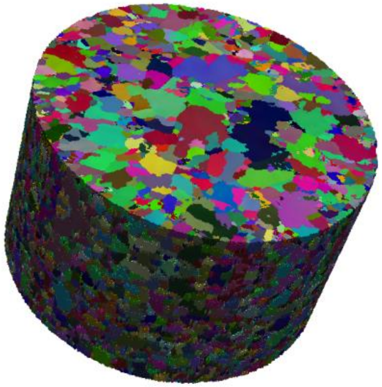
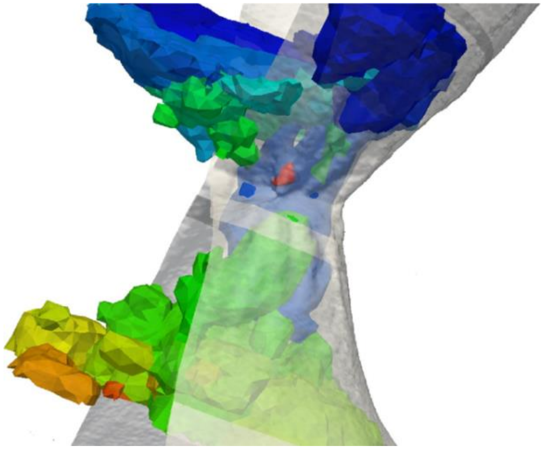
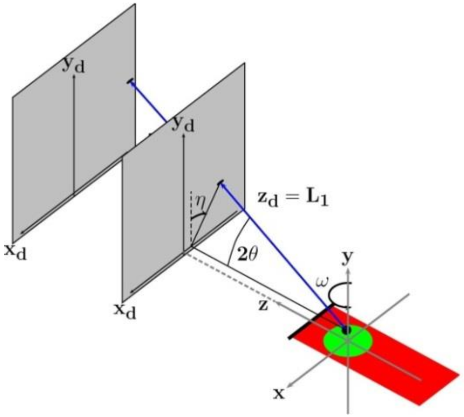
High Energy Diffraction Microscopy at Sector 1:
An Inside View of Materials' Responses

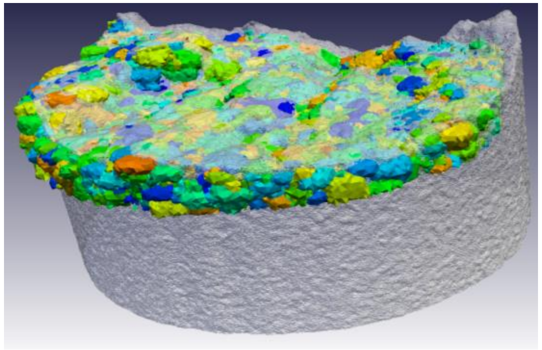




Bob Suter
Carnegie Mellon University
and
Visiting Scientist at APS

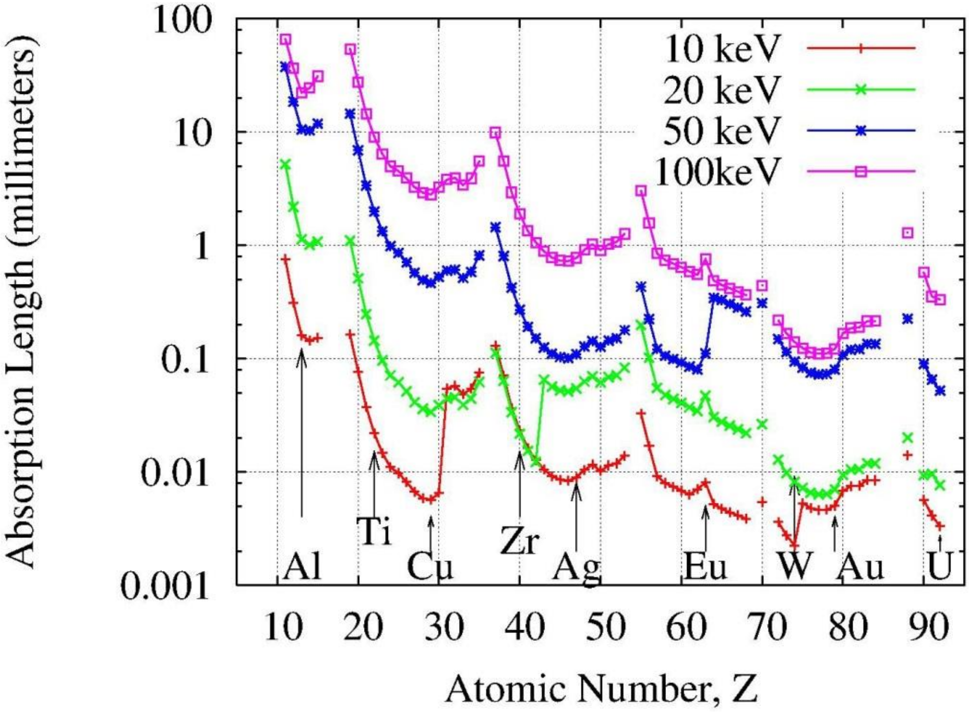


Thanks to:
Linda Young
Jay Schuren (AFRL)
Sector 1 staff
CMU graduate students



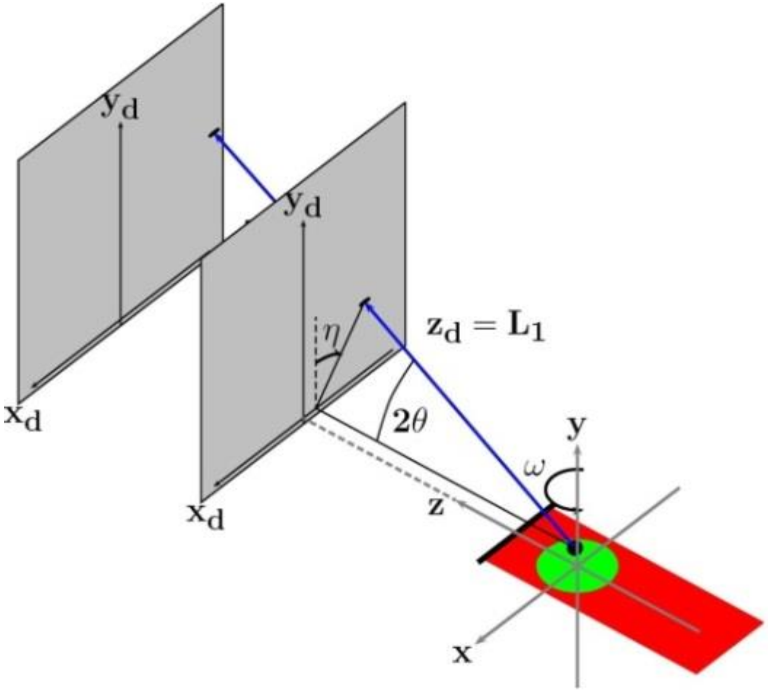
High Energy X-rays: > 50 keV

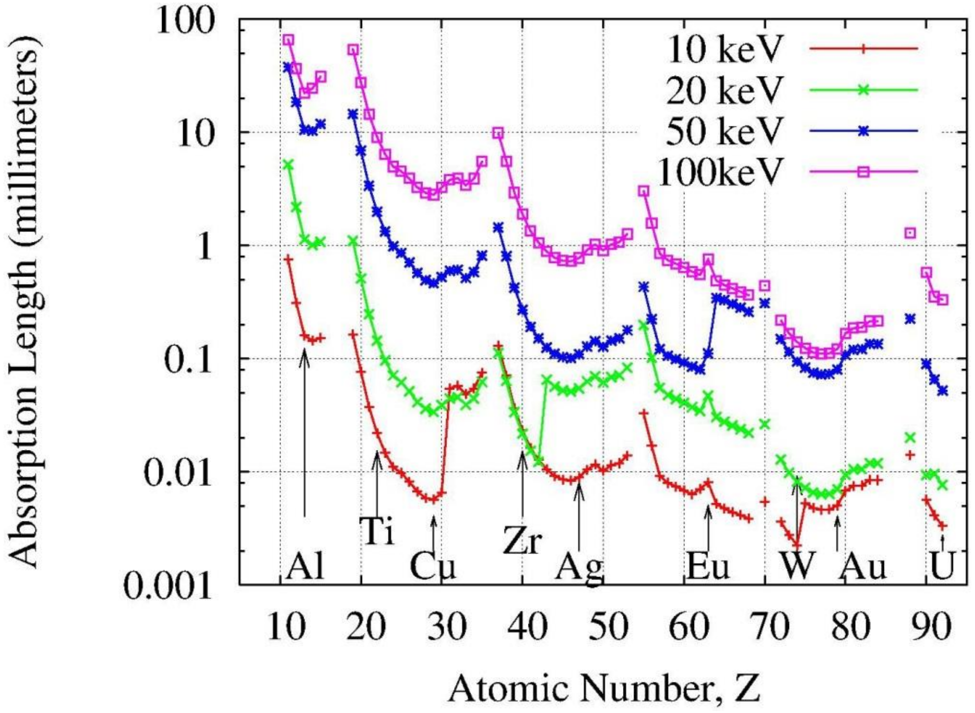
- **Penetrate millimeter dimensions**



High Energy X-rays: > 50 keV

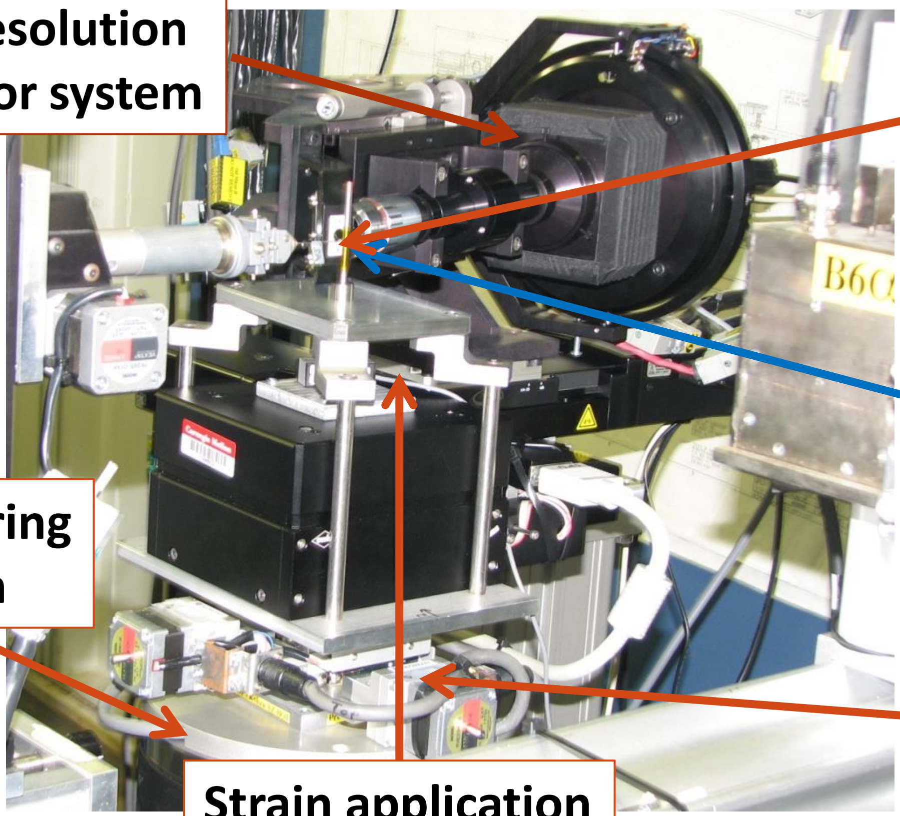
- **Penetrate millimeter dimensions**
- **Bragg diffraction at small angles**
- **Large reciprocal space coverage with small detector and one rotation**





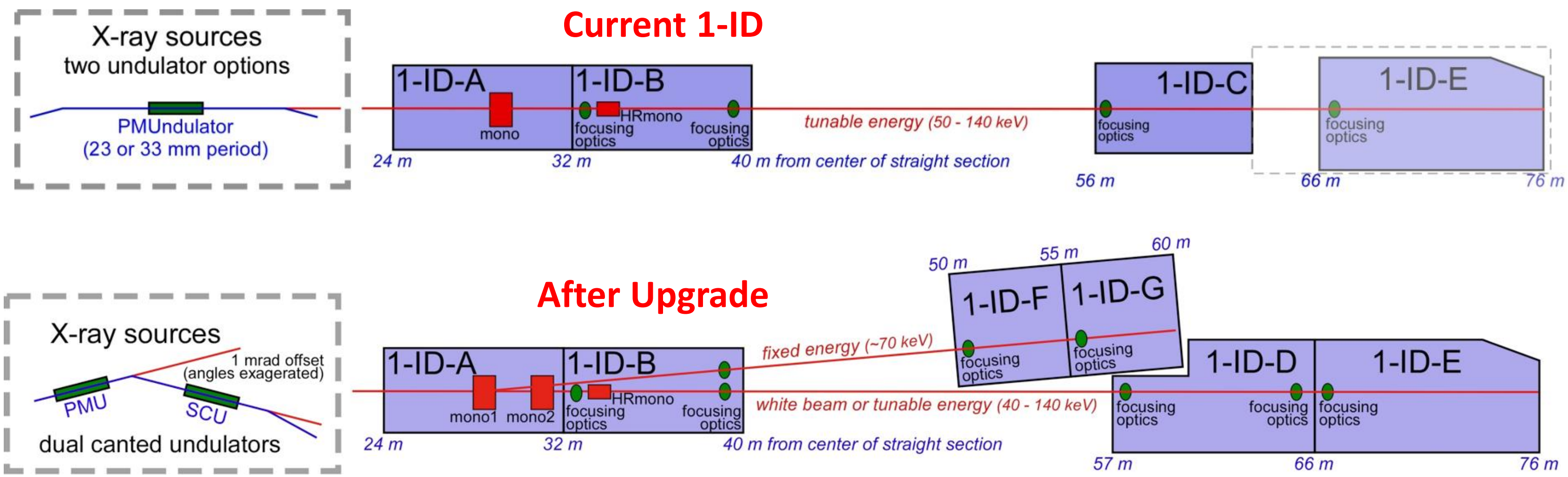
High Energy X-rays: > 50 keV

- Penetrate millimeter dimensions
- Bragg diffraction at small angles
- Large reciprocal space coverage with small detector and one rotation



What the APS does best:
High brilliance at high energies

- Sector 1
  - Dedicated high energy beamline(s)
  - Tailored undulator sources (SCU coming)
  - High resolution area detectors
  - Precision mechanics
  - Data pipeline to Orthros cluster

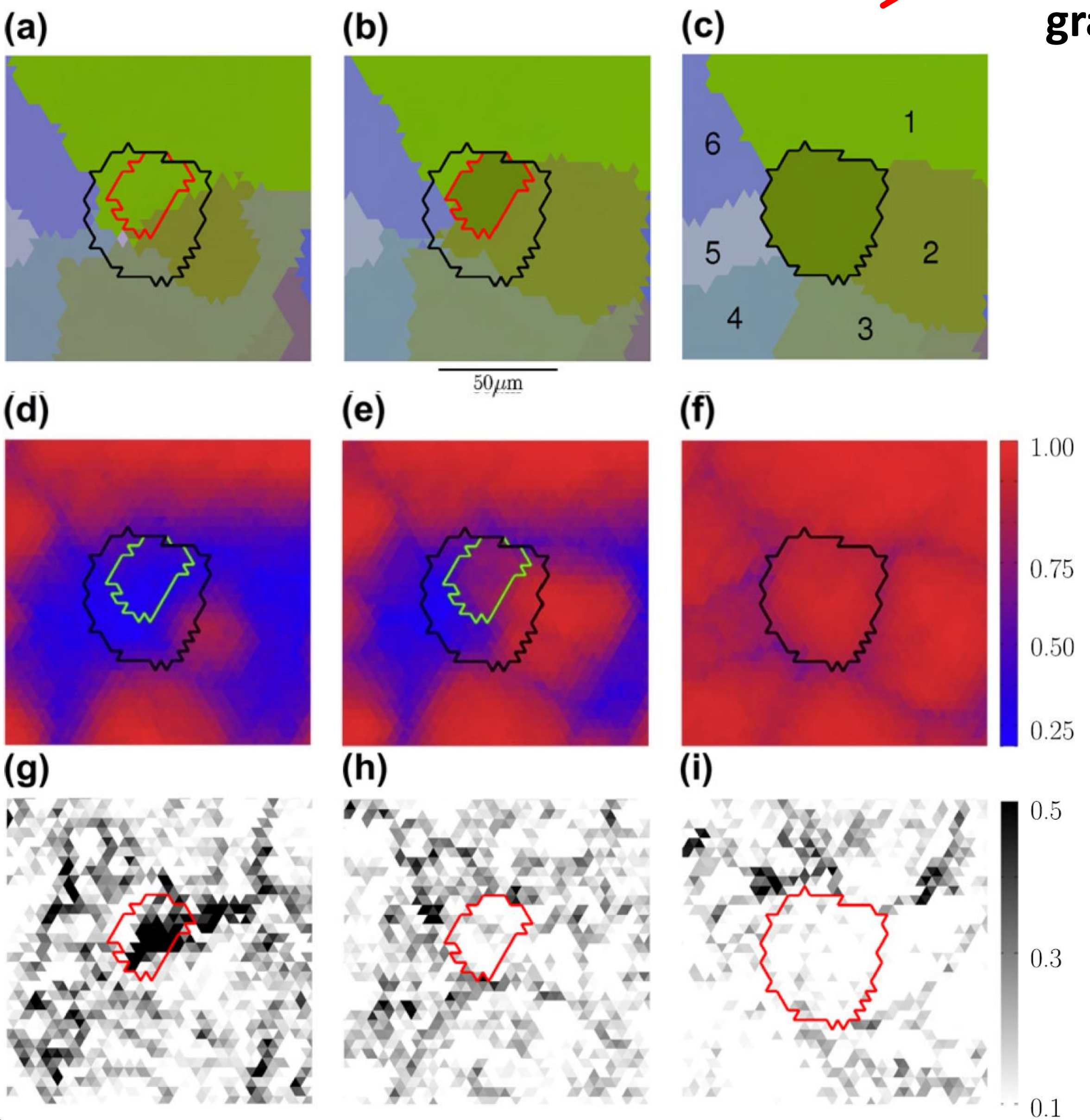


Hefferan et al, Acta Mat 2012

Recrystallization in pure Al

annealing

Voxel-based reconstruction shows new grain and nature of prior neighborhood



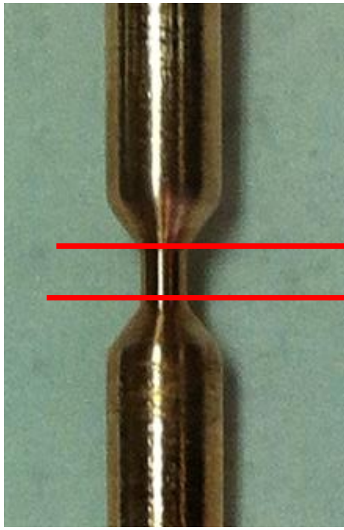
Lattice orientations

Confidence metric

KAM map: 0.5 deg scale

Example 2: Uniaxial Tensile Test on Copper

Onset of plastic response in single layer: fine strain steps

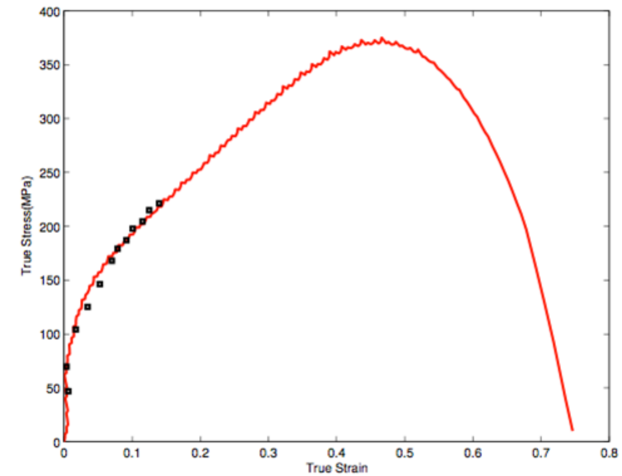


1 mm
gauge section

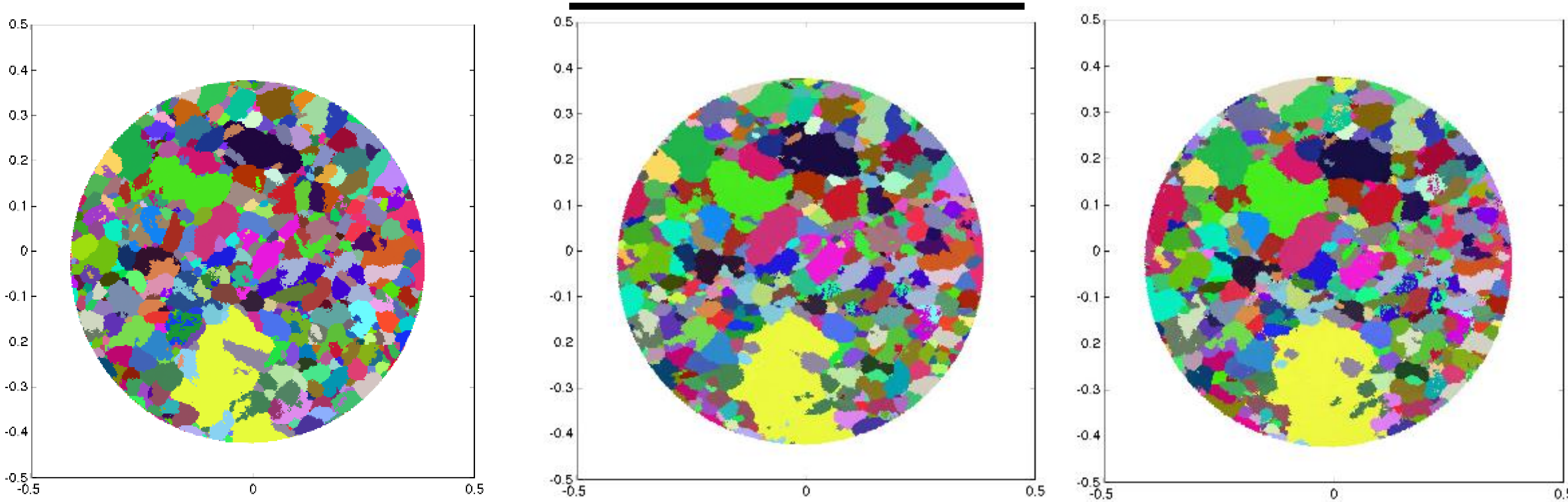
Sample:

Cu: 99.99% pure

Analysis of interior grains only



1 mm



0.06% Strain | **6% Strain** | **14% Strain**

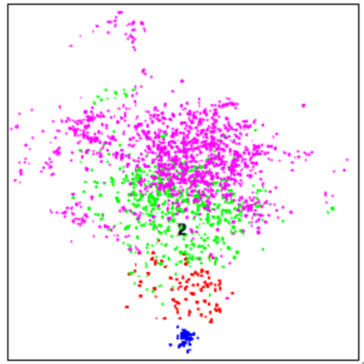
Voxel based tensile axis in crystal coordinates

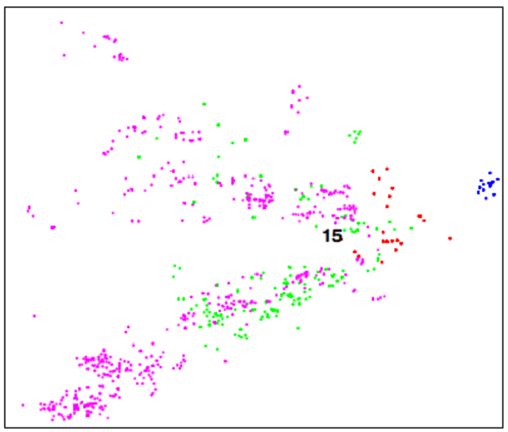
Spatially resolved rotation and breakup

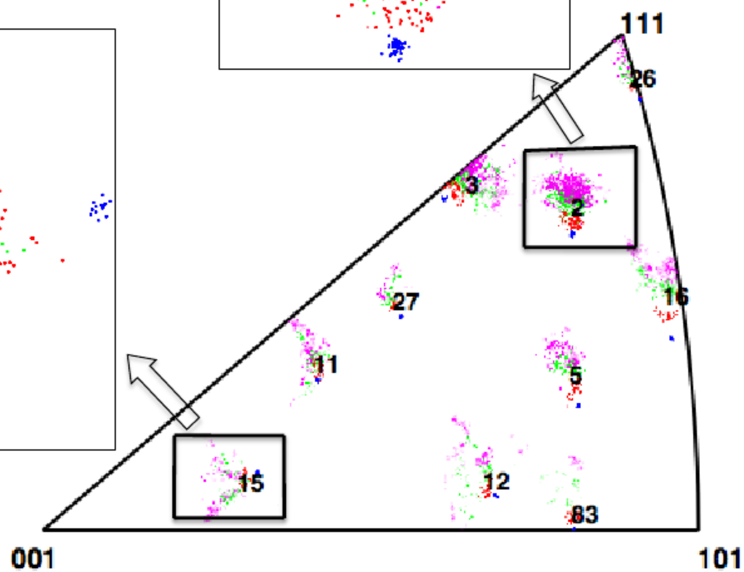
Pokharel, et al, in preparation

Strain levels

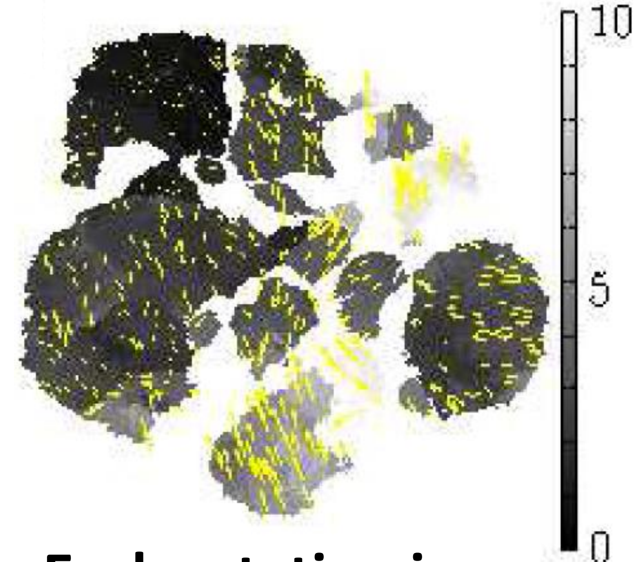
Blue 0%
Red 6%
Green 10%
Purple: 14%







Lattice rotation and bifurcation leading to broadening of scattering



Each rotation is spatially resolved within grain interiors

Li et al, J. Appl. Cryst. (2012)

AFRL-APS-CMU-LLNL-Cornell PUP

- Combine nf-, ff-HEDM, tomography
  - Coupled data collection
  - Coupled data reduction
  - Coupled interpretation
- Design, build, commission multi-technique compatible sample handling/environments

Allocations: Aug 2012, Dec 2012

Slides from J. Schuren presentation to APS SAC March, 2013

The Materials Genome Initiative

Goal: to decrease the time-to-market by over 50%

1. Develop a Materials Innovation Infrastructure
2. Achieve National goals in energy, security, and human welfare with advanced materials
3. Equipping the next generation materials workforce

"The inherently fragmented and multidisciplinary nature of this work demands scientists think of themselves not as an individual researcher, but as part of a powerful network collectively analyzing and using data generated by a larger community."

Argonne National Laboratory; DESY PETRA III; Lawrence Livermore National Laboratory

- Jon Almer
- Peter Kenesei
- Ali Mashayekhi
- Erika Benda
- Kurt Goetze
- Ulrich Lienert
- Joel Bernier
- Frankie Li

Carnegie Mellon University; Cornell University; CHESS

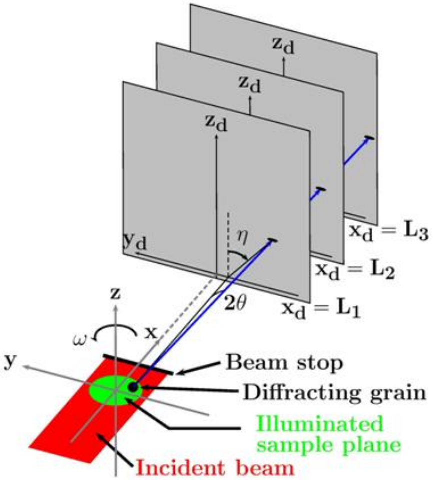
- Robert Suter
- Jon Lind
- Matt Miller
- Donald Boyce
- Sol Gruner
- Ernie Fontes
- Darren Dale

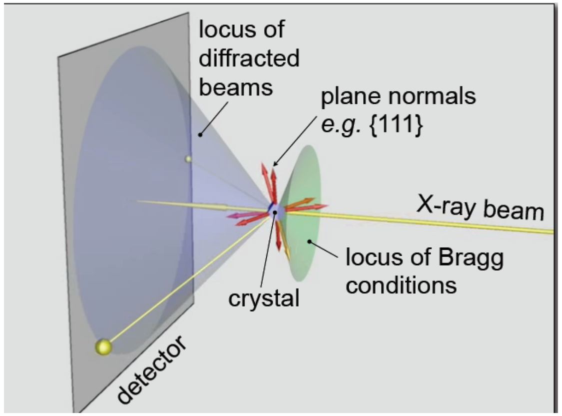
Illinois University of Illinois at Urbana-Champaign; Purdue University; PulseRay; nutonian Scientific Data Mining; Air Force Research Laboratory

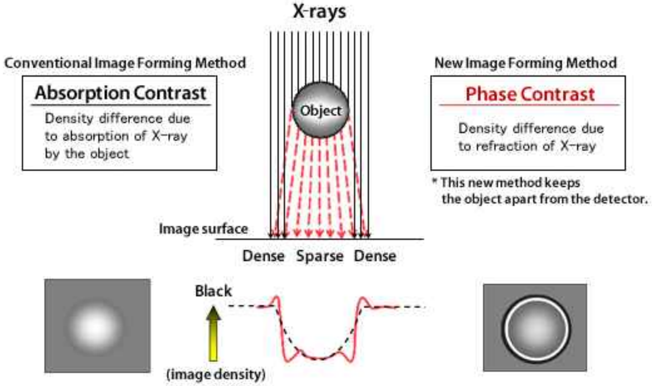
- Armand Beaudoin
- Michael Sangid
- Basil Blank
- Michael Schmidt
- Jay Schuren
- Paul Shade
- TJ Turner

Overview of the Techniques

Near Field Orientation Microscopy	Far Field Lattice Strain Techniques	Absorption Micro-Computed Tomography





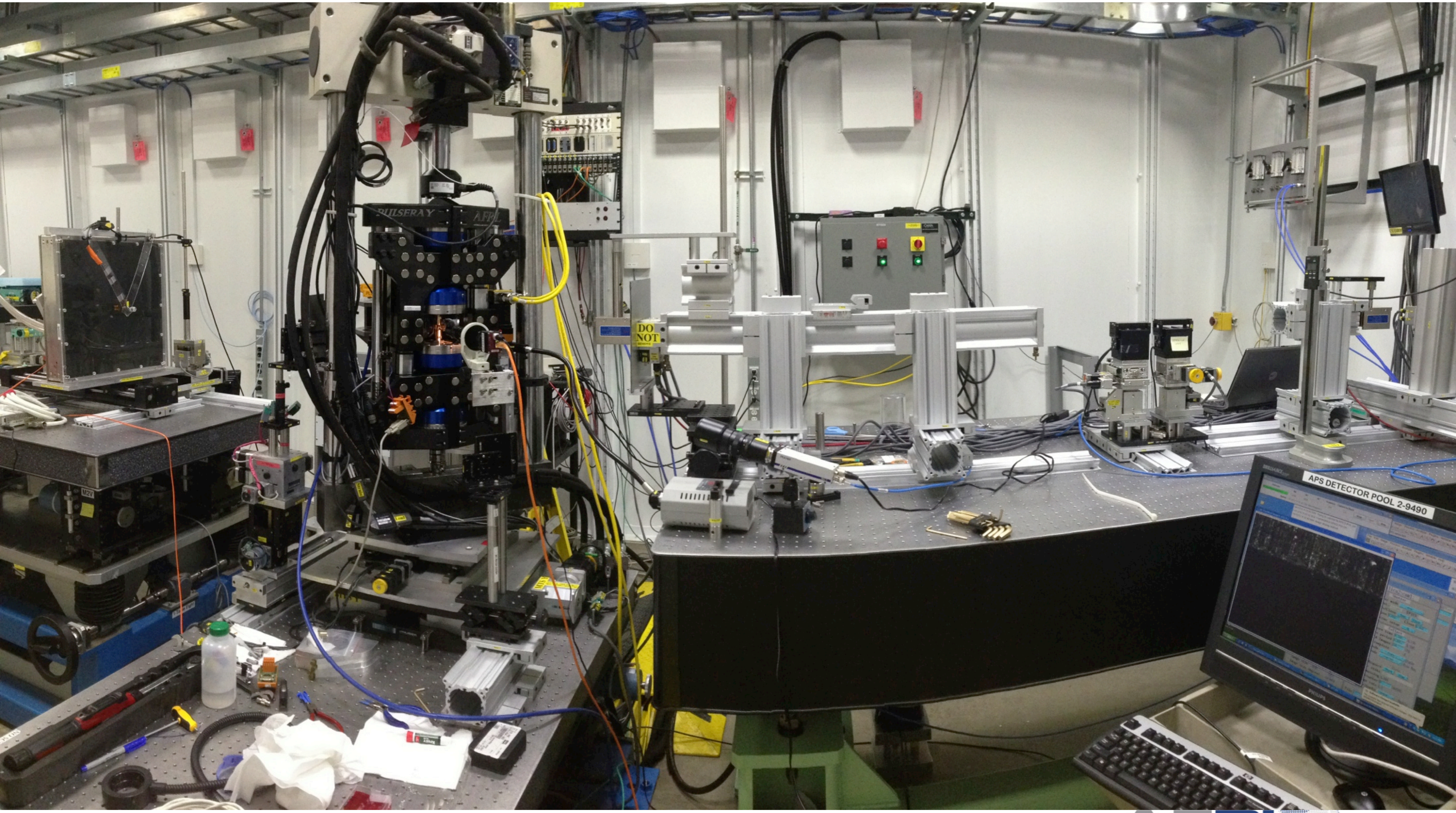


Provides:
grain shapes, subgrain orientation, subgrain strain(?)
X-ray Char.:
Line focused beam (~ 1.5mm x 2um)
Collection:
Take image at N different distances and rotate M times (NxM images). Move sample vertically to build up 3D volume
Processing:
Reconstruct distinct diffraction spots on detector

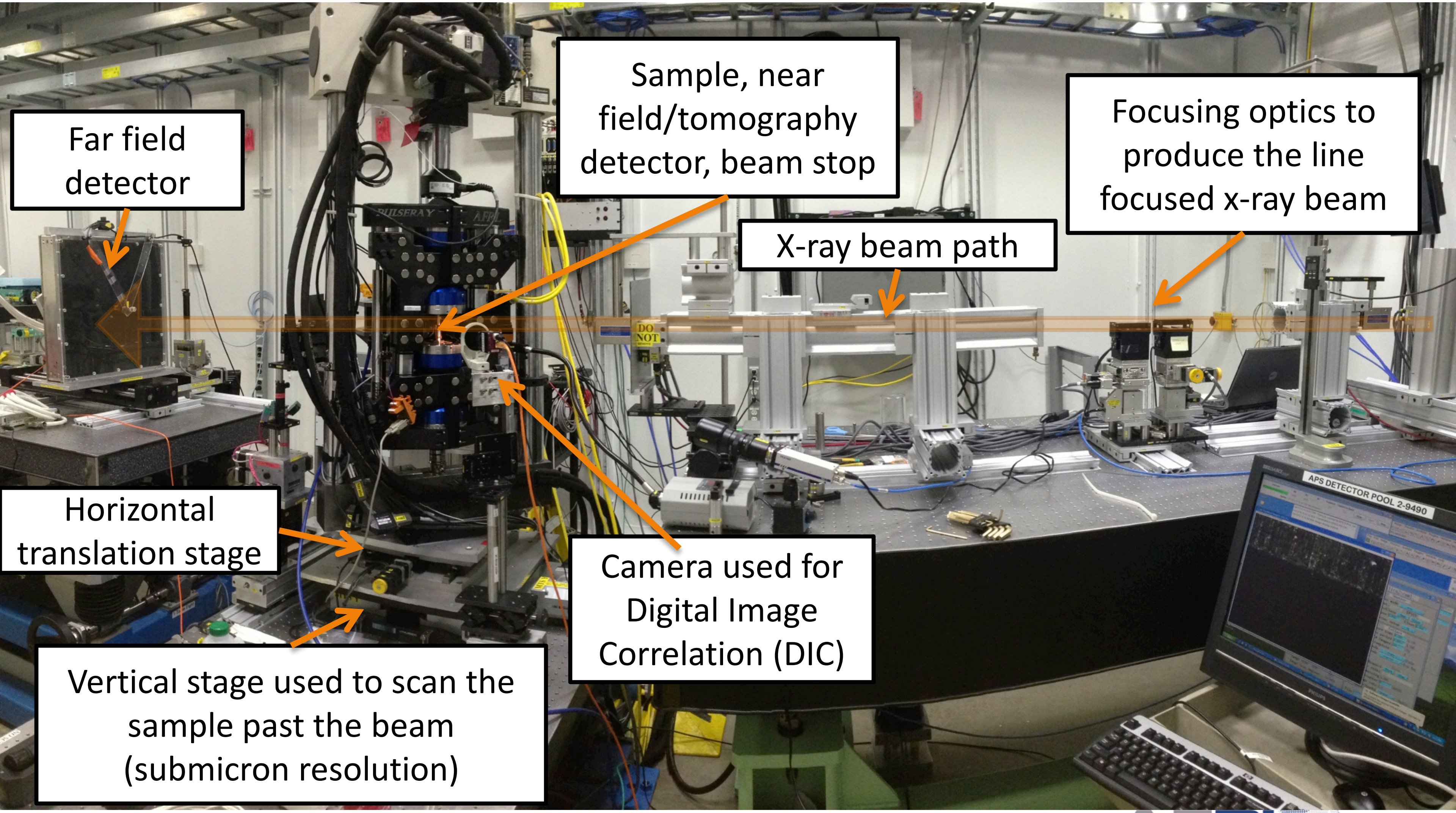
Provides:
grain volume, centroid, orientation, strain for individual grains
X-ray Char.:
Both line focused and box beam
Collection:
Take image during M rotation increments. Move sample vertically to build up 3D volume using line beam
Processing:
Back projection of diffraction spots w/ grain precession

Provides:
position/size of Inclusions, voids, cracks
X-ray Char.:
Both line focused and box beam
Collection:
Take image during M rotation increments. Move sample vertically to build up 3D volume using line beam
Processing:
Back projection of contrast within 2D image (mm^2) of direct beam

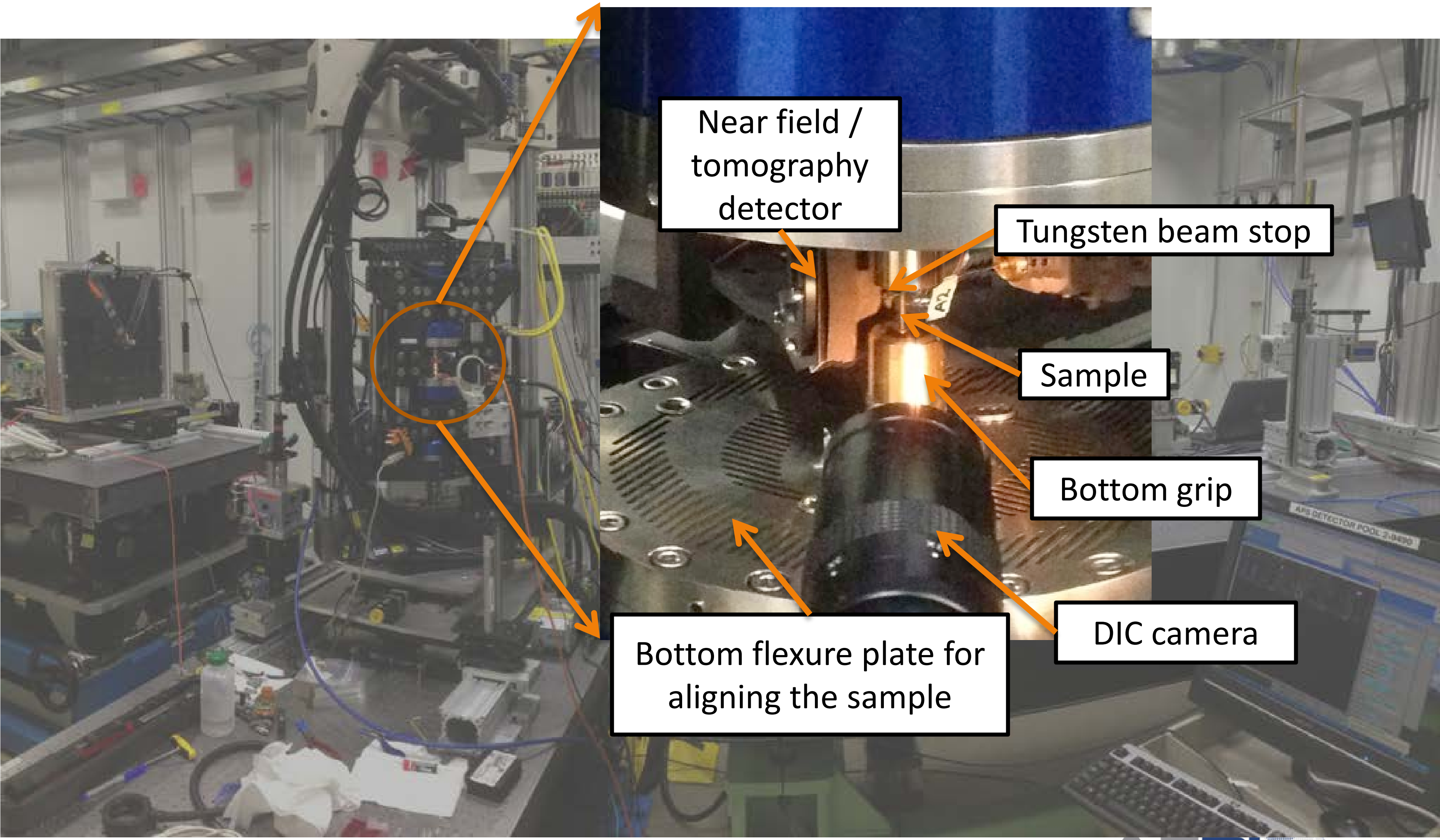
Experimental Setup at APS-1-ID-E



Experimental Setup at APS-1-ID-E



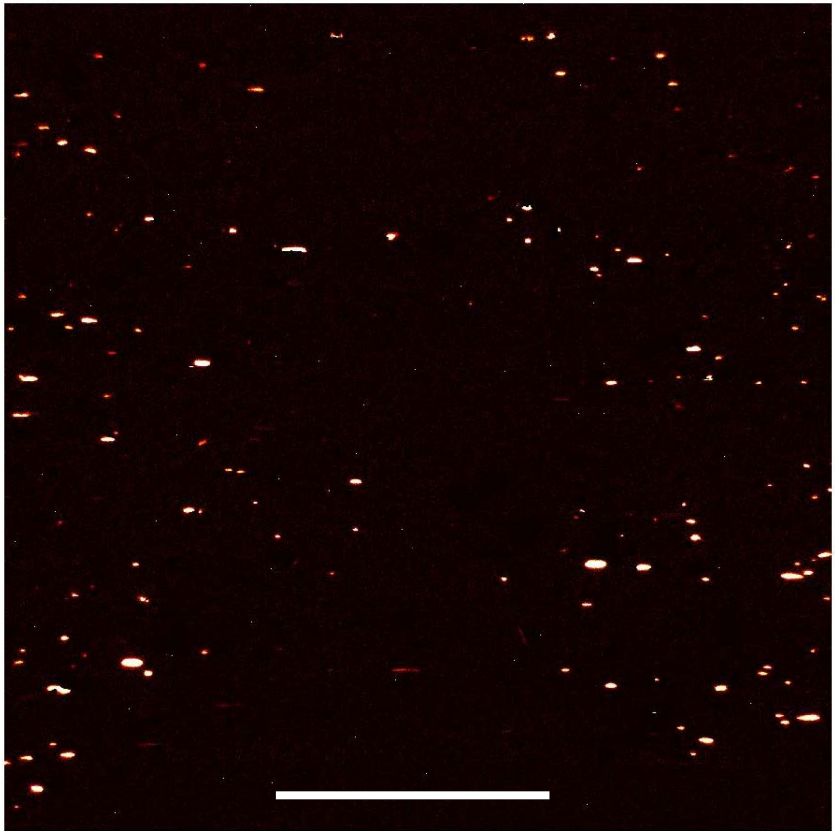
Experimental Setup at APS-1-ID-E



Concurrent Near-, Far-field, and Tomography

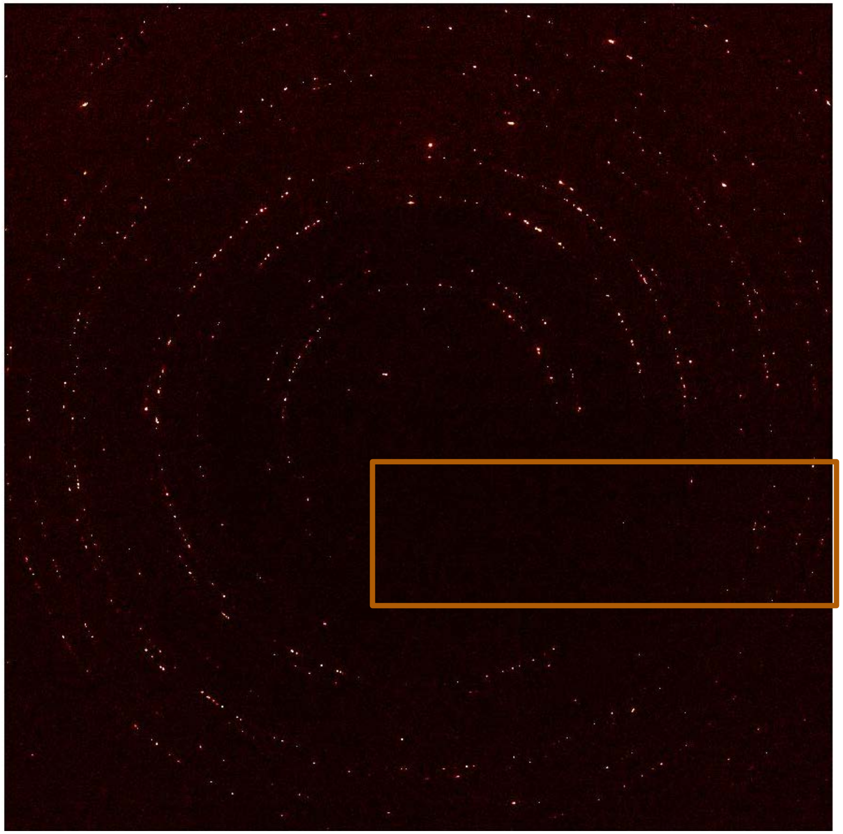
Sample: IN-100 (Ni superalloy)   energy = 51.954 keV

Near-field: D = 15 mm



3 mm

Far-field: D = 650 mm

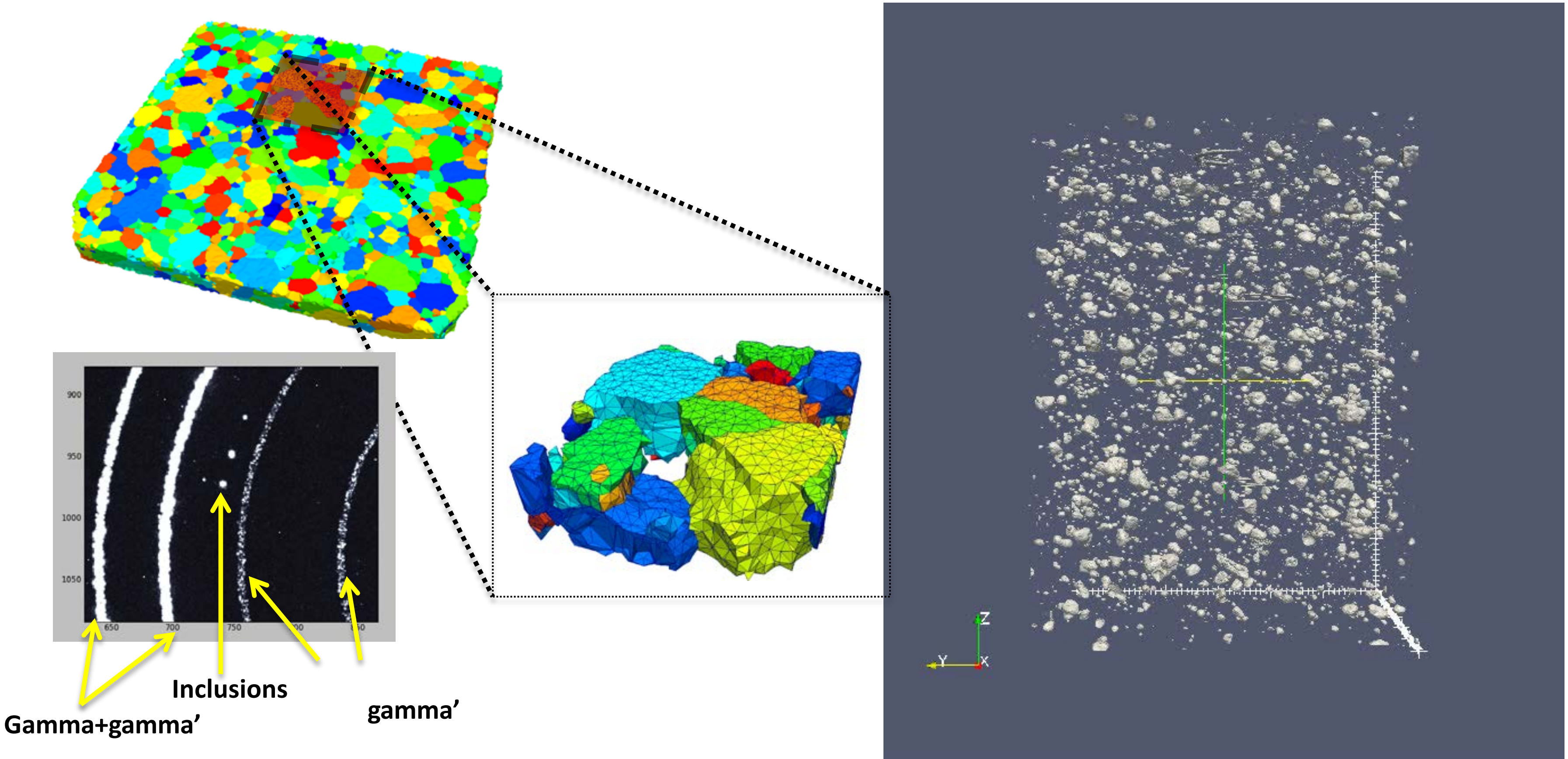


200 mm

Processing Nickel-based superalloys

•Thermally induced porosity

– **Overview:** TIP is thought to occur at grain boundary triple lines – using the full 3D dataset investigate coalescence statistics and the dependence on the local microstructure

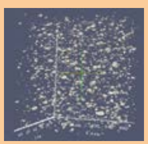


Thank You!

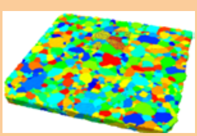
Developing HEDM tools to nondestructively characterize samples at the microstructure length scale far from the free surface during known thermomechanical test conditions

Integrate High Energy X-ray Techniques With Thermo-Mechanical Testing

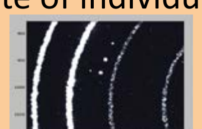
Absorption **Micro-Computed Tomography**
-- Position/size of Inclusions, voids, and cracks



Near Field Orientation Microscopy
-- Subgrain orientation information



Far Field Lattice Strain Techniques
-- Stress state of individual grains



Enable concurrent application to probe deforming materials

Future Research

Initial State

Quantify microstructure and stresses → Initialize model w/ microstructure from HEDM

Evolve sample via thermo-mechanical loading

Simulate the experimental boundary conditions

Deformed State

Quantify microstructure and stresses

Track evolution of both stresses and crystal orientations

Compare both the measured and simulated stresses and crystallographic orientations

Closed-Loop Model Development/Validation

Identify where results disagree, then develop/refine key aspects of model

Provide Validated Model for Component Designs