CANFSA

Center for Advanced Non-Ferrous Structural Alloys
An Industry/University Cooperative Research Center

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

Spring Meeting

April 12, 2022

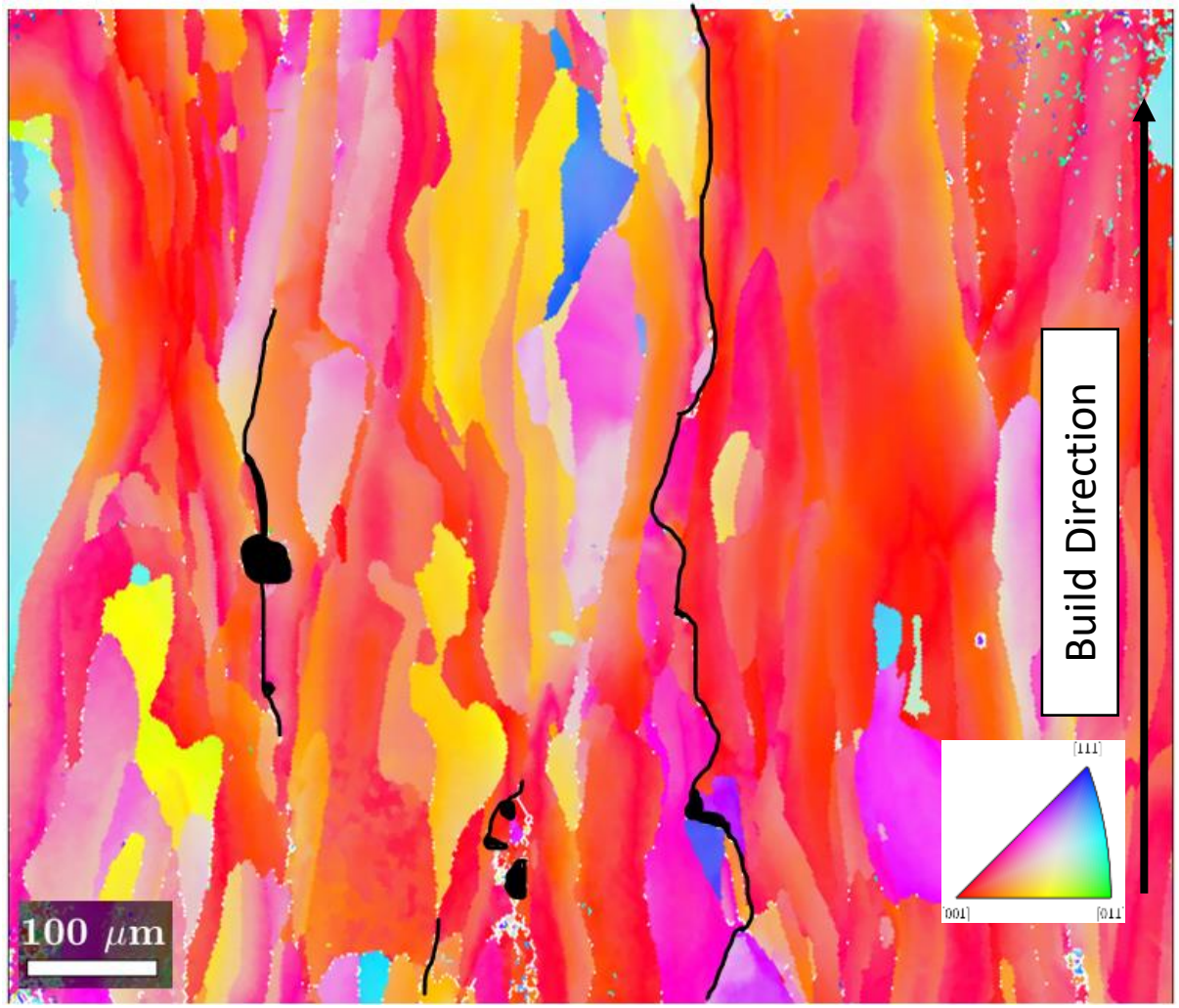
- Student: Chloe Johnson
- Advisor: Dr. Amy Clarke
- Thesis Committee: Dr. Kester Clarke, Dr. Michael Kaufman, Dr. Jeremy Iten, Dr. Garritt Tucker

COLORADO SCHOOL OF MINES

NSF

IOWA STATE UNIVERSITY

Project Motivation



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

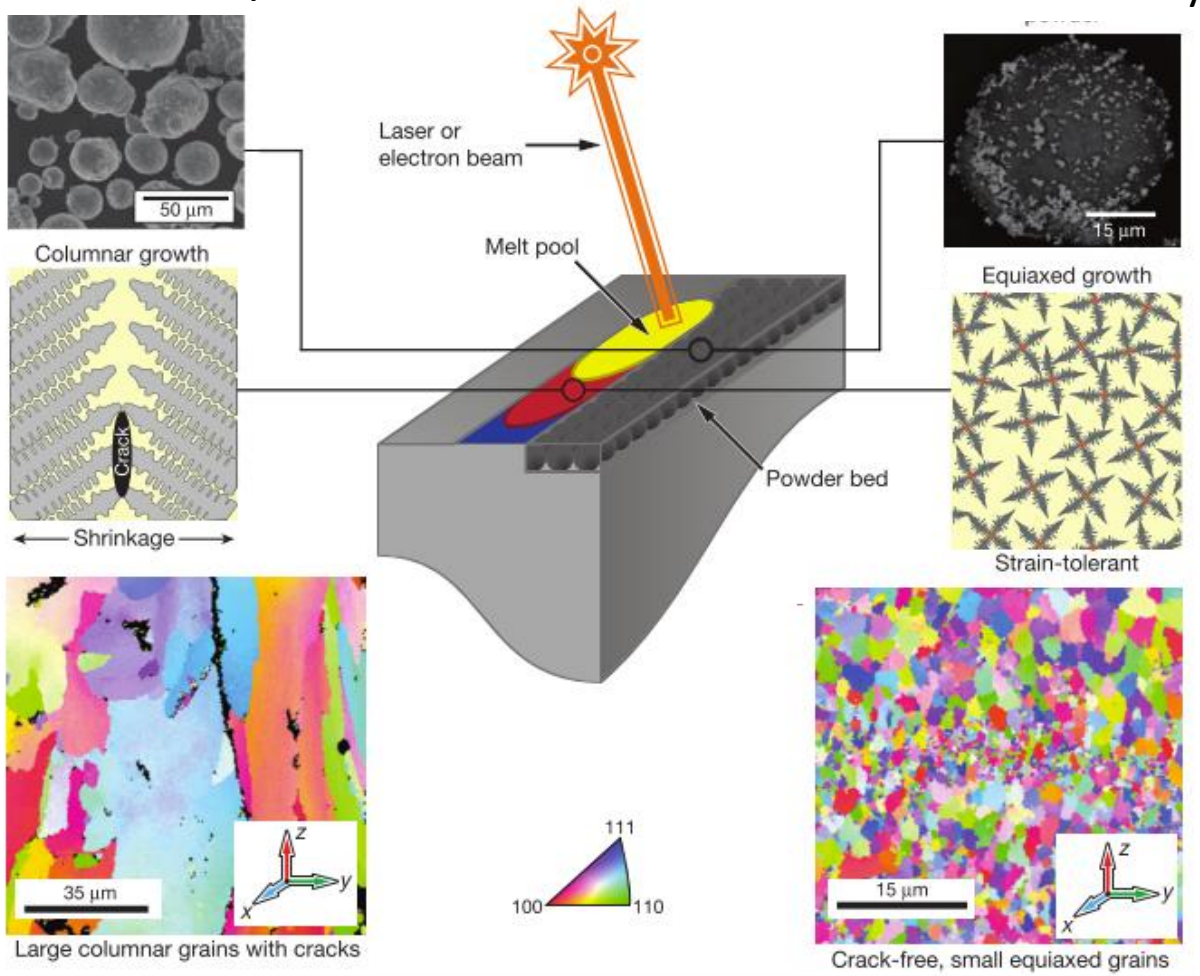
- AM presents method to print new geometries, like complex cooling channels, allowing for use of lower operating temperature alloys like Al alloys
- Al alloys currently used in AM are limited, and have mostly been casting alloys (e.g. AlSi10Mg)
- Under AM conditions, many Al alloys tend to form columnar grains, and are subject to solidification cracking
- These results imply a need for Al alloys designed specifically for AM

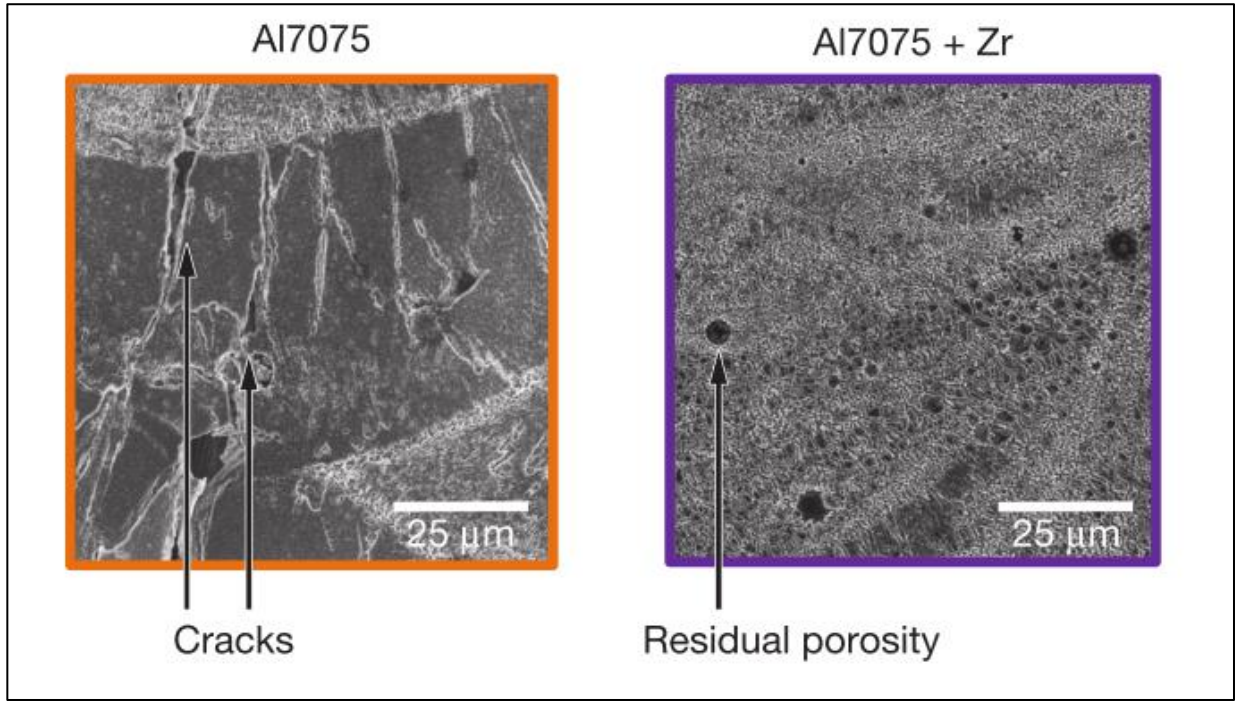
Grain Size Control via Inoculants in AM Alloy Powders

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Standard Alloys

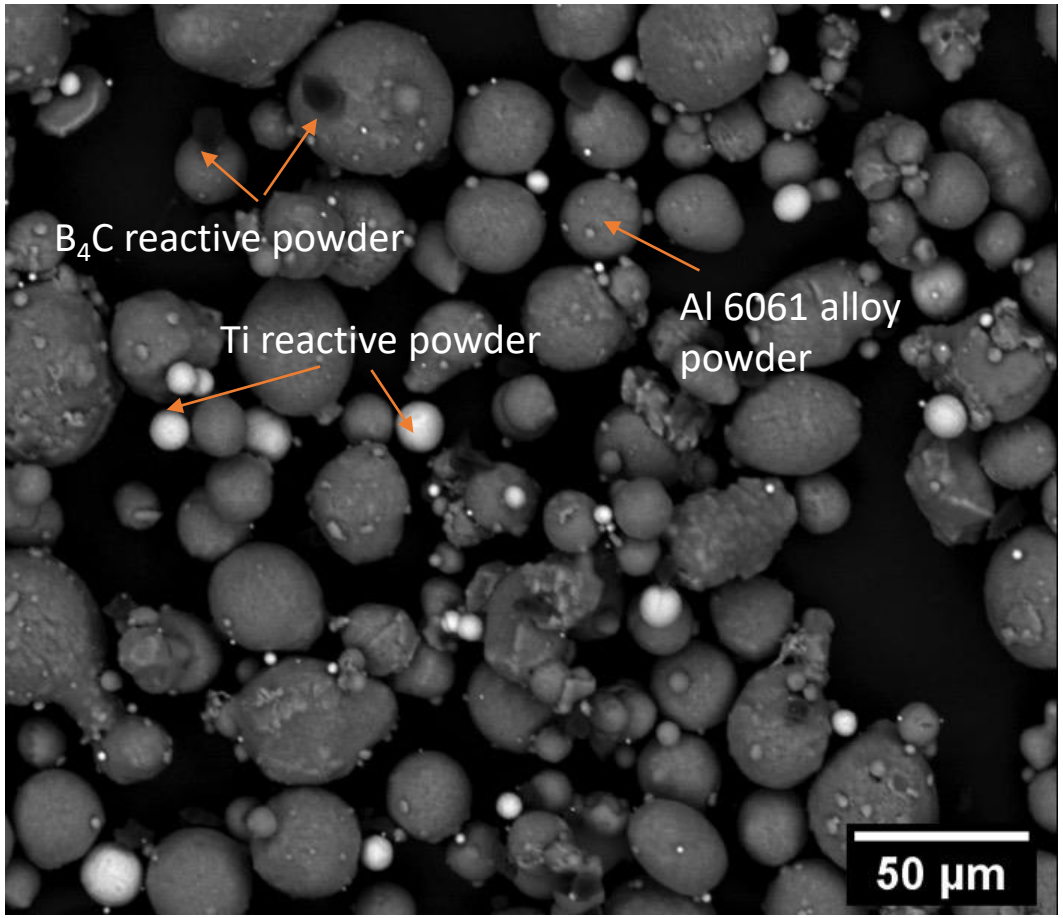
Inoculated Alloys



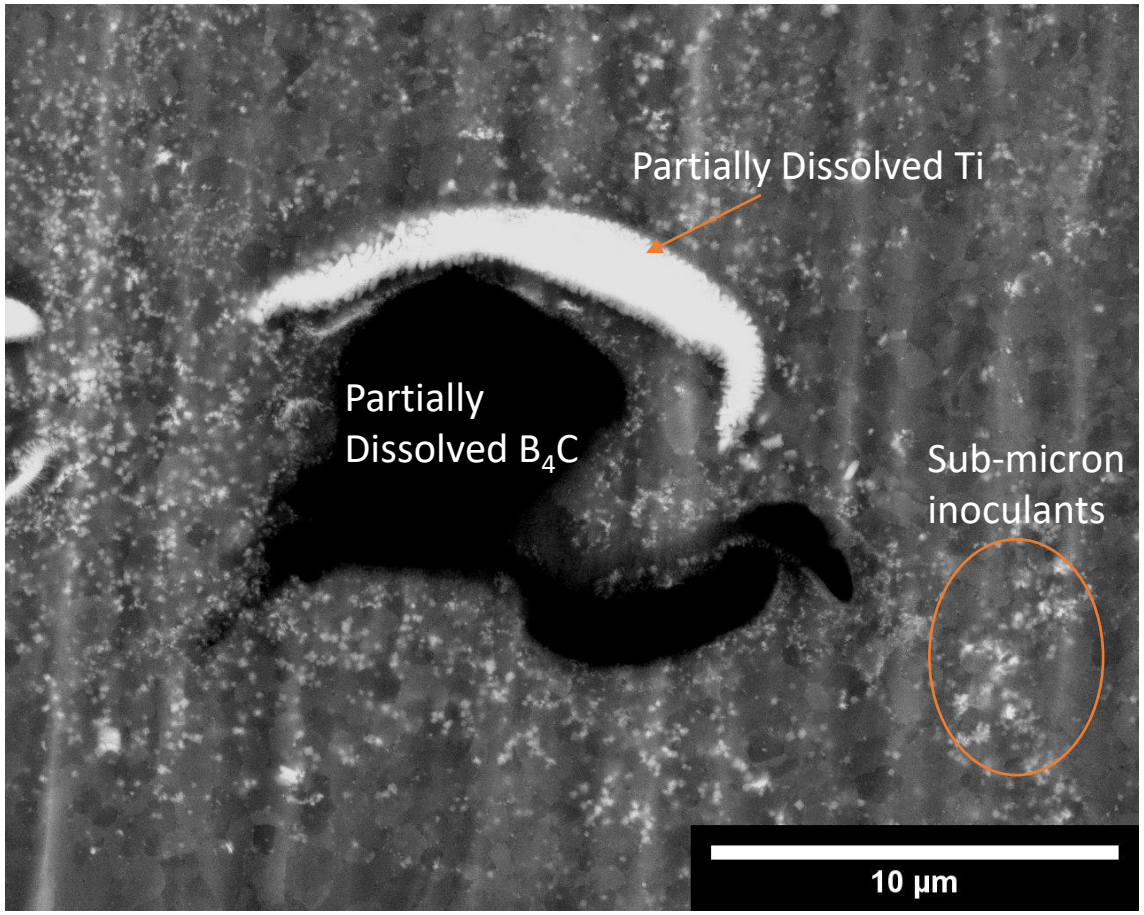


Taken from: Martin et. al, 3D Printing of High-Strength Aluminium Alloys, Nature (2017).

A6061 Reactive Additive Manufacturing (RAM) Alloy Designed for AM



BSE SEM image of A6061-RAM2 alloy powder, 2 refers to vol. % of Ti and B_4C in a ratio of 3:1

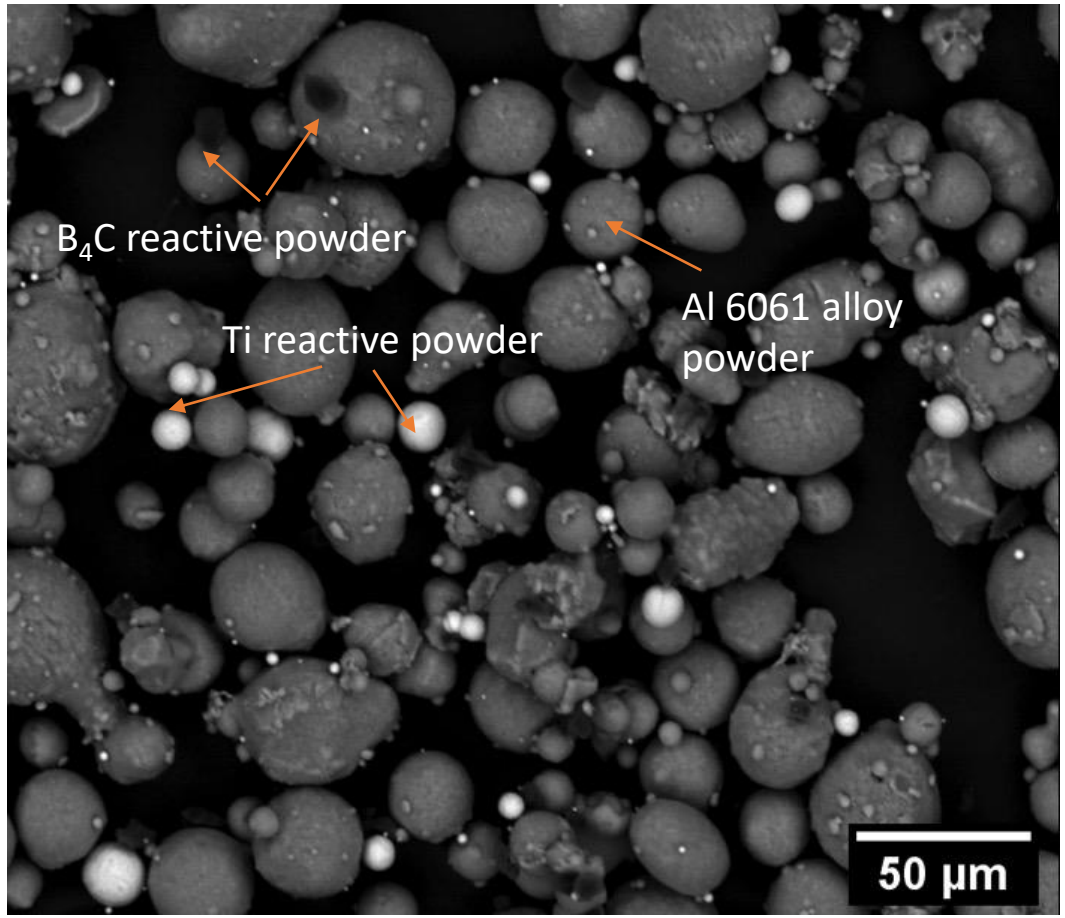


SEM back scatter image of as built A6061-RAM2

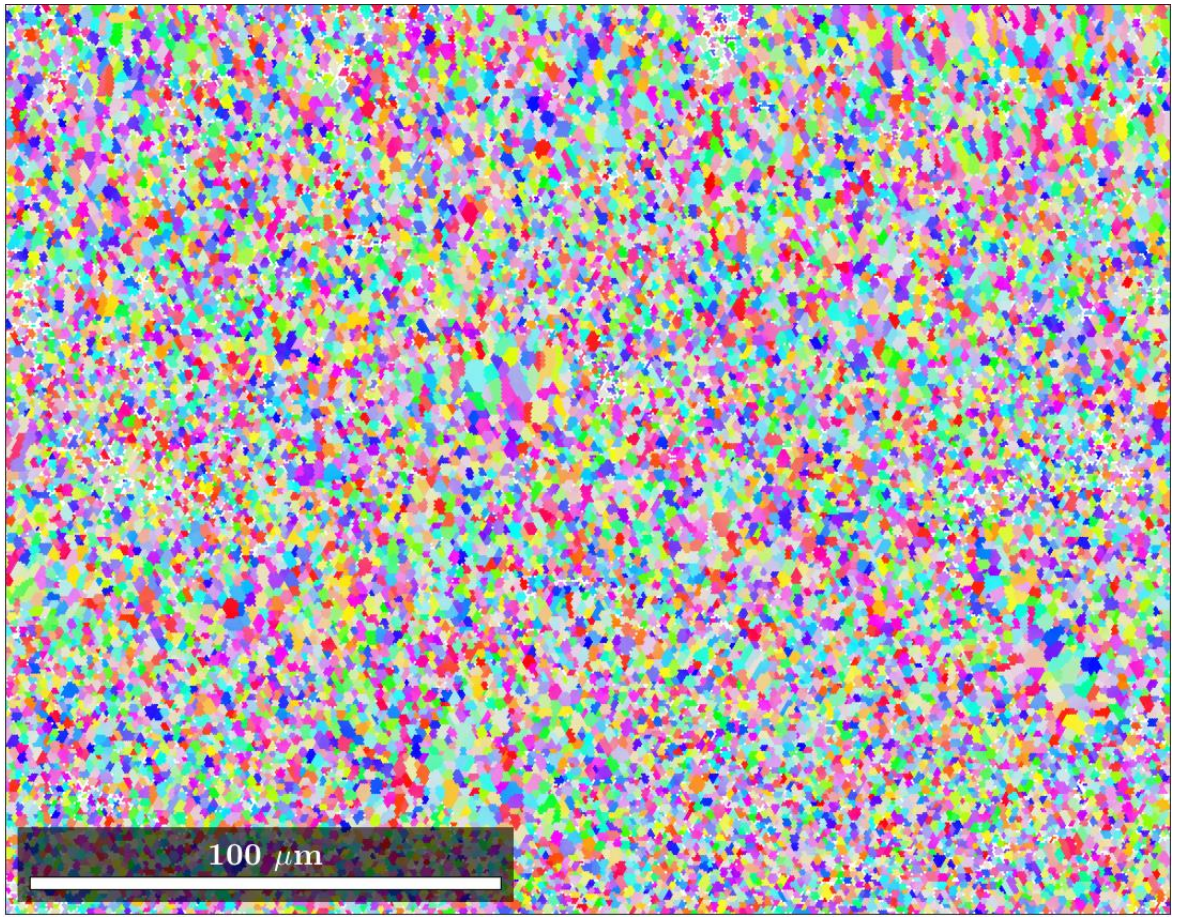
J. S. Neuchterlein & J. J. Iten, Reactive additive manufacturing, US Patent 20160271878 A1, priority 2015-03-17, published 2016-10-22.

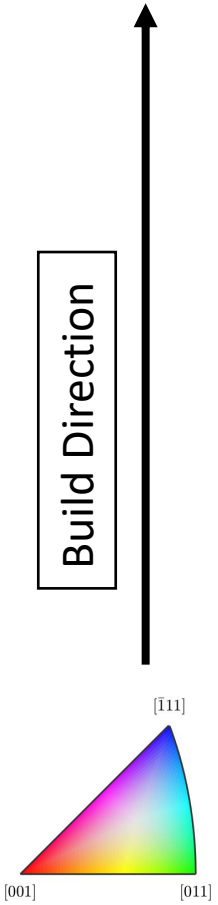
Al 6061 Reactive Additive Manufacturing (RAM) Alloy Designed for AM

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS



BSE SEM image of A6061-RAM2 alloy powder, 2 refers to vol. % of Ti and B_4C in a ratio of 3:1





EBSD IPF of as built A6061-RAM2

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

Project Objectives/Research Questions

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

1. How do solidification conditions, specifically solidification velocity and thermal gradient, impact grain refinement mechanisms in A6061-RAM alloys during laser powder bed processing?
2. How does starting reactive particle content in A6061-RAM alloys impact inoculant formation and grain refinement mechanisms during laser powder bed fusion?
3. What is the impact of particle content in as-built A6061-RAM alloys, built using laser powder bed fusion, on aging and precipitation strengthening in these alloys?

Research Question 2

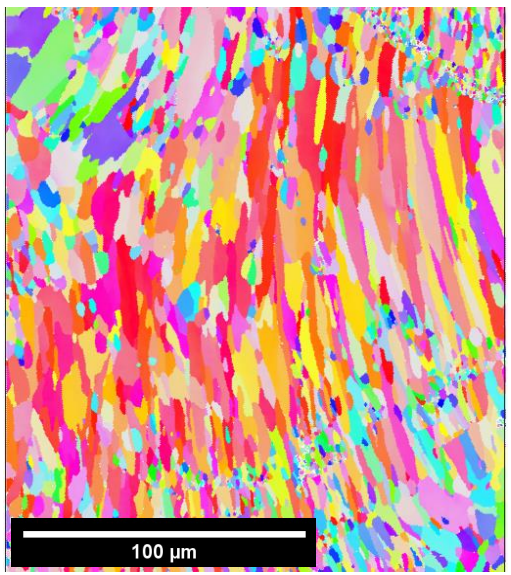
How does starting reactive particle content in A6061-RAM alloys impact inoculant formation and grain refinement mechanisms during laser powder bed fusion?

- Varied starting reactive particle content for samples containing both Ti and B_4C (0.5, 1, 2, 10 vol. %)
- Isolated impacts of Ti vs. B_4C on refinement
- Characterized grain structure and particle content in all alloys

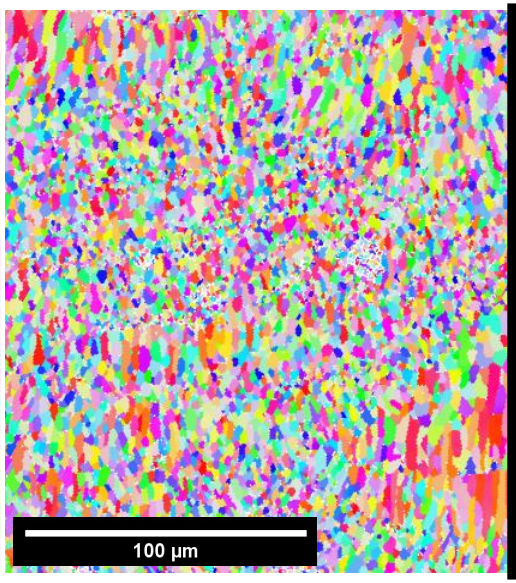
Degree of Refinement for Various Starting Reactive Particle Content

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

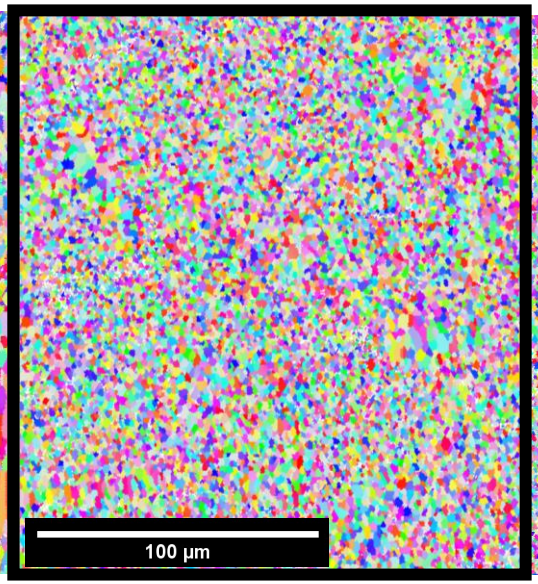
6061
Grain size
Area: 291.38 µm^2
Diameter: 12.59 µm
(range of 2.31-941.19 µm max diameter)



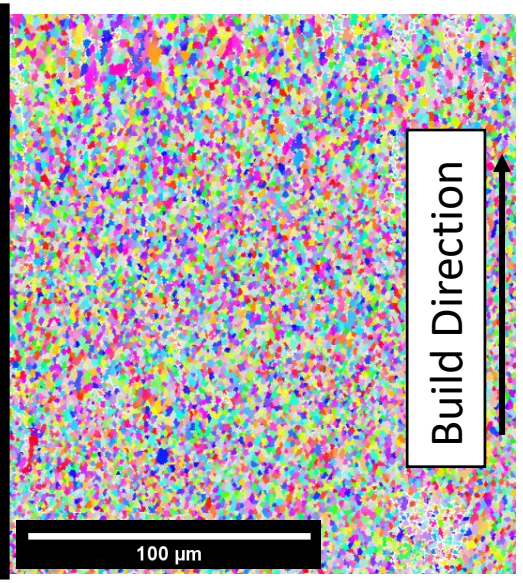
RAM 0.5
Grain size
Area: 8.87 µm^2
Diameter: 4.37 µm



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

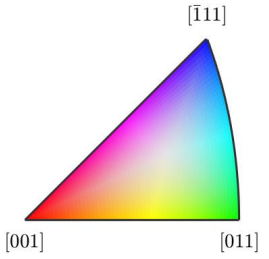


RAM 2
Grain size
Area: 1.91 µm^2
Diameter: 1.93 µm



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

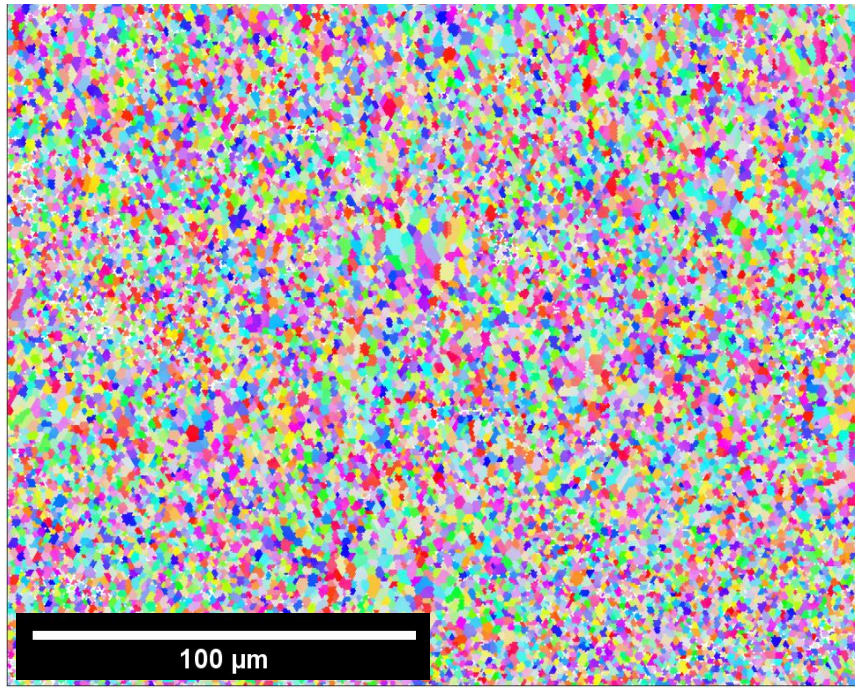
Increasing RAM Content



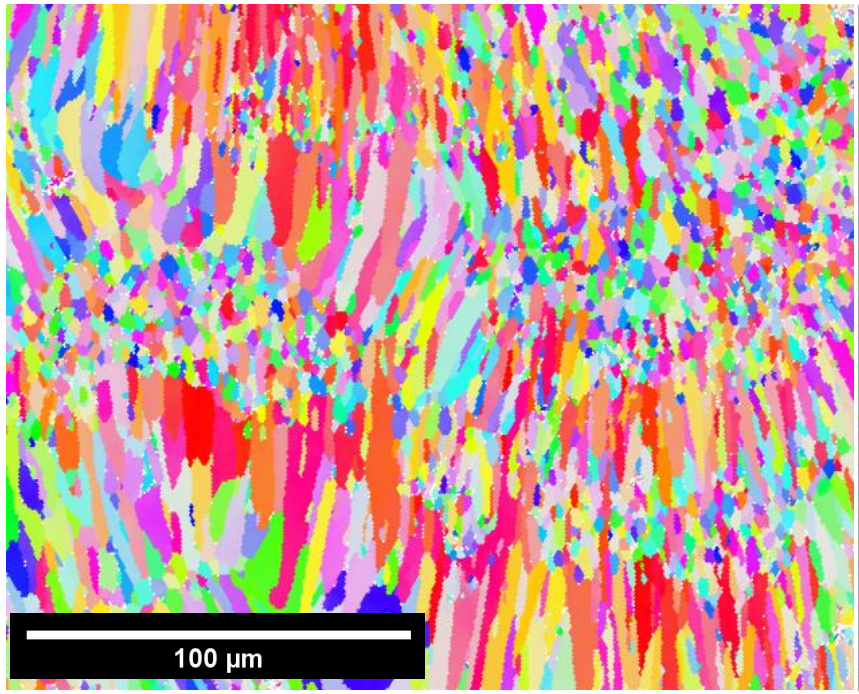
Impact of Individual Reactive Powders (B_4C or Ti) vs. A6061-RAM2 (B_4C + Ti)

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

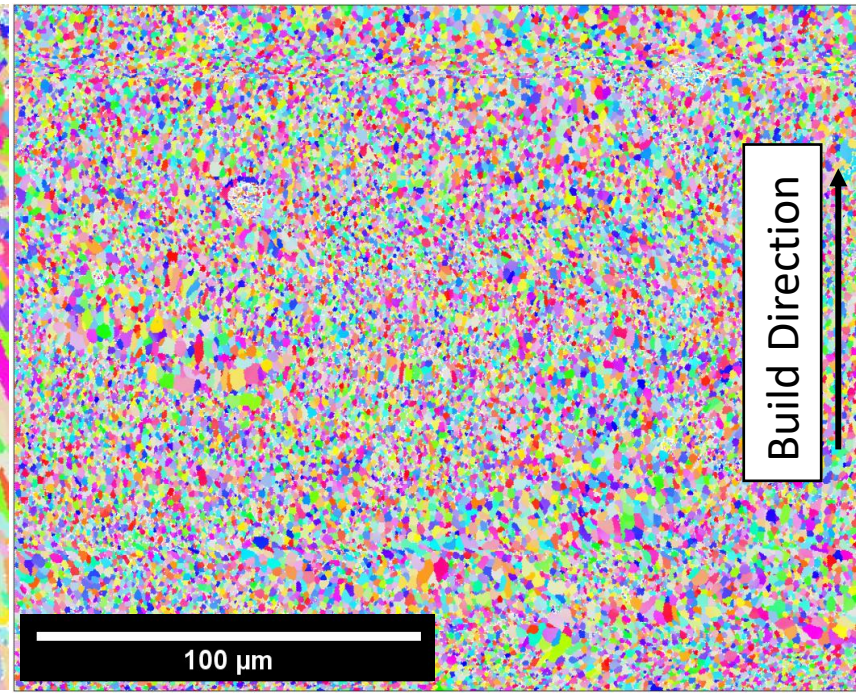
All builds were performed with 40 um layer thickness, 370 W, 0.2 mm hatch spacing



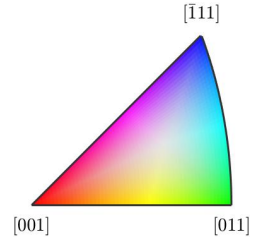
A6061-RAM2
1300 mm/s
Grain size
Area: 1.95 µm^2
Diameter: 1.96 µm



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



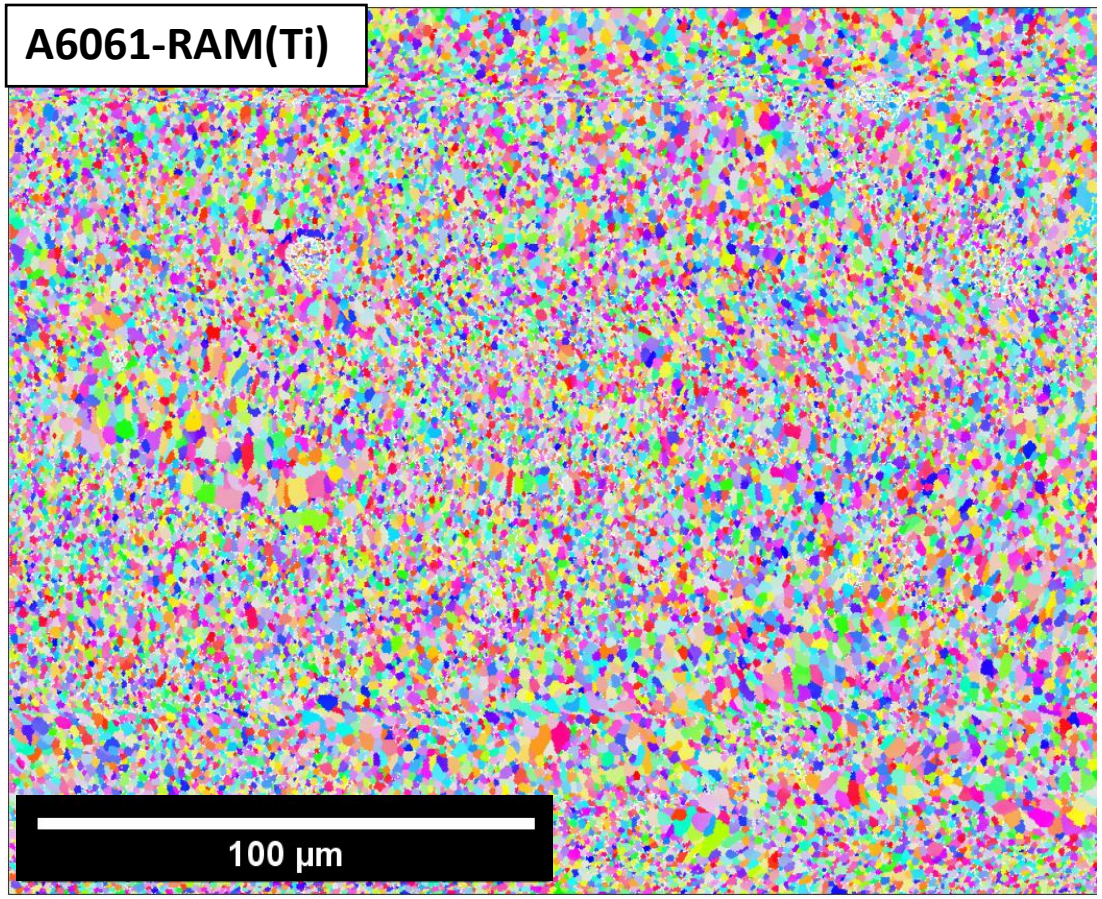
A6061-RAM: Ti
1300 mm/s
Grain size
Area: 0.73 µm^2
Diameter: 1.16 µm



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

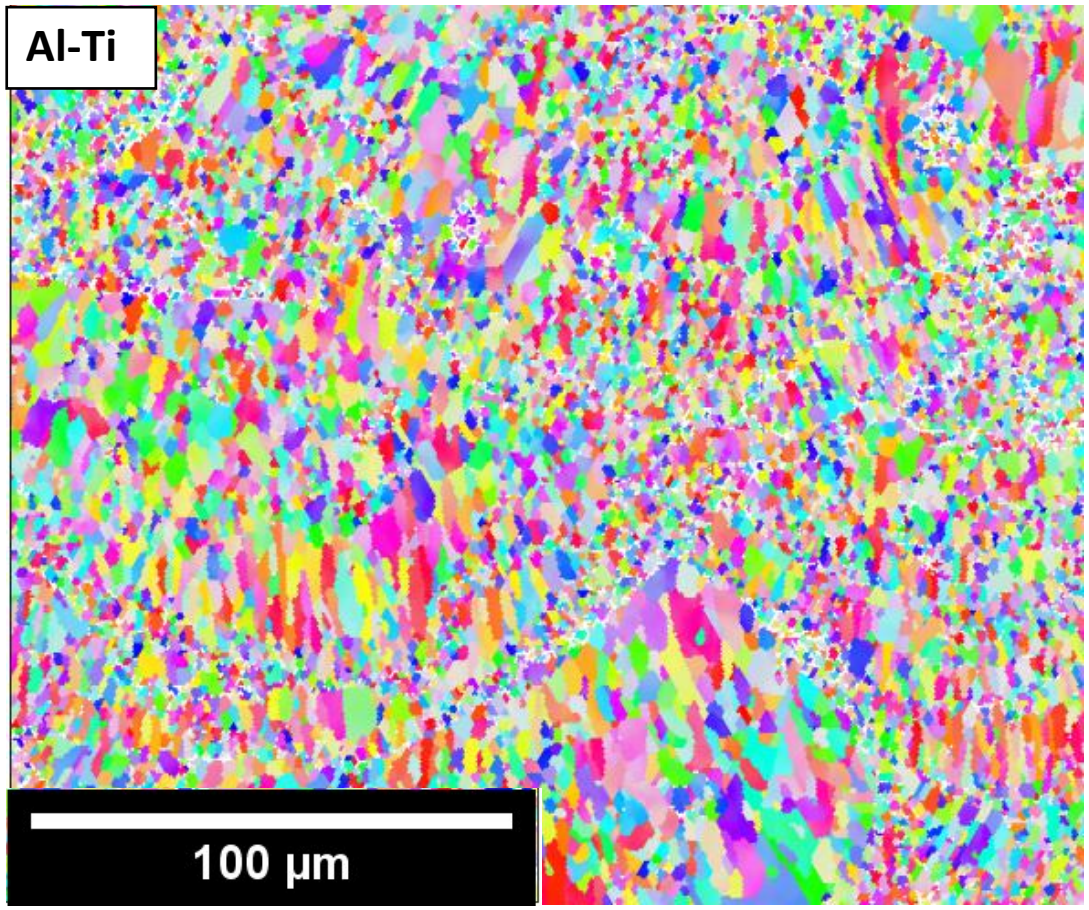
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

40 um layer thickness, 370 W, 1300 mm/s, and 0.2 mm hatch spacing



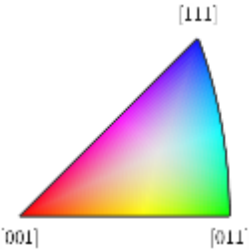
Grain size
Area: 0.73 µm^2
Diameter: 1.16 µm

30 um layer thickness, 370 W, 1300 mm/s, and 0.19 mm hatch spacing



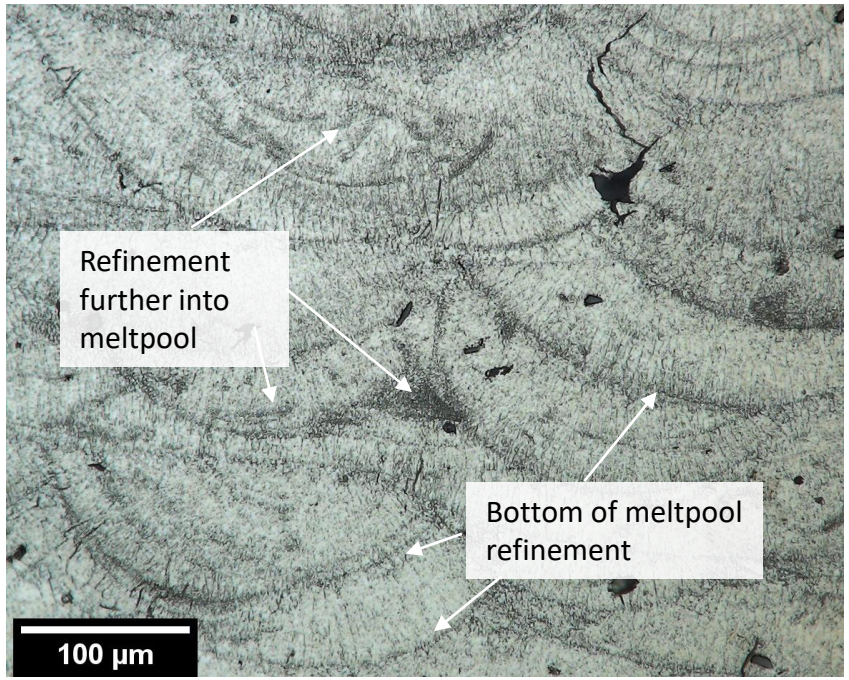
Build Direction

Grain size
Area: 2.83 µm^2
Diameter: 2.32 µm

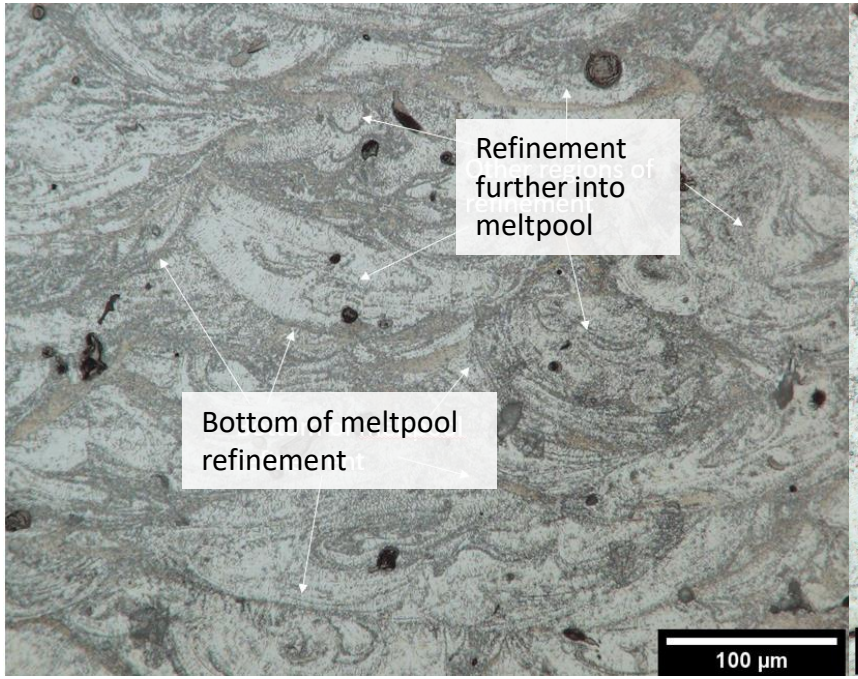


Refinement Locations in Samples with a CET

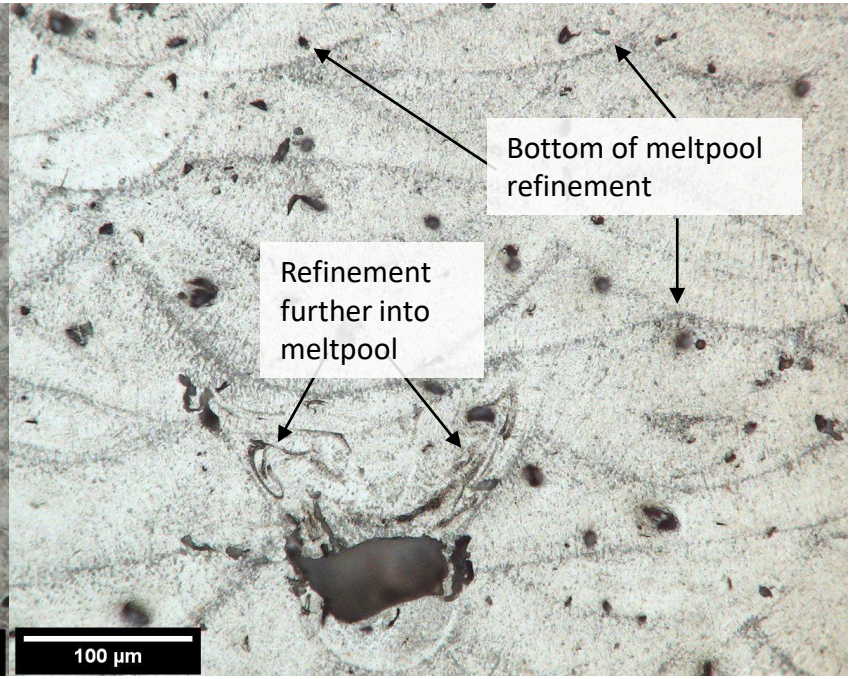
A6061-RAM0.5



Al-Ti

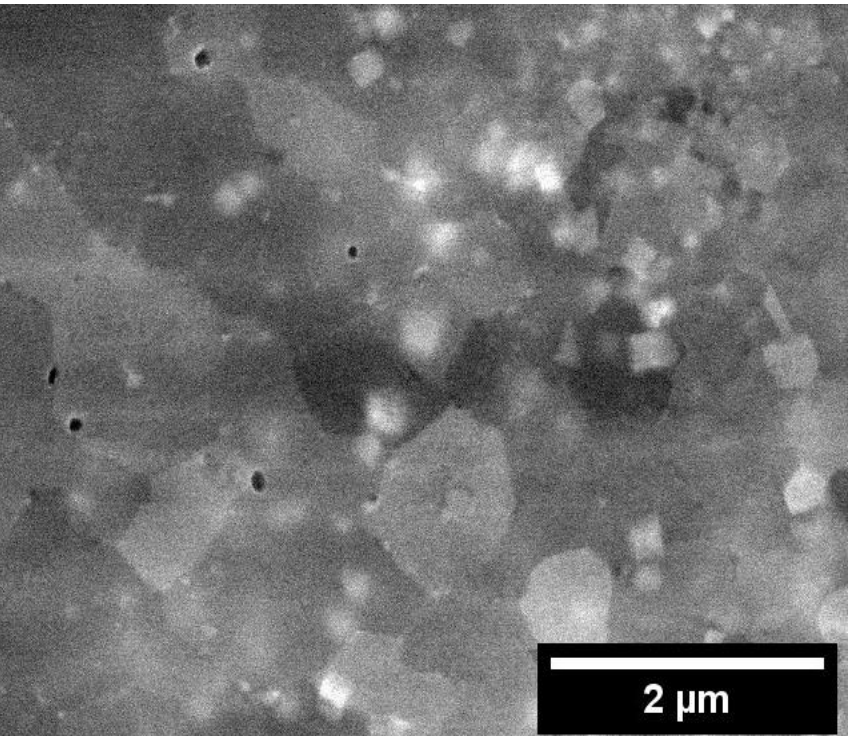


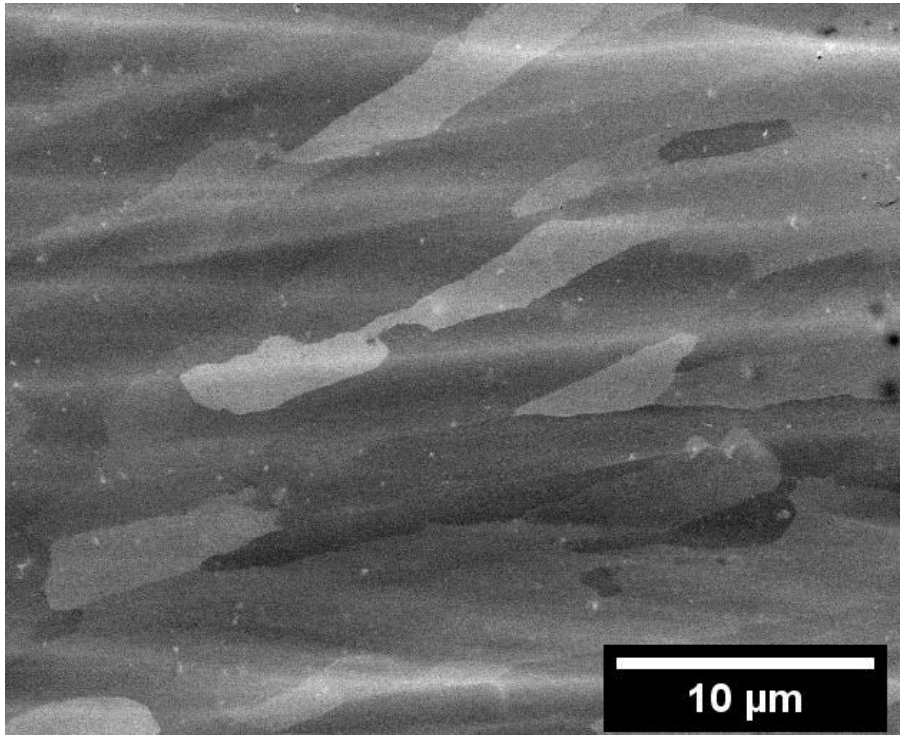
A6061-RAM(B_4C)



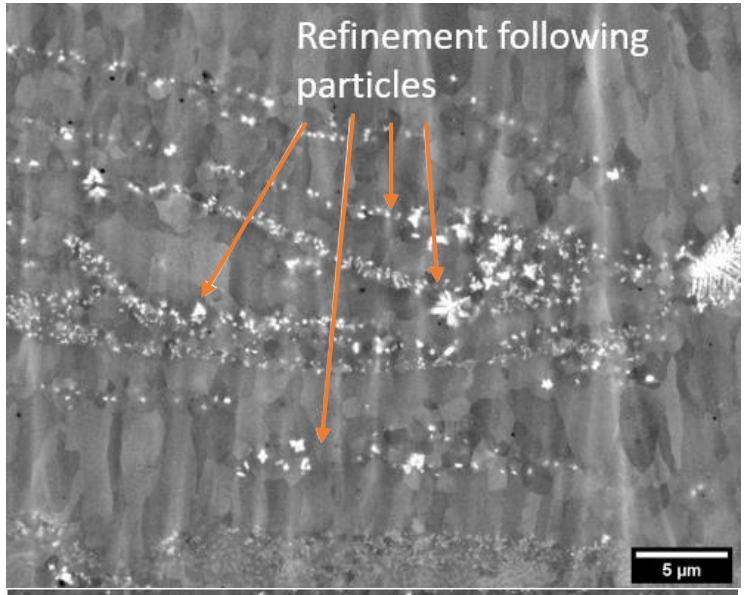
Light optical microscopy (LOM) images of samples etched with Kroll's Reagent

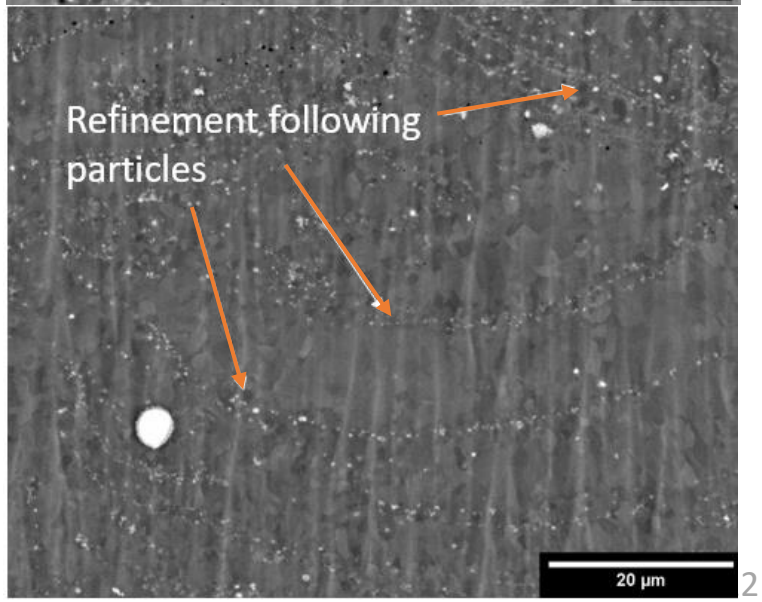
Refined vs. Columnar Regions: SEM Observations





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





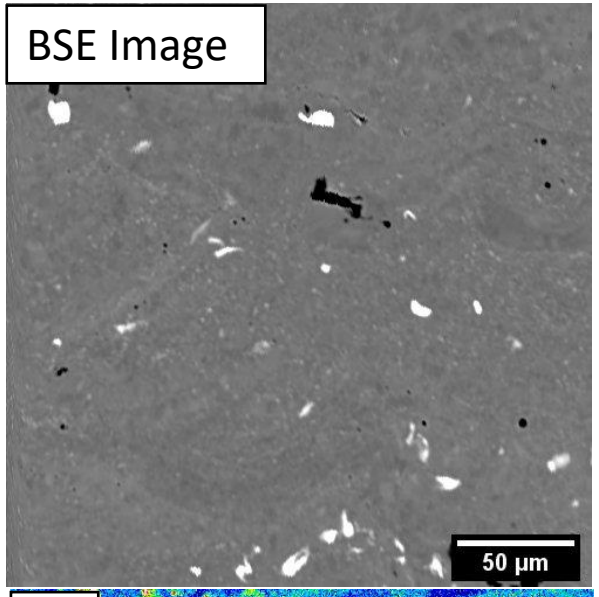
SEM-BSE Images taken from an Al-Ti sample

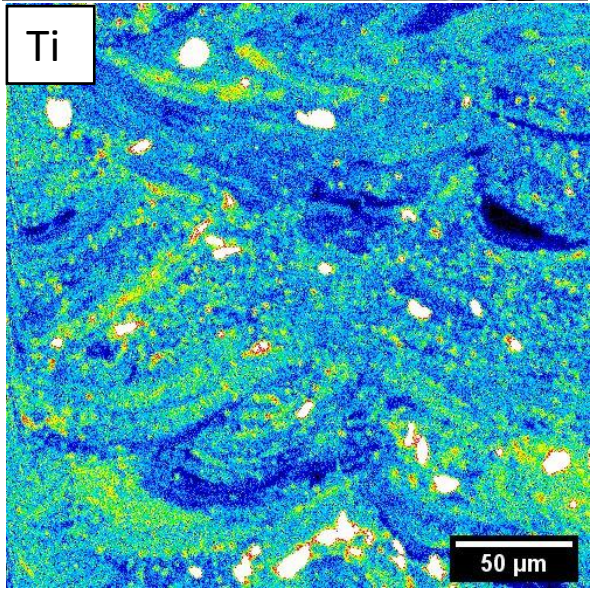
Electron Microprobe Maps of Solute Distribution

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

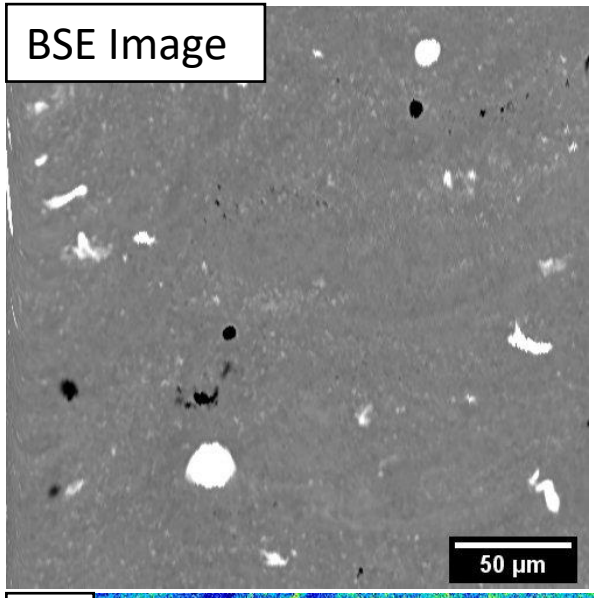
Increasing counts

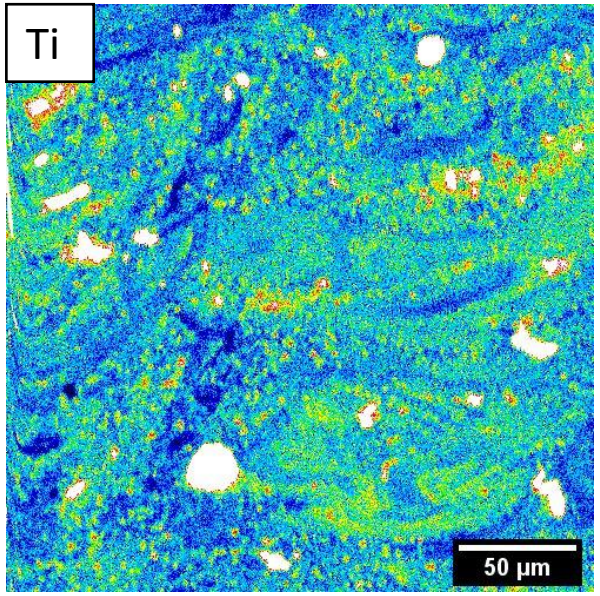
A6061-RAM2



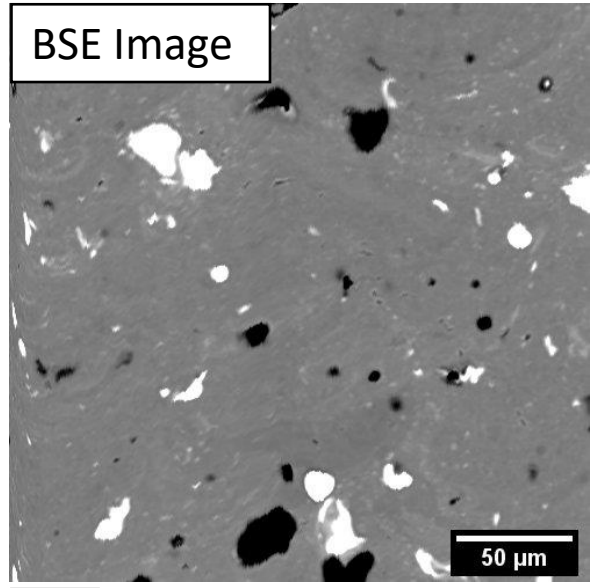


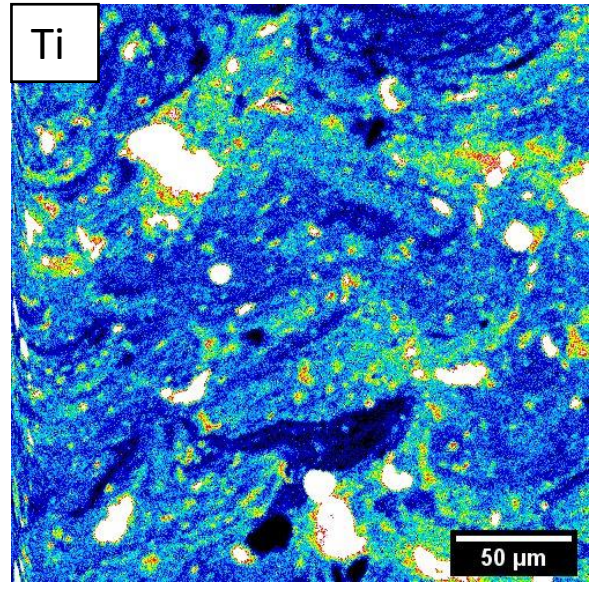
A6061-RAM(Ti)



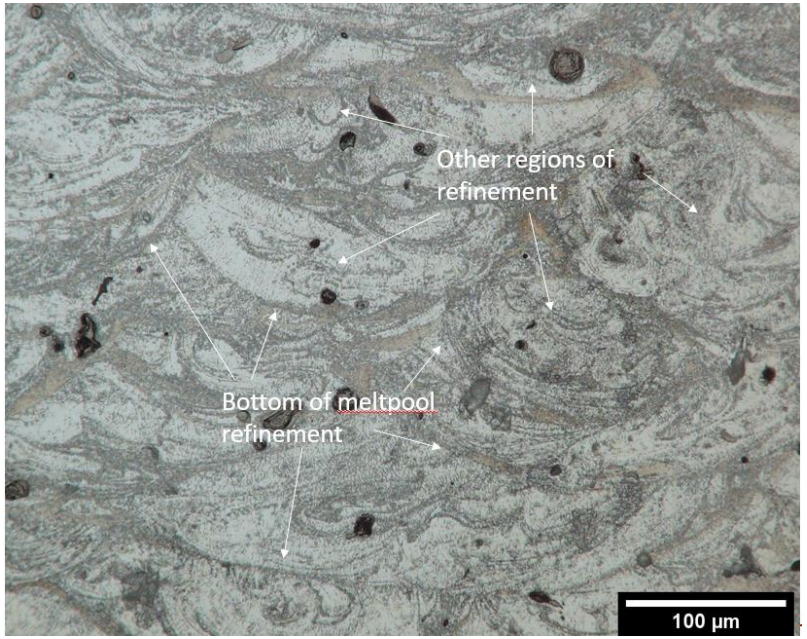


Al-Ti

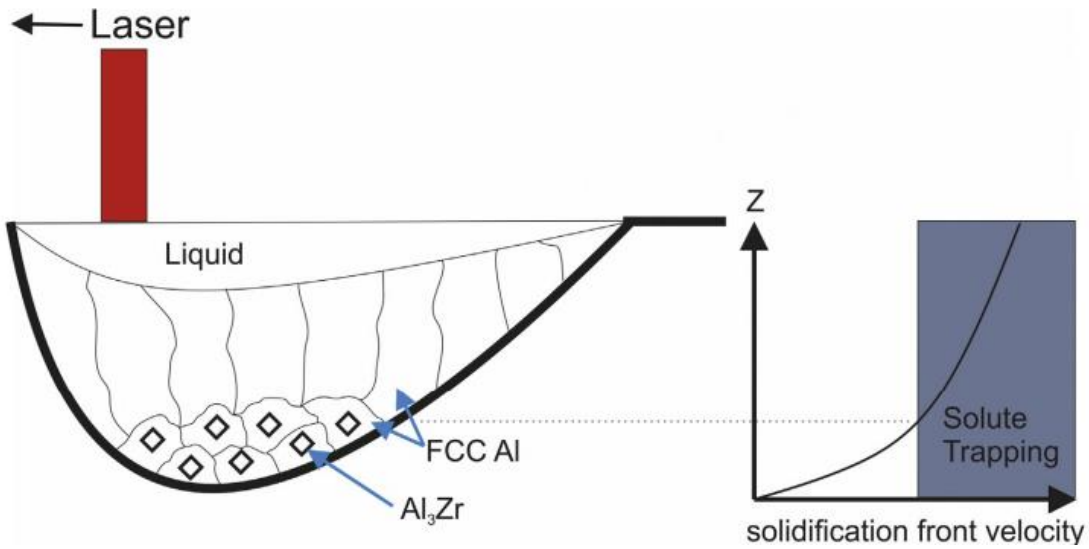




Factors Contributing to Refinement Locations: Bottom of the Melt Pool



LOM of etched Al-Ti as-built sample



Taken from: Griffiths et. al, Effect of laser rescanning on the grain microstructure of a selective laser melted Al-Mg-Zr alloy, Materials Characterization (2018).

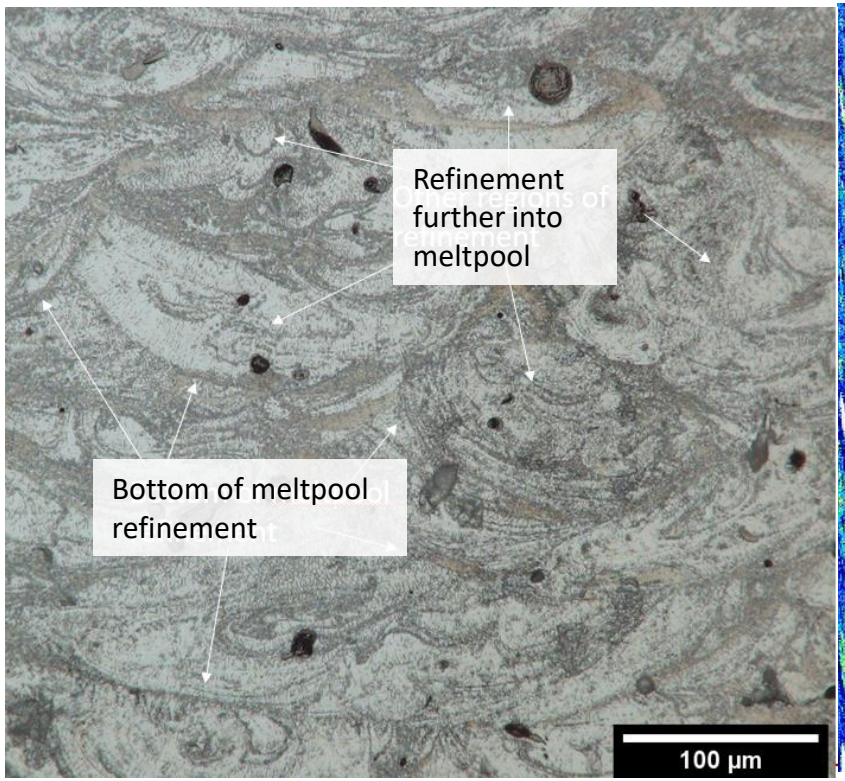
Nominal and measured (STEM EDS) Ti content for various alloys

Sample	Measured Ti (wt. %)	Nominal Ti (wt. %)
Al-Ti	0.96 ± 0.46	2.45
A6061-RAM(Ti)	0.8 ± 0.42	2.45
A6061-RAM2	1.12 ± 0.41	2.45
A6061-RAM10	0.69 ± 0.22	12.25

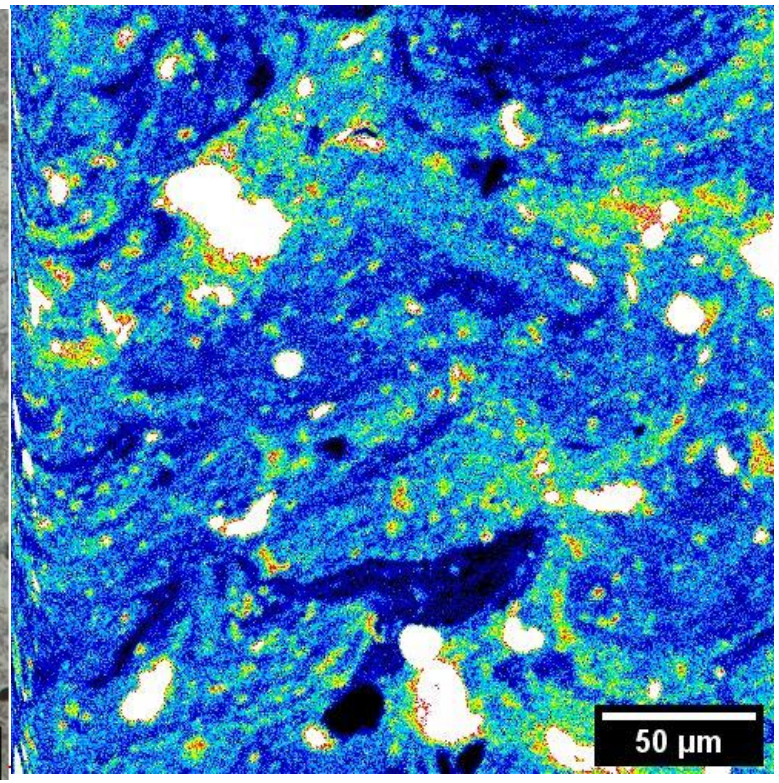
Note: maximum solubility of Ti in Al ~1.1 wt.%

Factors Contributing to Refinement Locations: Further into the Meltpool

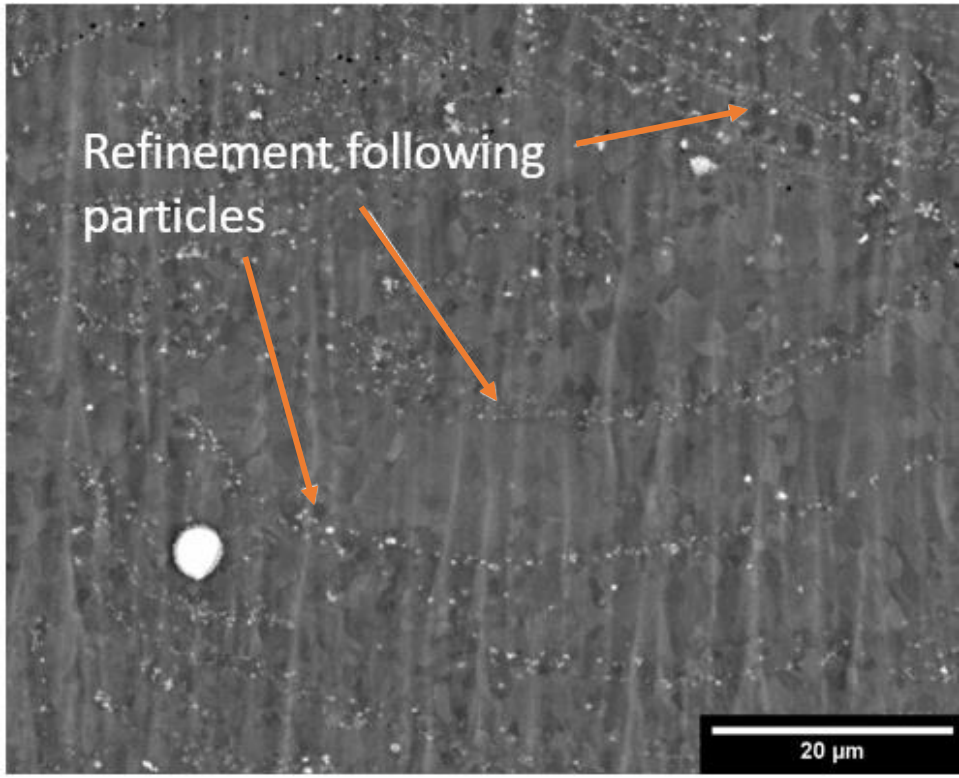
Al-Ti



Etched LOM Image

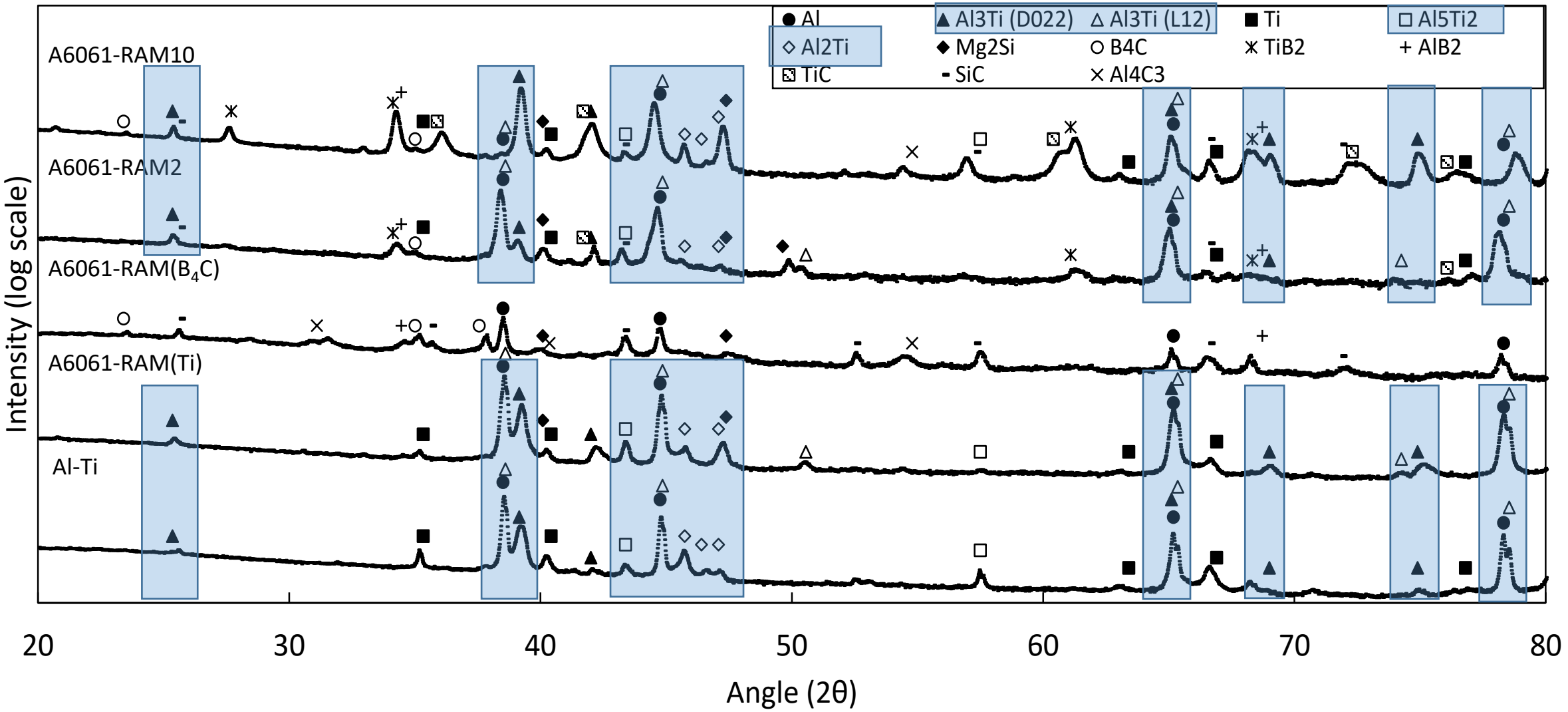


EPMA Ti map

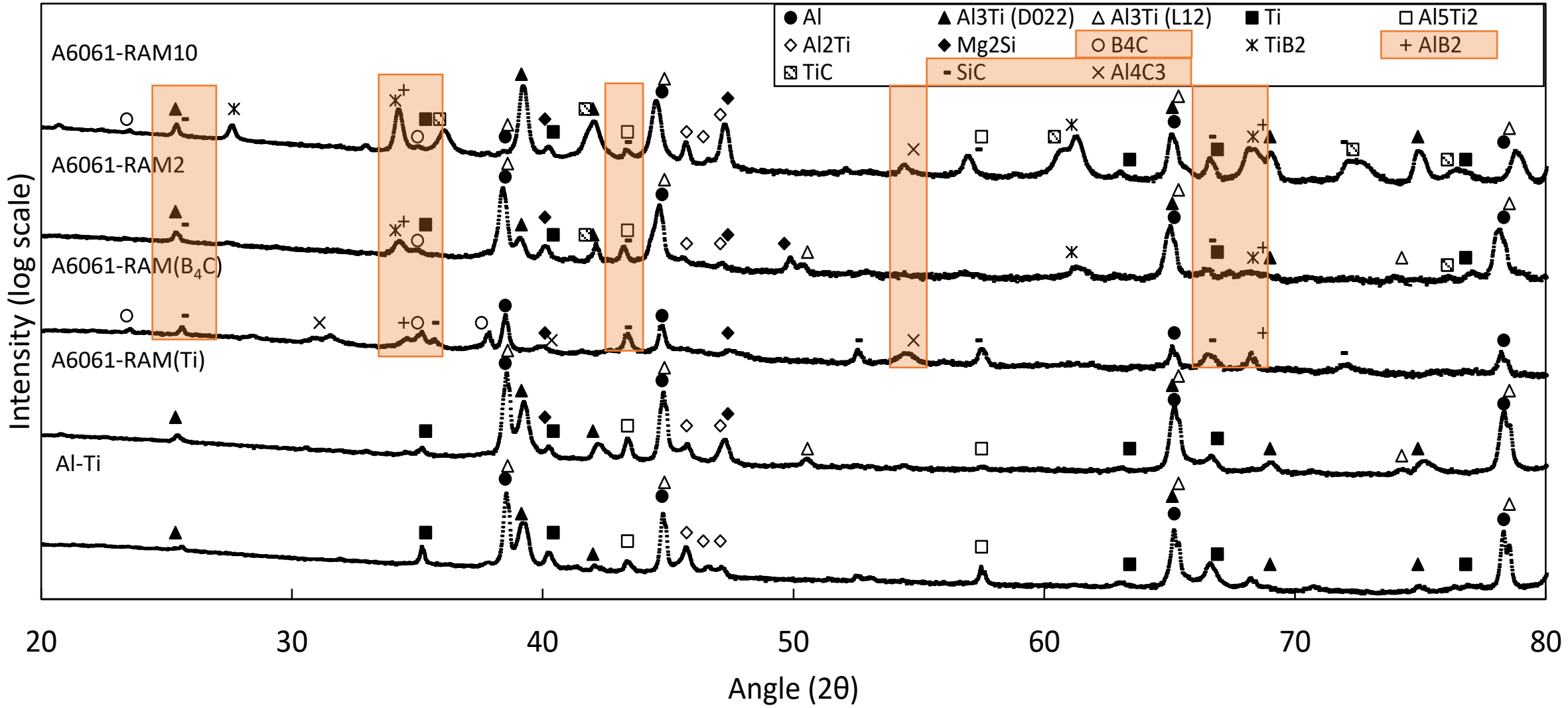


SEM BSE Image

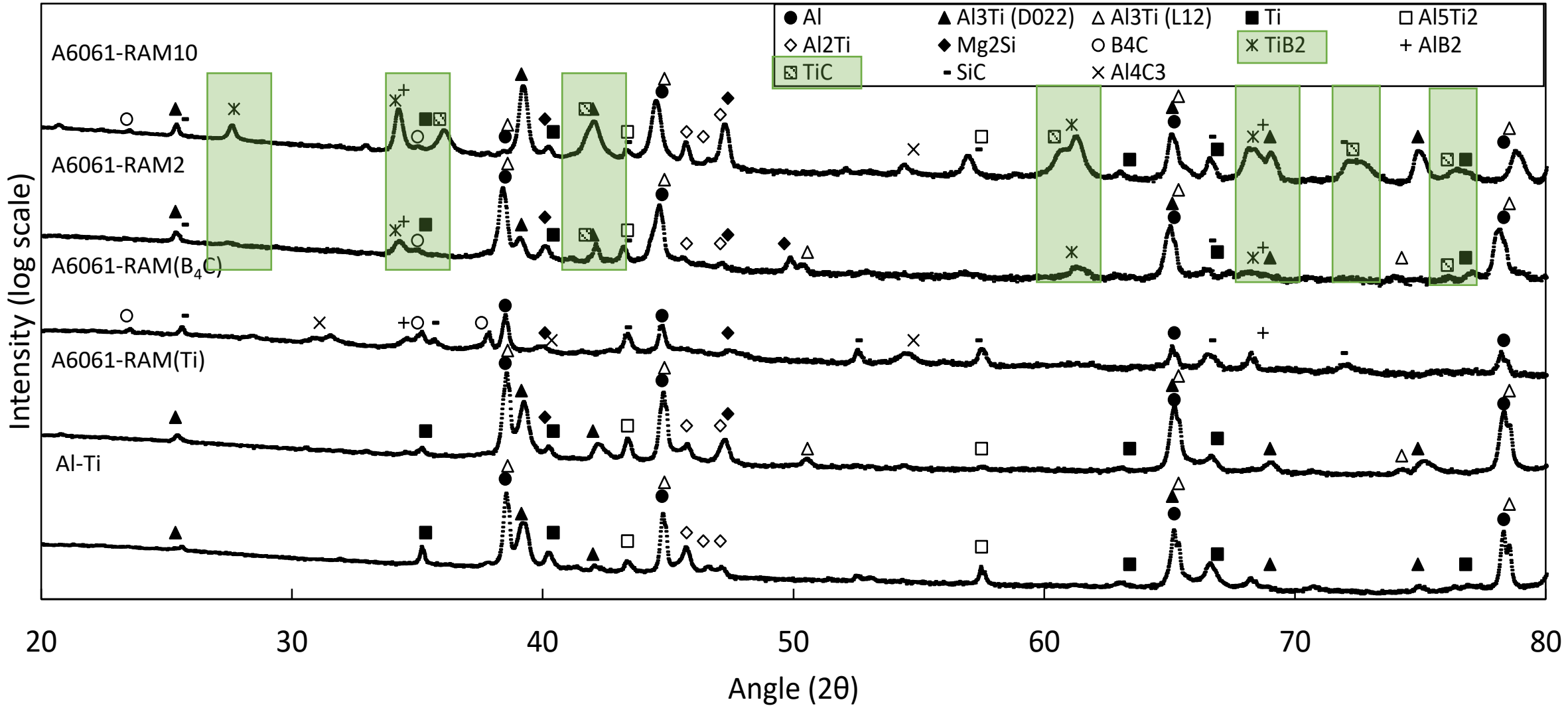
Particle Types (XRD) For Dissolved Matrix Samples



Particle Types (XRD) For Dissolved Matrix Samples



Particle Types (XRD) For Dissolved Matrix Samples



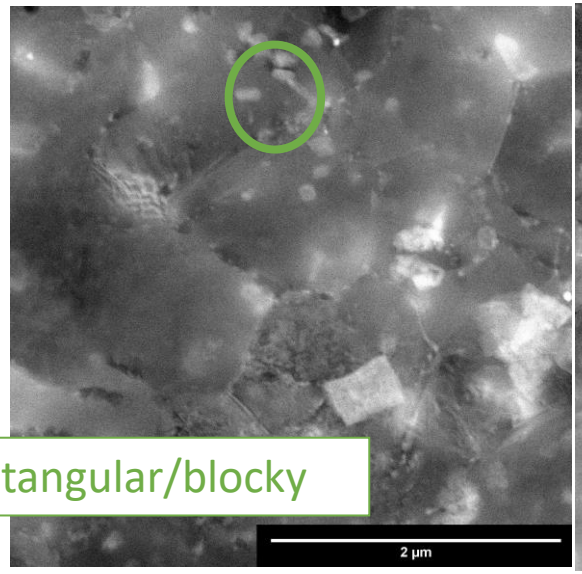
Observed Particle Morphologies A6061-RAM(Ti), Al-Ti, & A6061-RAM(B_4C) (HAADF STEM)

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

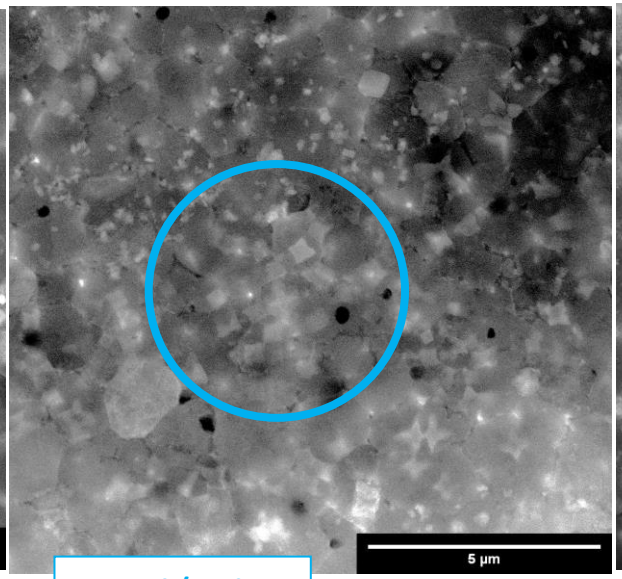
A6061-RAM(Ti)

Al-Ti

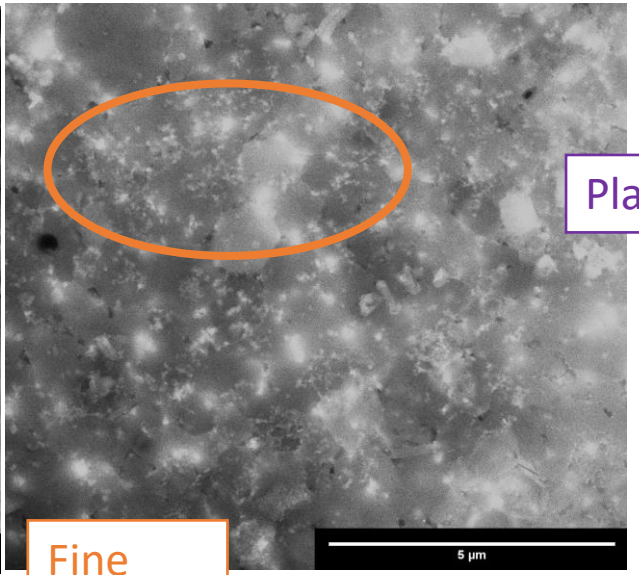
A6061-RAM(B_4C)



Rectangular/blocky

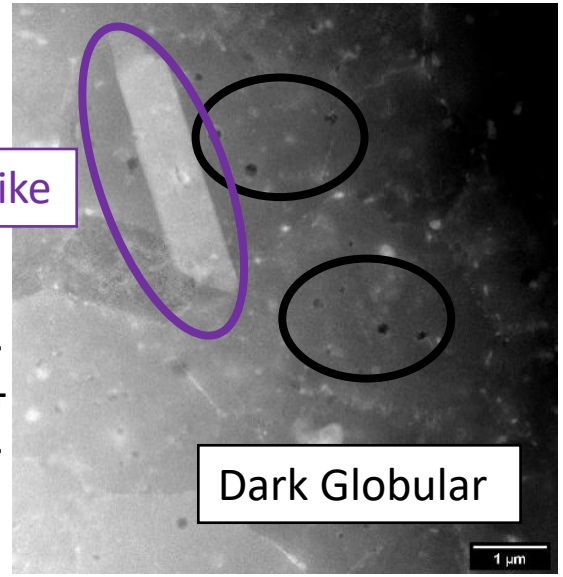


Petal/cubic

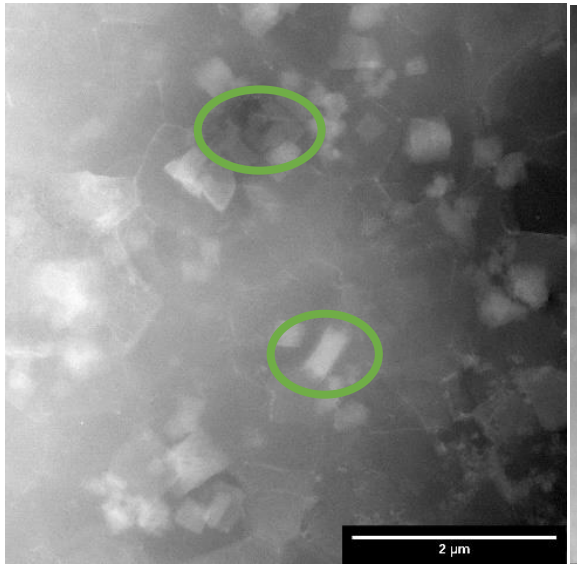


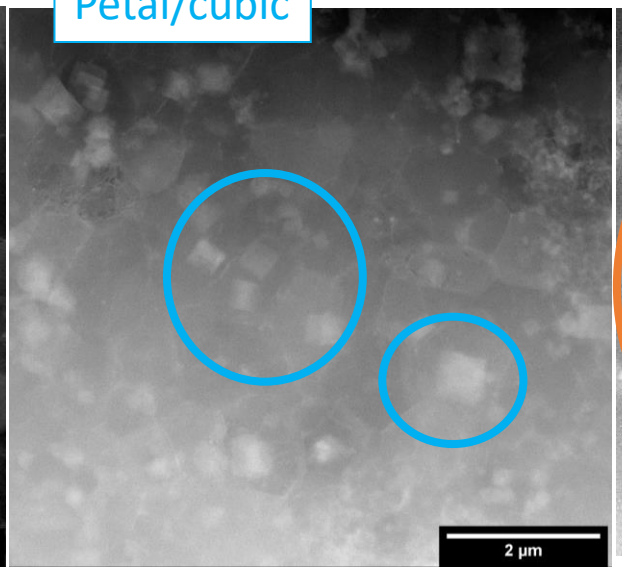
Fine Clusters

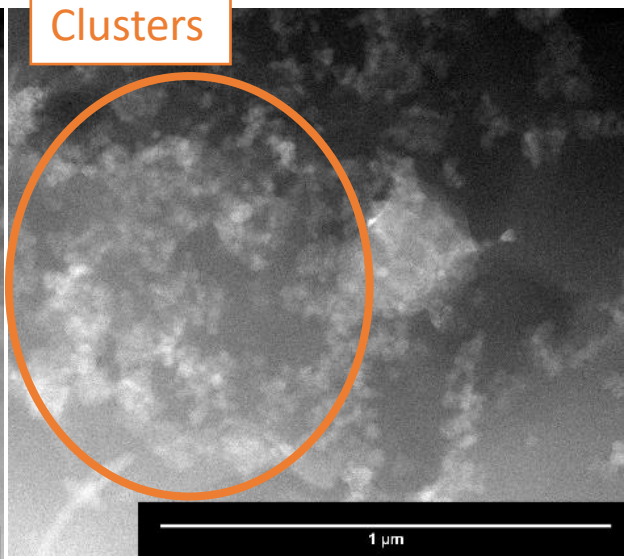
Platelike

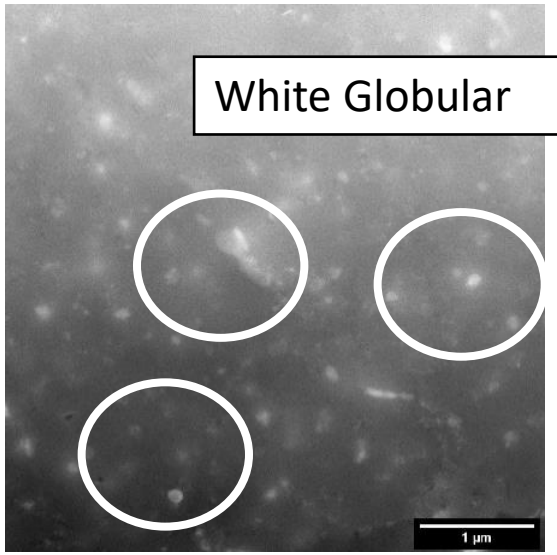


Dark Globular





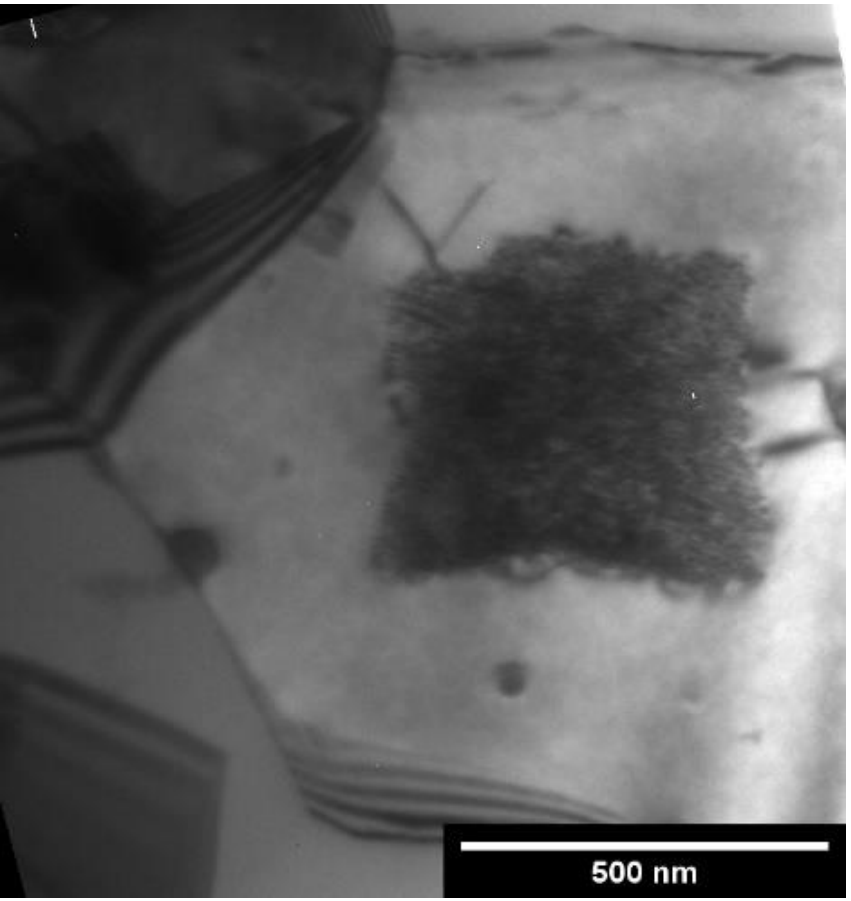




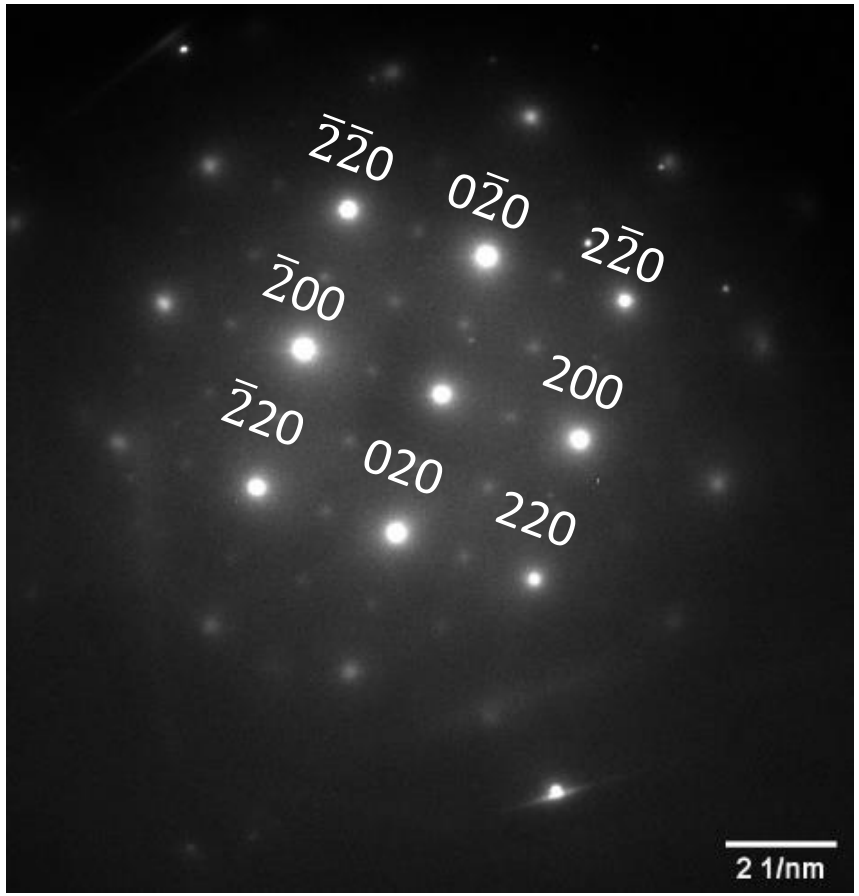
White Globular

Phase ID of Cubic/Petal Particle: Al-Ti

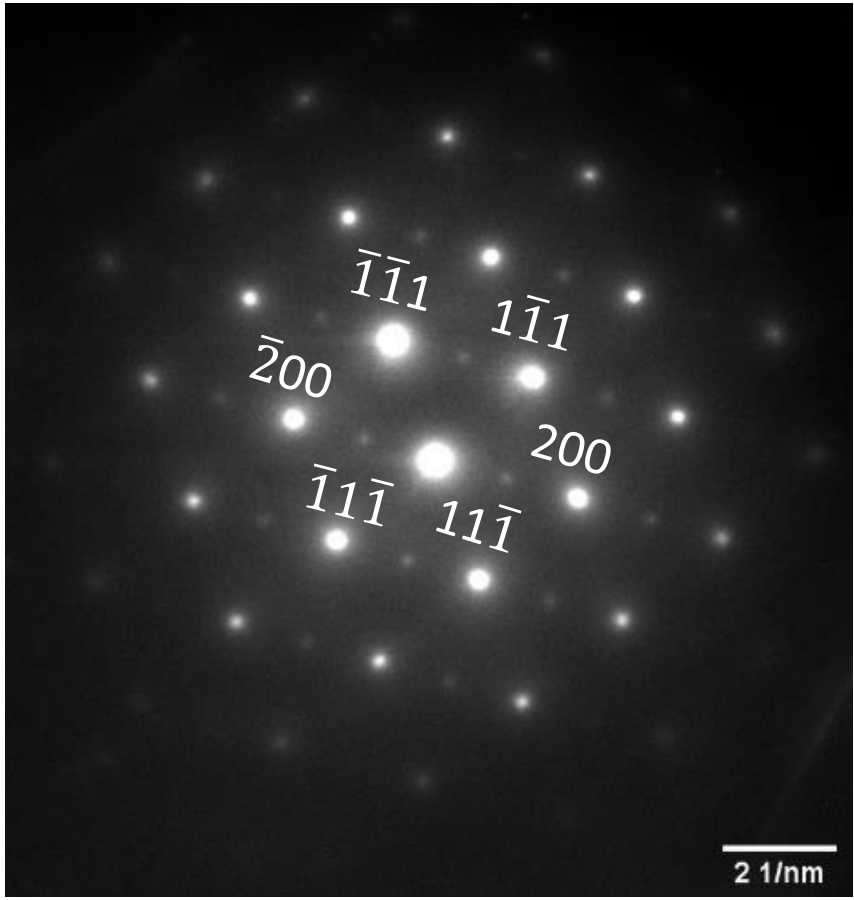
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS



DF TEM image taken down center reflection of SADP



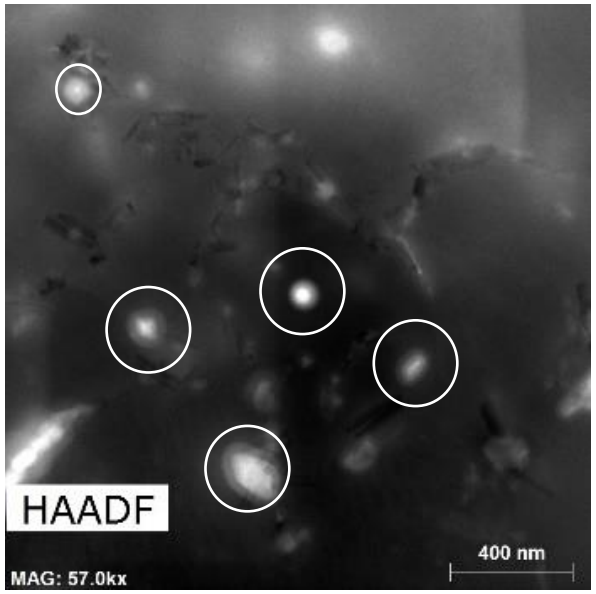
SADP from [001] zone axis

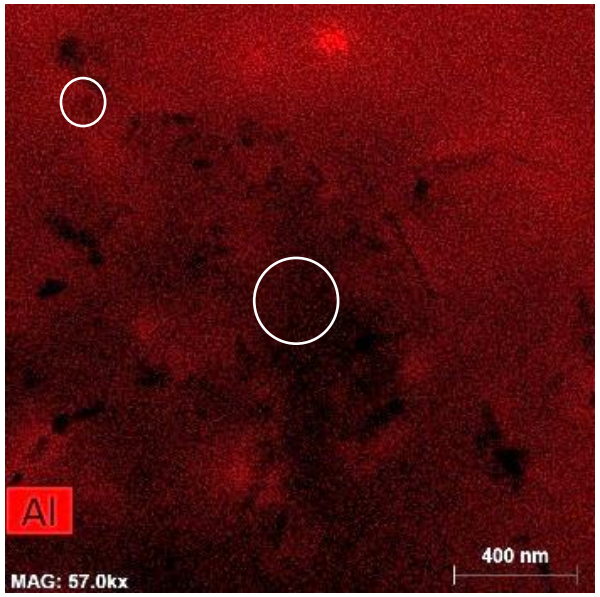


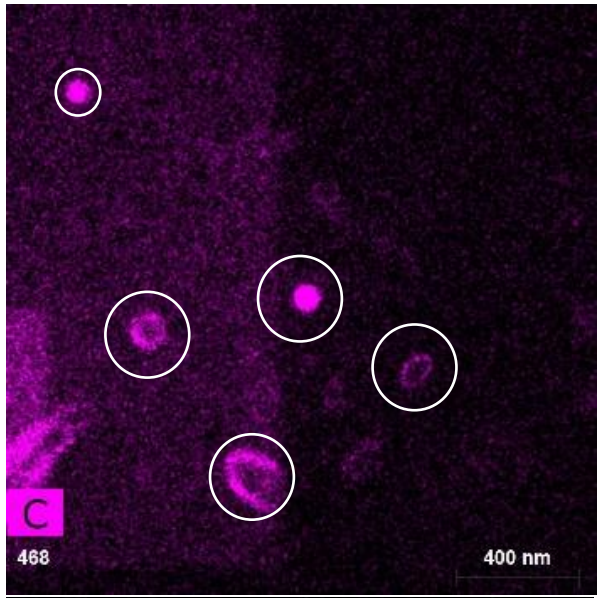
SADP from [011] zone axis

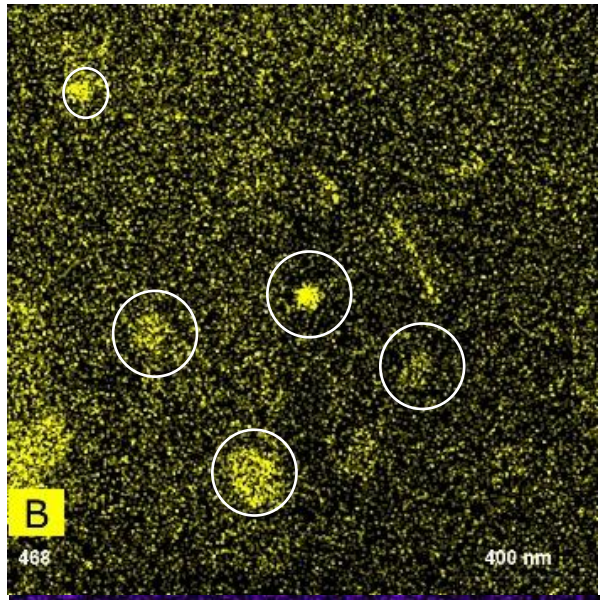
Chemical Analysis of Particles: A6061-RAM(B_4C) (STEM EDS)

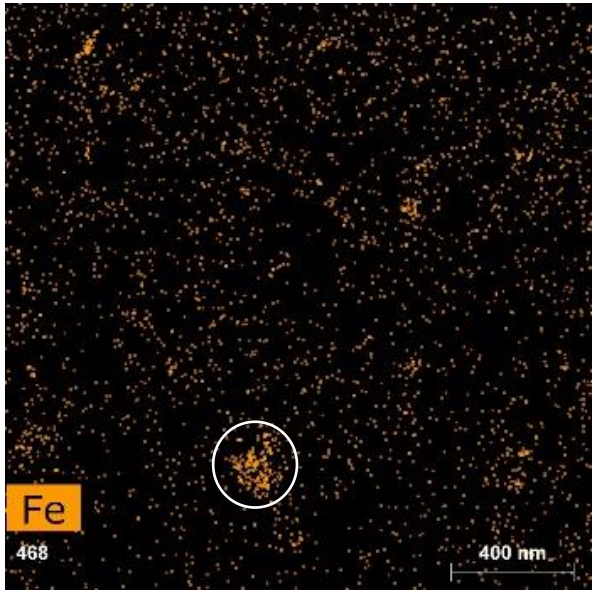
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

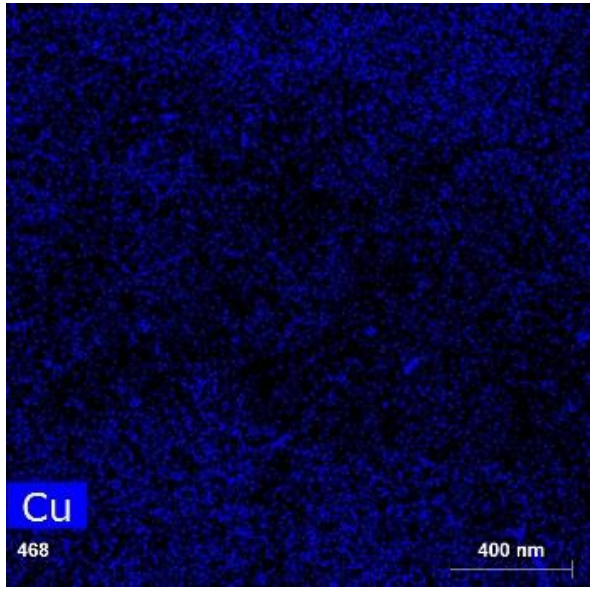


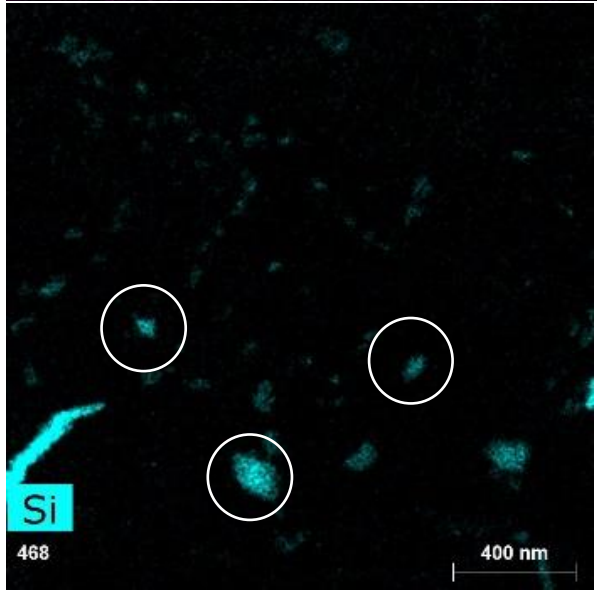


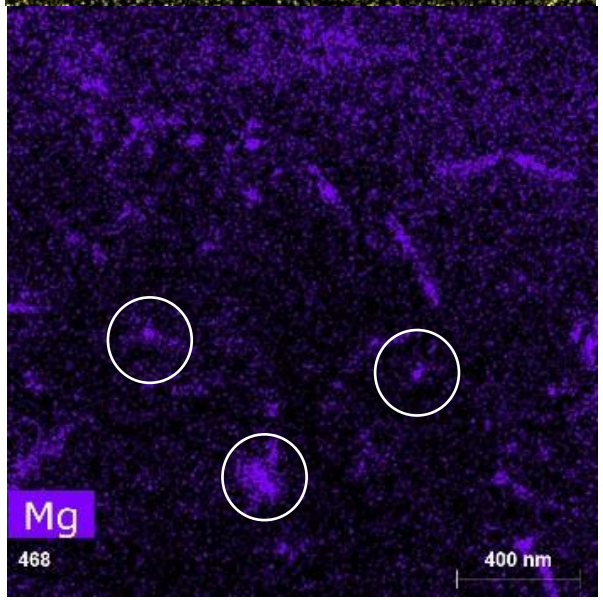










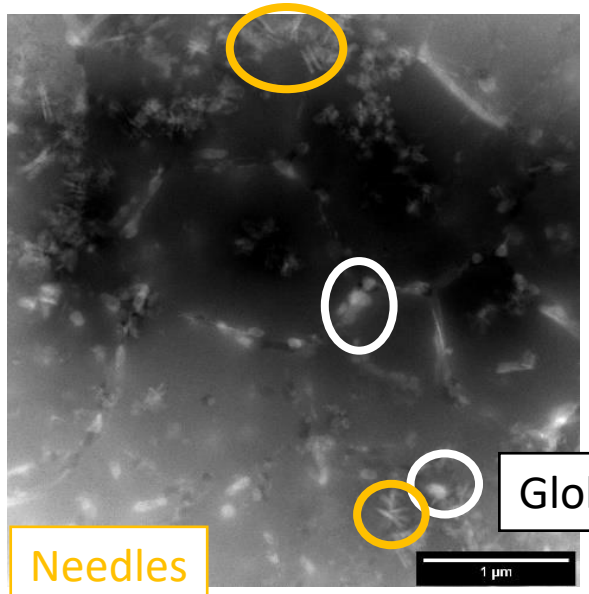


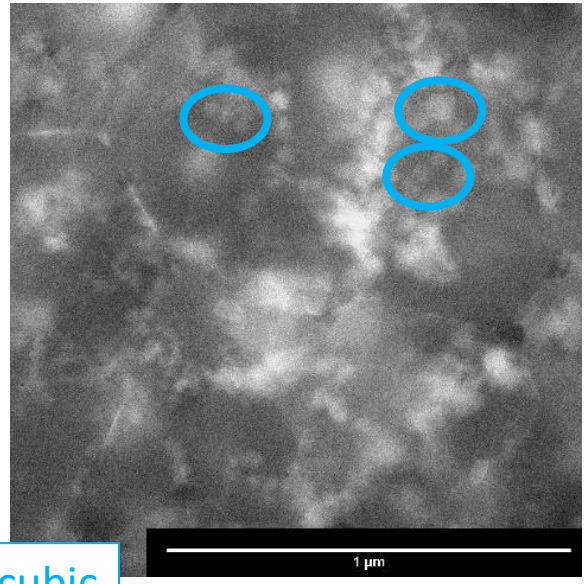
Observed Particle Morphologies A6061-RAM2 & A6061-RAM10 (HAADF STEM)

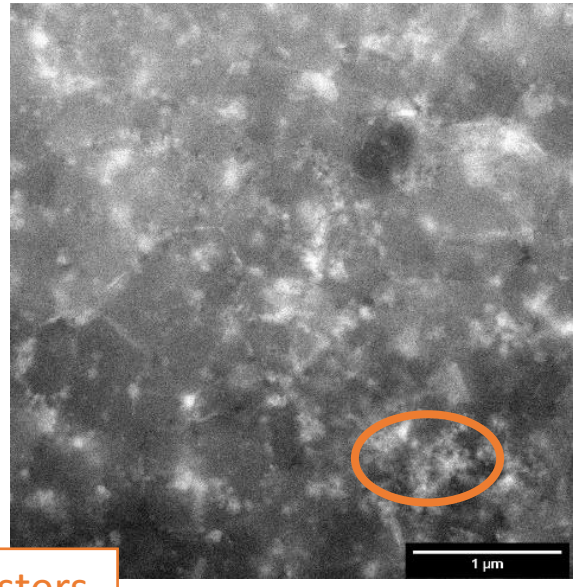
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

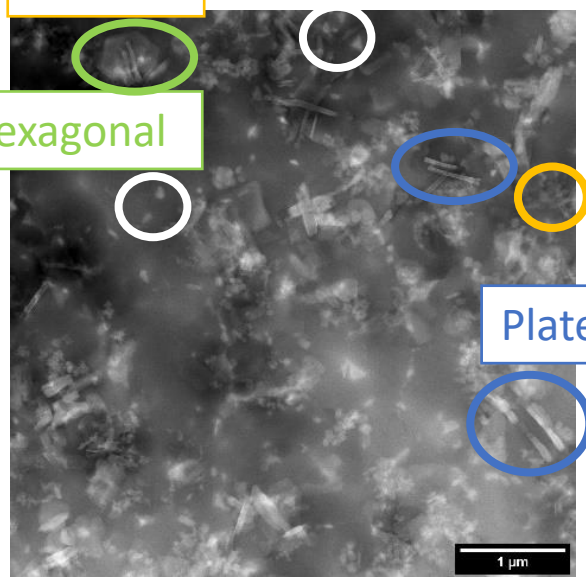
A6061-RAM2

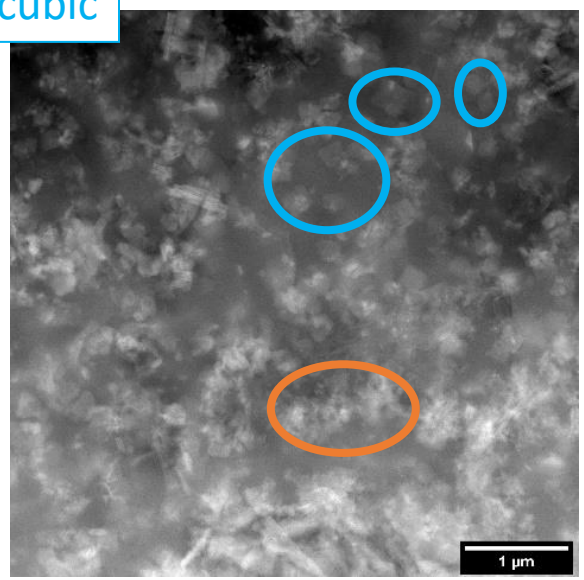
A6061-RAM10

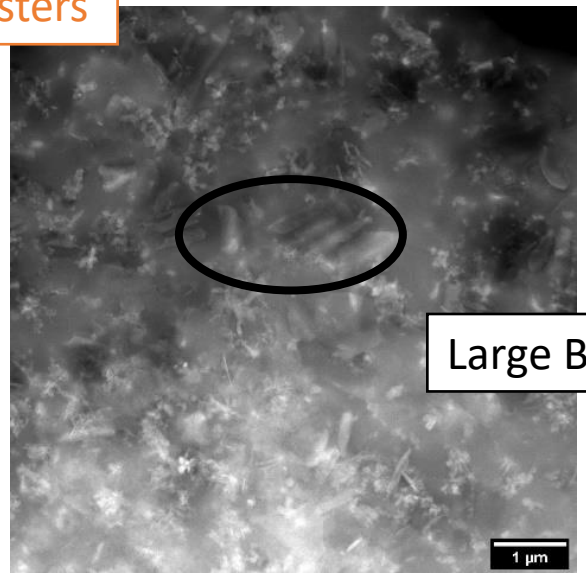






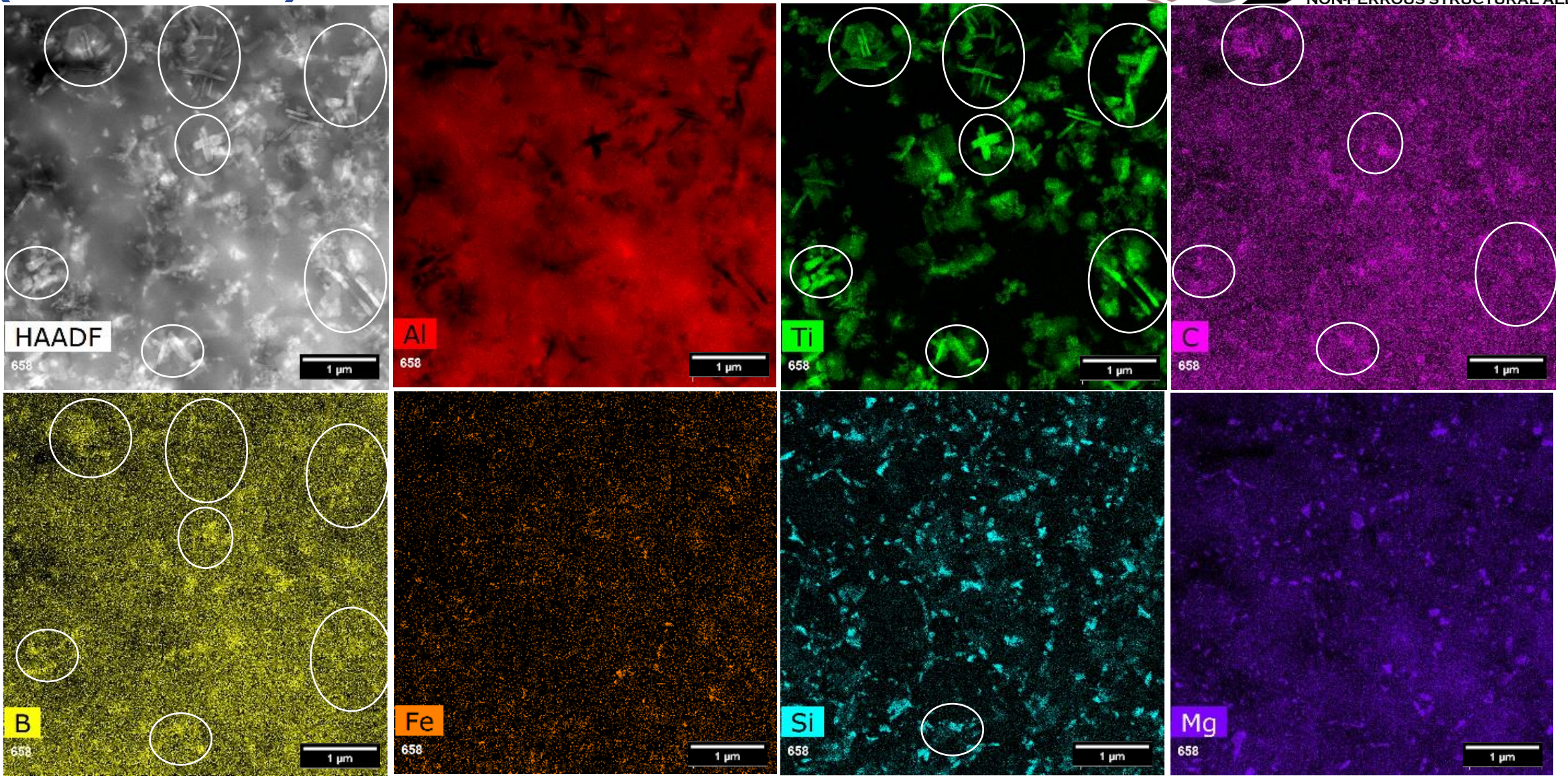






Chemical Analysis of Particles: A6061-RAM10 (STEM EDS)

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS



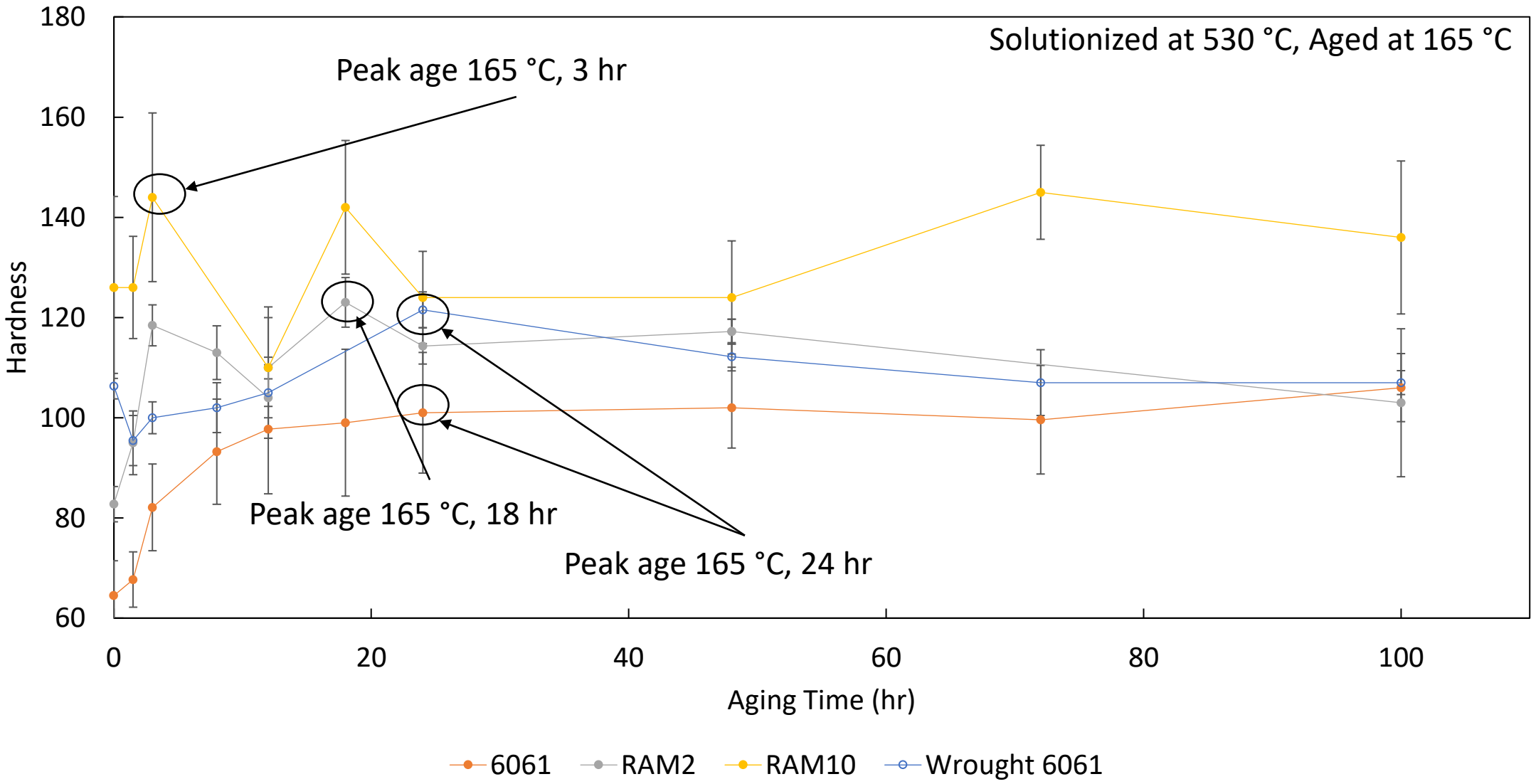
Research Question 3

What is the impact of particle content in as-built A6061-RAM alloys, built using laser powder bed fusion, on aging and precipitation strengthening in these alloys?

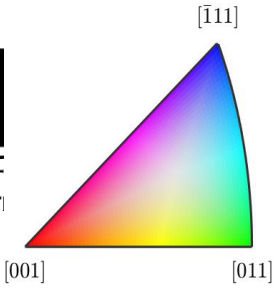
$$\text{Mg \& Si Clusters} \rightarrow \beta''(\text{nm, needle}) \rightarrow \beta'(\text{rod}) \rightarrow \beta$$

Solutionizing Temperature (°C)	Aging Temperature (°C)	Samples Aged in this Condition
530	150	A6061-RAM2, Wrought 6061
530	165	A6061-RAM2 & 10, Wrought 6061, AM 6061
530	175	A6061-RAM2, Wrought 6061
530	190	A6061-RAM2, Wrought 6061
500	165	A6061-RAM2, Wrought 6061

Microhardness for All Alloys Aged at 165°C



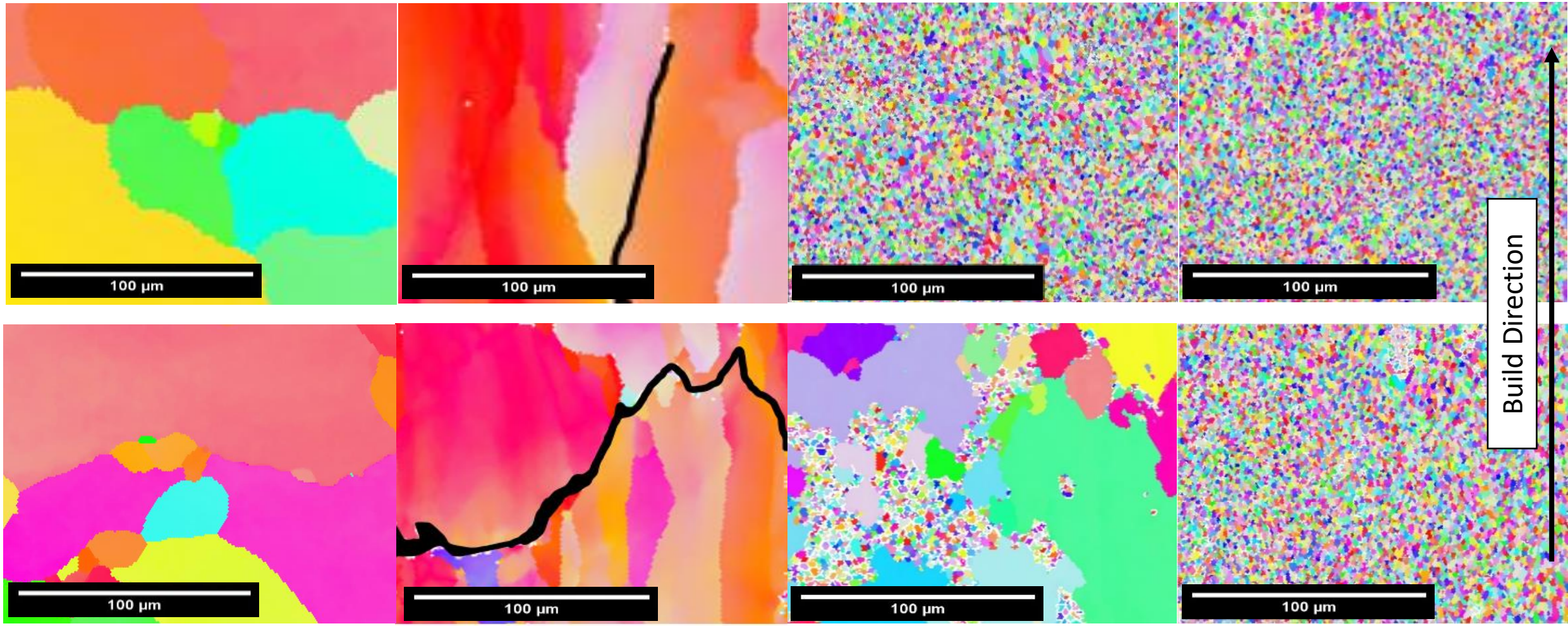
Microstructure of Solutionized Samples



6061 Wrought | AM 6061 | A6061-RAM2 | A6061-RAM10

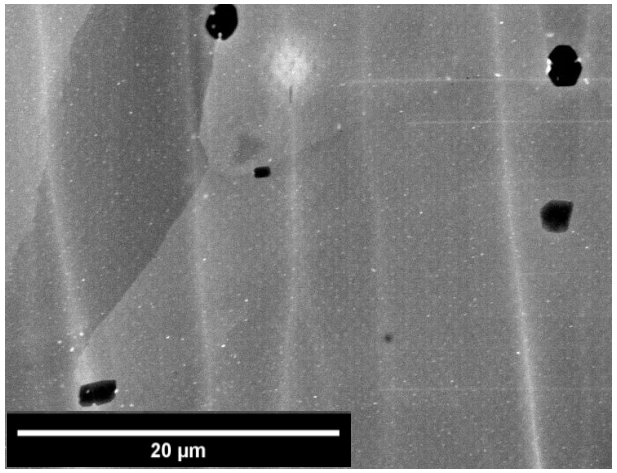
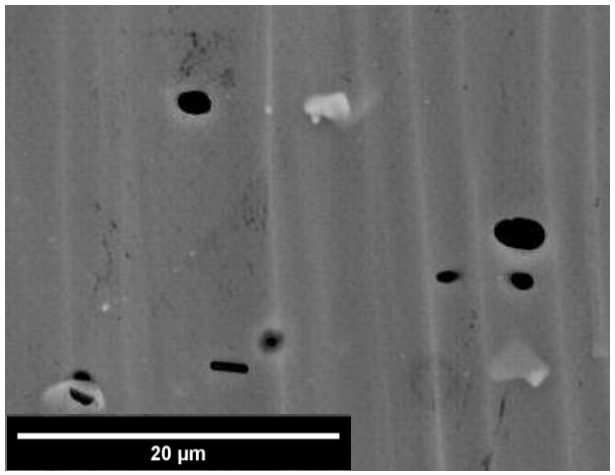
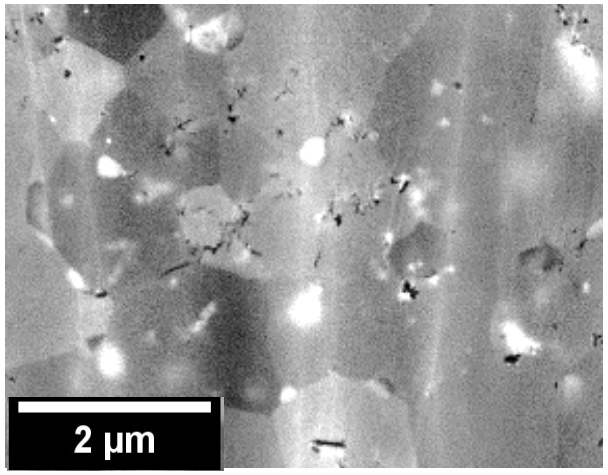
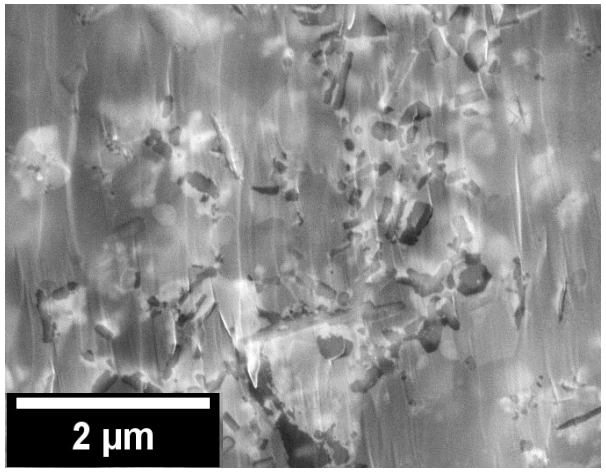
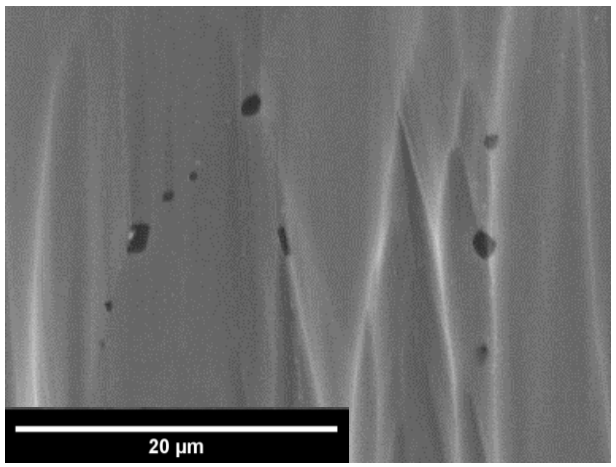
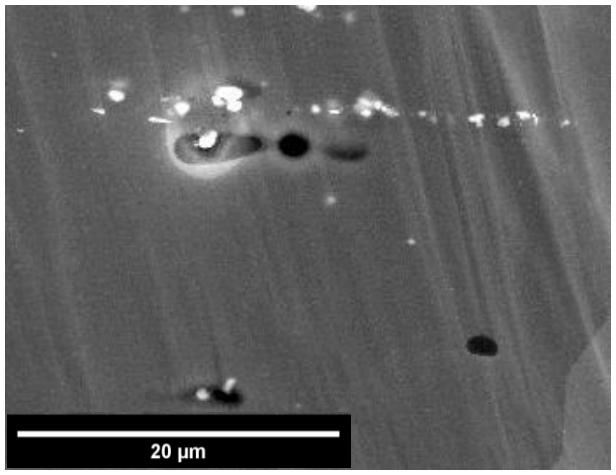
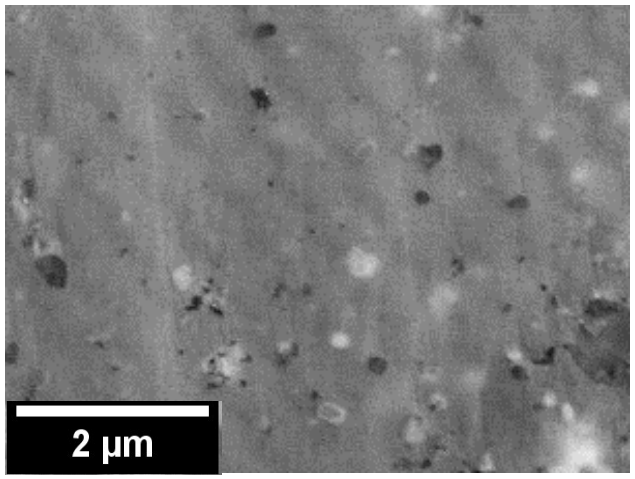
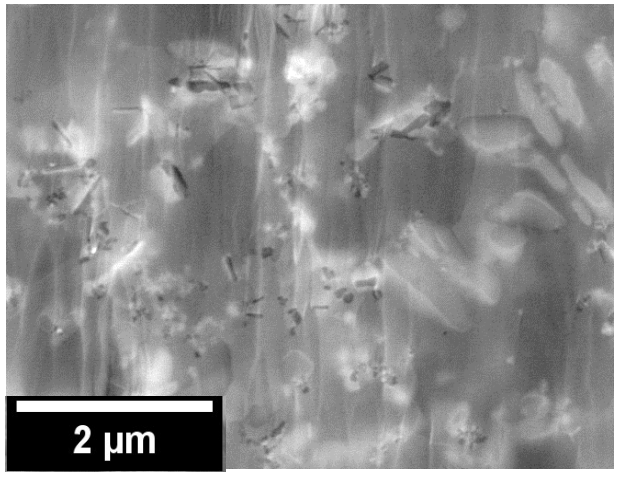
As Received/As Built

Solutionized at 530°C



Microstructural Development of Aged 6061/Wrought 6061 Samples

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

	6061 AM Build	6061 Wrought	A6061-RAM2	A6061-RAM10
Peak Aged 165°C				
Overaged 165°C				

SEM BSE Images

Contributions to Strengthening

- Orowan Strengthening

$$\Delta\sigma_{Orowan} = \frac{\varphi G_m b}{d_p}\left(\frac{6V_p}{\pi}\right)^{\frac{1}{3}}$$

Contribution	Wrought 6061	AM 6061	A6061-RAM2	A6061-RAM10
Orowan (MPa)	-	-	20.9	31.0

Contributions to Strengthening

- Orowan Strengthening

$$\Delta\sigma_{Orowan} = \frac{\varphi G_m b}{d_p}\left(\frac{6V_p}{\pi}\right)^{\frac{1}{3}}$$

- Hall-Petch (Grain size) Strengthening

$$\Delta\sigma_y = kd^{-1/2}$$

Contribution	Wrought 6061	AM 6061	A6061-RAM2	A6061-RAM10
Orowan (MPa)	-	-	20.9	31.0
Hall-Petch, average grain size (MPa)	20.3	26.1	44.1	55.5

Contributions to Strengthening

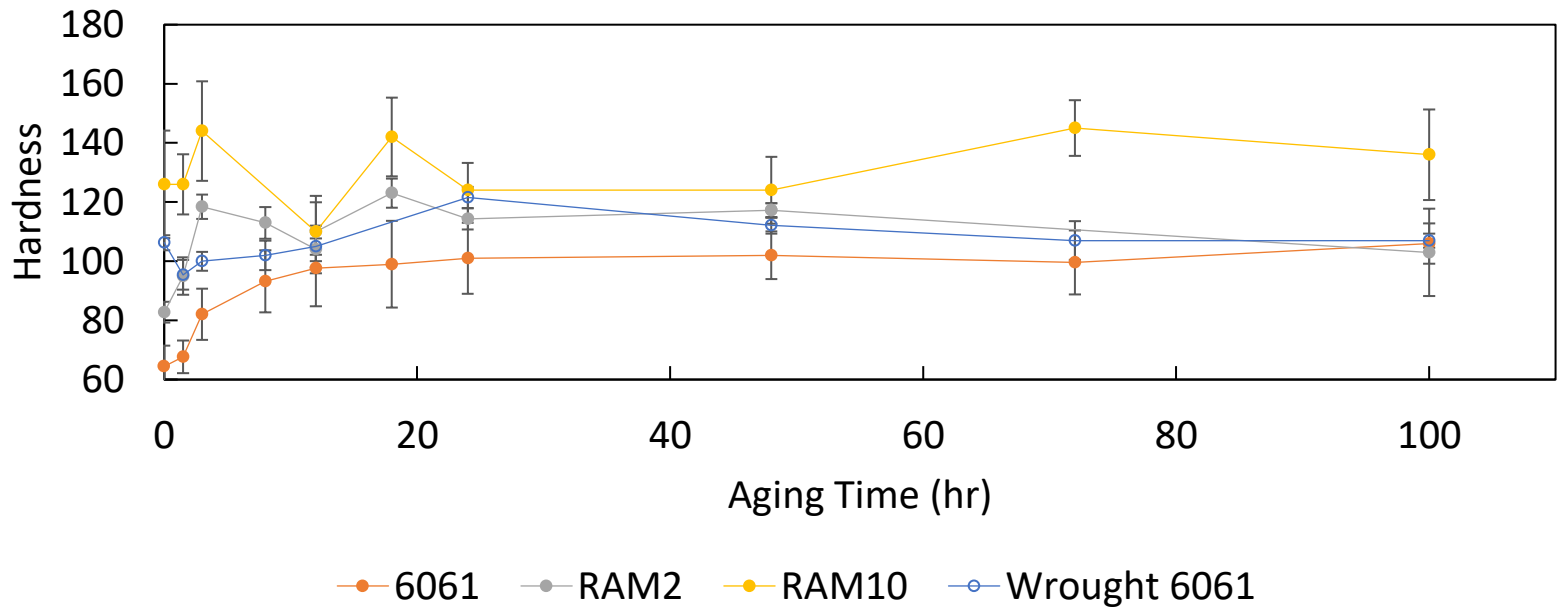
- Orowan Strengthening

$$\Delta\sigma_{Orowan} = \frac{\varphi G_m b}{d_p}\left(\frac{6V_p}{\pi}\right)^{\frac{1}{3}}$$

- Hall-Petch (Grain size) Strengthening

$$\Delta\sigma_y = kd^{-1/2}$$

Contribution	Wrought 6061	AM 6061	A6061-RAM2	A6061-RAM10
Orowan (MPa)	-	-	20.9	31.0
Hall-Petch, average grain size (MPa)	20.3	26.1	44.1	55.5
Hall-Petch max grain size (MPa)	5.1	3.0	6.6	27.3



Contributions to Strengthening

- Orowan Strengthening

$$\Delta\sigma_{Orowan} = \frac{\varphi G_m b}{d_p}\left(\frac{6V_p}{\pi}\right)^{\frac{1}{3}}$$

- Hall-Petch (Grain size) Strengthening

$$\Delta\sigma_y = kd^{-1/2}$$

- Load Bearing of Reinforcing Phase

$$\Delta\sigma_{load} = 1.5V_p\sigma_i$$

Contribution	Wrought 6061	AM 6061	A6061-RAM2	A6061-RAM10
Orowan (MPa)	-	-	20.9	31.0
Hall-Petch, average grain size (MPa)	20.3	26.1	44.1	55.5
Hall-Petch max grain size (MPa)	5.1	3.0	6.6	27.3

Summary & Conclusions

- For the laser parameters studied in in-situ imaging, all A6061-RAM2 melts achieved same level of refinement (avg. grain size ~2-3 µm) and show no signs of hot tearing
- Addition of Ti and/or B_4C refines microstructure compared to 6061, full refinement achieved in A6061-RAM2, A6061-RAM10, and A6061-RAM(Ti) and smallest grain size in A6061-RAM(Ti)
  - SiC, Ti borides and carbides, and Al_3Ti down to ~100 nm all served as inoculants, Al_3Ti refining most effectively for amount of initial reactive particles
  - Imply wider range of inoculant types and sizes can be used in AM vs. casting, and wider design space for consideration of which particle content and type could be used to achieve certain mechanical properties
  - Shape of refined regions in samples with CET suggest refinement is less dependent on solidification conditions and more dependent on where reactant particles, and thus solute, is distributed in the melt
- Formation of Mg_2Si precipitates during aging increases hardness of all alloys, however nucleation sites in all alloys studied (with a higher amount observed in RAM alloys) leads to coarser particles than expected β'' strengthening precipitate
- Orowan and Hall-Petch strengthening were estimated to have a large contribution towards strength in the A6061-RAM alloys and most likely account for the higher hardness values, with Hall-Petch providing more significant contribution

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Questions?

Thank you for listening!

chloe@elementum3d.com