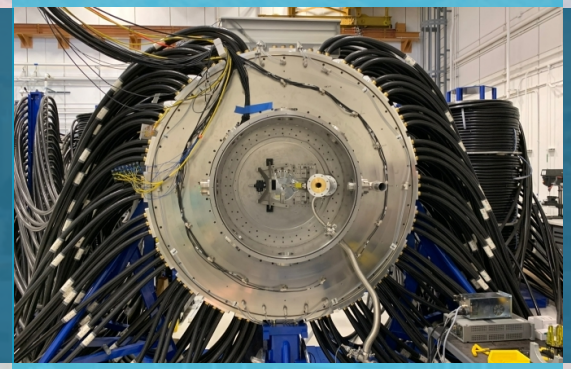
SAND2022-8941C

Sandia National Laboratories

X-ray diffraction of material under ramp compression on the Thor pulsed-power generator



PRESENTED BY
Tommy Ao

APS Shock Compression of Condensed Matter 2022
Anaheim, CA
July 10-15, 2022

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LABORATORY DIRECTED RESEARCH & DEVELOPMENT

Outline

Background

- Basics of x-ray diffraction (XRD) and dynamic compression
- Review of Thor components and operation

X-ray diffraction on Thor

- Hardware design and implementation
- Dynamic XRD results

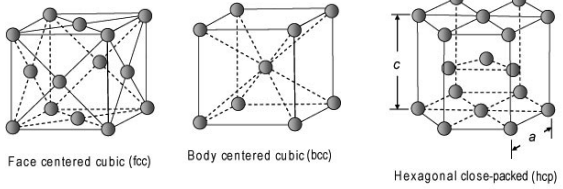
On-going developments

- Technical improvements
- Upcoming Thor-XRD experiments

Crystallography of solids & x-ray diffraction principles

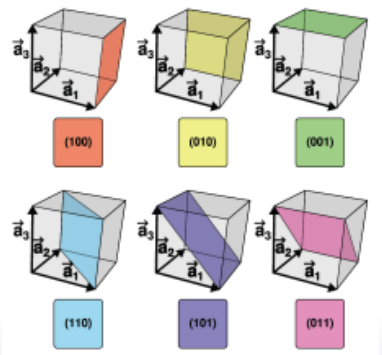
Solids with crystalline structure

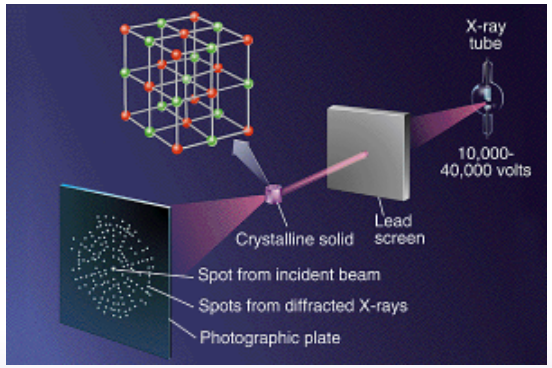
- Formed by atoms, molecules, or ions stacking in 3-dimensional space with a regular & repeating arrangement



- Lattice directions and planes
  - Miller indices are the reciprocal intercepts of the plane of unit cell axes

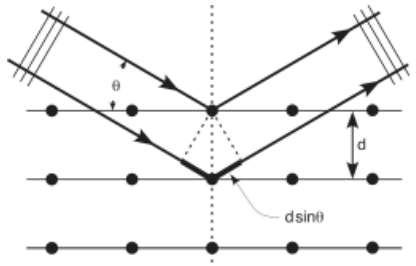
Miller indices in cubic crystals





X-ray crystallography

- Beam of x-rays strikes a crystal & causes beam to spread into many specific directions
- From angles and intensities of diffracted beams, 3-dimensional picture of the crystal is obtained



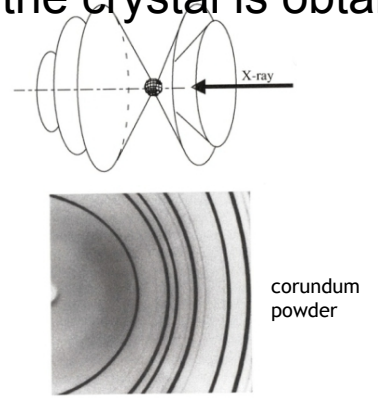
Bragg's Law:

Coherent reflections will occur for wavelength and crystal *d*-spacings that satisfy the condition:

$$n\lambda = 2dsin\theta$$

The scattering angle (half-angle of the Debye-Scherrer cone) is:

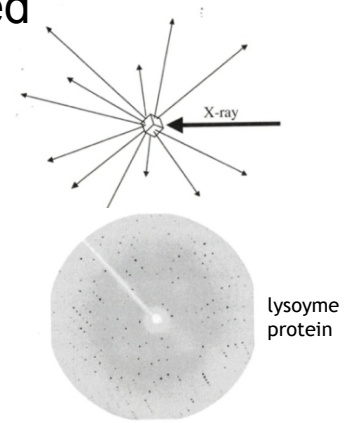
$$\varphi = 2\theta$$



Debye-Scherrer Cones:

Illuminate a polycrystalline sample with a **monochromatic** x-ray beam, scattering patterns called **Debye-Scherrer cones** are observed.

A huge number of crystals are randomly oriented, hence there is a large finite number of them with the correct orientation required



Laue Spots:

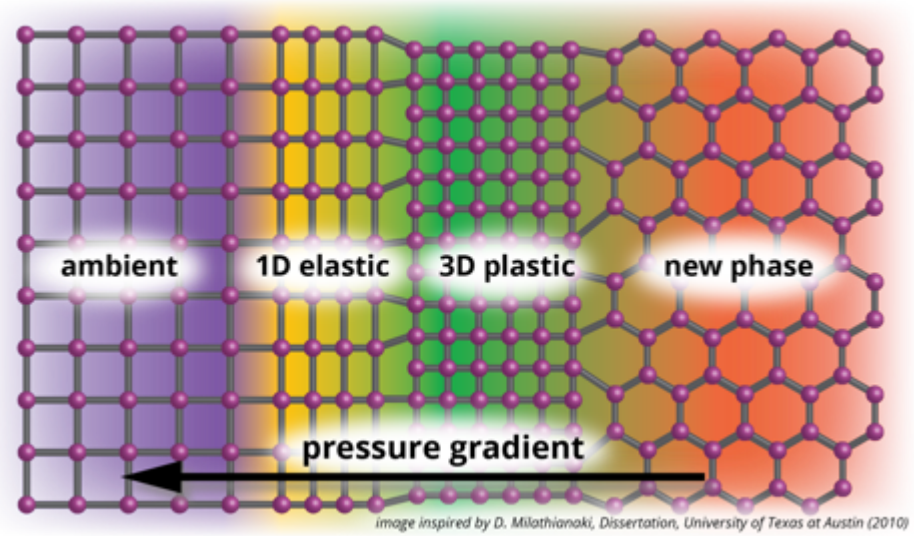
Illuminate a single crystal sample with a **continuum** x-ray beam, discrete reflections called **Laue spots** are observed.

For each set of reflecting Bragg planes in the sample, the wavelength required to meet Bragg's Law will exist because of the continuous nature of the x-ray source spectrum

X-ray diffraction (XRD) to infer dynamic material properties

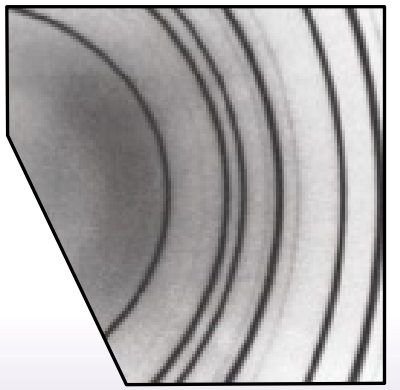
Goals

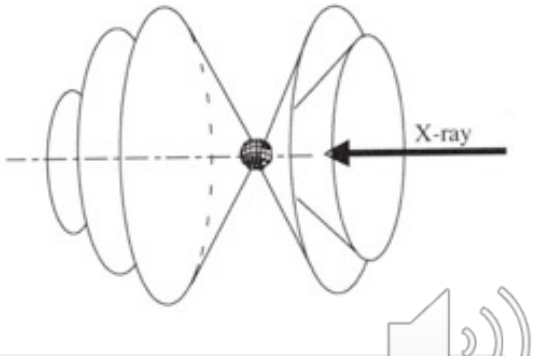
- Characterize both phase transitions and their kinetics that occur in dynamically compressed condensed matter on ns time scales and nm spatial scales
- Determine at atomic scale how materials behave under extreme pressure & temperature conditions; velocimetry gives only continuum scale information



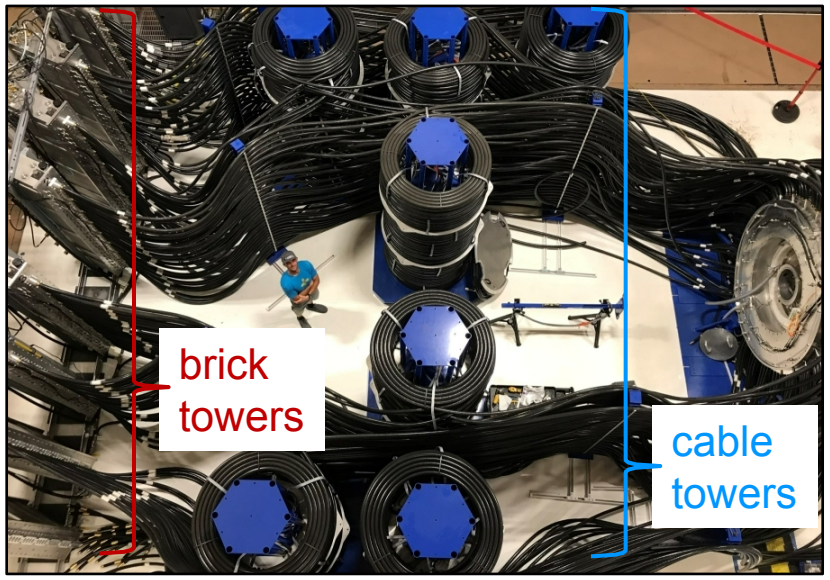
Approach

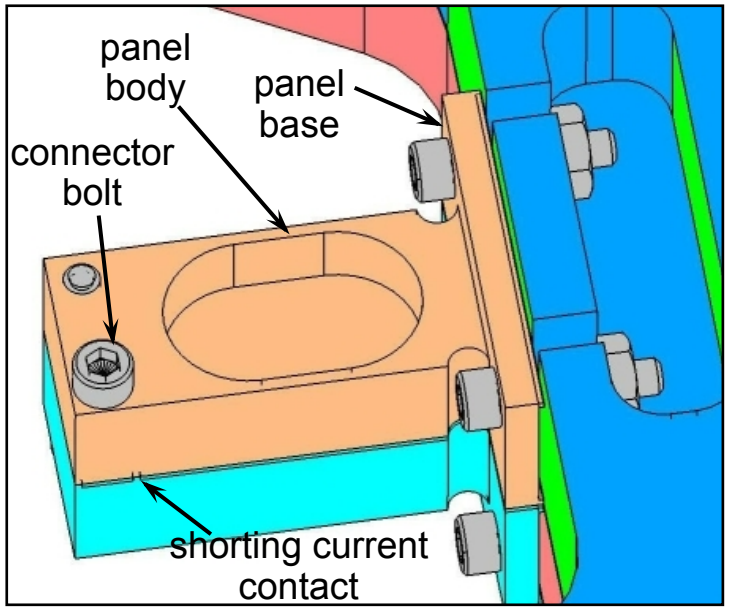
- Produce source x-rays with flash x-ray diode
- Generate high-pressure state with Thor pulsed-power driver
- Detect diffracted x-ray pattern with image plate

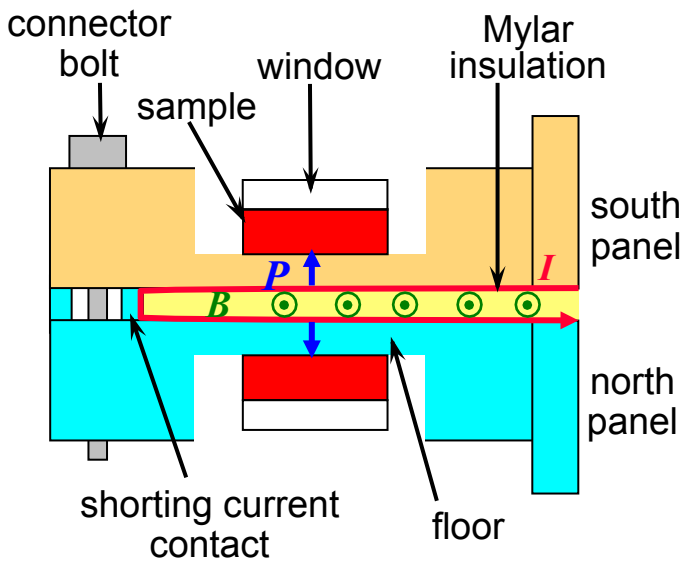


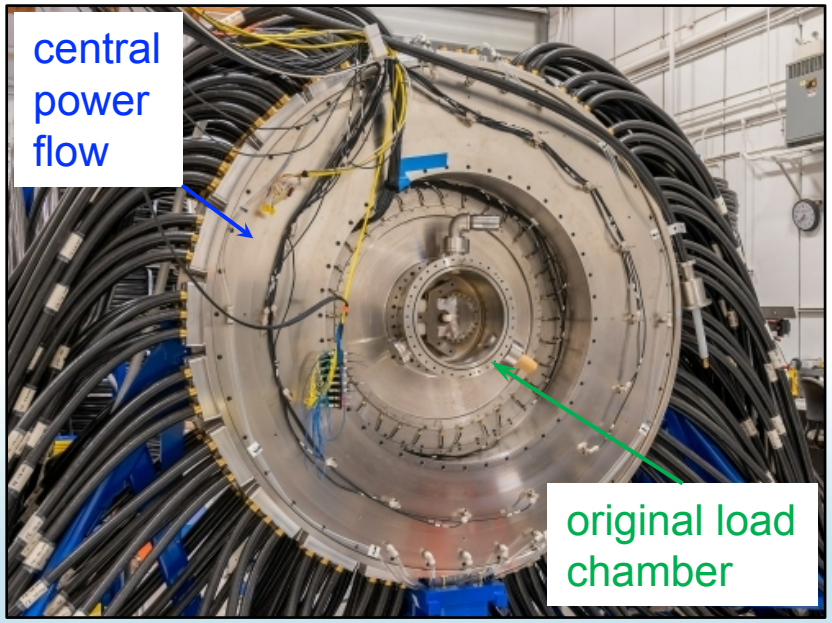


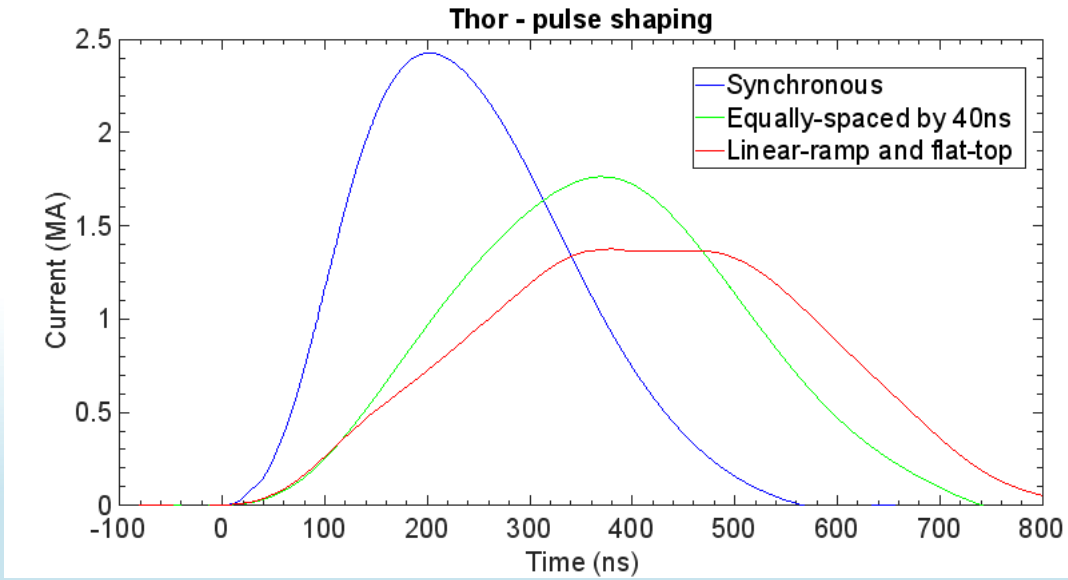
Review of Thor components and operation





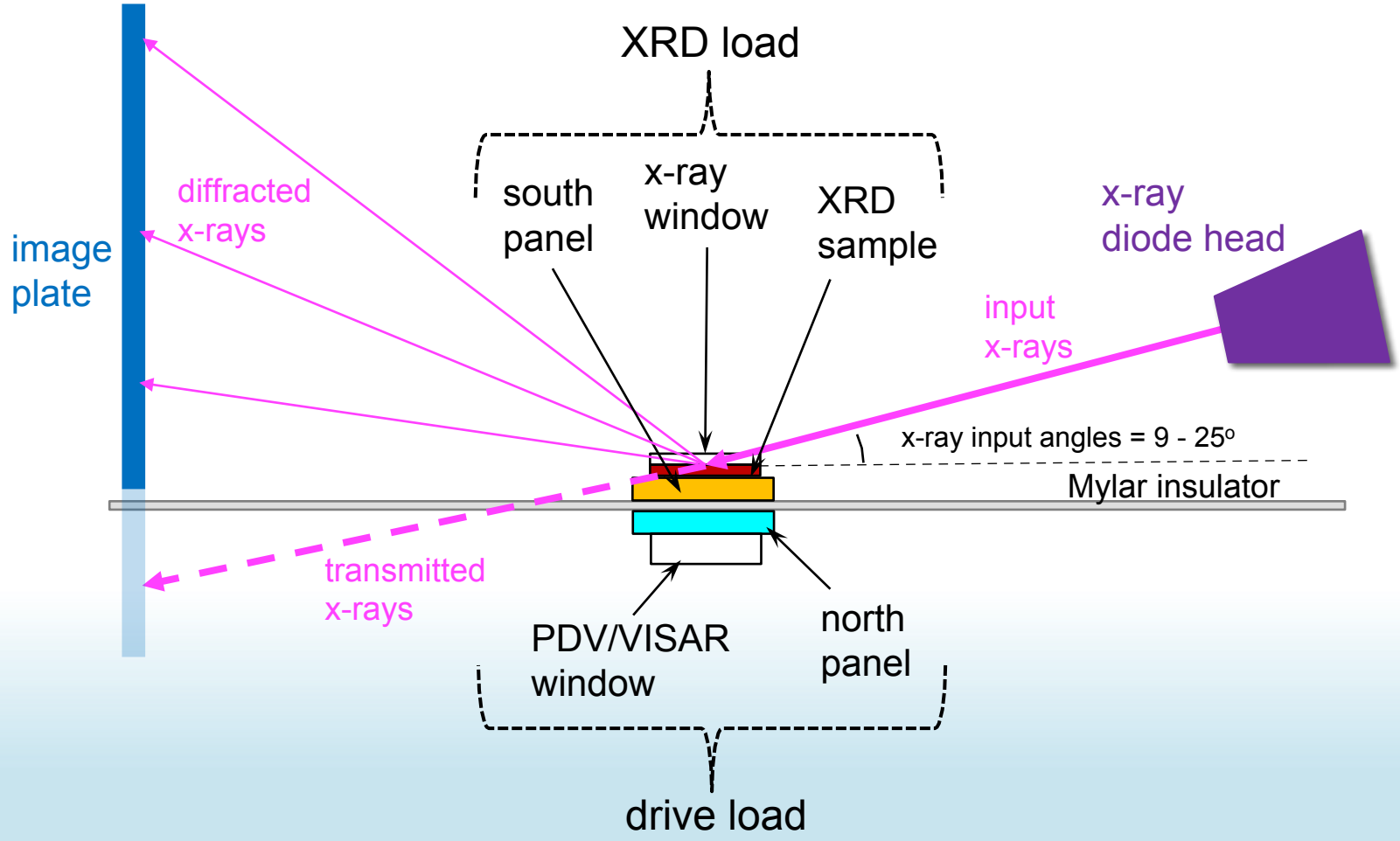






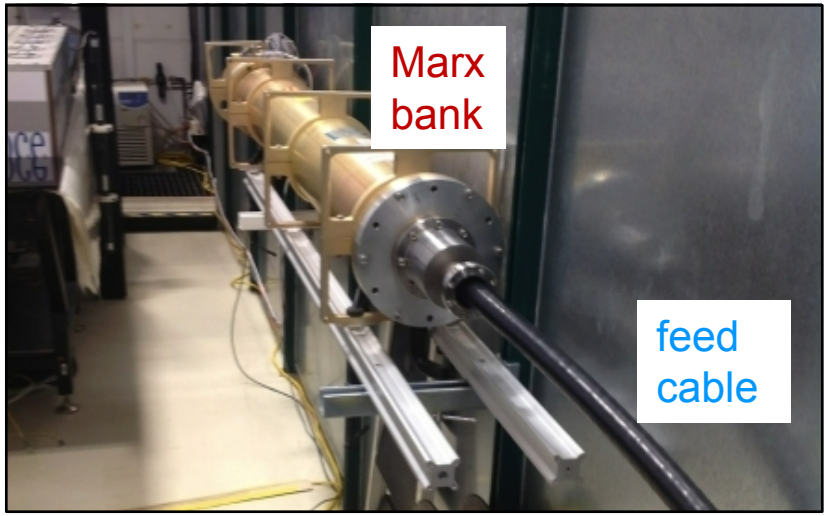
Geometry of input x-rays to load panel and diffracted x-rays

- XRD load on south panel; drive load on north panel
- Current flows on panels' inner surface perpendicular to plane of view



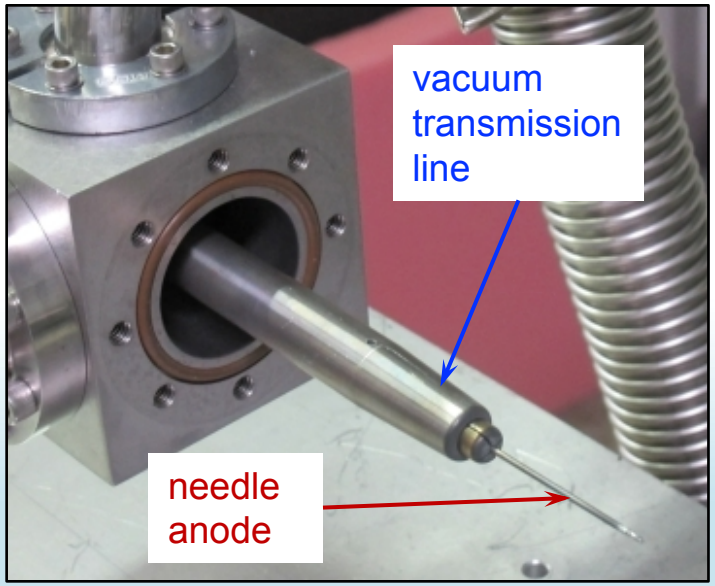
Combination of existing and new hardware

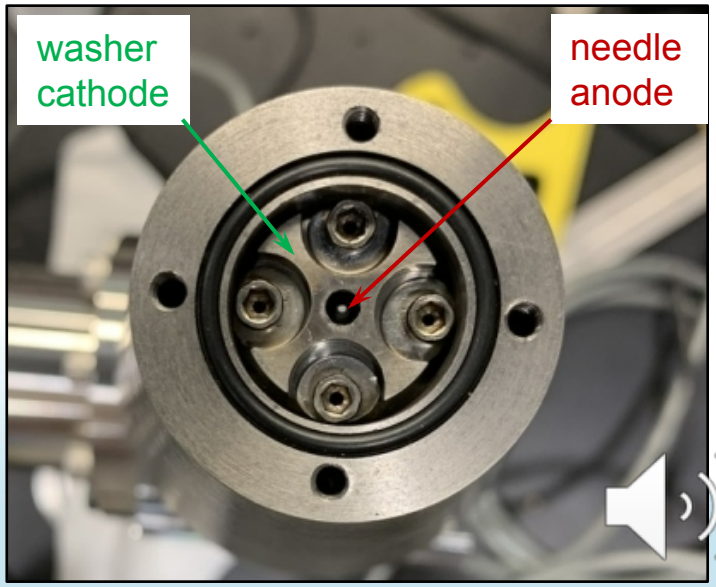
- 35-stage Marx bank high-voltage pulsed power generator charged to 30 kV
- Needle-and-washer electron beam diode via high-voltage coaxial cable



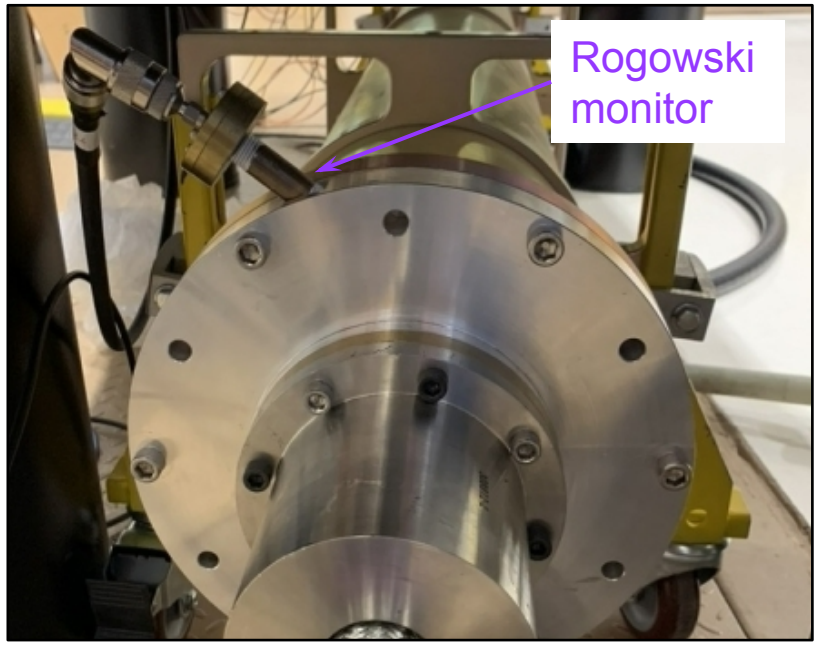
Morgan *et al.*, RSI **79**, 113904 (2008)

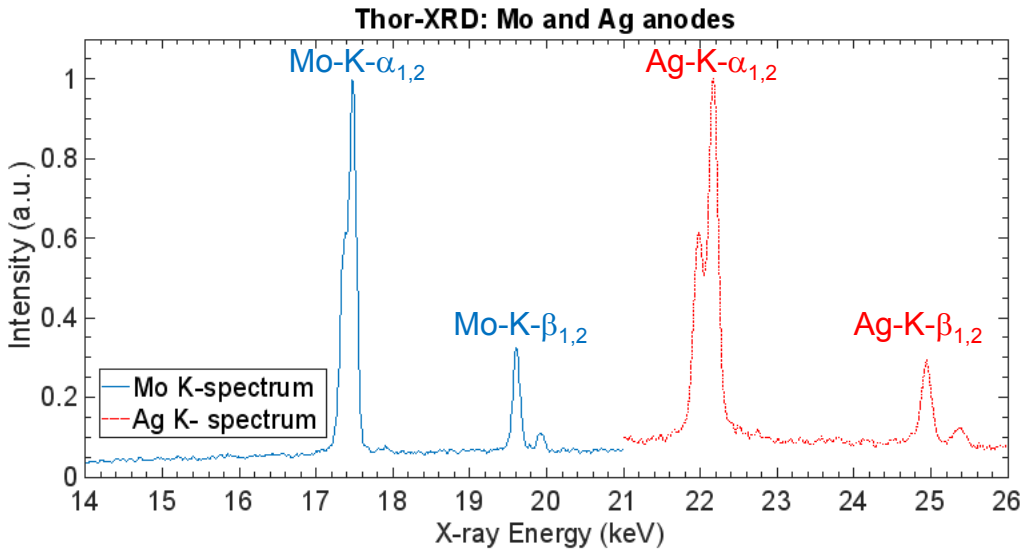
- Select anodes to produce various K_α emission
  - e.g. Cu: 8.0 keV; Mo: 17.4 keV, Ag: 22.1 keV
- Line and bremsstrahlung x-ray emission within 30 ns pulse

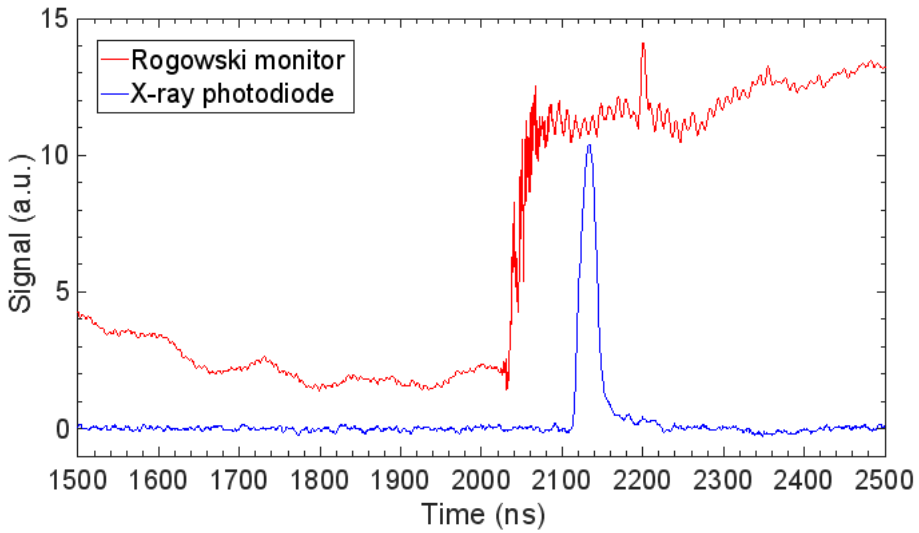


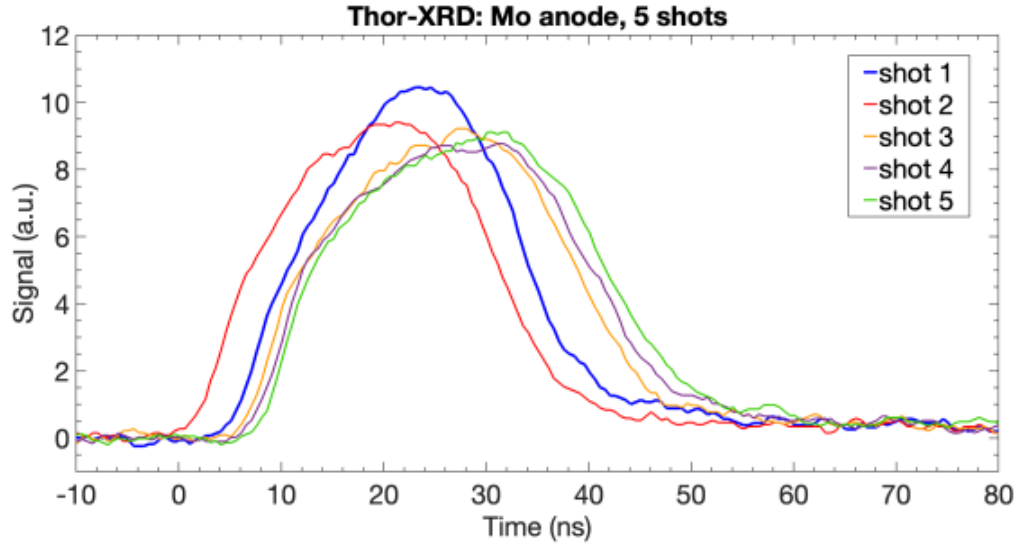


X-ray source parameters and performance



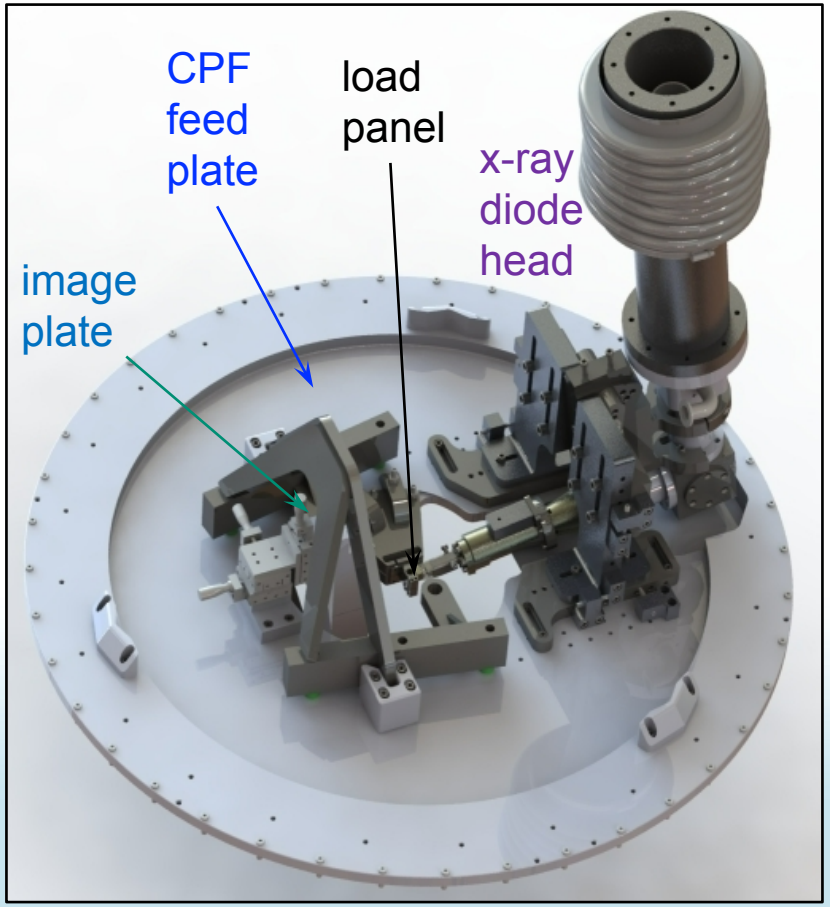


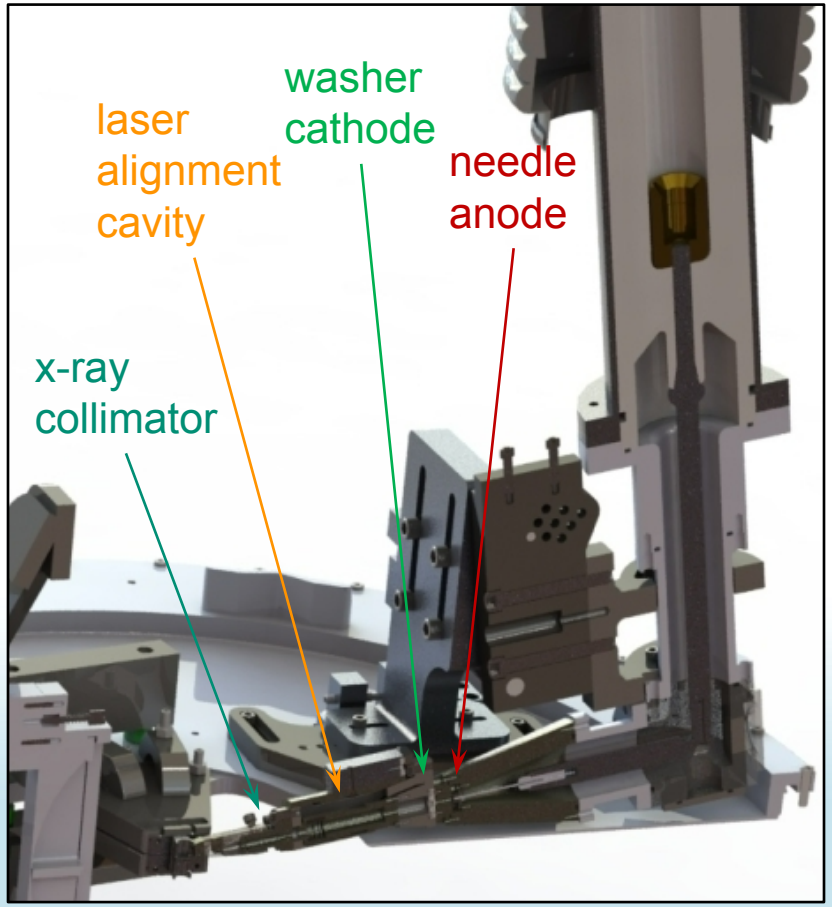




Conical x-ray diode head with 90º bend in transmission line

- Mounted directly to central power flow (CPF) feed plate
- Laser alignment to XRD load

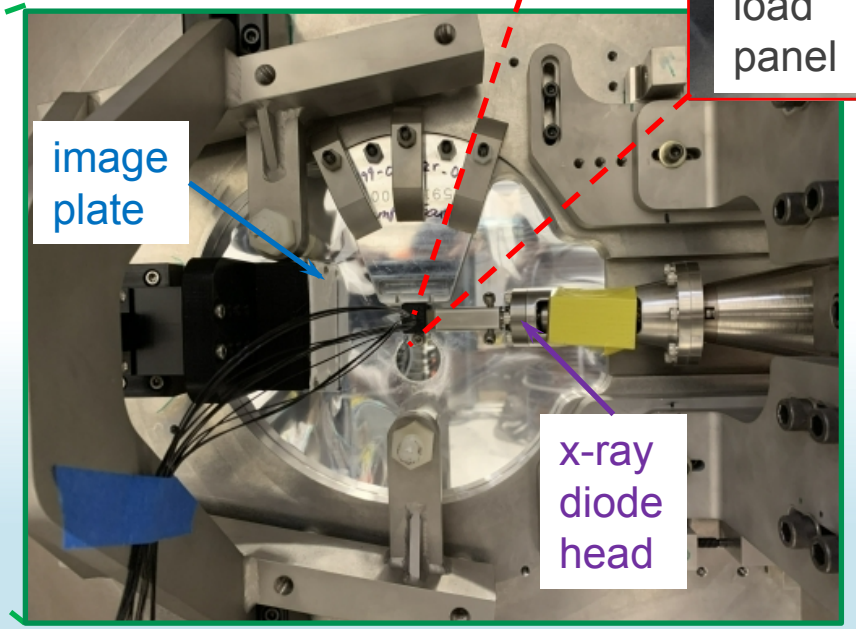


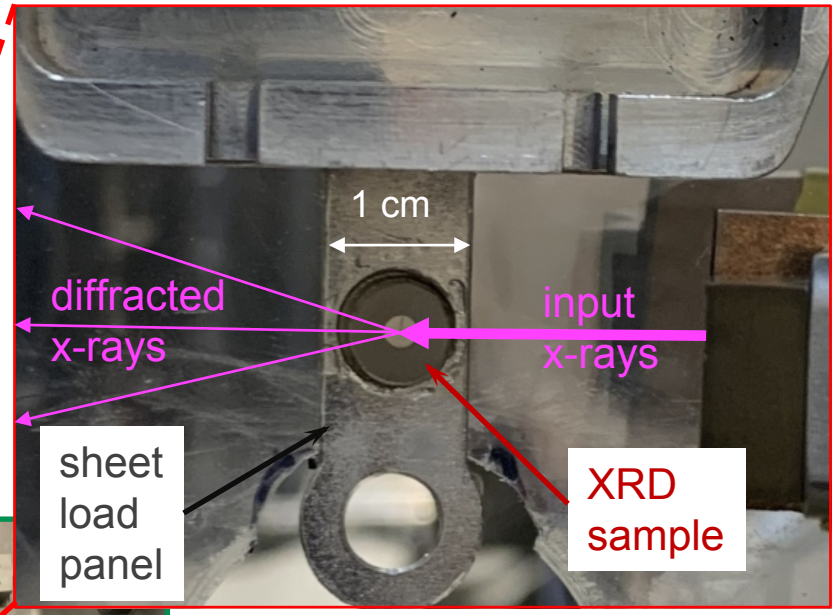


Implementing x-ray diode onto Thor

New hardware to accommodate x-ray diode, load panel, and image plate

- Modified central power flow plate
- Larger load chamber with opening x-ray diode
- Conical x-ray diode head with 90° turn
- Sheet load panel cut by waterjet

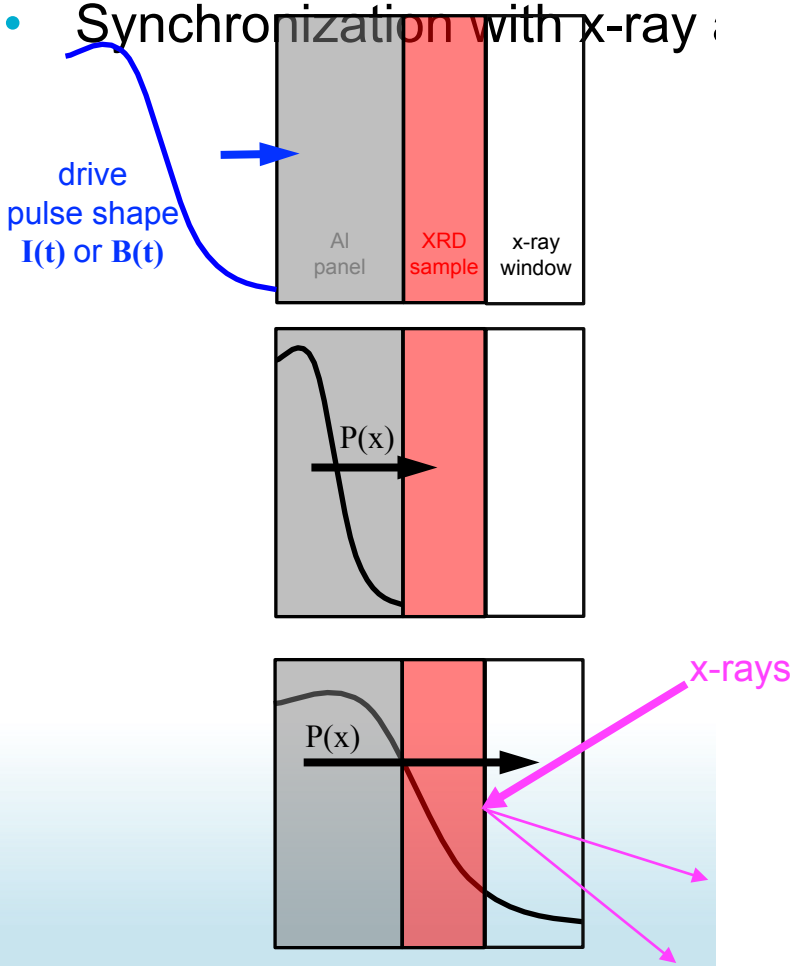


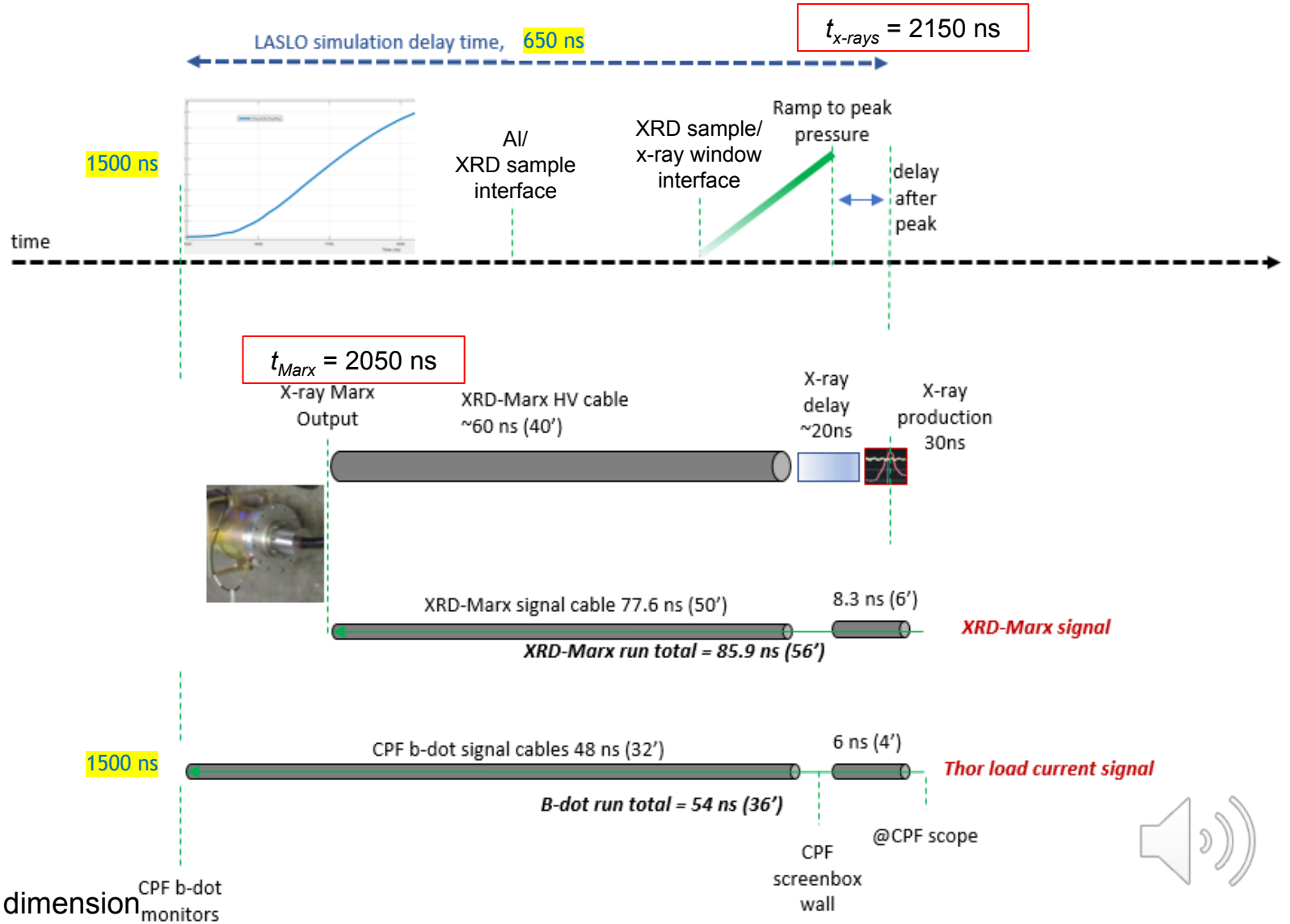


Design of Thor-XRD experiments

LASLO[1] simulations to design drive pulse shape and x-ray timing

- Ramp pressure; panel, sam
- Synchronization with x-ray



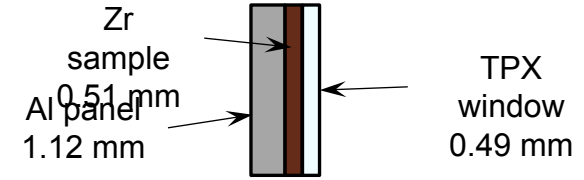


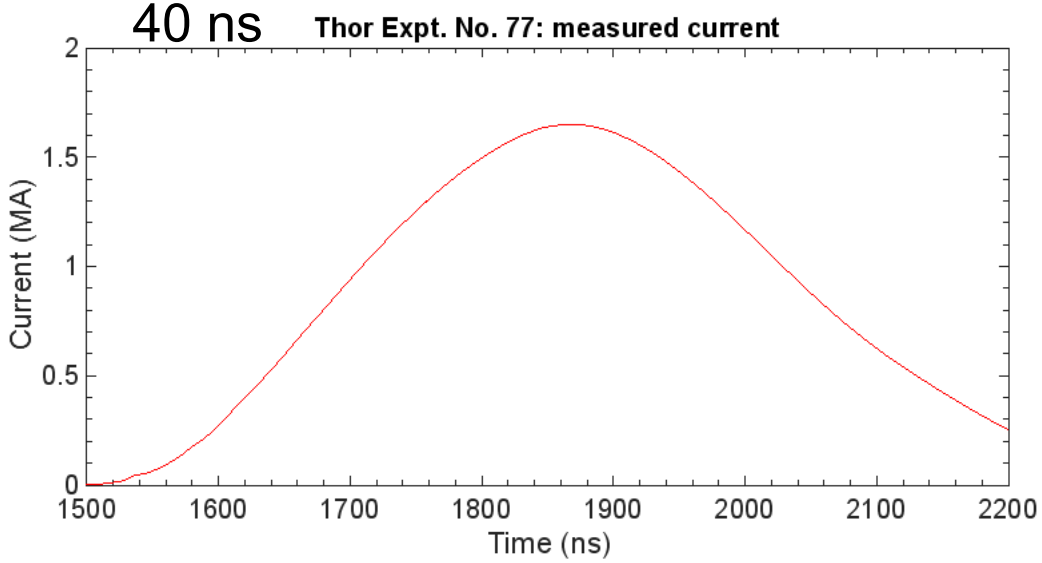
[1]Lagrangian Analysis and Simulation of Loading in One dimension

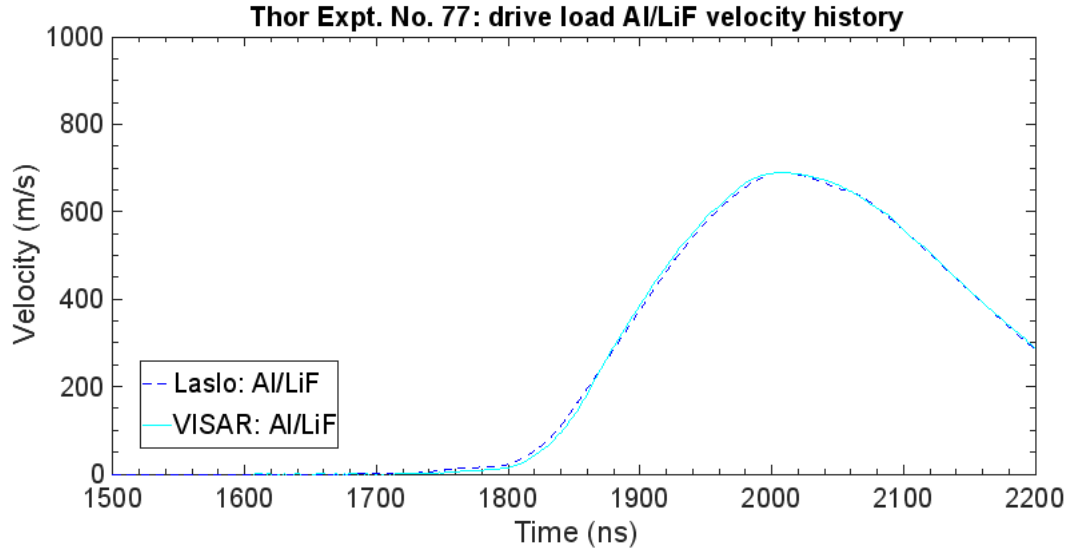
Thor Expt. No. 77: Al panel, Zr sample, TPX window

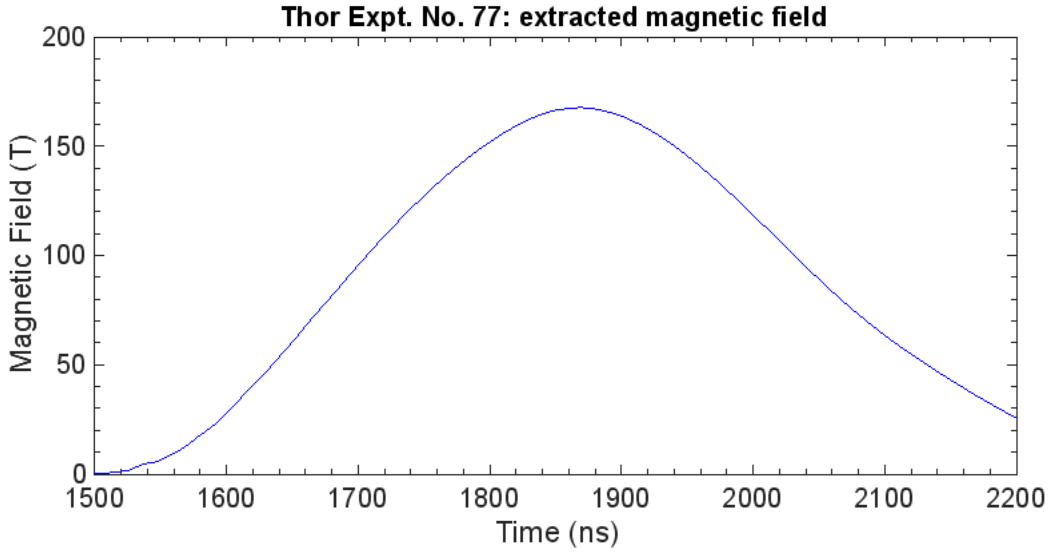
Initial Thor-XRD experiment

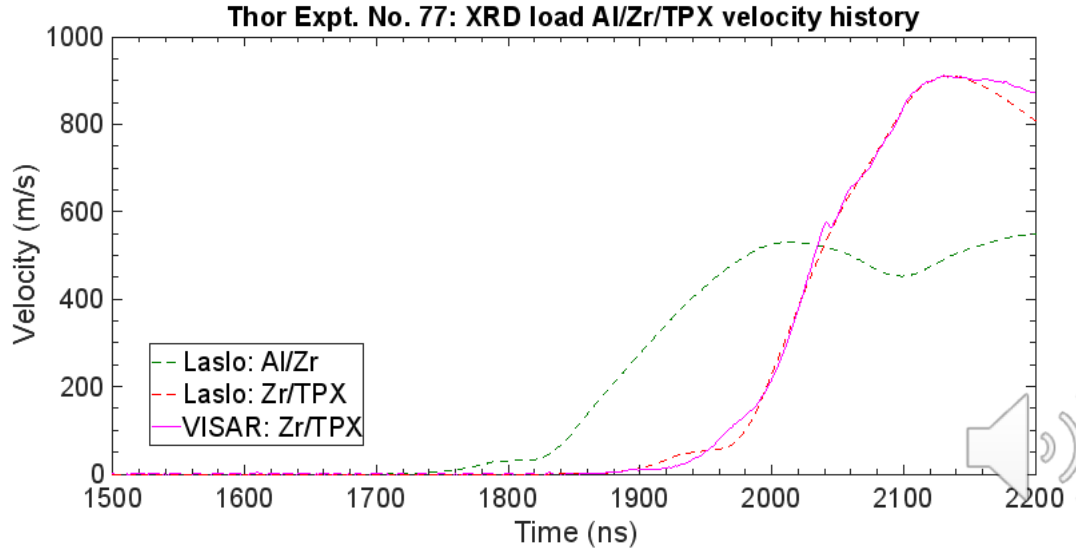
- "Linear" pulse shape: brick towers fired equally-spaced by 40 ns







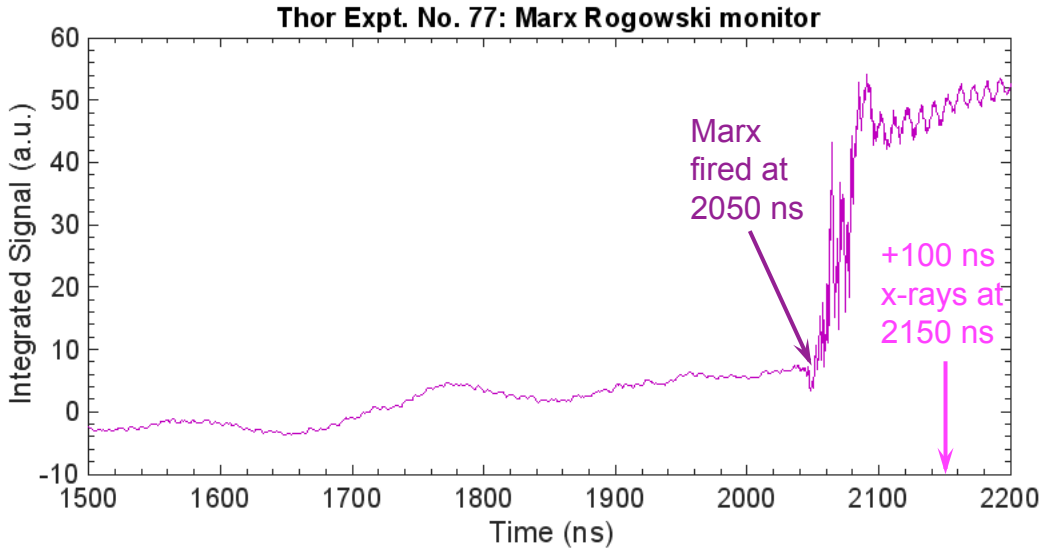


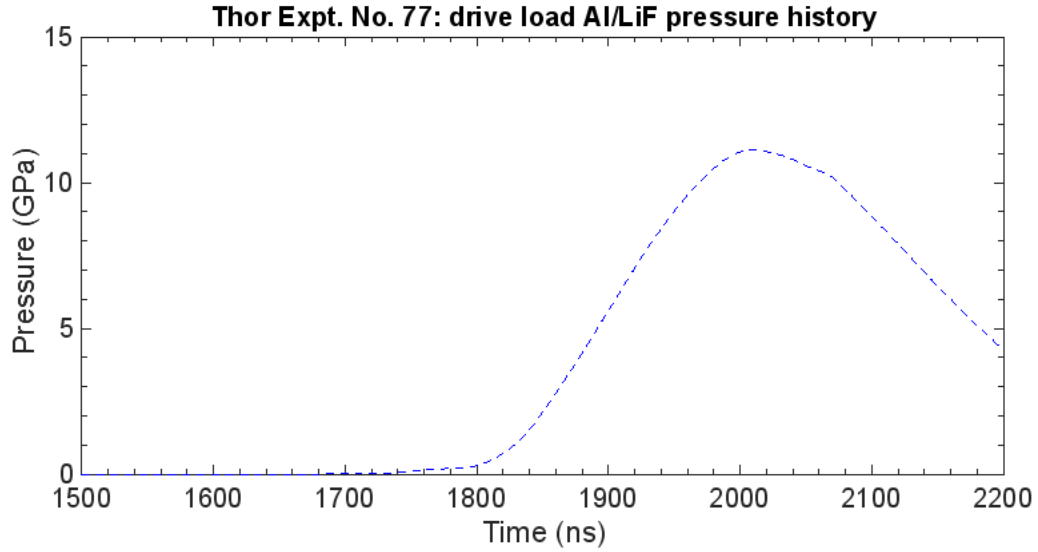


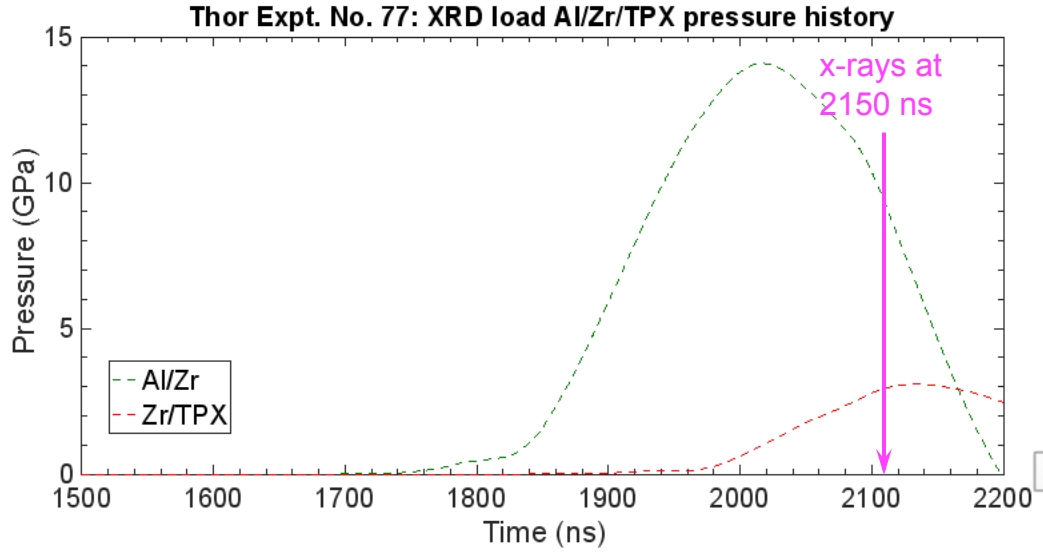
Thor Expt. No. 77: pressure history and x-ray timing

X-rays timed near peak of ramp

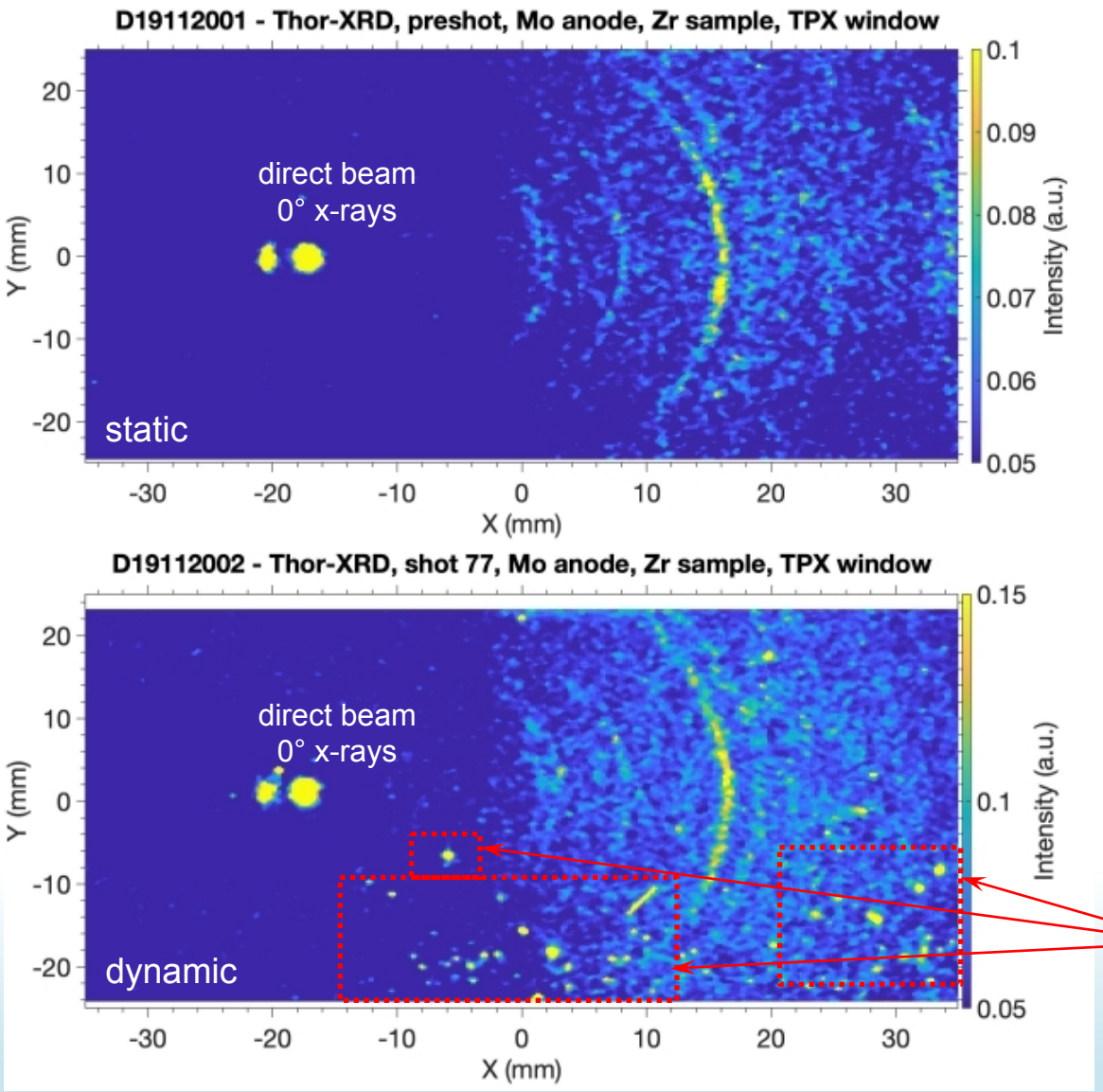
- Drive load peak pressure of 11 GPa
- XRD load peak pressure only 3 GPa due to TPX window





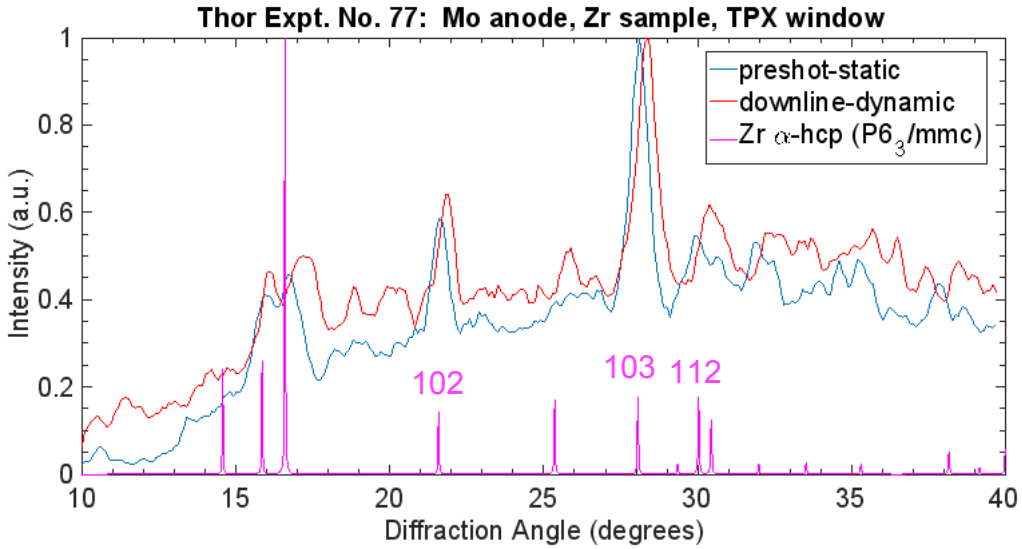


Thor Expt. No. 77: XRD measured Zr lattice compression



Similar static and dynamic XRD patterns

- No noticeable new rings/peaks: no phase change
- Shift in peaks due to lattice compression



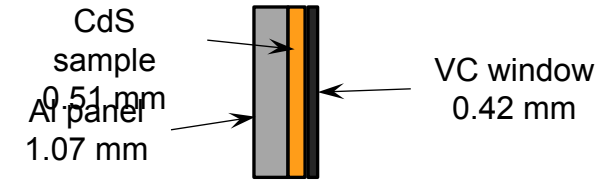
Artifacts on dynamic XRD pattern

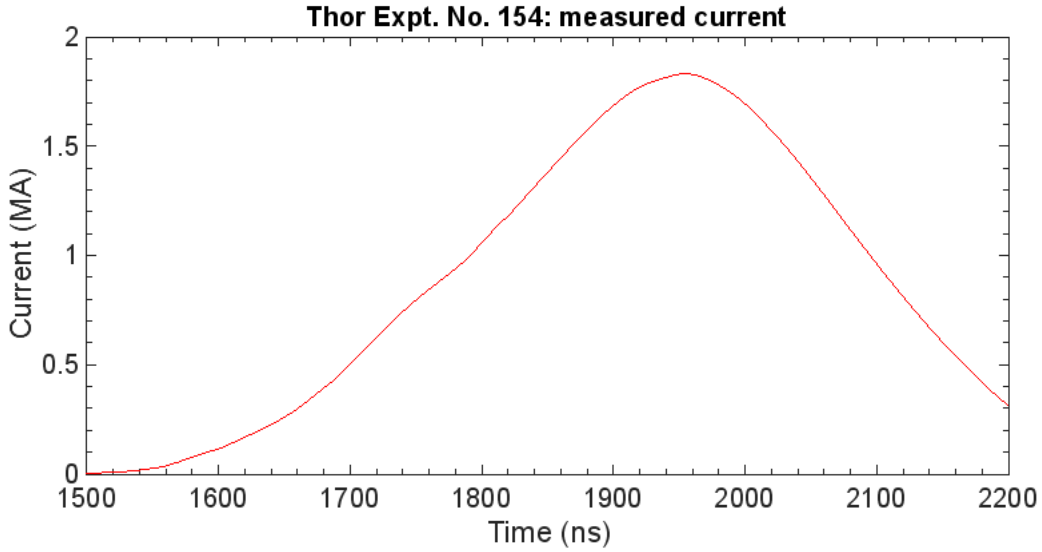
- Electric/magnetic fringing field from Thor firing
- Mitigated with better shielding of IP

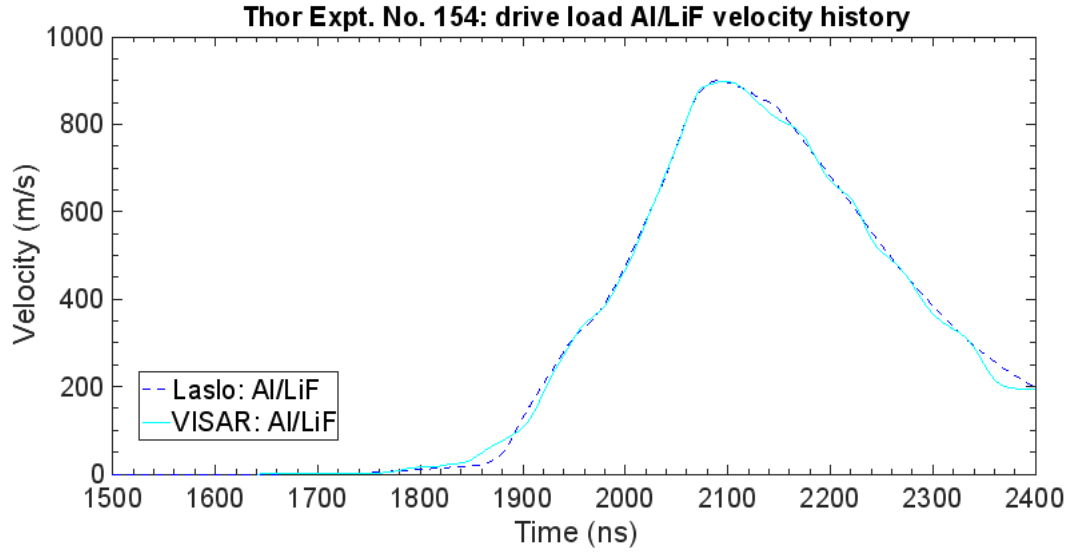
window

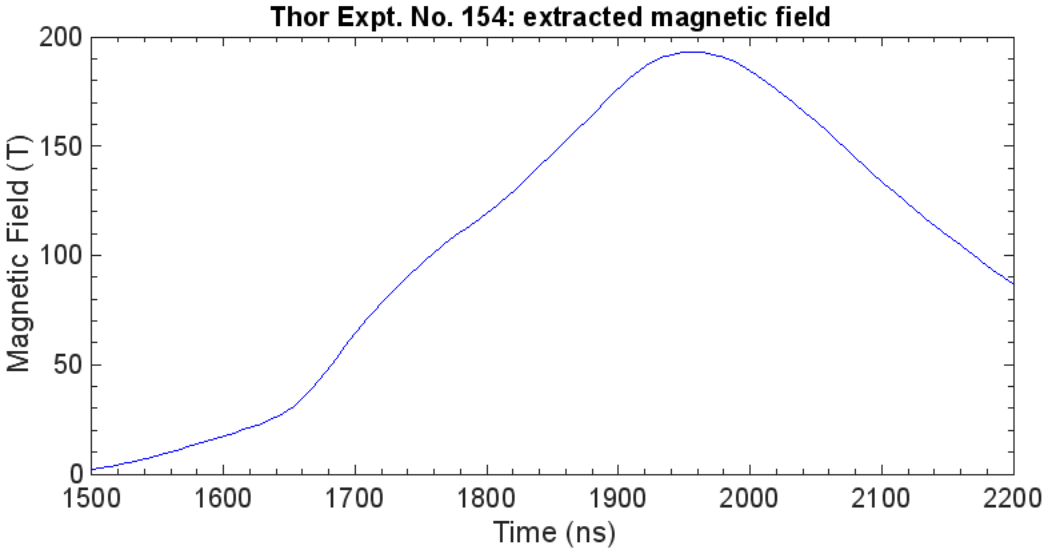
Recent Thor-XRD experiment

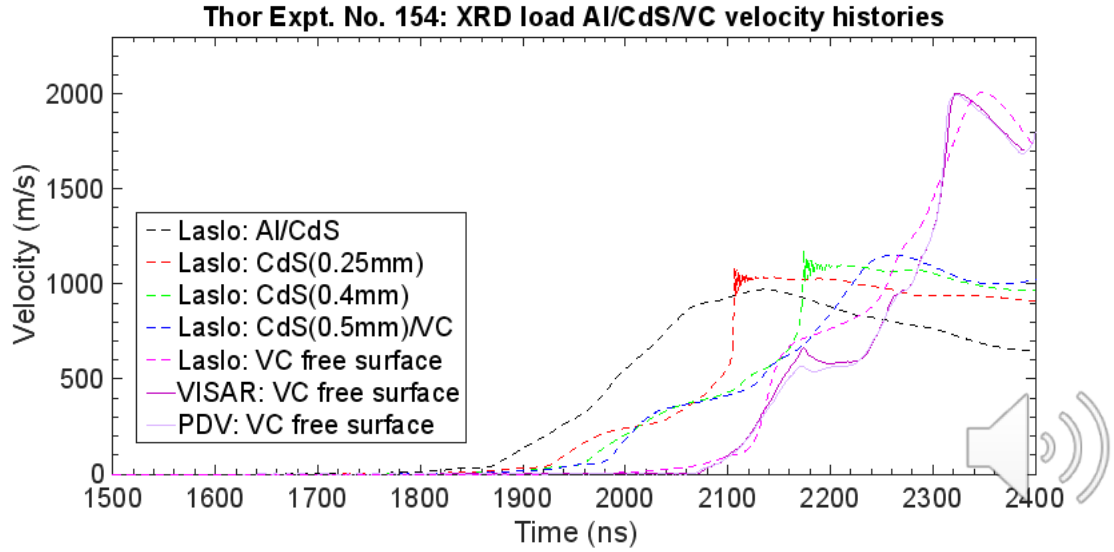
- VISAR/PDV only measure VC free surface









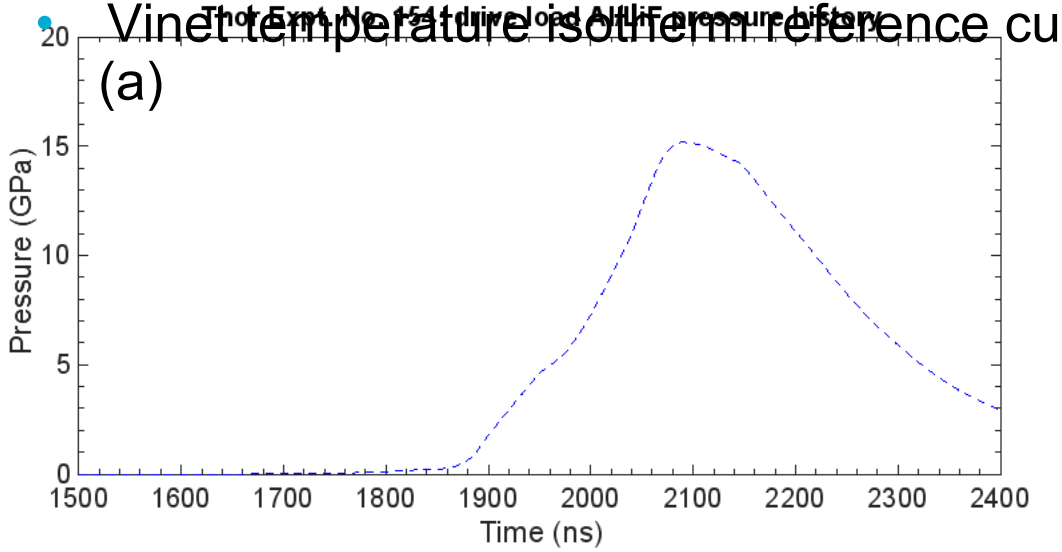


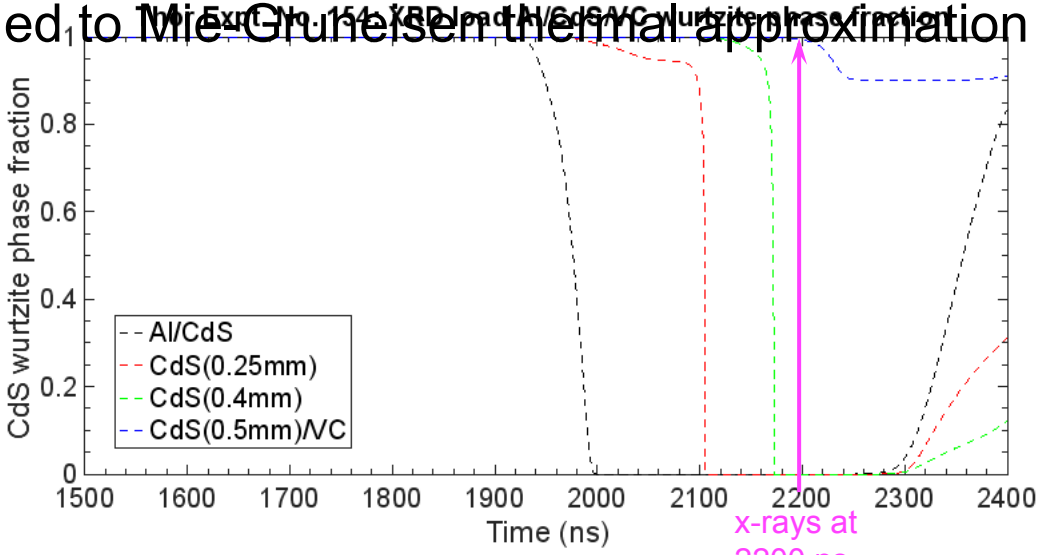
Thor Expt. No. 154: pressure and phase fraction histories

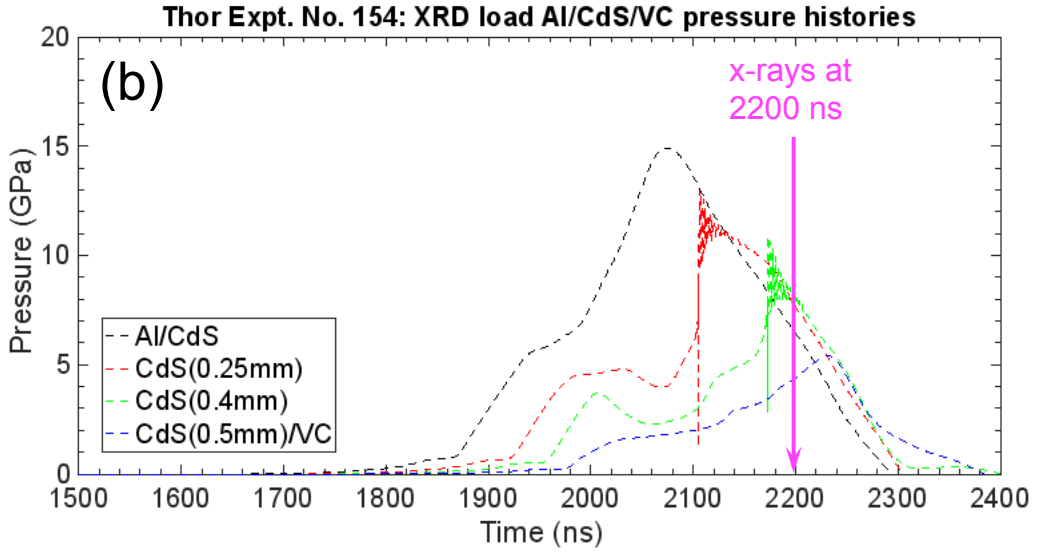
Phase transformation kinetics model for CdS ambient wurtzite and high-pressure rocksalt

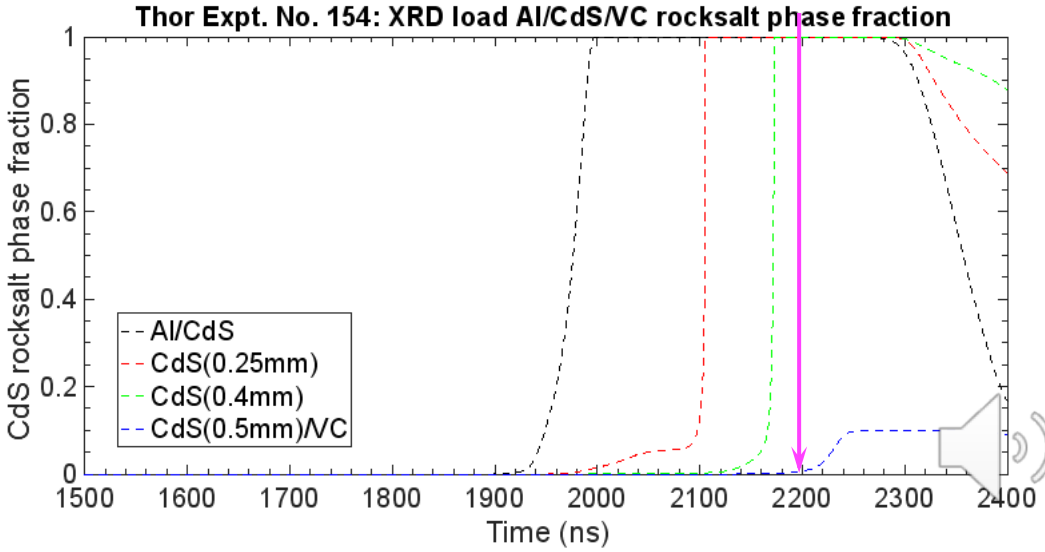
Made by J. Brown

- Vinet temperature isotherm reference curves coupled to Mie-Gruneisen thermal approximation





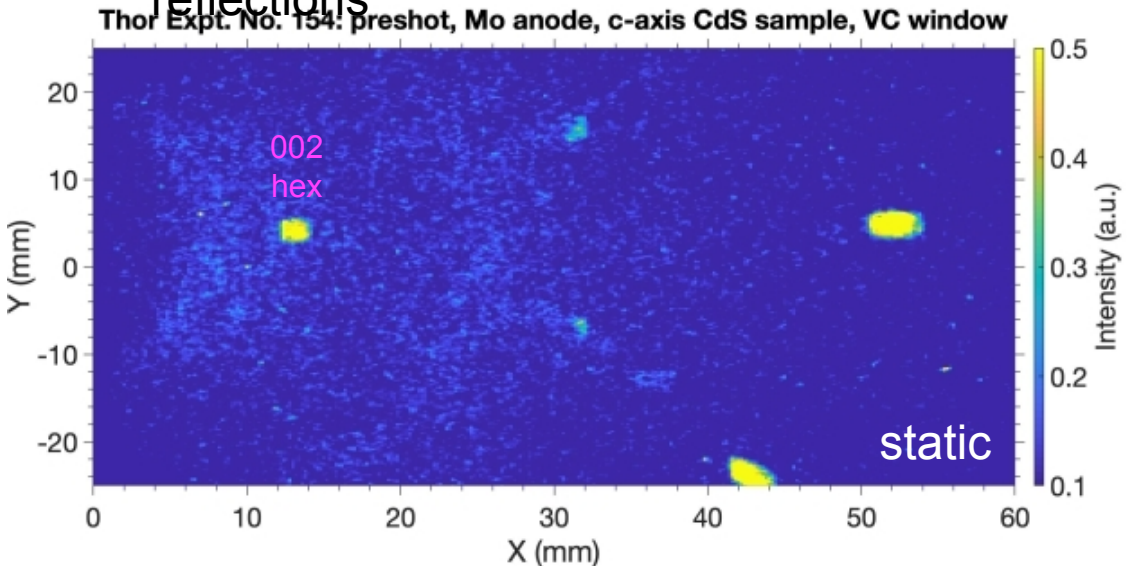


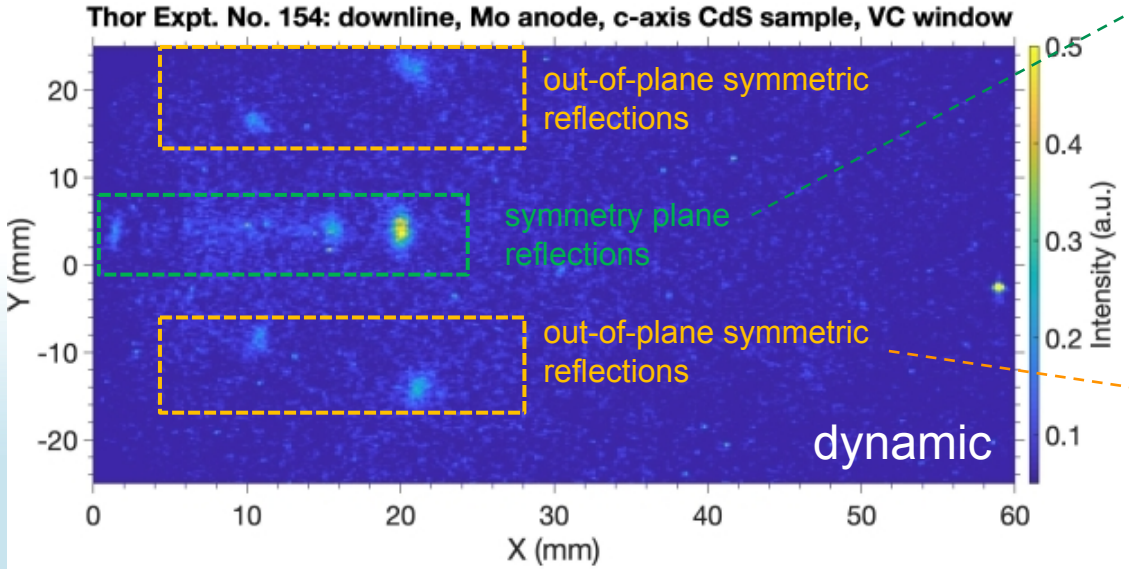


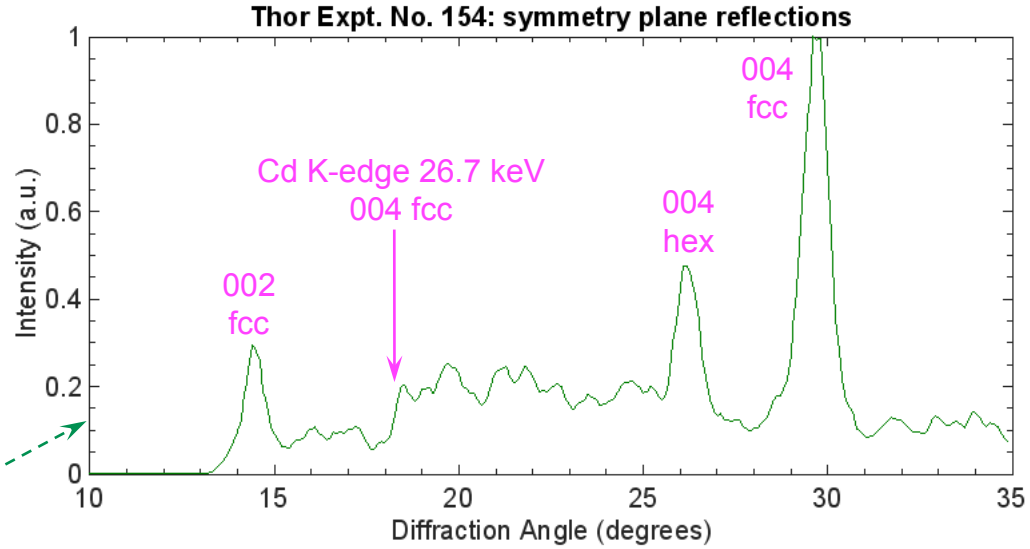
transition

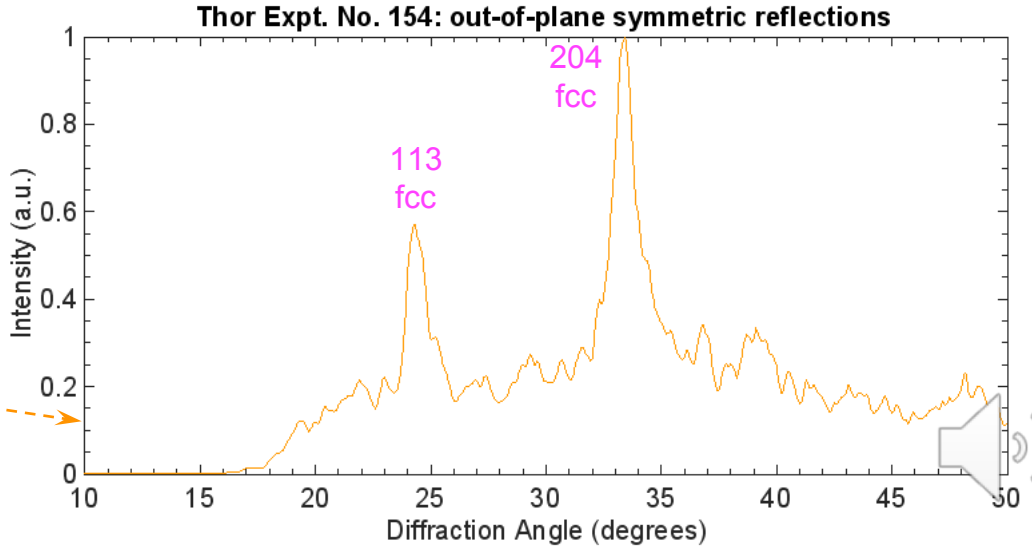
Dynamic XRD pattern clearly shows new set of Laue spots

- Spots along central axis are from “symmetry plane reflections” and off-axis spots are from “out-of-plane reflections”









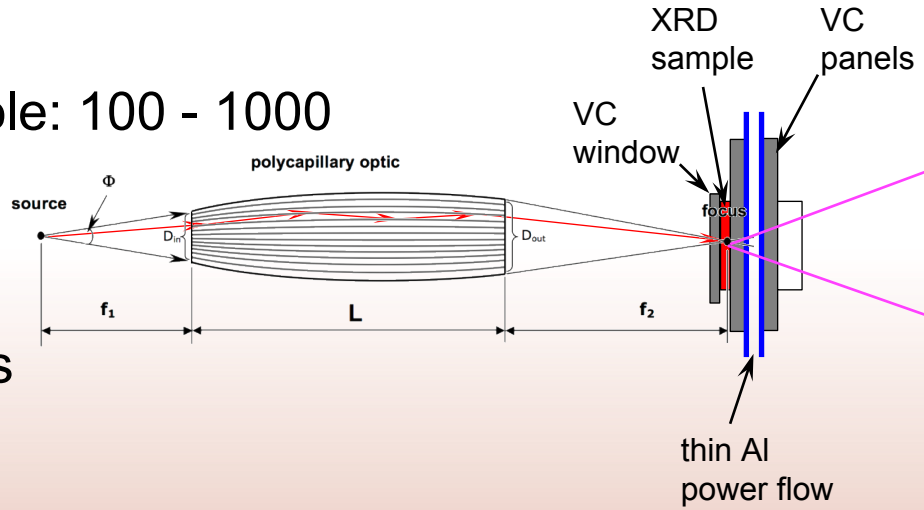
Improvements to Thor-XRD

Fielding Thor-XRD components

- Repeatability of IP data
  - Kinematic holder
  - Registration fiducials
- Mounting x-ray diode head to precision multi-axis stages within Thor load chamber
- Laser alignment reliability
- Higher ramp pressures

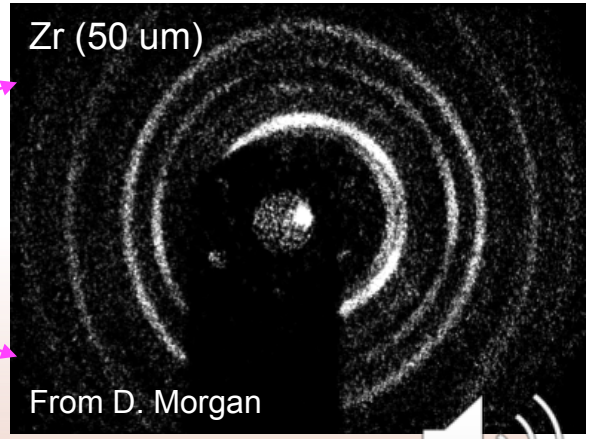
Increasing x-ray flux

- Polycapillary lens
  - X-ray flux gain vs. pinhole: 100 - 1000
- Transmission XRD



Pressed powder samples

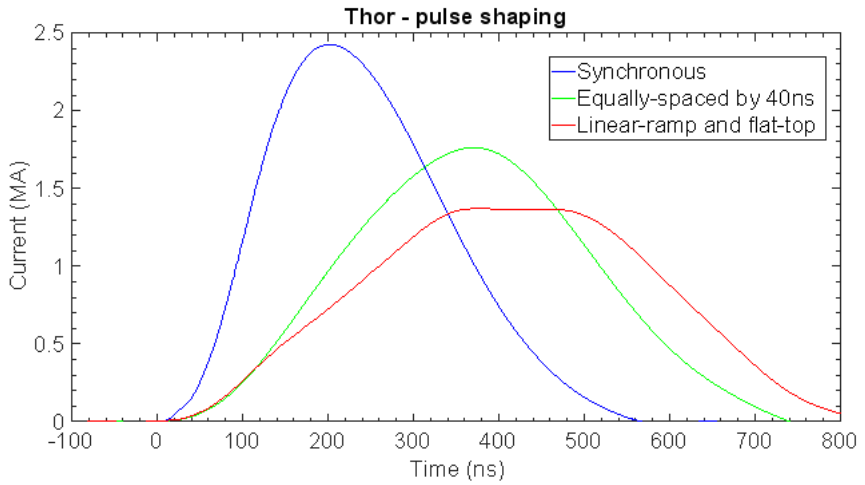
- Fine-tune fabrication process
- Thinner pellets



Upcoming Thor-XRD experiments

Single-crystal samples

- Investigating a-axis CdS samples
- Phase transformation kinetics with XRD at various times along ramp profile
- Modifying pulse shapes for varying ramp rates

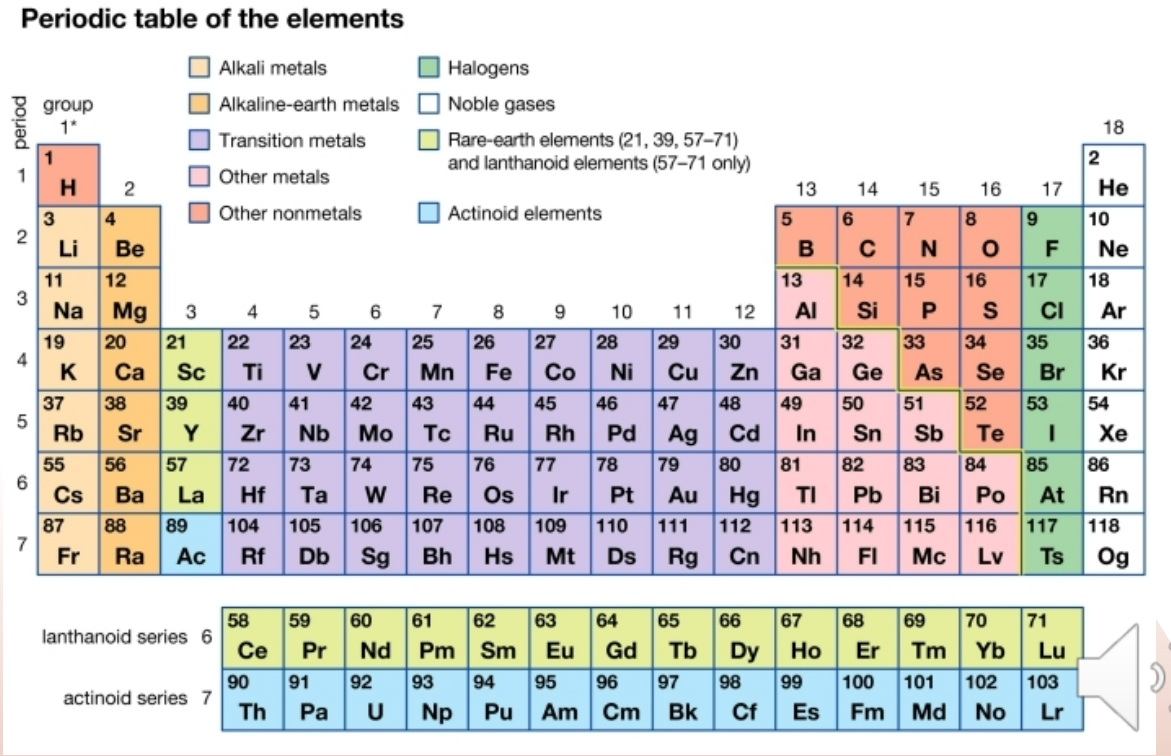


Pressed powder CdS

- Varying particle sizes

Polycrystalline samples

- Metals: e.g., Zr, Bi, Sn, etc.
- Powders: e.g., CaF_2, TiO_2, etc.



Summary

X-ray Diffraction on Thor

- Designed and implemented XRD capability on Thor
- Timed x-rays to ramp wave profile
- Dynamic XRD measured lattice compression of Zr sample
- Measured phase change of single-crystal CdS sample with dynamic XRD

On-going Developments

- Multiple technical improvements
- Continuing Thor-XRD experiments

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- D. V. Morgan, B. S. Stoltzfus, K. N. Austin, J. Usher, E. Breden, L. M. Pacheco, S. Dean, J. L. Brown, S. Duwal, H. Fan, P. Kalita, M. D. Knudson, M. A. Rodriguez, & J. M. D. Lane
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- T. Ao, D. V. Morgan, B. S. Stoltzfus, et al., Rev. Sci. Instrum. **93**, 053909 (2022)