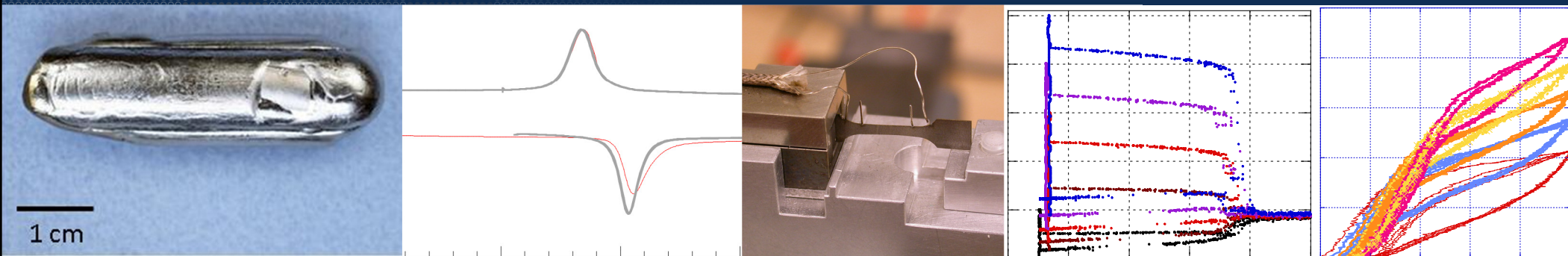




Exceptional service in the national interest



Shape Memory Effect and Superelasticity in a Ti-rich NiTiPt High Temperature Shape Memory Alloy

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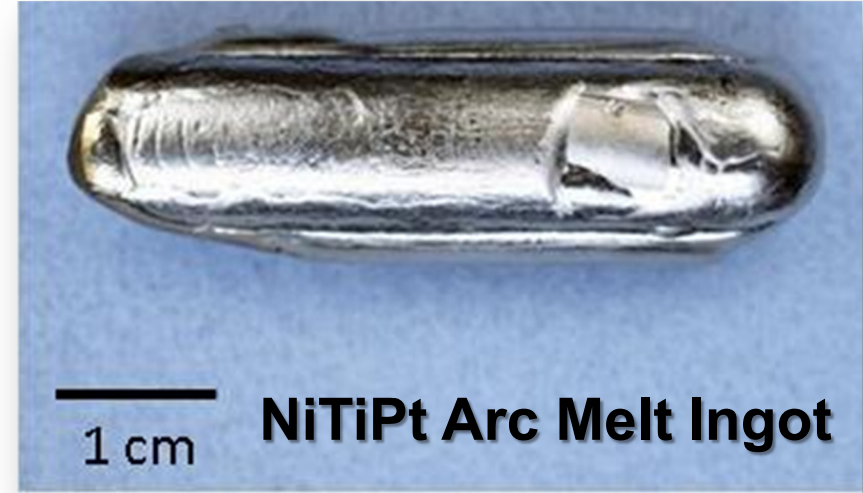
High Temperature SMAs

- Shape memory alloys (SMAs) that exhibit ***shape memory effect and superelasticity at high temperatures*** will enable a new class of mechanical sensors and actuators.
- Substitutional replacement of Ni with Pt, Pd in NiTi can increase shape memory transformation temperatures.
- Pt replacement can increase transformation temperatures up to 1000 °C (NiPt).
- Previous studies have focused primarily on alloys with ≥ 20 at.% Pt and transformation temperatures > 250 °C.

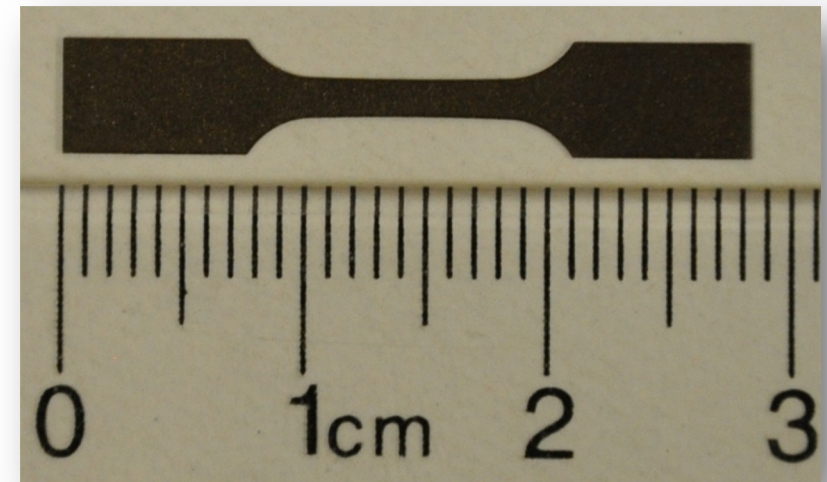
Desired Transformation Temperature Range
150-250 °C

Fabrication

- Targeted series of Ti-rich NiTiPt ternary alloys.
- Ternary alloys processed via **arc melting**.
- Followed by **homogenization**.
- Electro-discharge machined (EDM) mini-coupons to enable tension tests with available material.

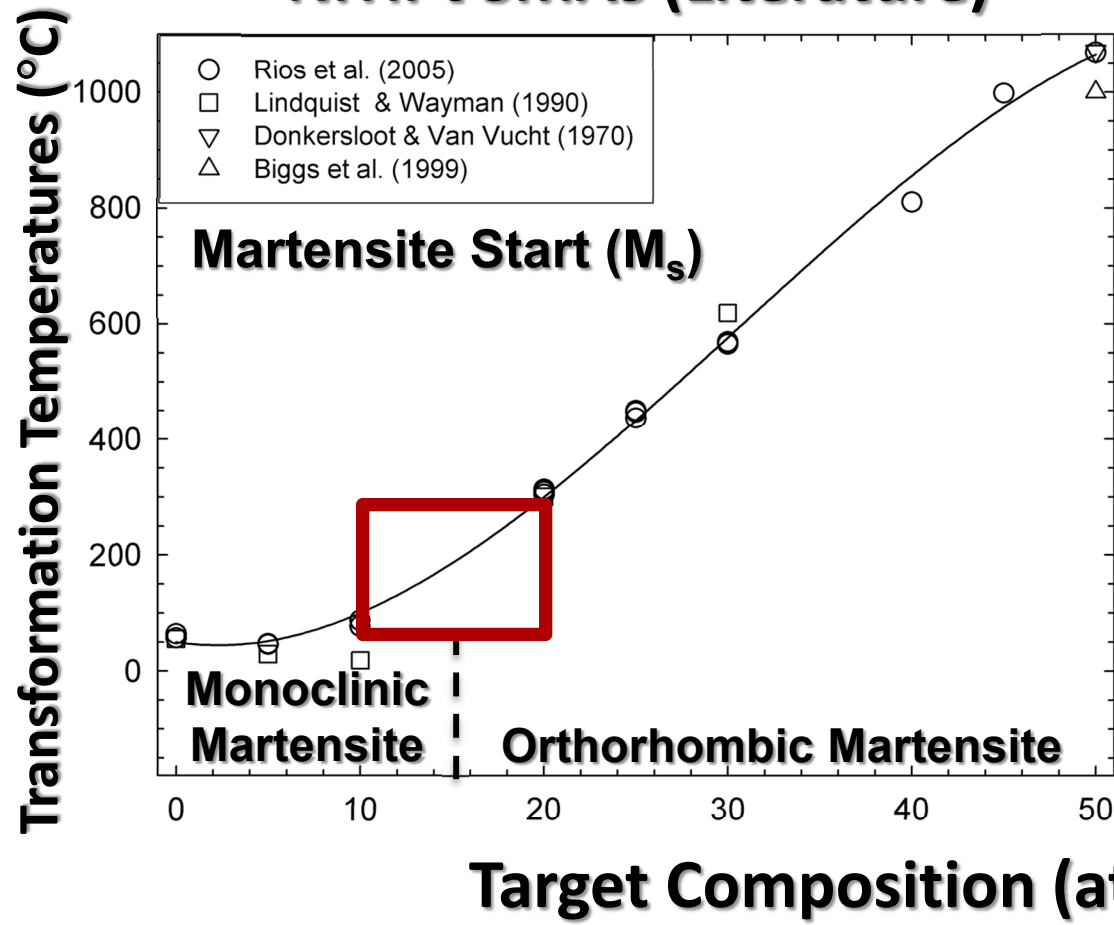


Target (at.% Ti)	Ingot Mass (g)	Homogenization
50.5	40	1050 °C, 60 hrs

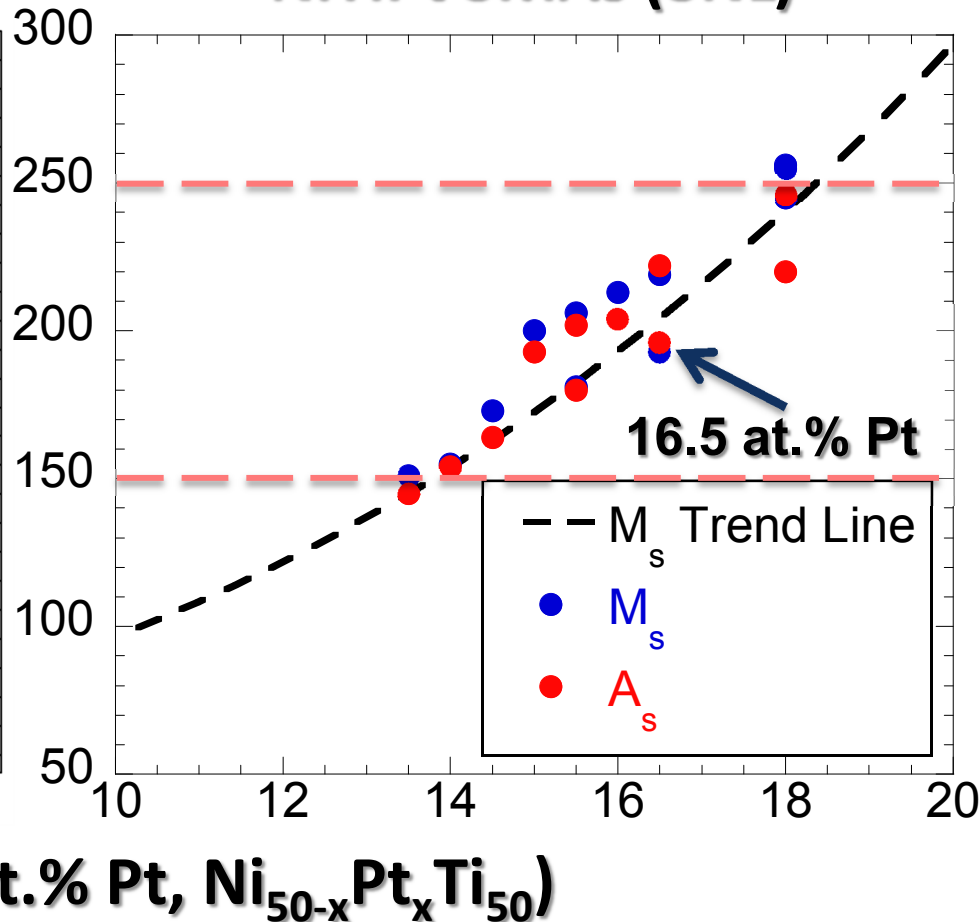


Choosing a Composition Based on A_s

NiTiPt SMAs (Literature)



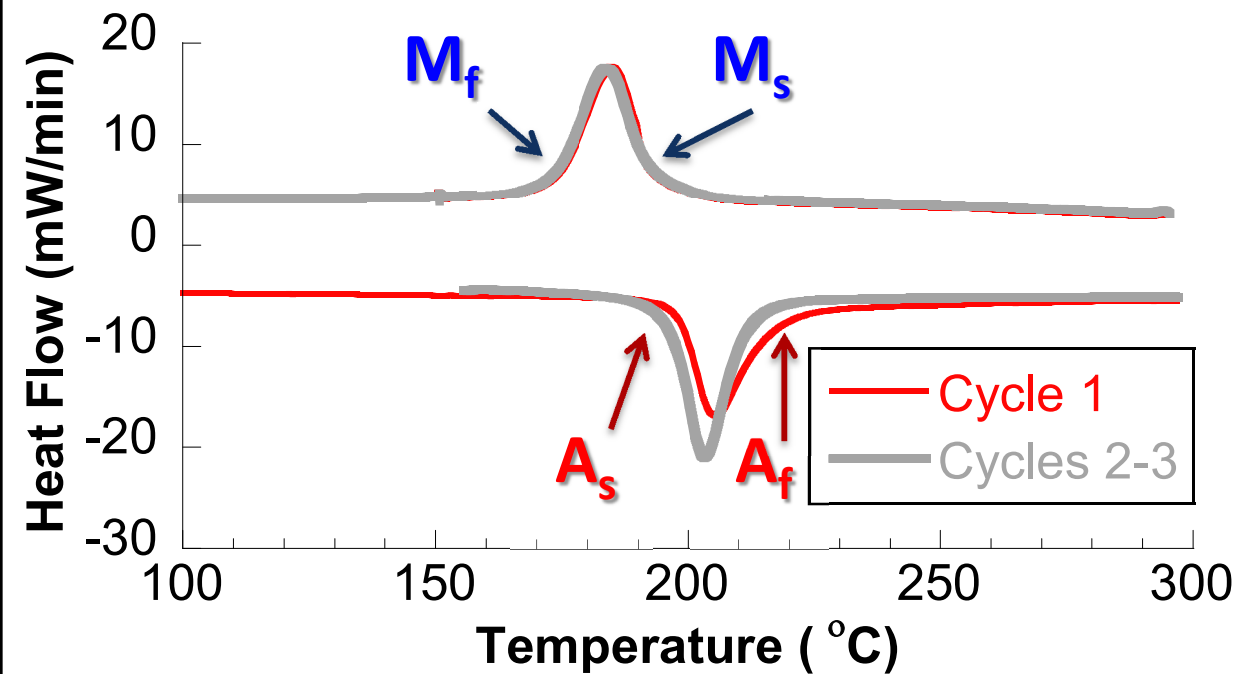
NiTiPt SMAs (SNL)



150-250 °C Transformation Temperatures = New Range of Compositions

Compositional and DSC Analysis

Target (at.% Pt)	ICP/MS (at.% Pt)	ICP/MS (at.% Ti)	ICP/MS (at.% Ni)
16.5	16.5	50.4	33.1



- Selected 16.5 at.% Pt ingot in center of transf. temperature range.
- Inductively coupled plasma mass spectroscopy (ICP/MS) verified composition.
- Differential Scanning Calorimetry (DSC) gives baseline transformation temperatures.
- DSC trace stabilizes in 2-3 thermal cycles.

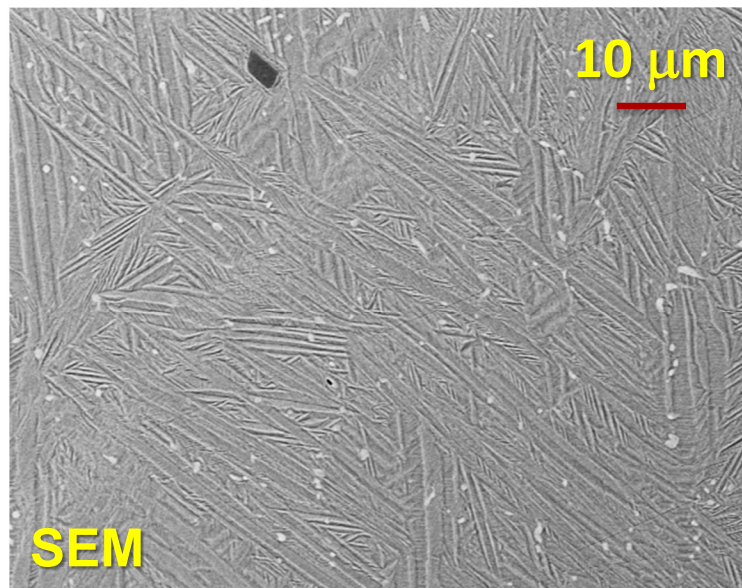
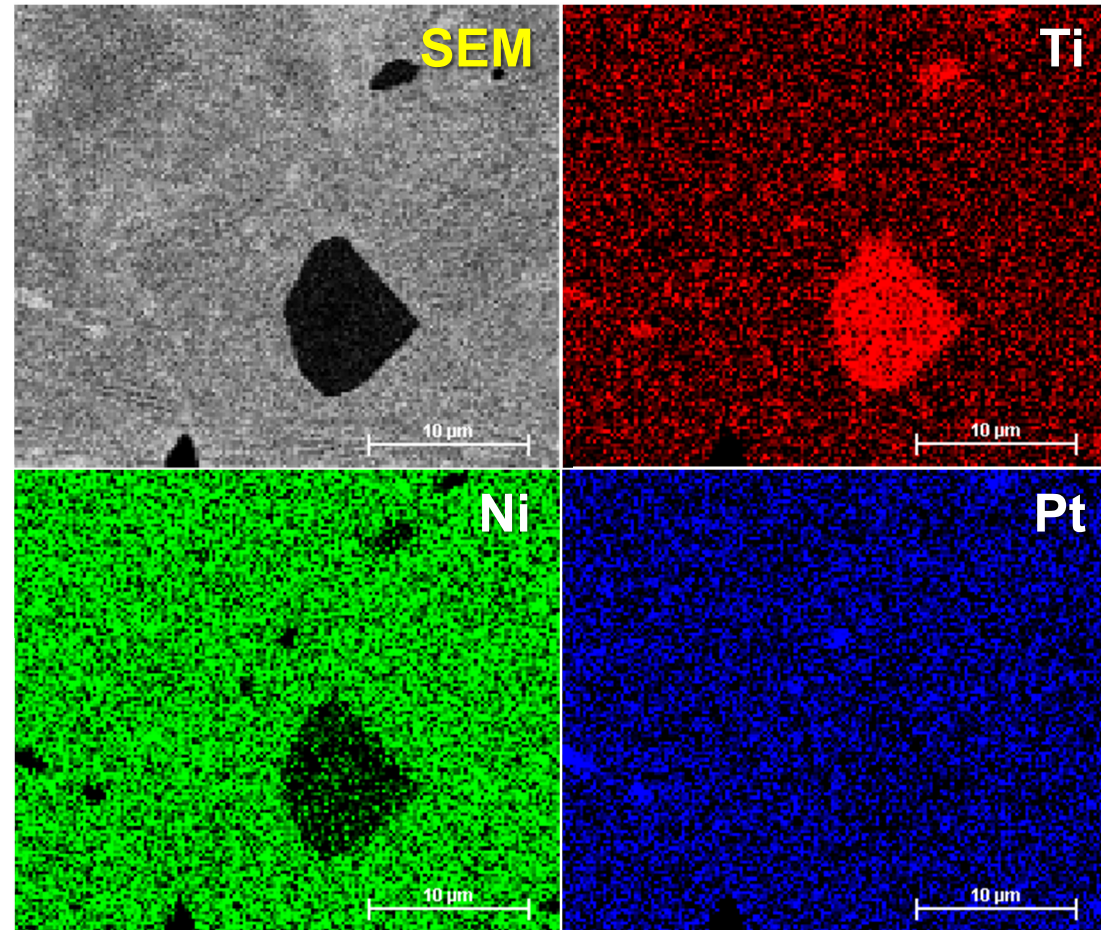
Martensite and Austenite

Start & Finish
Transformation
Temperatures

M_s (°C)	M_f (°C)	A_s (°C)	A_f (°C)
193	173	196	211

Energy-dispersive X-ray Spectroscopy

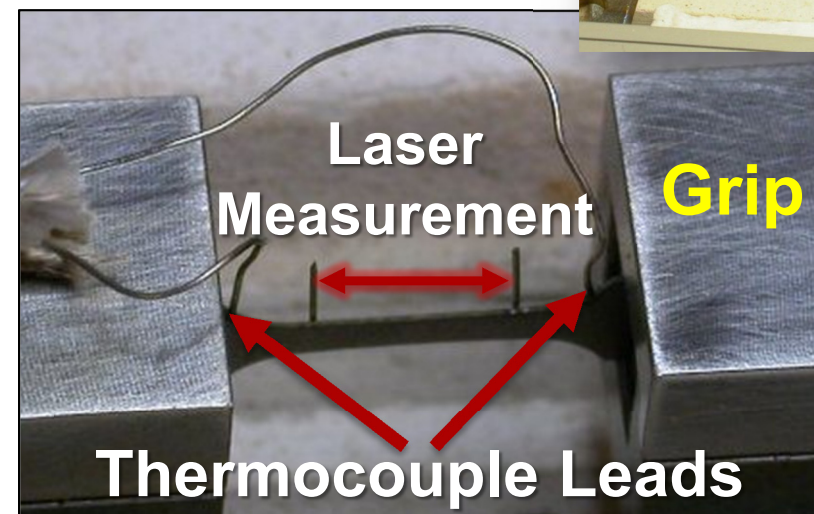
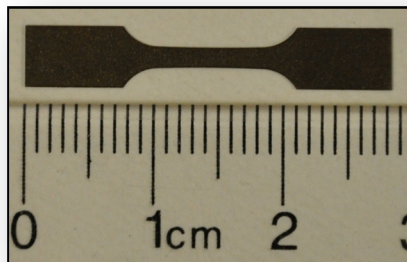
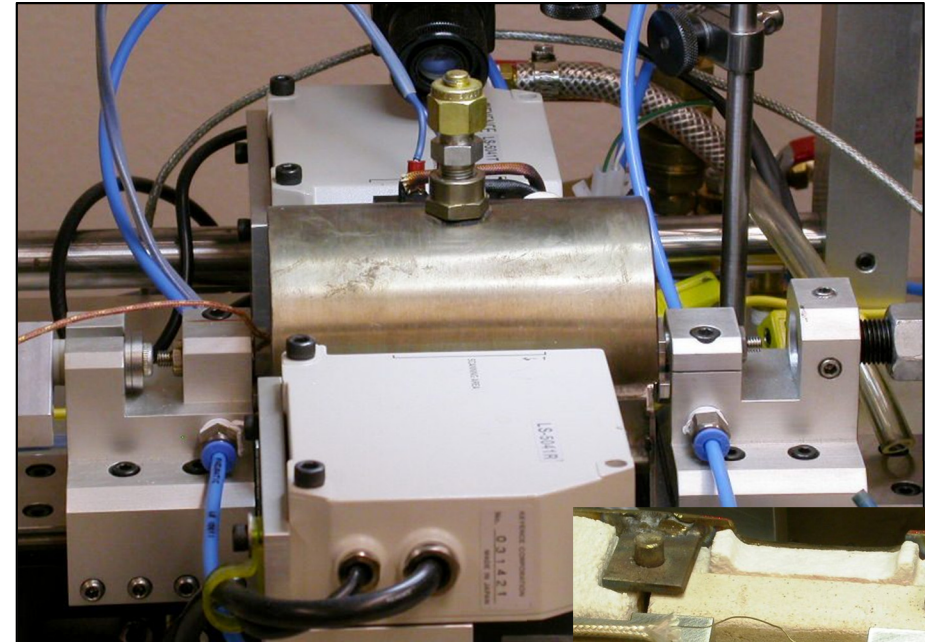
- Dark precipitates are Ti-rich.
- Light precipitates are Ti-rich, slightly Pt-rich, and Ni-lean relative to matrix.
- Light precipitates are possibly $Ti_2(Ni,Pt)$.



- Sample pulled to failure at RT, then grip section imaged.

Mini-tensile Test Setup

- Custom built horizontal servo-hydraulic frame with clamshell furnace.
- Interface 100 lb load cell.
- LS5041R-Keyence laser extensometer measures strain between spot-welded tabs in gauge section.
- Temperature controlled from thermocouple readout in gauge section.



Shape Memory Characterization

How does the new NiTiPt HTSMA recover shape under thermomechanical conditions?

- Shape Memory Effect (SME) Experiments
 - Determine how much tensile strain material can recover under no load via heating through transformation.
 - Study how shape recovery evolves over multiple cycles.
- Superelasticity Experiments
 - Determine if material exhibits large tensile strain recovery at high temperatures via stressing through transformation.
 - Superelasticity has not been demonstrated for HTSMAs in the literature.

SME Experiments

Test Program

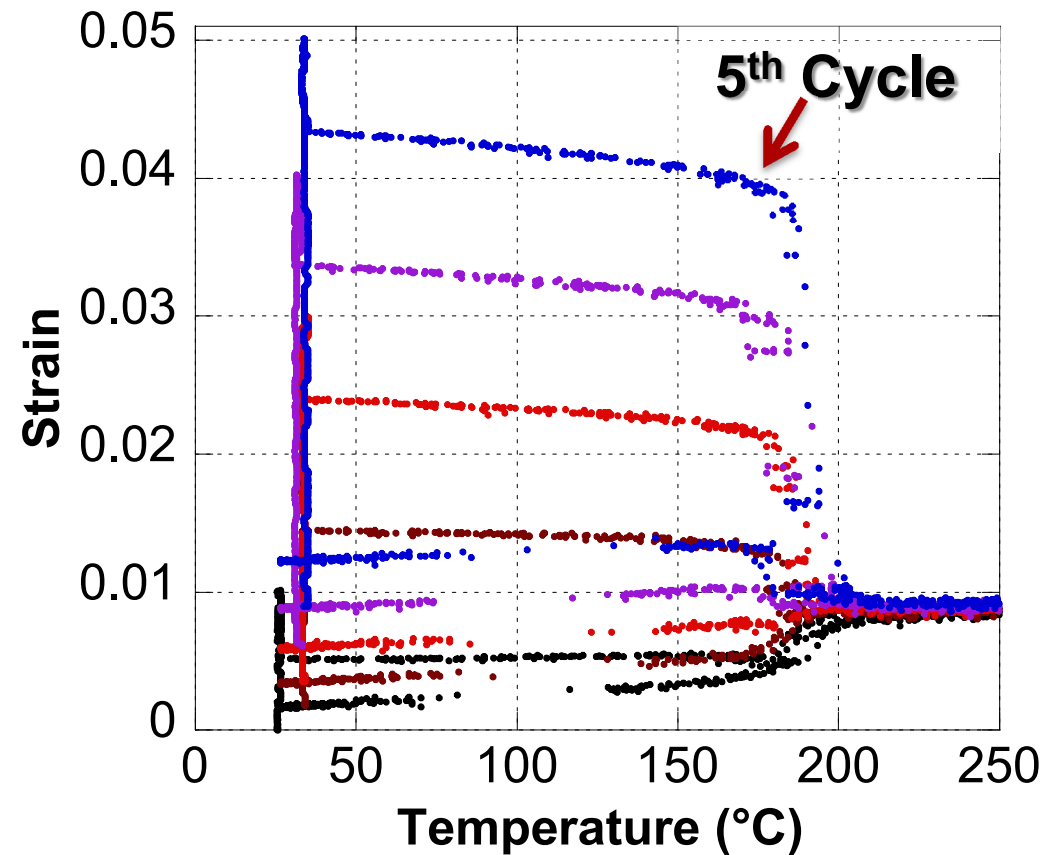
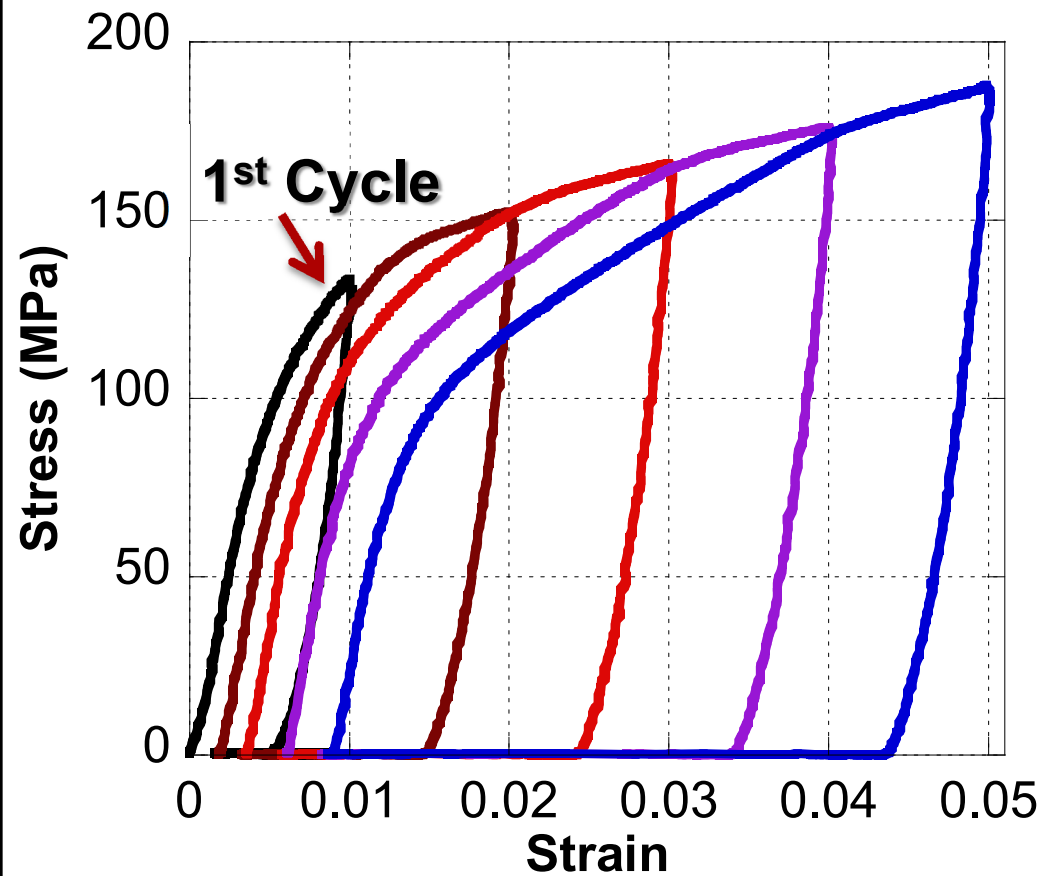
Effective Strain Rate
(1/s)

Heat Rate
(°C/min)

Pull to X% strain (stroke control) @RT; unload to 0 N; heat to A_f+30 °C @0 N, hold; cool.

4×10^{-4}

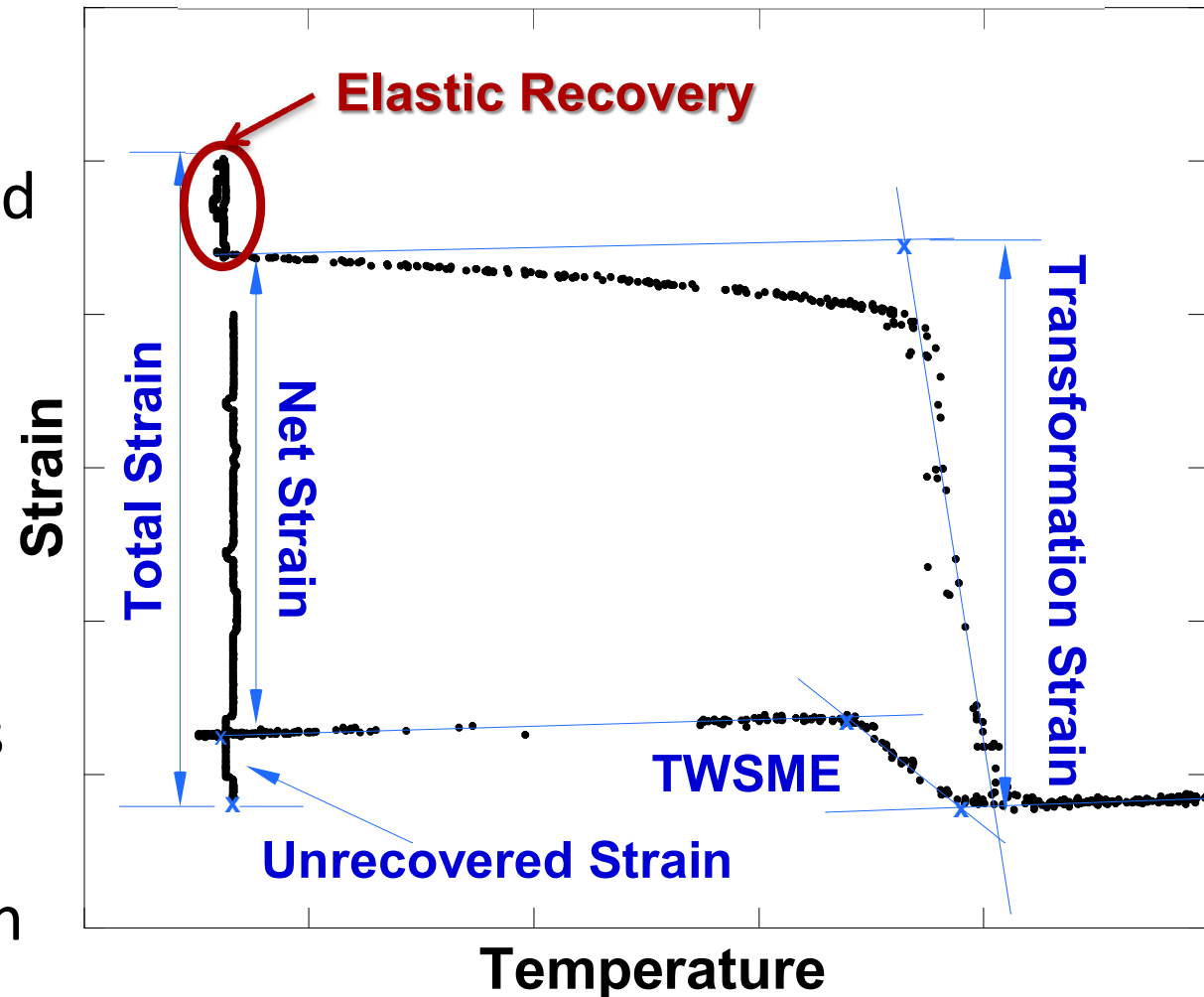
5-10



SME Strain Quantities

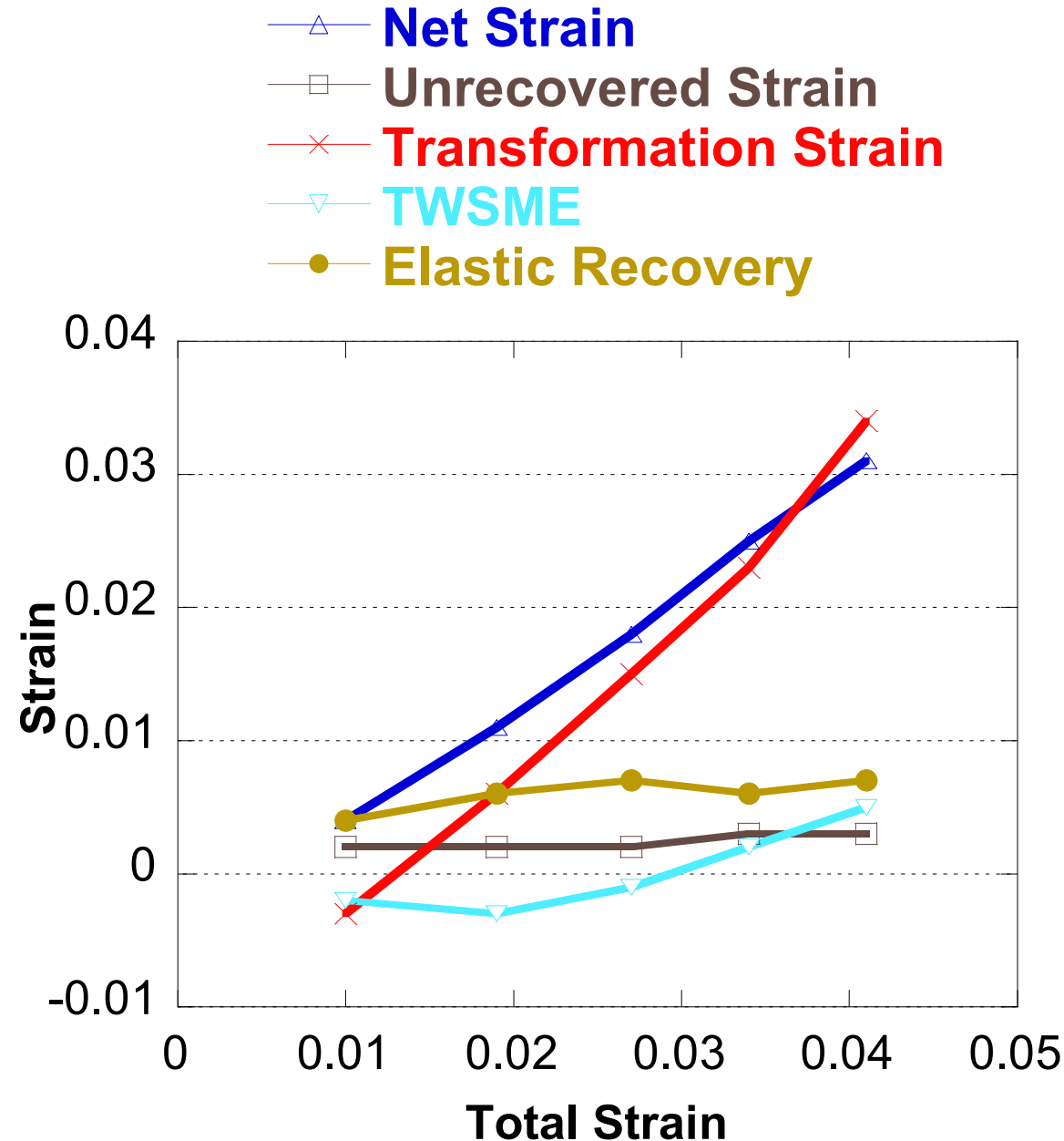
- **Total Strain:** how much the sample stretched under load.
- **Elastic Recovery:** zero-load strain prior to heating.
- **Transformation Strain:** strain recovered during heating.
- **Two-way SME (TWSME):** strain as sample lengthens upon cooling.
- **Unrecovered Strain:** strain remaining after full cycle.

Typical Shape Recovery Curve



Shape Recovery vs. Extension

- Information guides engineers on shape recovery limits of material.
- >2.5% strain recovery observed.
- Transformation Strain** increases as total stroke increases.
- TWSME** strain is near-zero at ~3% Total Strain; *study behavior at this strain level further.*



SME Experiments (Cyclic)

Test Program

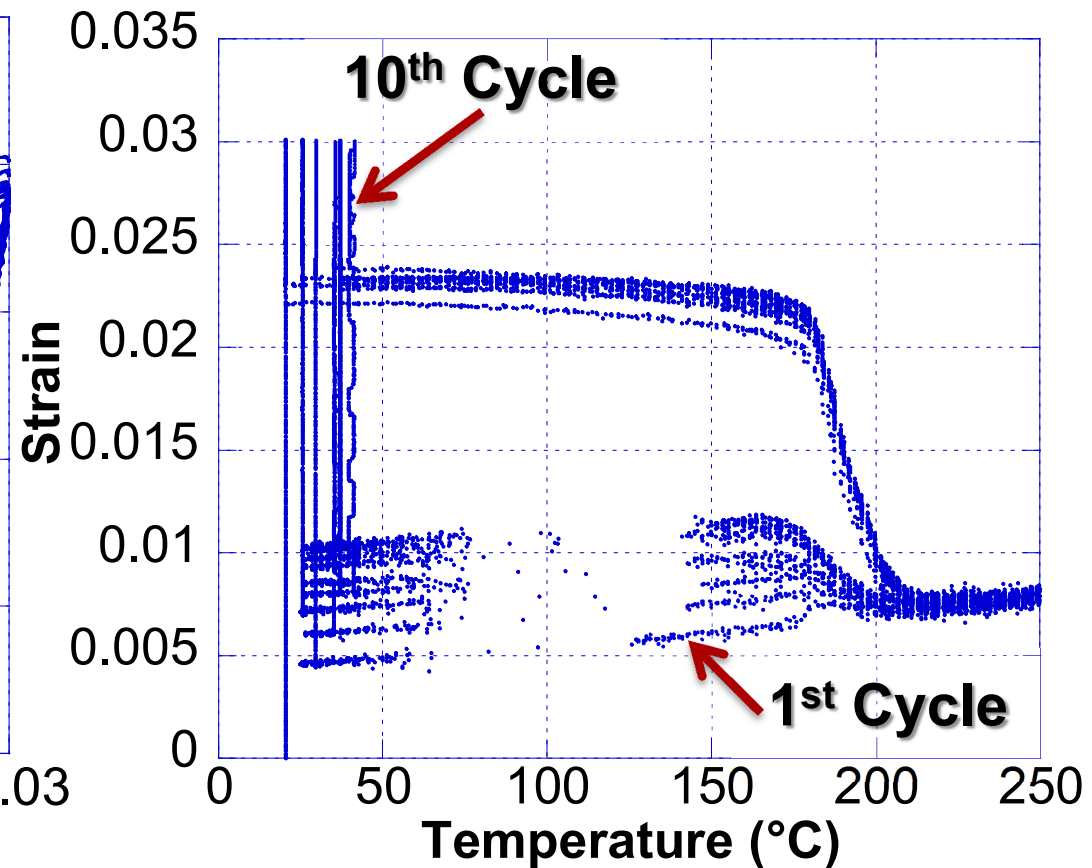
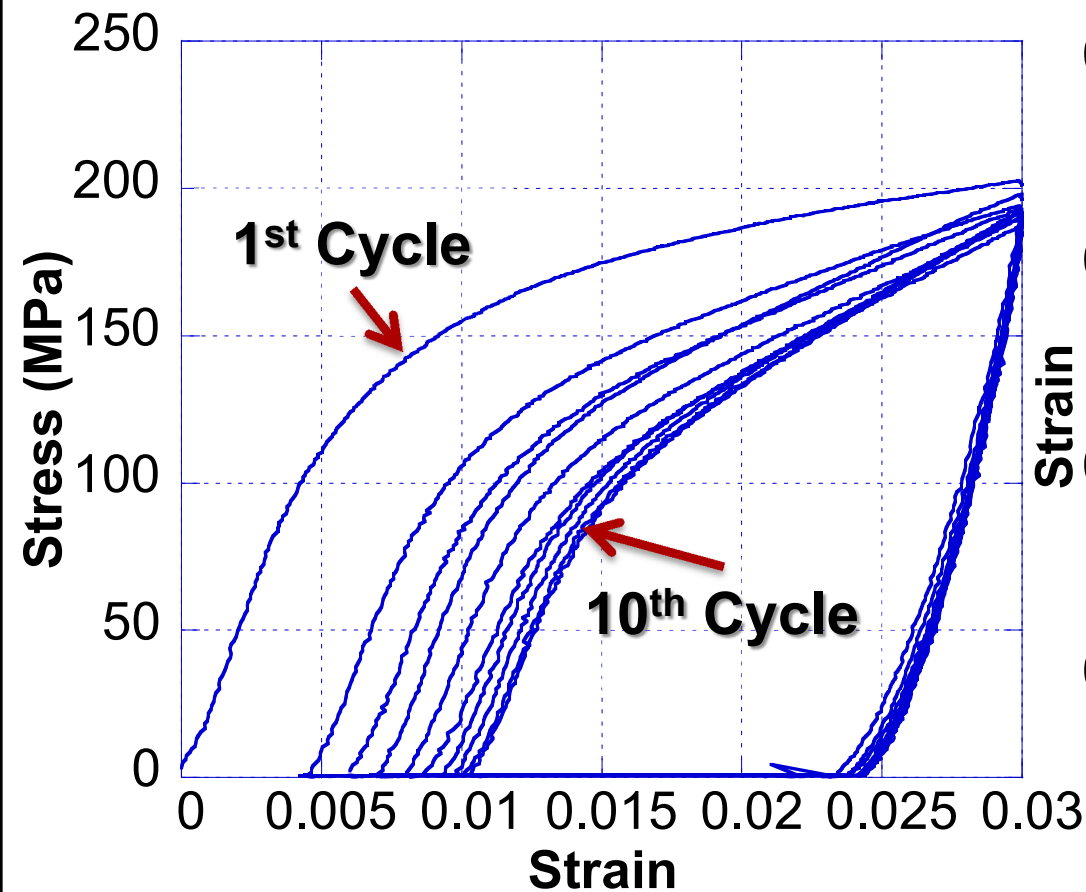
Effective Strain Rate
(1/s)

Heat Rate
(°C/min)

4×10^{-4}

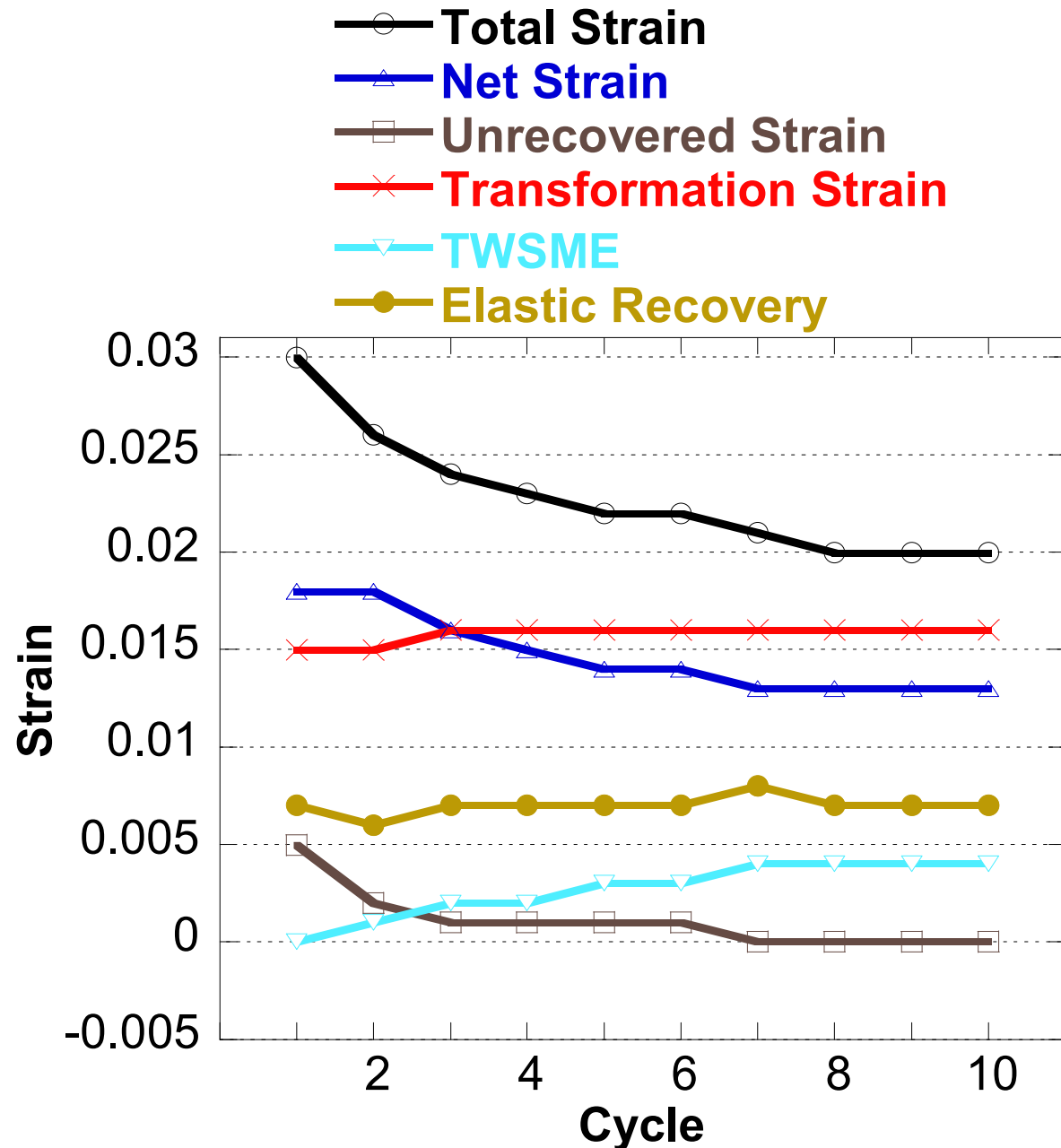
5-10

Pull to 103% of initial length (stroke control) @RT; unload to 0 N; heat to A_f+40 °C @0 N, hold; cool.



Shape Recovery Evolution

- Strain values stabilize over cycles.
- Total Strain** drops since test was stroke-limited & partial strain recovery occurred in early cycles.
- Unrecovered Strain** zeros out at 2% Total Strain.
- TWSME** stabilizes in 7 cycles.



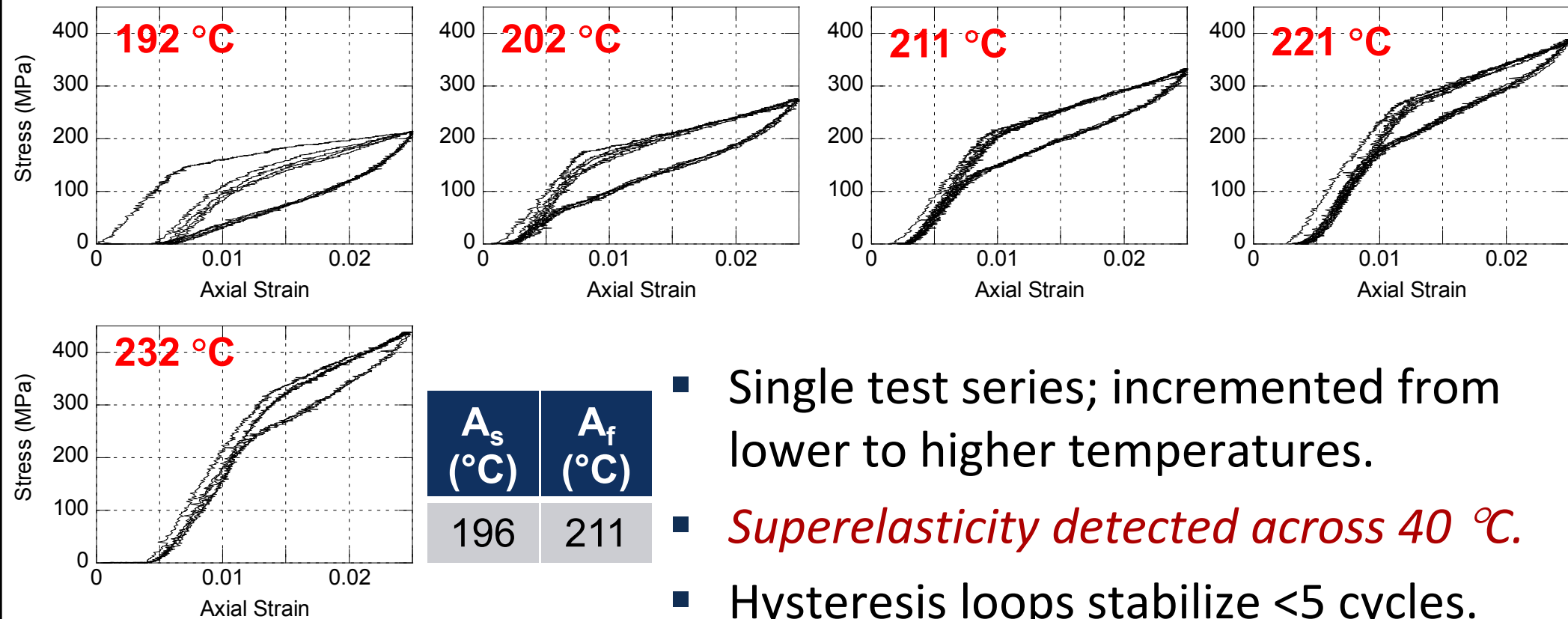
Superelasticity Experiments

Test Program

Effective Strain Rate
(1/s)

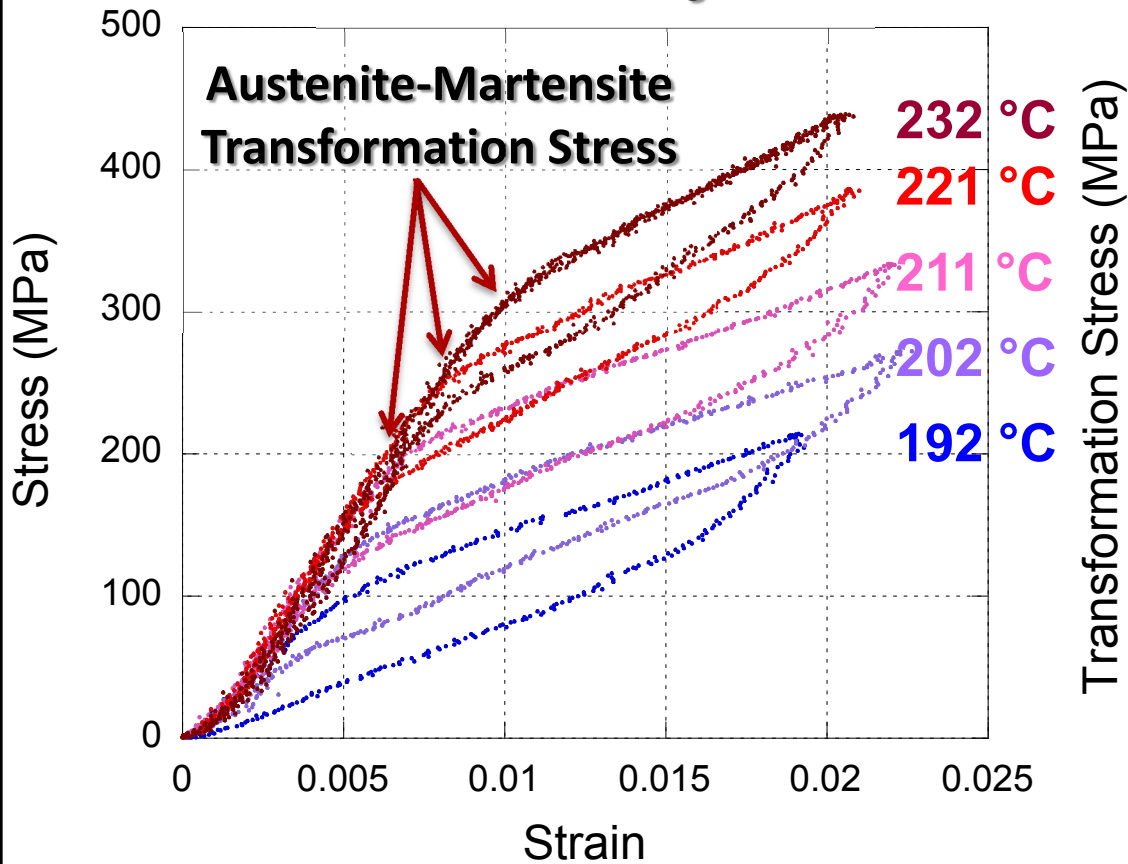
2×10^{-4}

Hold at temperature; pull to 2.5% of initial length; unload to 0 N; cycle load-stroke 4 more times; heat to $A_f + 40$ °C; hold; cool to next temperature; hold.

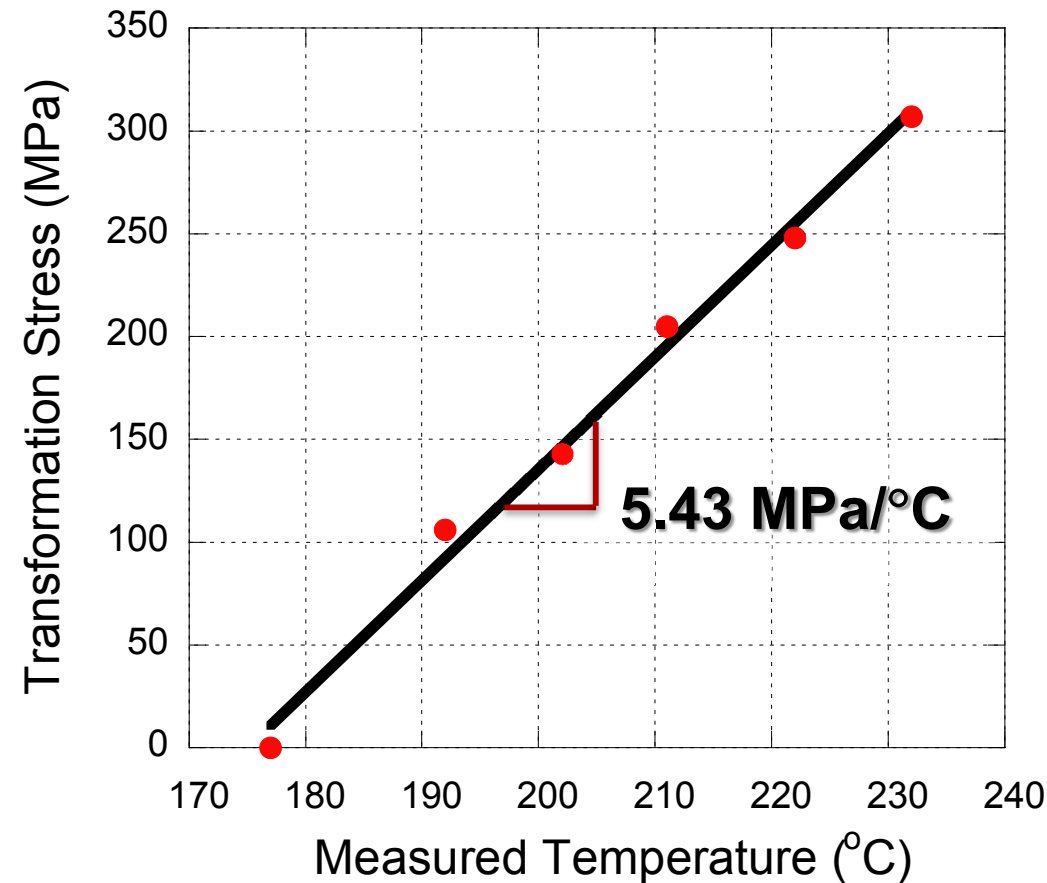


Transformation Stress

Stabilized Cycles



Austenite to Martensite



- Characteristic flag-shape superelasticity observed.
- Full recovery of $\sim 2.25\%$ strain.
- Transformation stress increases linearly with temperature.

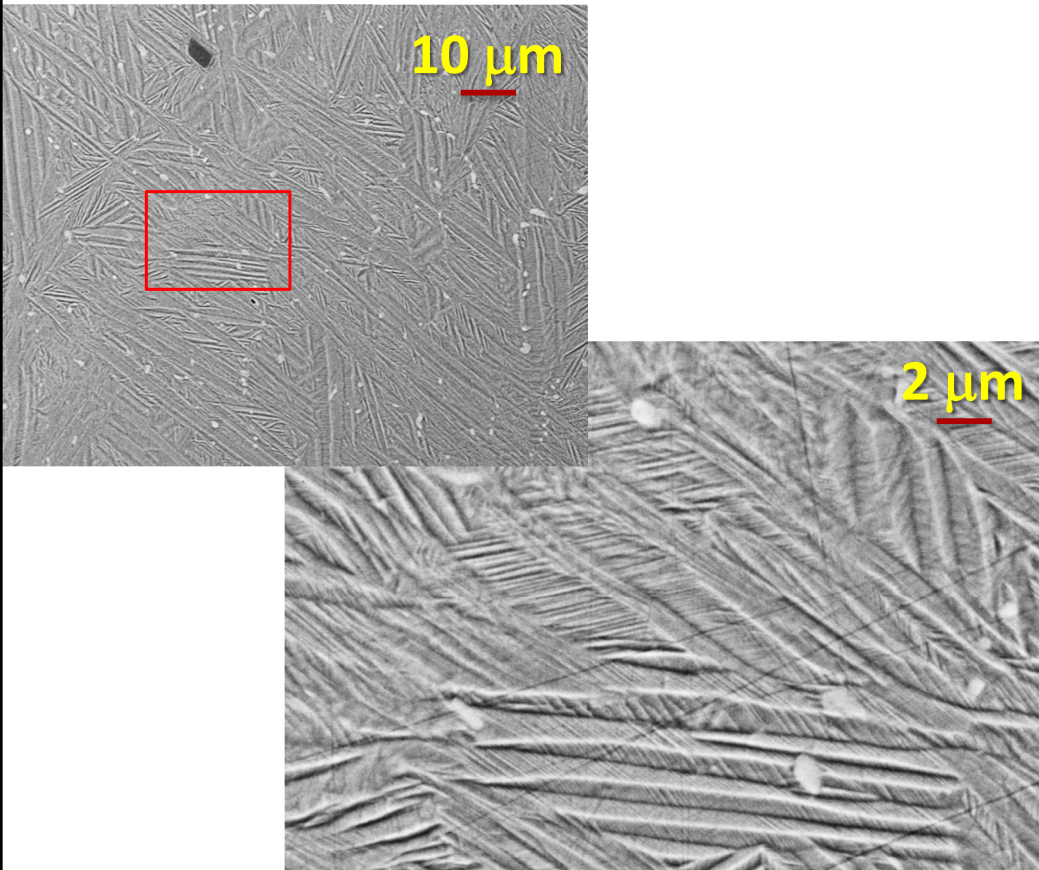
Concluding Remarks

- New range of Ti-rich Ni-Ti-Pt SMA's fabricated with 13.0 - 16.5 at.% Pt to find A_s ranging 150 °C to 250 °C.
- 16.5 at.% Pt variant with $A_s = 192$ °C chosen for further study.
- DSC, physical characterization, and thermomechanical tests performed.
- Target Ti-rich composition confirmed with ICP/MS.
- EDS shows Ti-rich precipitates.
- SME and Superelasticity demonstrated for the first time in these alloys.
- SME shows >2.5% tensile strain recovery; ~2.25% superelastic recovery.
- Two-way SME observed; increases with increased extension, stabilizes in few cycles.
- Superelasticity exhibits characteristic flag-shape and linear increase in Austenite-Martensite transformation stress.

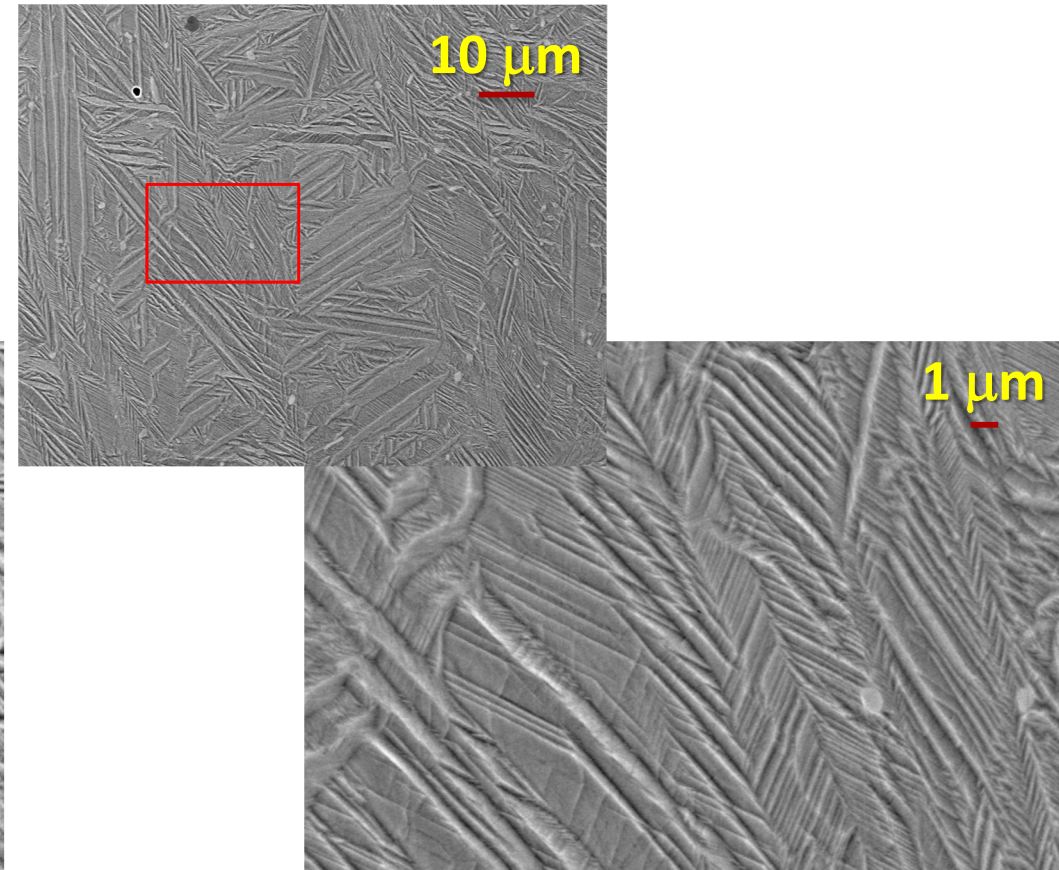
NOTES & DETAILS

Scanning Electron Microscopy

Grip Section



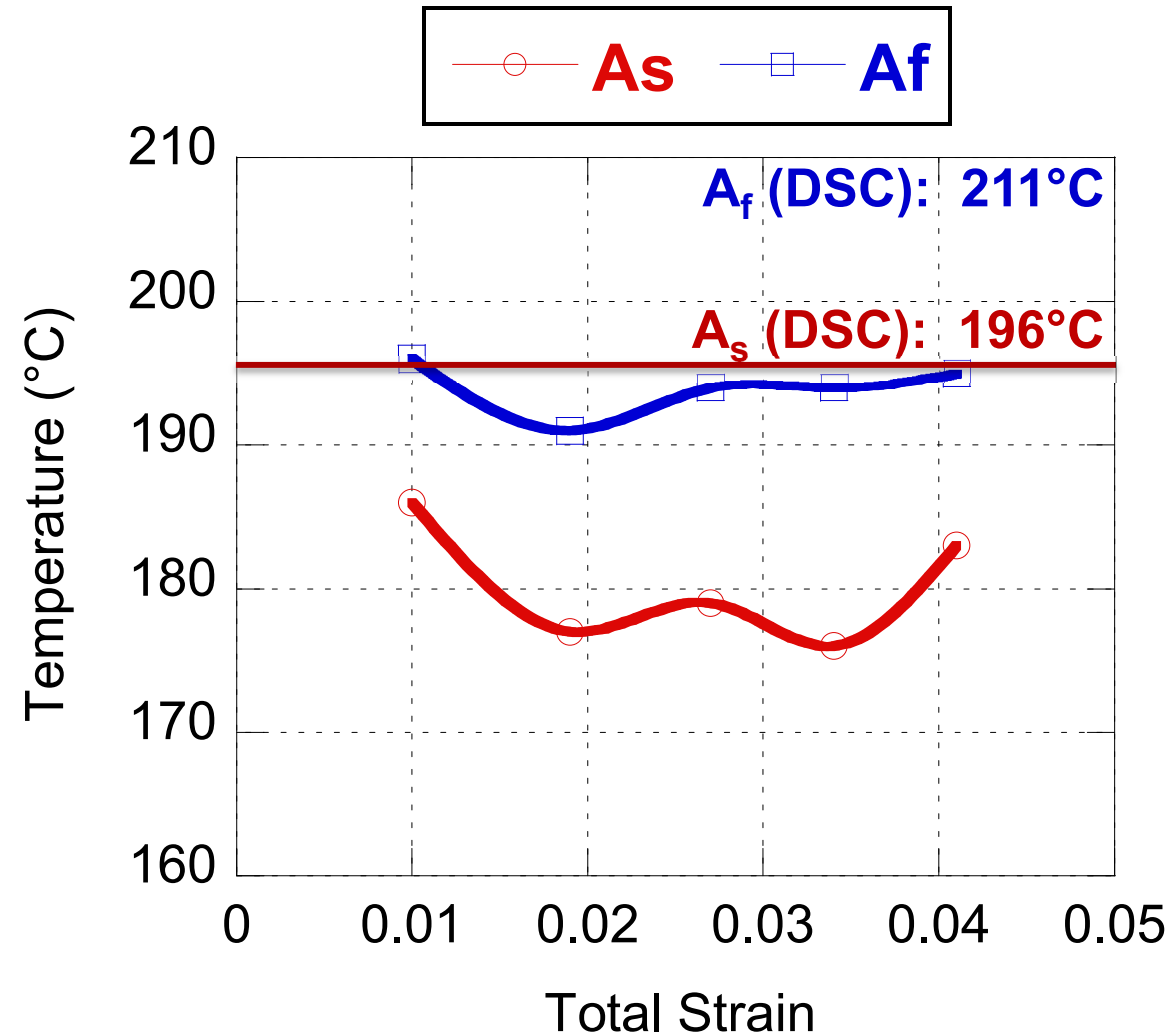
Middle of Gage



- Sample pulled to failure at RT, then imaged.
- No obvious evidence of detwinning in SEM images.
- Presence of white and dark precipitates.

SME Transformation Behavior

- Apparent transformation temperatures estimated from SME curves.
- Variability with respect to total strain.
- Tension samples show primary shape recovery $\sim 20^\circ\text{C}$ lower than DSC temperatures.
- Difference in recorded and samples temperatures partly to blame.



SME Transformation Behavior (Cyclic)

- Apparent transformation temperatures estimated from SME curves.
- Tension samples show primary shape recovery ~ 20 °C lower than DSC temperatures.
- Difference in recorded and samples temperatures partly to blame.

