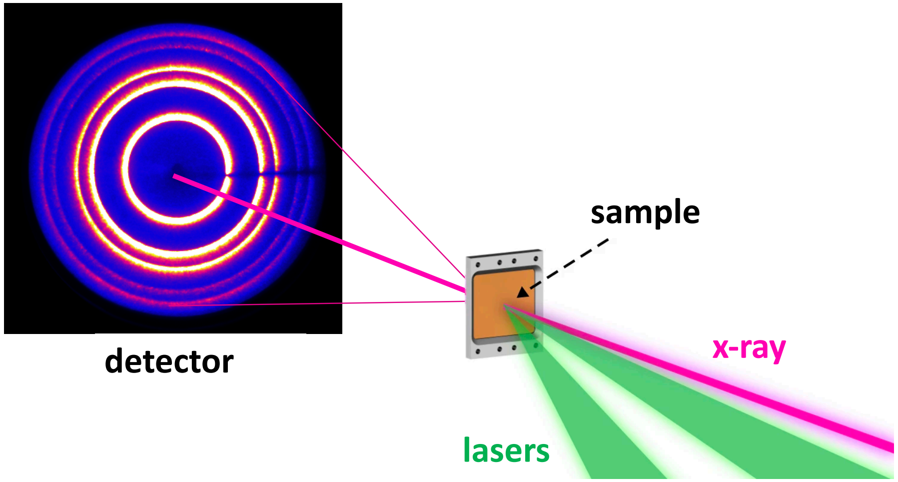
Light-source diffraction studies of phase transitions under shock loading

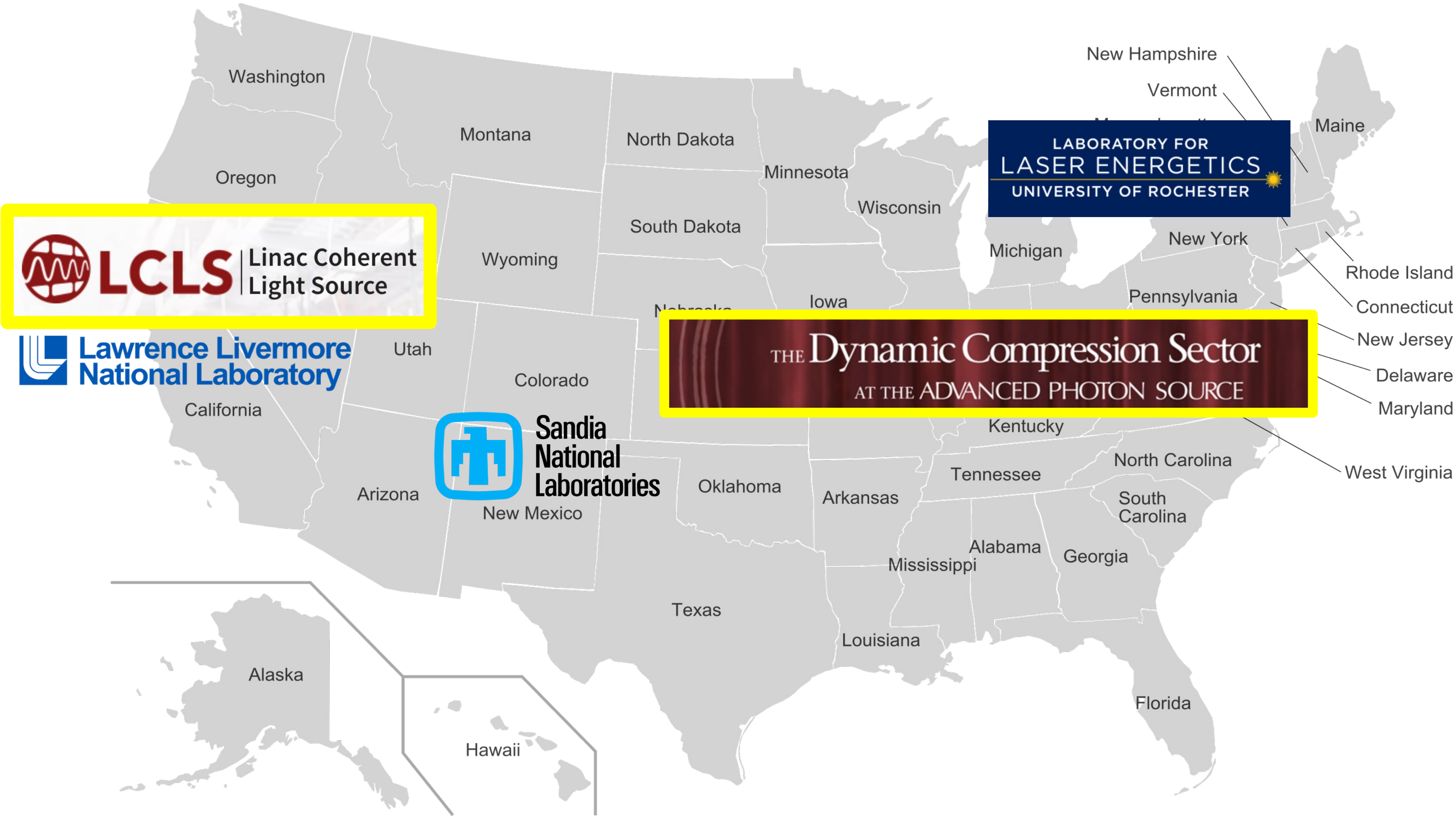


Sally June Tracy

Carnegie Institution for Science

HEDS seminar, LLNL - Feb. 18, 2022

New capabilities for dynamic compression



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Shock waves in crystalline materials

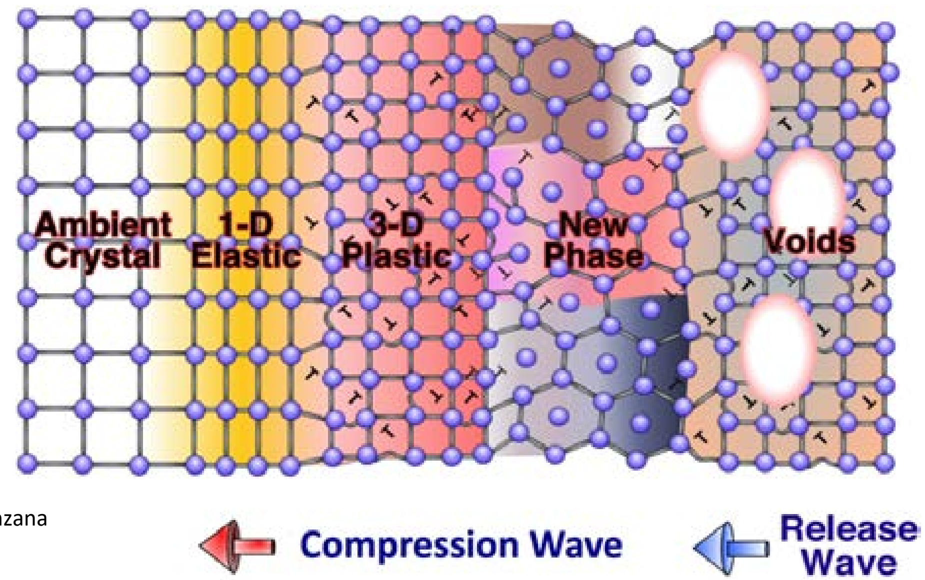

Image: Lorenzana

Atomic-length scale processes

- Elastic compression
- Plastic deformation
- Phase changes
- Kinetics and metastability

In situ **XRD can be critical to determine lattice-level structural information**

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X-ray diffraction on the ns time scale of shock loading events

LINAC Coherent Light Source (LCLS):
- ~10^{12} photons/pulse
- ~100 femtosecond pulses

Image: SLAC

Advanced Photon Source (APS):
- ~10^{9} photons/pulse
- ~100 picosecond pulses

Image: ANL

Highlight recent experiments:
- Laser-drive compression carbonates at the LCLS
- Gas–gun compression of ZnO & MgF_2 at the APS

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Carbonates in the deep Earth

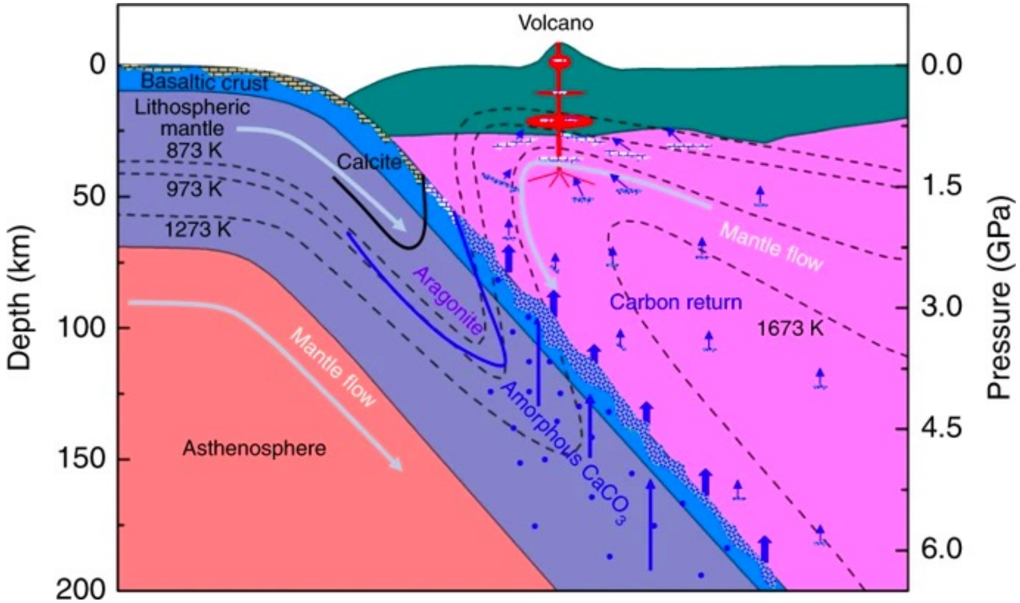

Fig. Huo et al., 2019

High-P-T phase stability of carbonates is fundamental for understanding the global carbon cycle and carbon storage in the deep Earth

Rapid loading of minerals during impact events

Interpretation of shock metamorphism
Understanding role of impact devolatilization

- Solid-state phase transitions
- Melting
- Dissociation
- Dissolution of solid residual phases

$$CaCO_3 \rightarrow CaO^{solid} + CO_2^{gas}$$

Laser-based shock experiments are effective in reproducing shock effects observed in naturally shocked minerals

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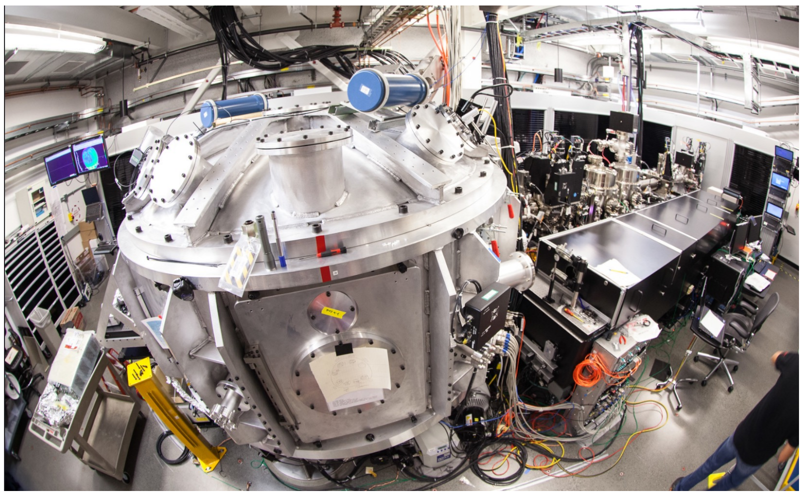
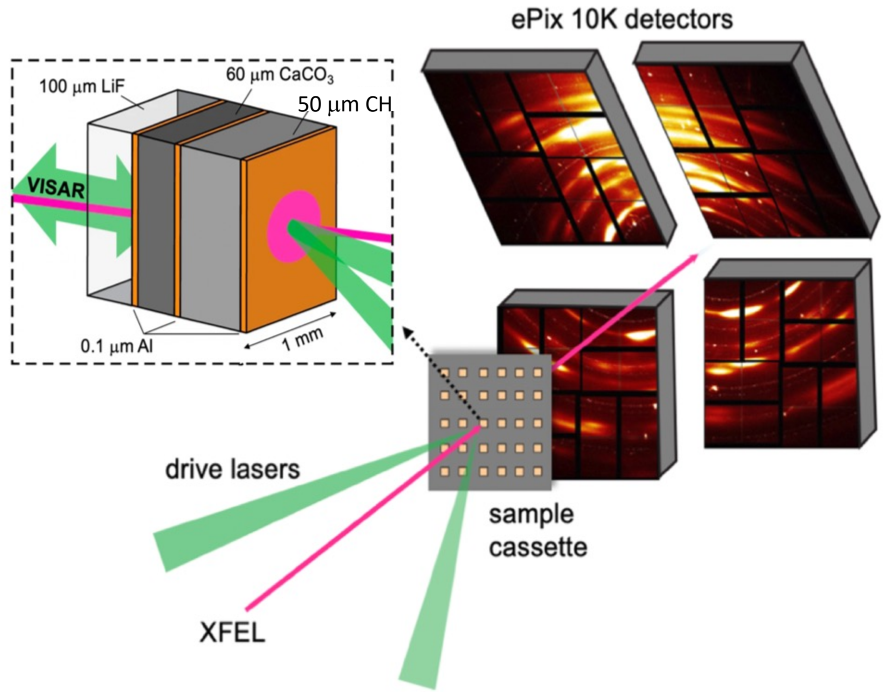
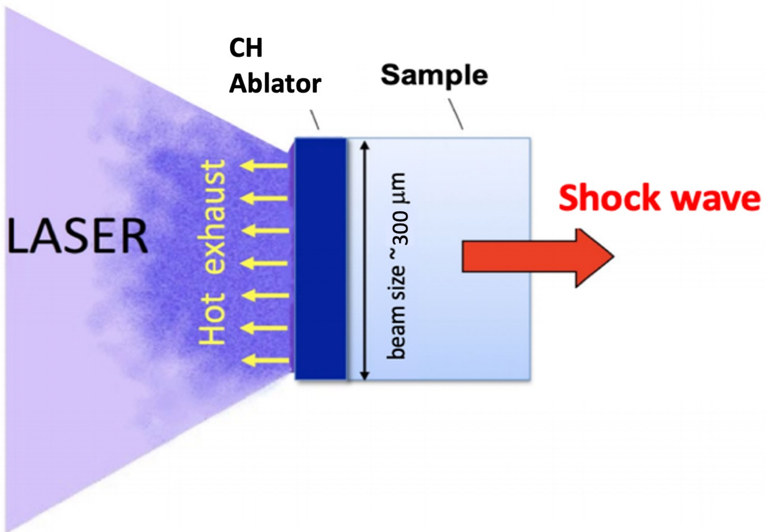
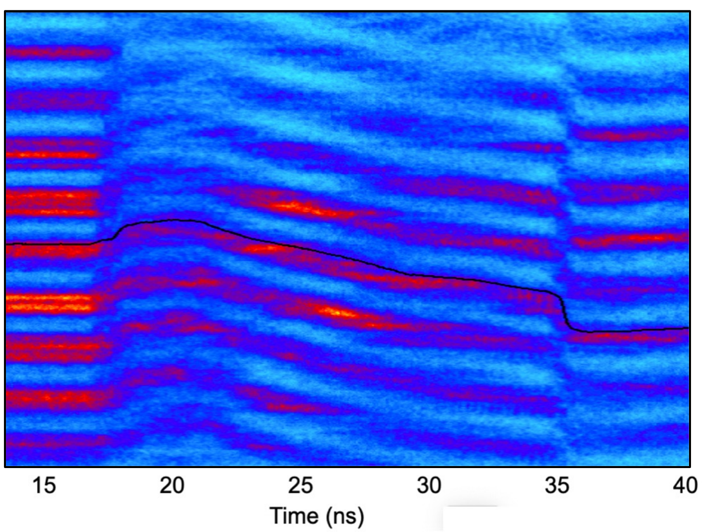
Pump-probe X-ray diffraction at Matter in Extreme Conditions (MEC) beamline





MEC Experiments





Samples:

Calcite – $CaCO_3$

- Limestone
- Calcite single crystals

Magnesite – $MgCO_3$

- Polycrystal (7% porosity)
- Natural gem

Drive Laser:

- 150 & 300-mm phase plates
- 10-15 ns flattop pulse
- Laser energies 10-70 J

XFEL:

- 9.5 & 14 keV
- 20-mm spot size

Line VISAR:

- Wave profiles collected at free surface or sample-LiF interface

- Pressure & shot timing determined via impedance matching

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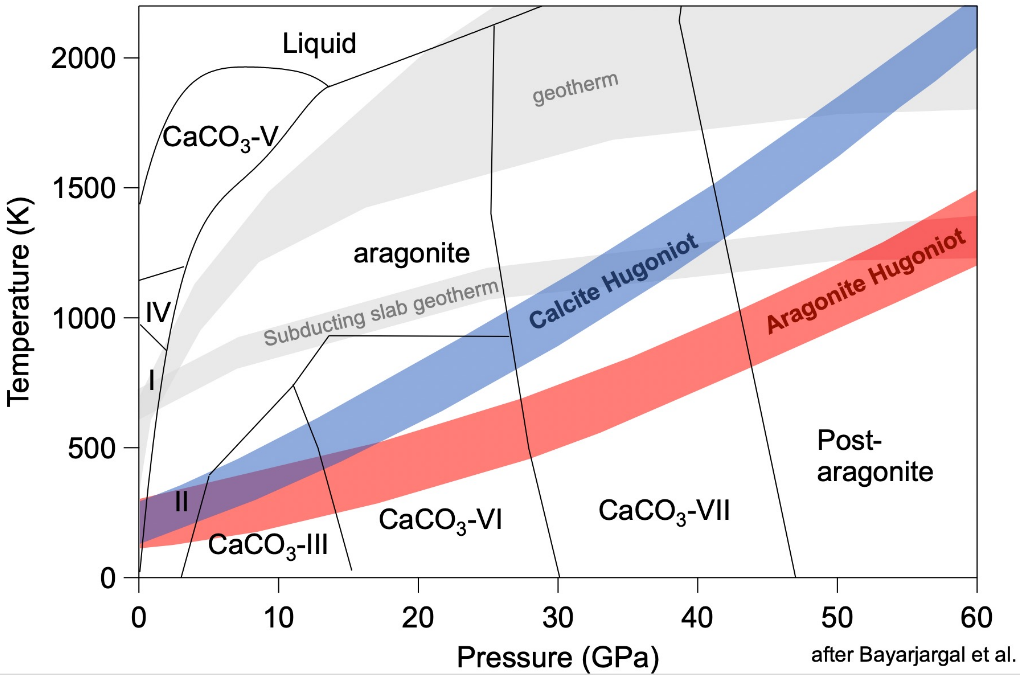
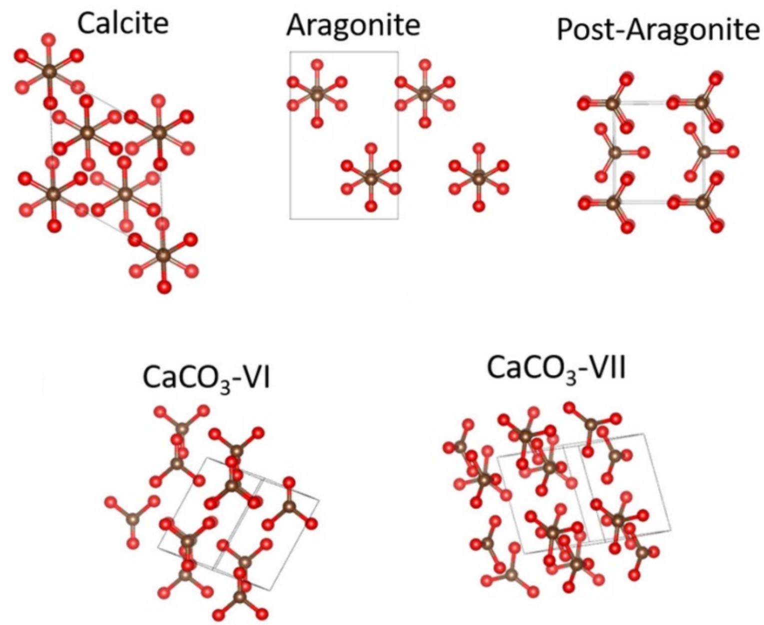
Complex polymorphism in calcium carbonate





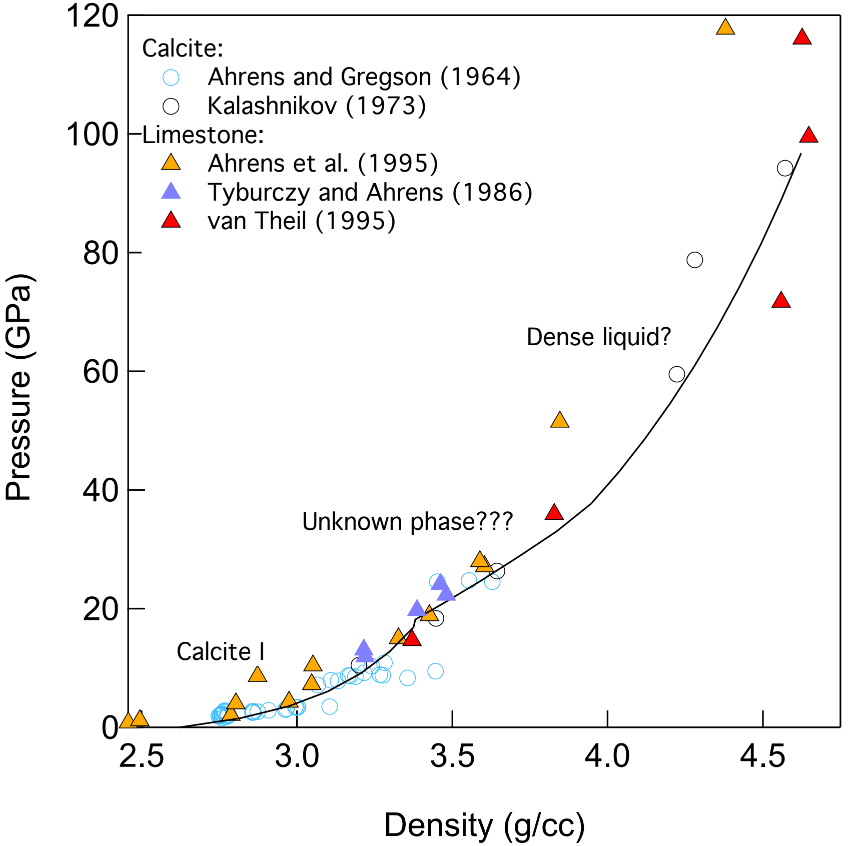
Rhombohedral $CaCO_3$-I transitions to a series of low-symmetry phases involving reorientation and tilting of the CO_3 groups

Early calcite shock-wave experiments

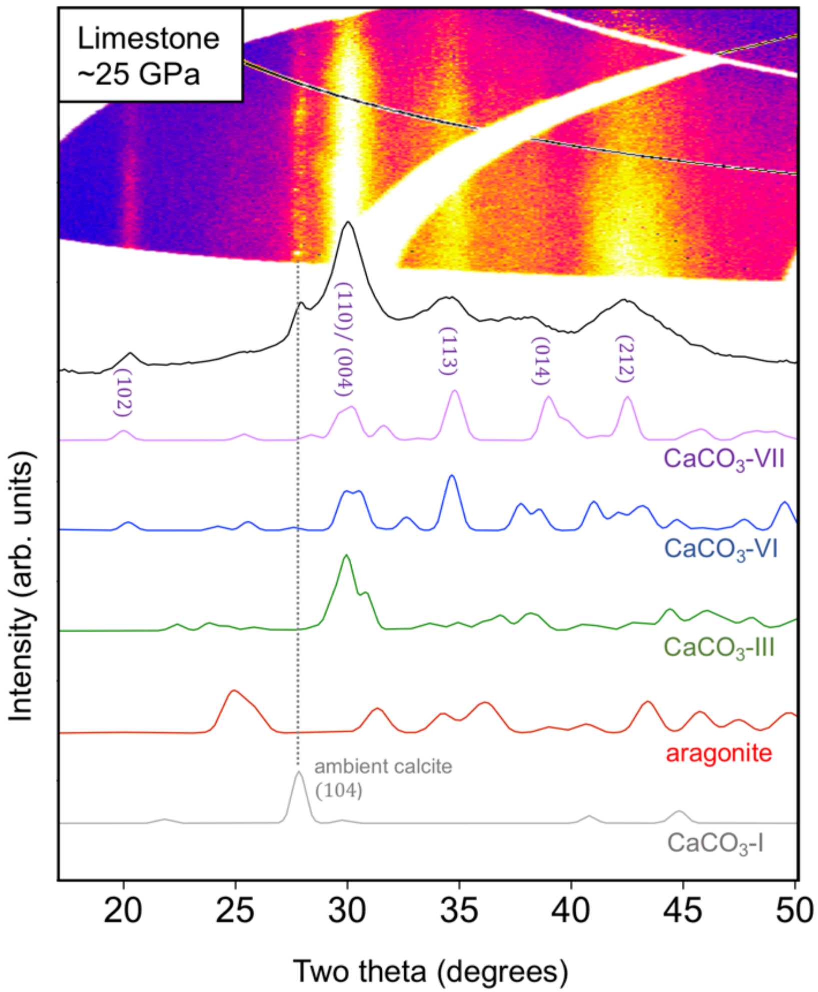
Gas-gun studies identified a phase transition ~20 GPa

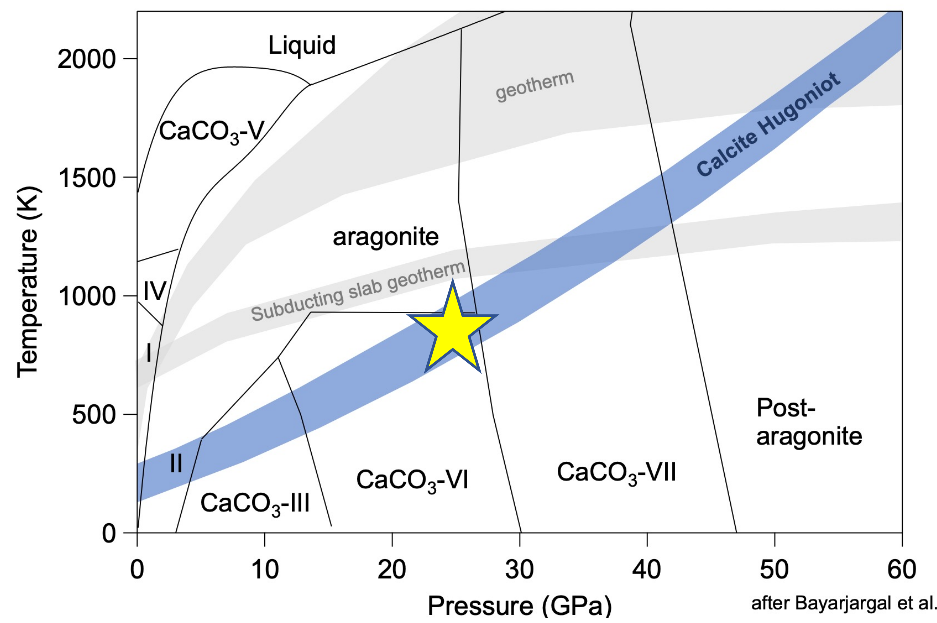
Based on thermodynamic considerations, high-pressure phase not consistent with the Calcite I-III phases known at the time

Conflicting results concerning degassing with reports ranging between 1-50 mole% devolatilization



Phase transition at 25 GPa





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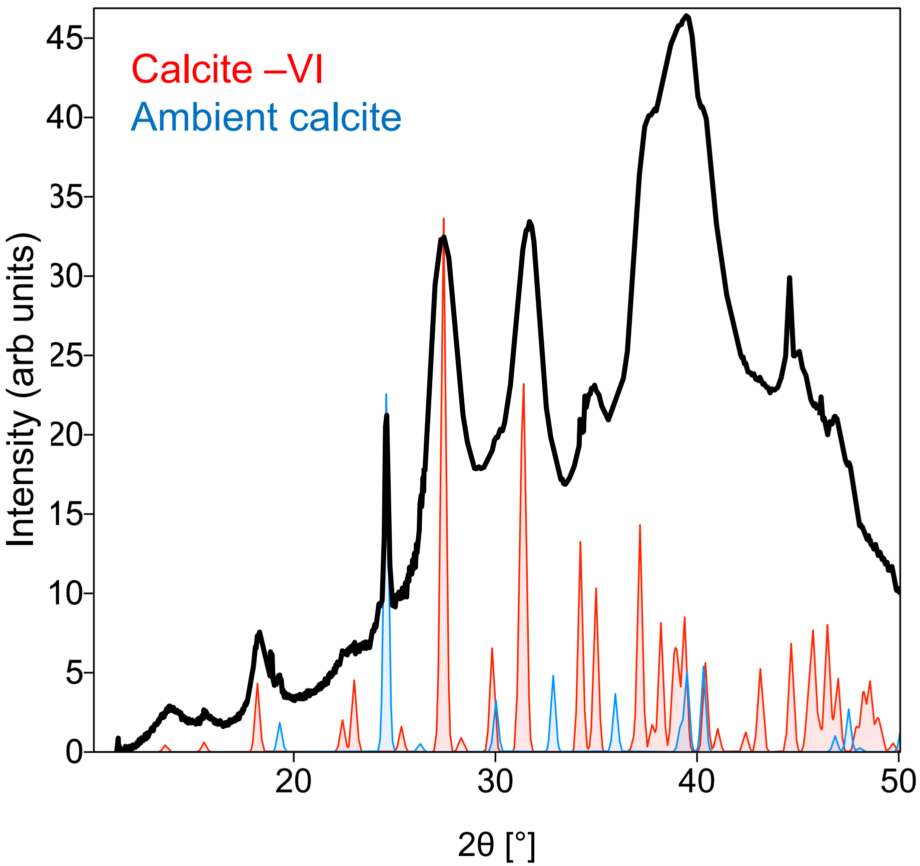
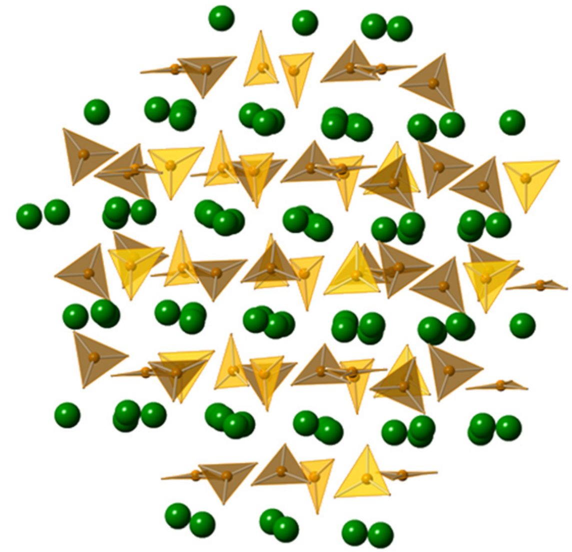
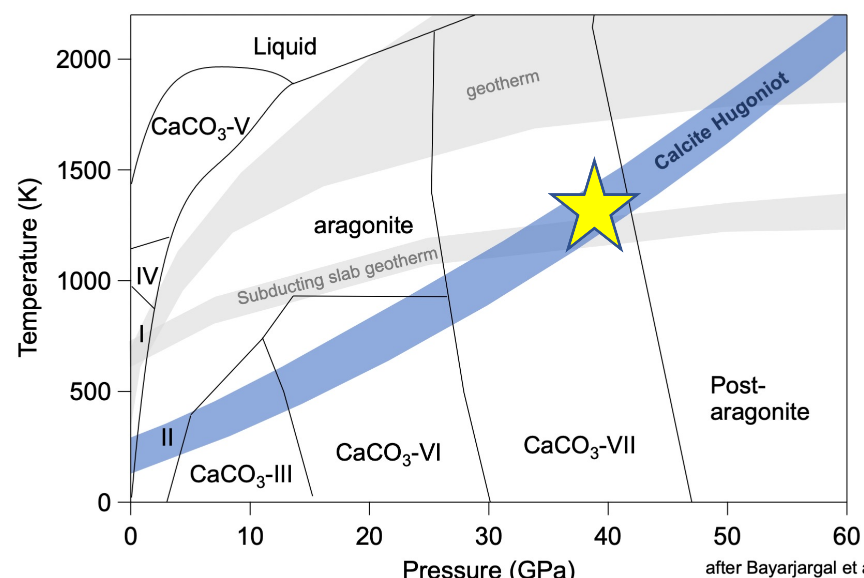
$CaCO_3$-VI at 40 GPa



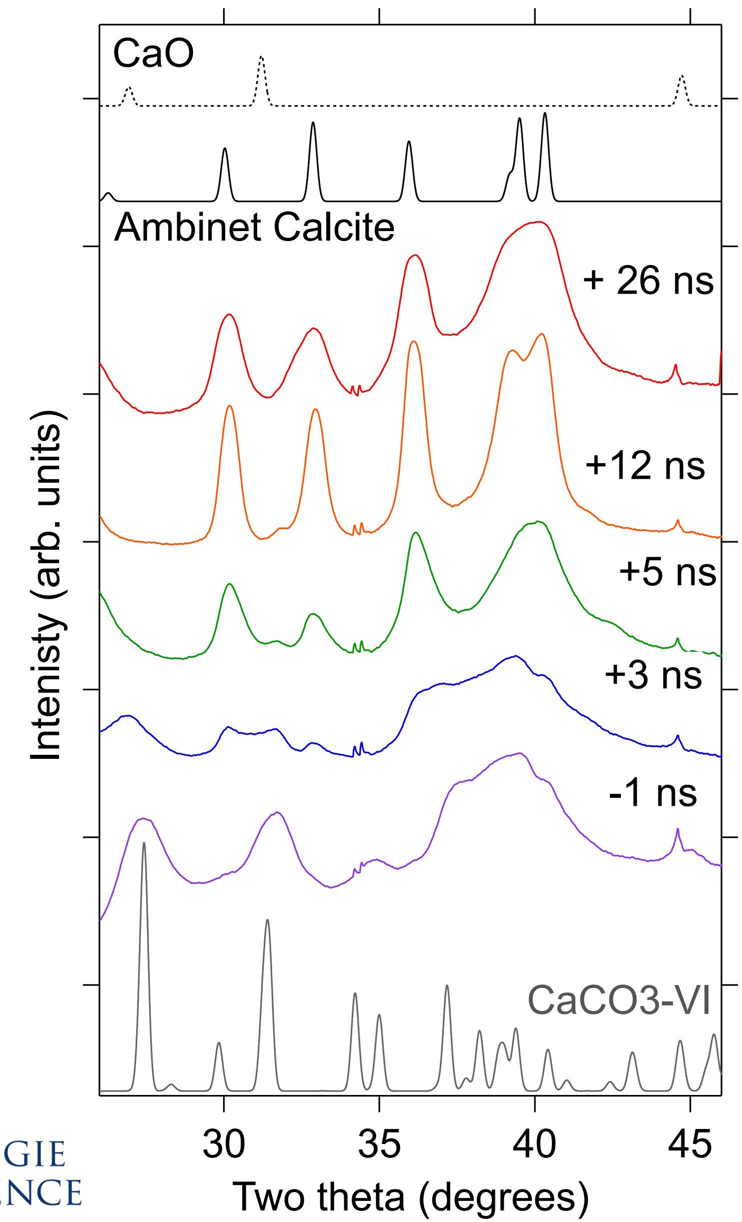


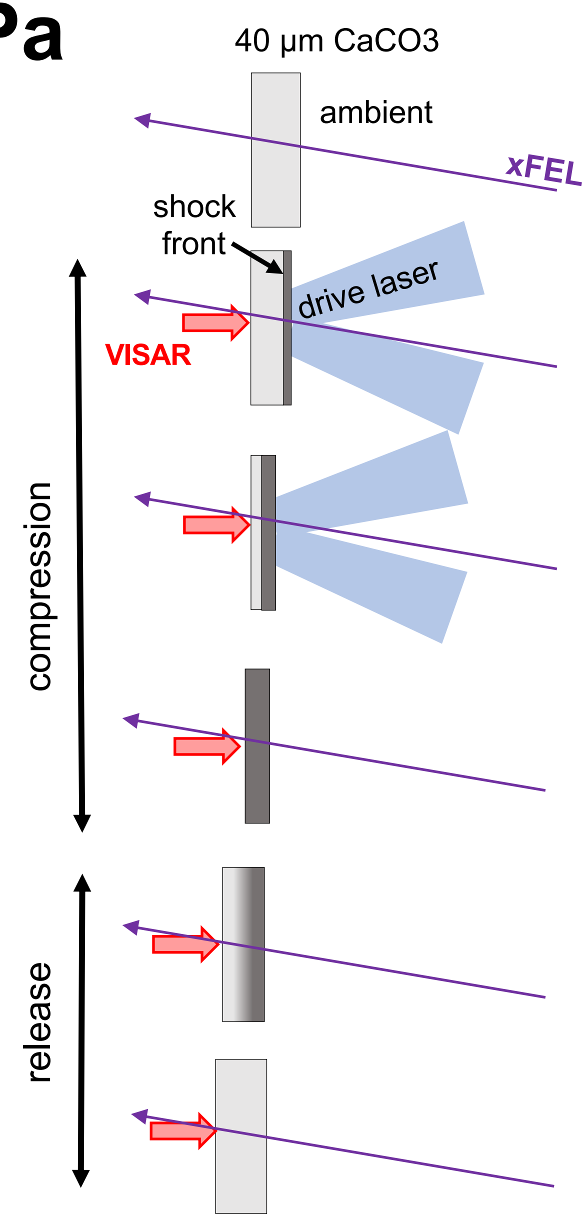
Triclinic calcite-VI



after Bayarjargal et al.

Release time series from 40 GPa





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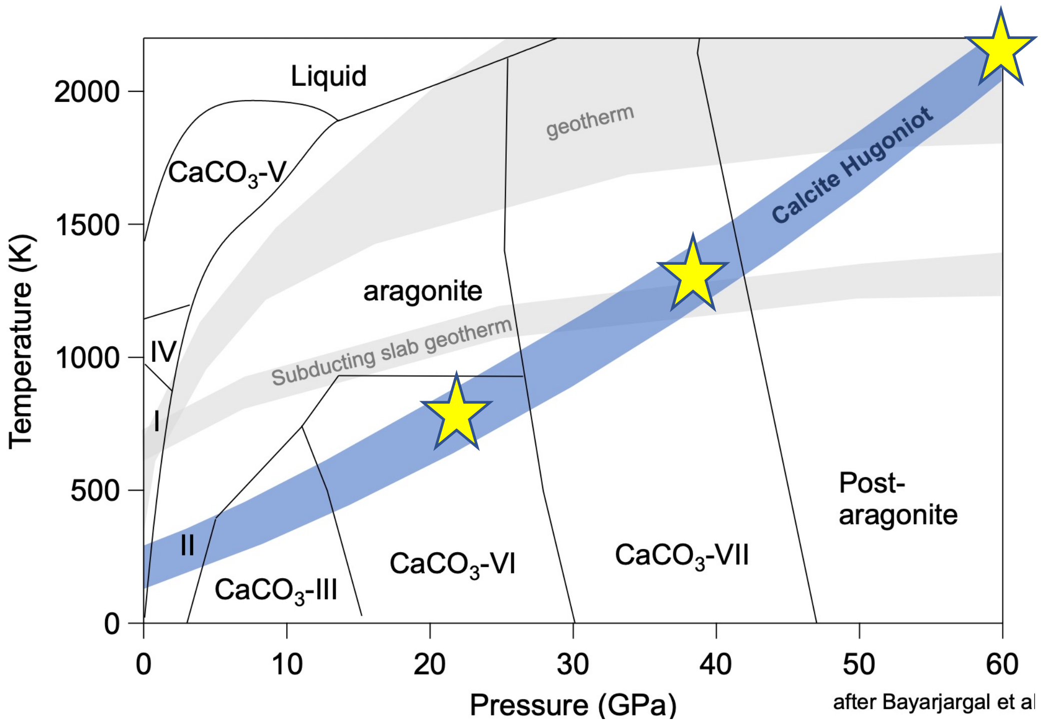
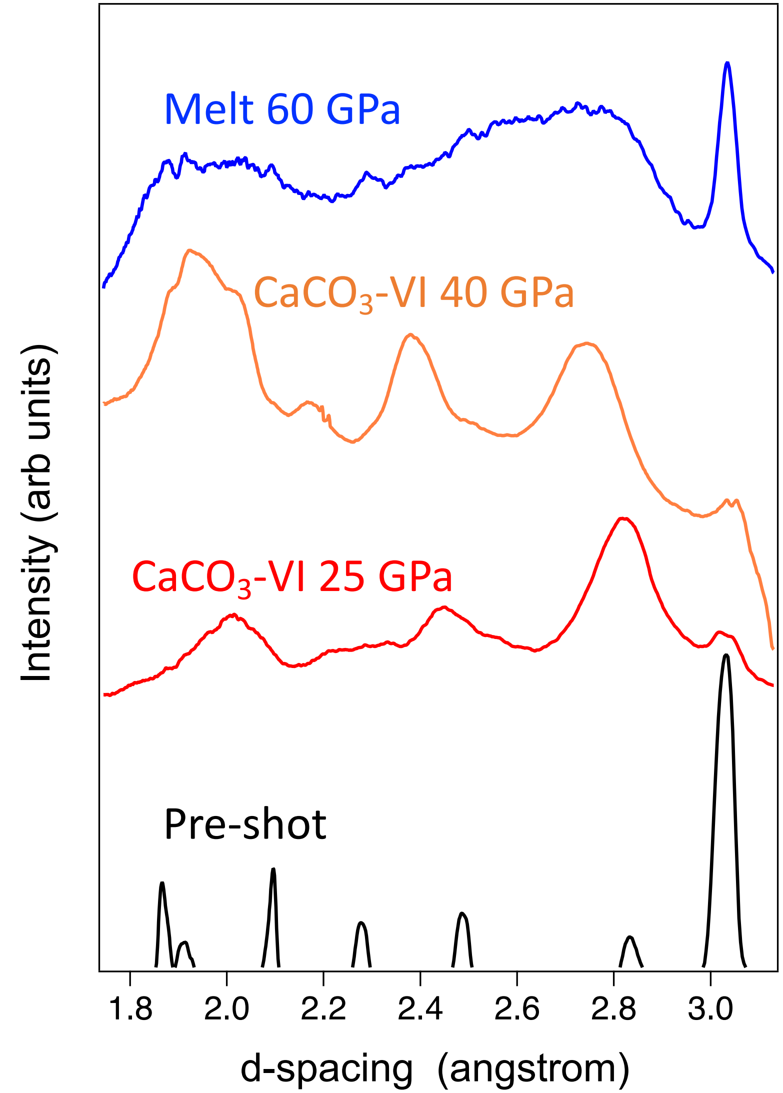
Shock melting at 60 GPa





Future Work:
Quantitative analysis of liquid scattering (14 keV)

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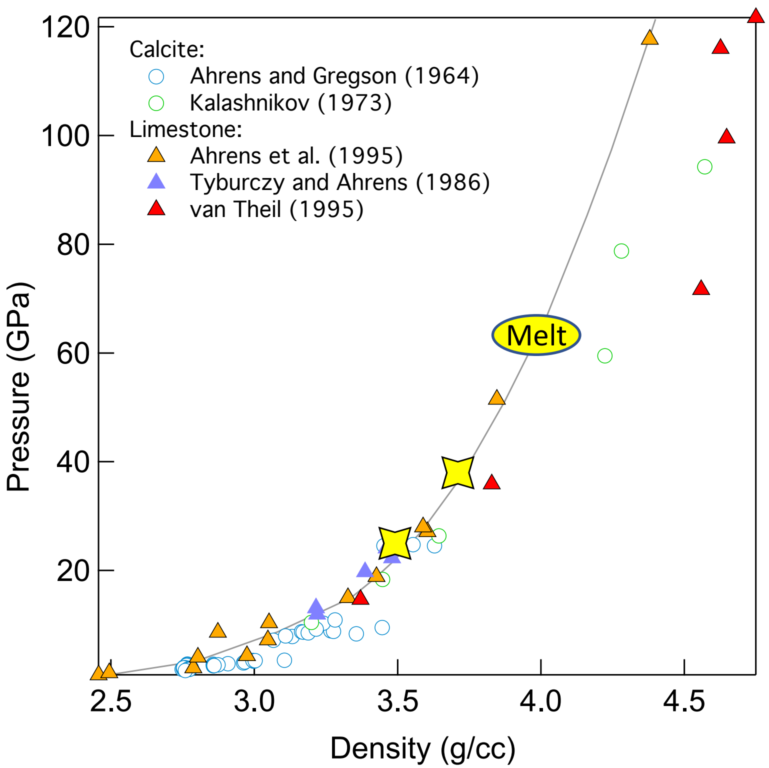
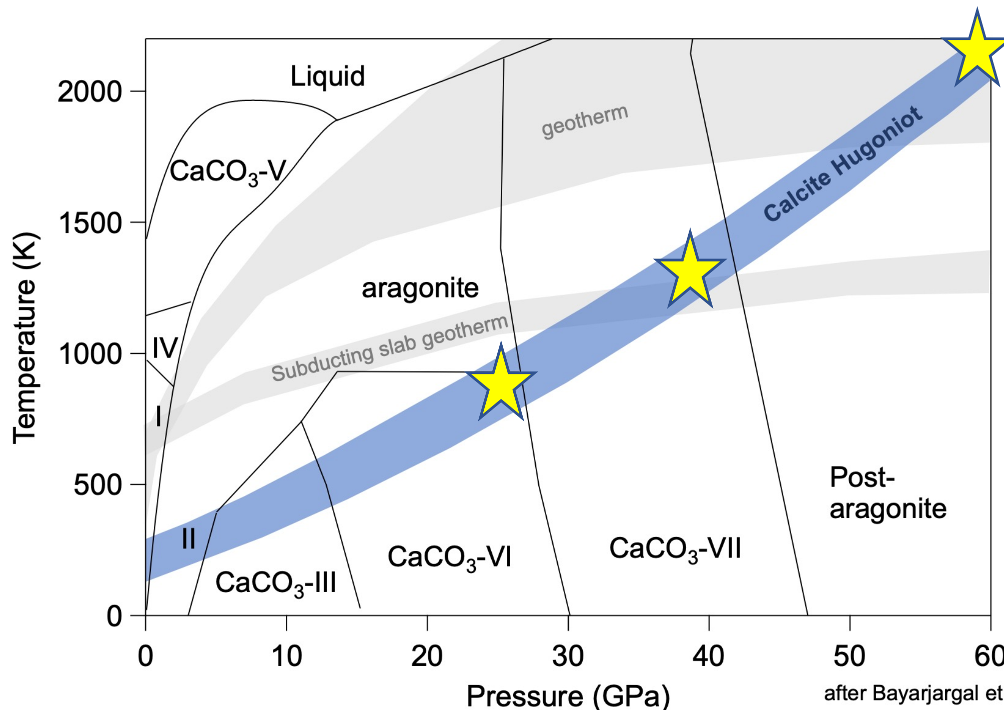
X-ray densities agree with gas-gun data





after Bayarjargal et al.

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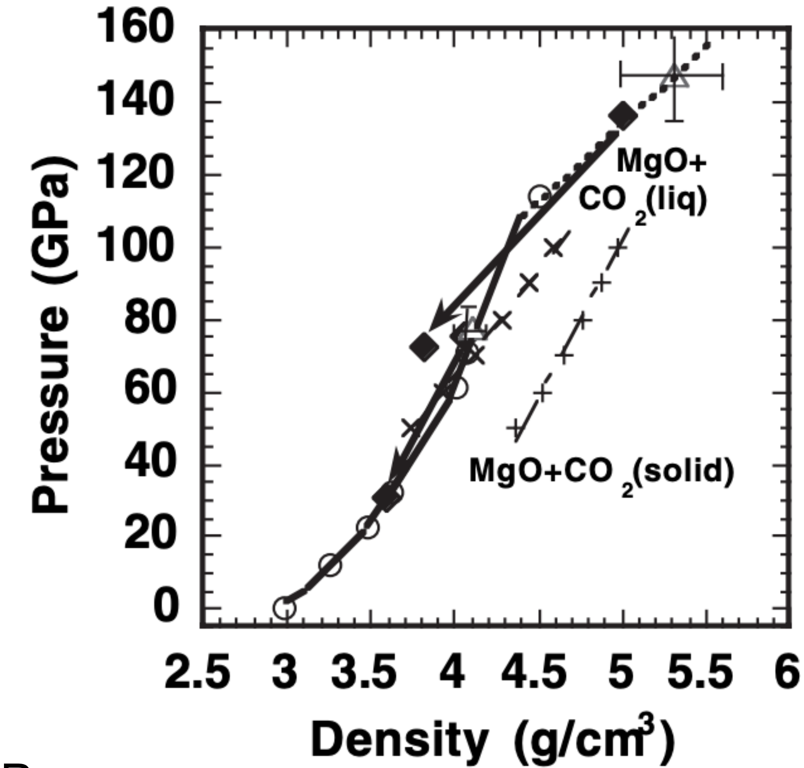
Magnesite - $MgCO_3$

Static:

- Stable up to 100 GPa, above which it undergoes a phase transition to an orthorhombic structure



Sekine et al., 2006

Gas-gun:

- Phase change on the Hugoniot near ~100 GPa
- A volume expansion on release was interpreted in terms of decomposition

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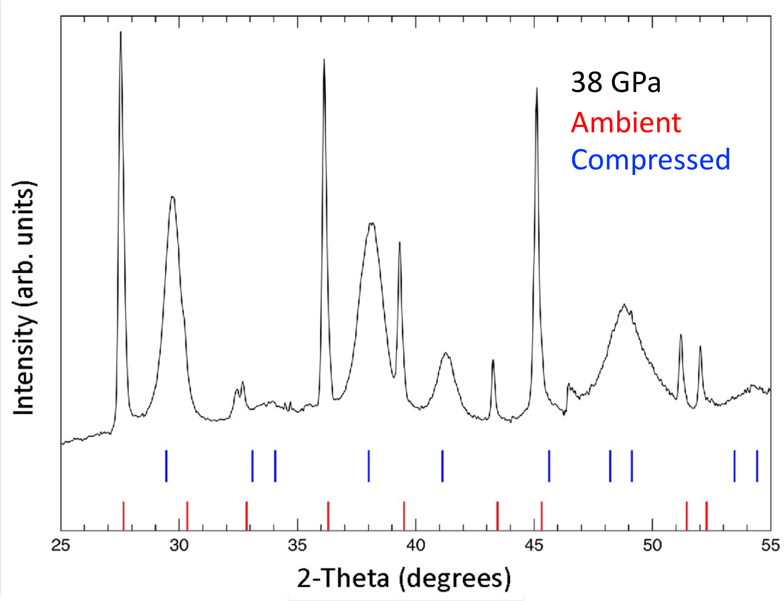
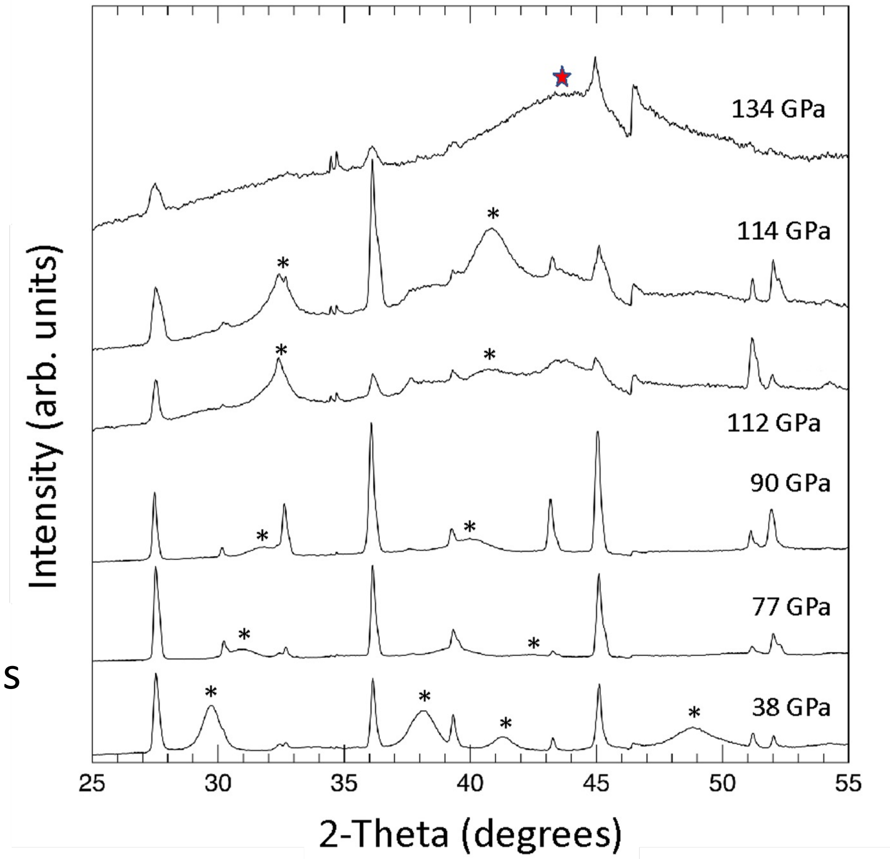
Magnesite-I stable up to melting at >120 GPa





Compression of $MgCO_3$-I with no phase changes up to 120 GPa

Melting above 120 GPa -- calculated shock temperature of 2.5-3K at this pressure

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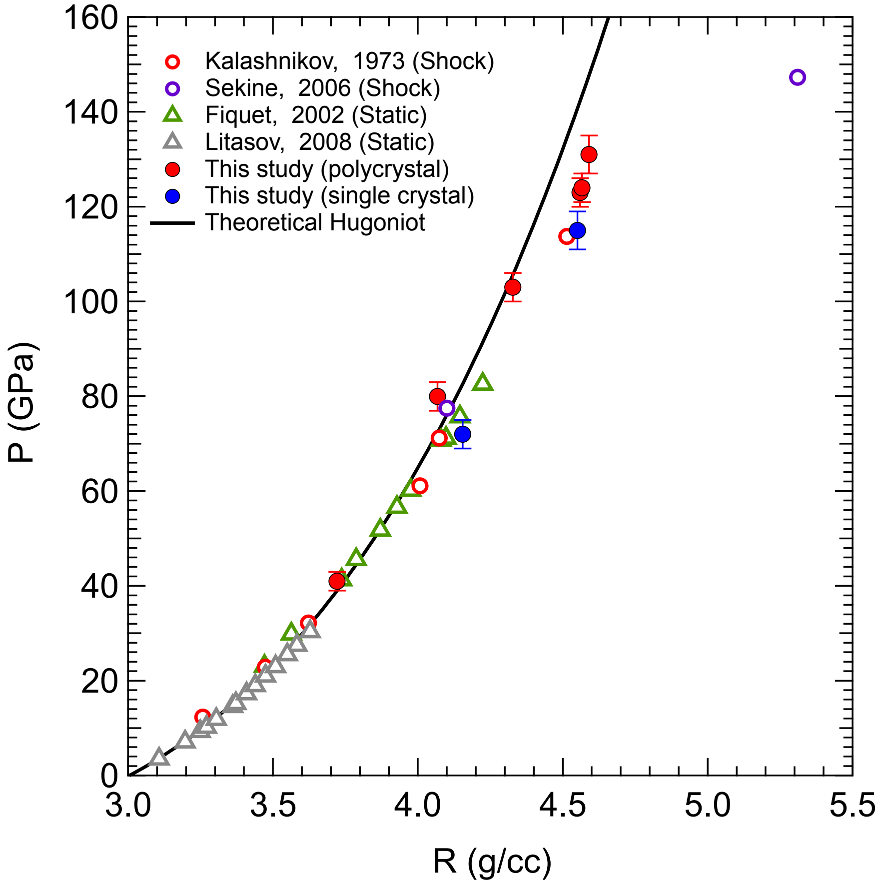
Magnesite Hugoniot

Pressure-density data derived from XRD

Consistent with past gas-gun results

Deviates from calculated Hugoniot based on static results above 90 GPa



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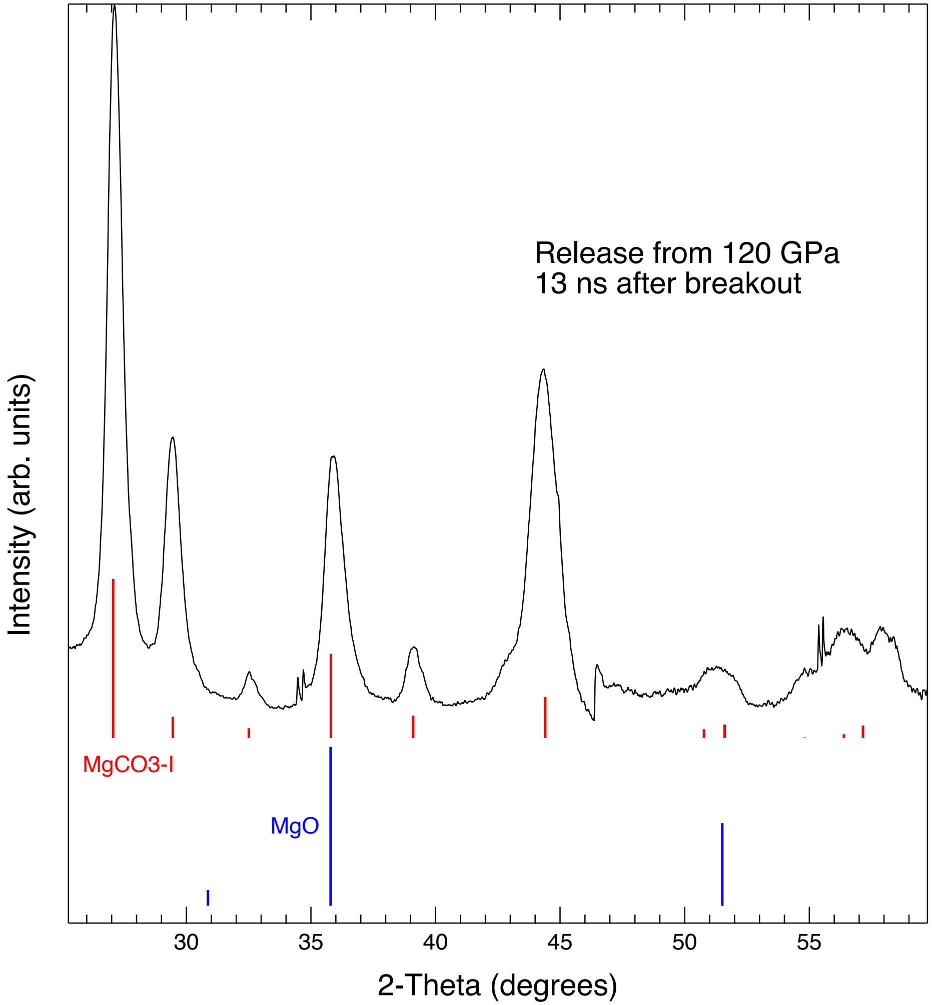
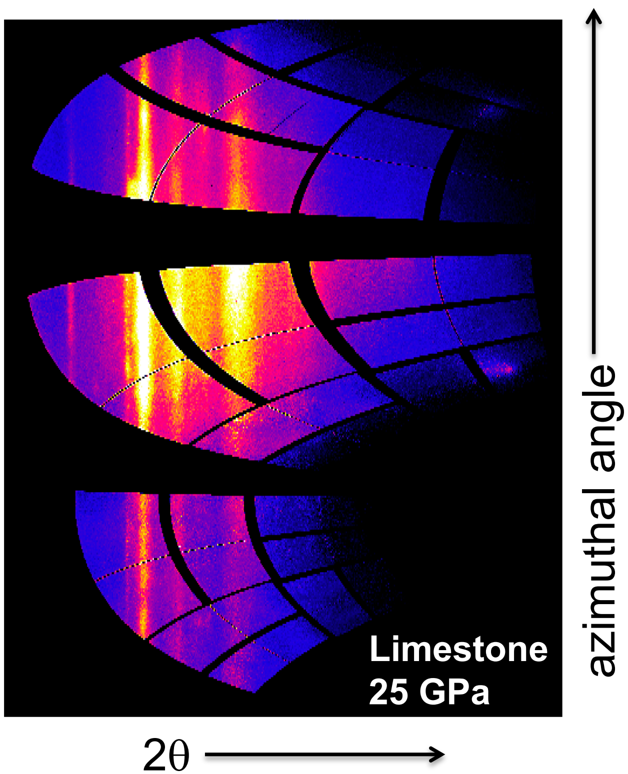
Retention of $MgCO_3$-I structure on release



Carbonate summary



Calcite:
- Crystallographic phase transformation to $CaCO_3$-VI
- Melting on Hugoniot above 60 GPa
- Reversion to $CaCO_3$-I on release with no evidence for devolatilization

Magnesite:
- Stable up to melting on the Hugoniot above 120 GPa
- Retention of $MgCO_3$-I on release

Shock experiments at XFEL:
- Allow us to resolve & differentiate low-symmetry crystal structures
- Provides means of carrying our detailed investigation of release behavior via pump-probe time series

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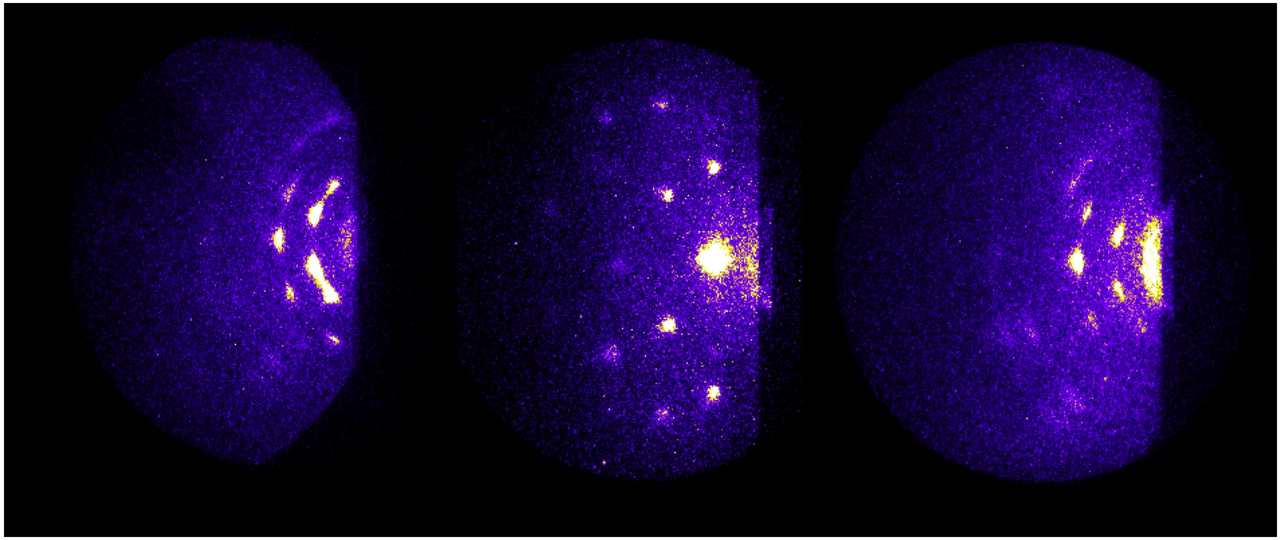
Phase transition in ZnO



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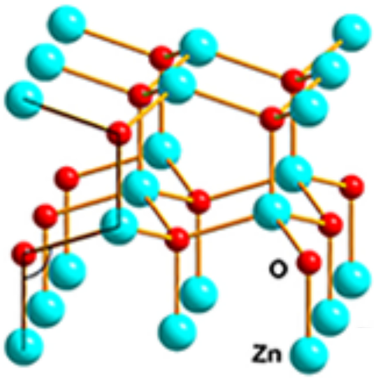
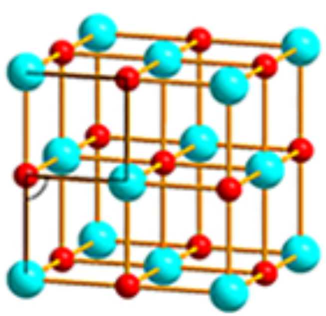
High pressure polymorphism in zinc oxide





- Crystallizes in a 4-coordinated wurtzite structure
- Phase transition a rocksalt phase at moderate pressure (9-16 GPa)
- Transition is common to many wurtzite and zincblende compounds
- Interest in finding routes to quench the ZnO rocksalt phase to ambient conditions due to its favorable optoelectronic properties
- Ultrafast XRD presents a unique capability to study this transformation in real time

Fig: adapted from Wang *et al.* (2018)

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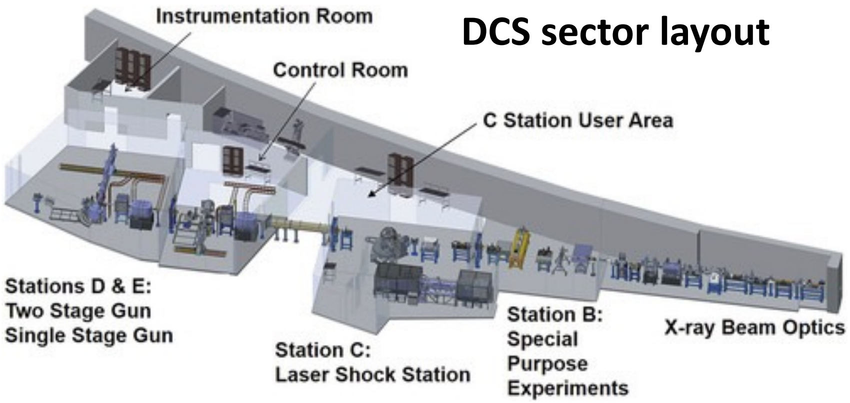
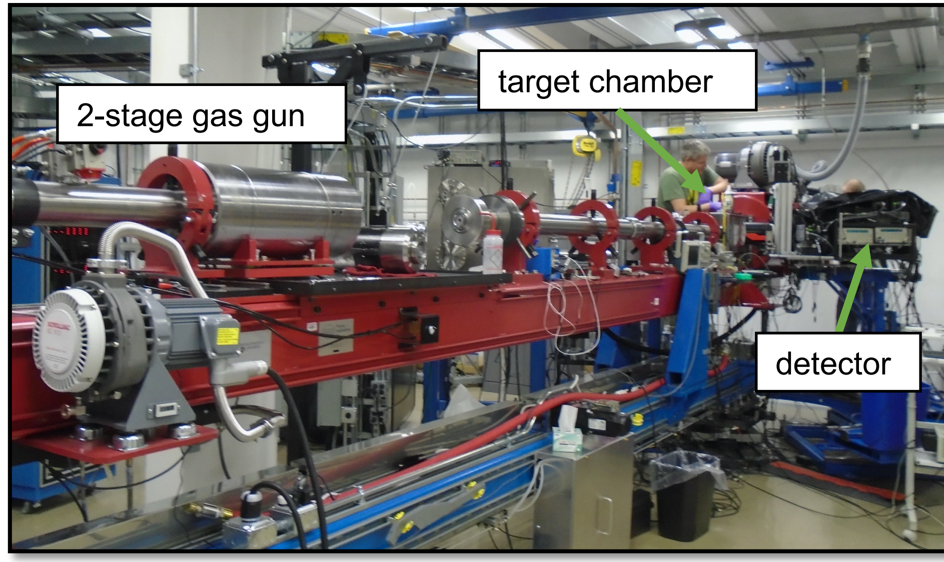
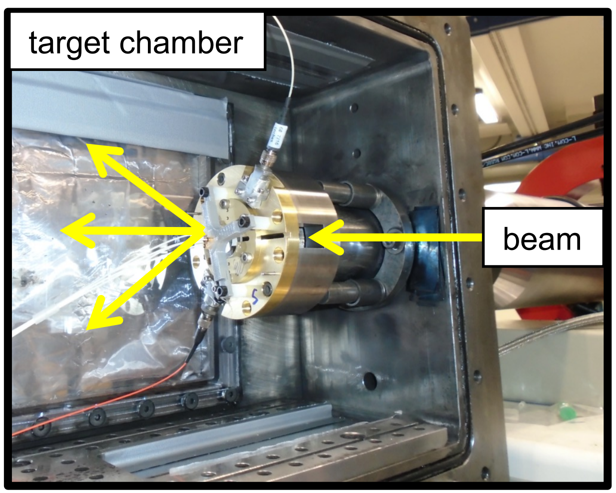
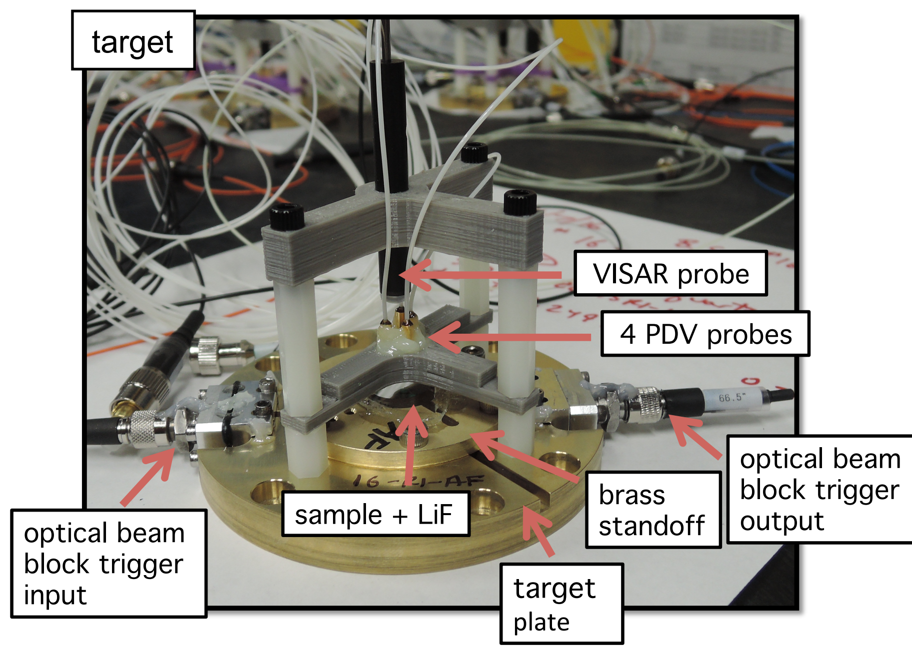
Dynamic Compression Sector (DCS):
Impact launcher at APS









THE Dynamic Compression Sector
AT THE ADVANCED PHOTON SOURCE

Multi-frame XRD at DCS

Take advantage of time structure of the synchrotron to collect a series of XRD frames

24-bunch mode well suited to ~100-ns time scale of gun experiments

At DCS collect four frames during the loading and release process for a given shot

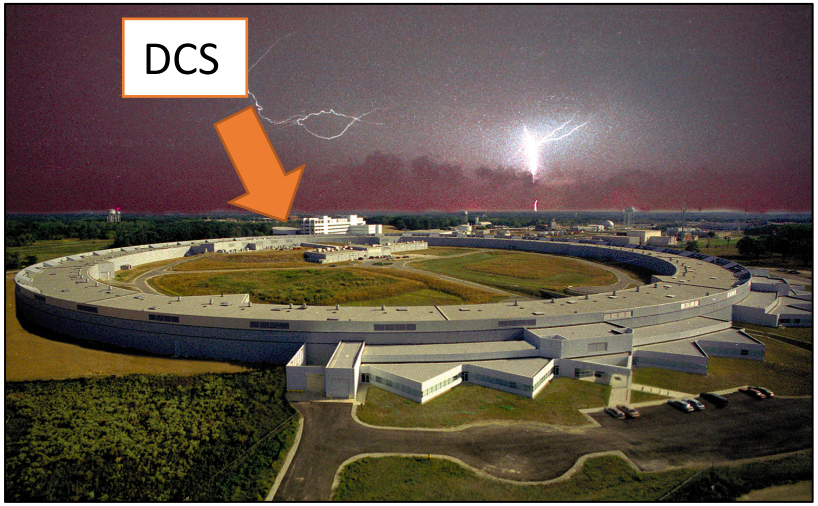
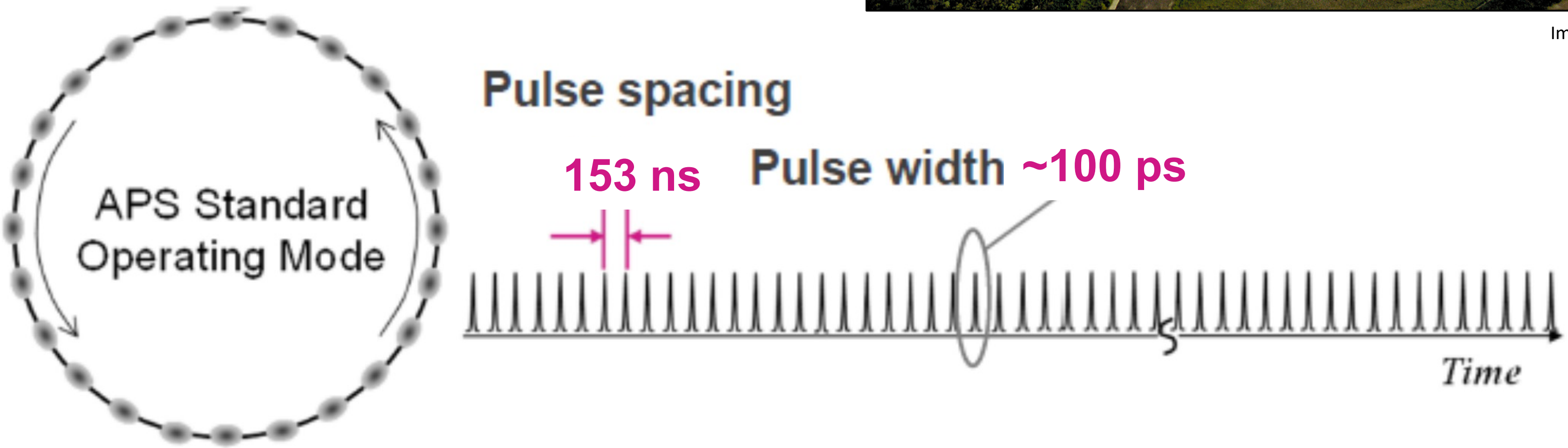


Image: ANL



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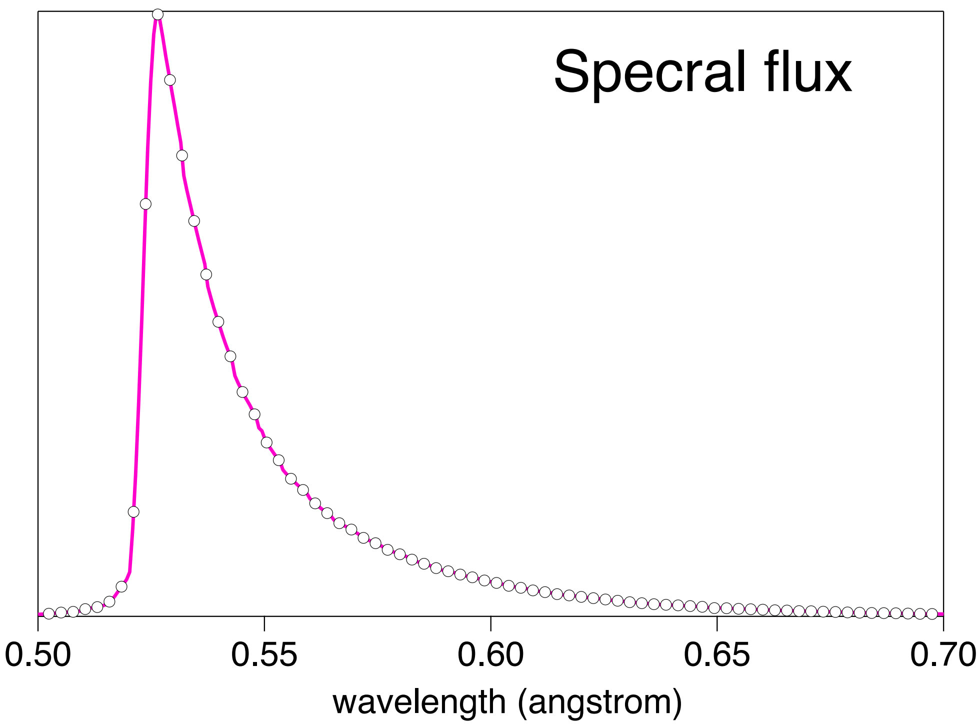
Pink beam X-ray diffraction

Single-pulse XRD experiments utilize pink beam to maximize photons delivered to target

Asymmetric wide-bandwidth spectral flux peaked at 24 keV



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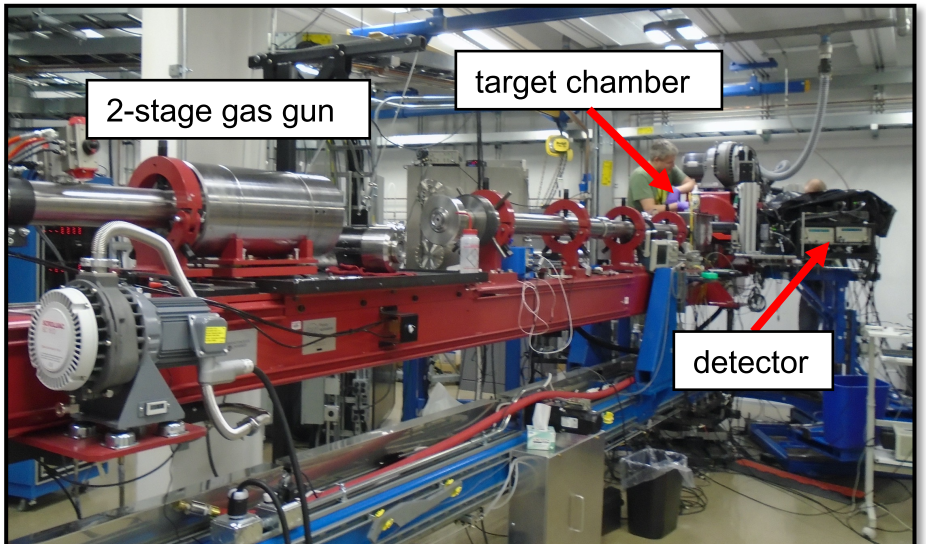
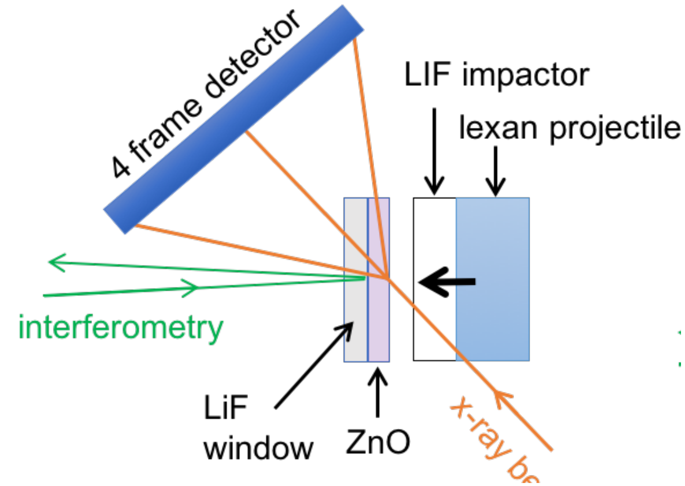
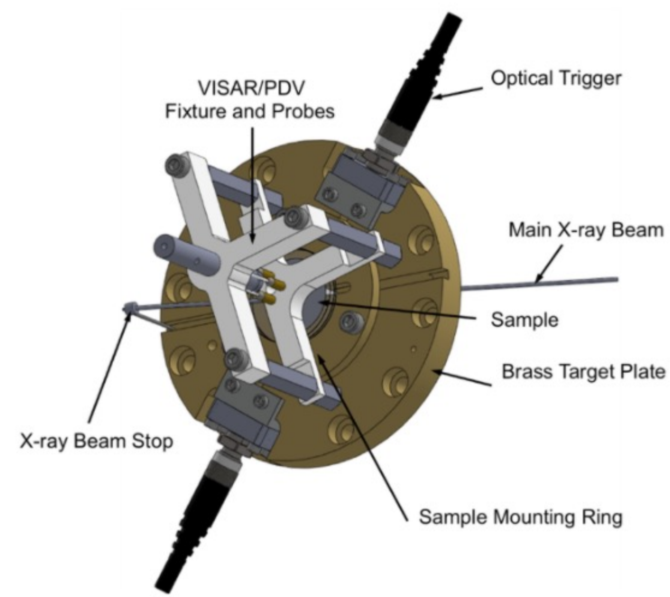
Transmission geometry using two-stage gun







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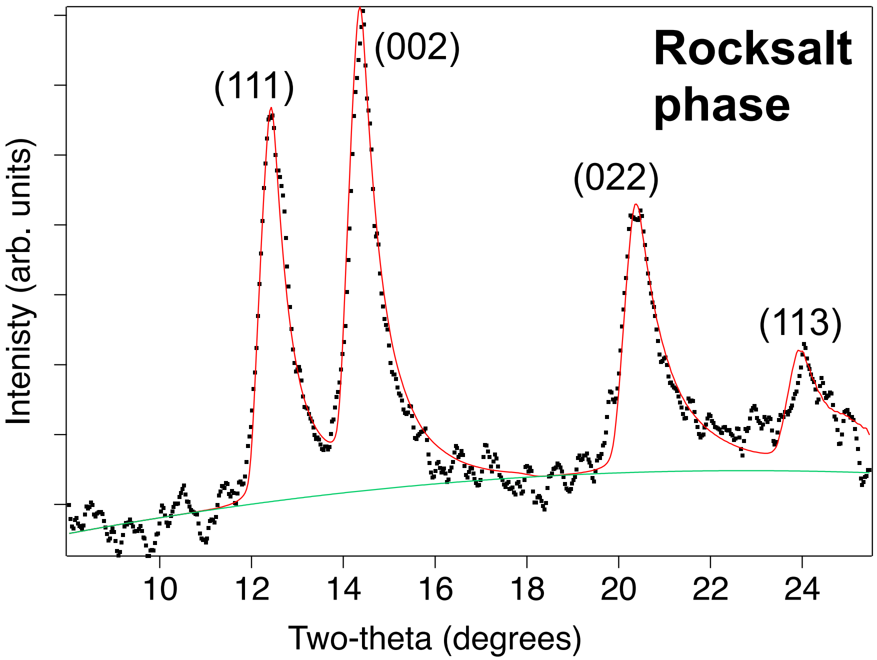
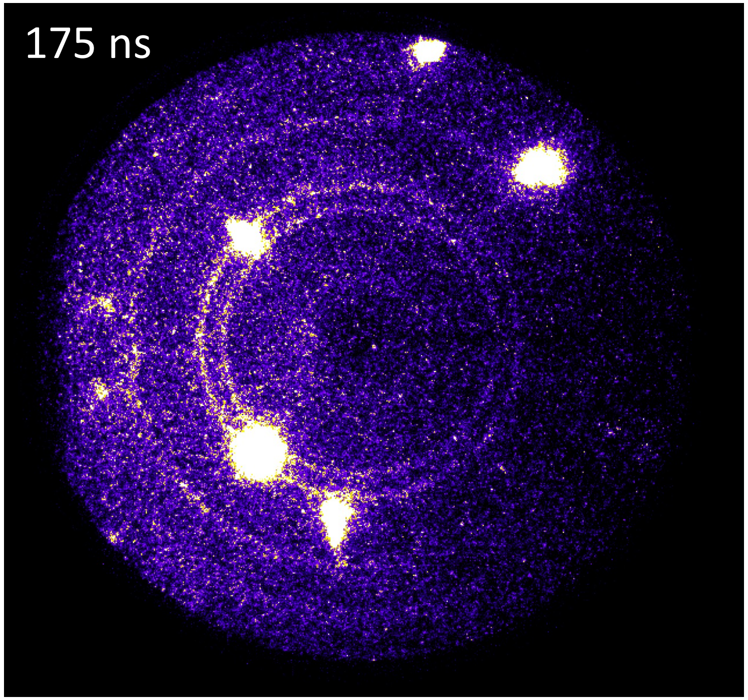
Polycrystalline material 20 GPa





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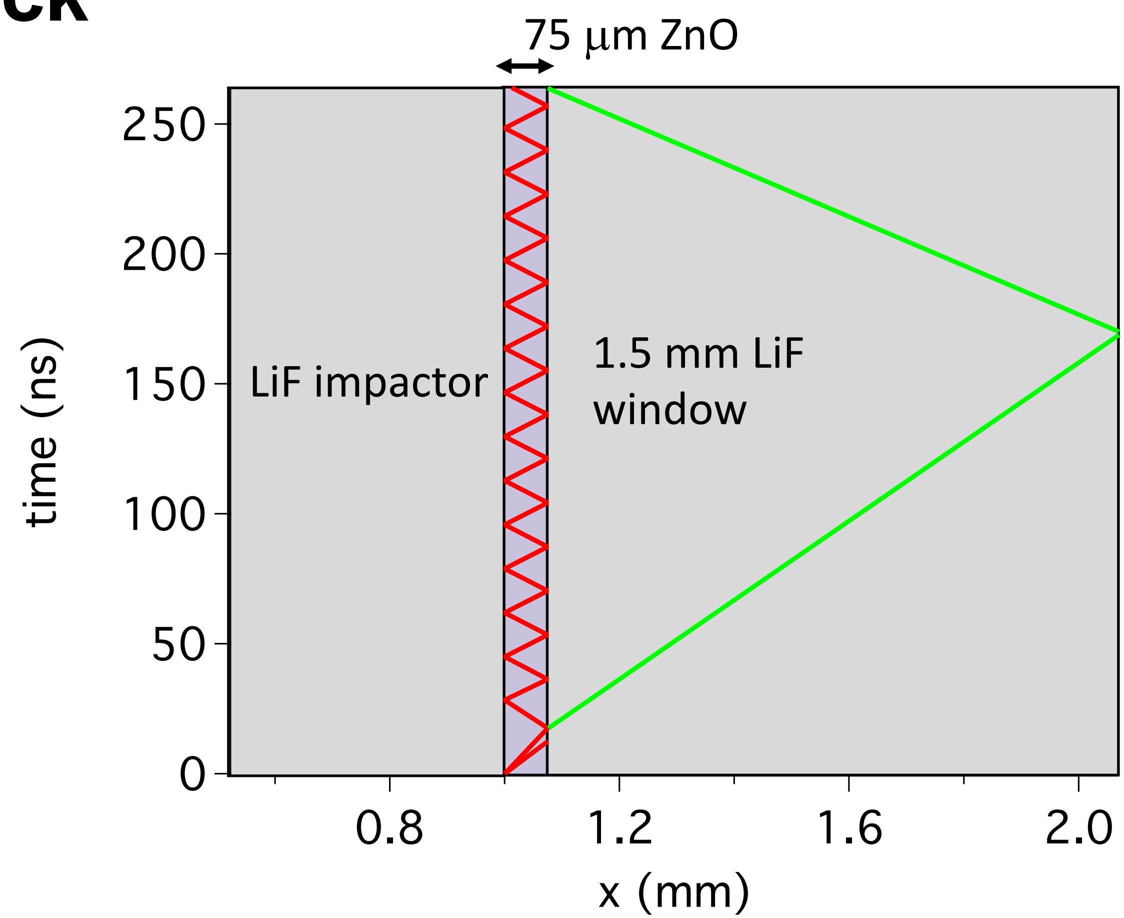
Reverberating shock

Absorption of ZnO:

Thin samples ~75 μm

Transit time ~20 ns

Caveat: Can't control x-ray probe time relative to impact to within 153 ns → can't ensure single shock state



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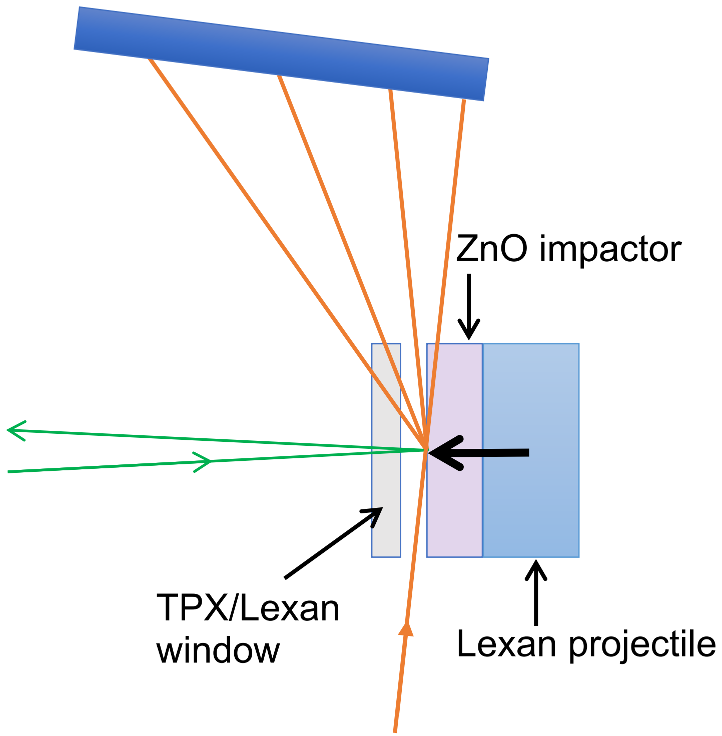
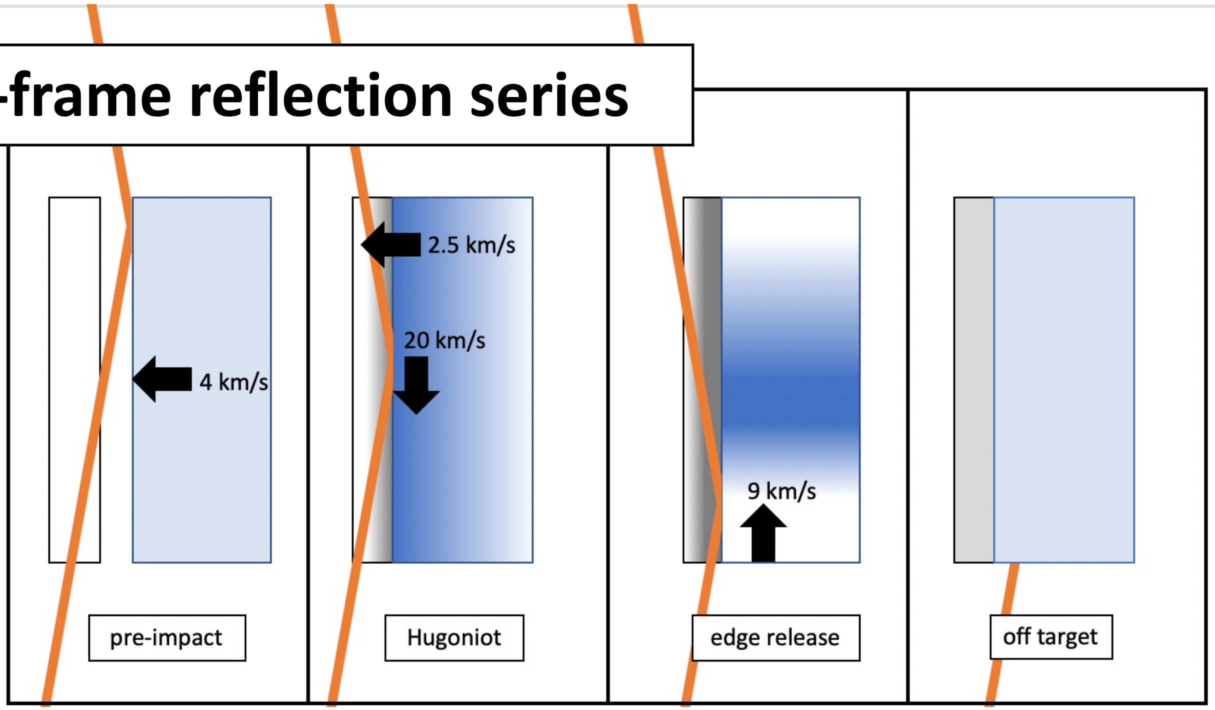
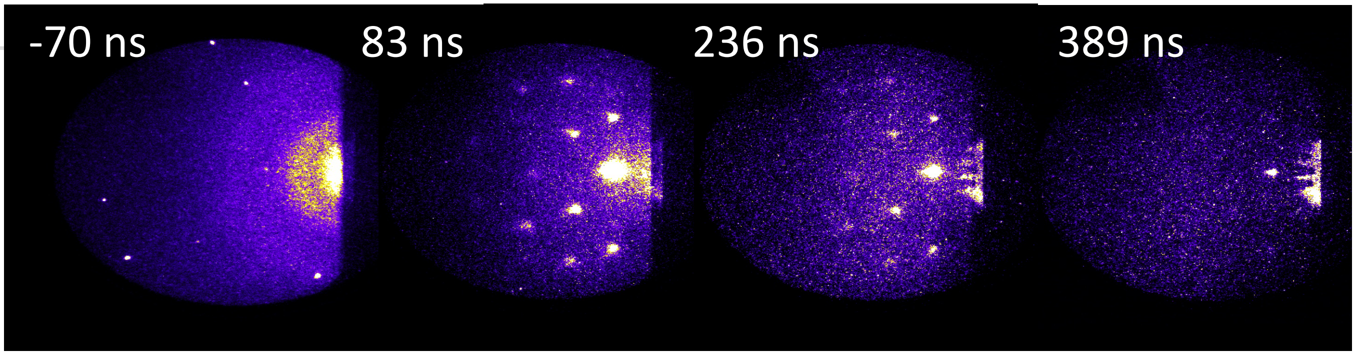
Single crystal shots in reflection geometry

- Significantly absorbing samples require reflection geometry
- Front surface impact shots: ZnO mounted in Lexan projectile and used as impactor
- Impact TPX/Lexan window
- To optimize 2-theta coverage and reduce low-angle cut off from sample absorption beam comes in at grazing angle 7°



4-frame reflection series





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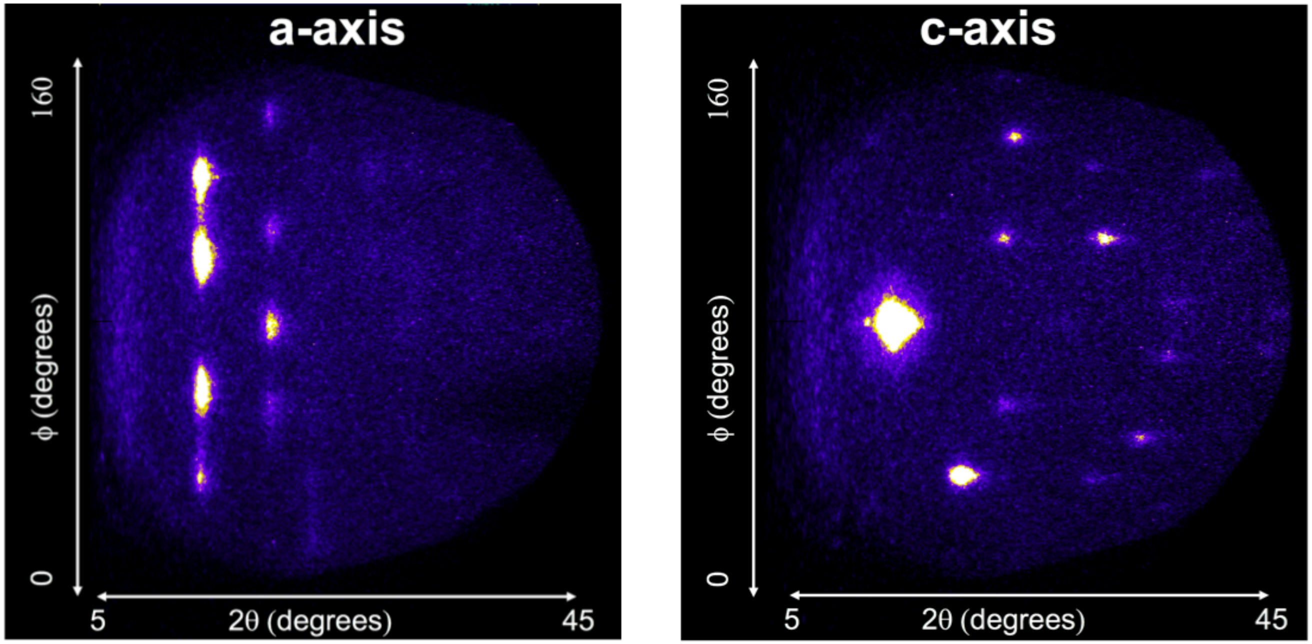
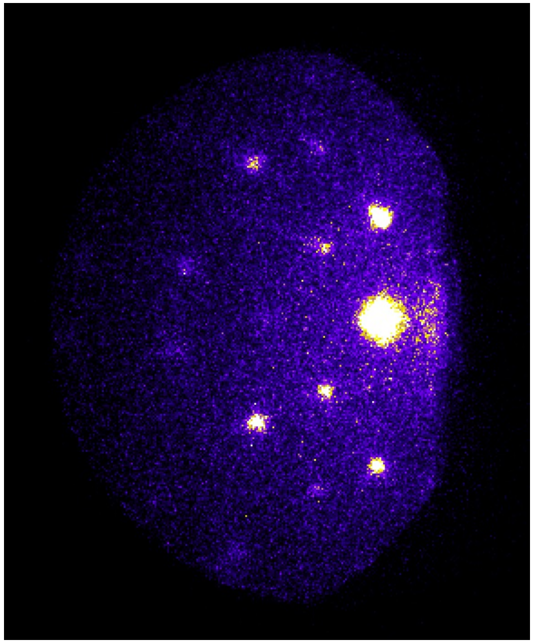
Oriented single crystals 20 GPa

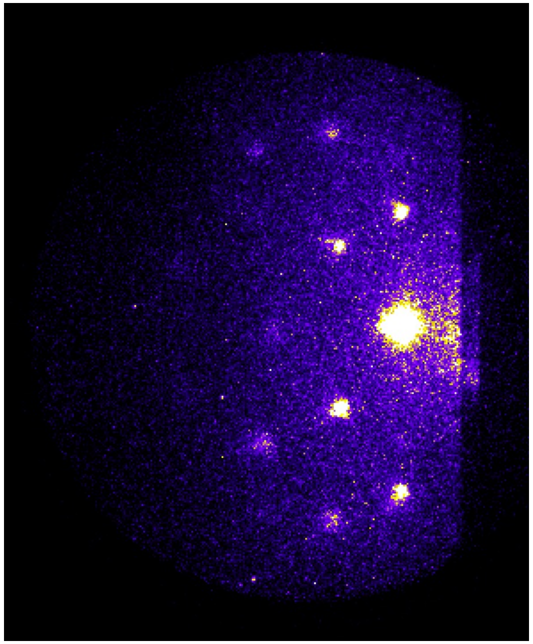


- Large crystallites preserved through the transformation
- Transformation to rocksalt phase with high degree texture

Reproducible transformation textures



ZnO (001)→ TPX



ZnO (001)→ Lexan

Reproducible pattern in terms of where we see the textured diffraction spots

Suggests we capture a reproducible transformation within the time scale of our measurements

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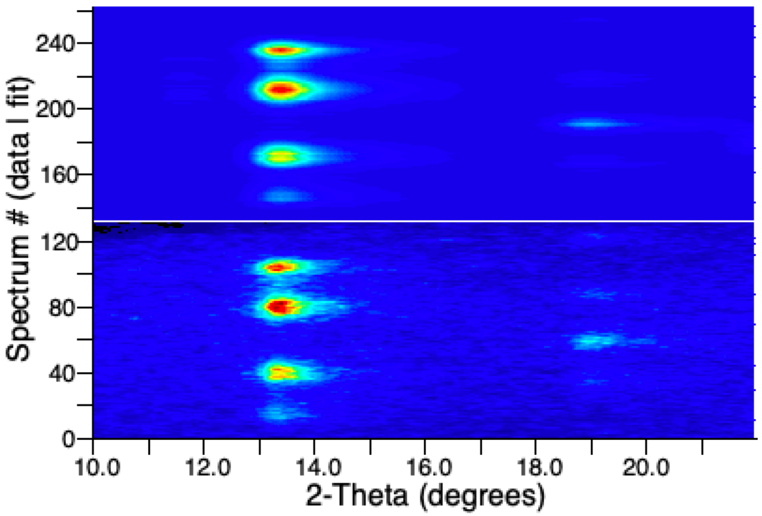
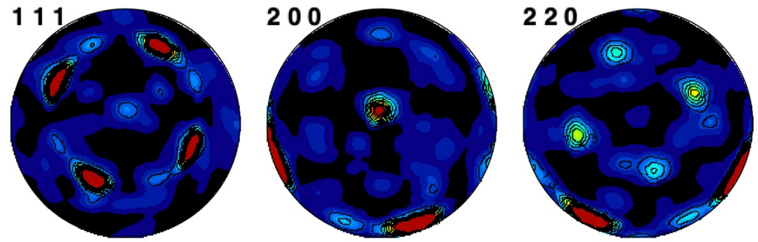
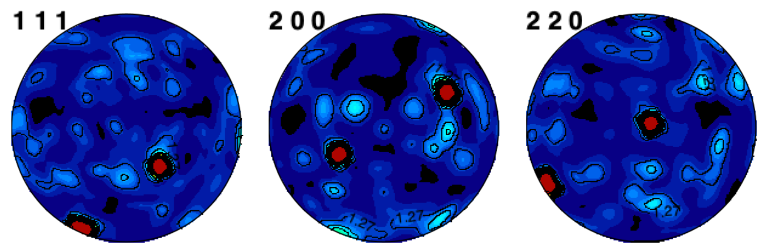
Texture analysis → orientation relations



C-axes loading:



A-axes loading:



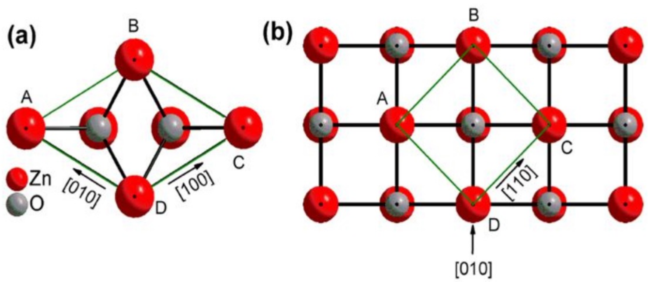


Fig: Tan et al. 2016.

Top views of wurtzite phase (**a**) and rocksalt phase (**b**) crystal structures.

RS (200) ~ wurtzite c-axes
RS (220) ~ wurtzite a-axes

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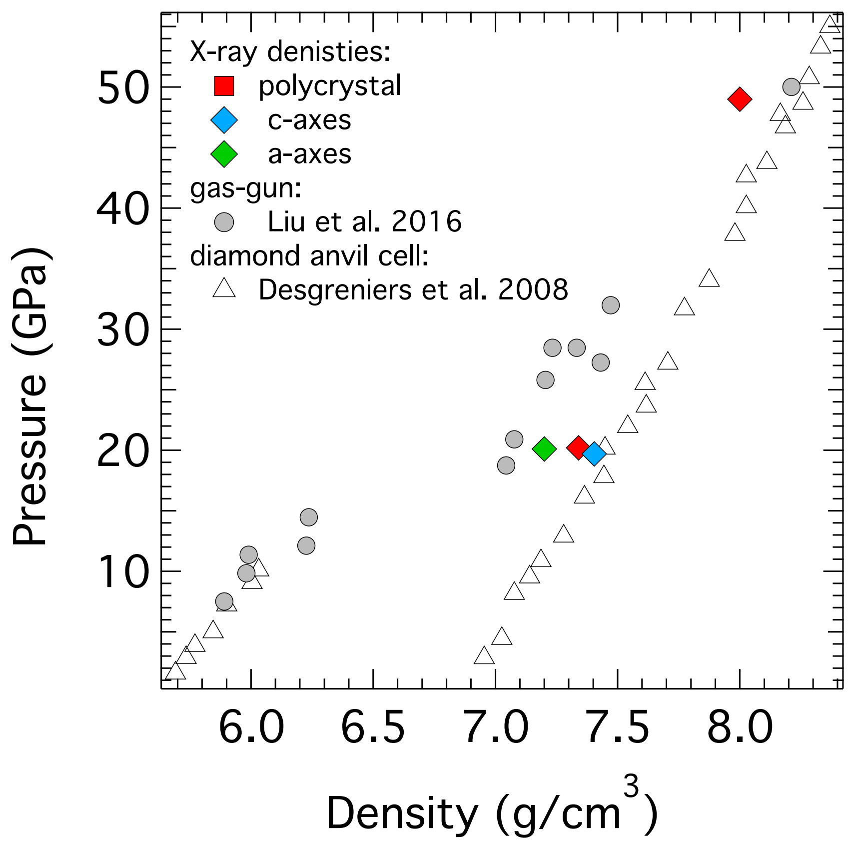
ZnO conclusions

In situ x-ray diffraction allows for crystallographic verification of the phase transition from WZ→ RS under shock compression

Single crystals show reproducible transformation textures with strong preferred orientation in transformed rocksalt phase



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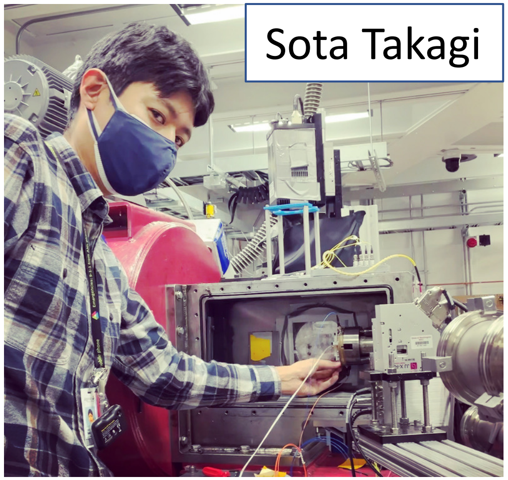
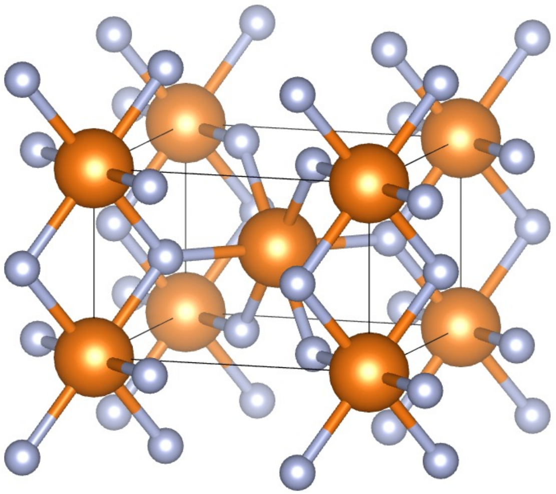
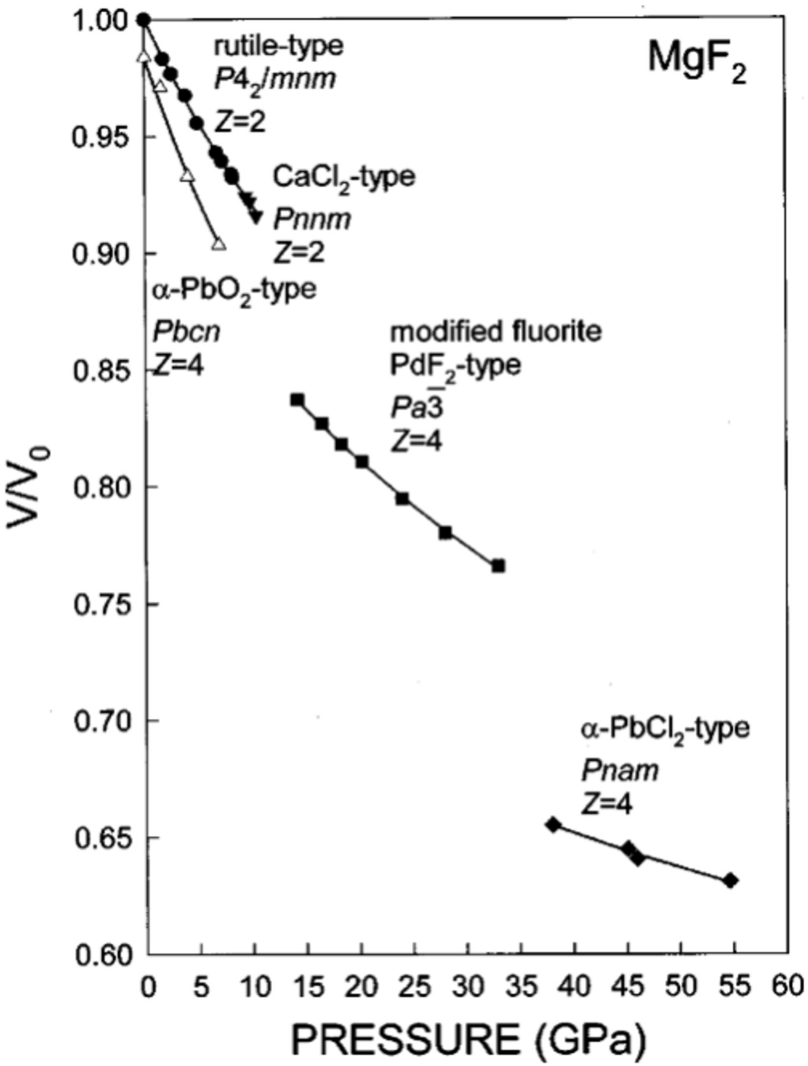
Phase transitions in MgF_2





Static: rutile→$CaCl_2$→ PdF_2

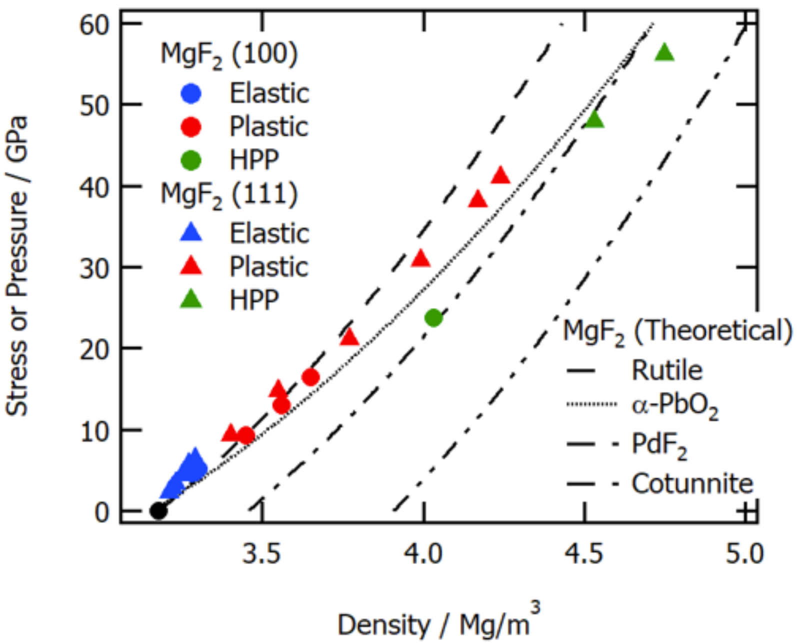


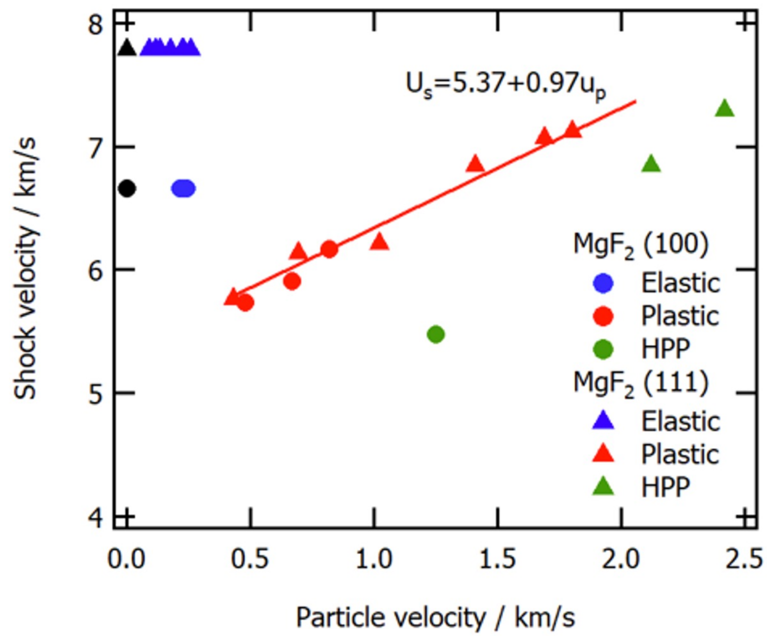
Haines, 2001

Carnegie Science

Phase transitions in MgF_2 under shock loading

Gun shock data collected at Kumamoto University:

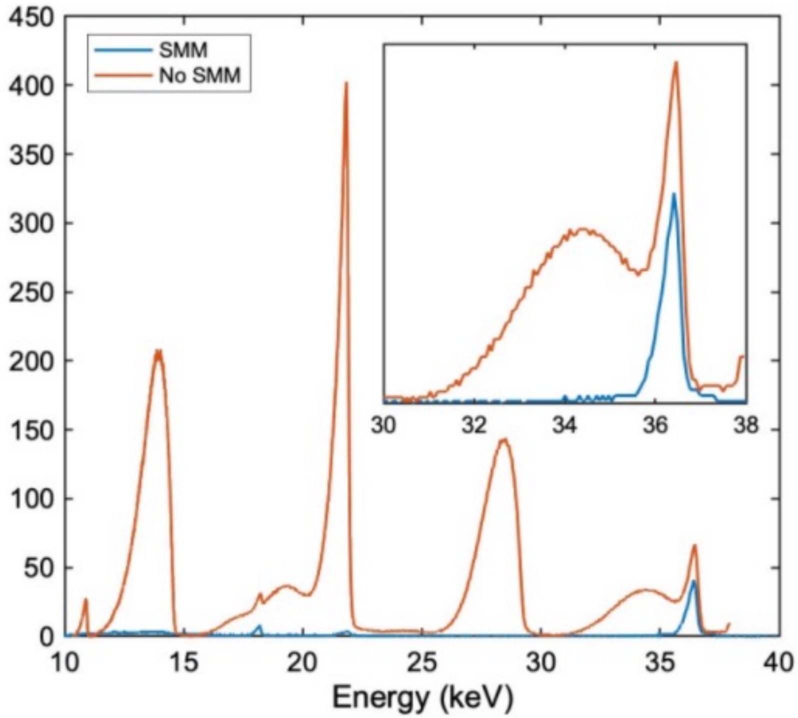


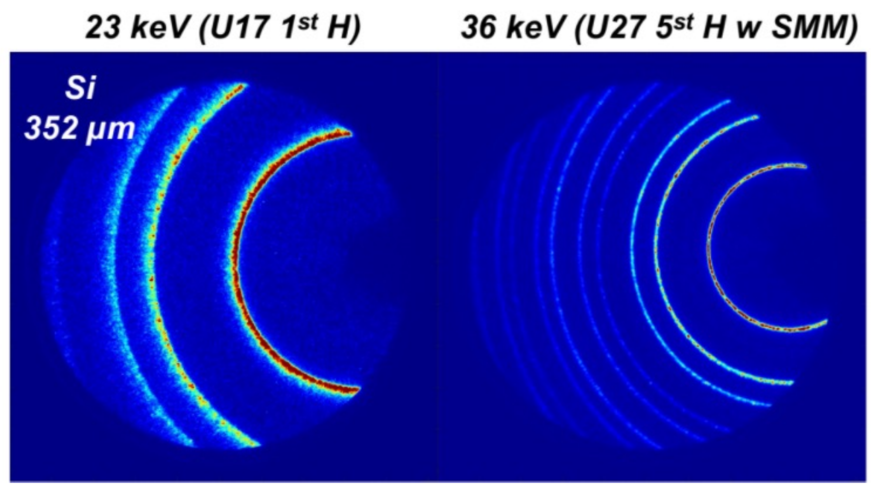


Questions:
What is structure of high-pressure phase?
What structure does the high-pressure phase revert to on release?

36 keV – Single Multilayer Monochromator

Isolate 5th harmonic of U27 (36 keV peak intensity)



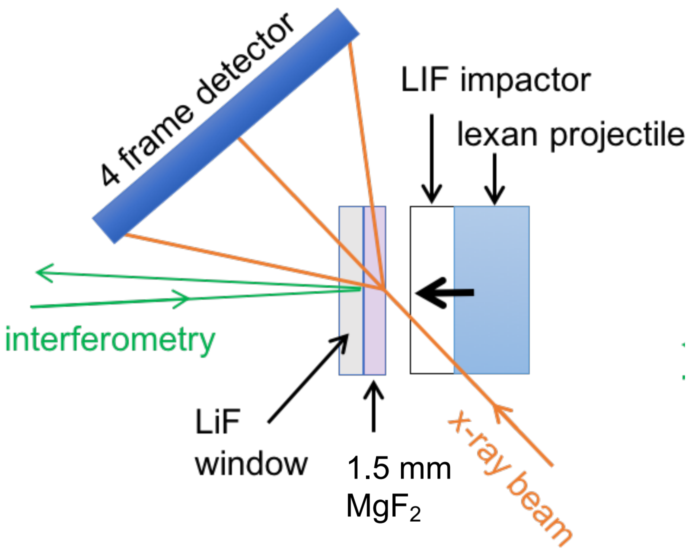


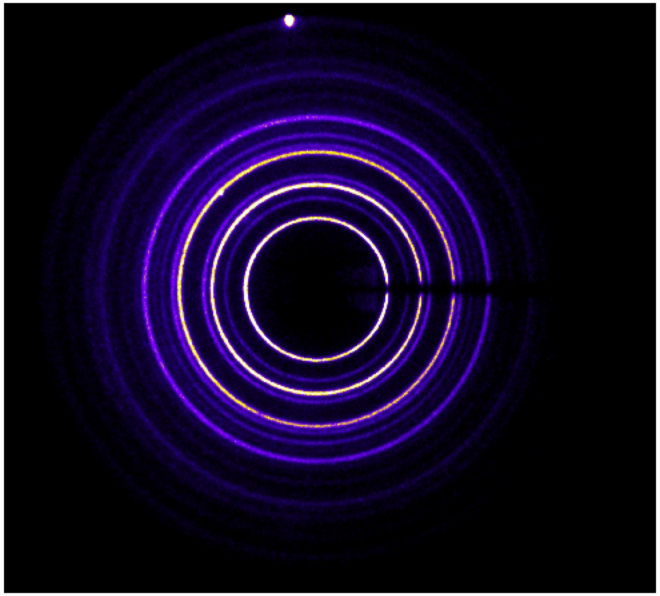
- High resolution diffraction peaks in single pulse
- Increased 2θ coverage to determine crystal structure
- High data quality for thick samples & high-Z materials

Carnegie Science

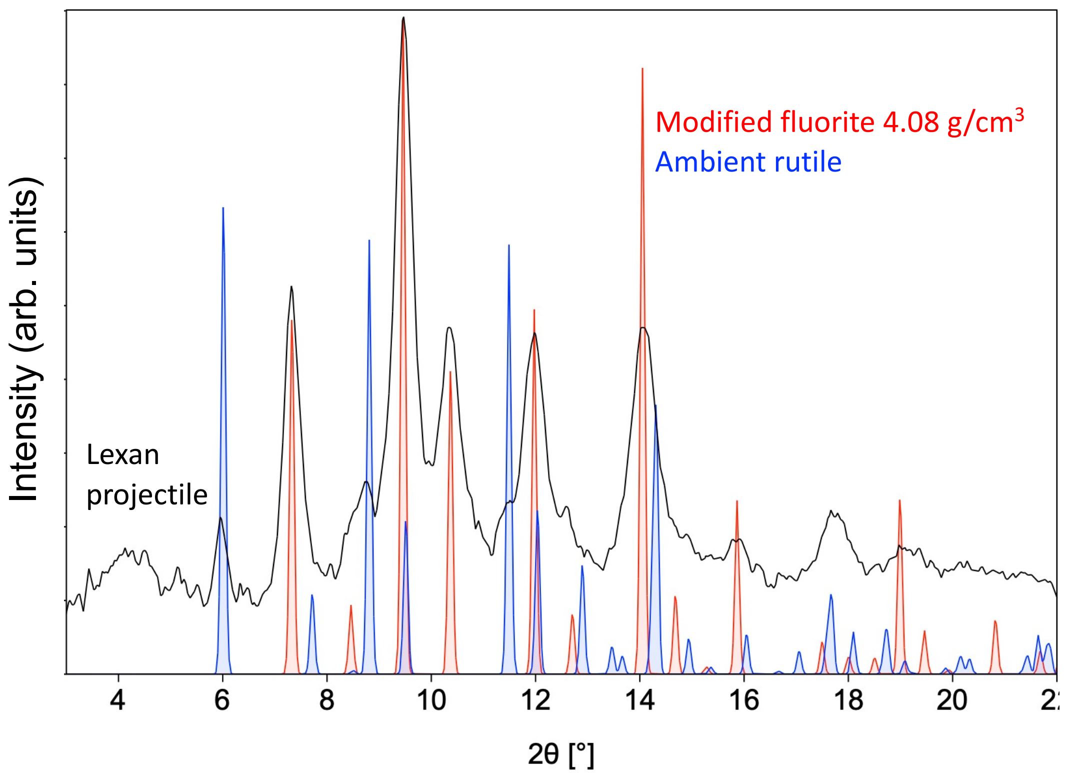
Two-stage gas gun shots at DCS

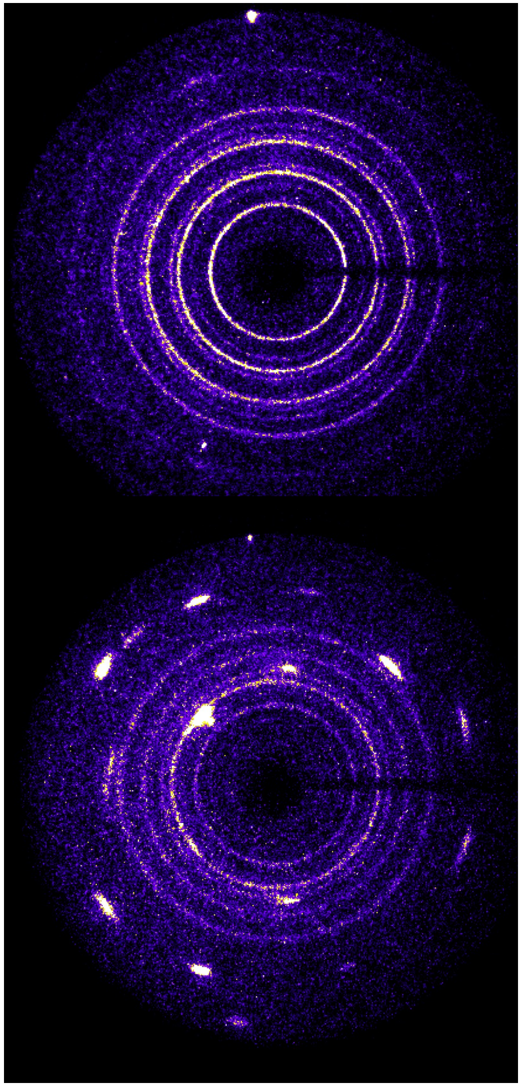
- Transmission geometry
- Sintered polycrystalline samples (94% density)
- ~1.5-mm thick MgF_2 samples with 0.7-mm LiF window
- 120-mm scintillator



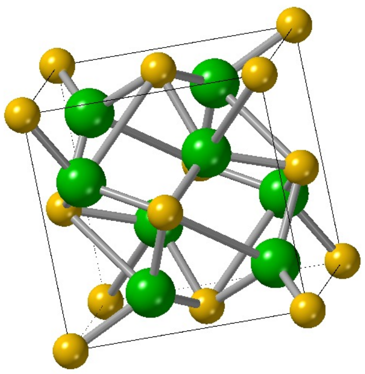


40 GPa – Modified fluorite phase

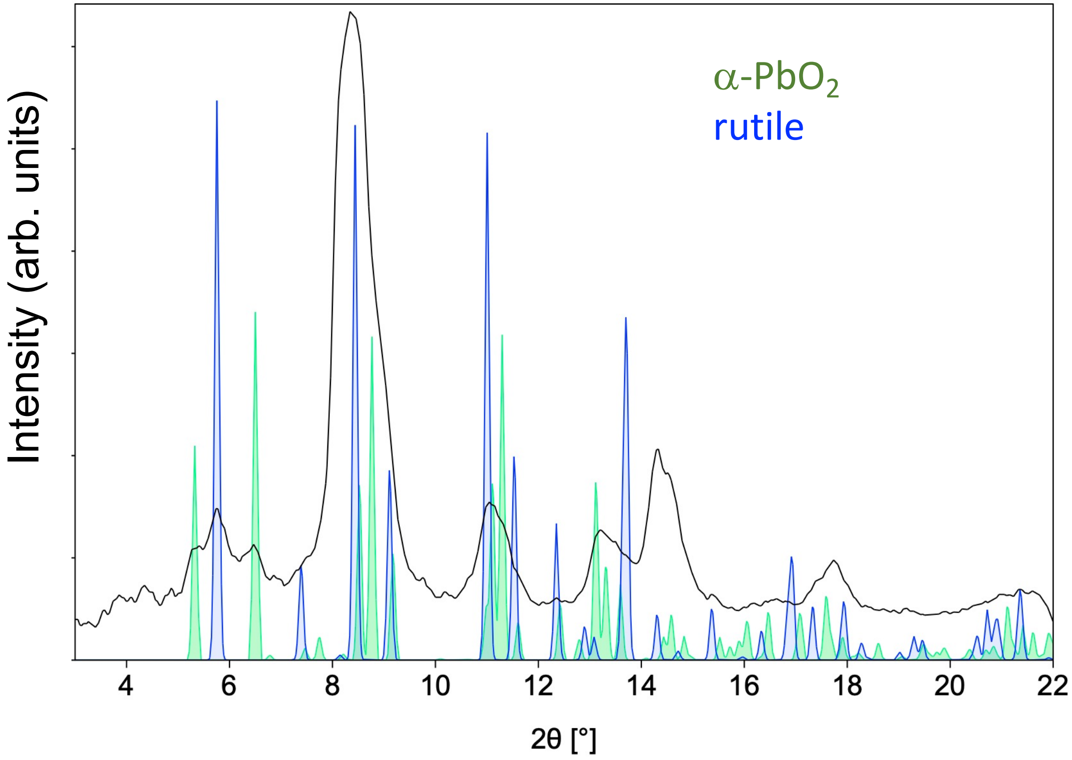


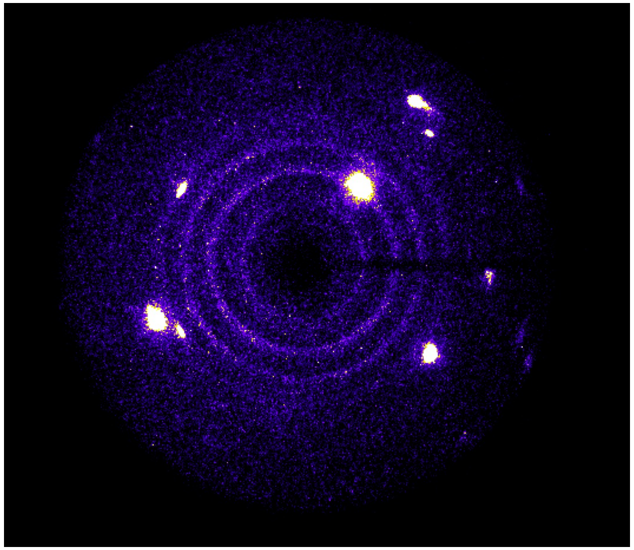


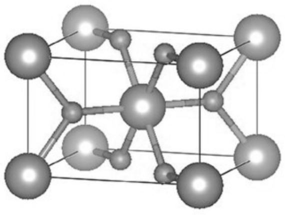
Transition to 6+2 coordinated modified fluorite phase



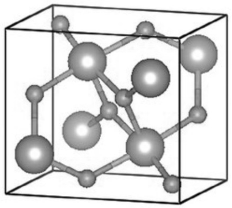
Release and reversion to α-PbO2 + rutile







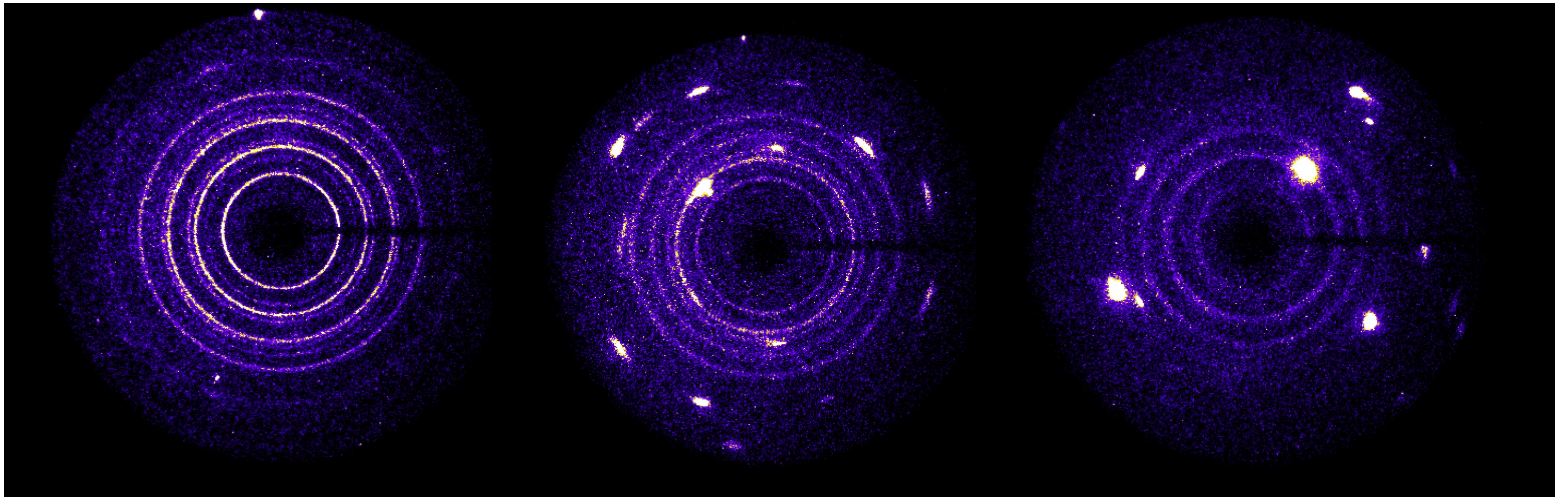
rutile



α-PbO$_2$

MgF_2 Conclusions

- *In situ* XRD allows for crystallographic verification of the phase transition to modified fluorite phase under plate-impact shock loading
- Reversion to mixture of α-PbO_2 and rutile phase on release
- Demonstration of new capabilities for 36 keV using SMM



CARNEGIE SCIENCE

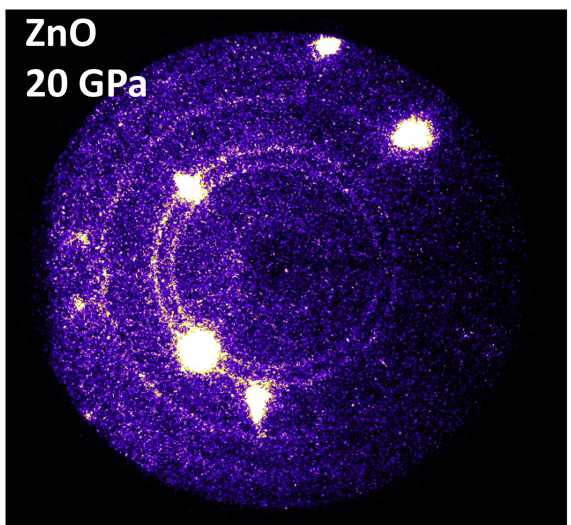
Summary & Conclusions

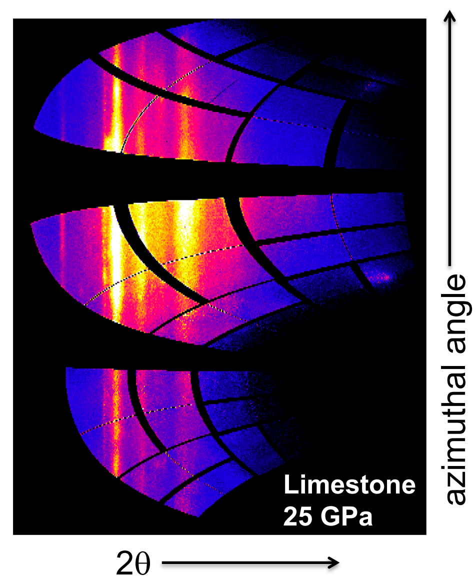
- Identification of phases that form & melting under shock loading for shock loading with both laser-driven and plate-impact drivers

- Ability to resolve low-symmetry crystal structures using single-shot XRD

- New insights into kinetics, metastability, and transformation mechanisms

Outlook: Higher energy X-rays as well as advancements in detectors & monochromators promise improved capabilities to study a broader range of materials including low & high-Z materials and liquid structures





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