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

Summer 2020 Videoconference

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Other Participants: Jonah Klemm-Toole (Mines)

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

- Analysis of *in-situ* radiography from APS on Ni and Al base alloys
- Top-down imaging of as-solidified melt pools

Metrics

Description	% Complete	Status
1. Literature review	15%	●
2. Analysis of APS beam line data	30%	●
3. Analysis of Dynamic Transmission Electron Microscopy (DTEM) of rapid solidification	0%	●
4. Simulation of experimental conditions	15%	●
5. Complementary *ex-situ* microstructural characterization	5%	●

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

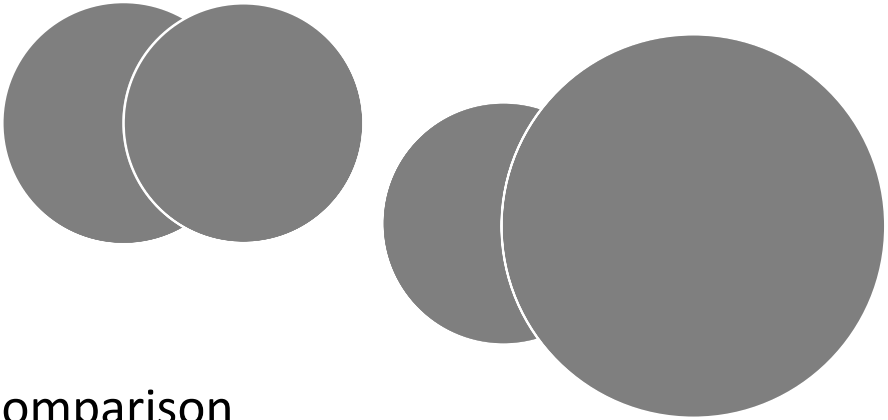
Left photo courtesy of wikimedia, right photo courtesy of NASA

Model Alloys of Choice

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

APS Experiments

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



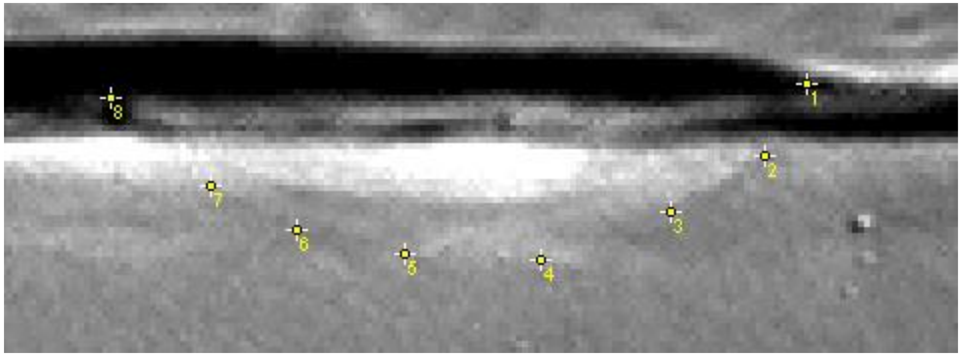
Alloy	Beam power [W]	Raster speed [m/s]	Notes
R2 [110], R2 [111], R4[100], R4 [110]	253.9	1.6	
	139.4	0.5	
	47.8	0.1	
	82.1/82.1	Spot melt	Edge of second pool intersects middle of first
	82.1/253.9	Spot melt	
R2 [110]	517.1	1.6	
	253.9	1	
R4 [100]	517.1	1.6	
	368.3	1.6	
	253.9	Spot melt	
Al-10Ag & Al-18Ag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

Recap of Previous Report

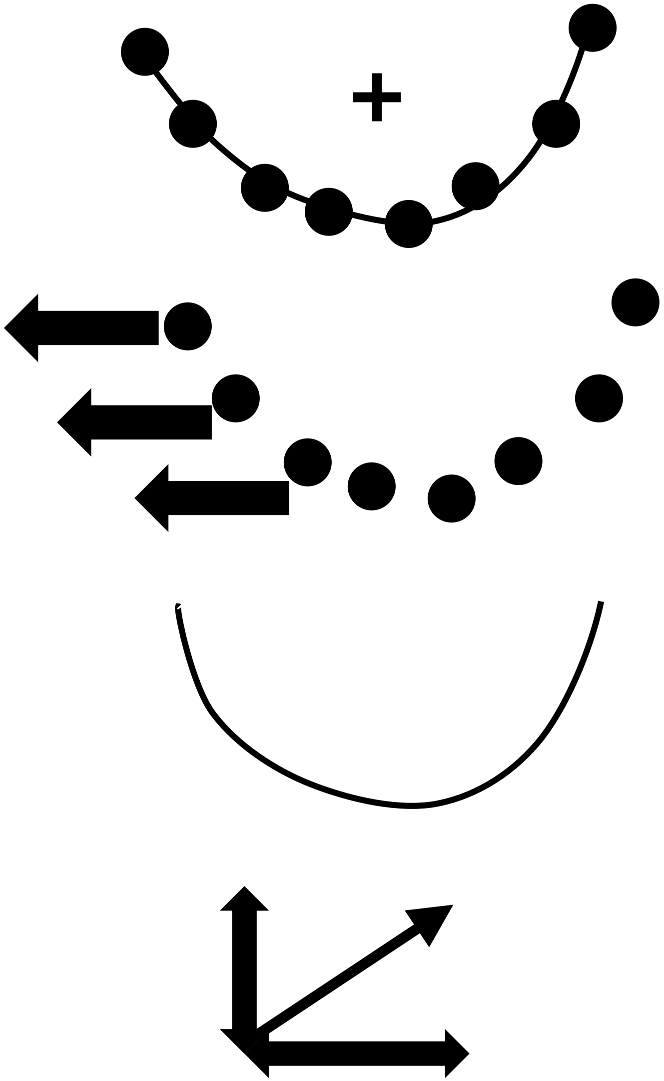
- Wrote a MATLAB program to perform Rosenthal-type simulations of conditions from APS
- Molten pool shape results differed between simulations and real melt pools
  - R2 and R4 matched for intermediate raster velocity, mismatched for low and high velocity
  - Al-Ag mismatched for most conditions
  - Non-steady-state behavior, especially keyholing, caused most extreme mismatches

Point Clicking for Interface Tracking

- Solid-liquid interface is tracked manually by clicking points along interface at different frames
  - Program developed by Gus Becker (thank you Gus!)
- Program outputs .csv file with x-y coordinates and frame numbers
- Conversion to physical distances and actual time stamps only requires a short unit conversion
- Finding the solidification velocity itself requires more data analysis

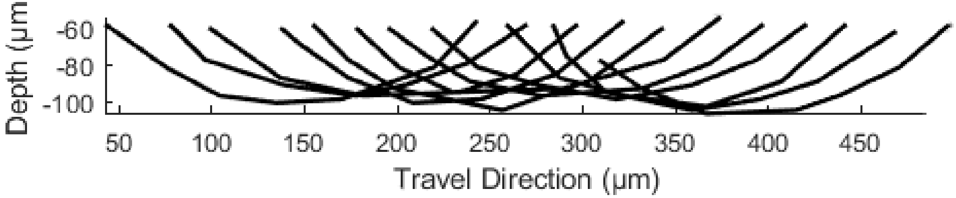


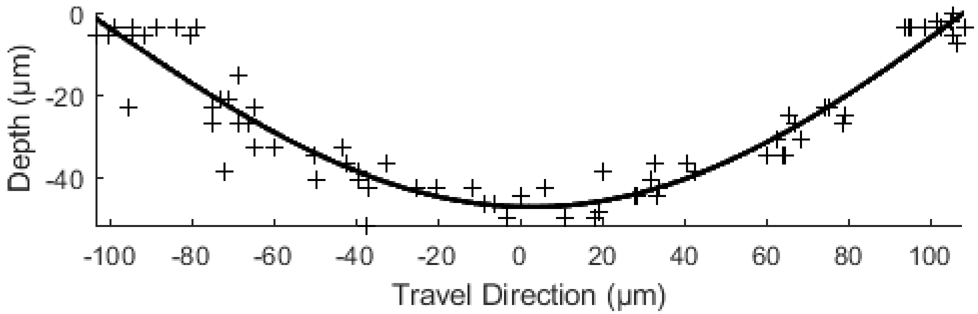
Analysis of Point Clicking for Rasters

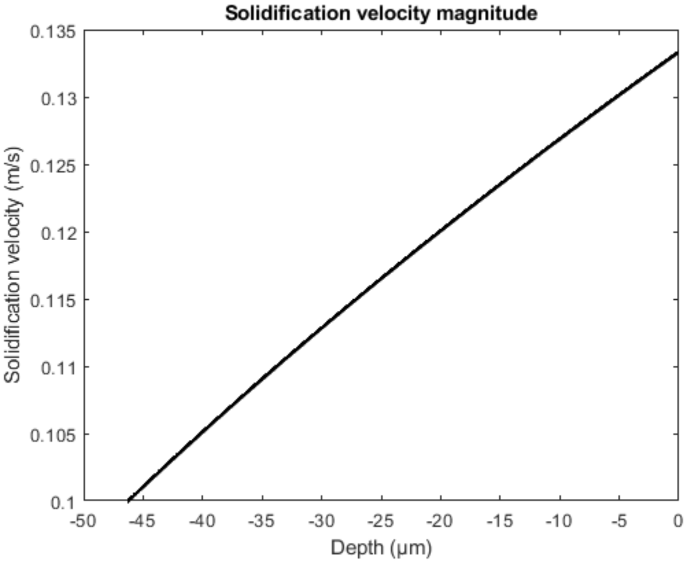


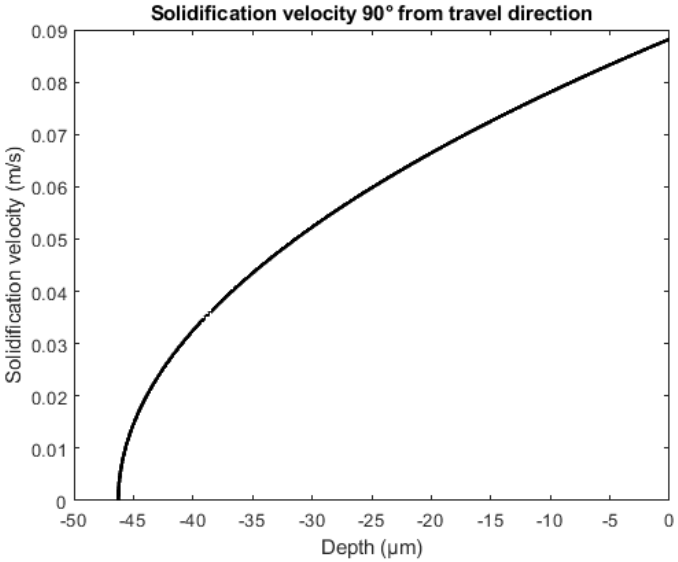
- Find a polynomial fit to each set of points, and use it to compute the centroid
- Translate all points so that their centroids are about x=o
- Find a new fit including every data point
- Extract vectors for x and y points of polynomial fit to the left of the absolute minimum along depth
- Find the change in x and y between each set of points and adjust vector magnitude so change in x is equal to the raster speed
- If needed for single crystals, compute projection of result onto unit vector of given angle

Example Results



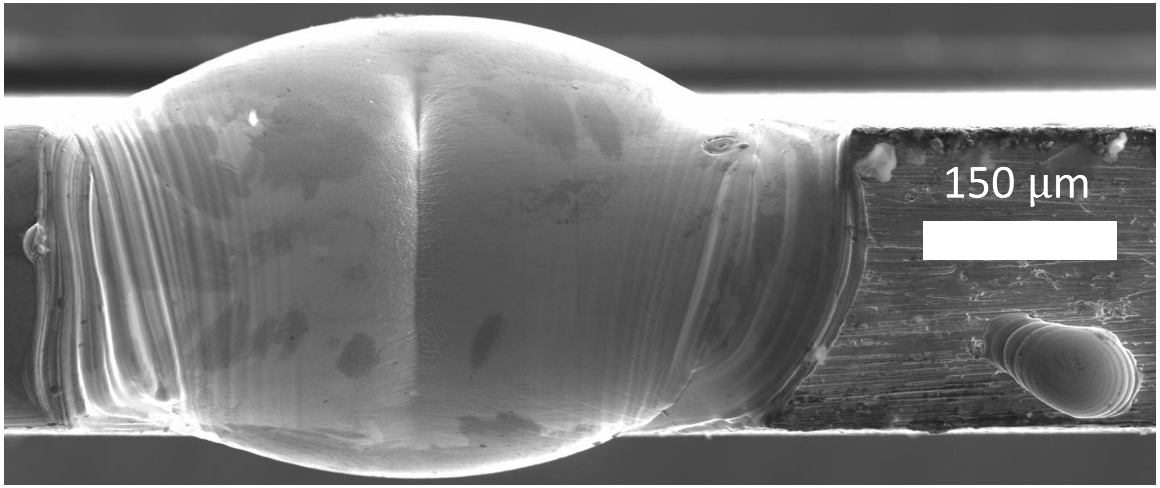


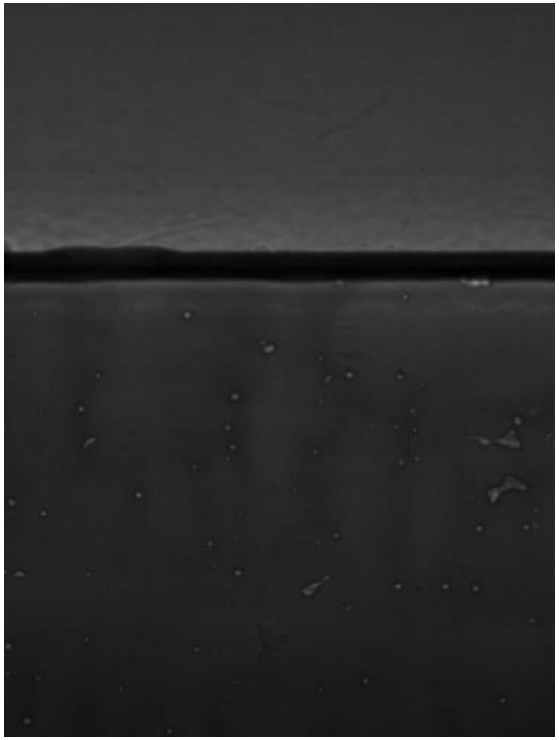




Top Down Imaging

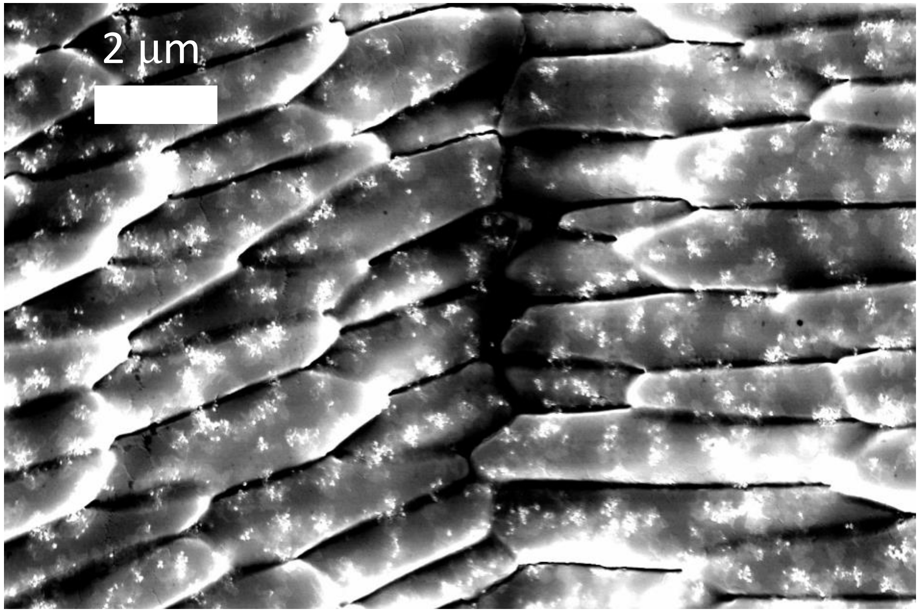
- Beginning of correlation between microstructural features and *in-situ* radiography
- For example, finding spatter that landed on the sample surface during keyholing

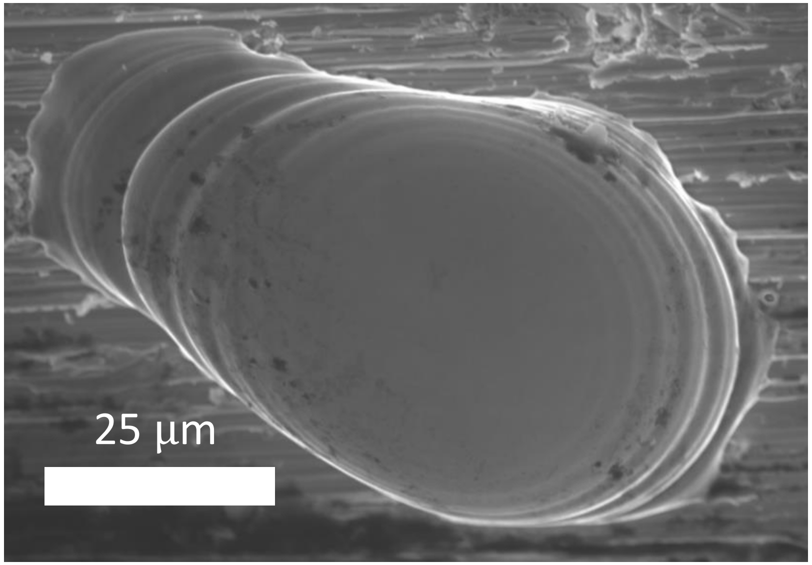




Spatter Solidifies under Uncontrolled Conditions

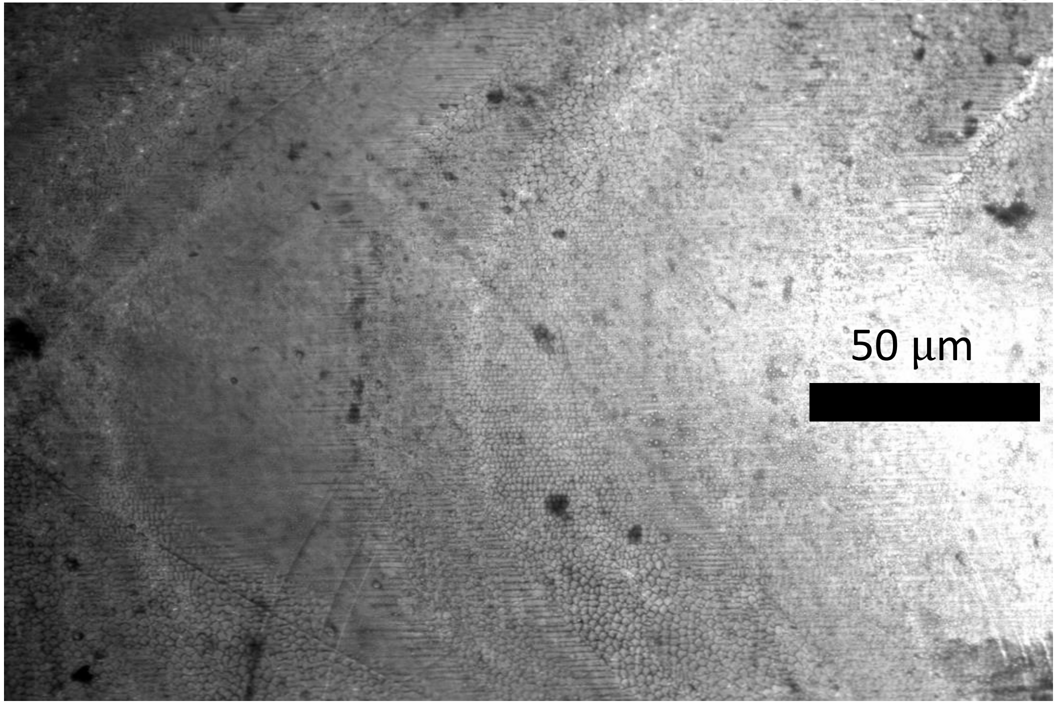
- Dendrites indicate single crystal / highly textured material near centerline
- No such indication within spatter



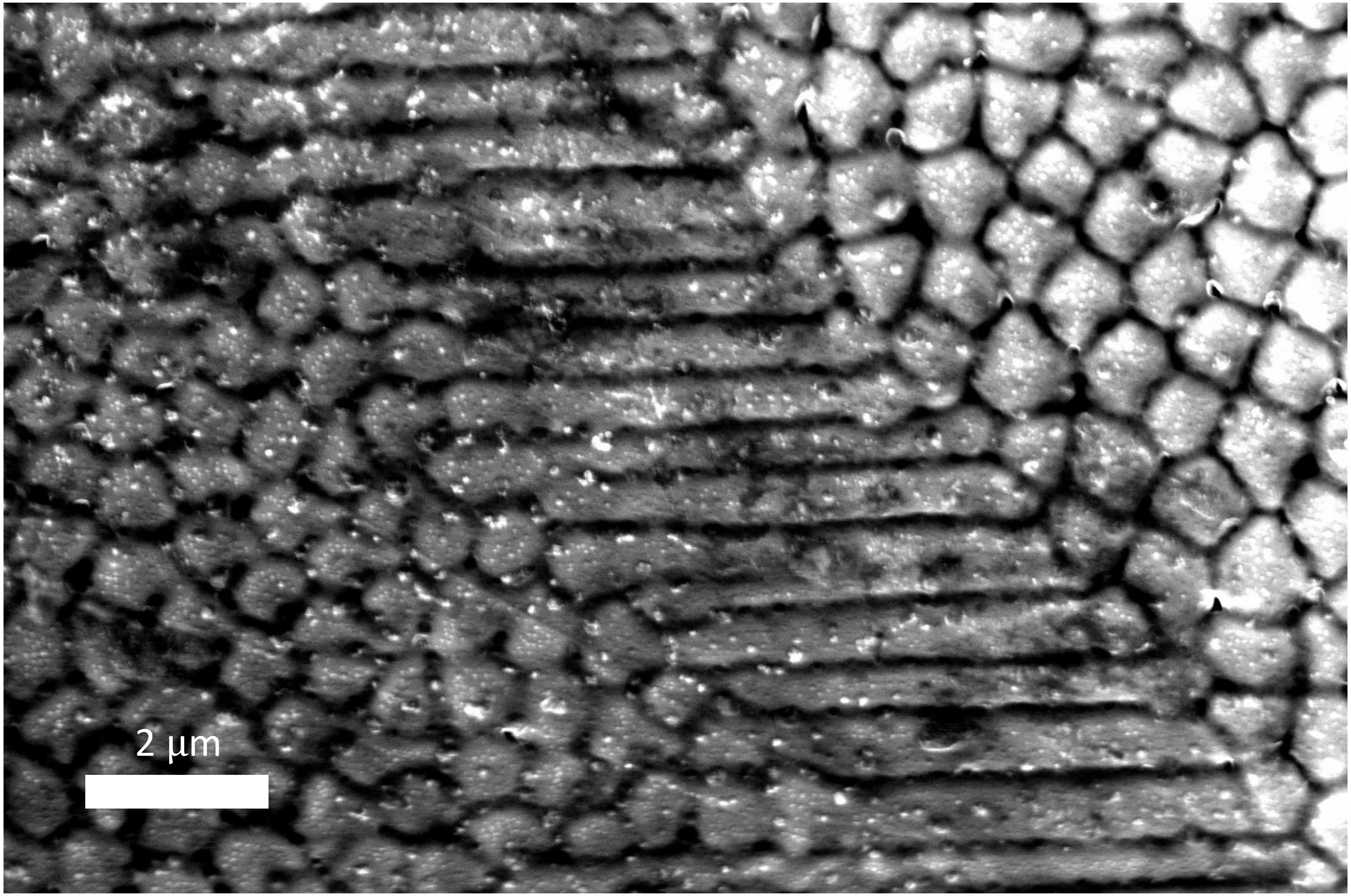


Dendrites Switching Growth Direction during Rasters

- Sample has a {100} orientation with respect to the travel direction
- Remaining a ‘single crystal’ requires dendrite growth along <100>, but multiple growth directions in <100> can be active

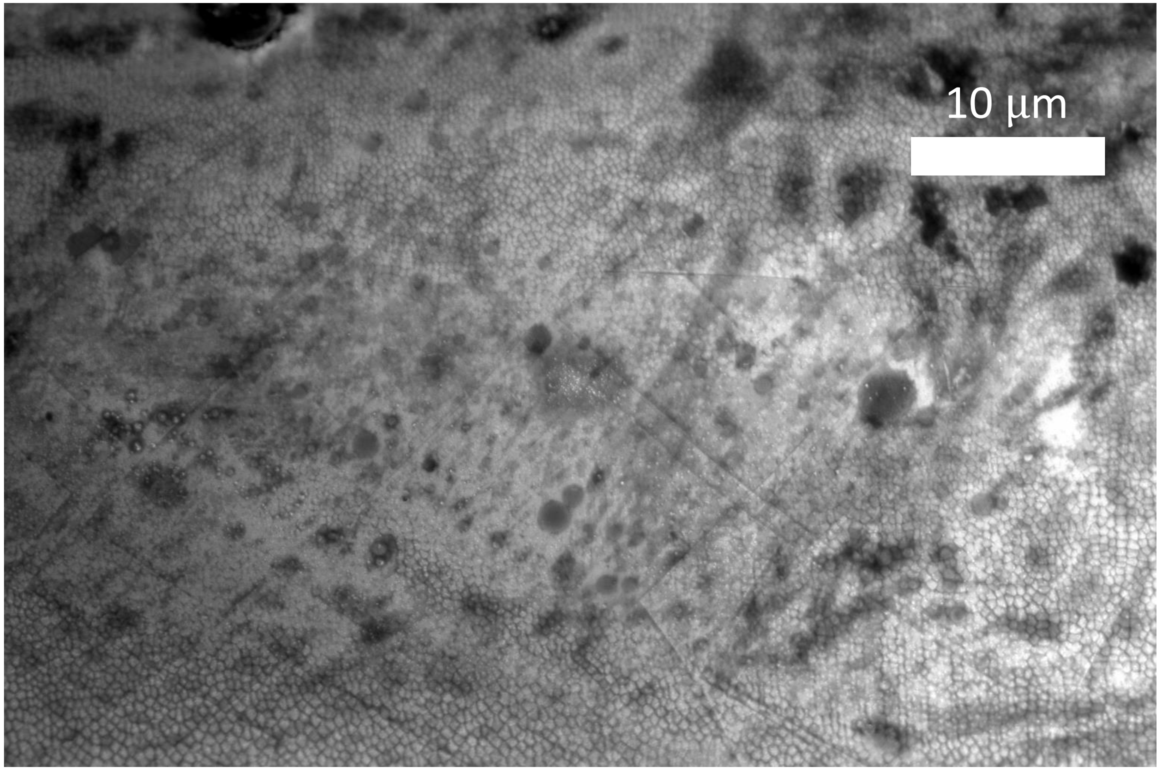


Same Dendrites at Higher Magnification

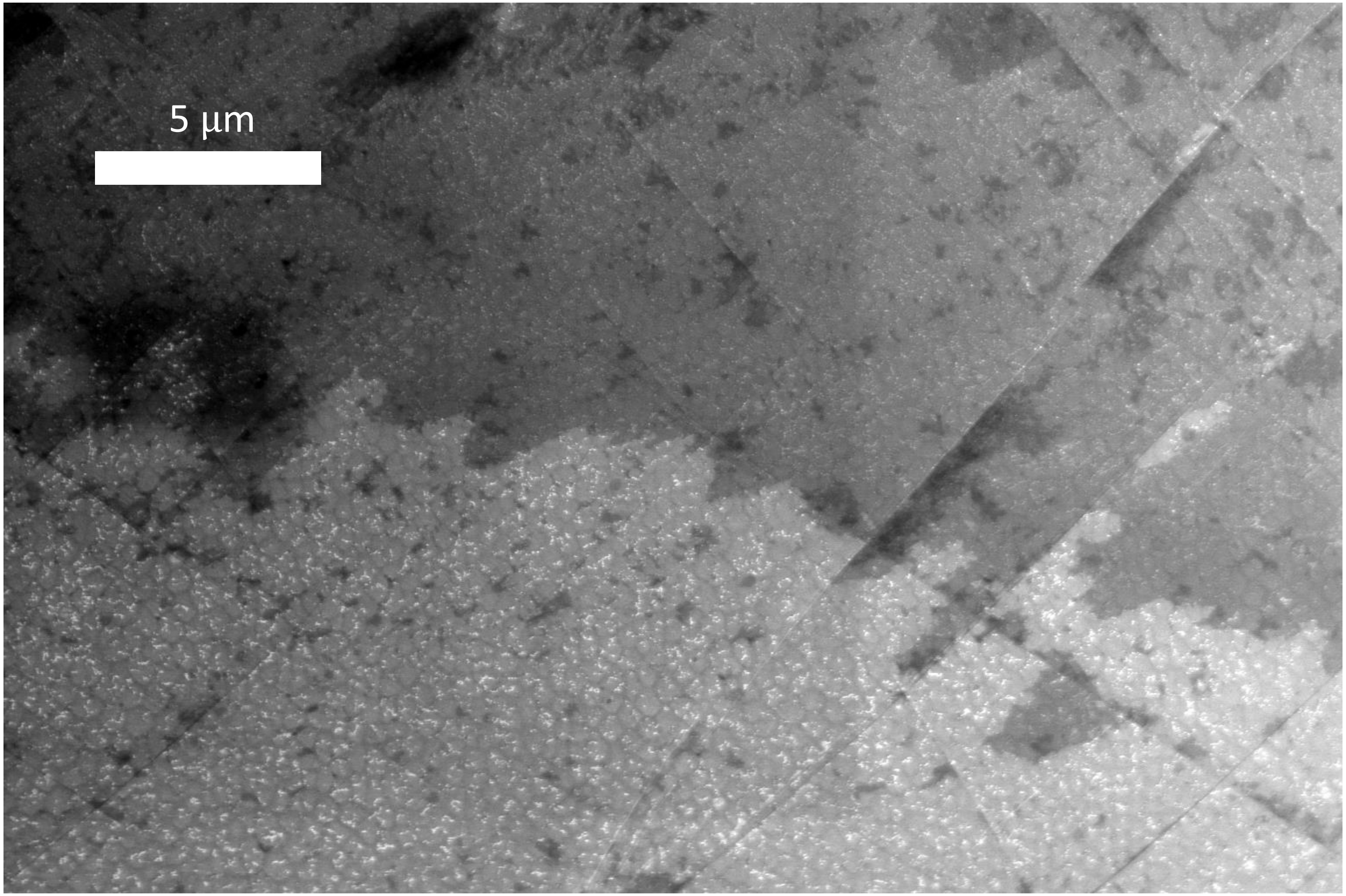


High Speed Raster

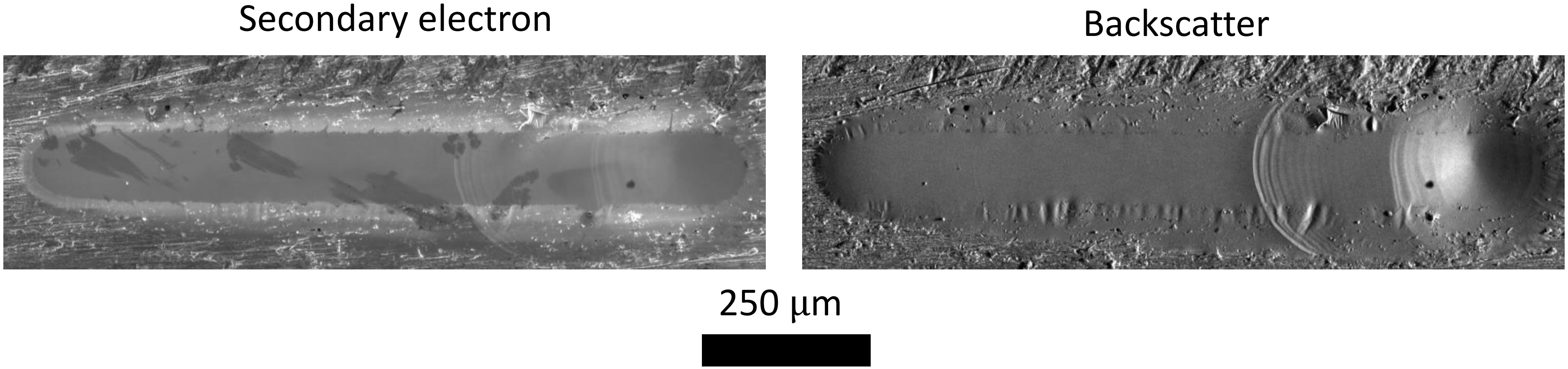
- Still {100} orientation but dendrite orientation remains consistent

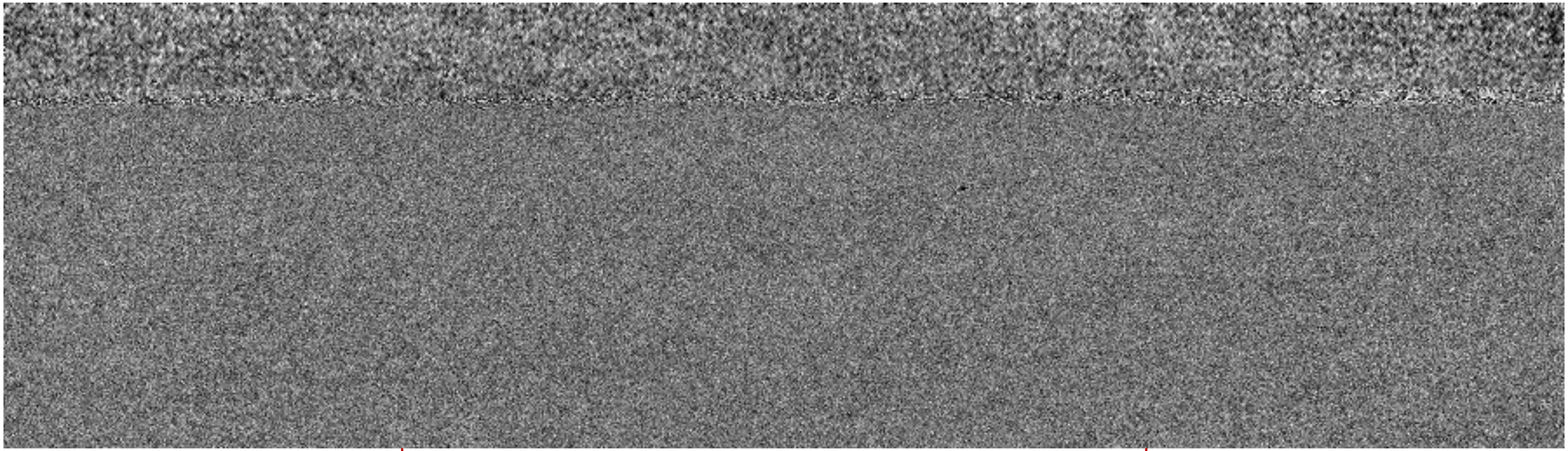


Dendrite Growth Direction Consistent for Stray Grains at High Raster Speed



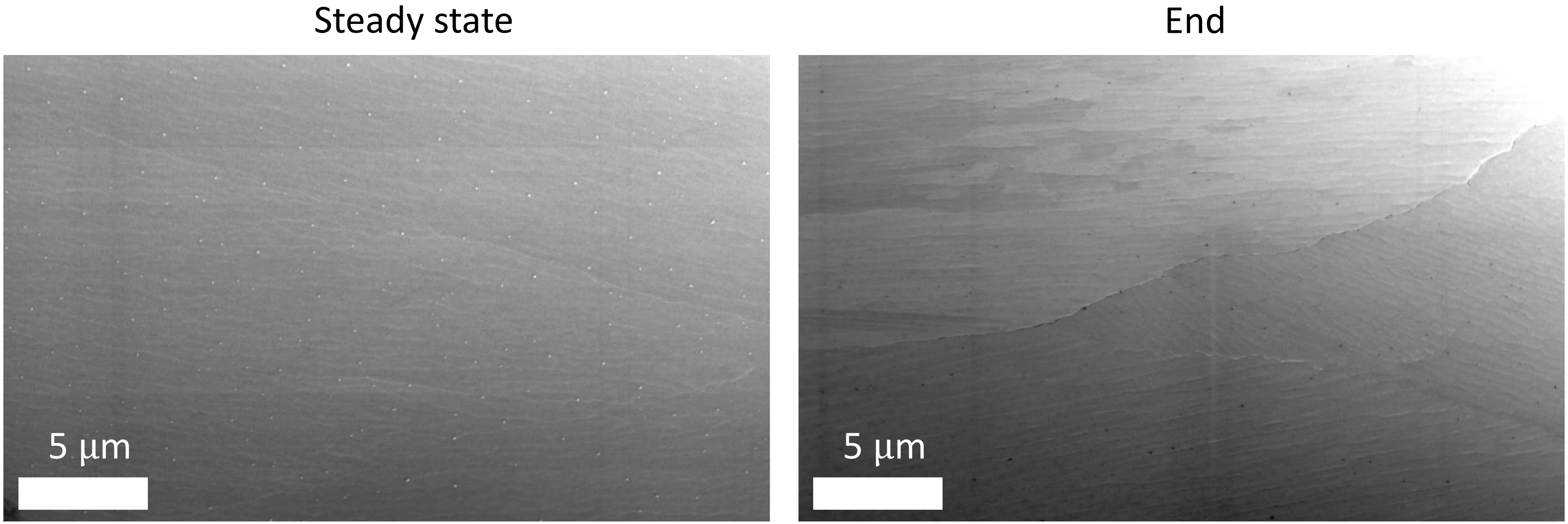
Slow Raster in Al – Ag



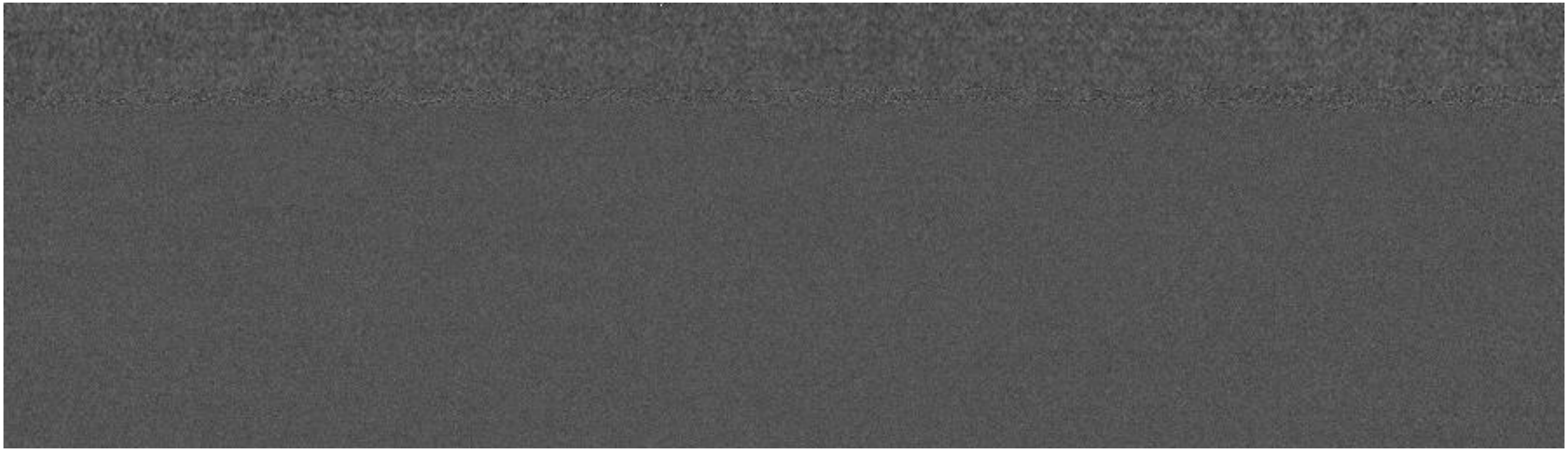


Microstructure Differs between Steady State and End of Raster

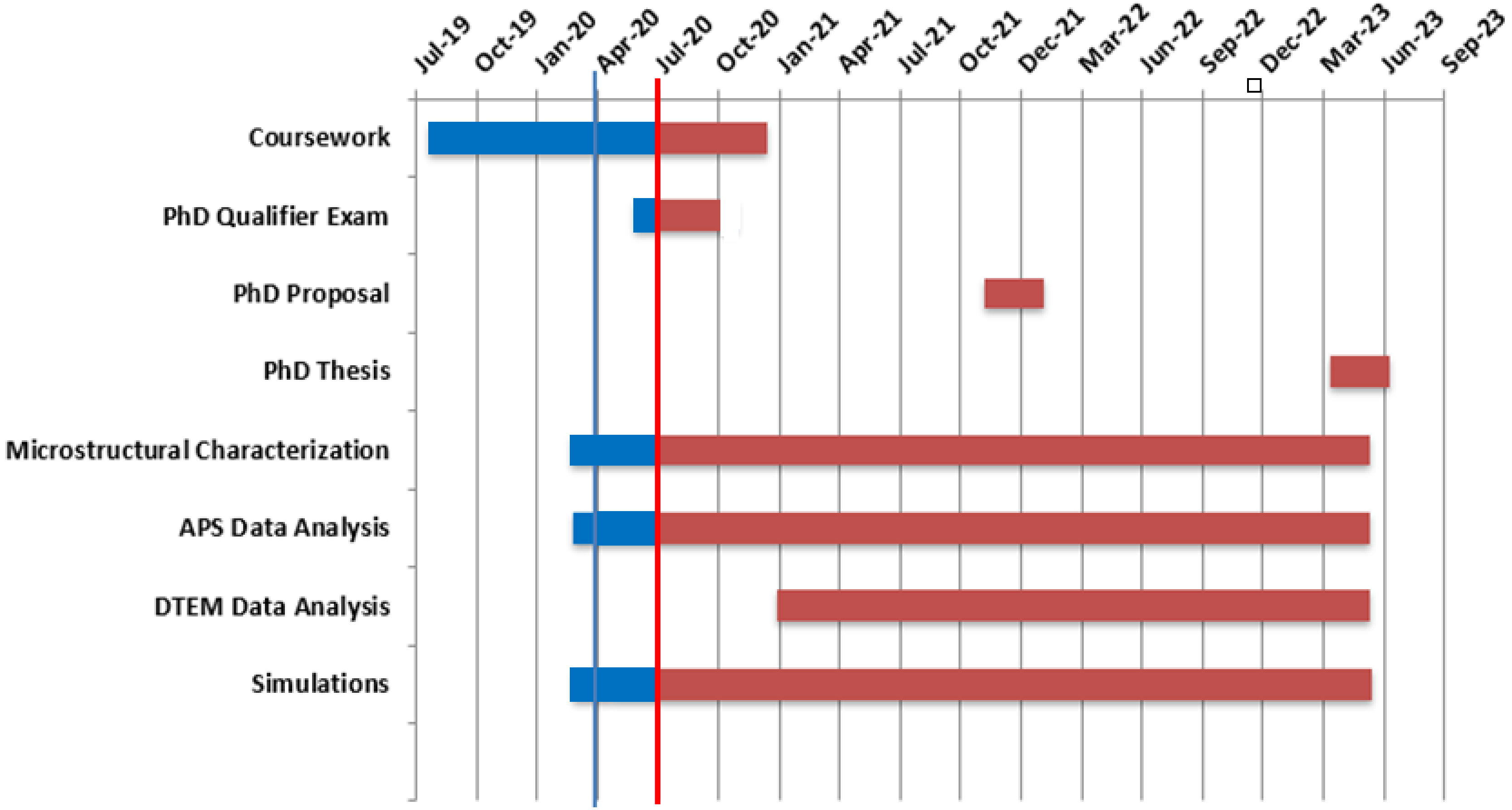
- Appears more seaweed like in steady state regime and columnar towards the end
- Microstructure at beginning difficult to see due to oxide layer



Fast Raster in Al-Ag



Progress


Jul-19
Oct-19
Jan-20
Apr-20
Jul-20
Oct-20
Jan-21
Apr-21
Jul-21
Oct-21
Dec-21
Mar-22
Jun-22
Sep-22
Dec-22
Mar-23
Jun-23
Sep-23
Coursework
PhD Qualifier Exam
PhD Proposal
PhD Thesis
Microstructural Characterization
APS Data Analysis
DTEM Data Analysis
Simulations

Challenges & Opportunities

- Analysis of spot melt velocities
  - Extension of normal lines
  - Intercepts along a line at a given angle
- Imaging Al – Ag with oxide layer present
  - Cannot see microstructure underneath oxide layer in some areas
  - Might need a method to remove oxide without affecting metal
- FLOW-3D simulations
  - Learning software
- Qualifying exam

Thank you!
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