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

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Iowa State University, Ames, IA
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Other Participants: Dong Ma (ORNL), Lawrence Allard (ORNL), Francisco Coury (Mines), Chloe Johnson (Mines), Yaofeng Guo (Mines), Benjamin Ellyson (Mines), Alec Saville (Mines), Gus Becker (Mines)

OAK RIDGE National Laboratory

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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.

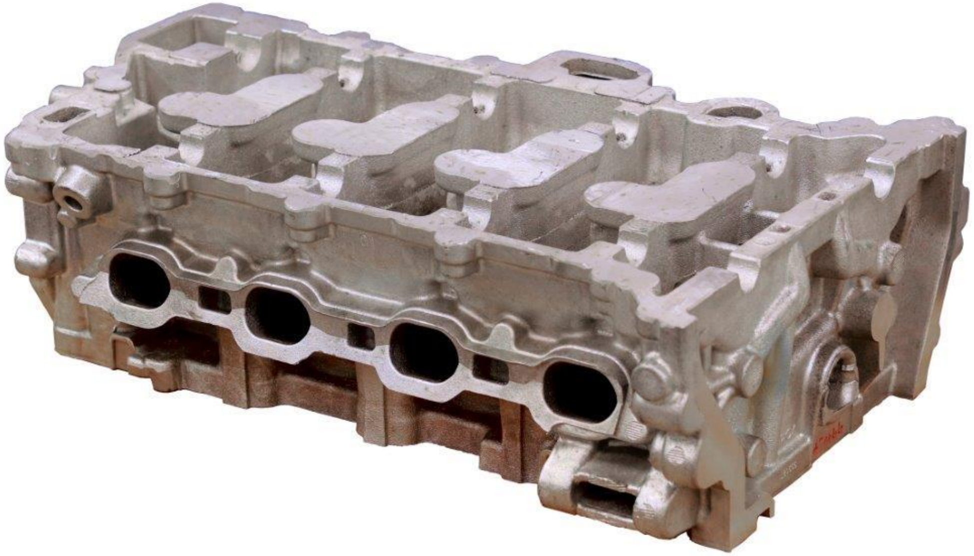
Recent Progress
- Implemented Kroner self-consistent model for elastic behavior
- Continued development for quantification of strain hardening mechanisms from energetic standpoint
- Interrupted aging study with Al-Cu, Al-Ag, and Al-Ag-Cu at 350°C performed at APS
- Implemented model to compare different aging conditions of 206 Al

Metrics

Description	% Complete	Status
1. Initial literature review	85%	●
2. In situ neutron diffraction, creep testing, and TXM	65%	●
3. Microstructural characterization pre- and post- creep and tension	50%	●
4. Qualitative assessment of neutron diffraction and mechanical test data	70%	●
5. Application and development of qualitative modelling to micro-scale diffraction data	40%	●

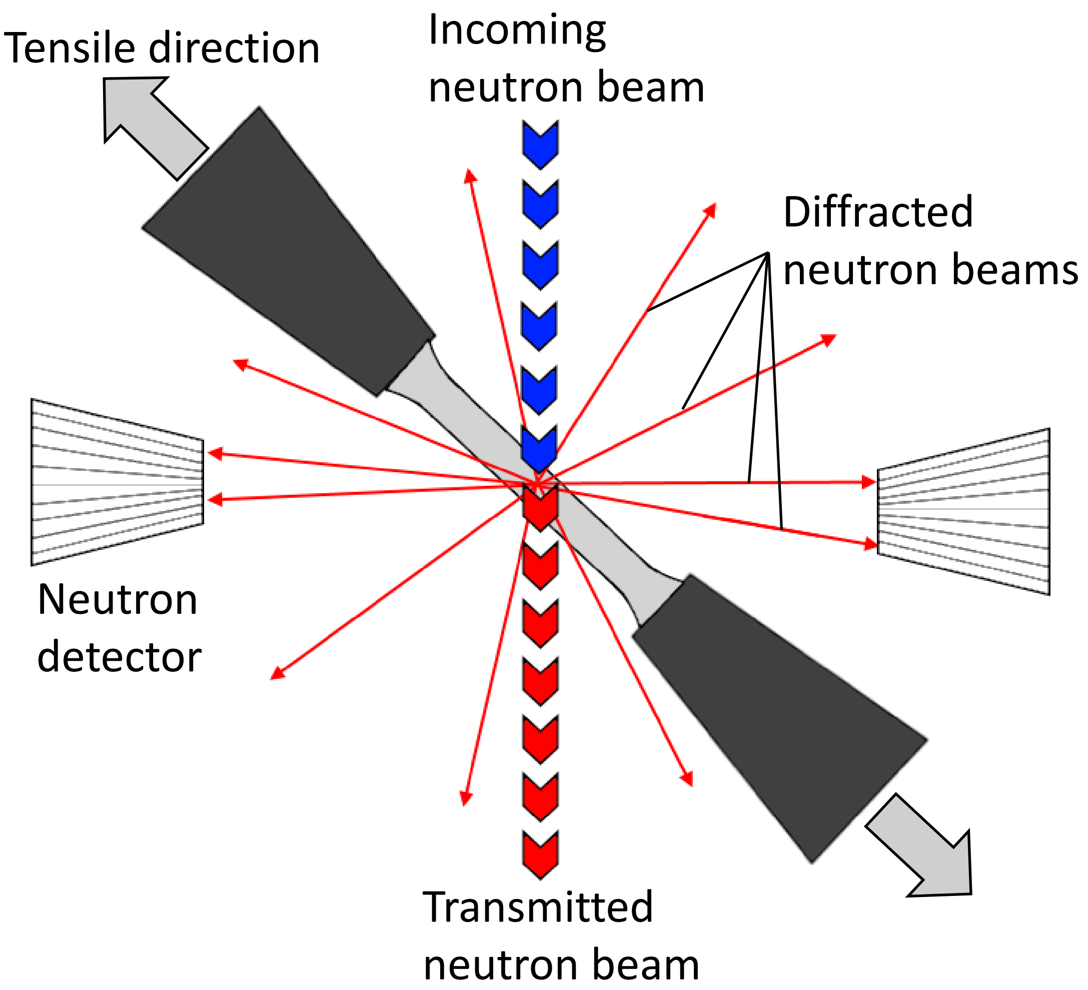
Industrial relevance

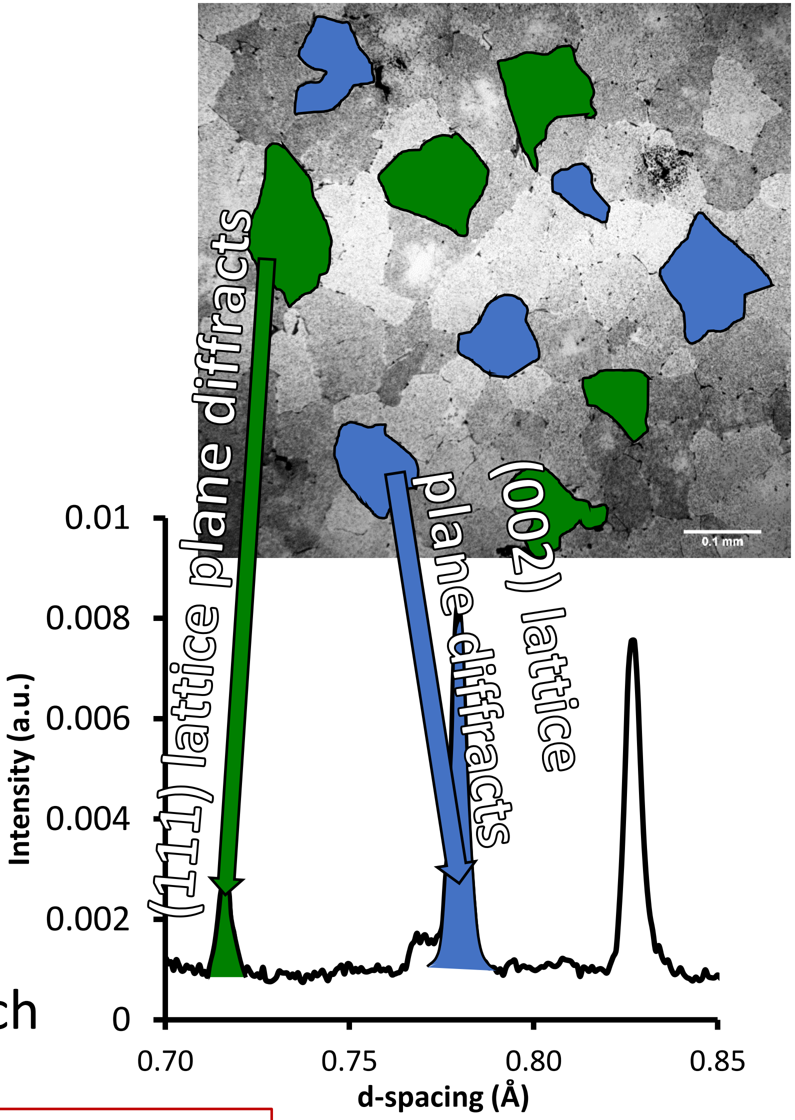
- Cast Al-Cu alloys have high strength, low density, and are easy to manufacture
  - Used in various industries such as for cylinder heads in light-duty engines
- Understanding of deformation mechanisms allow prediction of mechanical behavior
  - Strain hardening behavior commonly overlooked, but is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



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

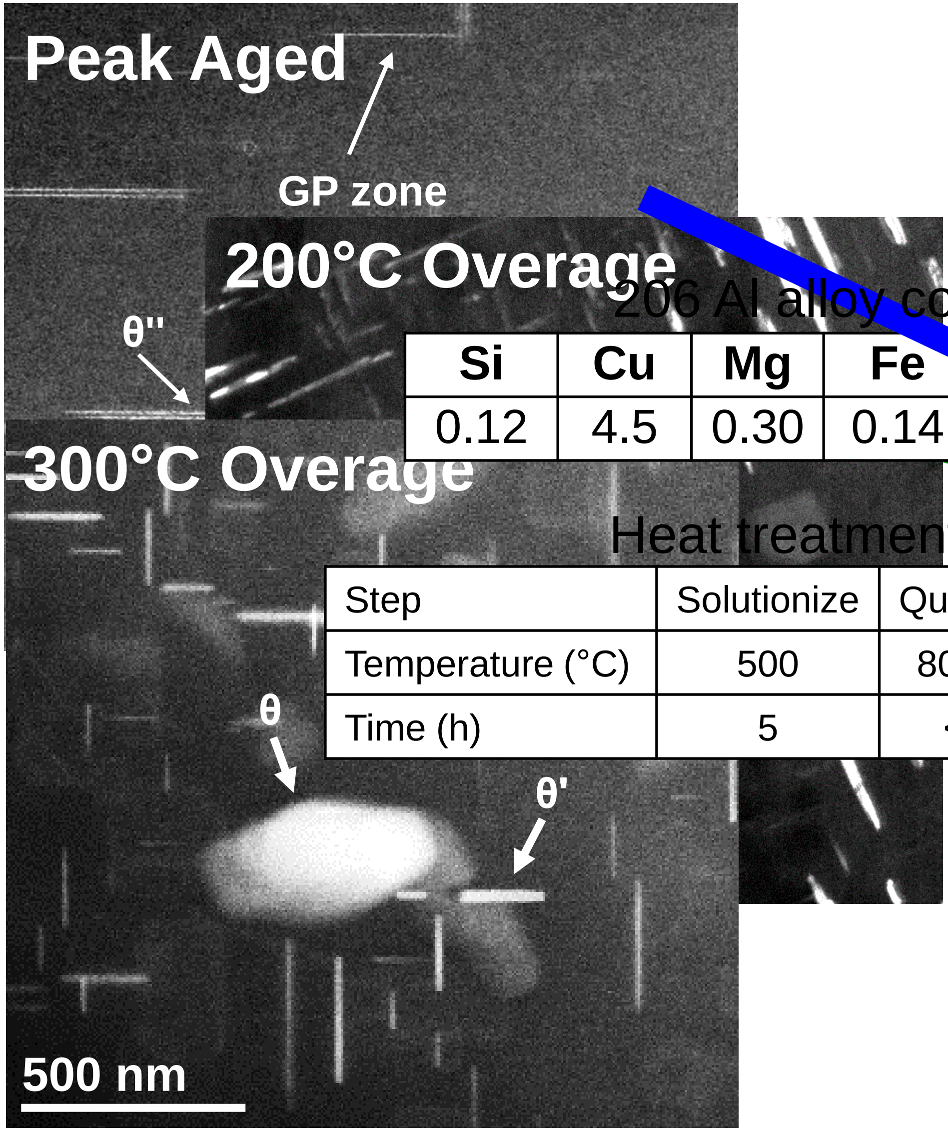
Neutron diffraction used to measure internal grain stresses

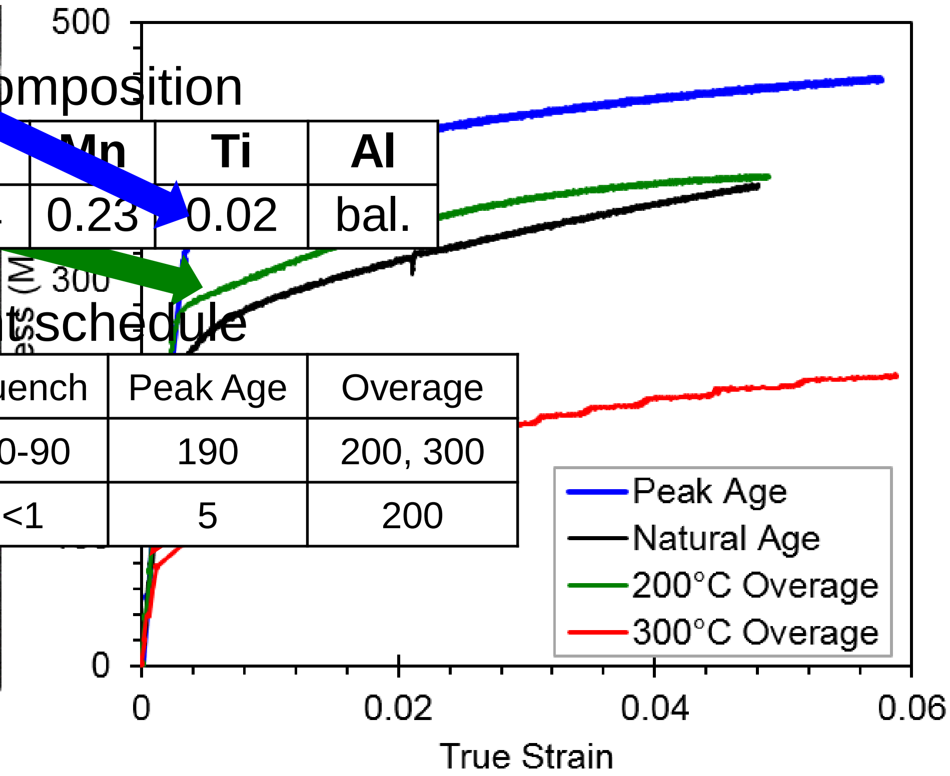




- Allows measurement of lattice strain, which is related to elastic internal stress

Background in the model system 206 Al





206 Al alloy composition

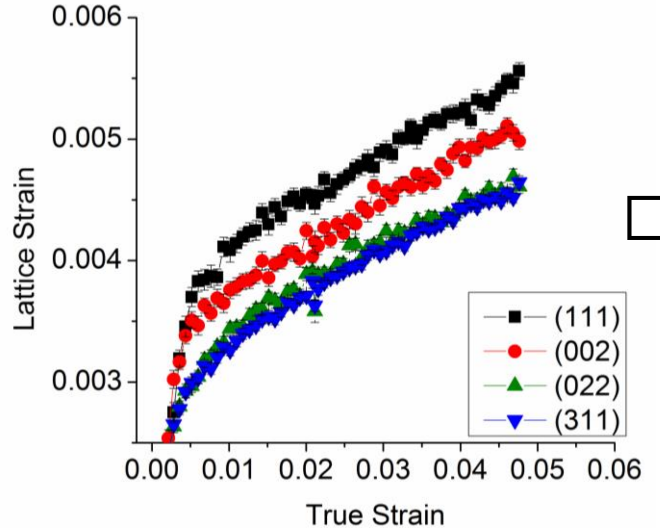
Si	Cu	Mg	Fe	Mn	Ti	Al
0.12	4.5	0.30	0.14	0.23	0.02	bal.

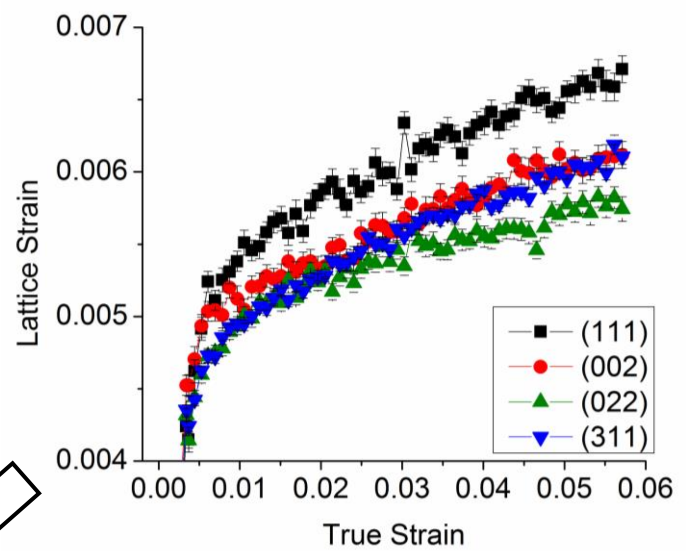
Heat treatment schedule

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

Anisotropy observed in neutron diffraction results

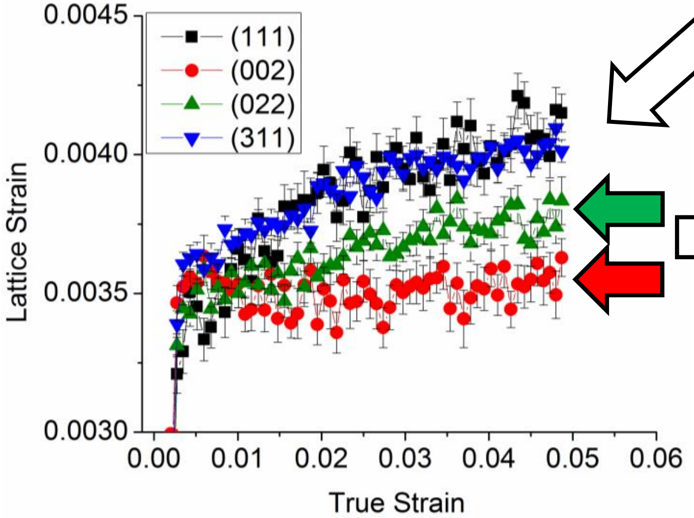
Natural Age

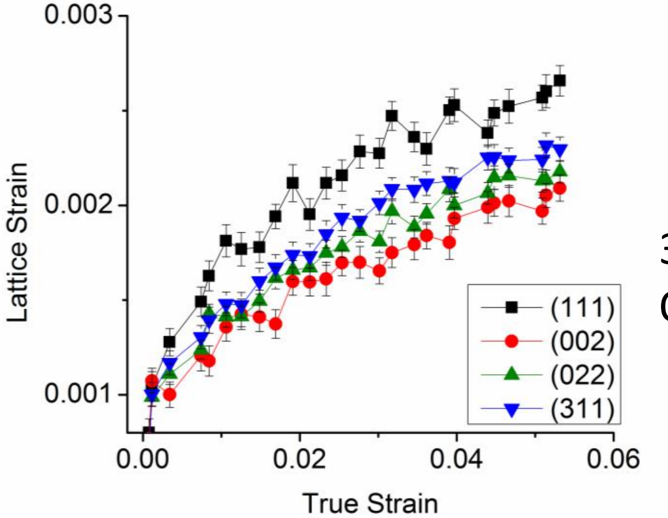




Peak Age

200°C Overage





300°C Overage

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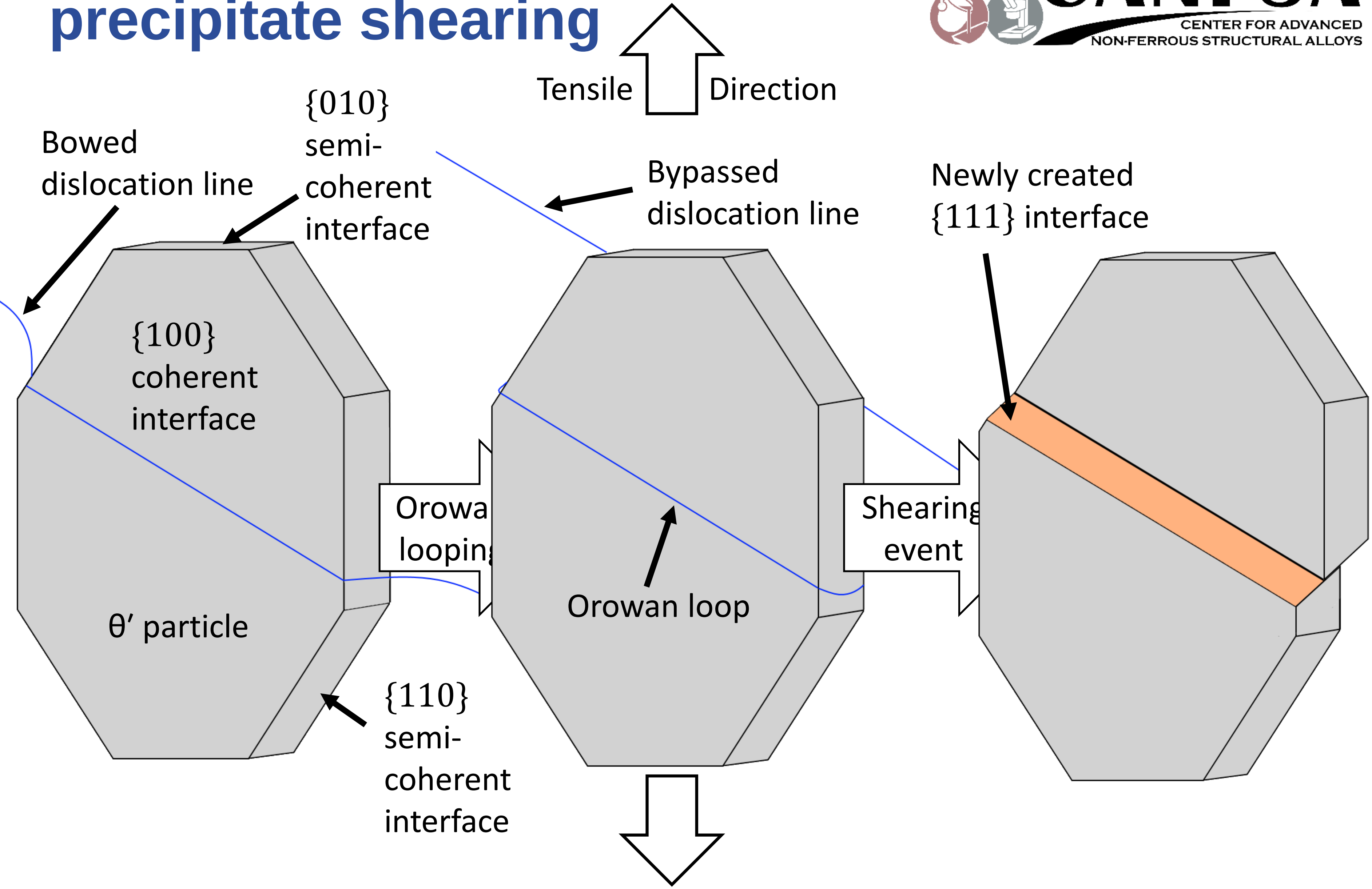
Mechanism of delayed precipitate shearing



Tensile Direction

{010} semi-coherent interface

Bowed dislocation line

Bypassed dislocation line

Newly created {111} interface

{100} coherent interface

Orowa loopin

θ′ particle

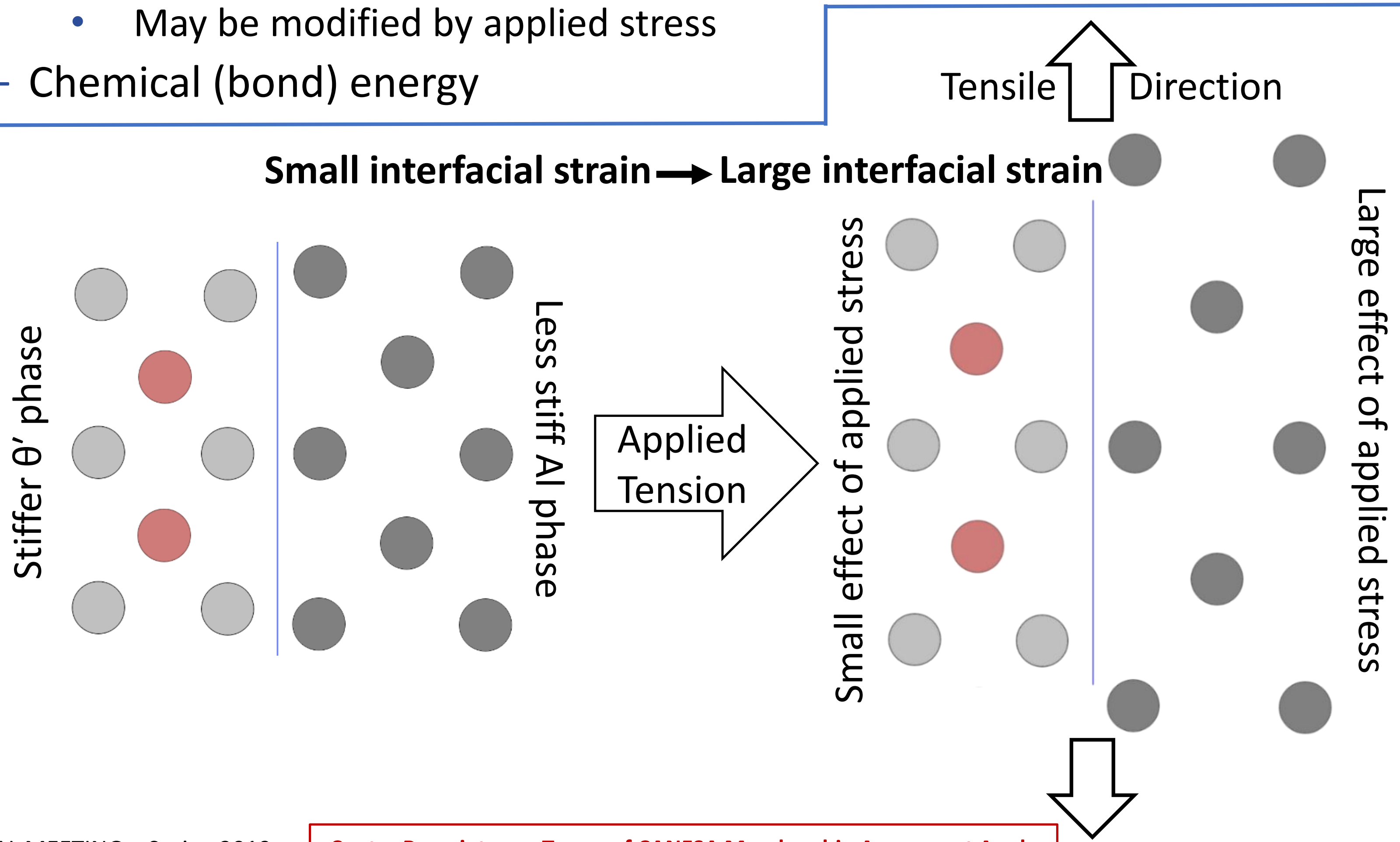
Orowan loop

Shearing event

{110} semi-coherent interface

Applied stress effects on precipitate shearing energy

- 2 major contributions to energy required for shearing:
  - – Lattice mismatch
    - May be modified by applied stress
  - – Chemical (bond) energy



Tensile Direction

Small interfacial strain → Large interfacial strain

Stiffer θ′ phase

Less stiff Al phase

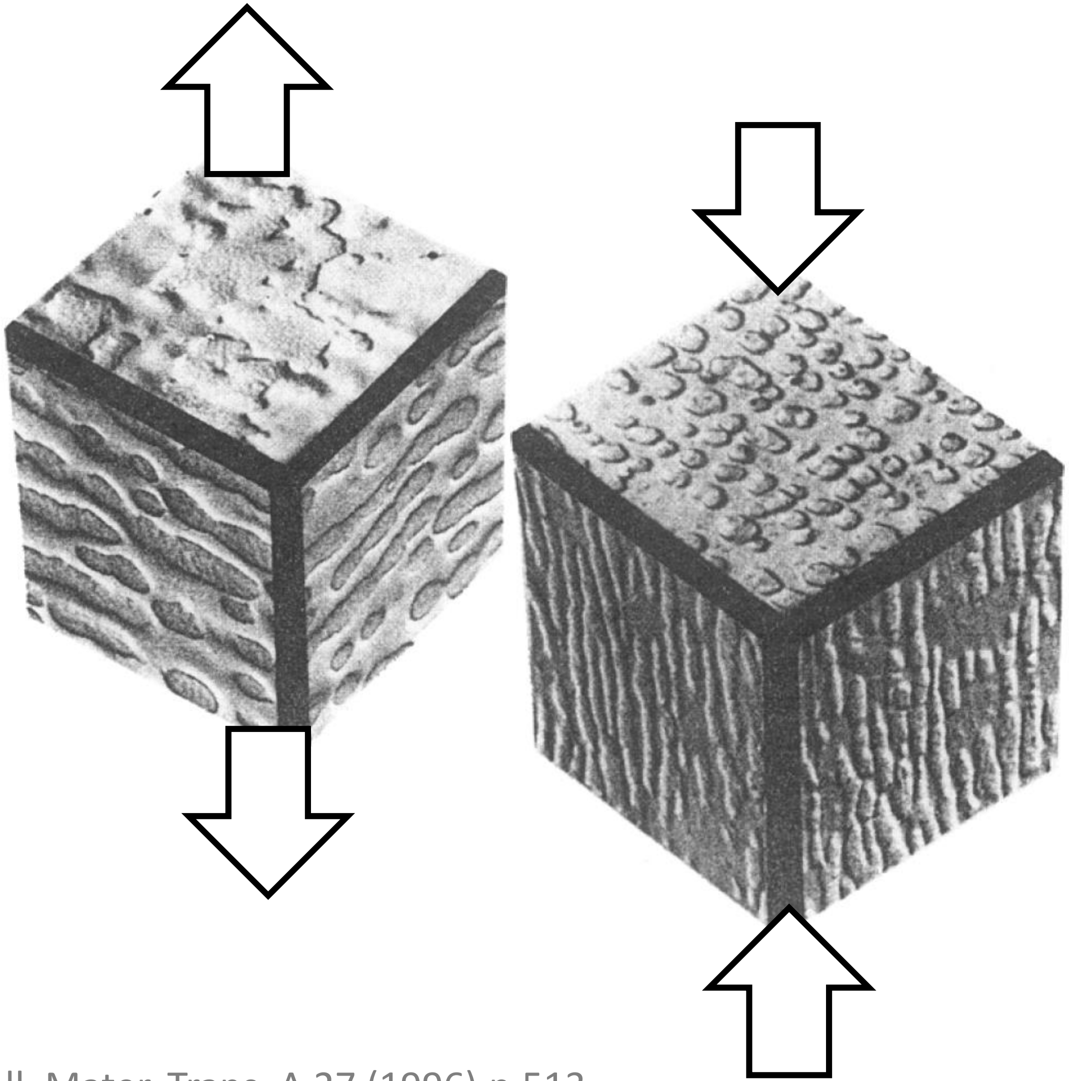
Applied Tension

Small effect of applied stress

Large effect of applied stress

Existing case: Rafting of γ-γ' structures in Ni superalloys

- Morphology of γ′ is changed in tension or compression
- $\gamma_{raft} \propto \sigma_{app} * \delta_{misfit} * \Delta Y$
- Hypothesis: Same effect can increase/decrease likelihood of precipitate shearing
  - Strain energy of newly created interface can be changed



F.R.N. Nabarro, Metall. Mater. Trans. A 27 (1996) p.513

Development of quantitative model for shearing energy

- Assumptions:
  - Strain energy on each side of interface is equal before stress is applied
  - Strain is uniform throughout precipitate (thin, but large diameter)
  - No strain field interactions between adjacent precipitates
- $f(\theta) = \frac{\sum_n (\cos(\theta) - \nu \sin(\theta))}{n}$
  - Averaged over multiplicity of applied stress direction, slip plane, and precipitate orientation

Symbol	Value
θ	Angle between σ_{app} and ϵ_0
n	Multiplicity
ν	Matrix Poisson's ratio
ϵ_0	Interfacial strain
C_γ	Stiffness component in strain direction
t_p	Precipitate thickness
A	Interface area
σ_{app}	Applied stress
D	Plate diameter

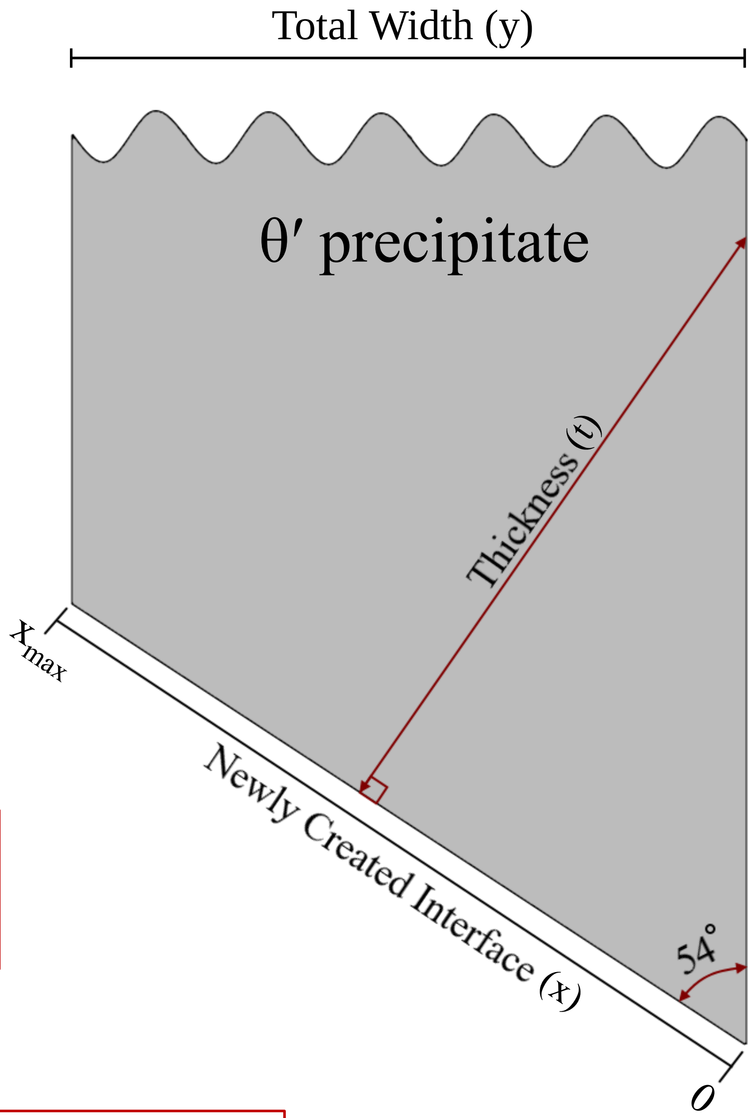
Geometric considerations in shearing energy model

- $E_{strain}^{ppt} = 2 * \epsilon_0 * C_\gamma * t_p * A$
- Interface not perpendicular to thickness of precipitate
  - $t_p = x * \tan(54)$
  - $A = xD$
  - $x_{max} = y * \sin(54)$

$$E_{strain}^{ppt} = 2 * \int_0^{x_{max}} \epsilon_0 C_\gamma xD \tan(54)\, dx$$

$$\Delta\sigma = \epsilon_0 C_\gamma - \left(\epsilon_0 C_\gamma + \sigma_{app} f(\theta)\right)$$

$$\boxed{\Delta E_{strain}^{ppt} = 2 * \int_0^{x_{max}} \sigma_{app} f(\theta) xD \tan(54)\, dx}$$



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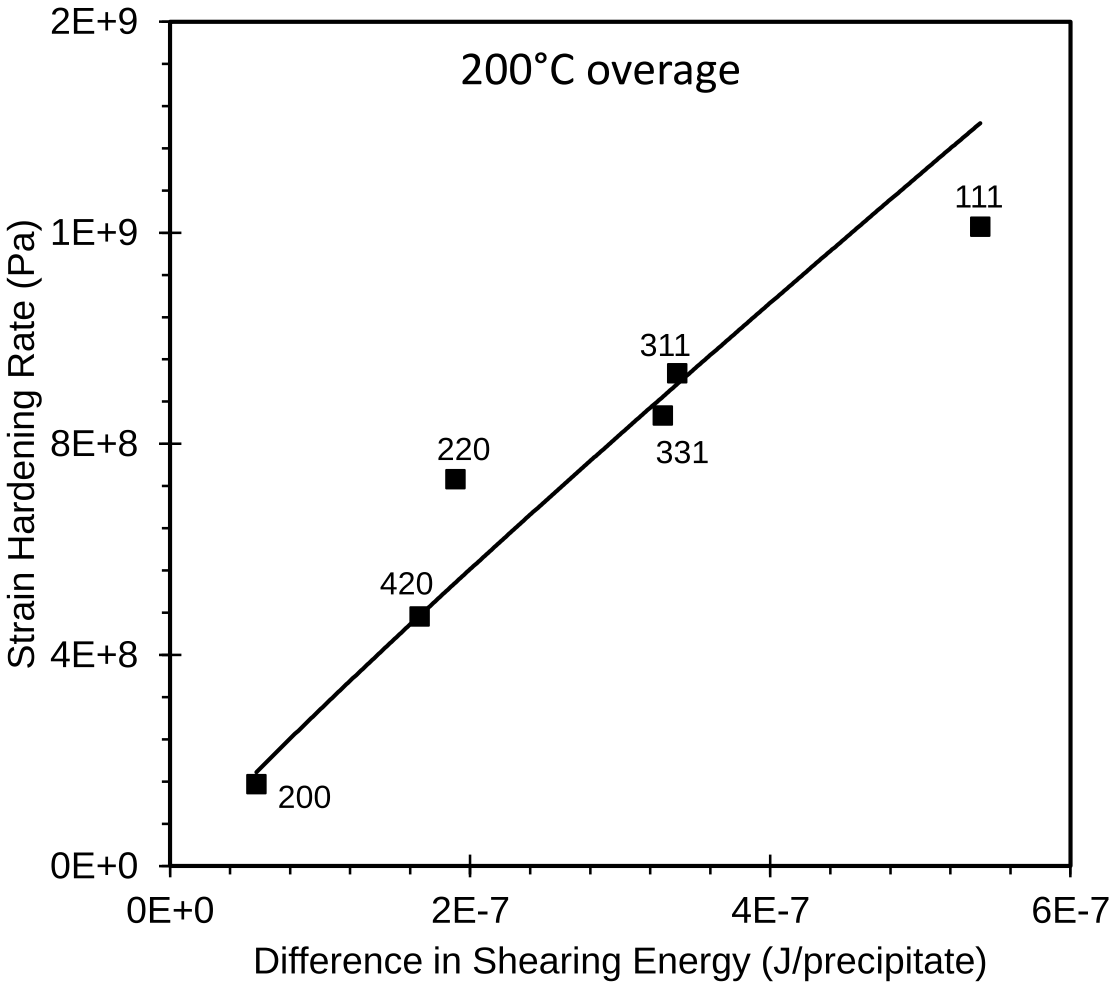
Comparison of modeling to experimental results

- Plot compares ΔE_{strain}^{ppt} with grain level strain hardening rate
- Correlation implies transition from shearing to looping controls strain hardening (in this condition)



Effect of precipitate size on the anisotropy behavior

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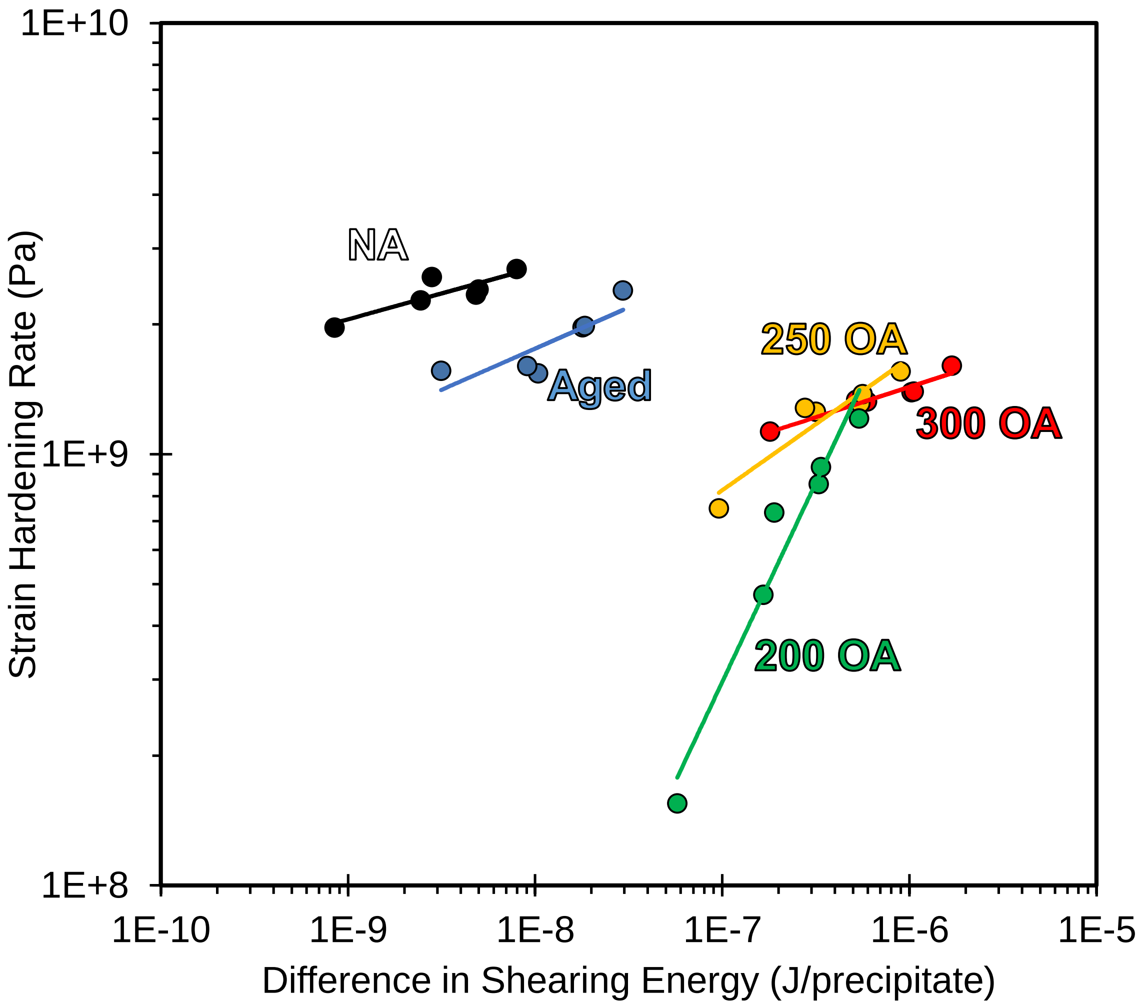
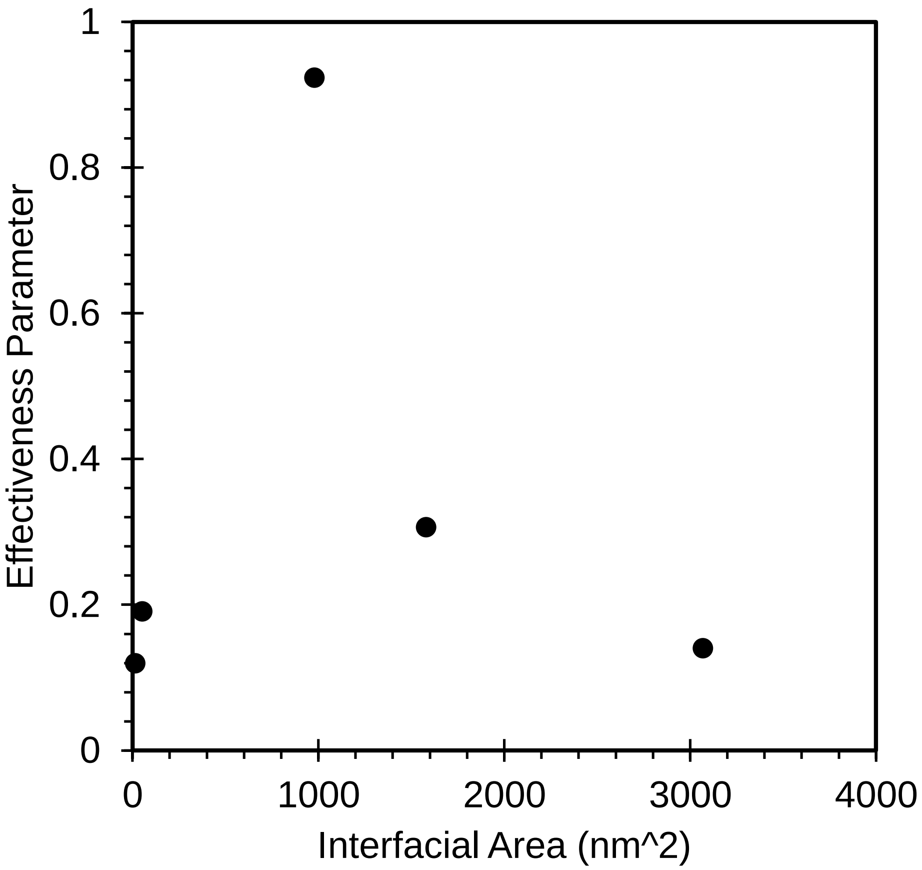
- Previous comparison applied to each condition of 206
- Shearing energy varies with precipitate size
- Slope of this plot shows magnitude of transition region control

Condition	Slope on Plot
NA	0.1199
PA	0.1909
200 OA	0.9238
250 OA	0.3067
300 OA	0.1403



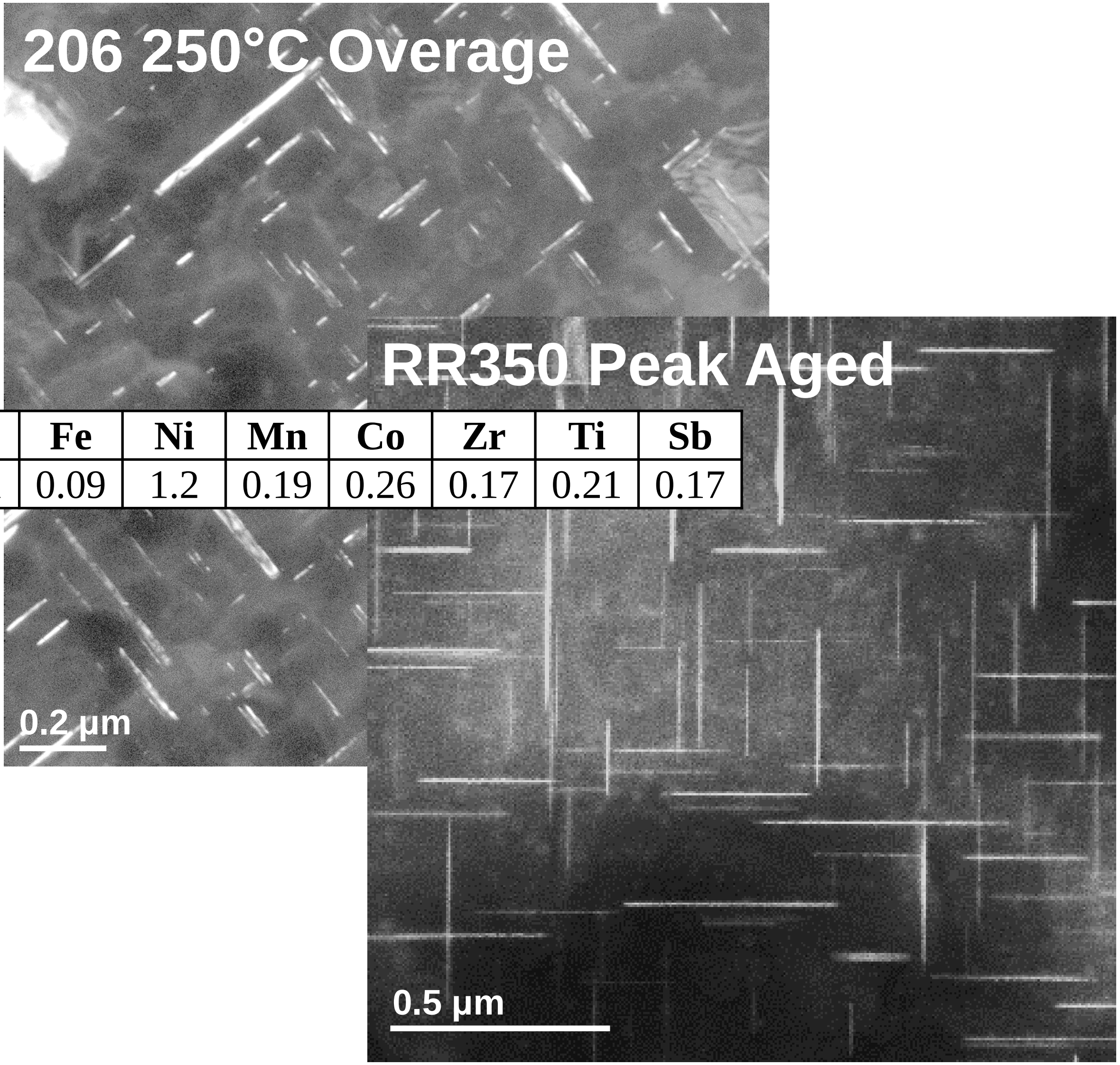
Bringing it all back together

- Plot shows slope from previous slide (“effectiveness parameter”) compared to area of shearing interface
- Appears as if transition between regimes occurs near 200°C overaged condition
- Note that the correlation occurs in all conditions – even ones without θ′
- May be able to apply similar logic to different precipitates



Adding another alloy – and another variable

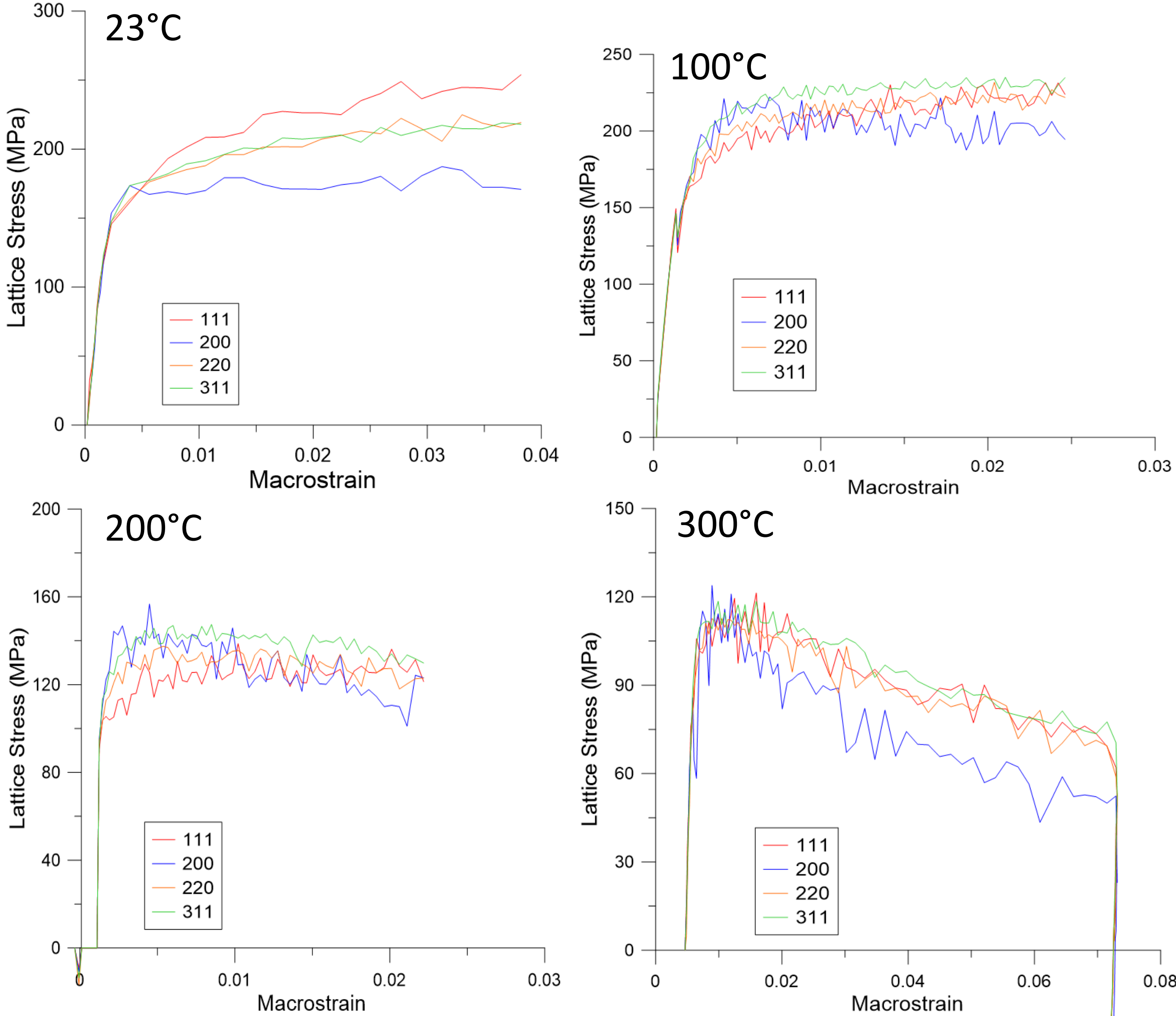
- RR350 is an alloy with a very thermally stable precipitate structure
- Will be varying testing temp with constant microstructure
- As-aged microstructure similar to 206 250°C overage



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

First look at RR350 elevated temperature data

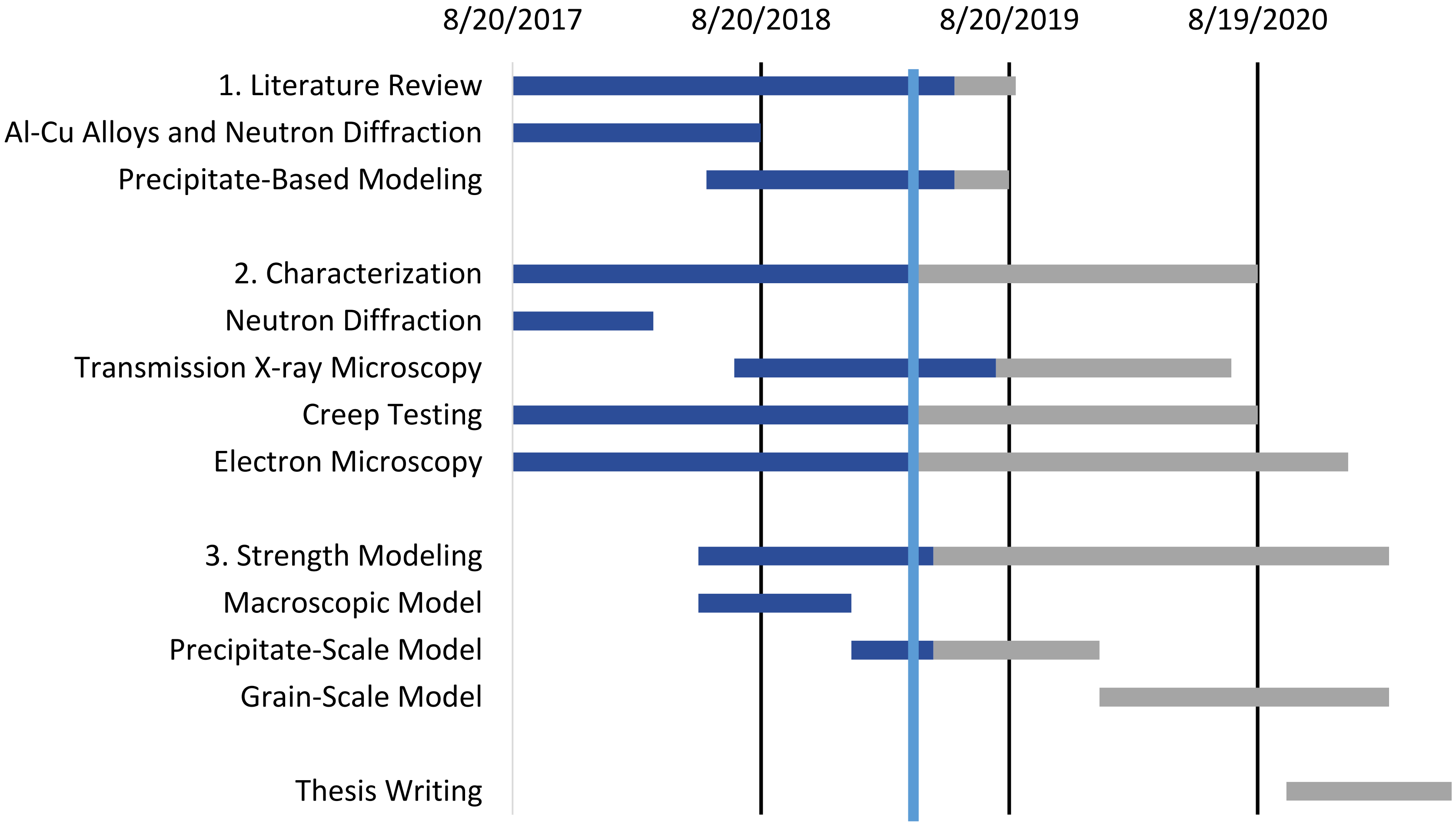
- Seems to be following similar grain orientation relationship
- Anisotropy seems to be lessening with increasing temperature
- Relationship will likely be more complicated than room temperature



Conclusions

- 206 shows a dependence of grain orientation on deformation mechanisms
- This behavior can be described with a simple model
- Magnitude of this dependence varies with precipitate size
- RR350 seems to follow similar behavior at room temperature, with lower magnitude of anisotropy with increasing temperature
- Next steps: Additional TEM and applying new energetic analysis to existing strain hardening models

Progress


8/20/2017
8/20/2018
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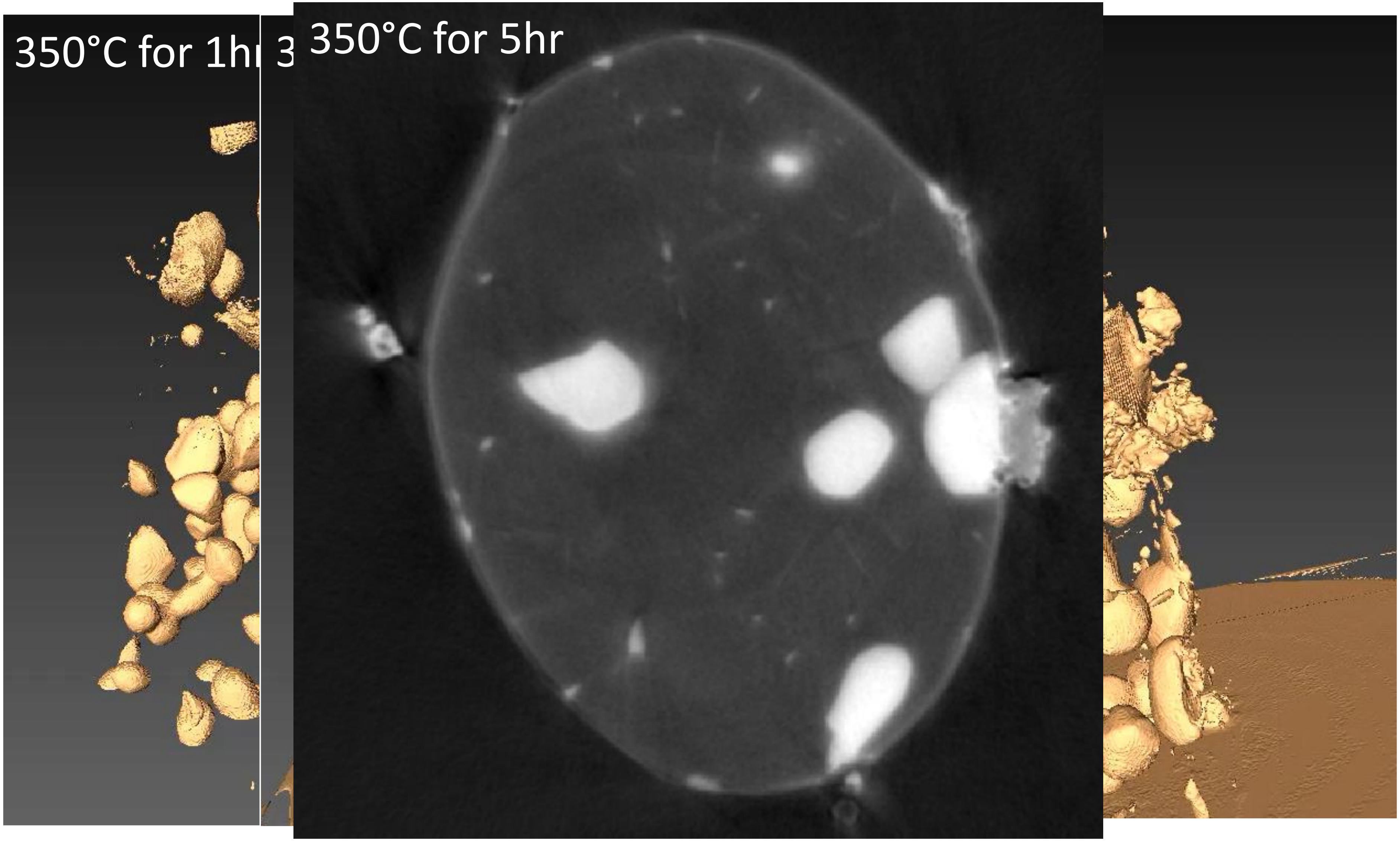
8/19/2020
1. Literature Review
Al-Cu Alloys and Neutron Diffraction
Precipitate-Based Modeling
2. Characterization
Neutron Diffraction
Transmission X-ray Microscopy
Creep Testing
Electron Microscopy
3. Strength Modeling
Macroscopic Model
Precipitate-Scale Model
Grain-Scale Model
Thesis Writing

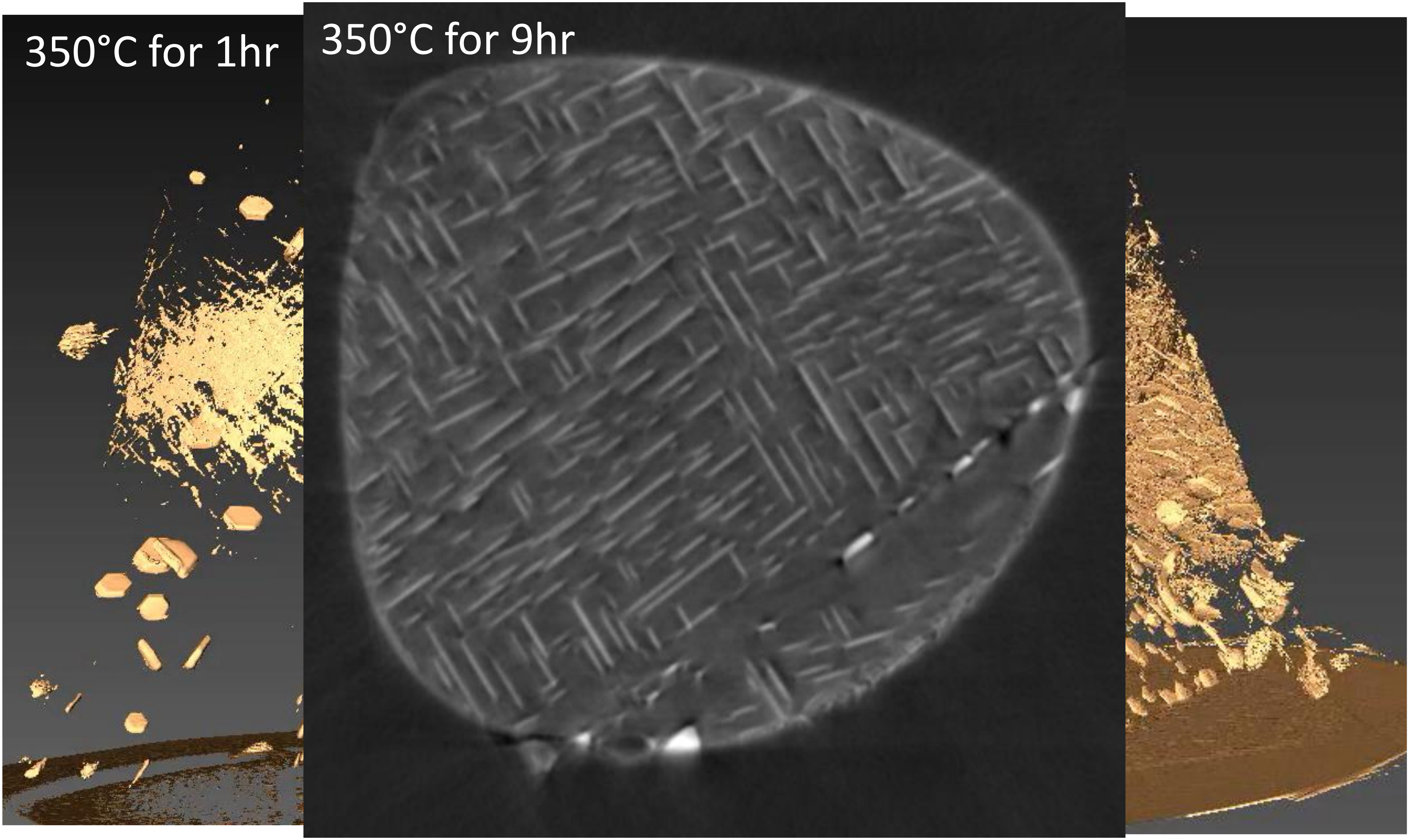
Thank you for your attention!

Questions?

3-D precipitate evolution in Al-5at%Cu observed with TXM



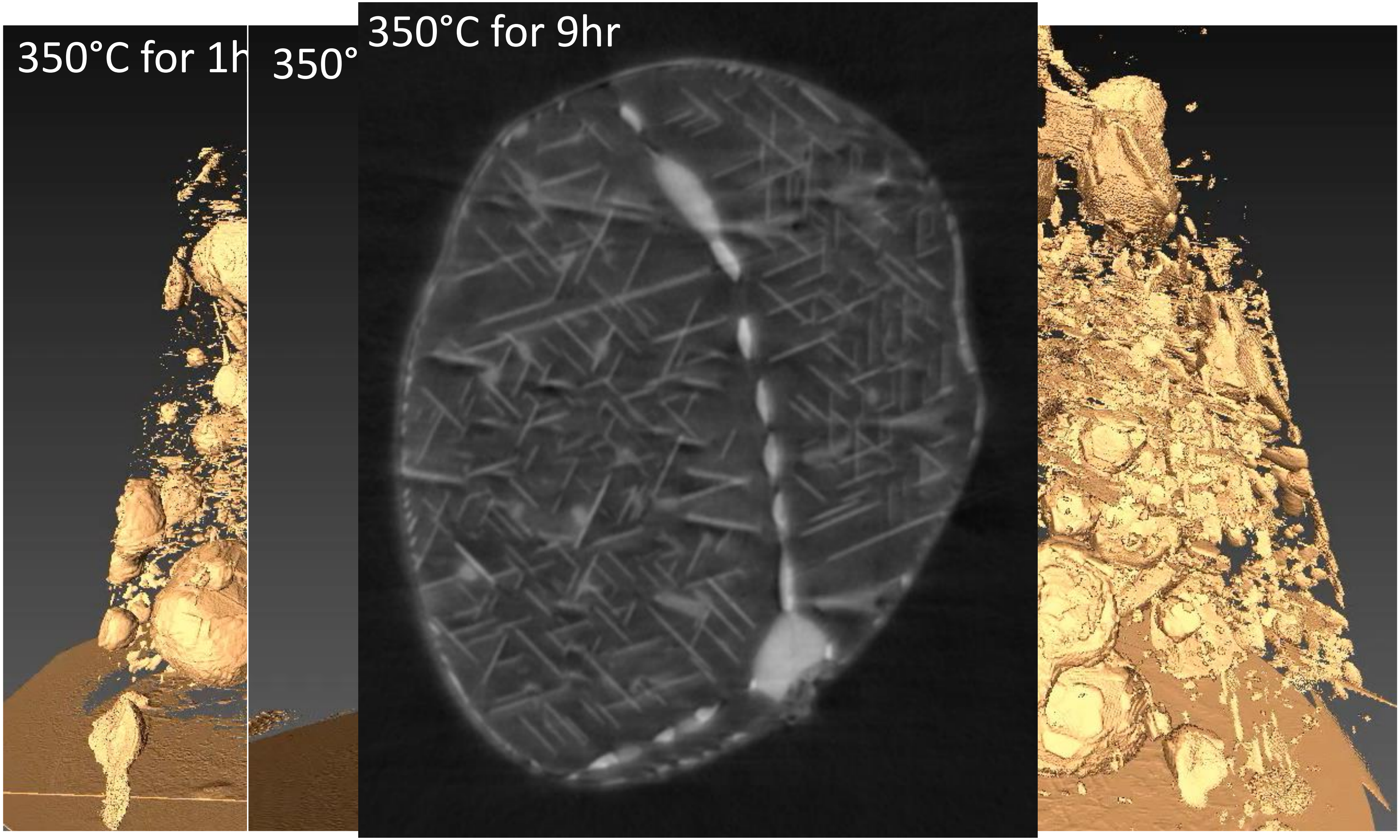
3-D precipitate evolution in Al-5at% Ag observed with TXM



350°C for 1hr

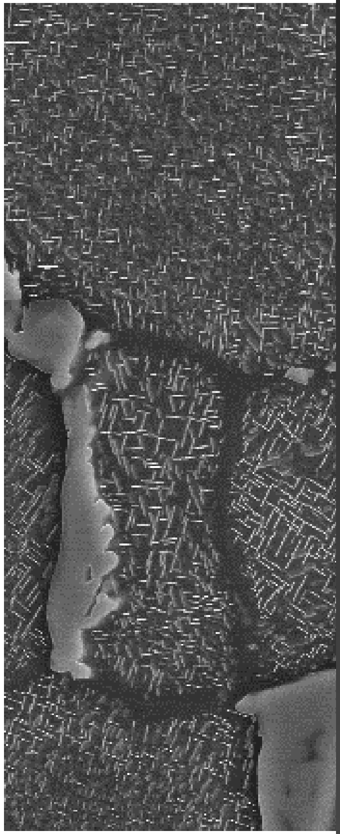
350°C for 9hr

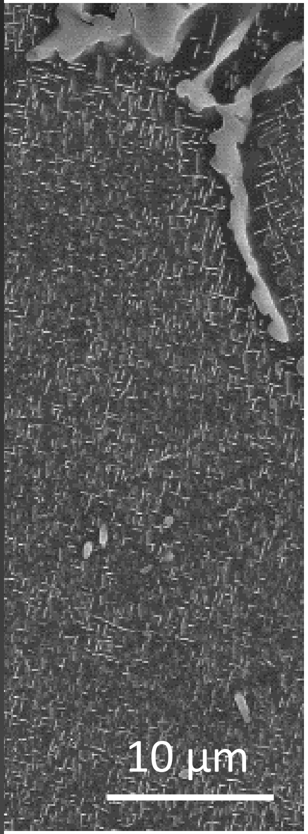
3-D precipitate evolution in Al-5at%Ag-5at%Cu observed with TXM



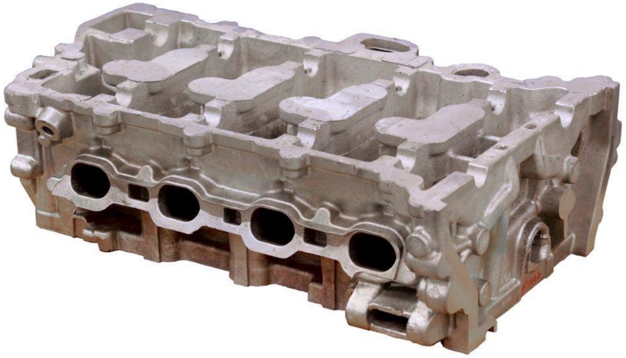
Extra Slides: Intergranular Precipitat

- “Chinese sc all Al, Cu, and Ni cont





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



Student: *Brian Milligan*

Faculty: *Amy Clarke*

Industrial Partners: *ORNL (Amit Shyam)*

Project Duration: *Sept. 2017 – May 2021*

Achievement

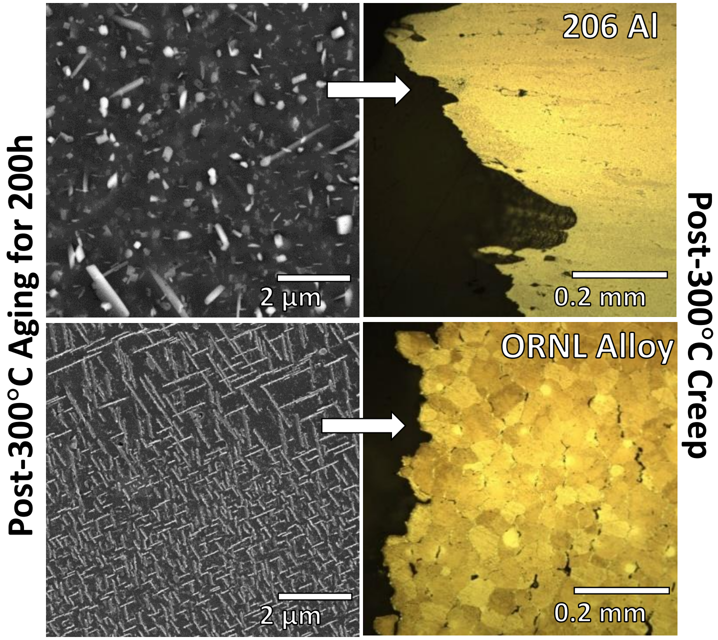
- Modeling of creep behavior in commercial and experimental Al-Cu alloys at high homologous temperature

Significance and Impact

- Thermally stable Al-Cu cylinder head alloys developed at ORNL outperform commercial alloys during creep loading, allowing for higher engine operating temperatures

Research Details

- Performed creep experiments and developed new low-stress microstructure-based creep model using results



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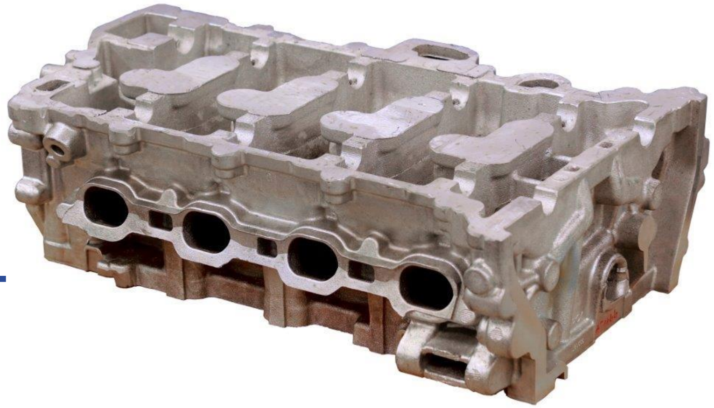

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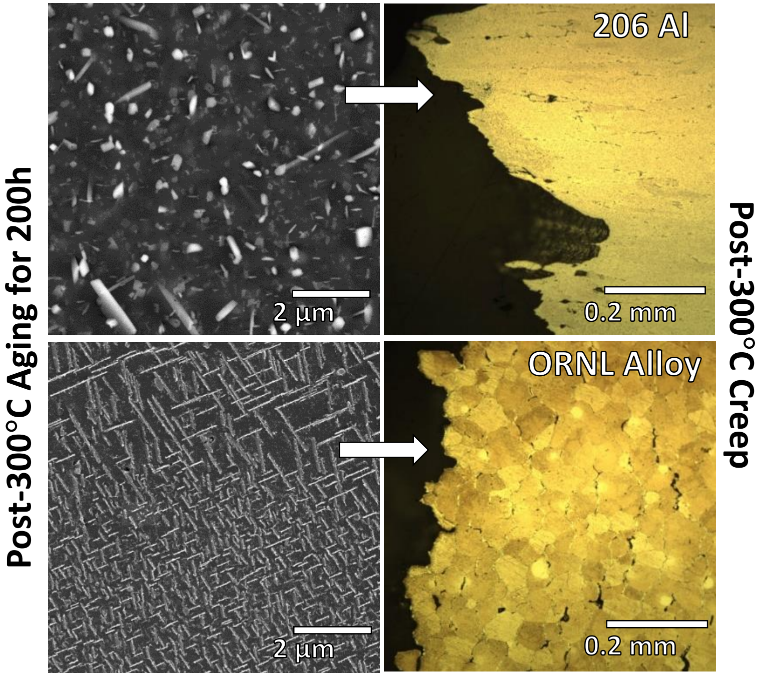

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Program Goal

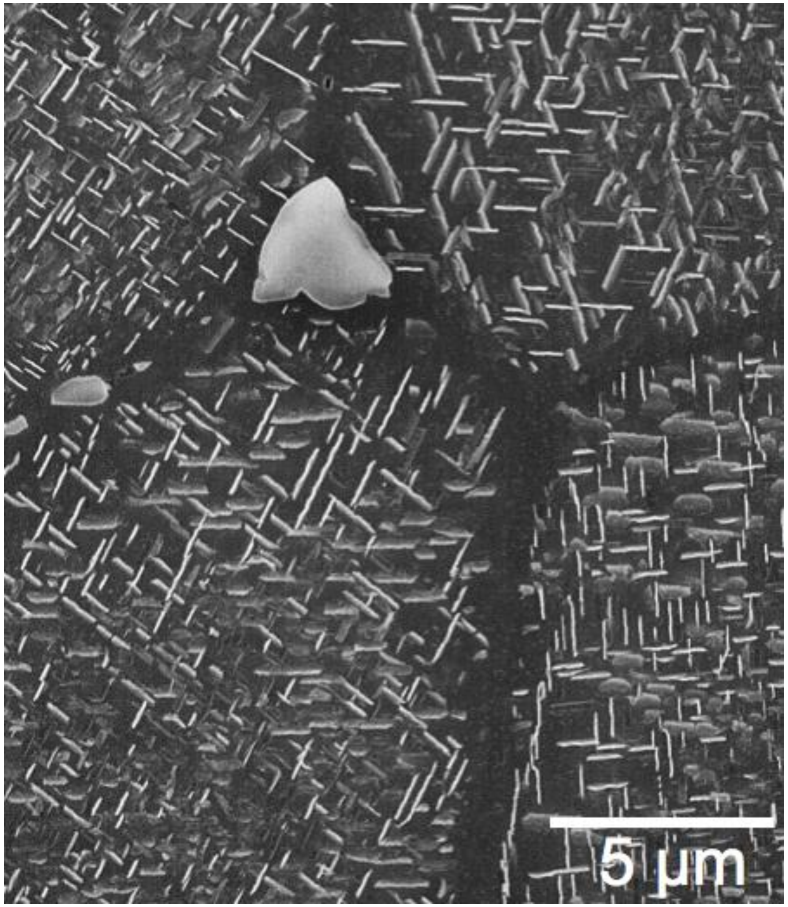
- Characterize the mechanical properties and microstructure of thermally stable Al-Cu alloys under various loading and aging conditions

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