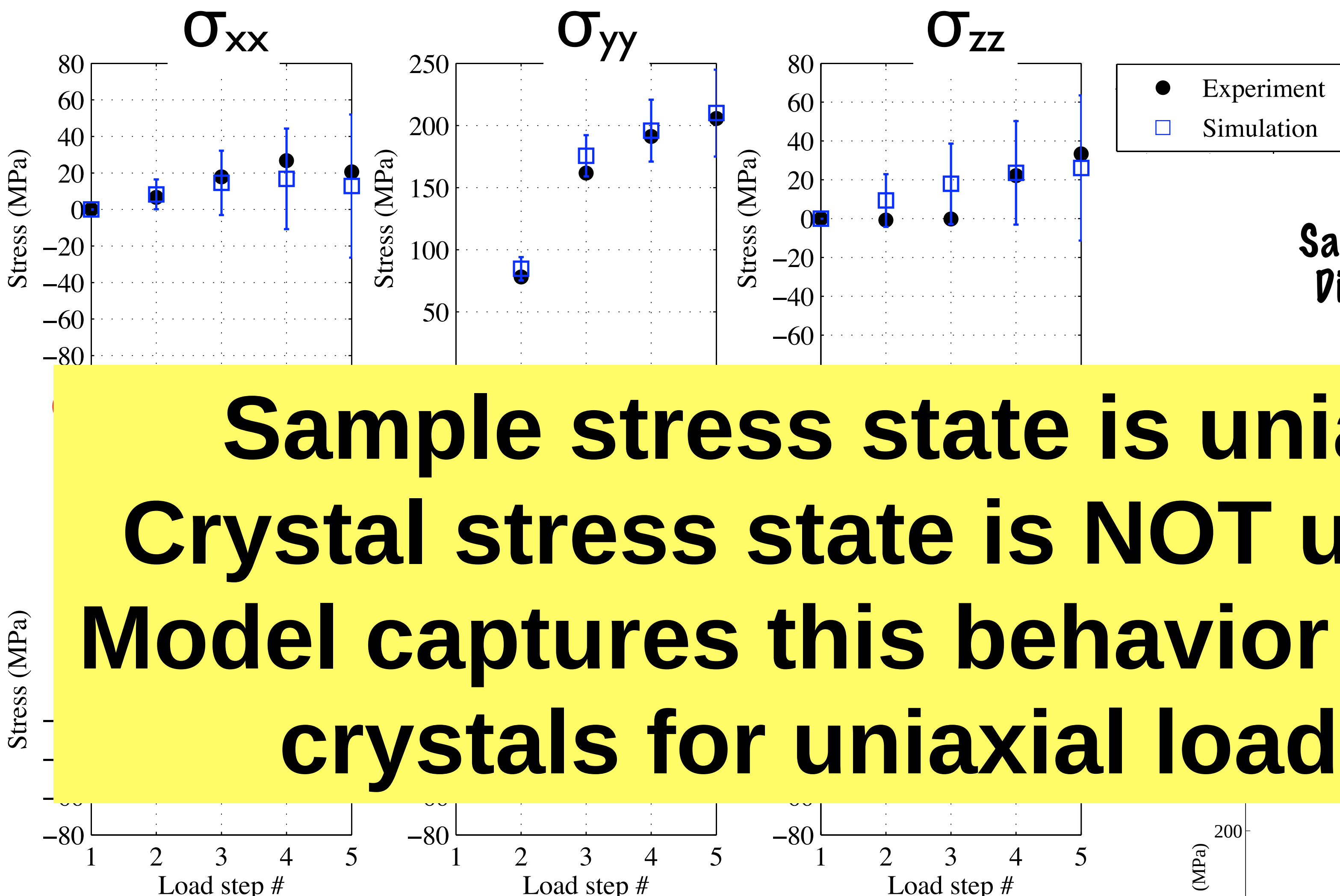
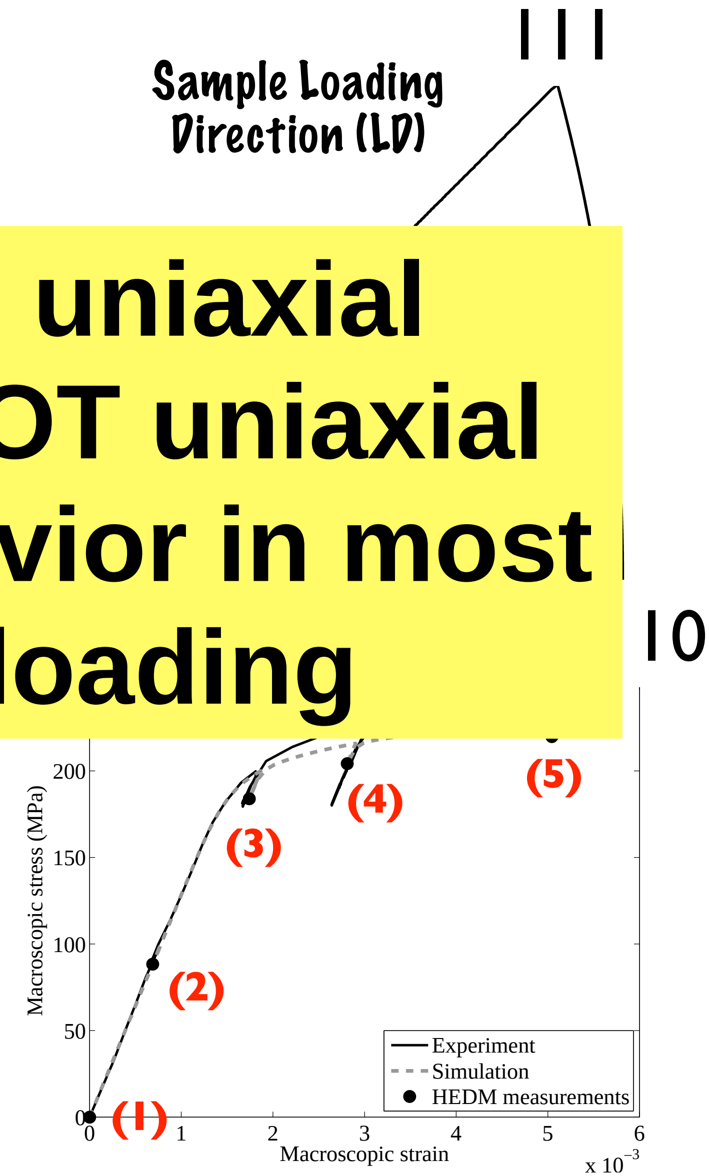
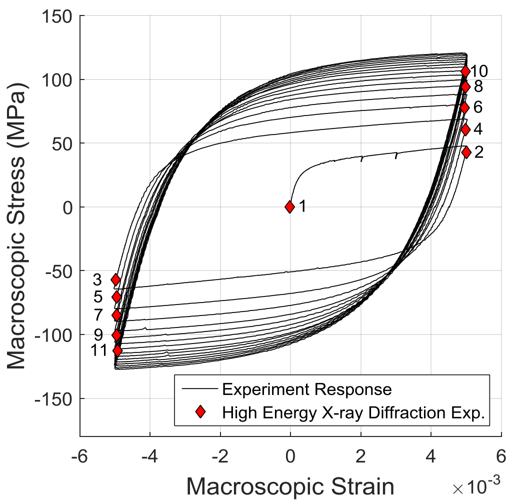




Sample stress state is uniaxial
Crystal stress state is NOT uniaxial
Model captures this behavior in most crystals for uniaxial loading

Wong et. al, 2013, Comp. Mat. Sci., 77, 456-466.

Cyclic Deformation of High Purity Copper

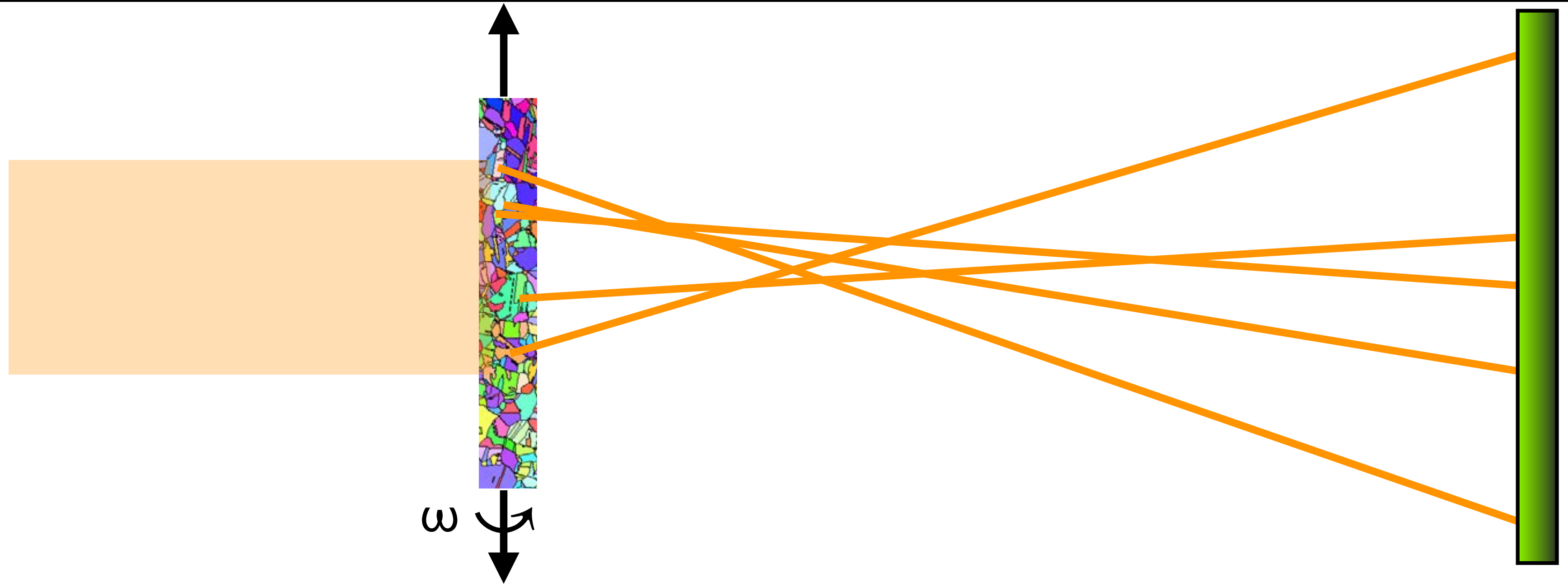


Obstalecki et al., Acta Mat., 75, 259-272, 2014

- **Fatigue Crack initiation in copper**
  - **Heterogeneous plastic slip**
- **Cyclic tests:**
  - **OFHC (99.9% pure copper)**
- **CHESS F2 & APS 1-ID**
- **Su Leen Wong & Robert Carson simulations (P. Dawson)**

Macroscale Stress-Strain

Forward projection: interface with the model



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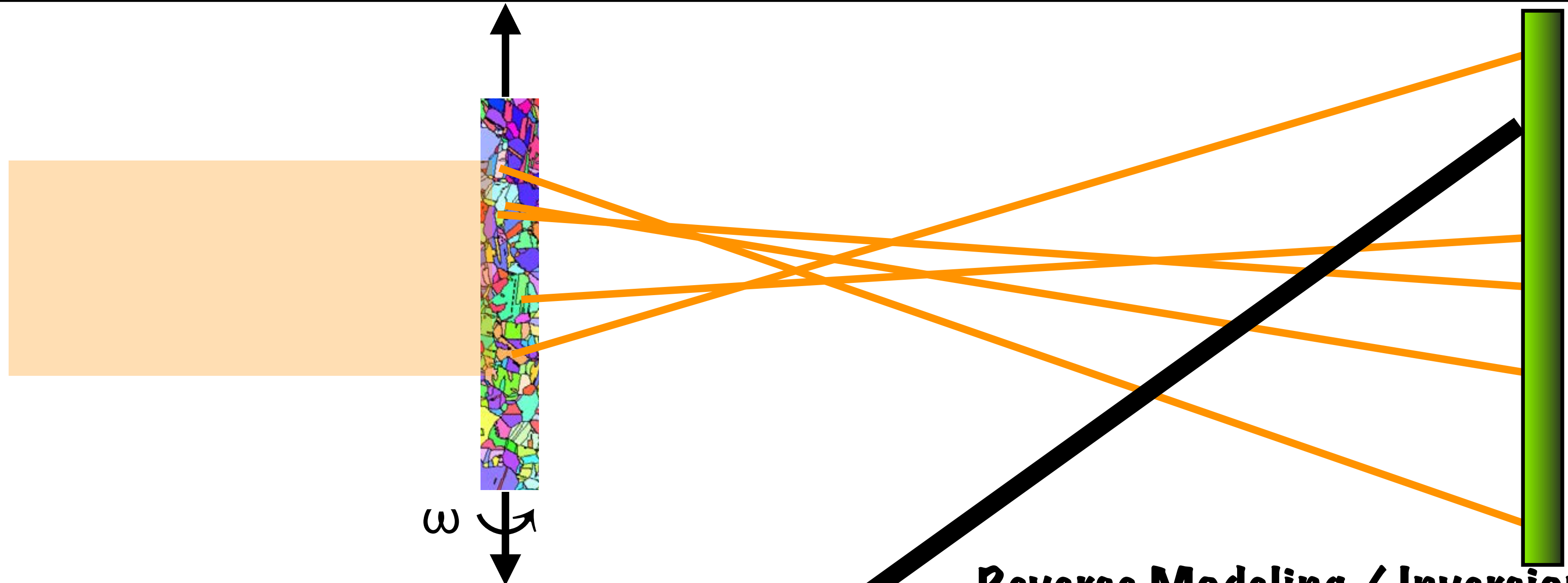
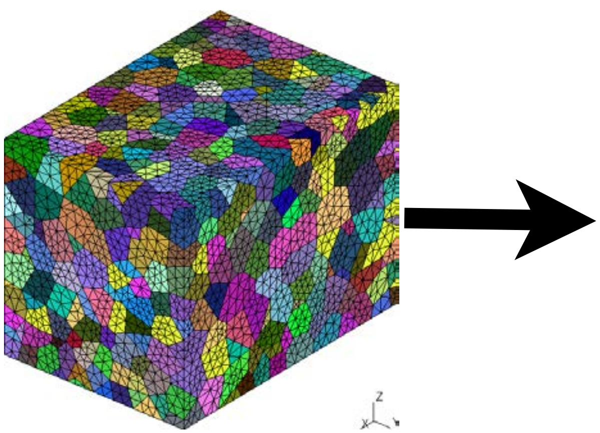
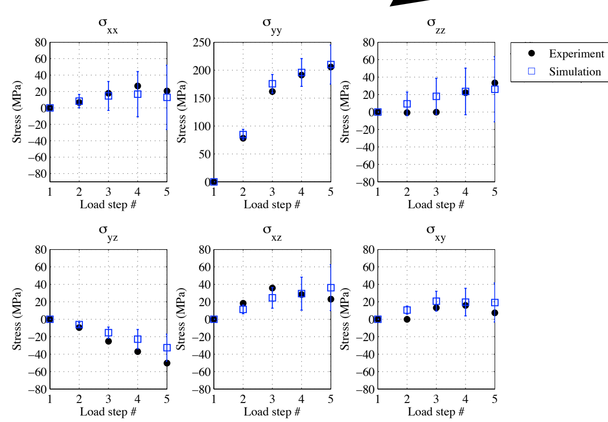




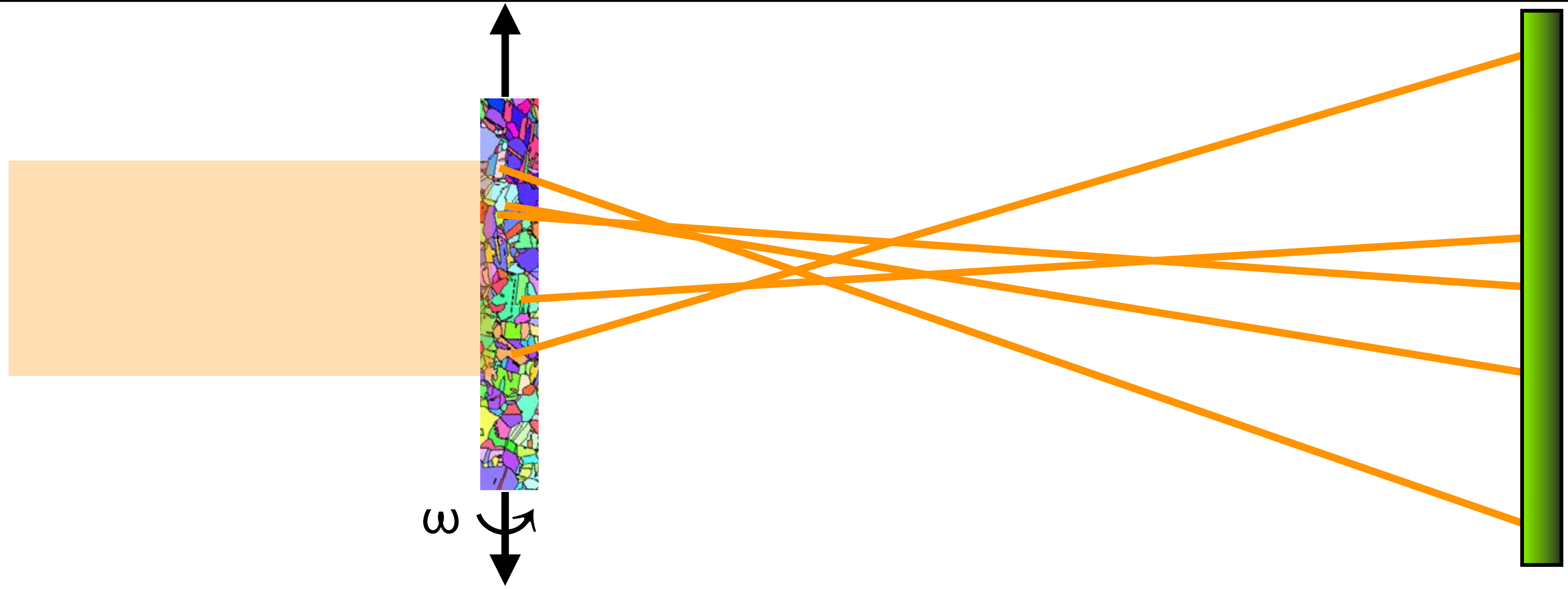


Crystal Stress Tensor

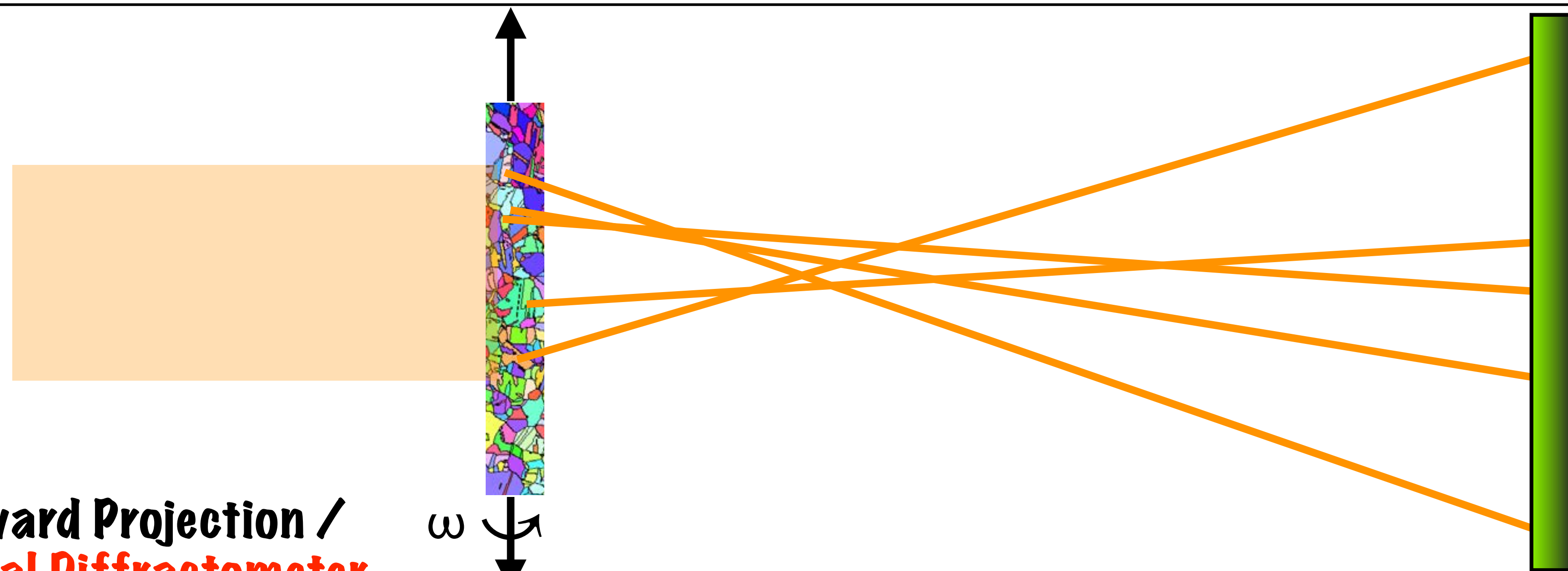
Reverse Modeling / Inversion

- Use data with the diffraction model to back-calculate:
  - lattice strains - stresses
  - orientation distributions
- Extract stresses and orientations from FEM model
  - Compare
- Iteration/hypothesis check?

Forward projection: interface with the model

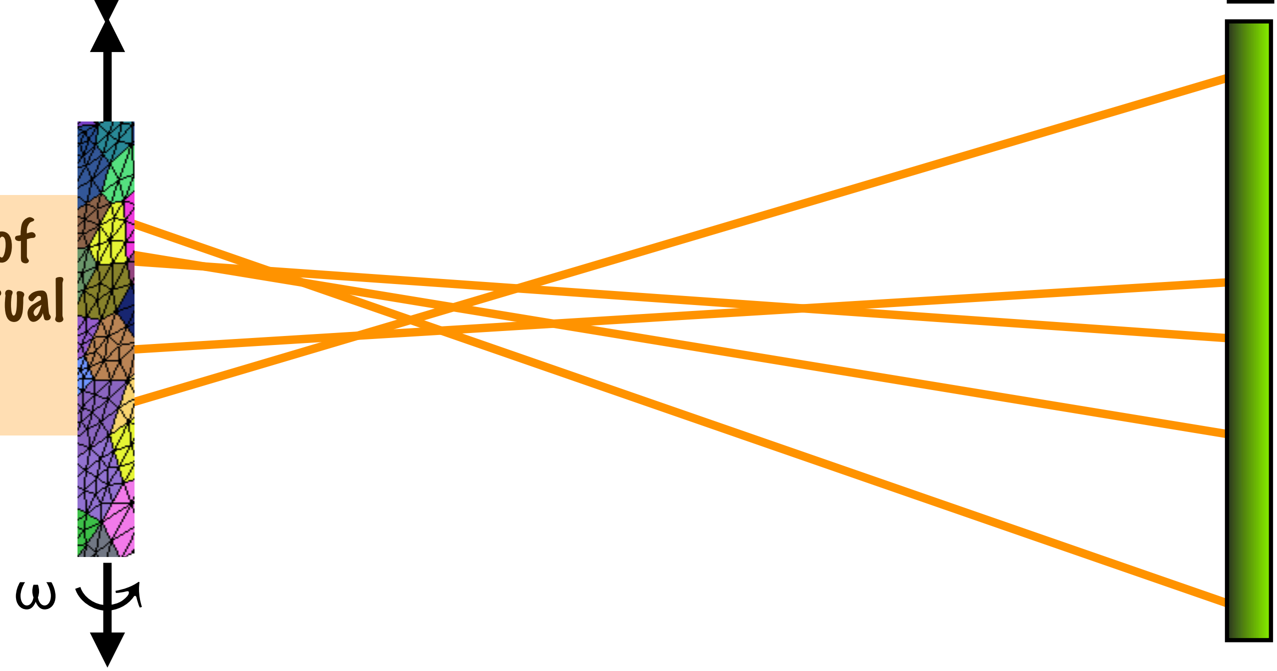


Forward projection: interface with the model



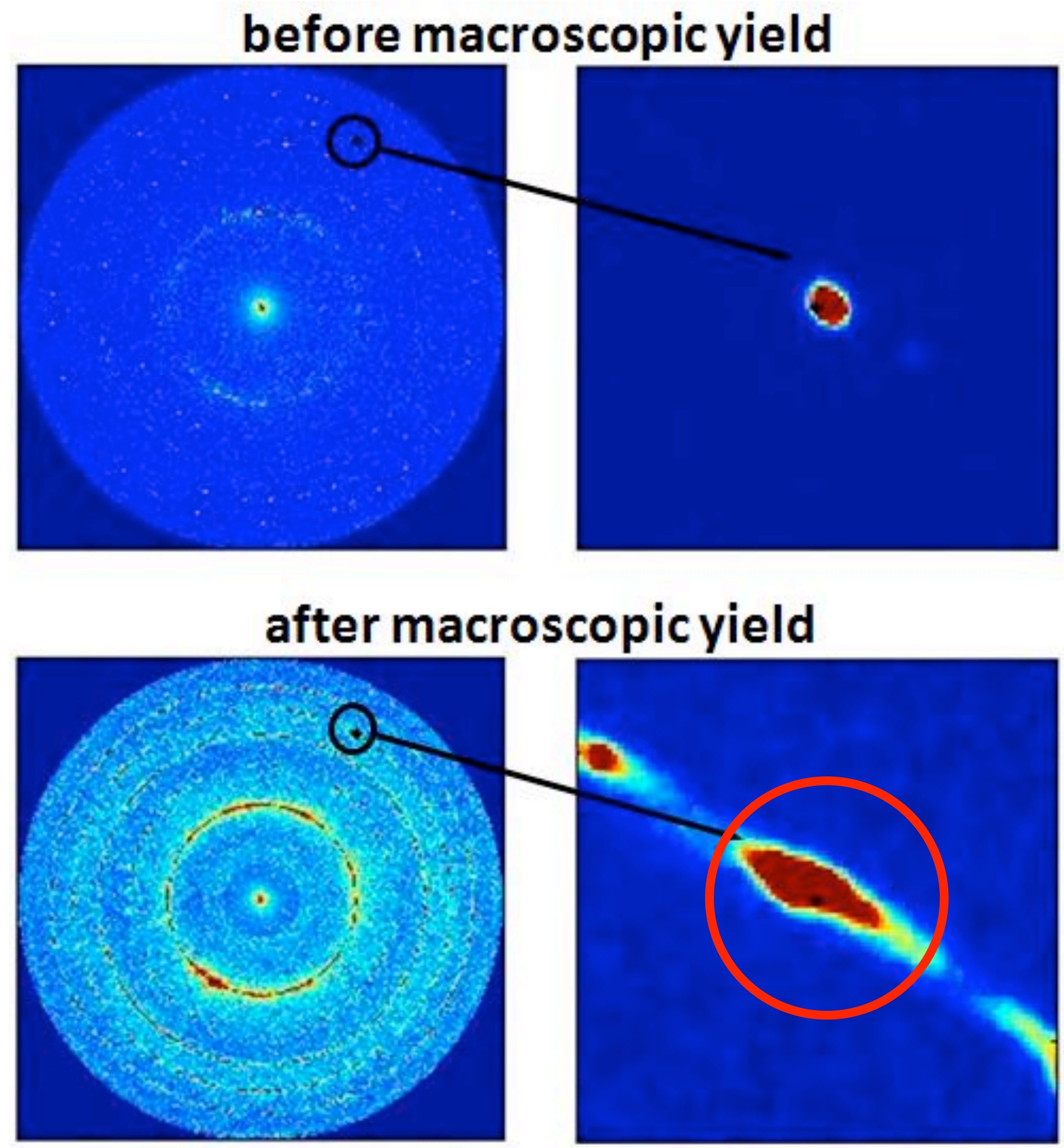
Forward Projection /
Virtual Diffractometer

- Put diffraction model into FEM
    - Accurately represent x-ray paths and detector
    - Distortion and orientation of virtual crystals within virtual diffraction data
    - Compare virtual and real detectors directly
- Hypothesis investigation
    - See impact directly



Diffracted Intensity Distribution - Far Field Detector

- Polycrystal Sample - rotate in ω
  - 100 - 1000 grains
  - 20 - 100 peaks per grain
- Each peak contains a projection of strain and orientation distributions within a grain
  - Post-yield "smearing" associated with plasticity - crystallographic slip
- To first approximation
  - Orientation spread: η (azimuthal) & ω
  - Strain spread: 2θ (radial) - traditional line broadening
- Moments of intensity distribution
  - Mean value (centroid)
  - Full Width Half Max (spread)



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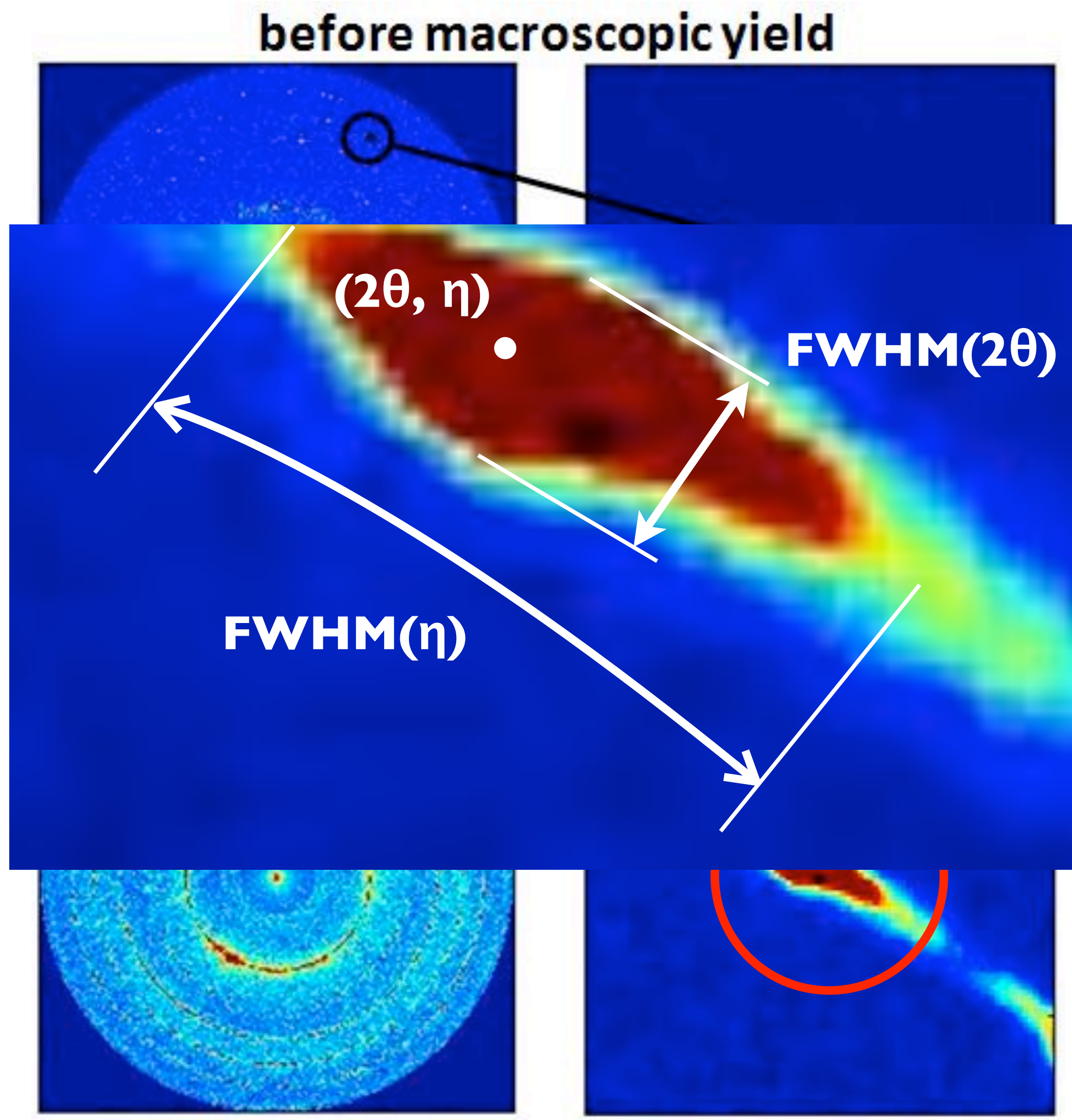
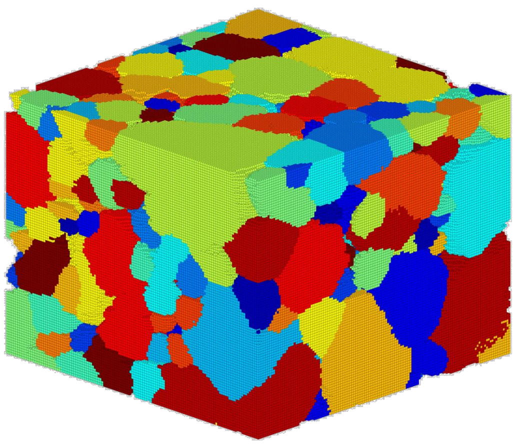
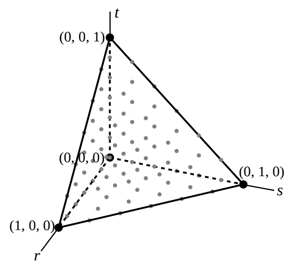
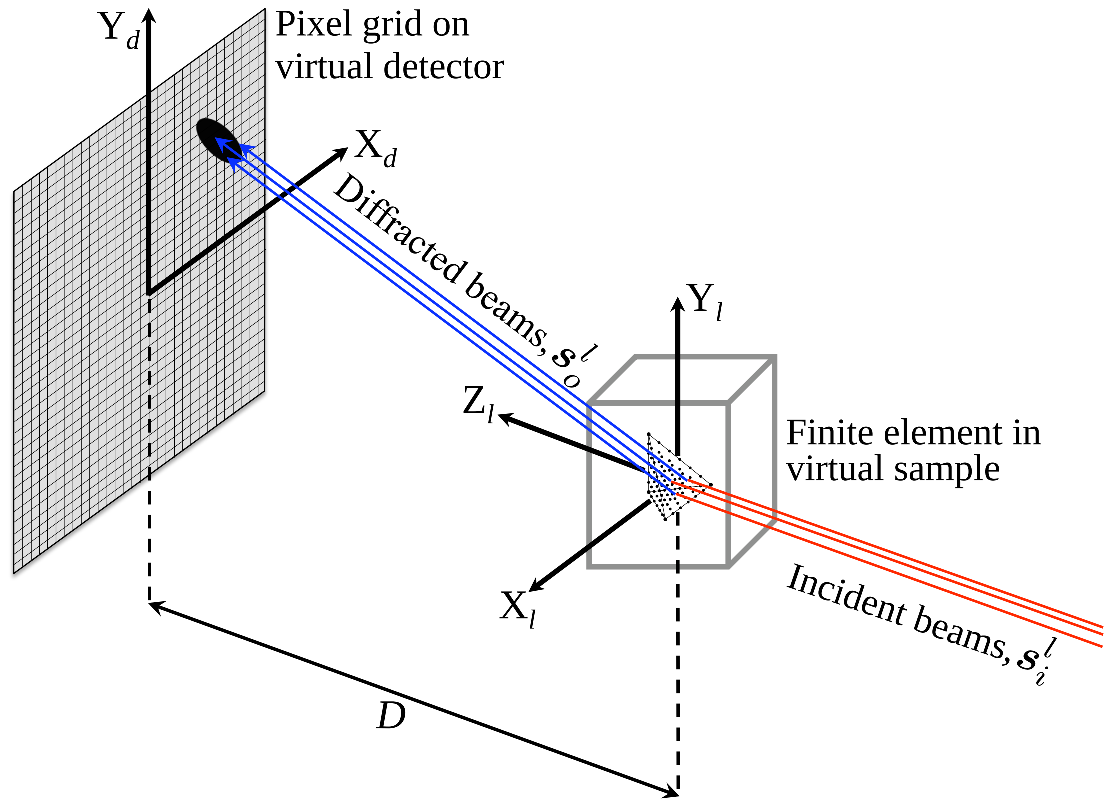


Finite Element Model - Virtual

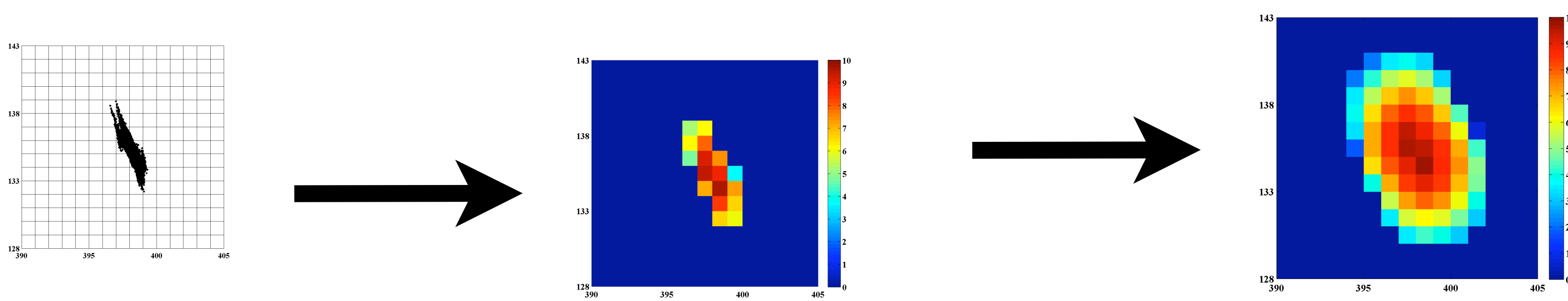






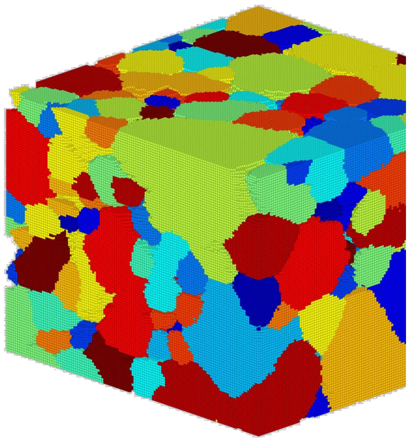
- Build a virtual polycrystal
  - Single crystal elasticity + plasticity
- ~100 scattering centers within each tetrahedral element
- Apply diffraction model - diffracted intensity projected onto detector (1)

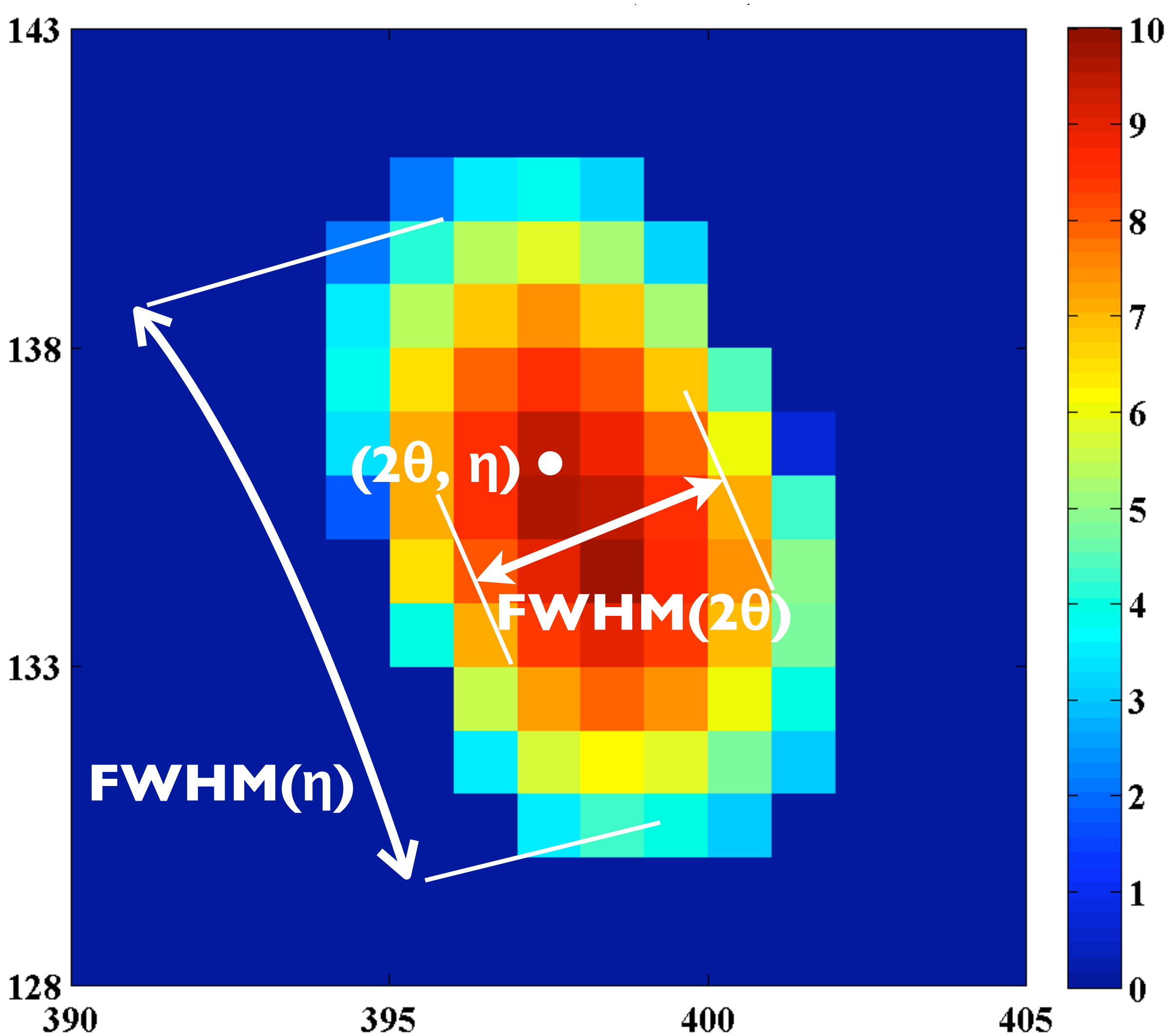
Wong et. al, 2013, Comp. Mat. Sci., 77, 456-466.

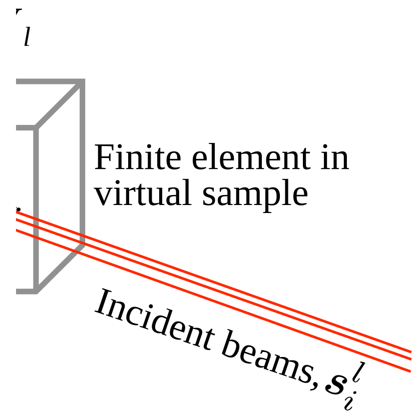


(1) (2) (3)

Finite Element Model - Virtual

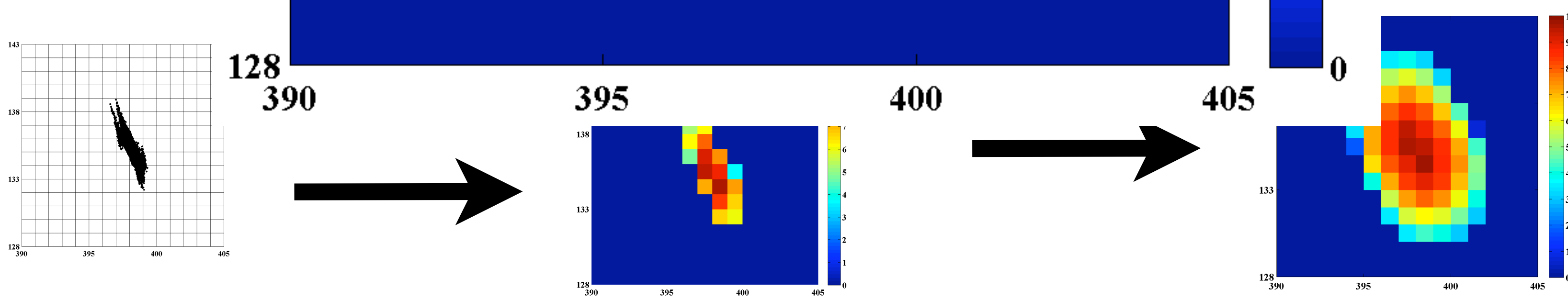






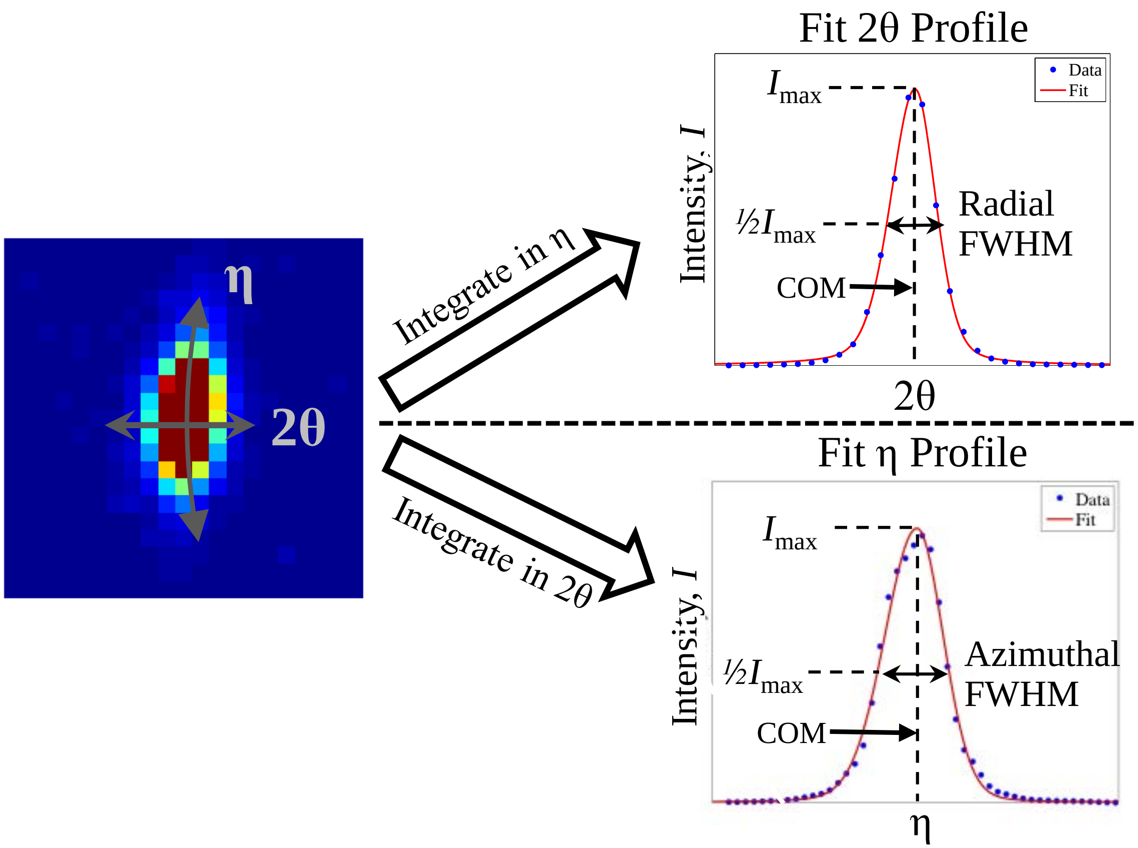
- Build a virtual p
  - Single crysta
- ~100 scattering
  tetrahedral elem
- Apply diffractior
  intensity project

lat. Sci., 77, 456-466.



(1) (2) (3)

Orientation and Strain Spread



- **From each peak (spot) extract simple center of mass (COM) and spread (FWHM) information**
  - **Radial - lattice strain**
  - **Azimuthal - orientation**
- **Using all spots for 1 crystal (20-100), compute *Θ* and *ζ***

$$(\Delta\eta_i^{FWHM})_n = (\eta_i^{FWHM})_n - (\eta_0^{FWHM})_n$$

$$(\Delta 2\theta_i^{FWHM})_n = (2\theta_i^{FWHM})_n - (2\theta_0^{FWHM})_n$$

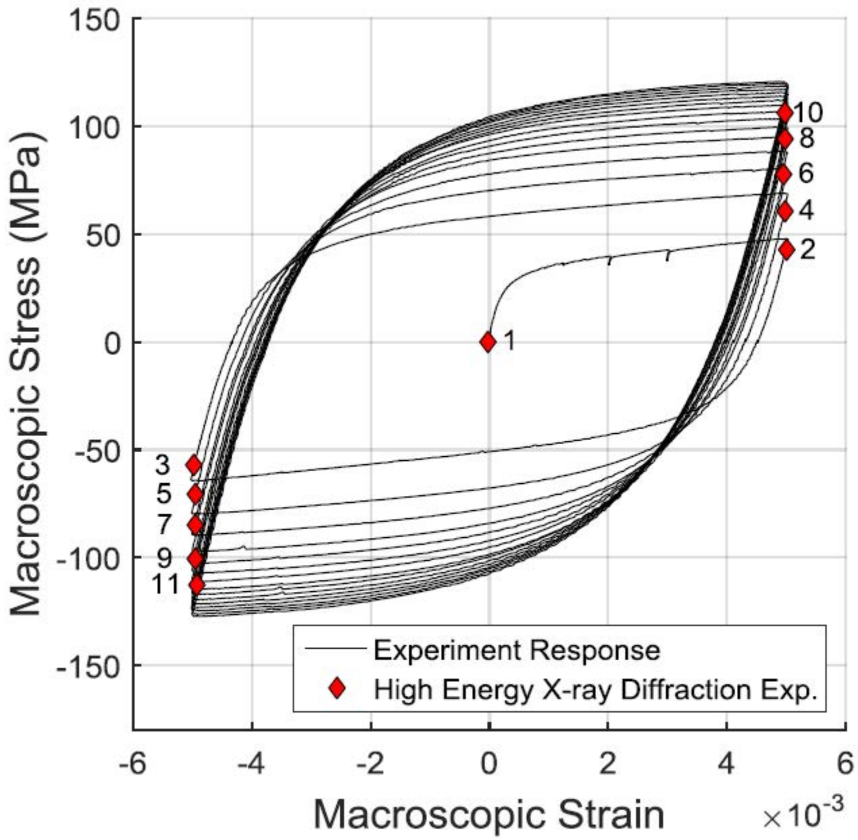
$$\zeta = \frac{1}{N}\sum_{n=1}^{N}(\Delta\eta_i^{FWHM})_n$$

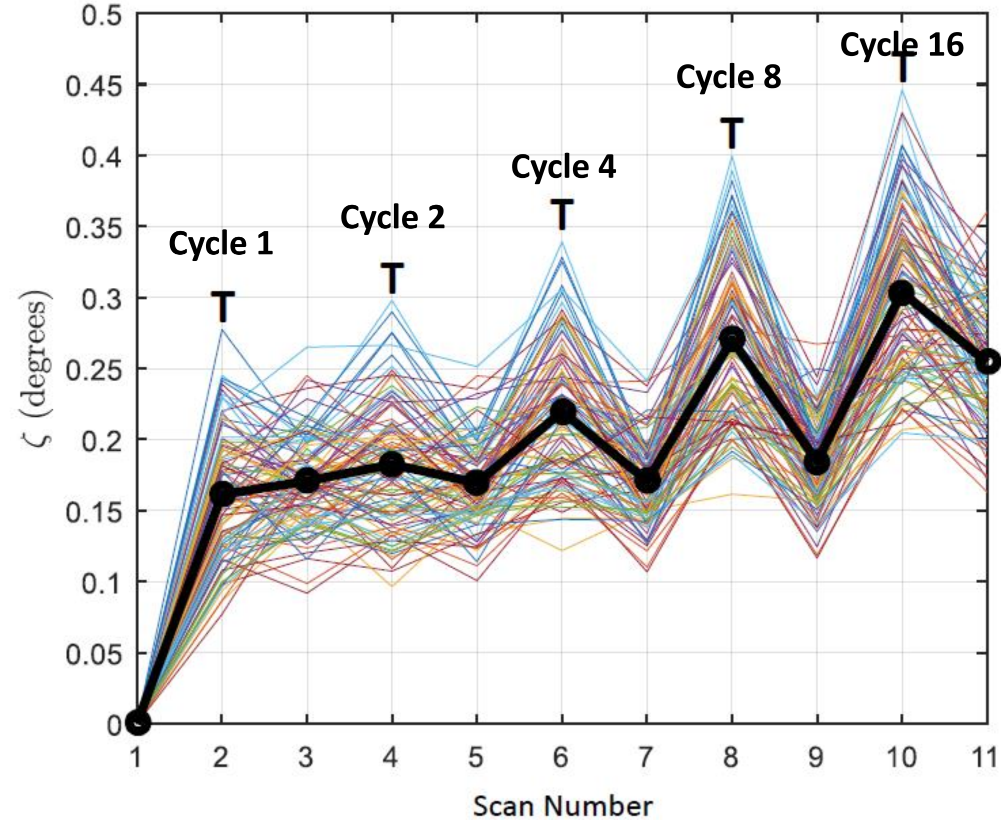
$$\Theta = \frac{1}{N}\sum_{n=1}^{N}(\Delta 2\theta_i^{FWHM})_n$$

Orientation

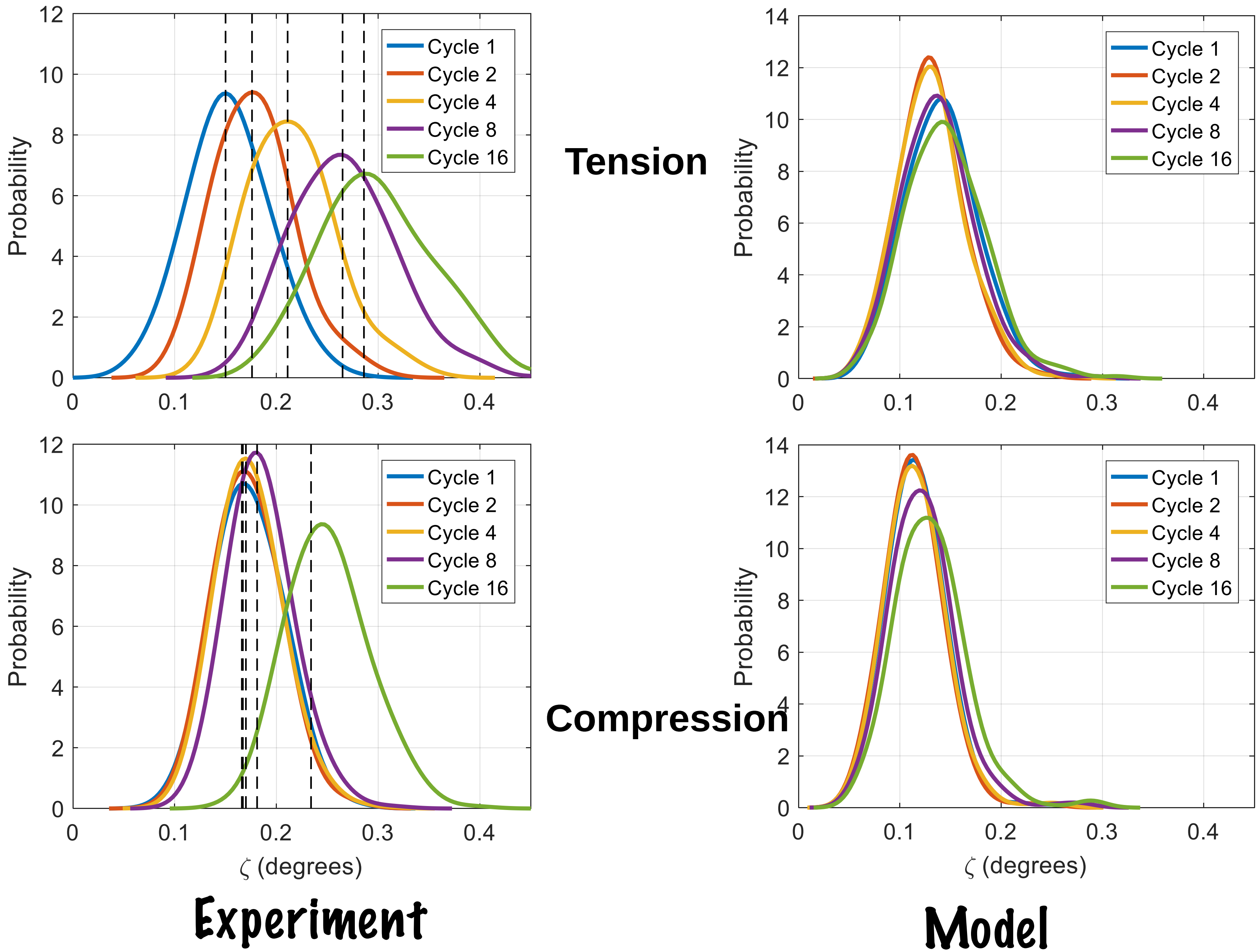
Lattice Strain

Evolution of ζ

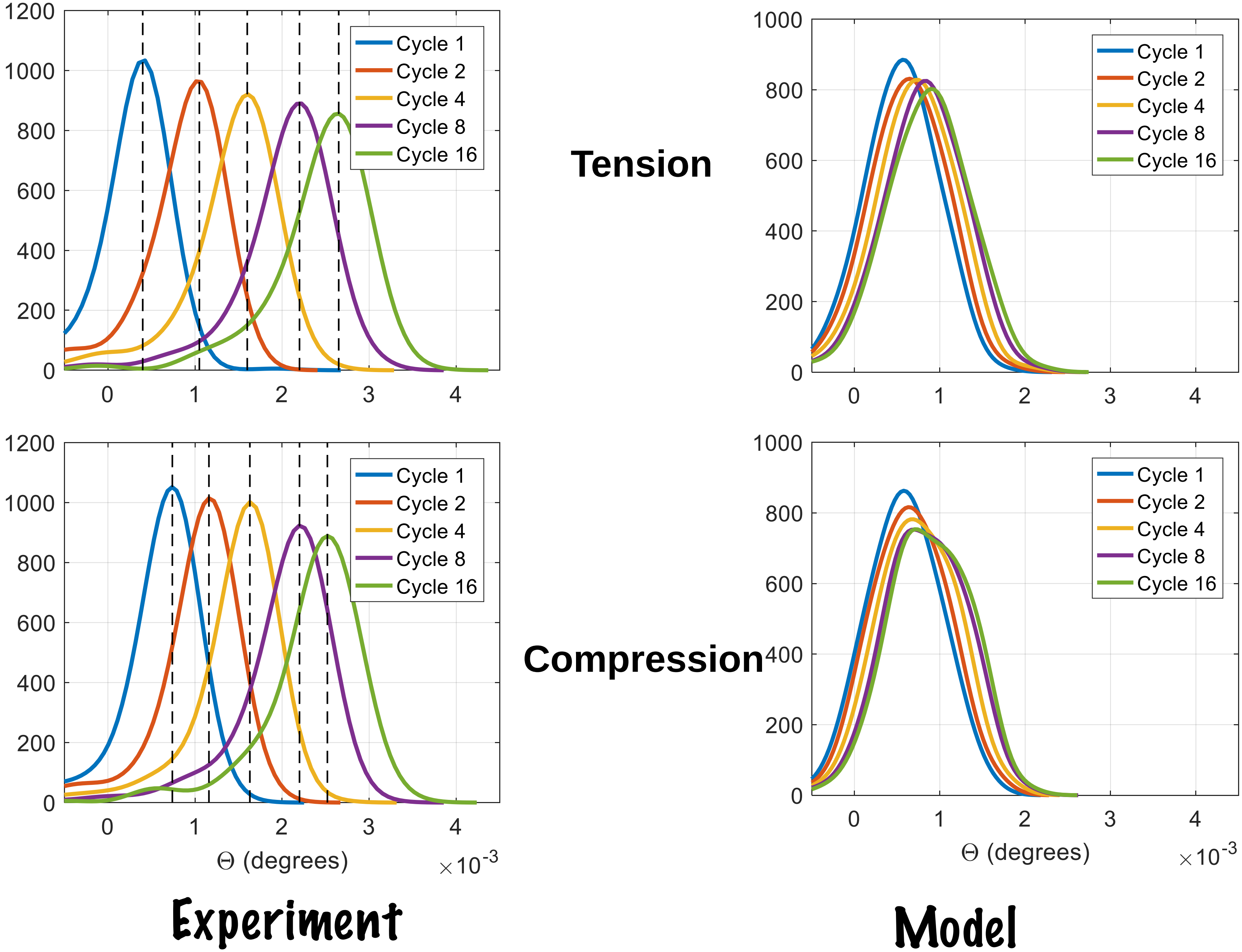




ζ Distribution over the aggregate



Φ Distribution (strains)



InSitμ@CHESS

- **Push the envelope of High Energy X-ray Diffraction (HEXD) methods**
  - **Create new methods, steal others**
- **Provide “enhanced support” of HEXD experiments**
  - **Meet designers, scientists, non-x-ray experts “half way”**
  - **Model and analysis support**
  - **Form partnerships: industry, national labs, university faculty**
- **Spectrum of Methods and Applications**
  - **Residual Stress**
    - Thick sections
    - Stress + chemistry
    - AM parts
  - ***In situ* Fatigue Crack Growth**
  - **Other *In situ* conditions**

CAT - First Beamtime Fall 2014

InSitμ People

- **Armand Beaudoin:** InSitμ Associate Director, UIUC emeritus Prof., distinguished industrial career, experiment/model interface
- **Darren Pagan:** Staff scientist, novel HEXD methods / data analysis / upgrade
- **Chris Budrow:** CHESS GRA working on residual stress
- **Ramya Nair:** Post-Doc: working on fracture in cement
- **Kelly Nygren:** Post-Doc: blending EM and HEXD
- **Eric Miller:** Tufts ECE Prof., signal processing, data science


Budrow


Pagan

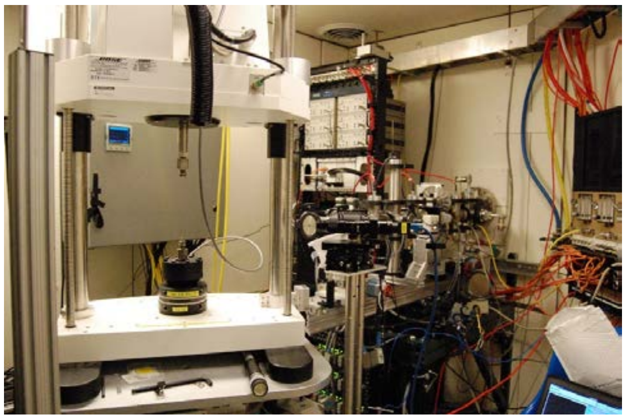

Beaudoin


Nair


Nygren

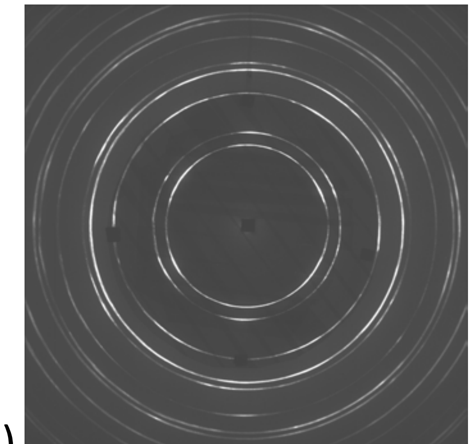

E. Miller

Fatigue crack growth - Aluminum

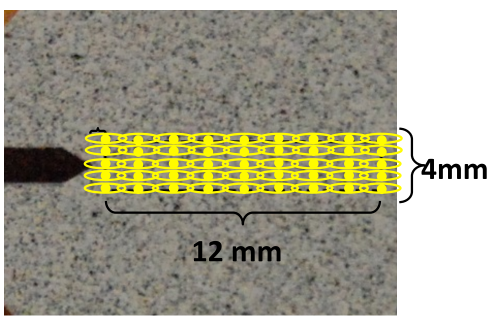


(a)

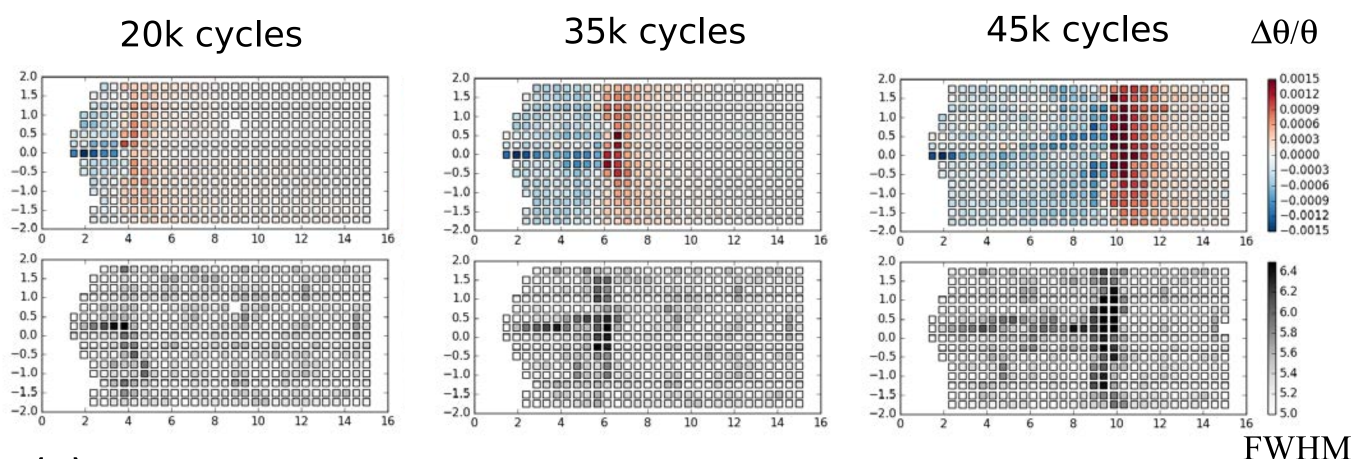
(b)



(c)



(d)

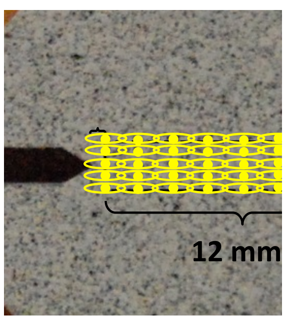


(e)

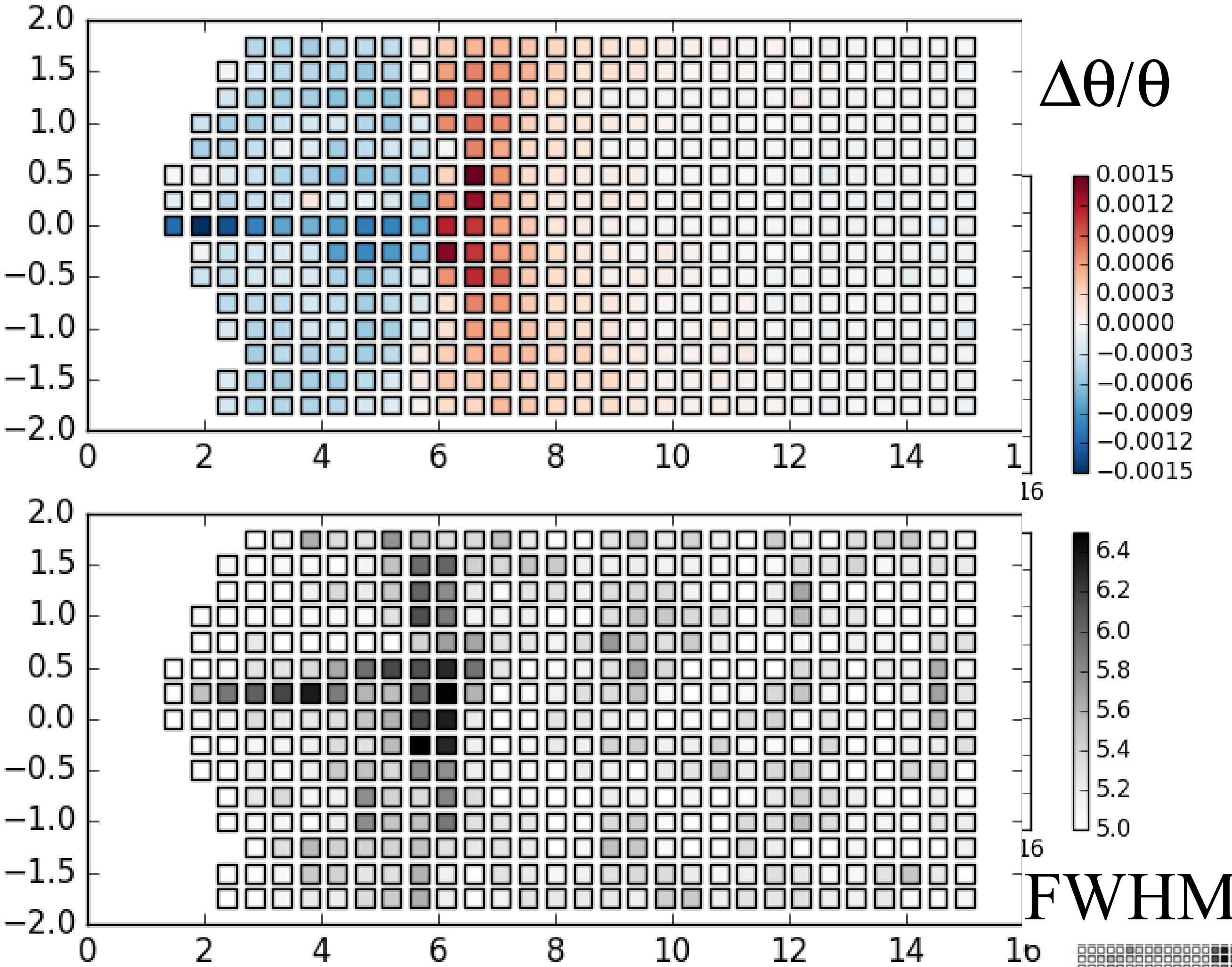
Fa n

35k cycles

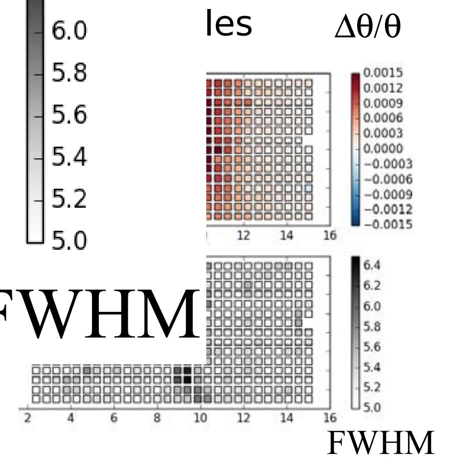
(a)



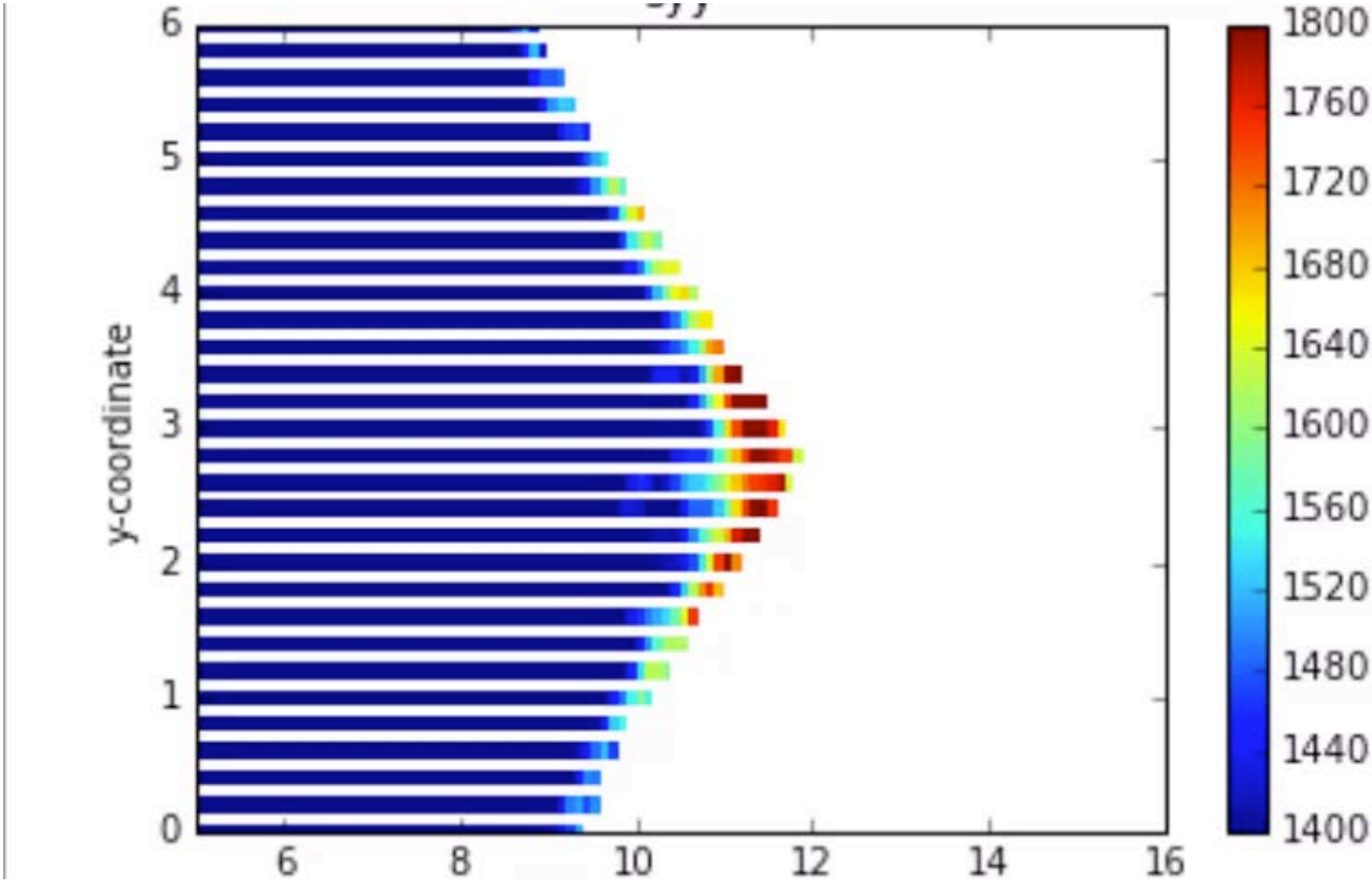
(d)



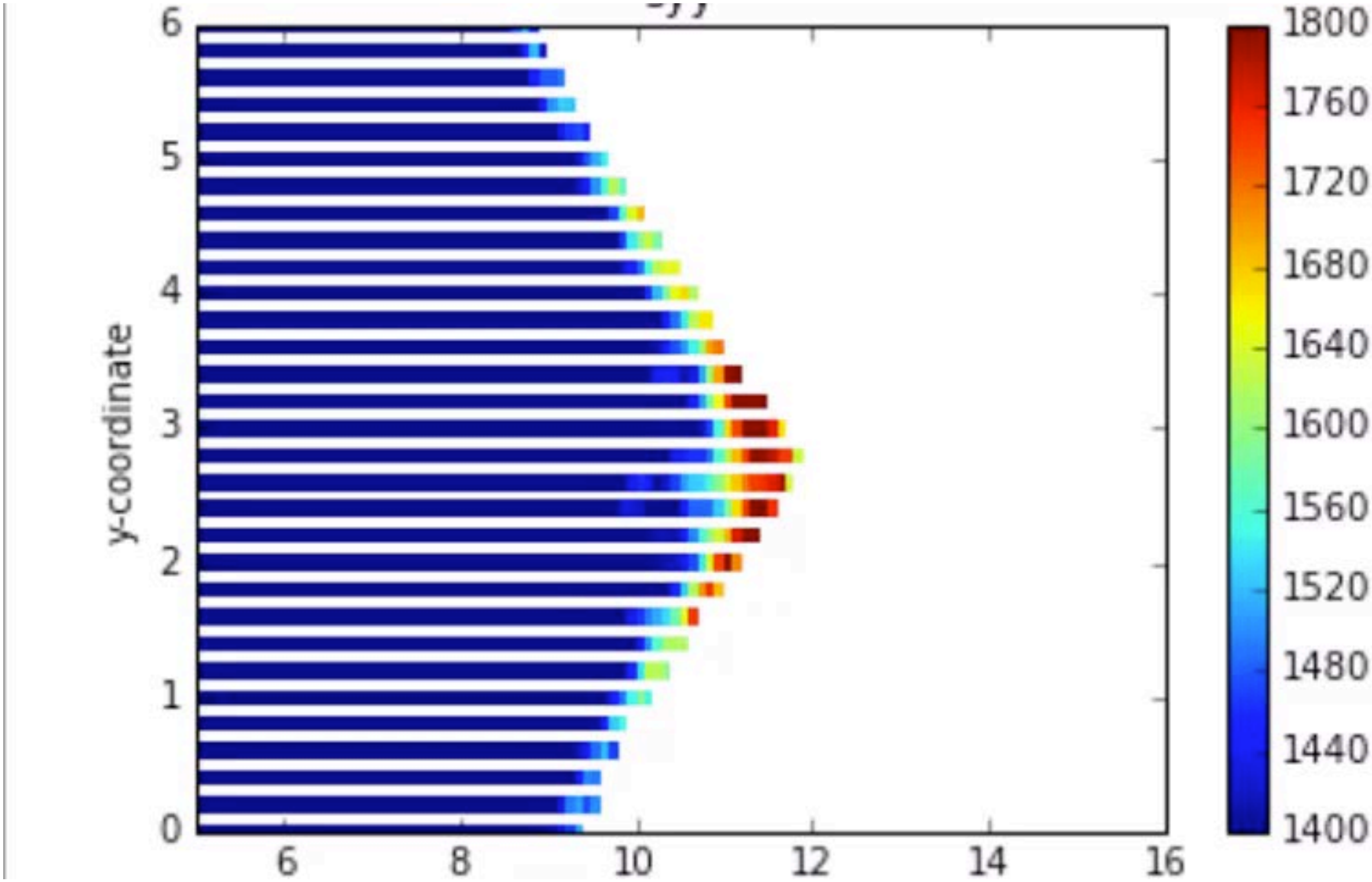
(e)



FCG in Ti-6-4 (Pilchak)



FCG in Ti-6-4 (Pilchak)



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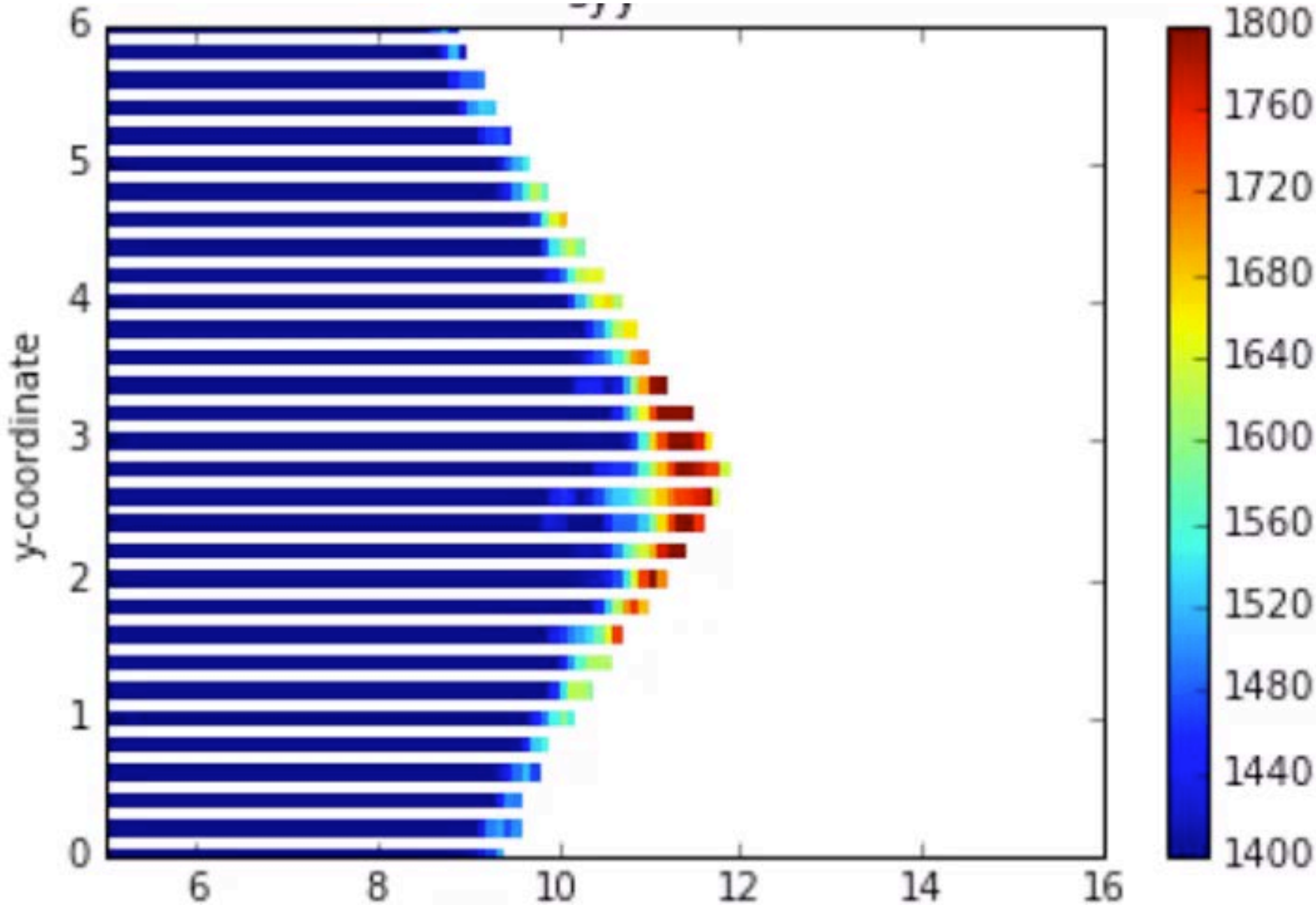
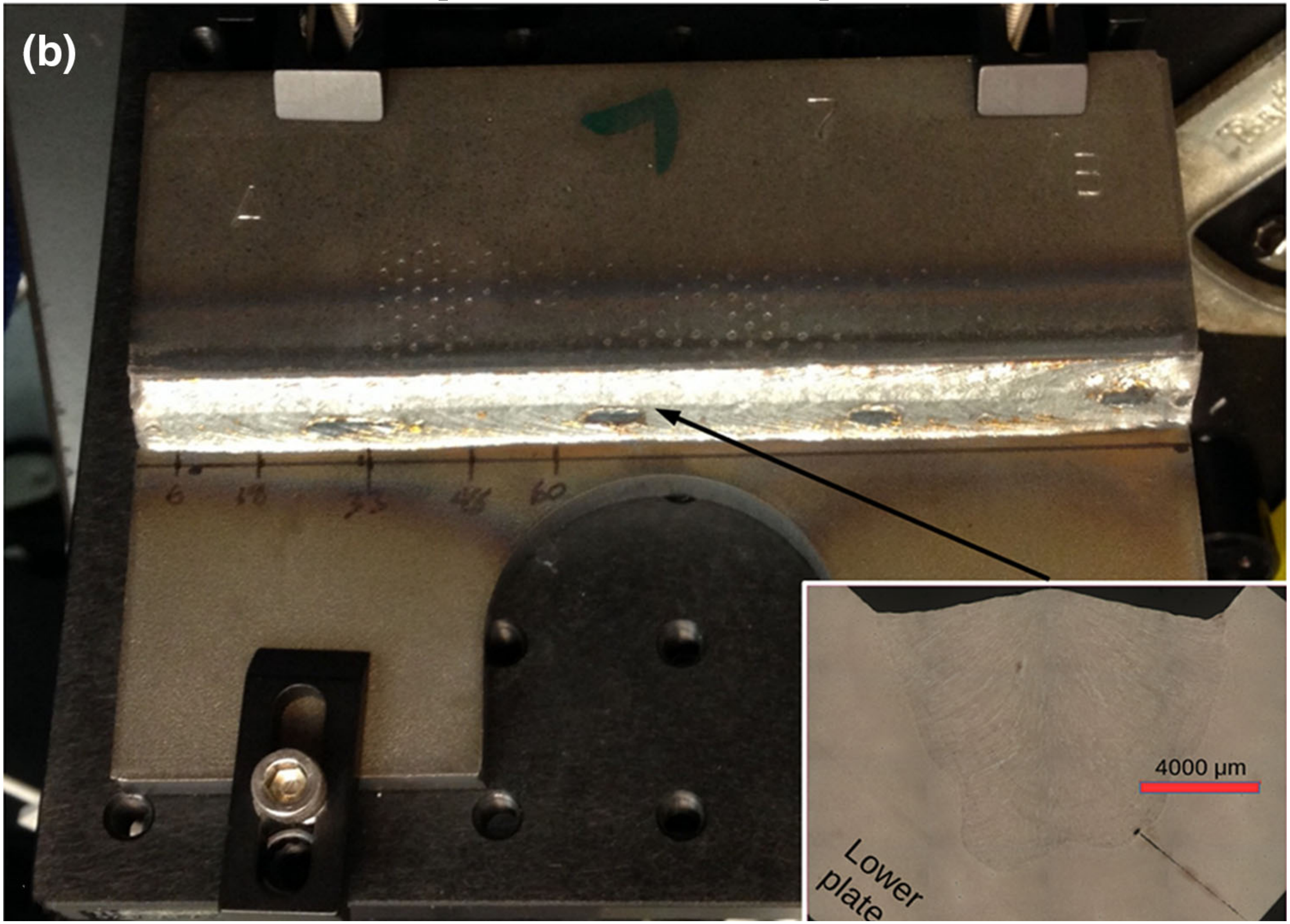


Welding Residual Stress Measurement Results

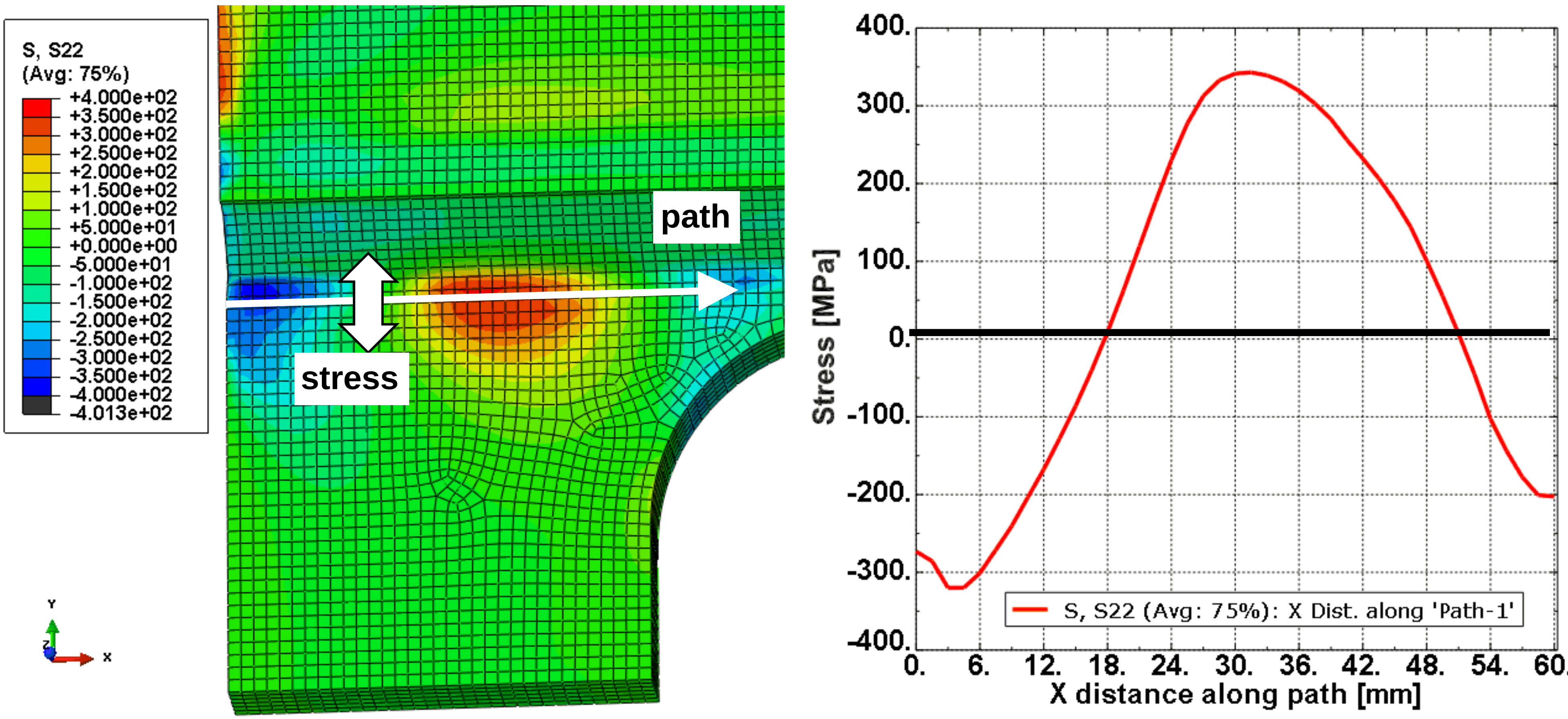
Justin Mach, Senior Engineer, Caterpillar
Armand Beaudoin, Industrial LIaison, InSitµ@CHESS
Matt Miller, Director, InSitµ@CHESS

Darren Dale, CHESS F2 Beamline Scientist / Associate Director, InSitµ@CHESS
Peter Ko, CHESS Research Associate
Graduate Research Assistants (Cornell University): Darren Pagan, Mark Obstalecki, & Chris Budrow
Graduate Research Assistant (University of Illinois at Urbana-Champaign): Kamalika Chatterjee

Lap Joint Sample



CAT Simulation result

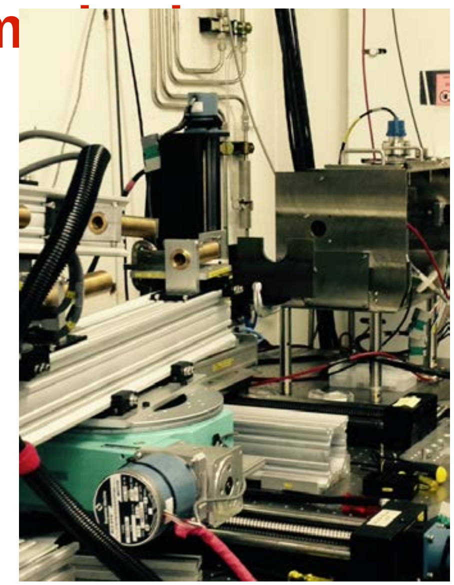


Residual stress resulting from welding process simulation.

CATERPILLAR®

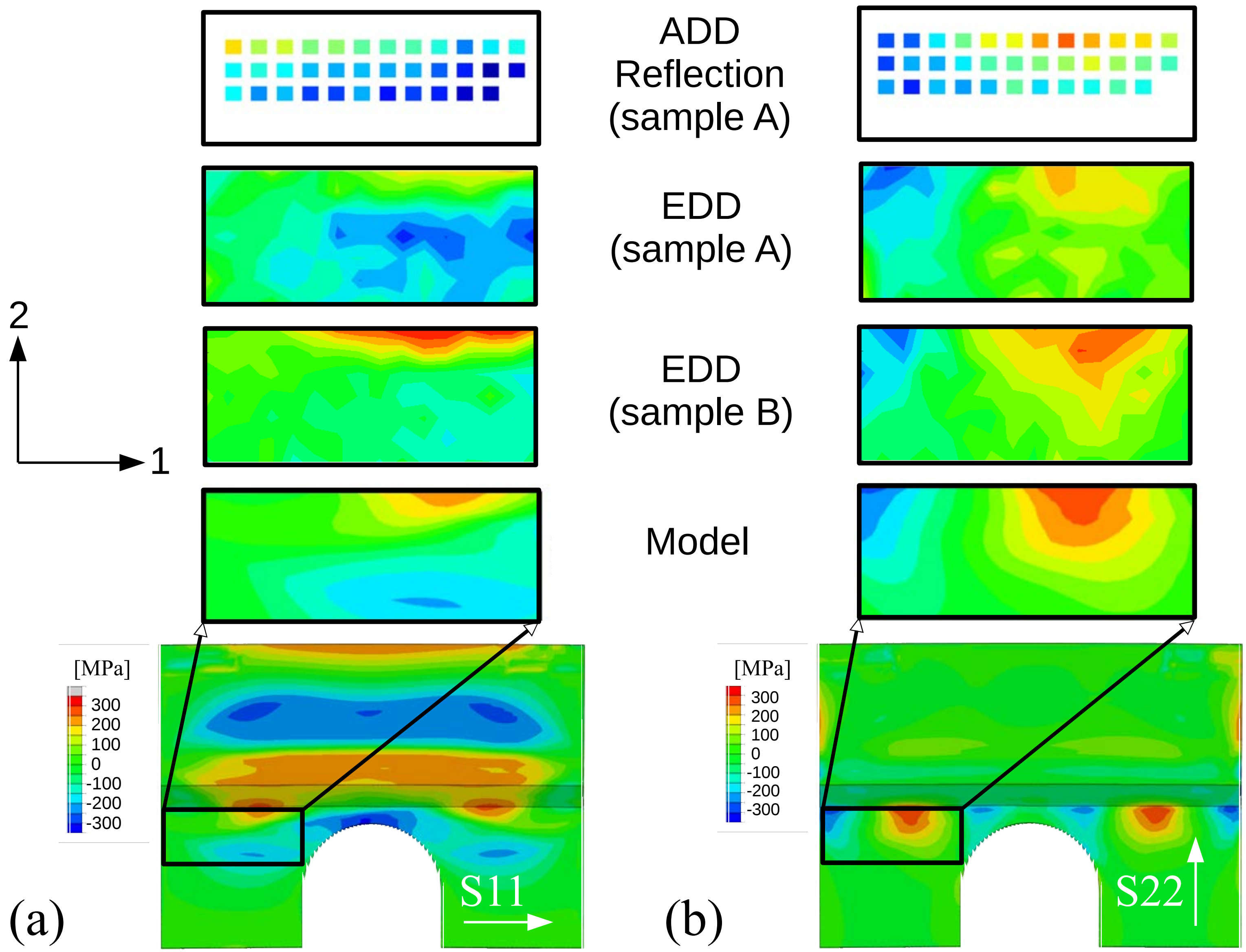
CAT Lap joint sample experimental plan

- **Welding model validation**
  - Simple sample "representative" of a real weld
  - 1/4" steel plate
- **Monochromatic reflection geometry**
  - Traditional $\sin^2\psi$ analysis
  - Replicate lab source experiment
  - Vary energy
  - CHESS F2
- **Polychromatic Energy Dispersive Diffraction (EDD) m**
  - Penetrate through the plate
  - Interrogate surface layer
  - Plane stress - rosette analysis
  - Advanced Photon Source (APS)



Mach, et. al.,JOM, 69:5, 393-399, 2017.

Summary of CAT Results



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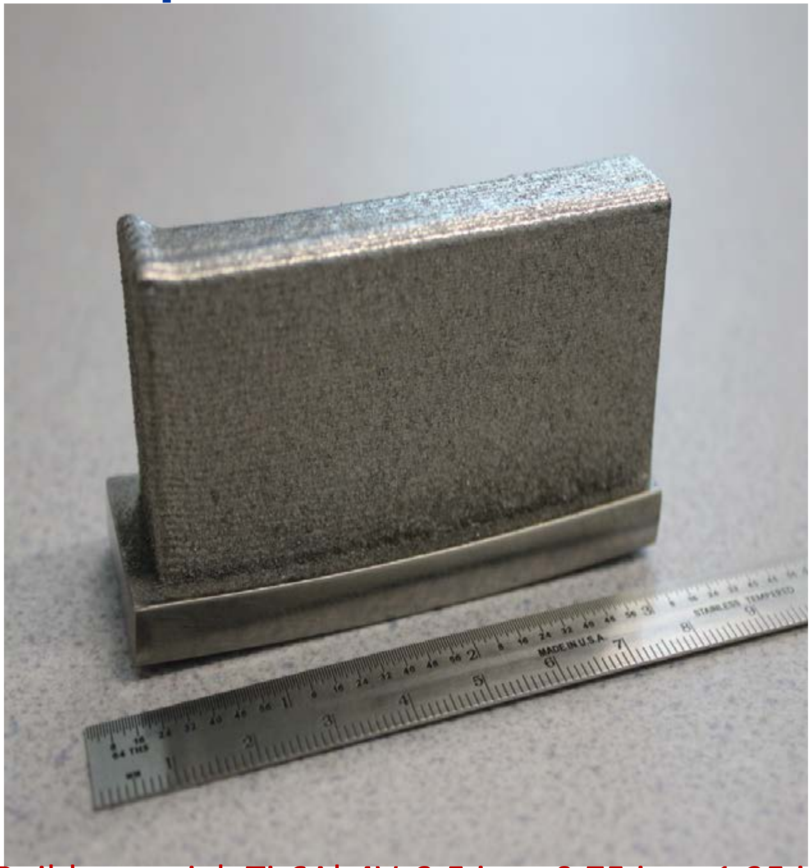
Additive Manufacturing: Residual Stress and Distortion

Distortion Example (Blown Powder)

Optomec LENS MR-7



Build material: Ti-6Al-4V, 3.5 in. x 0.75 in. x 1.25 in. (L x W x H)

Substrate: Ti-6Al-4V, 4 in. x 1 in. x 0.5 in. (L x W x H)

Residual Stress Induced Cracking

Powder Bed: EOS

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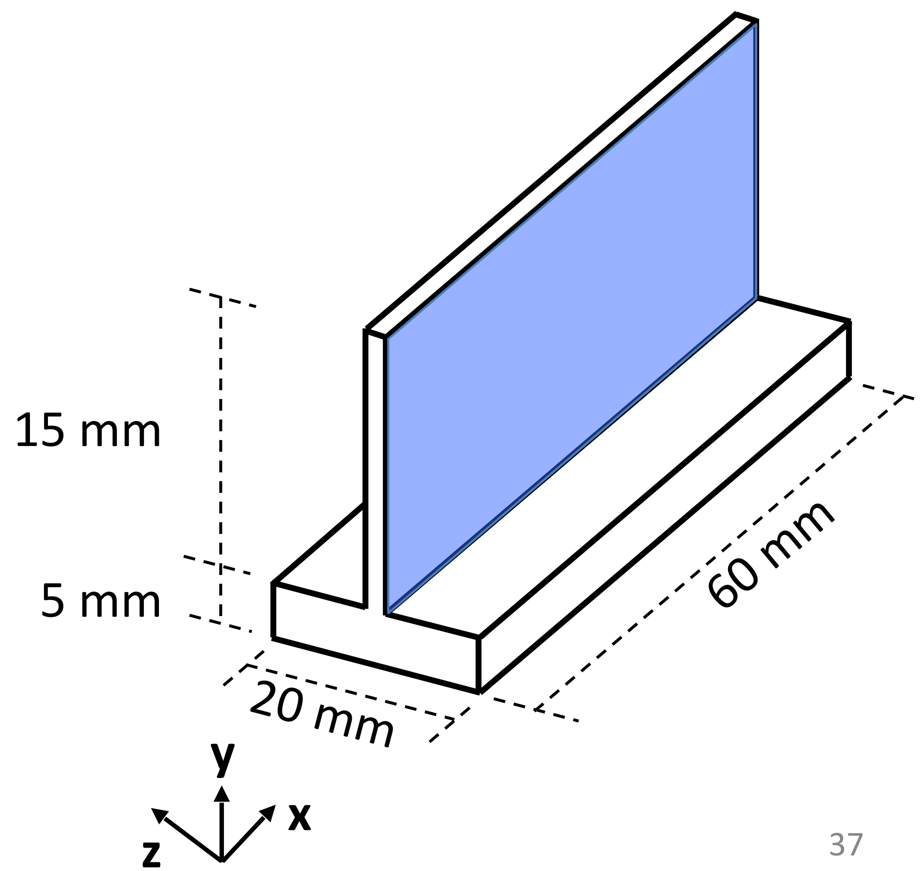
Courtesy Fred Lia and Wesley Mitchell
CIMP 3D, Penn State Univ.
& Jim Williams – Ohio State Univ.

Residual Stresses in AM Materials

- Monochromatic powder
- Measure gradient
  - Map strain over thin flange
  - Scans on grid of 0.5 x 0.5 mm
    - **3600 Measurements!**
- Thin section of sample
  - Plane Stress
- Sample rotated 180 degrees about y-axis
  - Stress result is average of "front" and "back" data.
- White beam measurements
  - Not shown here



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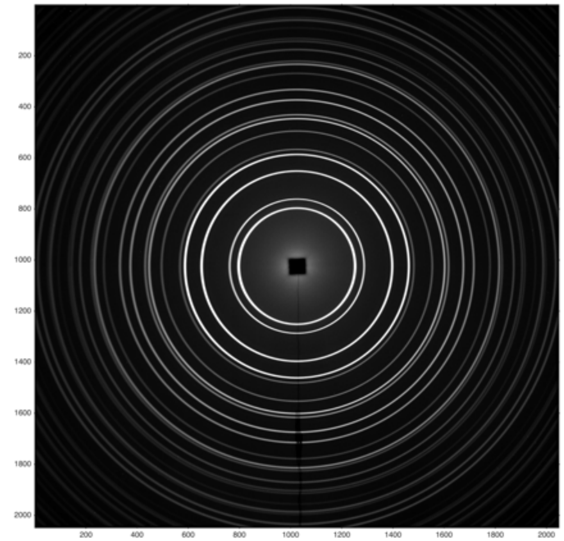

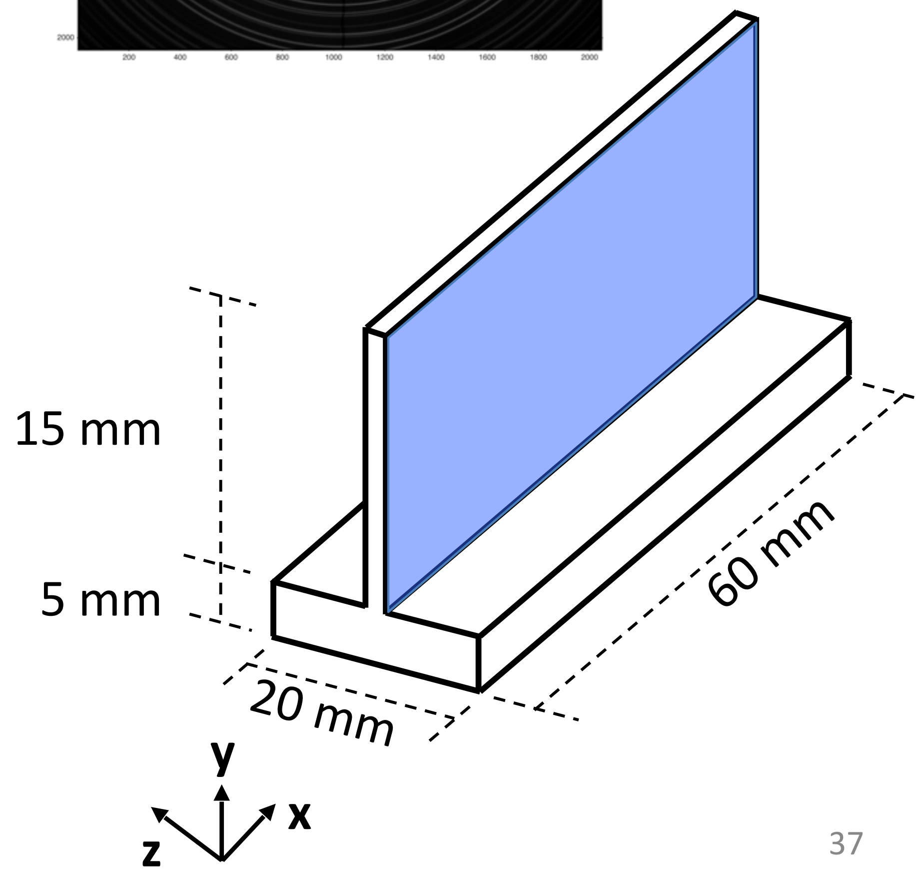

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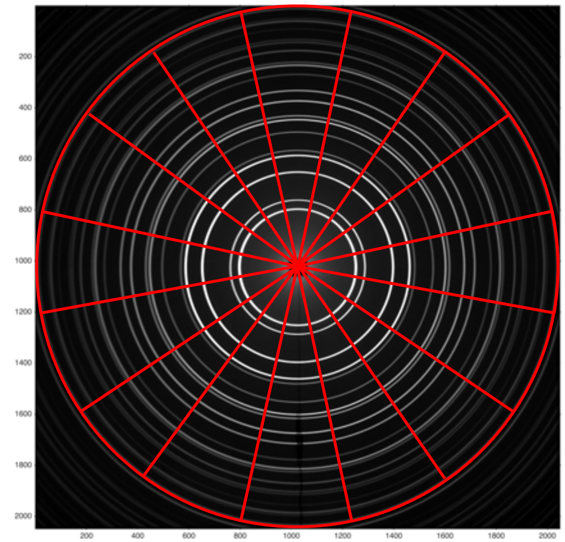
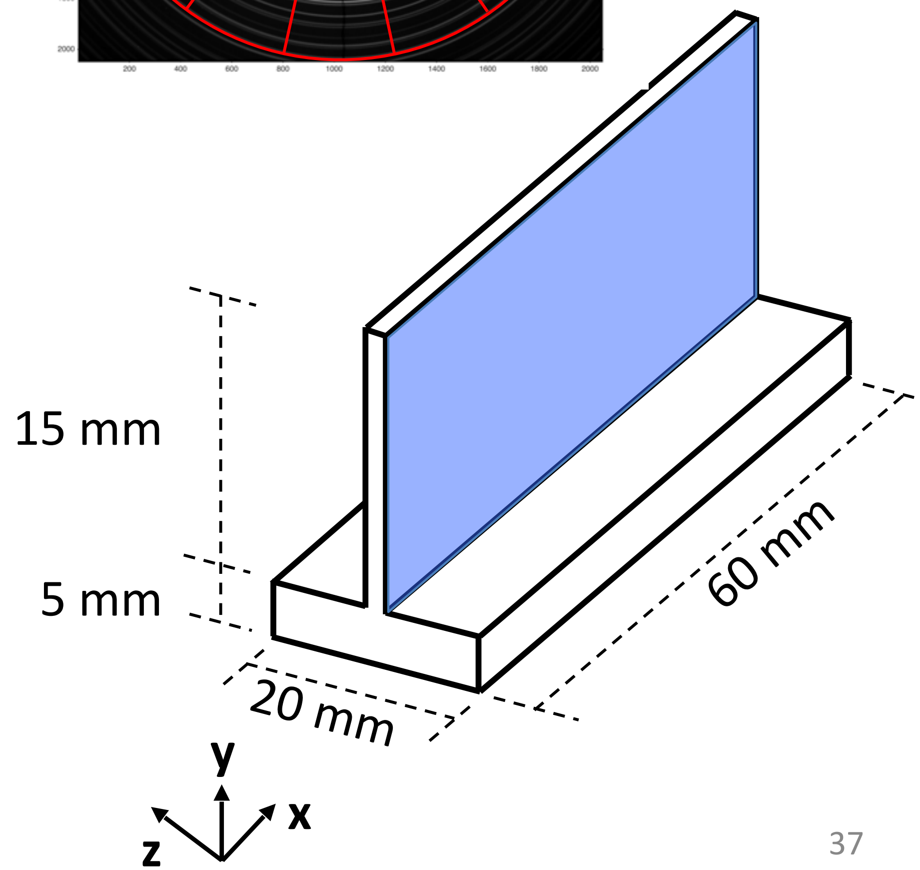
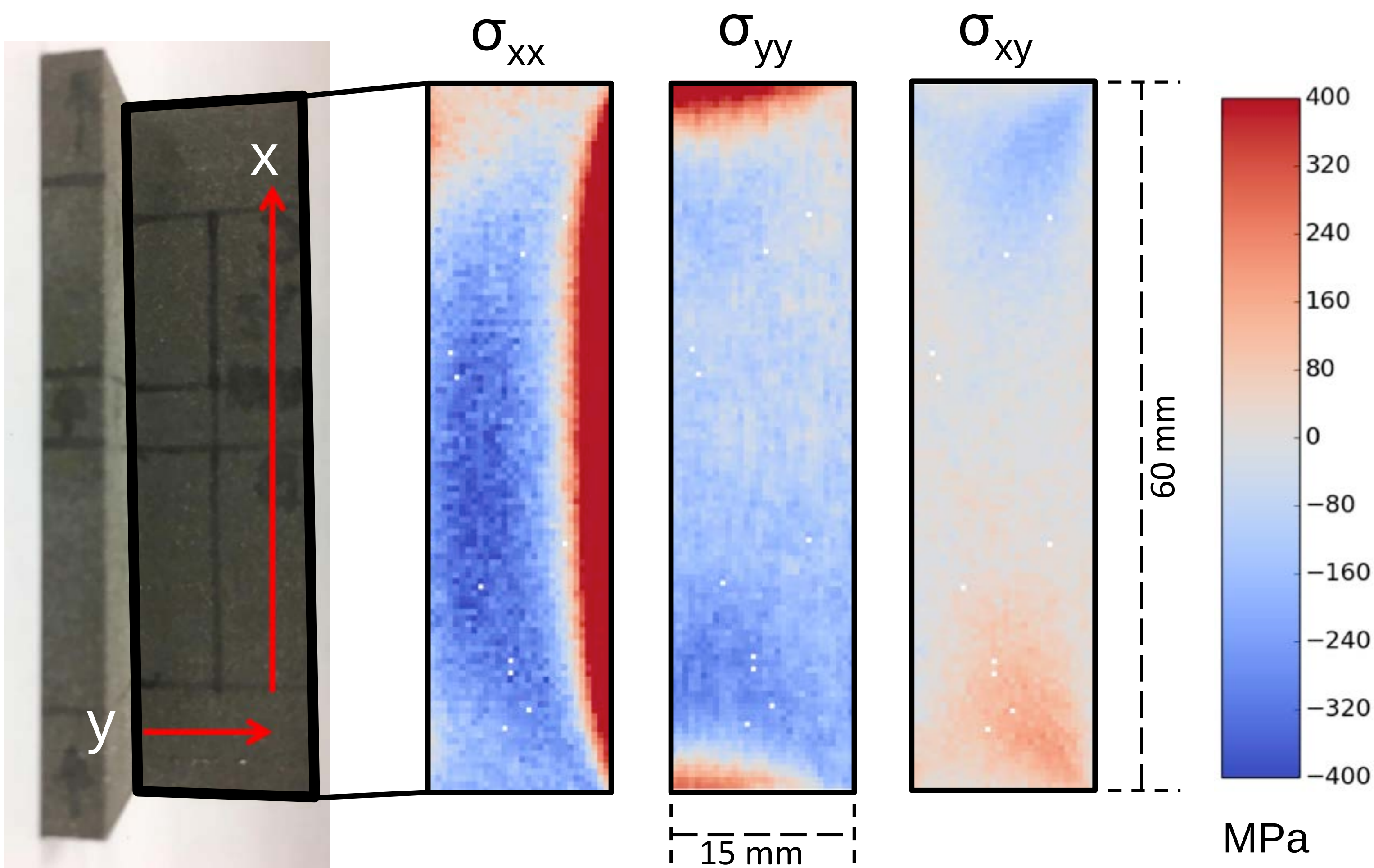


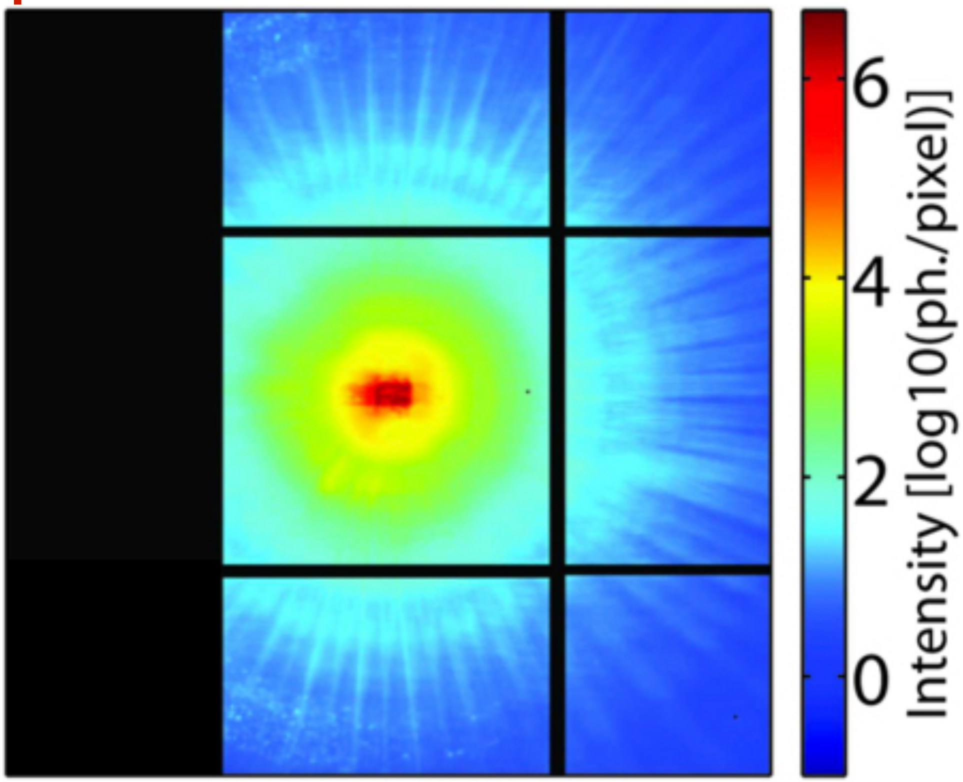


Additive Manufacturing: Results

- [1 1 0] reflection used to compute strain, with diffraction ring broken up into 10 degree arcs (for peak fitting).
- Isotropic Elasticity applied to calculate stress: E = 114 GPa, v = 0.342
- Boundary conditions were used to adjust lattice parameter, adjusting so that normal stresses at corners are zero.

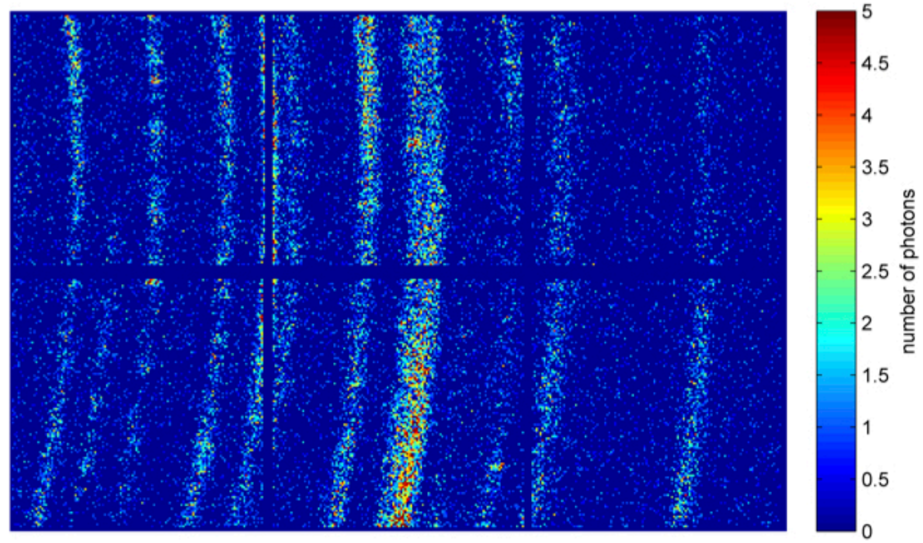


Detectors - new speeds and ranges



Coherent Diff 100 milliseconds

Brazing - 2 milliseconds



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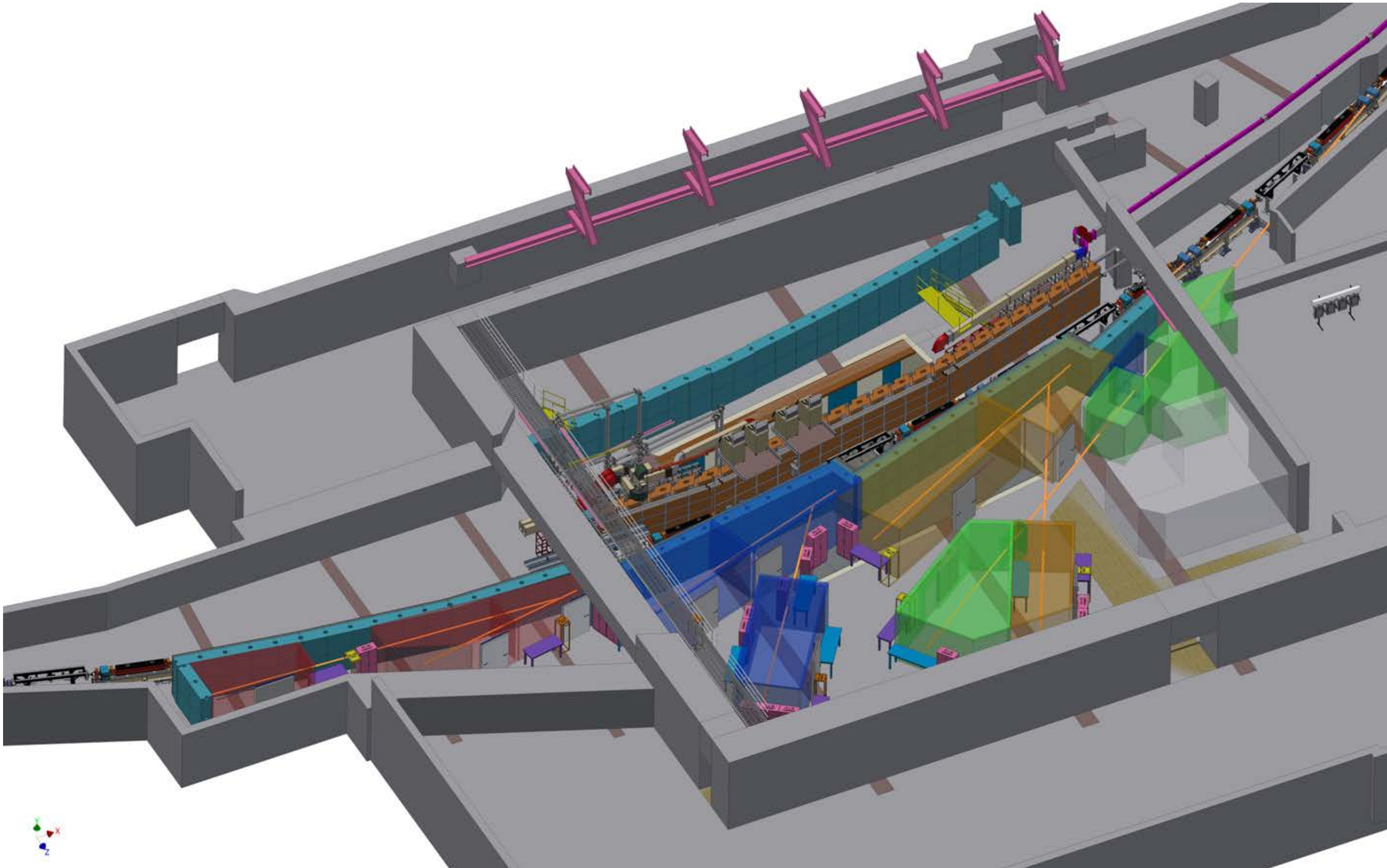
MM-PAD Detector: Sol Gruner, Cornell

- **150 micron pixels**
- **38mm x 57mm**
- **(Left) 10^7 x-rays / second; dynamic range 1 - 10^6 photons**
- **(Right) Seeing Al_3Ni Debye rings with 5 photon range**
- **CdTe for high energy**

MM-PAD Detector

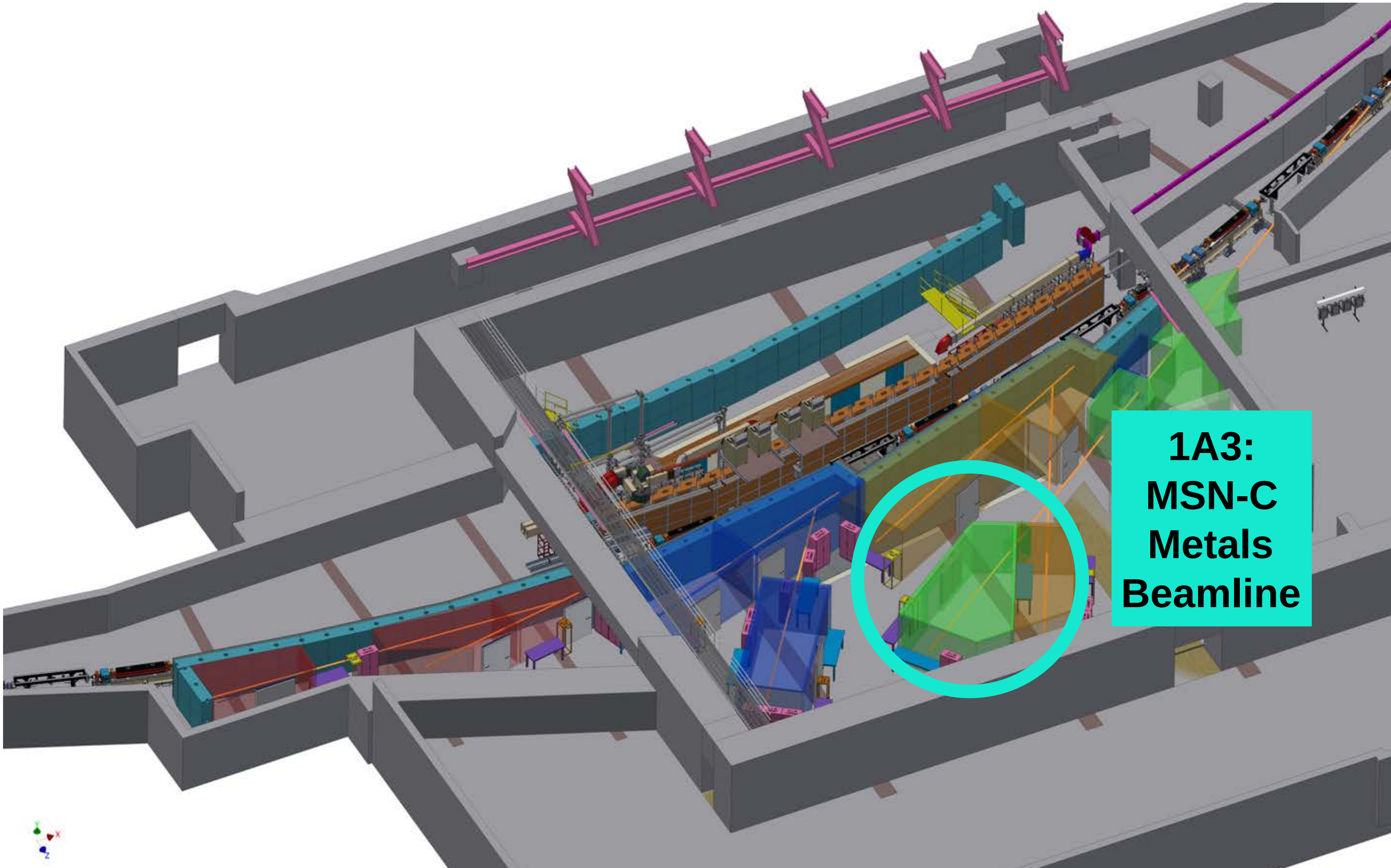
Tate et. al, Journal of Physics: Conference Series, 425(6): 062004, 2013. Giewekemeyer et. al, J. Synch. Rad 21(5): 1167–1174, Sep 2014.

CHESS-U Stations   MSN-C Beamlines



Cornell University
Cornell High Energy Synchrotron Source

CHESS-U Stations MSN-C Beamlines



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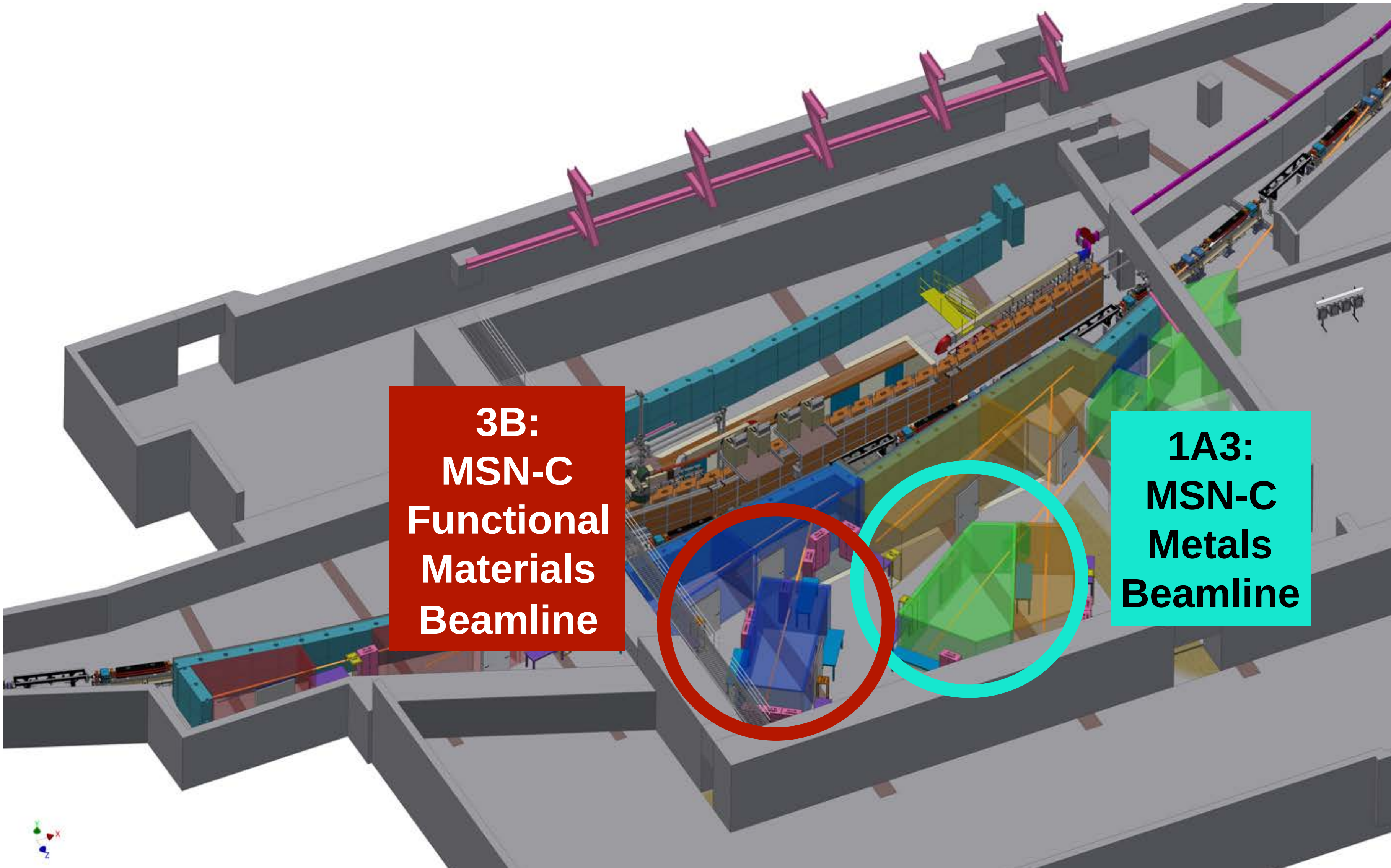

Conclusions / Advice

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