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*Title:* Continued Investigation of the Solid-Solid Phase Transitions in Zirconium

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## **Continued Investigation of the Solid-Solid Phase Transitions in Zirconium**

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Work has continued to refine and investigate differences in the Hugoniots for Zirconium with differing impurity levels. Past work has shown that interstitial oxygen inhibits the  $\alpha$ - $\omega$  phase transition but appears to have little effect on the  $\omega$ - $\beta$  phase transition. Further plate impact experiments have been carried out to determine differences in the Hugoniots for two of the three types of Zirconium samples studied in previous work.

# Continued Investigation of the Solid-Solid Phase Transitions in Zirconium



**P. A. Rigg, C. W. Greeff, and G. T. Gray, III**

**APS SCCM, June 26-July 1, 2011**

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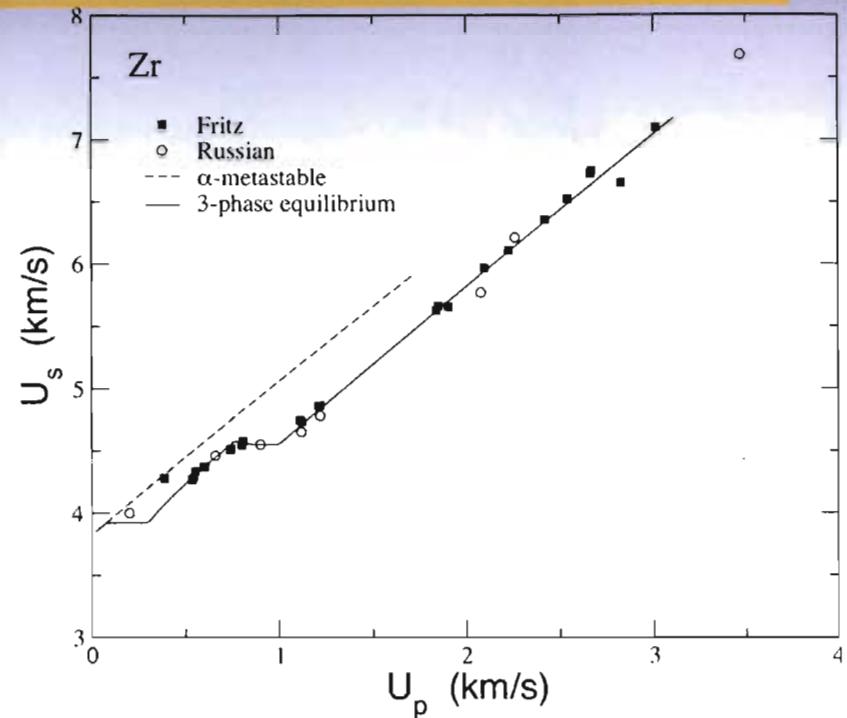
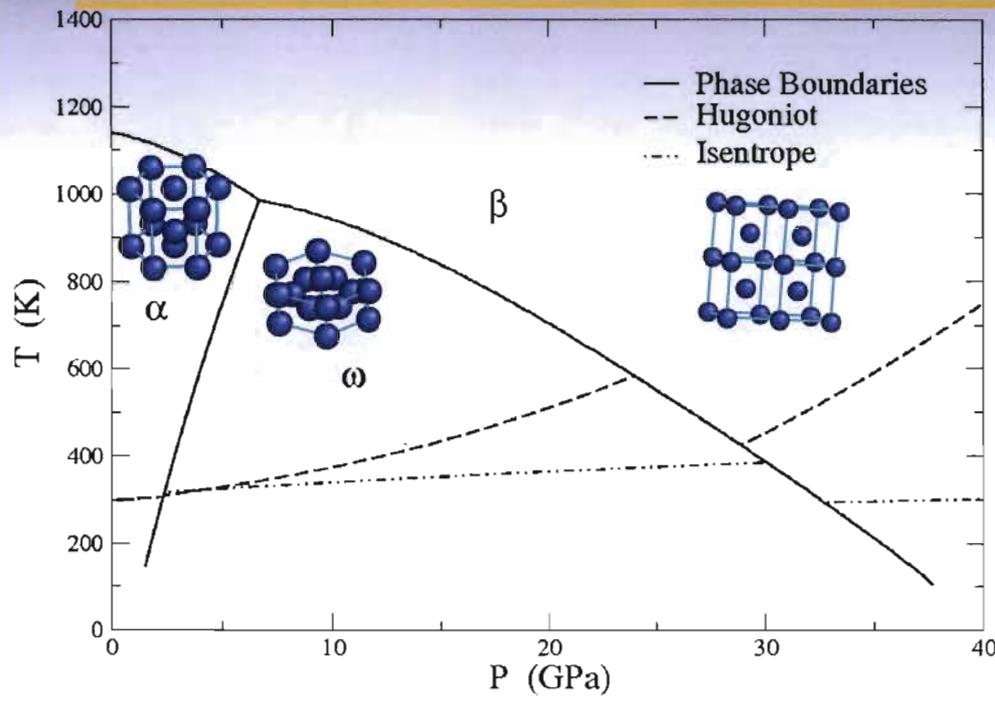
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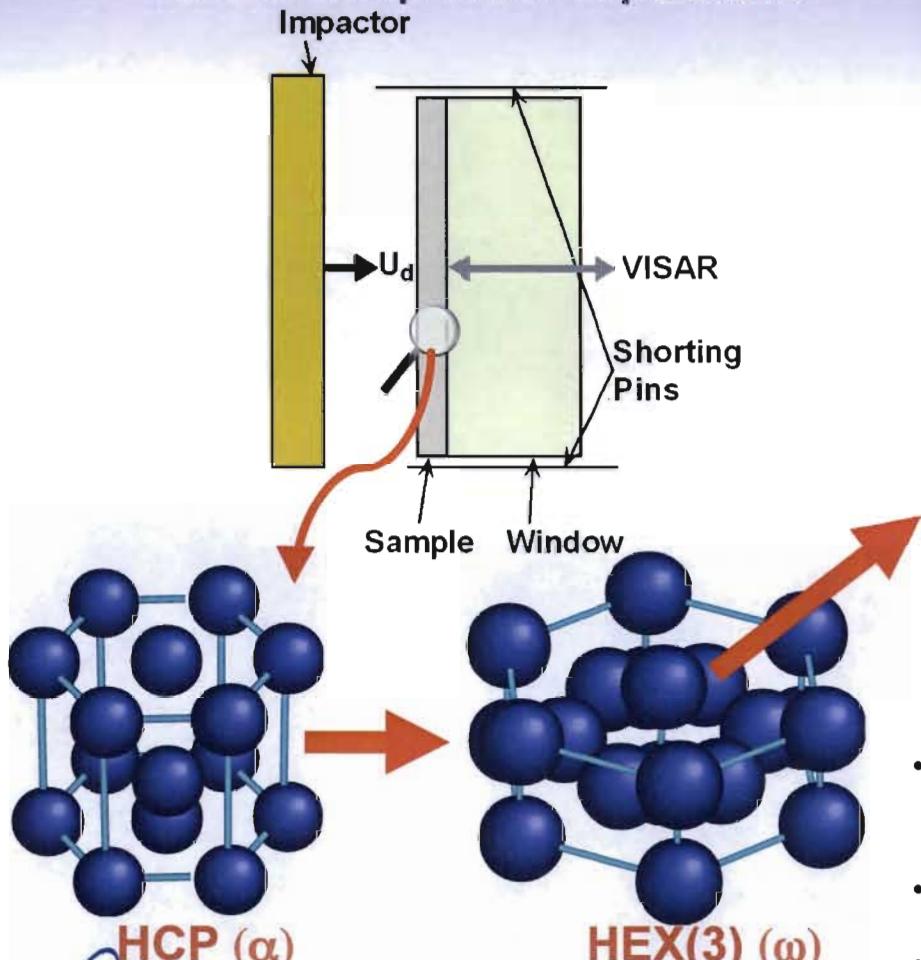
# Zirconium is well suited for investigating solid-solid phase transitions under dynamic loading conditions



- Three solid phases exist in Zr in pressure regimes easily accessible through shock and isentropic loading.
- Kinks in legacy  $U_s - U_p$  data indicate that transitions should be observable in shock compression experiments

# Velocimetry is used in shock compression experiments to detect phase transitions in real time

## Shock Compression Experiment

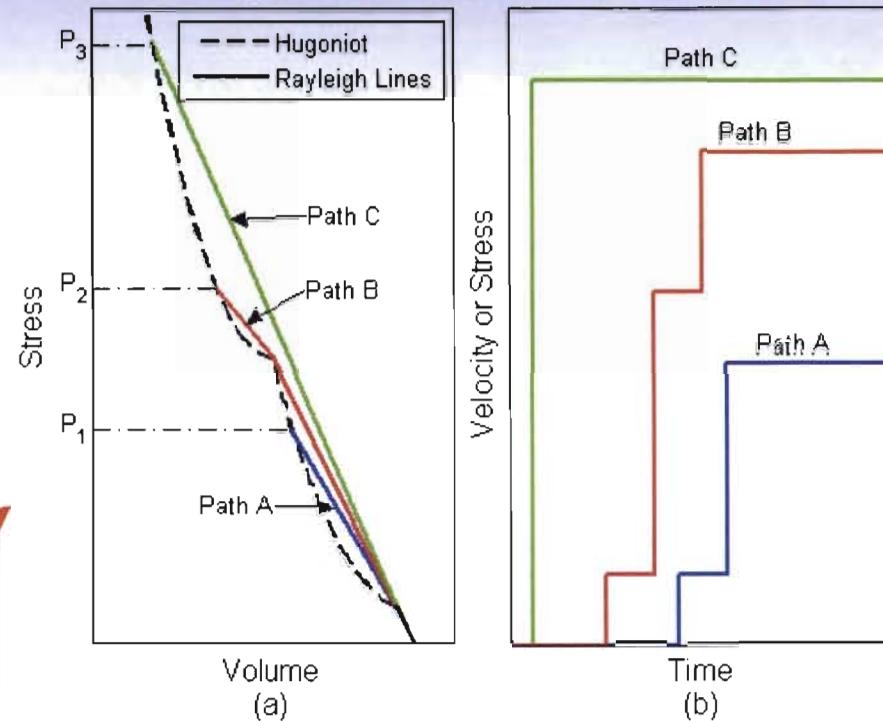


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## Loading Paths and Ideal Wave Profiles

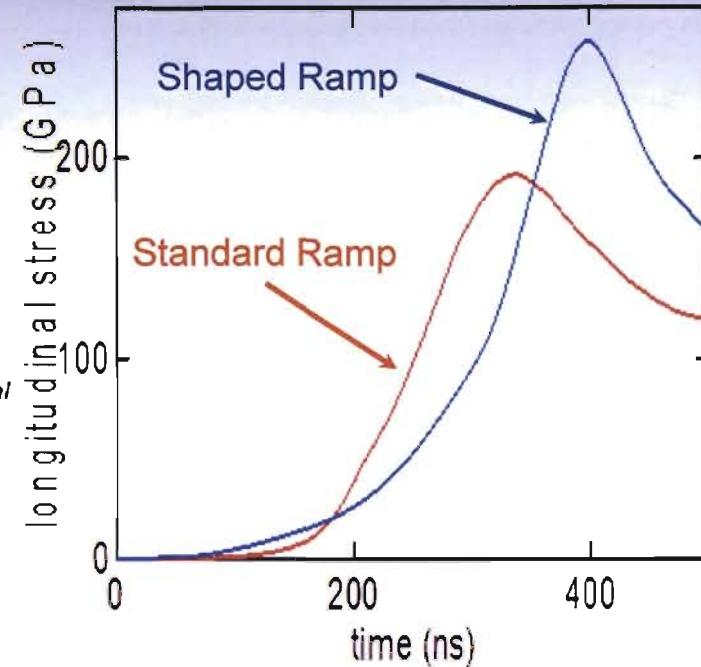
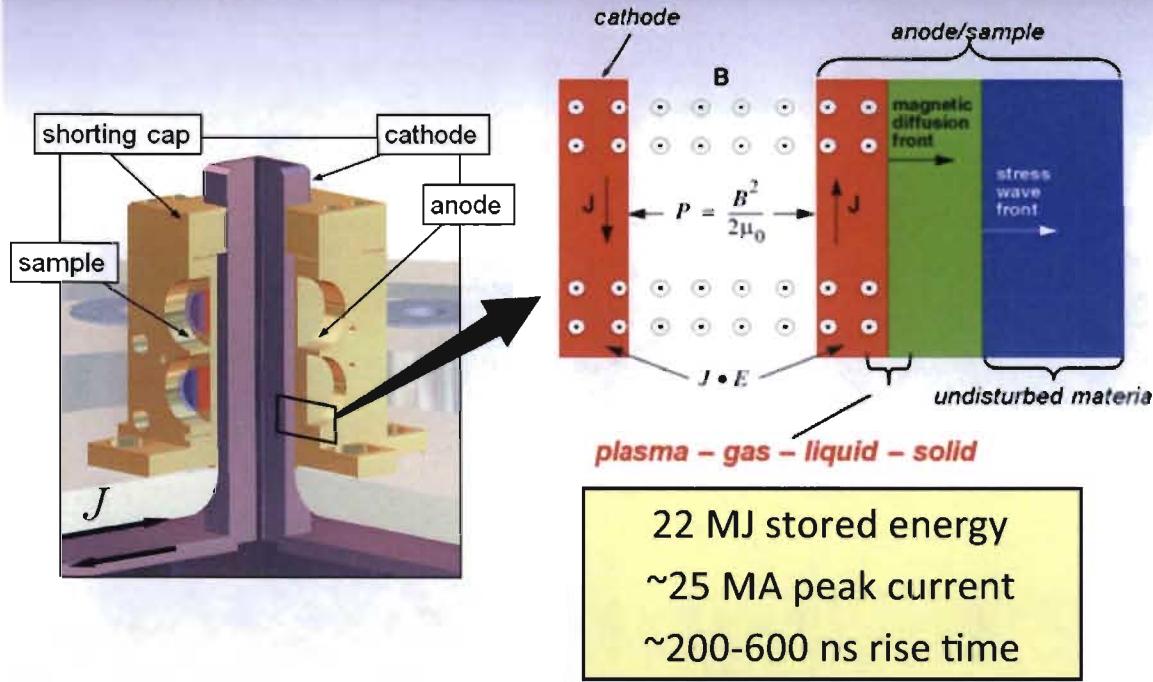


- A kink in the Hugoniot indicates a change in the material:
  - Elastic-plastic deformation
  - Phase transition
- When the Rayleigh line intersects these kinks, multiple shock waves are formed (Paths A & B).
- **Path C:** Changes still occur, but with no wave profile signature – **changes are “overdriven”**.

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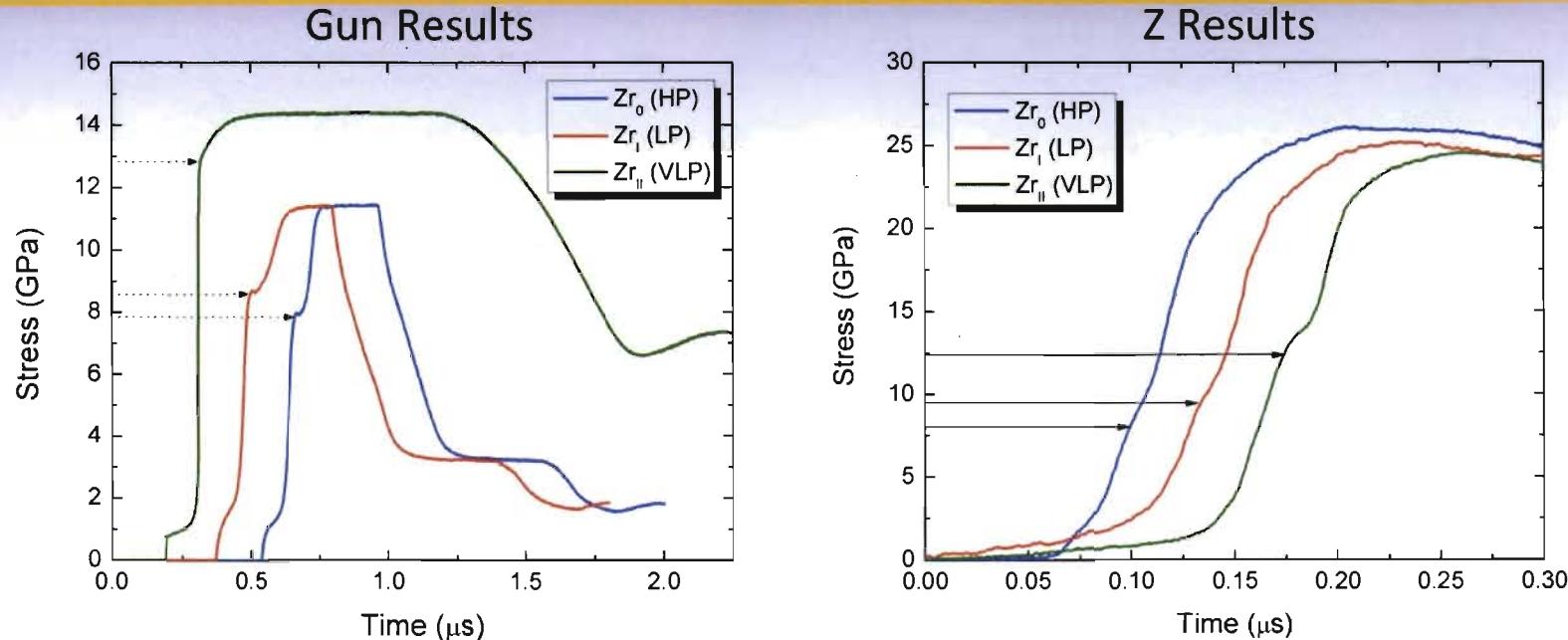
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# Isentropic compression data can provide additional information about the kinetics of phase transformations



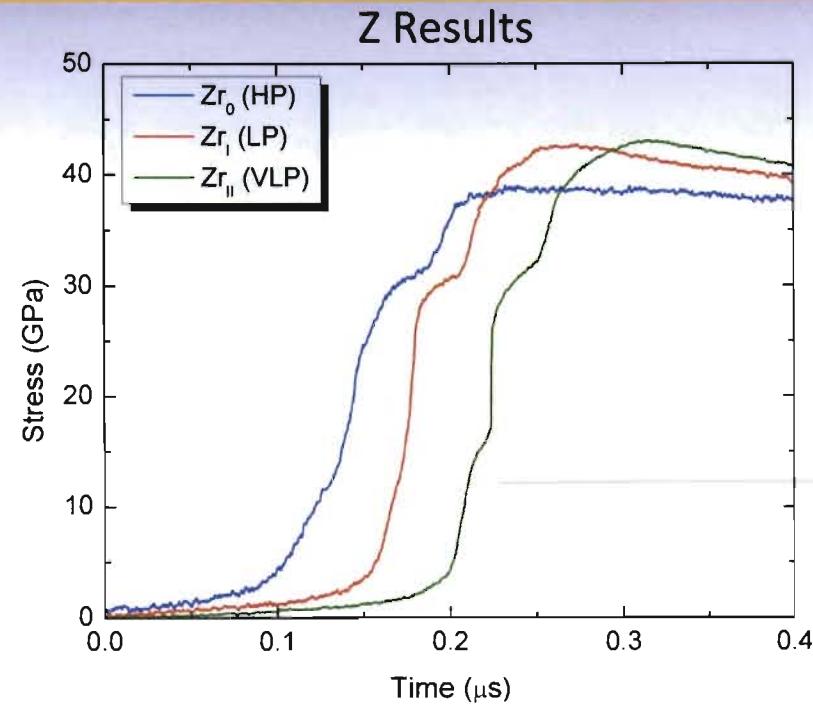
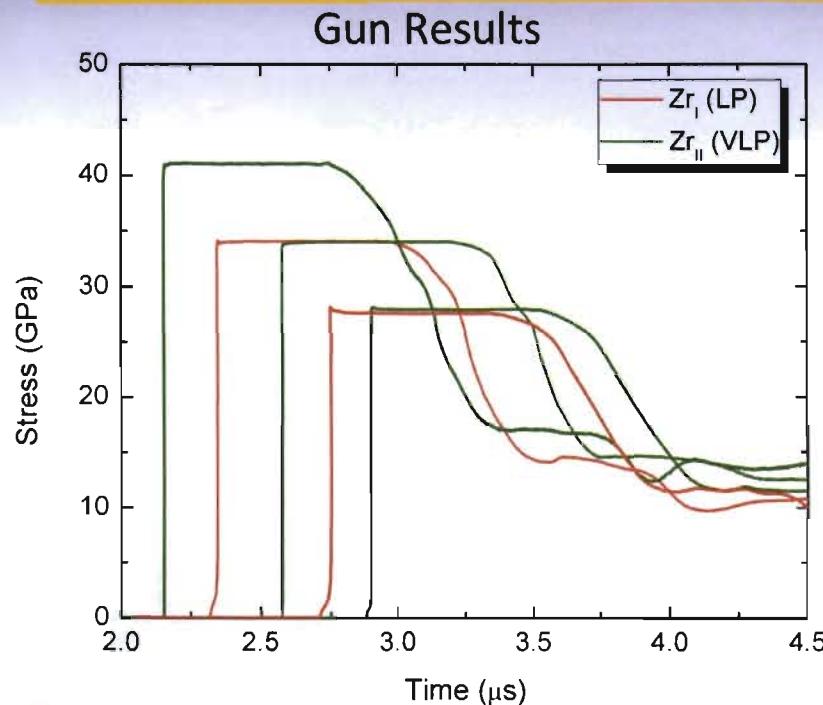
- Magnetic field between anode/cathode induces pressure on the sample.
- Pressure pulse temporally follows current
  - This can be tailored by dumping capacitors sequentially
- Materials closely follow isentrope when ramp loaded.
- Isentrope will follow Hugoniot very closely for several hundred kbar.
- Same phase boundaries are accessible through shock and isentropic loading.
- Phase transition cannot be “overdriven”.
- Lower strain-rates allow investigation of strain-rate effects on phase transitions.

# The presence of oxygen in Zr dramatically increases the stress at which the $\alpha - \omega$ phase transition occurs

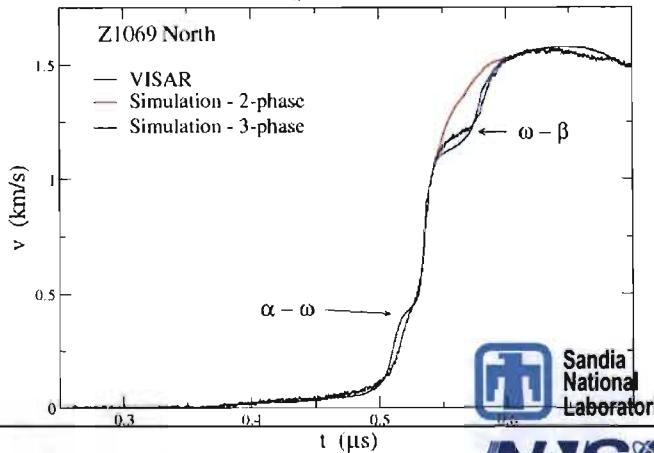


- Oxygen levels are < 50ppm for  $Zr_0$ , 390ppm for  $Zr_1$ , and 1200ppm for  $Zr_{II}$ .
- The phase transition signature is very subtle in the shocked impure sample and is overdriven just a few GPa higher.
- In contrast, the signature is more pronounced for the isentropically loaded impure sample indicating that strain rate is important.
- No rarefaction shock indicating  $\omega$  phase is retained upon release.

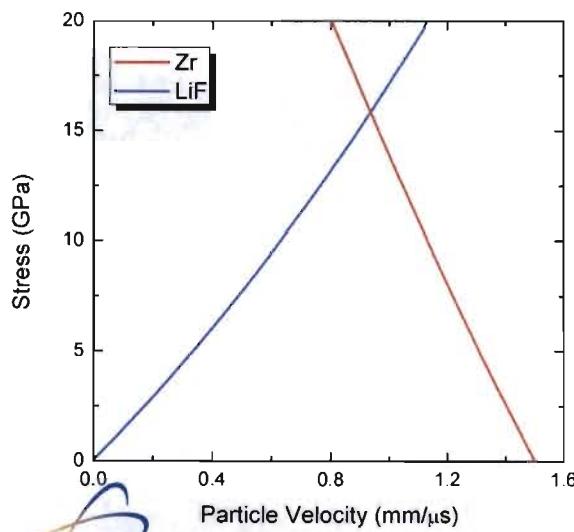
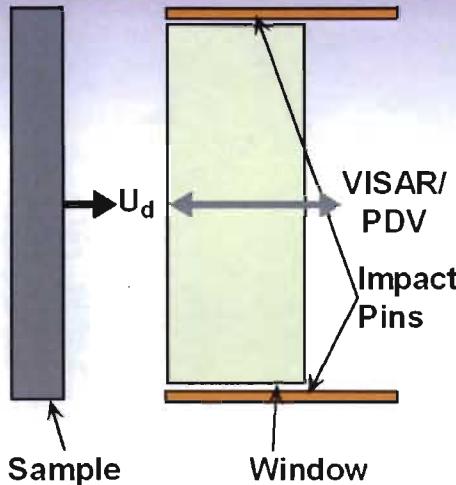
# Further experiments conducted to look for $\omega - \beta$ transition



- 3-wave structure not observed in gun experiments.
- Oxygen content does not affect  $\omega - \beta$  transition stress in Z experiments.
- Model matches Z data well. Shock data not modeled



# Front surface impact (FSI) experiments eliminate wave interactions due to multi-wave structure



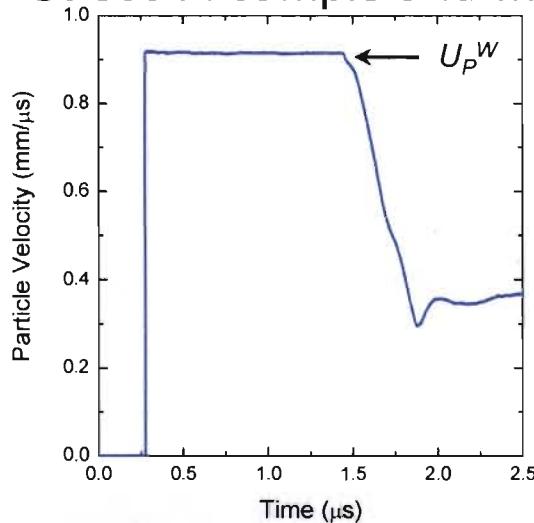
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- Sample is impacted on window directly.
- Measurement of projectile velocity,  $U_d$ , and particle velocity,  $U_P^W$ , needed to determine Hugoniot point:
  - Projectile velocity measured using shorting pins or PDV to 0.1%.
  - Particle velocity measured at impact using VISAR and PDV to ~0.5 - 1%.

- Stress in sample and window defined by window Hugoniot



- Shock Velocity determined from R-H Jump Conditions:

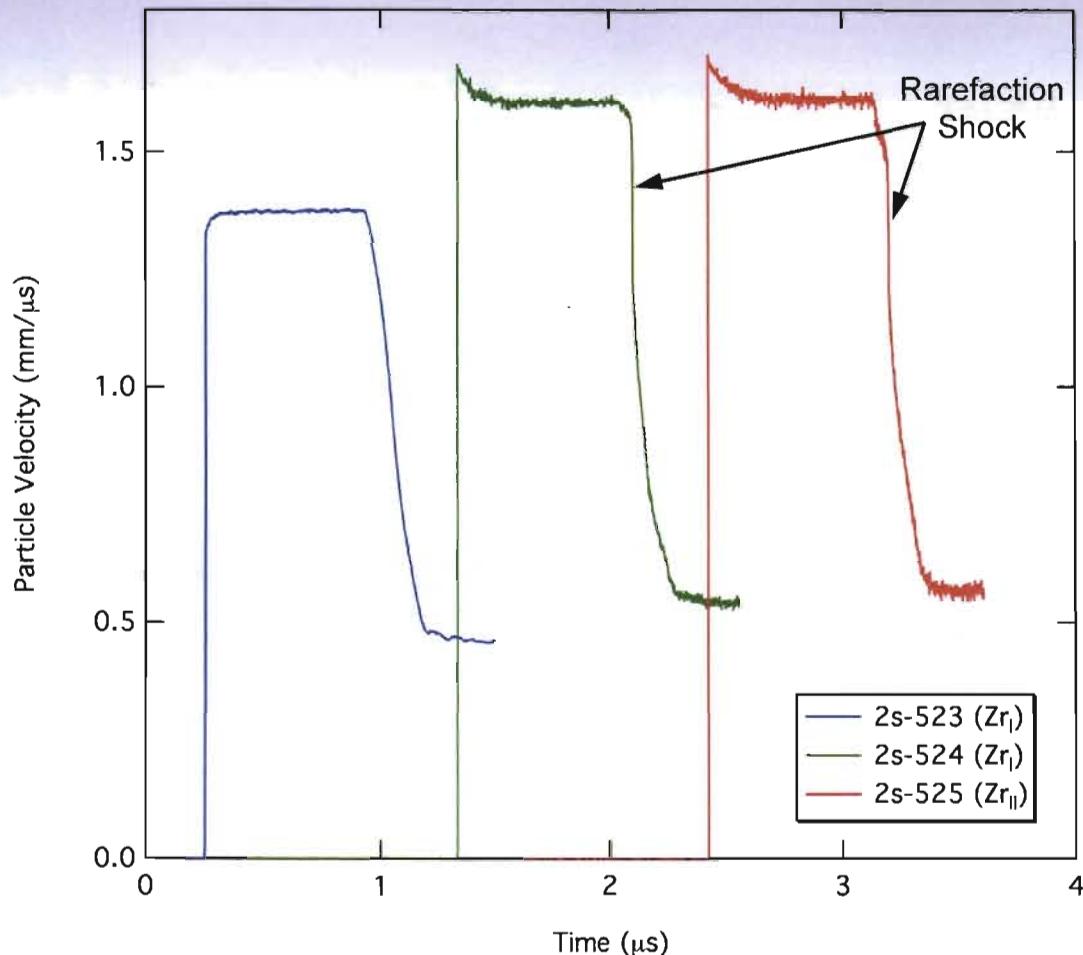
$$U_S^S = \frac{P - P_1}{\rho_1(U_d - U_P^W - U_{P1})}$$

- Must know initial conditions!

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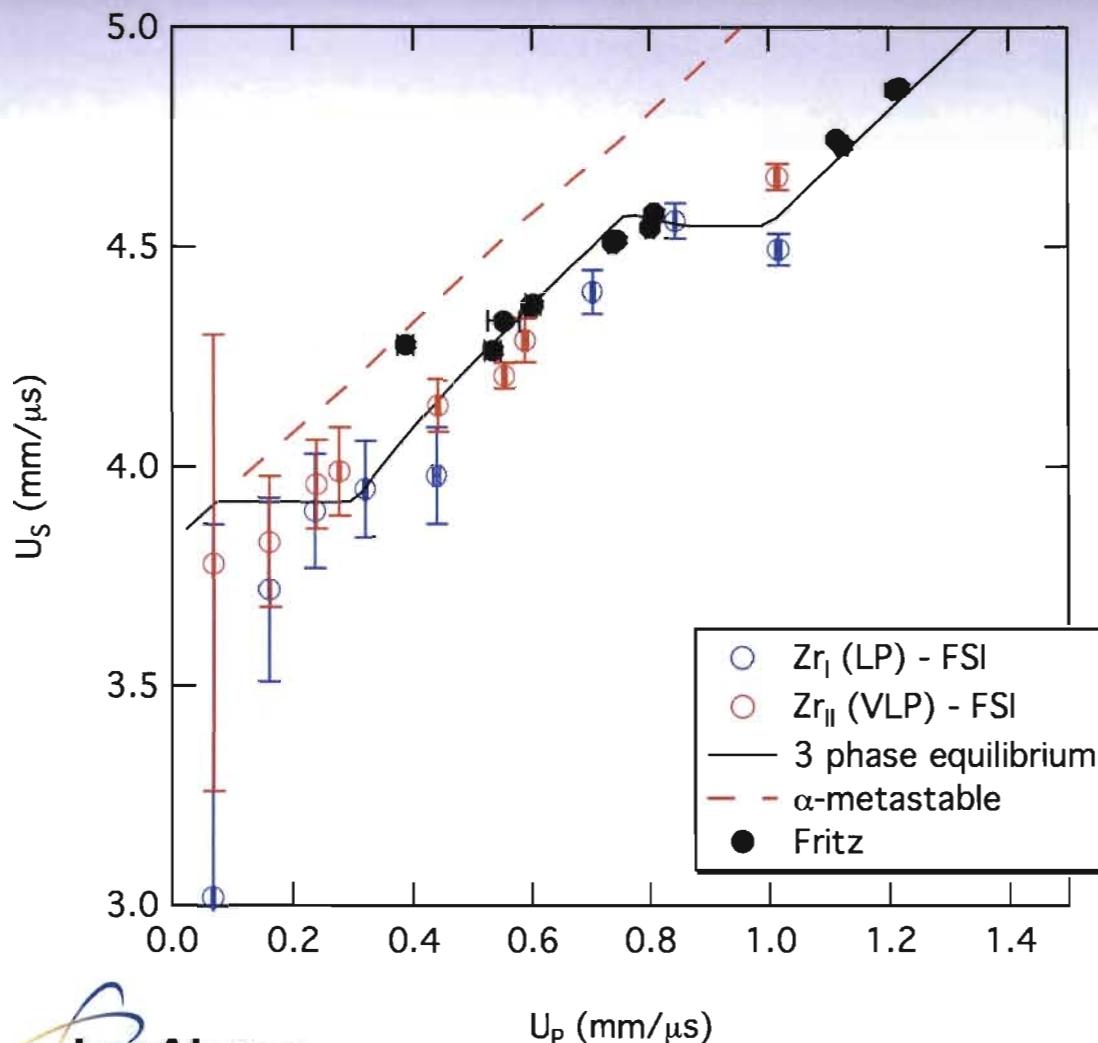
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## FSI experiments reveal $\beta$ to $\omega$ transition on release



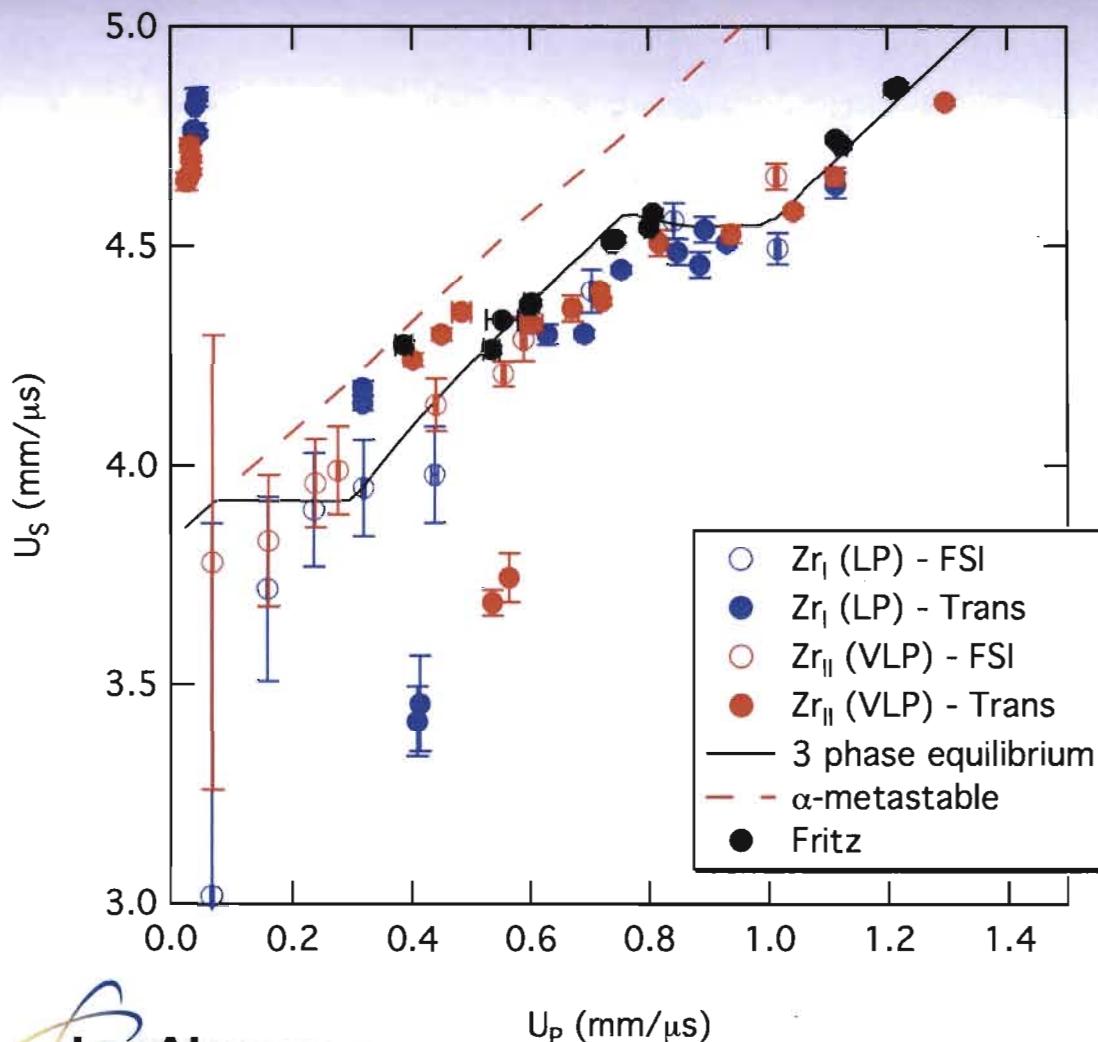
- Experiments conducted at 25 and 31 GPa on Zr<sub>I</sub> and 31 GPa on Zr<sub>II</sub> samples.
- At 25 GPa and below – Flat-top shock with ramped release
- At 31 GPa – Overshoot and relaxation at the front followed by shock formation on release
  - $\omega - \beta$  boundary crossed on shock
  - Kinetics of transition observed at shock front
  - Material reverts to  $\omega$  phase upon release

# Calculated Hugoniot is in very good agreement with data from front surface impact experiments



- Errors are large at low stresses due to uncertainties in initial state parameters.
- Results near the  $\alpha - \omega$  transition are more consistent with the calculated Hugoniot
  - Better measurement of equilibrium at transition??

# Calculated Hugoniot is in very good agreement with data from front surface impact experiments



- Errors are large at low stresses due to uncertainties in initial state parameters.
- Results near the  $\alpha - \omega$  transition are more consistent with the calculated Hugoniot
  - Better measurement of equilibrium at transition??
- Transmission experiments seem to be better for determining the  $\omega - \beta$  transition stress, but...

## Summary

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- Both front surface impact and transmission experiments were performed to investigate the influence of impurities on the solid-solid phase transitions in three purities of Zr.
- Presence of interstitial O<sub>2</sub> dramatically changes kinetics of  $\alpha - \omega$  phase transformation, but not the  $\omega - \beta$  transformation.
- Differences between shock- and isentropically loaded experiments shows kinetics are also influenced by the initial loading conditions.
- Greeff Equation of State includes kinetic model which successfully captures much of the phenomena observed experimentally.
- More work needed to successfully model Zr<sub>II</sub> material and simulation of gun experiments around  $\omega - \beta$  transition still needs to be done.
- Combination of several experimental techniques and theory are leading to a better understanding of phase transition kinetics.