

## ***Project 22: Development of Novel High Temperature Aluminum Alloys***

***Spring 2019 Semi-Annual Meeting  
Iowa State University, Ames, IA  
April 3-5, 2019***

*Student: Joe Jankowski (Mines)*

*Faculty: Michael Kaufman, Amy Clarke, Robert Field, Steve Midson (Mines)*

*Industrial Mentors: Krish Krishnamurthy (Honeywell), Paul Wilson (Boeing)*



# Project 22: Development of Novel High Temperature Aluminum Alloys



- Student: Joe Jankowski (Mines)
- Advisors: Michael Kaufman, Amy Clarke (Mines)

**Project Duration**  
PhD: June 2015 to August 2019

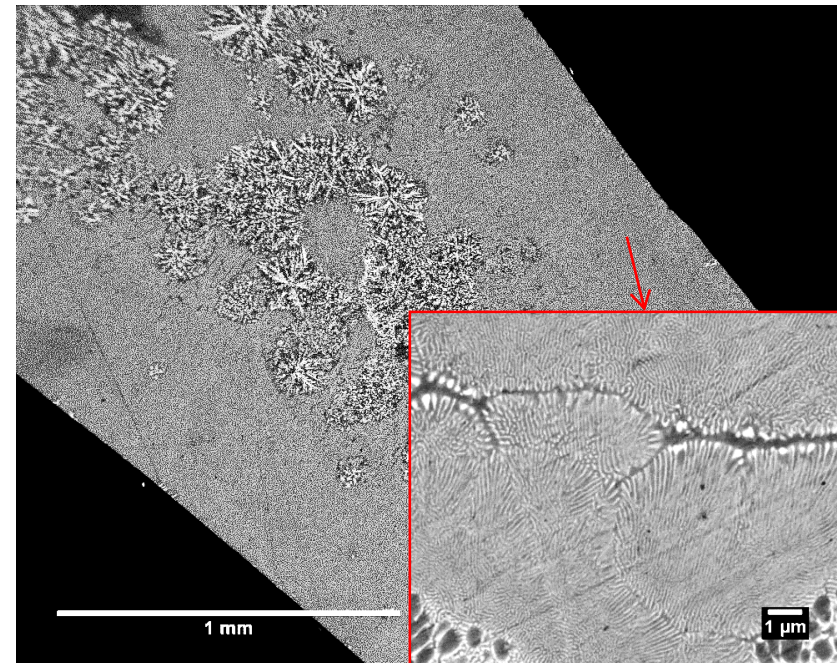
- **Problem:** Aluminum alloys with acceptable high temperature structural properties are expensive and difficult to produce.
- **Objective:** Develop high-temperature, high-strength Al alloys without use of rapid solidification by forming stable microeutectic.
- **Benefit:** Reduce production cost and increase selection of high performance high-temperature Al alloys.

- Recent Progress**
- Processing-microstructure study
  - Gas tungsten arc welding (GTAW) autogenous welding study feasibility demonstration
  - Compression testing of J35 alloy
  - SEM characterization of J35 microstructure
  - High accuracy density functional theory (DFT) modeling of  $\alpha$ -phase

Metrics		
Description	% Complete	Status
1. Develop experimental protocols for reproducible castings	100%	●
2. Mechanical testing and characterization of microeutectic-containing microstructures	90%	●
3. Develop crystallography / phase stability knowledge of $\alpha$ -phase	100%	●
4. Assess ability to produce microeutectic in chill castings	100%	●
5. Determine how fundamental solidification parameters affect microeutectic formation	50%	●

# Project Motivation

- Microeutectic between Al and  $\alpha\text{-Al}_{13}(\text{Fe},\text{V})_3\text{Si}$  in chill castings
- Hardness of microeutectic similar to RS8009
- Lower cooling rate than rapidly solidified alloys
  - $10^2\text{-}10^3$  K/s vs.  $10^4\text{-}10^6$  K/s
- Potential high-temperature Al structural alloy

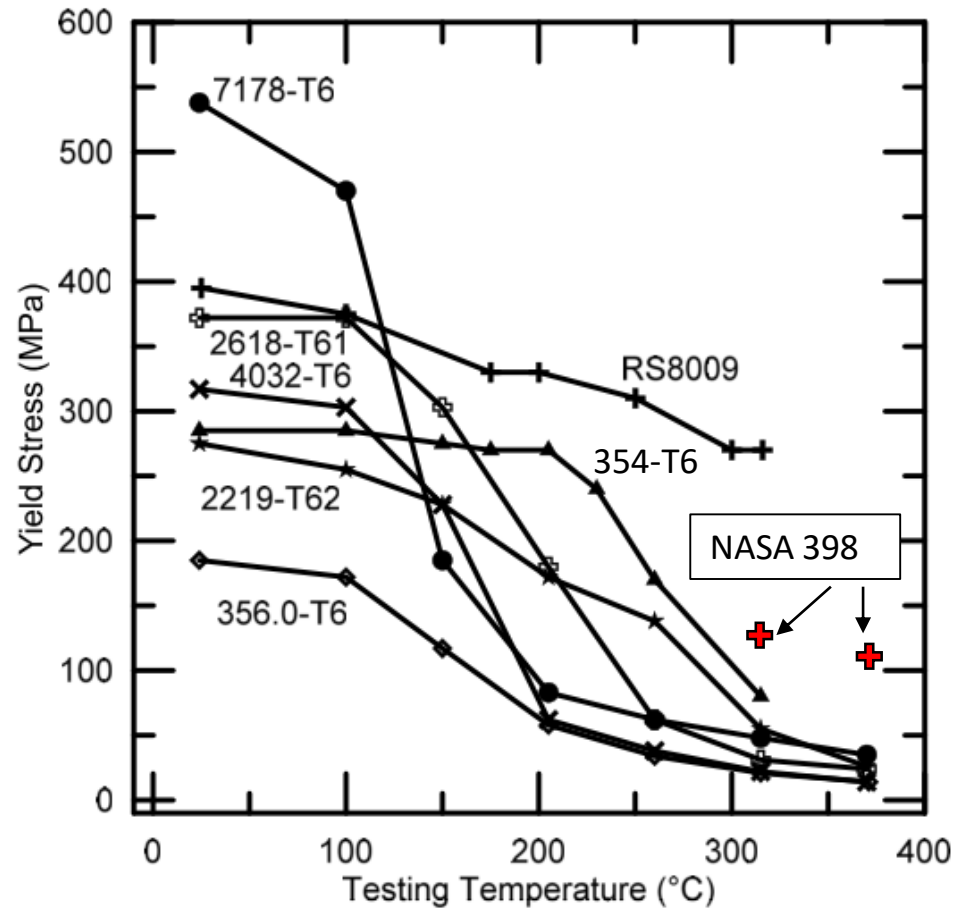


Alloy	Al (at%)	Fe (at%)	V (at%)	Si (at%)
RS8009	Bal.	4.3	0.7	1.7

# Industrial Relevance

- Development of a lower cost high-temperature Al structural alloy
- Identify alternative processing routes for high-temperature Al alloys
- Rapid screening of composition space for nonequilibrium systems

\*R. Marshall. Master's Thesis. (2016).



Mechanical properties of selected Al alloys (RS8009 is high-temperature alloy)\*



# Presentation Overview



- Prior Work
- Recent Progress
- Project Timeline

# Prior Work



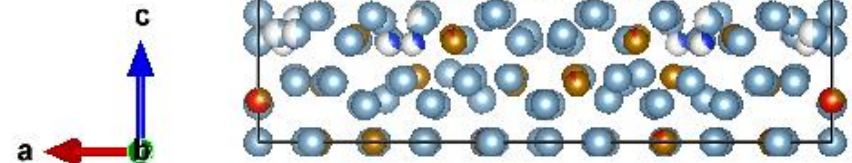
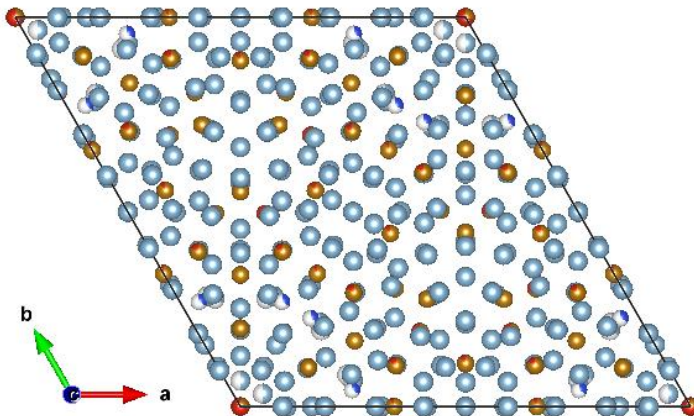
**Goal:** Develop alloy with high volume fraction of microeutectic constituent, improve understanding of system

## **Completed work**

- Structure determination of deleterious h-phase using powder diffraction
- Develop method of using density functional theory (DFT) to screen compositions
- Experimental validation of DFT
- Determination of cooling rates in Cu chill mold
- Selection of final alloys

# h-Phase Crystal Structure

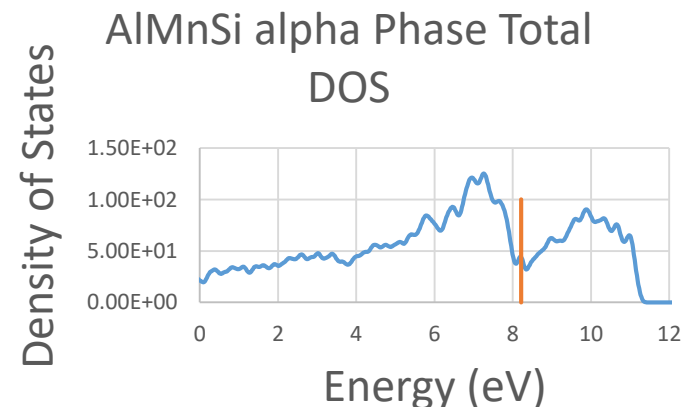
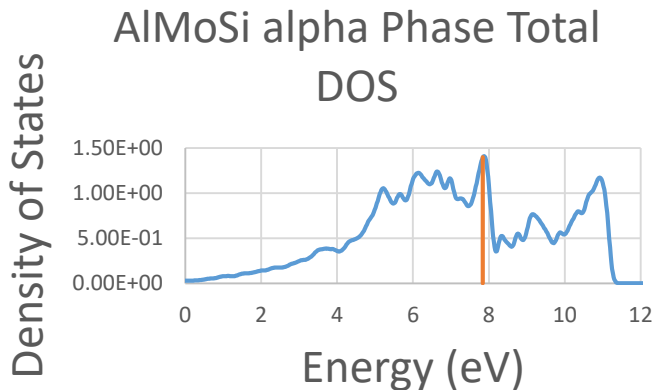
- Most complex intermetallic crystal structure solved by powder diffraction, ~472 atoms per unit cell, 4 different elements present
- Used synchrotron x-ray and neutron powder diffraction



# DFT Composition Screening

- Density of states (DOS)
  - Relatively inexpensive calculation
  - Large unit cells for  $\alpha$  and h-phase
  - Easier to get useful DOS vs. enthalpy/charge density
- Based on actual physics of system
- Intuitive visual representation of bonding interactions in 2D

$\alpha$ -AlXSi ternary phase	Area under curve from "stable" to calculated Fermi level	Deep pseudogap
Ti	-245	Yes
V	-117	Yes
Cr	-49	Yes
Mn	0 (reference)	Yes
Fe	22	Yes
Co	120	Borderline
Ni	145	No
Cu	130	No



# DFT Validation

- Method assesses possibility, does not determine if something will actually form
- Best for extrapolation from known system, for example Al-Fe-V-Si
- Results for  $\alpha$ -phase are promising
- Potential to accelerate alloy design in **all metallic systems**

Alloy System	Expected to form $\alpha$ -phase from theory?	Is $\alpha$ -phase experimentally observed?
Al-Fe-V-Si	Yes	Yes
Al-Fe-Cr-Si	Yes	Yes
Al-Co-Ti-Si	No	No
Al-Co-Cr-Si	No	No
Al-Fe-Mn-Cr-Si	Yes	Yes

# Final Alloy Selection



- Chosen with processability in mind
  - Equilibrium liquidus
- Cr vs. V
- V-containing alloys may contain h-phase or low-Si  $\alpha$ -phase
  - From WDS/XRD analysis

Alloy	Liquidus (°C, approx.)	Total TM (at%)
J35	730	3.4
J40	760	4.1
J45	780	4.6
J50	800	5.1
RS8009	850	5.0

Composition (at%)

Alloy	Al	Fe	Mn	Cr	Si
J35	Bal.	1.5	1.5	0.4	1.6
J40	Bal.	1.8	1.8	0.5	1.8
J45	Bal.	2.0	2.0	0.6	2.1
J50	Bal.	2.2	2.2	0.7	2.3



# Recent Progress



- Processing-microstructure study
- Compression testing
- GTAW autogenous welding solidification experiment

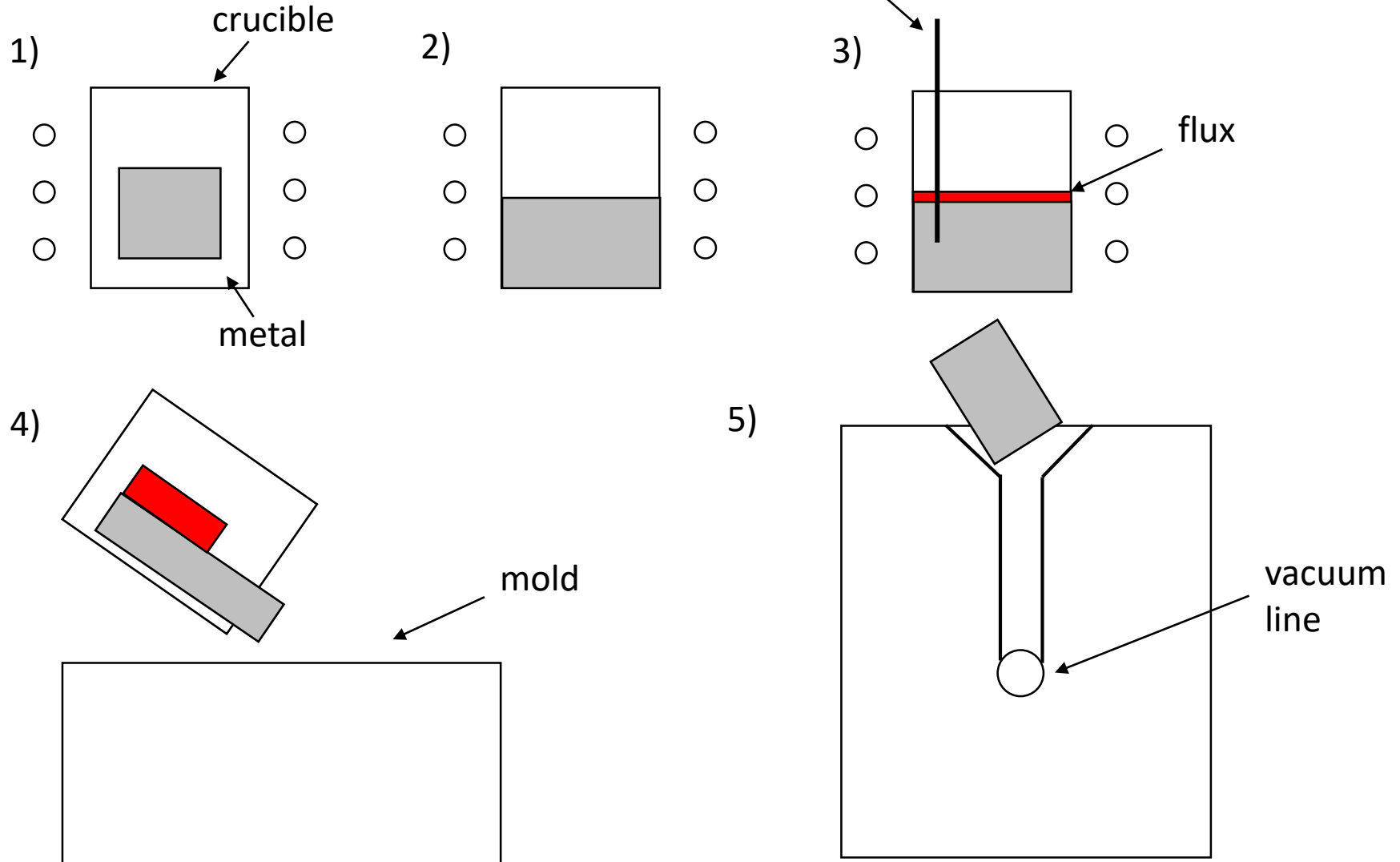
# Processing-Microstructure Study



**Goal:** Develop links between processing conditions and microstructure in chill mold castings

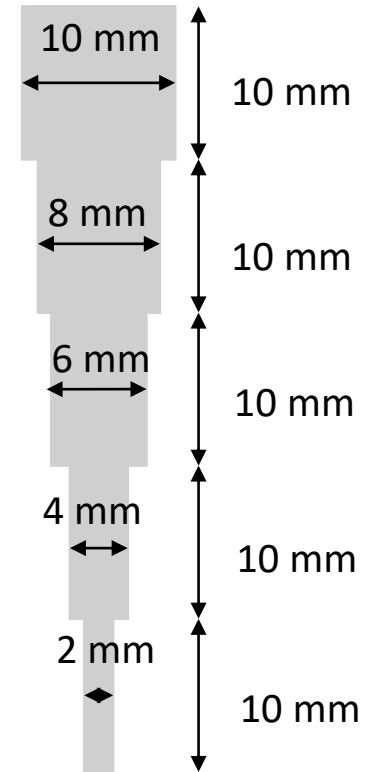
- Alloys being studied are highly processing sensitive
- Need to identify robust processing conditions
- Evaluate ability to produce microeutectic microstructure consistently

# Experimental Setup



# Variables in Study

- Melt superheat
  - Shown to be critical to microstructure formed
- Mold geometry / cooling rate
  - Stepped cylindrical mold shape
  - Variable solidification conditions in mold
- Alloy composition
  - Transition metal level has strong effect on microstructure



Composition (at%)

Alloy	Al	Fe	Mn	Cr	Si
J35	Bal.	1.5	1.5	0.4	1.6
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# Results of Study



- Difficult to find conditions that produce consistent microstructures through thickness
  - Found one particularly promising condition—J35 w/ 200 C superheat (6mm)

## Additional results

- Higher transition metal content enhances  $\alpha$ -phase nucleation
  - Narrows processing window for coupled growth
- Lower transition metal content promotes primary Al growth
  - Al dendrites with interdendritic Al- $\alpha$  microeutectic

# Mechanical Testing



**Goal:** Determine if microeutectic-containing alloys have high strength and microstructural stability at elevated temperatures

- Room + elevated temperature compression
  - Room temperature tests performed with 20 kip hydraulic MTS frame
  - Elevated temperature tests performed with Gleeble thermomechanical simulator
  - $10^{-3} \text{ s}^{-1}$  strain rate
- Evaluate degradation of strength at temperature over time
- Link performance to microstructures through characterization

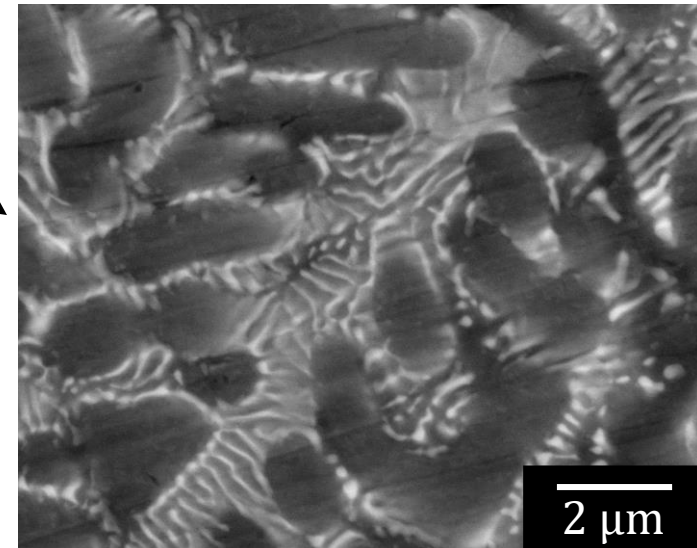
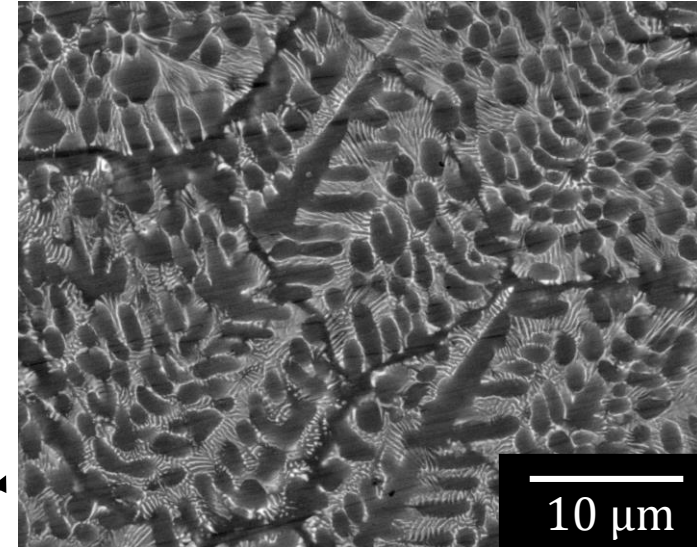


# J35 Microstructure Overview

- Previous mechanical testing identified “primary Al” as most desirable solidification morphology in alloys J35 and J40
  - Microhardness
  - Compression testing
- J35 cast at 200 °C superheat w.r.t. equilibrium liquidus (6 mm diameter)
- Cooling rates <1,000 K/s
  - Thin-walled die casting
  - Twin roll strip casting\*
- “primary Al”
  - Al dendrites (appear to be primary)
  - Solute-enriched lamellar microeutectic

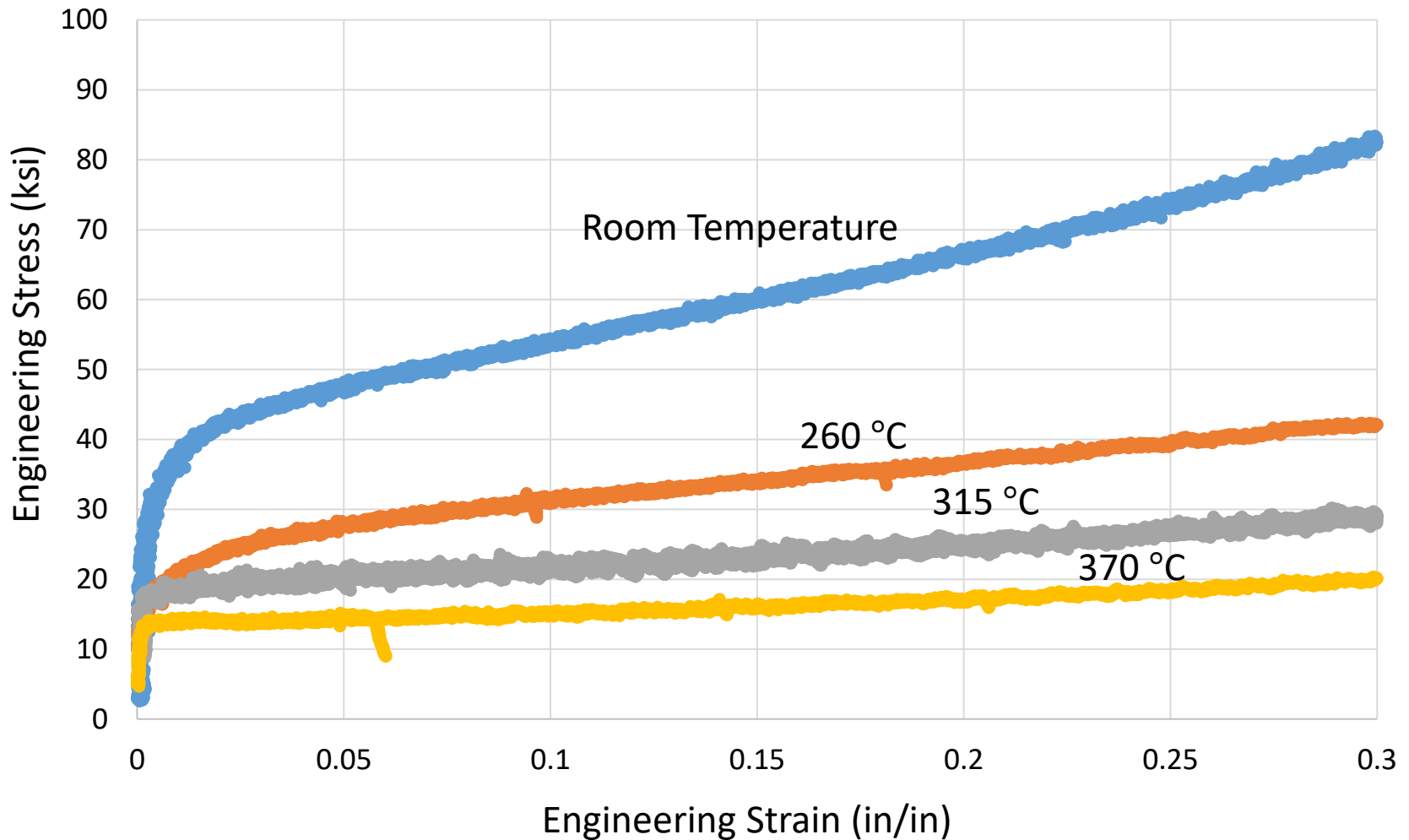
\*P.N. Anyalabechi. EPD Congress. (2004).

BSE images



# J35 Mechanical Properties

## J35 Stress-Strain Curves After 0.5hr at Temperature

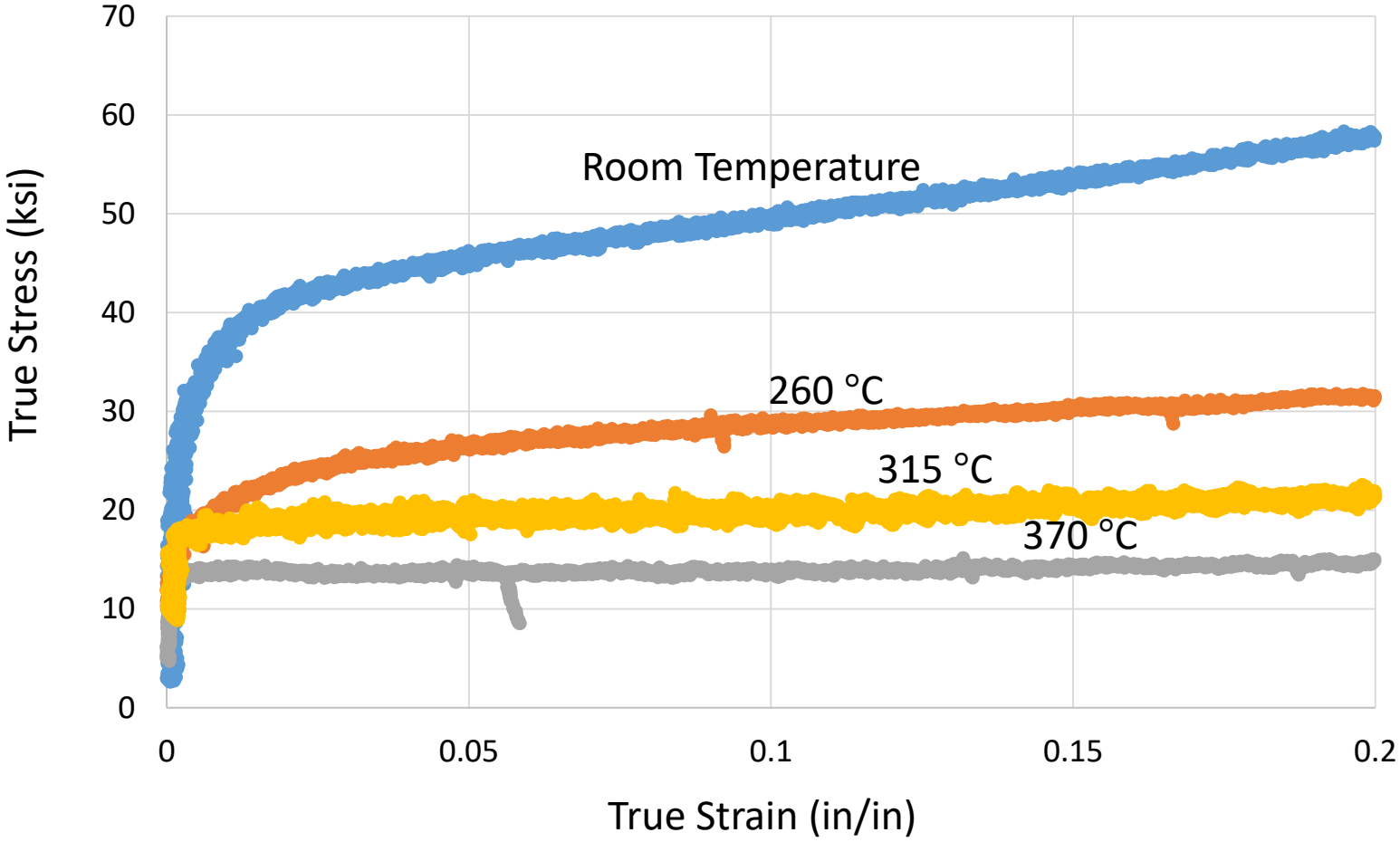


# J35 Mechanical Properties



estimate

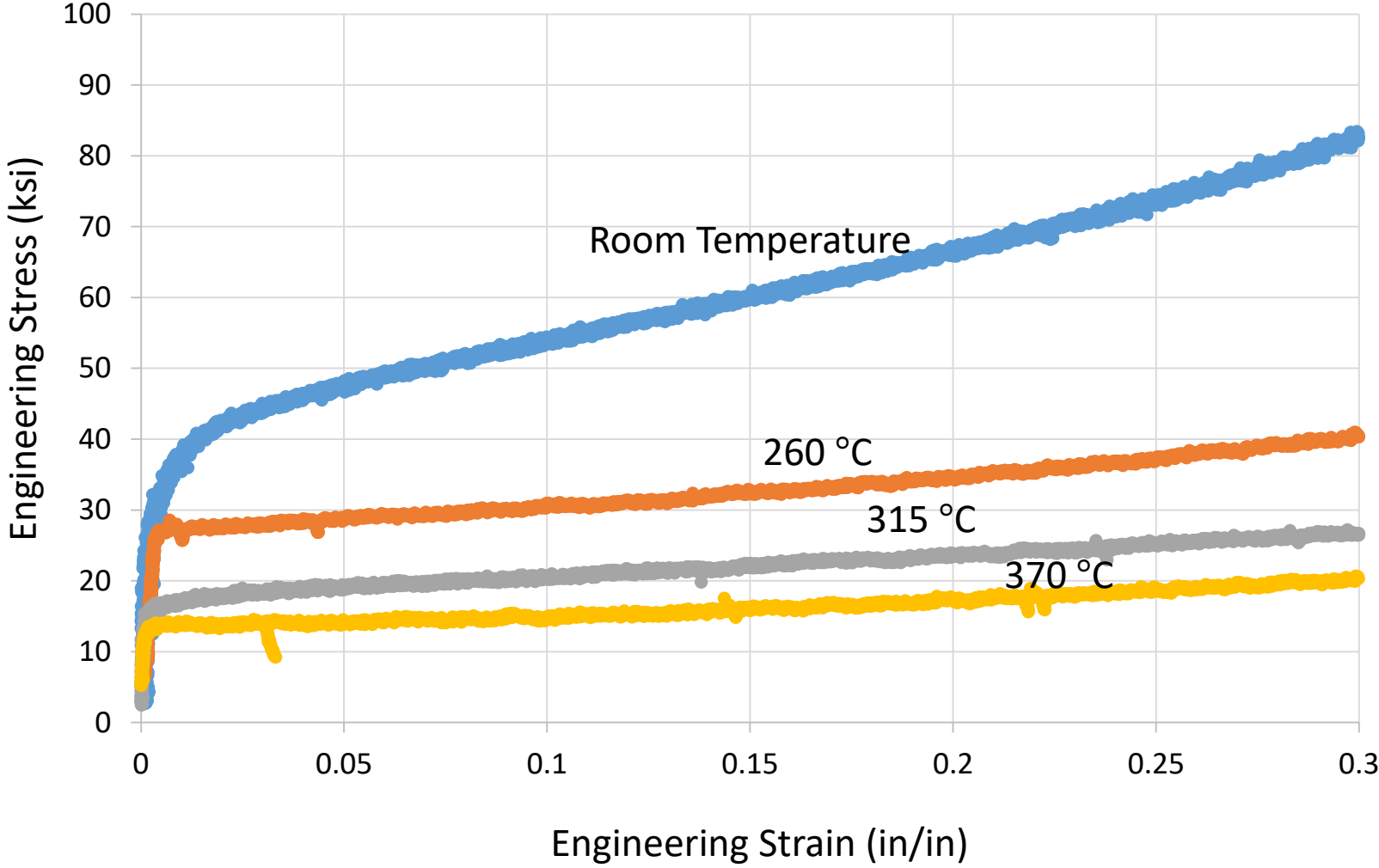
### J35 Stress-Strain Curves After 0.5hr at Temperature



# J35 Mechanical Properties



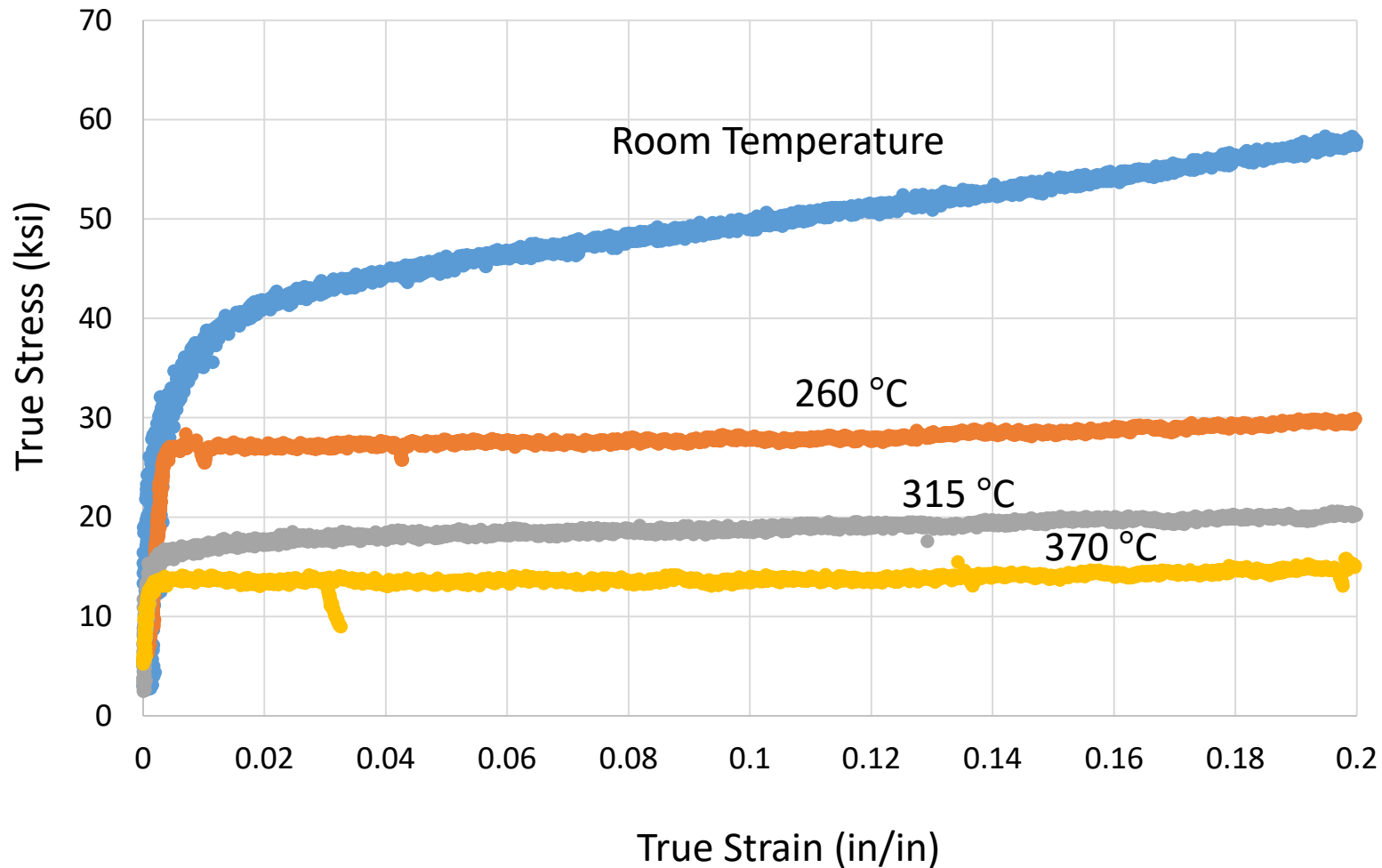
J35 Stress-Strain Curves After 100hr at Temperature



# J35 Mechanical Properties

estimate

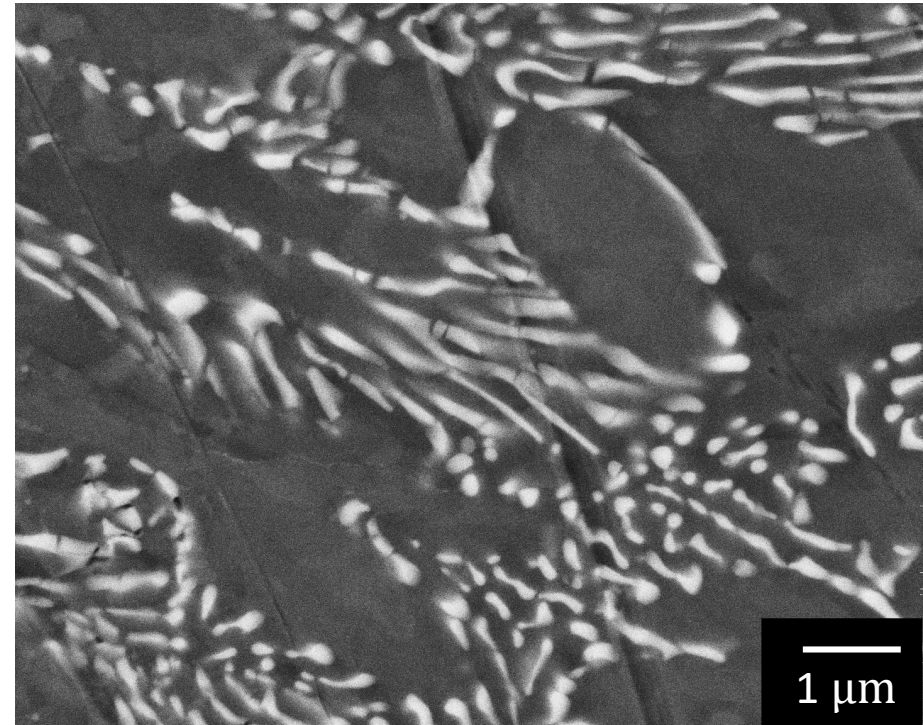
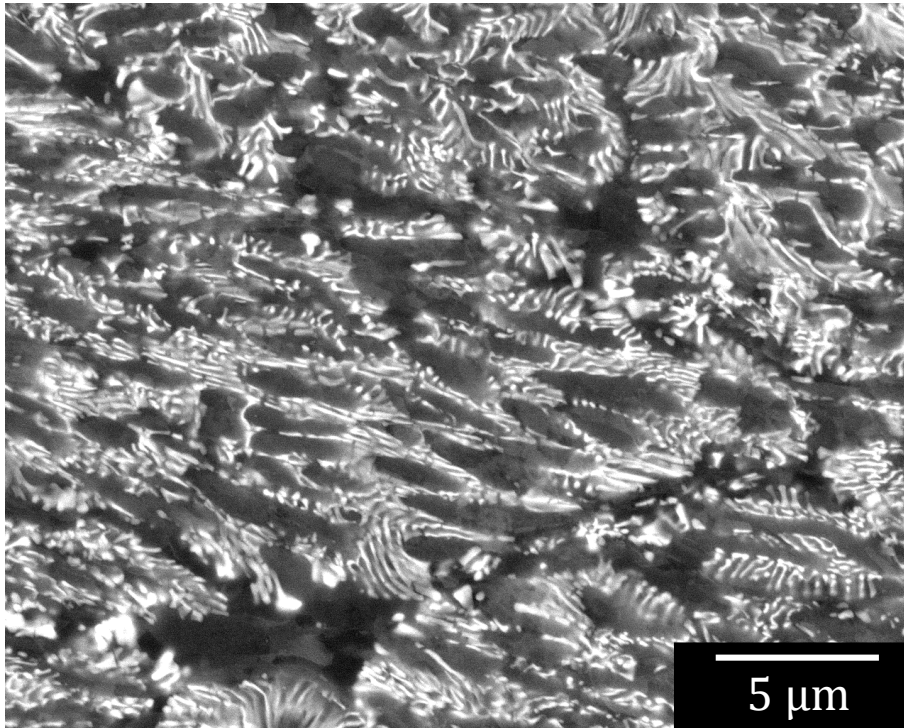
### J35 Stress-Strain Curves After 100hr at Temperature





# Deformed Microstructure

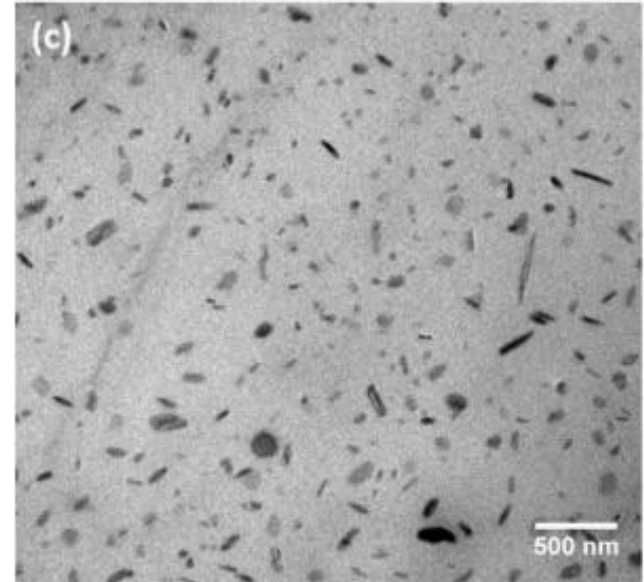
- J35, 100 hr at 260 °C
- Deformation breaks up dendritic as-cast microstructure
- Possibility for refinement of structure through deformation





# Potential Aging in J35

- 0.2% YS in J35 changed:
  - 20 ksi, 0.5 hr at 260 C
  - 27 ksi, 100 hr at 260 C
- Increase in strength consistent with aging
- $\alpha$ -phase known to precipitate out in solute-rich Al dendrites\*
- Needs further investigation



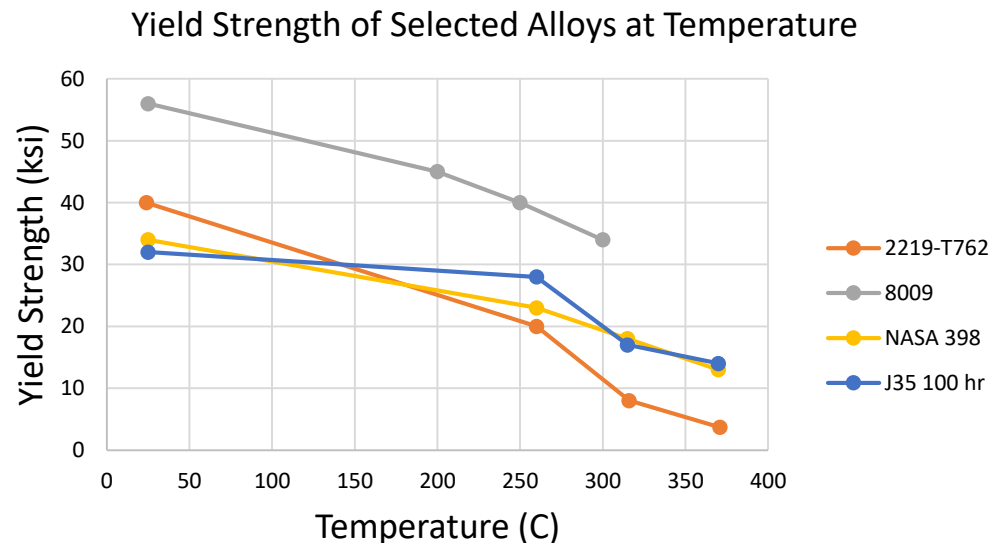
$\alpha$ -Al(Fe,Mn,Mo)Si dispersoids precipitated in an Al dendrite during an aging heat treatment for Al-7Si-0.5Cu-0.3Mg-0.1Fe-0.3Mo-0.5Mn\*

\*A.R. Farkoosh *et al.* (2015)

# Summary of Mechanical Testing



- Possible aging response at 260 °C
  - Further investigation needed
  - Potential to create hierarchical, thermally stable microstructure
- Measured yield strengths comparable to or exceed existing conventionally processed Al alloys
- Negligible degradation of mechanical properties with time
- Samples produced at cooling rates lower than 1000 K/s
  - Thin-walled die casting
  - Twin roll strip casting



# GTAW Solidification Study

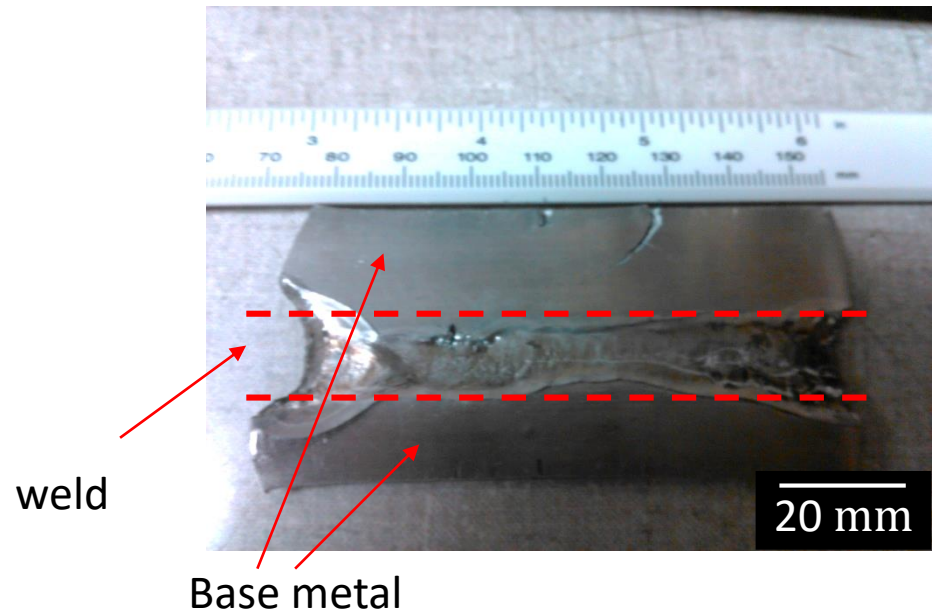


**Goal:** Link as-cast microstructures to fundamental solidification parameters

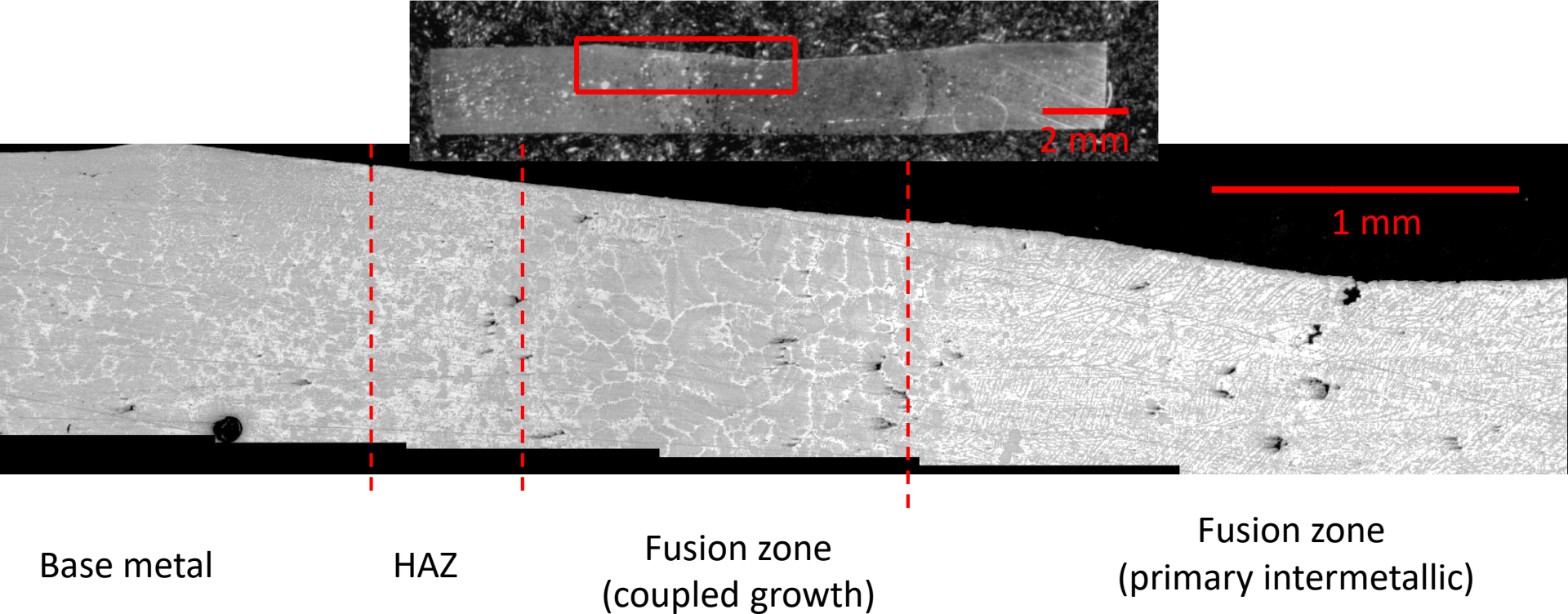
- Controlled solidification velocity
- Can estimate thermal gradients from cooling rates
- More generally applicable than processing-microstructure study

# Demonstration Weld

- Cast J35 plate with dimensions of 40x40x4 mm
- Rolled plate to approximately 80x40x2 mm
- Used GTAW with 110 A AC current, ~4 mm/s travel speed
- Able to achieve a melt pool in the plate



# Weld Microstructure



Base metal

HAZ

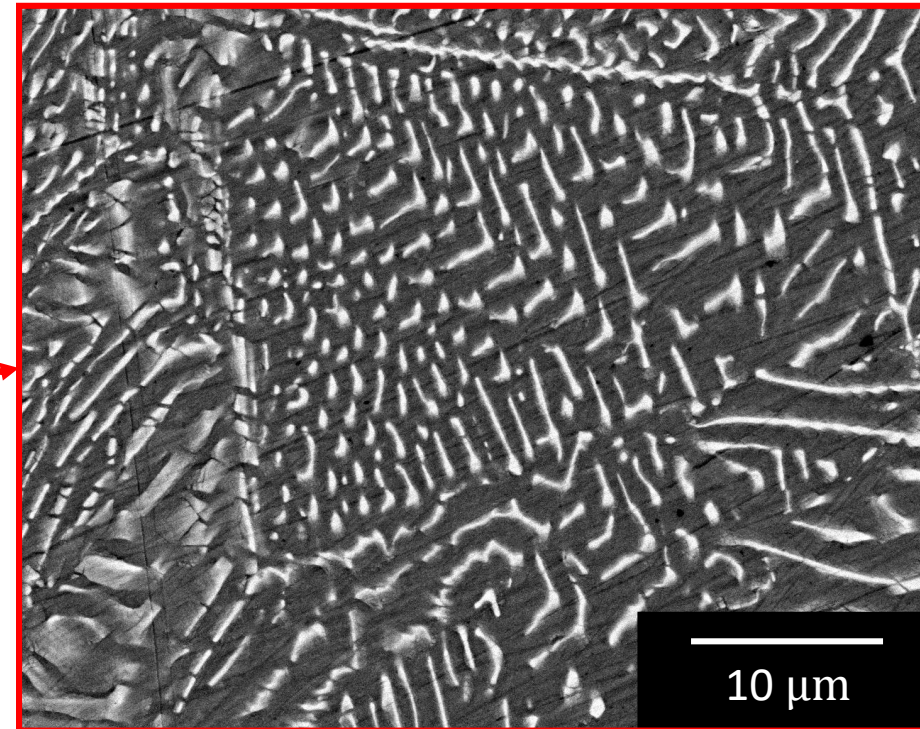
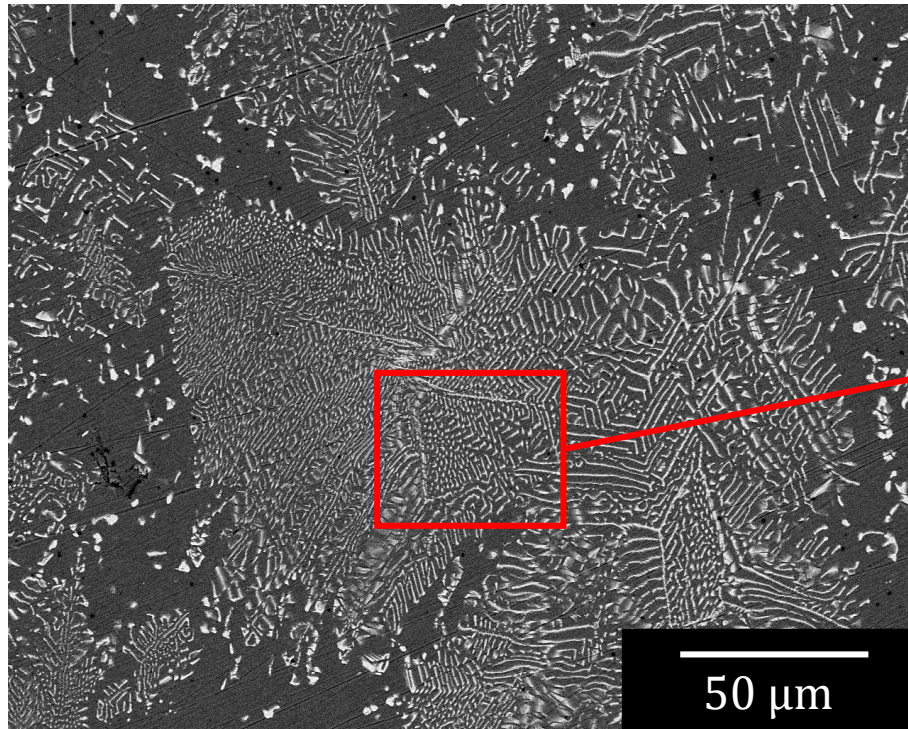
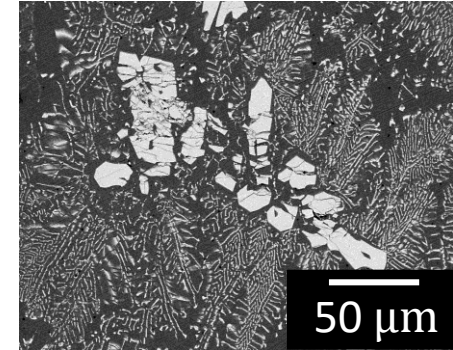
Fusion zone  
(coupled growth)

Fusion zone  
(primary intermetallic)



# Base Metal

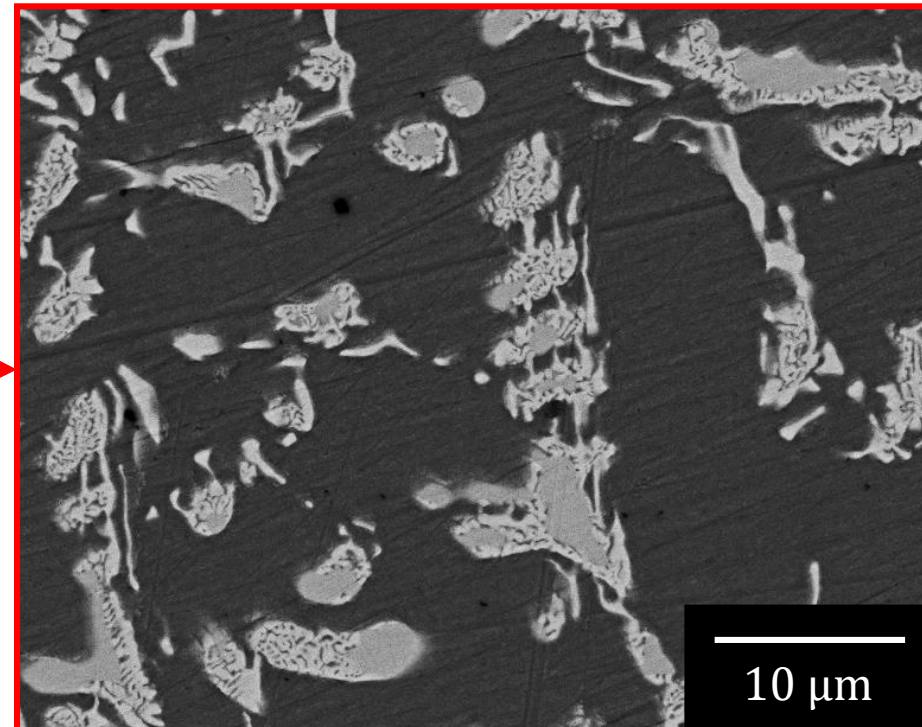
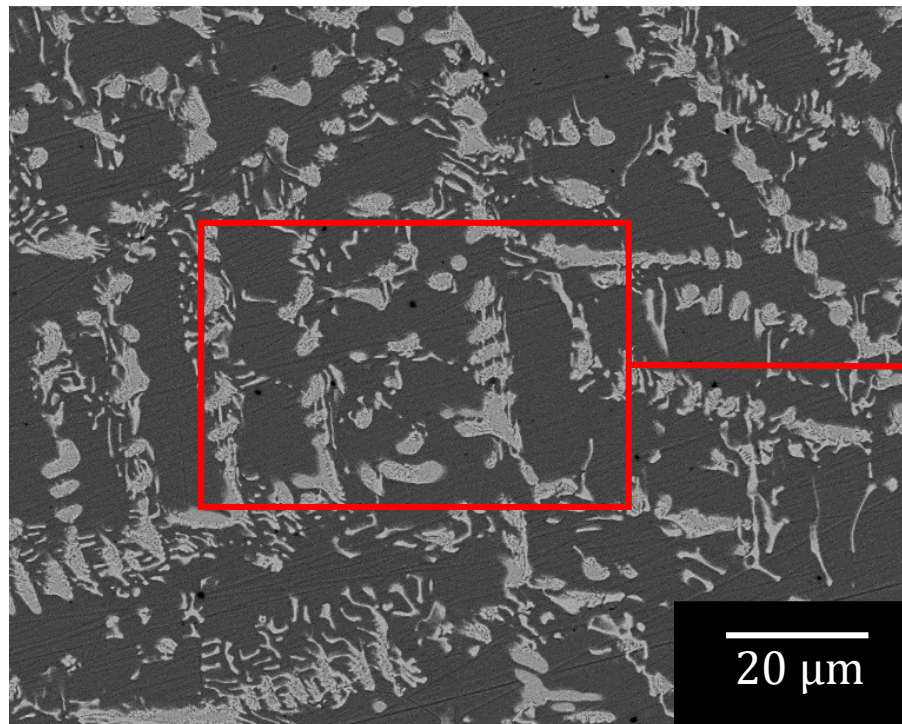
- Consists of coarse primary particles and coarse eutectic
- Fine scale relative to size of melt pool





# Fusion Zone (Center)

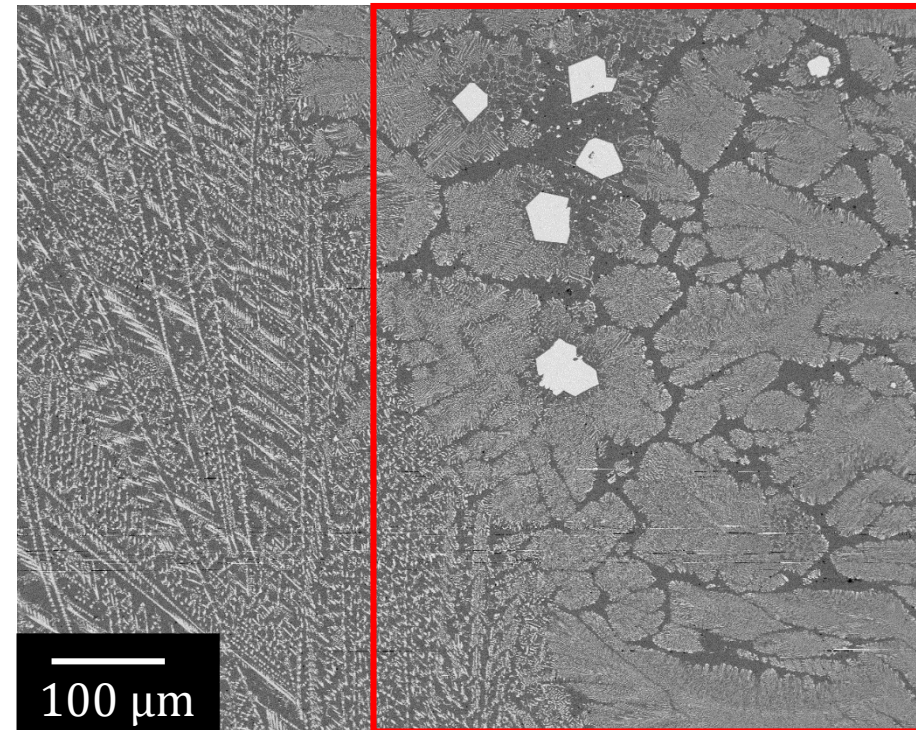
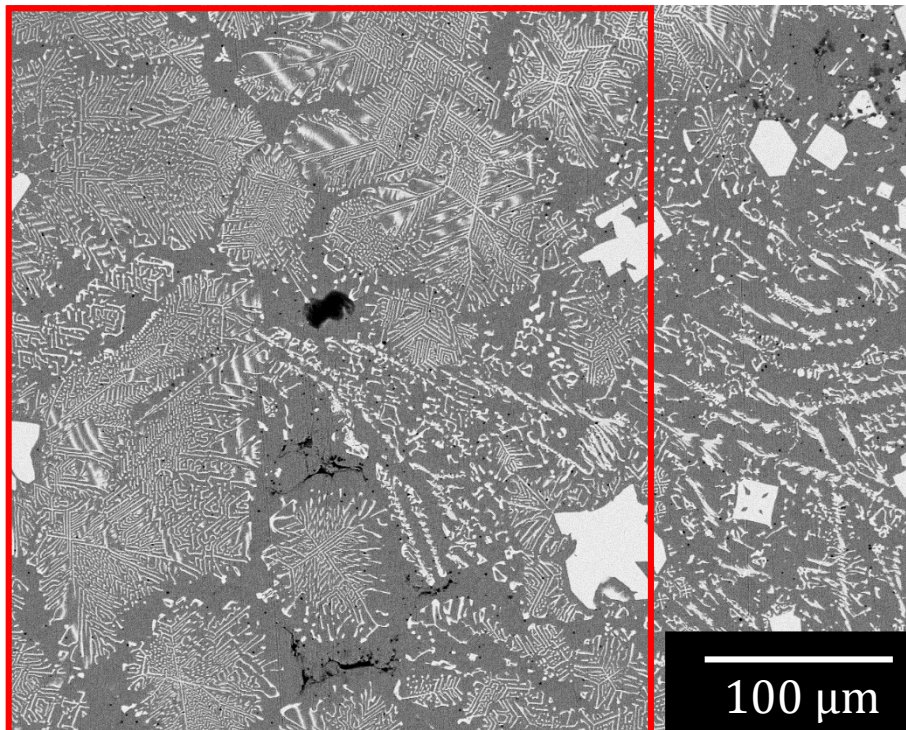
- Expected to have highest solidification velocity
- Microstructure looks like primary intermetallic formation





# Fusion Zone (Edge)

- Expected to have lowest solidification velocity
- “Eutectic islands” at edge of fusion zone
- Appears to contain partially re-melted  $\alpha$  particles



# GTAW Summary



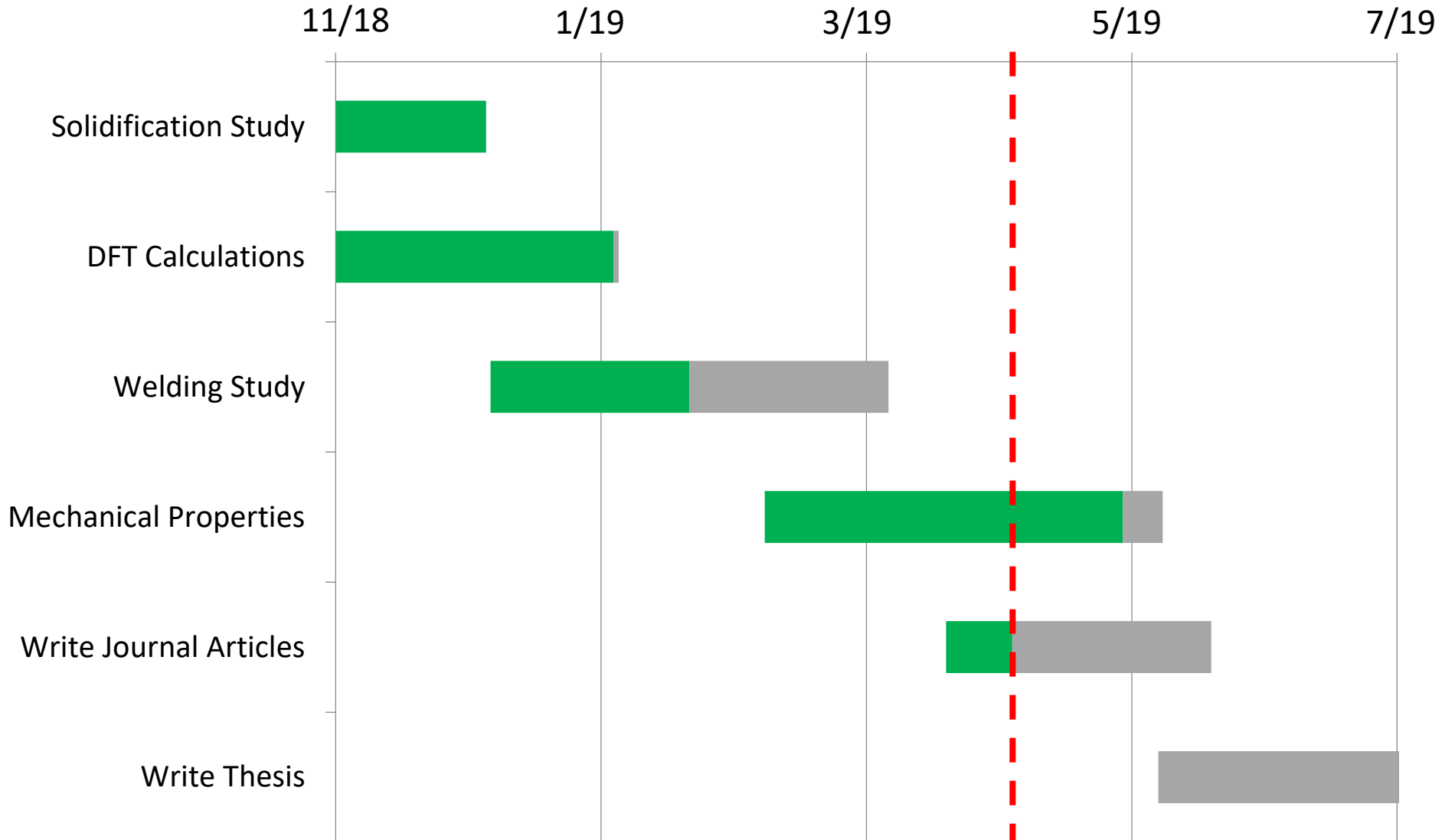
- Have already made welding plates and developed welding experiment for study—largest bottleneck
- Still need to perform autogenous welds at various travel speeds using automated GTAW setup
- Characterize microstructures using SEM
- Use results to link microstructures to solidification velocities, and potentially thermal gradients

# Future Work



- Investigate aging in J35
  - TEM characterization of 100 hr at 260 °C condition
  - Verify results with a second heat treatment
  - Try two-step heat treatment: 100 hr at 260 °C, 100 hr at 315 °C
- Complete autogenous GTAW solidification study
  - Complete welds
  - Characterize welds using SEM
- Write journal articles on completed research
- Write thesis

# Progress



Thank you!

Joe Jankowski

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# Project 22: Development of Novel High Temperature Al Alloys



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## Achievement

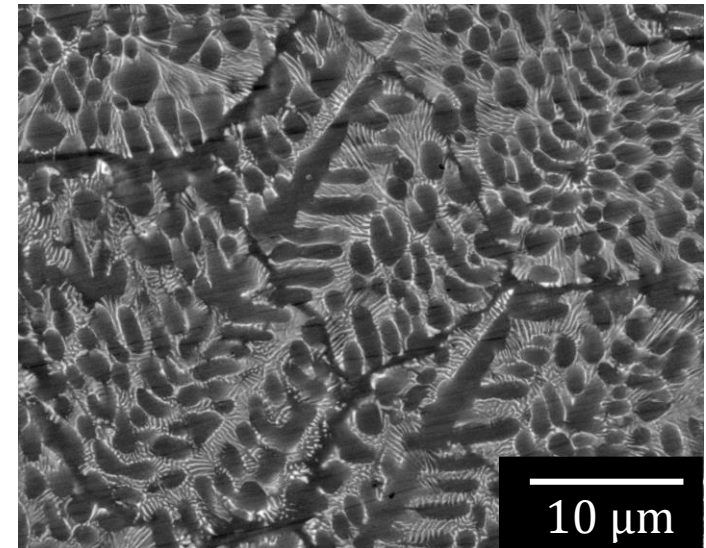
- Production of aluminum alloys with high strength at high homologous temperatures utilizing conventional processing routes, identified using *ab initio* DFT calculations

## Significance and Impact

- Reduced cost of high performance Al structural alloys, improved methods for metastable alloy design.

## Research Details

- Performed DFT calculations to determine promising alloy compositions and identified required solidification conditions to form lamellar microeutectic constituent.



10 μm

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## Program Goal

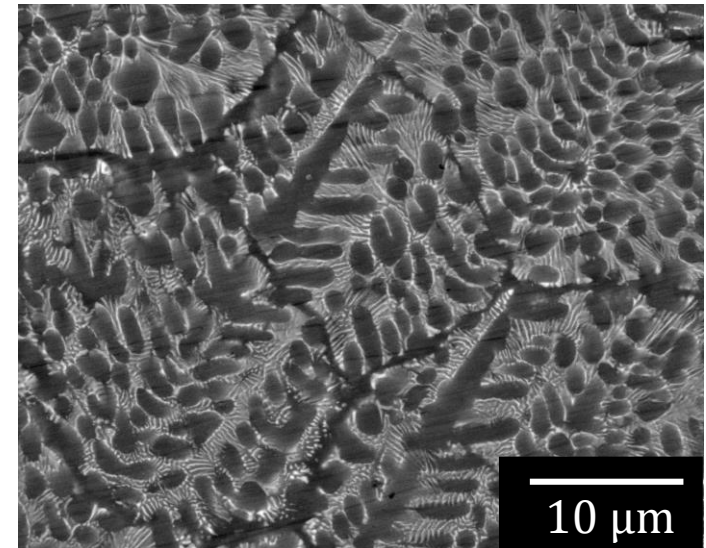
- Develop Al alloys with high volume fraction of microeutectic between Al and  $\alpha$ -phase intermetallic.

## Approach

- Utilize DFT calculations and characterization techniques to develop a new class of Al alloys based on the eutectic between Al and  $\alpha$ -phase.

## Benefits

- Improved scientific understanding of microeutectic, new high performance Al alloys, new method of metastable alloy design.





# Cu Chill Mold Cooling Rates

- Measure of repeatability
- Indicative of solidification conditions
- Evidence cooling rates are between  $10^2$  and  $10^3$  K/s

