Project 30: Microstructural Evolution of Metallic Alloys During Rapid Solidification (Leveraged)

Spring 2018 Semi-Annual Meeting
Colorado School of Mines, Golden, CO
April 11-12, 2018

Student: Chloe Johnson (CSM)
Faculty: Dr. Amy Clarke (CSM)
Industrial Mentor(s): TBD
Other Participants : Adam Stokes (CSM), Yaofeng Guo (CSM)

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Project 30: Microstructural Evolution of Alloys During Rapid Solidification Dashboard

- 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 with lesser known effects on subsequent solid state phase transformations

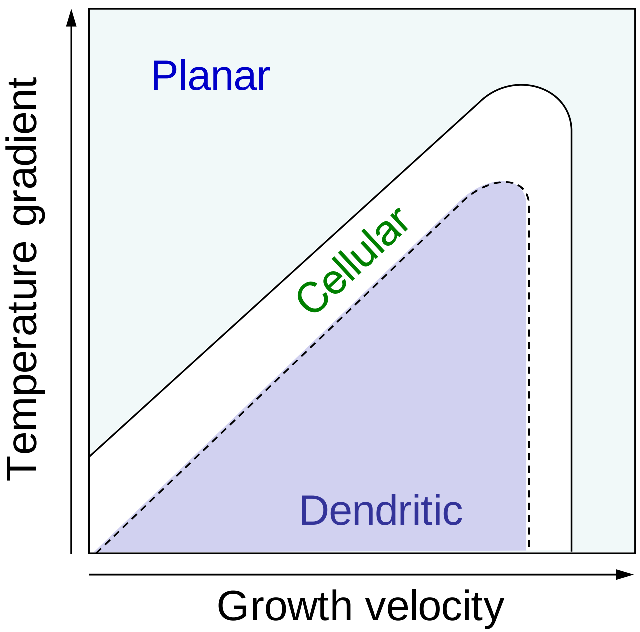
Objective: Understand the relationship of as-solidified microstructures to subsequent solid-state transformations and final microstructures and properties of alloys

Benefit: Inform models, leading to better predictions of microstructural evolution achieved by specific processing conditions

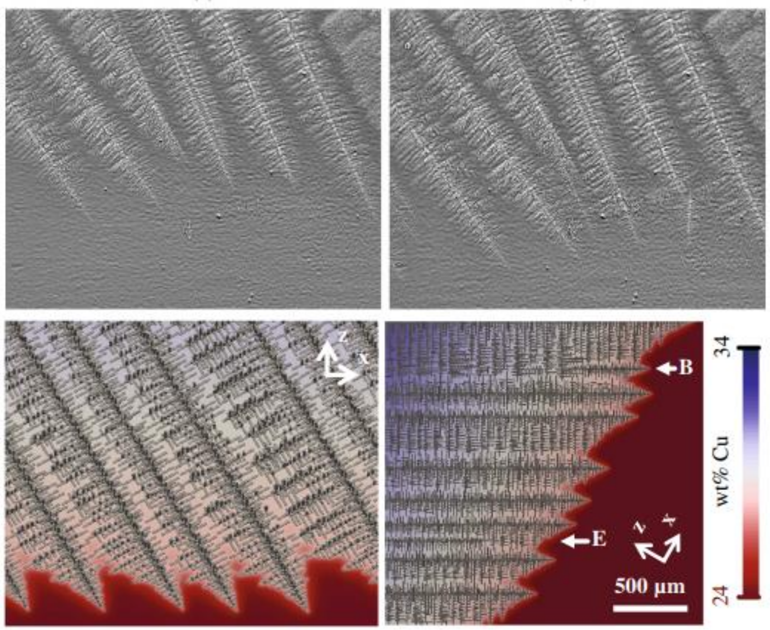
Recent Progress
- Literature review
- Alloy selection
- Sample acquisition
- Advanced Photon Source (APS) at Argonne National Laboratory user proposal submitted
- Dynamic Transmission Electron Microscopy (DTEM) collaboration with Lawrence Livermore National Laboratory

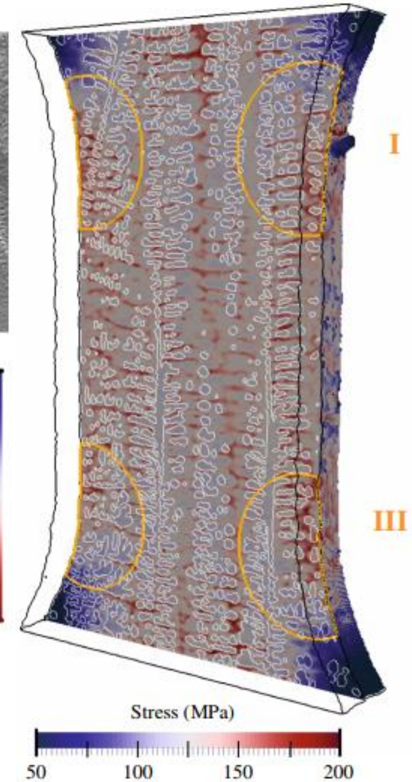
Metrics		
Description	**% Complete**	**Status**
1. Literature review	20%	●
2. Alloy selection	100%	●
2. Characterization (ex/in-situ) of samples solidified under rapid and conventional conditions	10%	●
3. In-situ solid state phase transformation experiments	0%	●
4. Evaluation of precipitation strengthening via micropillar compression	0%	●

Processing-Microstructure-Property Relationships



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





D. Tourret *et al.*, "From solidification processing to microstructure to mechanical properties: a multi-scale x-ray study of an Al-Cu alloy sample", *Metallurgical and Materials Transactions A,* **48,** 5529–5546 (2017).

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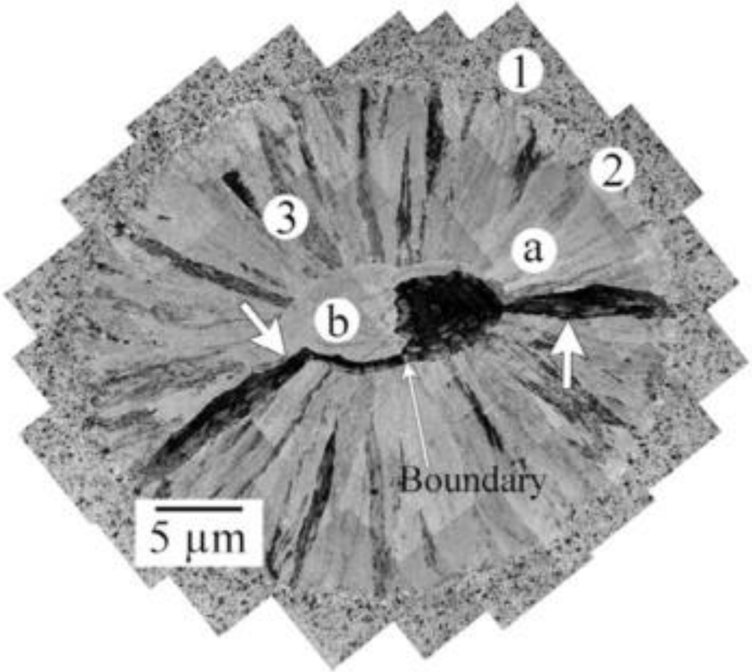
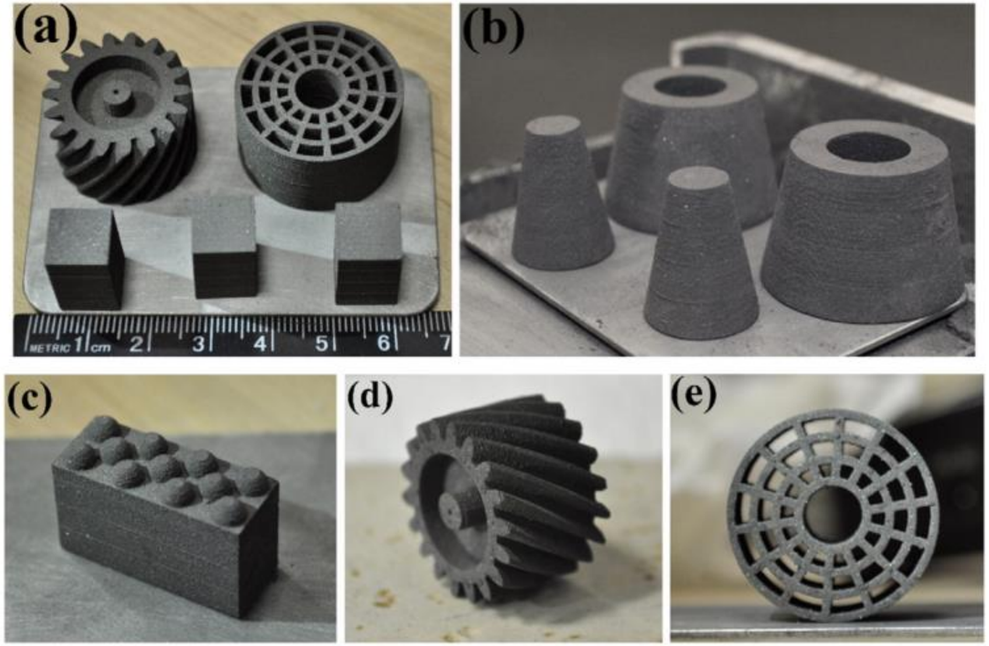
Industrial Relevance



Montage of conventional BF TEM images showing three distinct morphological zones in a resolidified Al-Cu alloy film.

J.T. McKeown *et al.*, "In situ transmission electron microscopy of crystal growth-mode transitions during rapid solidification of a hypoeutectic Al-Cu alloy", *Acta Materialia* **65,** 56–68 (2014).



3D-printed TiCx preforms with various geometries

A. Levy, A.Miriyev, A. Elliott, S.S. Babu, N. Frage, "Additive manufacturing of complex-shaped graded TiC/steel composites", *Materials and Design* **118,** 198–203 (2017).

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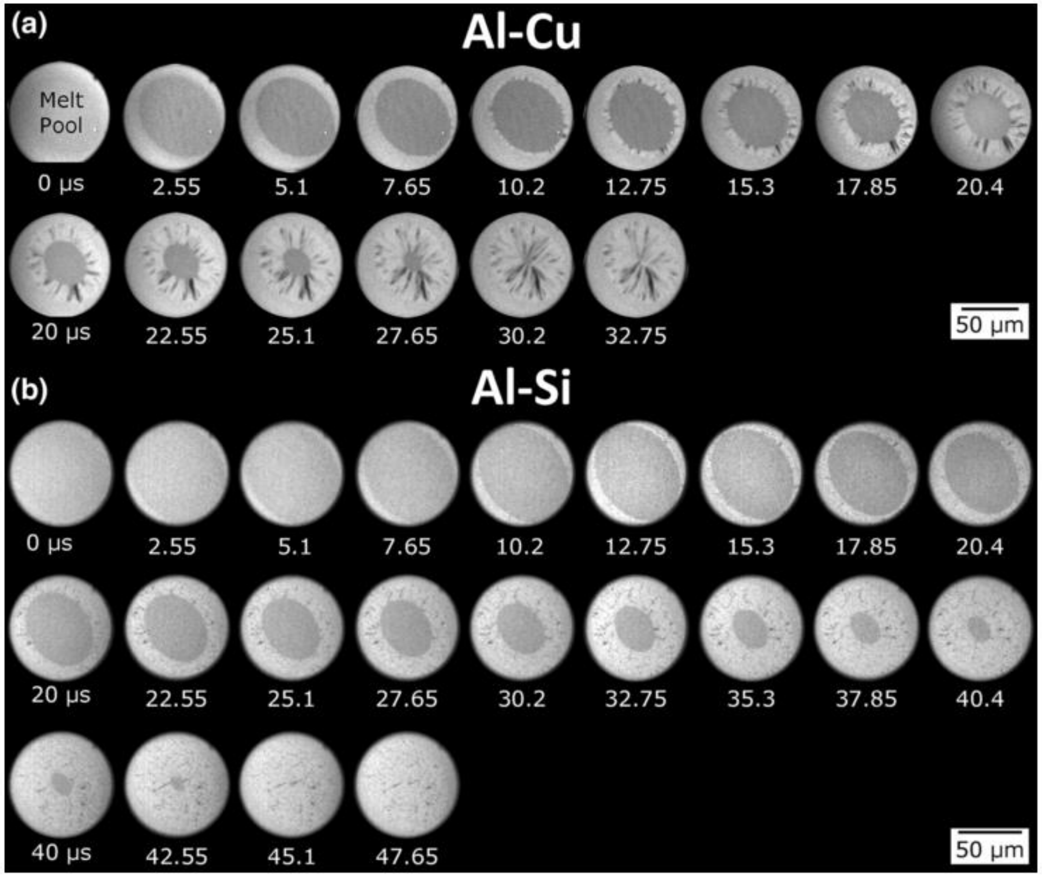
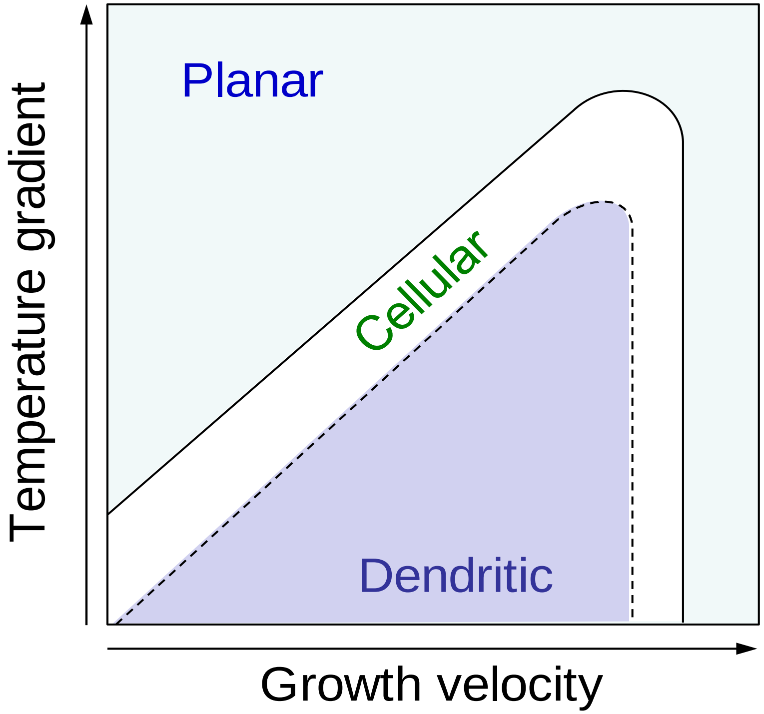
Solid-Liquid Transformations: Far from Equilibrium in the DTEM



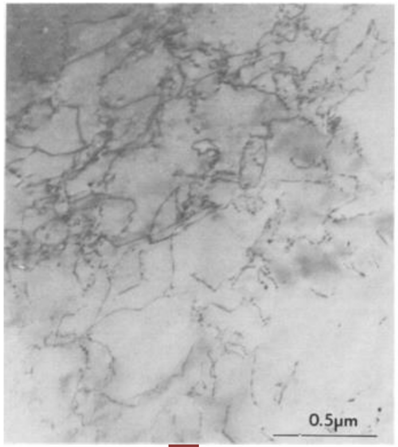


DTEM imaging during rapid solidification in (a) Al–4Cu and (b) Al–3Si alloy thin films after pulsed-laser melting [J.T. Mckeown et al., JOM 2016, 3:68].

Solidification and Precipitation in Al-Mn Alloys

< 5 wt% Mn

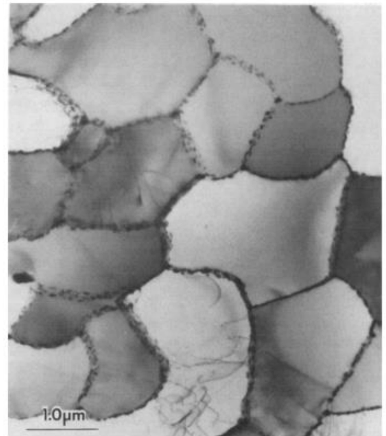
As Solidified Structure



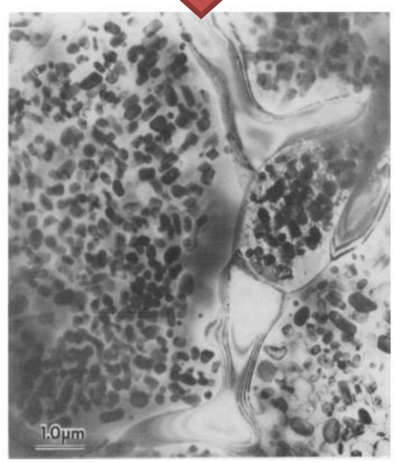
Aging 1 Hr at 450°C

5-15 wt% Mn

As Solidified Structure

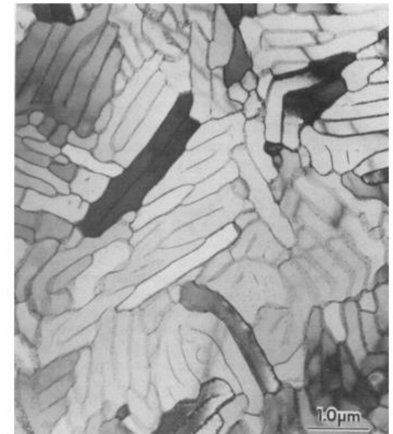


Aging 16 Hr at 450°C

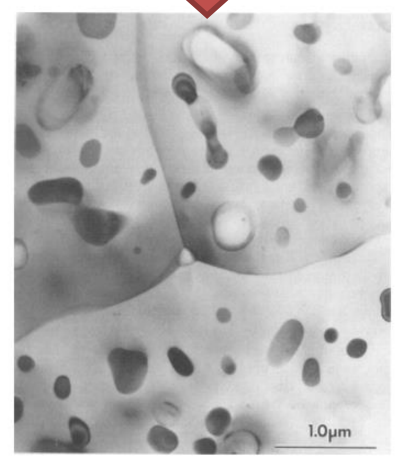


>15 wt% Mn

As Solidified Structure



Aging 6 Hr at 450°C



Taken From: Shechtman, D., Schaefer, R. J. & Biancaniello, F. S. Precipitation in rapidly solidified Al-Mn alloys. *Metallurgical Transactions A* **15,** 1987–1997 (1984).

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Proposed Experiments

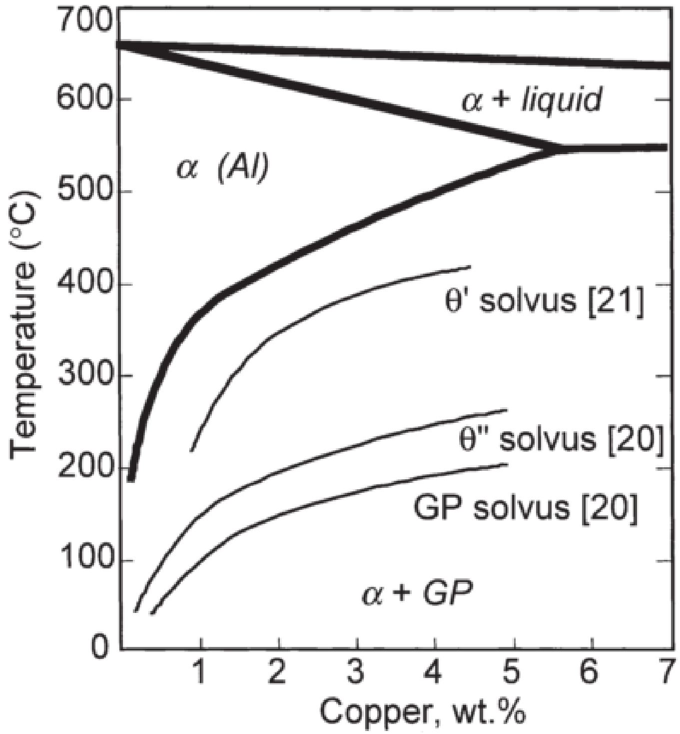
Phase Transition, Scale, Technique, and Synthesis Pathways that will be Explored for Different Aluminum Alloys				
Phase Transition	Scale	Technique	Synthesis Type	Alloy(s)
Solid-Liquid	Film, 2D/Small	DTEM @ LLNL	Far from Equilibrium	Al-Cu, Al-Ag
Solid-Liquid	Bulk, Small	2D & 4D X-ray Imaging @ APS	Near -> Far from Equilibrium	Al-Cu, Al-Ag
Solid-Solid	Film, 2D/Small	TEM/Hot Stage @ Mines, TEM/HRTEM/Hot Stage @ CINT-SNL	Near -> Far from Equilibrium	Al-Cu, Al-Ag, Al-Cu-Ag
Solid-Solid	Bulk, Small	4D TXM @ APS	Near Equilibrium	Al-Cu, Al-Ag, Al-Cu-Ag
Solid-Solid	Bulk, Small	APT @ Mines	Near -> Far from Equilibrium	Al-Cu, Al-Ag, Al-Cu-Ag

Aluminum Alloy Systems

Use as model systems for studying rapid solidification and solid-state phase transformations (e.g. precipitation)

Consider

- Al-Cu: GP zones ➔ θ” ➔ θ’➔ θ
- Al-Ag: GP zones ➔ γ’ ➔ γ
- Al-Cu-Ag: Combination



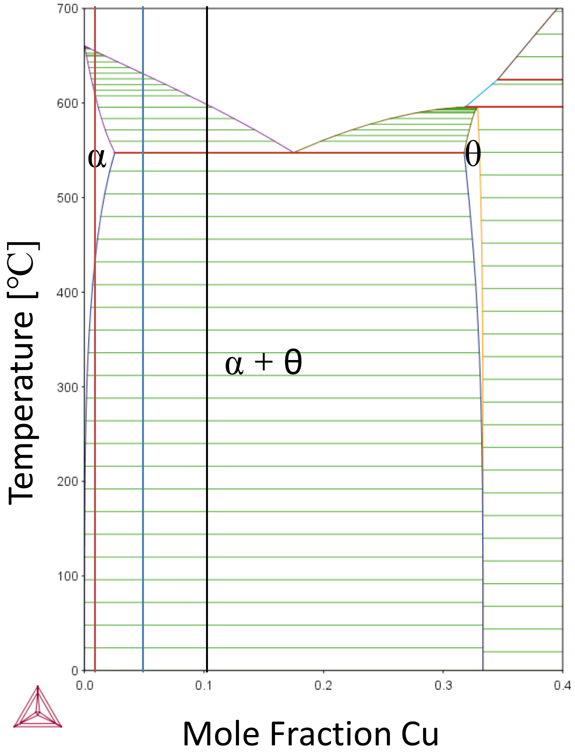
Al-rich corner of the Al–Cu phase diagram showing the metastable solvus boundaries for GP zones, θ” and θ’, together with the equilibrium solvus line for the θ phase.

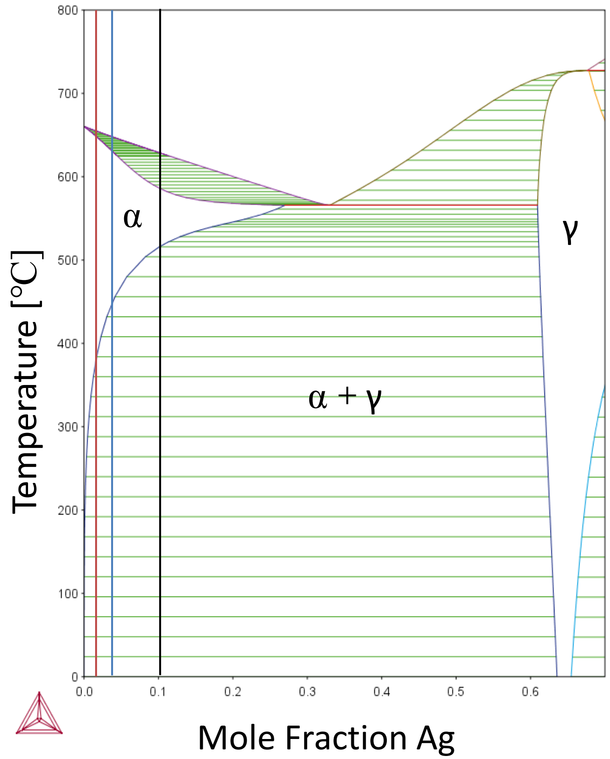
S.P. Ringer and K. Hono, “Microstructural evolution and age hardening in aluminium alloys: atom probe field-ion microscopy and transmission electron microscopy studies”, *Materials Characterization,* **44,** 101–131 (2000).

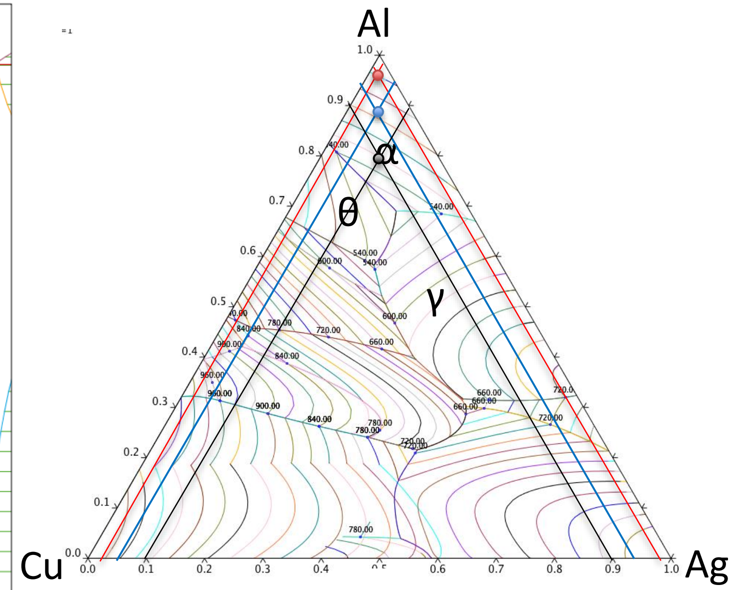
Alloy Selection: Al-Cu, Al-Ag, Al-Cu-Ag

Aluminum Alloy Compositions			
Alloy	Sample 1 wt. % (at. %)	Sample 2 wt. % (at. %)	Sample 3 wt. % (at. %)
Al-Cu	Al-1.9 Cu (Al-0.8 Cu)	Al-10 Cu (Al-4.5 Cu)	Al-20 Cu (Al-9.6 Cu)
Al-Ag	Al-3 Ag (Al-0.8 Ag)	Al-14.3 Ag (Al-4.5 Ag)	Al-30 Ag (Al-9.6 Ag)
Al-Cu-Ag	Al-1.9 Cu-3 Ag (Al-0.8 Cu-0.8 Ag)	Al-10 Cu-14.3 Ag (Al-4.5 Cu-4.5 Ag)	Al-20 Cu-30 Ag (Al-9.6 Cu-9.6 Ag)

Alloy Selection: CALPHAD Phase Verification

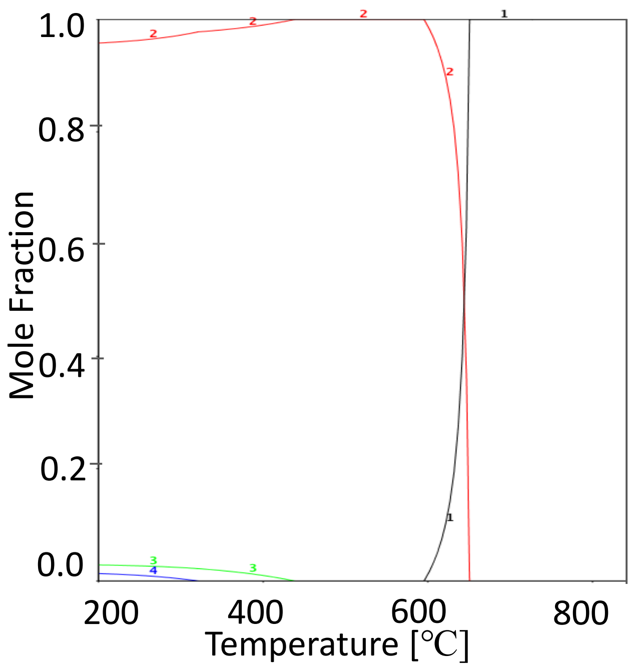




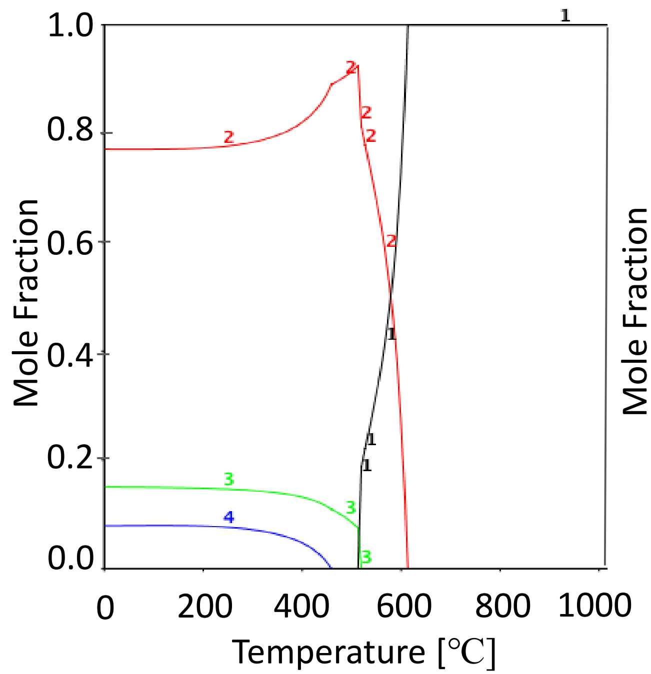


1 at. %	red line
5 at. %	blue line
10 at. %	black line

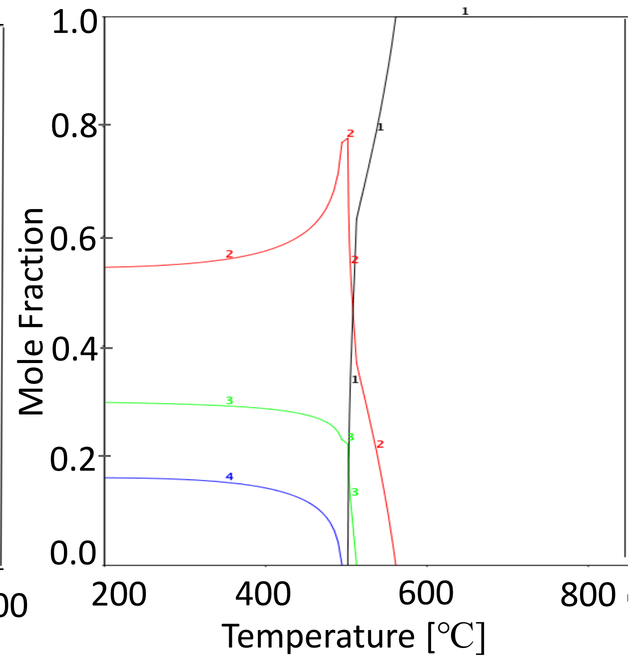
Alloy Selection: CALPHAD Phase Verification (Al-Cu-Ag)



Al-1 at.% Cu-1 at. % Ag



Al-5 at.% Cu-5 at. % Ag



Al-10 at.% Cu-10 at. % Ag

FCC_Al (α)	red
HCP_ $AlAg_2$ (γ)	blue
Al_2Cu (Θ)	green
Liquid	black

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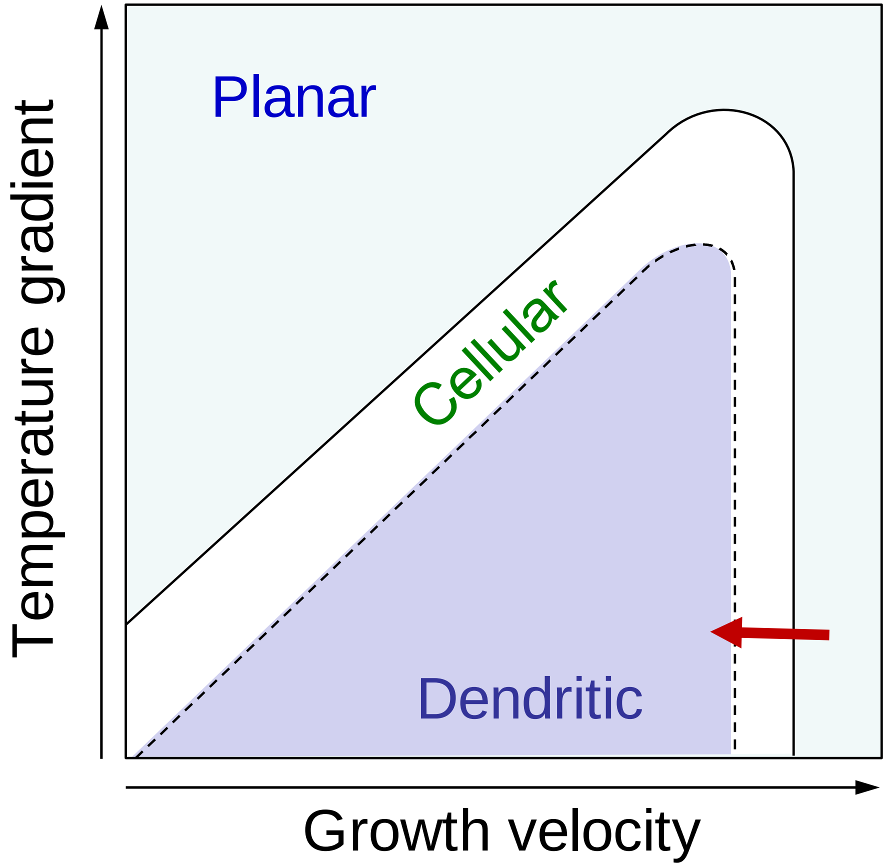
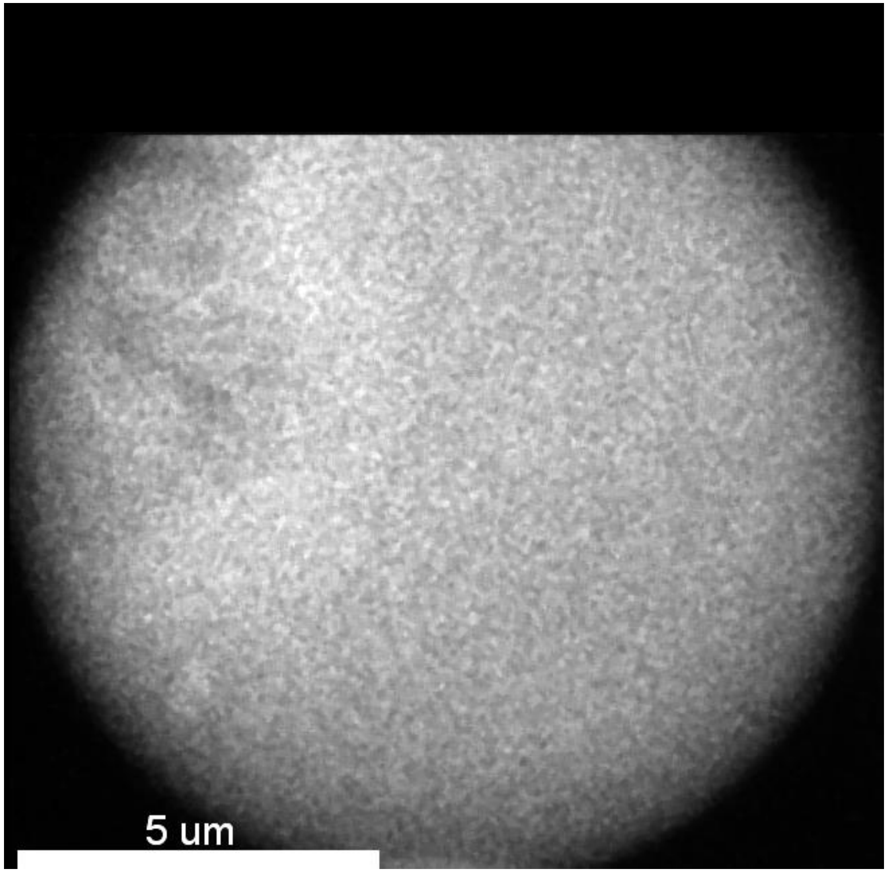
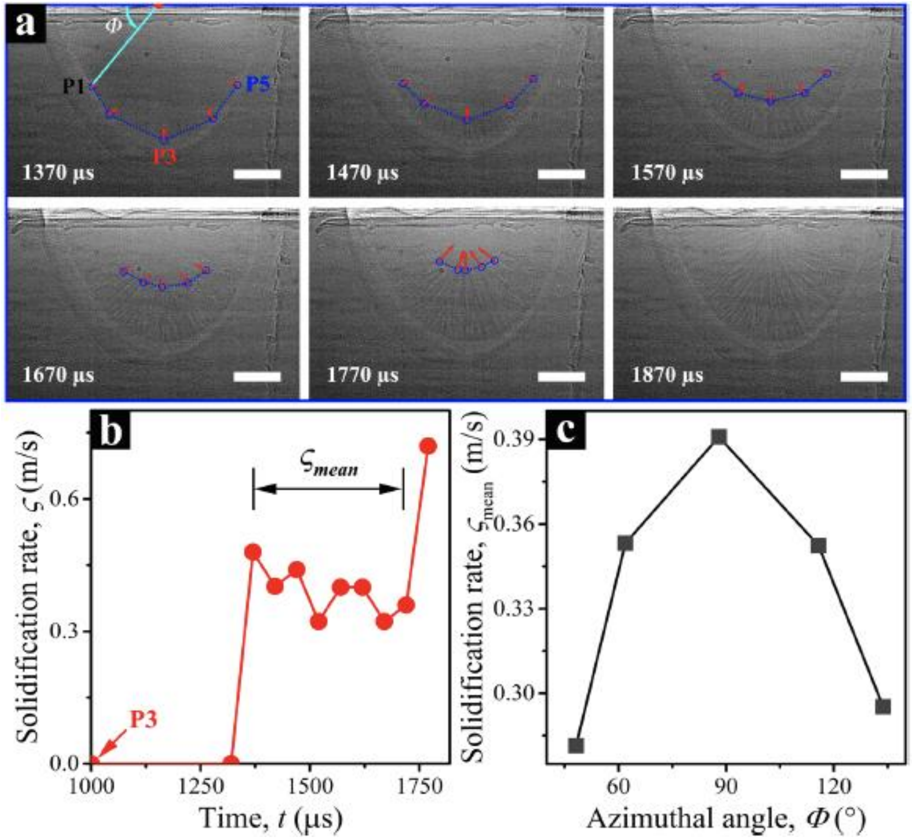
Hot-Stage DTEM Rapid Solidification Studies





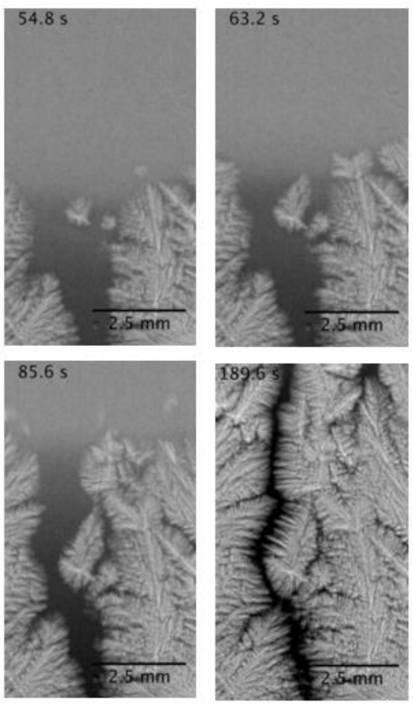
AM Solidification Studies at APS

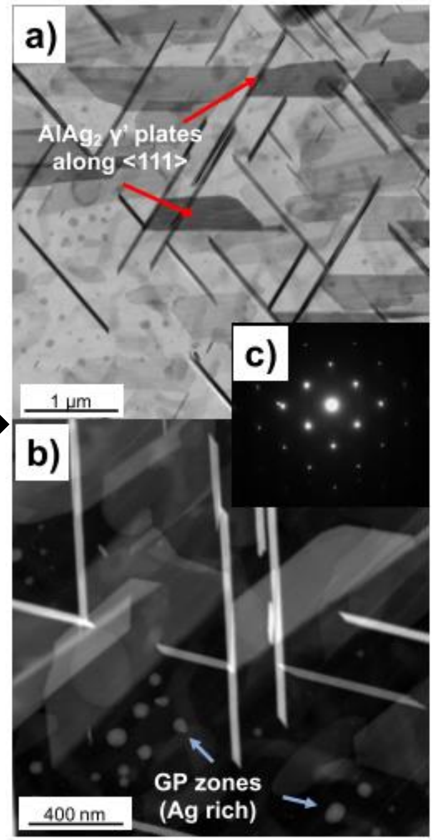
Right: Synchrotron x-ray imaging of a Ti-6Al-4V plate sample in laser melting processes and solidification rate measurements [C. Zhao et al., Scientific Reports 2017, 7:3602].

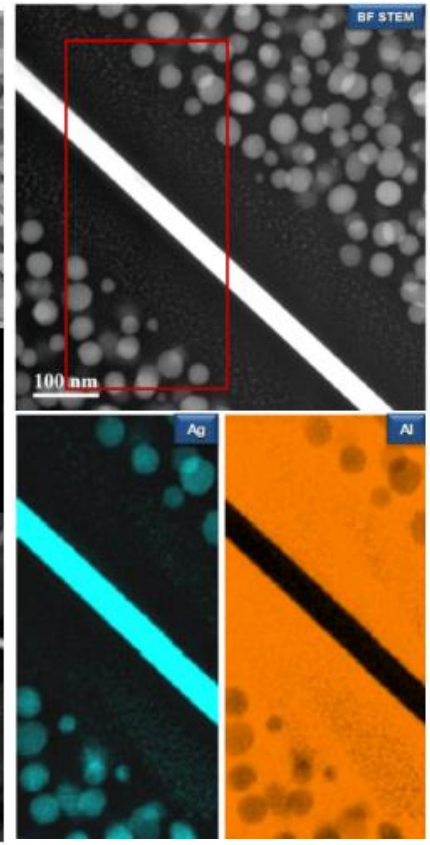


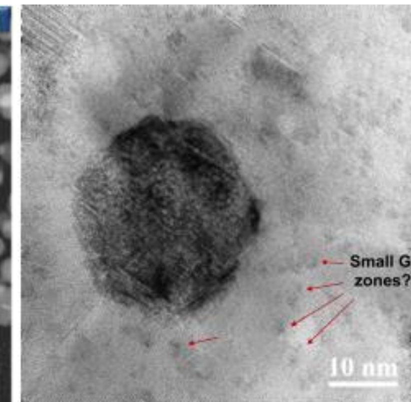
In-Situ Imaging of Al-Ag and Solid State Phase Transformations

High-energy x-ray imaging, highlighting Al-Ag solidification dynamics during controlled directional solidification



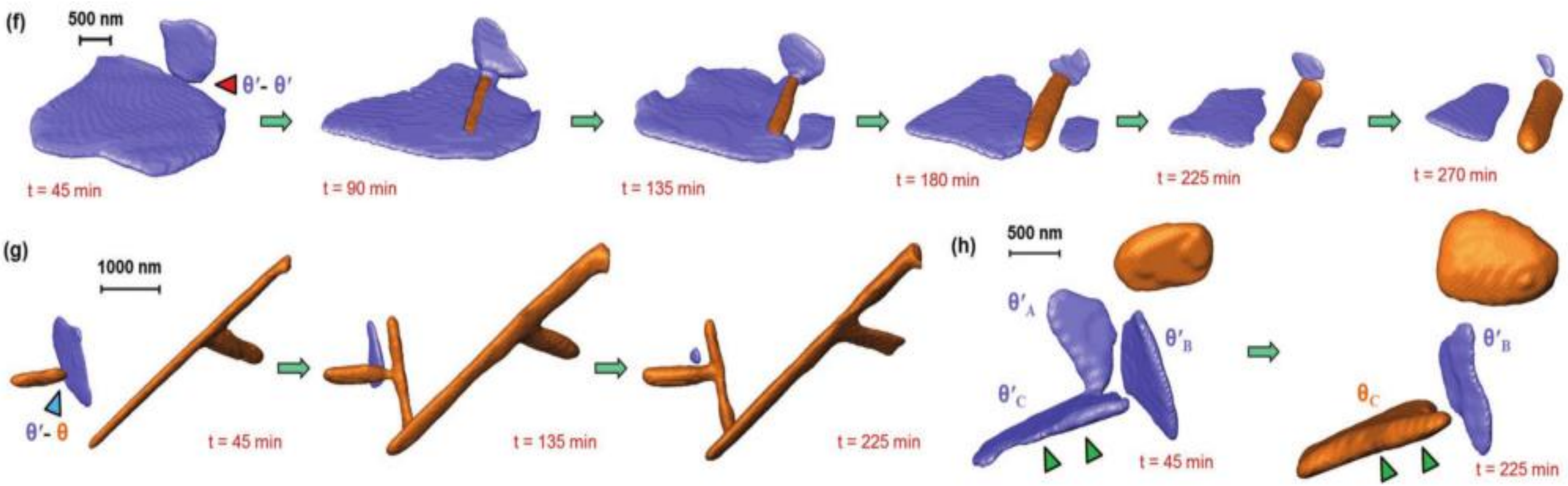






GP zones and γ' precipitation in an Al-Ag alloy imaged with advanced electron microscopy techniques

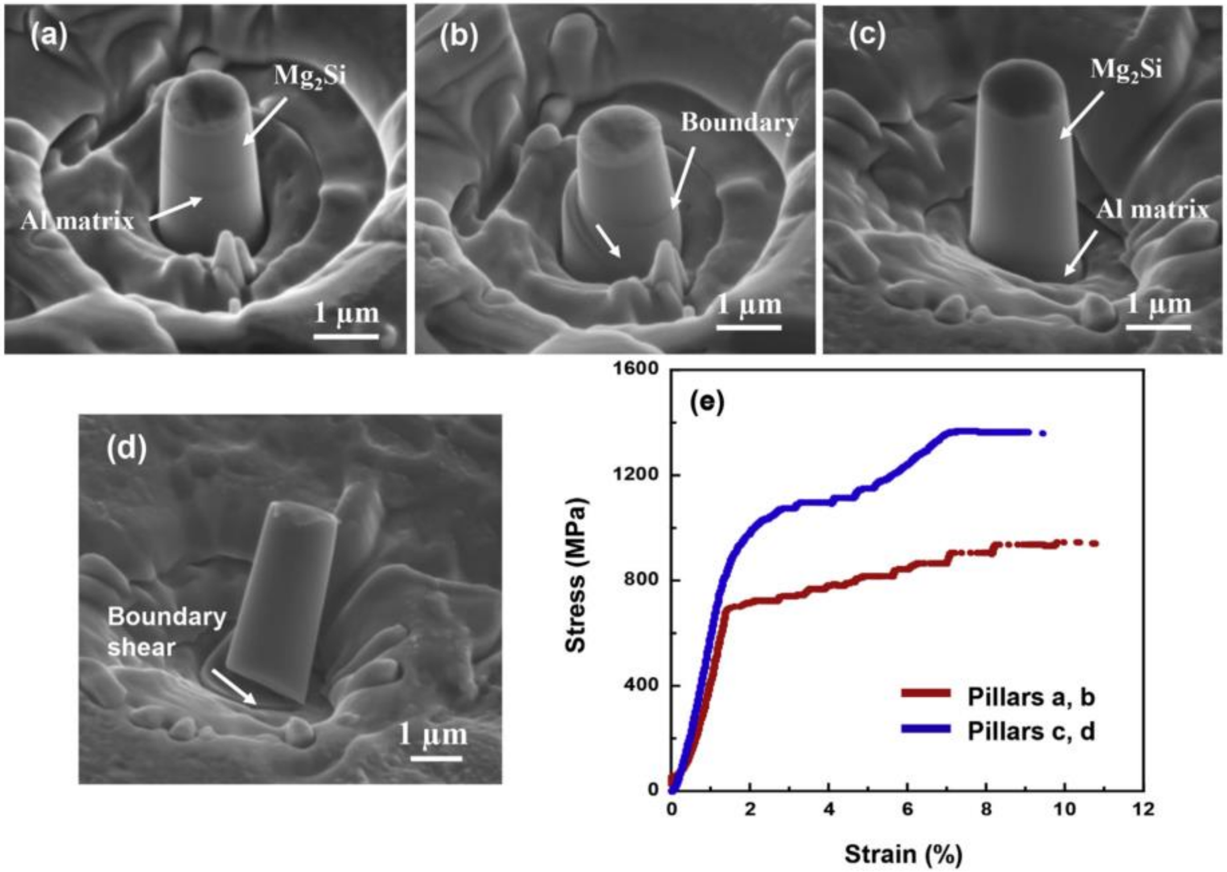
4-D X-ray Microscopy of Precipitation



(f) 3D nondestructive microstructural characterization using TXM:
(g) Nucleation and growth of θ from θ′ to θ ′ intersection.
(h) Varying transformation of different θ′ precipitates.

C.S. Kaira *et al.*, "Probing novel microstructural evolution mechanisms in aluminum alloys using 4D nanoscale characterization", *Advanced Materials* **29,** (2017).

Micropillar Compression Testing

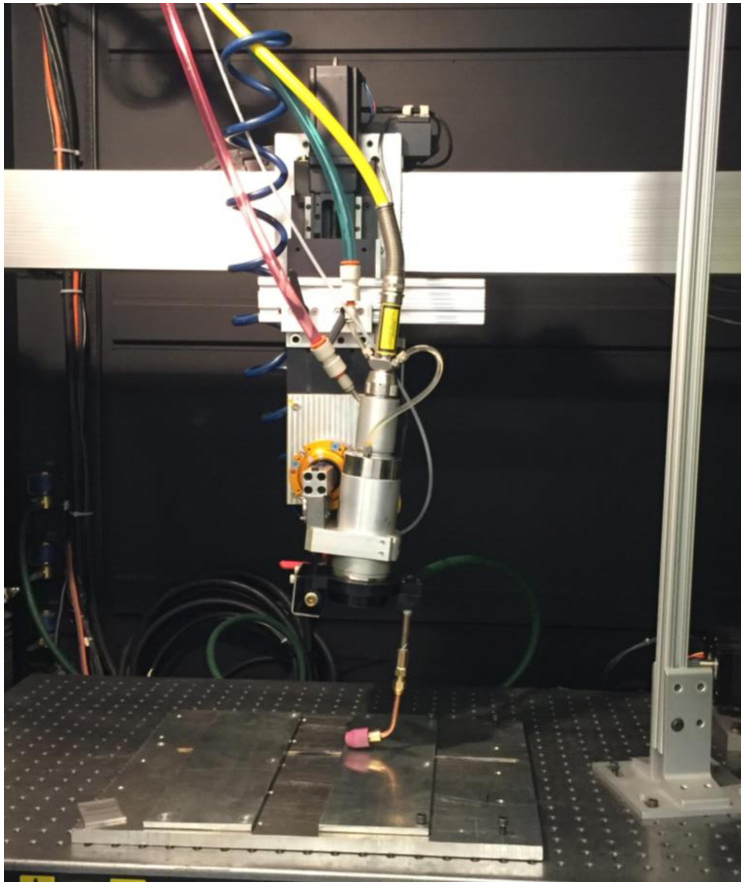


(a-d) Deformation of Si-bearing inclusion (Mg2Si) pillars with Al at the base and (e) stress-strain curves showing the decrease in stress values of Si-bearing inclusions due to presence of the Al matrix

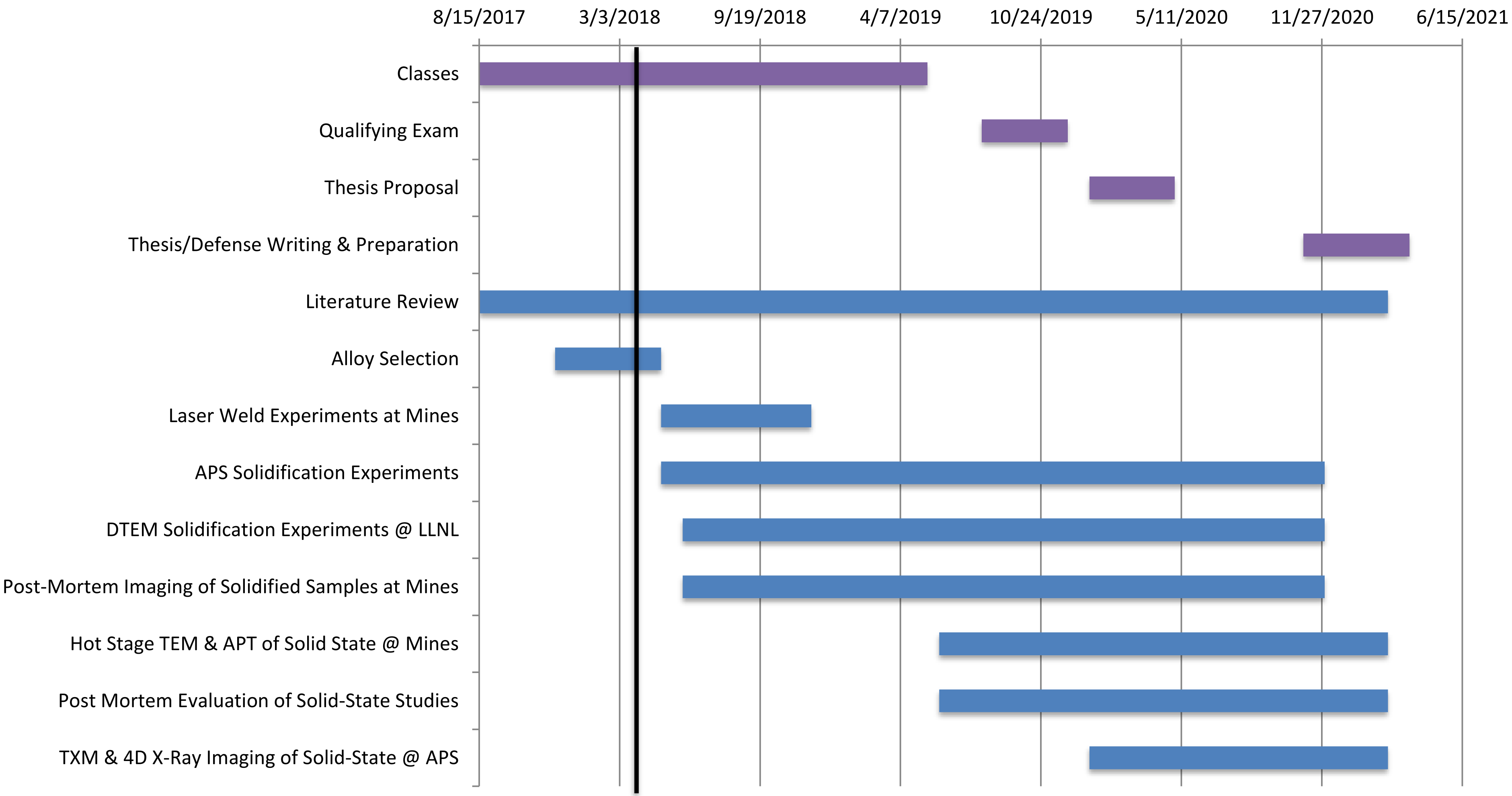
S.S. Singh, E. Guo, H. Xie, N. Chawla, "Mechanical properties of intermetallic inclusions in Al 7075 alloys by micropillar compression", *Intermetallics* **62,** 69–75 (2015).

Preparation for APS: Preliminary Rapid Solidification Studies

Laser welding set up at Mines.



Progress


8/15/2017
3/3/2018
9/19/2018
4/7/2019
10/24/2019
5/11/2020
11/27/2020
6/15/2021
Classes
Qualifying Exam
Thesis Proposal
Thesis/Defense Writing & Preparation
Literature Review
Alloy Selection
Laser Weld Experiments at Mines
APS Solidification Experiments
DTEM Solidification Experiments @ LLNL
Post-Mortem Imaging of Solidified Samples at Mines
Hot Stage TEM & APT of Solid State @ Mines
Post Mortem Evaluation of Solid-State Studies
TXM & 4D X-Ray Imaging of Solid-State @ APS

Project 30 – Microstructural Evolution of Metallic Alloys During Rapid Solidification

Graduate Student – Chloe Johnson (CSM)
Faculty/Advisors – Amy Clarke (CSM)
Industrial Mentors – TBD

Program Goal

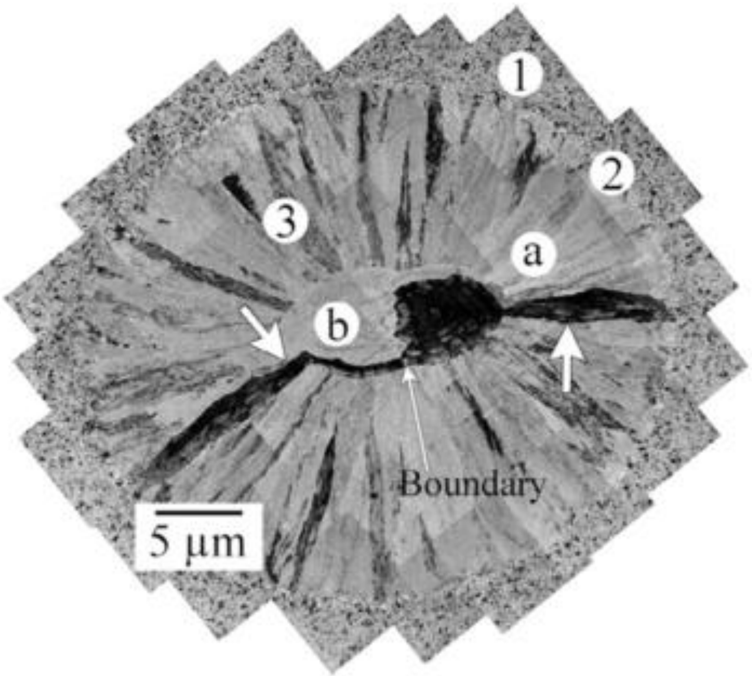
Understand the effect of rapid solidification on the as-solidified microstructure and subsequent solid-state phase transformations.

Approach

Use in-situ and post mortem imaging techniques to capture and characterize the mechanisms controlling microstructural development during far from equilibrium and equilibrium solidification.

Benefits

In-situ characterization of solid-liquid and solid-state phase transformations will give a full understanding of the solidification pathway and help inform models.



TEM image of a rapidly solidified Al-Cu alloy

Project Duration

Ph.D. August 2017 – May 2021

Thank you very much!

Chloe Johnson

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