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

**Fall 2018 Semi-Annual Meeting**
**Colorado School of Mines, Golden, CO**
**October 2-4, 2018**

Student: Benjamin Ellyson (Mines)
Faculty: Amy Clarke (Mines)
Industrial Mentors: Austin Mann (Boeing)
Other Participants: Yaofeng Guo (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

- Ti-1023 (wt.%) quasi-static compression study completed
- Initial Ti-1023 quasi-static tensile testing underway
- Deformation mechanism of Ti-1023 identified by TEM
- Initial microstructural characterization of Ti-15Mo (wt.%)

**Metrics**

| Description | % Complete | Status |
|---|---|---|
| 1. Literature review | 70% | ● |
| 2. Quasi-static mechanical characterization of Ti-1023 and Ti-15Mo | 40% | ● |
| 3. Dynamic testing of Ti-1023 and Ti-15Mo | 0% | ● |
| 4. Microstructural characterization of pre- and post- deformed samples | 30% | ● |
| 5. *In situ* characterization of microstructural evolution during deformation | 0% | ● |

# 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 at $10^{-3}$ $s^{-1}$ and 0.18 True Strain

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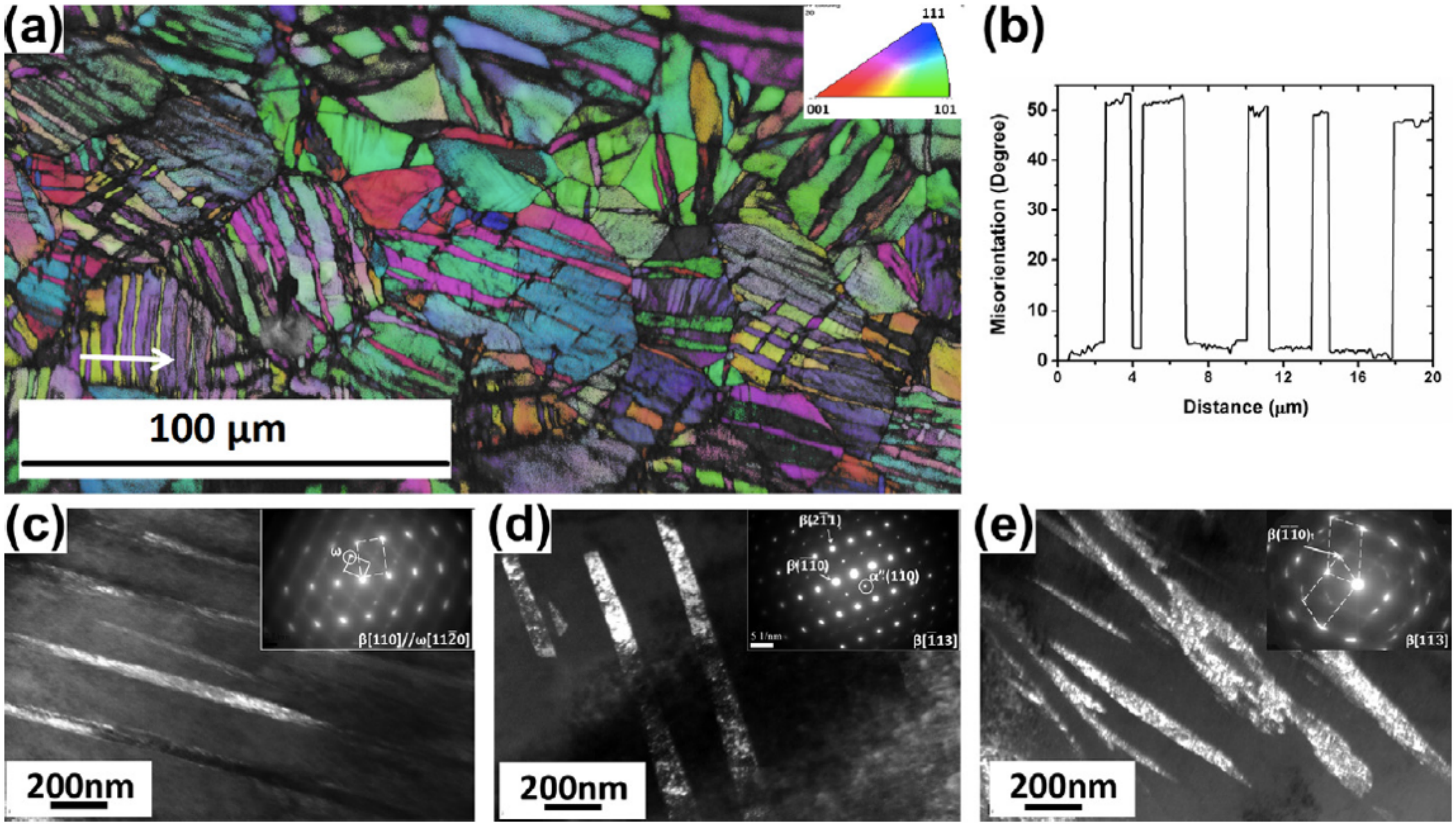


| Deformation-induced ω phase | Deformation-induced α” phase | {112}<111> β mechanical twinning |
|---|---|---|

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

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

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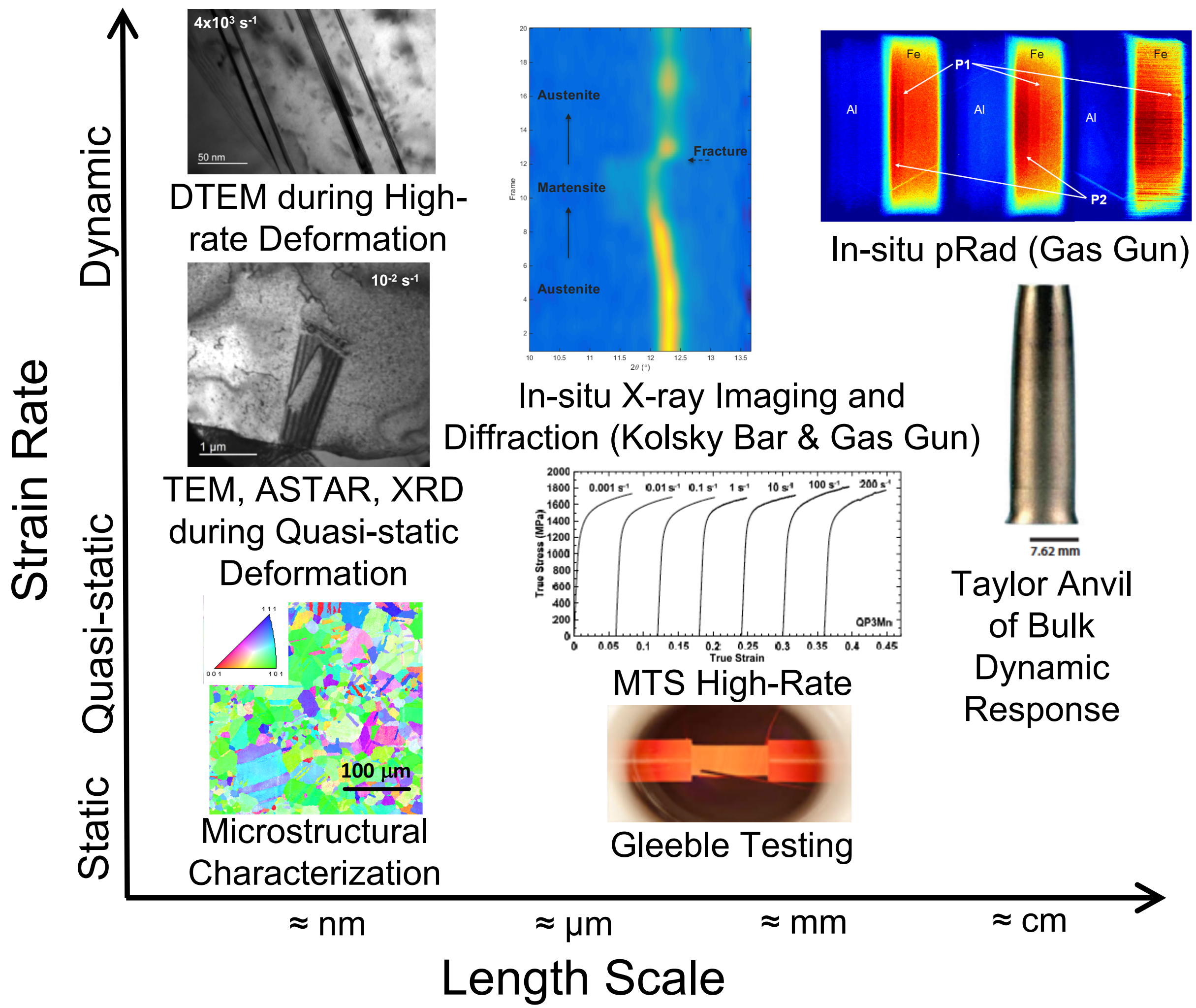

# The Effect of Strain Rate ($10^{-3}$, $10^{-1}$, $10^{1}$, $10^{2}$ $s^{-1}$) on Deformation Mechanisms during Compression of a Ti-10V-3Fe-3Al (wt.%) Alloy

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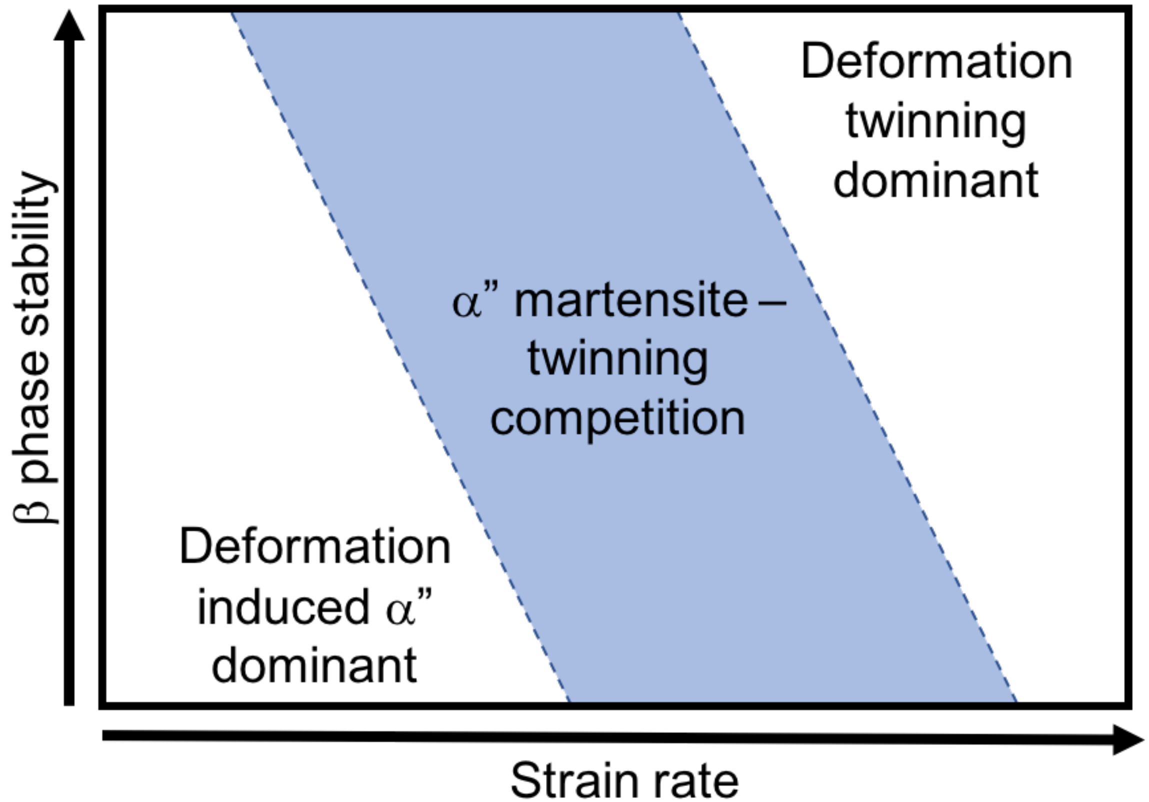
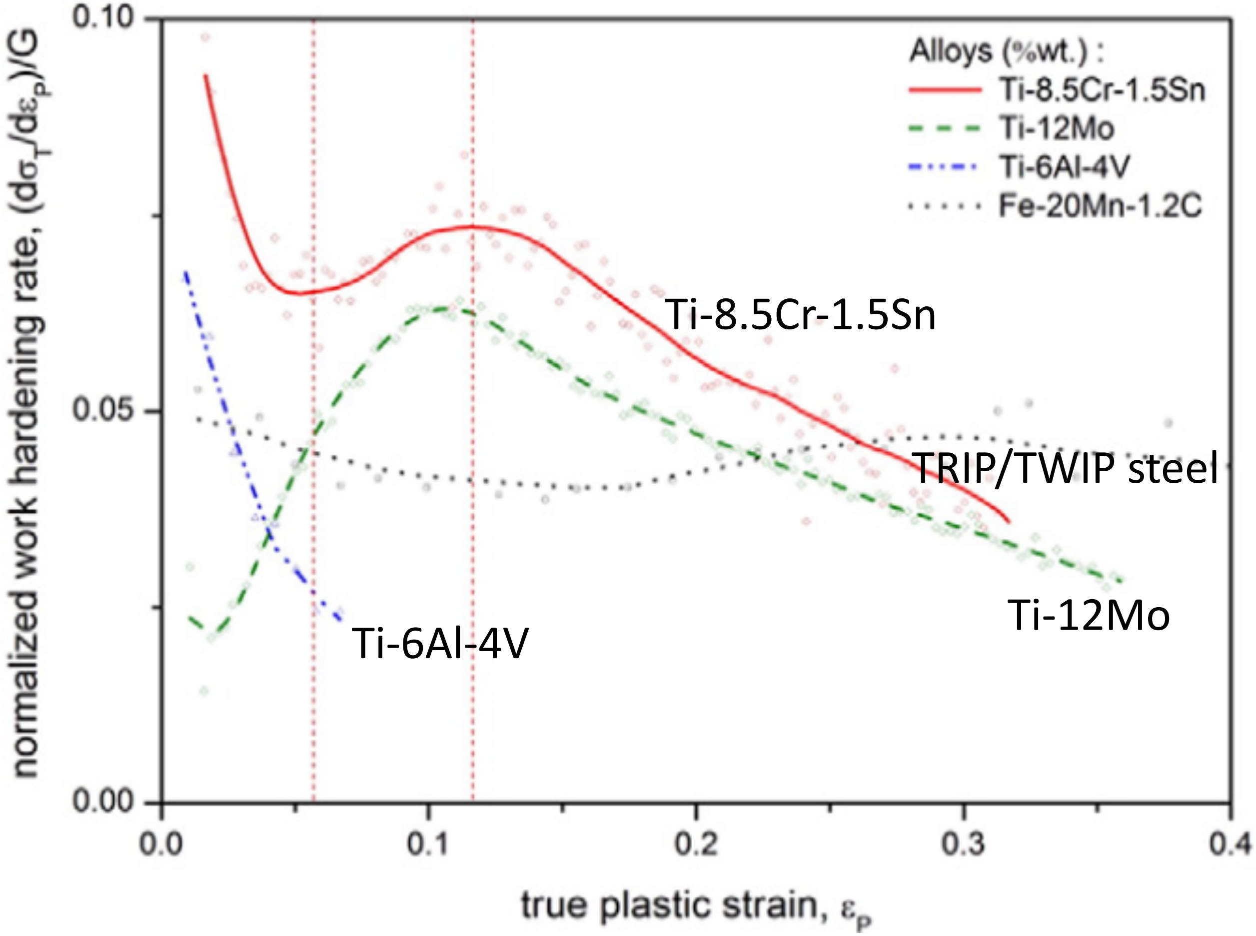
- **As strain rate increases:**
  - Stress-induced α” martensite (dominant) + {332}<113> β twinning + stress-induced ω phase + slip
  - Stress-induced α” martensite + {332}<113> β twinning + stress-induced ω phase + slip
  - Stress-induced α” martensite + {332}<113> β twinning (dominant) + stress-induced ω phase + slip




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

# Work Hardening and Evidence of TRIP/TWIP




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

# Improvements in Sample Preparation

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- Consulting Buhler, Struers, LECO and Oxford EBSD
- Reading literature

## Automatic mechanical polishing methods

- Fast, high quality and reproducibility

| grit/grain size | abrasive | duration (seconds) | load (N) | rotation (rpm) |
|---|---|---|---|---|
| 320 grit | SiC paper | until flat | 5 | 350/175 |
| 9 µm | diamond suspension | 300 | 5 | 150/75 |
| 0.02 µm | 5 OP-S + 1 $H_2O_2$ | 600 | 10 | 150/75 |

Notes: individual force applied and holder rotates in opposite direction with the platen.

# Comparison of Ti-1023 OM Images

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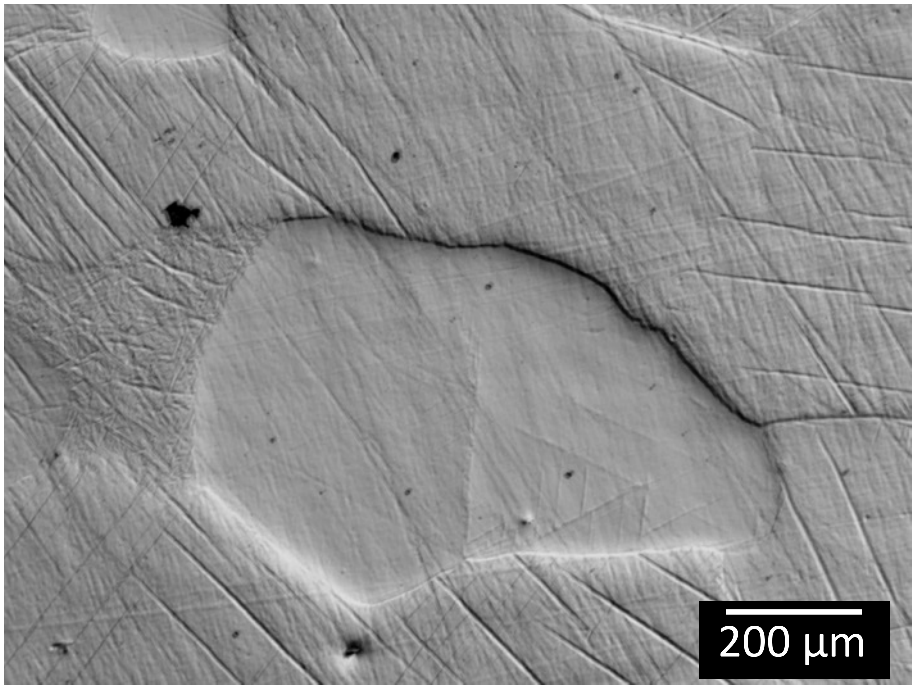
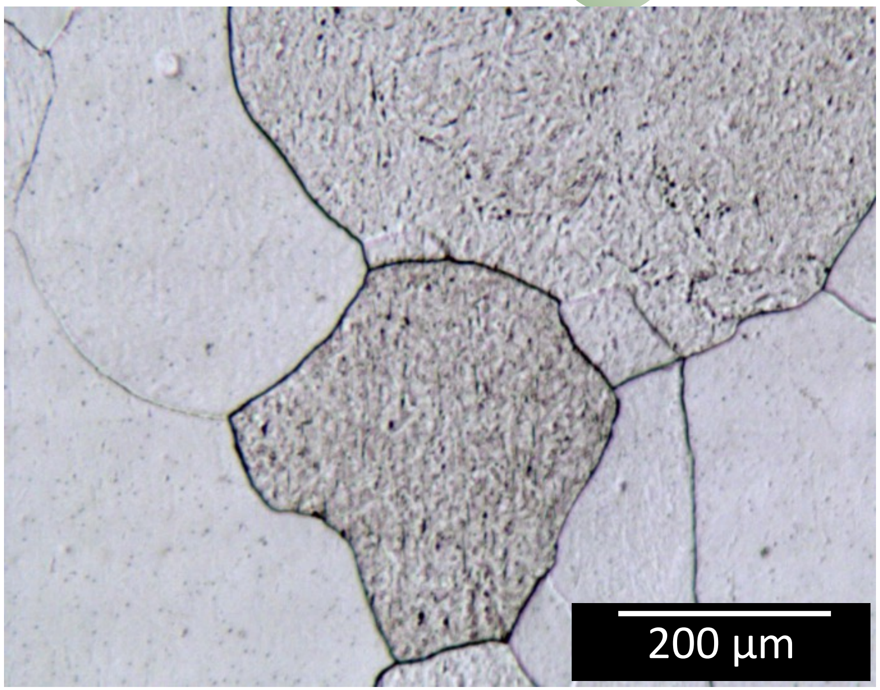



**previously**




**improved**

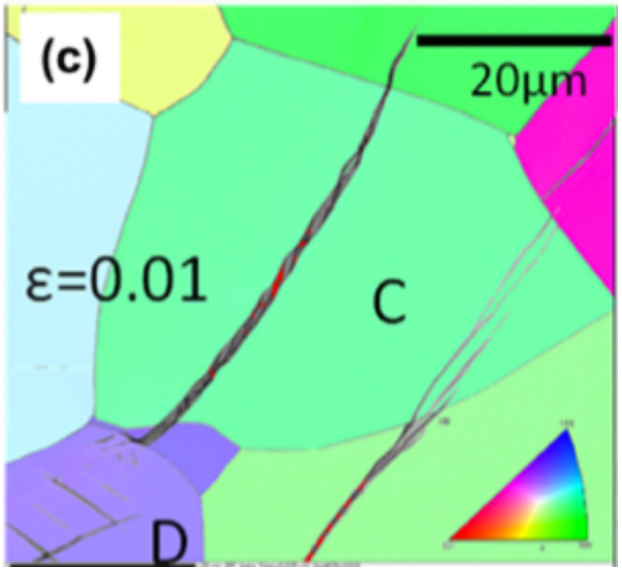
- Etchant
  - left: Kroll's reagent (6% $HNO_3$, 2% HF and 92% DI water)
  - right: 1% HF in DI water
- No more stress-induced martensites due to sample preparation

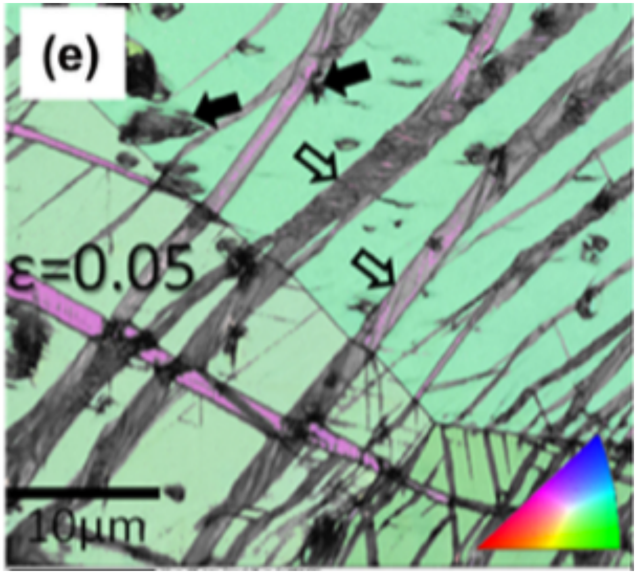
# Difficulty in Obtaining High Quality EBSD Results in Deformed Samples

The indexing level decreases very rapidly with strain, as illustrated by Fig. 3e and f. Only a few twins and martensitic plates can be indexed in this figure, while most of the deformation areas, e.g. the dark clusters (black arrows)

Ti-12Mo

**ε = 1%**




Ti-12Mo

**ε = 5%**

F. Sun et al. / Acta Materialia 61 (2013) 6406

# Preliminary Ti-1023 EBSD Results

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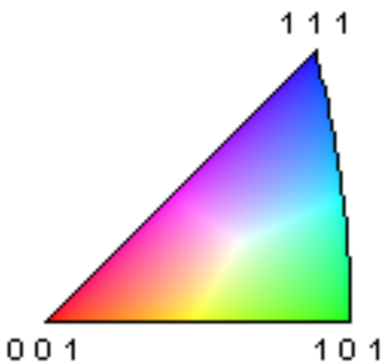
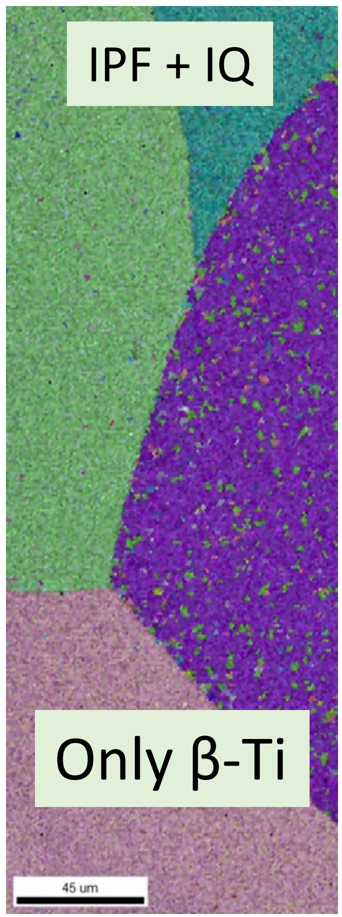
**before deformation**

**2hrs@1200°C**

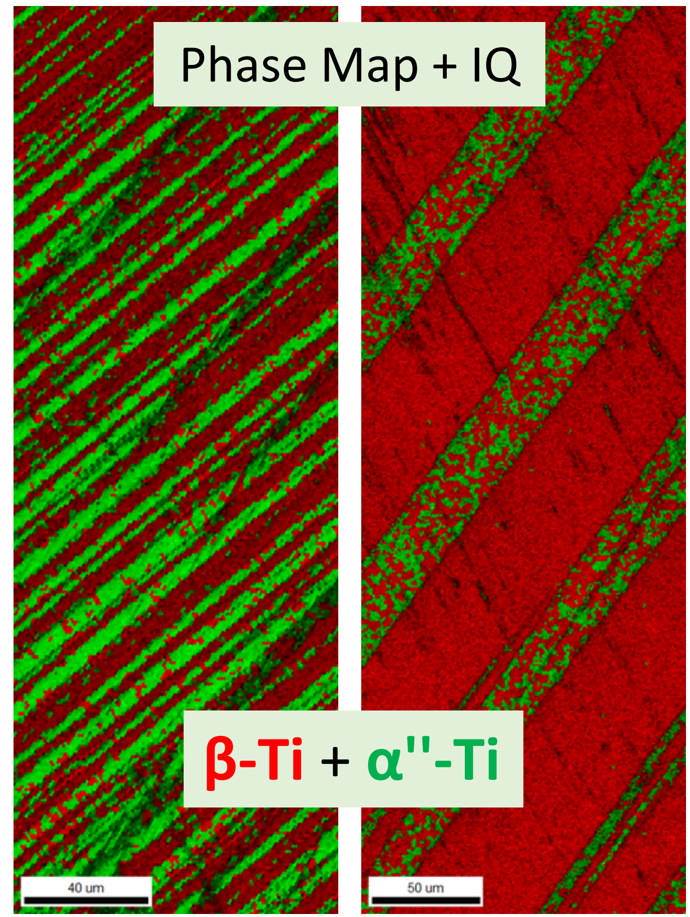
**CI: 0.4 - 0.8**




**after deformation, 5%**

**CI: ~ 0**



# Comparison of Ti-1023 Stress-Strain Behavior in Compression

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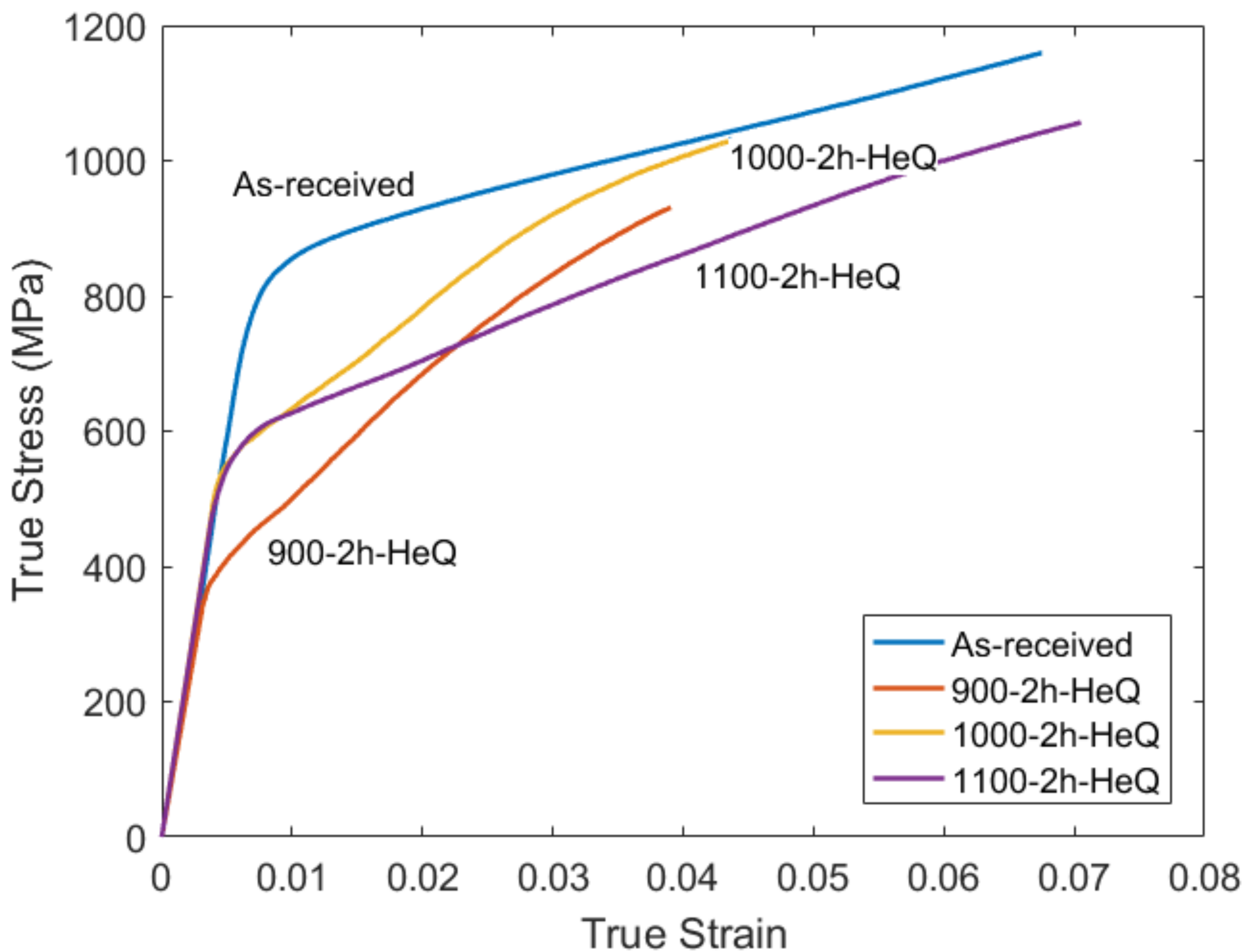

# Trend in 0.2% Offset Yield Stress in Compression

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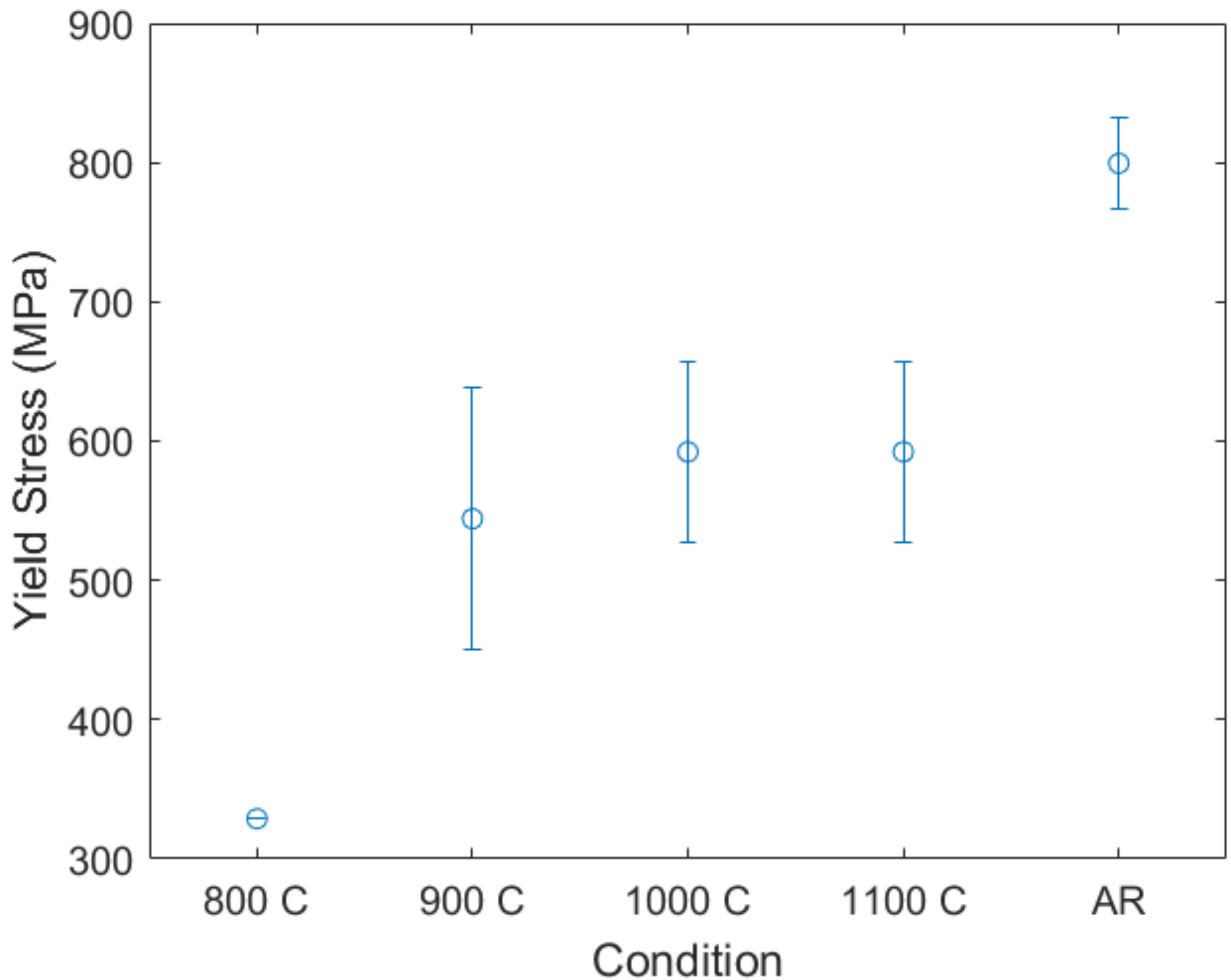
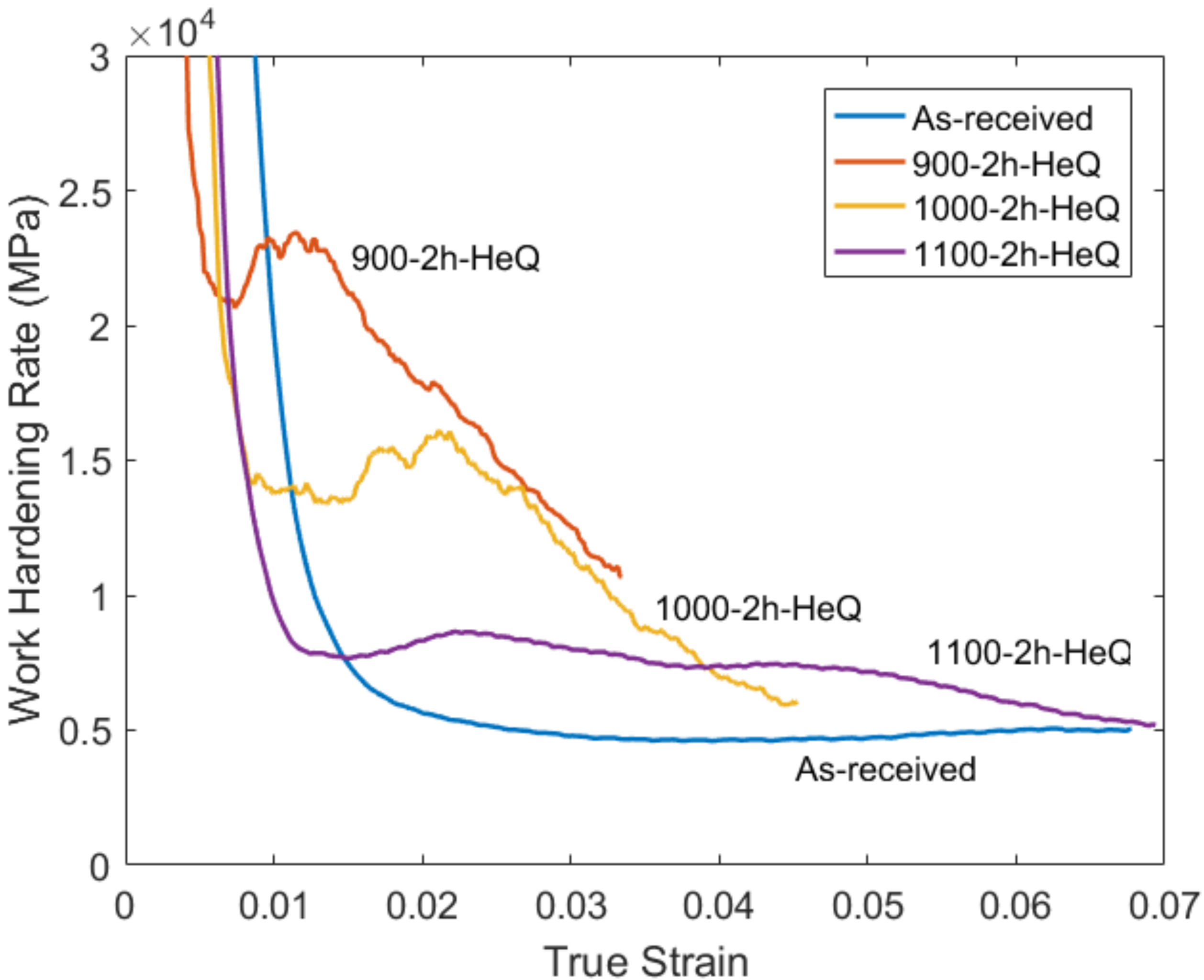
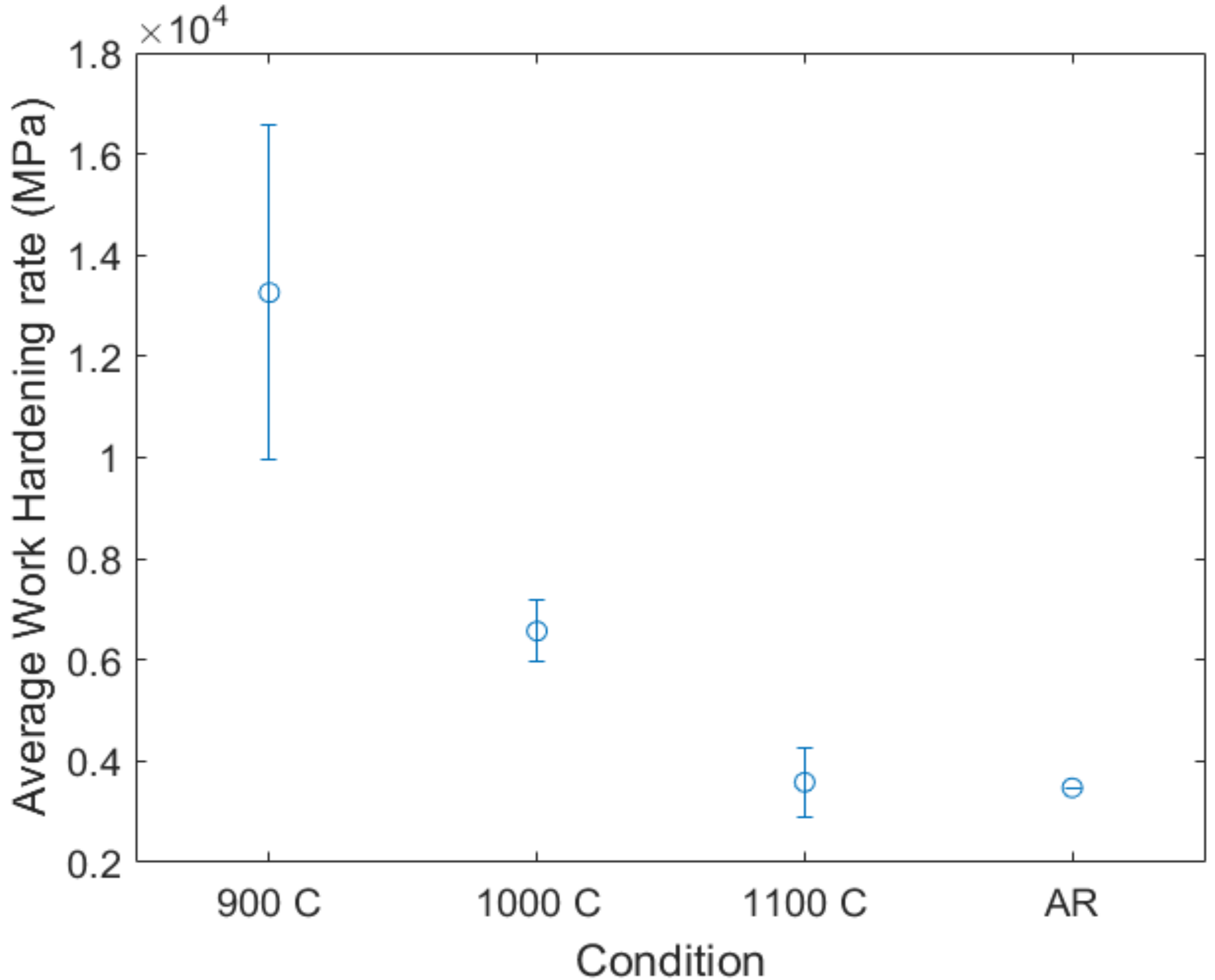



Ti-1023

# Instantaneous Work Hardening as a Function of Solution Temperature



# Trend in Average Work Hardening Behavior




Ti-1023

# Plan for Initial Ti-1023 Tensile Testing

- Lessons from compression study and literature:
  - Grain size/solution temperature has a controlling effect on work hardening behavior
  - Effect of grain size and holding temperature are convoluted

1. 700°C-1h for full annealing
2. 1100°C-3h for grain growth treatment for large grain sample
3. 850°C-1h for final homogenizing solution treatment

# Ti-1023 Microstructure and Grain Size

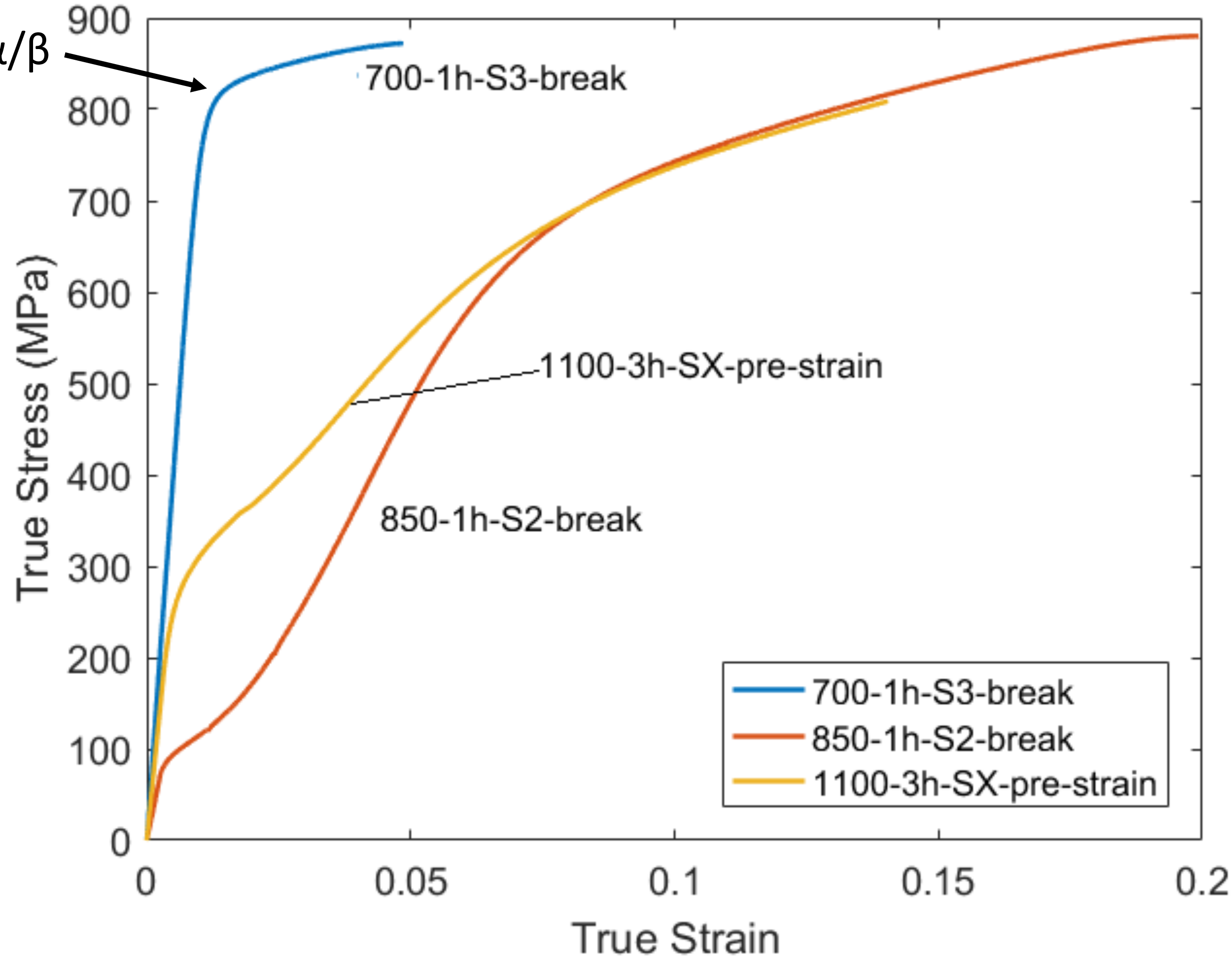
α/β treatment, 700 °C for 1h :

- Roughly 70 % primary α fraction
- Leaves A/R prior β grain boundaries un-modified

| Heat treatment | Grain size |
|---|---|
| 850 °C for 1h, small grain state | ~ 150 µm |
| 1100 °C for 3h, large grain state | ~ 1200 µm |

# Ti-1023 Stress-Strain Behavior in Tension

Typical behavior for fully stable α/β



# Trend inTi-1023 0.2% Offset Yield Stress in Tension

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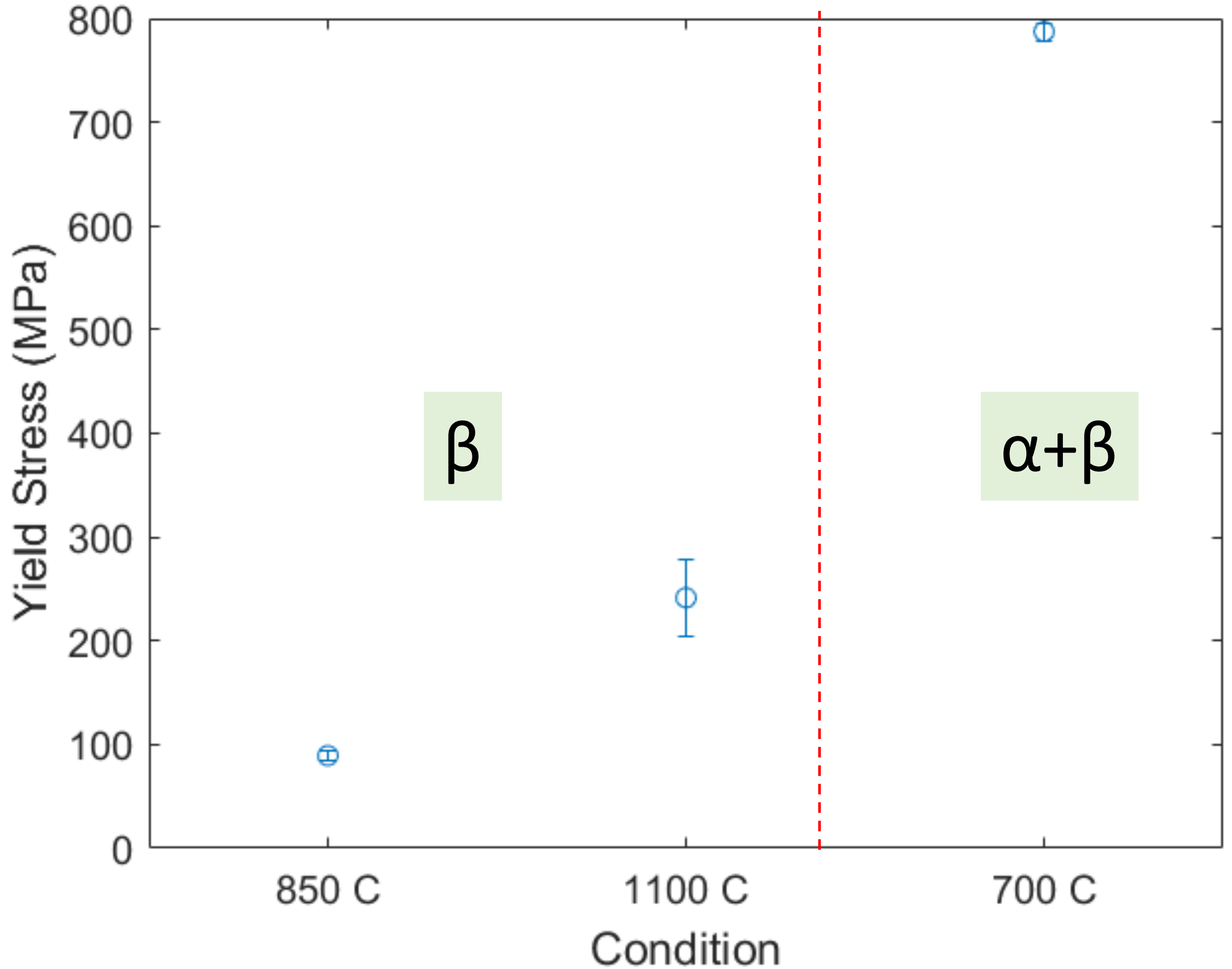

# Ti-1023 Instantaneous Work Hardening Behavior in Tension

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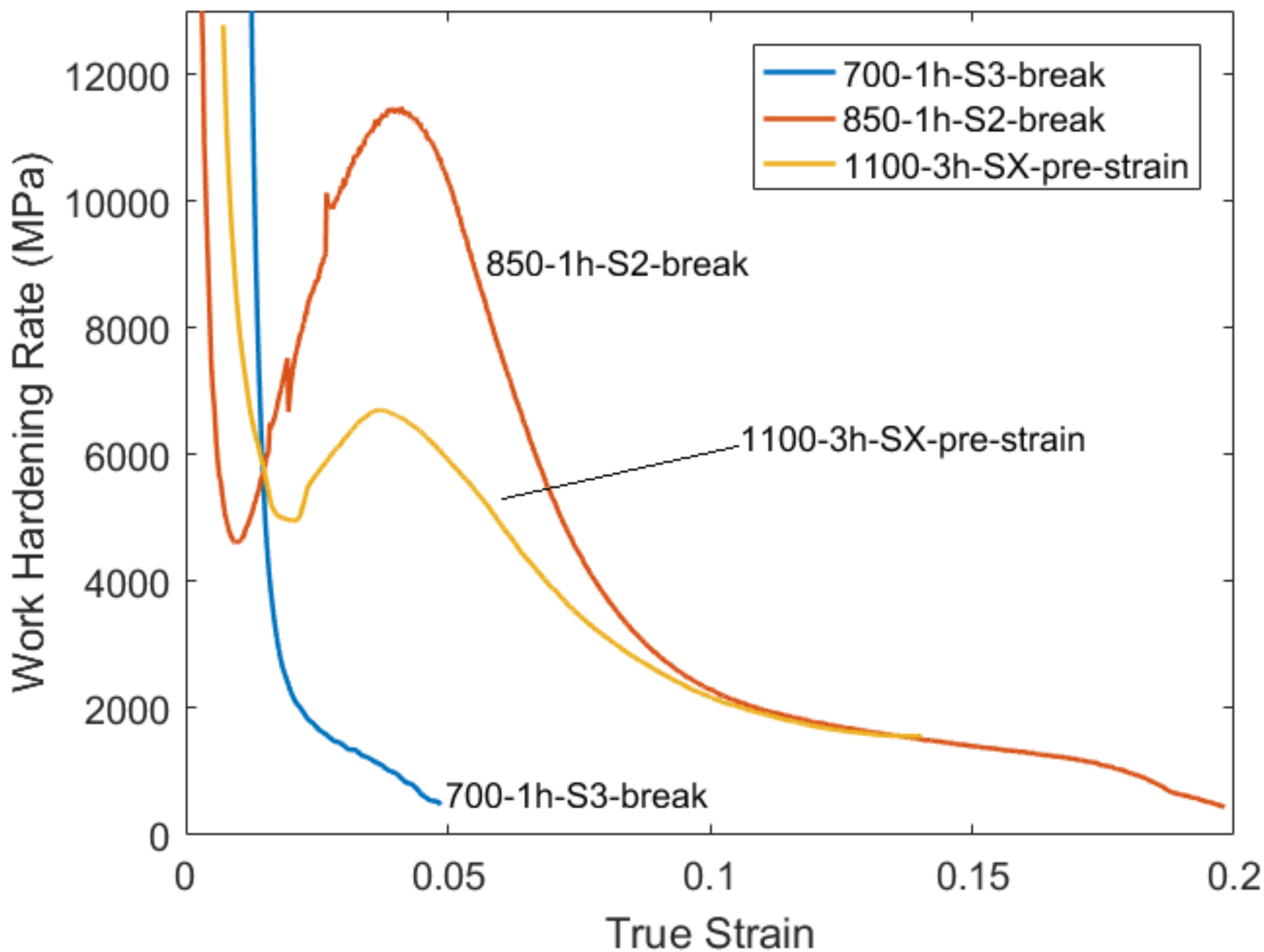
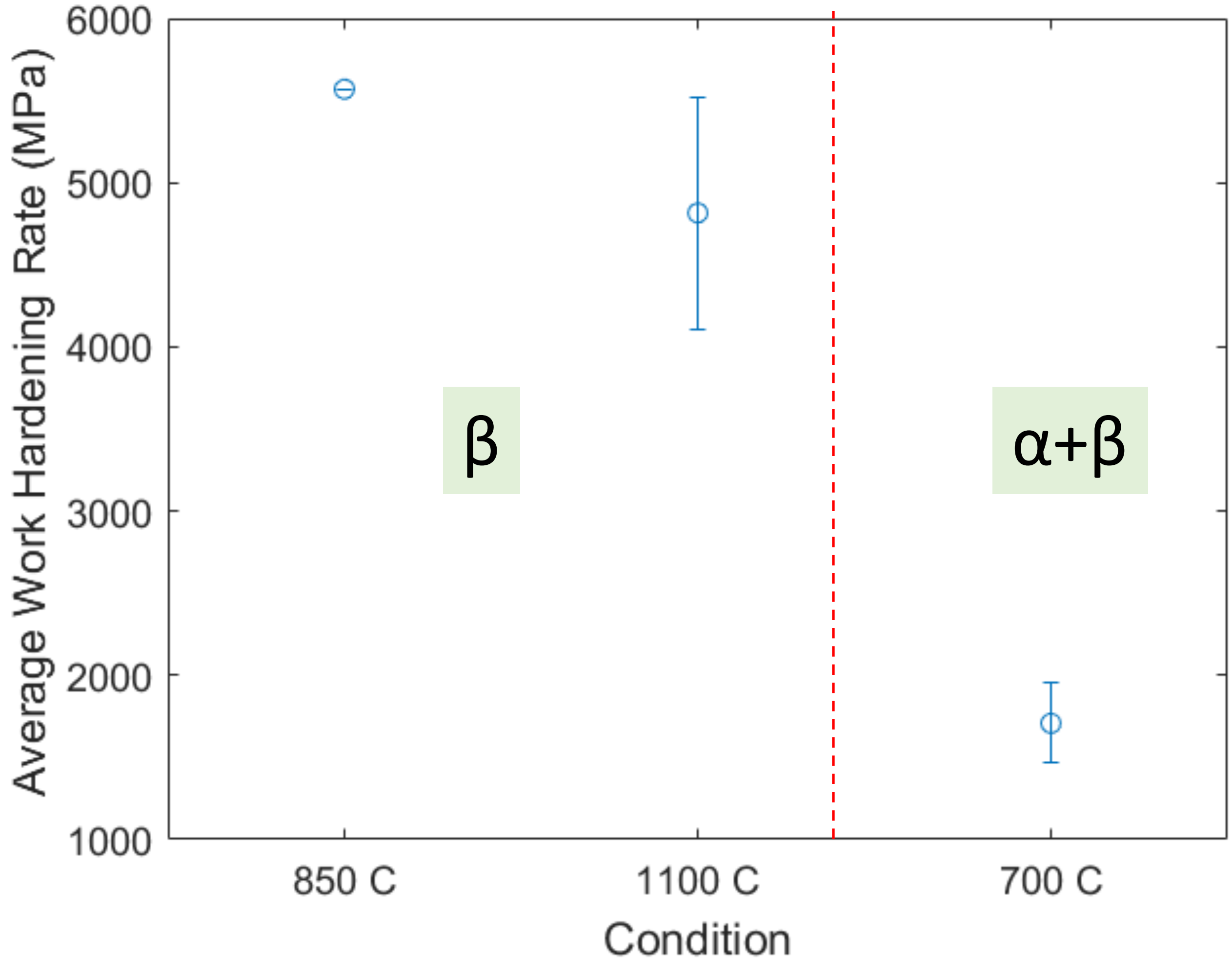


# Trend in Ti-1023 Average Work Hardening Behavior



# Ti-1023 Summary

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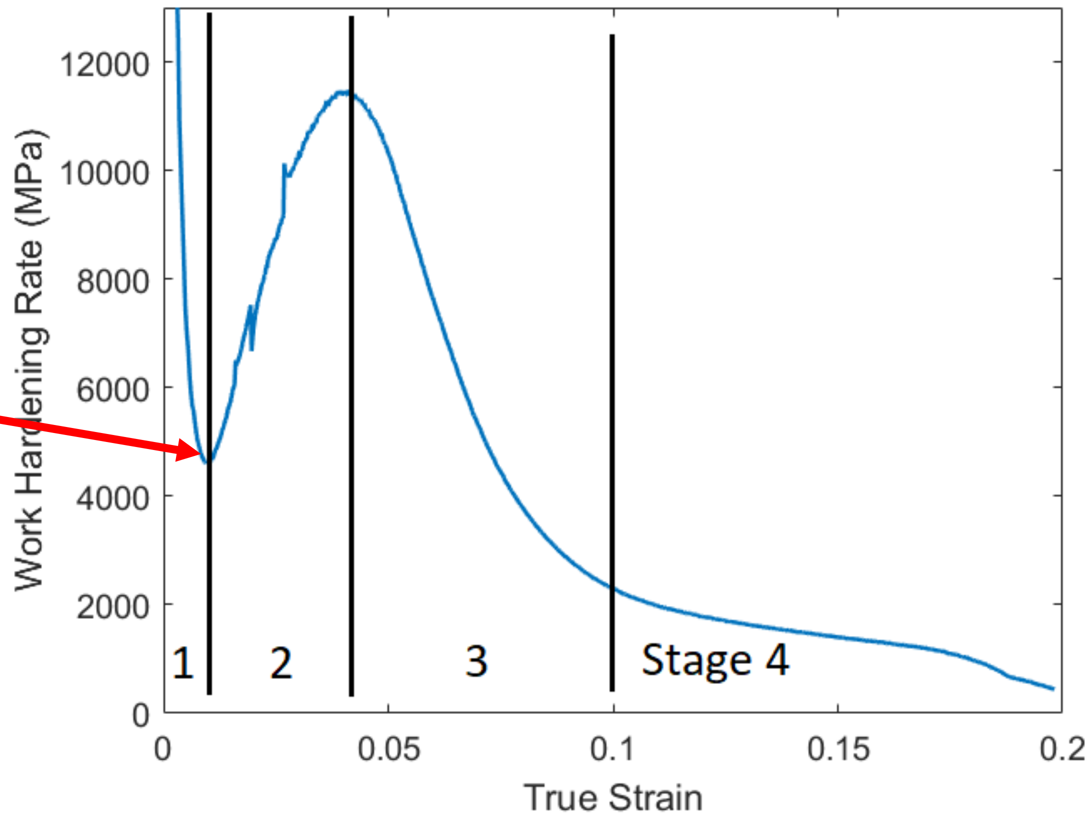
- Compression study shows:
  - Work hardening peak tends to be broader as solution temperature increases
  - Yield stress dependence on solution temperature exhibits a large spread
- Initial tensile testing shows:
  - Grain size and beta chemical stability are controlling factors for work hardening behavior
  - TRIP/TWIP occurs even at large grain sizes
  - Yield stress and work hardening trend with grain size and chemical stability are clear in tension

# Stages of Work Hardening in Ti-1023

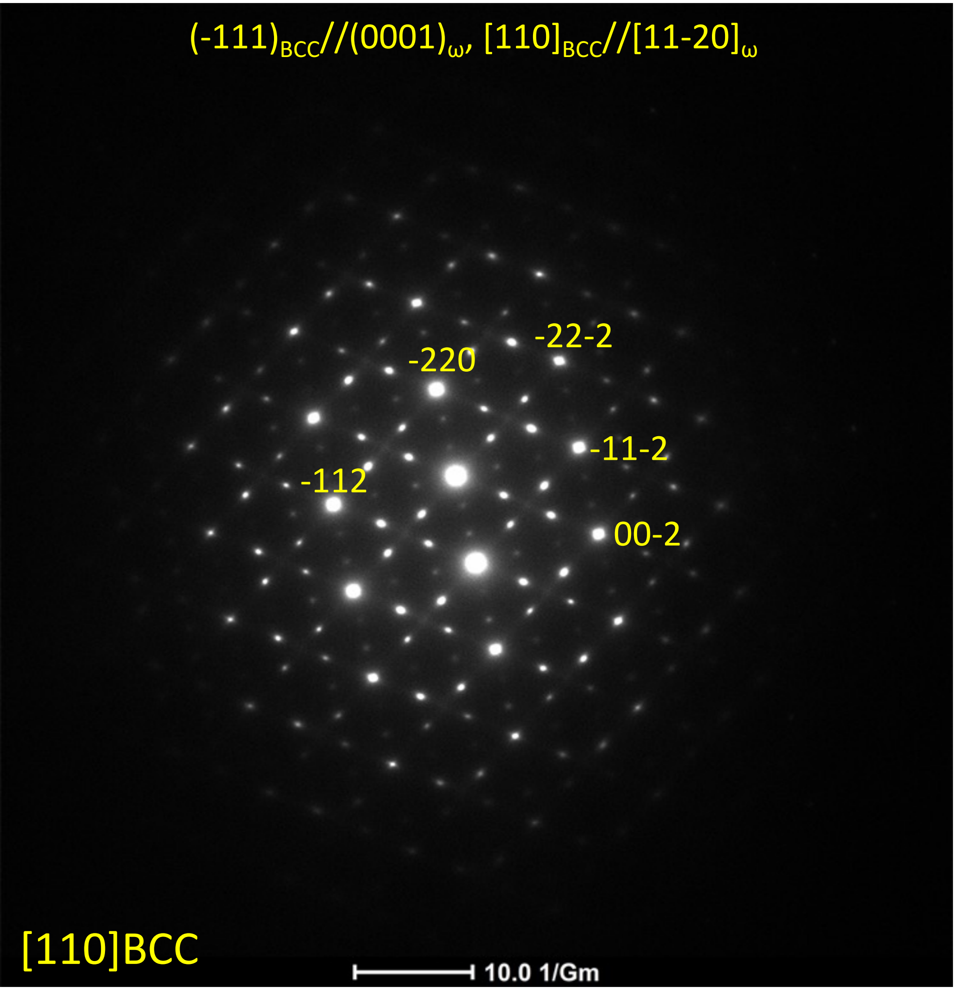
TEM characterization of Interrupted tensile tests :

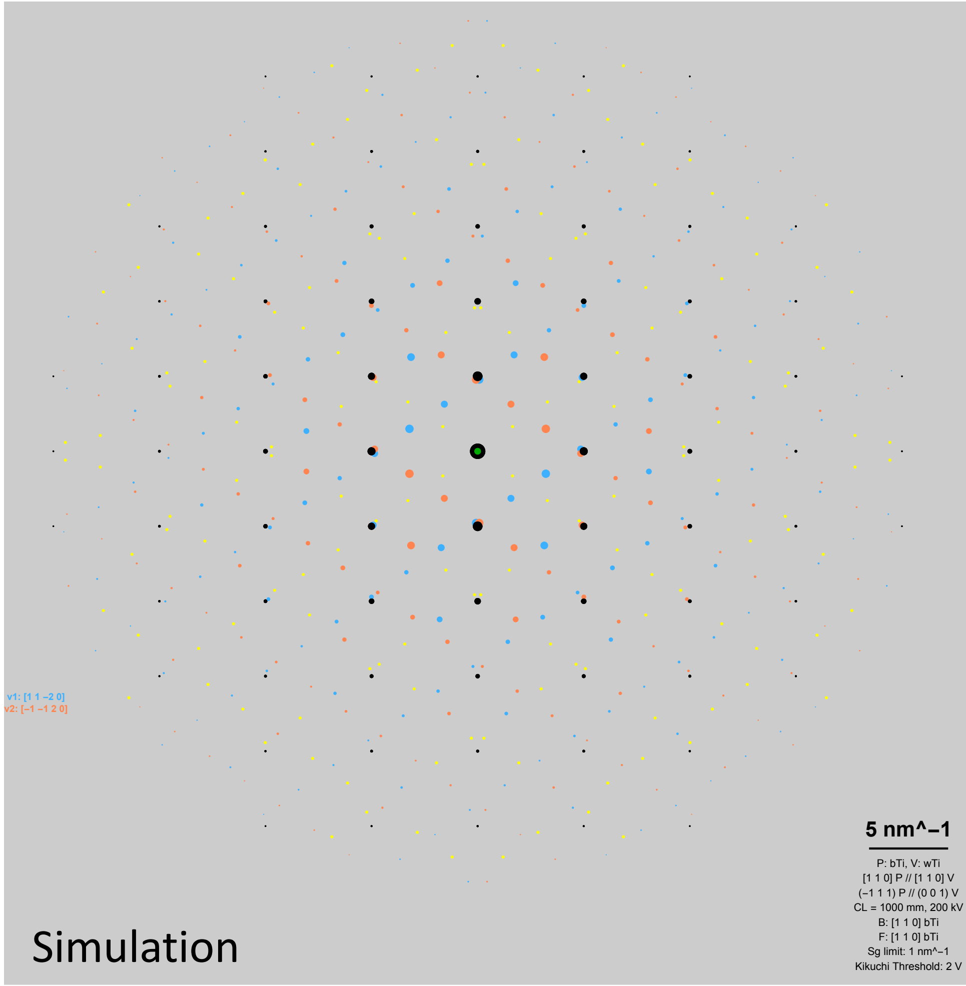
- ✓ 0.5% plastic strain
- 1.7% plastic strain



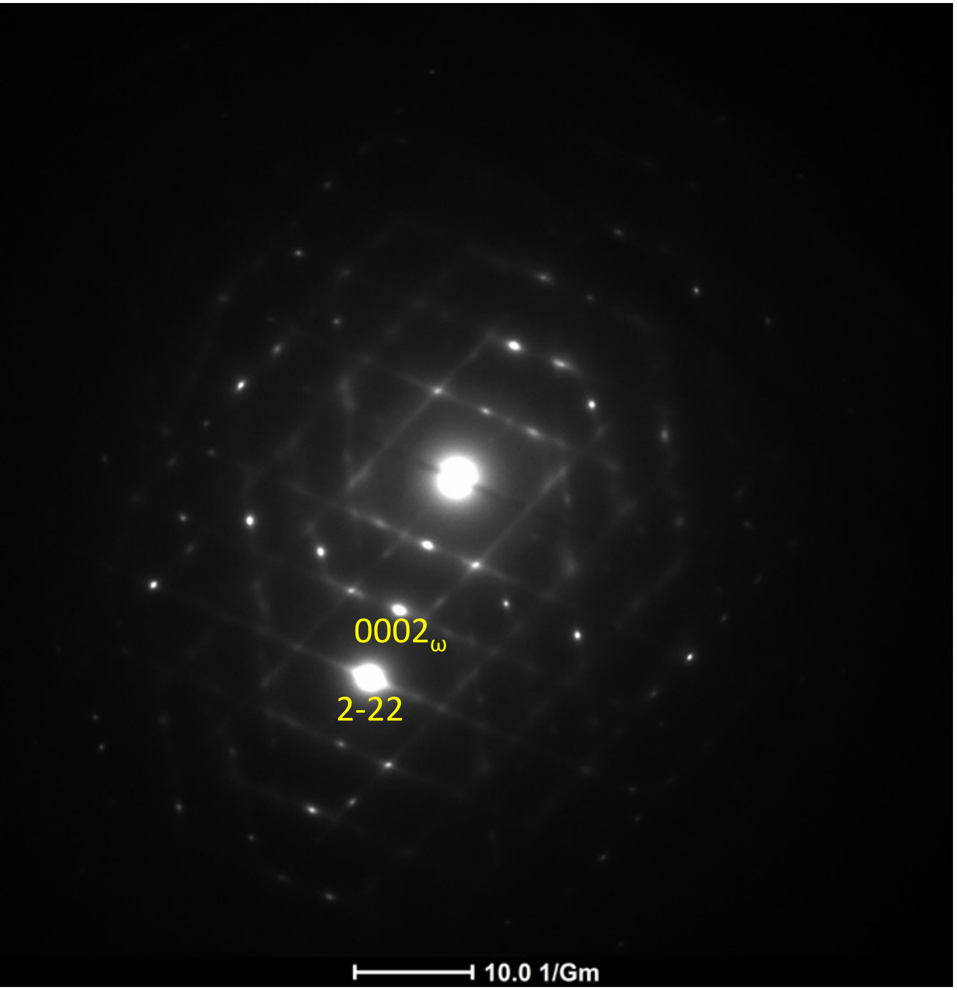
# Undeformed Ti-1023

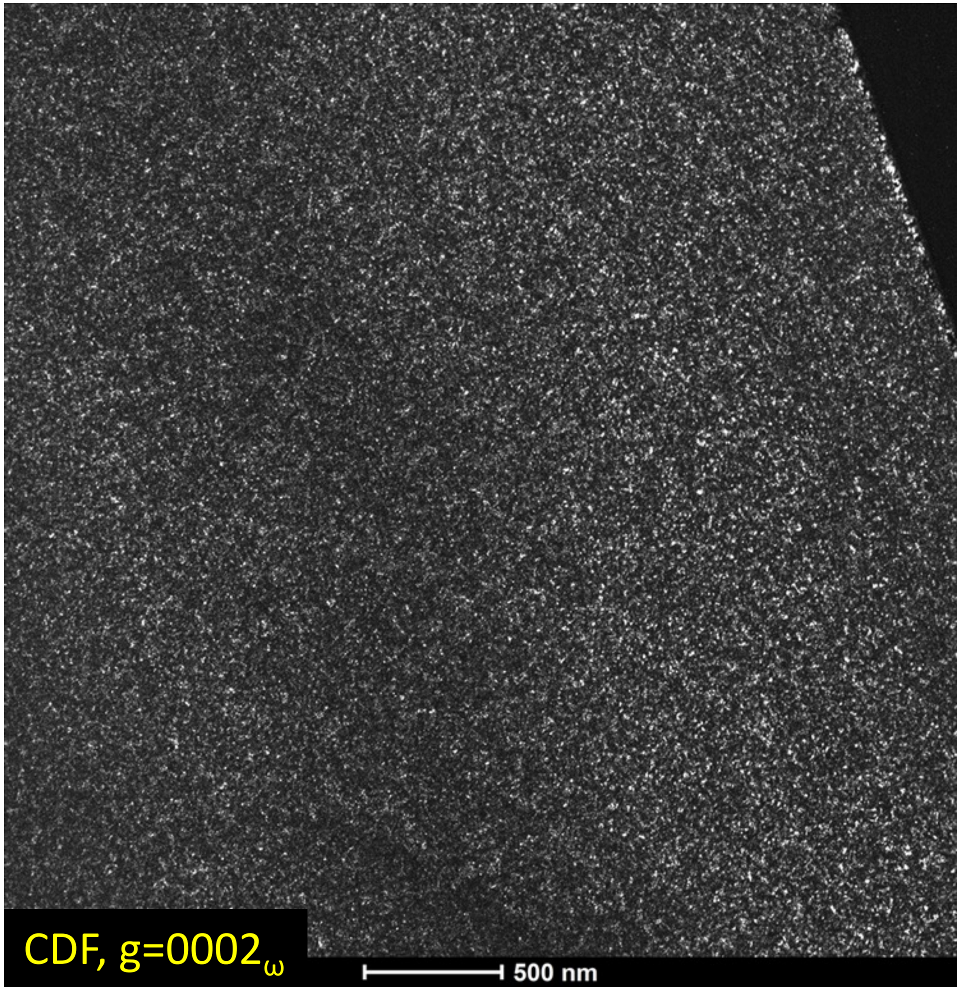
1h@850°C, small grain size



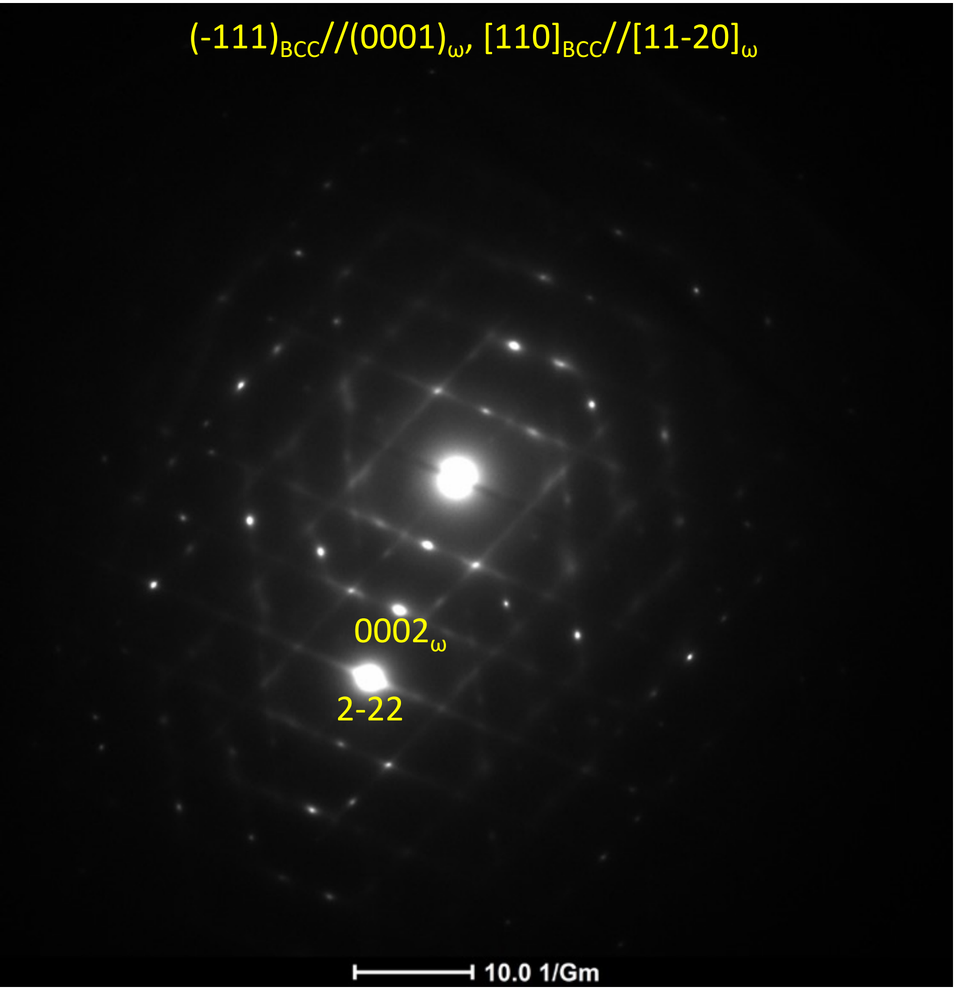



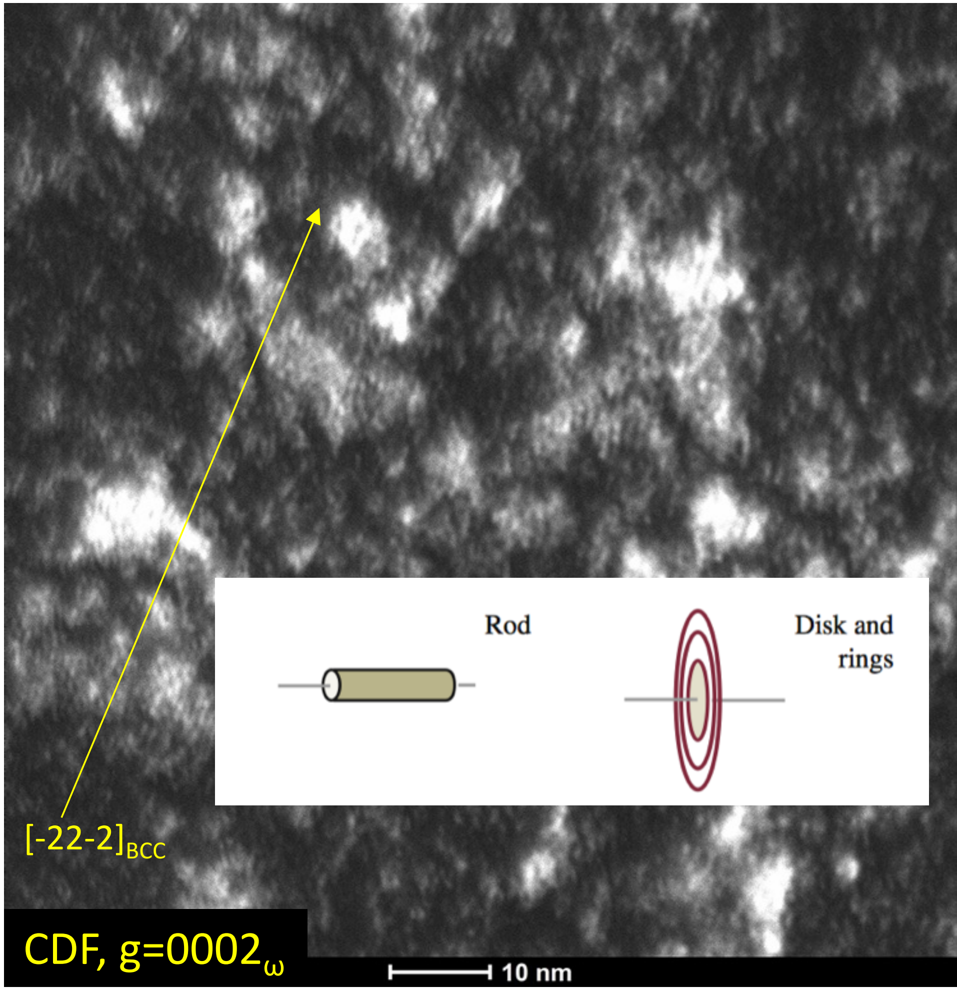
# Distribution of Athermal ω Phase in Ti-1023



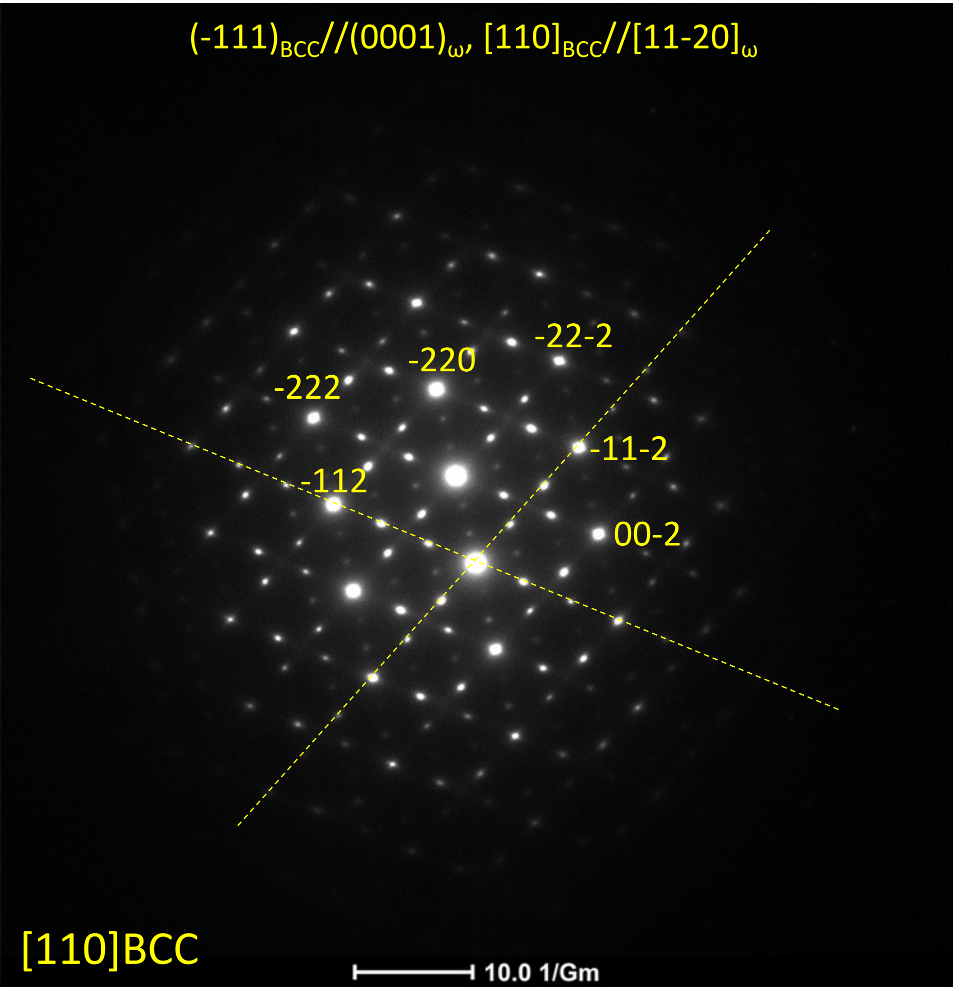



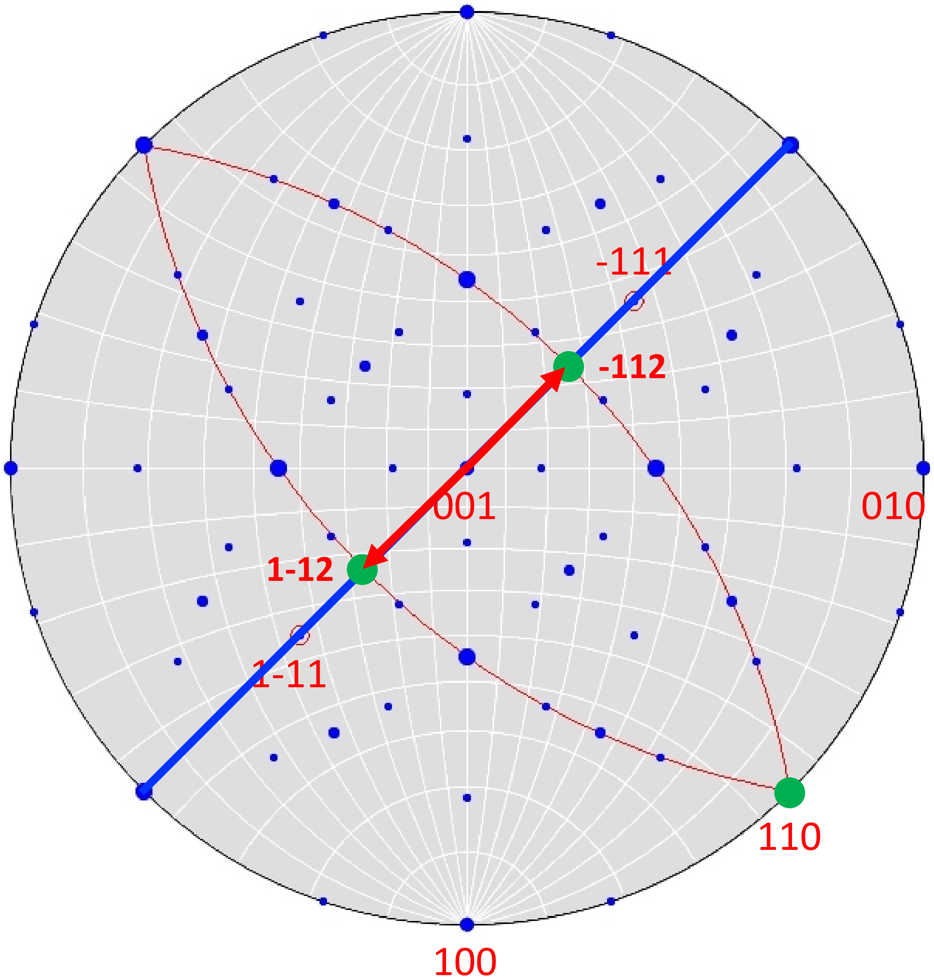
# Morphology of Athermal ω Phase in Ti-1023



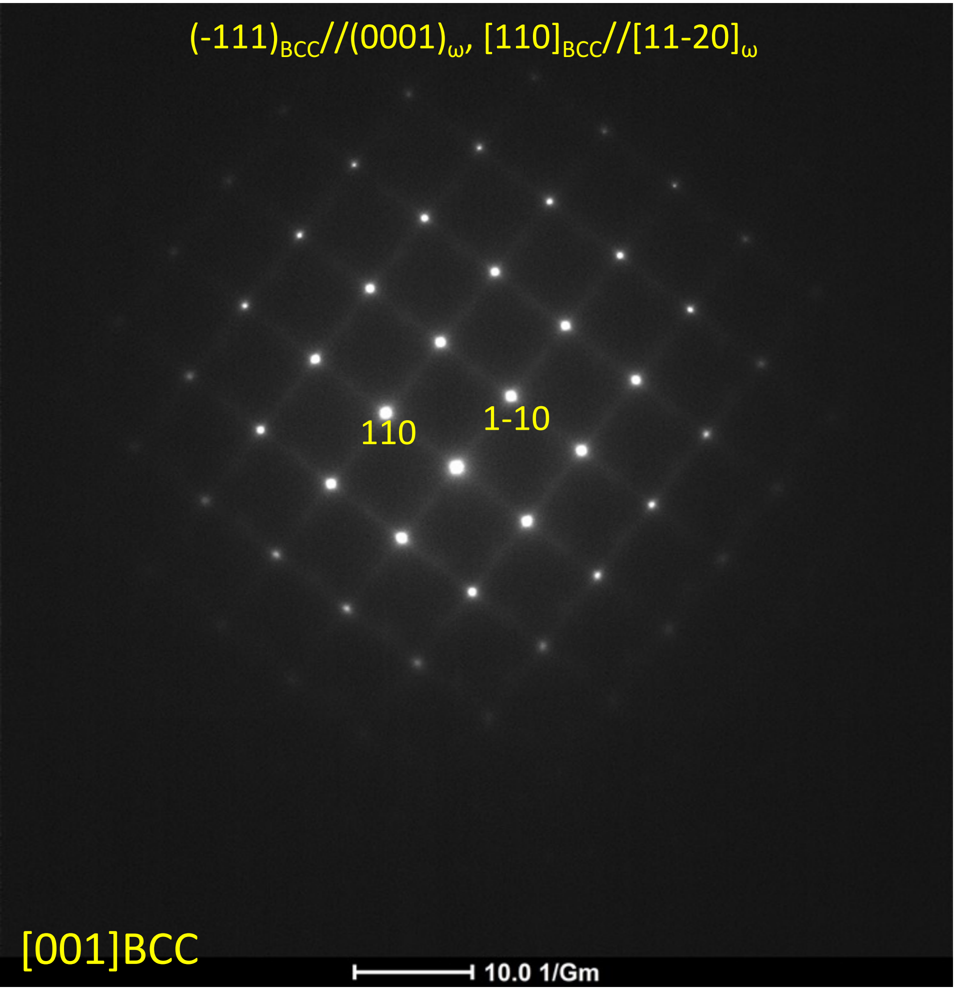



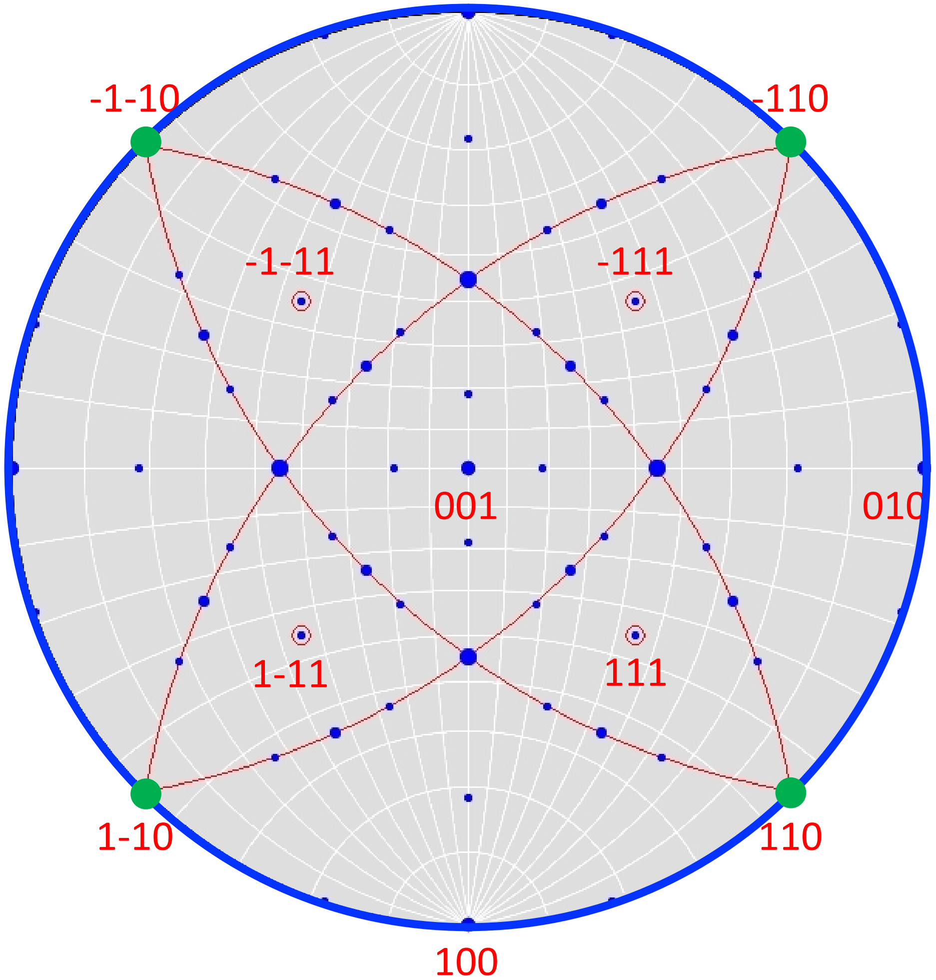
# Streaking in Diffraction Pattern



# Streaking in Diffraction Pattern

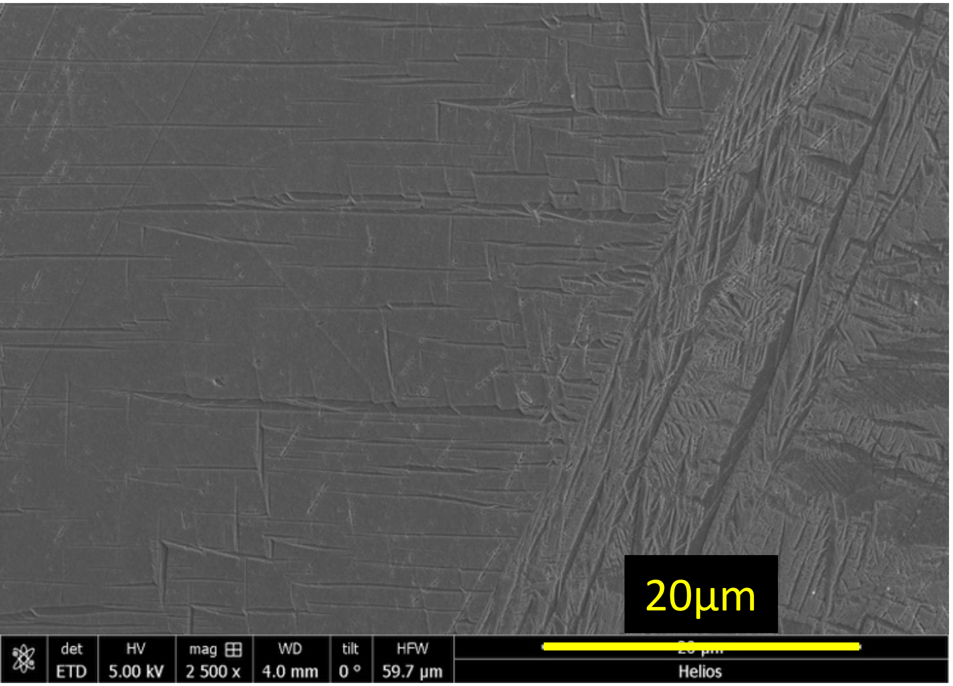


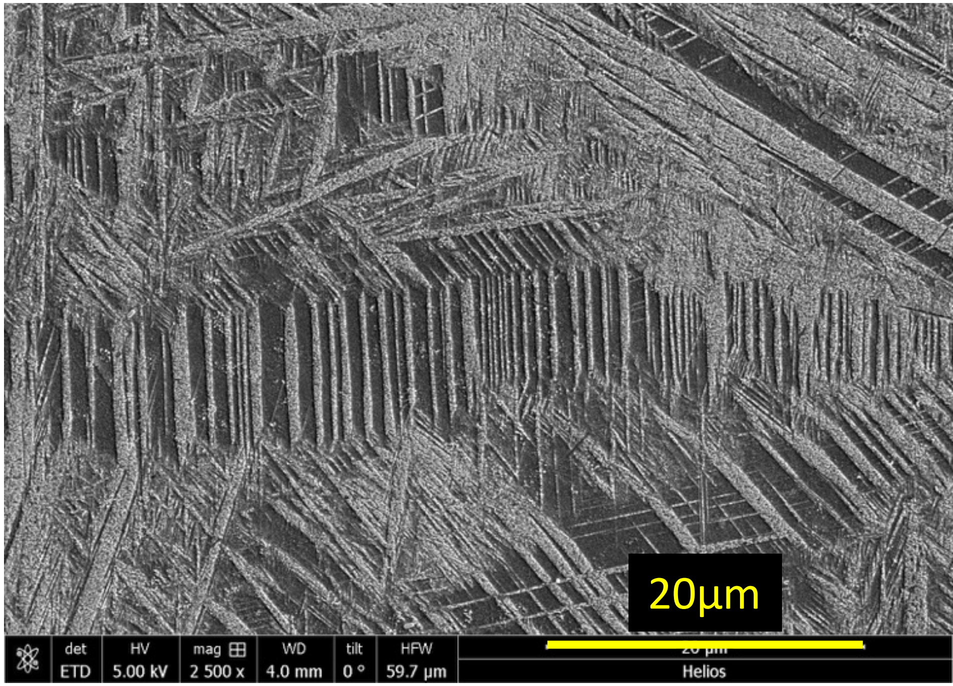
# Deformed Ti-1023 (Tension)

1h@850°C, small grain size

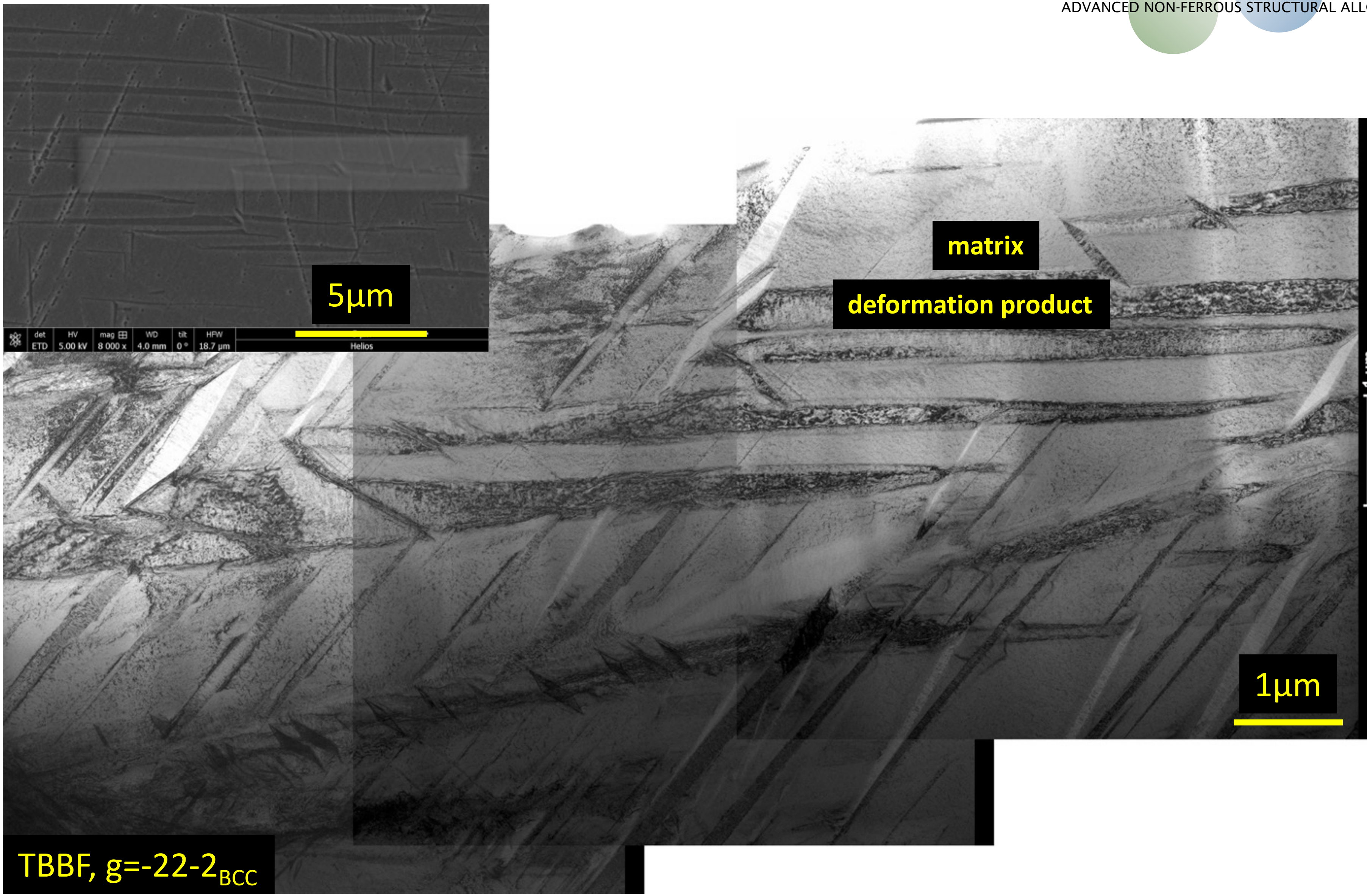
0.5%




1.7%



# 0.5% Deformed Ti-1023 (Tension)



# Matrix: Undeformed vs 0.5% Deformed

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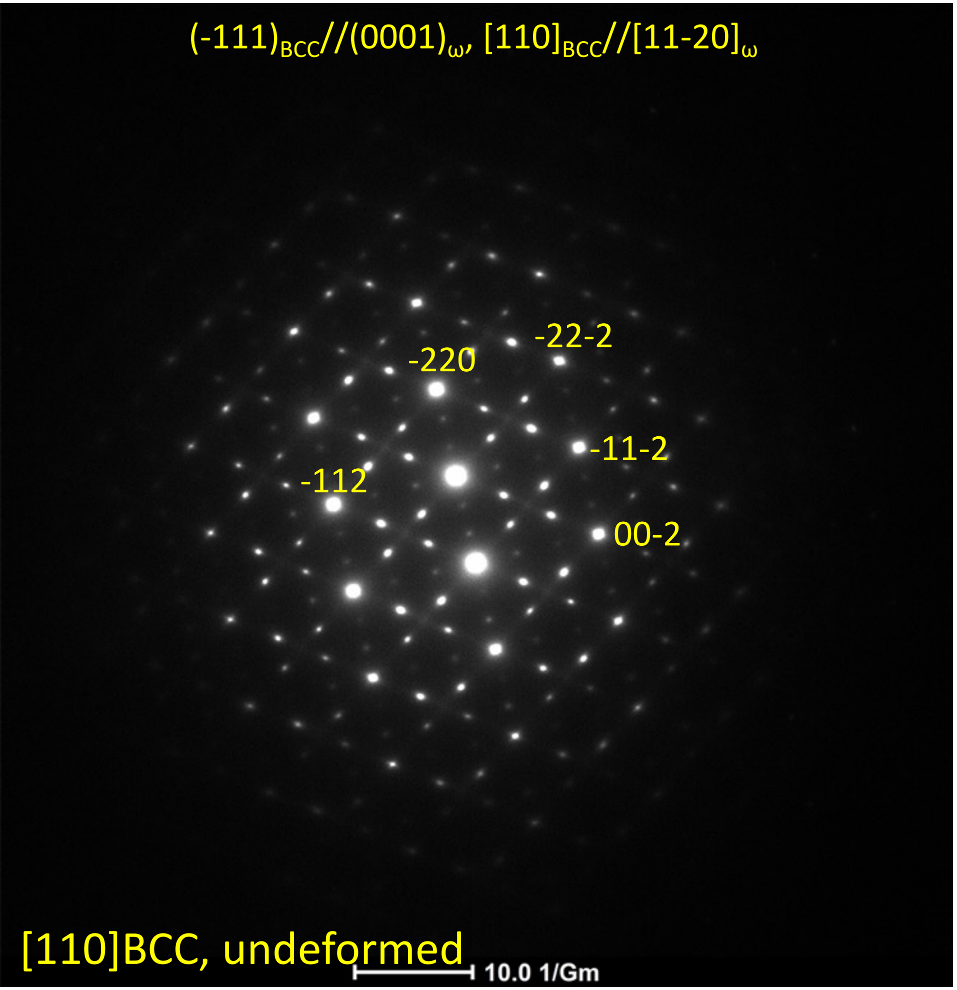
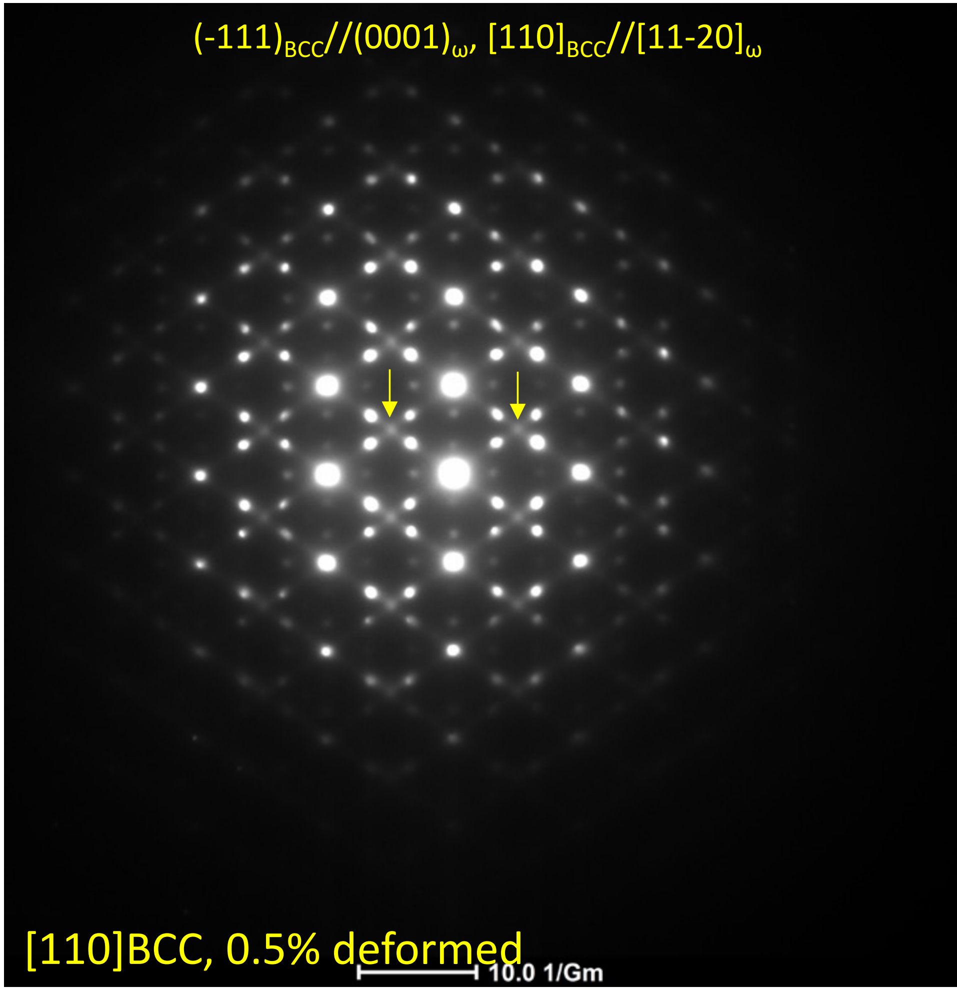
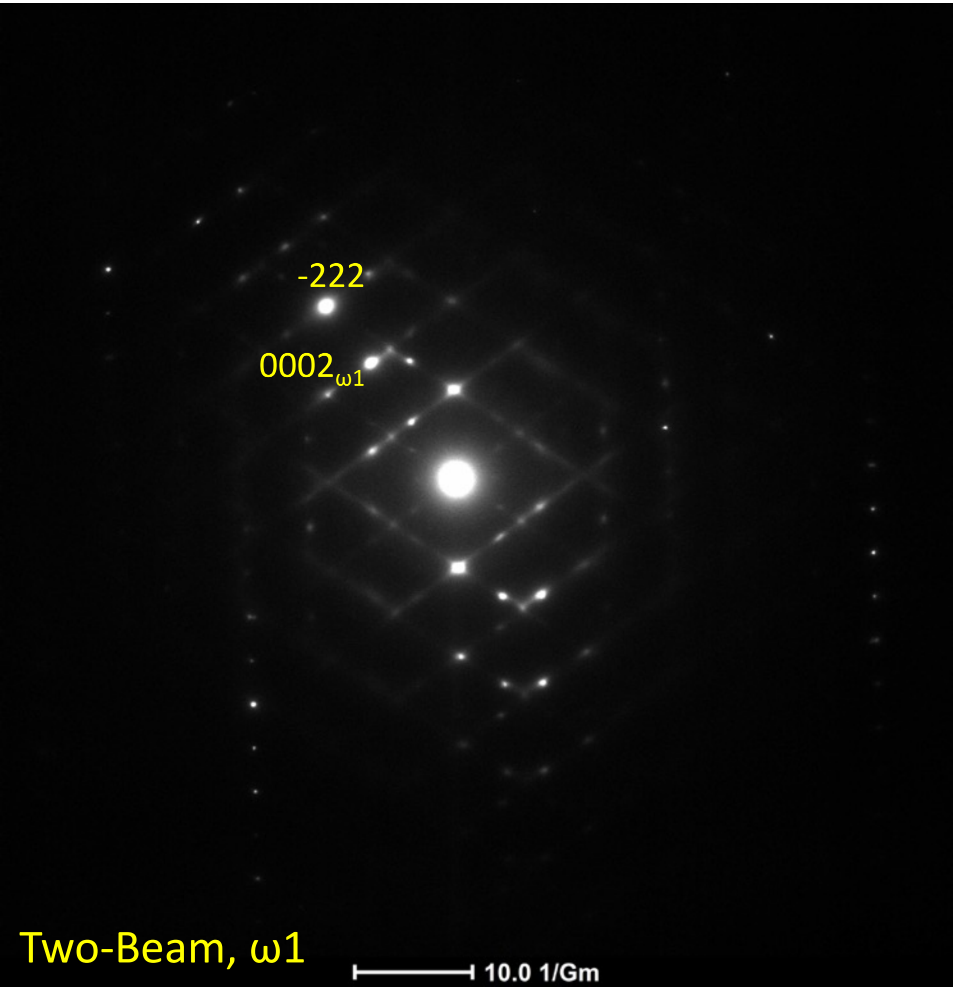
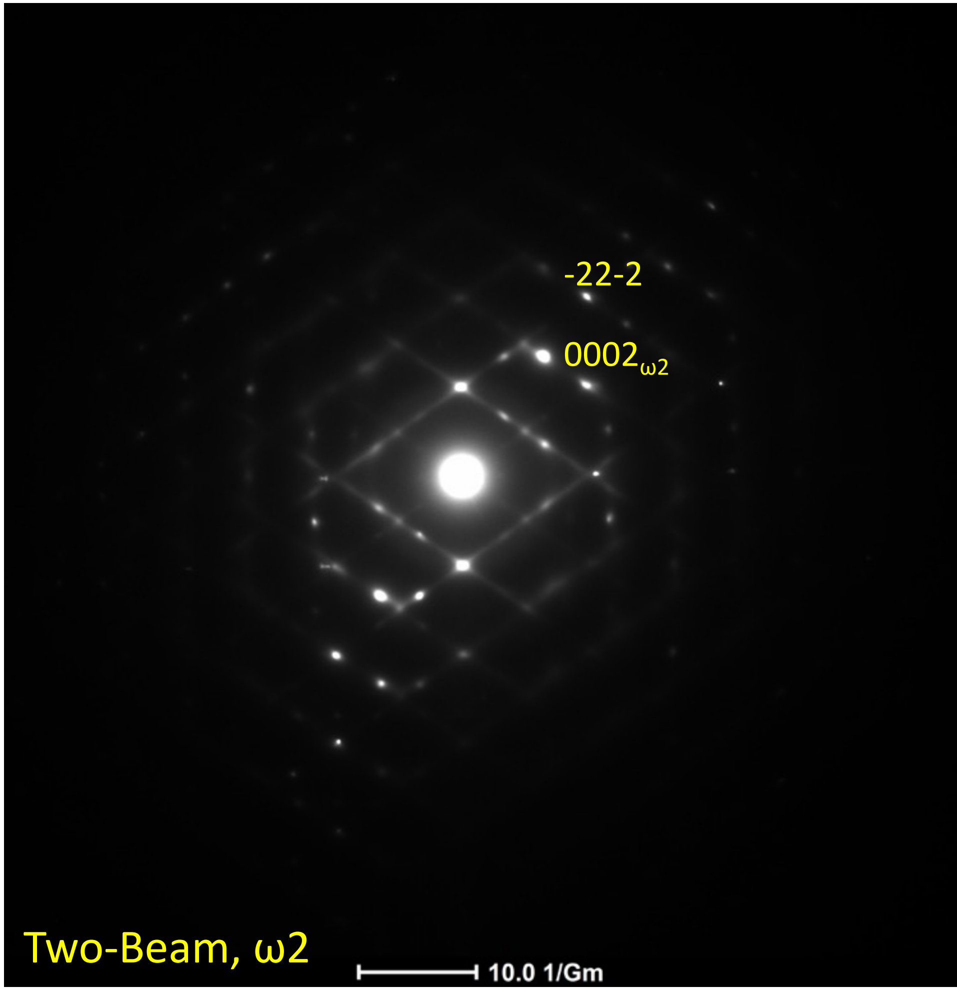


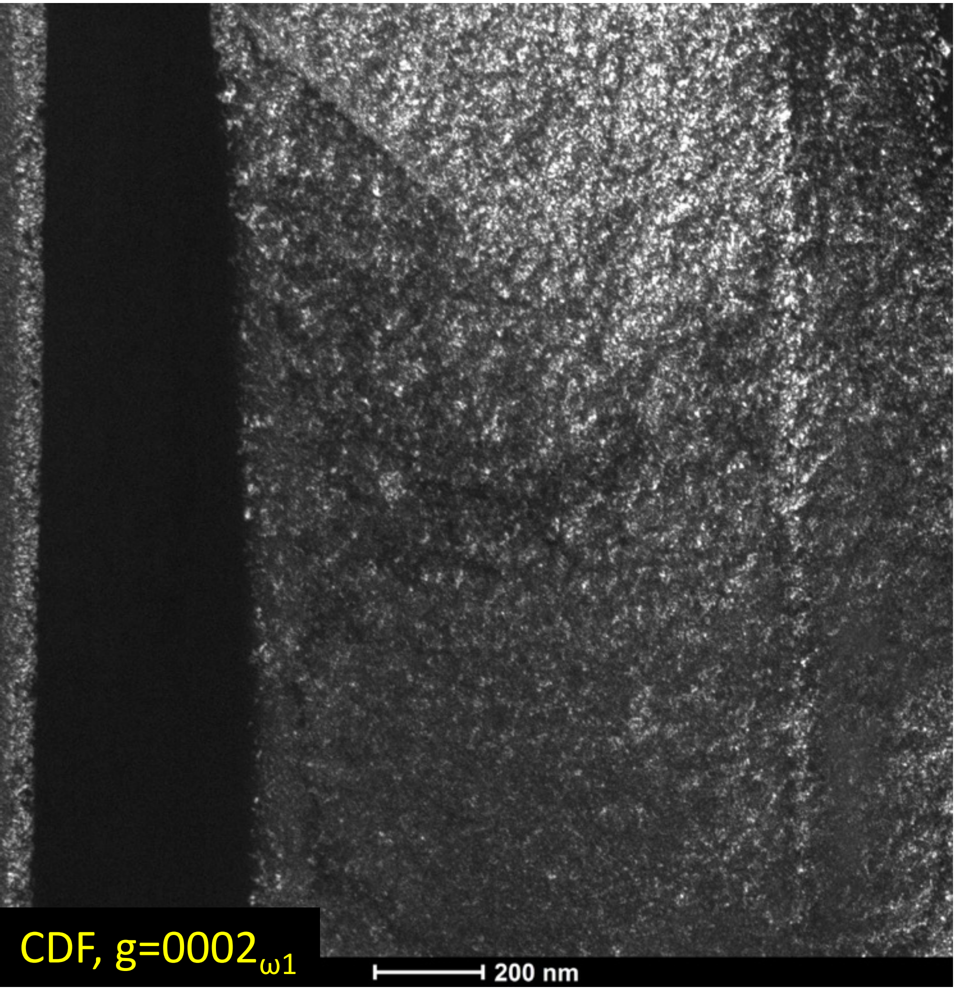



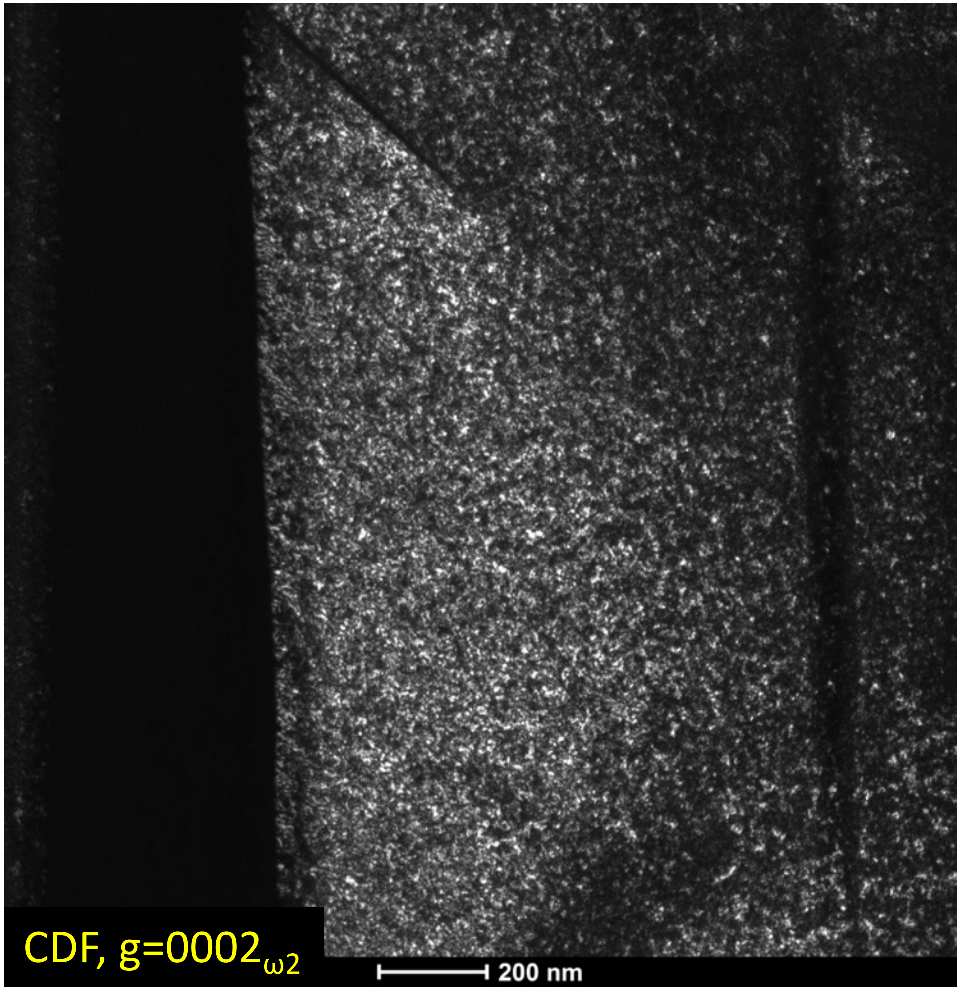
# Two Variants of Athermal ω in Ti-1023



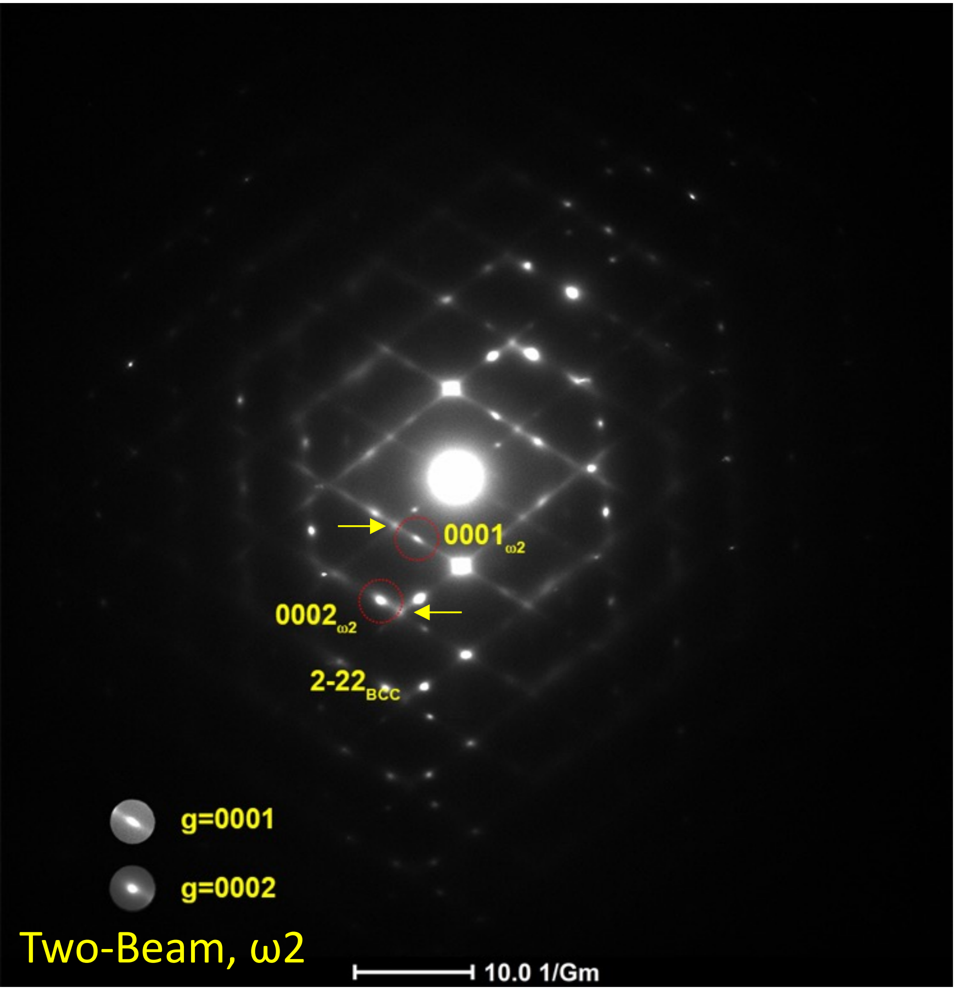



# Distribution of Athermal ω in 0.5% Deformed Ti-1023



# No ω Phase Appeared in CDFs



# CDFs using g=$0002_{\omega 1/2}$

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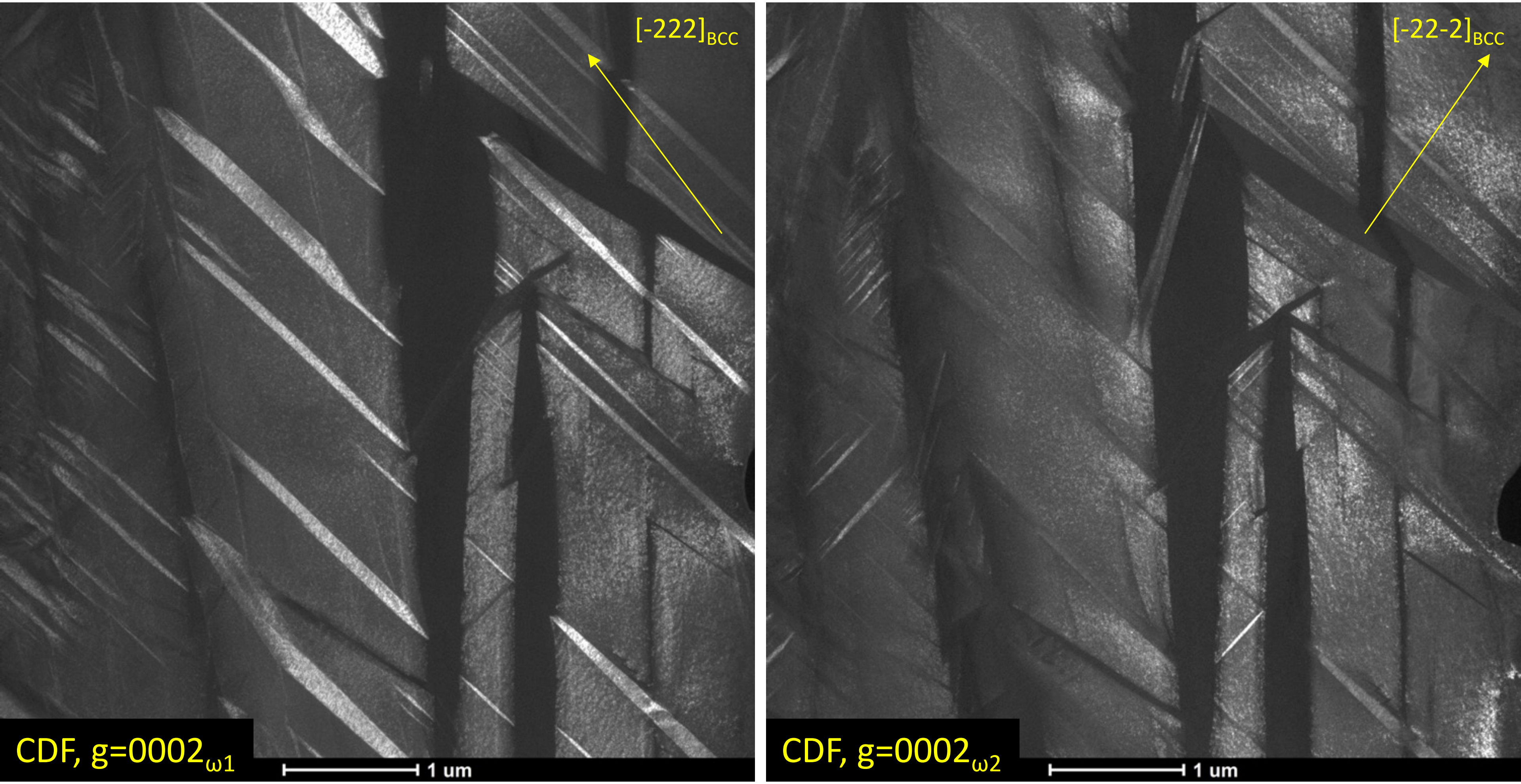

# Nature of Martensite: α' or α''?

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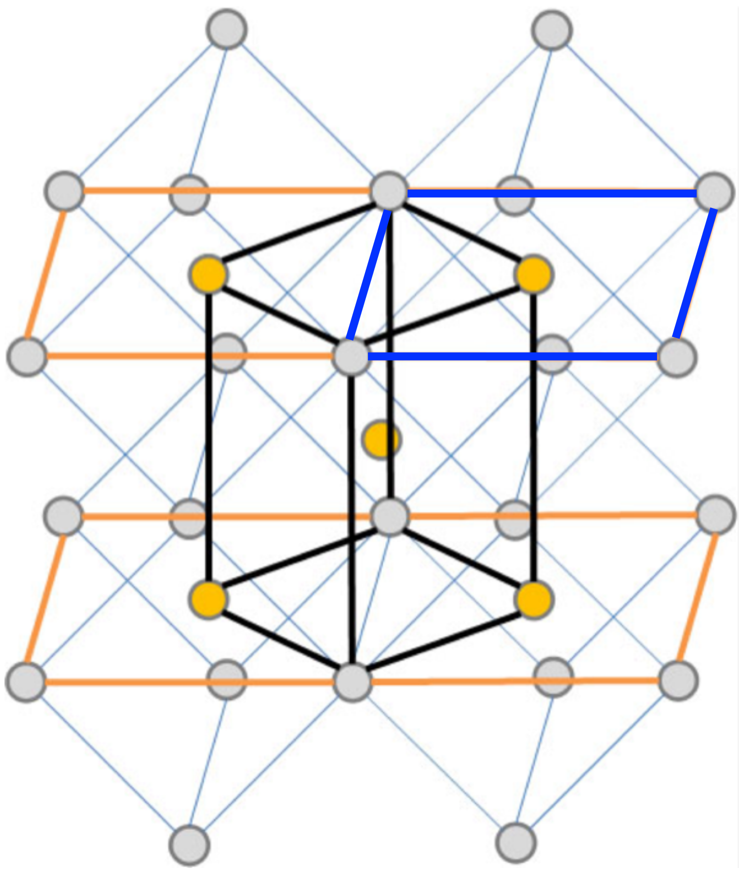
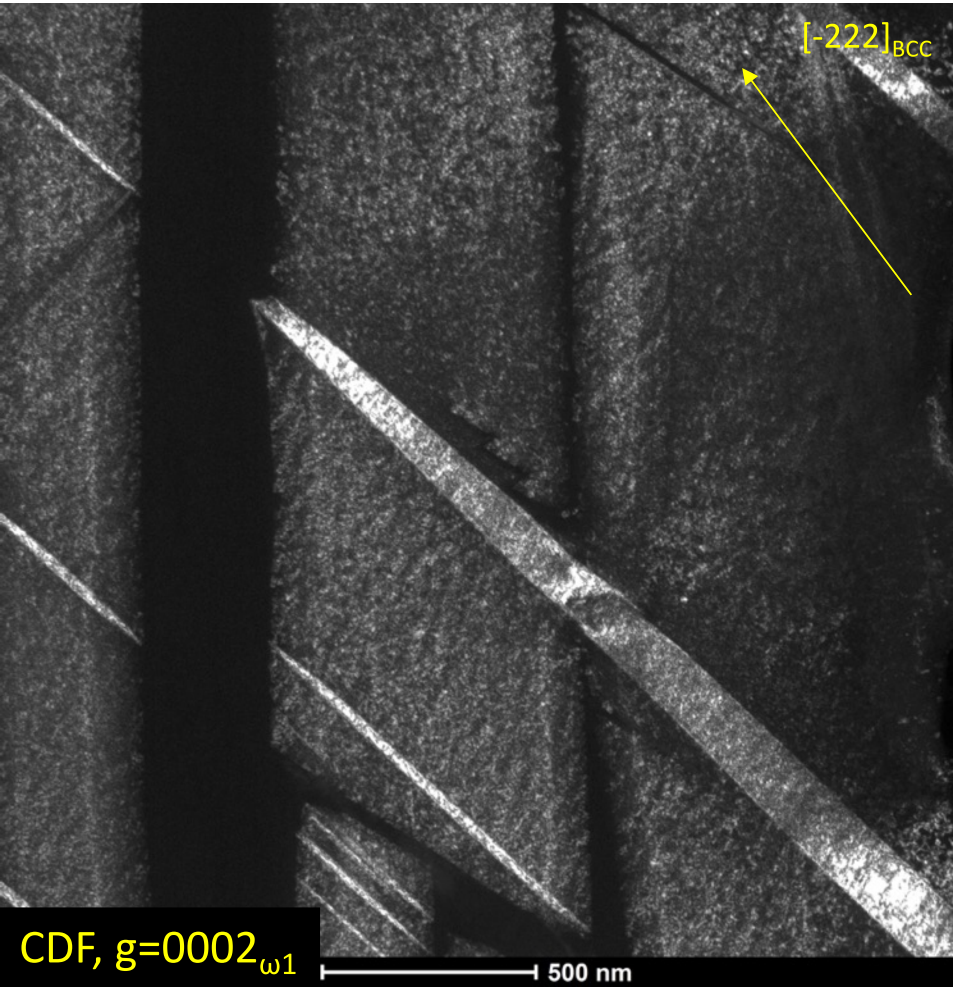
- $(110)_{BCC}//(0001)_{\alpha/\alpha'}$, $[1\text{-}11]_{BCC}//[2\text{-}1\text{-}10]_{\alpha/\alpha'}$ (Burgers OR)
- $(1\text{-}10)_{BCC}//(010)_{\alpha''}$, $[110]_{BCC}//[001]_{\alpha''}$
- Duerig* reported **α''** in heat-treated Ti-10-2-3 alloy.

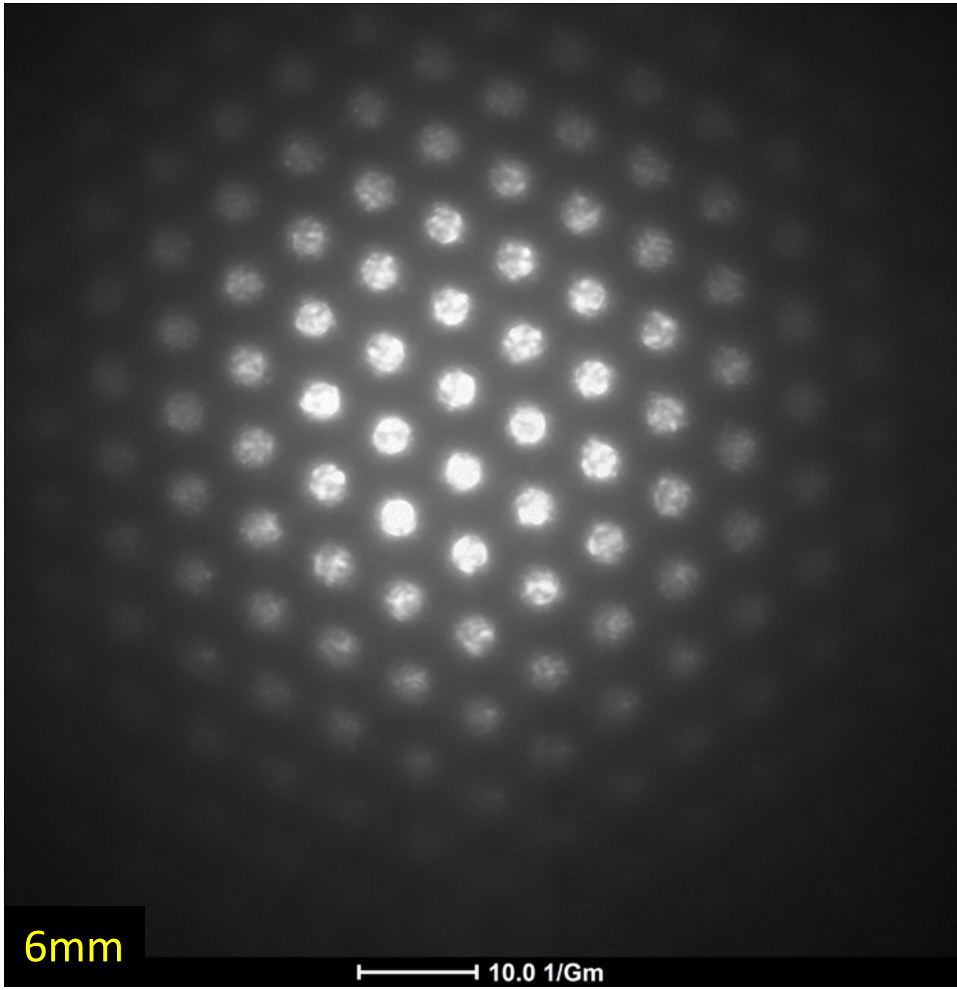


| Zone Axis Correspondence between α' and α'' in $[110]_{BCC}$ | |
|---|---|
| α' (hex) | α'' (ort) |
| v1, [0001] | v1, [001] |
| v2, [000-1] | |
| v3, [10-10] | v2, [010] |
| v4, [-1010] | |
| v5, [5-4-13] or [2-11]<br>v8, [-541-3] or [-21-1]<br>v10, [5-4-1-3] or [2-1-1]<br>v12, [-5413] or [-211] | v3, [211]<br>v4, [-211]<br>v5, [2-11]<br>v6, [-21-1] |

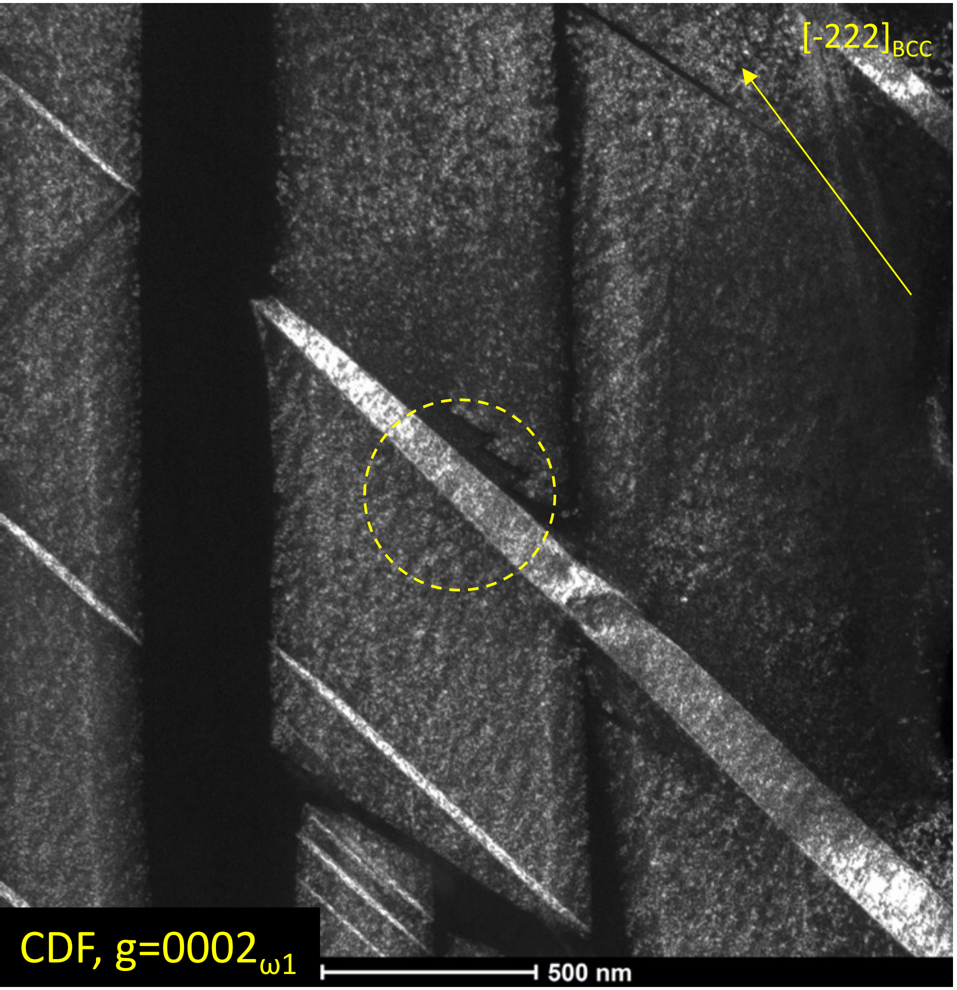
[*] T.W. Duerig, et al., Acta Metall. 30 (1982) 2161–2172.

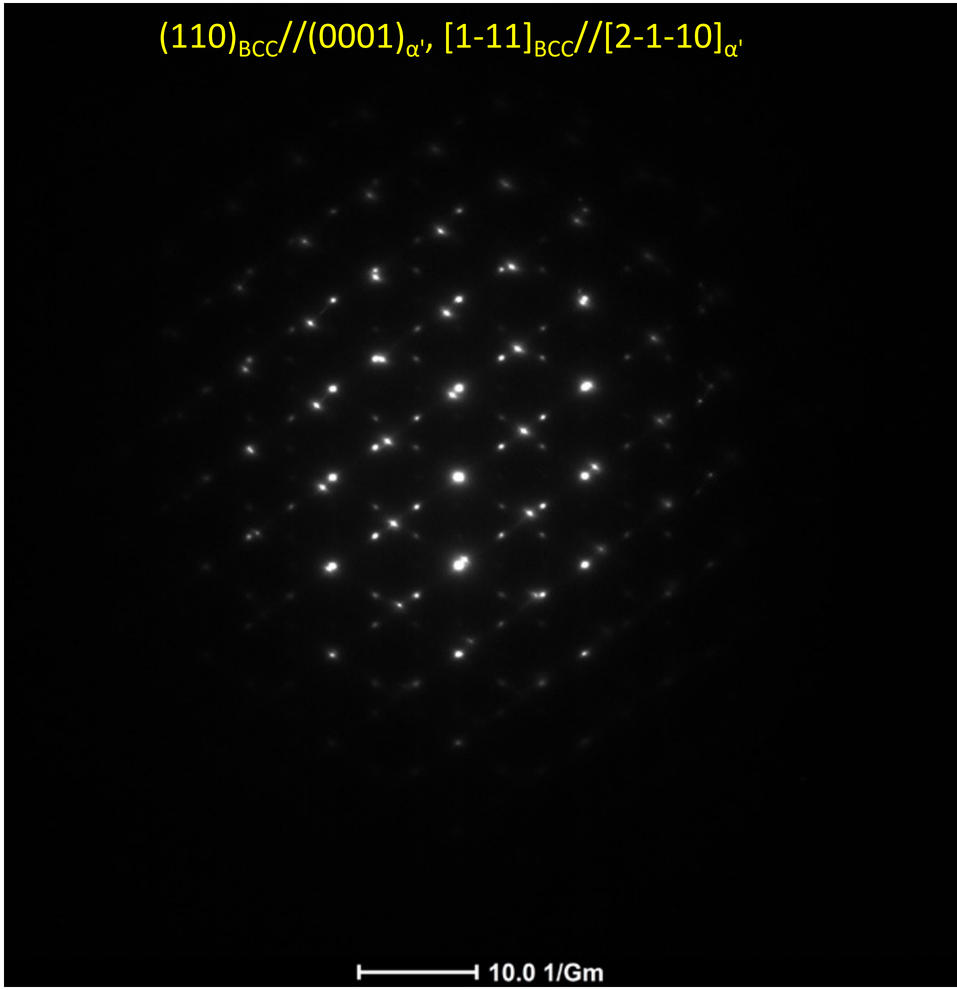
# Evidence 1: $[0001]_{\alpha'}$ or $[001]_{\alpha''}$



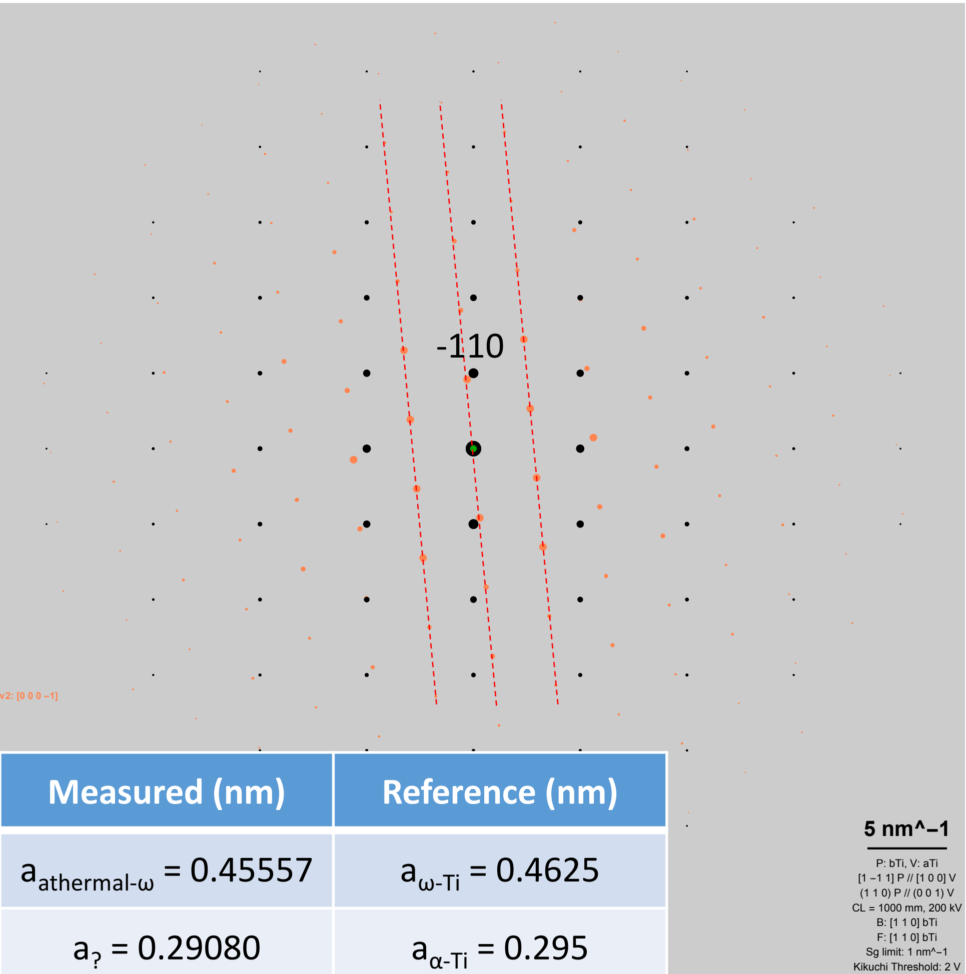



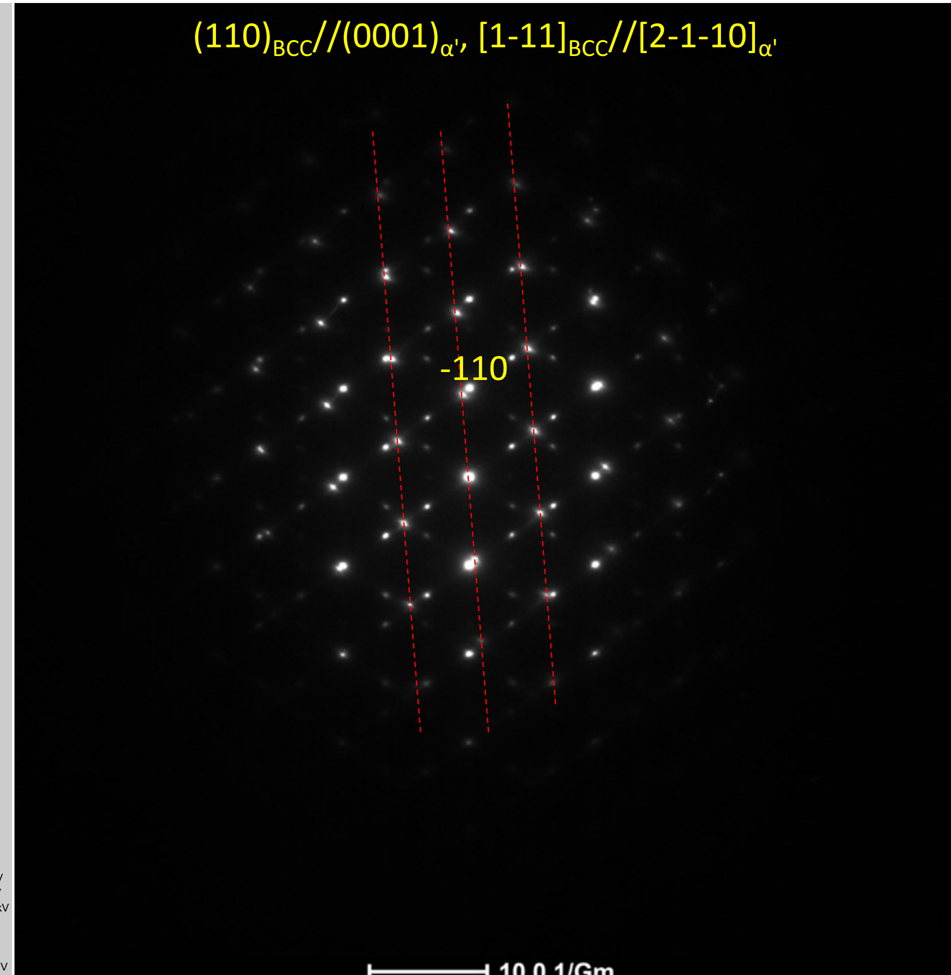
# Evidence 1: $[0001]_{\alpha'}$ or $[001]_{\alpha''}$



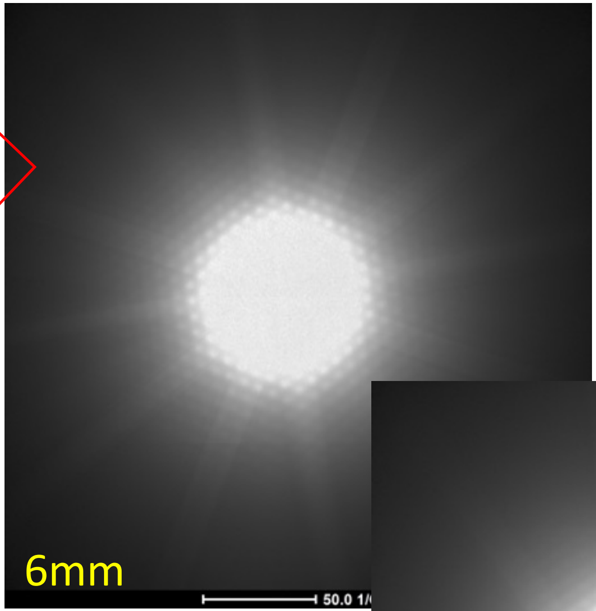
# Evidence 1: $[0001]_{\alpha'}$ or $[001]_{\alpha''}$

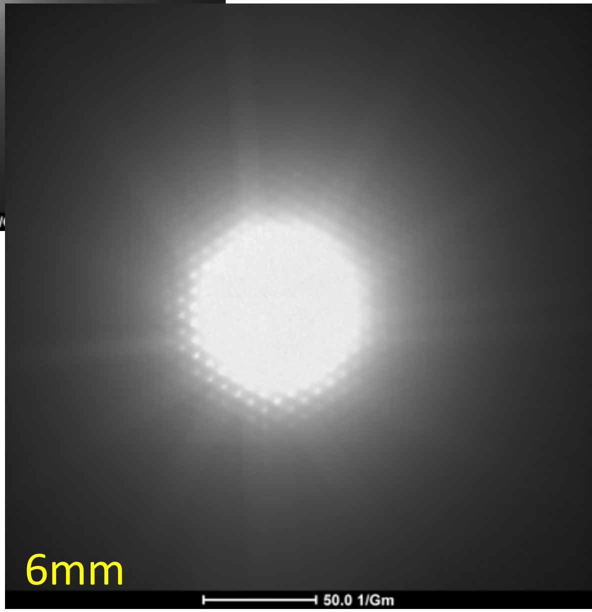


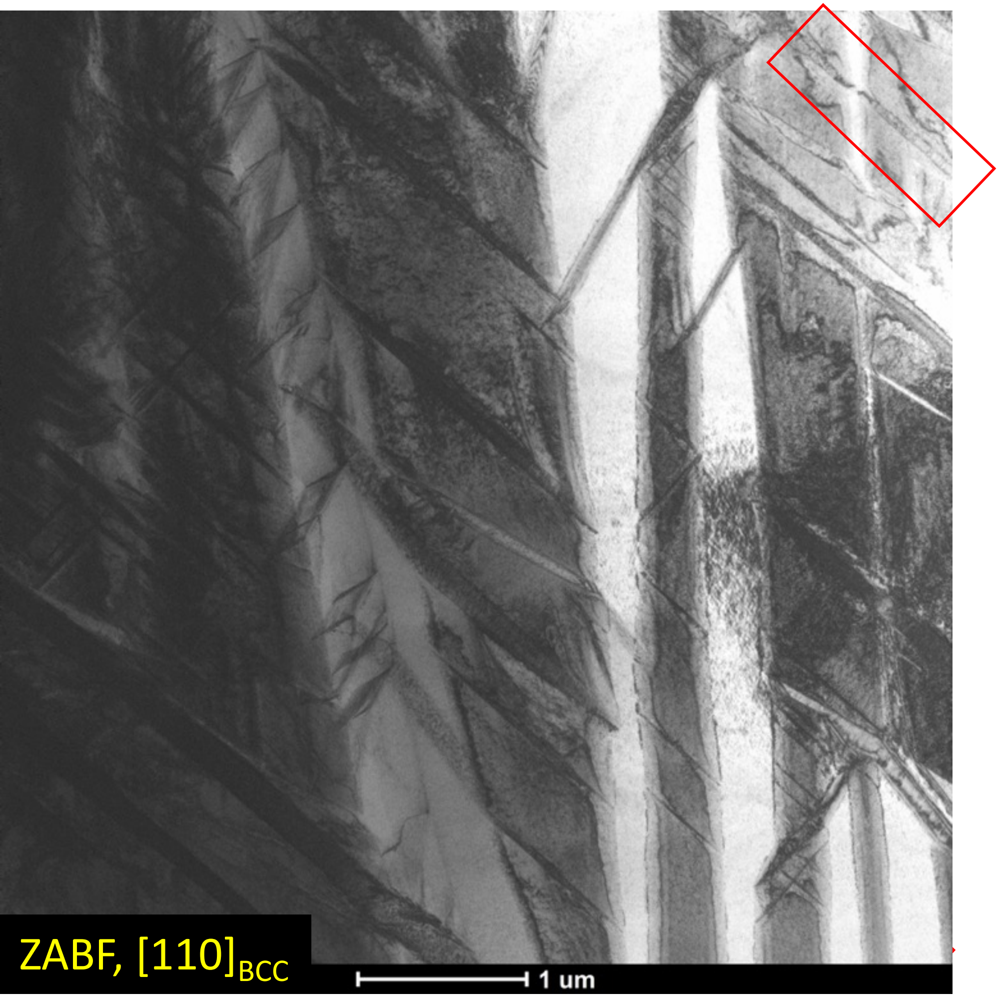

| Measured (nm) | Reference (nm) |
|---|---|
| $a_{athermal\text{-}\omega} = 0.45557$ | $a_{\omega\text{-}Ti} = 0.4625$ |
| $a_{?} = 0.29080$ | $a_{\alpha\text{-}Ti} = 0.295$ |

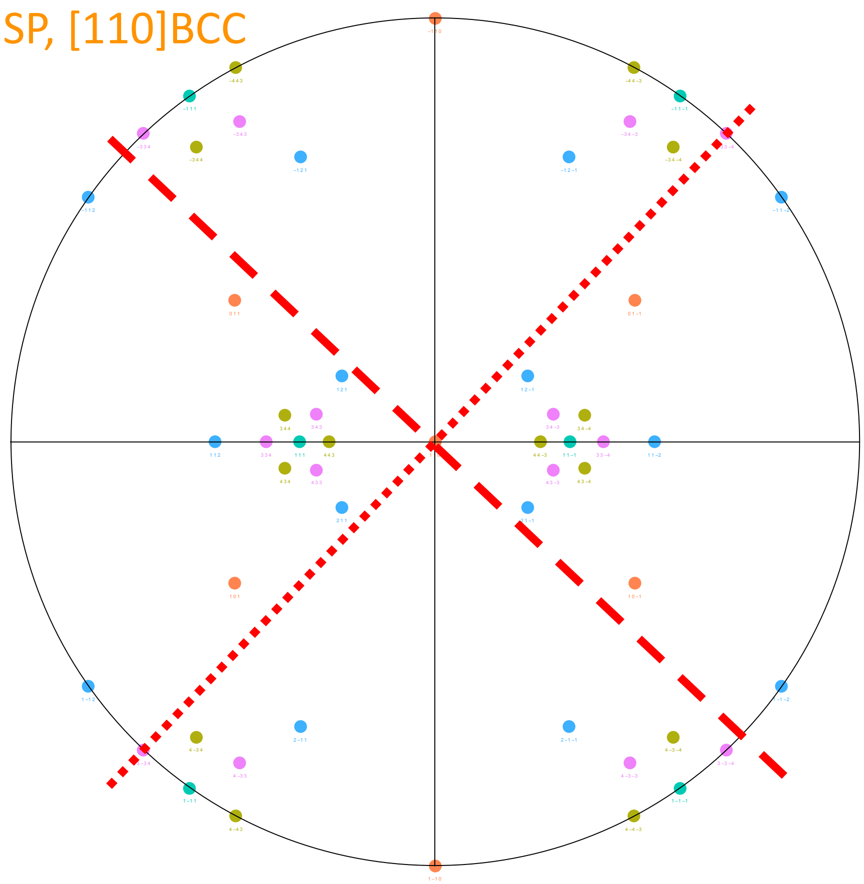
# Evidence 2: Two Variants

# Evidence 3: Habit Plane




{112}, {110}, {111}, {334}, {344}

# Evidence 3: Habit Plane

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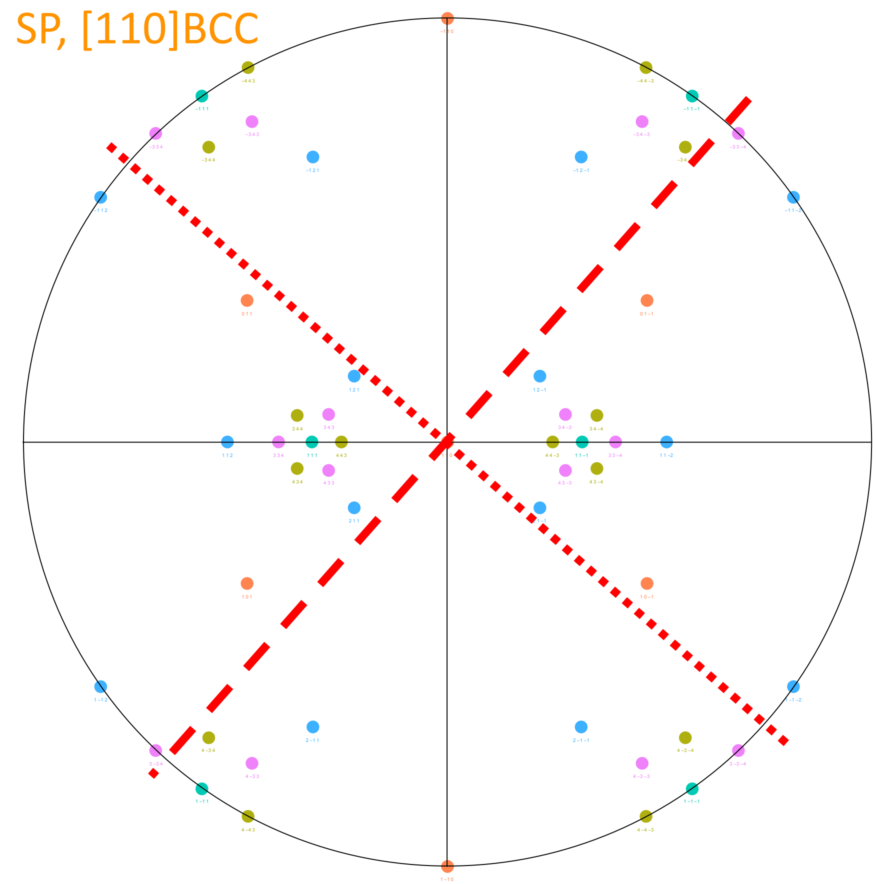
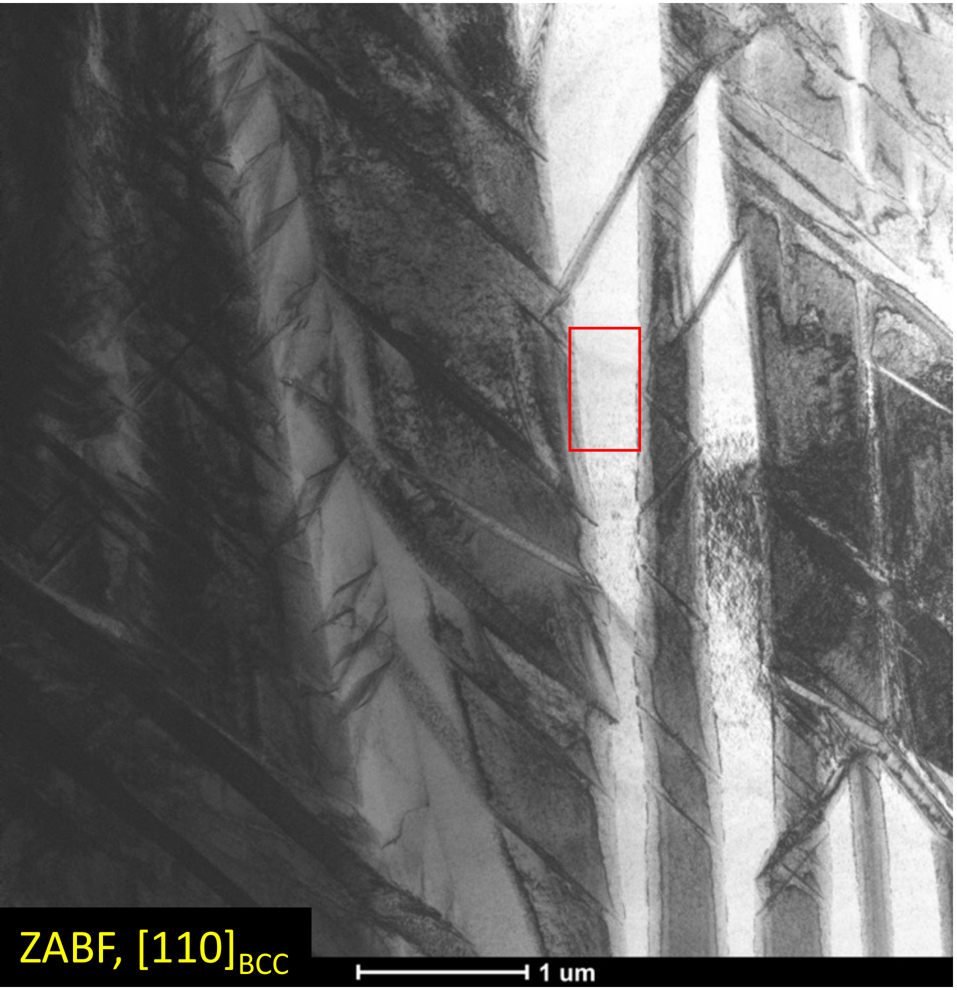
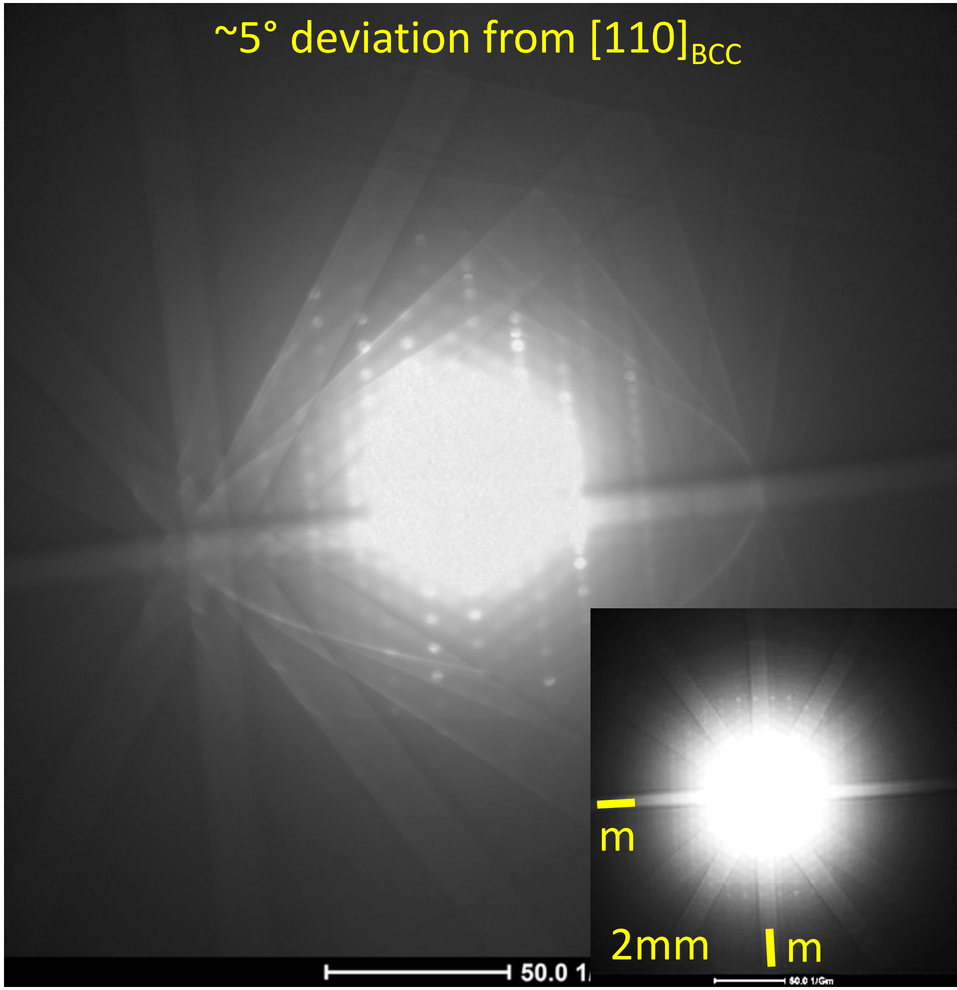



{112}, {110}, {111}, {334}, {344}

# Evidence 4: [10-10]$_{\alpha'}$ or [010]$_{\alpha''}$

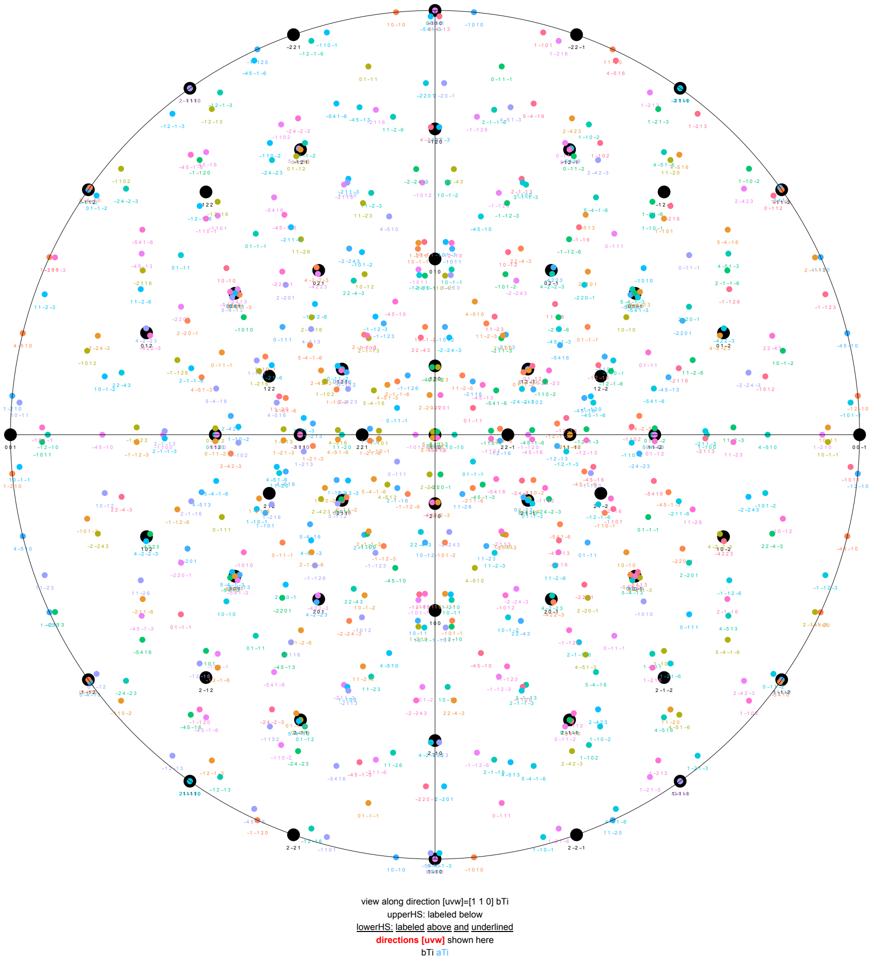


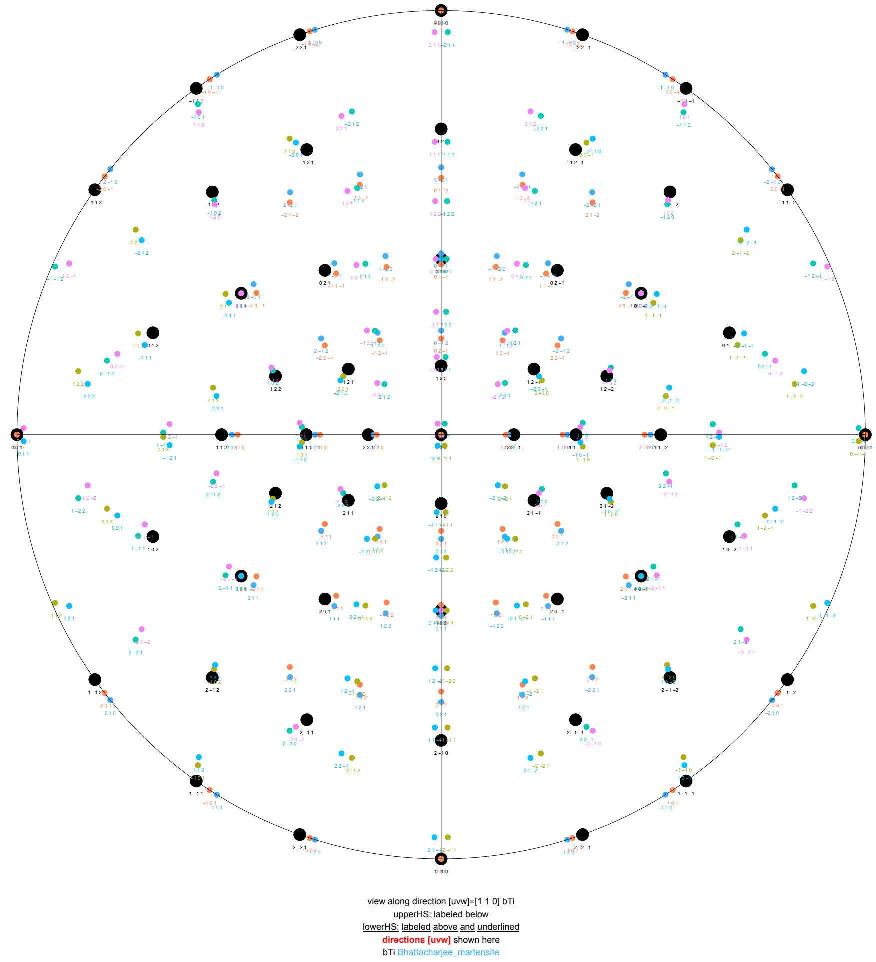
# Evidence 4: [10-10]$_{\alpha'}$ or [010]$_{\alpha''}$

CANFSA
Center for
ADVANCED NON-FERROUS STRUCTURAL ALLOYS

SP, [110]$_{BCC}$, 12 α' variants

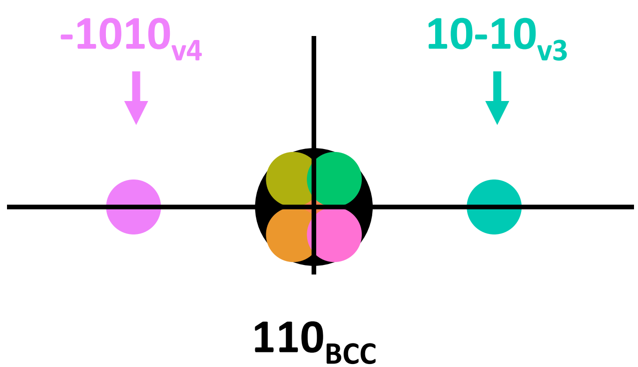



SP, [110]$_{BCC}$, 6 α'' variants



# Evidence 4: $[10\text{-}10]_{\alpha'}$ or $[010]_{\alpha''}$

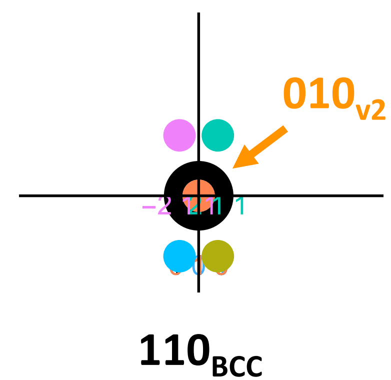
SP, $[110]_{BCC}$, 12 α' variants




~5° deviation

P,ind=1
V1,ind=2
V2,ind=3
V3,ind=4
V4,ind=5
V5,ind=6
V6,ind=7
V7,ind=8
V8,ind=9
V9,ind=10
V10,ind=11
V11,ind=12
V12,ind=13

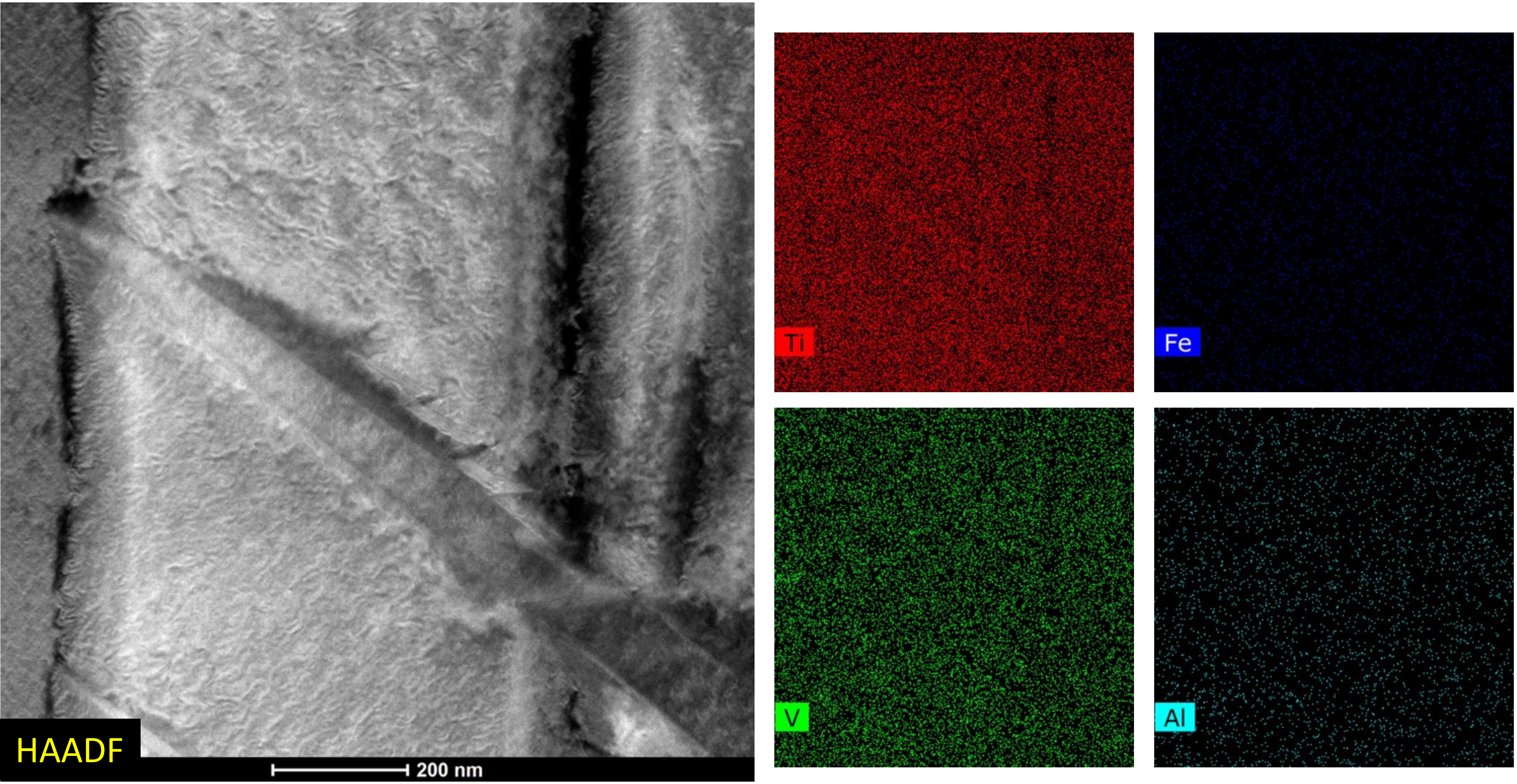
SP, $[110]_{BCC}$, 6 α'' variants




exactly parallel

P,ind=1
V1,ind=2
V2,ind=3
V3,ind=4
V4,ind=5
V5,ind=6
V6,ind=7

# Evidence 5: Diffusionless Transformation



# Conclusion

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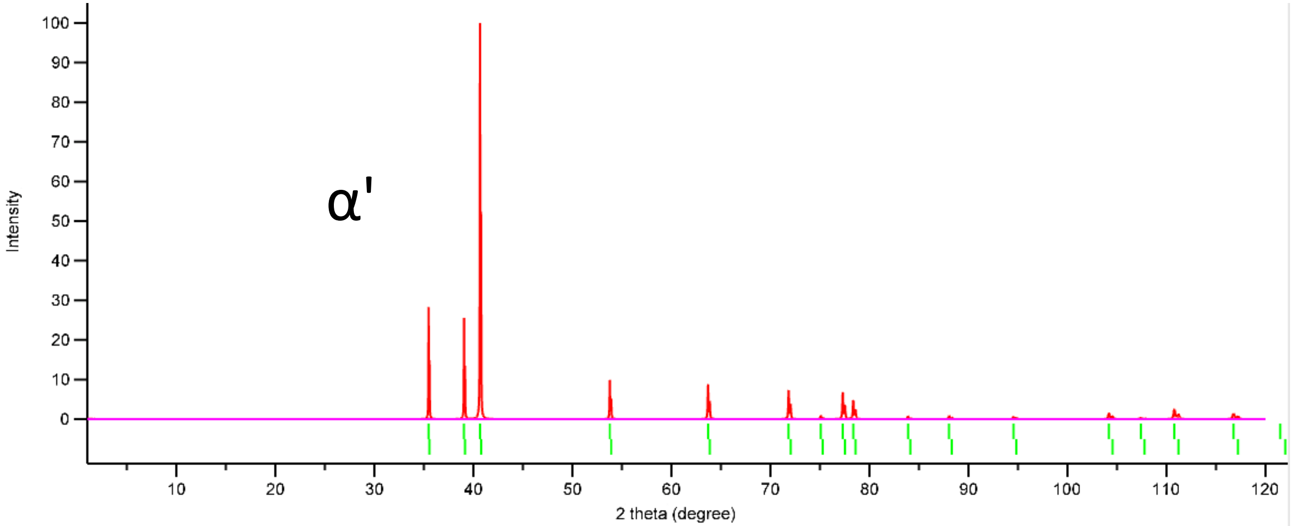
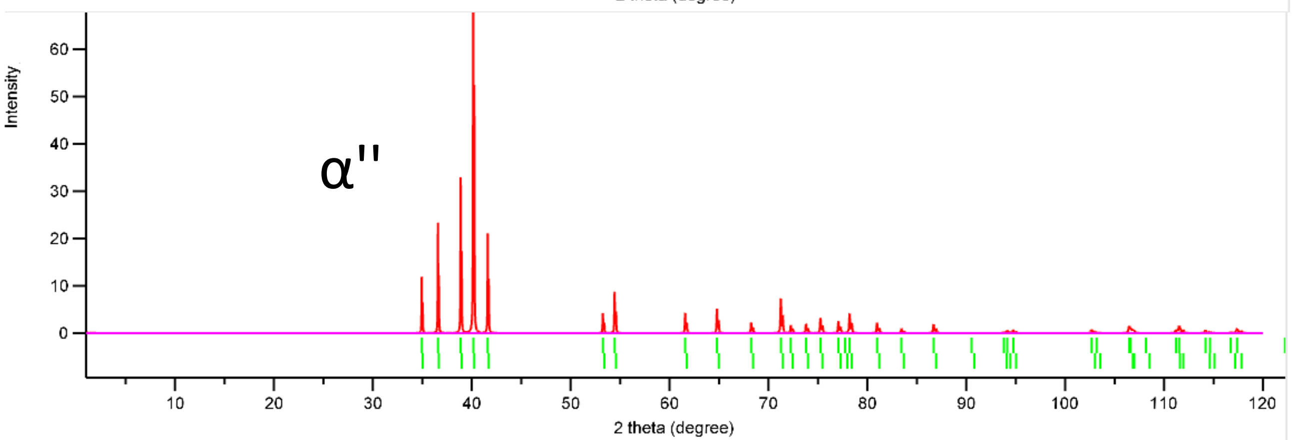
- The deformation product is more likely to be hexagonal **α'** instead of orthorhombic α''.
- XRD will be done to check whether it is α' or α''.



# Different Martensite Variants

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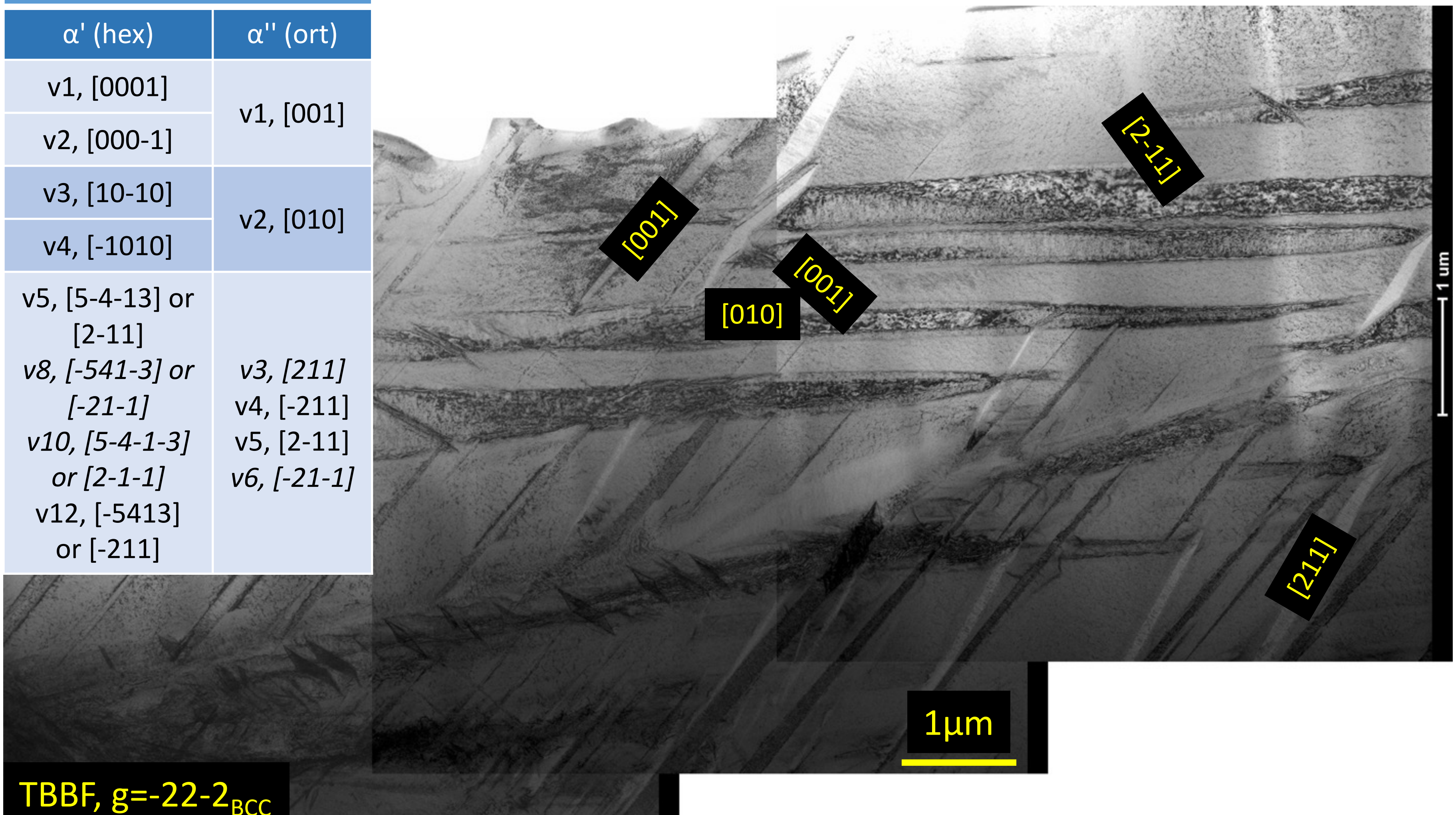
| Zone Axis Correspondence between α' and α'' in $[110]_{BCC}$ | |
|---|---|
| α' (hex) | α'' (ort) |
| v1, [0001] | v1, [001] |
| v2, [000-1] | |
| v3, [10-10] | v2, [010] |
| v4, [-1010] | |
| v5, [5-4-13] or [2-11]<br>*v8, [-541-3] or [-21-1]*<br>*v10, [5-4-1-3] or [2-1-1]*<br>v12, [-5413] or [-211] | *v3, [211]*<br>v4, [-211]<br>v5, [2-11]<br>*v6, [-21-1]* |



# Interaction of Different Martensite Variants

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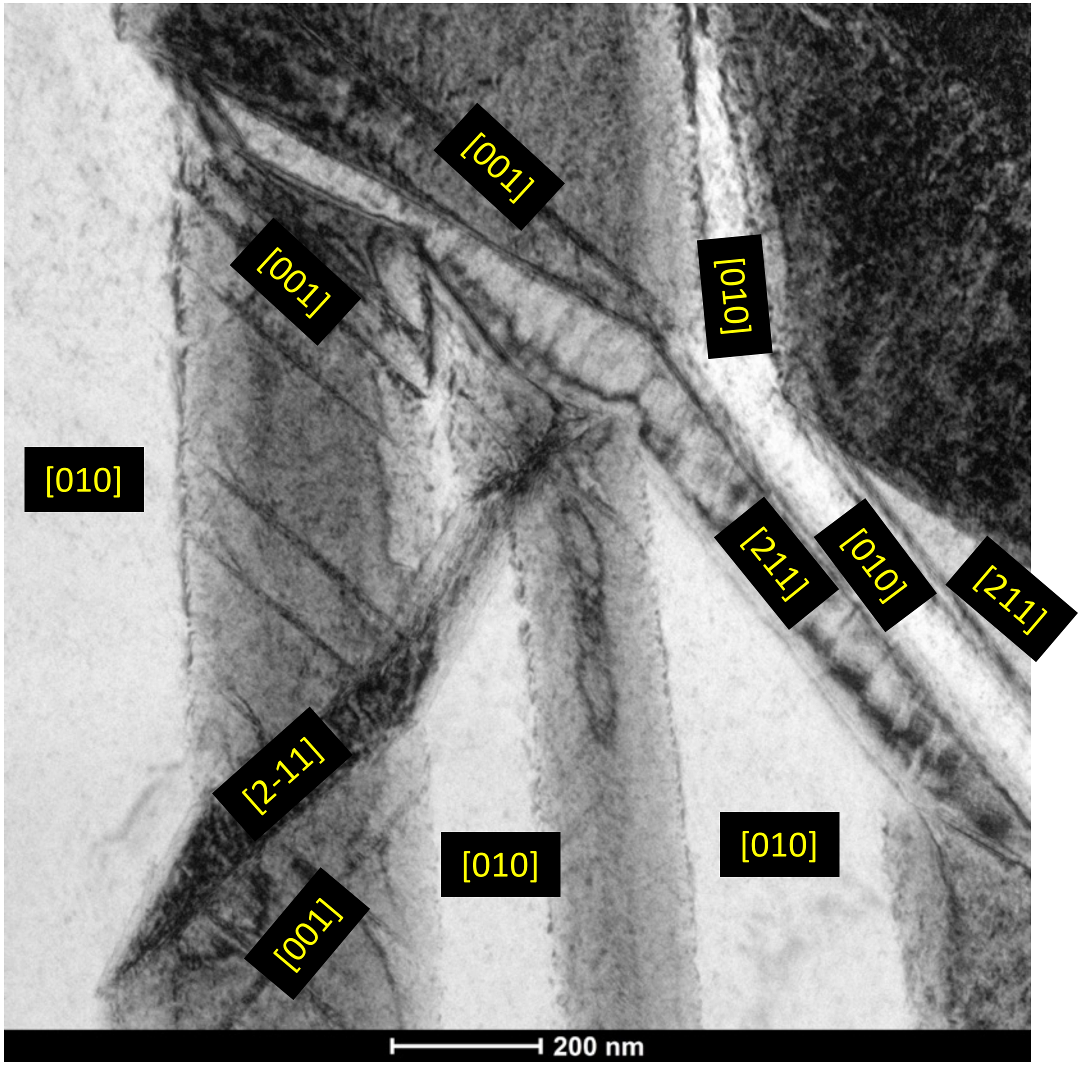
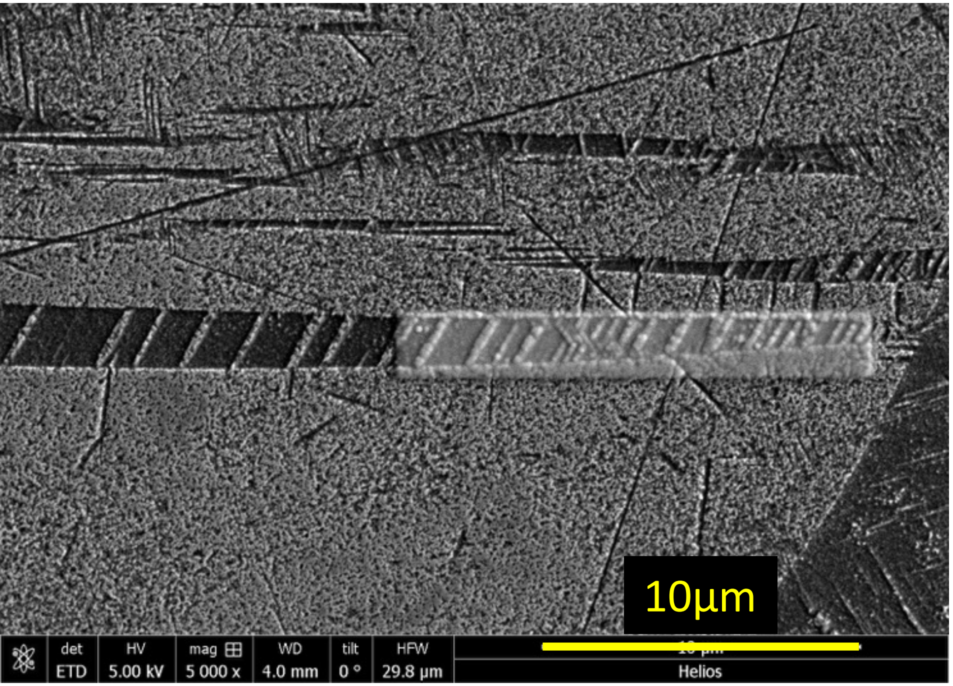


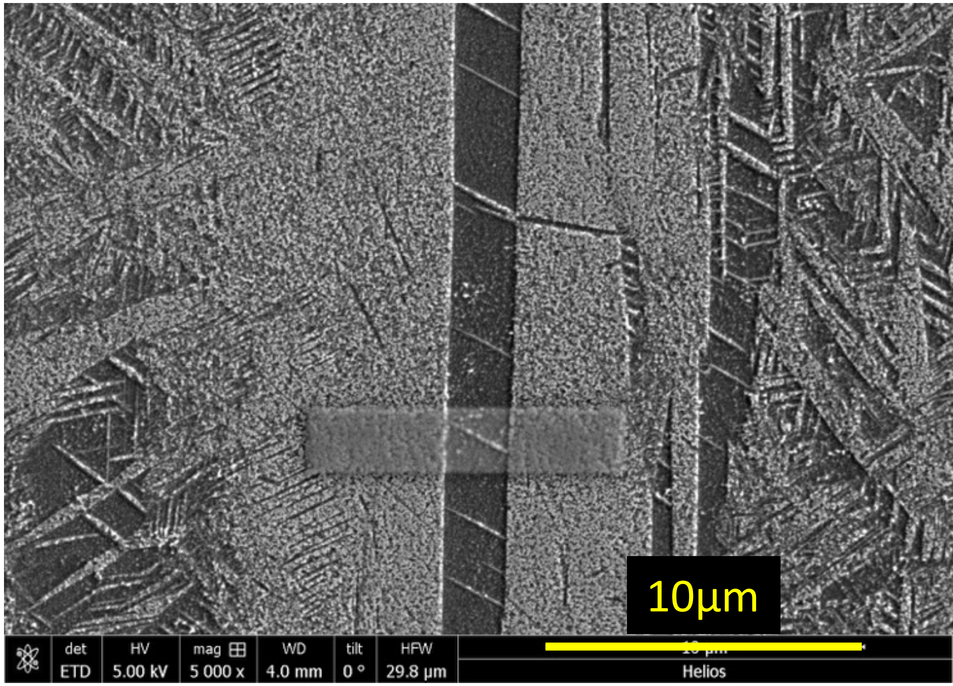
# 1.7% Deformed Ti-1023 (Tension)

1h@850°C, small grain size

lift-out-A




lift-out-C



# 1.7% Deformed Ti-1023 (Tension)

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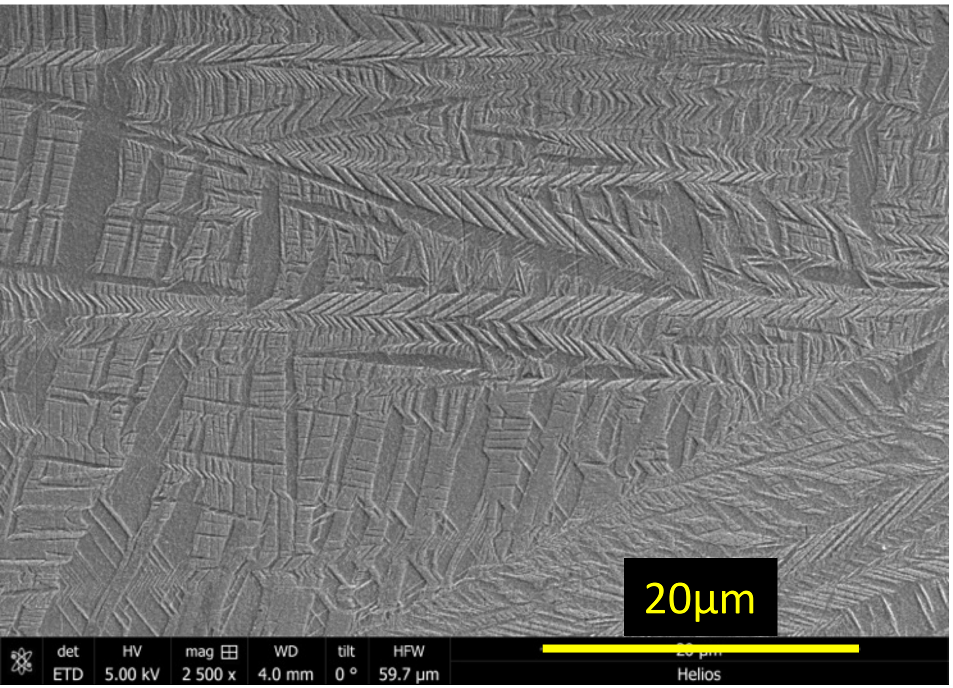
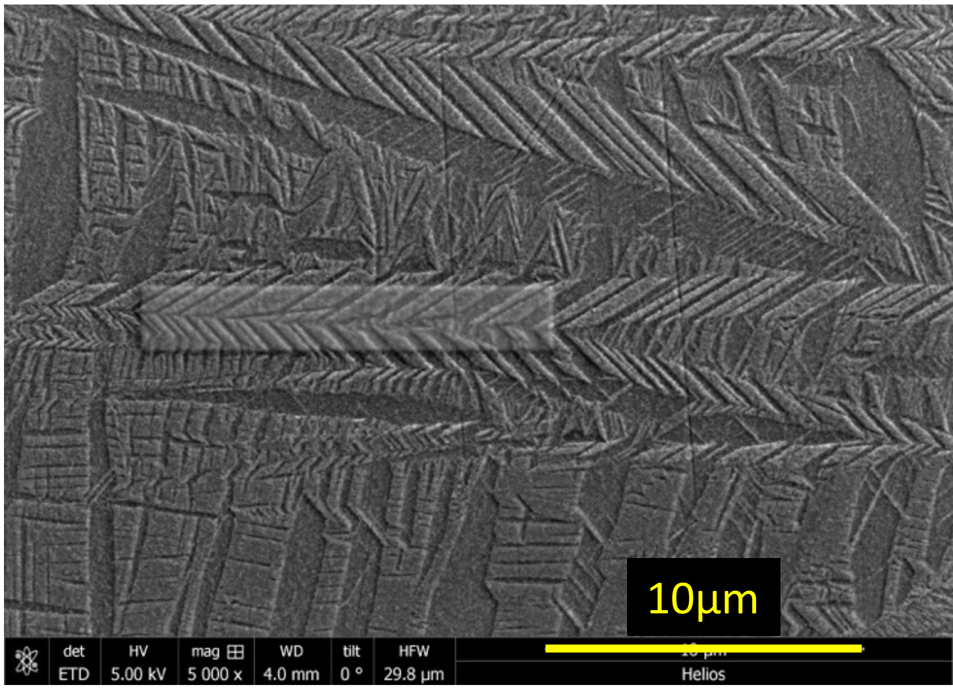
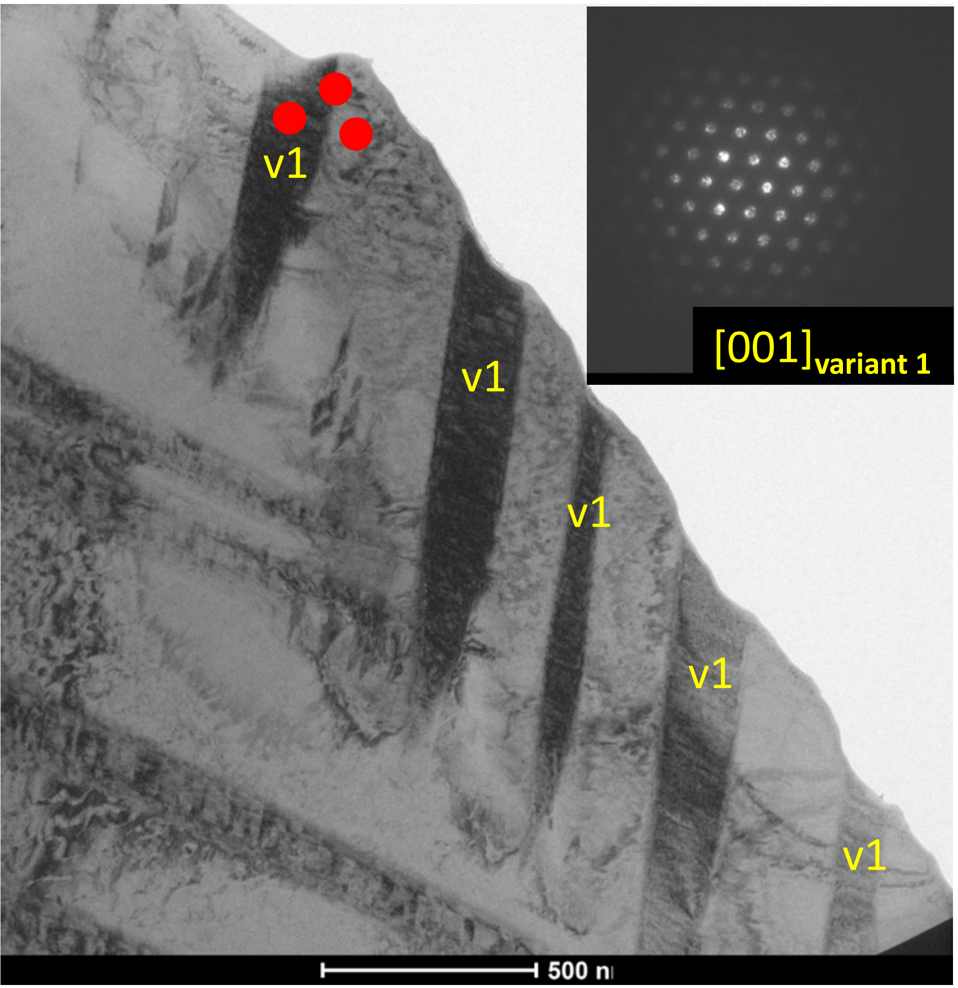
# 5% Deformed Ti-1023 (Compression)

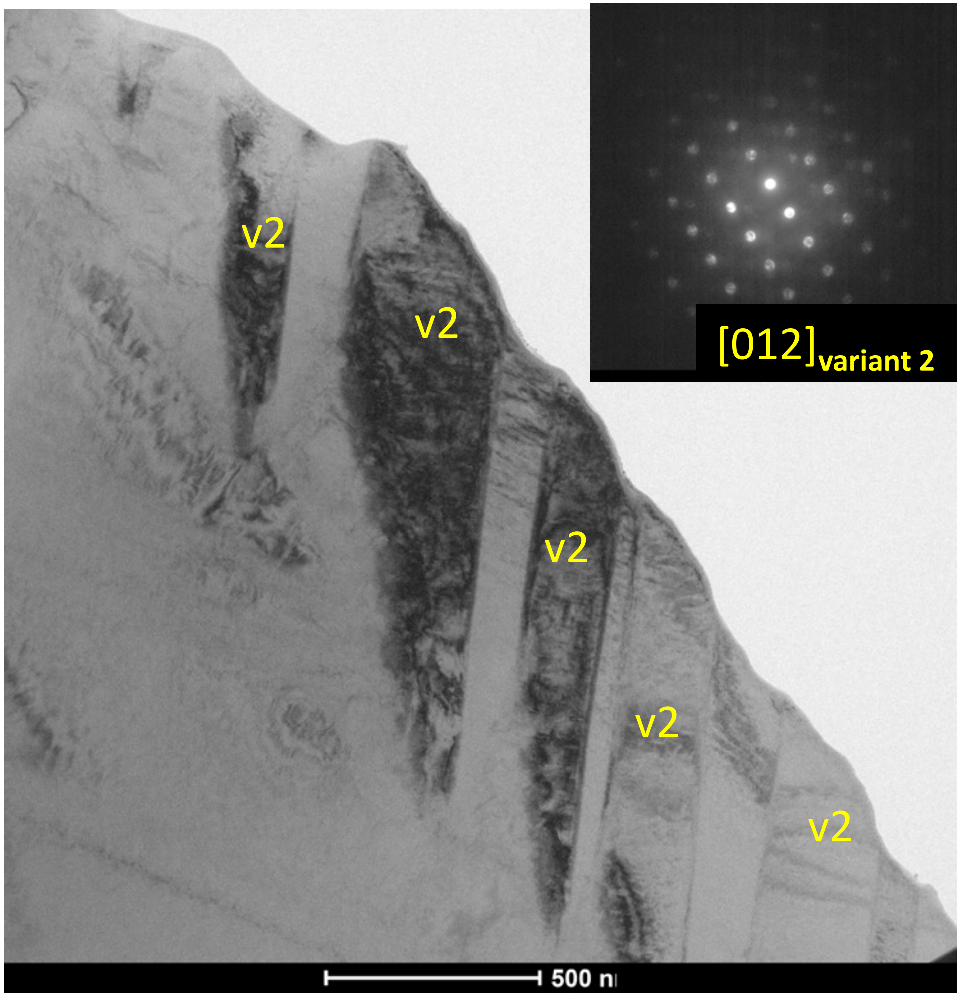
2h@900°C followed by water quenching



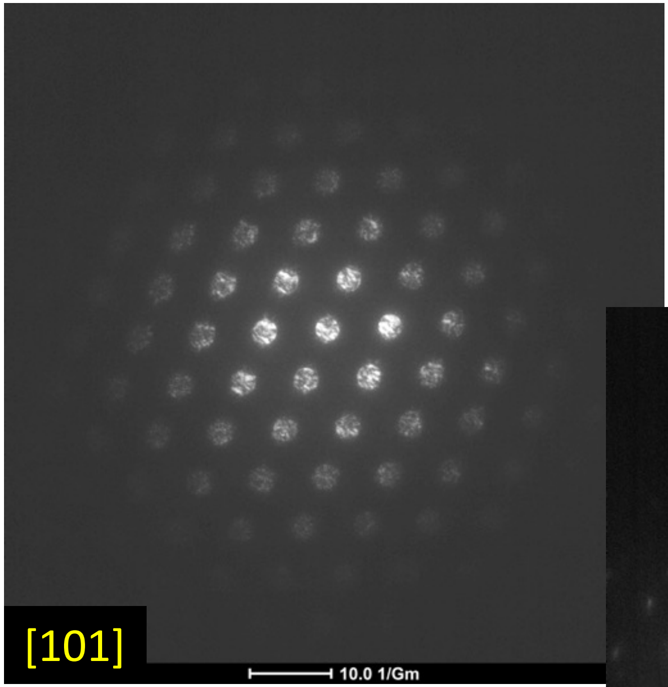



# Fully Martensitic Fishbone



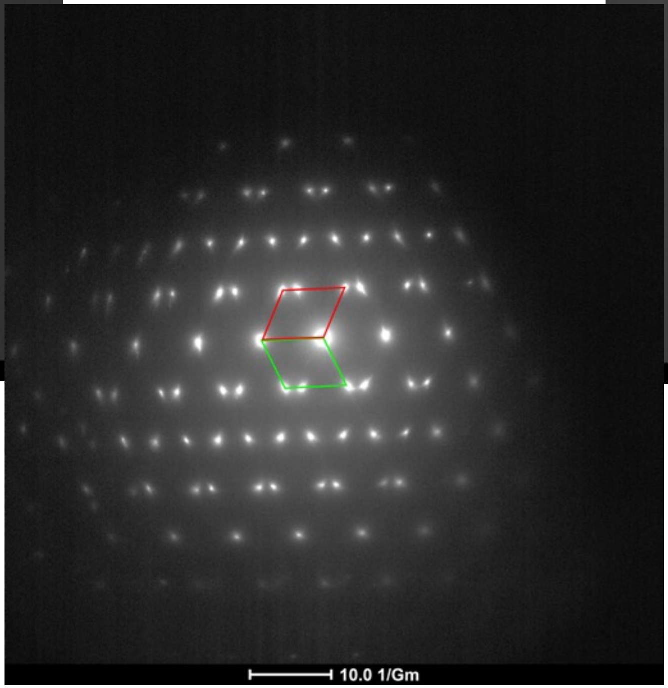



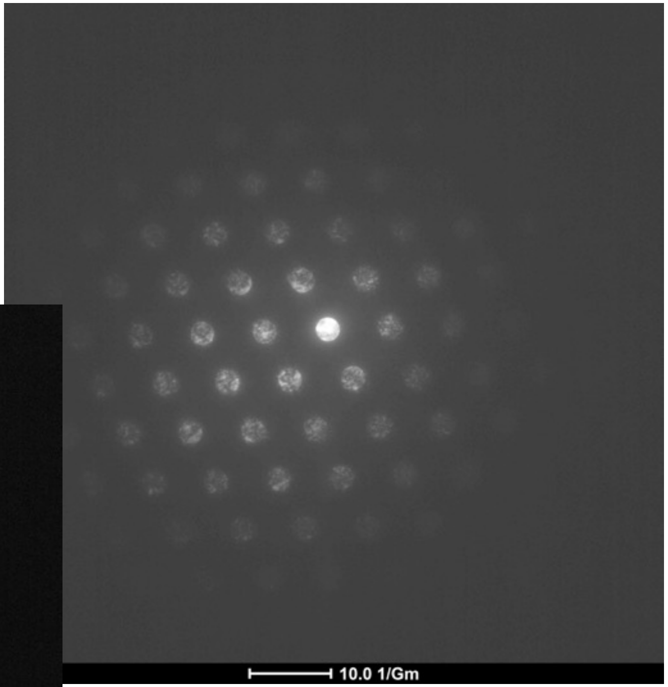
# Twinned Martensites




variant 1

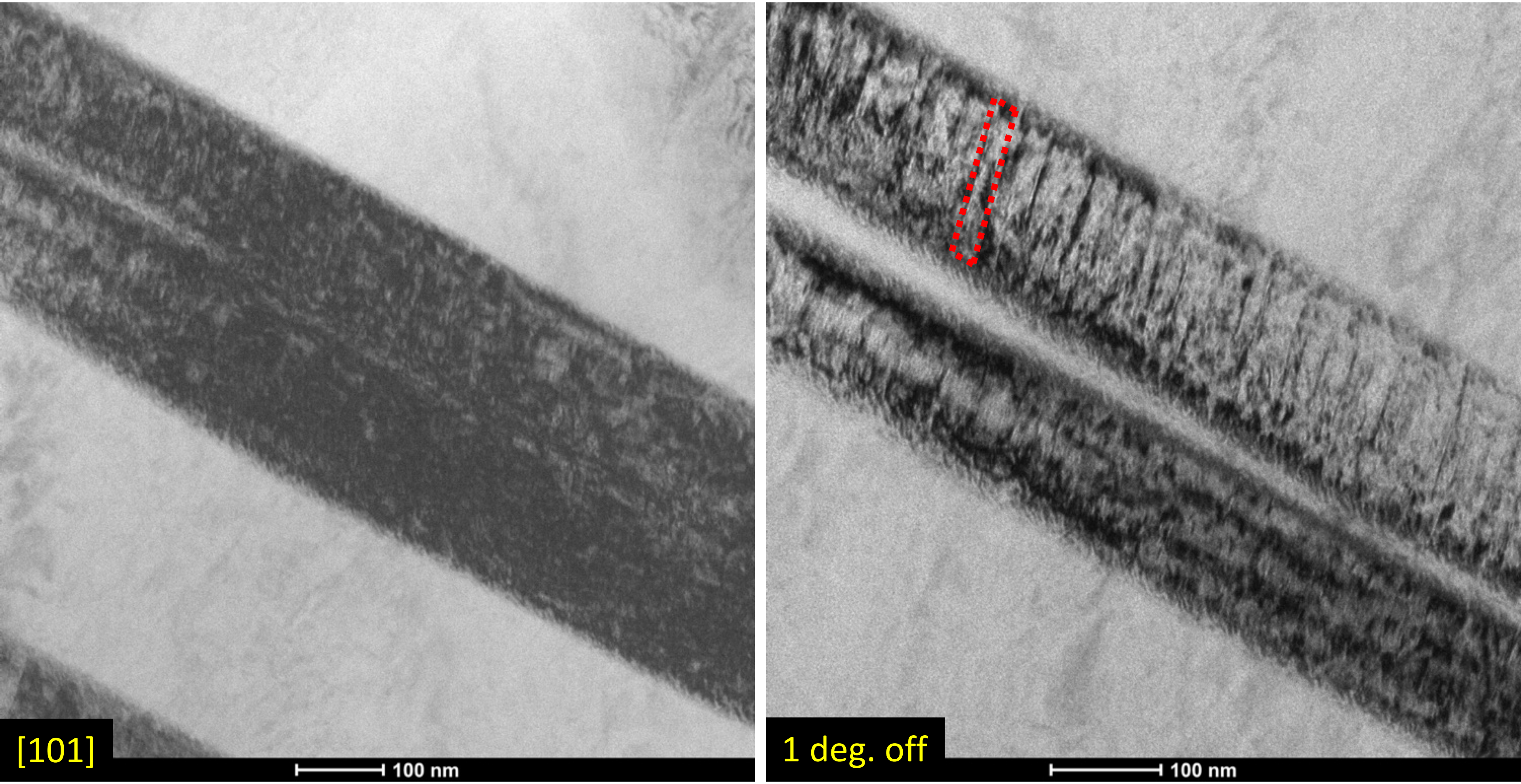
interface




variant 2

# Substructure within Each Variant



# Summary

- Very fast evolution of deformation products at low plastic strains
- No twinning or stress-induced ω was observed
- Martensite is more likely to be α' instead of α''
- Different martensitic variants were indexed

# Updated Progress Summary

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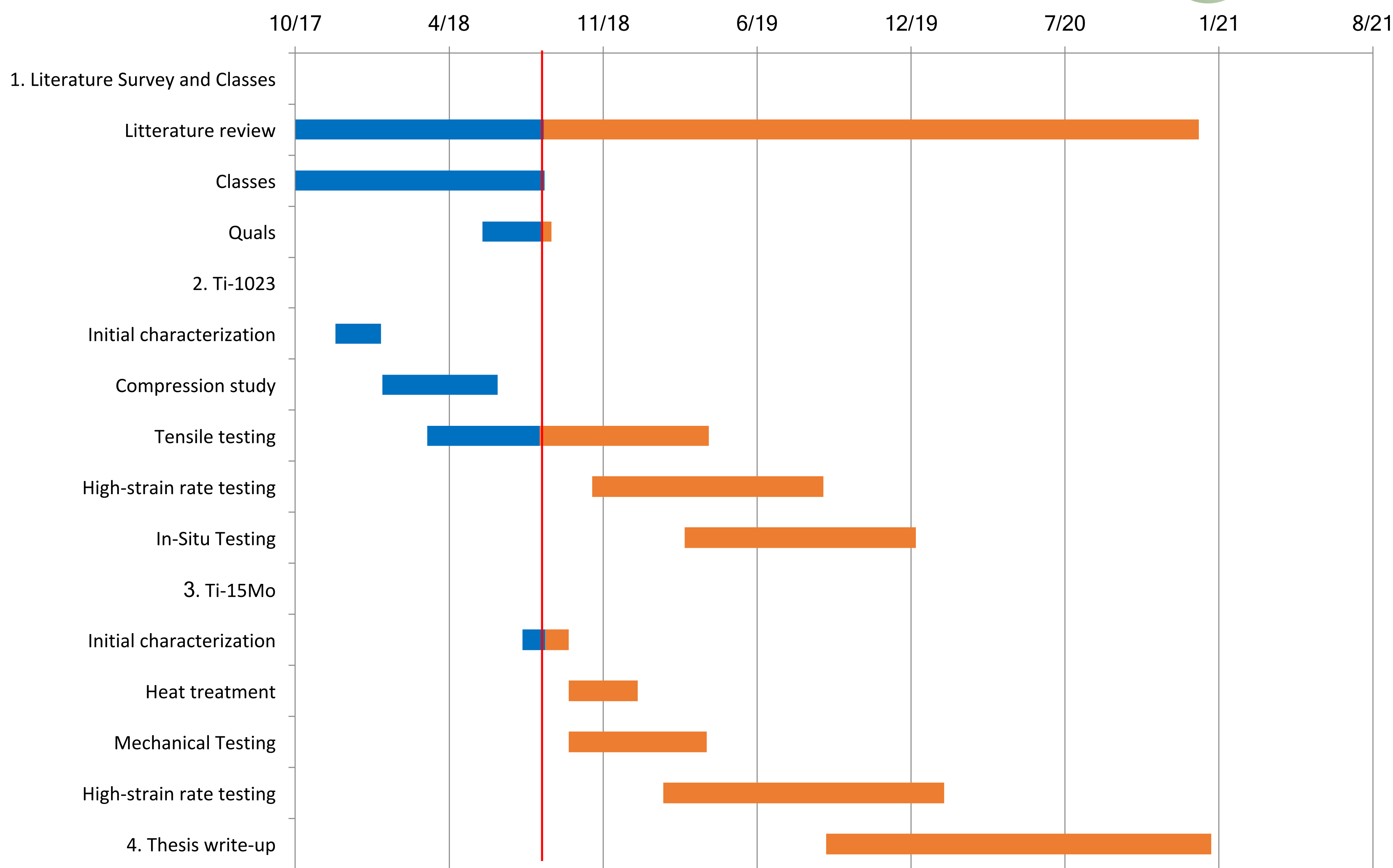

Center for ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Thank you very much!

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Yaofeng Guo

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# Center for Advanced Non-Ferrous Structural Alloys

*An Industry/University Cooperative Research Center*

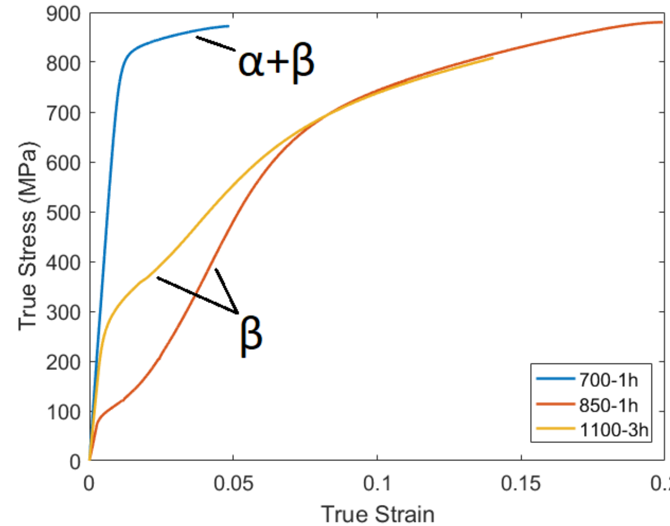
## Project 33a-L: In-situ Studies of Strain Rate Effects on Phase Transformations and Microstructural Evolution in β-Titanium

**Student:** *Benjamin Ellyson*

**Faculty:** *Amy Clarke*

**Industrial Partners:** Boeing (Austin Mann) *ARFL (TBD)*

**Project Duration:** *Sept. 2017 – May 2021*




### Achievement

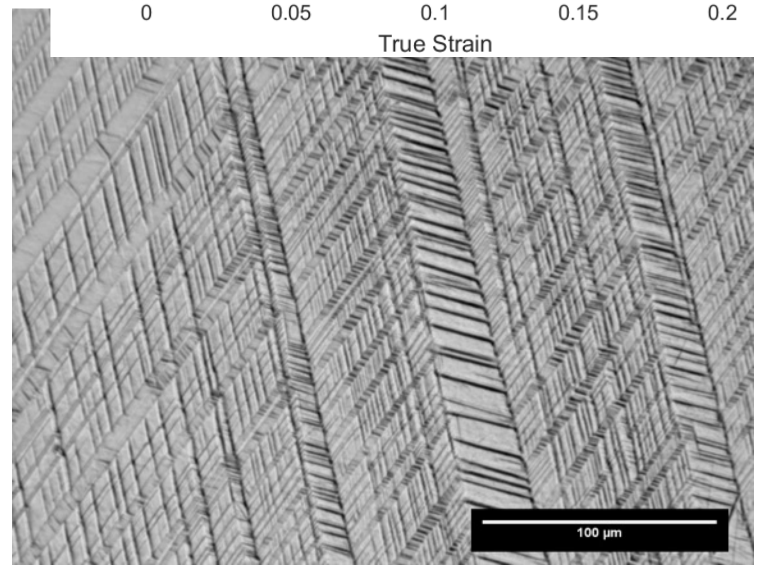
- Develop alloy design methodology to tailor microstructural evolution and mechanical properties for blast resistance

### Significance and Impact

- New TRIP/TWIP metastable β-titanium alloys represent a new class of lightweight blast resistant armor and greatly extend formability to complex structural parts

### Research Details

- Testing over multiple scales (macroscopic to fine scale) and strain rates (quasi-static to dynamic) will inform alloy design



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

CANFSA
Center for ADVANCED NON-FERROUS STRUCTURAL ALLOYS

**Student:** *Benjamin Ellyson*

**Faculty:** *Amy Clarke*

**Industrial Partners:** *ORNL (Amit Shyam)*

**Project Duration:** *Sept. 2017 – May 2021*

## Achievement

- Develop alloy design methodology to tailor microstructural evolution and mechanical properties for blast resistance

## Significance and Impact

- New TRIP/TWIP metastable β-titanium alloys represent a new class of lightweight blast resistant armor and greatly extend formability to complex structural parts

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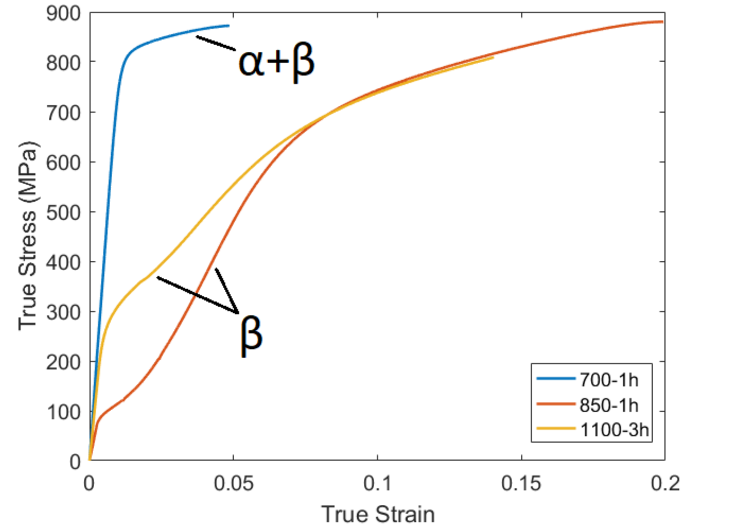


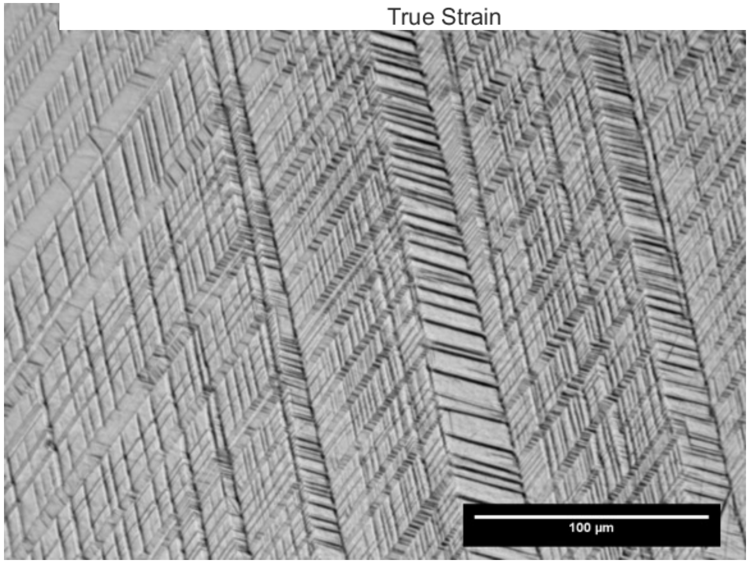

# Project 33a-L: In-situ Studies of Strain Rate Effects on Phase Transformations and Microstructural Evolution in β-Titanium

CANFSA
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**Student:** *Benjamin Ellyson*

**Faculty:** *Amy Clarke*

**Industrial Partners:** *Boeing (Austin Mann)*

**Project Duration:** *Sept. 2017 – May 2021*

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**Program Goal**

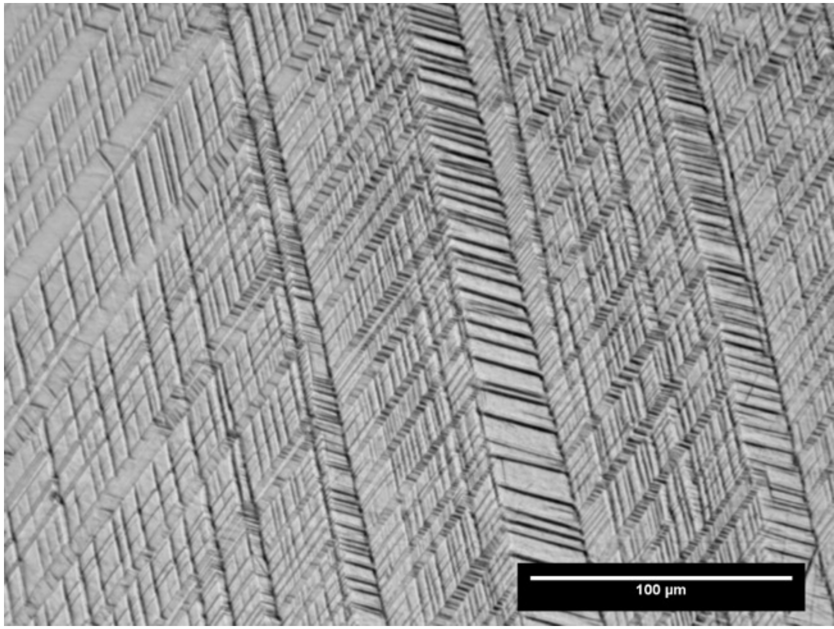
- understand and model microstructural evolution in metastable β Titanium alloys to tailor properties for blast and crash resistance

**Approach**

- Utilize testing and in-situ testing and characterization over multiple length scales and decades of strain rates to understand metastable deformation mechanisms

**Benefits**

- Improved understanding of microstructural evolution in metastable β Titanium alloys and formulation of novel alloys with tailored performance




Microstructural evolution in Ti-1023 compressed to 5%