

Project #33: In-situ Studies of Strain Rate Effects on Phase Transformations and Microstructural Evolution in β -Titanium and Multi-Principle Element Alloys

**Spring 2018 Semi-Annual Meeting
Colorado School of Mines, Golden, CO
April 11-12, 2018**

Student: Benjamin Ellyson (Mines)

Faculty: Amy Clarke (Mines)

Industrial Mentor(s): TBD

Other Participants: Yaofeng Guo (Mines)



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

- 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

- Heat treatment performed to obtain β phase microstructures that exhibit TRIP/TWIP
- Compressive testing of solution treated samples partially completed
- Investigation of low-temperature, short soak time treatments is under way
- Initial thermo-mechanical testing completed in the Gleeble

Metrics

Description	% Complete	Status
1. β solution treatments	95%	●
2. Literature review	50%	●
3. High-throughput quasi-static compression testing to β solution heat treatment	90%	●
4. Optical microstructural characterization of pre and post compression conditions	80%	●
5. EBSD/TEM microstructural characterization of pre and post compression conditions	10%	●



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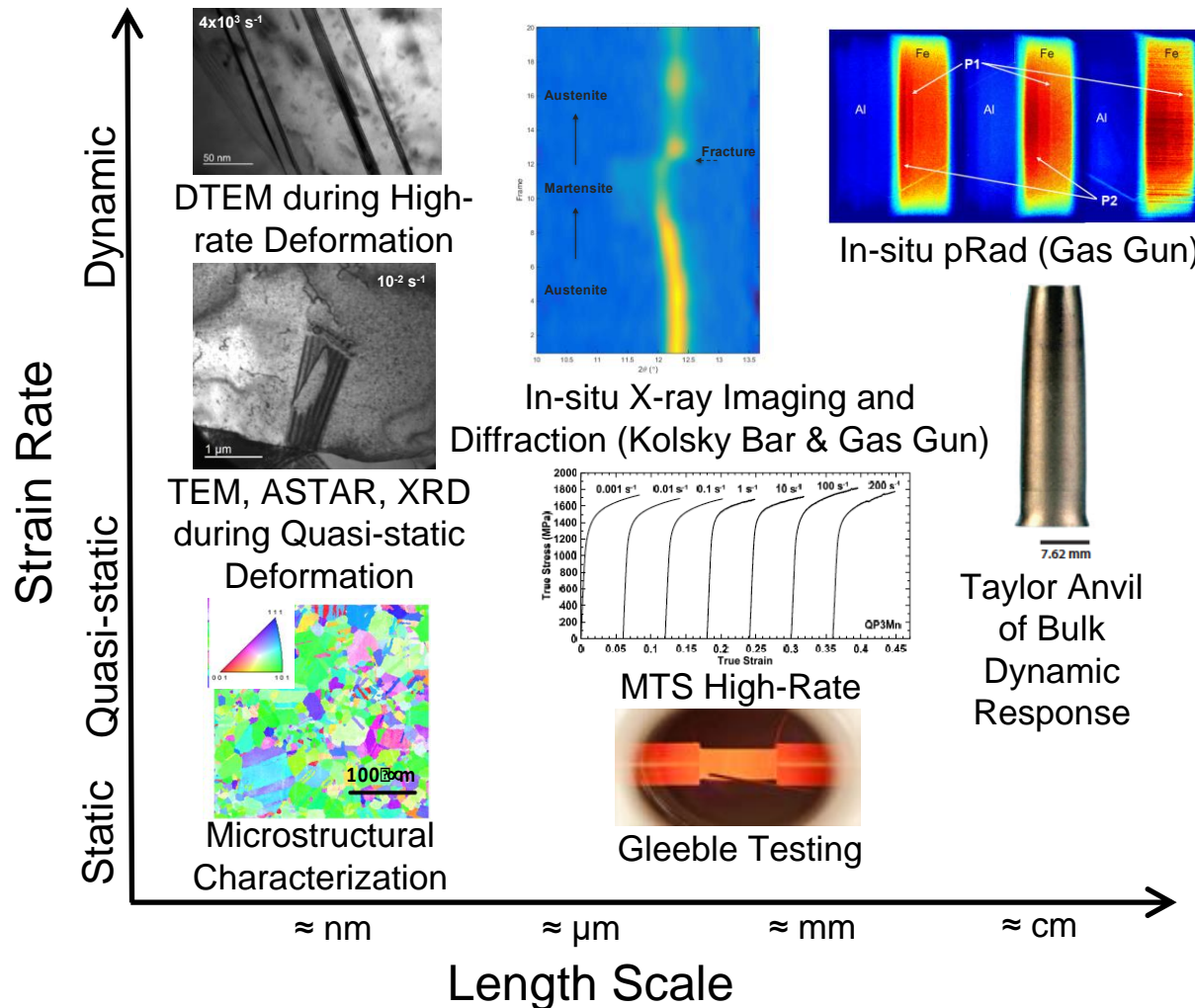
Industrial Relevance: Development of Blast Resistant Materials for the Navy

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

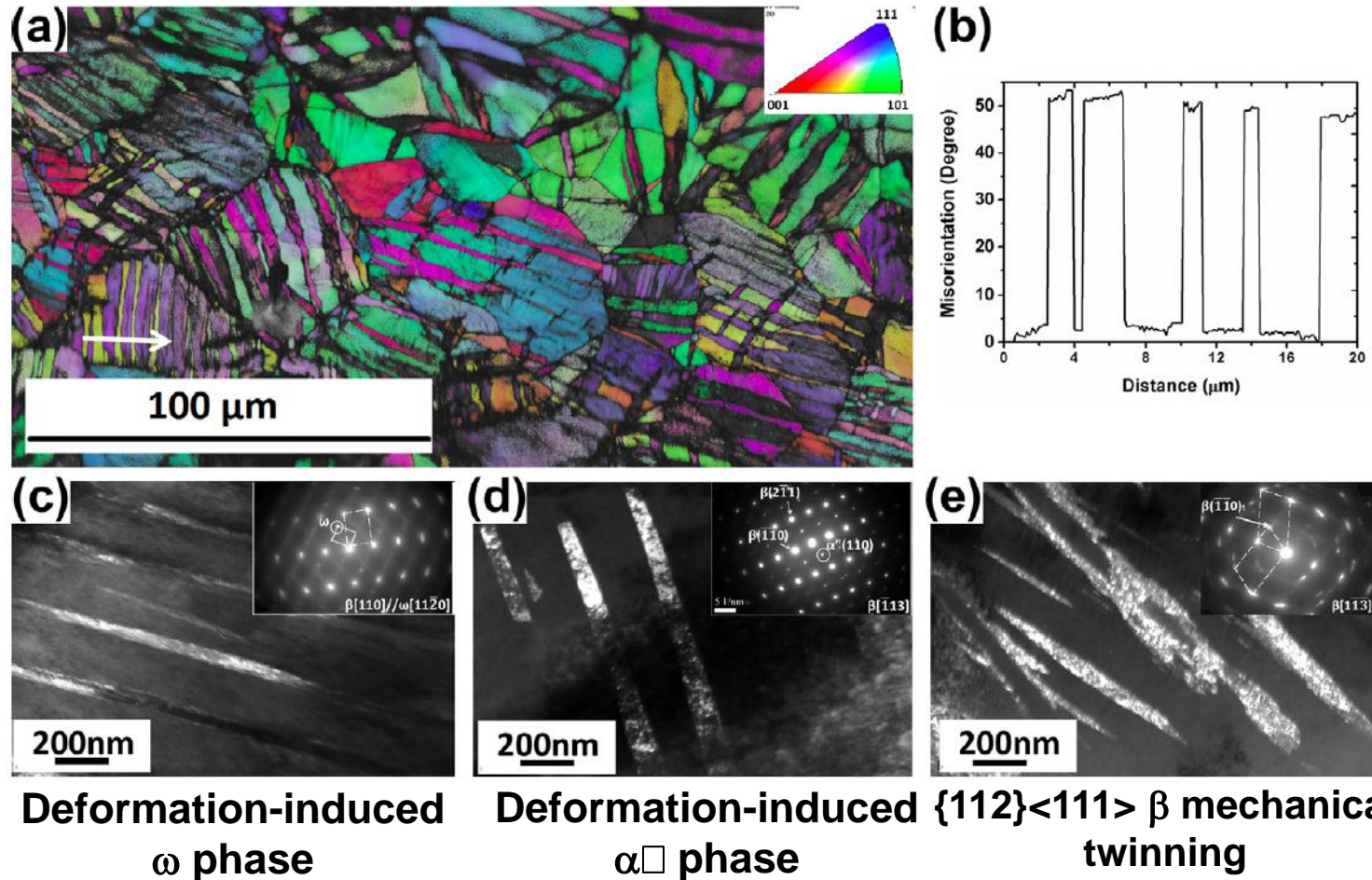


<https://www.onr.navy.mil/Science-Technology/Departments/Code-33>

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



Ti-25Nb-3Zr-3Mo-2Sn (wt.%) Alloy Microstructure After Deformation at 10^{-3} s^{-1} and 0.18 True Strain

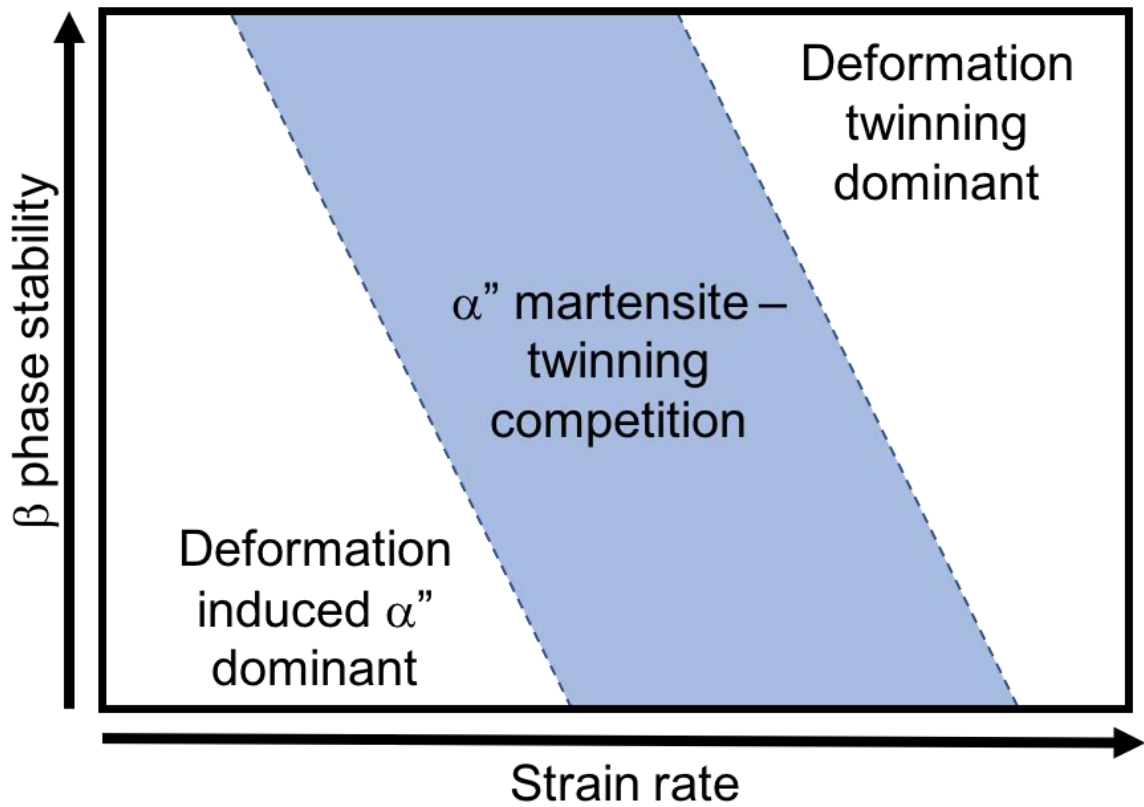


H. Zhan, et al. 107 Scripta Materialia (2015): 34-37

6 The Effect of Strain Rate (10^{-3} , 10^{-1} , 10^1 , 10^2 s $^{-1}$) on Deformation Mechanisms during Compression of a Ti-10V-3Fe-3Al (wt.%) Alloy

• As strain rate increases:

- Stress-induced α'' martensite (dominant) + $\{332\}\langle 113\rangle$ β twinning + stress-induced ω phase + slip
- Stress-induced α'' martensite + $\{332\}\langle 113\rangle$ β twinning + stress-induced ω phase + slip
- Stress-induced α'' martensite + $\{332\}\langle 113\rangle$ β twinning (dominant) + stress-induced ω phase + slip



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

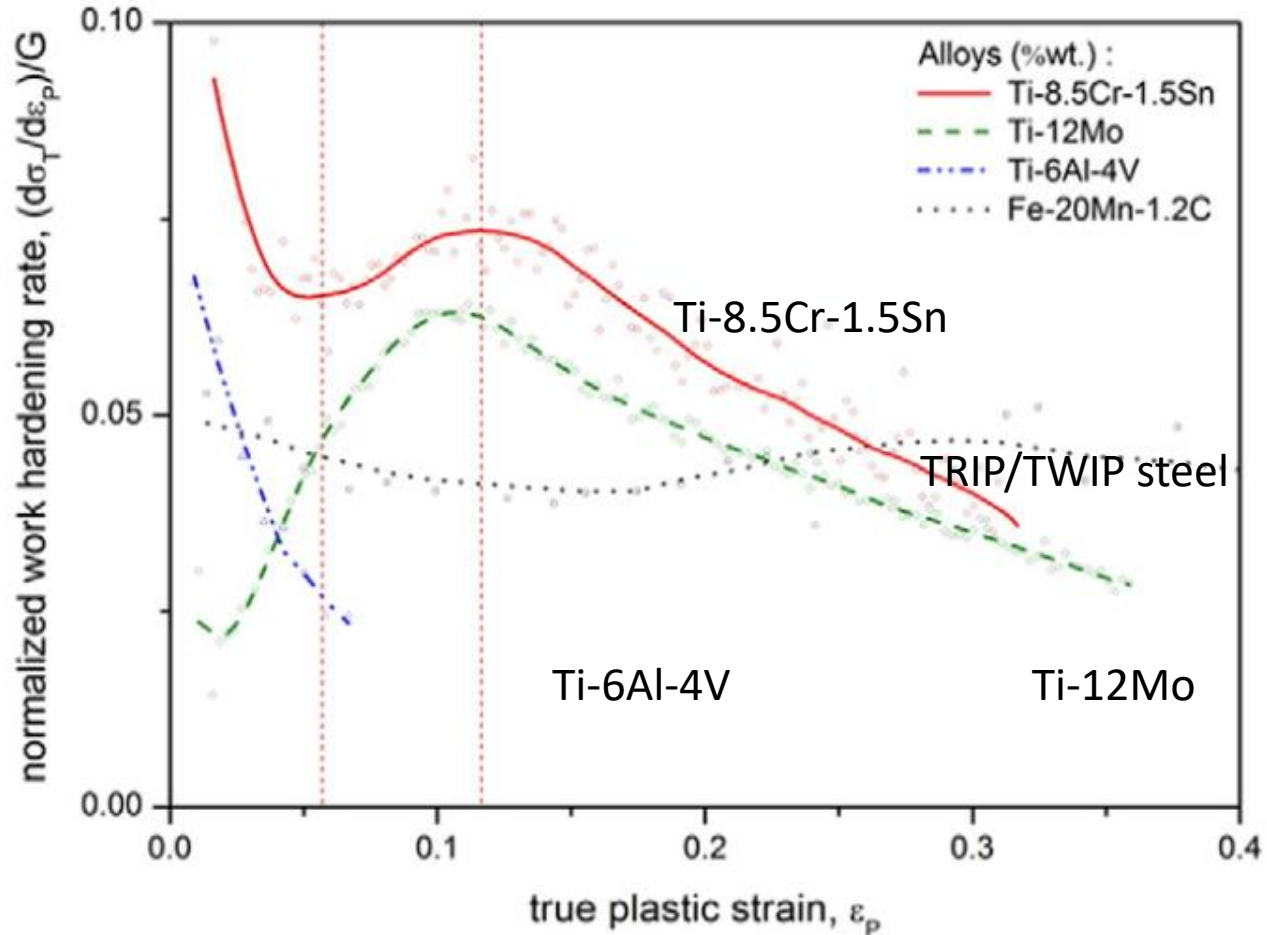


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7 Work Hardening and Evidence of TRIP/TWIP



Brozek, C., et al. Scripta Materialia 114 (2016): 60-64



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Ti-1023 Heat Treatments to Obtain Fully β Microstructures

To promote TRIP/TWIP:

- Single phase β microstructure
- Fully homogenized
- Small grain size promotes TRIP
- Large grain size promotes TWIP

Li, C., et al. *Materials Science and Engineering: A* 528.18 (2011): 5854-5860
Bhattacharjee, A., et al. *Scripta materialia* 53.2 (2005): 195-200



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Solution Heat Treatment Matrix

- Hold temperature and quench rate effect on martensite fraction
- Response surface for martensite phase fraction dependence
- Water and helium to vary quench rate

Temp (°C)	Time (h)	Quench medium
900	2	Water or He
1000	2	Water or He
1100	2	Water or He
1200	2	Water or He

Sample Nomenclature

Sample denomination will be as follows:

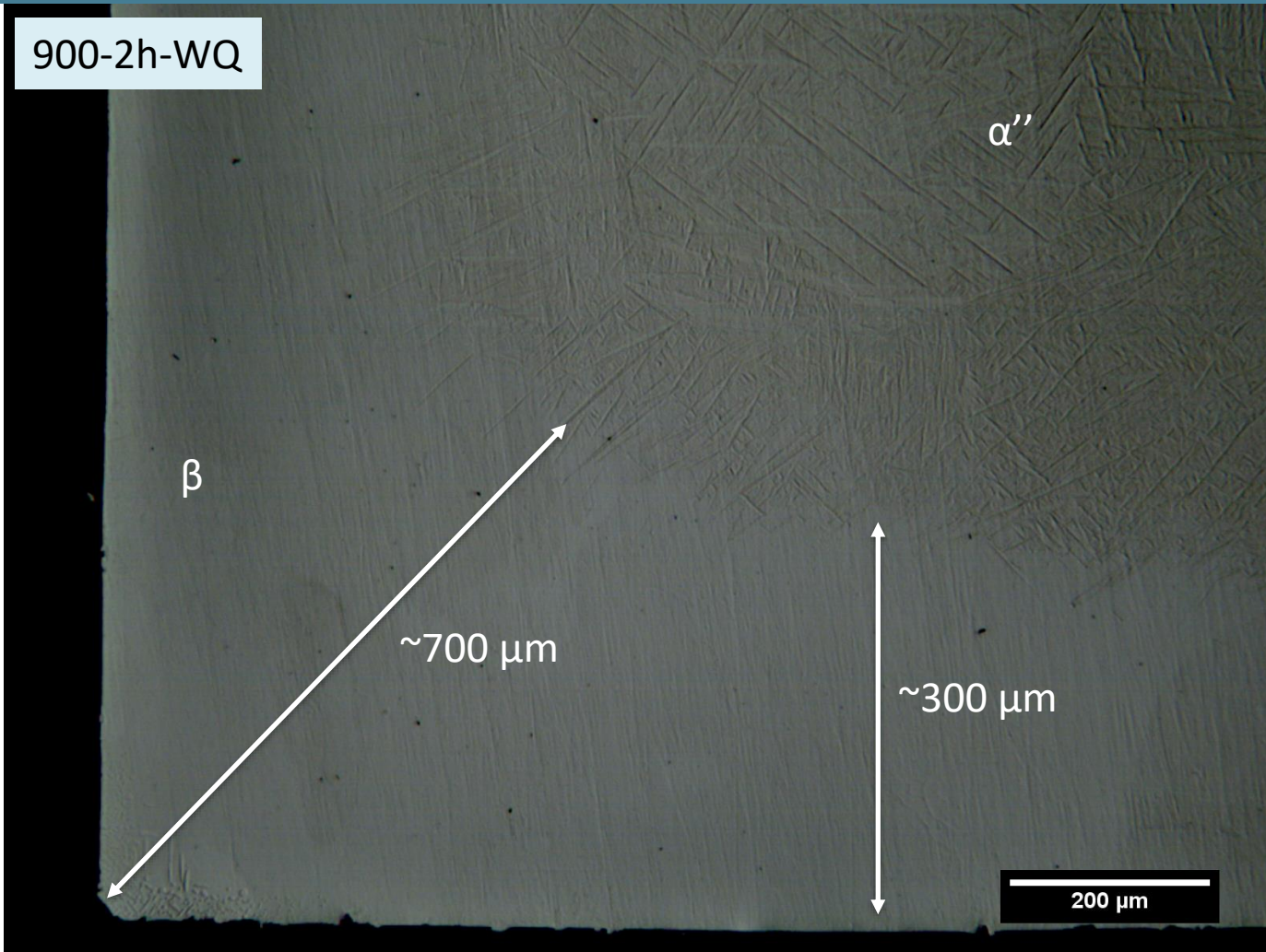
Temperature-time-quench-condition

900-2h-WQ-pre

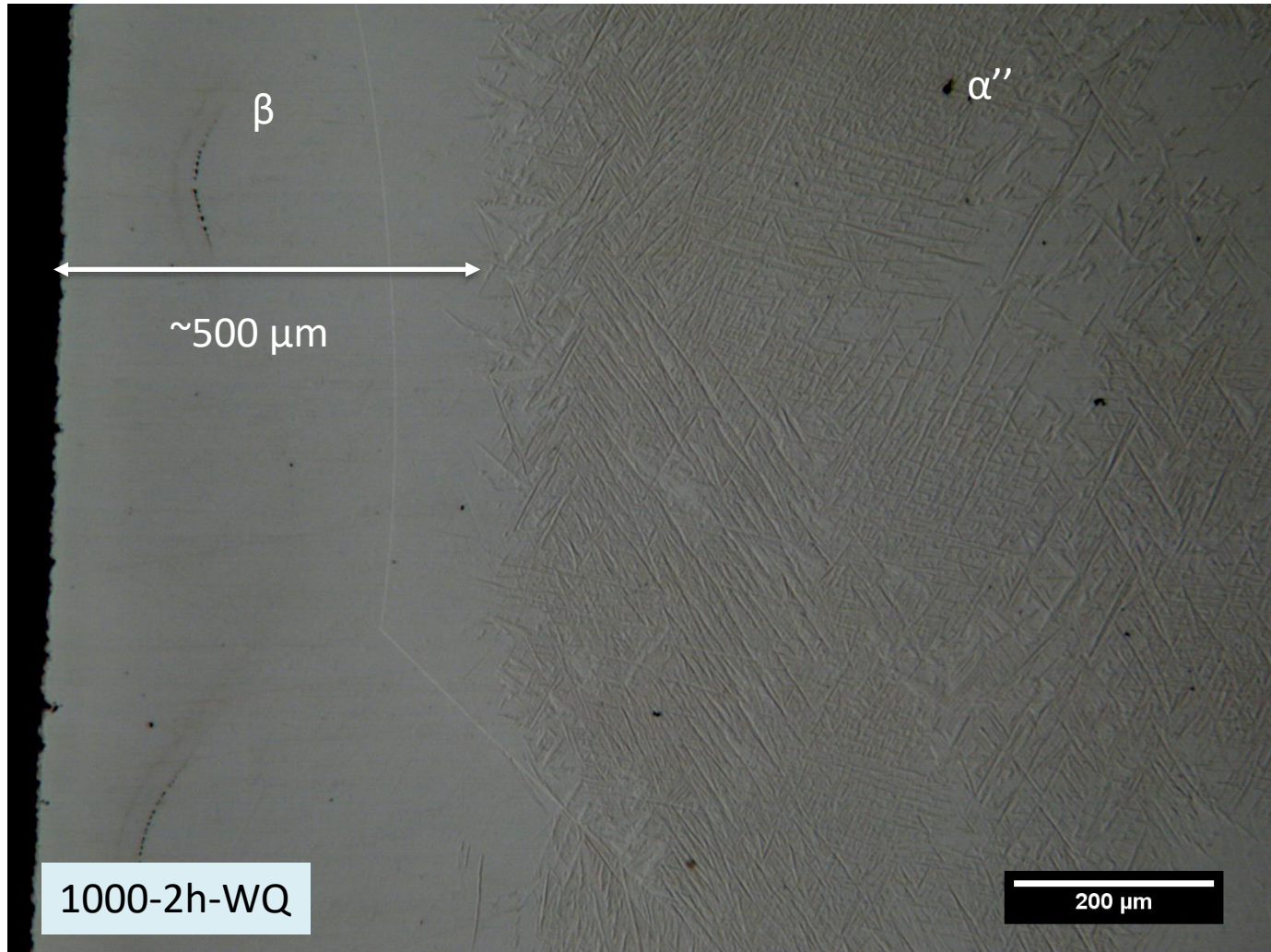
900-2h-WQ-post

900-2h-WQ-10⁻²

Preliminary Water Quench Results



Representative of 1000°C and 1100°C



Challenges in Obtaining Fully β Microstructures in Ti-1023

- Obtaining a fully-retained β structure in Ti-1023 requires severe quench rates and thin sections

Bhattacharjee, A., et al. *Scripta materialia* 53.2 (2005): 195-200

Duerig, T. W., et al. *Acta Metallurgica* 30.12 (1982): 2161-2172

Neelakantan, S., et al. *Materials Science and Technology* 25.11 (2009): 1351-1358

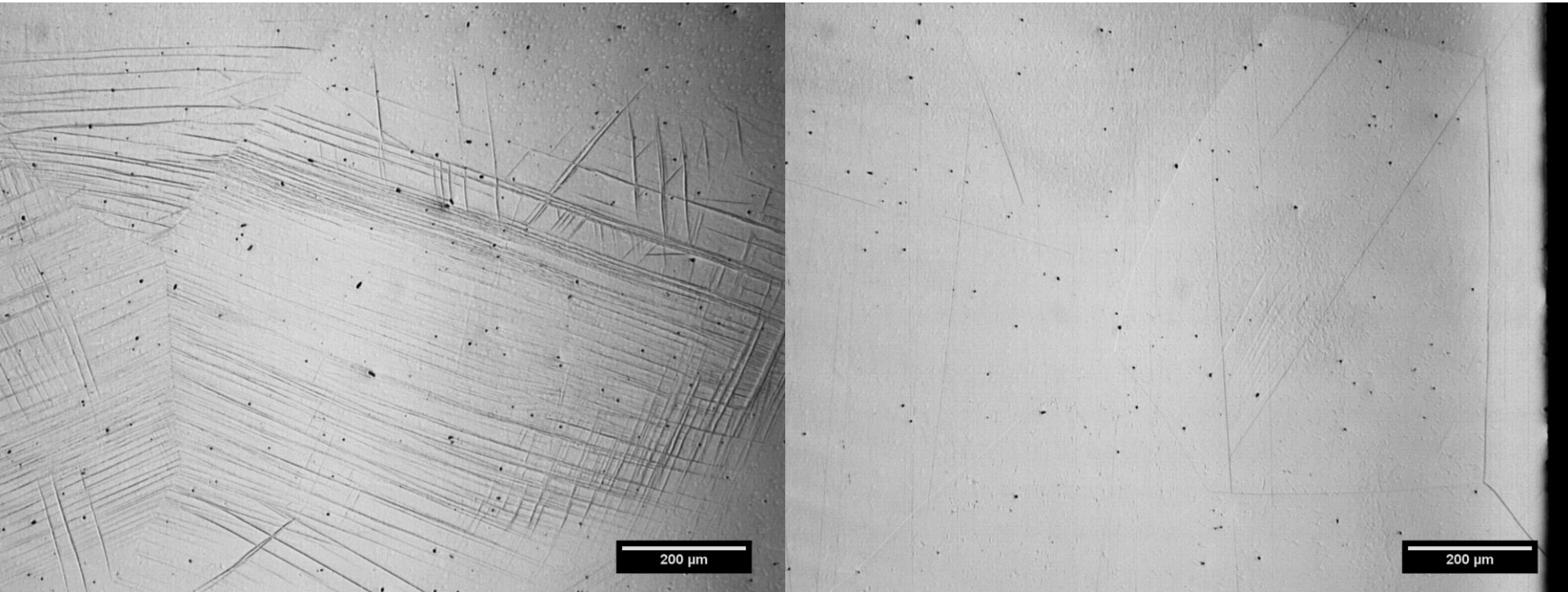


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1200-2h-WQ Exhibits Larger Grain Size and Inhomogeneous Response



Less nucleation sites for martensite

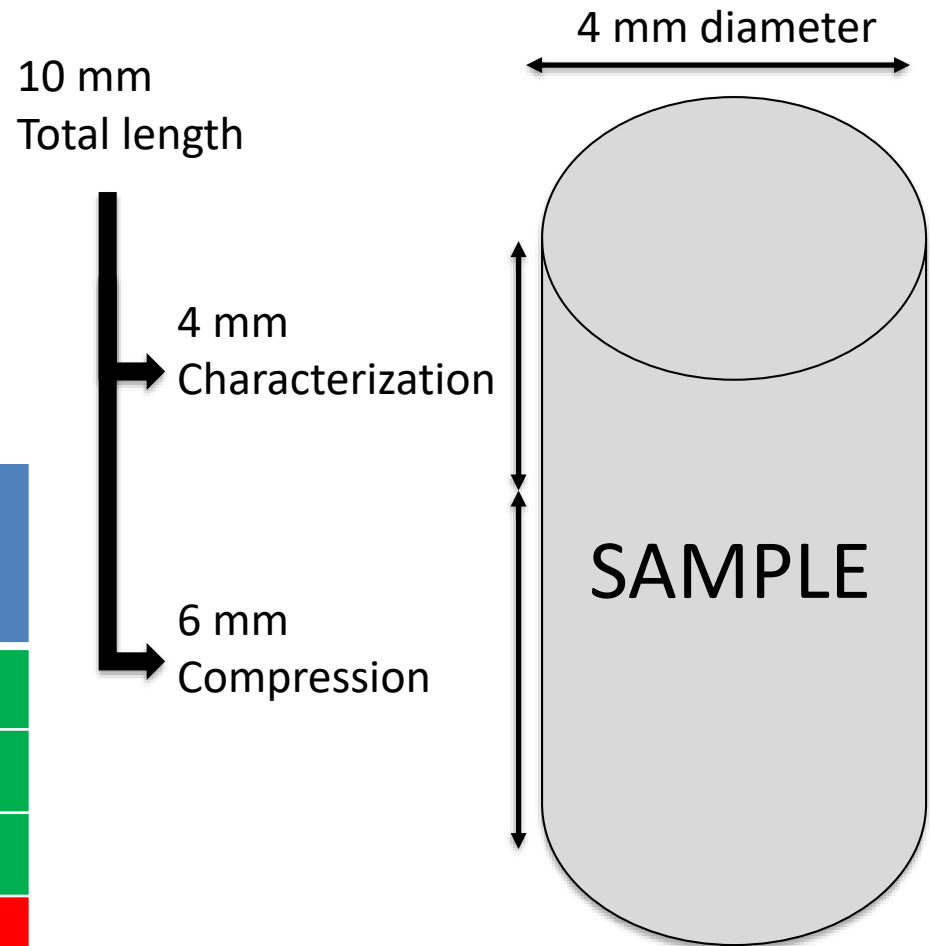
Lessons Learned

Factors controlling the ability to produce fully β microstructures:

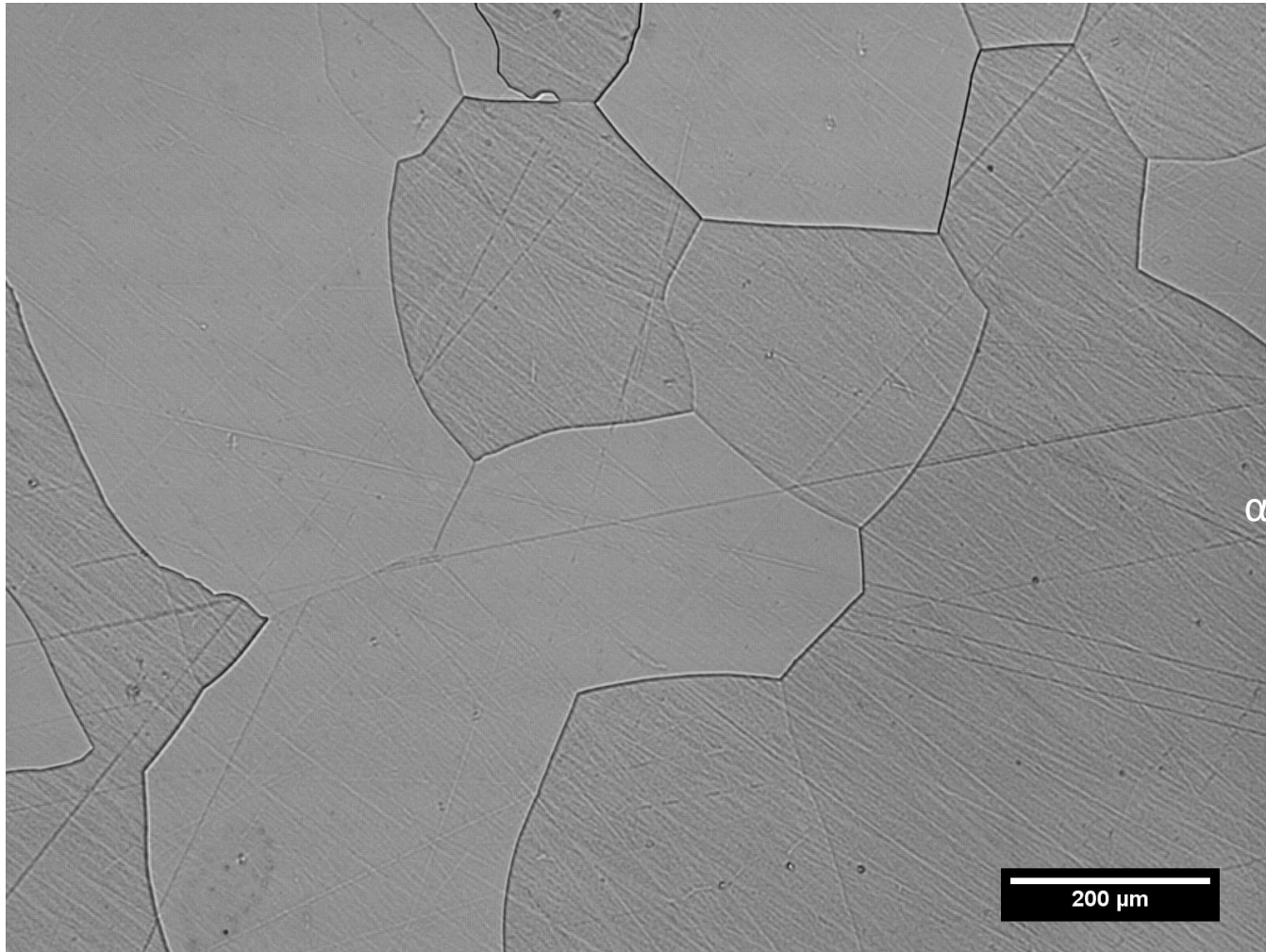
- Sample size
- Quench delay
- Quench rate
- Grain size

Sample Geometry - Compression

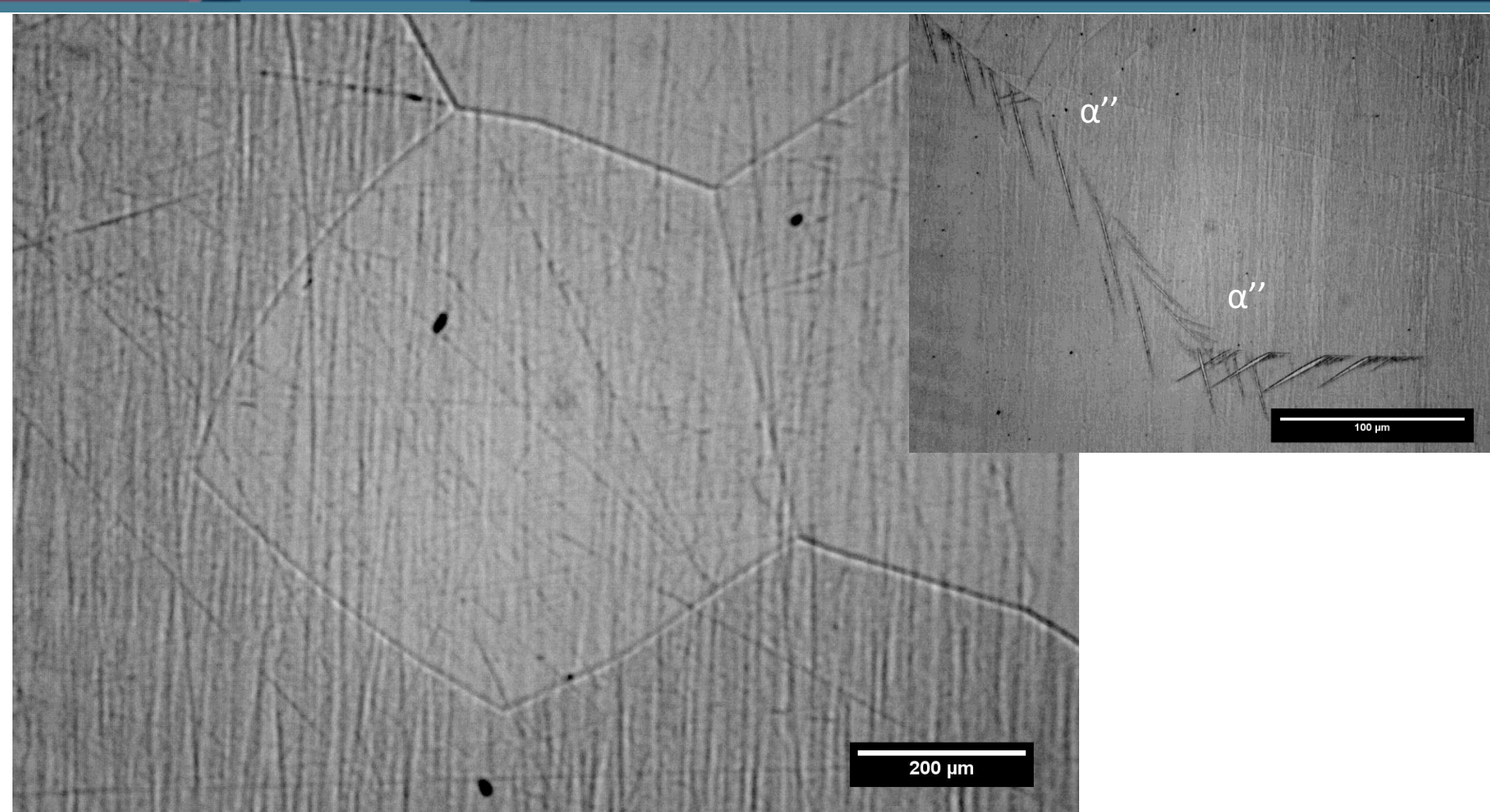
Temperature (°C)	Helium Quench	Water Quench
900		
1000		
1100		
1200		



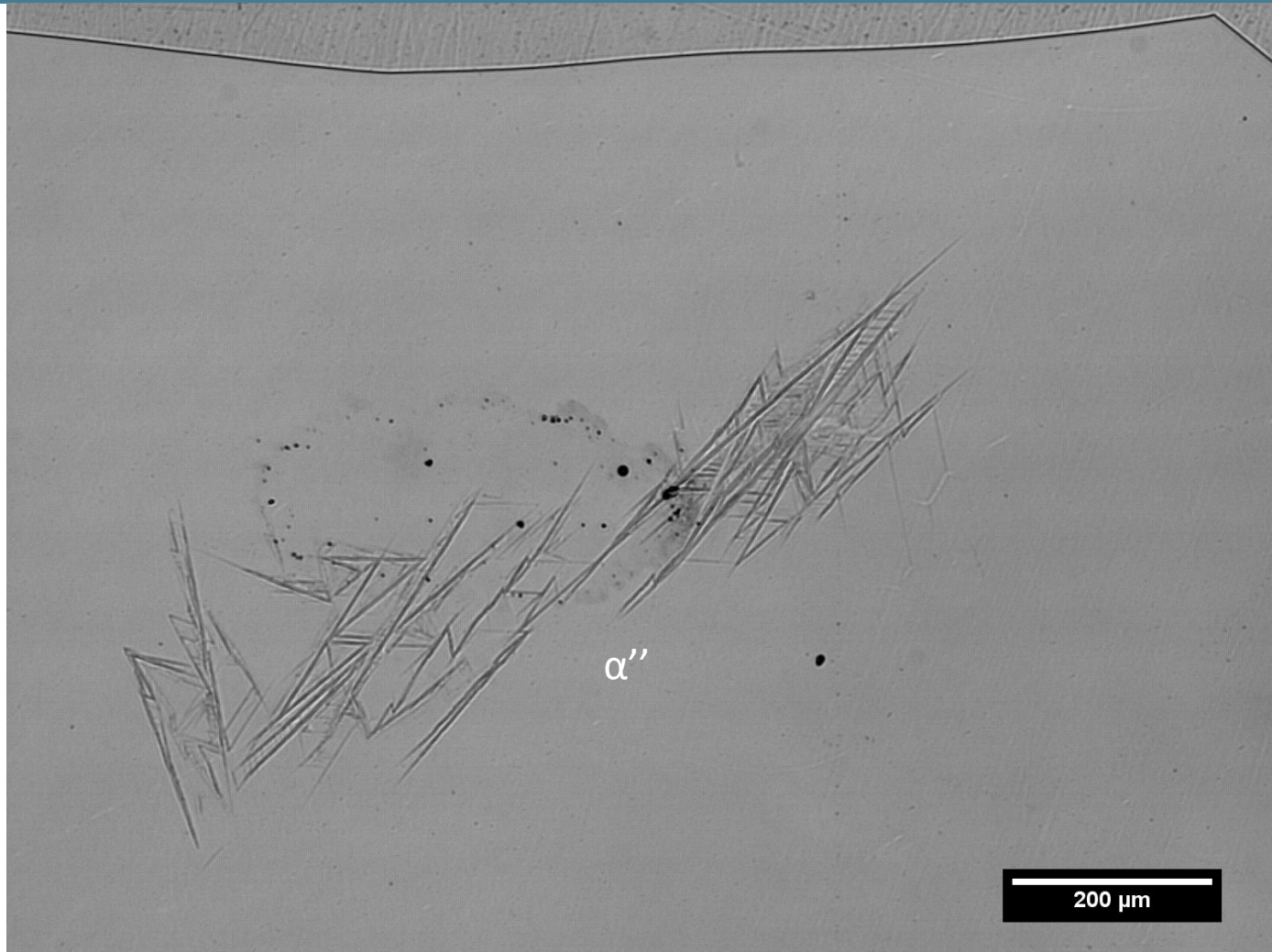
900-2h-HeQ-pre Exhibits Equiaxed β



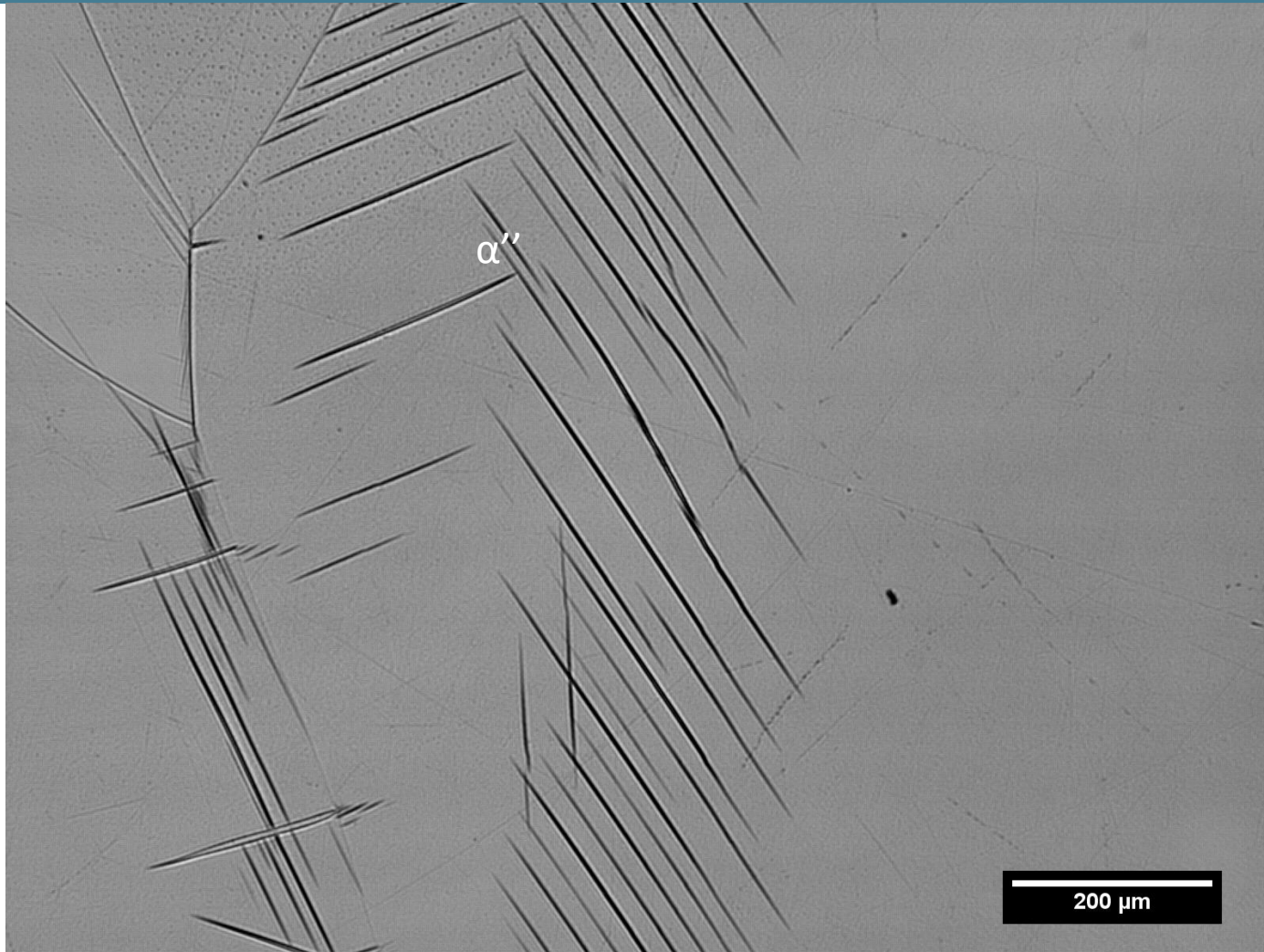
1000C-2h-WQ-pre equiaxed β



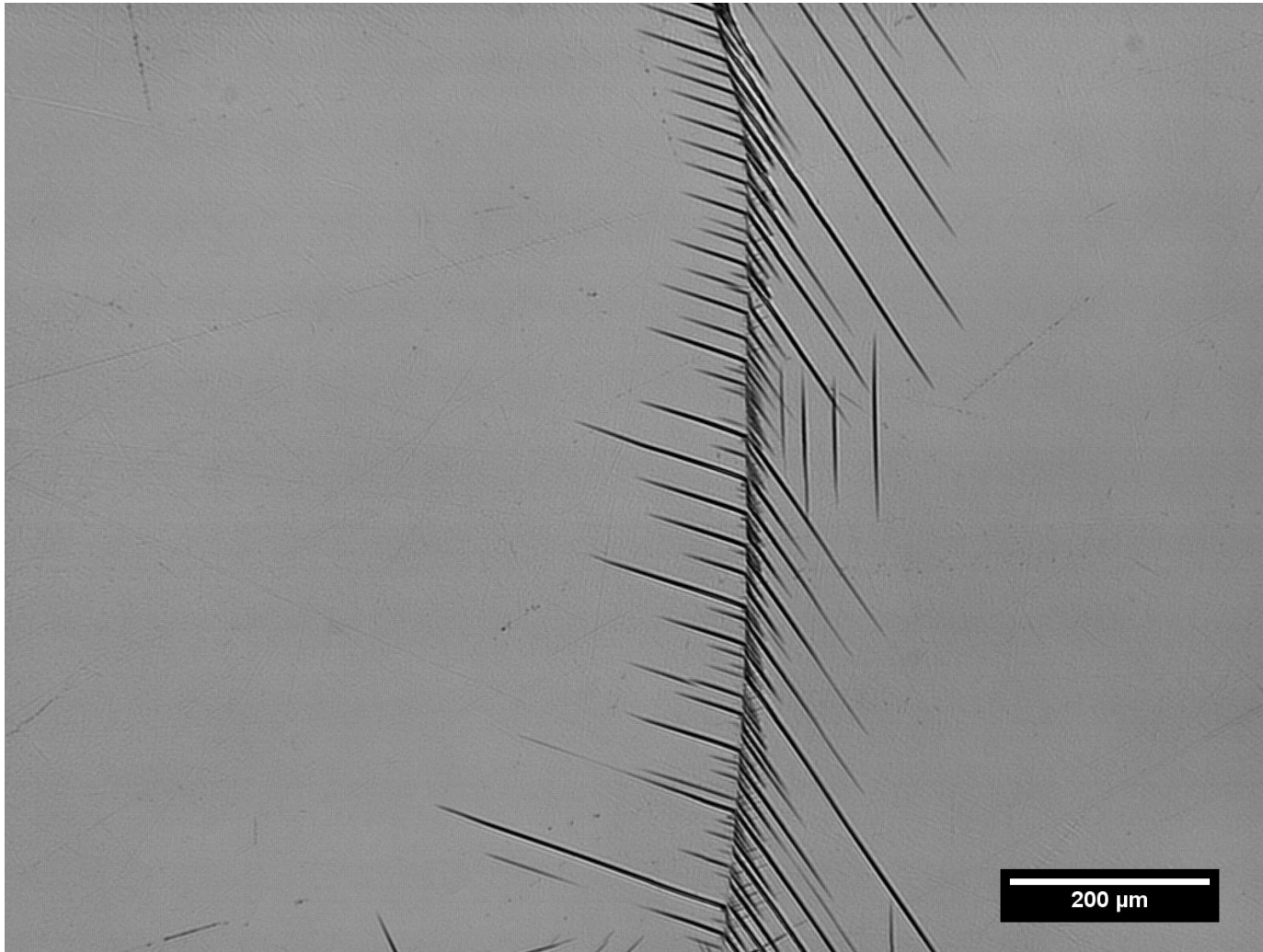
1100C-2h-HeQ-pre equiaxed β



Traces of α'' in the 1200-2h-HeQ-pre



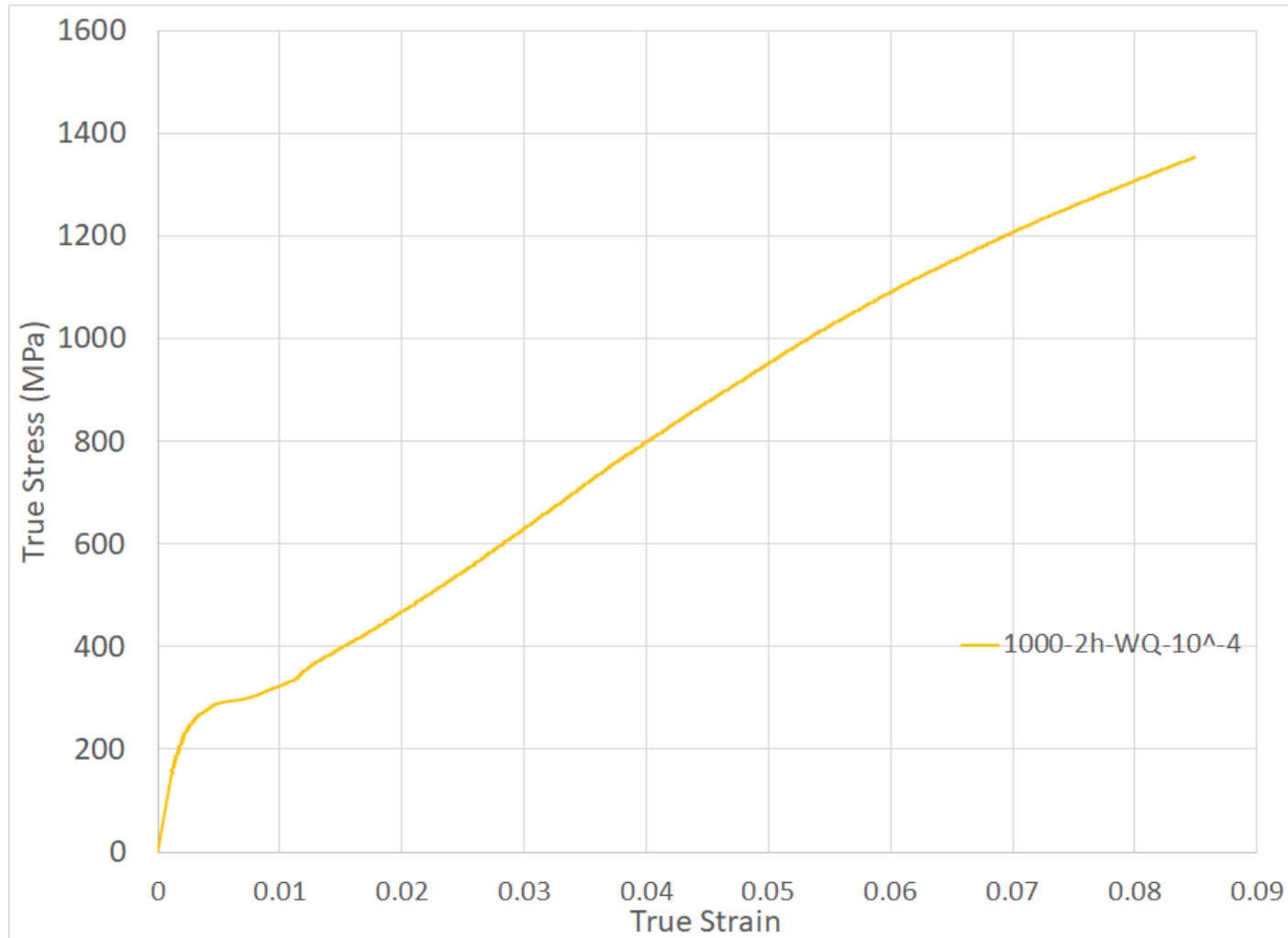
Only a Single Grain Boundary in the 1200-2h-HeQ-pre cross-section



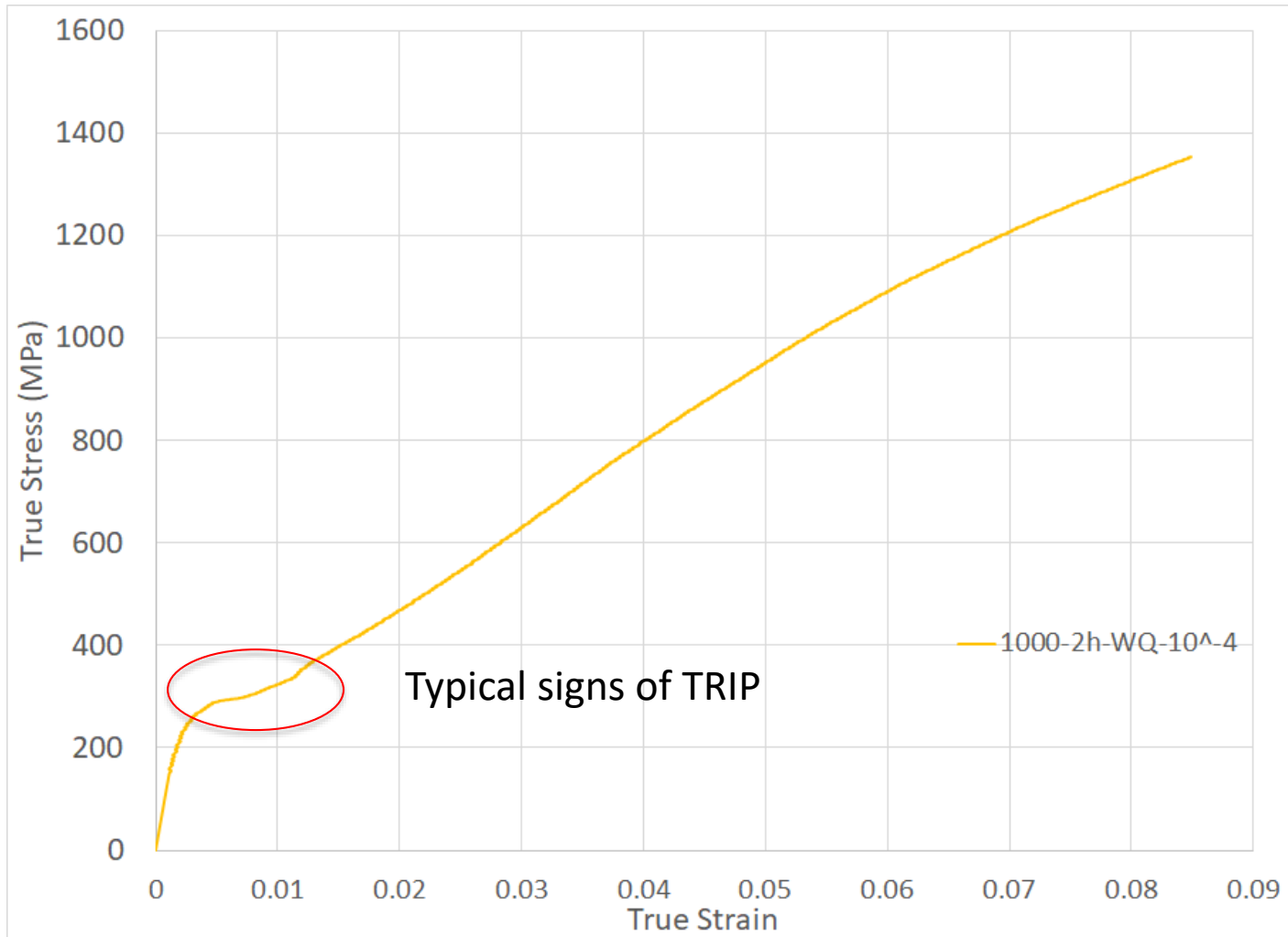
Optical Microstructural Characterization Summary

Condition	Martensite Fraction	# of Grains in $\Phi 4$ mm Cross Section
900-2h-WQ	~ 15%	~50
900-2h-HeQ	~ 2%	~ 60
1000-2h-WQ	< 1%	~ 17
1000-2h-HeQ	~ 0%	~ 12
1100-2h-HeQ	~ 2%	~ 3
1200-2h-HeQ	~ 6% (only @ GB)	~ 2

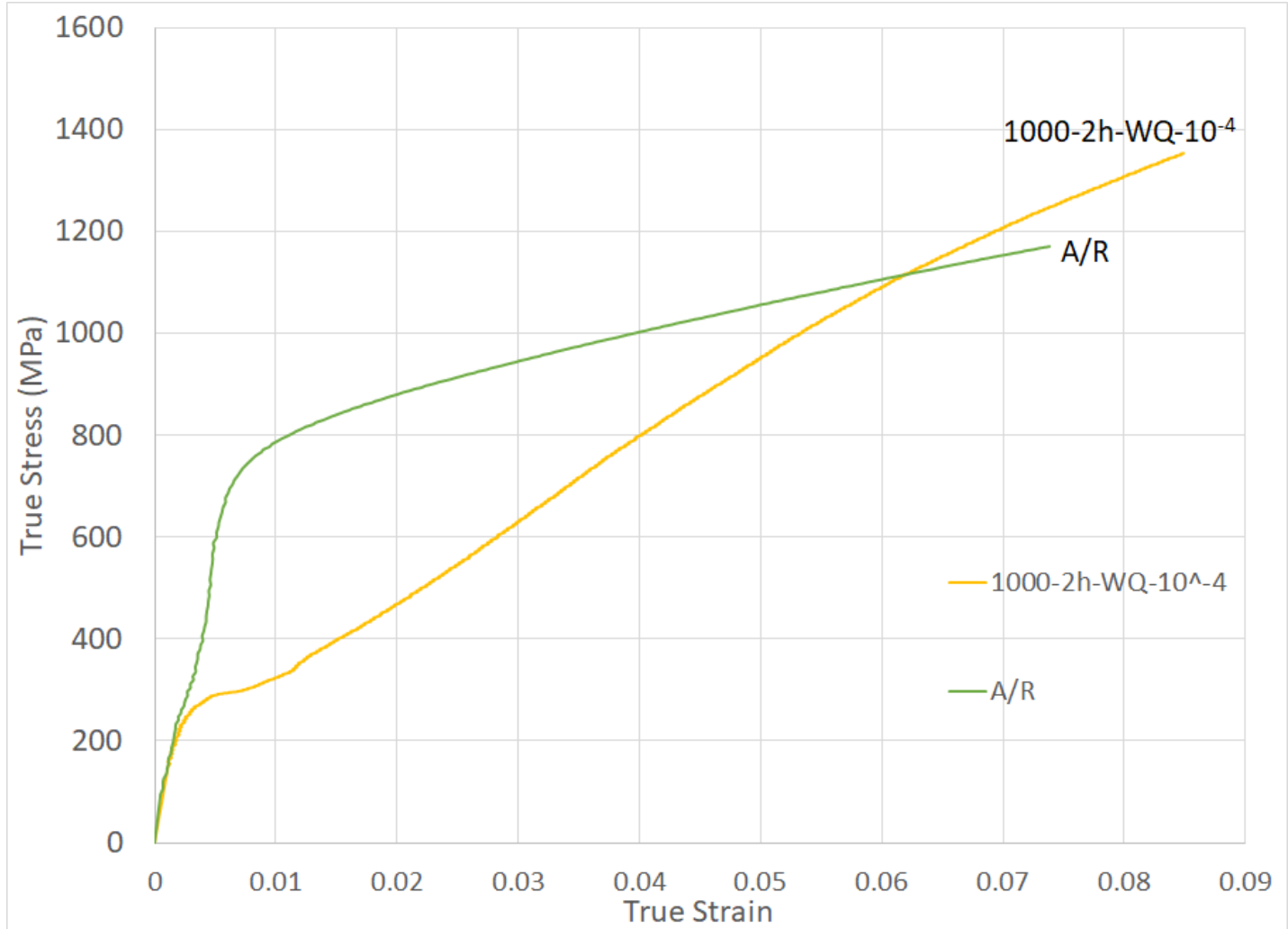
Compression of 1000-2h-WQ at 10^{-4} /s



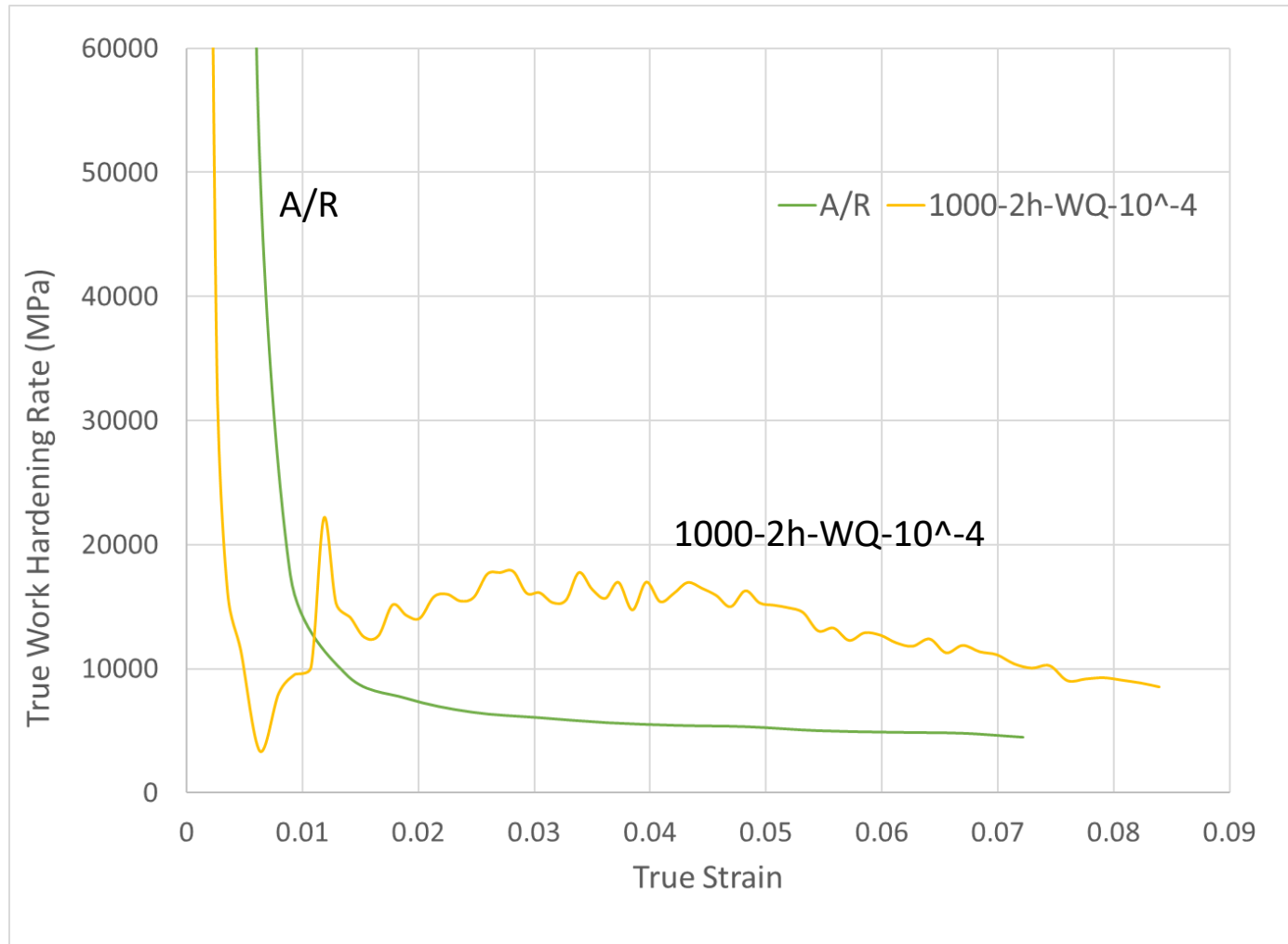
Compression of 1000-2h-WQ at 10^{-4} /s



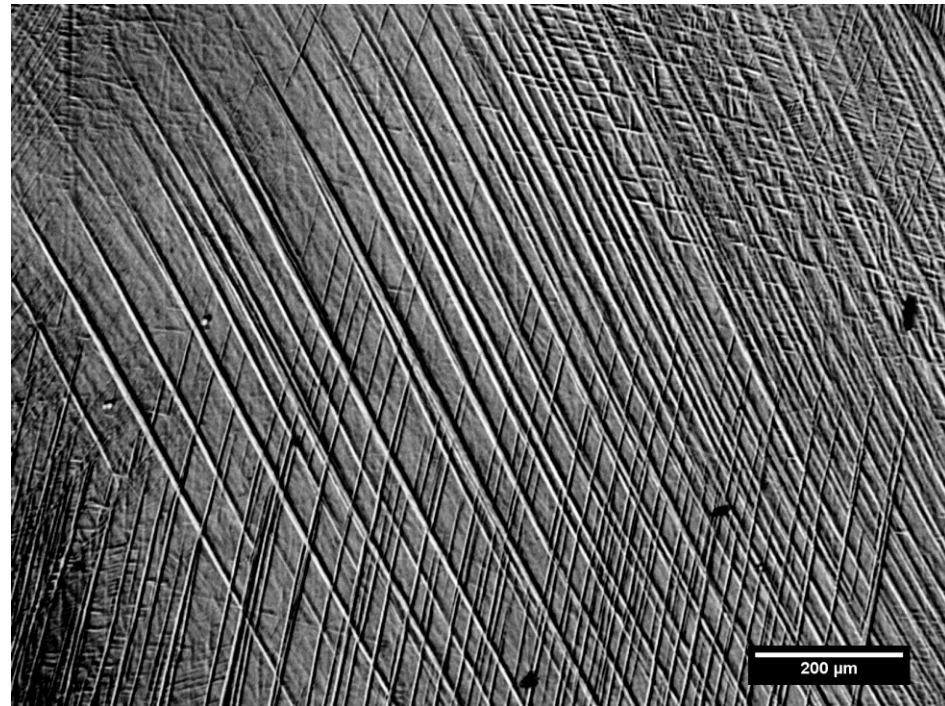
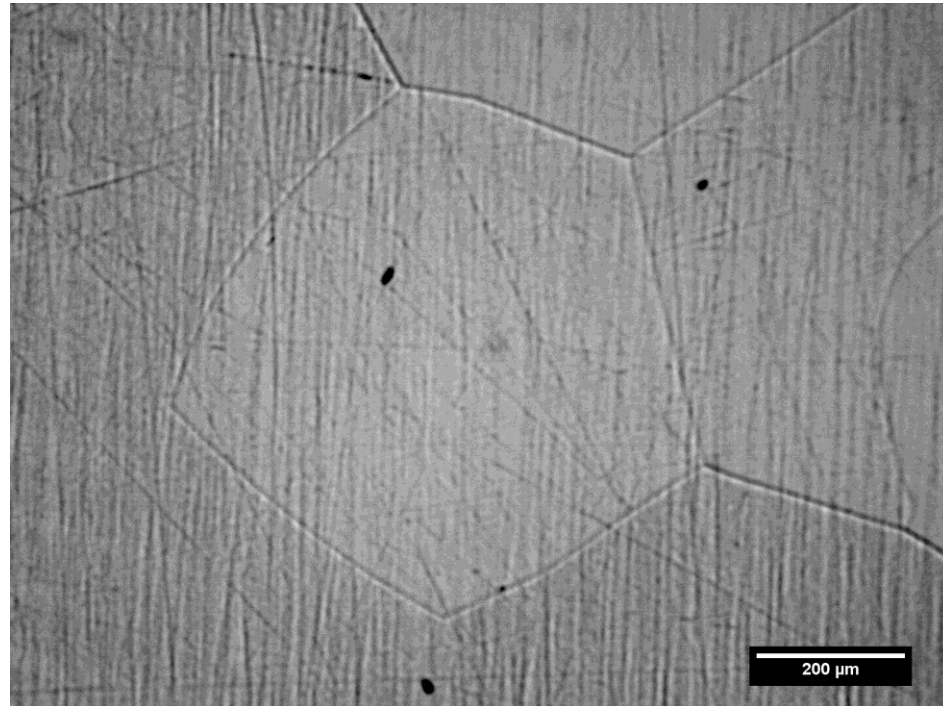
Comparison of As-received and 1000-2h-WQ-10⁻⁴



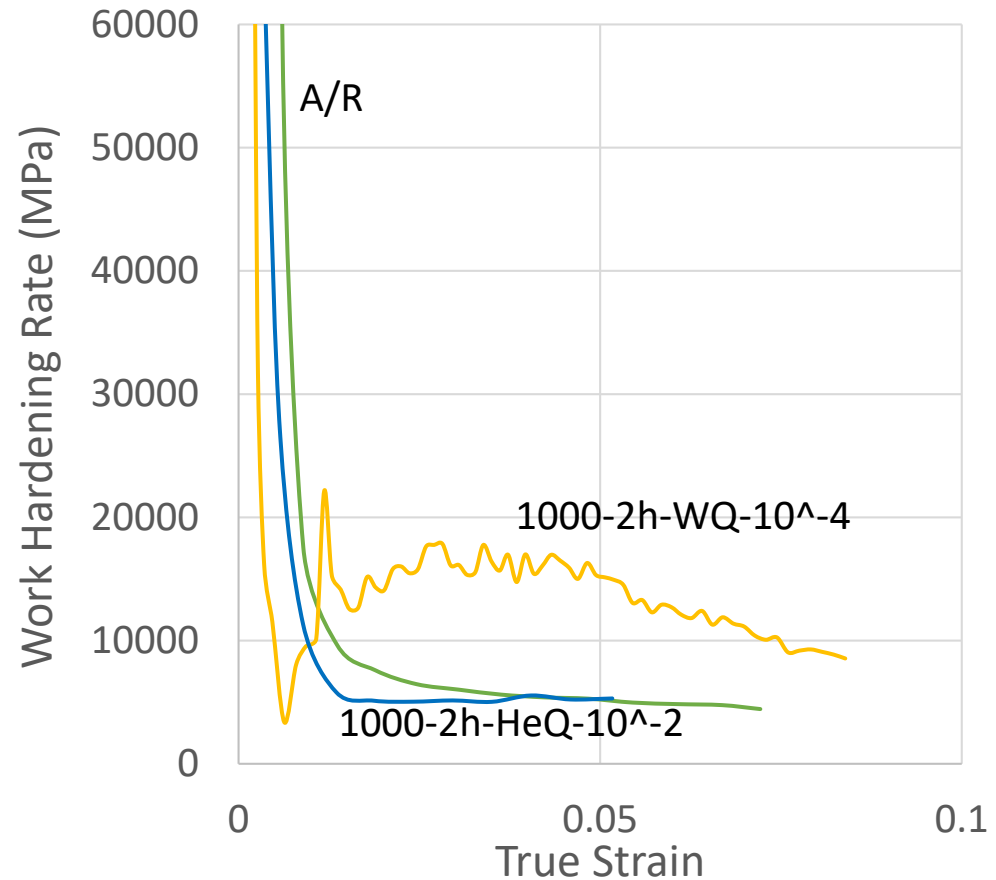
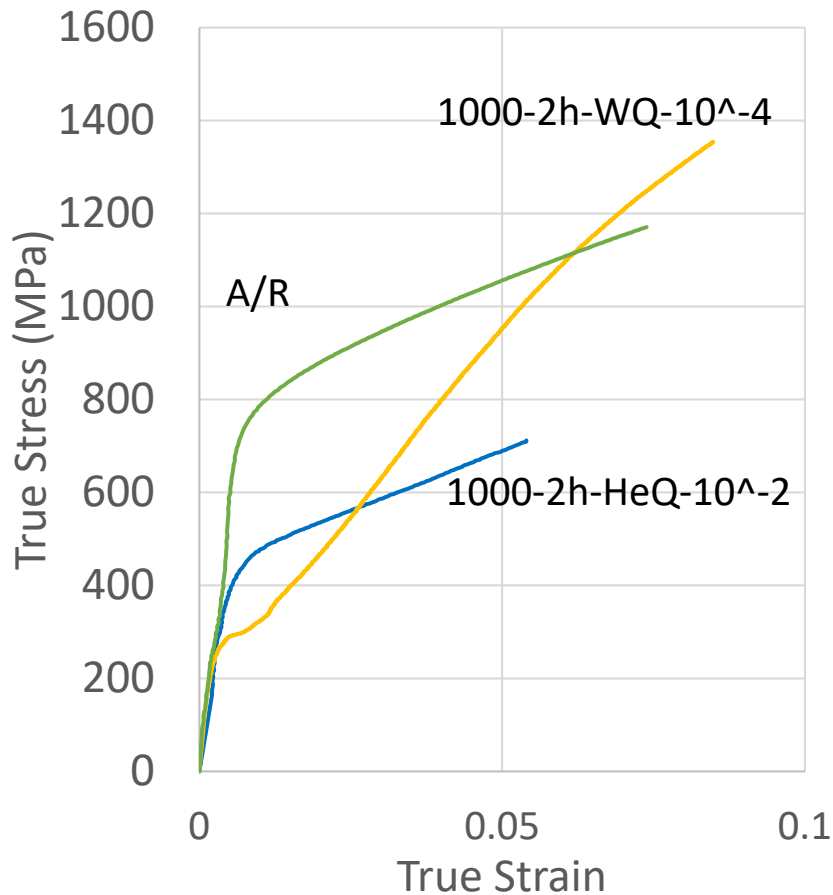
Instantaneous Work Hardening Rate comparison



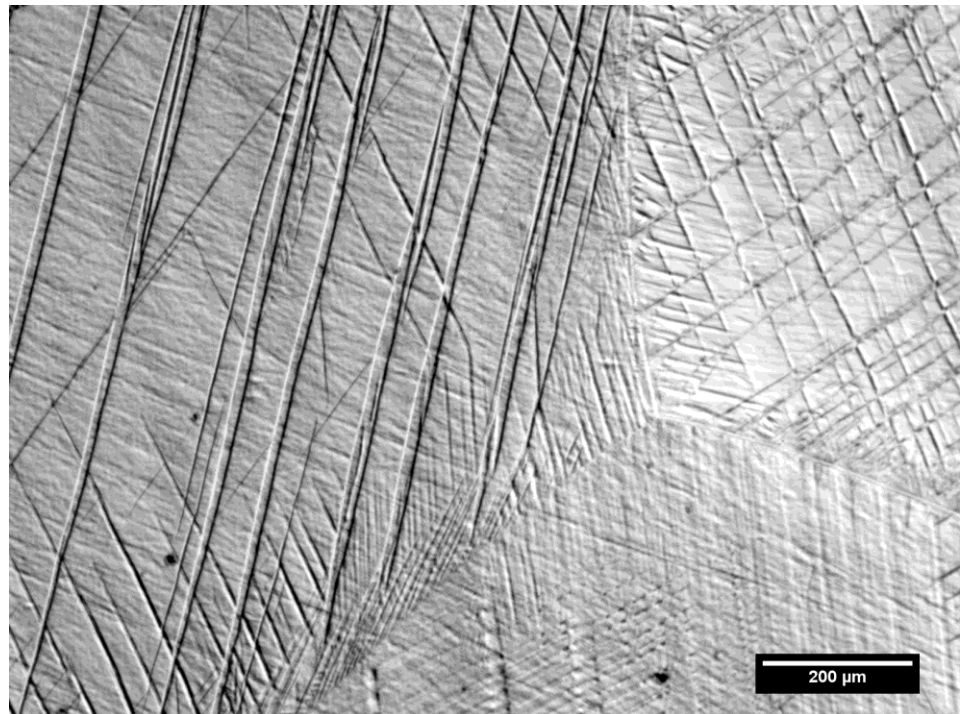
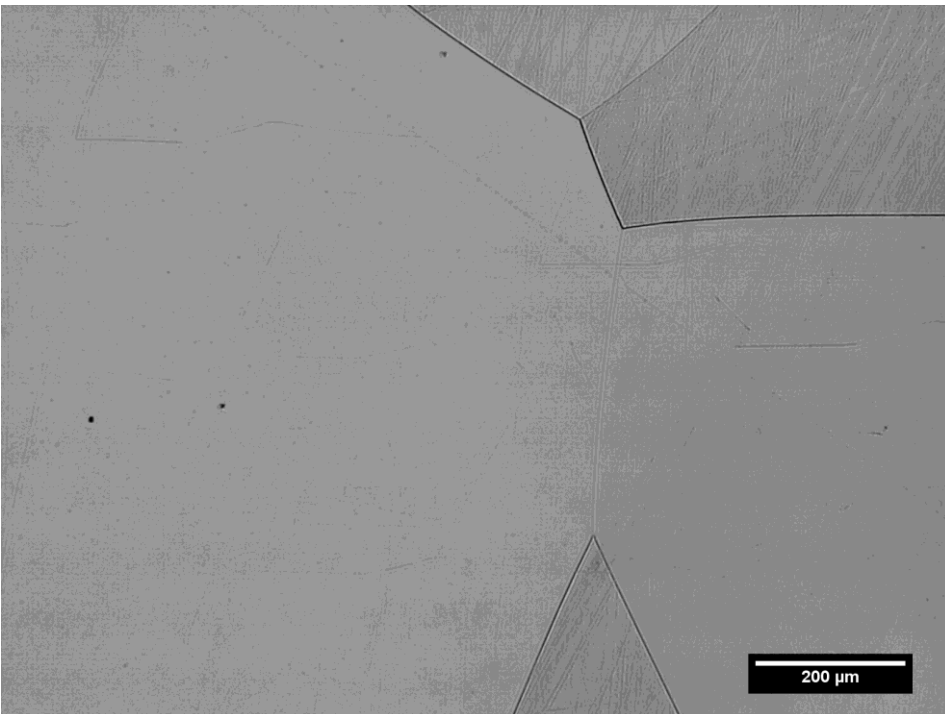
Comparison of Pre- and Post-Compression Microstructure of 1000-2h-WQ-10⁻⁴



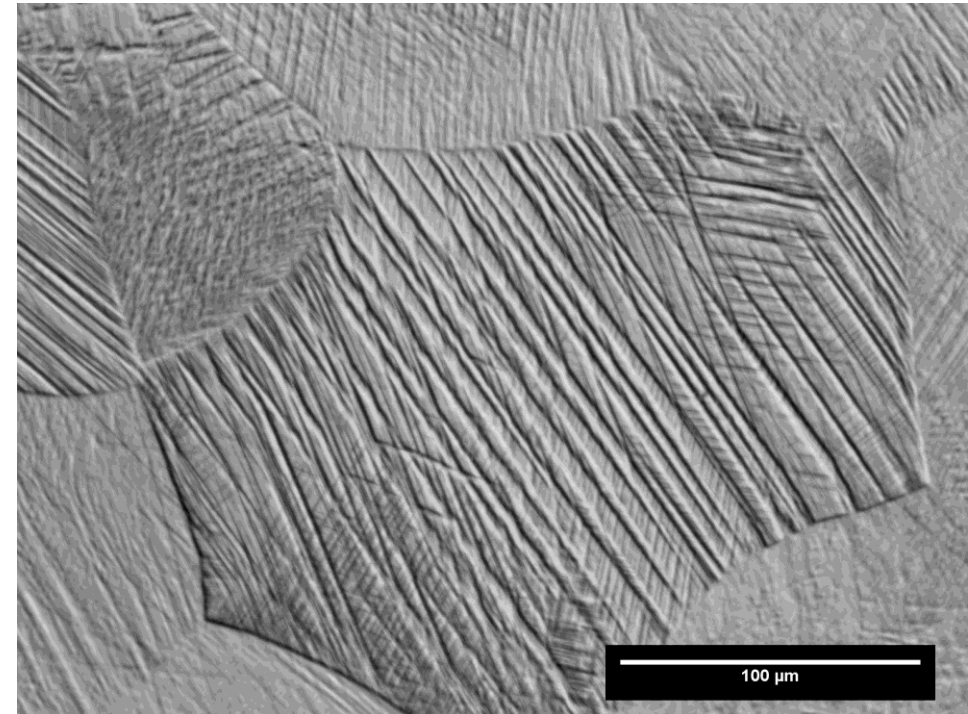
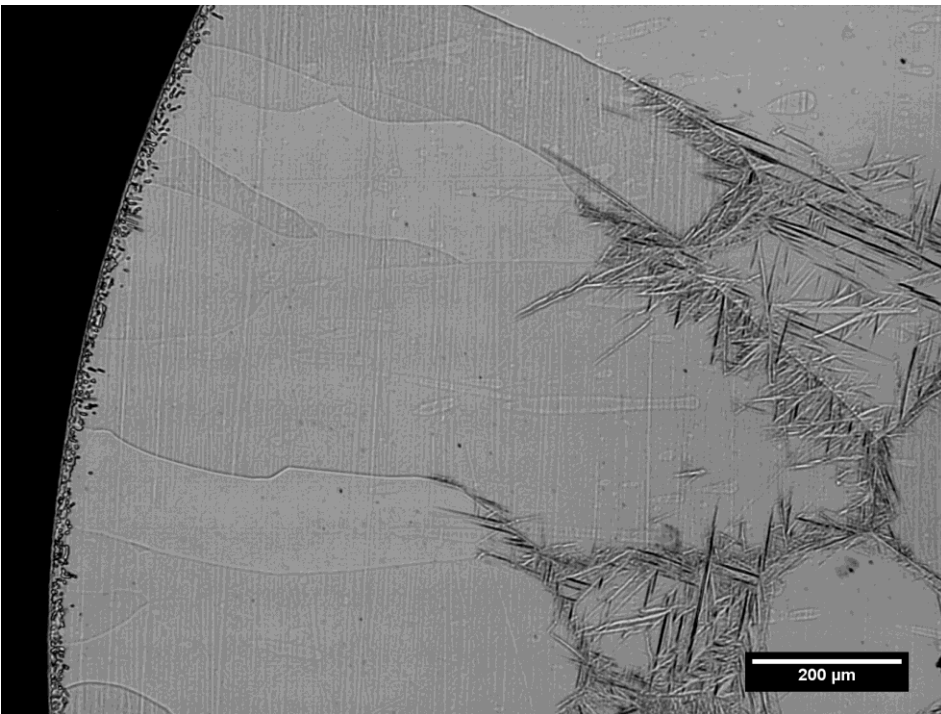
Stress-Strain Behavior does not Suggest TRIP/TWIP ...



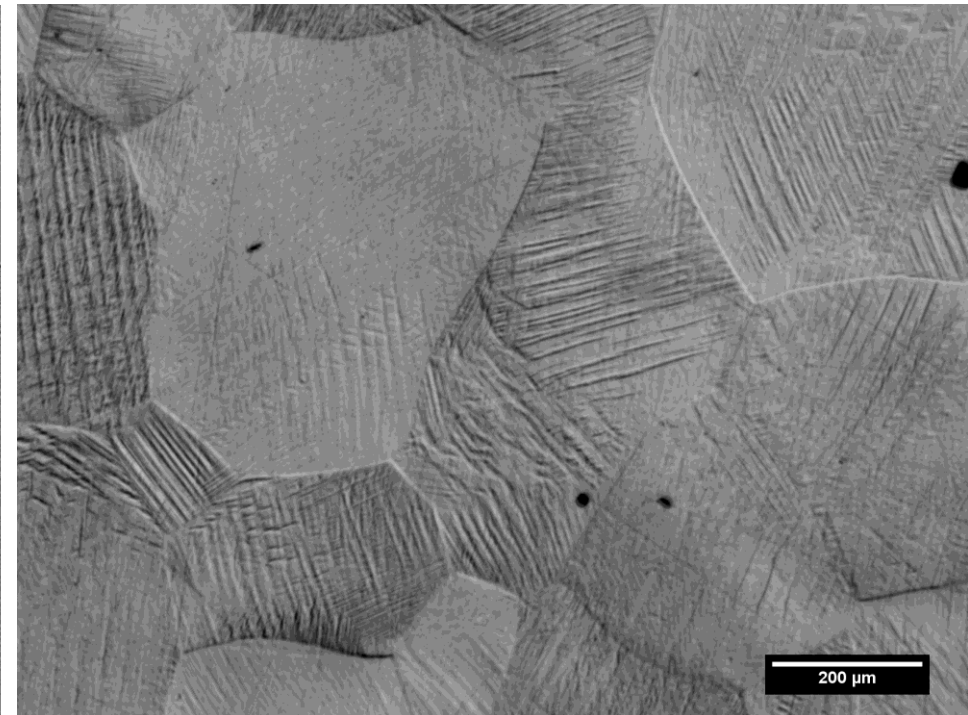
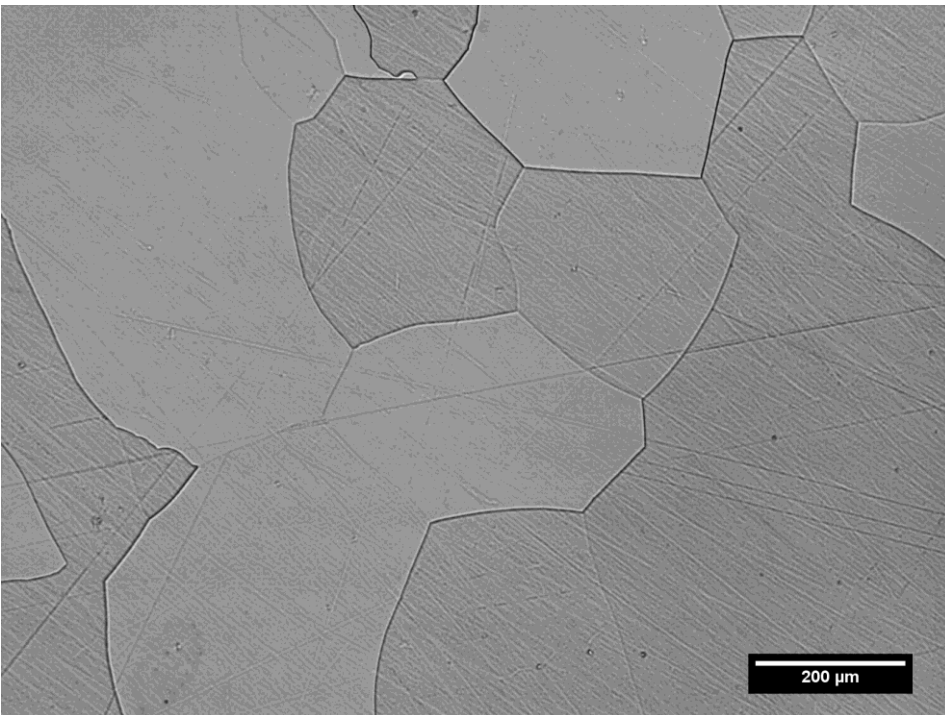
Comparison of Pre- and Post-Compression Microstructure of 1000-2h-HeQ-10⁻²



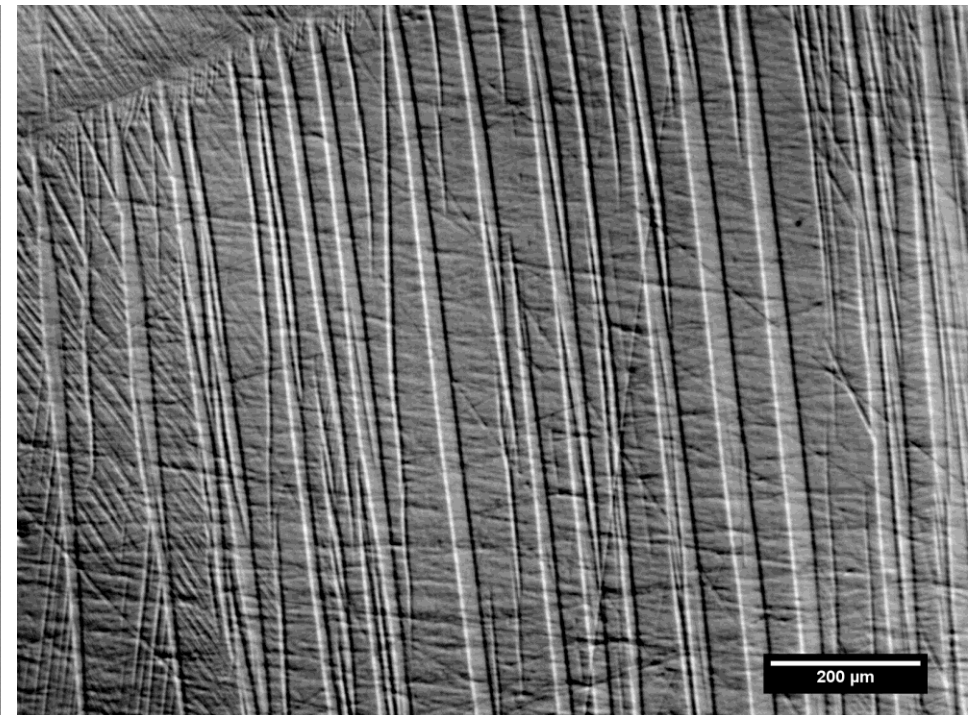
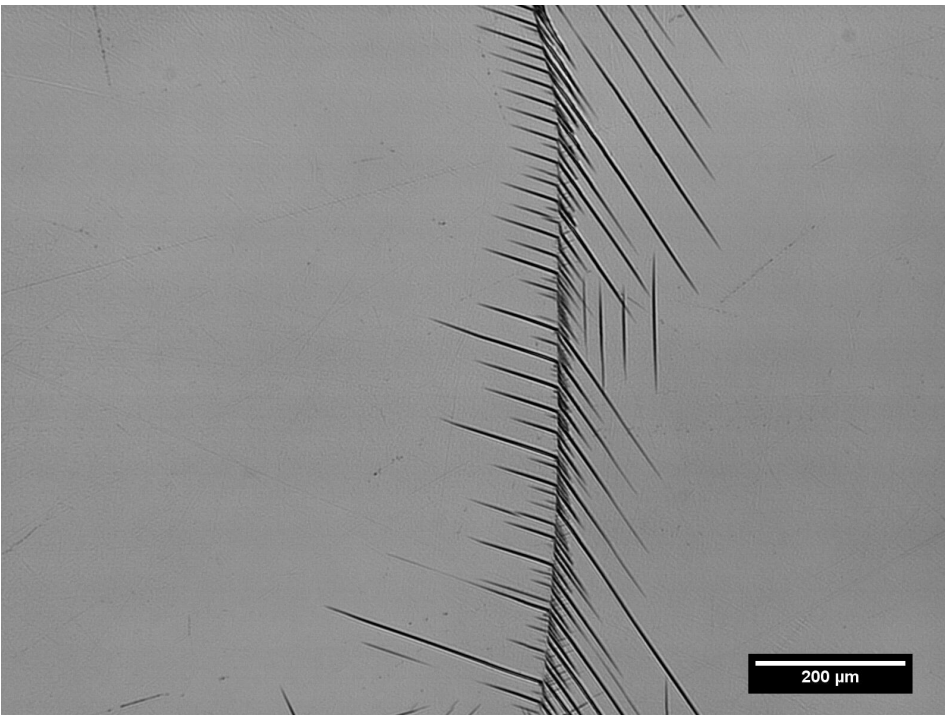
Comparison of Pre- and Post-Compression Microstructure of 900-2h-WQ-10⁻²



Comparison of Pre- and Post-Compression Microstructure of 900-2h-WQ-10⁻²



Comparison of Pre- and Post-Compression Microstructure of 1200-2h-HeQ-10⁻²



Compression Study Summary

Every heat treatment investigated shows TRIP/TWIP in post-compression microstructure

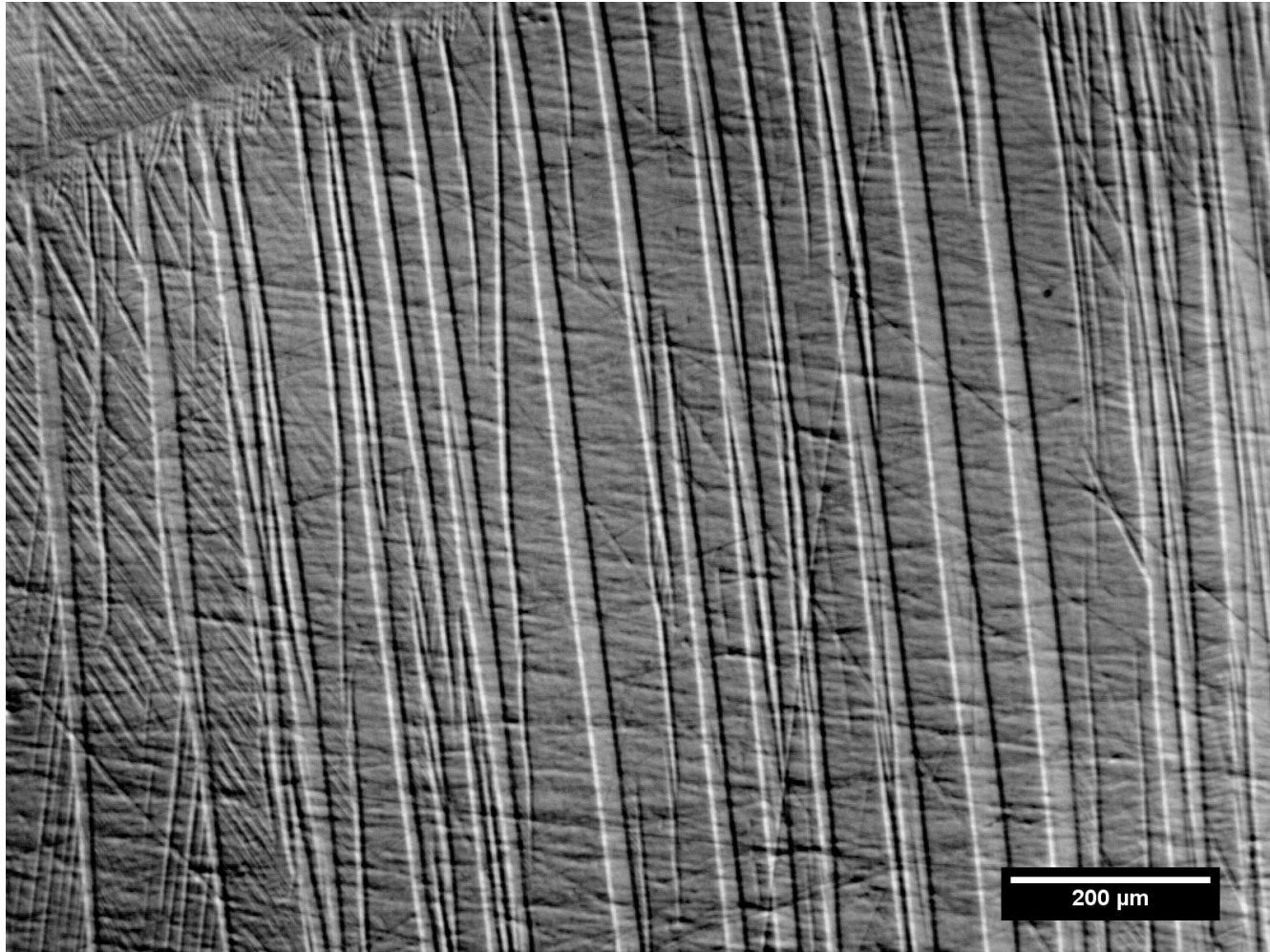


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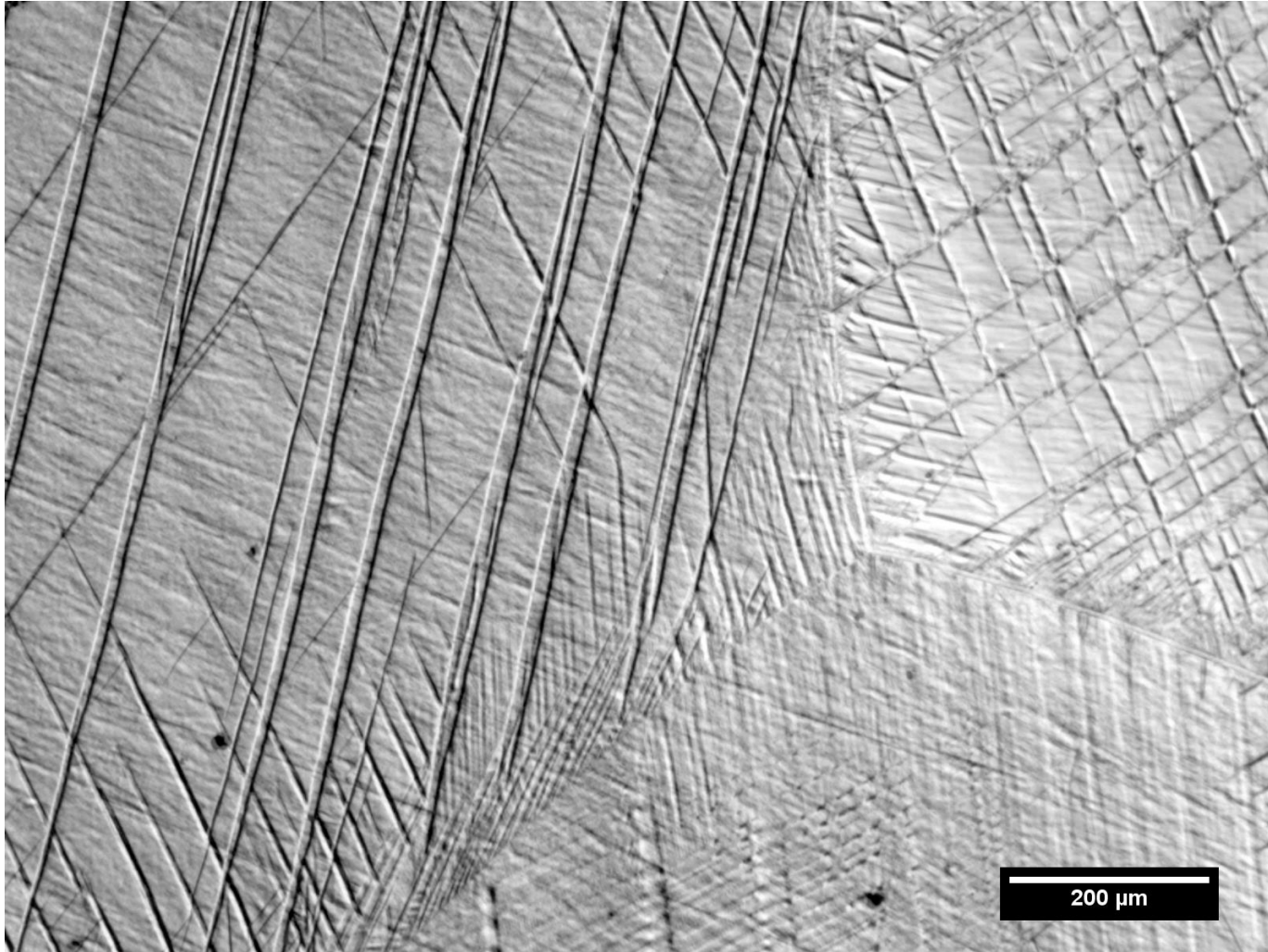


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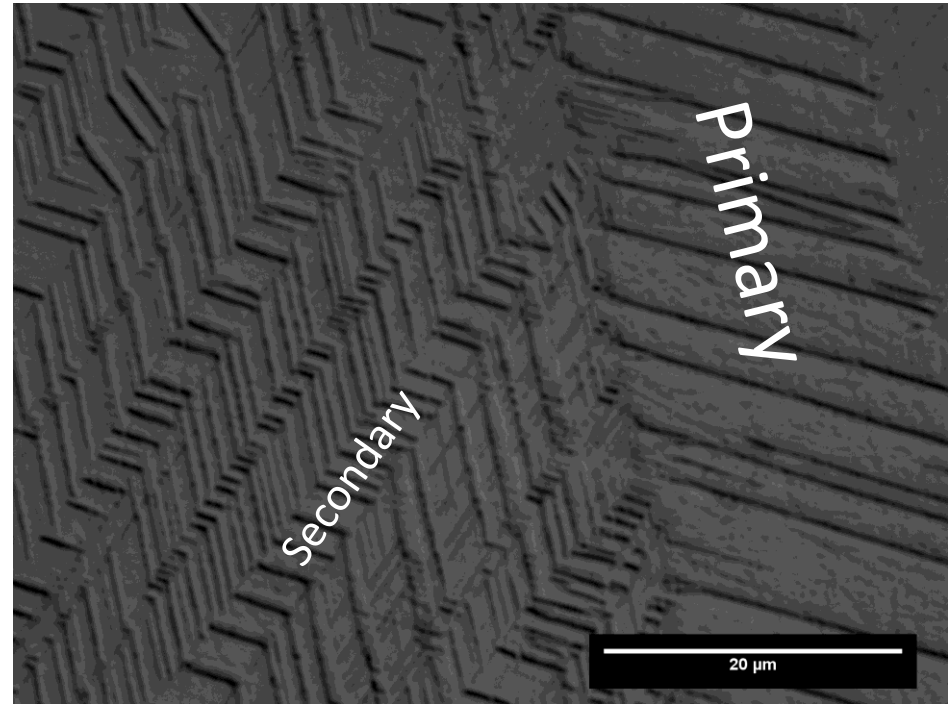
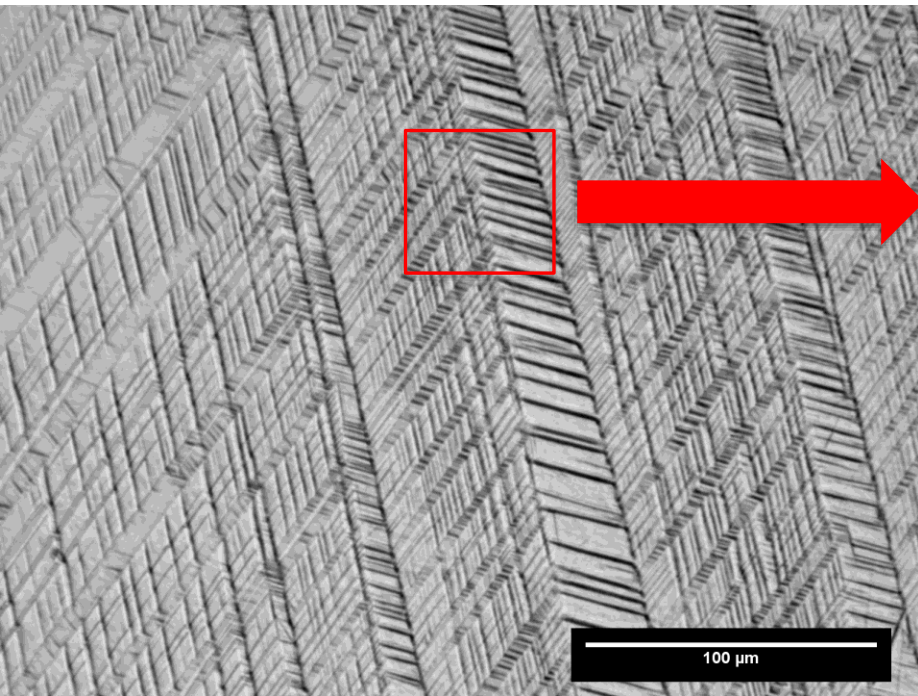
Orientation of Product Similar Within a Single Grain



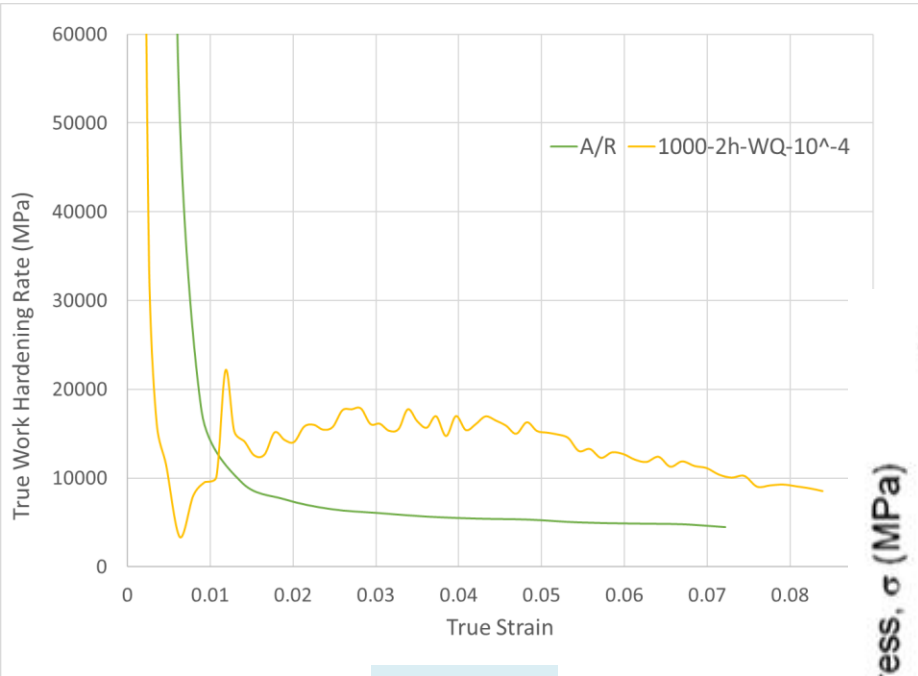
Multiple Variants Can Be Present in a Single Grain



Hierarchy of Deformation Product

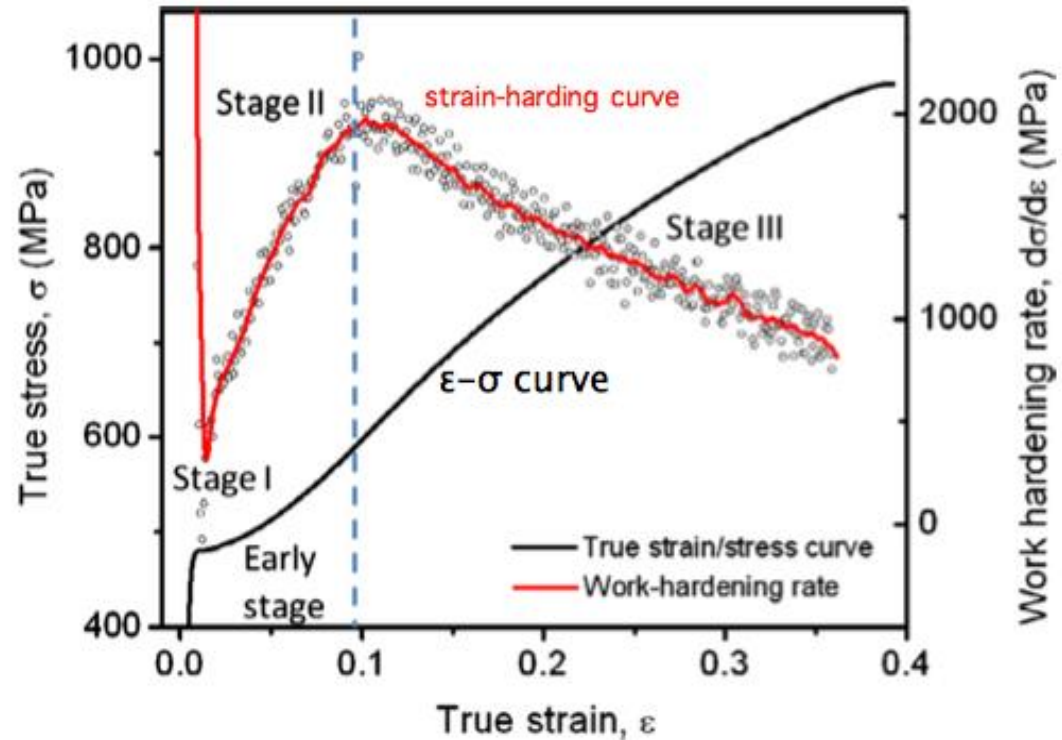


Work Hardening Rate and TRIP/TWIP Effect



Ti-1023

Ti-12Mo (wt.%)



Sun, F. et al. 61 Acta Materialia (2013): 6406-6417

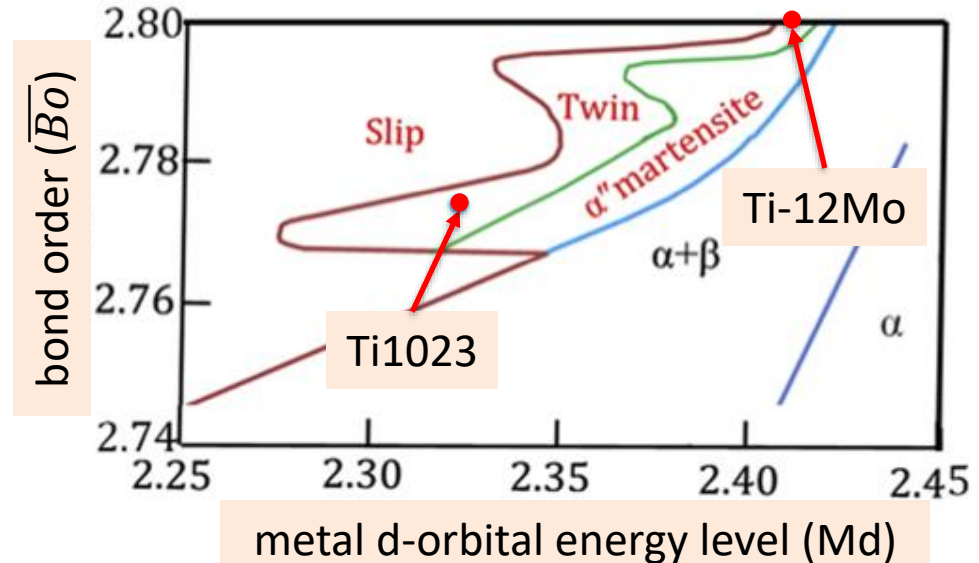


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Deformation Modes in β -Ti Alloys at Room Temperature

- Twinning
 - $\{112\}\langle 111\rangle$ twinning
 - $\{332\}\langle 113\rangle$ twinning
- Stress induced martensite
- Stress induced ω phase
- Slip

Morinaga's phase stability map

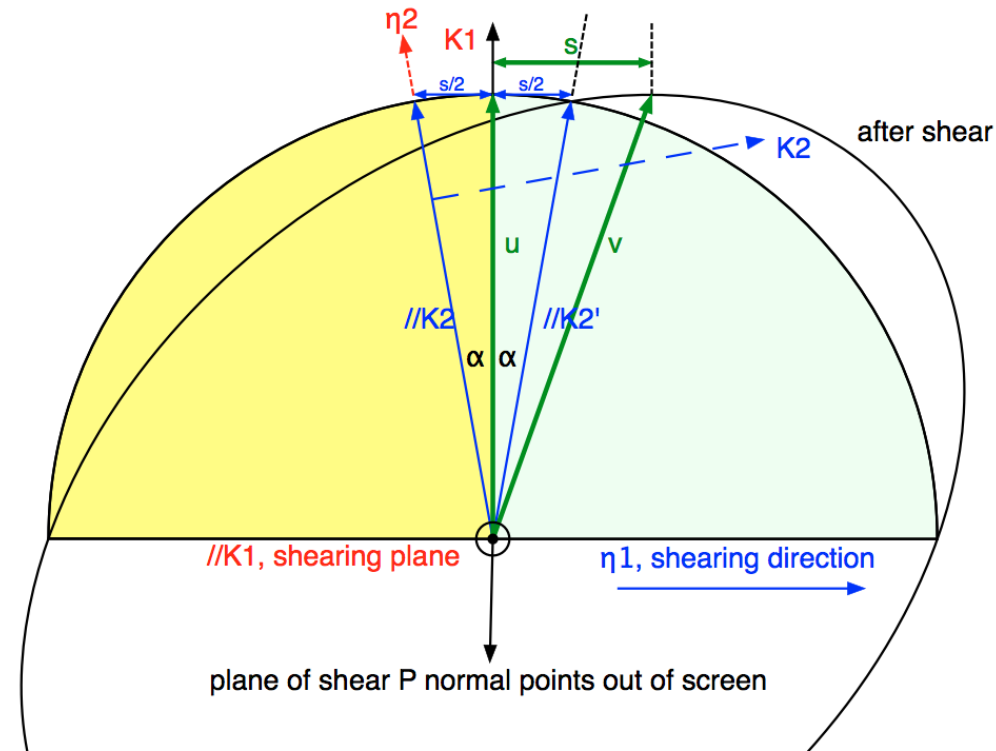


Ahmed, M., et al. 104 Acta Materialia (2016): 190-200

Two Twinning Systems

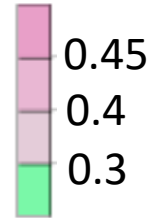
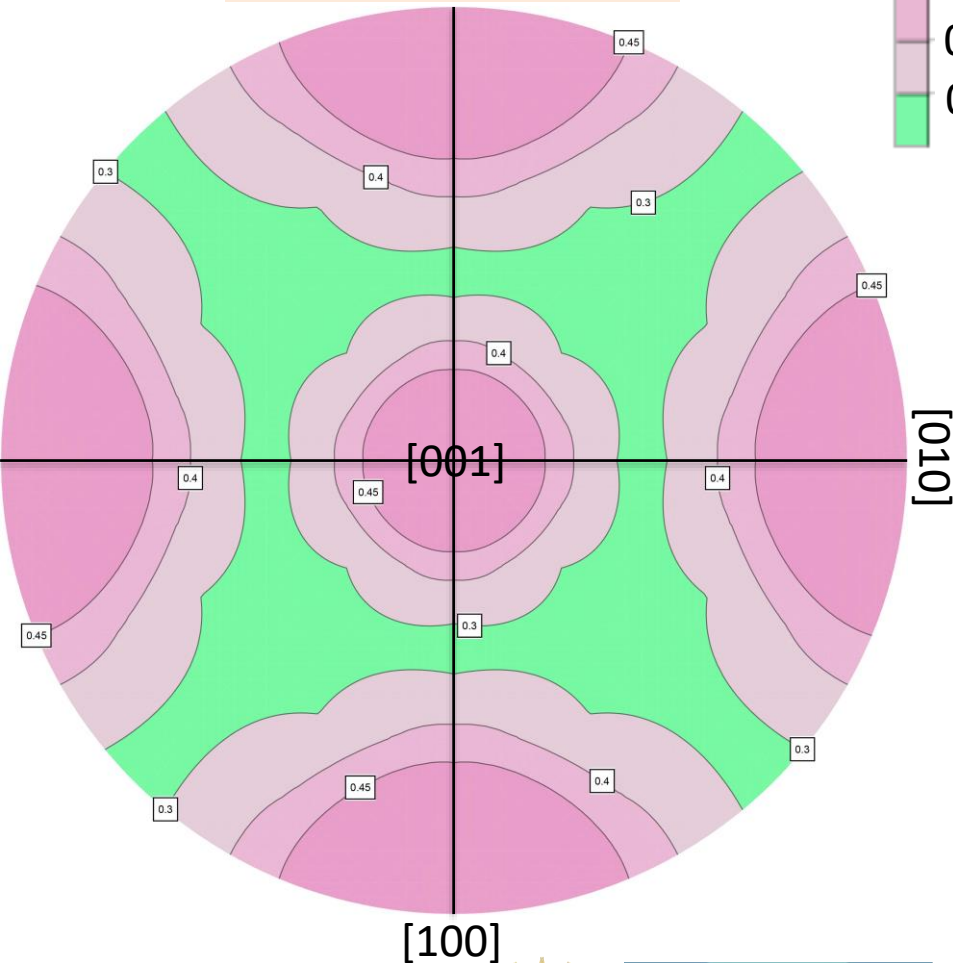
{112}<111> twinning	{332}<113> twinning
(112)[$\bar{1}\bar{1}\bar{1}$]	(332)[11 $\bar{3}$]
(121)[$\bar{1}\bar{1}\bar{1}$]	(323)[1 $\bar{3}\bar{1}$]
(211)[1 $\bar{1}\bar{1}$]	(233)[$\bar{3}\bar{1}\bar{1}$]
($\bar{1}\bar{1}\bar{2}$)[1 $\bar{1}\bar{1}$]	(3 $\bar{3}\bar{2}$)[1 $\bar{1}\bar{3}$]
($\bar{1}\bar{2}\bar{1}$)[11 $\bar{1}$]	(3 $\bar{2}\bar{3}$)[131]
($\bar{2}\bar{1}\bar{1}$)[$\bar{1}\bar{1}\bar{1}$]	(2 $\bar{3}\bar{3}$)[$\bar{3}\bar{1}\bar{1}$]
(1 $\bar{1}\bar{2}$)[$\bar{1}\bar{1}\bar{1}$]	($\bar{3}\bar{3}\bar{2}$)[$\bar{1}\bar{1}\bar{3}$]
(1 $\bar{2}\bar{1}$)[$\bar{1}\bar{1}\bar{1}$]	($\bar{3}\bar{2}\bar{3}$)[$\bar{1}\bar{3}\bar{1}$]
(2 $\bar{1}\bar{1}$)[11 $\bar{1}$]	($\bar{2}\bar{3}\bar{3}$)[311]
(11 $\bar{2}$)[$\bar{1}\bar{1}\bar{1}$]	(33 $\bar{2}$)[113]
(12 $\bar{1}$)[$\bar{1}\bar{1}\bar{1}$]	(32 $\bar{3}$)[1 $\bar{3}\bar{1}$]
(21 $\bar{1}$)[1 $\bar{1}\bar{1}$]	(23 $\bar{3}$)[$\bar{3}\bar{1}\bar{1}$]

- Polarization of twinning and operative twinning

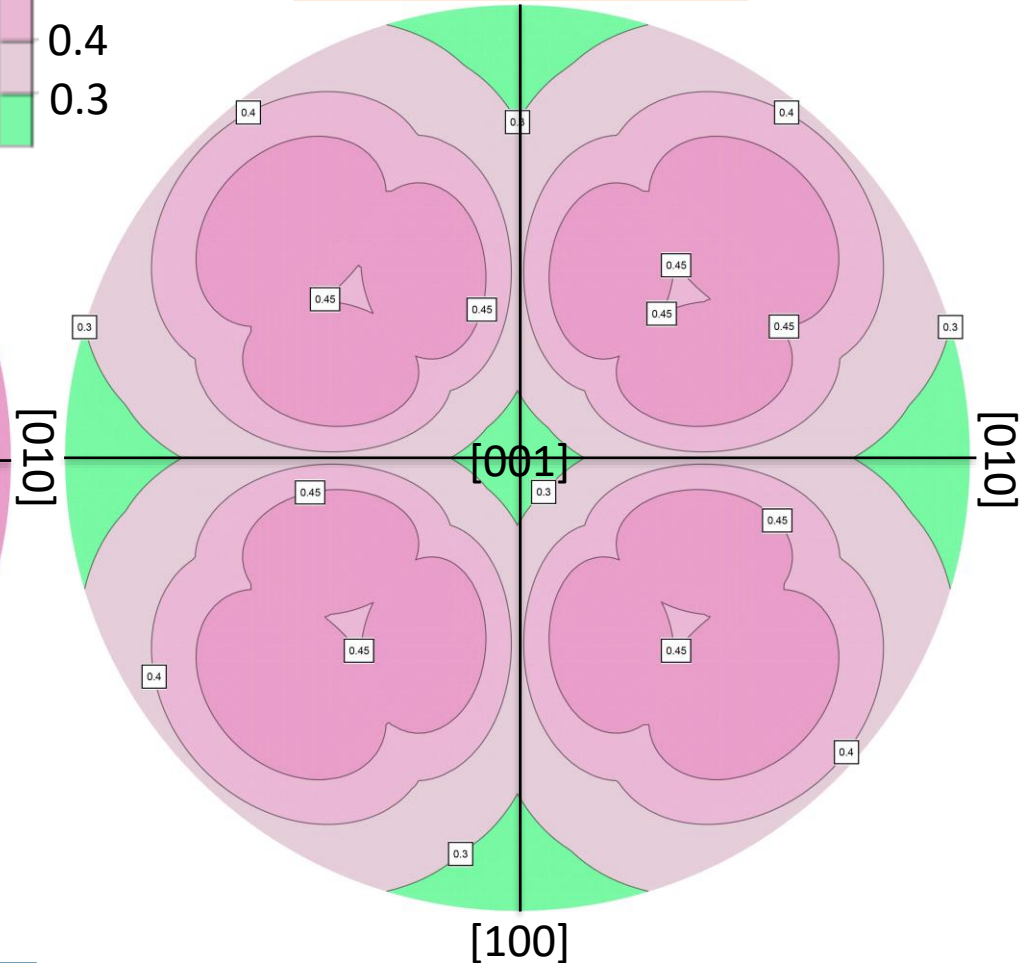


Schmid Factor of Two Twinning Systems

{112}<111> Twinning



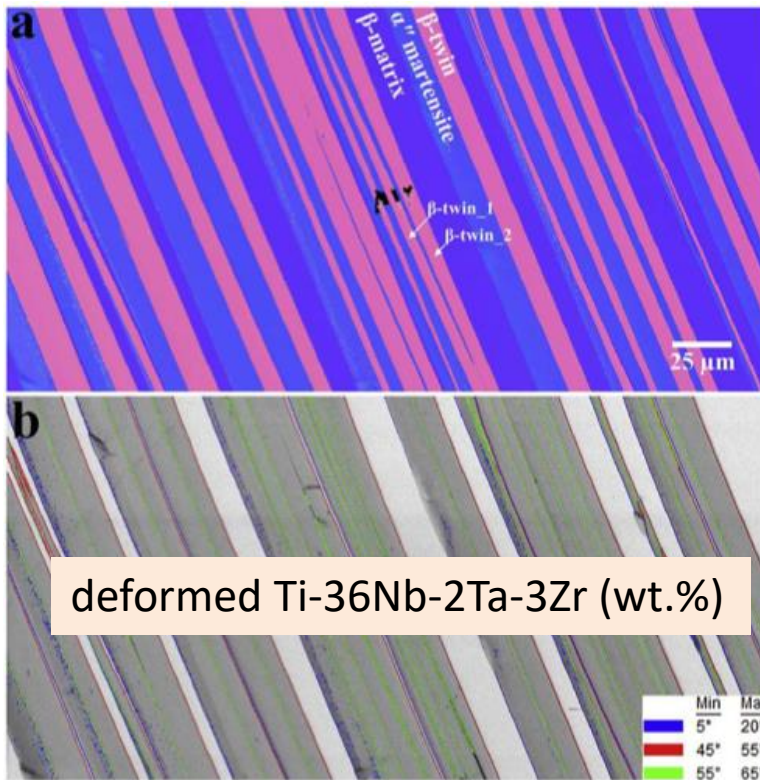
{332}<113> Twinning



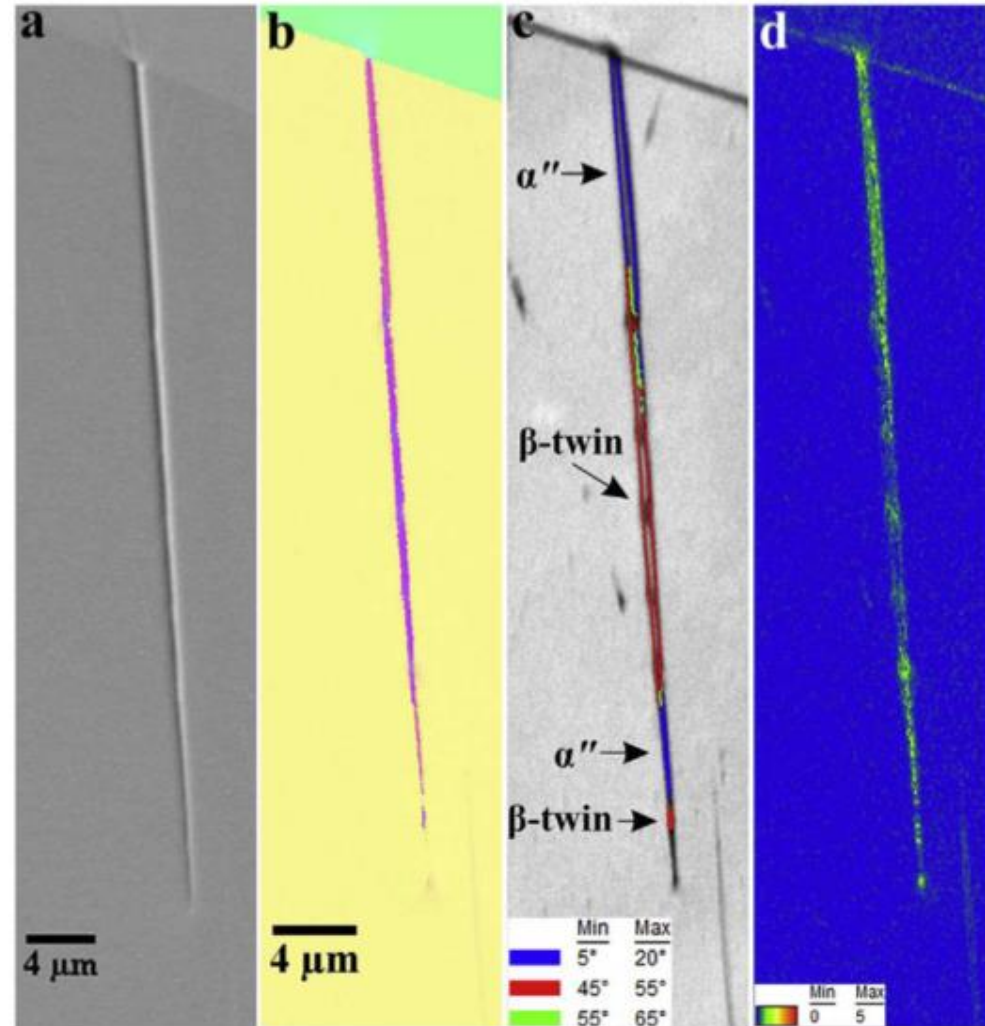
Coupled {332} Twinning and Stress Induced Martensitic Transformation

Misorientation

- {112} twinning: $\Sigma 3$, 60° - $\langle 111 \rangle$
- {332} twinning: $\Sigma 11$, 50.57° - $\langle 110 \rangle$



deformed Ti-36Nb-2Ta-3Zr (wt.%)



Lai, M.J., et al. 111 Acta Materialia (2016): 173-186

Ongoing EBSD Characterization

- Post-mortem EBSD of deformed samples
- In situ EBSD during deformation
- Understanding the coupling of mechanisms in Ti-1023 will inform future Ti-alloy experiments

Summary

- Compression study nearly complete:
 - Fully equiaxed β microstructure with little/no martensite
 - Evidence of TRIP/TWIP behavior
- EBSD and TEM microstructural characterization is underway
- Tensile testing of Ti-1023 based upon compression testing results
- Ti-15Mo from ATI available

Progress

Gantt Chart for Project 33

Apr/18 May/18 Jul/18 Aug/18 Oct/18 Dec/18 Jan/19



Thank you very much!

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References

- [1] Brozek, C., et al. "A β -titanium alloy with extra high strain-hardening rate: design and mechanical properties." *Scripta Materialia* 114 (2016): 60-64.
- [2] Li, C., et al. "Influence of α morphology and volume fraction on the stress-induced martensitic transformation in Ti-10V-2Fe-3Al." *Materials Science and Engineering: A* 528.18 (2011): 5854-5860.
- [3] Bhattacharjee, A., et al. "Effect of β grain size on stress induced martensitic transformation in β solution treated Ti-10V-2Fe-3Al alloy." *Scripta materialia* 53.2 (2005): 195-200.
- [4] Duerig, T. W., et al. "Formation and reversion of stress induced martensite in Ti-10V-2Fe-3Al." *Acta Metallurgica* 30.12 (1982): 2161-2172.
- [5] Neelakantan, S., et al. "Plasticity induced transformation in a metastable β Ti-1023 alloy by controlled heat treatments." *Materials Science and Technology* 25.11 (2009): 1351-1358.
- [6] Sun, F., et al. "Investigation of early stage deformation mechanisms in a metastable β titanium alloy showing combined twinning-induced plasticity and transformation-induced plasticity effects." *Acta Materialia* (2013): 6406-6417.
- [7] Ahmed, M., et al. "Strain rate dependence of deformation-induced transformation and twinning in a metastable titanium alloy." *Acta Materialia* (2016): 190-200.
- [8] Lai, M.J., et al. "On the mechanism of {332} twinning in metastable β titanium alloys." *Acta Materialia* (2016): 173-186.