Project 36C-L: Combining In-Situ and Ex-Situ Characterization to Understand Crystallographic Texture Development in Additive Manufacturing

Fall 2019 Semi-Annual Meeting
Colorado School of Mines, Golden, CO
October 9 - 11, 2019

Staff: Jonah Klemm-Toole (Mines)

Faculty: Amy Clarke (Mines) and Kester Clarke (Mines)

Industrial Mentors: TBD

Center Proprietary – Terms of CANFSA Membership Agreement Apply

Overview

- Motivation and Background
- Experimental Design
- In-situ and Ex-situ Characterization Results
- Preliminary Modeling and Simulations
- Summary
- Continuing Work
- Challenges and Opportunities

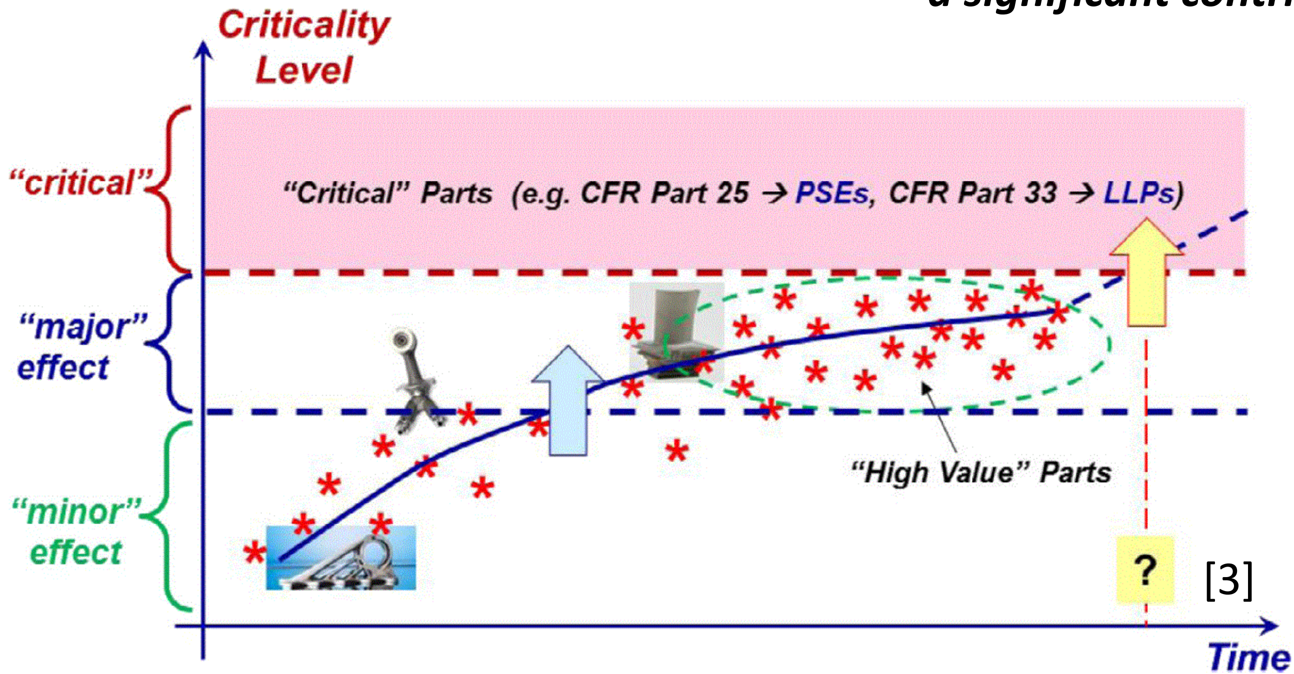
Motivation

National Academy of Sciences [1]

Federal Aviation Administration [2]

Airforce Research Laboratory [2]

→ ***Anisotropic mechanical properties*** limit the application of additively manufactured (AM) metal parts to high value, failure critical applications. ***Crystallographic texture is likely a significant contributor to anisotropy.***

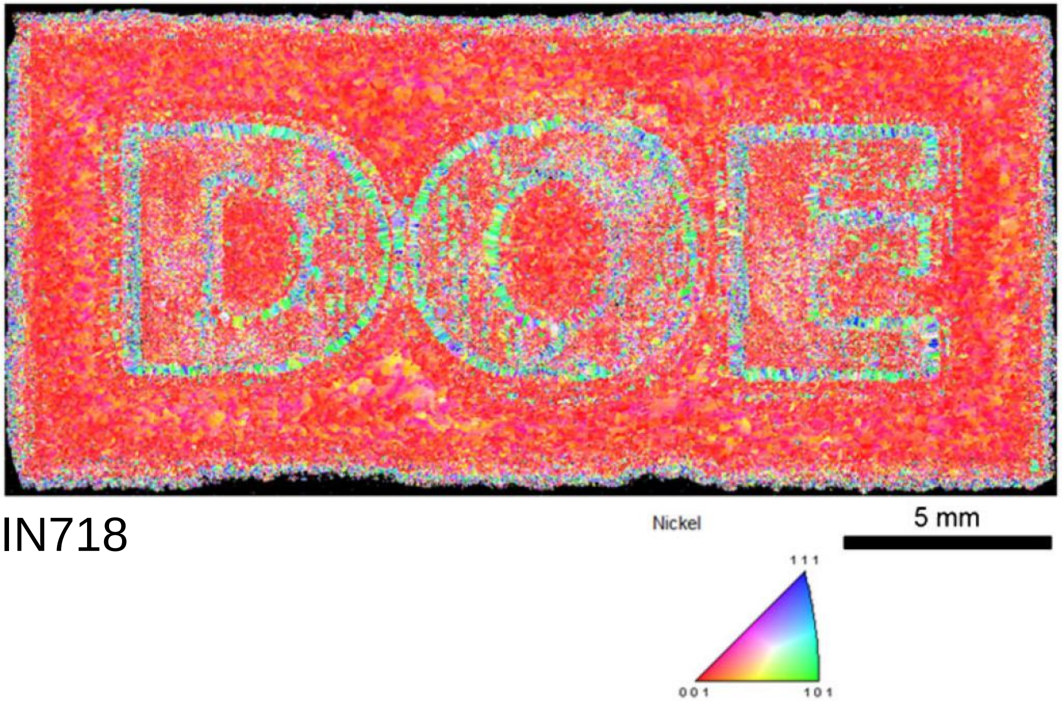


[3]

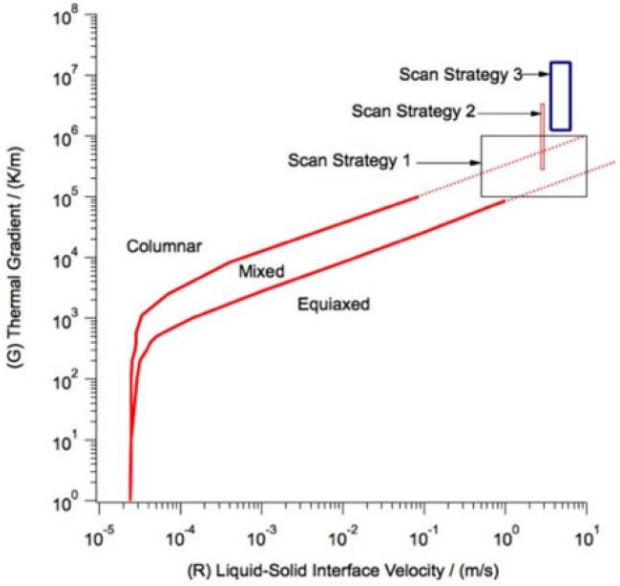
Insufficient understanding of processing-microstructure-mechanical property relationships limits application of AM

[1] D. Snyder, Nat. Acad. Sci., Washington D.C., pp. 47-50, 2015
[2] M. Seifi *et al.*, *JOM,* vol. 69, no. 3, pp. 439-455, 2017.
[3] M. Gorelik, *Int. J. Fat*, vol. 94, pp. 168-177, 2017.

Controlling Texture in AM



IN718



G and R calculated from heat transfer models

Large gradients and low velocities lead to columnar grains with preferred $\langle 100 \rangle$ directions parallel to the build direction

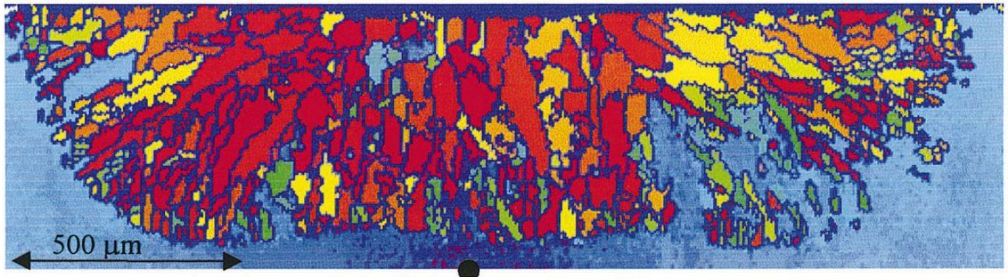
Smaller gradients and higher velocities induce the columnar to equiaxed transition (CET) and lead to random orientations

R.R. Dehoff, M.M. Kirka, W.J. James, H. Bilheux, A.S. Tremsin, L.E. Lowe, and S.S. Babu, “Site Specific Control of Crystallographic Grain Orientation Through Electron Beam Additive Manufacturing,” *Mater Sci Tech,* vol. 31, no. 8, 2015.

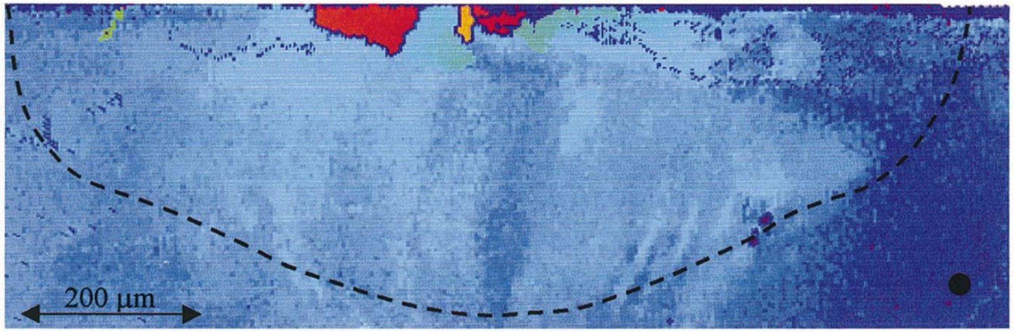
Controlling Texture During Laser Welding of Single Crystals

New grain orientations nucleated

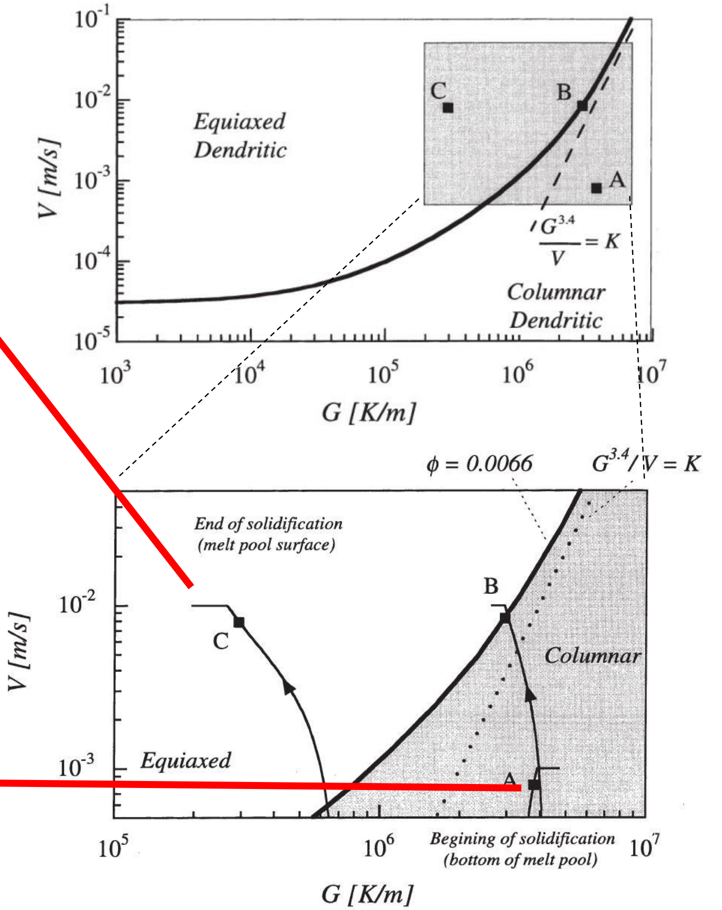
CMSX-4



Most of weld maintains ⟨100⟩ orientation of substrate



G and V calculated from heat transfer models



Gradient decreases and velocity increases from bottom to top of melt pool which induces the CET and random grain orientations

M. Gaumann, C. Bezecon, P. Canaliz, and W. Kurz, “Single Crystal Laser Deposition of Superalloys: Processing-Microstructure Maps,” *Acta Mater,* vol. 49, 2001.

Experimental Objectives

- **Develop a deeper understanding of texture development in AM Ni-Based Superalloys**
  - *Start with understanding microstructure and texture in isolated spot melts and rasters on single crystal samples*
  - *Proceed to influences of multiple melting cycles and overlapping spot melts/rasters*
- **Evaluate the influences of several parameters on the CET and subsequent crystallographic orientations after solidification**
  - *Initial substrate orientation (beyond* $\langle 100 \rangle$ *orientation)*
  - *Laser power*
  - *Composition*
- **Directly measure solidification velocity using the AM Simulator at the Advanced Photon Source (APS) at Argonne National Lab**
  - *Will use velocity measurements to calibrate heat transfer models in order to calculate thermal gradients*

Experimental Set Up – Laser Melting + In-Situ Radiography

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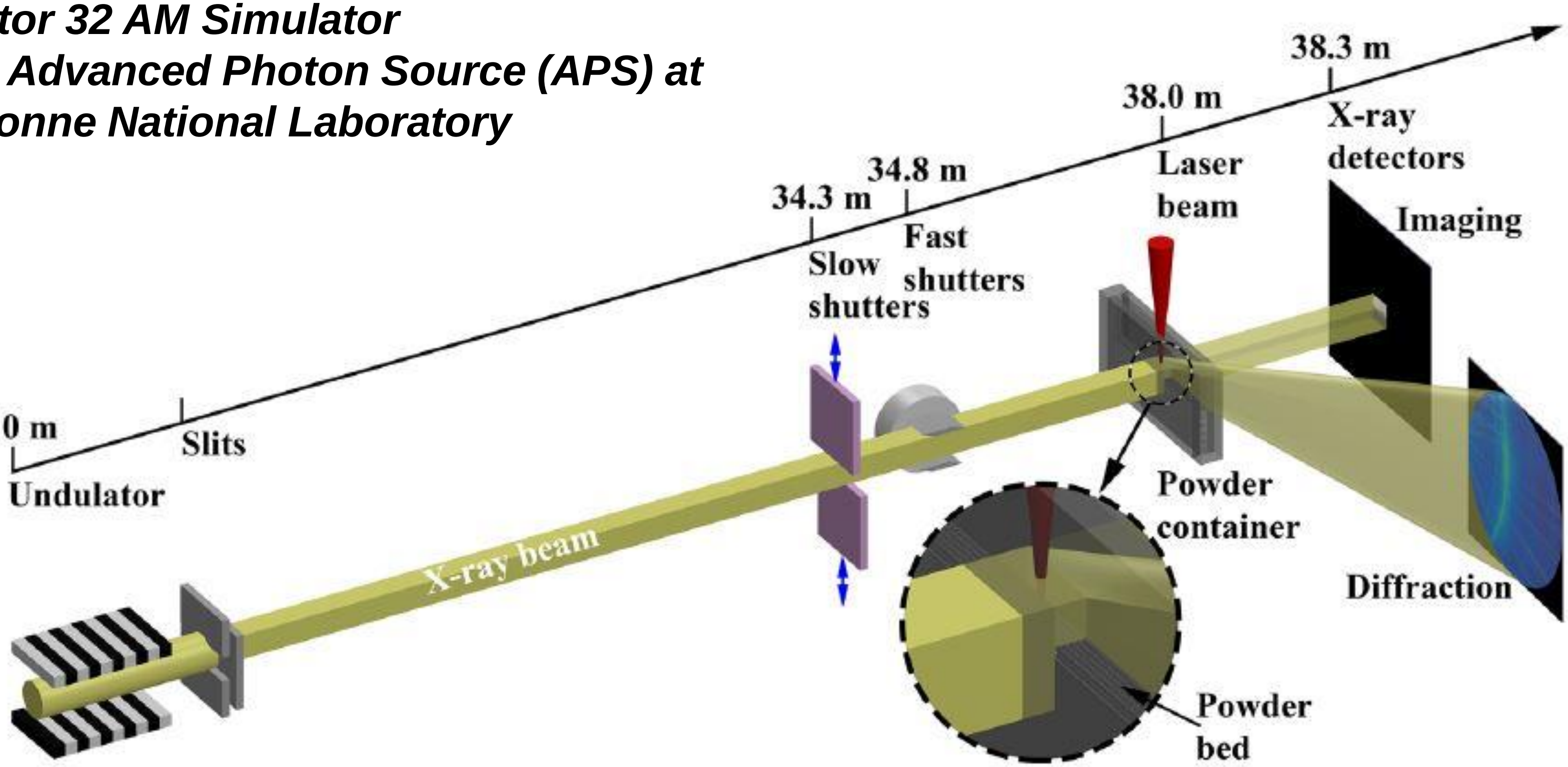
Sector 32 AM Simulator
The Advanced Photon Source (APS) at Argonne National Laboratory



High speed radiography allows for tracking melting and solidification with high temporal resolution

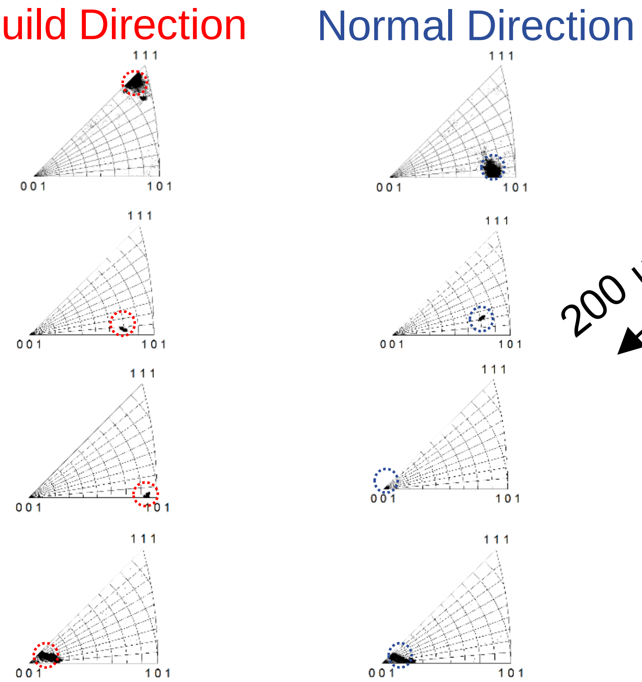
C. Zhao, K. Fezzaa, R.W. Cunningham, H. Wen, F. De Carlo, L. Chen, A. Rollett, T. Sun, "Real-Time monitoring of laser powder bed fusion process using high-speed X-ray imaging and diffraction," *Scientific Reports,* 7:3602, 2017.

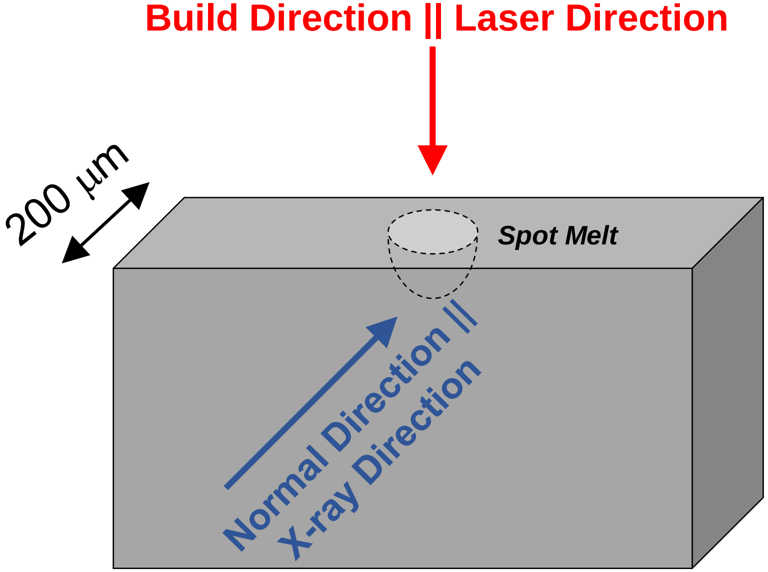
Sample Conditions

- **Spot Melting Parameters**
  - 106, 156, 208, 260 W for 1 ms
- **Compositions**
  - R2: Ni – 1.9Mo – 6.6 Al
  - R4: Ni – 22.2Mo – 2.8 Al

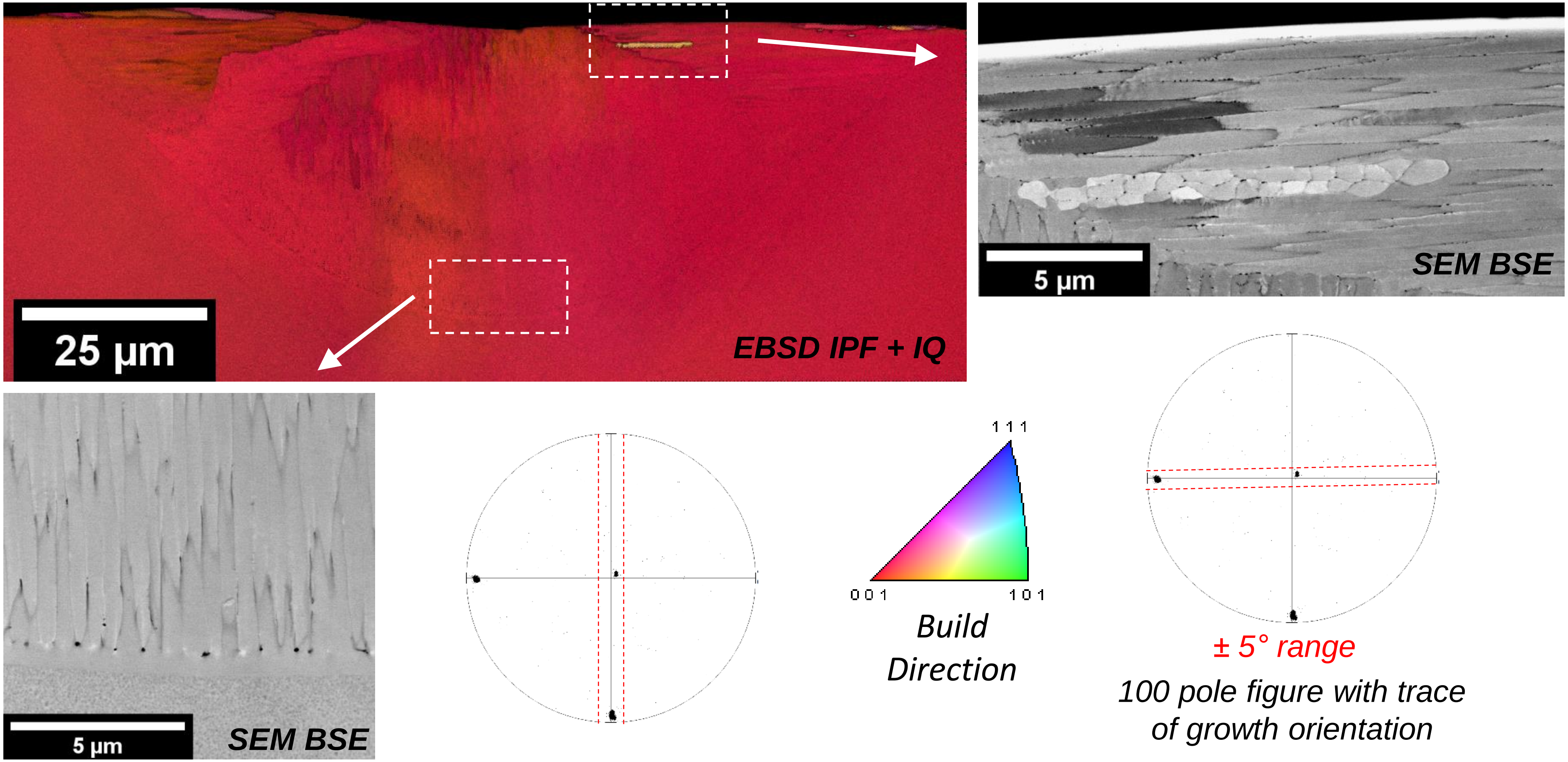
Provided by T. Pollock (UCSB)
(Fahrmann *et al*. Mat Sci Eng A 1999)

- **Single Crystal Orientations**
  - R2-111
  - R2-110
  - R4-110
  - R4-100





Ex-Situ EBSD + SEM to Determine Growth Orientations

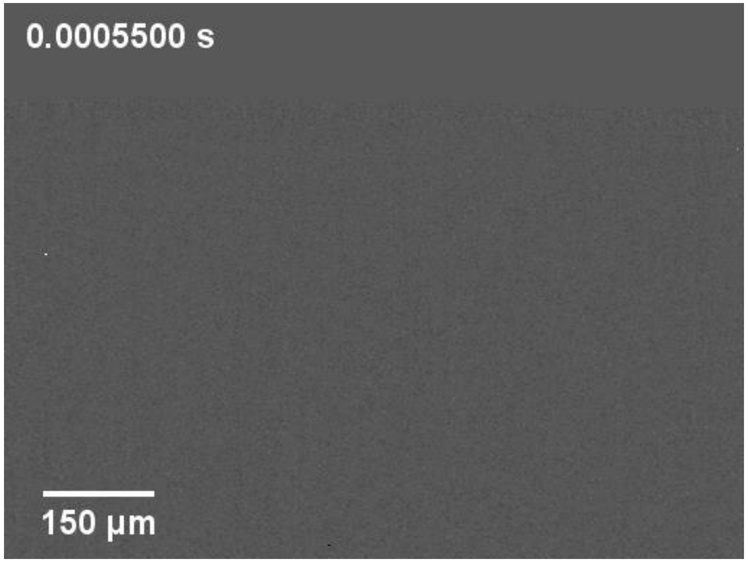


100 pole figure with trace of growth orientation

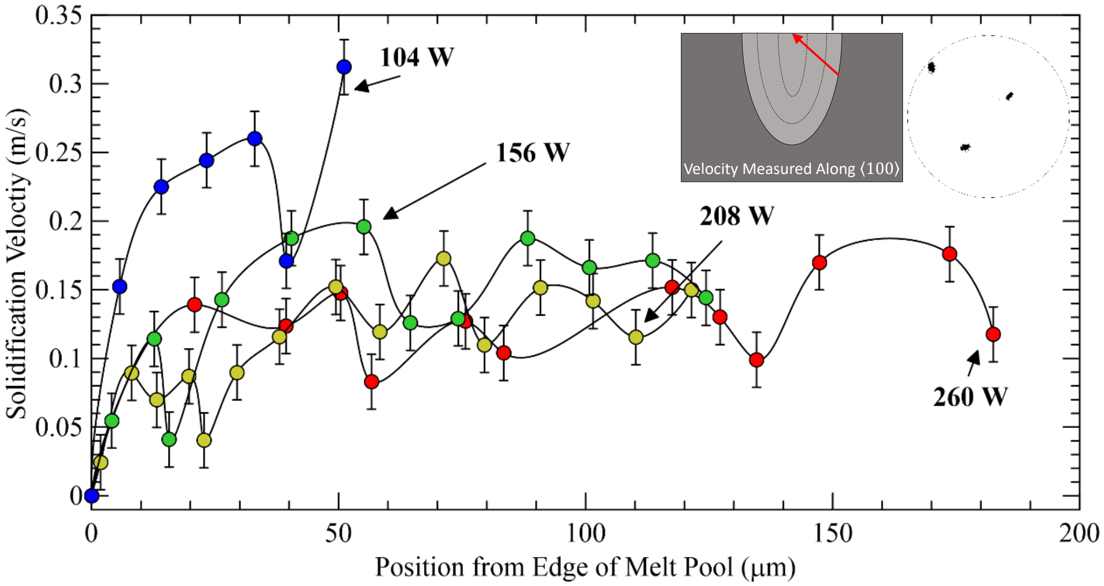
Growth orientations determined by EBSD + SEM in order enable dendrite tip velocity measurements from radiography

Analysis of In-Situ Radiography

Radiography of R2-111
260 W 1 ms



Solidification Velocity Measurements for R2-111

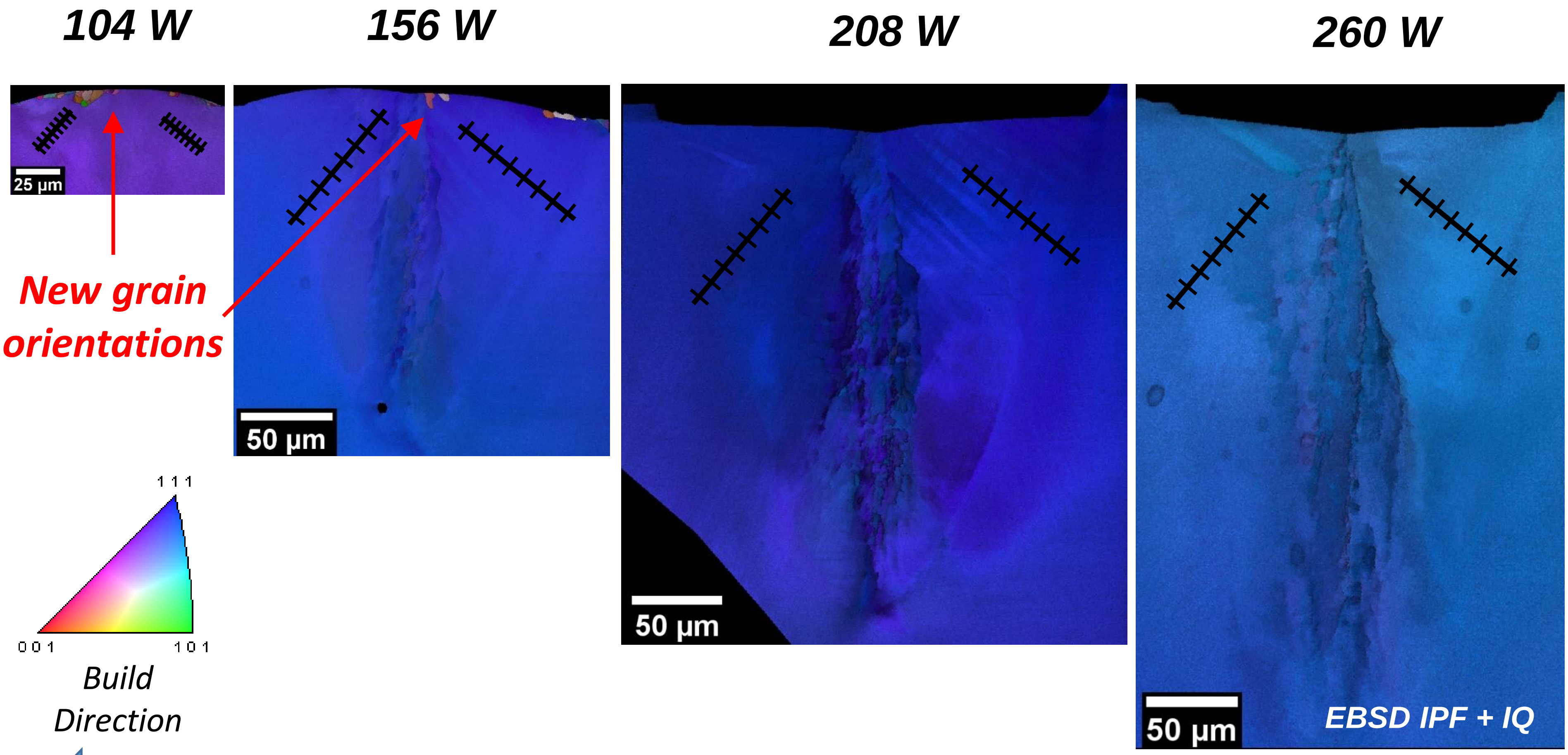


Thanks to Gus Becker at Mines for ImageJ scripts!

Solidification velocity increases from bottom to top of pool (confirming prediction from heat transfer models)

Lower laser power leads to higher solidification velocity near top of pool

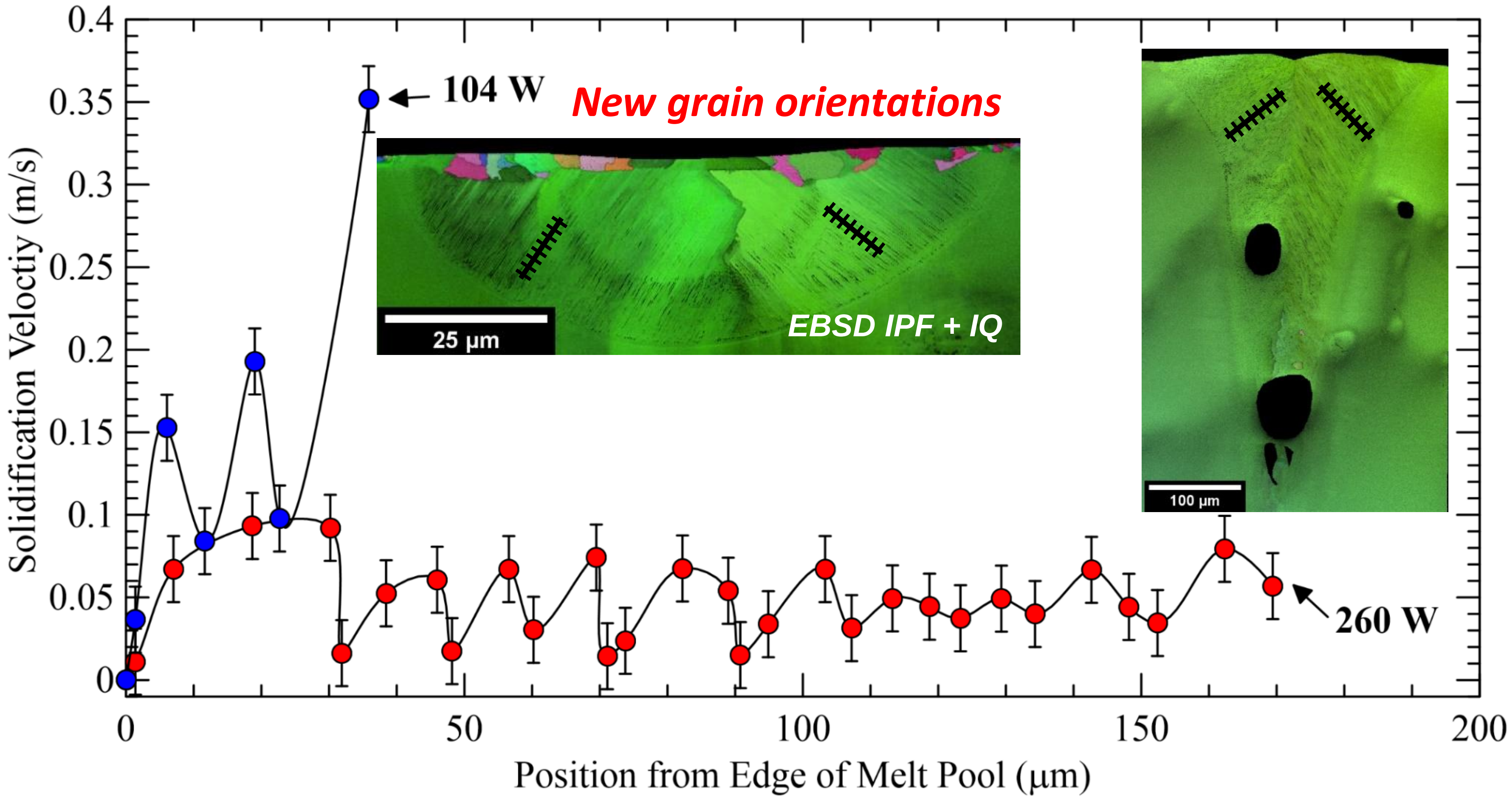
EBSD of R2-111 Effect of Laser Power



New grain orientations

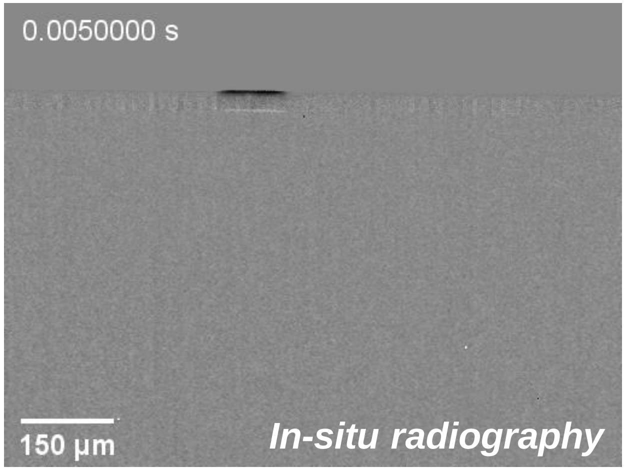
Lower laser power, higher solidification velocity, greater propensity to induce CET

Solidification Velocity and EBSD Results for R4-110

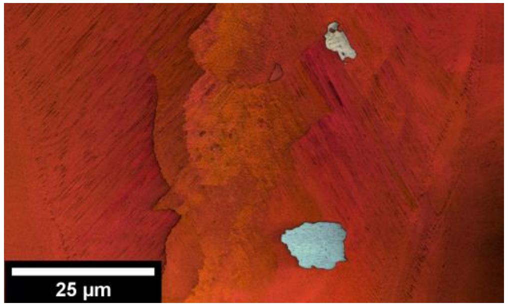


Lower laser power exhibits higher solidification velocity and new grain orientations at top of pool (CET induced) – same trend as with R2-111

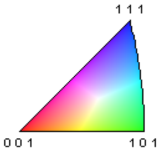
Keyhole Collapse Causing CET in R4-110 260 W



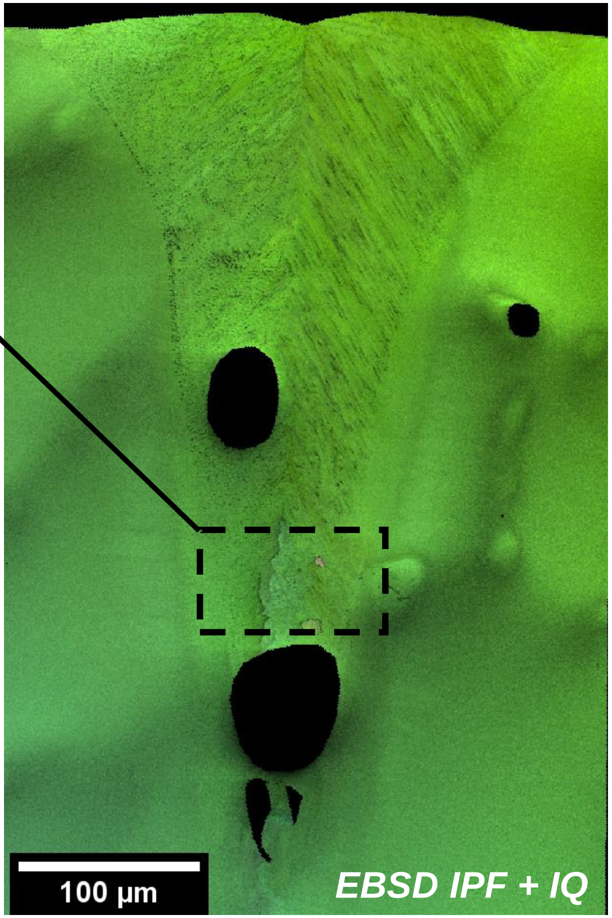
Normal Direction

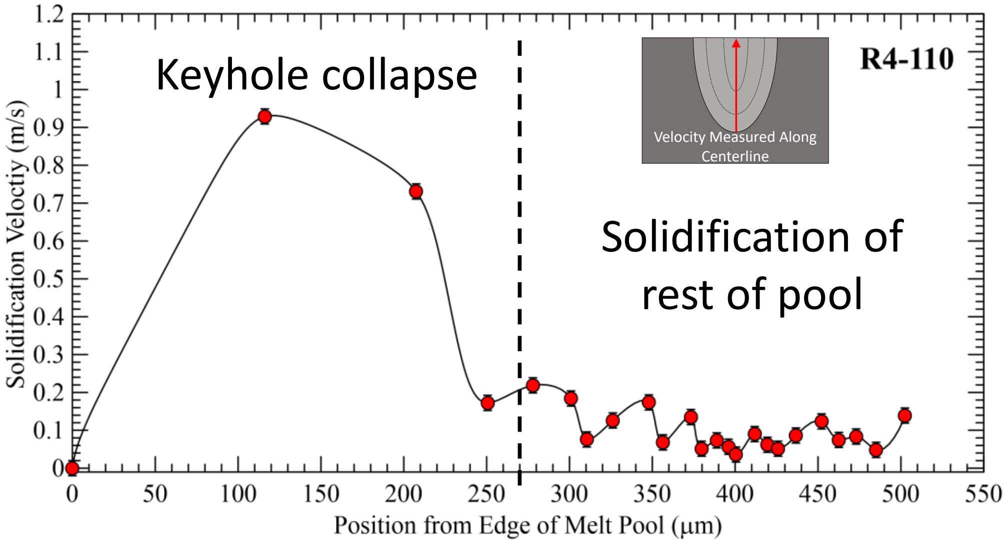


New grain orientations



Build Direction



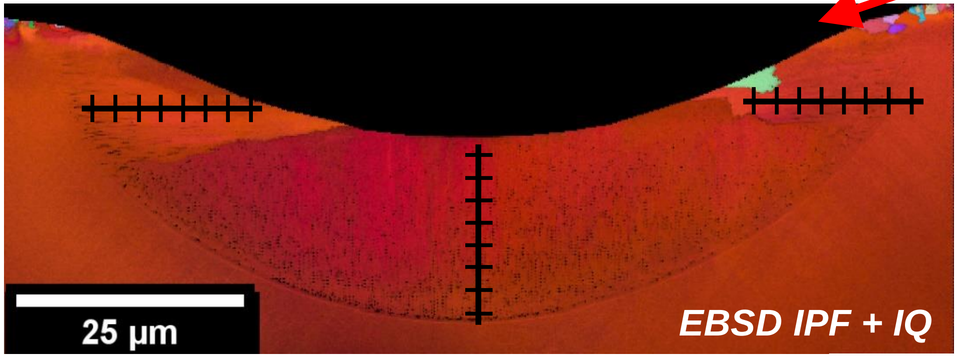


High solidification velocity during collapse of keyhole induced the CET

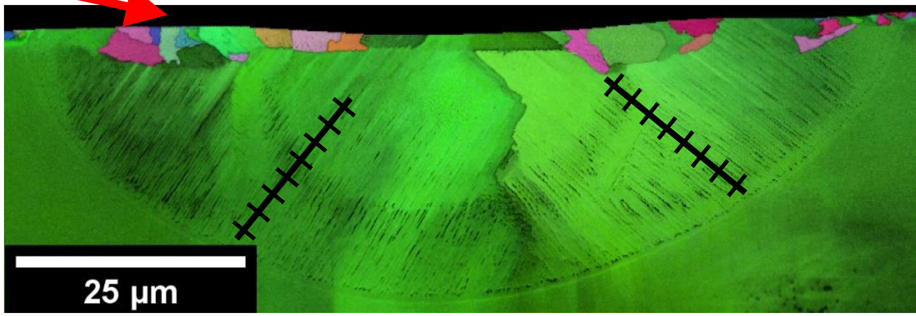
Influence of Substrate Orientation

New grain orientations

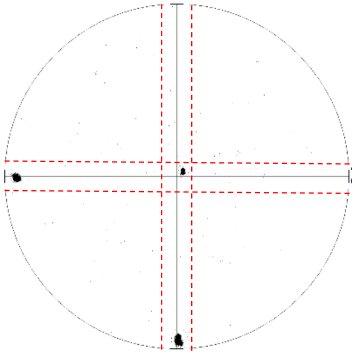
R4-100 106 W



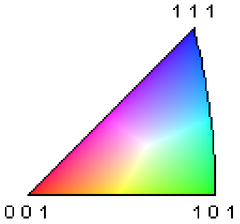
R4-110 106 W



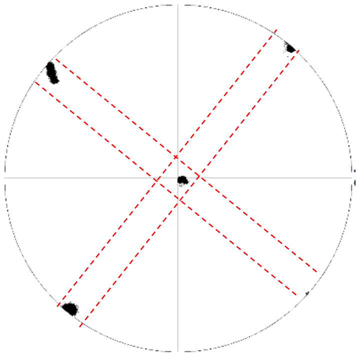
100 Pole Figure w/ traces of growth orientations



± 5° range



Build Direction



± 5° range

100 Pole Figure w/ traces of growth orientations

Both orientations show growth along $\langle 100 \rangle$ orientations and new grain orientations at top of weld (CET) – solidification velocity similar for both substrate orientations

Influence of Composition on Microstructure Development

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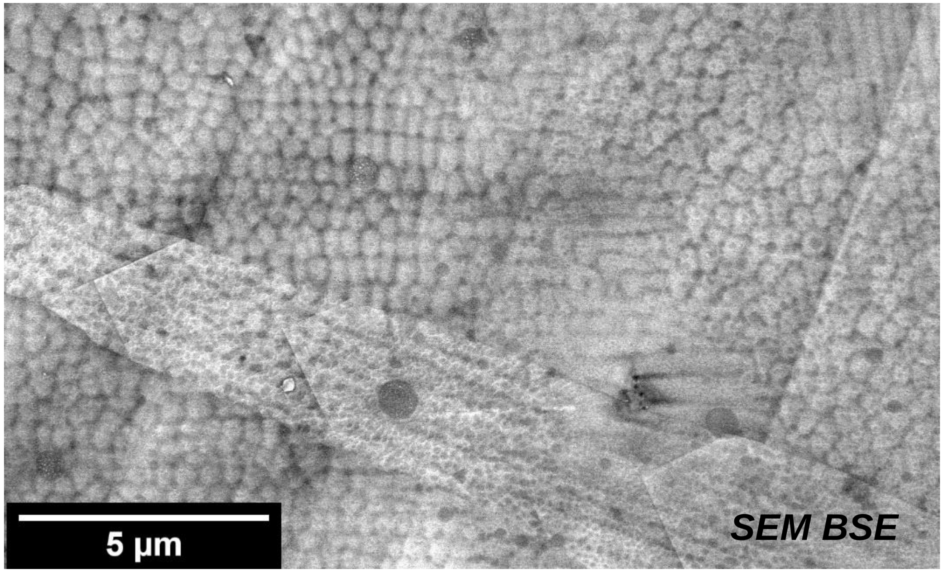
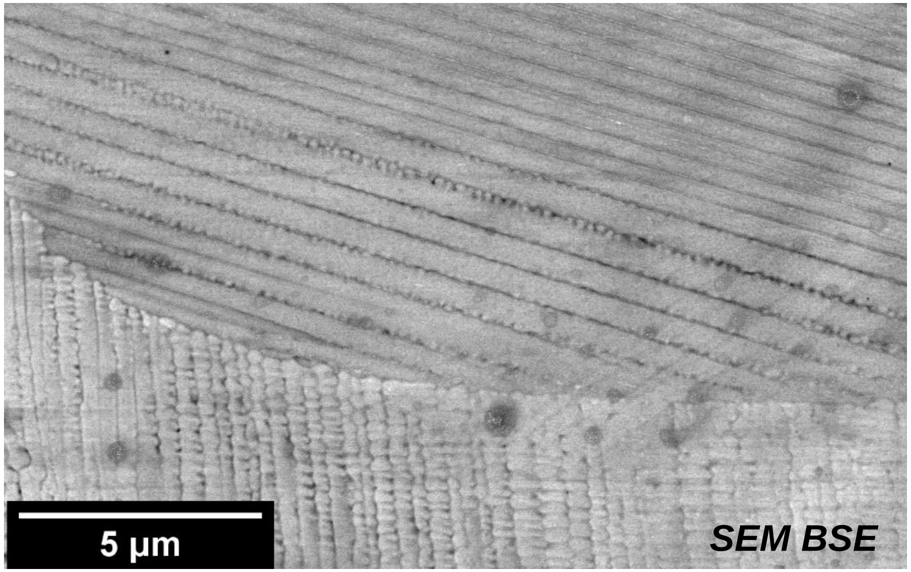
R2-110 106 W



Secondary arms NOT observed

R4-110 106 W

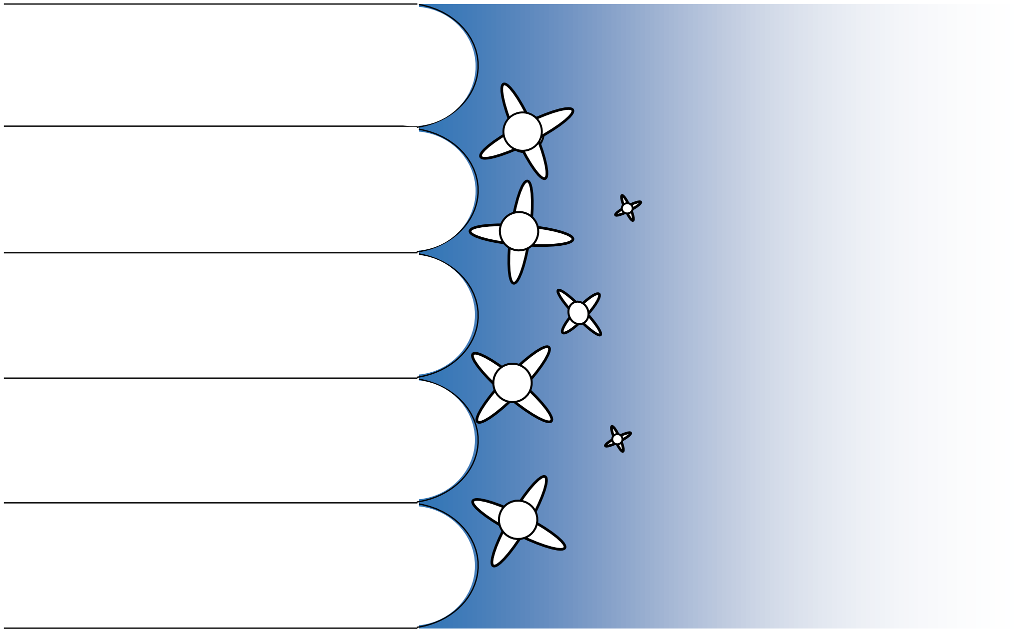


Secondary arms observable

Alloy R4 (higher Mo content) shows well developed secondary dendrite arms, whereas no secondary arms are observed in R2 (lower Mo content)

Indicates Mo content affects solidification microstructure – likely greater undercooling in liquid ahead of solidifying front

Columnar to Equiaxed Transition Model



- **Gäumann modification to Hunt CET model**
- **Uses Kurz-Giovanola-Trivedi (KGT) model to calculate undercooling during columnar growth**
- **Incorporates velocity dependent solute partitioning**
- **Thermodynamic data taken from ThermoCalc TCNI8 database**
- **Nucleation site density N_0 calibrated to e-beam powder bed fusion AM IN718 samples**

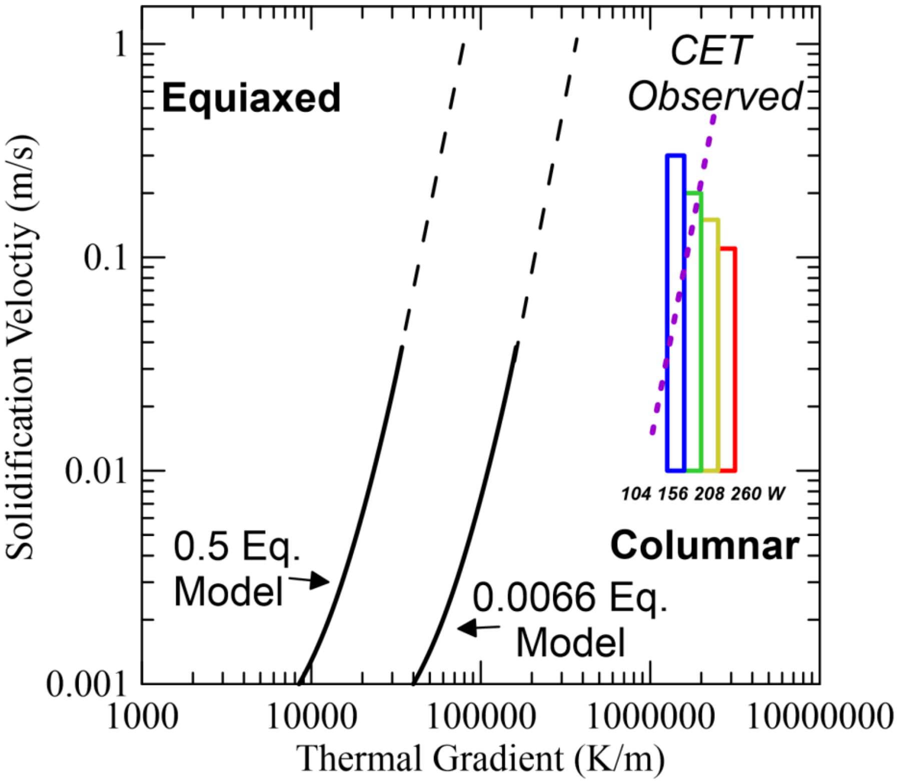
$$G = \frac{1}{n+1}\sqrt[3]{\frac{-4\pi N_0}{3\ln(1-\phi)}}\,\Delta T\left(1-\frac{\Delta T_n^{n+1}}{\Delta T^{n+1}}\right)$$

Thanks to Michael Haines at UTK for running simulations!

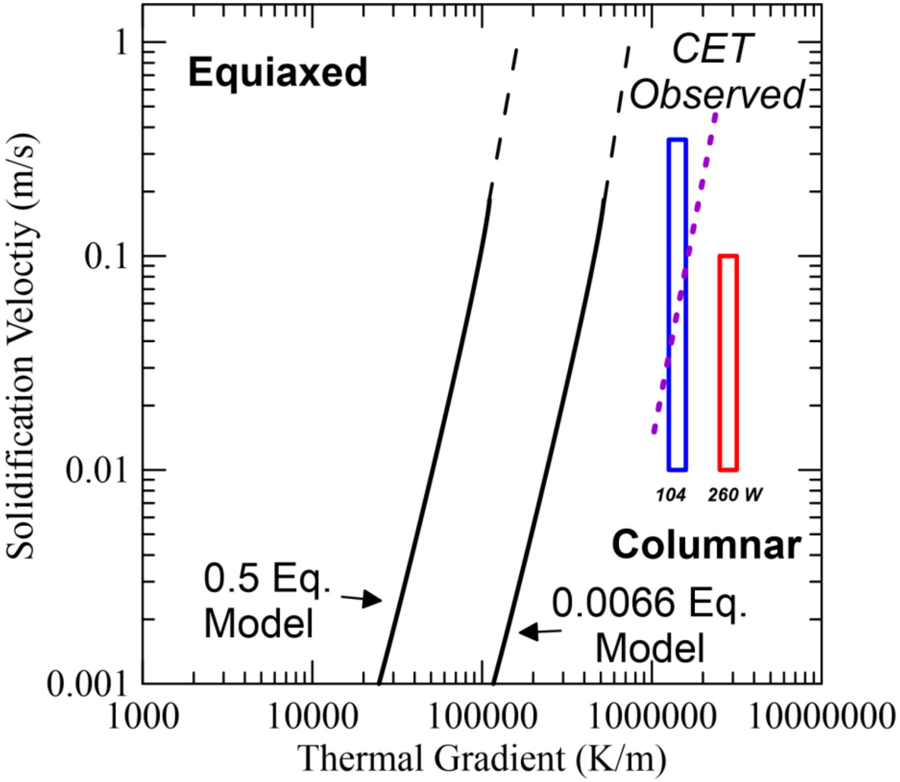
M. Haines, A. Plotowski, C.L. Frederick, E.J. Schwalback, S.S. Babu, "A sensitivity analysis of the columnar to equiaxed transition for Ni-based superalloys in electron beam additive manufacturing," *Computational Materials Science, 155*, 2018.

Comparison of CET Simulation to Experimental Results

R2-111



R4-100 & R4-110



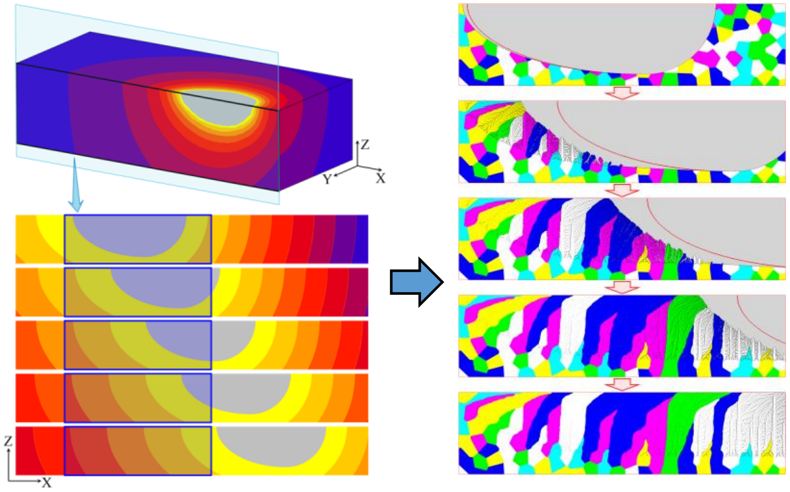
More sophisticated modeling is needed to get better estimates of thermal gradients, and more data (at higher velocities) is needed to calibrate model (particularly N_0) to experimental results

Preliminary Computational Simulations

Overall strategy

1) Perform 3D finite element thermal simulations

2) Use thermal field as boundary condition of 2D phase-field solidification simulations



F. Yu, et al., Int. J. Heat & Mass Transf. 142 (2019) 118450

Preliminary results

Cross section of a polycrystalline Al-2wt%Cu melt pool solidification

Perspectives:

- Massive parallelization on graphic cards (GPUs)
- Application to Ni-based alloys
- Explore laser parameters, resulting melt pool shape (and melt pool stability) on dendritic grain growth competition and grain texture selection

Courtesy D. Tourret (IMDEA Materials, Spain)

Calibrating models to experimental results on polycrystalline binary Al alloys, then will proceed to ternary Ni alloy single crystal samples

Summary

- **Use of AM Simulator at APS allows for in-situ characterization of solidification velocity at time scales relevant to laser powder bed fusion processing**
  - *Solidification velocities on the order of* 10^{-2} *– 1 m/s are measured*
  - *Higher velocities are measured at the end of solidification (at the top of the melt pool) and with lower laser power*
  - *Combination with ex-situ EBSD and SEM allows the evaluation of dendrite tip velocity from radiography data*
- **New grain orientations are observed at the end of solidification with lower laser powers for all conditions**
  - *New grains likely nucleate due to higher solidification velocities at top of pool inducing the CET (also observed during collapse of deep keyhole at higher power)*
  - *Laser power is the most significant parameter evaluated on spot melts compared to substrate orientation and composition*
- **Overall crystallographic orientation (texture) of spot melt is strongly influenced by substrate orientation**
  - *It is possible that single crystals with any orientation may be made with AM provided a suitably oriented starting block is used and the CET is not induced in the weld pools*

Continuing Work

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- **Evaluate intermediate laser powers for all conditions**
  - *Only lowest and highest powers for all conditions completed*
  - *All powers completed for R2-111*
- **Evaluate rasters and multiple spot melts**
- **Evaluate rasters at higher solidification velocities**
  - *During next beam time allotment, will generate samples with higher solidification velocities in order to have conditions with greater volume fractions of equiaxed grains*
- **Work on refining and calibrating CET model**
  - *Work with Michael Haines to see if algorithm can produce solutions at higher velocities*
  - *Collaborate to get more sophisticated heat transfer models to calculate thermal gradients*
  - *Include higher velocity samples with greater fractions of equiaxed grains to calibrate CET model*

Challenges & Opportunities

- **CET model is highly sensitive to nucleation site density which cannot be directly measured**
  - *Nucleation site density is back calculated from equiaxed grain fraction measurements for a given sample geometry/process and alloy.*
  - *Can the model be predictive? Is it useful outside of understanding CET in a particular sample geometry/alloy?*
  - *Should more focus go into more sophisticated models that include 2D effects and explicitly evaluate nucleation ahead of the advancing columnar front?*
- **General trends of how parameters influence the CET can enable better microstructure/texture control in AM**
  - *Is there more specific information/understanding that would be impactful to industry?*

Thank You!

Jonah Klemm-Toole

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Back Up Slides

Ex-Situ Scanning Electron Microscopy (SEM) – Top of Spot Melts

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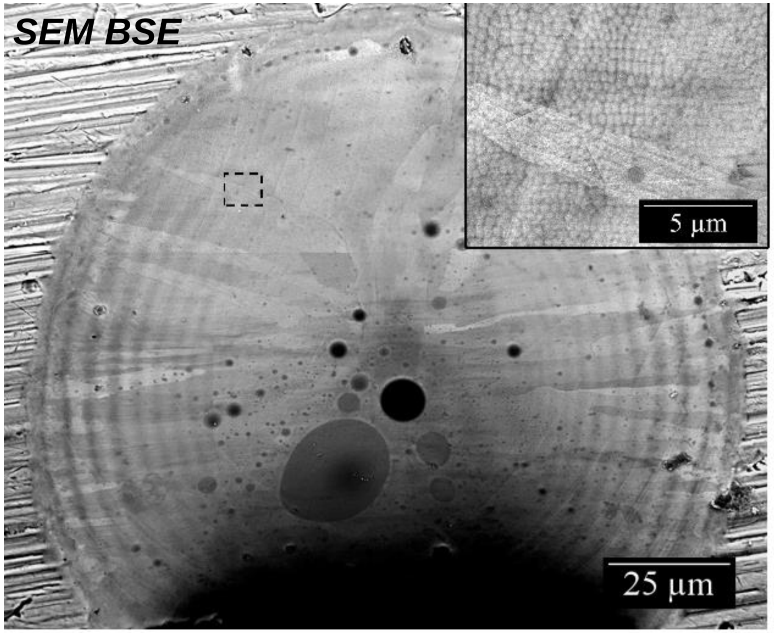
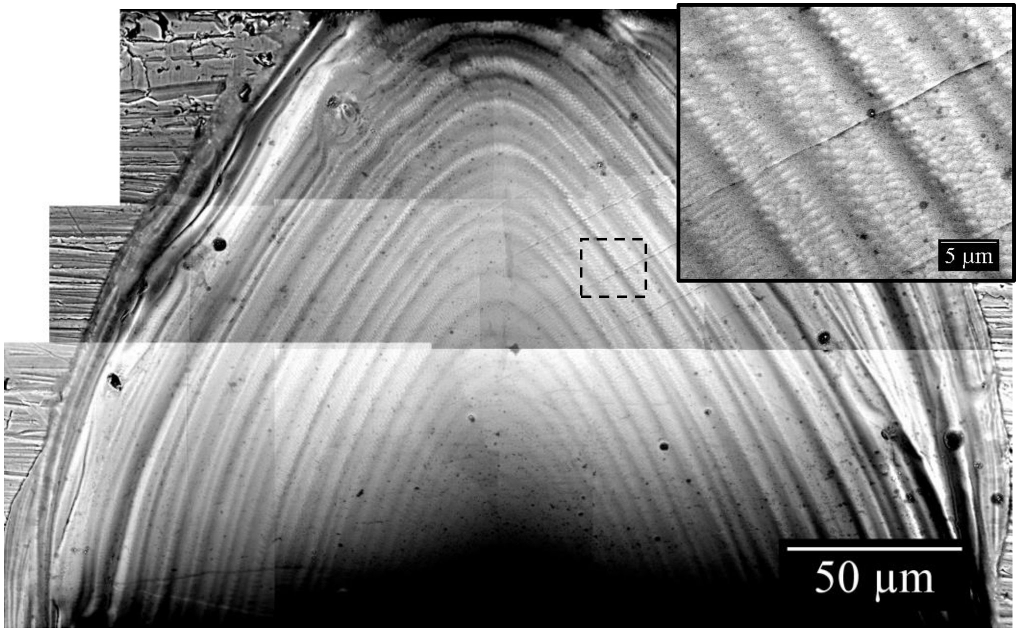
R2-111 106 W 1 ms



Cell/Dendrite Spacing ~ 340 nm +/- 25 nm

R2-111 260 W 1 ms



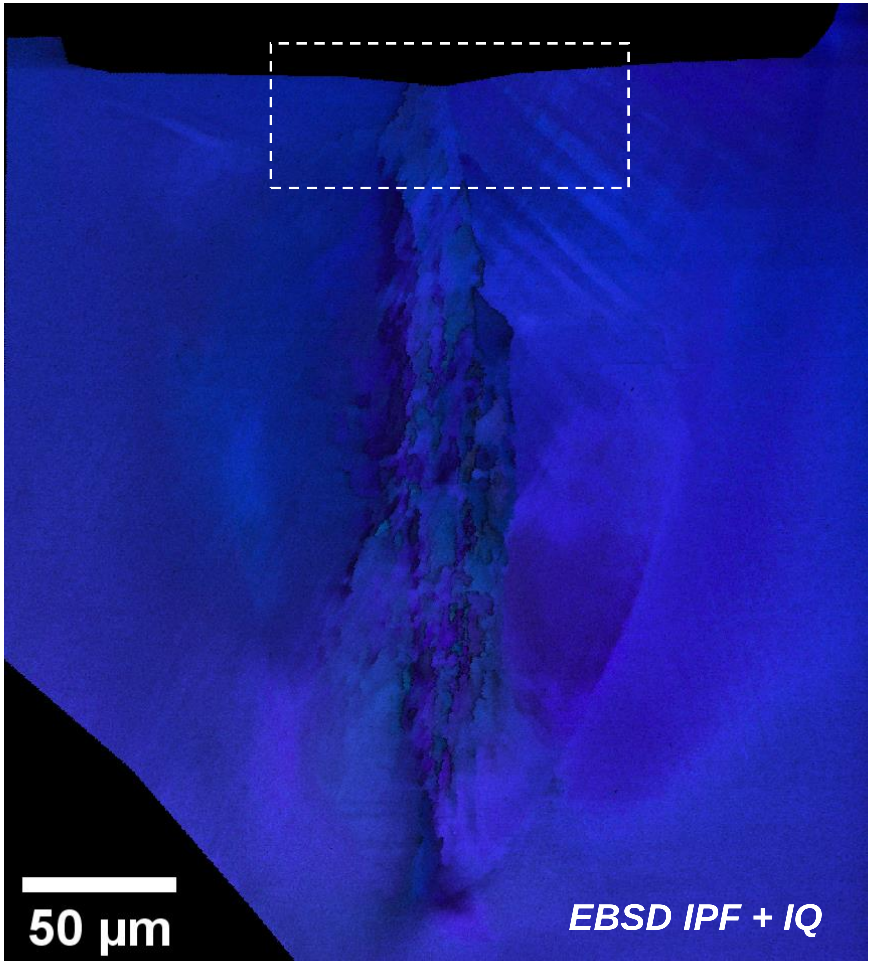
Cell/Dendrite Spacing ~ 900 nm +/- 75 nm

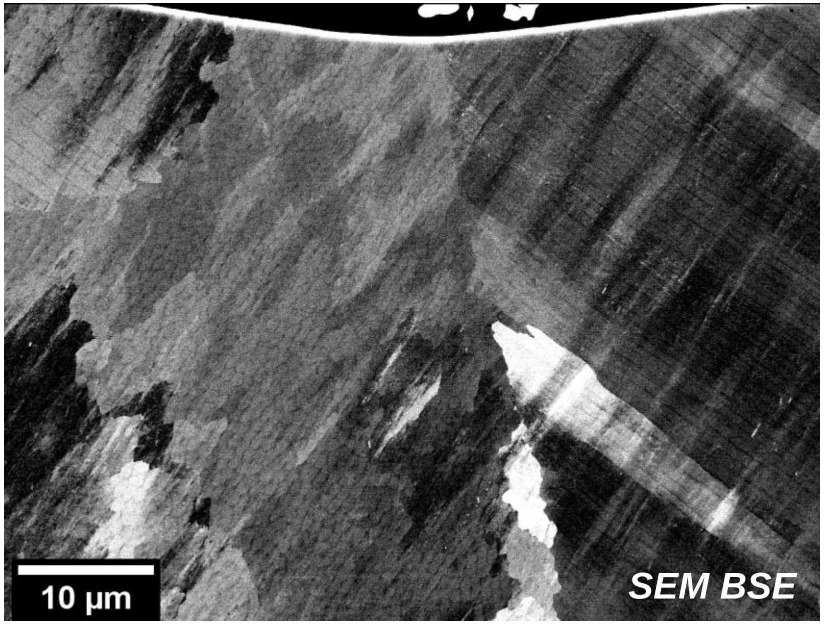
Finer Cell/Dendrite Spacing Indicates Higher Cooling Rate with Lower Laser Power

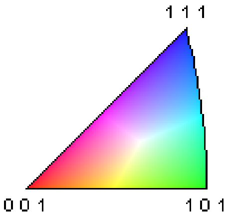
Different Cell/Dendrite Orientations Observed at Top of Lower Laser Power Spot Melt– Suggests Nucleation of Different Orientation (CET)

Growth Orientations of R2-111

R2-111 208 W







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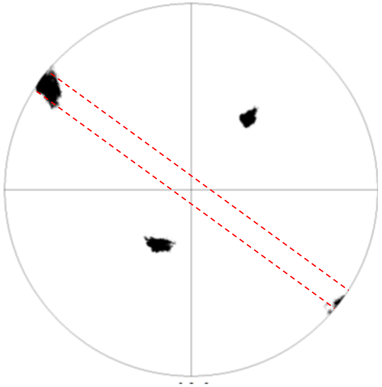
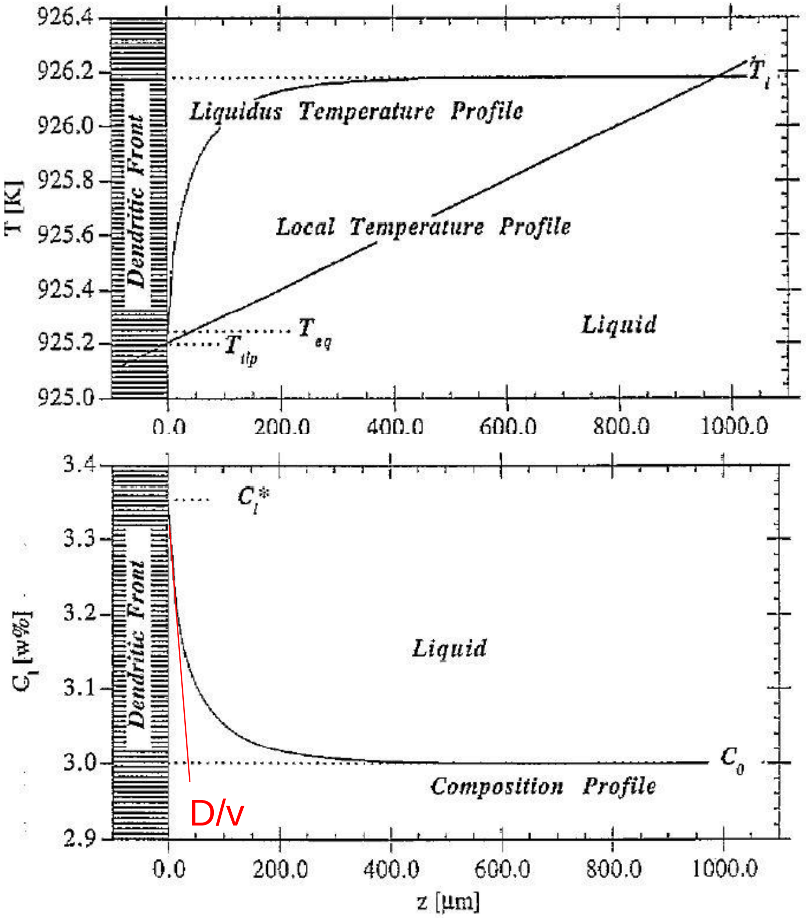
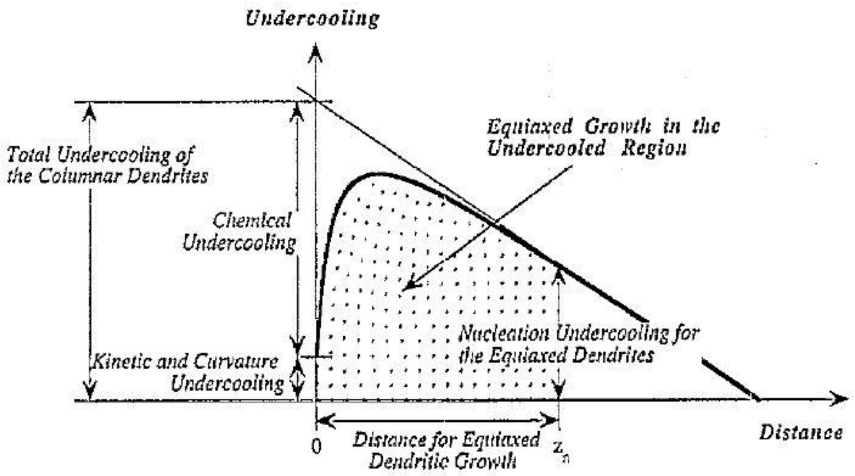


100 Pole Figure

Influence of Solidification Velocity & Composition





Higher velocities and solutes with lower diffusivities lead to greater undercooling in the liquid ahead of the advancing columnar front and increase the nucleation and growth rate of "equiaxed" grains, i.e. more likely to induce the CET.

M. Gaumann, R. Trivedi, and W. Kurz, "Nucleation ahead of the advancing interface in directional solidification," Materials Science and Engineering A226-228, 1997, pp. 763-769.

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Staff: *Jonah Klemm-Toole*

Faculty: *Amy Clarke and Kester Clarke*

Industrial Partners: *TBD*

Project Duration: *Feb 2019 – Feb 2021*

Achievement

- Determined that lower laser power leads to new grain nucleation due to higher solidification velocities inducing the columnar to equiaxed transition (CET)

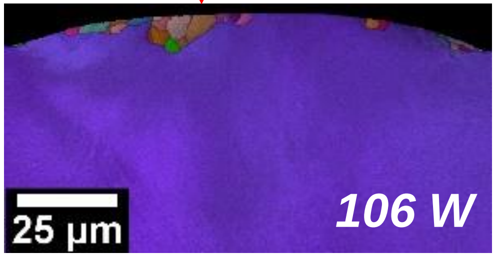
Significance and Impact

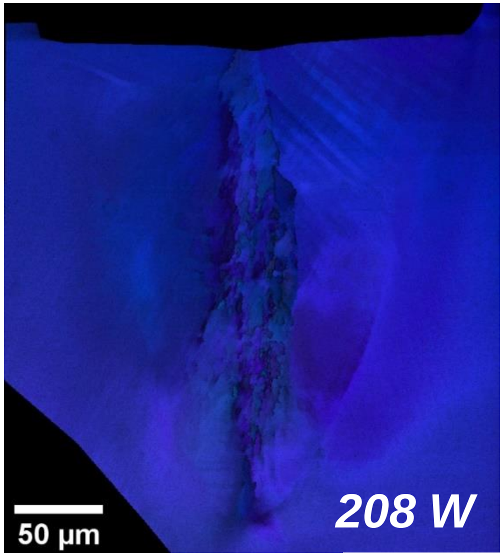
- A better understanding of how to control crystallographic texture and microstructure selection in AM can enable location and orientation specific property design

Research Details

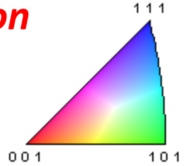
- Obtained in-situ radiography during laser melting and solidification at the Advanced Photon Source at Argonnne National Laboratory

New grain orientations





Substrate orientation maintained



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Program Goal

- Develop deeper fundamental understanding of crystallographic texture development in metal additive manufacturing

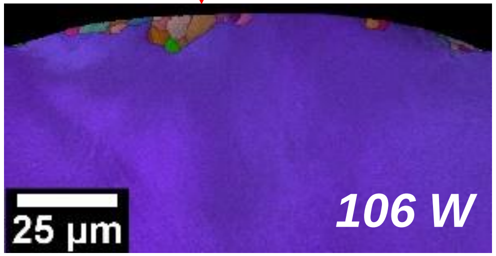
Approach

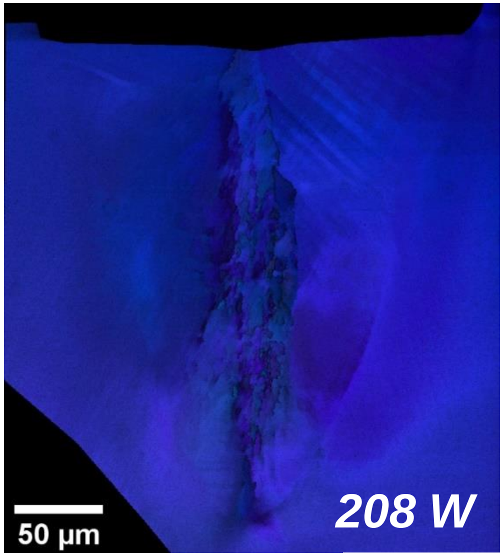
- Combine in-situ radiography during solidification and ex-situ EBSD and SEM to measure solidification velocity and relate solidification conditions to columnar to equiaxed transition models

Benefits

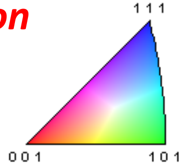
- Control of columnar to equiaxed transition can enable the production of highly oriented “single crystals” of any orientation with AM

New grain orientations





Substrate orientation maintained



Build Direction