THE NATIONAL GEM CONSORTIUM

National Aeronautics and Space Administration

Characterizing Microstructure and Properties of a Niobium Alloy Subject to Various Heat-Treatments

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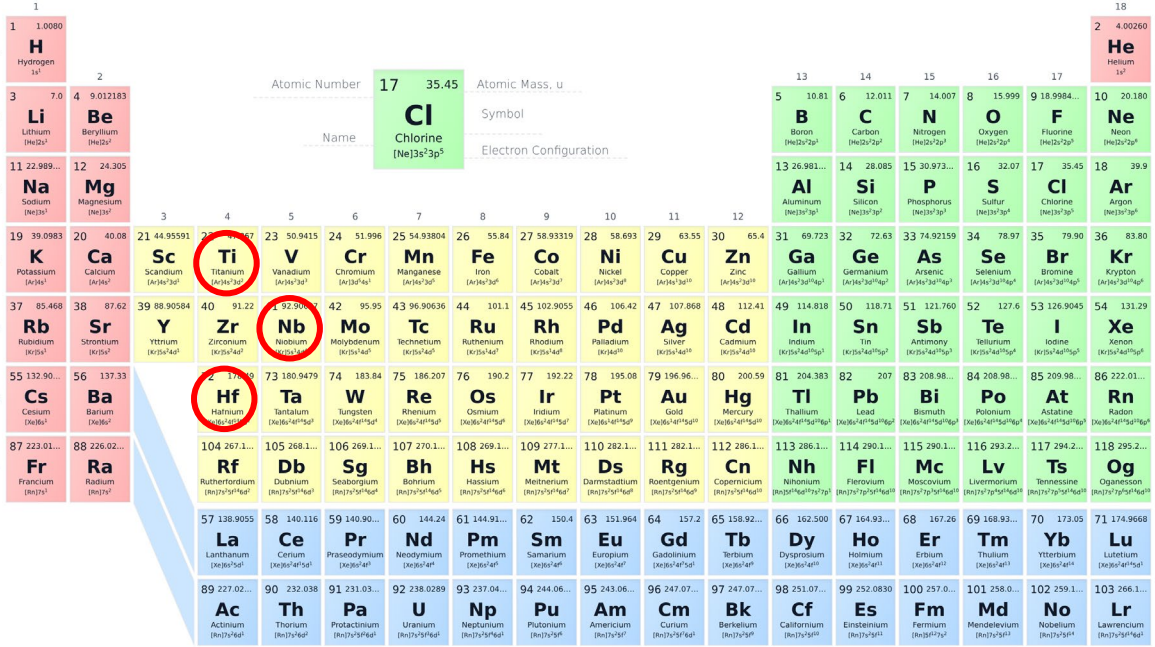
NASA STEM

Introduction

- Nb alloy, C-103, with 10 wt.% Hf, and 1 wt.% Ti
  - Nb-10Hf-1Ti
- Has relatively high strength, good ductility, and maintains a density less than 9 g/cc

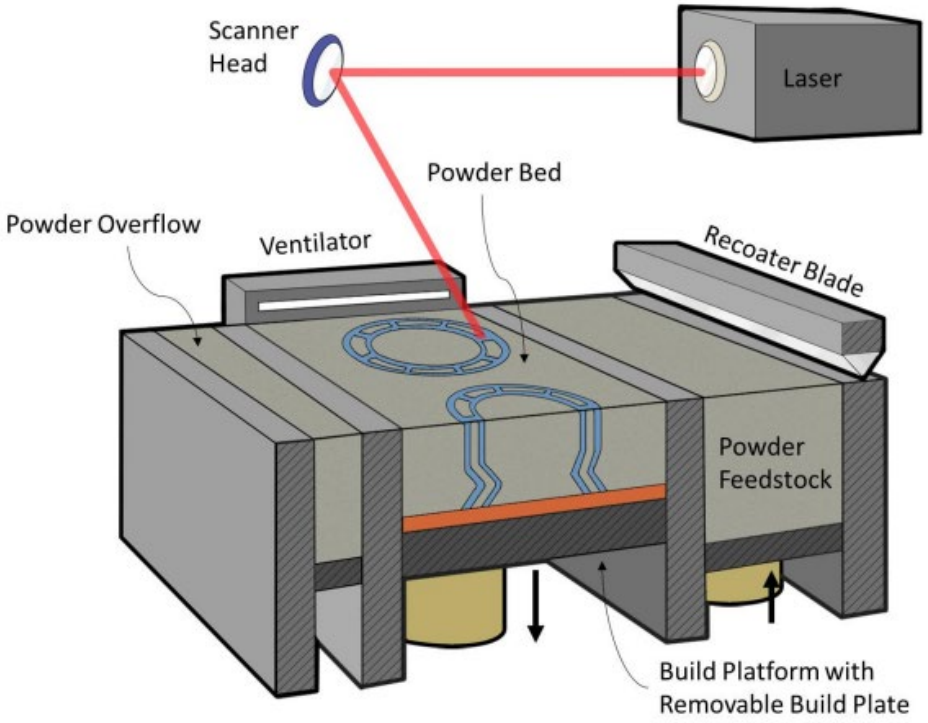


Used to make the apollo command service module, reaction control system R-4D thruster.[1]

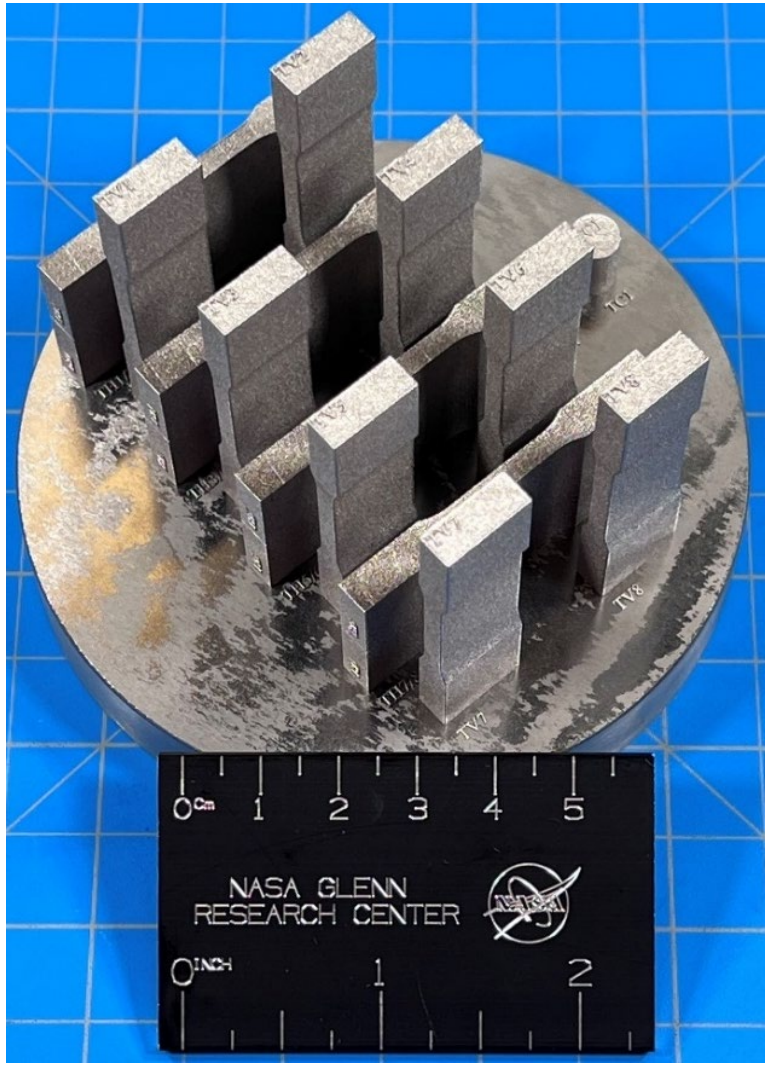
1. Omar Mireles, "Refractory Alloy Additive Manufacture Build Optimization (RAAMBO) - NASA Technical Reports Server (NTRS)," *NASA*, June 14, 2022, accessed August 11, 2023, https://ntrs.nasa.gov/citations/20220008787.

Fabrication of Specimens

- Specimens fabricated by laser powder-bed fusion.
- Fabricated in build (vertical) and transverse (horizontal) directions.



Schematic of laser-powder bed fusion.[2]



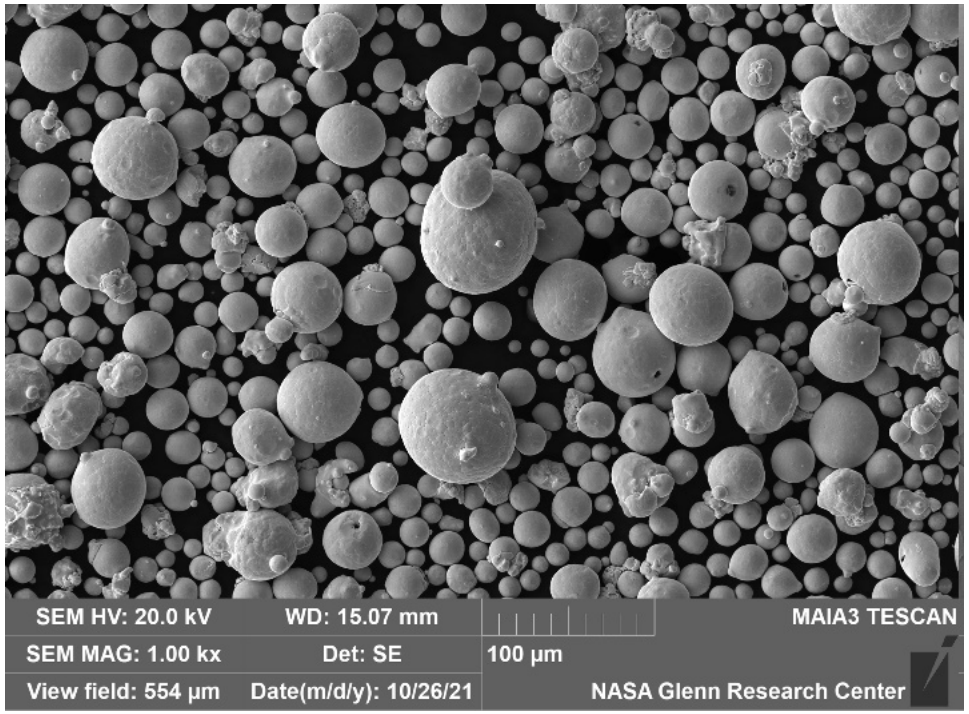
2. Paul Gradl, Omar Mireles, and Nathan Andrews, "Introduction to Additive Manufacturing for Propulsion and Energy Systems - NASA Technical Reports Server (NTRS)," *NASA*, n.d., accessed August 11, 2023, https://ntrs.nasa.gov/citations/20210019758.

Powder Material

- Spherical powder was used in manufacturing
- Analysis of the powders was conducted to verify shape and chemistry:
  - Powder size distribution was conducted using laser-scattering

D_{10}, (µm)	D_{50}, (µm)	D_{90}, (µm)	Density, (g/cm^3)	Hall Flow, (sec/50)
15.4	28.6	49.5	8.83	11

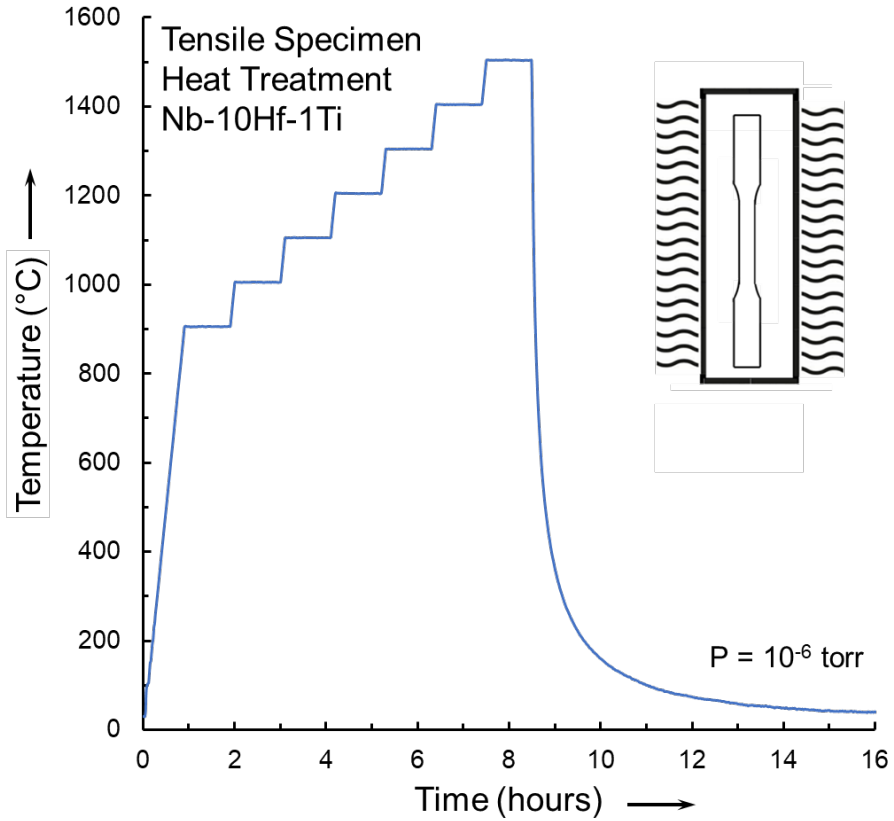
  - Inductively Coupled Plasma mass spectrometry for metals
  - LECO combustion analysis for other elements

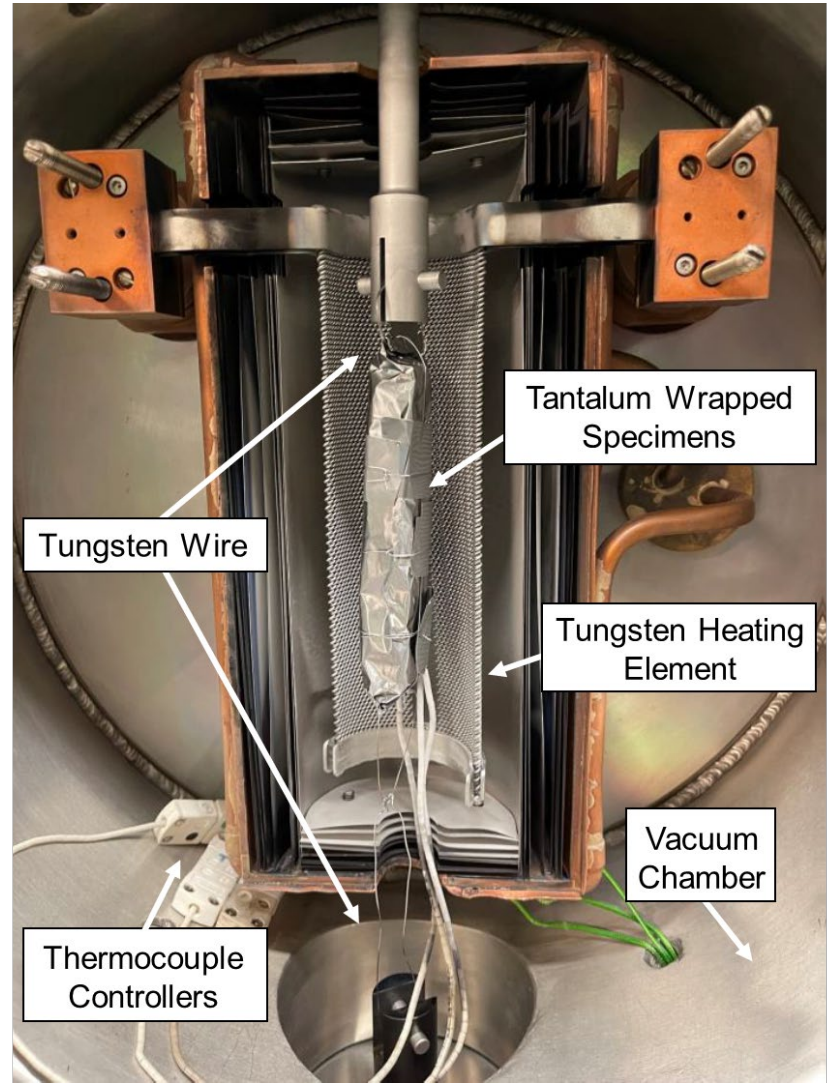


Element	Nb (wt.%)	Hf (wt.%)	Ti (wt.%)	O (ppm)	N (ppm)	C (ppm)	Zr (ppm)	W (ppm)
Powder	Bal.	9.77	0.97	430	52	50	320	2800
ASTM B654	Bal.	9-11	0.7-1.3	<250	<100	<150	<7000	<5000

High-Vacuum Annealing (HVA)

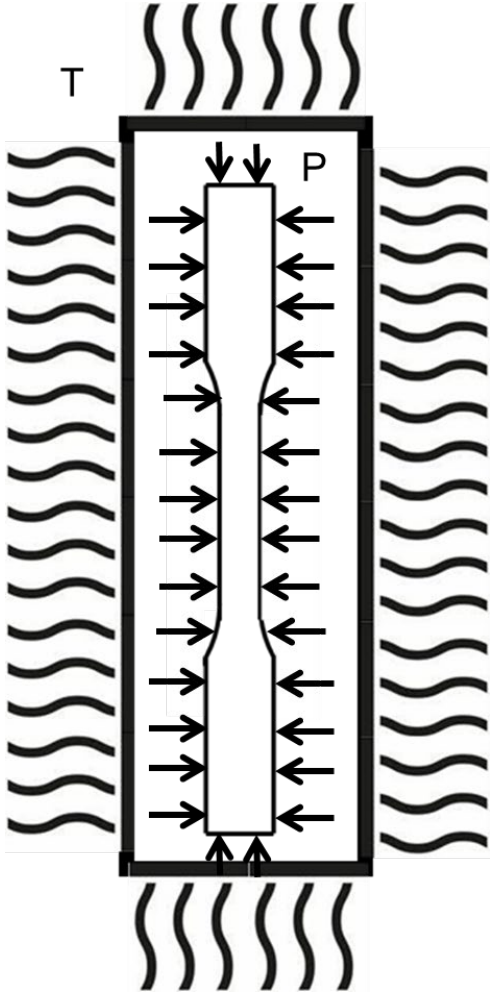
- High vacuum (10^{-6} torr) furnace was used to heat specimens.
- Specimens were wrapped in Ta foil to prevent oxidation.



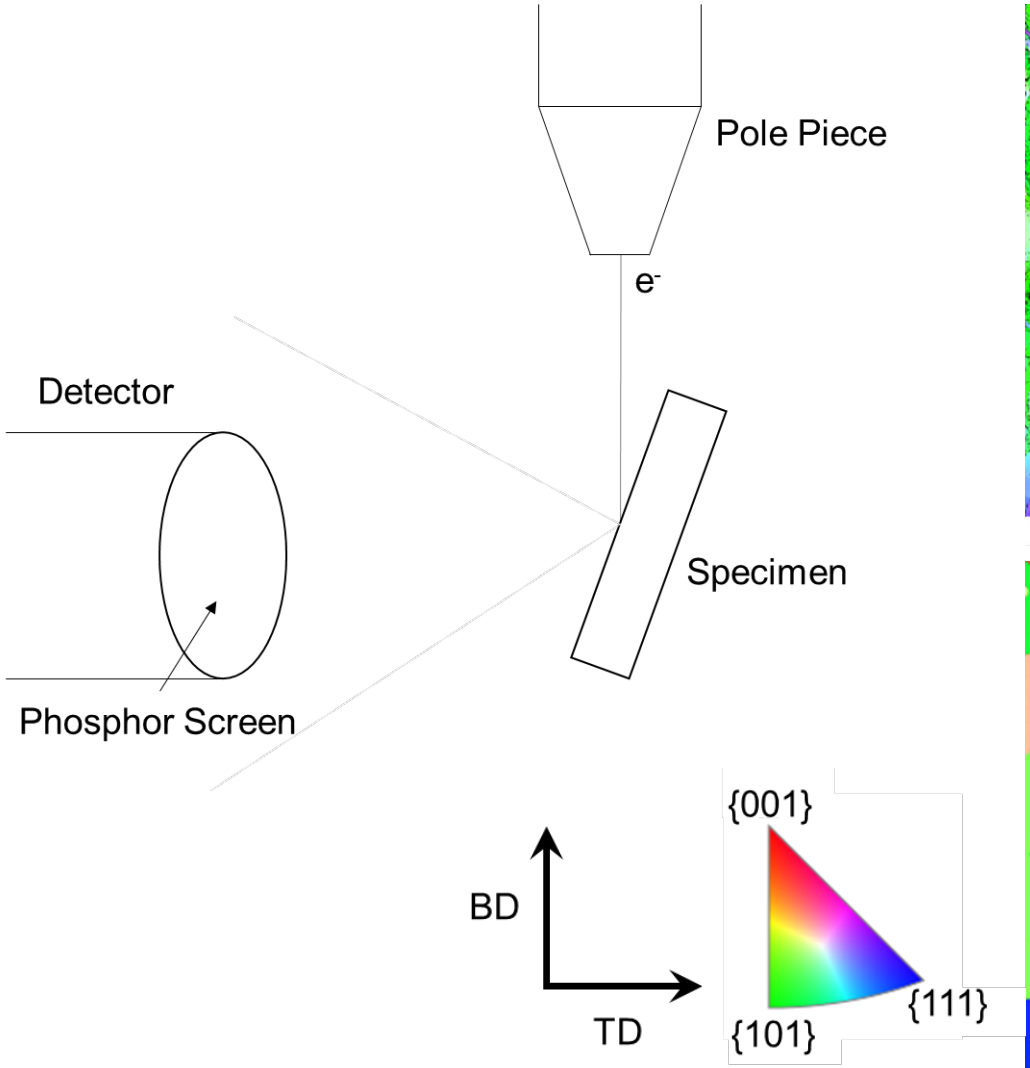


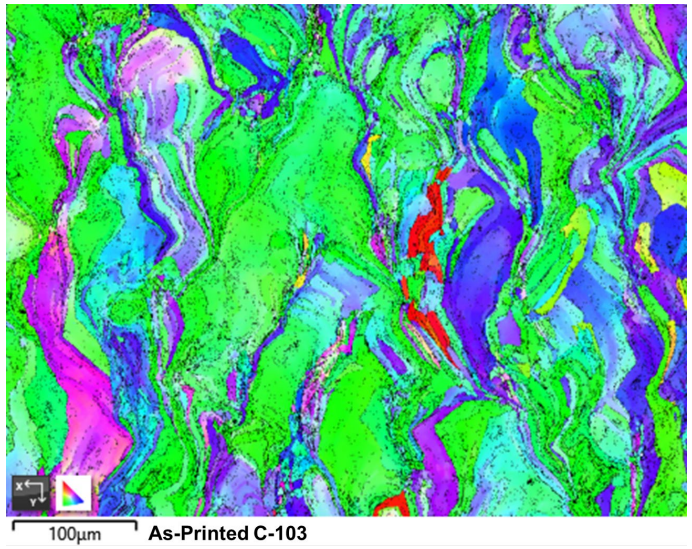
Hot Isostatic Pressing (HIP)

- HIP is a process of heating and pressurizing inert gas around a specimen.
- This is used to collapse surface pores, close cracks, and initiate recrystallization.
- Often used to obtain better microstructure and more isotropic properties in AM parts.

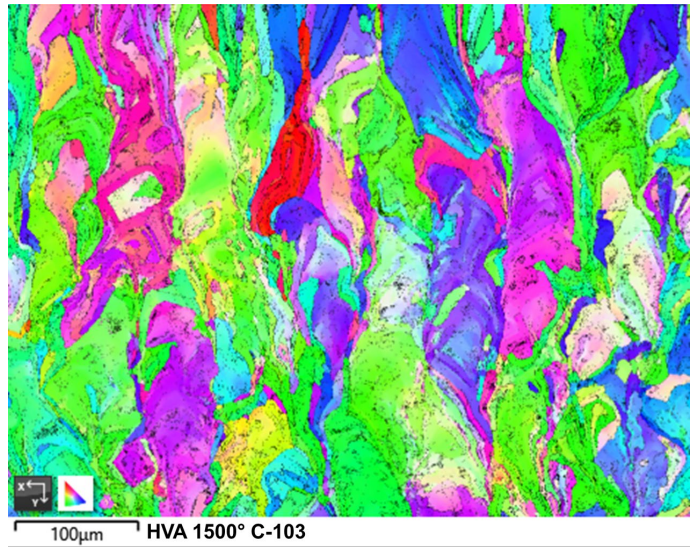


Microstructure Characterization - EBSD

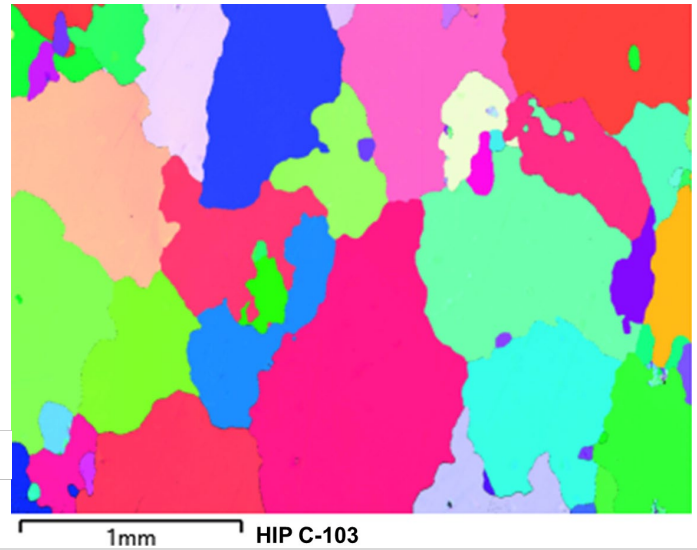




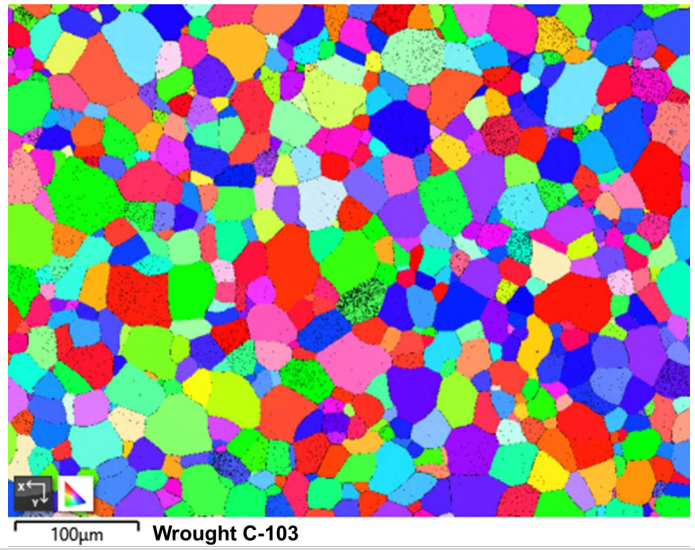
As-Printed C-103



HVA 1500° C-103

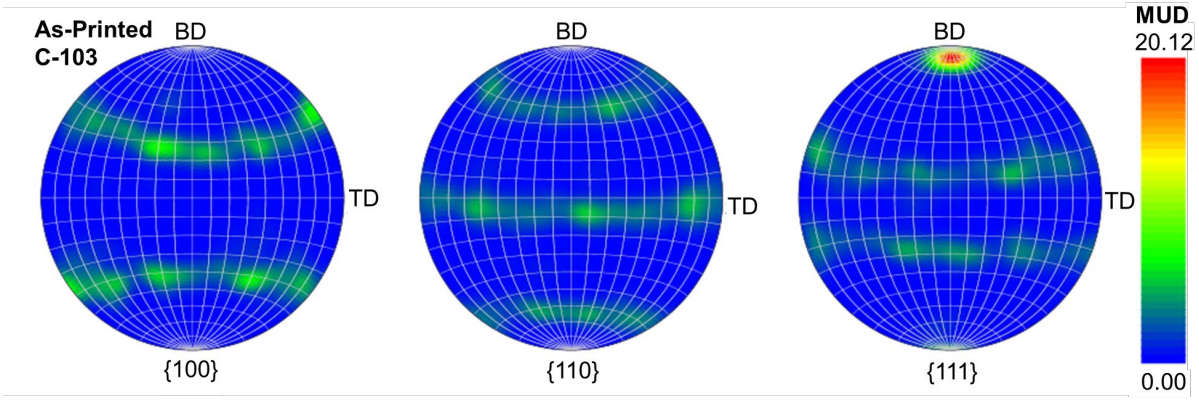


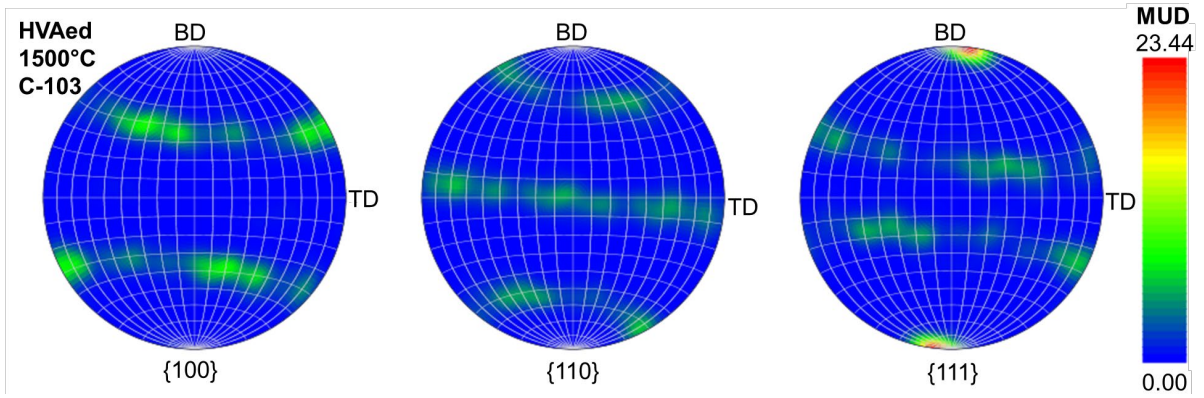
HIP C-103

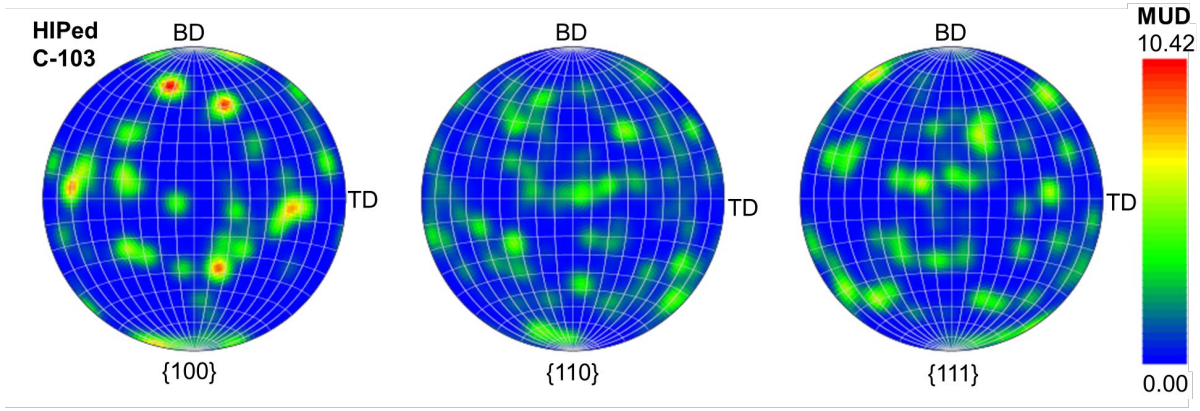


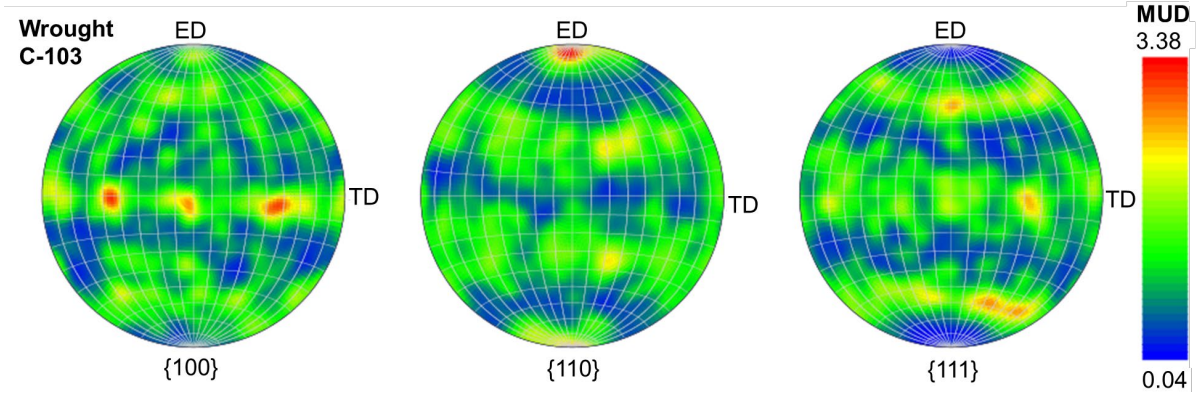
Wrought C-103

Pole-Figures (Texture Identification)



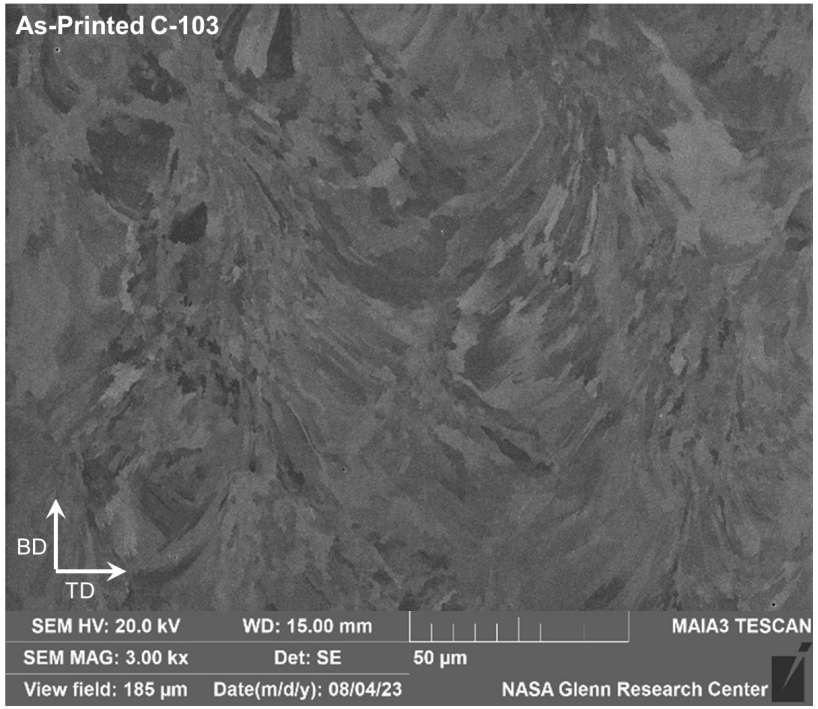


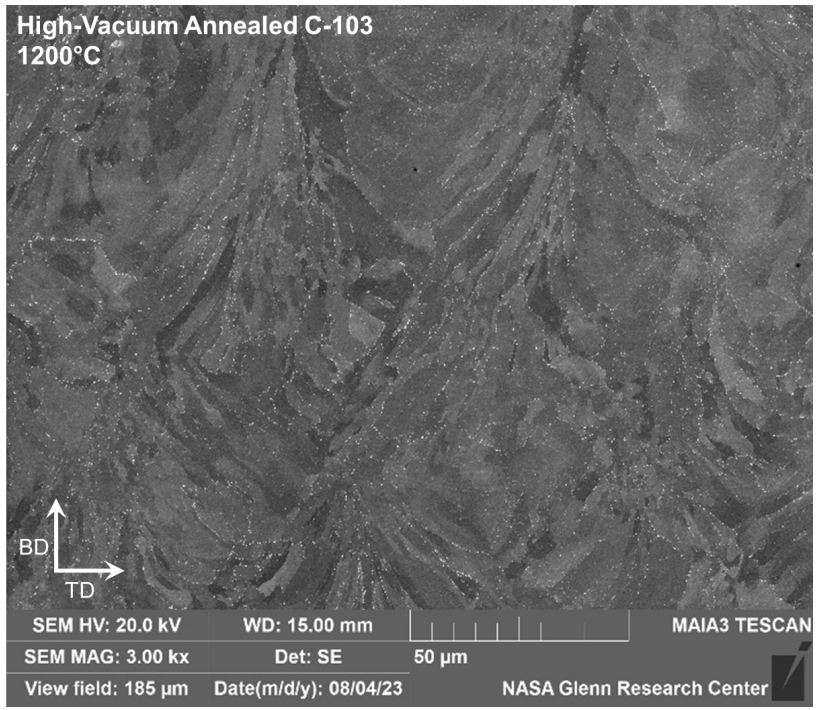


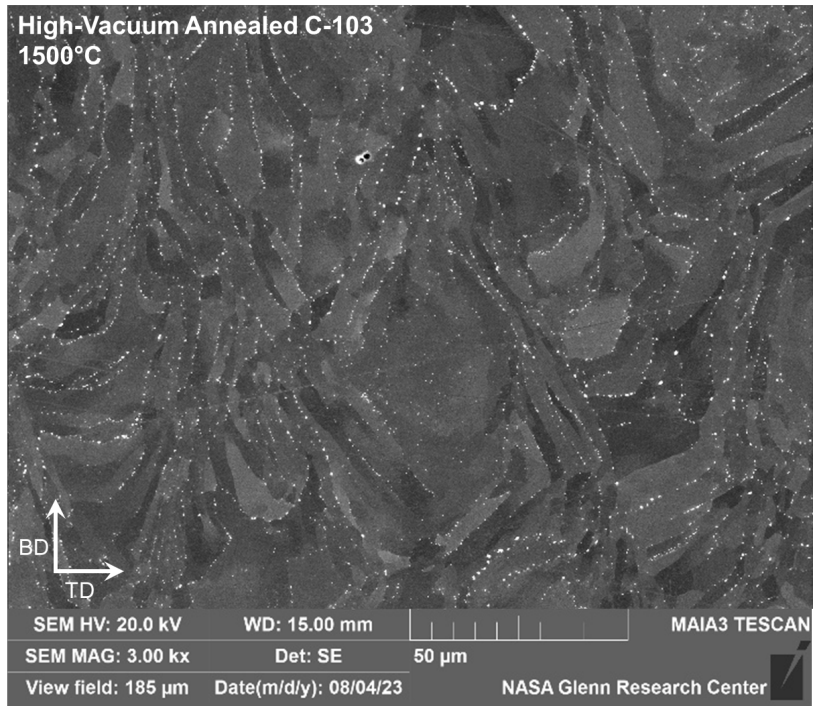


Growth of Precipitates

Increasing Temperature







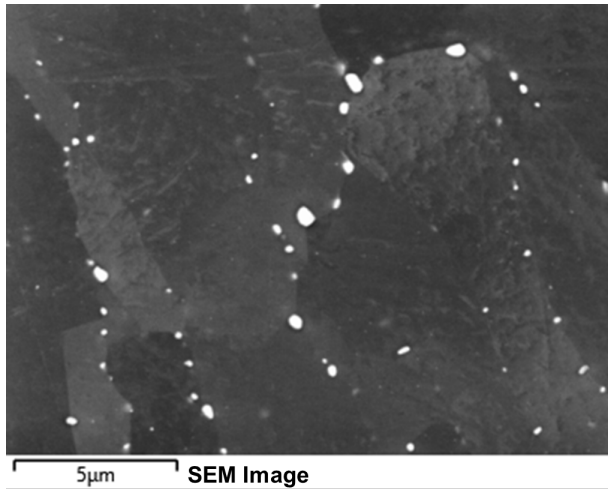
As-Printed

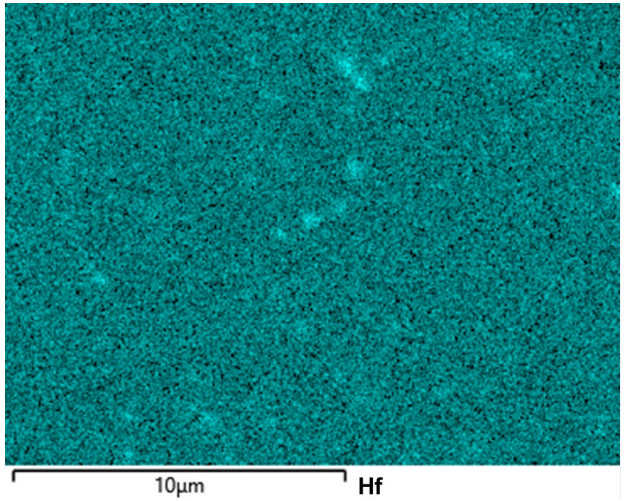
1200°C

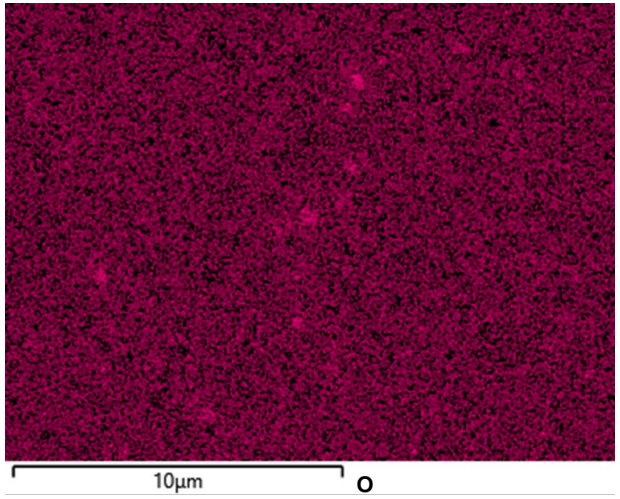
1500°C

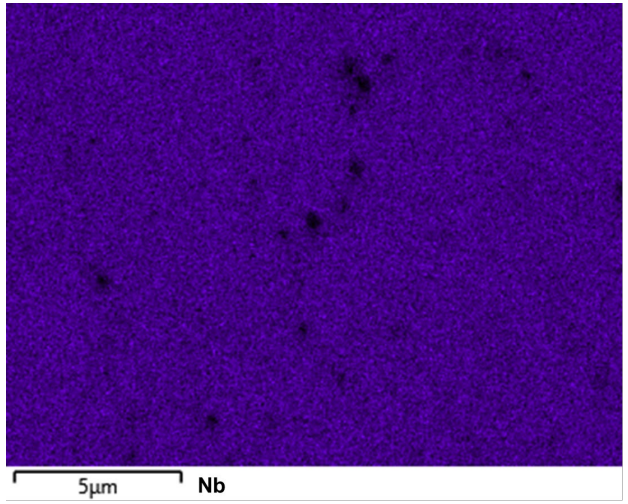
Precipitates grow exponentially with temperature.

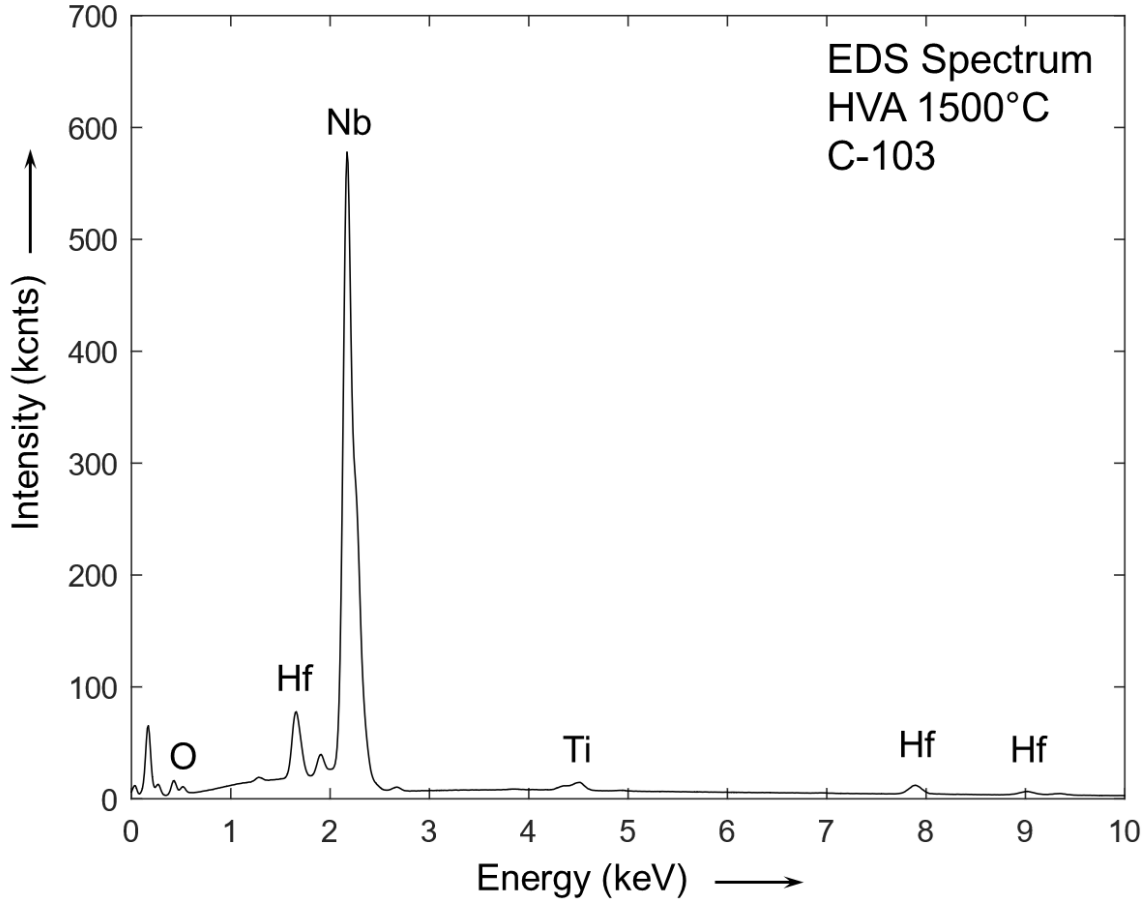
Evaluation of Precipitates





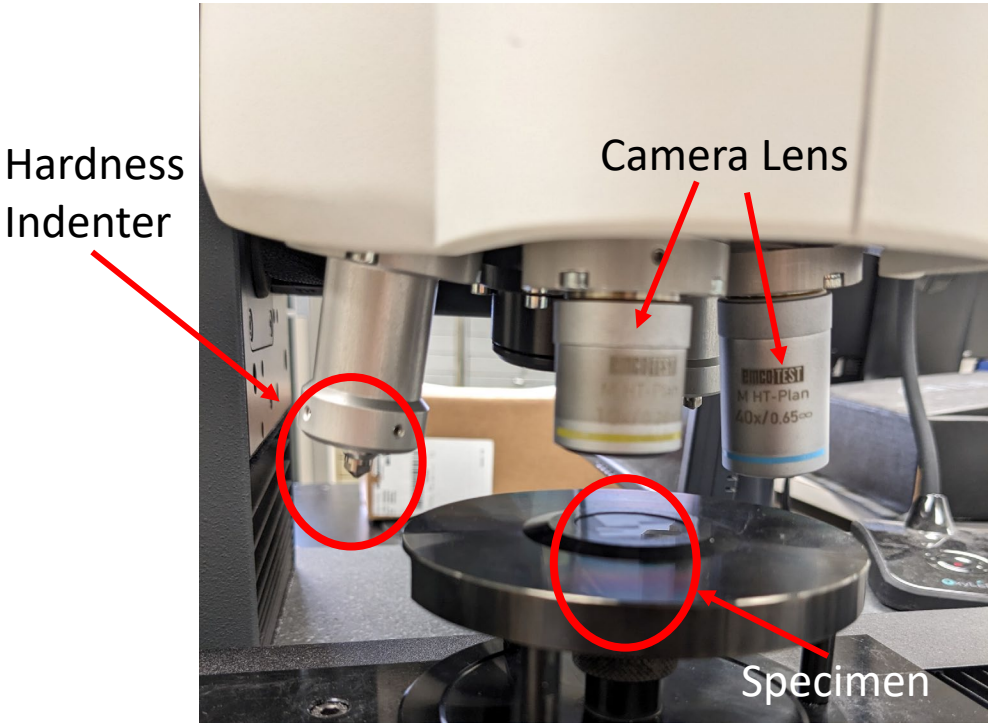


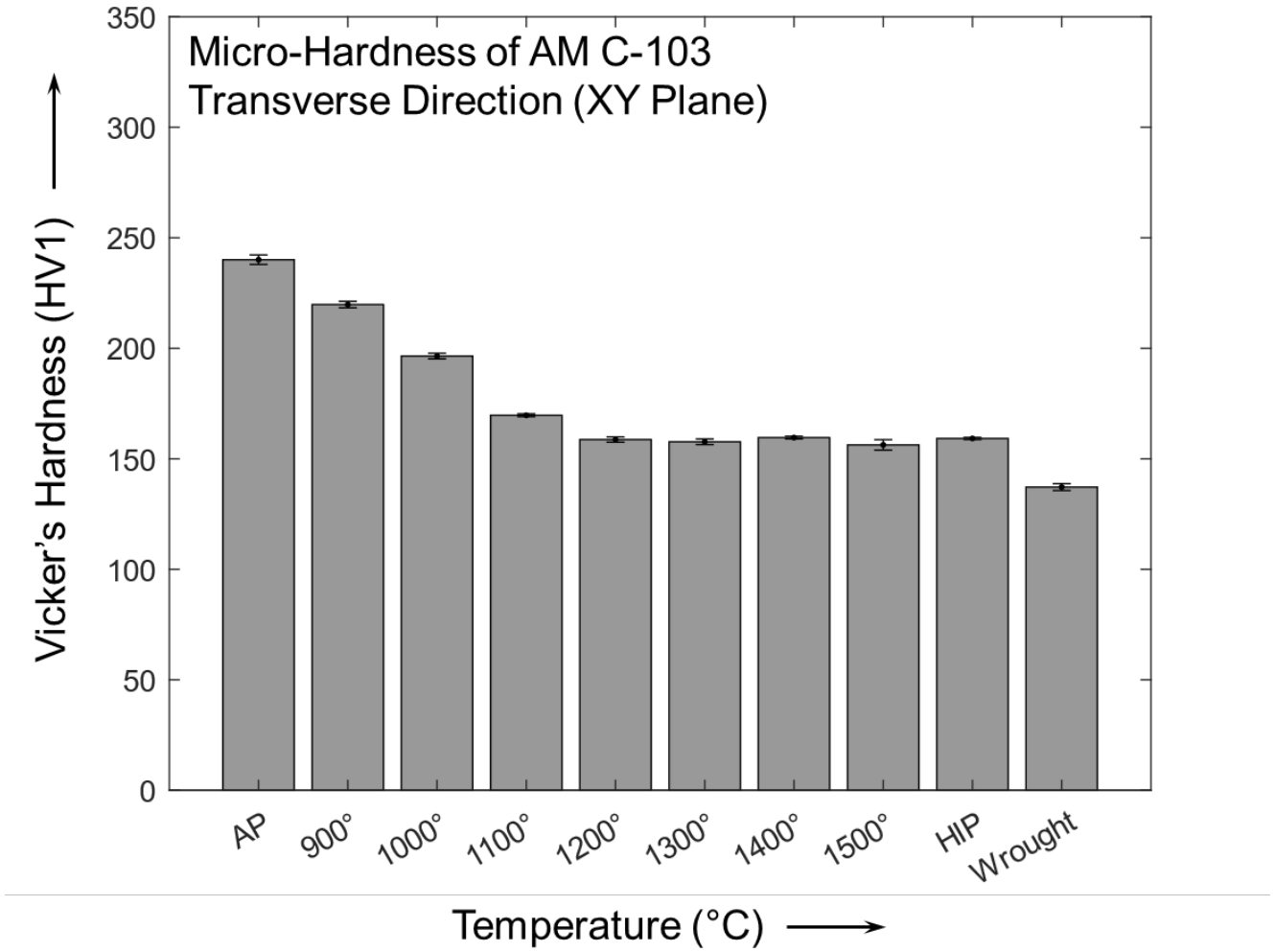




Micro-Hardness Measurements

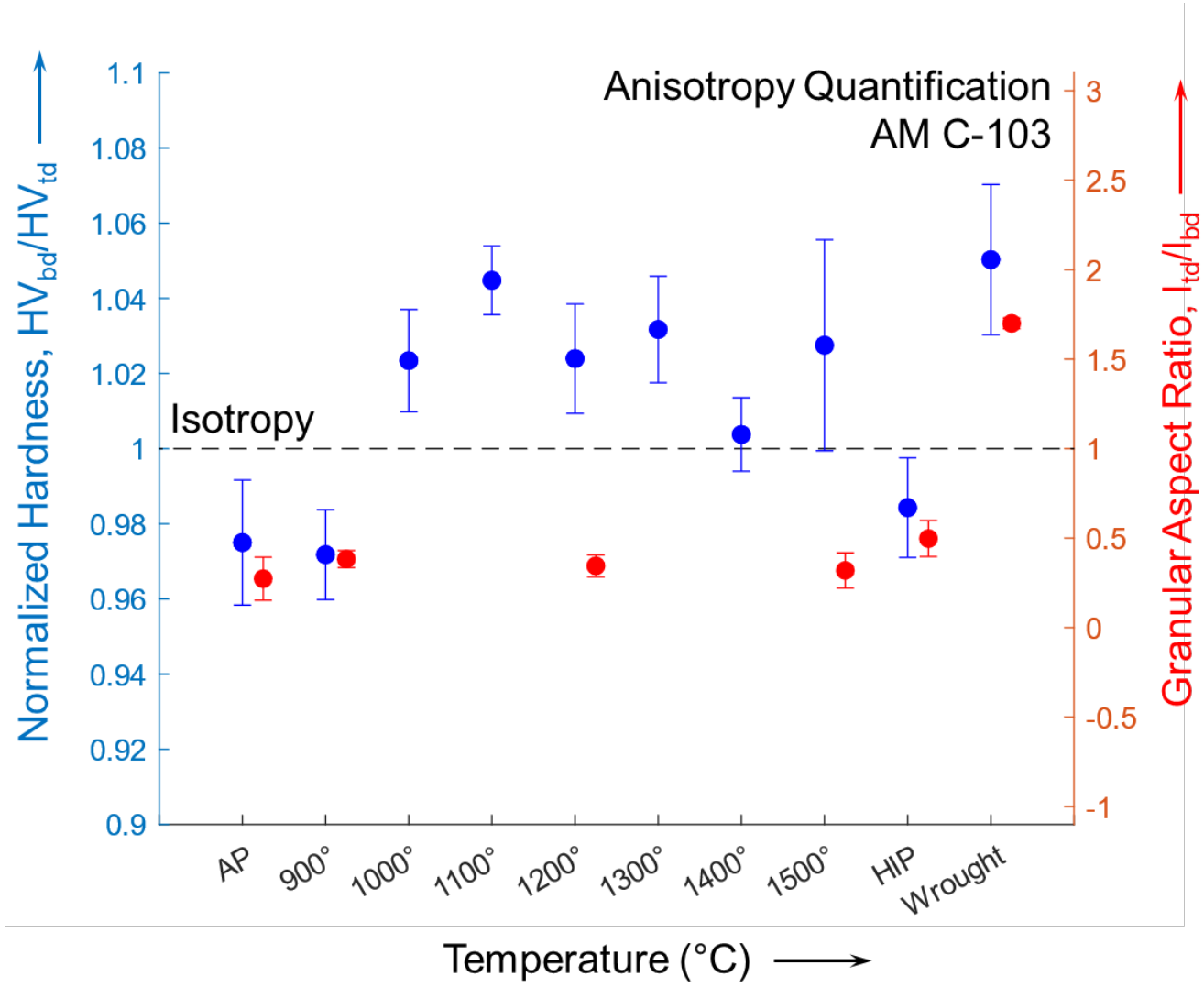
- Conducted micro-hardness testing on all specimens in both build and transverse directions.
- Used 1 kg load and followed ASTM Standard B721





Anisotropy Quantification

- Anisotropy is the difference in material properties with direction.
- Anisotropy in hardness correlates well with granular aspect ratio (geometric anisotropy of grains) for
  - As-printed
  - HIPed
  - Wrought
- Large change in HVA from 900° to 1000°C.
- Anisotropy for HVA is driven by an anisotropic growth of oxides weakening TD more than the BD



Conclusions

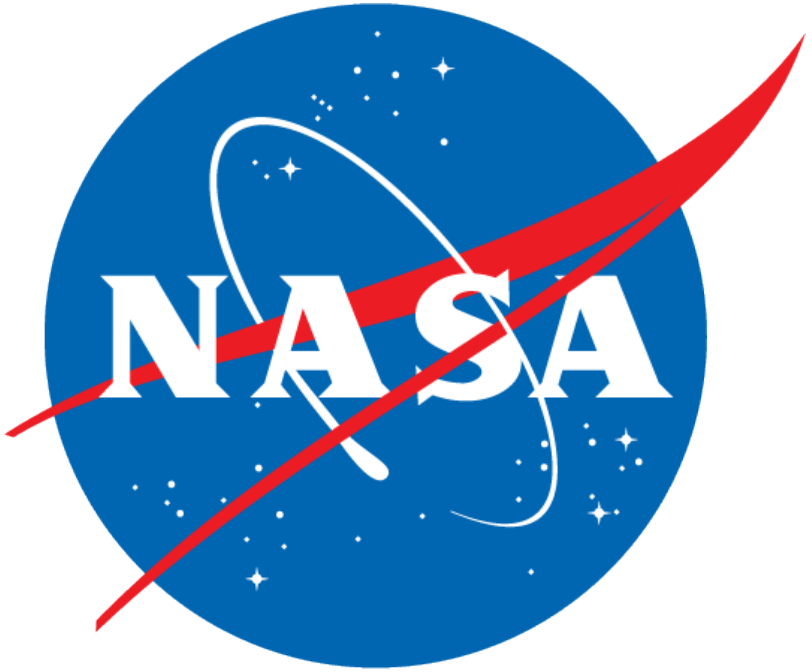
1. Recrystallization and texture change did not occur during high-vacuum annealing, but did occur in hot isostatic pressing.

2. HfO_2 formed in all specimens and grew exponentially with temperature until saturated. Oxide formation showed a strong negative correlation with hardness.

3. Anisotropy of the AP and HIPed specimens was driven by grain aspect ratio, while anisotropy in HVAed specimens depended on oxide growth.

4. Overall, HVA and HIP cannot be used interchangeably but both may serve as viable options for future material engineering endeavors.

Thank you for your attention.



Questions?