NARI

Single-Crystal High-Temperature Shape-Memory Alloys

ARMD Seedling Fund Phase I

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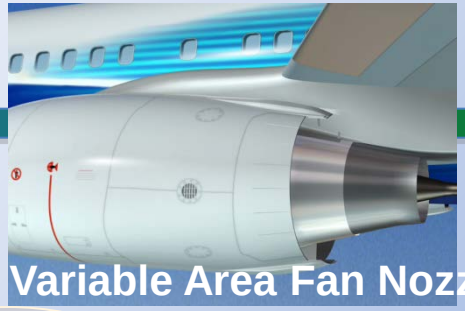
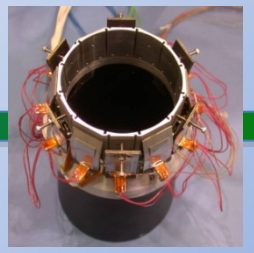
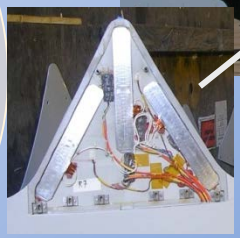


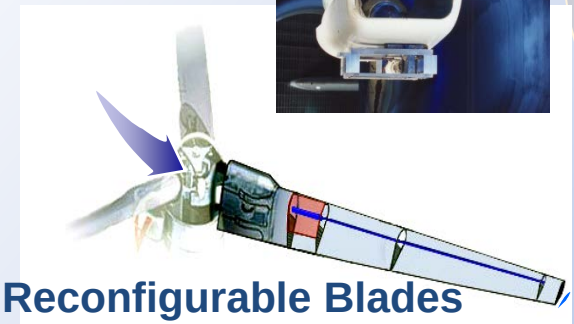


Variable
Geometry
Inlets

Shape Memory Alloys Enable the Development Adaptive Structures

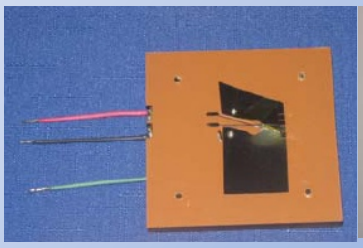


Variable
Geometry
Chevron



Reconfigurable Blades

Flaps, Tabs, Vortex Generators



Variable
Geometry
Aerodynamic Surfaces

Objectives

- Grow high-temperature, high purity, shape-memory alloy (SMA) single-crystals using the Czochralski method
- Analyze composition, microstructure, and orientation using electron microscopy and X-ray
- Perform thermo-mechanical testing on samples and compare to polycrystalline and Bridgeman-grown single-crystal samples

Shape Memory Behavior

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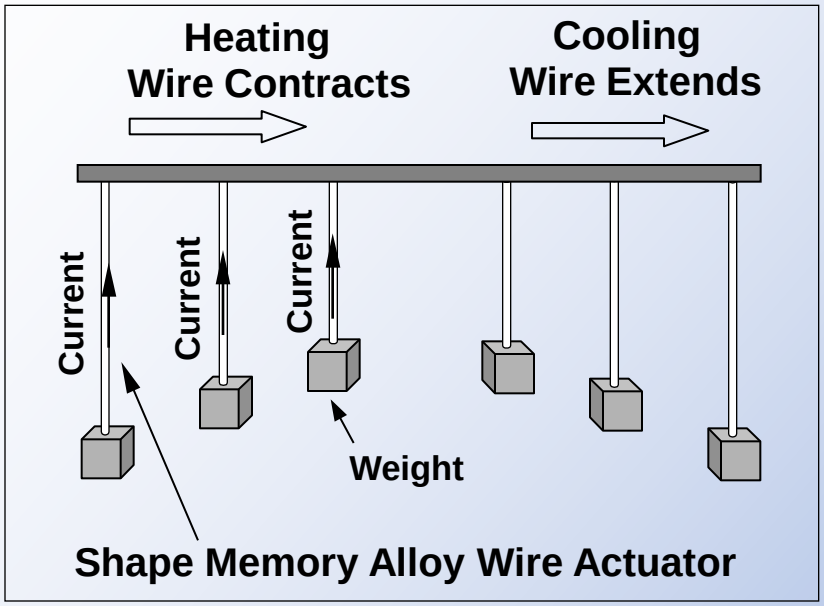

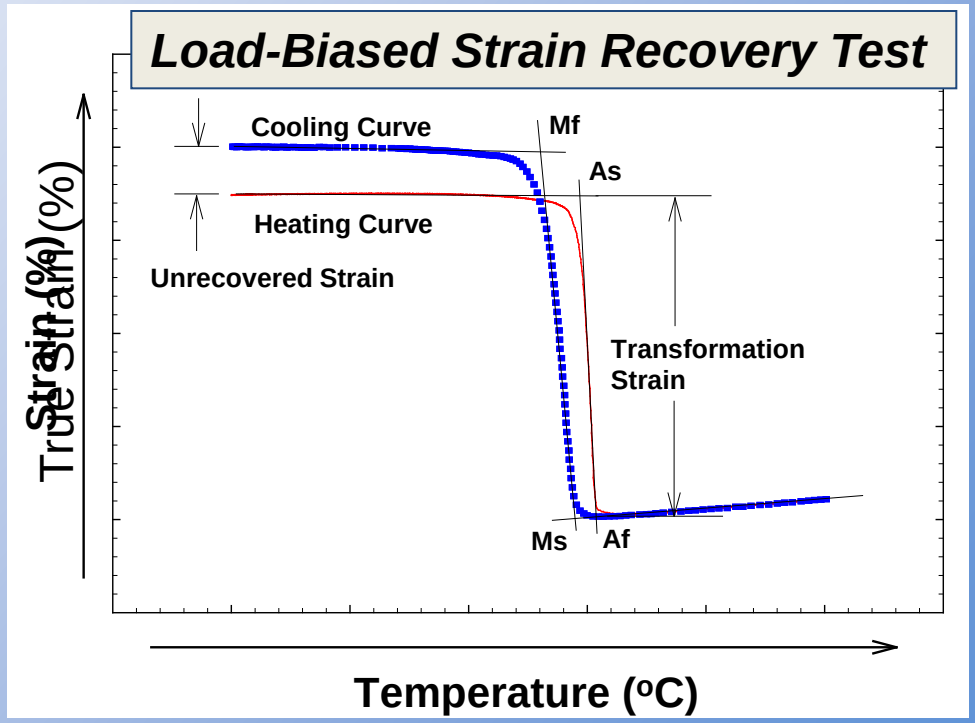

Measure:

a) Transformation Temps M_f, M_s, A_s, A_f

b) Transformation Strain -> Work Output

c) Unrecovered Strain -> Dimensional Stability

$$W = \int \sigma d\varepsilon$$

Advantages of SMA-Based Actuation Systems

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- High force (per volume/weight)
- Compact
- Easily integrated on existing systems
- Eliminates extraneous systems
  - hydraulics, pneumatics, mechanical
- Robust
- Simple, frictionless, quiet
- Low Maintenance

Motor:
Torque 66 in-lbs
25 lbs

Gear box :
190 in-lbs
16 lbs

SMA Rotary Actuator :
150 in-lbs
1 lbs

BOEING

IDEAL FOR HIGH FORCE, LARGE STROKE, LOW CYCLE

Opportunities for SMA Actuators on Jet Aircraft

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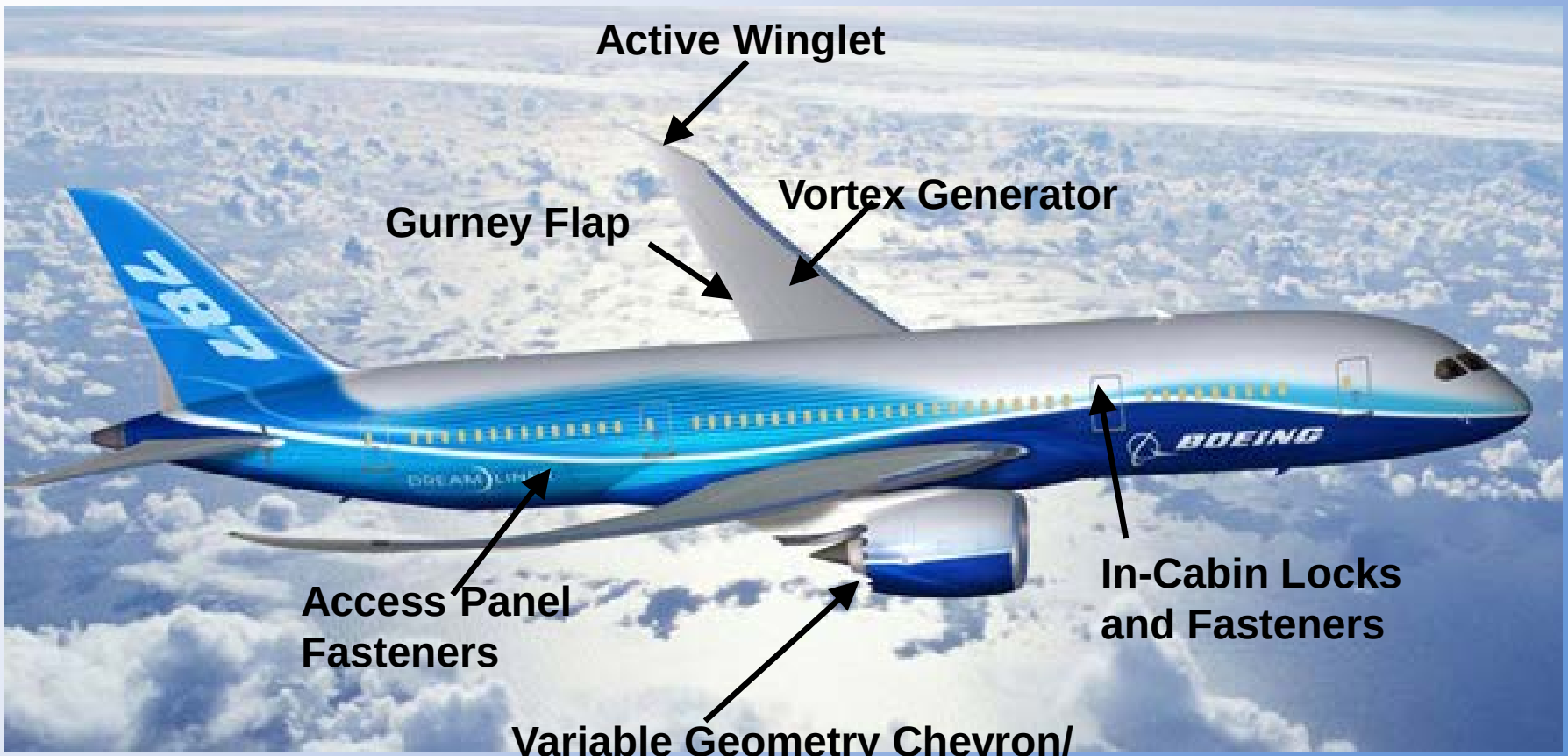
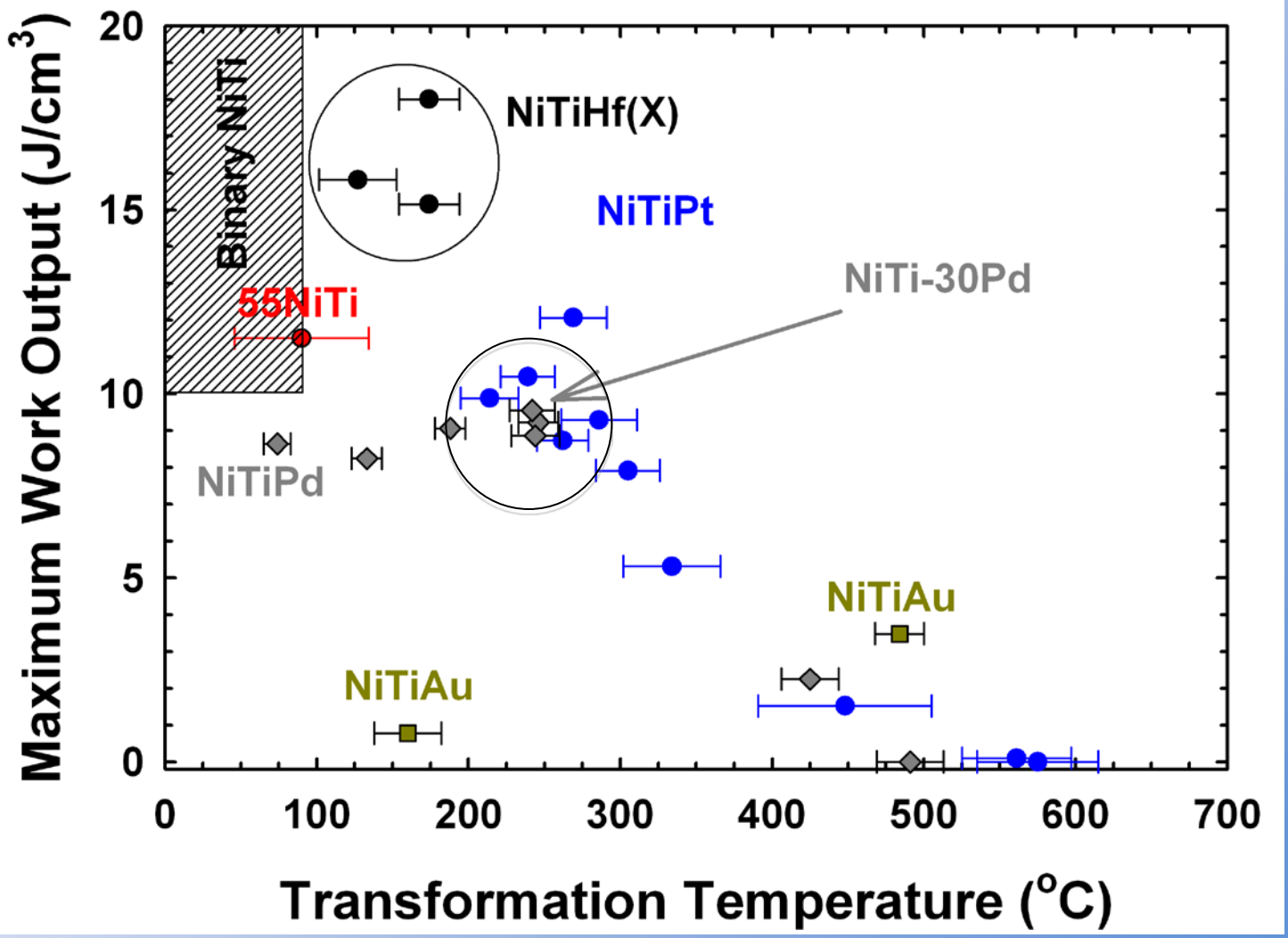


Maximum Work Output of Polycrystalline SMAs



The Innovation

- HTSMAs – high work output in a small volume
- Single-crystal HTSMA's – high stress capability & tailorable properties
  - High transformation strain - high work at low(er) stress
  - Extreme stress capability – high work output while maximizing compactness

Background

- Russian Ni-Ti-Hf SX HTSMA previously tested
  - Produced by Bridgeman method in graphite crucible
  - Large carbides due to reaction with carbon in crucible
  - One-off lab experiment
  - Future collaboration not feasible
- GRC tri-arc Czochralski grower
  - Three arcs aid temperature uniformity
  - Copper cold-hearth – eliminates additional carbon contamination

Single Crystal Growth Methods

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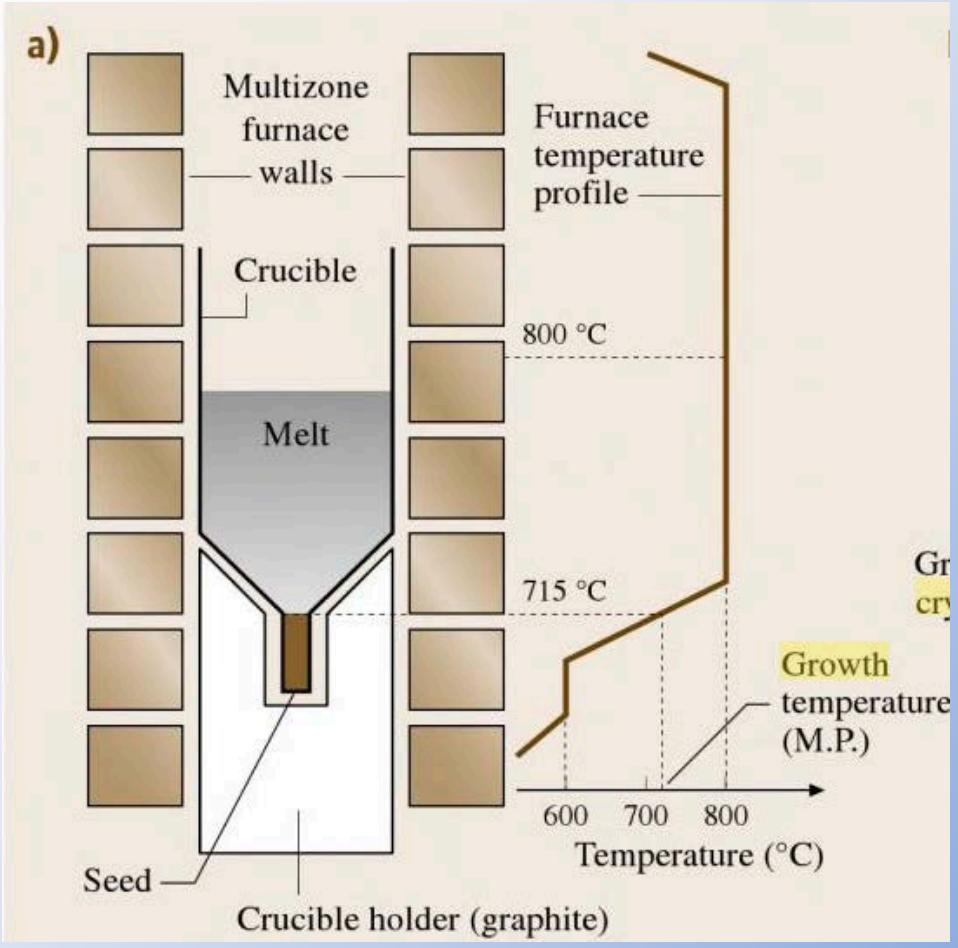
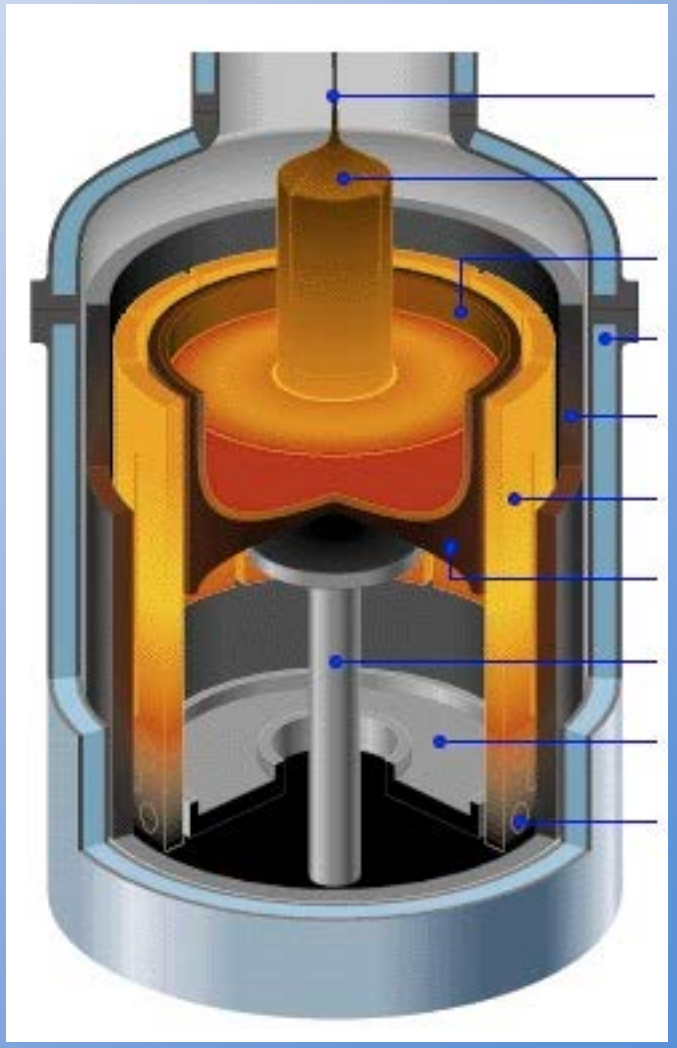
- Bridgeman Method



Czochralski Method



*Springer Handbook of Crystal Growth, (2010) Springer-Verlag, Berlin Heidelberg, p288

Tri-Arc Czochralski Method

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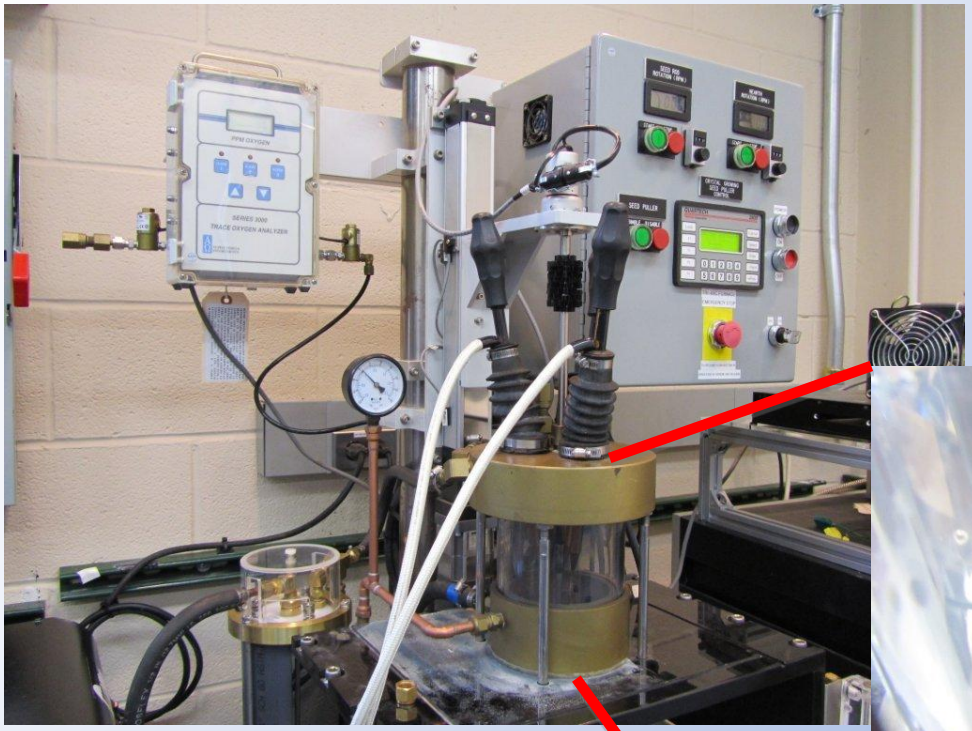

Czochralski Growth Chamber

SMA Ingot With Pull Rod and Arc Melting Stingers

Single-Crystal Growth Movie

Results

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- Crystals of Ni-Ti-Hf, Ni-Ti-Pd, & Ni-Ti-Zr grown
- Nucleated using Al_2O_3 rod, then W
- ~3mm rod used to grow Ni-Ti-Pd
- Switched to larger 6.3mm rods

1st Gen Ni-Ti-Pd Crystals Already Grown

2nd Gen Ni-Ti-Hf Crystals

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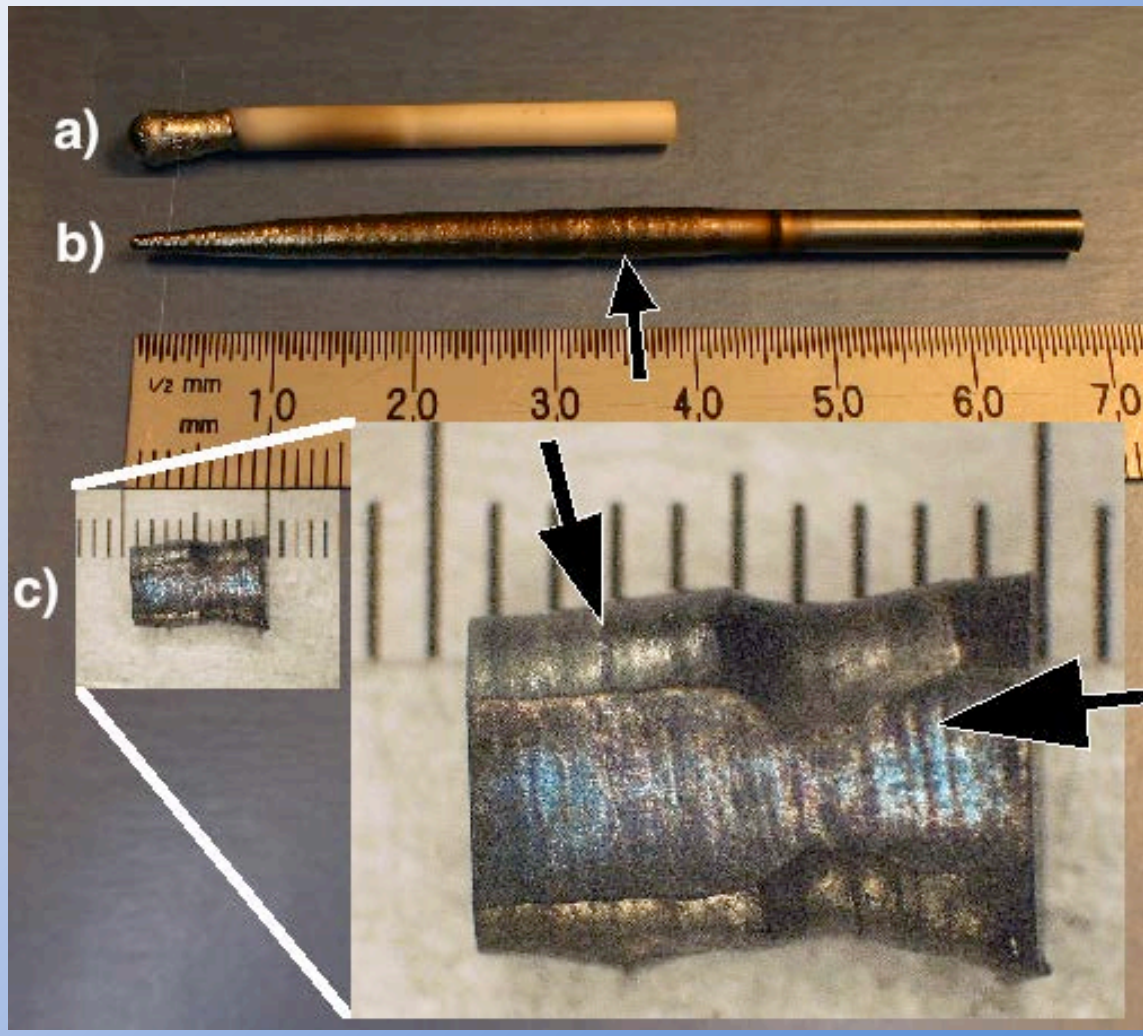
Samples and Growth Banding

- Growth banding
  - Occurs in all CZ crystals
  - Can be due to temperature fluctuations
    - (not a problem)
  - Or chemical inhomogeneity
    - (problem?)



Banding Effects in Ni-Ti-Hf

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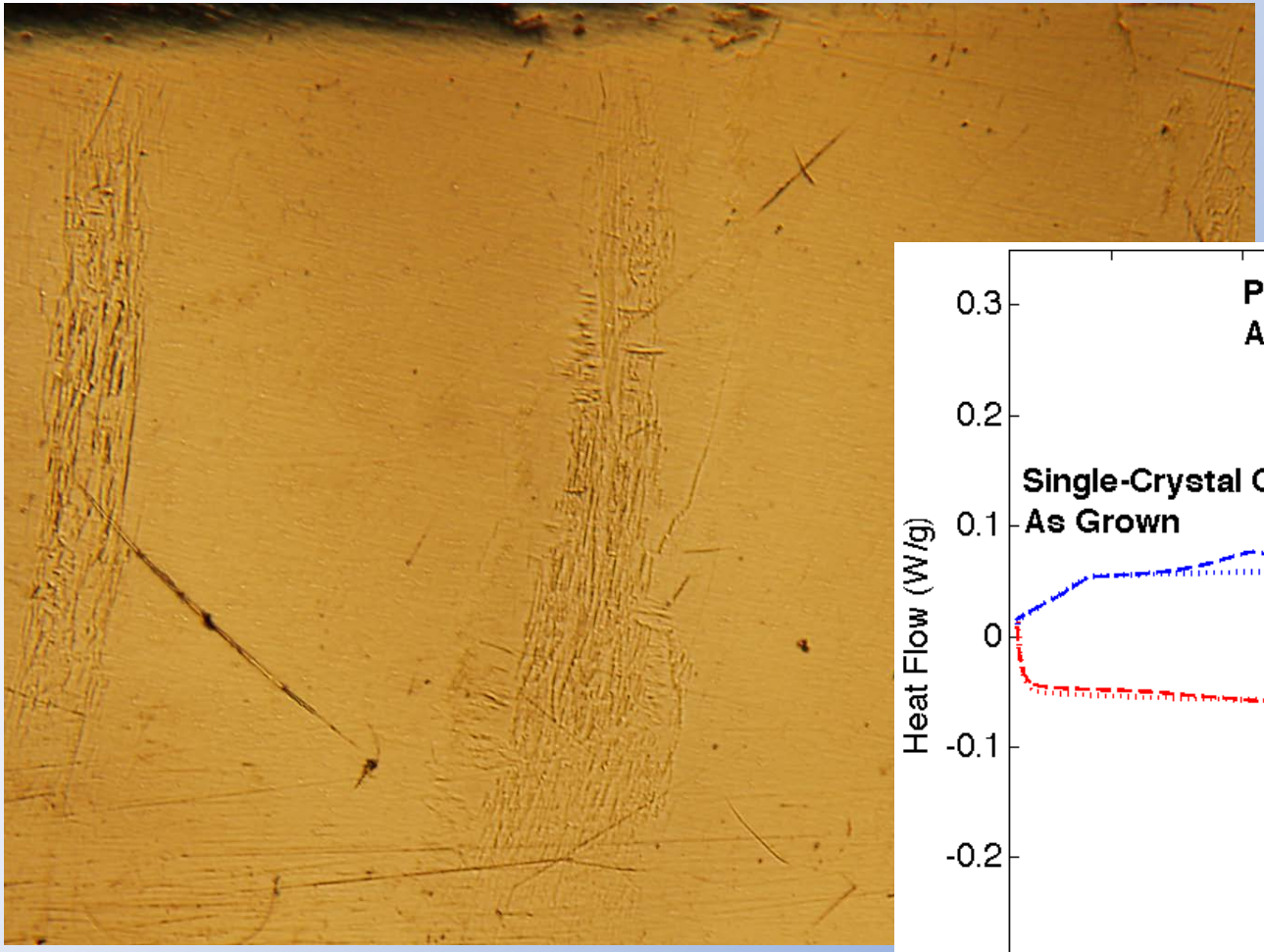
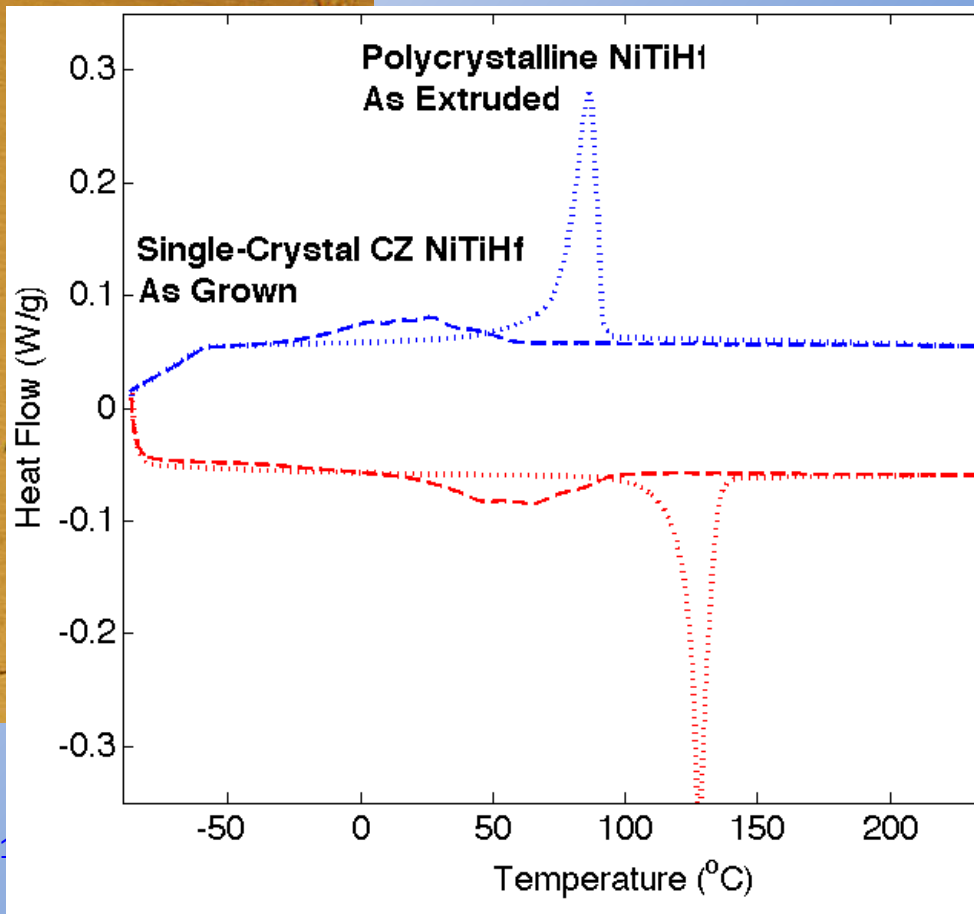
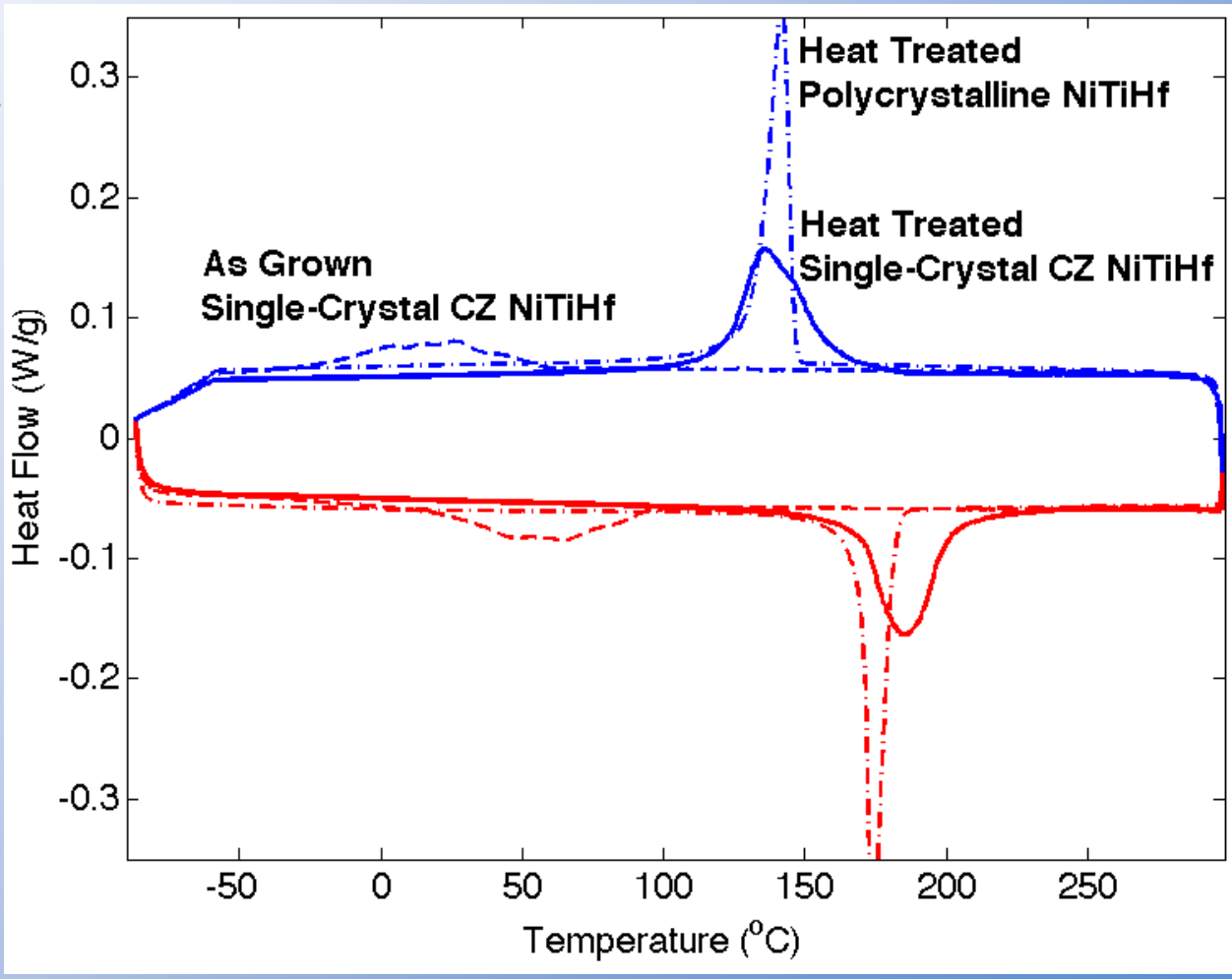




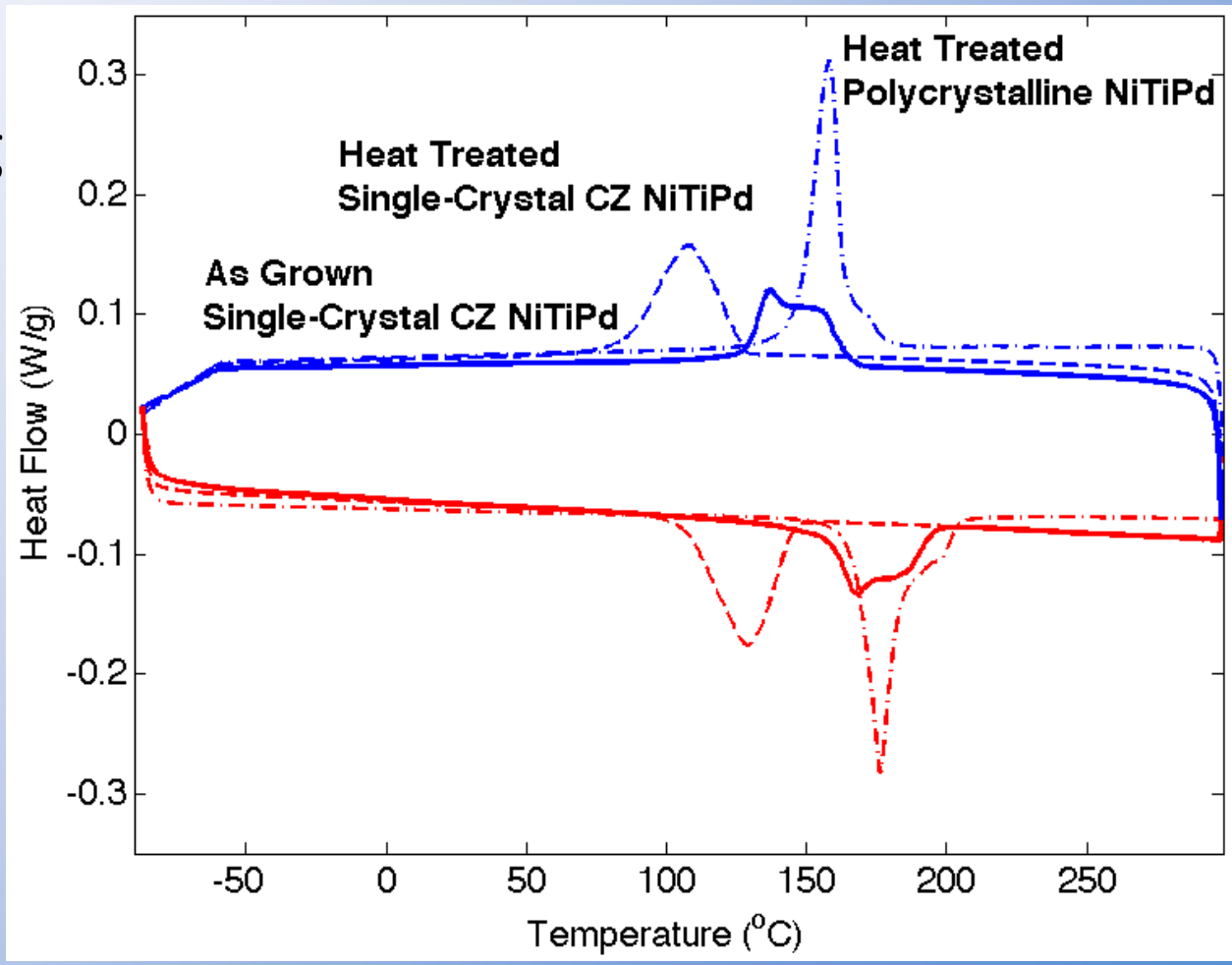
Ni-29.7Ti-20Hf

- As Grown
  - Broad – banding
  - Low Tf – Ni
- Heat treated
  - High, narrow Tf
  - Similar in polycrystal and single-crystal



Ni-49.7Ti-30Pd

- As Grown
  - Broad – banding
  - Low Tf – Ni
- Heat treated
  - High, narrow Tf
  - Similar in polycrystal and single-crystal



SEM of Ni-49.7Ti-30Pd

- Banding visible
  - Martensite in bands
- Also particles
  - W
  - TiC

SEM of Ni-29.7Ti-20Hf

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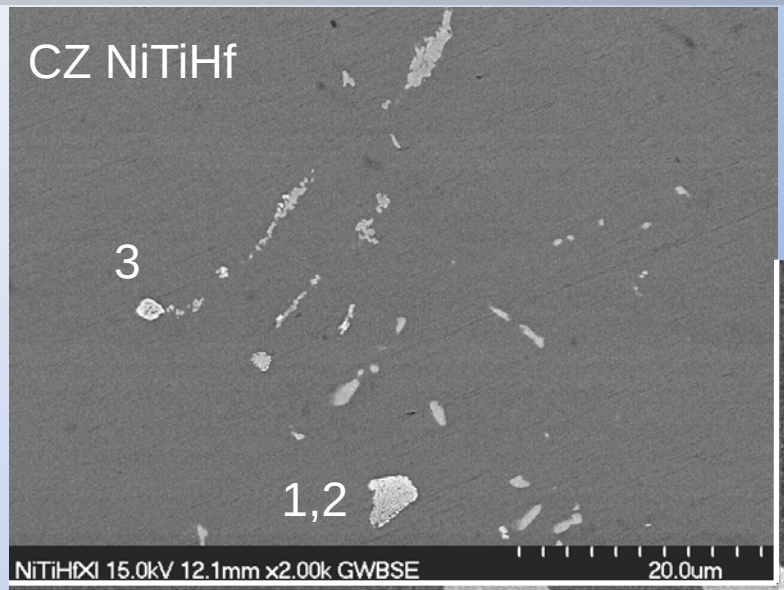
- Particles present
  - 1) TiC 2) HfC
  - 3) W
  - Much smaller in GRC CZ grown SX
  - Russian crystal grown in graphite crucible



Chemical Analysis Comparison Feedstock and Single Crystal (SX)

Table 1: Chemical Analysis of HTSMA Single Crystal Samples and Precursor Hot Top Material (at%).						
Sample	**Ni**	**Ti**	**Hf**	**Zr**	**Pd**	**W**
NiTiHf Feedstock	**50.22**	**29.17**	**20.14**	**0.33**		
SX Hf T	**50.2**	**29**	**20.4**	**0.3**		**0.15**
SX Hf B	**50.2**	**29**	**20.3**	**0.31**		**0.16**
NiTiPd Feedstock	**20.01**	**48.9**			**31.09**	
SX Pd B	**19**	**48.9**		**0.04**	**31.8**	**0.28**

Sample composition measured from top (near pull rod) and bottom (near melt)

Composition of samples is equivalent to that of the original hot top material.

Transformation Strain Ni-29.7Ti-20Hf

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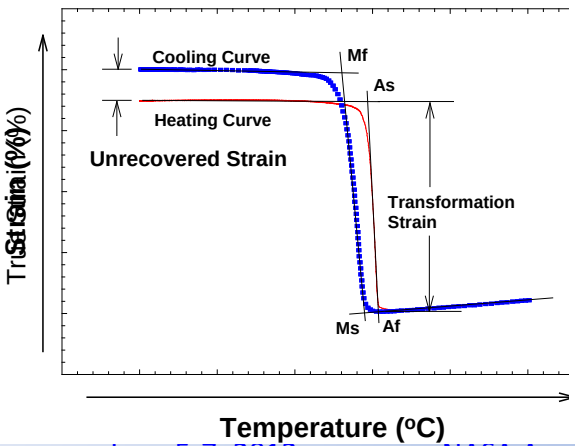
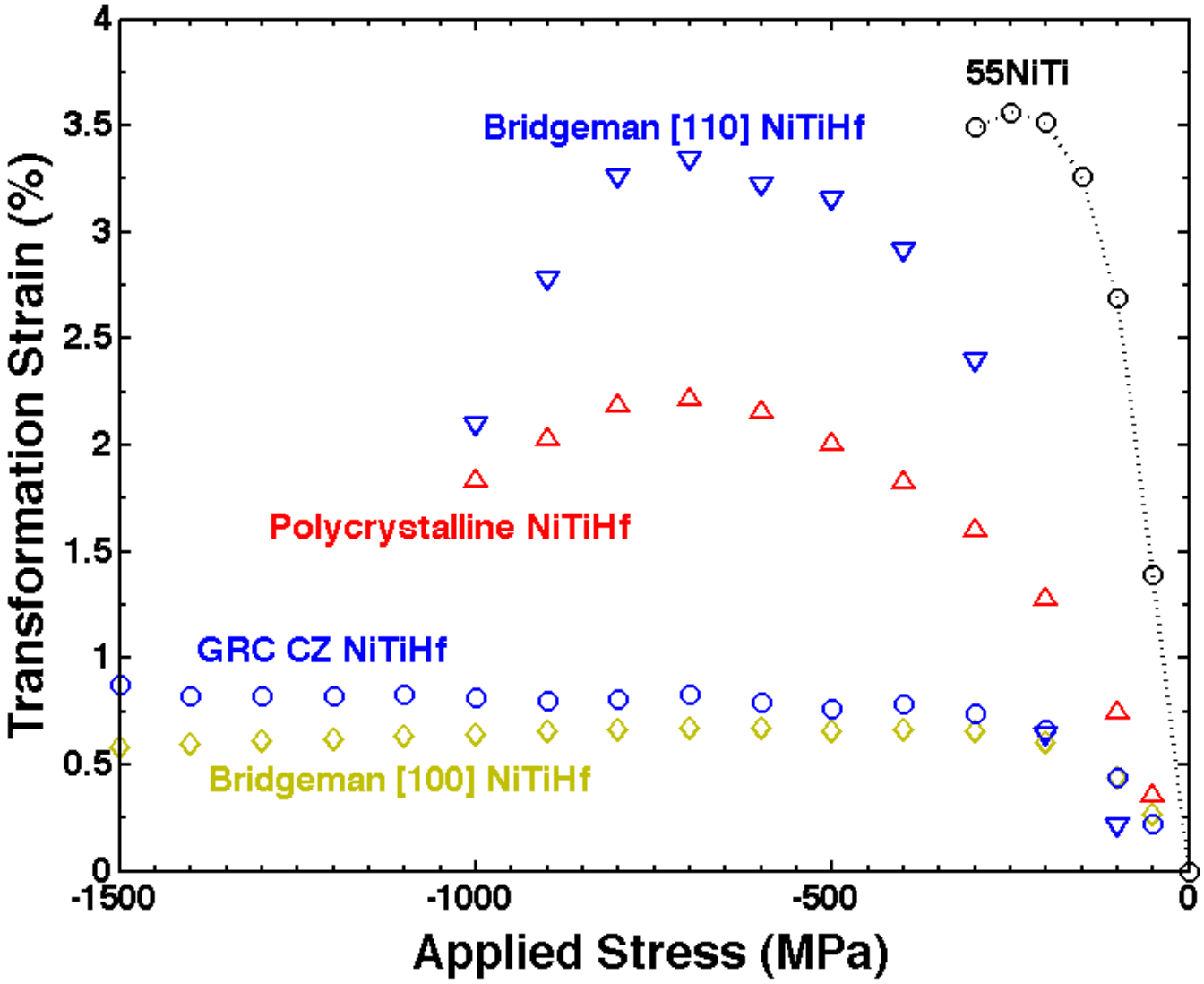
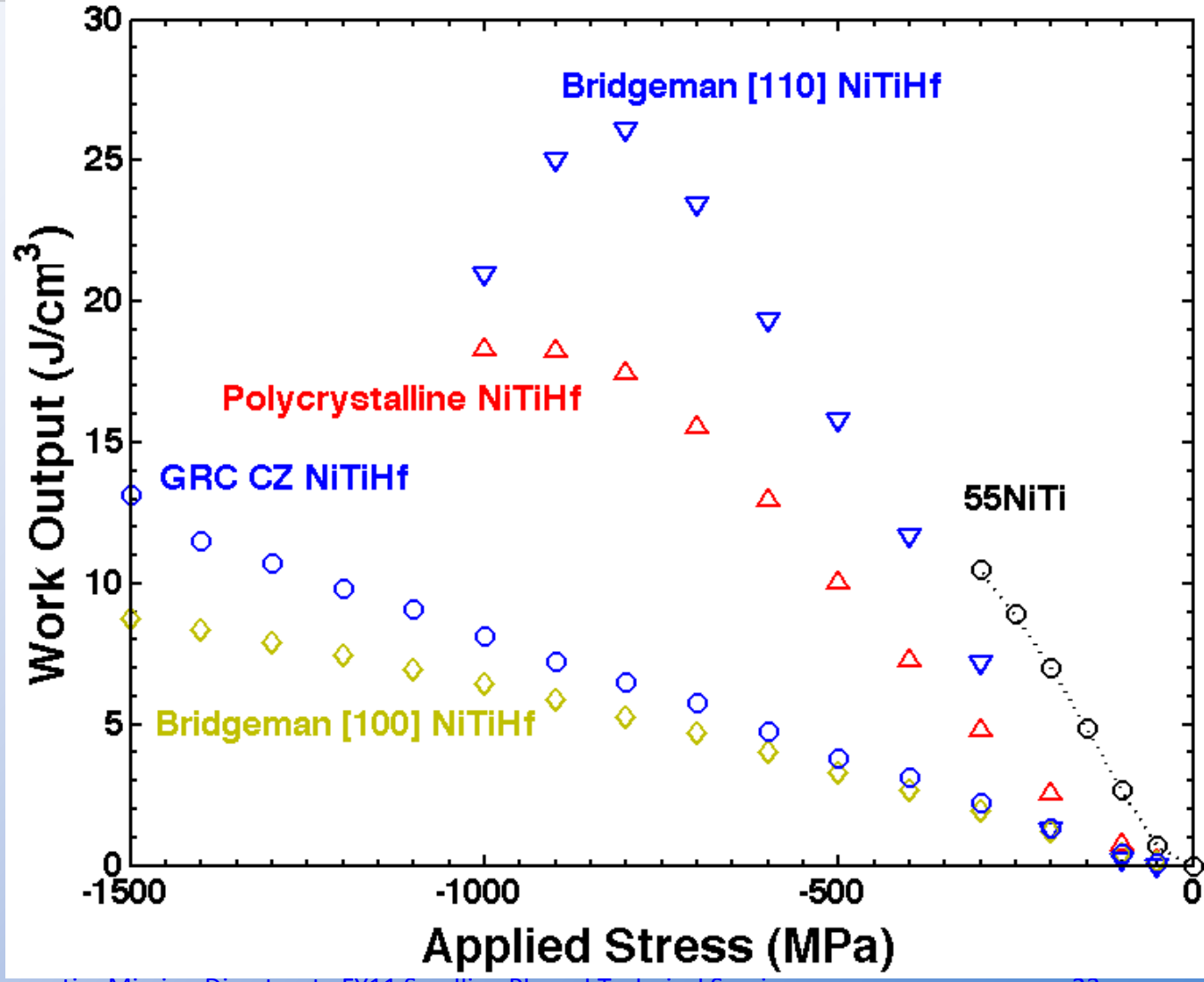
- Moderate TfS
  - Higher than Russian SX
  - Extremely high stress capability





Work Output Ni-29.7Ti-20Hf

- Work is high due to high stress capability



Dimensional Stability Ni-29.7Ti-20Hf

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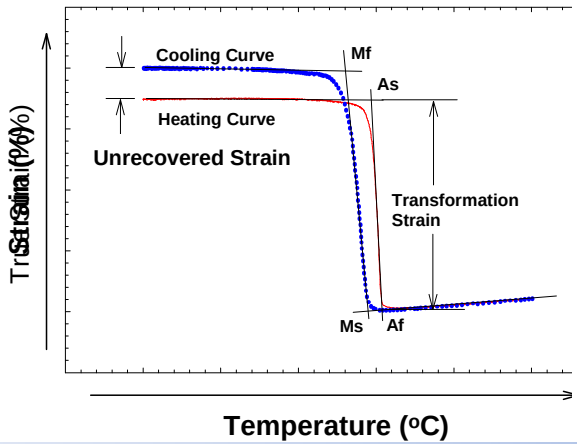
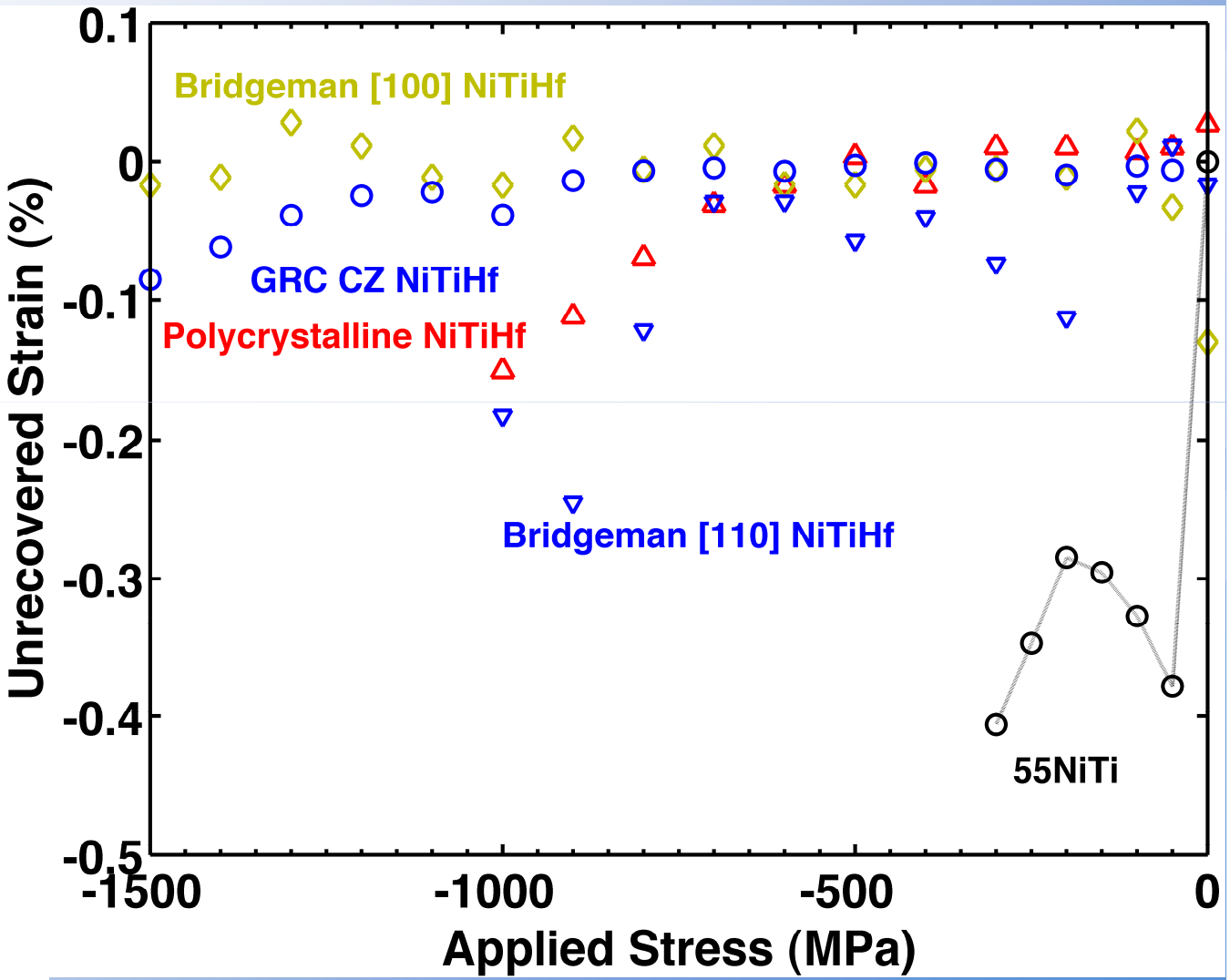
- Stability equivalent to that of Russian crystals





Transformation Strain Ni-49.7Ti-30Pd

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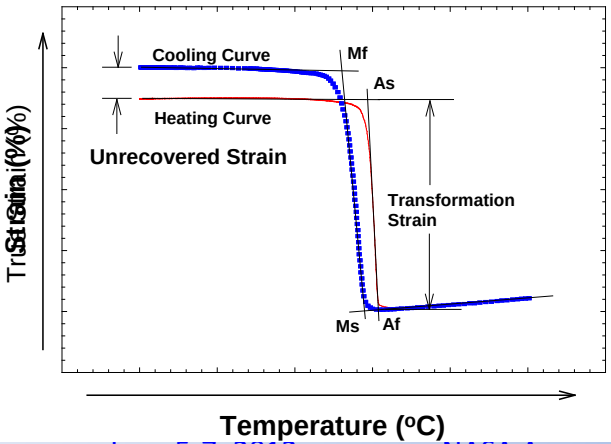
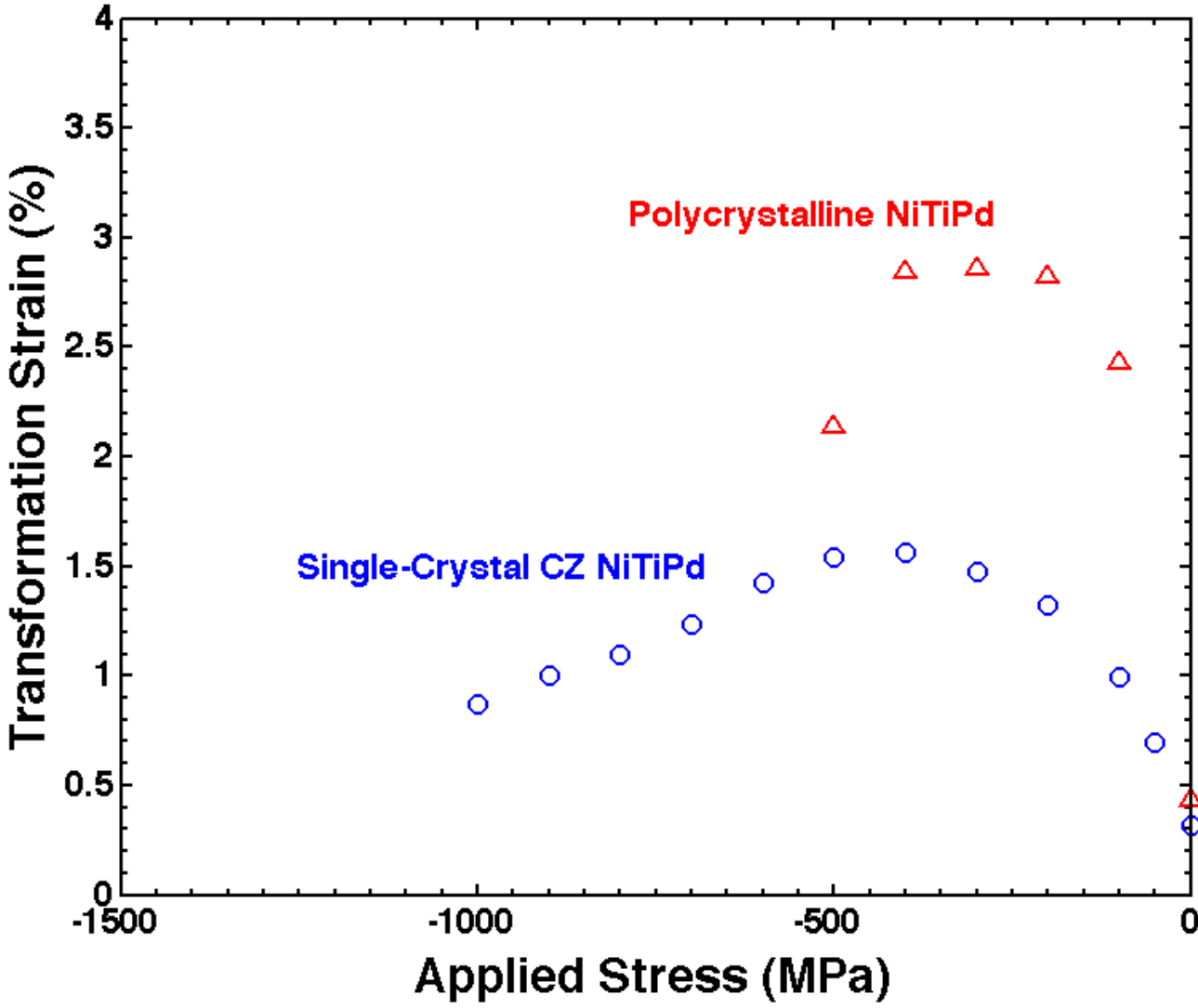
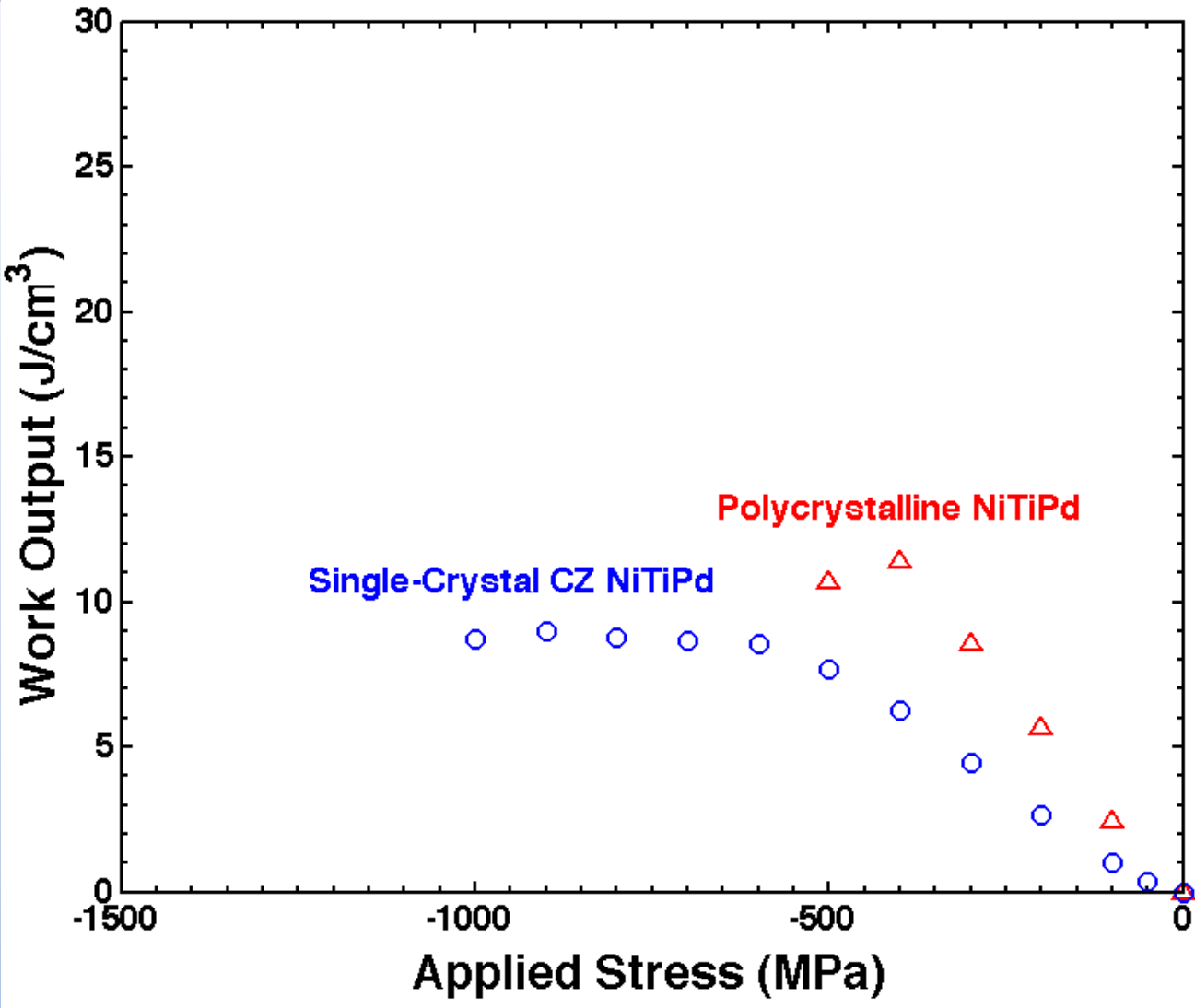
- Moderate TfS
  - Lower than polycrystal
  - High stress capability





Work Output Ni-49.7Ti-30Pd

- Work is high due to high stress capability



Dimensional Stability Ni-49.7Ti-30Pd

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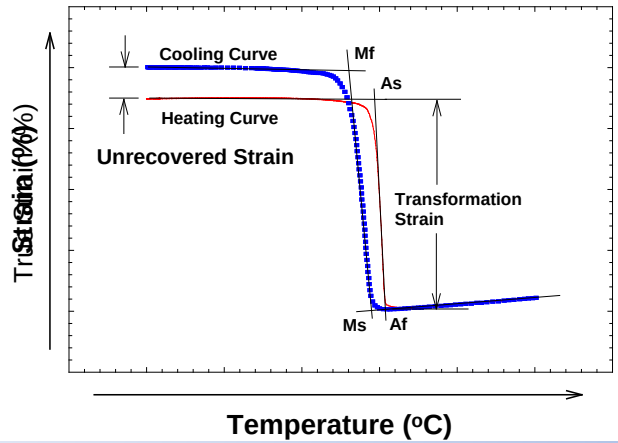
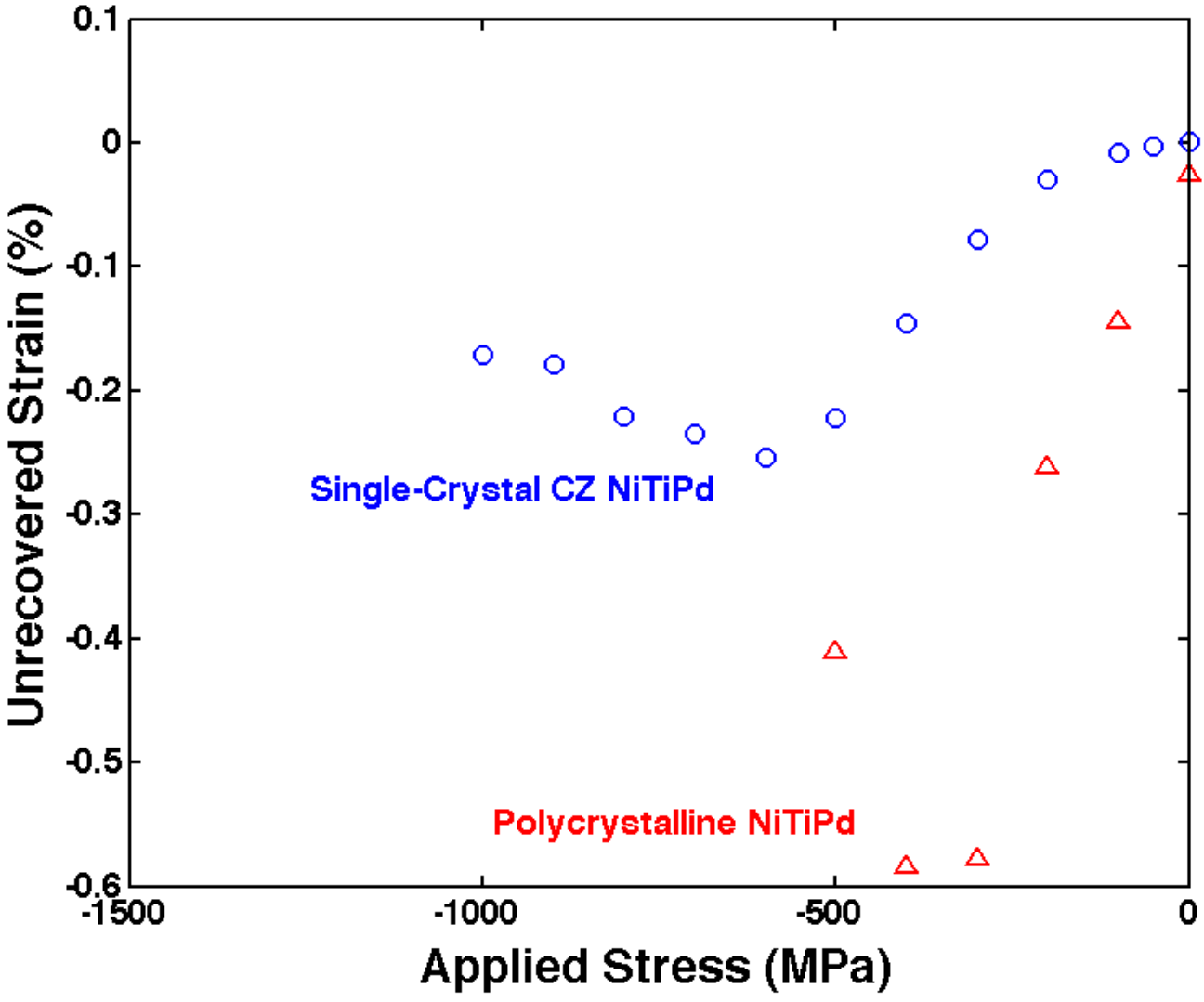
- Stability greater than polycrystal





Remainder of Phase I

- X-ray in progress to confirm orientation and quality of crystals, ie low angle boundaries
- Mechanical testing of remaining crystals
  - Ni-Ti-Zr, and several additional Ni-Ti-Hf

Payoffs/Impact of Innovation

- Tailorable properties
  - High force (stress)
  - High displacement (transformation strain)
  - Hybrid properties
- Properties unobtainable in polycrystalline materials

Summary of Accomplishments

- Successfully demonstrated Czochralski process for fabricating oriented SMA crystals
  - High stress capability
  - High work output
- Cleanest SX grown
  - Lower amount of TiC/HfC
  - Finer sized particles

Plans for Dissemination

- Enough data for one paper/TM after additional tests are completed
- Conference presentation FY13

Next Steps – Phase II

- Determine growth conditions for producing large (>6mm) and small (<2mm) diameter single-crystals
- Produce crystals of two different orientations
  - Seeding
  - Cutting to orientation (requires larger crystals)
- Deliver growth conditions, microstructure, and property data for two sizes and two orientations of single-crystals
- Produce HTSMA SX using carbon-free feedstock

Ultimate Phase II Goal

- Grow crystals that are several inches in diameter!!
- Next step for single-crystal SMAs to be Commercially Feasible