Project 29: Identification of Deformation Mechanisms of Thermally Stable Cast Al-Cu Alloys via Neutron Diffraction (Leveraged)

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

Student: Brian Milligan (Mines)
Faculty: Amy Clarke (Mines)
Industrial Mentor(s): Amit Shyam (ORNL)
Other Participants : Dong Ma (ORNL), Lawrence Allard (ORNL), Francisco Coury (Mines)

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

Project Duration

PhD: August 2017 to May 2021

Problem

Thermally stable cast Al-Cu alloys developed at ORNL require characterization of mechanical properties.

Objective

Apply in-situ neutron diffraction, SEM, TEM, and traditional mechanical testing to better understand the mechanical behavior of these alloys.

Benefit

Improvement of properties of these alloys, as well as furthering scientific understanding of precipitation strengthened Al alloys.

Recent Progress

- Paper on creep properties of Al alloys at 300 and 350 °C in preparation.
- Paper on room temperature deformation mechanisms in 206 Al in preparation.
- Identified deformation mechanisms in 206 Al under various aging conditions
- Quantification of precipitate cutting underway.
- User proposal submitted to APS at Argonne National Laboratory to study precipitation and growth kinetics using TXM.

Metrics		
Description	**% Complete**	**Status**
1. Literature review	80%	●
2. *In situ* neutron diffraction at the SNS, and creep testing at CSM and ORNL	80%	●
3. Microstructural characterization pre- and post- creep and tension	60%	●
4. Analysis of neutron diffraction data	80%	●
5. Development of models for grain orientation-dependent tensile and creep properties	40%	●

206 Alloy Composition and Aging Conditions

206 Al composition

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

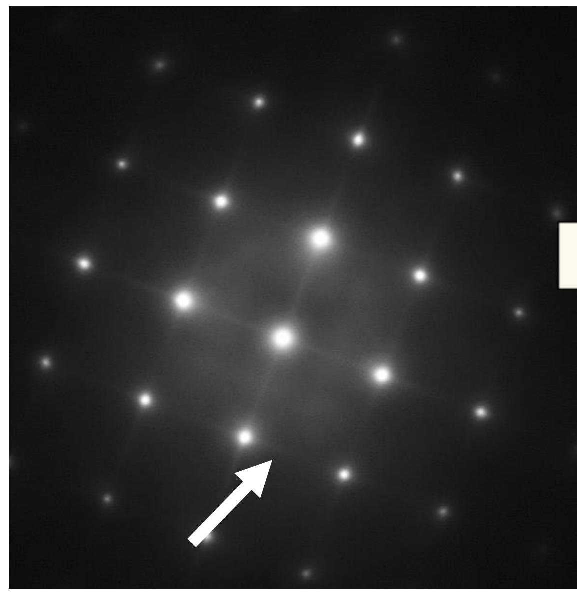
- 206 Al is a common Al-Cu alloy usually strengthened with GPI and GPII precipitates
- Various heat treatments have been applied to study the mechanical properties with various microstructures

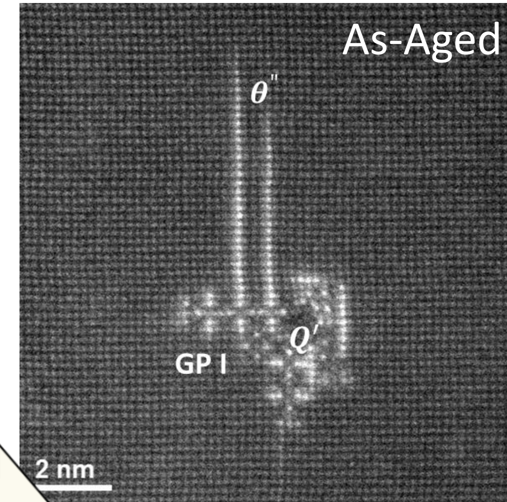
Heat treatment schedule

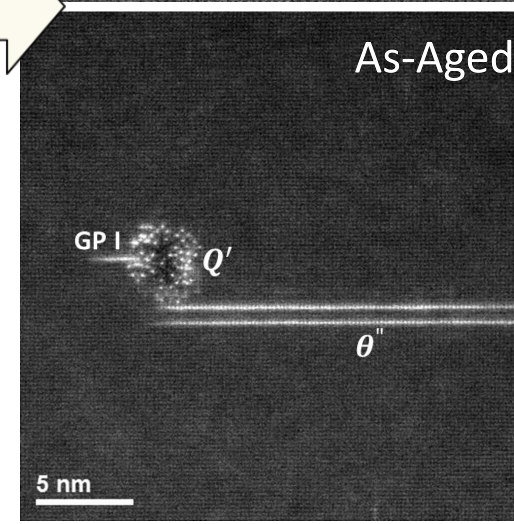
Step	Solutionizing	Quench	Peak Aging	Overaging
Temperature (°C)	500	80-90	190	200, 250, 300
Time (h)	1	<1	5	200

As-Quenched and Aged Microstructures (GP Zone-Strengthened Conditions)

- As-quenched and aged at 190 °C for 5 h
- Both conditions primarily strengthened by GP zones
  - As-quenched is a supersaturated solid solution with GPI zones
  - As-aged contains GPI and GPII zones



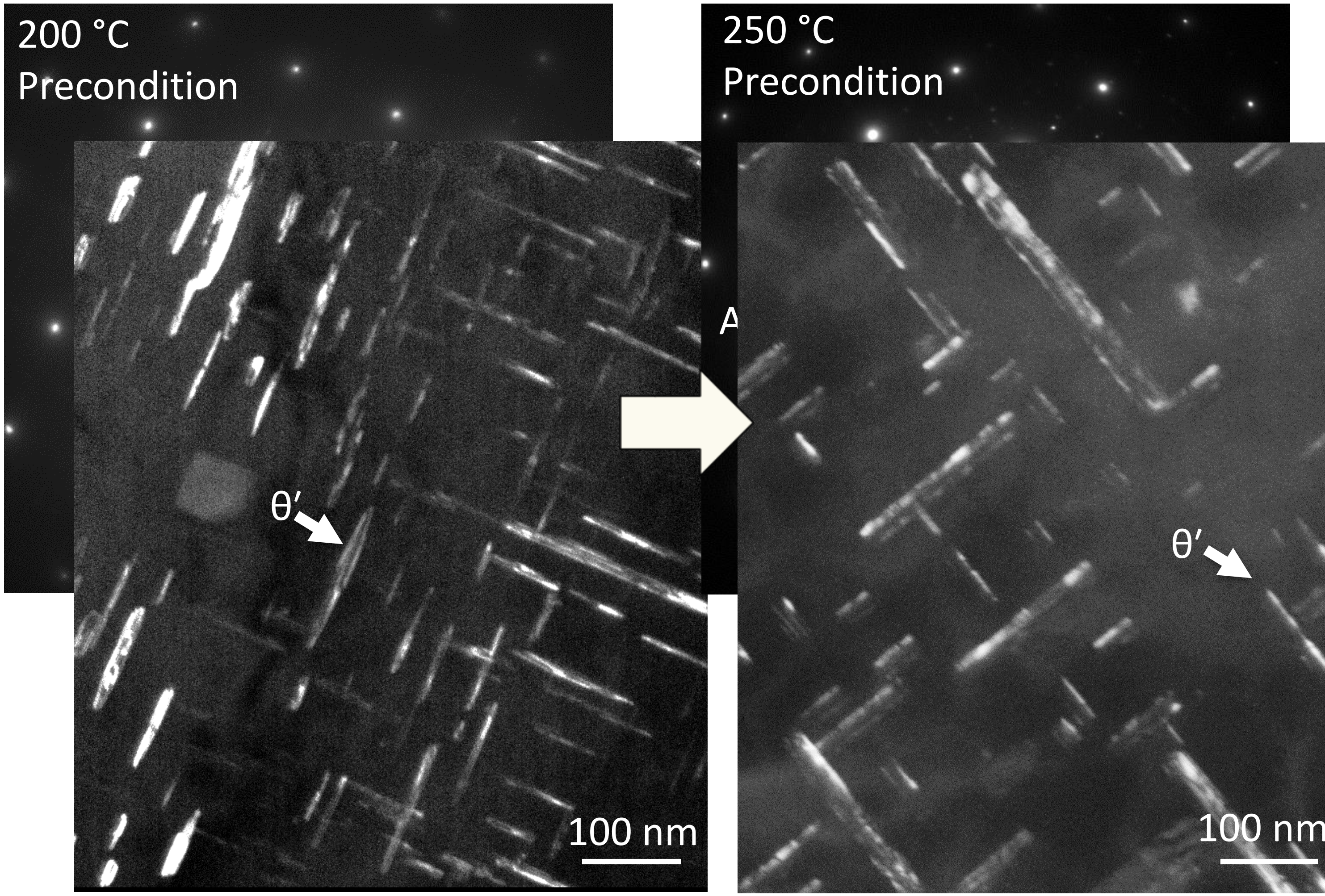




Zone axis (001) in all images.

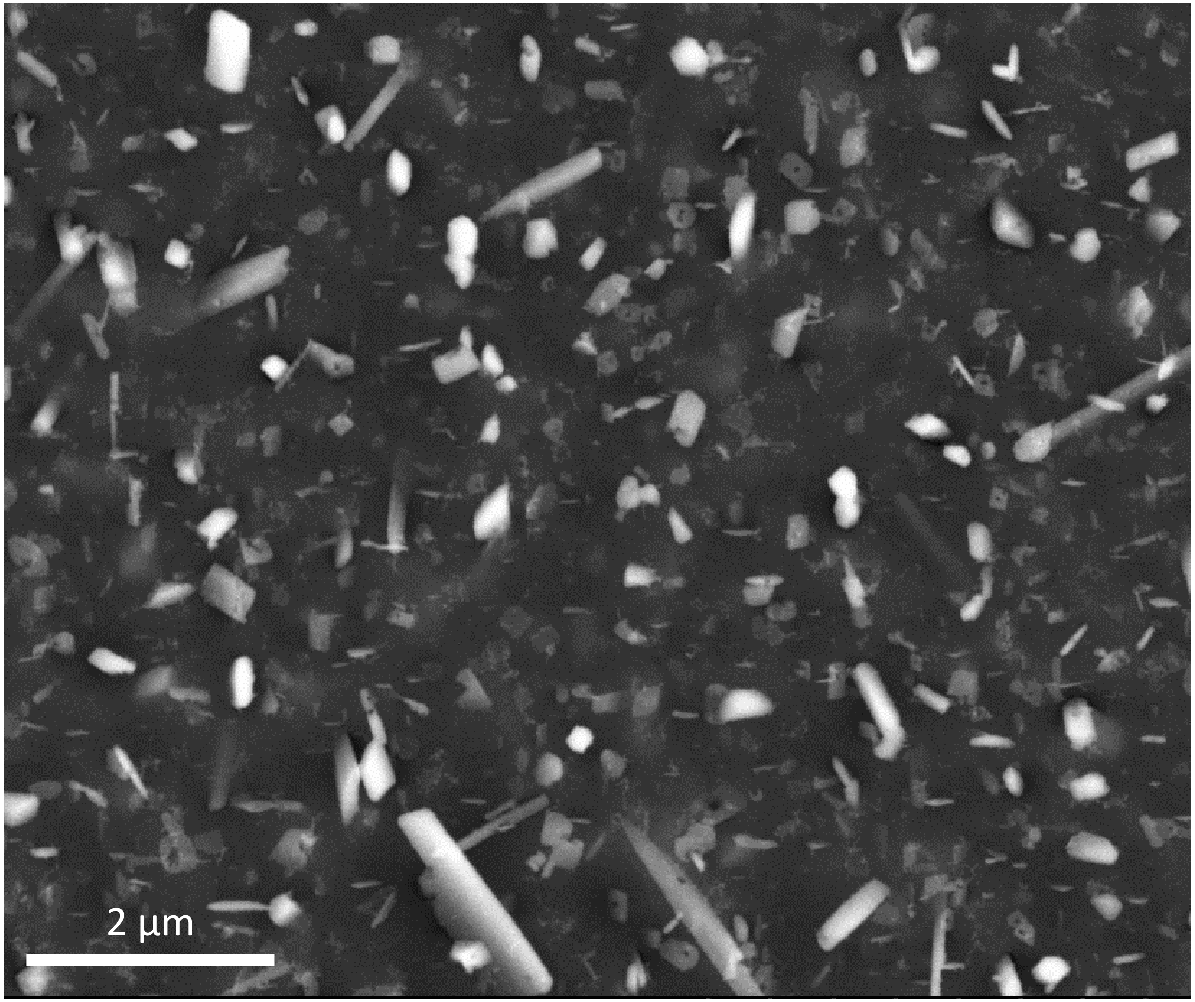
200 and 250 °C Overaged Microstructures (θ'-Strengthened Conditions)

- Aged at 190 °C for 5 h & overaged at 200 or 250 °C for 200 h
- Primarily θ′ in both conditions

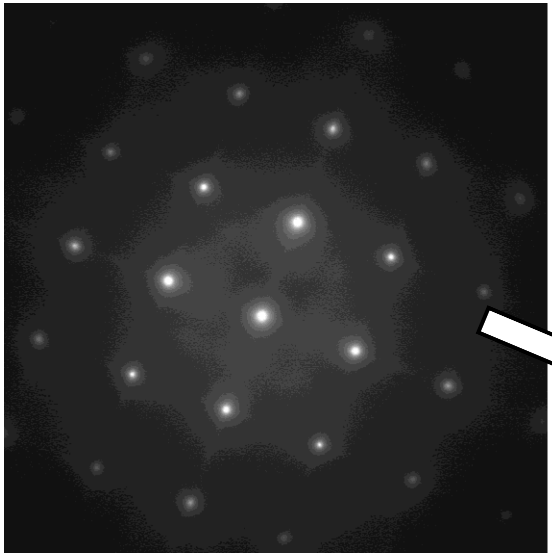


300 °C Overaged Microstructure (Mostly θ)

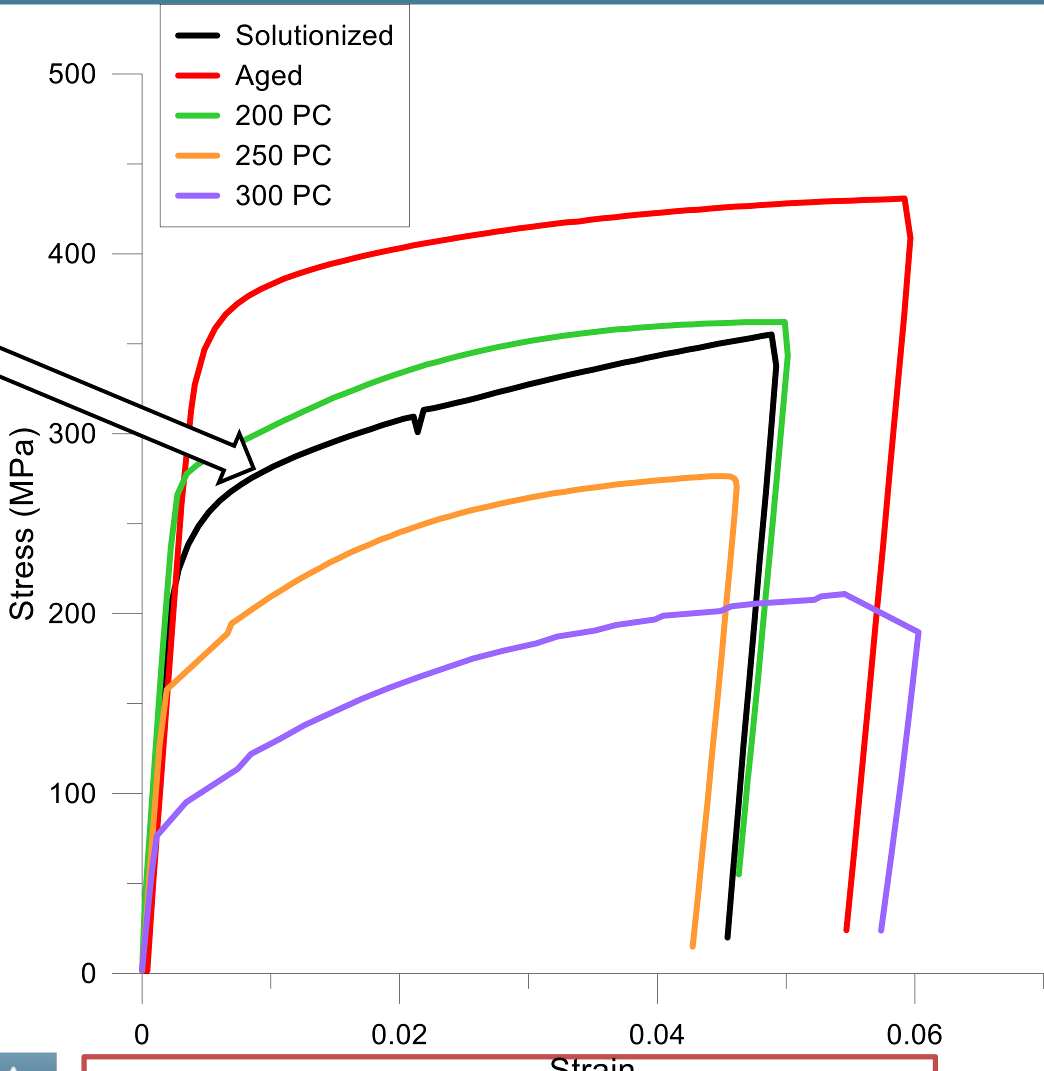
- Aged at 190 °C for 5 h and overaged at 300 °C for 200 h
  - Mostly θ
  - Coarse θ is less effective at strengthening than GP zones or θ′
- Some coarse retained θ′



Bulk Tensile Results



Condition	0.2% YS (MPa)	Precipitates
As-Quenched	255	GPI
Peak Aged	365	GPI, GPII, θ″
200 °C Overaged	290	θ″, θ′
250 °C Overaged	175	θ′
300 °C Overaged	95	θ



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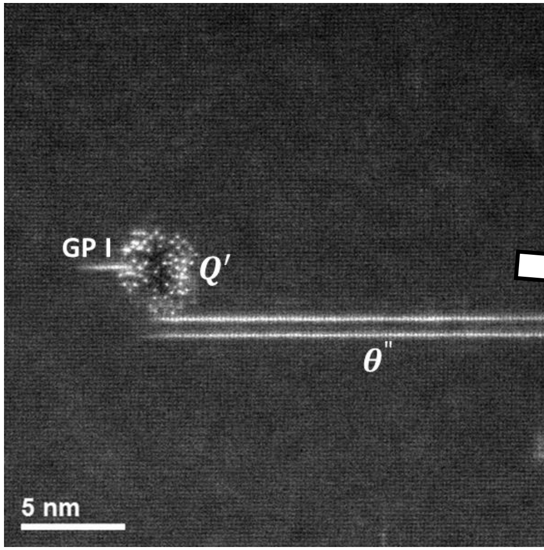

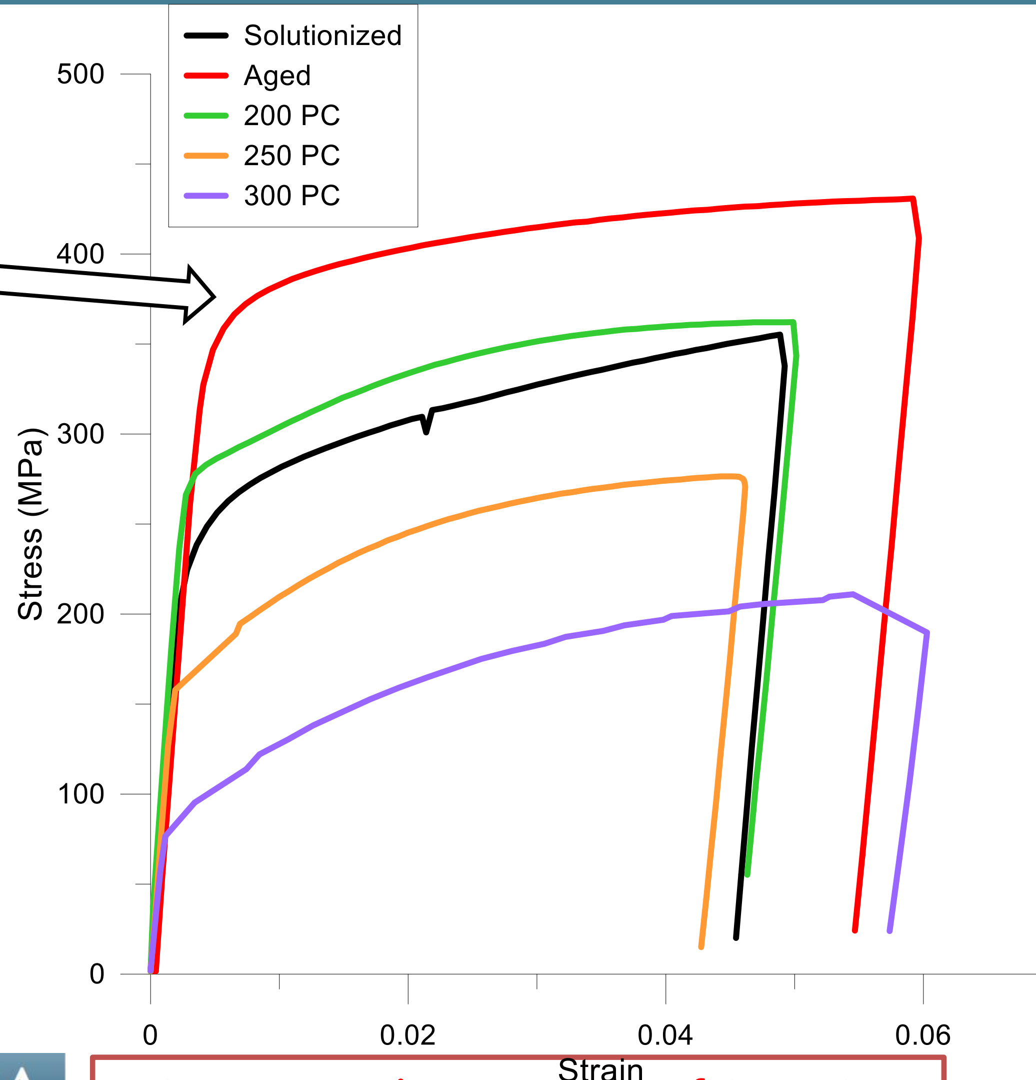

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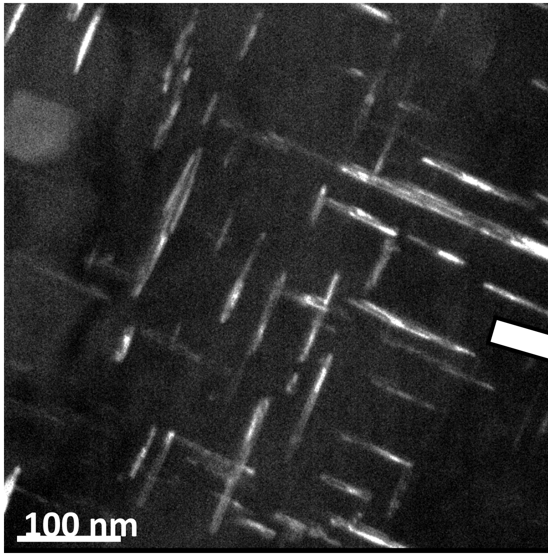

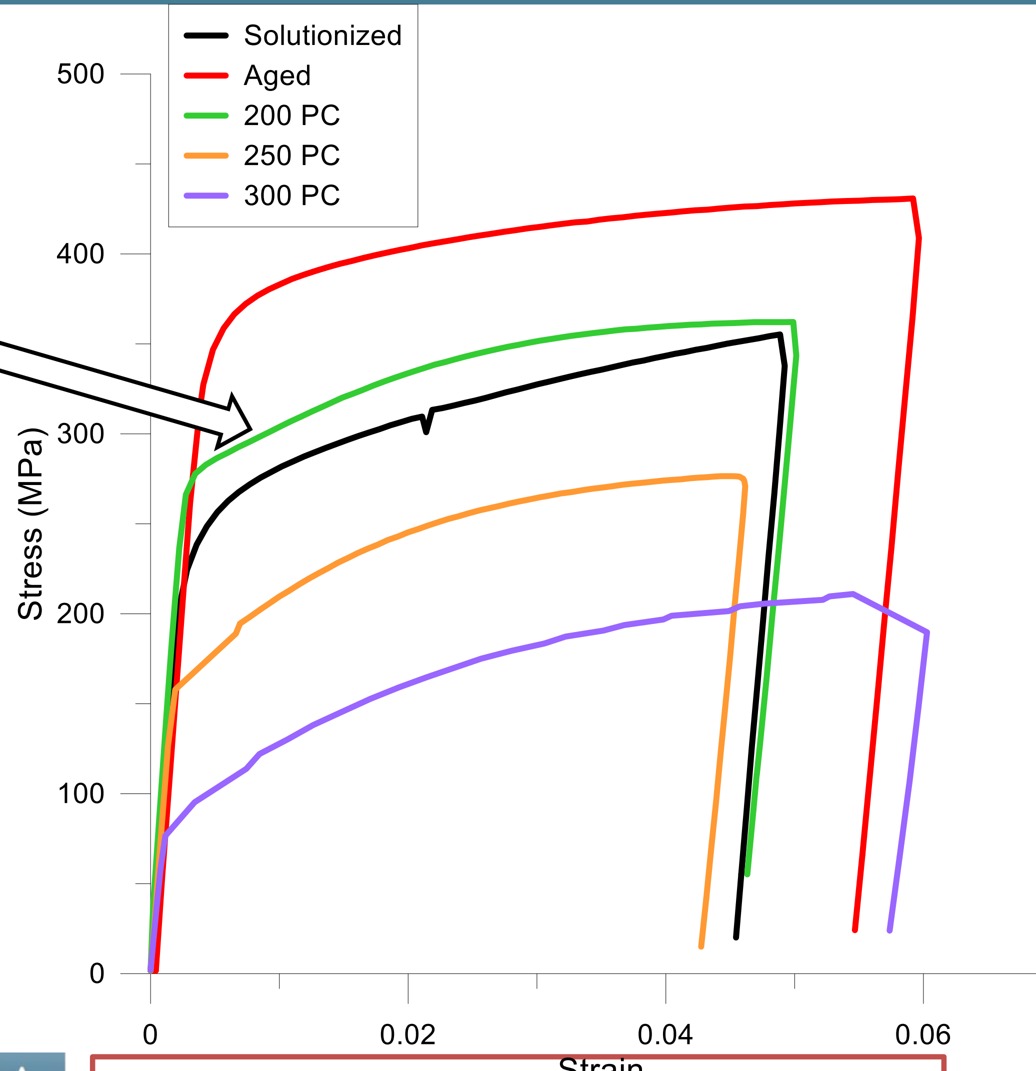

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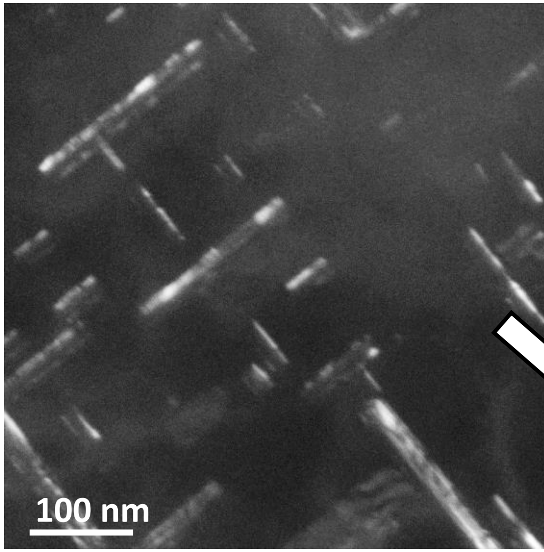

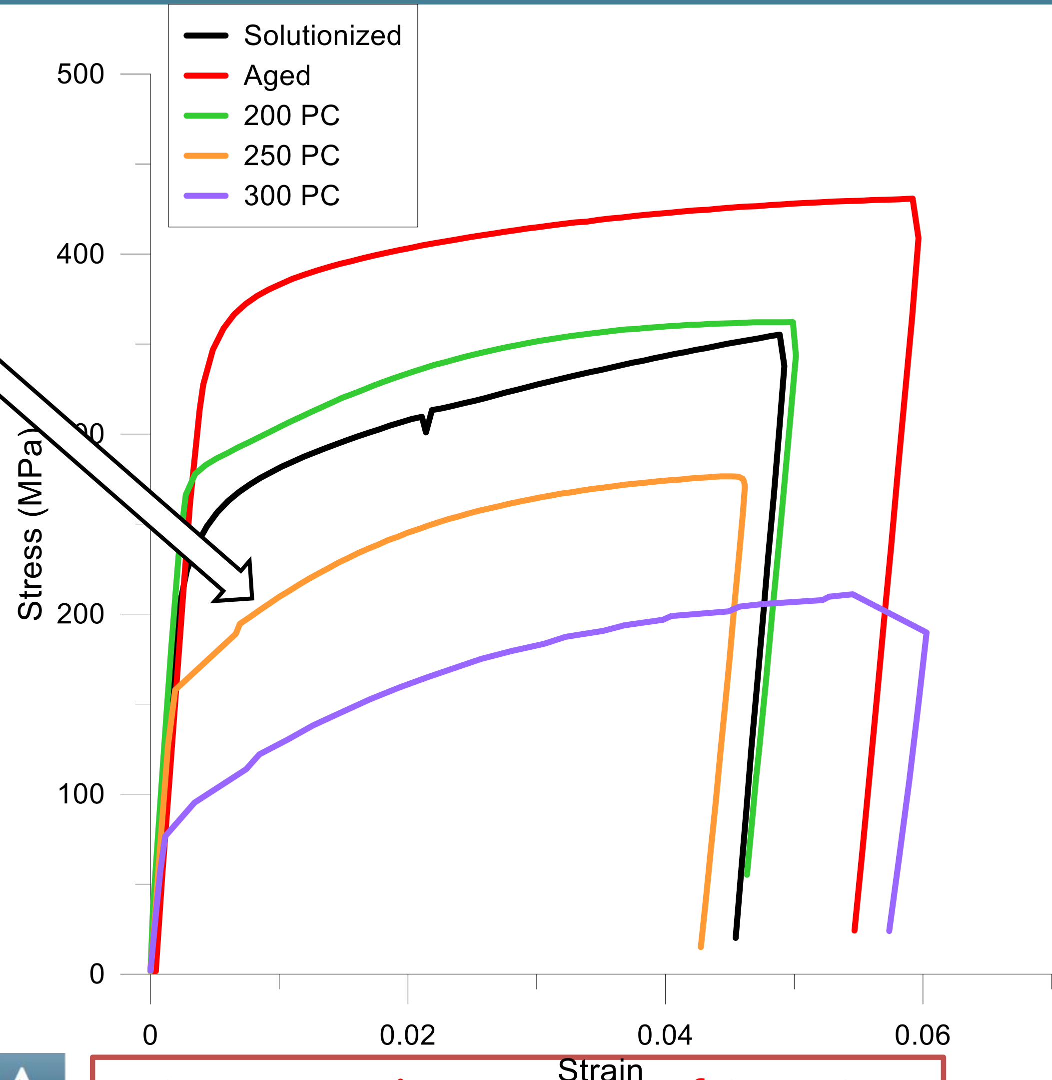

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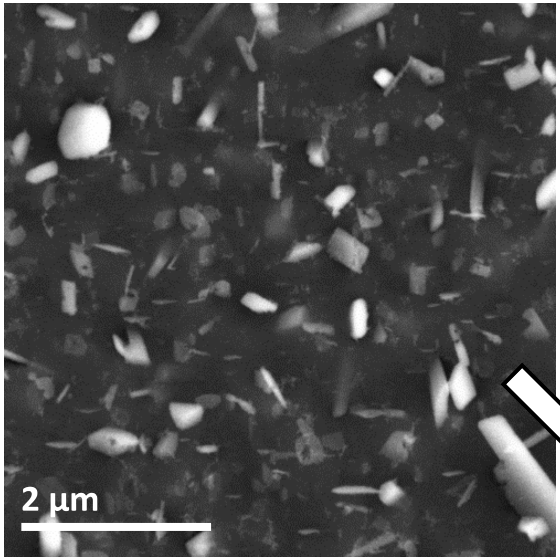

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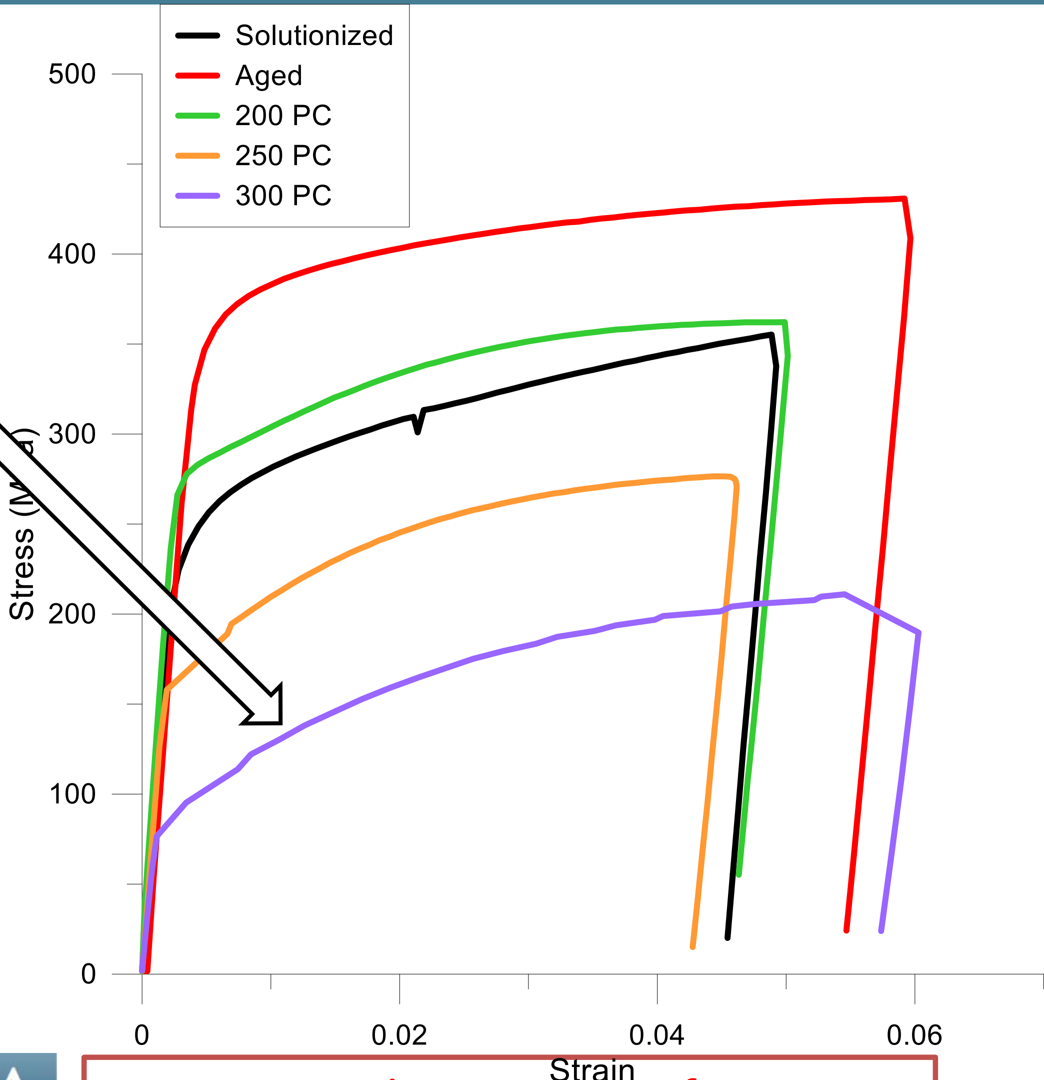
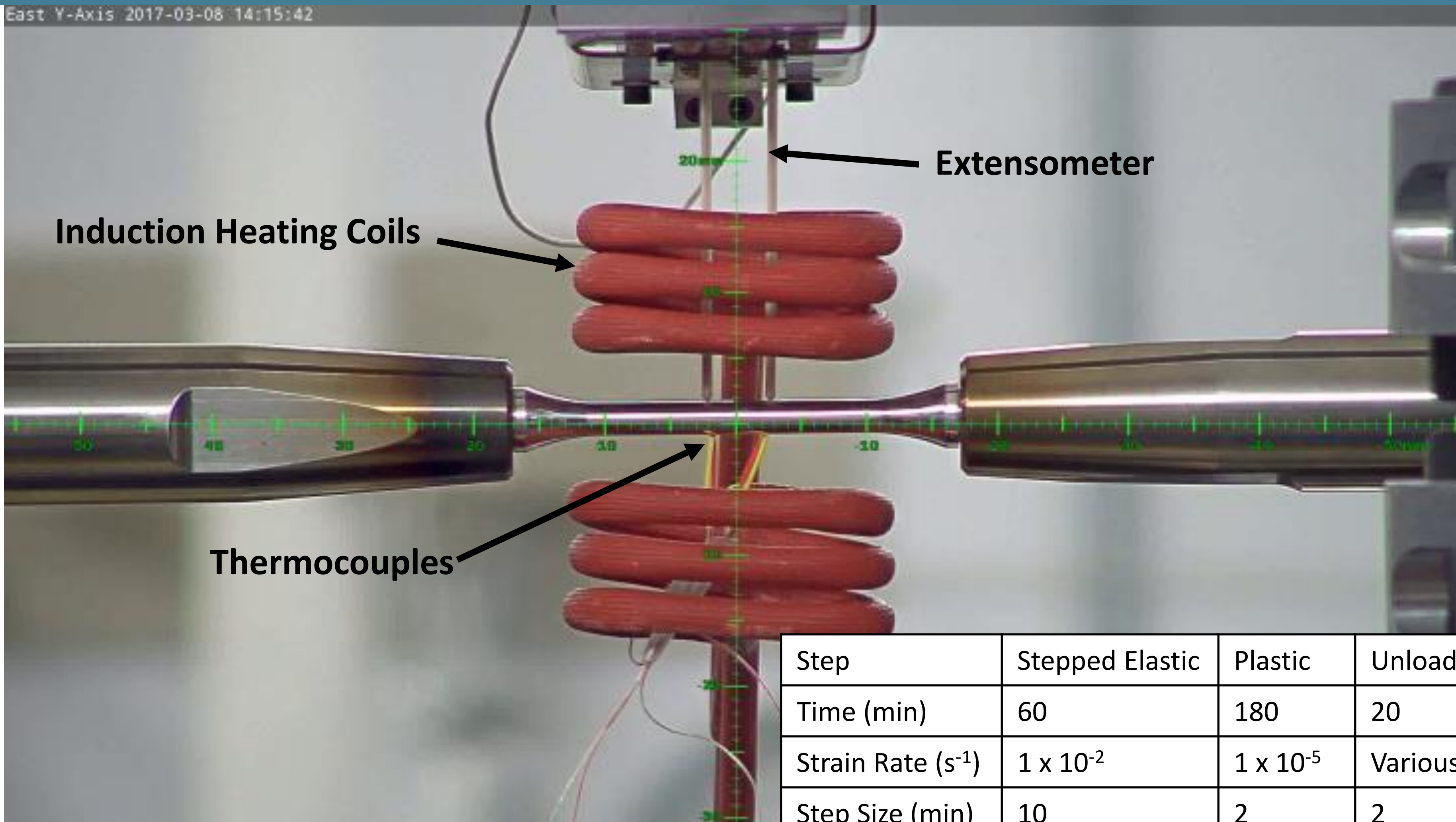
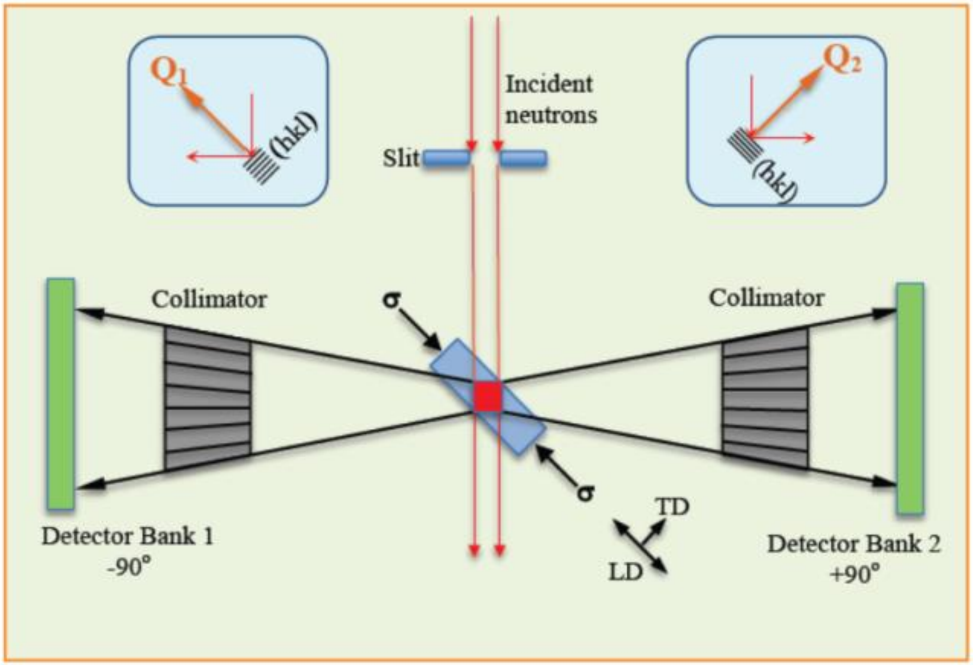


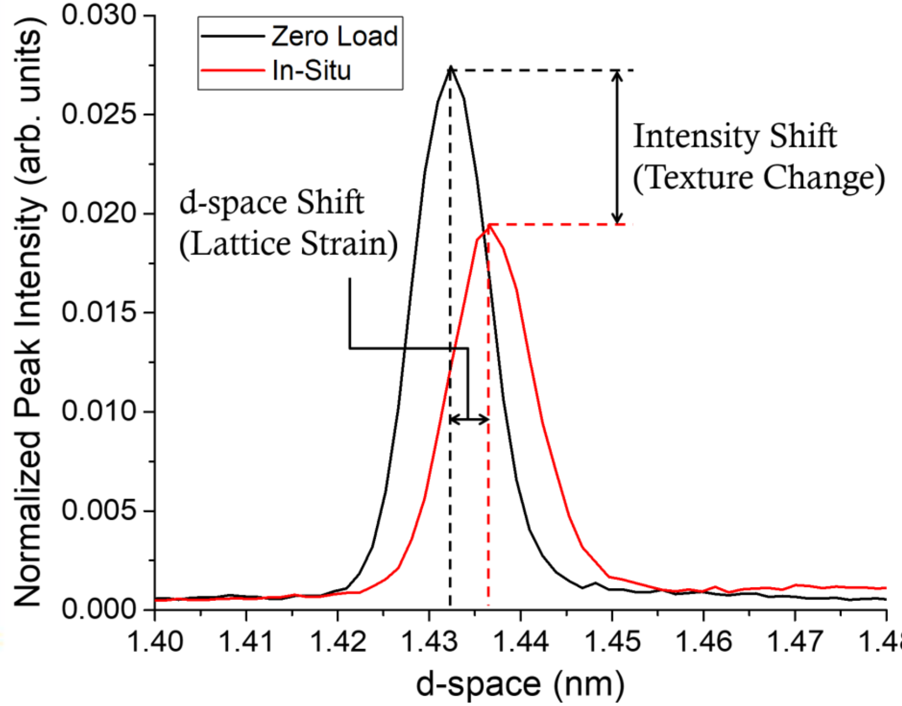
In-Situ Neutron Diffraction at ORNL Spallation Neutron Source (SNS) – VULCAN Beamline



Step	Stepped Elastic	Plastic	Unload
Time (min)	60	180	20
Strain Rate (s^{-1})	1×10^{-2}	1×10^{-5}	Various
Step Size (min)	10	2	2

Neutron Diffraction Experimental Setup at VULCAN

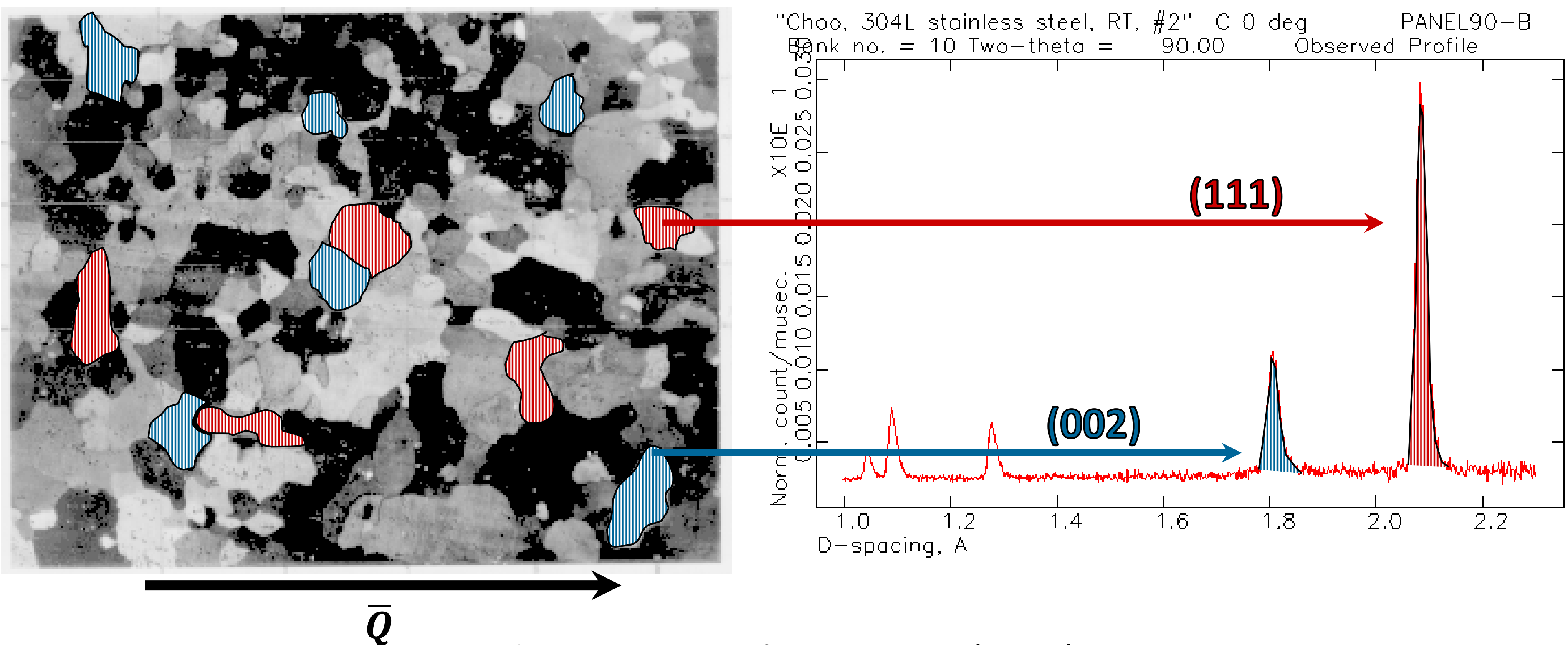




Neutron Diffraction Allows Separation of Individual Grain Orientation Mechanical Behavior

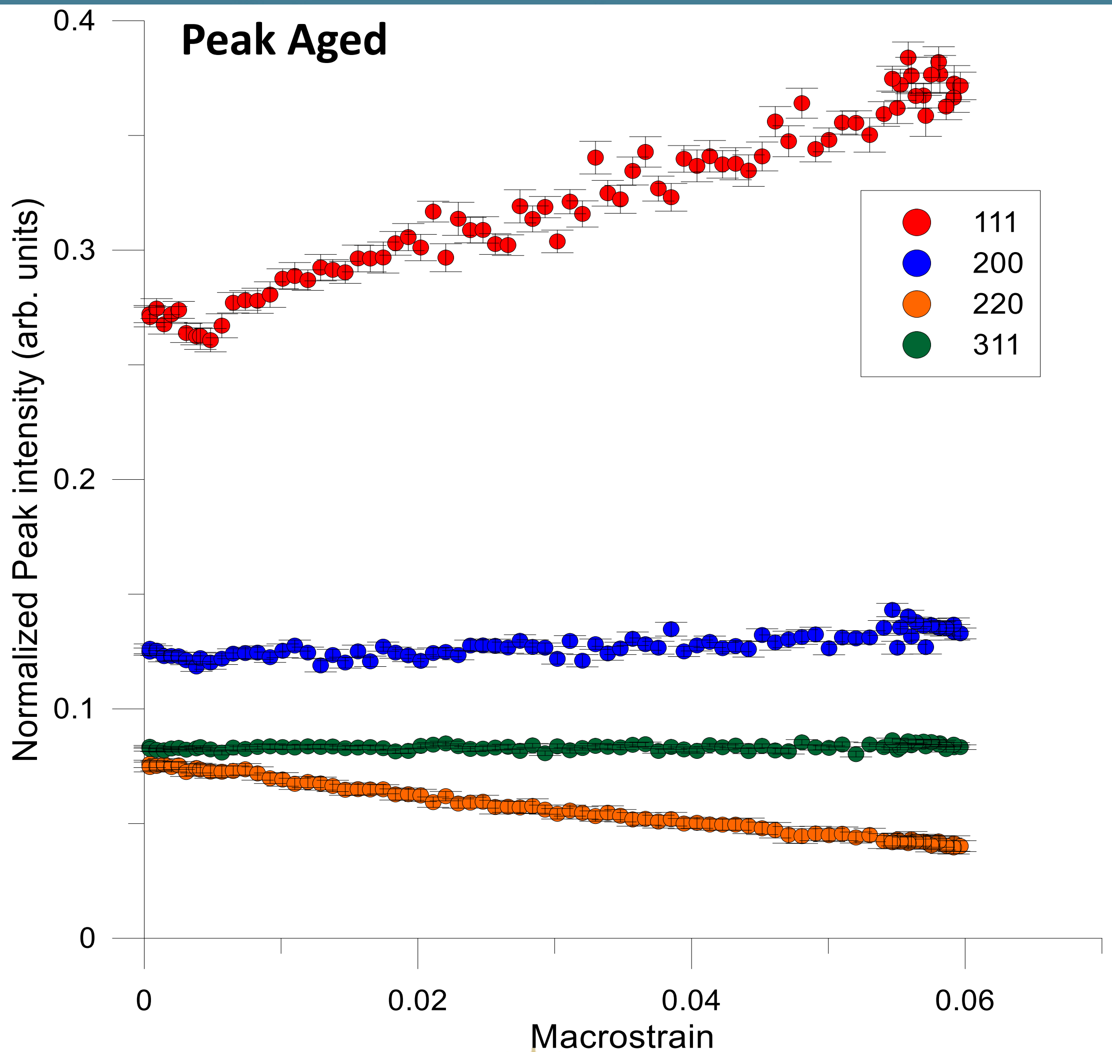
Polycrystalline Aggregate of grains

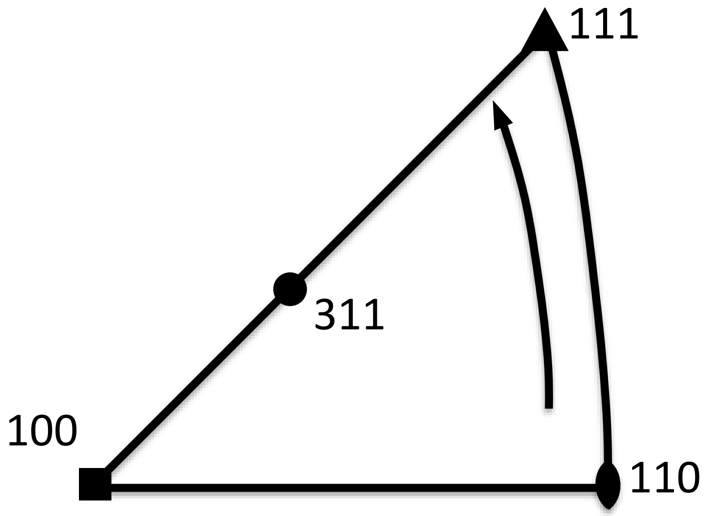
Stainless Steel



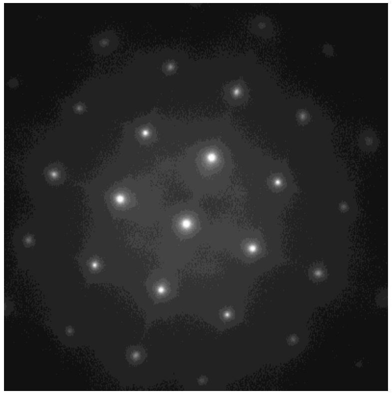
Slide Courtesy of Don Brown (LANL)

Grain Rotation in the Peak Aged Condition

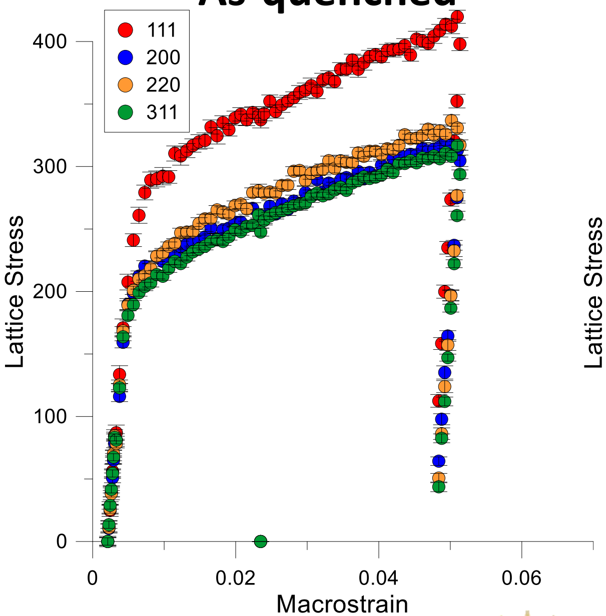


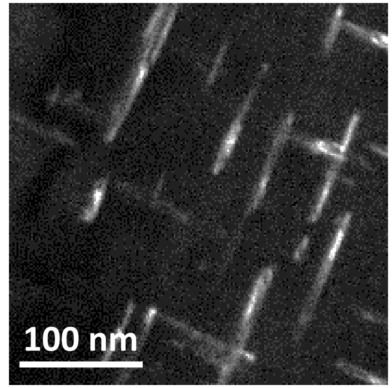


Plastic Anisotropy Increases with the Presence of Shearable Precipitates

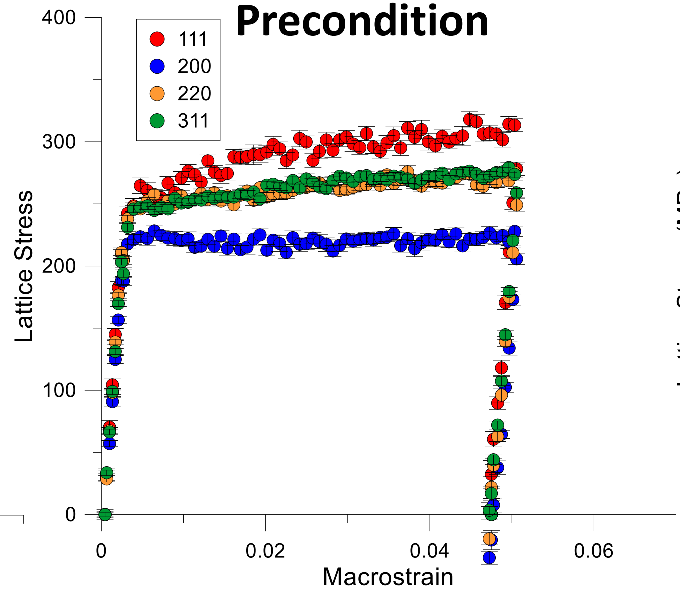


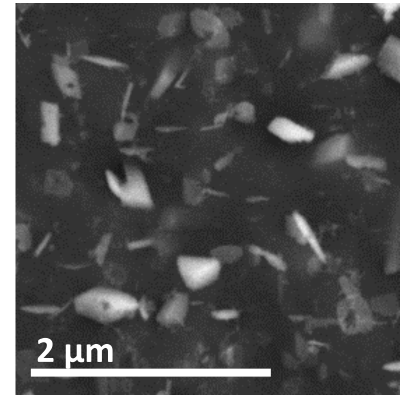
As-quenched



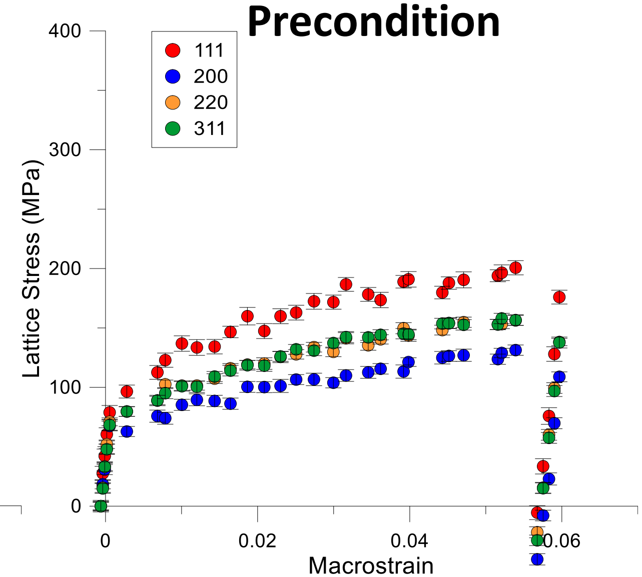


200 °C Precondition



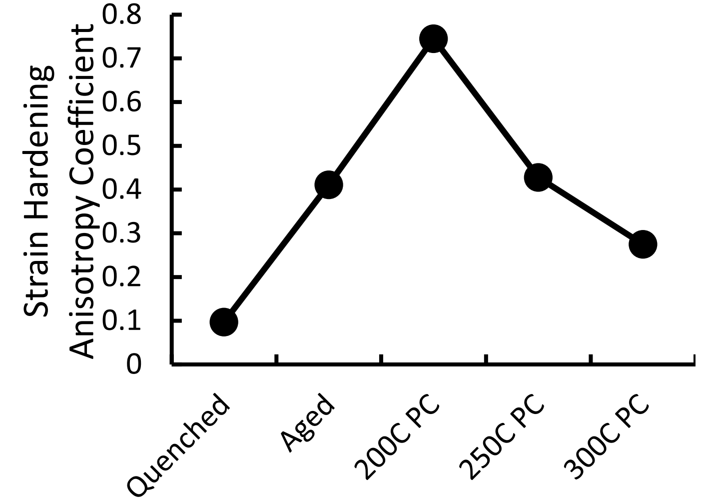


300 °C Precondition

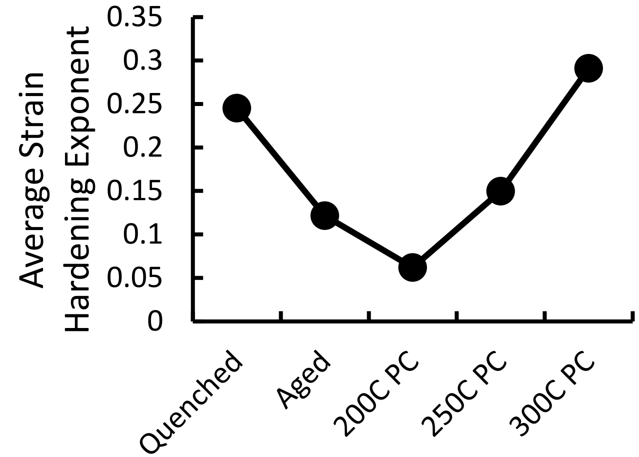


Deformation Mechanism Shifts from Precipitate Cutting to Orowan Looping

- Plot (right) shows strain hardening anisotropy, defined as the fraction difference between measured strain hardening exponents
- Deformation mechanism shift from mostly precipitate cutting to Orowan looping
- Lack of precipitate cutting causes as-quenched and 300°C overaged condition to act similarly.



Aging condition	Major precipitates	Possible strengthening mechanisms
Solutionized	GPI	Solute drag, precipitate cutting
Peak Aged	GPI, GPII, θ′′	Precipitate cutting
200 °C Overaged	θ′′, θ′	Precipitate cutting, Orowan looping
250 °C Overaged	θ′	Precipitate cutting, Orowan looping
300 °C Overaged	θ	Orowan looping



RR350 Alloy Composition and Aging Conditions

RR350 Composition

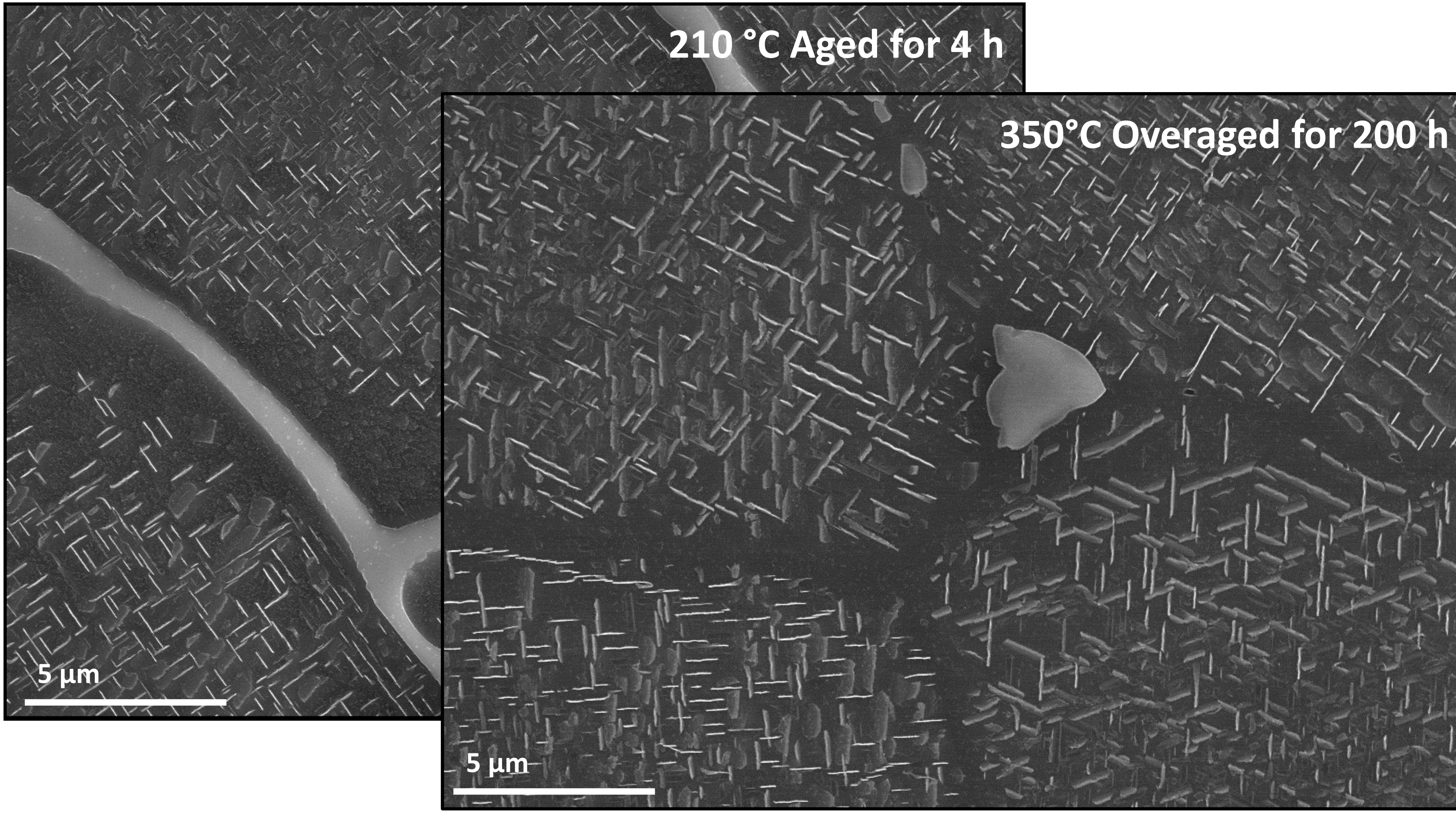
Element	Si	Cu	Zn	Fe	Ni	Mn	Co	Zr	Ti	Sb	Al
Wt%	0.04	4.8	0.01	0.09	1.2	0.19	0.26	0.17	0.21	0.17	bal.

- RR350 is an alloy developed for applications under creep conditions, strengthened with θ′ precipitates
- Grain boundaries have Ni-, Cu-, Al- containing intermetallics, which are detrimental to ductility but improve creep resistance

Heat treatment schedule

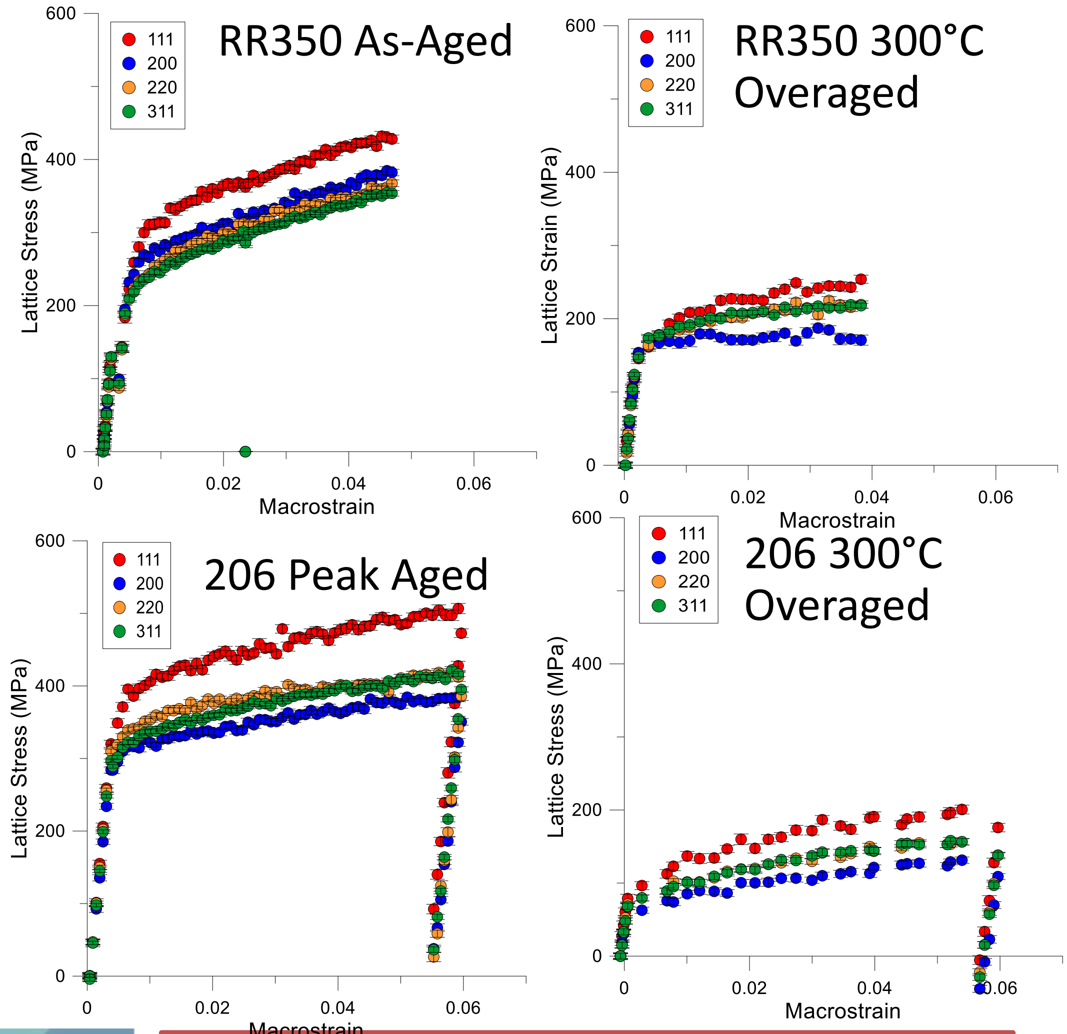
Step	Solutionizing	Quench	Aging	Overaging
Temperature (°C)	535	80-90	210	100, 300, 350
Time (h)	1	<1	4	200

Microstructure in RR350 Relatively Unchanged during Overaging



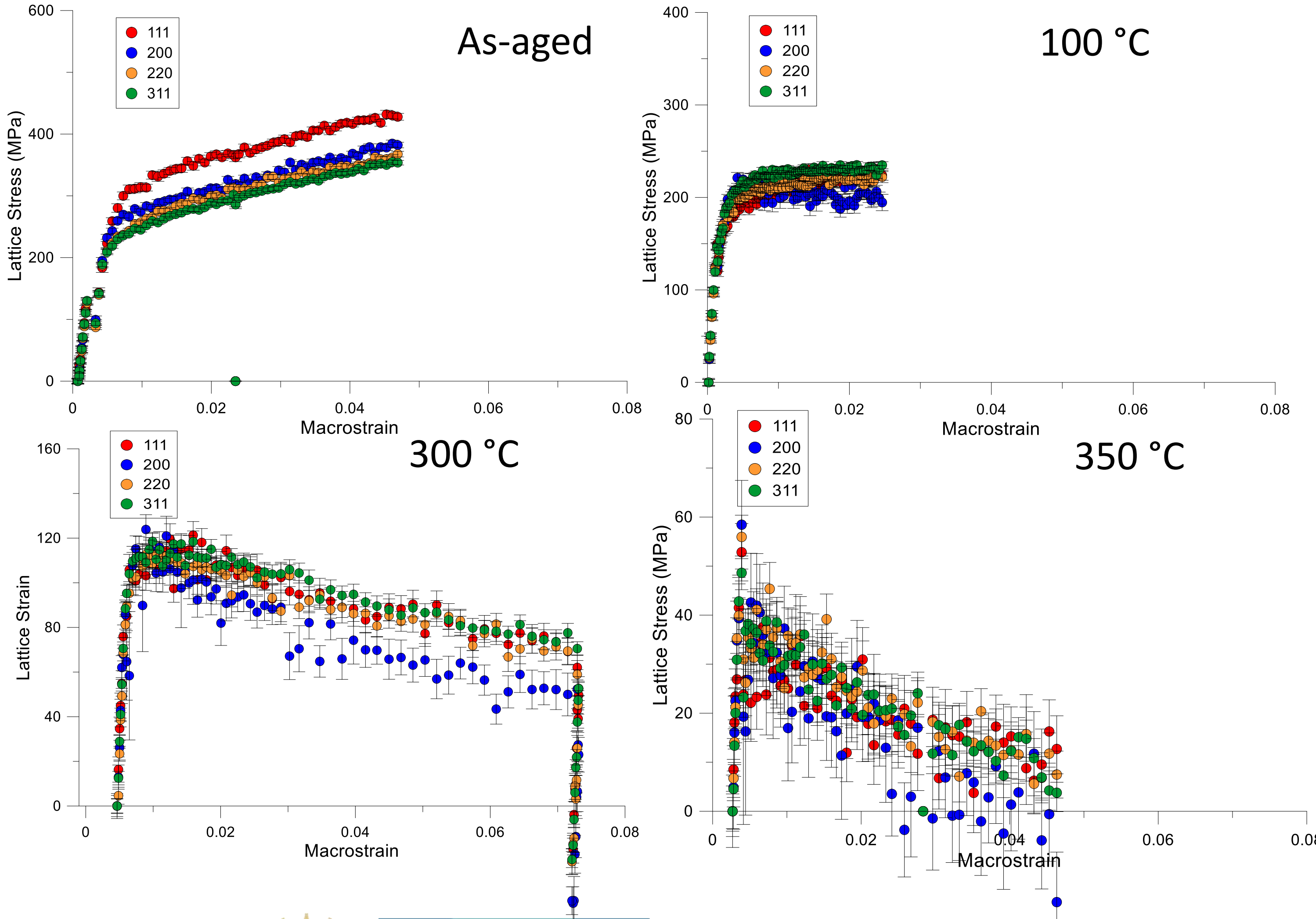
Comparison of 206 to RR350 (Thermal Stability)

- RR350 loses less strength during over aging than 206
- Reduction in strength during aging is lessened after some time



Center Proprietary – Terms of CANFSA Membership Agreement Apply

RR350 Precipitate Shearing May Become Active At Certain Temperatures



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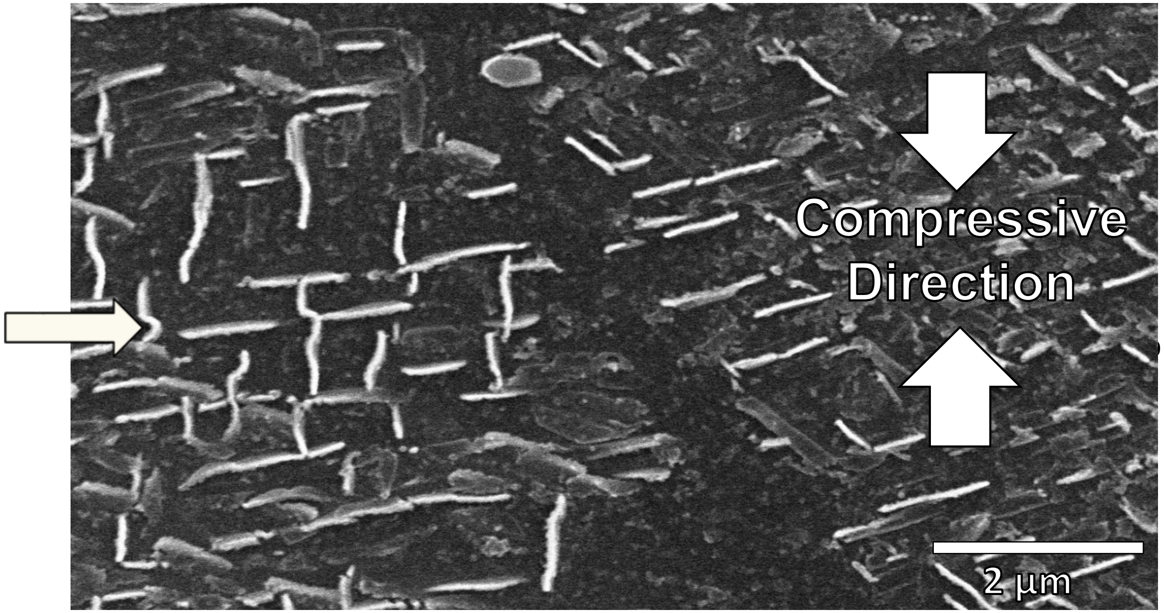
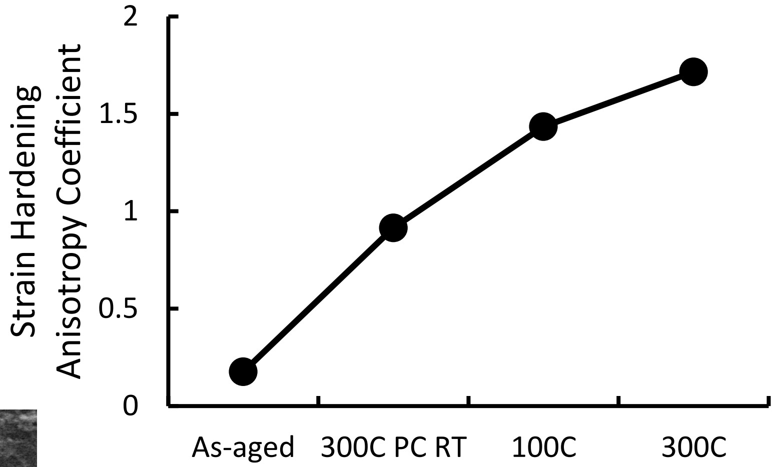
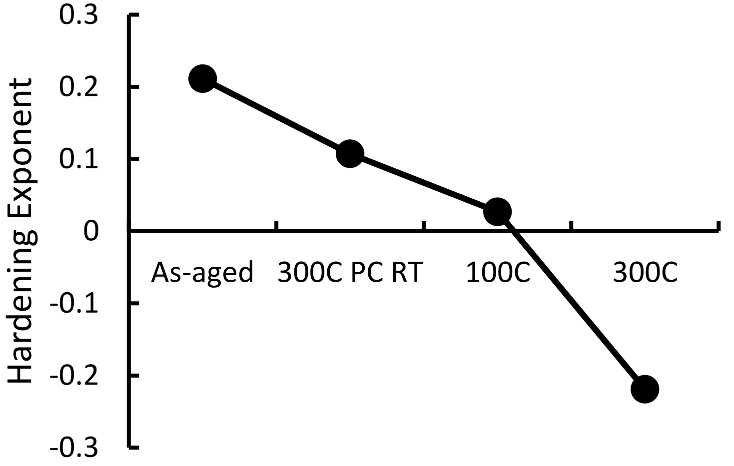
Mechanism Changes with Temperature in RR350

- Similar strain hardening anisotropy to 206 room temperature
- Activation of a similar precipitate cutting mechanism at higher temperatures
- θ′ precipitates in RR350 still relatively thick compared to 206 precipitates







Conclusions

- 206 precipitate sizes range from nanometers to microns
- Precipitate cutting controls a significant amount of the grain orientation-dependent mechanical behavior in 206
- Significant grain rotation and texture development occurs during tension in 206 and RR350
- Strength decreases and anisotropy increases with increasing temperature in RR350

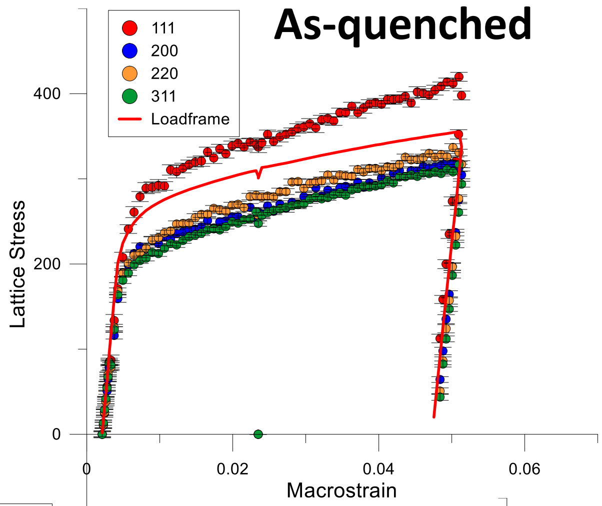
Future Work

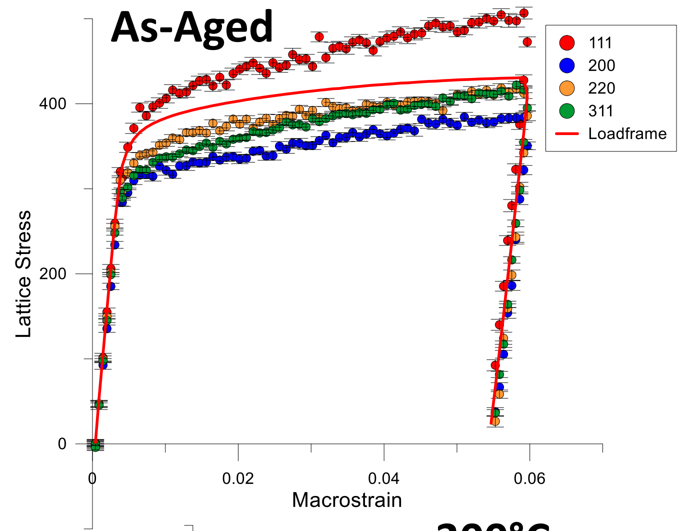
- High resolution TEM to observe GP zones in the 206 as-quenched condition
- TEM defect study to observe precipitate cutting
- Quantitative modeling of precipitate cutting
- Ex-situ and in-situ (neutron diffraction) studies of creep in various Al-Cu alloys
- User proposal submitted to the Advanced Photon Source at Argonne National Laboratory to study precipitation and growth kinetics using transmission x-ray microscopy
- Summer internship at ORNL

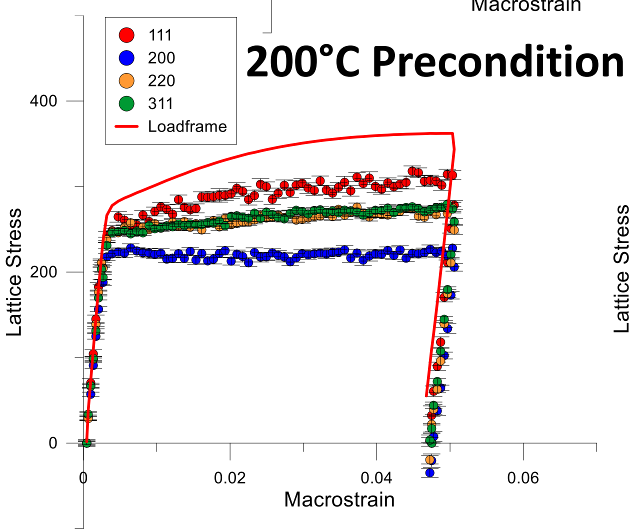
Acknowledgement of Funding Sources

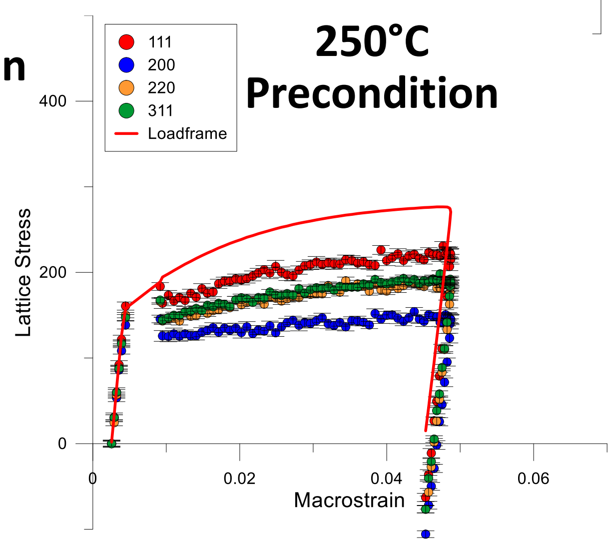
- Laboratory Directed Research and Development (LDRD), Oak Ridge National Laboratory
- Center for Advanced Non-Ferrous Structural Alloys (CANFSA), an Industry-University Cooperative Research Center sponsored by the National Science Foundation, at the Colorado School of Mines and Iowa State University.
- Propulsion Materials Program, Office of Vehicle Technologies, Energy Efficiency and Renewable Energy (EERE), US DOE
- Scientific User Facilities Division, Office of Basic Energy Sciences, US Department of Energy, at Oak Ridge National Laboratory under contract DE-AC05-00OR22725 with UT-Battelle.
- AJC's Early Career Award from the U.S. DOE, Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, Grant No. DE-SC0016061

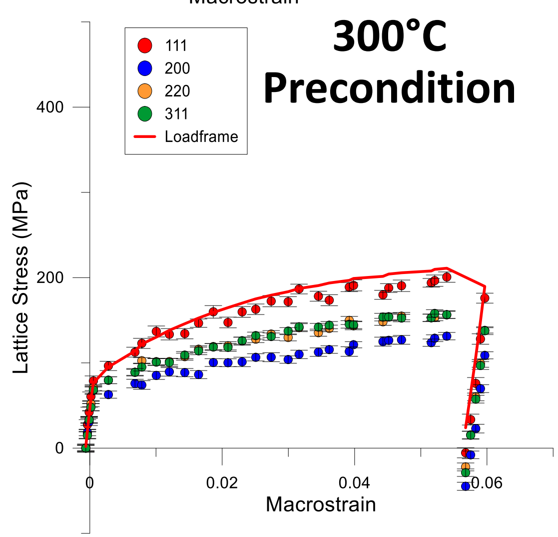
Extra Slides – Lattice Stress in All Aging Conditions











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

Graduate Student – Brian Milligan (CSM)
Faculty/Advisors – A. Clarke (CSM)
Industrial Mentors – A. Shyam (Oak Ridge National Lab.)

Program Goal

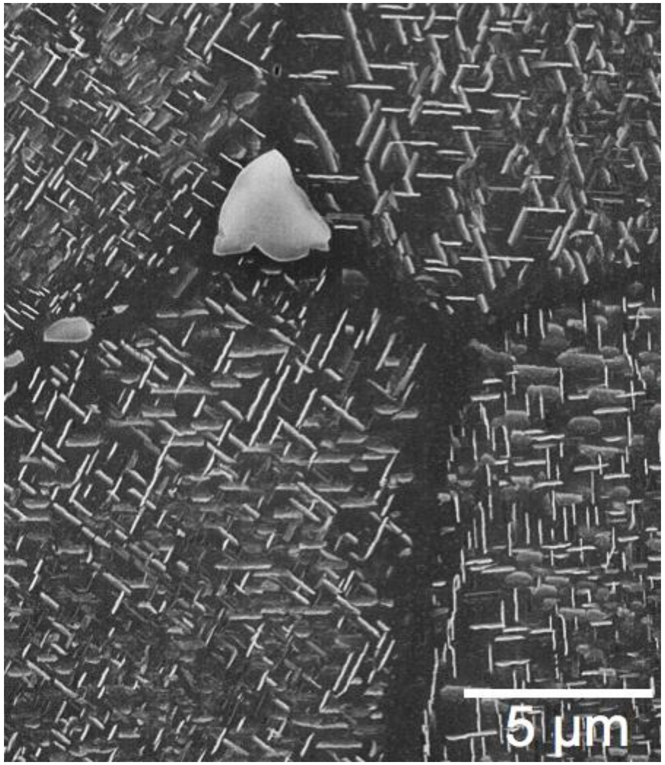
Characterize the mechanical properties and microstructure of thermally stable Al-Cu alloys under various loading and aging conditions

Approach

Utilize neutron diffraction, microscopy, and mechanical testing to identify deformation mechanisms ex-situ and in-situ

Benefits

Improved scientific understanding of mechanical properties in Al-Cu alloys as well as insight into how to improve their performance at high temperature



Precipitation in RR350 aluminum alloy

Project Duration

Aug. 2017 to May 2021