In-Situ Micromechanics at CHESS

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Cornell High Energy Synchrotron Source

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COMPRES Workshop, 2018

Outline

- Background of CHESS
- CHESS-U
- Overview of structural materials capabilities at CHESS
- Recent Highlights
- Future of structural materials at CHESS

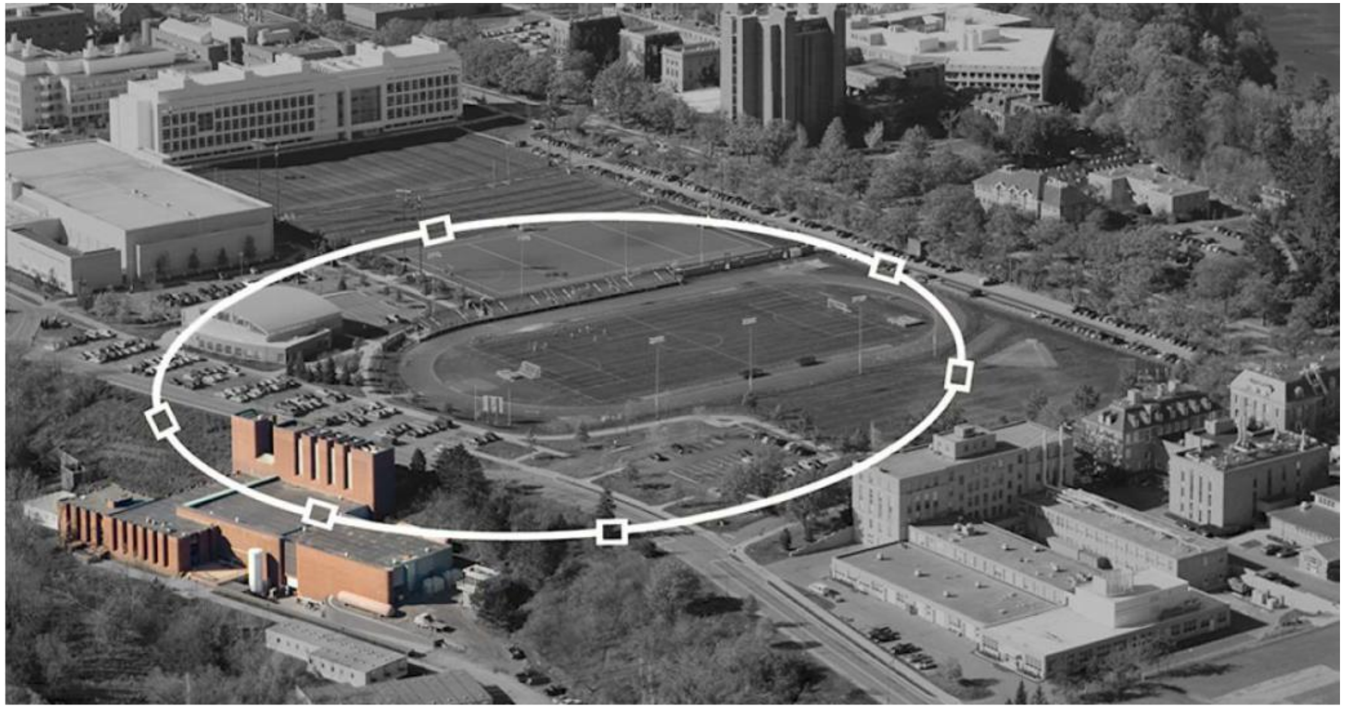
Cornell High Energy Synchrotron Source (CHESS)

Storage Ring:

- 768 m circumference
- 5.3 GeV (1 of 5 in world)
- 120 mA of e- and e+ simultaneously
- 5 stories underground

CHESS:

- 1300 user visits/year
- 11 experimental stations
- 3800 hours/year of user operation
- 2 Nobel Prizes
- $20M/year budget from NSF



CHESS Philosophy

- CHESS is a primarily NSF-funded National User Facility located on a university campus
  - CHESS directly funds 10-20 graduate students and post-docs (co-advised with professors on campus)
- Education of students (graduate and undergraduate) and users is an important part of our mission
- Students get hands-on experience operating (and changing) a beamline for their experiments
- New users are able to develop their experiments (>30% first time synchrotron users) with beamline staff and experienced users are able to develop new cutting-edge experiments

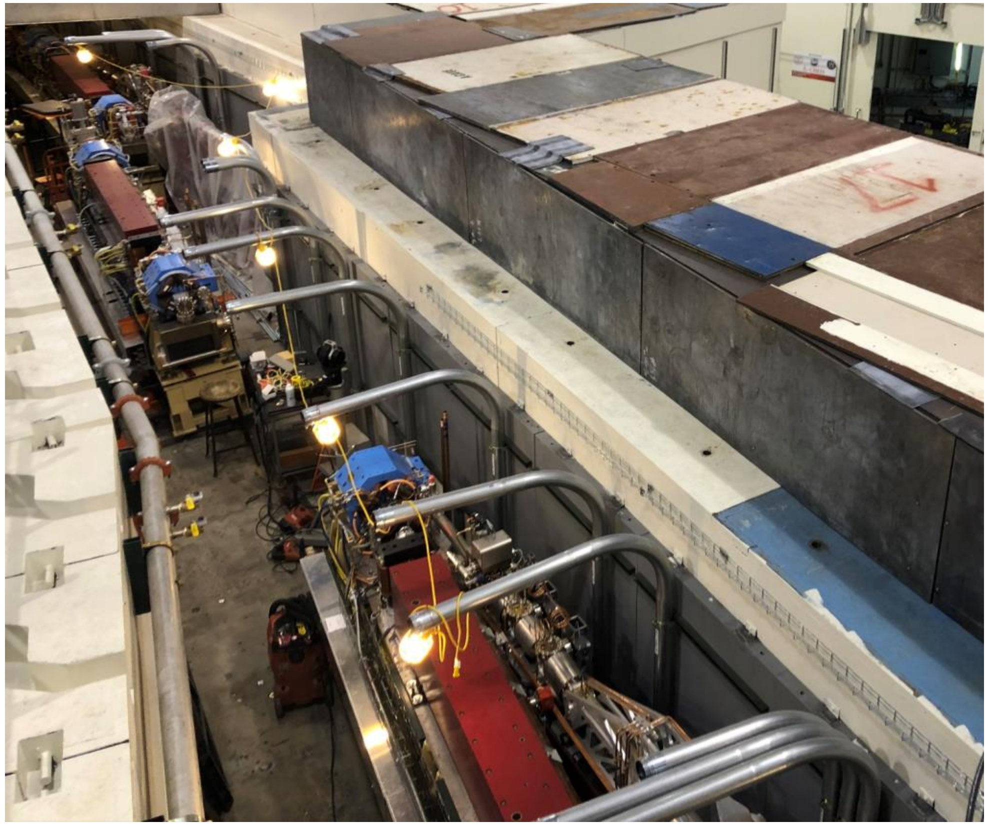
CHESS-U

CHESS is currently undergoing and upgrade funded by the state of New York (CHESS-U)

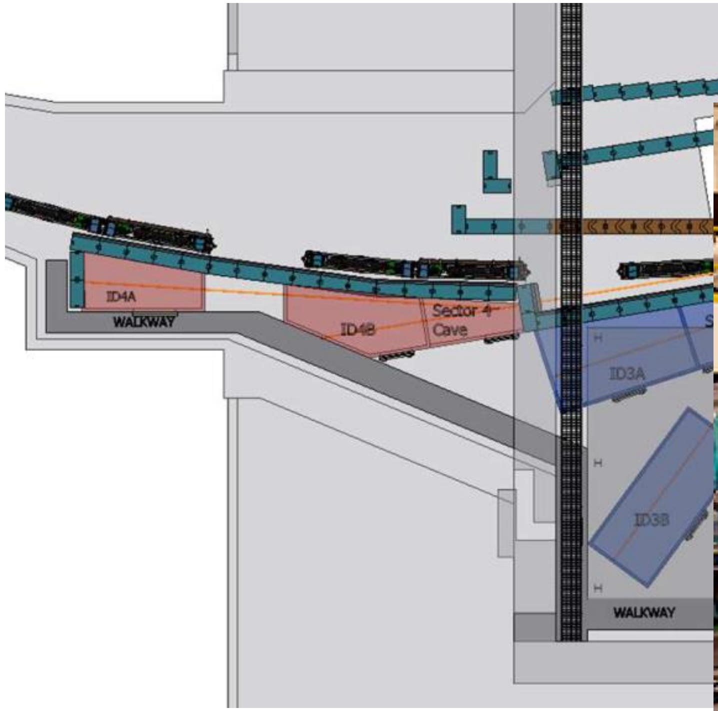
- **Expected completion April, 2019**

As part of the upgrade:

- CHESS will be moving to single beam (e- only) operations
- Replace ~1/6 of the storage ring with new double bend achromat magnets
- Electron beam energy will be increased from 5.3 -> 6.0 GeV
- Electron beam current will be raised from 120mA to 200mA
- All beamlines will be fed by insertion devices (CHESS Compact Undulators)
- 7 new (larger!) X-ray experimental stations

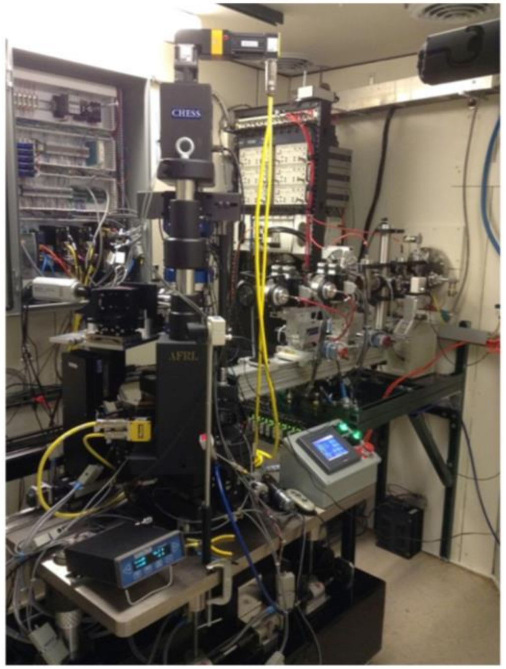


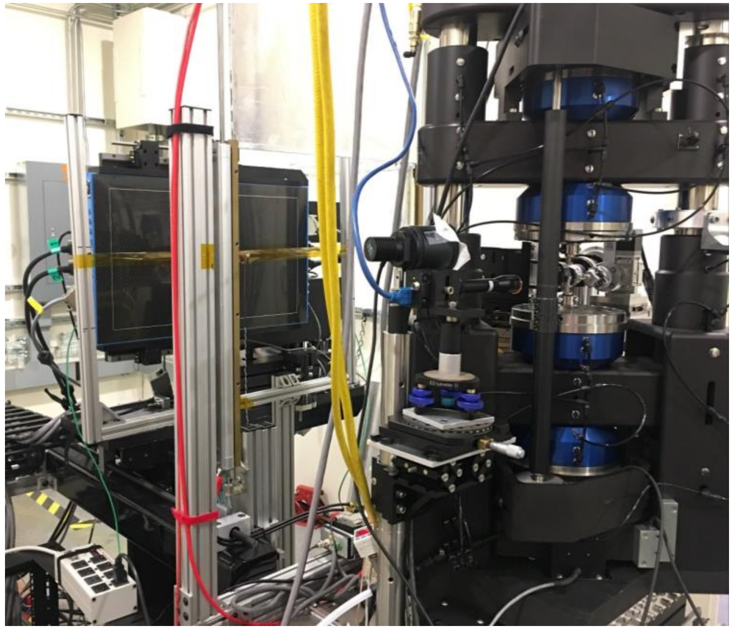
CHESS-U New Hutches



Structural Materials at CHESS

- The structural materials program at CHESS was operated out of the F2 station
- Focus was placed on high-energy (>30keV) **diffraction** techniques to study microstructure micromechanical response
  - Complimentary absorption tomography was available
- With CHESS-U, all structural materials capabilities presented today will still be available and primarily transferred to the new Forming and Shaping Technology beamline





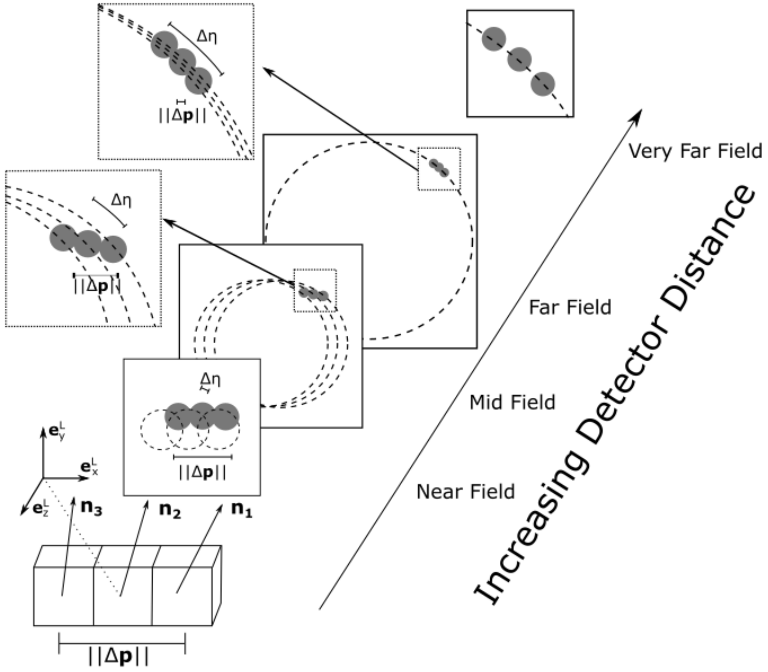
Techniques 1

Far-field high energy diffraction microscopy, ff-HEDM (monochromatic)

- Measure grain average strain tensors, center of masses, and crystal lattice orientations
- Works best with (1) grain diameters varying from 10 µm to 500 µm, (2) relatively uniform size distribution also helps, (3) low to medium dislocation content
- Uses large panel area detectors sitting between 0.5 and 1.5 mm from the sample (2 x Dexela 2923 or 1 x GE 41RT+)
- Single volume scan times are ~5 minutes
- Minimum ~100° sample rotation necessary, more angular range the better
- Needs Distinct Peaks!

Near-field high-energy diffraction microscopy, nf-HEDM (monochromatic)

- Measure grain morphology and distributions of orientation within grains (~2um resolution)
- Works best with (1) grain diameters above 10 µm, (2) low to medium dislocation content
- Uses scintillator optical camera pairing sitting **5 mm to 15 mm** from the sample (LuAG:Ce)
- Box beam algorithm used can probe volumes up to 150 µm tall
- Single volume scan times are ~2hrs, entire samples take 8-10 hrs
- The reconstruction algorithm requires ff-HEDM data to seed the grain reconstruction



Near field measurements are more sensitive to positions of diffracting volumes, while far-field measurements are more sensitive to orientation and strain state of diffracting volumes

*All monochromatic techniques can be performed from 30-80 keV

Techniques 2

Transmission Powder Diffraction (monochromatic, far-field)

- Measure pole figures (texture), strain pole figures (fiber orientation averaged strains), pair distribution functions, 2D distributions of strain/stress
- Works best with fine grained materials and does not have any defect content restrictions
- Single exposures take seconds, while full pole figures can be measured in minutes
- Uses large panel area detectors sitting between 0.5 and 1.5 mm (2 x Dexela 2923 or 1 x GE 41RT)

Energy Dispersive Diffraction (polychromatic)

- Measure spatially resolved distributions of strain / texture in large volumes (cm)
- Works best with fine grained materials, but heavily textured materials can be problematic
- X-rays range from 50-200 keV
- Can penetrate through bulky sample environments
- Uses germanium single crystal sensor with XMAP signal processor for energy determination

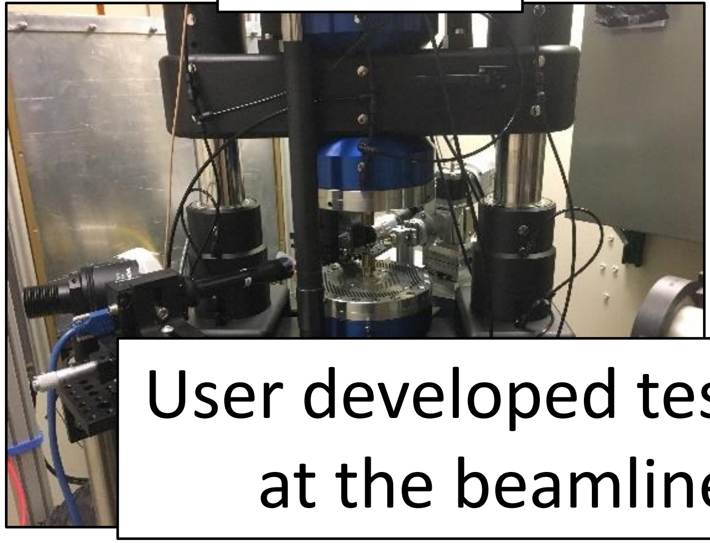
Absorption Computed Tomography (monochromatic)

- Measure variation of porosity/cracks or phase compositions
- Works best with materials whose phase compositions have large variation in density/ atomic number or known voids
- Uses scintillator optical camera pairing sitting 5mm to 100 mm (LuAG:Ce)

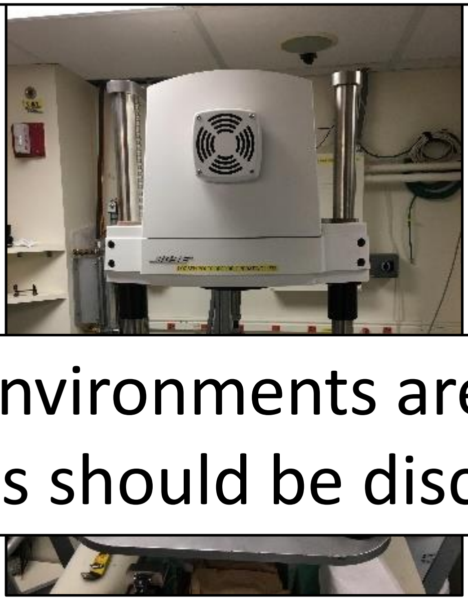
*All monochromatic techniques can be performed from 30-80 keV

Testing Environments

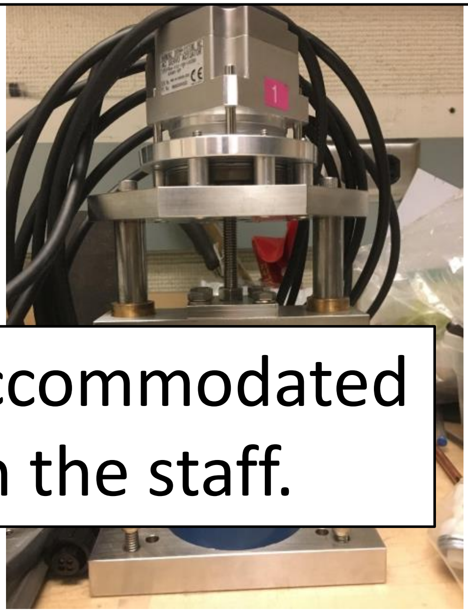
RAMS2



Electro Magnetic Load Frame



Compact Load Frame



User developed testing environments are readily accommodated at the beamline. Plans should be discussed with the staff.

RAMS2

- Full reversed cyclic loading (2000N T and 1000N C)
- Furnace Available (1000C Max)
- Demanding specimen design
- Compatible Techniques:
    - Far-field HEDM
    - Near-field HEDM
    - Powder Diffraction
    - Absorption Tomography

Electro Magnetic Load Frame

- Full reversed cyclic loading (2500N)
- High Frequency (100 Hz Max)
- Flexible specimen design
- Compatible Techniques:
    - Far-field HEDM
    - Powder Diffraction
    - Absorption Tomography

Compact Load Frame

- Full reversed cyclic loading (~2000N)
- Pressure Cell Coming Soon (100MPa Max)
- Flexible specimen design
- Compatible Techniques:
    - Far-field HEDM
    - Powder Diffraction
    - Absorption Tomography
    - Energy Dispersive Diffraction

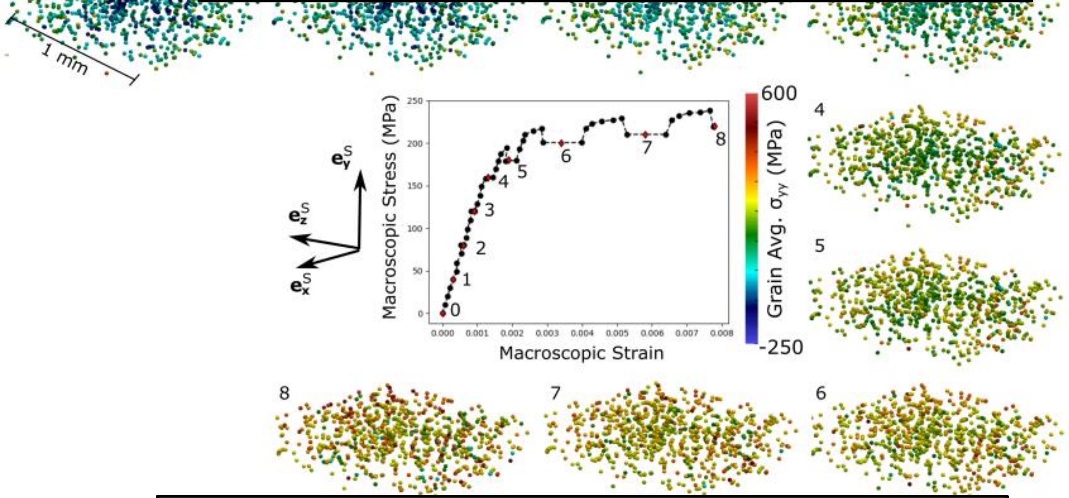
Highlight Reel

- Overview
- Techniques / equipment we used
- Why we used a given technique / equipment
- What we measured
- What we learned

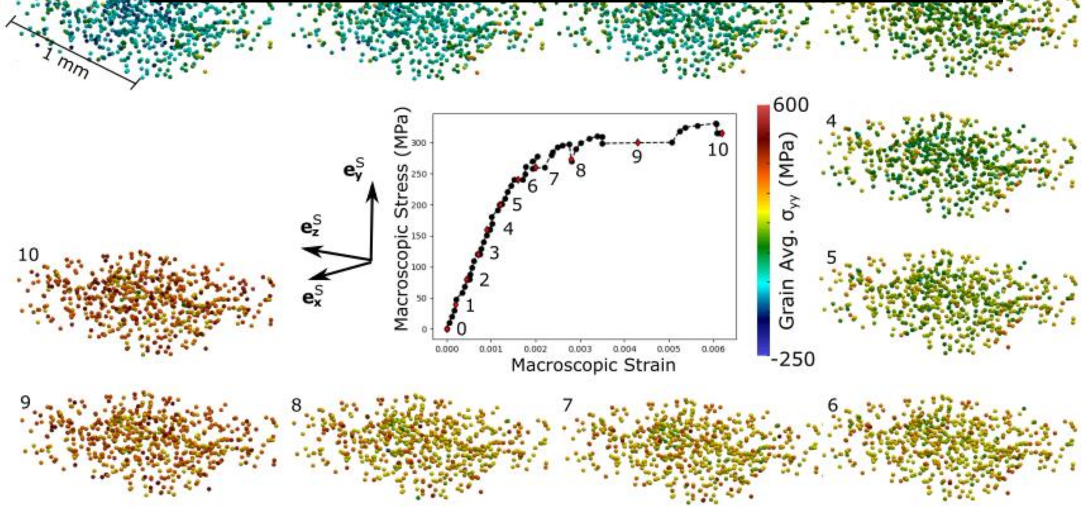
Far-field High Energy Diffraction Microstructural Response Comparisons

- Micromechanical responses of wrought and powder metallurgy hot-isostatic pressed (PM-HIP) 316L Stainless Steel samples were tracked during uniaxial tension
- ff-HEDM / RAMS2
- Grains were relatively large ~100 µm and the defect content was fairly low due to heat treatments
- Full grain average strain and stress tensors were measured from hundreds of grains at each load step
- The traditionally wrought 316L was found to have a wider distribution of stresses at each load step, prevalence of higher stressed grains may lead to more prevalent failure nucleation

316L Wrought, Grain Average σ_{yy} (Loading)

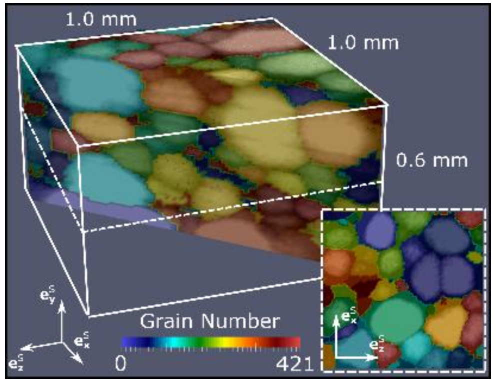


316L PM-HIP , Grain Average σ_{yy} (Loading)



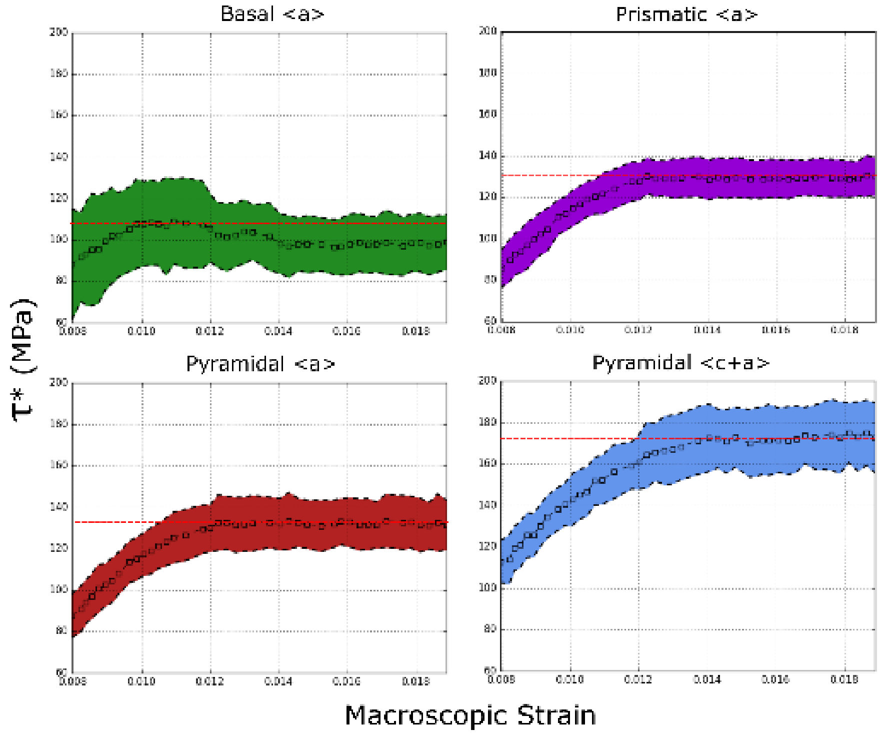
Guillen, Wharry, et.al., Mat. Sci. Eng. A., *Accepted*

Far-field High Energy Diffraction Microscopy at Elevated Temperatures



- The average slip system strengths of different slip systems in hexagonal Ti-7Al were probed during a uniaxial tension test at elevated temperature
- ff-HEDM / RAMS2 with furnace
- Grains were relatively large ~100 um and the defect content was fairly low due to heat treatments
- Full grain average strain and stress tensors were measured from hundreds of grains at each load steps, stresses were resolved onto slip systems and the distributions analyzed
- Strong interactions between dislocations and precipitates leading to softening/localization observed at room temperature were significantly reduced as temperature was increased

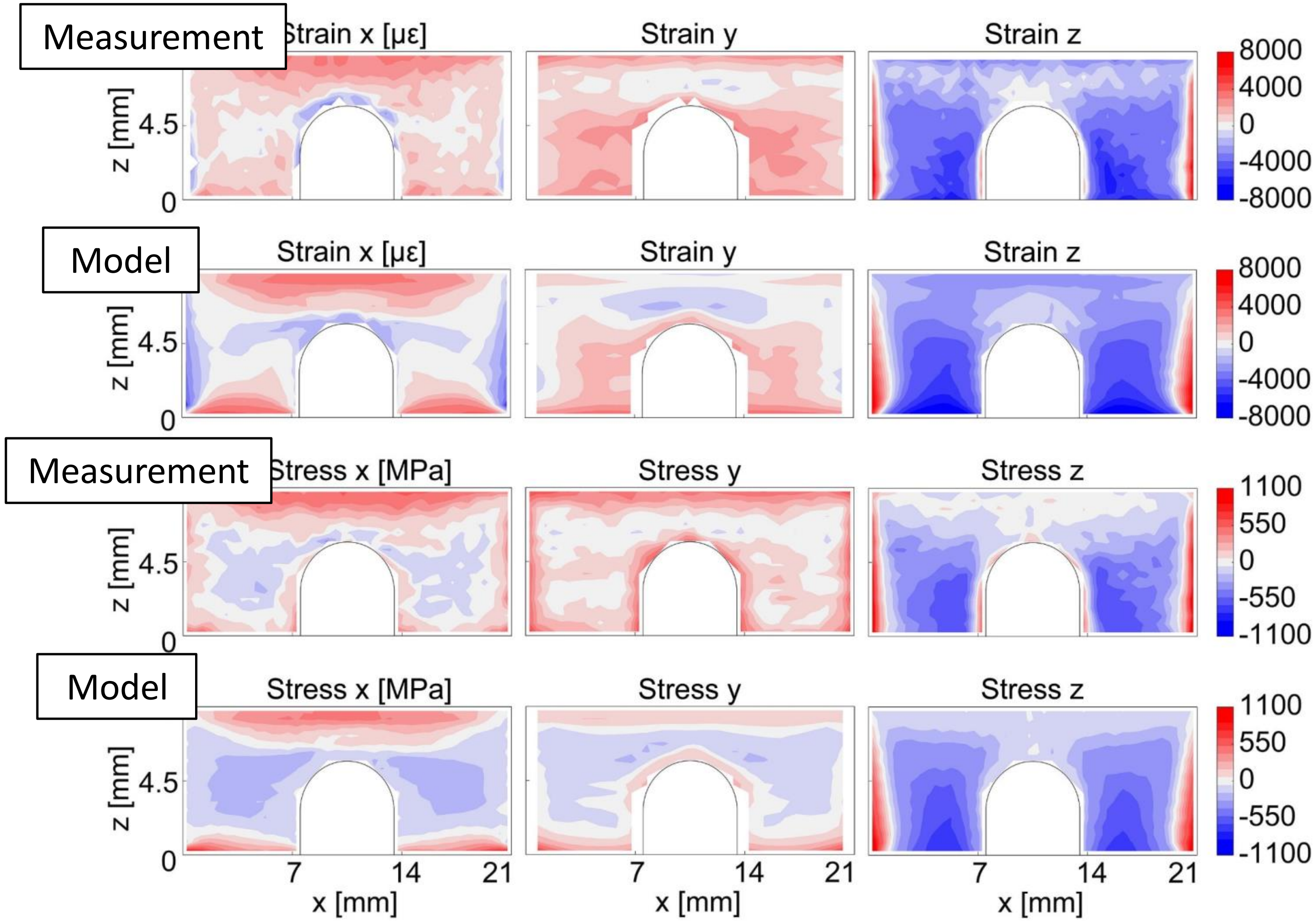
Ti-7Al Slip System Strengths 355°C



Pagan, Bernier, Shade, et al, Scripta Materialia, (2018)

Using Energy Dispersive Diffraction to Measure 3-D Stress Fields

- Residual strain and stress fields were measured in additively manufactured Ti-6Al-4V specimens (ex-situ) and compared to a full thermomechanical model of the build process
- Energy dispersive diffraction
- Samples were relatively large so high-energy EDD was able to penetrate through the sample and isolate diffraction volumes
- 3D fields of three perpendicular strain components were measured and then used to calculated normal stresses
- The X-ray data provided a valuable validation of the thermomechanical model (Diablo)



Strantza, et. al, Materials Letters, (2018)

Moving Forward - FAST

PROPOSED

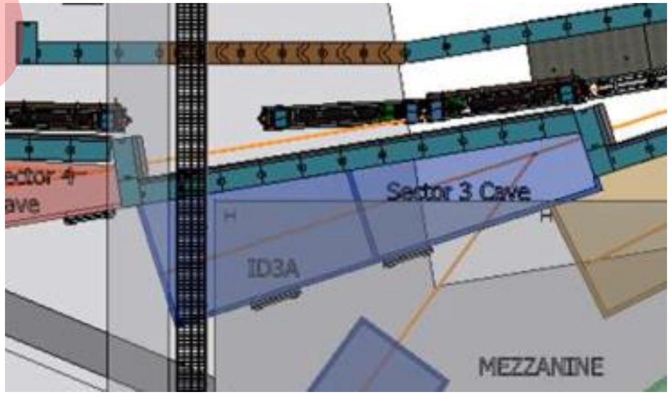
Beginning in April 2019, structural materials will transition to the Forming and Shaping Technology (FAST) beamline located in the new ID3A hutch.

All described monochromatic techniques will be available, but there will be a new focus on deploying optics and detectors for studying rapid processes (**phase transformations and texture evolution! strain?**) using high-energy diffraction

- Insertion device Cornell Compact Undulator

- "Increased Resolution Mode": Double Bounce Cryo Cooled Silicon (30-70keV)
    - ($\frac{\Delta \mathbf{E}}{\mathbf{E}} < \mathbf{10^{-4}}$,**~3e11ph/s** @ 60keV, 1 mm x 1 mm)
    - Grain-by-grain strain measurements, 3-D orientation mapping, micro computed tomography, powder diffraction (orientation and strain pole figures), pair distribution function measurements
- "Wide-Bandwidth Mode": Cryogenically Cooled Multilayer W:CB4 Monochromator (30-60keV) **ETA 2020**
    - ($\frac{\Delta \mathbf{E}}{\mathbf{E}} = \mathbf{3 \times 10^{-2}}$, **~1e13ph/s** @ 60keV , 1 mm x 1 mm)
    - High-speed (µs-ms scale) X-ray diffraction measurements for quantification of phase fractions, temperature, strain, and texture and complimentary radiography
- Max. Beam Size: ~4mm (H) x ~1mm (V)

- Along, with the beamline upgrade, new computation resources (computing cluster) will be available to users to help process data and run simulations (**Explicit Crystal Plasticity FEM**)



Thanks for Listening!