Characterization of a New Phase and Its Effect on the Work Characteristics of a Near-Stoichiometric $Ni_{30}Pt_{20}Ti_{50}$ High-Temperature Shape Memory Alloy (HTSMA)

A new phase observed in a nominal $Ni_{30}Pt_{20}Ti_{50}$ (at.%) high temperature shape memory alloy has been characterized using transmission electron microscopy and 3-D atom probe tomography. This phase forms homogenously in the B2 austenite matrix by a nucleation and growth mechanism and results in a concomitant increase in the martensitic transformation temperature of the base alloy. Although the structure of this phase typically contains a high density of faults making characterization difficult, it appears to be trigonal (-3m point group) with a_o~1.28 nm and c_o~1.4 nm. Precipitation of this phase increases the microhardness of the alloy substantially over that of the solution treated and quenched single-phase material. The effect of precipitation strengthening on the work characteristics of the alloy has been explored through load-biased strain-temperature testing in the solution-treated condition and after aging at 500 ºC for times ranging from 1 to 256 hours. Work output was found to increase in the aged alloy as a result of an increase in transformation strain, but was not very sensitive to aging time. The amount of permanent deformation that occurred during thermal cycling under load was small but increased with increasing aging time and stress. Nevertheless, the dimensional stability of the alloy at short aging times (1-4 hours) was still very good making it a potentially useful material for high-temperature actuator applications. *(Support by the NASA Fundamental Aeronautics Program, Supersonics Project and the analytical facilities at the Center for Advanced Research and Technology at the University of North Texas are gratefully acknowledged.)*

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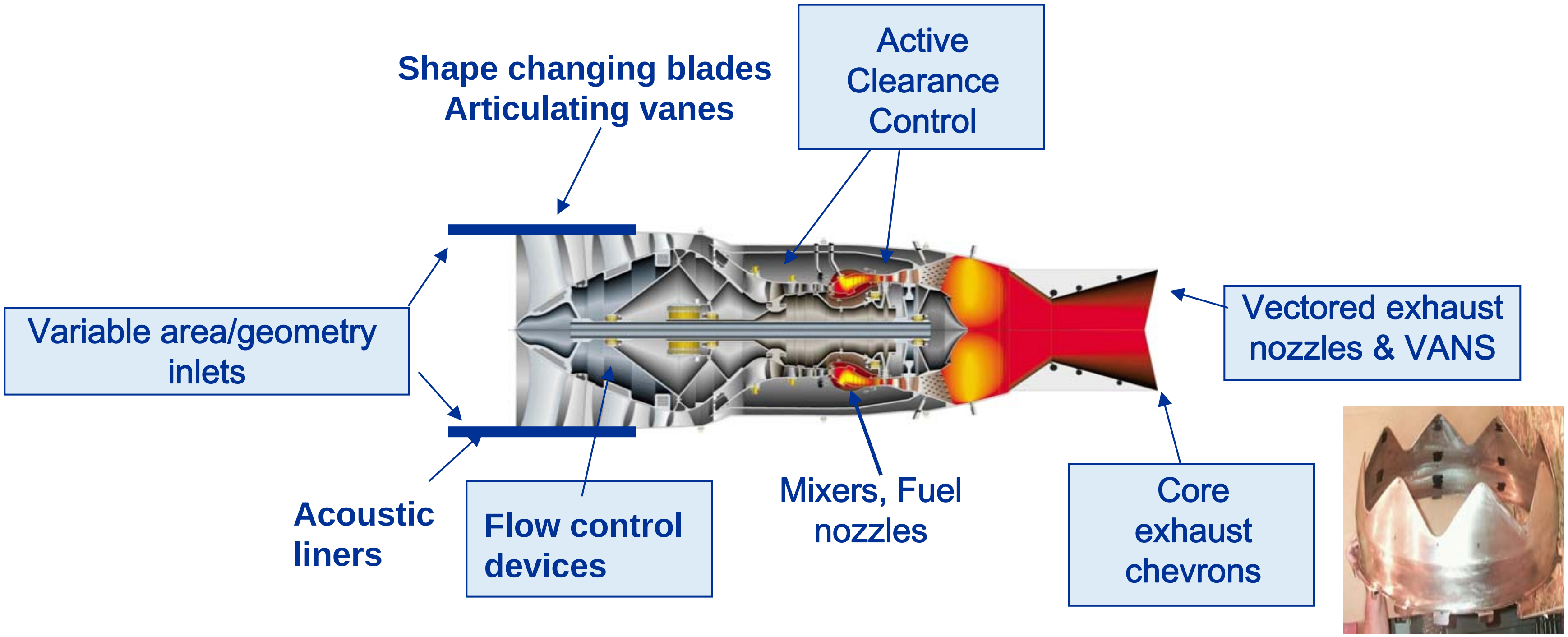
High-Temperature Shape Memory Alloys are an enabling technology to a host of “smart” structures in jet engines

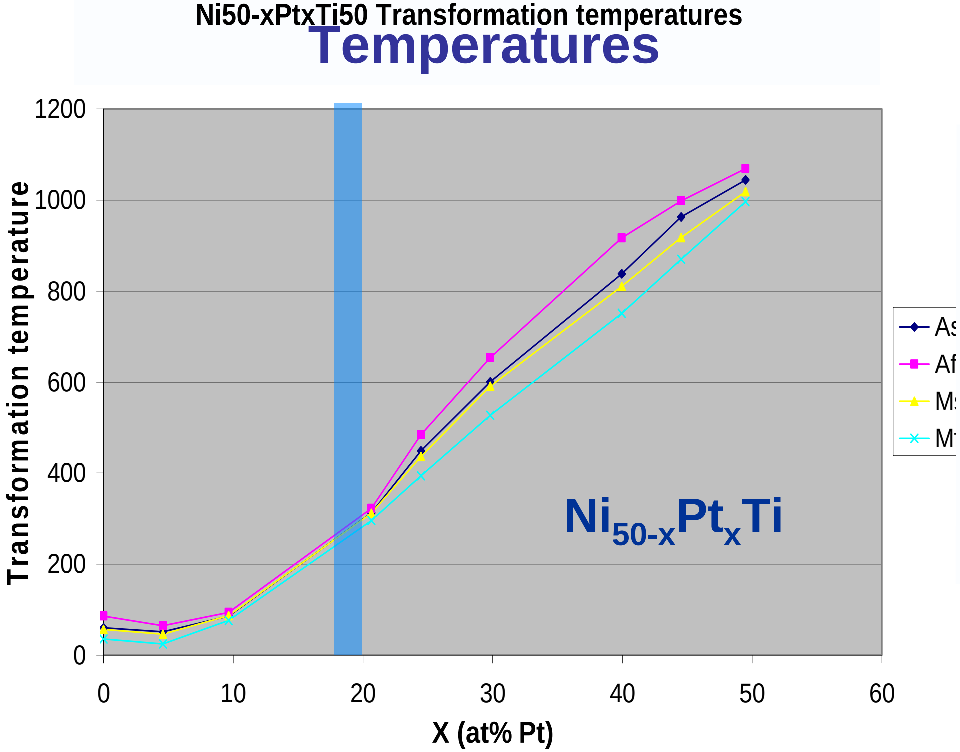


Advantages of HTSMA

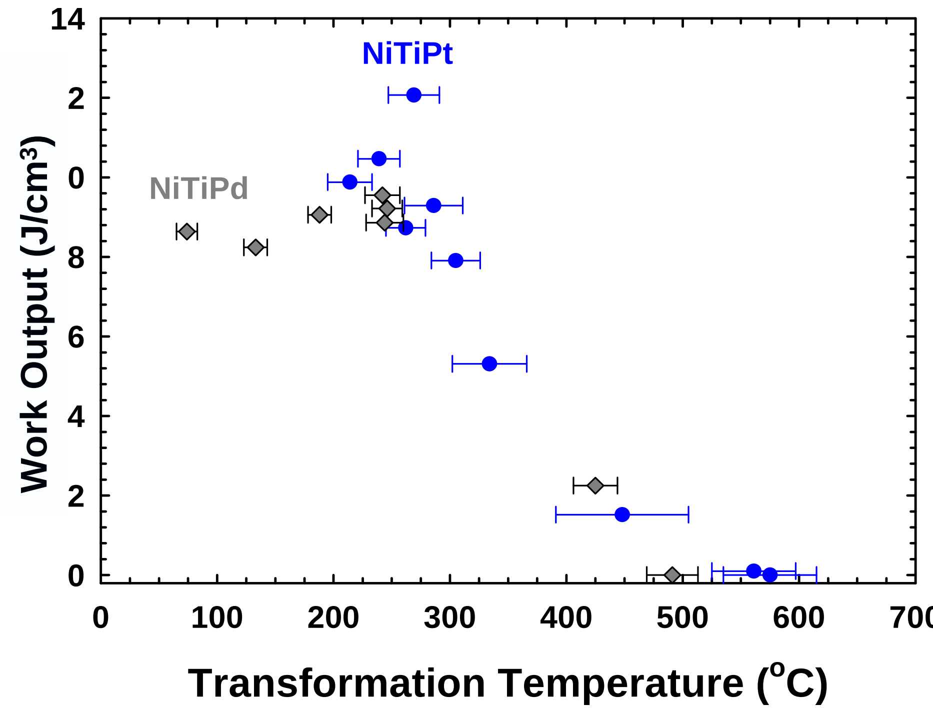
- **High force per volume/weight - compact, lightweight**
- **Solid State - eliminates hydraulics, pneumatics, mechanical systems**
  simple, frictionless, quiet, maintenance free
- **Passive control - eliminates sensors, electronics**
- **Can be actively controlled for high-force, precision movements**

Introduction

Effect of Pt on Transformation Temperatures



Work Behavior of HTSMA



10 J/cm³ is equivalent to a 44 mil rod, 25” long, lifting a 110 lb weight 0.5”

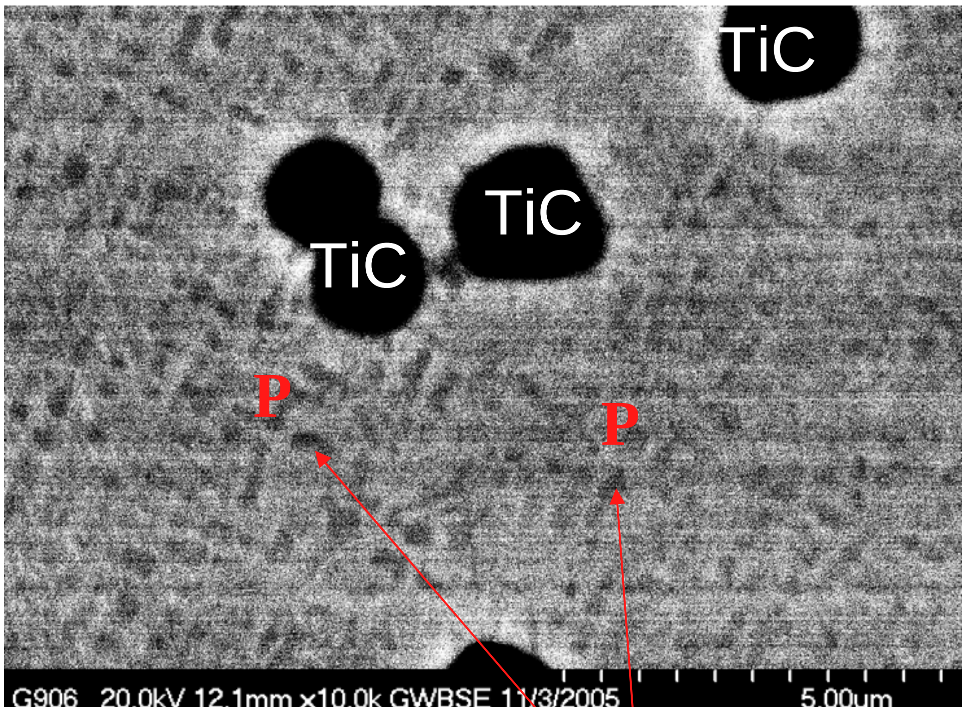
4 J/cm³ is equivalent to a 44 mil rod, 25” long, lifting a 44 lb weight 0.5”

Experimental

- **Alloys vacuum induction melted in graphite crucibles.**
- **Ingots homogenized and then canned and extruded.**
- **Compositional analysis (at.%) – 30.09Ni-19.33Pt-49.76Ti-0.46C-0.29O-0.03N-0.01Cu-0.03Fe.**
- **Thermal analysis (DSC/DTA)**
- **Microstructural Analysis (SEM, TEM, 3DAP, HAADF, HRTEM)**
- **Mechanical properties (hardness, load biased thermal cycling tests, etc).**

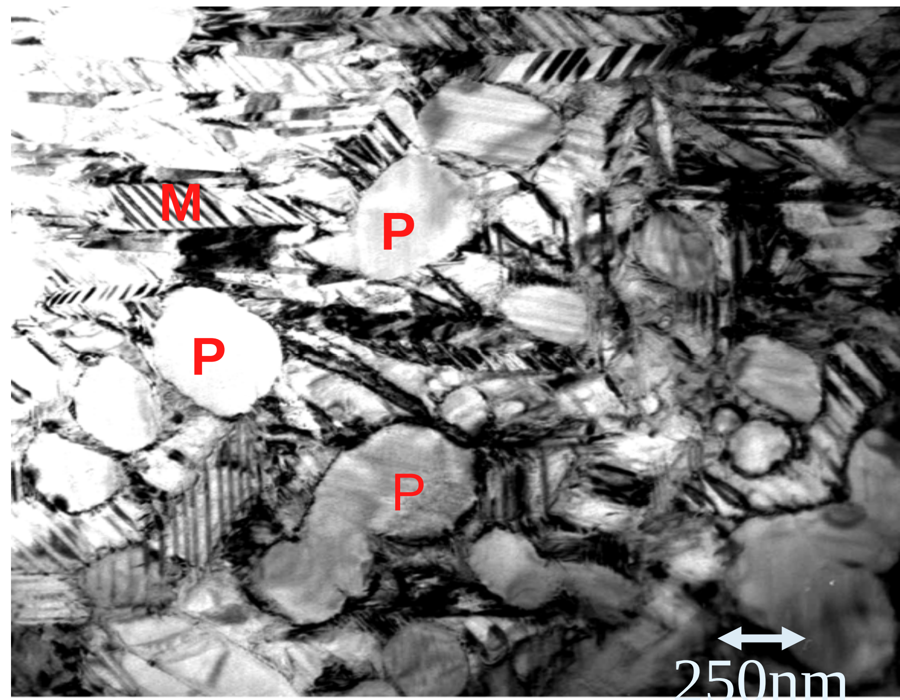
Microstructure: Alloy aged at 500ºC/1024h

SEM



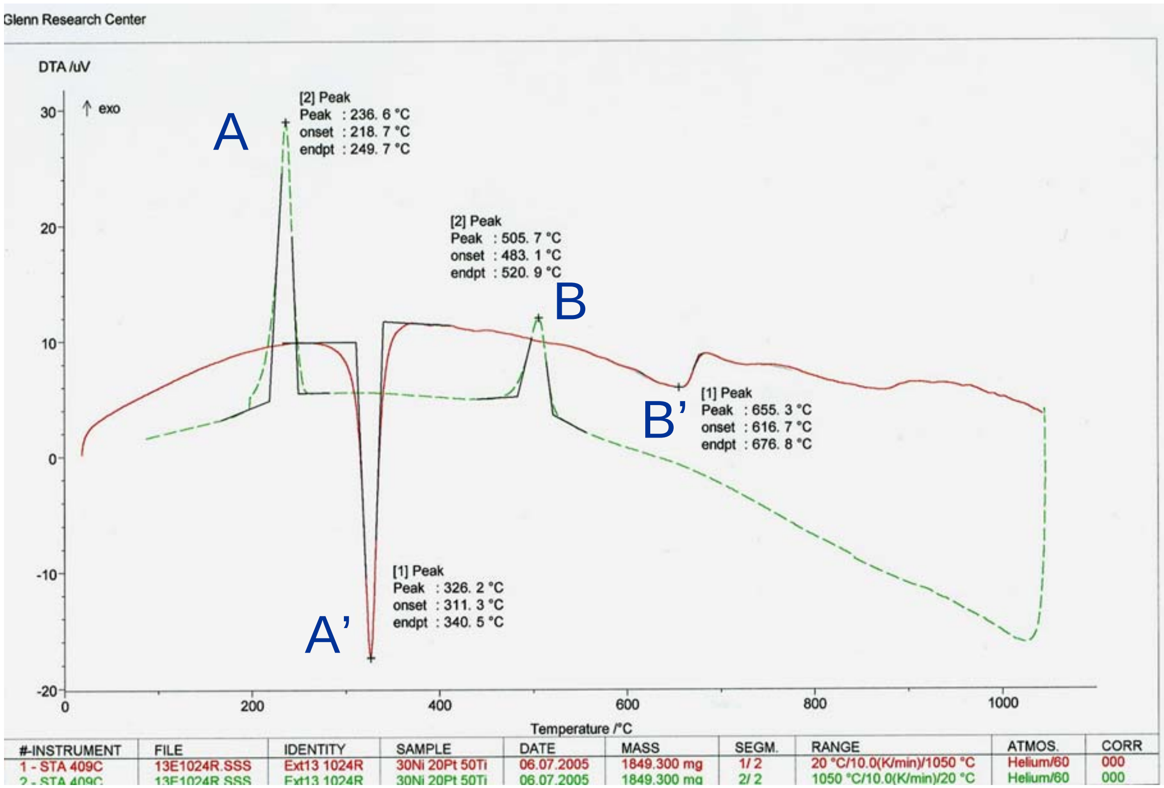
Fine ~0.5μm phase P (black)

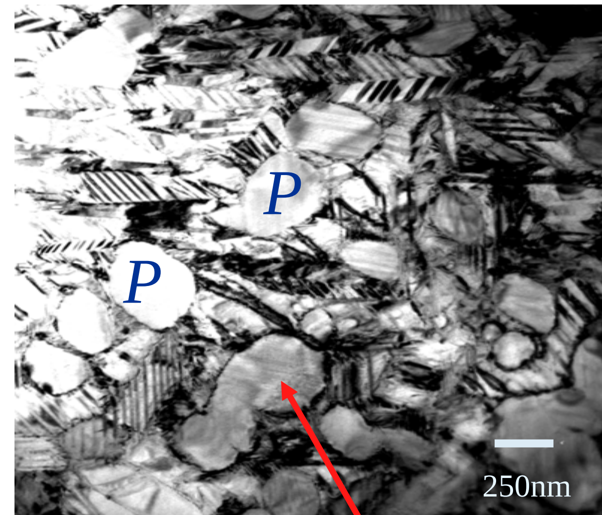
TEM



Fine Phase (P) in a martensite matrix (M)

DTA Scan: Ext. 13 sample annealed 500º C/1024h

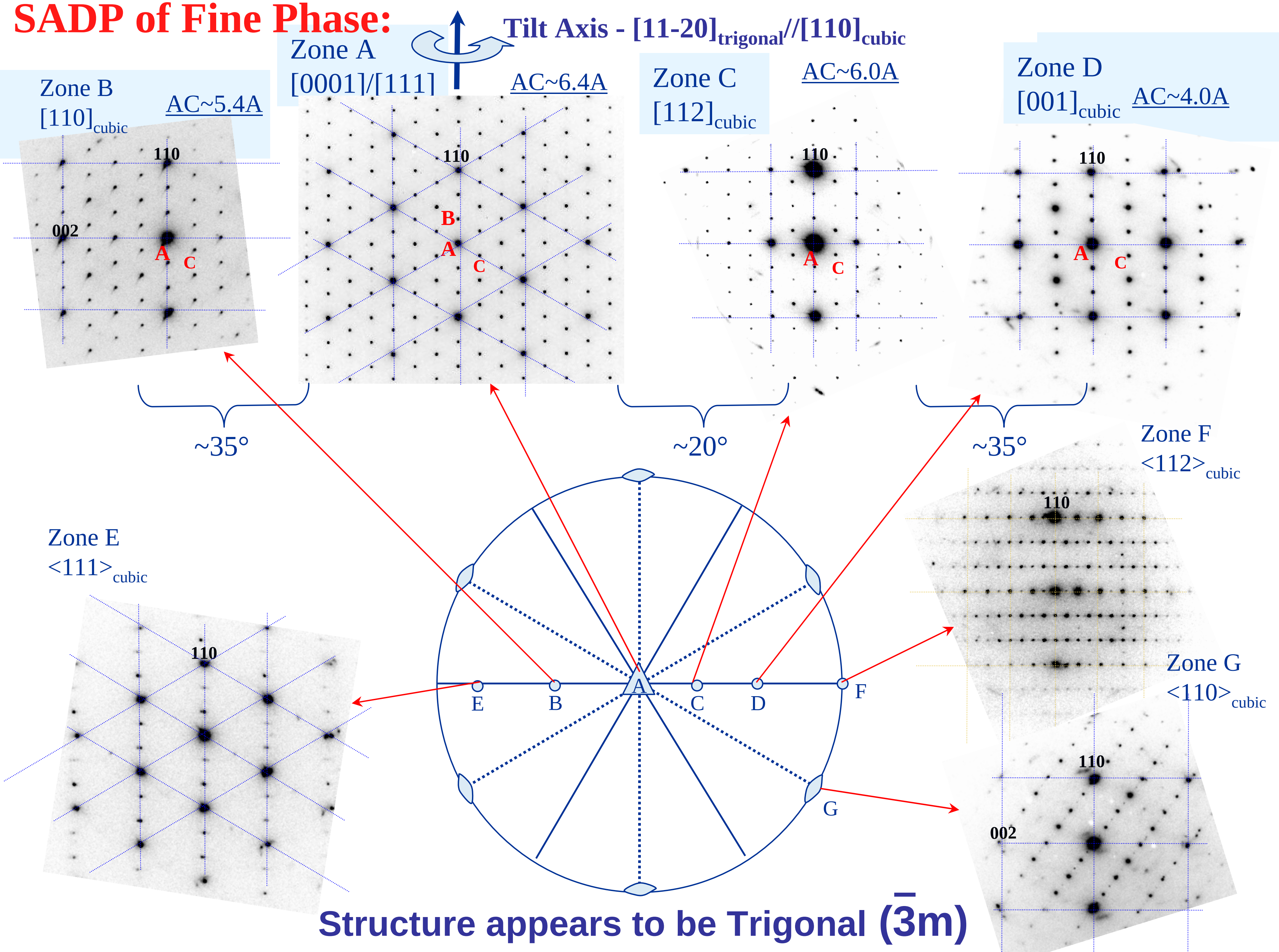




Fine phase, P

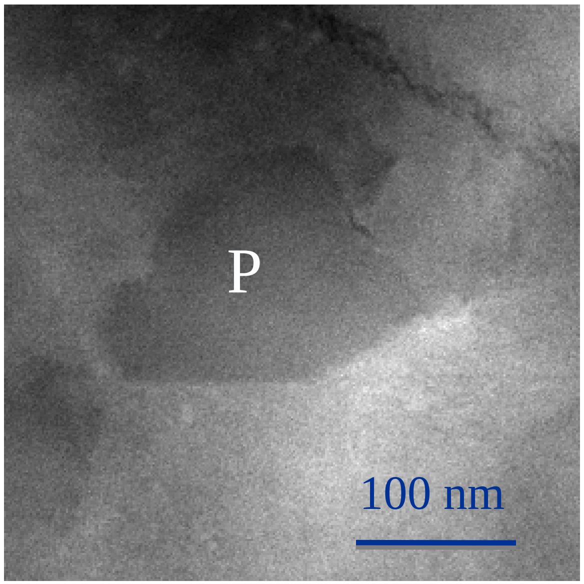
- **The low temperature peaks (A,A') are due to the austenite-to-martensite and the reverse transformation.**
- **The higher temperature peaks (B,B') are likely due to the fine phase P.**
- **Quenching from 800°C results in elimination of the fine phase P.**

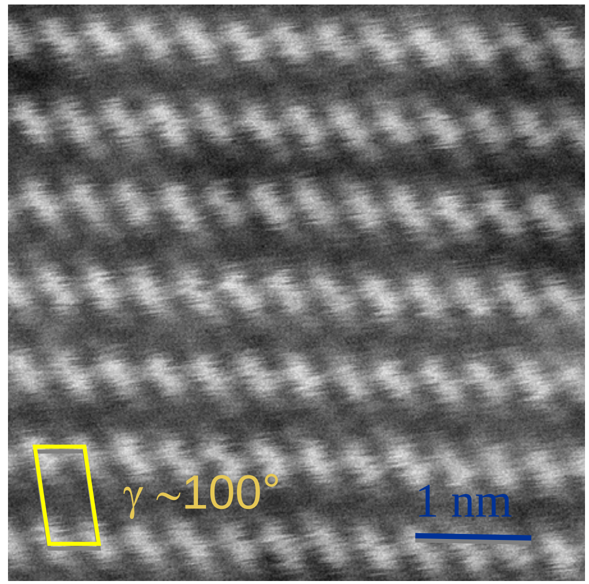
SADP of Fine Phase:

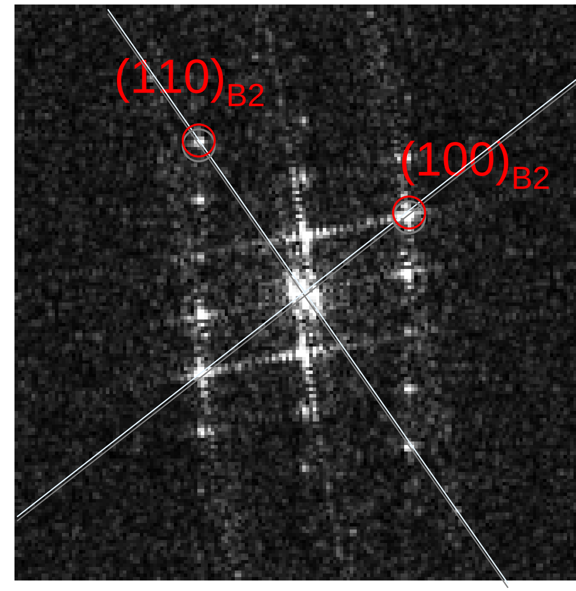


Structure appears to be Trigonal ($\bar{3}m$)

HAADF Imaging of the precipitates – contrast proportional to Z^2 (Z_{Pt}=78, Z_{Ni}=28, Z_{Ti}=22)



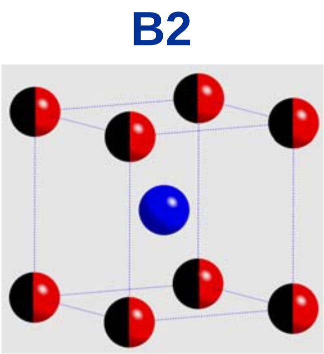


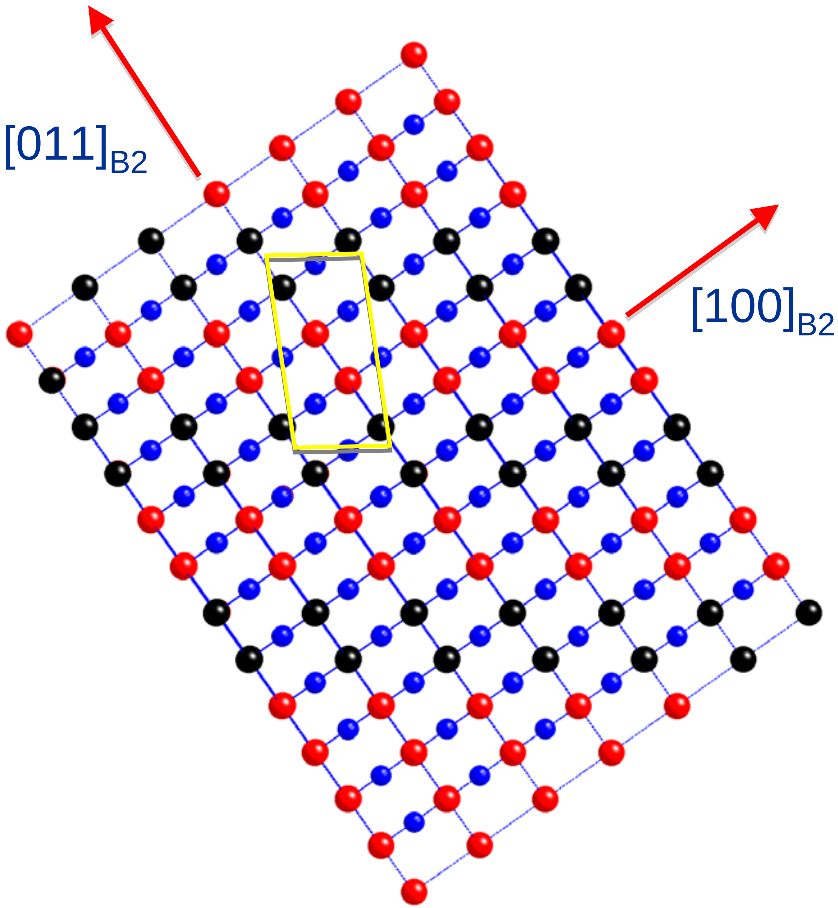


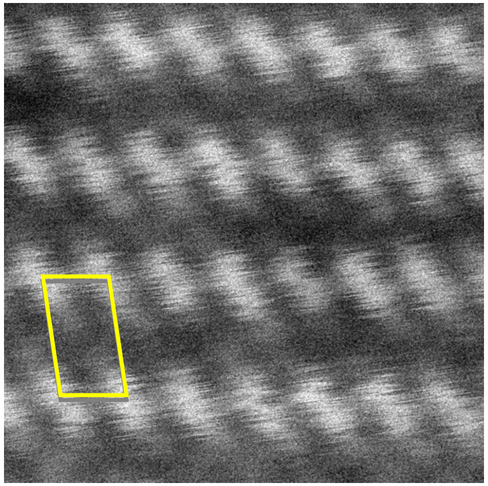
- Observation of precipitate in the “cubic” [110] orientation.
- HAADF contrast dominated by dumbbell-like motif represents two closely positioned Pt rich columns.
- 2D projection has oblique symmetry a ~ 3.7Å, b ~ 6.9Å, γ ~100°.
- Fourier transform consistent with diffraction pattern G, [110]cubic.

Hypothesis: Fine Phase P is

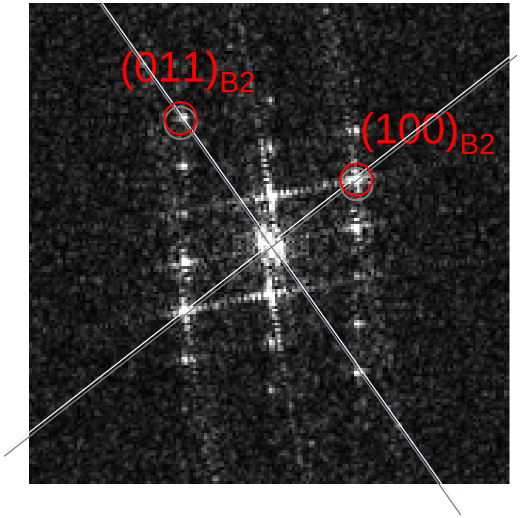
- Trigonal based on a B2 crystal structure where Ni and Pt substitute each other on the Ni sub-lattice.
- In addition, there is an ordering of Pt on the Ni sub-lattice of B2 structure.





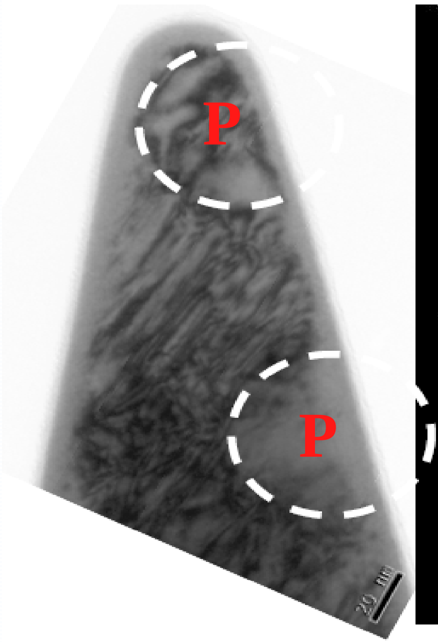


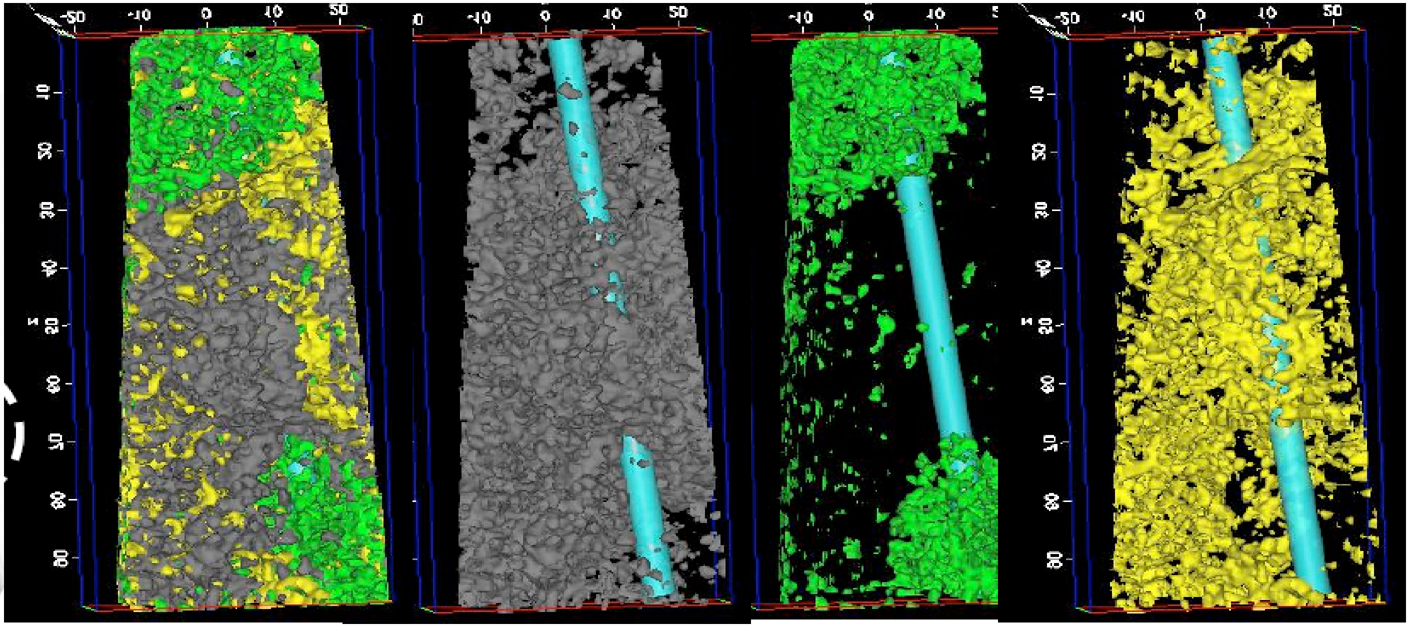
HAADF image

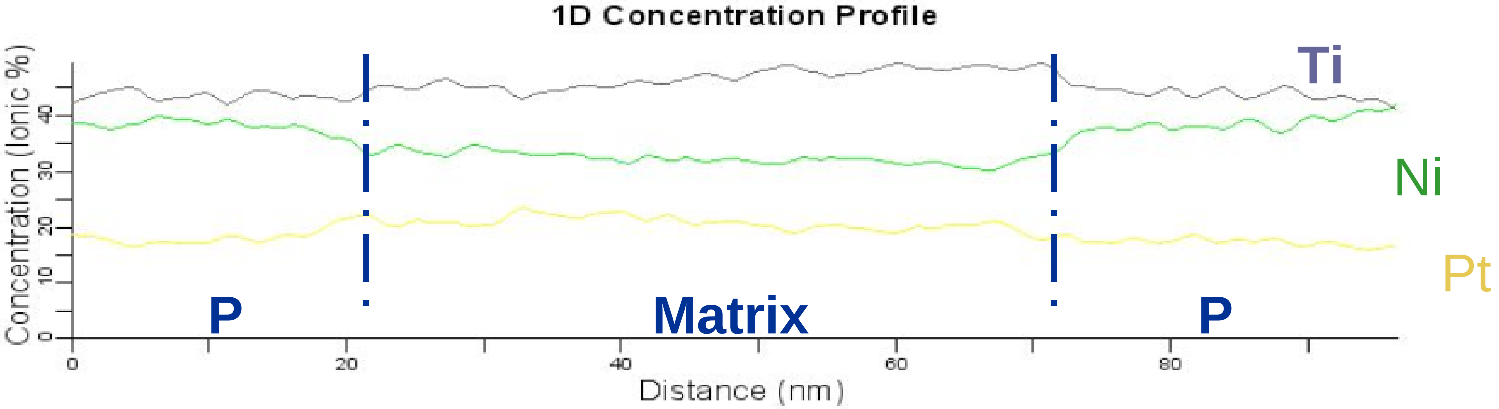


3D Atom Probe Analysis:

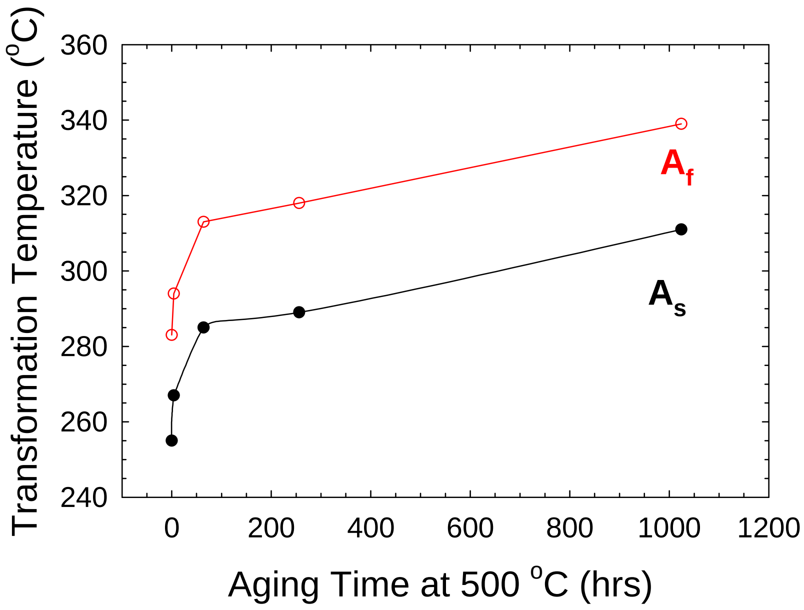
Fine phase is rich in Ni and slightly depleted in Pt







Transformation Temperatures: Effect of Aging Time at 500°C on the Stress-Free Trans. Temp.

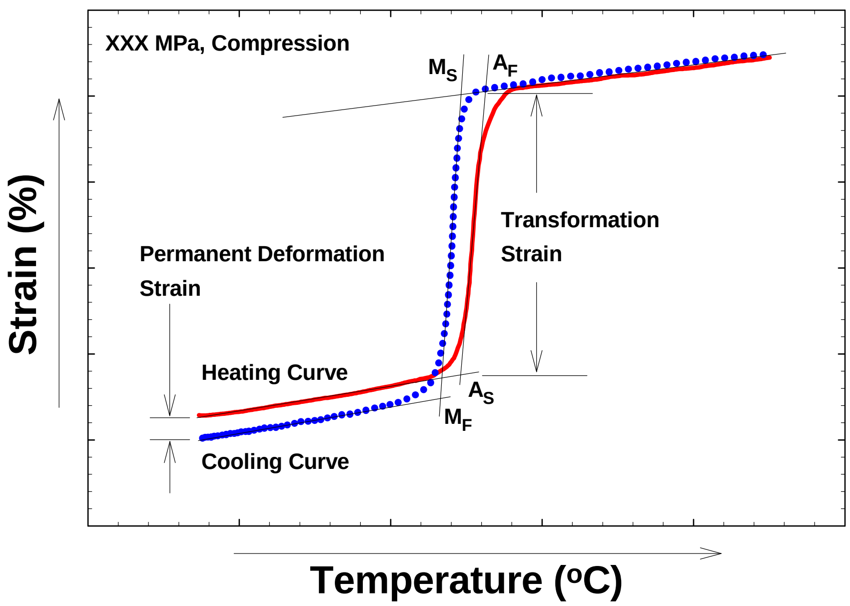


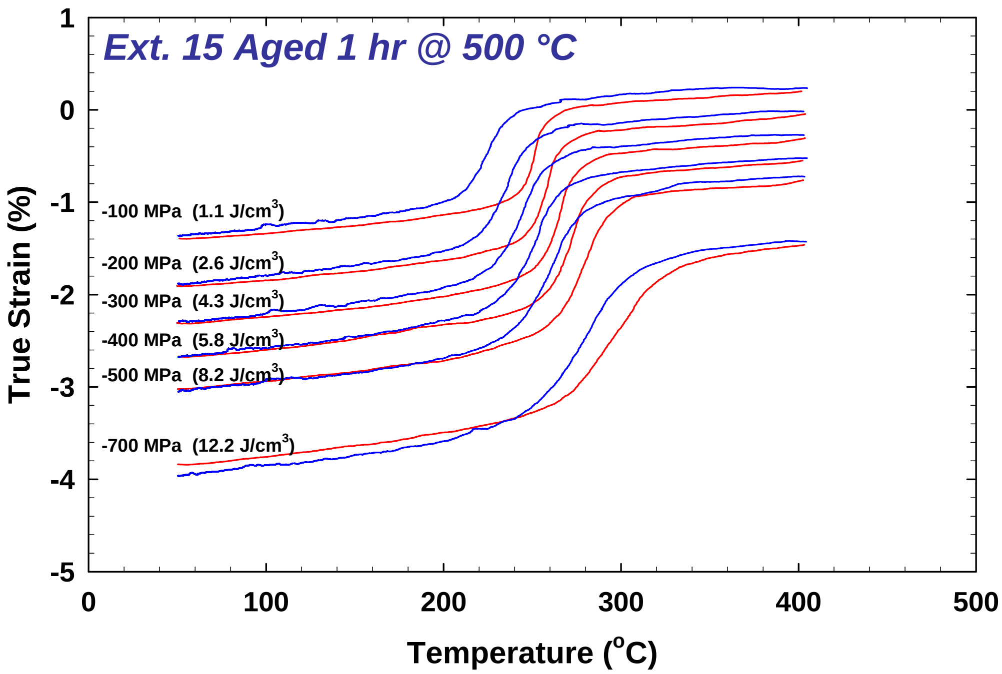
Alloy Soln. Treated 800°C/30min + WQ + Aged 500°C/(0-1024h)

Transformation Temperatures

Time (hrs)	A_p	A_s	A_f	M_s	M_f	M_p
0	267	255	283	229	200	214
4	279	267	294	239	211	225
64	298	285	313	260	232	245
256	303	289	318	266	237	251
1024	326	311	339	288	256	273

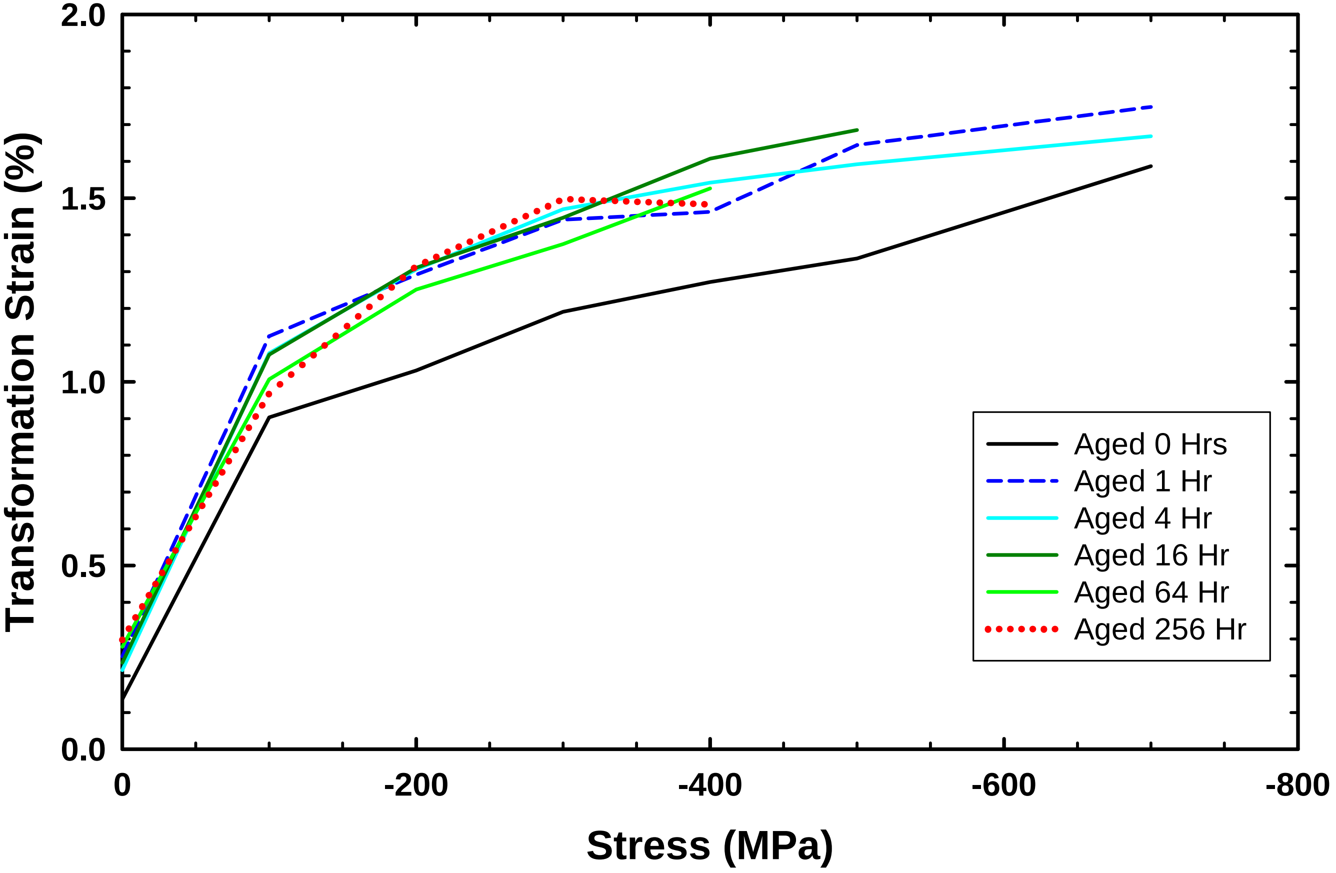
Load Biased Thermal Cycling in Compression to Determine Shape Memory Response Under Load (For Actuator Type Applications)





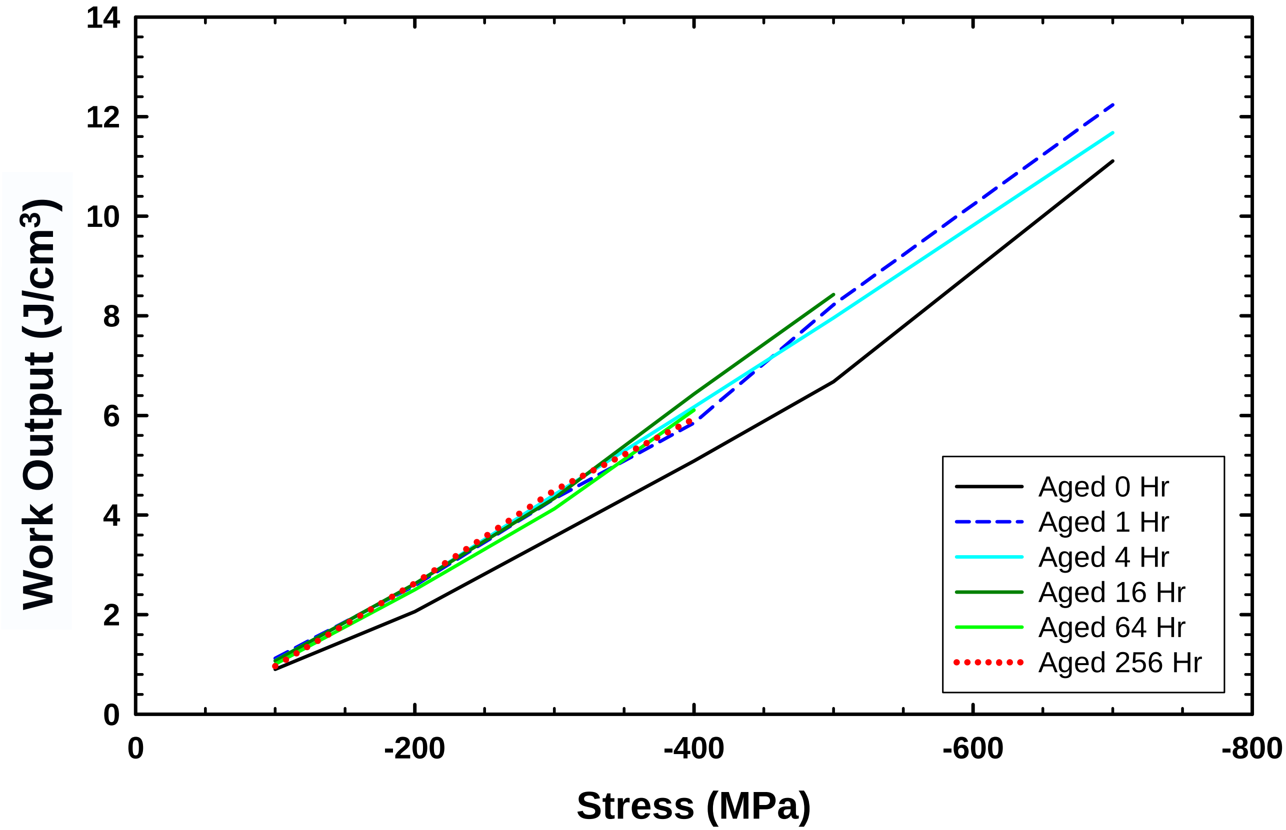
- Definition of properties determined from load-biased thermal cycling under a constant load.
- Indicated for each curve is the Applied stress level and the equivalent work output (= transformation strain x applied stress).

Transformation Strain vs. Stress after Aging for Various Times at 500 °C



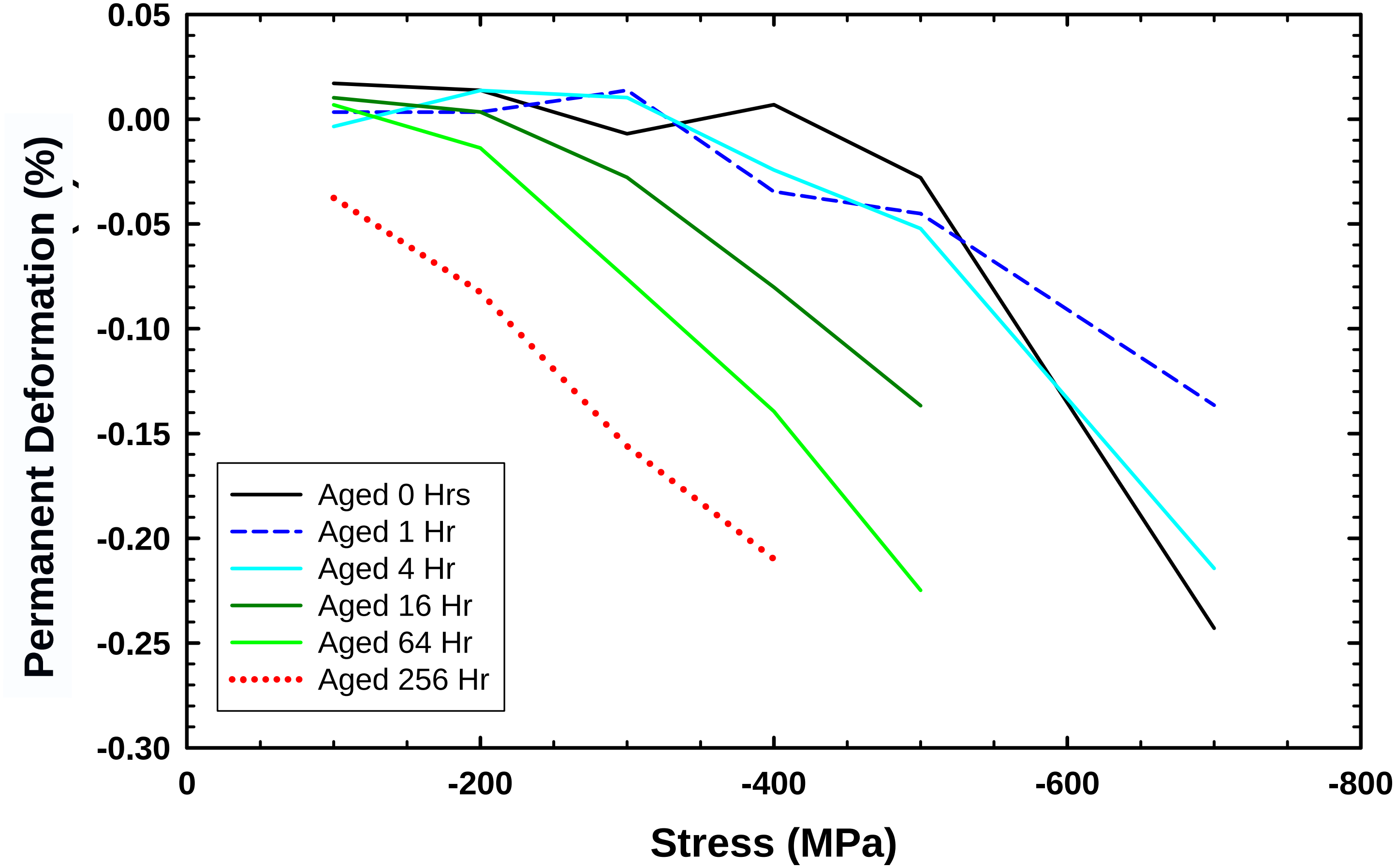
- **Aging increases the amount of transformation strain.**

Work Output vs. Stress after Aging for Various Times at 500 °C



- **Work output (proportional to transformation strain) increases with increasing stress and is independent of aging time.**

Permanent Deformation vs. Stress after Aging for Various Times at 500°C



- **Aging at times longer than 4 hr causes an increase in the amount of dimensional instability (equivalent to permanent deformation) that occurs with each load-biased thermal cycle**

Summary

- A new phase has been identified in a $Ni_{30}Pt_{20}Ti_{50}$ alloy.
- The new phase is Ni rich and slightly Pt lean (lower Z contrast).
- The crystal structure appears to be Trigonal ($\bar{3}m$). Atom positions are based on the B2 structure but with ordering of Pt on Ni sites.
- Aging increases the transformation temperatures for the material
  - *Enhancing the optimum use temperature of the material.*
- Aging also results in an increase in the transformation strain, and hence work performed at a given stress level, compared to the precipitate-free material.
- Unfortunately, aging does increase the amount of permanent deformation that occurs at a given stress level, however the effect is minimal at aging times of 1-4 hrs.

For use in high-temperature actuator applications or adaptive structures – the optimum condition for the $Ni_{30}Pt_{20}Ti_{50}$ (at.%) alloy is a solution treatment and water quench followed by aging 1-4 hrs at 500 °C