CANFSA

Center for Advanced Non-Ferrous Structural Alloys

An Industry/University Cooperative Research Center

Project 37b-L: Rationalization of Liquid/Solid and Solid/Solid Interface Instabilities During Thermal-Mechanical Transients of Metal Additive Manufactuing (ISU)

Spring 2019 Semi-Annual Meeting
Iowa State University, Ames, IA
April 3-5, 2019

Student: Matt Kenney (*ISU)*

Faculty: Priyanka Agrawal (ISU), Maria Quintana(ISU)

Industrial Mentors: Prof. Peter C Collins (ISU)

MURI members: S. Babu (UTK), A. Clark (CSM), J. R. Jinschek (OSU), T. Pollock (UCSB), Z. Kong (VT), S. Ringer and X. Liao (Univ. of Sydney), S. Primig (Univ. of South Wales) and TEAM

Industrial Mentors: Billy Short and Jennifer Wolk (ONR)

Project 37b-L: Rationalization of Liquid/Solid and Solid/Solid Interface Instabilities During Thermal-Mechanical Transients of Metal Additive Manufactuing (ISU)

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

- Student: Matt Kenney (ISU)
- Advisor(s): Prof. Peter Collins (ISU)

Project Duration
August 2018 to August 2021

- Problem: Understand the thermal gradients in an AM build as a function of different scan strategies by studying the microstructure.
- Objective: To understand the science behind the relation between thermal gradients and the microstructure and texture evolution
- Benefit: Optimize the final cost and mechanical properties of the AM component

Recent Progress

- Ti-6Al-4V AM samples with three scan strategies are imaged using optical microscope and SEM in BSE mode
- Texture studies are carried out using EBSD along and across the AM build direction
- Precession Electron Diffraction parameters have been optimized.
- SEM characterization of Inconel 738 sample.

Metrics

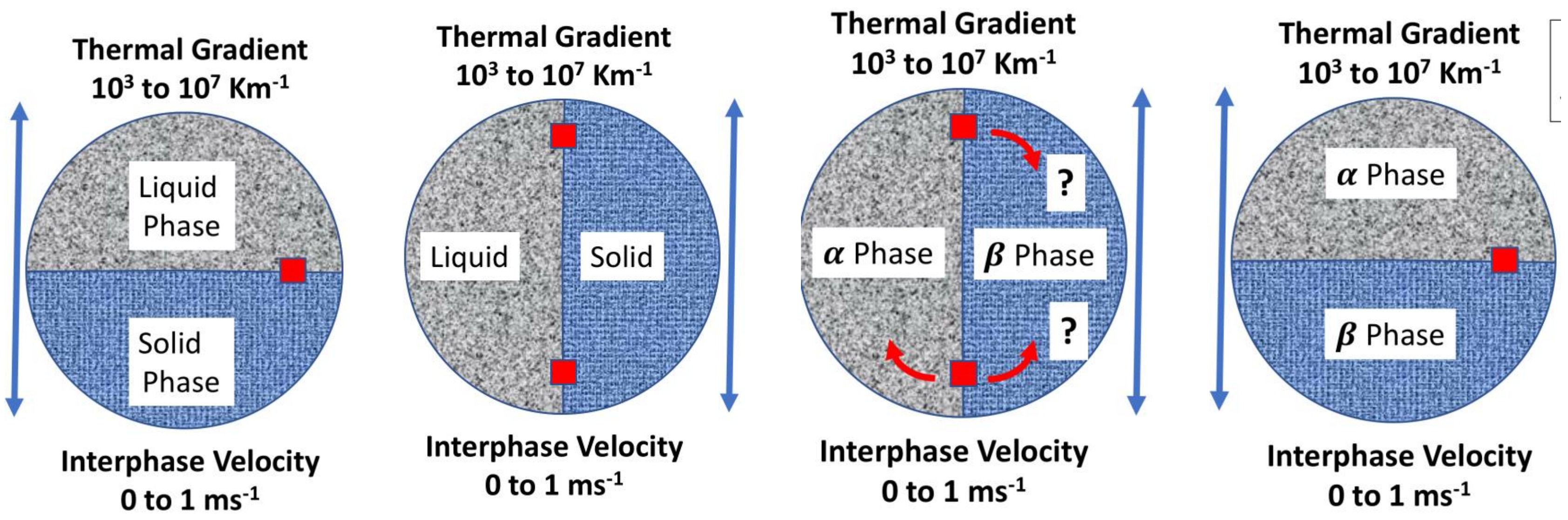
Description	% Complete	Status
1. Sample preparation for optical, SEM-BSE, EBSD and TEM	45%	● (green)
2. Literature review	20%	● (green)
3. Texture scans – EBSD,SRAS, and ASTAR PED	25%	● (green)
4. Prior beta orientation and other analysis	5%	● (yellow)
5. Relate thermal gradients to microstructure and the final mechanical properties	0%	● (red)

Industrial Relevance

- Understanding underlying behavior of different AM strategies on resulting microstructure and mechanical properties of metallic printed parts
- Build a scientific basis into Integrated Computational Materials Engineering (ICME) predictions of AM knowledge gap areas (nano and micro scale regimes of length and time)
- Reduce trial and error phase of AM design and manufacture curve

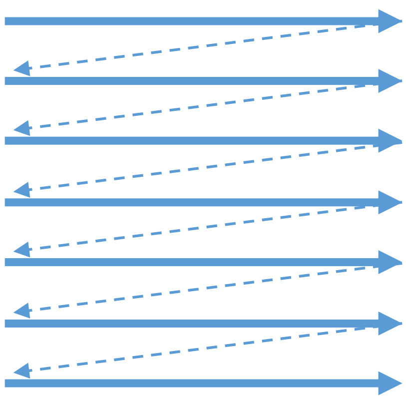
Background

- In AM, interphase instabilities occur with large spatial-temporal thermo-mechanical gyrations
- The focus is to understand L-S and S-S phase transformation which will depend on the thermal gradient



Research interests:

Three different AM scan strategies are selected to understand fundamental research questions. The different scan strategies will change the thermal gradient: Raster, Dehoff and Random



Ordering of Raster Fill

Ordering of Dehoff Fill

Ordering of Random Fill

Research interests:

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

QUESTIONS:

- Will there be a local equilibrium at the interface due to thermal gradient?
- What will it be in the case of different scan strategies?
- How the interfaces will move with the change in thermal gradient?
- Will there be a change in microstructure and will the microstructures evolve from bottom to top of the build?
- Will there be a change in the scale of microstructures?
- Will there be a change in crystallographic variants?
- Will there be a change in defect population?

ONR-MURI plan

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Genesis of Controlled Alloy Samples

- Lack of Knowledge: Liquid-Solid & Solid-Solid Interface stability during T-σ Transients
- Ni-Al-Cr and Ti-Al-V Model Alloy Design (All)
- Samples with μ-structure control EBM (UTK)
- Thermo-Mechanical Driver Setup (UTK)

In-situ Measurements

- In-situ Transmission electron Microscopy (OSU,ISU& AUSMURI)
- In-situ Scanning Electron Microscopy (AUSMURI)
- In-situ Synchrotron, Photon & Neutron Measurement (All)
- In-situ Phonon Based Crystal Texture Measurement (ISU)
- In-situ Thermal & 3D Displacement Imaging (VT)

Ex-situ Measurements

- Ex-situ Atom Probe Analysis (UT, CSM & AUSMURI)
- Ex-situ TEM& SEM Analyses (All)
- Ex-situ Tri-Beam Characterization (UCSB)
- Ex-situ Optical Characterization (All)

End-point & Data Curation

- ICME & Data Analyses (All & DOE & DOD Labs & AUSMURI)
- Dream-3D framework for Data & Sample Curation (All & DOE and DOD Labs.)
- Physical Metallurgy of Metal Alloy Phase Interfaces with Thermal Gradients and Gyrations

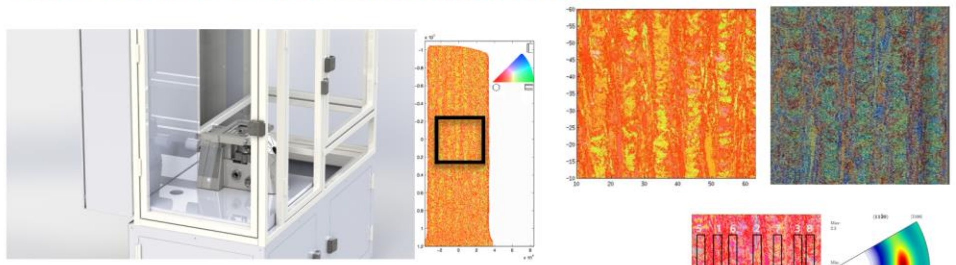
MURI: UT: University of Tennessee, Knoxville; CSM: Colorado School of Mines; Iowa State University (ISU) ; The Ohio State University (OSU); University of California, Santa Barbara (UCSB); and Virginia Polytechnic Institute and State University (VT) ;
AUSMURI; U. Sydney and The University of South Wales

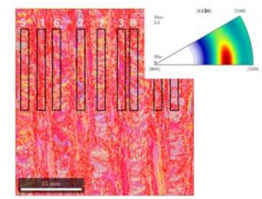
Research interests:

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

E-PBF Material

Spatially Resolved Acoustic Spectroscopy

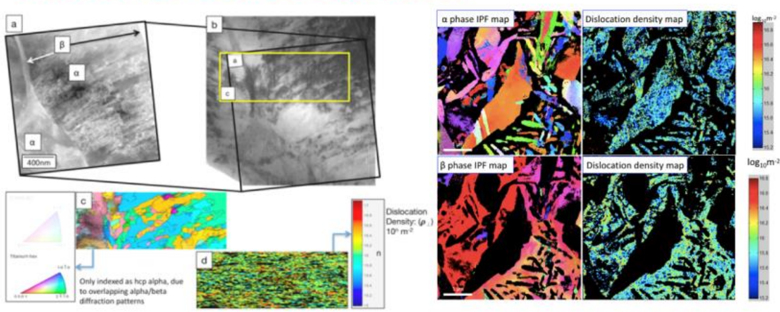


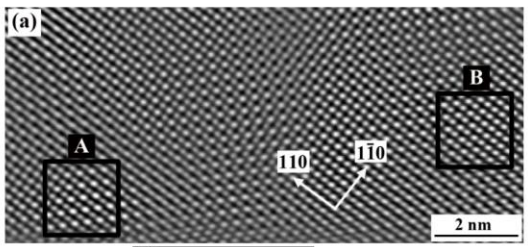


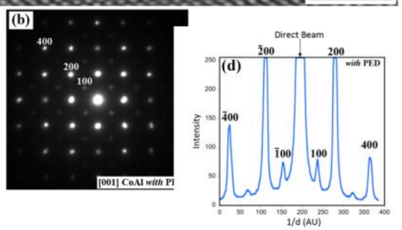
In-situ measurements of texture and elastic moduli.
First data fusion to recover 3rd Euler angle (right)
New applications of SRAS to elevated temperatures.

In-situ Phonon Based Crystal Texture Measurement (ISU)

Precession Electron Diffraction







Accurate assessments of phases and defects with nm resolution.
Integrated with spatial information of composition.
Local order determination. Integrated with DD/CP-FEM models?

Ex-situ TEM& SEM Analyses (All)

Outline of the project

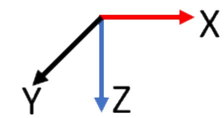
- Three Ti64 AM and Inconel 738 builds with different scan strategies are provided by ONRL – Raster(L), Random (R) and Dehoff (D)

- **TASKS:**
  1. Imaging – Macro, Optical and SEM-BSE
  2. Texture
     a) SRAS – Spatially Resolved Acoustic Spectroscopy (macro-texture)
     b) EBSD – Electron Back scattered diffraction (SEM) (micro-texture)
     c) PED – Precession Electron Diffraction (TEM) (nano-texture)
  3. Analysis of the 2D and 3D texture data to determine - the prior β orientation, reconstruction of β grain size, volume fraction of the phases, α laths size and aspect ratio, etc.
  4. Develop the understanding to relate thermal gradient to the microstructural evolution

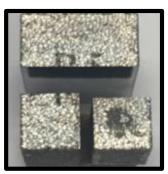
Material

CANFSA — CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

- 3 Ti64 AM builds – Raster(L), Random (R) and Dehoff (D)
- 1 Inconel 738 build has been received - Random
- ISU is provided with L5, R5 and D5 samples
- Z is the build direction for all the samples
- XY and XZ planes are polished for imaging and texture studies

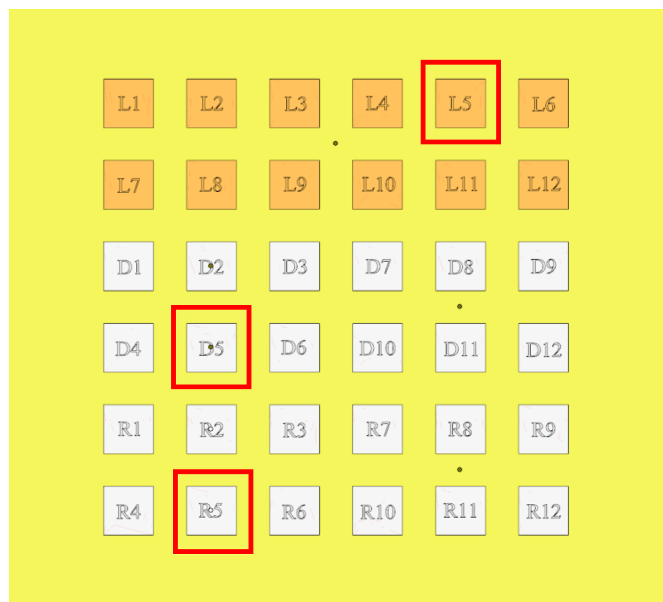


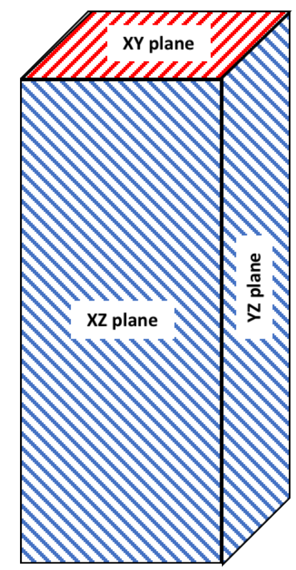
Raster (L5)



Random (R5)

Dehoff (D5)



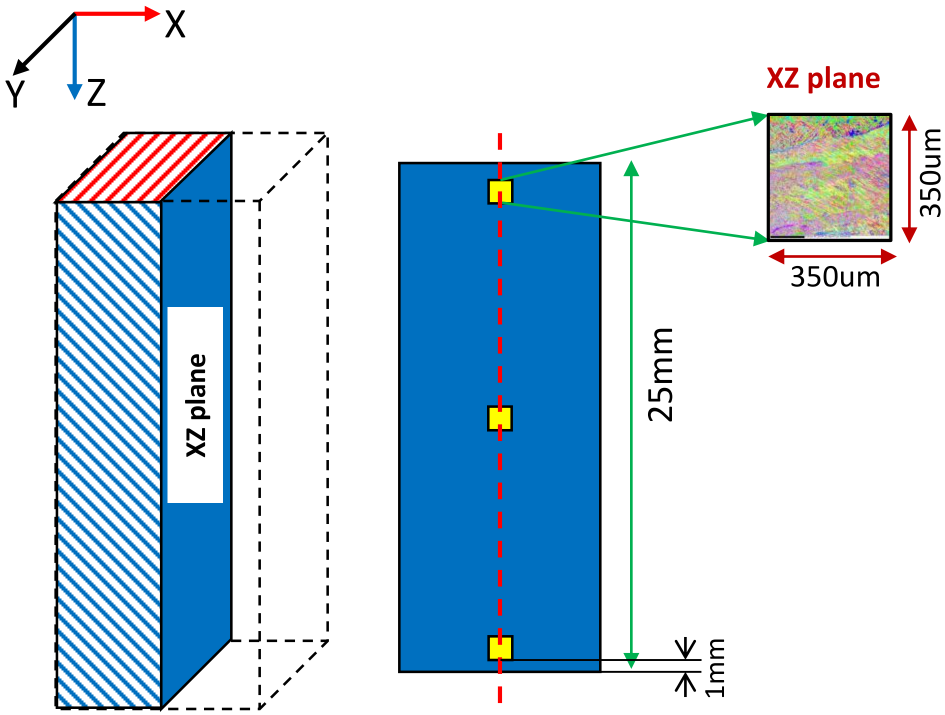


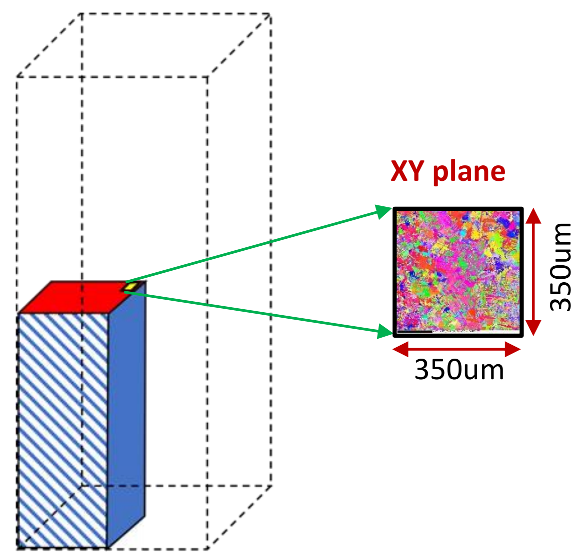
15x15x25mm

L5 - has vertical features
R5 - melt pools
D5 - has horizonal features

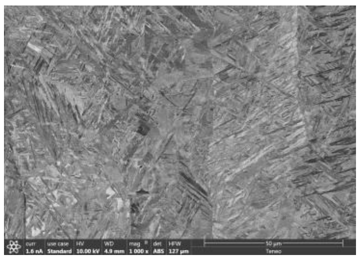
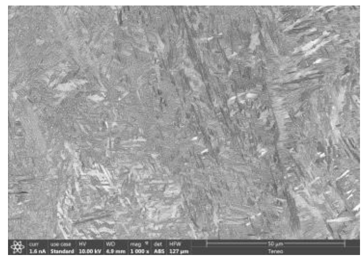
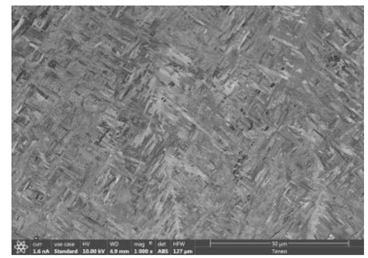
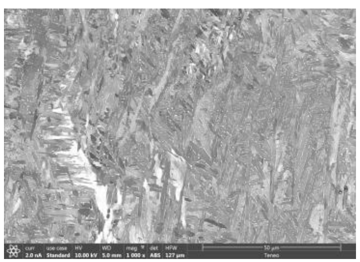
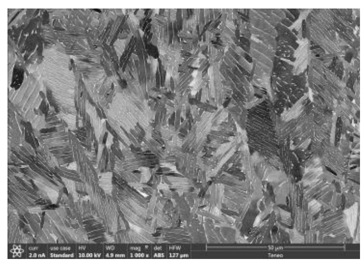
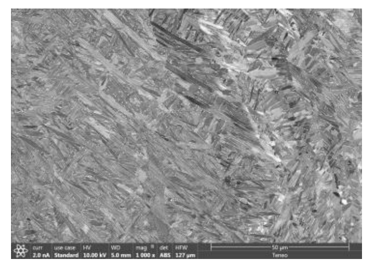
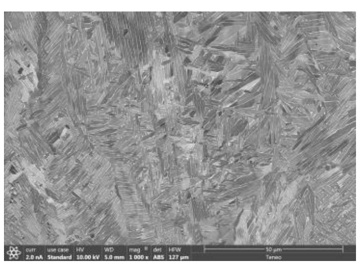
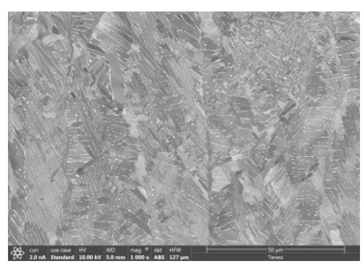
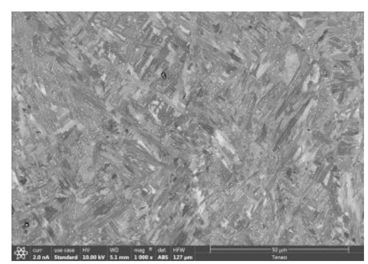
Texture – EBSD in XZ and XY plane

- Ti64 and Inconel samples – L5, R5 and D5
- XY and XZ planes are polished for texture analysis
- 350x350ums area with 0.5um step size, 8x8 binning
- EBSD to obtain micro-texture and compare with SRAS (macro-texture) (ISU), PED ASTAR (ISU), Neutron scattering (CSM) and Synchrotron (UTK)

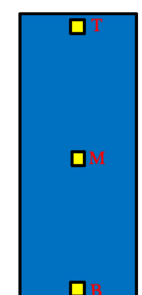




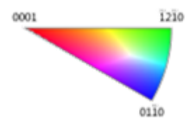
Imaging: SEM-BSE mode in XZ plane

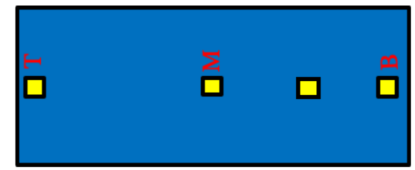
	Top	Middle	Bottom
L5			
D5			
R5			

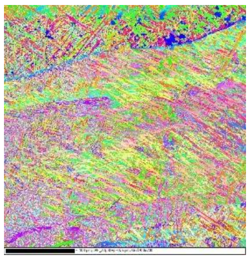
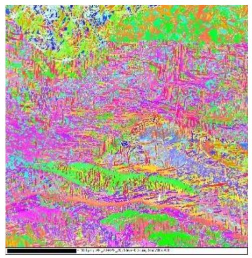
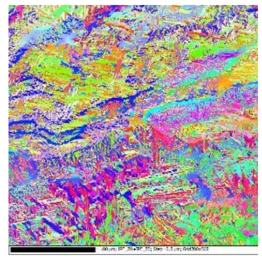
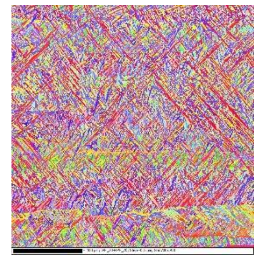
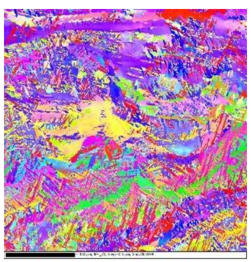
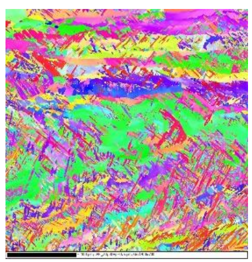
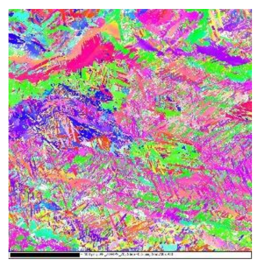
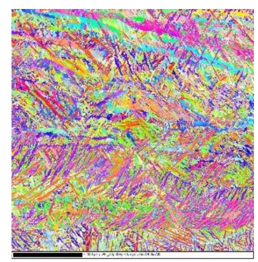
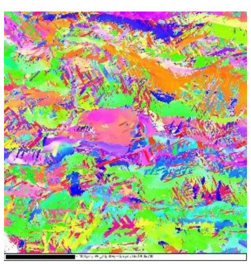
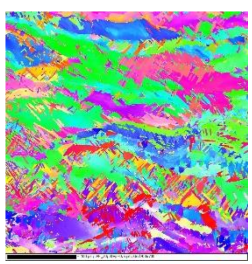
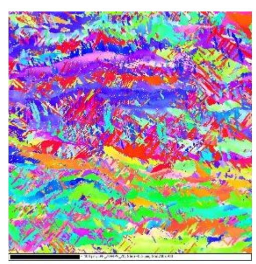
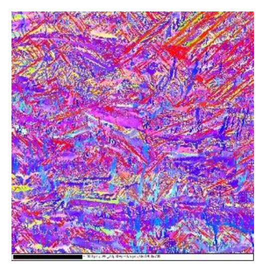
- Columnar morphology is observed in all the samples
- The columnar grains are at an angle to the longitudinal axis (growth and cooling rate)
- L5 is with finer morphologies as compared to R5 and D5. R5 seem to have coarsest morphologies
- All the samples show a combination of basket-weave and lamellar morphologies where R5 seems to have greater fraction of lamellar morphology



EBSD in XZ plane





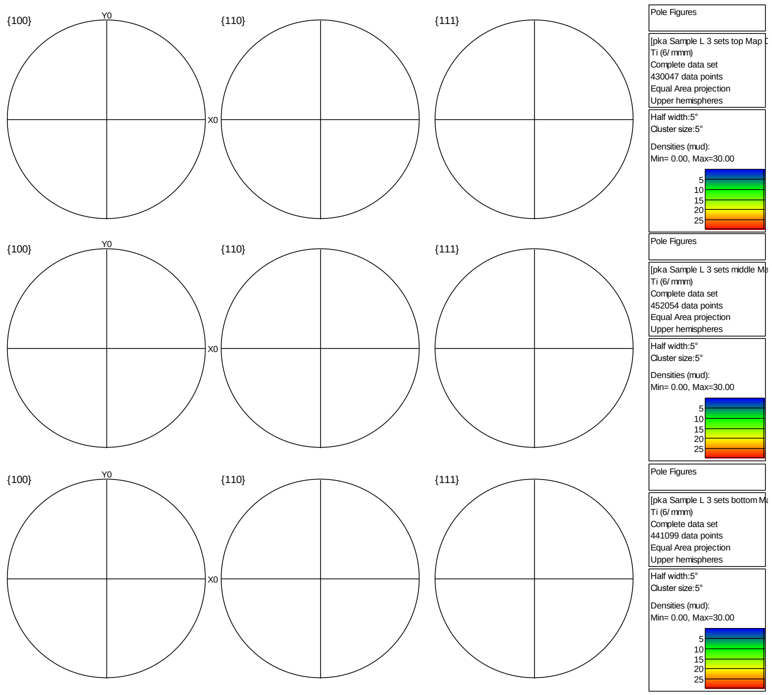
	top	middle	1/4th from bottom	bottom
L				
D				
R				

- Microstructure evolves from bottom to top along the build direction → basket-weave to colony structure
- There is not much difference between texture of middle and top
- Next steps could be obtaining
  a) volume fraction of colony and lamellar morphology from B to T
  b) HT beta orientation
  c) prior beta grain size
  d) alpha lath size

EBSD – L5 in XZ plane

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

Alpha

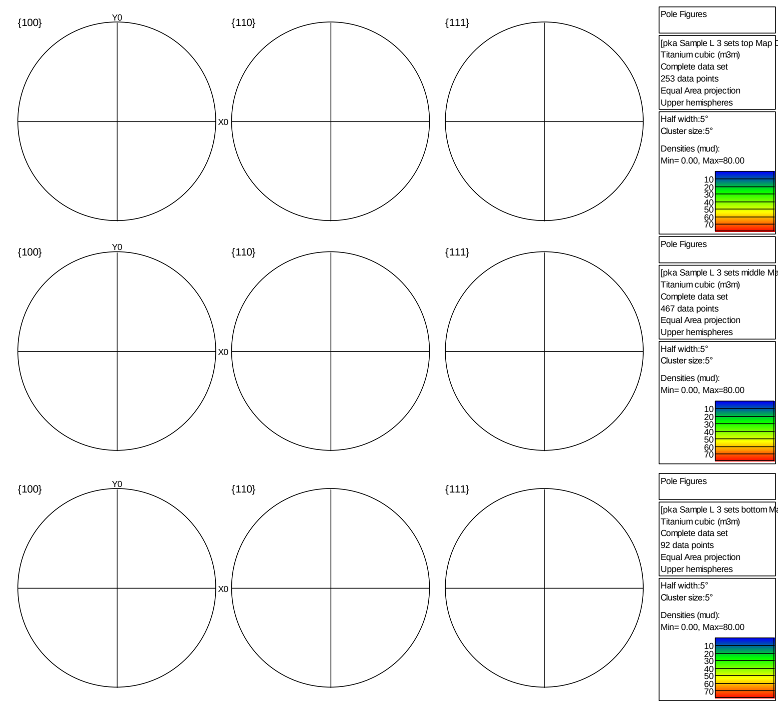


T

M

B

Beta

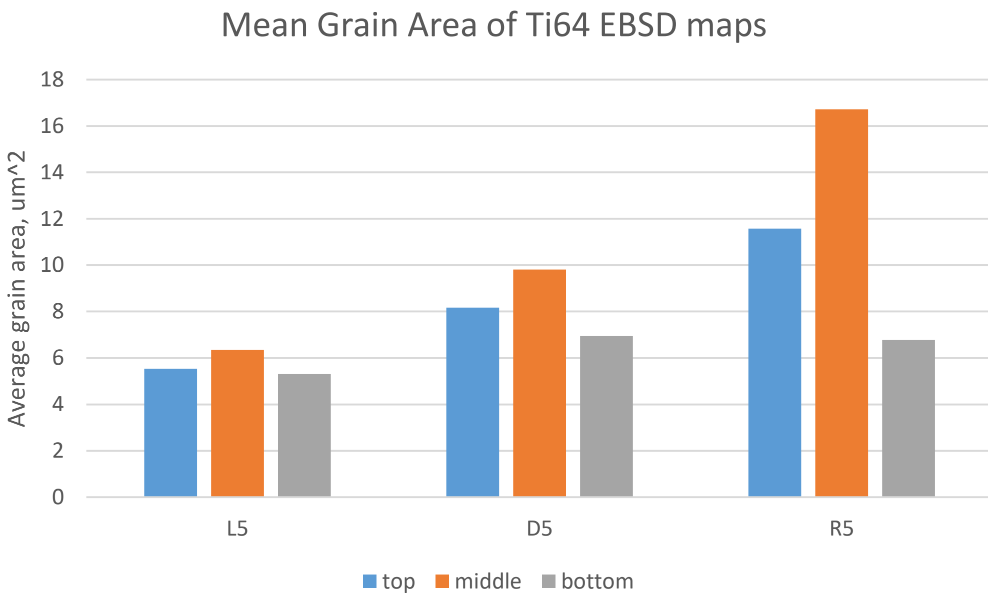


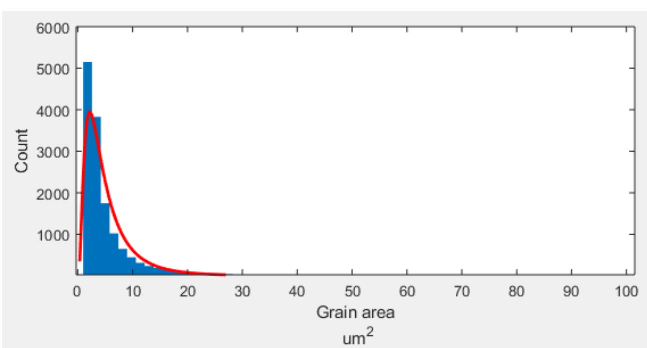
- The scale for alpha and beta is not same
- Alpha texture seem to be stronger at the bottom and weakens along the build direction to the top
- Beta texture shows similar trend but more statistics is required before confirming the trend
- Similar is the trend for D and R samples from bottom to top

Sample L	alpha	beta
Top	26.54	37.01
Middle	27.04	42.06
Bottom	29.37	79.87

Grain Size – Lognormal fit

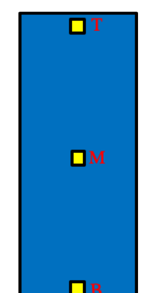
CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

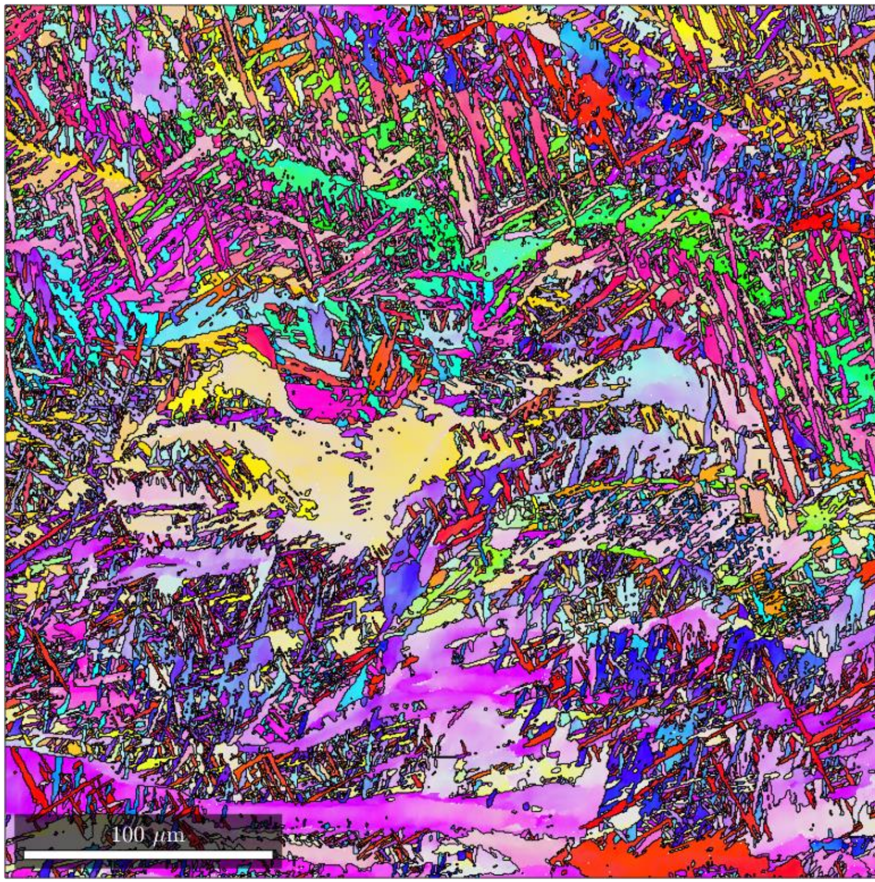




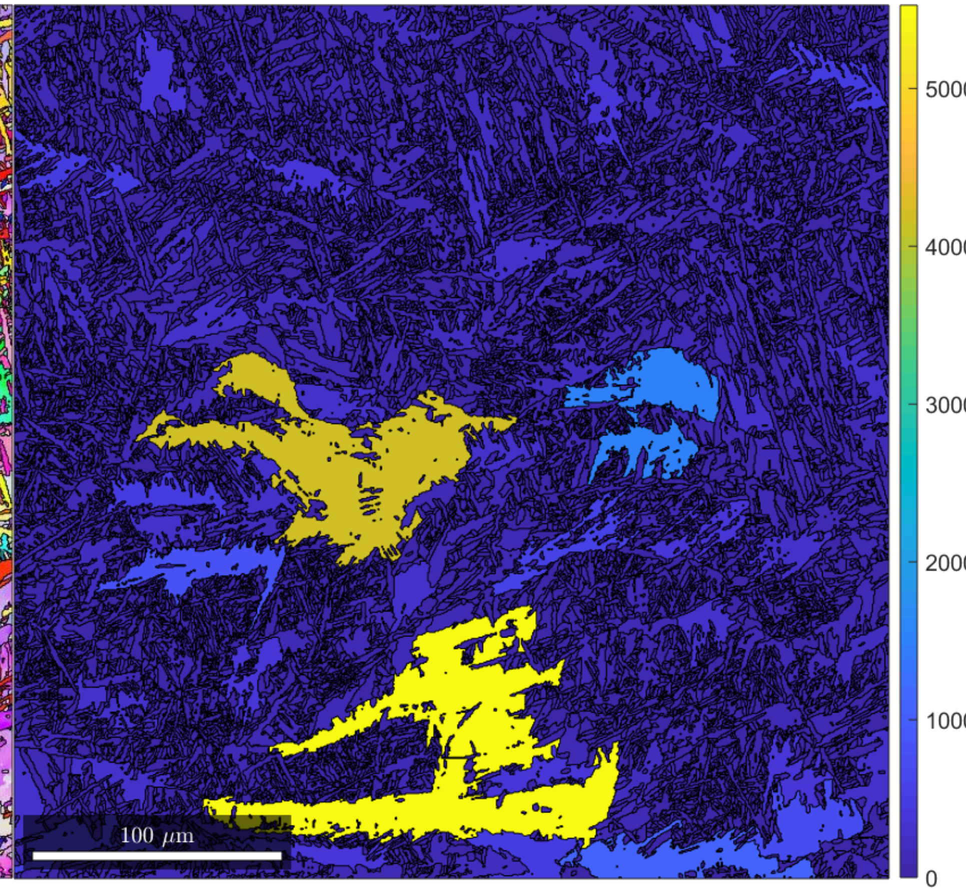
- Quantitative comparison of average grain size
- Data were fit to a lognormal distribution

	top	middle	bottom
L	Mu: 1.40 Sigma: 0.79 Mean: 5.54	Mu: 1.48 Sigma: 0.86 Mean: 6.36	Mu: 1.38 Sigma: 0.76 Mean: 5.30
D	Mu: 1.60 Sigma: 1.00 Mean: 8.17	Mu: 1.71 Sigma: 1.08 Mean: 9.81	Mu: 1.55 Sigma: 0.88 Mean: 6.94
R	Mu: 1.81 Sigma: 1.13 Mean: 11.57	Mu: 2.06 Sigma: 1.23 Mean: 16.7	Mu: 1.50 Sigma: 0.91 Mean: 6.78





Ti64 D3 top-grain orientations



Ti64 D3 top-grain area

Summary from the experiments

CANFSA
CENTER FOR ADVANCED
NON-FERROUS STRUCTURAL ALLOYS

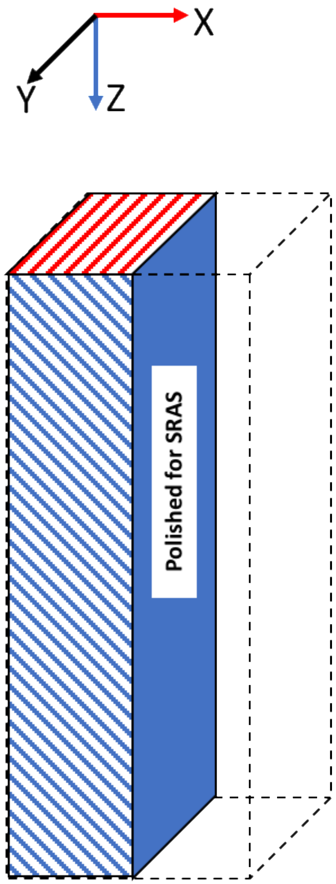
Imaging

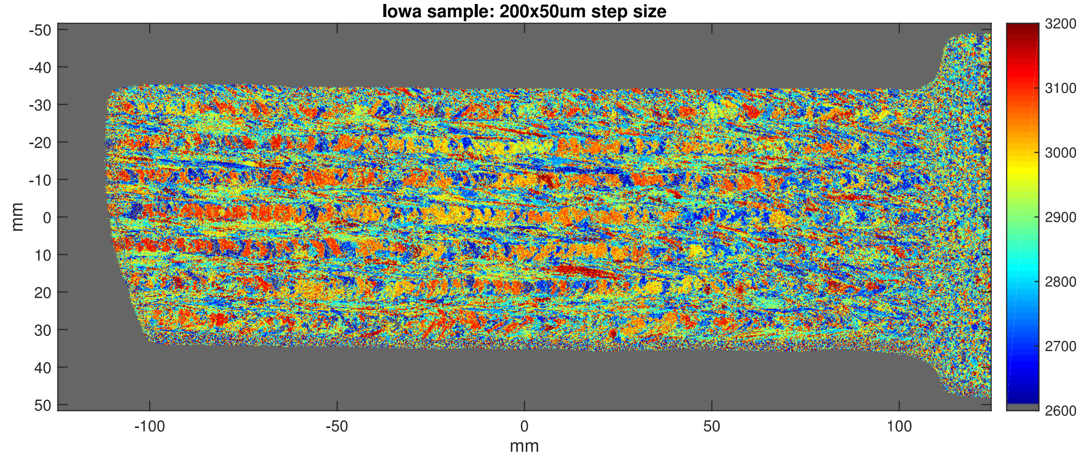
- From bottom to top – the microstructure is evolving from basket-weave to colony structure
- L5 has finer morphologies as compared to R5 and D5 for both XY and XZ planes. R5 seem to have coarser morphologies as compared to D5
- All the samples show combination of basket-weave and lamellar morphologies where R5 seems to have greater fraction of lamellar morphology

Texture:

- Bottom to top – the texture seem to be evolving from strong to random (weaker)
- L seem to have stronger texture than D and R

SRAS (Spatially Resolved Acoustic Spectroscopy)

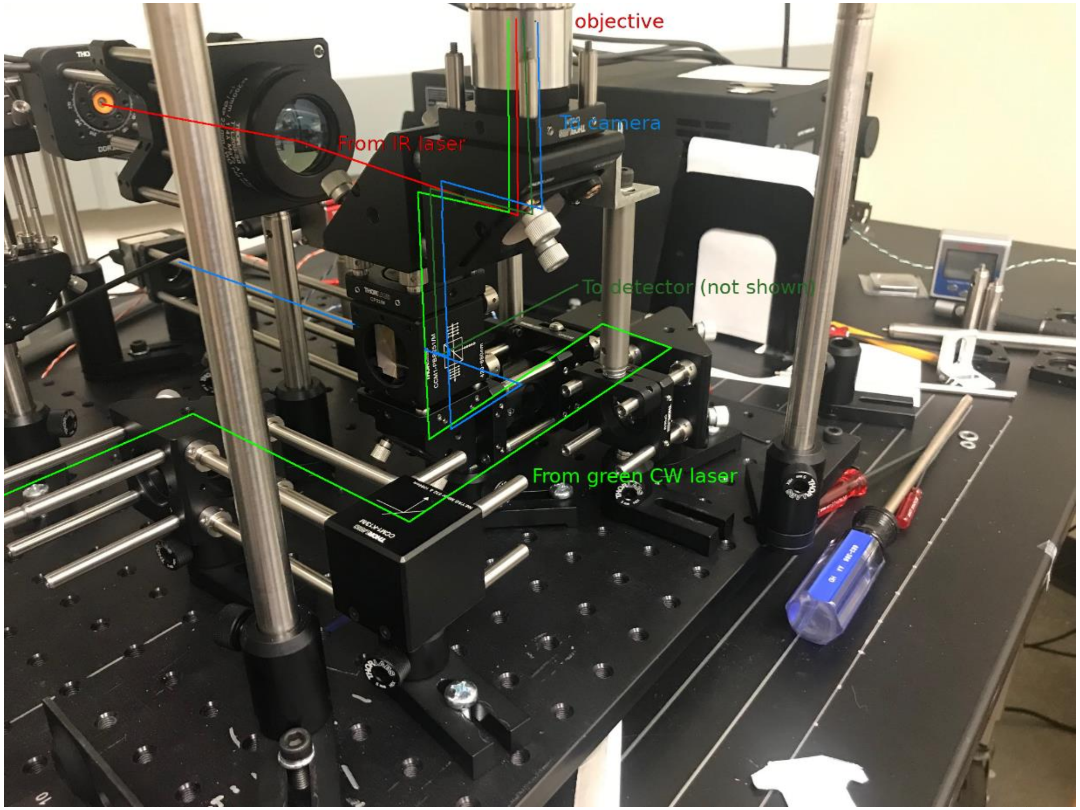




- Three Ti64 samples – L5, R5 and D5 and Inconel R5
- XZ plane is polished for SRAS using RoboMet.3D
- It will help to obtain the texture information of a larger area and also across the build in less time
- Surface quality for SRAS is similar to that for SEM
- This is particularly attractive to additive manufacturing parts where texture and grain size is directly related to the manufacturing conditions (speed and type of laser trajectory)

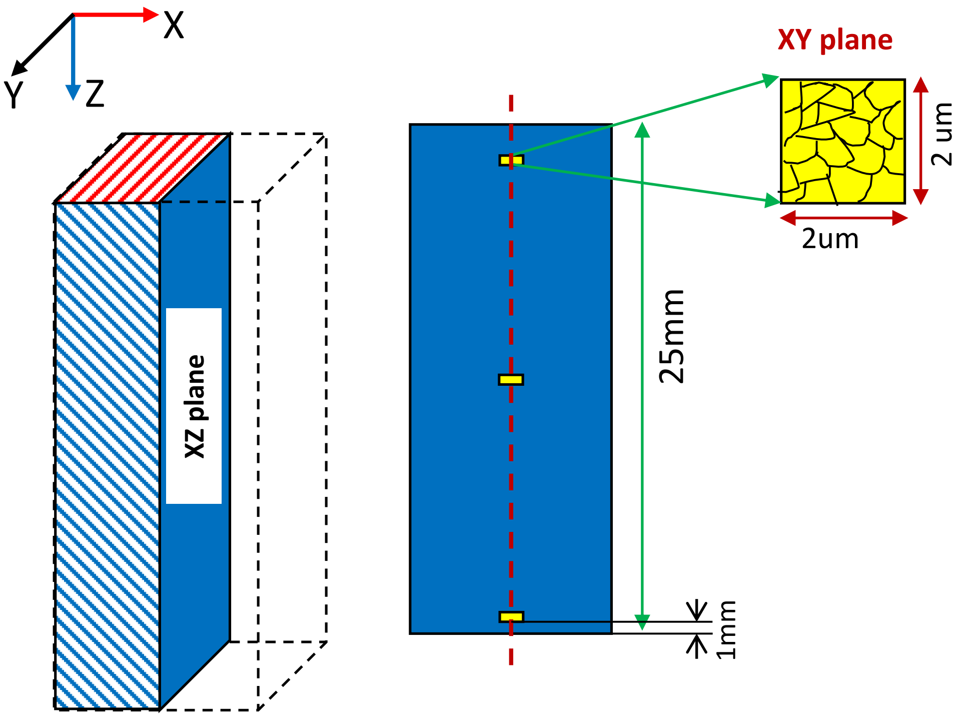
SRAS Development

- SRAS is still in progress.
- Pete and Thomas have made huge progress in the past few weeks.



PED (Precession Electron Diffraction)

CANFSA CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

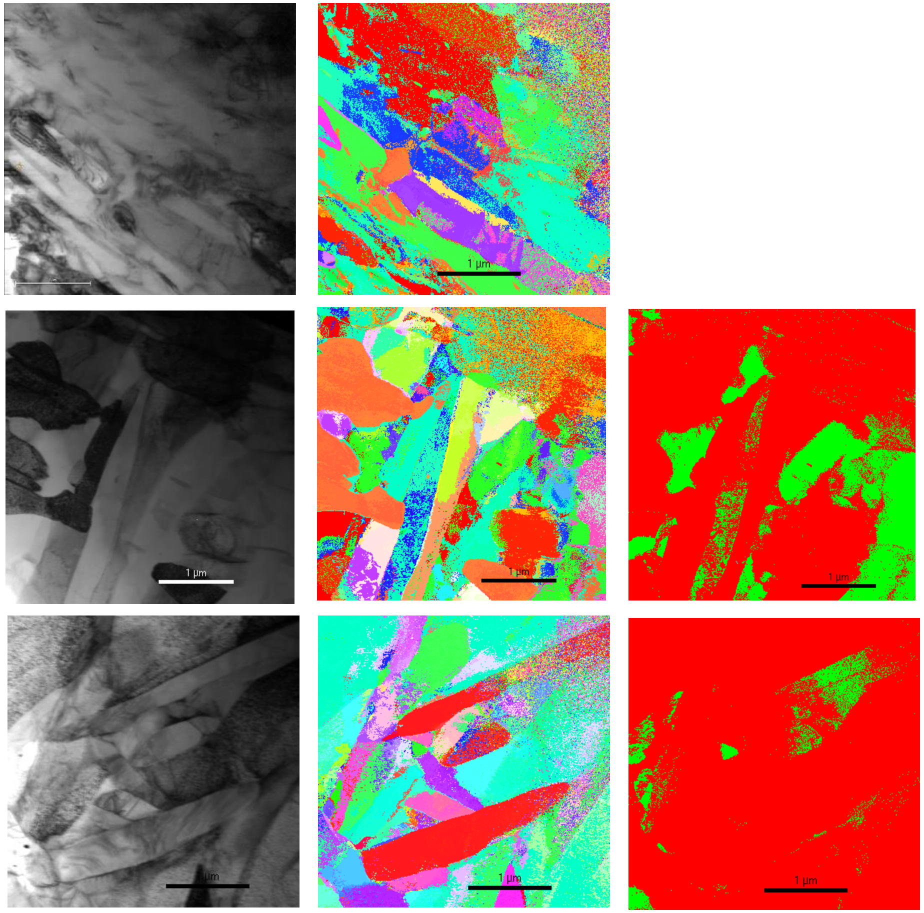


- Three samples – L5, R5 and D5
- Top, middle and bottom for all the three samples
- Imaging via TEM and to check for the presence of secondary alpha
- PED scans – to obtain a) texture information and b) the dislocation density using the MATLAB code developed by Iman et al[1]

[1] Iman G. et al, *Acta Materialia*, **79**, pp 203-214, 2014

PED Issues

- Initially we had issues collecting clean, well-indexed results.
- Parameters were then optimized to improve results.

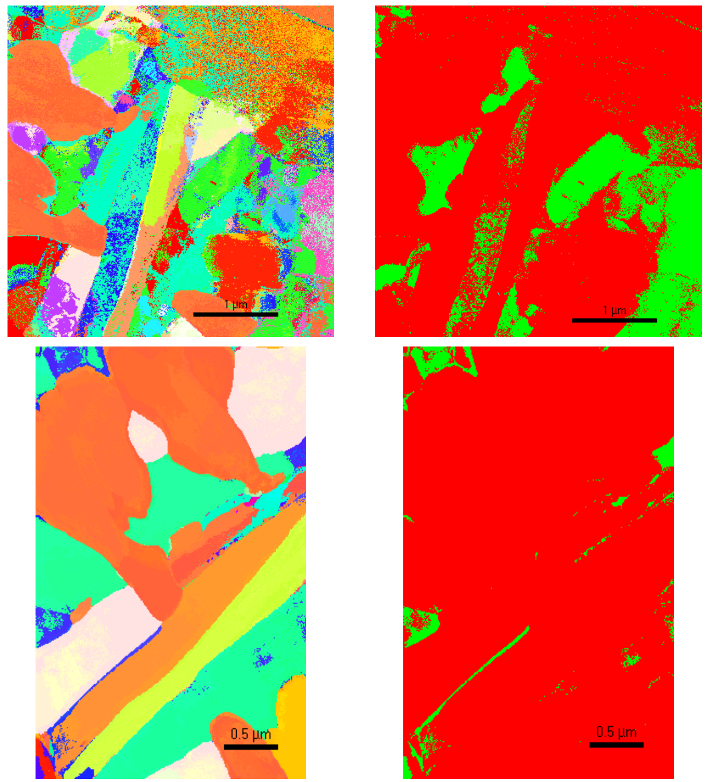


23

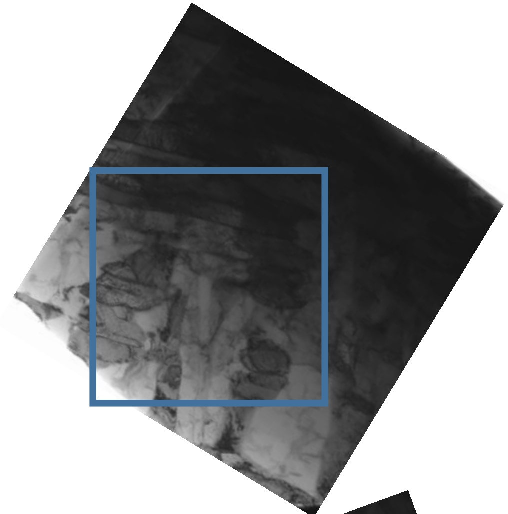
Preliminary PED Data

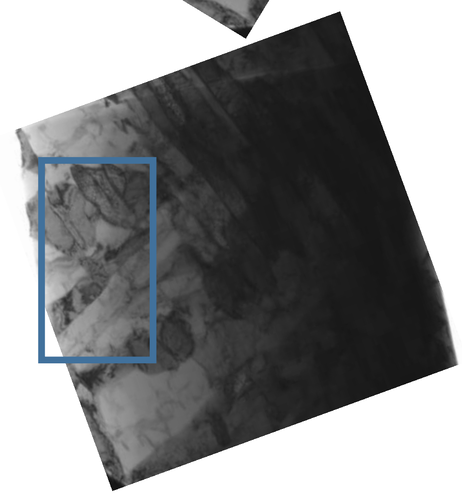
Alpha+Beta texture

Before and after the optimization of parameters
- same step size and camera length



L5 Top





Center Proprietary – Terms of CANFSA Membership Agreement Apply

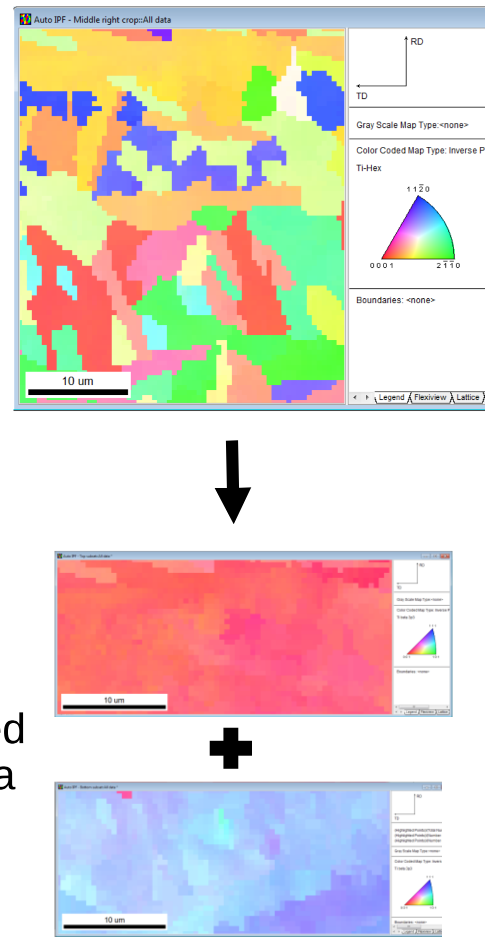
Determination of HT-beta orientation

CANFSA
CENTER FOR ADVANCED NON-FERROUS STRUCTURAL ALLOYS

- Determination of high temperature beta orientation is useful but difficult to measure
  - In-situ orientation measurement at temperature
  - Small volume fraction of retained beta at room temperature
- Using Burgers orientation relationship between BCC beta and HCP alpha, beta orientation can be determined from room temperature alpha orientation measurements

$$\{110\}_\beta \parallel (0001)_\alpha$$

$$\langle 111\rangle_\beta \parallel \langle 2\bar{1}\bar{1}0\rangle_\alpha$$



- Initial code written by Iman Ghamarian
- Current implementation is very slow
  - Speed up is necessary and can be improved with parallelization during rewrite
- Implementation fails across beta grain boundaries
  - Could be addressed by manually masking data along prior beta grain boundaries
  - Applying network theory and only comparing neighboring grains during initial parent phase segmentation could help segment grains and provide speed up

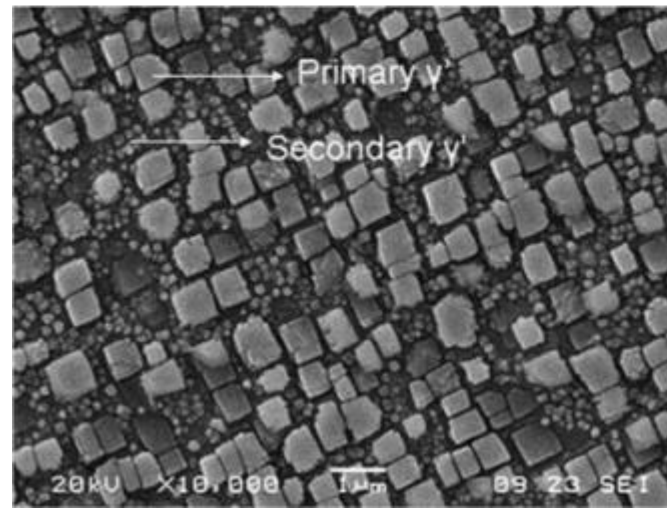
Glavicic et al., 2002. Mater. Sci. Eng., A. 351(1-2), 258-264

Tari et al., 2012. J. Appl. Crystallogr. 46(1), 210-215

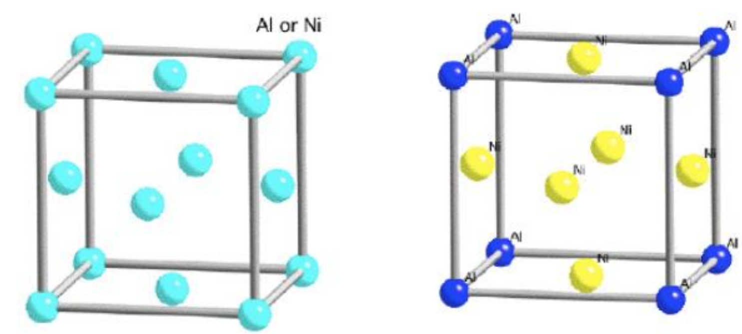
Humbert & Gey, 2002. J. Appl. Crystallogr. 35(4), 401-405

Inconel 738

- High temperature, creep resistant alloy (gas turbine blades)
- Austenitic Y matrix (FCC) with Y' precipitates (FCC)
- Interface between matrix and precipitates is semi-coherent and prevents dislocation motion while still allowing some ductility
- Few studies have been found with this materials system and none relating to electron beam powder bed AM



SEM of Solution Heat treatment in In738 showing primary and secondary Y' precipitates



Crystal structure of γ

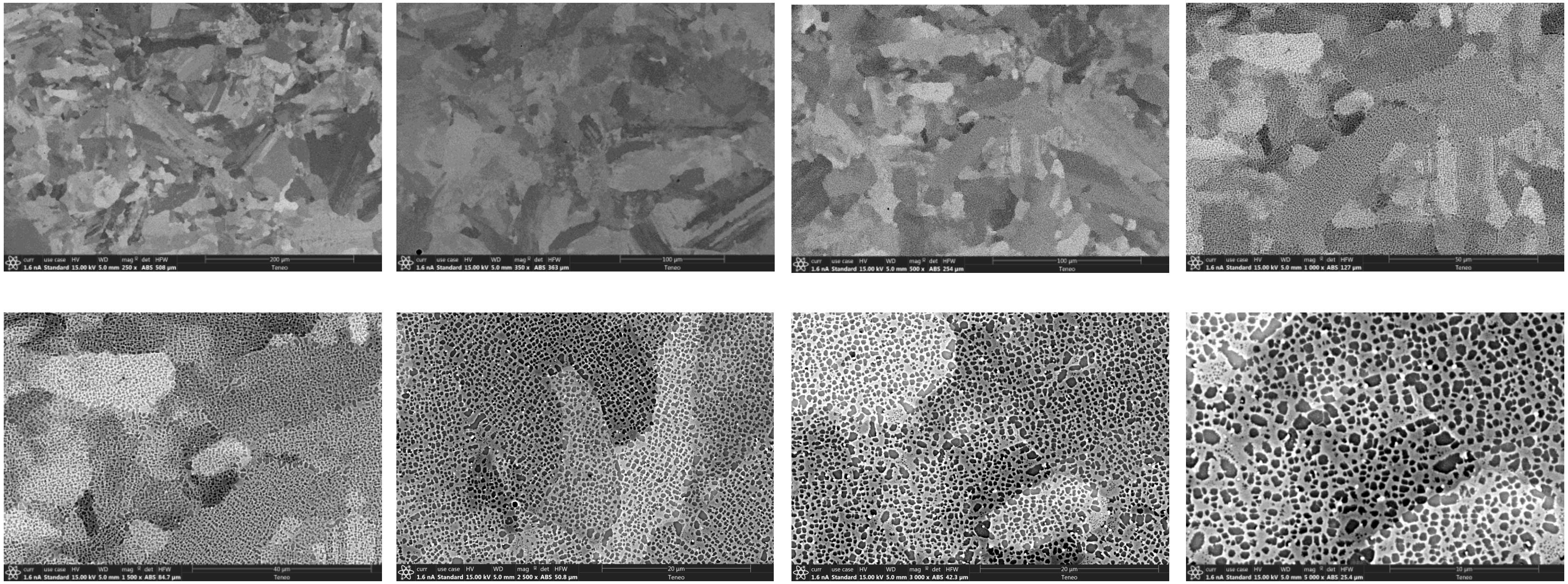
Crystal structure of γ'

Crystal structure Y, Y' phase within In738

Development of a γ' Precipitation Hardening Nickel Base Superalloy for Additive Manufacturing. Abdul Shaafi Shaikh, 2018 Department of Industrial and Materials Science, Chalmers University of Technology, Gothenburg, Sweden

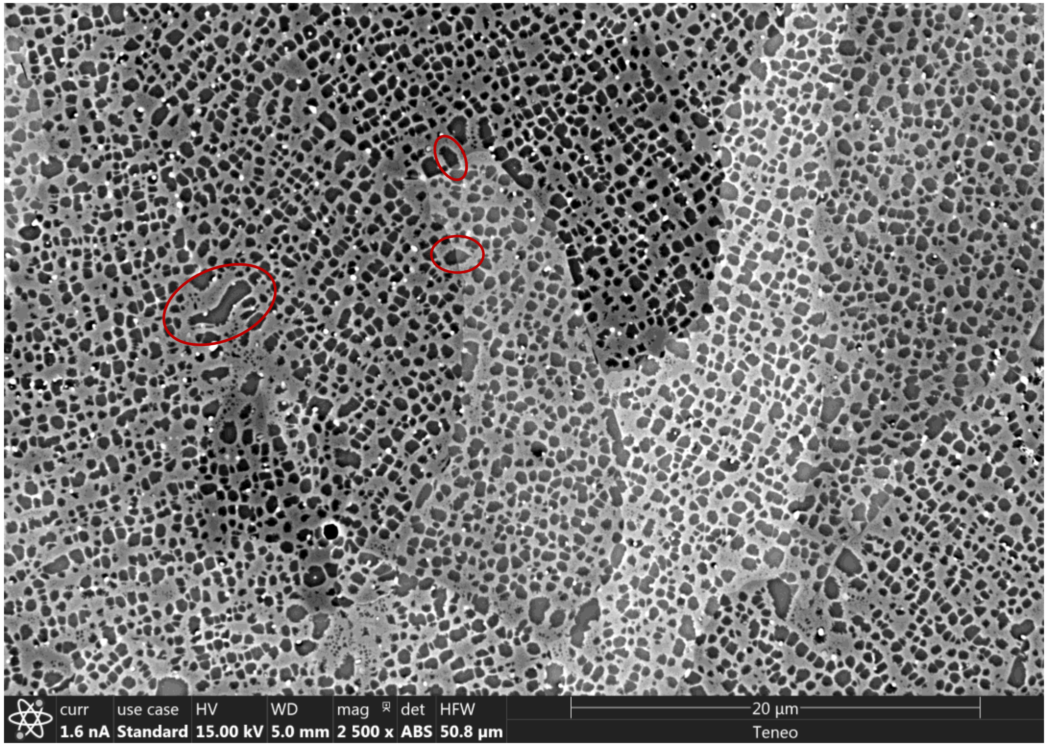
SEM - Inconel 738 – R5

XZ plane - Top



SEM - Inconel 738 – R5

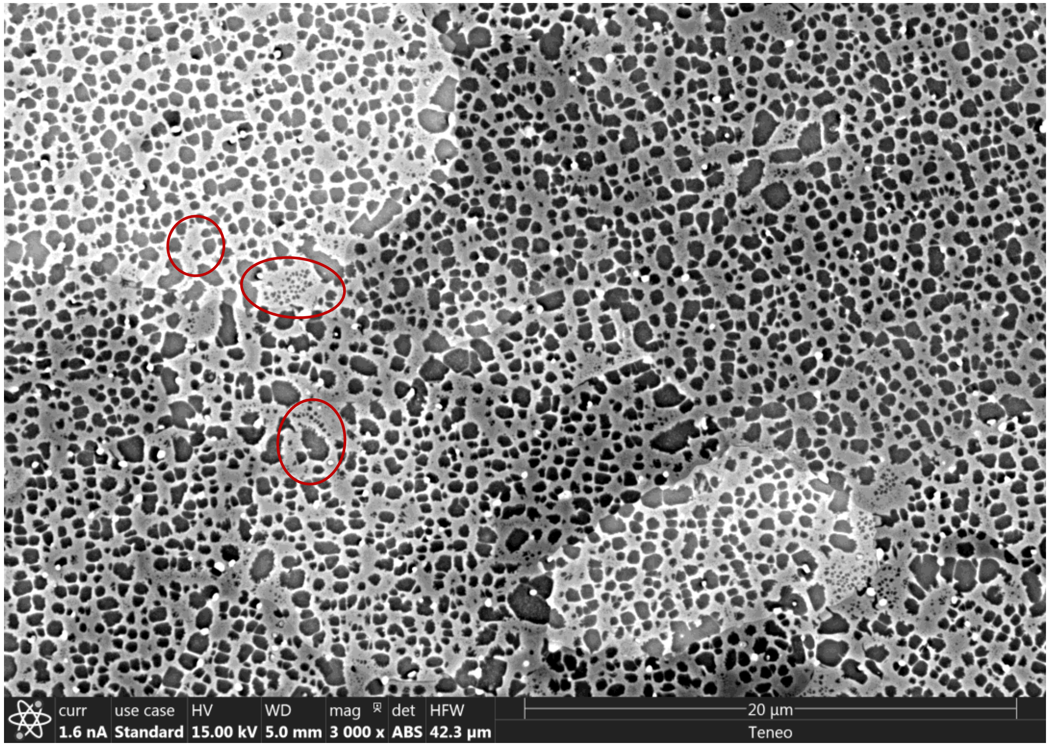
XZ plane - Top



- Gamma prime with same contrast in different grains (similar contrast)
- Other cases: contrast of gamma prime changes at the boundary but maintains coherency
- Coherency of gamma prime precipitates persists even with changes in orientation (contrast)
- With similar contrast there is a sharp change in coherency leading to sharp boundaries

SEM - Inconel 738 – R5

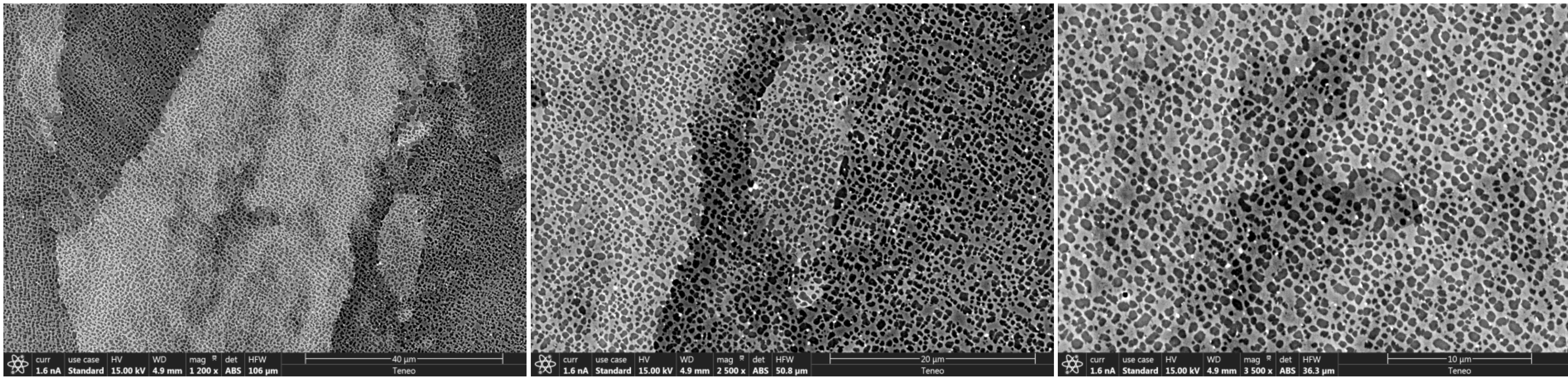
XZ plane - Top



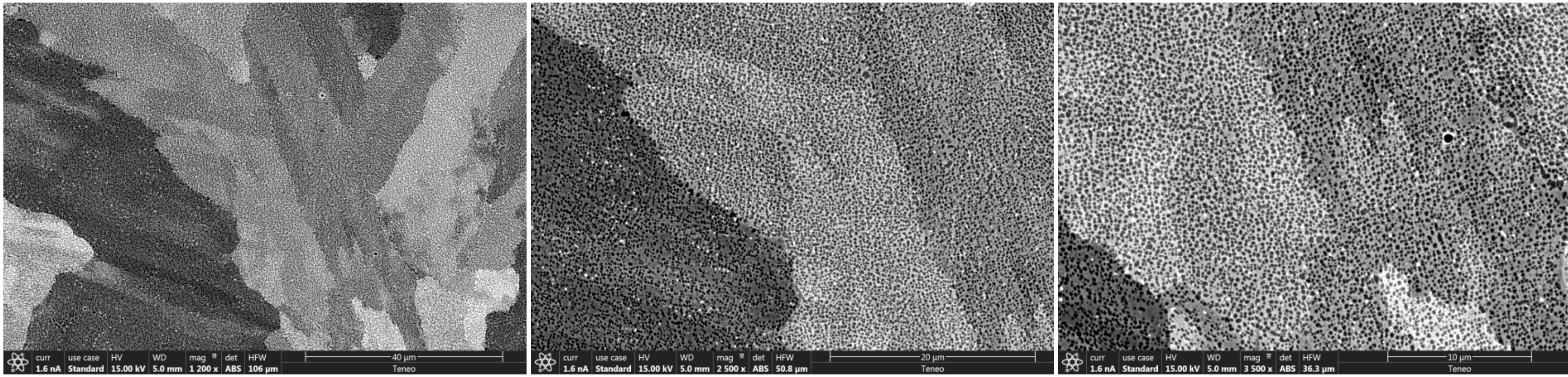
- Secondary gamma prime?
- Coarsening of primary gamma prime along the boundaries
- Gamma prime “finger-like” features could be due to instabilities during AM processing, change in local composition, arrest by solutes, etc

SEM - Inconel 738 – R5

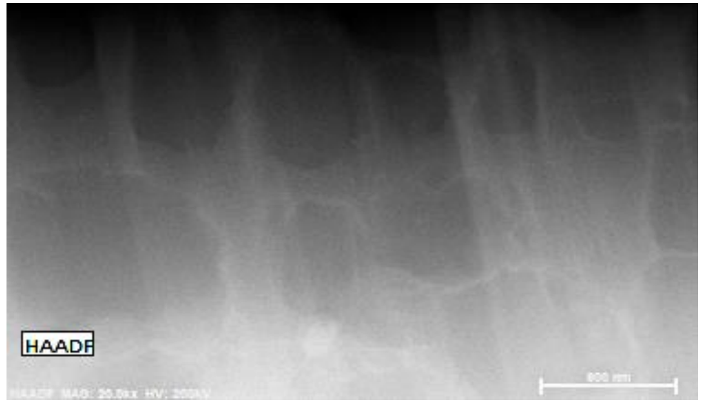
Top

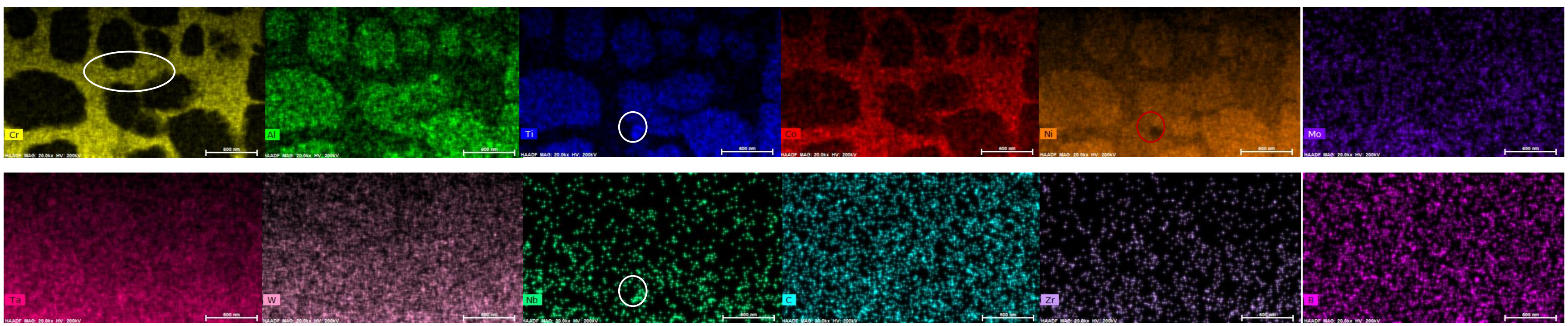


Bottom



TEM EDS



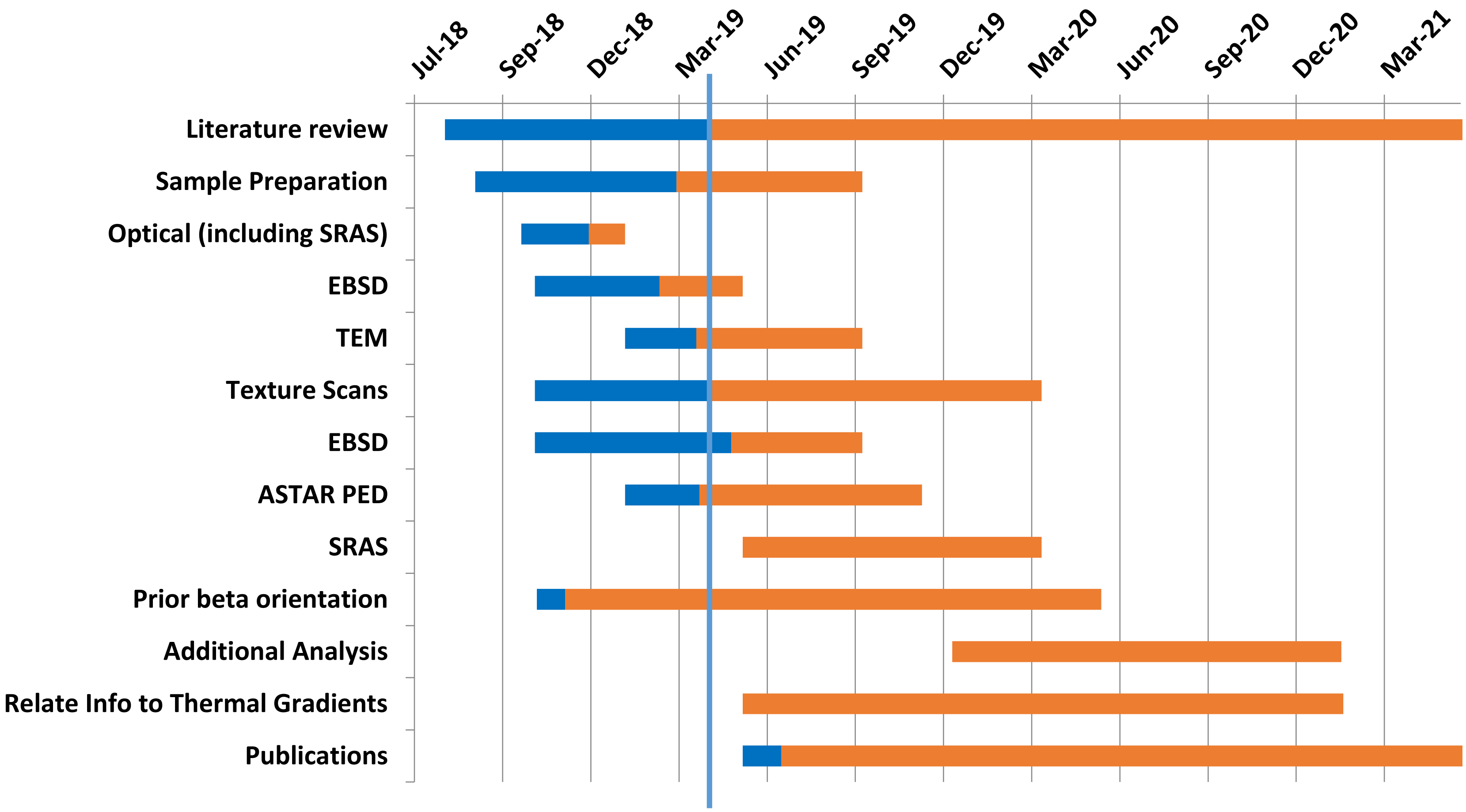


Ni depleted zones correspond to Ti- and Nb-rich zones.
Ti map shows a Ti precipitate, but not clear if it is (Ti,Nb)C or (Ti, Nb)boride.
Cr map shows some precipitates, it is not evident if they are carbides or borides.

Inconel 738

- Top has coarser morphology as compared to the bottom of the build as expected
- More cuboid precipitates at bottom
- Finer gamma, finer gamma prime in bottom than top
- Carbides/borides appear consistently throughout the microstructure

Progress


Jul-18
Sep-18
Dec-18
Mar-19
Jun-19
Sep-19
Dec-19
Mar-20
Jun-20
Sep-20
Dec-20
Mar-21
Literature review
Sample Preparation
Optical (including SRAS)
EBSD
TEM
Texture Scans
EBSD
ASTAR PED
SRAS
Prior beta orientation
Additional Analysis
Relate Info to Thermal Gradients
Publications

Plans in coming months

- EBSD data will be collected on Inconel sample.
- Detailed analysis of the texture data from EBSD will be carried out to determine - the prior β orientation, reconstruction of β grain size, volume fraction of the phases, α laths size and aspect ratio, etc.
- PED ASTAR scans on FIB foils will be carried out for texture data at nano-scale and to quantify the defect density
- SRAS scans will be performed when the system is operational
- An understanding will be developed to related the thermal gradients with the current information of microstructure and texture evolution with different scan strategies and along the build direction

Thank you!

Matt Kenney
mjkenney@iastate.edu