CANFSA
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An Industry/University Cooperative Research Center

# Project 29-L: Identification of Deformation Mechanisms in Thermally Stable Cast Al-Cu Alloys via Neutron Diffraction

**Summer Teleconference**

**July 8, 2020**


- Student: Brian Milligan (Mines)
- Faculty: Dr. Amy Clarke (Mines)
- Industrial Mentors: Amit Shyam (ORNL), John Carpenter (LANL)

Oak Ridge National Laboratory

NSF
Colorado School of Mines
Iowa State University

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- Student: Brian Milligan (Mines)
- Advisor(s): Amy Clarke (Mines), Amit Shyam (ORNL)


**Project Duration**

Ph.D.: August 2017 to May 2021

Problem
- Deformation and phase transformation behavior at a micro-scale in Al-Cu alloys is not well understood.

Objective
- Apply in-situ neutron diffraction, SEM, TEM, mechanical testing, and synchrotron X-ray imaging to better understand the mechanical behavior and phase transformations in these alloys.

Benefit
- Improvement of properties of thermally stable Al-Cu alloys (including new ORNL alloy), as well as furthering scientific understanding of precipitation strengthened Al alloys.

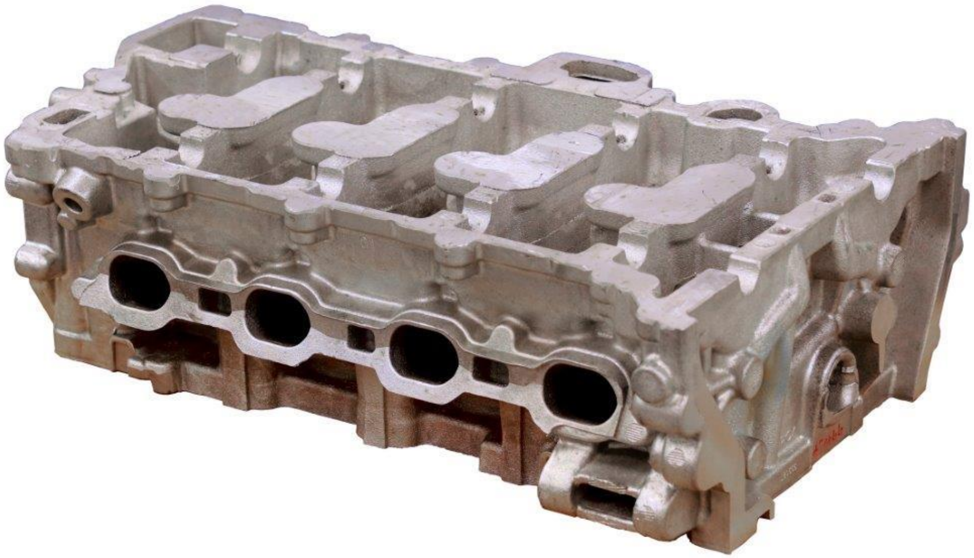
Recent Progress
- Refined previous load transfer model to predict matrix behavior and precipitate yielding behavior
- Continued microscopy study of alloy 206 post-mortem with Lawrence Allard
- Ready to submit paper on alloy 206 room temperature neutron diffraction
- Began analysis of precipitate lattice strains during elevated temperature tensile testing and creep in alloy RR350

**Metrics**

| Description | % Complete | Status |
|---|---|---|
| 1. Initial literature review | 90% | ● |
| 2. In situ neutron diffraction, creep testing, and TXM | 100% | ● |
| 3. Microstructural characterization pre- and post- creep and tension | 70% | ● |
| 4. Qualitative assessment of neutron diffraction and mechanical test data | 80% | ● |
| 5. Application and development of qualitative modelling to micro-scale diffraction data | 70% | ● |

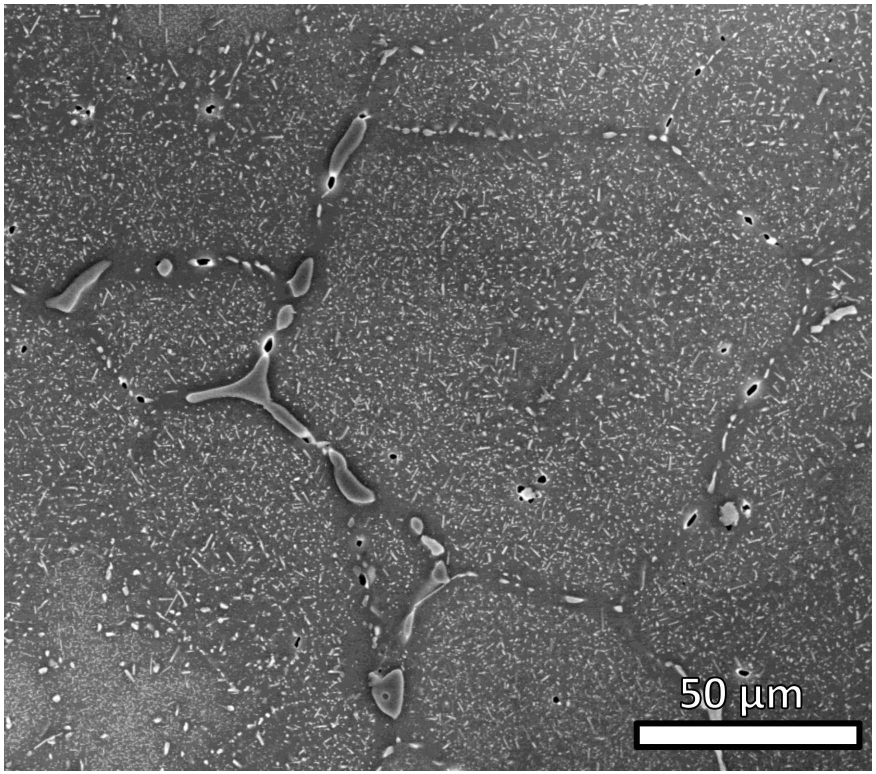
# Industrial Relevance

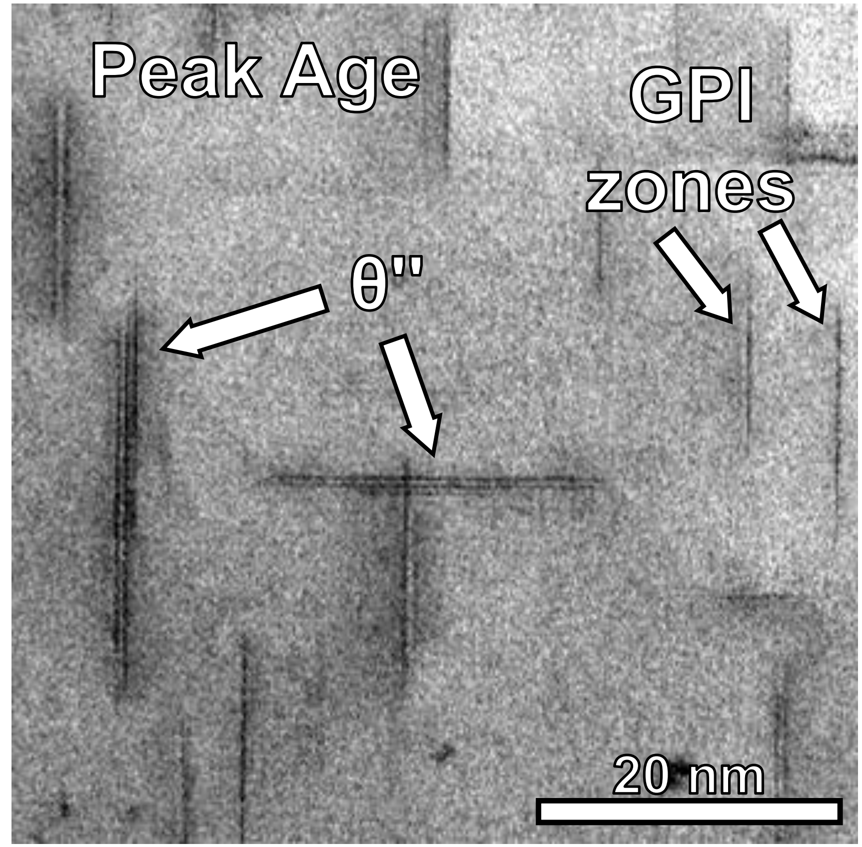
- Cast Al-Cu alloys have high strength, low density, and are very castable
  - Used in various industries such as for cylinder heads in light-duty engines
- Understanding of deformation mechanisms allow prediction of mechanical behavior
  - Strain hardening behavior commonly overlooked, but is relevant for fatigue life



Cylinder head cast with ORNL ACMZ alloy.
Credit: Jason Richards (ORNL)

# Al-Cu Alloy 206 used for Room Temperature Testing



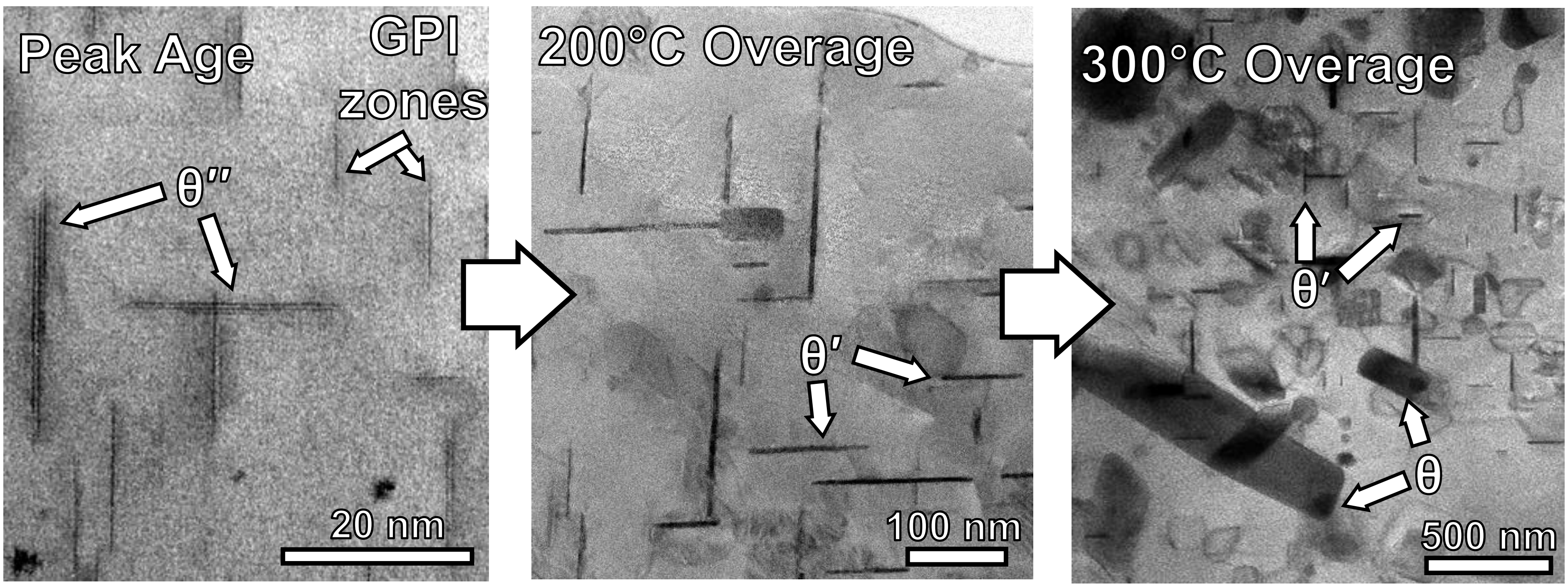

Composition of Alloy 206 (wt%)

| Si | Cu | Mg | Fe | Mn | Ti | Al |
|---|---|---|---|---|---|---|
| 0.12 | 4.5 | 0.30 | 0.14 | 0.23 | 0.02 | bal. |

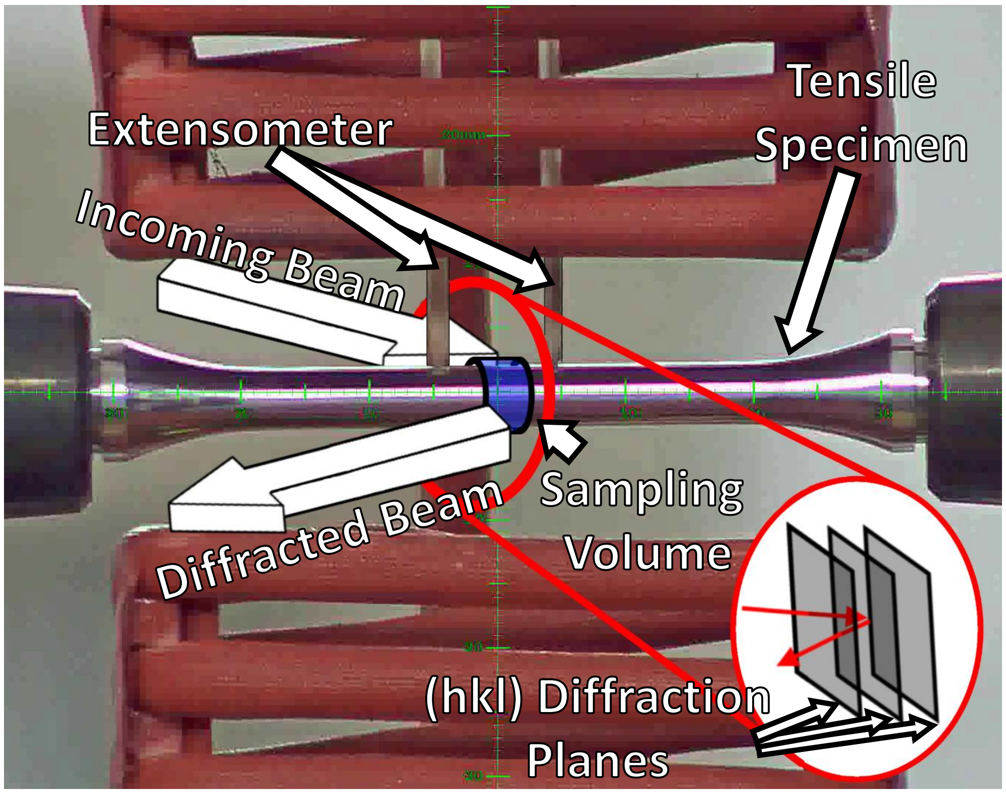
STEM images courtesy of Lawrence Allard. Zone axis is [001].

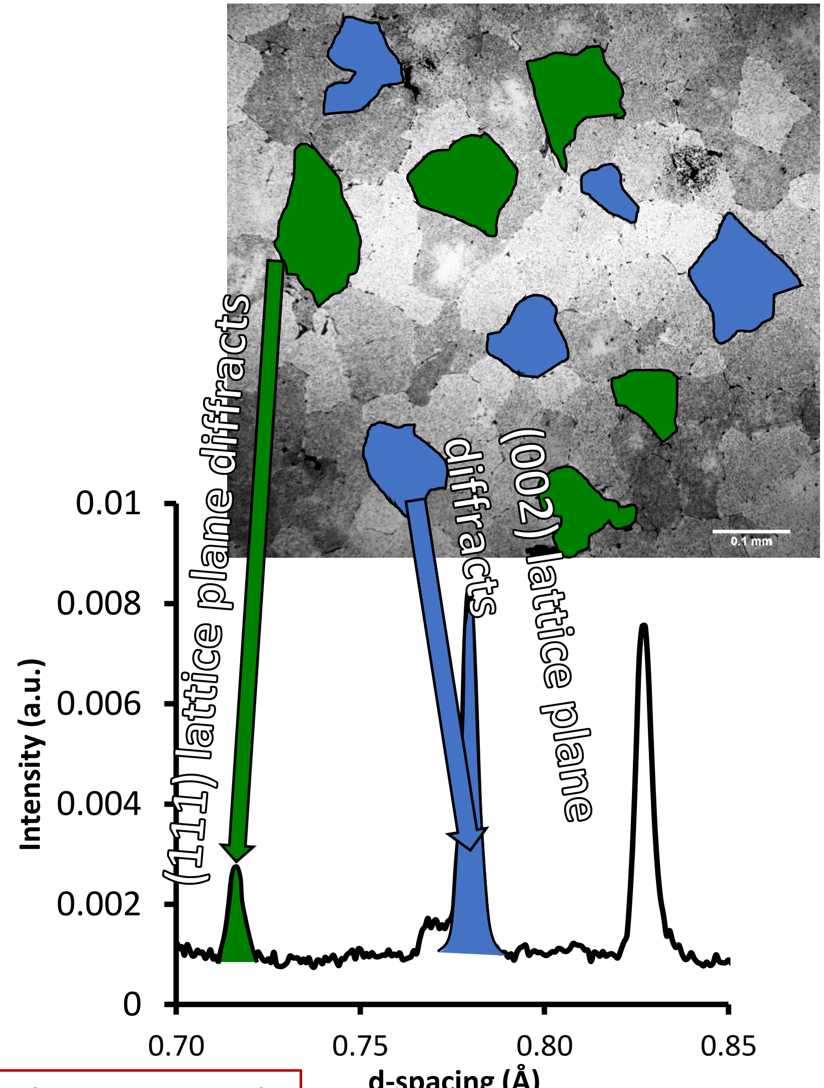
# Heat Treatments → Microstructure

| Step | Solutionize | Water Quench | Peak Age | Overage |
|---|---|---|---|---|
| Temp. (°C) | 500 | 80-90 | 190 | 200, 300 |
| Time (h) | 5 | <1 | 5 | 200 |

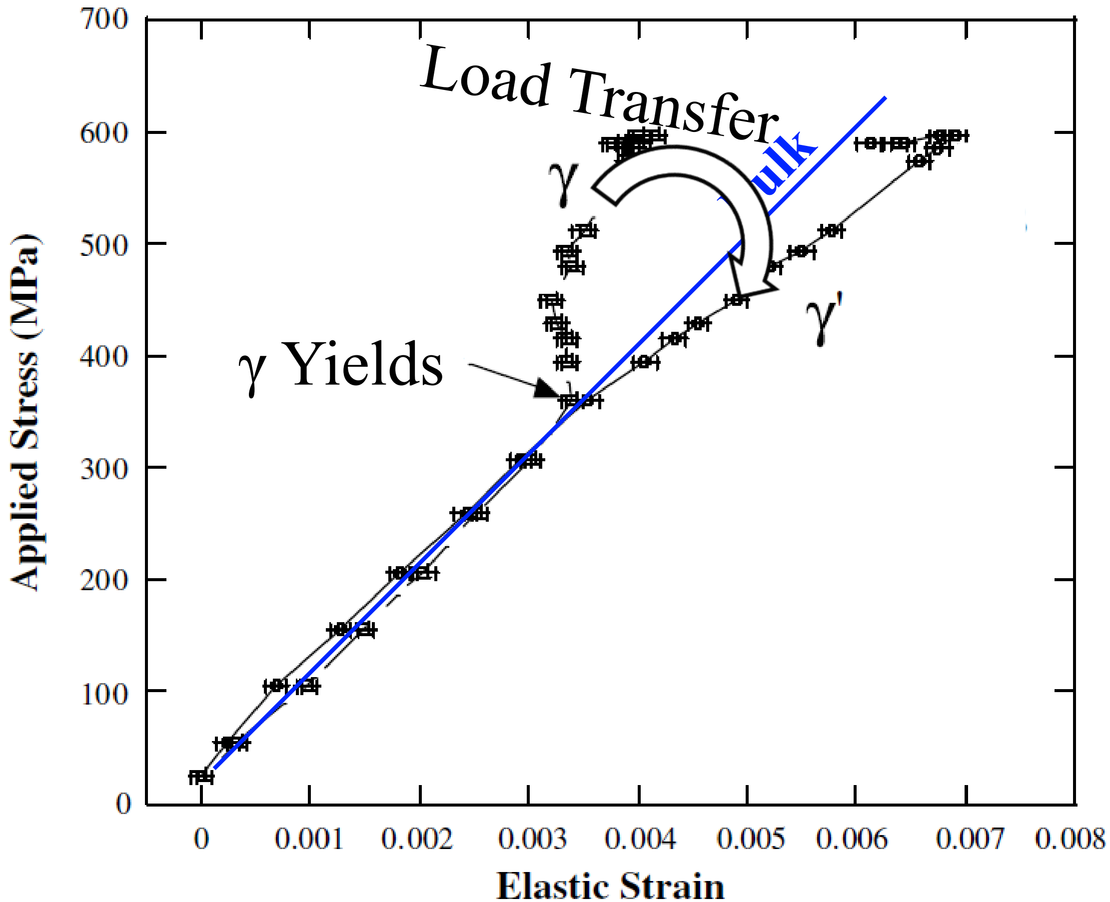


# Neutron Diffraction Experiments at SNS VULCAN



# Y-plot Demonstrates Load Transfer Between Phases




PWA 1422 superalloy, reproduced from [1]

# Peak Age: θ″ Precipitates Shear Easily, No Load Transfer

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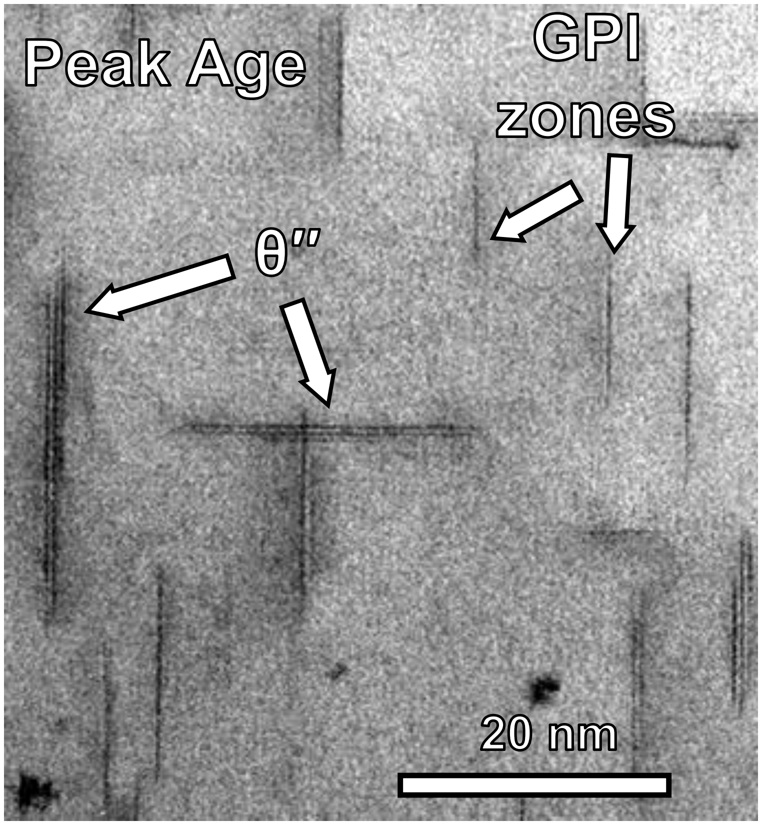
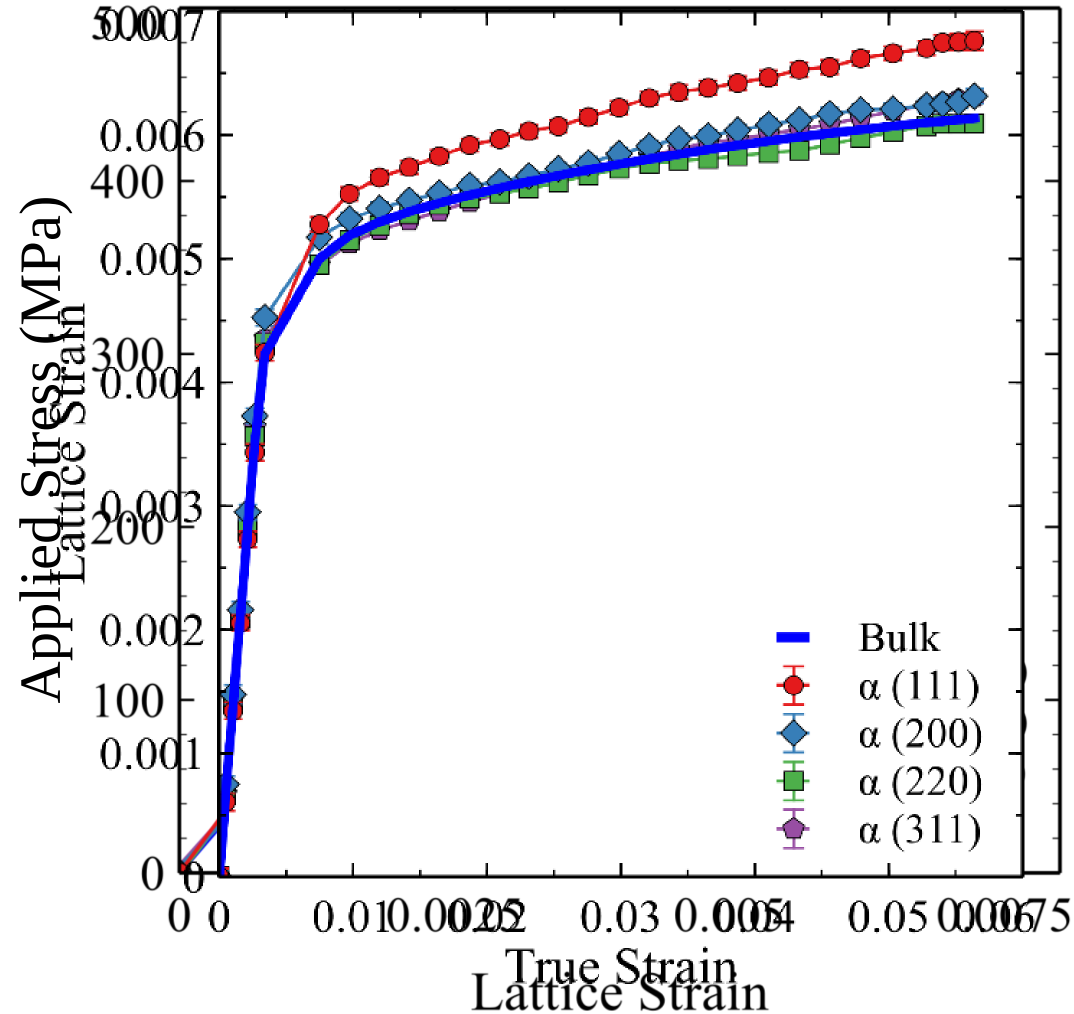
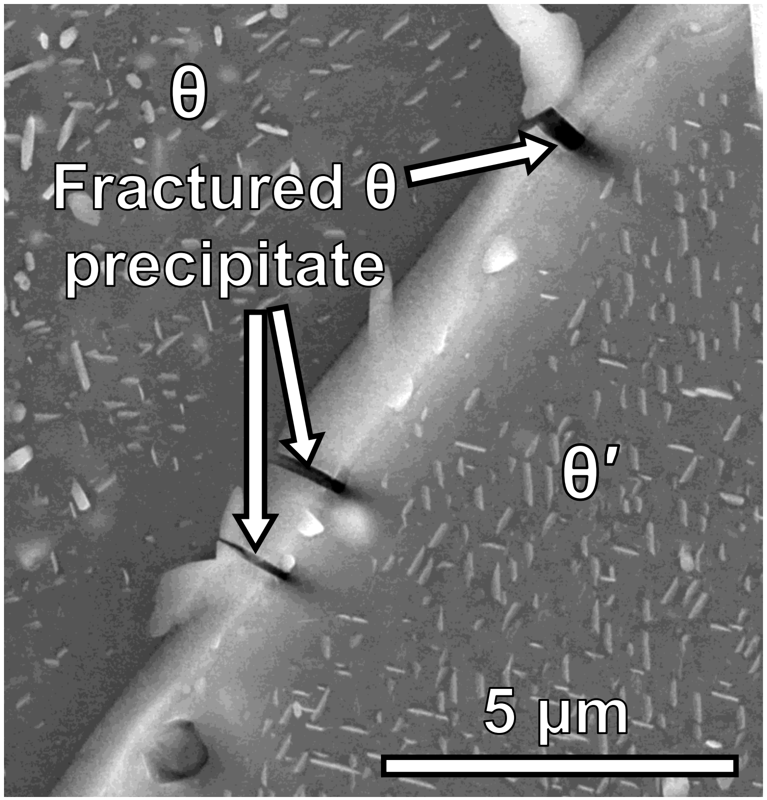
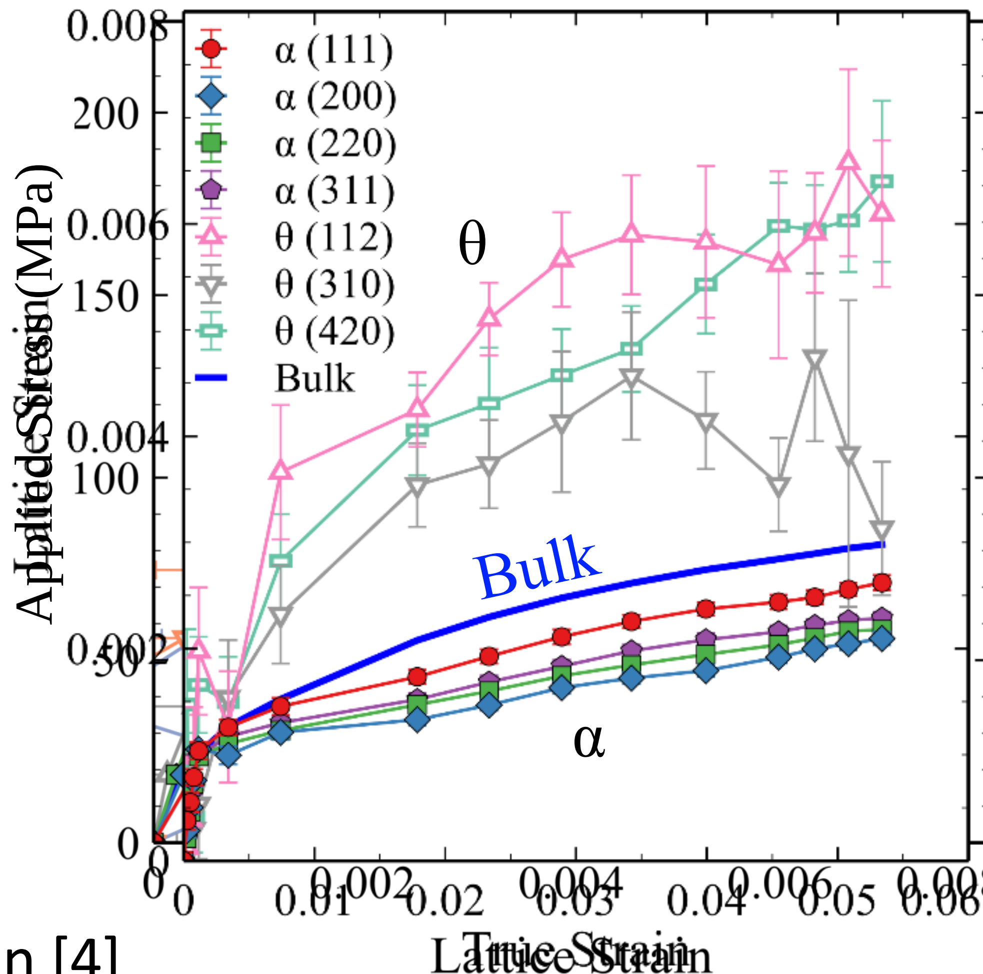



- θ″ precipitates easily sheared by dislocations [2]

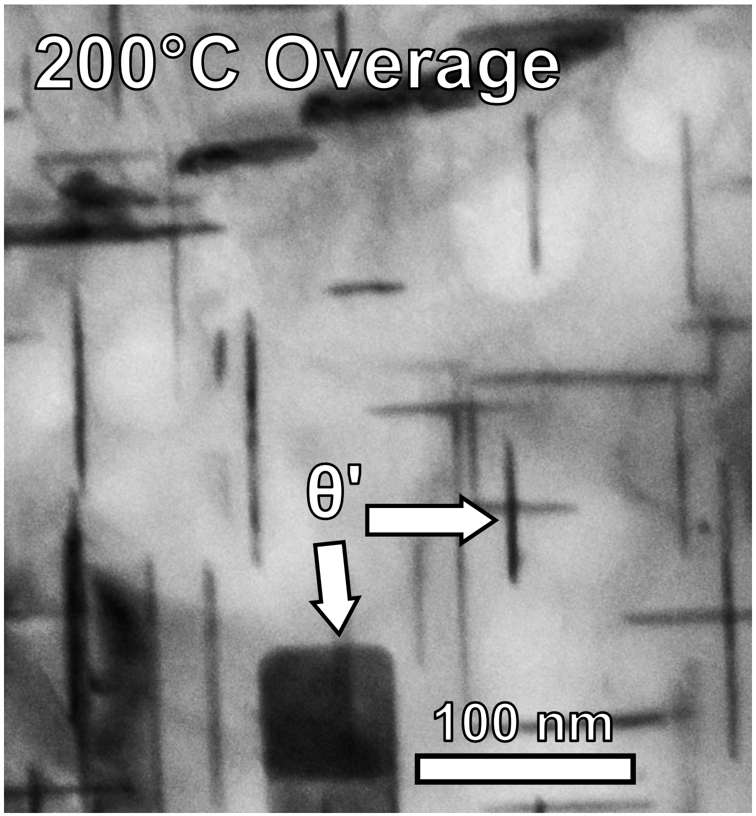
# 300°C Overage: Orowan Looping, Load Transfer and Precipitate Fracture

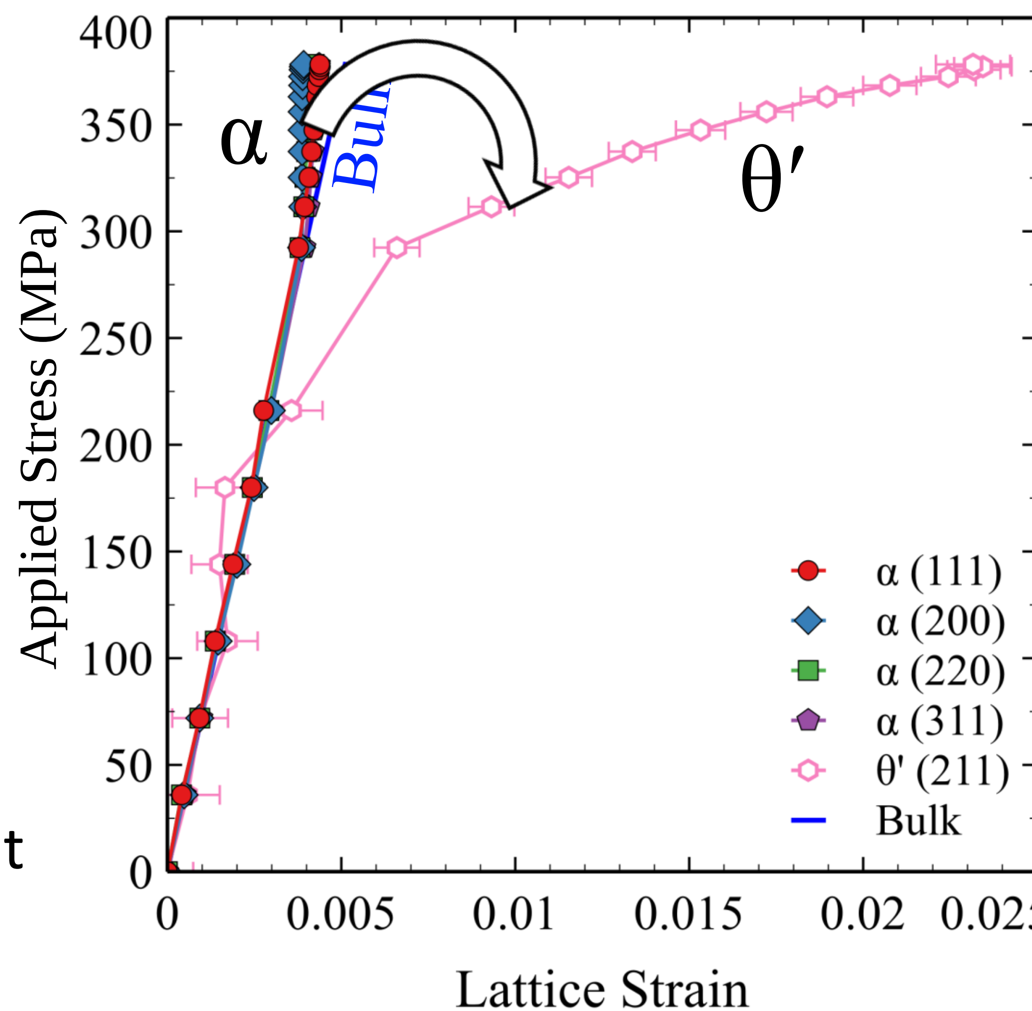



- θ precipitates un-shearable [3]
- Precipitate fracture at high strain [4]

# 200°C Overage: Orowan Looping, Load Transfer, and Anisotropy

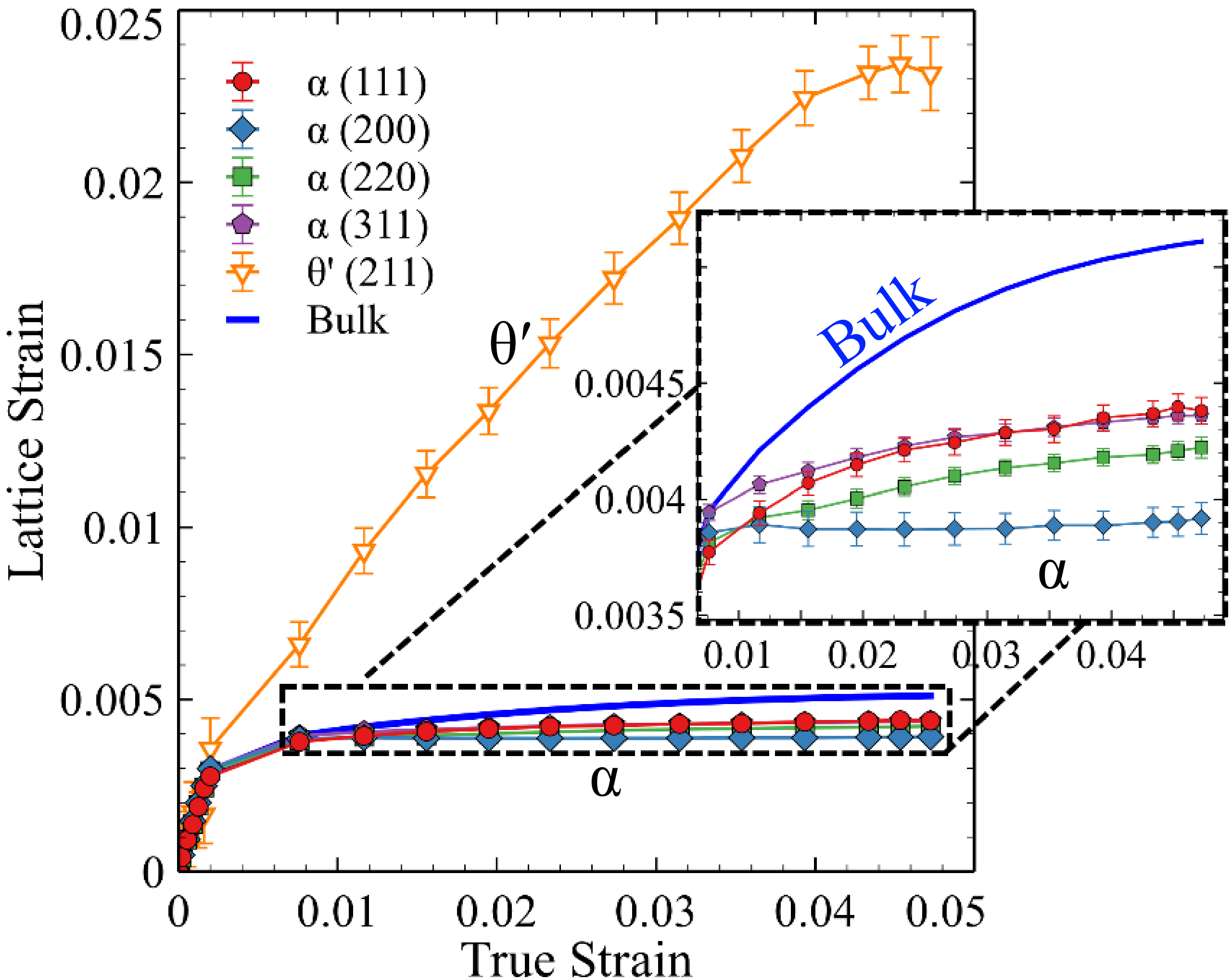


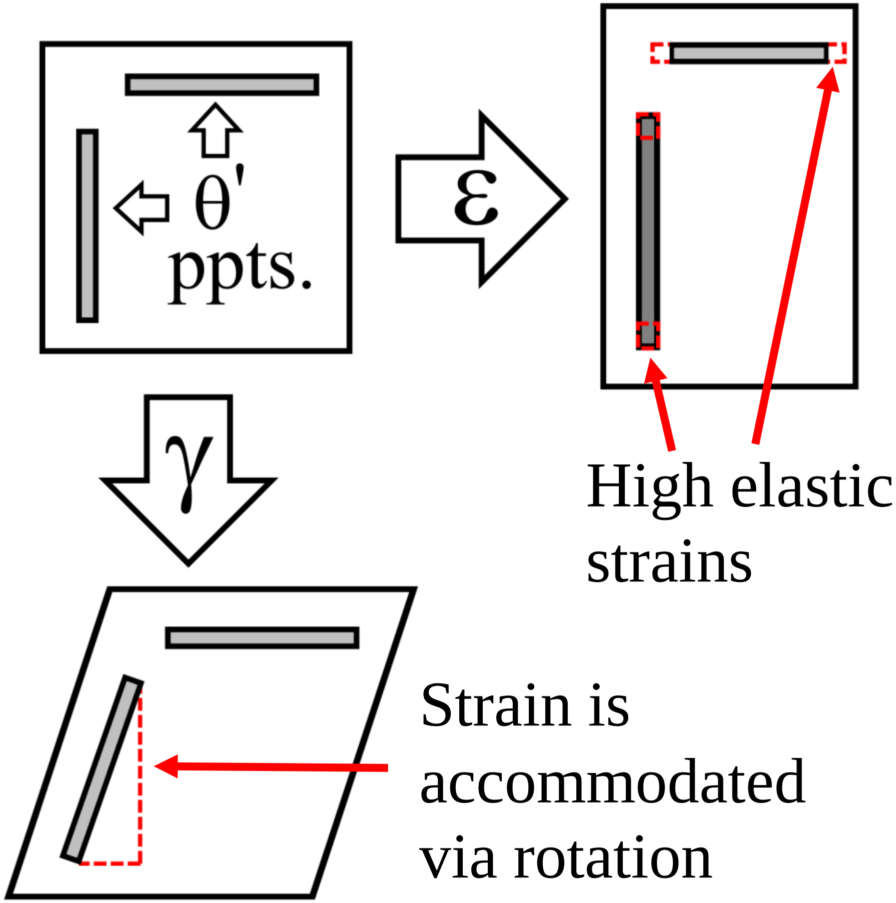

- θ′ precipitates un-shearable at bulk yield point [3]

# Anisotropic Load Transfer Causes Anisotropic Strain Hardening

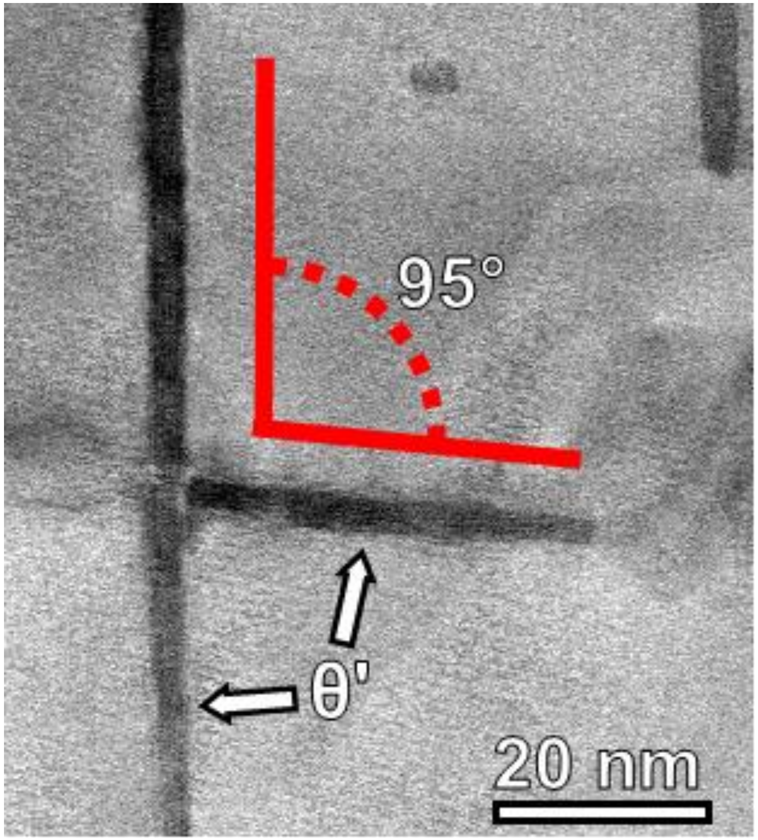




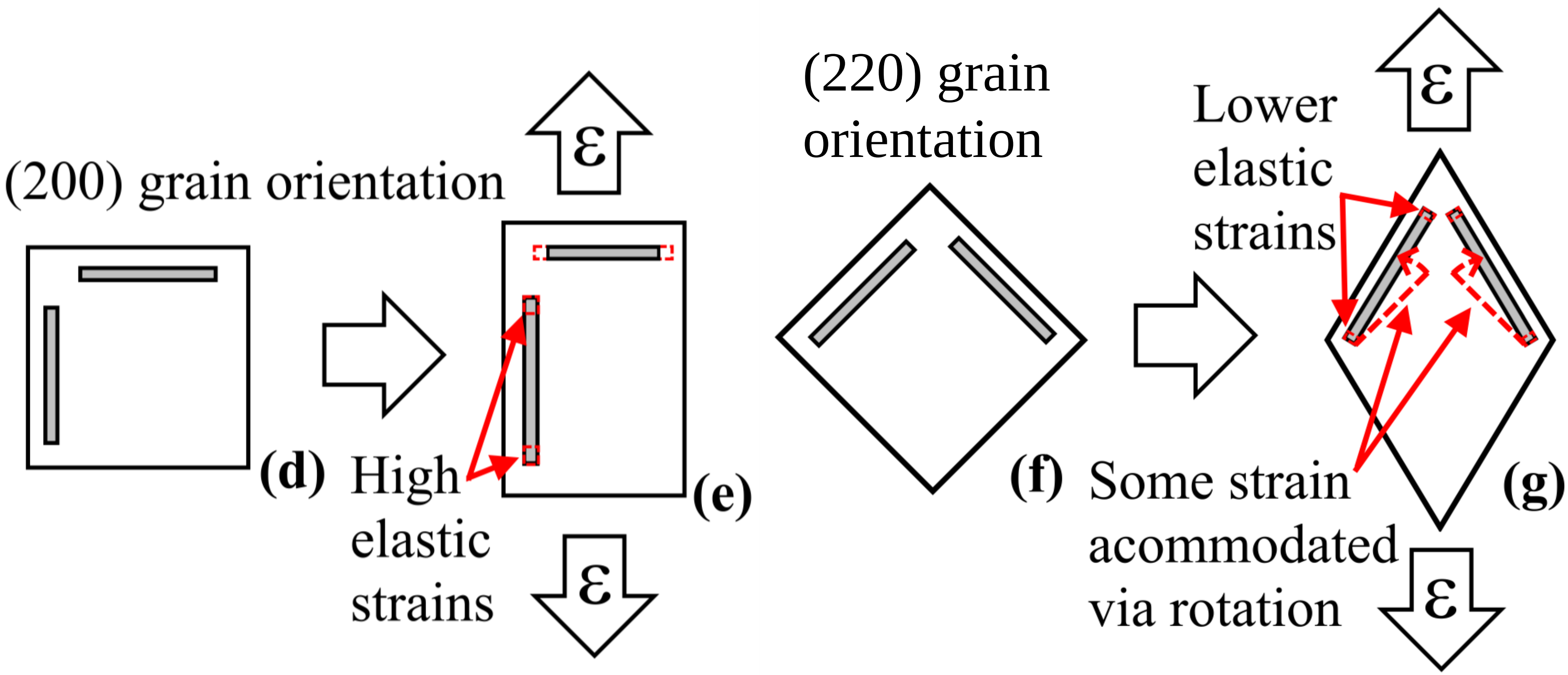
# Anisotropic Load Transfer Caused by Precipitate Rotation




Post-mortem 206 200°C overaged sample

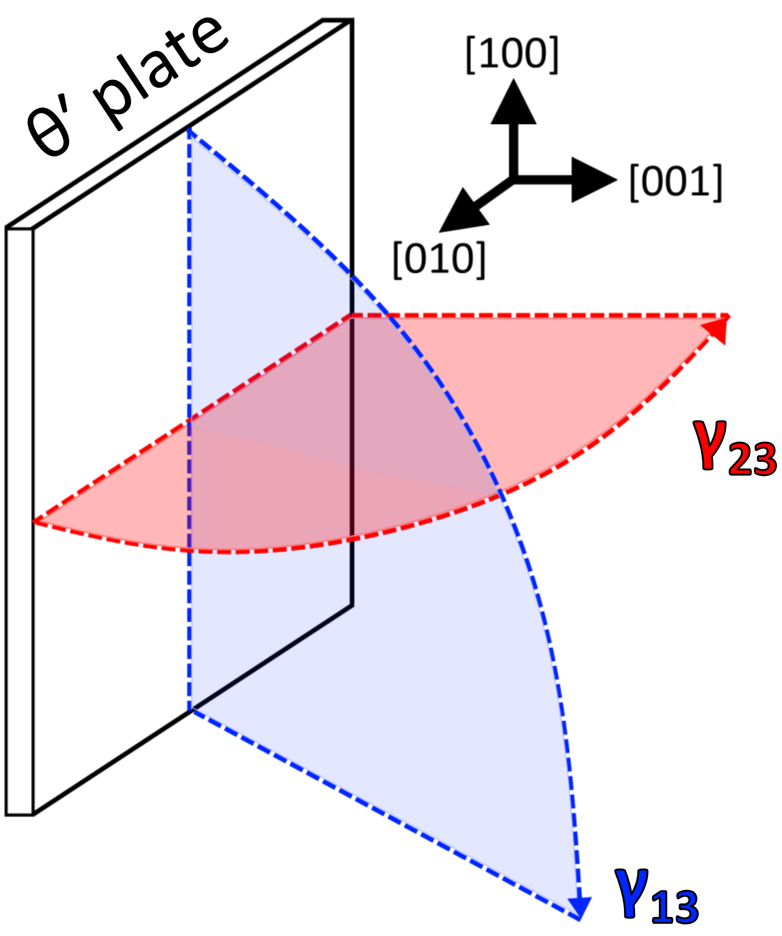


# (200) Grain Orientation has Highest Load Transfer

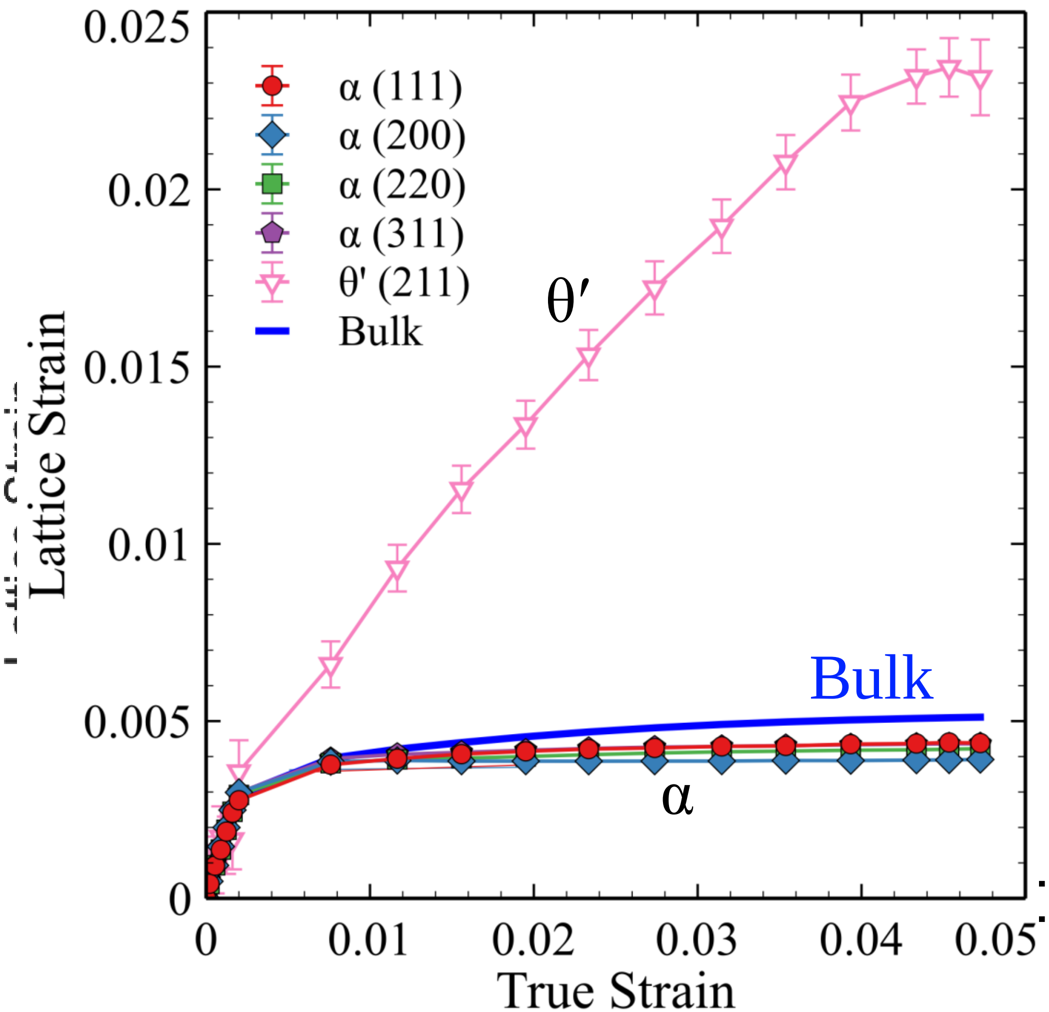


# Modeling Precipitate Rotation: Assumptions

- Uniaxial bulk strain
- Iso-strain conditions
- Precipitates do not yield
- $\gamma_{13}$ and $\gamma_{23}$ are accommodated *via* rotation [6]
- Applied stress and stress from load transfer do not interact



# Modeling Precipitate Rotation: Total Lattice Strain




$$\sigma_{total}^{hkl} = \sigma_{app} + \sigma_{transfer}$$

$$\epsilon_{lattice}^{hkl} = \frac{\sigma_{total}^{hkl}}{E^{hkl}}$$

*Assuming precipitate does not yield

$$\sigma_{transfer} = \epsilon_{transfer} * E^{hkl}$$

$$\epsilon_{lattice}^{hkl} = \frac{\sigma_{app}}{E^{hkl}} + \epsilon_{transfer}$$

# Transfer Strain 1. Calculate Specimen Strain State

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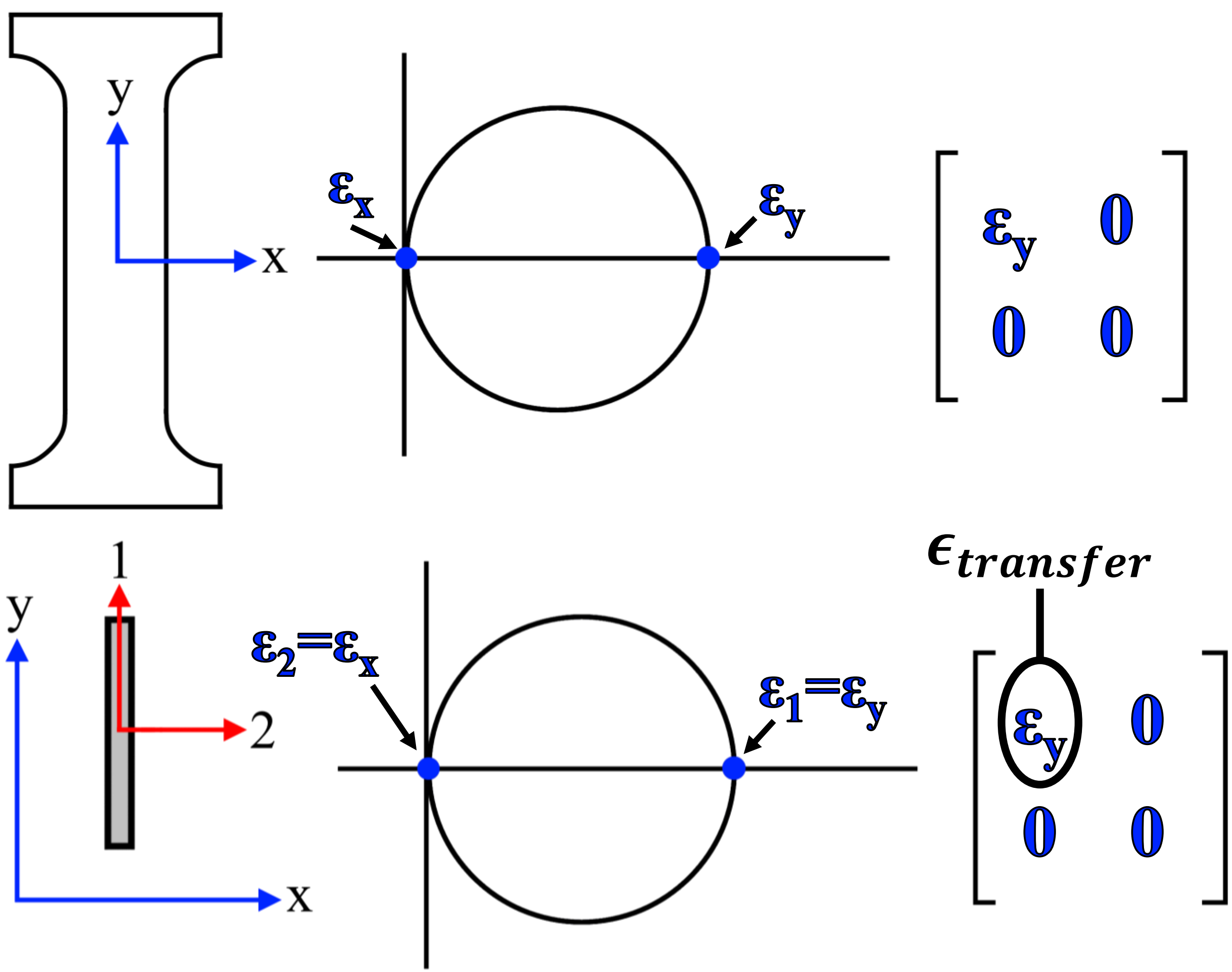
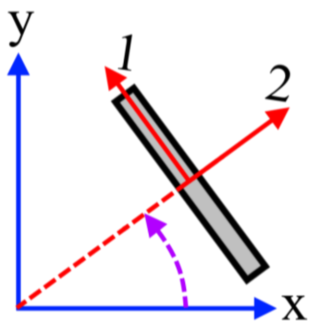
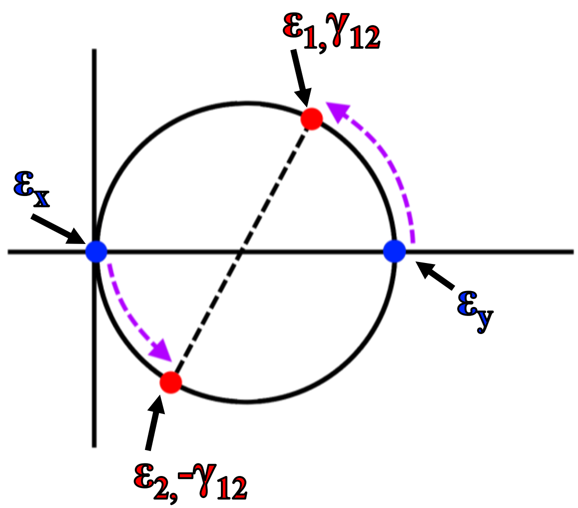



(200) type precipitate

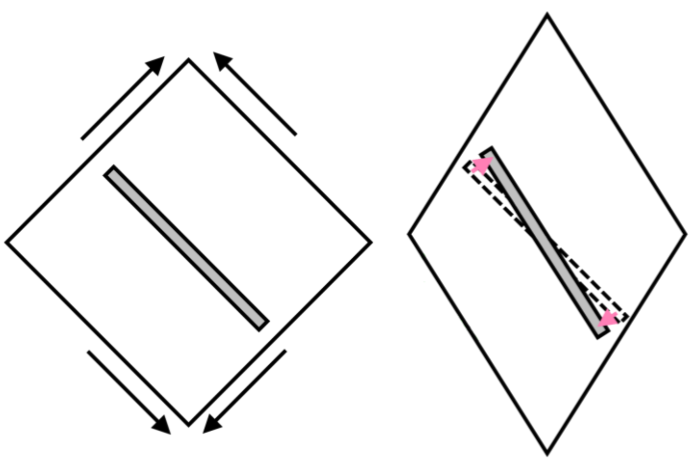
# Transfer Strain 2. Rotate Strain State to Precipitate Axes

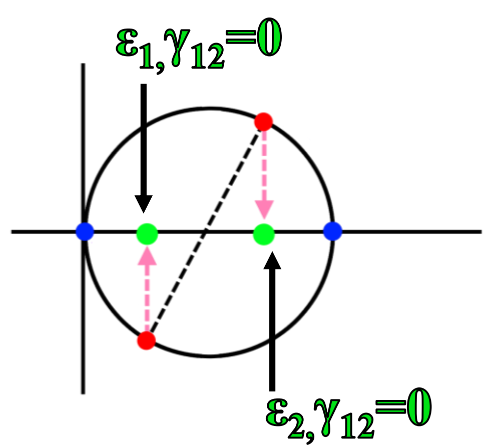
(111) type precipitate



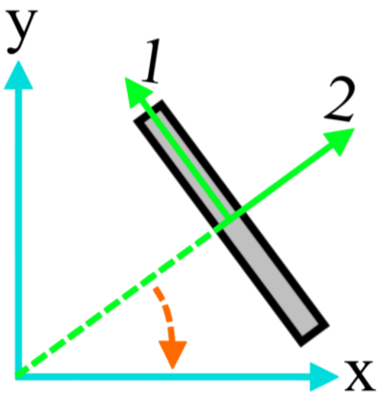

$$\begin{bmatrix} \varepsilon_y & 0 \\ 0 & 0 \end{bmatrix} \Rightarrow \begin{bmatrix} .9\varepsilon_y & .3\varepsilon_y \\ -.3\varepsilon_y & .1\varepsilon_y \end{bmatrix}$$

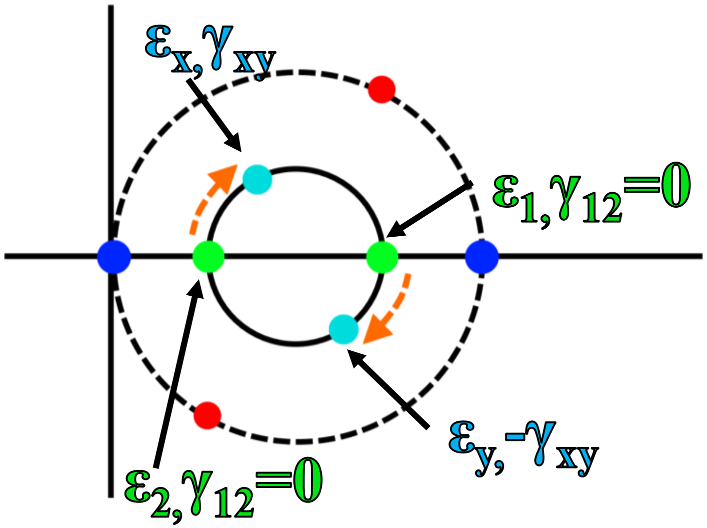





$$\begin{bmatrix} .9\varepsilon_y & .3\varepsilon_y \\ -.3\varepsilon_y & .1\varepsilon_y \end{bmatrix} \Rightarrow \begin{bmatrix} .9\varepsilon_y & 0 \\ 0 & .1\varepsilon_y \end{bmatrix}$$

# Transfer Strain 3. Rotate Back to Sample Axes



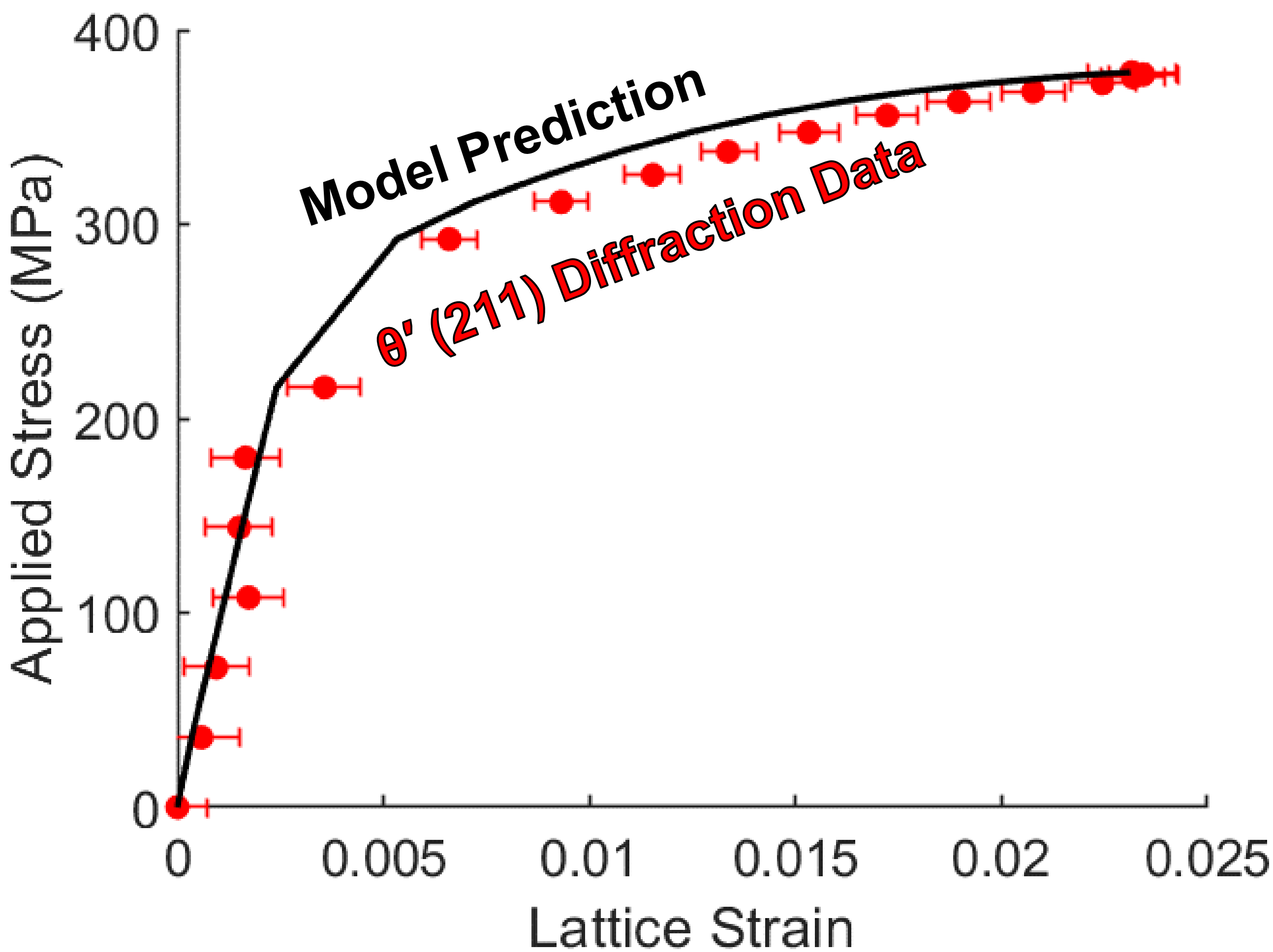




$$\begin{bmatrix} .9\varepsilon_y & 0 \\ 0 & .1\varepsilon_y \end{bmatrix} \Rightarrow \begin{bmatrix} .78\varepsilon_y & -.24\varepsilon_y \\ .24\varepsilon_y & .18\varepsilon_y \end{bmatrix}$$

$\epsilon_{transfer}$

| Precipitate | Rotation | $\epsilon_{transfer}$ |
|---|---|---|
| (200) | 0° | $\varepsilon_y$ |
| (111) | 36° | $0.78\varepsilon_y$ |

# Modeling Precipitate Rotation: Comparison to Experiments




Model attempted to predict behavior of precipitates in (422) grain orientation, data is for (211) θ′ precipitates

# Can we Predict Matrix Behavior?

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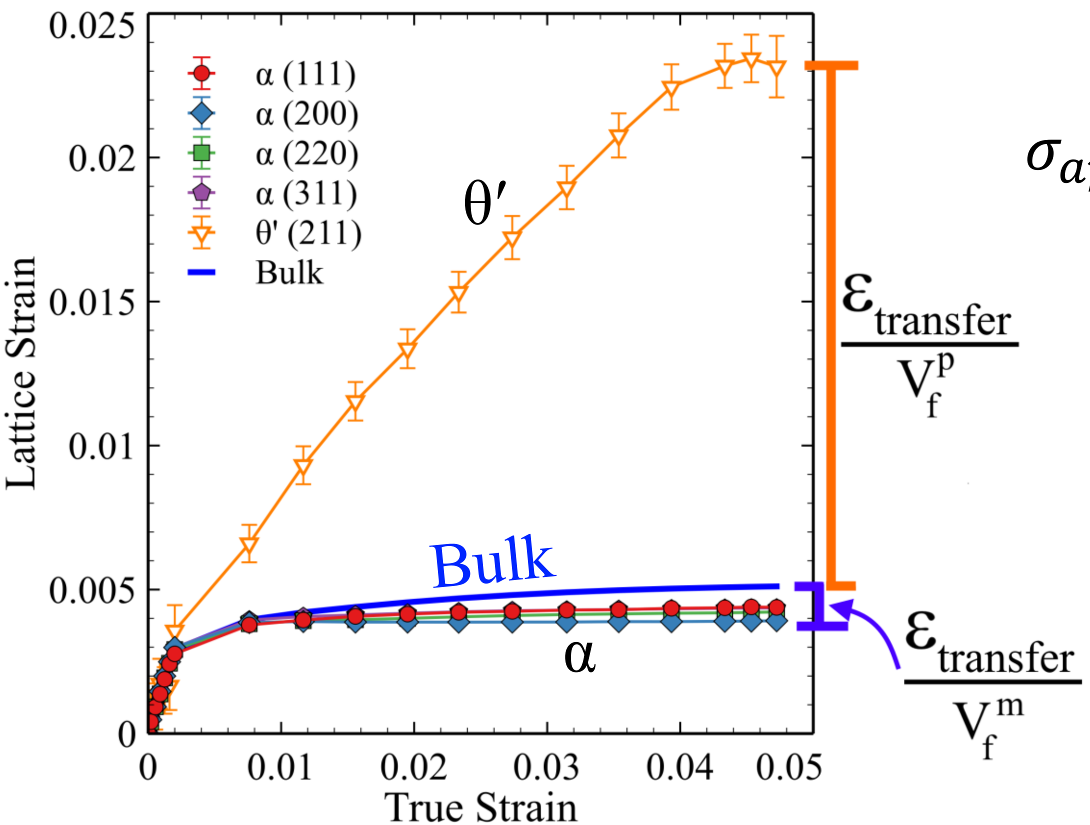
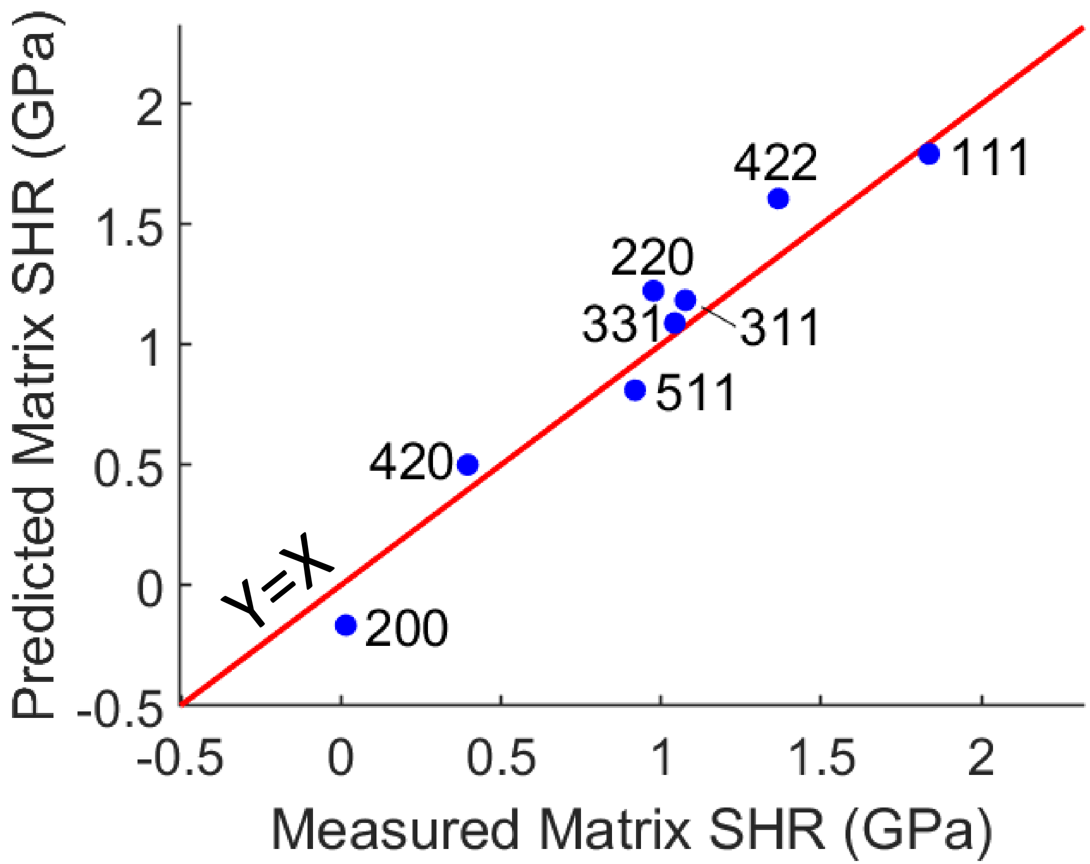



$$\sigma_{app} = \sigma_p V_f^p + \sigma_{matrix}^{hkl}(1 - V_f^p)$$

$$\sigma_{matrix}^{hkl} = \frac{\sigma_{app} - \sigma_p V_f^p}{1 - V_f^p}$$

Measured at 1.75% (STEM)

# Precipitate Rotation: Effect on Matrix Behavior




Measured from 0.5-3.5% strain

# Intro to RR350: Preliminary Work at Elevated Temperature

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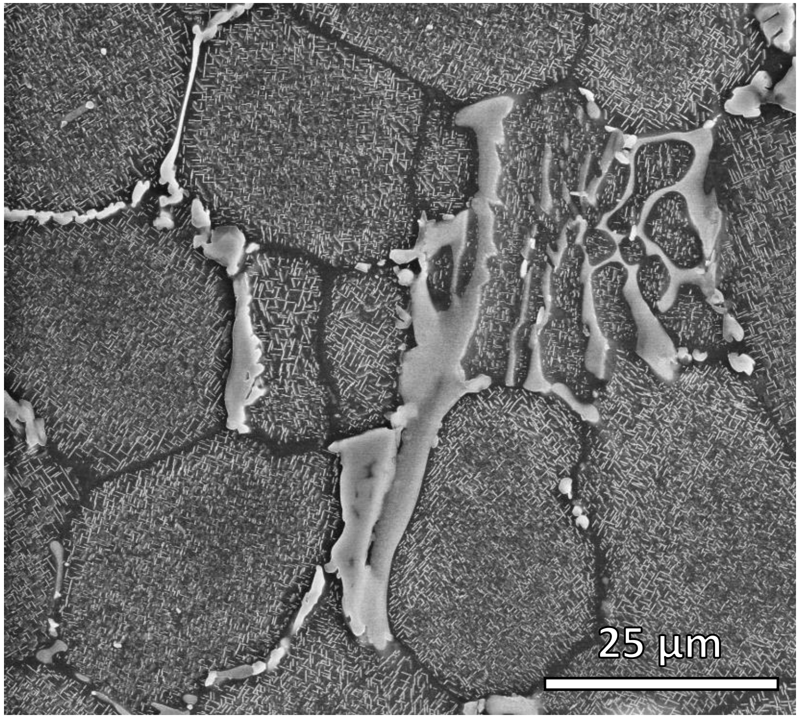
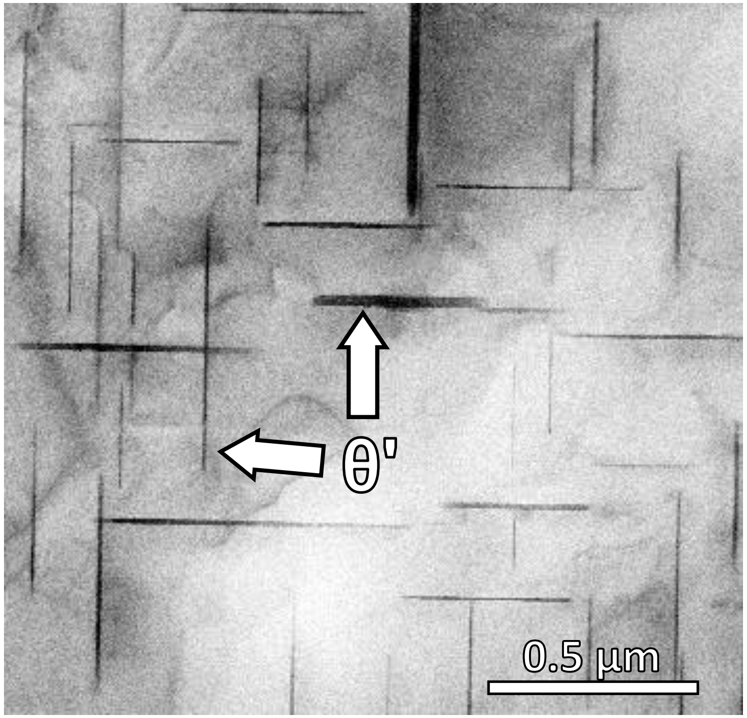
Composition of Alloy RR350 (wt%)

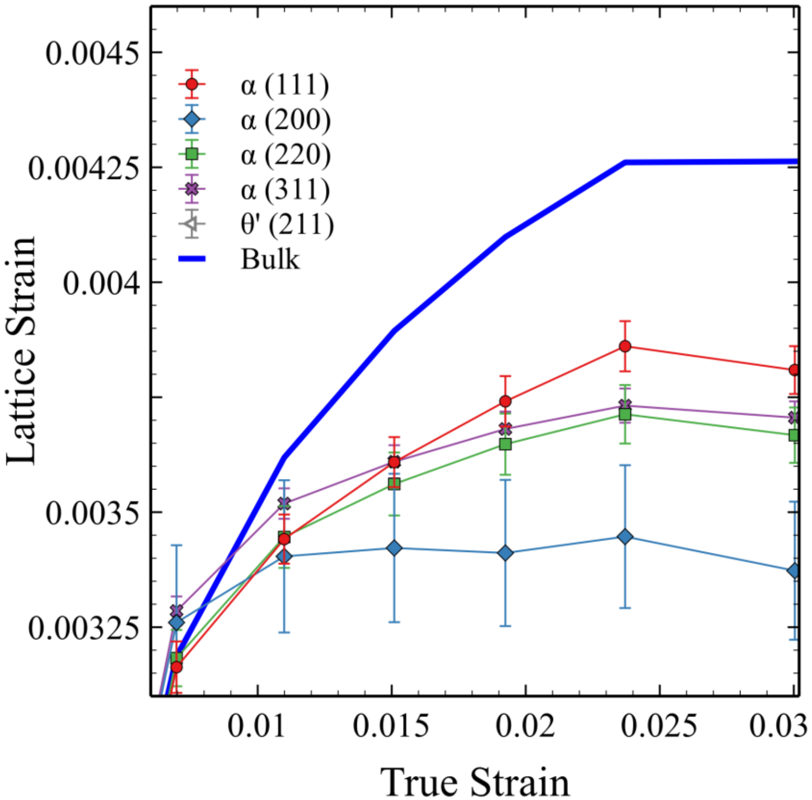
| Cu | Mn | Zr | Si | Zn | Fe | Ni | Co | Ti | Sb |
|---|---|---|---|---|---|---|---|---|---|
| 4.8 | 0.19 | 0.17 | 0.05 | 0.01 | 0.09 | 1.2 | 0.26 | 0.21 | 0.17 |

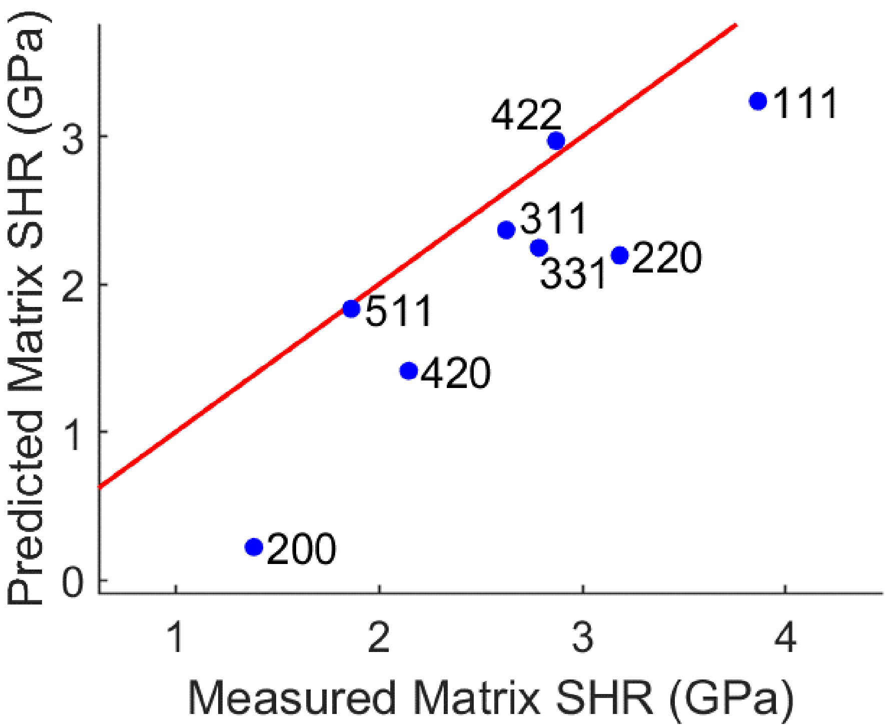


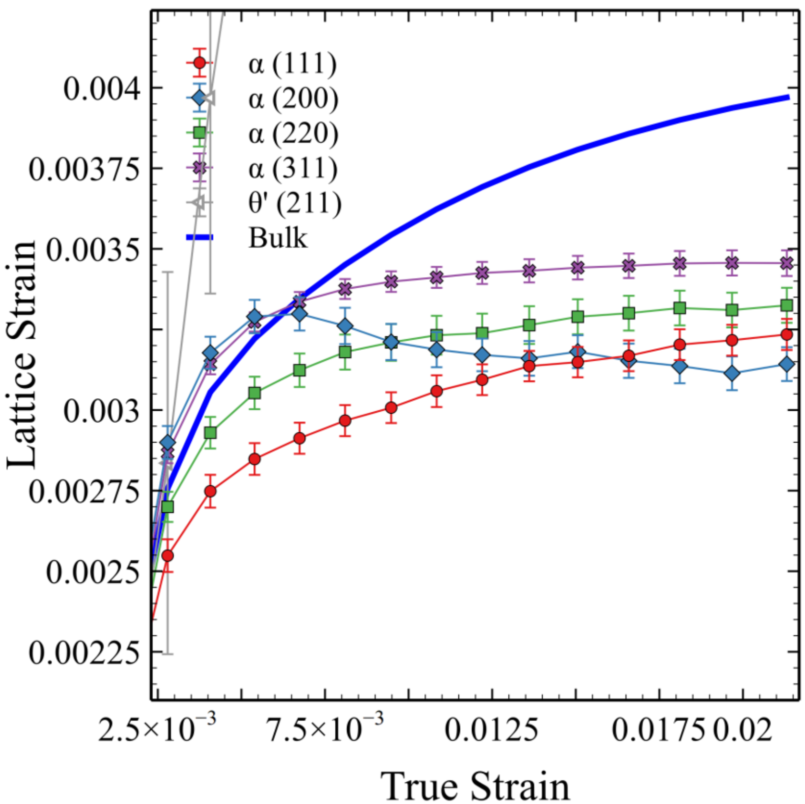

| Solutionize | Water Quench | Age | Overage |
|---|---|---|---|
| 535°C for 12hr | 80-90°C | 240°C for 5hr | Test Temp. for 200hr |

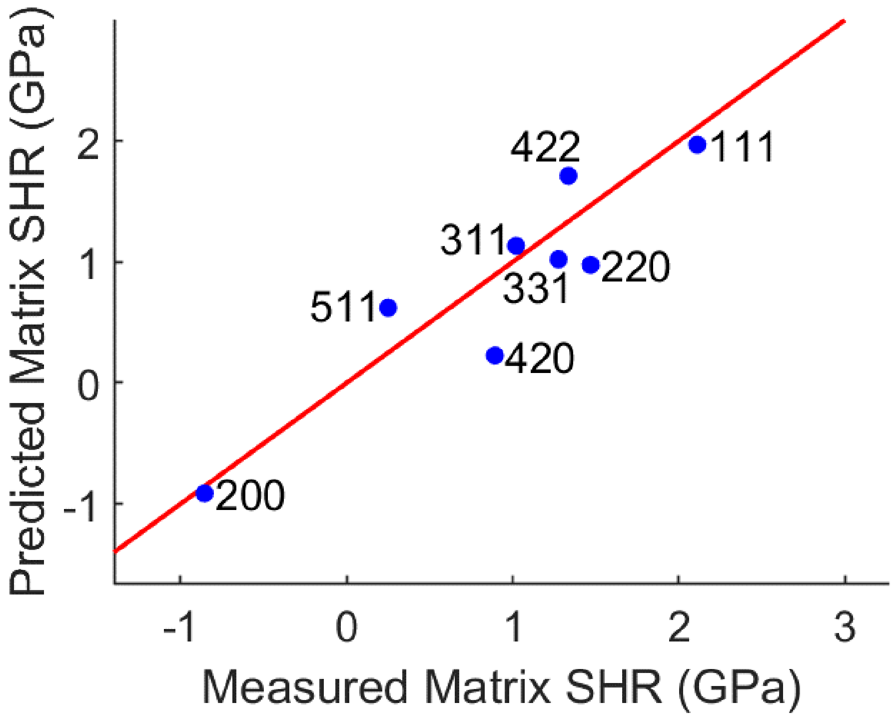
# RR350 As Aged Room Temperature Tension: Similar to 206



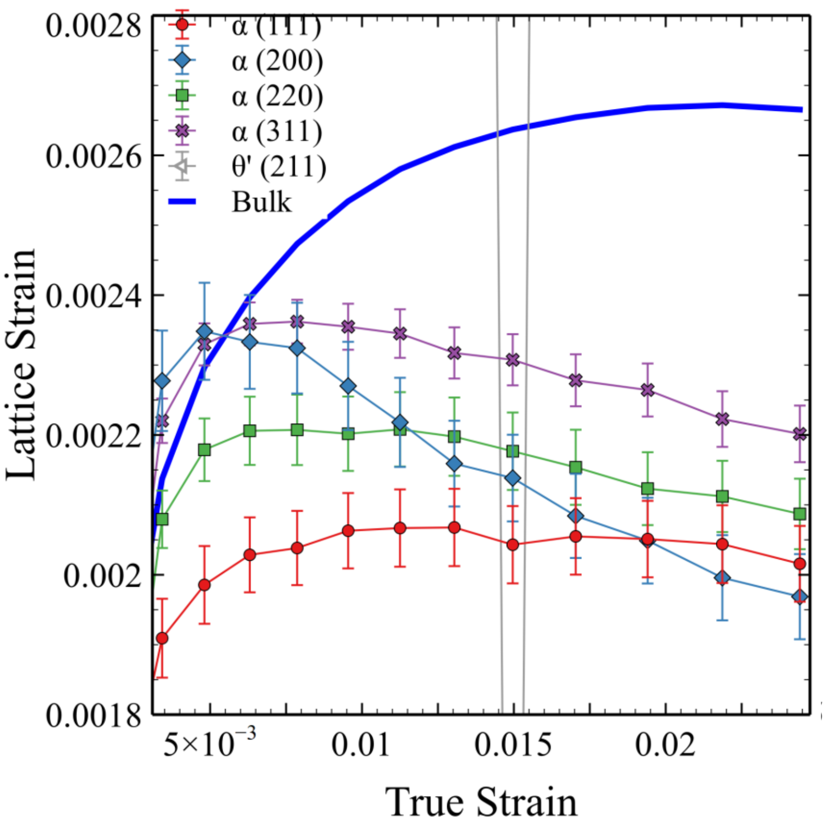



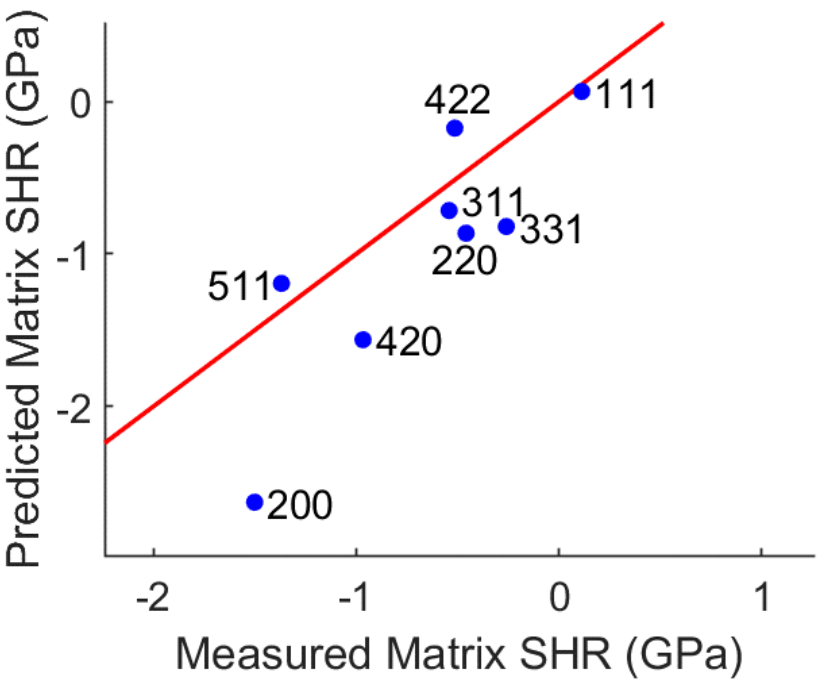
# RR350 100°C Tension: Similar to Room Temp.



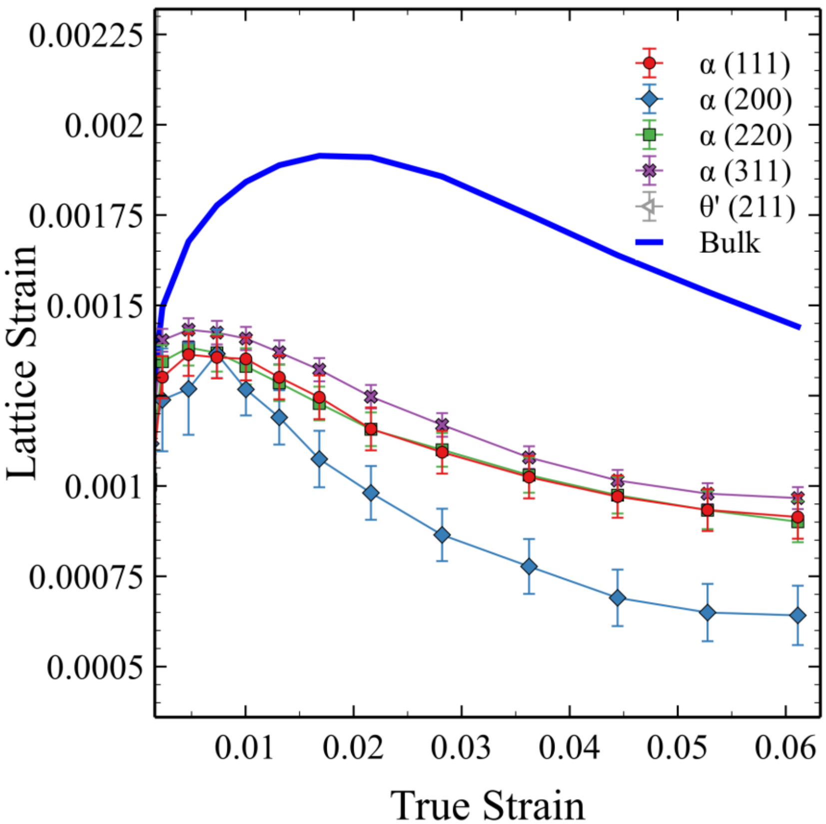



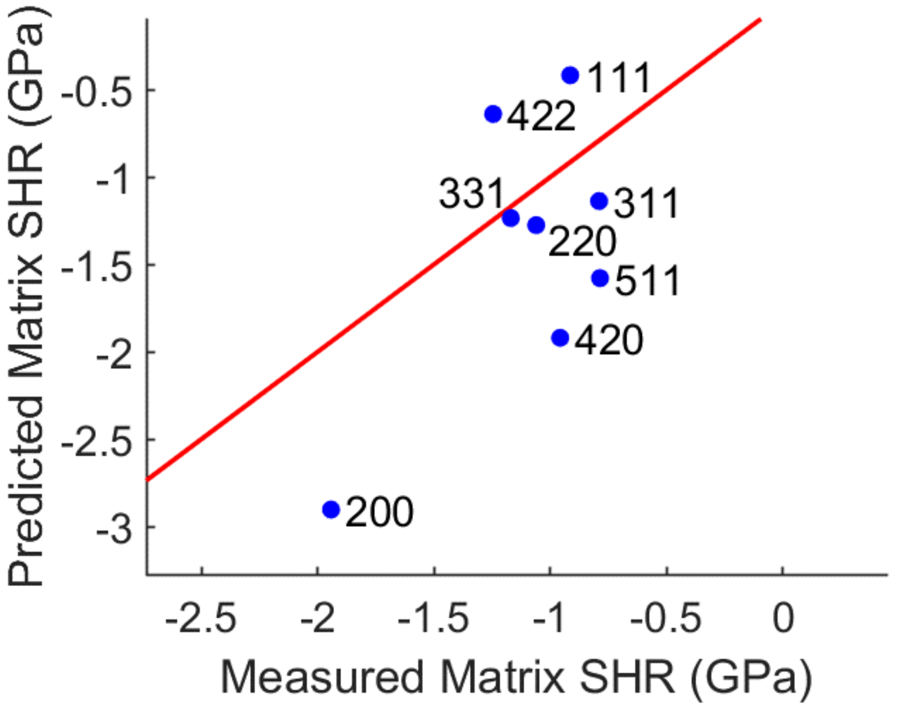
# RR350 200°C Tension: Anisotropic; Less Load Transfer



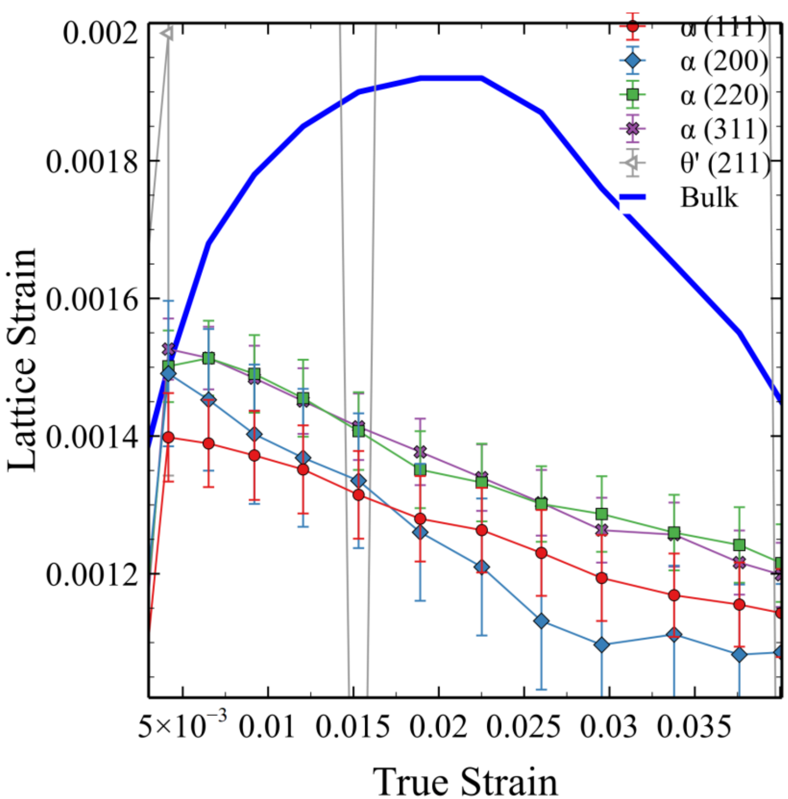



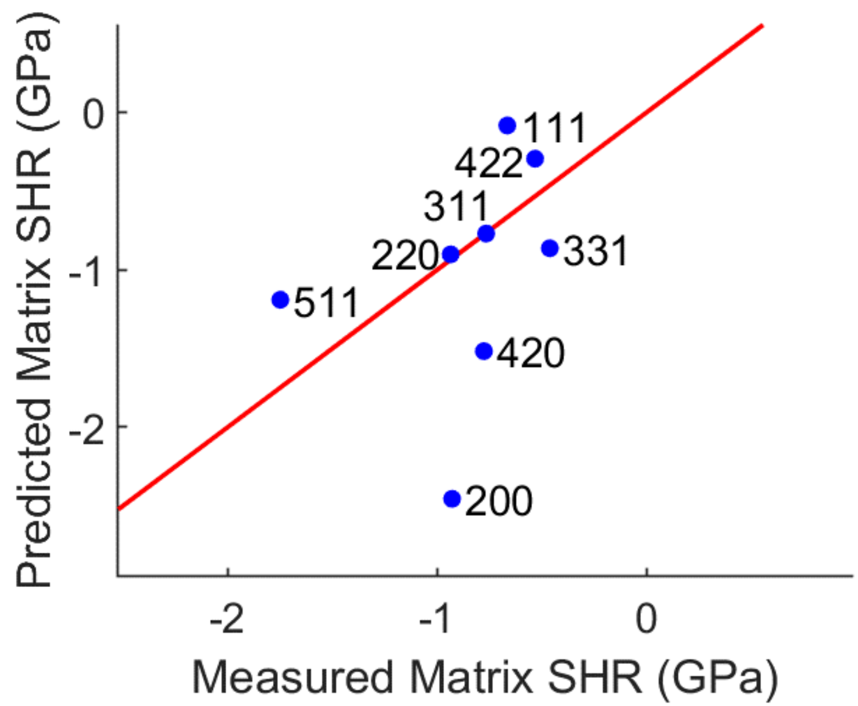
# RR350 300°C Tension: Nearly Isotropic; Less Load Transfer



# RR350 350°C Tension: Nearly Isotropic; θ′ Transforming

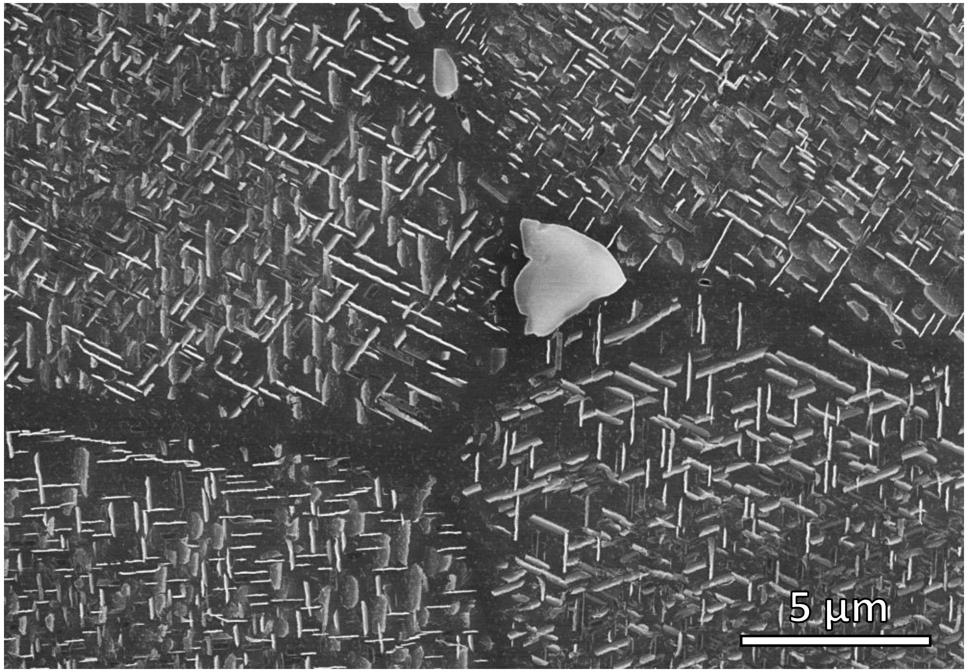


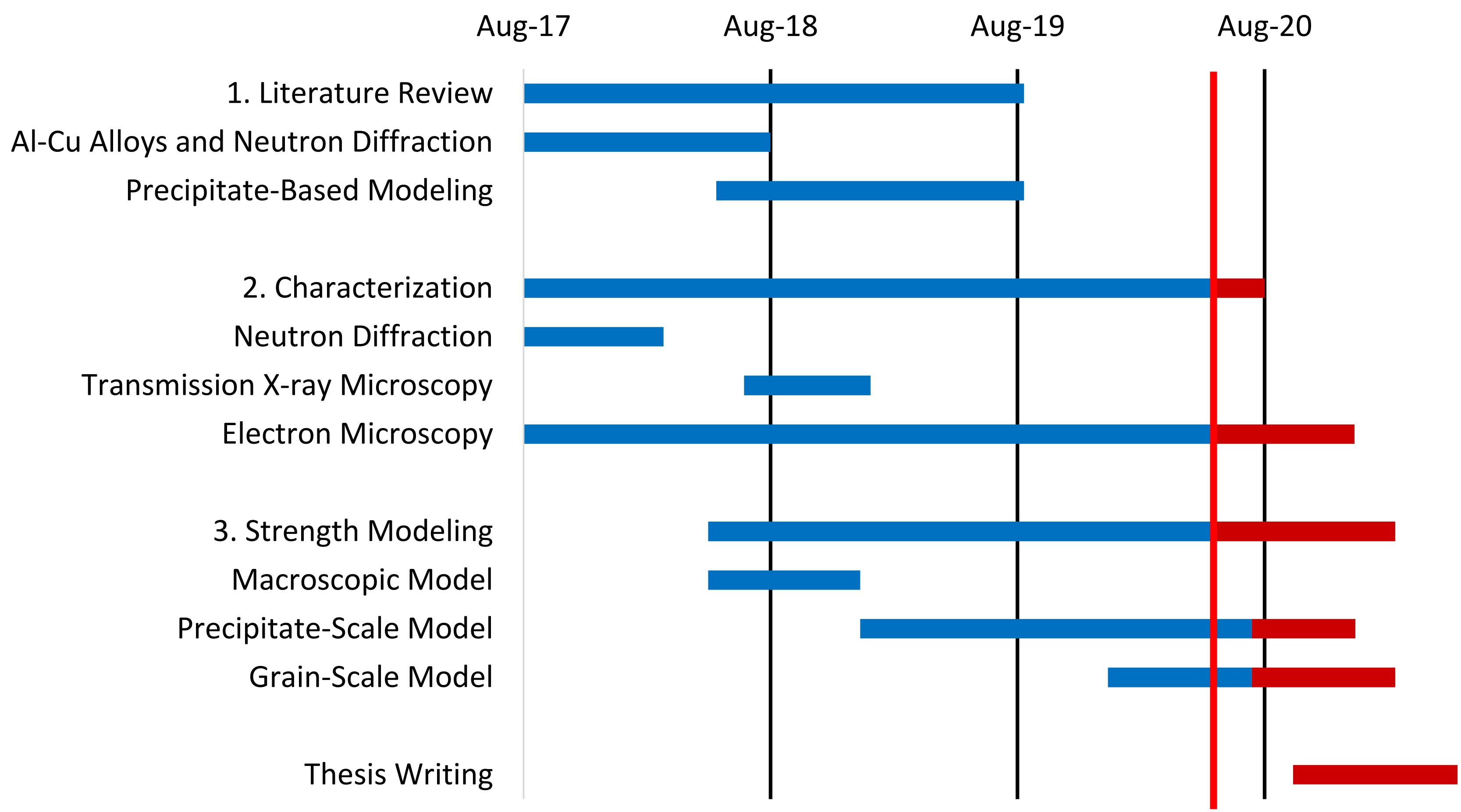
# Conclusions and Limitations of High Temperature Tests

- Model still works up to 200°C as-is
- Higher temperature?
  - Precipitates might yield at lower stress
  - Recovery might mitigate load transfer
  - Microstructure changes at 350°C

RR350 350°C overage



# Progress


Aug-17
Aug-18
Aug-19
Aug-20
1. Literature Review
Al-Cu Alloys and Neutron Diffraction
Precipitate-Based Modeling
2. Characterization
Neutron Diffraction
Transmission X-ray Microscopy
Electron Microscopy
3. Strength Modeling
Macroscopic Model
Precipitate-Scale Model
Grain-Scale Model
Thesis Writing

# Challenges & Opportunities

- Applying this model to other systems with plate-shaped precipitates may be interesting
  - Ex: $\gamma''$ strengthened superalloys (like Inconel 718)
- Perfect load transfer and precipitate rotation does not seem to explain higher temperature behavior
  - Recovery and climb may reduce load transfer
- Prediction of fracture behavior?
  - Local, individual phase stress will be more useful than aggregate stress in predicting crack initiation of grain boundary precipitates or voids

Thank you!

Brian Milligan
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# References


[1] S. Ma *et al*, CRSS of γ-γ′ phases from in situ neutron diffraction of a directionally solidified superalloy tension tested at 900°C, Acta Materialia 56 (2008) 4102-4113

[2] S. D. Dahlgren, Coherency Stresses, Composition and Dislocation Interactions for θ" Precipitates in Age-Hardened Al-Cu, Metallurgical and Materials Transactions A 7 (1976) 1401-1405

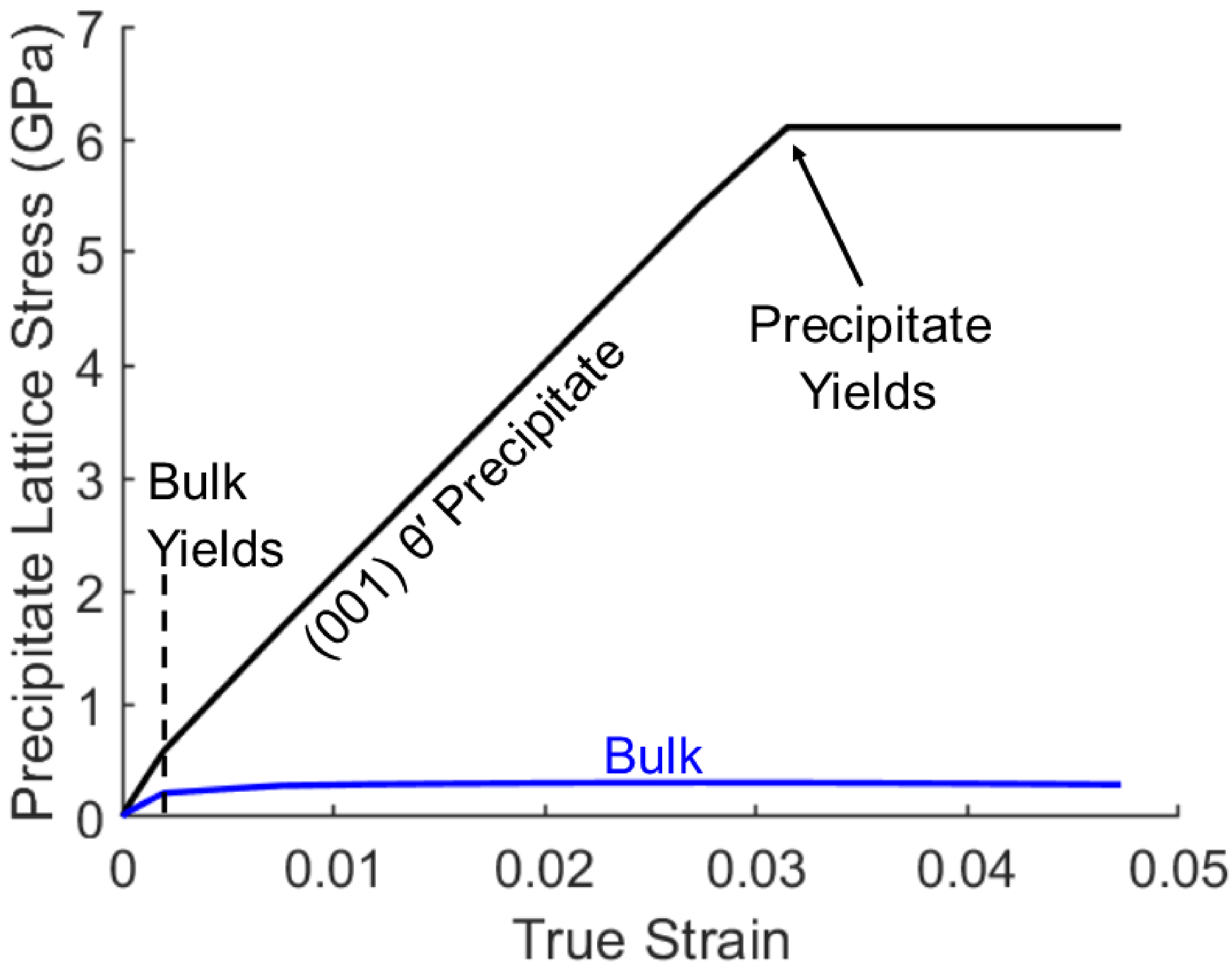
[3] I. Adlakha *et al*, Revealing the atomistic nature of dislocation-precipitate interactions in Al-Cu alloys, Journal of Alloys and Compounds 797 (2019) 325-333

[4] S. Bahl *et al*, Effect of copper content on the tensile elongation of Al–Cu–Mn–Zr alloys: Experiments and finite element simulations, Materials Science and Engineering A (2020) 138801

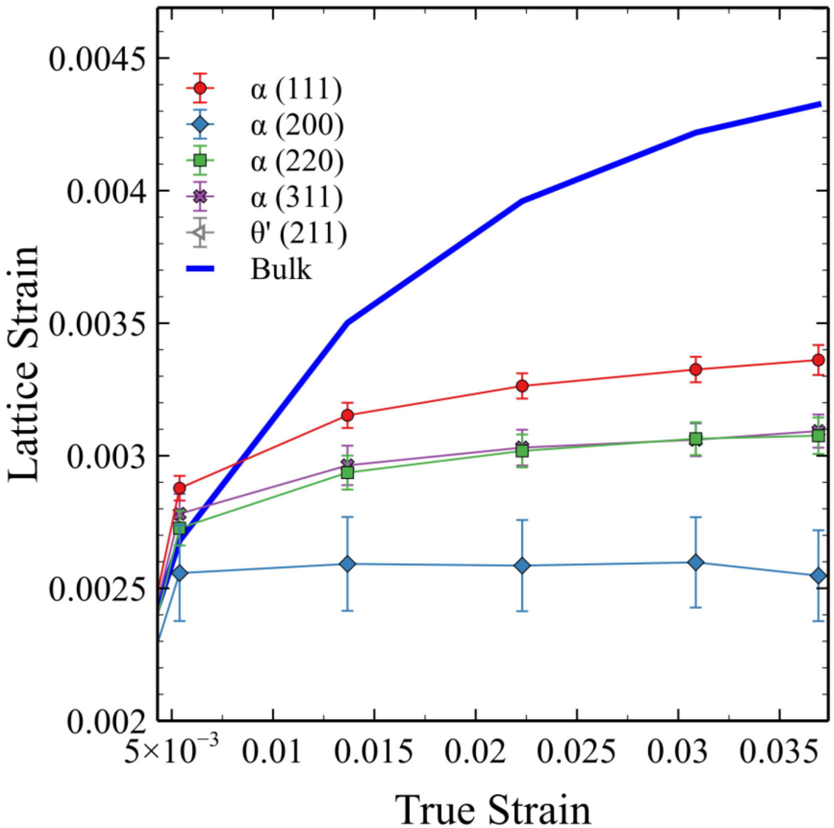
[5] P. Shower *et al*, The effects of microstructural stability on the compressive response of two cast aluminum alloys up to 300 °C, Materials Science and Engineering A 700 (2017) 519-527

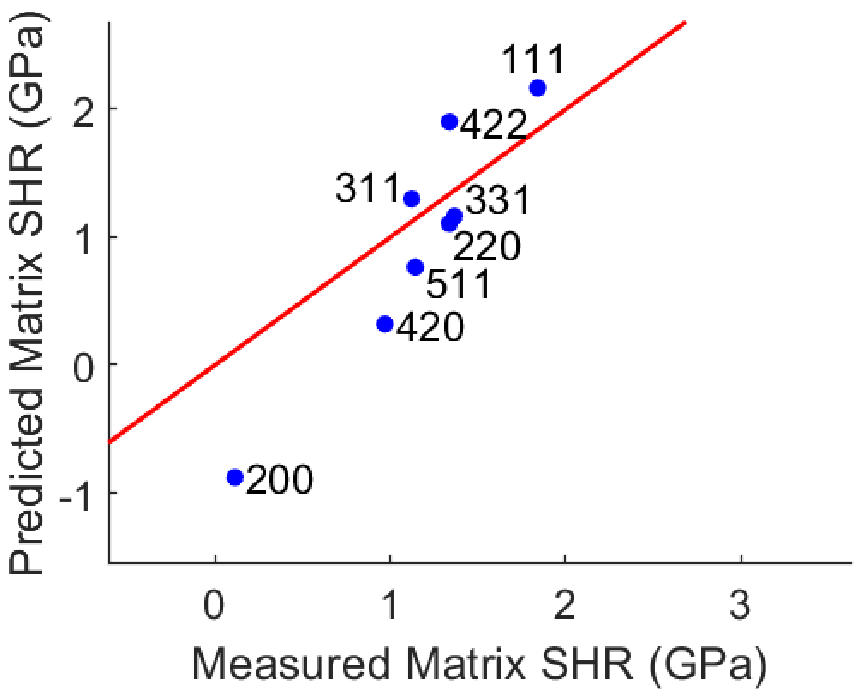
[6] W. F. Hosford, R. H. Zeisloft, The anisotropy of age-hardened Al-4 pct Cu single crystals during plane-strain compression, Metallurgical Transactions 3 (1972) 113-121

# What if the Precipitates Yield?



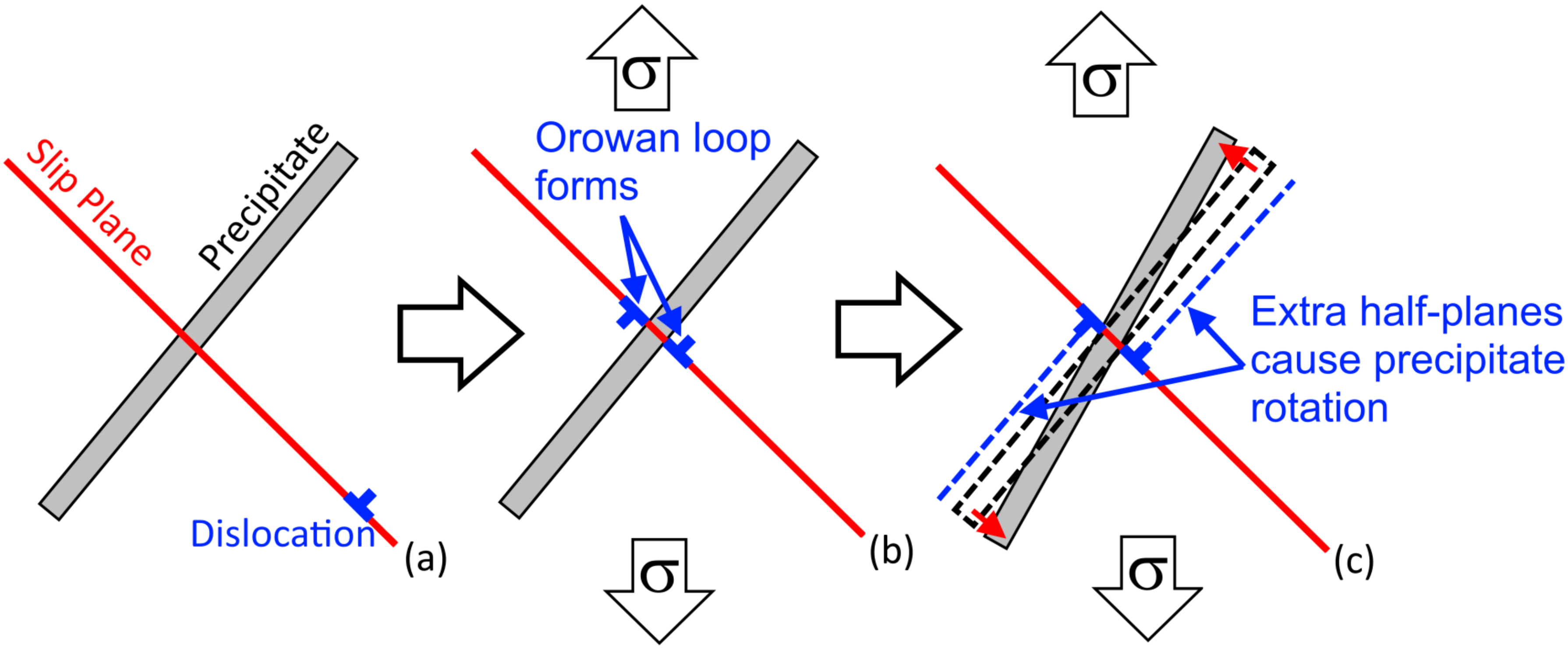
# RR350 300° Overaged RT Tension:



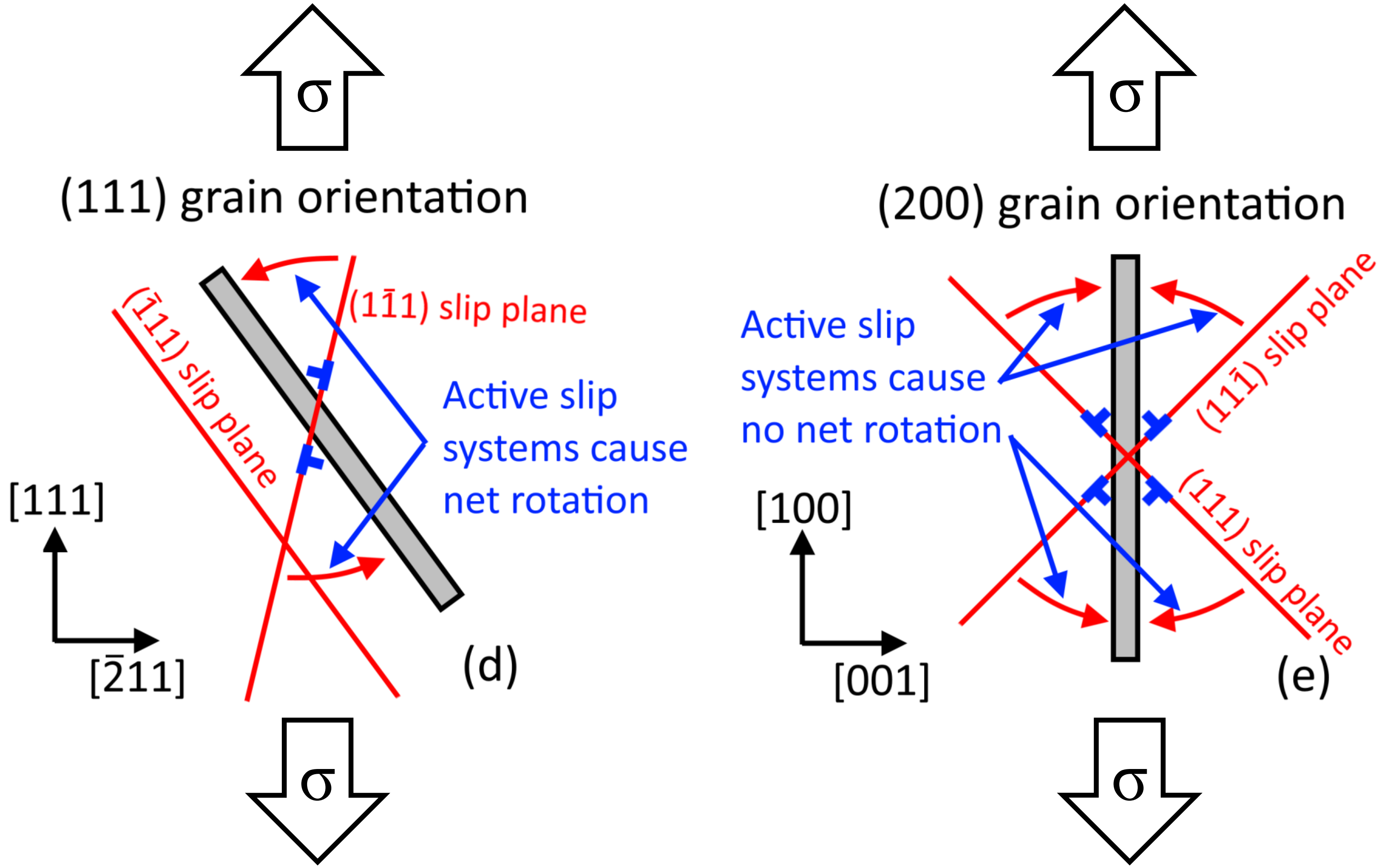



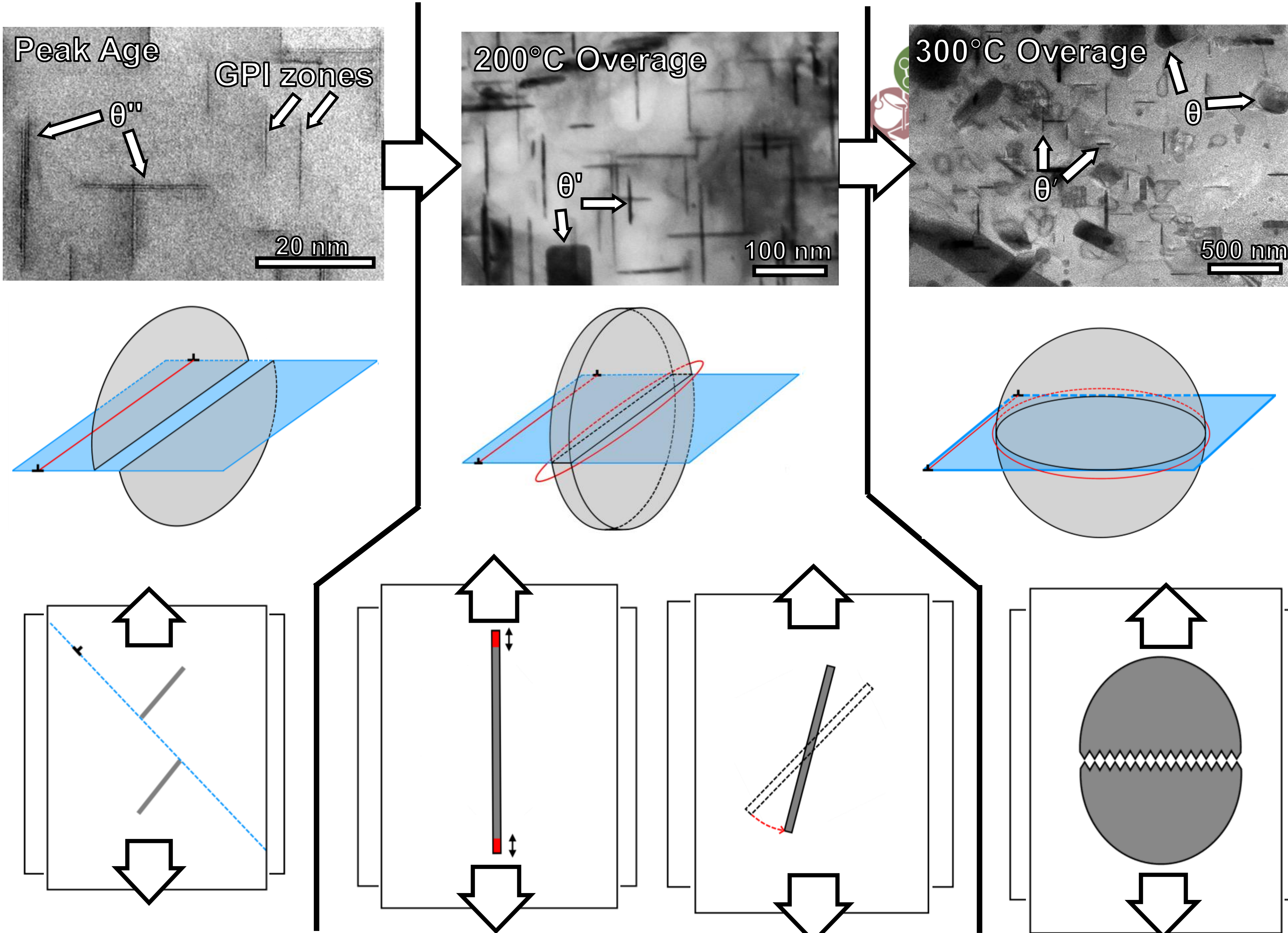

Note: Measured 0.5-3.5% strain due to chopping issues

# Dislocation Mechanism for Precipitate Rotation



# Anisotropic Rotation Dislocation Mechanism





Peak Age
GPI zones
θ''
20 nm
200°C Overage
θ'
100 nm
300°C Overage
θ
θ'
500 nm

# Acknowledgements

Research sponsored by:

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- The Department of Energy, Energy Efficiency & Renewable Energy, Vehicles Technologies Office (VTO), Powertrain Materials Core Program (Jerry Gibbs, VTO Materials)

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