Project 36E-L: In-Situ Characterization of Microstructural Evolution During Simulated Additive Manufacturing in Model Alloys

Semi-annual Fall Meeting

October 2021

- Student: Brian Rodgers (Mines)
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- Industrial Mentors: Joe McKeown (LLNL), Edwin Schwalbach (AFRL), Neil Carlson (LANL)
- Other Participants: Jonah Klemm-Toole (Mines), Joe McKeown (LLNL), Alain Karma (Northeastern Univ.)

Project 36E-L: In-Situ Characterization of Microstructural Evolution During Simulated Additive Manufacturing in Model Alloys

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- Student: Brian Rodgers (Mines)
- Advisor(s): Amy Clarke (Mines)

Project Duration
PhD: September 2019 to March 2023

- Problem: Aerospace components are difficult to produce conventionally, but the effects of additive manufacturing (AM) on microstructural evolution are not understood enough to replace conventional manufacturing.
- Objective: Develop an understanding of solidification behavior in model alloys under AM conditions by *in-situ* characterization.
- Benefit: Microstructural control for additive manufacturing of aerospace components.

Recent Progress
- Determined G-V profile of Ni melt pools using SYSWELD simulations
- Found DOT in Al-Ag samples at higher solidification velocities
- Showed DOT occurs at higher velocities in Al-Ge
- Analyzed microstructures of Al-Ag and Al-Ge DTEM samples`

Metrics		
Description	**% Complete**	**Status**
1. Literature review	60%	●
2. Analysis of APS beam line data	90%	●
3. Analysis of Dynamic Transmission Electron Microscopy (DTEM) of rapid solidification	50%	●
4. Simulation of experimental conditions	50%	●
5. Complementary *ex-situ* characterization	55%	●

Model alloys of choice

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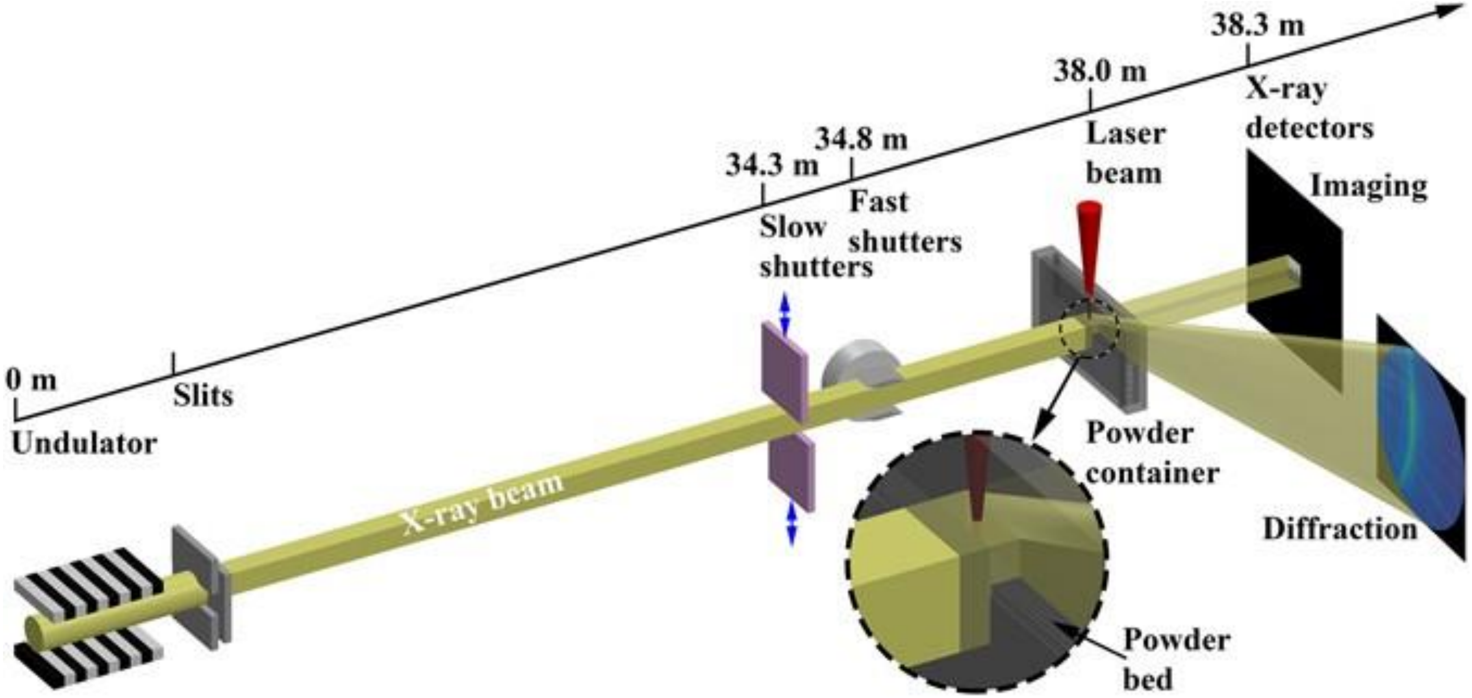
- Ni-Al-Mo single crystals
  - R2: Ni-6.6Al-1.9Mo (at%)
  - R4: Ni-2.8Al-22.2Mo (at%)
  - Directly observe CET
- Al-Ag binary system
  - Al-10Ag and Al-18Ag (at%) APS samples
  - Assorted DTEM compositions
  - Strong partitioning
- Al-Ge binary system
  - Assorted DTEM compositions
  - Differences in bonding type

Beamline 32 AM simulator overview

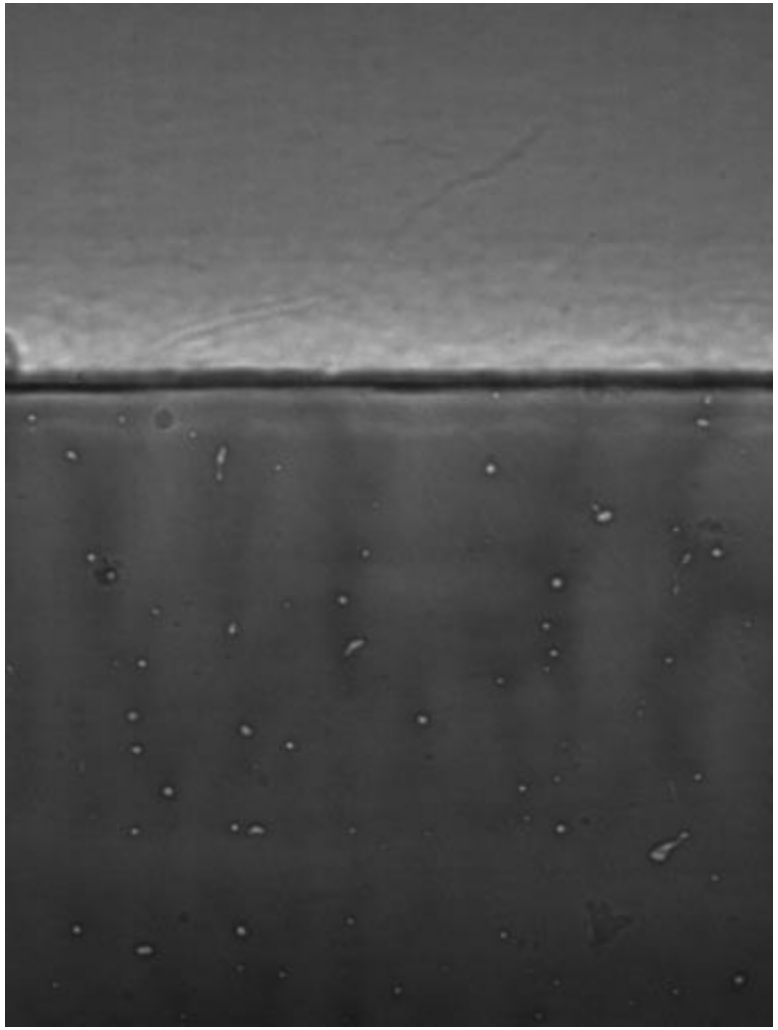
- Laser rasters on surface
- Synchrotron source transmits x-rays through samples simultaneously
- Scintillator converts x-rays to visible light captured by high speed camera
- Able to measure solid-liquid interfacial velocity



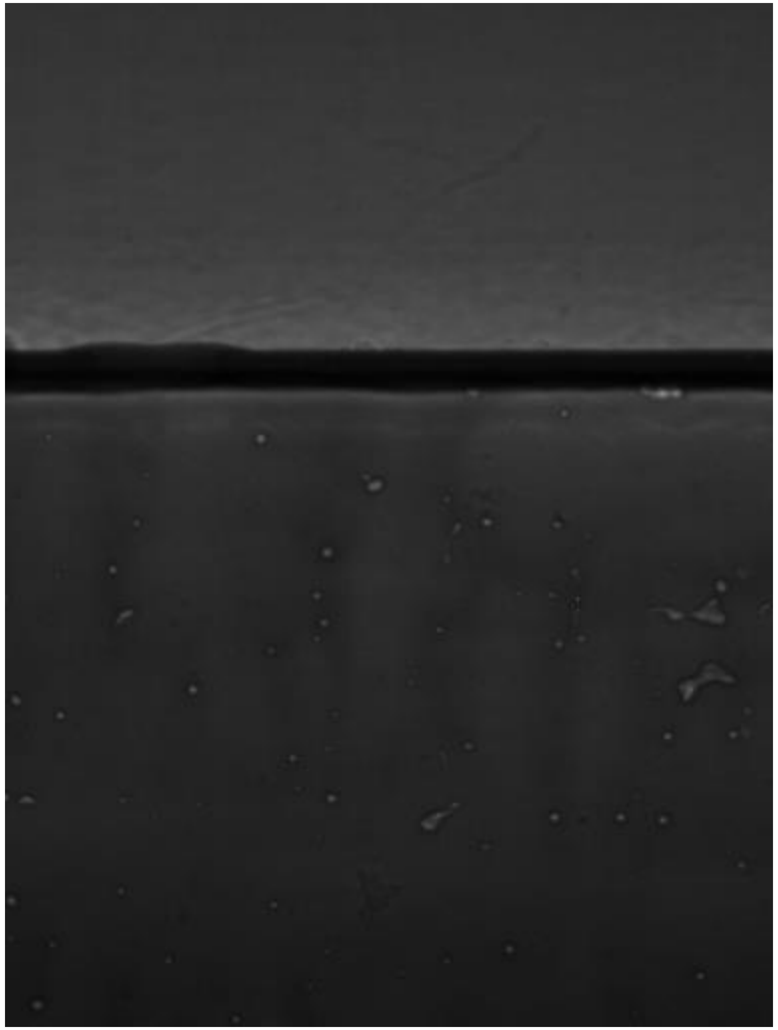
"Real-time monitoring of laser powder bed fusion process using high-speed X-ray imaging and diffraction", C. Zhao, K. Fezzaa, R.W. Cunningham, H. Wen, F.D. Carlo, L. Chen, A.D. Roller, T. Sun, *Sci. Reports.,* 2017

Ni spot melt radiography

Conduction



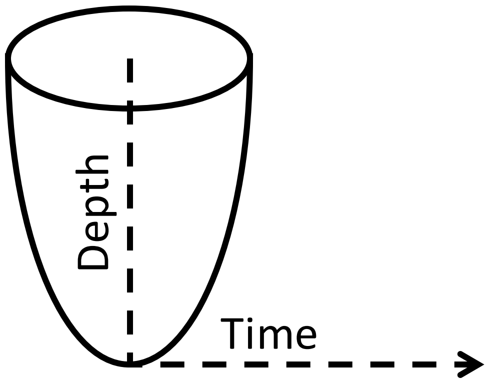
Keyhole

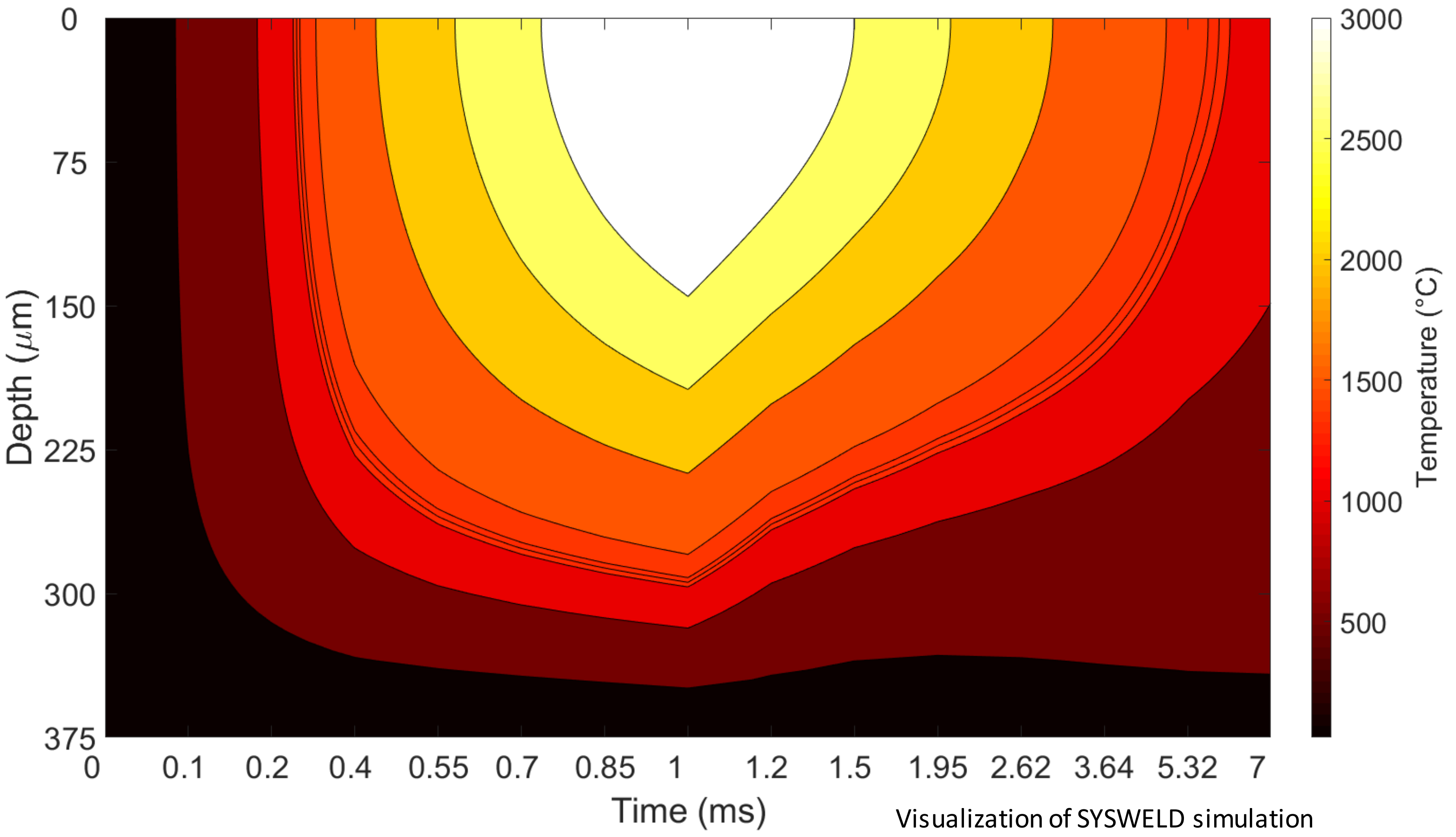


Radiography provides velocity only, not thermal gradient

SYSWELD simulations

- SYSWELD assumptions include:
  - Same properties between solid and liquid
  - Conduction only
  - No vaporization
  - IN625 properties
- Goal is to find G and V
- Simulating spot melts
  - 1 ms duration
  - Varying power





Visualization of SYSWELD simulation for 197 W spot melts

Interface velocity behavior differs with power

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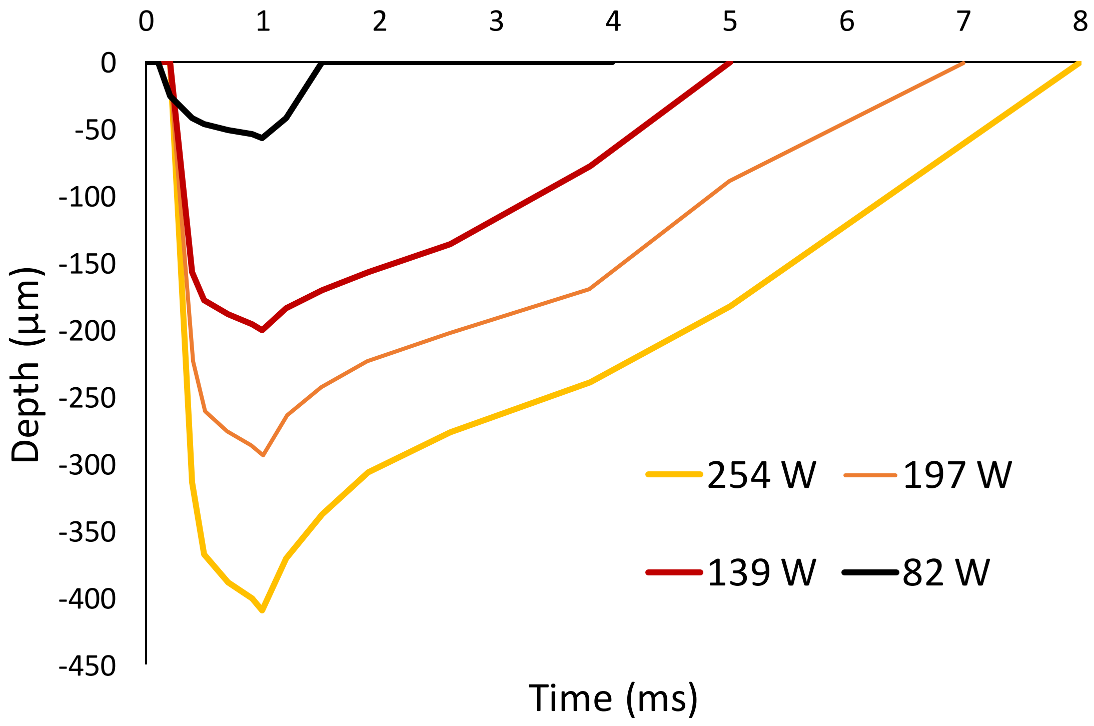
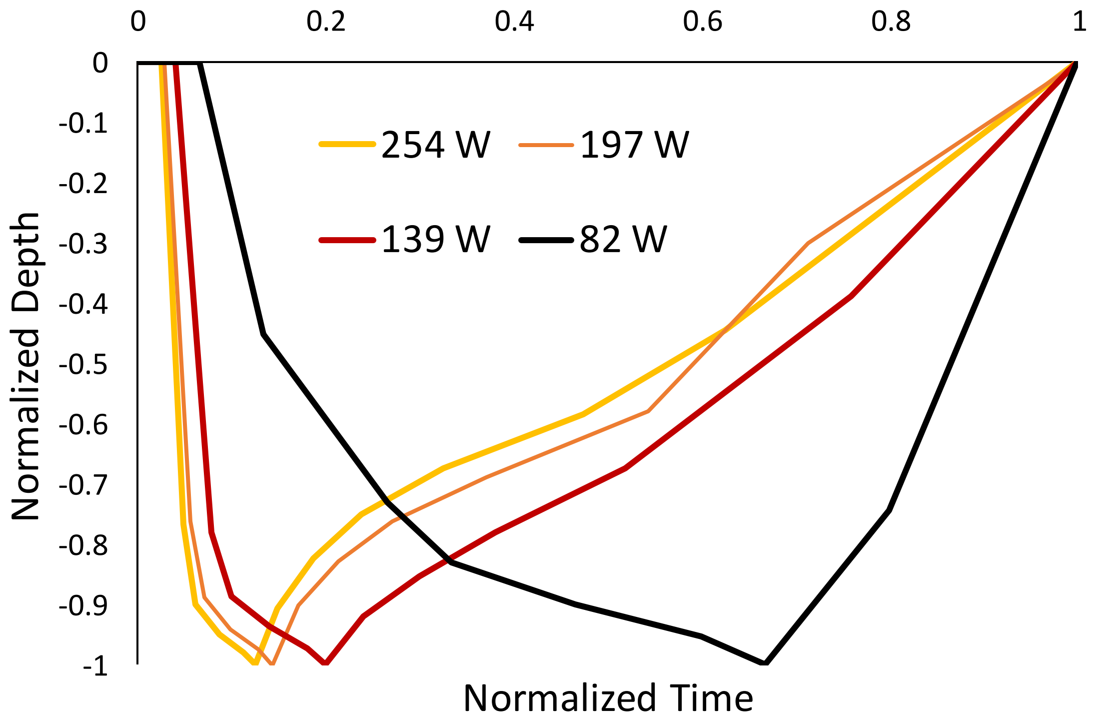
- Higher power influences the shape of the velocity profile, not just the magnitude
- Velocity does not change monotonically
- Normalizing depth and time by their respective maximum helps with visualization

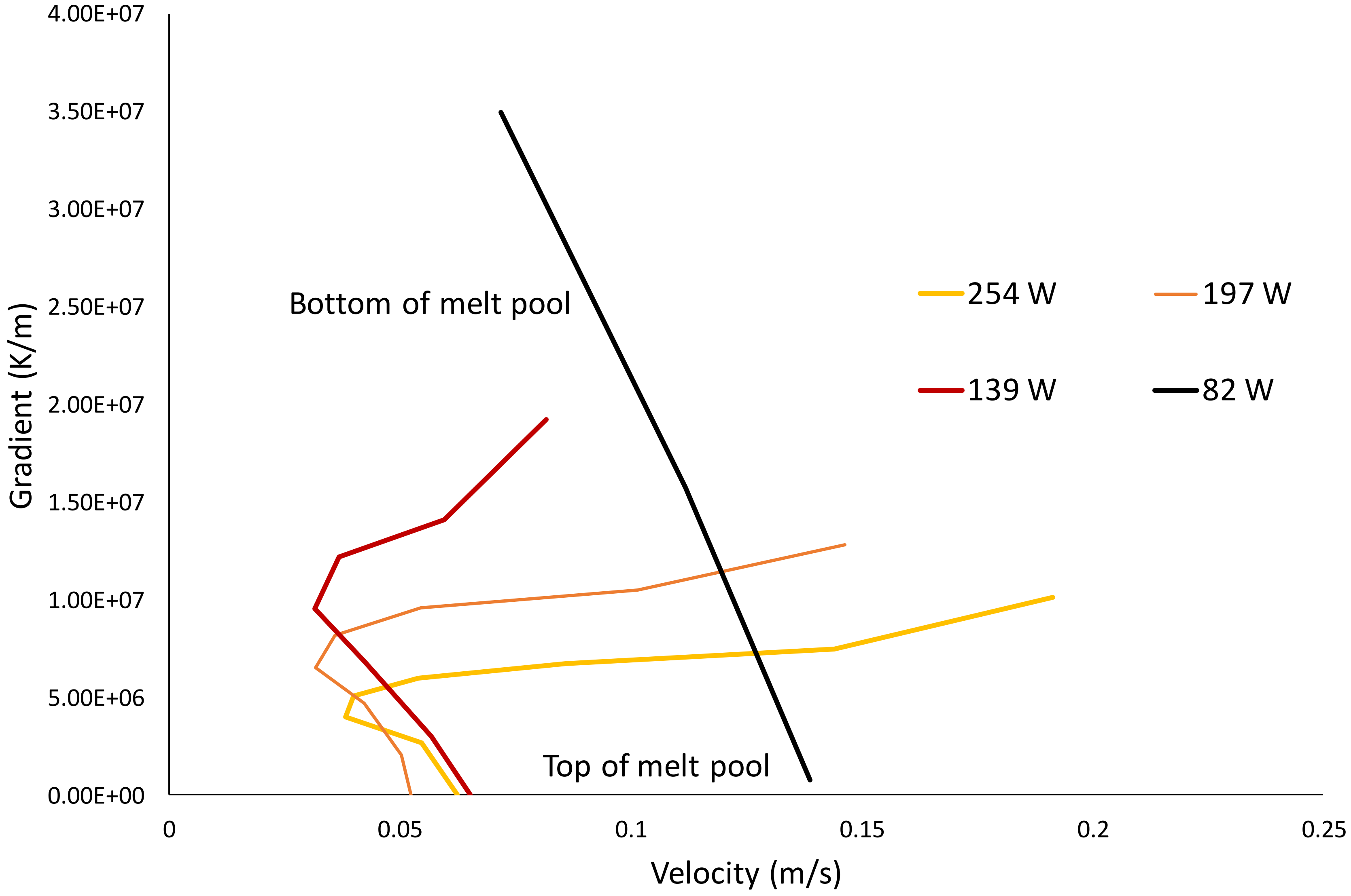


Visualization of SYSWELD simulation results



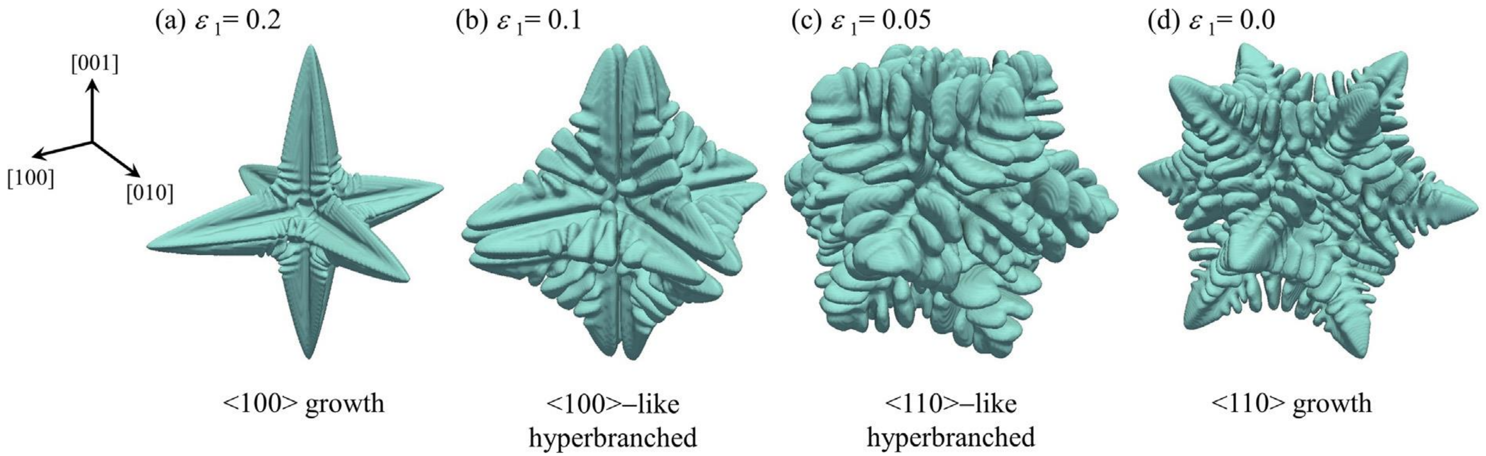
Visualization of SYSWELD simulation results

Summary of G-V results

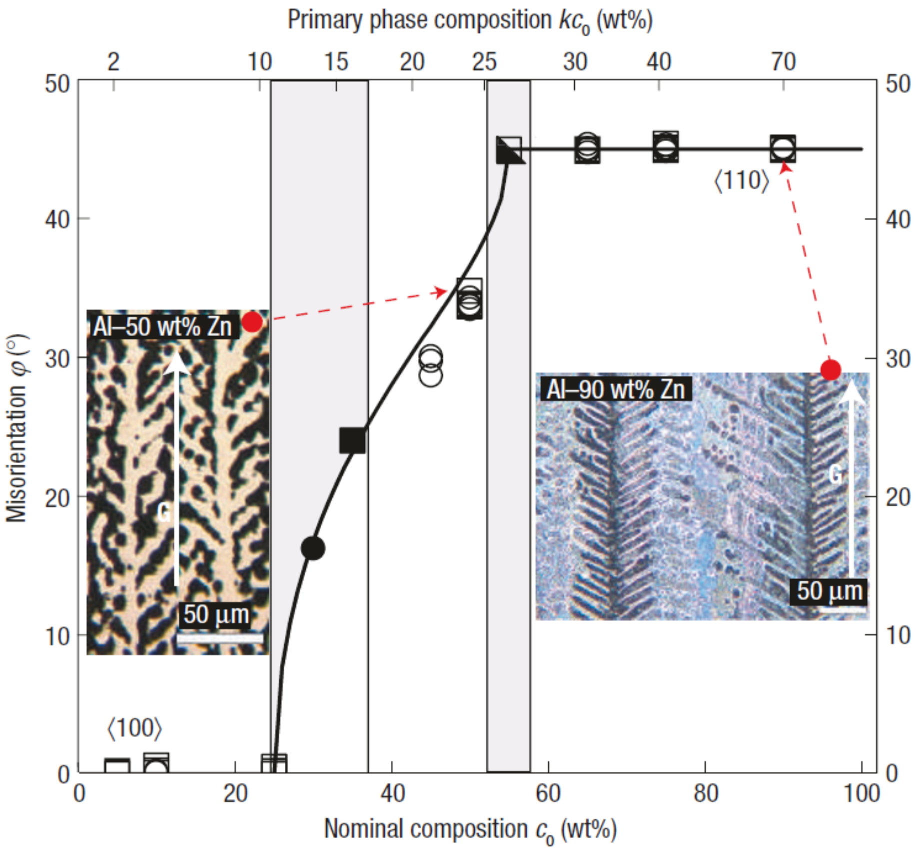


Dendrite Orientation Transition (DOT) refresher

- Dendrites usually grow along a fixed direction
- Some systems experience a shift in growth direction with solute additions
- Attributable to changes in interfacial energy anisotropy



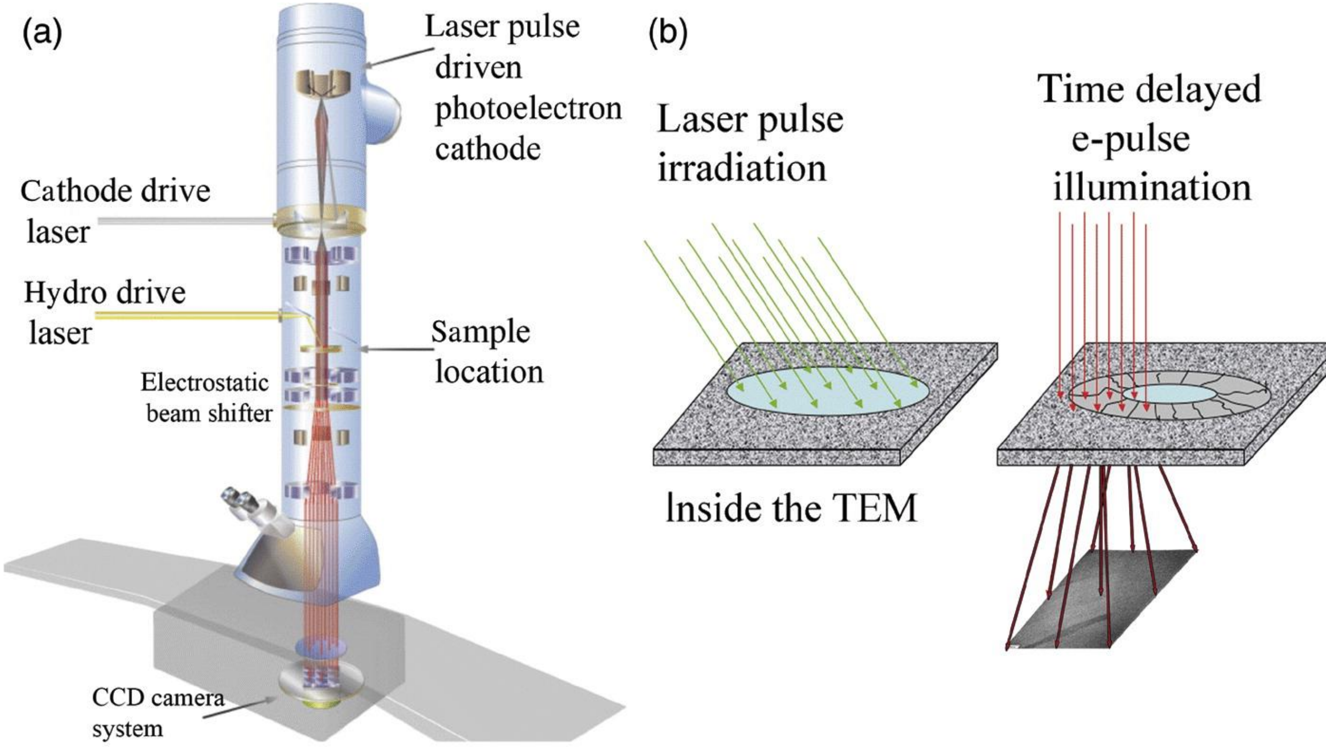
"A parametric study of morphology selection in equiaxed dendritic solidification", G. Kim, T. Takaki, Y. Shibuta, K. Matsuura, M. Ohno, *Comp. Mat. Sci.*, 2019



"Orientation selection in dendritic evolution", T. Haxhimali, A. Karma, F. Gonazles, M. Rappaz, *Nature*, 2006

DTEM experimental setup

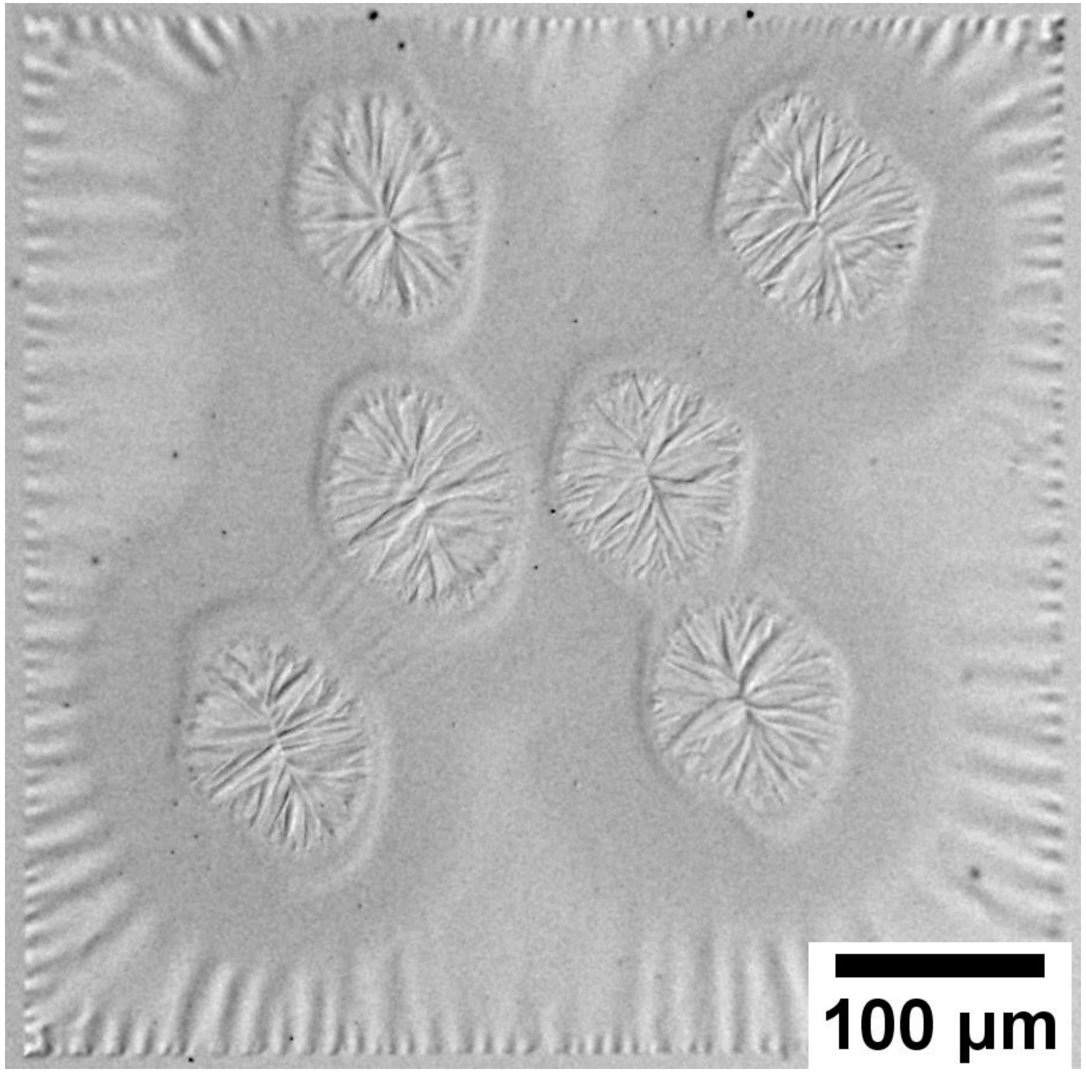
- Amorphous silicon nitride substrate with electron transparent window
- Thin film of alloy made by sputtering
- Laser melts region of alloy within window region
- A nine frame movie starting after some delay is produced by deflecting the beam to different regions of the detector



“Revealing the transient states of rapid solidification in aluminum thin films using ultrafast in situ transmission electron microscopy”, A. Kulovits, J. Wiezorek, T. LaGrange, B. Reed, G. Campbell, *Phil. Mag. Lett.,* 2011

Looking for DOT in Al-Ag DTEM foils

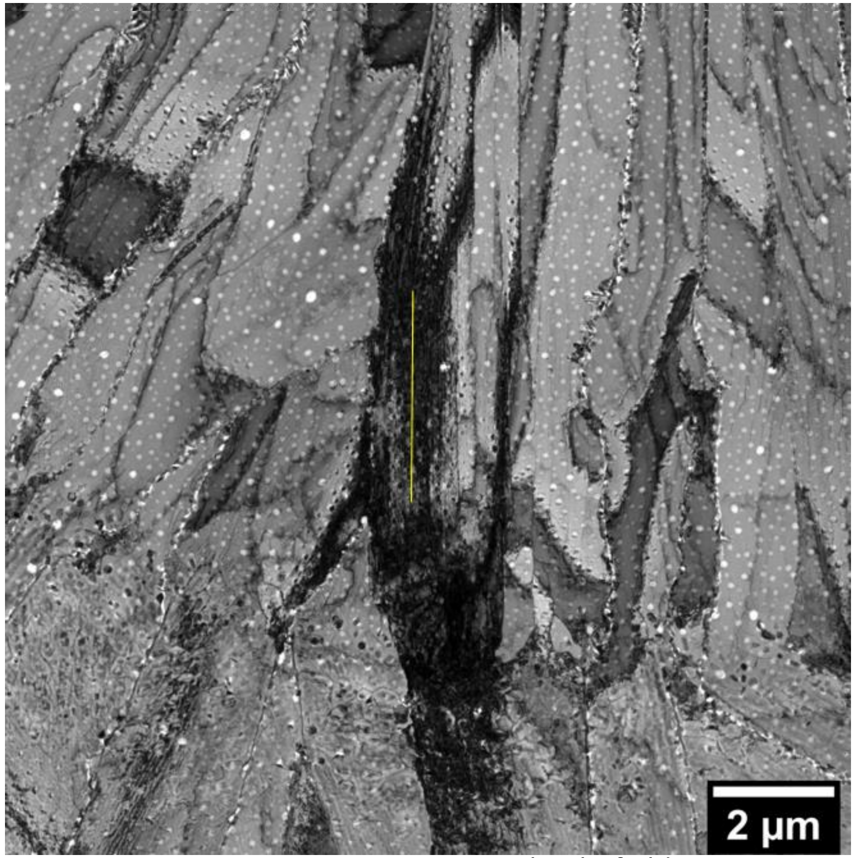
- DTEM differs from APS melts
  - Exclusively columnar dendrites
  - Faster interface velocity
  - Little convection
- So, do <110> dendrites still occur?



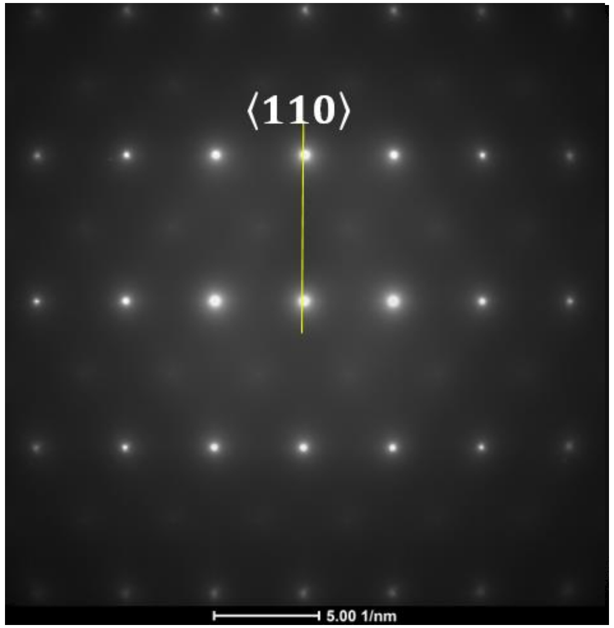
Visible light stereoscope

Finding DOT dendrites in Al-Ag DTEM

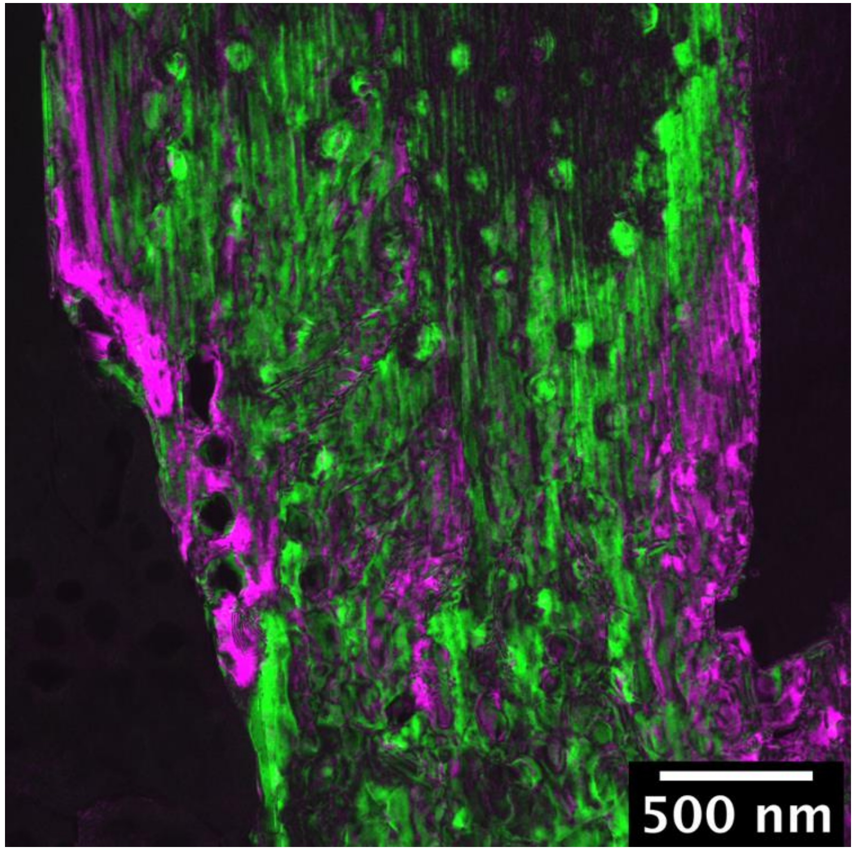
- Cannot get growth direction orthogonal to foil to directly diffract
- Trace analysis of SADP instead
- Dark field confirms entire grain is crystallographically consistent



Down zone bright field



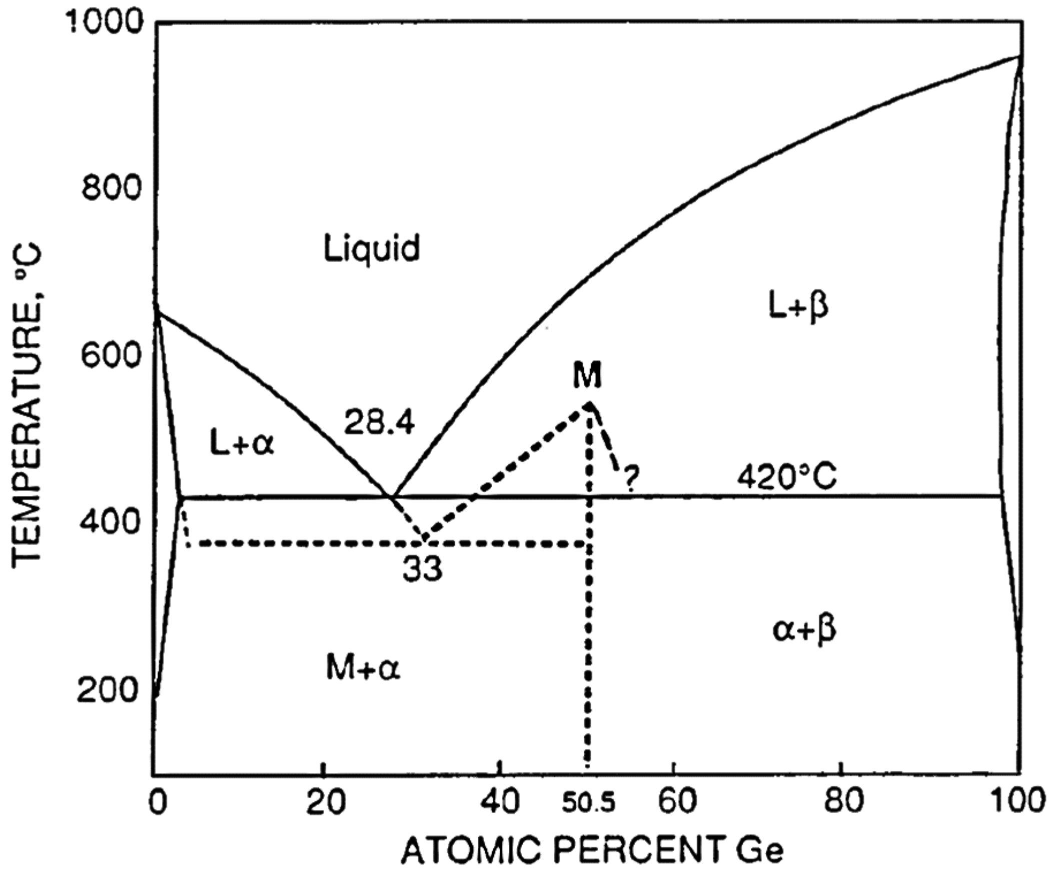
211 zone axis SADP



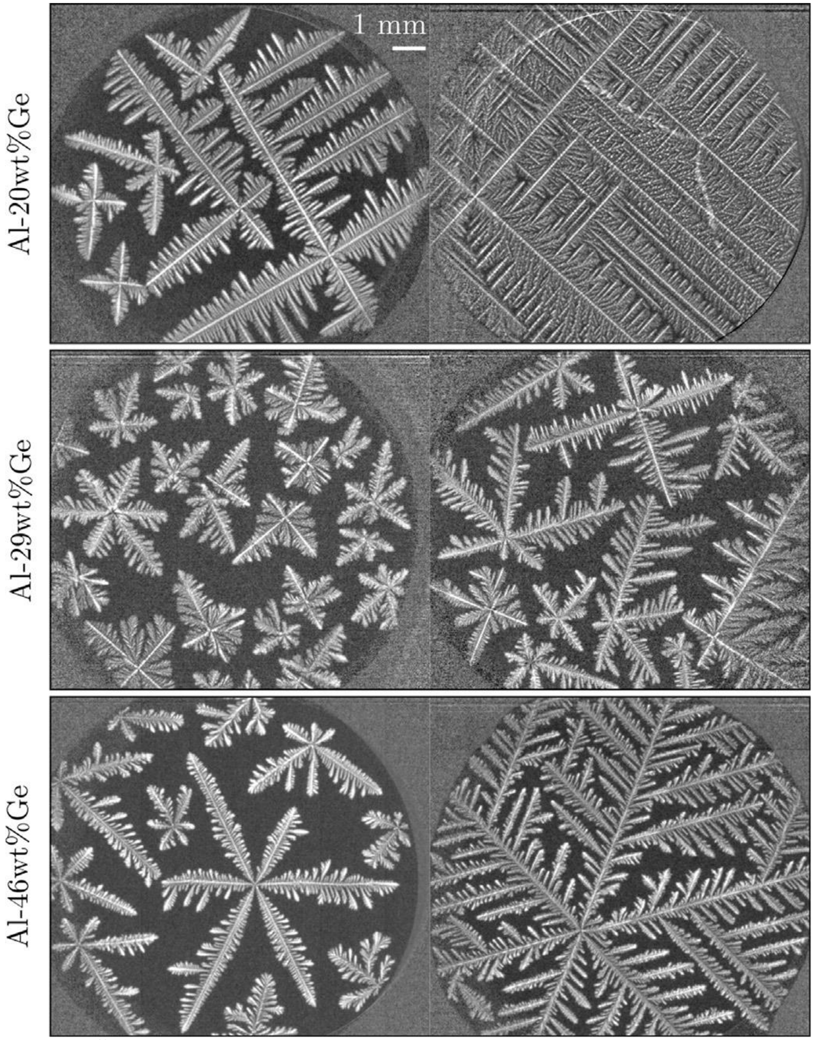
Composite dark field

Examining old Al-Ge DTEM foils

- Publication from another group shows DOT in Al-Ge system, but at lower velocities



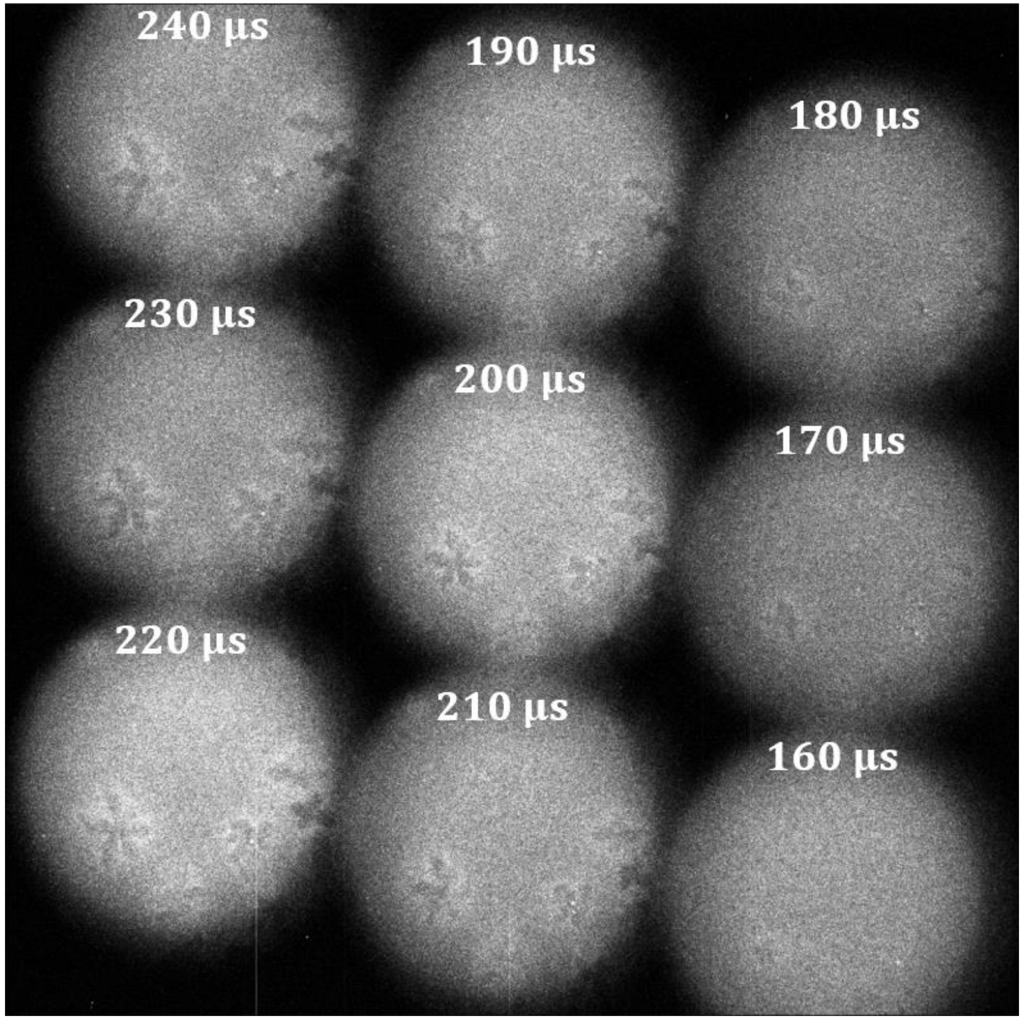
"Nonequilibrium Behavior in the Al-Ge Alloy System: Insights into the Metastable Phase Diagram", T. Laoui, M. Kaufman, *Met. Trans. A*, 1991



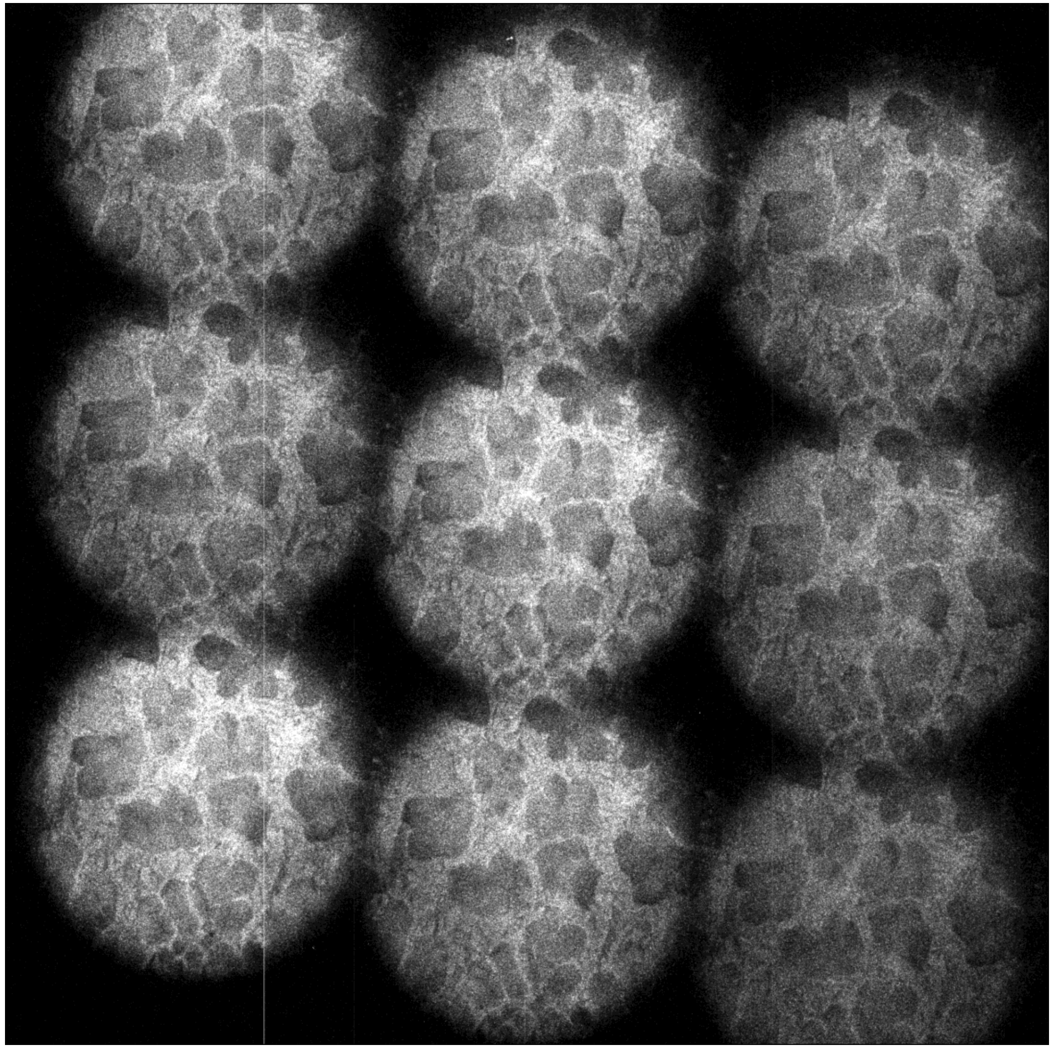
"Dendrite orientation transition in Al-Ge alloys", M. Becker, J. Dantzig, M. Kolbe, S. Wiese, F. Kargl, *Acta Mat.*, 2018

Solidification starts with primary Ge

- EDS scans of as-sputtered region confirm hypereutectic compositions



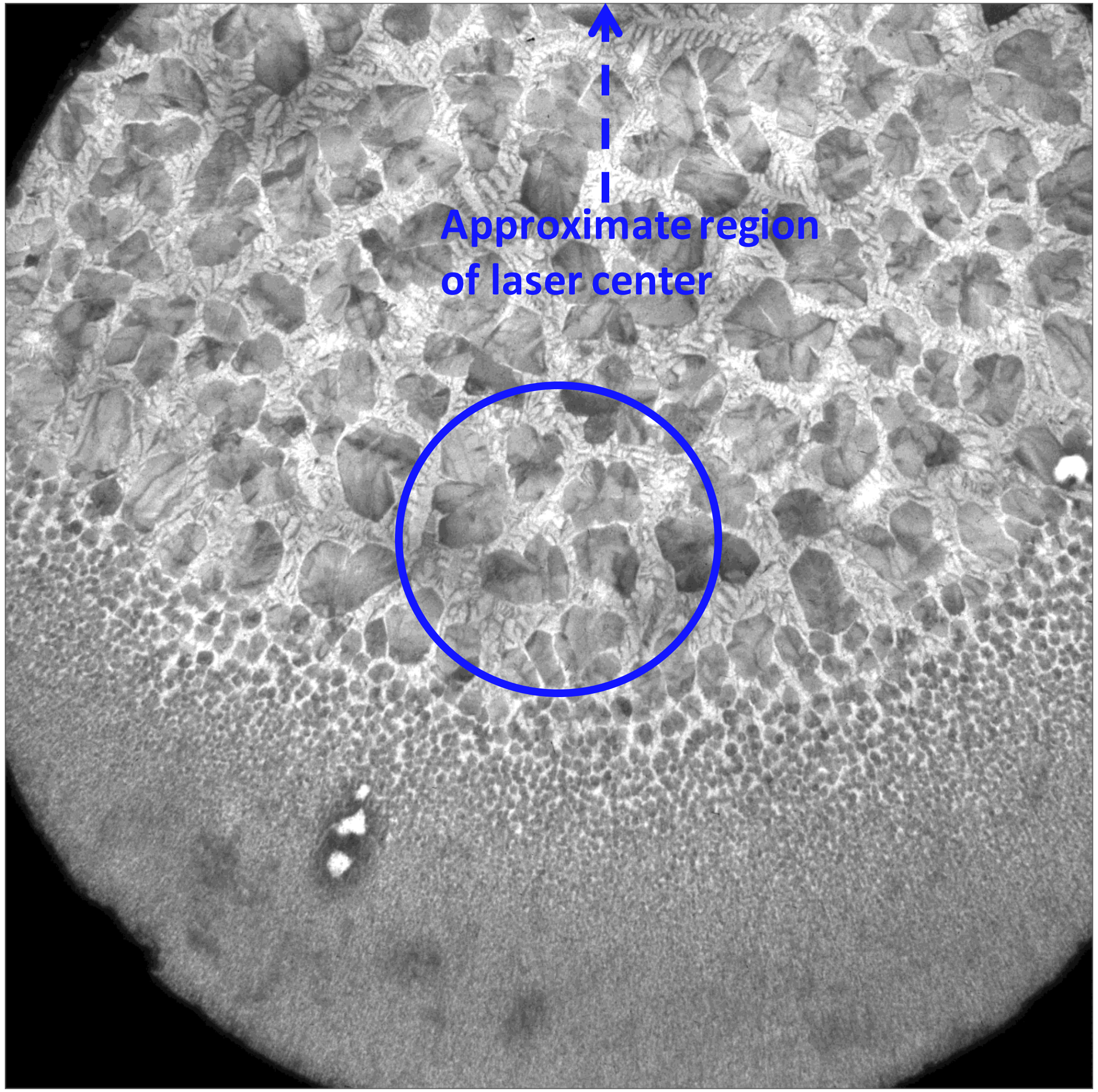
DTEM, 160 µs delay, 10 µs interval



DTEM after solidification

Solidification finishes with dendrites

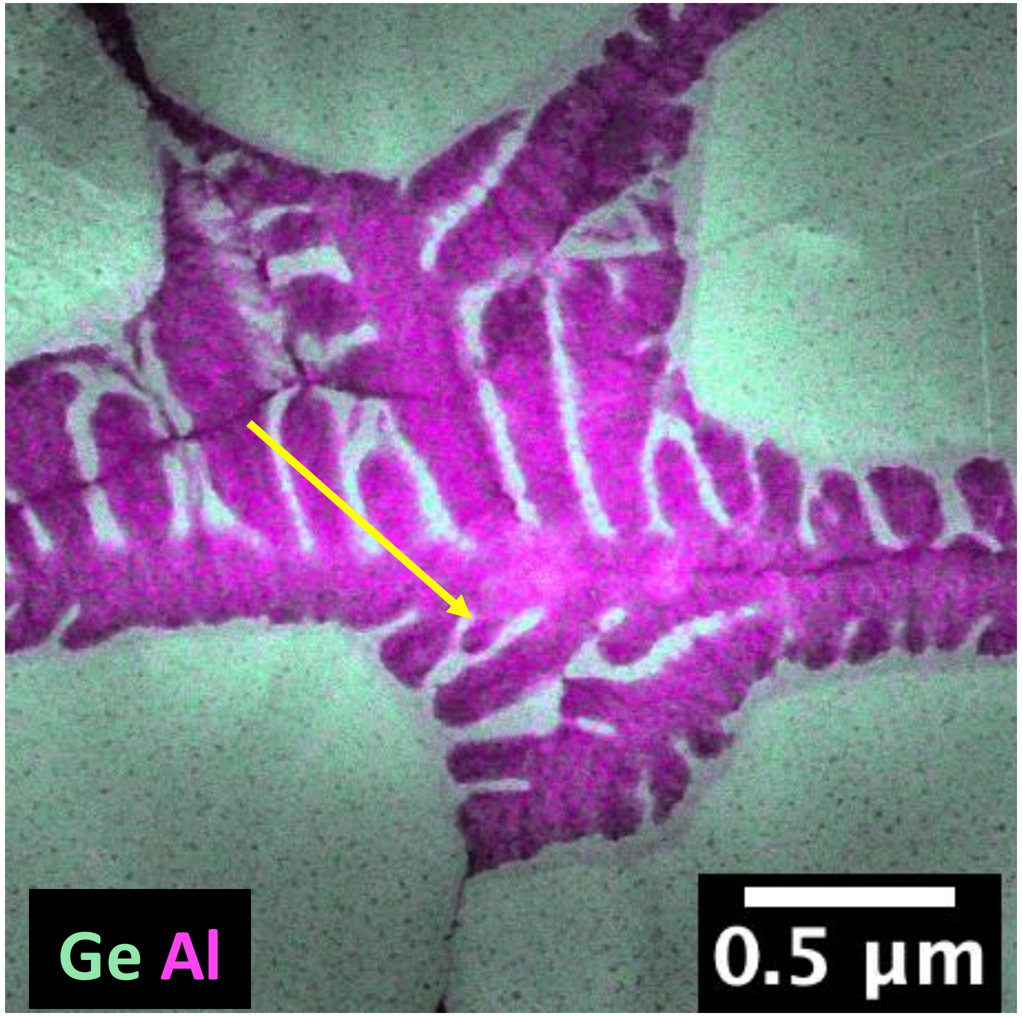
- Spaces between Ge crystals filled with dendrites and interdendritic liquid
- Shell develops around Ge crystals as well



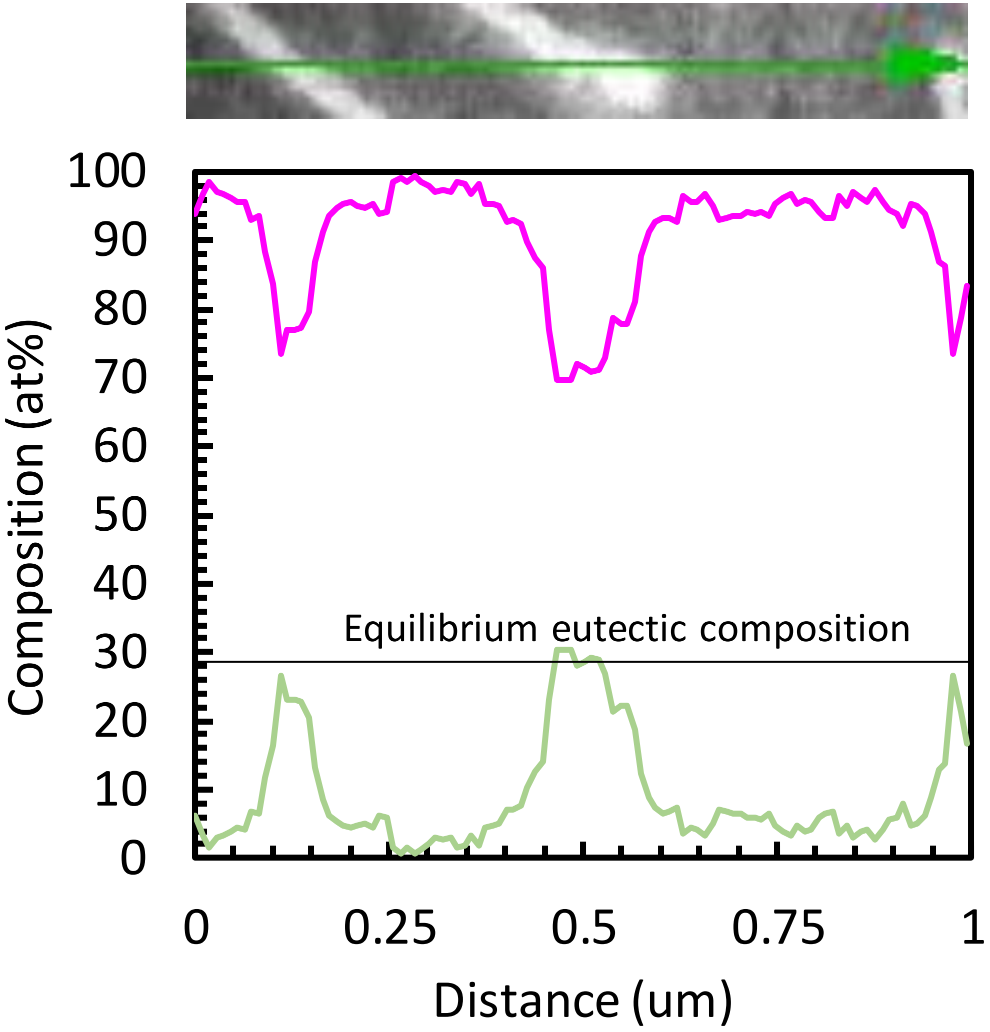
Bright field after DTEM

Dendrites are Al

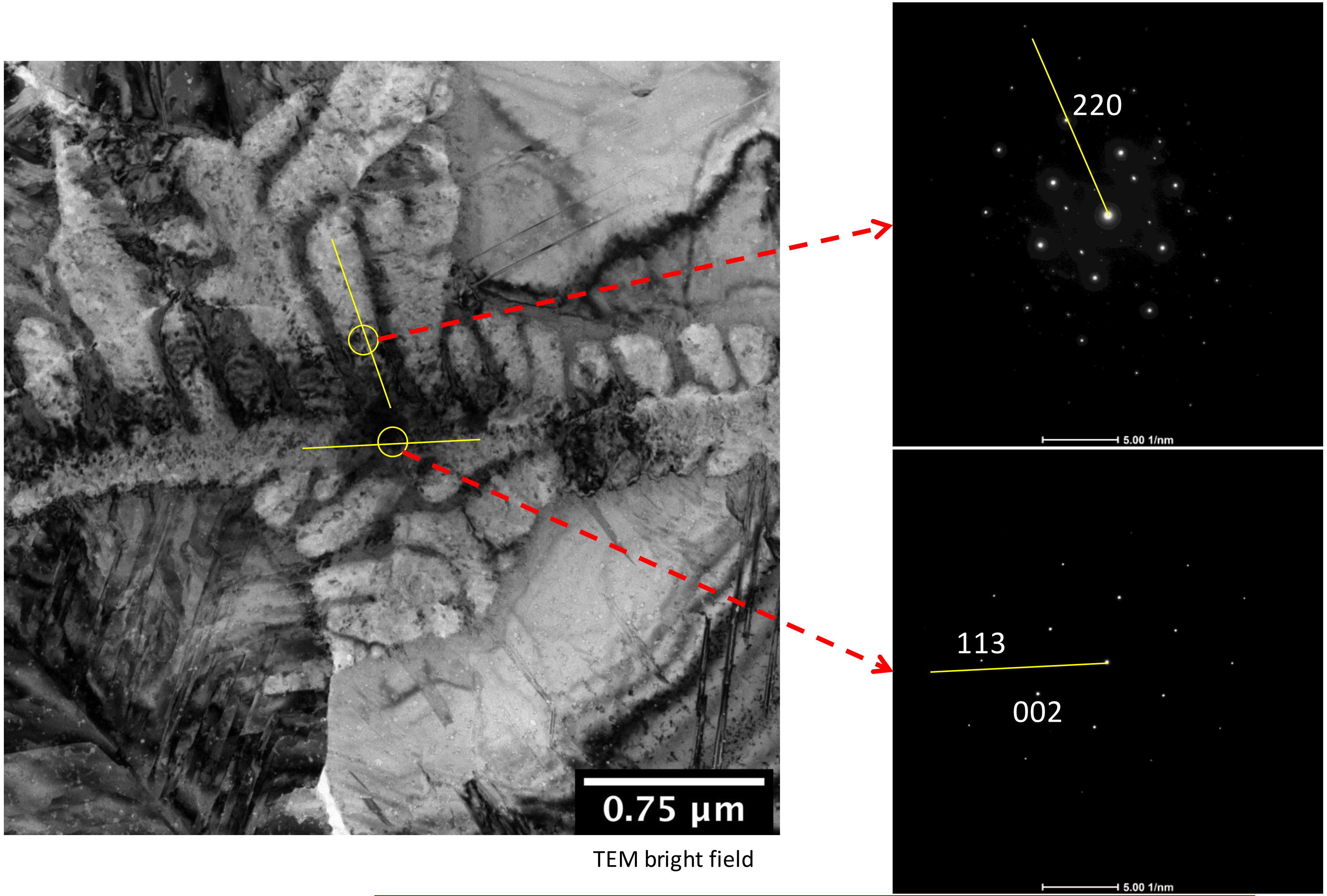
- Surprising for a hypereutectic alloy



STEM-EDS

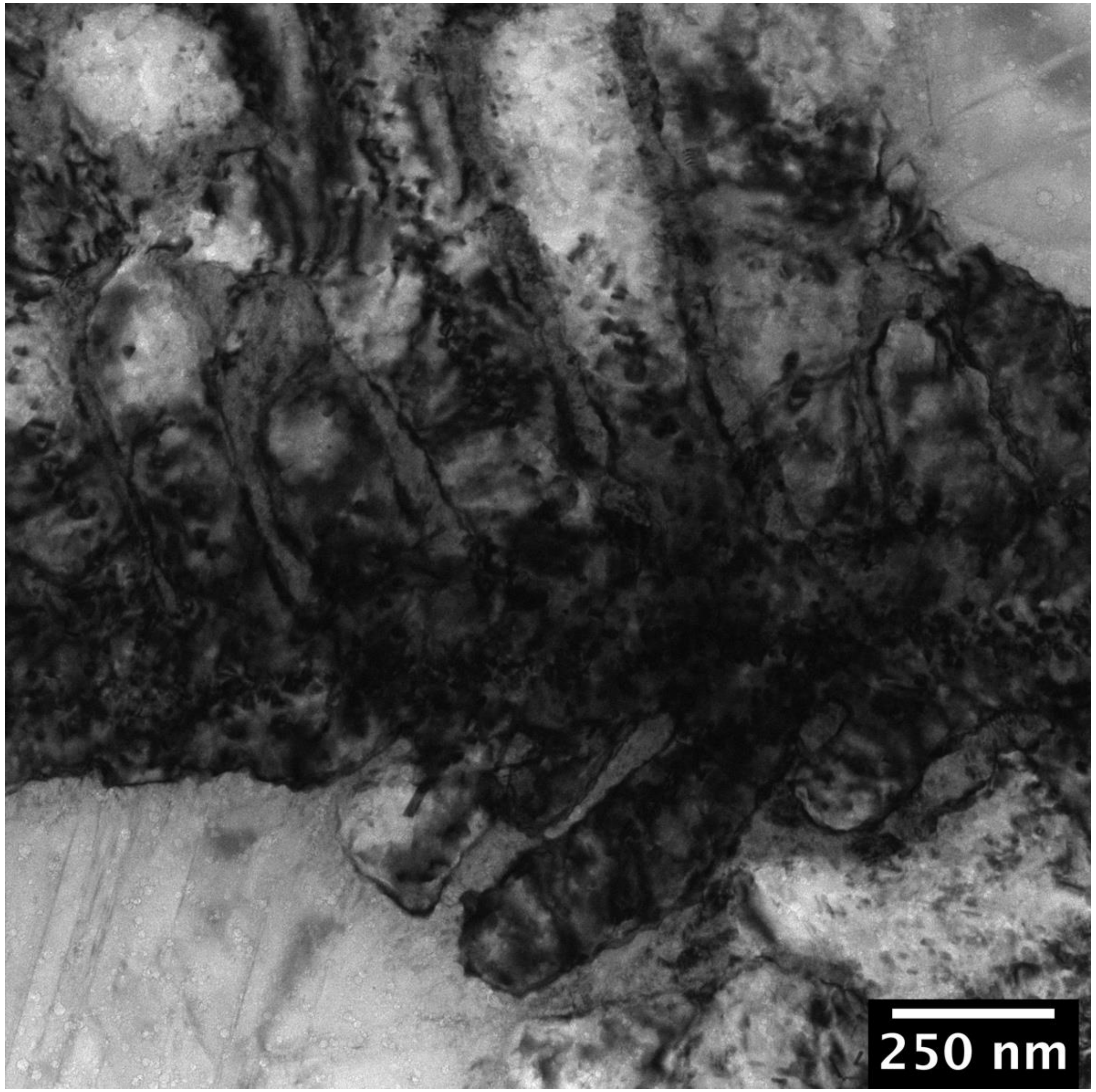


SADP analysis of growth directions

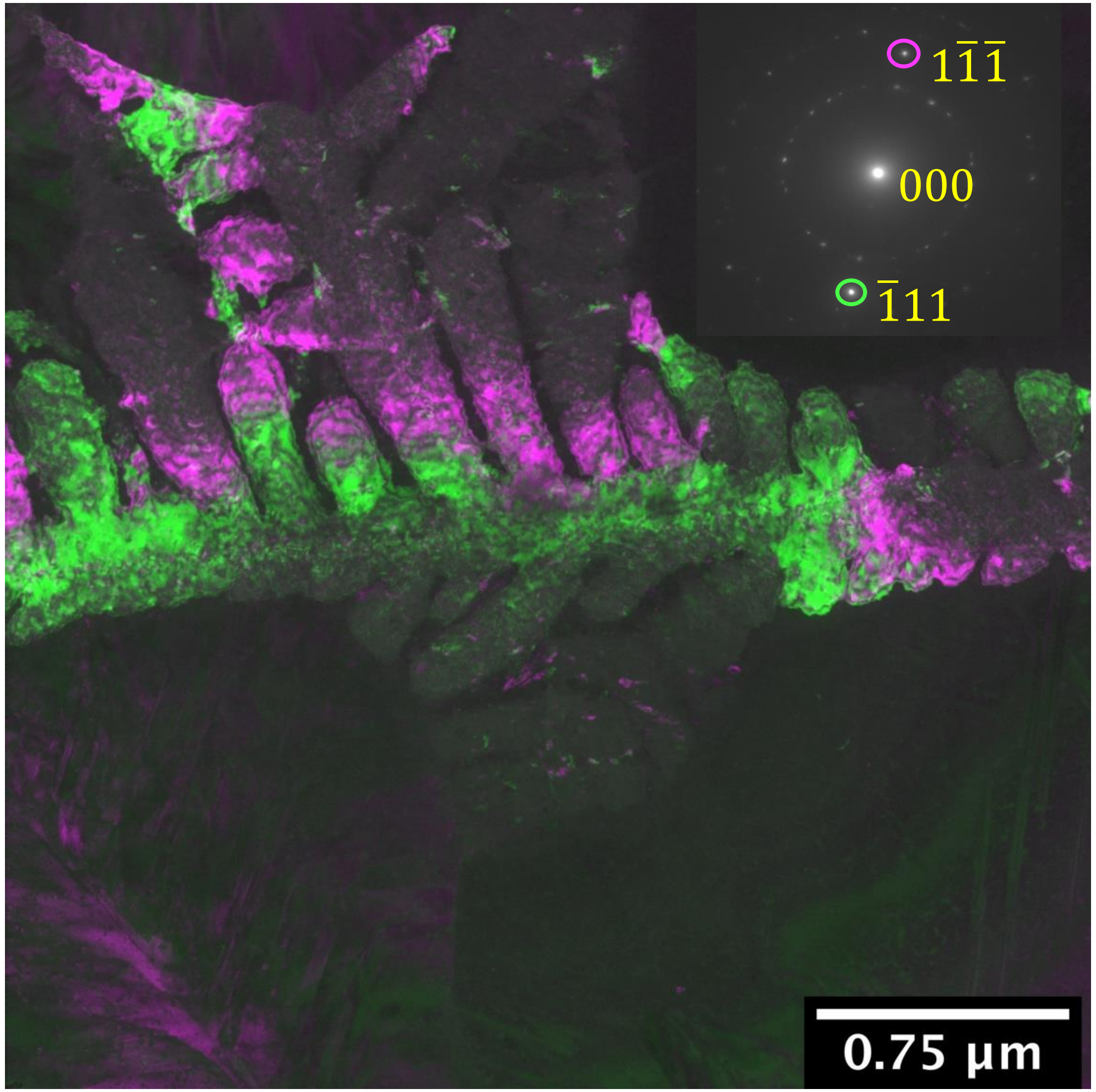


TEM bright field

Dark field confirms it is one crystal

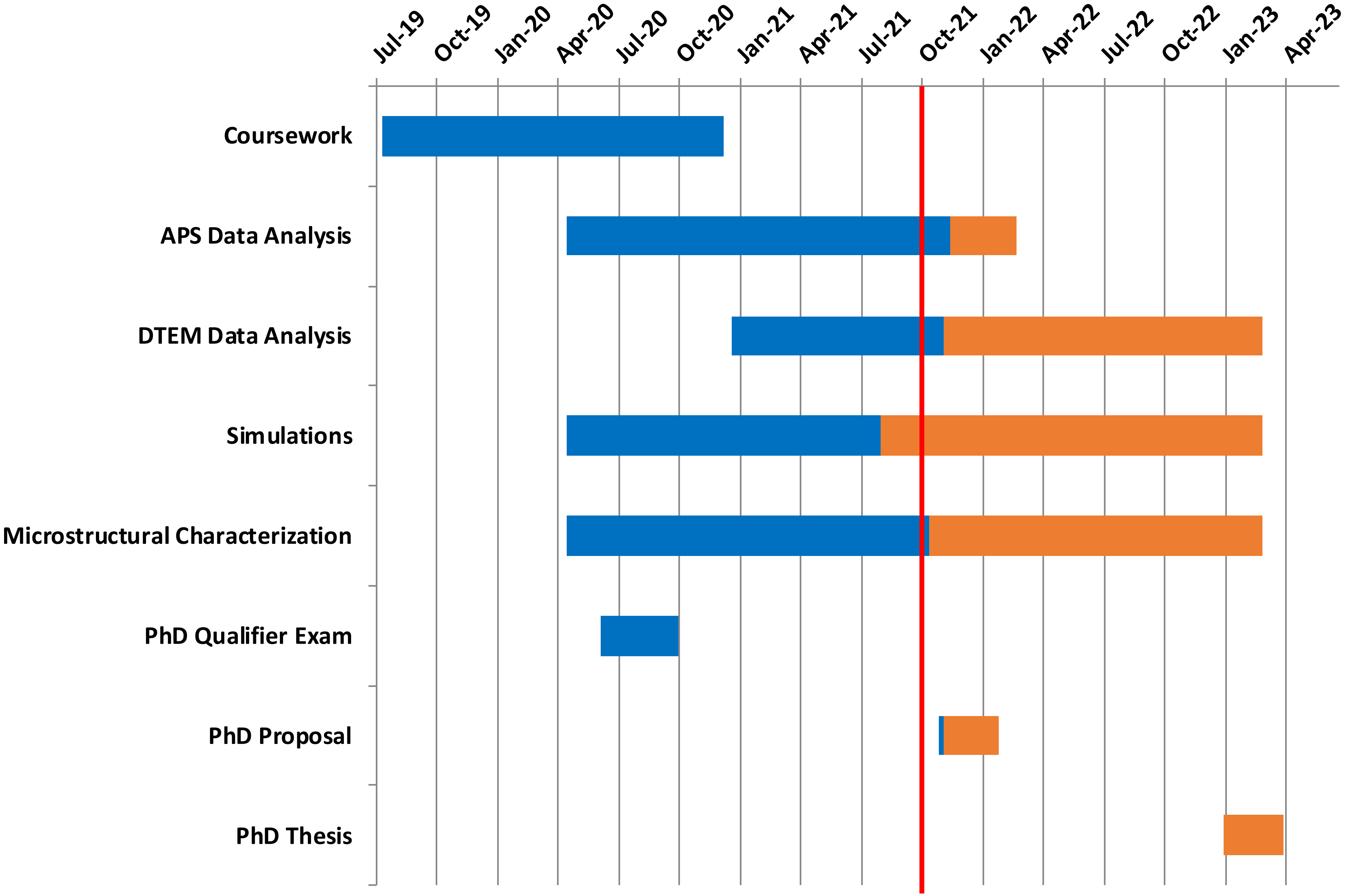


TEM bright field down zone



TEM dark field

Gantt Chart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Coursework
APS Data Analysis
DTEM Data Analysis
Simulations
Microstructural Characterization
PhD Qualifier Exam
PhD Proposal
PhD Thesis

Challenges & Opportunities

- Residency at LANL
  - Working outside of Colorado
  - New equipment to learn, but also exciting new capabilities
    - Sputtering to create DTEM samples
    - Simulations towards solid-liquid interfacial energy anisotropy
    - Electron microscopes

Thank you!

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