Microstructural Characterization Of Additively Manufactured U6Nb During Heat Treatment and Deformation

B. Clausen¹, D. W. Brown¹, T. A. Sisneros¹, S. C. Vogel¹, A. Wu²

1.) Los Alamos National Lab
2.) Lawrence Livermore National Lab

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Advanced Qualification of New Materials Requires a Detailed Understanding of the Linkage Between Processing, Microstructure and Process.
UNb is Complicated


3.5-6.5 wt% : \(\alpha''\)
Monoclinic

6.5-7 wt% : \(\gamma^0\)
Tetragonal

7-8.75 wt% : \(\gamma^0\)
Tetragonal
The UNb Alloys are Shape Memory Alloys


De-twinning of martensitic variants


Stress induced phase transformation.

Structure of AM’ed U6Nb Differs From That Conventionally Produced

Conventionally Produced Material

Additively Manufactured Material

- Something in the microstructure is stabilizing the $\gamma_0$ phase. It is enhanced after heat treating.
  - Other metal impurities?
  - Interstitial oxygen?
  - Oxygen binding with U, effectively increasing Nb concentration?
Heat Treating of U6Nb Completed In-Situ on SMARTS

![Image of lab equipment and a graph showing temperature versus time. The graph has a blue line labeled Tc, and the x-axis is labeled Time (ksec.) with values ranging from 0 to 43.2, and the y-axis is labeled Temperature (C) with values ranging from 0 to 1200. The graph shows a linear decrease in temperature with time.]
Everything we can monitor, chemistry, stress, texture and dislocation density is constant during hold at 1000°C.

Not sensitive to grain growth.

Conclude that microstructural changes happen during heating.

We will have time this spring at APS to monitor microstructural evolution during heat up.
We Performed Deformation In-Situ to Monitor the Microstructure
Development of U6Nb Diffraction Pattern During Tensile Deformation

U6Nb Large Strain, Run number 32747

Normalized intensity [-] vs. d-spacing [Å]
Traditional U6Nb Deforms By Multiple Mechanisms

AM’ed U6Nb Also Has Sigmoidal Flow Curve
Evolution of Diffraction Pattern is Distinct From Conventional U6Nb

AddMan AP U6Nb, Run number 89512

Normalized intensity [-] vs. d-spacing [Å]
Plastic Deformation Accommodated by Stress Induced Phase Transformation

Different initial phase excludes the 2 primary deformation modes of conventional U6Nb.
Phase Evolution is Quantified by Diffraction

As Manufactured

Heat Treated

Phase Fraction
True Strain (%)

Phase Fraction
True Strain (%)
Conclusions

• We have used neutron diffraction to monitor the microstructural evolution of conventional and AM’ed U6Nb under different conditions.
  – As manufactured and heat treated material do not have same crystal structure as wrought U6Nb (α´´).
    – As-manufactured: 2 phase α´´ and γ₀.
    – Heat treated: γ₀.
  – We observe no microstructural changes during 10hrs hold at 1000C.
  – Deformation induced transformation to the α´´ phase.

• Neutron diffraction limited to processes with time scales of minutes-10’s of minutes.

• Current capabilities at APS (1ID) will allow us to measure similar quantities with ~40μs integration time (4 frames).
  – Changes the scale of processes we can study, e.g. microstructural development following deposition.

• MaRIE capabilities will reduce this integration time to <ns, allowing us to study the initial solidification of the printed metal.
  – e.g. solute segregation during solidification.
  – Might need MHz data collection rates.
Variation of Lattice Parameter With [Nb]
Stress Induced $\alpha''$ Looks Like U6Nb
Deformation of AM Material is Reminiscent of Y12 Material

\[ \varepsilon = \varepsilon_p + \varepsilon_e \]
AM U6NB Heat Treating

- AP @ RT
- AP First @ T
- AP 10hrs @ T
- HT @ RT

Normalized Intensity [Arb. Units]
d-spacing [Å]

- 100°C/min to 1000°C.
- 10 hours at 1000°C.
- Oil Quench
How Does the Microstructure Evolve During Heat Treatment?