Heating Rate Dependent Microstructural Evolution of Worked Titanium Alloys: AKA How to Bring Your Windows Laptop to Its Knees.

[1]Brown, D.W., [1]Carver, D. T.,[1]Savage, D.J., [1]Clausen, B., [2]Strantza, M. [3]Zepeda-Alarcon,, [5]Pagan, D., [5]Shanks, K.

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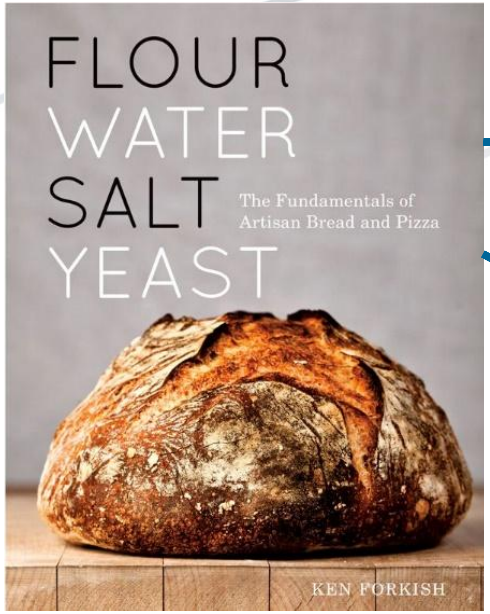
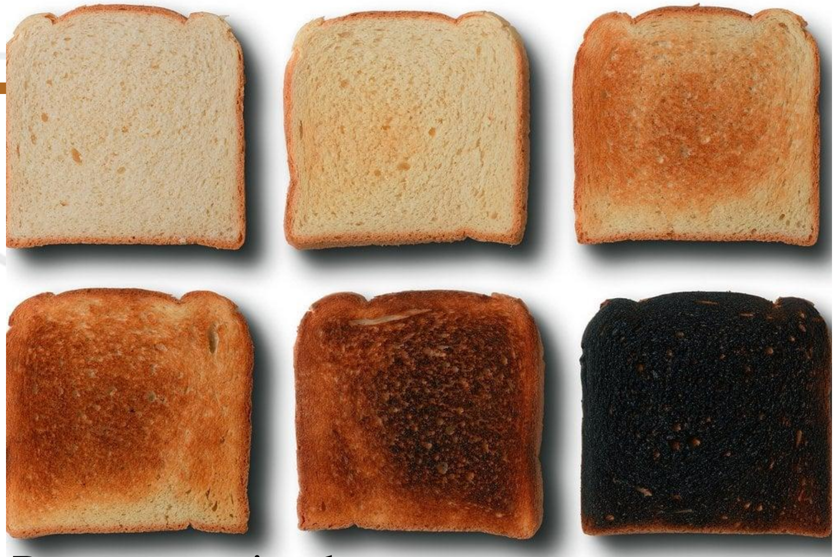
Microstructure Matters!



Highly
Recommended

Process

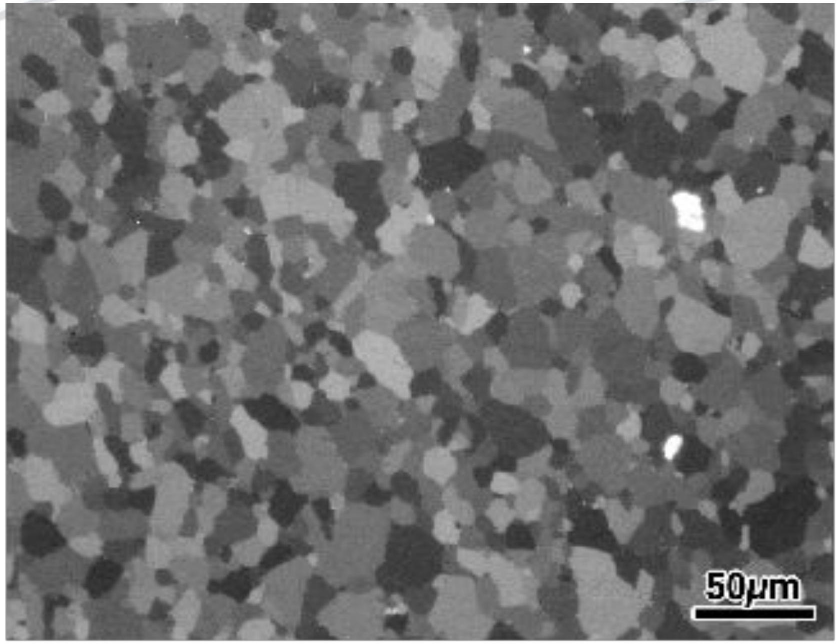
The ingredients are the same, the Properties are different. These are set by the Processing.



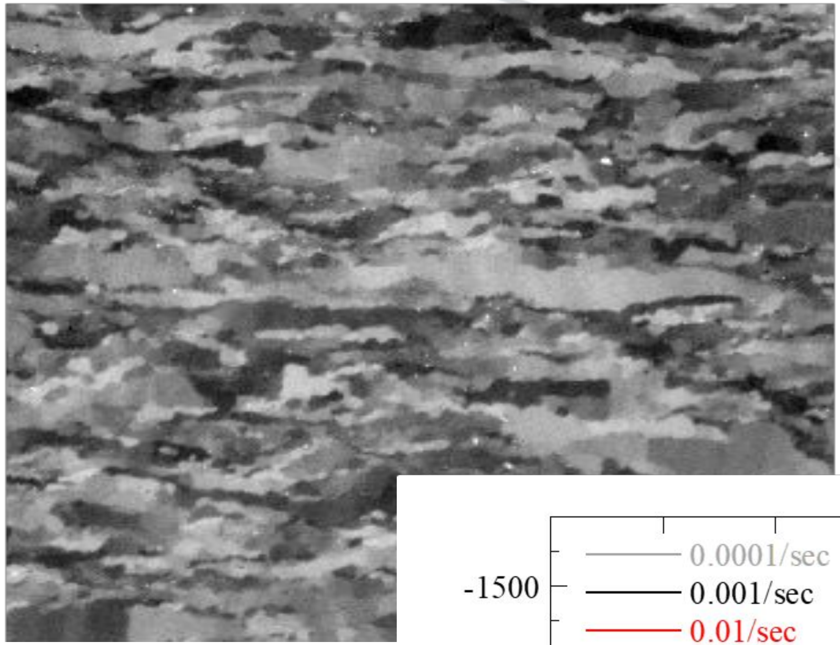
Post processing heat treatment

Aging

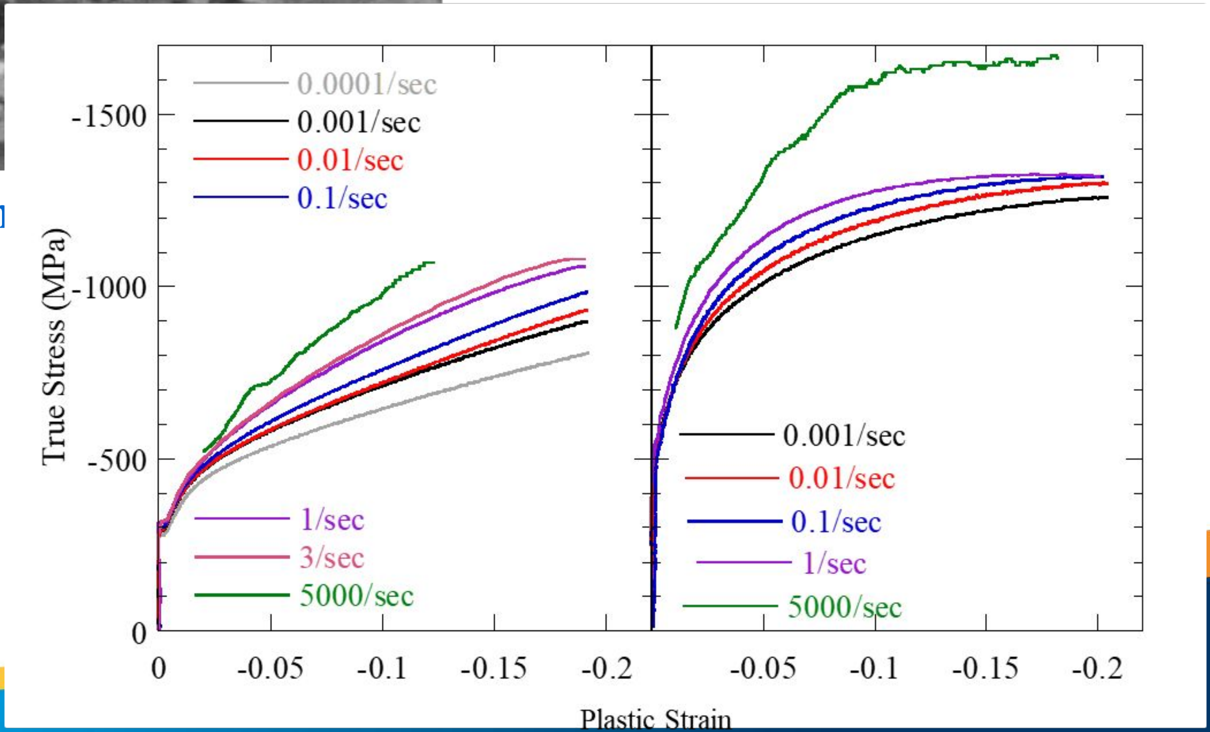
In Materials, We Call This The Process/Structure/Property Relationship.



Hot-Pressed Beryllium

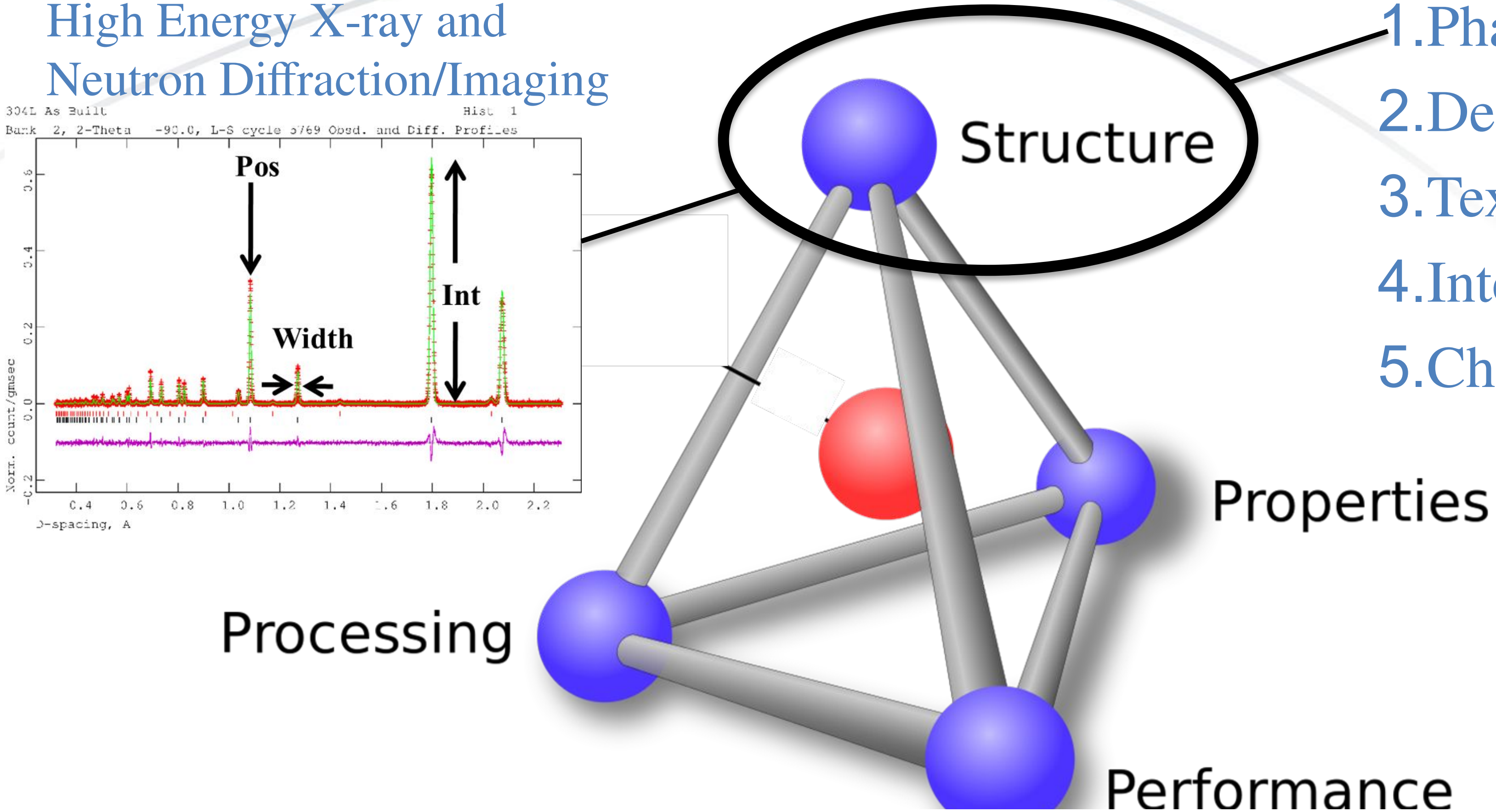


Rolled (wrough



The Goal of My Team is to Provide Experimental Support of Model Development Across the PSPP Relationship

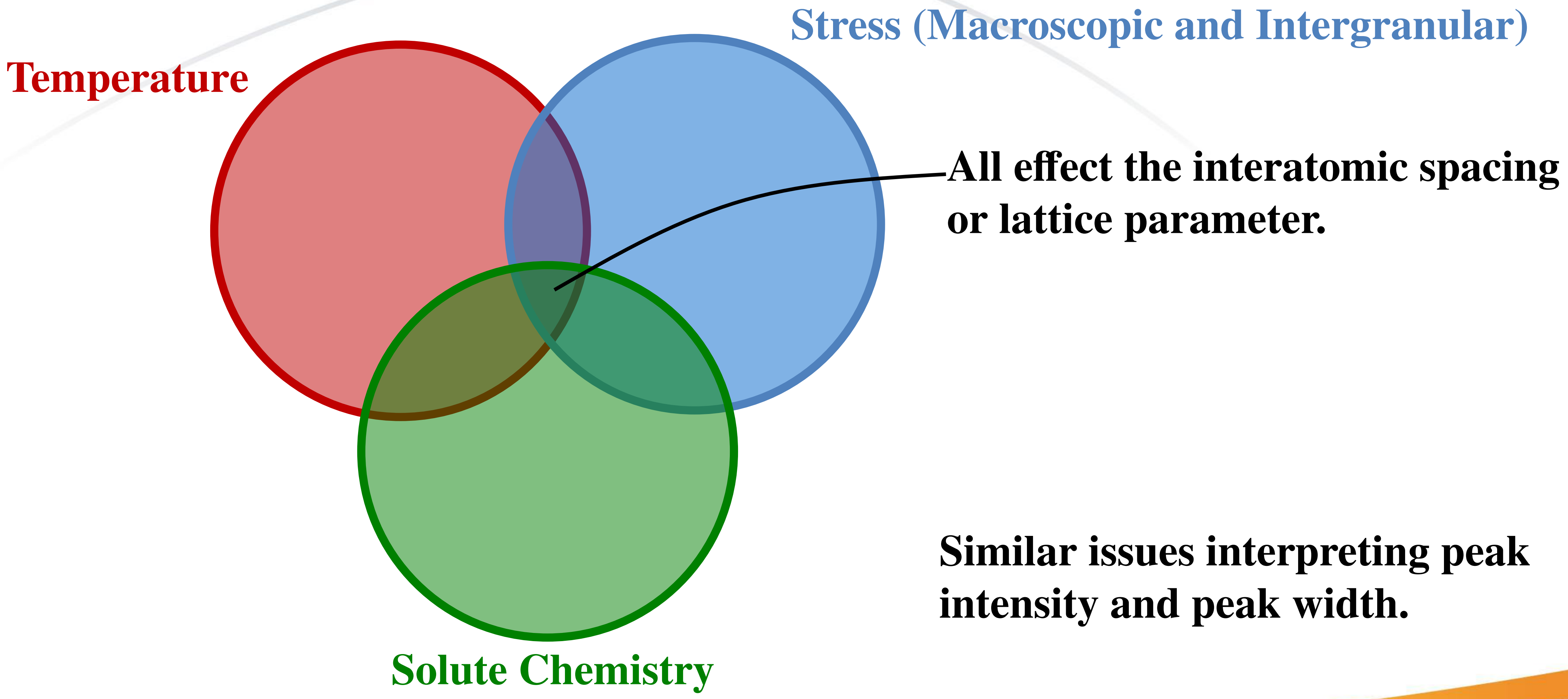
High Energy X-ray and Neutron Diffraction/Imaging



1. Phase
2. Defects
3. Texture
4. Internal Stress
5. Chemistry

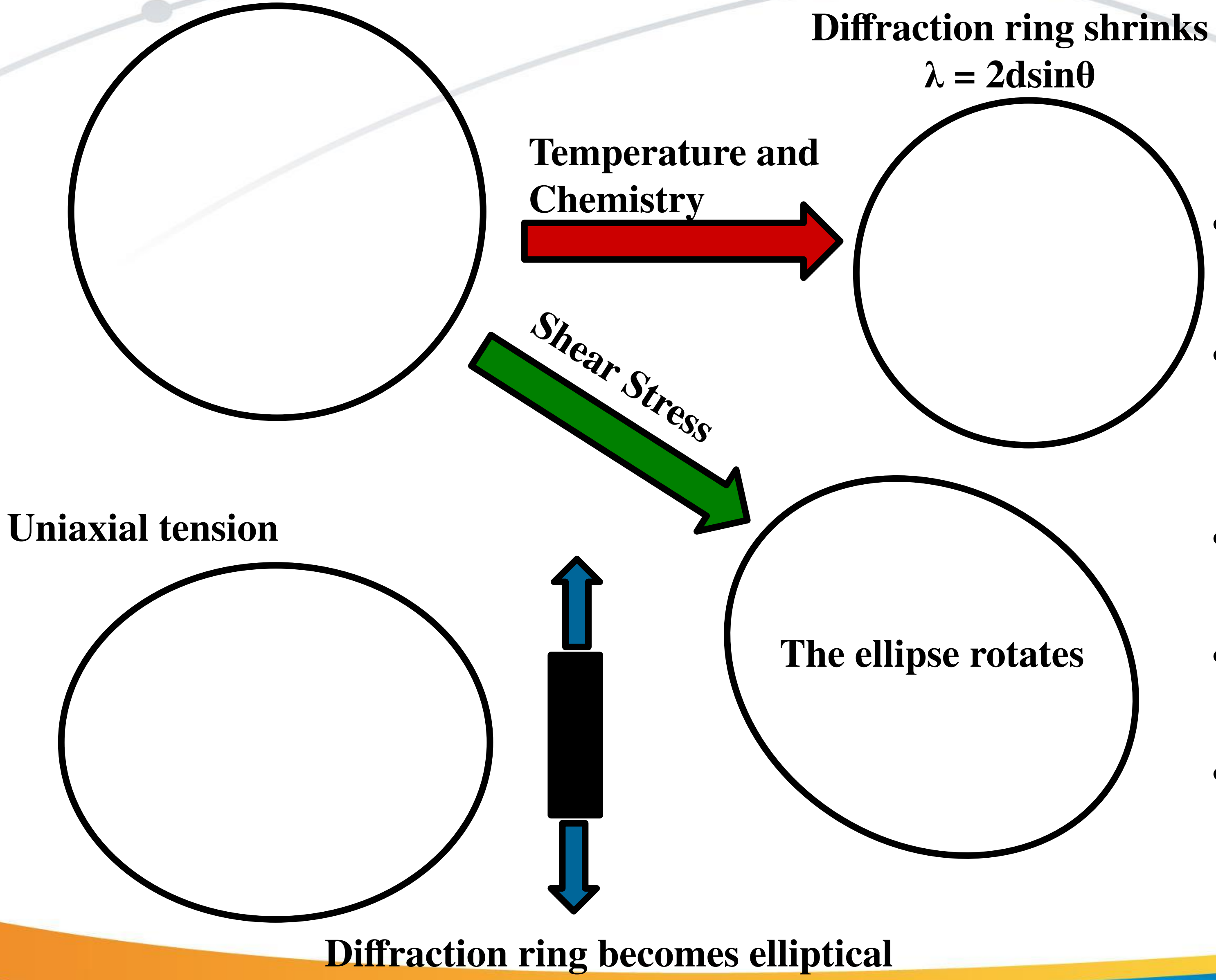
- **Our mission is to provide quantitative microstructural features under relevant conditions.**
  - **For the development and validate of predictive process/performance models.**
  - **Models and data must bridge length and time scales.**

It is Difficult to De-Convolute Multiple Microstructural Features on Parameters Derived From Diffraction Patterns.



Similar issues interpreting peak intensity and peak width.

Need to be Quantitative Places Demands on Detectors



- Δa/a~0.000100
  - Sets pixel size.
- Necessary to measure full diffraction rings to separate temperature/chemistry from stress.
  - Big detector.
- Macro and intergranular stress separated by measuring multiple rings.
- External knowledge necessary to separate temperature from solute chemistry.
- Similar issues to separate texture/phase/DW.

Ti-6Al-4V (Ti64) is a Popular Materials for Additive Manufacture (Powder Bed Fusion)

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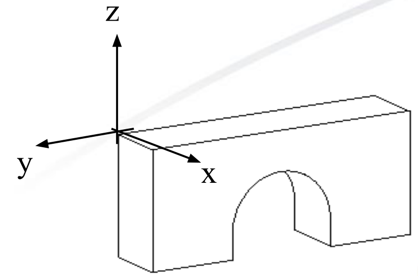
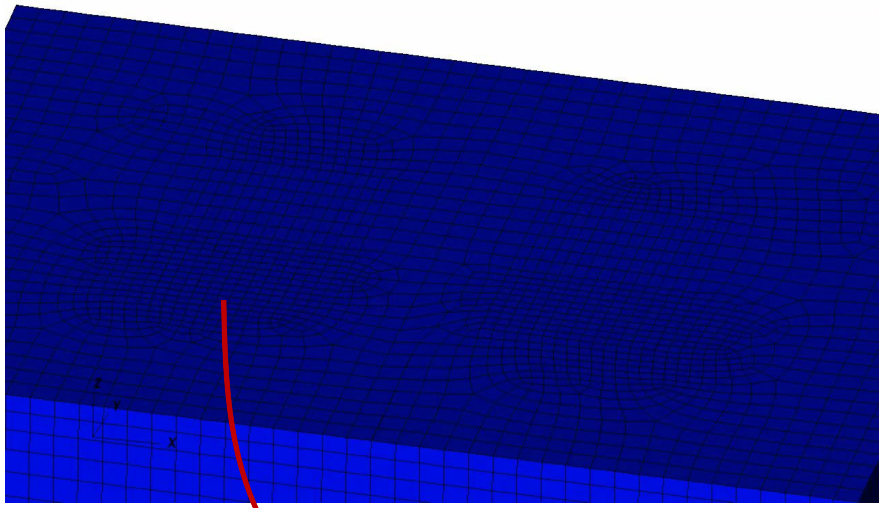
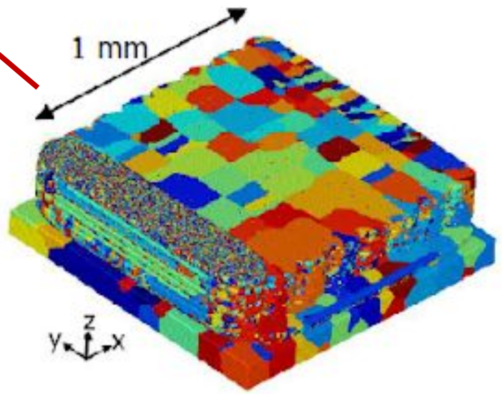
Heavily utilized in aerospace industry, expensive, large buy/fly ratio.



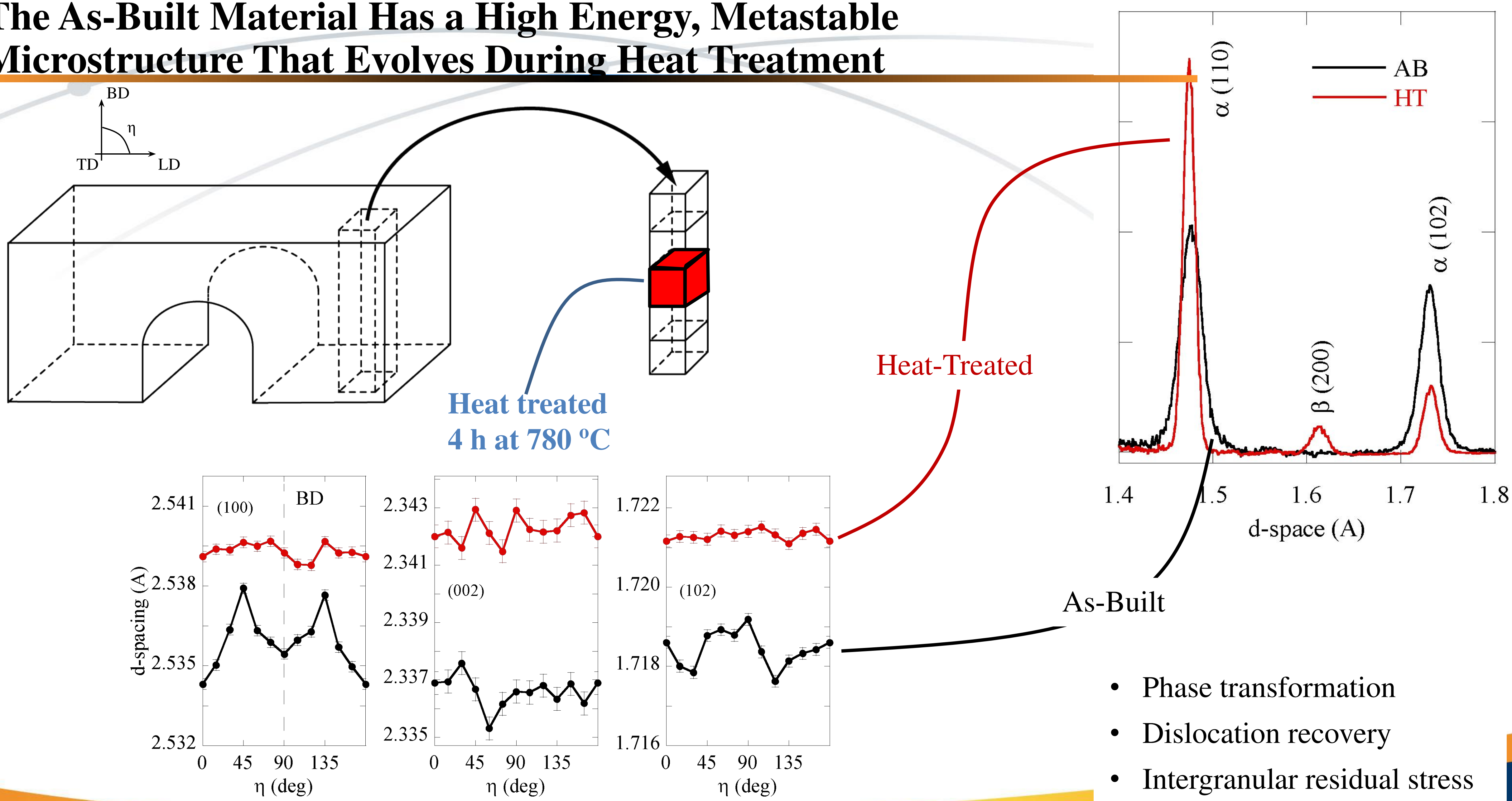




- A1: continuous scan, aligned with x-axis, 90 degree rotation between layers
- A2: continuous scan, at 45 degree to x-axis, 90 degree rotation between layers
- B1: island scan, 5mm by 5mm islands, aligned with x-axis, 90 degree rotation between layers
- B2: island scan, 5mm by 5mm islands, at 45 degree to x-axis, 90 degree rotation between layers

[1] M. Strantza, R.K. Ganeriwala, B. Clausen, T.Q. Phan, L.E. Levine, D. Pagan, W.E. King, N.E. Hodge, D.W. Brown, *Mat. Lett.*, 2018, vol. 231, pp. 221-224.
[2] R.K. Ganeriwala, M. Strantza, W.E. King, B. Clausen, T.Q. Phan, L.E. Levine, D.W. Brown, N.E. Hodge, *Additive Manufacturing*, 2019, vol. 27, pp. 489-502.
[3] M. Strantza, R.K. Ganeriwala, B. Clausen, T.Q. Phan, L.E. Levine, D.C. Pagan, J.P.C. Ruff, W.E. King, N.S. Johnson, R.M. Martinez, V. Anghel, G. Rafailov, D.W. Brown, *Additive Manufacturing*, 2021, vol. Accepted

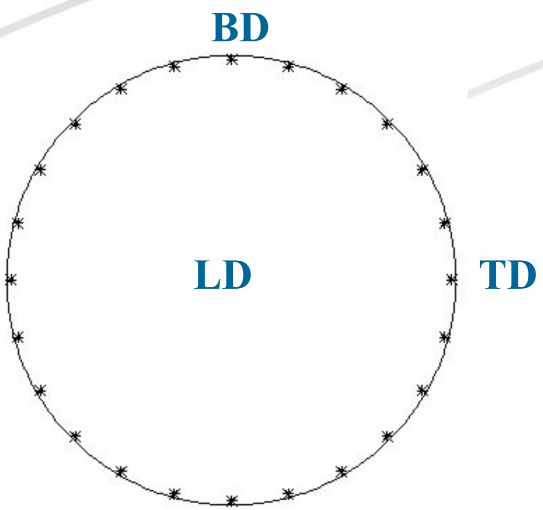
The As-Built Material Has a High Energy, Metastable Microstructure That Evolves During Heat Treatment

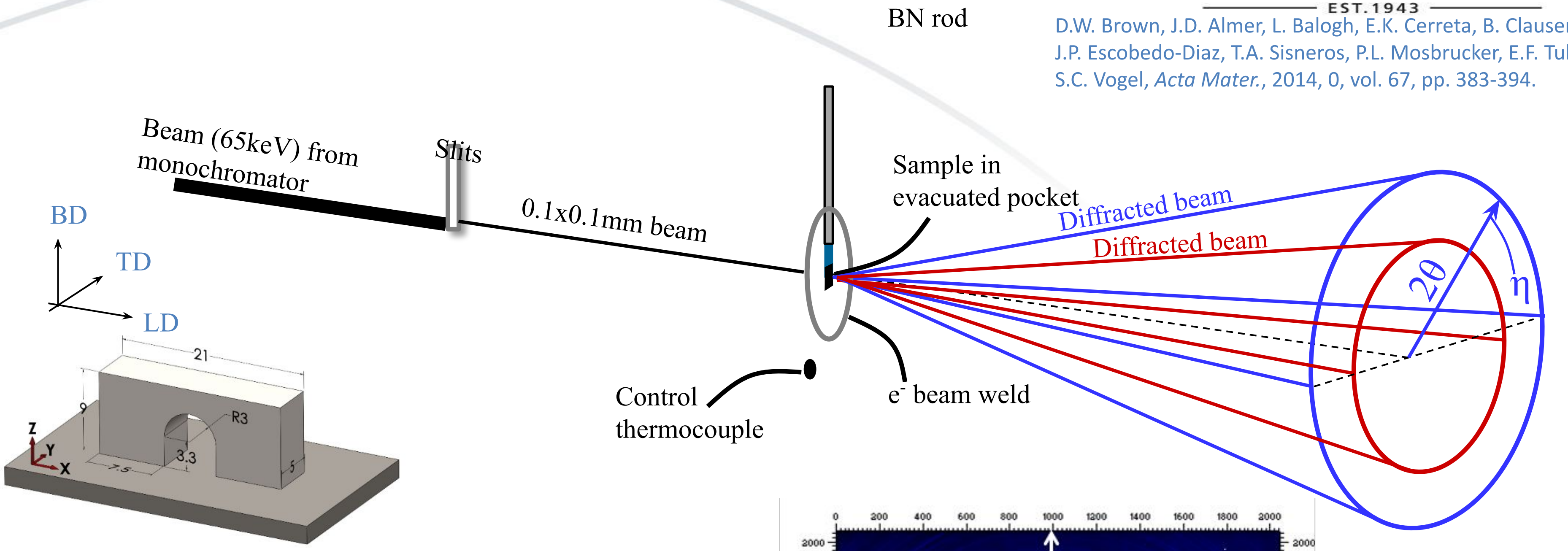


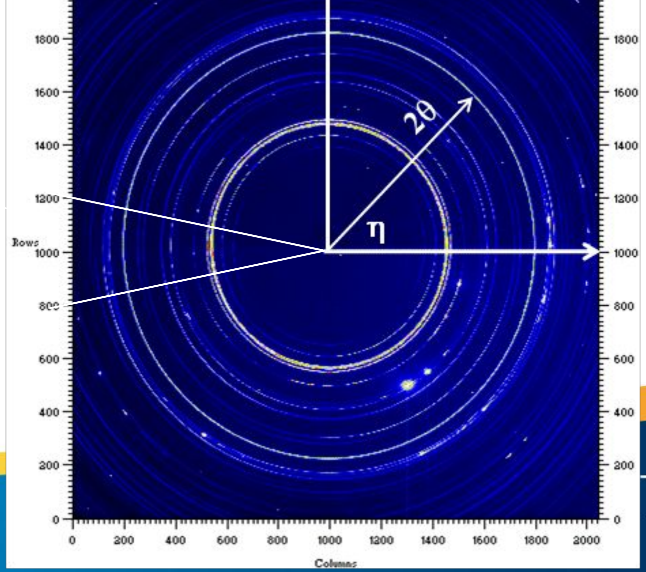
- Phase transformation
- Dislocation recovery
- Intergranular residual stress
- Solute chemistry distribution

Residual Stress Measurements Motivated In-Situ High Energy X-Ray Diffraction Measurements During Heat Treating

D.W. Brown, J.D. Almer, L. Balogh, E.K. Cerreta, B. Clausen, J.P. Escobedo-Diaz, T.A. Sisneros, P.L. Mosbrucker, E.F. Tulk, S.C. Vogel, *Acta Mater.*, 2014, 0, vol. 67, pp. 383-394.







- Debye cones provide diffraction vectors nearly normal to the incident beam.
- Integrate rings to get an average response.
- Bin (cake) the data over twenty-four 15deg intervals to provide orientation dependence.
- Lattice parameter of container used to determine temperature.

D. W. Brown, V. Anghel, L. Balogh, B. Clausen, N. S. Johnson, R. M. Martinez, D. C. Pagan, G. Rafailov, L. Ravkov, M. Strantza, and E. Zepeda-Alarcon: Metall. Mater. Trans. A 2021, pp. 5165-81.

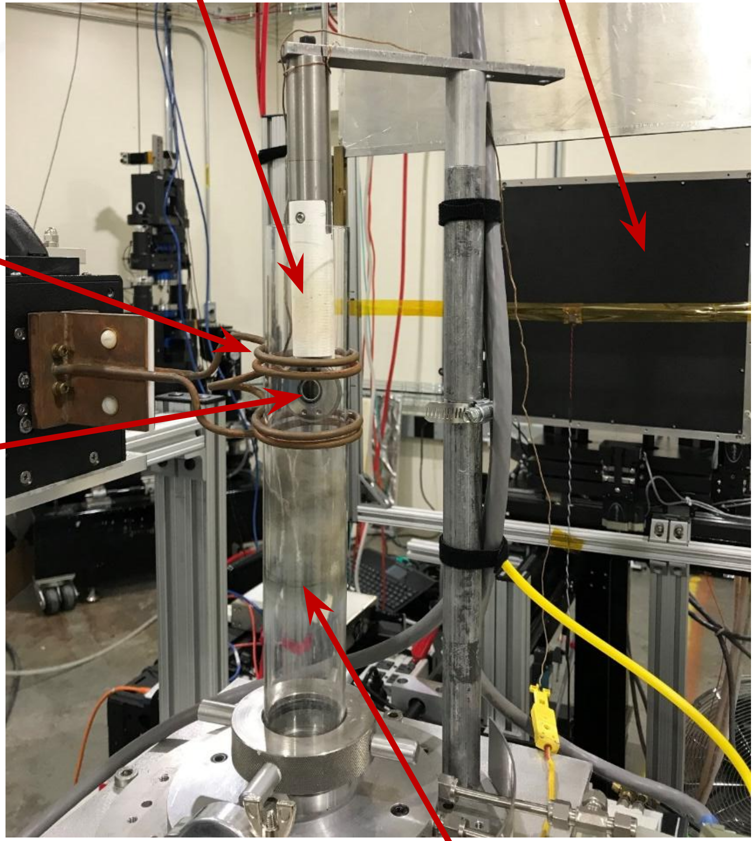
This is What it Looks Like For Real

Detector : 2000's technology, 8Hz (200 images), 14 bit pixel depth

BN Isolating Rod

RF Coils

Sample

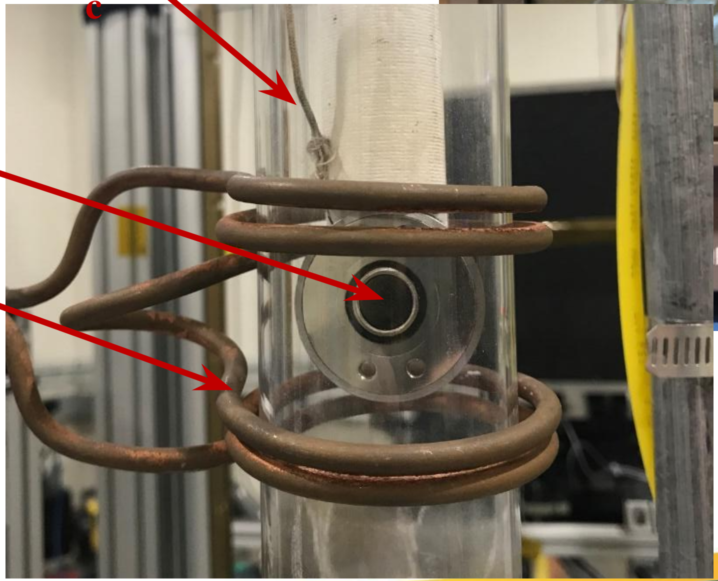


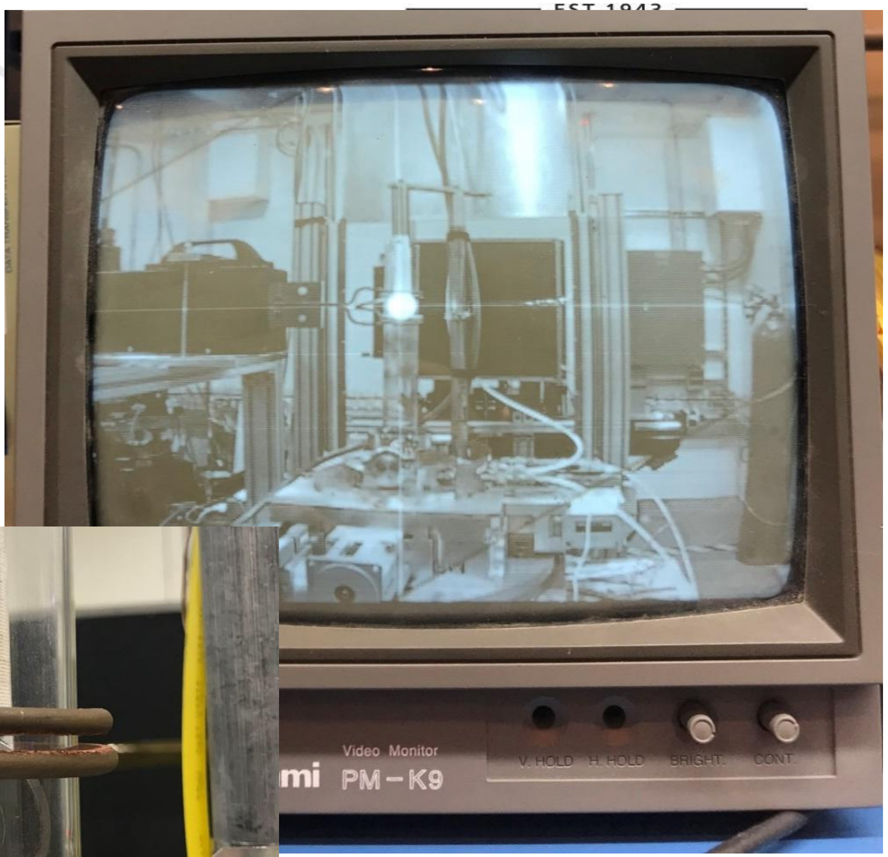
Flowing Argon

T c

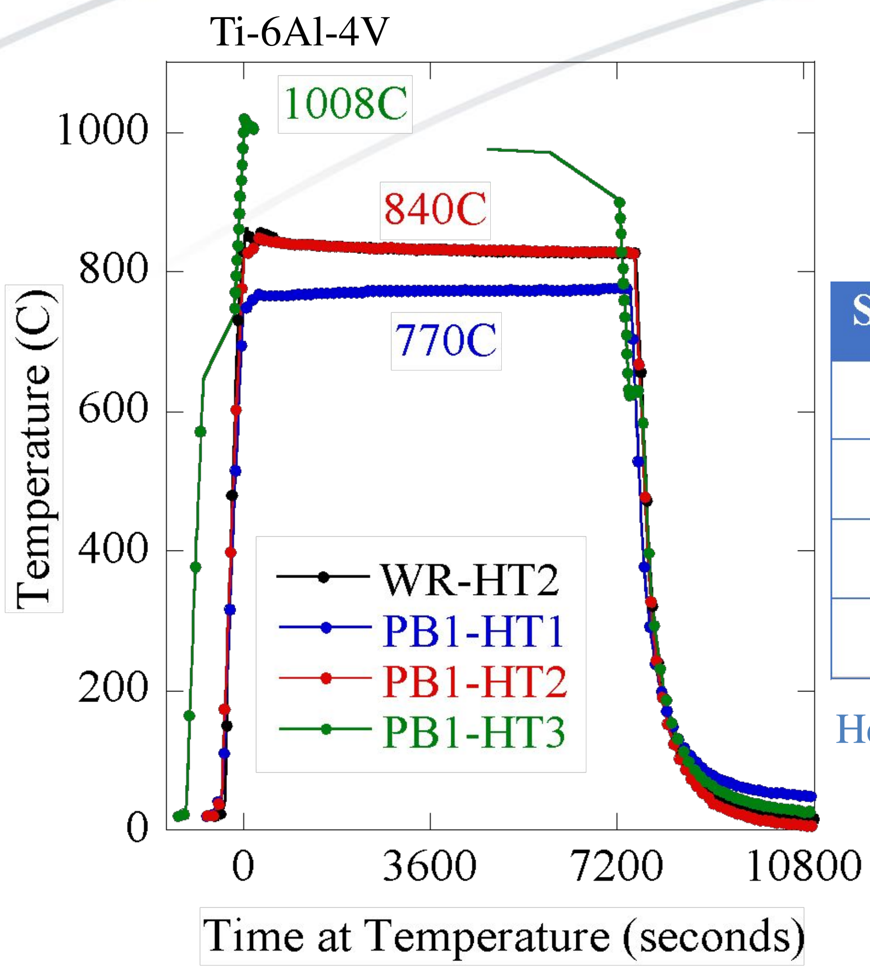
Sample

RF Coils





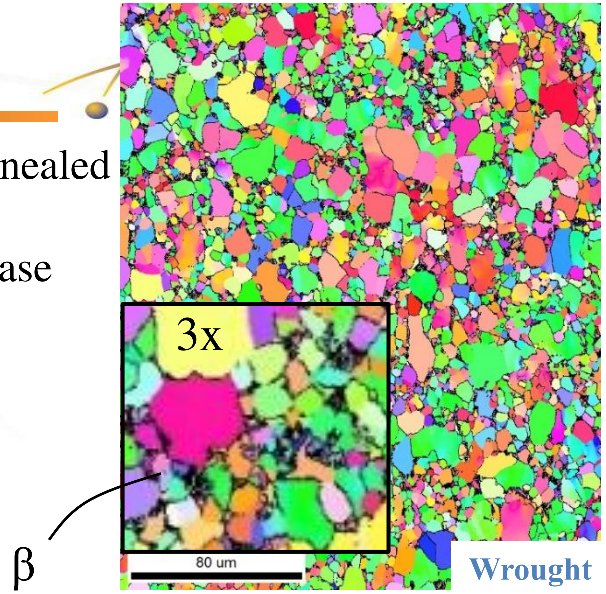
Our First Experiments Focused on Industrial Heat Treatment Processes

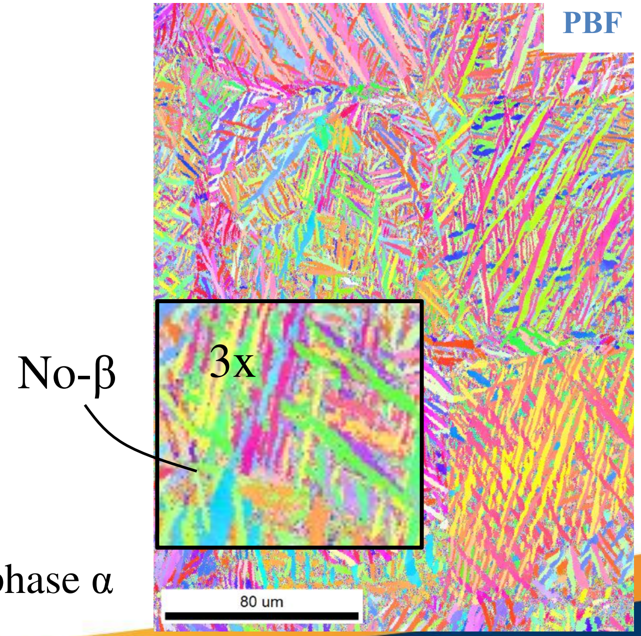


Sample Name	Material	T(C)
WR-HT2	Wrought	840
PB1-HT1	PBF	770
PB1-HT2	PBF	840
PB1-HT3	PBF	1008

Heating and cooling rate 100 C/min

- Wrought Material: Hot-rolled and annealed followed by an air cool.
- Equilibrium microstructure, 6% β phase
  V mostly in β.

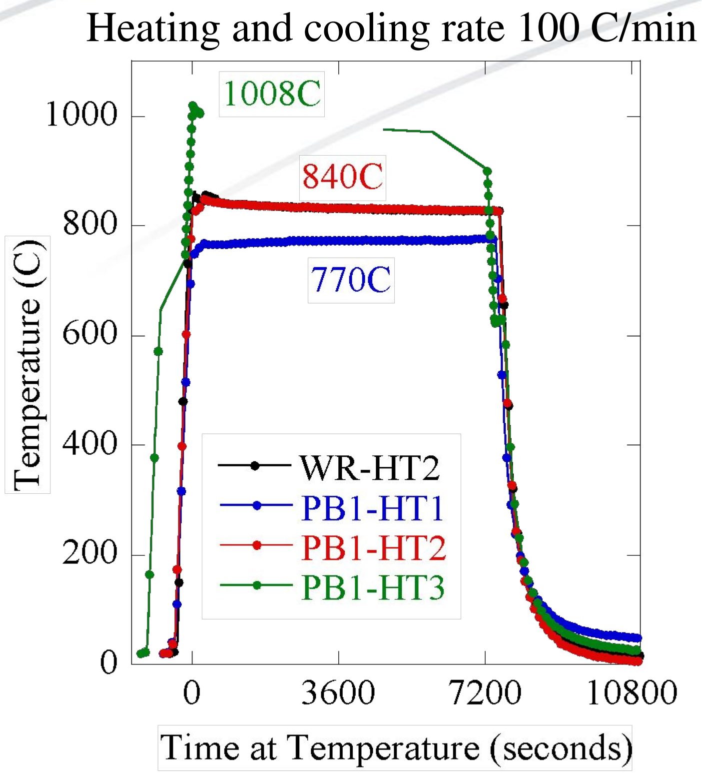




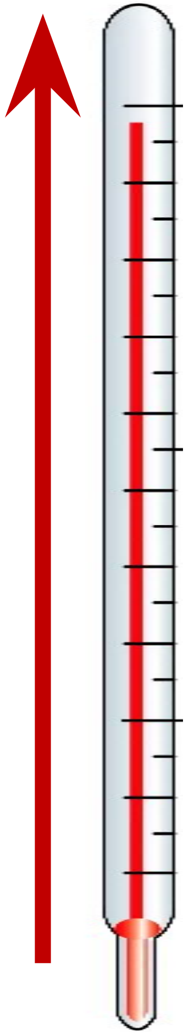
- PBF Material: 10^6C/min quench.
- Metastable microstructure, single phase α
  V trapped in α.

Physics and measurement rate were consistent.

Summary: In-Situ Diffraction Enables Intelligent Design of a Post-Build Heat Treatment to Achieve Specific Properties



RT (300K):Single phase α′, metastable microstructure, high dislocation density, intergranular residual stresses.

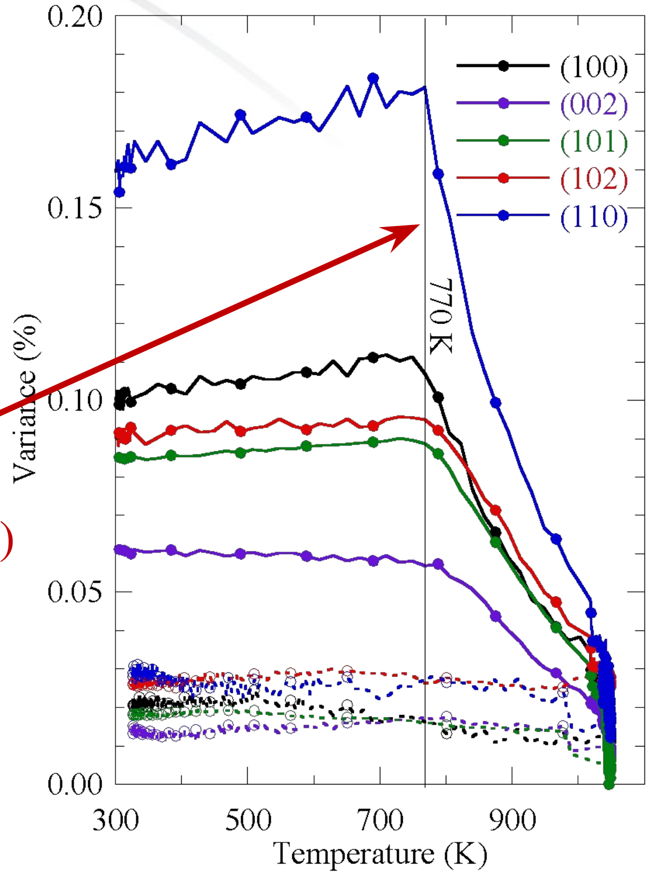


Summary: In-Situ Diffraction Enables Intelligent Design of a Post-Build Heat Treatment to Achieve Specific Properties

500°C (770K) Recovery and stress relief occur concurrently.

RT (300K):Single phase α, metastable microstructure, high dislocation density, intergranular residual stresses.

Recovery (dislocation annihilation)



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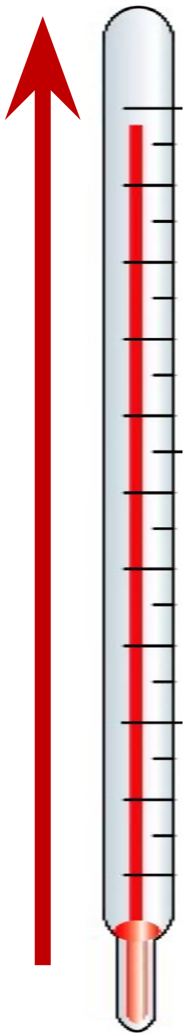
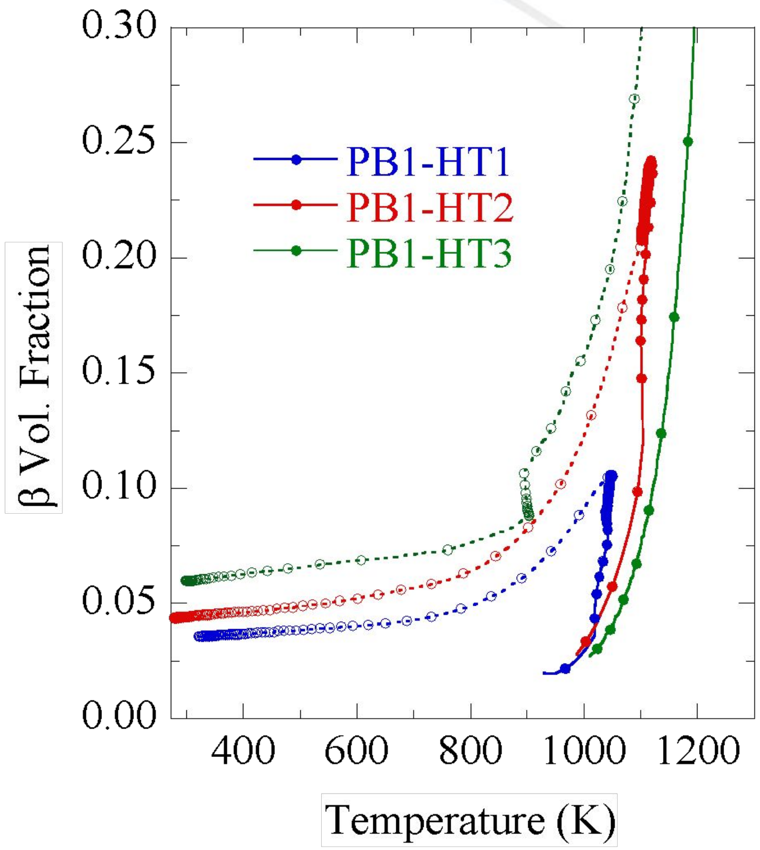


~600°C (900K) α→β initiates

500°C (770K) Recovery and stress relief occur concurrently.

RT (300K):Single phase α, metastable microstructure, high dislocation density, intergranular residual stresses.



Summary: In-Situ Diffraction Enables Intelligent Design of a Post-Build Heat Treatment to Achieve Specific Properties

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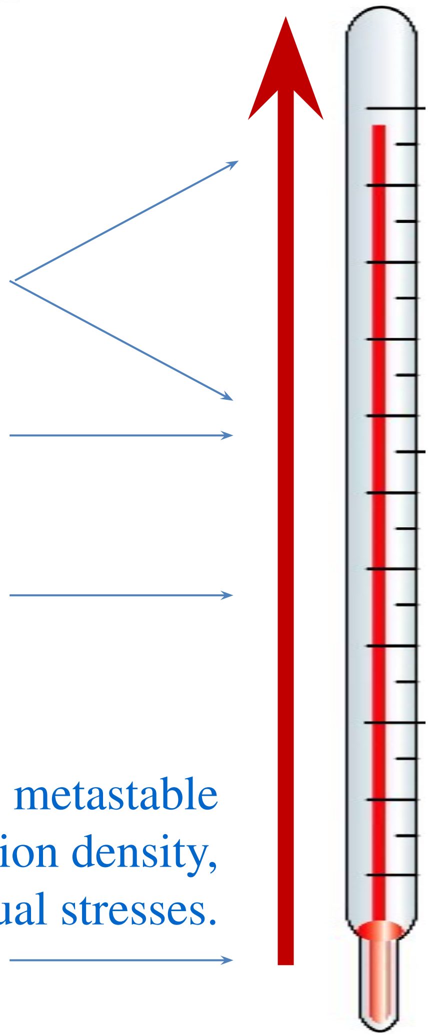
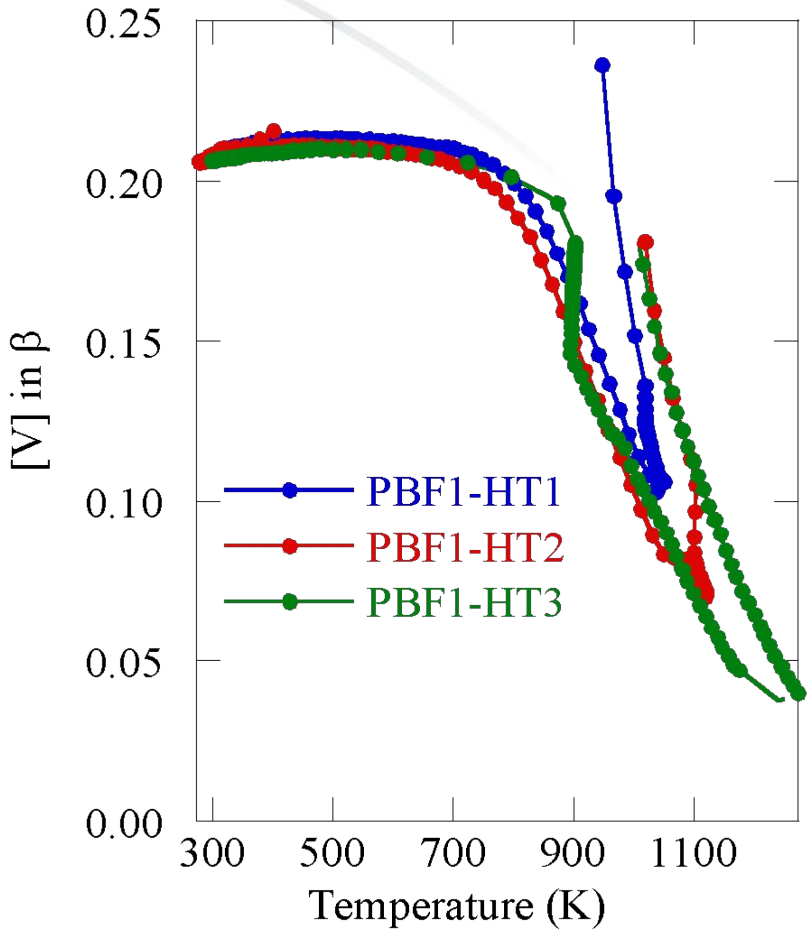
Vanadium redistribution

~600°C (900K) α→β initiates

500°C (770K) Recovery and stress relief occur concurrently.

RT (300K): Single phase α, metastable microstructure, high dislocation density, intergranular residual stresses.





Summary: In-Situ Diffraction Enables Intelligent Design of a Post-Build Heat Treatment to Achieve Specific Properties

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1008°C (1281K) β Transus

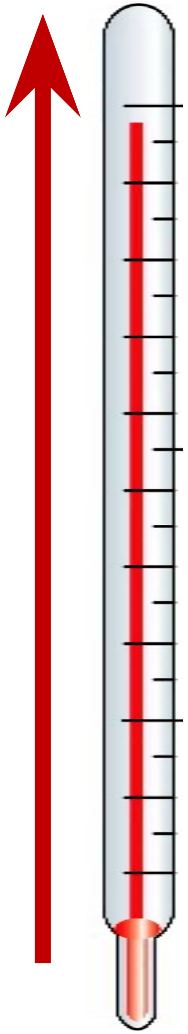
Grain/Lathe Growth

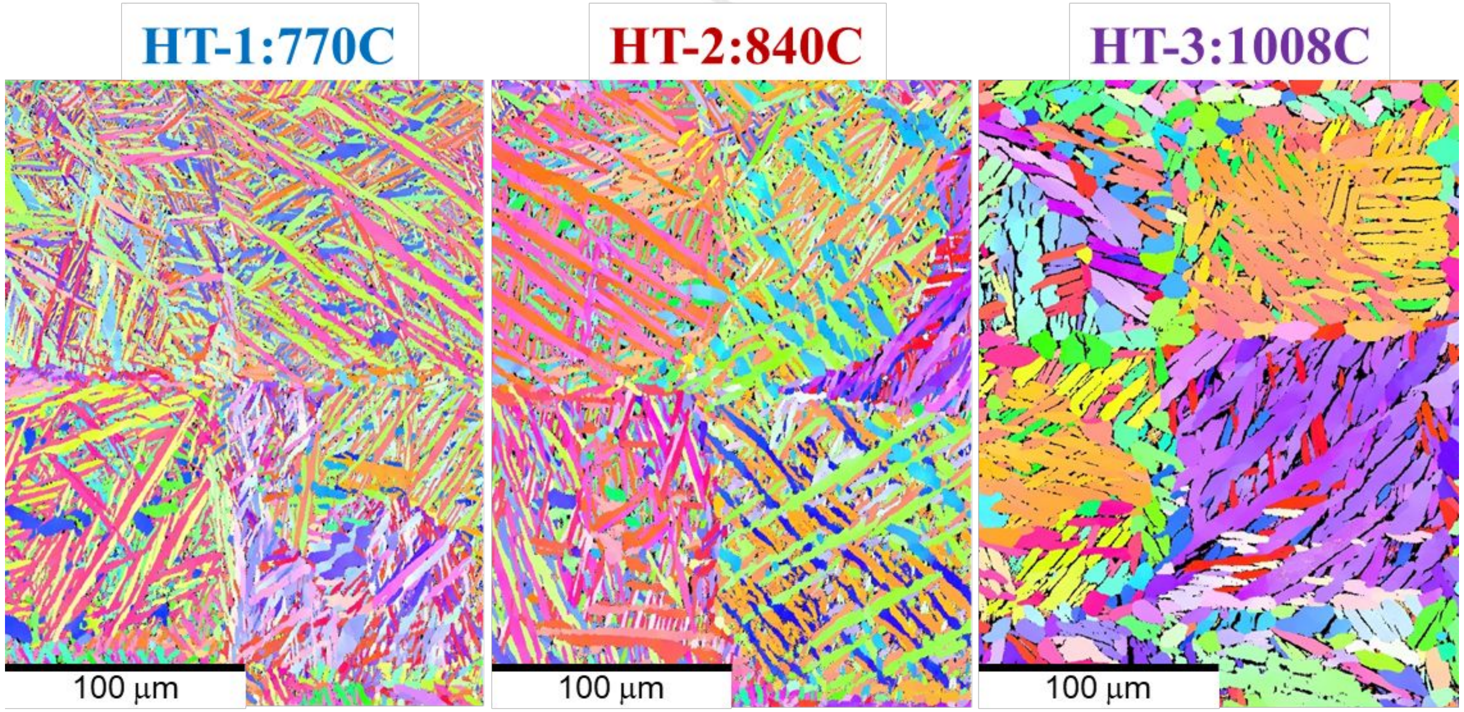
Vanadium redistribution

~600°C (900K) α→β initiates

500°C (770K) Recovery and stress relief occur concurrently.

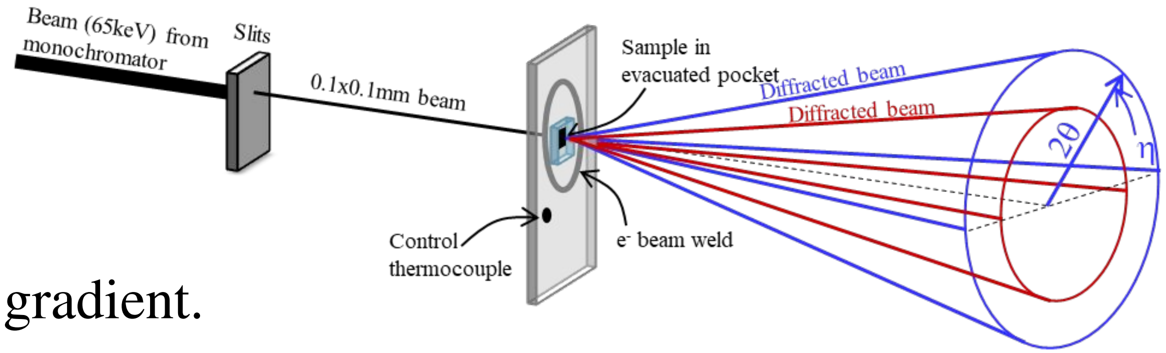
RT (300K):Single phase α, metastable microstructure, high dislocation density, intergranular residual stresses.





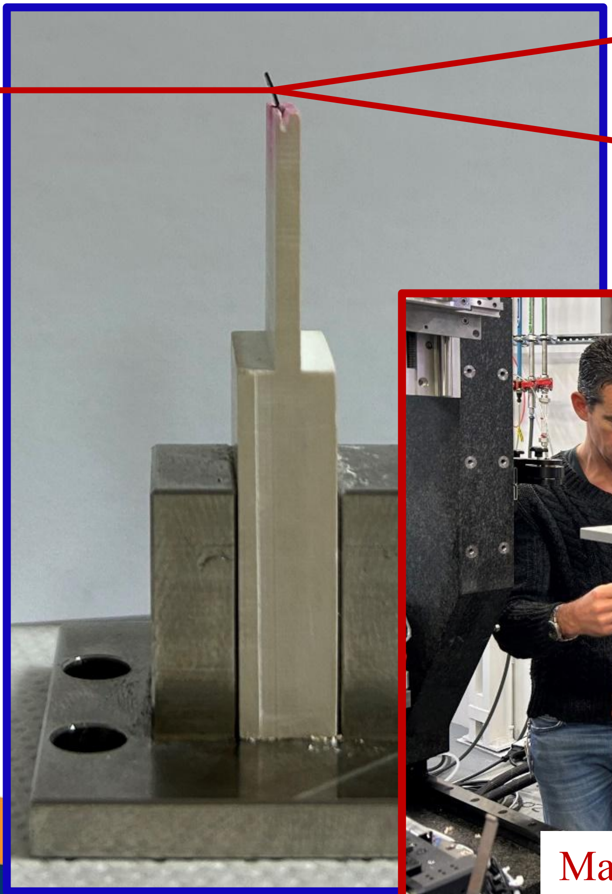
Data Rate (Detector Readout and Incident Flux) Set the Target Heating Rate

- Our target lattice parameter uncertainty (relative) is 100ppm (100με
  - Enables stress analysis, solute chemistry determination and line profile ana
- CTE of most metals is ~10ppm/°C.
- We can tolerate ~10°C of blur through dT/dt or dT/dx without worsening our resolution.
  - 1kHz data rate = 10,000°C/sec.
- Ok, how do we 1.) heat that fast (with acceptable surface/center thermal gradient) and 2.) how do we monitor temperature (±10°C) that fast?
  - Answer to 2.) is we cannot.
- Choices for heating…
  - High energy electron beam.
  - Direct current through sample.
  - Induction.
  - Laser.
- Surface heating limits sample thickness to control gradient.
  - 0.2mm thick (per Travis).
  - Limits coupling for induction.

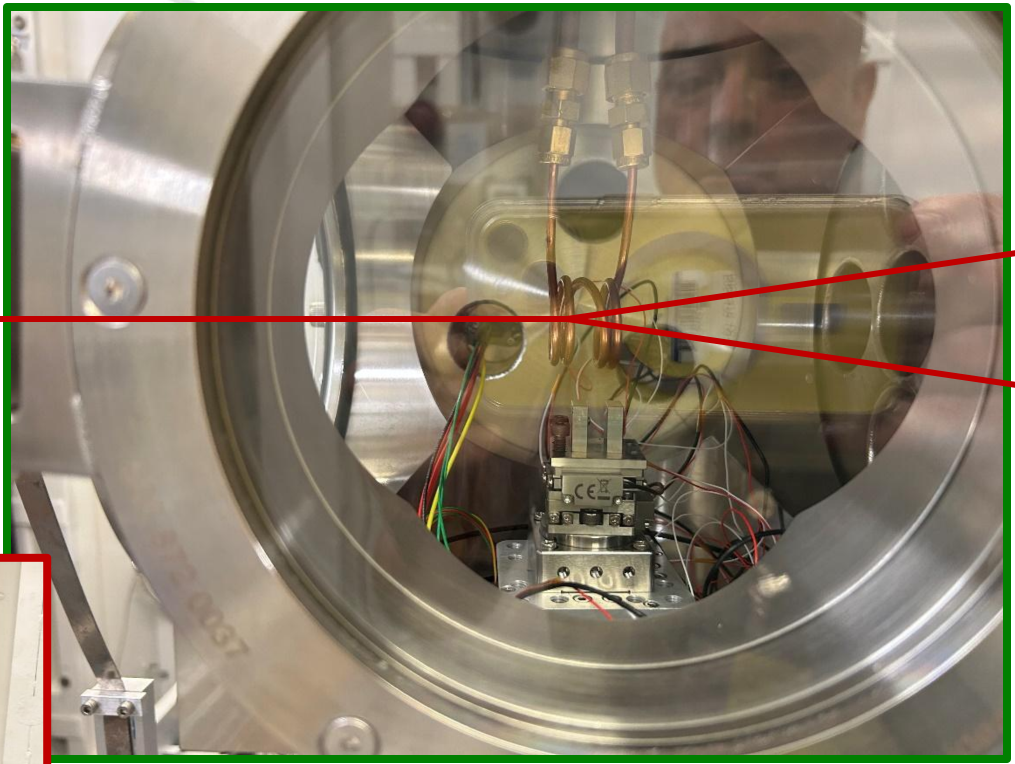


Rapid Heating Requires Modification of the Sample and the Environment

- Target 10,000 C/sec
- 3 x 4 x 0.2 mm Ti64 Sample
- Thin sample to minimize thermal gradient
    - ΔT<10C from surface to center
- No external temperature measurement



Marco DiMichiel and Travis Carver



- Induction heating supplies rapid power increase.
- Surface heating, drives gradient in beam direction
- Small sample, poor conductivity resulted in maximum heating rate of 1500 C/sec (in Ti).
- e-gun was preferred heating method (volumetric), but could not get it in time.

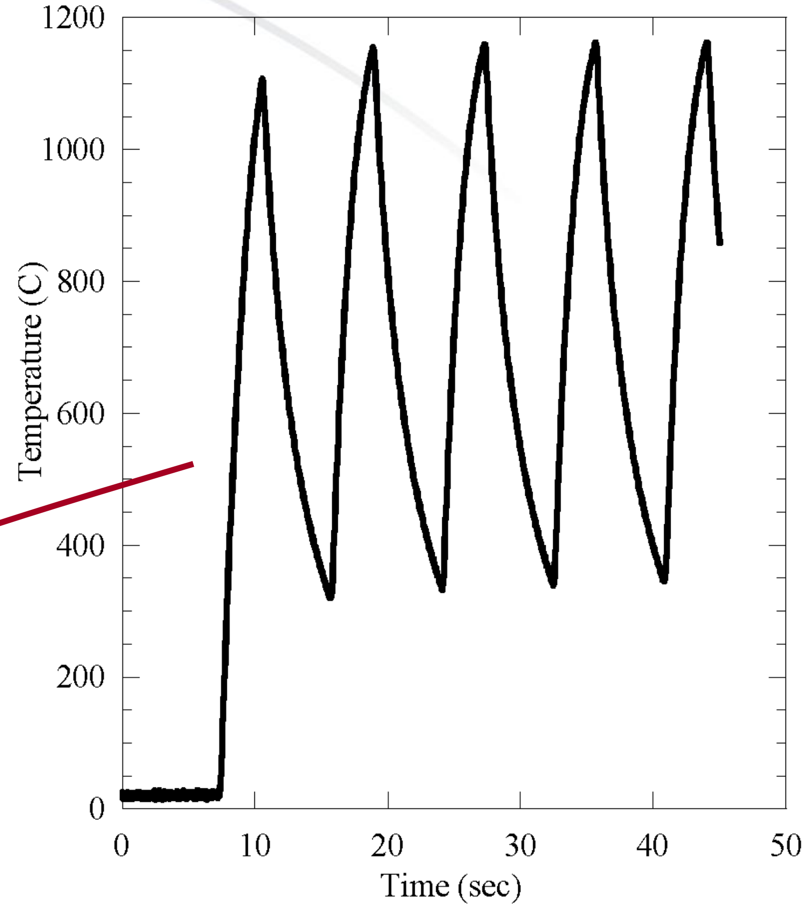
Collecting Data at 1kHZ Creates Its Own Problems

- Detector speed determined by requirements:
    - $\Delta q/q$ (FWHM) ~ 0.1%
    - $\Delta d/d$ (peak center) ~100ppm
    - Need ~5 complete rings to separate microstructural effects (e.g. texture and phase).
- 4 Ti64 Samples: 0.5 C/sec, 5 C/sec, 50 C/sec, 500 C/sec
    - 75,000 2D diffraction images on 500 C/sec sample.
    - Caked into 24 1D patterns/image
- 2 Cycling samples
- Corresponding samples with cp Ti (heated faster).
- Some other stuff
- Total of ~1M Diffraction Images.
- GSAS on my laptop takes ~7 seconds to refine 1 pattern.

Stefano Checchia and Eric Peterson

Completed 5 Cycles on AM Ti64 With Max Heating Rate of ~500C/sec

- 22000 Diffraction images in 50 Sec.
- Also completed 10 cycles with power decreasing 5%/cycle to simulate repeated deposition (DED).
- But this data belongs to someone else !



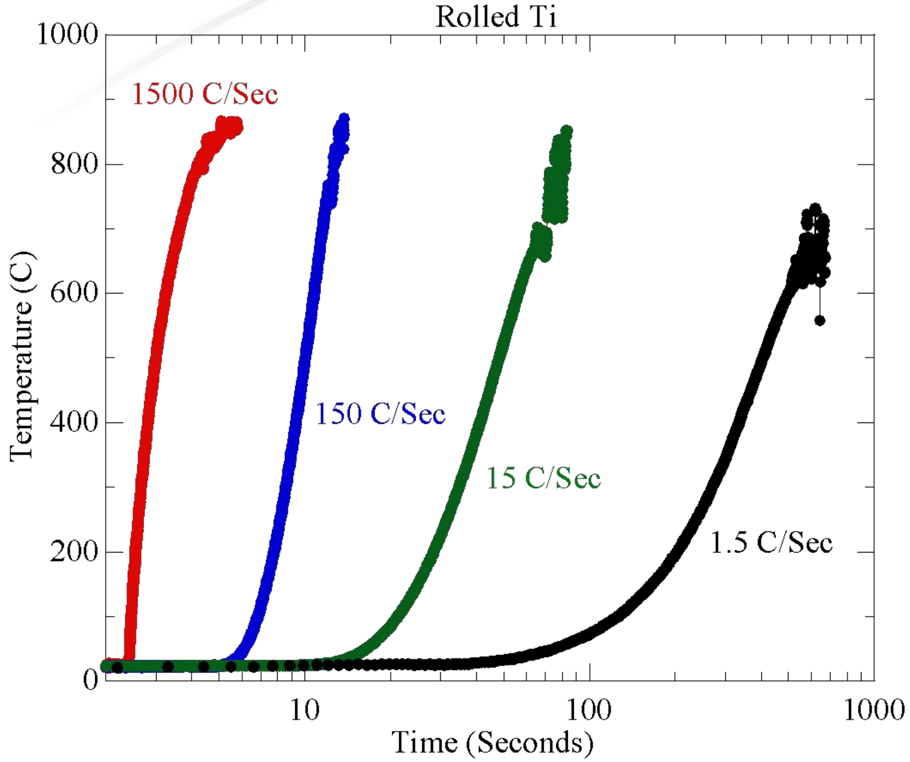
Temperature determined from lattice parameter and known (?) thermal expansion of Ti64.

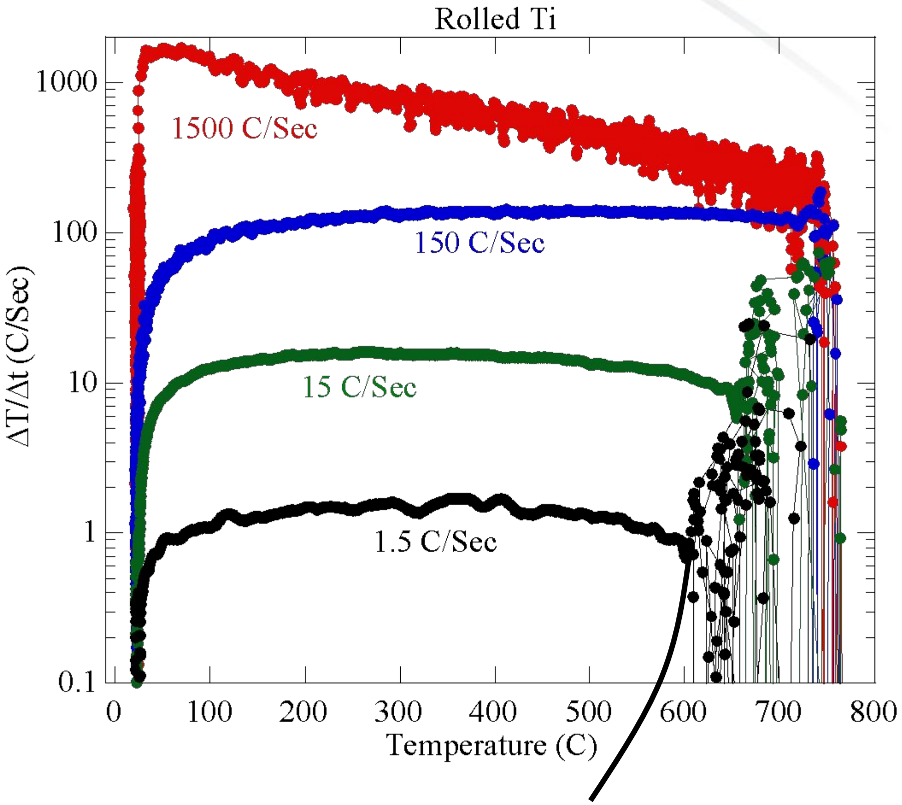
Microstructure Evolution as a Function of Heating Rate

- Temperature determined by lattice parameter and known CTE [1].
- Heating rate varied over 4 orders of magnitude.

Rolled CP-Ti Grade 1[2] (Goodfellow)

Grade	C	O	N	H	Fe	Other (total)
1	0.08	0.18	0.03	0.015	0.2	0.4





Lattice parameter fit becomes unstable.

Function of heating rate

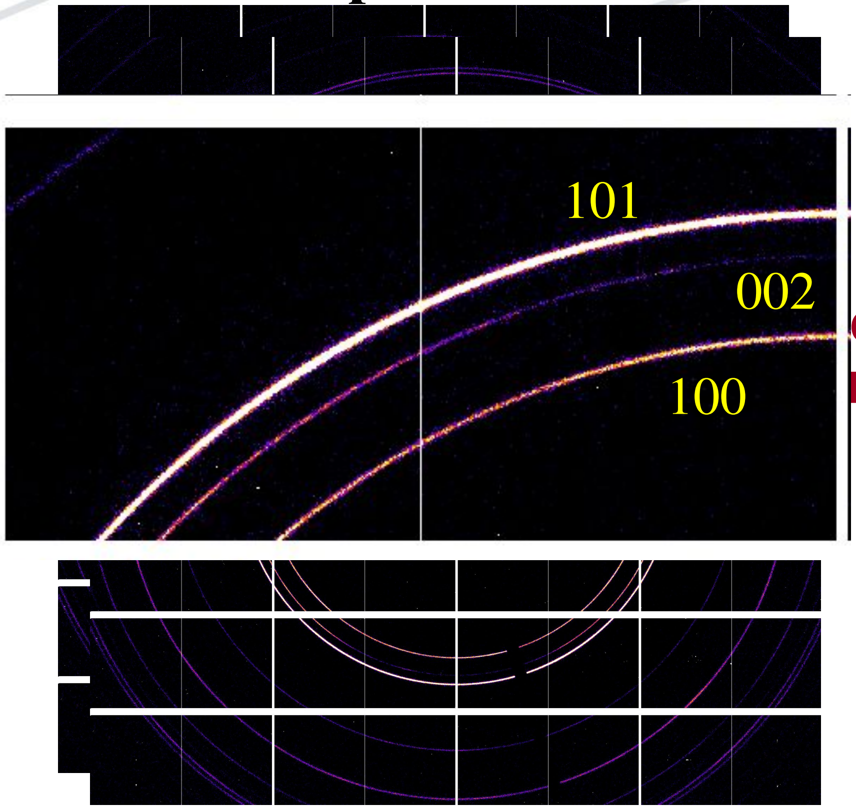
Well below β Transus.

[1] Touloukian, YS, Kirby, RK, Taylor, RE, Desai, PD, Thermal Expansion: Metallic Elements and Alloys, Plenum Publishing Company, New York, 1975.

[2] Asme, Specification for Titanium and Titanium Alloy Strip, Sheet, and Plate Sb-265, Grade 1—Unalloyed titanium, 2013.12

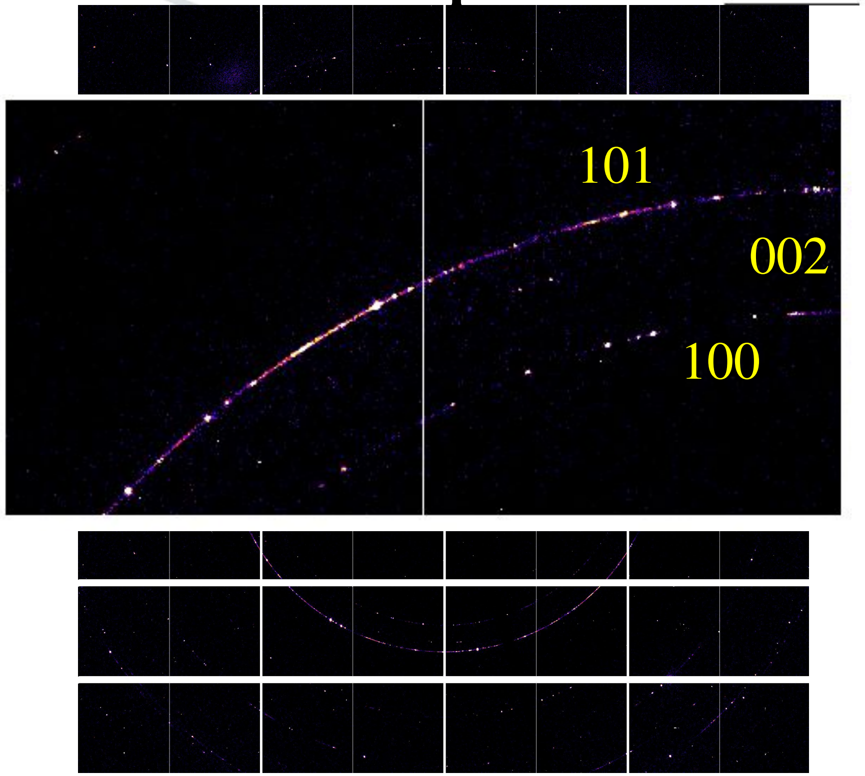
Diffraction Rings Become "Spotty" at High Temperature

Room Temperature



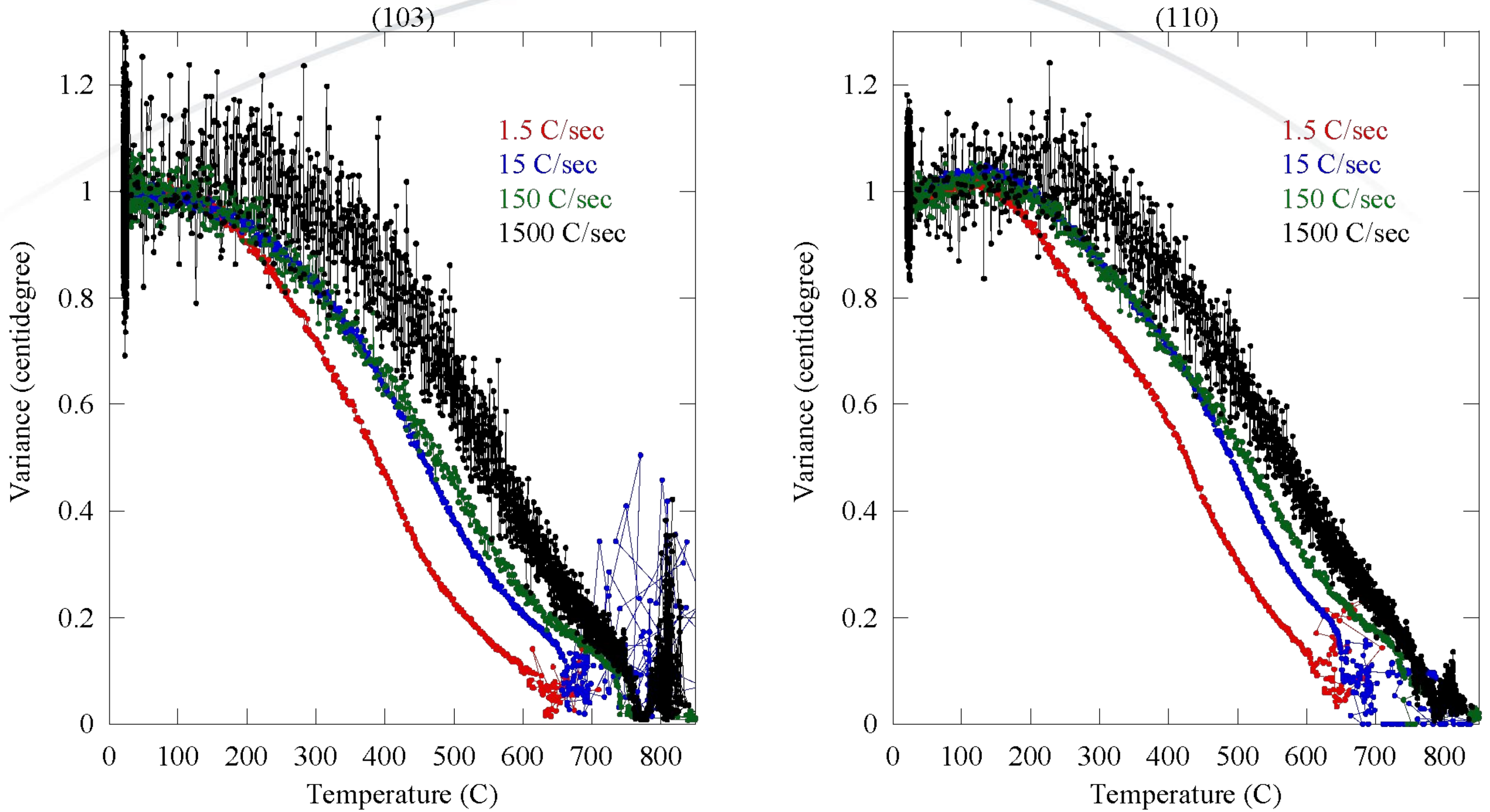
eat Pulse

Elevated Temperature

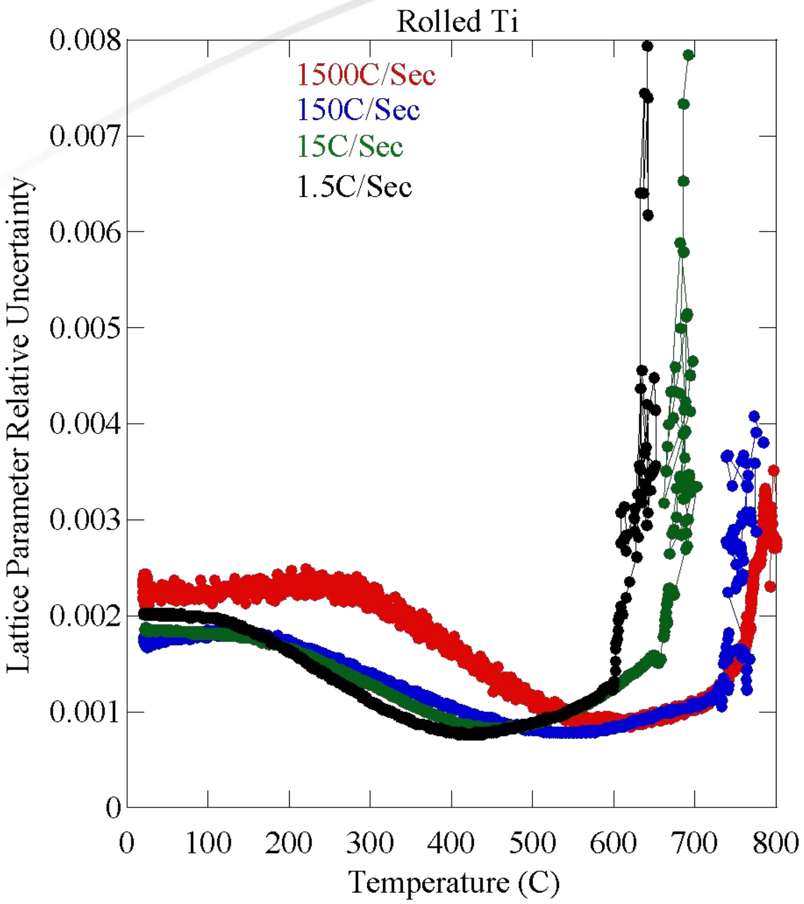


- Spottiness caused by 2 things…
  1. Grain growth
  2. Decrease in mosaicity
- We will associate it with recrystallization.

Dislocation Recovery is Heating Rate Dependent

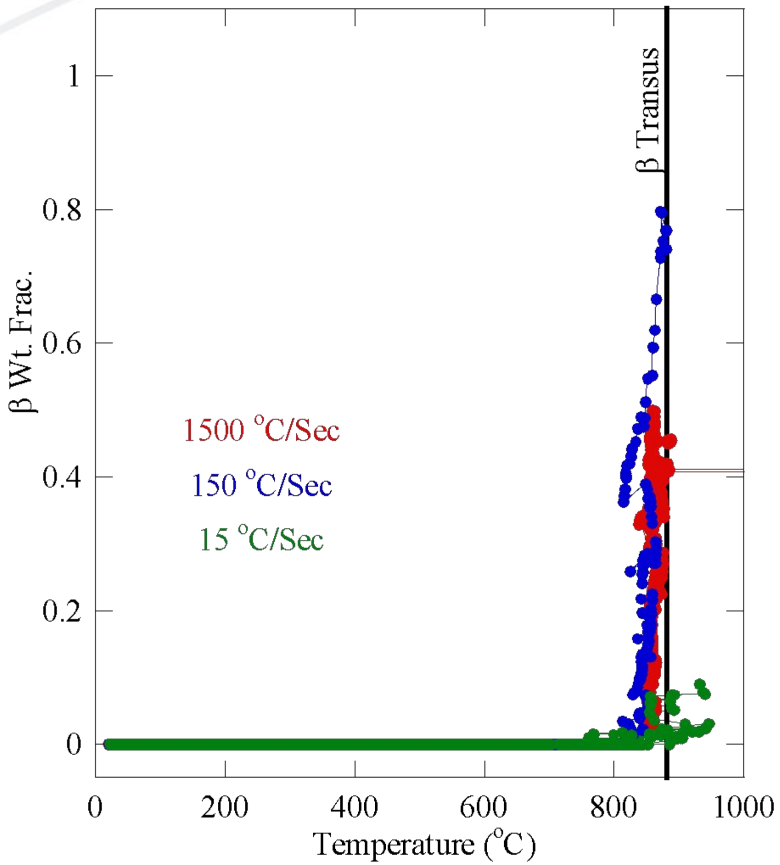


Recrystallization is Observed to Be Heating Rate Dependent



- The rapid increase in lattice parameter uncertainty is a result of the rings getting “spotty”.
- If we associate this with RX, then the obvious conclusion is that RX is heating rate dependent.
- Working to define a statistical function to quantify this effect (Matt, I might need another 6 months!).

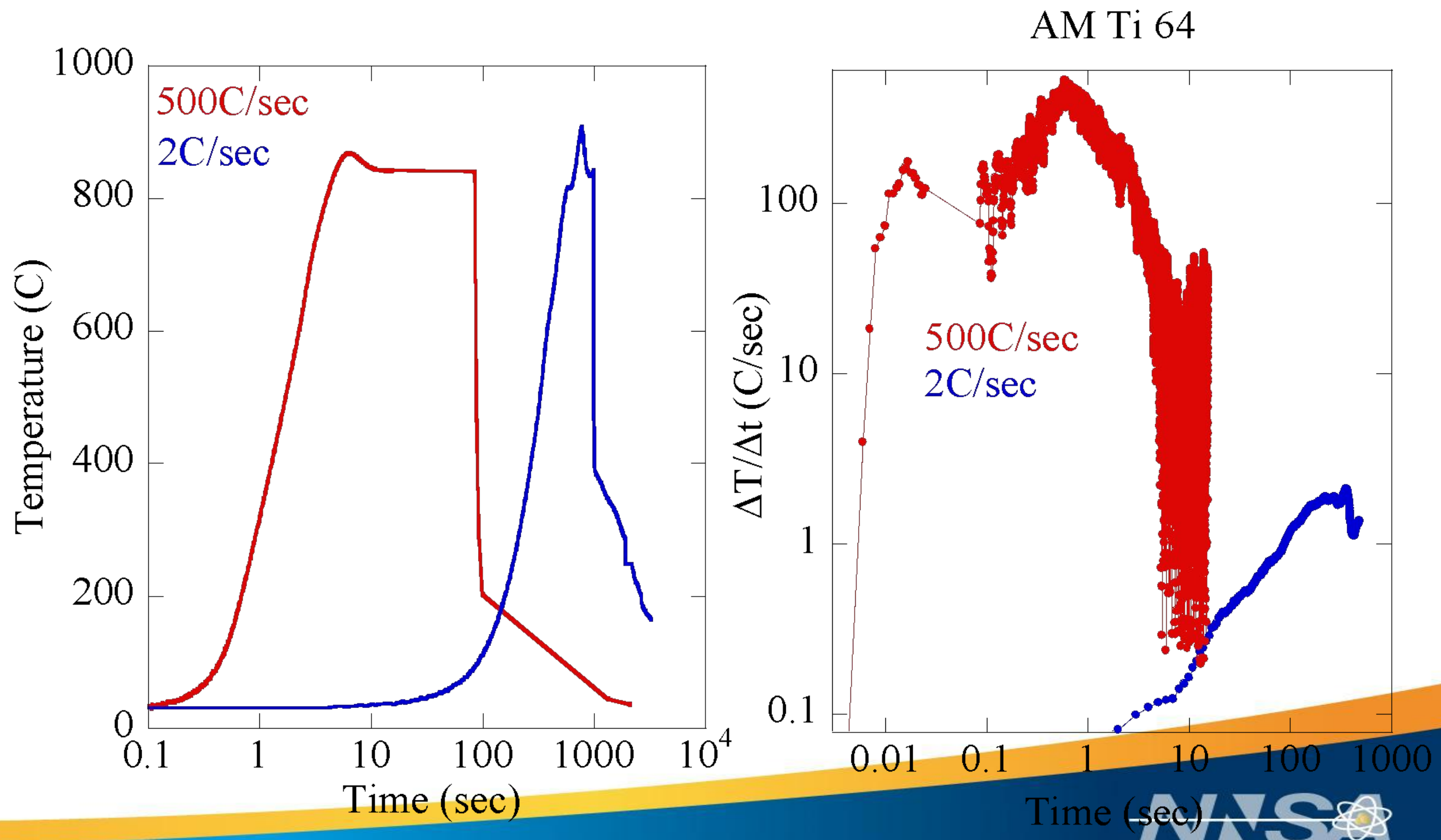
Transformation To β Is Not Heating Rate Dependent (At All)



- This is expected for a nominally pure material.
- Absolute phases fractions should not be believed.
- Provides a check on temperature calibration.

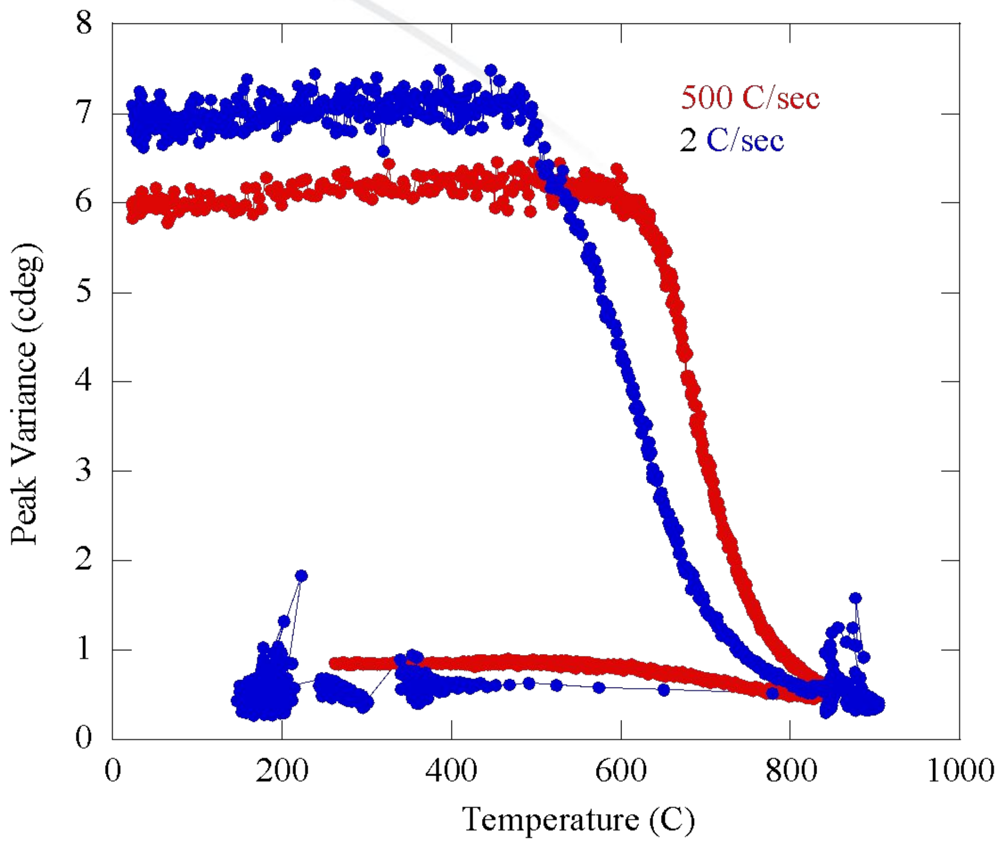
Microstructure Evolution as a Function of Heating Rate in AM Ti64

- Heating rates over 4 orders of magnitude.
- Monitor dislocation density, internal stress, phase, and Vanadium concentration.
- Preliminary analysis at this point.
- Cannot ignore solute chemistry !



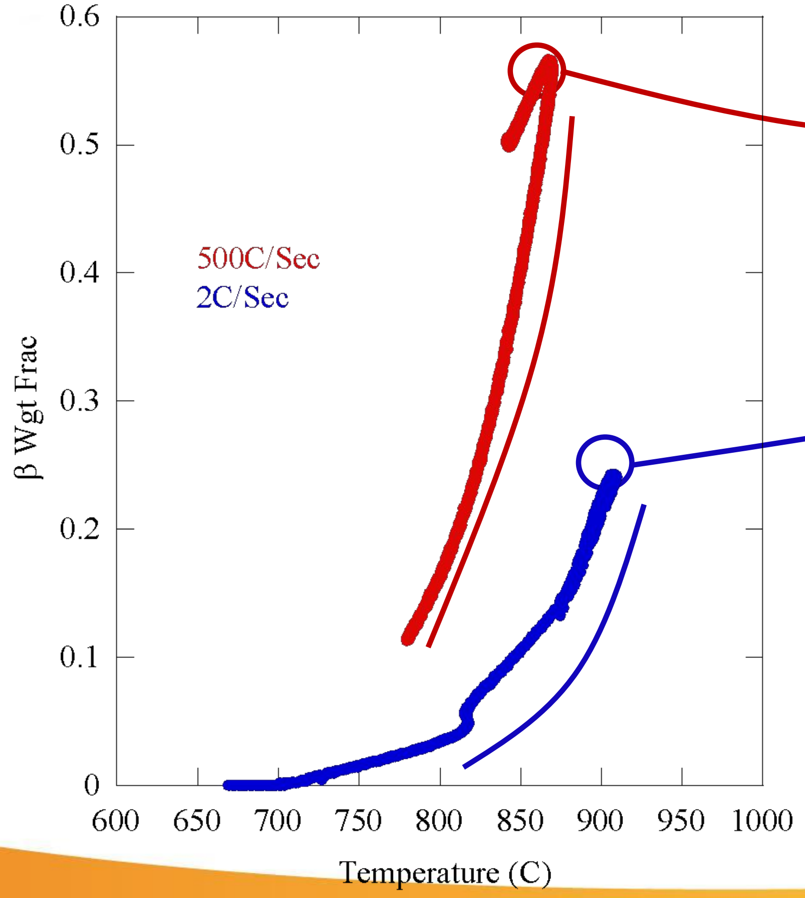
Rate Dependence of Dislocation Recovery is as Expected

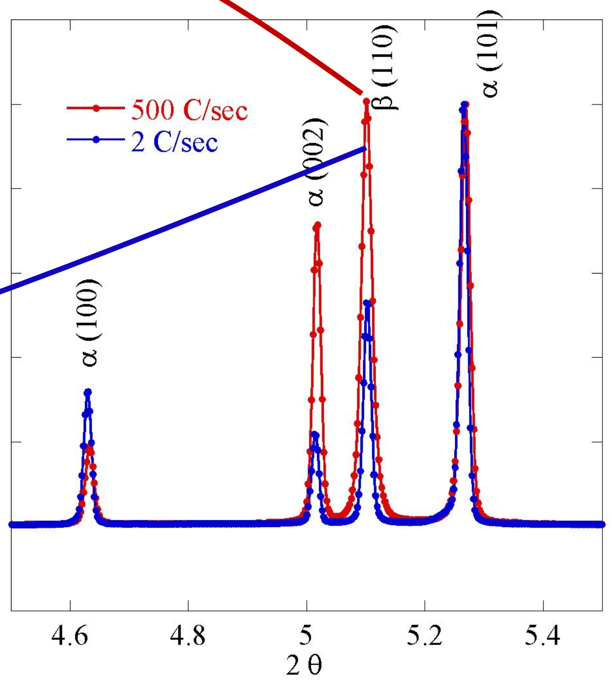
- Dislocation motion is governed by diffusion.
- Lags when heated quickly, delaying recovery.



Phase Transformation Does Not Behave as Expected

- Expect V diffusion to control phase transformation, again delaying it.
- But observation does not agree with that.





- Reminder, preliminary analysis!

Conclusions

- High-energy X-ray (and neutron) diffraction can be used to monitor microstructural evolution during simulated processing.
  - AM, post-build heat treating, welding, forging, machining…
  - Phase, texture, dislocation density, internal stress, temperature…
- New developments in x-ray sources and detection enable experimental data rates up to and exceeding 1kHz (with sufficient q- or d-resolution).
- Rolled cp-Ti
  - Dislocation recovery and RX are observed to be heating rate dependent.
  - $\alpha \rightarrow \beta$ transformation is not (over 3 orders of magnitude dT/dt)
- Powder Bed Fusion Ti-6Al-4V (preliminary analysis)
  - Dislocation recovery is heating rate dependent.
  - $\alpha \rightarrow \beta$ transformation is not as expected.
- The results can be used to design processing route or, if coupled with mechanical testing, to advance understanding of the process/structure/properties relationship.
- Currently pushing experiments to ever higher data rates and faster kinetic processes.

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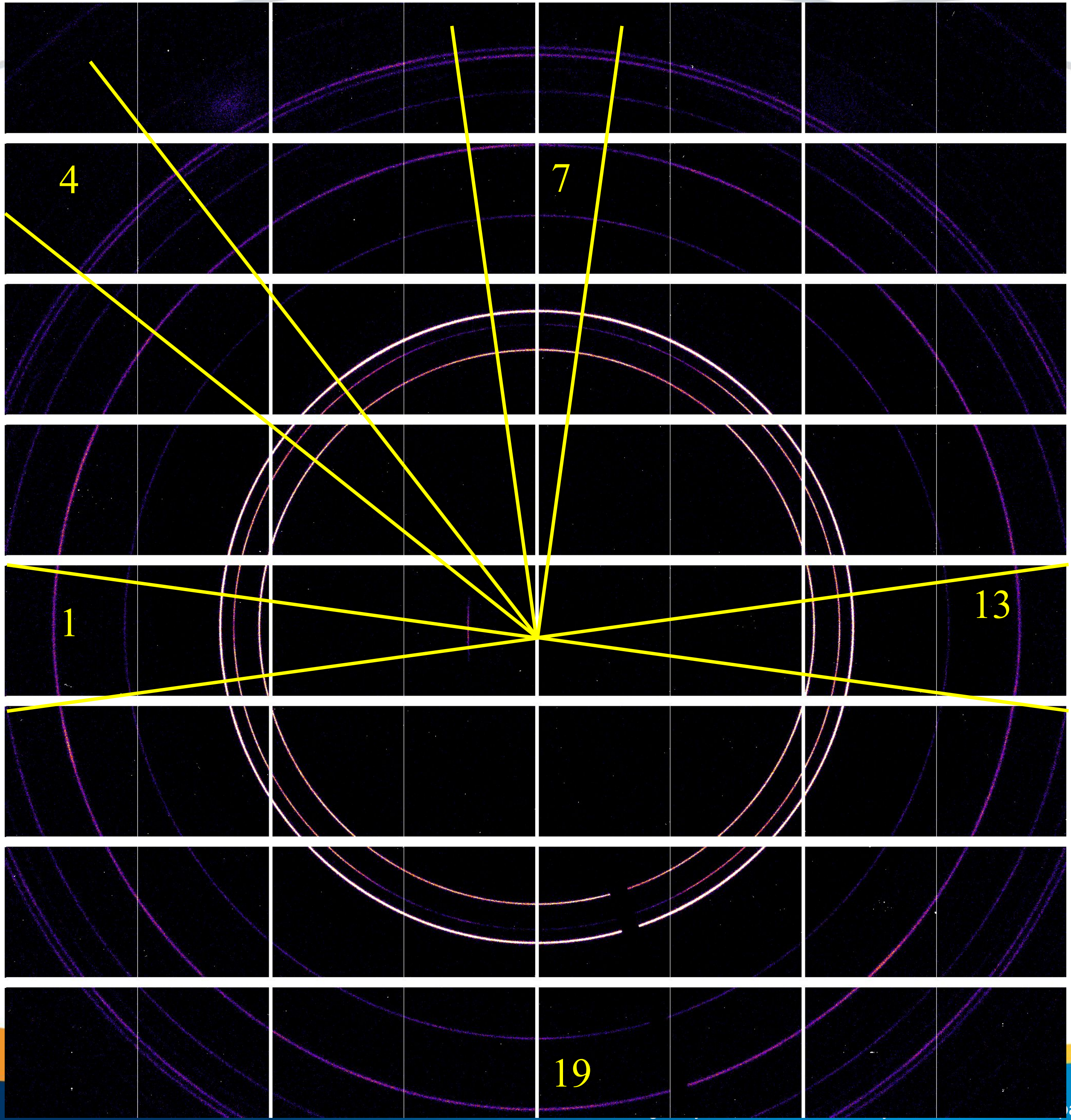
  - AM, post-build heat treating, welding, forging, machining…
  - Phase, texture, dislocation density, internal stress, temperature…
- Powder Bed Fusion Ti-6Al-4V
  - Initial meta stable microstructure is vanadium supersaturated single phase α′ with fine laths, high internal stress and dislocation density.
  - Dislocation recover and stress relief happen concomitantly from 770K to 870K (500°C-600°C).
  - β phase becomes significant at 940K (613°C), increases to 100% at transus temperature of 1281K (1008°C).
  - Vanadium expelled from α to β as β phase grows.
  - Returns to near equilibrium β content and vanadium distribution at RT.
- The results can be used to design processing route or, if coupled with mechanical testing, to advance understanding of the process/structure/properties relationship.
- Currently pushing experiments to ever higher data rates and faster kinetic processes.

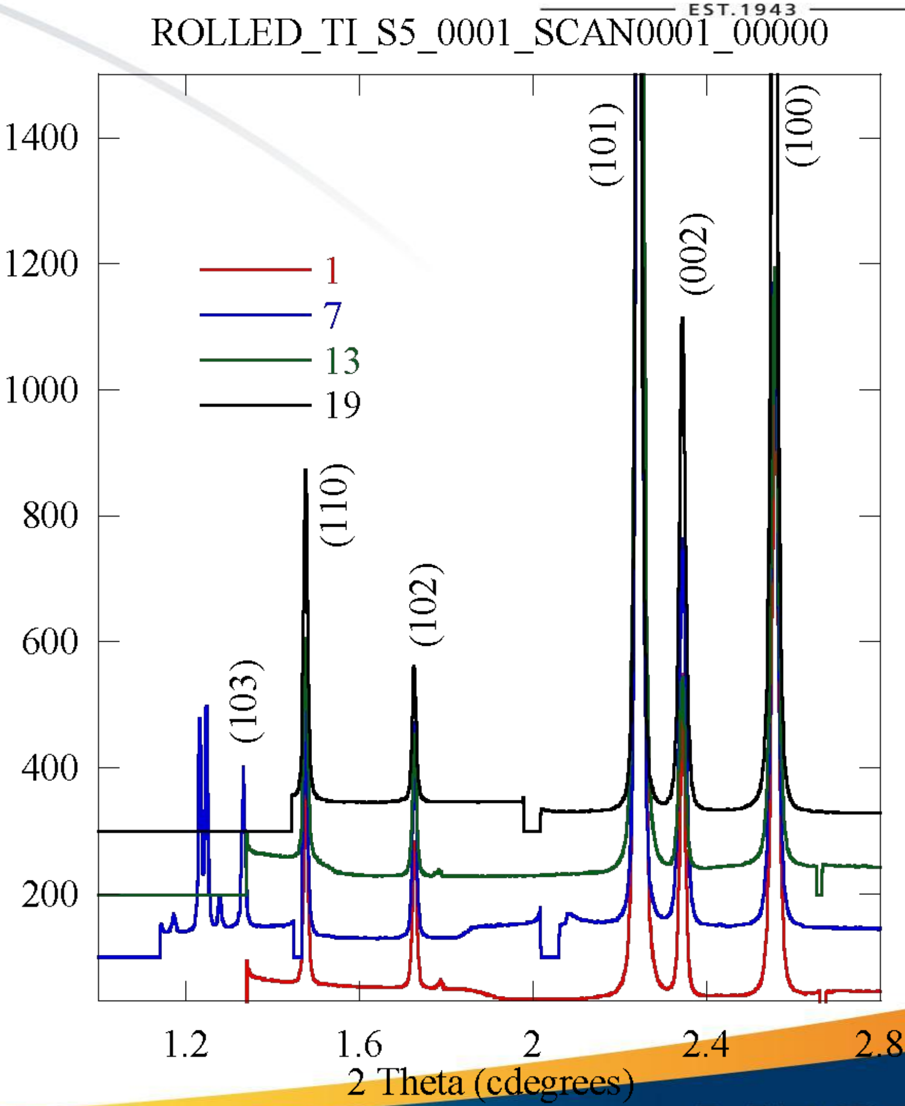
Even Though We Own a Neutron Source, We Choose to Use the Best Tool to Solve the Problem at Hand

Means frequent trips to x-ray sources, e.g. CHESS.

CHESS
CORNELL HIGH ENERGY SYNCHROTRON SOURCE

We Are Probing Several hkl's Along 1 Sample Direction





The Eiger 2 XE CdTe Detector Enables the Collection of High-Resolution Diffraction Data at Rates Relevant for Manufacture and Performance Environments.

Characteristics…

1) 32cm x 32cm Area coverage
2) 75um x 75um pixel pitch (18.1M
3) ~1000 Hz frame rate (16M versi
(Continuous readout, full area)
4) 20-bit pixel bin depth
5) Optimized for 20keV-100keV X-Rays
6) Count Rate: 10^7 ph/s/pixel
7) External User Gating
8) Arrived at LANL in July, en route to