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

Fall Meeting

October 13th – 15th 2020

- Student: Benjamin Ellyson (Mines)
- Faculty: 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 methodology and tailor microstructures and properties
- Benefit: Novel titanium alloys for blast and crash resistant applications

Recent Progress

- Electron backscatter diffraction (EBSD) of APS specimens underway
- Investigation of transition to TRIP inhibited behavior for aged Ti-1023
- Dilatometry of Ti-15Mo and Ti-1023 is near completion
- First principles calculations of binary metastable titanium alloys underway

Metrics		
Description	**% Complete**	**Status**
1. Literature review	80%	●
2. Quasi-static mechanical characterization of Ti-1023 and Ti-15Mo	95%	●
3. Dynamic testing of Ti-1023 and Ti-15Mo	80%	●
4. Microstructural characterization of pre- and post-deformed samples	70%	●
5. *In situ* characterization of microstructural evolution during deformation	60%	●

Industrial Relevance: Development of Blast Resistant Materials for the Navy

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- **Cellular Materials Program**
  - Multifunctional structures
  - **Blast resistance**
  - Thermal management

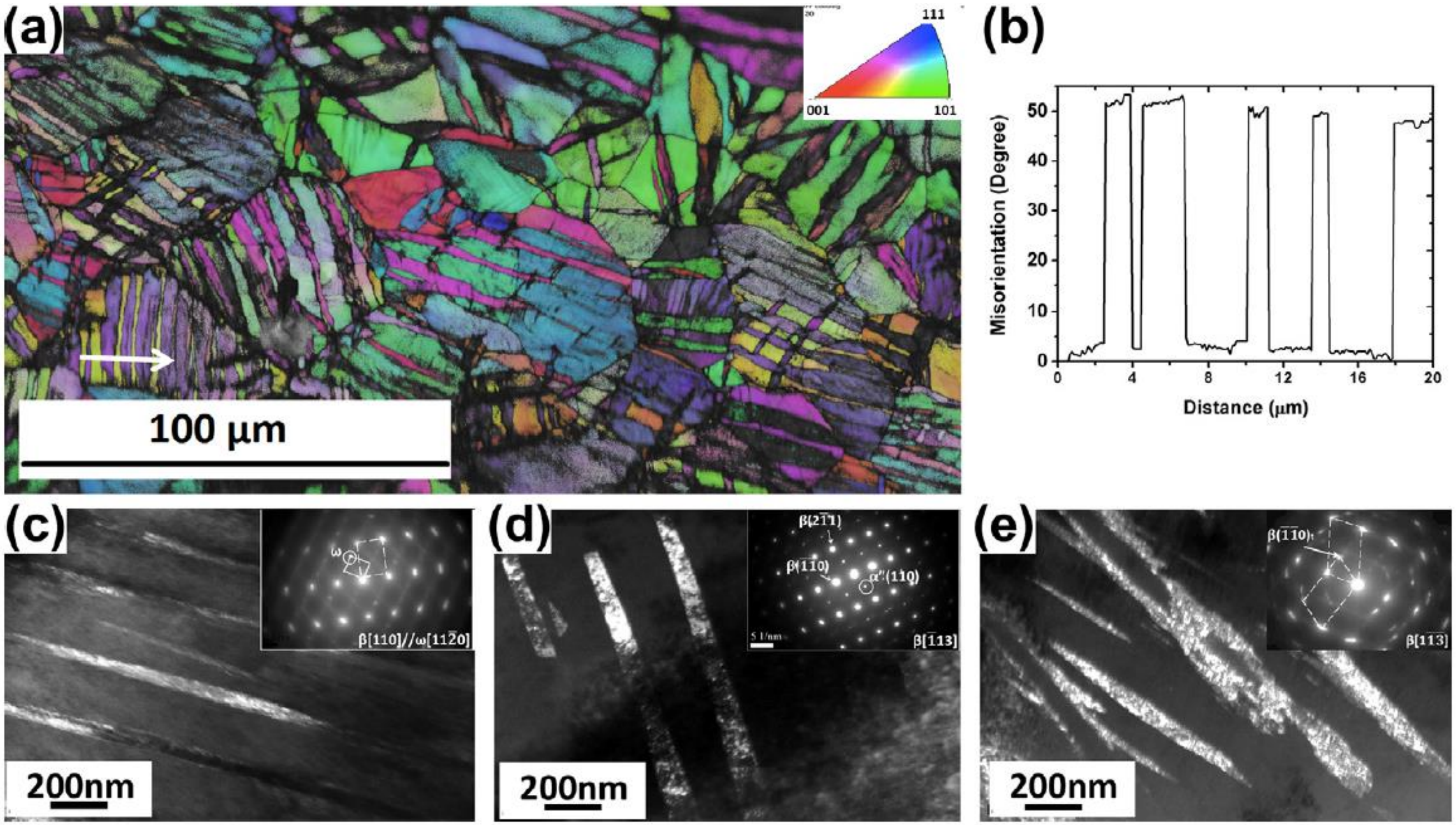
- **Propulsion Materials Program**
  - **Aircraft and marine engines**

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

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

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

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



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

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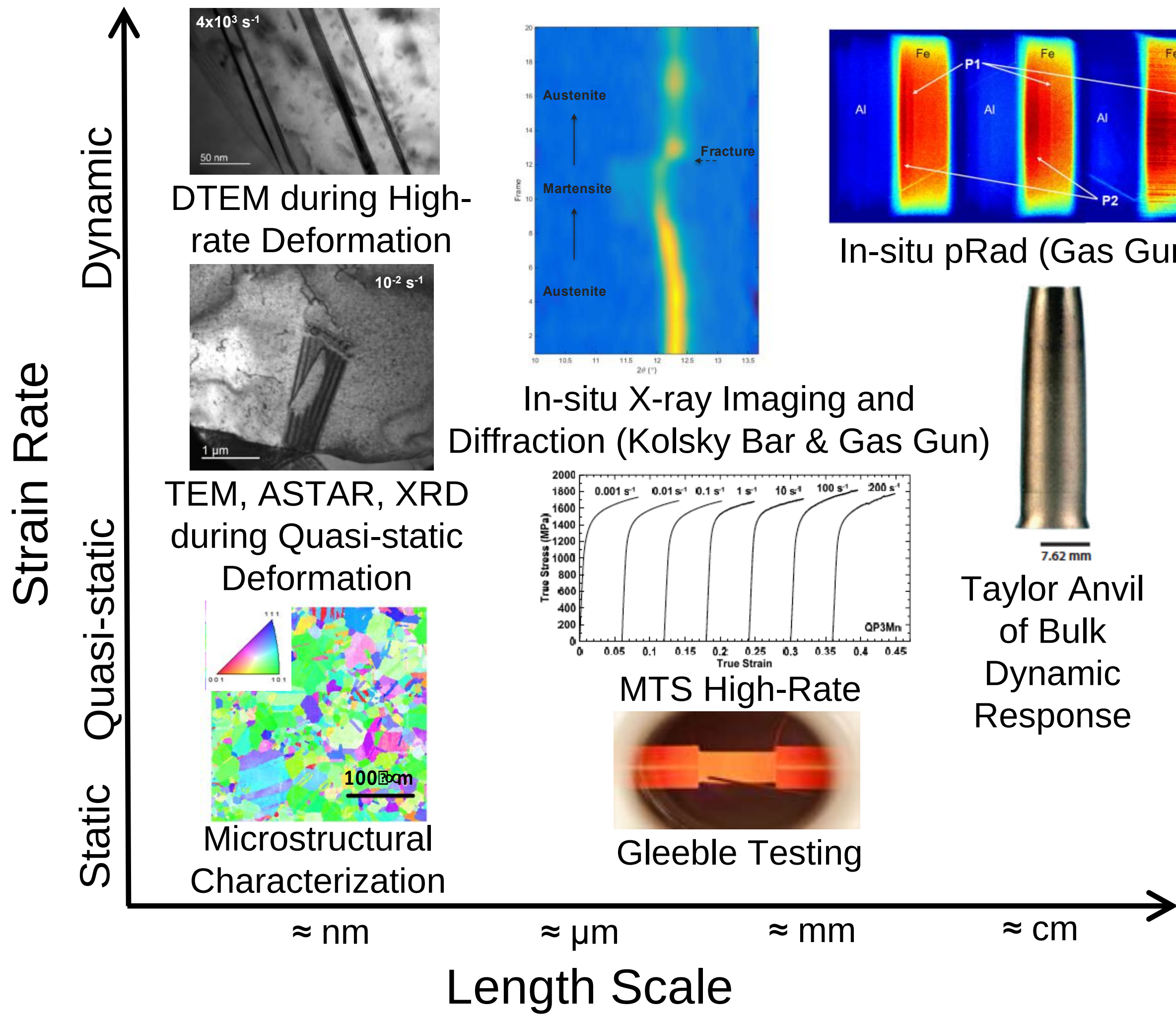
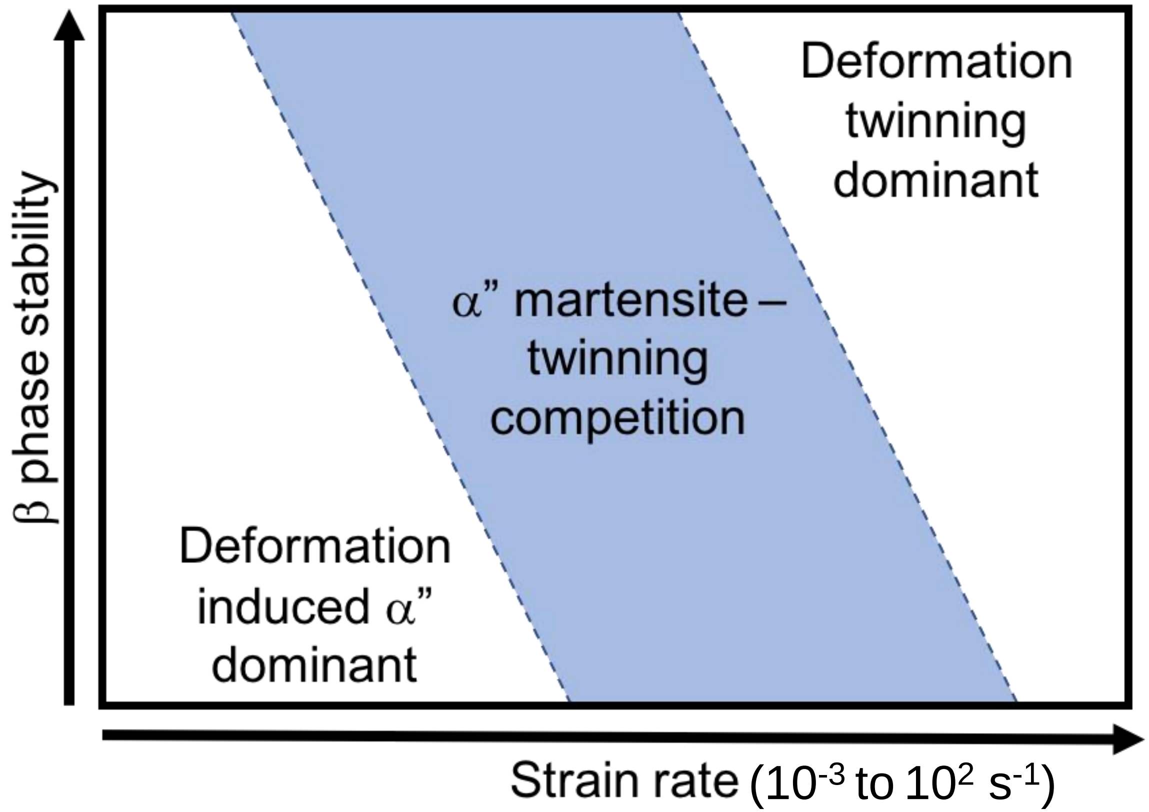


TRIP: Transformation Induced Plasticity
TWIP: TWinning Induced Plasticity

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



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

As-Quenched Quasi-static Tension of TRIP Ti-1023

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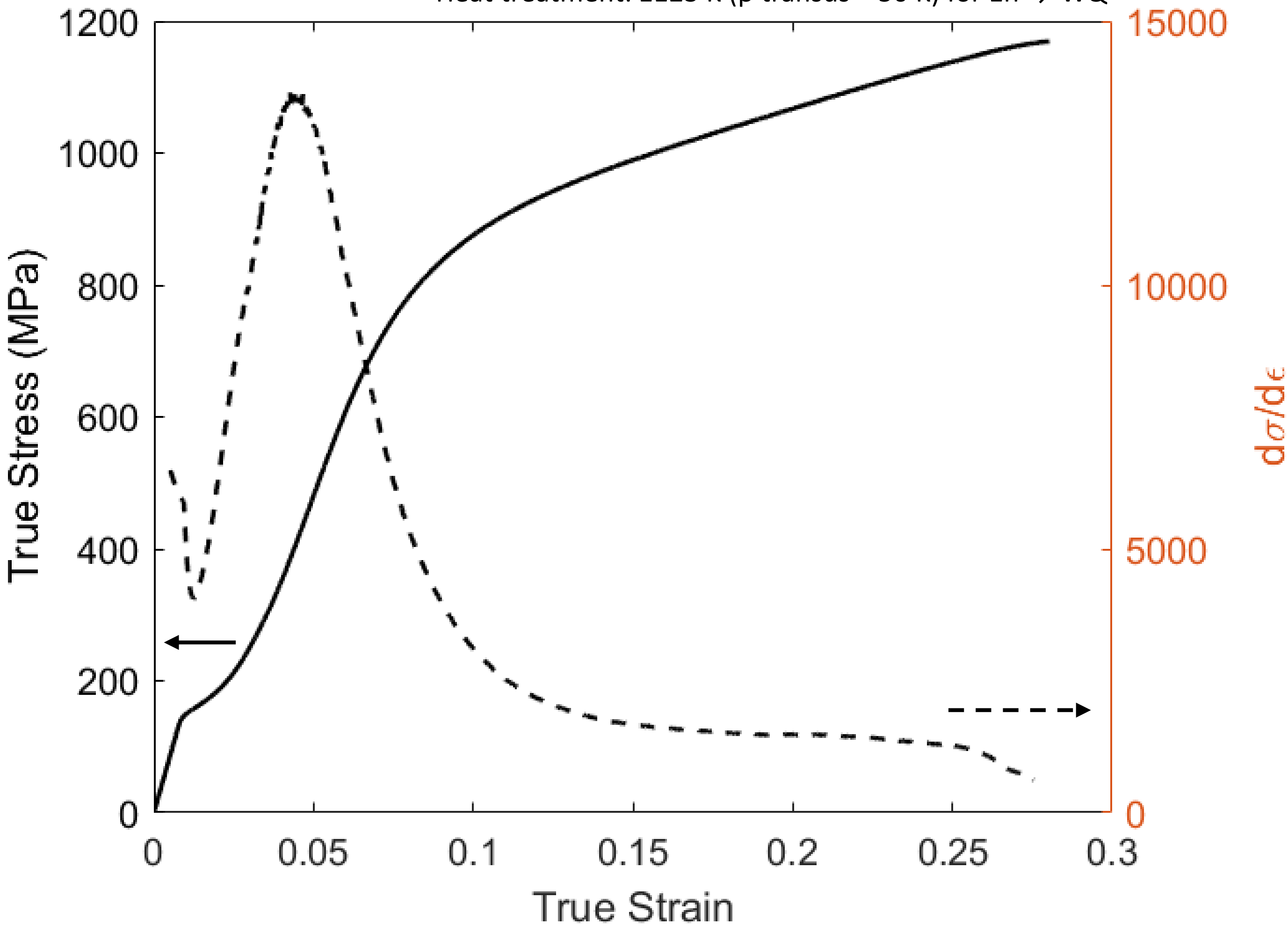
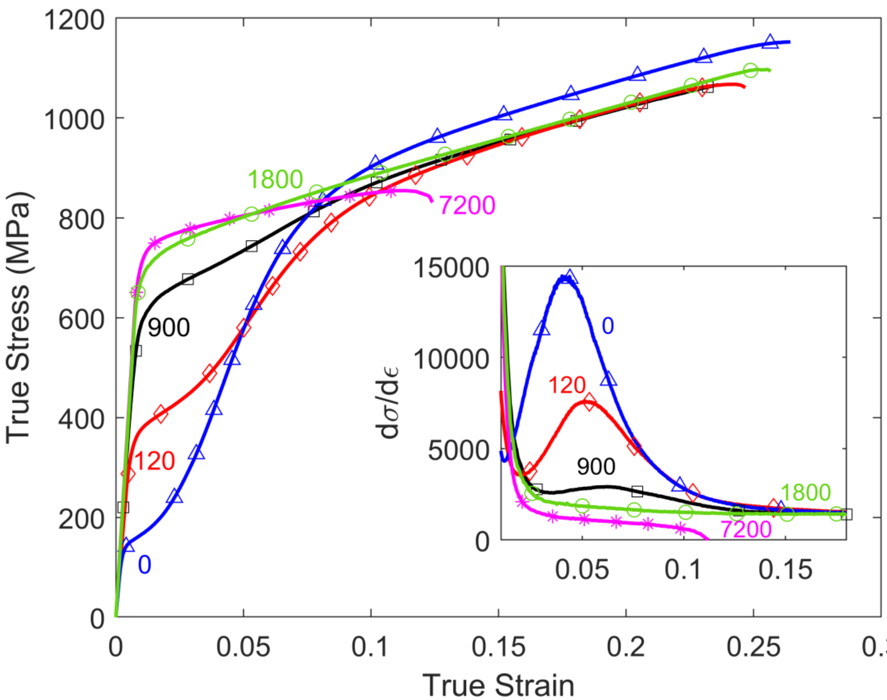
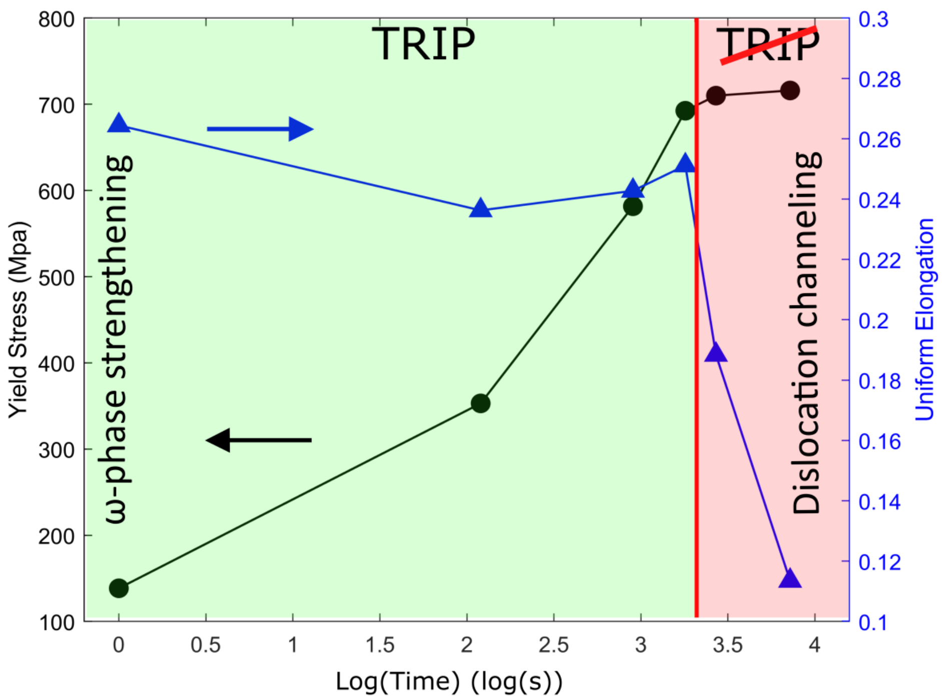
Heat treatment: 1123 K (β transus + 50 K) for 1h → WQ



Low-Temperature Aging of TRIP Ti-1023

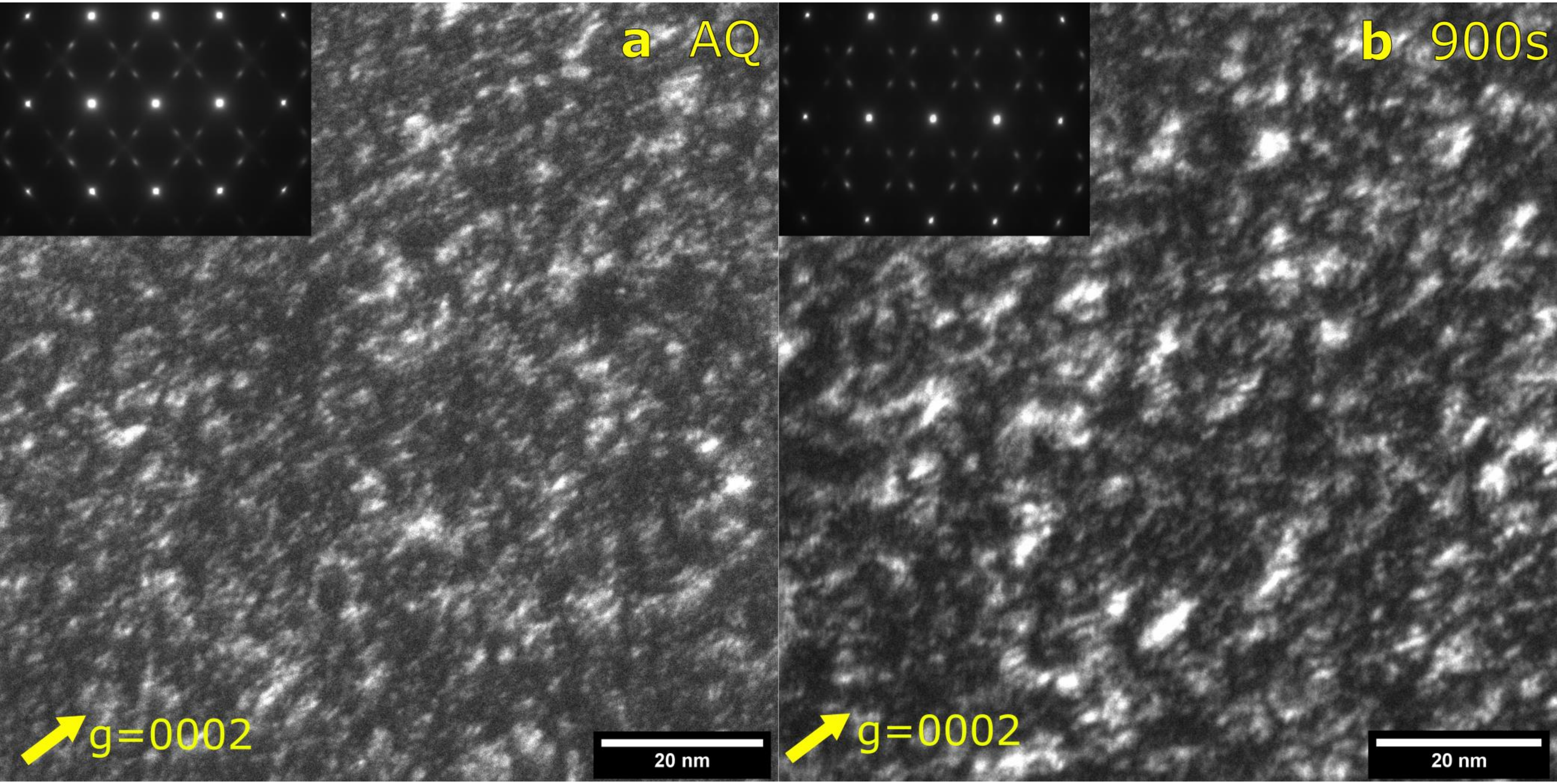
Tensile deformation at $10^{-3}s^{-1}$





Aging for indicated number of seconds at 423 K

ω-phase in 423 K Aged Ti-1023



Transition To TRIP Inhibited

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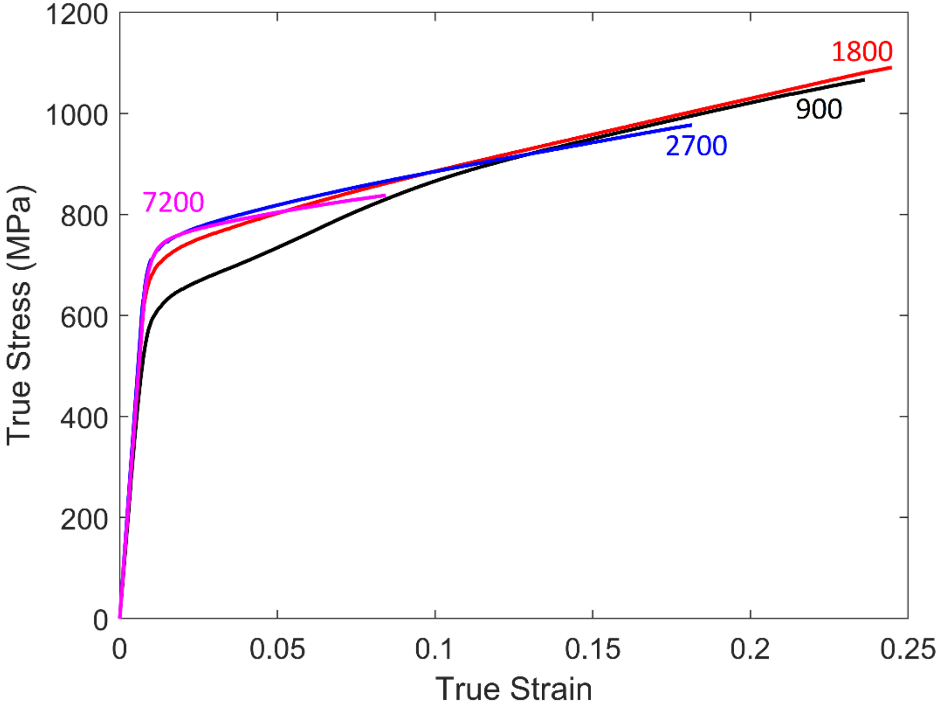
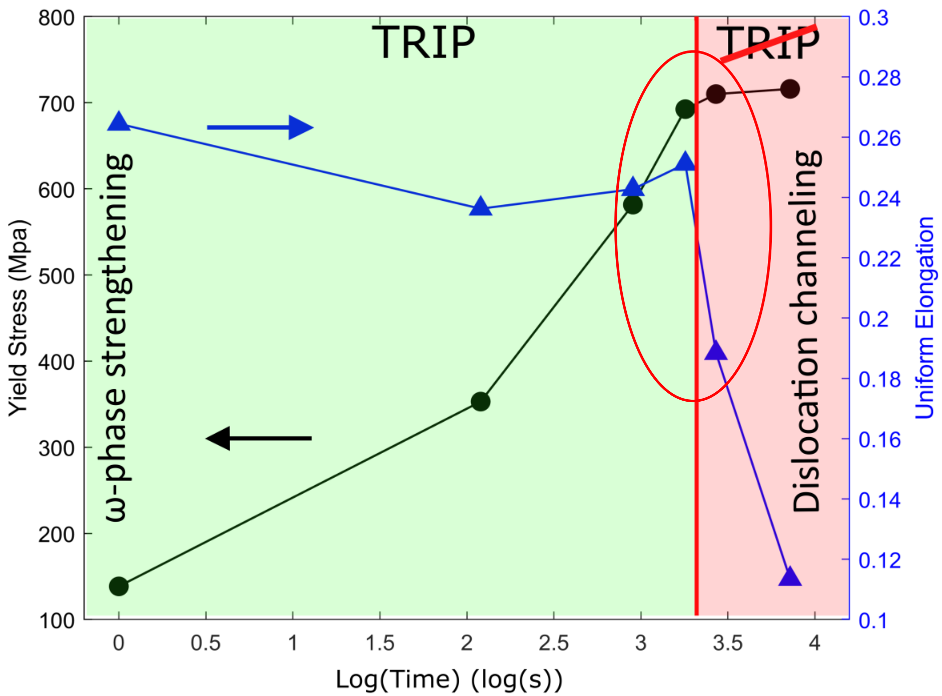
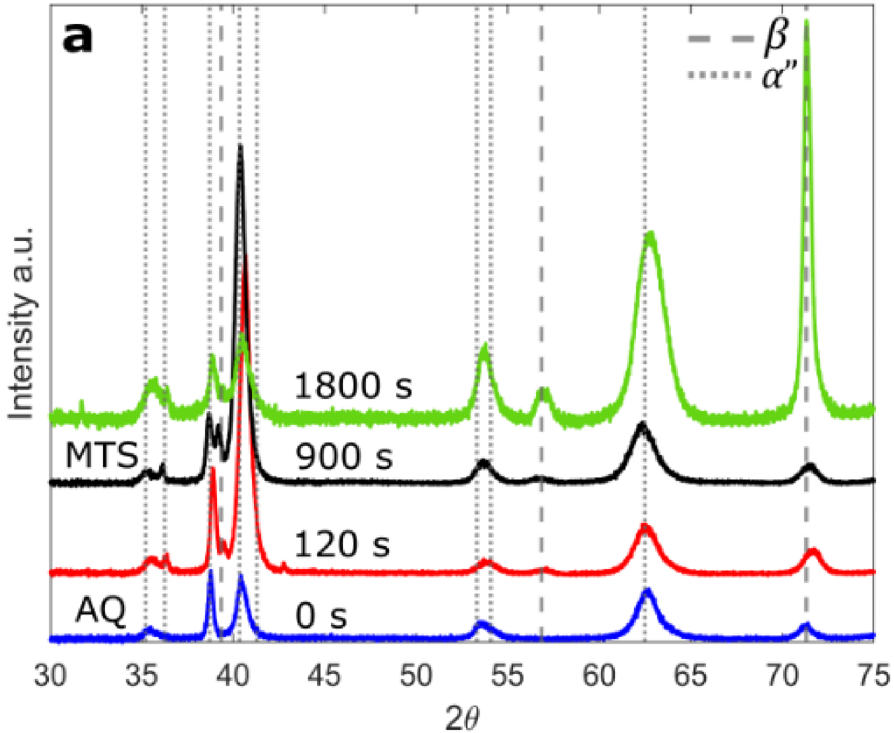
Tensile deformation at $10^{-3}s^{-1}$

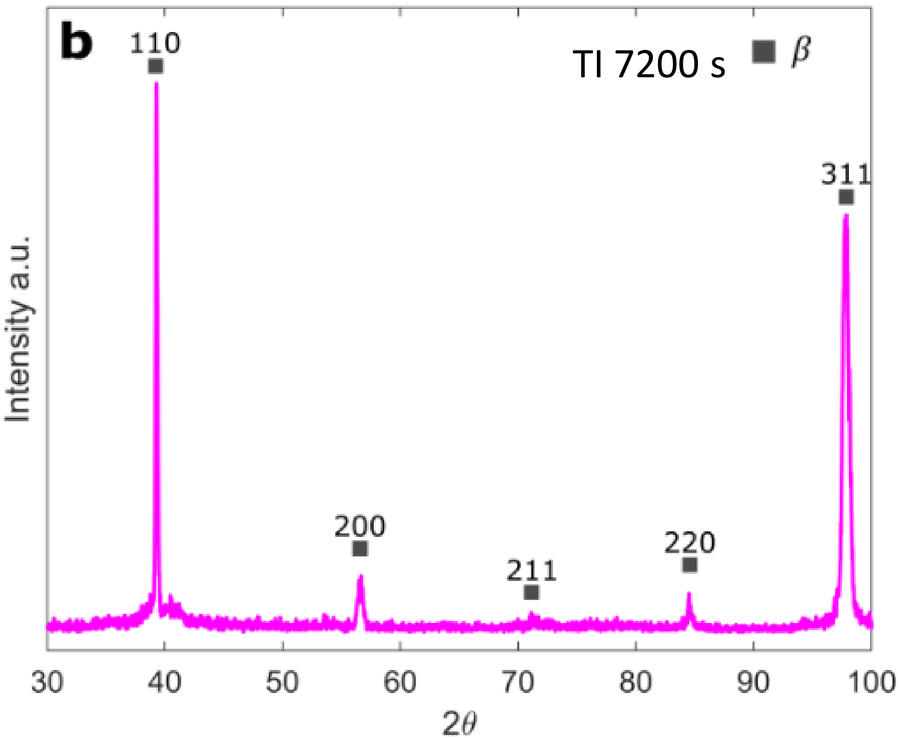




Aging for indicated number of seconds at 423 K

EBSD & XRD of Interrupted Tensile Tests of Aged Ti-1023





Xray diffractograms from fractured gage section

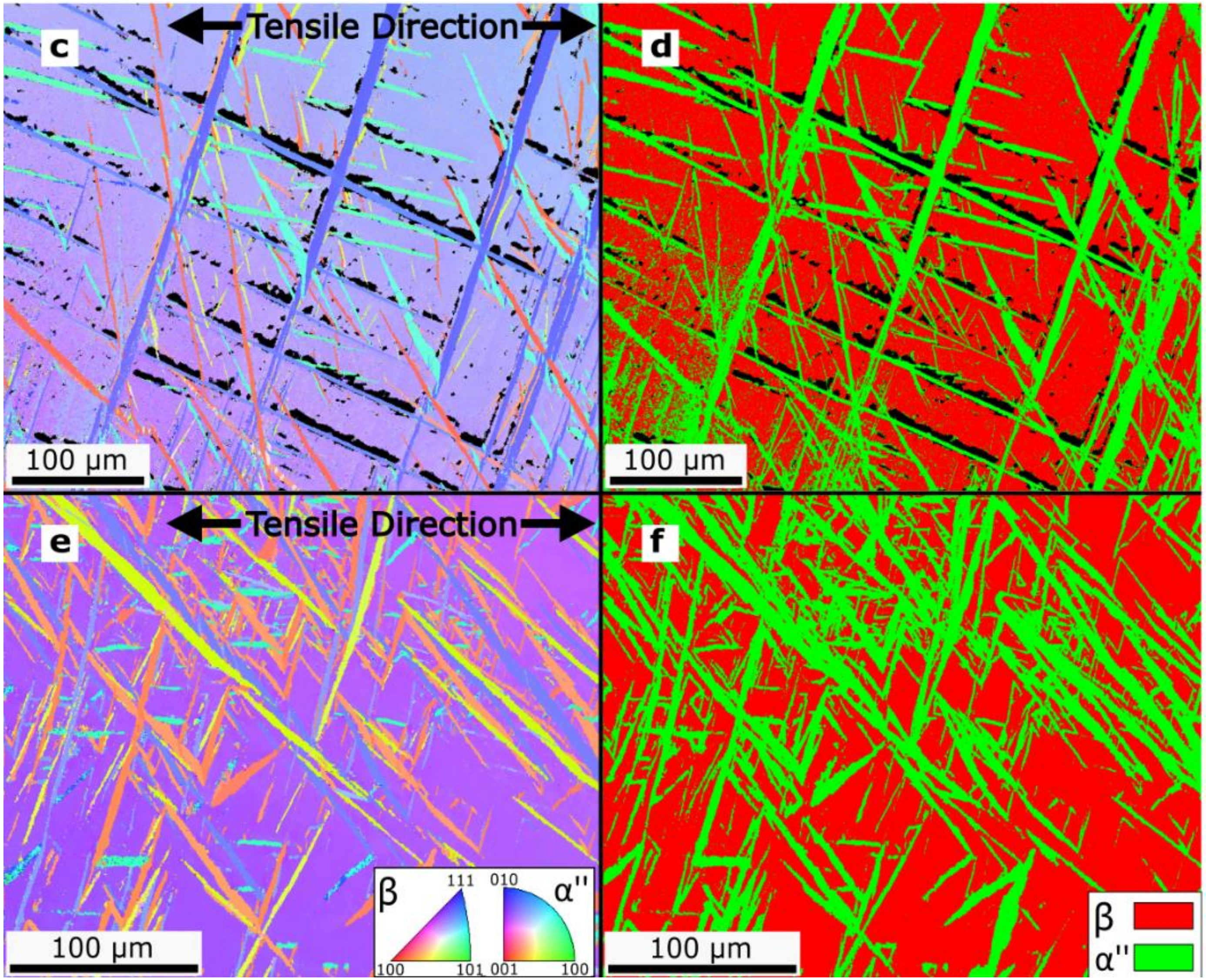
EBSD of Interrupted Tensile Tests of Aged Ti-1023

As-quenched

0.5% Plastic strain

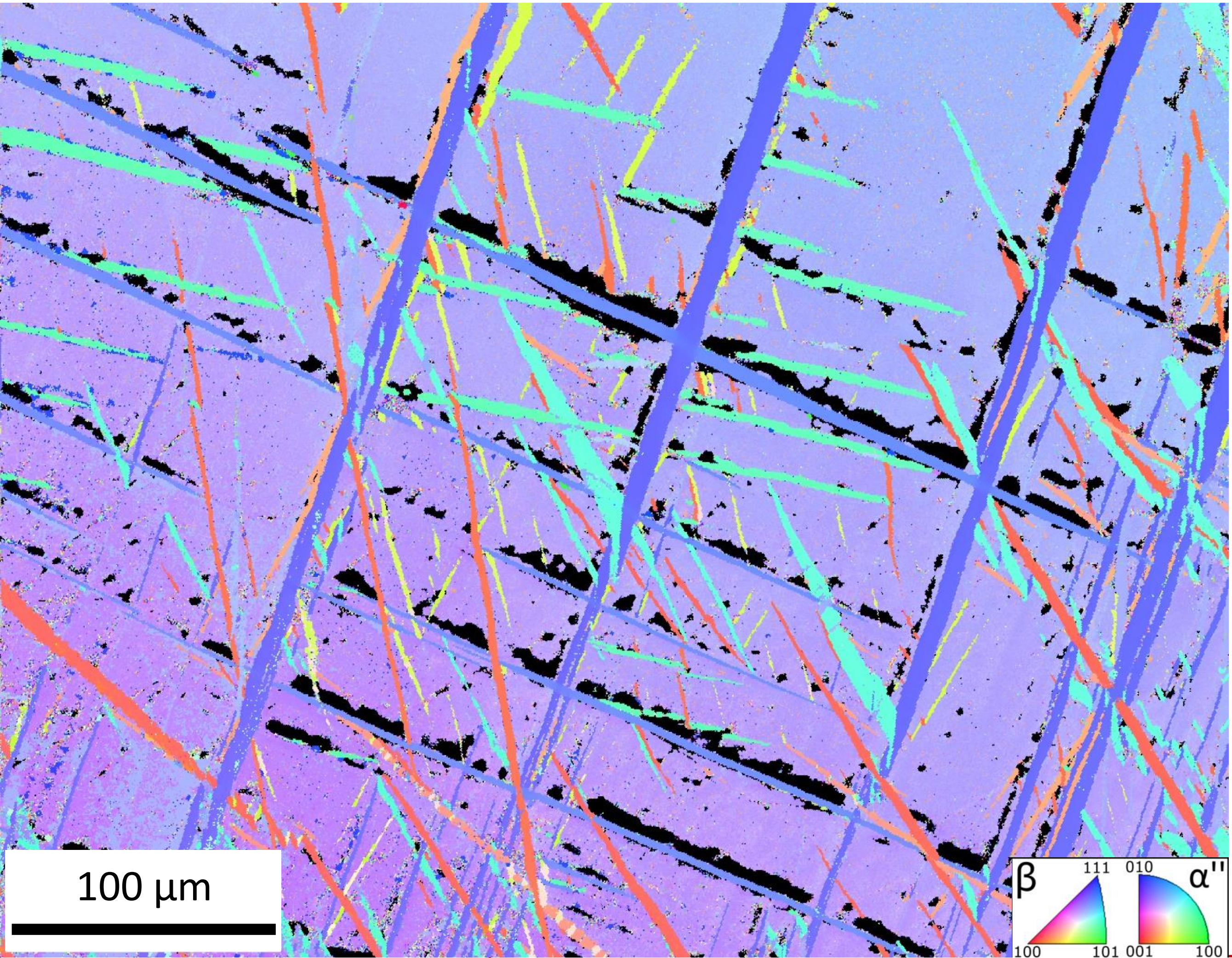
900 s at 423 K

0.5% Plastic strain



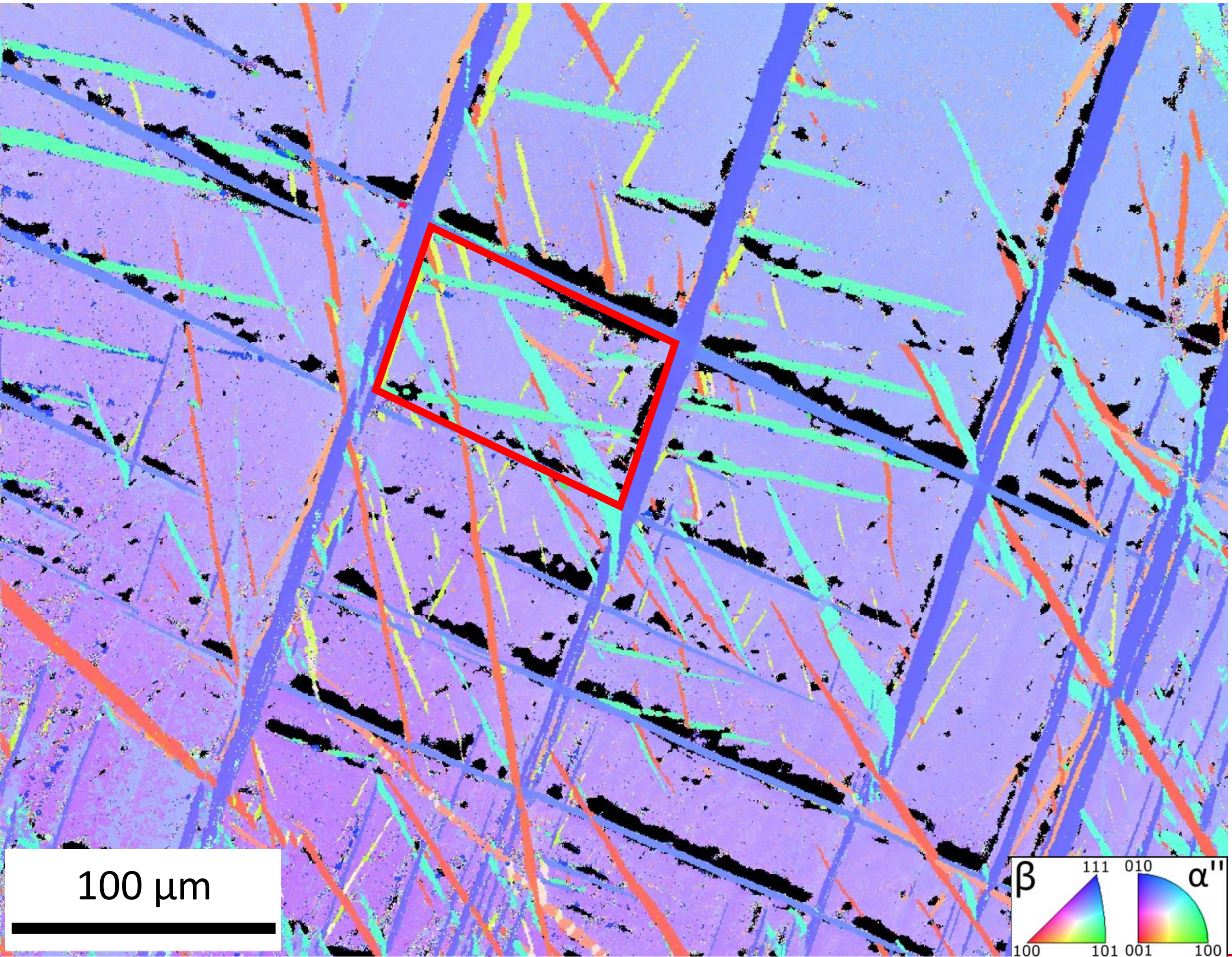
Hierarchy of Microstructural Evolution

As-quenched, deformed 0.5% plastic strain at $10^{-3}s^{-1}$



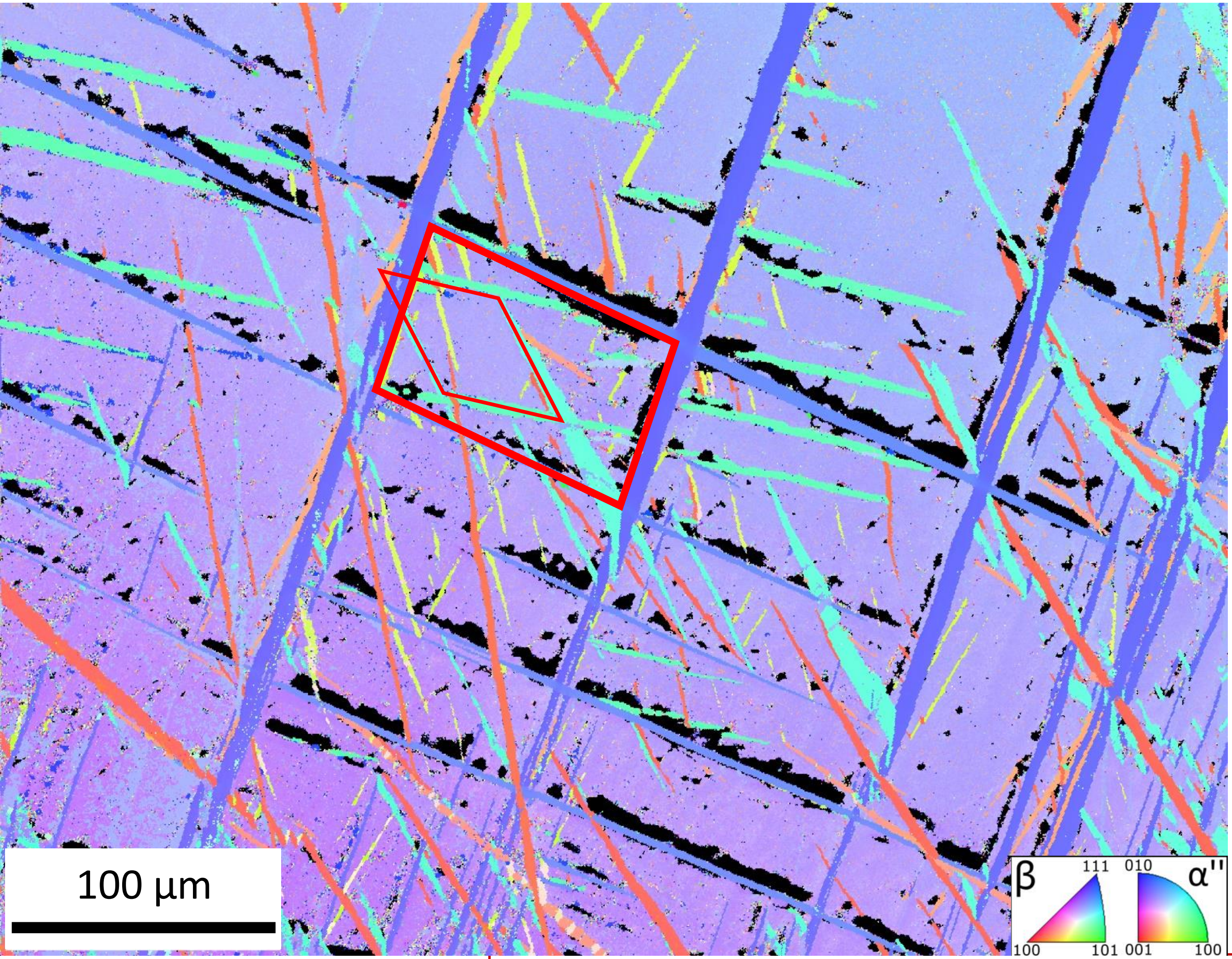
Dynamic Hall-Petch effect: Average slip length decreases with plastic strain

Hierarchy of Microstructural Evolution



Primary product

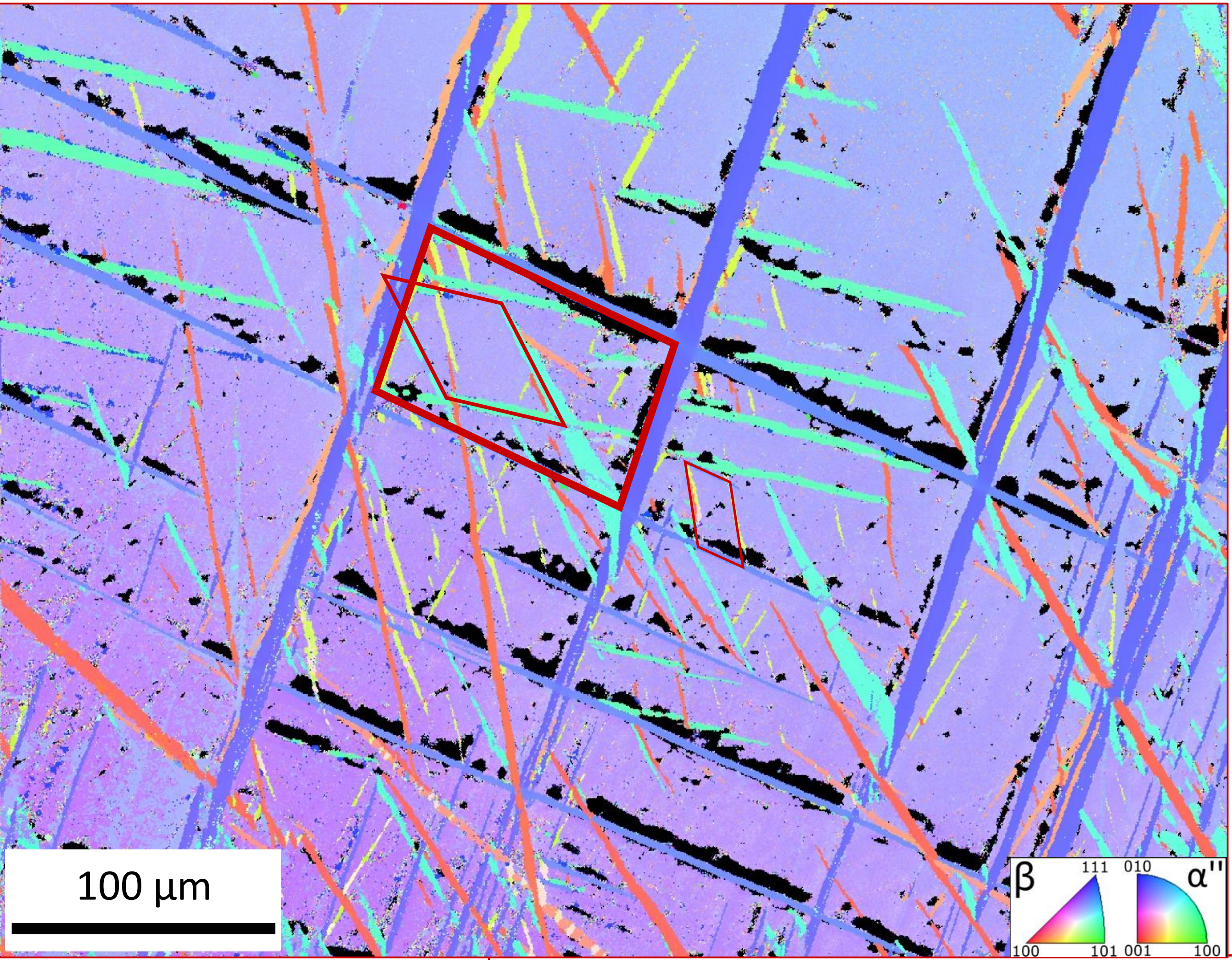
Hierarchy of Microstructural Evolution



Primary product

Secondary product

Hierarchy of Microstructural Evolution



Primary product

Secondary product

Tertiary product

Without mentioning fine scale structure!

Fine Scale Structure (TEM)

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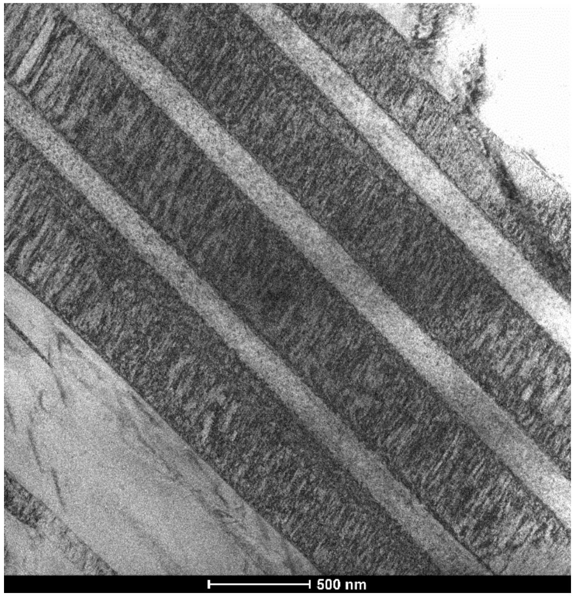
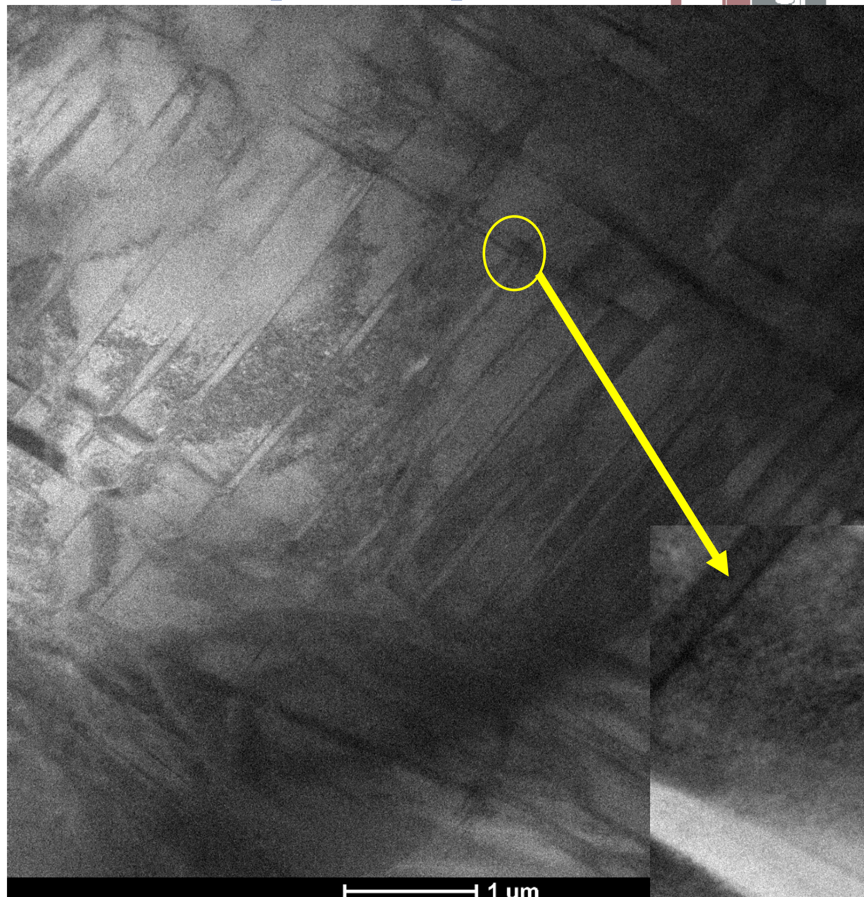
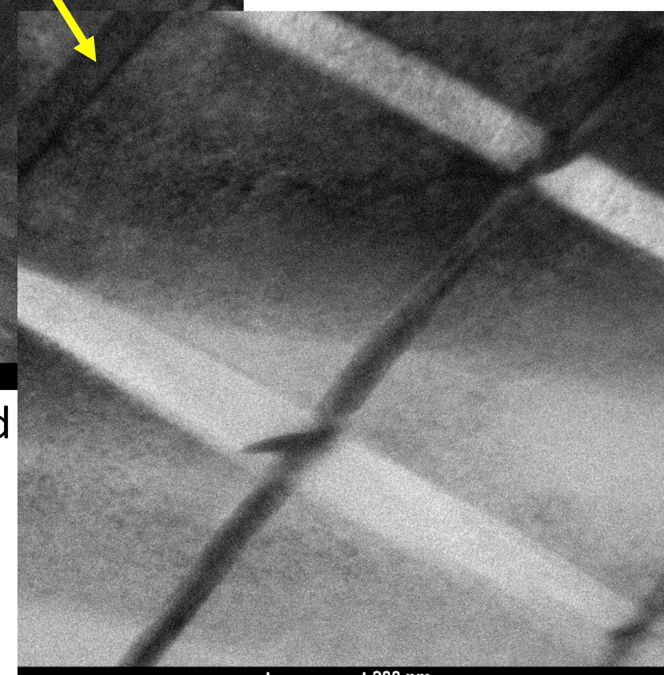
900 s at 423 K



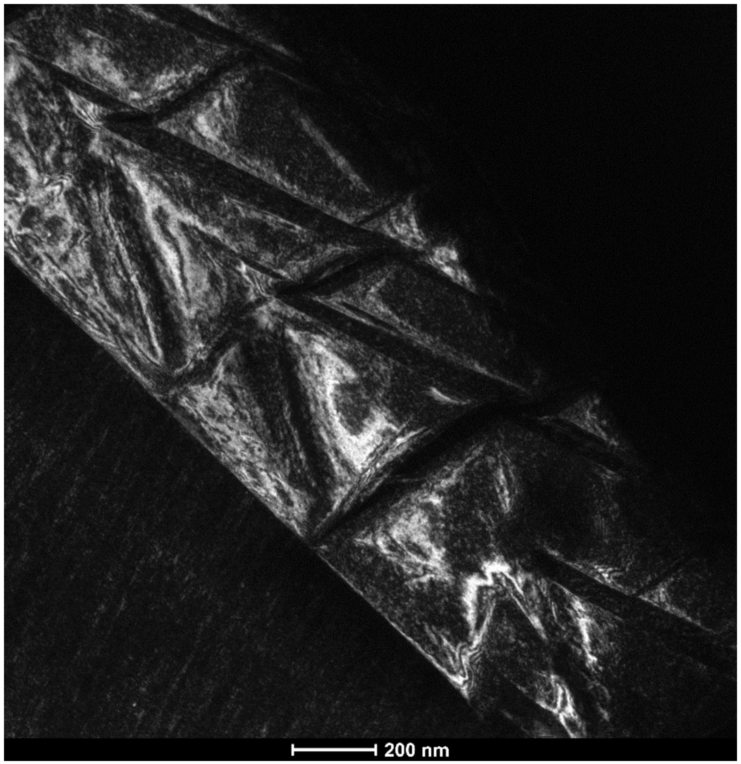


Fractured



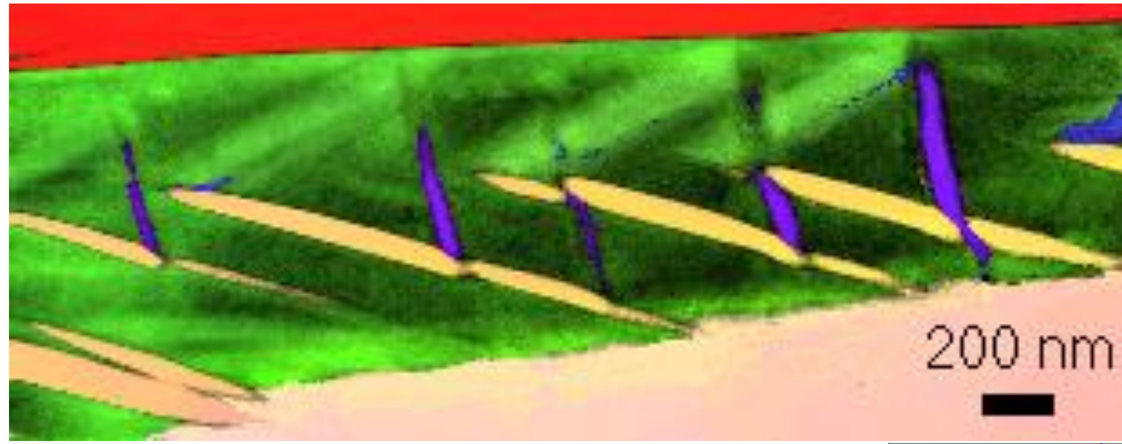
0.5% Plastic strain

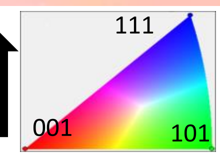
Fine Scale Structure (TEM/ASTAR)

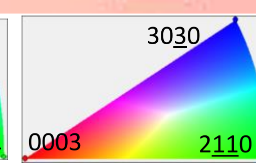


Dark field image taken from $\{112\}_\beta$

Orientation*Index map



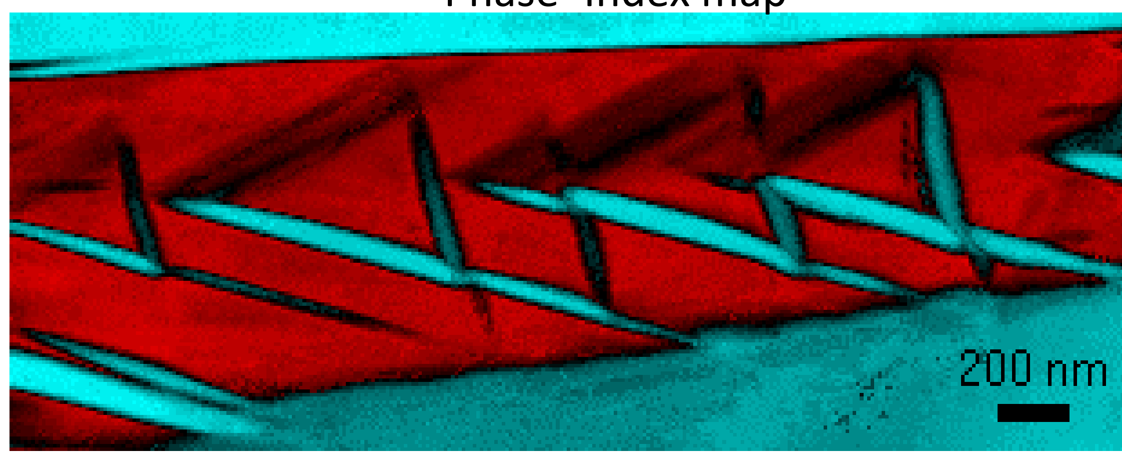




Martensite

β Phase

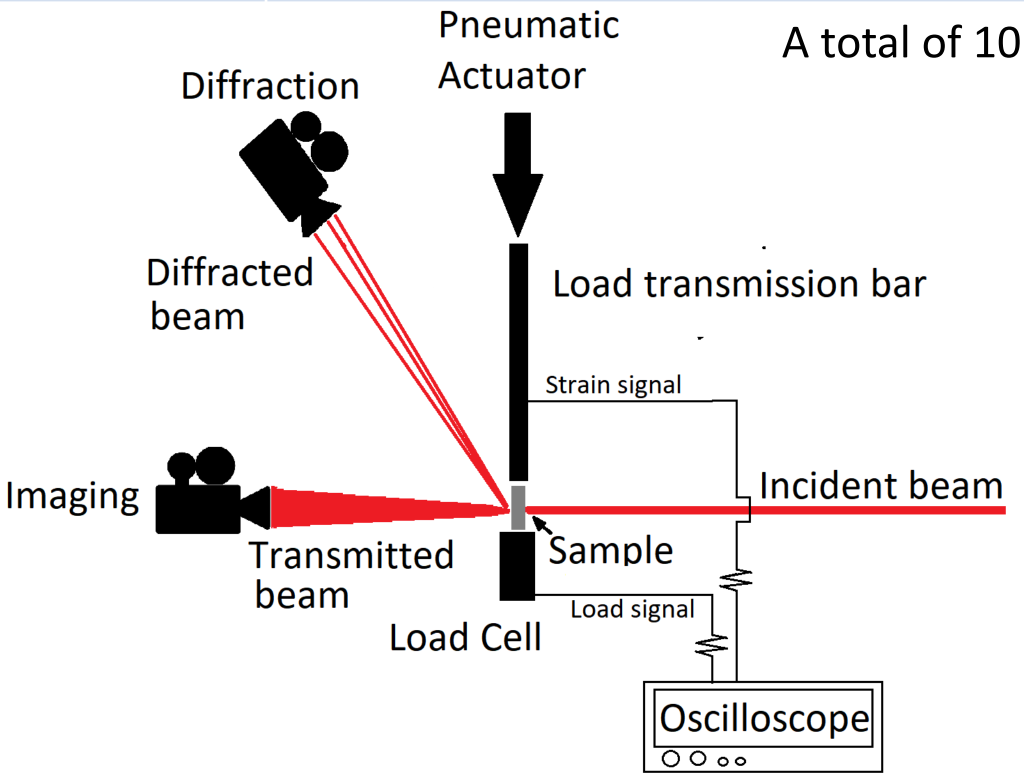
Phase*Index map



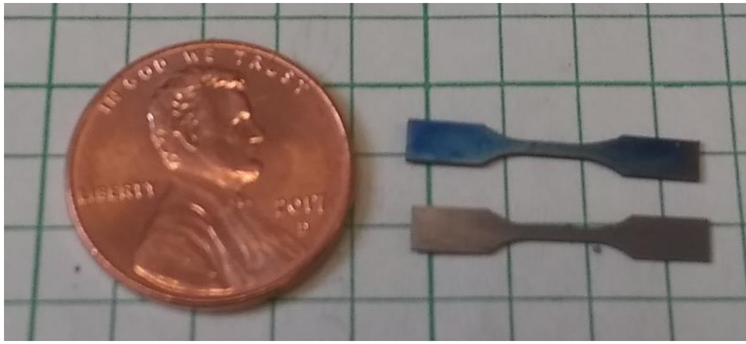
ASTAR courtesy of Jing at NanoMEGAS

APS 32-ID Pressure Bar Testing

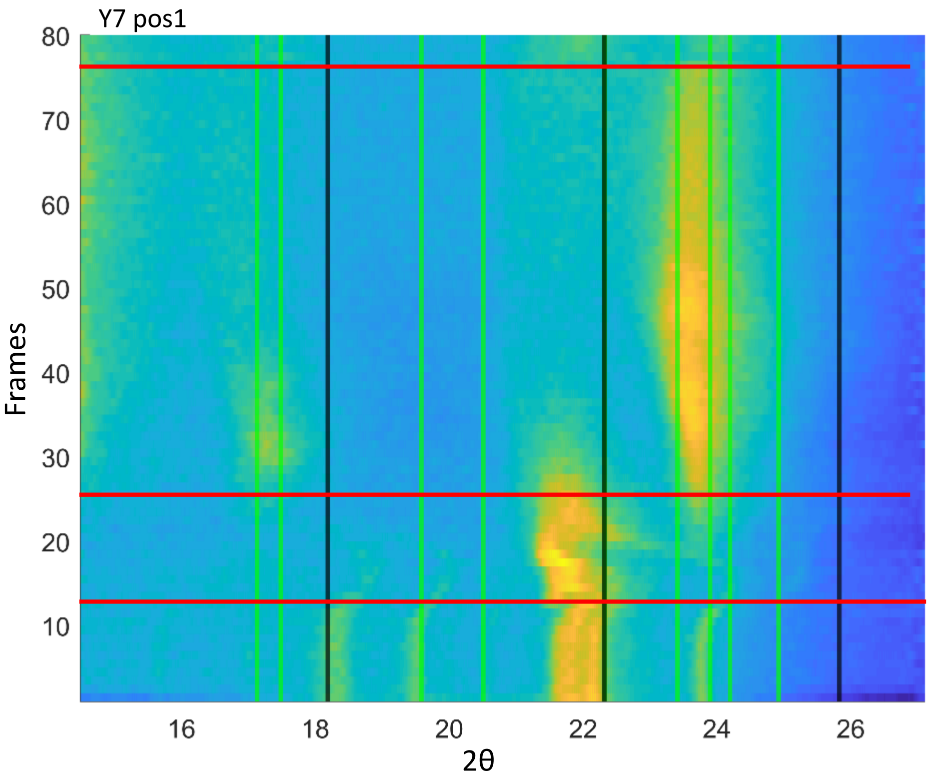
Alloys (wt %)	Name of condition and Heat Treatments		
Ti-10V-2Fe-3Al	AQ 1123K-0.5h→WQ / TRIP	MTS AQ+423K-900s / TRIP	TI AQ+423K-7200s / SLIP
Ti-15Mo	AQ 800-1h→WQ / TWIP		
Ti-12Mo	AQ 820-1h→WQ / TRIP+TWIP		

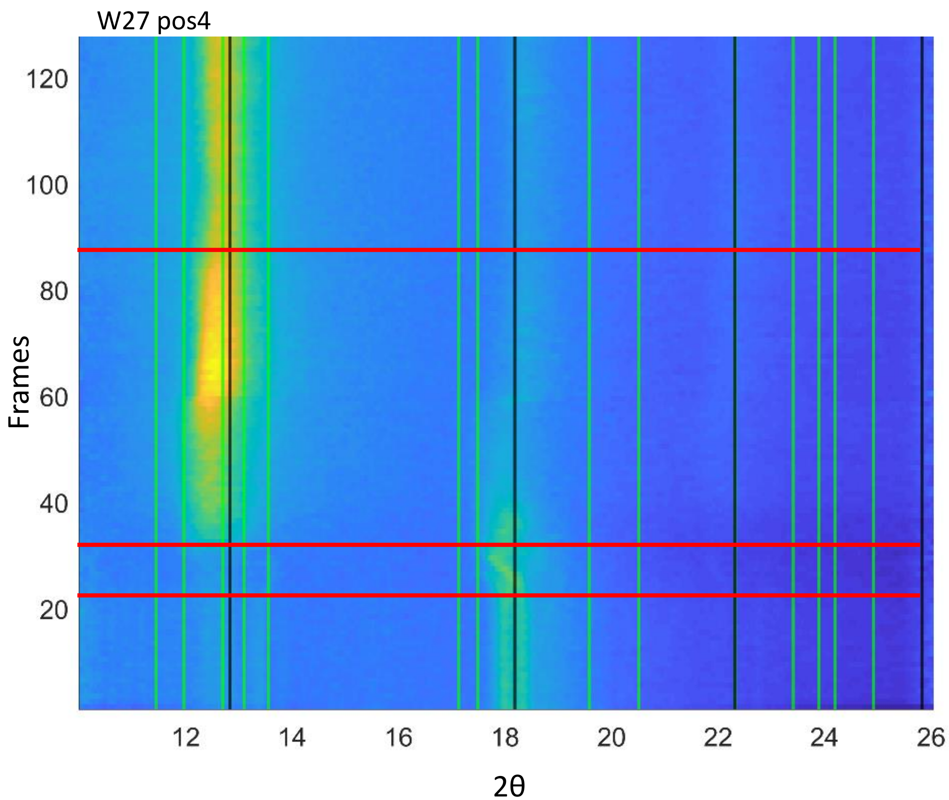


A total of 102 β–Ti samples were tested in Feb 2020



Twinning vs Transformation





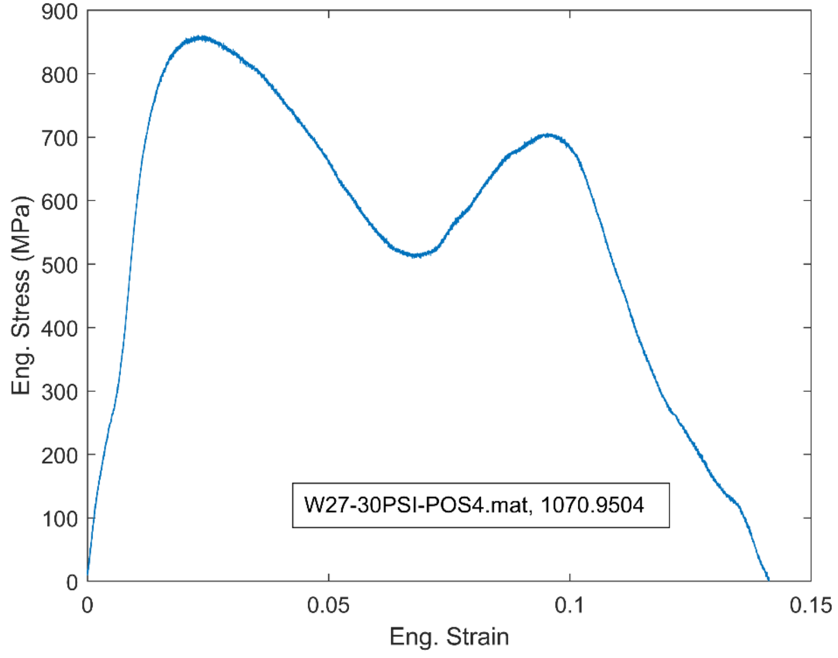
TRIP Ti-1023

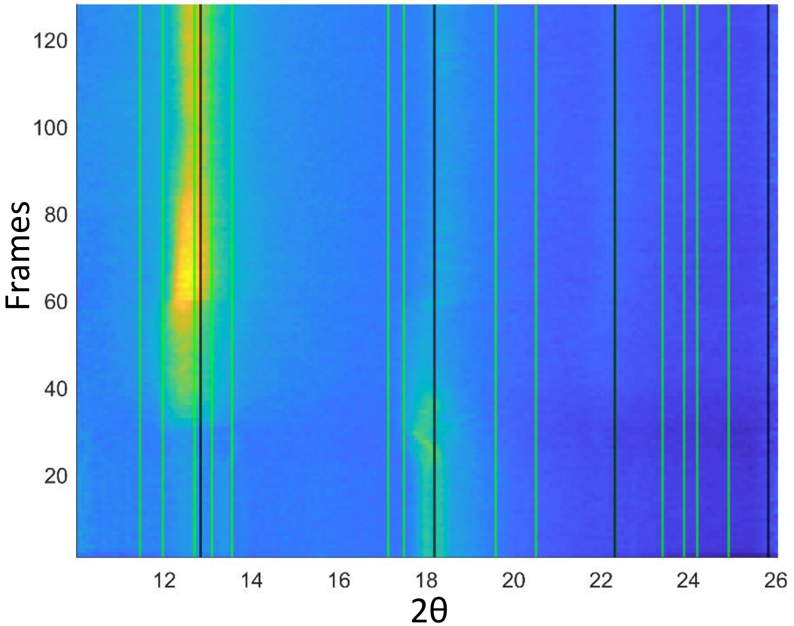
TWIP Ti-15Mo

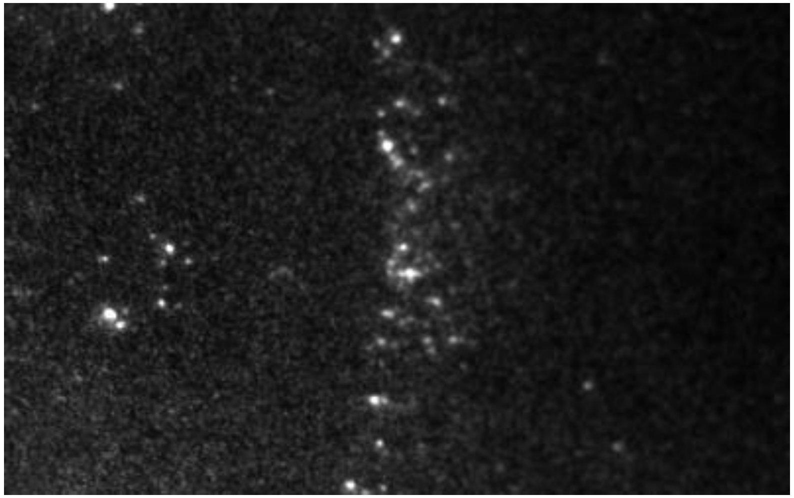
Green: α”
Black: β

Deformation at ~1000 s^{-1}

In-situ TWIP Ti-15Mo Data

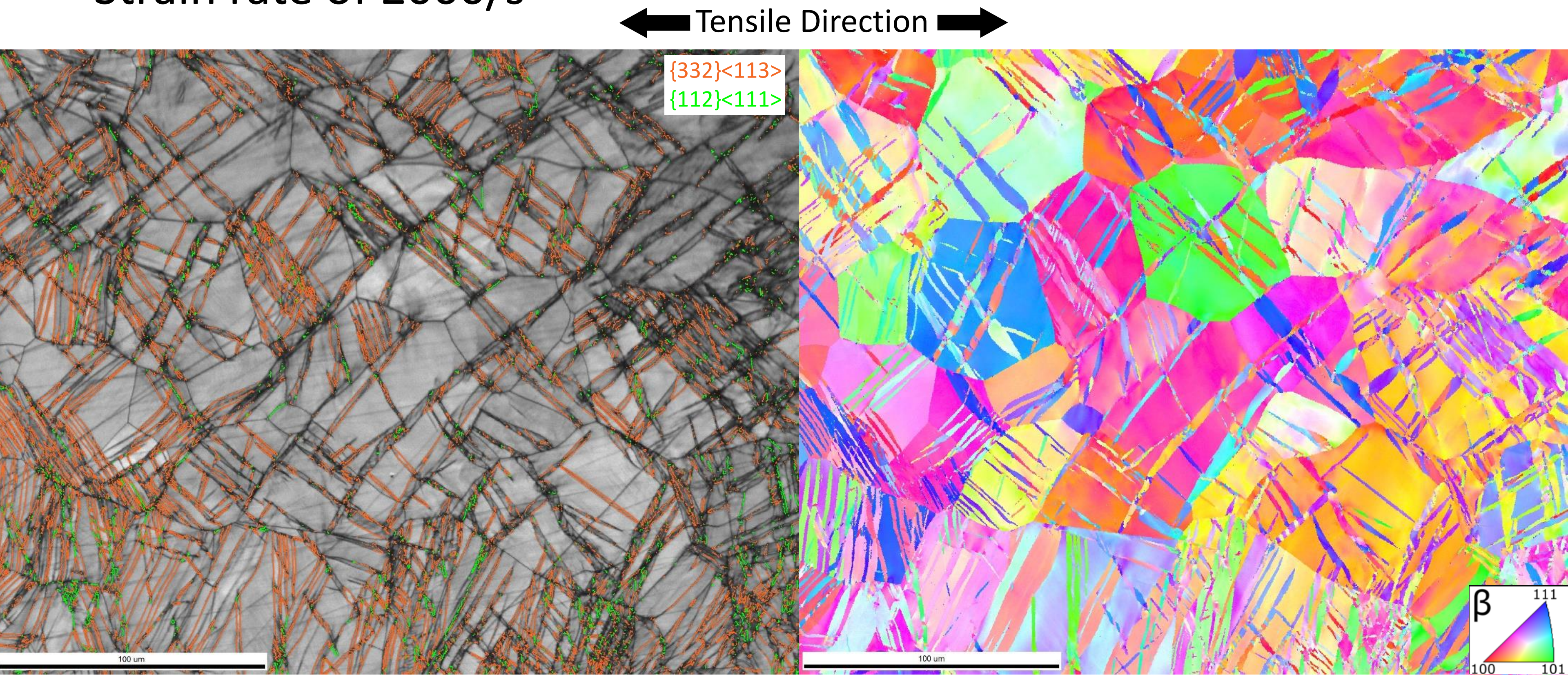






Ti-15Mo High Rate Tension EBSD Overview

- Tested at 75 psi in position 2
- Strain rate of 2000/s

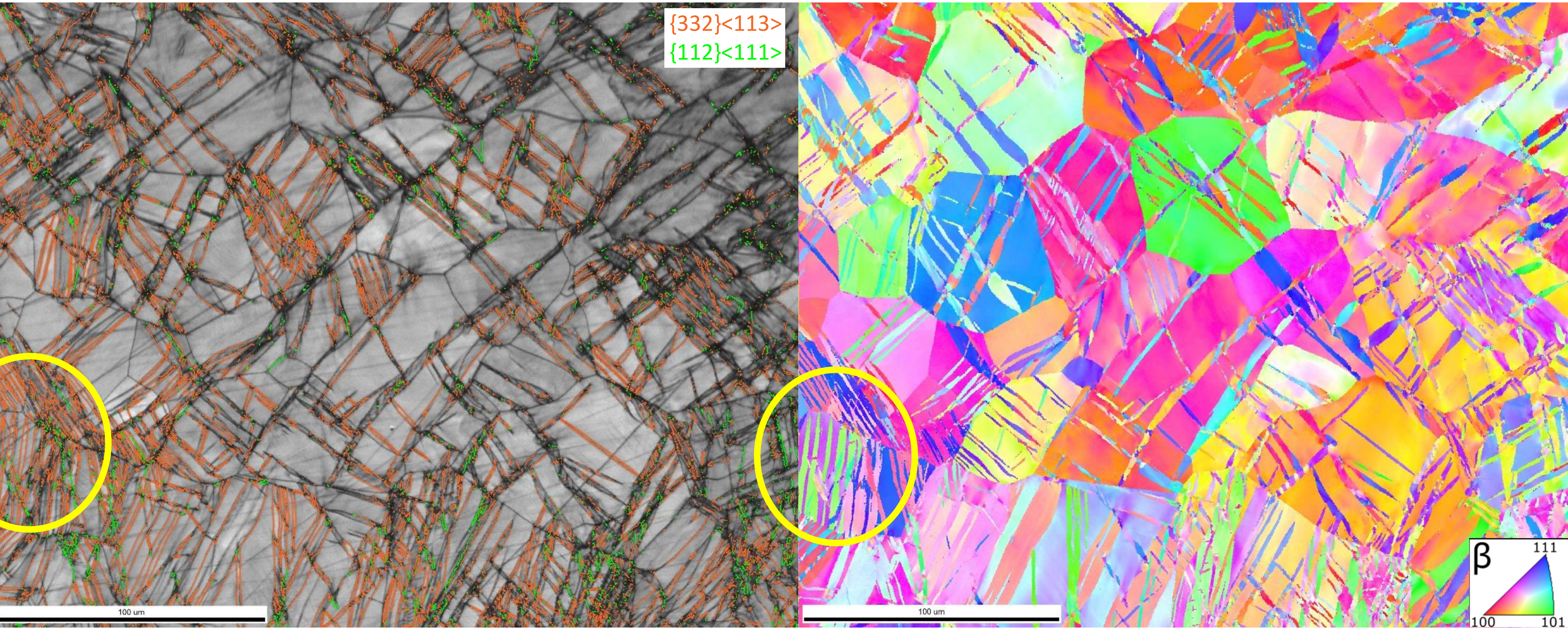


IQ map + Twin Boundaries

IPF map

Ti-15Mo High Rate Tension EBSD Overview

- Tested at 75 psi in position 2
- Strain rate of 2000/s



Primary {332}<113> Twins

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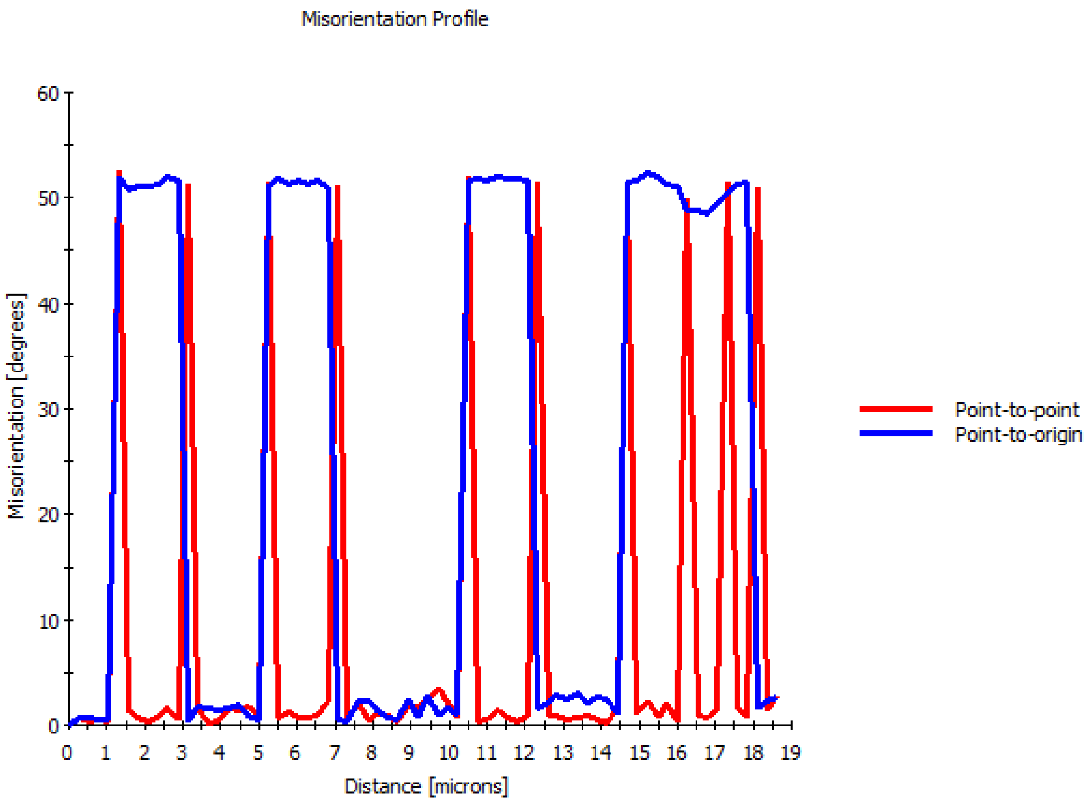
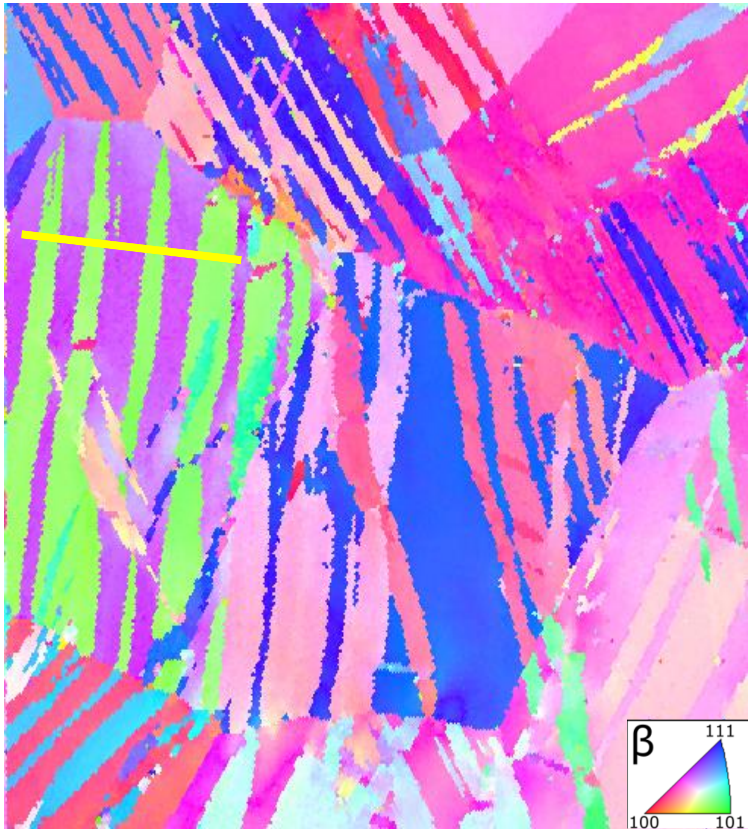
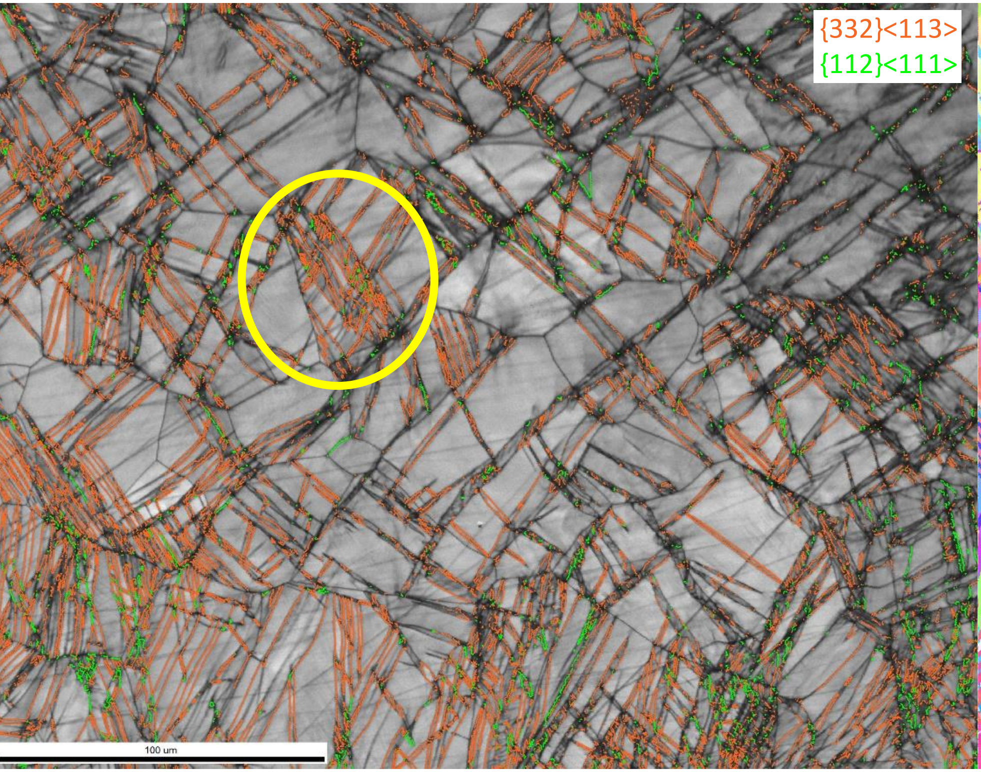


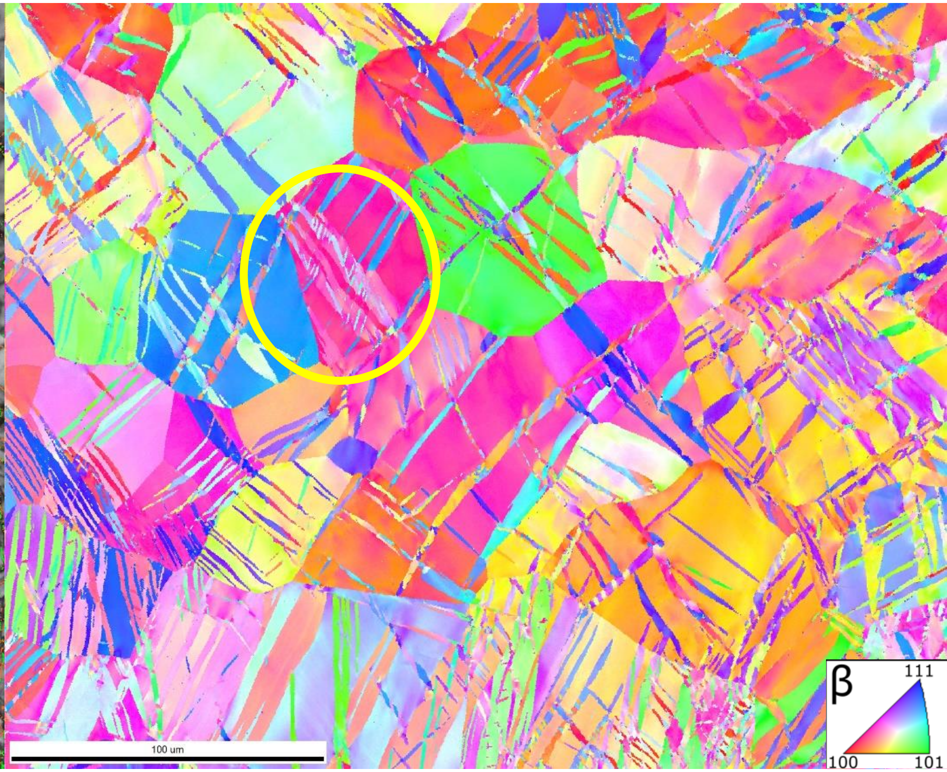


{332}<113> are characterized by a Σ11 CSL boundary with 50.5° misorientation relative to [110]

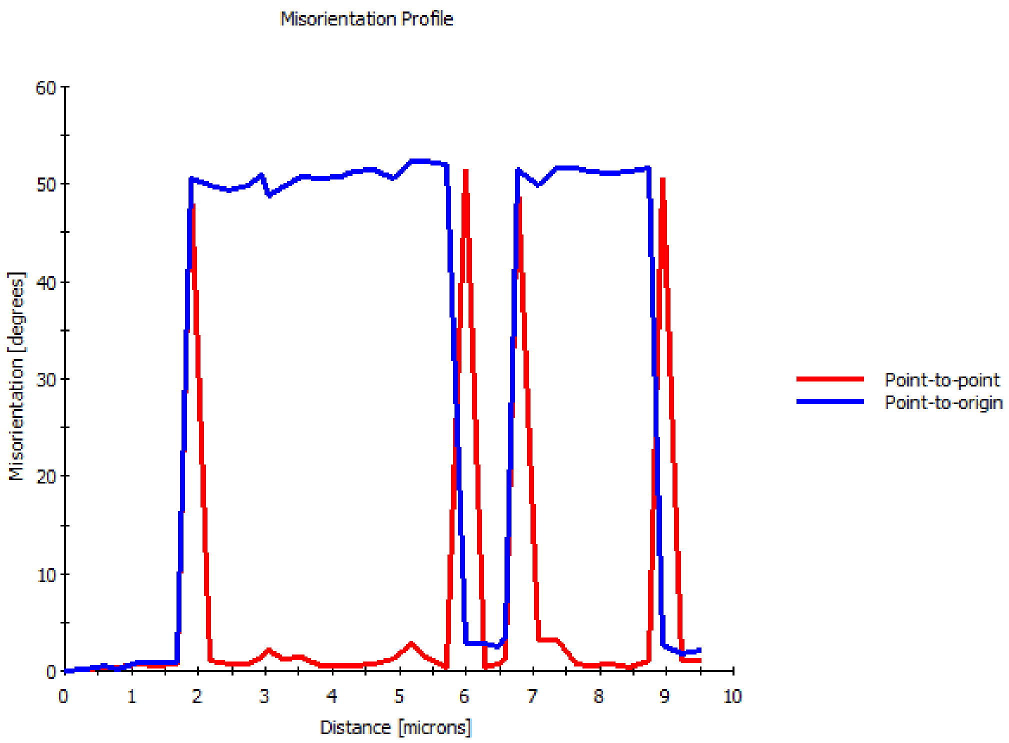
Ti-15Mo High Rate Tension EBSD Overview

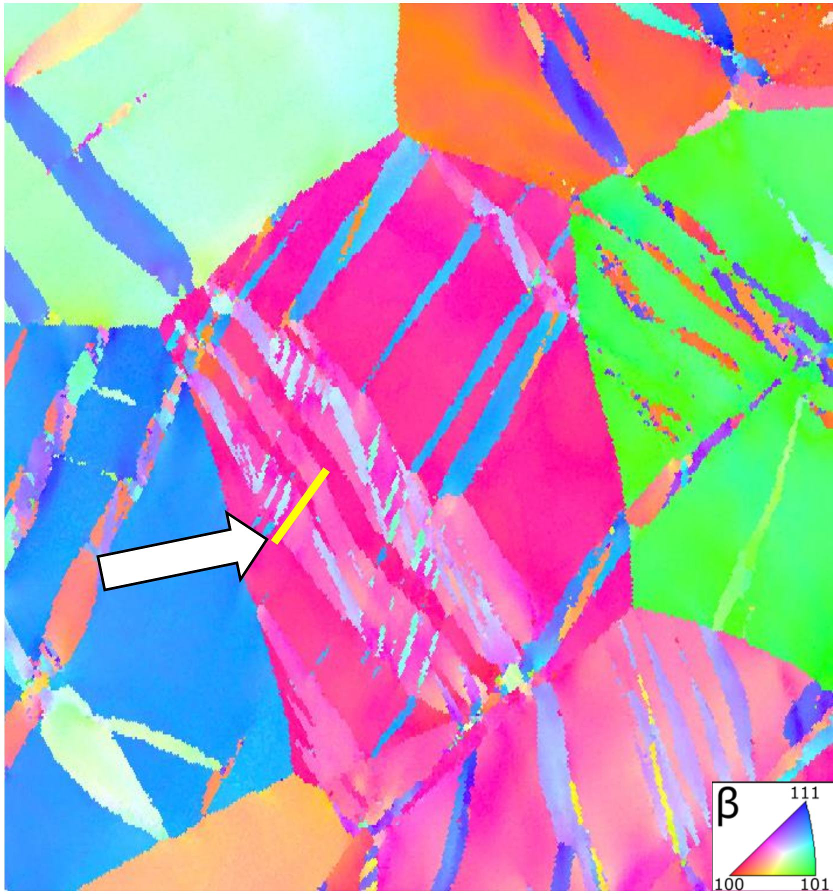
- Tested at 75 psi in position 2
- Strain rate of 2000/s



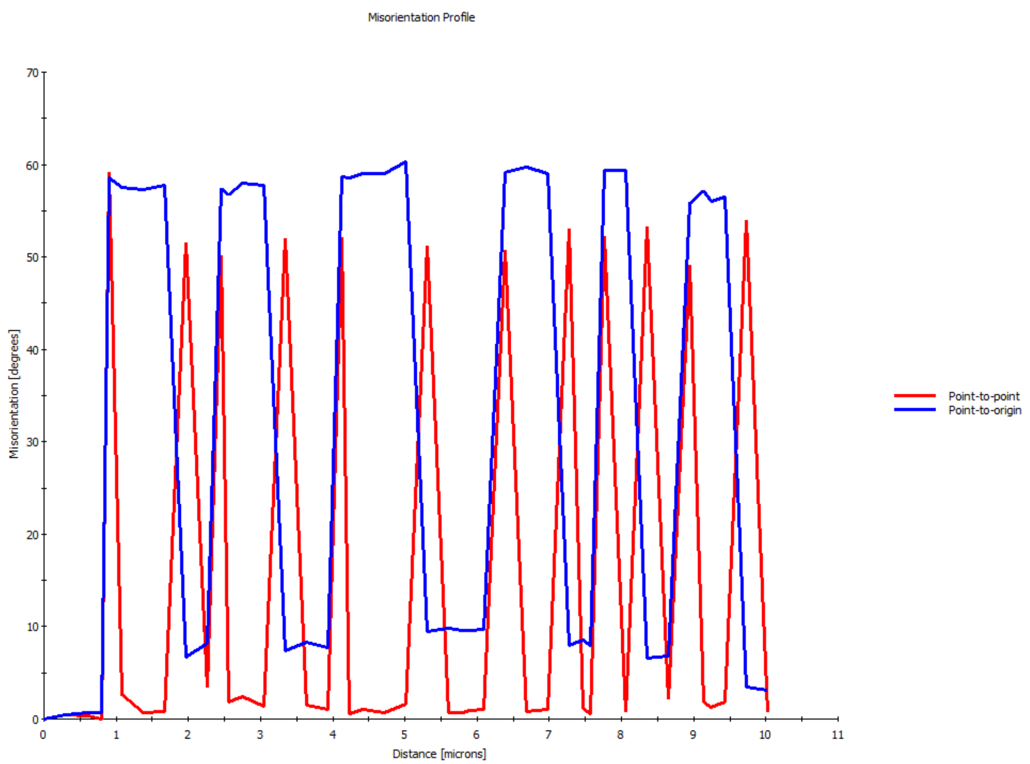


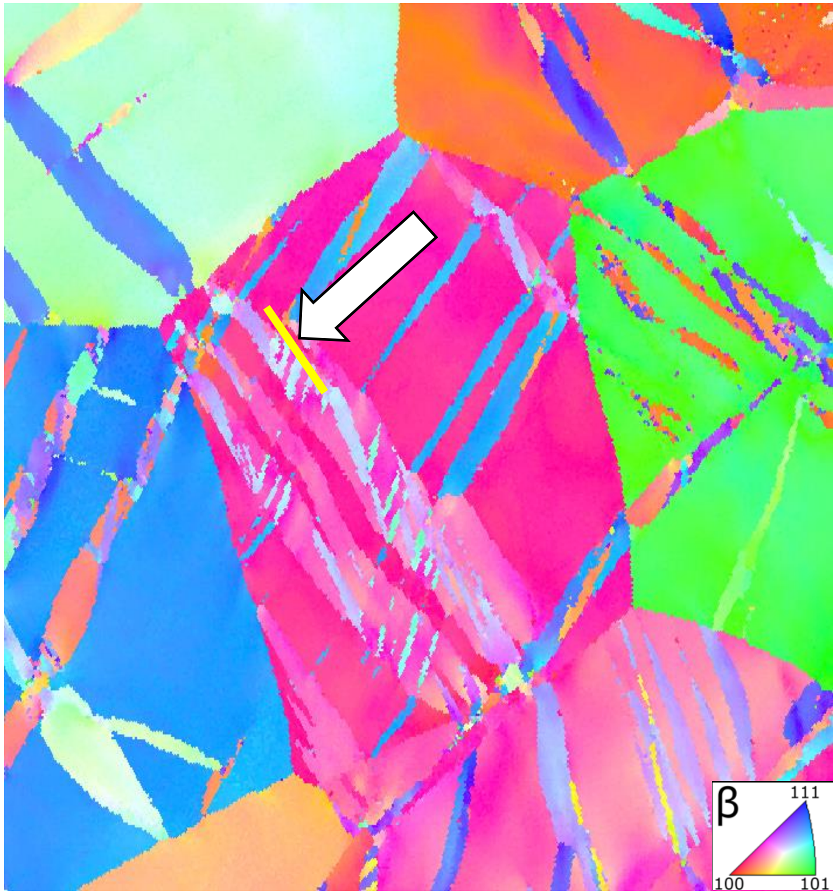
Primary {332}<113> Twins





Secondary {332}<113> Twins

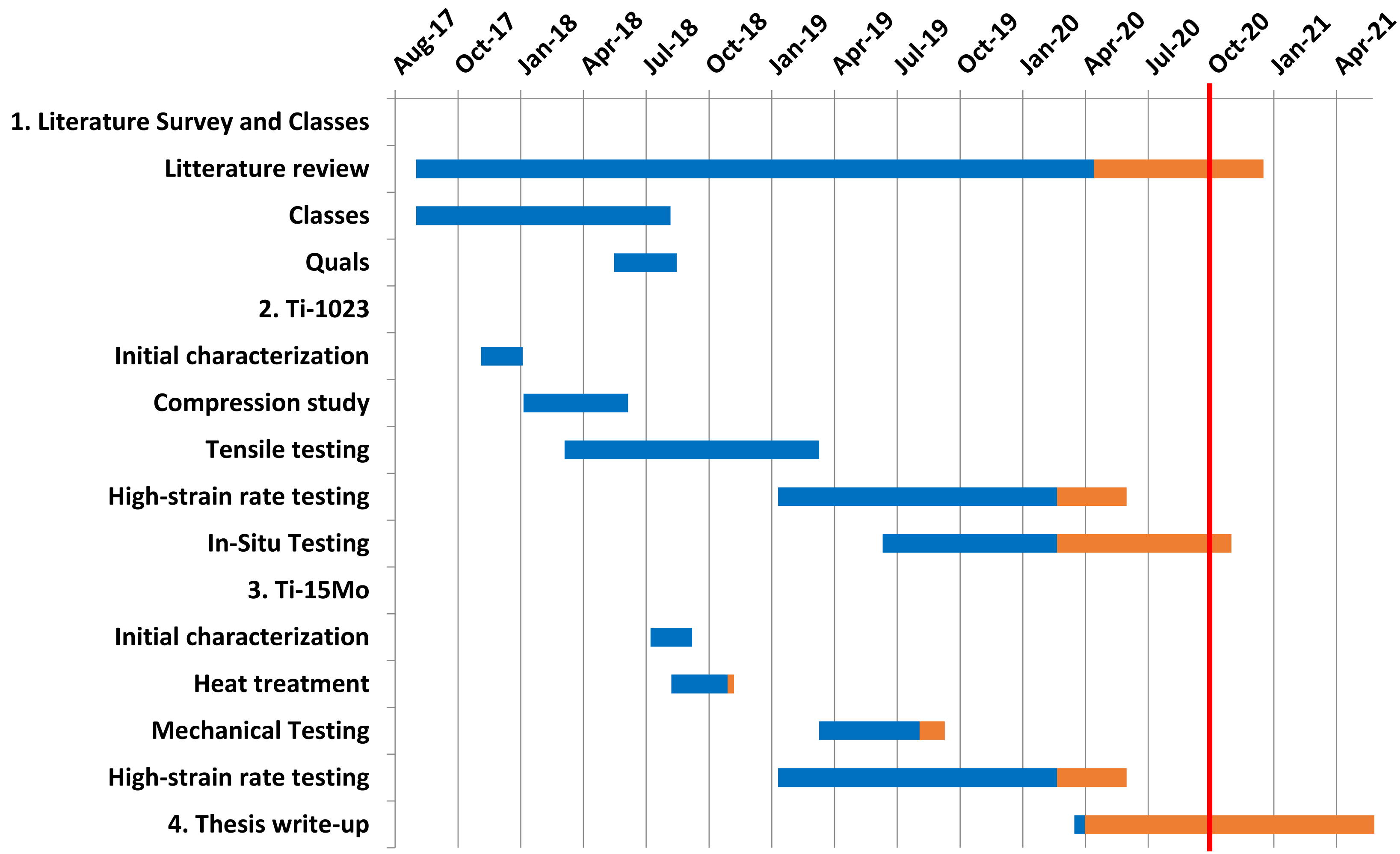




Conclusions

- EBSD is working great!
- Low-temperature aged Ti-1023 remains TRIP dominant, implying that aging affects the transformation stress without affecting chemical stability
- Ti-15Mo remains TWIP dominant at high-rates
- High rate deformation of Ti-15Mo activates {112}<111> twinning, which is unreported
- A publication was prepared on the effect of low T aging on the strength of Ti-1023

Progress



Aug-17
Oct-17
Jan-18
Apr-18
Jul-18
Oct-18
Jan-19
Apr-19
Jul-19
Oct-19
Jan-20
Apr-20
Jul-20
Oct-20
Jan-21
Apr-21
1. Literature Survey and Classes
Litterature review
Classes
Quals
2. Ti-1023
Initial characterization
Compression study
Tensile testing
High-strain rate testing
In-Situ Testing
3. Ti-15Mo
Initial characterization
Heat treatment
Mechanical Testing
High-strain rate testing
4. Thesis write-up

Thank you!

Benjamin Ellyson
bellyson@mines.edu

Challenges & Opportunities

Challenges

- TEM has been down recently
- Ductility will have to be measured directly on APS specimens (and/or from radiography data)

Opportunities

- ω-phase strengthening provides increased yield strength without tradeoff to ductility
- Transition to TI condition is gradual, suggesting a larger window for in-service properties

Synthesis of Tested Materials

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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**

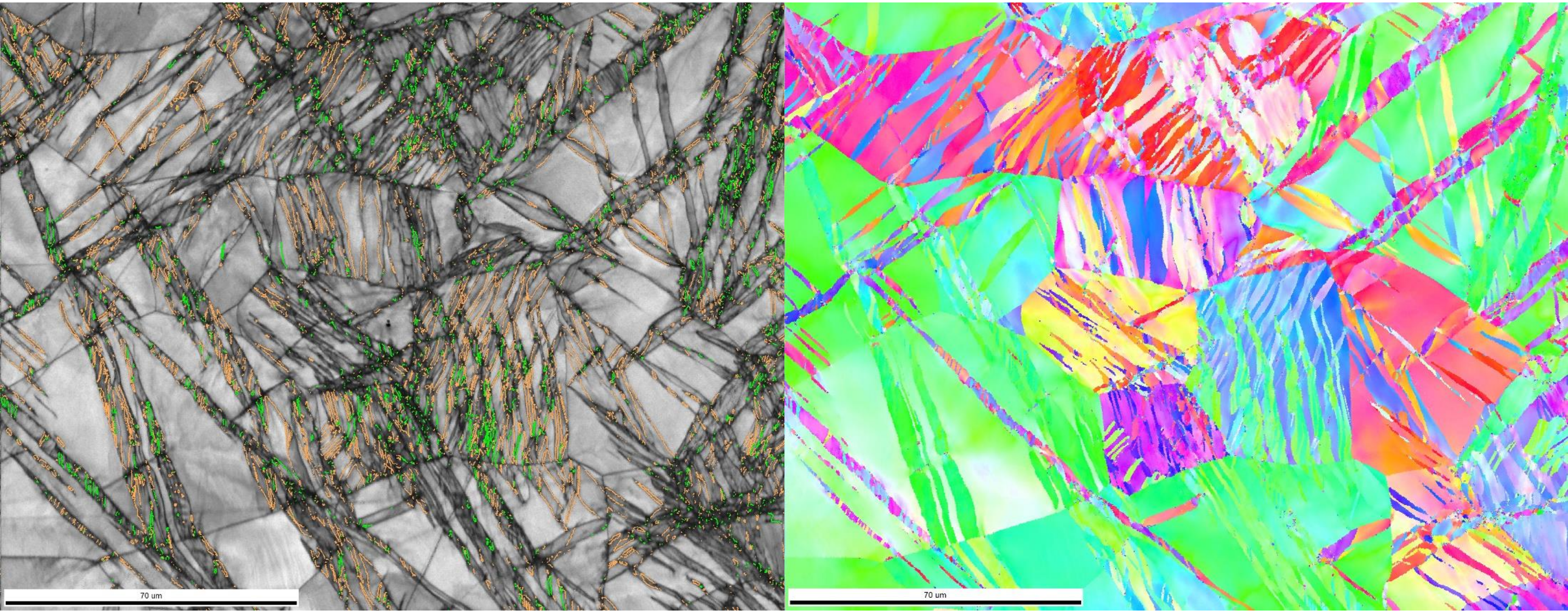
Secondary studies accompanying current work:

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.

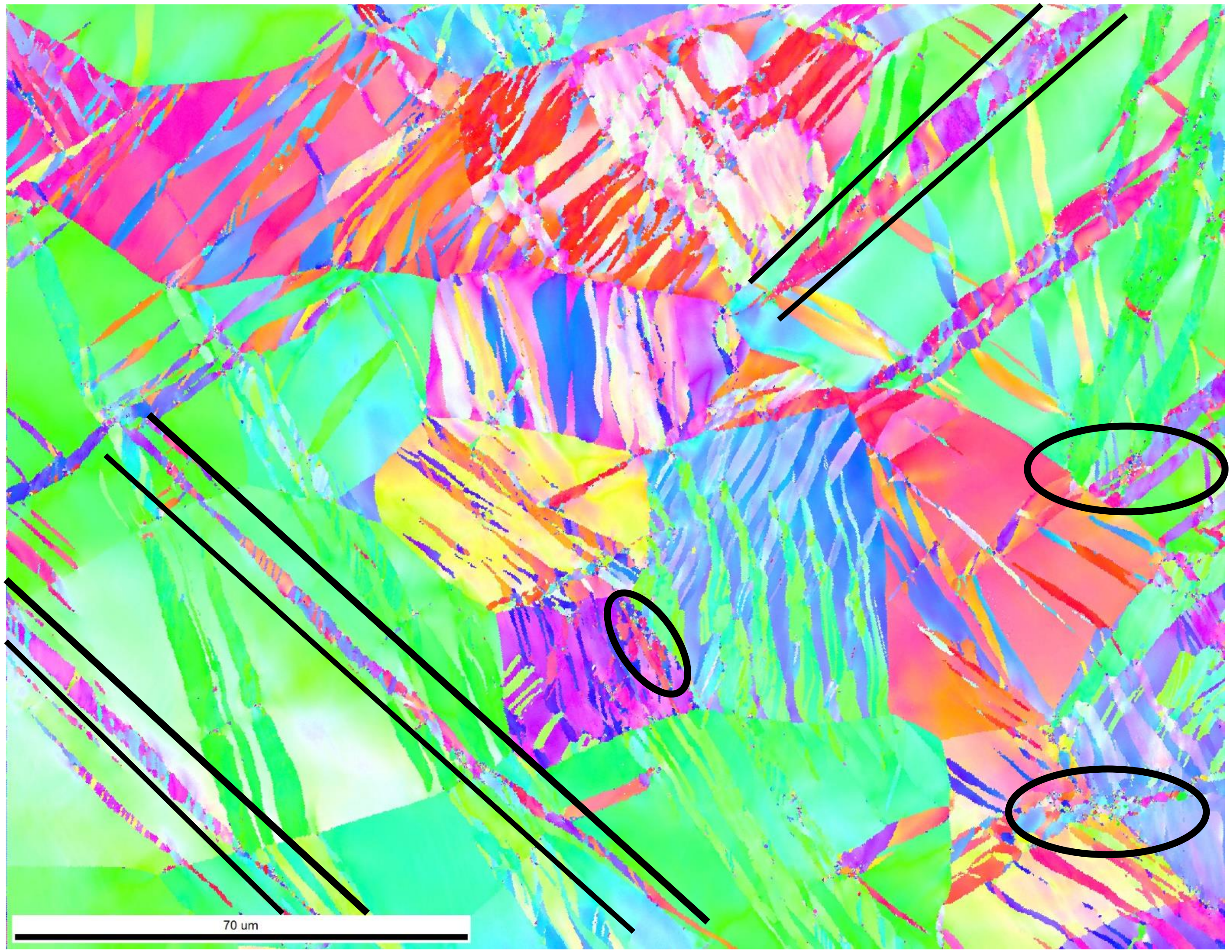
APS-W15 A4

- Tested at 30 psi in pos 4
- Strain rate of 1000/s

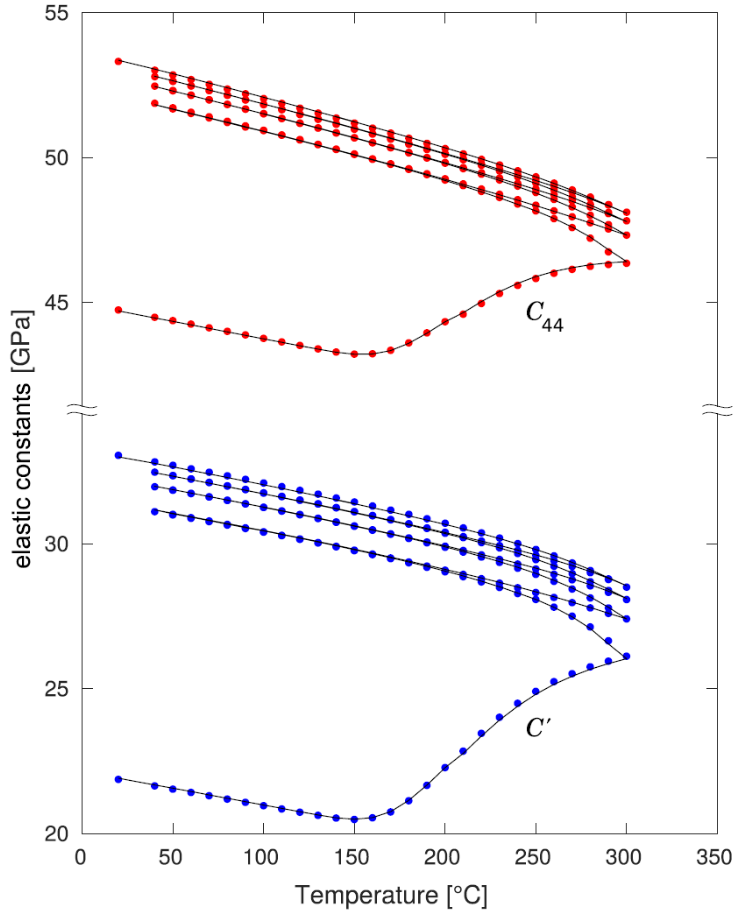
Tensile Direction

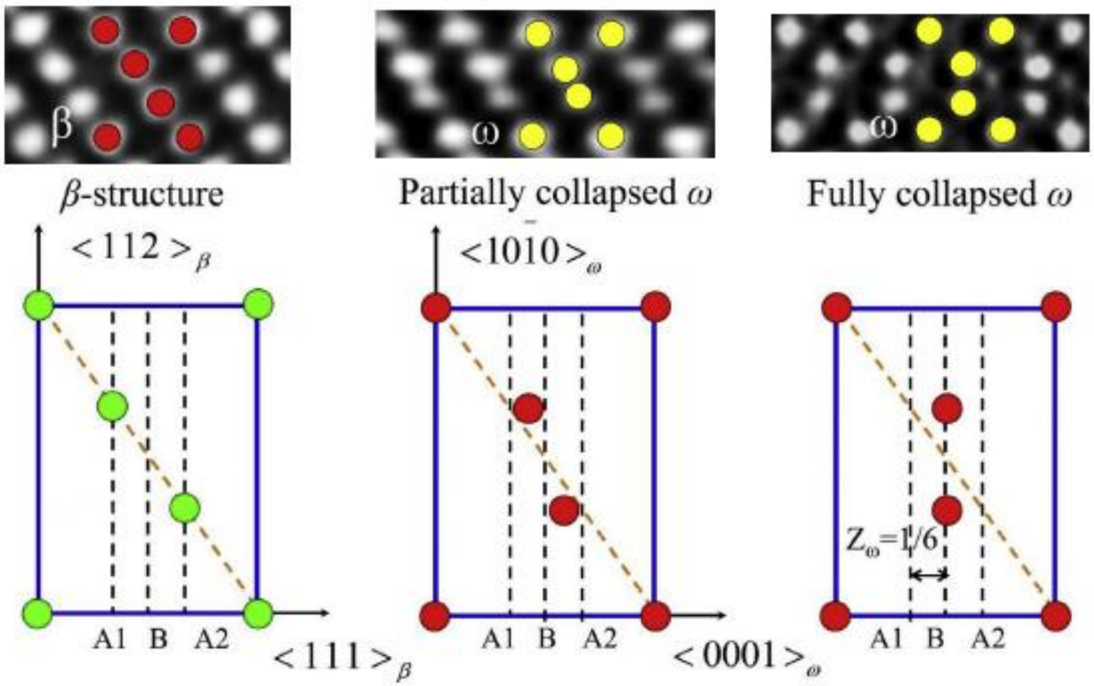


Amorphous or Nano Grains



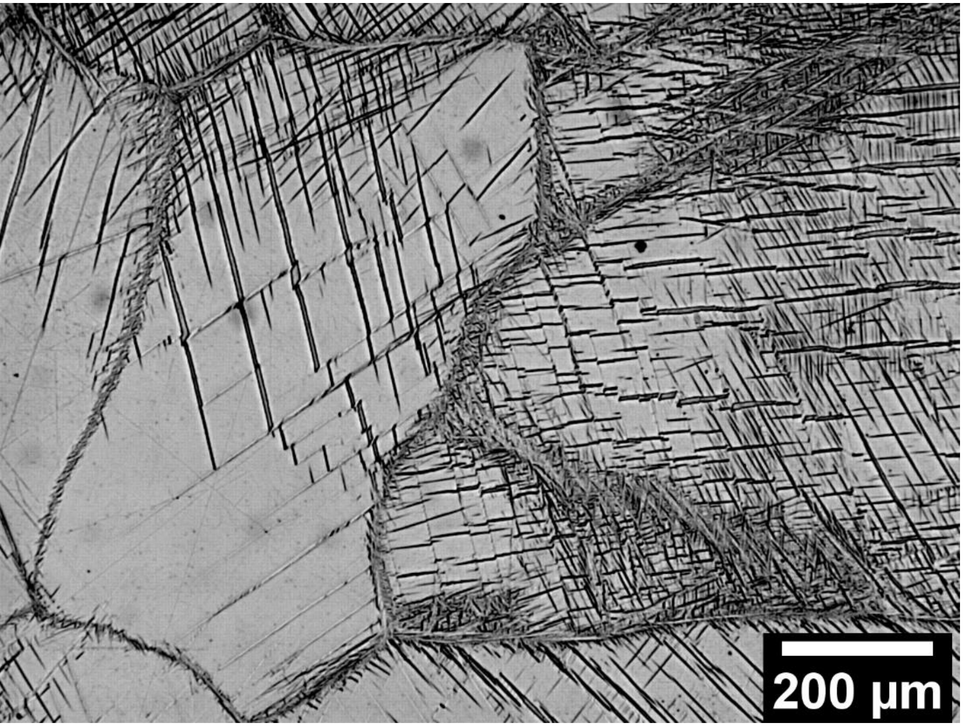
What is happening w/ ω-phase



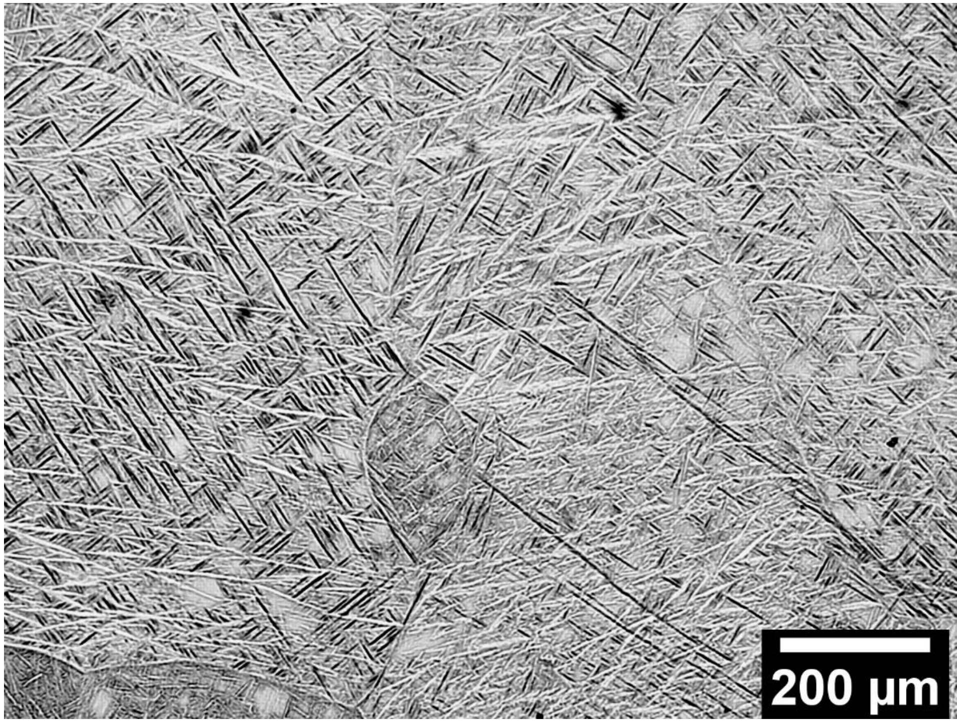


Comparison of Post-Yield Microstructure of Ti-1023

As-Quenched



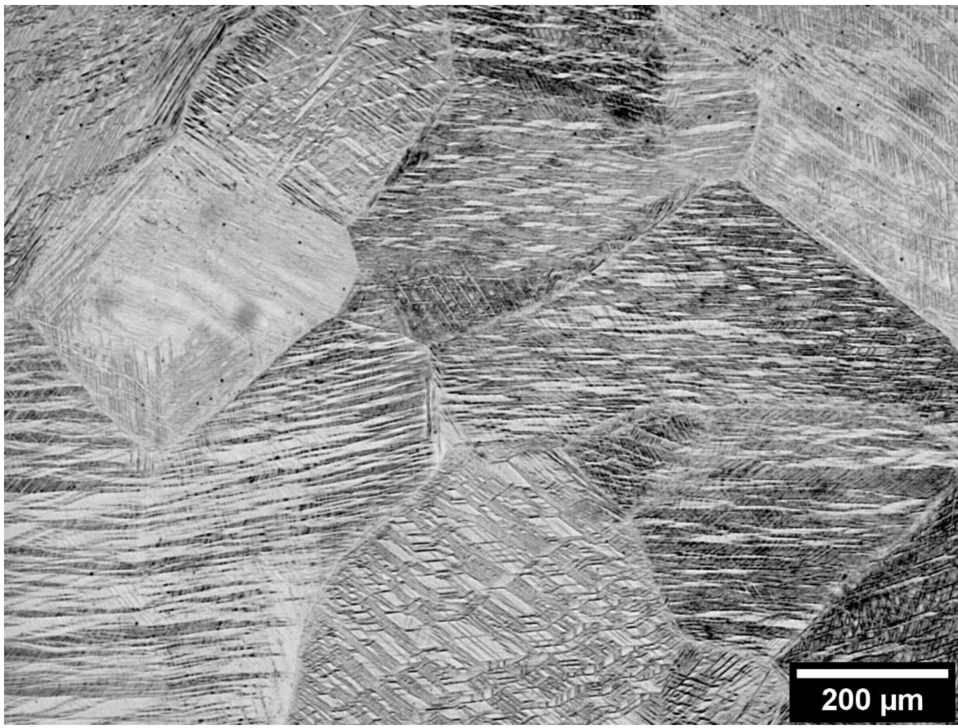
Aged 900s at 423K



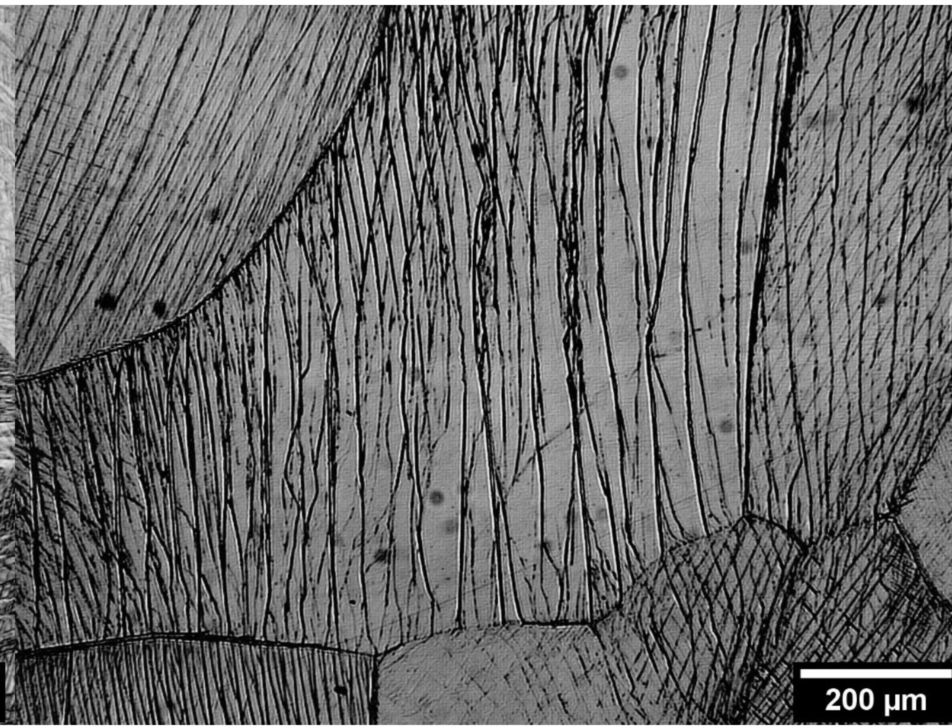
Deformed by a 0.5% plastic strain in tension at 10^{-3}/s

Comparison of Microstructure of Failed Tensile Specimens of Ti-1023

As-Quenched

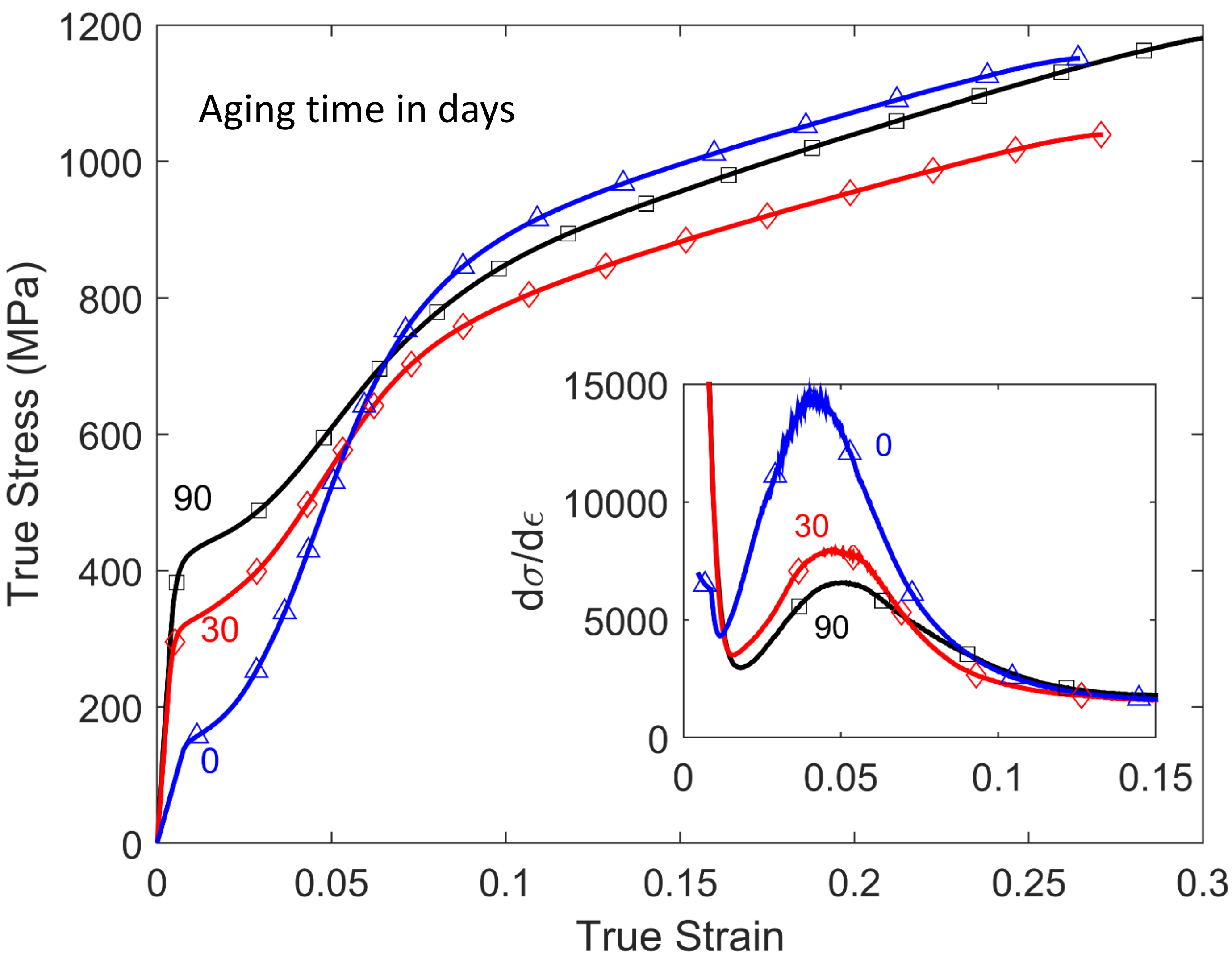


Aged 7200s at 423K

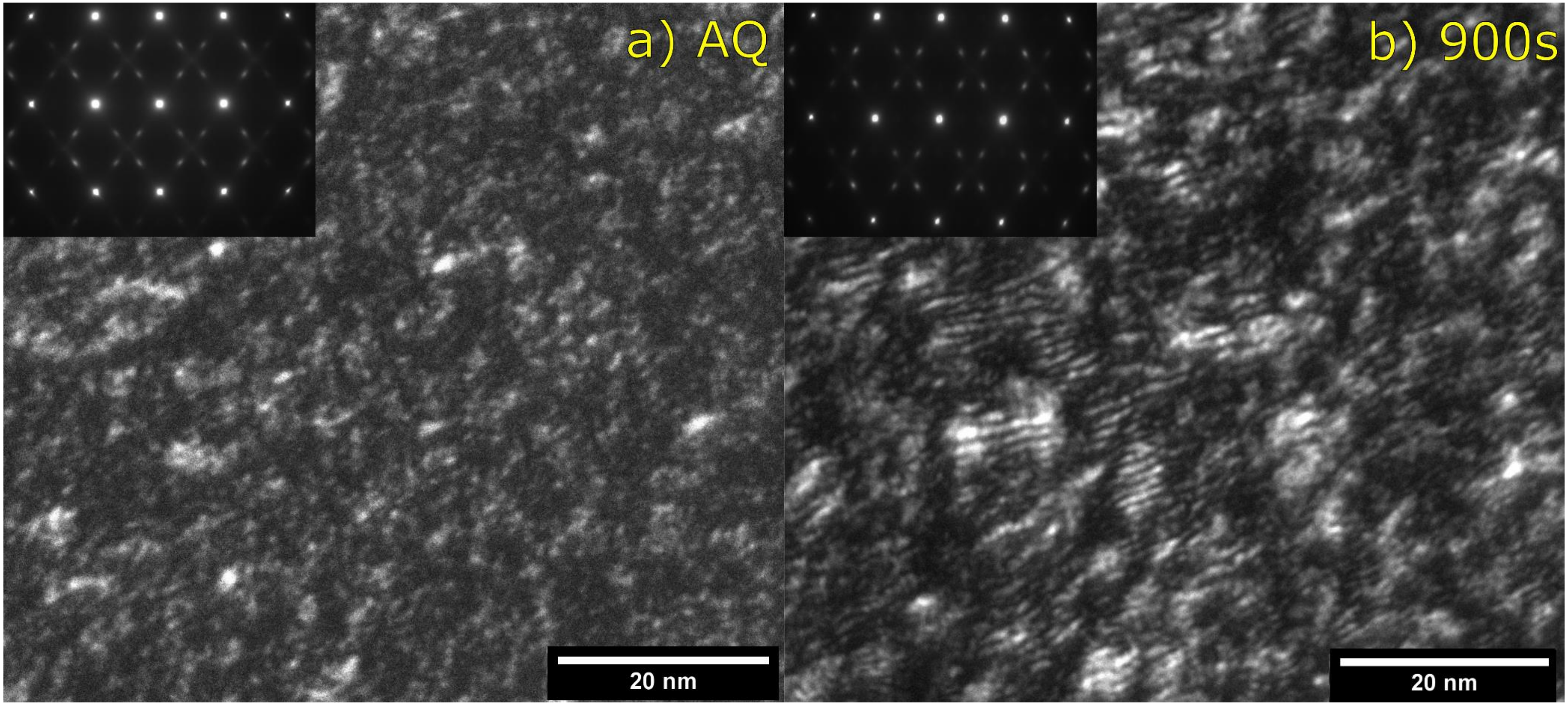


All images are from specimens failed in tension at 10^{-3}/s

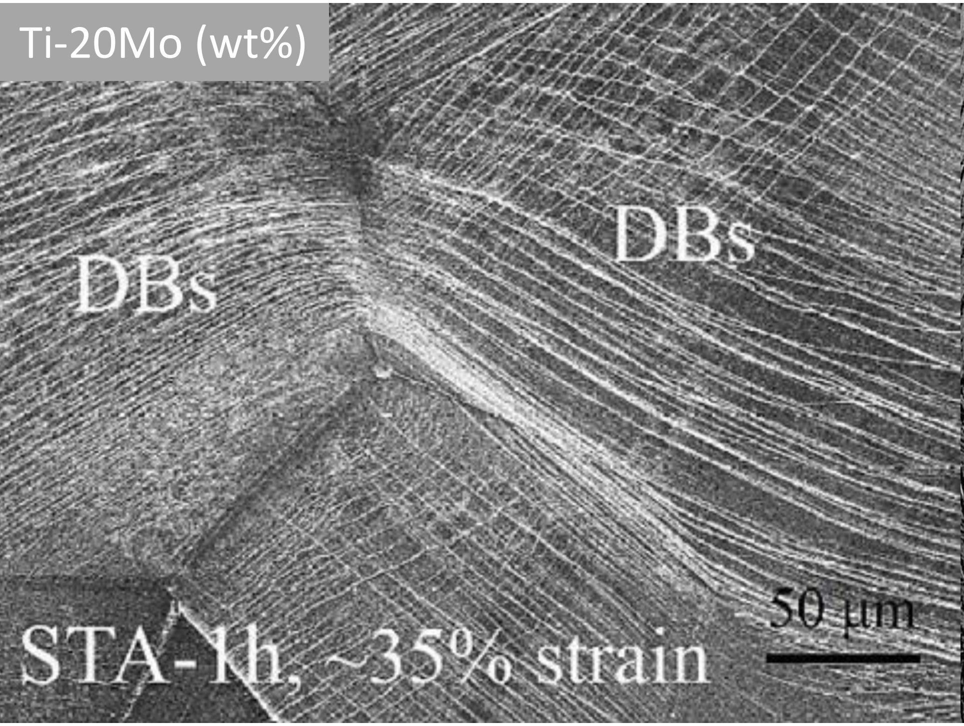
Natural Aging in Ti-1023



TEM of ω-phase Aging

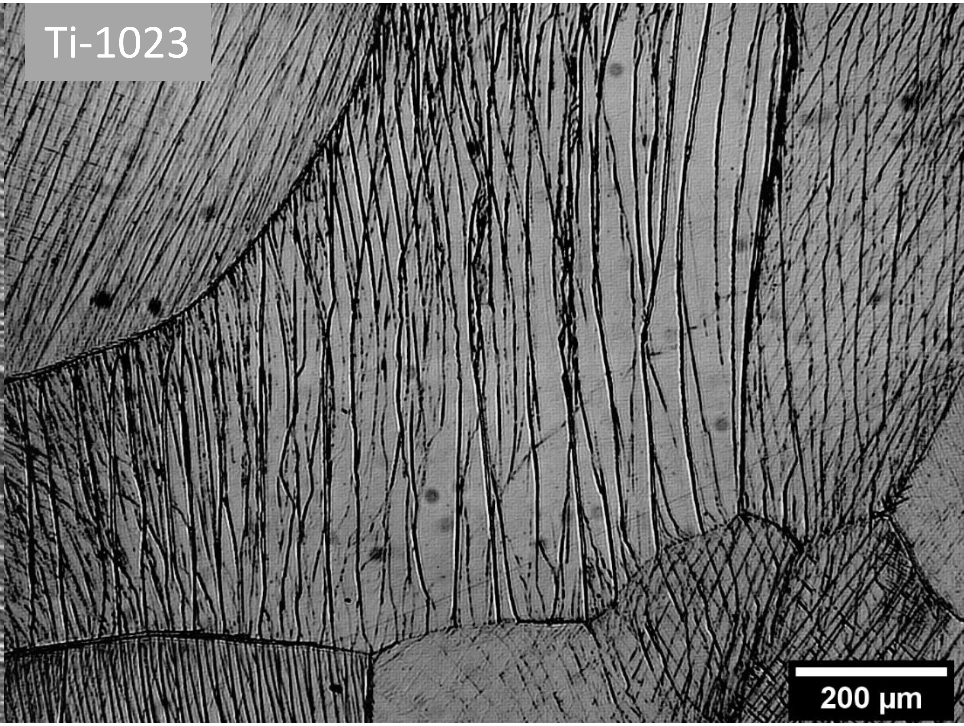


Comparison of Over-Aged (7200s, 423K) Microstructure of Ti-1023



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

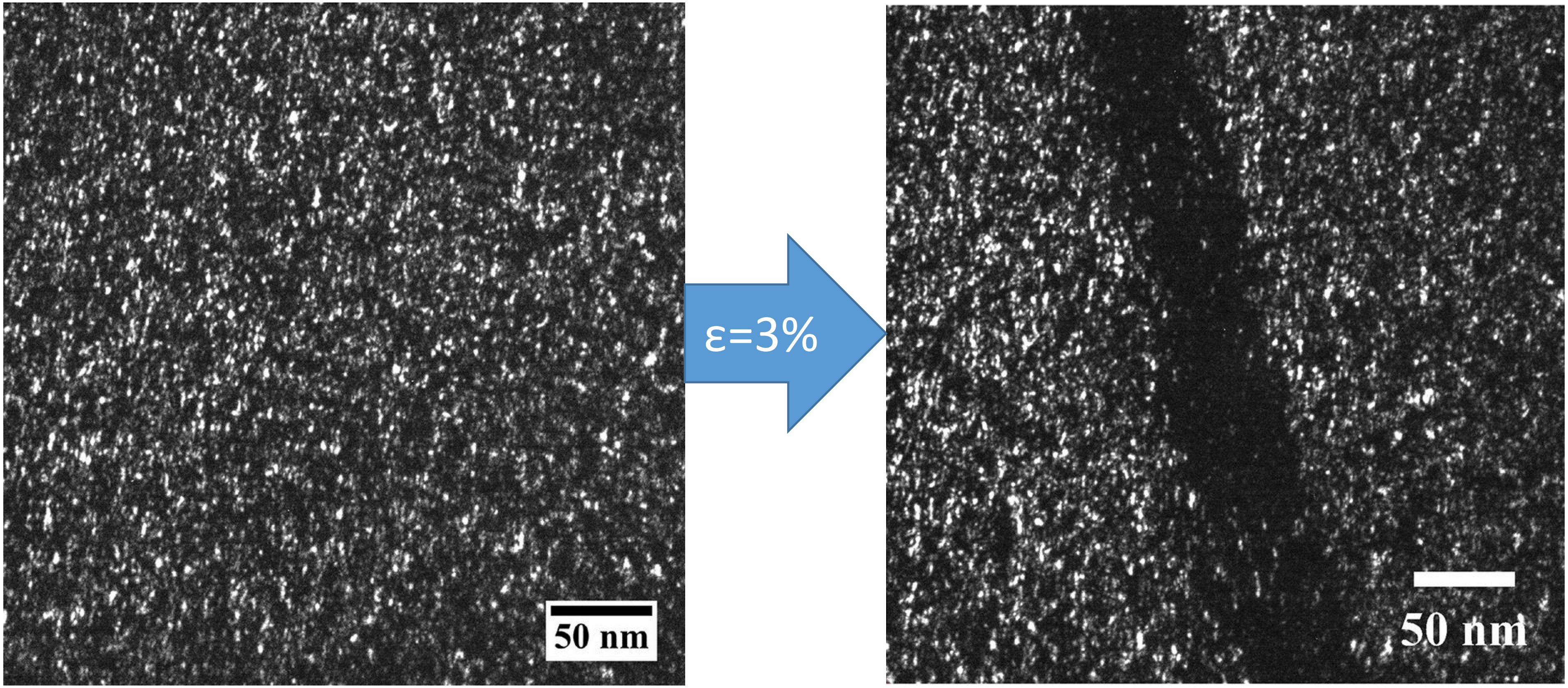
DBs : Dislocation bands



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

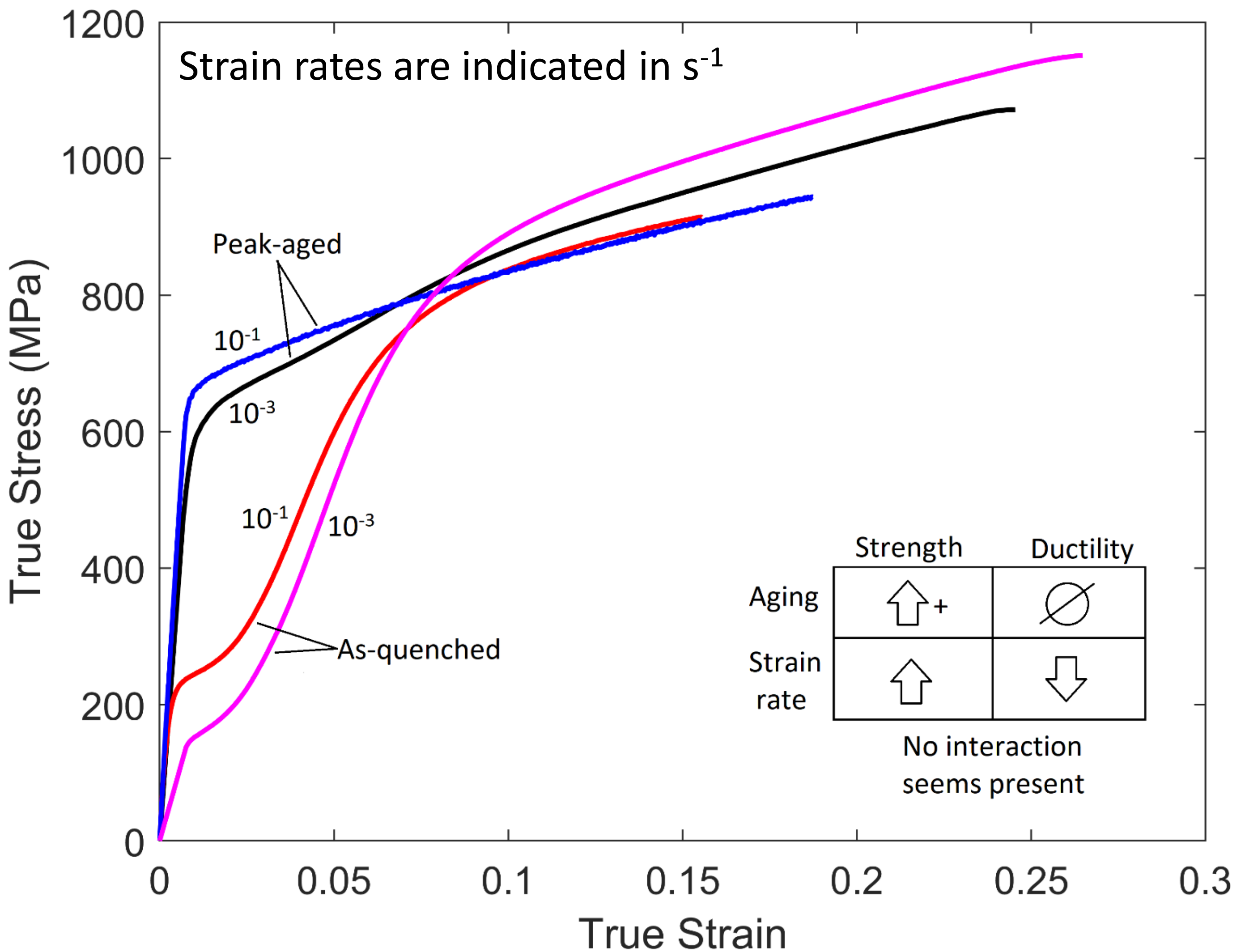
Artificial Aging of Ti-1023 for 7200s at 423K inhibits stress-induced martensite and causes dislocation bands to form

Dislocation Channeling



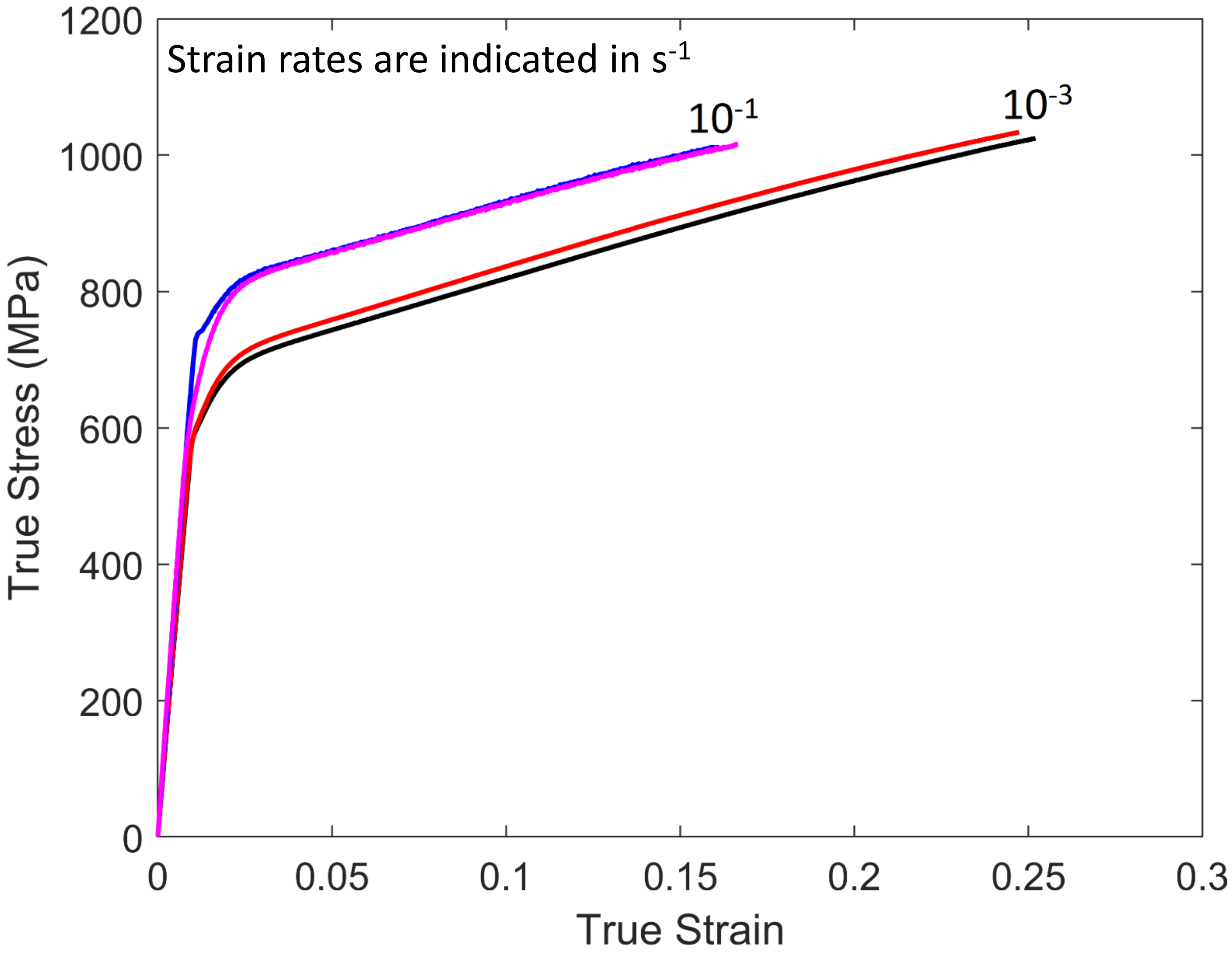
Lai et al. *Acta Materialia* 151 (2018): 67-77.

Strain Rate Effects on TRIP Ti-1023

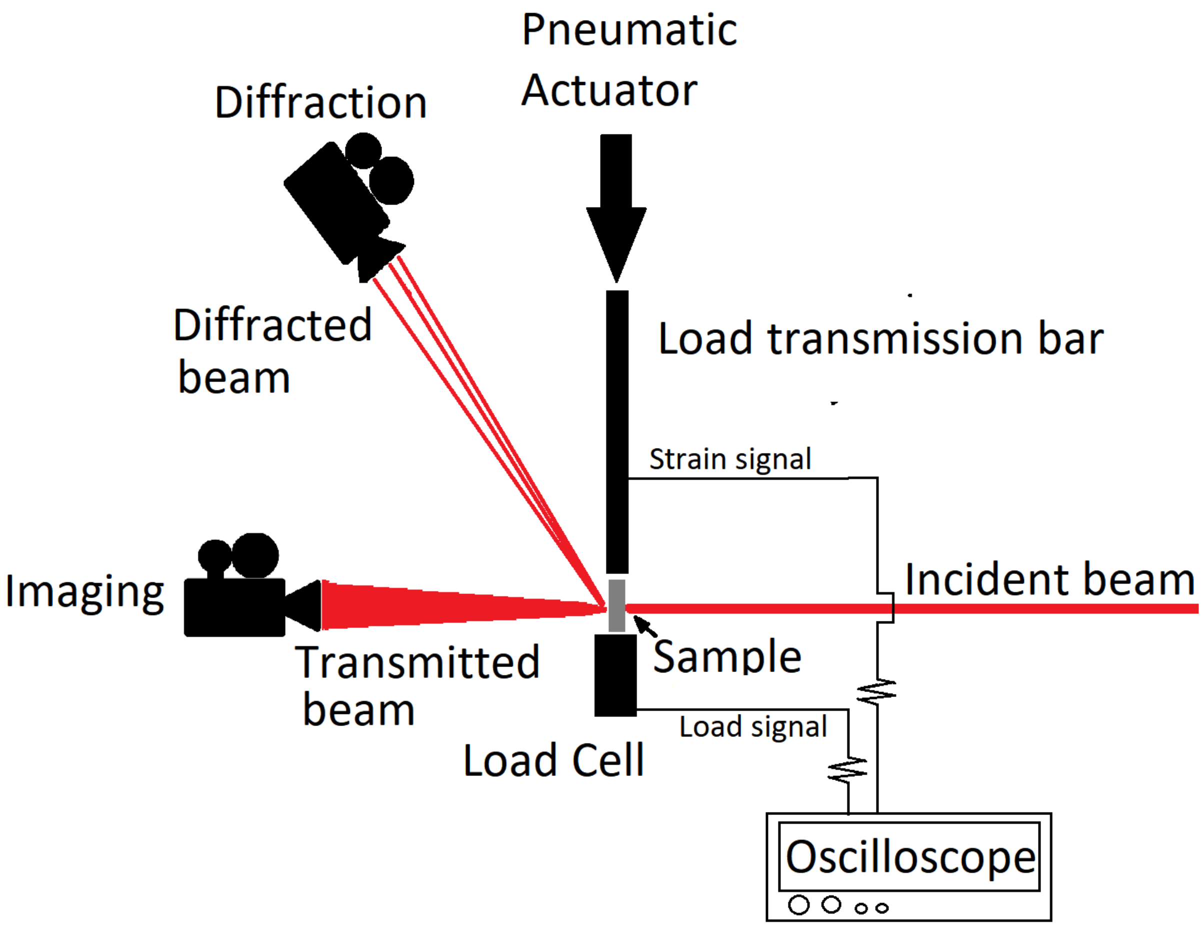


Peak-aged is 900 s at 423K

Strain Rate Effect in TWIP Ti-15Mo

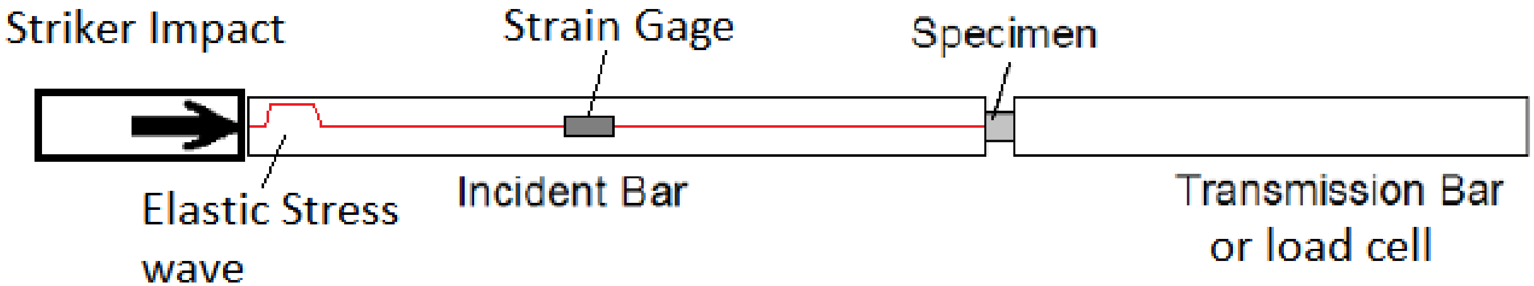


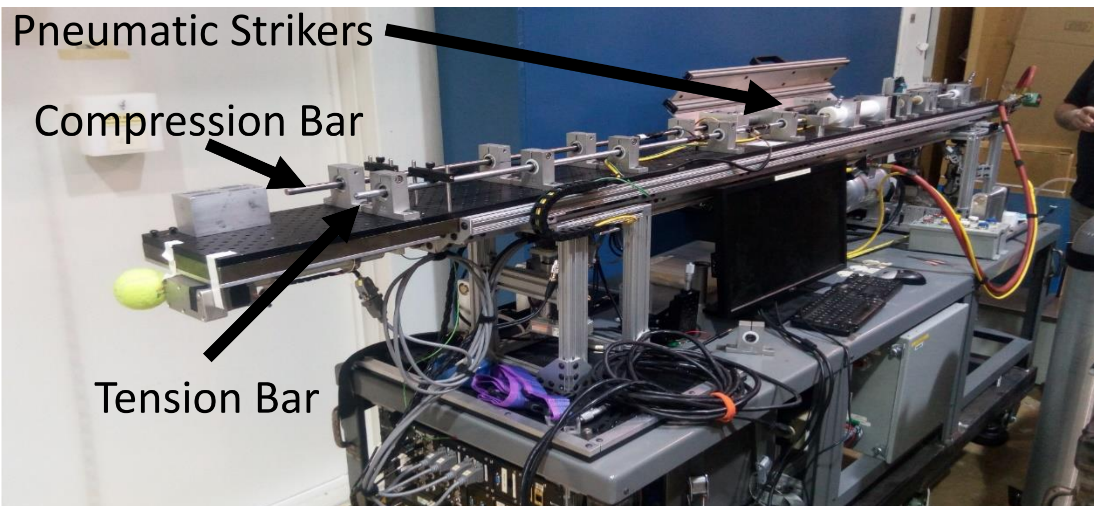
APS High-Rate In-situ Kolsky Bar



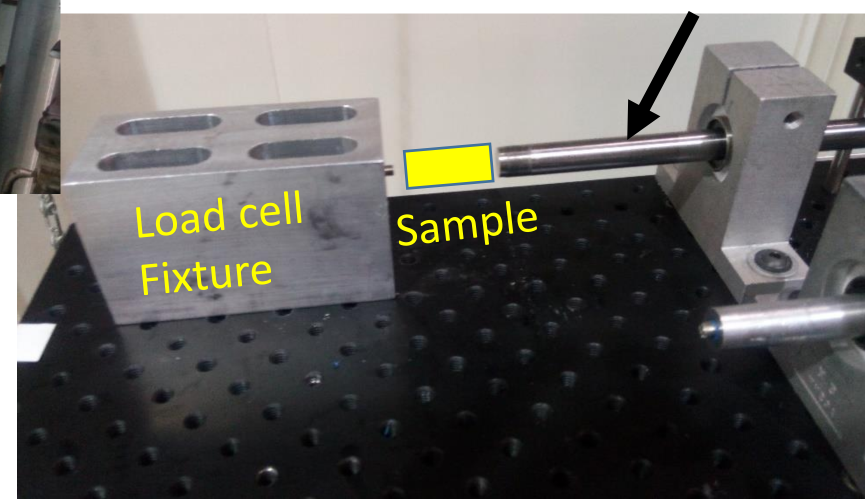
- Strain rates from 10^2 to 10^3/s in tension and compression
- Simultaneous measurement of diffraction, imaging and stress-strain data
- Time resolved data at high sampling rate, $2x10^{-8}$ s for mechanical data and $2x10^{-5}$ s for diffraction data

APS Kolsky Bar Setup

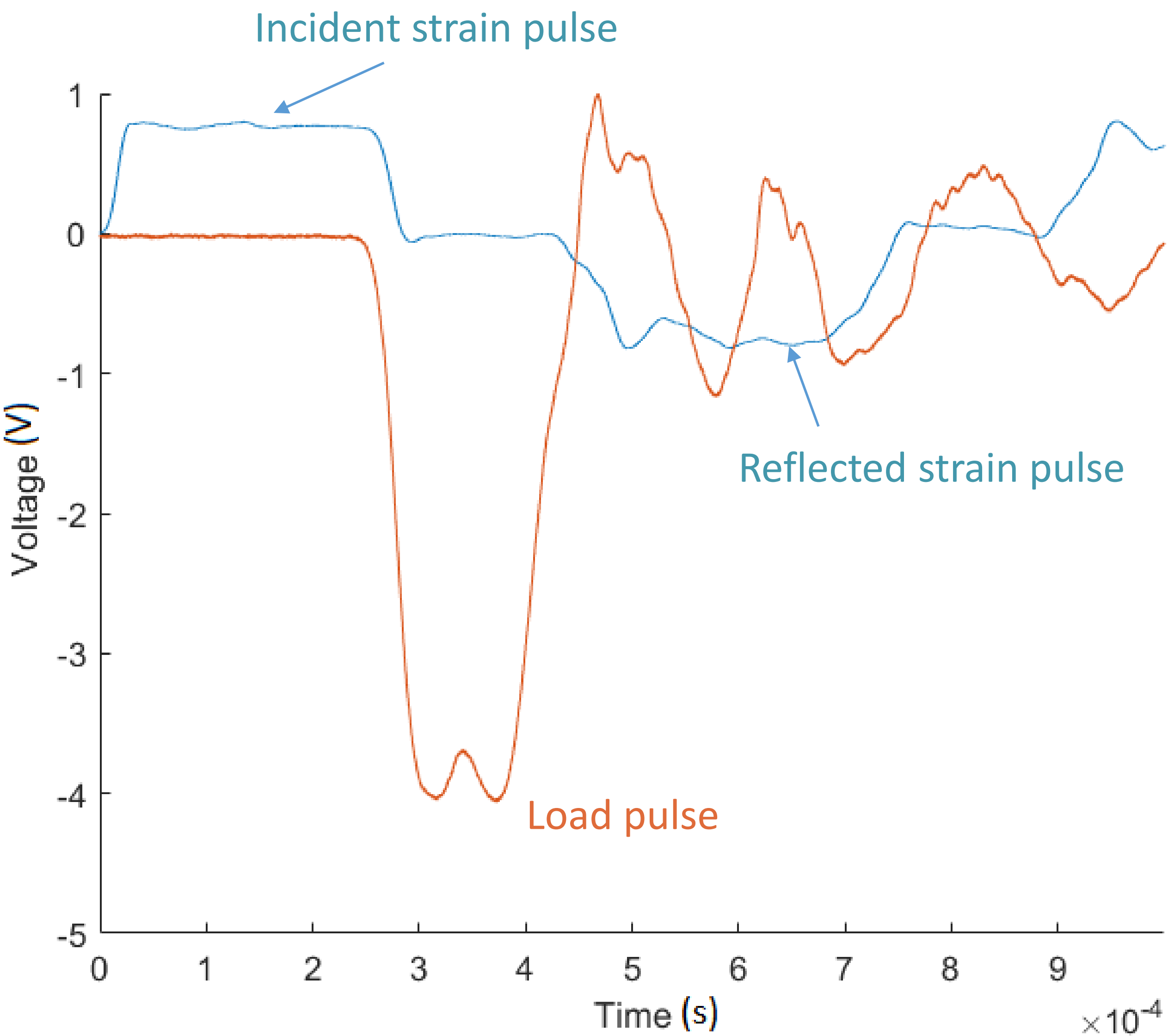




Load Transmission Bar



Kolsky Bar Raw Data



Post-Processing: Mechanical

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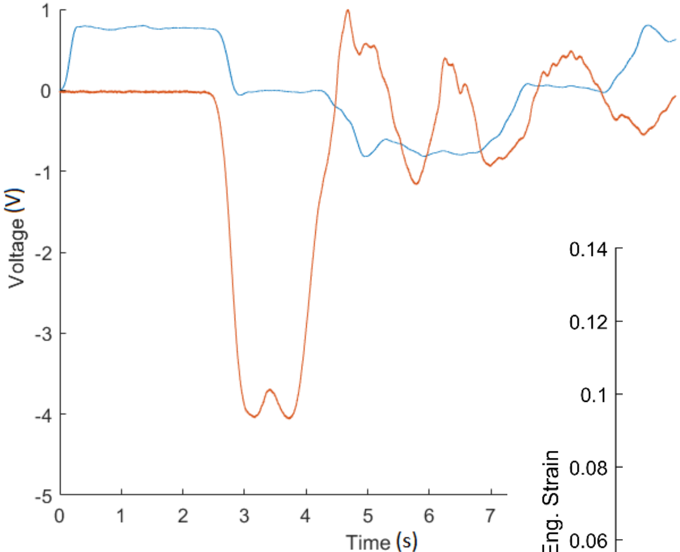
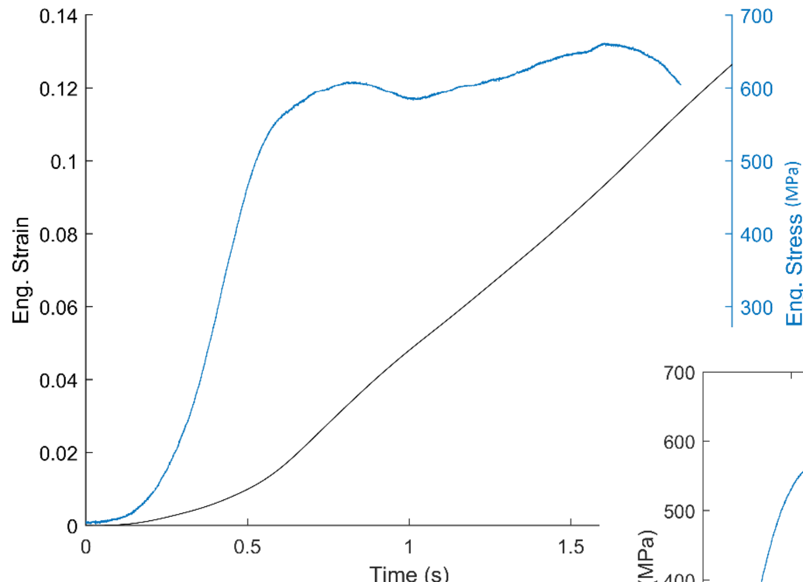
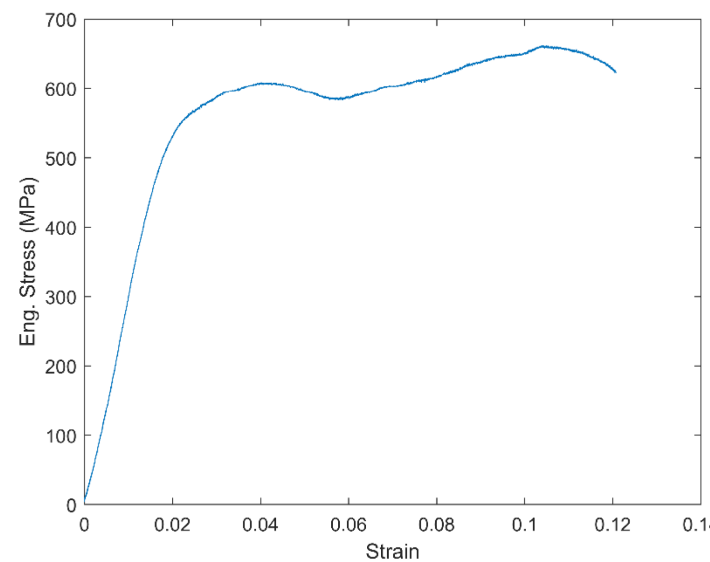


Raw data



Time-resolved strain and stress

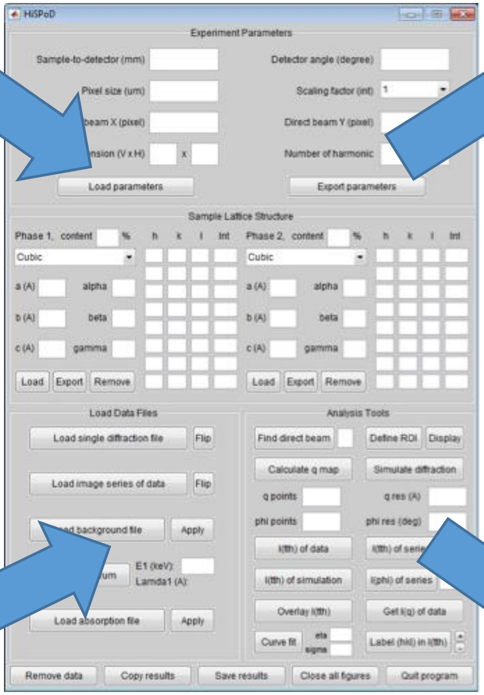
Stress-strain curve

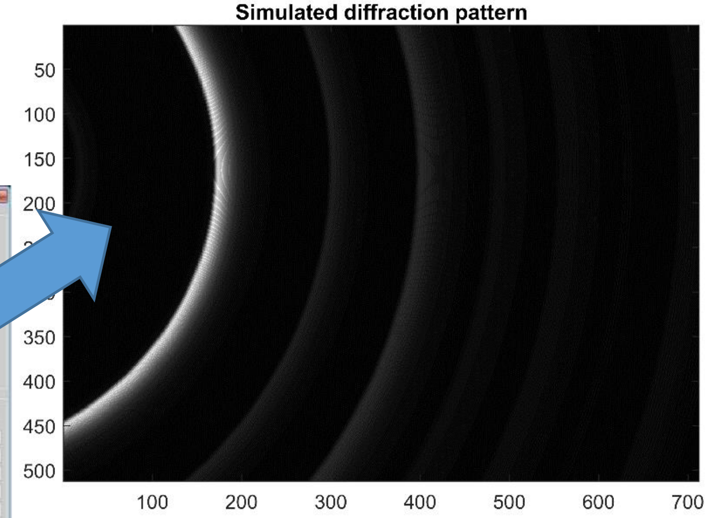


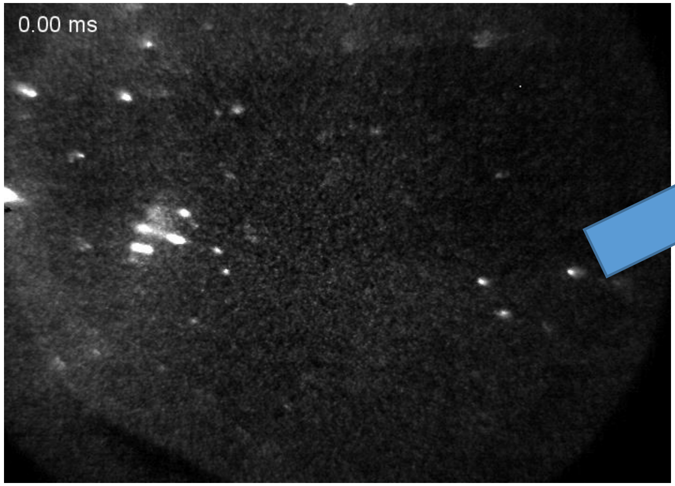
Post-Processing: Diffraction

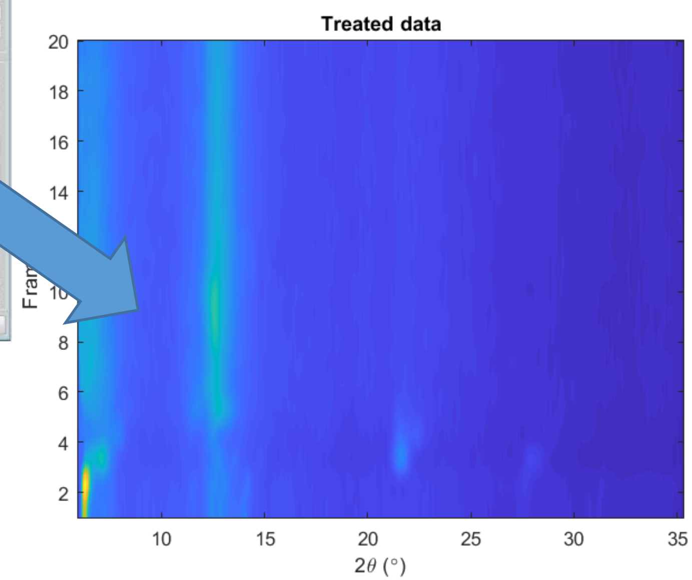
Lattice parameter
Space group
Structure factor
Volume fraction
Beam position
Energy spectrum

HiSPoD



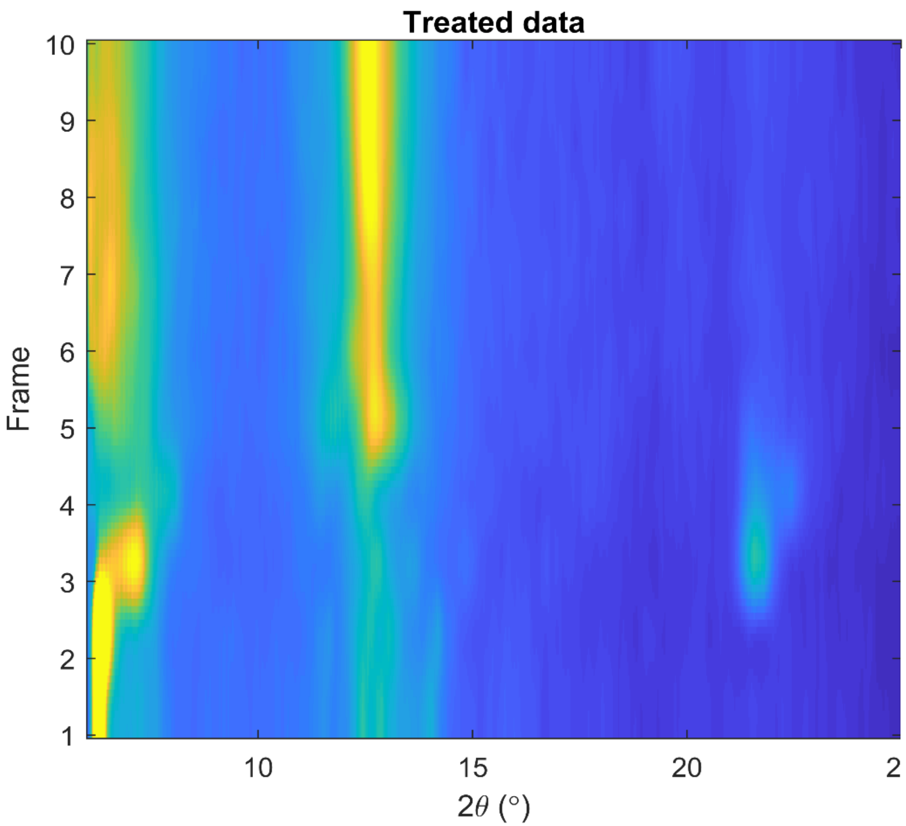


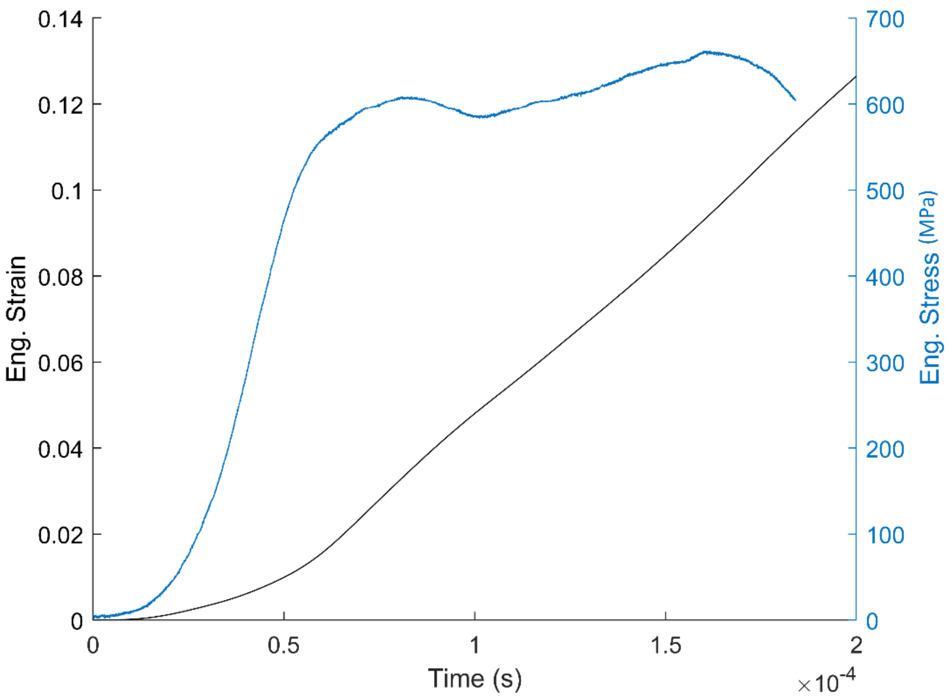




Example from a Ti-1023 Sample

Exact synching remains to be done

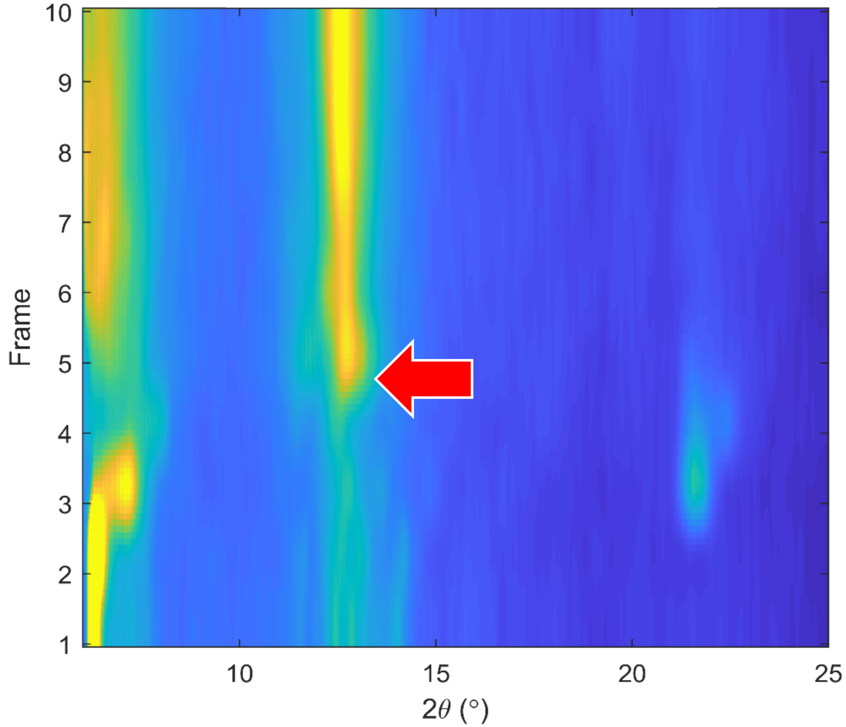




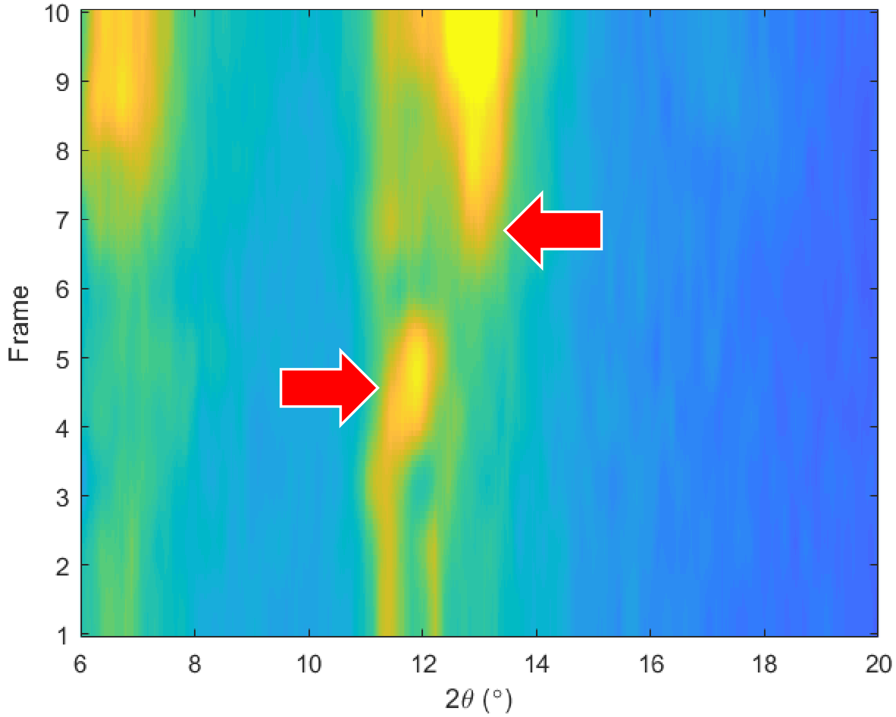
Each frame corresponds to 20 µs interval

Interpreting Diffraction Data

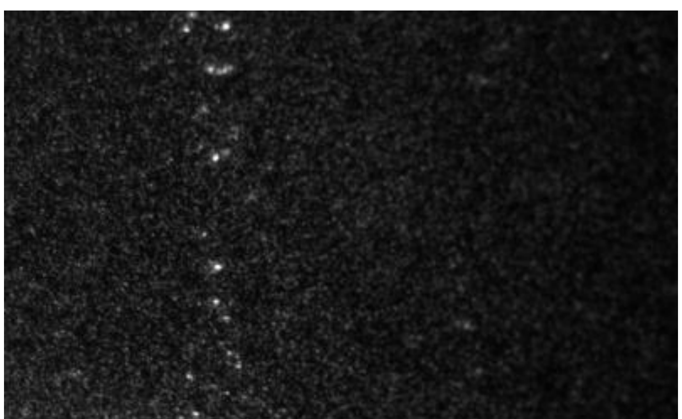
Ti-1023 sample F45



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

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