Microstructural Characterization Of Additively Manufactured U6Nb During Heat Treatment and Deformation

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

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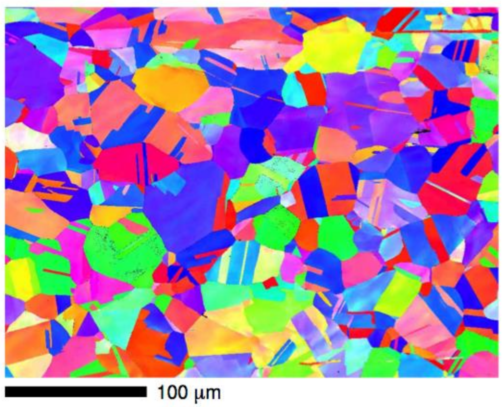
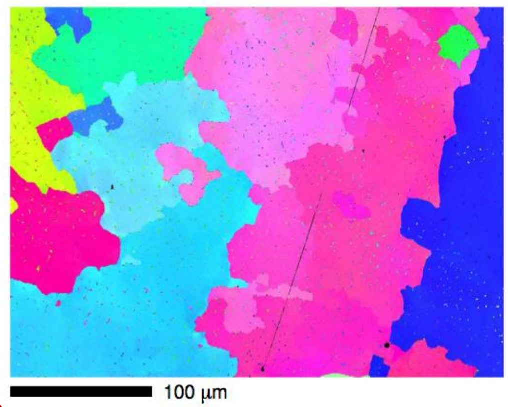
Advanced Qualification of New Materials Requires a Detailed Understanding of the Linkage Between Processing, Microstructure and Process.

Wrought
1.2 % Ferrite/Martensite (Ferrite Scope)



LENS 2 kW
2.3 % Ferrite/Martensite (Ferrite Scope)



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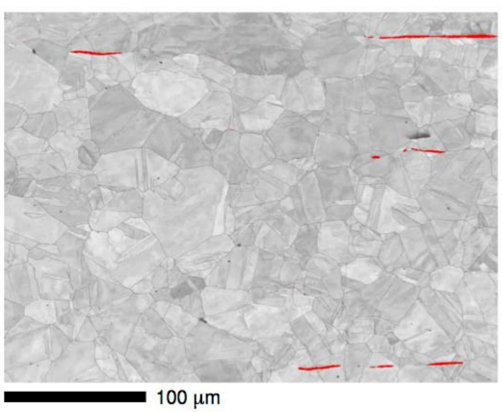

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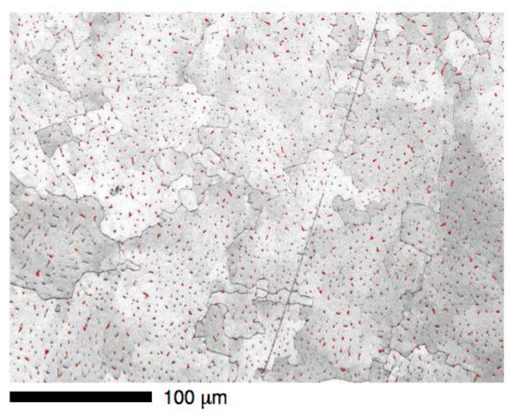
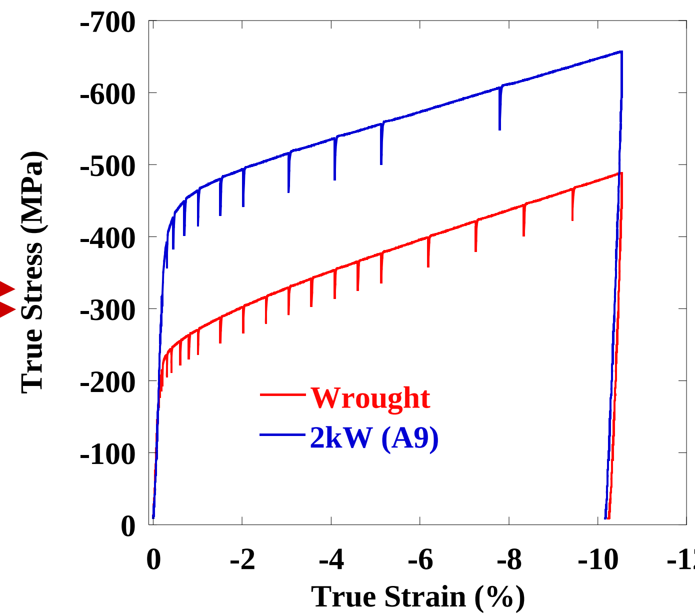
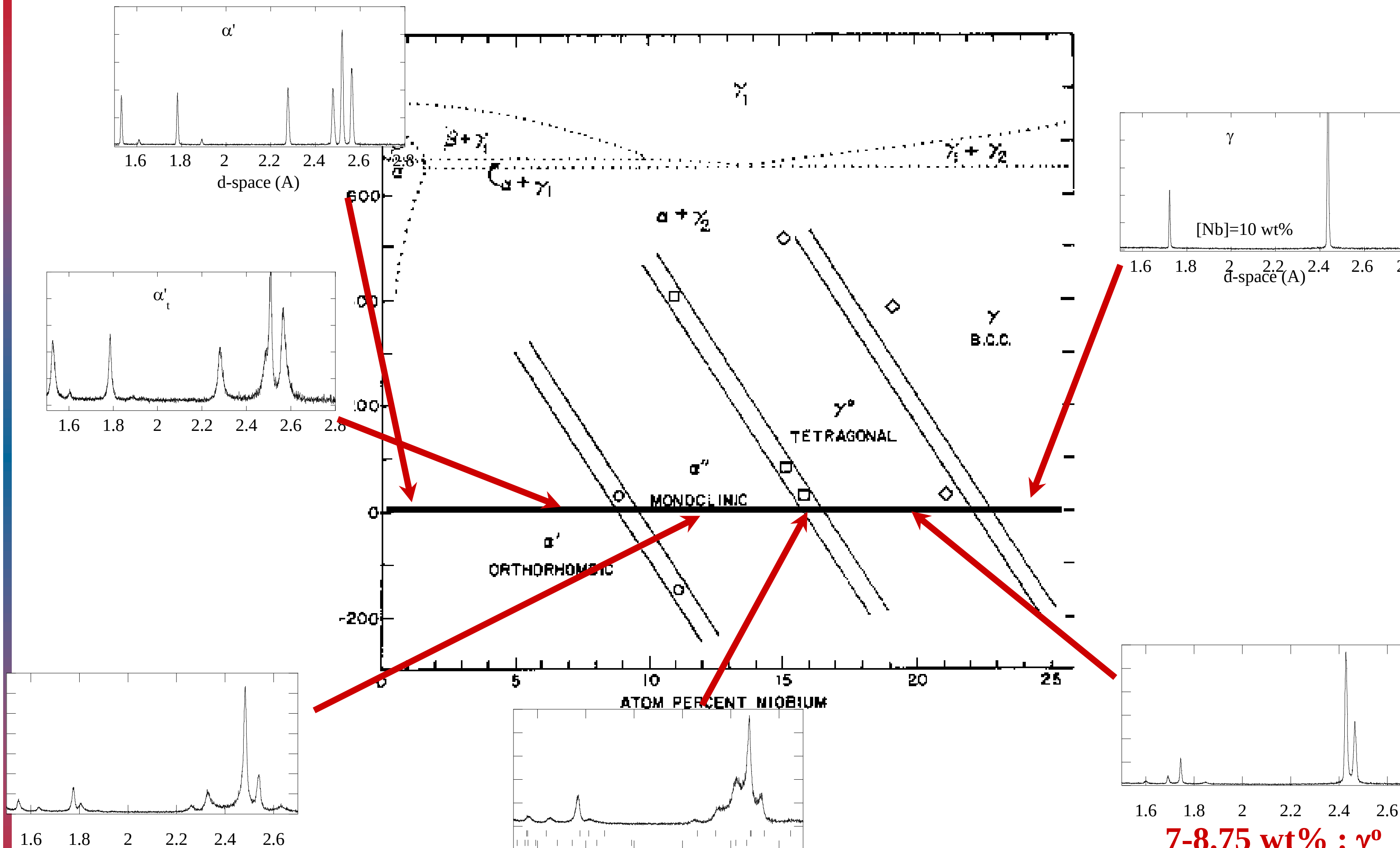




UNb is Complicated



3.5-6.5 wt% : α´´
Monoclinic

6.5-7 wt% : γ°
Tetragonal

7-8.75 wt% : γ°
Tetragonal

Jackson, R.J., *Reversible Martensitic Transformations between Transition Phases of Uranium-Base Niobium Alloys*, 1970, Rocky Flats Division, The Dow Chemical Company: Golden Colorado.

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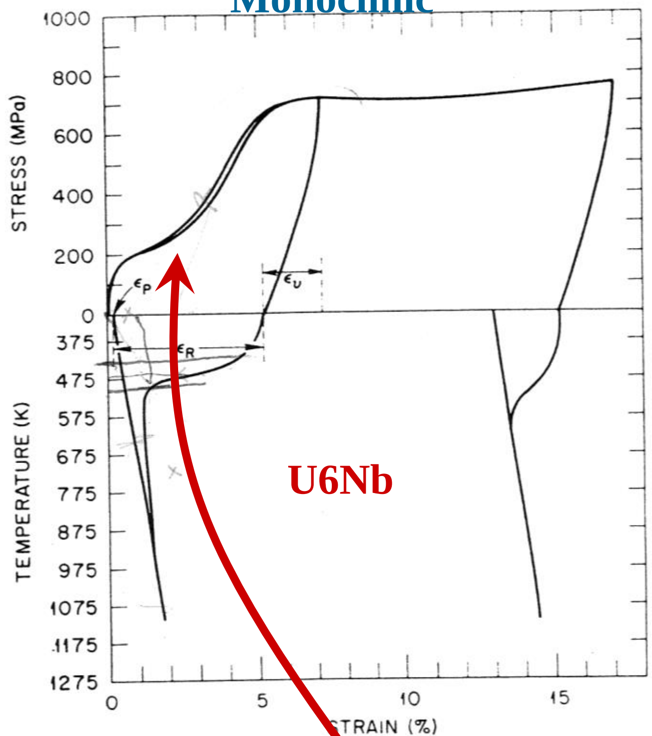
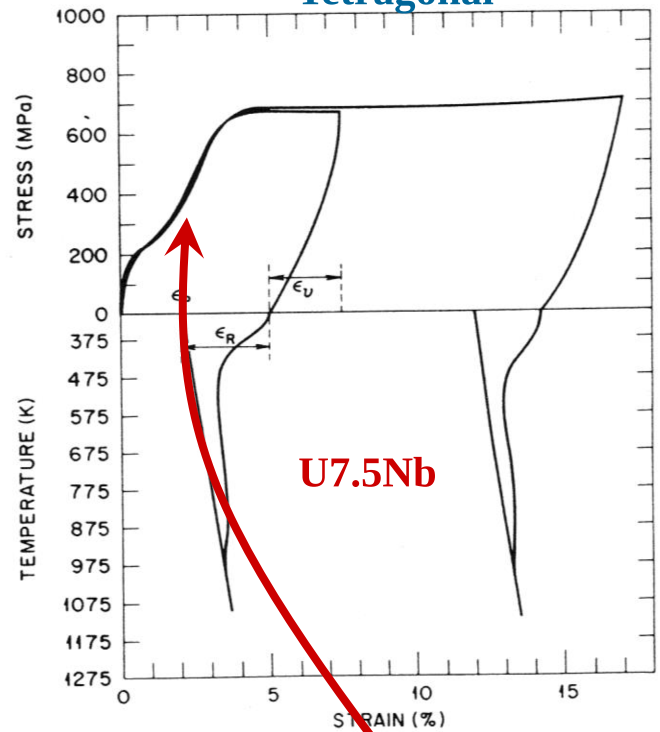
The UNb Alloys are Shape Memory Alloys

Vandermeer, Ogle, Northcutt, Met Trans A, Vol 12, 1981

Monoclinic



Tetragonal



De-twinning of martensitic variants

Stress induced phase transformation.

D.W. Brown, M.A.M. Bourke, P.S. Dunn, R.D. Field, M.G. Stout, D.J. Thoma, Met. Trans. A, 32 (2001) 2219-2228.

D.W. Brown, M.A.M. Bourke, R.D. Field, W.L. Hults, D.F. Teter, D.J. Thoma, S.C. Vogel, Mat Sci Eng A, 421 (2006) 15-21.

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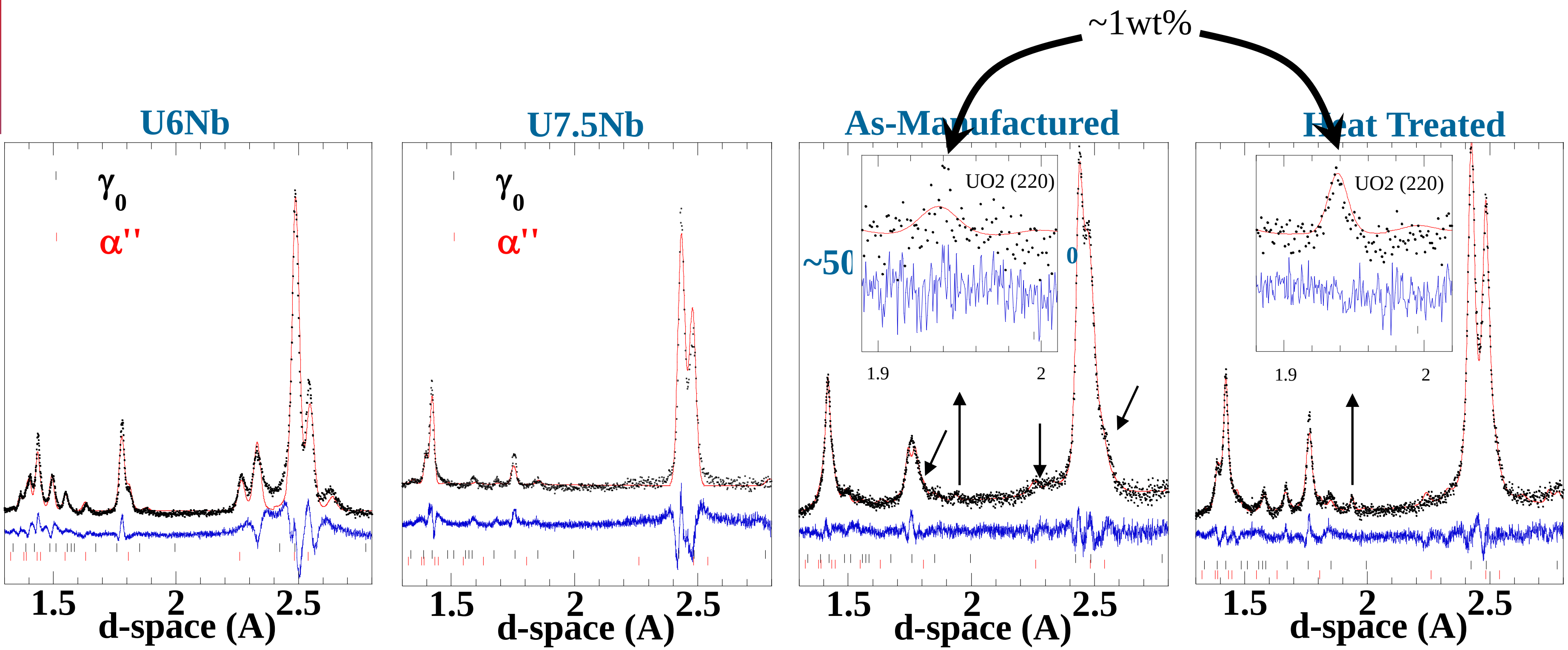
Structure of AM'ed U6Nb Differs From That Conventionally Produced



Conventionally Produced Material

Additively Manufactured Material

- Something in the microstructure is stabilizing the γ_0 phase. It is enhanced after heat treating.
  - Other metal impurities?
  - Interstitial oxygen?
  - Oxygen binding with U, effectively increasing Nb concentration?

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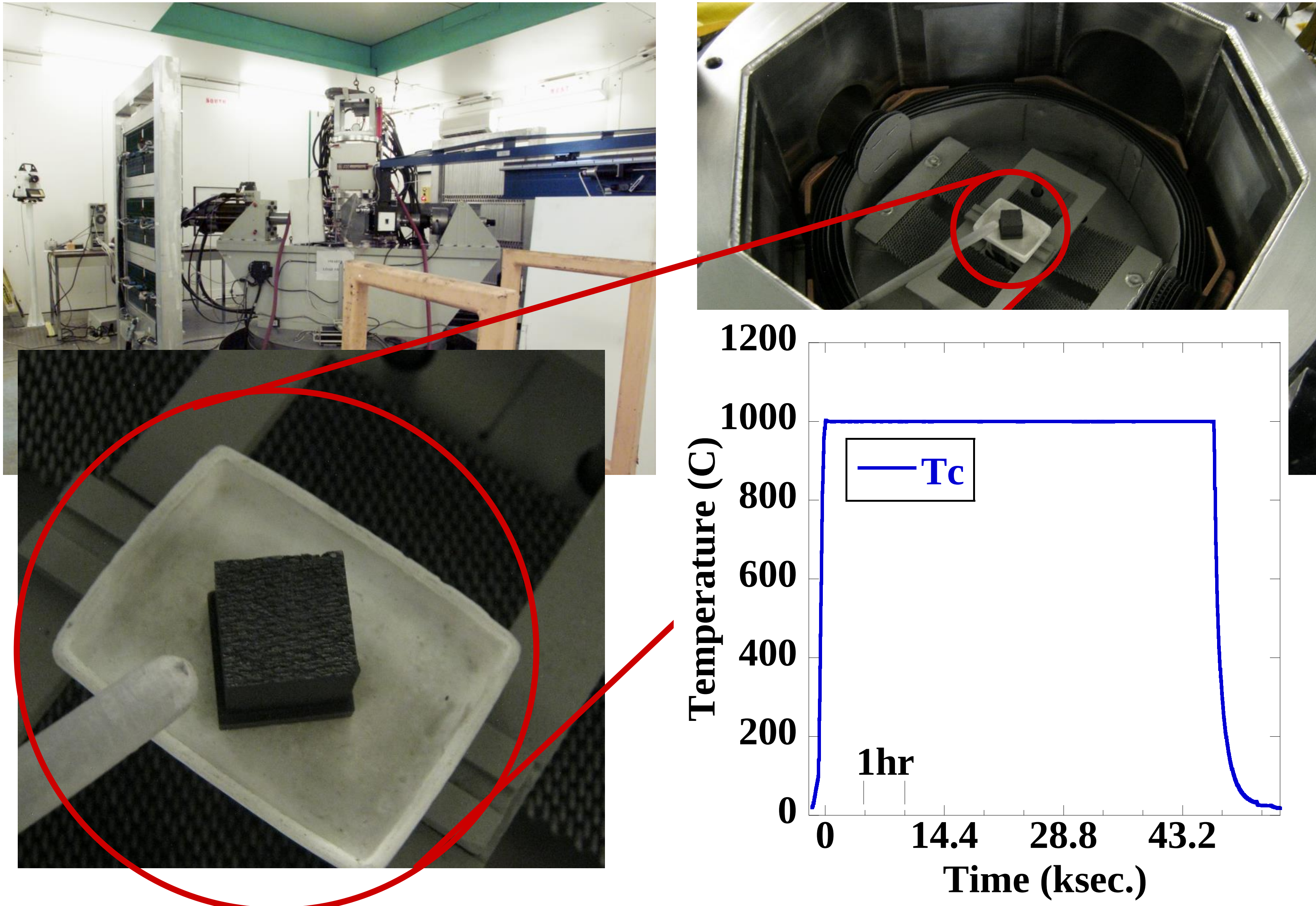
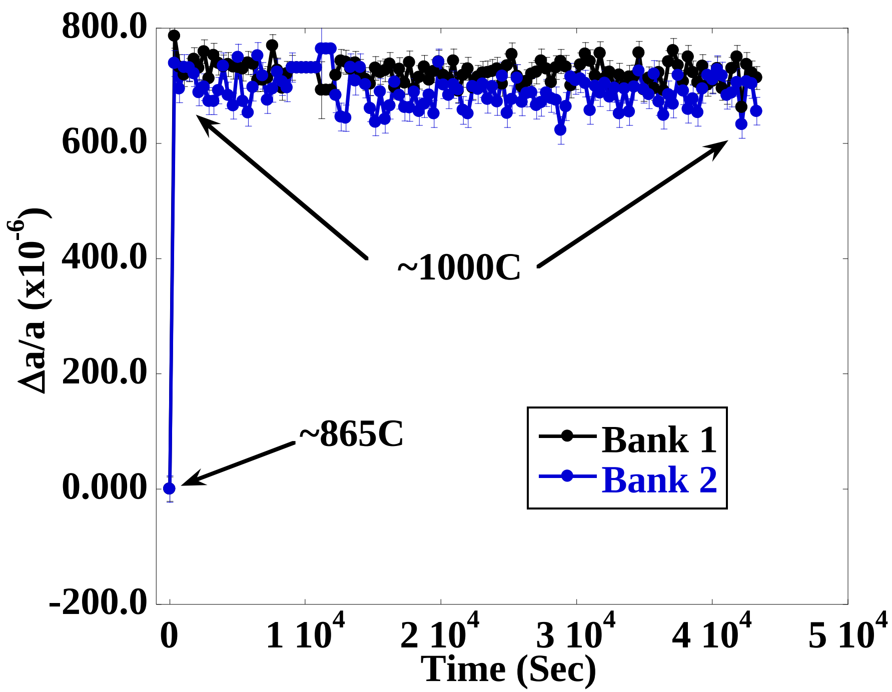
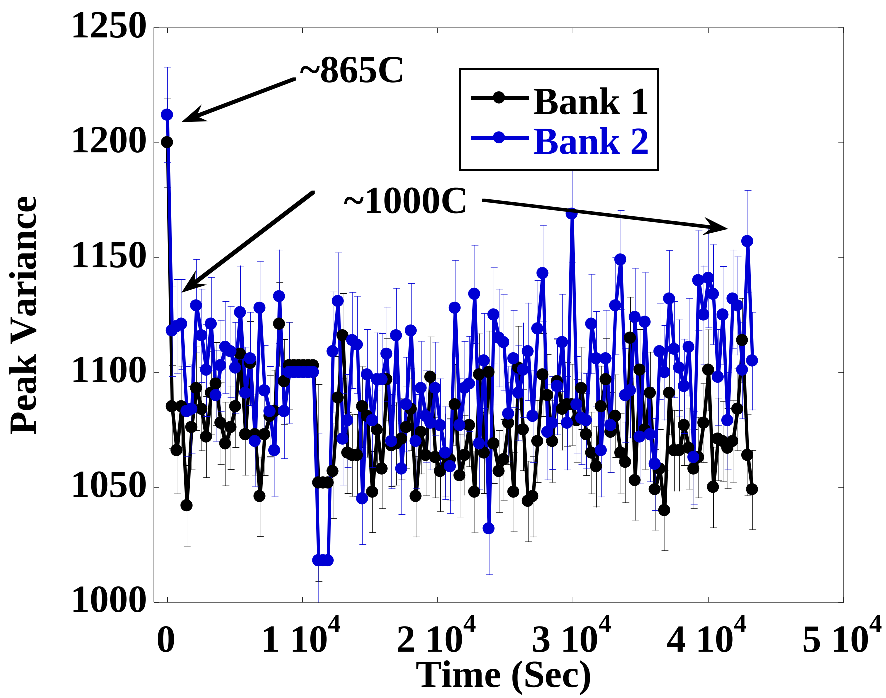
Heat Treating of U6Nb Completed In-Situ on SMARTS



How Does the Microstructure Evolve During Heat Treatment?





- Everything we can monitor, chemistry, stress, texture and dislocation density is constant during hold at 1000C.
- 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.

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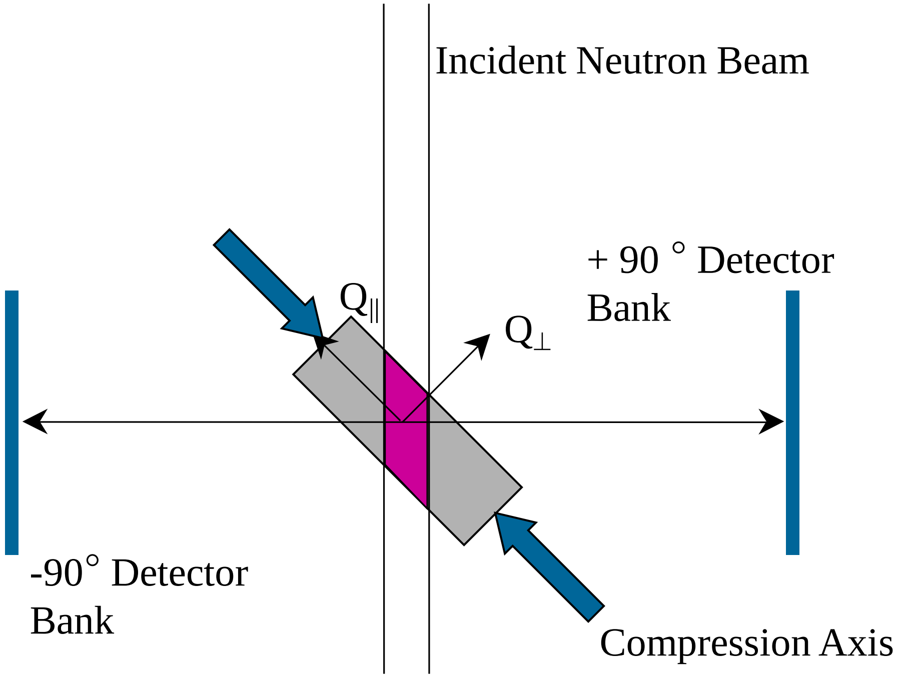
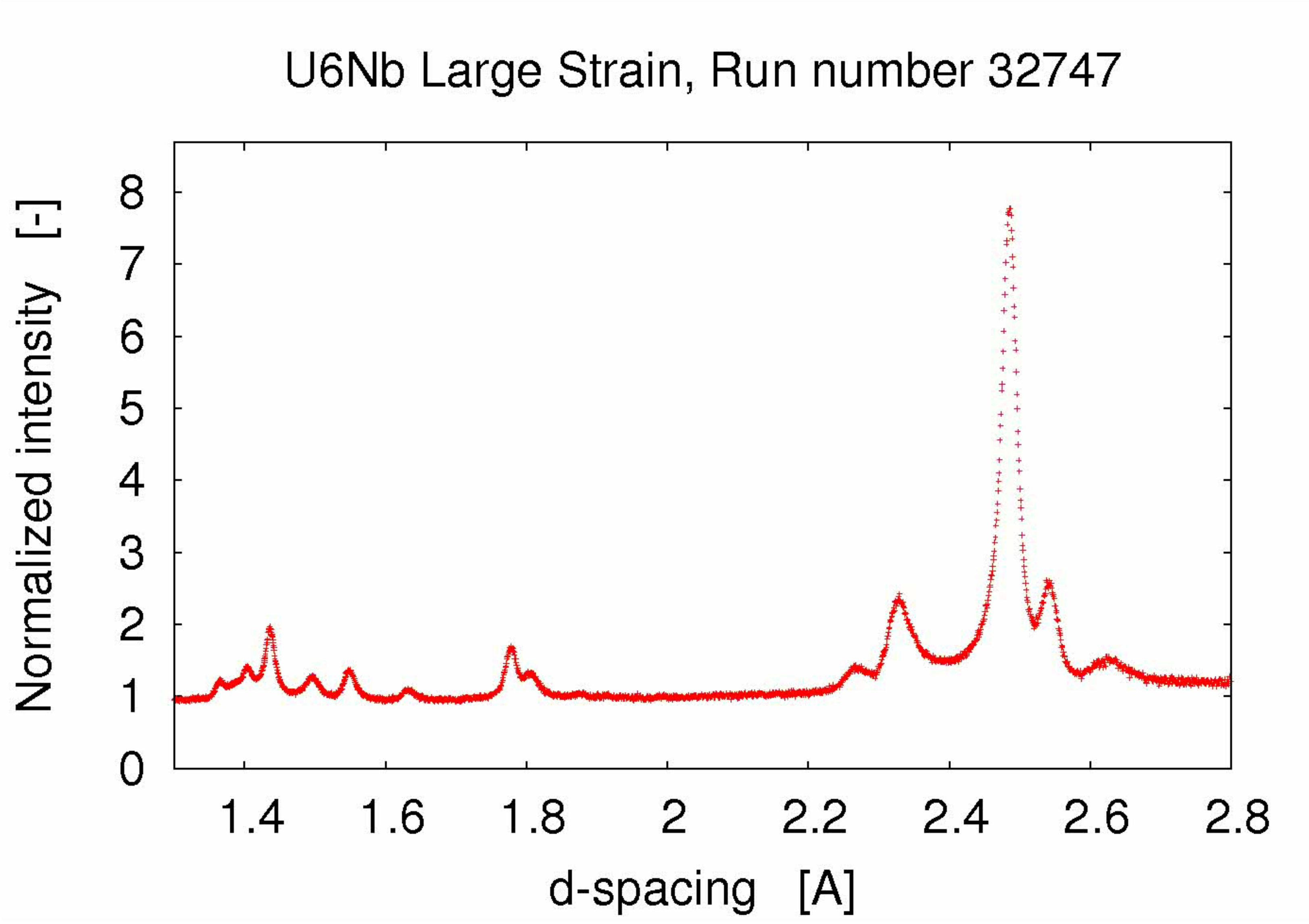
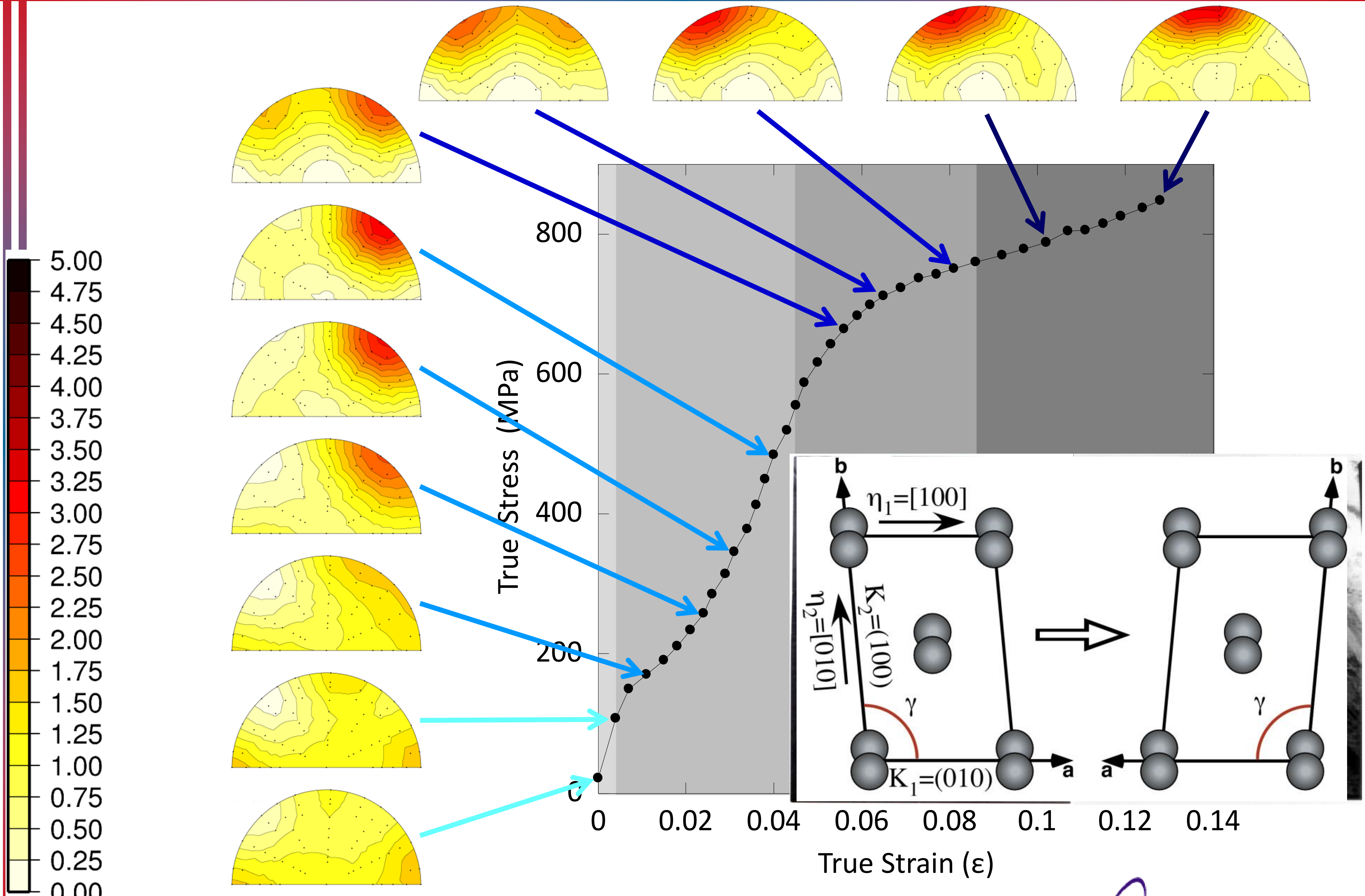
We Performed Deformation In-Situ to Monitor the Microstructure



Development of U6Nb Diffraction Pattern During Tensile Deformation



Traditional U6Nb Deforms By Multiple Mechanisms



C.N. Tupper, D.W. Brown, R.D. Field, T.A. Sisneros, B. Clausen, Met. Trans. A, 43A (2012) 520-530.

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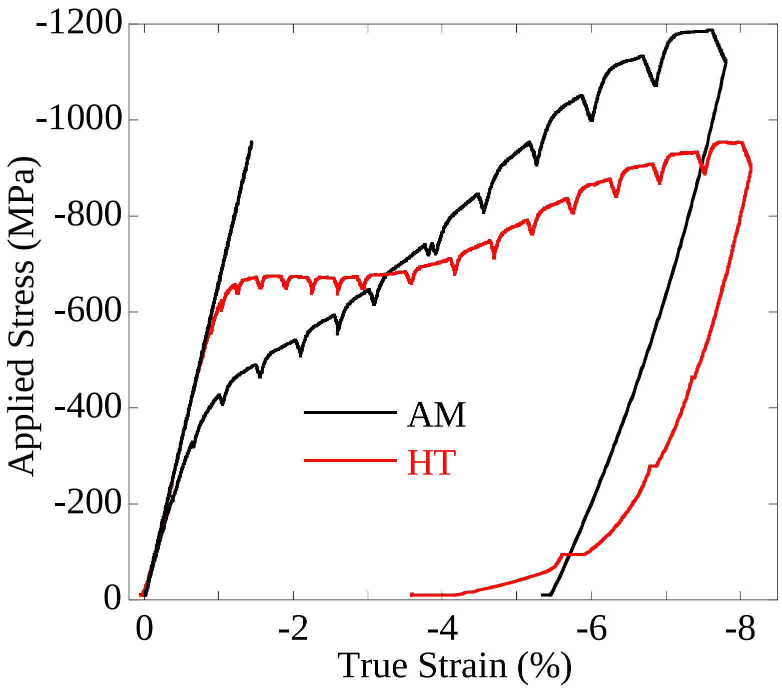
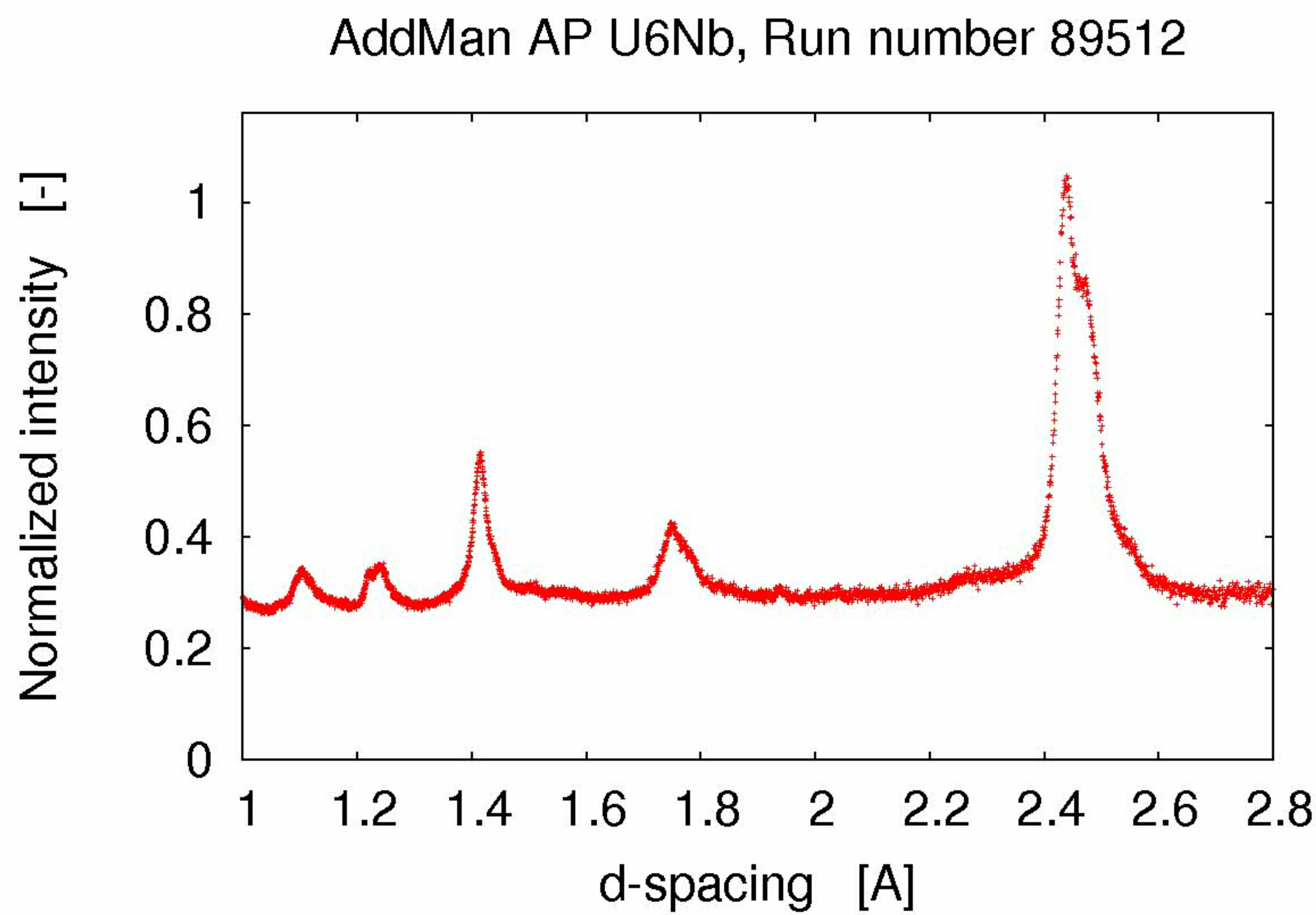
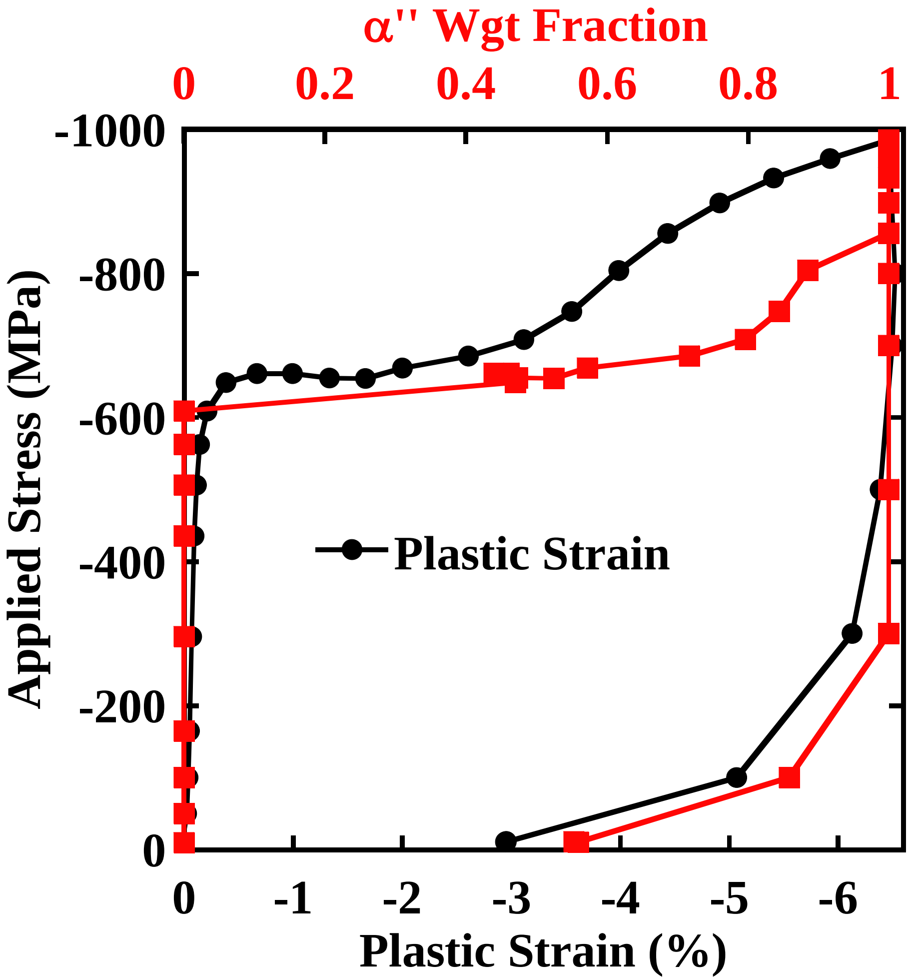
AM'ed U6Nb Also Has Sigmoidal Flow Curve



Evolution of Diffraction Pattern is Distinct From Conventional U6Nb



Plastic Deformation Accommodated by Stress Induced Phase Transformation



Different initial phase excludes the 2 primary deformation modes of conventional U6Nb.

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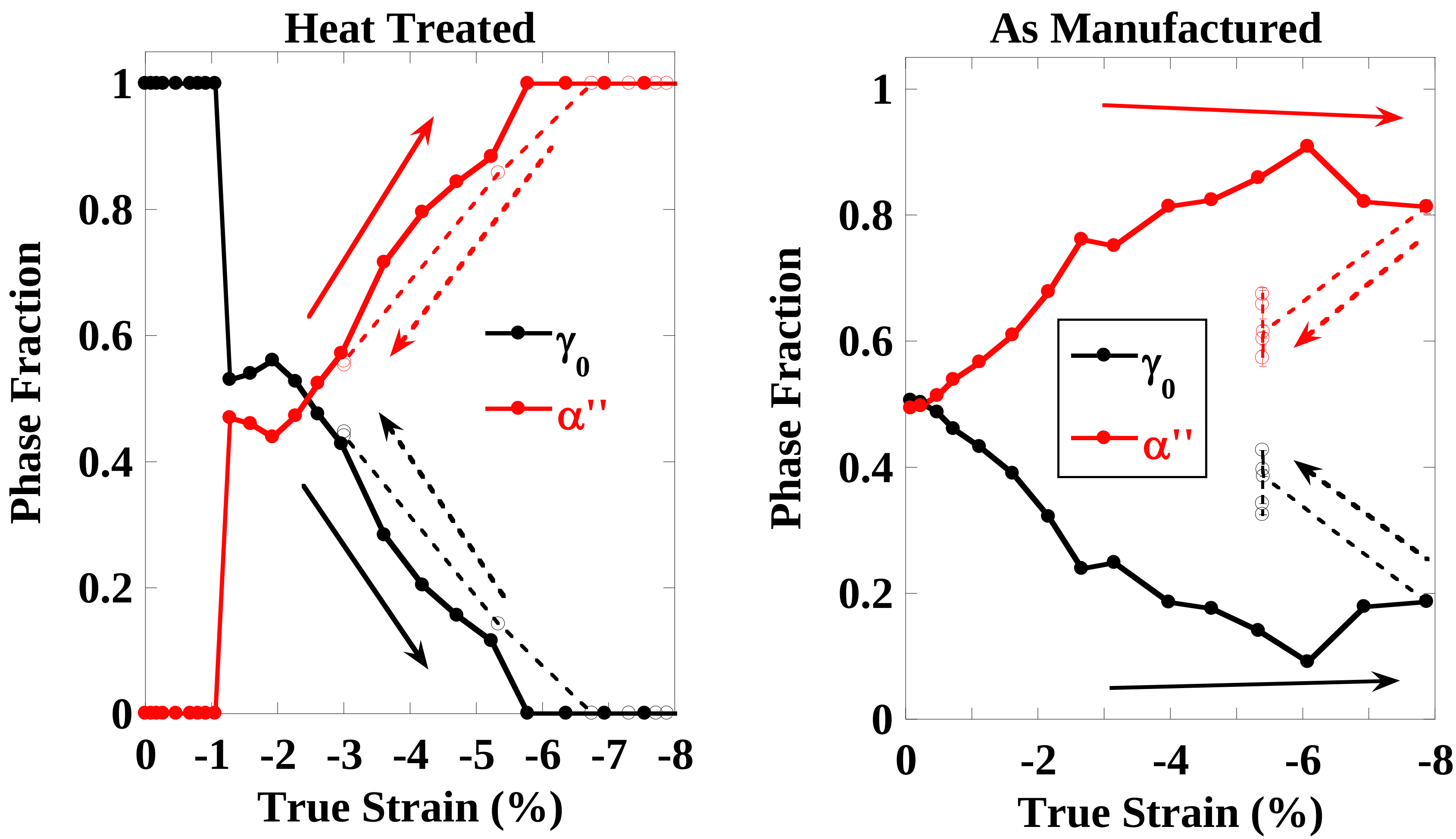
Phase Evolution is Quantified by Diffraction



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 γ_0.
    - Heat treated: γ_0.
  - 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.

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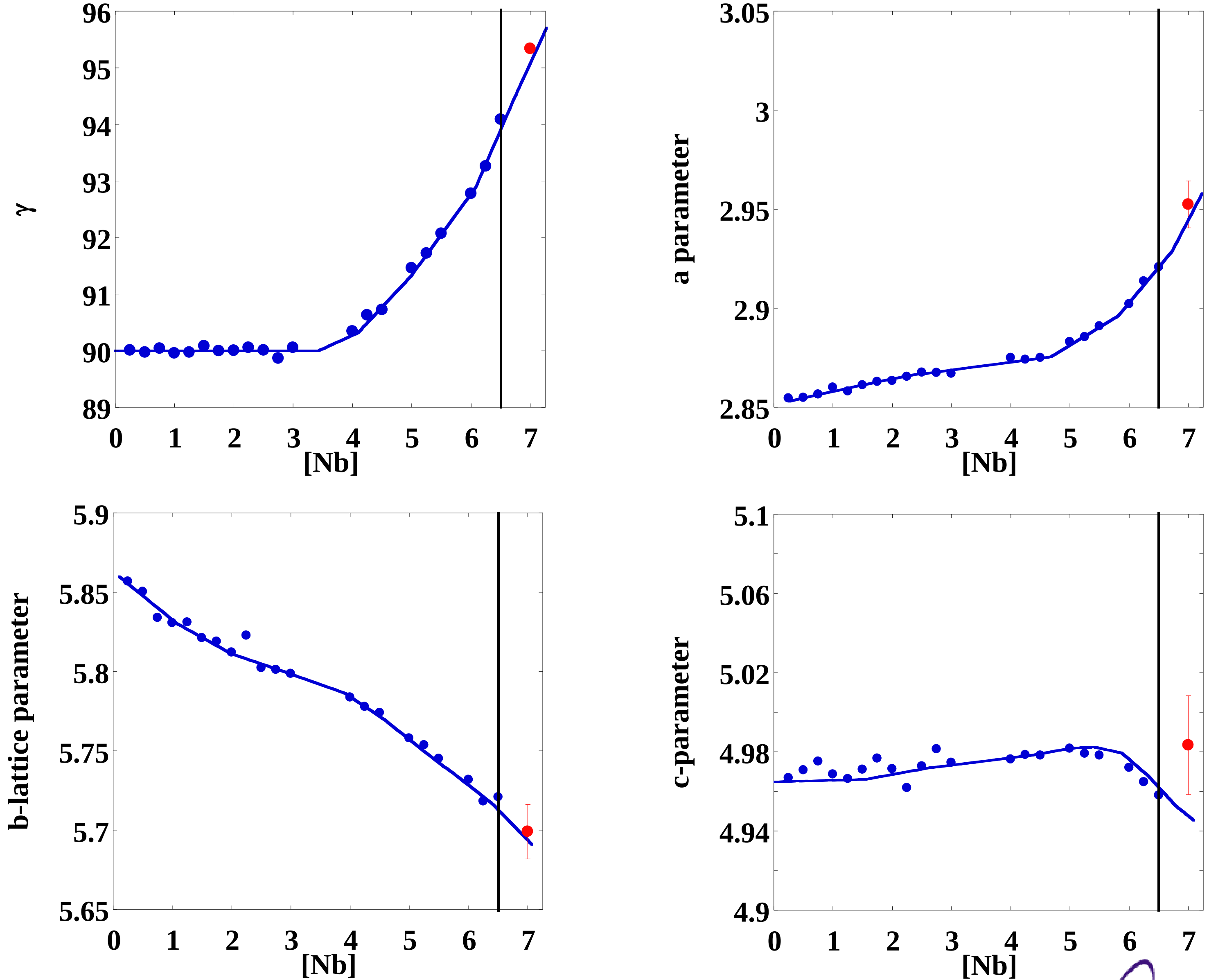
Variation of Lattice Parameter With [Nb]



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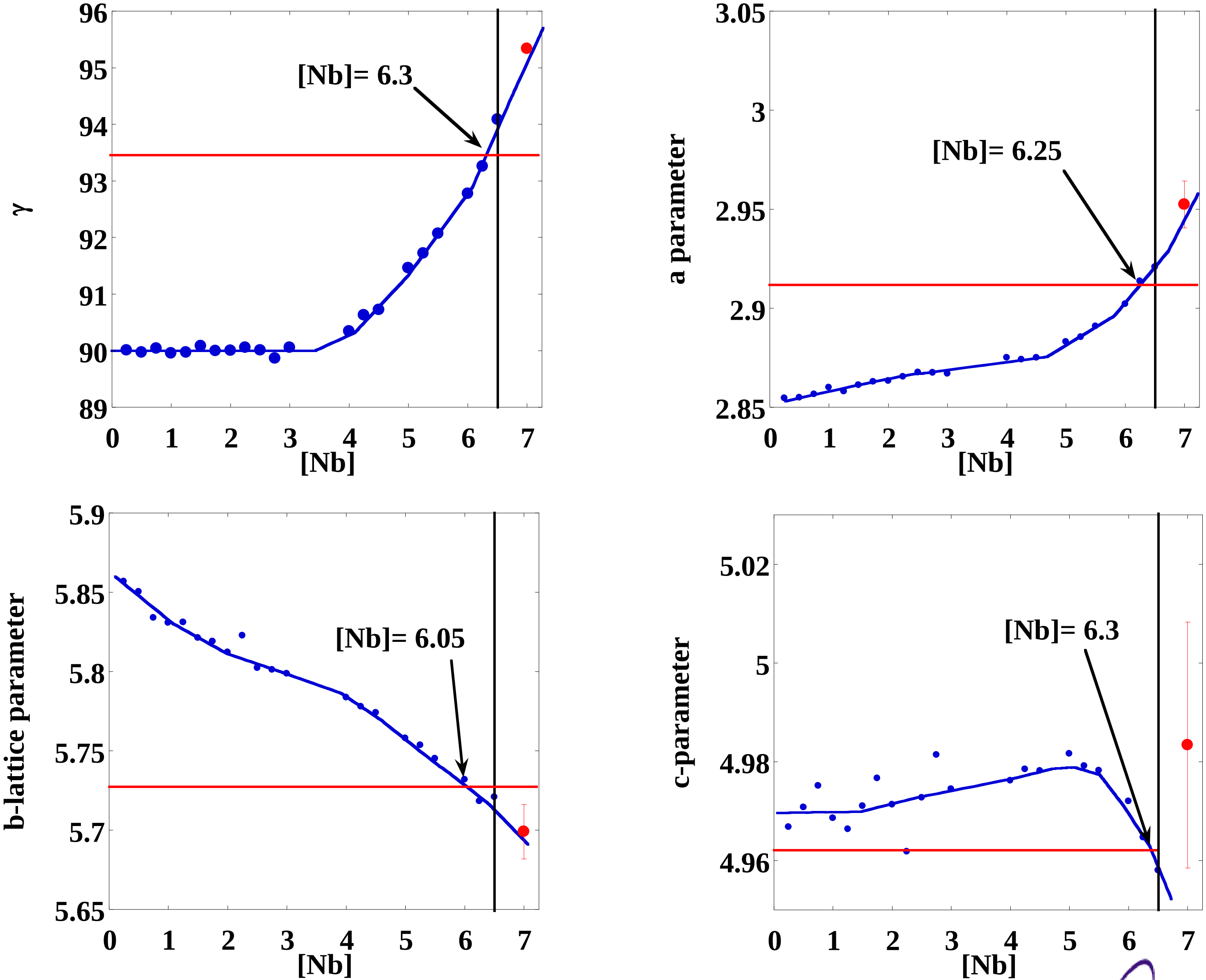
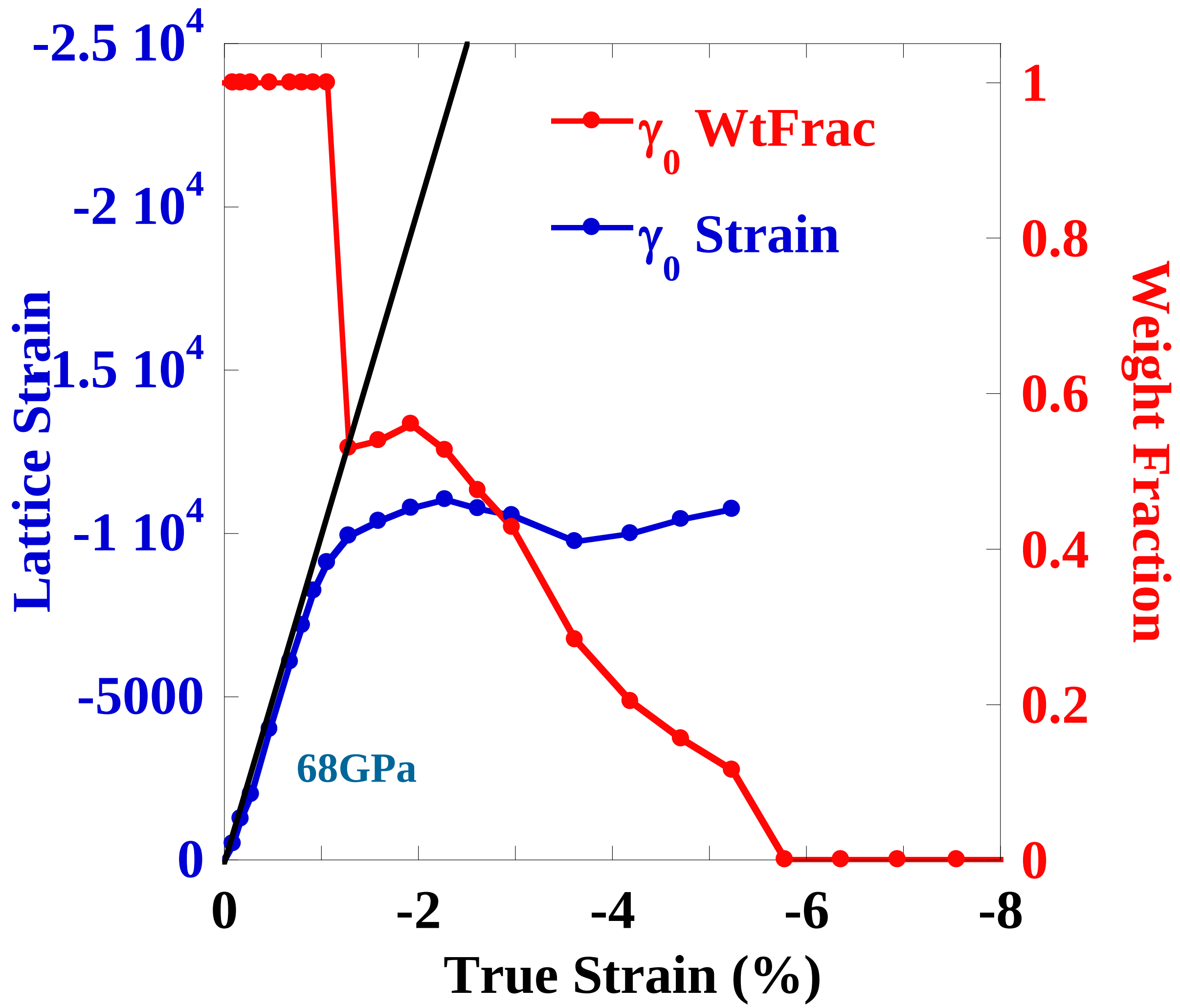
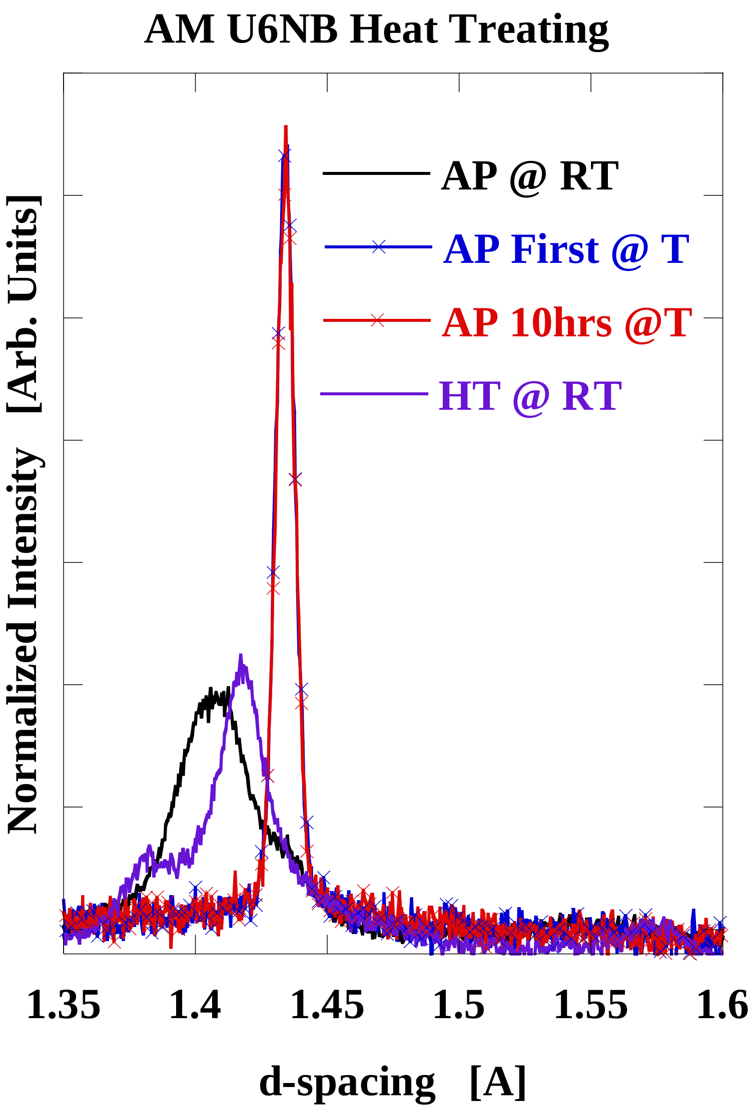
Stress Induced α´´ Looks Like U6Nb

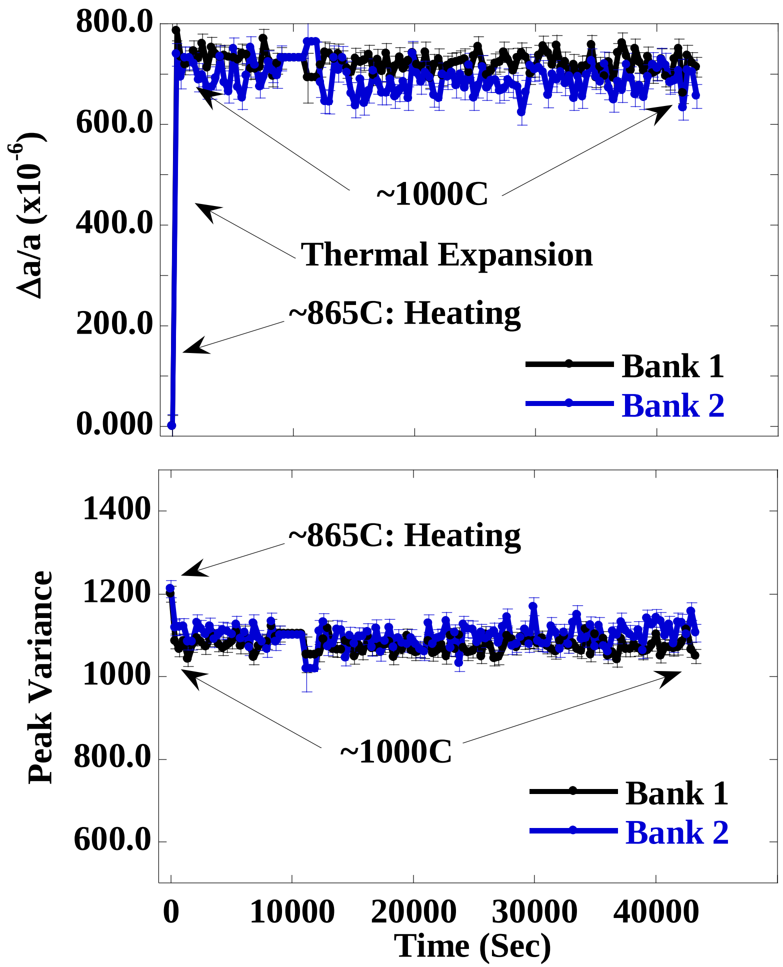


Deformation of AM Material is Reminiscent of Y12 Material



$$\varepsilon = \varepsilon_p + \varepsilon_e$$





- 100C/min to 1000C.
- 10 hours at 1000C.
- Oil Quench

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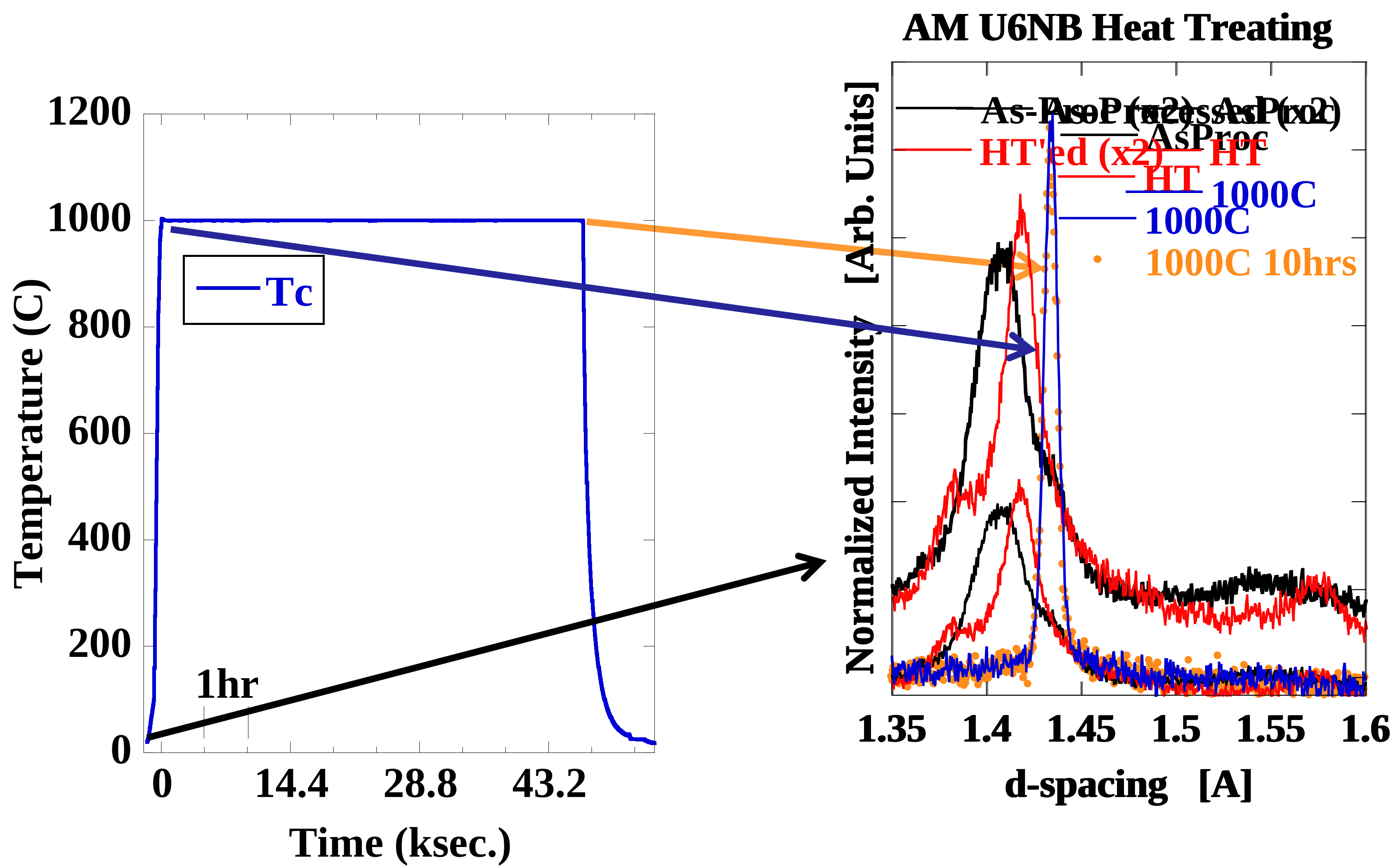
How Does the Microstructure Evolve During Heat Treatment?



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