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

Spring Meeting
April 7th – 9th, 2020

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Industrial Mentors: TBD

Multidisciplinary University Research Initiative (MURI)

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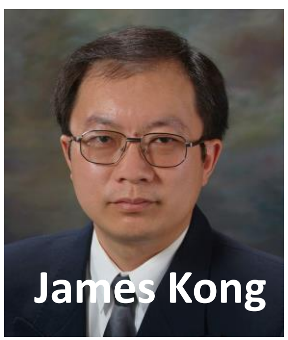

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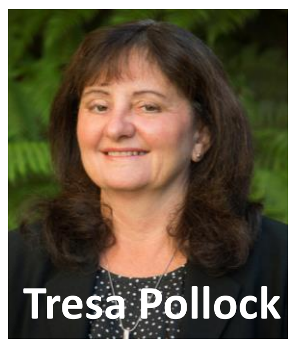

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Overview

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

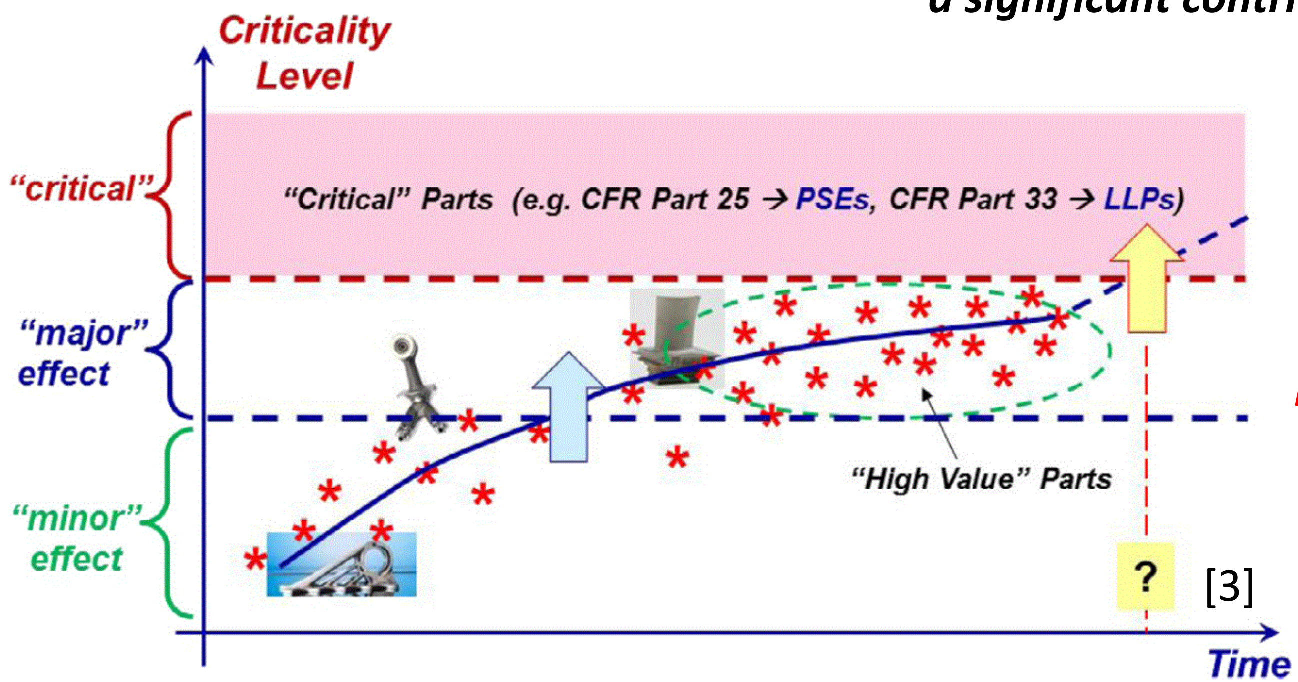
Motivation

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.***



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

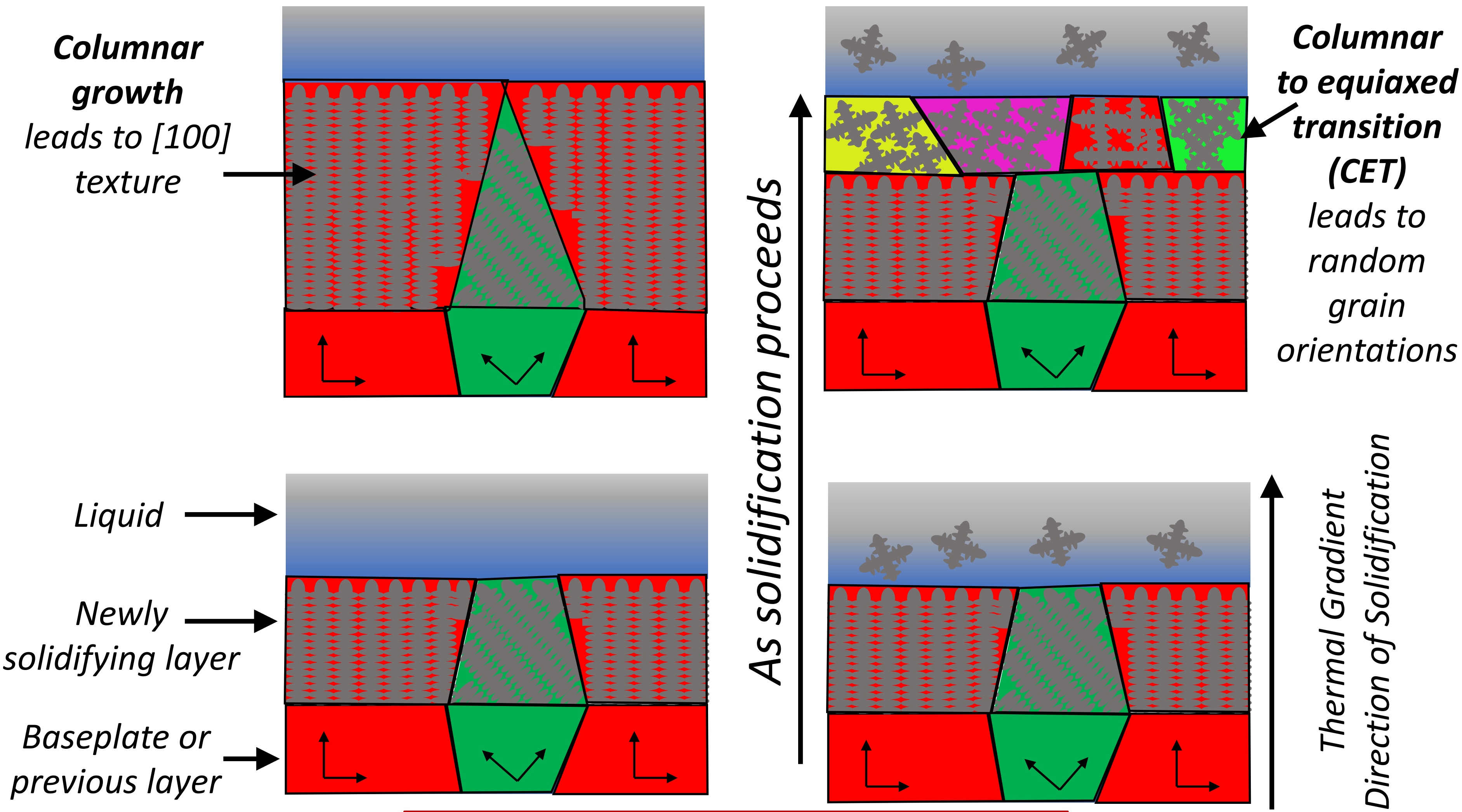
[2] M. Seifi *et al*., *JOM,* vol. 69, no. 3, pp. 439-455, 2017.

[3] M. Gorelik, *Int. J. Fat*, vol. 94, pp. 168-177, 2017.

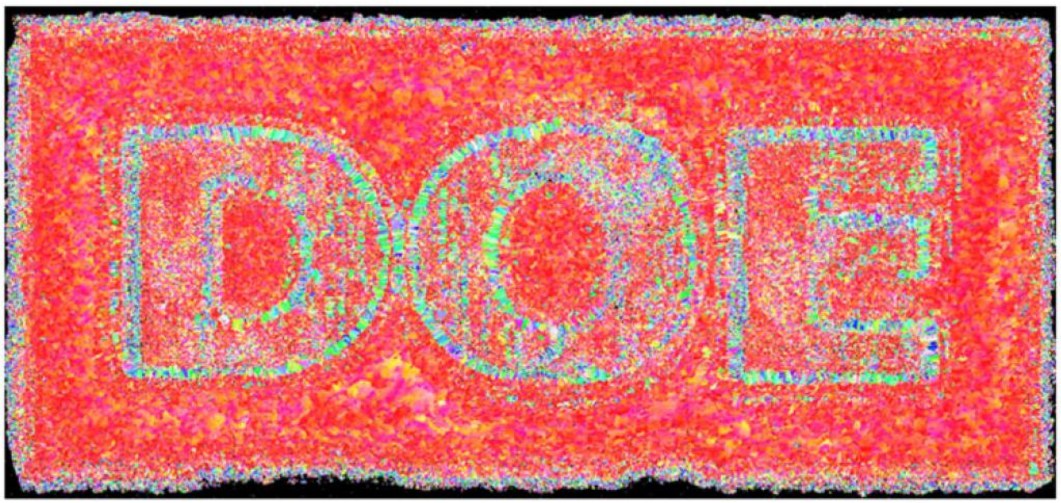
Texture Formation/Disruption in Additive Manufacturing

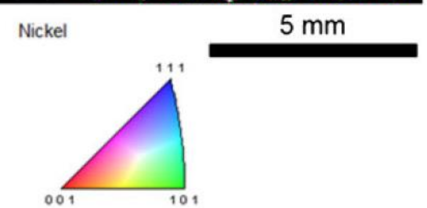
Texture Formation

Texture Disruption

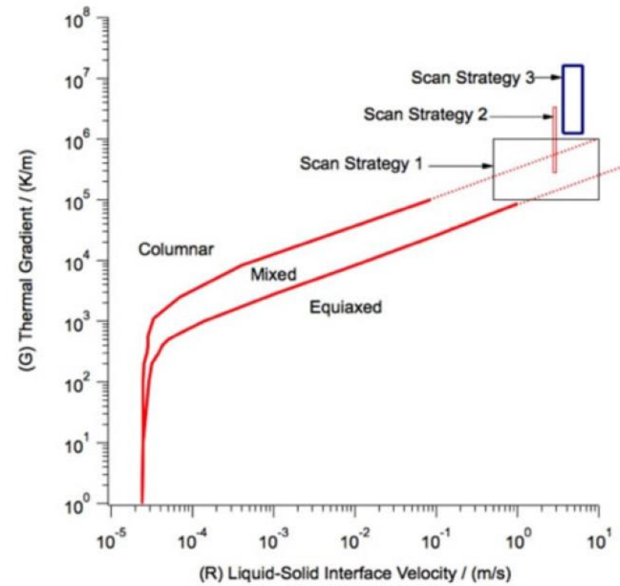


Controlling Texture in AM





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

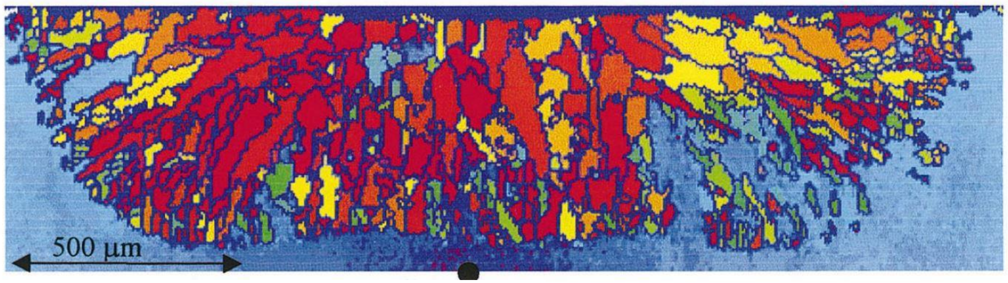
Smaller gradients and higher velocities induce the columnar to equiaxed transition (CET) and lead to random orientations

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.

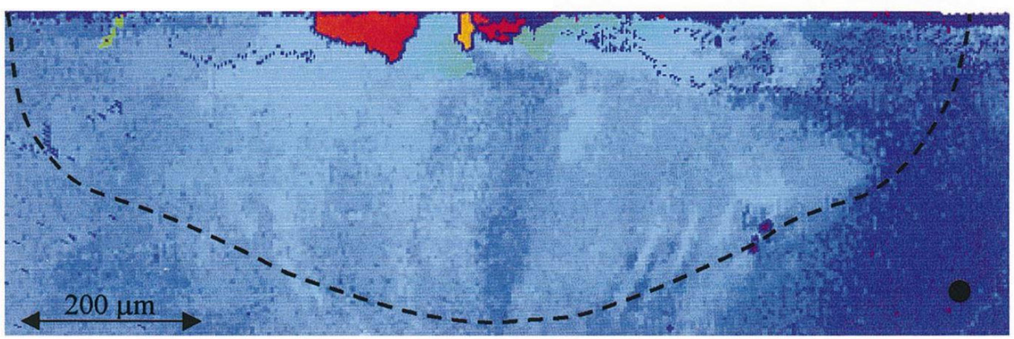
Controlling Texture During Laser Welding of Single Crystals

New grain orientations nucleated

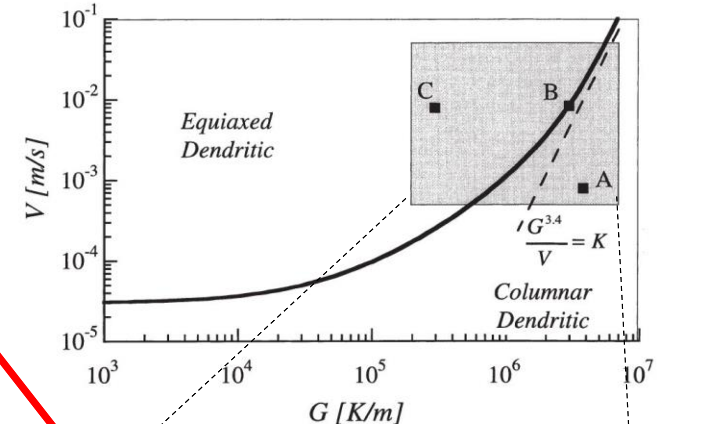
CMSX-4

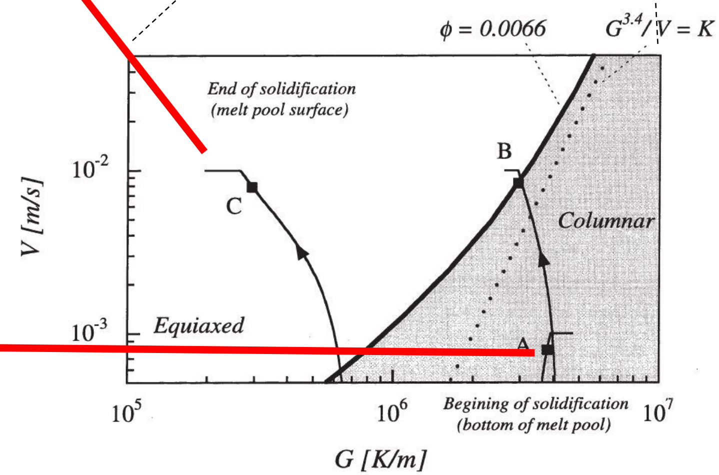


Most of weld maintains [100] orientation of substrate



G and V calculated from heat transfer models





Gradient decreases and velocity increases from bottom to top of melt pool induces 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**
- **Evaluate the influences of several parameters on the CET and subsequent crystallographic orientations after solidification**
  - *Spot and raster melt strategies*
  - *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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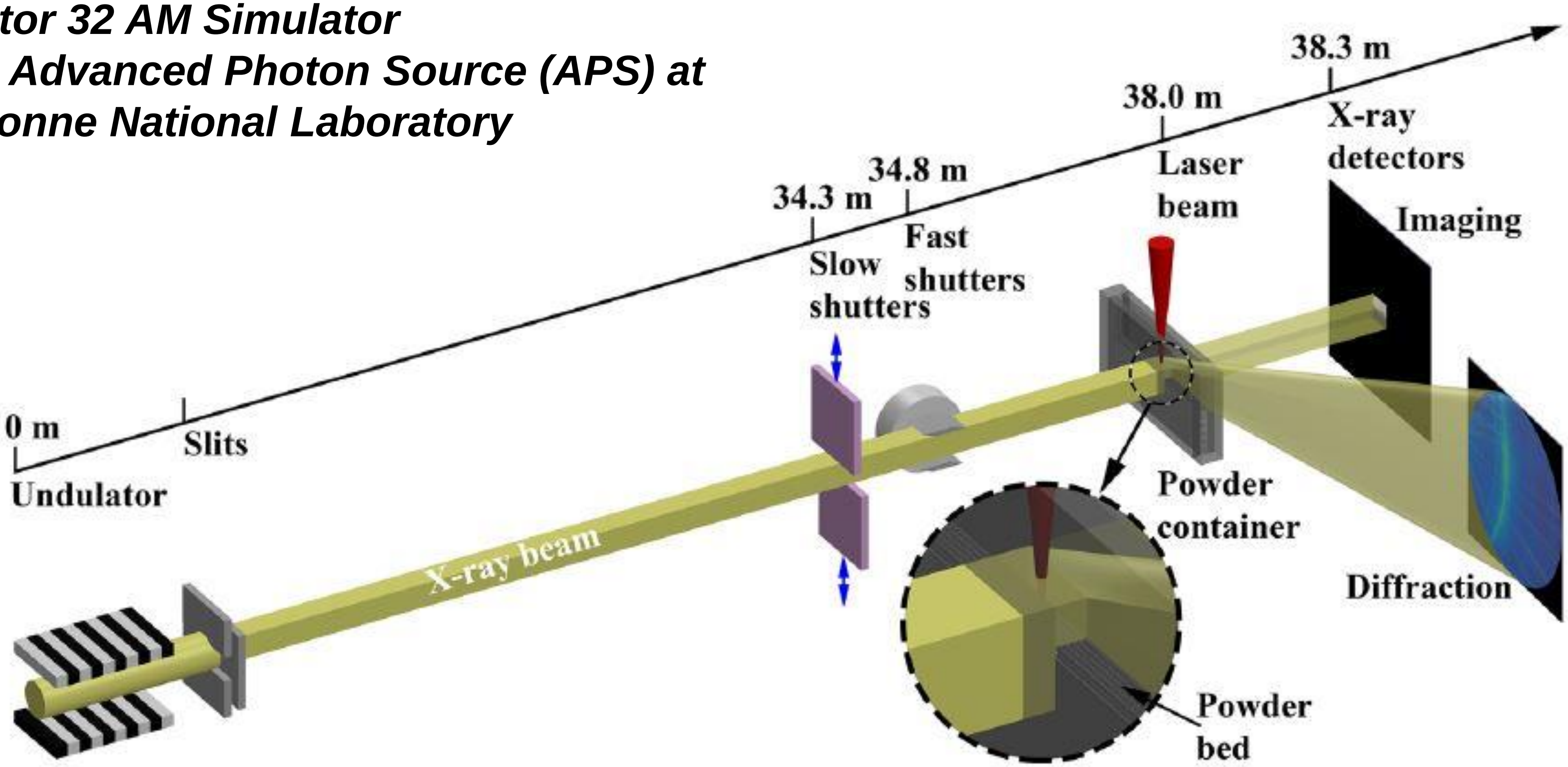
Sector 32 AM Simulator
The Advanced Photon Source (APS) at Argonne National Laboratory



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

- **Melting Parameters**
  - 106, 156, 208, 260 W for 1 ms (spot), 156 W 0.5 m/s (raster)
- **Compositions**
  - Ni – 1.9Mo – 6.6 Al
  - Ni – 22.2Mo – 2.8 Al

Same γ' volume fraction
Provided by T. Pollock (UCSB)
(Fahrmann *et al*. Mat Sci Eng A 1999)

- **Single Crystal Orientations**

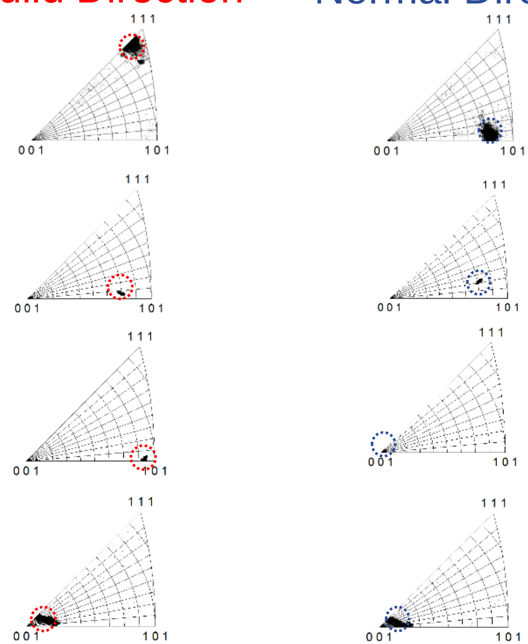
Build Direction | Normal Direction

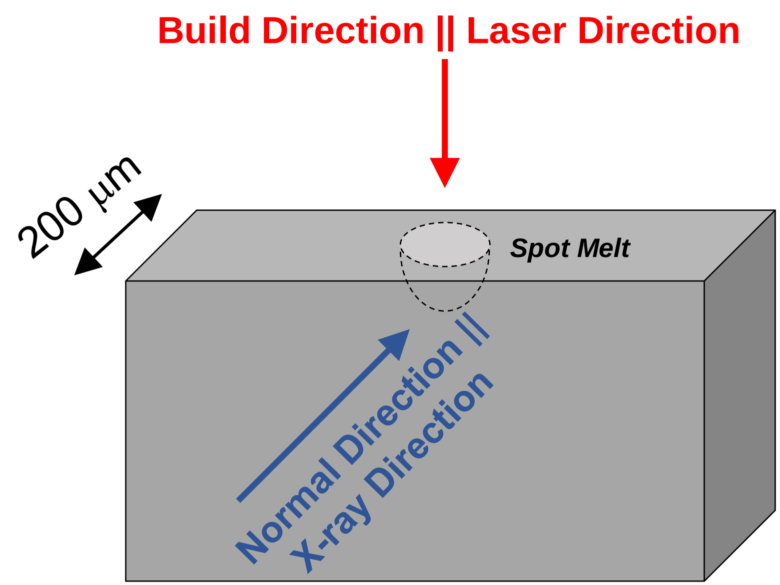
Ni-1.9Mo-6.6Al-111

Ni-1.9Mo-6.6Al-110

Ni-22.2Mo-2.8Al-110

Ni-22.2Mo-2.8Al-100





EBSD + SEM to Determine Growth Orientations

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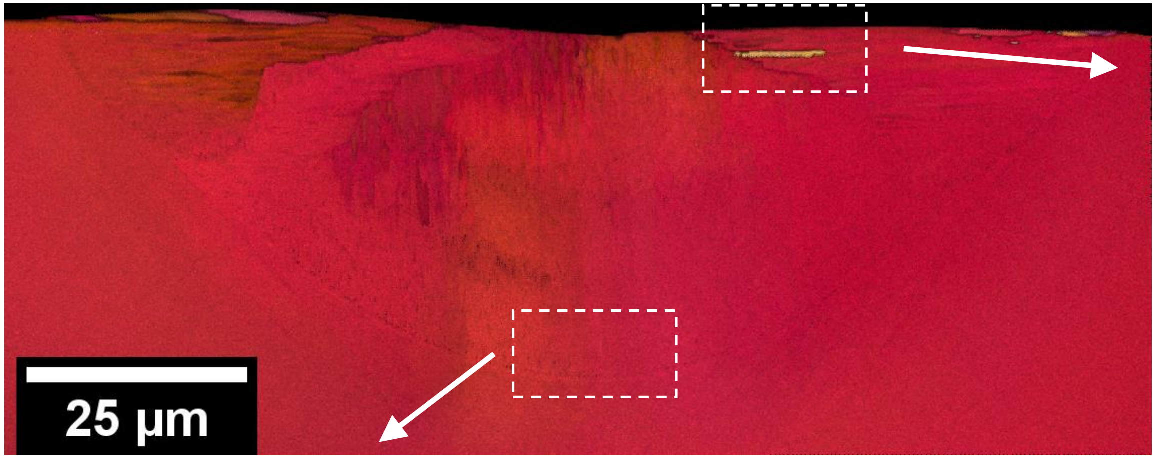
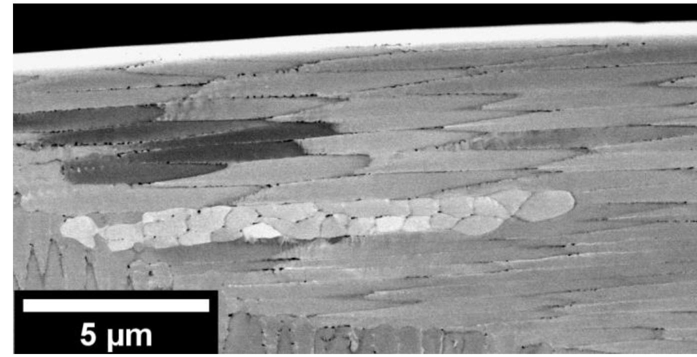
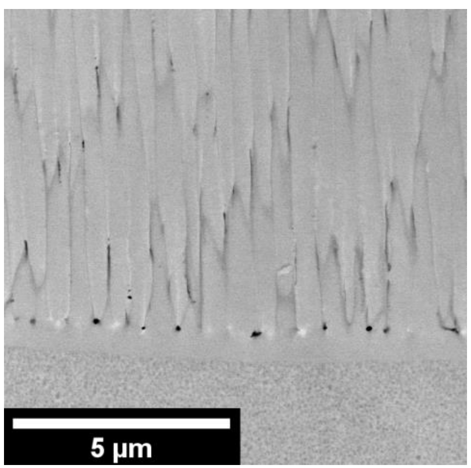
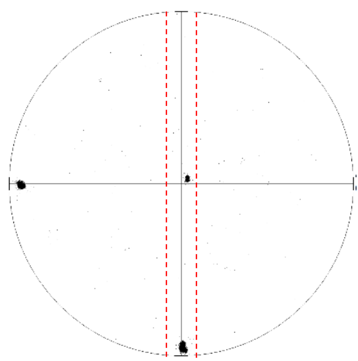
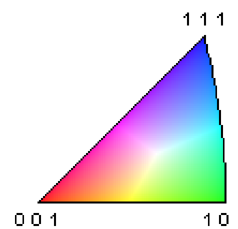




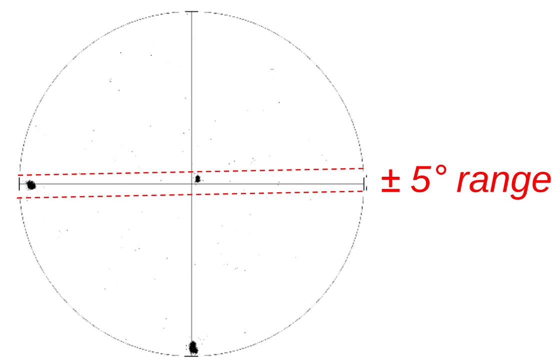




100 Pole Figure



Build Direction

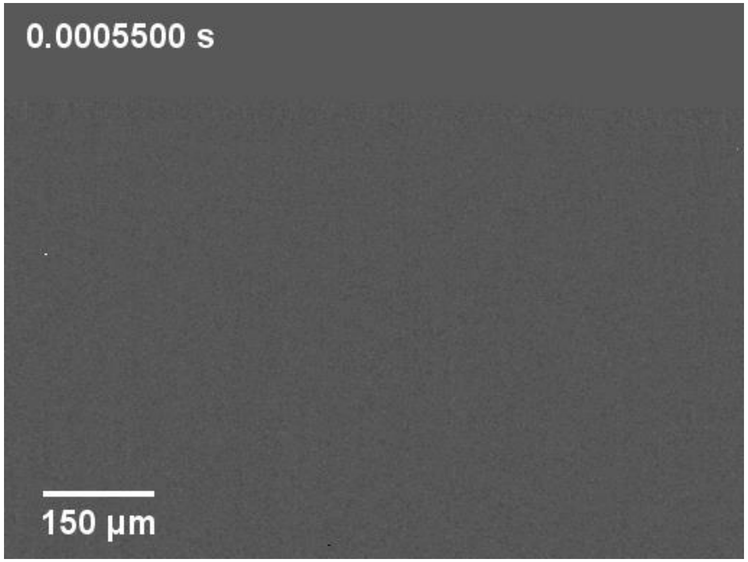


100 Pole Figure

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

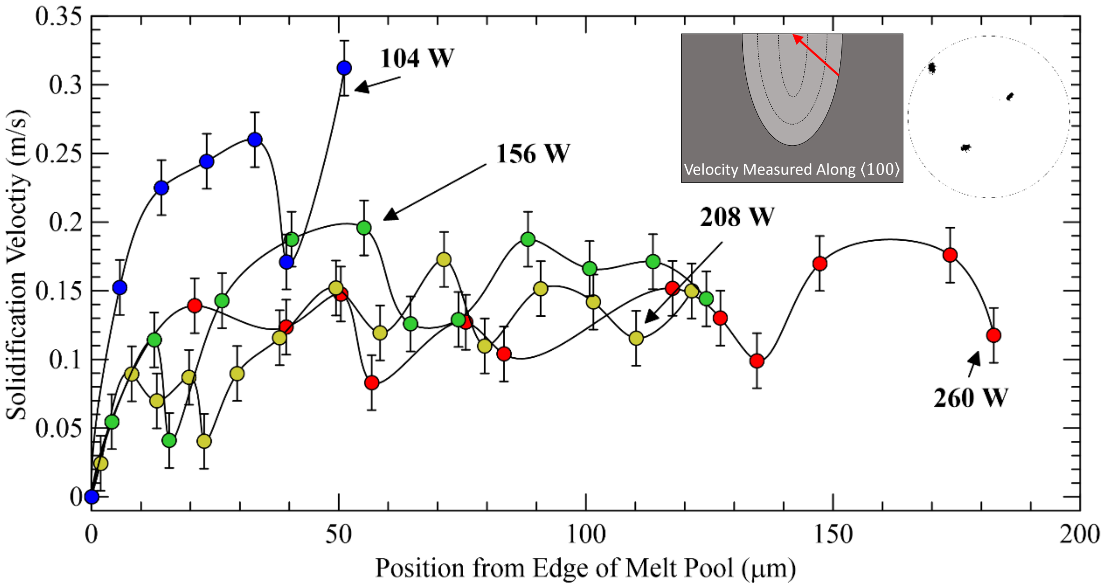
Analysis of In-Situ Radiography

Radiography of Ni-1.9Mo-6.6Al-111 260 W 1 ms



Thanks to Gus Becker at Mines for ImageJ scripts!

Solidification Velocity Measurements for Ni-1.9Mo-6.6Al-111



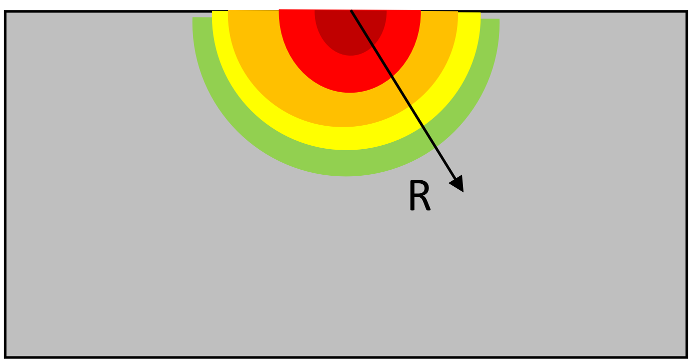
Solidification velocity increases from bottom to top of pool (confirming prediction from heat transfer models)

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

Simple Conduction Heat Transfer Models

Spot Melts

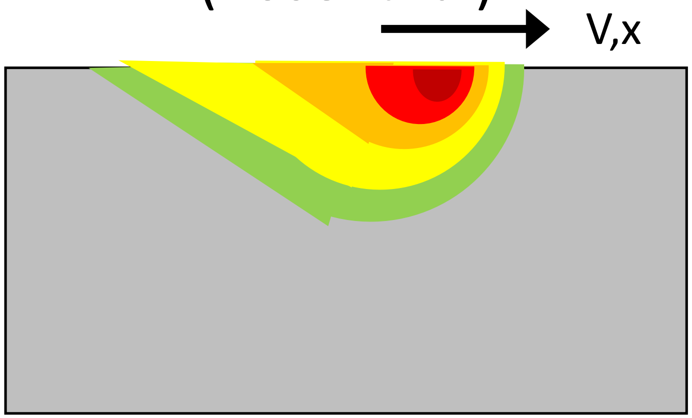
Instantaneous Stationary Point Heat Source in a Semi-infinite Body



$$T - T_0 = \frac{\beta Q}{\rho C (4\pi\alpha t)^{1.5}} e^{\left(\frac{R^2}{4\alpha t}\right)}$$

Rasters

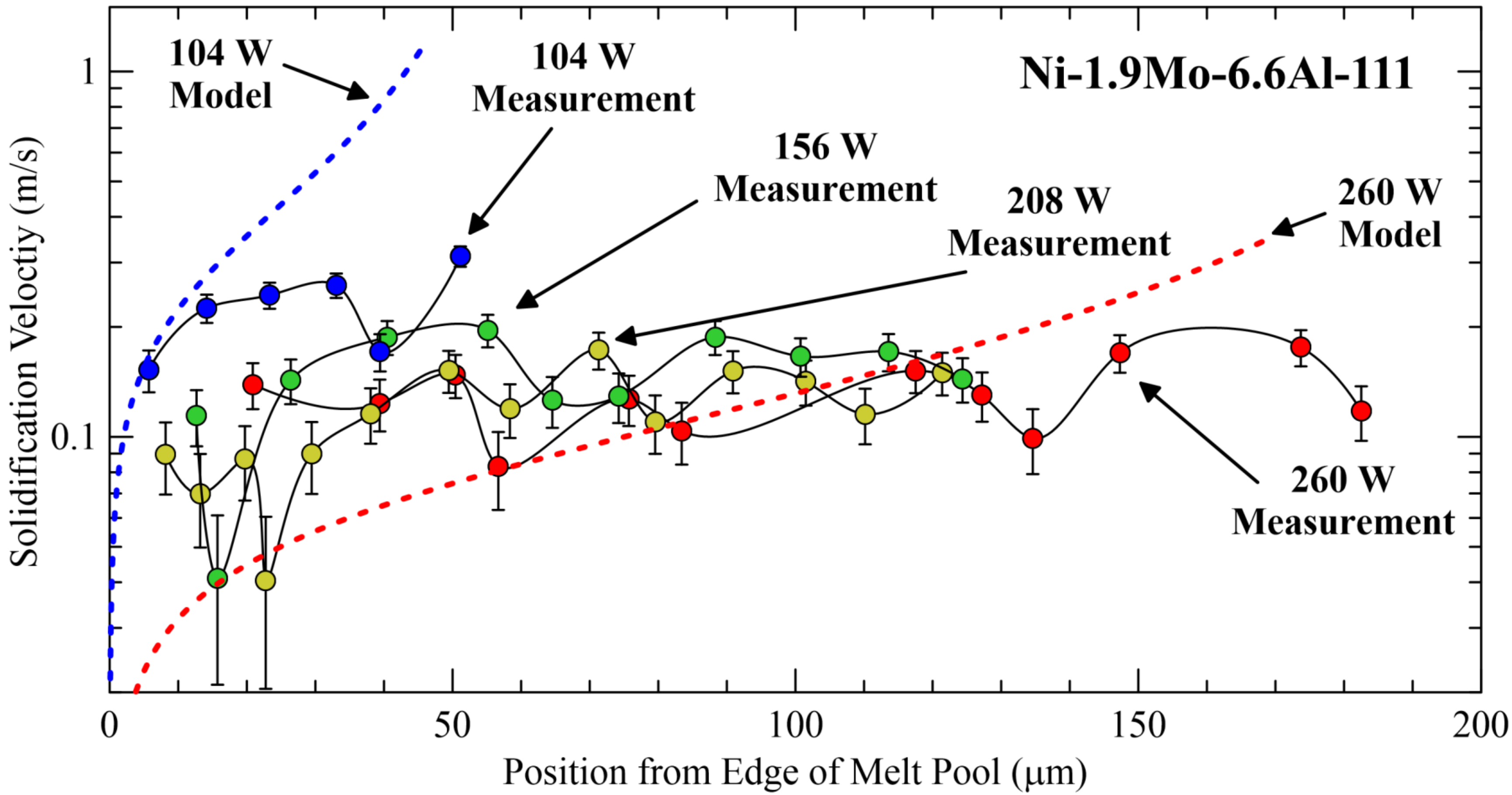
Moving Point Heat Source in an Semi-infinite Body (Rosenthal)



$$T - T_0 = \frac{\beta P}{2\pi k R} e^{\left(-\frac{V(R+x)}{2\alpha}\right)}$$

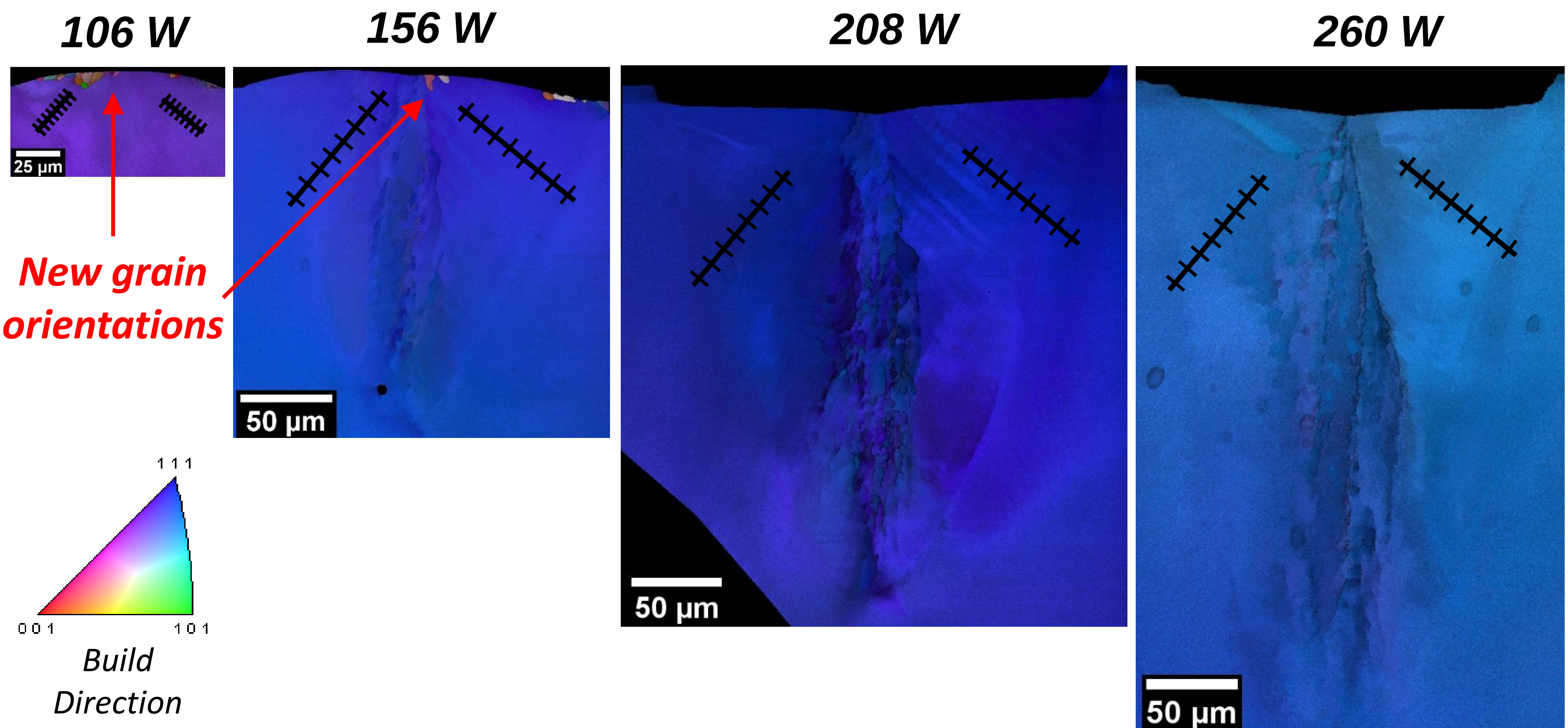
The simple heat transfer models do not account for actual specimen dimensions, laser energy distribution, convection, or latent heat of fusion

Comparison of Heat Transfer Models to Measurements – Spot Melts



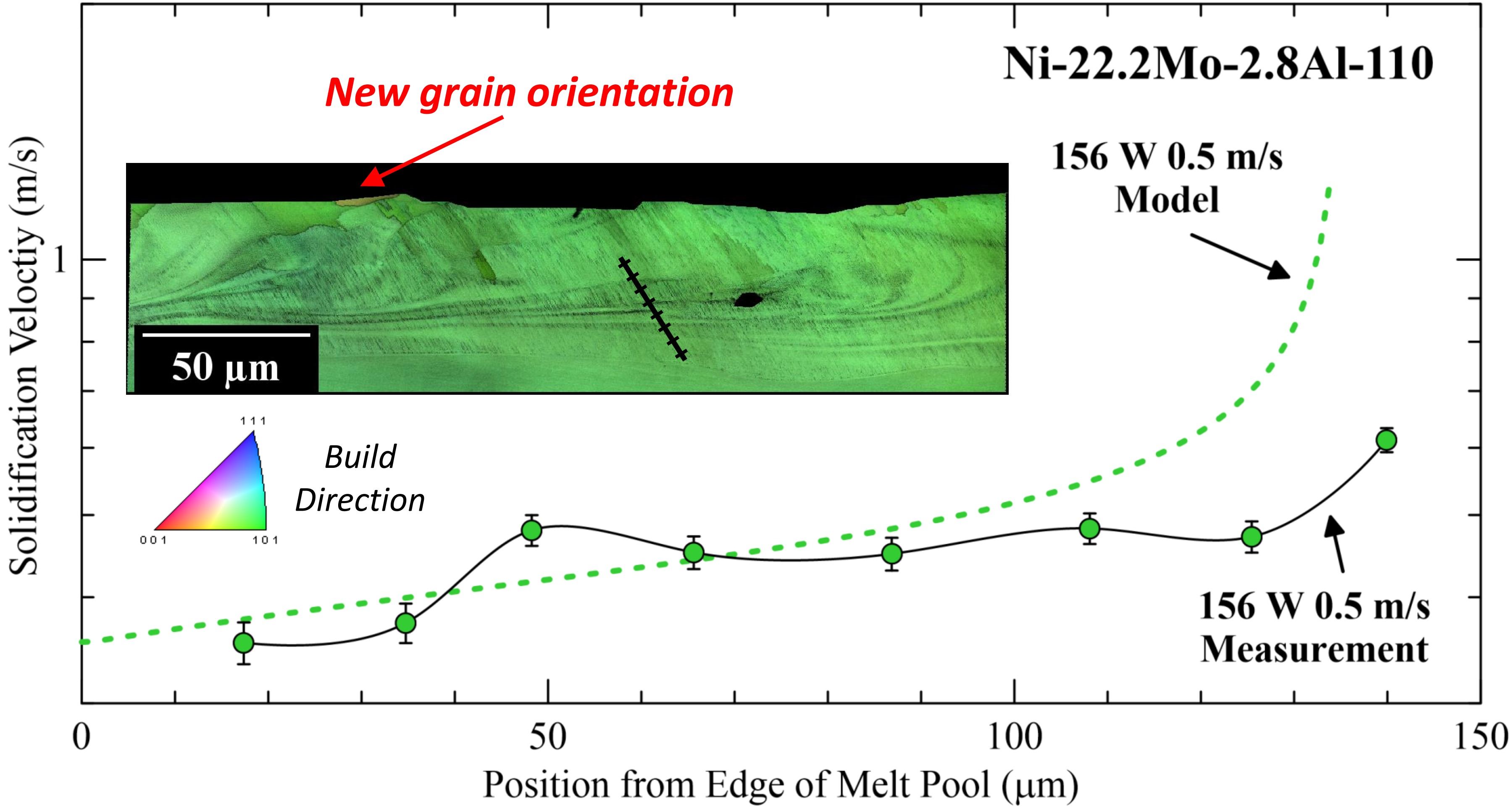
The simple heat transfer model for spot melts qualitatively predict trends, but it over estimates solidification velocity likely due to infinite body assumption

EBSD of Ni-1.9Mo-6.6Al-111 Spot Melts



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

Solidification Velocity and EBSD
Ni-22.2Mo-2.8Al-110 Raster



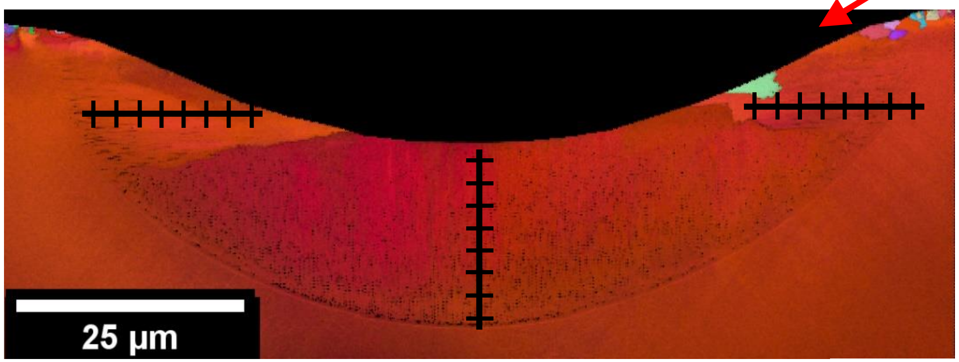
Better agreement between model and measurement for raster, and higher solidification velocities are associated with new grains at the top of the pool

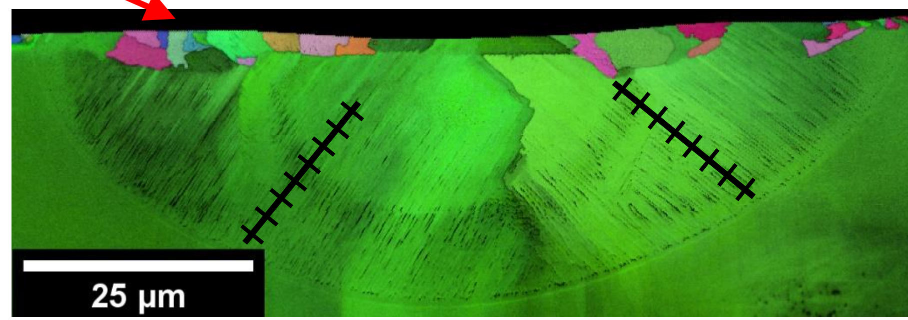
Influence of Substrate Orientation

New grain orientations (CET)

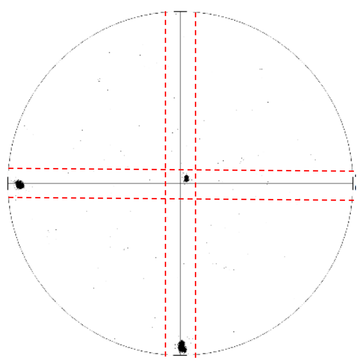
Ni-22.2Mo-2.8Al-100 106 W

Ni-22.0Mo-2.8Al-110 106 W

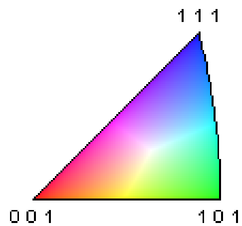




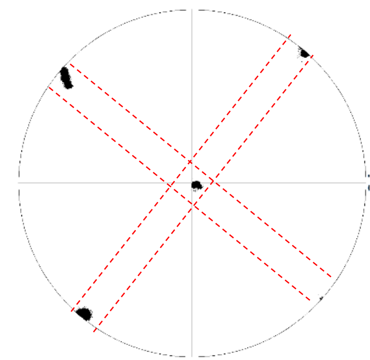
100 Pole Figure w/ traces of growth orientations



± 5° range



Build Direction



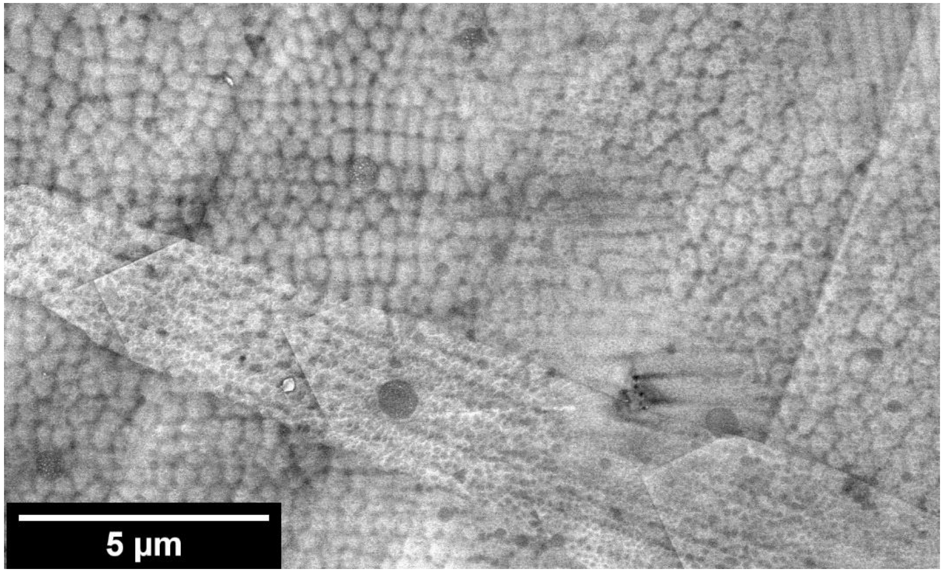
± 5° range

100 Pole Figure w/ traces of growth orientations

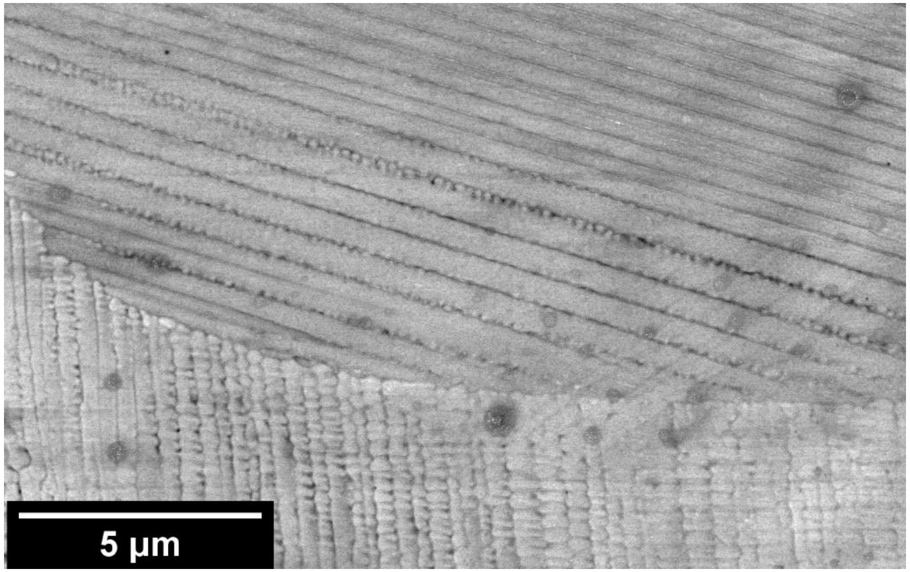
Both substrate orientations show growth parallel to ⟨100⟩ directions indicating dendritic solidification (observed in all conditions)

Influence of Composition on Microstructure Development

_Ni-1.9Mo-6.6Al-110 106 W_



_Ni-22.2Mo-2.8Al-110 106 W_



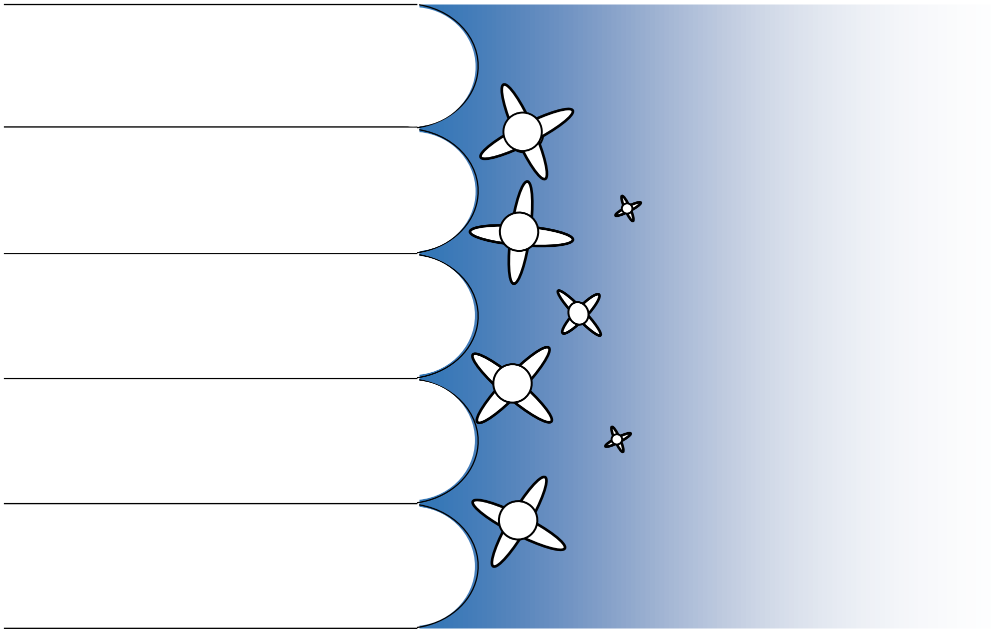
Secondary arms NOT observed

Secondary arms observable

Ni-22.2Mo-2.8Al (higher Mo content) shows well developed secondary dendrite arms, whereas no secondary arms are observed in Ni-1.9Mo-6.6Al (lower Mo content)

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

Columnar to Equiaxed Transition (CET) 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**

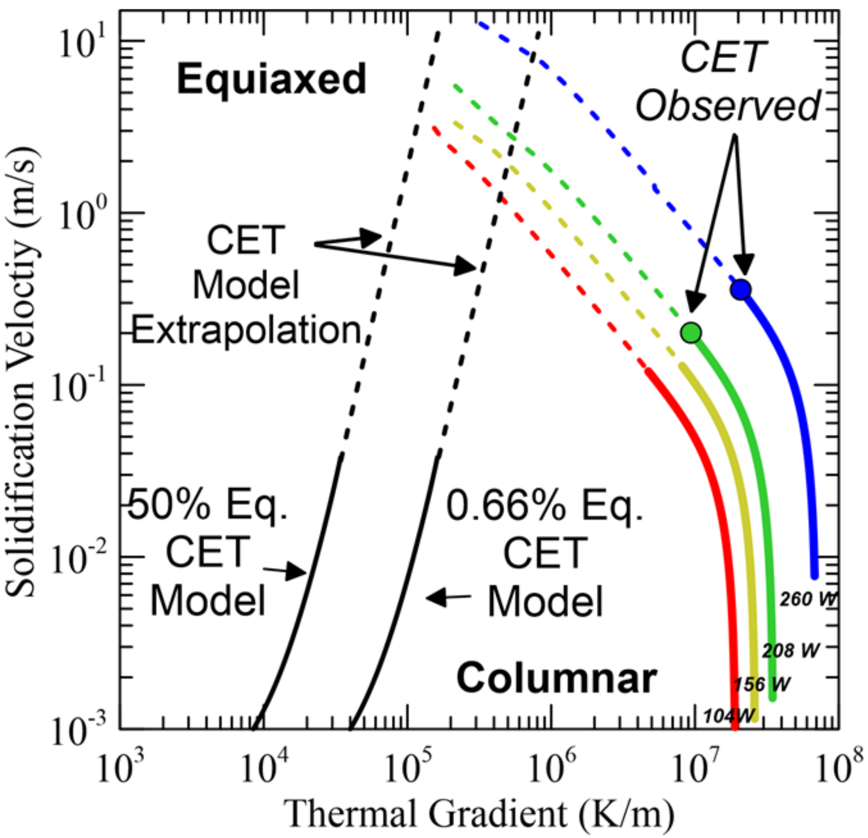
$$G = \frac{1}{n+1}\sqrt[3]{\frac{-4\pi N_0}{3\ln(1-\phi)}}\,\Delta T\left(1-\frac{\Delta T_n^{n+1}}{\Delta T^{n+1}}\right)$$

Thanks to Michael Haines at UTK for running simulations!

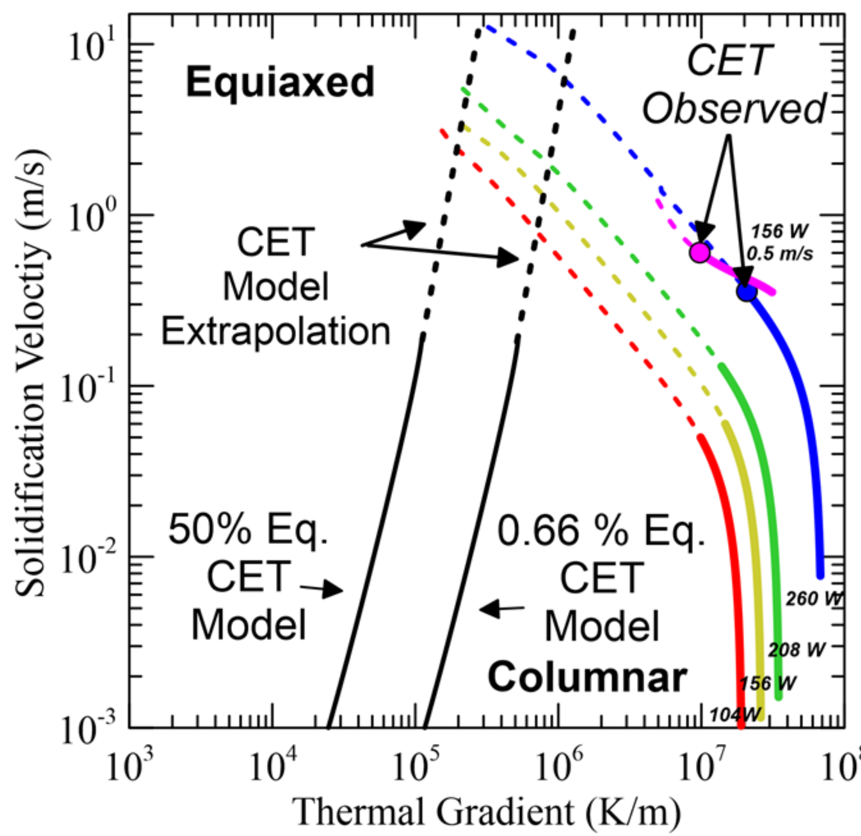
M. Haines, A. Plotowski, C.L. Frederick, E.J. Schwalback, S.S. Babu, "A sensitivity analysis of the columnar to equiaxed transition for Ni-based superalloys in electron beam additive manufacturing," *Computational Materials Science, 155*, 2018.

Comparison of CET Simulation to Experimental Results

Ni-1.9Mo-6.6Al-111



Ni-22.2Mo-2.8Al-110



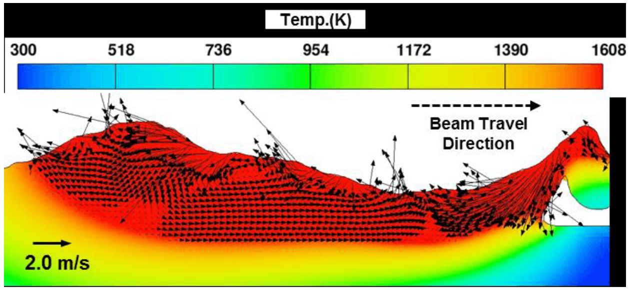
More sophisticated modeling is needed to get better estimates of thermal gradients, and more data (at higher velocities) is needed to calibrate model (particularly N_0) to experimental results

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*
  - *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*
  - ***Laser power is the most significant parameter evaluated** on spot melts compared to substrate orientation and composition.*
- **The simple heat transfer model used to simulate rasters better predicts solidification velocity compared to the model used to simulate spot melts**
  - *More sophisticated modeling is needed to get better estimates of thermal gradients in order to calibrate CET models*

Continuing Work

- **Perform more sophisticated simulations to get better estimates of thermal gradients**
  - *Realistic boundary conditions*
  - *Convection*
  - *Laser power distribution*

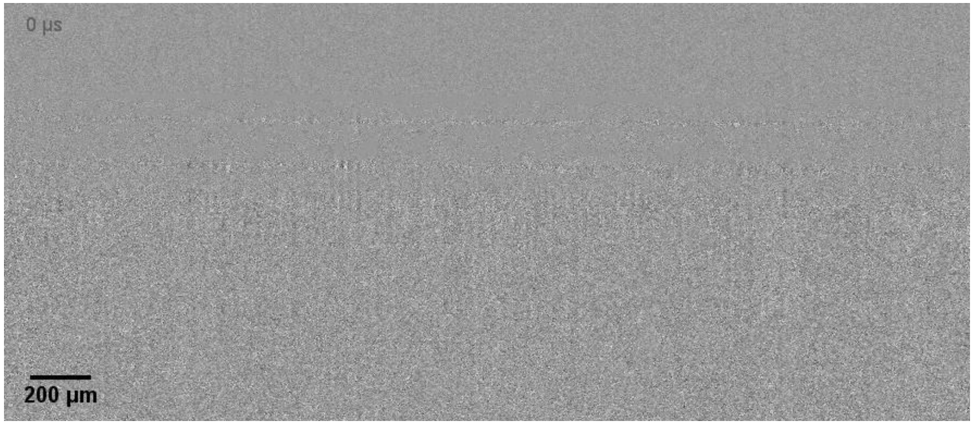


FLOW-3D

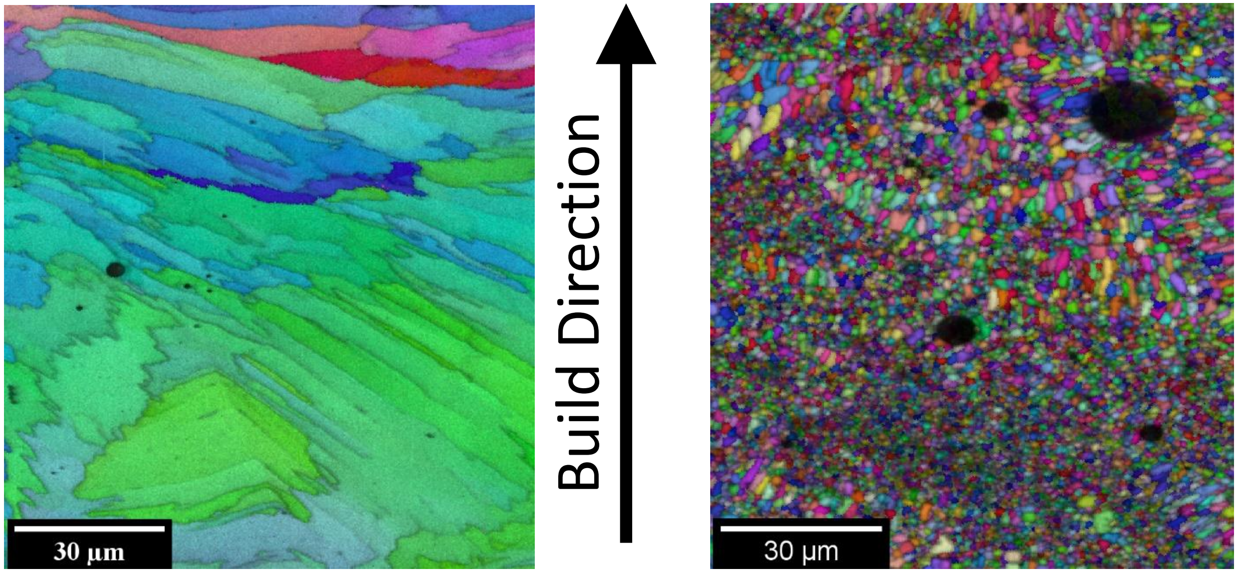
https://www.flow3d.com/resources/case-studies/

- **Evaluate rasters at higher solidification velocities (Brian Rodgers)**
  - *Higher velocity rasters are expected to result in higher fractions of equiaxed grains and provide valuable data for the CET model*
- **Calibrate CET model**
  - *Combine radiography, EBSD, and thermal gradient calculations to calibrate nucleation site density in the CET model*
- **Write Journal Articles**
- **Begin work on HRL aluminum samples from 2019 APS Beamtime**

Continuing Work – APS Experiments on HRL Inoculated Al



In-situ radiography of pure Al powder on pure Al build



Pure Al HRL Build – EBSD IPF + IQ **Inoculated Al HRL Build – EBSD IPF + IQ**

Preliminary EBSD on HRL builds to confirm sample preparation techniques

Challenges & Opportunities

- **What is the correct level of sophistication for modeling APS experiments?**
  - *The **geometry** of the samples results in almost 2D heat transfer – very different from semi-infinite plate - **can be addressed with FLOW-3D***
  - ***Convective flow** is observable in radiography, so effects of convection need to be accounted for in heat transfer simulations – **can be addressed with FLOW-3D***
  - *There are **no thermo-physical or thermo-chemical properties** for the alloys under investigation, so must use values for commercial alloys like IN718. **How much error does this cause?***
- **Would a sensitivity study be of interest to the sponsors?**
  - *The relative influences of geometry, convection, laser characteristics, and material properties can be evaluated on thermal conditions to determine most important factors.*

Thank You!

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Acknowledgements

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- Use of the Advanced Photon Source, an Office of Science User Facility operated for the U.S. DOE Office of Science by Argonne National Laboratory, was supported by the U.S. DOE under Contract No. DE-AC02-06CH11357; X-ray data were collected at the Sector 32-ID-B beamline.

Effects of Long Shifts at the Beamline….

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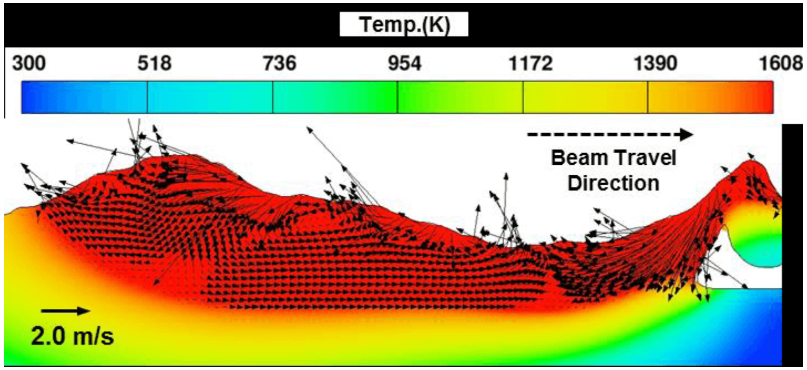
Back Up Slides

Further Modeling and Simulations

Overall strategy

1) Perform 3D conduction plus convection thermal simulations using FLOW-3D

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



https://www.flow3d.com/resources/case-studies/

Preliminary results

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

Perspectives:

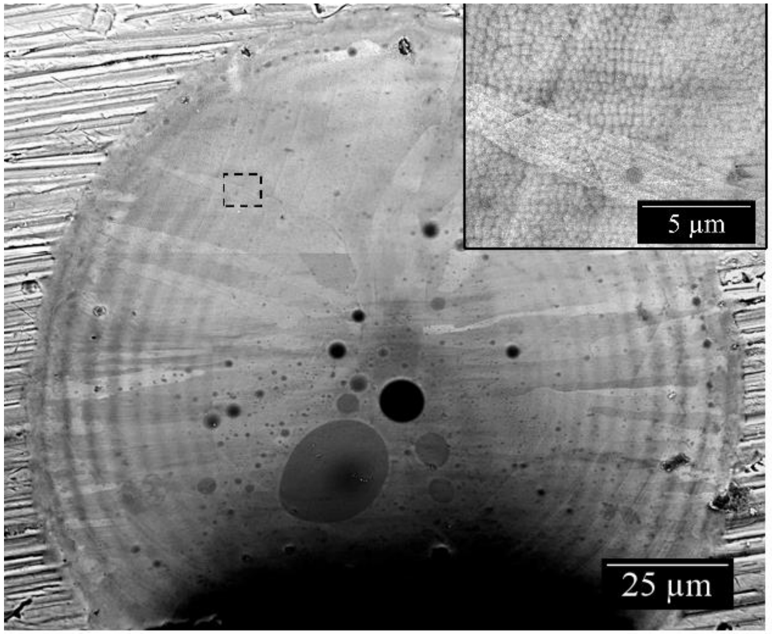
- Massive parallelization on graphic cards (GPUs)
- Application to Ni-based alloys
- Explore laser parameters, resulting melt pool shape (and melt pool stability) on dendritic grain growth competition and grain texture selection

Courtesy D. Tourret (IMDEA Materials, Spain)

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

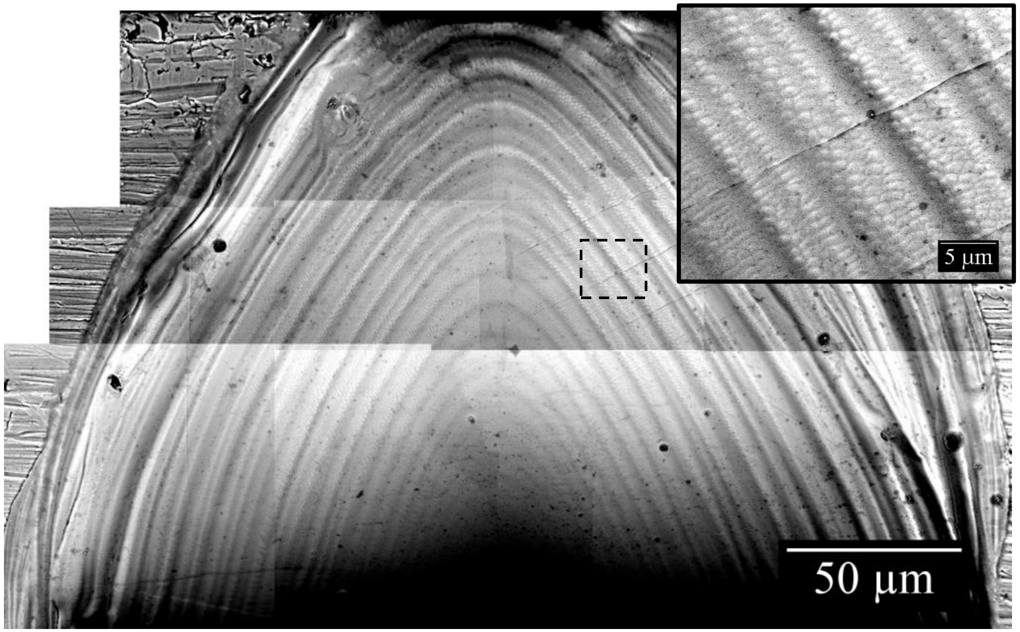
Ex-Situ Scanning Electron Microscopy (SEM) – Top of Spot Melts

R2-111 106 W 1 ms



Dendrite Spacing ~ 340 nm +/- 25 nm

R2-111 260 W 1 ms

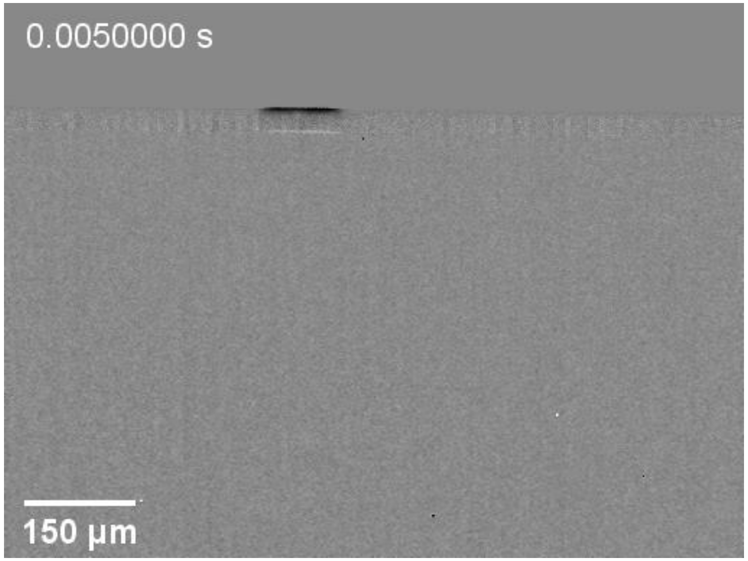


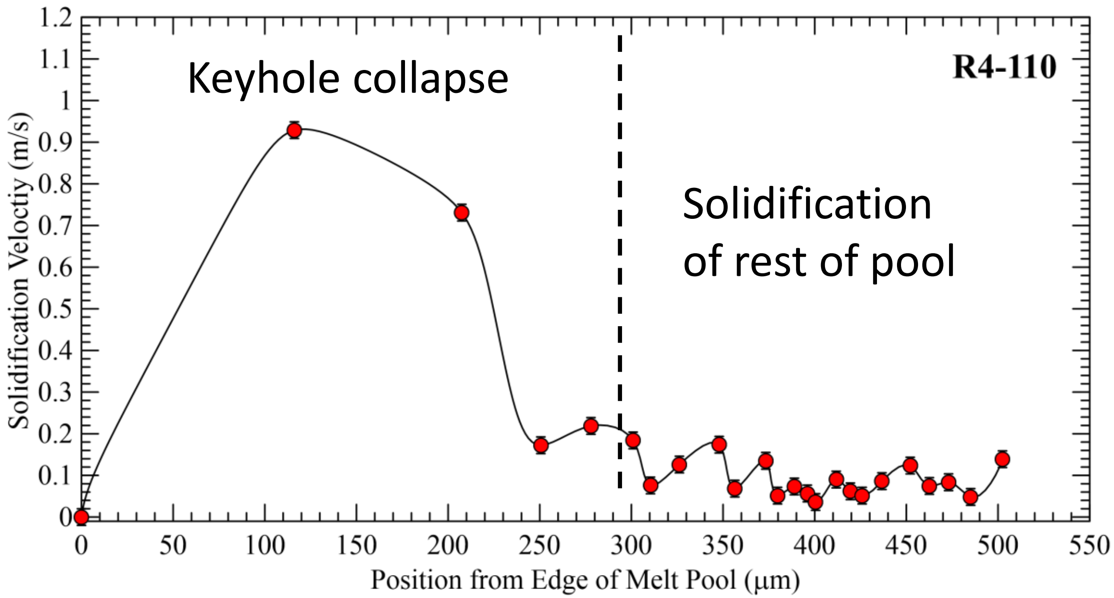
Dendrite Spacing ~ 900 nm +/- 75 nm

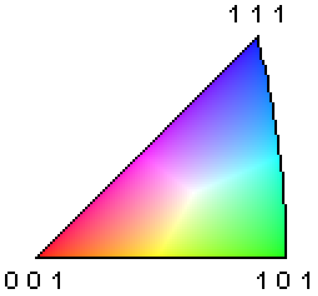
Finer Cell/Dendrite Spacing Indicates Higher Cooling Rate with Lower Laser Power

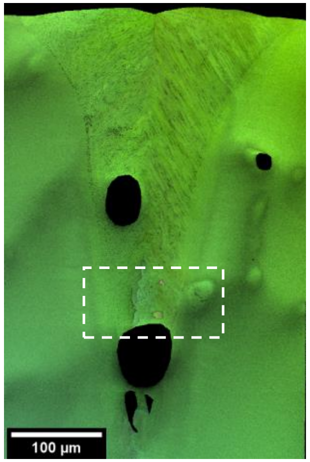
Different Cell/Dendrite Orientations Observed at Top of Lower Laser Power Spot Melt– Suggests Nucleation of Different Orientation (CET)

Keyhole Collapse Causing CET

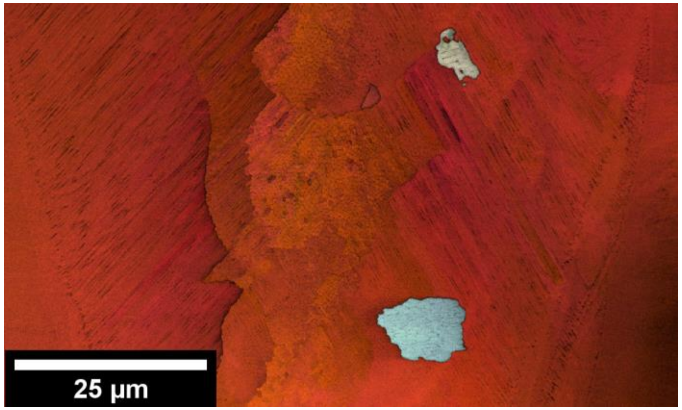








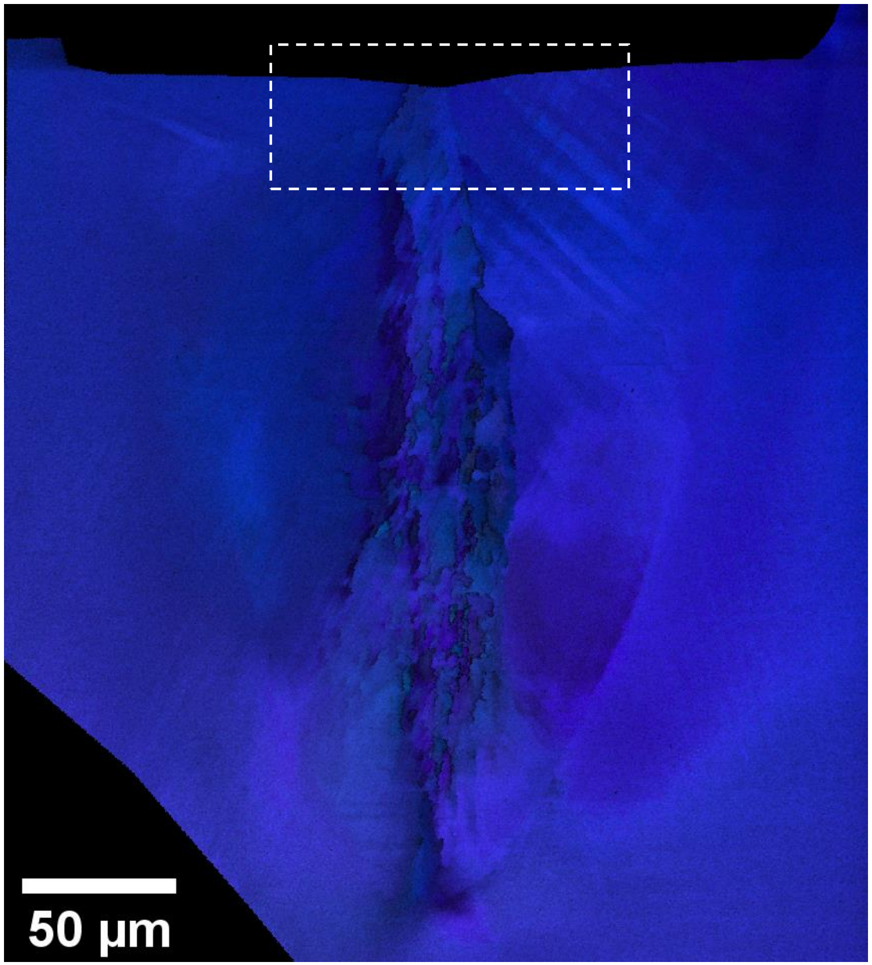
Build Direction

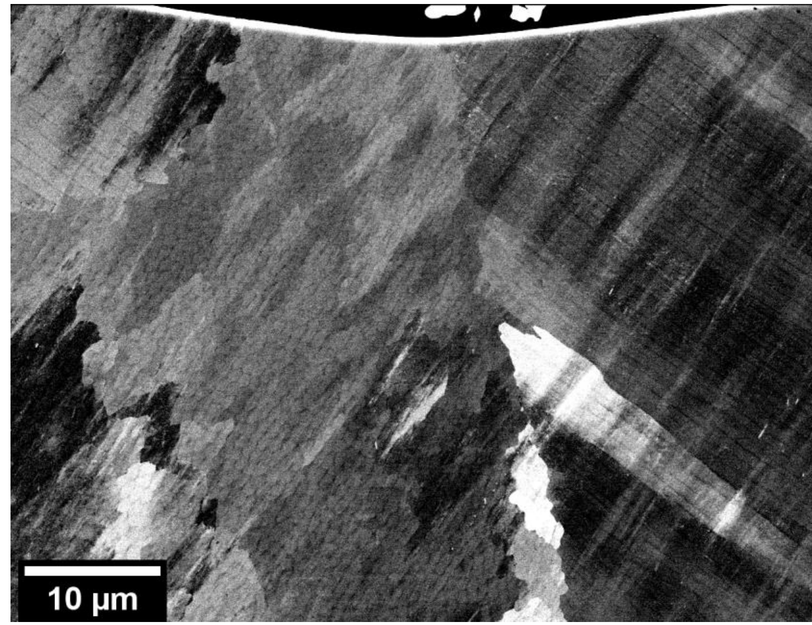


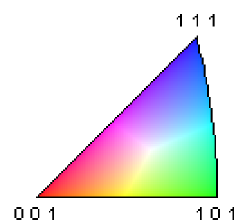
Normal Direction

Growth Orientations of R2-111

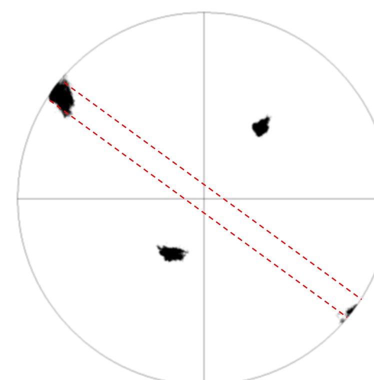
R2-111 208 W



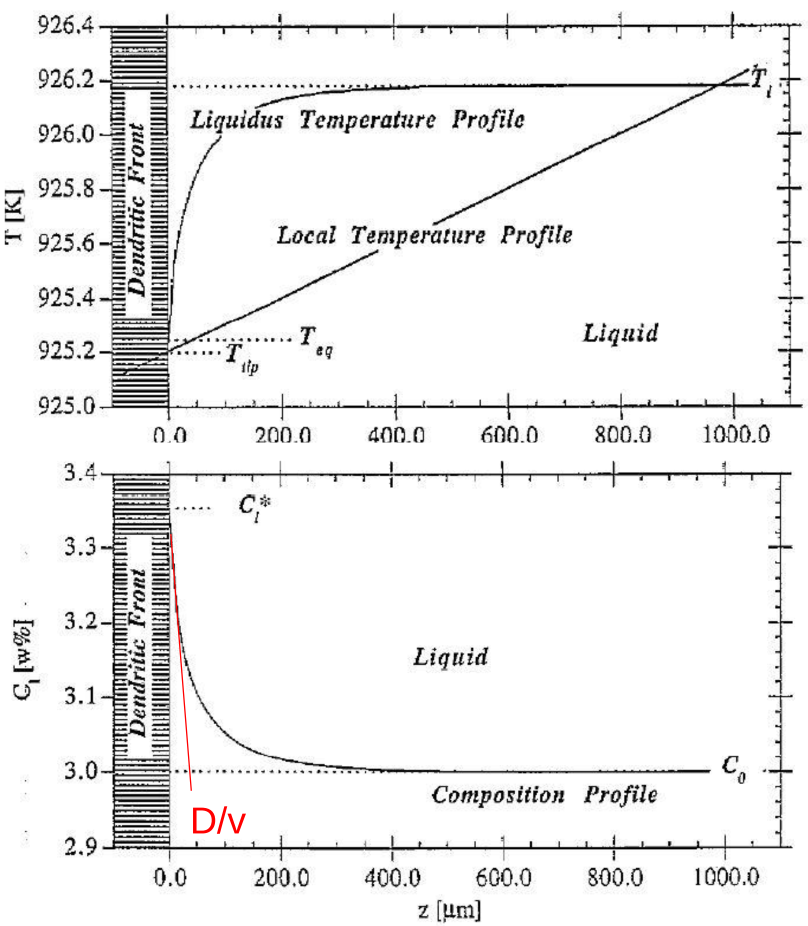


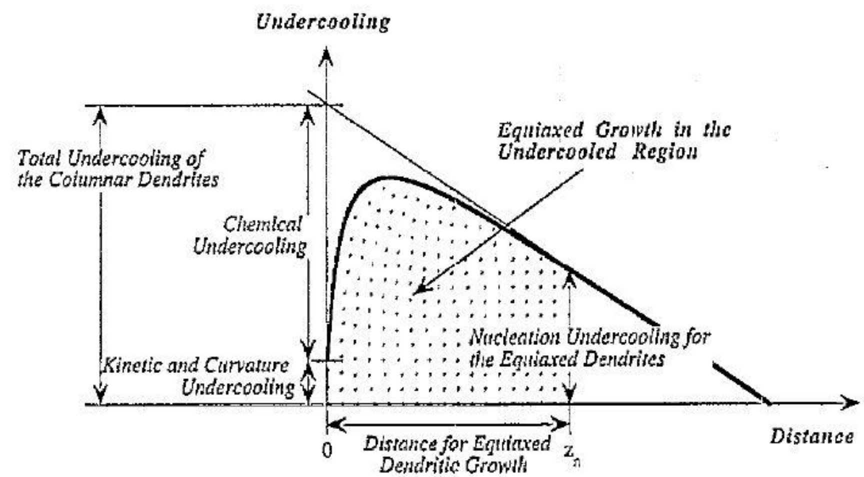


Build Direction



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.

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

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)

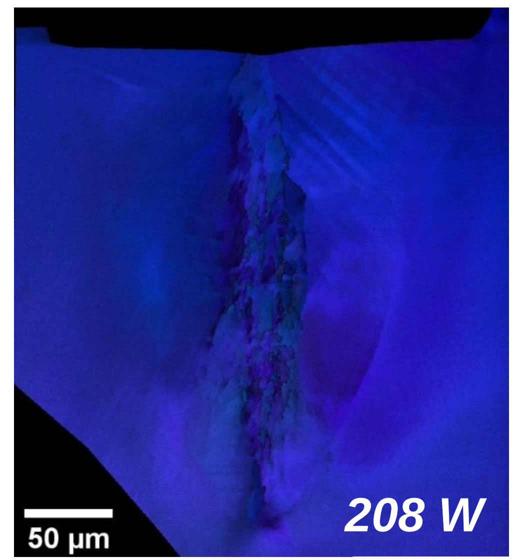
Significance and Impact

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

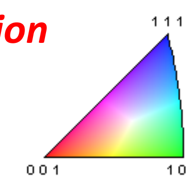
Research Details

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

New grain orientations



Substrate orientation maintained



Build Direction

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

CANFSA CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

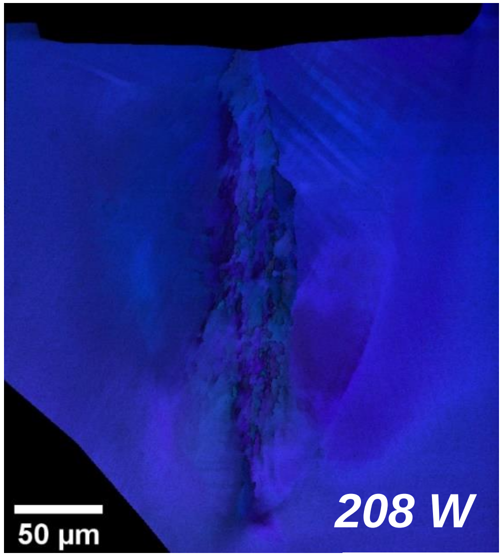
Staff: *Jonah Klemm-Toole*

Faculty: *Amy Clarke and Kester Clarke*

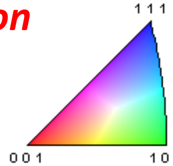
Industrial Partners: *TBD*

Project Duration: *Feb 2019 – Feb 2021*

New grain orientations



Substrate orientation maintained



Build Direction

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