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

Fall Meeting

October 13th – 15th 2020

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

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 August 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.

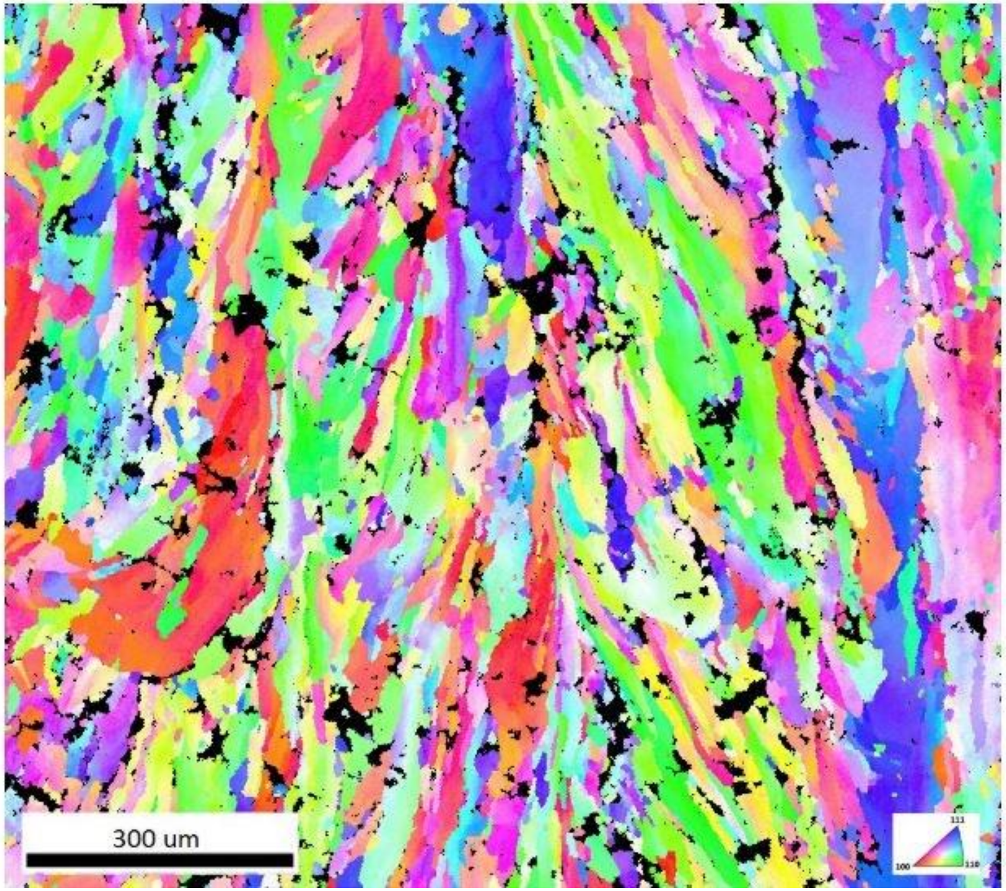
Recent Progress

- Characterization of grain size and morphology in A6061-RAM2 and A6061-RAM10 alloys from in-situ experiments at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL)
- Initial characterization of particle species formed in as-built A6061-RAM10
- NSF sponsored internship at Elementum 3D

Metrics

Description	% Complete	Status
1. Literature review	70%	●
2. Investigation of RAM (reactive additive manufacturing) on grain refinement mechanisms	10%	●
3. Correlation of measured and modeled solidification conditions to microstructural features and grain refinement	60%	●
4. Effect of inoculants and unreacted particles on post-processing heat treatment	20%	●

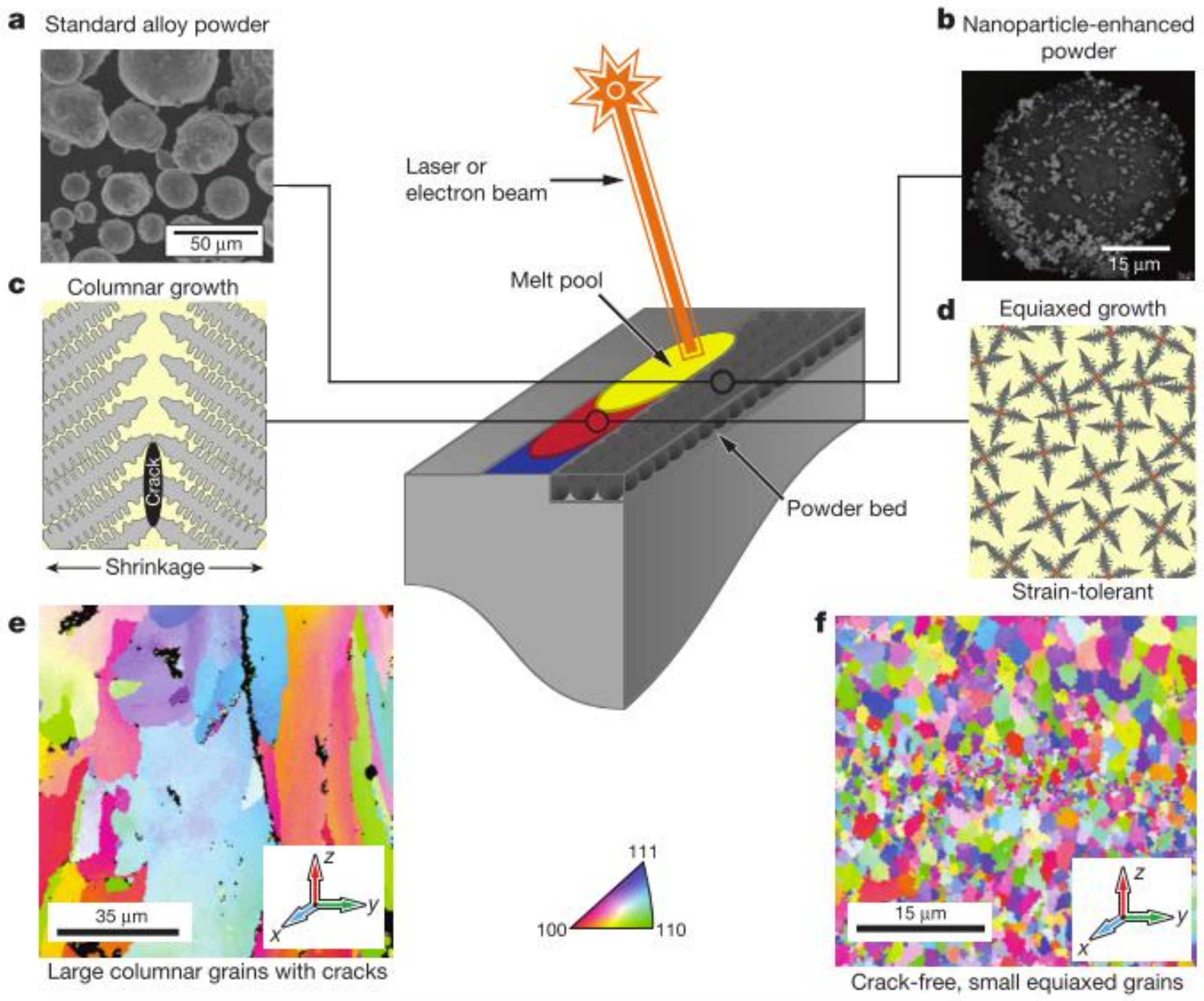
Industrial Relevance



Inverse pole figure of 3D-printed stock 7075, build direction is vertical to the page. Taken from J. H. Martin et al. *Nature,* 549 (2017) 365-369.

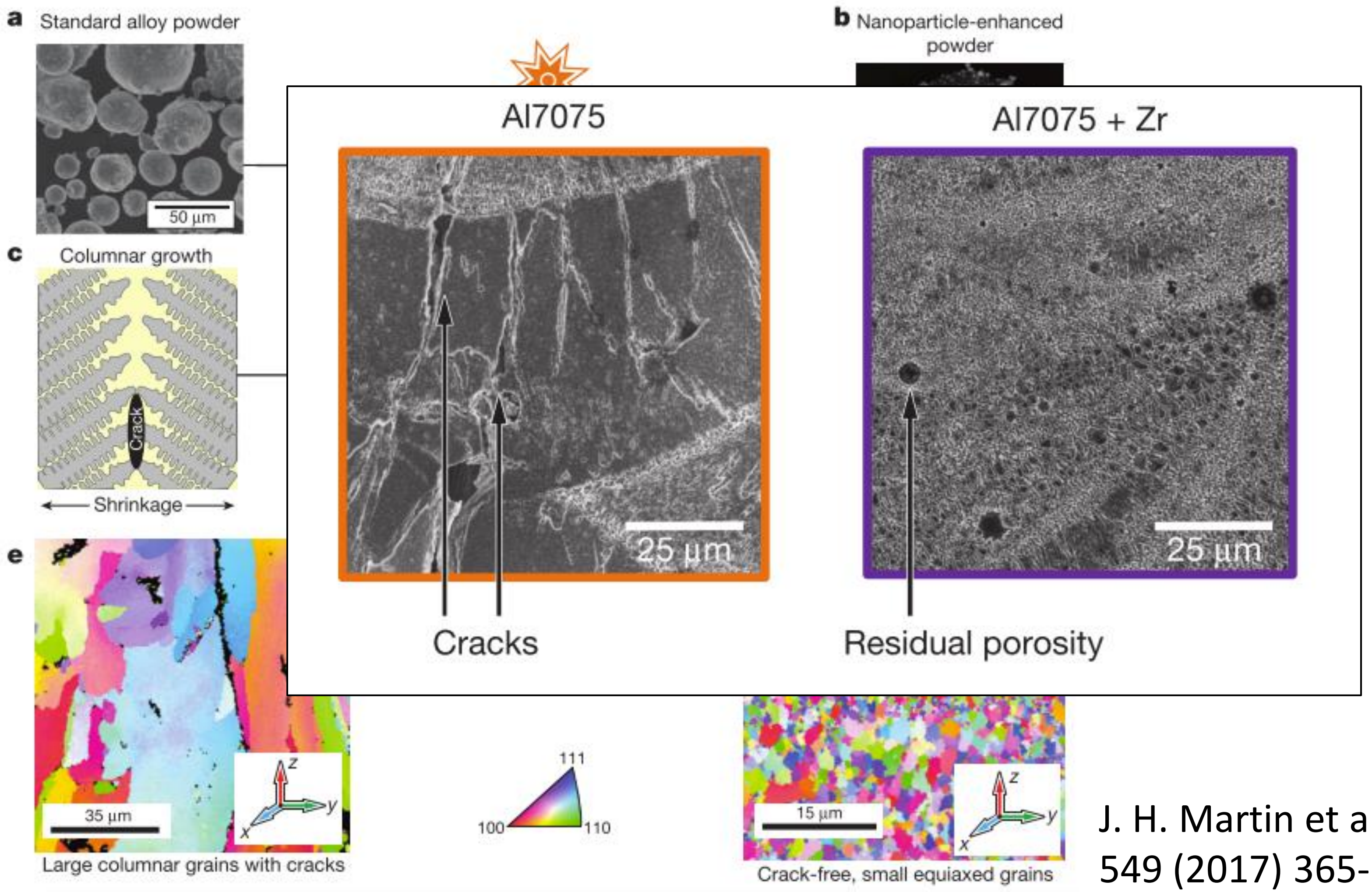
- Aluminum alloys currently used in AM are limited, and have mostly been casting alloys (e.g. AlSi10Mg)
- Under AM conditions, many aluminum alloys tend to form columnar grains, and are subject to solidification cracking
- These results imply a need for alloys designed specifically for AM

Grain Size Control via Innoculants in AM Alloy Powders



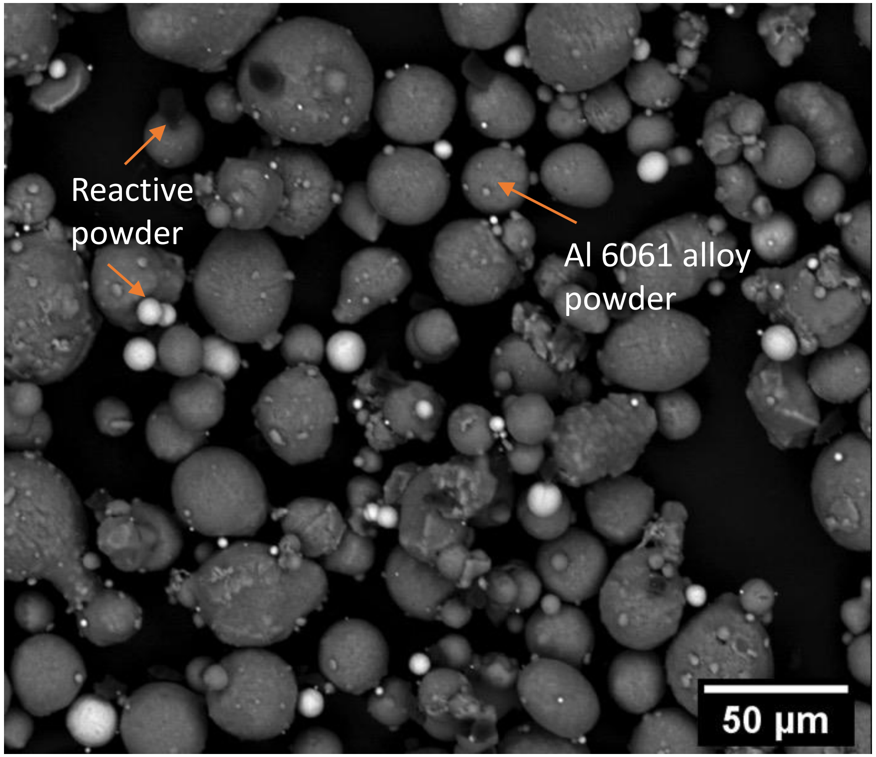
J. H. Martin et al. *Nature,* 549 (2017) 365-369.

Grain Size Control via Innoculants in AM Alloy Powders

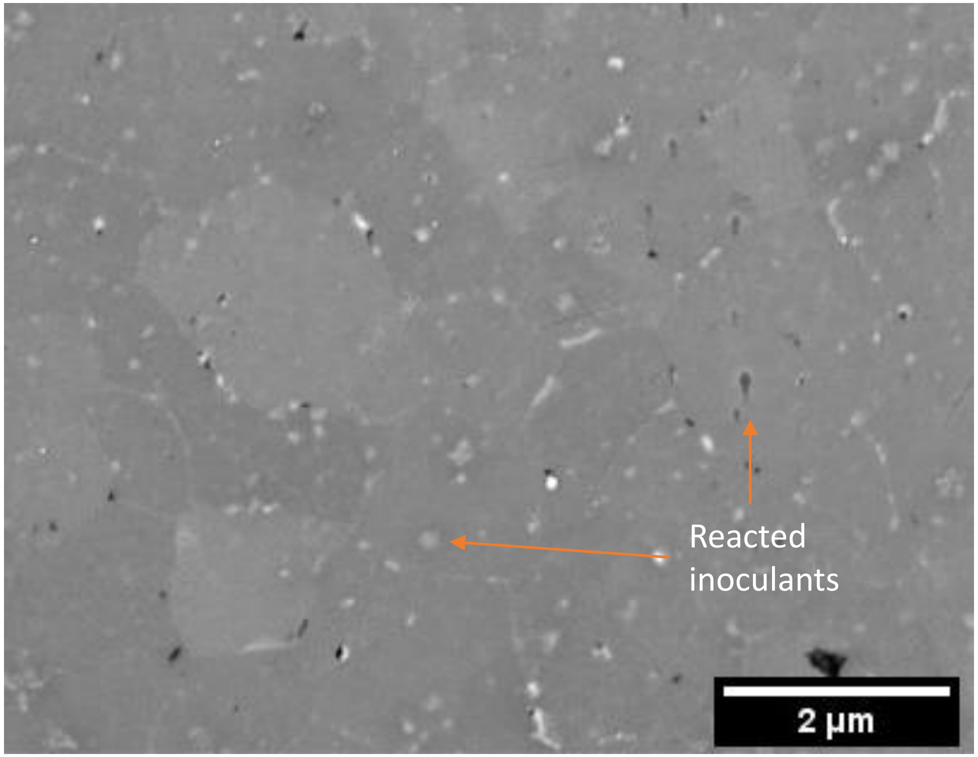


J. H. Martin et al. *Nature,* 549 (2017) 365-369.

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.

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

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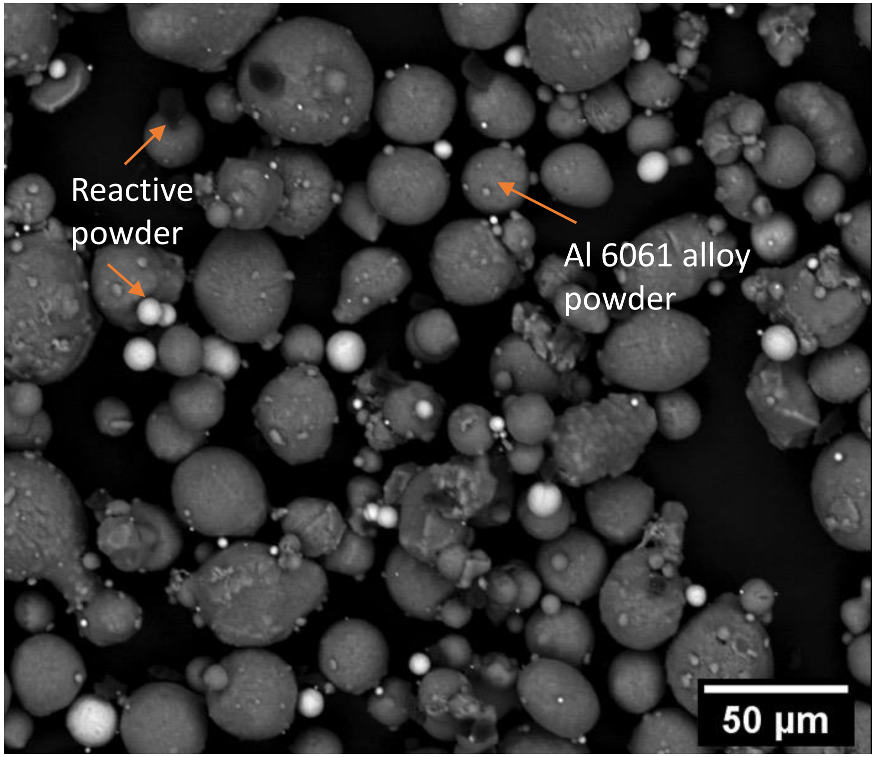
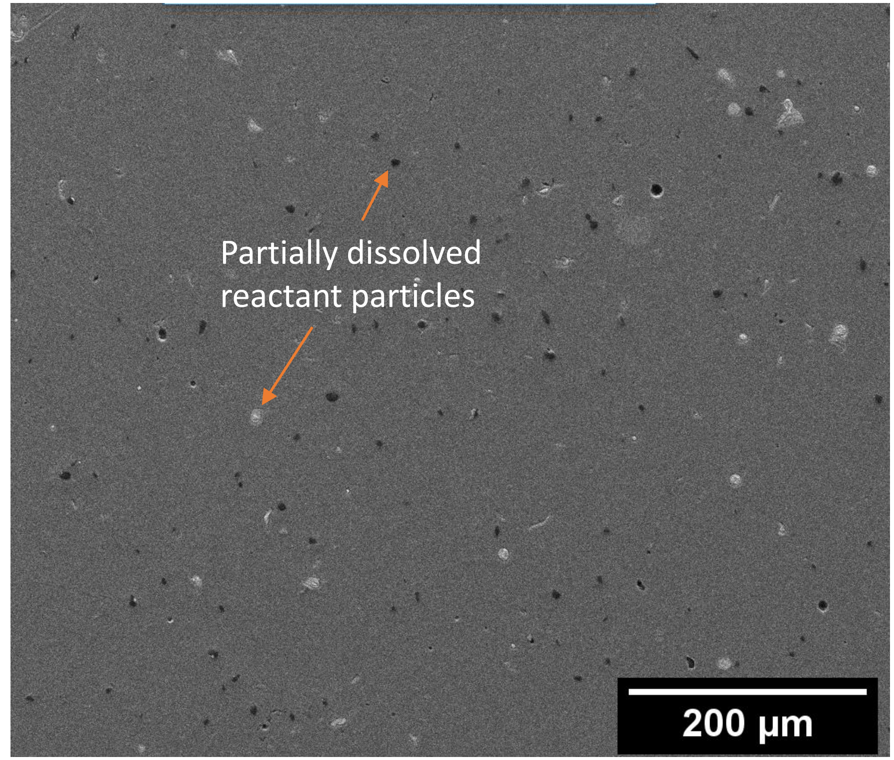


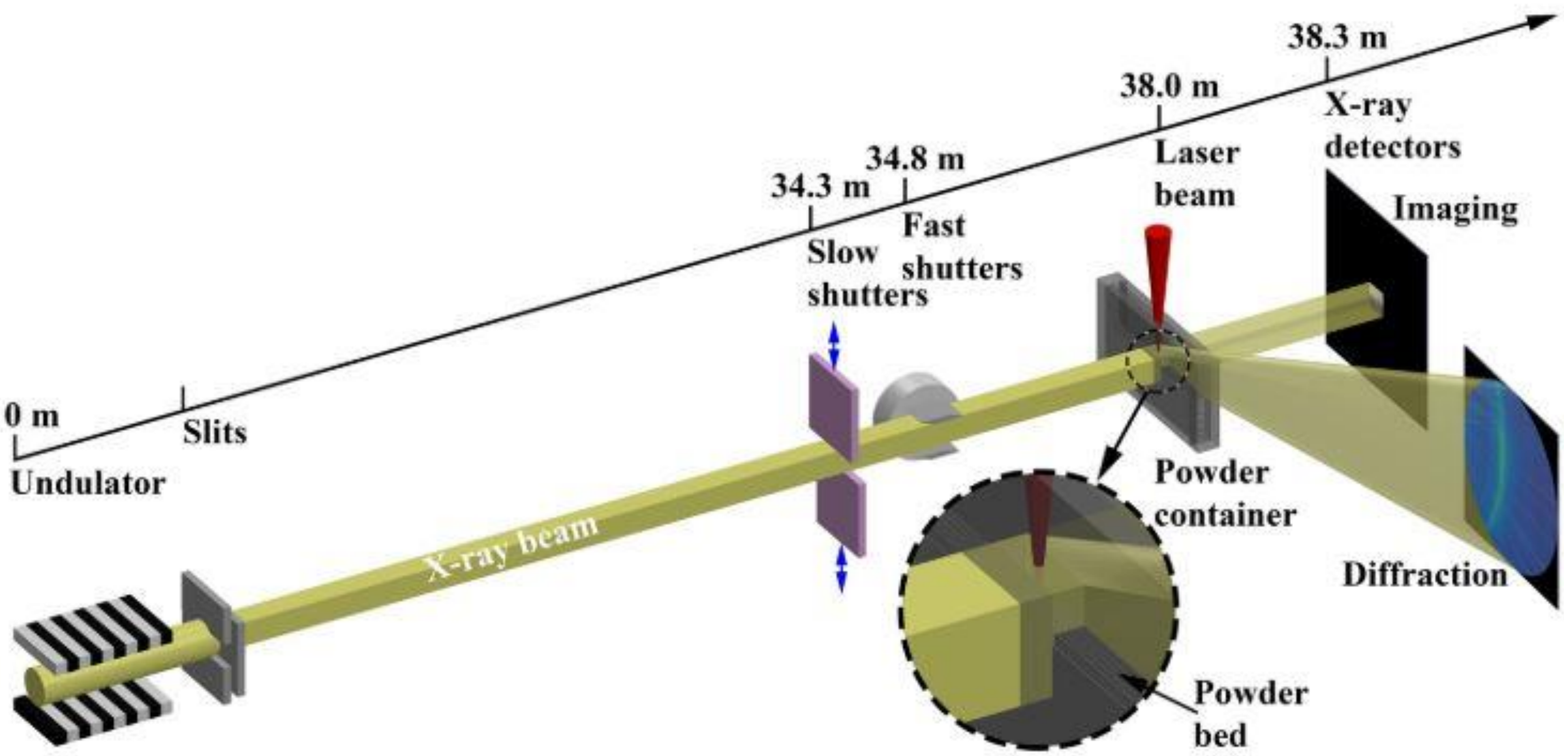
BSE SEM image of Al 6061 RAM 2% alloy powder



SEM image of as built Al 6061 RAM 2%

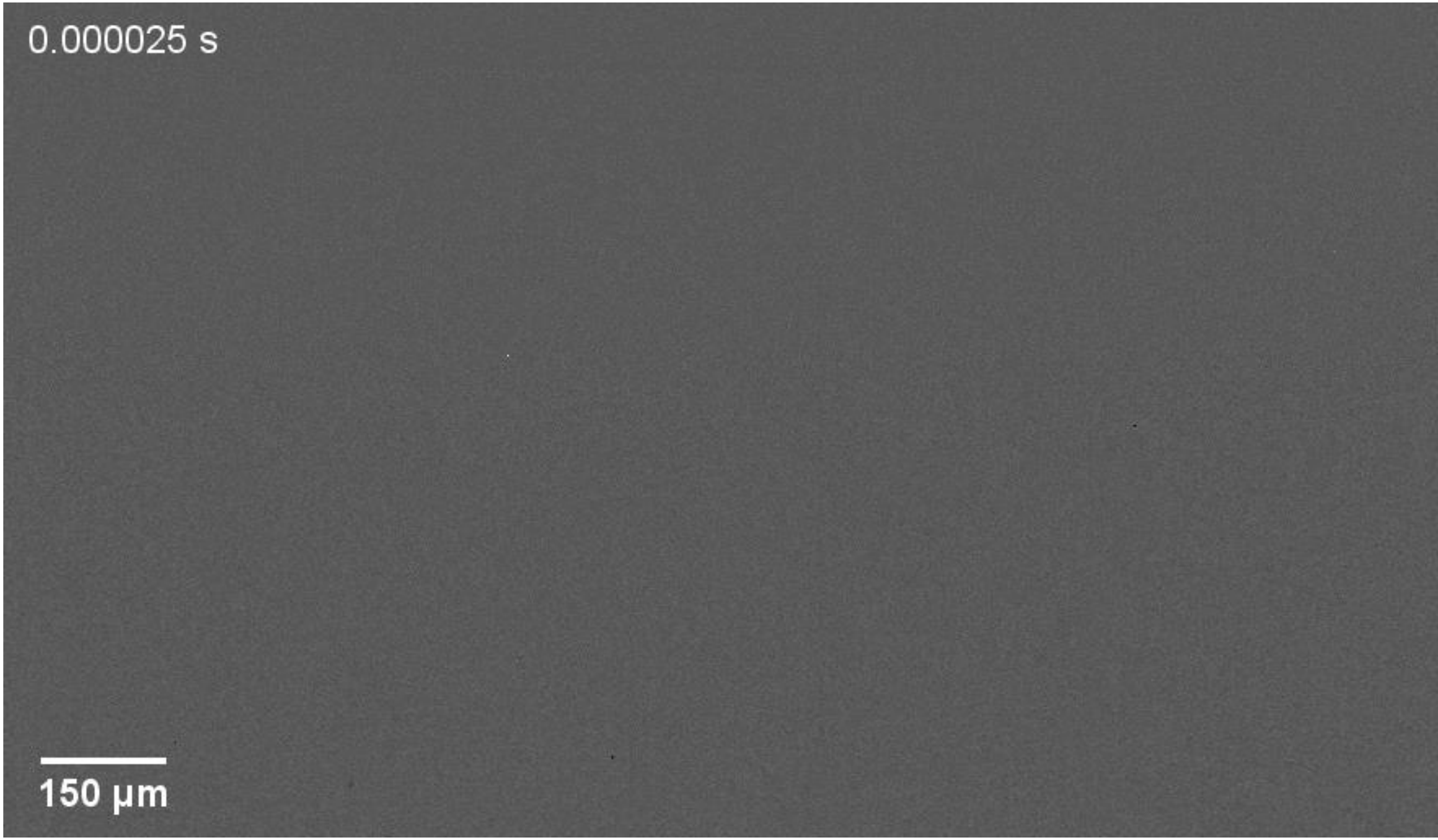
J. S. Neuchterlein & J. J. Iten, Reactive additive manufacturing, US Patent 20160271878 A1, priority 2015-03-17, published 2016-10-22.

Advanced Photon Source (APS) Additive Manufacturing Simulator Set-up



Schematic of AM simulator used for in-situ experiments at ANL.
Taken from: C. Zhao et al., *Scientific Reports,* 7 (2017) 1-11.

Image Processing: Tracking of S/L Interface



Animation of laser pass on 6061 wrought + 6061 powder, 416 W, 0.5 m/s

Acknowledgement to Gus Becker for image processing

Sample Sets & Laser Parameters Used for In-situ Experiments

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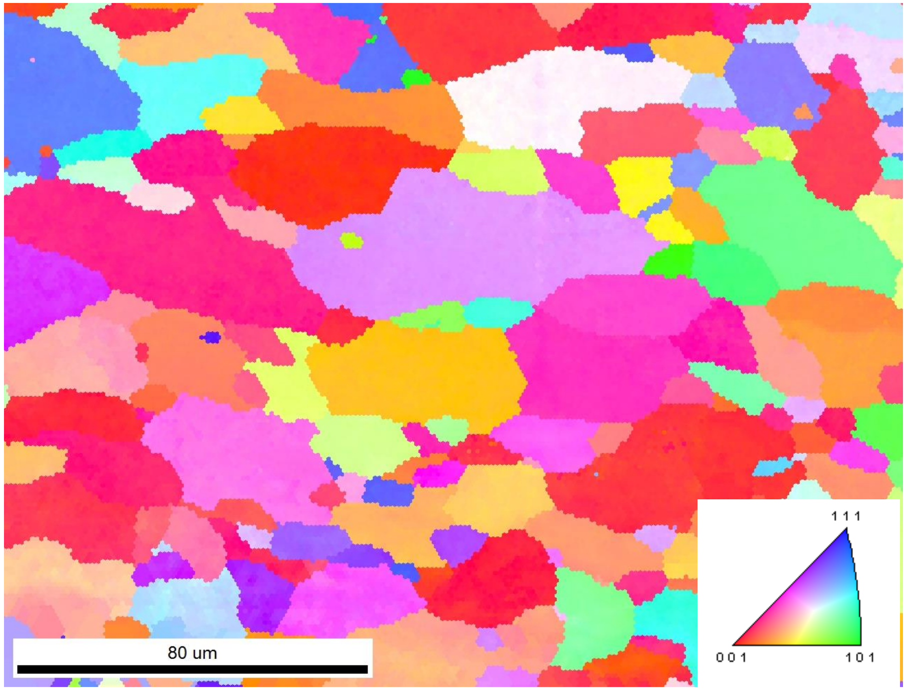
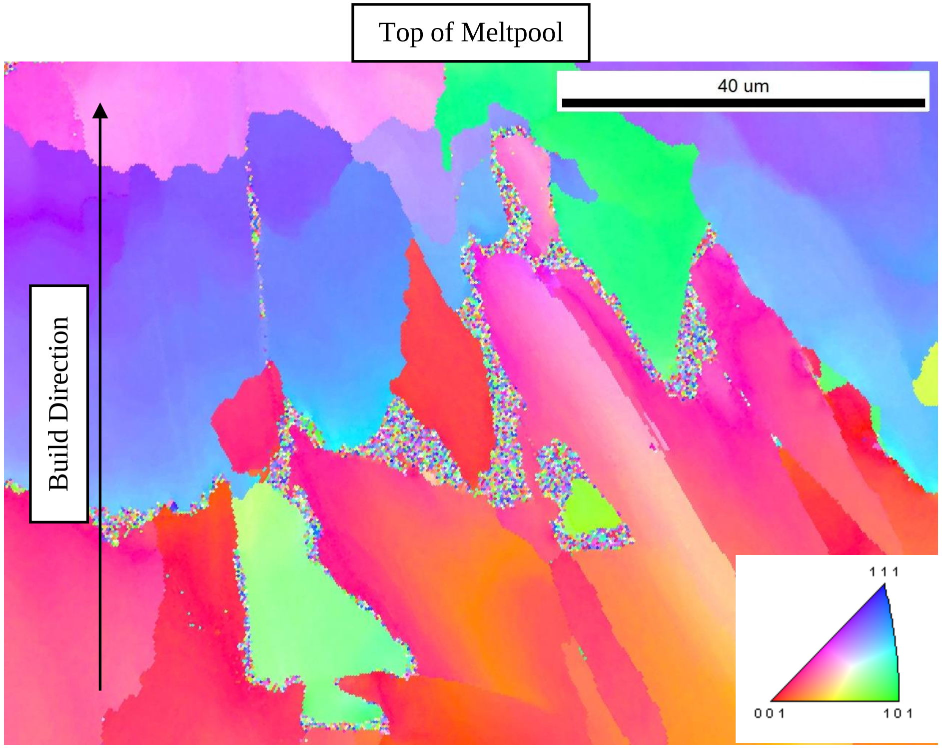
Base Plate	Powder
A6061-RAM2 Build	A6061-RAM2
A6061-RAM10 Build	A6061-RAM10
Wrought 6061	A6061-RAM10

Sample Number	Power (W)	Speed (m/s)	Linear Energy Density (J/m)
1	311	0.5	622
2	397	1	397
3	397	1.5	265
4	540	1.5	360
5	540	2	270

Microstructure of Wrought 6061 Base Plate & 6061 LPBF Raster



Wrought 6061 base plate



From single raster on 6061 wrought base plate with a layer of 6061 powder (426 W, 0.5 m/s)

Microstructure & Observed Hot-Cracking of 6061 Raster

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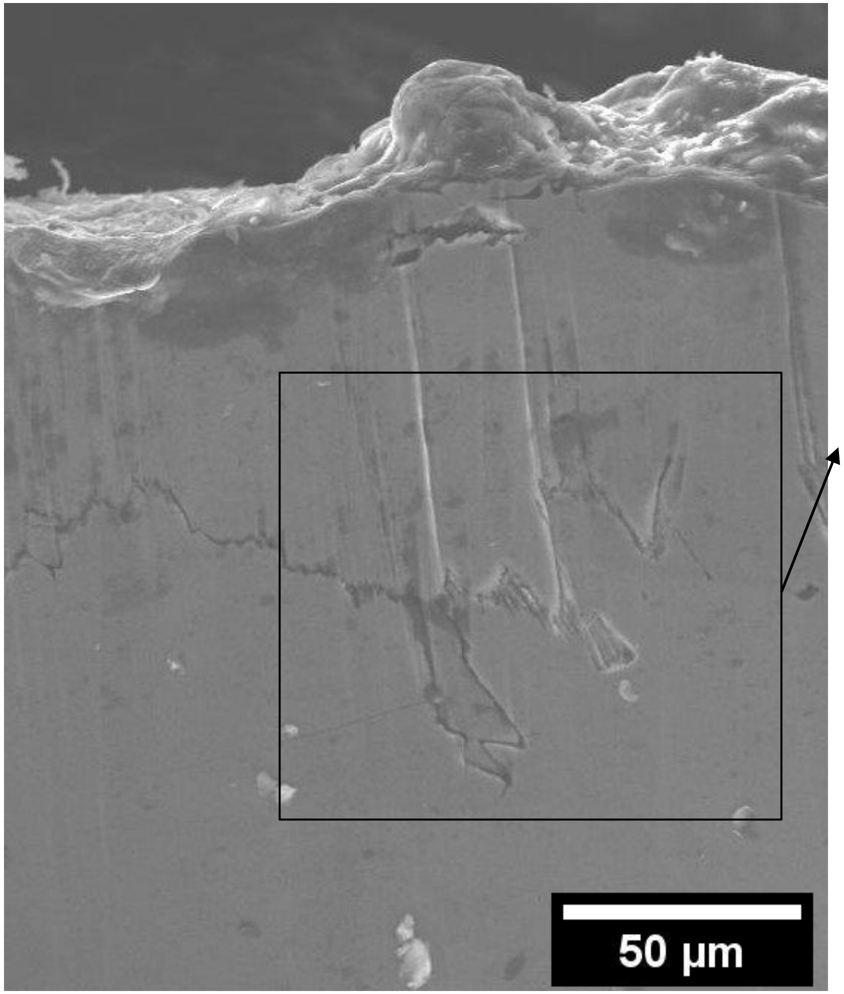
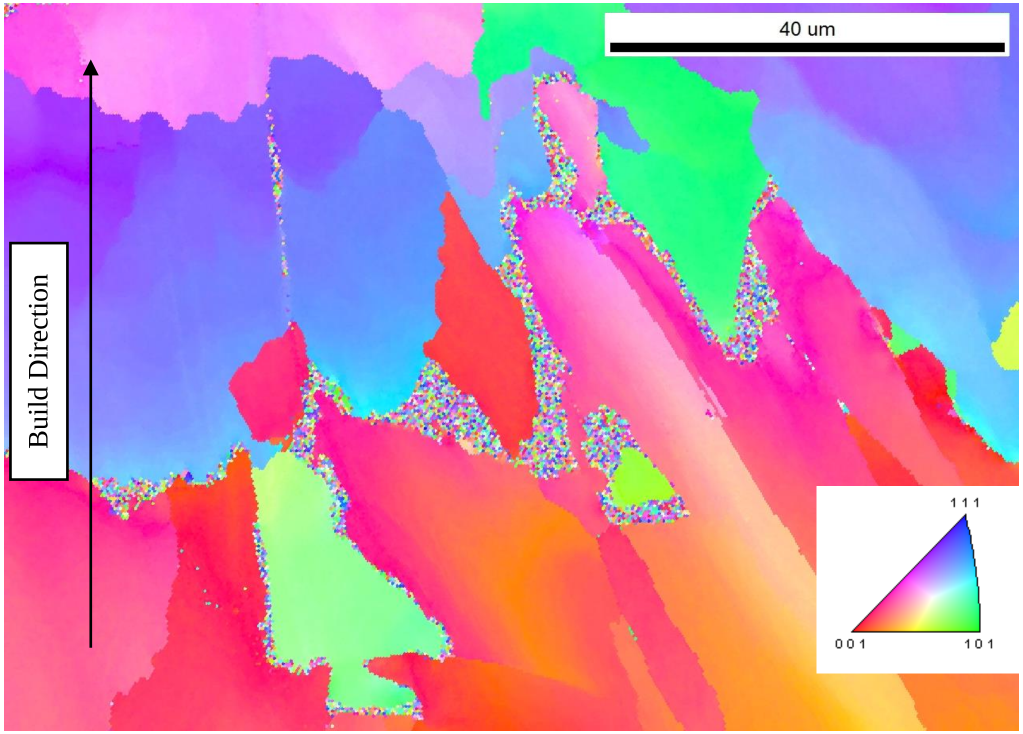


location of EBSD IPF on right, taken from middle of raster



From single raster on 6061 wrought base plate with a layer of 6061 powder (426 W, 0.5 m/s)

Changes in Grain Size with Laser Parameters (RAM2 Build/RAM2 Powder)

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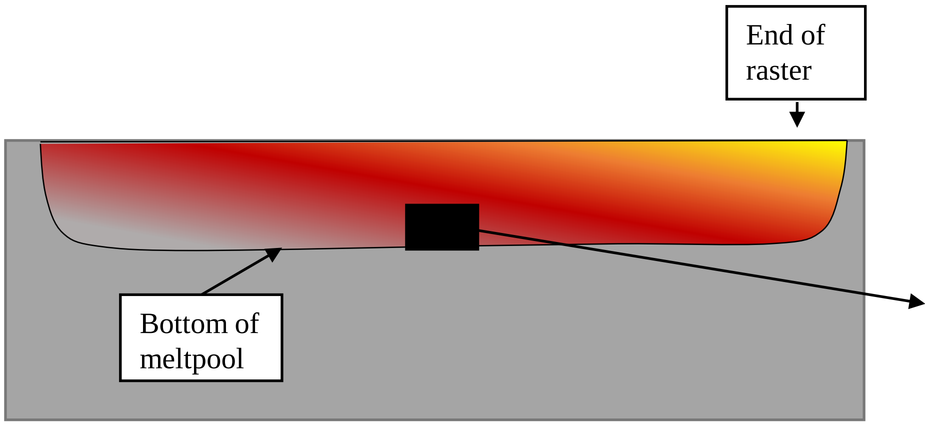
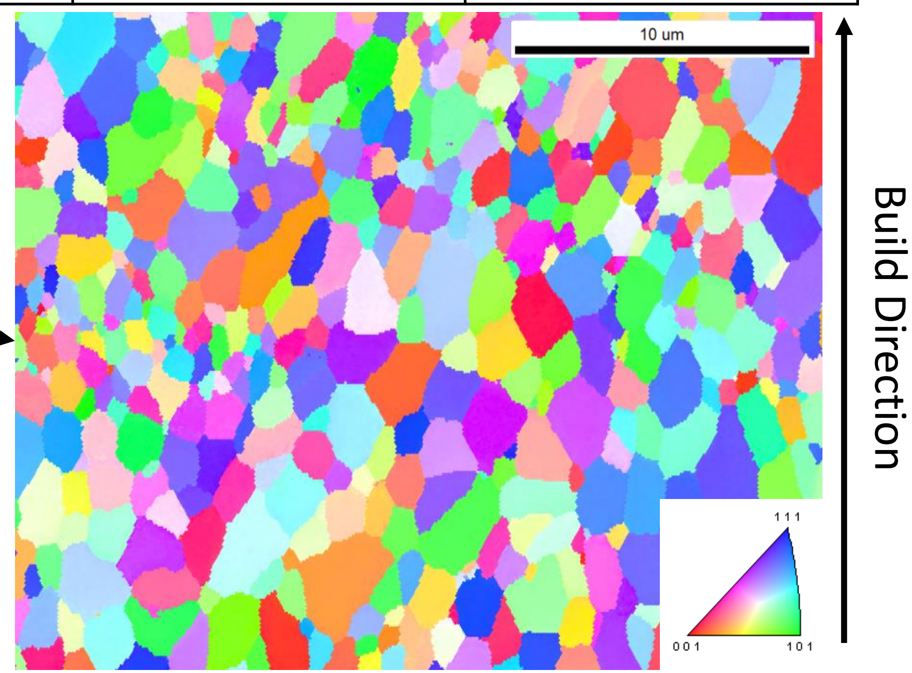
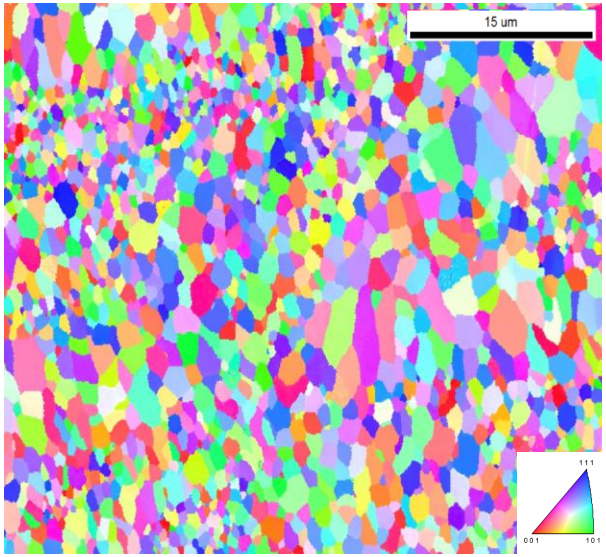
Sample Number	Power (W)	Speed (m/s)	Linear Energy Density (J/m)	Average Grain Area (μm²)	Average Grain Diameter (μm)
1	311	0.5	622	0.91	1.27
2	397	1	397	0.71	1.19
3	397	1.5	265	-	-
4	540	1.5	360	2.08	1.97
5	540	2	270	0.70	1.14



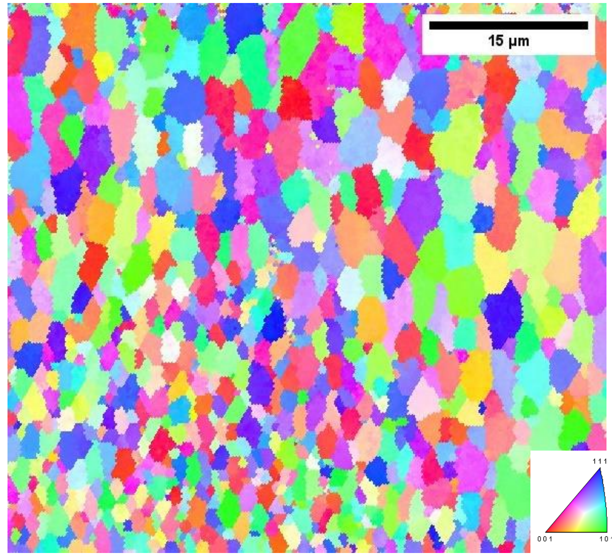


Above: Location of EBSD scan in single raster
Right: Sample 1 (see table) for an A6061-RAM2 Build with A6061-RAM2 powder

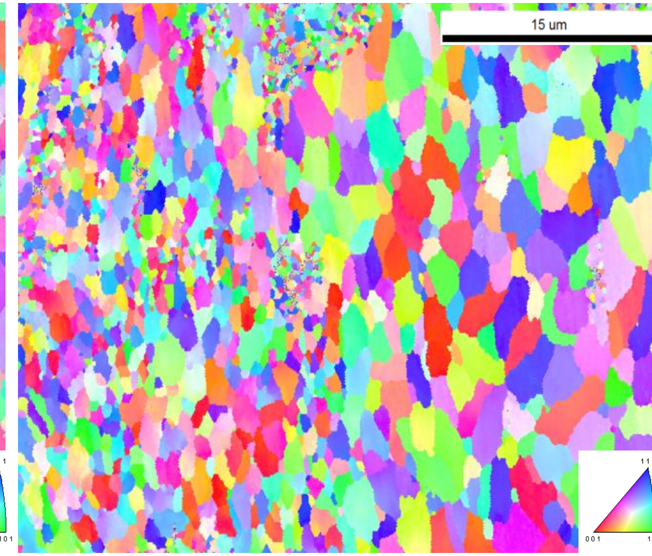
Changes in Grain Size with Laser Parameters (RAM2 Build/RAM2 Powder)



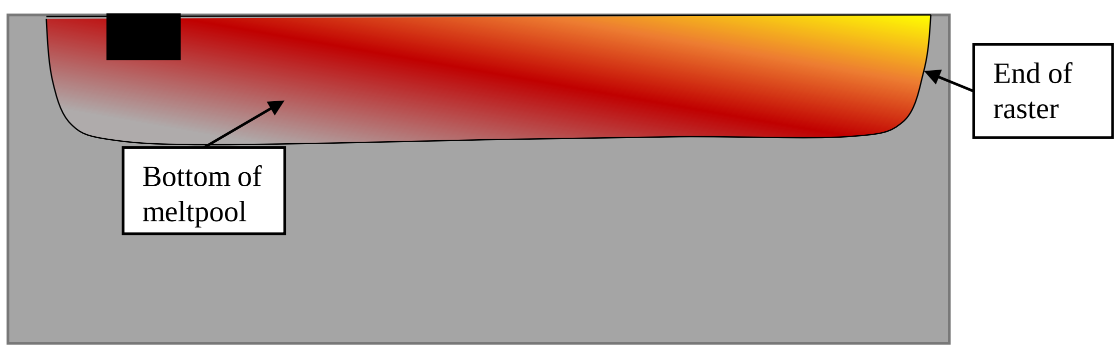
Sample 2 (397 W, 1 m/s)



Sample 4 (540 W, 1.5 m/s)



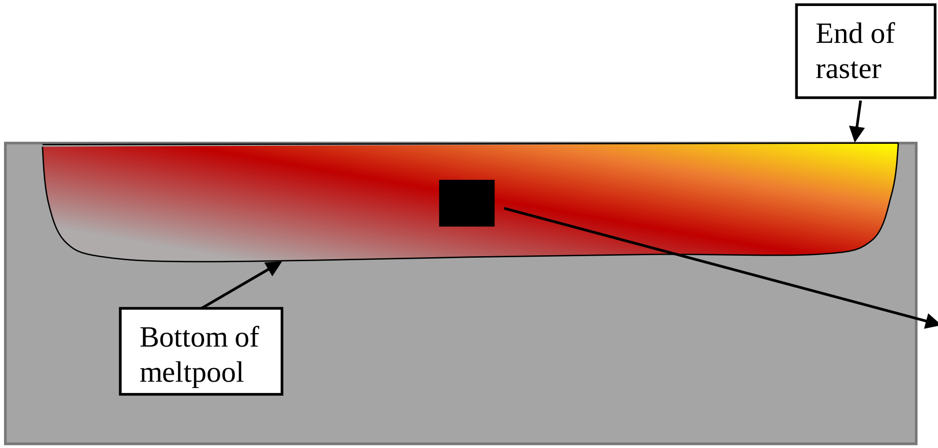
Sample 5 (540 W, 2 m/s)

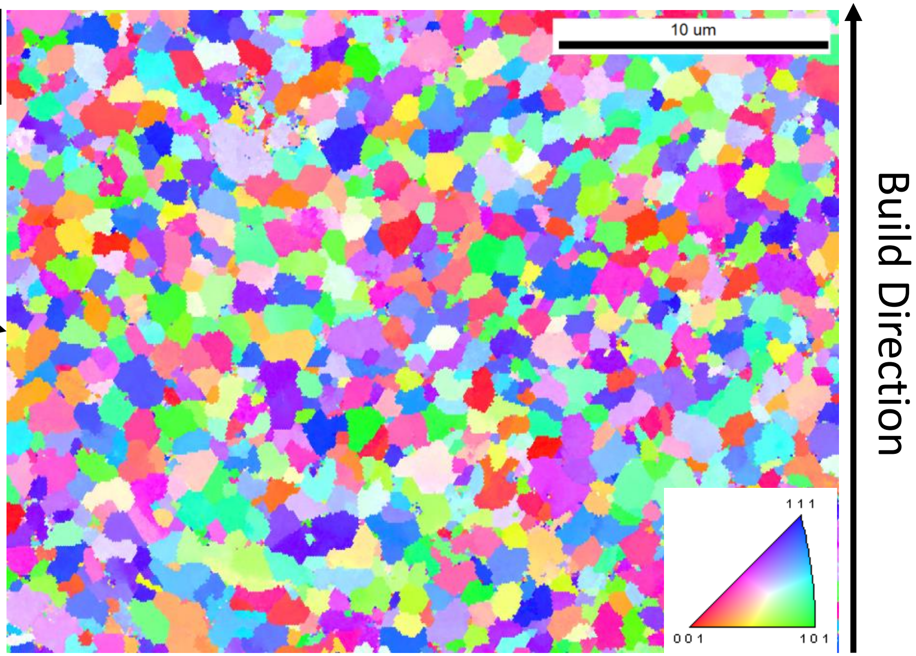


Schematic of single raster showing location for EBSD IPFs above

Changes in Grain Size with Laser Parameters (RAM10 Build/RAM10 Powder)

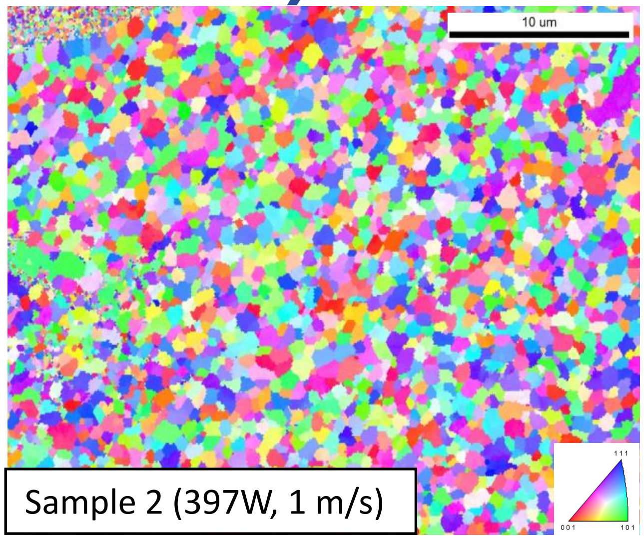
Sample Number	Power (W)	Speed (m/s)	Linear Energy Density (J/m)	Average Grain Area (μm^2)	Average Grain Diameter (µm)
1	311	0.5	622	0.49	0.88
2	397	1	397	0.53	0.95
3	397	1.5	265	0.27	0.73
4	540	1.5	360	0.38	0.83
5	540	2	270	0.19	0.61



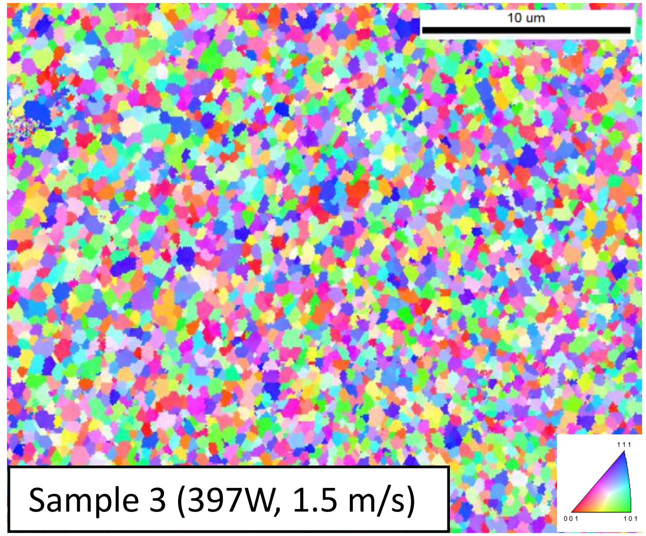


Above: Location of EBSD scan in single raster
Right: Sample 1 (see table) for an A6061-RAM10 Build with A6061-RAM10 powder

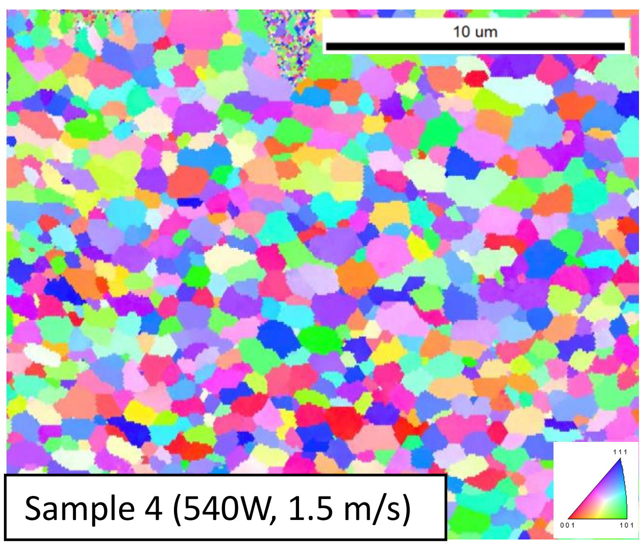
Changes in Grain Size with Laser Parameters (RAM10 Build/RAM10 Powder)



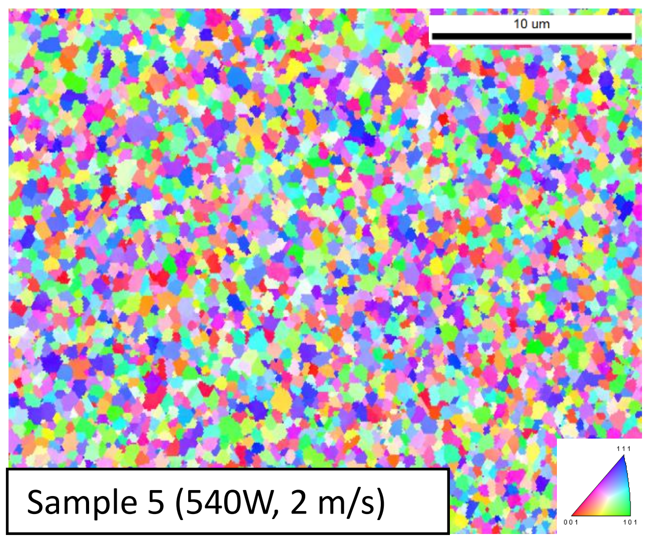
Sample 2 (397W, 1 m/s)



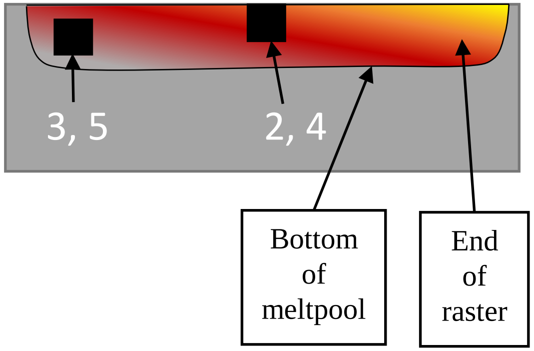
Sample 3 (397W, 1.5 m/s)



Sample 4 (540W, 1.5 m/s)

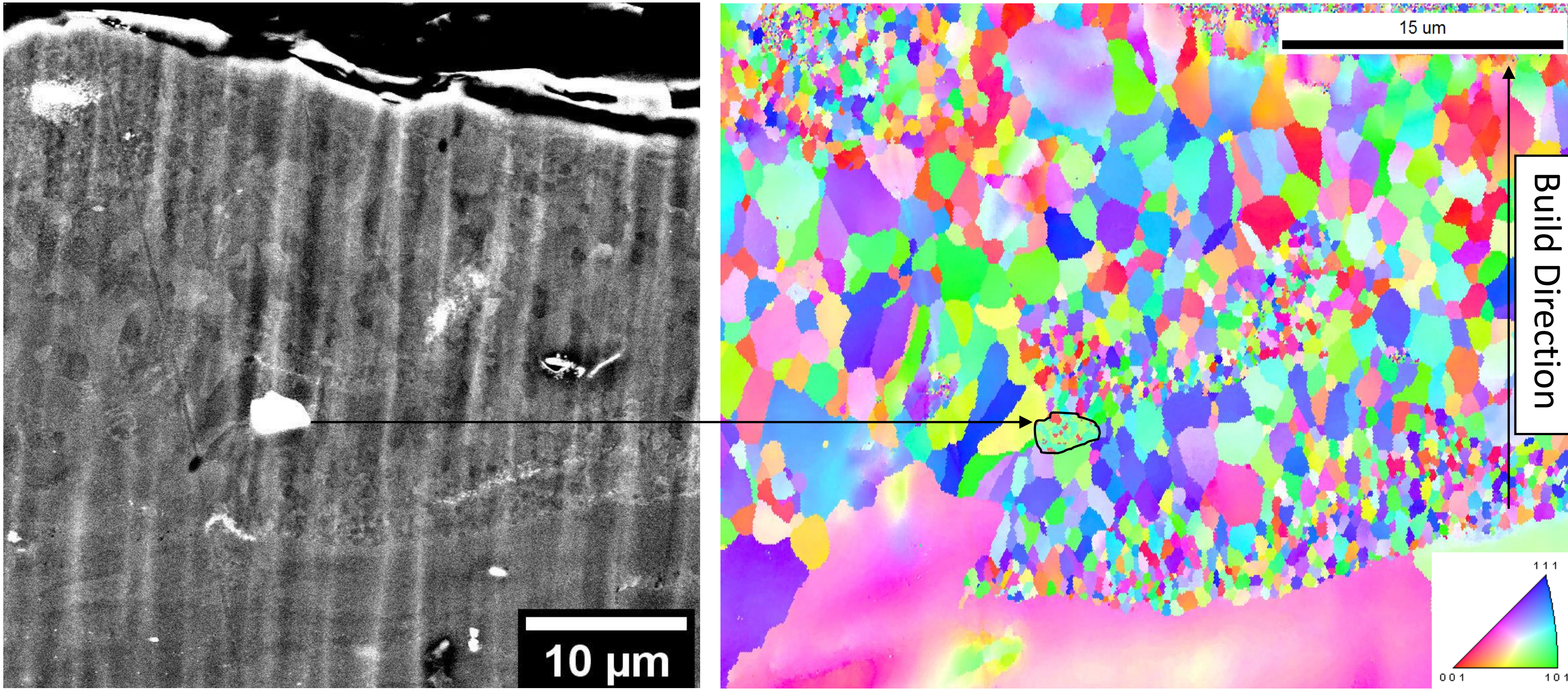


Sample 5 (540W, 2 m/s)



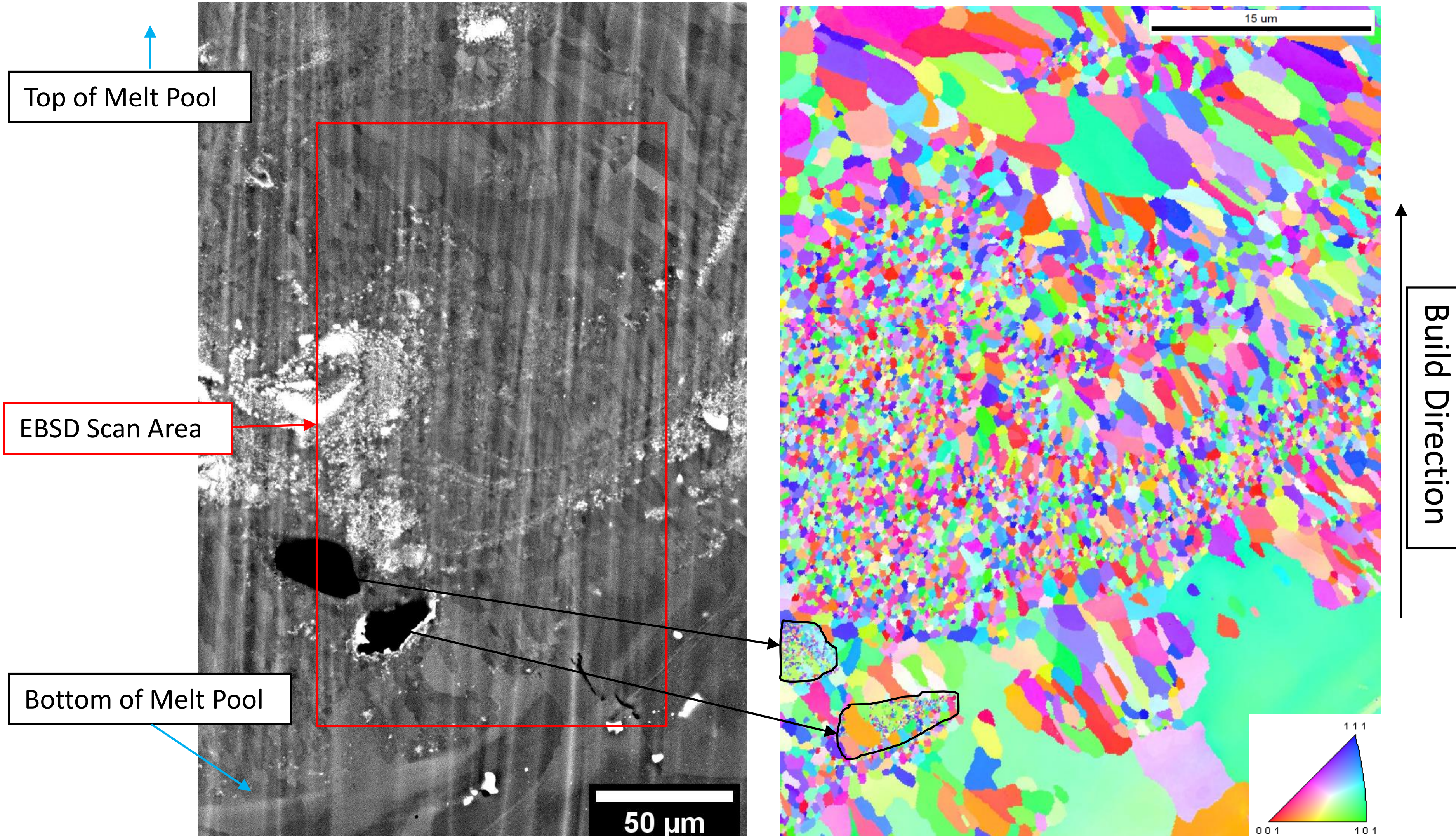
Above: Schematic showing location of EBSD IPFs for each sample number in the single raster

Microstructure of 6061 Wrought /A6061-RAM10 Powder Layer Samples



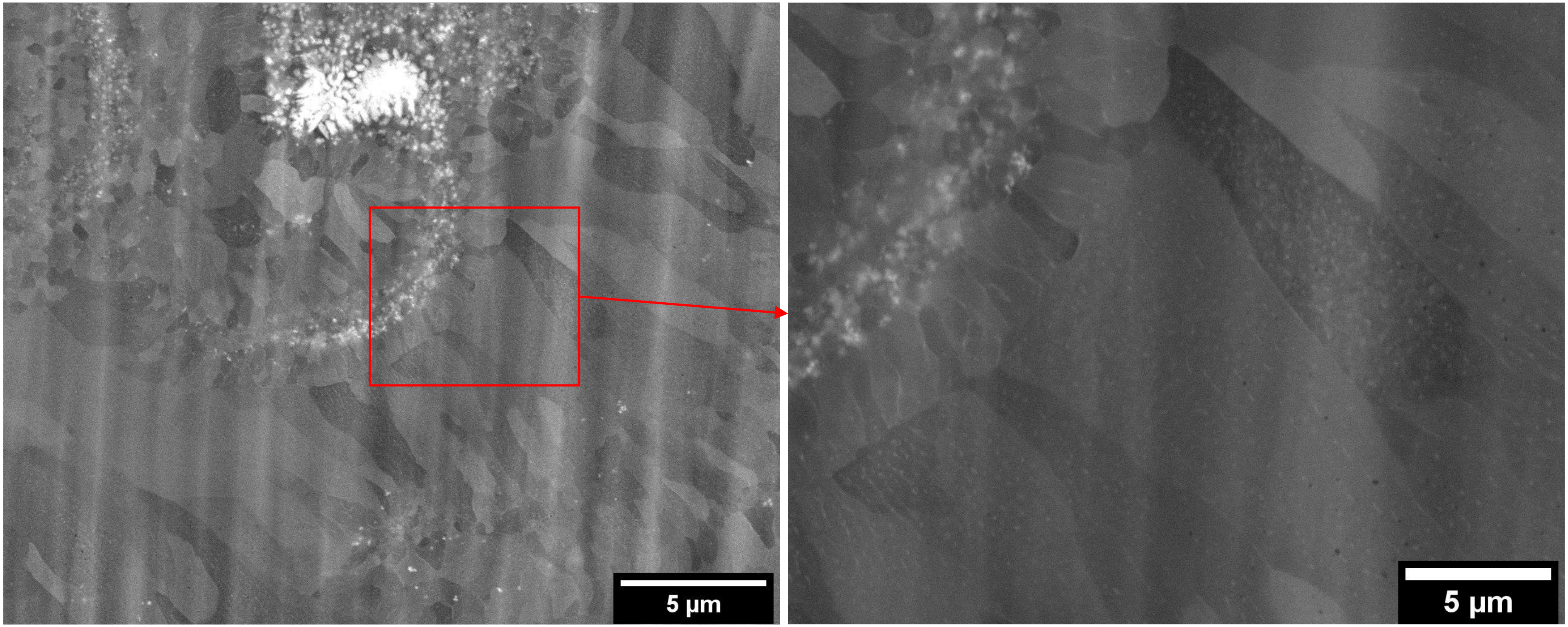
6061 wrought plate with A6061-RAM10 powder (497 W, 1.5 m/s) taken near end of raster

Microstructure of “Hybrid” (6061 Base/A6061-RAM10 Powder Layer) Samples



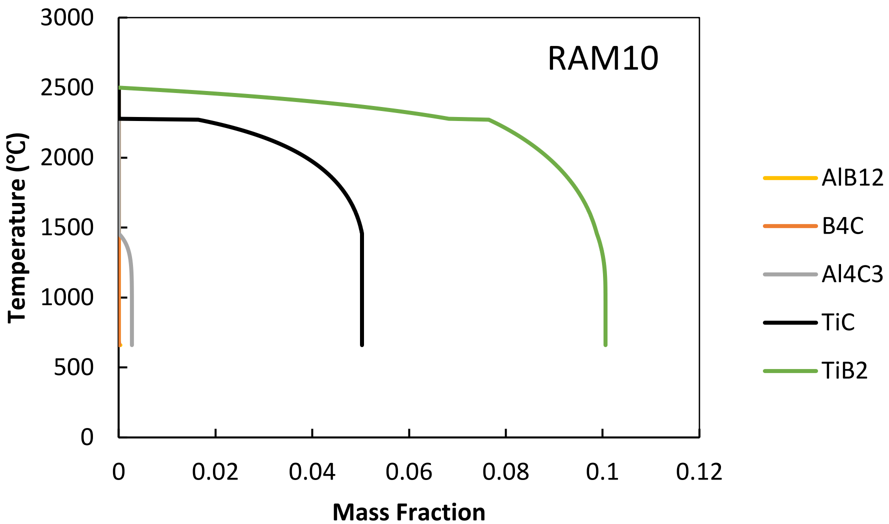
6061 wrought with A6061-RAM10 powder (540 W, 2 m/s) at termination of raster

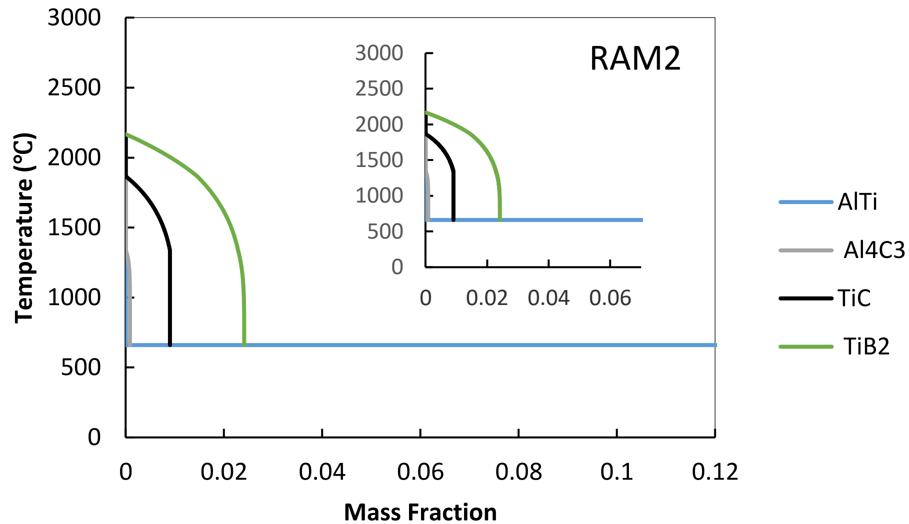
Microstructure of “Hybrid” (6061 Base/A6061-RAM10 Powder Layer) Samples

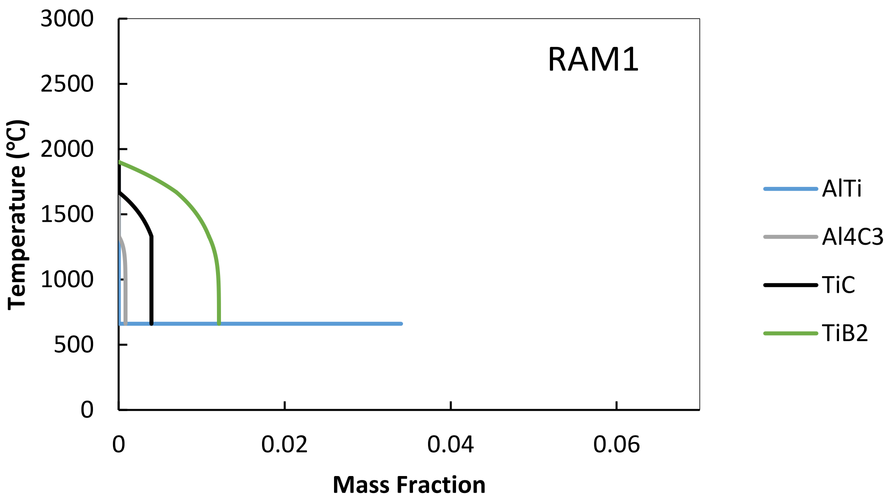


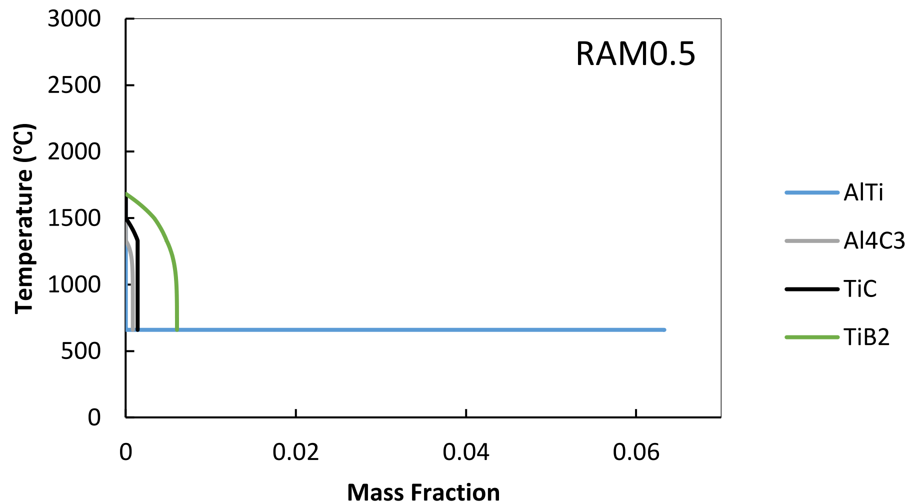
Wrought 6061 with A6061-RAM10powder (540 W, 2 m/s) at termination of raster

Thermo-Calc Predicted Phases in Alloys with Various Starting RAM Concentrations

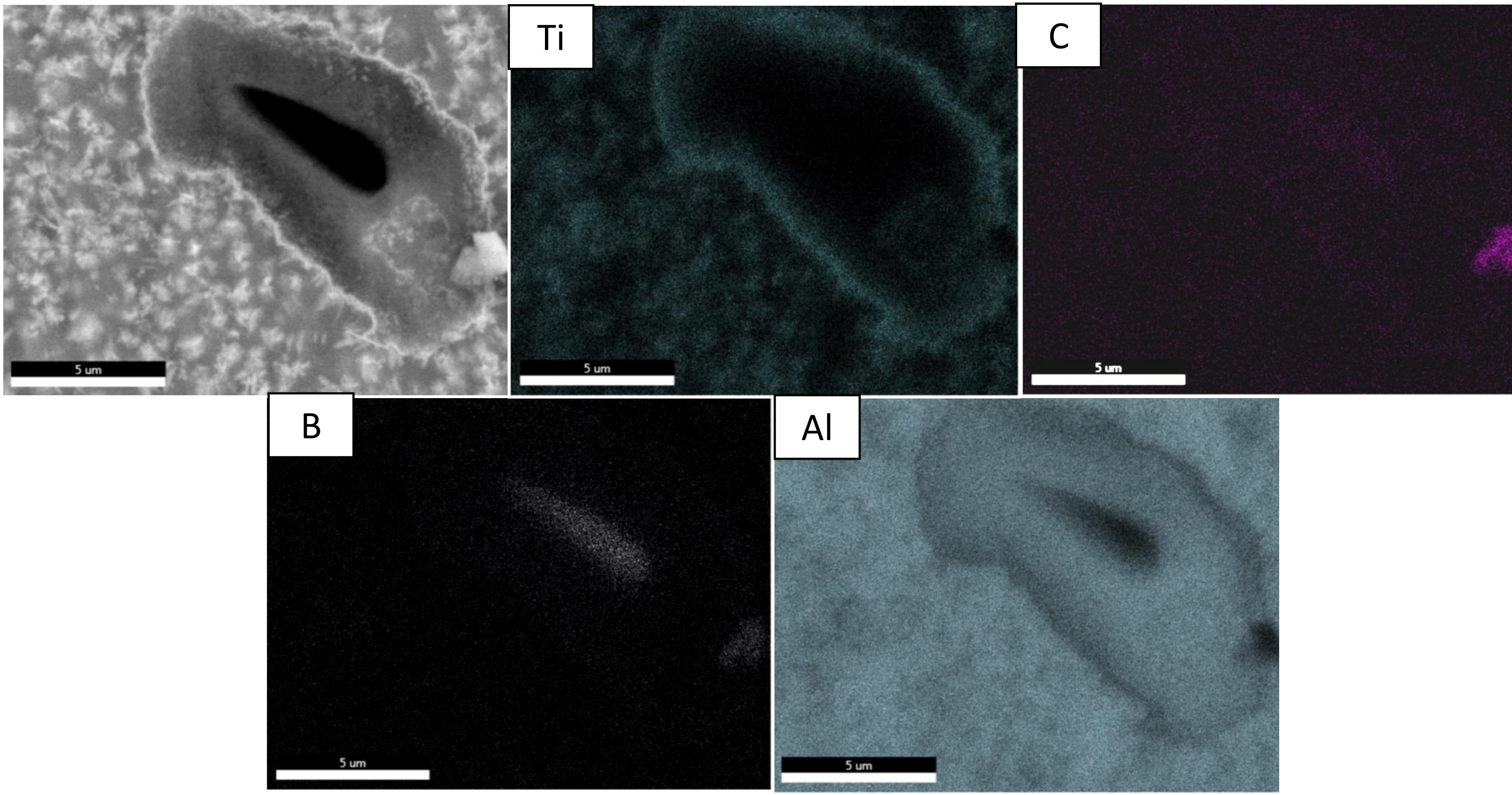






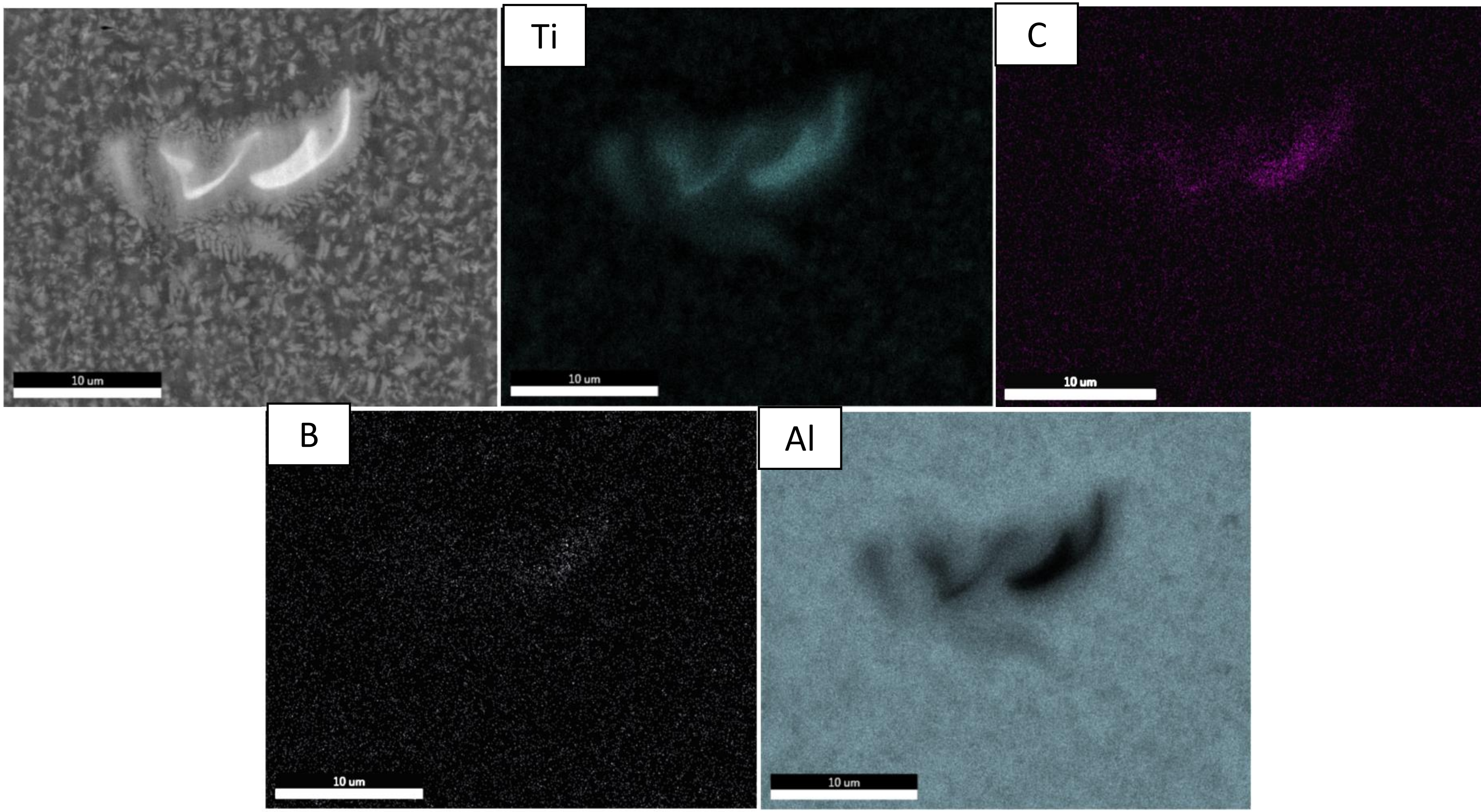


As-Built A6061-RAM10: B_4C Particle



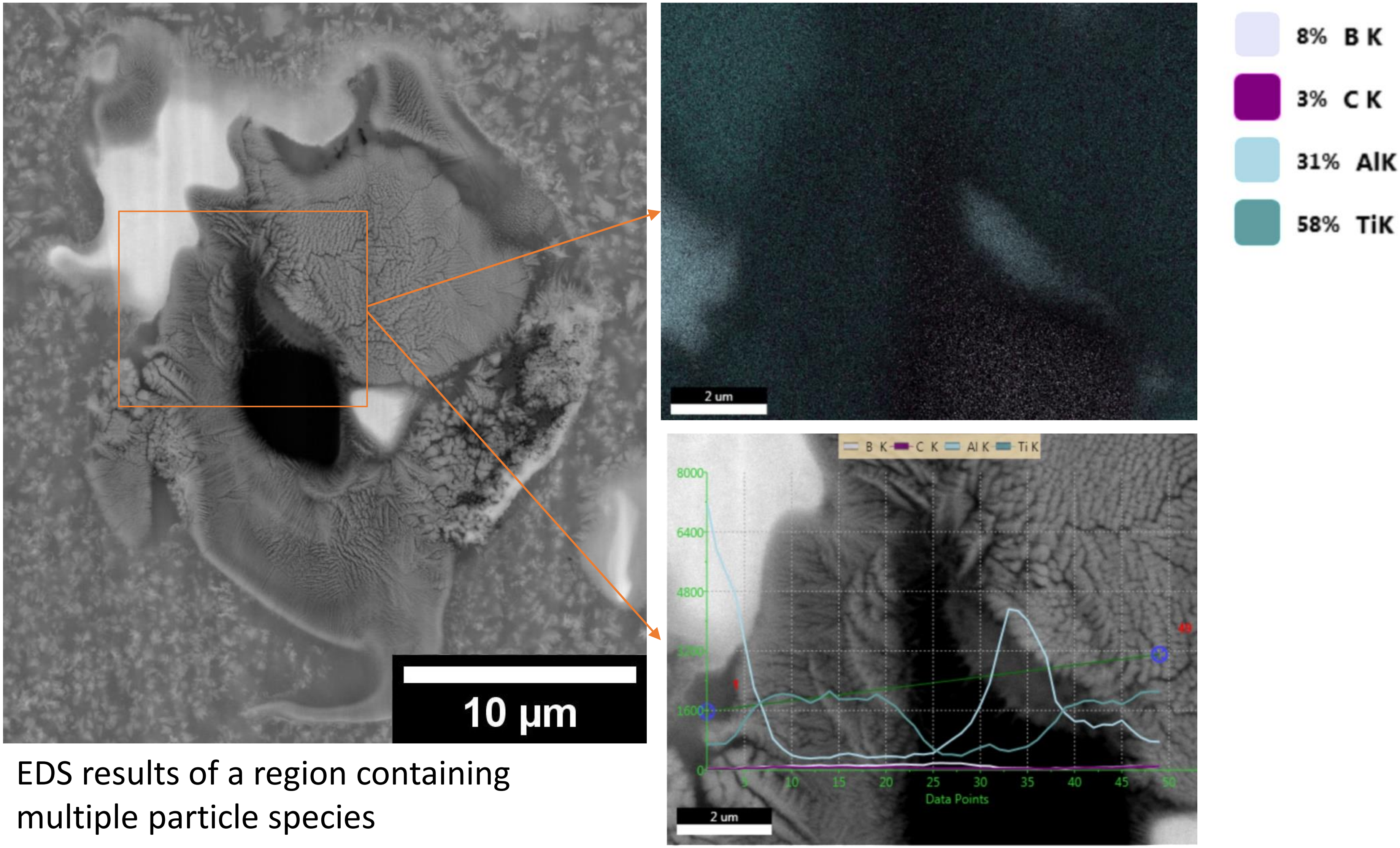
EDS results around B_4C (black) particle in A6061-RAM10 meltpool

As-Built A6061-RAM10: Ti Particle



EDS results around Ti (white) particle in A6061-RAM10 meltpool

As-Built A6061-RAM10: Diversity of Particle Species

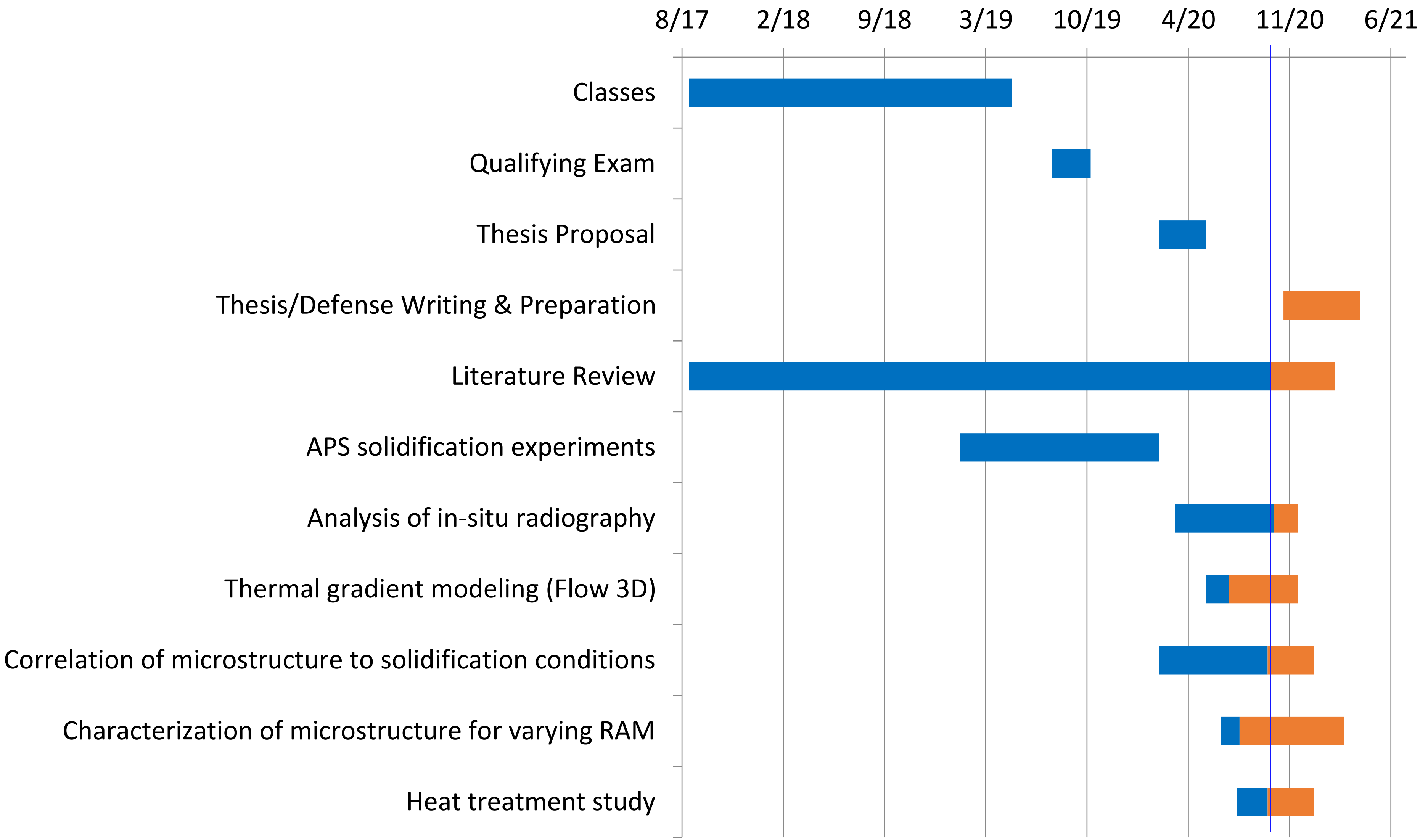


EDS results of a region containing multiple particle species

Conclusions & Future Work

- A6061-RAM alloys show similar grain sizes and morphologies for all solidification conditions investigated
  - investigated further in remaining conditions/samples from APS experiments
- Unidentified Al and Ti rich particle species forming in A6061-RAM10
  - EDS provides insight into particle species, further work with other techniques (TEM, XRD, etc.) may be needed
  - Effect RAM particle contents (i.e. RAM10, RAM2, RAM1, and RAM0.5) on particle formation and final microstructure will also be investigated

Progress



8/17
2/18
9/18
3/19
10/19
4/20
11/20
6/21
Classes
Qualifying Exam
Thesis Proposal
Thesis/Defense Writing & Preparation
Literature Review
APS solidification experiments
Analysis of in-situ radiography
Thermal gradient modeling (Flow 3D)
Correlation of microstructure to solidification conditions
Characterization of microstructure for varying RAM
Heat treatment study

Challenges & Opportunities

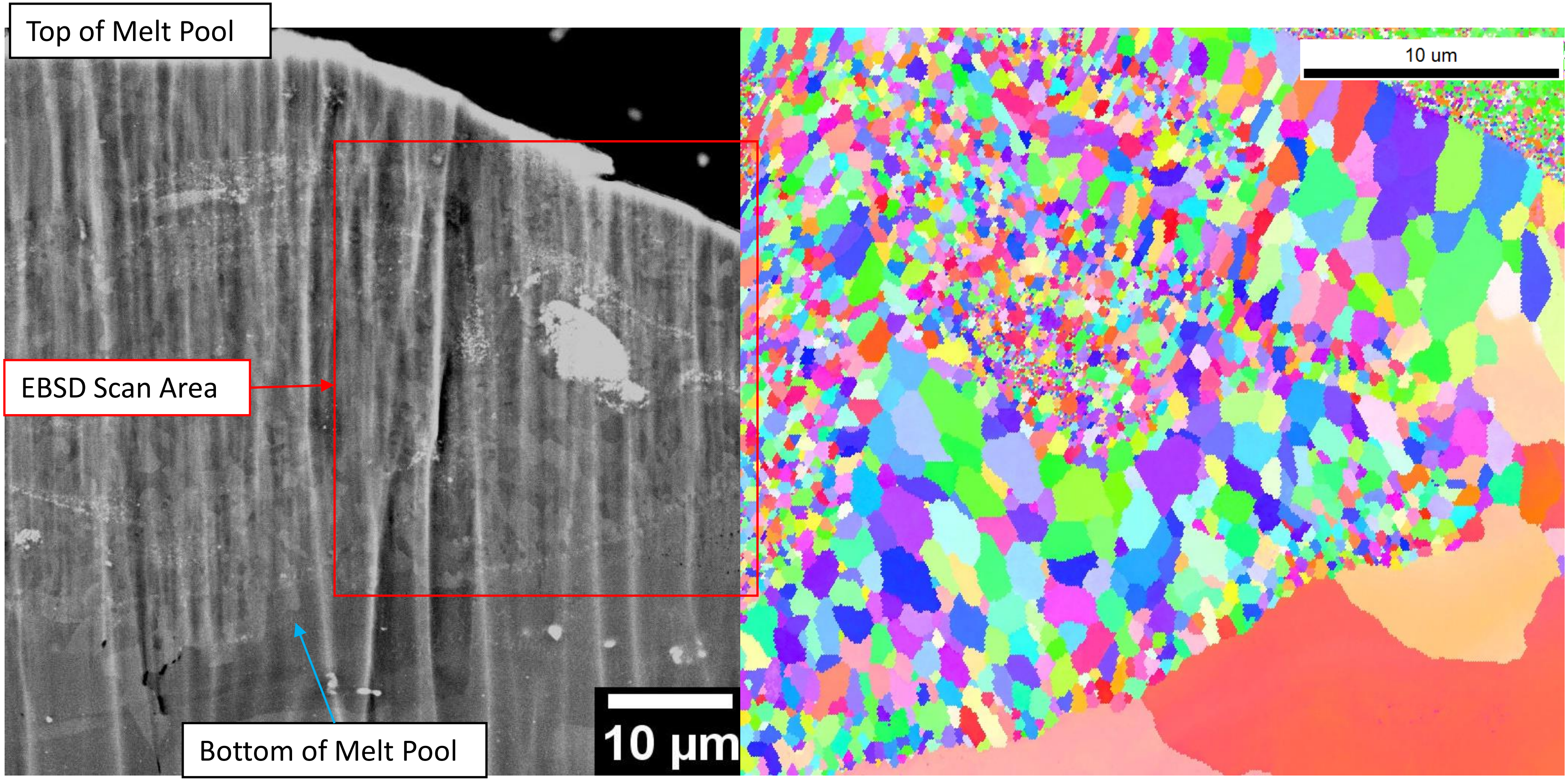
- Challenges
  - Effectively capturing all significant areas in microstructure of single raster scans
  - Identifying various particle species in A6061-RAM alloys with different RAM particle contents
- Opportunities
  - Identifying which particles contribute most to refinement by comparing regions of more or less refinement in 6061 wrought/A6061-RAM10 powder samples
  - Investigating effect of starting RAM particle content on final microstructure to inform design of in-situ inoculated alloys for AM of Al

Thank you!

Chloe Johnson

chloejohnson@mines.edu

Microstructure of “Hybrid” (6061 Base/A6061-RAM10 Powder Layer) Samples



Taken from middle of raster from melt pool performed using 497 W, 1.5 m/s

Changes in Grain Size with Laser Parameters (RAM2 Build/RAM2 Powder)

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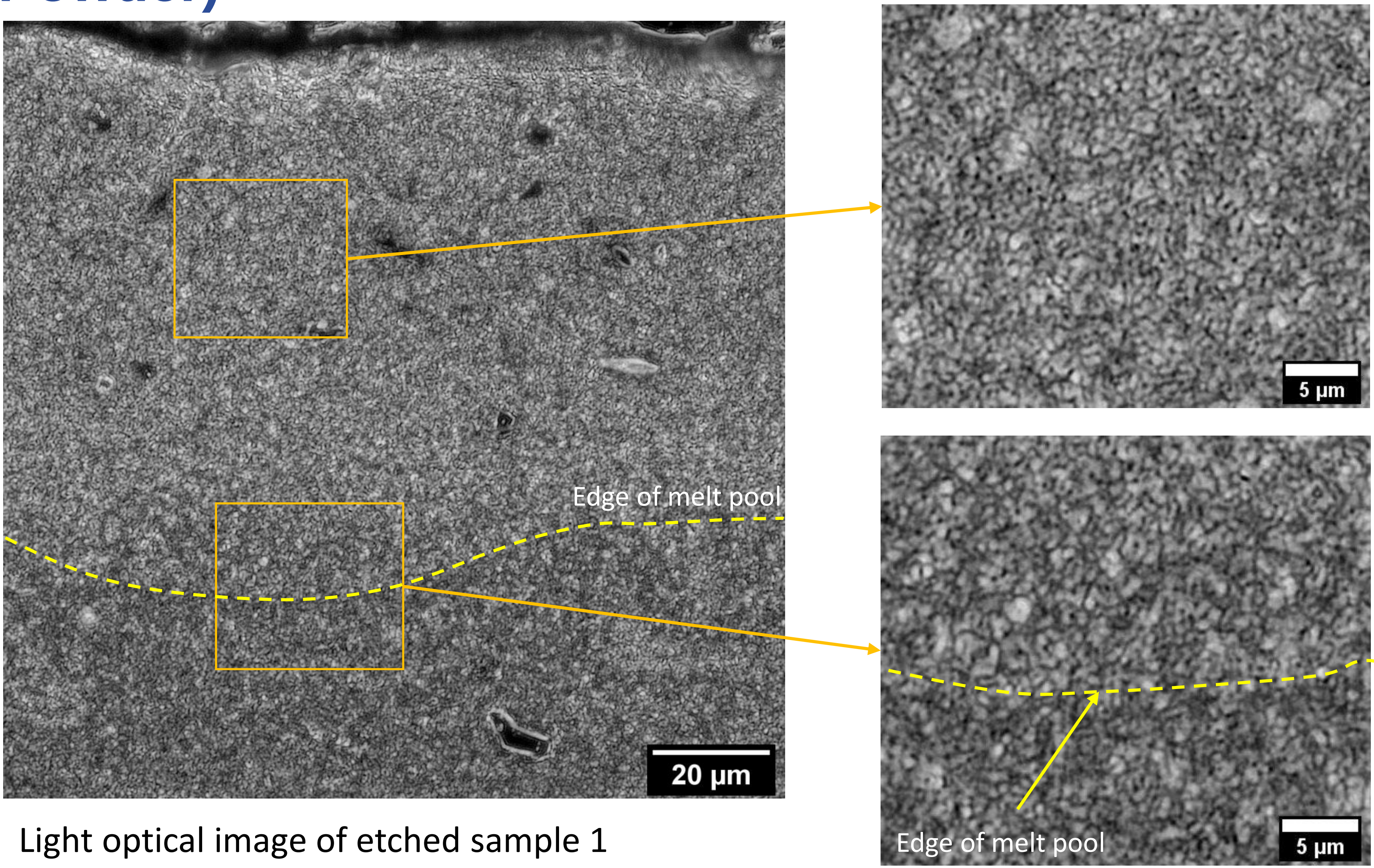

Light optical image of etched sample 1

Changes in Grain Size with Laser Parameters (RAM2 Build/RAM2 Powder)

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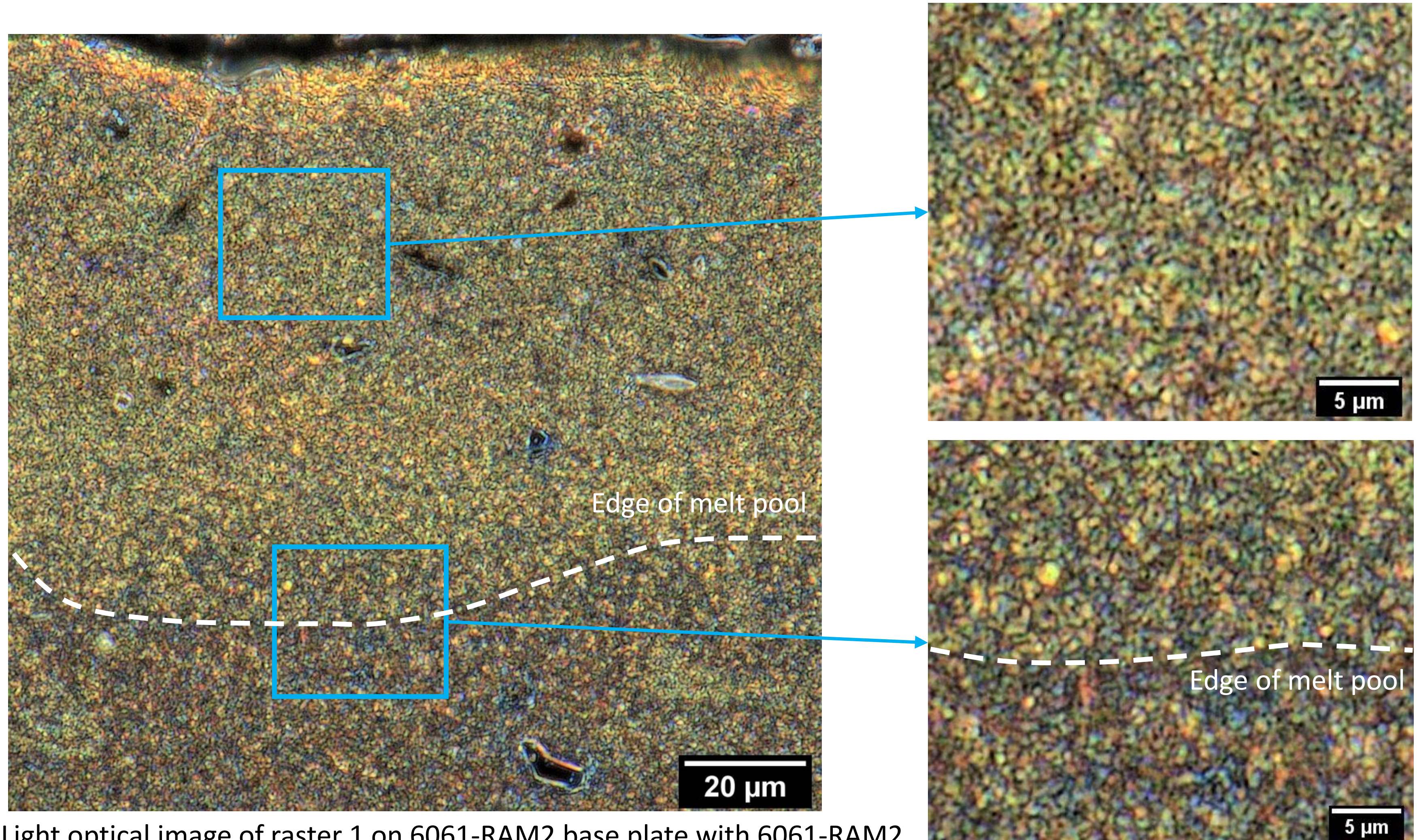

Light optical image of raster 1 on 6061-RAM2 base plate with 6061-RAM2 powder etched with two step etchant, including Weck's Reagent