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

Project 30: Microstructural Development of Metallic Alloys during Rapid Solidification

Spring 2019 Semi-Annual Meeting
Iowa State University, Ames, IA
April 3-5, 2019

Student: Chloe Johnson (Mines)

Faculty: Amy Clarke (Mines).

Industrial Mentors: Los Alamos National Lab, Boeing

Other Participants: Yaofeng Guo (Mines), Joe Jankowski (Mines), Gus Becker (Mines), Francisco Coury (UFSCAR), Joe McKeown (LLNL)

NSF COLORADO SCHOOL OF MINES IOWA STATE UNIVERSITY

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 and the evolution is not well understood.
- Objective: Understand the relationship of rapid solidification conditions to 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

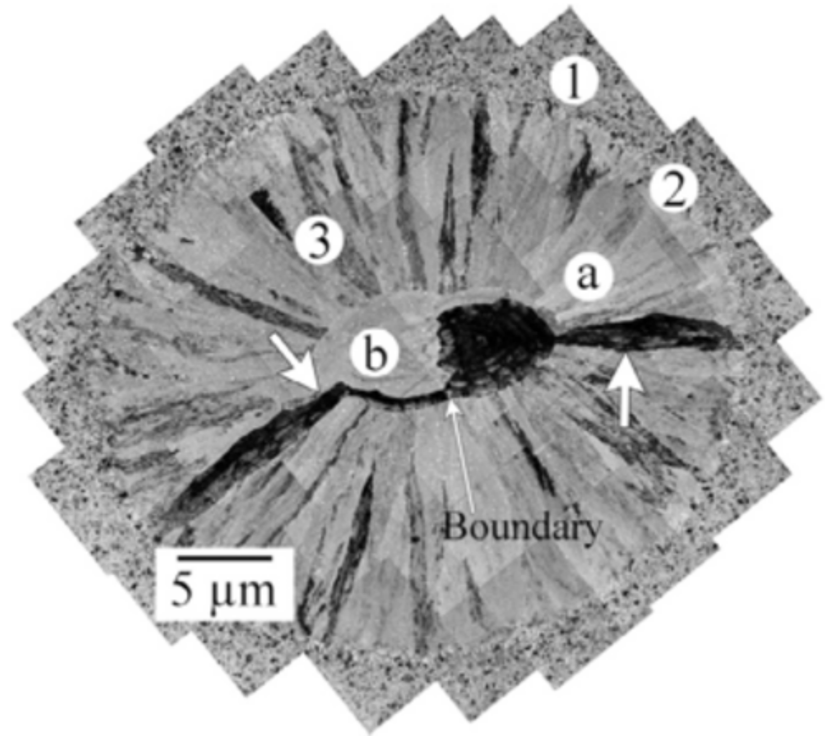
- Image analysis of Dynamic Transmission Electron Microscopy (DTEM) data of Al-Ge alloys
- Initial processing of Al-Ag DTEM data
- Initial look at microstructure from Elementum 3D Al 6061 Metal Matrix Composite (MMC) alloy designed for AM
- Experiments performed on Al-Ag and Al 6061 MMC using AM simulator at the Advanced Photon Source (APS) at Argonne National Lab (ANL)

Metrics

Description	% Complete	Status
1. Literature review & alloy selection	45%	●
2. In/ex-situ characterization of metastable phase formation in rapidly solidified, faceting Al-Ge alloys	50%	●
3. Investigation of novel rapidly solidified structures in binary and ternary model Al alloys	15%	●
4. Evaluation of grain size effects in Al MMC alloys via in/ex-situ rapid solidification	10%	●

Industrial Relevance

- Solidification conditions have a significant impact on final microstructure
- Rapid solidification generates novel microstructures, metastable phases, and a variety of grain morphologies
- Understanding processing/ microstructure relationships for rapid solidification will inform manufacturing processes

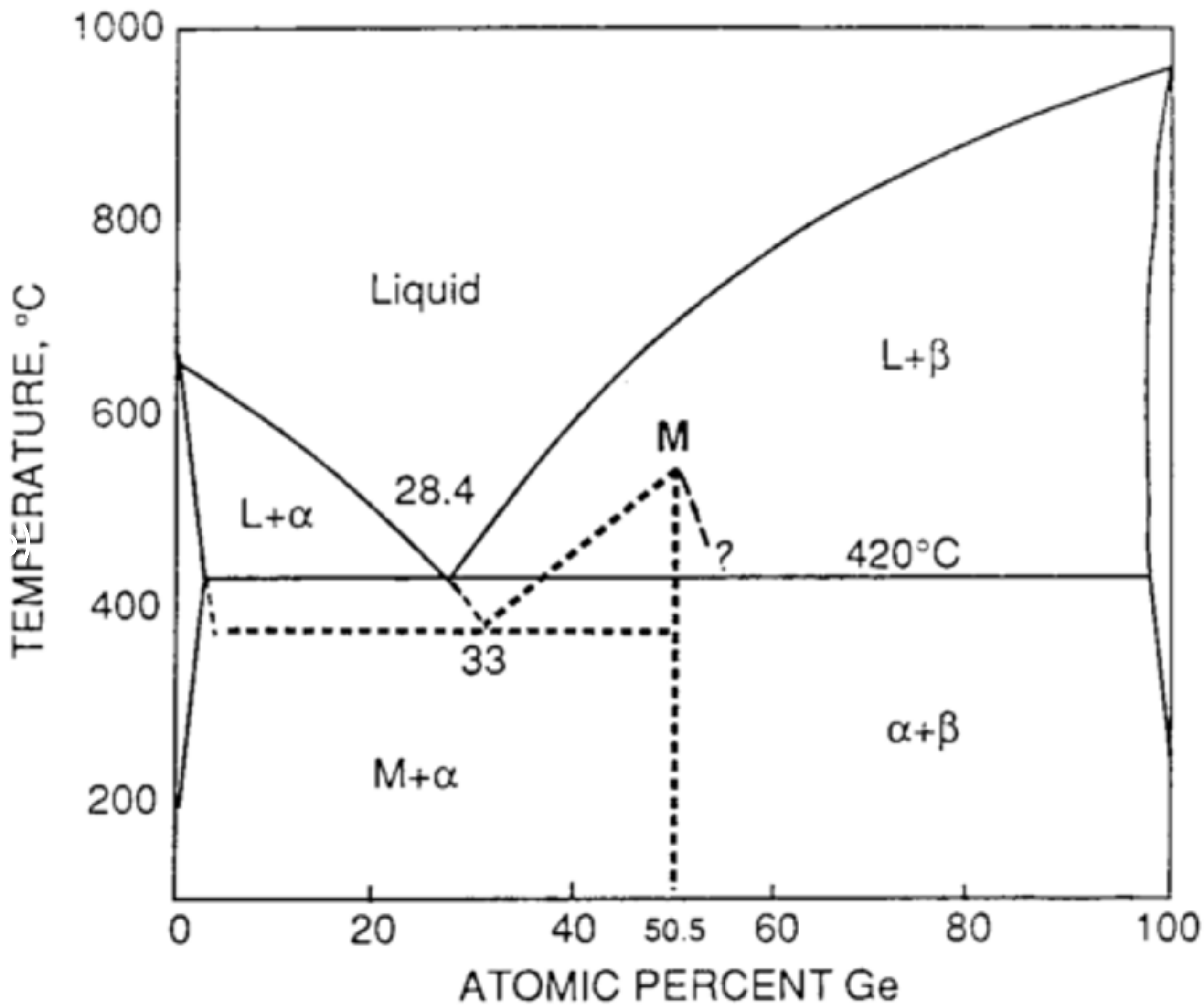


Post-mortem TEM image of Al-Cu alloy film after DTEM solidification.
J.T. McKeown *et al.*, *Acta Materialia* (2014).

Overview

- Observed trends in Al-Ge microstructures from DTEM experiments
- Initial results from AM simulator at APS
  - Al 6061 MMC alloy
  - Al-Ag (model alloy)
- Future Work

Previous Work: Development of Metastable Phase Diagram



Proposed metastable phase diagram for monoclinic (M) phase.

T. Laoui & M. J. Kaufman, *Met Trans A* (1991).

Previous Work: Development of Metastable Phase Diagram

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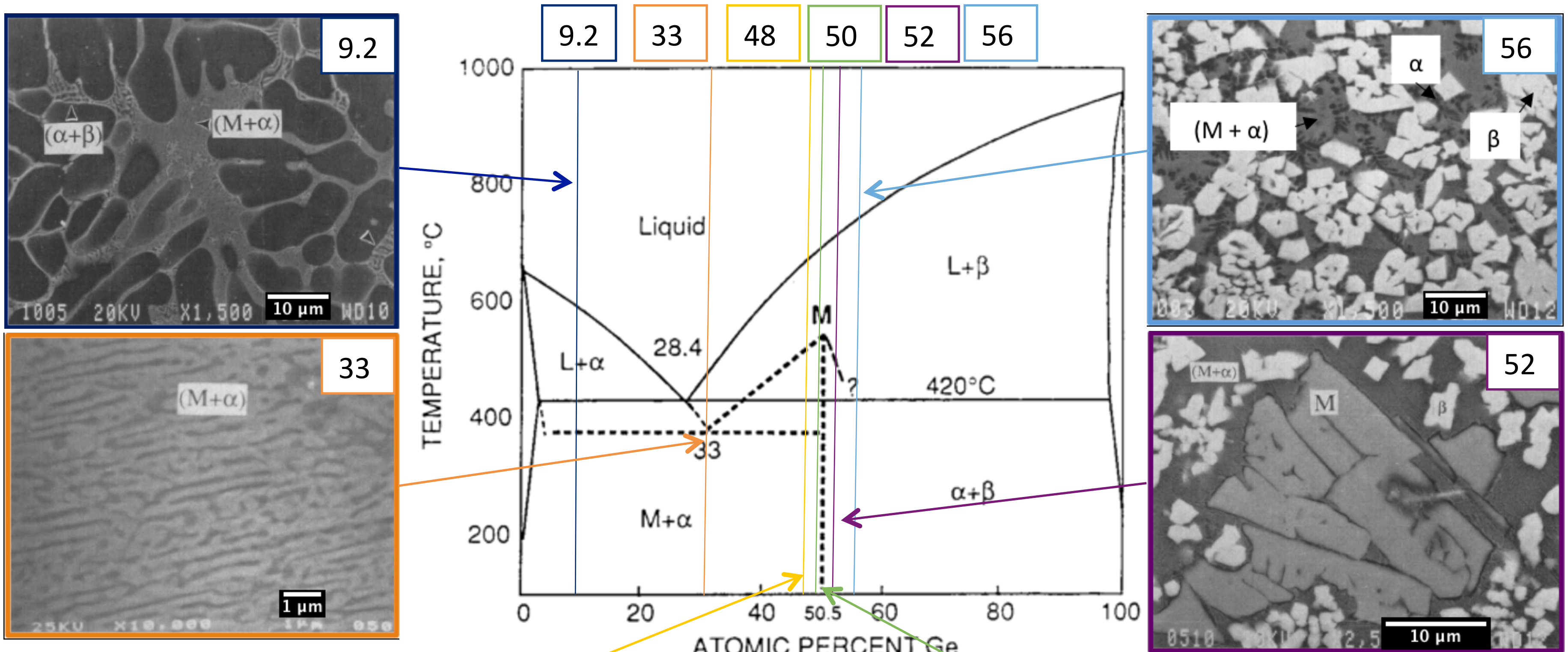

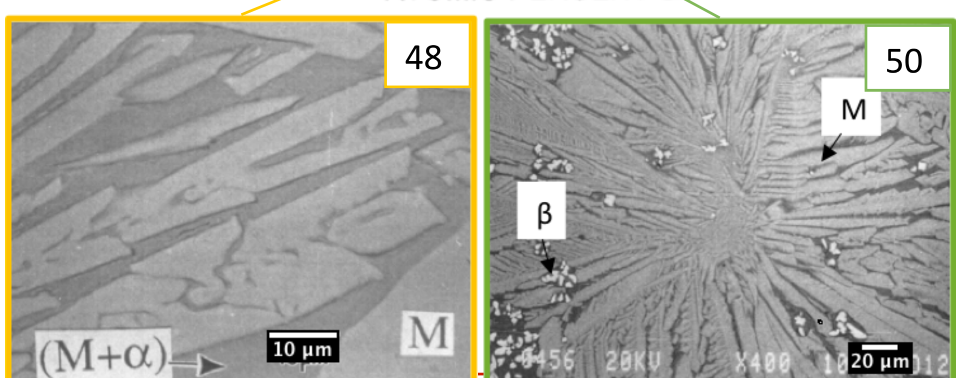

M – Monoclinic phase (43-45 at. % Ge)

α – FCC Al solid solution (0-3 at. % Ge)

β – Diamond cubic Ge solid solution (99-100 at. % Ge)

SEM images of observed phases in various Al-Ge samples.
T. Laoui & M. J. Kaufman, *Met Trans A* (1991).

DTEM of Al-Ge: Our Interests

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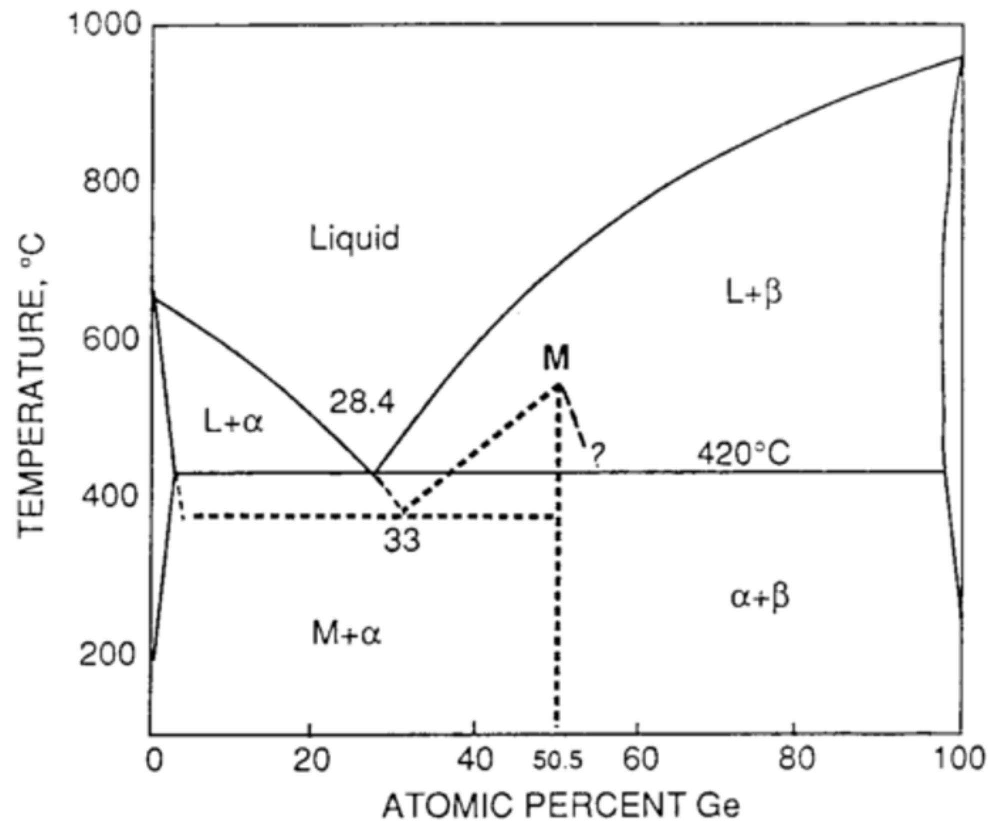
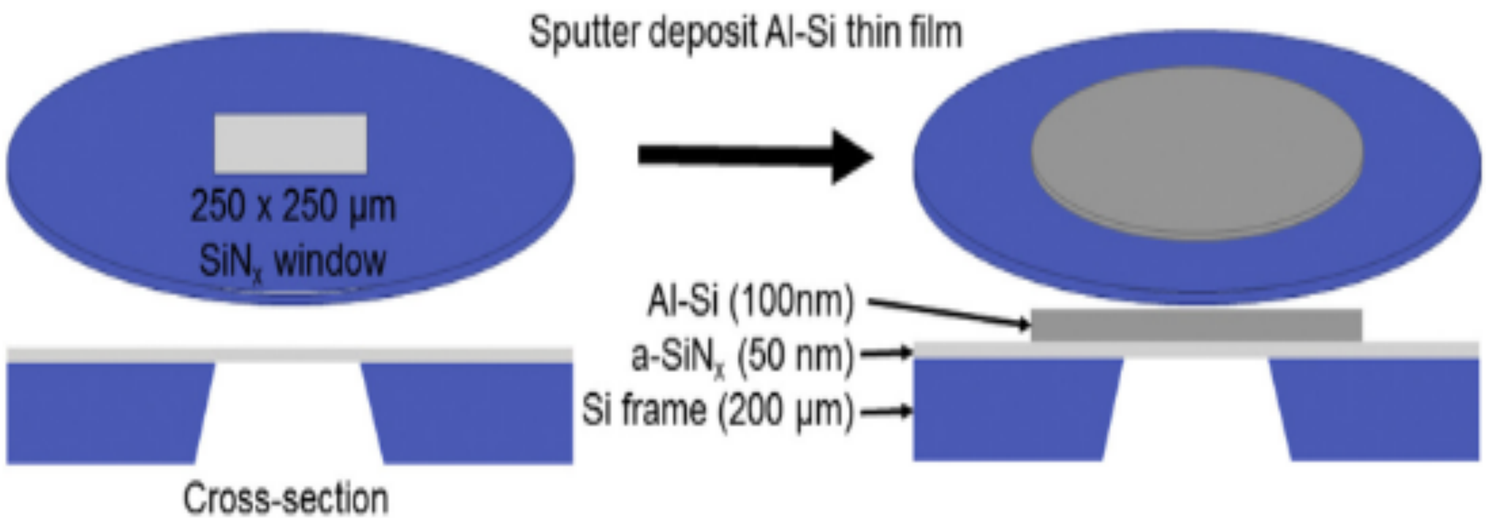
- Explore proposed metastable phase diagram that includes M phase
- Investigate phase transformation pathway and if M phase remelts in the liquid prior to the formation of β phase at compositions above 50.5 at. %



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

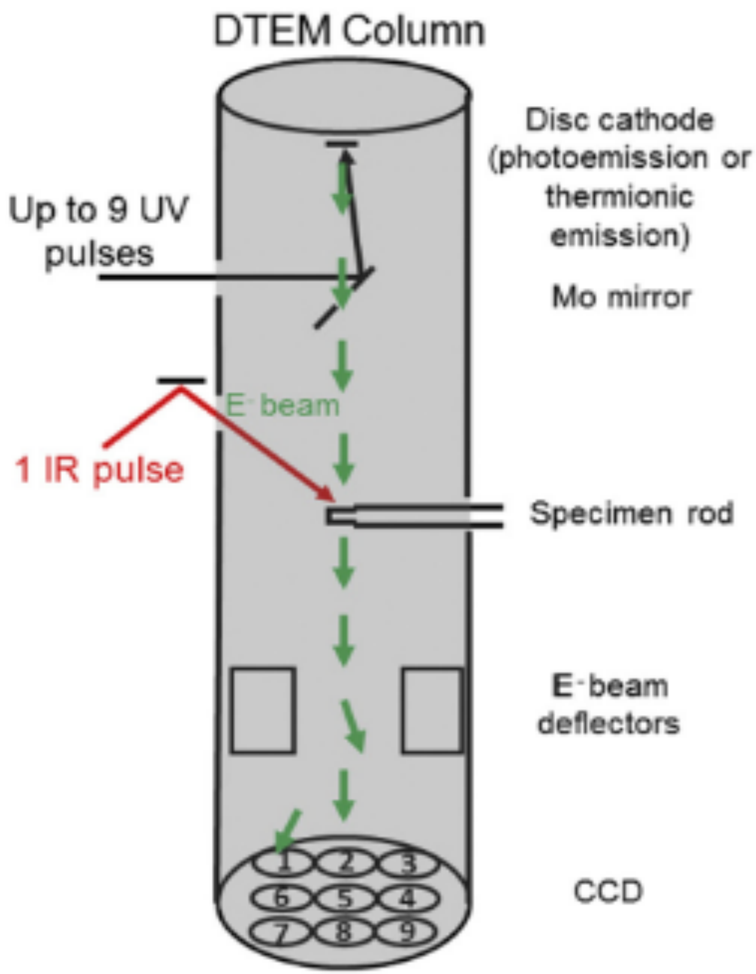
Dynamic Transmission Electron Microscopy (DTEM): Sample Prep



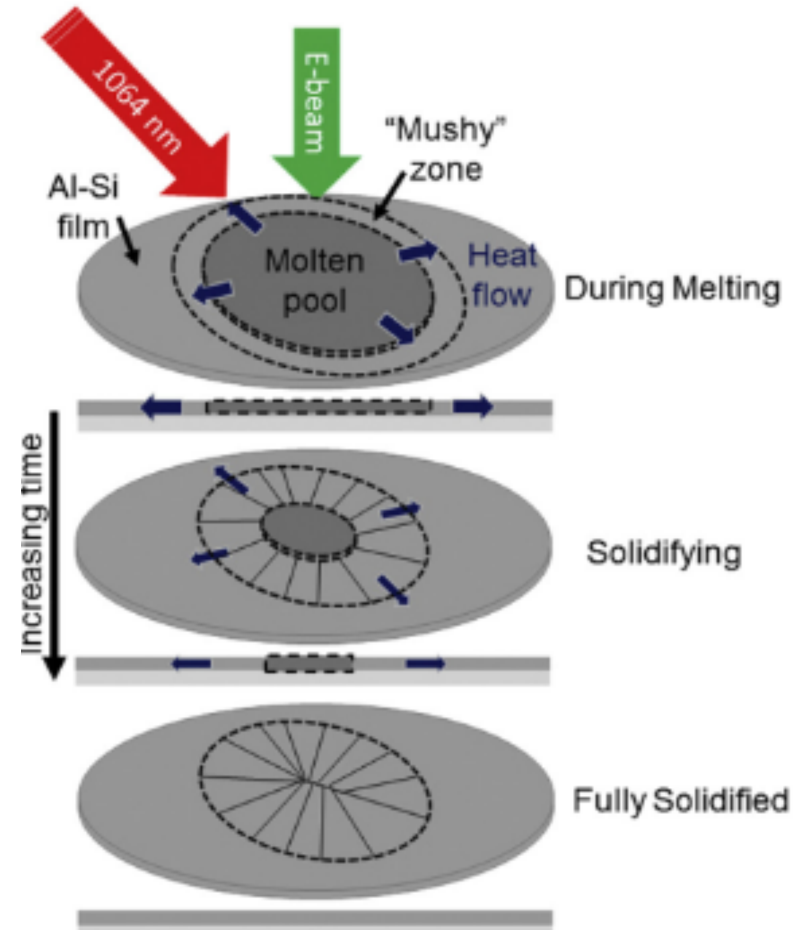
Schematic of sample preparation for DTEM, consisting of a thin film sputter deposited onto a Si-N substrate with a window in the center

J. D. Roehling et al., *Acta Met* (2017).

Dynamic Transmission Electron Microscopy (DTEM): Technique



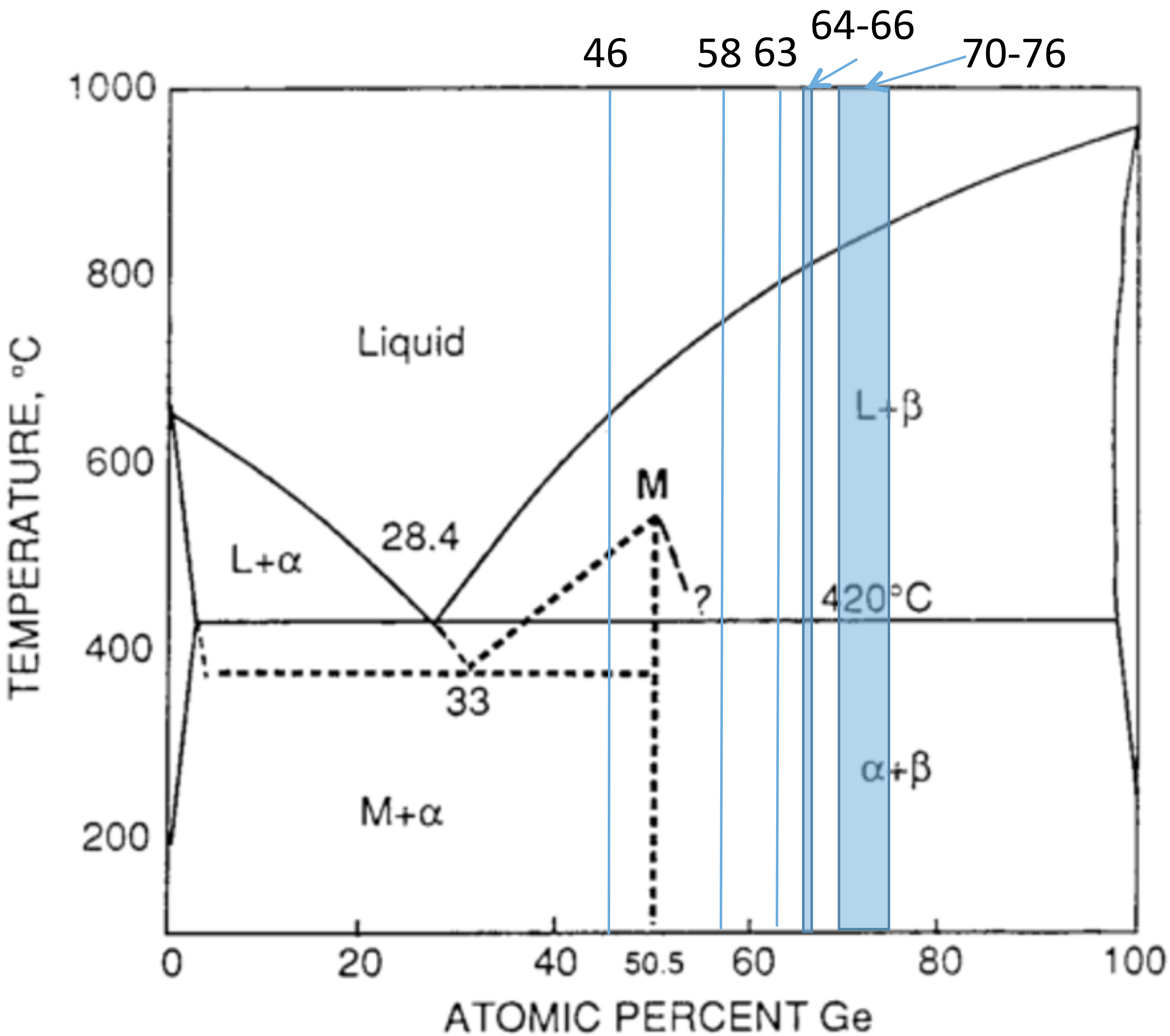
Schematic of DTEM column, where E-beam deflectors allow the collection of 9 images at microsecond intervals



Solidification of a melt pool generated using DTEM

J. D. Roehling et al., *Acta Met* (2017).

Al-Ge Sample Compositions



- Sample compositions were targeted to be to greater and less than 50.5 at. % Ge
- The compositions tested were 46, 58, 63, 64-66, and 70-76 at. % Ge

Observed Microstructure Below 50.5 at. % Ge

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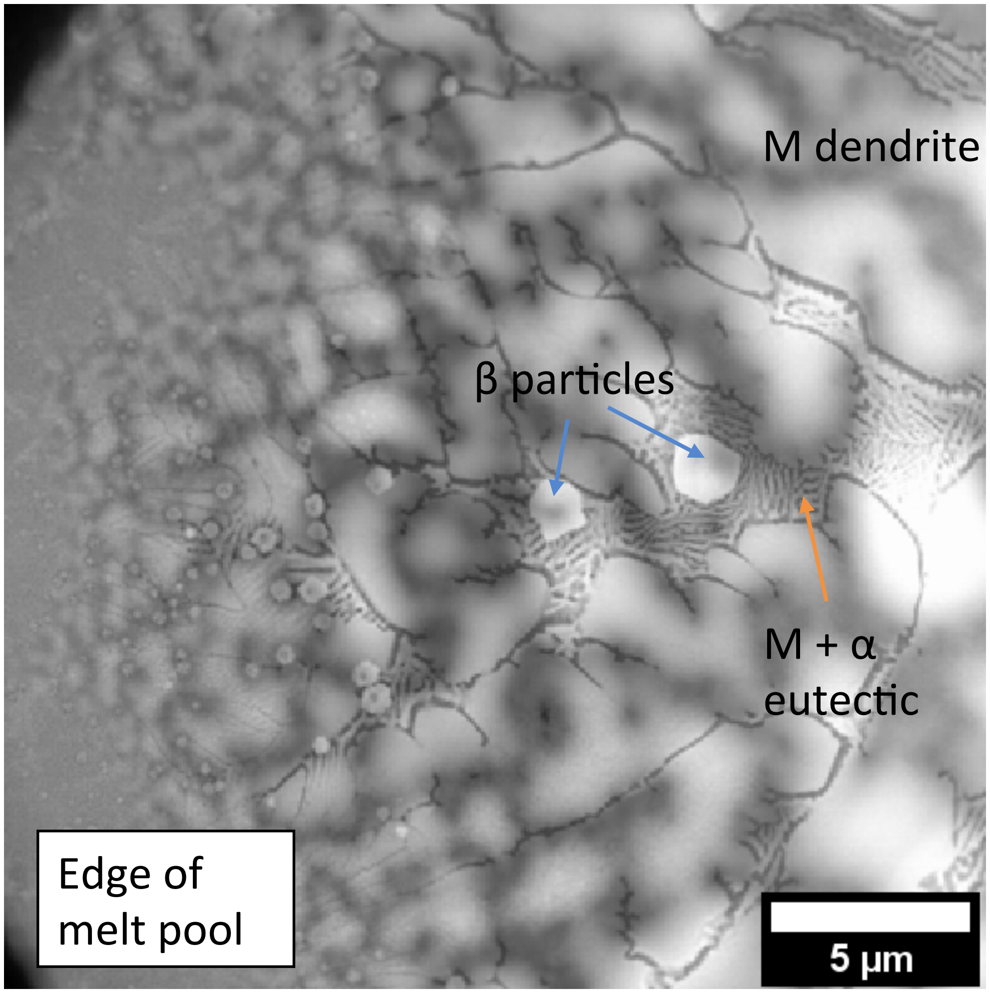

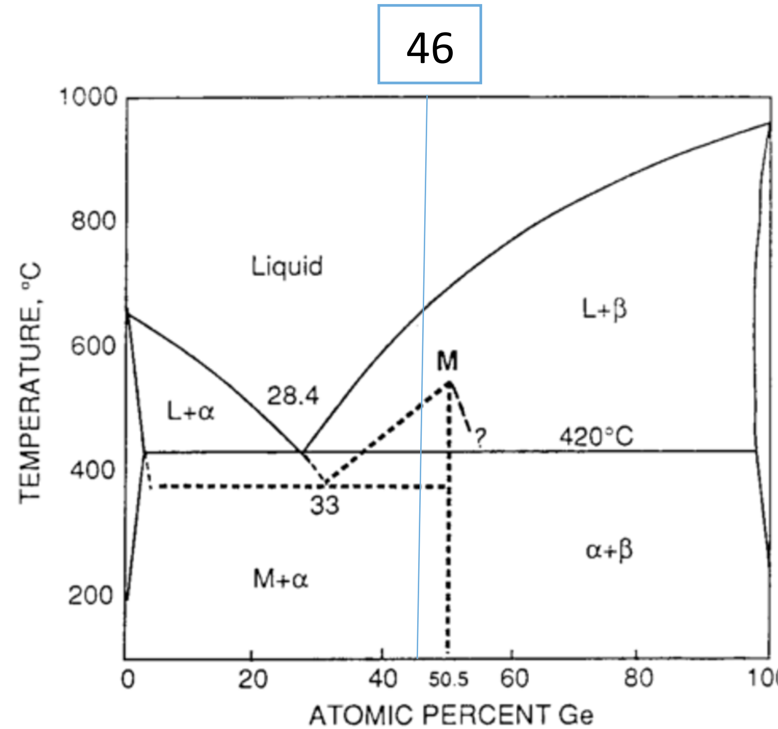

TEM image taken by Francisco Coury

Microstructures Above 50.5 at. % Ge: Multiple Regions

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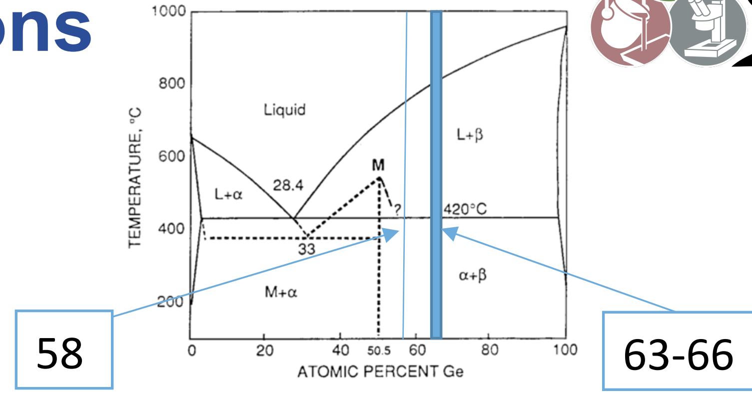

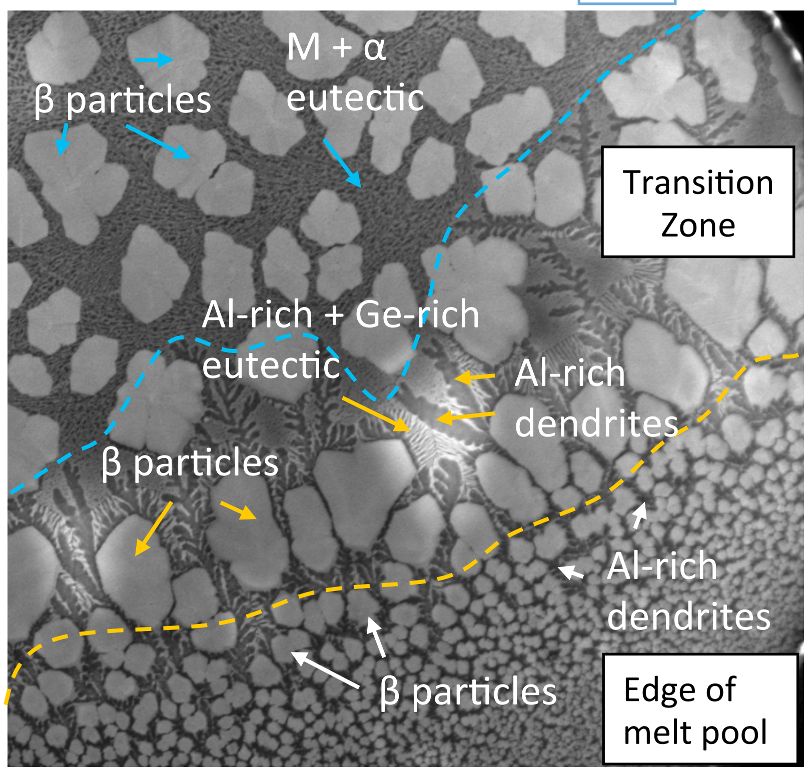

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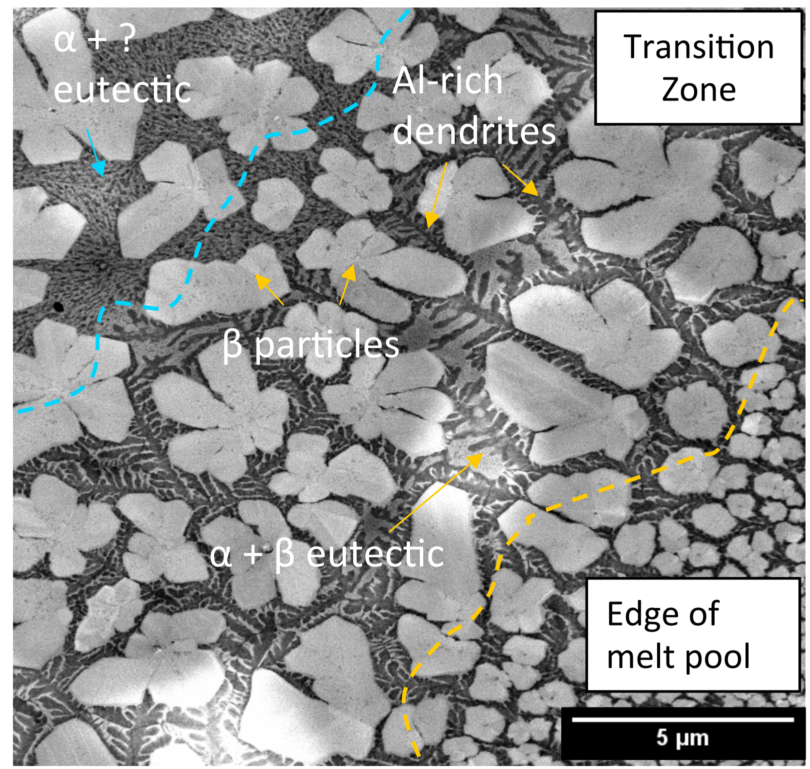
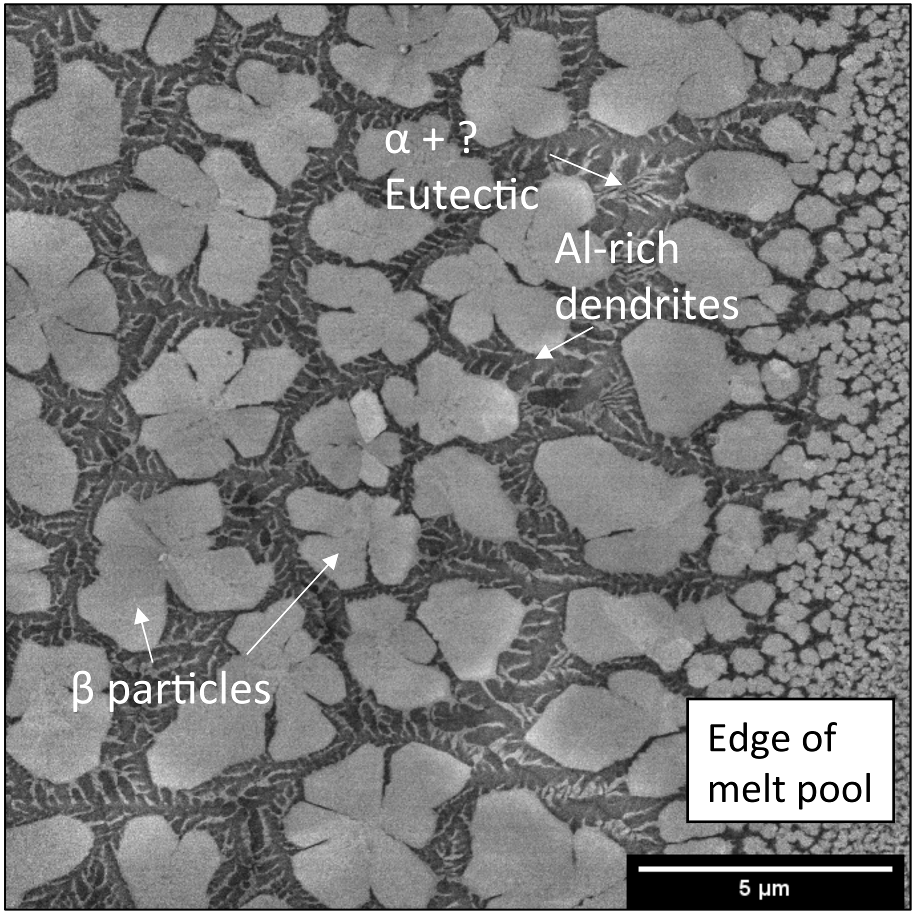
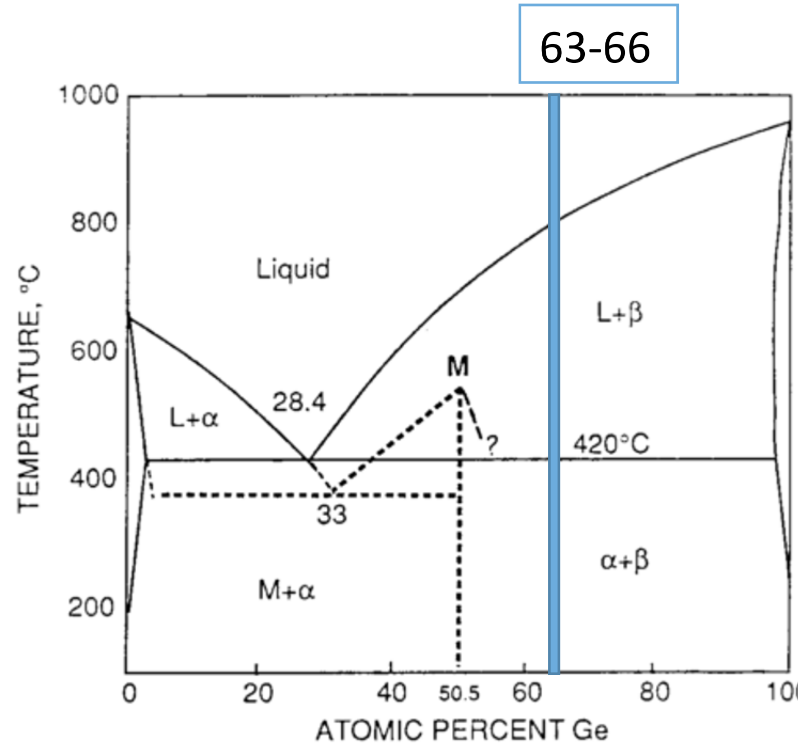


HAADF STEM image taken by Chloe Johnson

Microstructures Above 50.5 at. % Ge: Single Region



HAADF STEM image taken by Chloe Johnson



Microstructural Selection: Multiple vs. Single Regions

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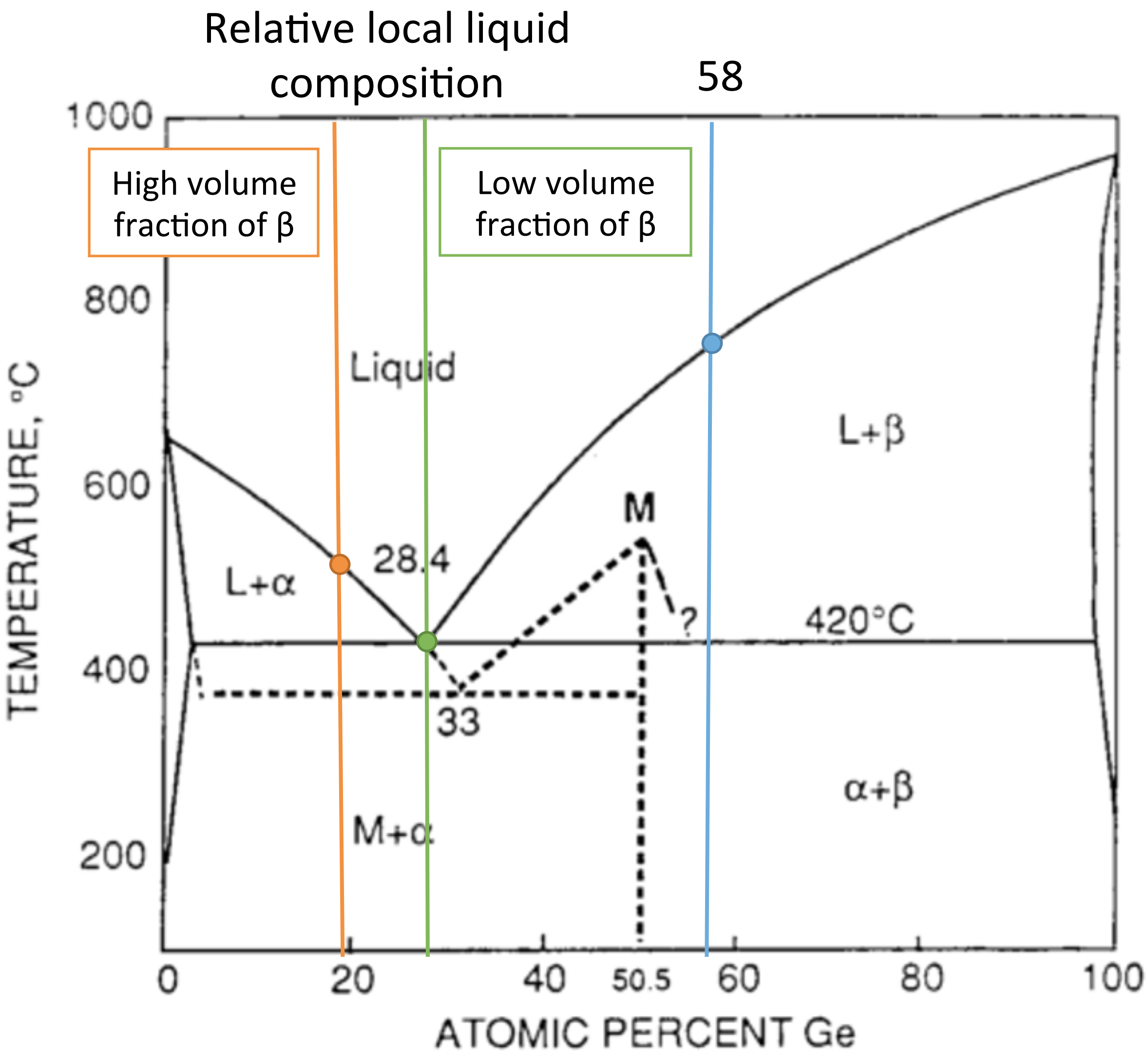

Consider 58 at. % Ge:

- Solidification starts with the formation of β particles at various regions in the melt pool
- If higher volume fractions form, Al rich dendrites form lowering the constitutional undercooling in the remaining liquid
- If lower volume fractions form, dendrites do not form and the undercooling is high enough for a metastable eutectic to form

Microstructures Above 50.5 at. % Ge: Highest Ge Content

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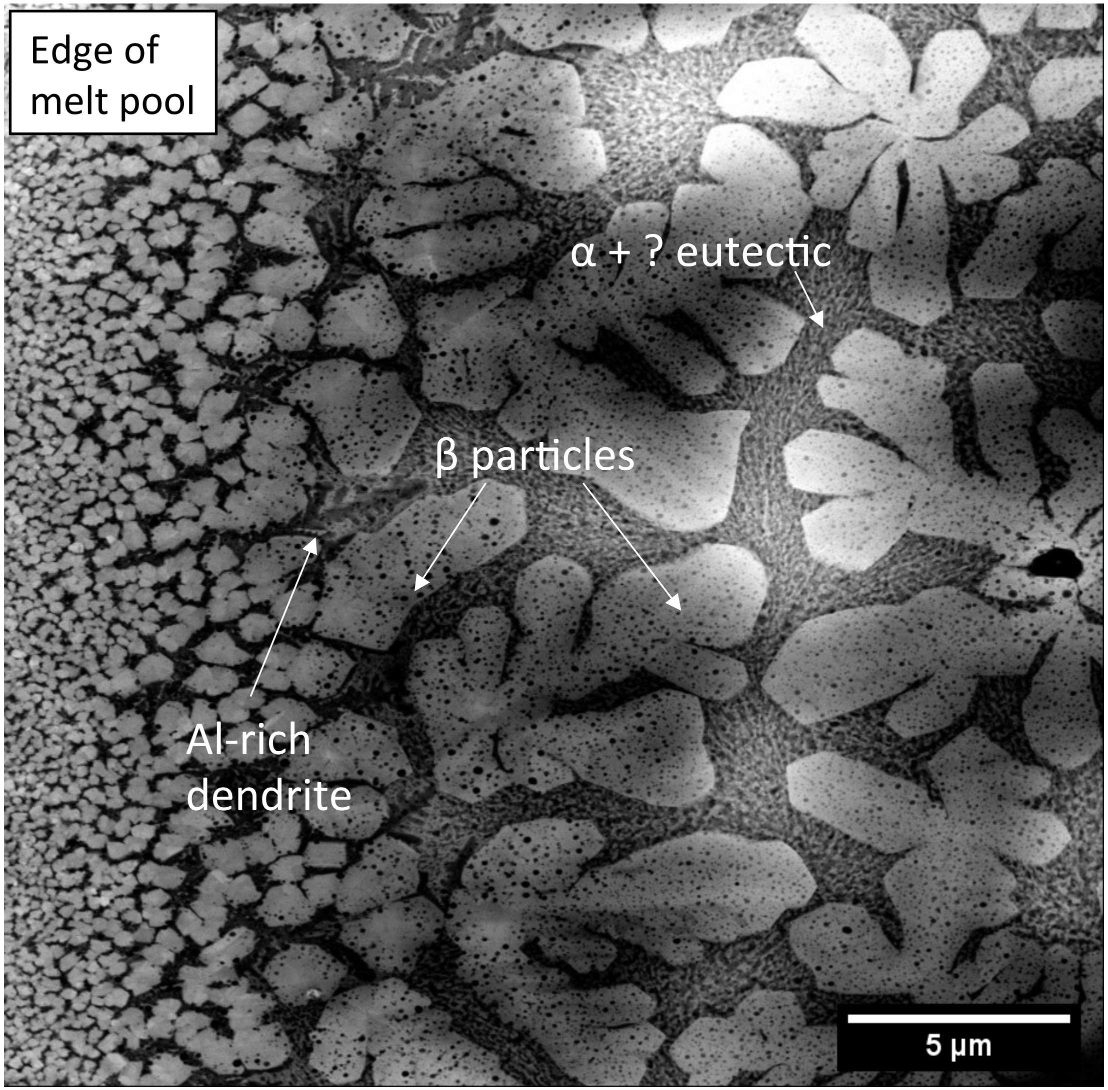
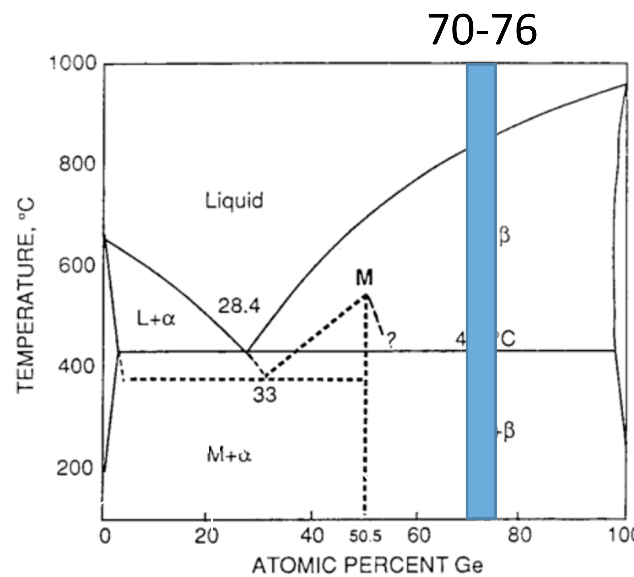
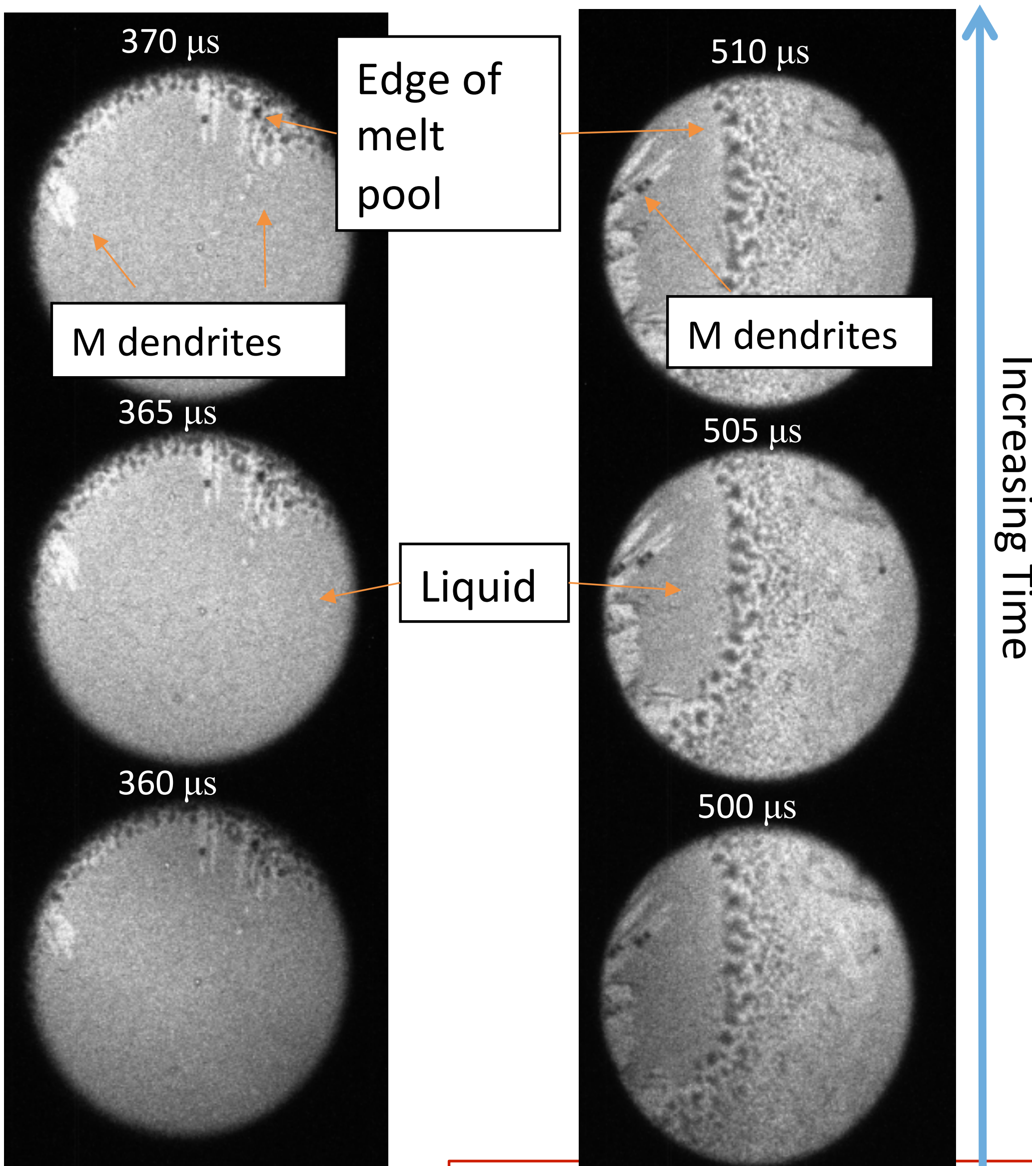
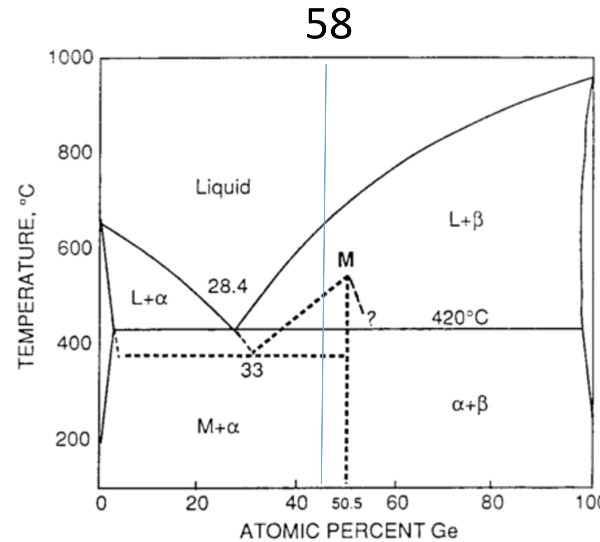




HAADF STEM image taken by Yaofeng Guo

Appearance of M Phase Dendrites Below 50.5 at. % Ge





- M phase observed in dynamic data growing from edges and other areas in the melt pool

Does M Phase Remelt Above 50.5 at. % Ge?

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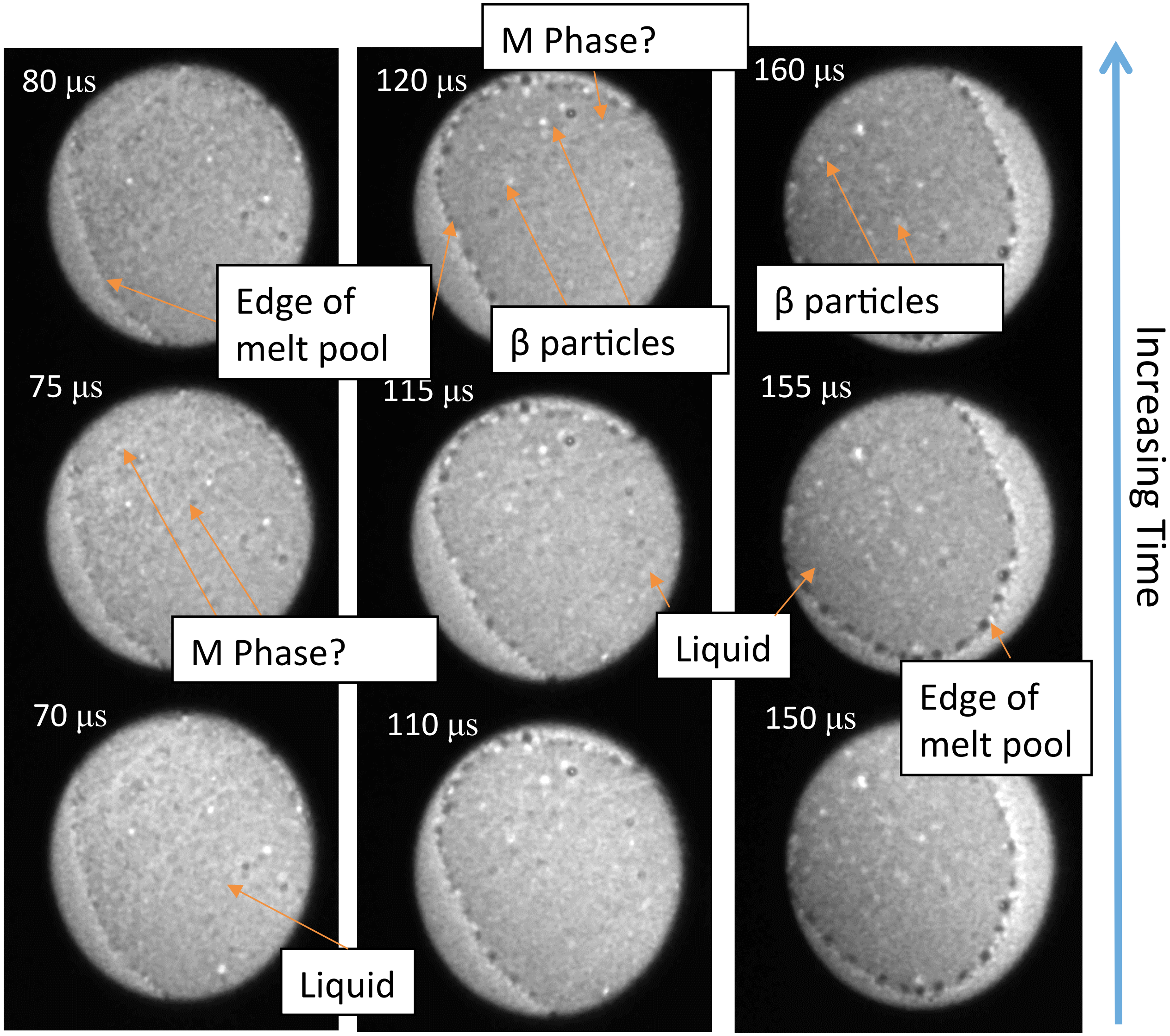

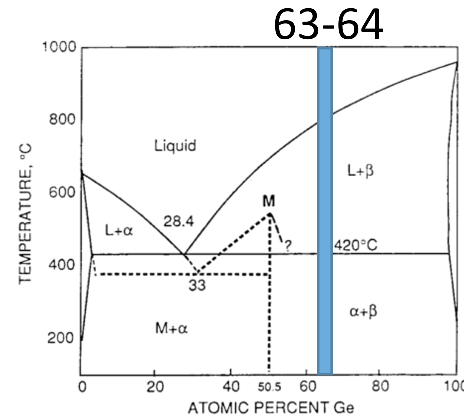

- Again, see areas of what could be M phase
- These disappear and β particles form

Summary

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- DTEM allows for the examination of microstructural evolution dynamics
- Local solute conditions in the liquid dictate the microstructure in the melt pool
- So far, results are consistent with the metastable phase diagram and findings of T. Laoui & M. J. Kaufman
- Remelting of M phase above 50.5 at. % Ge is currently inconclusive, but further DTEM experiments will be performed
- Identification of eutectics above 58 at. % Ge needs to be performed to help inform extension of metastable eutectic

APS Additive Manufacturing Simulator Set-up

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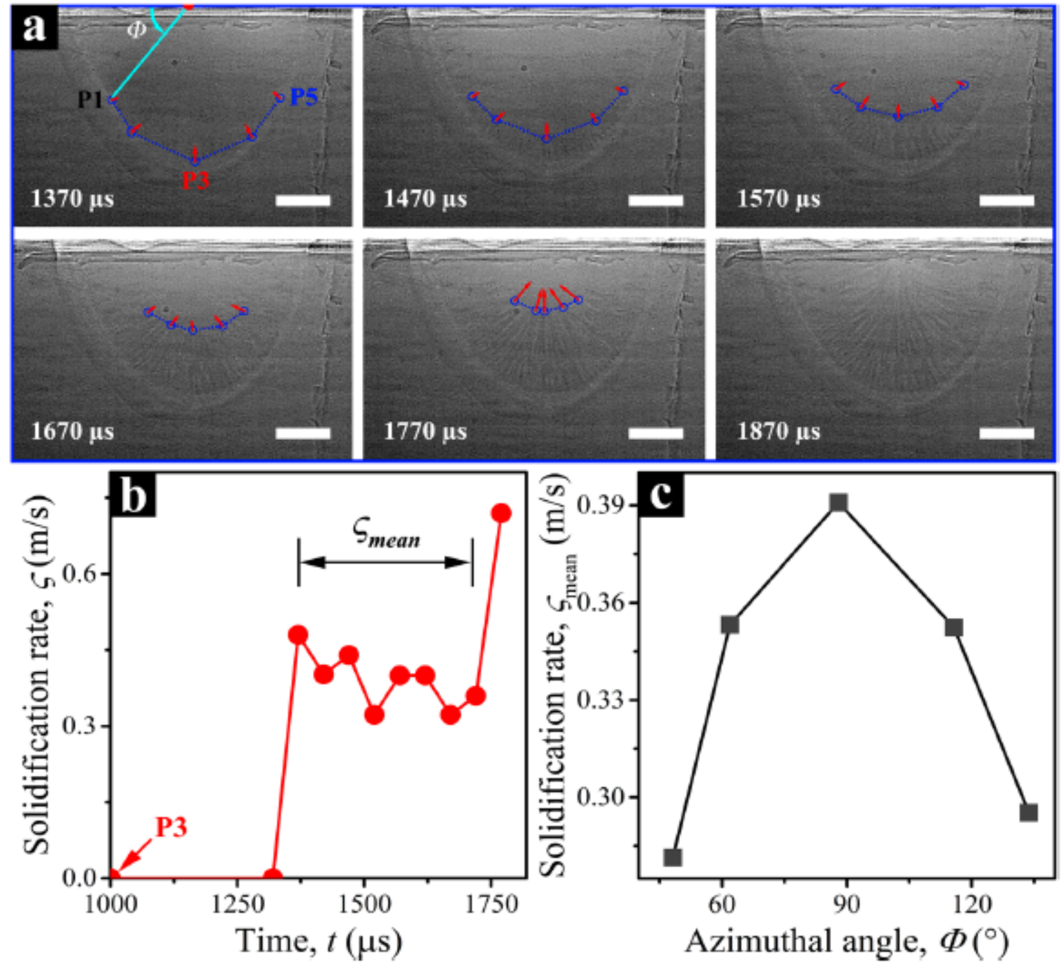
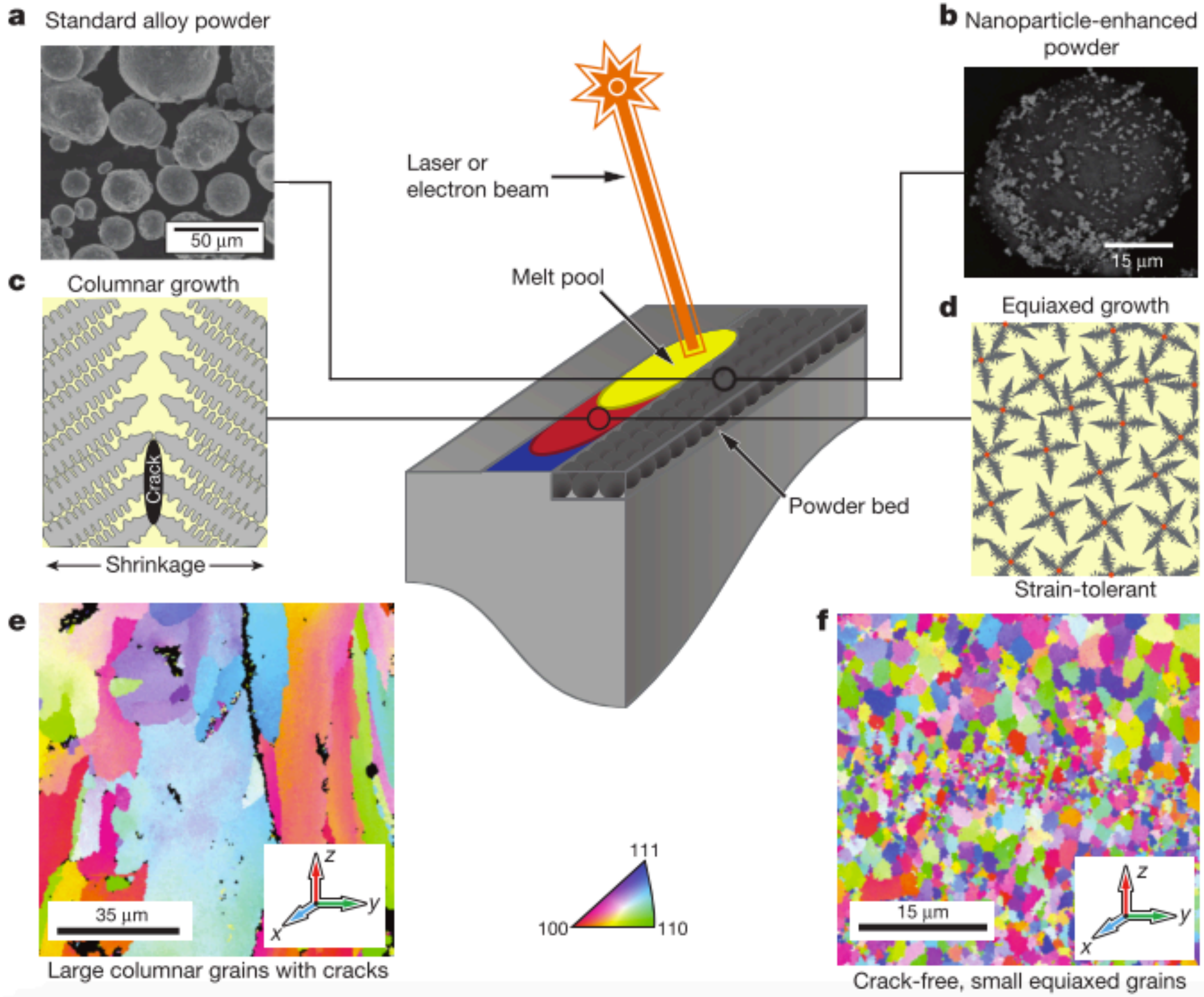


Synchrotron x-ray imaging of a Ti-6Al-4V plate sample in laser melting processes and solidification rate measurements. C. Zhao et al., *Scientific Reports* (2017).

Grain Size Control via Innoculants in AM Alloy Powders



J. H. Martin et al. *Nature* (2017).

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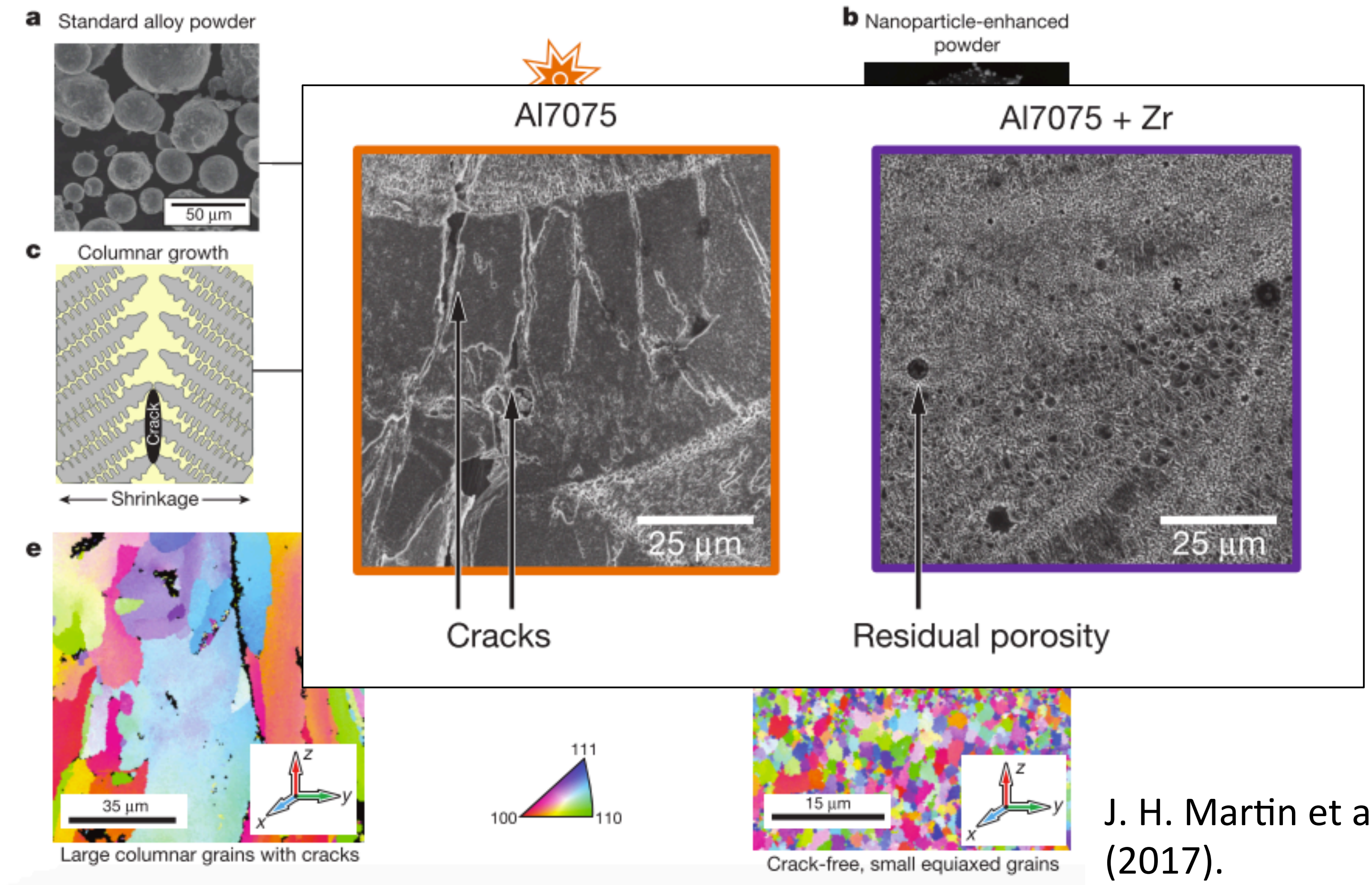

J. H. Martin et al. *Nature* (2017).

Al 6061 MMC Alloy Designed for AM: Initial Characterization

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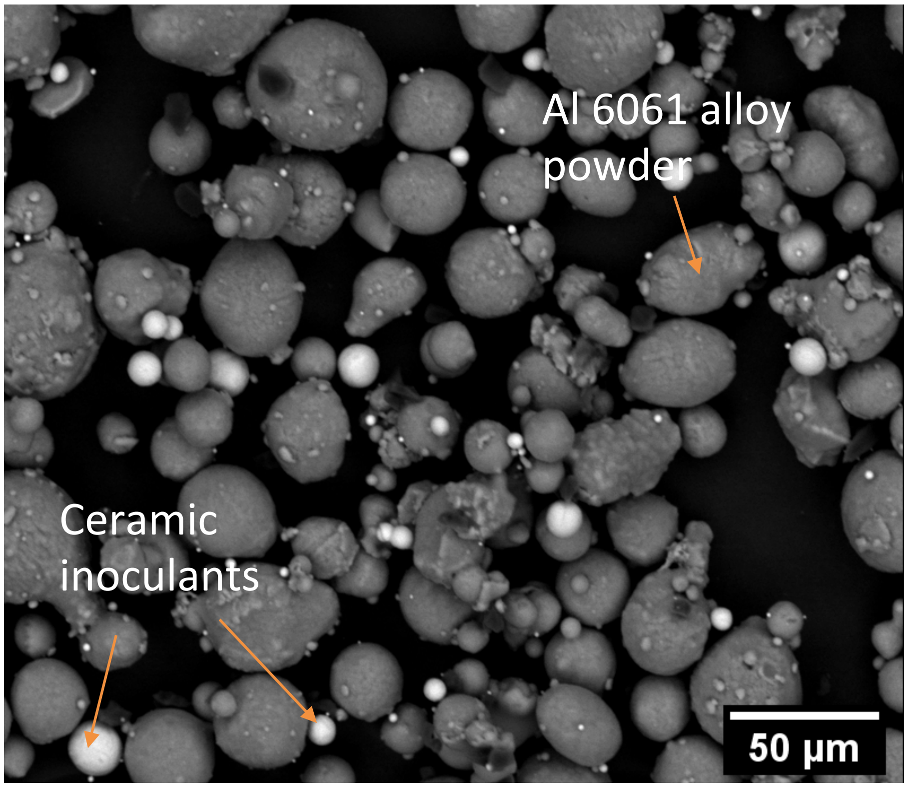
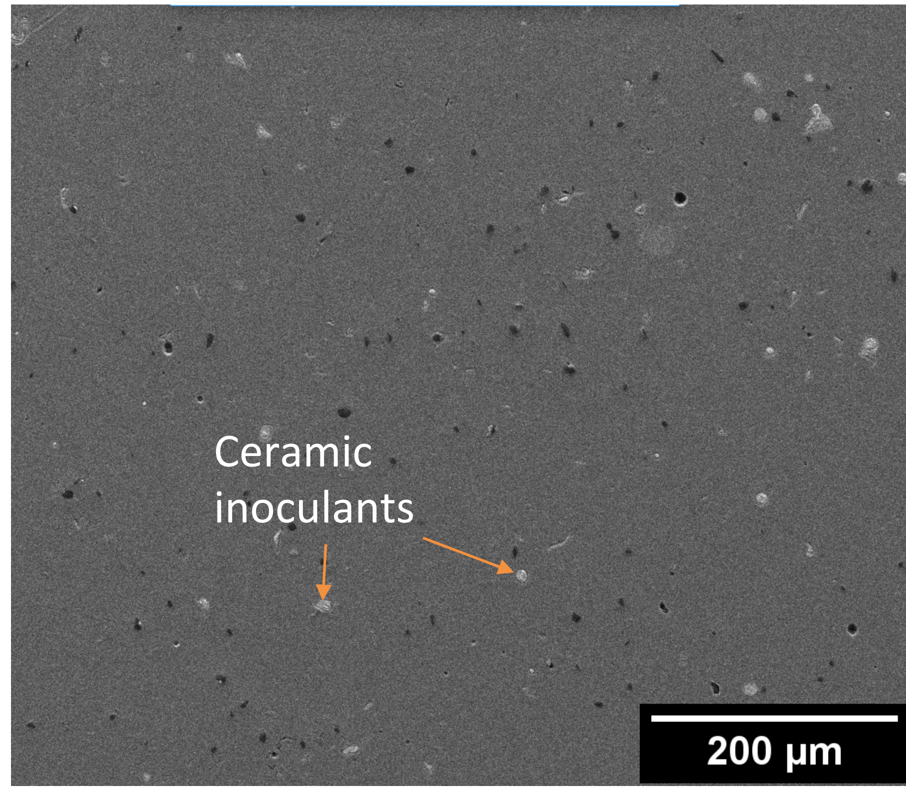


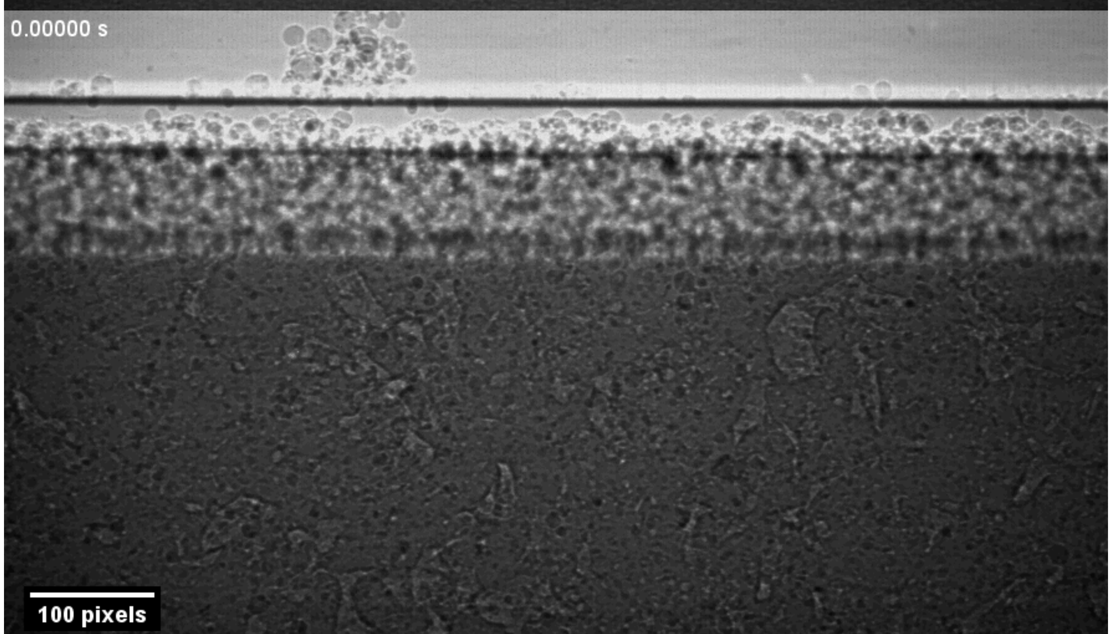
BSE SEM image of Al 6061 MMC alloy powder taken by Chloe Johnson



SEM image of Al 6061 MMC AM build taken by Chloe Johnson

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

Animation of laser pass on Wrought 6061 plate (no powder)
100 W, 0.5 m/s



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

Al-10 at. % Ag Model Alloy: Goals and Experimental Plans

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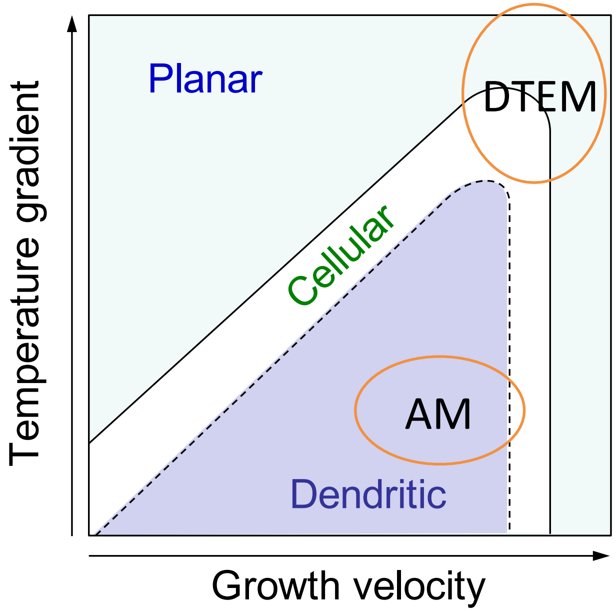
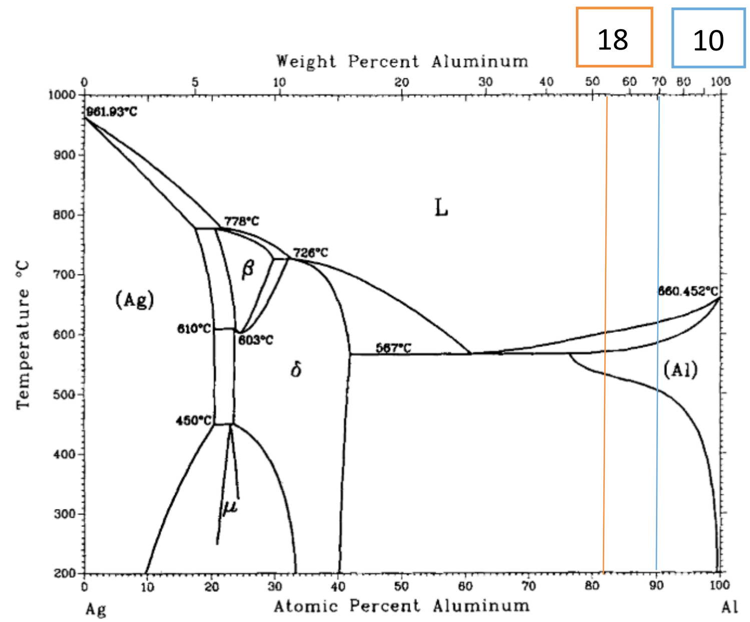
- Investigate affects of microsegregation/solute distribution on final microstructure in rapid solidification, as well as inform solidification models
- Initial rapid solidification experiments were performed using DTEM, which generates high G & R conditions
- Recent experiments using the AM simulator generated lower G & high R values, expanding the range of solidification conditions studied

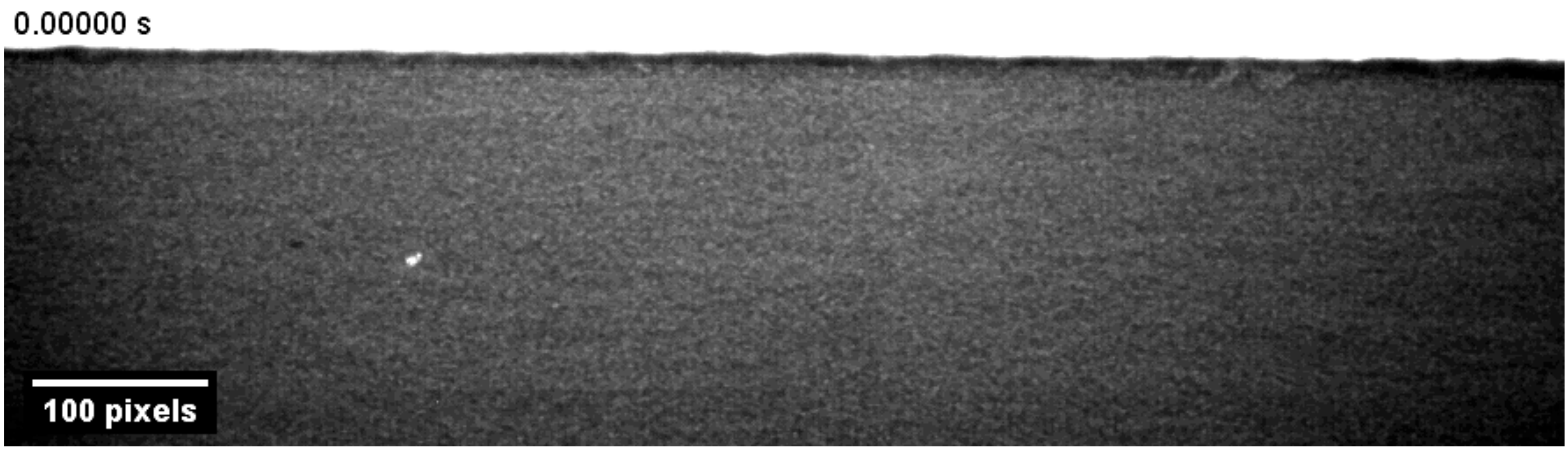


(e.g. [Kurz, Fisher, *Fundamentals of solidification*])



Al-Ag phase diagram (A. J. McAlister, *Bulletin of Alloy Phase Diagrams* (1987))

Al-10 at. % Ag Model Alloy: Animation from AM Simulator



Animation of laser pass done using the AM simulator on Al-10 at. % Ag
360 W, 1 m/s

Future Work

- Al Ge
  - Continue identification of microstructural trends
  - DTEM remelting of M phase
  - Further analysis of local solute conditions
- Al 6061 MMC alloy for AM
  - Post-mortem microstructural evaluation on APS samples
  - Further analysis of grain structure in as-built samples
- Al Ag
  - Post-mortem microstructural evaluation on APS samples
  - Further DTEM experiments
  - Comparison of microstructures from various solidification conditions

Progress

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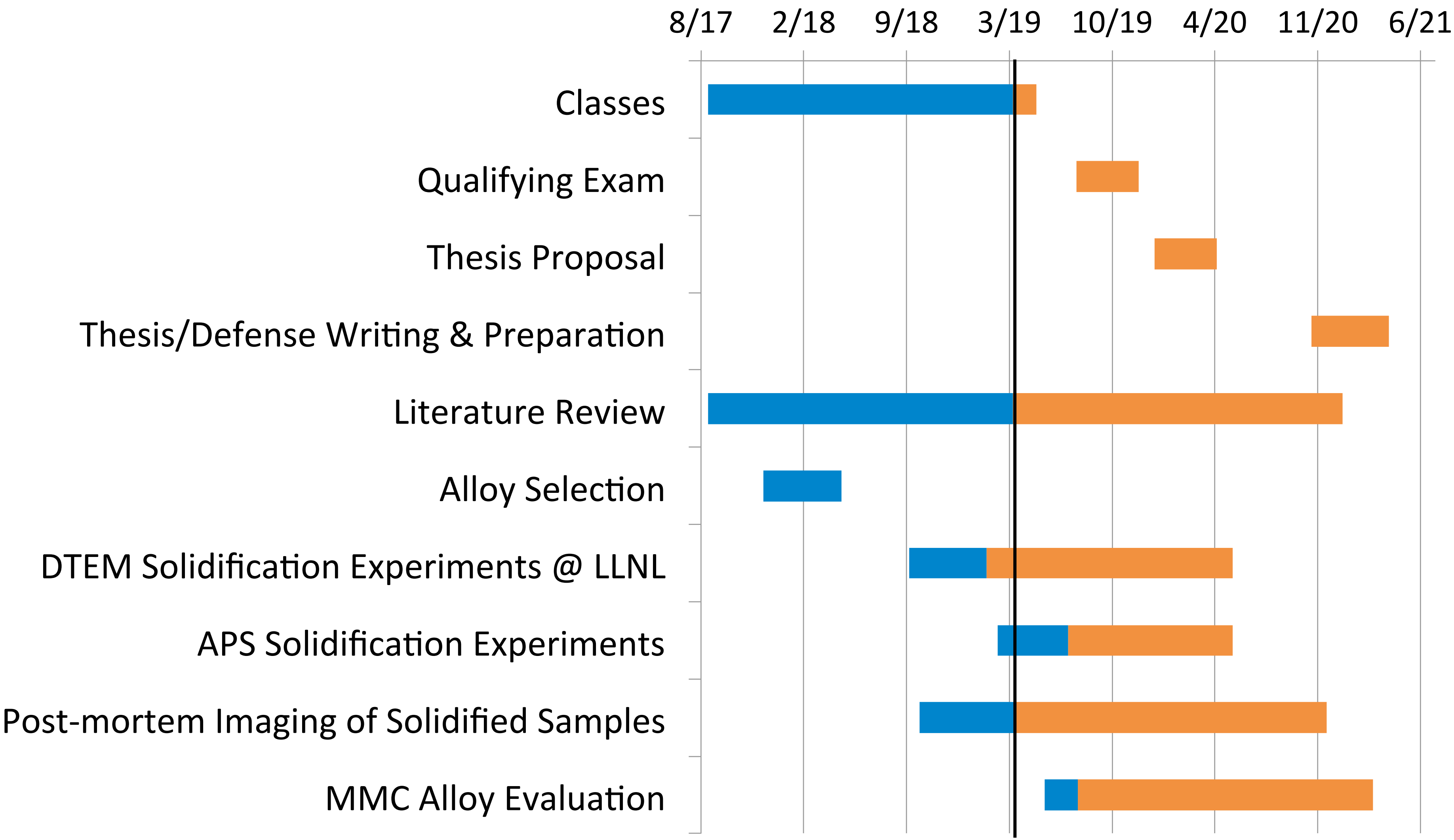

Thank you very much!

Chloe Johnson

chloejohnson@mines.edu