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Dynamic compression induced phase transitions of ZrW_2O_8, $(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$, and $(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})_2O_3$

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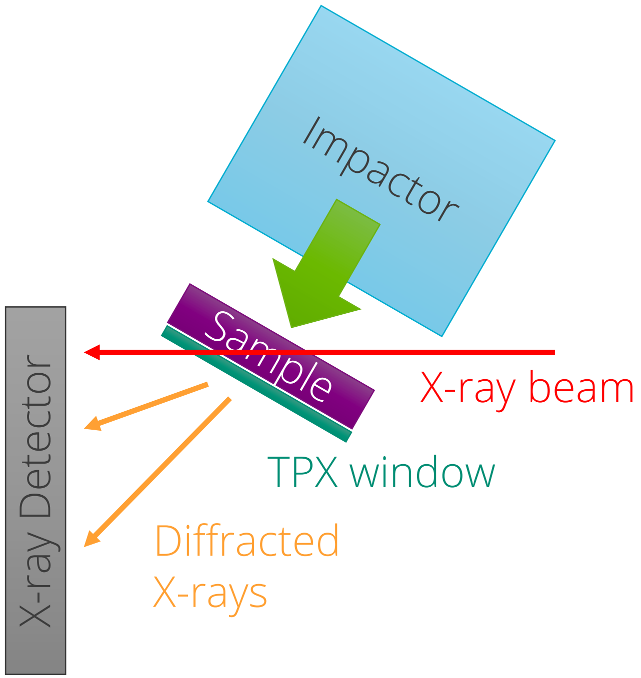
47th International Conference and Expo on Advanced Ceramics and Composites (ICACC2023)

S1: Mechanical Testing, and Characterization of Ceramics and Composites

January 25th, 8:50 – 9:10 AM

Dynamic Compression with ***In Situ*** X-ray Diffraction (XRD)

- Impactor (e.g., LiF or Lexan) hits sample at km/s → shock wave in sample
- *In situ* XRD probes phase transformations



Four X-ray detectors imaging at ~153 ns time spacing

- Recently developed capability at the Dynamic Compression Sector (DCS) facility at Argonne National Laboratory
- Understand behavior of compression-driven phase transformations, their pathways, and kinetics
- Prior dynamic compression XRD work has largely focused on monatomic solids with limited work on single crystal binary materials

Examine the impact of dynamic compression on phase stability in compositionally complex and "meta-stable" oxides

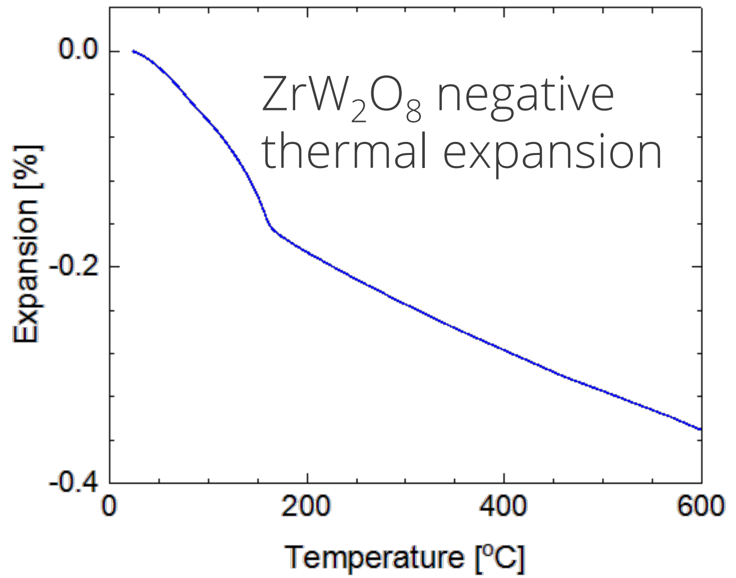
Materials

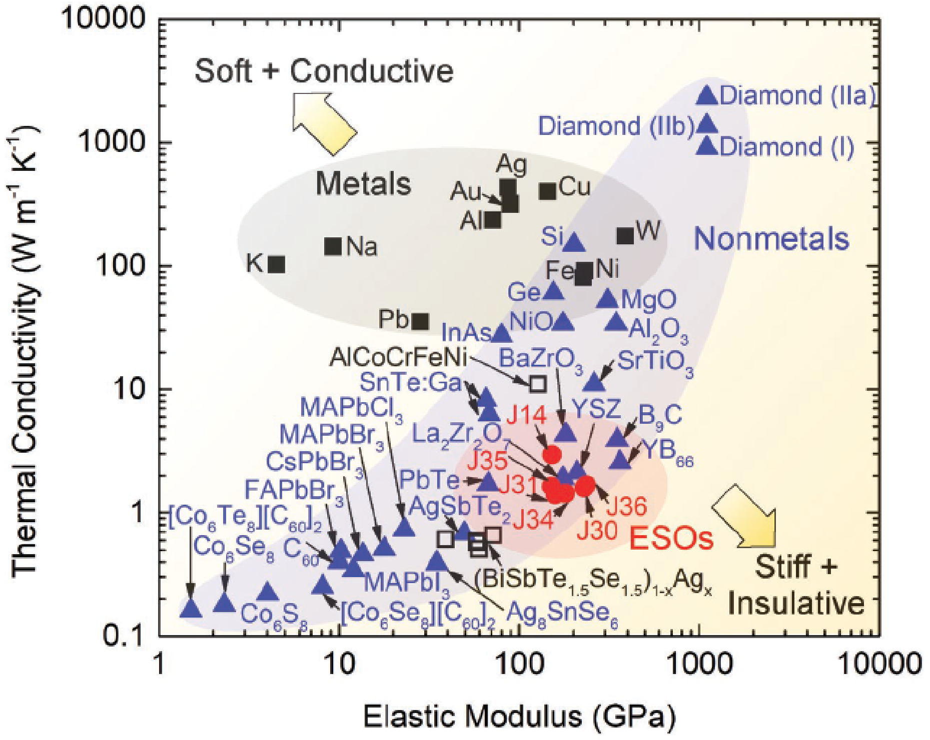
ZrW_2O_8 negative thermal expansion oxide

- Mitigate thermal stresses and strains, e.g., in radioactive waste storage[1] and high precision optical systems
- Cubic, kinetically-stable below ~700 °C

Compositionally complex oxides (CCOs)

- Low thermal conductivity[2], e.g., for thermal barriers
- May be metastable, i.e., entropy stabilized oxide (ESO)
- $(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$ (ESO[3])
- $(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})_2O_3$





[1]E. Kim, Chem. Phys. Lett. 744 (2020) 137172
[3]C.M. Rost, Nature Comm. 6 (2015) 8485

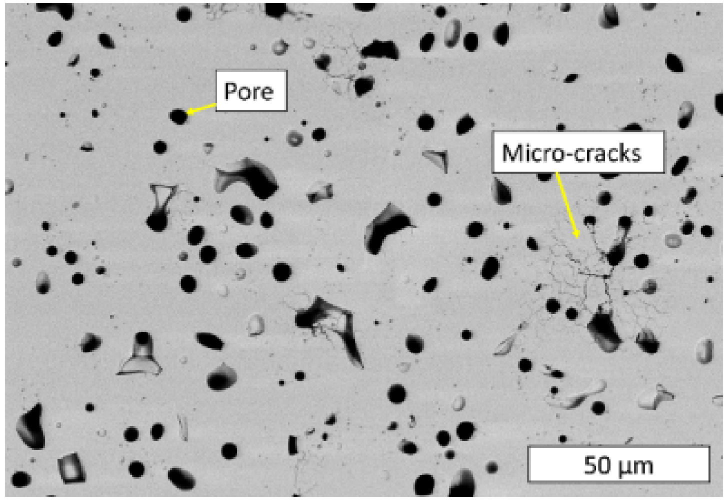
[2]J. Braun, Adv. Mater. 30 (2018) 1805004

Sample Fabrication, Purity, and Microstructure

Solid state reaction using precursor oxides and/or carbonates

ZrW_2O_8 "tungstate"

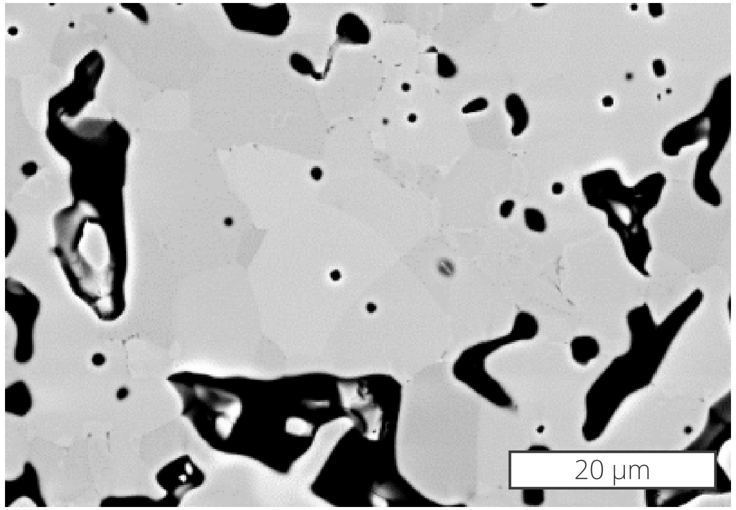
- 1160 °C (4 h) sinter and air-quenched to room temperature
- 95% cubic ZrW_2O_8
- 89% dense
- Some micro-cracking
- ~50 μm thick wafer for DCS



Back-scatter electron microscopy

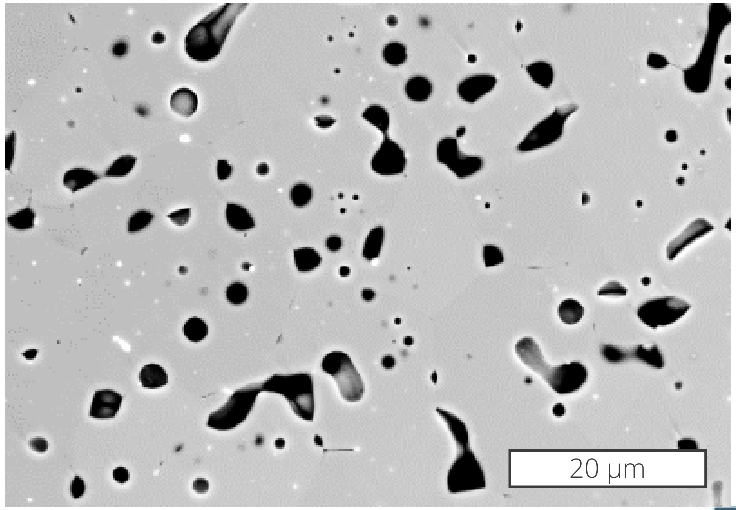
$(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})_2O_3$ "bixbyite"

- 1600 °C (12 h) sinter and furnace-quench
- Single phase bixbyite
- 83% dense
- ~150 μm thick wafer for DCS

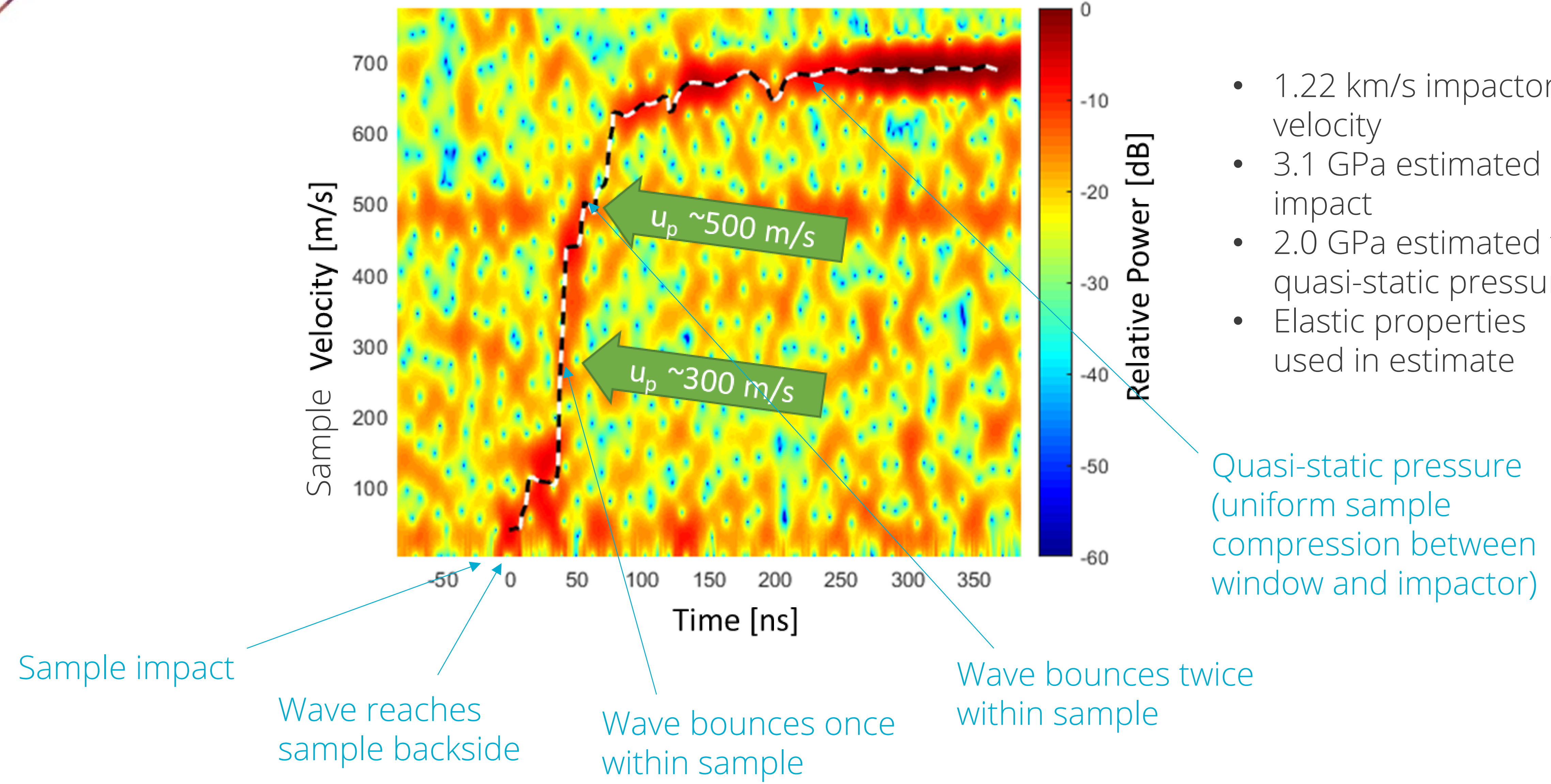


$(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$ "rock salt"

- 1100 °C (12 h) sinter (no calcine) and air-quench (below 1000 °C)
- Single phase rock salt
- 92% dense
- ~250 μm thick wafer for DCS



ZrW_2O_8 Dynamic Compression -Photon doppler velocimetry (PDV)



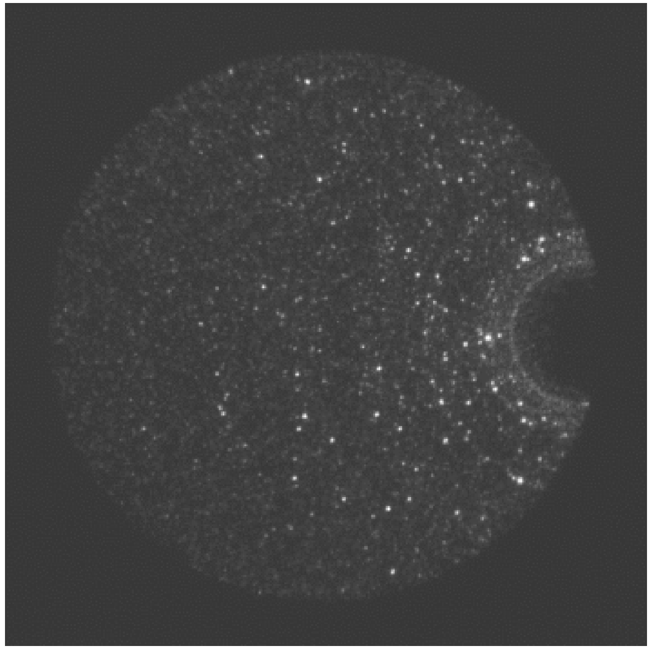
- 1.22 km/s impactor velocity
- 3.1 GPa estimated at impact
- 2.0 GPa estimated for quasi-static pressure
- Elastic properties used in estimate

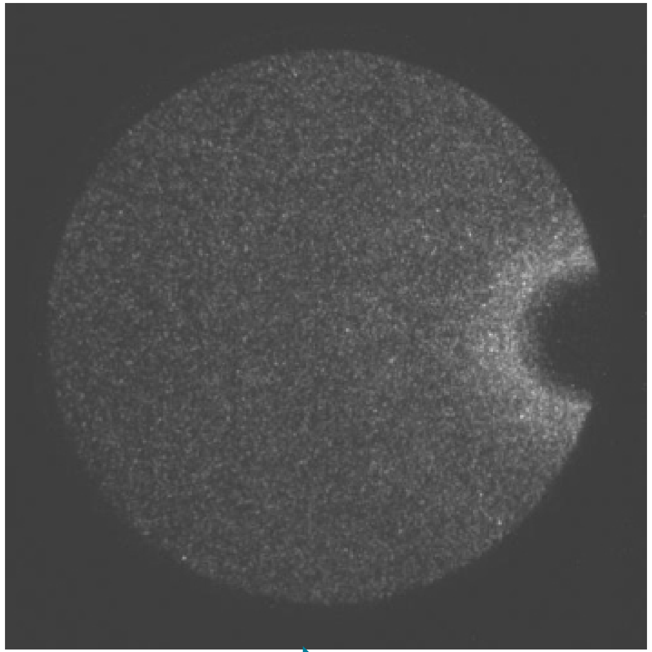
Pressure Induced Amorphization (PIA) at 3.1 GPa

X-ray diffraction

Before Impact

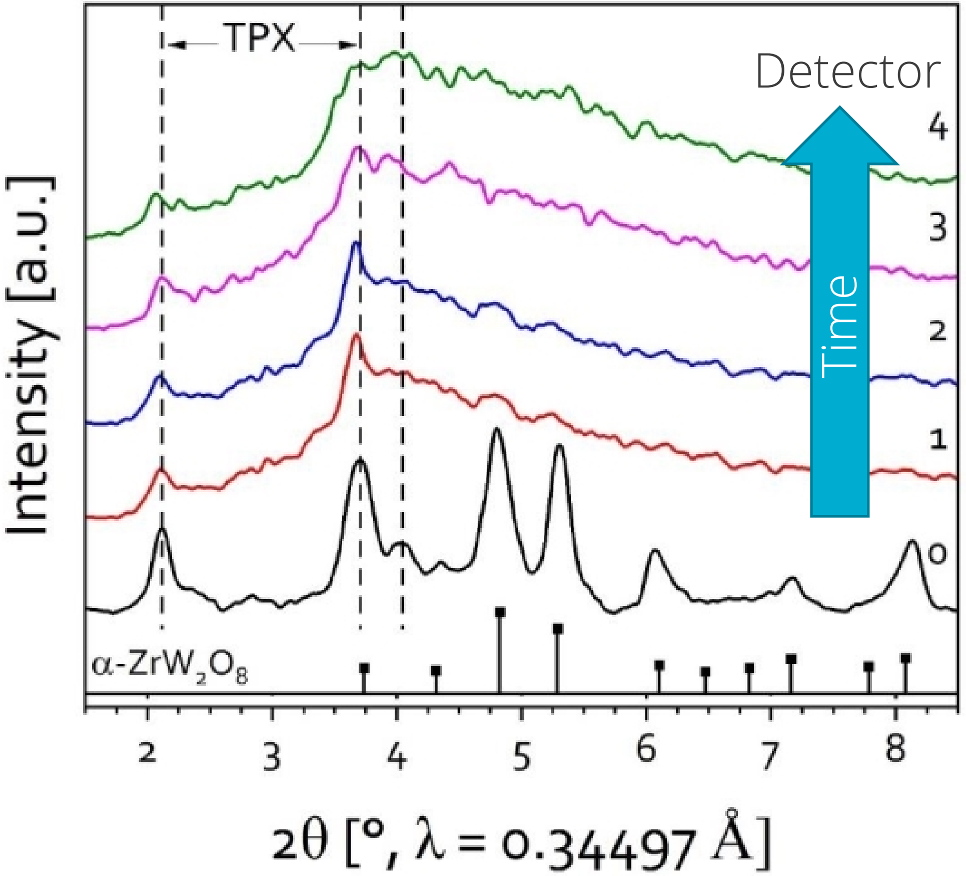
After Impact





Amorphization after impact

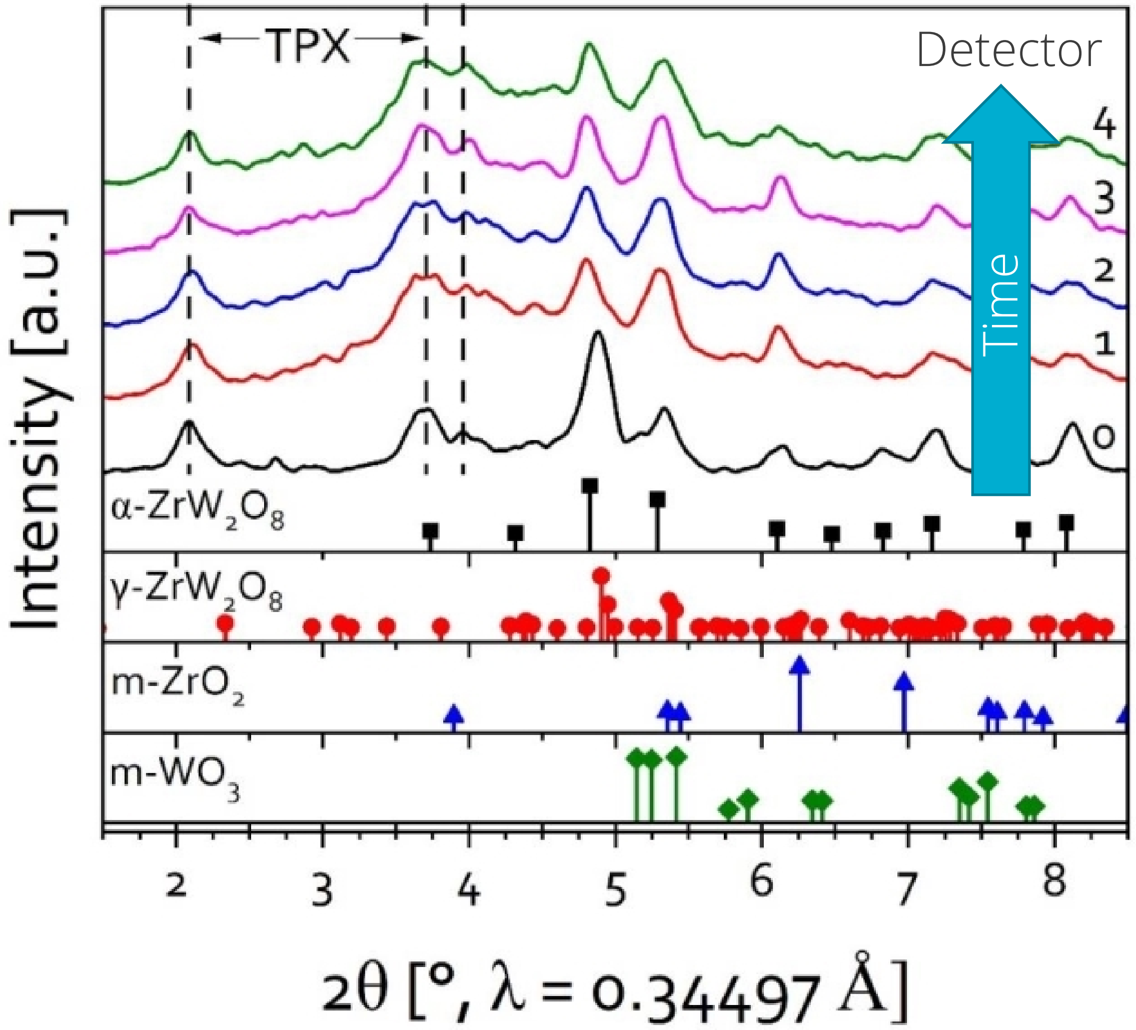
Integrated XRD images



3.1 GPa PIA consistent with quasi-static values of 1.5 - 3.5 GPa[1]

[1]Perottoni, Science, 280 (1998) 886

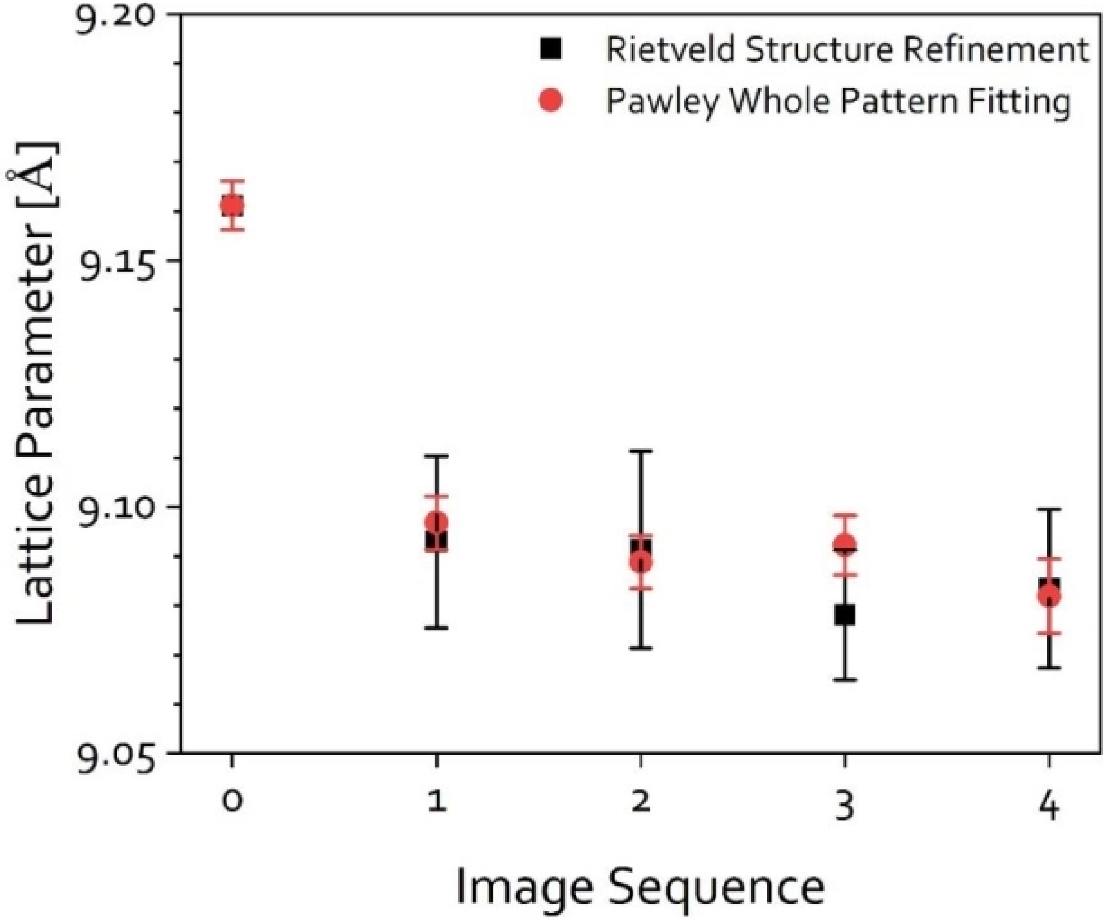
No Obvious Phase Change at Lower, 1.8 GPa Peak Stress



- 1.0 GPa quasi-static stress
- Orthorhombic (γ) at >0.2 GPa from quasi-static data[1] – not clearly observed here

[1]Perottoni, Science, 280 (1998) 886

ZrW_2O_8 Lattice Parameter Compression

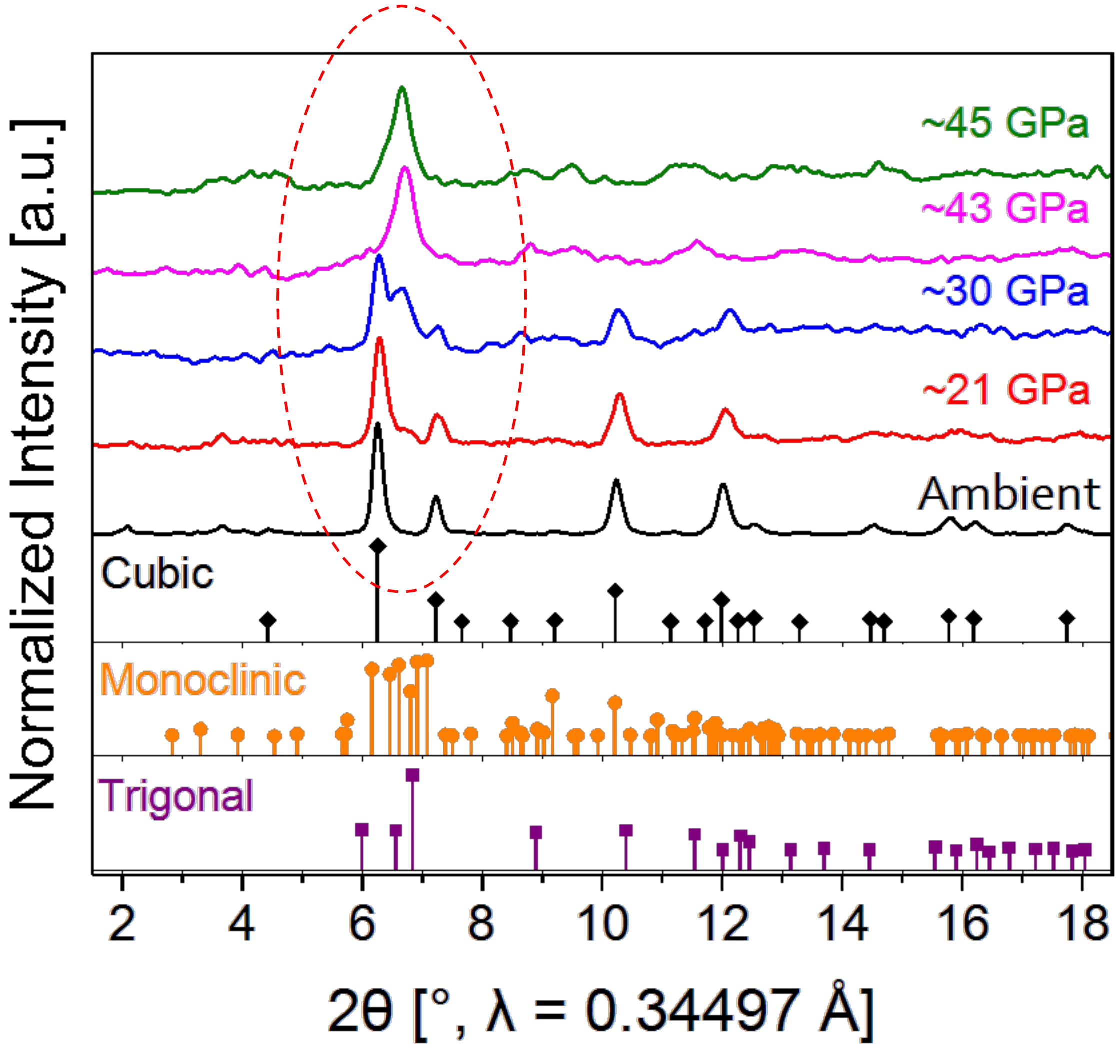


1.0 GPa quasi-static stress

0.8% compressive strain

Less than 1.1% compressive strain expected in quasi-static condition

Phase Transformations in $(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})_2O_3$



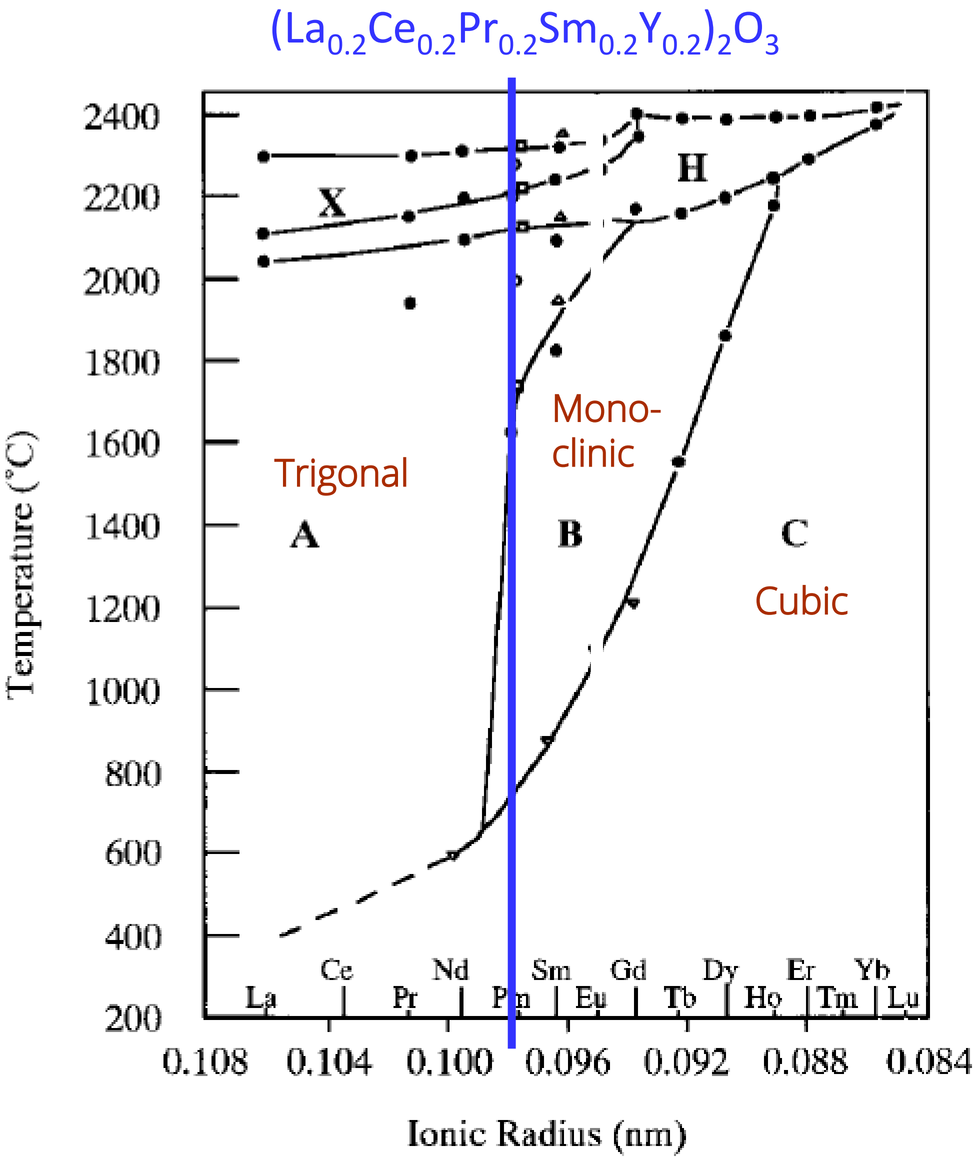
- Multiple shock induced phase transformations!
- In contrast to quasi-static amorphization at ~16-30 GPa for $(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})O_2$[2]

[2]Cheng, Comm. Chem., 2 (2019) 114

*Low PDV resolution → peak stress **estimates** based on CeO_2 and UO_2 Hugoniots*[1]

[1]Marsh, LASL Shock Hugoniot Data (1980)

Phase Transformations in Rare Earth M_2O_3



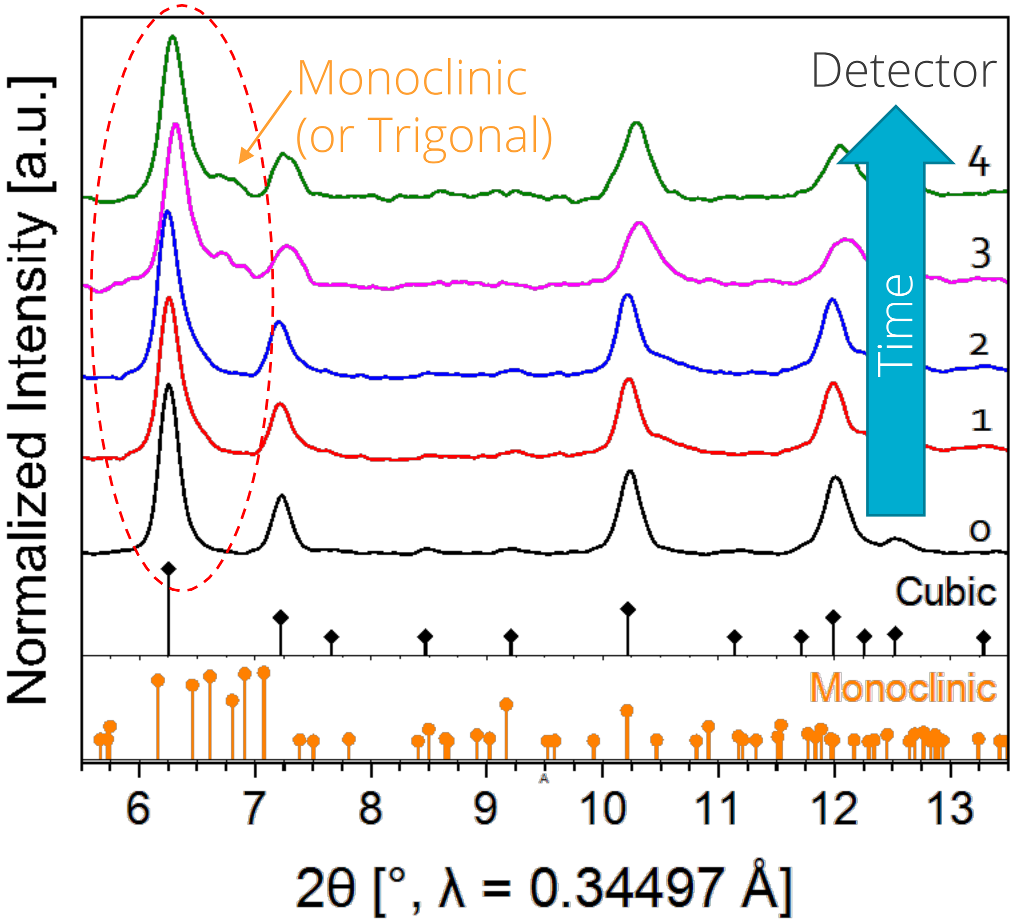
Adachi, Chem. Rev., 98 (1998) 1479

- Cubic → monoclinic → trigonal phase transformation reported for increasing temperature
- Similar phase formation observed for present shock measurements
- Shock in ~100 ns, limited atomic diffusion for phase transformations

Kinetic Delay in Phase Transformation

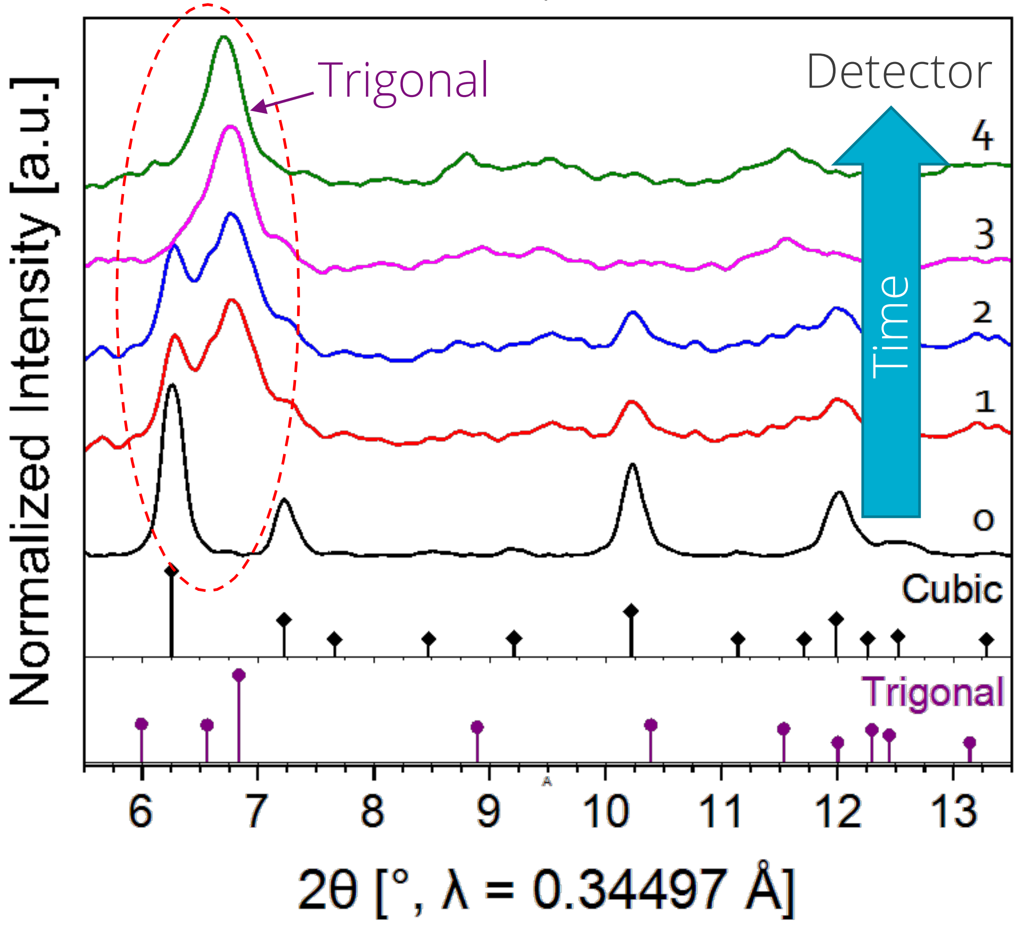
$(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})_2O_3$

~21 GPa peak stress



~300 ns needed for monoclinic to form

~43 GPa peak stress



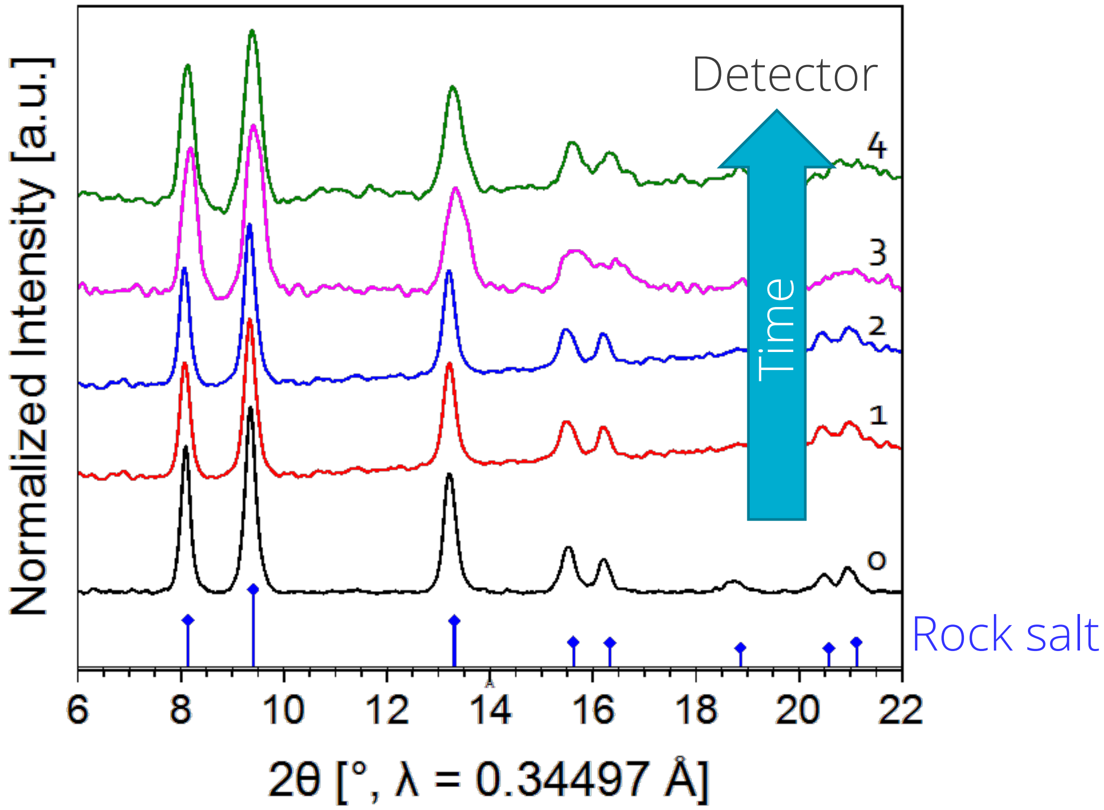
- Start of trigonal formation immediately
- ~300 ns until approximate transformation complete

Both phase transformations require 100s ns to form under dynamic compression

Dynamic Compression of $(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$

~24 GPa peak stress

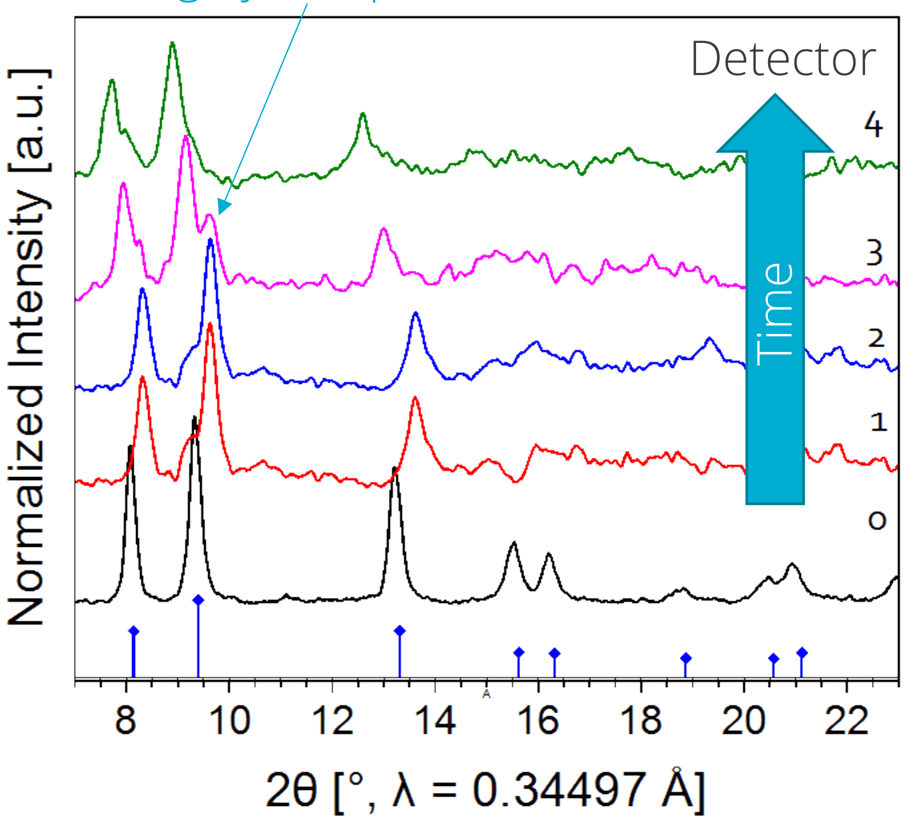
No change in phase



Single phase in quasi-static (meas. up to 48.2 GPa)[2,3]

~96 GPa peak stress

Highly compressed (5 – 9%) rock salt phase?



Possible shocked vs. unshocked microstructure features?

Low PDV resolution → peak stress **estimates** based on MgO Hugoniot[1]

[1]Marsh, LASL Shock Hugoniot Data (1980); [2]Chen, J. Phys. Chem. C 123 (2019) 17735; [3]Yue, Scripta Mater. 219 (2022) 114879

Summary

- ZrW_2O_8 - tungstate
  - Amorphization at 3.0 GPa peak stress, consistent with quasi-static literature results
  - Lattice parameter compression less than expected at 1.0 GPa
- $(La_{0.2}Ce_{0.2}Pr_{0.2}Sm_{0.2}Y_{0.2})_2O_3$ – bixbyite
  - Cubic → monoclinic → trigonal phase transformations induced by shock
  - Contrary to amorphization observed in literature
  - Kinetically delayed phase transformations during shock
- $(Cu_{0.2}Co_{0.2}Mg_{0.2}Ni_{0.2}Zn_{0.2})O$ – rock salt
  - Consistent “low” pressure phase stability with literature (~24 GPa)
  - Possible highly compressed rock salt phase at high pressure (~96 GPa)

Acknowledgements

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