Phase Changes in Ni-Ti Under Shock-Loading

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Alloy Design
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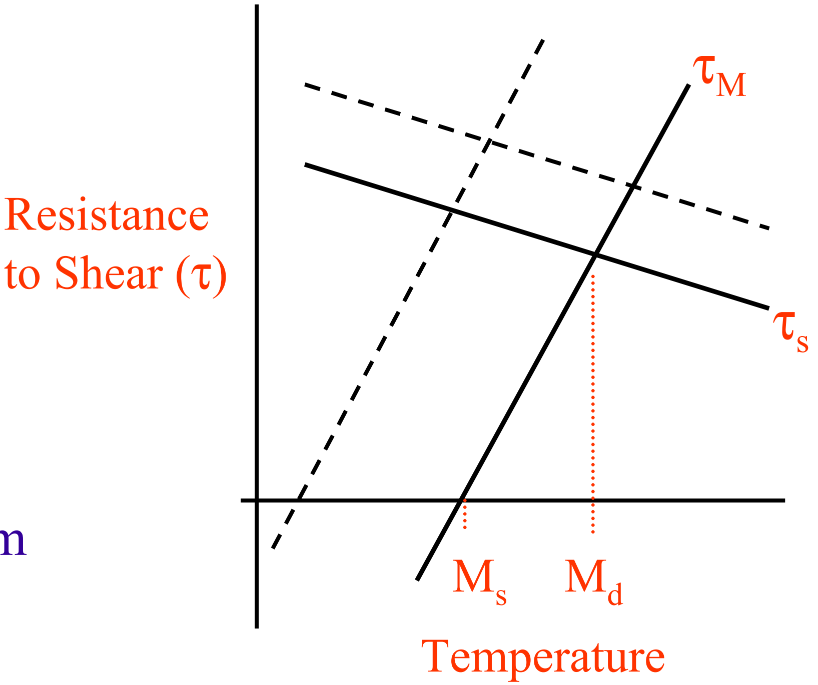
Stress-Induced Martensite

Martensite and plastic flow compete to cause shape change (*Scheil, 1932*)



Patel and Cohen analysis (1953)

- Add shear + dilatational energy term to free energy sum: $U = \tau\gamma_o + \sigma\varepsilon_o$
- Good agreement for Fe-Ni (-C)
  - Uniaxial tension > uniaxial compression
  - Hydrostatic compression has negative effect (stabilizes austenite)

Phase Stability in Ni-Ti

- Parent phase undergoes martensitic transition(s)
  - Shape memory effect (*Buehler et al., 1963*)
- Equiatomic NiTi decomposition
  - B2 → IC → (T_R) R-Phase → (M_s) B19' Martensite

    B2, IC: Ordered BCC (CsCl); R-Phase: Rhombohedral; B19' Martensite: Monoclinic
- Off-stoichiometric and ternary alloys
  - T_R and M_s shift
  - Ni-Rich: precipitation of Ni_4Ti_3, Ni_3Ti_2, Ni_3Ti
  - Little known about Ni_xTi_{100-x} when $x > 52$

Shock-Induced Martensite Experiments

- Why do shock loading experiments?
  - Control of time variable
  - Distinguish isothermal vs. athermal martensite
  - Reveal nucleation sites from incompletely grown martensite units
- Prior work
  - Gas-gun experiments limited to temporal resolutions of > 100ns
  - Fe-Ni (-Mn) (-C)
    - Meyers and Guimares (1976)
    - Thadhani and Meyers (1986)
    - Chang and Meyers (1988)
    - Sano, Chang, Meyers and Nemat-Nasser (1992)
  - Ni-Ti (-Fe)
    - Thakur, Thadhani and Schwarz (1997)

Goals of This Work

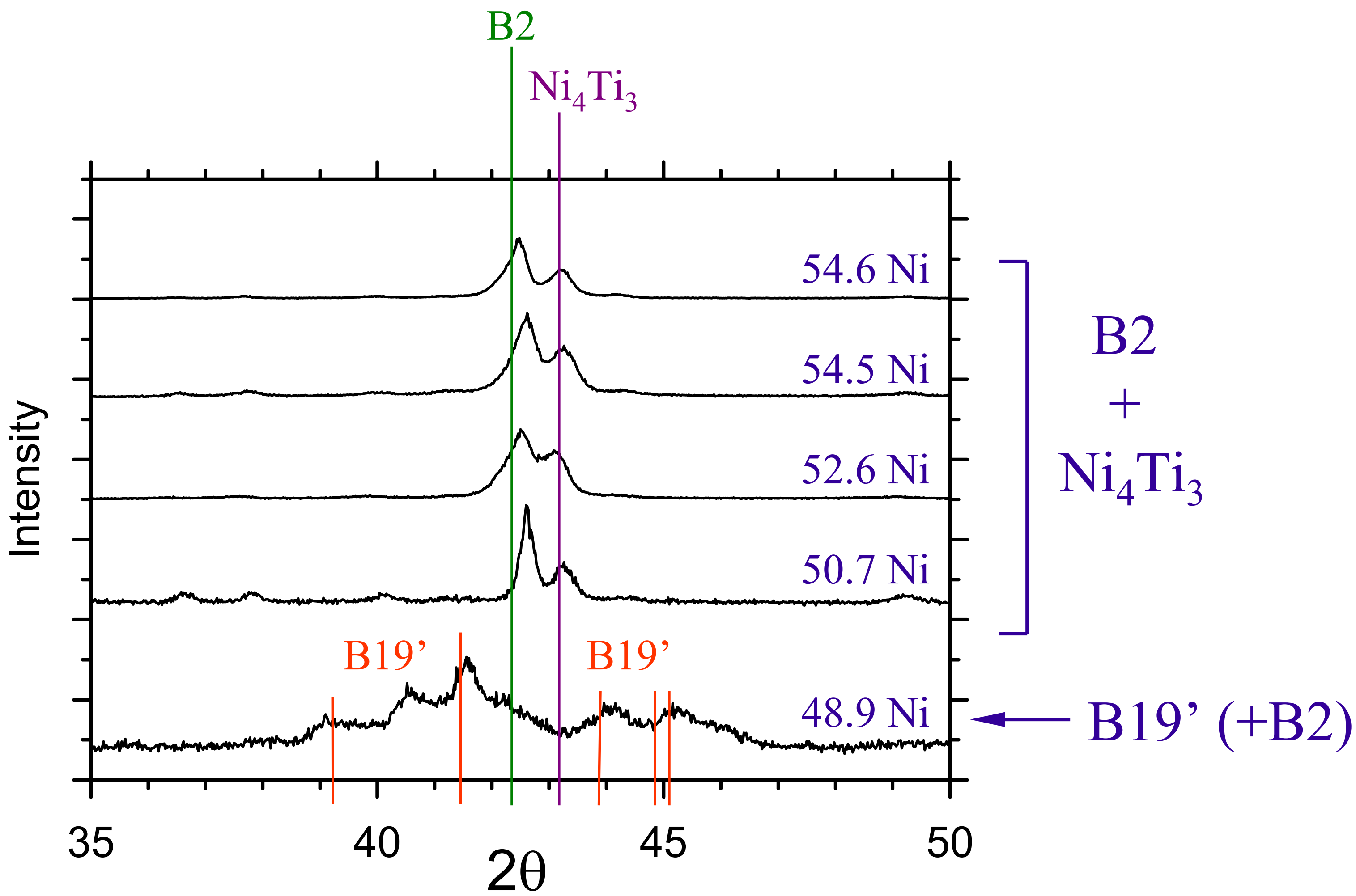
- Characterize Ni-Ti Alloys With High-Ni
  - Martensitic transitions
  - Intermetallic compound formation
  - Phase competition during B2 decomposition

- Investigate Effect of Laser Shock-Loading
  - Stress-induced martensite
  - Ultra-short stress pulses (~1 ns)
  - High stresses (~10 GPa)

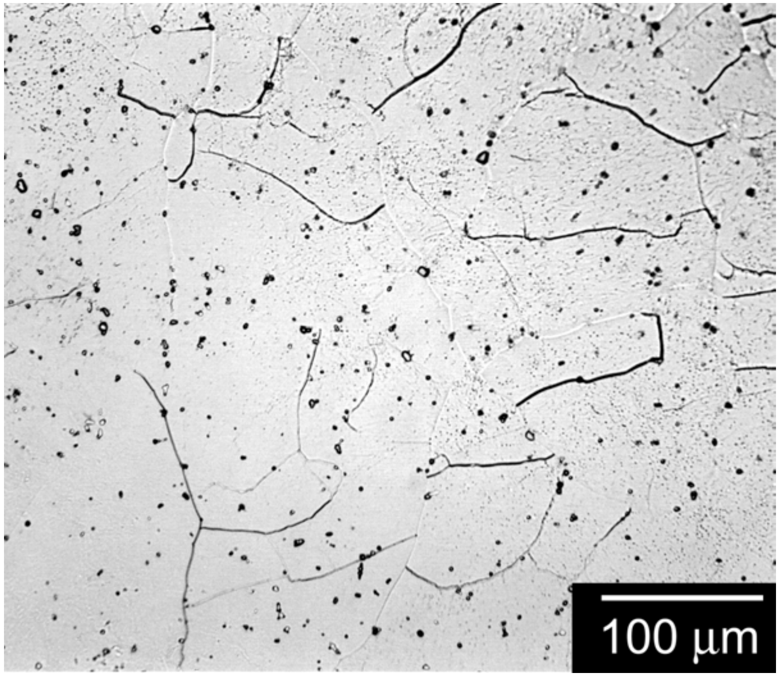
Experimental

- Alloy Processing
  - Binary Ni-Ti arc melted:
    - 48.9, 50.7, 52.6, 54.5, 54.6 At.% Ni
  - Homogenization at 1100^{o}C – 50 hours, water quench
- Shock-Loading in LANL Trident Laser
  - Direct drive shots - microstructural alteration
    - 2 shots per alloy (1.8 and 3.6 ns)
  - Symmetric impact (flyer plate) shots - equation of state
- Characterization
  - Optical microscopy, SEM, TEM
  - Resistivity and dilatometry
  - X-Ray diffraction (XRD)
  - Laser probing of back surface velocity during loading

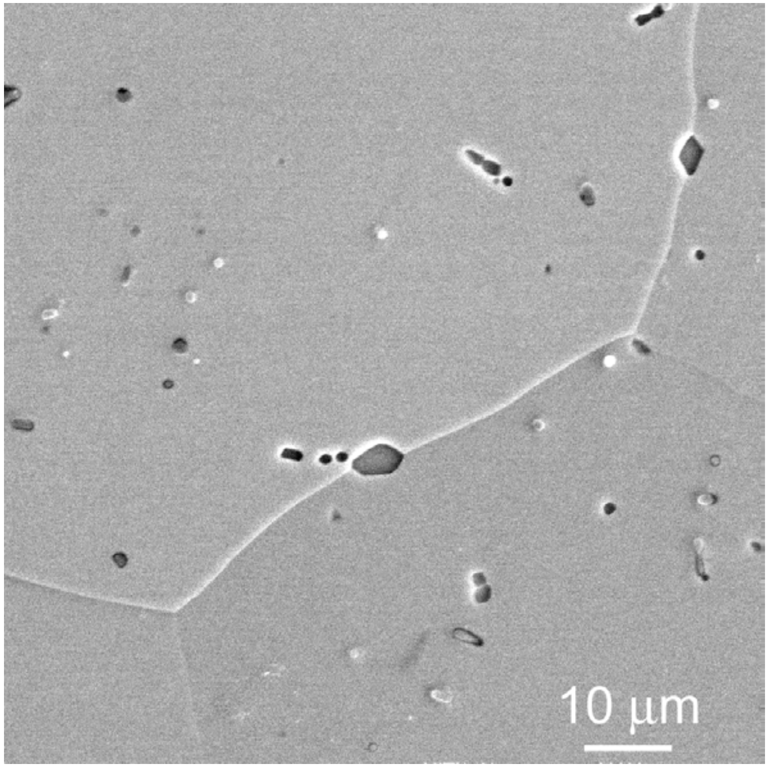
XRD: As-Quenched



As-Quenched Microstructure



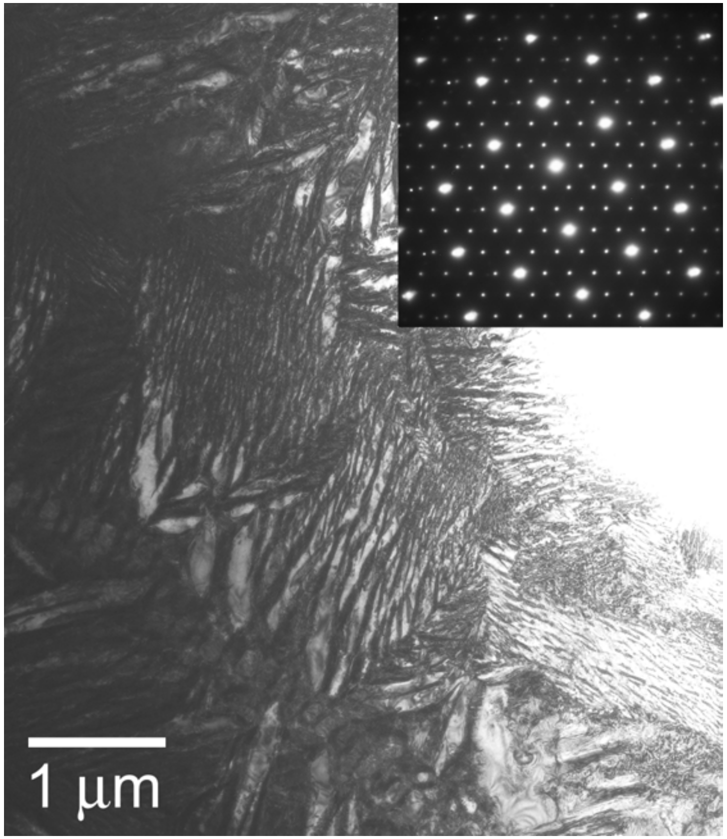
52.6 Ni



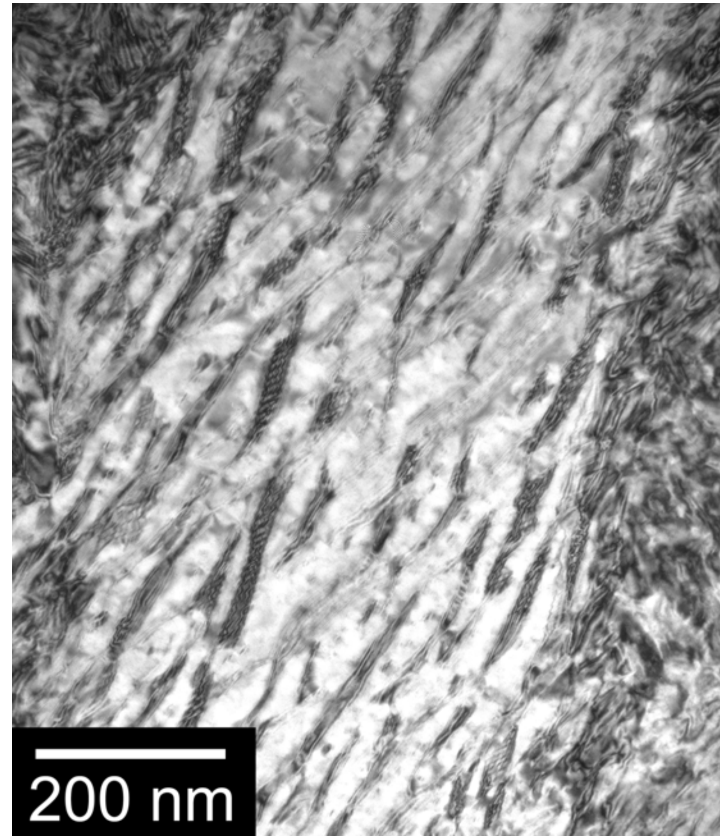
54.5 Ni

TEM-SAD and SEM-EDS show inclusions are Ti_4Ni_2O

Ni_4Ti_3 Precipitation



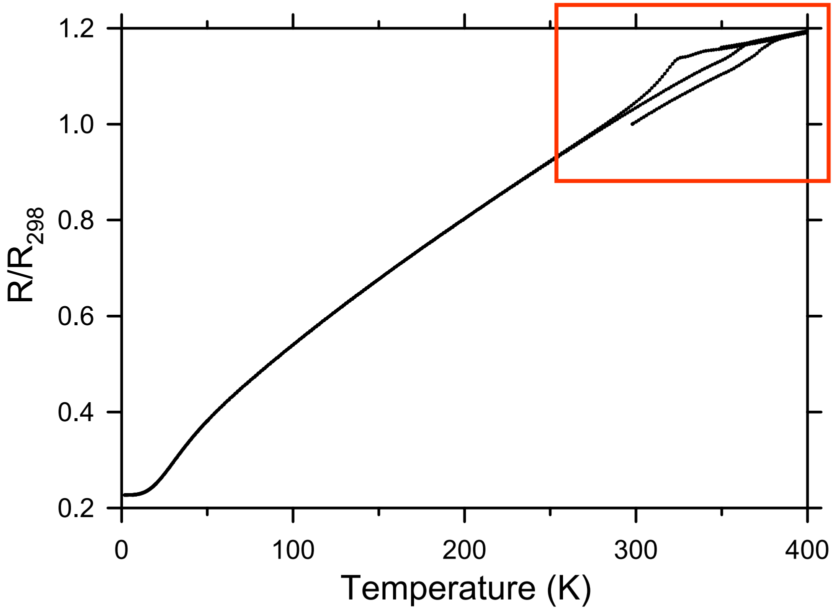
54.6 Ni

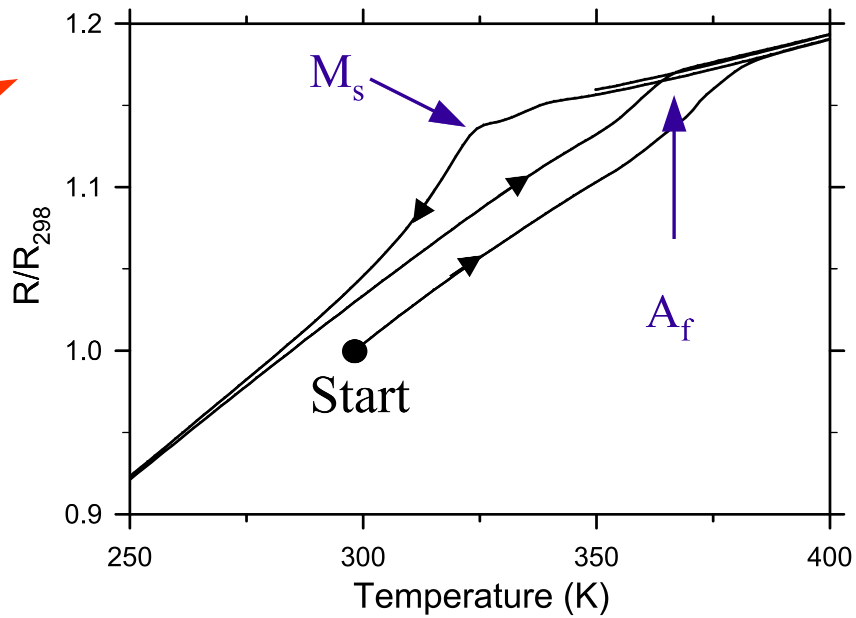


No R or B19’ in 50.7, 52.6, 54.5, 54.6 Ni

Resistivity

48.9 Ni



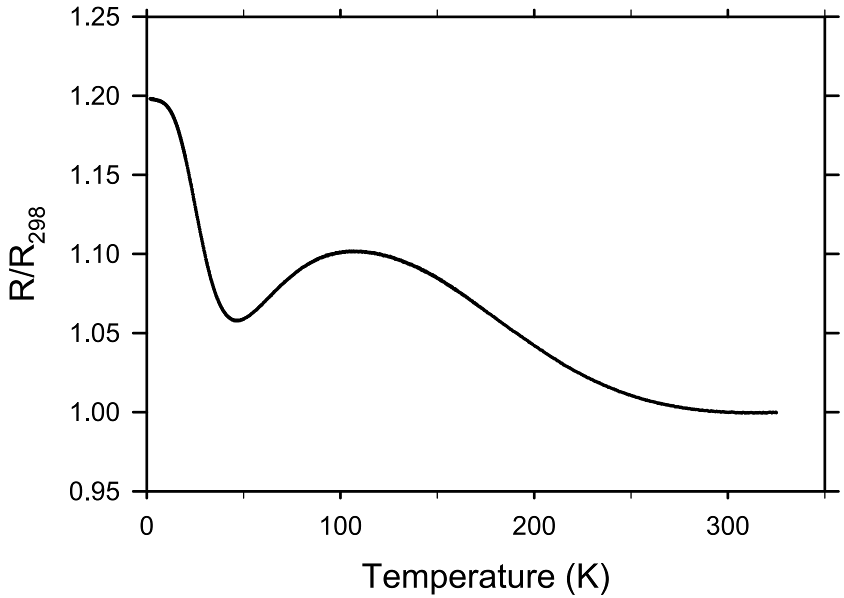


M_s = 323K (50°C)
A_f = 363K (90°C)

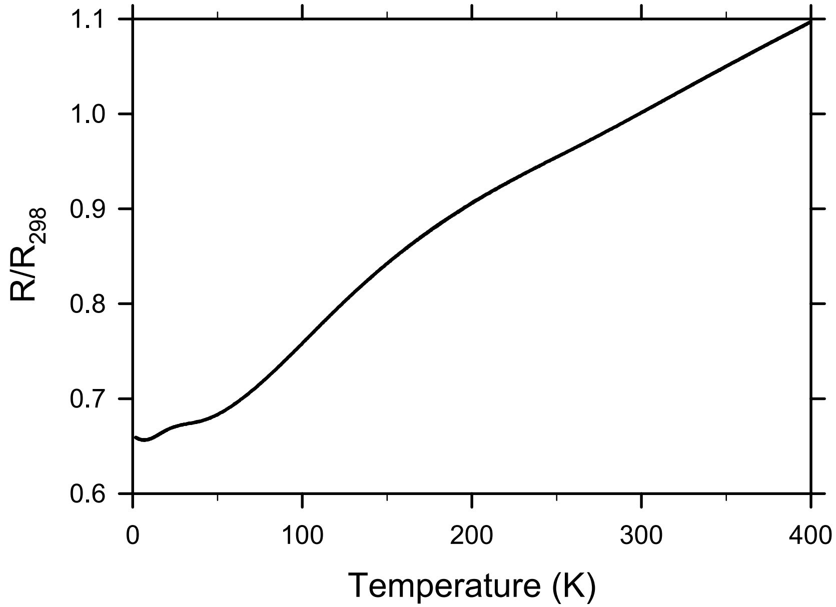
Same hysteresis found by dilatometry and DSC

Resistivity

No Thermal Hysteresis



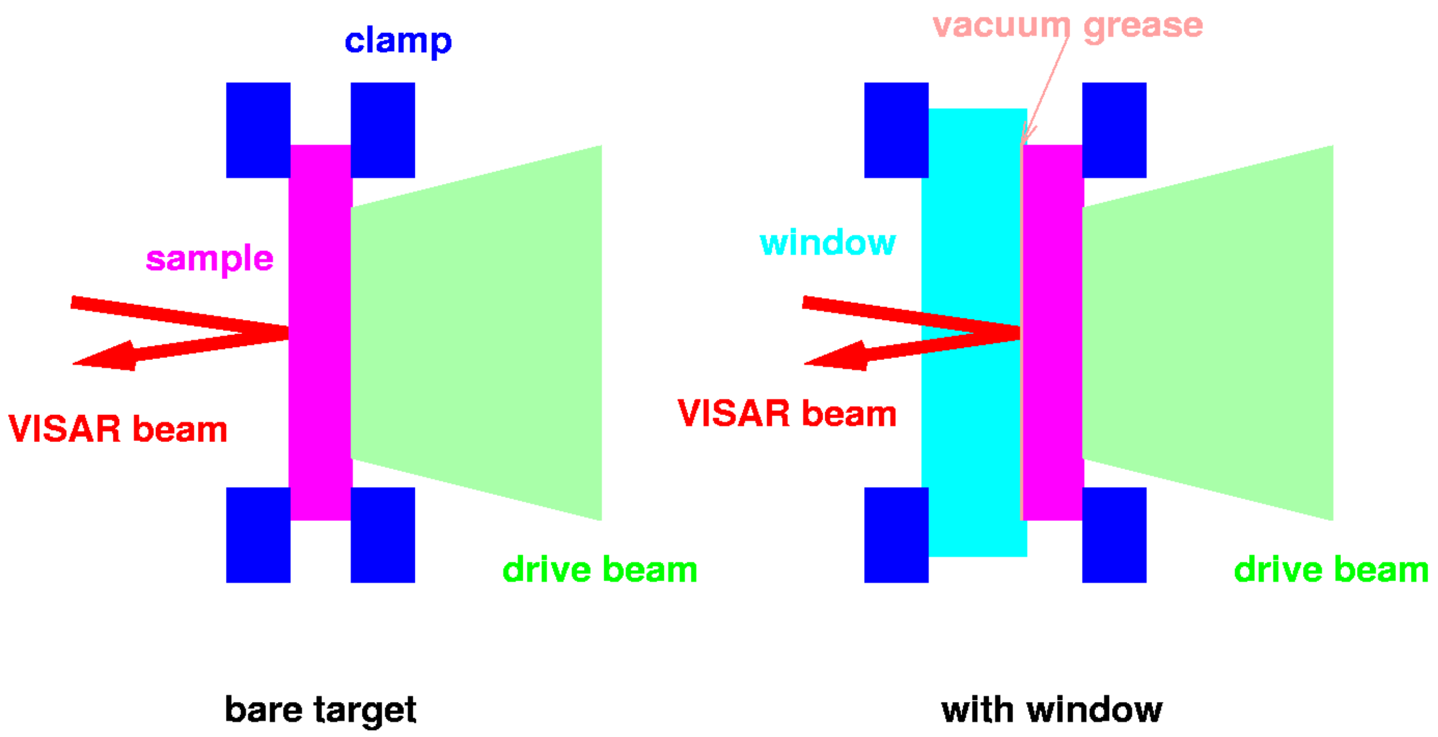
50.7 Ni



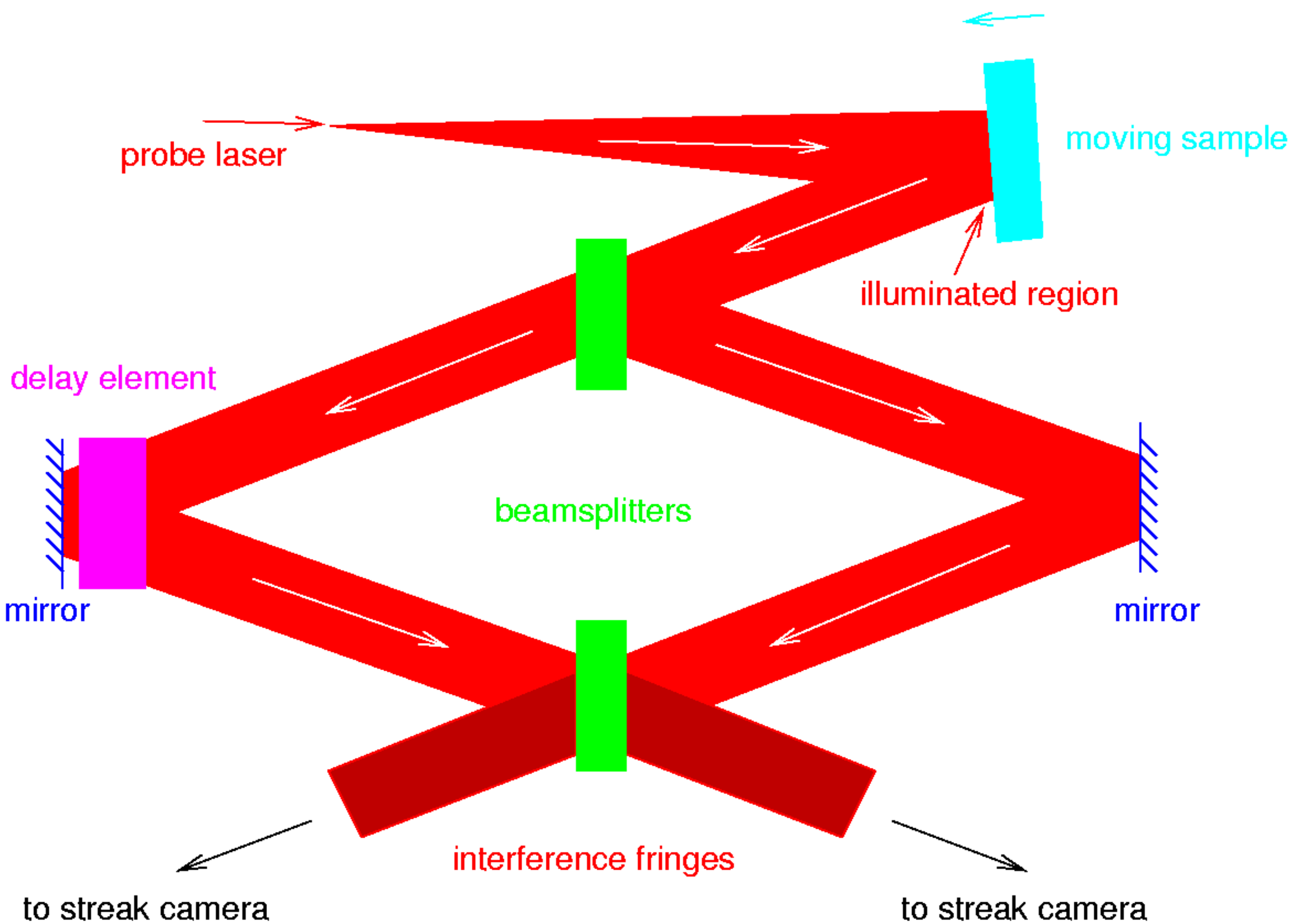
54.5 Ni

No hysteresis found over –100 to +1000°C by dilatometry in 50.7, 52.6, 55.4, 54.6 Ni

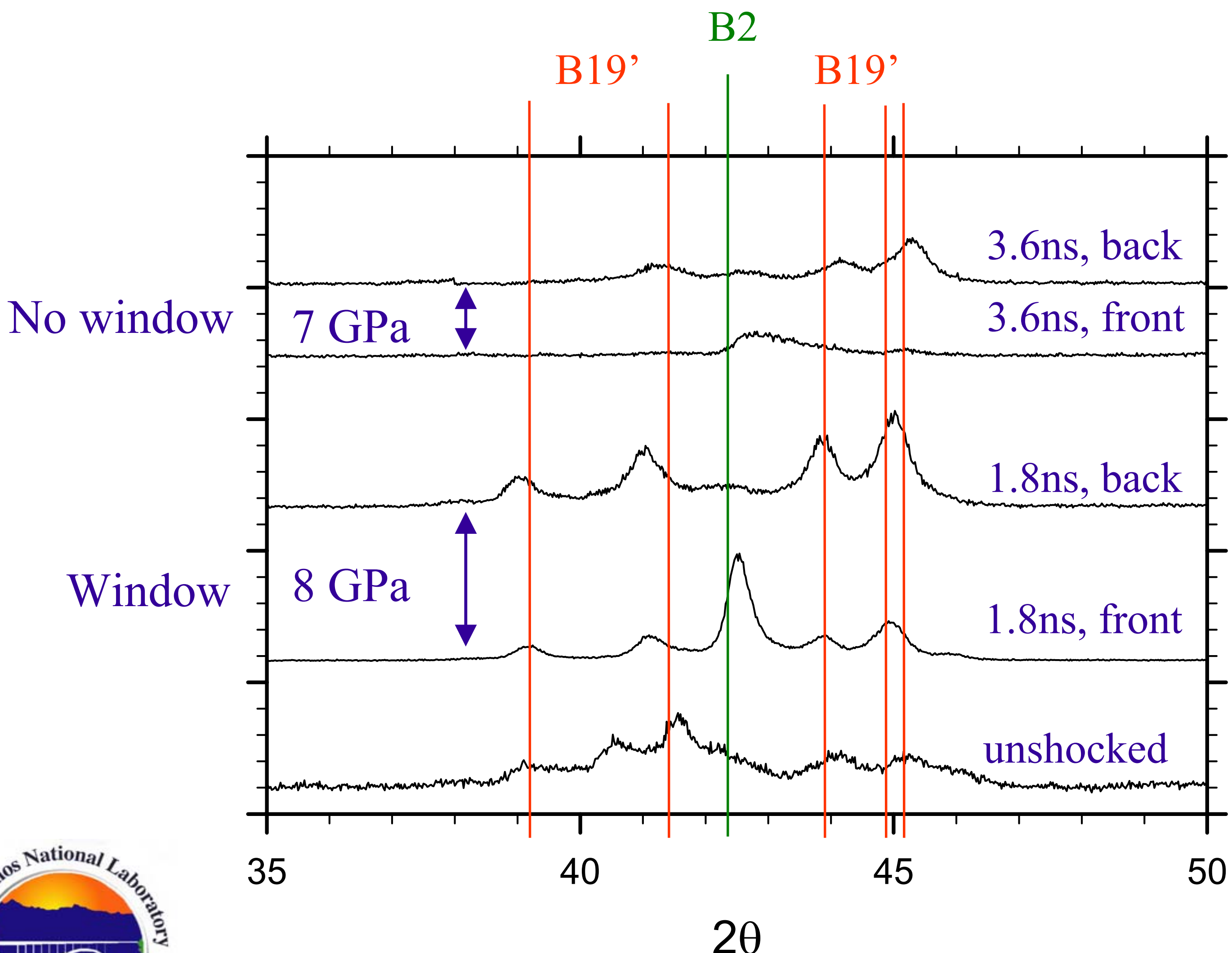
Trident Laser Setup



VISAR Probes



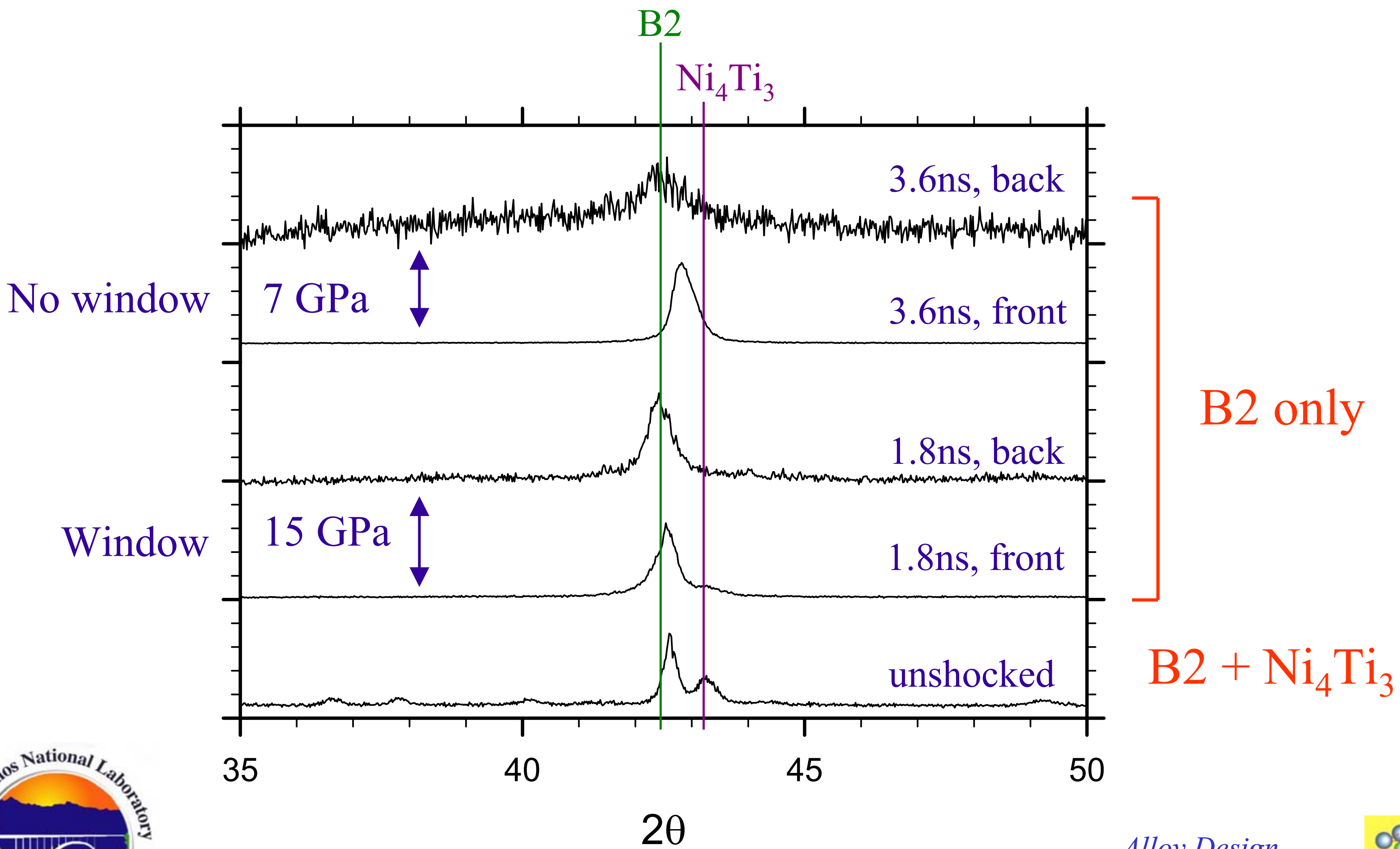
Shocked 48.9 Ni



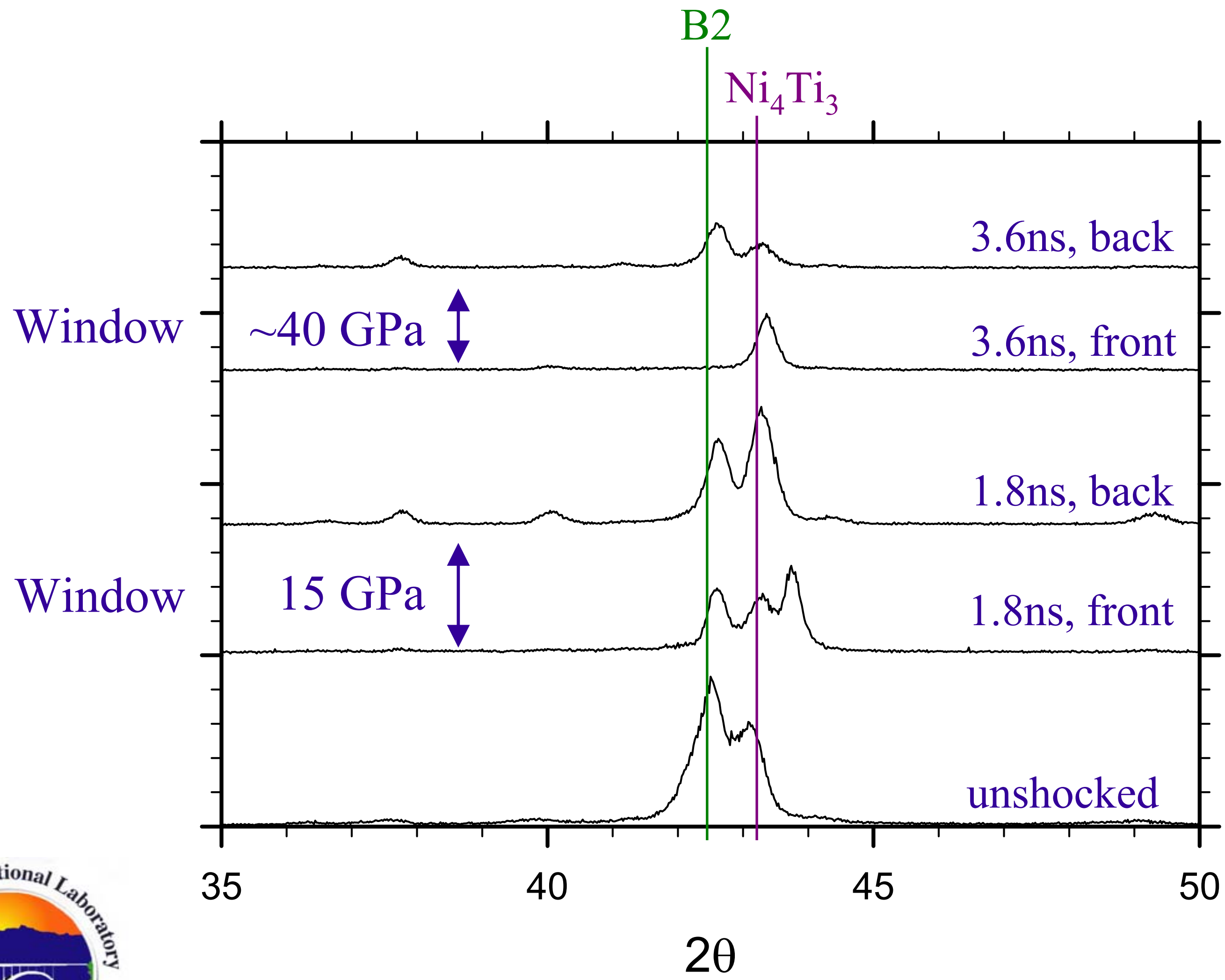
Reverse reaction (B19' ⟶ B2) on front side

Little change on back side

Shocked 50.7 Ni



Shocked 52.6 Ni

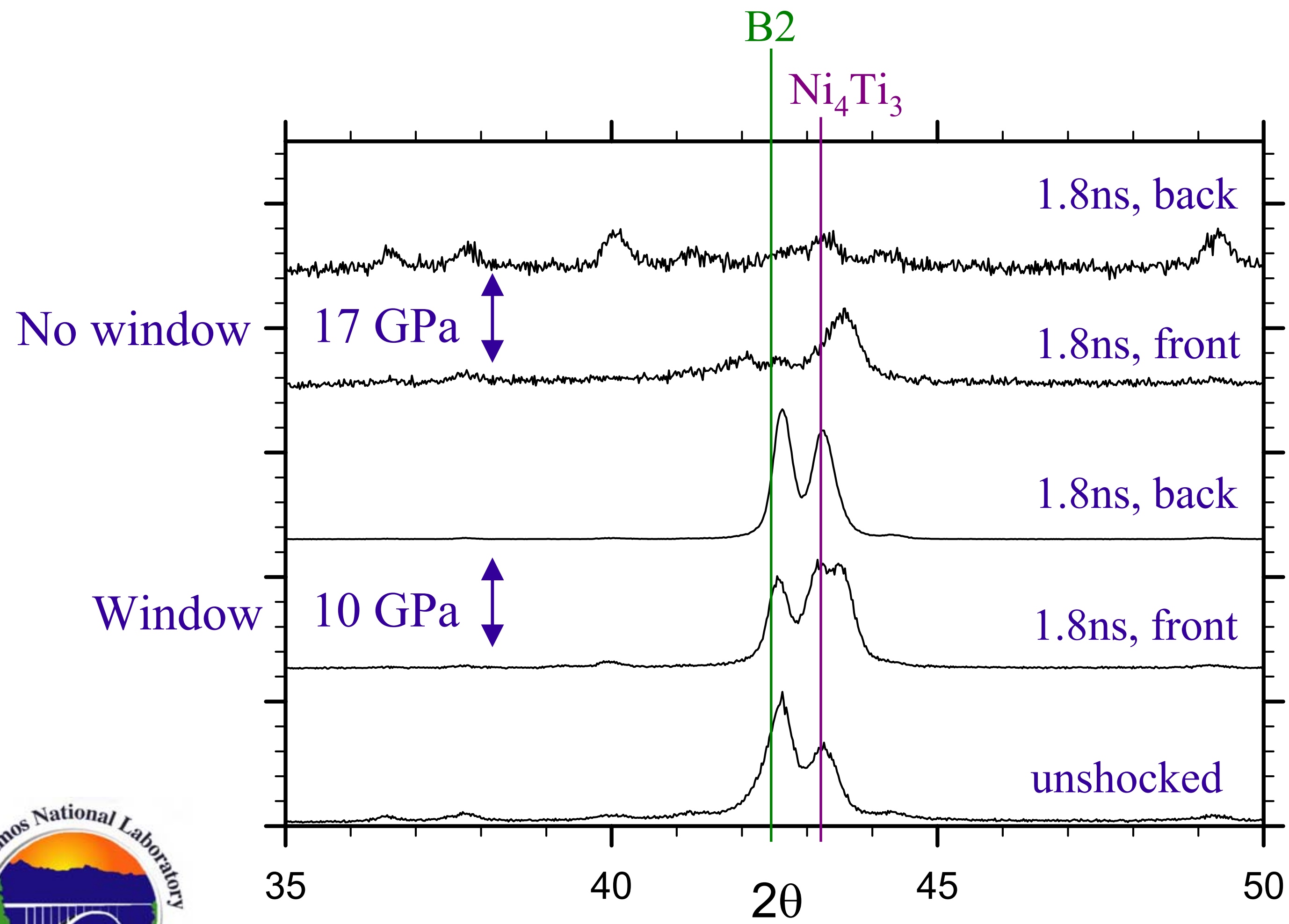


More Ni_4Ti_3

Higher 3.6ns pressure made little difference

$B2 + Ni_4Ti_3$

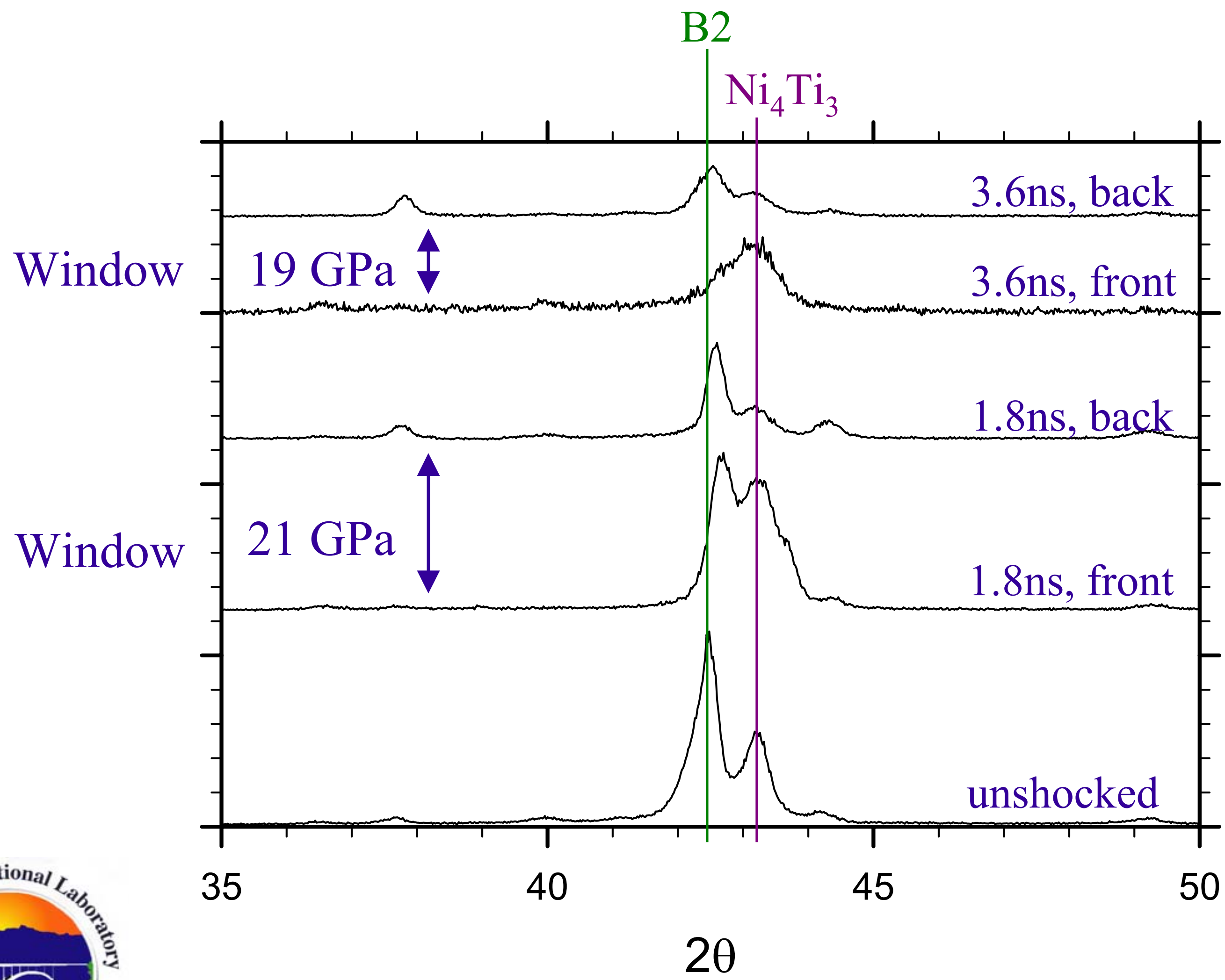
Shocked 54.5 Ni



Without window gives bigger effect than with window at constant time

B2 + Ni_4Ti_3

Shocked 54.6 Ni



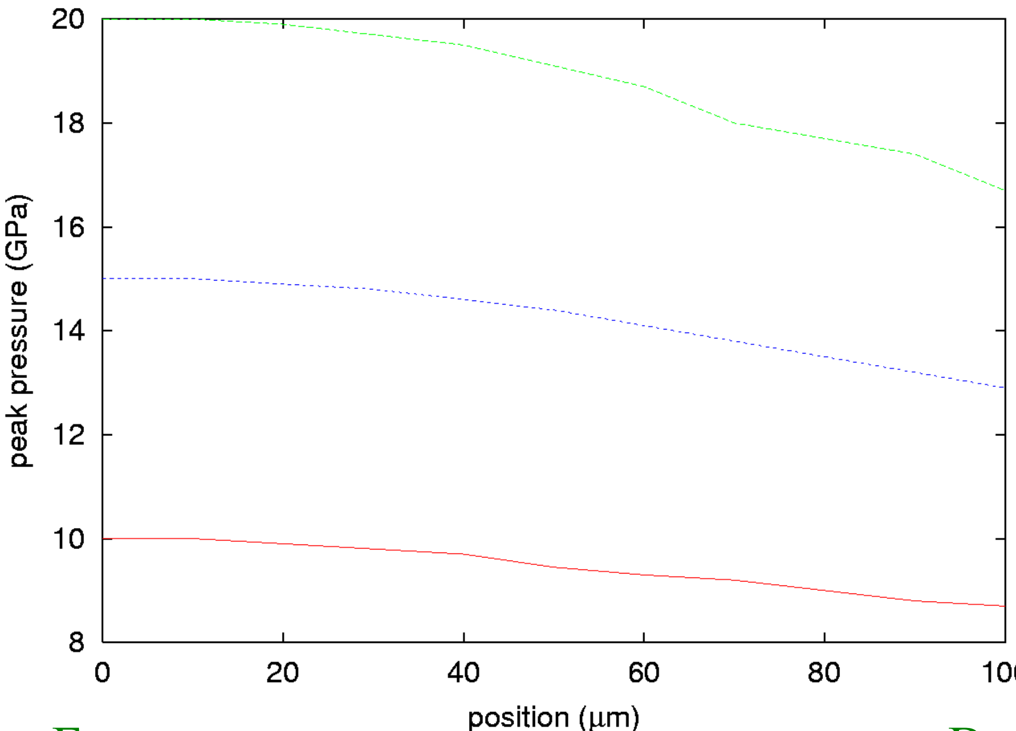
More Ni_4Ti_3 on front side

Little change on back side

B2 + Ni_4Ti_3

Stress-Time Profiles

- Peak compressive stress experienced during initial shock wave transit
- Calculated from EOS data
- Less decay than expected
- Tensile wave is reflected from the back face
  - Much less reflection with the window



Front face

Back face

Results Summary & Interpretation

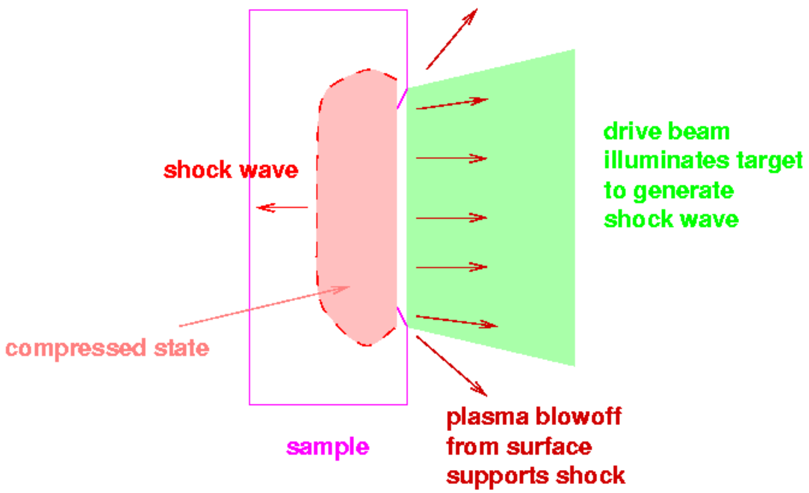
- Martensitic Transformations
  - 48.9 Ni: Reverse transformation: B19’ ⟶ B2
  - Others: No martensite in AQ or shock-loaded
- Ni_4Ti_3 Occurrence
  - AQ: Found in all alloys except 48.9 Ni
  - Shock Loading: decreased in 50.7 Ni, increased in 52.6, 54.5, 54.6
- Stress alone is insufficient to explain these results
- Thermal spike in shocked region can be important
  - Temp. calculated to rise to 300-350^{o}C for 10-20 GPa
  - Formation of B2 (48.9 Ni) and Ni_4Ti_3 (other alloys) can result
  - Expect rise only to 60-80^{o}C for 1-2 GPa (future expts.)
- B2 stabilized (48.9 Ni) due to shock-induced defects

Equation of state for NiTi

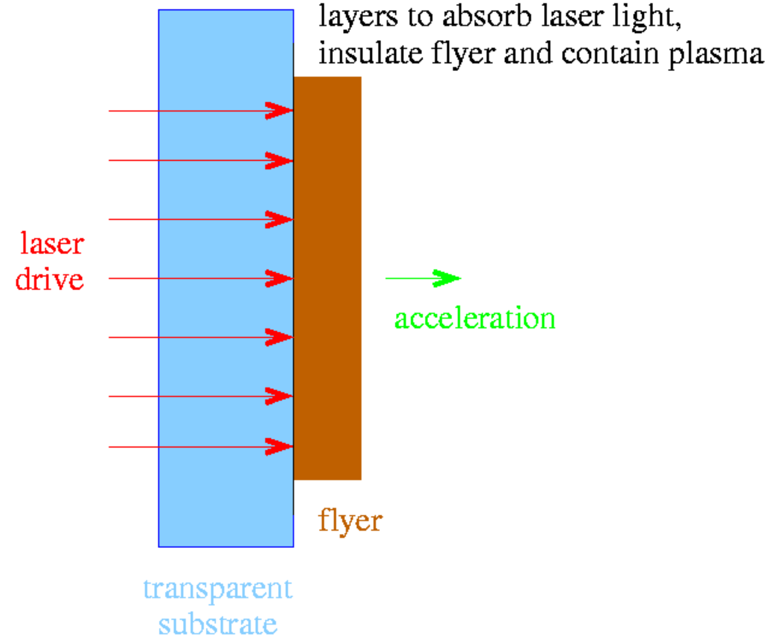
- Necessary to help interpret direct drive experiments
- Theoretical equation of state
  - Ab initio quantum mechanics (cold curve only)
  - Grueneisen model of lattice-thermal contribution
  - Correction to reproduce observed lattice spacing at STP
- Flyer impact experiments to measure Hugoniot

Laser-generated shocks at TRIDENT

Direct drive



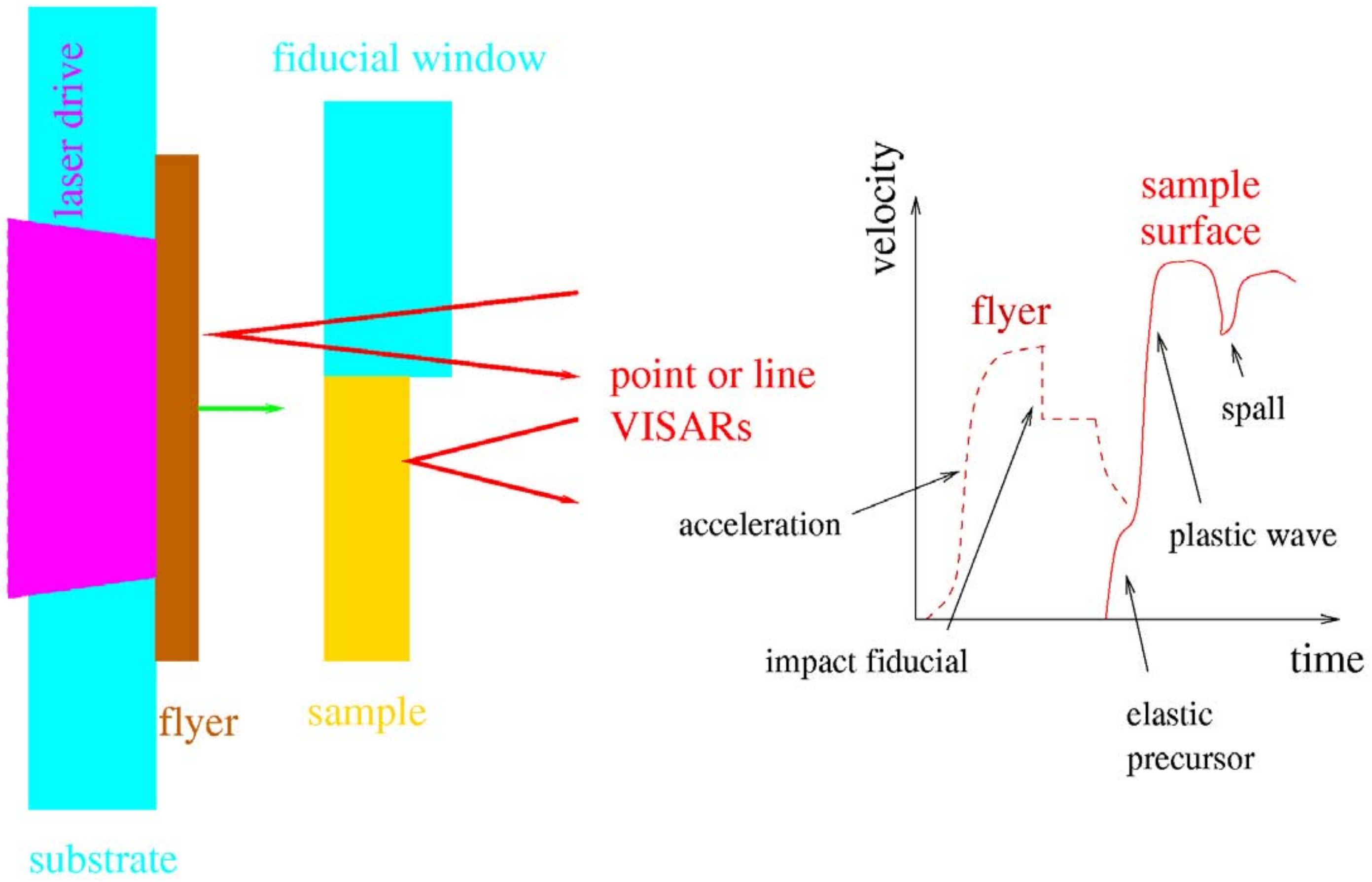
Laser-launched flyer plate



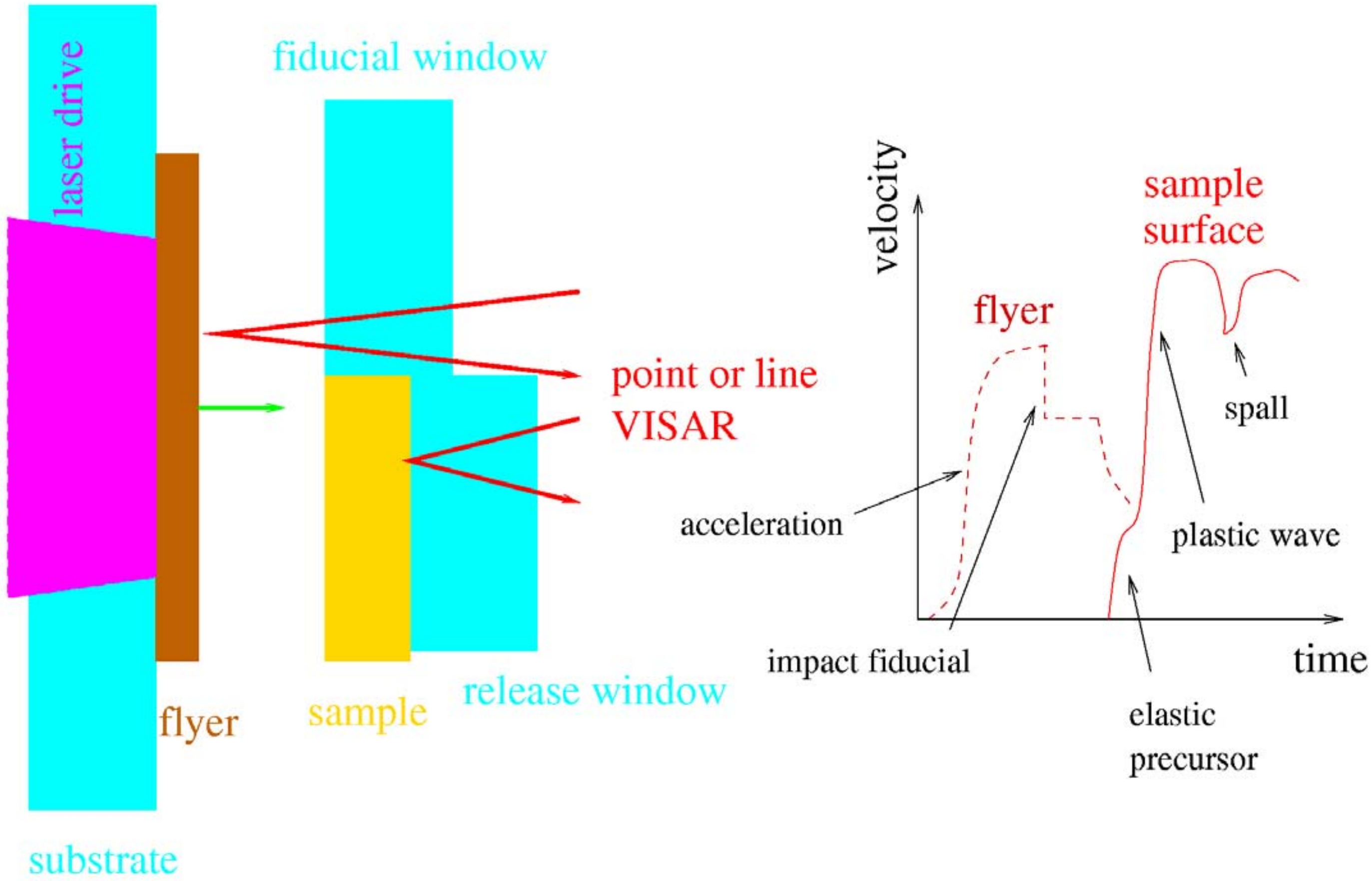
TRIDENT: flexible intensity profiles
~1 to 1000 J, ~0.2 to 500 ns
3 beams
~10 shots / day

Lasers can fill a niche at small scale (single to a few crystals) and pressures at or above gas gun and explosively-driven experiments.

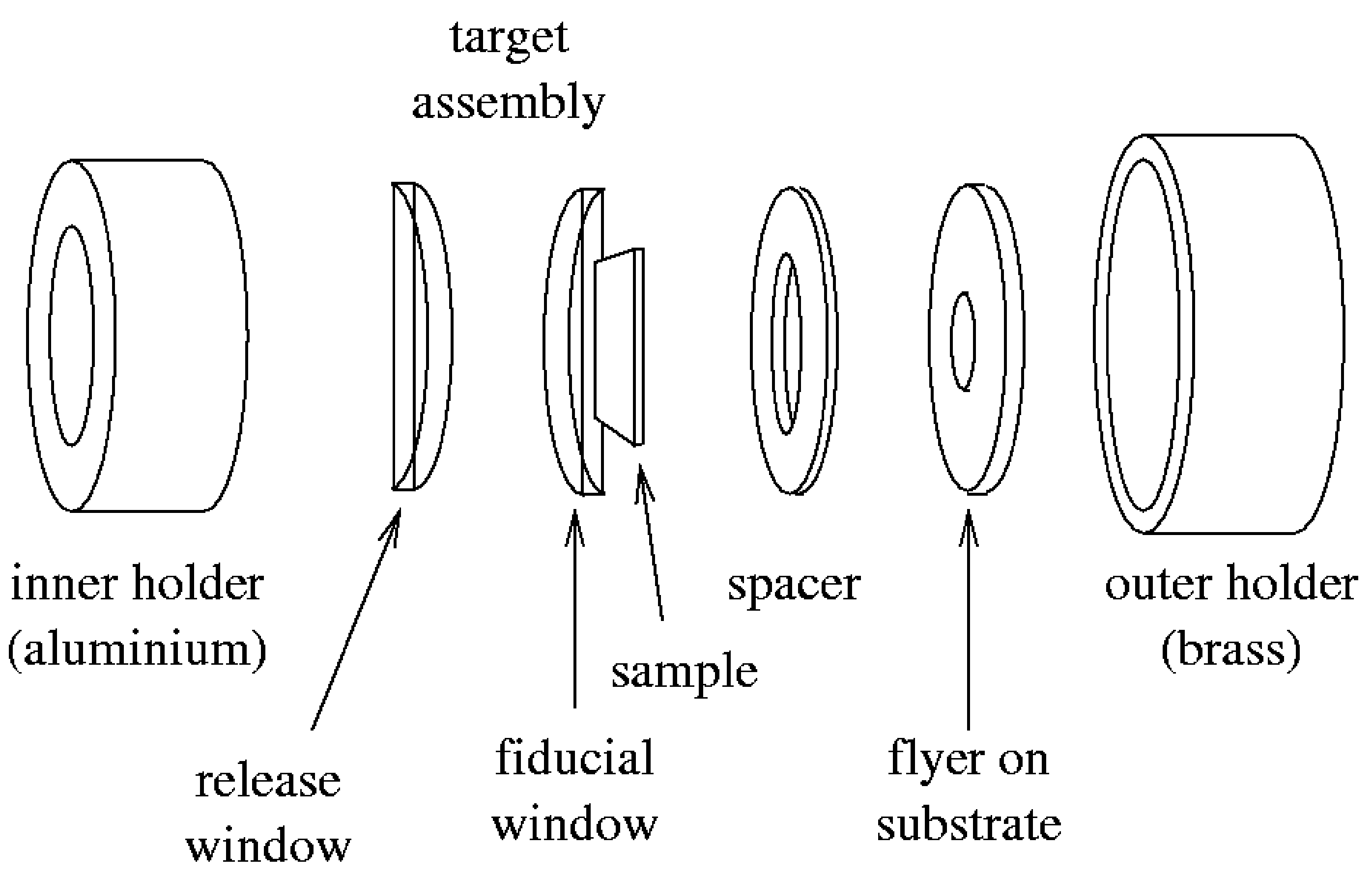
Equation of state / strength: impact fiducial design



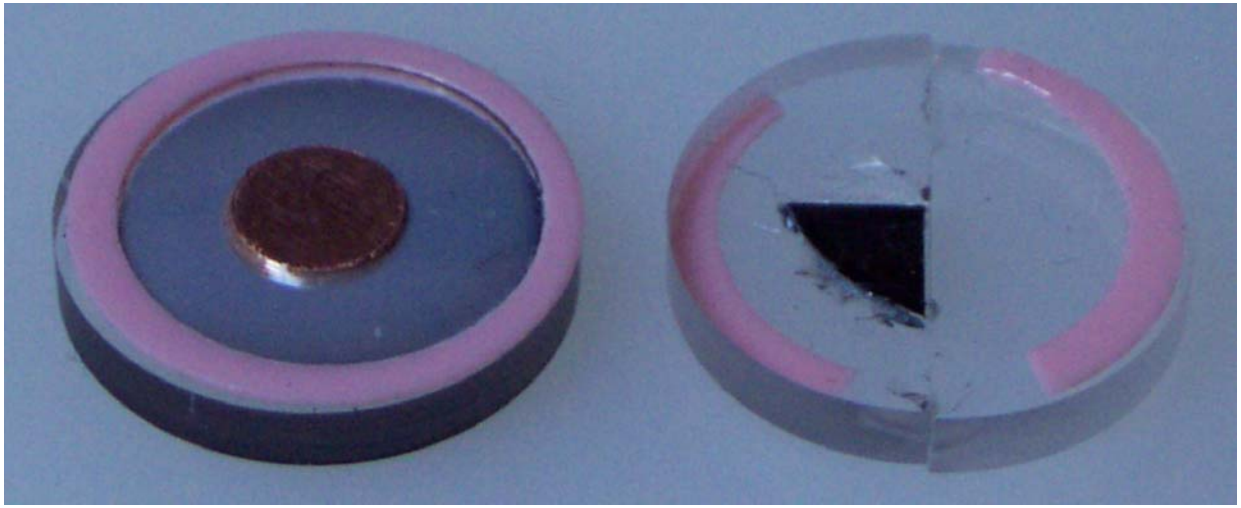
Equation of state / strength: step-window design



Step-window: target assembly

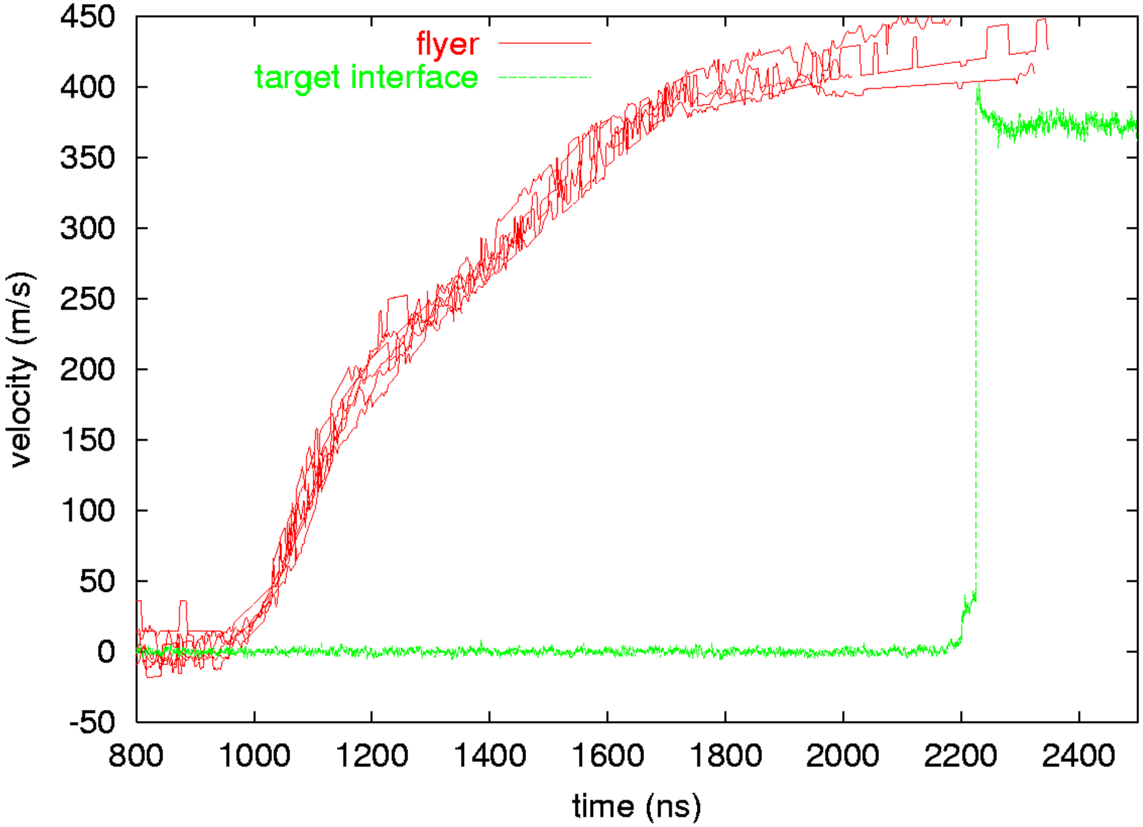


Step-window assembly



TRIDENT shot 14143

Acceleration and shock records



Drive pulse to ~600 ns

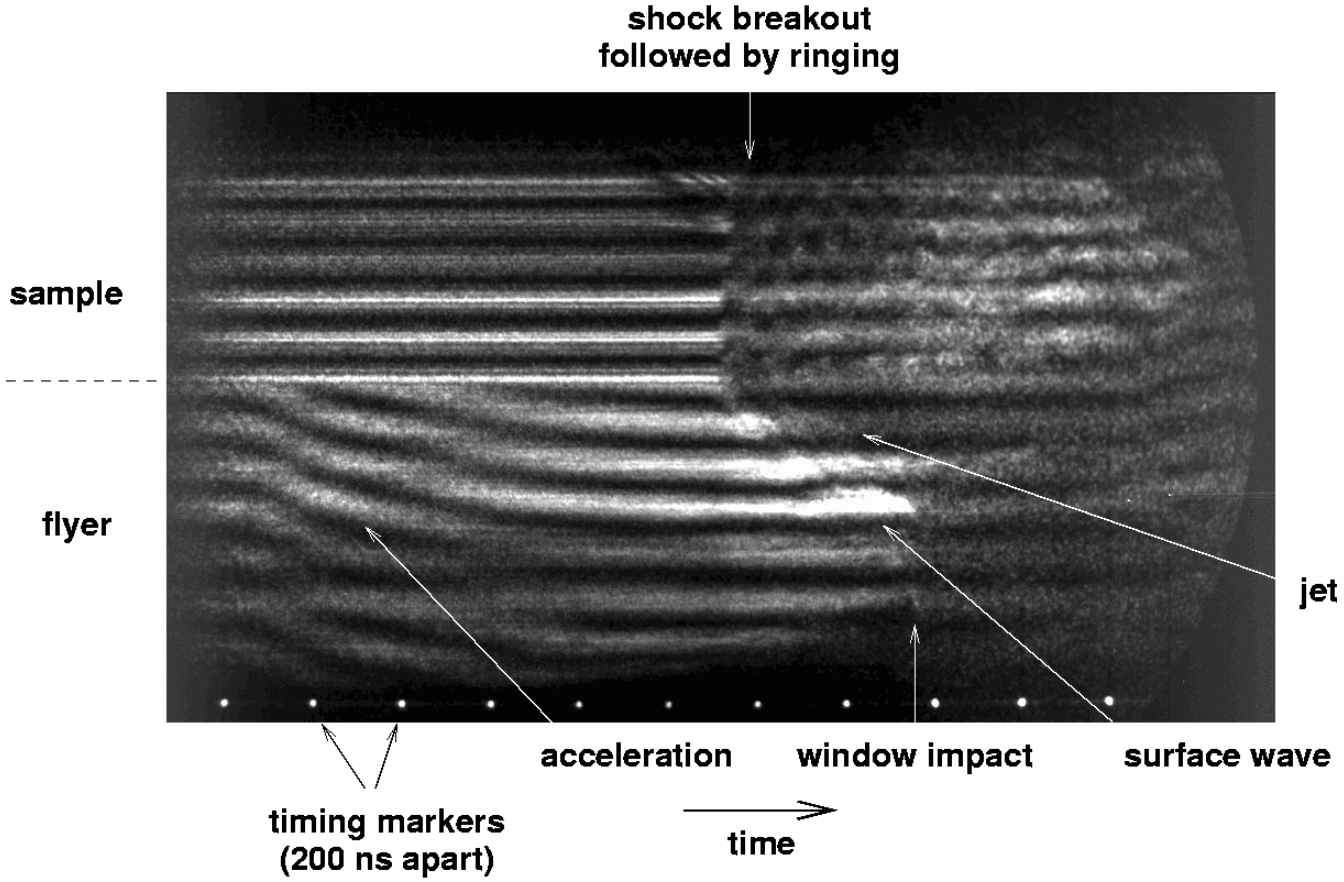
Flyers up to ~300 μm thick

Several hundred m/s with a few J.

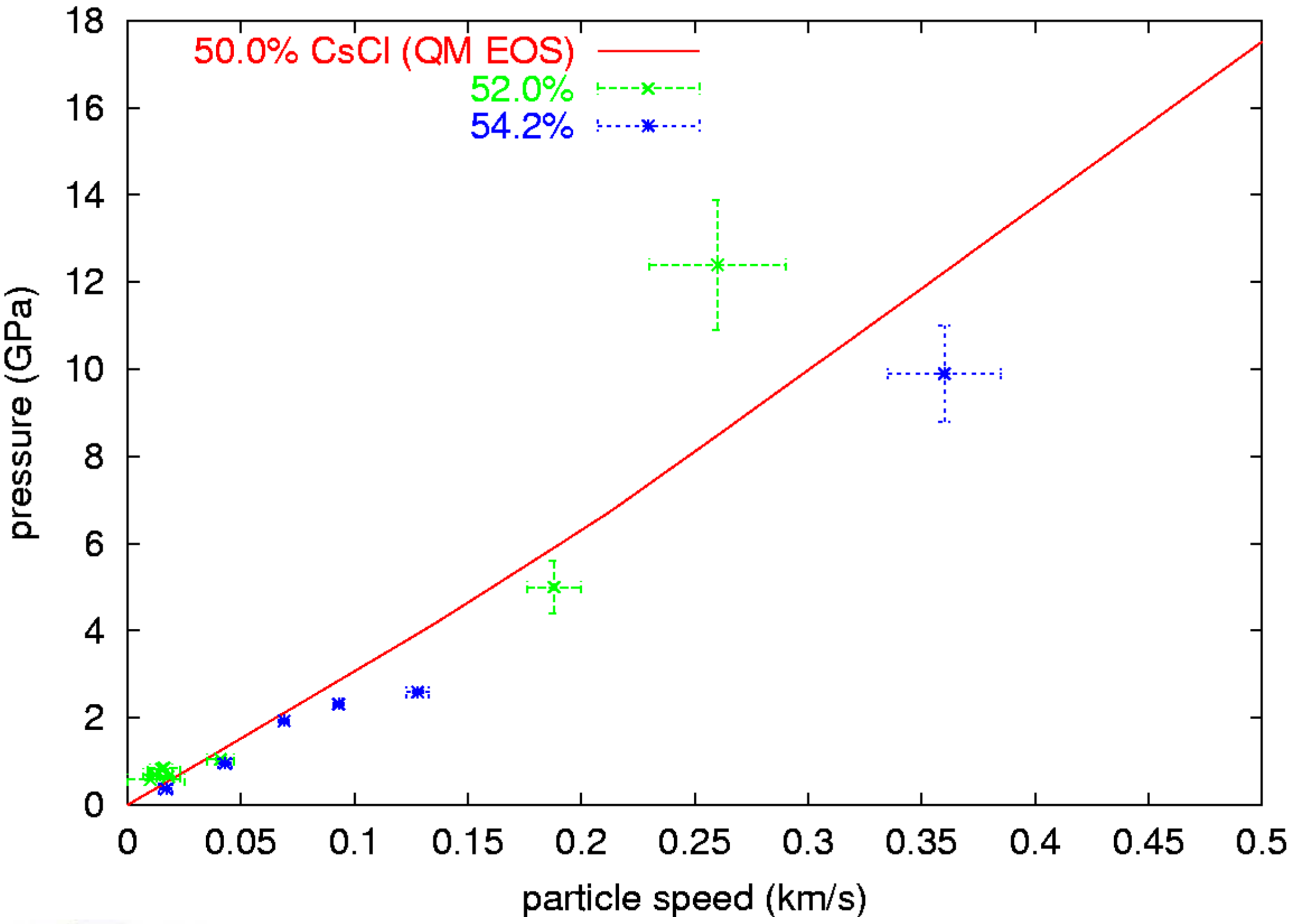
Point + line VISAR
⇒ EOS, strength, spall, phase boundaries.

Example:
TRIDENT shot 14123
NiTi flyer, NiTi target, PMMA window

Line VISAR records whole experiment



Shock Hugoniot for NiTi alloys

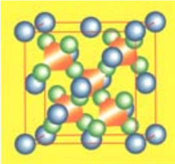


EOS data for NiTi samples, different compositions (atomic %).

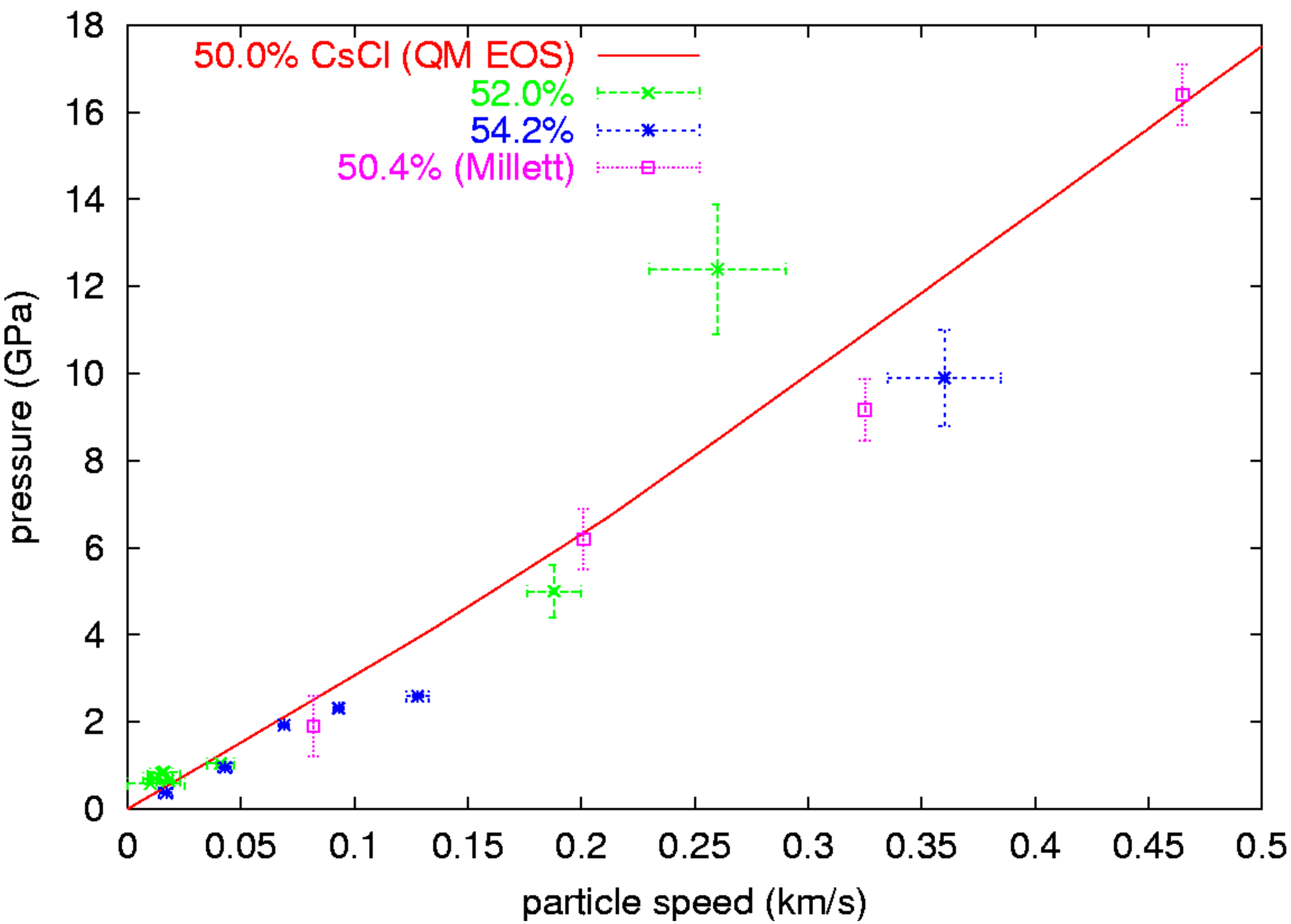
From TRIDENT flyer shots, Dec 01 and Mar 02.

Technique evolving and improving.

Obtained data for Cu, in agreement with published EOS.



Shock Hugoniot for NiTi alloys



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