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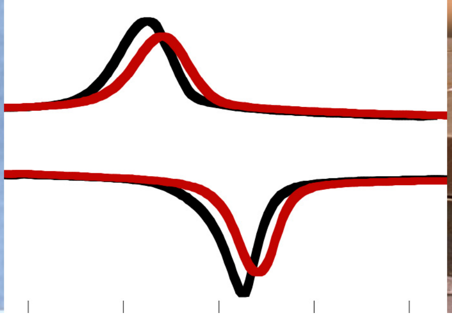
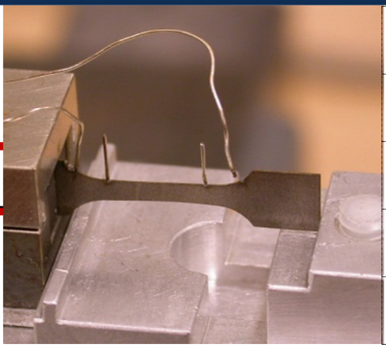
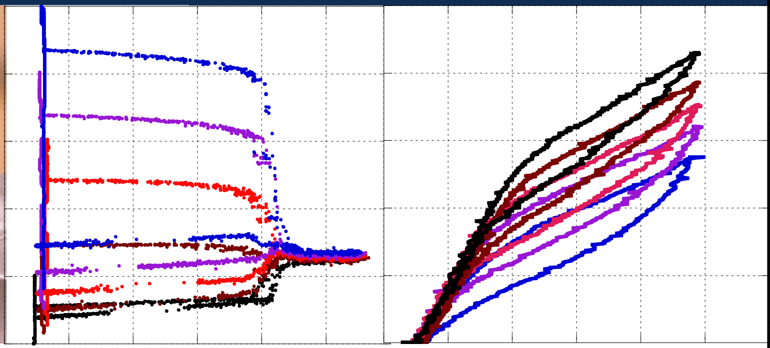

Shape Memory Effect and Superelasticity in NiTiPd High Temperature Shape Memory Alloys

Drs. Jordan E. Massad, **Thomas E. Buchheit**,
James R. McElhanon, Donald F. Susan
Sandia National Laboratories

Dr. Ronald D. Noebe
NASA Glenn Research Center

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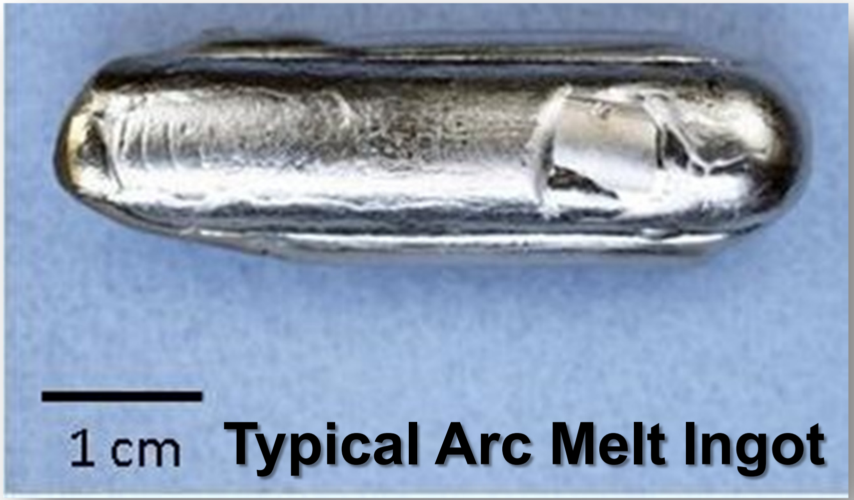
High Temperature SMAs

- Shape memory alloys (SMAs) that exhibit ***shape memory effect <u>and</u> 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.
- Pd replacement can increase transformation temperatures up to 550 °C ($Ti_{50}Pd_{50}$).
- For transformation temperatures >250 °C, previous studies of Ti-rich NiTiPd have focused primarily on ≥30 at.% Pd.
- Bigelow et al. (2007) investigated Ti-rich 25 at.% Pd that transforms ~190 °C.

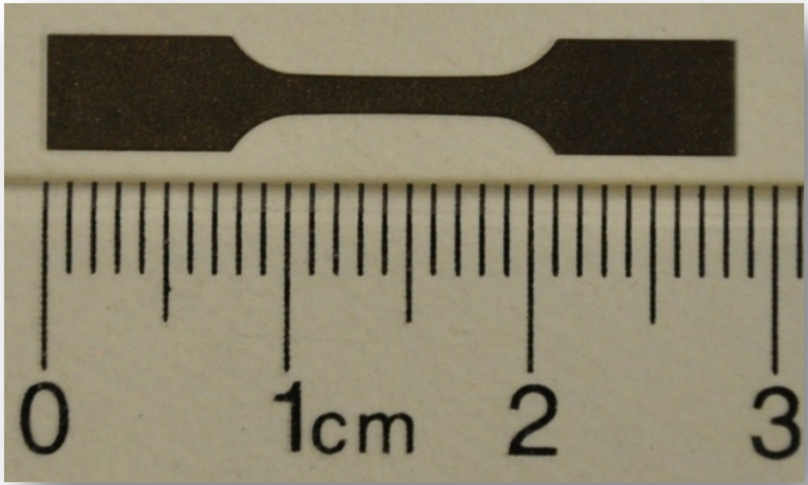
Desired Transformation Temperature Range 150-250 °C

Fabrication

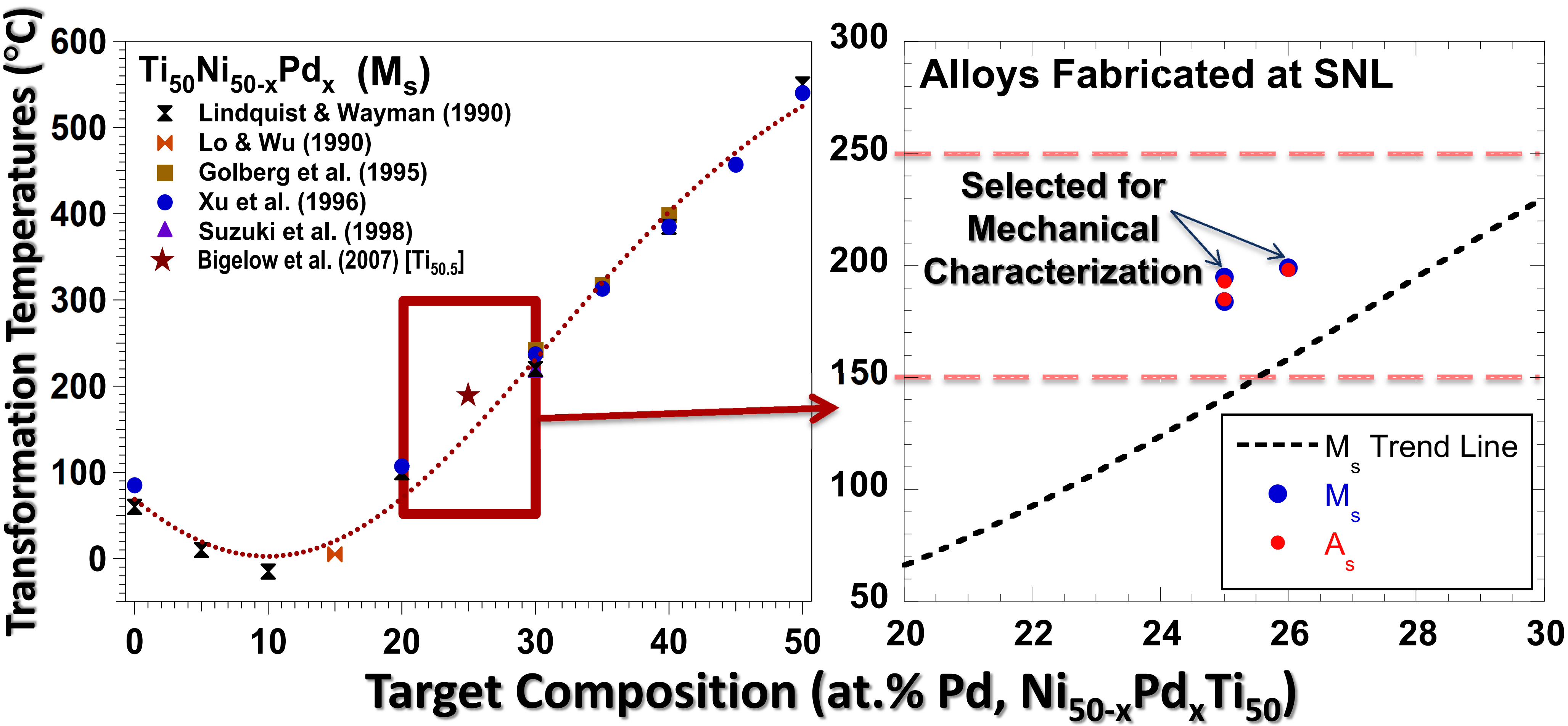
- Targeted Ti-rich NiTiPd ternary alloys.
- Ternary alloys processed via **arc melting.**
- Followed by **homogenization**.
- Electro-discharge machined (EDM) mini-coupons to enable <u>*tension tests*</u> with small amount of material.



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



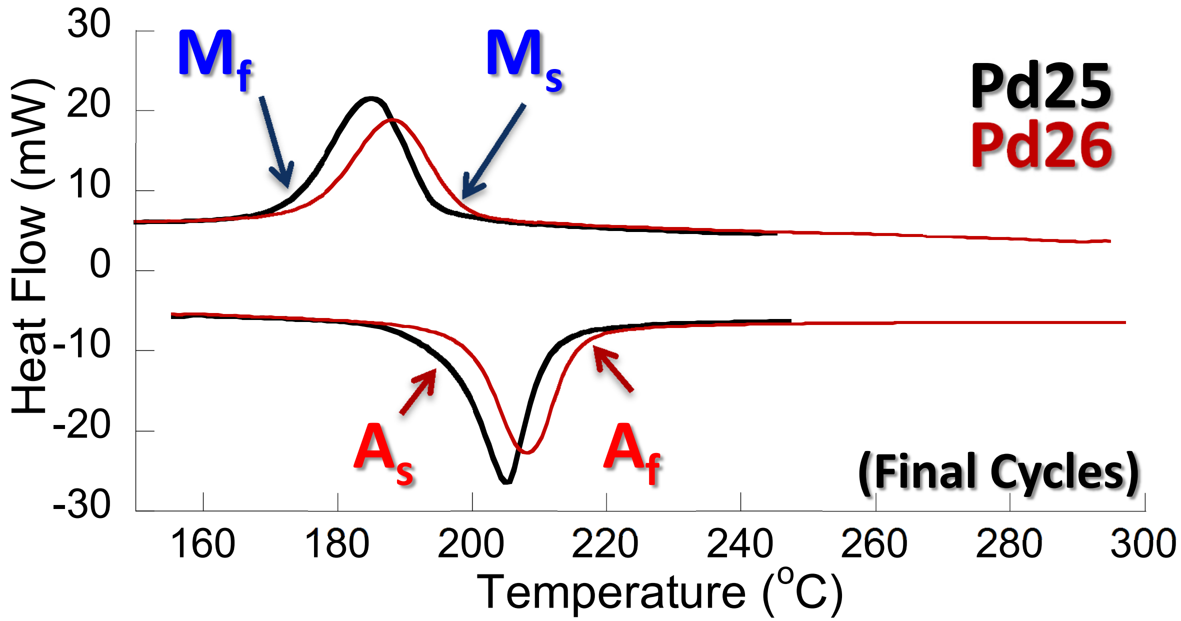
Choosing a Composition in NiTiPd



- Historical data indicates that Ti-rich, 20-30 at.% Pd compositions may produce desired transformation temperatures.
- Investigate two compositions: **25 at.% Pd (Pd25)** and **26 at.% Pd (Pd26)**.

Compositional and DSC Analysis

Target (at.% Pd)	ICP/MS (at.% Pd)	ICP/MS (at.% Ti)	ICP/MS (at.% Ni)
25	25.4	49.8	24.1
26	26.4	49.8	22.8



- Inductively coupled plasma mass spectroscopy (ICP/MS) verified composition with small Cu impurities.
- Differential Scanning Calorimetry (DSC) gives baseline transformation temperatures.
- DSC trace stabilizes in 2-3 thermal cycles.

Alloy	M_f	M_s	A_s	A_f
Pd25	172	195	193	212
Pd26	175	199	198	217

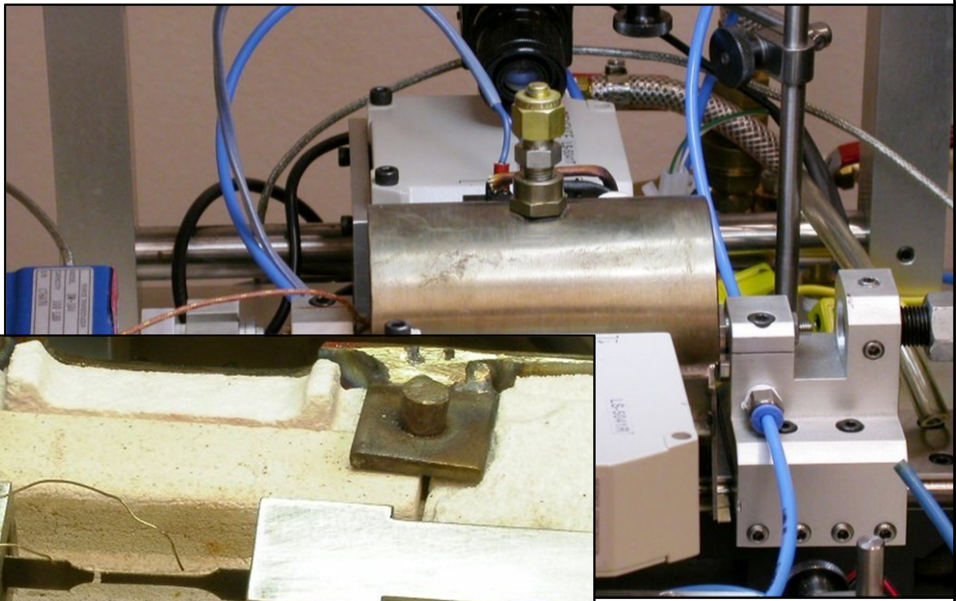
Martensite and Austenite Start & Finish Transformation Temperatures (°C)

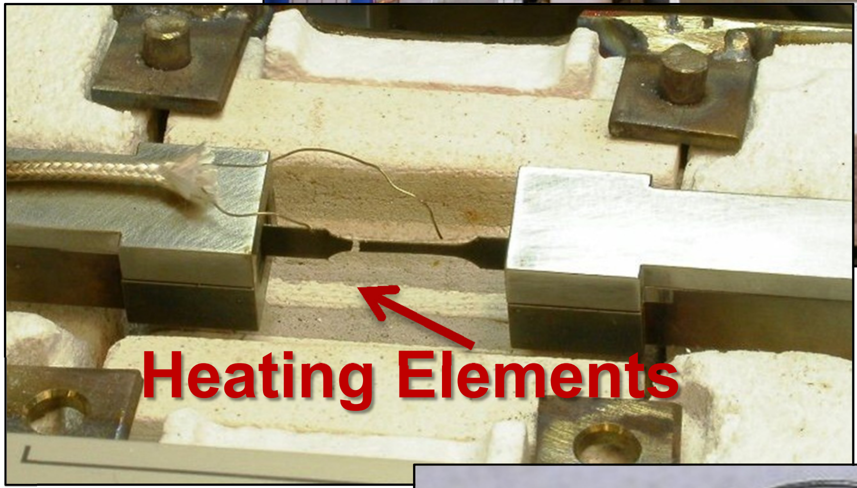
Pd25 (°C) (Bigelow, 2007)

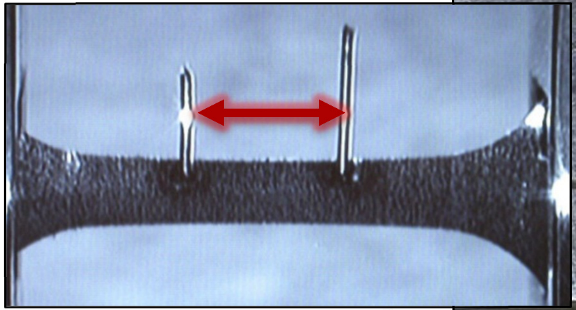
M_f	M_s	A_s	A_f
178	190	193	197

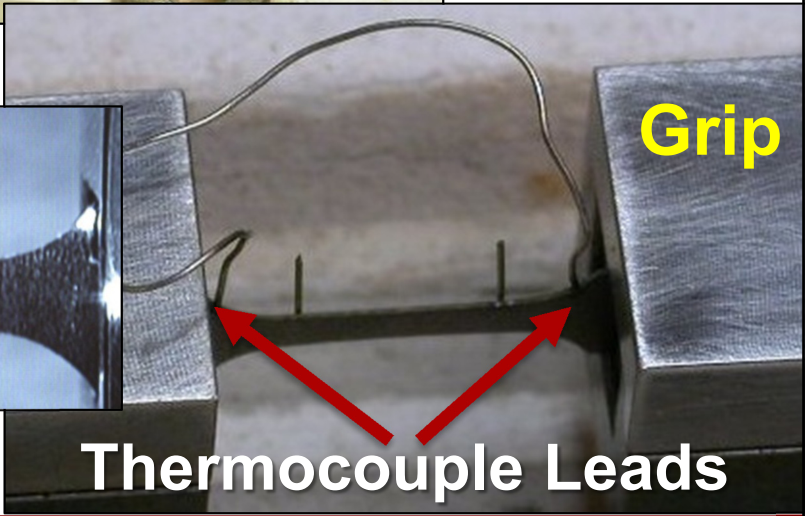
Mini-tensile Test Setup

- 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 readout from thermocouple leads.









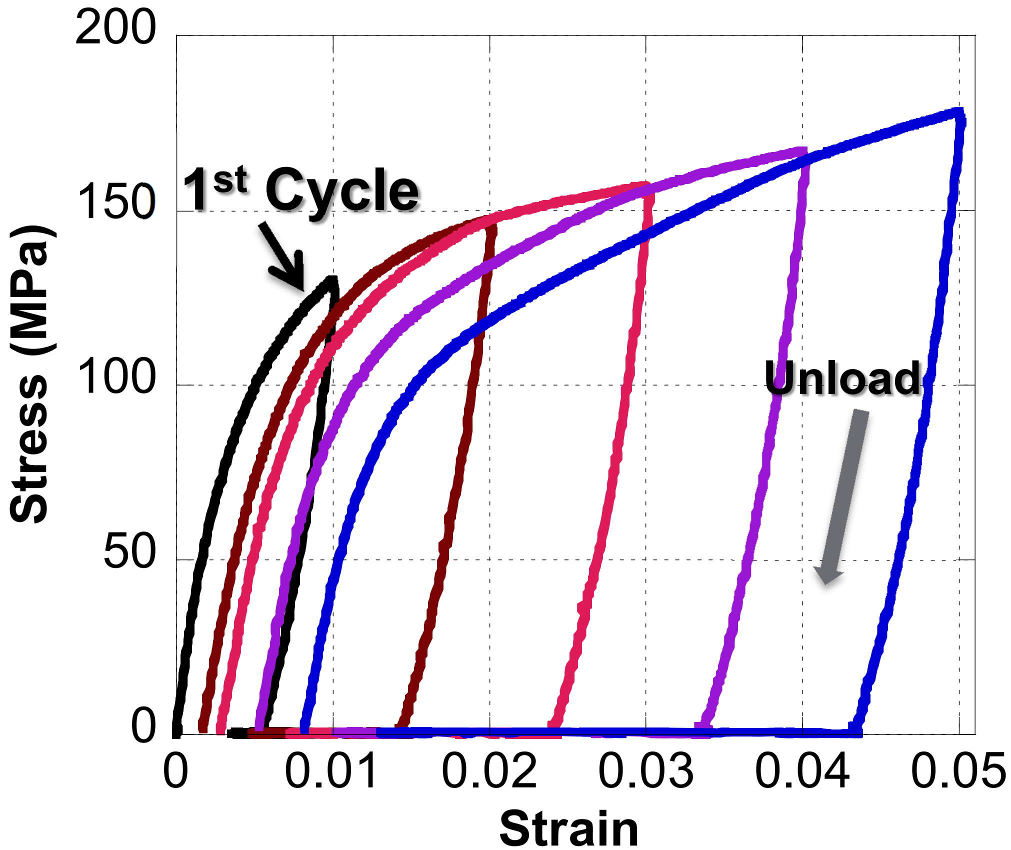
Shape Memory Characterization

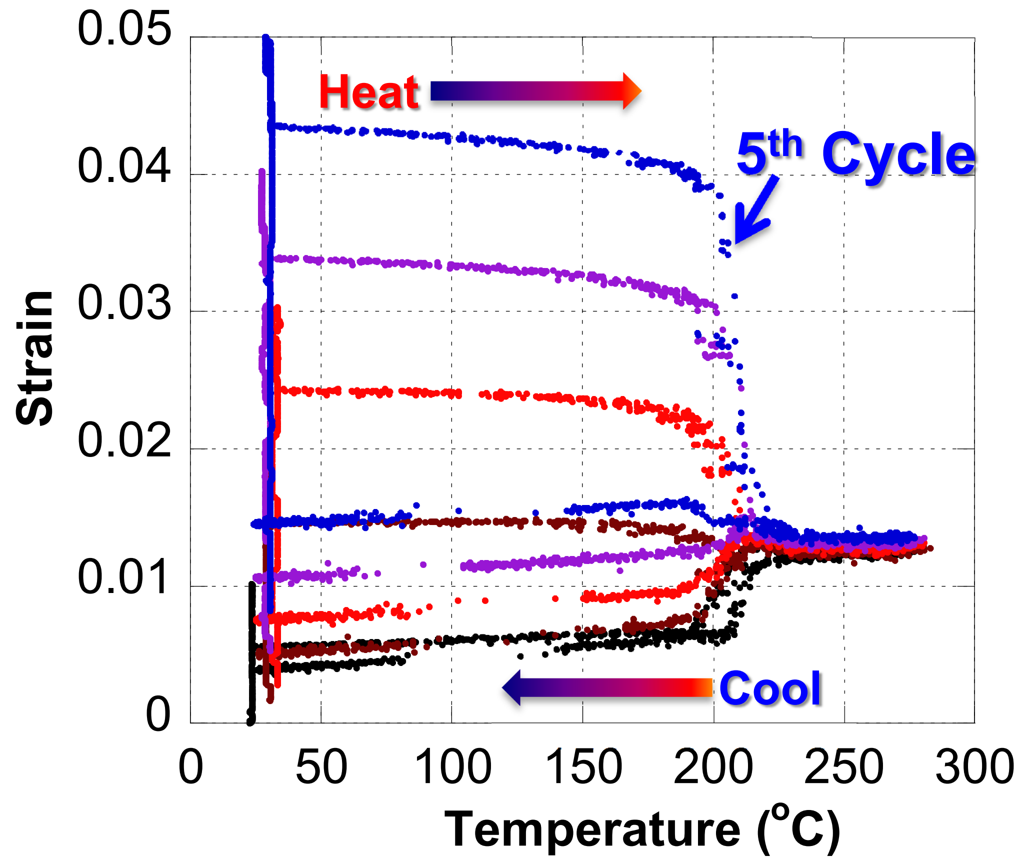
Mechanical characterization tasks are guided by end-use considerations.

- Device Considerations
  - Deform or set SMA devices in Martensite, then heat to recover deformation under no load; may be reset multiple times.
  - SMA devices may be deformed at high temperatures (Austenite).
- 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.
- To assess alternatives, compare NiTiPd data to Ti-rich $NiTi_{50.5}Pt_{15.5}$ with similar transformation temperatures.

SME Experiments

Pd26

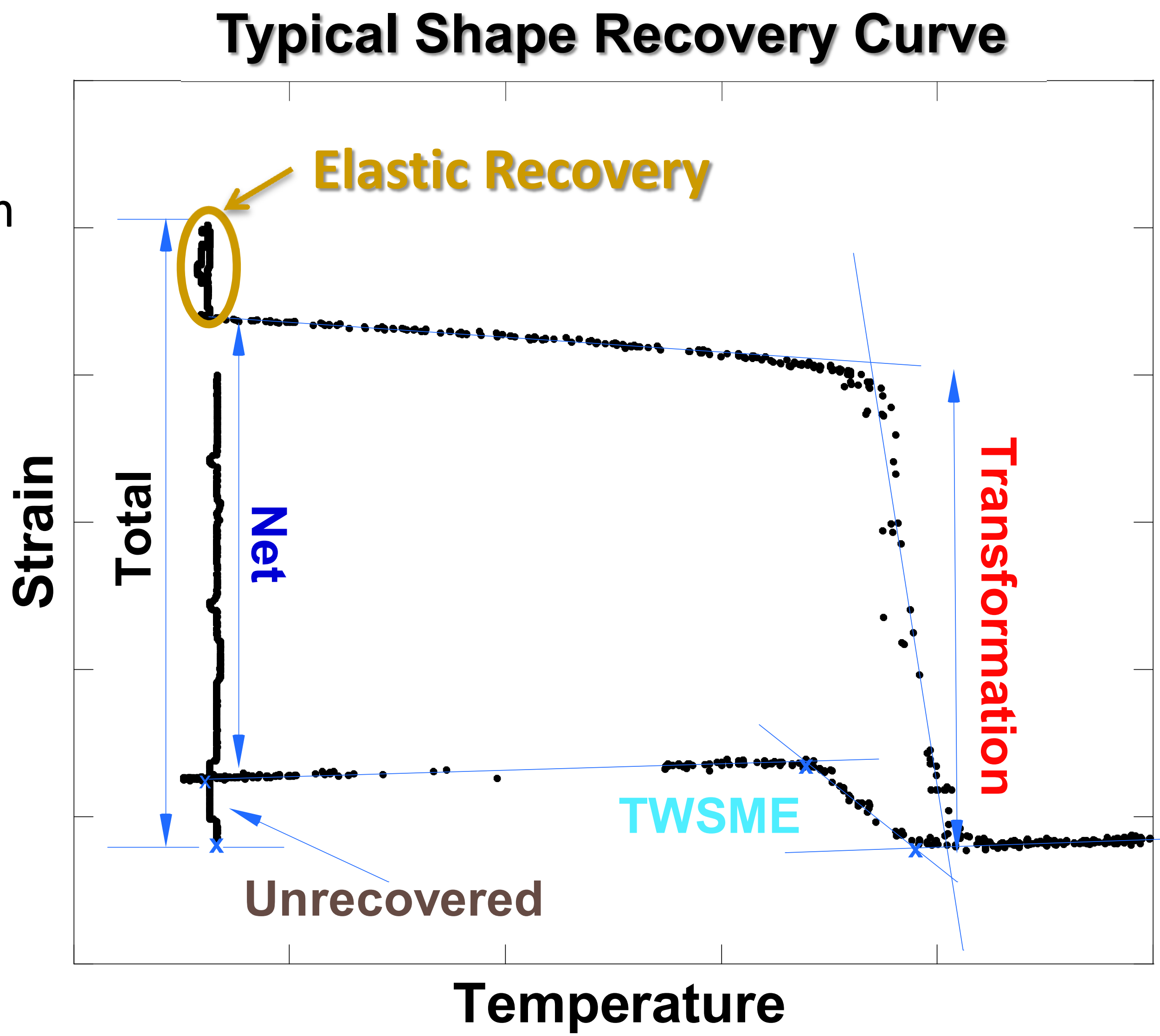




- Single sample, stroke controlled test to *increasing* stroke values.
- Load-unload at RT ($4 \times 10^{-4}\ s^{-1}$).
- Load-free heat to recovery to A_f + 60 °C (5 °C/min).
- Load-free cool-down shows changing two-way SME (TWSME).

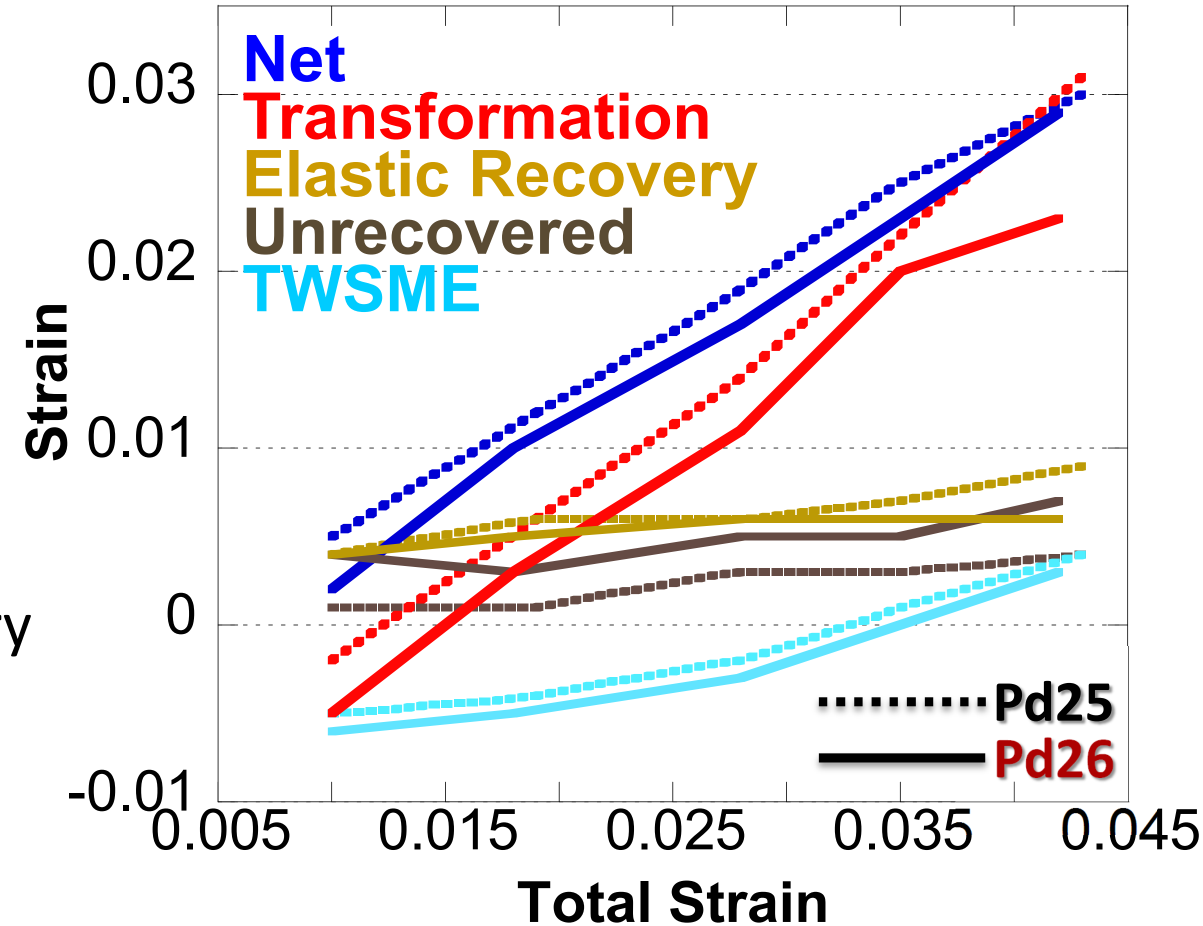
SME Strain Quantities

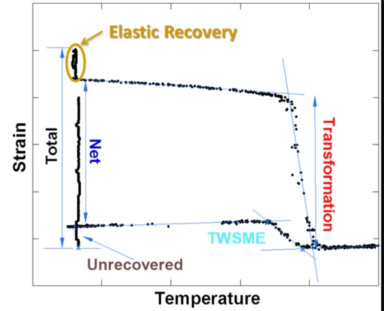
- **Total Strain**: how much the sample stretched under load.
- **Elastic Recovery**: strain recovered on unloading at room temperature.
- **Transformation Strain**: strain recovered during transition to Austenite.
- **Two-way SME (TWSME)**: strain change as sample cools to Martensite.
- **Unrecovered Strain**: strain remaining after full cycle.
- **Net Strain**: final strain recovered at RT due to *thermal* cycle.



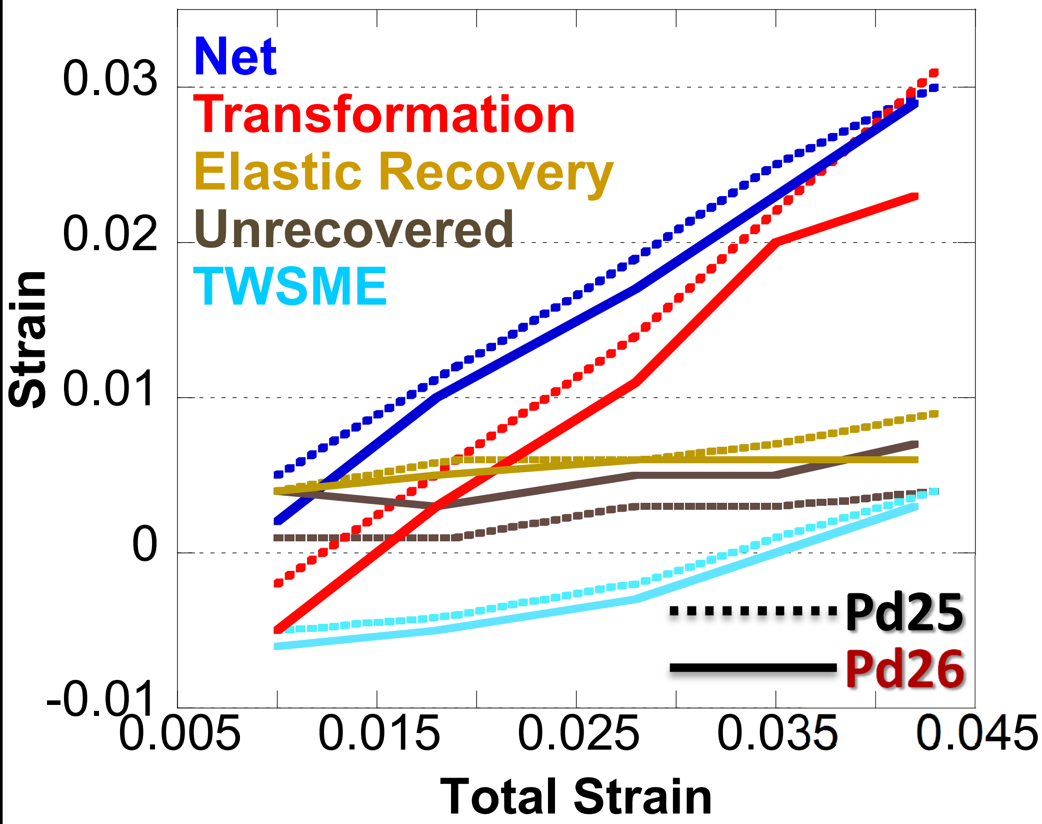
Shape Recovery vs. Total Strain

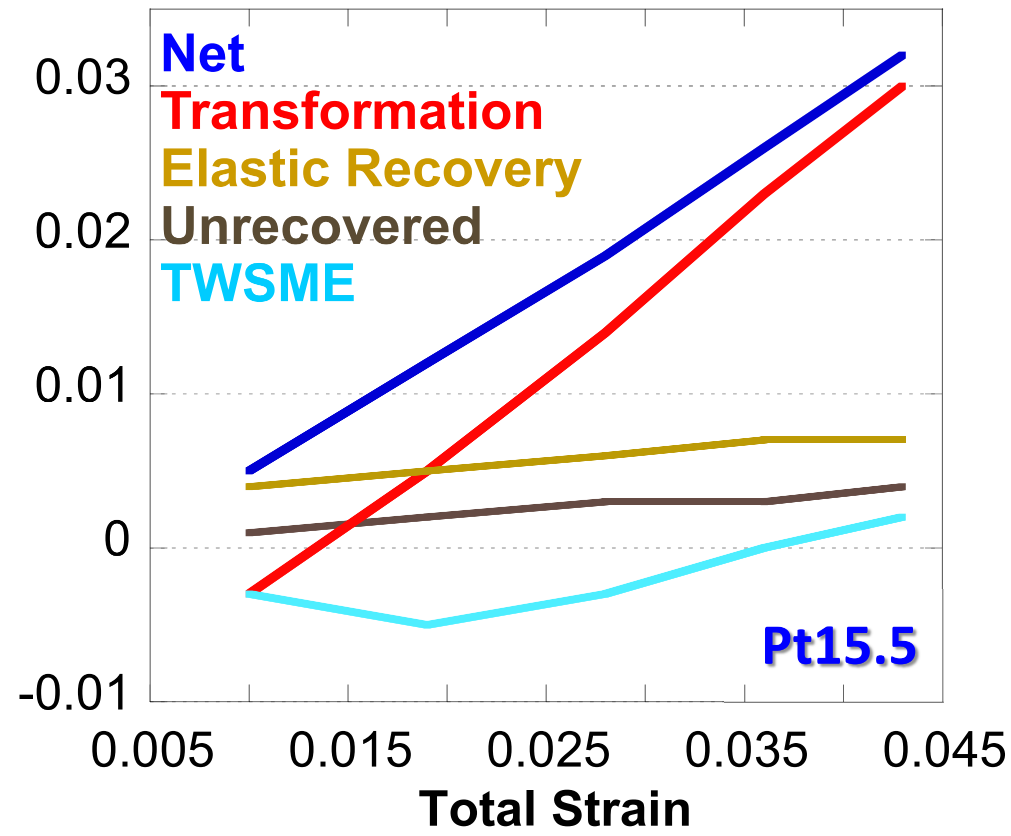
- **Transformation Strain** increases as total stroke increases.
- Up to 3% **Net Strain** recovery observed.
- **TWSME** strain is near-zero at ~3.5% **Total Strain**.
- Samples show similar recovery response.
- Data determines how much SMA should be stretched to recover a desired deformation.





Shape Recovery vs. Total Strain





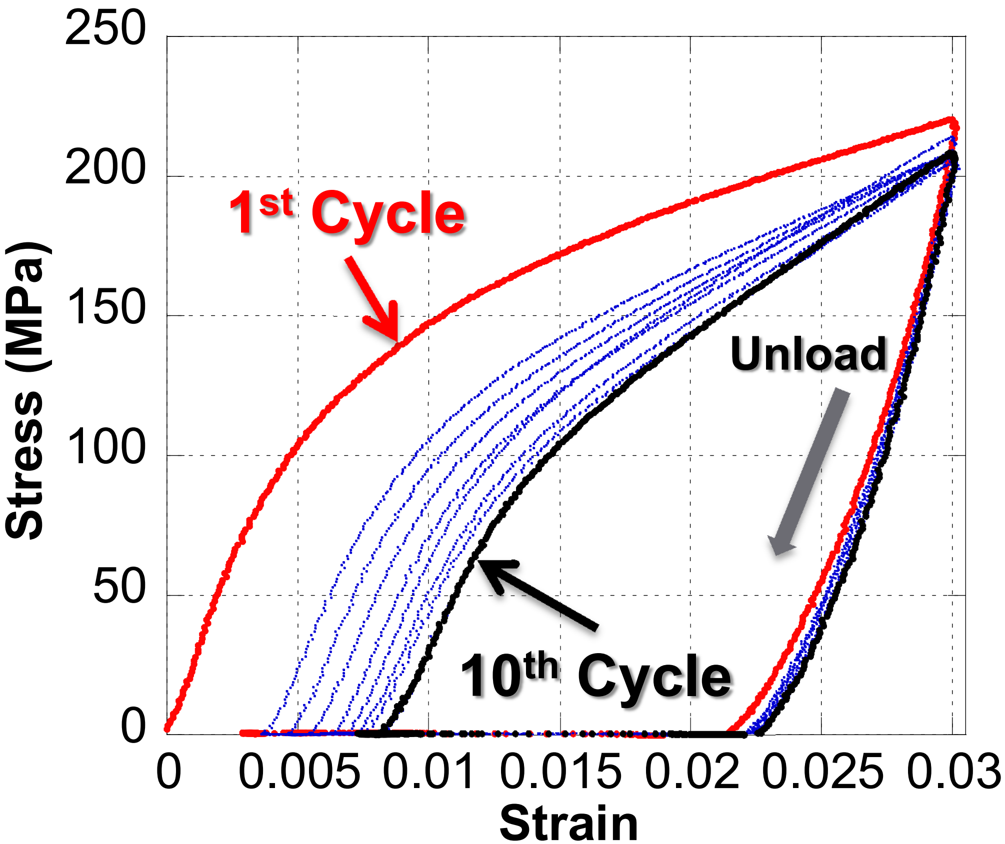
- **Pt15.5** SME similar to that of **Pd25** & **Pd26**.
- All show minimum TWSME at ~3.5% **Total Strain**.

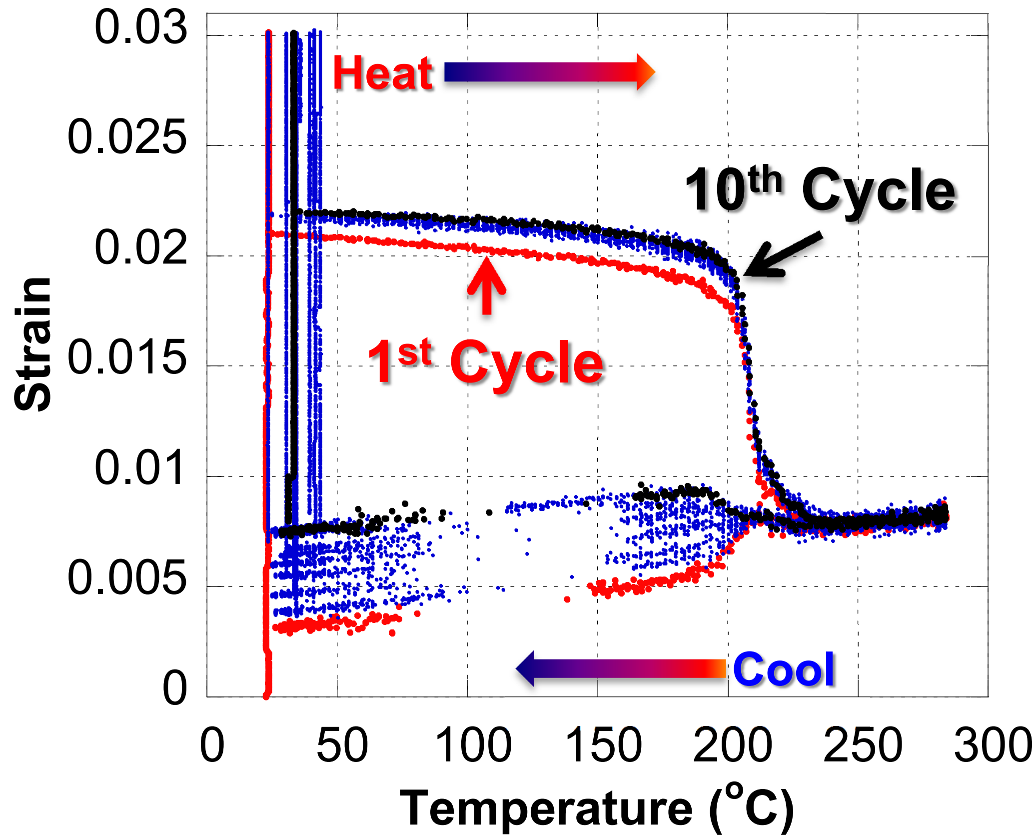
Alloy	M_f	M_s	A_s	A_f
Pd25	172	195	193	212
Pd26	175	199	198	217
Pt15.5	179	206	202	222

(°C)

SME Experiments (Cyclic)

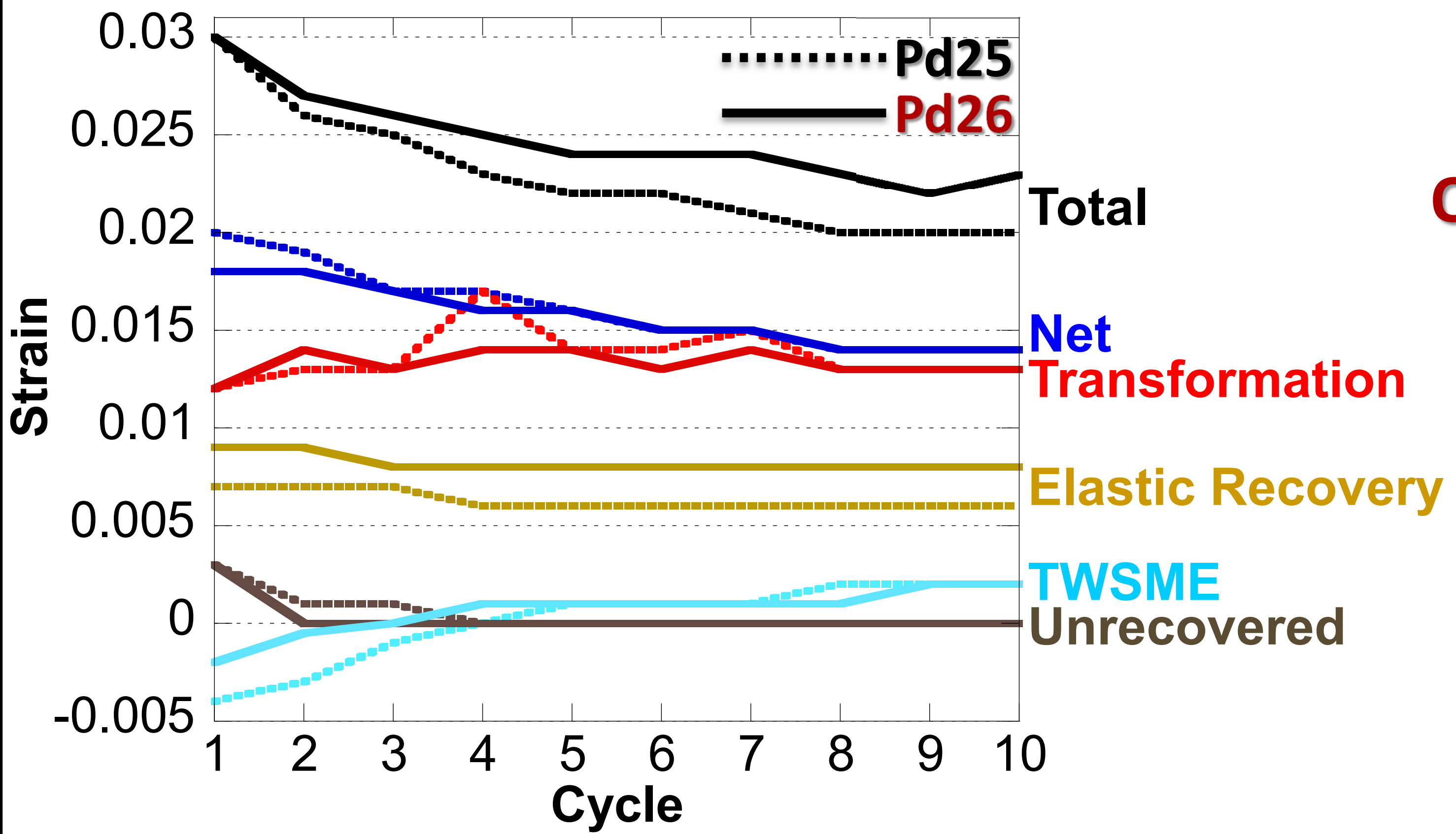
Pd26



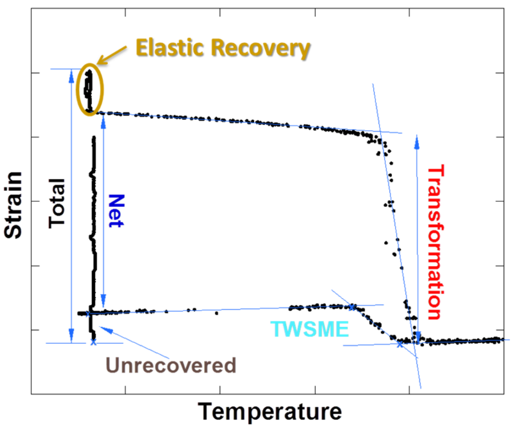


- Single sample, stroke controlled test to *fixed* stroke value.
- Load-unload at RT (4×10^{-4} s^{-1}).
- Load-free heat to recovery to A_f + 60 °C (5 °C/min).
- Load-free cool-down shows *evolving* TWSME.

Shape Recovery Evolution

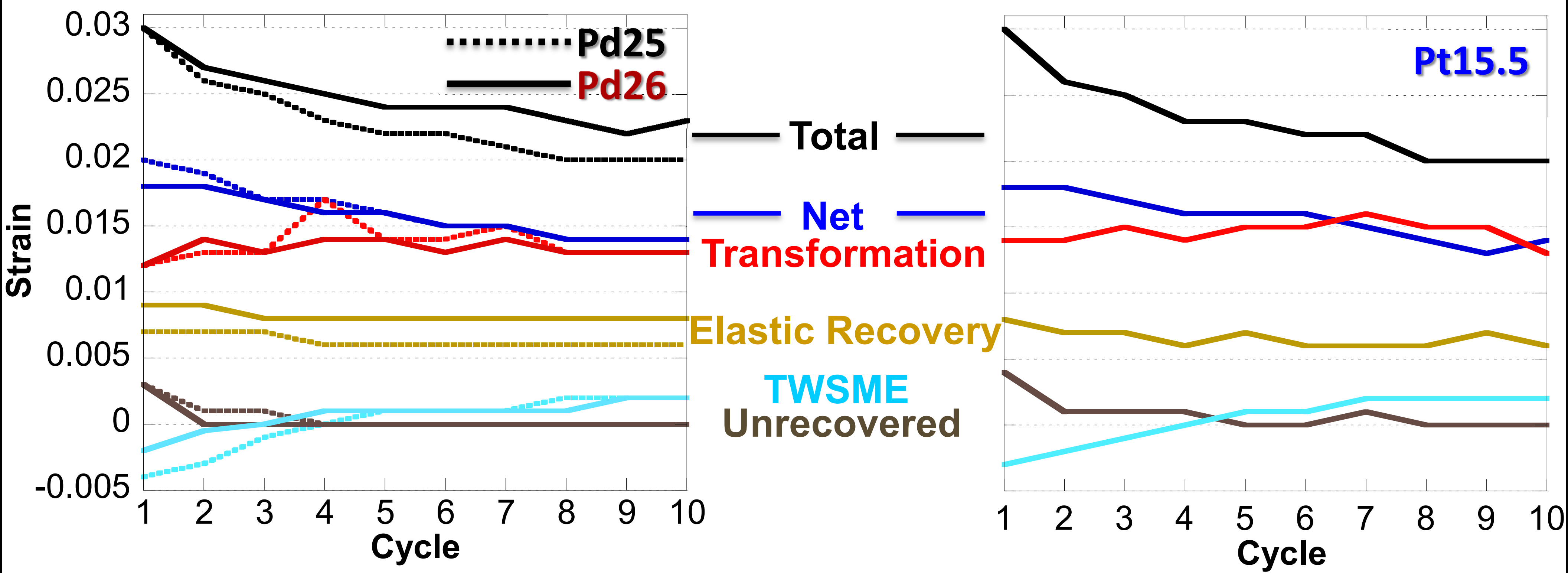


Strain Values Evolve Over Repeated Cycles

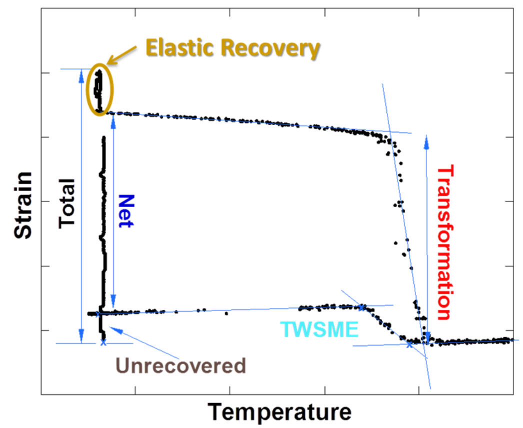


- **Total Strain** drops since test was stroke-limited & partial strain recovery occurred in early cycles.
- **Net Strain** stabilizes at 1.4% with ~2% **Total Strain**.
- **Unrecovered Strain** vanishes in 2-4 cycles at ~2.5% **Total Strain**.
- **TWSME** stabilizes in 8-9 cycles, vanishes in 3-4 cycles.

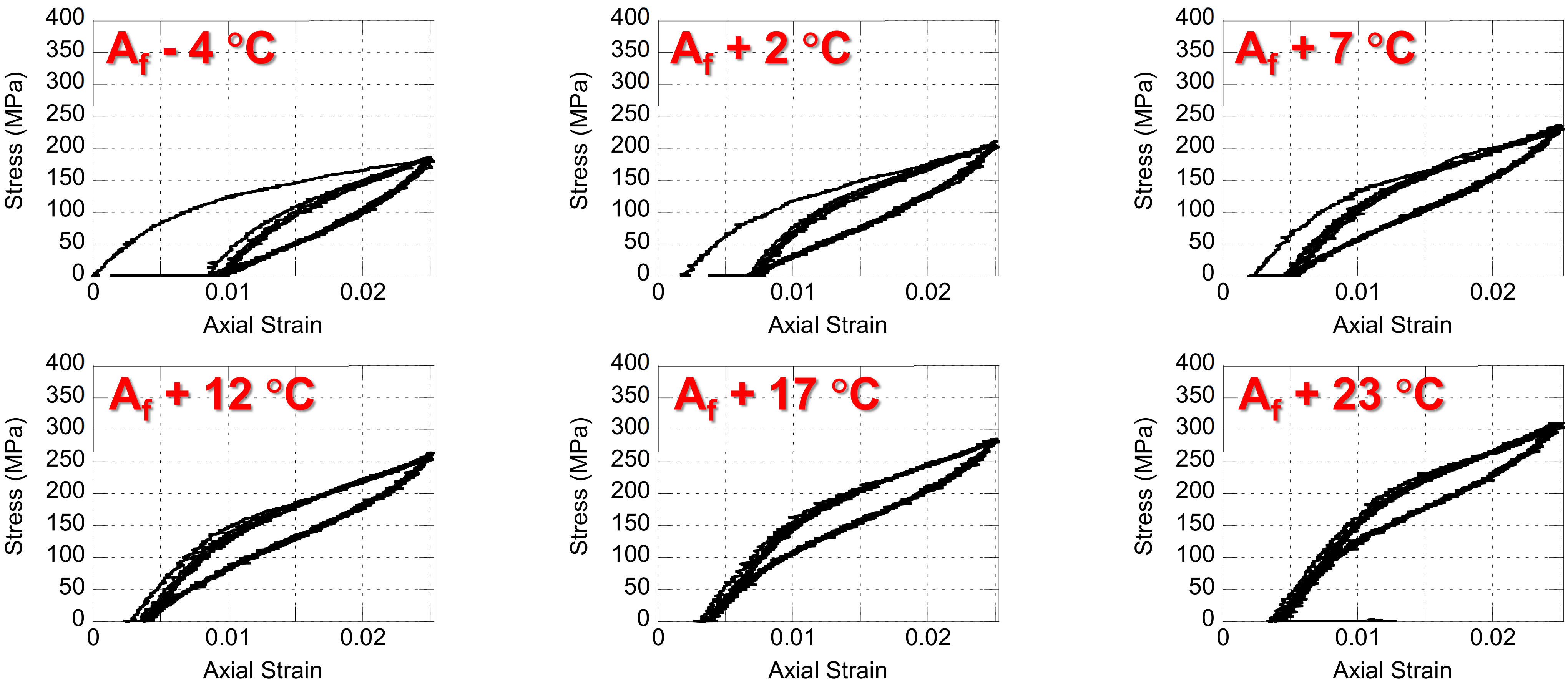
Shape Recovery Evolution



- **Pt15.5** evolution is similar to that of **Pd25** & **Pd26**.
- Minimum **TWSME** at ~2.3% **Total Strain**.



Superelasticity Experiments

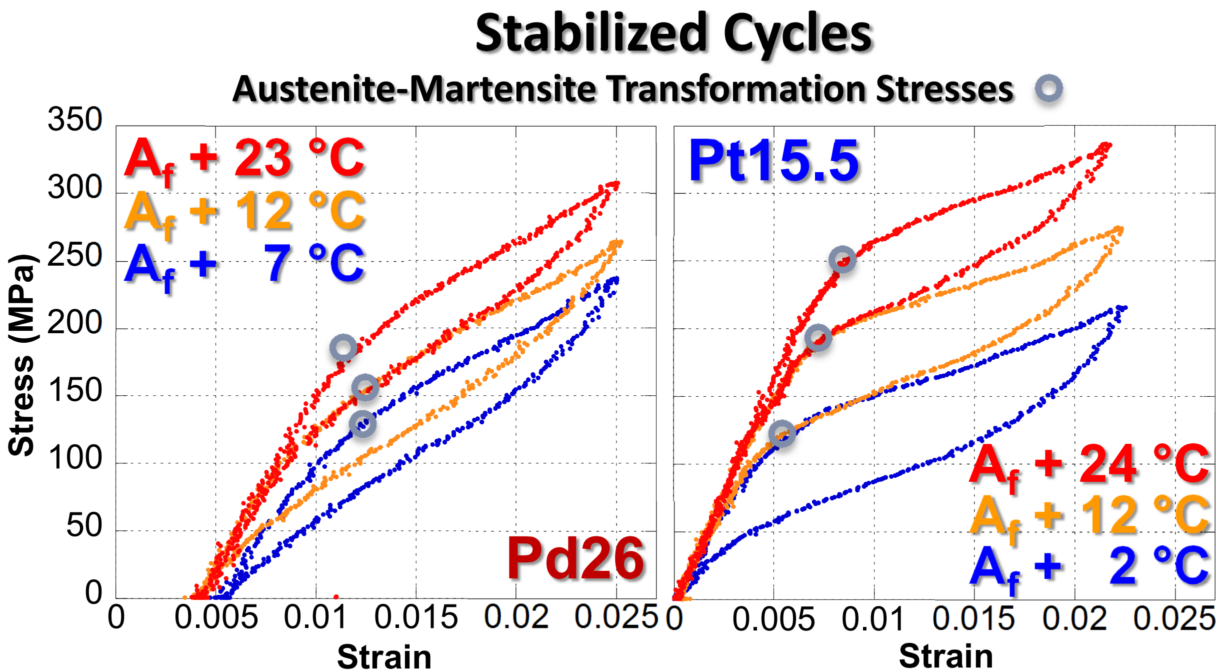


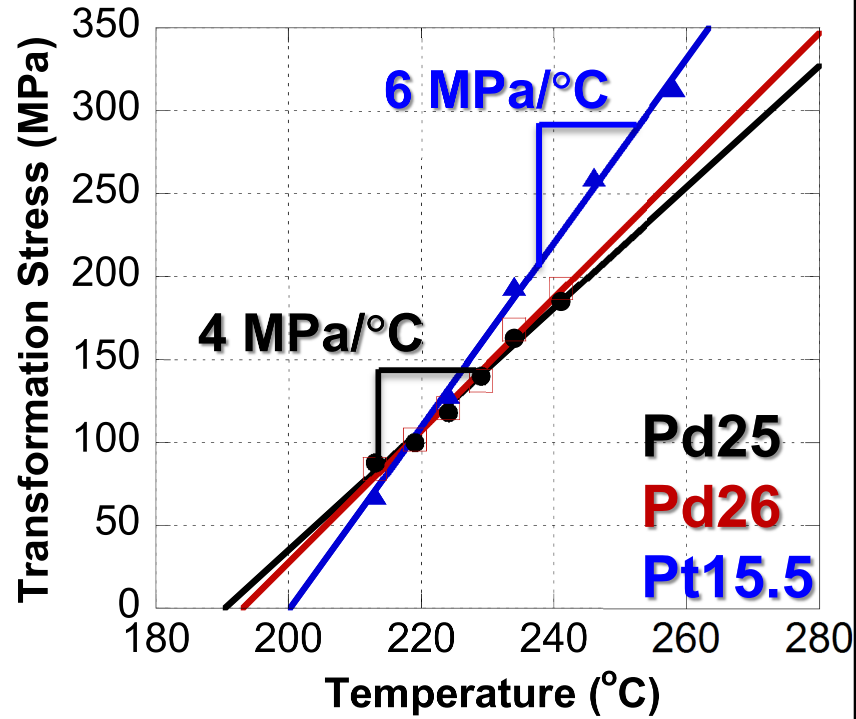
Pd26

M_s (°C)	A_f (°C)
199	217

- Single sample, stroke limited test series.
- Incremented from lower to higher temperatures each reached from A_f + 40 °C.
- Hysteresis loops stabilize <5 cycles.
- Superelasticity detected across at least 10 °C.

Transformation Stress





- Characteristic flag-shape superelasticity observed with recovery of ~2.0% (Pd) and ~2.3% (Pt) strain.
- **Pd26** shows considerably more hardening during transformation.
- **Pt15.5** Austenite slightly stiffer than **Pd26** (33 vs. 25 GPa).
- Transformation stress slope is similar between Pd compositions, but **Pt15.5** is steeper.

A_f (°C)

Pd25	Pd26	Pt15.5
212	217	222

Concluding Remarks

- Ti-rich NiTiPd SMAs fabricated with 25, 26 at.% Pd to achieve A_s in 150 °C to 250 °C range.
- Target composition confirmed with ICP/MS.
- Both SME and Superelasticity demonstrated in this high temperature range.

- **Pd25** and **Pd26** behave similarly:
  - Recover >2.5% tensile strain recovery via SME, ~2% via Superelasticity.
  - Two-way SME observed; increases with increased extension, stabilizes in few cycles; can possibly be minimized under certain conditions.
  - Superelasticity exhibits characteristic flag-shape, stabilizes in few cycles.
  - Austenite-Martensite transformation stress increases at 4 MPa/°C.

- **NiTiPd** compared to **$NiTi_{50.5}Pt_{15.5}$**:
  - SME behavior is similar among these samples.
  - Superelastic recovery is similar, but **Pt15.5** shows less transformation hardening and transformation stress increases more with temperature.

Alloys with very different compositions may offer similar shape memory response.