CANFSA

Center for Advanced Non-Ferrous Structural Alloys

An Industry/University Cooperative Research Center

Project 34: In-situ Observation of Phase and Texture Evolution Preceding Abnormal Grain Growth in Ni-based Aerospace Alloys

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Colorado School of Mines, Golden, CO
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Project 34: In-situ Observation of Phase and Texture Evolution Preceding Abnormal Grain Growth in Ni-based Superalloys

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- Student: Byron McArthur (Mines)
- Advisors: Amy Clarke, Kester Clarke (Mines)

Project Duration

PhD: Nov 2017. to Dec. 2020

- Problem: Abnormal grain growth (AGG) in Ni-based superalloys (RR-1000) significantly reduces mechanical properties and occurs as a result of forging parameters.
- Objective: Determine the mechanism of abnormal grain growth in Ni-based superalloys using ex-situ and in-situ characterization techniques.
- Benefit: Improved mechanical properties for turbine disk alloys.

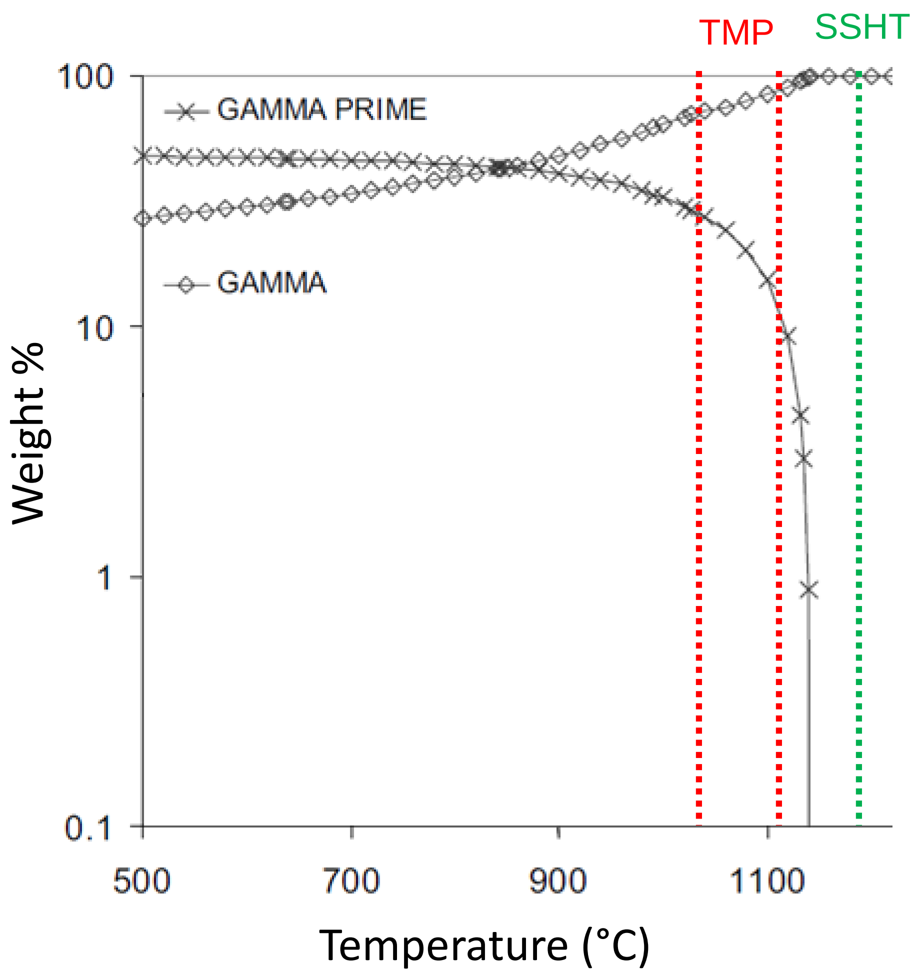
Recent Progress

- Performed microstructural simulations to evaluate effects of soft impingement on matrix grain size
- Internship at Air Force Research Labs
- Developing theory of AGG

Metrics		
Description	**% Complete**	**Status**
1. Literature review	75%	●
2. Explore abnormal grain growth forging parameters for RR1000	75%	●
3. Ex-situ and interrupted material testing and characterization	50%	●
4. Develop and test theory to explain abnormal grain growth phenomena	50%	●
5. Perform in-situ microscopy with a synchrotron source (HEDM) to demonstrate phenomena	0%	●

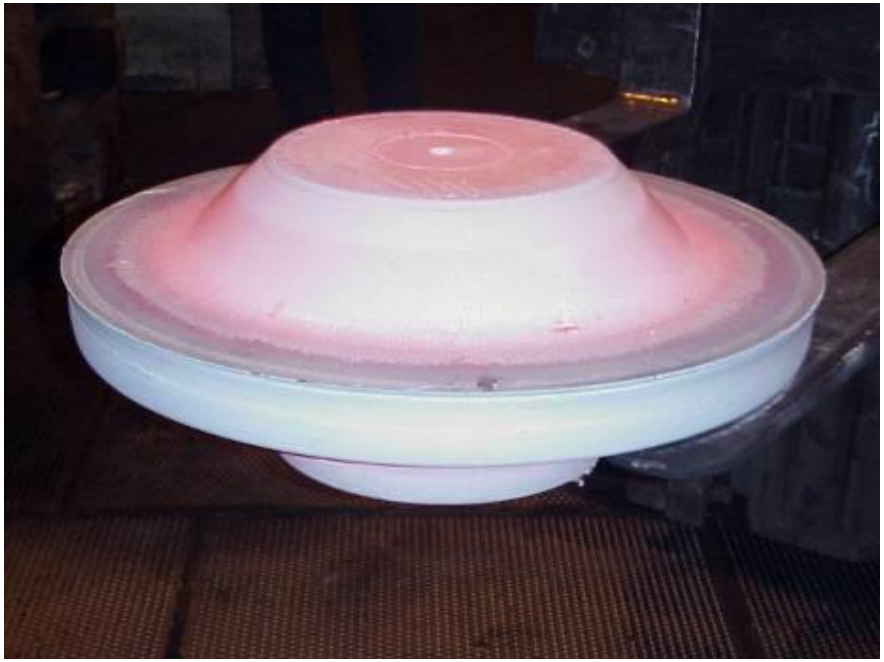
Material: RR-1000, γ-γ’

- Processing:
  - Powder metallurgy
  - Hot isostatic pressure compaction
  - Extruded at 5:1 ratio
  - Isothermal forging: 1035-1110°C
    - Performed in Gleeble®
  - SSHT: 1150-1170°C
    - Performed in dilatometer
- Critical AGG parameters:
  - Strain
  - Strain rate
  - Heating rate to super solvus hold
  - Forging temperature



Isothermal Forging

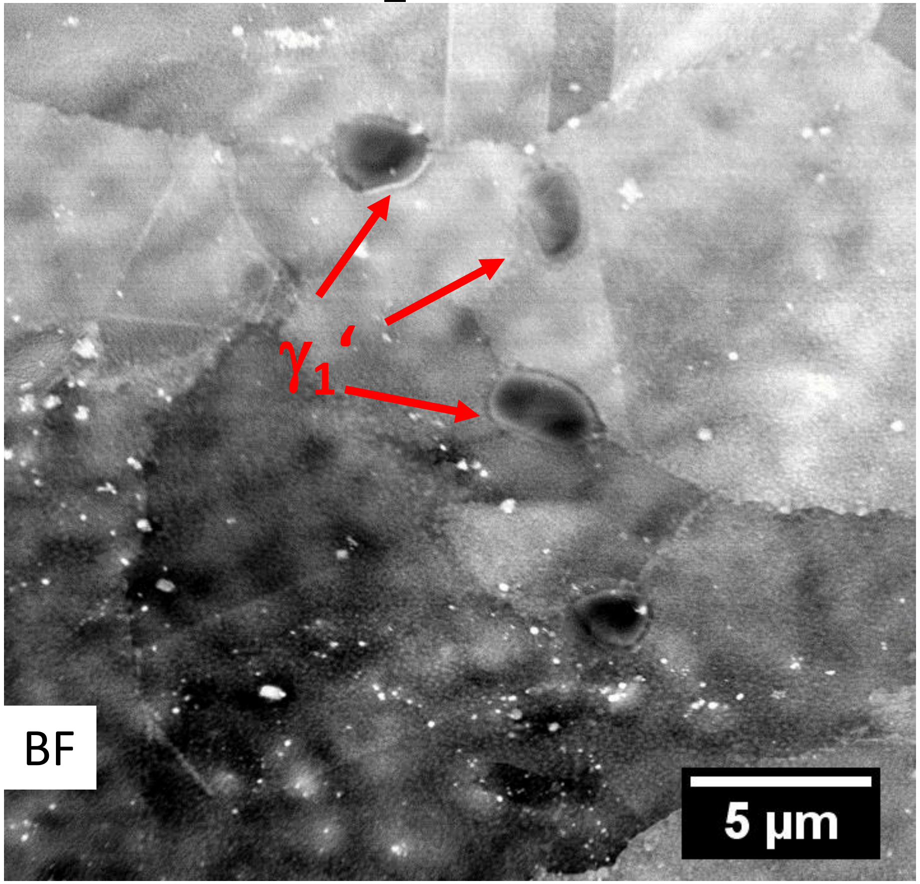
- Sub- γ’ solvus temperature
- Low strain rate
- Maintain superplastic deformation for decreased forging loads
- Primary γ’ pins γ grain boundaries
  - Secondary γ’ less effective or dissolved
- Low stored energy accumulation
  - Grain boundary sliding (Coble creep)
  - Dynamic recovery
  - Dynamic recrystallization



Mitchell, R. J., Lemsky, J. A., Ramanathan, R., Li, H. Y., Perkins, K. M., & Connor, L. D. *Superalloys 2008, pp.* 347–356.

As-received Material

Low γ_1' Fraction



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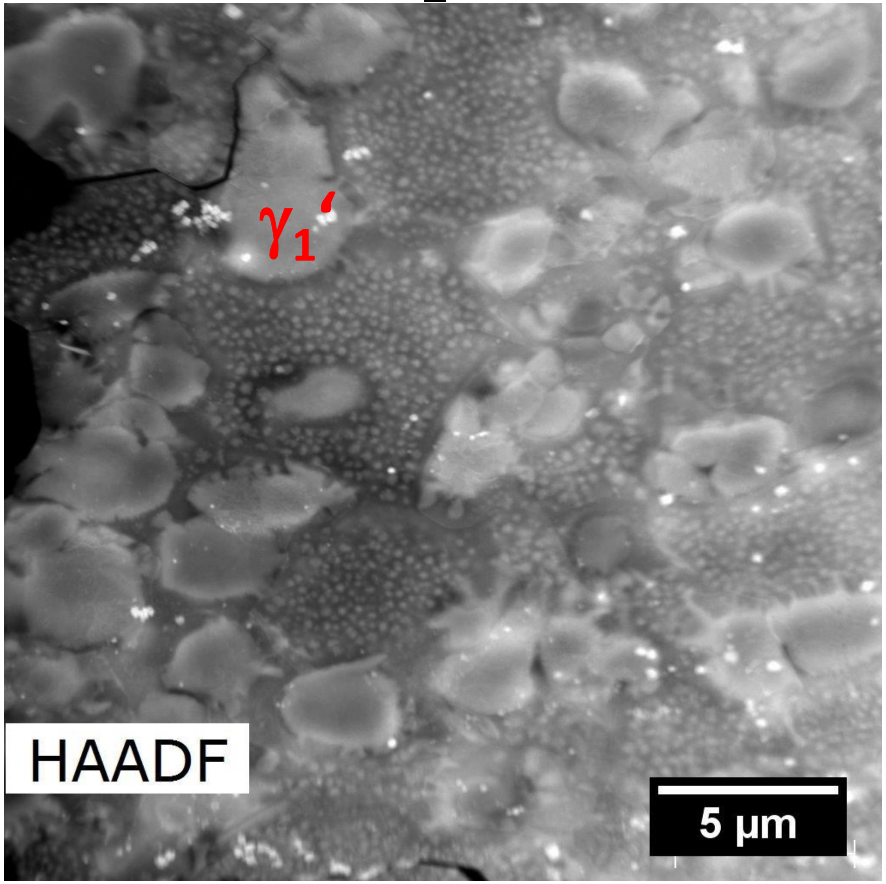

Thanks to Yaofeng Guo for TEM imaging

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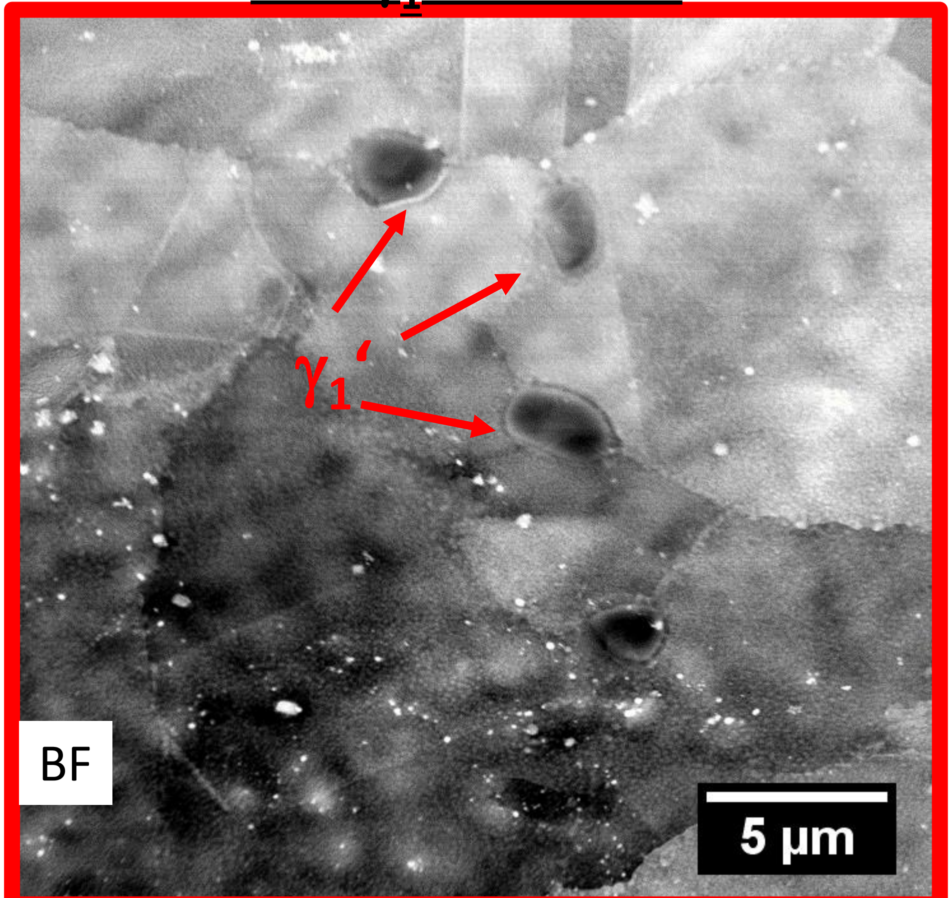

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Experimental Procedure

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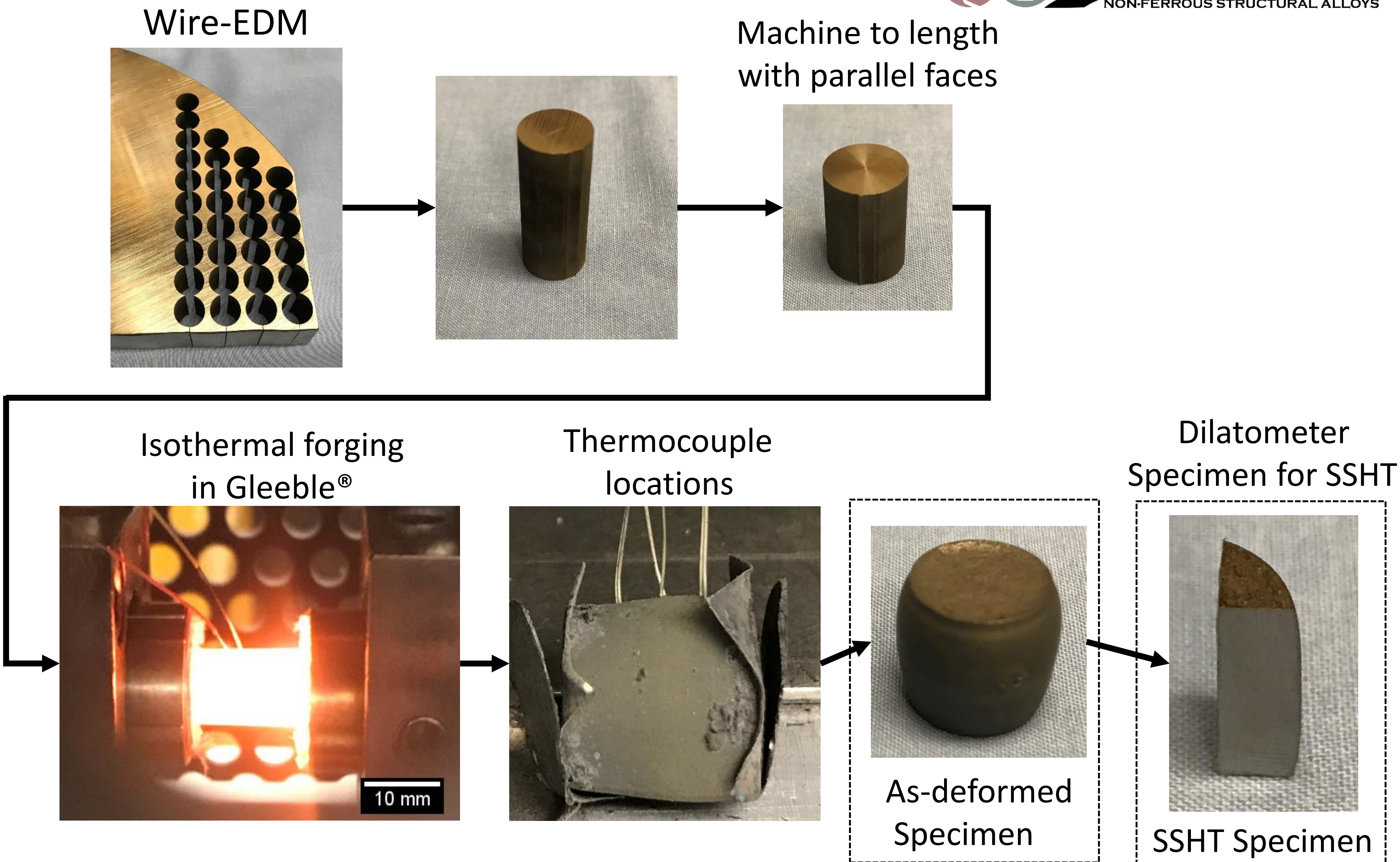
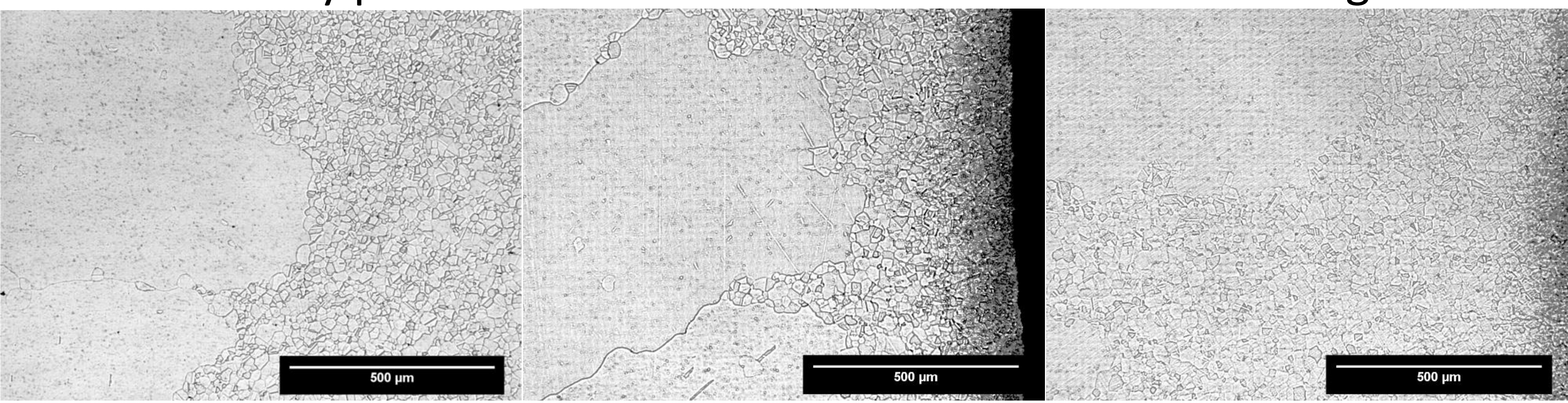


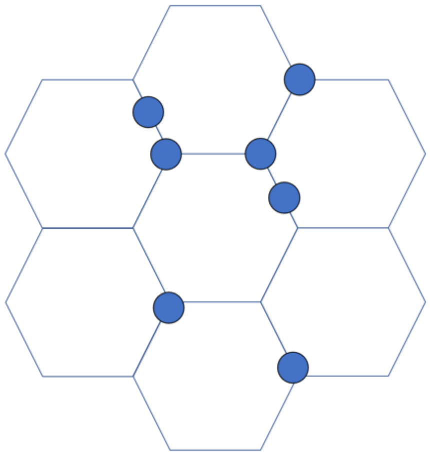
Summary of Prior Work & Results

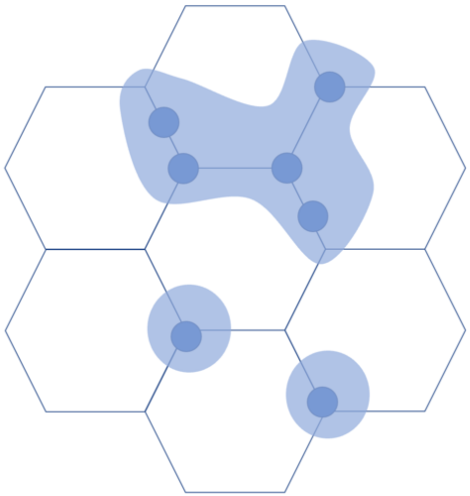
- Replicate industrial processing that leads to AGG in lab-scale testing
- Determine influential processing parameters and their roles
  - Varying parameters shift AGG to regions of more/less stored energy
- Consistently produce AGG via Gleeble TMP and heat treating

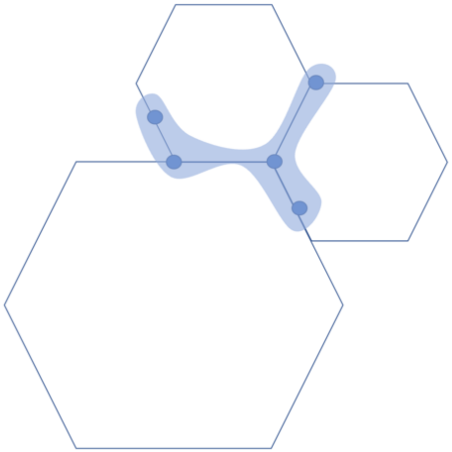


- Begin exploring observation and characterization techniques
  - Ex-situ, interrupted, and in-situ

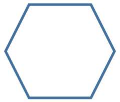
Soft Impingement Theory

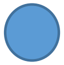


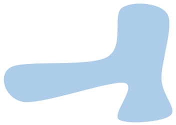




- Secondary γ′ dissolved
- Spatial and size distribution of primary γ′ creates solute (Cr) clouds/clustering
- Soft impingement slows dissolution rates of γ′ locally
- γ grains in areas of lower primary γ′ density grow more rapidly to create AGG

 = γ grain

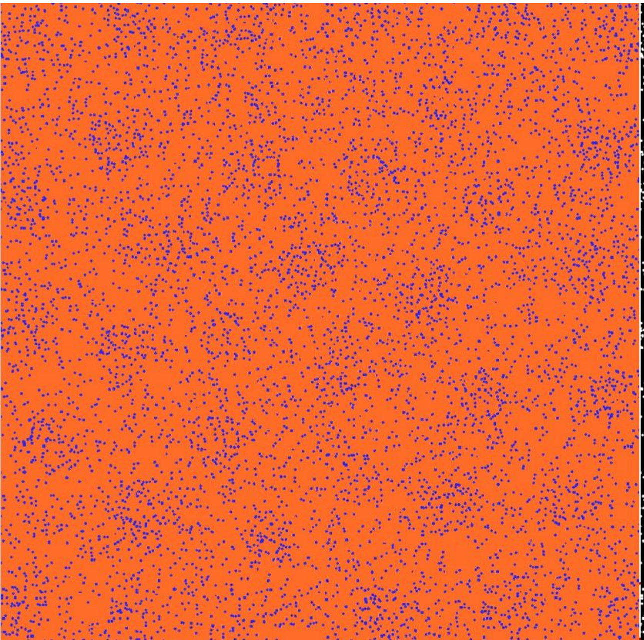
 = γ′ grain

 = Solute cloud

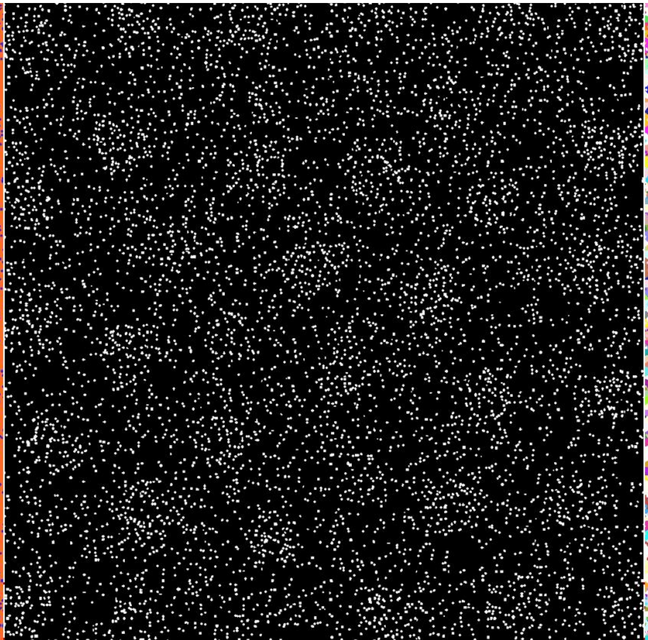
Soft Impingement Modeling

- Evaluate effects of spatially clustered γ' on γ grain size distribution during heat treating
- Initial microstructure of γ' within a γ matrix created with Dream.3D
- Evolved microstructure with a hybrid model
  - SPPARKS, Hybrid Potts Phase-field
- Figures represent ~500x500µm region of material
  - Simulation is unitless

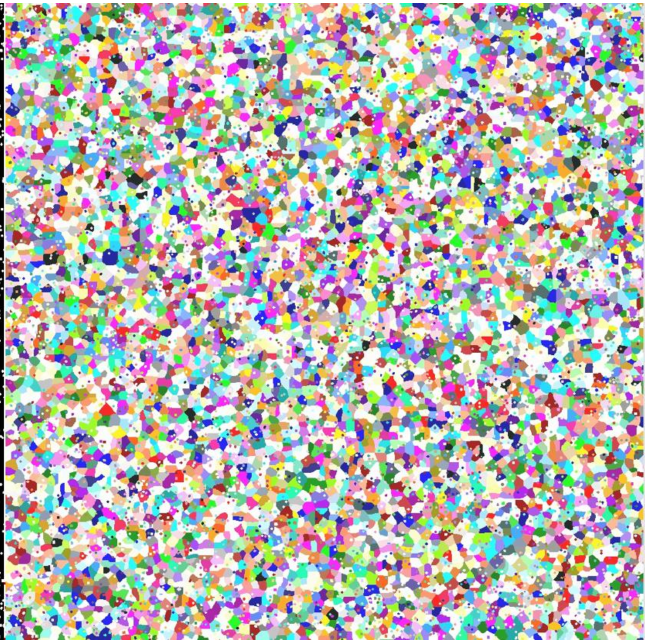
Composition



Phase



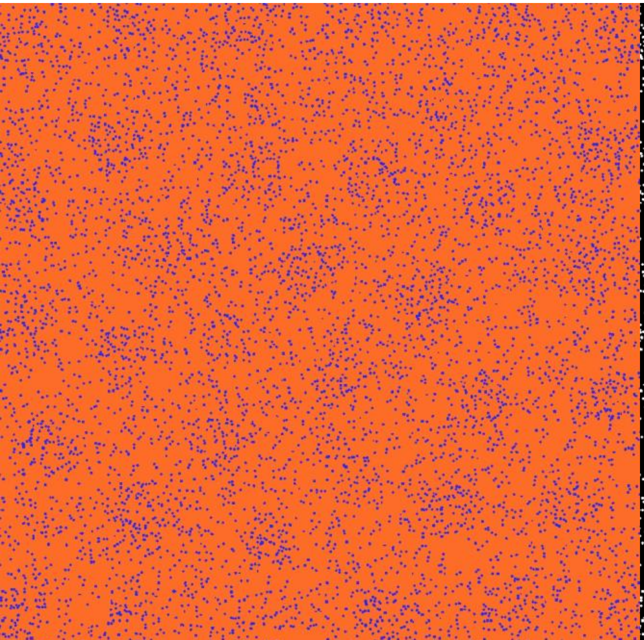
Grain ID



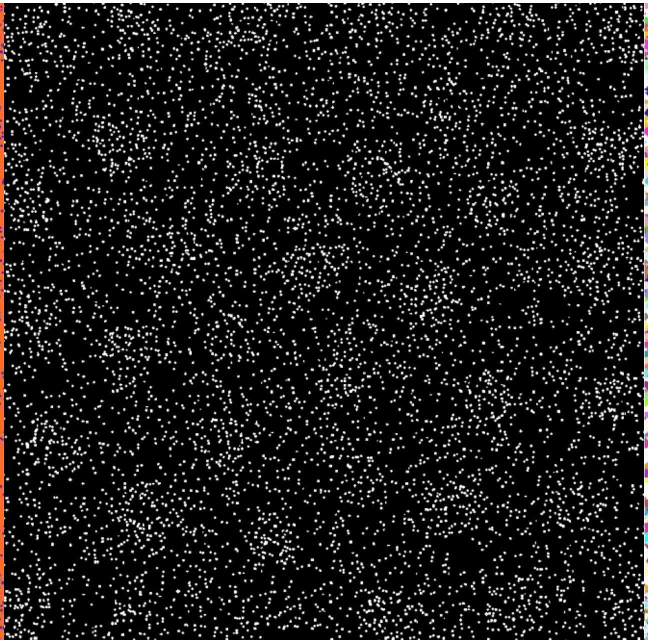
Soft Impingement Modeling

- Spatial clustering delays precipitate dissolution
- Precipitates effective at pinning matrix grain boundaries
- Bimodal matrix grain size temporarily
- Matrix grain size equalizes

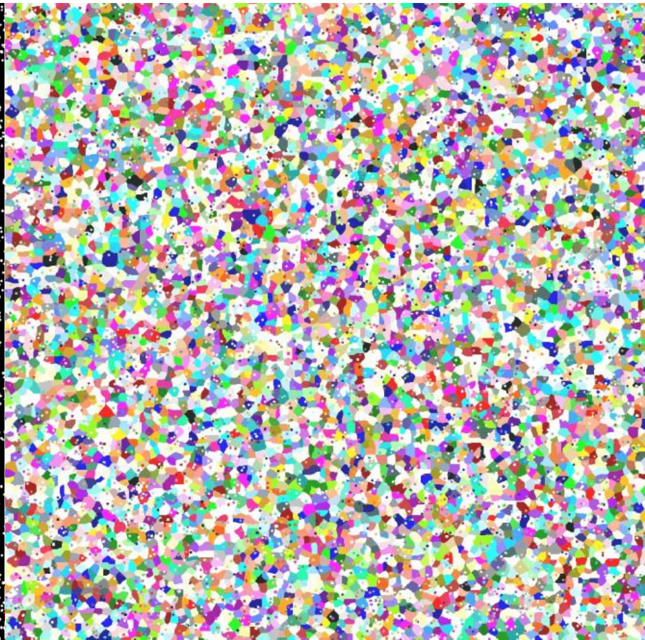
Composition



Phase



Grain ID



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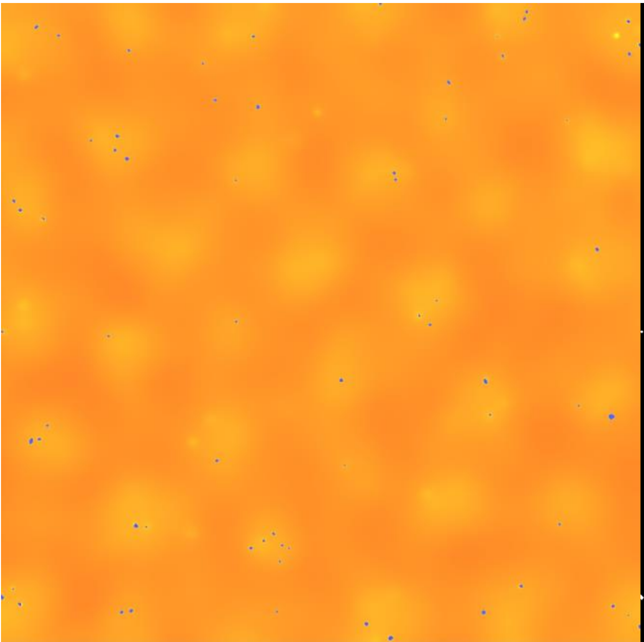
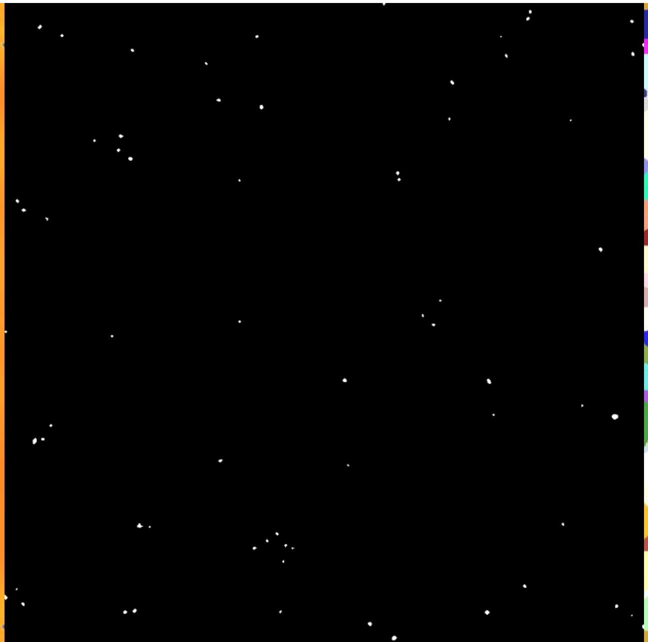
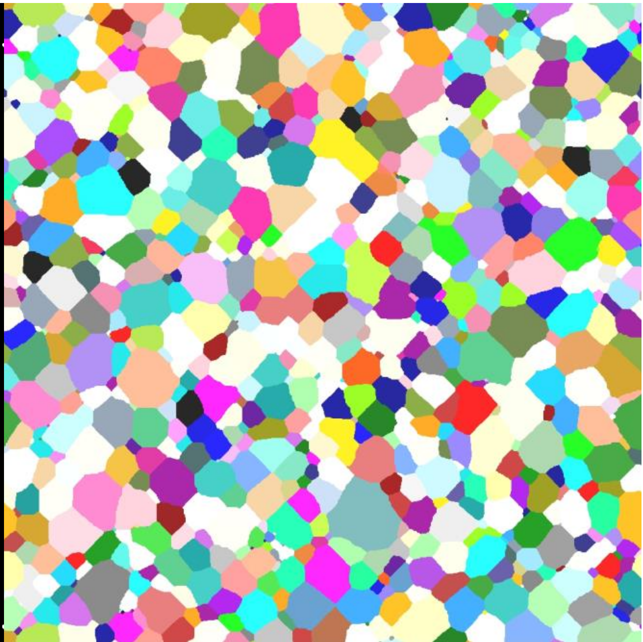


Phase



Grain ID



Soft Impingement (Cont.)

- Possible spatial clustering of γ' due to elemental segregation in alloyed powder or deformation assisted γ' dissolution may create regions of lower γ' phase fraction
- Leads to increased local γ grain size in early stages of grain growth that could propagate during heat treating
- Modeling accounts for boundary energy, but no stored energy contributions as a driving force to accelerate grain growth
- Current experimental results do not appear to support this theory
  - Low deformation and strain rate required
  - More work needed

Coherent γ Recrystallization

Hetero-epitaxial Recrystallization (HERX) (Charpagne)

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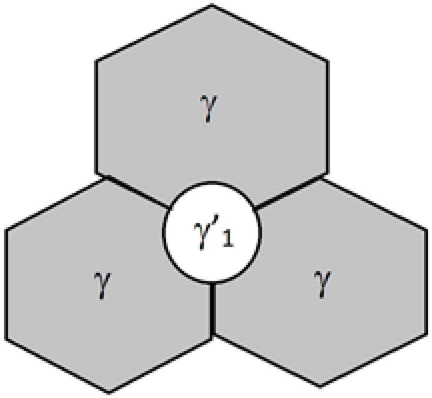
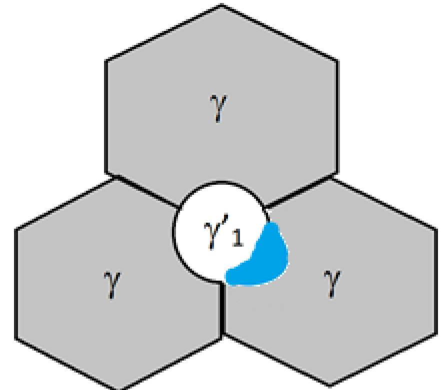
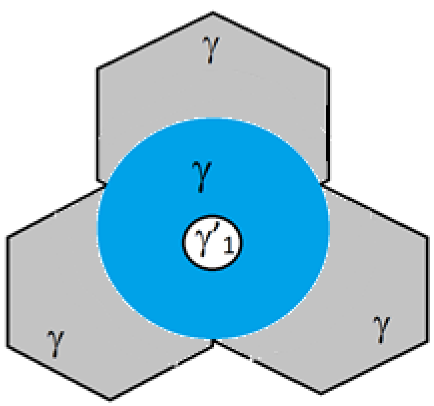


γ'_1 is incoherent with neighboring γ



γ nucleates coherently off γ'_1 or during order-disorder transformation



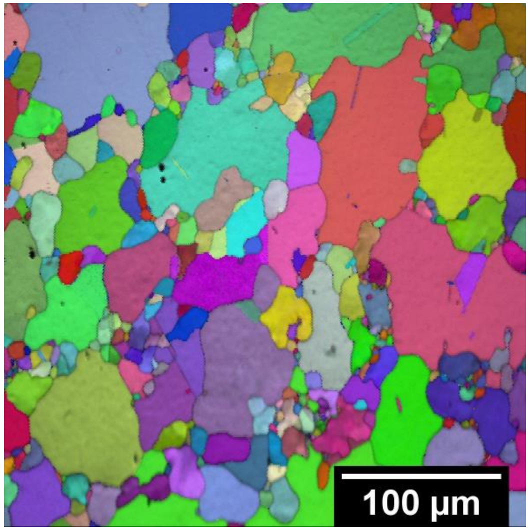
Growth to consume stored energy in γ

- Possible γ- γ' disorder-order transformation induced, or coherent nucleation
- Reduced energy barrier for nucleation
- Explains required heating rate
  - HERX occurs before RX
  - Growth until hard impingement
- γ'_1 serves as nucleation sites
- Different than particle stimulated nucleation

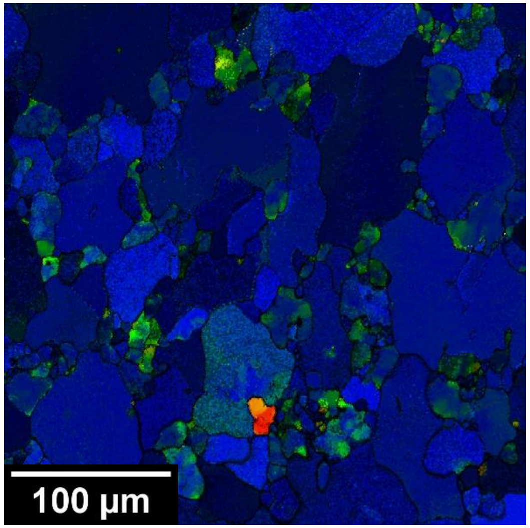
Thermomechanical Processing and Characterization Plans

- Perform "Step-Load" isothermal forging at various strains to map deformation mechanisms and relate to AGG conditions
- Interrupted heat treating to determine changes in stored energy during recovery, recrystallization, and grain growth
  - Sub-, super-, and critical strain rates for AGG
- Special attention to stored energy near primary- γ'

Orientation Map



Grain Reference Orientation Deviation



Future Work

Stored Energy Characterization

- Analyze the γ and γ' dislocation structures
  - As received, forged, interrupted heat treated, final
  - TEM & SEM-EBSD
  - Identify instances of HERX (dynamic or static)

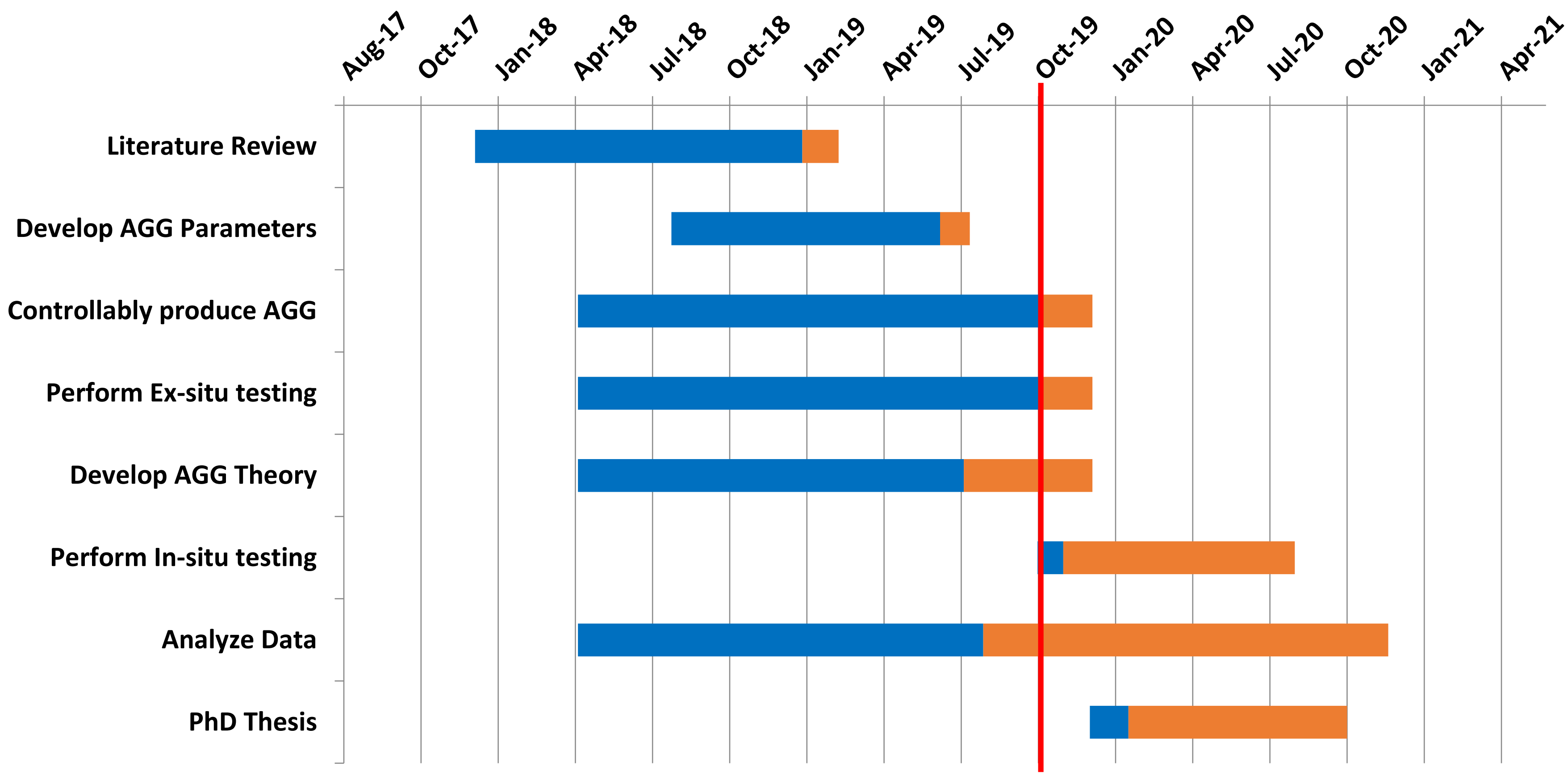
Interrupted Processing

- Perform heat treating of AGG producing material
- Track growth of γ and dissolution of γ' approaching γ'-solvus

γ' Spatial Distribution

- Quantify distribution of γ' throughout material
  - Track dissolution during TMP & heat treating

Progress


Aug-17
Oct-17
Jan-18
Apr-18
Jul-18
Oct-18
Jan-19
Apr-19
Jul-19
Oct-19
Jan-20
Apr-20
Jul-20
Oct-20
Jan-21
Apr-21
Literature Review
Develop AGG Parameters
Controllably produce AGG
Perform Ex-situ testing
Develop AGG Theory
Perform In-situ testing
Analyze Data
PhD Thesis

Challenges & Opportunities

- Working on improving conventional TEM foil preparation technique
  - 20:80 Perchloric:Methanol, -20°C, 16-25V, 80µm foil
  - More area than FIB liftouts
- Improving Gleeble capabilities
  - Reducing thermal gradients (Kaowool wrapped specimen)
  - Adjust PID settings for high temperature, low displacement rate
- Contemplating adding model alloys
  - Compare effects of ordering by creating CuAu ($L1_2$ ordered) or AlAg (disordered) systems with second phase precipitates
  - Simple binary system may show HERX in a more controlled manner, with similar precipitate dissolution rates

Thank you!

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