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

Spring Meeting

April 7th – 9th 2020

Student: Byron McArthur (Mines)

Faculty: Amy Clarke, Kester Clarke, Michael Kaufman (Mines)

Industrial Mentor: Eric Payton (AFRL), Kevin Severs (ATI)

NSF | Colorado School of Mines | Iowa State University

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, Michael Kaufman (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.

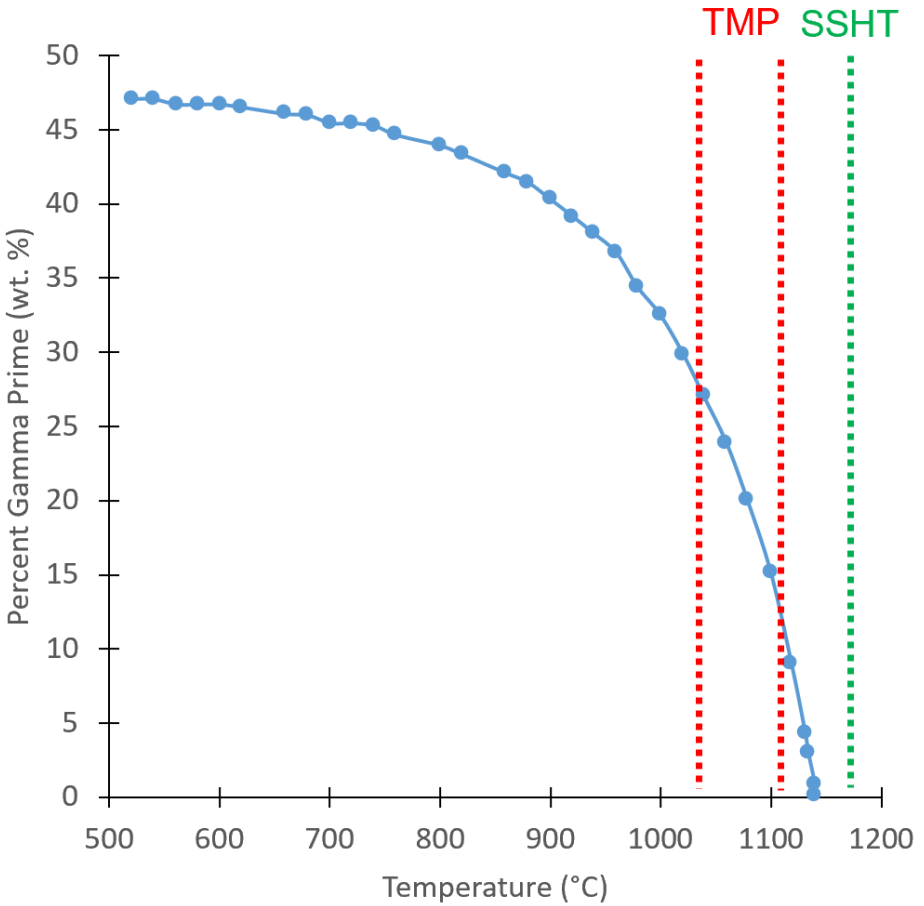
Recent Progress

- Developing mechanistic theory for AGG
- Strain rate sensitivity testing of RR1000
- Performing mesoscopic model testing of AGG theory

Metrics		
Description	**% Complete**	**Status**
1. Literature review	90%	●
2. Explore abnormal grain growth forging parameters for RR1000	90%	●
3. Ex-situ and interrupted material testing and characterization	50%	●
4. Develop and test theory to explain abnormal grain growth phenomena	75%	●
5. Perform model testing to observe mechanisms	50%	●

Material: RR-1000, γ-γ'

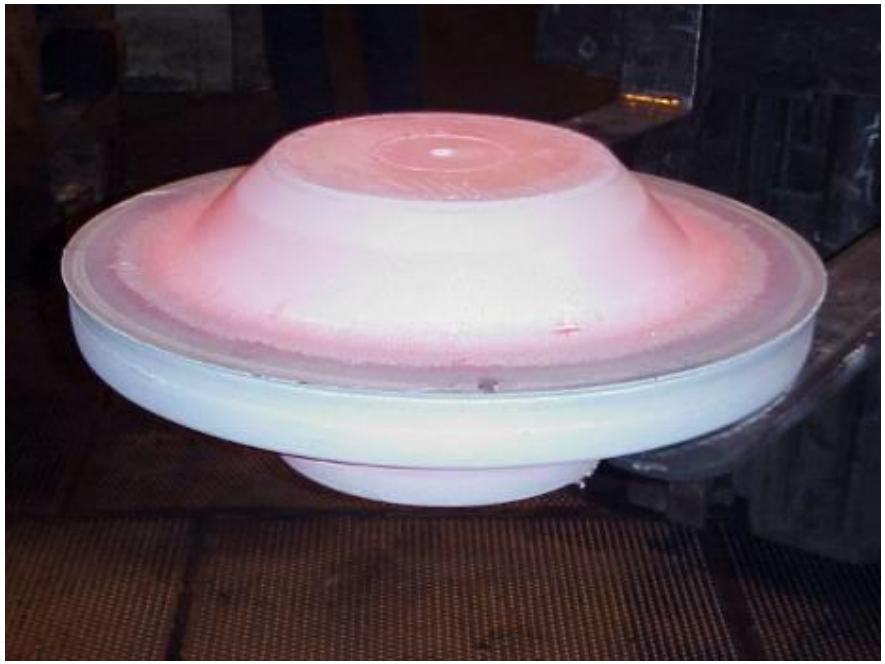
- Processing:
  - Powder metallurgy
  - Hot isostatic pressure compaction
  - Extruded at 5:1 ratio
  - Isothermal forging: 1035-1110°C
    - Performed in Gleeble®
  - Super solvus heat treatment (SSHT)
    - 1150-1170°C
    - Performed in dilatometer
- Critical AGG parameters:
  - Strain
  - Strain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



M.C. Hardy, B. Zirbel, G. Shen, R. Shankar. Developing damage tolerance and creep resistance in a high strength nickel alloy for disc applications, Superalloys 2004 83-90 (2004).

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

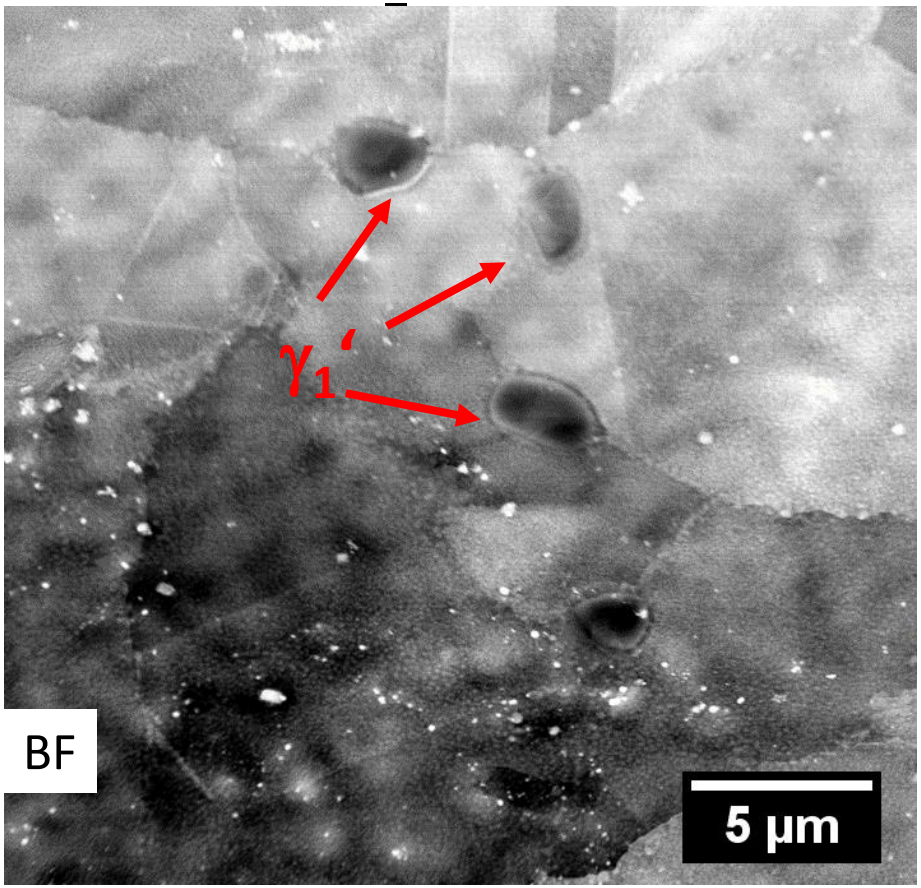


Forging ~1 Meter Diameter

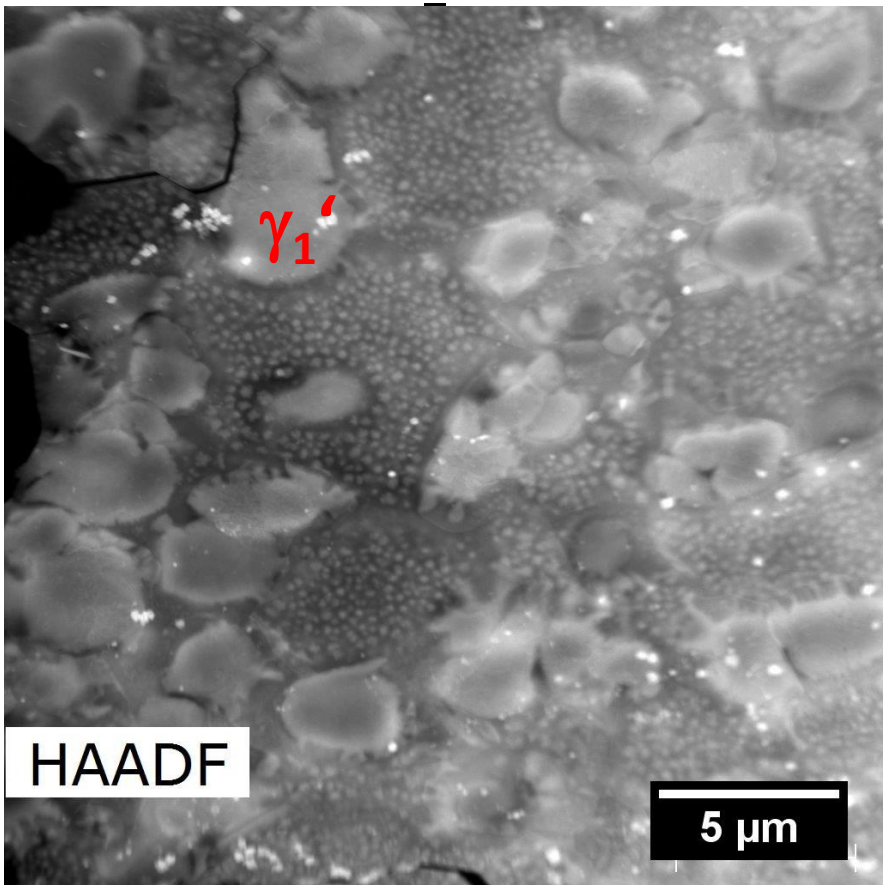
Mitchell, R. J., Lemsky, J. A., Ramanathan, R., Li, H. Y., Perkins, K. M., & Connor, L. D. *Superalloys 2008, pp.* 347–356.

As-received Material

Low γ_1' Fraction



High γ_1' Fraction



Thanks to Yaofeng Guo for TEM imaging

As-received Material

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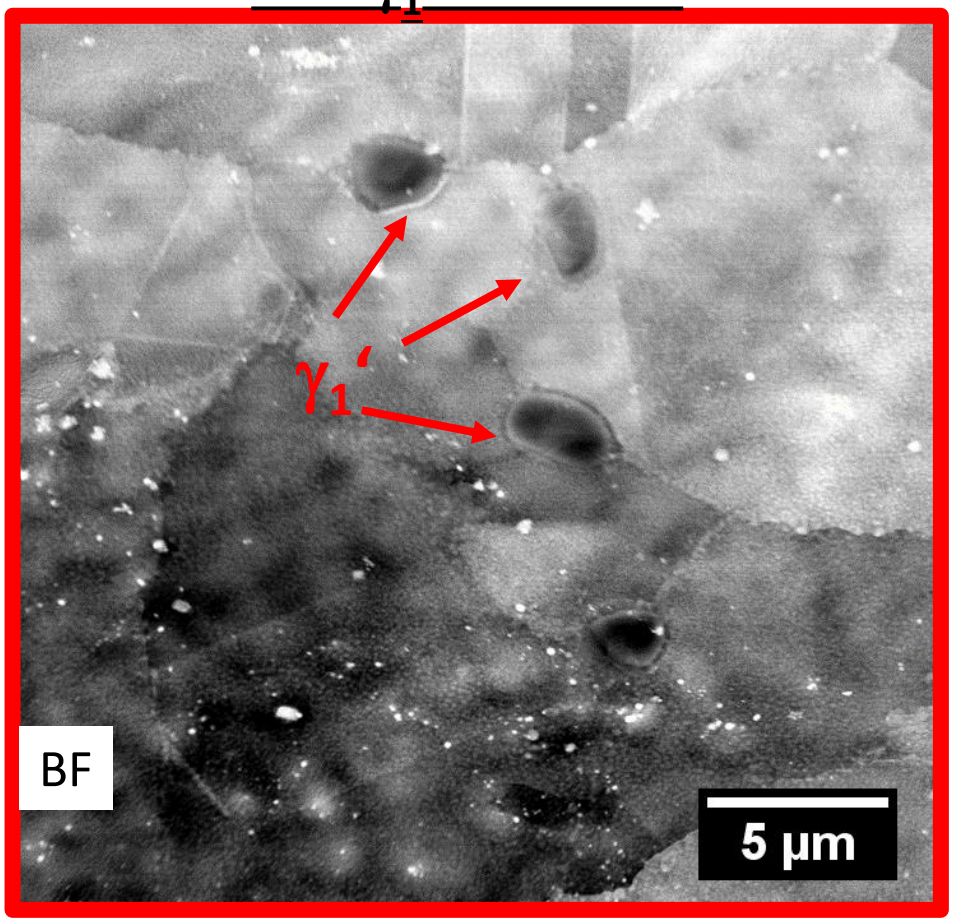
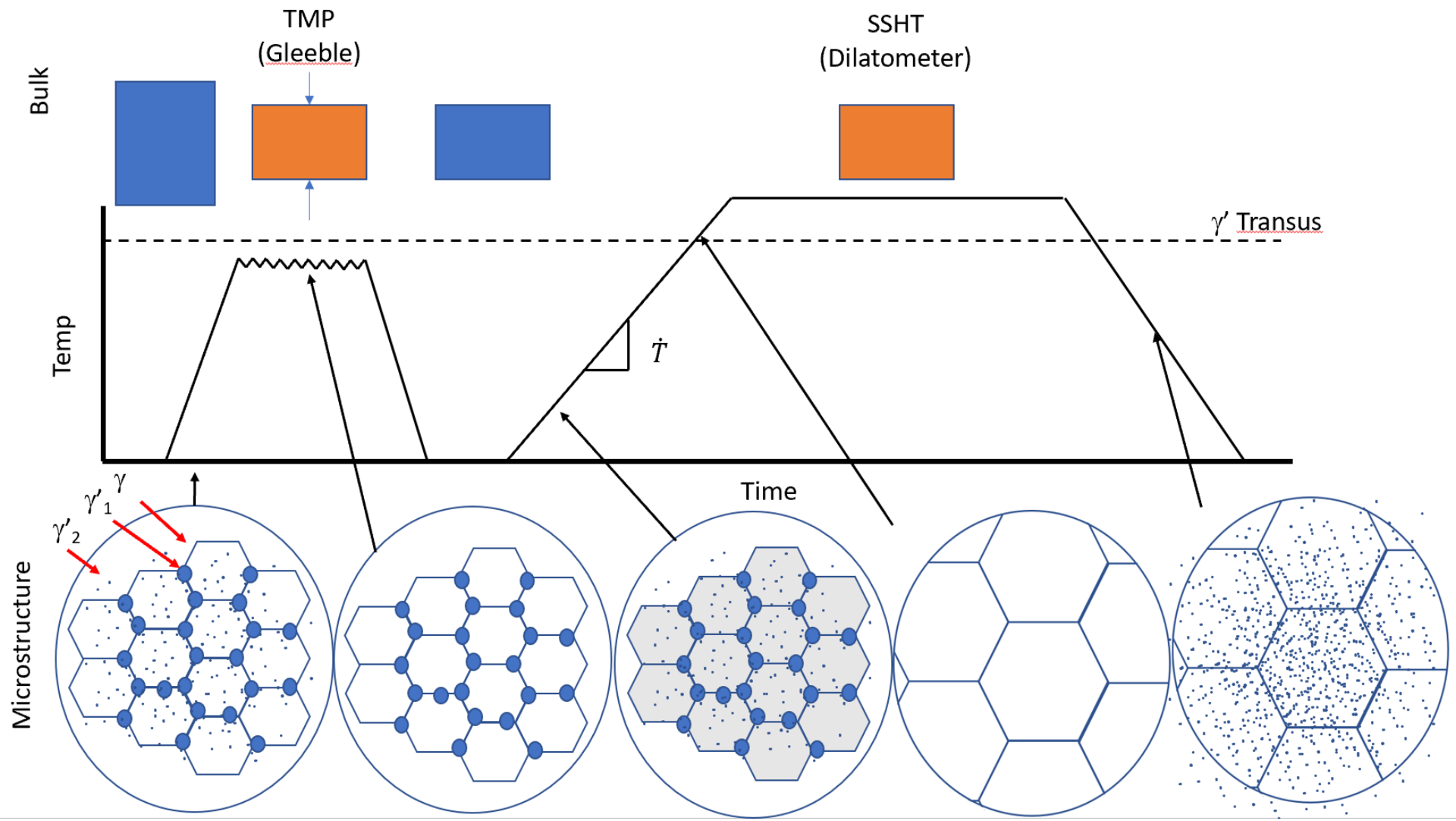
Low γ_1' Fraction



- Lower γ_1' demonstrated AGG
- Higher γ_1' showed no AGG
- γ' size influences deformation mechanism, stored energy, and recrystallization
- Higher γ_1' may be used later for further development or support of AGG theory

Thanks to Yaofeng Guo for TEM imaging

Processing and Microstructure



Experimental Procedure

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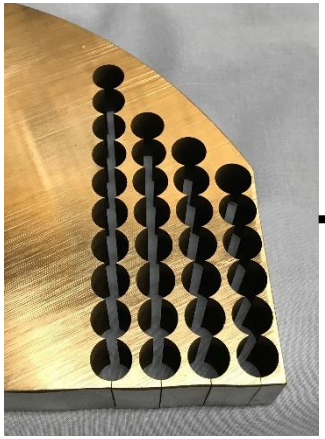
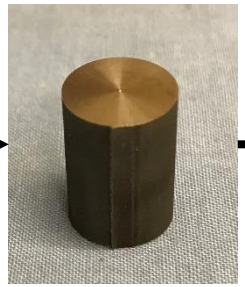
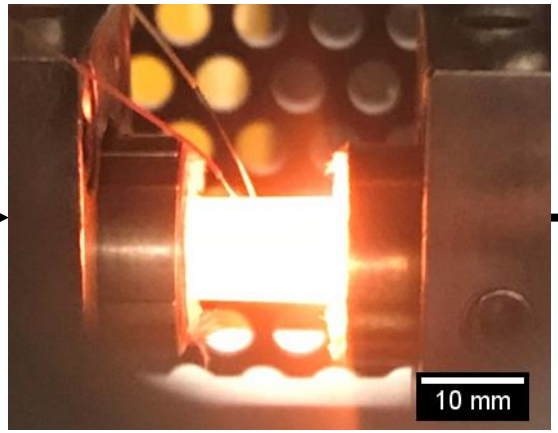
Wire-EDM



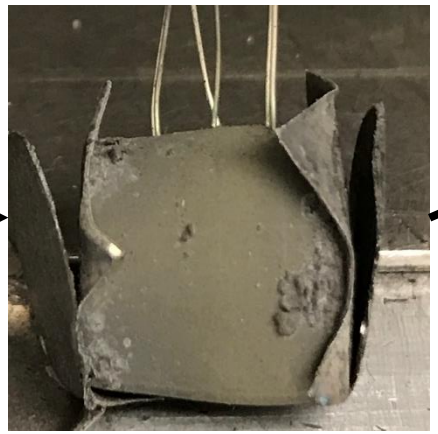
Machine to length with parallel faces



Isothermal forging in Gleeble®



Thermocouple locations



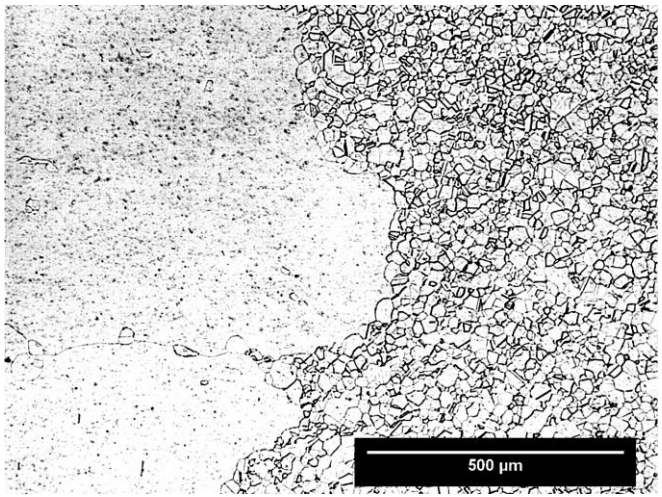
As-deformed Specimen

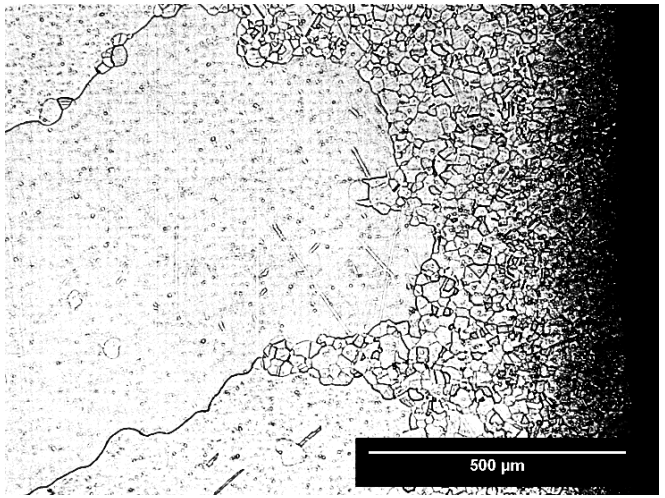
Dilatometer Specimen for SSHT

SSHT Specimen

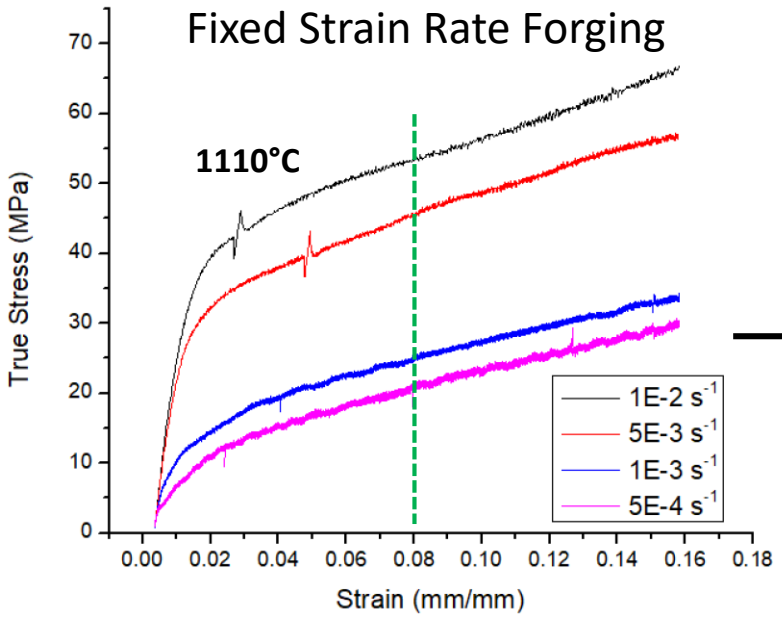
Summary of Prior Work & Results

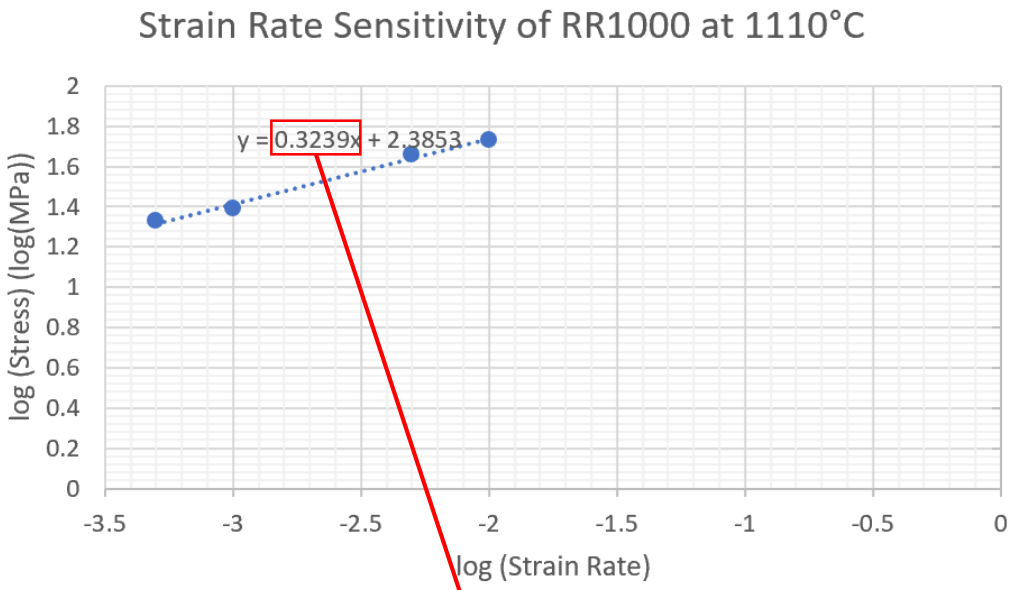
- Replicate industrial processing that leads to AGG in lab-scale testing
  - High temperature, low strain & strain rate, low super-solvus heating rate
- Determine influential processing parameters and their roles
  - Varying parameters shift AGG to regions of more/less stored energy
- Consistently produce AGG via Gleeble TMP and heat treating
- Exploring experimental techniques to detail phenomena

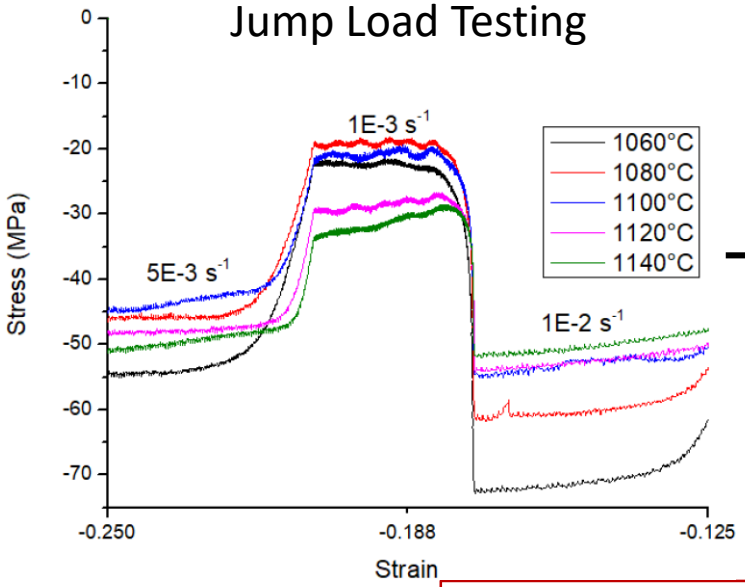


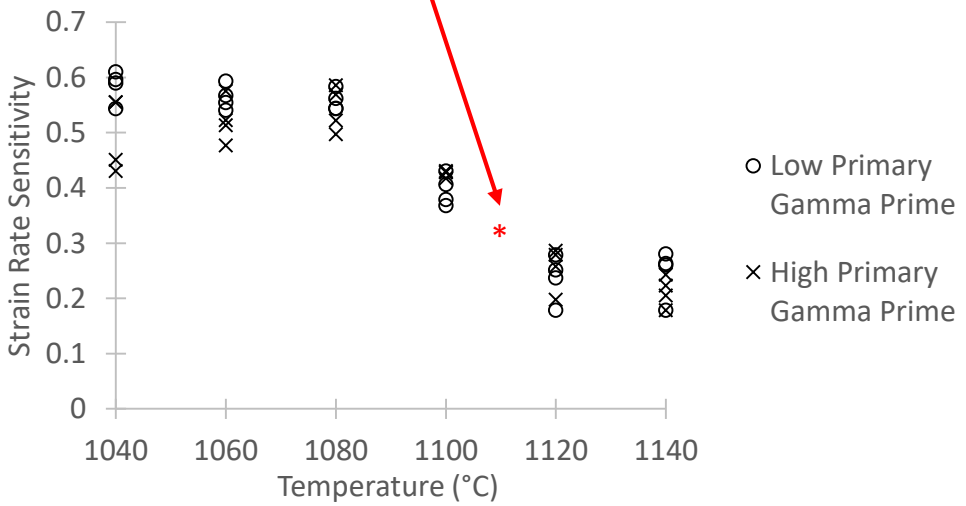


Strain Rate Sensitivity





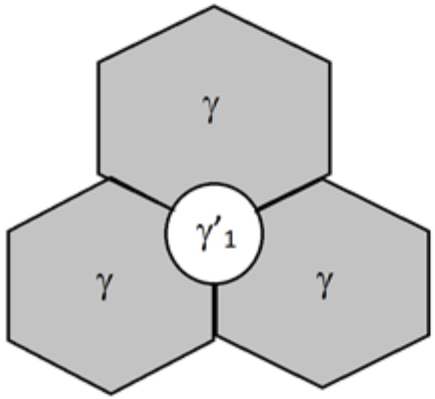




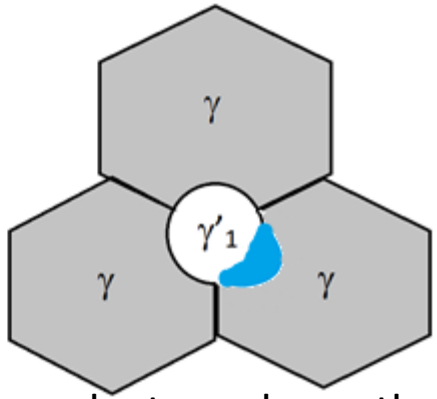
Theory of Abnormal Grain Growth

Coherent γ Recrystallization

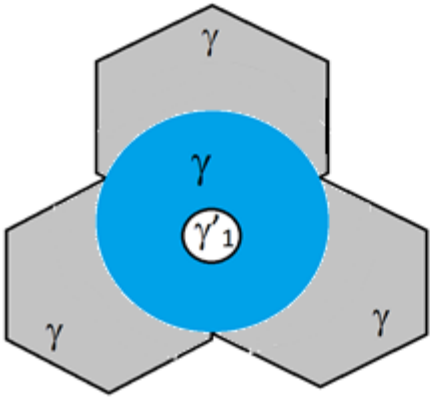
Hetero-epitaxial Recrystallization (HERX) (Charpagne)



γ'_1 is incoherent with neighboring γ



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

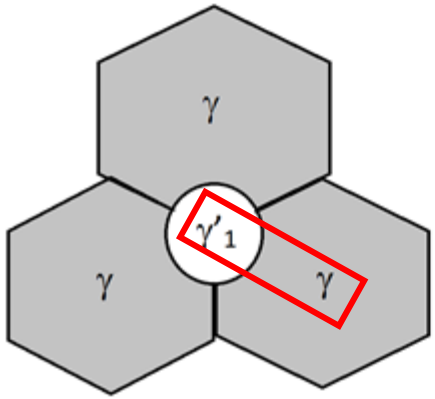


Growth to consume stored energy in γ

- 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

Coherent γ Recrystallization

Hetero-epitaxial Recrystallization (HERX) (Charpagne)



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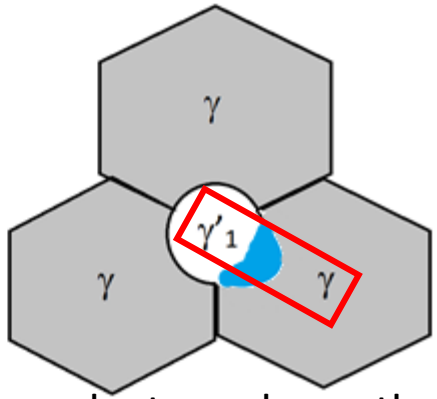

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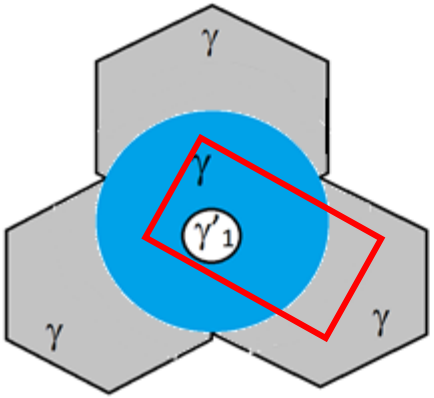
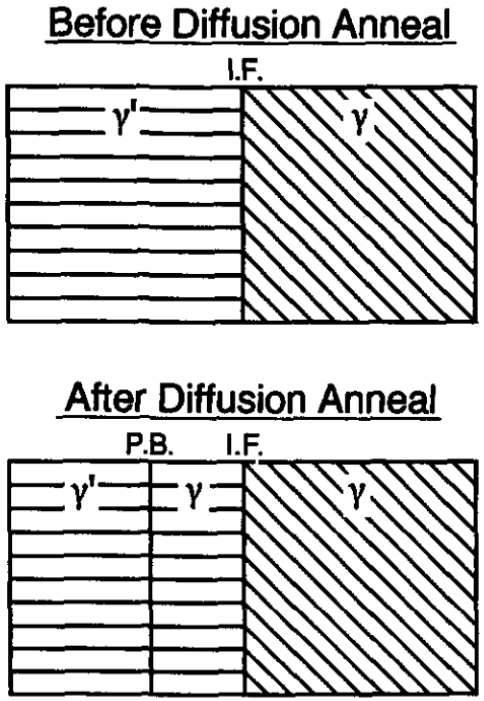


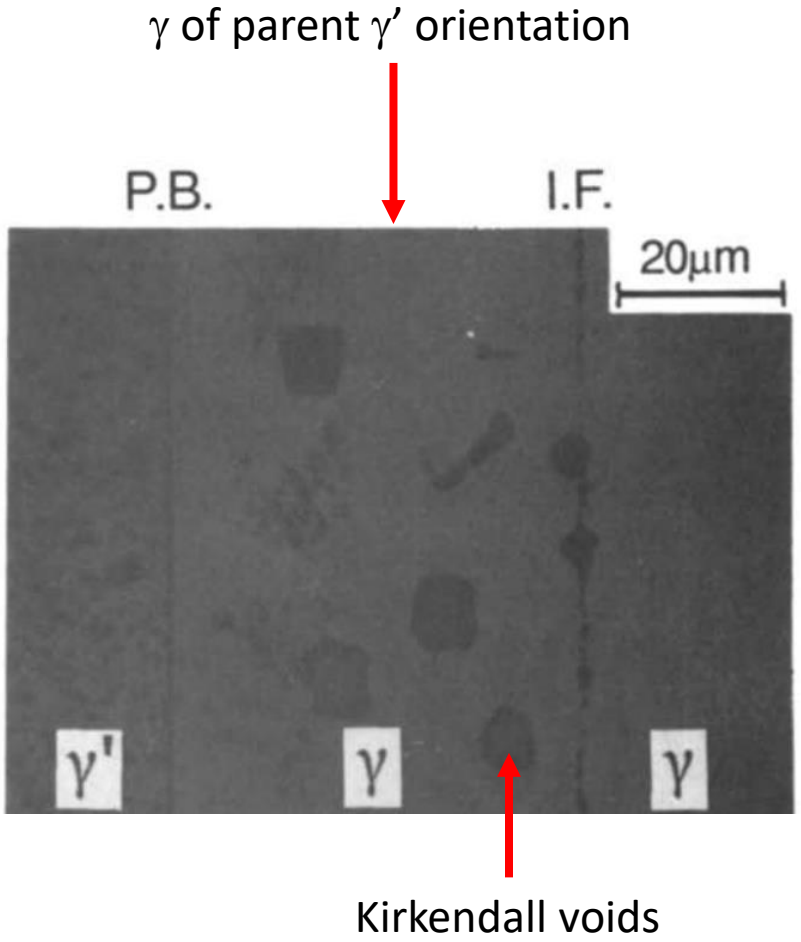
Growth to consume stored energy in γ

- Want to observe microstructural development at micron scale
- Occurs at ~1080-1110°C
- Rapidly in limited instances
- Statistically difficult with 2-D sectioning
- Propose to do mesoscopic model experiments

γ'- γ Diffusion

- Ni3Al-Ni (γ'- γ) diffusion couple annealed
- Al in γ' diffused to γ, forms γ
- New γ of parent γ' orientation
- Supports theory of stable γ- γ boundary formed in location of parent γ'- γ boundary

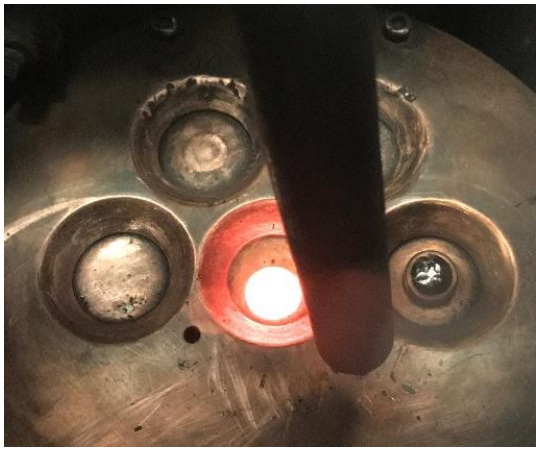


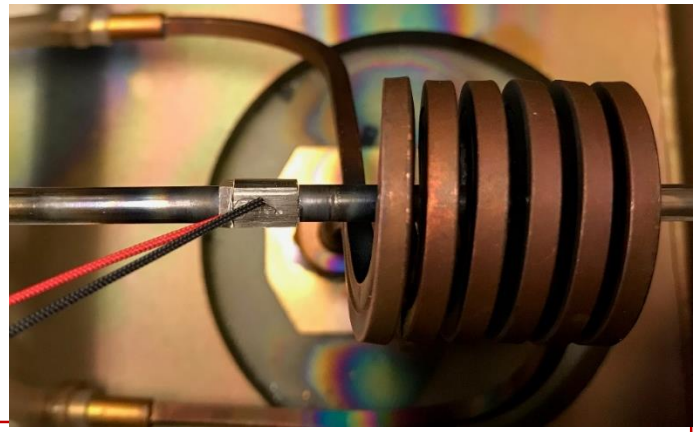


M. Watanabe, Z. Horita, D.J. Smith, M.R. McCartney, T. Sano, M. Nemoto, Electron microscopy study of Ni/Ni3Al diffusion-couple interface-I. Microstructural observation and microchemical analysis, Acta Metall. Mater. 42 (1994) 3381–3387

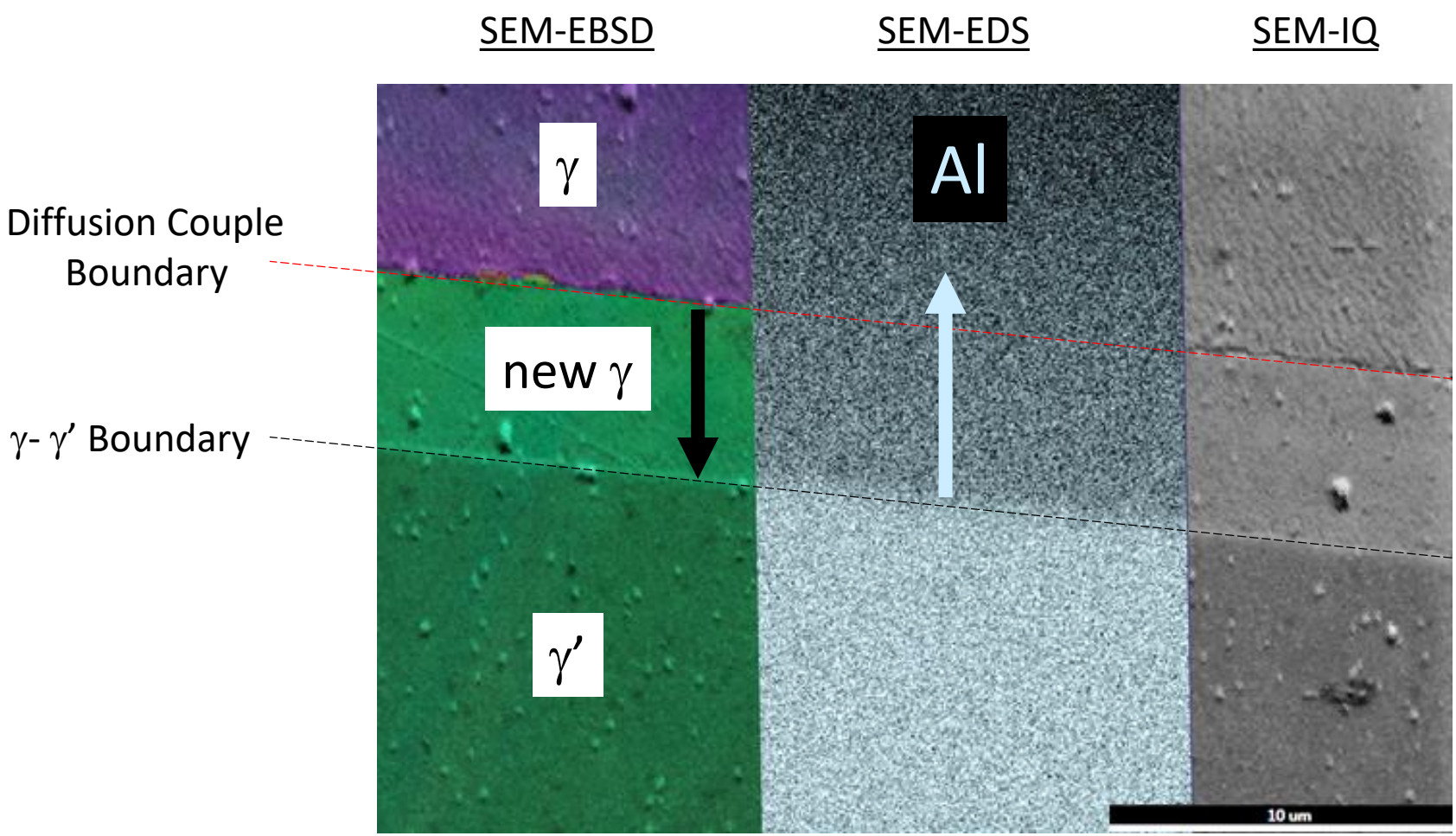
Mesoscopic AGG Study

- Diffusion couple to create γ-γ’ interface
- γ’ created through Ni_3Al button melted specimen
  - Stoichiometric composition
  - Homogenized at 1000°C for 24 hours
- Pure nickel for γ
- Aluminum diffusion driving γ’ - γ transformation
- Polished, flat surfaces in dilatometer (10^{-6} torr)
- 1000°C for 4 hours





Diffusion Couple γ - γ'



In Progress Testing
Growth of Stable γ AGG Nuclei

- Specimens of Ni-200 (99.8% Ni), IN-625 deformed in tension
  - 5,10,15% deformation
  - Homogeneous deformation in gauge section
- Deformation to act as stored energy for grain growth driving force, but insufficient for recrystallization at diffusion couple temperatures
- Adding super-solvus heat treated RR1000 (stable γ grain size) to test matrix
  - Secondary γ' may act to delay traditional recrystallization until primary γ'
- Expect to observe extensive growth of new γ (formerly γ') to consume deformed γ

Future Work

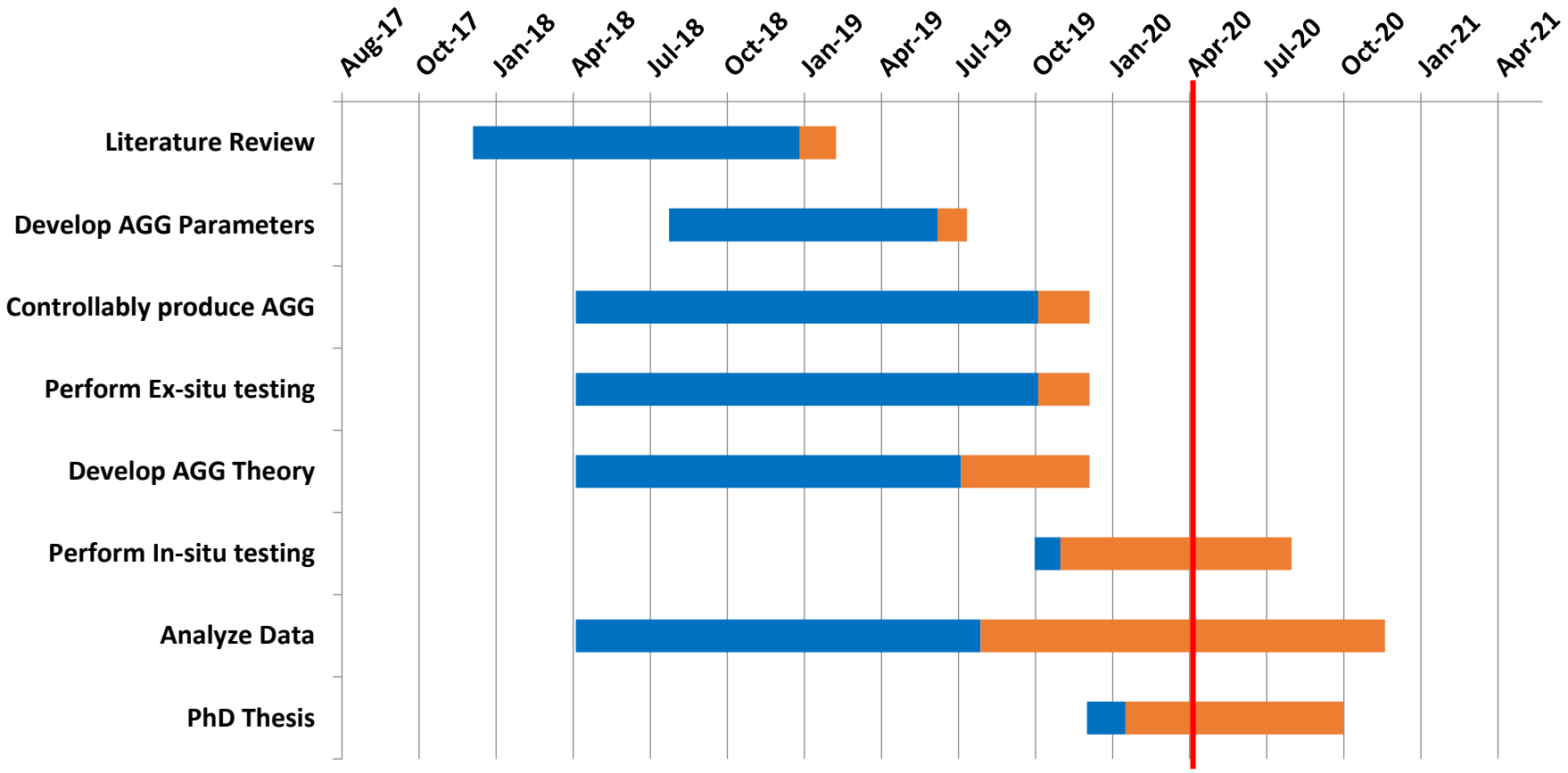
Diffusion Couple Experiments

- Establish critical strain just below recrystallization at diffusion couple time and temperature
- Demonstrate growth of strain free γ nuclei to consume deformed γ

Alleviating AGG Phenomena

- Provide possible processing routes that prevent or reduce AGG phenomena
  - Post-forging short SSHT
  - Lower temperature or high strain rate 'bump' after isothermal forging
- Supports mechanistic understanding of mechanism

Progress



Challenges & Opportunities

- Additional insight into isothermal forging of turbine discs may be helpful for providing processing suggestions to avoid AGG
- Performing flat grinding & polishing for diffusion couple surfaces is difficult
  - Sub-micron fluctuations limit bonded surfaces
  - Currently hand grinding & polishing
  - 4mm diameter surface
- Information on recrystallization of low deformation nickel and IN-625 would be helpful

Thank you!

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