

***Project 36E-L: In-Situ Characterization of  
Microstructural Evolution During Simulated Additive  
Manufacturing in Model Alloys***

***Fall Meeting***

***October 13<sup>th</sup> – 15<sup>th</sup> 2020***

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Membership Agreement Apply**

# Project 36E-L: In-Situ Characterization of Microstructural Evolution During Simulated Additive Manufacturing in Model Alloys



- Student: Brian Rodgers (Mines)
- Advisor(s): Amy Clarke (Mines)

**Project Duration**  
PhD: September 2019 to March 2023

- **Problem:** Aerospace components are difficult to produce conventionally, but the effects of additive manufacturing (AM) on microstructural evolution are not understood enough to replace conventional manufacturing.
- **Objective:** Develop an understanding of solidification behavior in model alloys under AM conditions by *in-situ* characterization.
- **Benefit:** Microstructural control for additive manufacturing of aerospace components.

- Recent Progress**
- Top-down imaging of as-solidified melt pools
  - Qualifying exam

Metrics		
Description	% Complete	Status
1. Literature review	25%	●
2. Analysis of APS beam line data	40%	●
3. Analysis of Dynamic Transmission Electron Microscopy (DTEM) of rapid solidification	0%	●
4. Simulation of experimental conditions	15%	●
5. Complementary <i>ex-situ</i> characterization	15%	●

# Industrial Relevance

Enhanced microstructure/properties:

- Control during AM

Applications:

- Turbine components
- Aerospace components

Benefits:

- Faster production time
- Greater geometric flexibility
- Reduced processing cost
- Enhanced performance
- Low volume/lean manufacturing and customization



Left photo courtesy of  
Wikimedia, right photo  
courtesy of NASA

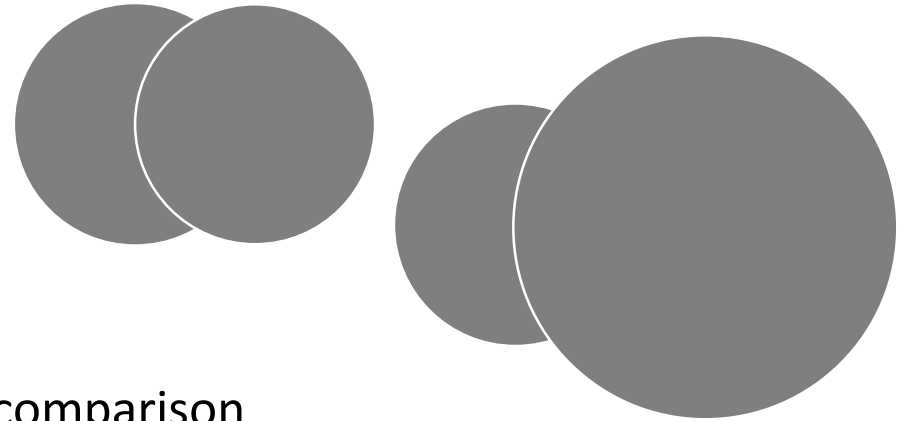
# Model Alloys of Choice



- Two alloy systems:
  - Ni-Al-Mo
  - Al-Ag
- Ni-base system consists of two alloys with same equilibrium  $\gamma'$  volume fraction supplied as single crystals
  - R2: Ni-6.6Al-1.9Mo (at%)
  - R4: Ni-2.8Al-22.2Mo (at%)
- Al-Ag binary system consists of two different fractions of silver
  - Al-10Ag (at%)
  - Al-18Ag (at%)

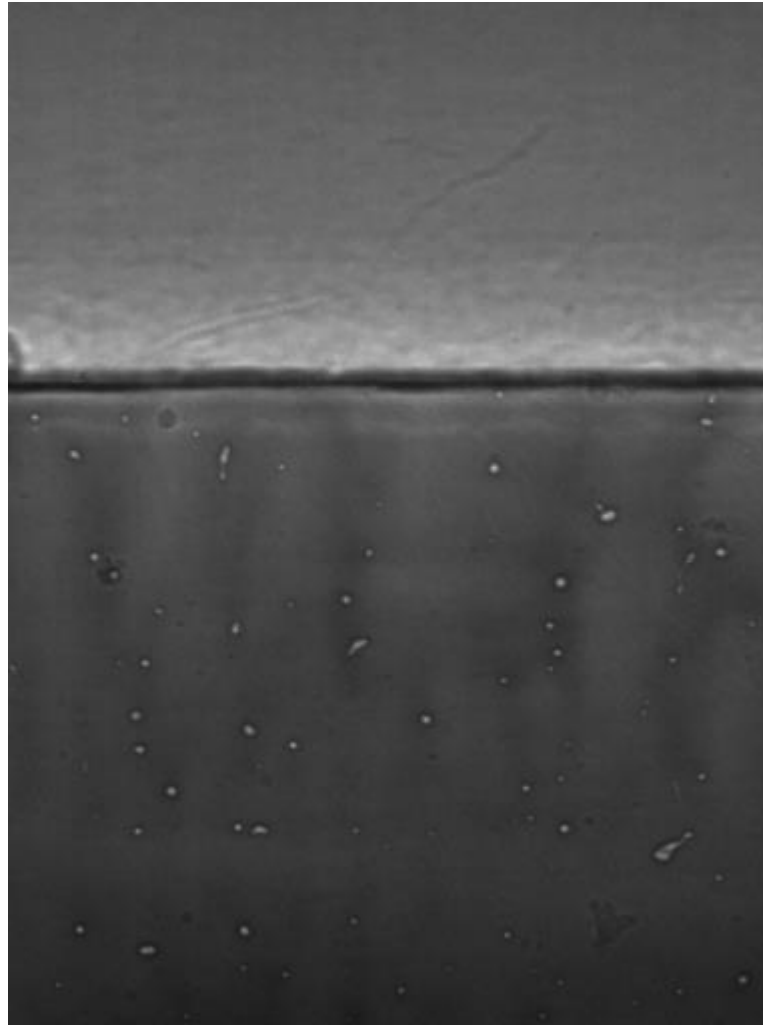
# APS Experiments

- R2 and R4 alloys:
  - Rasters at constant heat input
  - Overlapping spot melts
- Al-Ag system:
  - Rasters and re-rasters for DTEM comparison

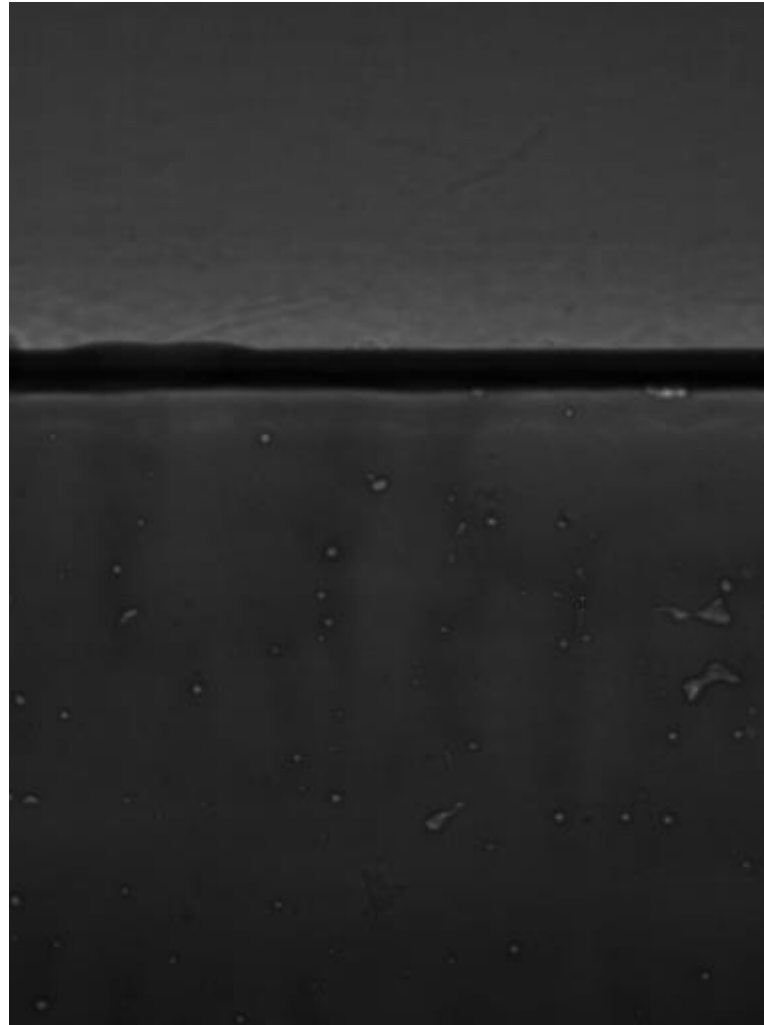


Alloy	Beam power [W]	Raster speed [m/s]	Notes
R2 [110], R2 [111], R4[100], R4 [110]	253.9	1.6	
	139.4	0.5	
	47.8	0.1	
	82.1/82.1	Spot melt	Edge of second pool intersects middle of first
	82.1/253.9	Spot melt	
R2 [110]	517.1	1.6	
	253.9	1	
R4 [100]	517.1	1.6	
	368.3	1.6	
	253.9	Spot melt	
Al-10Ag & Al-18Ag	282.5/282.5	0.1/0.1	Re-rasters with 100% overlap
	368.3/368.3	2/2	
	282.5	0.1	
	368.3	2	

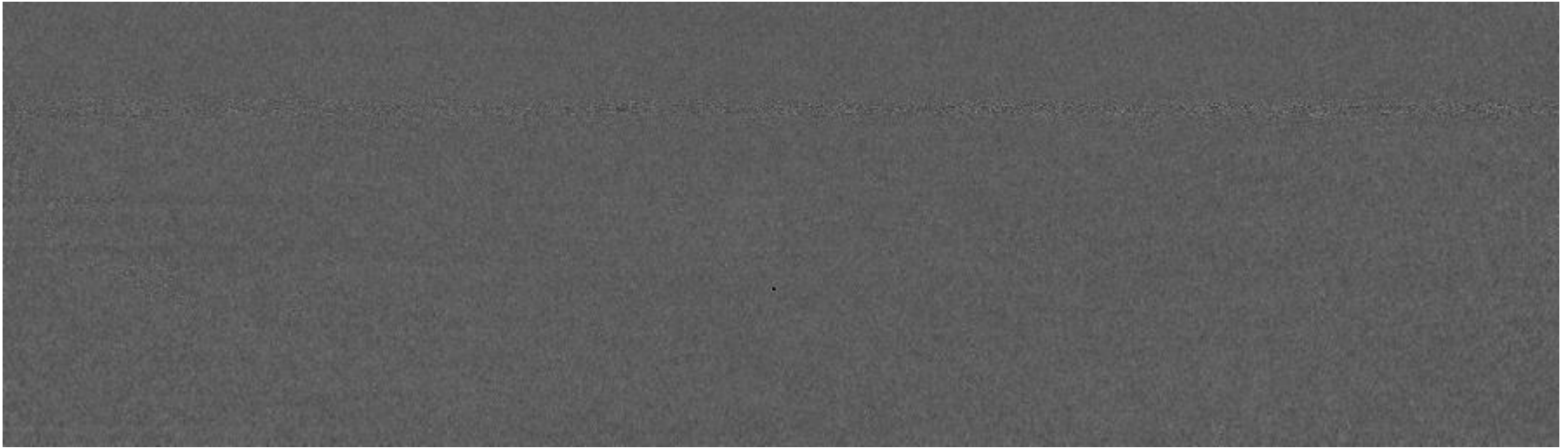
# Spot Melt



# High Power Spot Melt



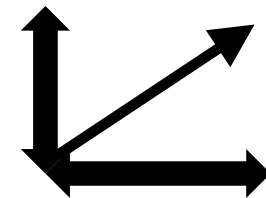
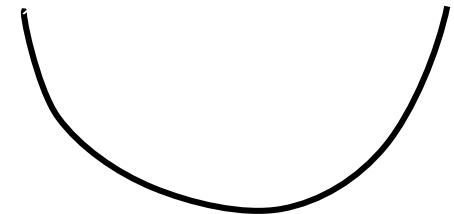
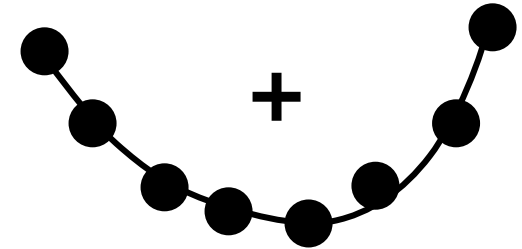
# Raster





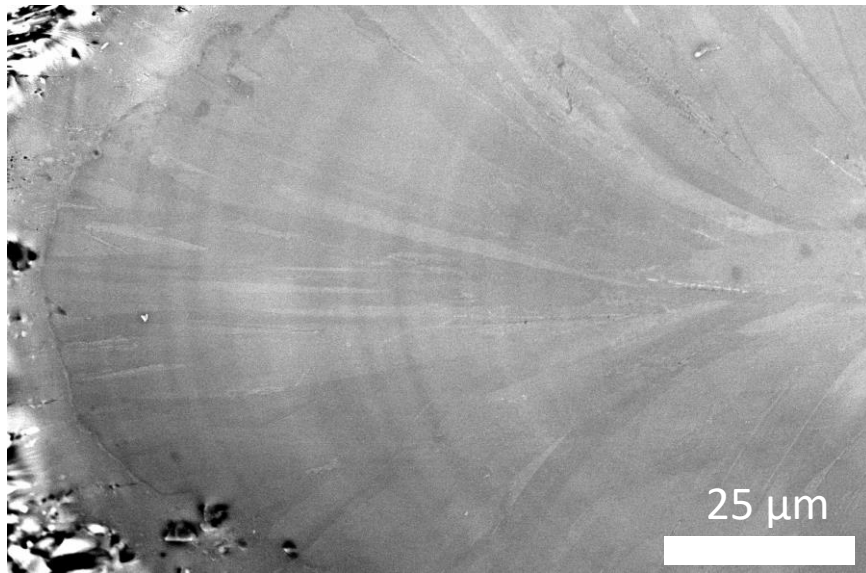
# Recap of Previous Report

- Analysis of solid-liquid interface velocity based on melt pool shape
- Polynomial fit to manually found points
- Analyze change in position as polynomial function moves through a line
- Adjust magnitude to match known raster speed
- If needed for single crystals, compute projection of result onto unit vector of given angle matching dendrite angle

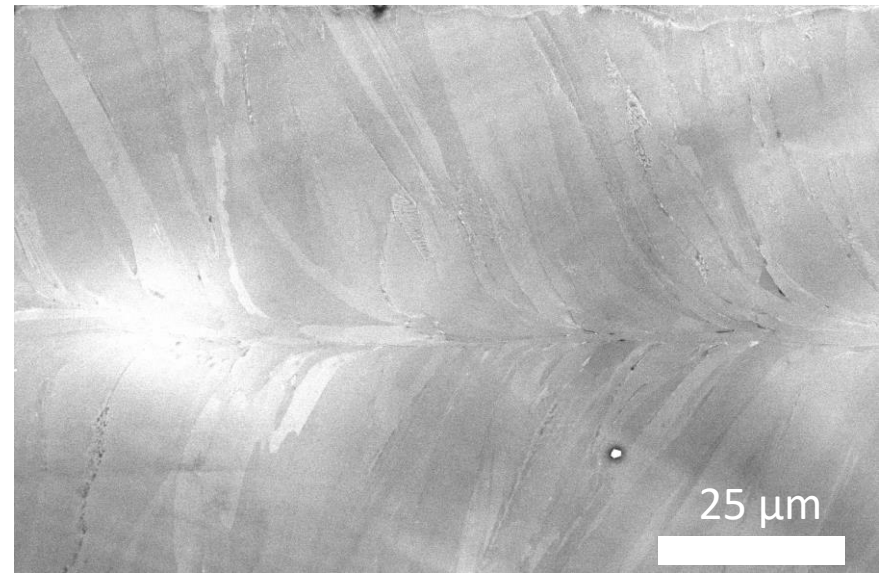


# Conventional Columnar Grains in Al-Ag

- Under some conditions, Al-Ag samples develop long columnar grains following the heat source
- This is commonly observed in welds
- Angle changes as pool achieves steady state, also typical behavior



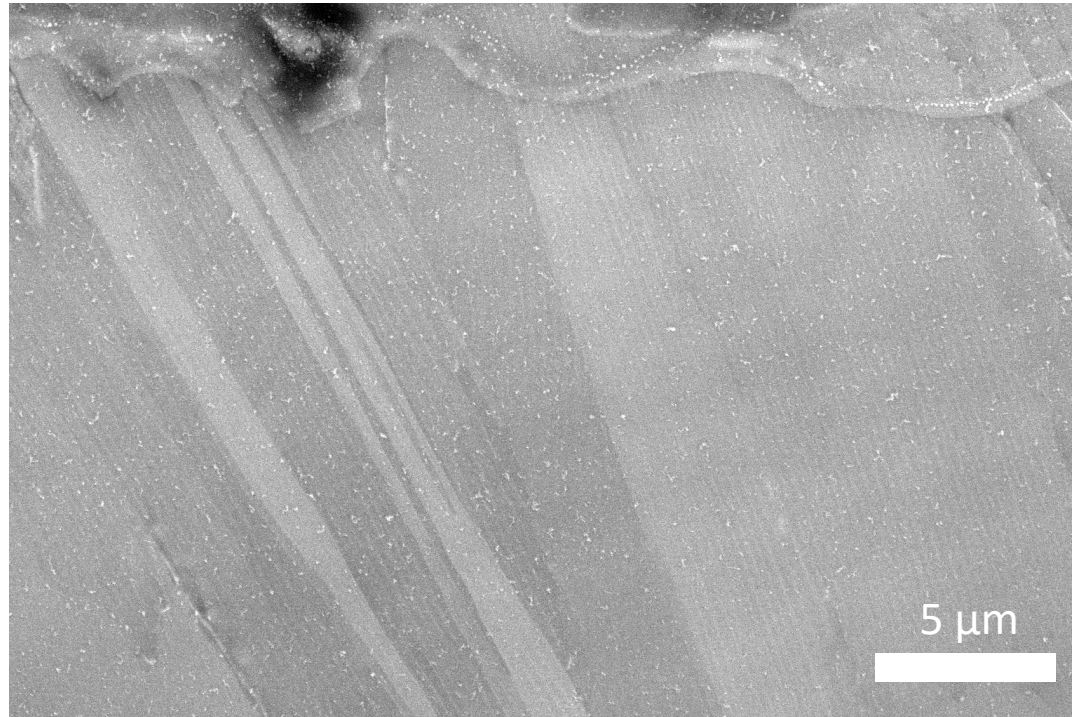
SEM, backscatter



SEM, backscatter

# Silver Partitioning in Columnar Grains

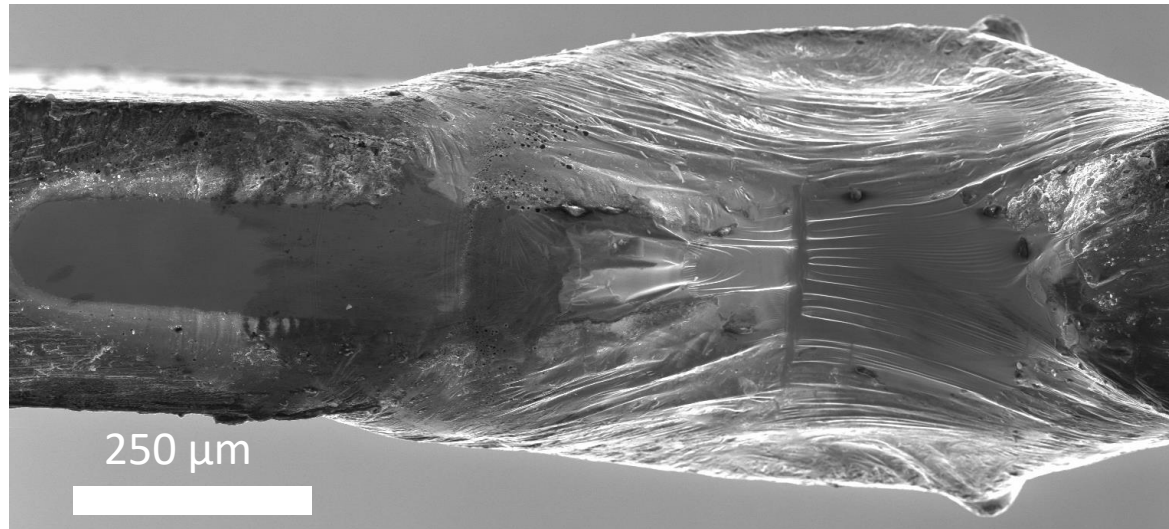
- Composition is not uniform within the columnar grains
- Silver partitions within the grains according to two different patterns seen thus far
- Straight lines and wavy lines of Ag partitioning



SEM, backscatter

# Al-10Ag Re-Raster

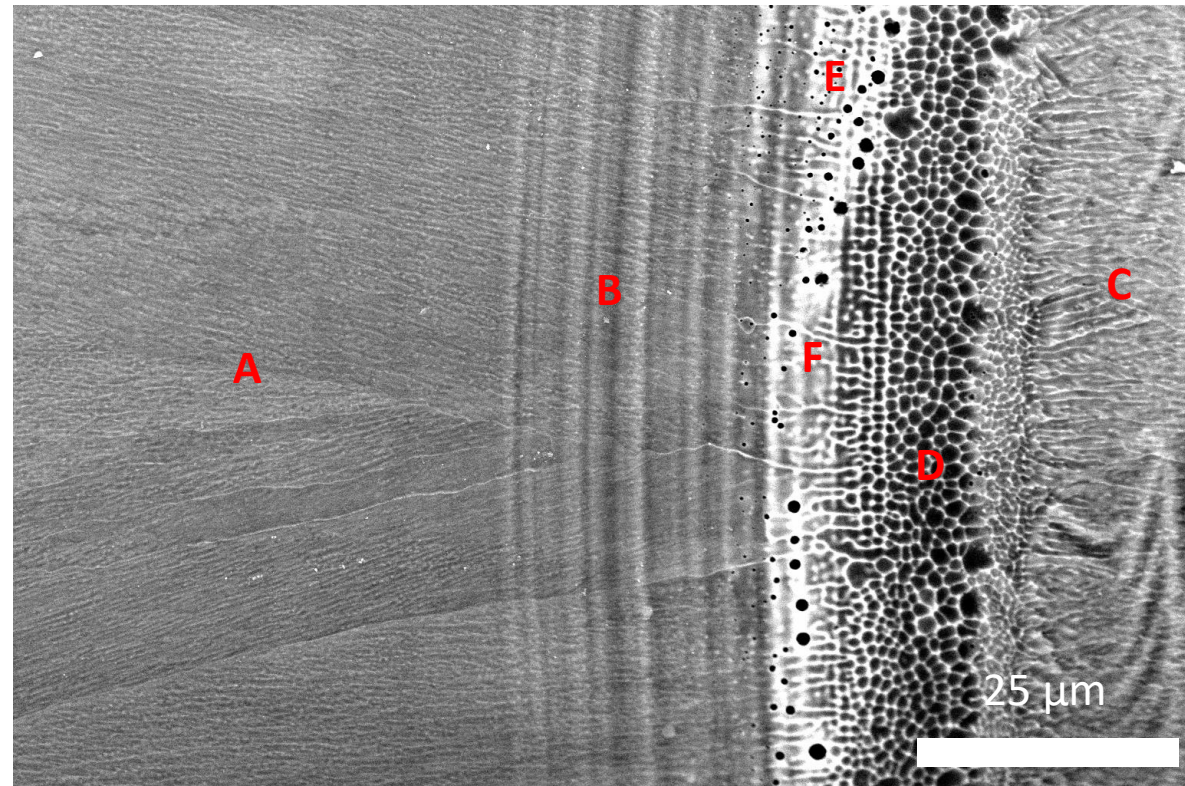
- Re-rastered sample where both rasters transition from conduction mode to keyhole mode due to transient heat flow
- Region where solid-liquid interface was during transition displays several different microstructures



SEM, secondary electron

# Microstructures in Transition Region

- A – Columnar grains
- B – Ripples from fluid flow
- C – Columnar grains with increased oxidation
- D – Cellular
- E – Pores
- F – Shown next slide, only seen here (so far)



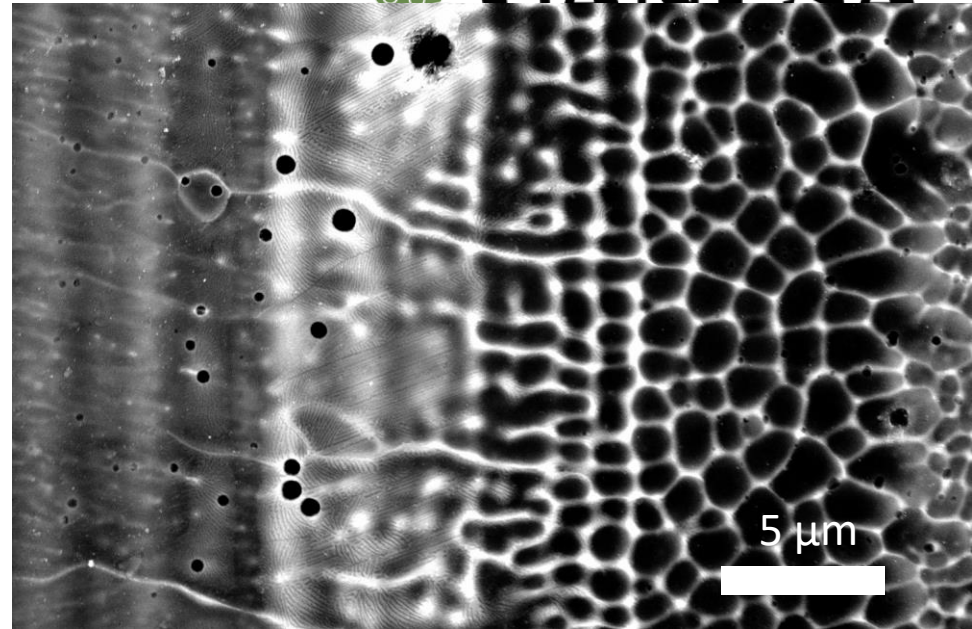
SEM, backscatter

# Unique Microstructure in Al-10Ag Re-Raster

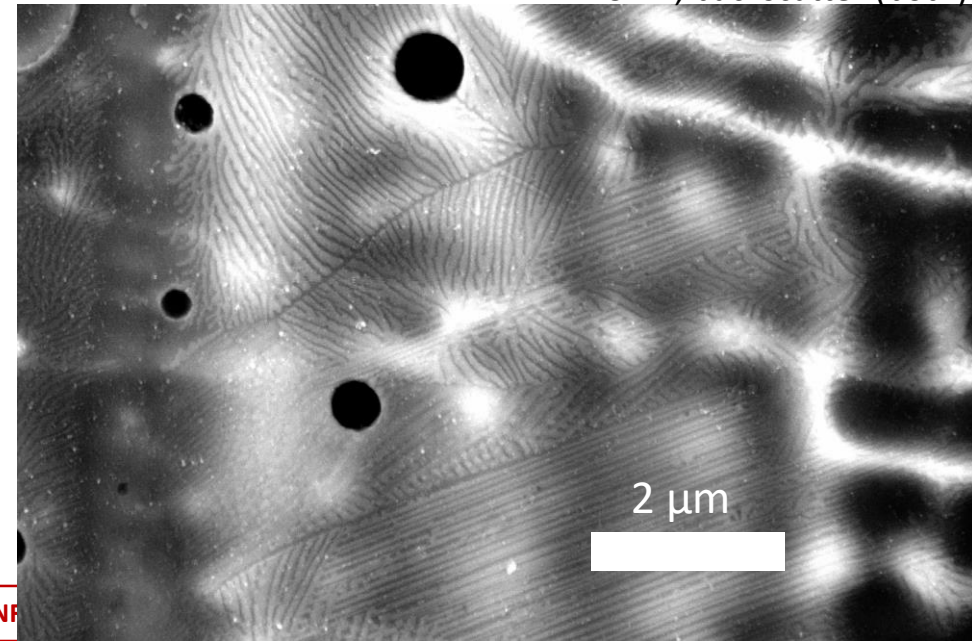


CANESA

- Partitioning of Ag on two length scales simultaneously
- Ag rich lines continuous between columnar region, through this region, into cellular region
- Fine alternating layers of Ag and Al rich phases
- Seaweed like structures also apparent
- Similar features seen in DTEM



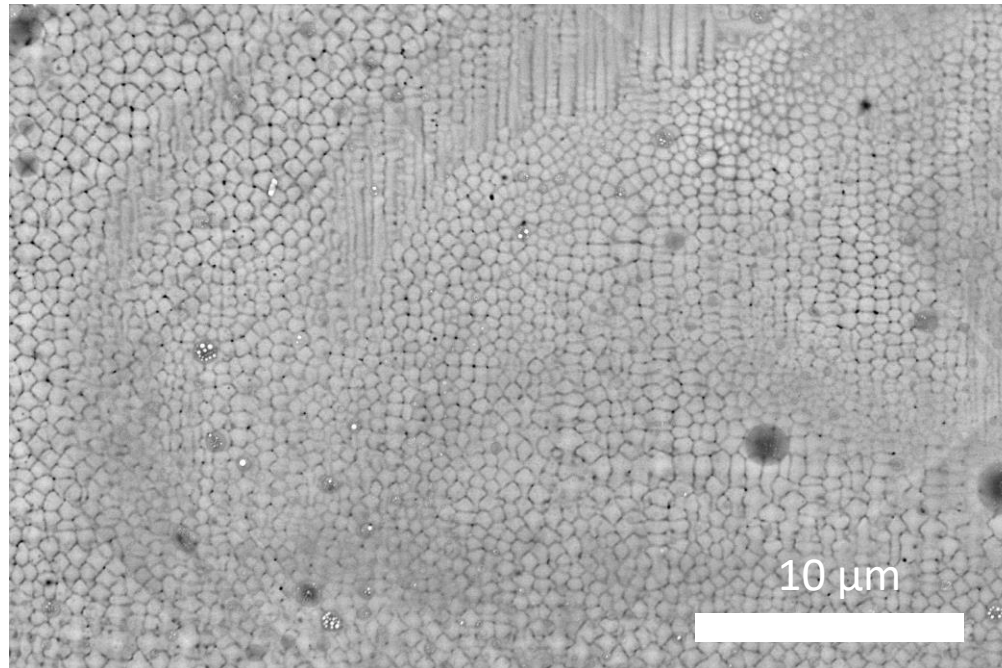
SEM, backscatter (both)



2 μm

# Dendritic Solidification in R4 <100> Oriented

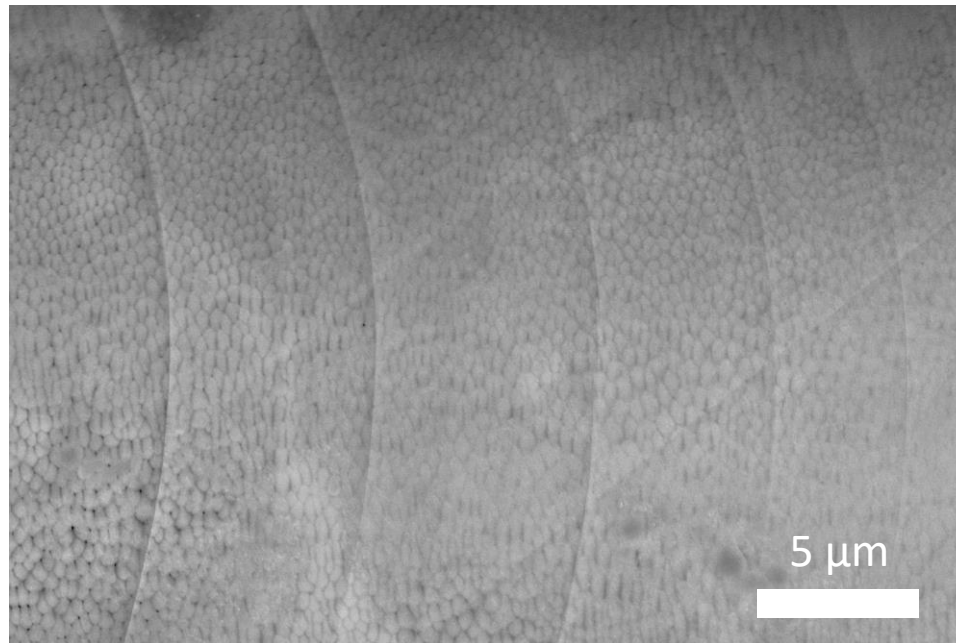
- The microstructure of many R4 melts can be readily identified as dendritic in nature
- Growth following crystallographic directions rather than maximum thermal gradient



SEM, backscatter

# Some Regions Cannot be Confirmed Dendritic

- Top-down imaging alone cannot confirm if this is dendritic or cellular
- Also a rare case of a region in R2 where the solidification structure can be readily distinguished

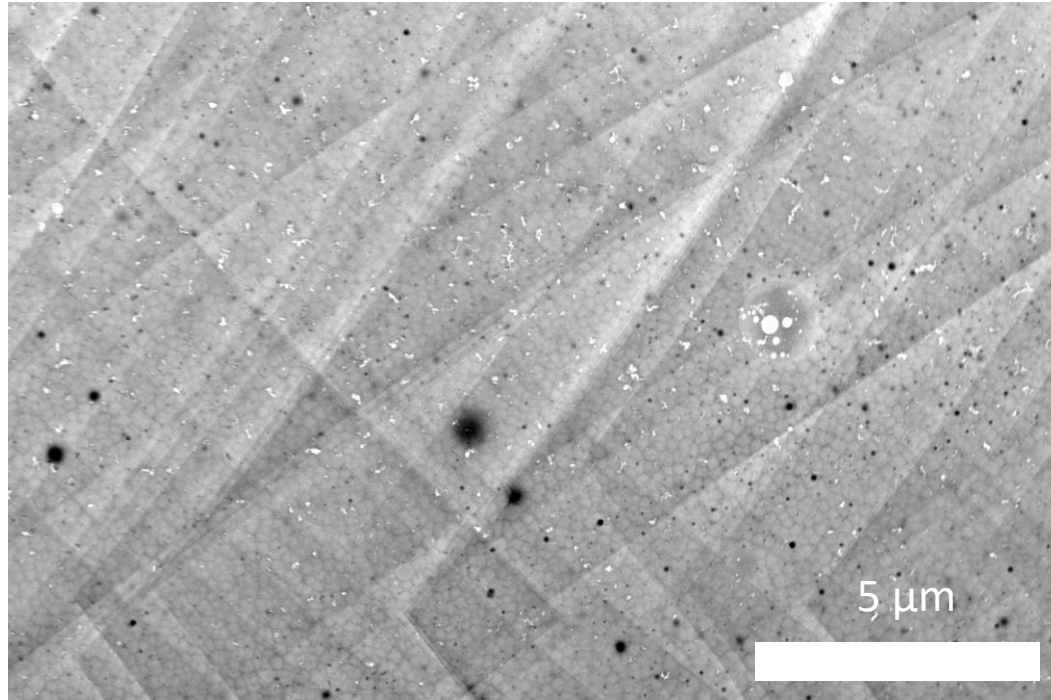


SEM, backscatter



# Slip Lines may Confirm “Single Crystal” Dendritic Structure

- Same ambiguous dendritic or cellular structure is present
- Lines are present in a pattern unlikely for fluid flow to have created, may be caused by dislocation movement



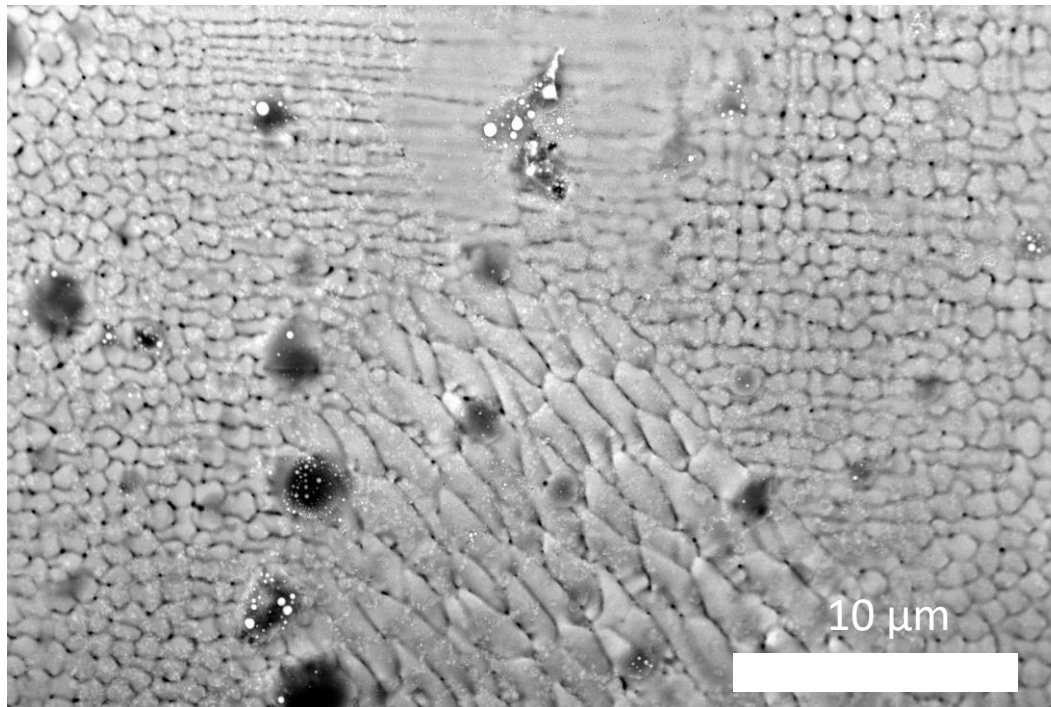
SEM, backscatter

# New Grain Orientations Nucleate in Spot Melts and Raster Starts



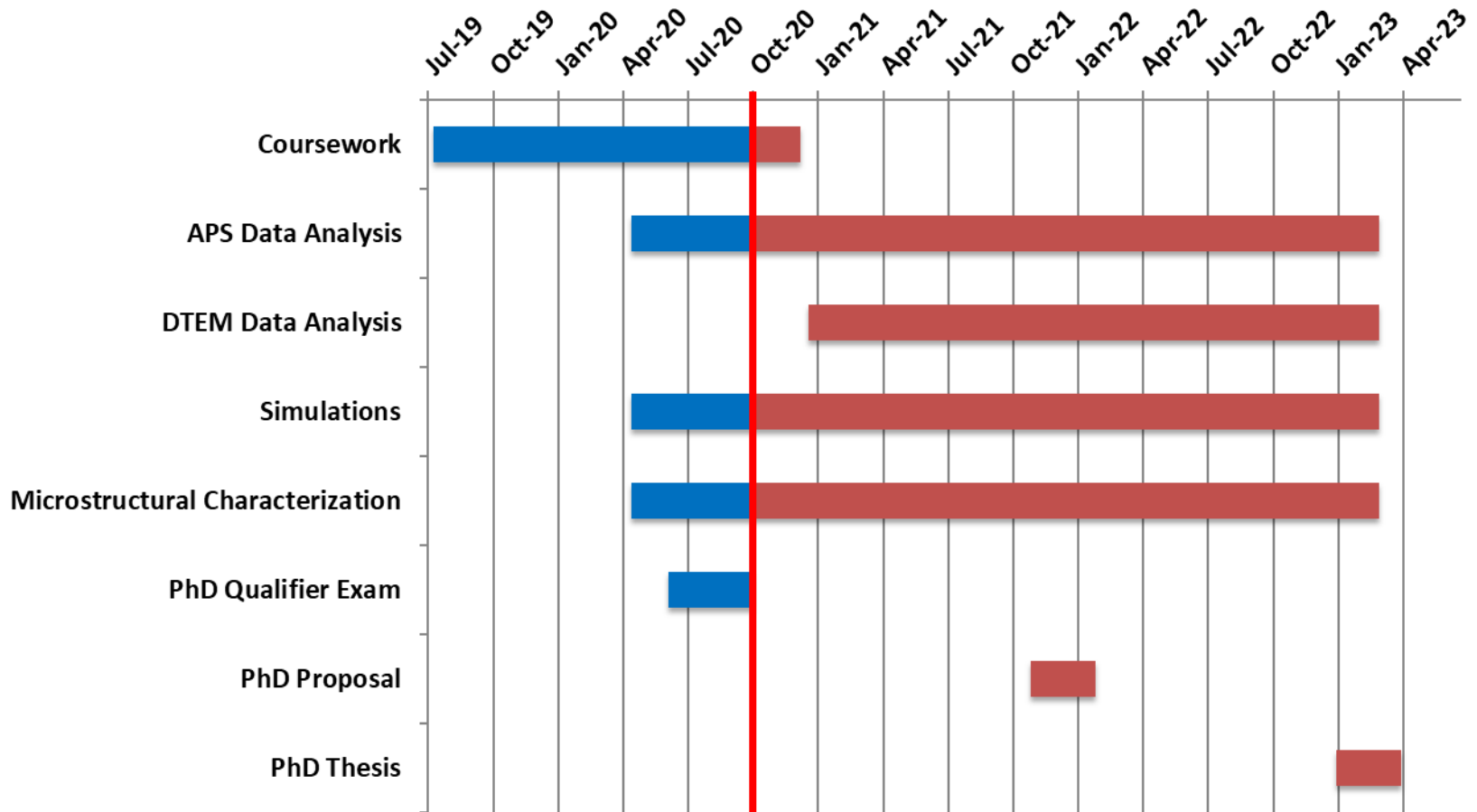
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CENTER FOR ADVANCED  
NON-FERROUS STRUCTURAL ALLOYS

- New grain orientations only seen in these areas
- No new grain nucleation seen in steady state of rasters *so far*, not proof it has not occurred



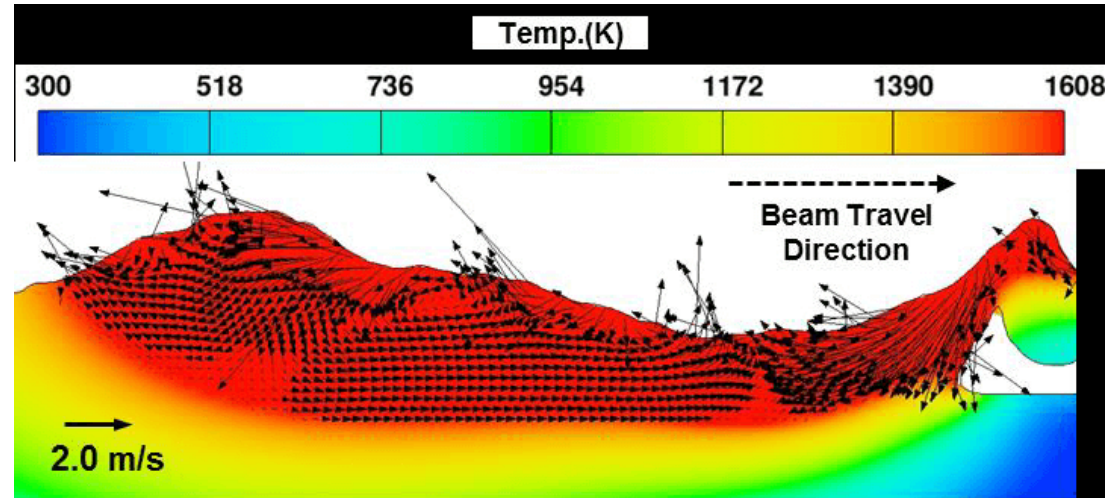
SEM, backscatter

# Progress



# Challenges & Opportunities

- Microstructure analysis
  - Determining 3D structure, needing view from other angles
  - Identifying structure seen in Al-Ag
- FLOW-3D simulations
  - Learning software



Thank you!  
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