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Single-Crystal High-Temperature Shape-Memory Alloys

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NASA Aeronautics Research Mission Directorate (ARMD)

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Innovations

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- Shape-Memory Alloys represent solid state actuation
  - High work output in a small volume
- High-Temperature Shape-Memory Alloys (HTSMAs)
  - Extend the range of transformation temperatures > 100C
  - Above the limit of commercial alloys: < 90C
- Single-crystals allow measurement of orientation specific mechanical behavior of materials
  - Different orientations have different behaviors
  - High transformation strain -> high work at low(er) stress
  - Extreme stress capability -> high work output while maximizing compactness

Shape Memory Behavior - Actuation

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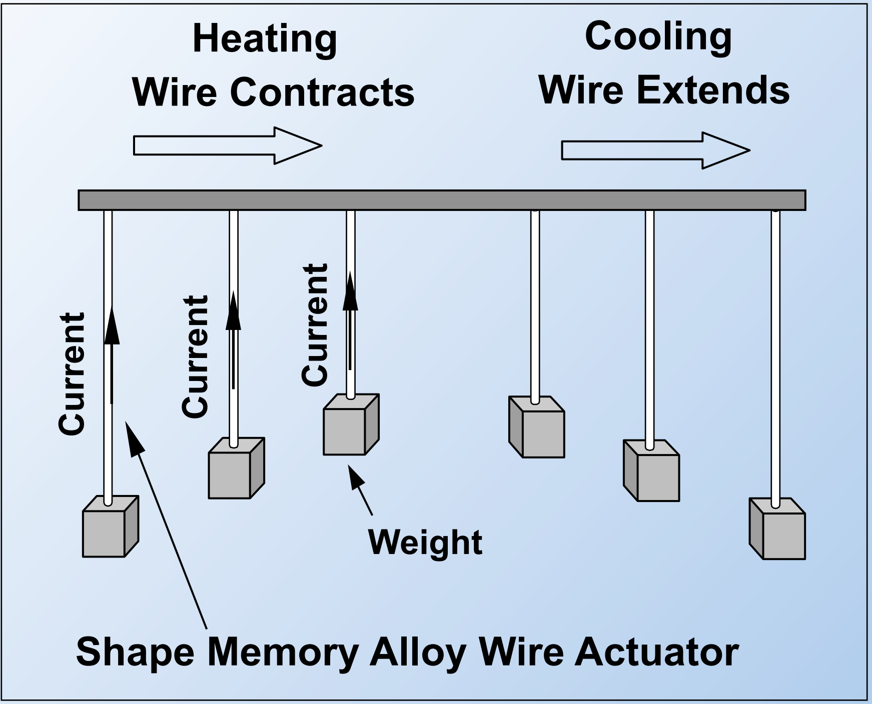

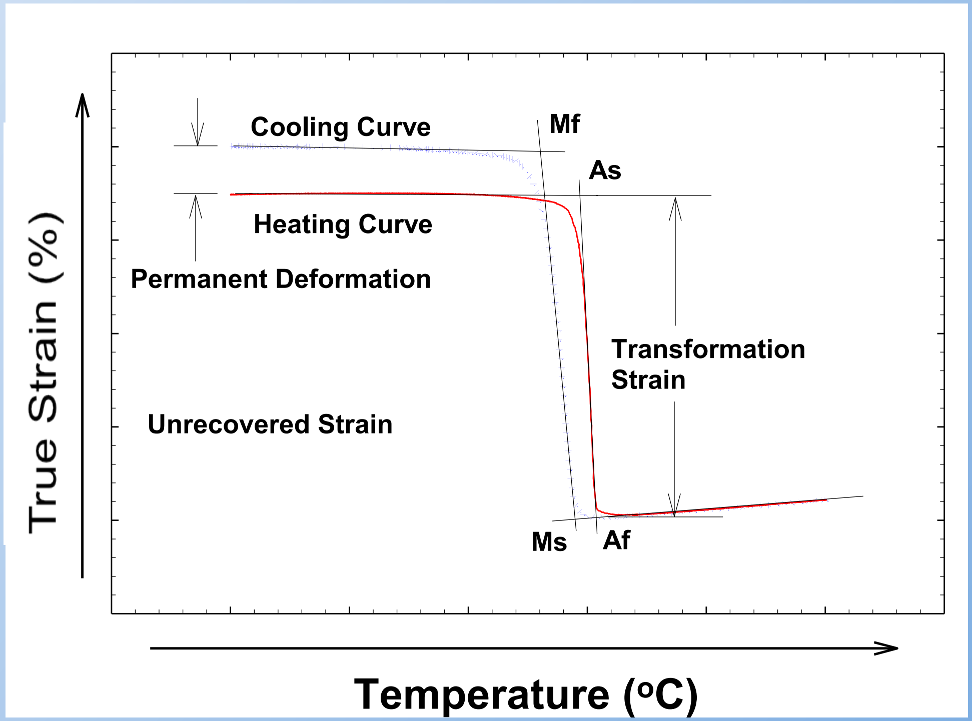

Load biased shape-memory behavior

Measure strain vs temperature as a function of load:

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

- 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

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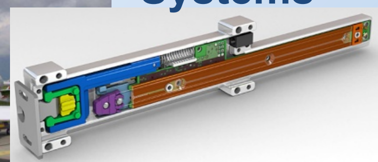
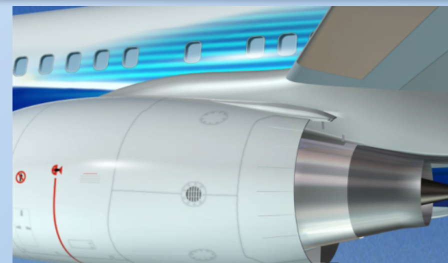
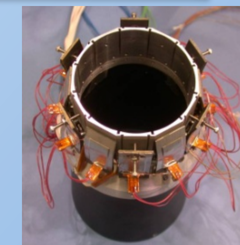
Smart Fastening Systems



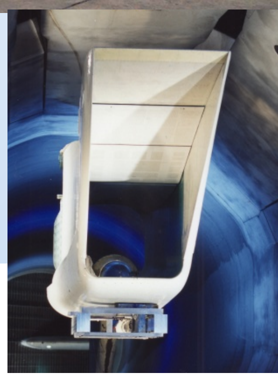




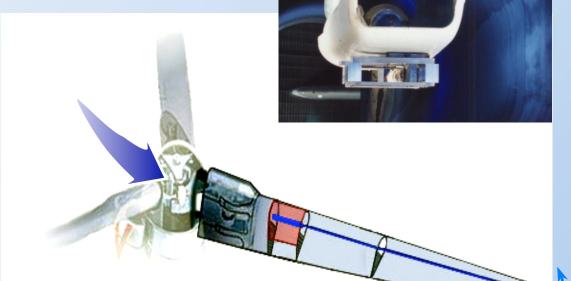
Variable Area Fan Nozzle

High Temperature Shape Memory Alloys Enable the Development Adaptive Structures

Variable Geometry Inlets

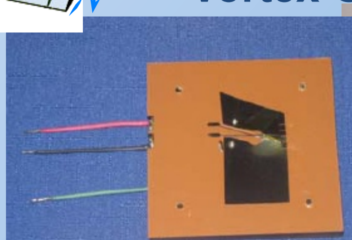


Variable Geometry Chevron



Reconfigurable Blades

Vortex Generators, Flaps, and Tabs



Variable Geometry Aerodynamic Surfaces

GRC High-Temperature Shape-Memory Alloy Development

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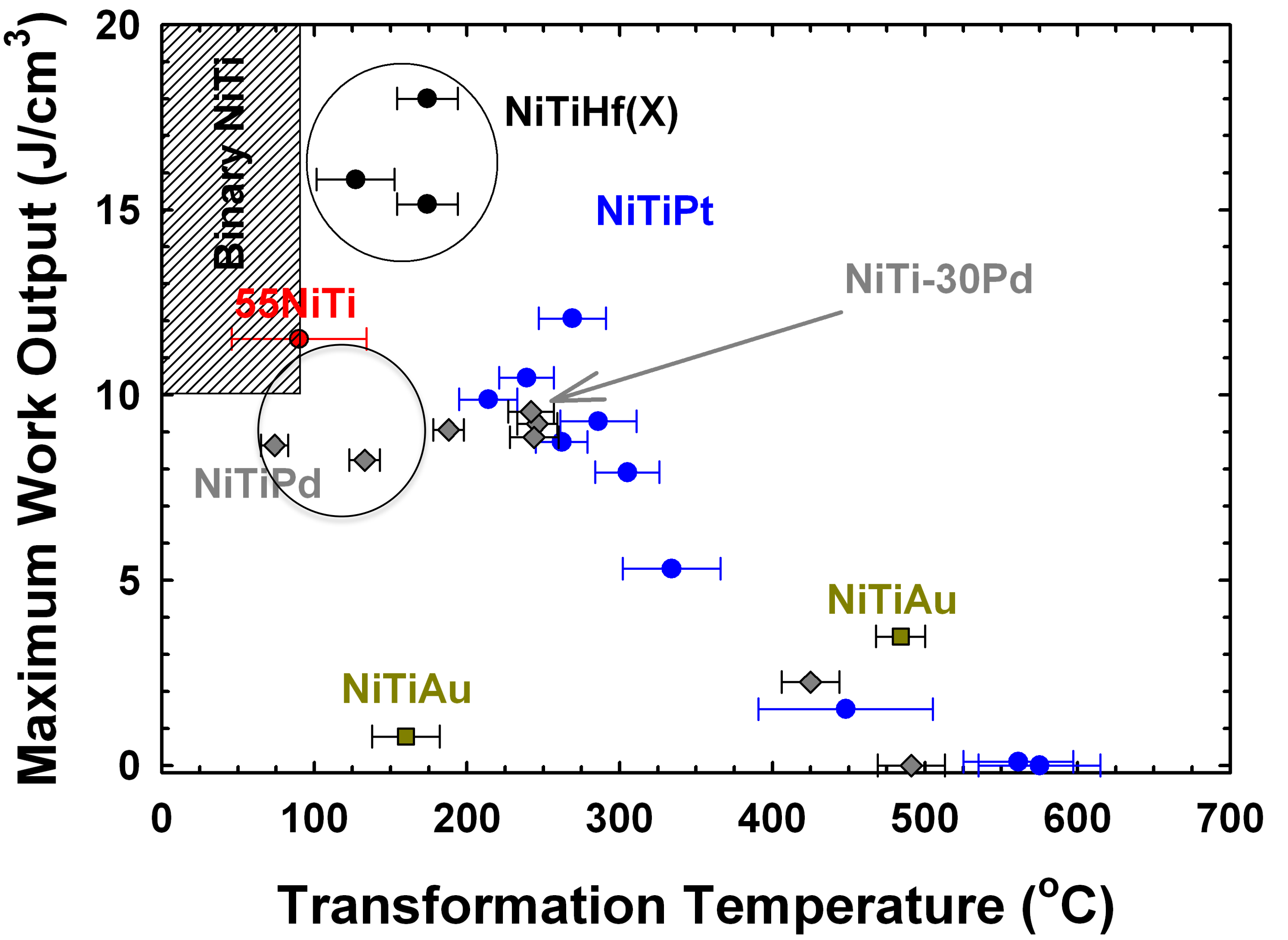
- Binary NiTi and 55NiTi
  - Limit of commerical alloy capability
- NiTiX (X= Au,Hf,Pd,Pt)
  - GRC alloys



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

Single Crystals

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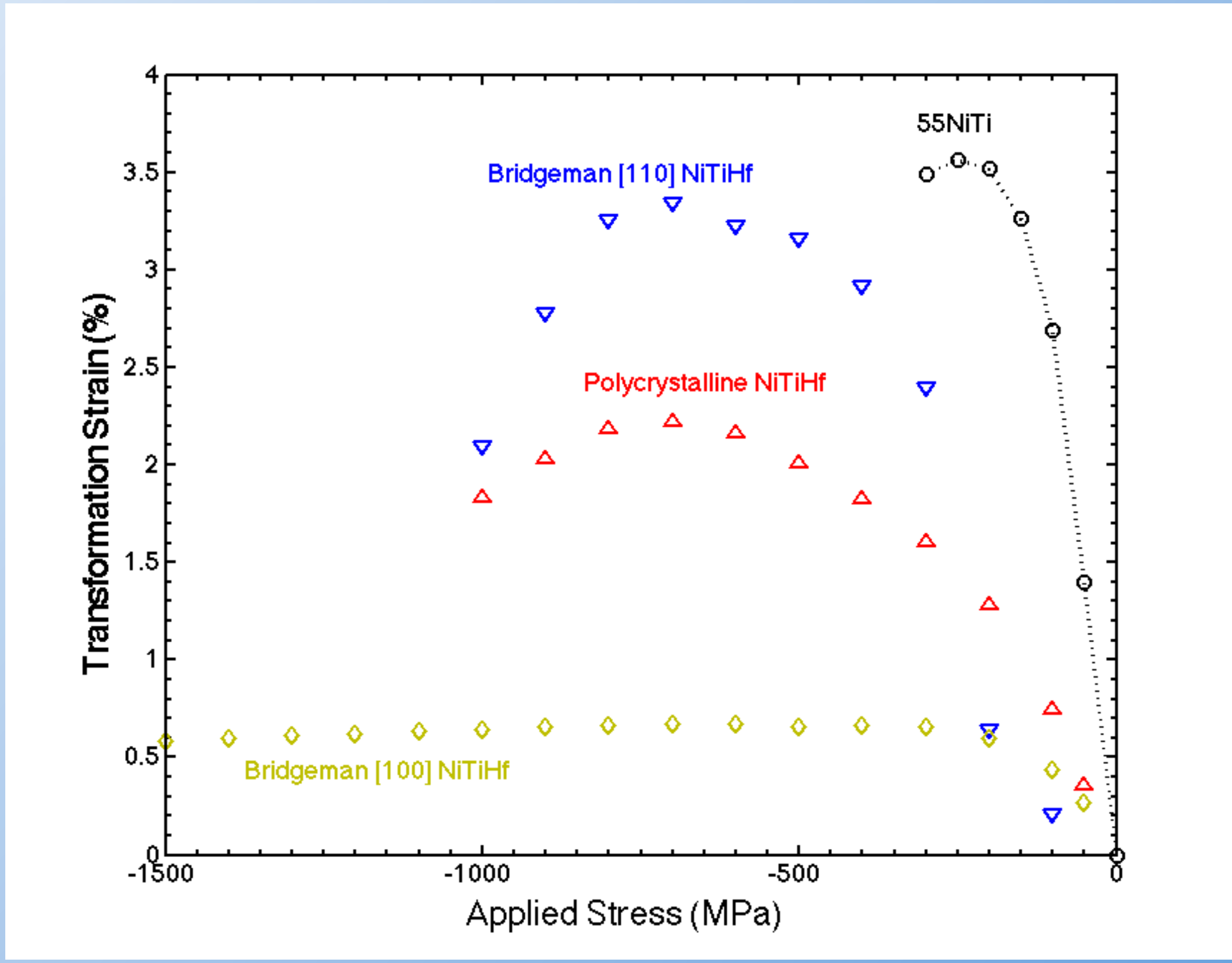
- Give us ability to tailor material properties
  - Larger stroke
  - High strength



Goals

- Develop infrastructure to grow single-crystals in-house at GRC
- Demonstrate ability to grow single-crystal high-temperature shape-memory alloys (NiTiHf)
  - Ability to control crystal size
- Demonstrate ability to tailor mechanical properties via orientation control

Technical Approach

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- Modify GRC's Czochralski (CZ) furnace to grow single-crystal or highly-oriented directionally solidified crystals
  - Czochralski process allows for diameter control
  - Tri-arc unit removes the need for graphite crucible as is required for Bridgeman growth
- Produce large diameter samples using CZ
- Model transformation strain capability of different orientations

Technical Approach

- Microstructural analysis
  - Are multiple grains present
  - How did they grow – link to process parameters if possible
- X-ray diffraction to determine orientation of grains
- Mechanical testing to determine
  - Load-biased shape-memory behavior – transformation strain, temperature, unrecovered strain, work output

Single Crystal Growth Methods

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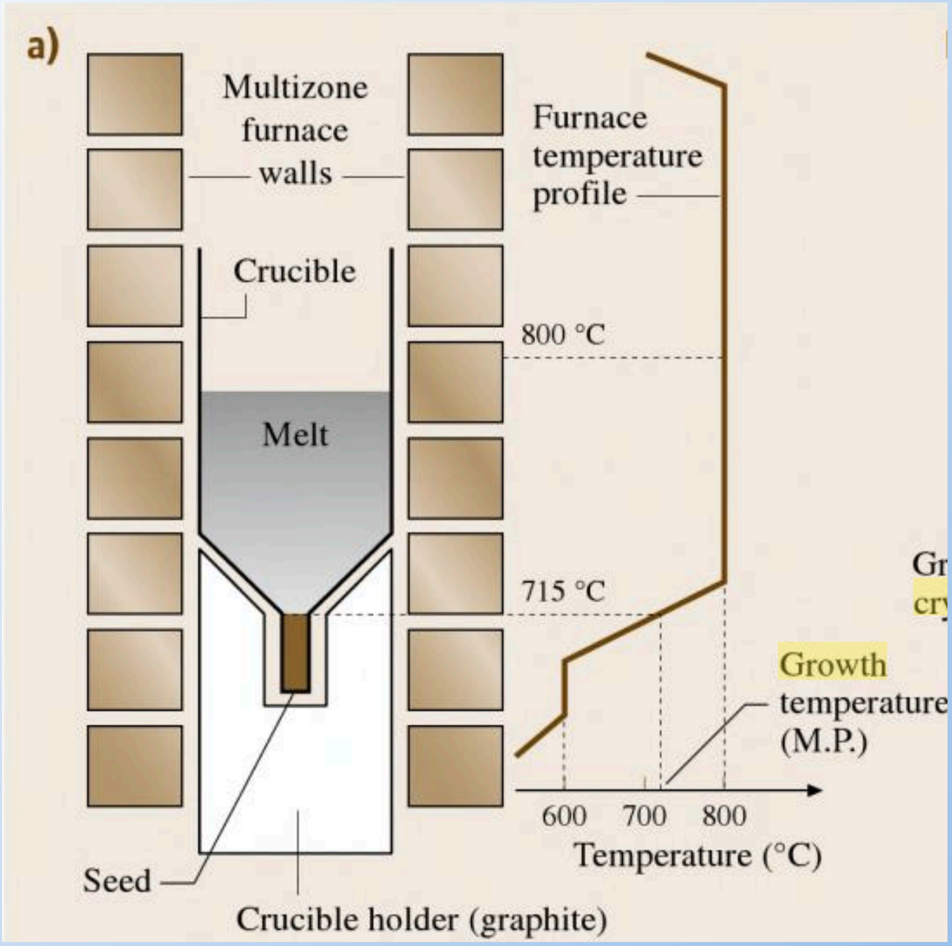
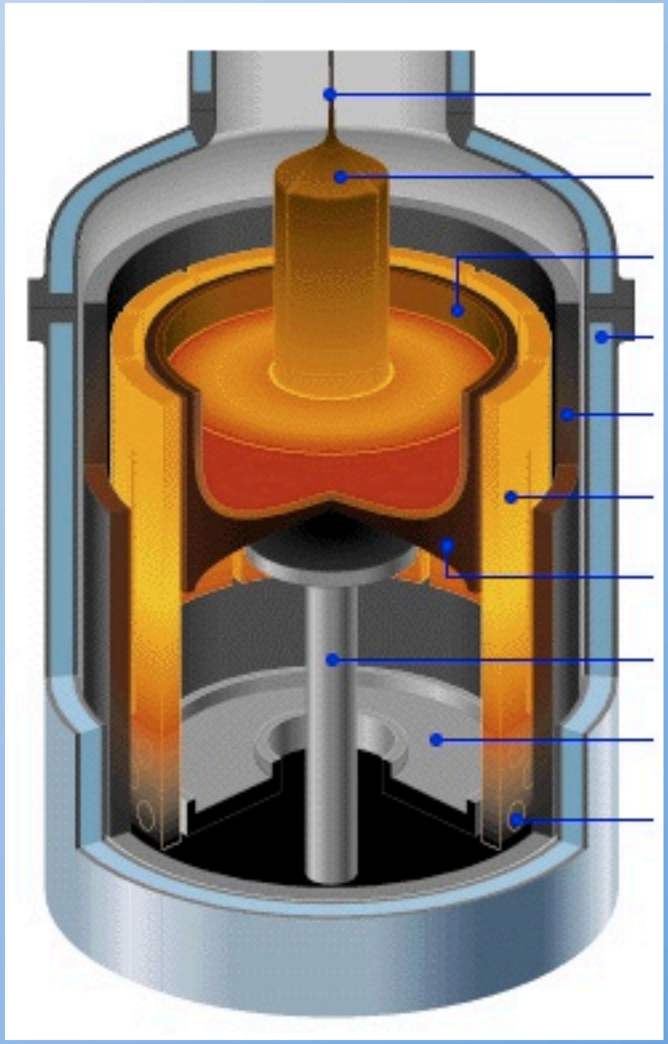
- Bridgeman Method



Czochralski Method



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

GRC's Tri-Arc Czochralski Furnace

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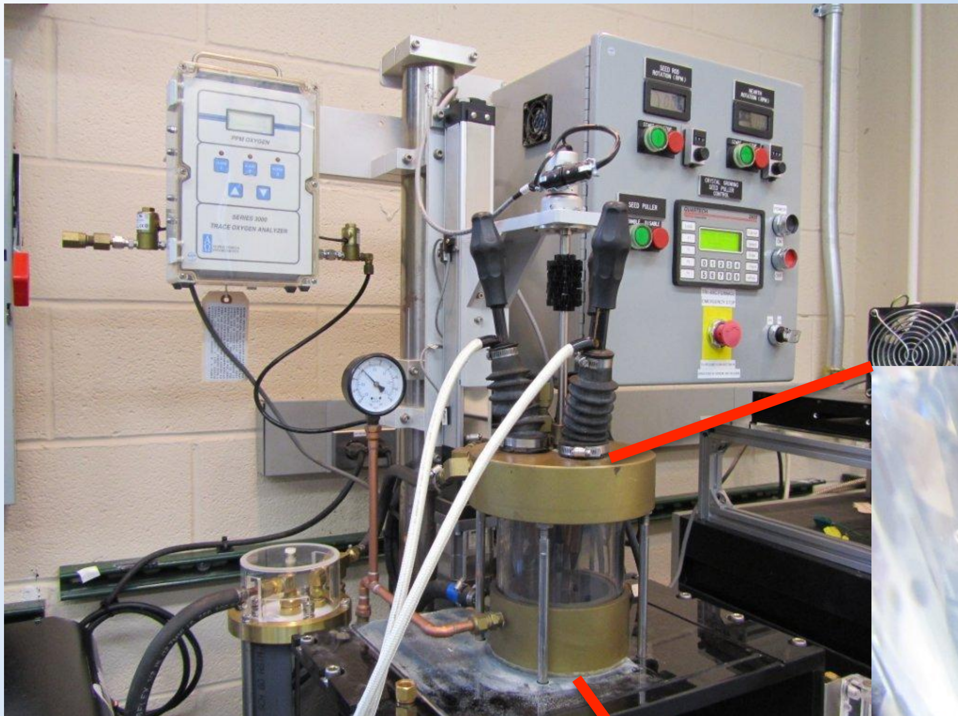

Czochralski Growth
Chamber

SMA Ingot With Pull Rod and
Arc Melting Stingers

Mechanisms Investigated for Initiating Crystal Growth

- Solidification of melt onto pull rod occurs by nucleation and growth:
  - W pull rods – Used because of high melting point, good thermal conductivity
    - End shapes – flat, pointed, and cavity
- Or by templating onto pre-existing grains of the same composition
  - NiTiHf polycrystalline pull rods
    - Shapes - flat, pointed, pointed offset
  - NiTiHf single crystal seed
    - Preferred method

Tungsten Pull Rod - Flat

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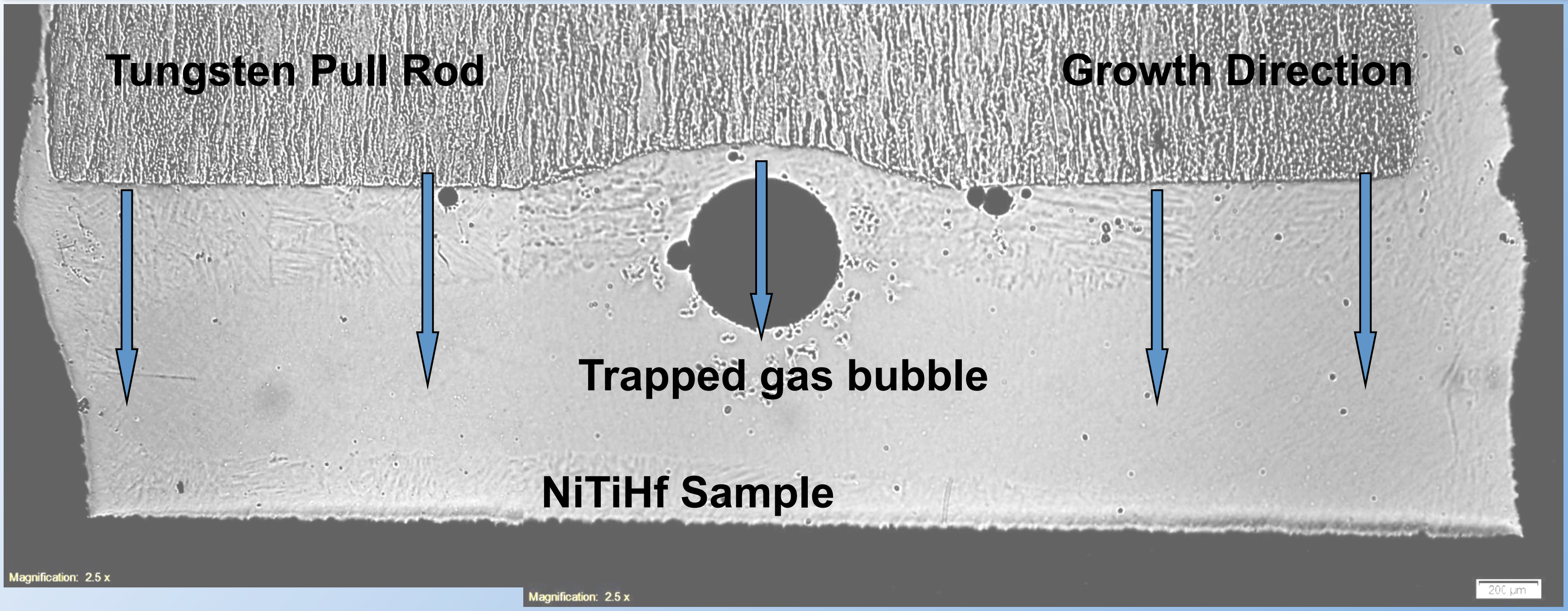

- Grains nucleate on end of pull rod
  - Preferentially oriented grains grow along growth axis
  - Pull rod shape does not aid in grain elimination

Flat Tungsten Pull Rod

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Growth Direction

- NiTiHf liquid wicks up on W pull rod

W pull rod

Pull rod/melt interface

NiTiHf sample

Tungsten Pull Rod - Pointed

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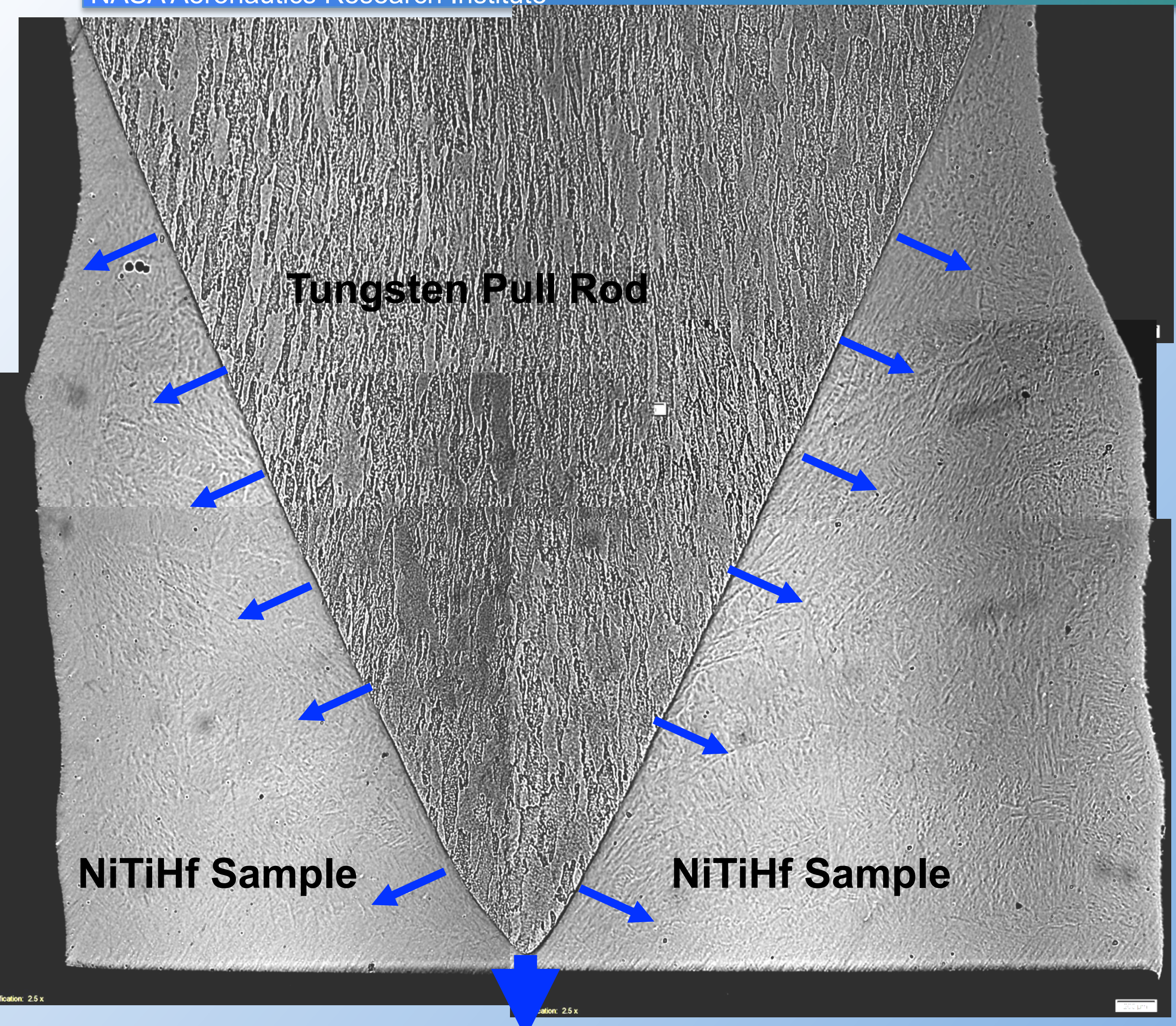

Grains nucleate on sides and tip

- Growth along maximum thermal gradient
- Grain elimination by divergence

Tungsten Pull Rod with Point

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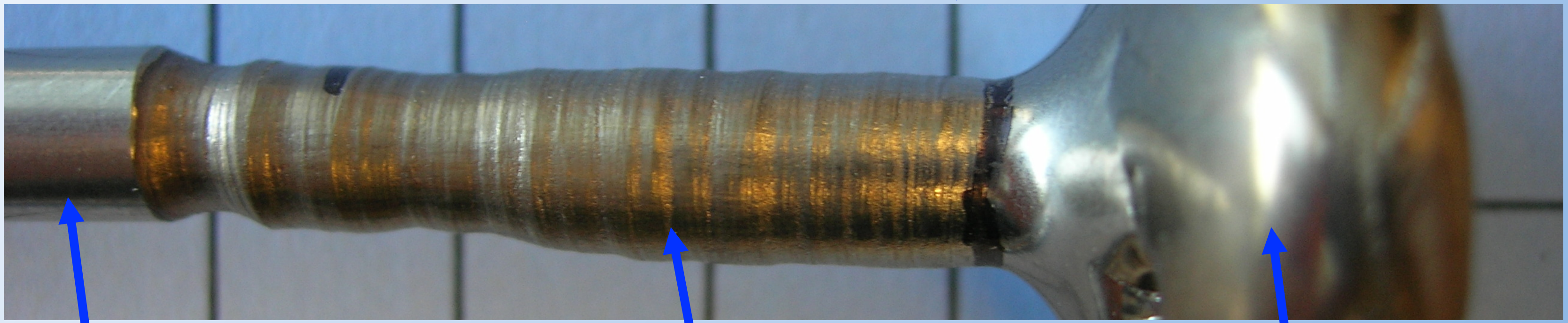
Growth Direction



NiTiHf sample

W pull rod with point

Solidified melt pool

Tungsten Pull Rod - Cavity

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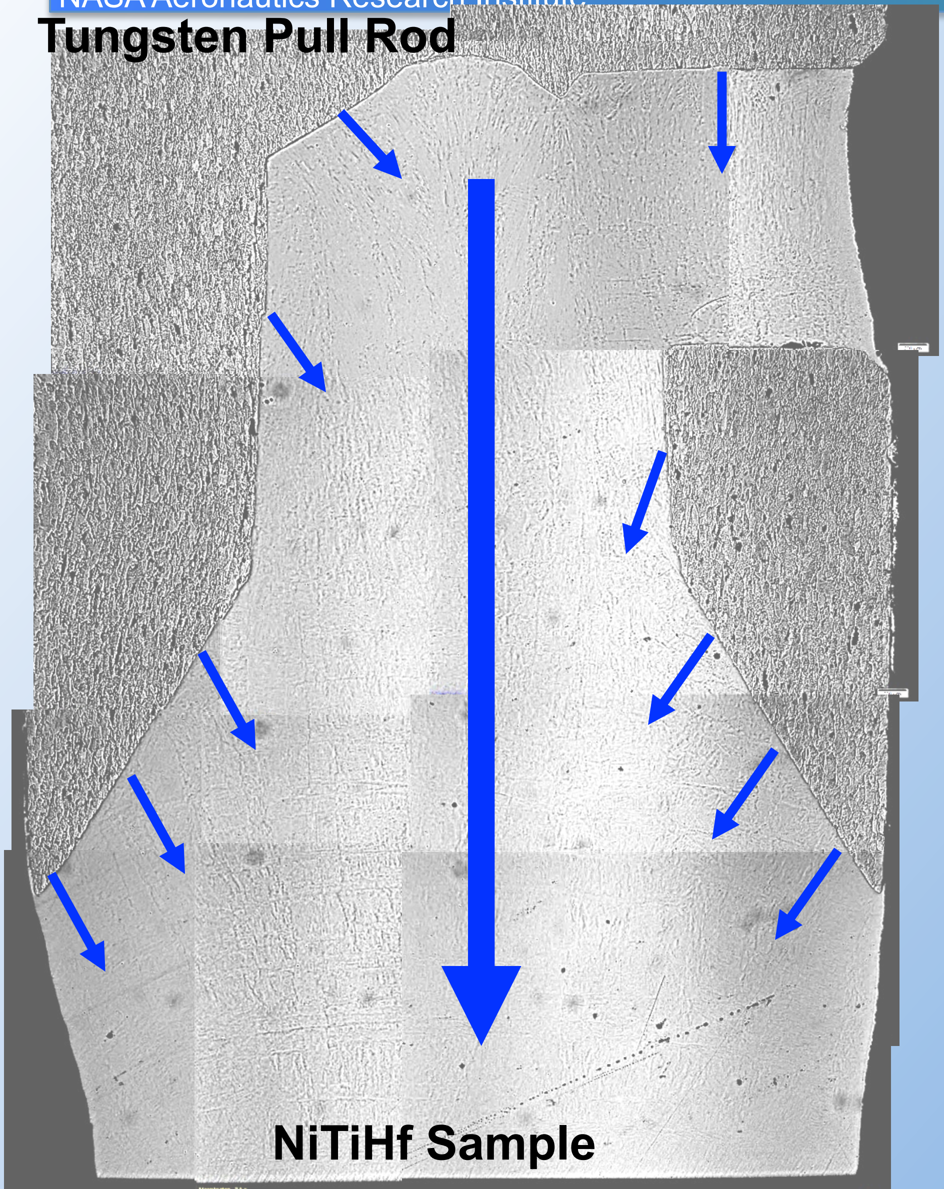

- Grains nucleate on interior surface of cavity
  - Grains from interior surface converge on core and extinguish
  - Causes grain elimination similar to necking

Tungsten Pull Rod with Cavity

W pull rod

Pull rod/melt interface

NiTiHf sample

- Sample diameter controlled by withdrawal speed and/or melt temperature (amperage)
  - Used to form neck and shoulder/body

NiTiHf Polycrystalline Pull Rods

- Flat end
  - Multiple grains at interface
- Pointed end
  - Minimize number of grains for templating
- Offset point
  - Minimize grains for templating
  - Cause sideways growth

NiTiHf Single Crystal Seed

- Preferred method
  - (Assuming single-crystal seed)
- Begin with one grain
  - End with one grain (hopefully)
- Initial trial with single-crystal seed
  - Grown using [001] seed
- Will use as [340] seed
  - Grow off of face

X-Ray Diffraction (XRD) Setup

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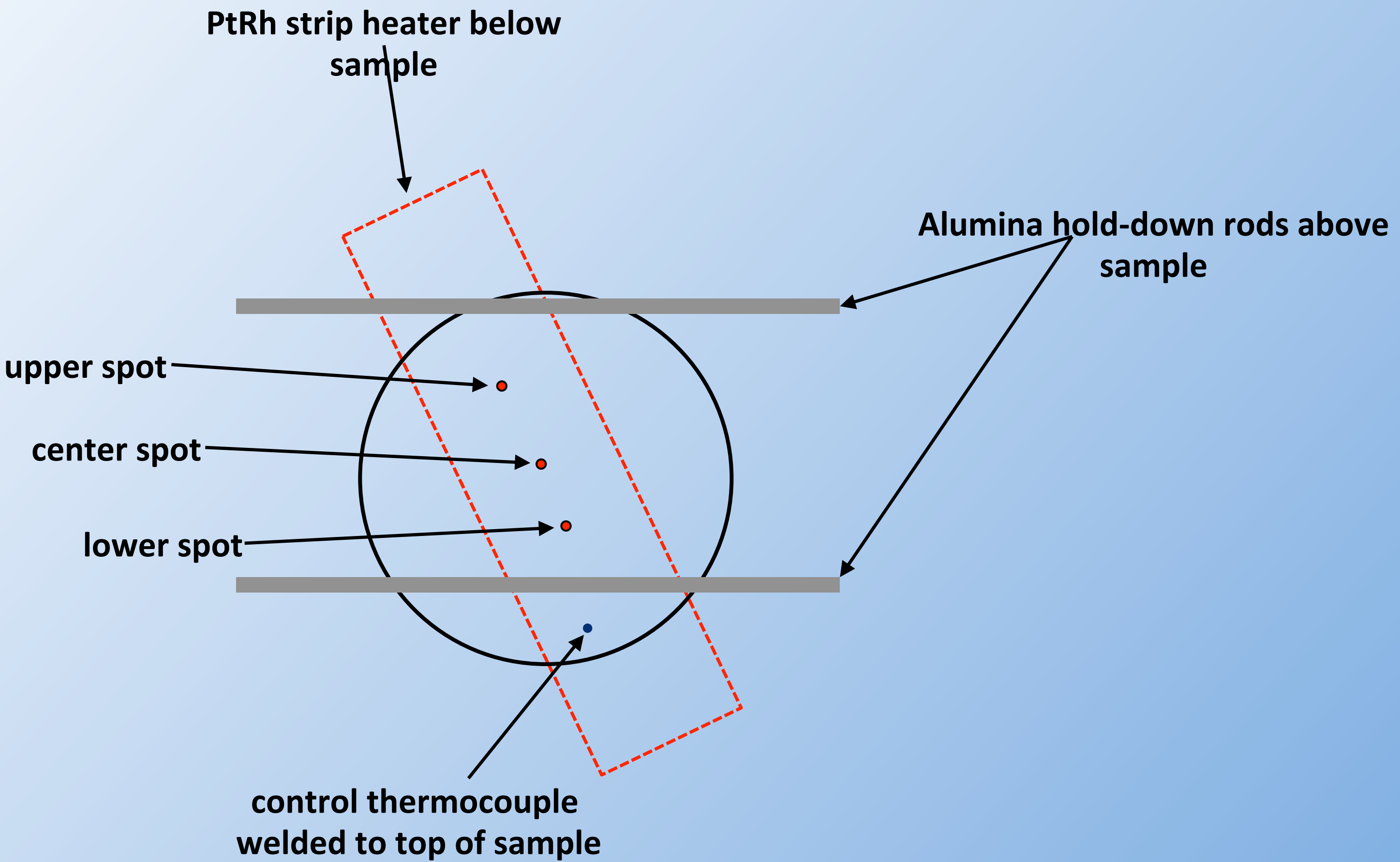
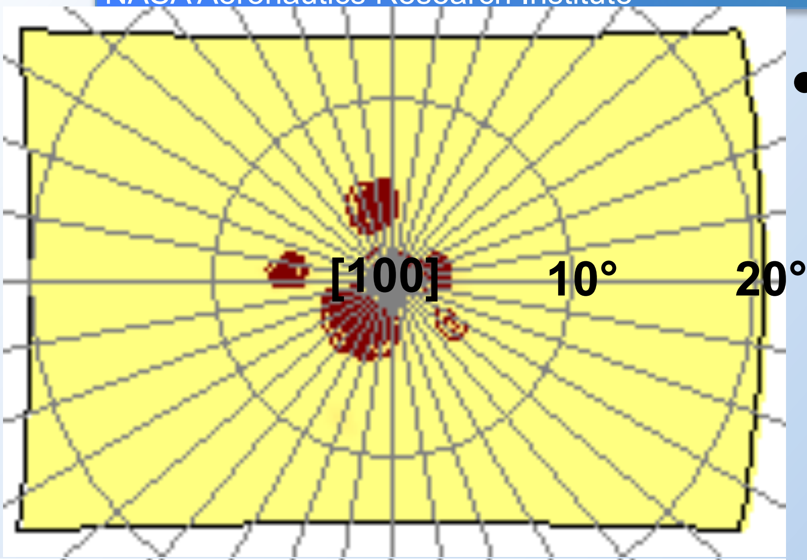
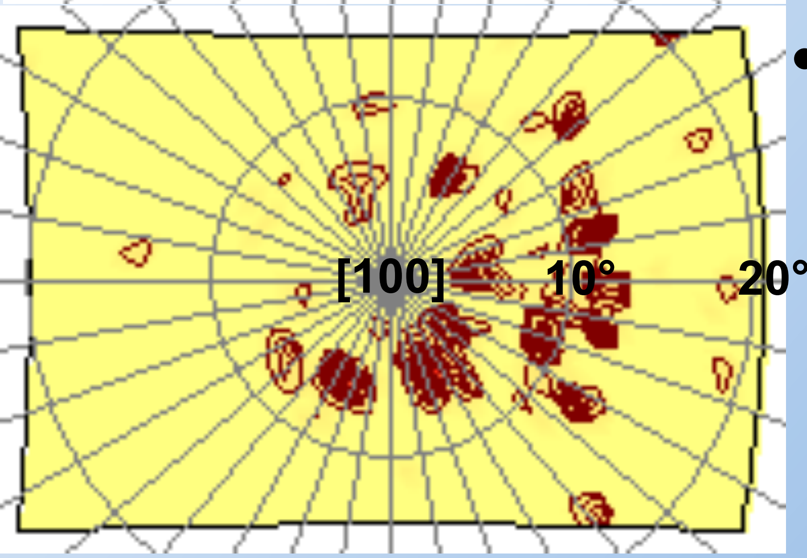


NiTiHf XRD Results (Pole Figures)

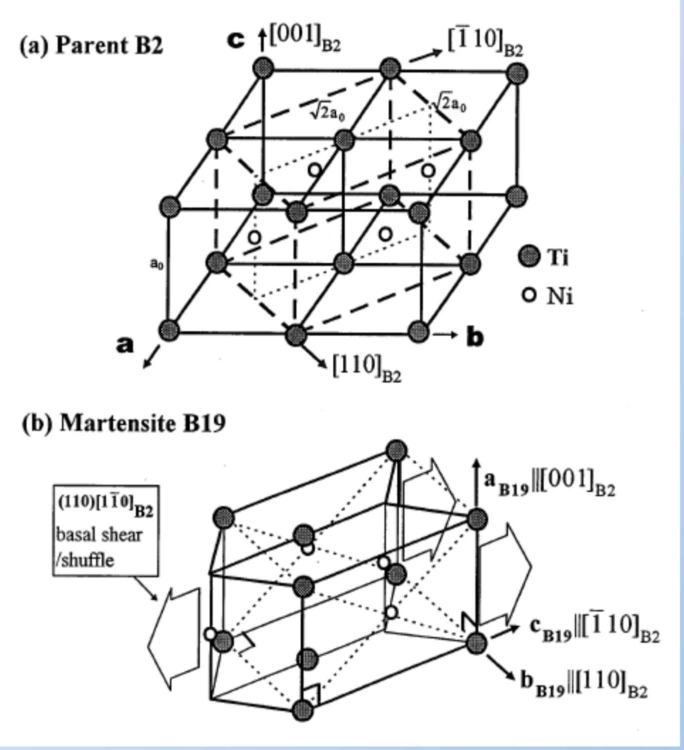




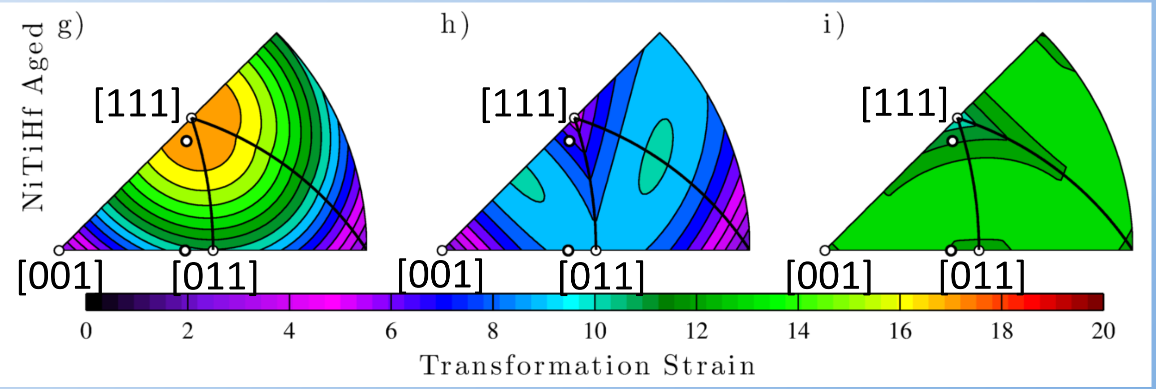
- Results from NiTiHf Single Crystal Seed [001]
  - Few Subgrains within 5°of the [100] direction
  - Average subgrain size = 280μm
- Results from NiTiHf Polycrystalline pull rod
  - Several grains/subgrains within 15°of the [100] direction
  - Average grain/subgrain size = 160μm

NiTiHf Transformation Modeling

Transformation calculation between B2 and single variant of orthorhombic B19 martensite



Theoretical Recoverable Transformation Strain Under Different Loading Conditions



Tension   Compression   Shear

Table of recoverable strain in compression

Orientation	Theory	Observed
[001]	1.72	0.67
[110]	9.36	3.34
[111]	7.19	2.60

Mechanical Testing

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Compression samples machined to ~ 1:2 diameter to length ratio and sized depending on CZ sample size

Reverse load tensile/ compression samples machined with 5mm dia x 14mm long gage

Tensile Load-Biased Shape-Memory Behavior

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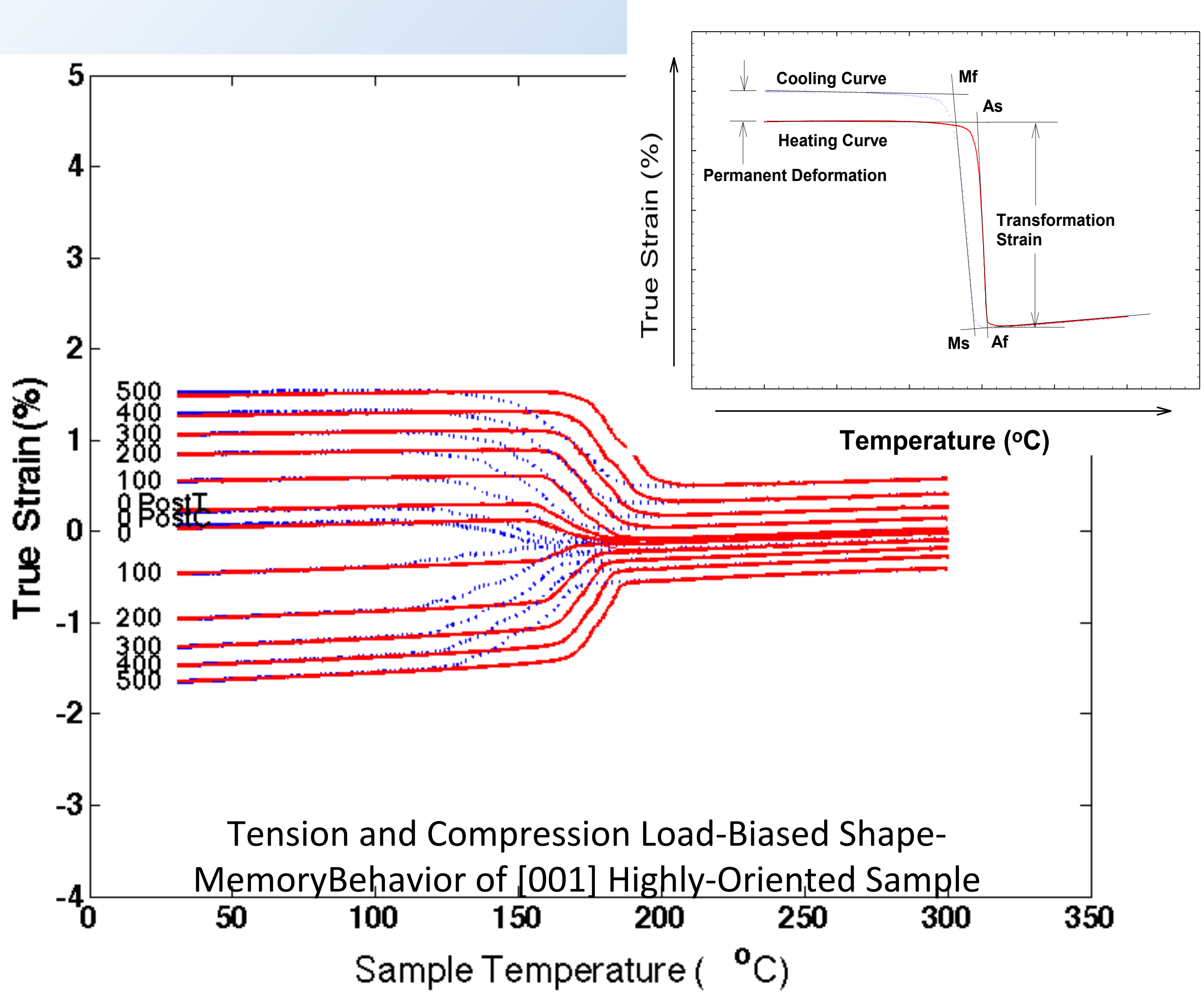
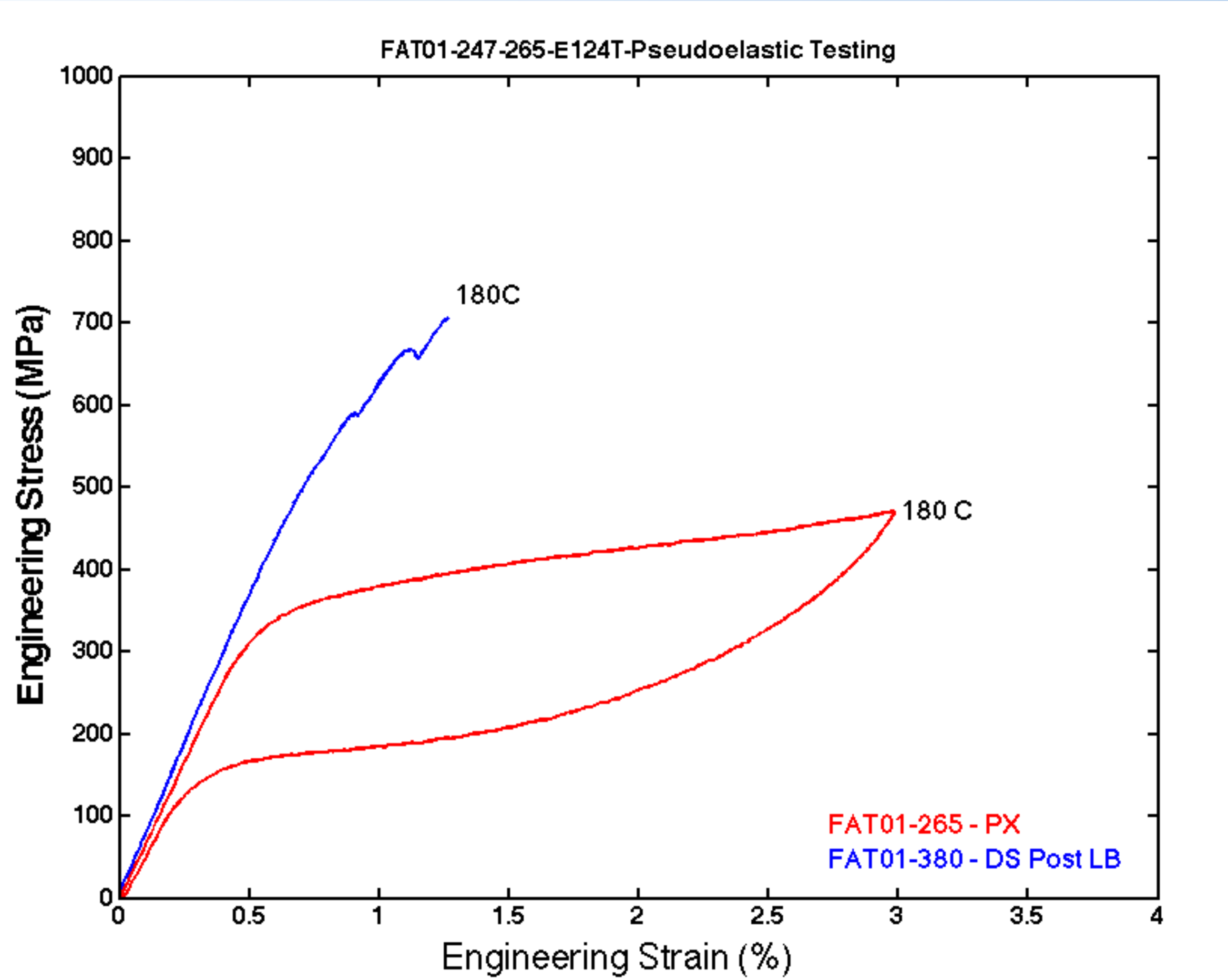
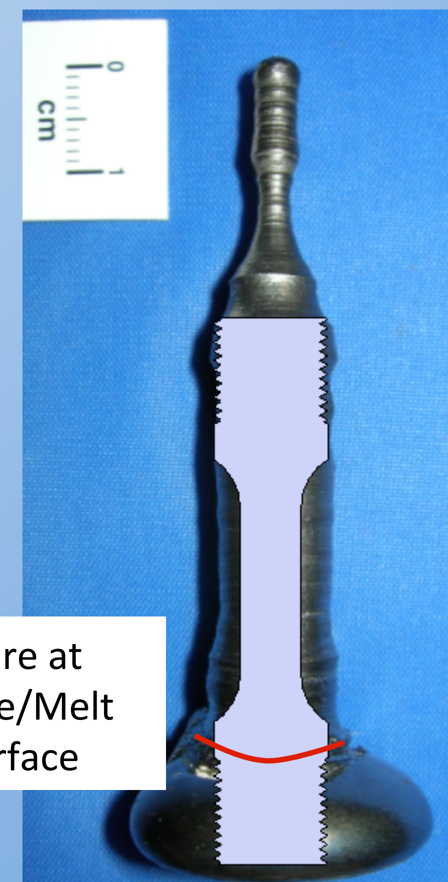


Tension and Compression Load-Biased Shape-MemoryBehavior of [001] Highly-Oriented Sample

First tensile load-biased data for any single crystal HTSMA

Stress-strain comparison – polycrystalline vs highly oriented [100] samples





Failure at Sample/Melt Interface

Transformation Strain in Compression GRC CZ Samples

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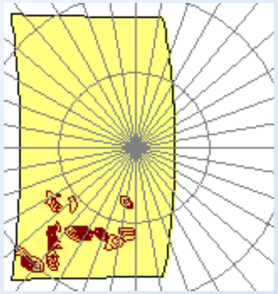
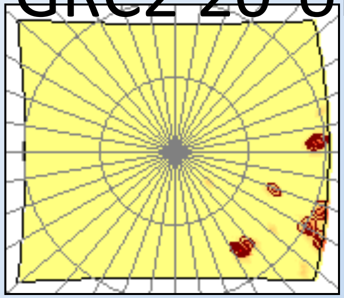
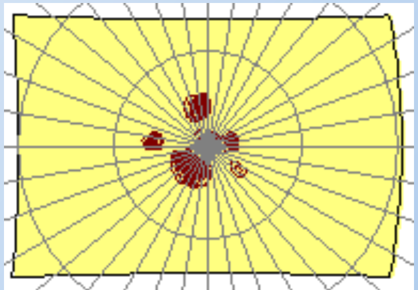
- GRC1 Near [001]

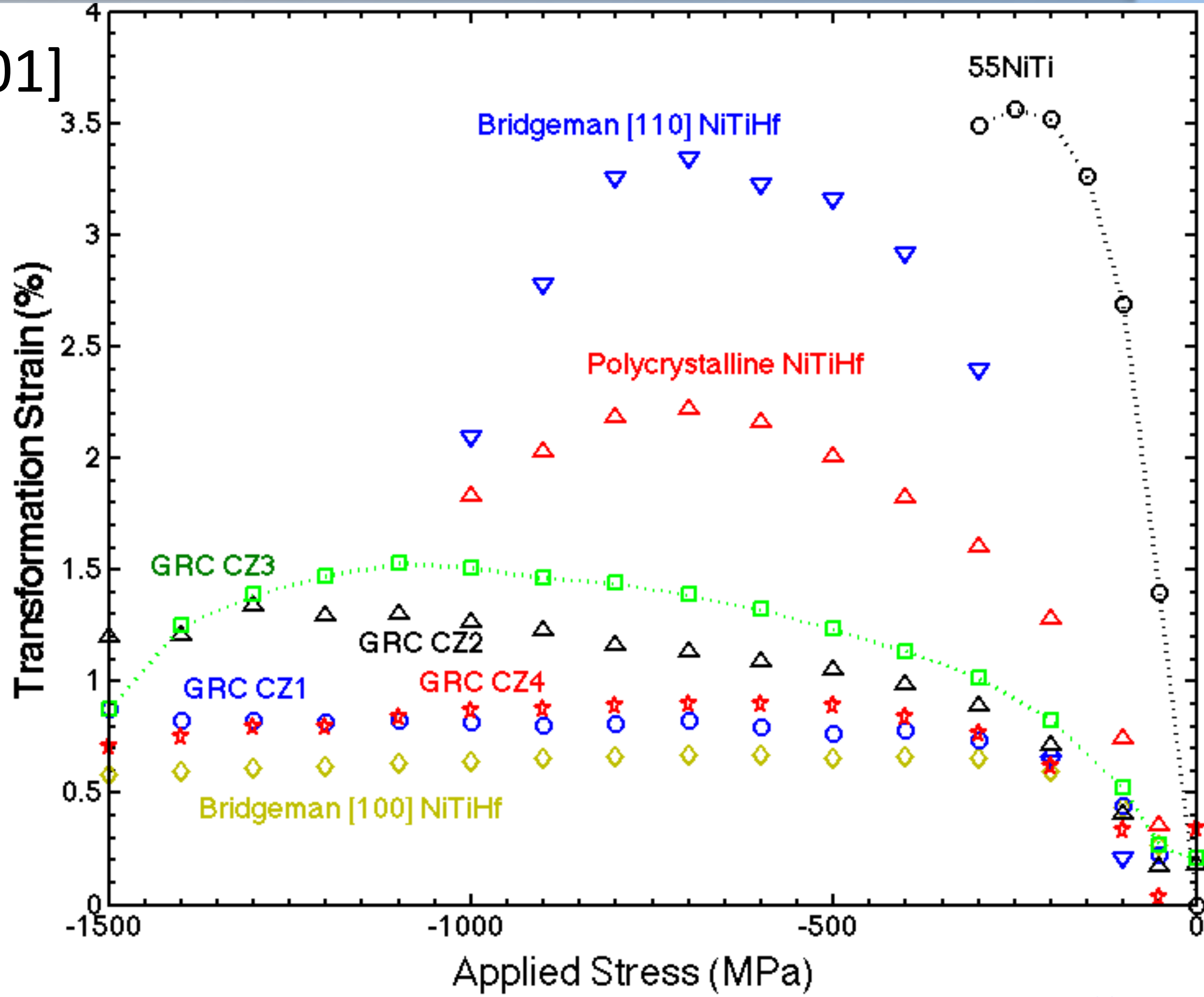


- GRC2 20°off



- GRC3?
- GRC4 Seeded





Work Output In Compression

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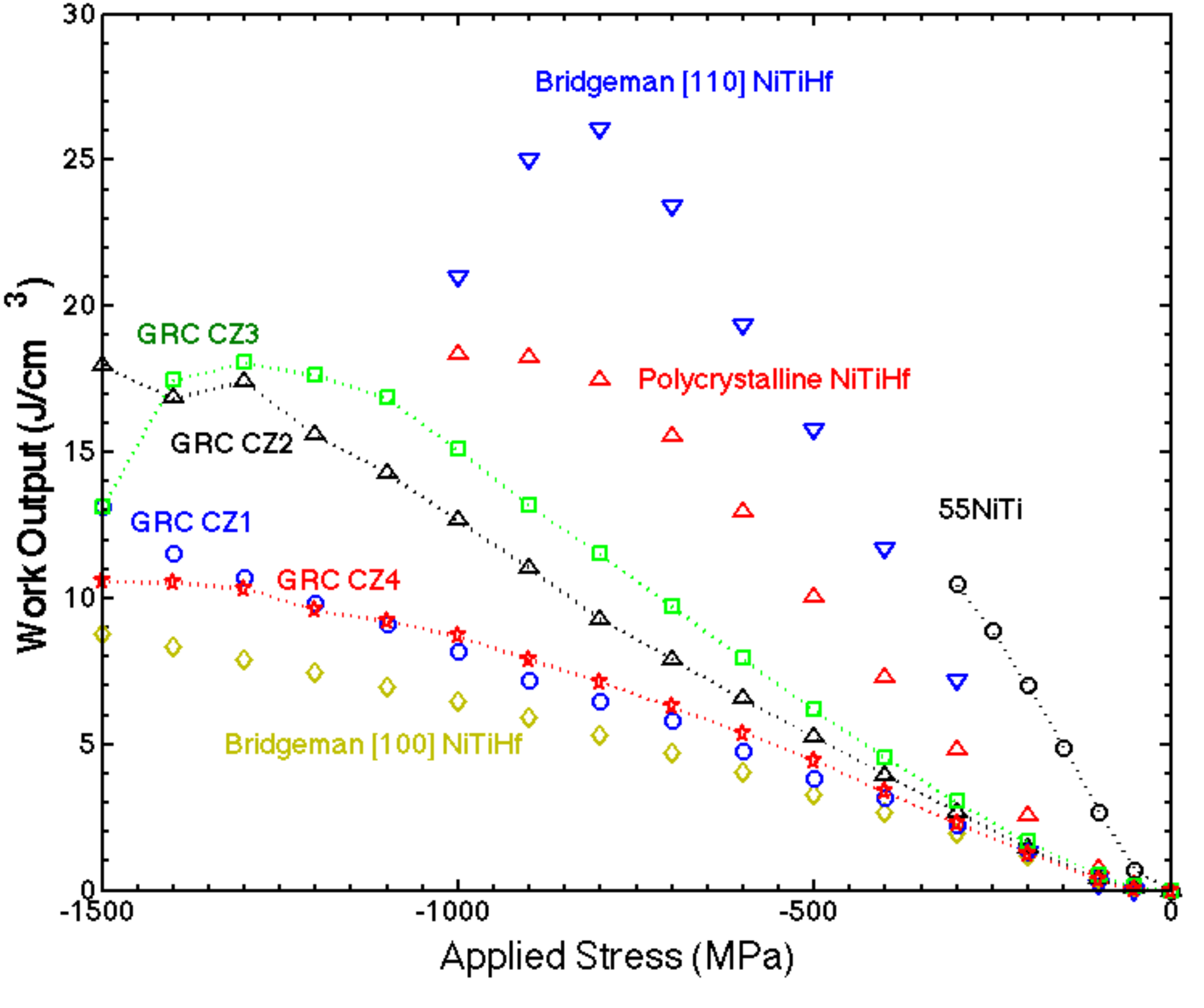
- Work is high due to high stress capability
  - NiTi has low stress capability



Dimensional Stability of NiTiHf

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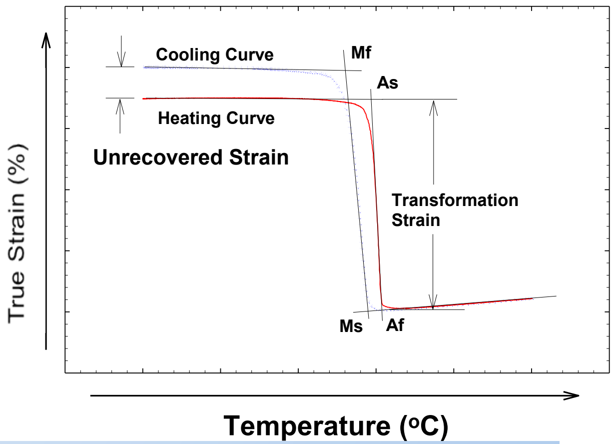
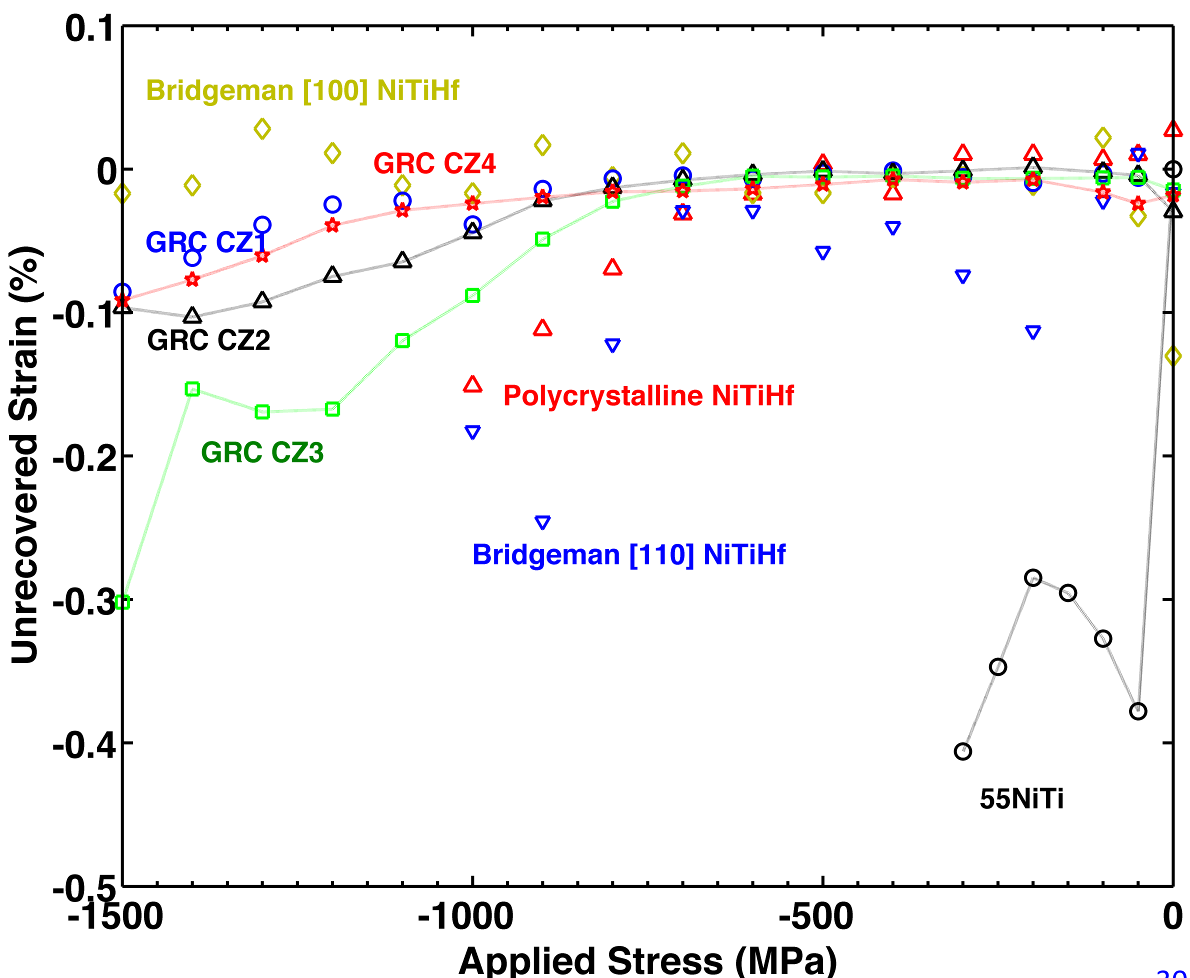
- Highly stable behavior to extremely high stresses





Remainder of Phase II

- X-ray in progress to confirm orientation and quality of crystals, ie low angle boundaries
- Mechanical testing of remaining crystals
  - NiTiHf seeded crystals – [001] and [340] orientations

Payoffs/Impact of Innovation

- Highly oriented samples behave like single crystals
  - Easier to manufacture – may improve commercial viability
- Tailorable properties
  - Very high force (stresses to 1.5GPa or more)
  - High displacement (transformation strain)
  - Hybrid properties
- Properties unobtainable in polycrystalline materials

Plans for Dissemination

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- Patent application on rotating hearth/collar
- Paper on NiTiHf single-crystals incorporating modeling data

Next Steps

- Can be transitioned into projects for aeronautics, space, resource collection, and resource utilization
  - High force and/or strain useful for applications where extreme high stress needed
  - Will be utilized in a recently won CIF project