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

Summer Meeting

June 30th , 2020

- Student: Benjamin Ellyson(Mines)
- Faculty: Prof. Amy Clarke (Mines)
- Industrial Mentors: Austin Mann (Boeing), Clarissa Yablinsky (LANL), John Foltz (ATI)
- Other Participants: Jonah Klemm-Toole (Mines)

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

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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 methodologies and tailor microstructures and properties
- Benefit: Novel titanium alloys for blast and crash resistant applications

Recent Progress

- Transmission electron microscopy (TEM) of aged Ti-10V-2Fe-3Al (wt.%) (Ti-10-2-3) tensile specimens is underway
- New high-strength low-temperature aged condition discovered for Ti-10-2-3
- Progress has been made in processing in-situ Advanced Photon Source (APS) data

Metrics

Description	% Complete	Status
1. Literature review	80%	● (green)
2. Quasi-static mechanical characterization of Ti-10-2-3 and Ti-15Mo	95%	● (green)
3. Dynamic testing of Ti-10-2-3 and Ti-15Mo	70%	● (green)
4. Microstructural characterization of pre- and post-deformation samples	70%	● (green)
5. *In situ* characterization of microstructural evolution during deformation	40%	● (yellow)

Industrial Relevance: Development of Blast Resistant Materials for the Navy

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- **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

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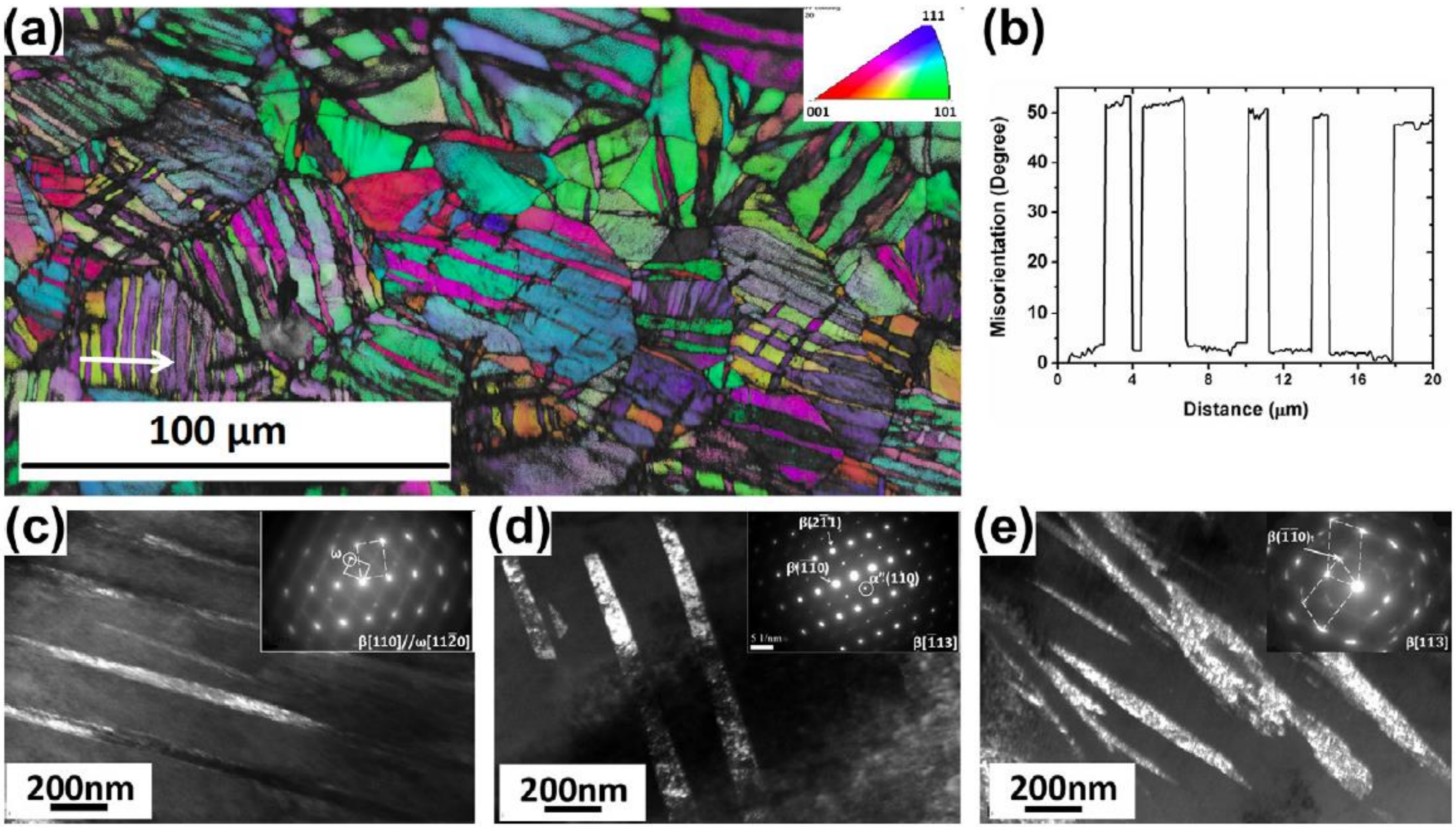
Compression at 10^{-3} to 0.18 true strain

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

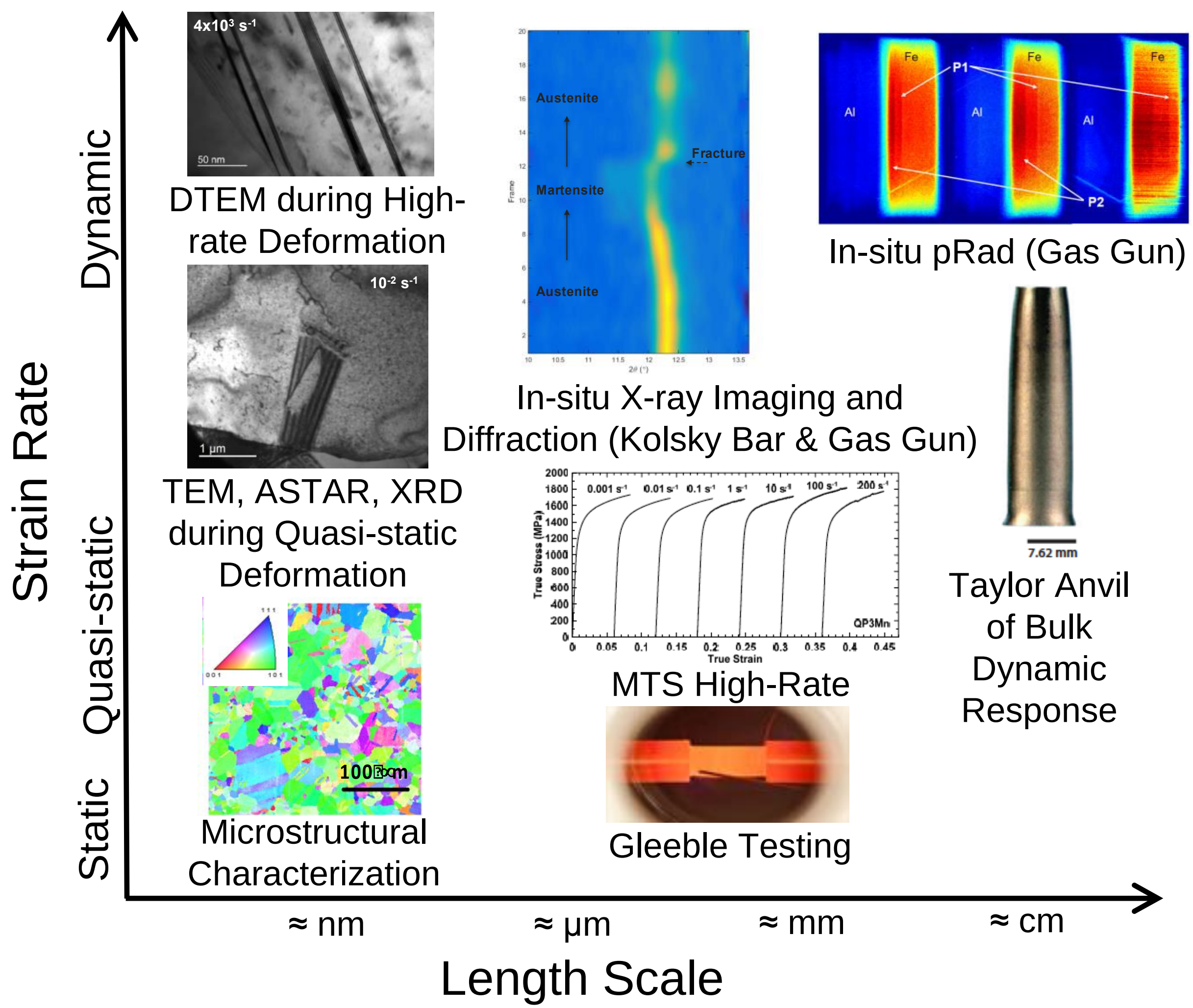


Deformation-induced ω phase | **Deformation-induced α'' phase** | **{112}<111> β mechanical twinning**

TRIP: Transformation Induced Plasticity

TWIP: TWinning Induced Plasticity

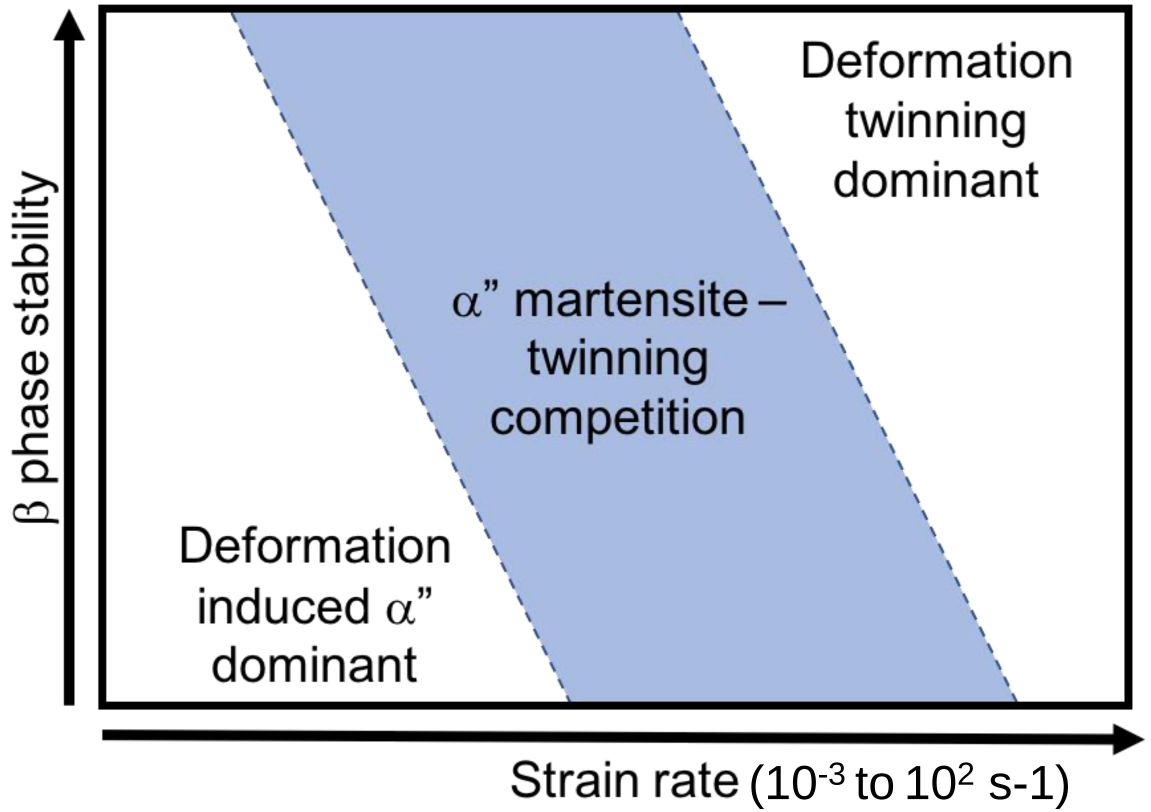
Multi-scale Studies of TRIP/TWIP During High Rate Deformation



TRIP: Transformation Induced Plasticity, TWIP: TWinning Induced Plasticity, DTEM: Dynamic Transmission Electron Microscopy, TEM: Transmission Electron Microscopy, XRD: X-Ray Diffraction, pRad: Proton Radiography, ASTAR: Automatic Crystal Orientation and Phase Mapping , MTS: Materials Test Systems

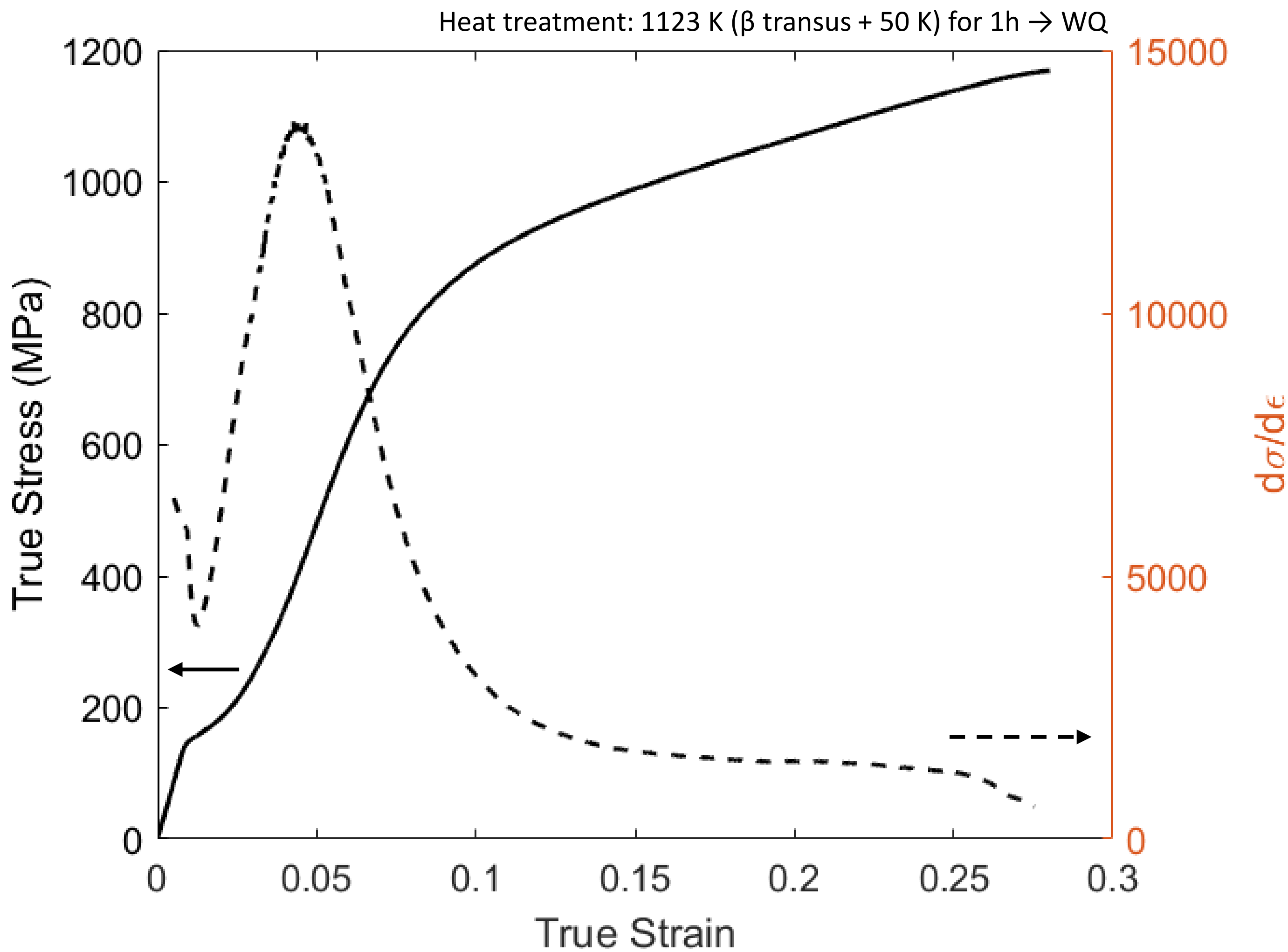
The Effect of Strain Rate on Deformation Mechanisms During Compression of a Ti-10V-3Fe-3Al (wt.%) Alloy

- **Deformation mechanisms present at all strain rates :**
  - Stress-induced α" martensite
  - {332}<113> β twinning
  - Stress-induced ω phase
  - Slip

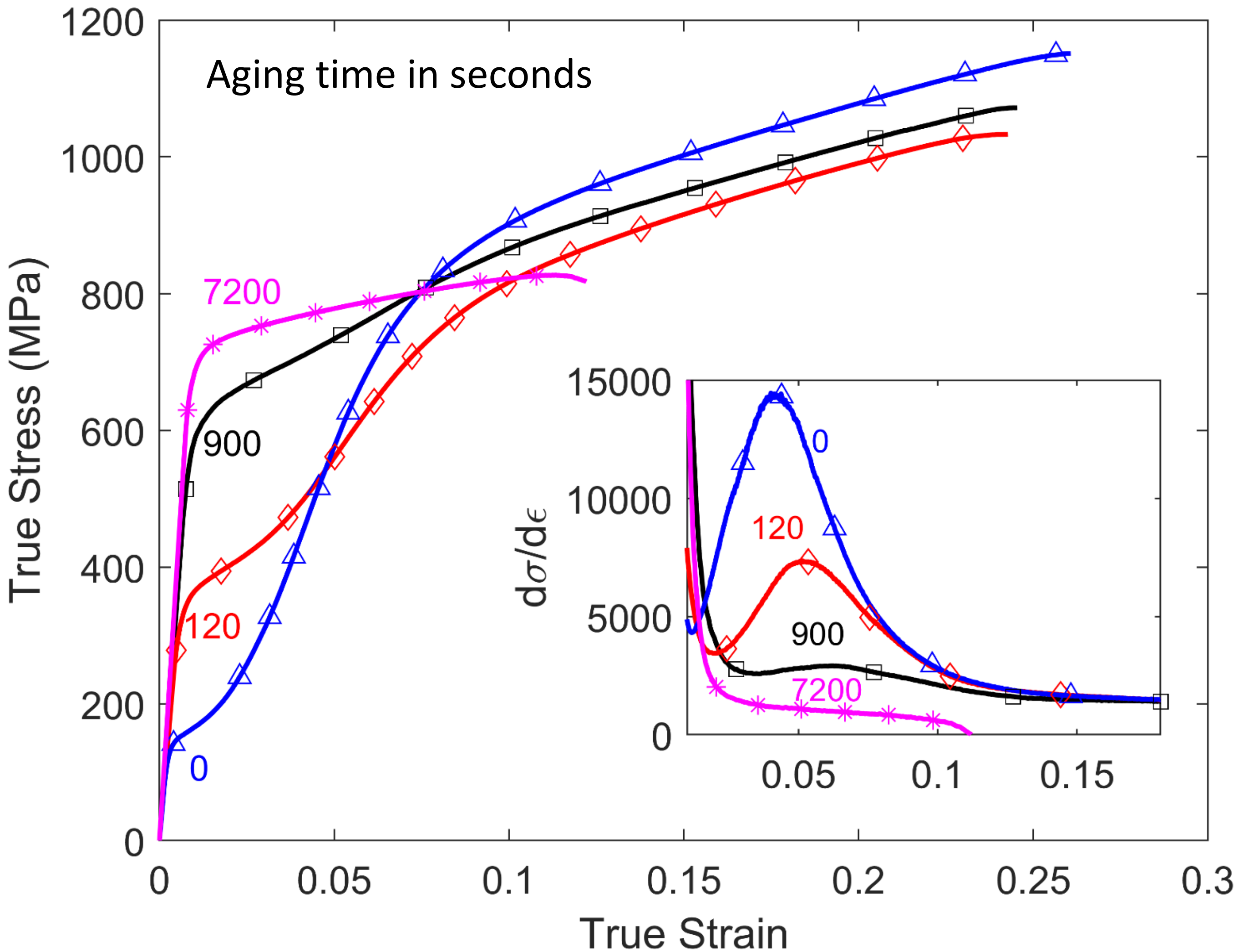


Ahmed, M., et al. 104 Acta Materialia (2016): 190-200

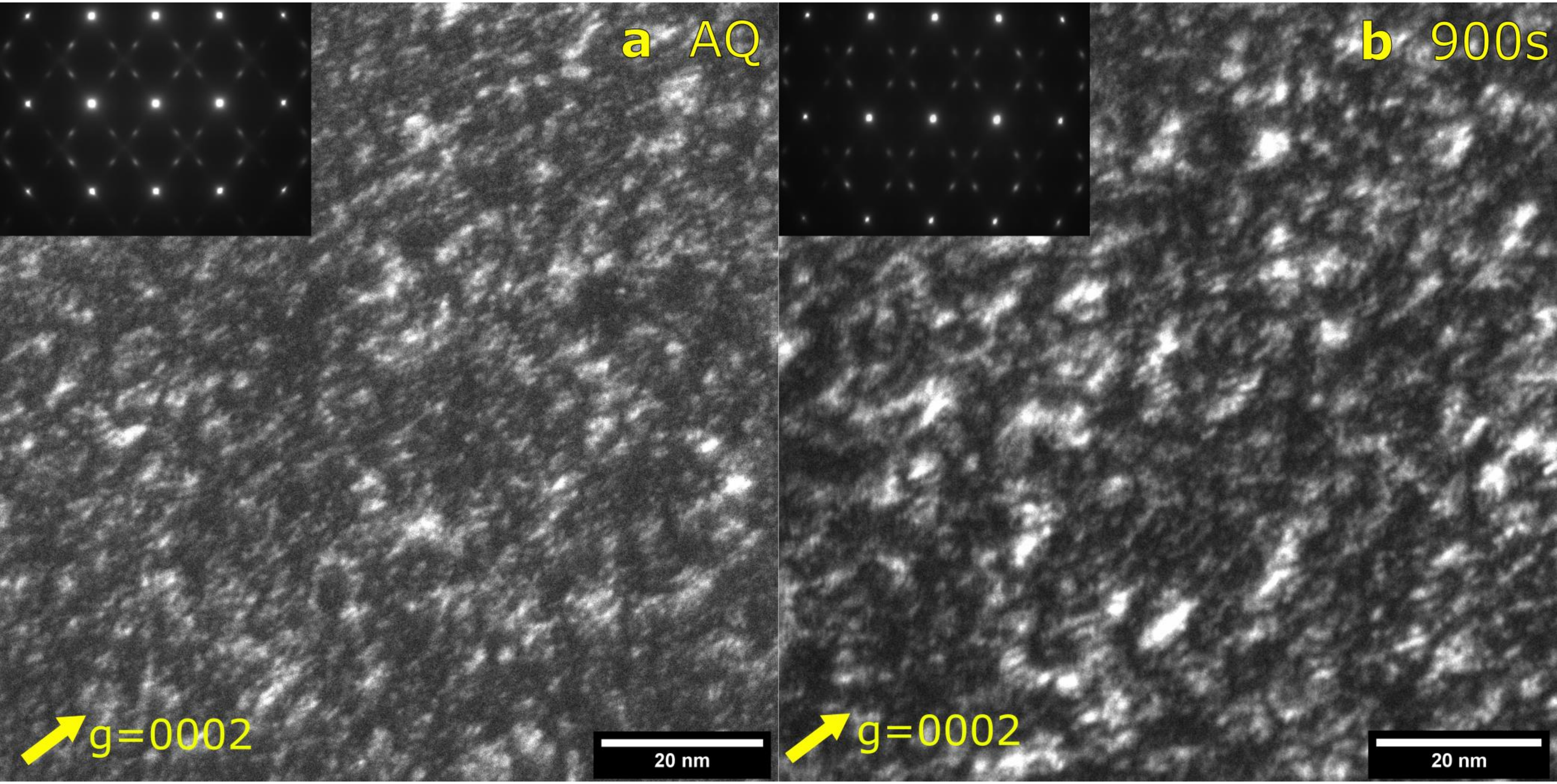
As-Quenched Quasi-static Tension of Ti-10-2-3



Artificial Aging of Ti-10-2-3 at 423K and Quasi-static Tensile Testing

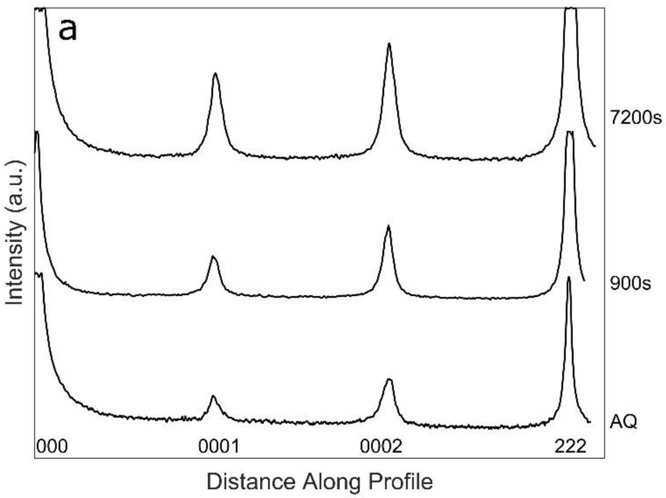


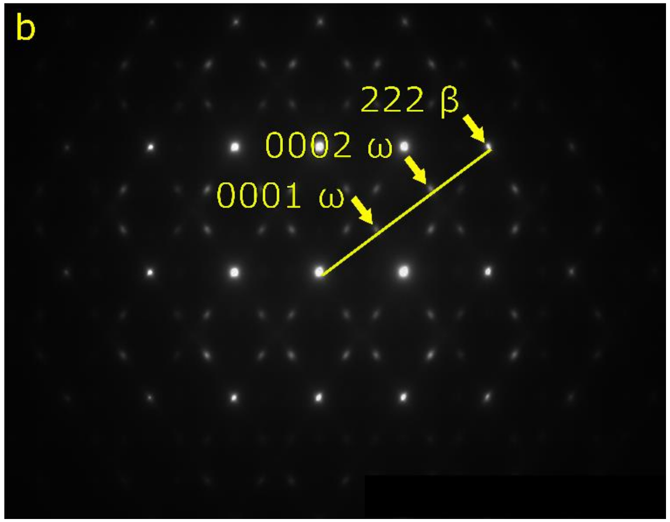
ω-phase in Aged Ti-10-2-3



Ex-situ Aging

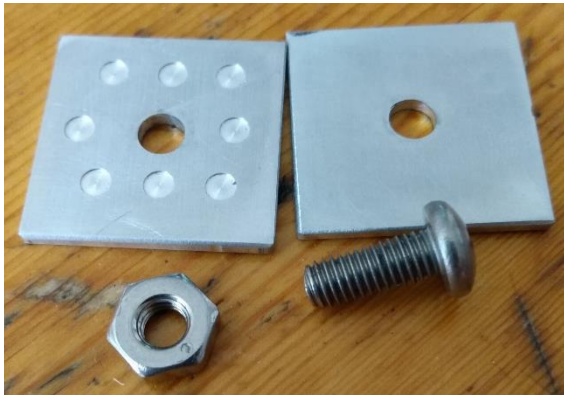
- Developed an ex-situ solution to ω-phase aging characterization





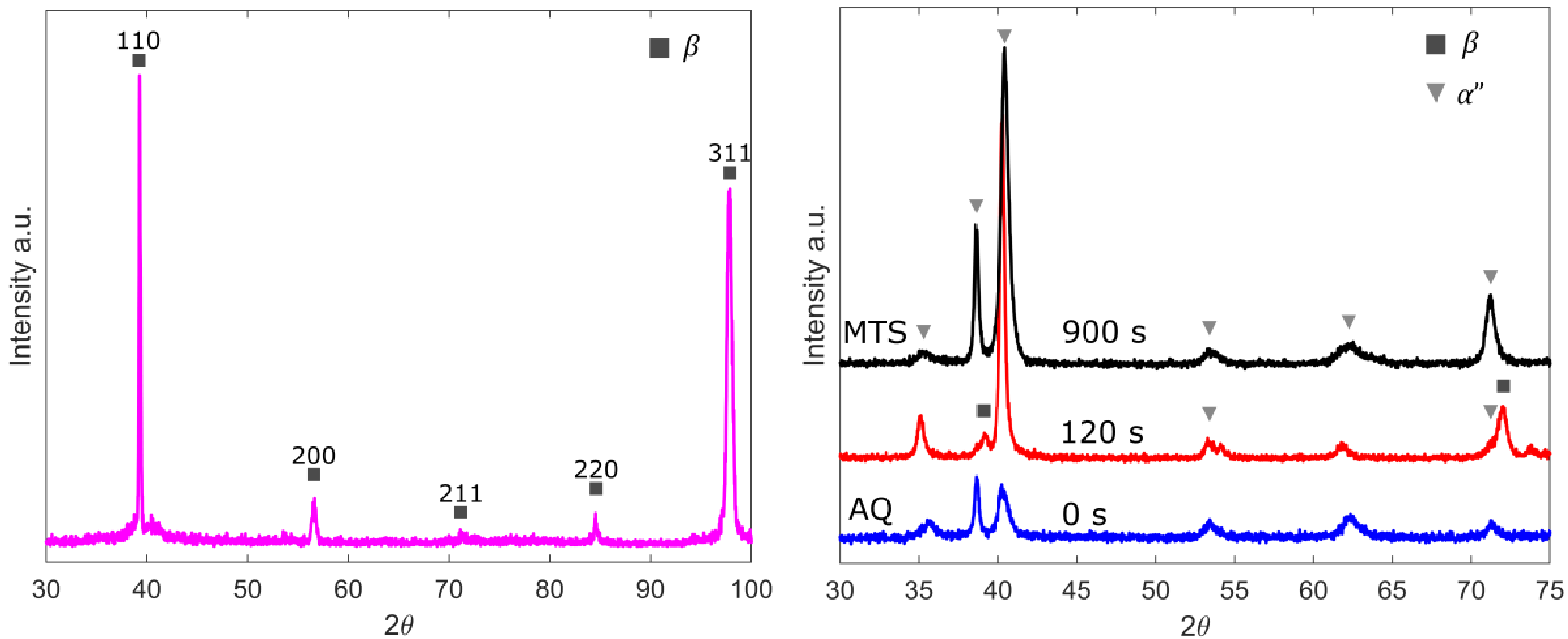
Problem : “Comparability” of diffraction and DF measurements

Solution : Ex-situ TEM aging setup



Transition to TRIP Inhibited (TI) Condition

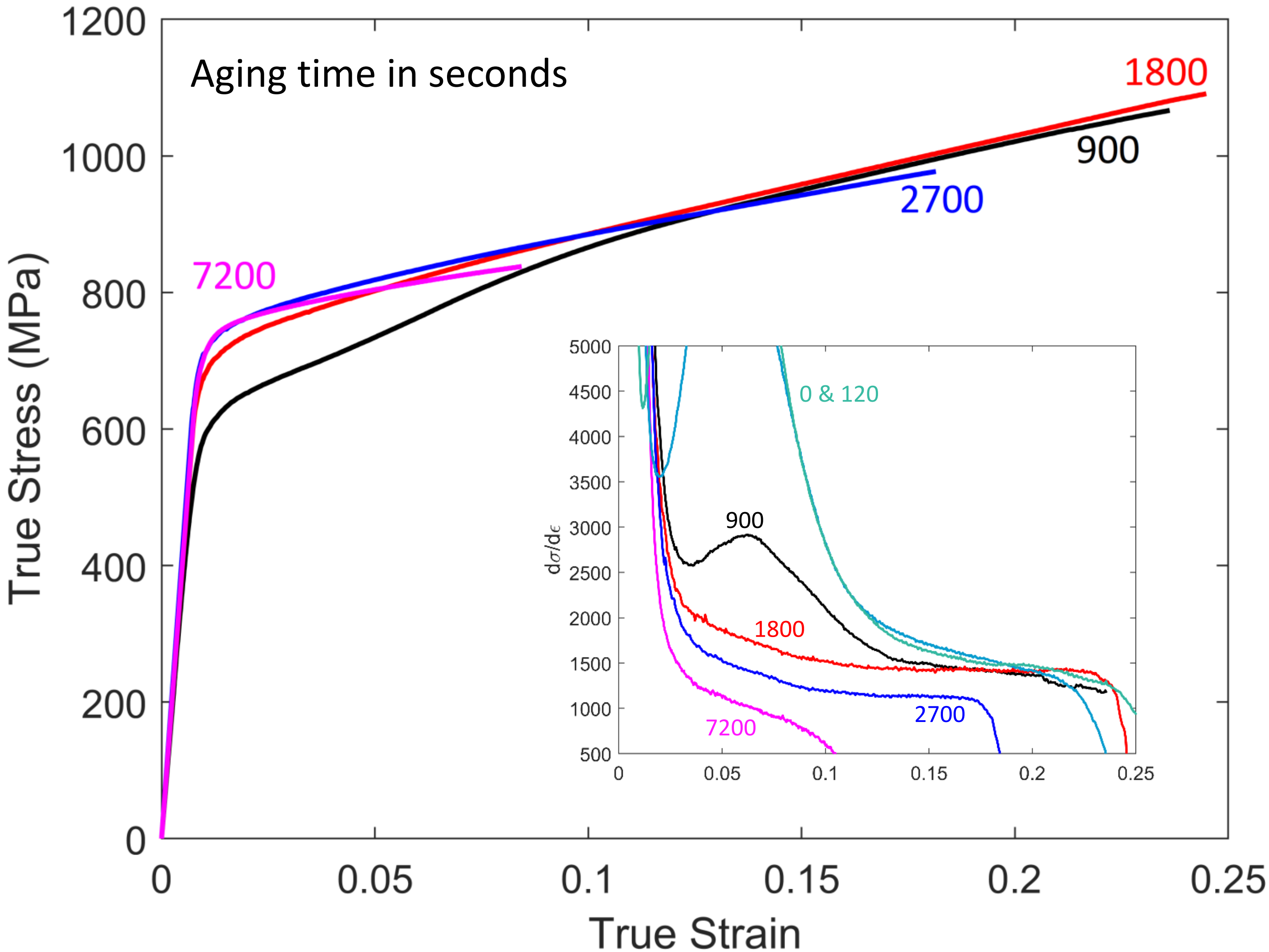
Aged 7200 s @ 423 K & fractured in tension at 10^{-3}



Similar to results reported in :
Chen, Wei, et al. *Acta Materialia,* 2019, 170, 187-204.
Lai, M. J., Tong Li, and Dierk Raabe. *Acta Materialia,* 2018, 151, 67-77.
Mantri, S. A., et al. *Scripta Materialia,* 2017, 130, 69-73.
Wang, Weilin, et al. *Materials & Design,* 2020, 186, 108282.

Over-aging of Ti-10-2-3 inhibits stress-induced martensite and causes dislocation bands to form

Transition to TRIP Inhibited



Center Proprietary – Terms of CANFSA Membership Agreement Apply

ω-phase Strengthening of Stress-induced Martensite in Ti-10-2-3: Overview and Limits

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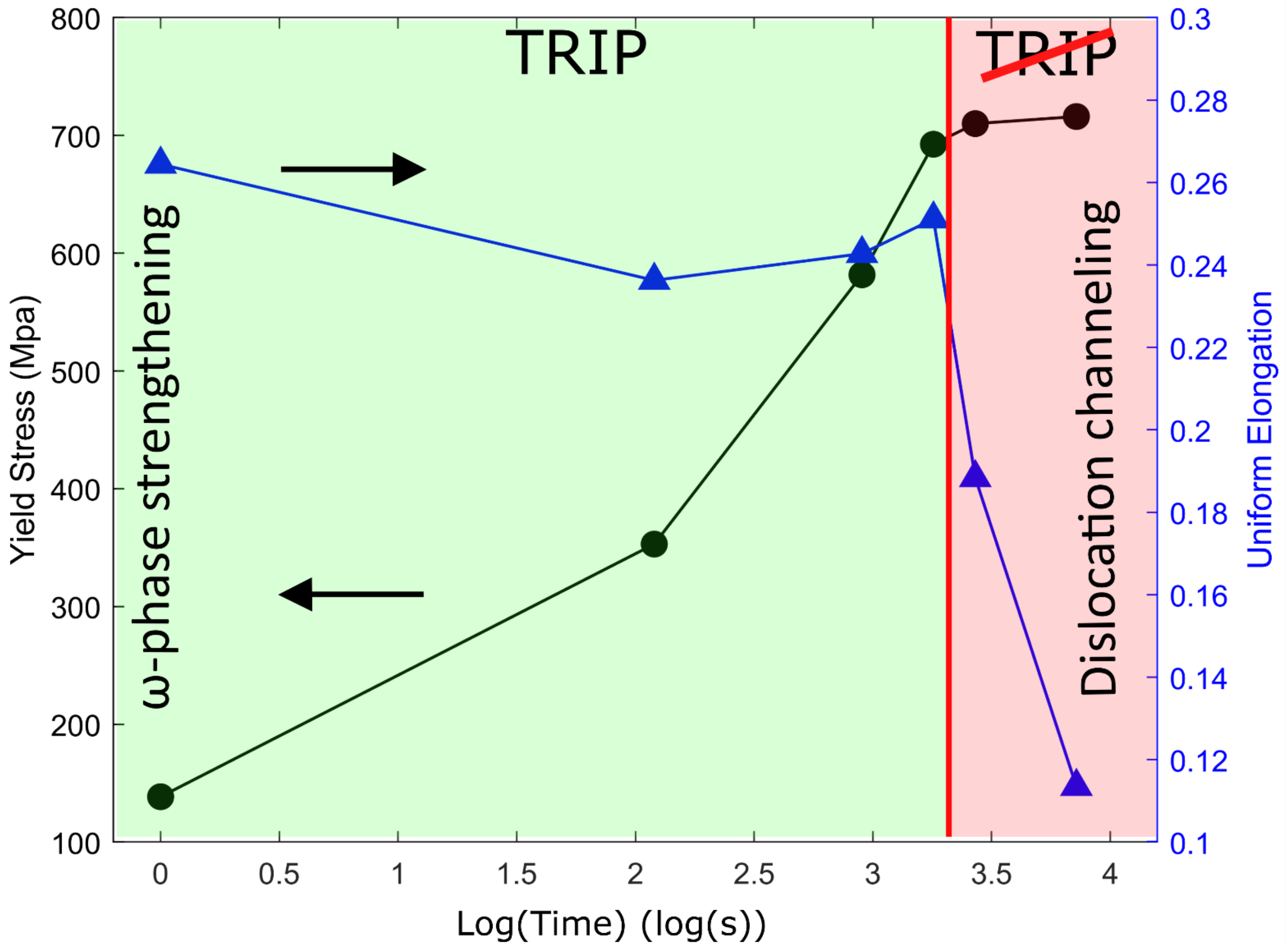
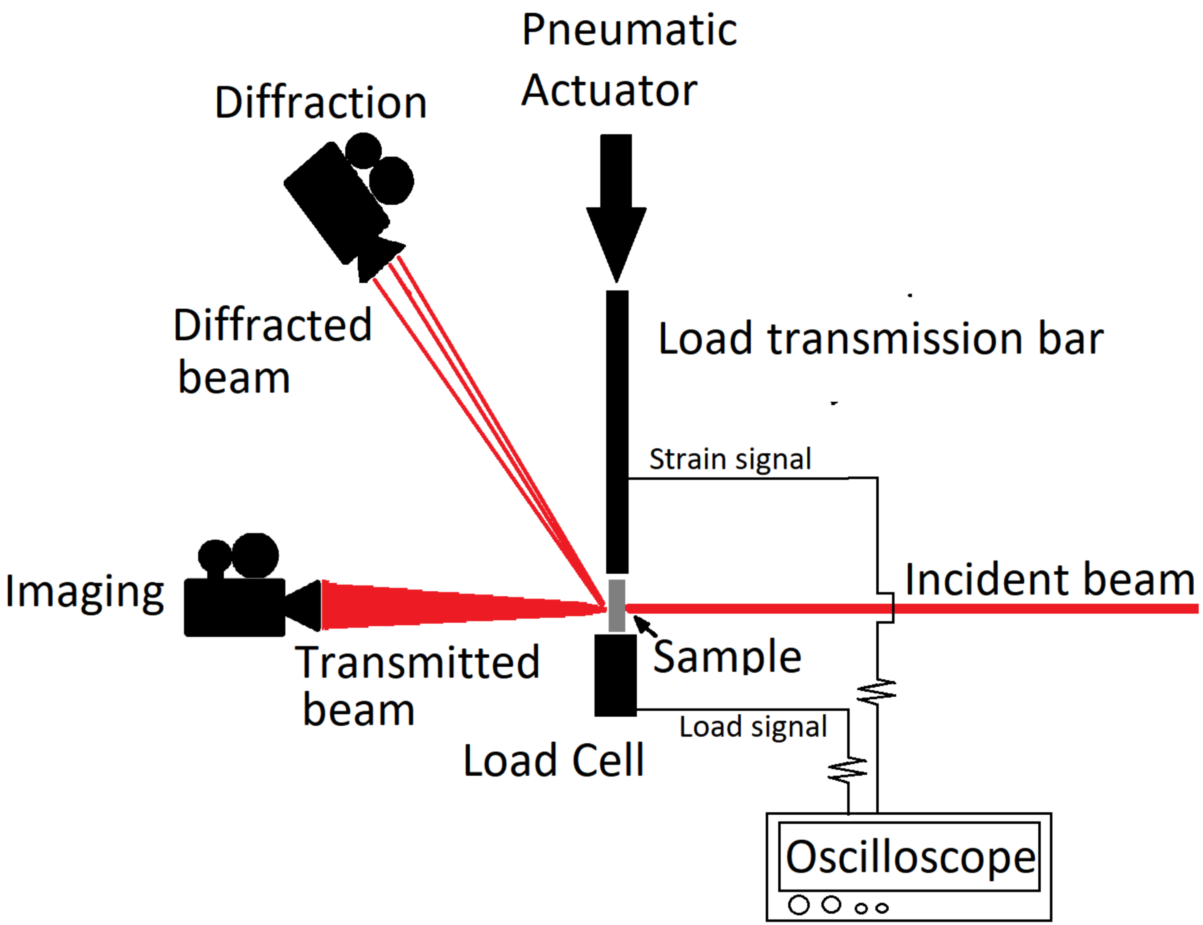


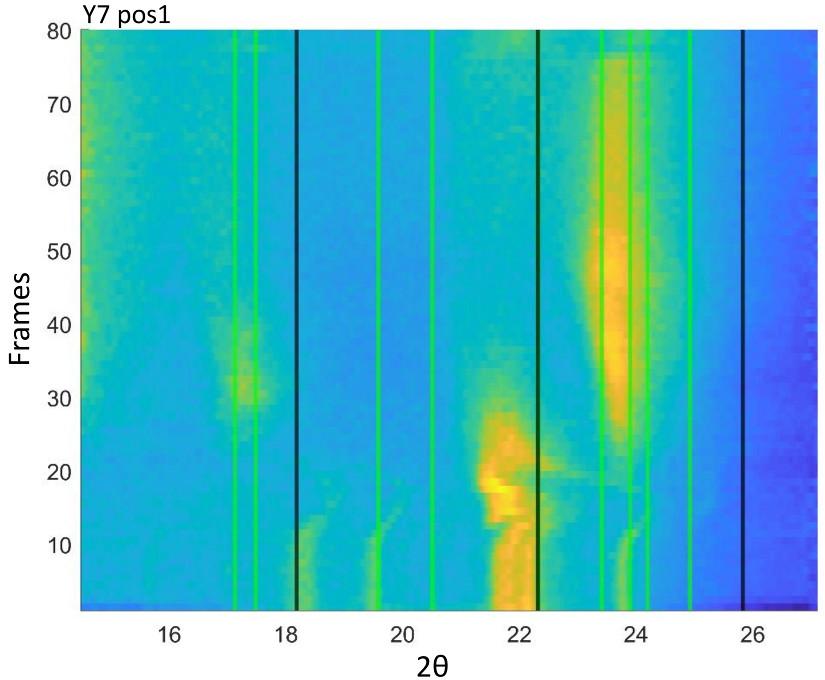
APS High-Rate In-situ Kolsky Bar



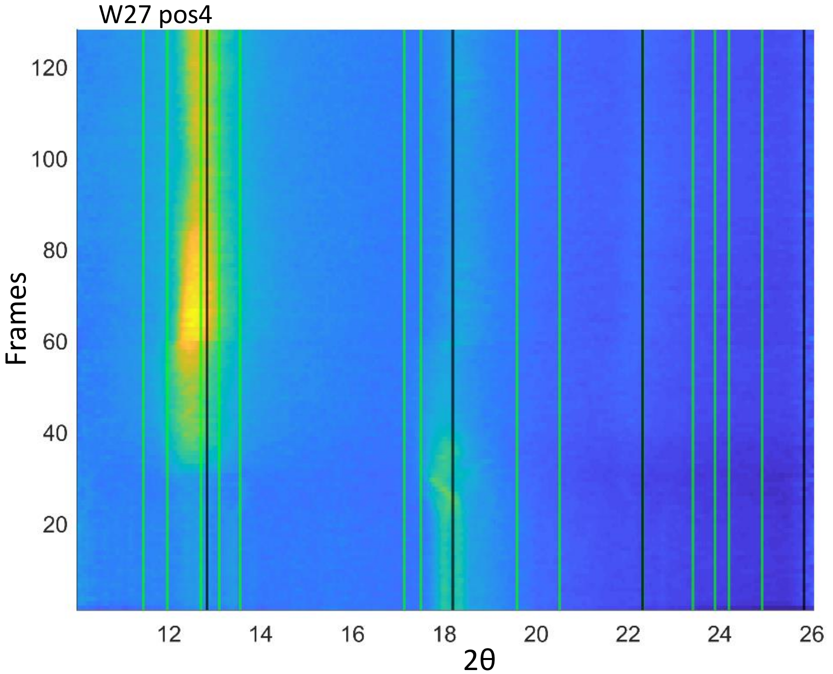
- Strain rates from 10^2 to 10^3/s in tension and compression
- Simultaneous measurement of diffraction, imaging and stress-strain data
- Time resolved data at high sampling rate, 2×10^{-8} s for mechanical data and 2×10^{-5} s for diffraction data

Twinning vs Transformation

Roughly:
Loading begins at frame 25
Yield occurs at frame 30
Fracture occurs at frame 80



Fresh-quenched Ti-1023 exhibits transformation



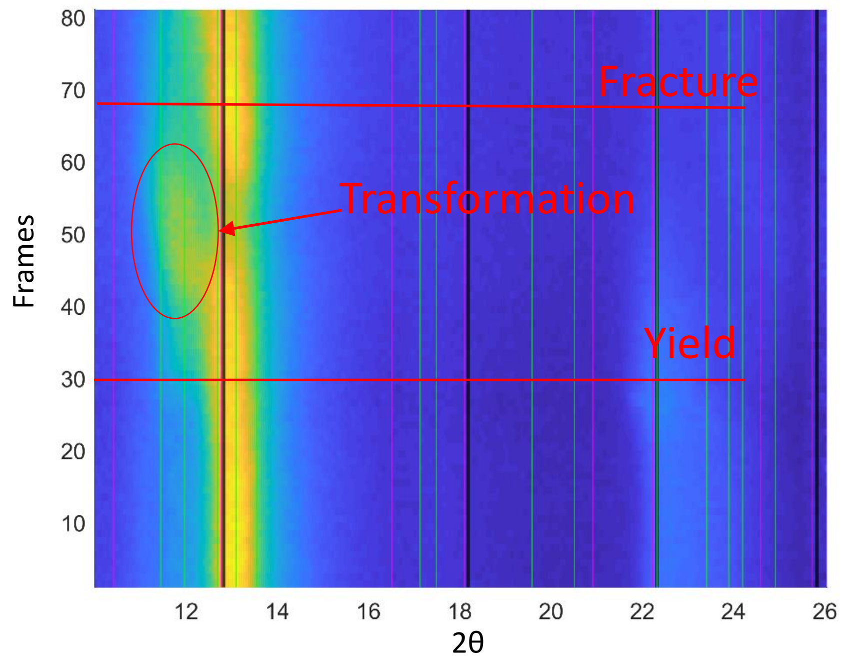
As-quenched Ti-15Mo exhibits deformation twinning

Green: α”
Black: β

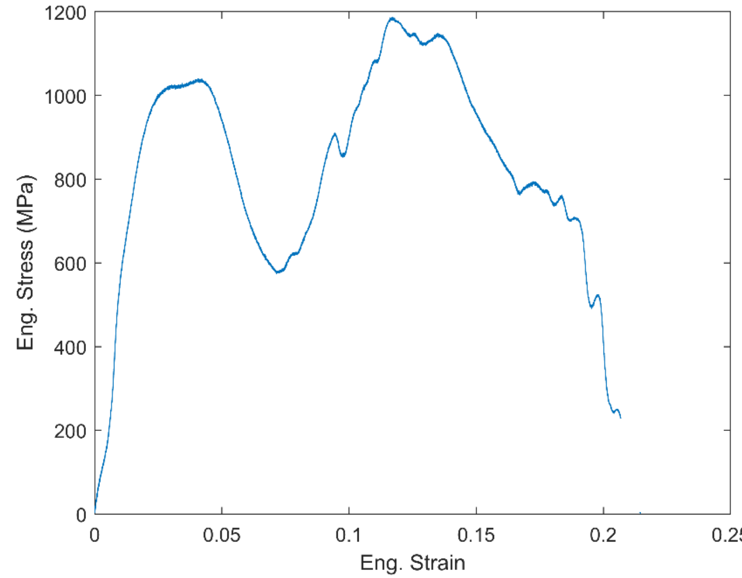
Deformation at ~1000 s^{-1}

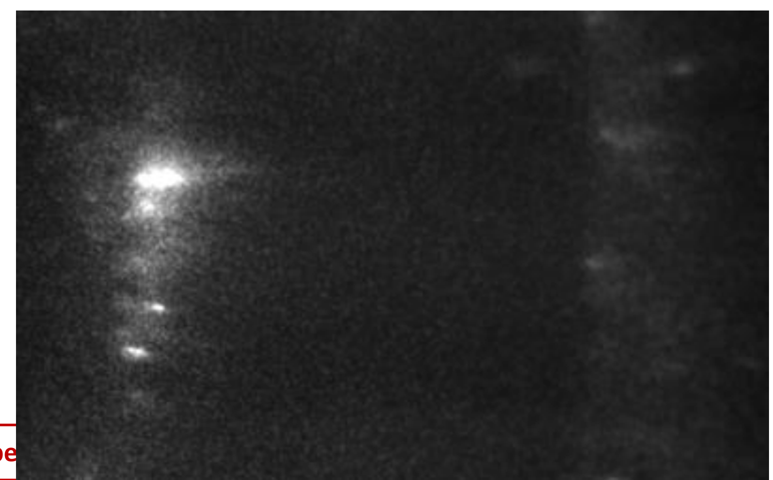
APS Initial Results

Evidence of reversion at higher strain?



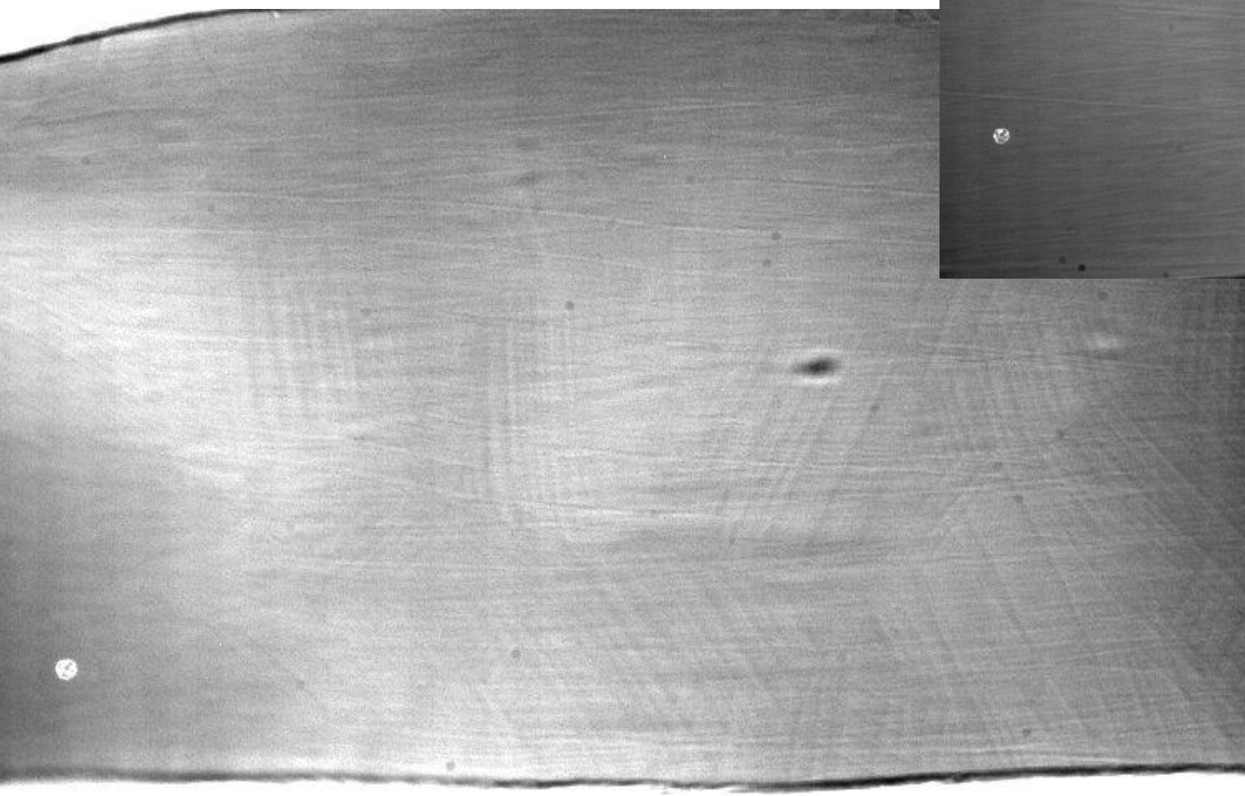
Ti-1023 in MTS condition deformed in tension at $1000s^{-1}$

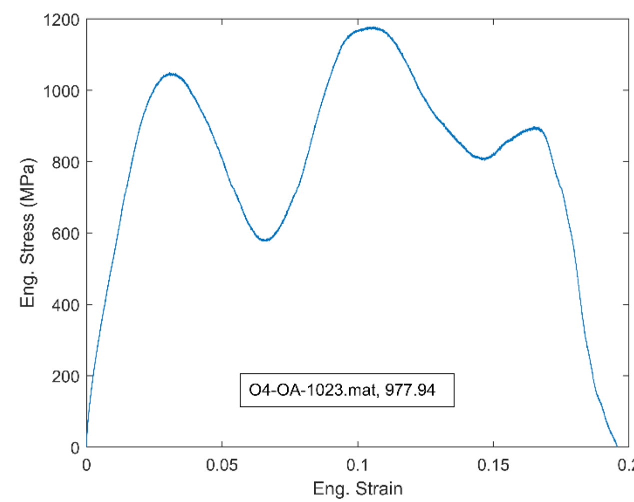




Twinning in TI Radiography

Evidence of deformation twinning at high strain rate seems to cause TWIP and increased ductility!





Progress

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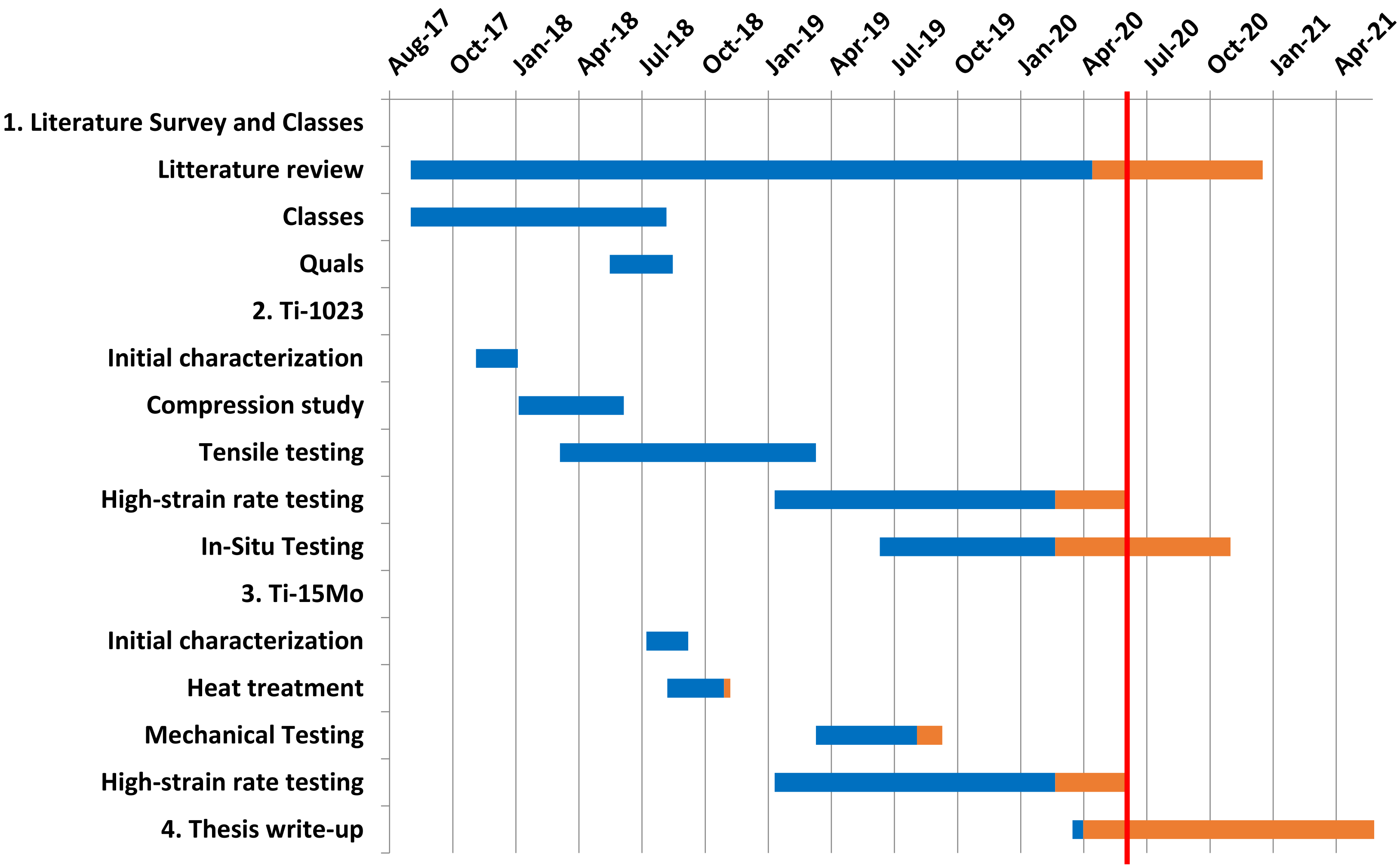


Challenges & Opportunities

Challenges

- COVID-19 has put a pause to many collaborations
- Proper EBSD indexing of martensite remains to be accomplished

Opportunities

- A publication on the effect of low temperature aging on the strength/ductility of Ti-10-2-3 has been prepared and is ready for submission
- APT reconstructions from Prof. Banerjee are on their way!
- High-strain rate microstructural evolution seems to be markedly different from quasi-static in Ti-1023

Thank you!

Benjamin Ellyson

bellyson@mines.edu

EBSD Trial and Error

- Martensite is very close in symmetry to BCC : classic indexing cannot differ between the two
- Fine scale of transformation product leads to significant pattern overlap
- β-Titanium alloys are very prone to surface deformation, which strongly affects EBSD reliability
- New electrolyte has proven very reliable at producing high-quality electro-polished surface (perchloric, hydrochloric, methanol & butoxy-ethanol solution)
- New spherical indexing algorithm from De Graef et al. is much more robust to pattern overlap and pseudo-symmetry

Synthesis of Tested Materials

Alloy	State	Strain Rate		
		~10^{-3}	~10^{-1}	~10^{2}-10^{3}
1023	AQ	CSM/CHESS	CSM	LANL/APS
1023	MTS	CSM/CHESS	CSM	LANL/APS
1023	TI	CSM/CHESS	CSM	APS
15Mo	AQ	CSM/CHESS	CSM	LANL/APS
12Mo	AQ	X	X	APS

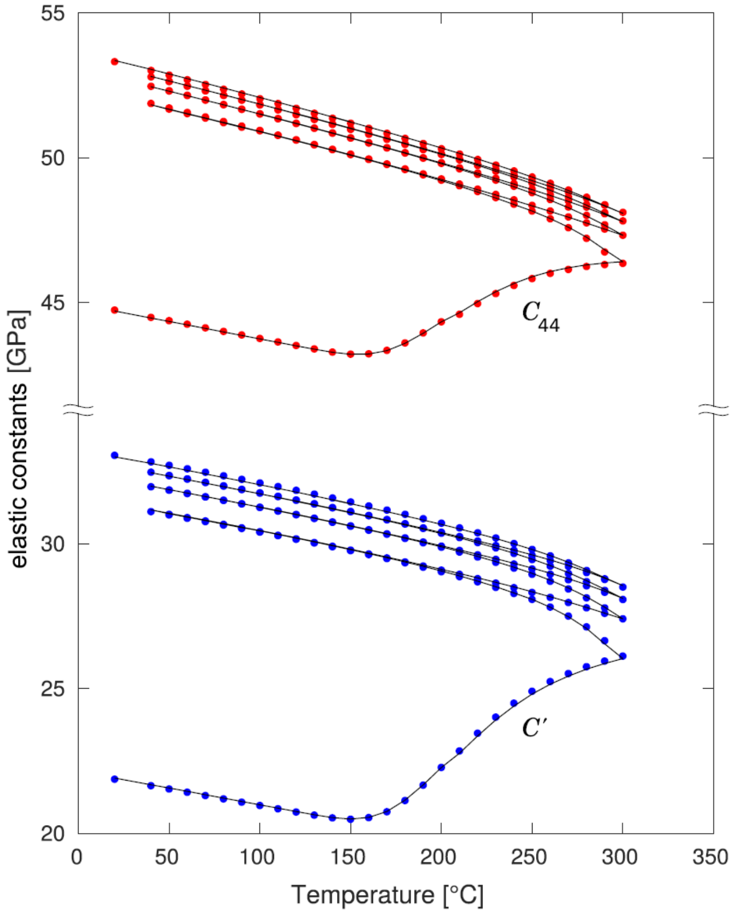
2 Over-arching studies are being conducted:

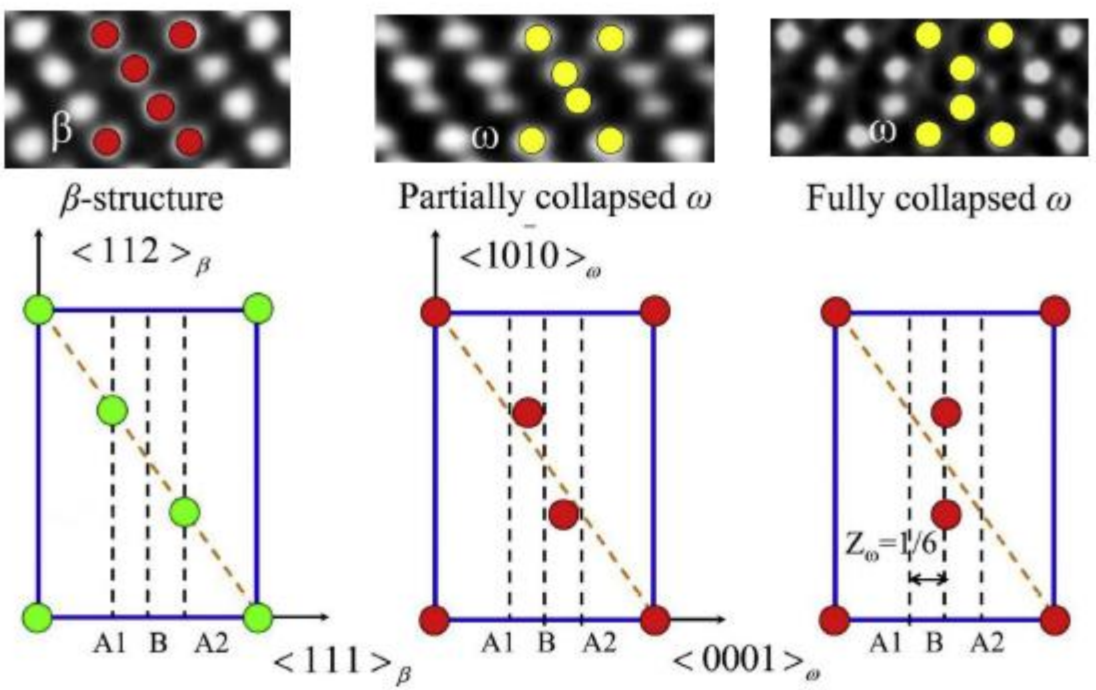
1. **Effects of low-temperature aging and strain rate on deformation of Ti-1023**
2. **Effects of composition on deformation at high rate in Ti-Mo system**

Secondary studies accompanying current work:

1. Effect of strain rate on deformation structure of Ti-15Mo
2. Investigating the effect of the low-temperature aging on ω phase in Ti-1023
3. Investigating the nature of competition between ω and α” during quasi-static def.

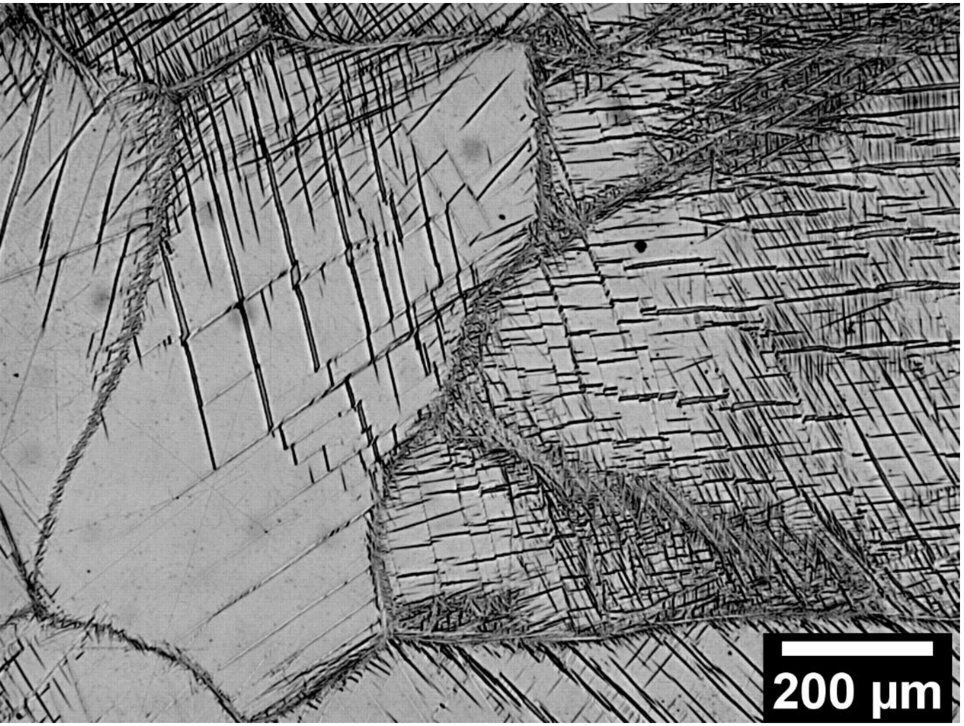
What is happening w/ ω-phase



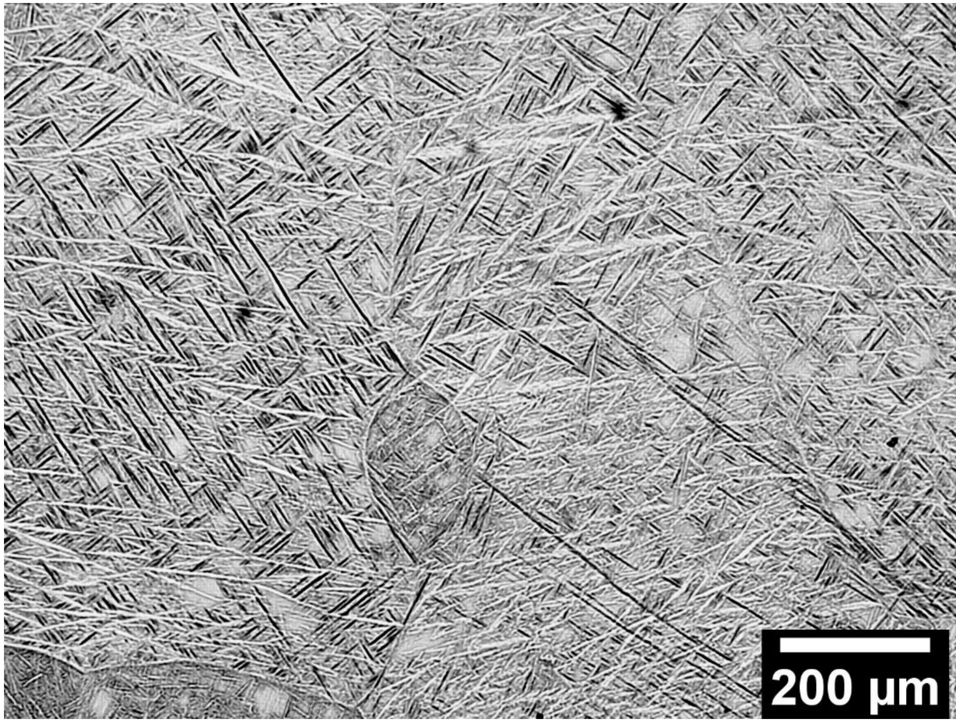


Comparison of Post-Yield Microstructure of Ti-1023

As-Quenched



Aged 900s at 423K



Deformed by a 0.5% plastic strain in tension at 10^{-3}/s

Comparison of Microstructure of Failed Tensile Specimens of Ti-1023

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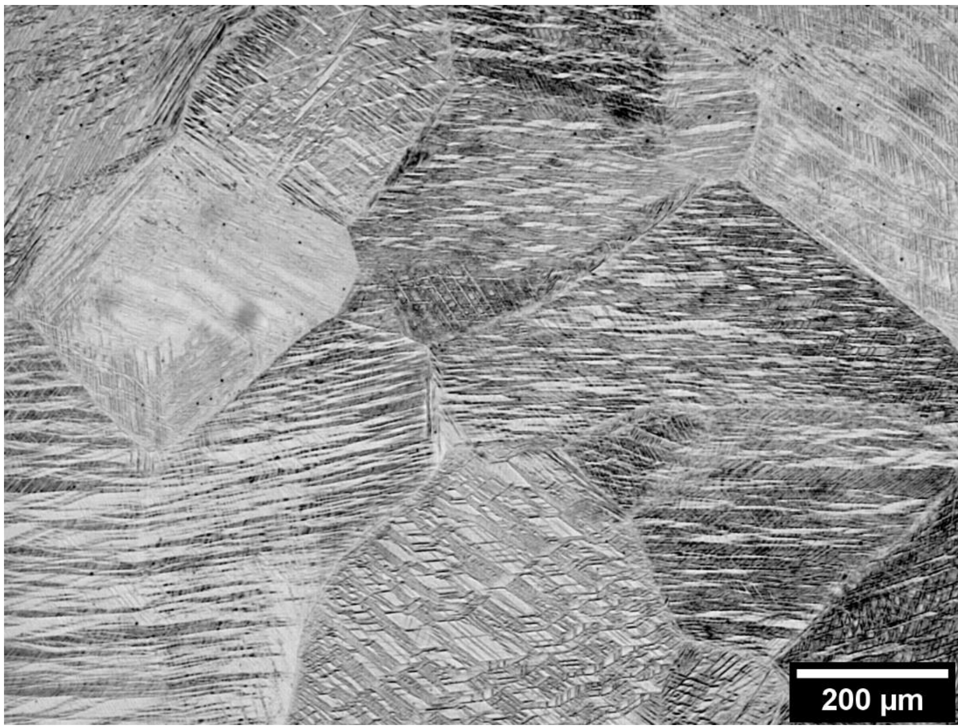
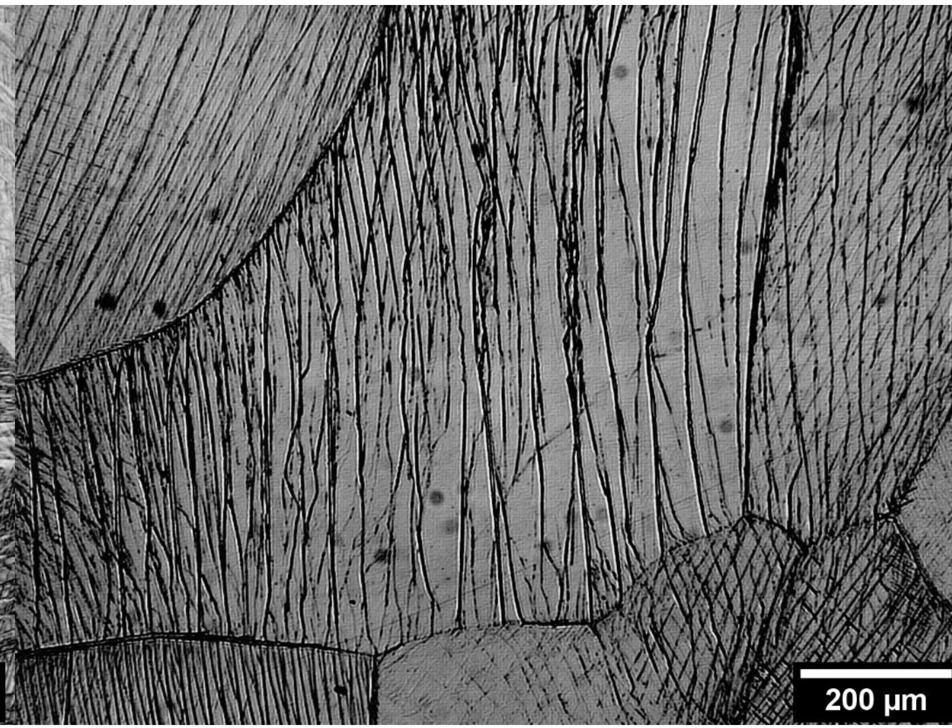
As-Quenched

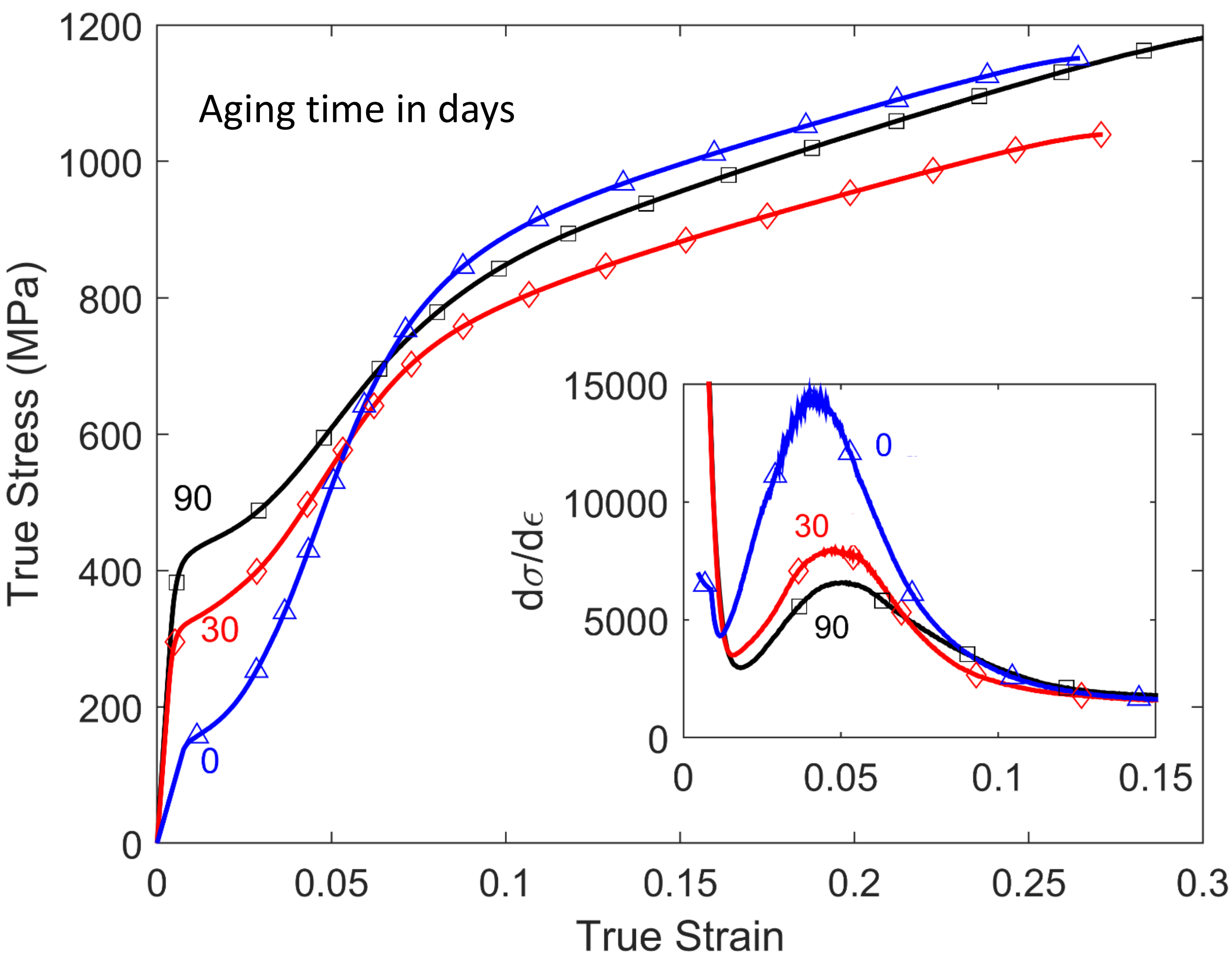


Aged 7200s at 423K

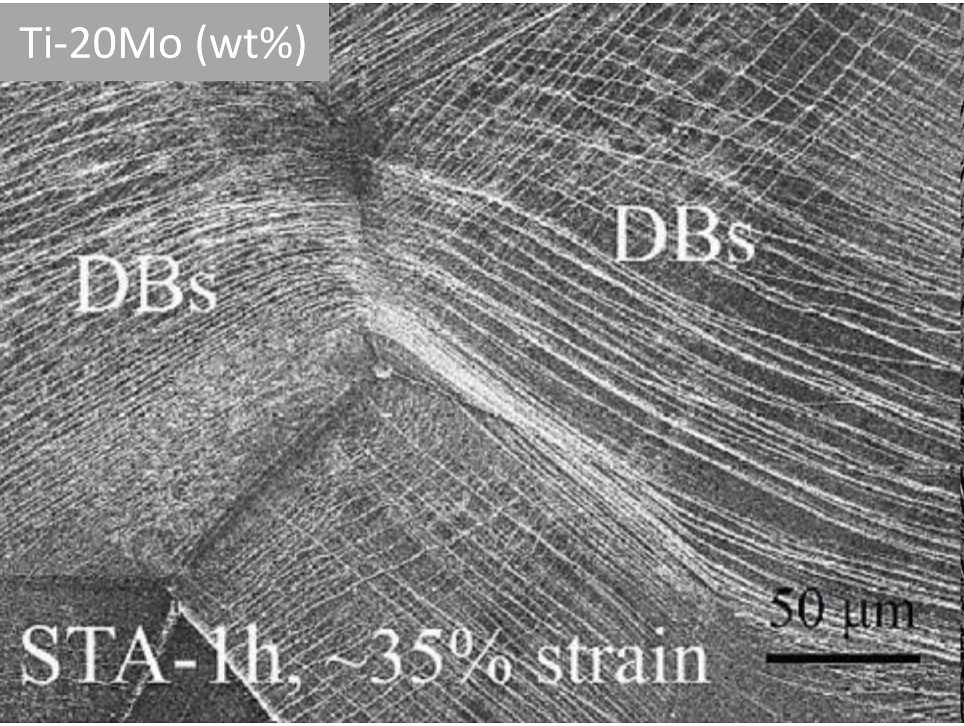


All images are from specimens failed in tension at 10^{-3}/s

Natural Aging in Ti-1023

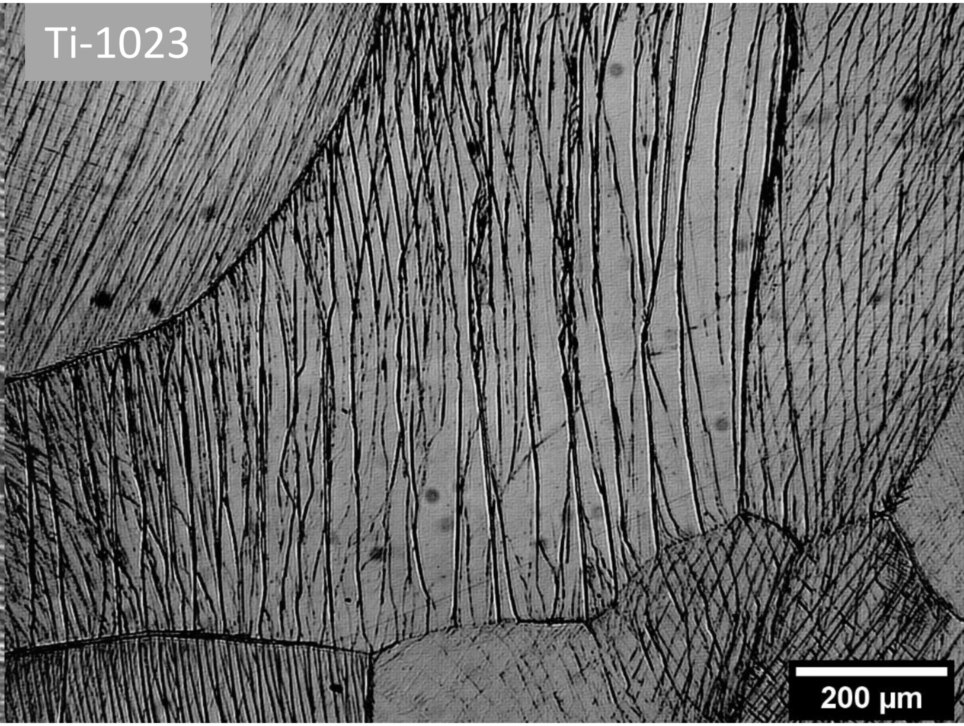


Comparison of Over-Aged (7200s, 423K) Microstructure of Ti-1023



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

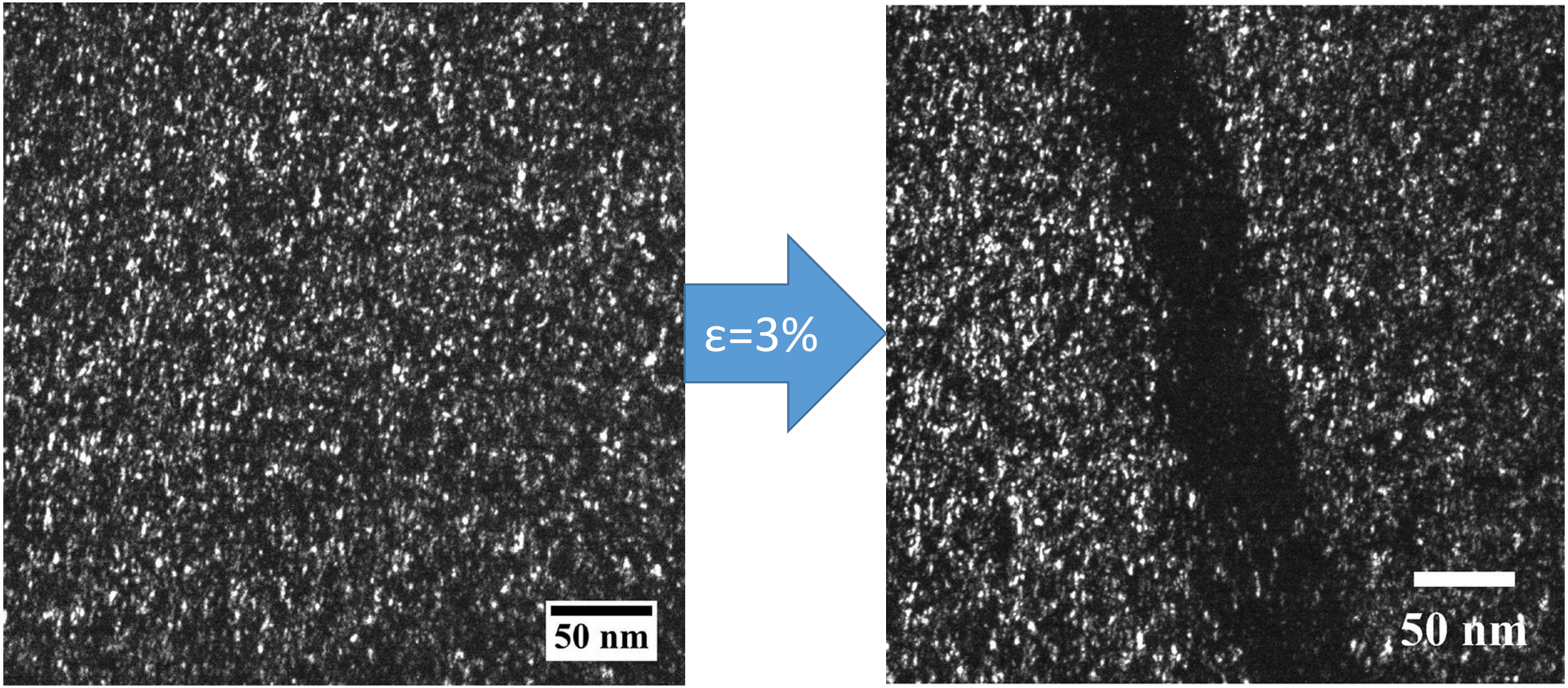
DBs : Dislocation bands



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

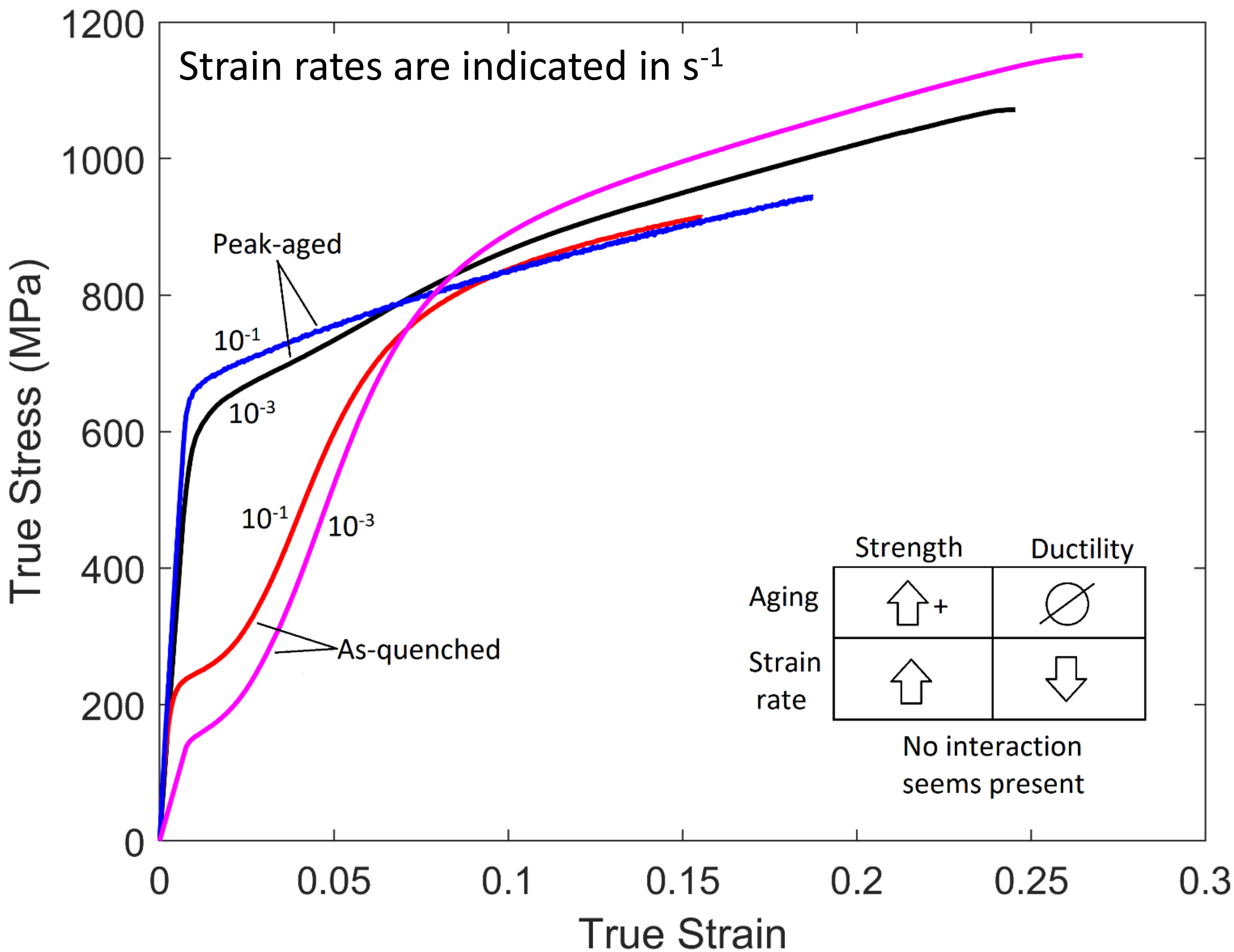
Artificial Aging of Ti-1023 for 7200s at 423K inhibits stress-induced martensite and causes dislocation bands to form

Dislocation Channeling



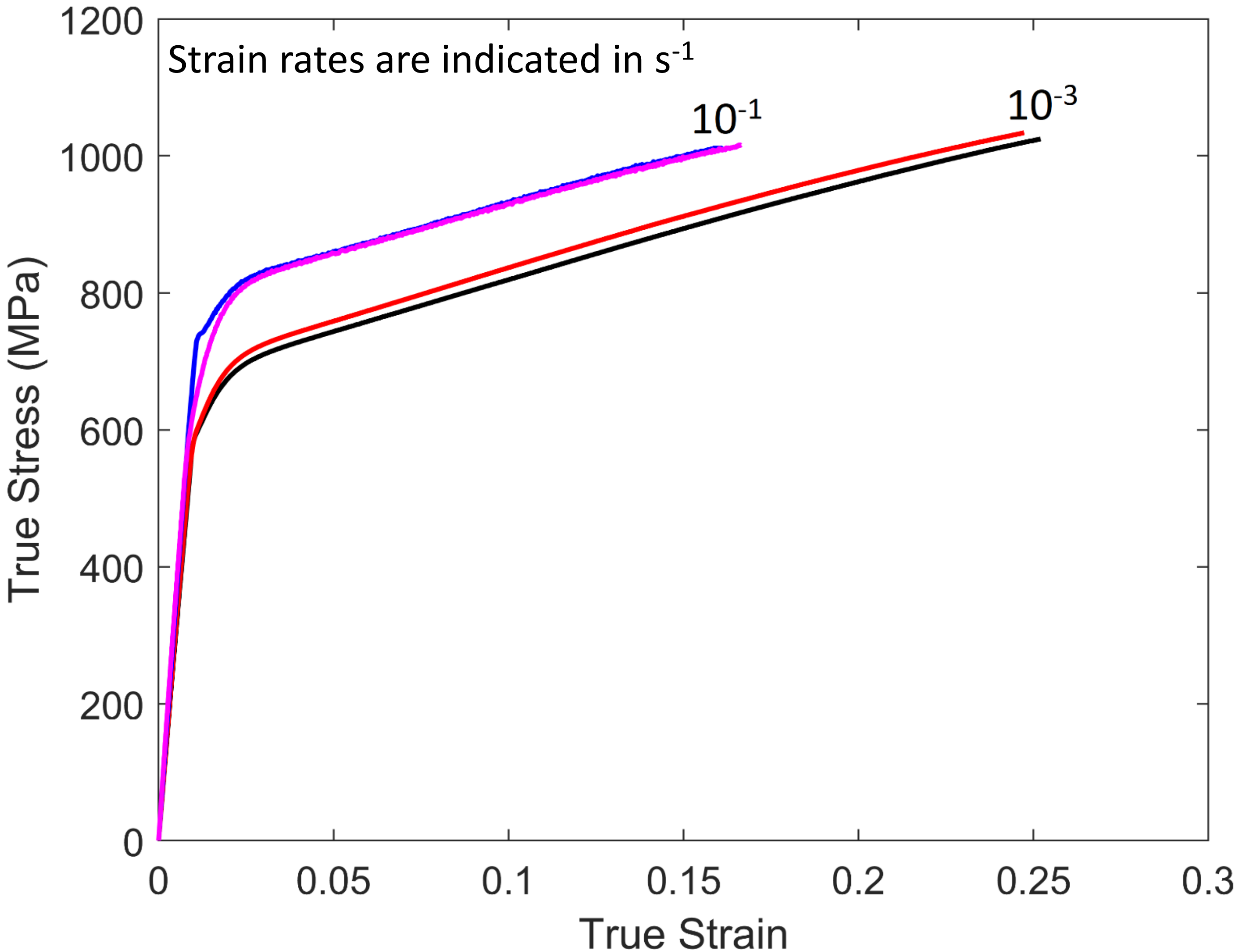
Lai et al. *Acta Materialia* 151 (2018): 67-77.

Strain Rate Effects on TRIP Ti-1023

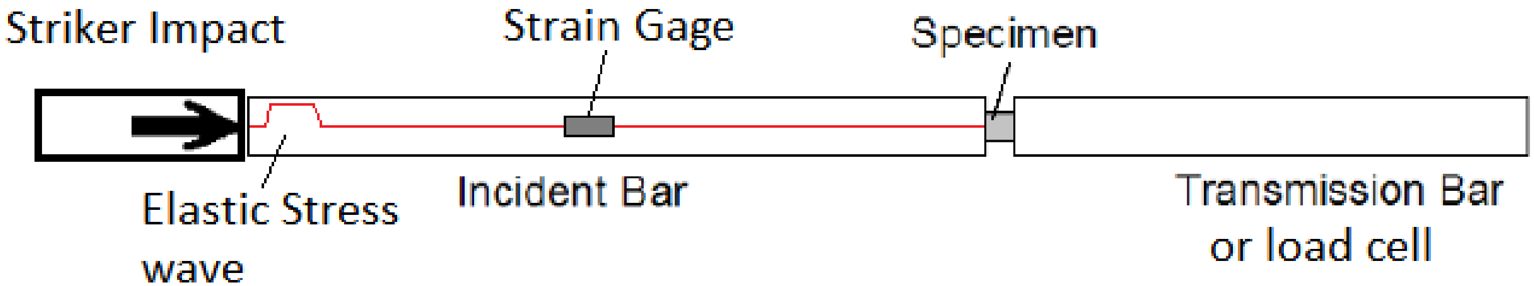


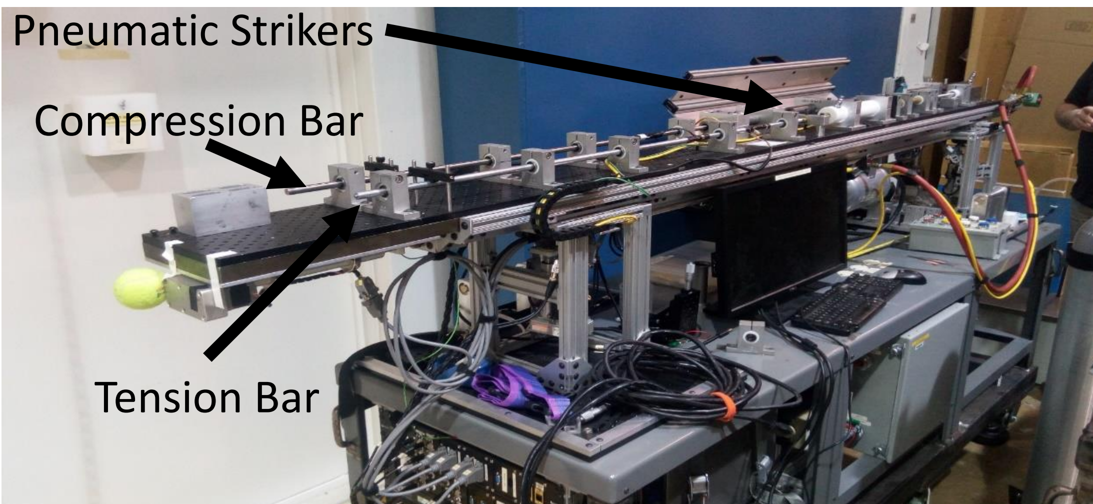
Peak-aged is 900 s at 423K

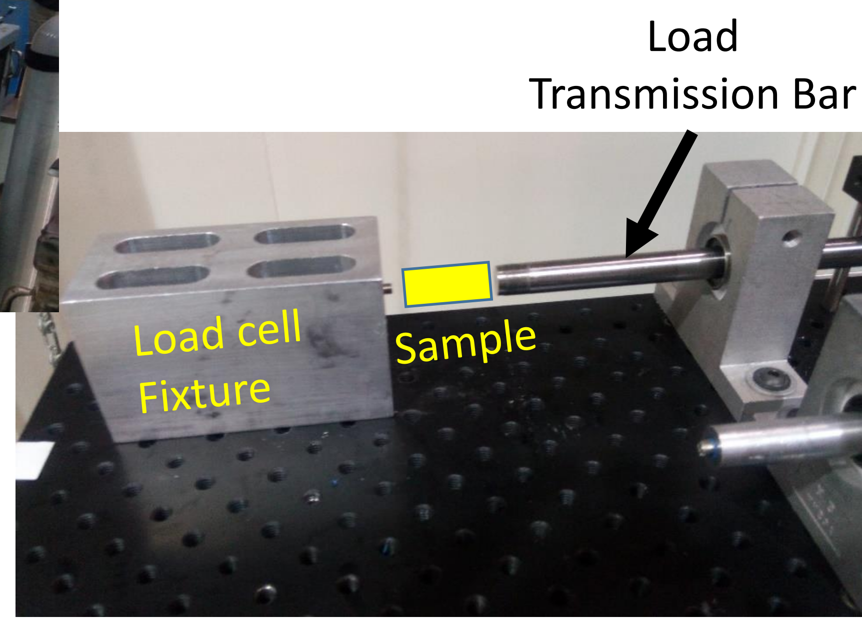
Strain Rate Effect in TWIP Ti-15Mo



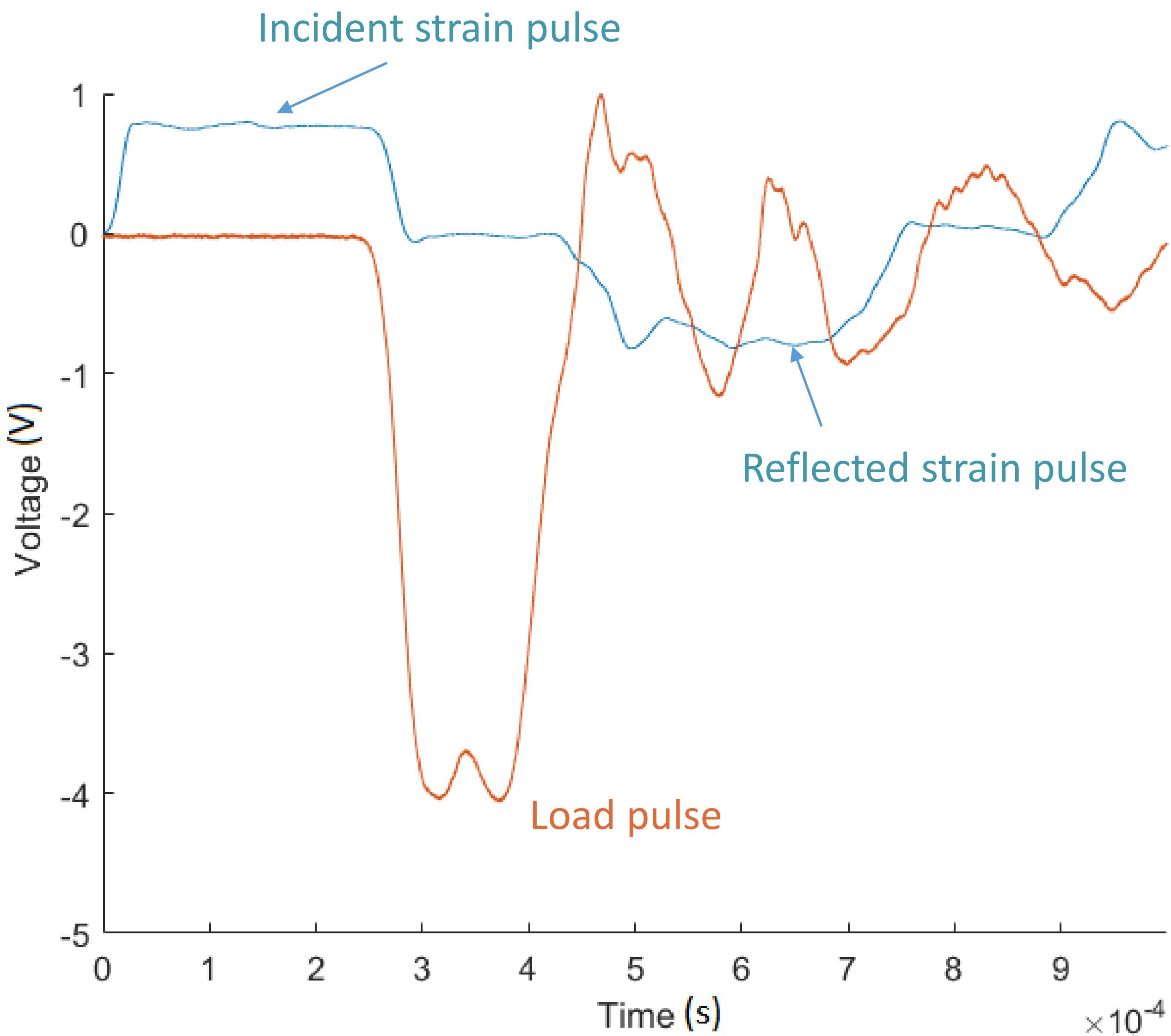
APS Kolsky Bar Setup







Kolsky Bar Raw Data



Post-Processing: Mechanical

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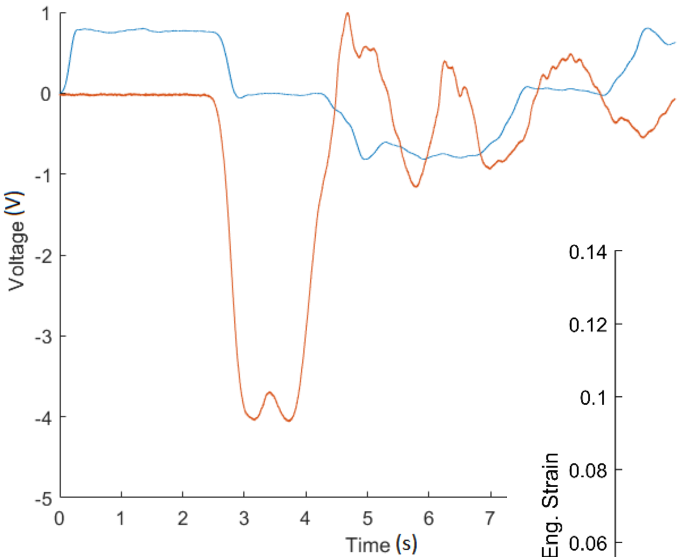
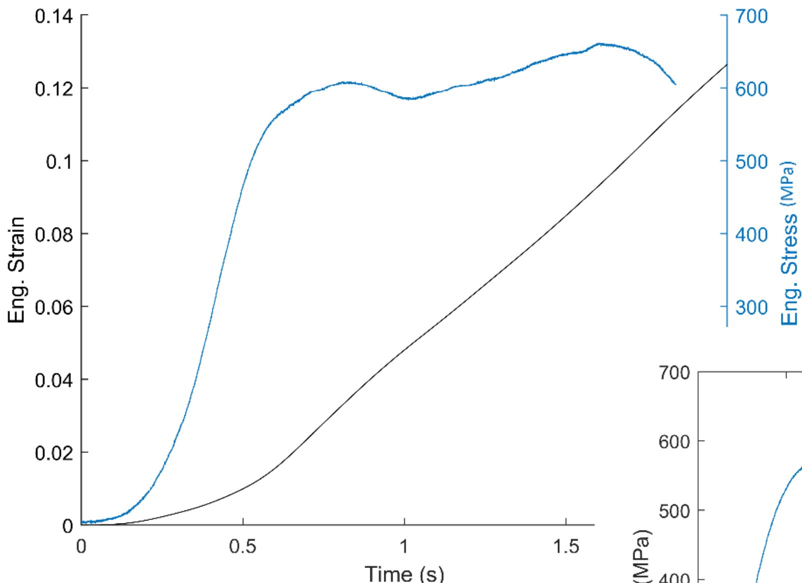
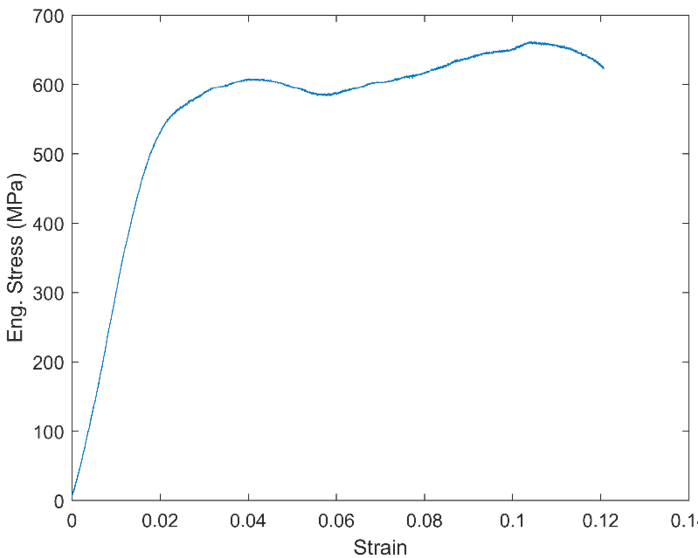


Raw data



Time-resolved strain and stress

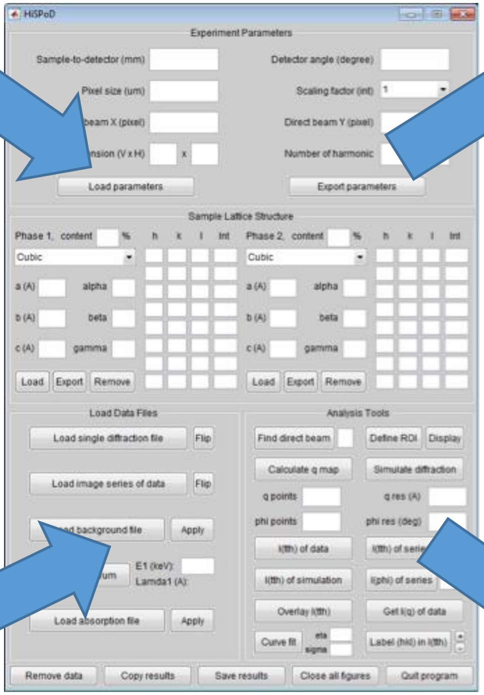
Stress-strain curve

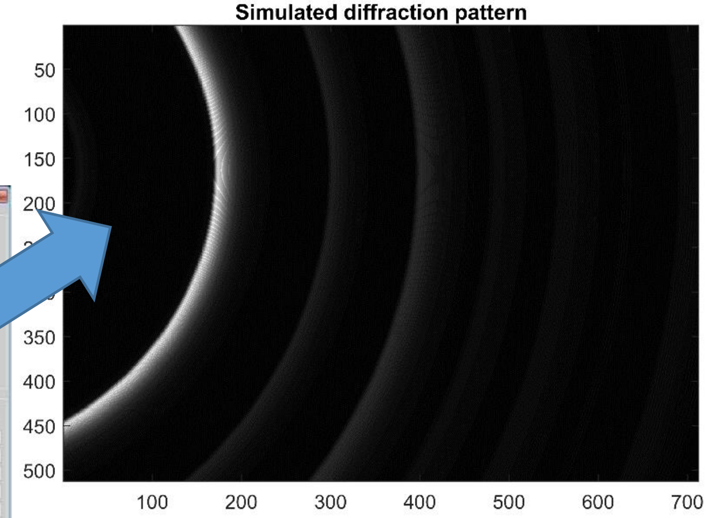


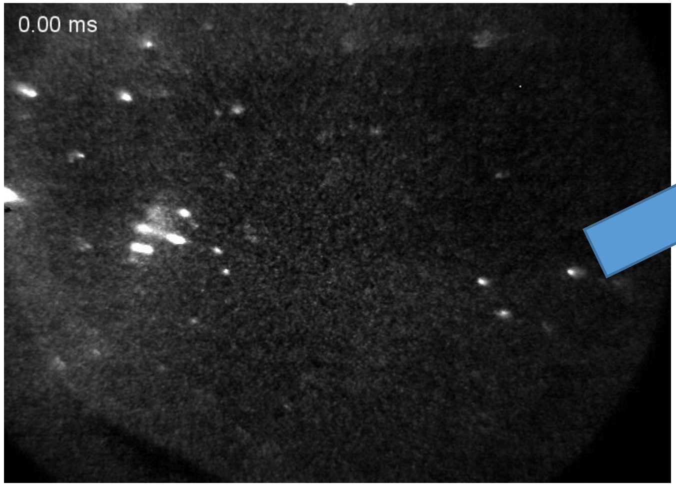
Post-Processing: Diffraction

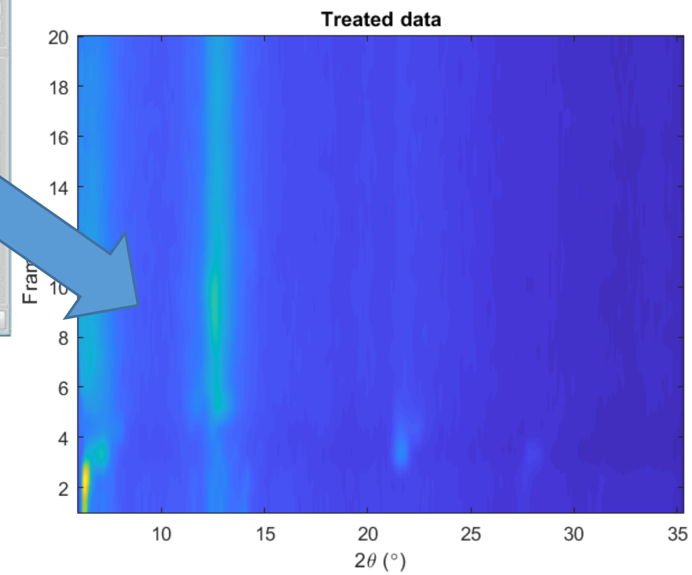
Lattice parameter
Space group
Structure factor
Volume fraction
Beam position
Energy spectrum

HiSPoD



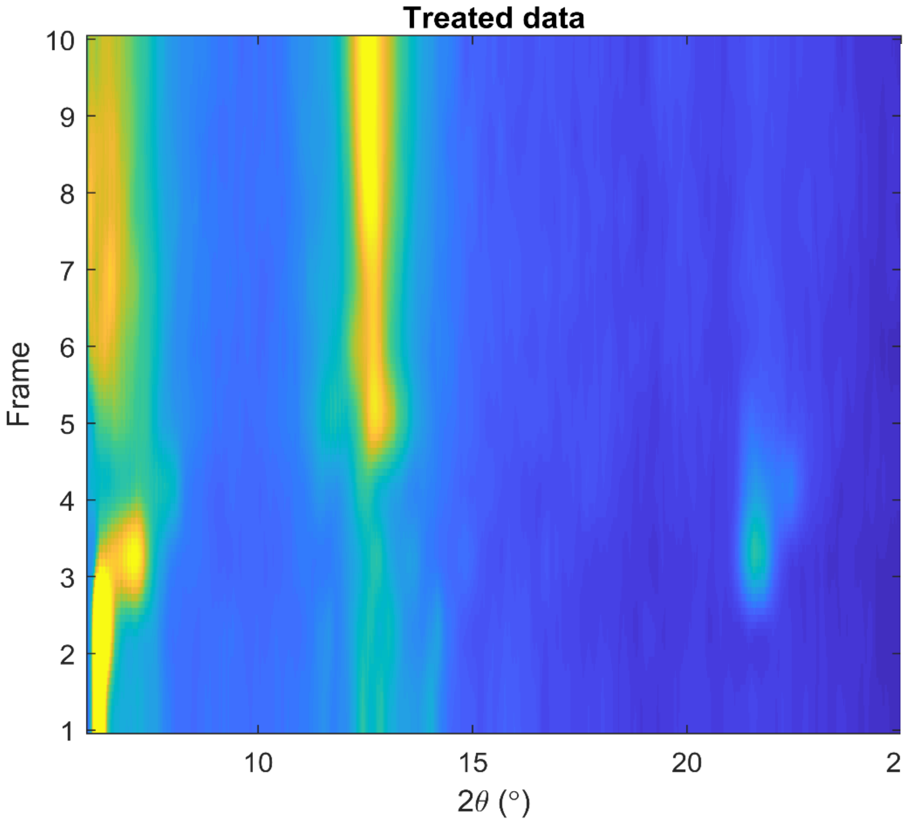


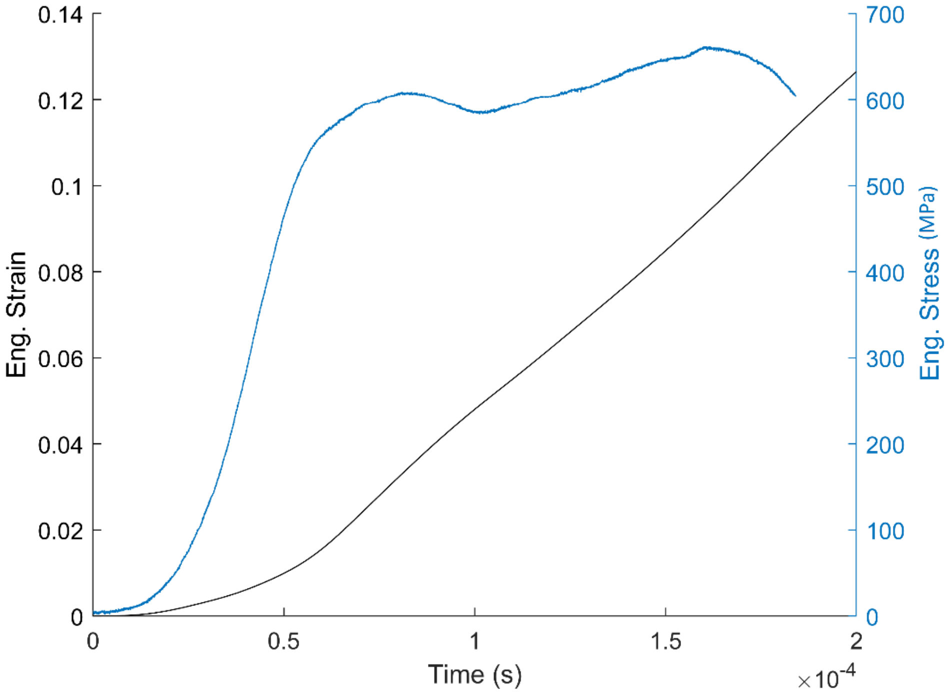




Example from a Ti-1023 Sample

Exact synching remains to be done

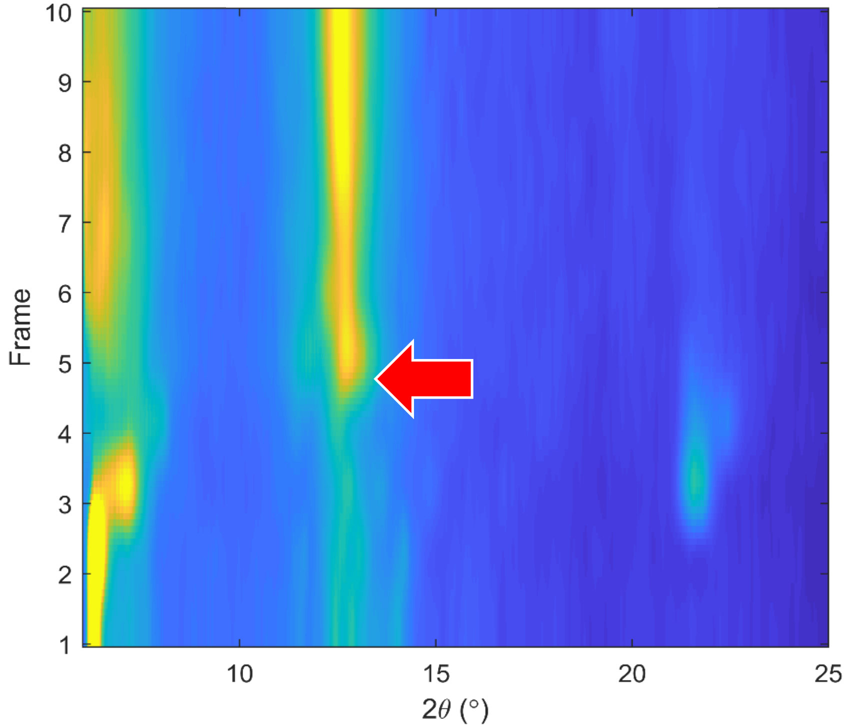




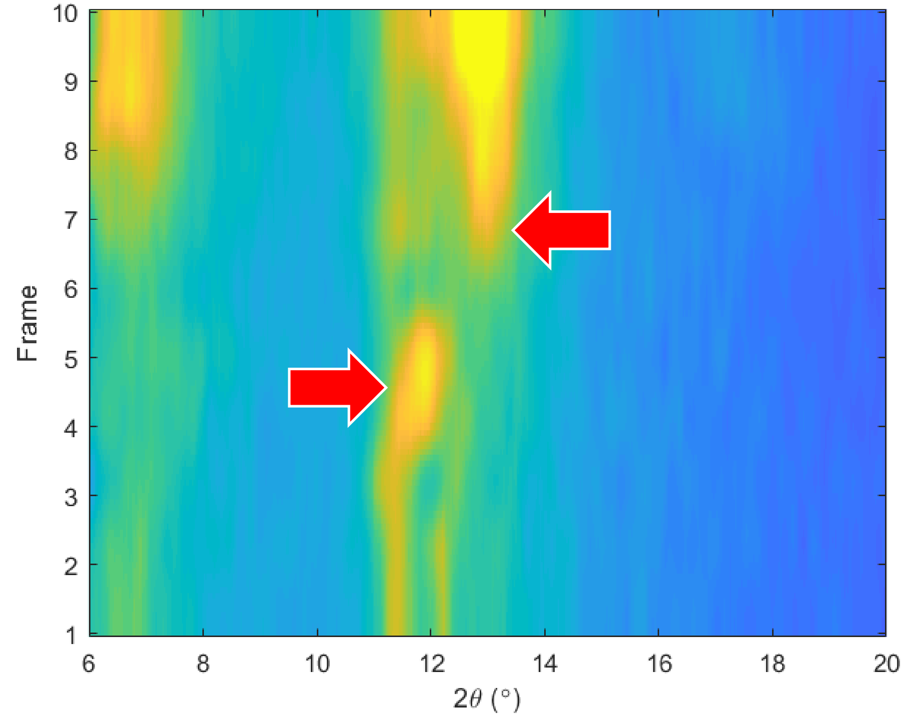
Each frame corresponds to 20 μs interval

Interpreting Diffraction Data

Ti-1023 sample F45

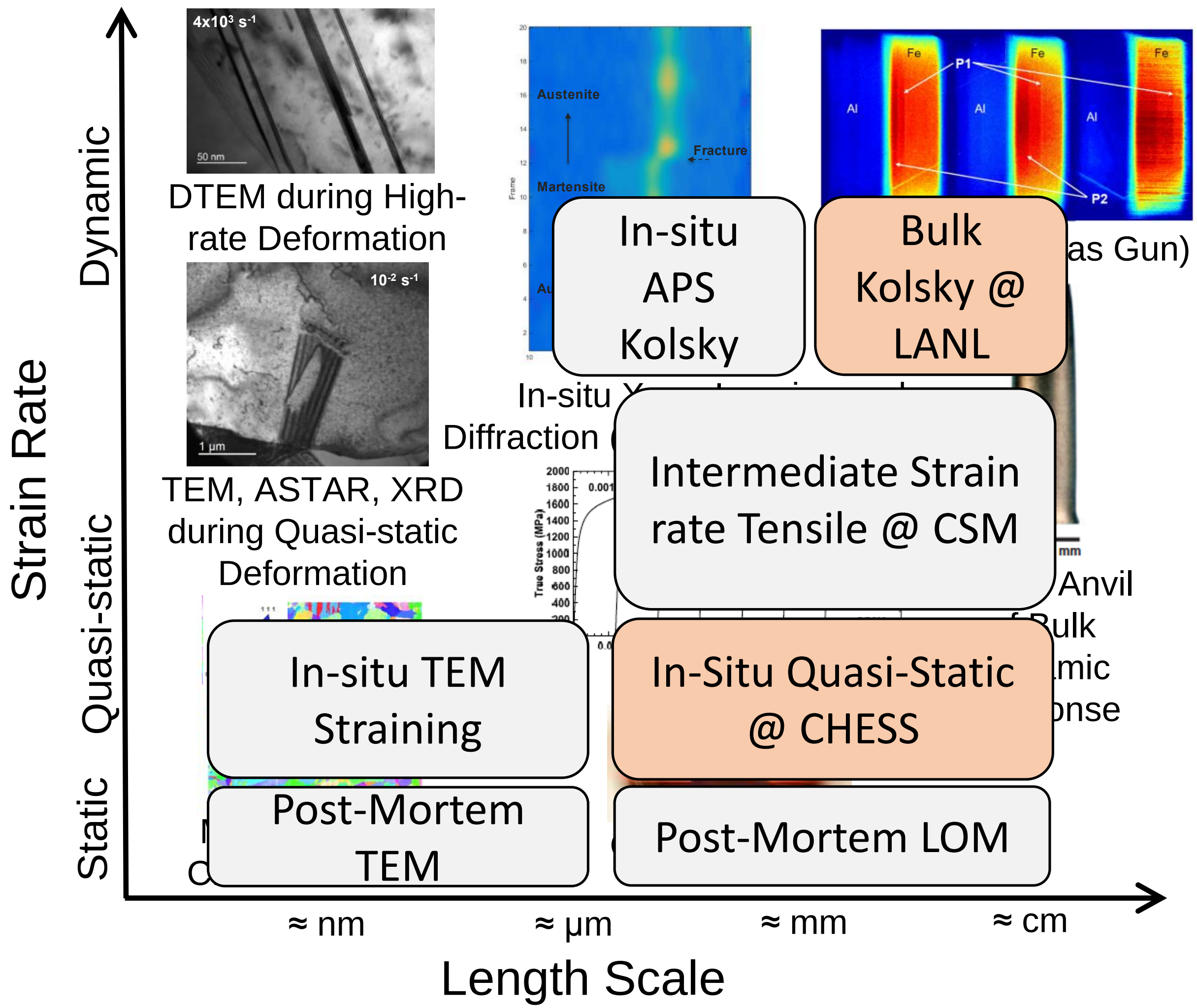


Ti-1023 sample F10



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

Strain Rate Vs. Length Scale Overview: Current Progress



ω-phase in Aged Ti-10-2-3

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