Project 30: Microstructural Development of Metallic Alloys during Rapid Solidification

Fall 2019 Semi-Annual Meeting
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Other Participants: Gus Becker (Mines), Jonah Klemm-Toole (Mines), Yaofeng Guo (Mines), Francisco Coury (UFSCAR), Joe Mckeown (LLNL)

Project 30: Microstructural Evolution of Metallic Alloys during Rapid Solidification

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

Project Duration
PhD: August 2017 to May 2021

- Problem: Rapid solidification results in novel as-solidified microstructures, the evolution of which is not well understood.
- Objective: Understand the correlation of rapid solidification conditions to the development of metastable phases, novel microstructures, and grain morphology effects in aluminum alloys.
- Benefit: Inform microstructural prediction to improve alloy design and solidification conditions for rapid solidification processes (e.g. additive manufacturing (AM)).

Recent Progress

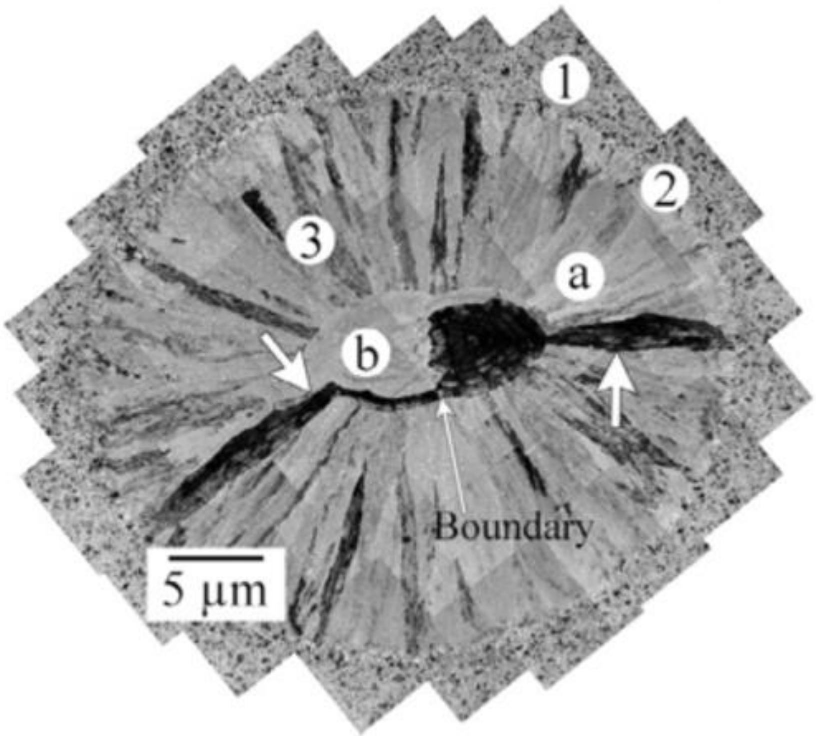
- Initial image processing of in-situ solidification experiments performed using the Additive Manufacturing (AM) simulator at the Advanced Photon Source (APS) at Argonne National Lab (ANL)
- Top-down characterization of 6061 Reactive Metal Powder (RAM) alloy designed for AM by Elementum 3D & wrought 6061 from experiments performed using the AM simulator at the APS at ANL
- Phase identification and investigation of microstructural selection in Al-Ge samples generated using dynamic transmission electron microscopy (DTEM)
- Preparing for qualifying exam in October 2019

Metrics

Description	% Complete	Status
1. Literature review	50%	●
2. In/ex-situ characterization of metastable phase formation in rapidly solidified, faceting Al-Ge alloys	85%	●
3. In/ex-situ investigation of grain morphology and size in Al 6061 and 6061 RAM alloys	15%	●
4. Evaluation of effect of solidification conditions on microstructural development in RAM AM builds (performed in collaboration with Elementum 3D)	5%	●

Industrial Relevance

- Solidification conditions have a significant impact on final microstructure
- Rapid solidification generates novel microstructures, metastable phases, and a variety of grain morphologies
- Binary/model alloys present an opportunity to understand these novel microstructures on a fundamental level that can serve to inform industrial processes



Post-mortem TEM image of Al-Cu alloy film after DTEM solidification.

J.T. McKeown *et al.*, *Acta Materialia,* 65 (2014) 56-68.

Industrial Relevance

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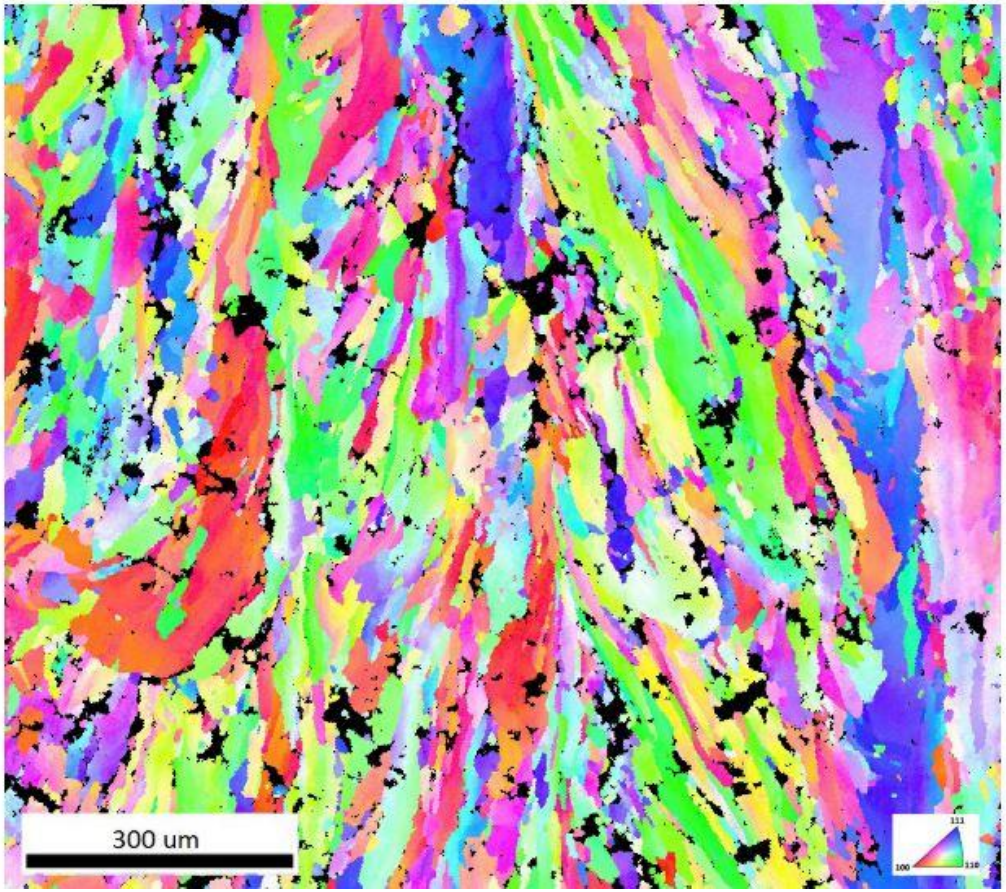
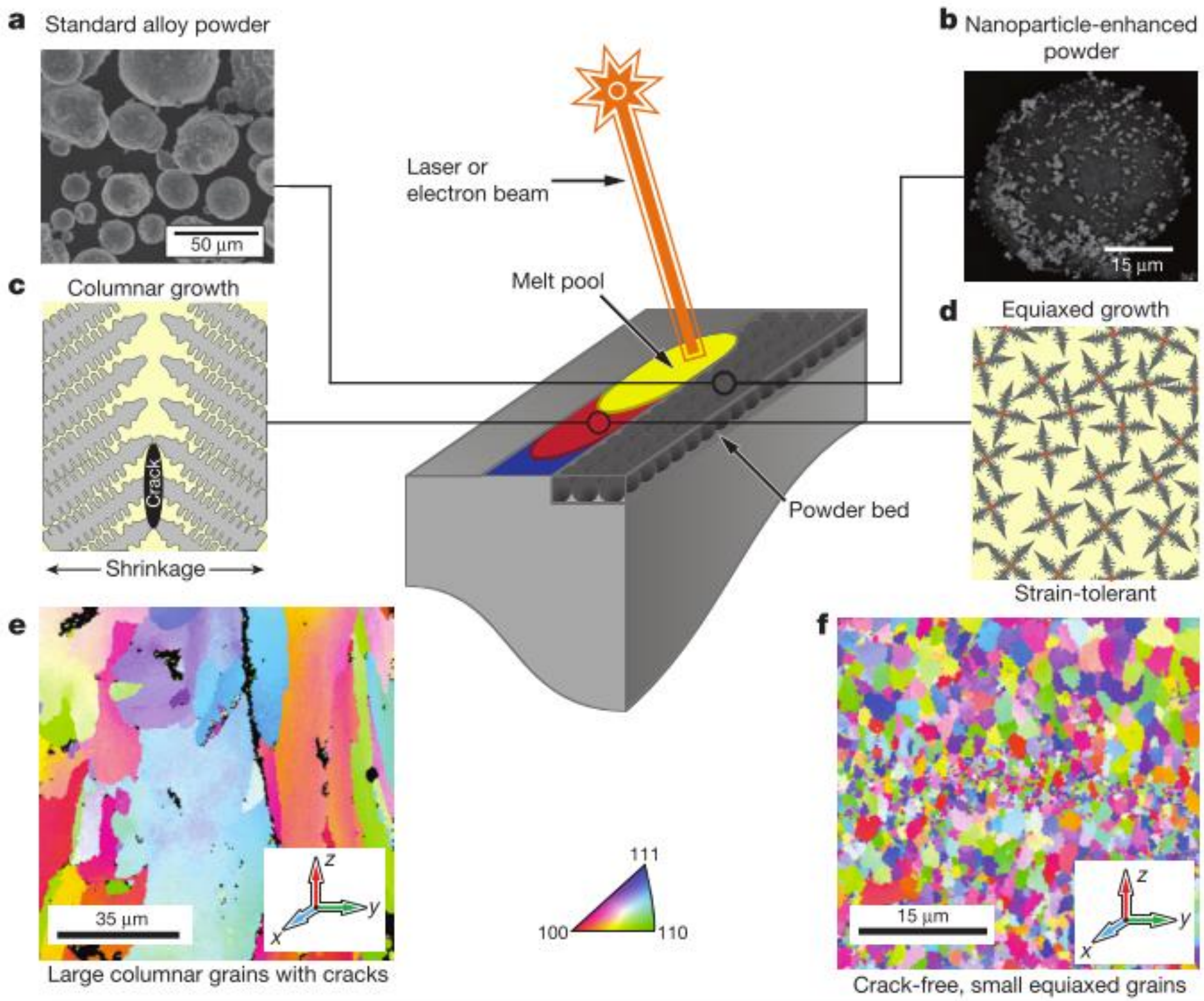


Inverse pole figure of 3D-printed stock 7075, build direction is vertical to the page. Taken from J. H. Martin et al. *Nature,* 549 (2017) 365-369.

- Aluminum alloys currently used in AM are mostly traditional stock alloys (e.g. 7075, 6061, 2024)
- Under AM conditions these 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



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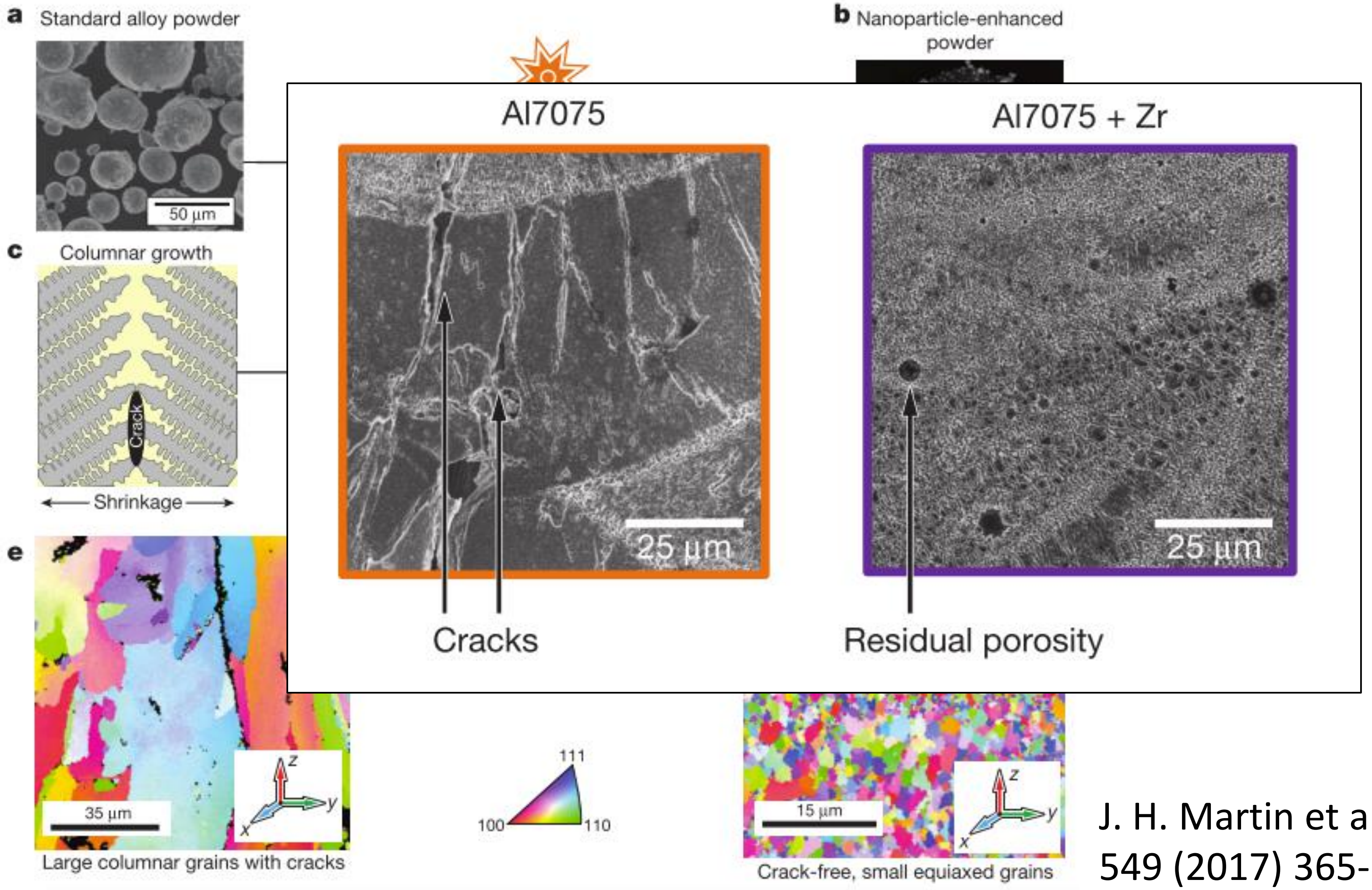
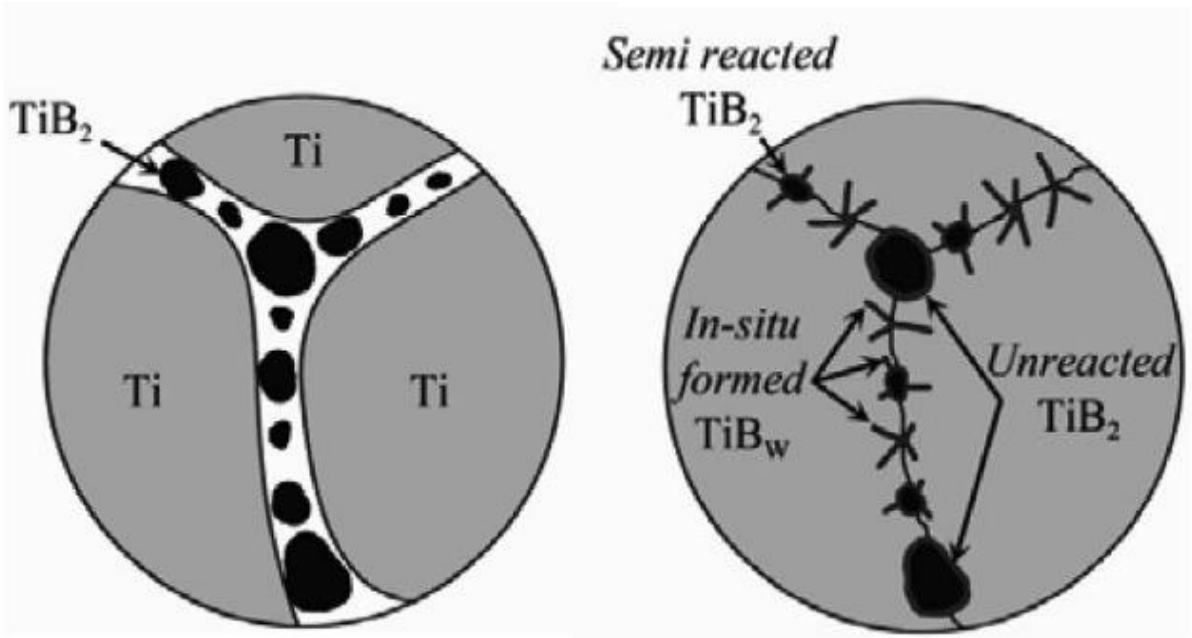


J. H. Martin et al. *Nature,* 549 (2017) 365-369.

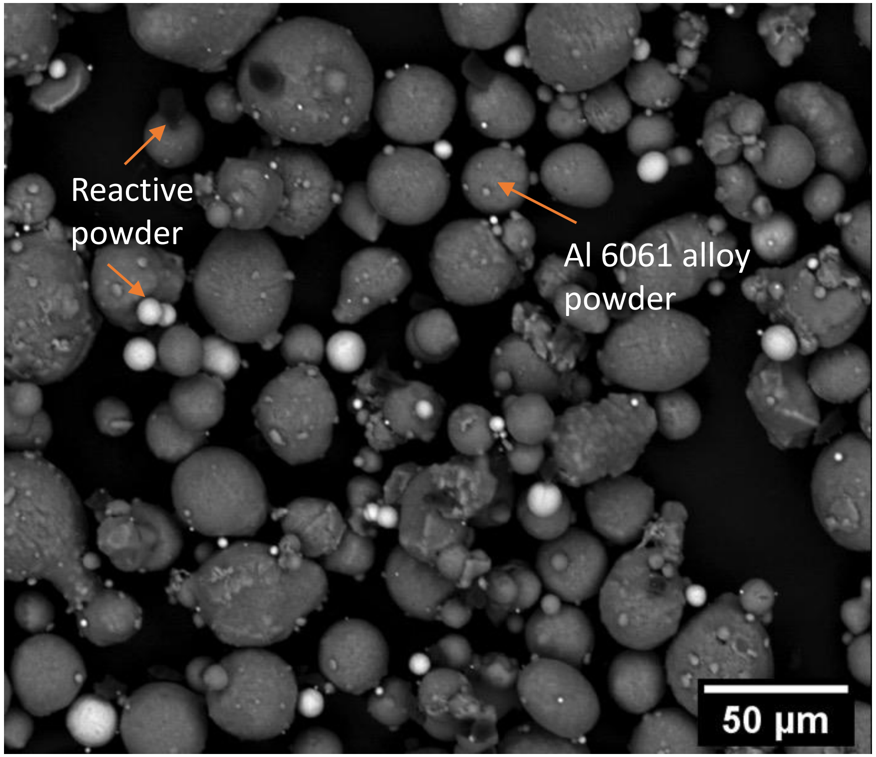
Reactive Metal Powder (RAM) Alloys for AM



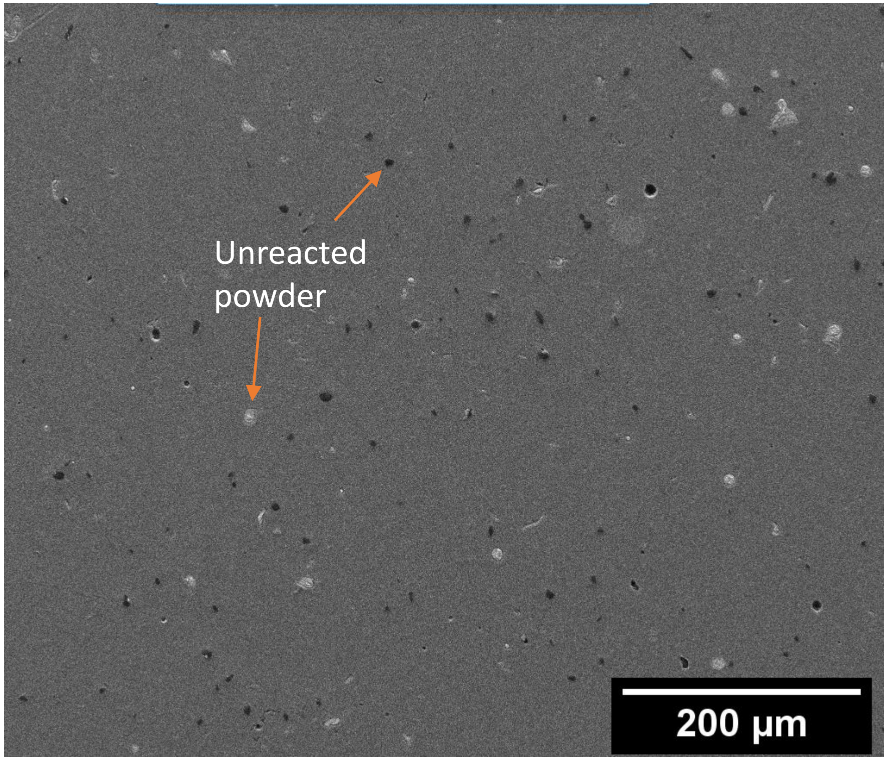
Schematic of in-situ MMC/RAM process for Ti matrix and TiB_2 particles. Taken from H. Attar et al, *International Journal of Machine Tools and Manufacturing,* 133 (2018) 85-102.

- Also known as in-situ MMC alloys
- Powders of two different components react with each other (or the matrix) to form inoculant particles
- Advantages of RAM:
  - Uniform distribution
  - Enhanced interfacial bonding
  - Can pin grain boundaries

Al 6061 Reactive Metal Powder (RAM) Alloy Designed for AM: Initial Characterization

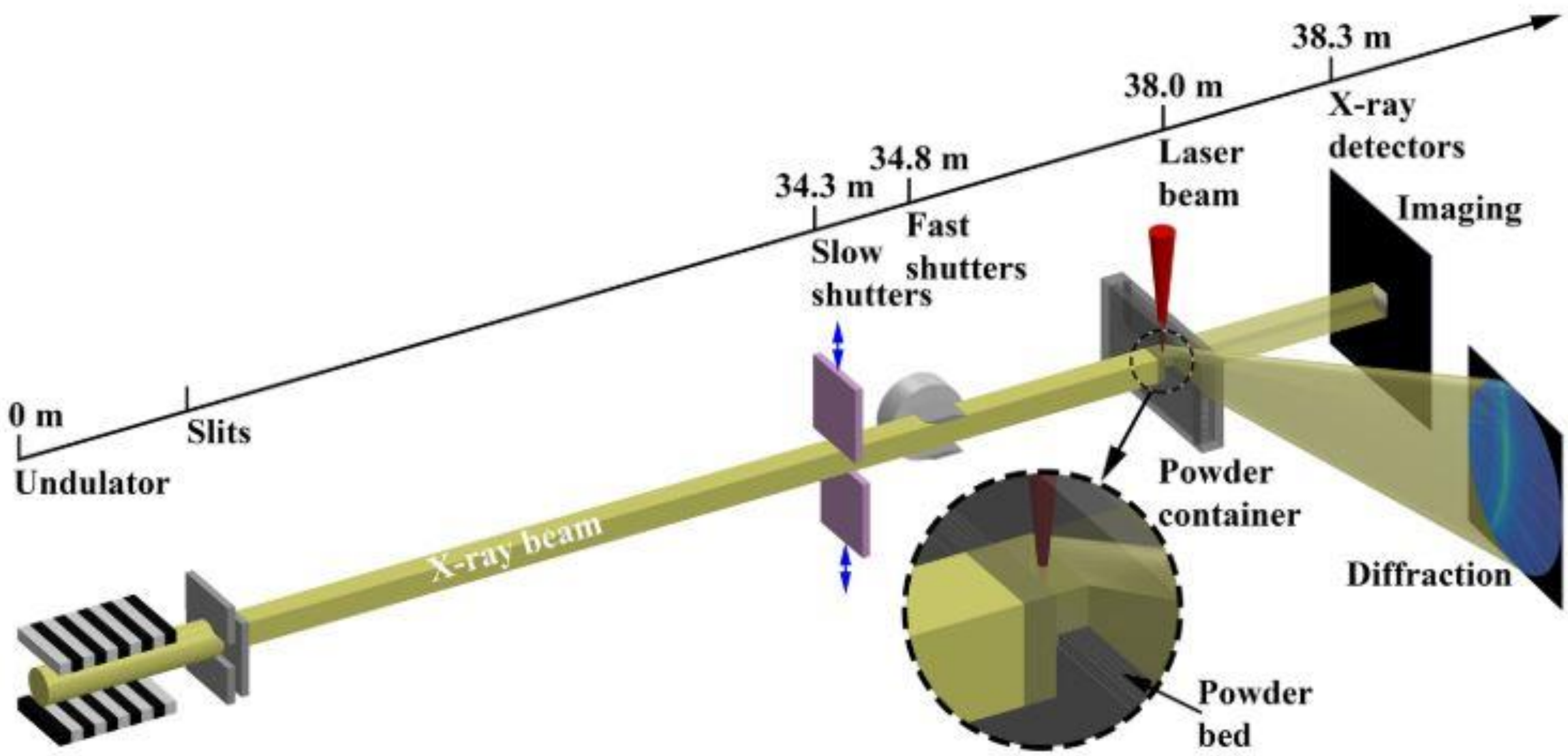


BSE SEM image of Al 6061 RAM alloy powder



SEM image of Al 6061 RAM AM build

APS Additive Manufacturing Simulator Set-up



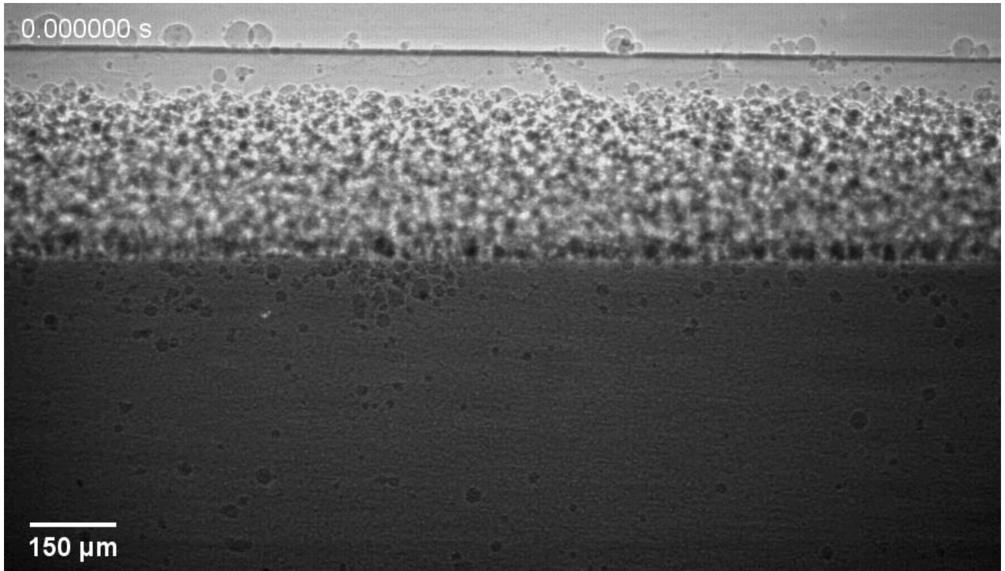
Schematic of AM simulator used for in-situ experiments at ANL.
Taken from: C. Zhao et al., *Scientific Reports,* 7 (2017) 1-11.

Selected Samples & Laser Parameters

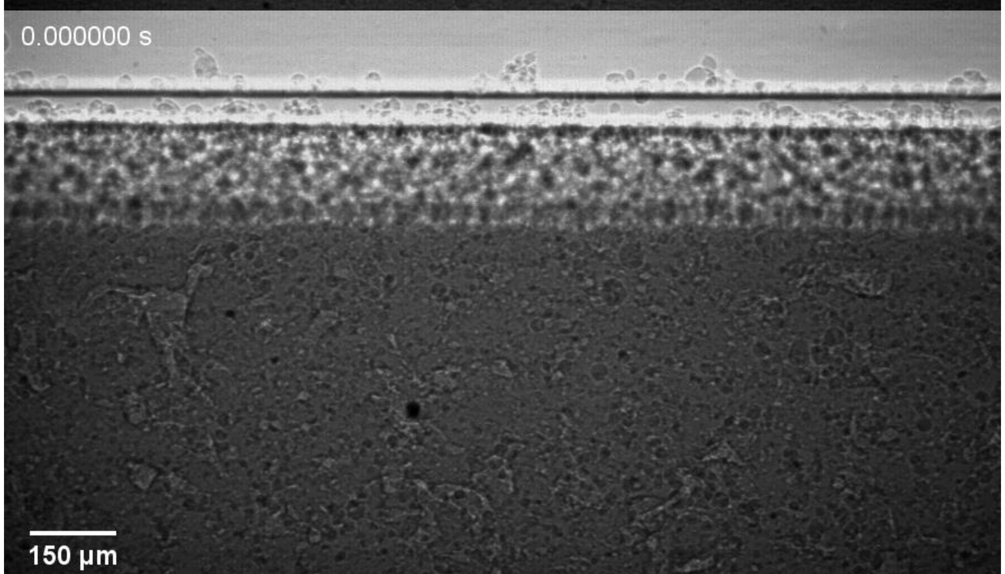
- Samples were made to be 1-1.5 mm thick and 40-50 mm in length
- Experiments were performed on 3 types of samples:
  - Al 6061 wrought base plate with 6061 powder
  - Al 6061 wrought base plate with 6061 RAM powder
  - Al 6061 RAM AM build base plate with 6061 RAM powder
- The same iterations of laser passes were performed on each of these samples:

Experimental parameters for laser passes		
Iteration	Laser Power (W)	Laser Speed (m/s)
1	416	0.3
2	416	0.5
3	520	0.3
4	520	0.5

APS AM Simulator Animations: Al 6061 MMC Alloy vs. Wrought 6061

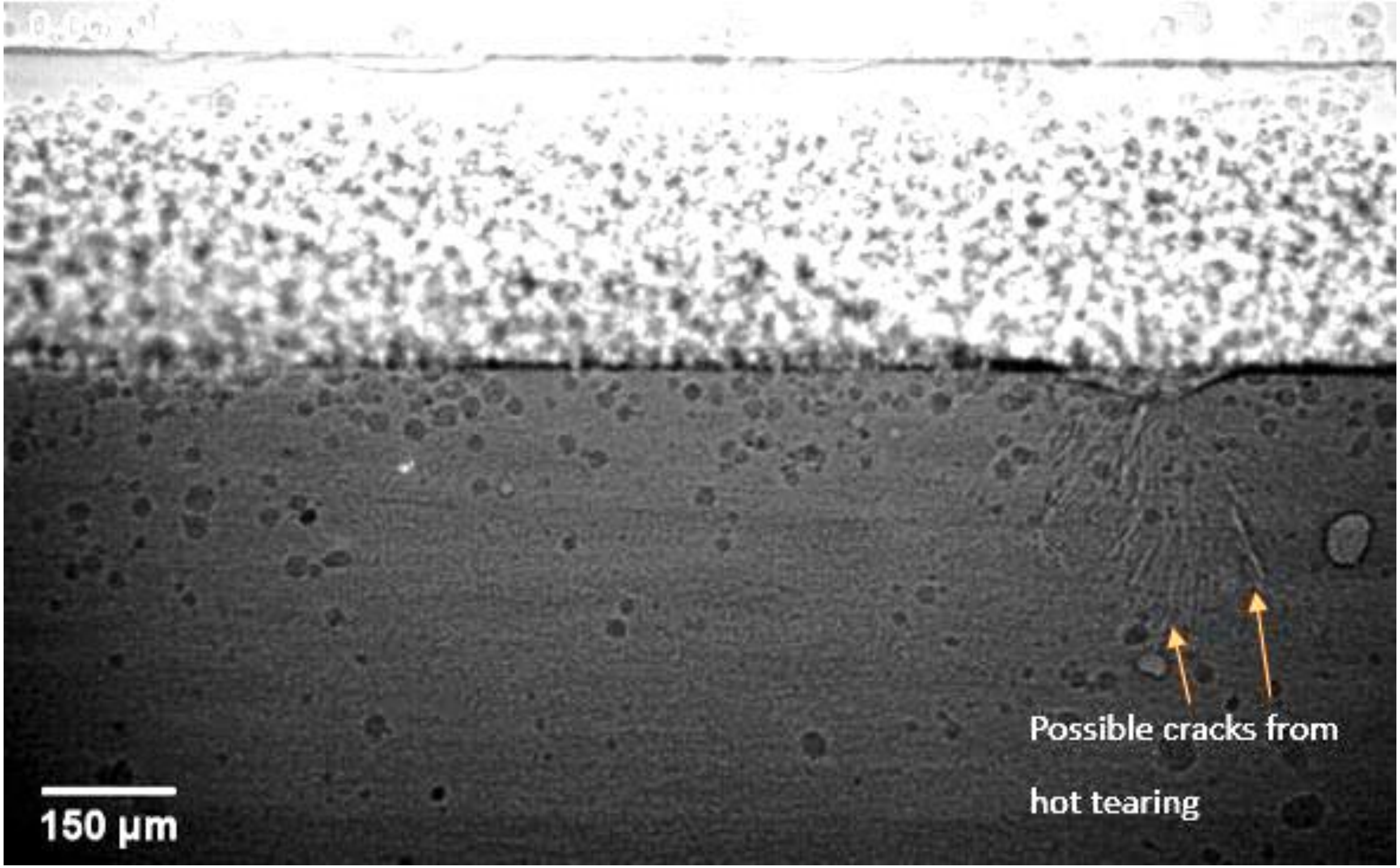


Animation of laser pass on 6061 wrought + 6061 powder, 416 W, 0.5 m/s



Animation of laser pass on Al 6061 RAM build + Al 6061 RAM powder 416 W, 0.5 m/s

Possible Solidification Cracking Observed in Al 6061



Radiography image of laser pass on 6061 wrought + 6061 powder (laser settings of 416 W, 0.5 m/s) showing possible cracking immediately following solidification.

Top Down View Imaging of 6061 & 6061 RAM Alloys

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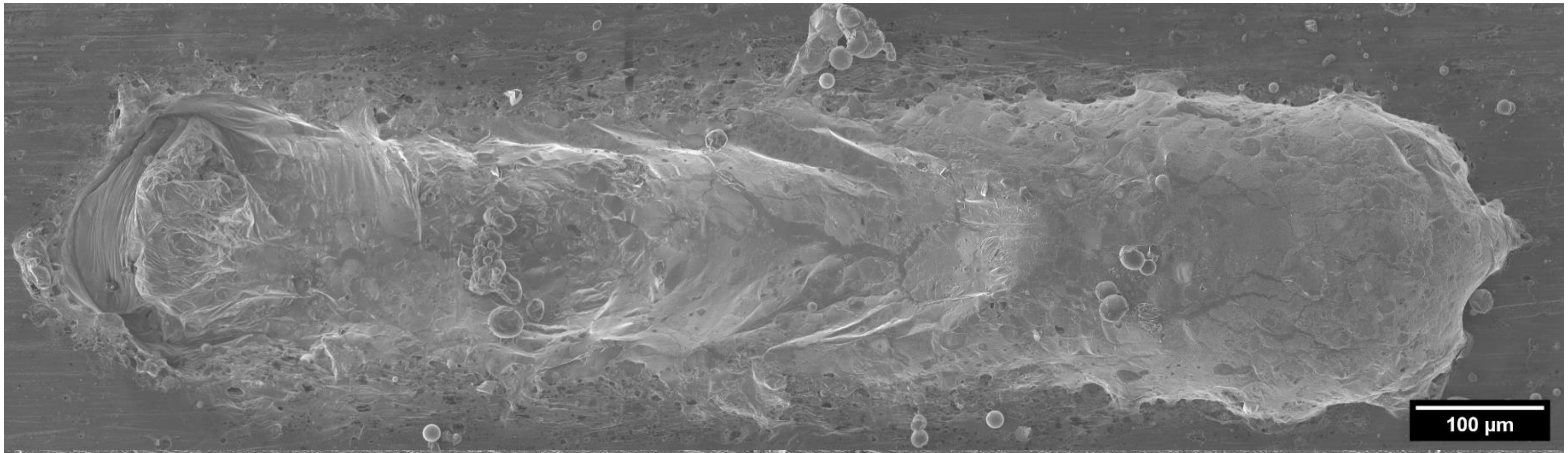
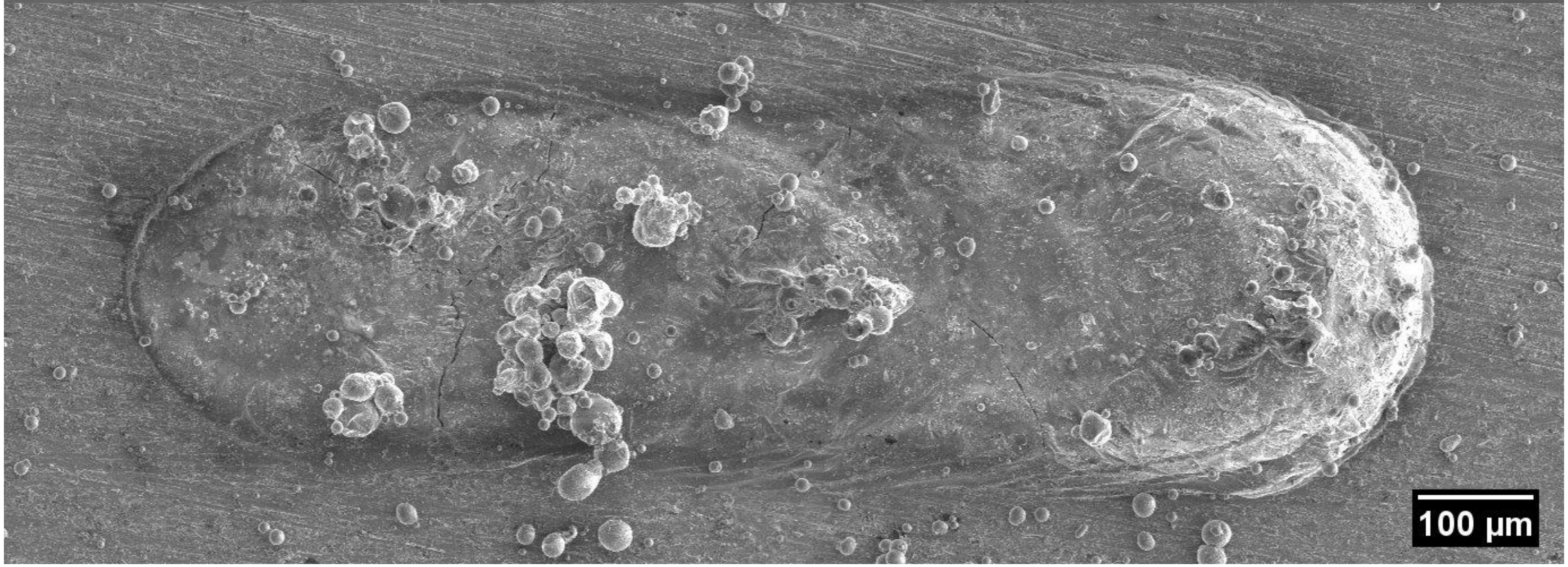
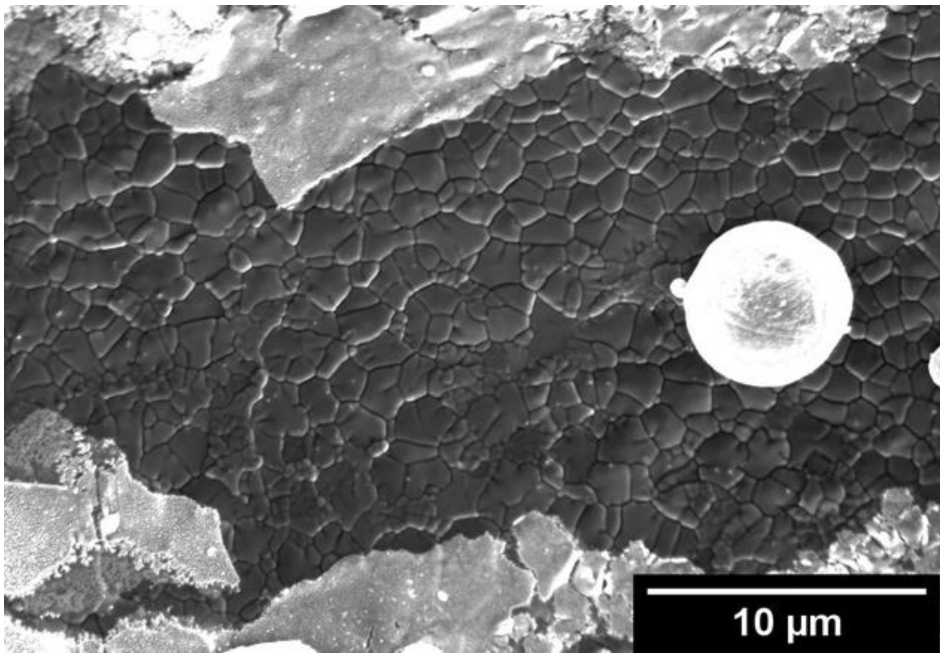


Secondary electron SEM image of laser raster of Al 6061 RAM build + Al 6061 RAM powder
416 W, 0.5 m/s

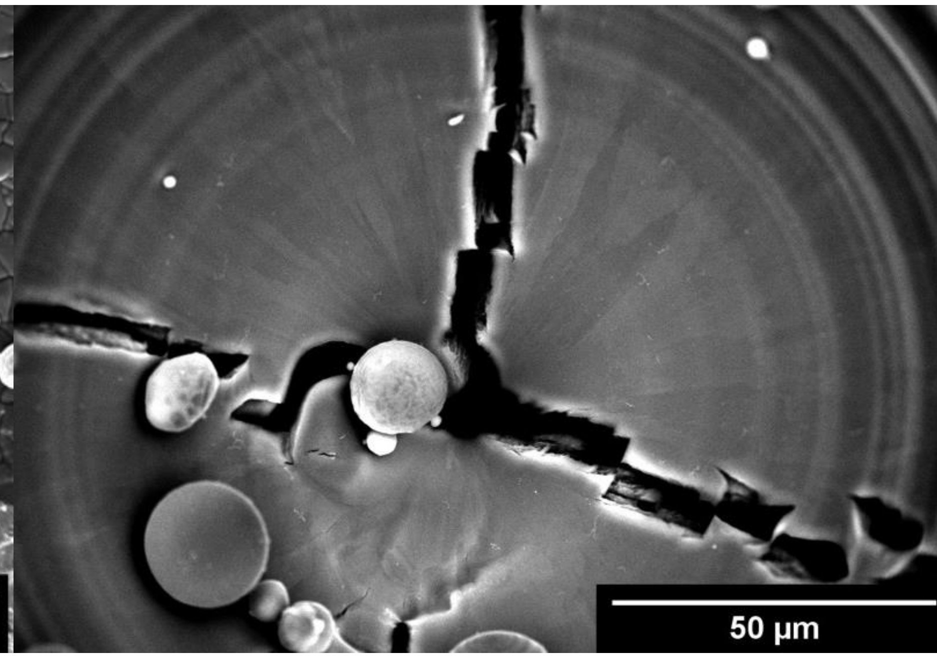


Secondary electron SEM Image laser raster of 6061 wrought + 6061 powder, 416 W, 0.5 m/s

Observed Grain Structure in Top Down View Images

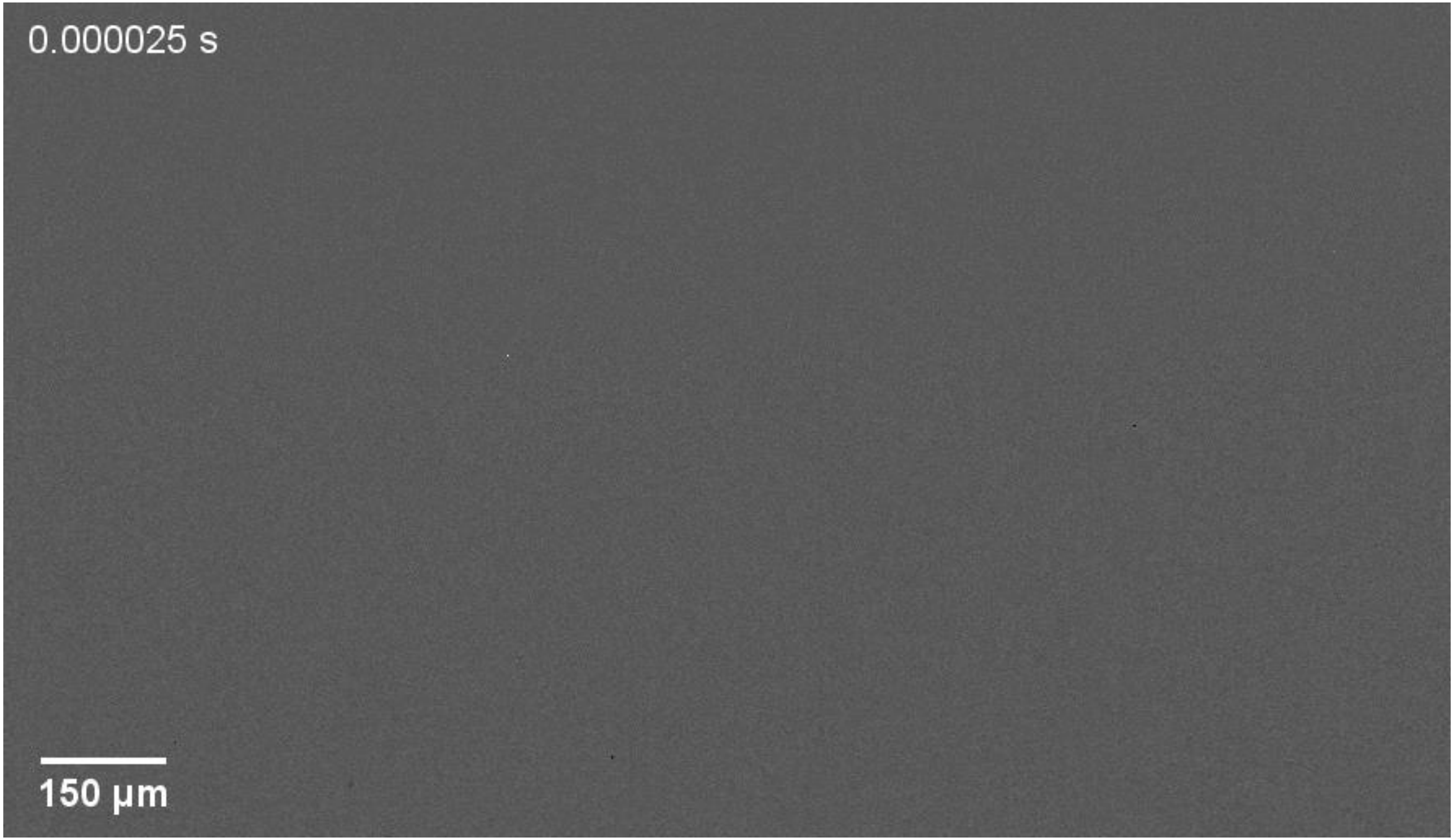


Secondary electron SEM image of laser raster of Al 6061 RAM build + Al 6061 RAM powder, 416 W, 0.3 m/s



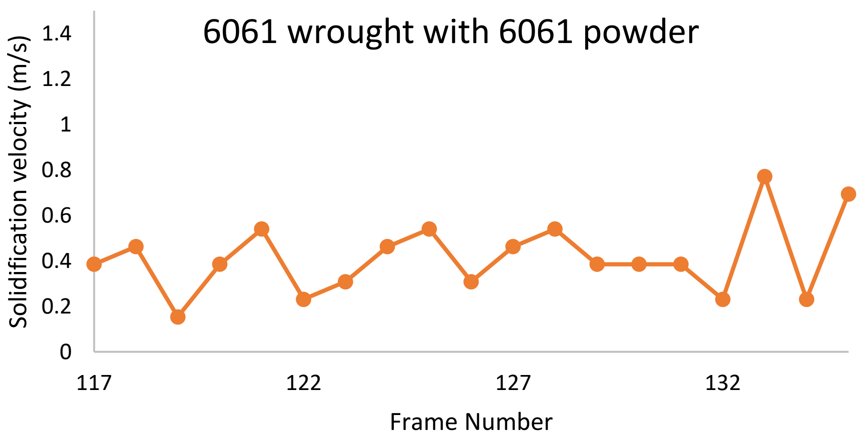
Backscatter SEM image of laser spot weld of 6061 wrought + 6061 powder, 520 W, 2 ms dwell time

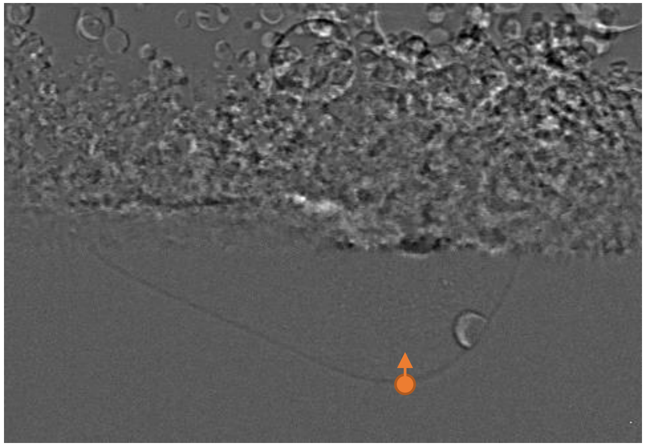
Image Processing: Tracking of S/L Interface



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

Solidification Velocity Measurements





Radiography image of melt pool and point used for velocity measurements

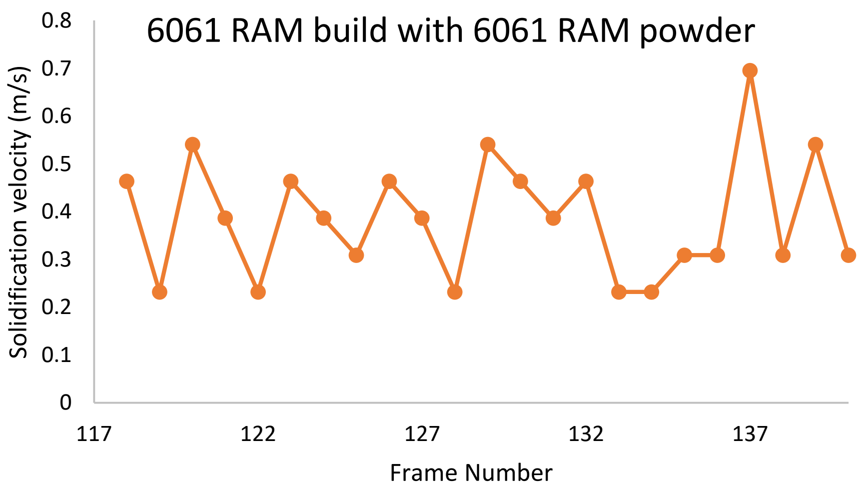


Table 30.1: Average velocity and standard deviation values for each material

Base Plate	Powder	Average velocity (m/s)	Standard deviation
6061 wrought	6061	0.40	0.1654
6061 RAM AM build	6061 RAM	0.39	0.1226

Future Work

- Identify grain morphology (columnar vs. equiaxed) in the cross-sections of these melt pools
- Correlate observed microstructures in samples to solidification conditions
- Use initial data as a stepping stone to plan future, more complex, experiments

Metastable Monoclinic Phase in Al-Ge Alloys: Our Interests

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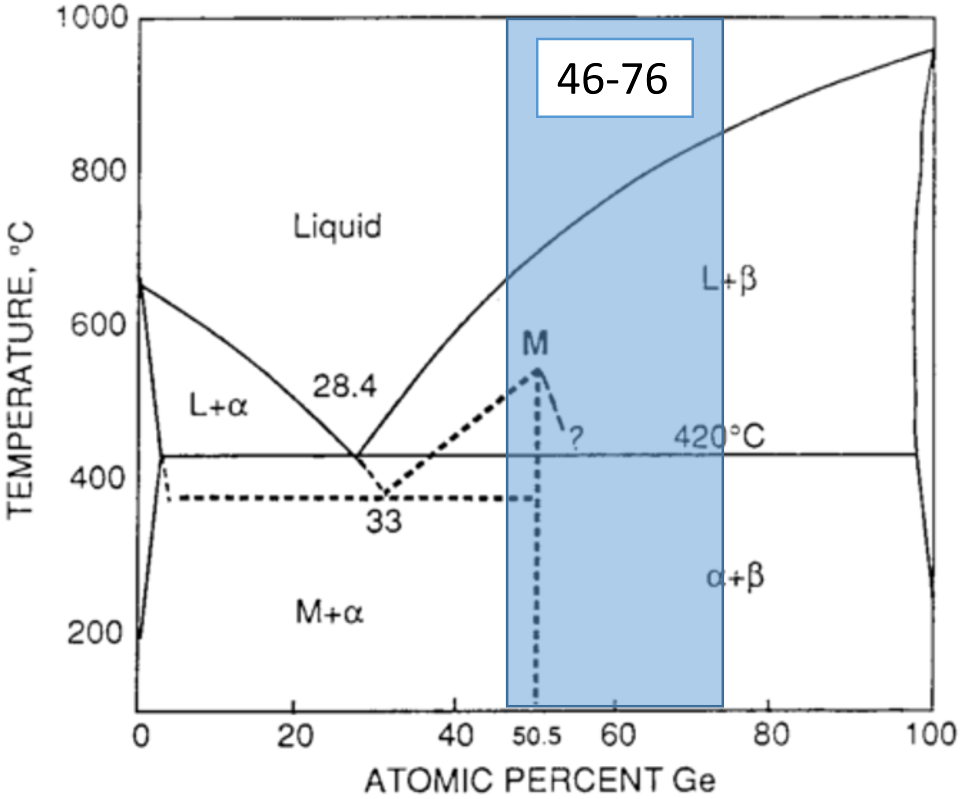
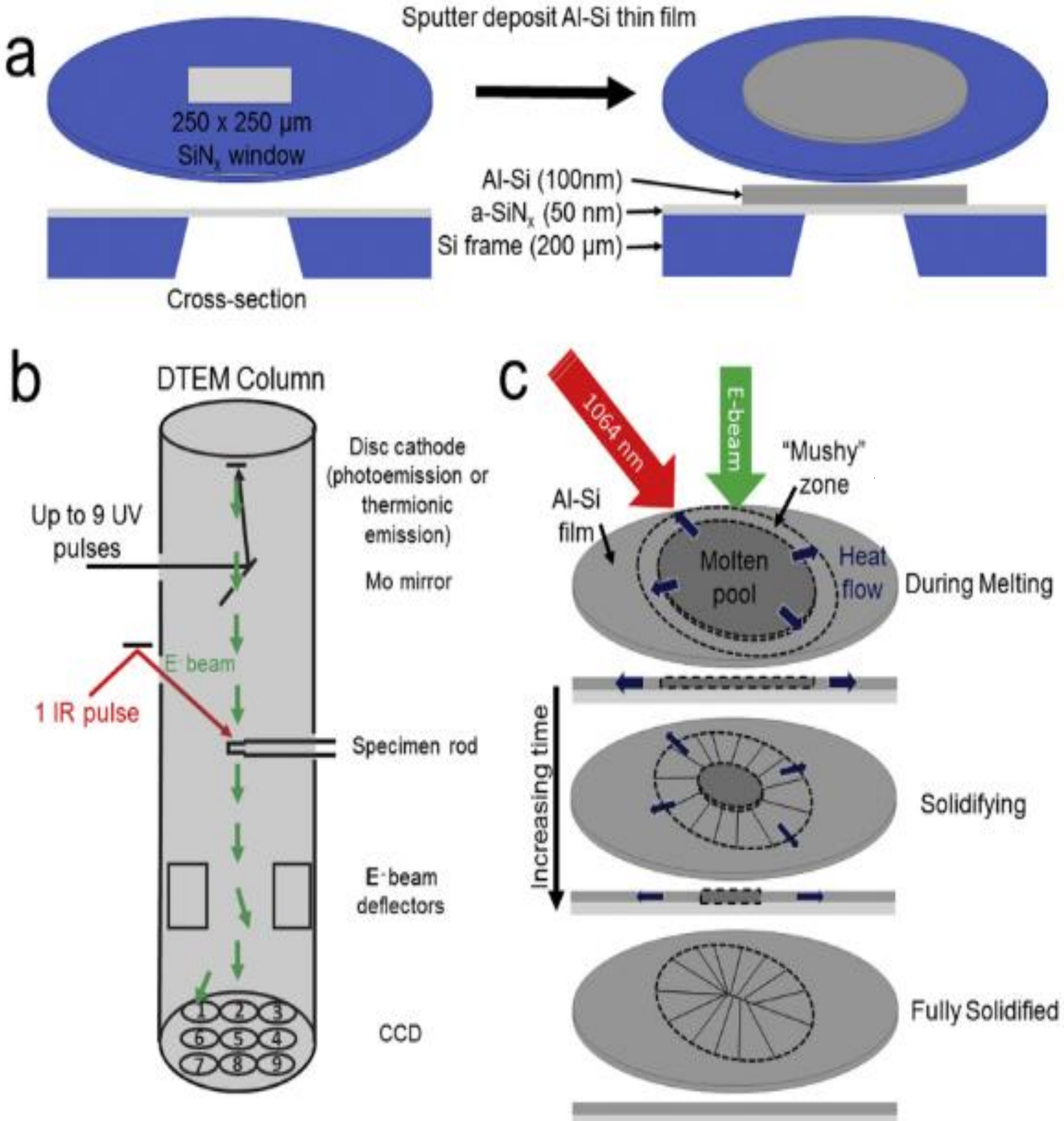
- Explore proposed metastable phase diagram that includes M phase
- Samples ranged from 46-76 at.%Ge



Proposed metastable phase diagram for monoclinic (M) phase.
T. Laoui & M. J. Kaufman., *Met Trans A* (1991).

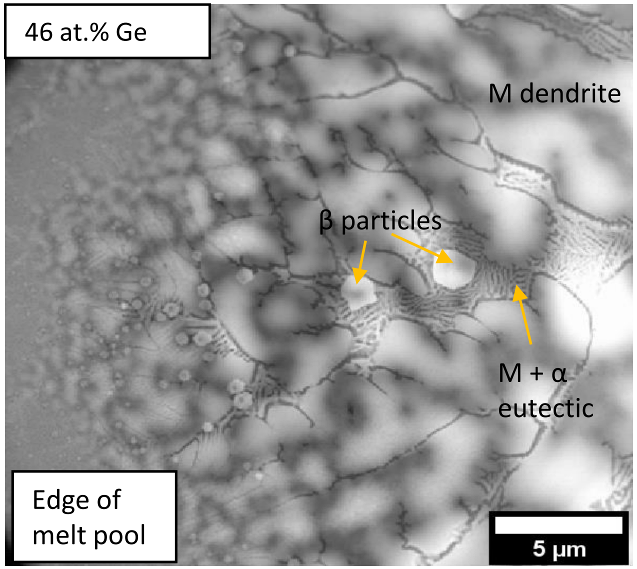
Dynamic Transmission Electron Microscopy (DTEM)



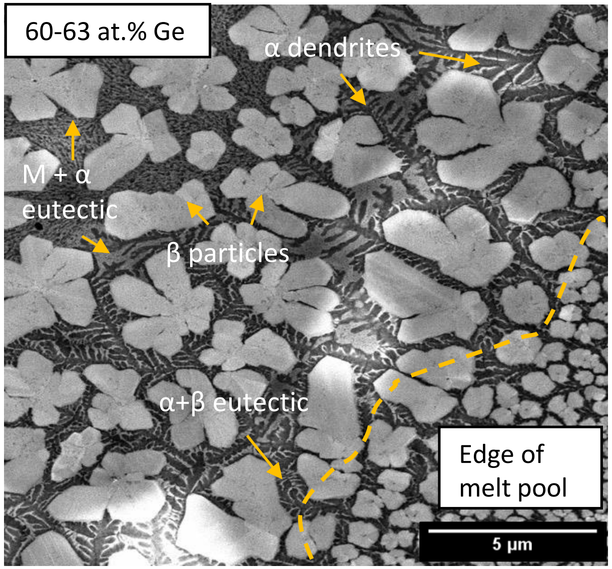
J. D. Roehling et al., *Acta Mat,* 131:22-30 (2017).

Observed Microstructural regions

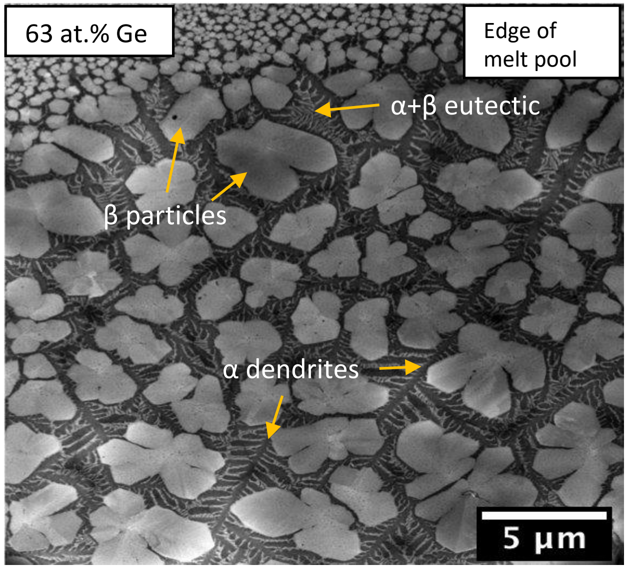
- Below 50.5 at.% Ge, M phase was the primary phase, surrounded by M + α eutectic
- Above 50.5 at.% Ge, β phase was the primary phase, surrounded by a few different types of microstructures:
  - α-dendrites surrounded by either α + β or M + α eutectic
  - M + α eutectic



TEM image taken by Francisco Coury

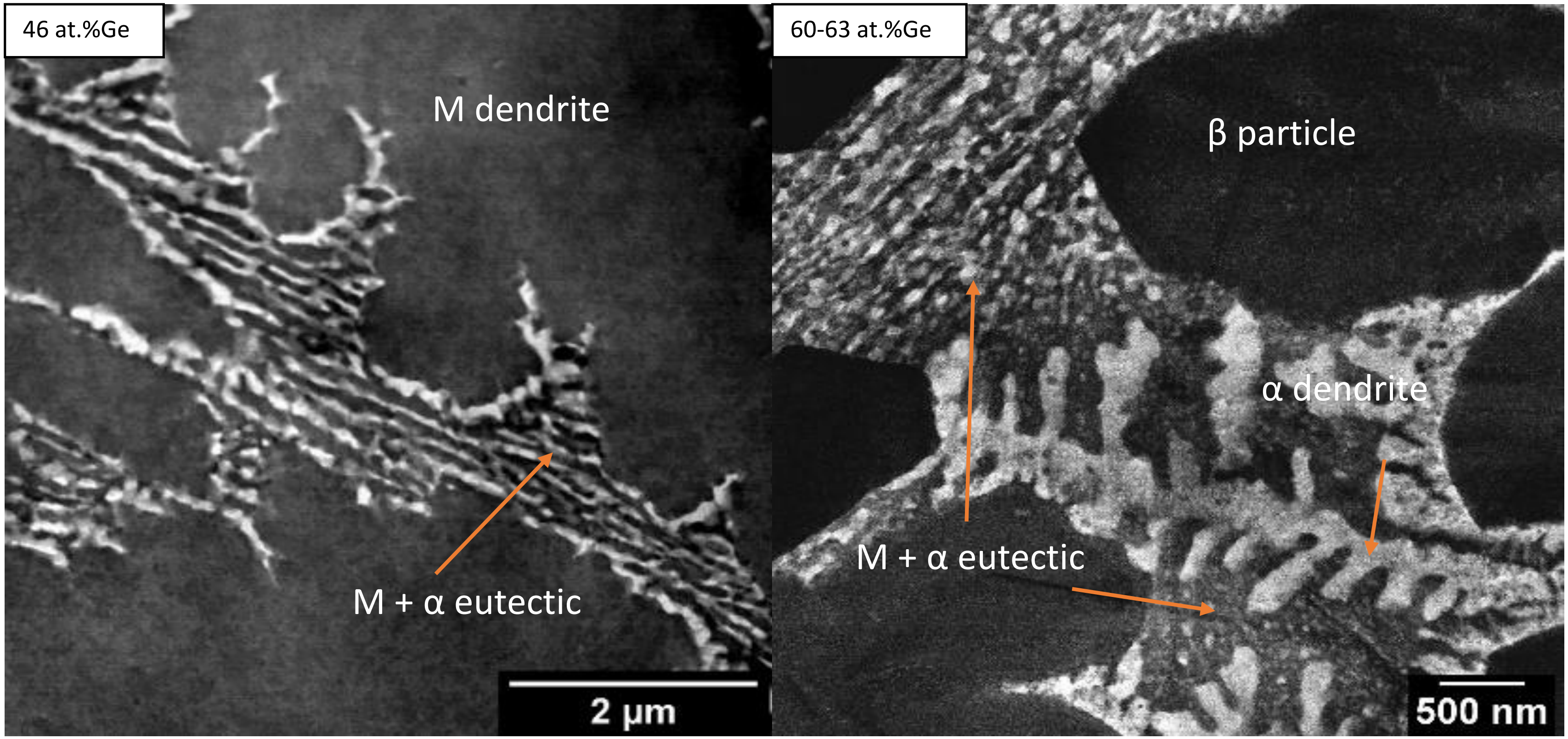


HAADF TEM image



HAADF TEM image taken by Yaofeng Guo

Morphologies of M+α Eutectic

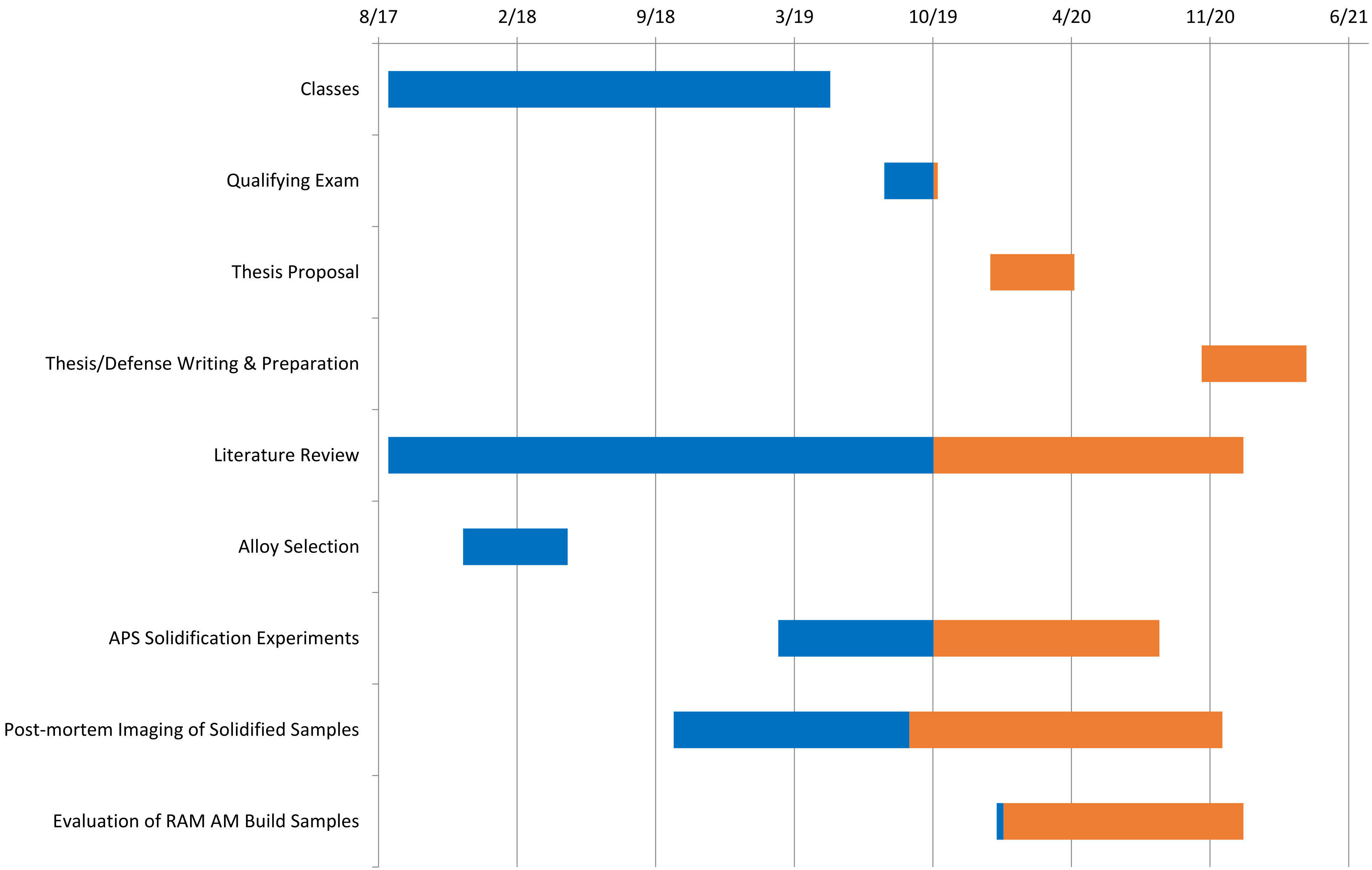


BF STEM images taken by Francisco Coury and Yaofeng Guo

Conclusions & Future Work

- Local microstructure between primary phases is determined by local solute conditions
- Various morphologies of M+α eutectic have been observed based on the primary phase that forms and local solute conditions

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
Alloy Selection
APS Solidification Experiments
Post-mortem Imaging of Solidified Samples
Evaluation of RAM AM Build Samples

Challenges & Opportunities

- Sample preparation & EBSD of Al samples
- Project goals:
  - Compare G & R in RAM vs. traditional 6061
    - G & R relationship can trigger grain morphology transitions in 6061 (not fully explored), while the effects in RAM alloys have not been evaluated
    - Consider experiments using: multiple laser passes/re-melts at varying time delay, settings, & offset (in-situ), preheating temperature (ex-situ)
  - Effect of delay time on re-melt zone size, possible cracking (solidification/residual stress), fraction of reacted powder (RAM), and grain size

Thank you!

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APS Additive Manufacturing Simulator Set-up

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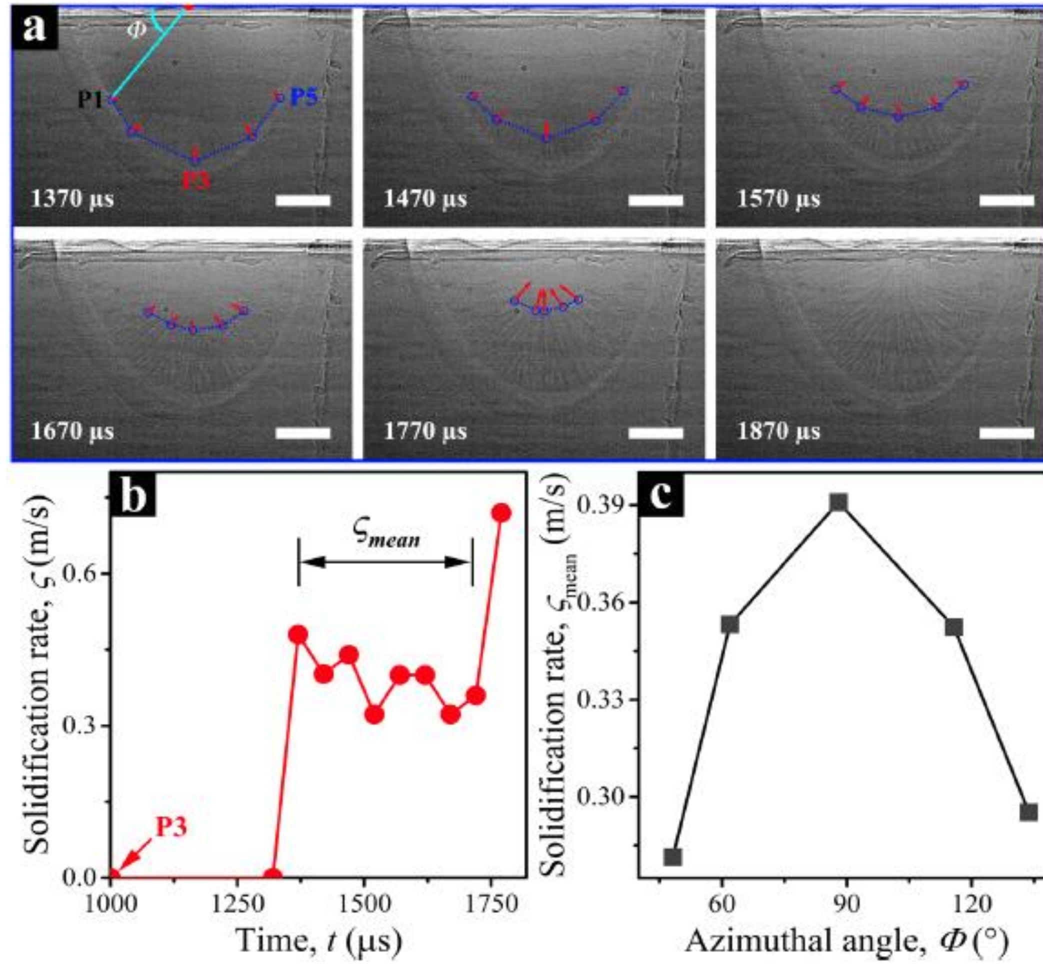
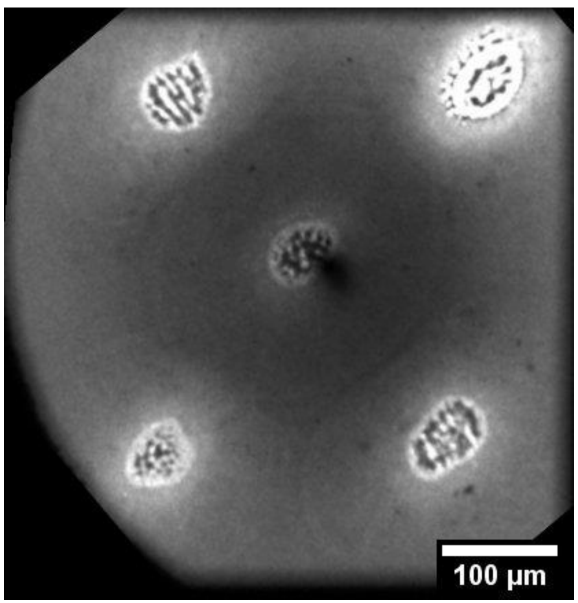


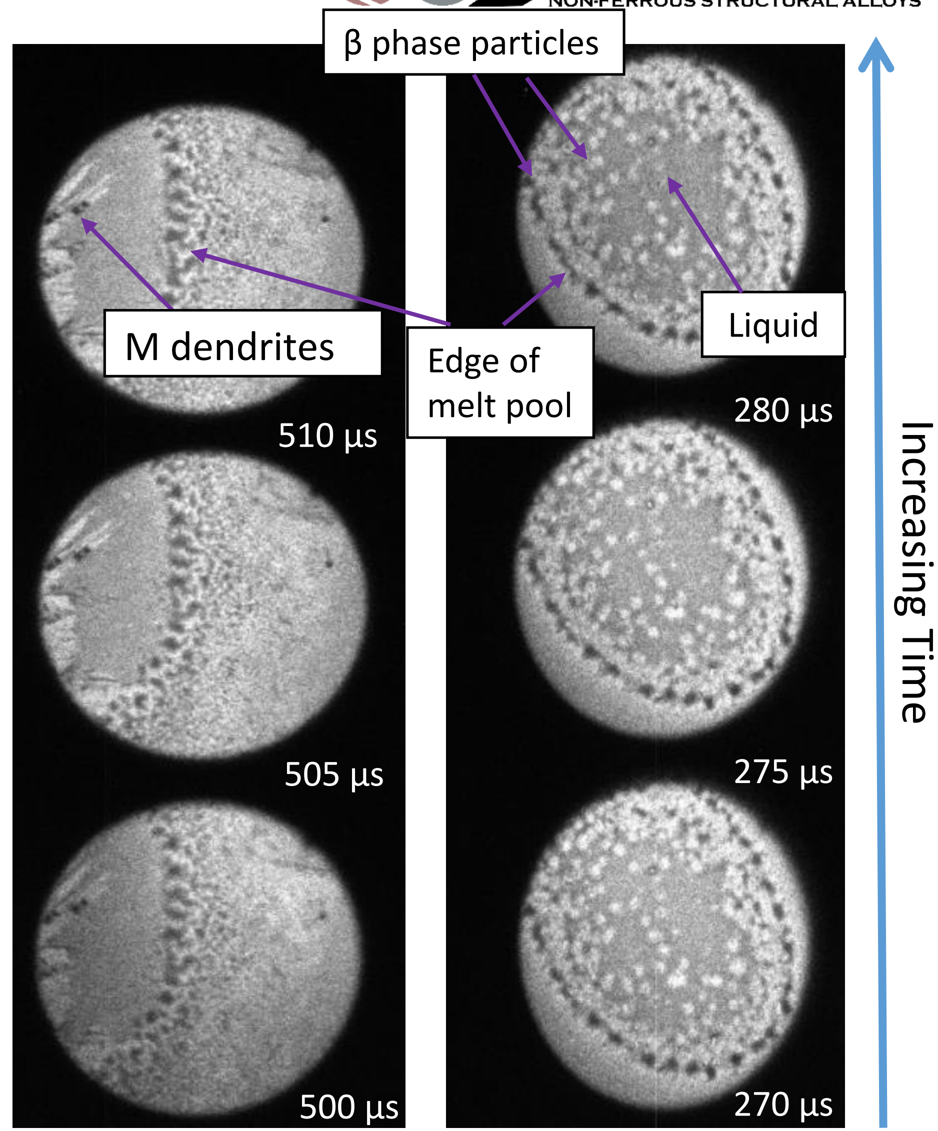
Synchrotron x-ray imaging of a Ti-6Al-4V plate sample in laser melting processes and solidification rate measurements. C. Zhao et al., *Scientific Reports* (2017).

Al-Ge DTEM Melt Pools

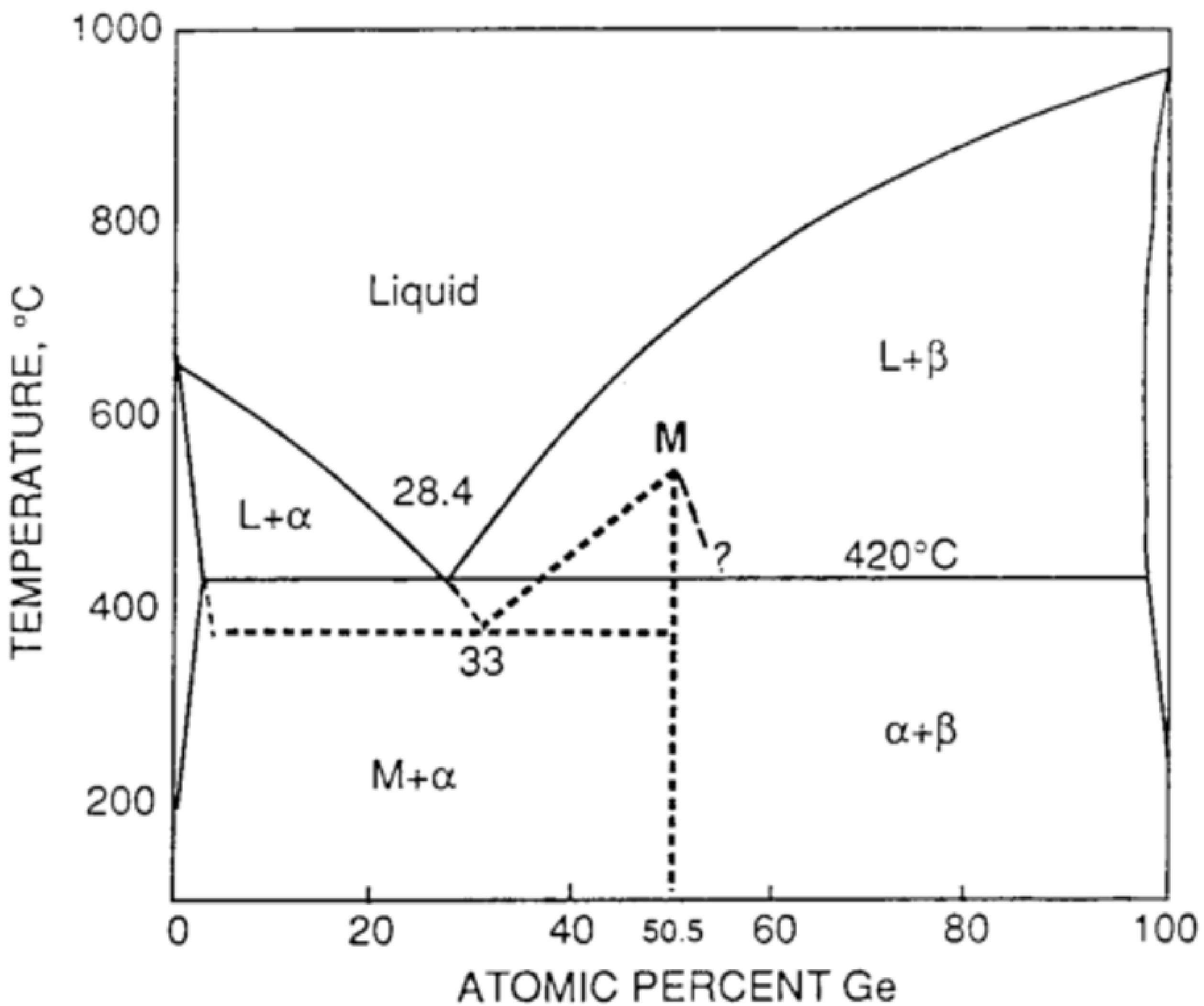
- Melt pools are roughly 50-70 µm in diameter
- β phase particles formed throughout the melt pool
- Average solidification velocity was found to be ~ 0.08-0.15 m/s



Left: TEM image of 70-76 at.% Ge film with 5 melt pools taken by Yaofeng Guo
Right: Sequence of 9 images of a melt pool taken using DTEM on a 46 (left) and 63 at.% Ge sample (right).



Microstructural Selection: Dendritic vs. Eutectic Regions



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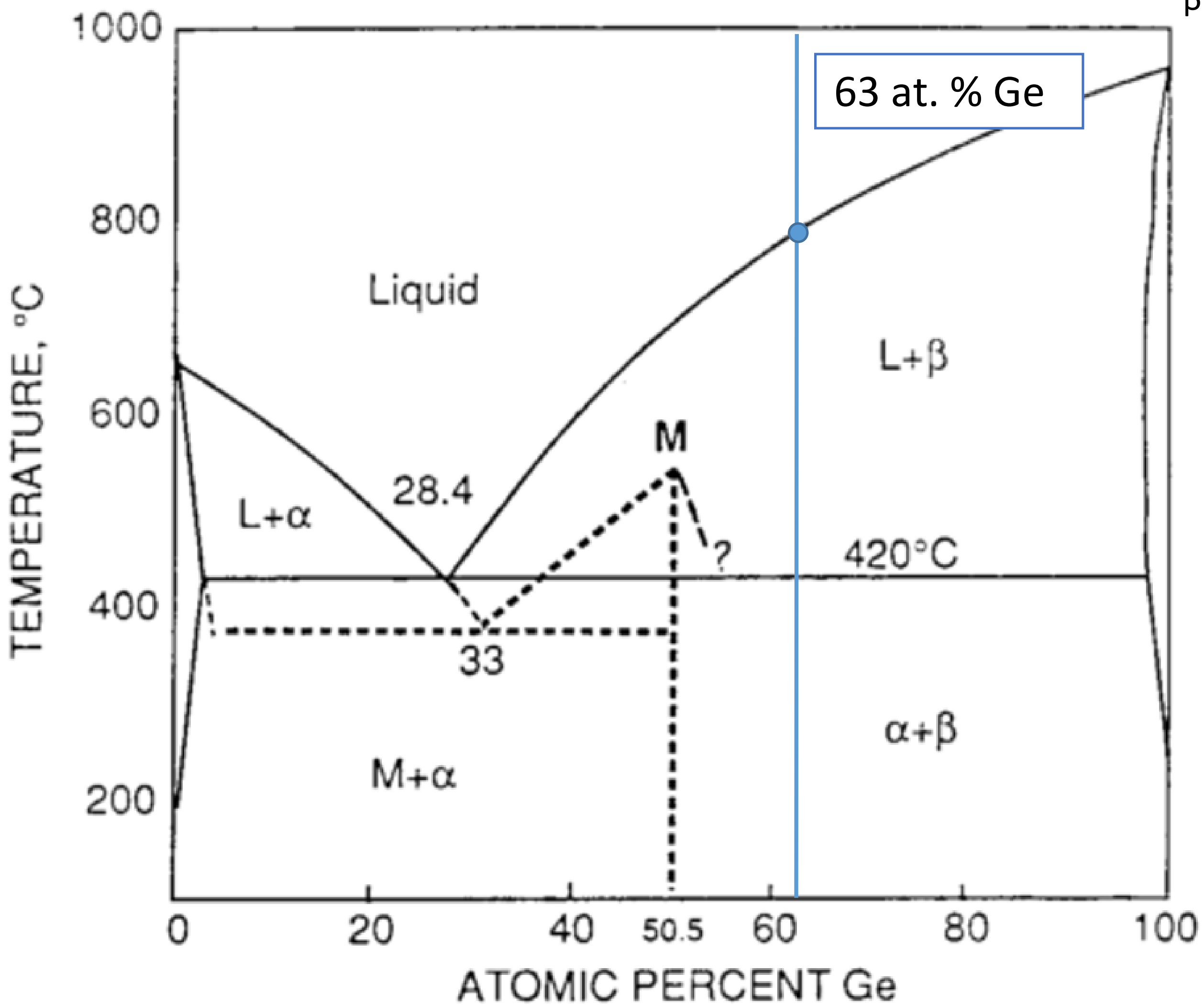

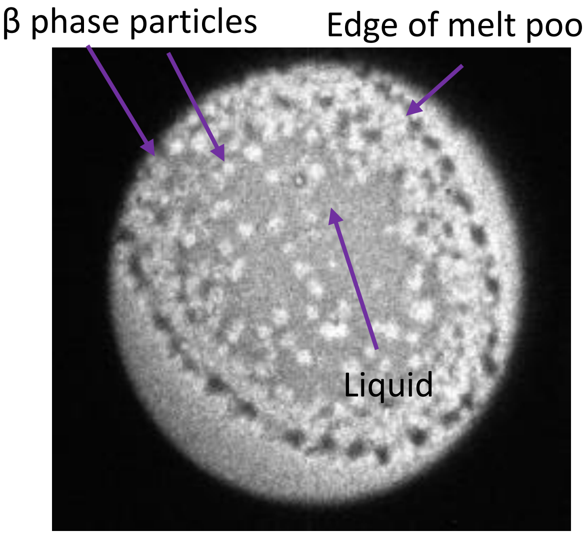

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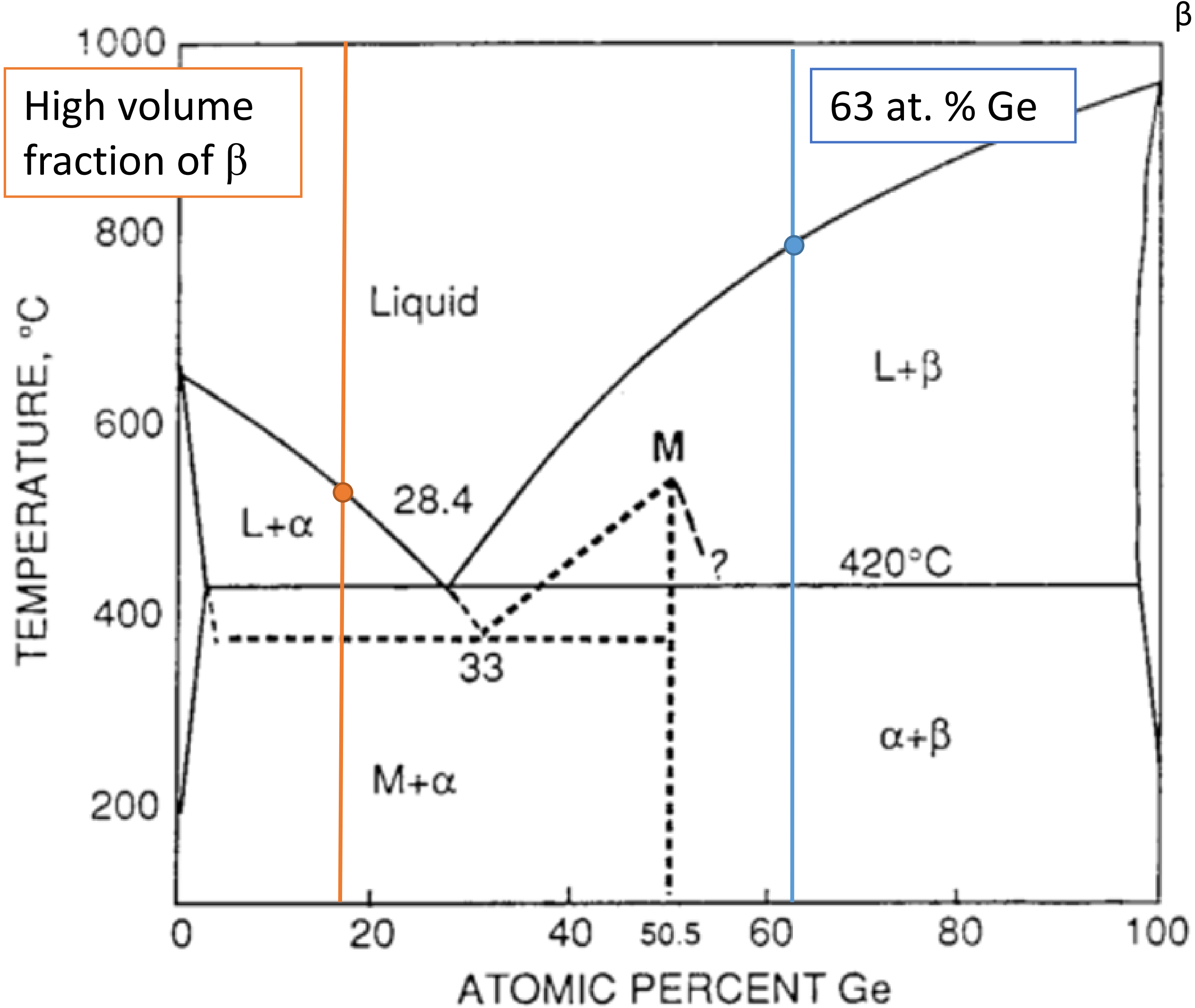

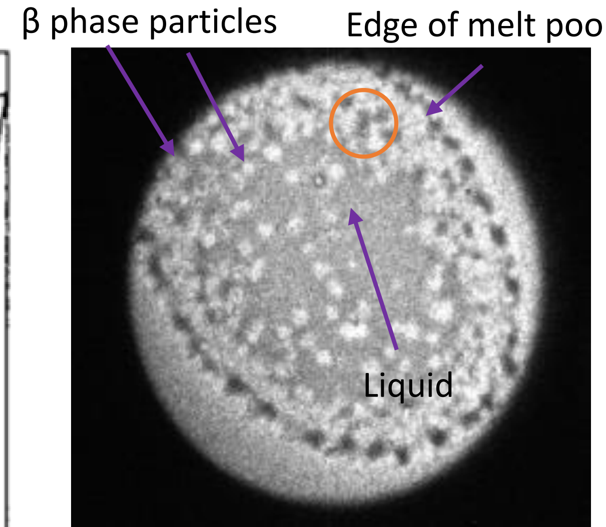

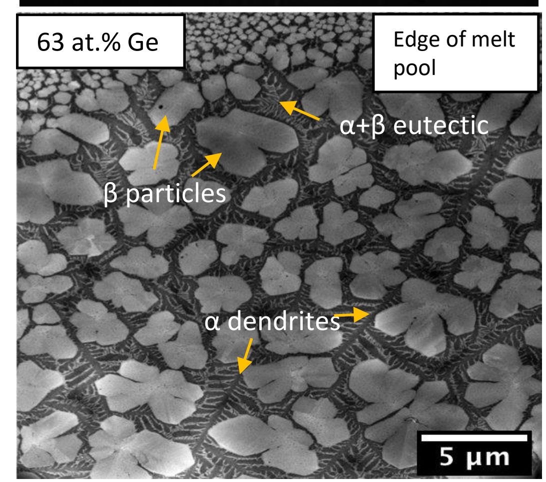

HAADF TEM image taken by Yaofeng Guo

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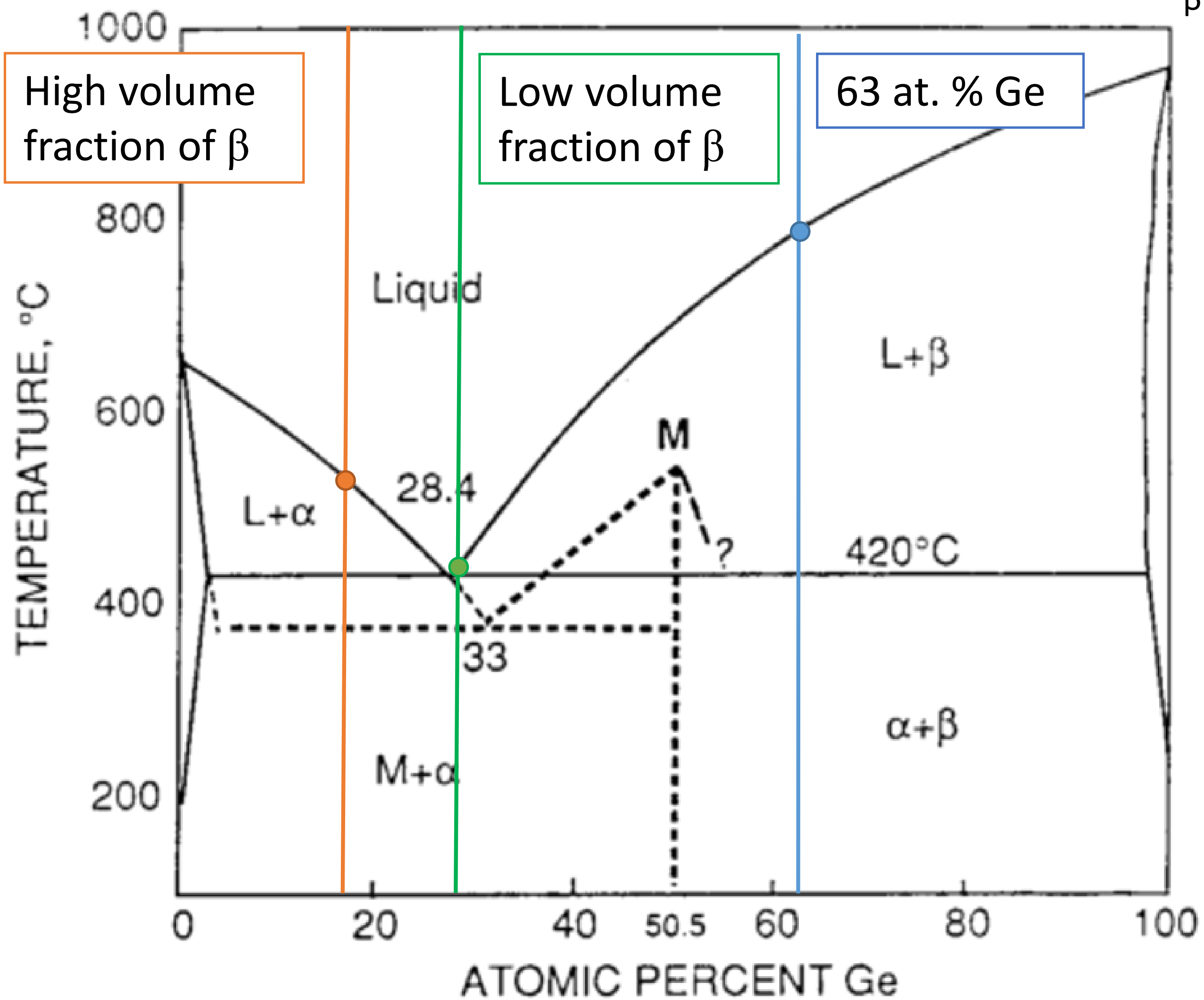
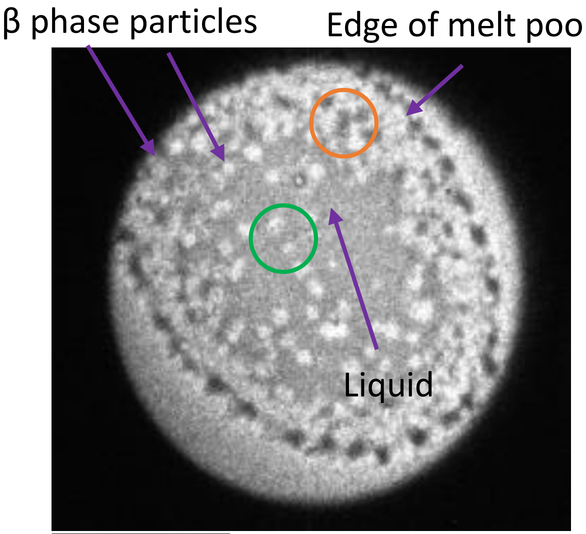
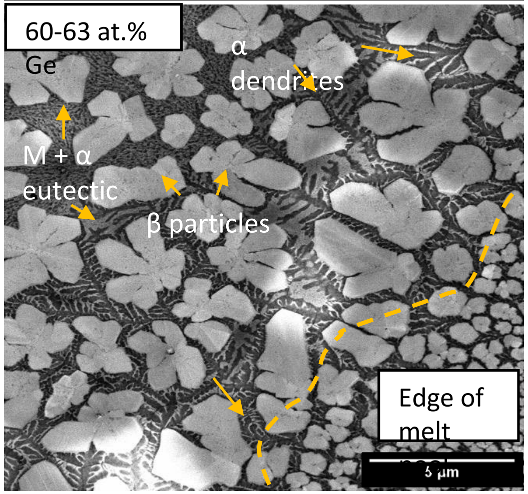
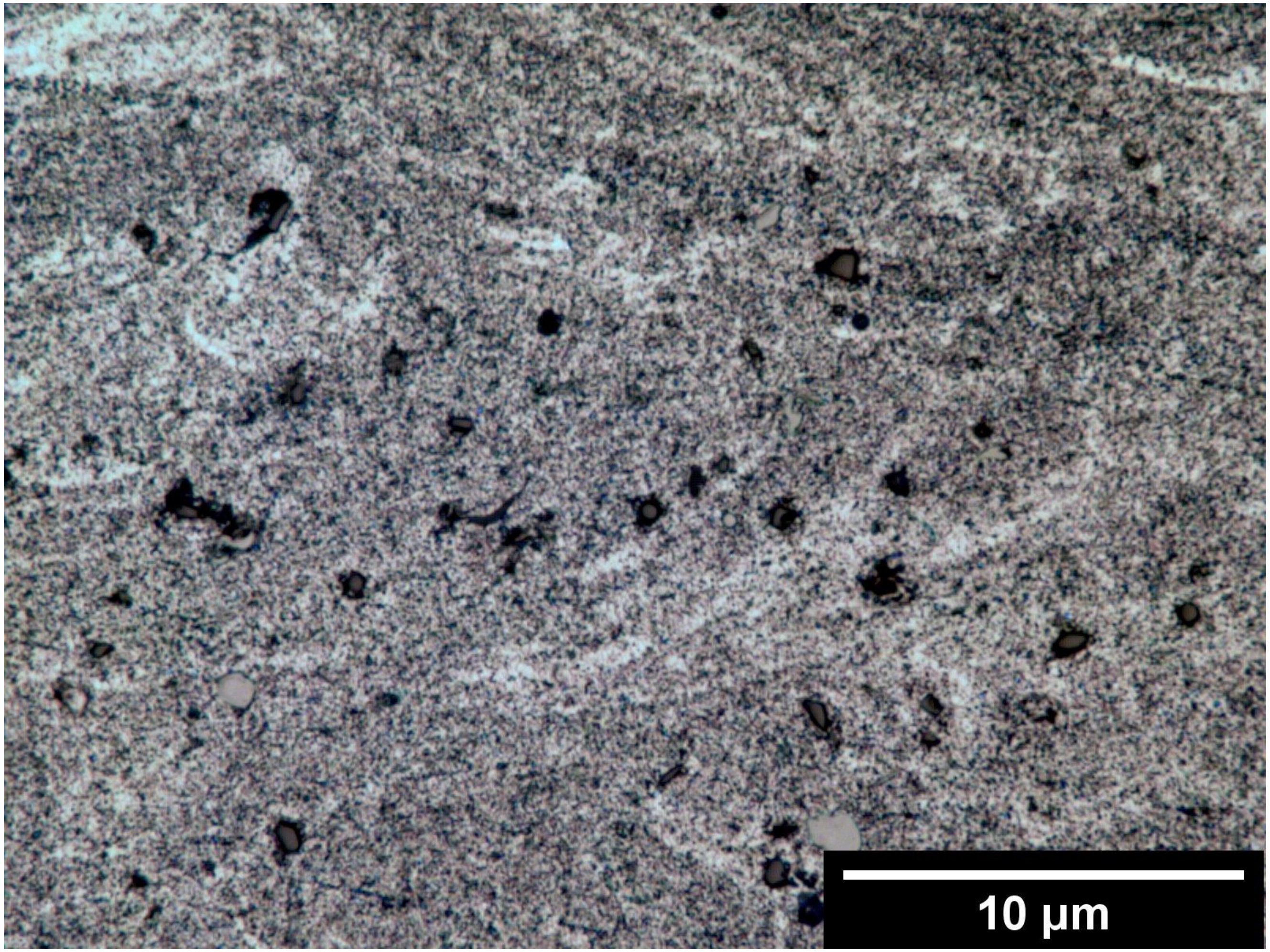






HAADF TEM image

Initial Characterization of RAM 6061

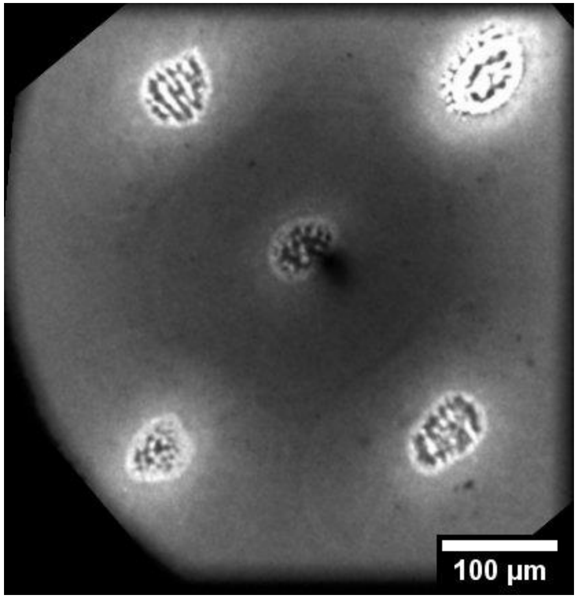


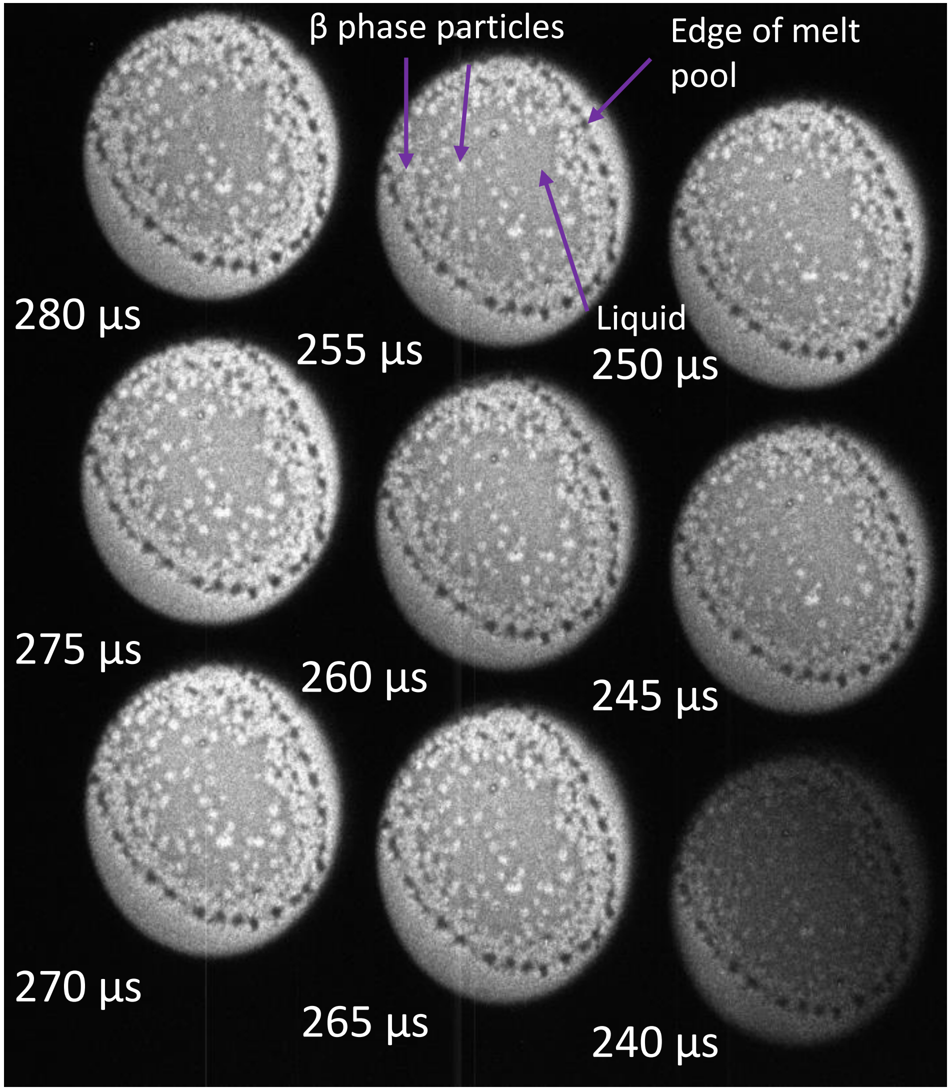
Overview

- Alloy design for AM
  - Metal matrix composite (MMC) & reactive metal powder (RAM) alloys
  - Additive manufacturing simulator at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL)
  - Initial post-mortem findings from top down imaging
  - Extraction of solidification velocity from radiography
- Metastable phase development in Al-Ge
  - Previous observation of metastable monoclinic phase in Al-Ge
  - Dynamic transmission electron microscopy (DTEM)
  - Observed microstructures & suggested formation conditions

Al-Ge DTEM Melt Pools

- Melt pools are roughly 50-70 µm in diameter
- β phase particles formed throughout the melt pool
- Average solidification velocity was found to be ~ 0.08-0.15 m/s





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