Project 30-L: Mechanisms of Grain Refinement in Laser Powder Bed Fusion of In-Situ Metal Matrix Composite 6061 Aluminum Alloys

Fall Meetings

October 2021

- Student: Chloe Johnson (Mines)
- Faculty: Dr. Amy Clarke (Mines)
- Industrial Mentors: Clarissa Yablinsky (LANL), John Carpenter (LANL), Jeremy Iten (Elementum 3D)
- Other Participants: Joe McKeown (LLNL), Jonah Klemm-Toole (Mines)

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Project 30: Mechanisms of Grain Refinement in Laser Powder Bed Fusion of In-Situ Metal Matrix Composite 6061 Aluminum Alloys

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- Student: Chloe Johnson (Mines)
- Advisor(s): Amy Clarke (Mines)

Project Duration
PhD: August 2017 to December 2021

- Problem: While in-situ inoculation presents a method to eliminate hot tearing and columnar growth in additive manufacturing (AM) of aluminum alloys, the mechanisms of grain refinement under rapid solidification conditions are not well understood.
- Objective: Understand how solidification conditions and the in-situ inoculation process affect mechanisms controlling grain refinement in inoculated alloys in AM.
- Benefit: Inform alloy design and identify refinement mechanisms for in-situ inoculated alloys used in AM solidification conditions.

Recent Progress

- Identification of particle types forming in samples containing various starting reacting particle content in RAM alloys
- Correlation of particle types, locations, and other microstructural features to refinement mechanisms for various RAM alloys
- Paper and thesis writing in preparation for thesis defense

Metrics

Description	% Complete	Status
1. Literature review	90%	●
2. Investigation of RAM (reactive additive manufacturing) on grain refinement mechanisms	90%	●
3. Correlation of measured and modeled solidification conditions to microstructural features and grain refinement	100%	●
4. Effect of inoculants and unreacted particles on post-processing heat treatment	100%	●

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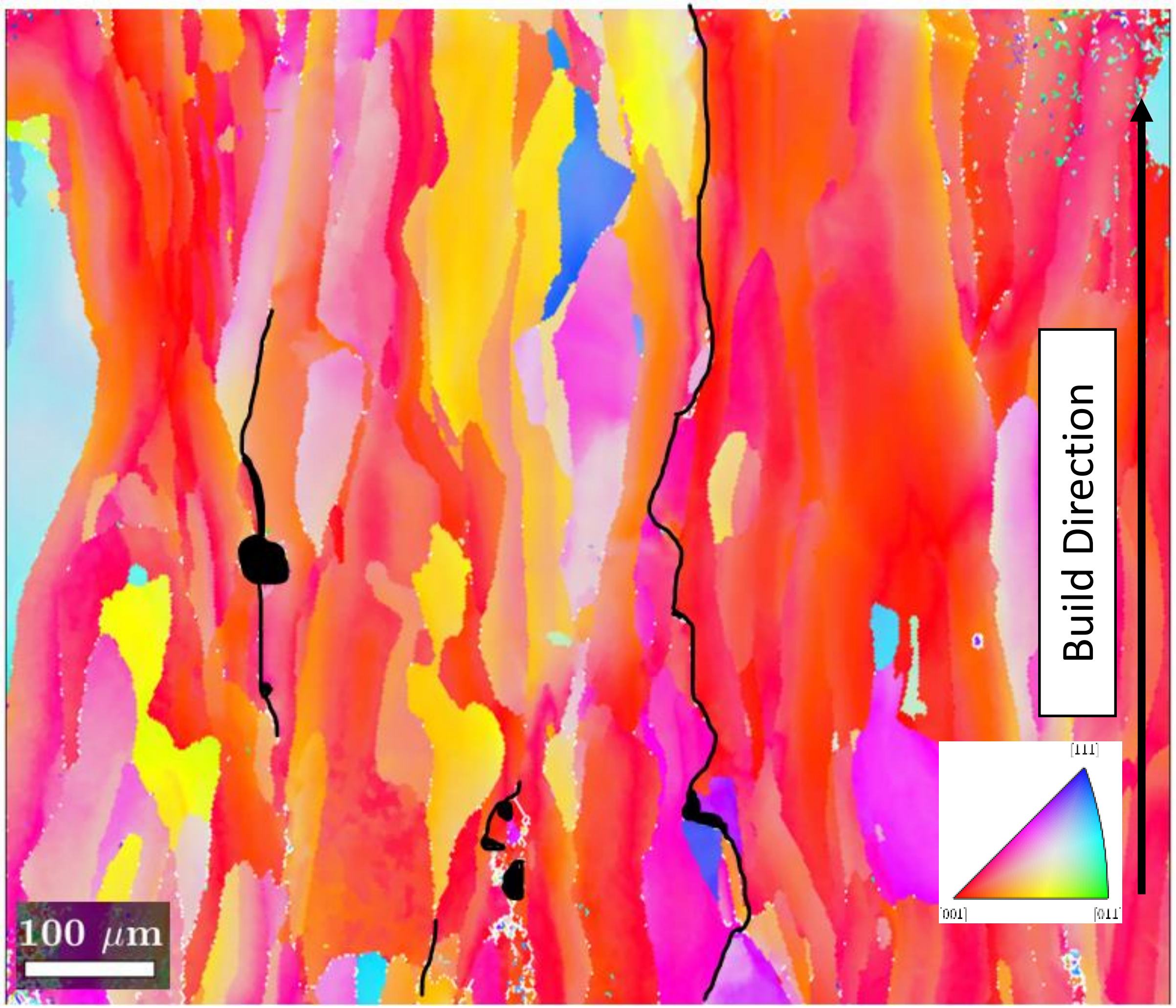
Industrial Relevance



Inverse pole figure of 3D-printed stock 6061, black regions show areas of observed solidification cracking in SEM imaging

- Aluminum alloys currently used in AM are limited, and have mostly been casting alloys (e.g. AlSi10Mg)
- Under AM conditions, many aluminum alloys tend to form columnar grains, and are subject to solidification cracking
- These results imply a need for alloys designed specifically for AM

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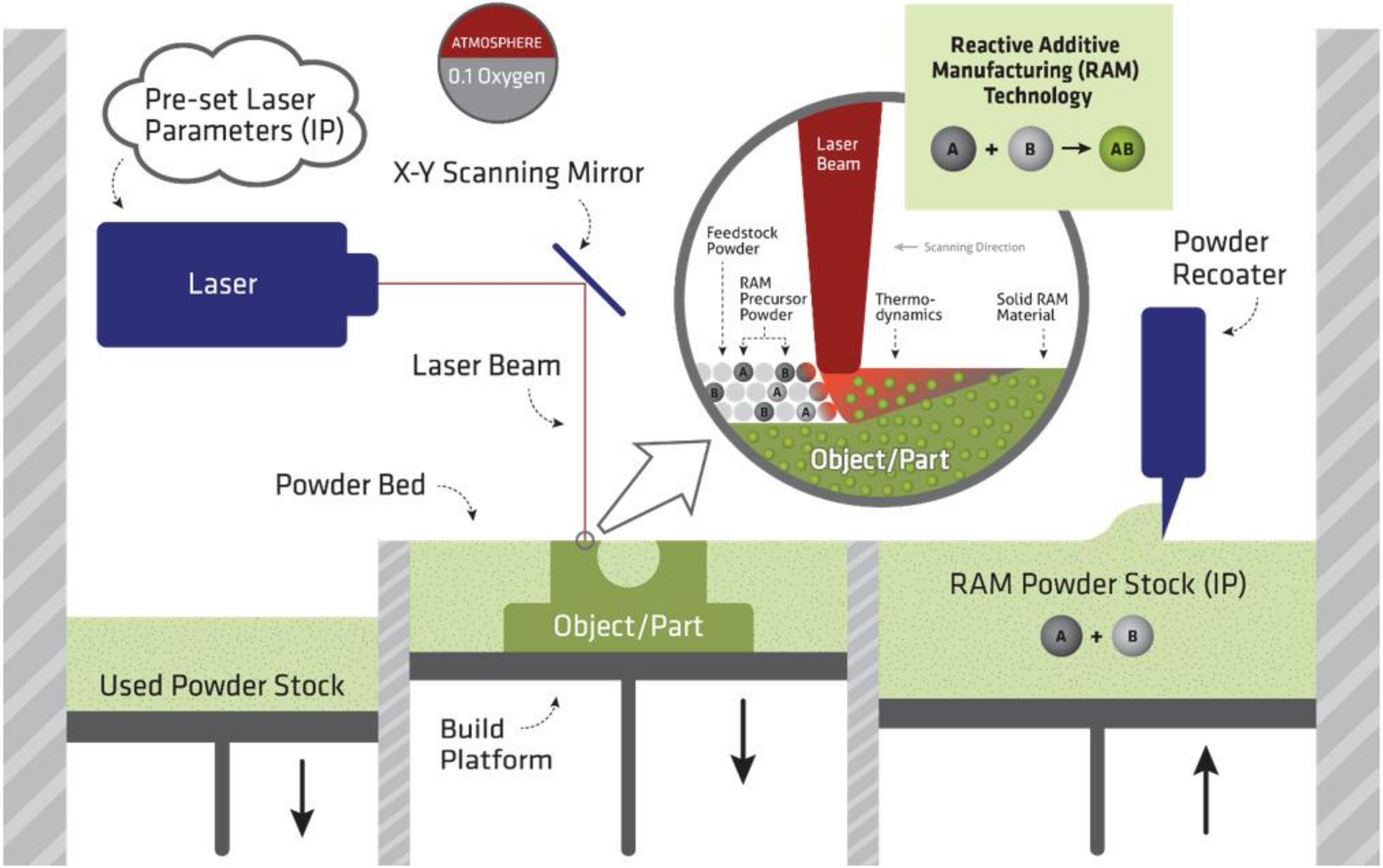
Reactive Additive Manufacturing (RAM)



Schematic provided by Elementum 3D

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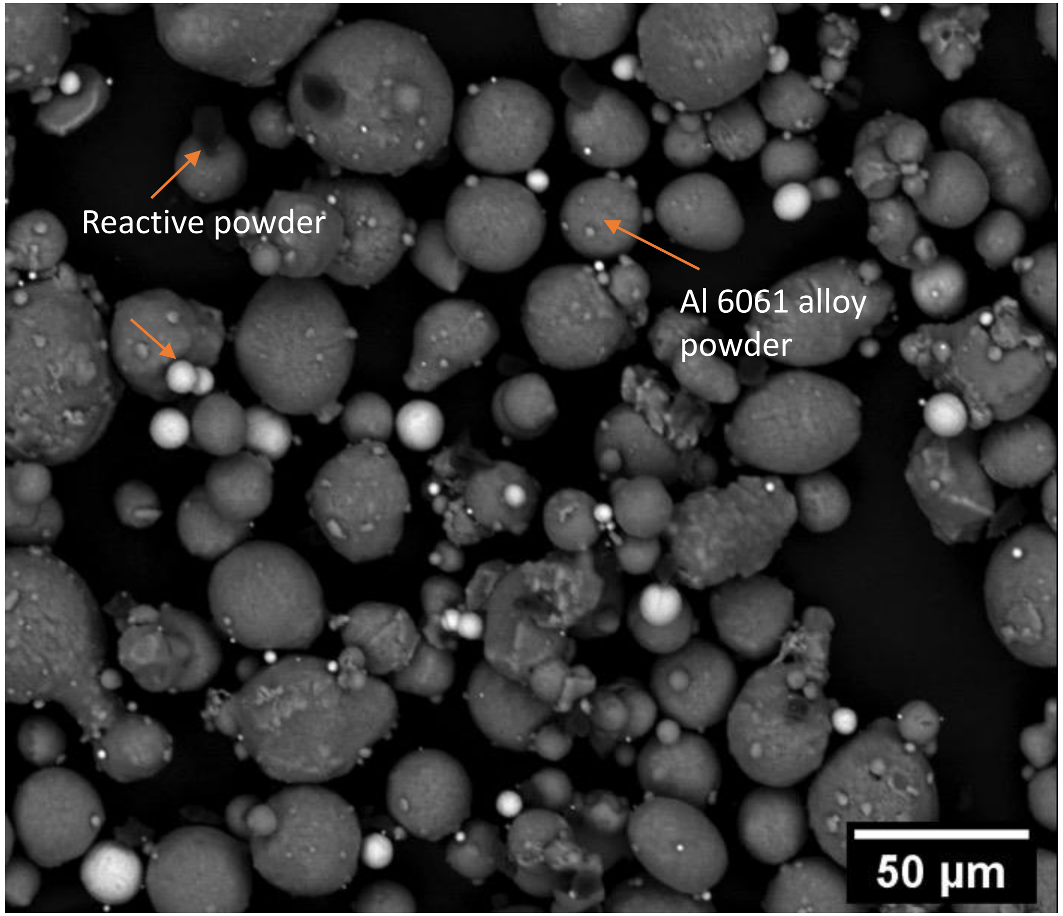
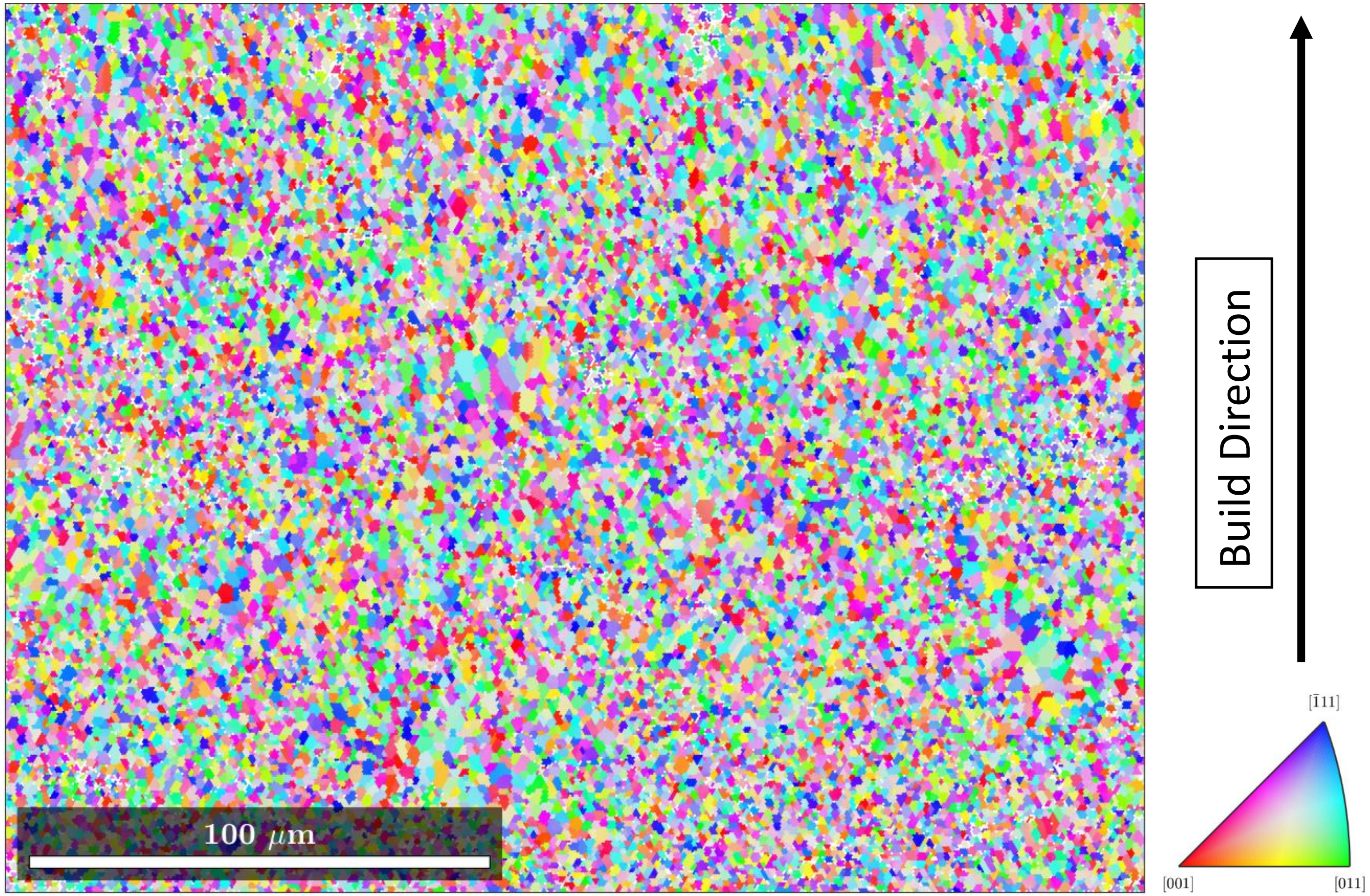
Al 6061 Reactive Additive Manufacturing (RAM) Alloy Designed for AM



BSE SEM image of Al 6061 RAM 2% alloy powder



SEM image of as built Al 6061 RAM 2%

J. S. Neuchterlein & J. J. Iten, Reactive additive manufacturing, US Patent 20160271878 A1, priority 2015-03-17, published 2016-10-22.

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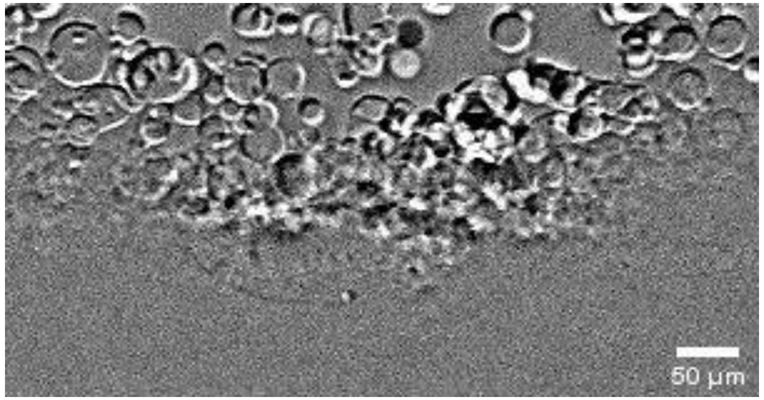
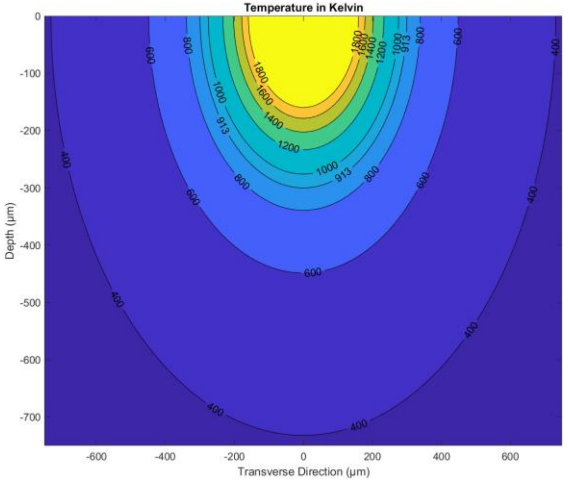
Project Scope

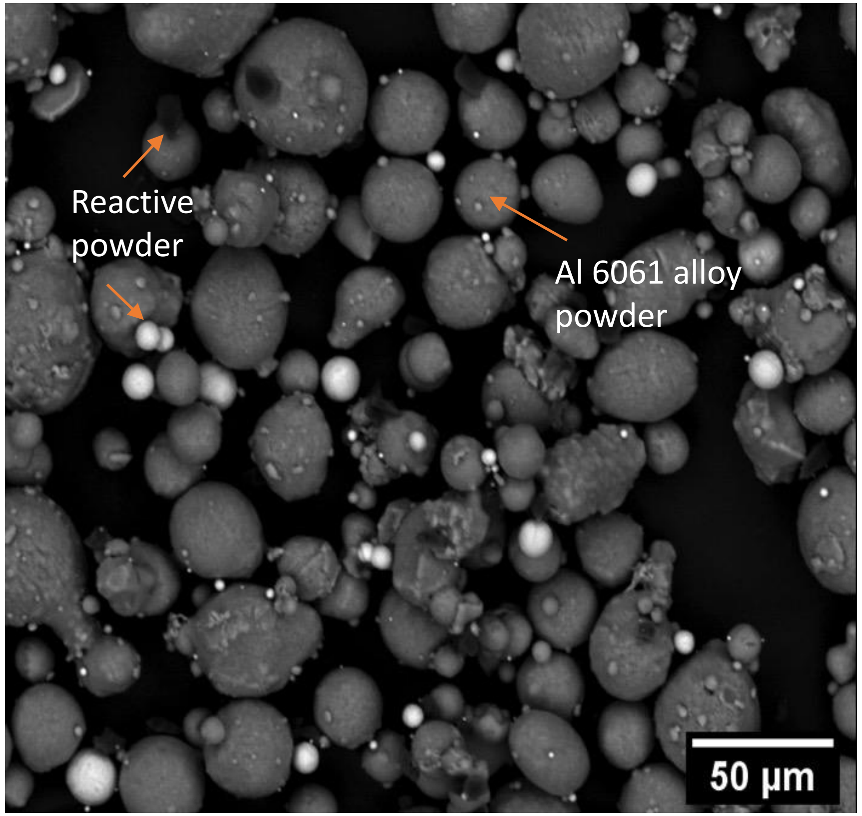
Overall Focus: What are the grain refinement mechanisms in AM of A6061-RAM alloys?

Focus Area 1: Influence of solidification conditions



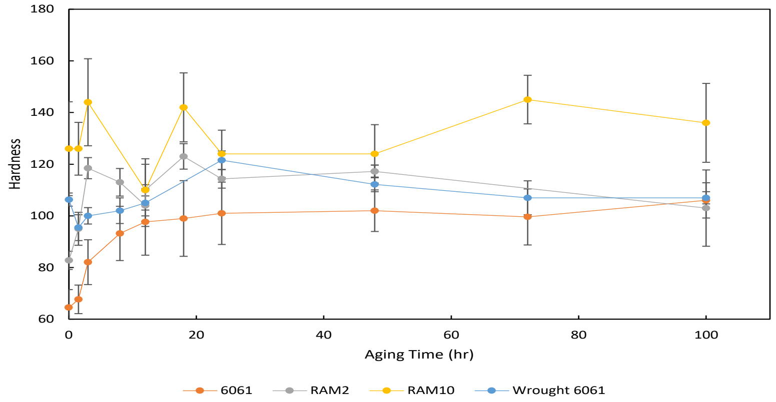


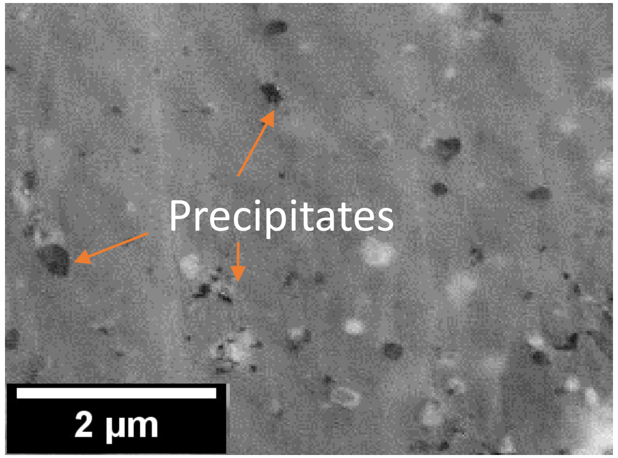
Focus Area 2: Impact of reactant particle content



SEM BSE Image of A6061-RAM2 Powder

Focus Area 3: Effect of particles on final structure after heat treatment





SEM BSE Image of A6061-RAM2 build aged at 165°C for 18h

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Degree of Refinement for Various Starting Reactive Particle Content

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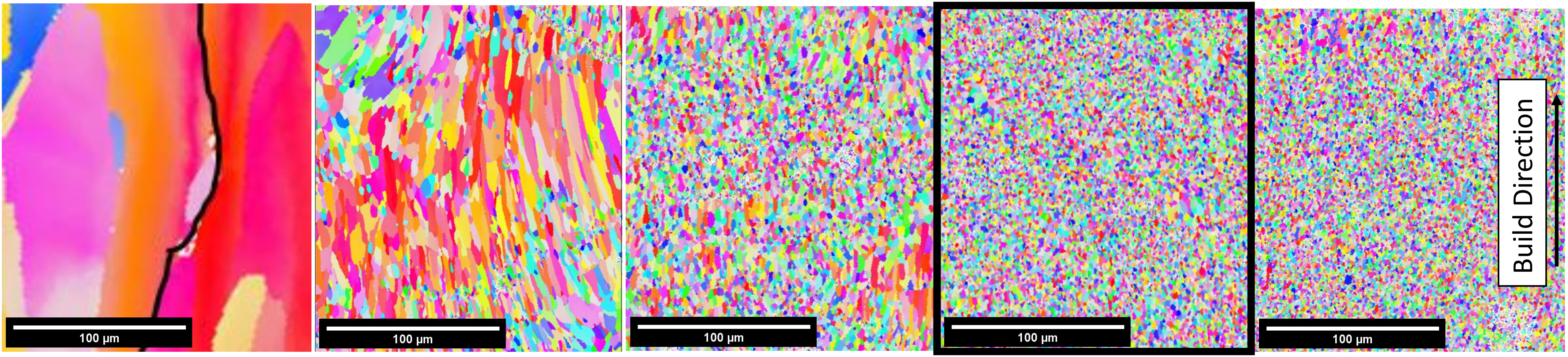


6061
Grain size:
Area: 291.38 µm^2
Diameter: 12.59 µm
(range of 2.31-941.19 µm max diameter)

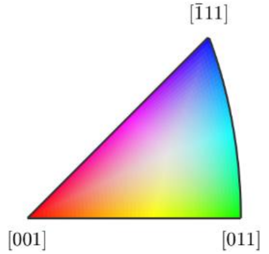
RAM 0.5
Grain size:
Area: 8.87 µm^2
Diameter: 4.37 µm

RAM 1
Grain size:
Area: 3.84 µm^2
Diameter: 2.80 µm

RAM 2
Grain size:
Area: 1.91 µm^2
Diameter: 1.93 µm

RAM 10
Grain size:
Area: 1.74 µm^2
Diameter: 1.87 µm

Increasing RAM Content



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Impact of Individual Reactive Powders (B_4C or Ti) vs. A6061-RAM2 (B_4C + Ti)

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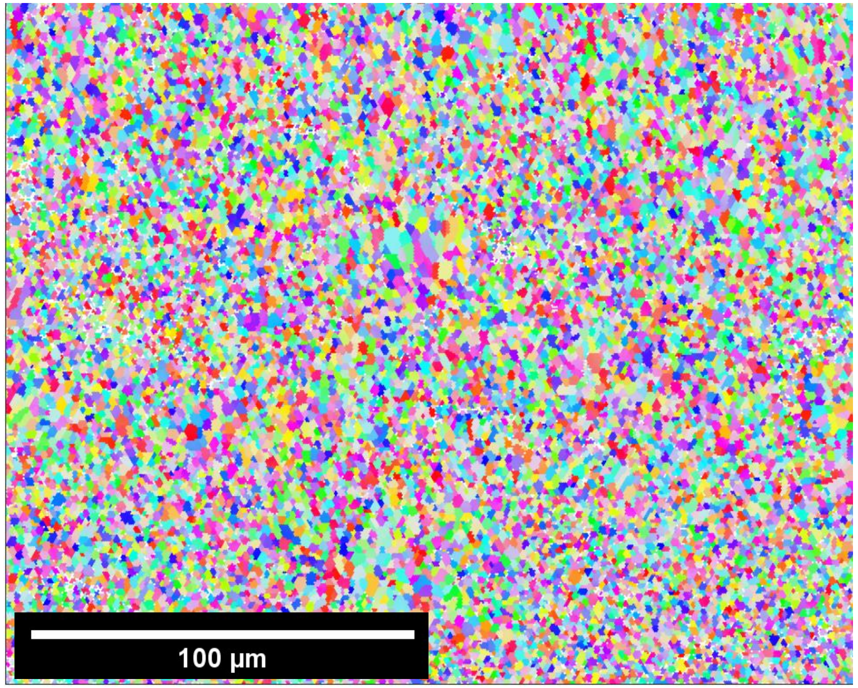
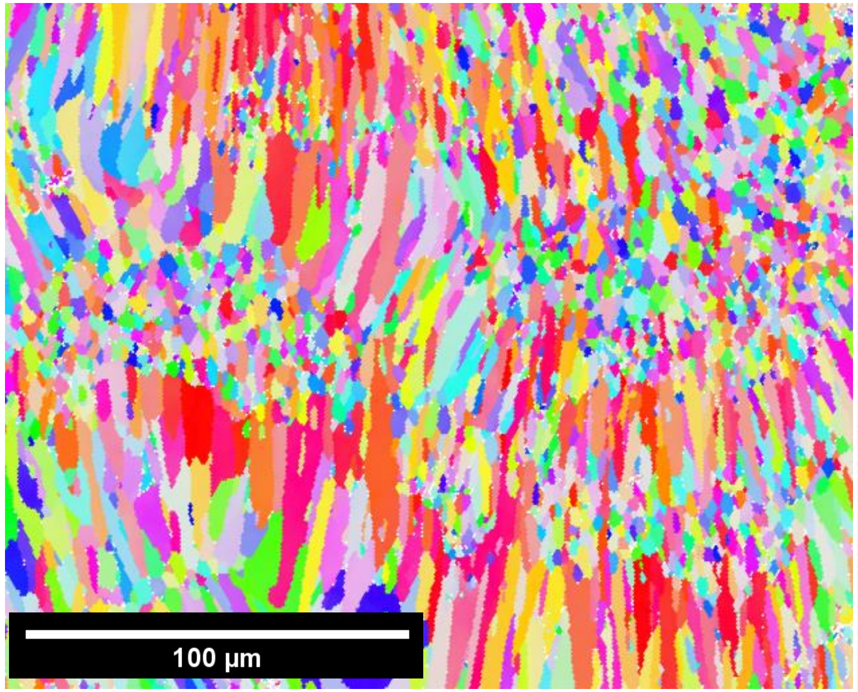
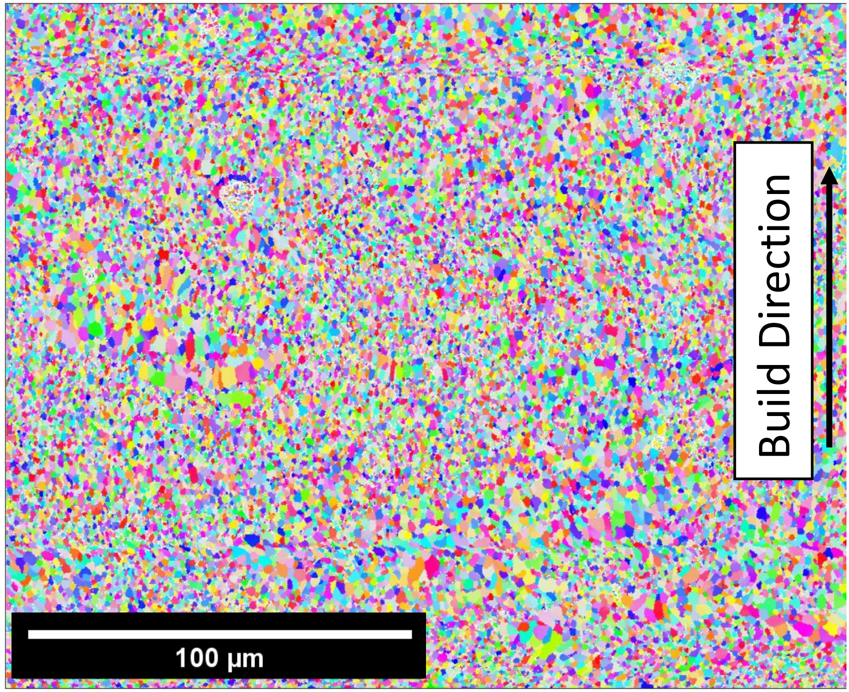
All builds were performed with 40 um layer thickness, 370 W, 0.2 mm hatch spacing



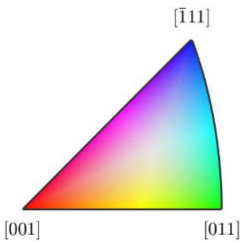
A6061-RAM2
1300 mm/s
Grain size:
Area: 1.95 µm^2
Diameter: 1.96 µm



A6061-RAM: B_4C
1300 mm/s
Grain size:
Area: 9.65 µm^2
Diameter: 4.81 µm



A6061-RAM: Ti
1300 mm/s
Grain size:
Area: 0.73 µm^2
Diameter: 1.16 µm



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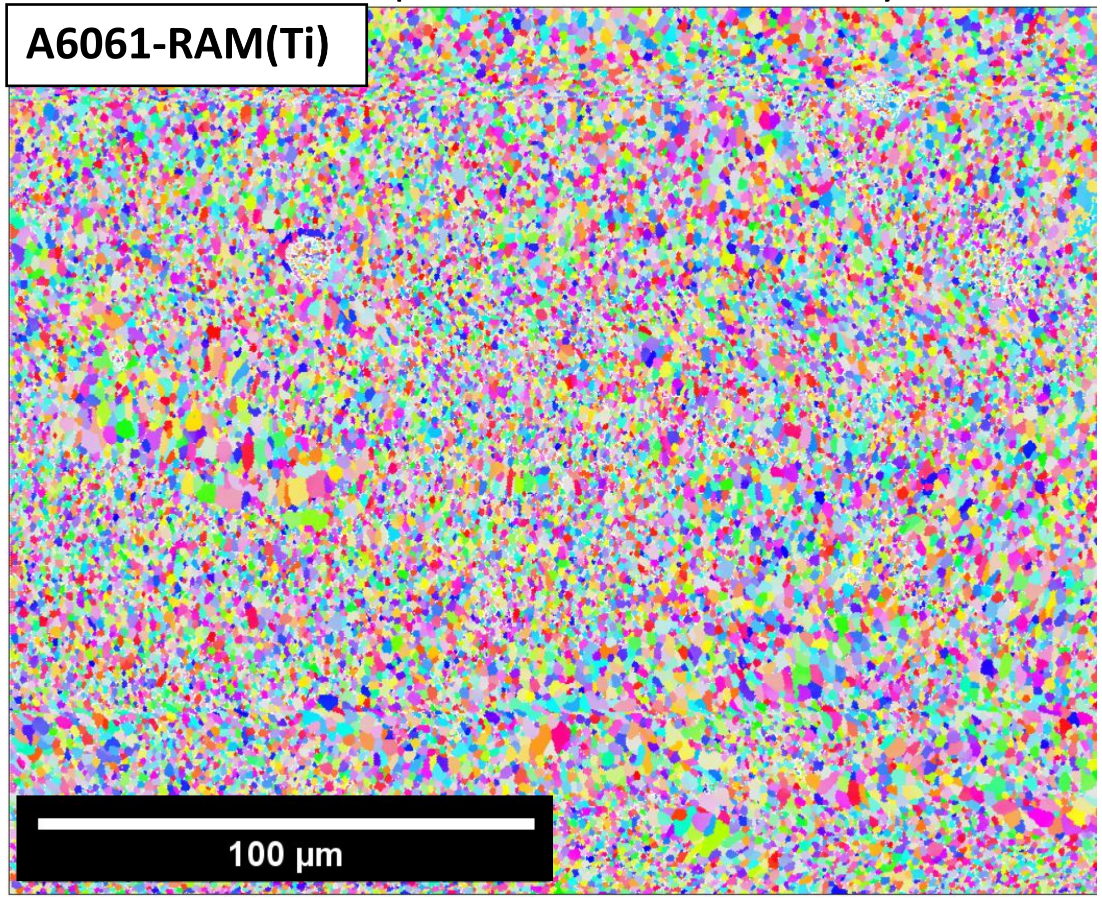
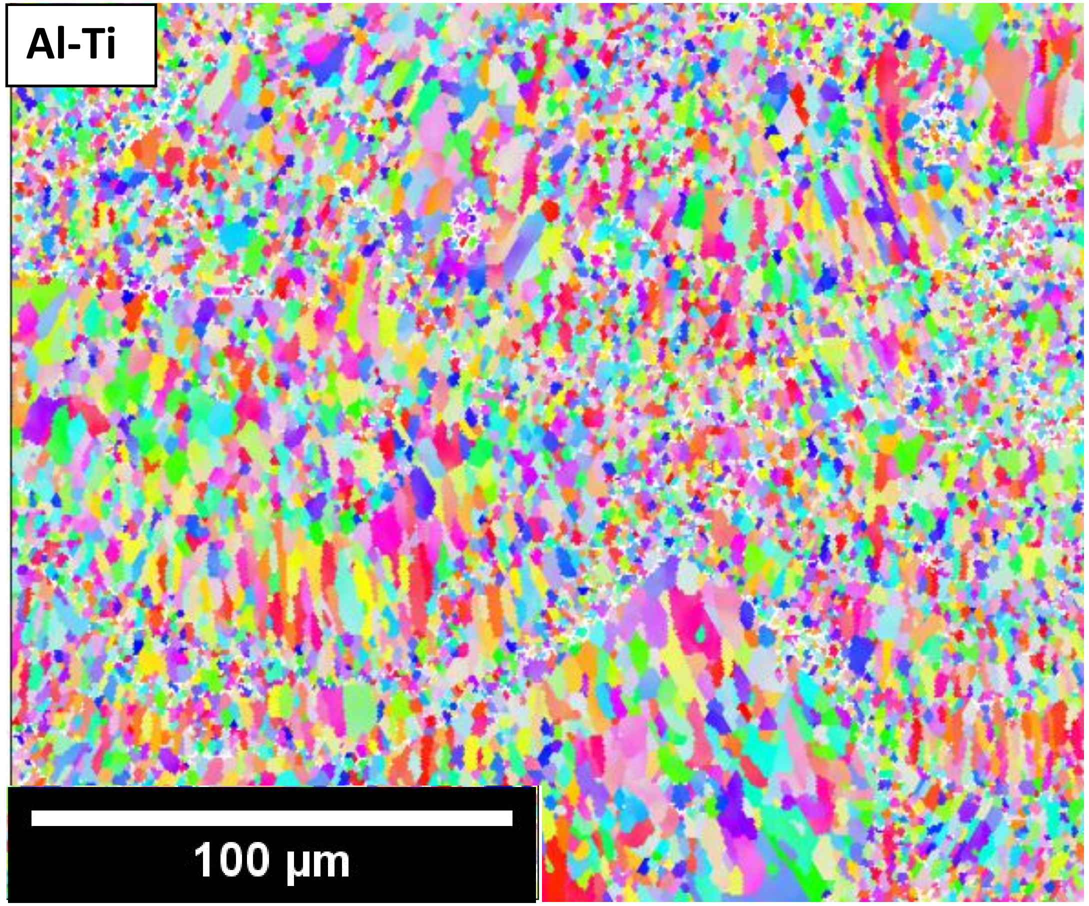
Comparison of Grain Structure A6061-RAM(Ti) vs. Al-Ti

All builds were performed with 40 um layer thickness, 370 W, 1300 mm/s, and 0.2 mm hatch spacing

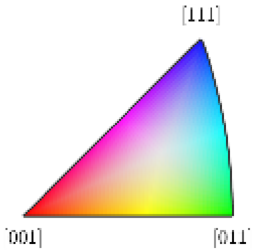


Grain size:
Area: 0.73 μm²
Diameter: 1.16 μm



Build Direction

Grain size:
Area: 2.83 μm²
Diameter: 2.32 μm



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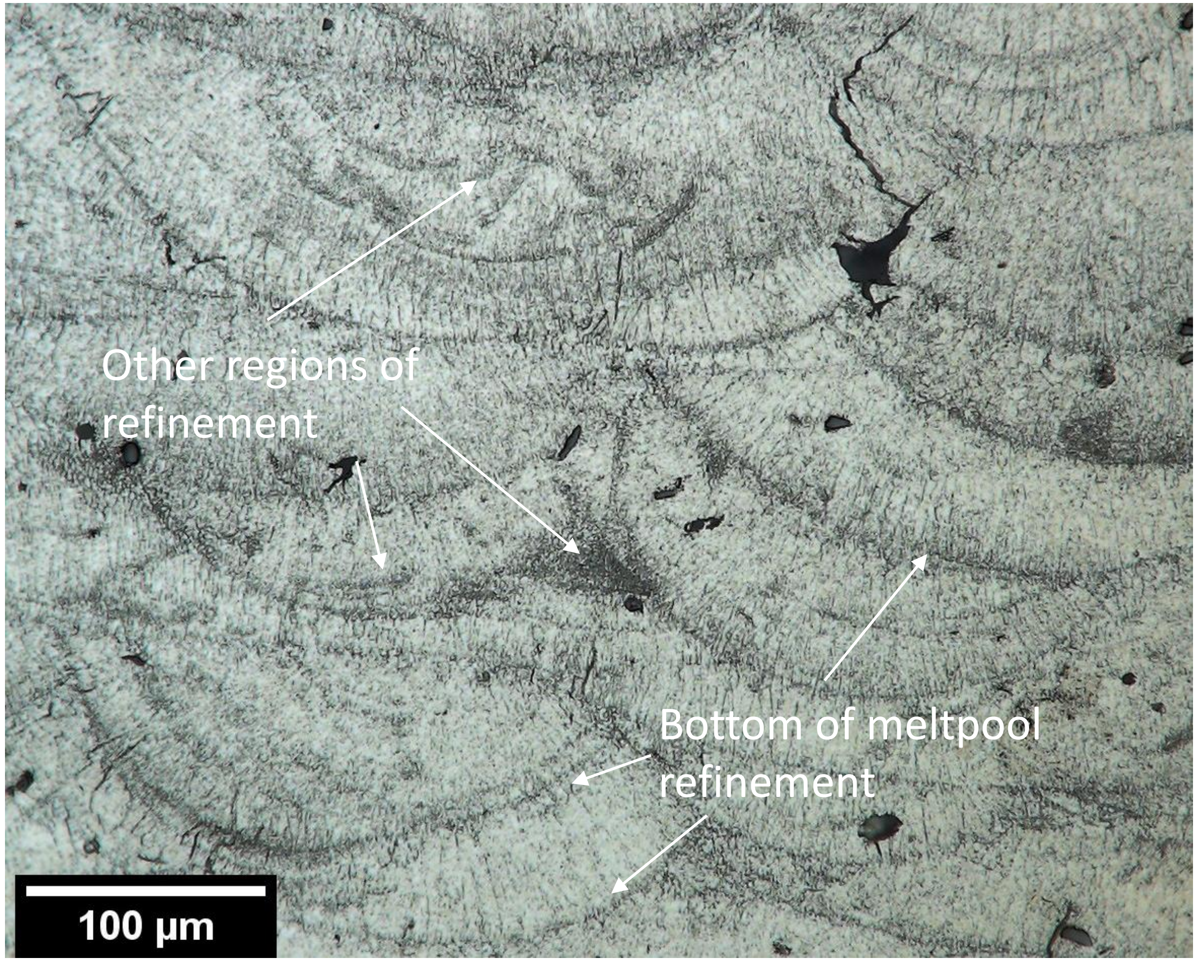
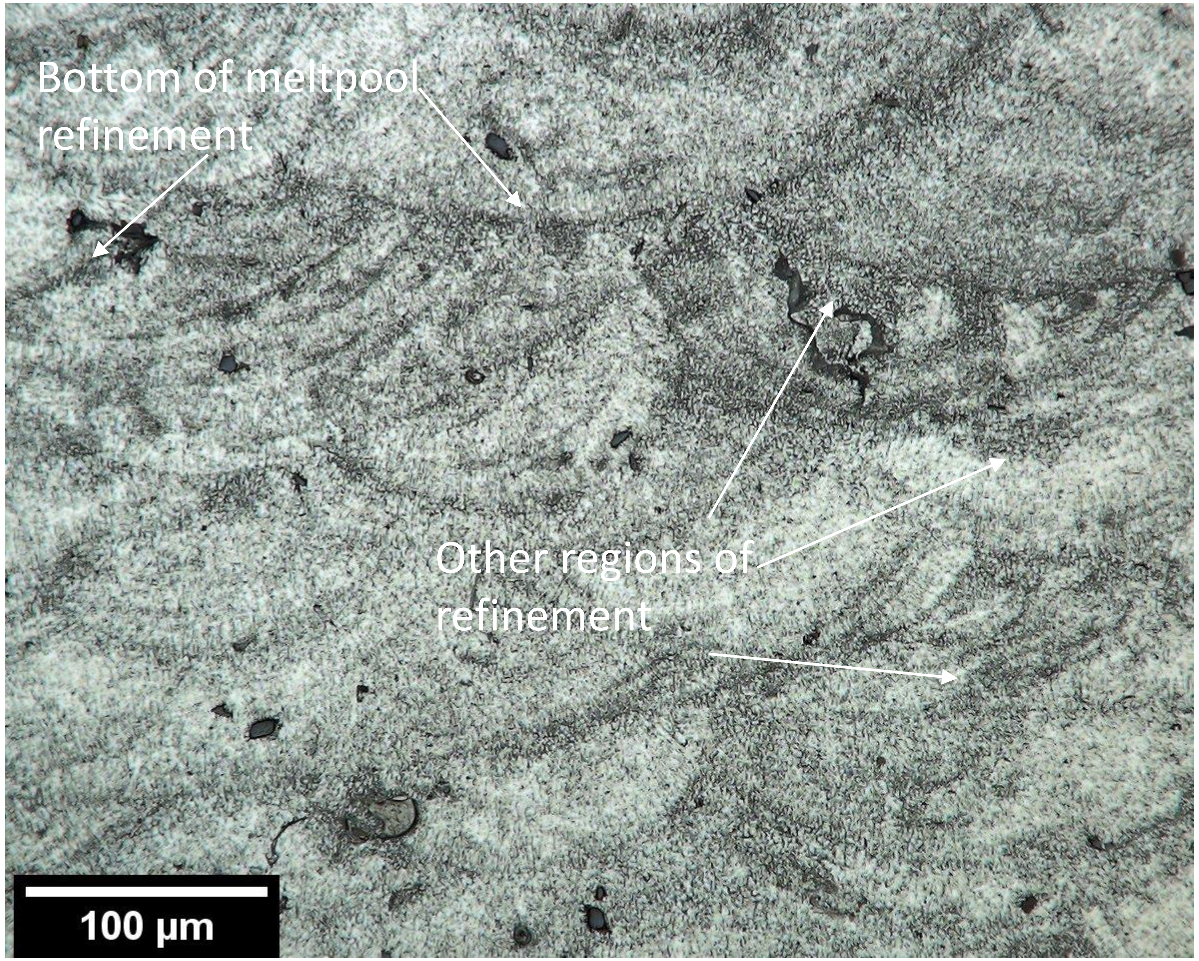
Refinement Locations (Lower Reactive Particle Content)

A6061-RAM0.5



A6061-RAM1



Samples etched with Kroll's Reagent

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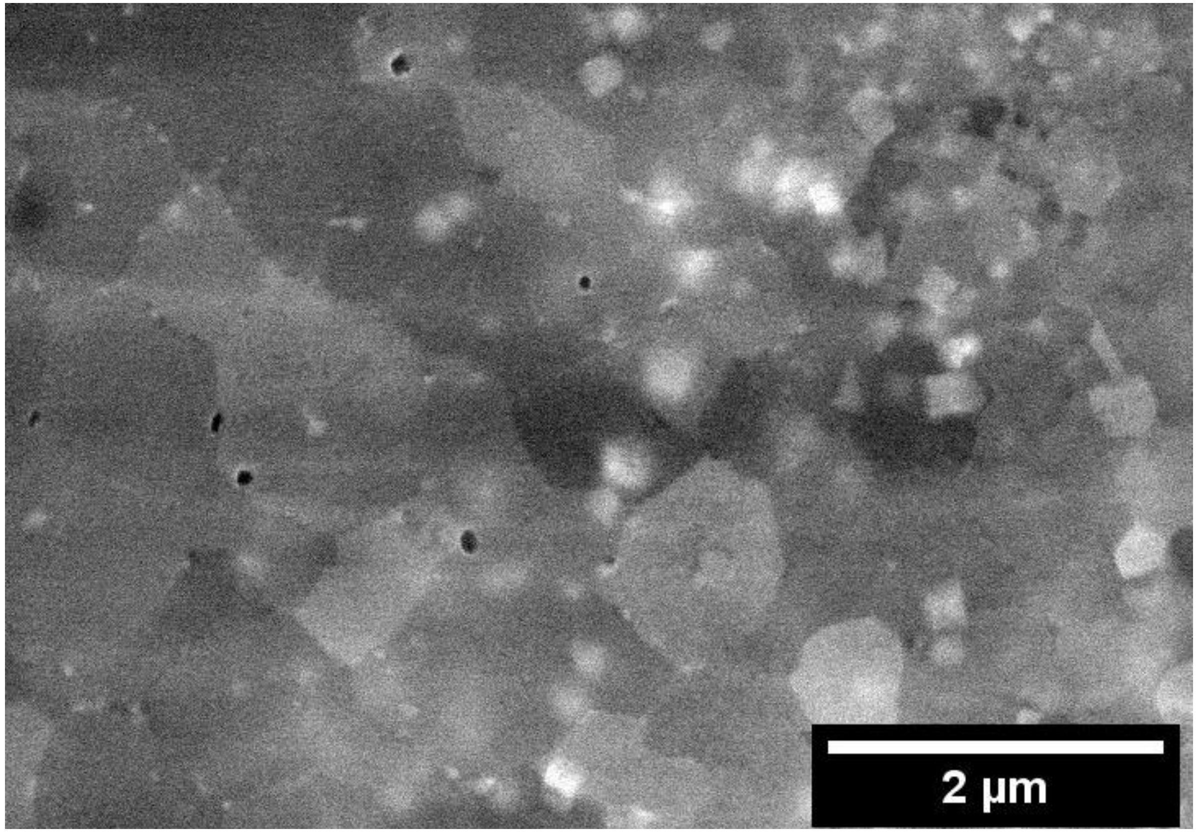
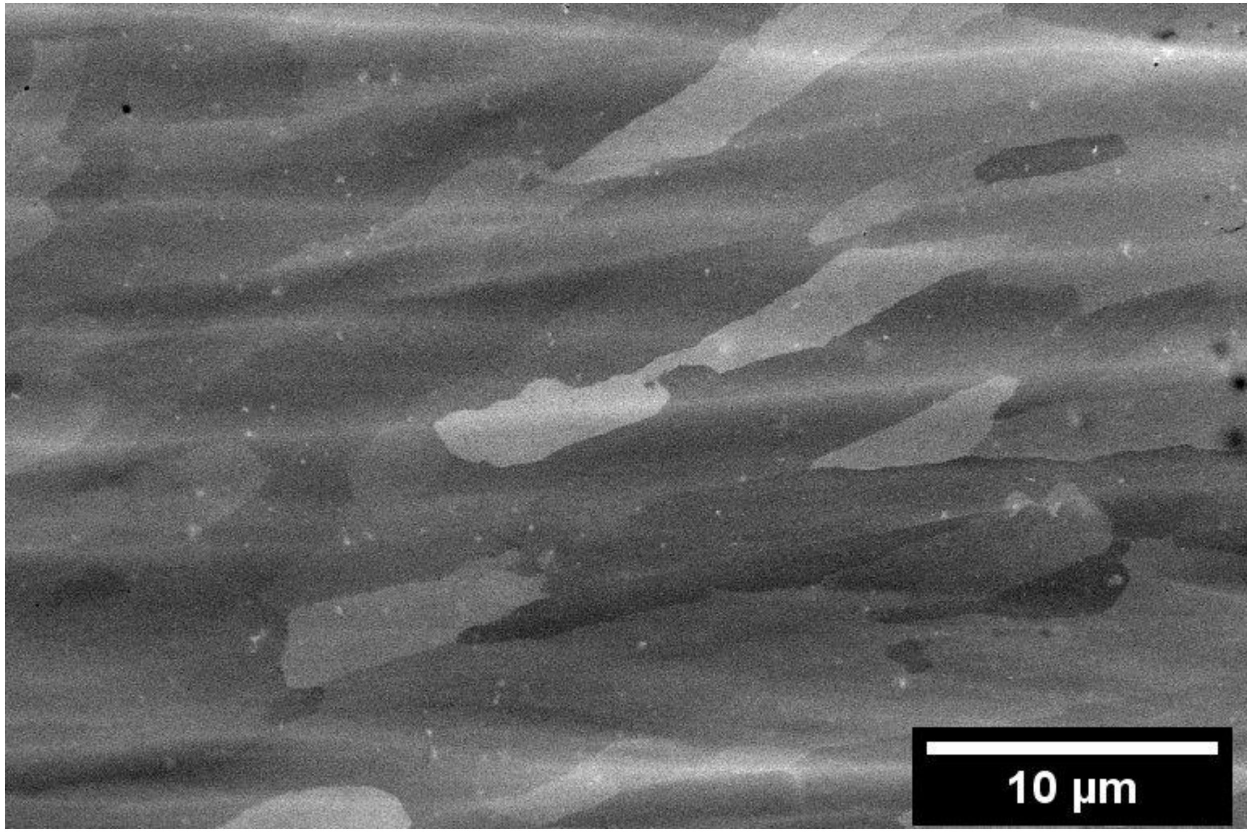
Refined vs. Columnar Regions for Low RAM Content Alloys





SEM-BSE Images taken from an A6061-RAM0.5 sample

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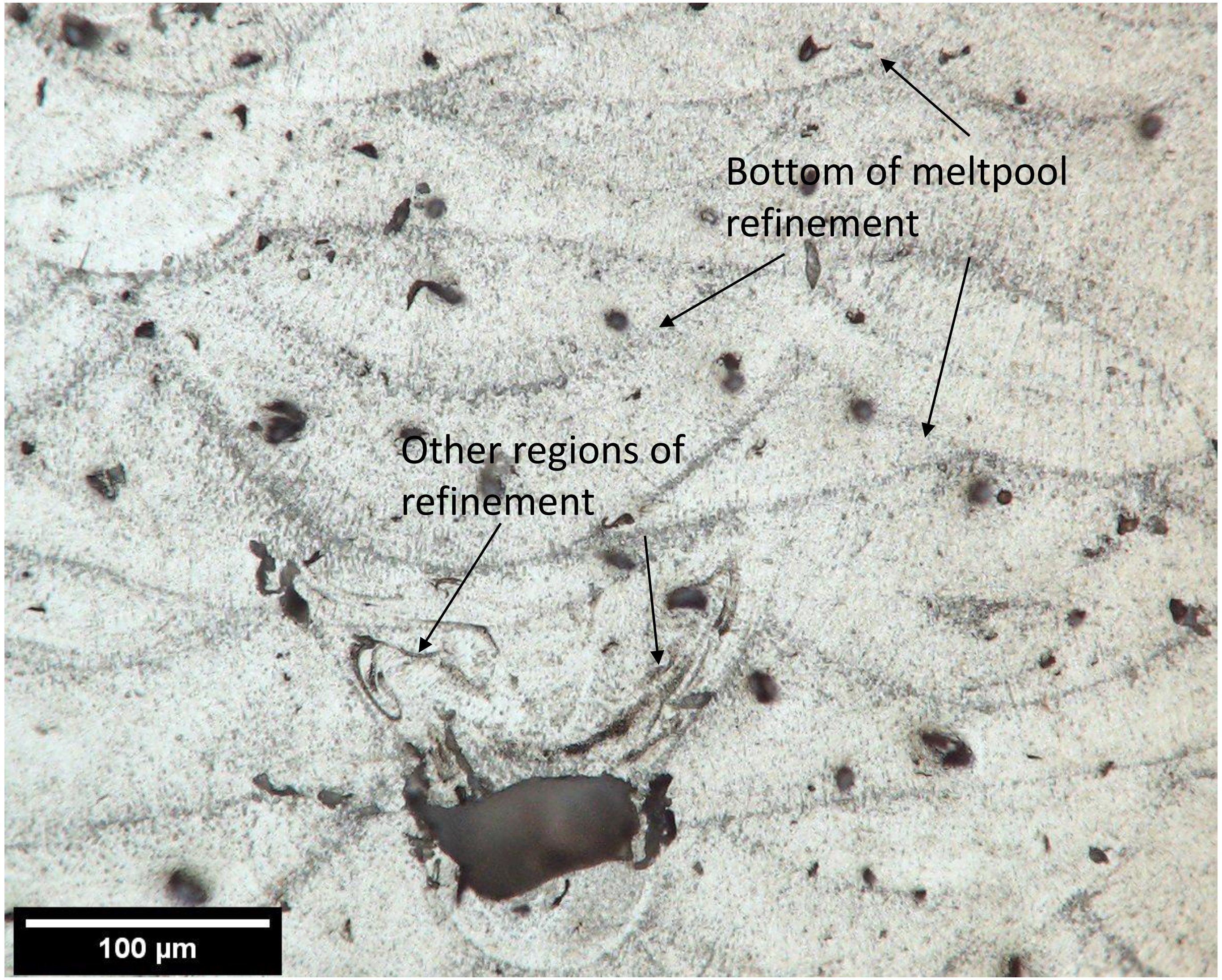
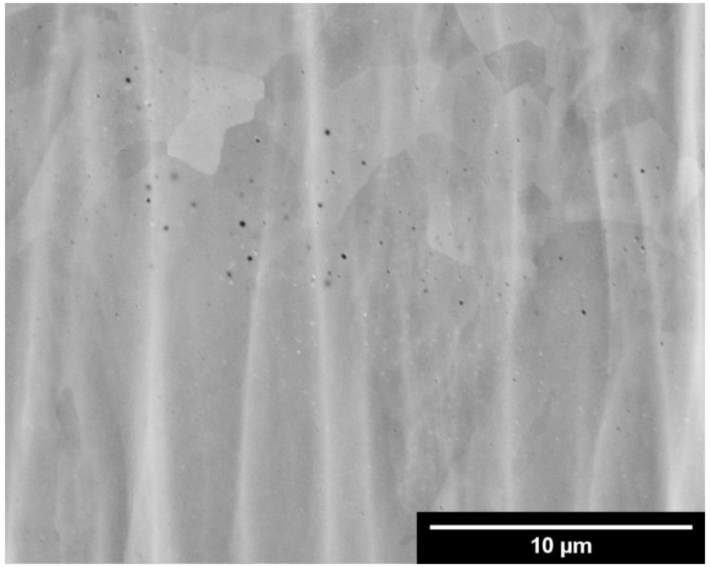
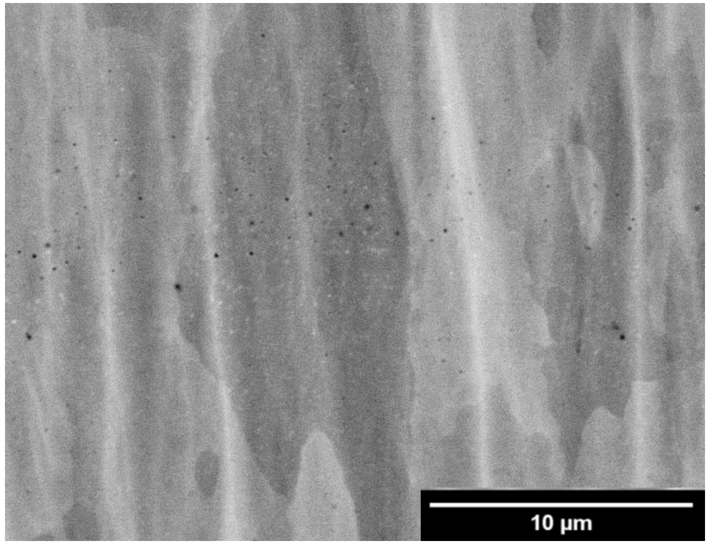
Refinement Locations A6061-RAM(B_4C)







Samples etched with Kroll's Reagent

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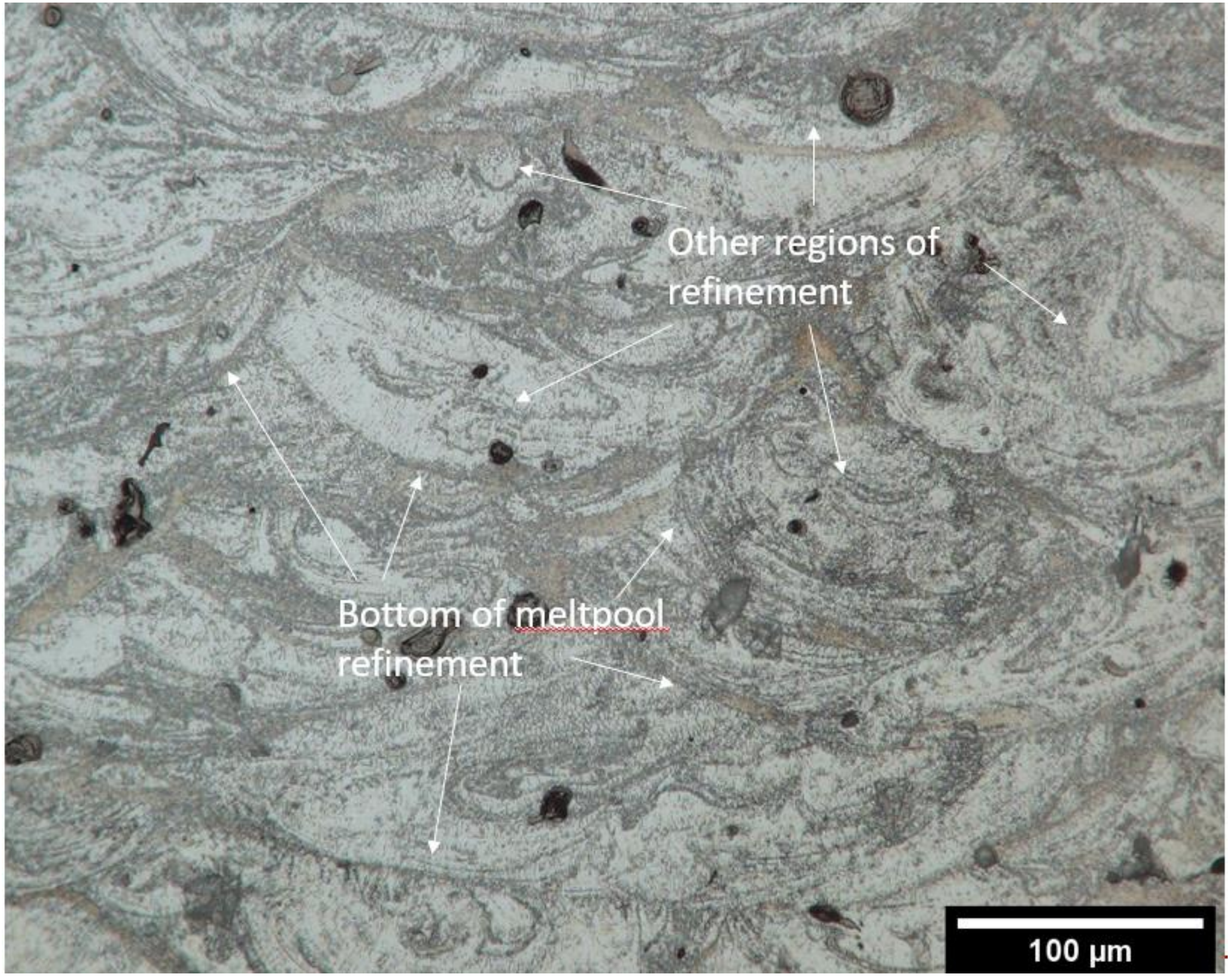
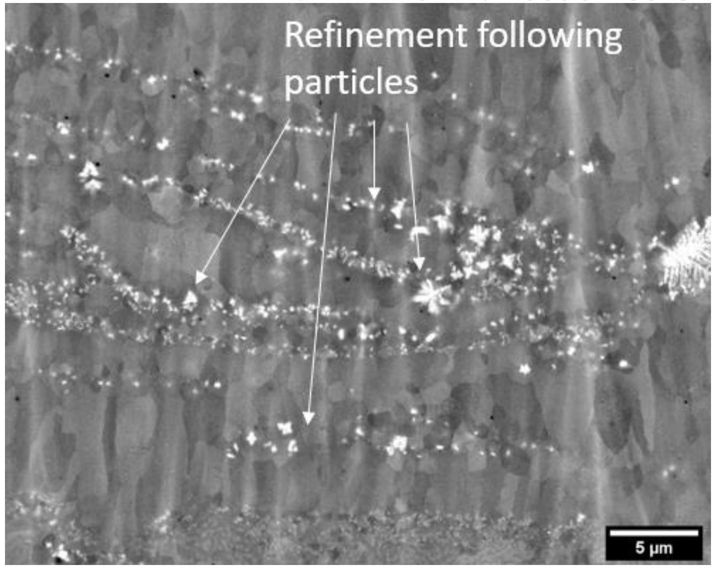
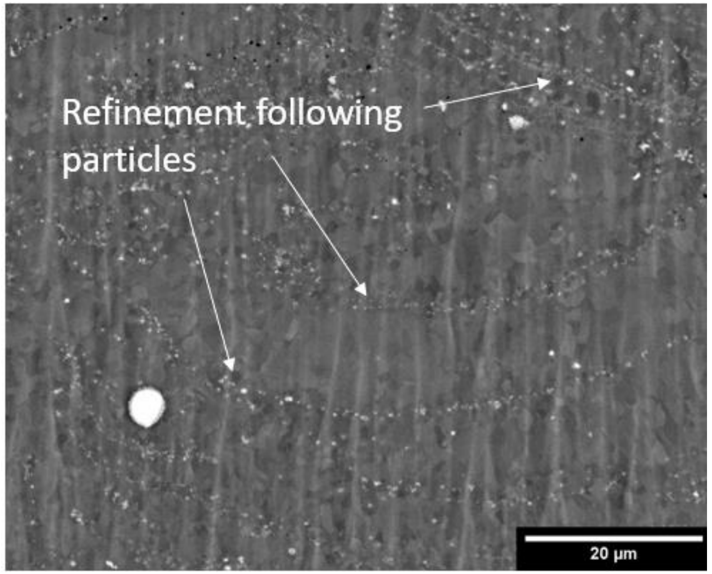
Refinement Locations in Al-Ti







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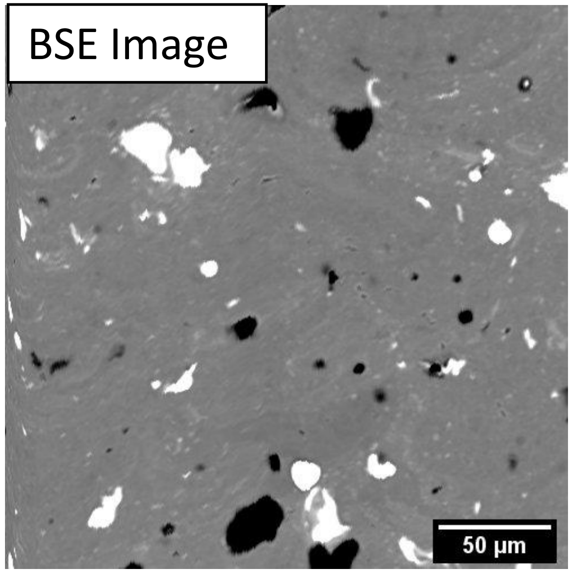
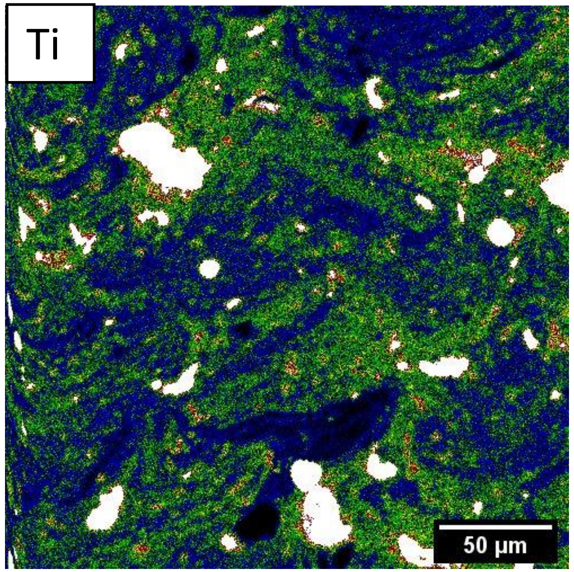
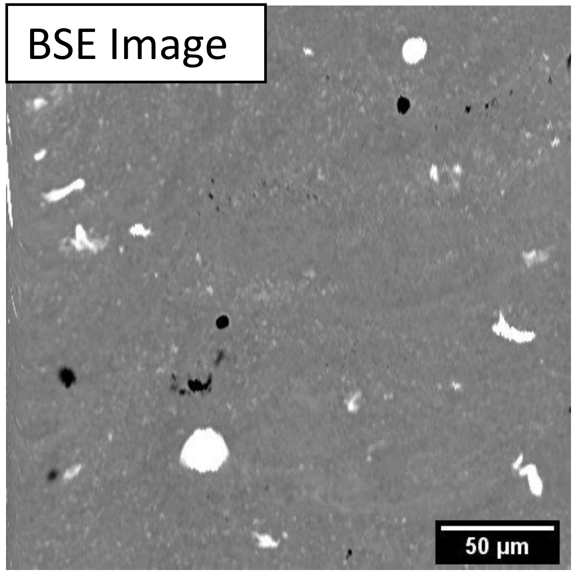
Electron Microprobe Maps of Solute Distribution

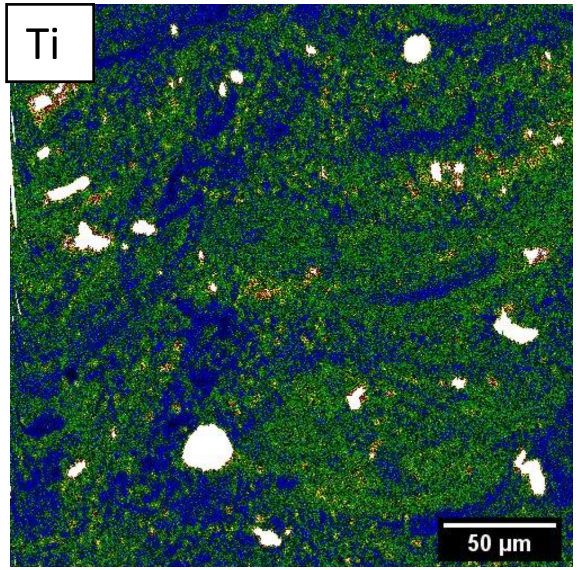
Al-Ti

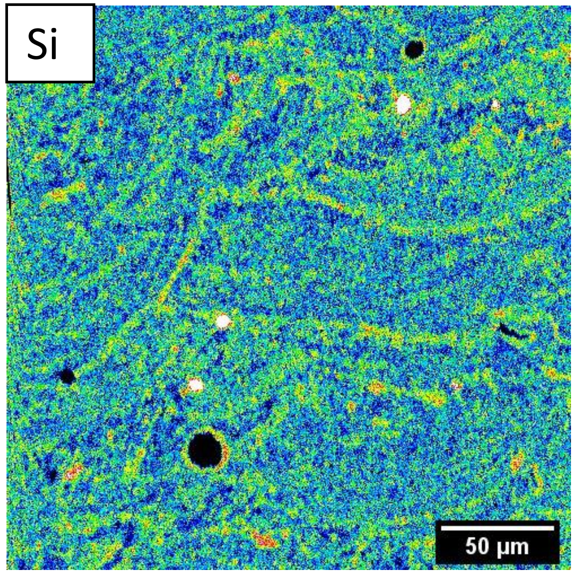




A6061-RAM(Ti)







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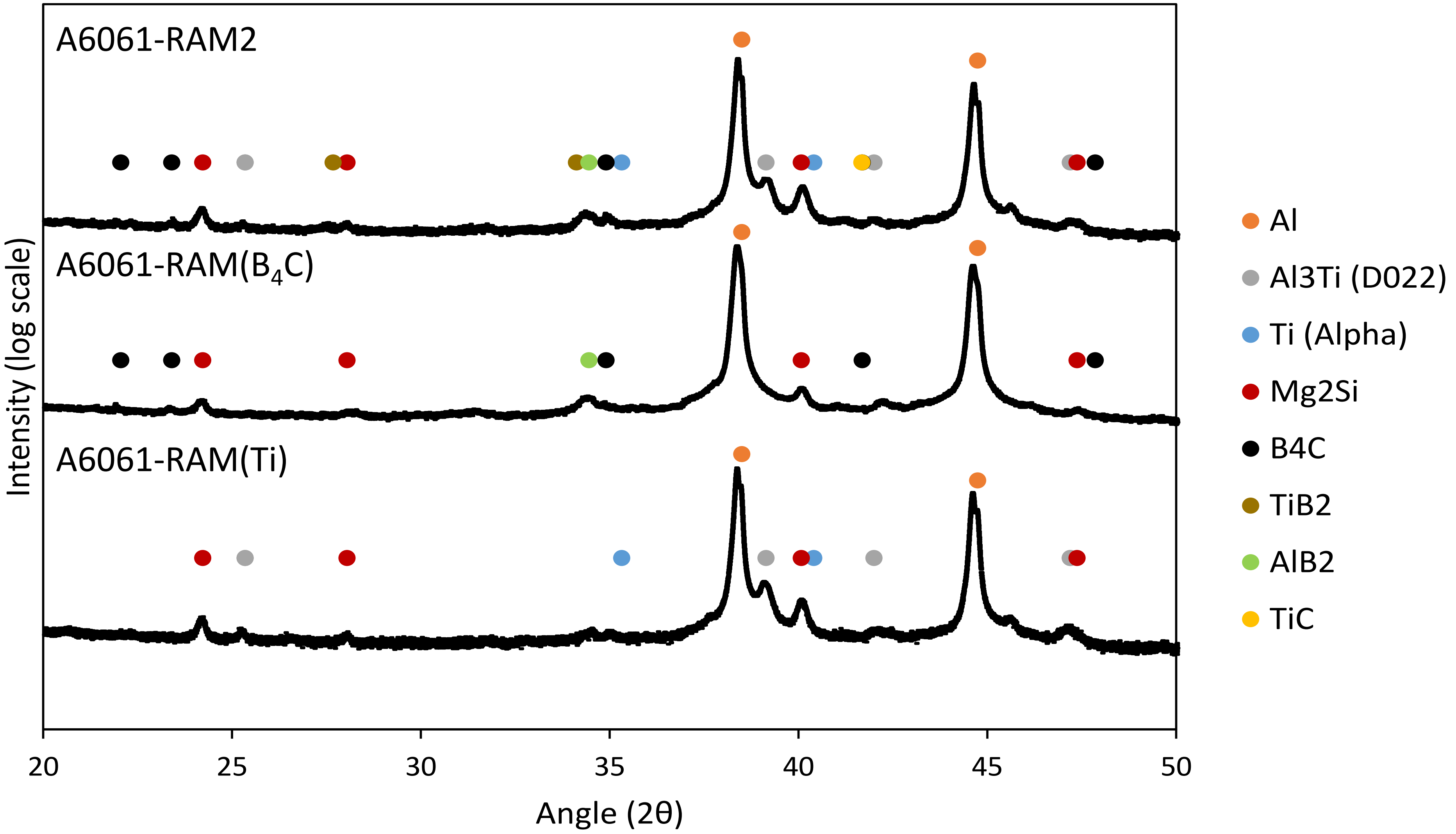
Particle Types (XRD) For Individual Reactive Powders



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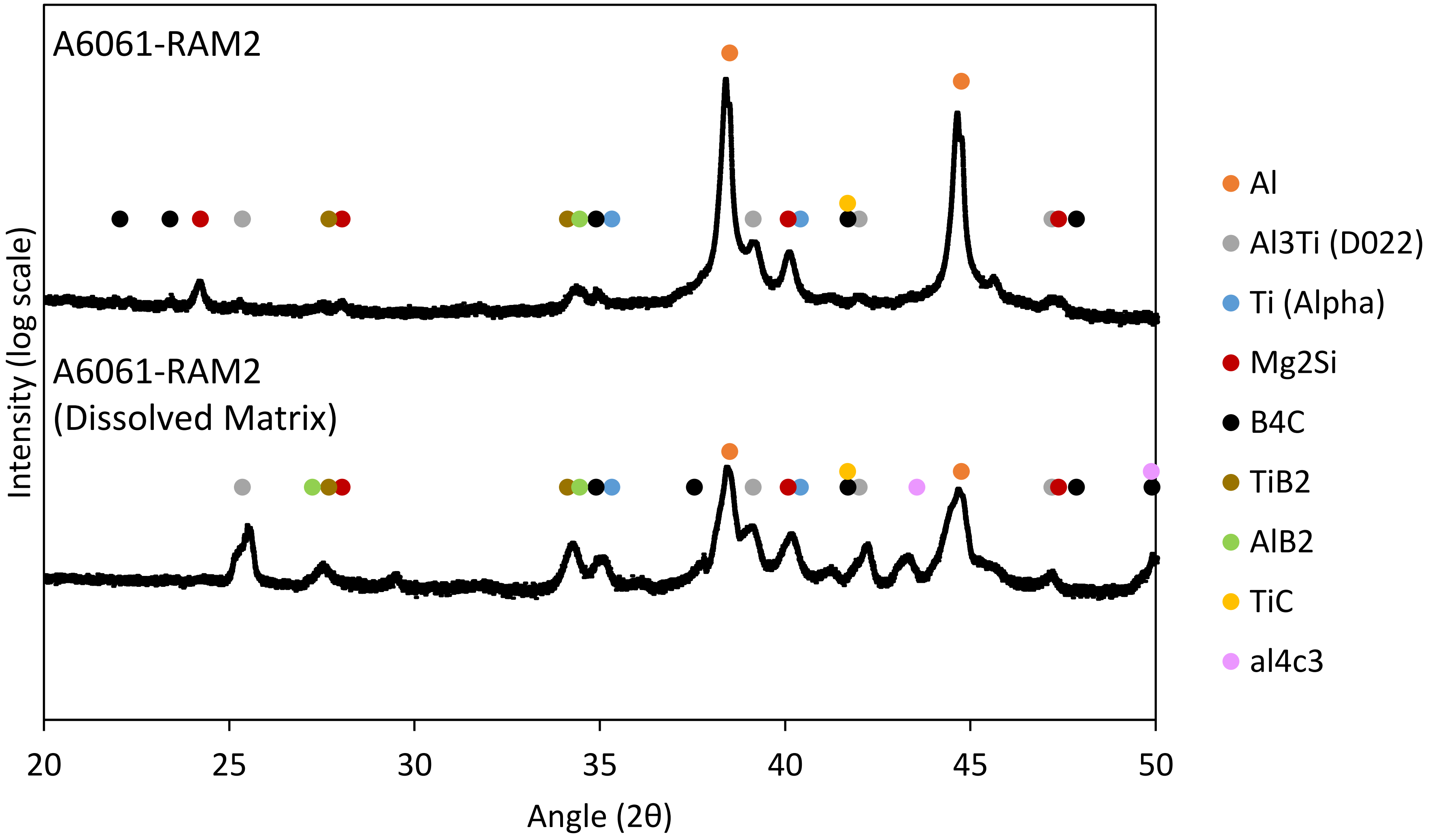
Particle Types (XRD) For A6061-RAM2 Dissolved Matrix



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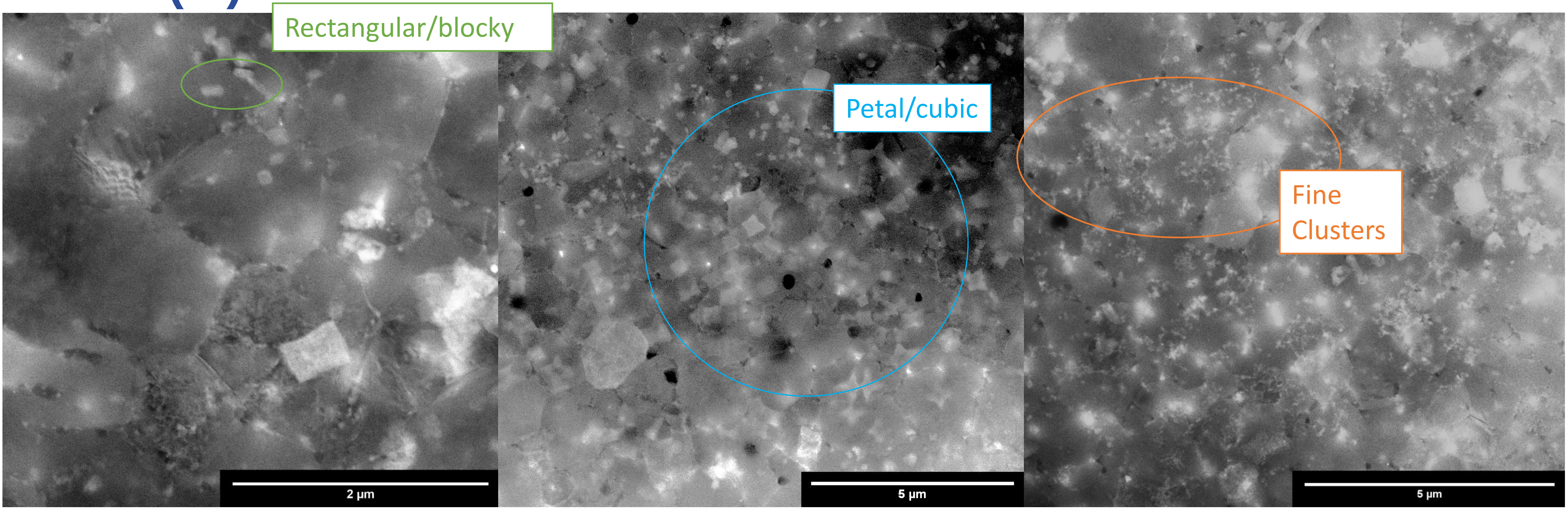
Observed Particle Morphologies A6061-RAM(Ti)



Observed Particle Morphologies Al-Ti

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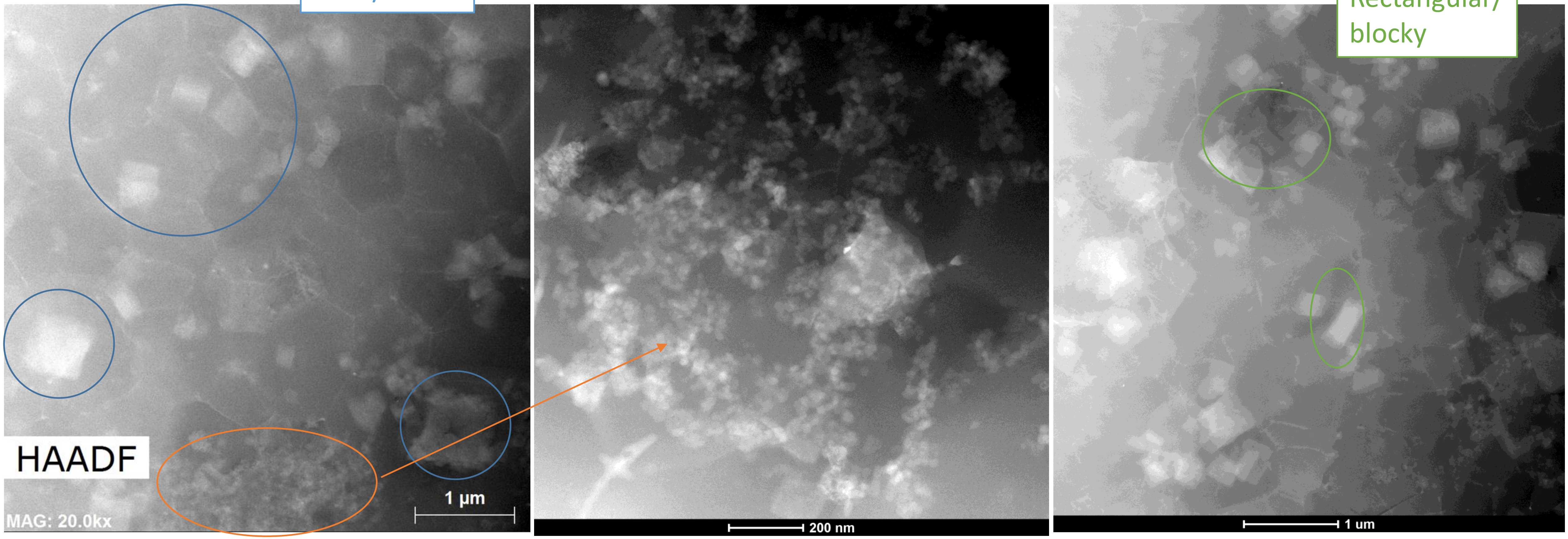

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Particle Characteristics Al-Ti and A6061-RAM (Ti)

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Characteristics of particles observed in selected RAM alloys

Alloy	Morphology	Size (nm)	Chemistry (wt. %)	Location
Al-Ti	Petal/cubic	140 ± 110	Al: 80.6 ± 6.7 Ti: 18.4 ± 5.7 Al/Ti = 4.8 ± 1.7	center of grains, other regions of grains/over boundaries
	Rectangular/blocky	310 ± 90 x 150 ± 40	Al: 75.1 ± 4.0 Ti: 20.5 ± 4.6 Al/Ti = 3.9 ± 1.2	center of grains, other regions of grains/over boundaries
	Fine	<30	Al: 91.2 ± 1.1 Ti: 8.4 ± 1.1 Al/Ti: 11.1 ± 1.7	dispersed throughout matrix
A6061-RAM(Ti)	Petal/cubic	180 ± 120	Al: 75.7 ± 5.1 Ti: 21.6 ± 4.8 Al/Ti = 3.76 ± 1.3	center of grains, other regions of grains/over boundaries
	Rectangular/blocky	320 ± 110 x 90 ± 30	Al: 70.72 ± 5.5 Ti: 25.14 ± 5.74 Al/Ti = 2.97 ± 0.82	center of grains, other regions of grains/over boundaries
	Fine	<30	Al: 86.67 ± 5.06 Ti: 10.30 ± 3.75 Al/Ti: 9.62 ± 4.09	dispersed throughout matrix

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Summary & Future Work

- RAM alloys containing different reactant particle content were considered to investigate the impact of reactant particle content on grain refinement
  - Increasing vol. % of two reactive constituents increased the amount and degree of refinement, although this was not significant past 2 vol. %
  - Of the two constituents (Ti and B_4C) Ti was found to contribute more to refinement and achieve results similar to the combination of the two, while B_4C was found to only minorly refine the microstructure
- In samples containing both refined regions and columnar regions, refined regions were found to correspond to particle containing locations determined by where reactant particles were distributed in the melt
- XRD showed the presence of a variety of particles in these materials, with many small peaks corresponding to small volume fractions being observed; these small volume fraction phases will be observed more using TEM analysis

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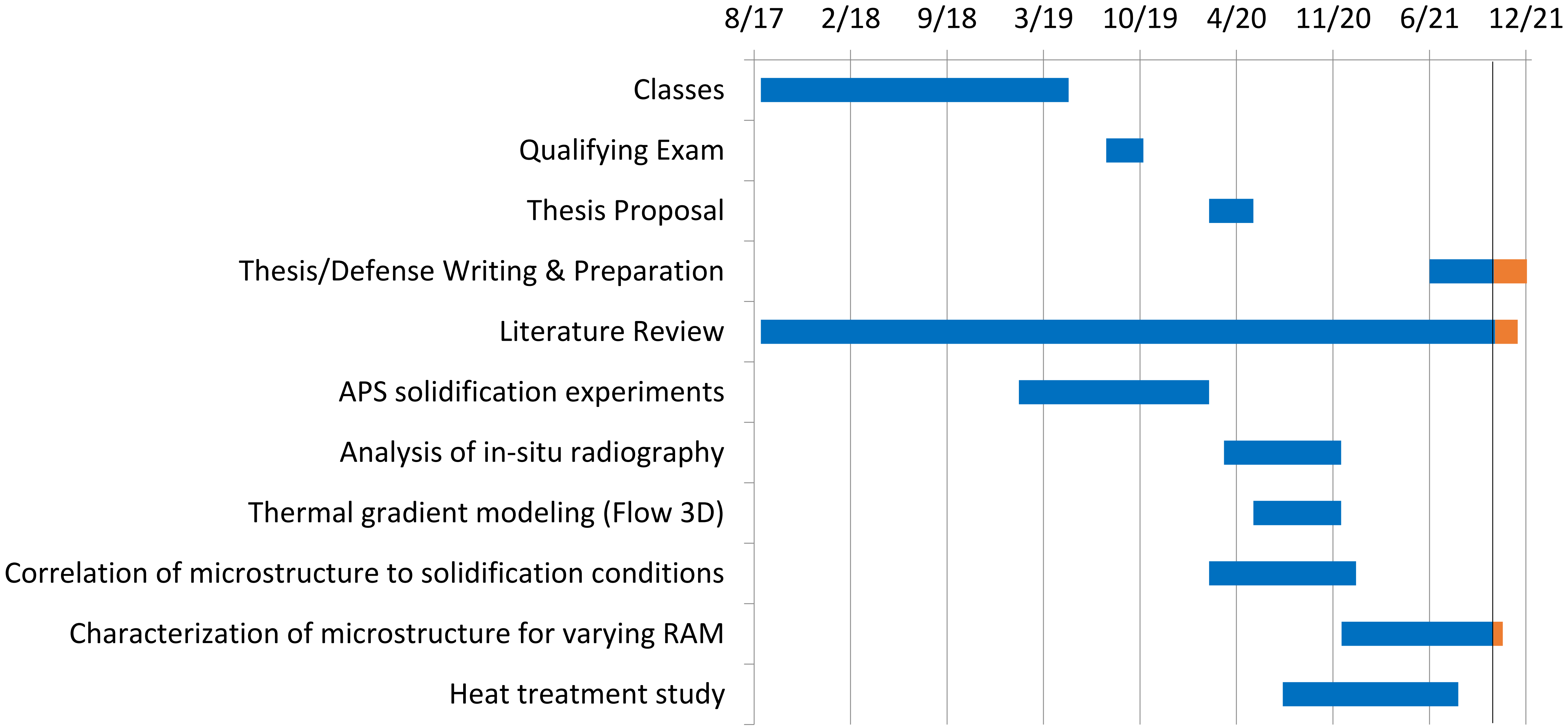
Progress



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Challenges & Opportunities

Challenges

- Finishing up TEM capture of various particle morphologies and chemistries in remaining alloys

Opportunities

- Identifying impact of inoculants on refinement to help inform alloy design for refinement using the RAM process

Thank you!

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