Effects of Severe Plastic Deformation and Sc Addition on the Recoverability of TiNiPd High Temperature Shape Memory Alloys

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Outline

- Challenges
- Objectives
- Materials Processing
- Experimental Results
  - Material Characterization
  - Thermo-mechanical Testing
- Conclusion
- Future Work

Challenges

Conventional TiNi ~ 100℃

Ti(Ni,Pd) ~ 100-400℃

Operating Temperature ↑ Challenges ↑

Long term stability

- Microstructure (recovery, recrystallization)
- Phase structure (decomposition)
- Resistance to oxidation

Plasticity

- Resistance to dislocation slip (ease of dislocation motion and decrease in the critical stress for slip)

Objectives

Enhance the recoverability of a $Ti_{50.5}Ni_{24.5}Pd_{25}$ (at. %) HTSMA

Increase the resistance to dislocation slip

Quaternary alloying with 0.5 at. % Sc (by solid solution hardening)

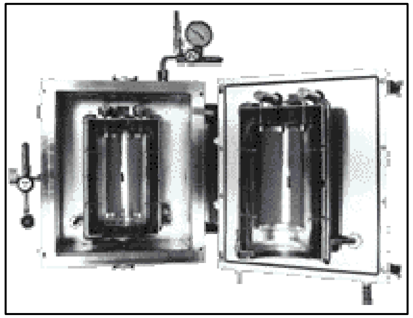
- Strong site preference for Ti
- Increases the formation energy, significantly affecting the bond strength in the alloy
- Increases the lattice parameter, thus the internal strain

Severe plastic deformation using equal channel angular extrusion (ECAE)

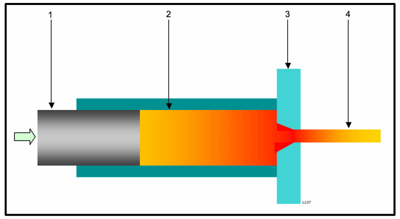
- Grain refinement
- Work hardening

Materials Processing

1. Vacuum induction melting of elemental constituents (Ni, Ti, Pd, Sc)

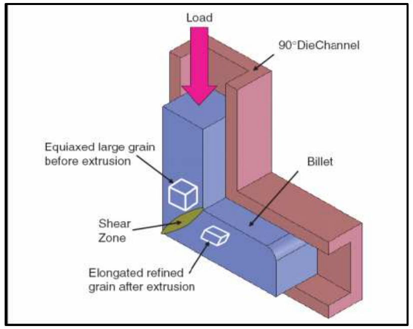


2. Homogenization of ingots at 1050℃ for 72 hours



3. Extrusion at 900℃ with an area reduction ratio of 7:1

As-extruded Billet



4. Severe plastic deformation of the as-extruded billet via ECAE

5. Preparation of microtensile specimens from the as-extruded and ECAE'ed billet by electrical discharge machining (EDM)

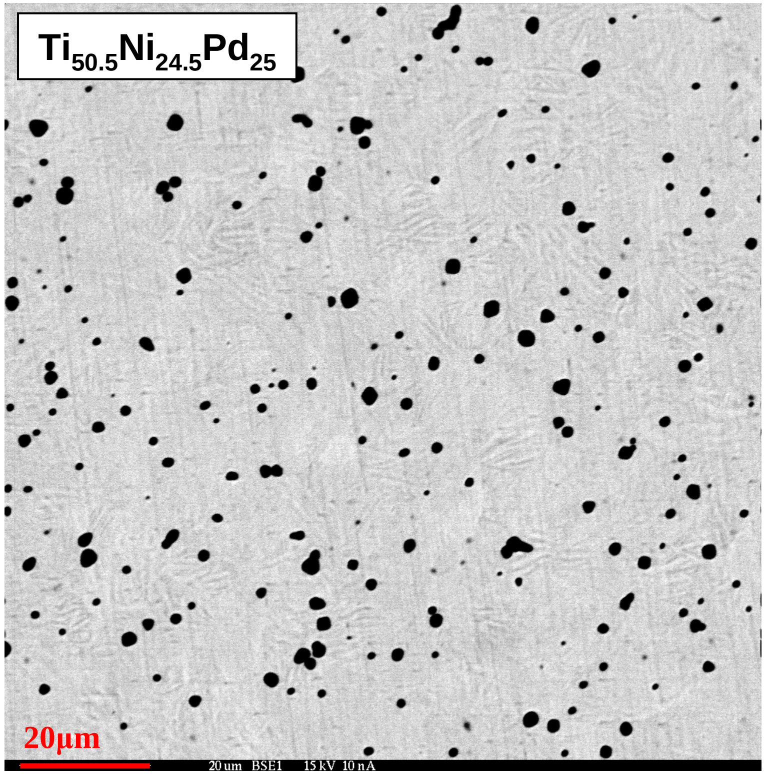
Experimental Results

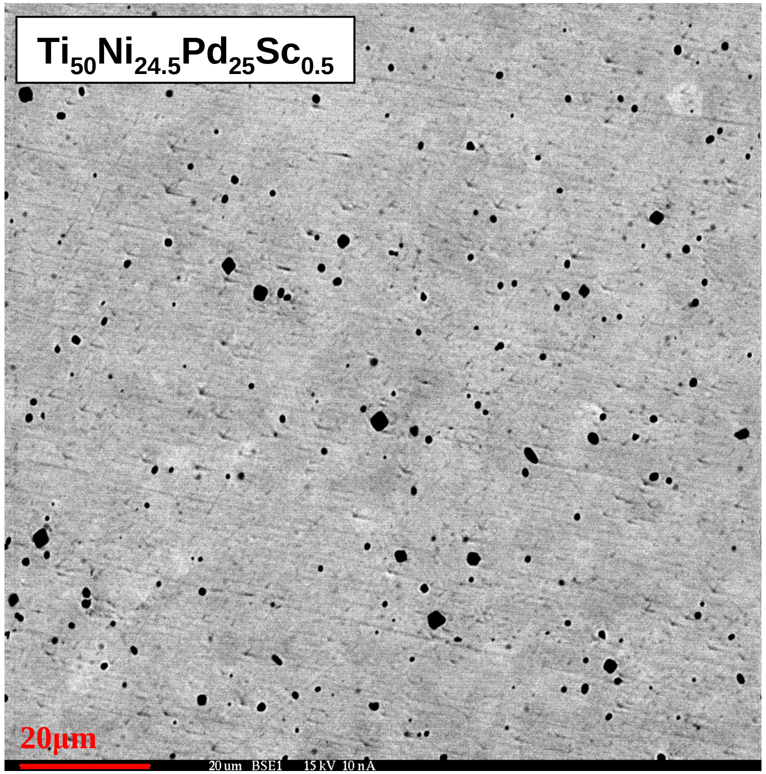
Part I

Addition of 0.5% Sc to the as-extruded $Ti_{50.5}Ni_{24.5}Pd_{25}$

$Ti_{50.5}Ni_{24.5}Pd_{25} \rightarrow Ti_{50}Ni_{24.5}Pd_{25}Sc_{0.5}$

Material Characterization





Grain size ≈ 5 – 15µm (Avg. 10µm)

Volume fraction of precipitates ≈ 3.4%

Average particle size ≈1.48±0.58µm

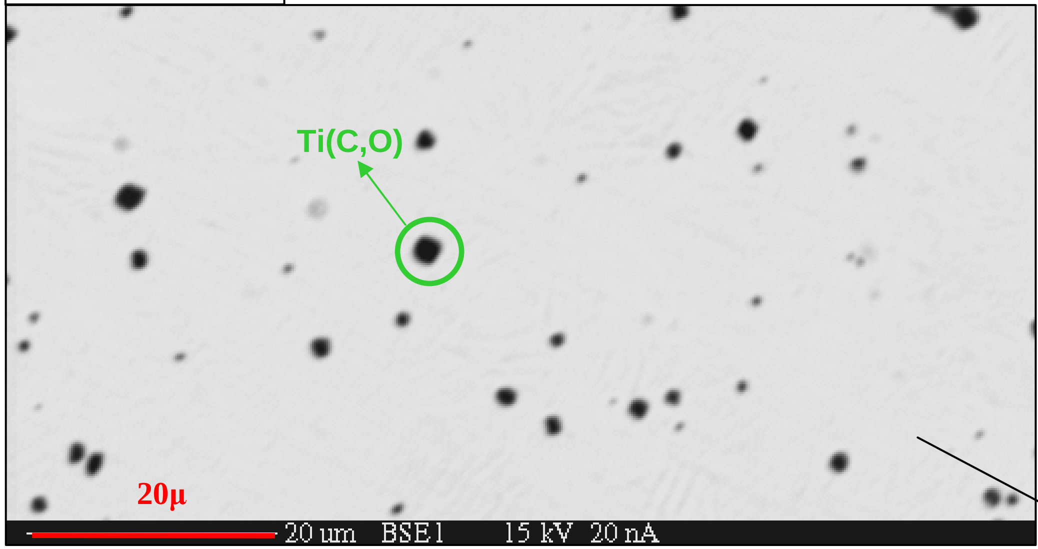
Grain size ≈ 5 – 10µm (Avg. 7µm)

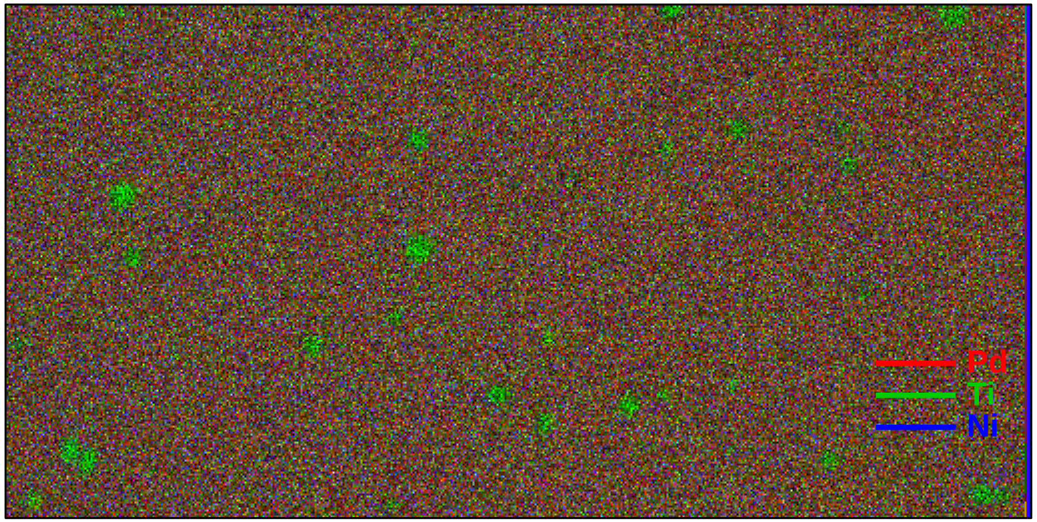
Volume fraction of precipitates ≈ 1.4%

Average particle size ≈0.94±0.44µm

Material Characterization

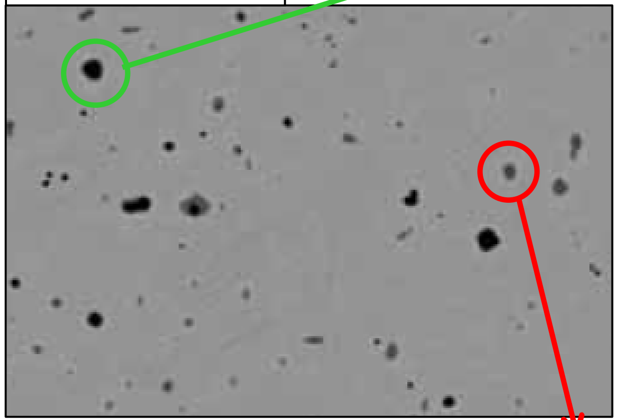
$Ti_{50.5}Ni_{24.5}Pd_{25}$





$Ti_{50.5}Ni_{24.5}Pd_{25}$

BLACK PHASE



Glen Bigelow, M.S. Thesis

GRAY PHASE
$Ti_2(Ni,Pd)$

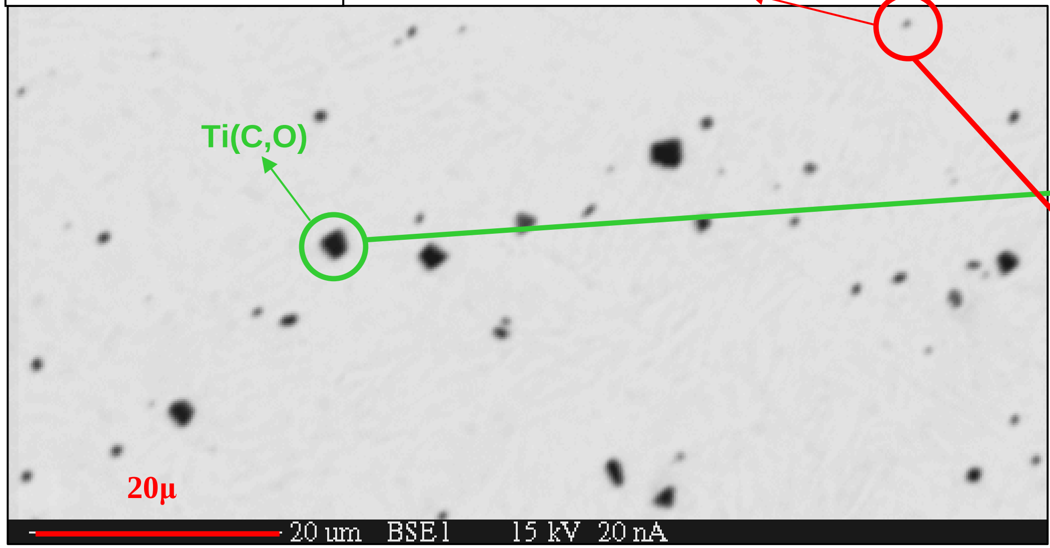
Barely visible gray phases. Hard to define the composition with EDS.

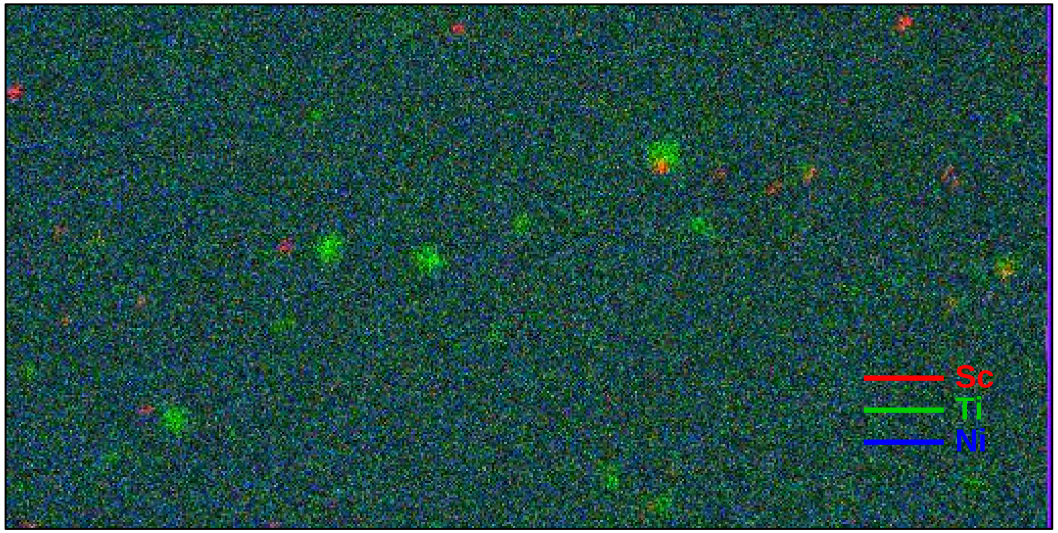
Matrix Composition from WDS Analysis

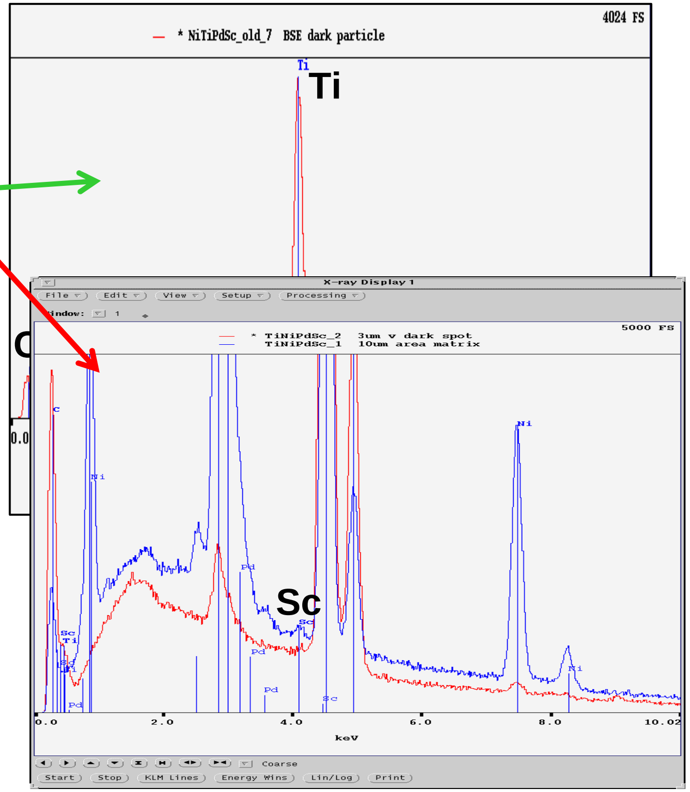
	Ti	Ni	Pd
at. %	49.62 ±0.5	24.42 ±0.25	25.96 ±0.25

Material Characterization

$Ti_{50}Ni_{24.5}Pd_{25}Sc_{0.5}$



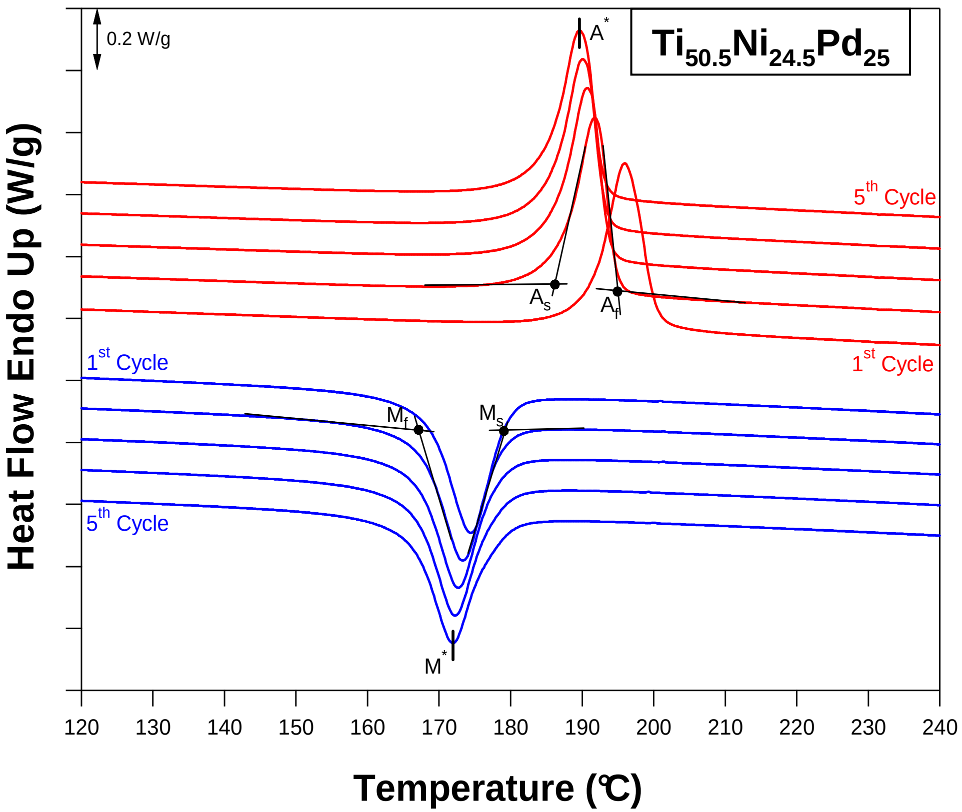


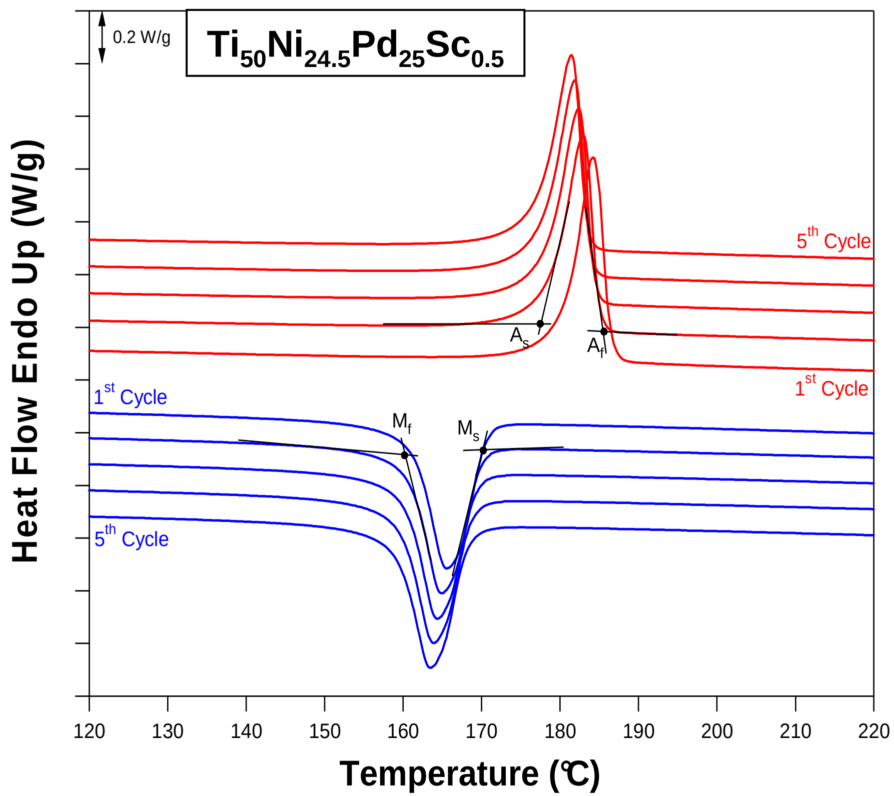


Matrix Composition from WDS Analysis

	Ti	Ni	Pd	Sc
at. %	49.41 ±0.5	25.11 ±0.25	25.02 ±0.25	0.46 ±0.05

Thermo-mechanical Testing





M_s = 179.4℃

A_f = 195.1℃

Decreased transformation temperatures

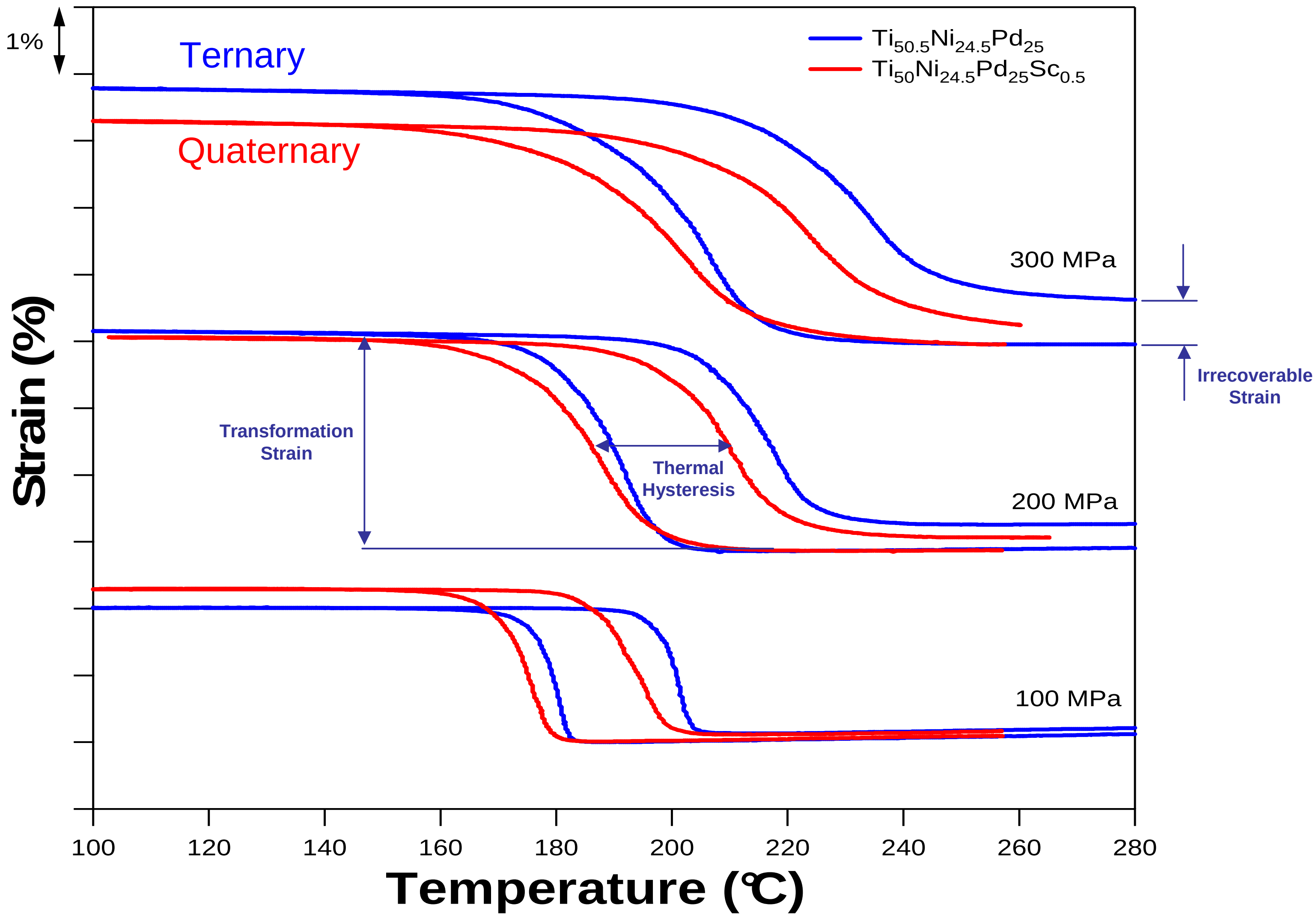
M_s = 170.1℃

Increased cyclic stability

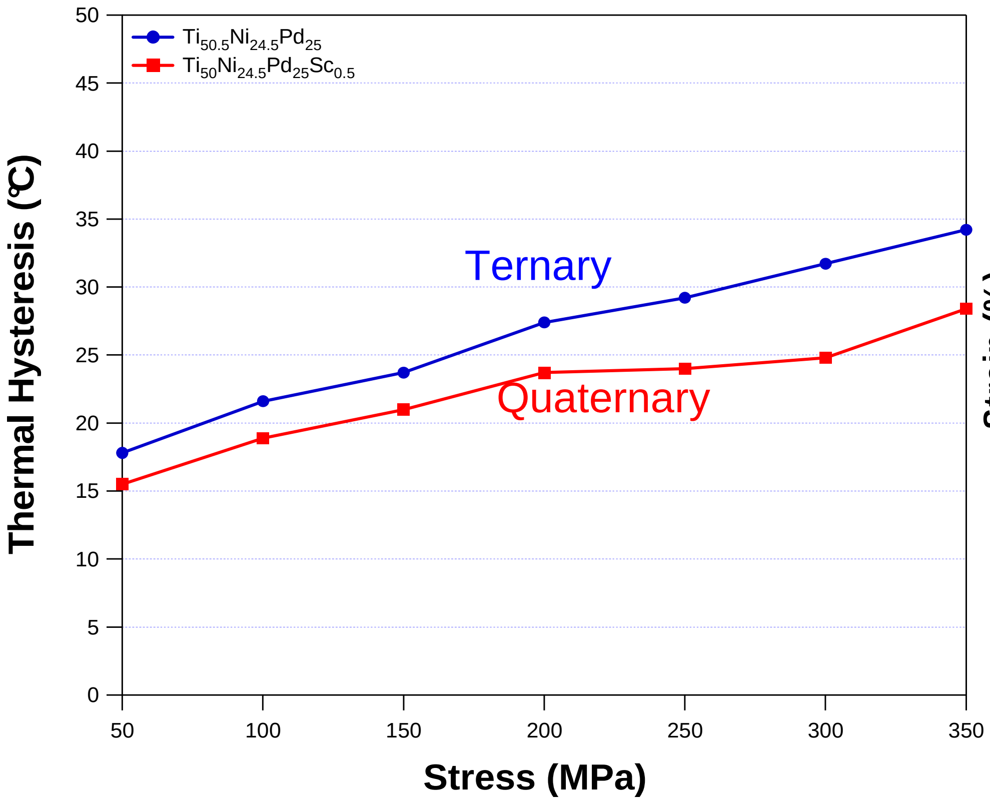
A_f = 185.1℃

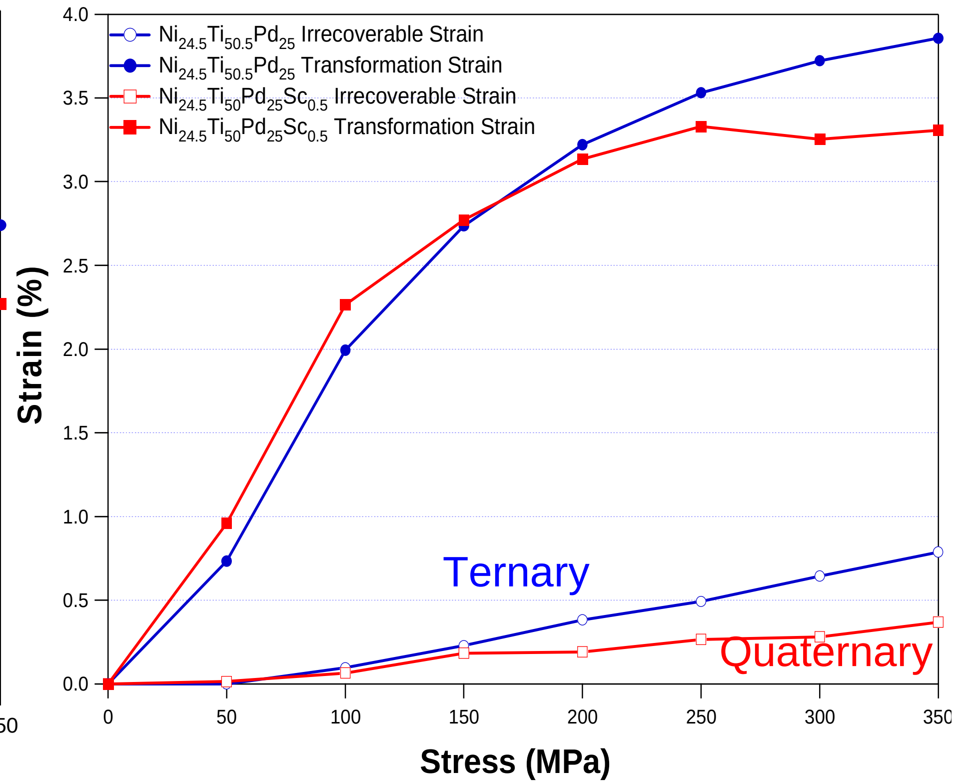
1℃ peak shift per cycle after the 2nd cycle. **0.6℃** peak shift per cycle after the 2nd cycle.

Thermo-mechanical Testing



Thermo-mechanical Testing





Addition of the quaternary element Sc:

- Decreases the amount of accumulated irrecoverable strain
- Decreases the thermal hysteresis
- Slightly decreases the transformation strain levels

Experimental Results

Part II

ECAE of $TiNiPd_{25}Sc_{0.5}$

Heat Treatment of ECAE'ed $TiNiPd_{25}Sc_{0.5}$

ECAE Conditions

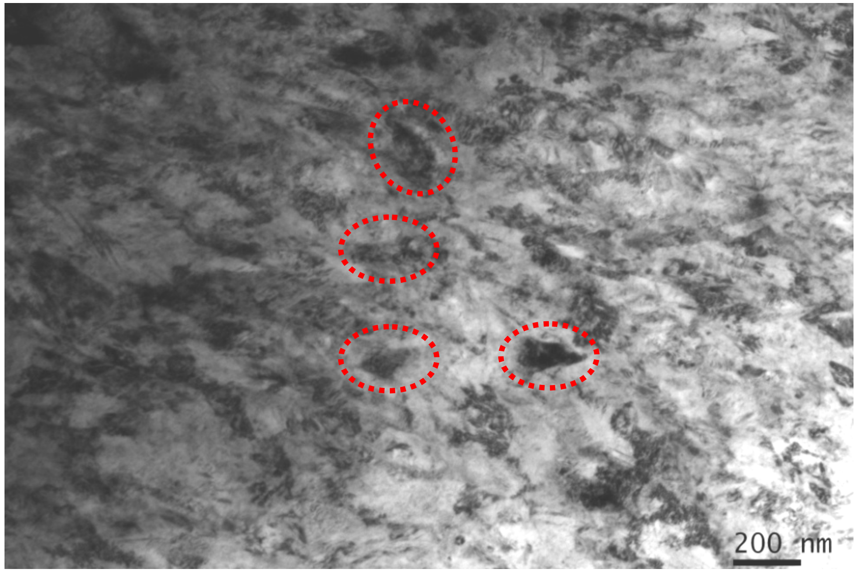
- 4E 425℃ for $Ti_{50}Ni_{24.5}Pd_{25}Sc_{0.5}$
- 4E 425℃ for $Ti_{49.5}Ni_{25}Pd_{25}Sc_{0.5}$

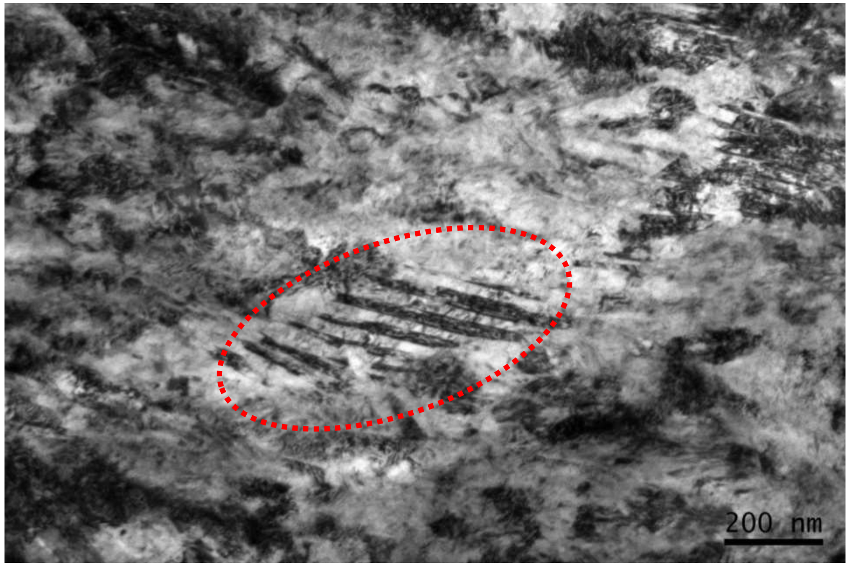
Heat Treatment

300℃, 1 hour followed by air cooling

Material Characterization

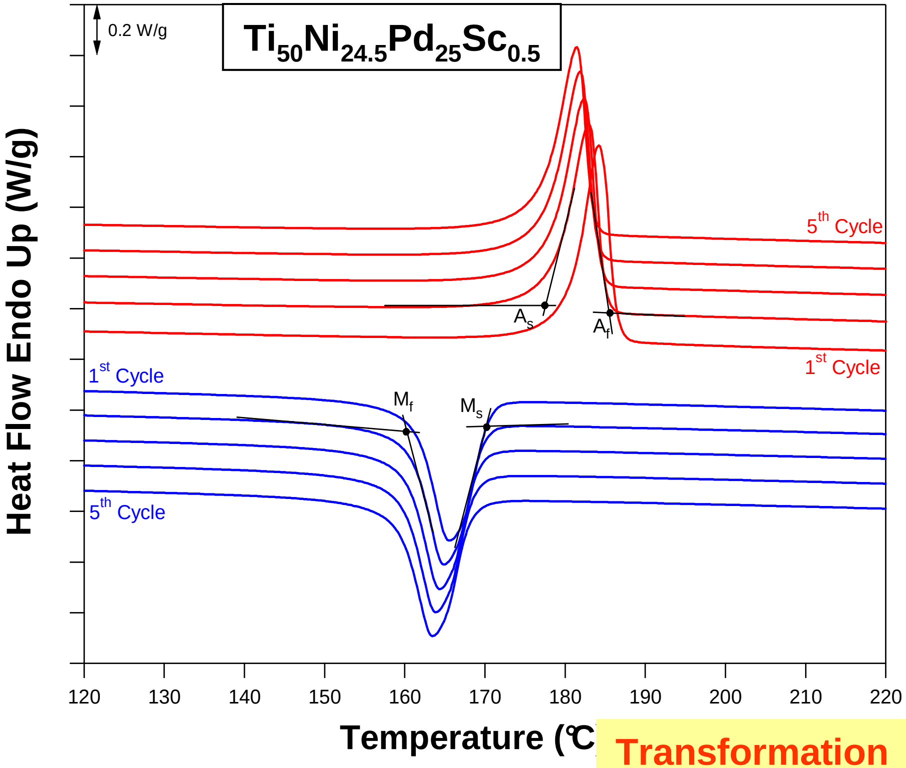
ECAE'ed $Ti_{50}Ni_{24.5}Pd_{25}Sc_{0.5}$, RT

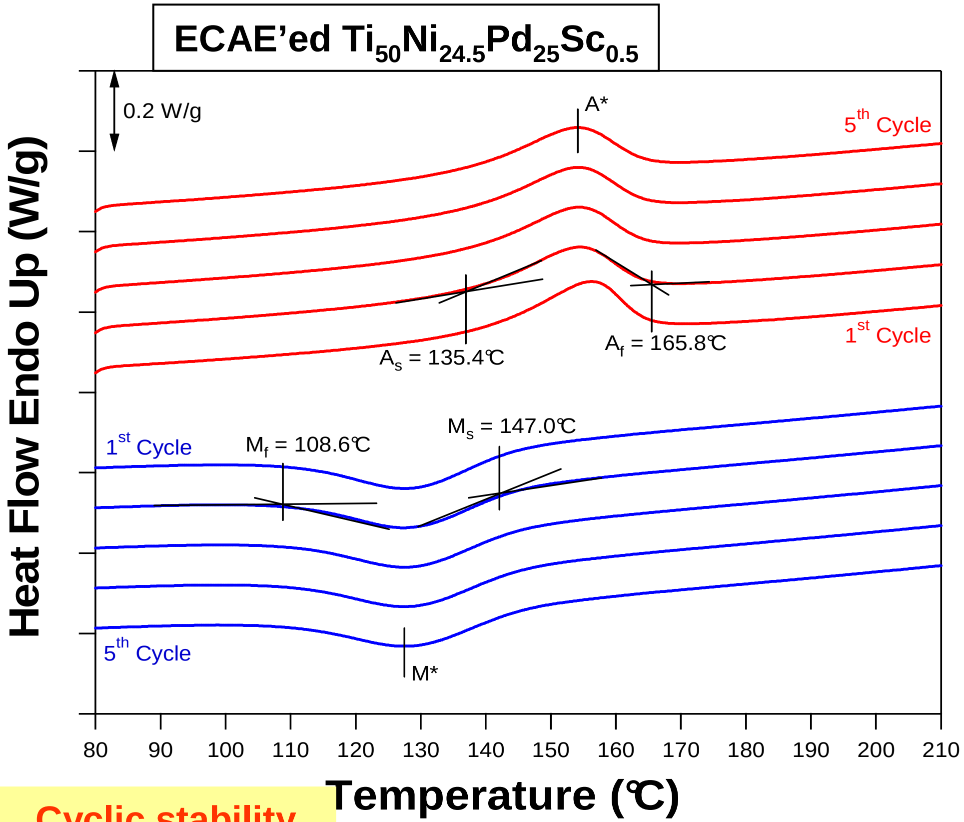




- Nano sized (150-200 nm) grains due to heavy deformation
- B19' martensite structure

Thermo-mechanical Testing





Transformation temperatures decreased further

Cyclic stability increased further

M_s = 170.1℃

A_f = 185.1℃

M_s = 147.0.1℃

A_f = 165.8℃

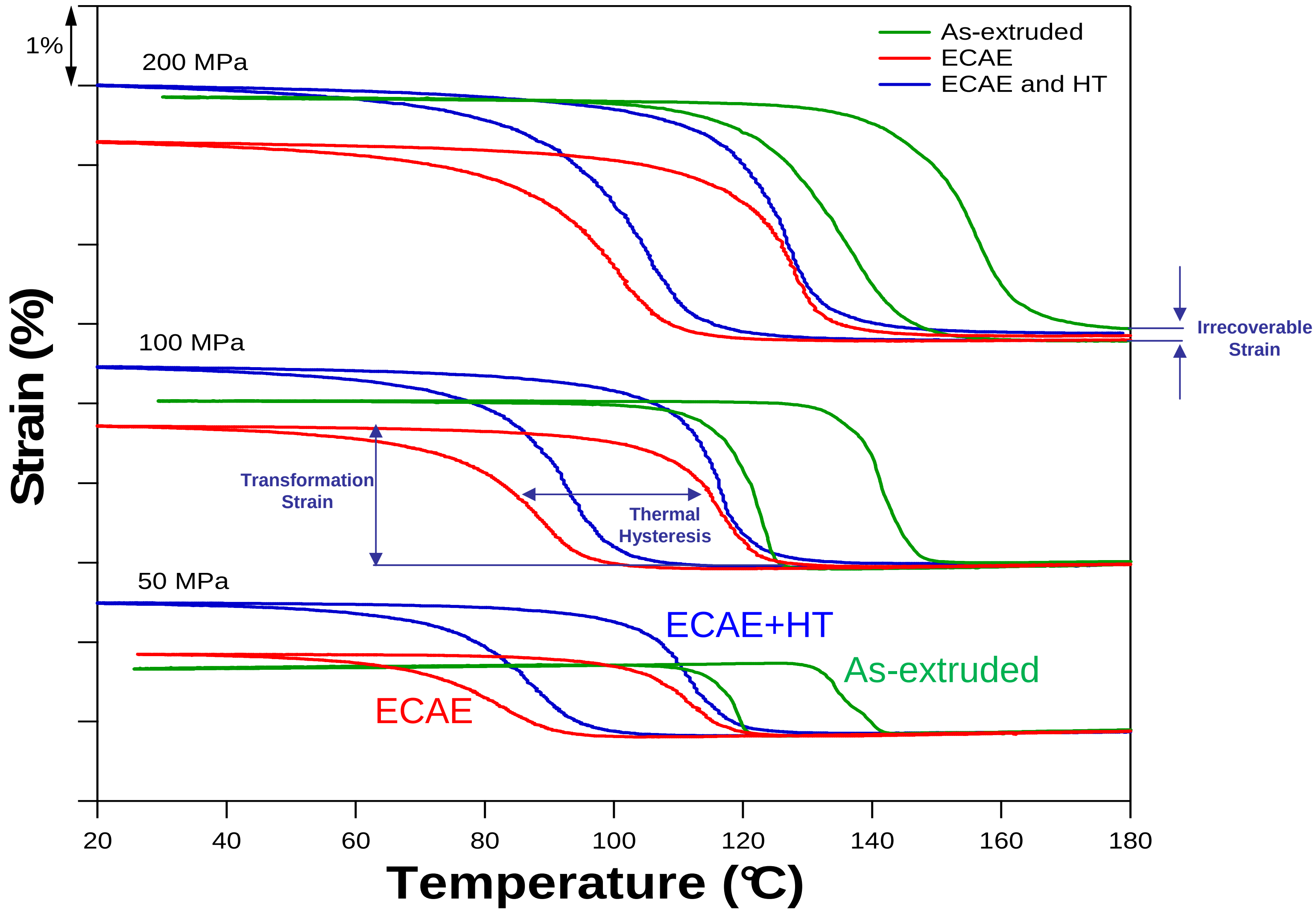
0.6℃ peak shift per cycle after the 2nd cycle.

No peak shift per cycle after the 2nd cycle.

Thermo-mechanical Testing

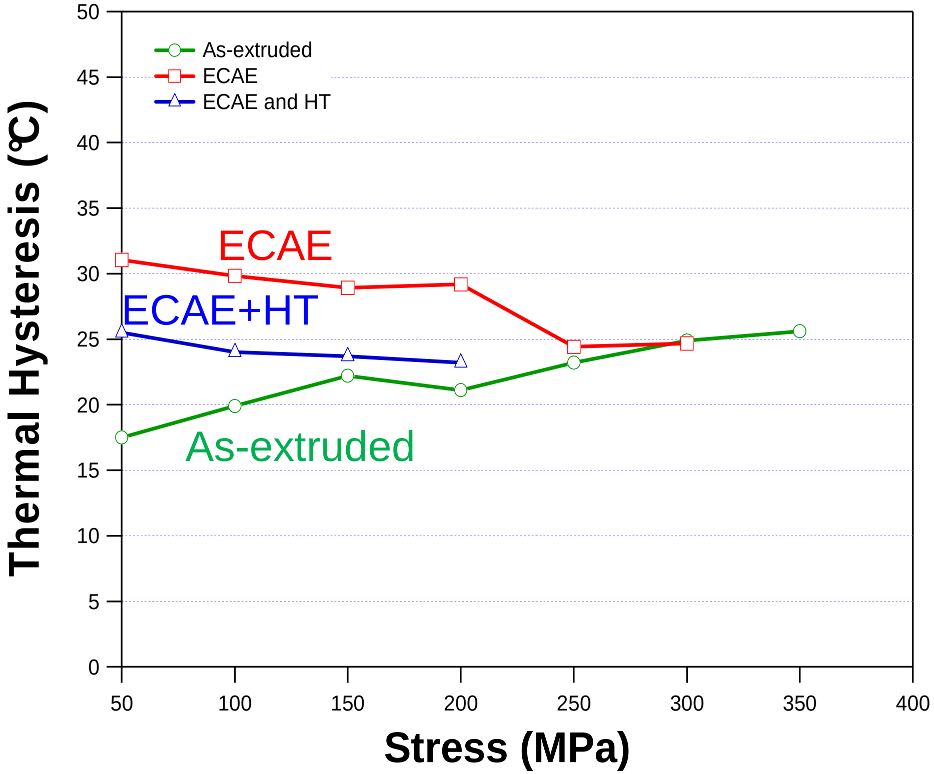
Texas A&M University

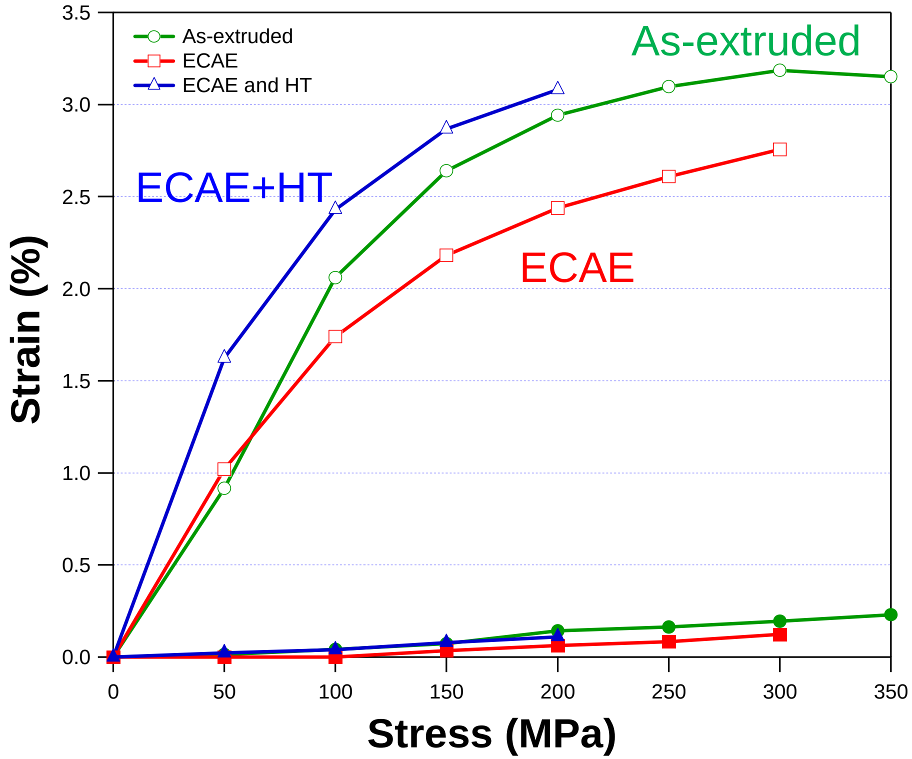
Department of Mechanical Engineering



Thermo-mechanical Testing

Texas A&M University | Department of Mechanical Engineering





Composition: $Ti_{49.5}Ni_{25}Pd_{25}Sc_{0.5}$

- ECAE depresses the transformation temperatures
- ECAE decreases the amount of accumulated irrecoverable strain
- Heat treatment after ECAE increases the transformation temperatures and improves the strain output beyond that of the as-extruded material for a given stress level.

Conclusions

- Quaternary addition of Sc improved the shape memory properties of TiNiPd by improving thermal and mechanical cyclic stability.

- Quaternary addition of Sc changed the structure of martensite from B19 to B19'.

- ECAE followed by heat treatment further improved the shape memory properties of TiNiPdSc.

- Both methods can eliminate the necessity of costly and long training cycles before the HTSMA is used in an application.

Future Work

- Addition of different levels of Sc
- Different ECAE routes and heat treatments
- Isobaric thermal cyclic tests

Thank you!

Appendix

Element	Atomic Number	Atomic weight (g/mol)	Density	Melting Point (℃)	Crystal Structure	Bond Length (pm)	Atomic radius (pm)	E (GPa)	G (GPa)	Hardness (B)
Titanium	22	47,867	4,506	1668	HCP	289,6	140	116	44	716
Nickel	28	58,693	8,908	1455	FCC	249,2	135	200	76	700
Palladium	46	106,42	12,023	1555	FCC	275,1	140	121	44	373 ?
Scandium	21	44,955	2,985	1541	HCP	321,2	160	74,4	29,1	750