

Project 33a-L: In-Situ Studies of Strain Rate Effects on Phase Transformations and Microstructural Evolution in β Titanium

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- Industrial Mentors: Austin Mann (Boeing)
Clarissa Yablinski (LANL)
- Other Participants: Jonah Klemm-Toole (Mines)



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- Student: Benjamin Ellyson (Mines)
- Advisor(s): Amy Clarke (Mines)

Project Duration
PhD: September 2017 to May 2021

- **Problem:** Uniform elongation and work hardening of titanium alloys restricts applications.
- **Objective:** Fundamentally understand microstructural evolution in metastable β titanium alloys to develop an alloy design methodology and tailor microstructures and properties.
- **Benefit:** Novel titanium alloys for blast and crash resistant applications

- Recent Progress**
- Ti-10V-2Fe-3Al (wt.%) (Ti-1023) aging study concluded
 - Quasi-static Ti-15Mo (wt%) tensile testing completed
 - Intermediate strain rate testing of Ti-1023 and Ti-15Mo partially completed
 - APS high-rate mechanical and diffraction data screened and post-processed

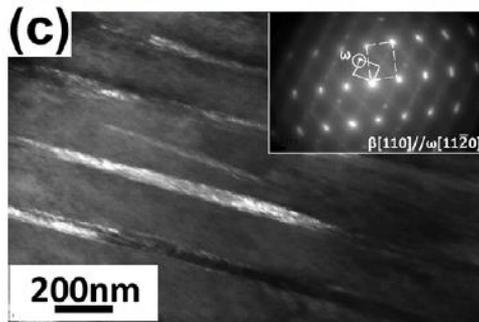
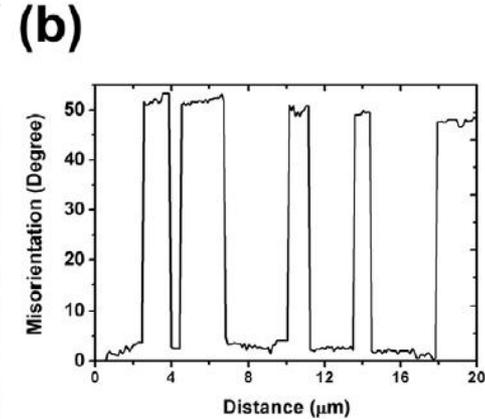
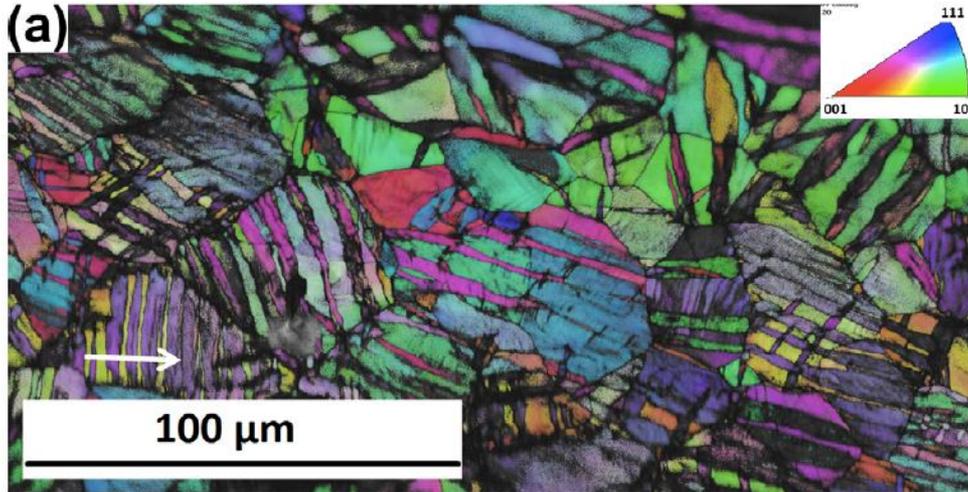
Metrics		
Description	% Complete	Status
1. Literature review	80%	●
2. Quasi-static mechanical characterization of Ti-1023 and Ti-15Mo	90%	●
3. Dynamic testing of Ti-1023 and Ti-15Mo	60%	●
4. Microstructural characterization of pre- and post-deformed samples	50%	●
5. <i>In situ</i> characterization of microstructural evolution during deformation	30%	●

- **Cellular Materials Program**
 - Multifunctional structures
 - **Blast resistance**
 - Thermal management
- **Propulsion Materials Program**
 - **Aircraft and marine engines**

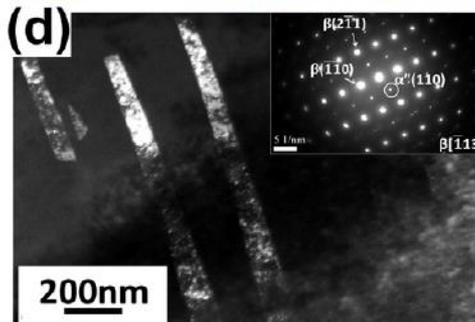


<https://www.onr.navy.mil/Science-Technology/Departments/Code-33>

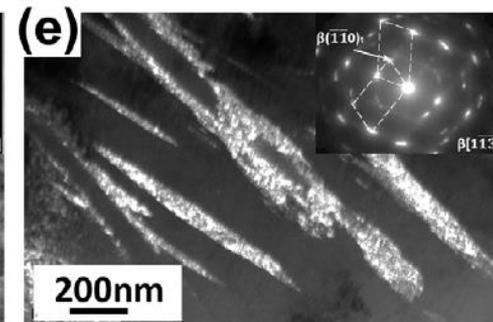
Ti-25Nb-3Zr-3Mo-2Sn (wt.%) Alloy Microstructure After Deformation



Deformation-induced
 ω phase



Deformation-induced
 α'' phase

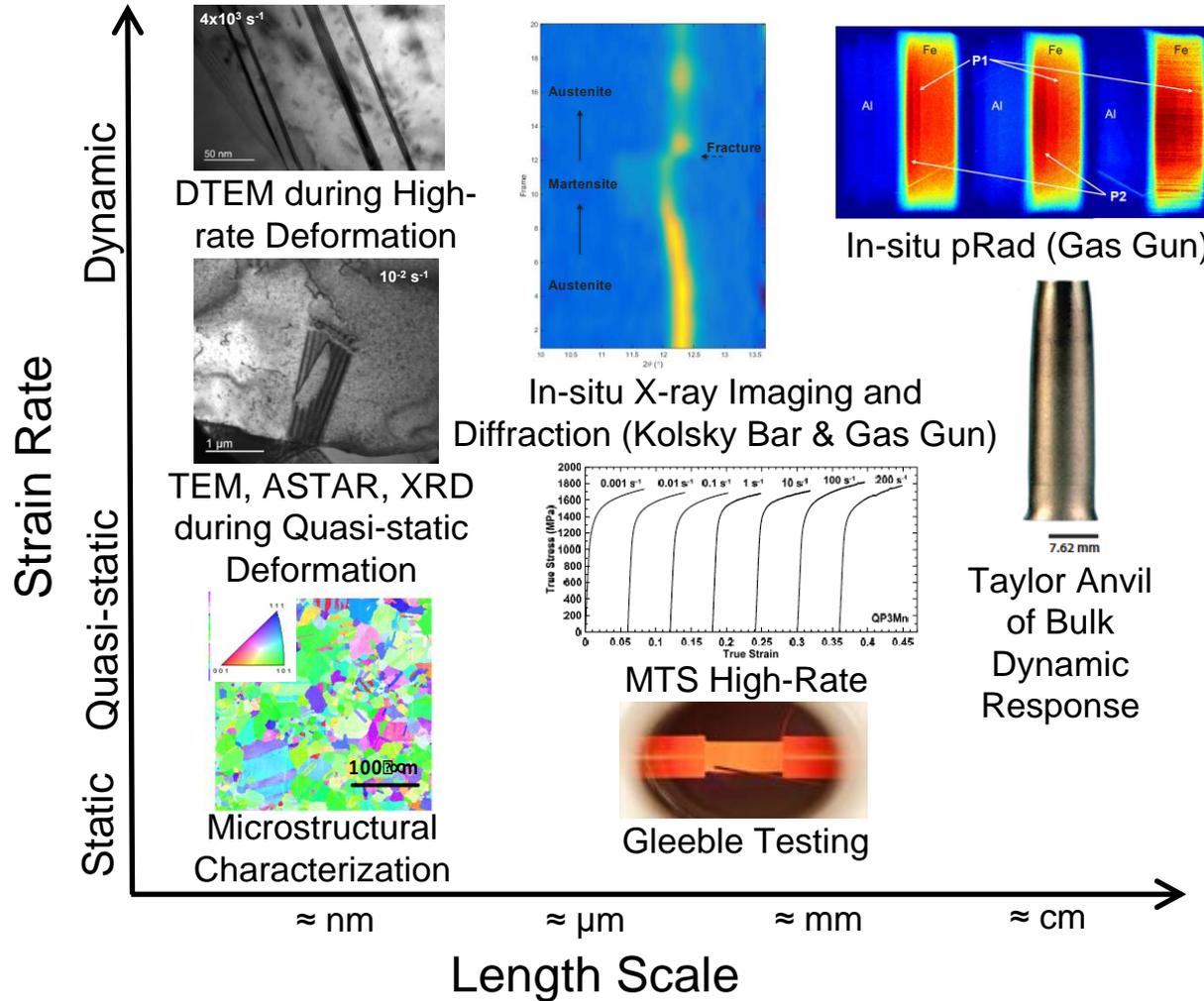


Deformation-induced
 β mechanical twinning

Compression at 10^{-3} to 0.18 true strain

H. Zhan, et al. 107 Scripta Materialia (2015): 34-37

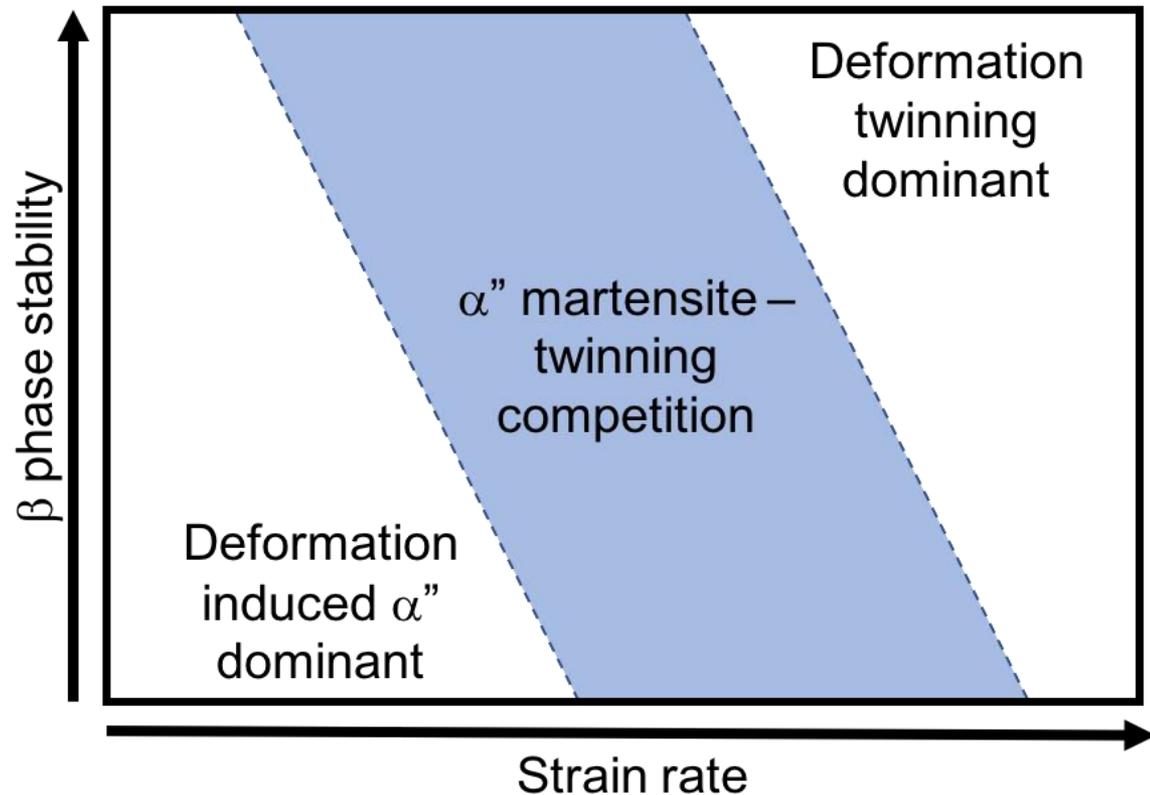
Multi-scale Studies of TRIP/TWIP During High Rate Deformation



The Effect of Strain Rate on Deformation Mechanisms During Compression of a Ti-10V-3Fe-3Al (wt.%) Alloy

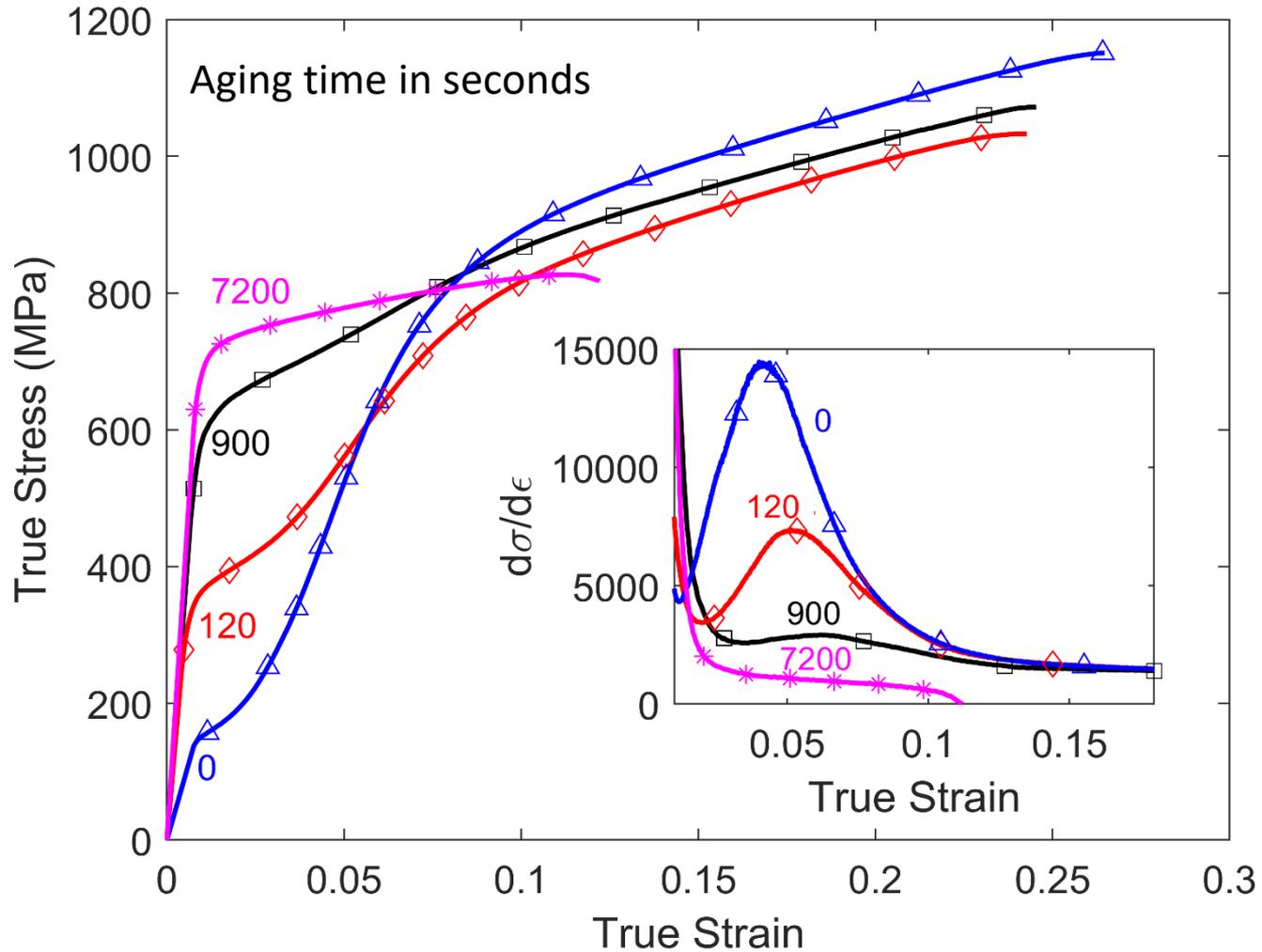
- **Deformation mechanisms present at all strain rates:**

- Stress-induced α'' martensite
- $\{332\}\langle 113 \rangle$ β twinning
- Stress-induced ω phase
- Slip

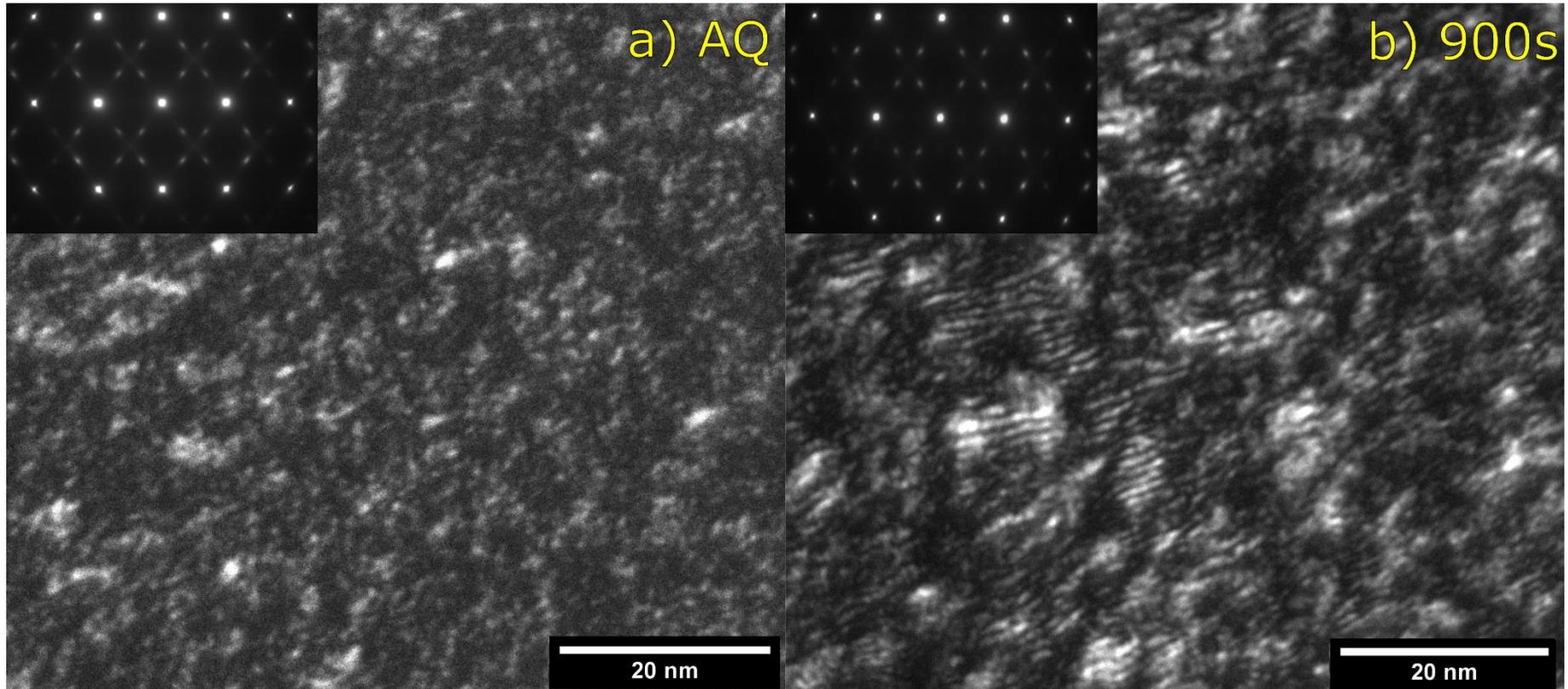


Ahmed, M., et al. 104 Acta Materialia (2016): 190-200

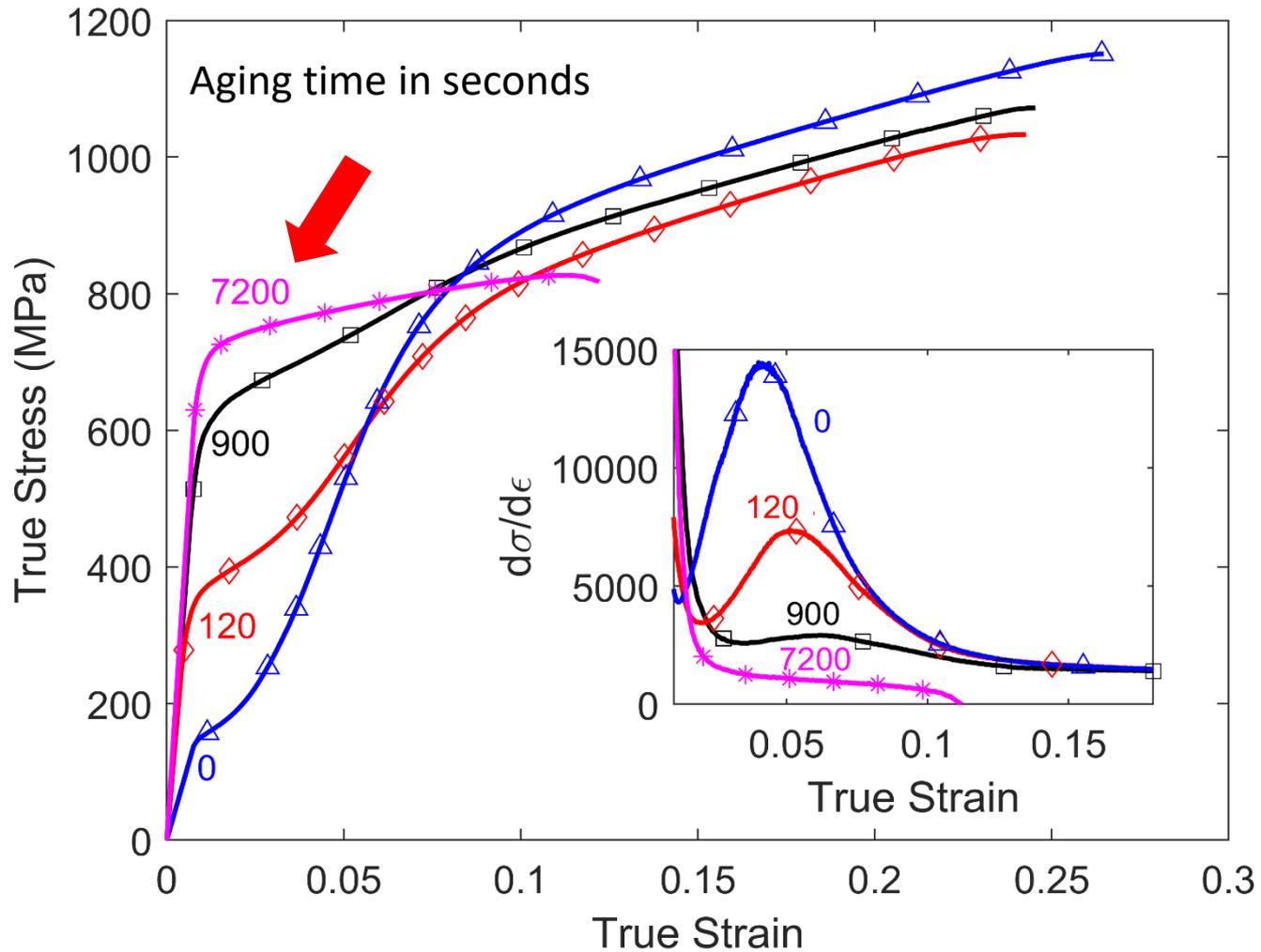
Artificial Aging in Ti-1023 at 423K



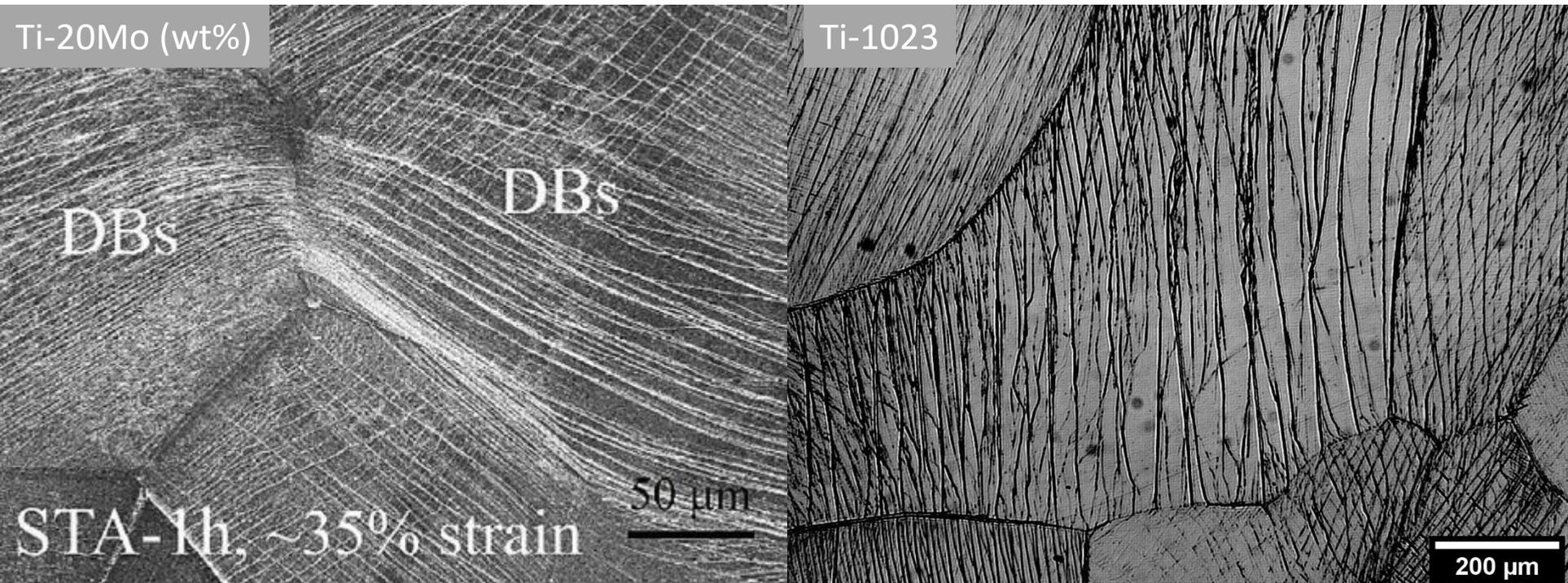
TEM of ω -phase Aging



Over-Aged Specimen (7200s)



Comparison of Over-Aged (7200s, 423K) Microstructure of Ti-1023



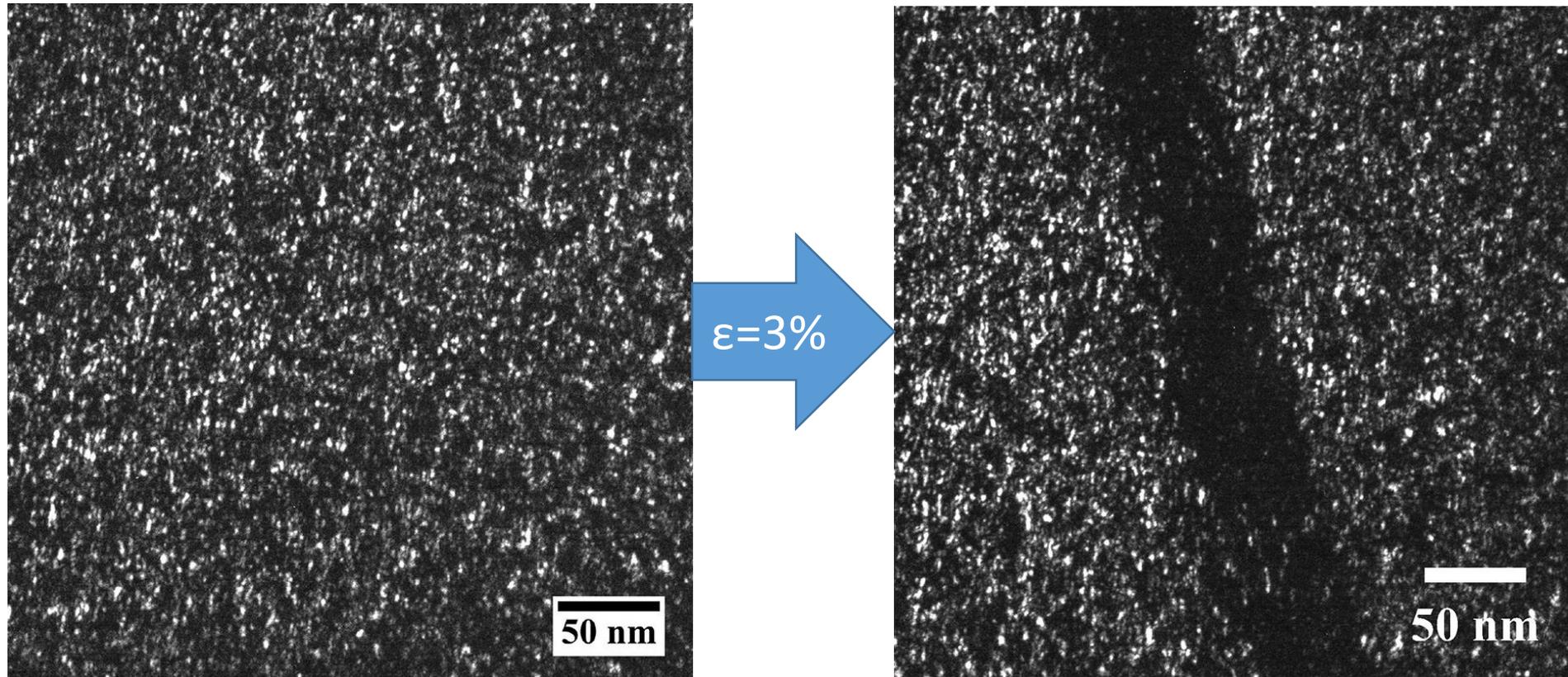
Chen et al., *Acta Materialia* (2019).

DBs : Dislocation bands

XRD indicates that the material is still single phase β , i.e. no martensite is present

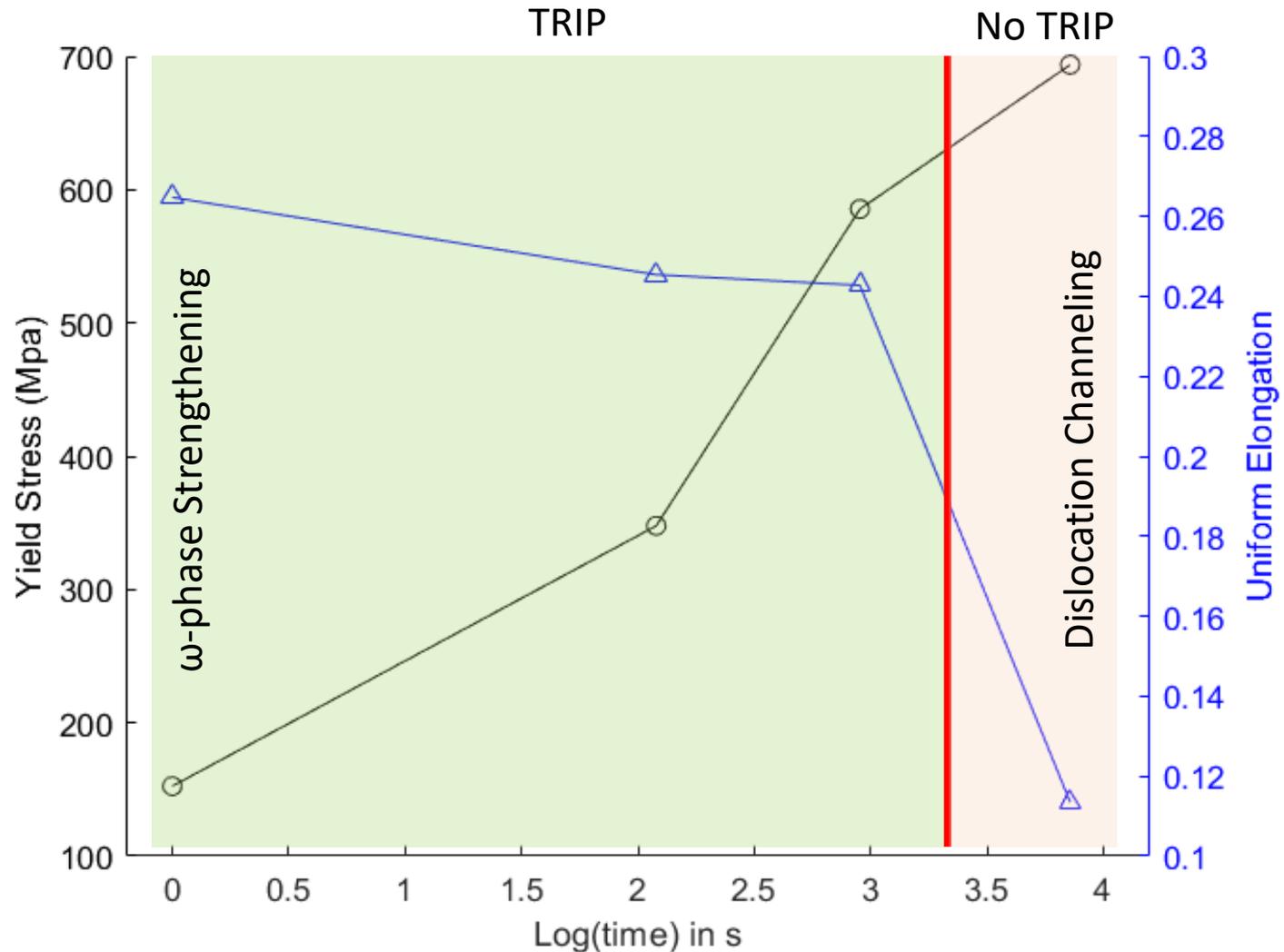
Artificial Aging of Ti-1023 for 7200s at 423K inhibits stress-induced martensite and causes dislocation bands to form

Dislocation Channeling

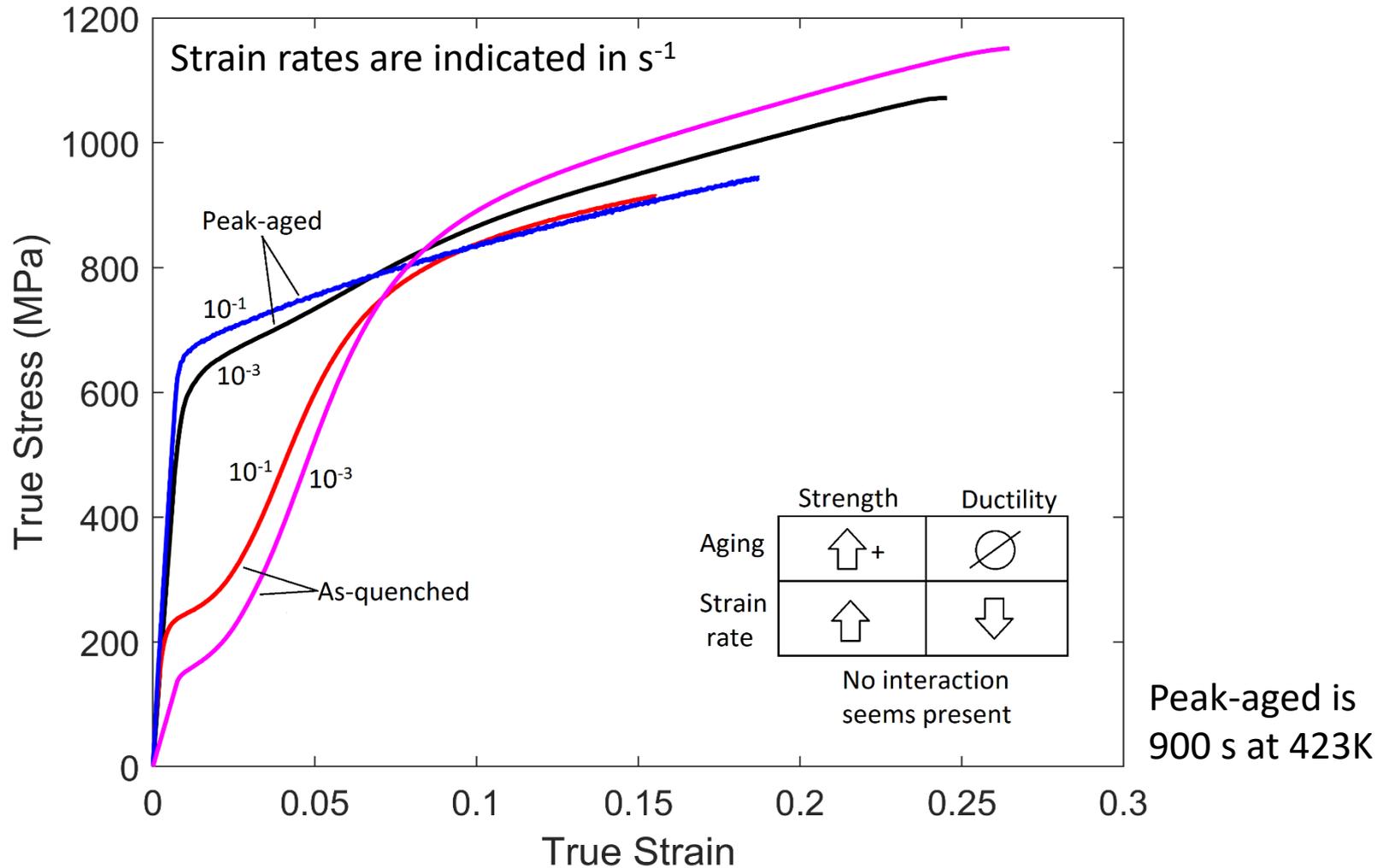


Lai et al. *Acta Materialia* 151 (2018): 67-77.

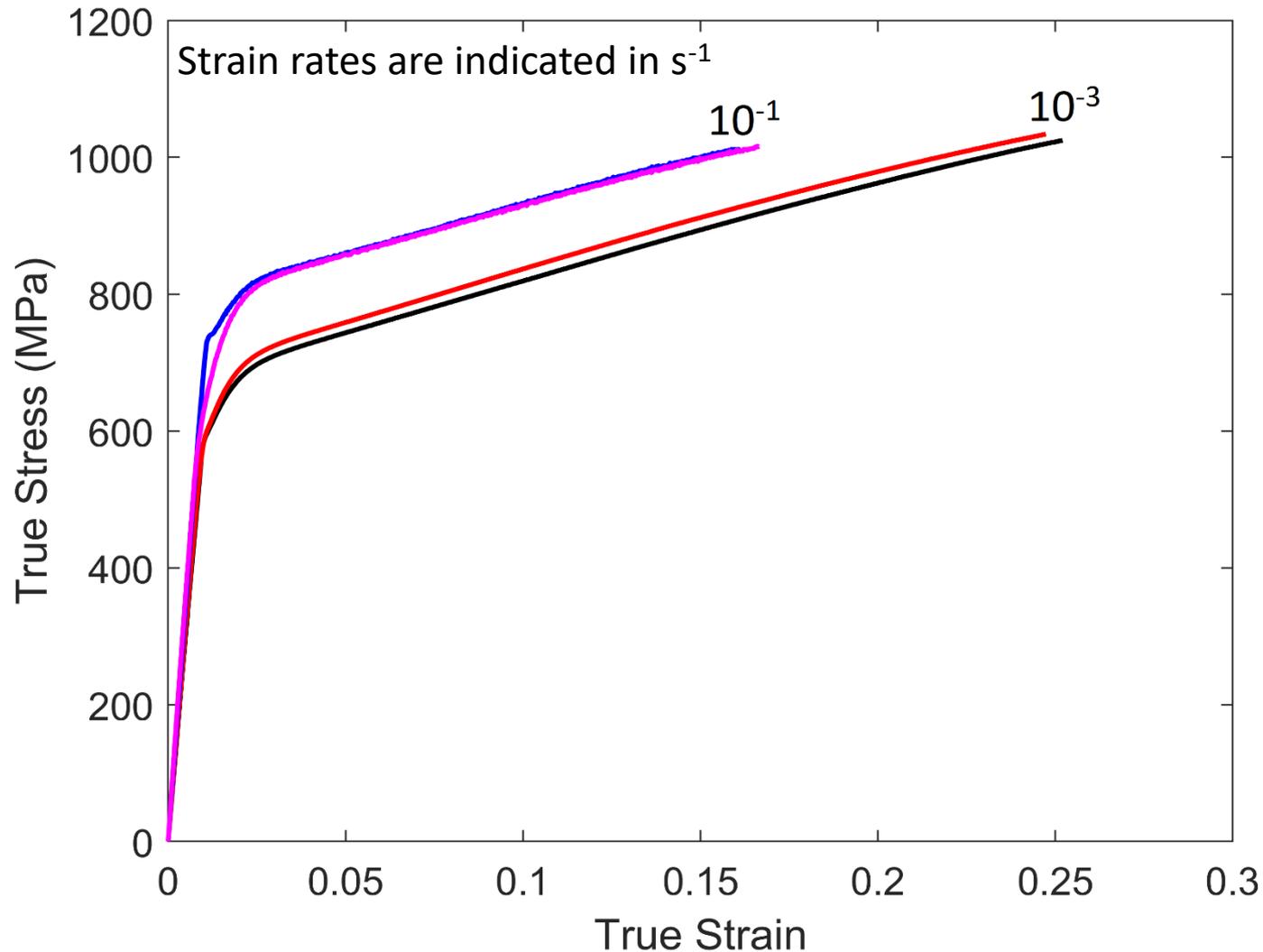
ω -phase Strengthening of Stress-induced Martensite in Ti-1023: Overview and Limits



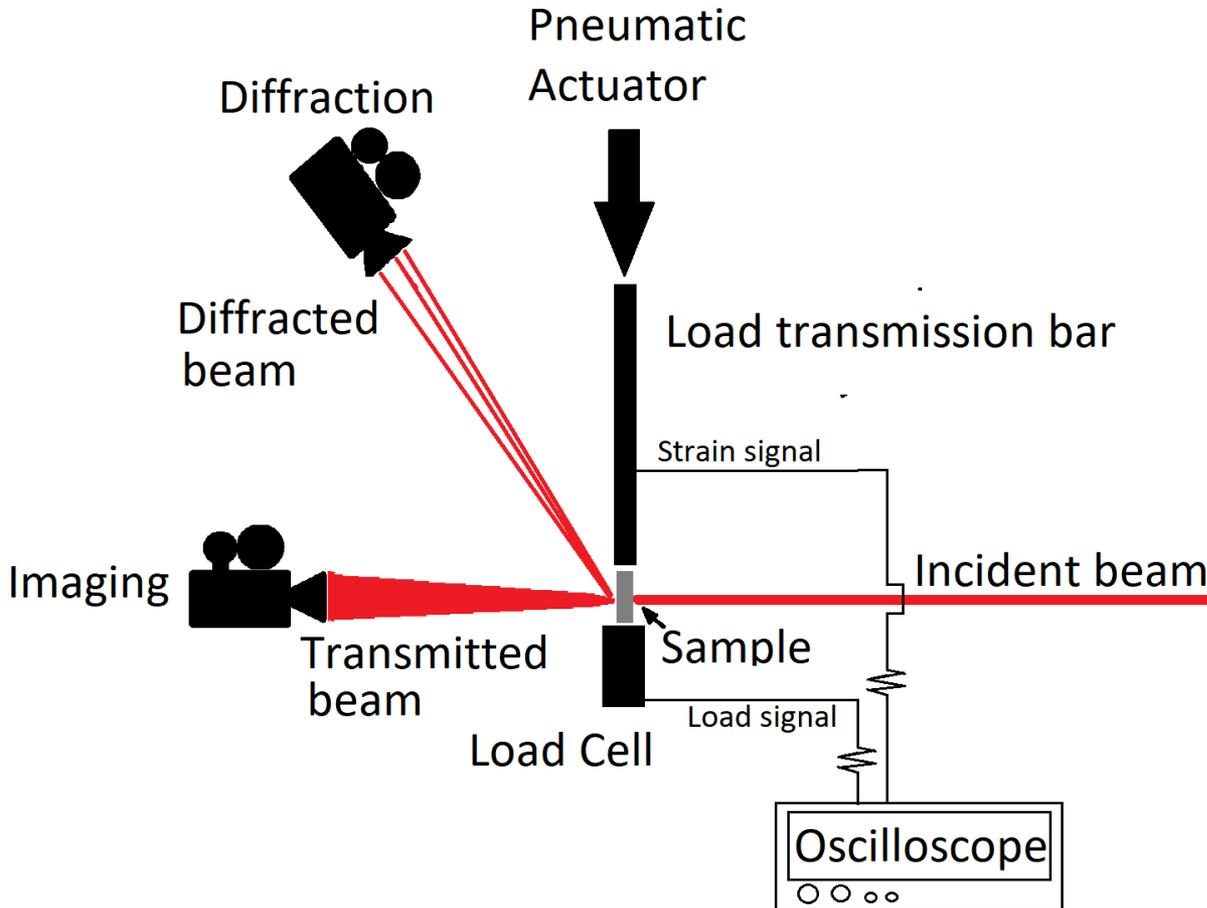
Strain Rate Effects on TRIP Ti-1023



Strain Rate Effect in TWIP Ti-15Mo

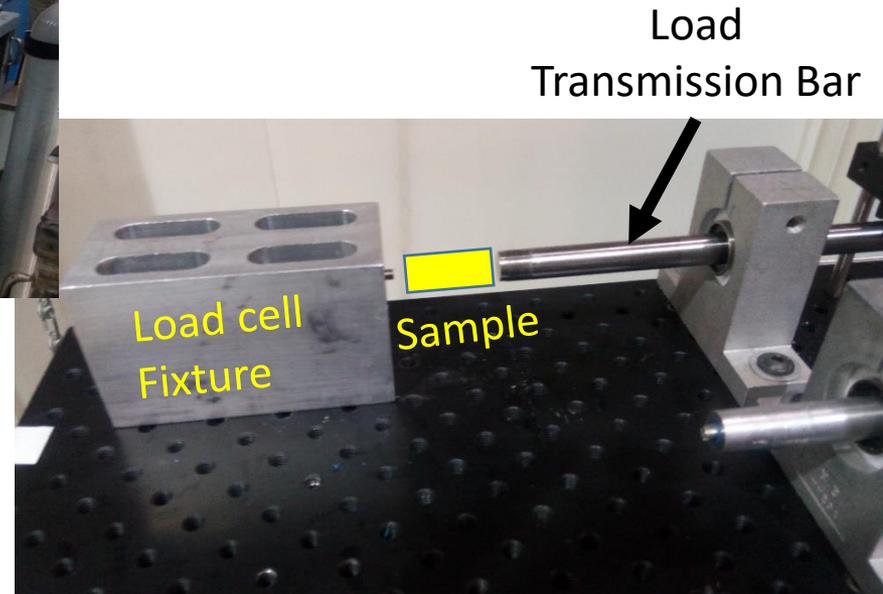
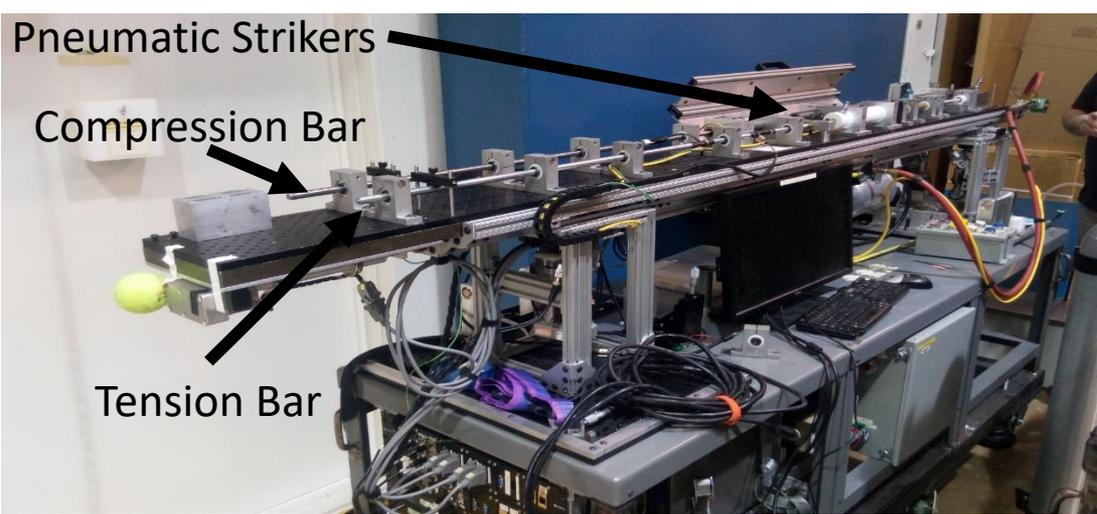
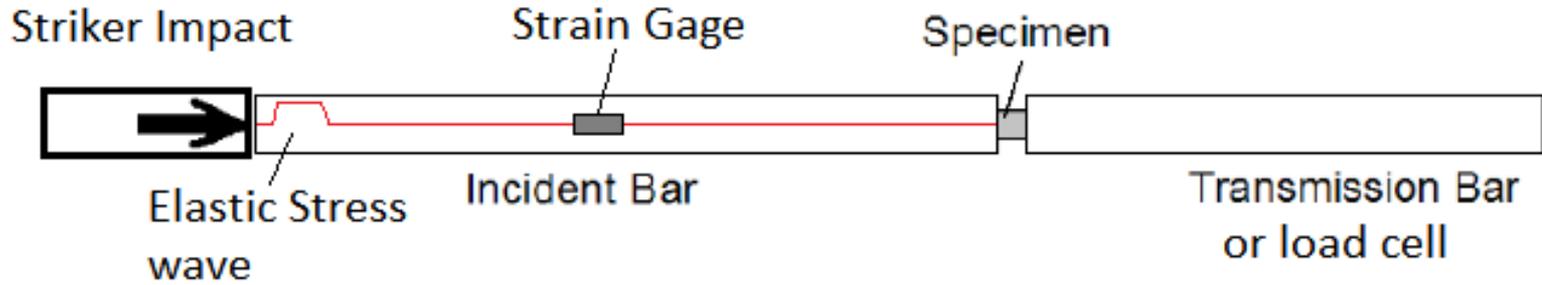


APS High-Rate In-situ Kolsky Bar

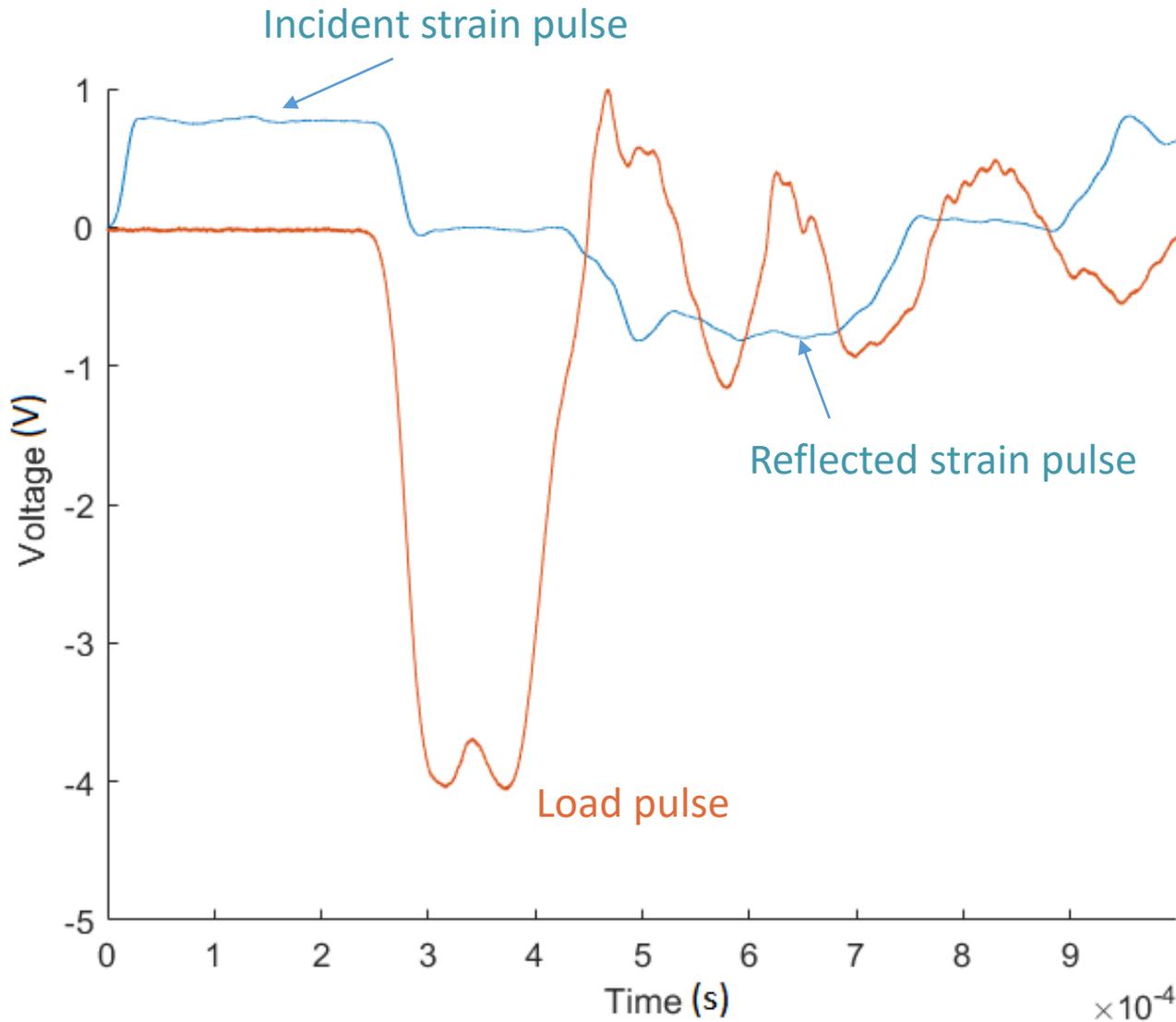


- Strain rates from 10^2 to 10^3 /s in tension and compression
- Simultaneous measurement of diffraction, imaging and stress-strain data
- Time resolved data at high sampling rate, 2×10^{-8} s for mechanical data and 2×10^{-5} s for diffraction data

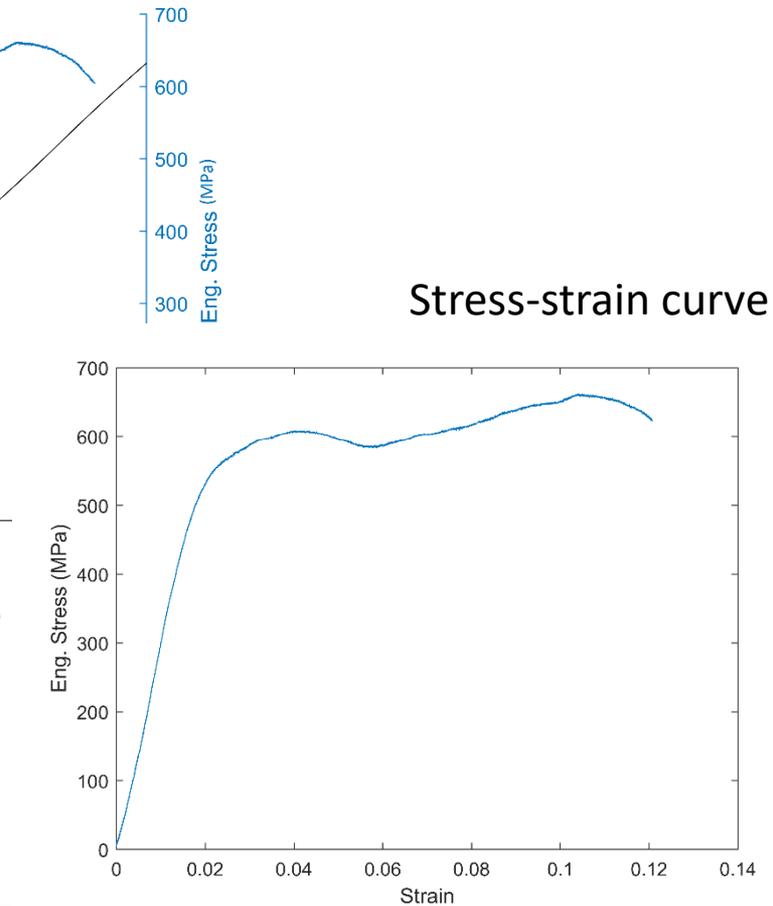
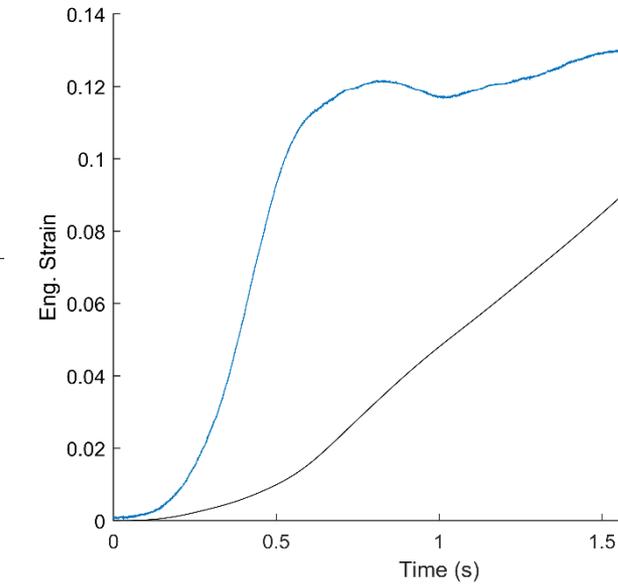
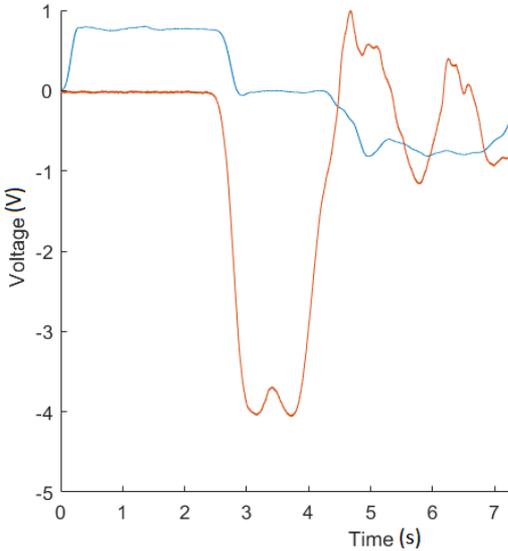
APS Kolsky Bar Setup



Kolsky Bar Raw Data



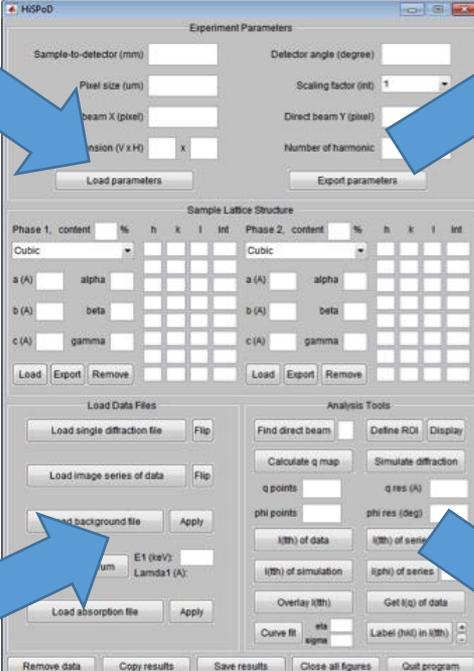
Post-Processing: Mechanical



Post-Processing: Diffraction

Lattice parameter
Space group
Structure factor
Volume fraction
Beam position
Energy spectrum

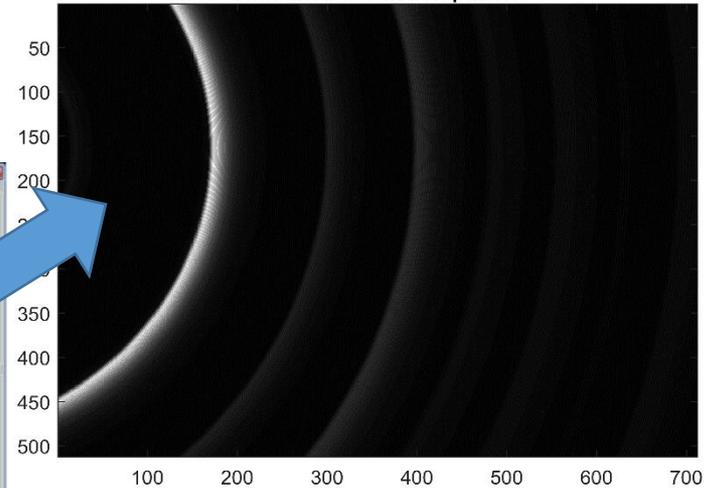
HiSPoD



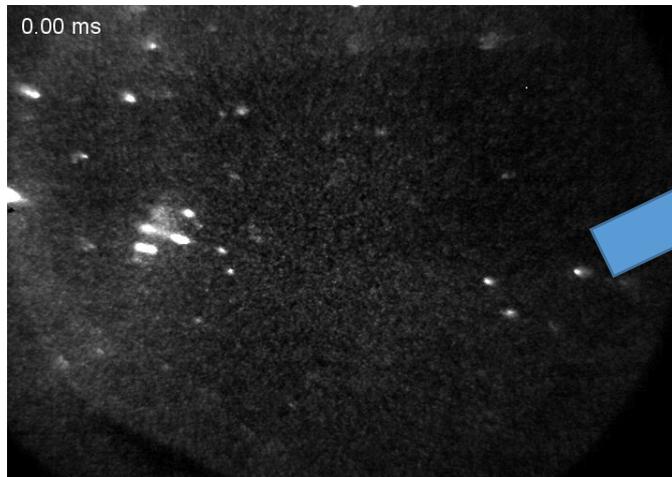
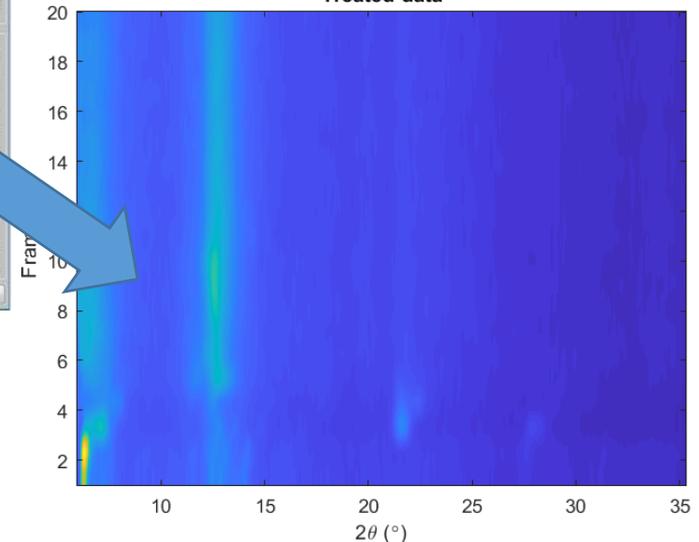
The HiSPoD interface includes sections for:

- Experiment Parameters:** Sample-to-detector (mm), Detector angle (degree), Pixel size (um), Scaling factor (int), Beam X (pixel), Direct beam Y (pixel), Dimension (V x H), Number of harmonic.
- Sample Lattice Structure:** Two phases (Phase 1 and Phase 2) with content, h, k, l, and Int values. Lattice parameters a(A), alpha, b(A), beta, c(A), gamma are provided for each phase.
- Load Data Files:** Load single diffraction file, Load image series of data, Load background file, Load absorption file.
- Analysis Tools:** Find direct beam, Define ROI, Display, Calculate q map, Simulate diffraction, q points, q res (A), phi points, phi res (deg), I(th) of data, I(th) of series, I(th) of simulation, I(phi) of series, Overlay I(th), Get I(q) of data, Curve fit, eta, Label (hkl) in I(th), sigma.

Simulated diffraction pattern

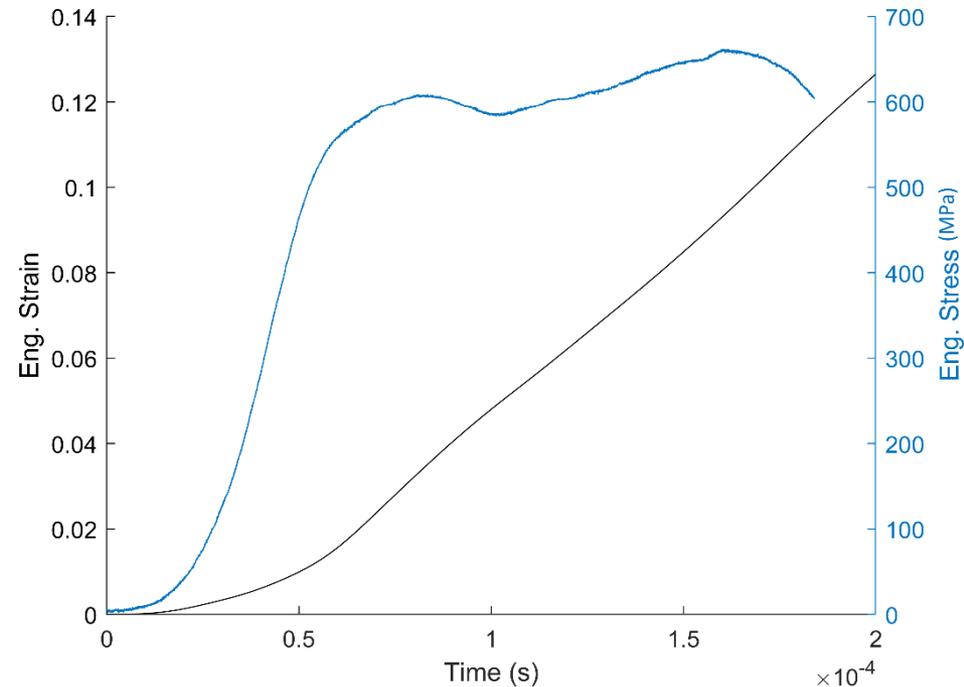
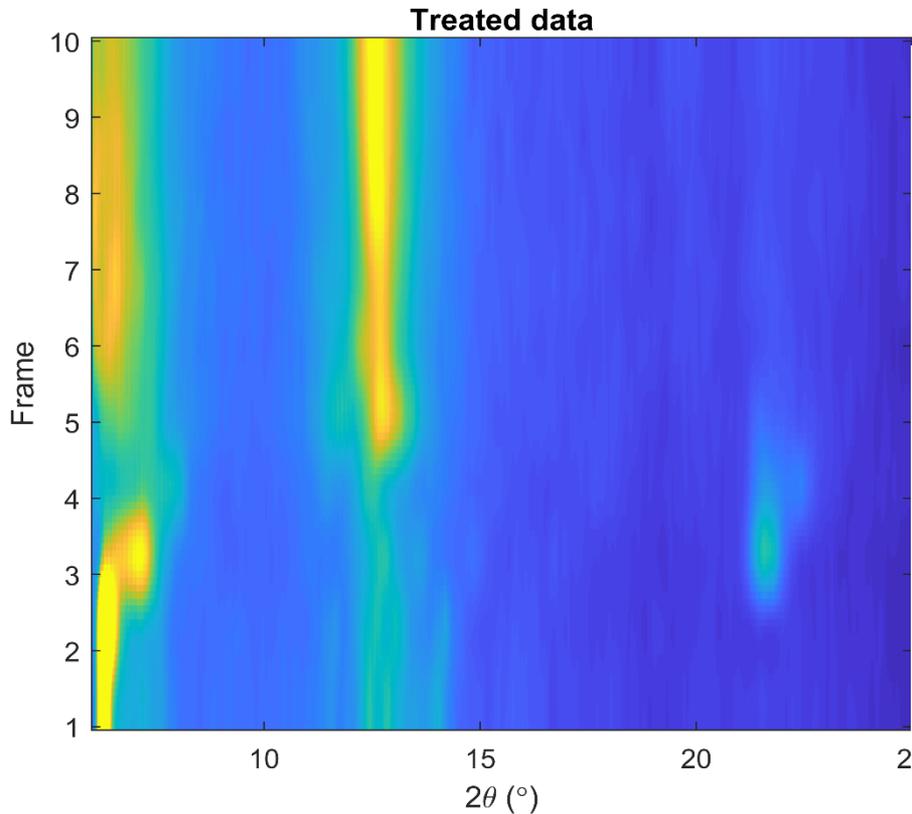


Treated data



Example from a Ti-1023 Sample

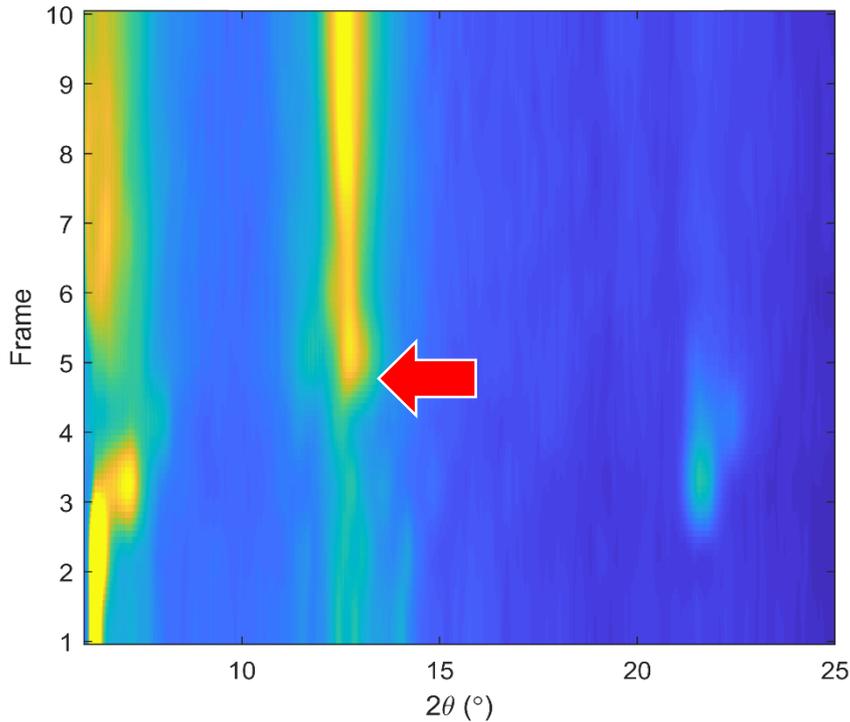
Exact synching remains to be done



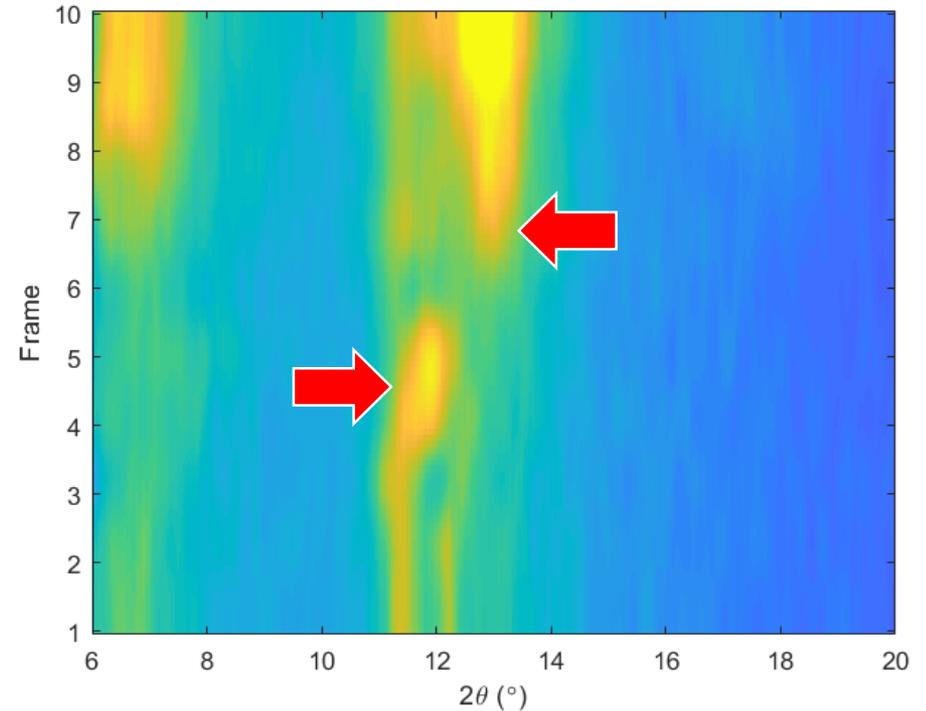
Each frame corresponds to 20 μ s interval

Interpreting Diffraction Data

Ti-1023 sample F45



Ti-1023 sample F10



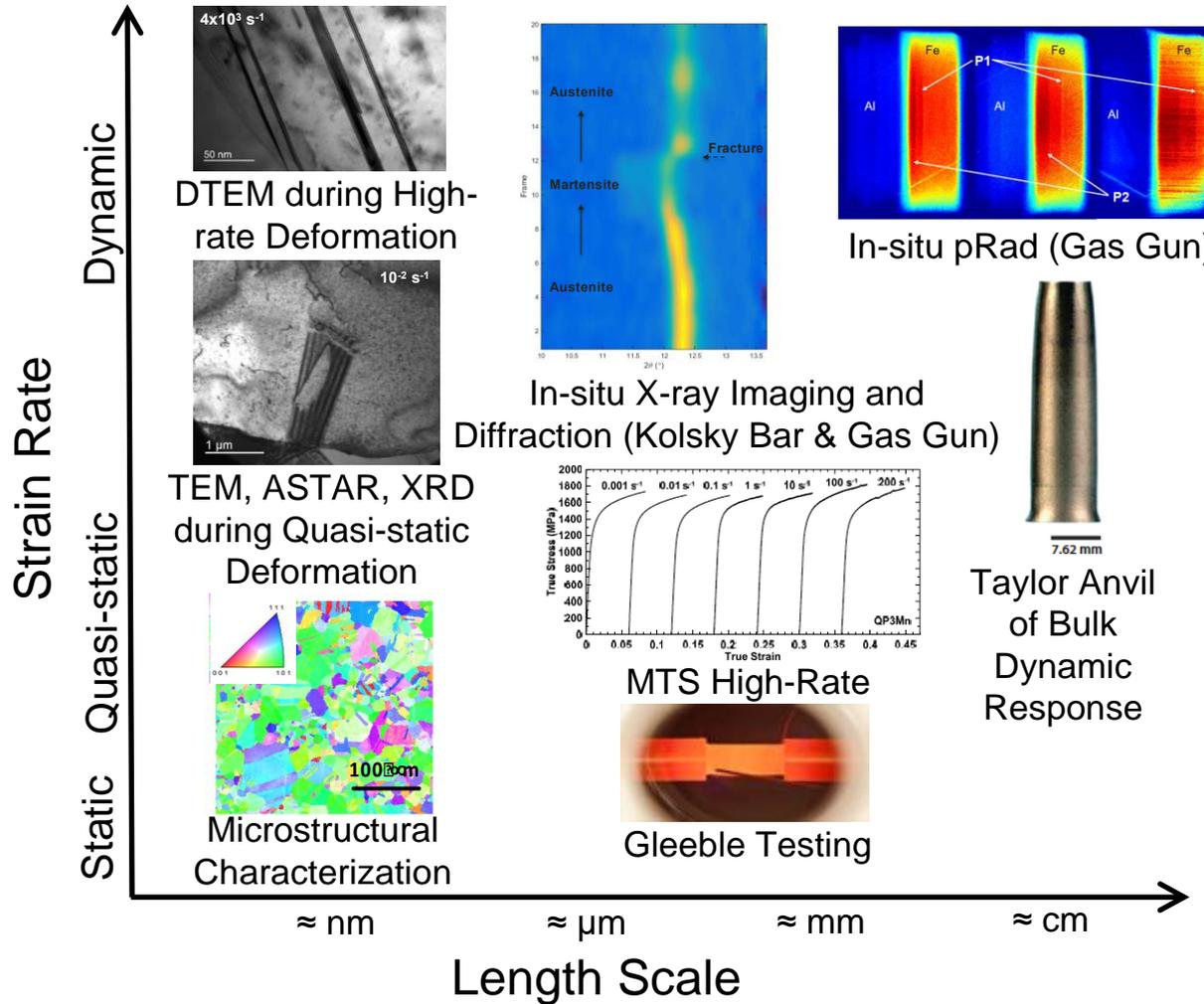
- Shift of peak intensity in 2θ indicates a phase change
- Increase in peak intensity indicates crystallite size refinement (twinning)

Upcoming work

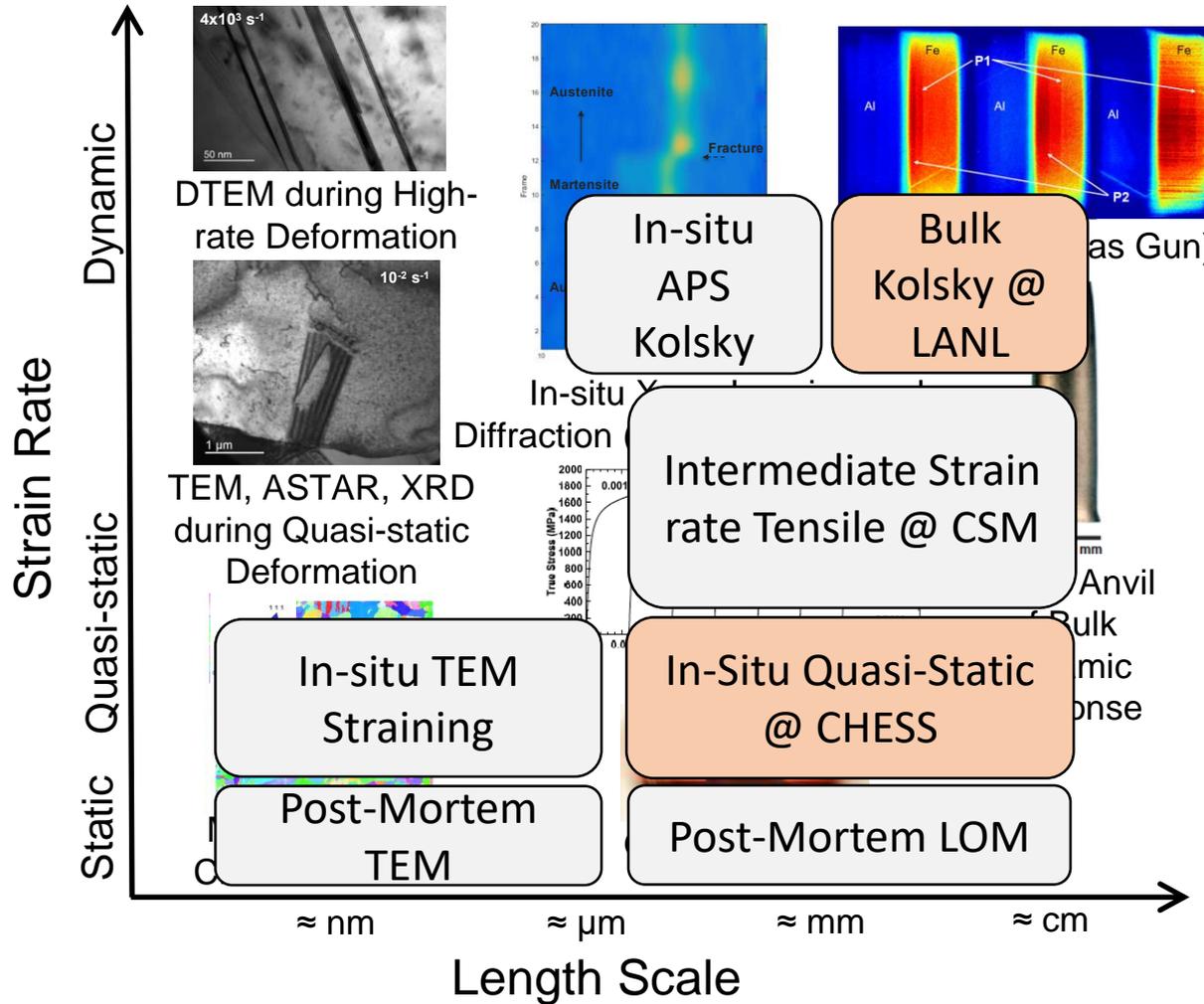


- Synchronize and analyze mechanical/diffraction data from APS
 - ~46 valid data sets: 19 Ti-1023, 12 Ti-15Mo, 10 Ti-3510 and 5 TiZrNb
 - Bulk Kolsky bar testing at LANL
- Finish intermediate strain rate testing of Ti-1023 and Ti-15Mo
- TEM characterization of deformed material
 - In-situ quasi-static TEM straining at LLNL
- Investigate TWIP control mechanisms
- More Beamtime!
 - 2020 Winter/Spring trimester beamtime request approved at APS
 - CHESS proposal for in-situ quasi-static tensile testing has also been approved for the new year (2020)

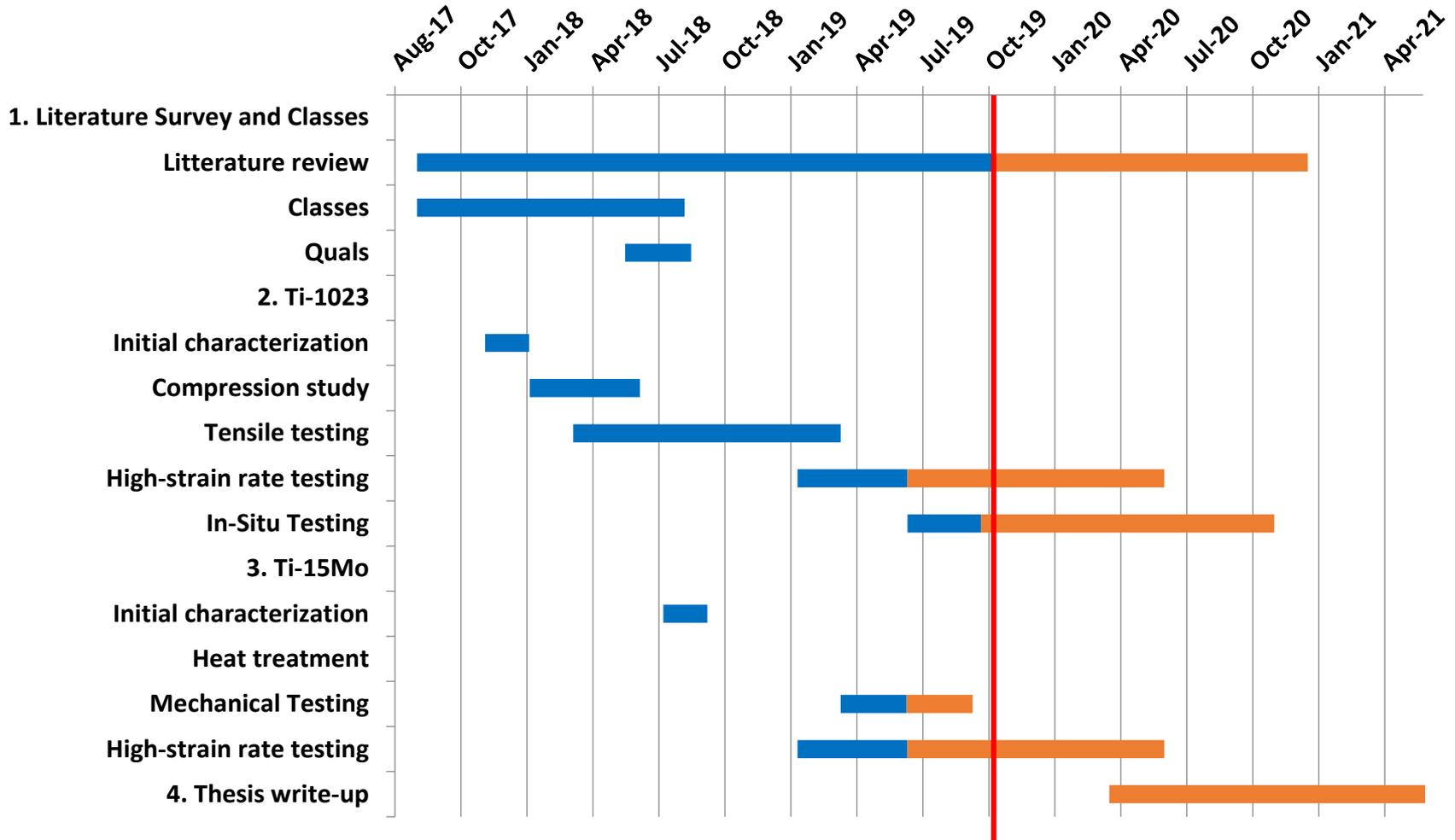
Strain Rate Vs. Length Scale Overview: Current Progress



Strain Rate Vs. Length Scale Overview: Current Progress



Progress



Challenges & Opportunities



- Fine-grained material for Synchrotron experiments is necessary for high-quality diffraction data and consistent small-scale mechanical data
- ω -phase strengthening of TRIP alloys presents a significant opportunity for novel alloy and heat treatment development
- Natural aging of TRIP Ti alloys also presents a novel un-explored avenue in Ti alloy design

Thank you!

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