Project 30-L: Microstructural Evolution of Metallic Alloys during Rapid Solidification

Fall 2018 Semi-Annual Meeting
Colorado School of Mines, Golden, CO
October 2-4, 2018

Student: Chloe Johnson (Mines)

Faculty: Amy Clarke (Mines)

Industrial Mentors: Los Alamos National Laboratory, Boeing

Other Participants: Yaofeng Guo (Mines), Joe Jankowski (Mines), Francisco Coury (Mines), Joe McKeown (Lawrence Livermore National Laboratory)

Project 30: Microstructural Evolution of Metallic Alloys during Rapid Solidification

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Center for ADVANCED NON-FERROUS STRUCTURAL ALLOYS

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

Project Duration
PhD: August 2017 to May 2021

- Problem: Microstructural evolution during rapid solidification is not well understood.
- Objective: Understand the formation of unique microstructures (e.g. metastable phases, grain sizes and morphologies, local chemistry) in aluminum alloys.
- Benefit: Understand and control microstructural evolution during rapid solidification to inform industrial processes (e.g. additive manufacturing, welding, etc.) and the development of microstructural models.

Recent Progress
- Surveyed different techniques for rapid solidification that produce a range of solidification processing conditions that will be used in the project
- Received aluminum 6061 metal matrix composite (MMC) powder for future in-situ solidification studies
- Laser welding studies performed to evaluate solidification conditions and resultant microstructure
- User proposal submitted to the Advanced Photon Source (APS) at Argonne National Laboratory for in-situ imaging of simulated additive manufacturing
- Study metastable phases and microstructural evolution in Al-Ge with dynamic transmission electron microscopy (DTEM)

Metrics

Description	% Complete	Status
1. Literature review	25%	●
2. Alloy selection and survey of rapid solidification processing techniques	40%	●
3. In-situ imaging of binary and MMC aluminum alloys	20%	●
4. Post-mortem microstructural characterization of phases and microstructures	5%	●
5. Correlate microstructural evolution during rapid solidification to (local) processing conditions	0%	●

Industrial Relevance

Rapid Solidification

↓

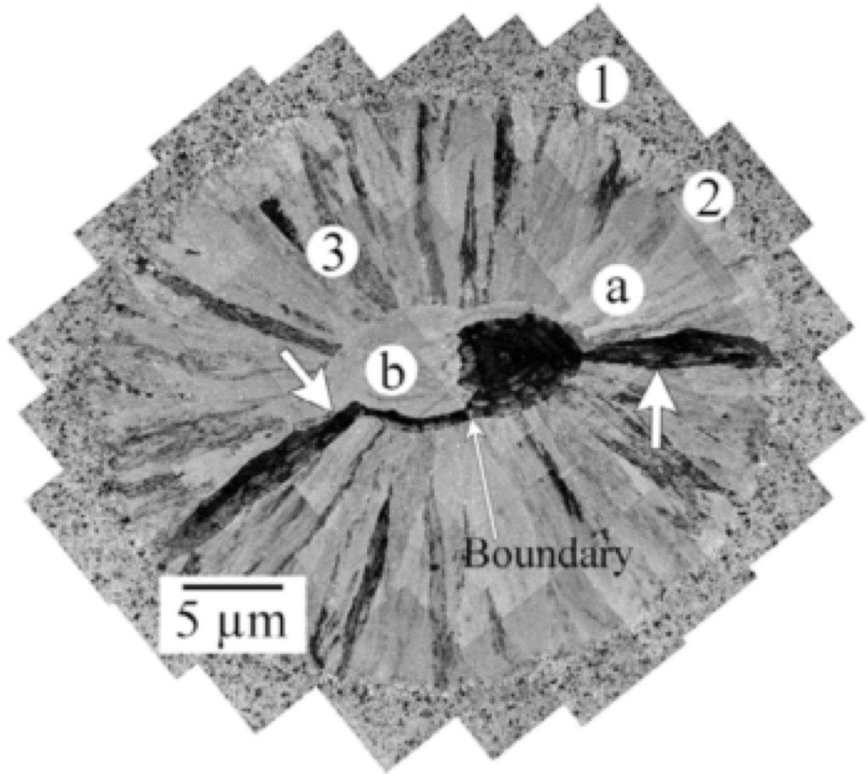
Microstructure

↓

e. g. Metastable Phases
Grain size/morphology,
Local chemistry

↓

Industrial Processes
(e. g. Additive Manufacturing)



Post-mortem TEM of an Al-7at.%Cu alloy film after DTEM solidification [J.T. McKeown et al., Acta Materialia (2014), 65].

Overview

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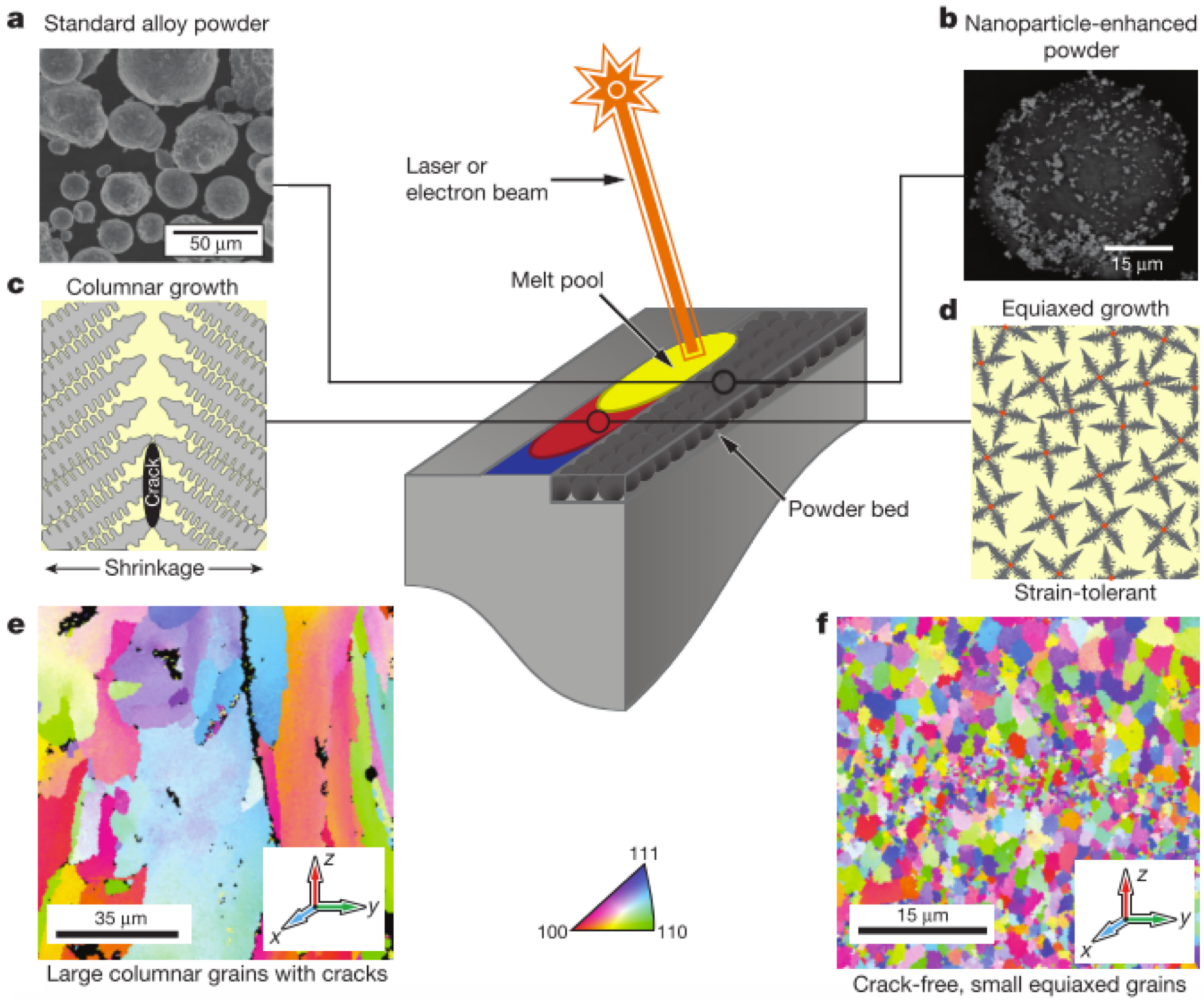
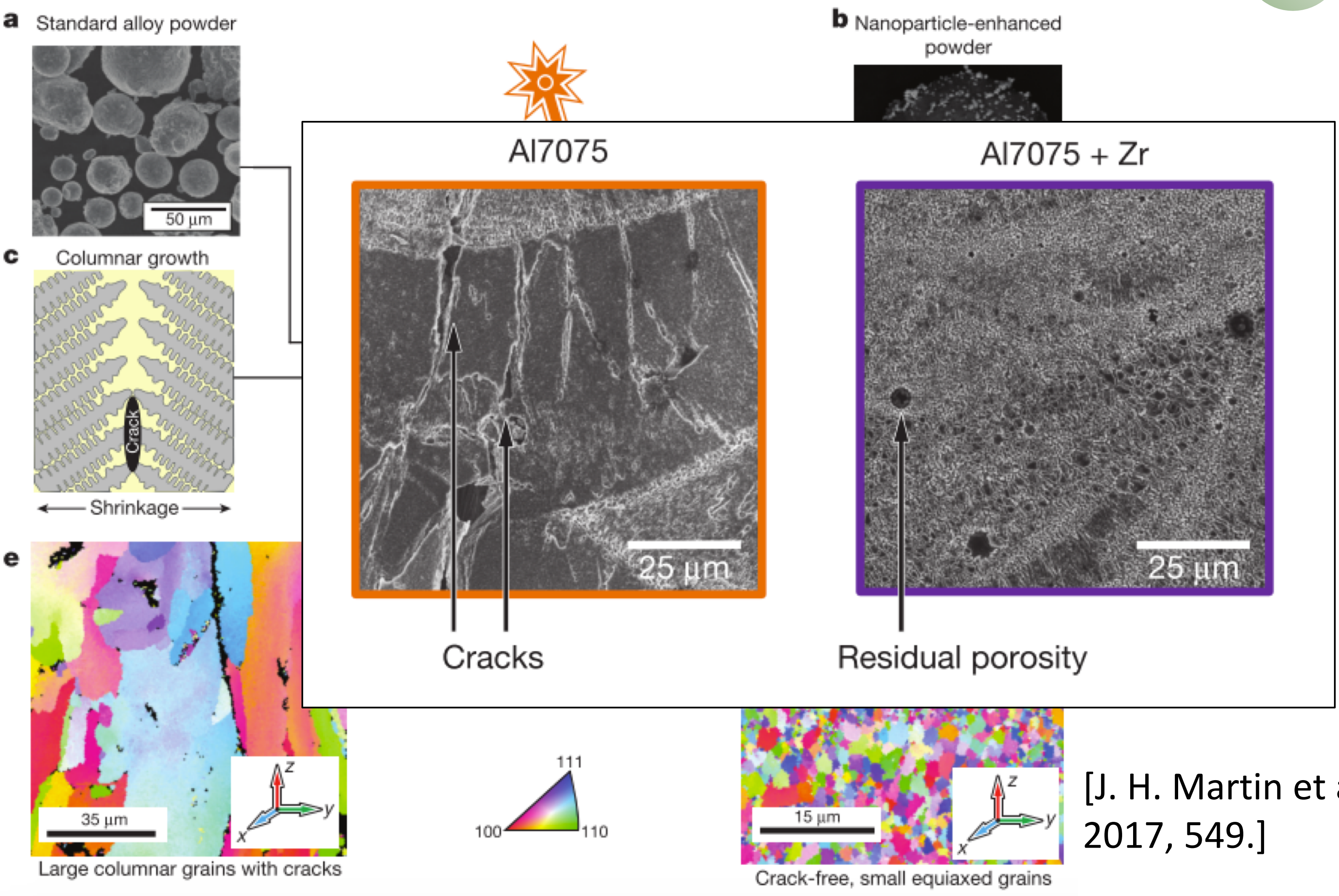
- Introduce an aluminum metal matrix composite (MMC) alloy powder for potential in-situ imaging of rapid solidification
- Recent progress
  - Rapid solidification experiments at Mines (laser welding)
  - Analysis of in-situ experiments with DTEM for Al-Ge alloys
- Future Work
  - Melt spinning
  - Post-mortem analysis of DTEM samples

Grain Size Control via Inoculation



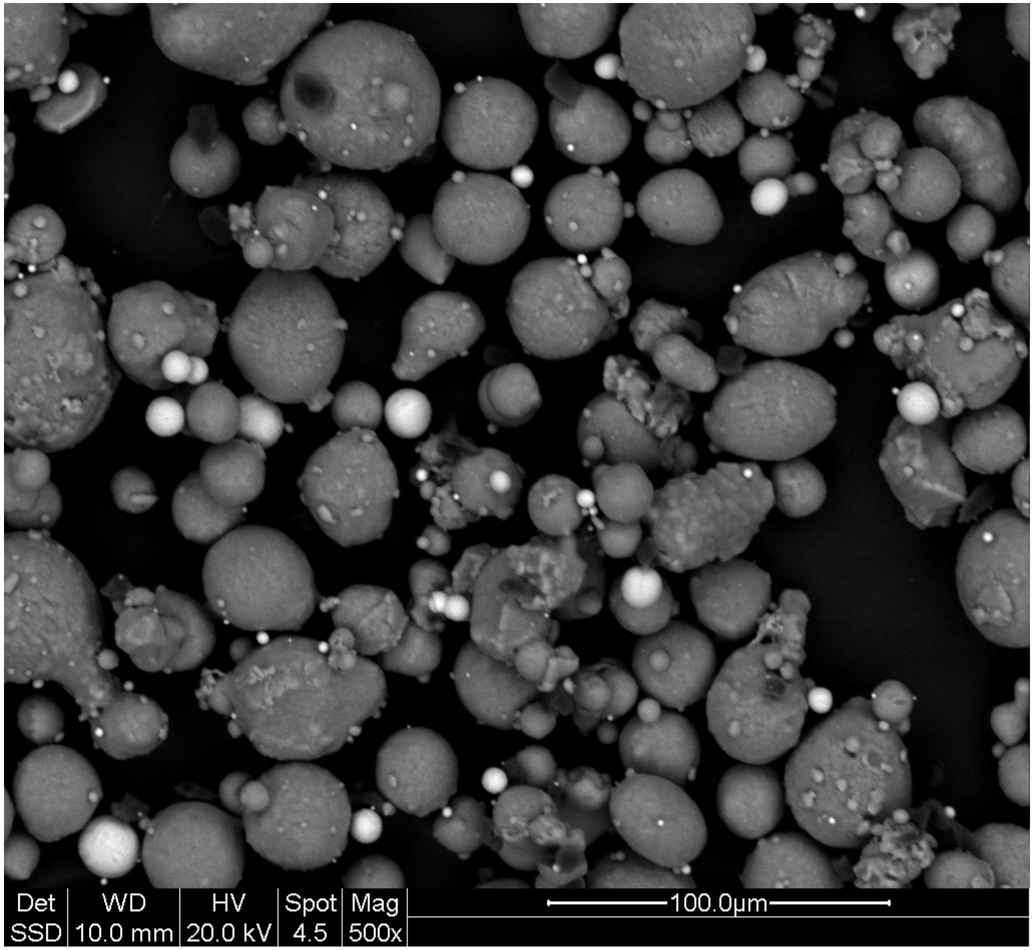
[J. H. Martin et al, Nature 2017, 549.]

Grain Size Control via Inoculation



[J. H. Martin et al, Nature 2017, 549.]

Al 6061 MMC Powder (Elementum 3D)



Particle Size	
Range of Al6061 Particle Diameter (μm)	1-50
Range of Ceramic Particle Diameter (μm)	1-20

In-situ AM Solidification Studies at APS

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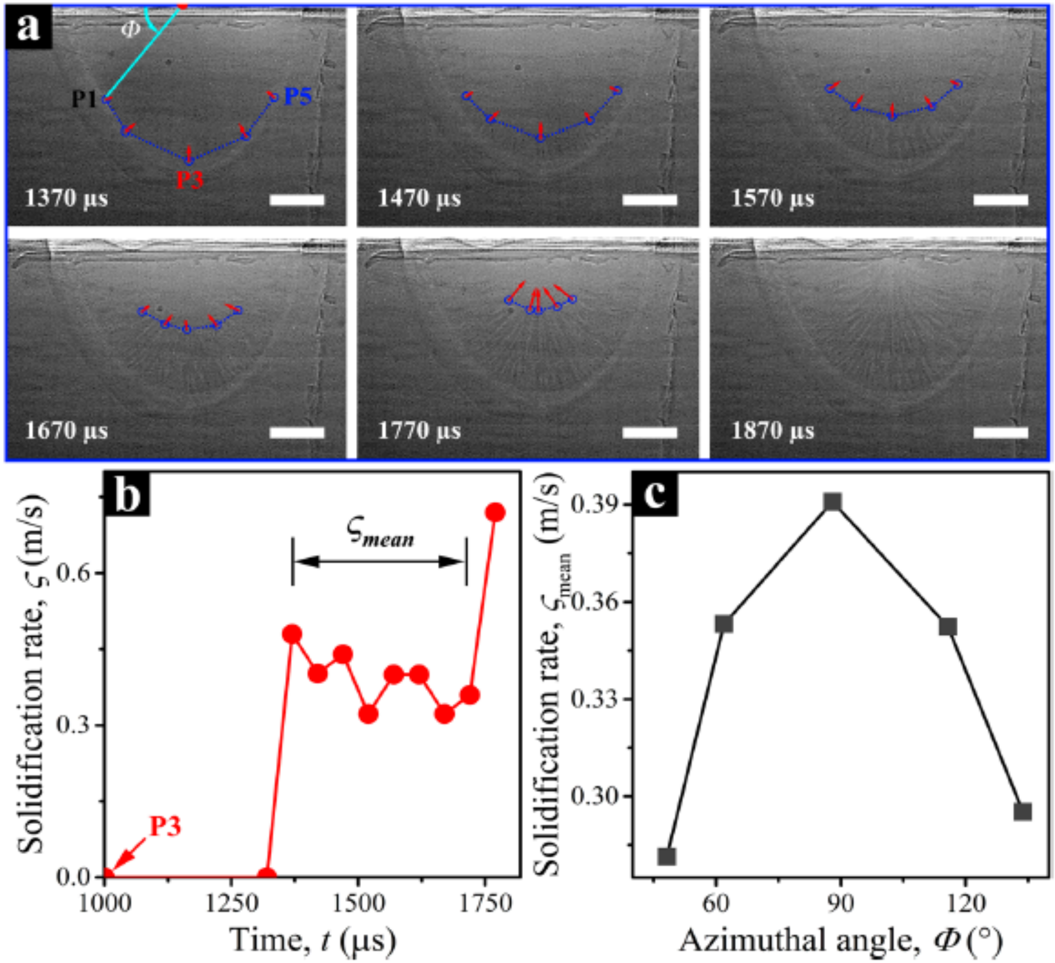

Synchrotron x-ray imaging of a Ti-6Al-4V plate sample in laser melting processes and solidification rate measurements [C. Zhao et al., Scientific Reports 2017, 7:3602].

Recent Progress: Laser Welding at Mines

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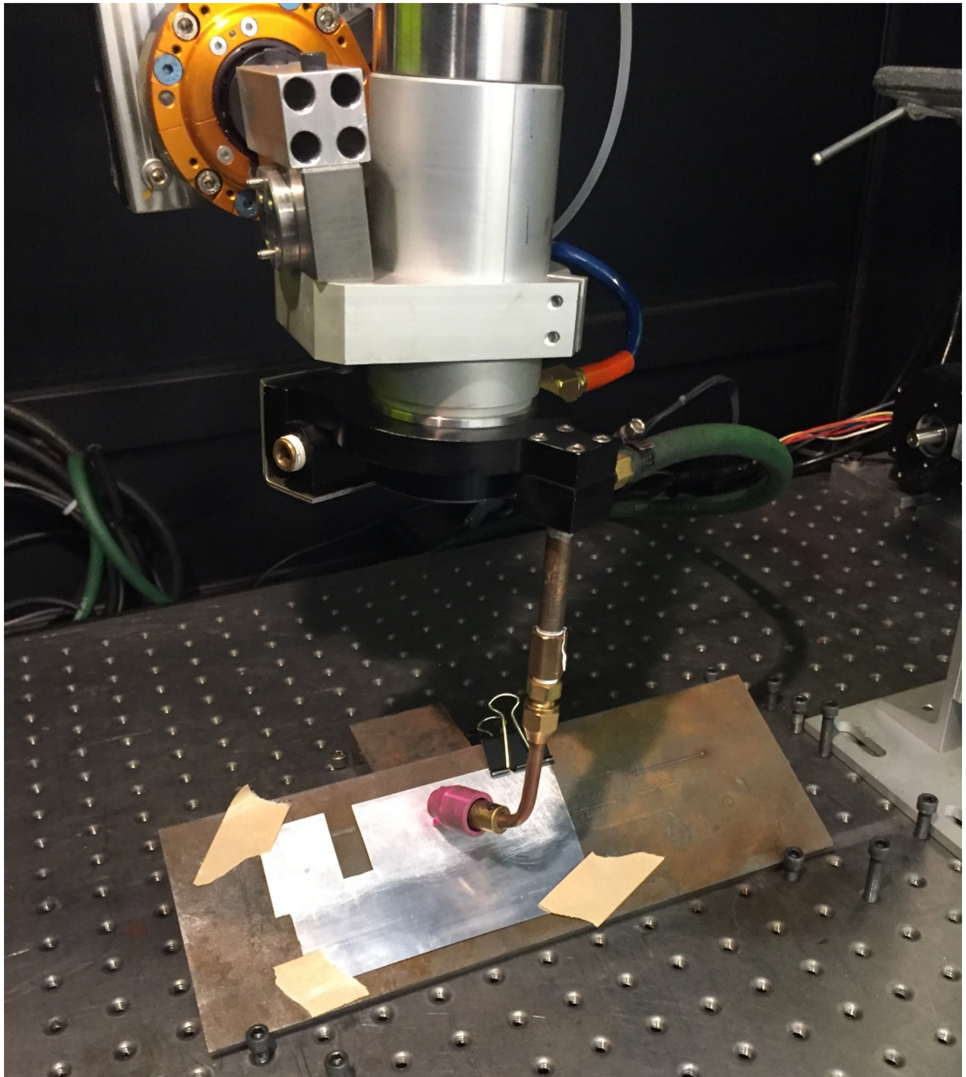
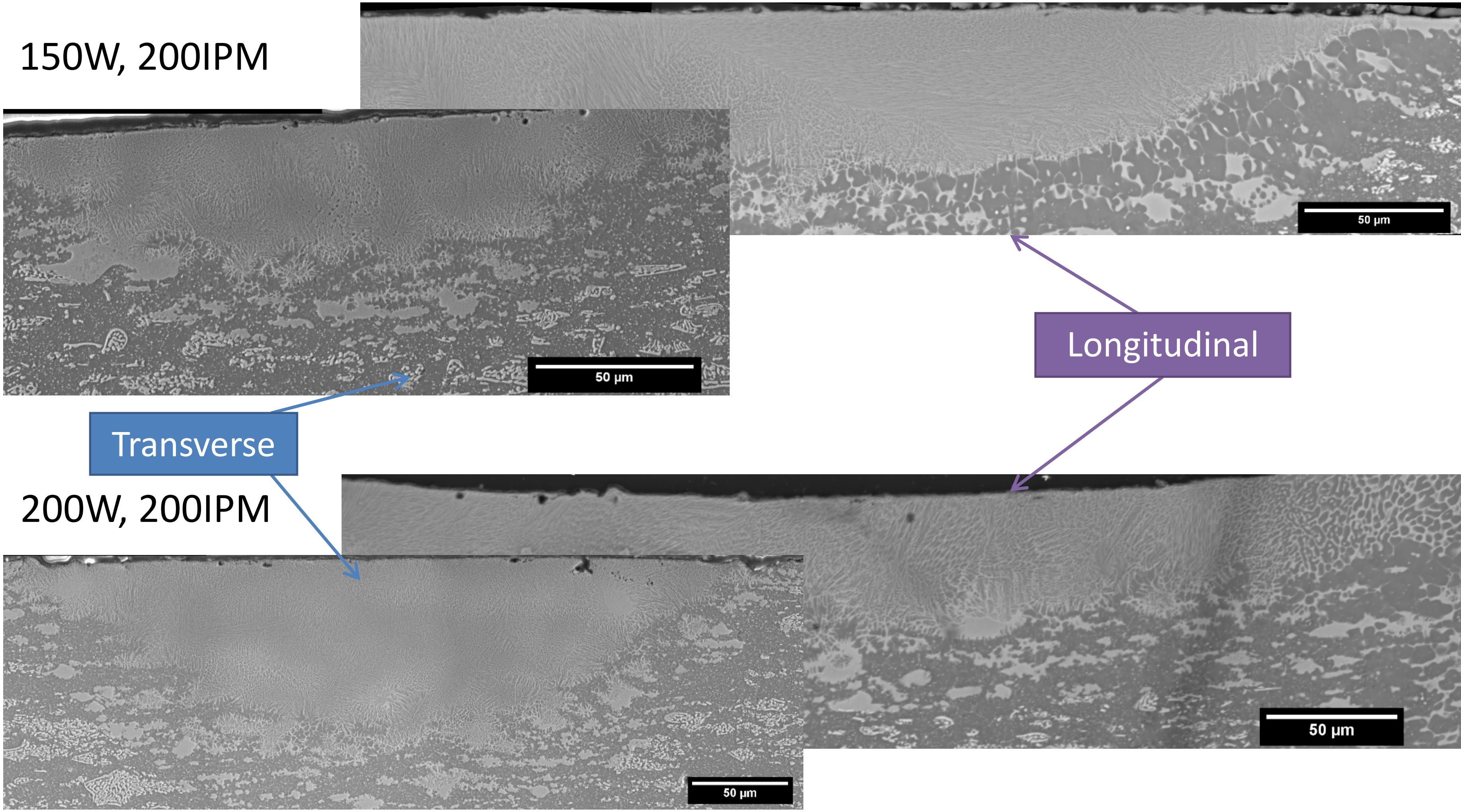


Laser Welding Conditions	
Power (W)	Speed (IPM, mm/s)
150	100, 42
	200, 85
200	100, 42
	200, 85

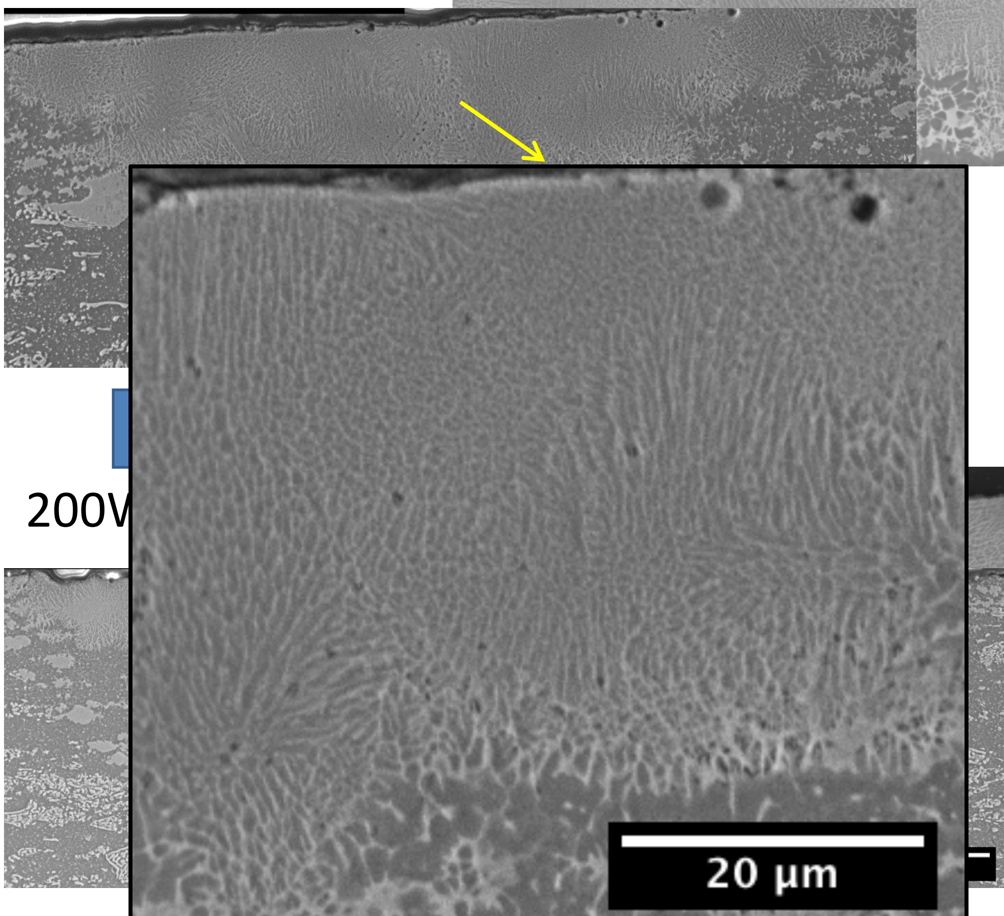
Laser welding of Al-7 at.% Cu foil at CSM.

Microstructural Characterization of Laser Welds

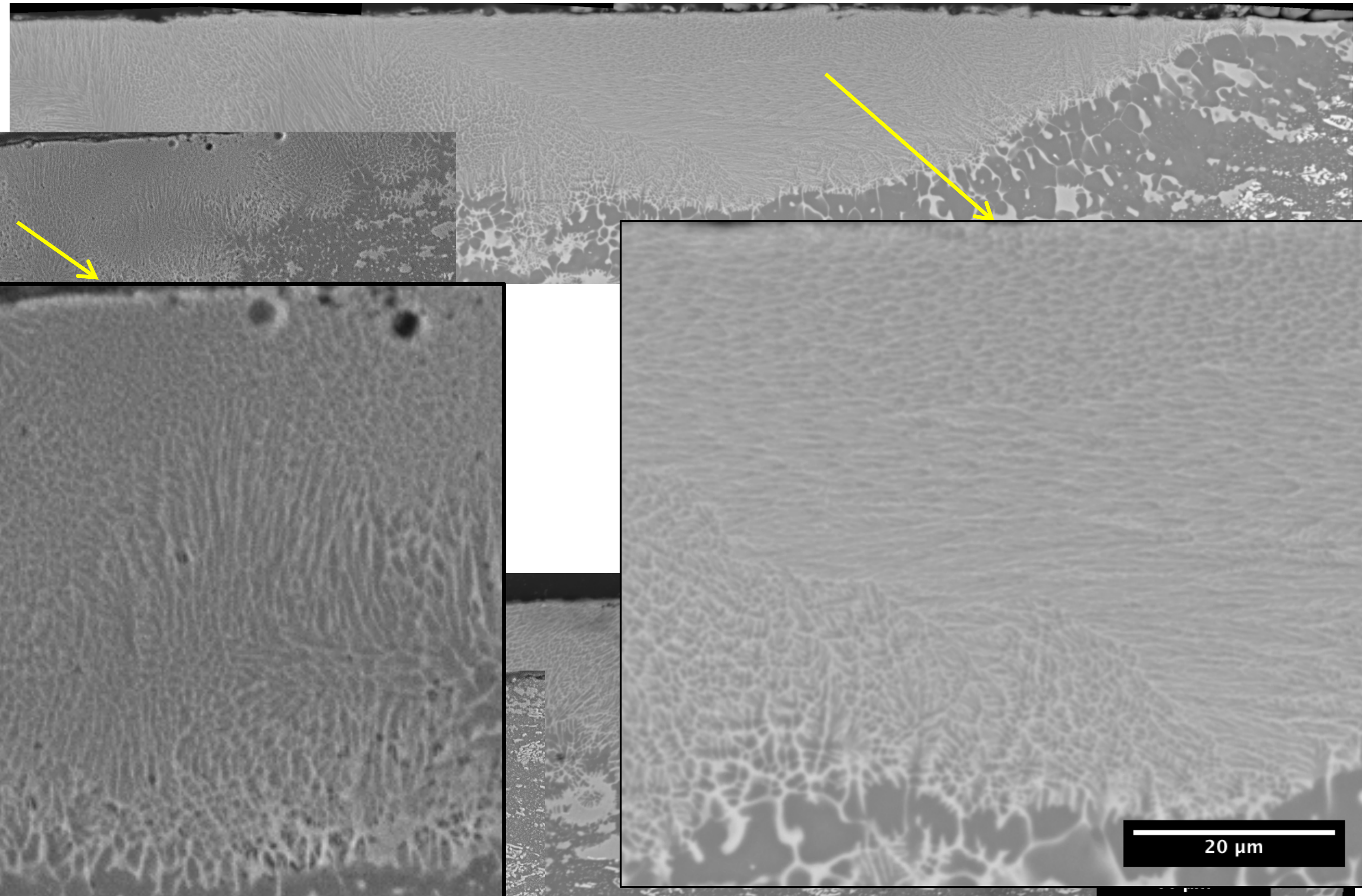


Microstructural Characterization of Laser Welds

150W, 200IPM



200V



Microstructural Characterization of Laser Welds

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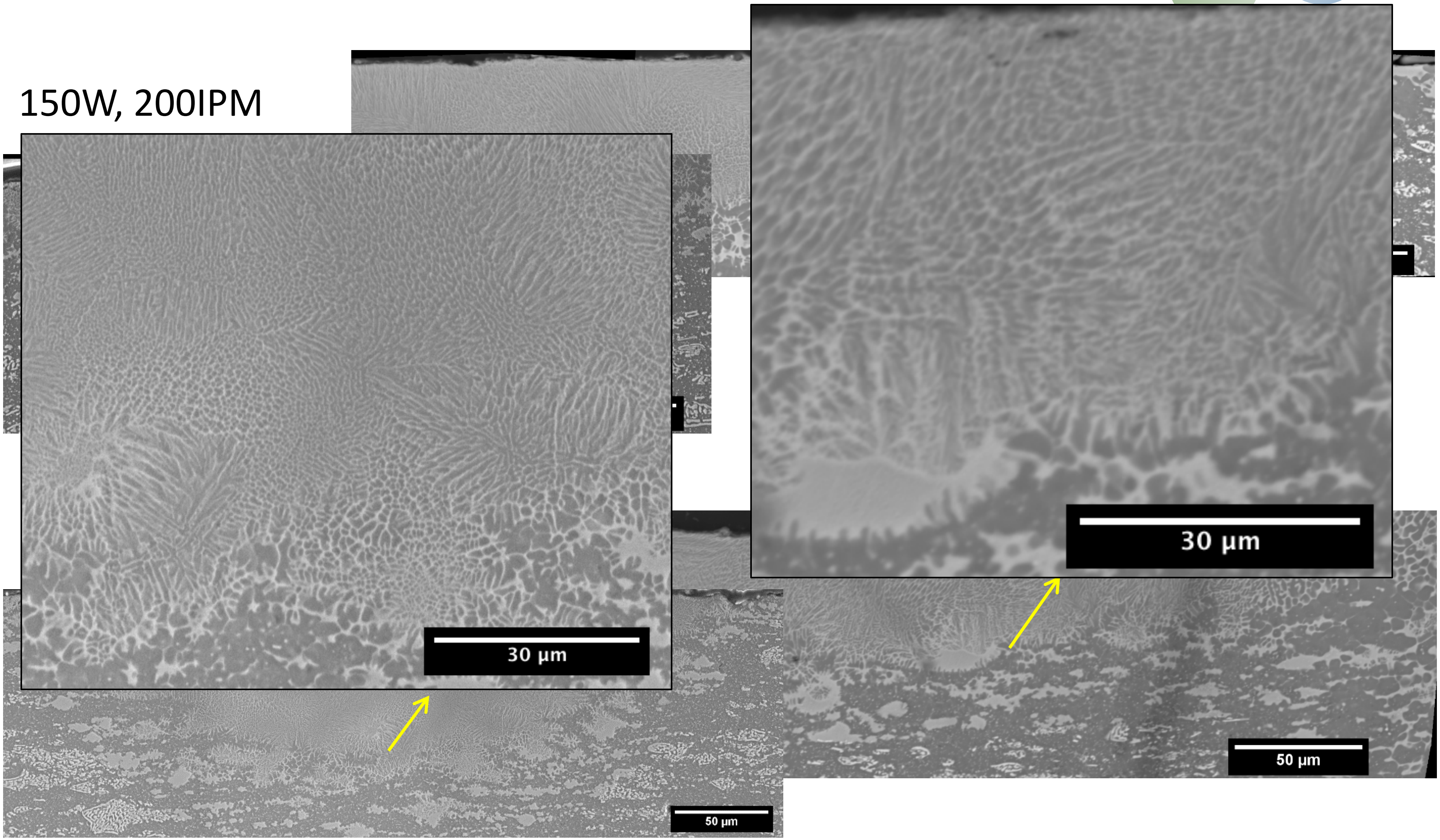
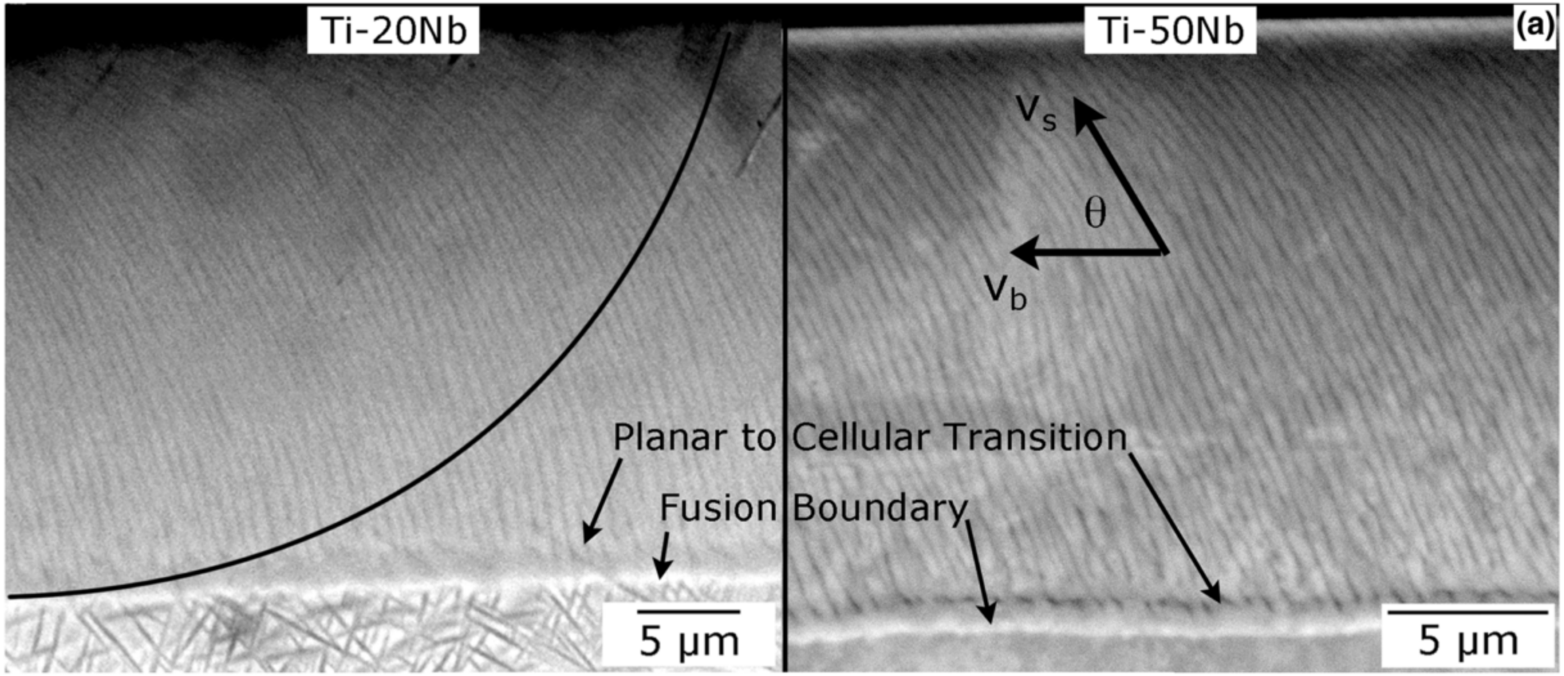
150W, 200IPM



Solidification Velocity Calculations



Example of solidification velocity calculations from a laser welding experiment. [J. D. Roehling et. al, JOM 2018.]

$$V_s = V_b \cos(\theta)$$

V_s – Solidification Velocity
V_b – Laser Scan Speed
Θ – Angle between the two velocity vectors

Solidification Velocity

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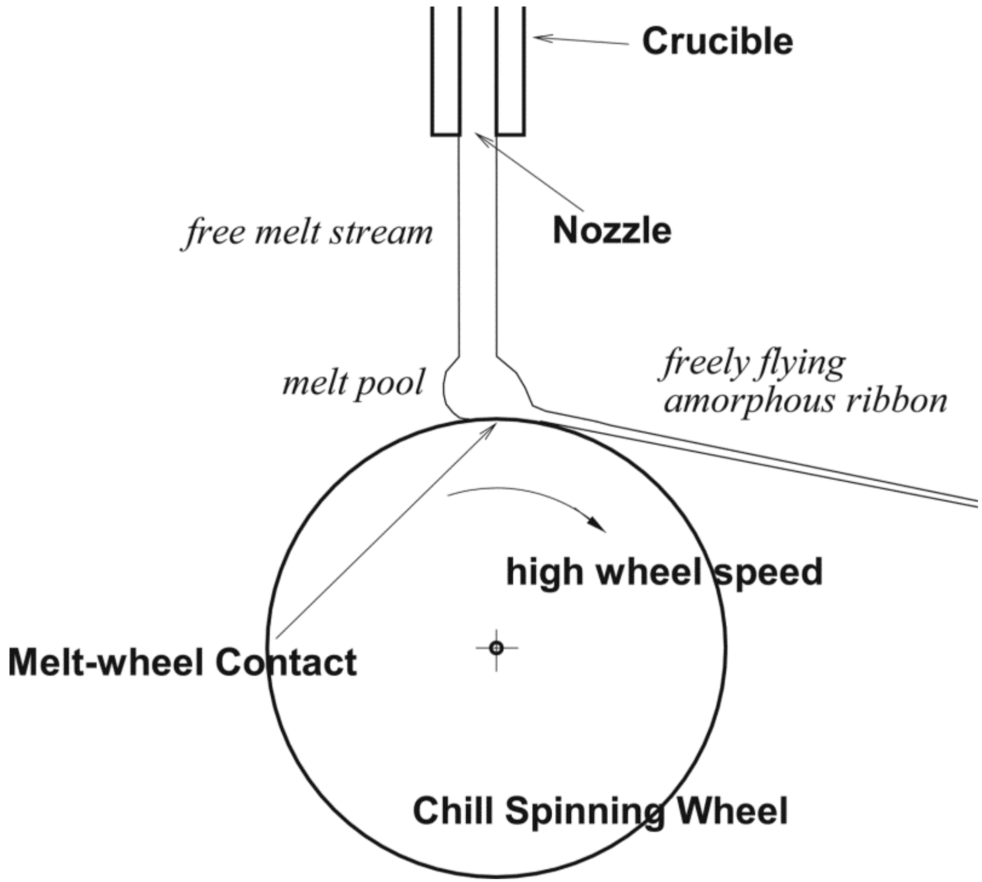
Solidification Velocity of Laser Welds	
Welding condition	Average Solidification Velocity (mm/s)
150 W, 42 mm/s	40
150 W, 85 mm/s	53
200 W, 42 mm/s	39
200 W, 85 mm/s	46

Solidification Velocity from Literature	
Welding condition	Average Solidification Velocity (mm/s)
1200-1750 W, 100-4000 mm/s	200-600
1200-1750 W, 1-1000 mm/s	10-2000
1000-1500 W, 200-4000 mm/s	200->500

[S. Gill, Acta Metallurgica 1992, 40; S. C. Gill, Acta Metallurgica 1993, 41; M. Zimmermann, Acta Metallurgica 1989, 12]

Melt Spinning at Mines



- Preparing crucibles:
  - Coated (BN, Yttria) & shaped quartz tubes
  - Boron nitride, machined
- Achieve another rapid solidification condition to compare with other studies
- Refine studies for off-site experiments

Example of DTEM Experiment: Al-4Cu

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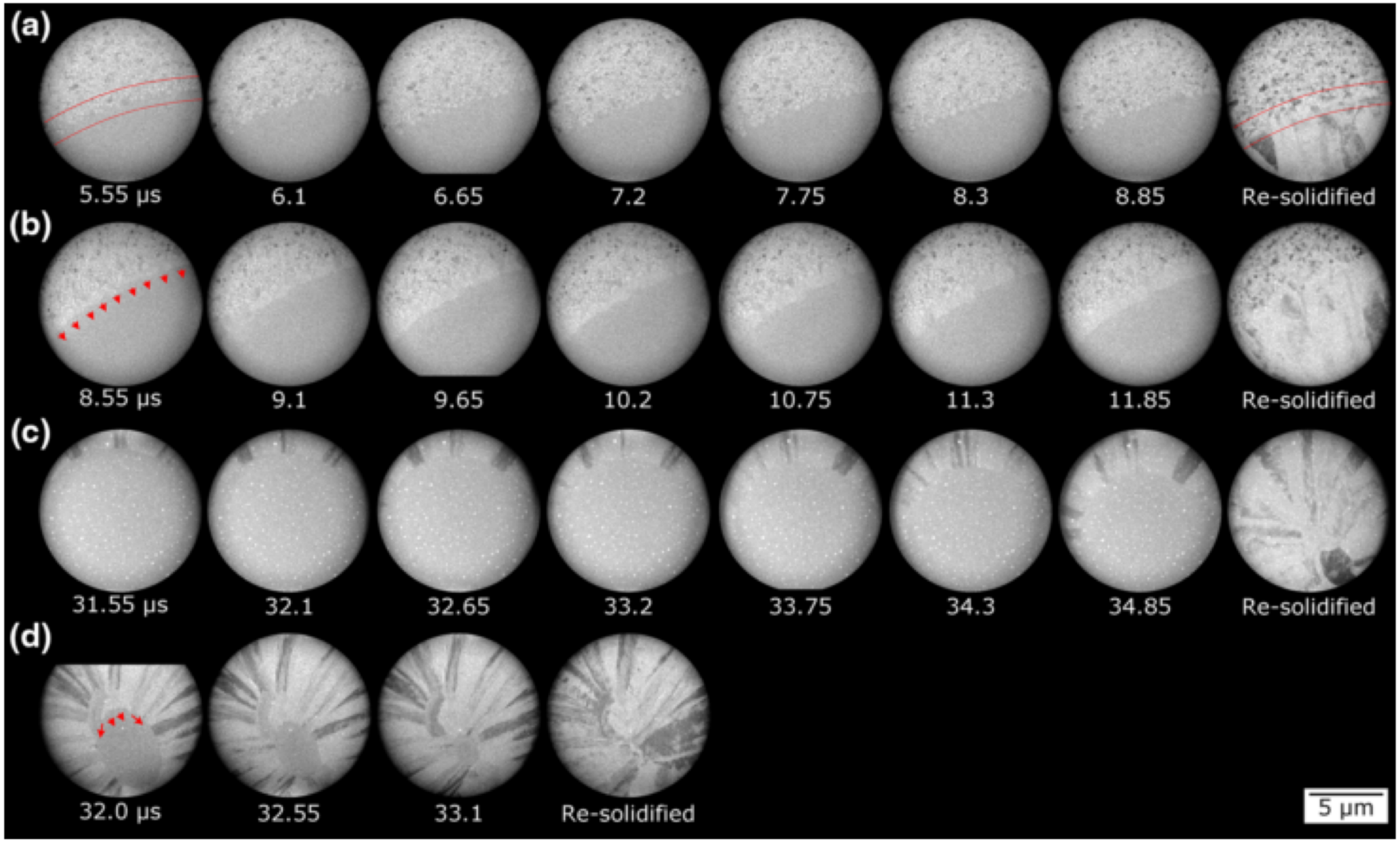
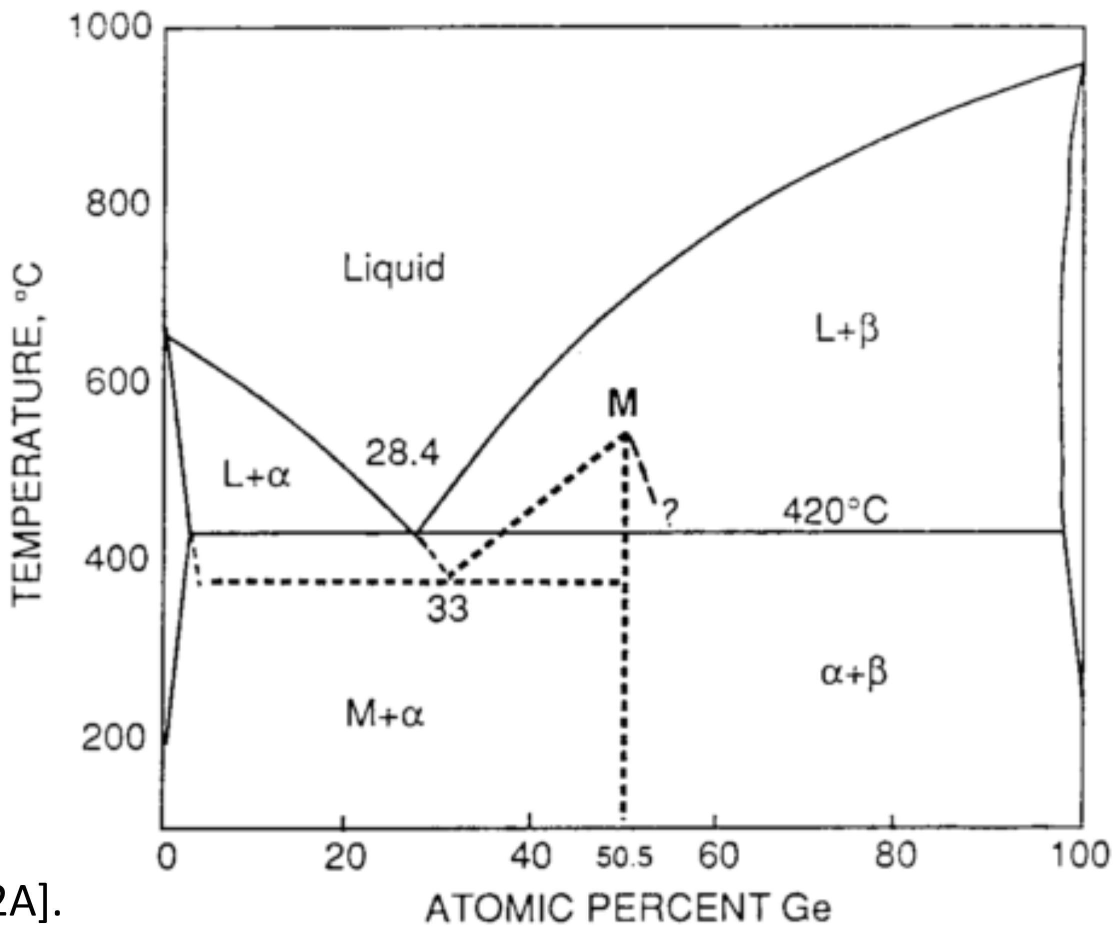


DTEM of an Al–4at.%Cu alloy thin-film during rapid solidification [J.T. McKeown et al., JOM 2016, 68].

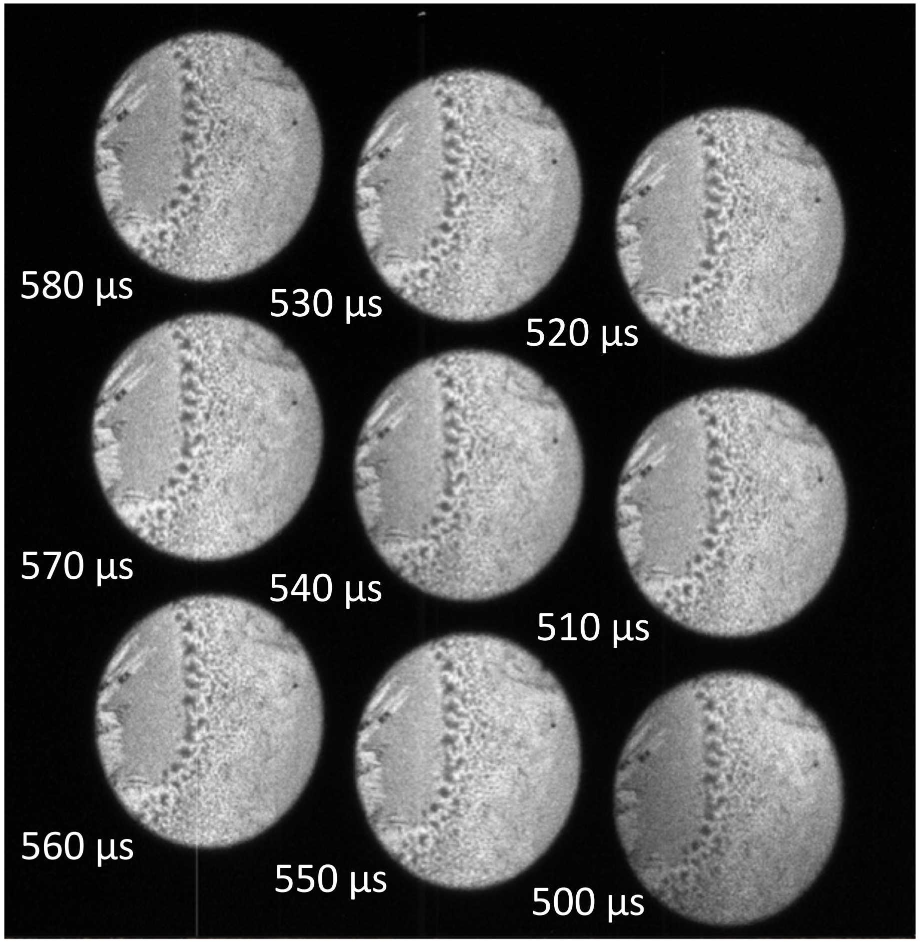
DTEM of Al-Ge: Metastable Phase Diagram of Monoclinic (M) Phase

Table 30.2: Nominal and Measured Compositions of Al-Ge	
Nominal Composition (at. % Ge)	Measured Composition (at. % Ge)
33	46
40	58
43	63
53	70-76

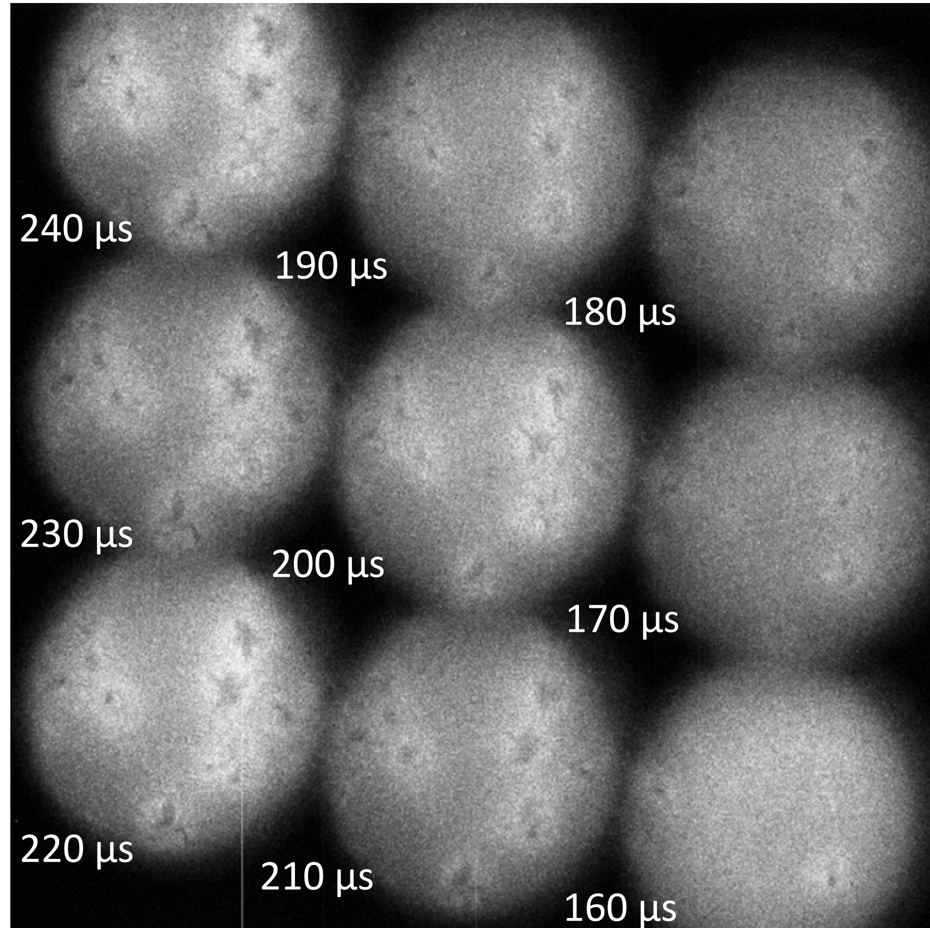


[Laoui et. al, Met. Trans. A 1991, 22A].

Initial Dynamic Data: Below and Above 50.5at.%Ge



Al-33at.%Ge (46at.%Ge)



Al-53at.%Ge (70-76at.%Ge)

Below 50.5at.%Ge: Al-33at.%Ge (46 at.%Ge)

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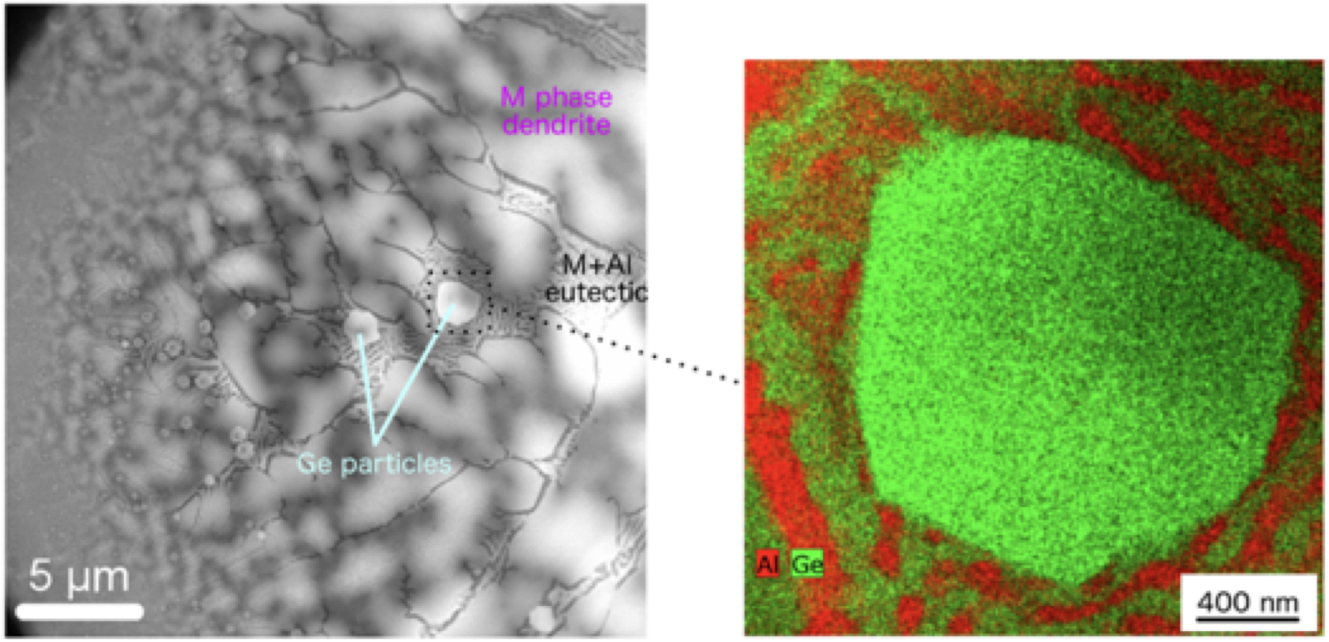
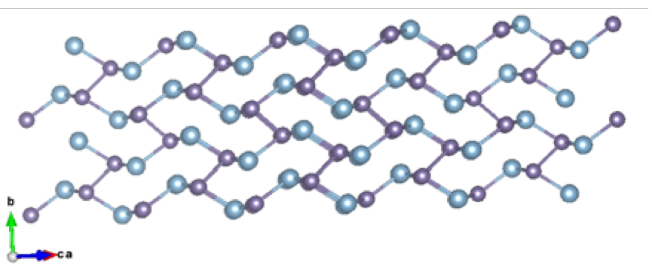
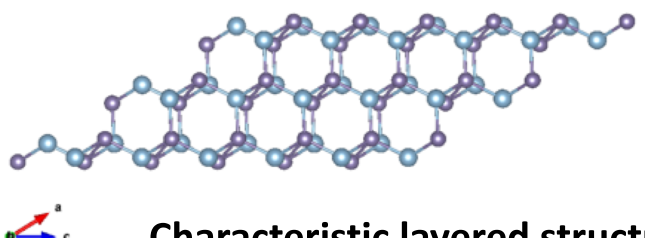




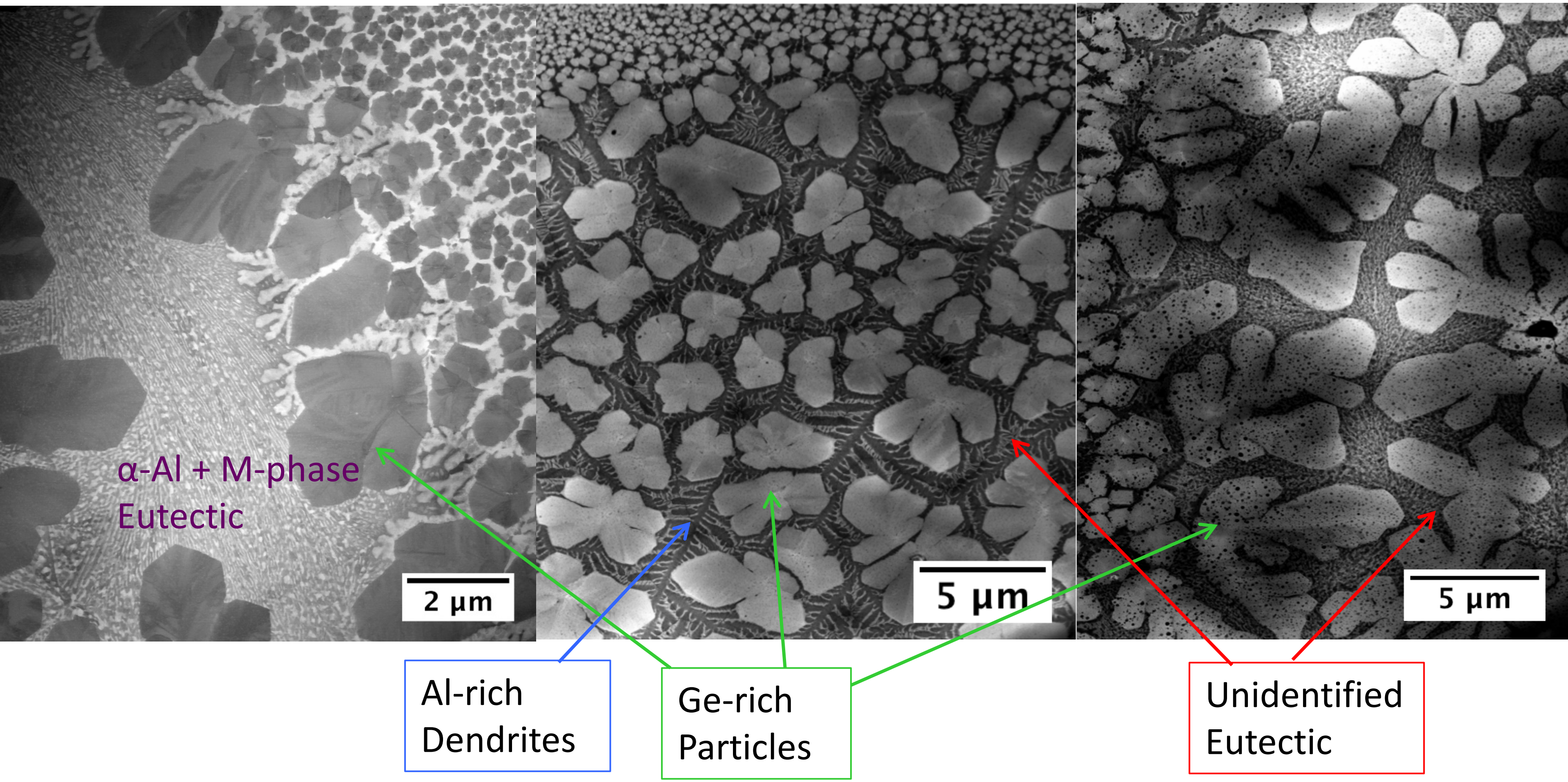
(P12$_1$/c1, a=6.73 A, b=5.82 A, c=8.05 A, =32.16)



Characteristic layered structure and 6-member rings of the monoclinic M-phase
Blue atoms = Al,
purple atoms = Ge

Above 50.5 at.%Ge

Al-40at.%Ge (58at.%Ge) | Al-43at.%Ge (63at.%Ge) | Al-53at.%Ge (70-76at.%Ge)



Future Plans

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- Additional Al-Ge DTEM experiments on compositions above and below 50.5 at.%Ge
  - Below: More complete understanding of M-phase dendrite growth and eutectic (α-phase + M-phase)
  - Above: Capture proposed formation and re-melting of M-phase
  - Use image analysis to determine phase fractions, measure phase compositions, compare with proposed metastable phase diagram (undercooling?)
- Melt spinning of binary Al alloys (Al-Cu, Al-Ag, Al-Cu-Ag)
- In-situ imaging of Al-Cu, Al-Ag, and Al-Cu-Ag alloys and 6061 MMC alloy powder

Progress

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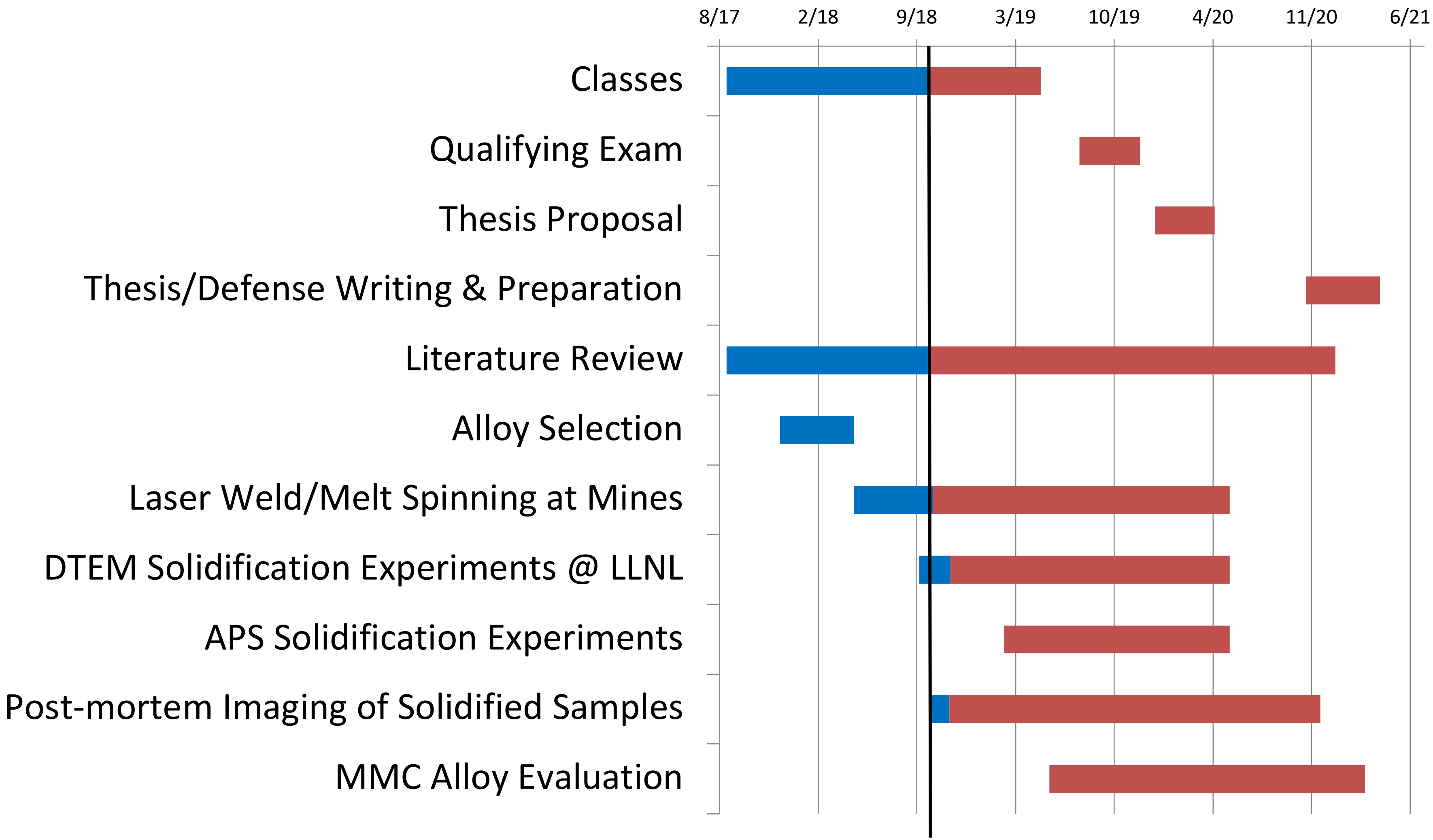

Thank you very much!

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References

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- [30.2] J. Mckeown, A. Kulovits, C. Liu, K. Zweiacker, B. W. Reed, T. LaGrange, J. M. K. Wiezorek, G. H. Campbell, In situ transmission electron microscopy of crystal growth-mode transitions during rapid solidification of a hypoeutectic Al-Cu alloy, Acta Metallurgica et Materialia, 65 (2014) 56-68.
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- [30.6] D. Tourret, J. C. E. Mertens, E. Lieberman, S. D. Imhoff, J. W. Gibbs, K. Henderson, K. Fezzaa, A. L. Deriy, T. Sun, R. A. Lebensohn, B. M. Patterson, A. J. Clarke, 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, 48A (2017) 5529-5546.
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Project 30 – Microstructural Evolution of Metallic Alloys During Rapid Solidification

Student: *Chloe Johnson*

Faculty: *Amy Clarke*

Industrial Partners: *LANL, Boeing*

Project Duration: *Aug. 2017 – May 2021*

Achievement

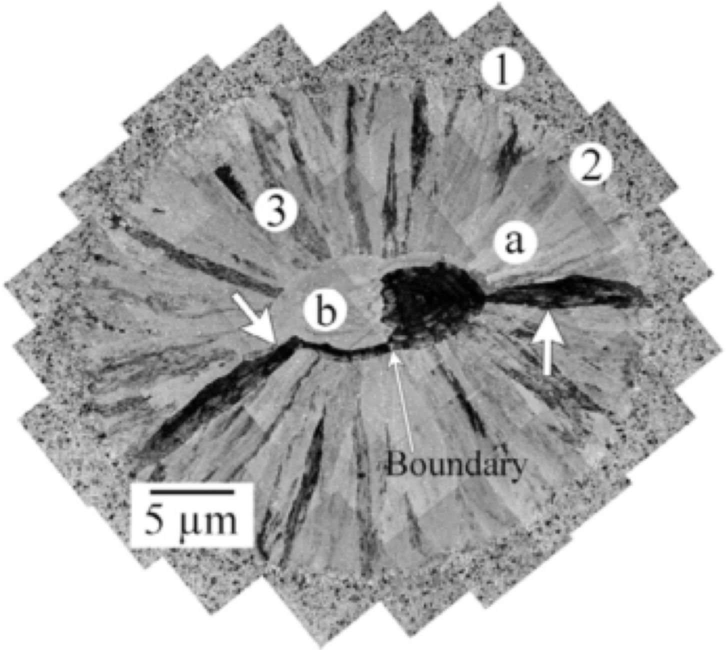
- Understand the relationship between rapid solidification conditions and development of metastable phases, grain sizes and morphologies, and local chemistries.

Significance and Impact

- Understand and control microstructural evolution during rapid solidification to inform industrial processes (e.g. additive manufacturing, welding, etc.).

Research Details

- Evaluate the effect of solidification conditions on the resultant microstructures and phases in chosen aluminum alloys (binary, ternary, MMC) using in/ex-situ techniques.



Post-mortem TEM of an Al-7at.%Cu alloy film after DTEM solidification [J.T. McKeown et al., Acta Materialia 2014, 65].

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Student: *Chloe Johnson*

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Project Duration: *Aug. 2017 – May 2021*

Program Goal

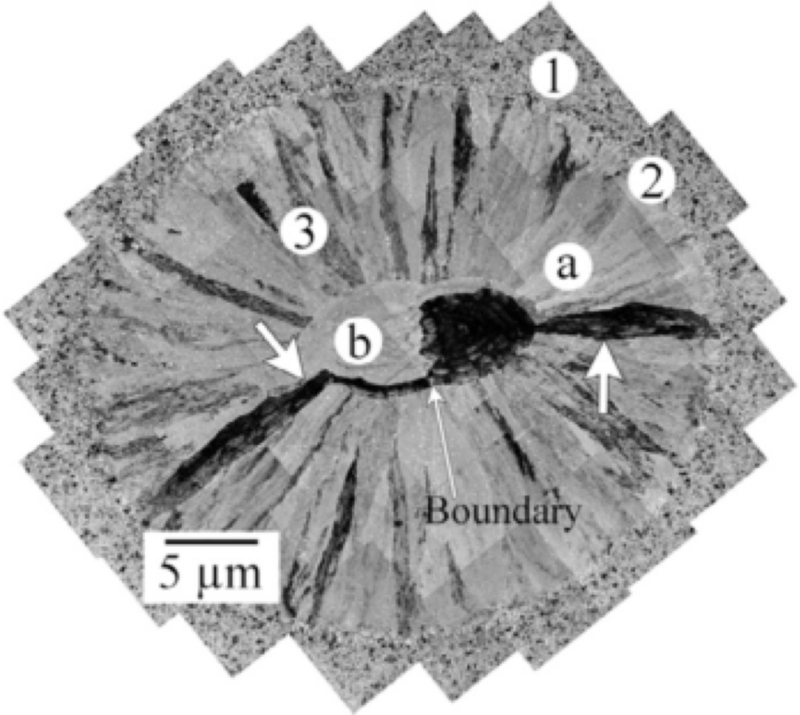
- Understand the relationship between rapid solidification conditions and development of metastable phases, grain sizes and morphologies, and local chemistries.

Approach

- Evaluate the effect of solidification conditions on the resultant microstructures and phases in a variety of aluminum alloys (binary, ternary, MMC) using in/ex-situ techniques.

Benefits

- Understand and control microstructural evolution during rapid solidification to inform industrial processes (e.g. additive manufacturing, welding, etc.).



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Achievement

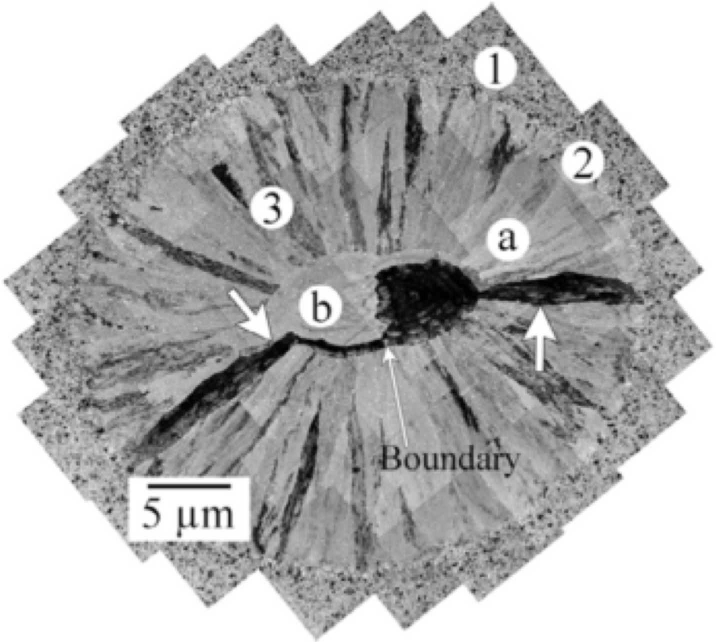
- Understand the relationship between rapid solidification conditions and development of metastable phases, grain sizes and morphologies, and local chemistries.

Significance and Impact

- Understand and control microstructural evolution during rapid solidification to inform industrial processes (e.g. additive manufacturing, welding, etc.).

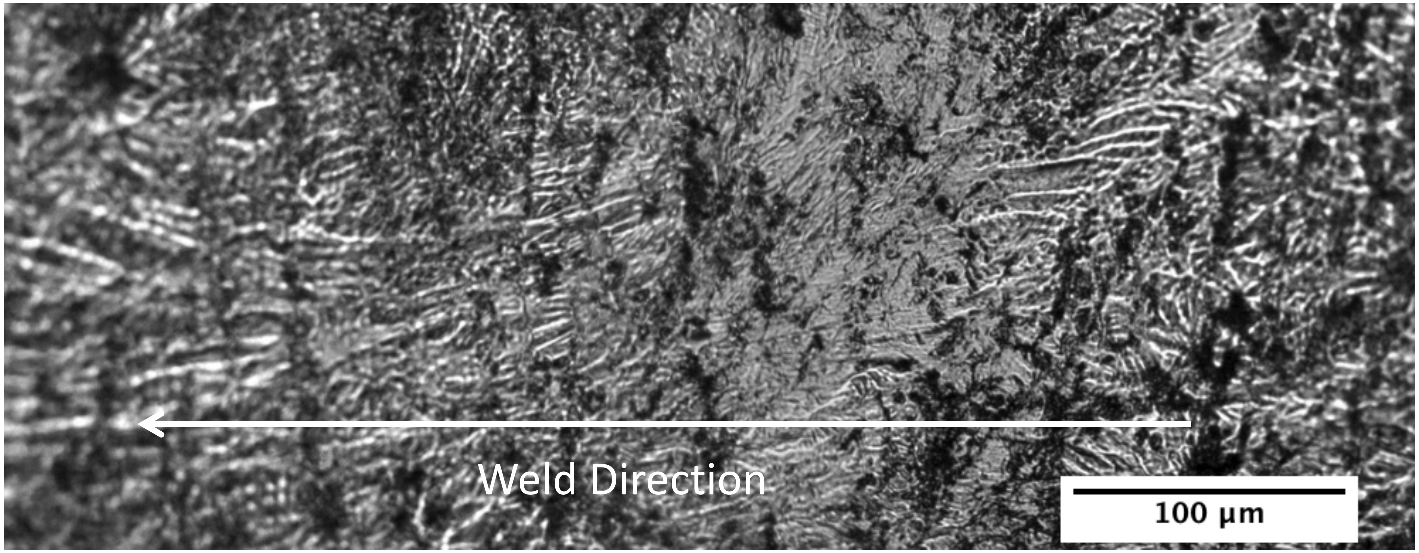
Research Details

- Perform rapid solidification experiments on wide variety of aluminum alloys (binary, ternary, MMC) using in/ex-situ imaging techniques.



Post-mortem TEM of an Al-7at.%Cu alloy film after DTEM solidification [J.T. McKeown et al., Acta Materialia 2014, 65].

Microstructural Characterization of Welds: Comparison to Literature



Light optical photograph of a laser weld done at CSM from the top of the weld.

Grain structure of laser weld from (a) a 3D grain growth model and (b) experimental EBSD data [H.L. Wei et al, Acta Mater. 2017, 122].