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

Fall Meeting

October 13th – 15th 2020

Student: Byron McArthur (Mines)

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

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

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 March. 2021

- 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

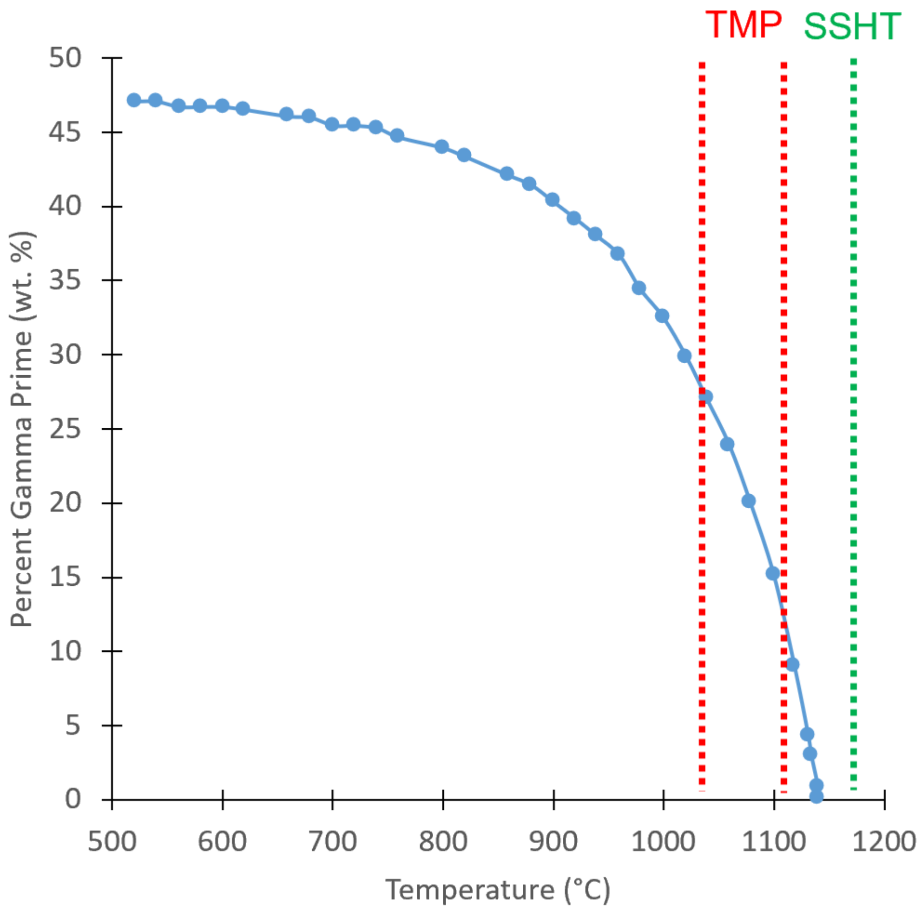
- Developing mechanistic theory for AGG
- Performed interrupted heat treatment processing to determine mechanisms for abnormal grain growth

Metrics

Description	% Complete	Status
1. Literature review	95%	●
2. Explore abnormal grain growth forging parameters for RR1000	95%	●
3. Ex-situ and interrupted material testing and characterization	90%	●
4. Develop and test theory to explain abnormal grain growth phenomena	90%	●
5. Propose and test methods for alleviating abnormal grain growth in industrial processing	75%	●

Material: RR1000, γ-γ'

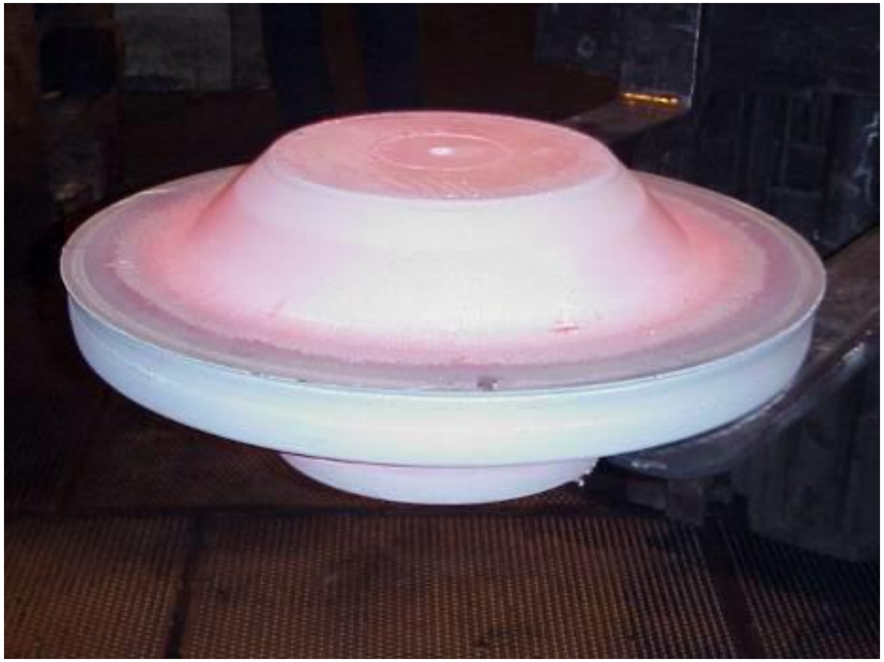
- Processing:
  - Powder metallurgy
  - Hot isostatic pressure compaction
  - Extruded at 5:1 ratio
  - Isothermal forging: 1035-1110°C
    - Performed in Gleeble®
  - Super solvus heat treatment (SSHT)
    - 1150-1170°C
    - Performed in dilatometer
- Critical AGG parameters:
  - Strain
  - Strain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



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

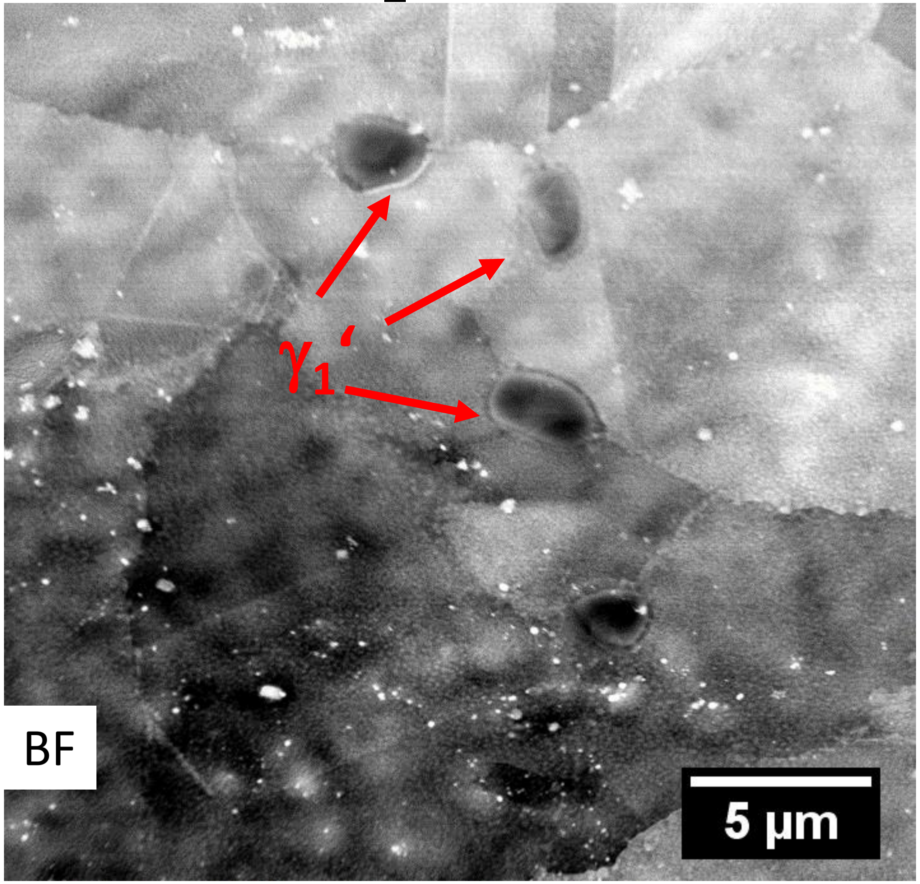


Forging ~1 Meter Diameter

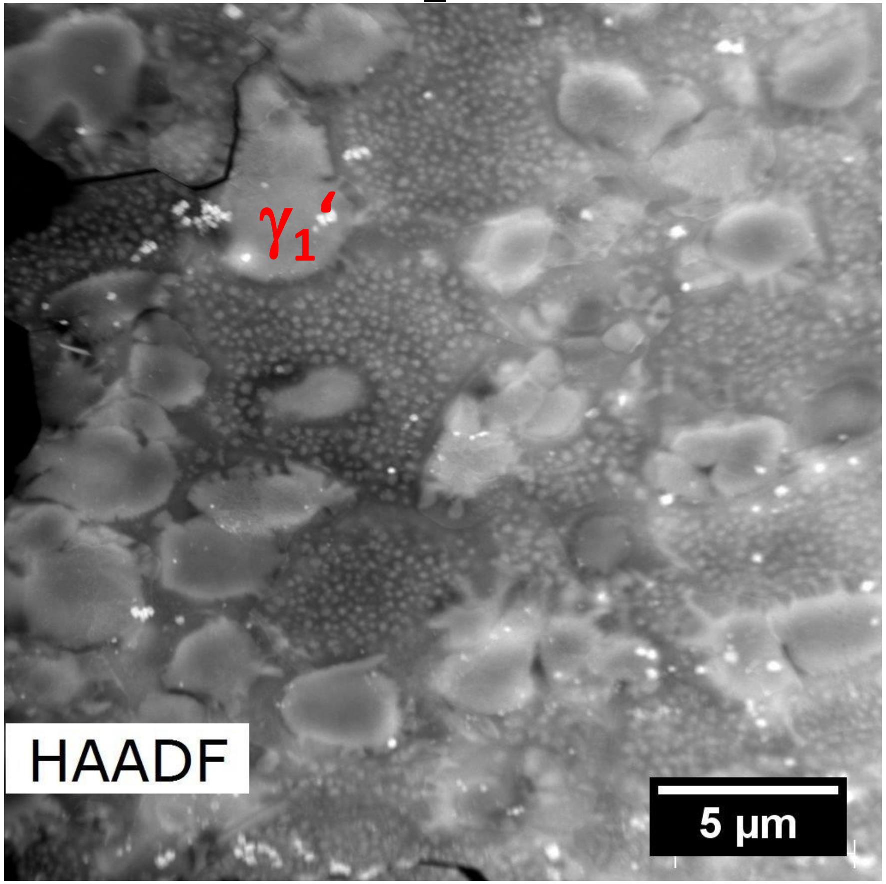
Mitchell, R. J., Lemsky, J. A., Ramanathan, R., Li, H. Y., Perkins, K. M., & Connor, L. D. *Superalloys 2008, pp.* 347–356.

As-received Material

Low γ_1' Fraction



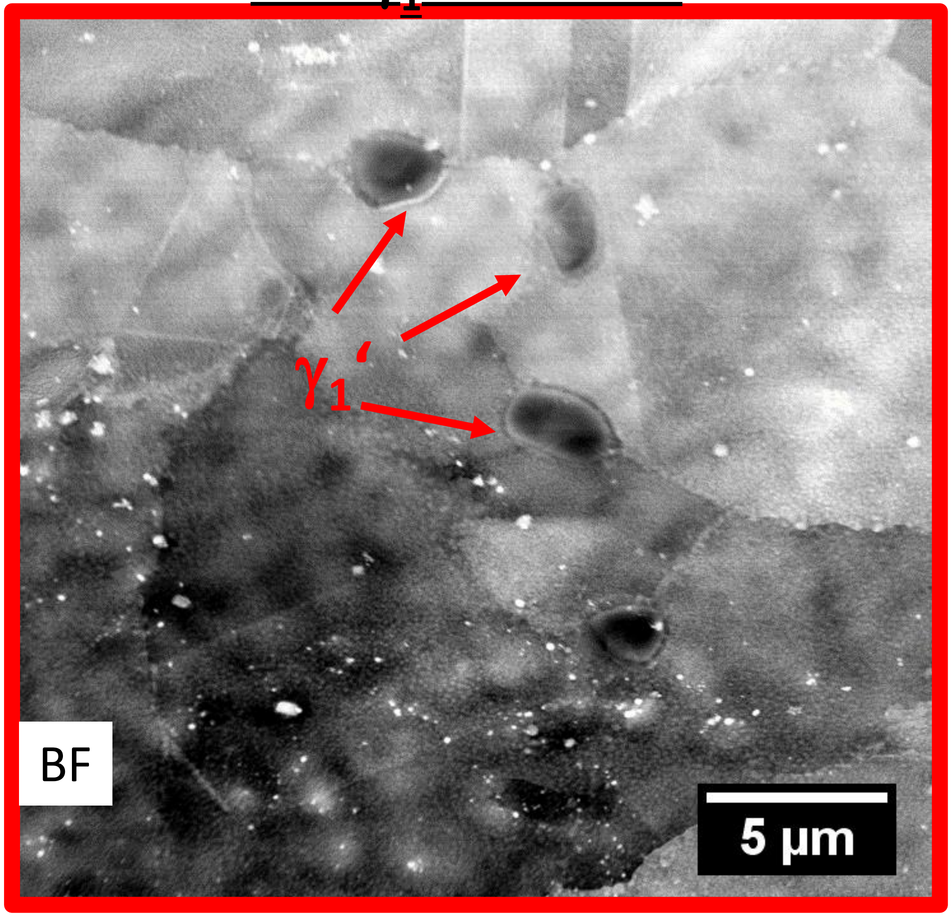
High γ_1' Fraction



Thanks to Yaofeng Guo for TEM imaging

As-received Material

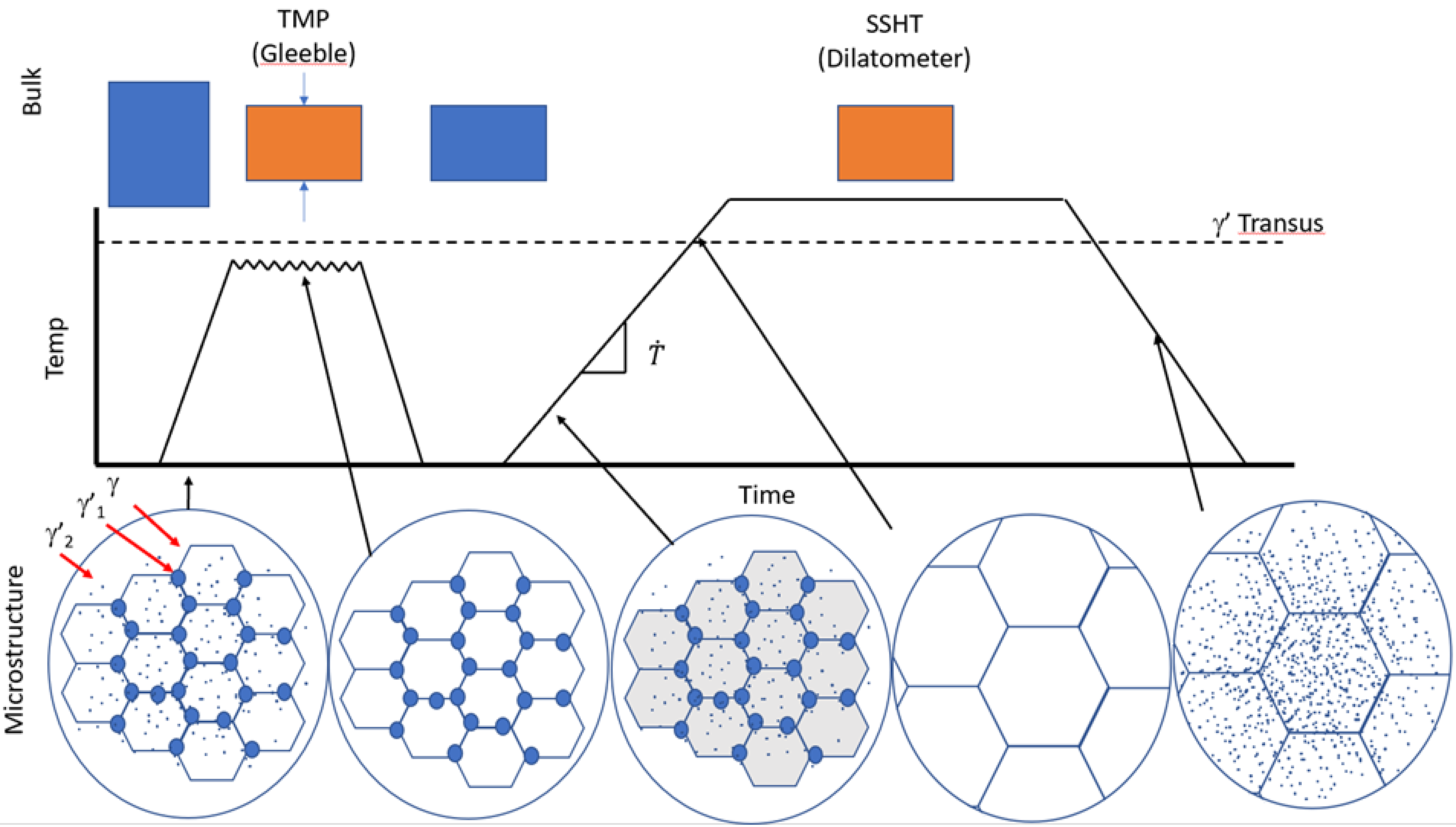
Low γ_1' Fraction



- 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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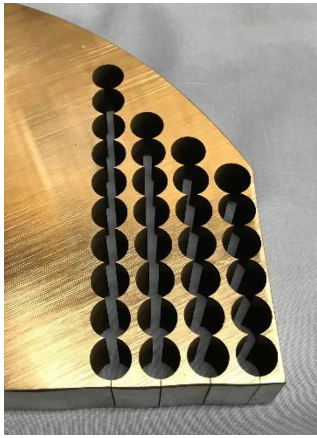
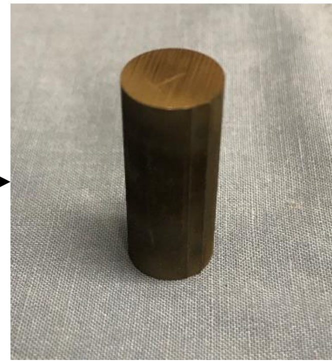
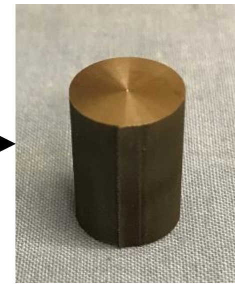
Wire-EDM

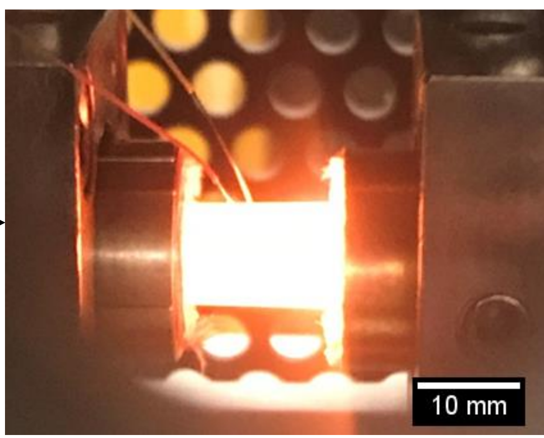




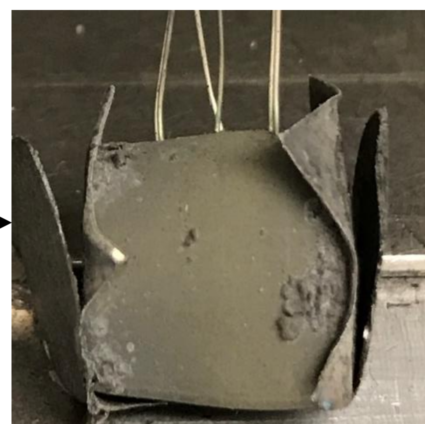
Machine to length with parallel faces



Isothermal forging in Gleeble®

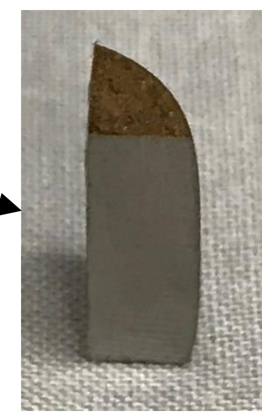


Thermocouple locations



As-deformed Specimen

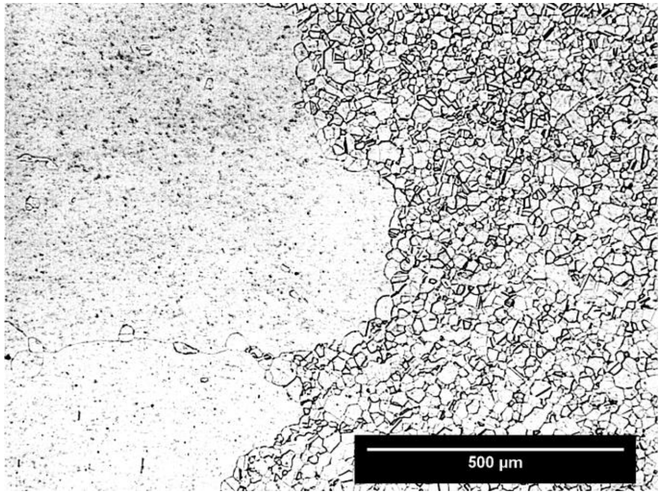
Dilatometer Specimen for SSHT

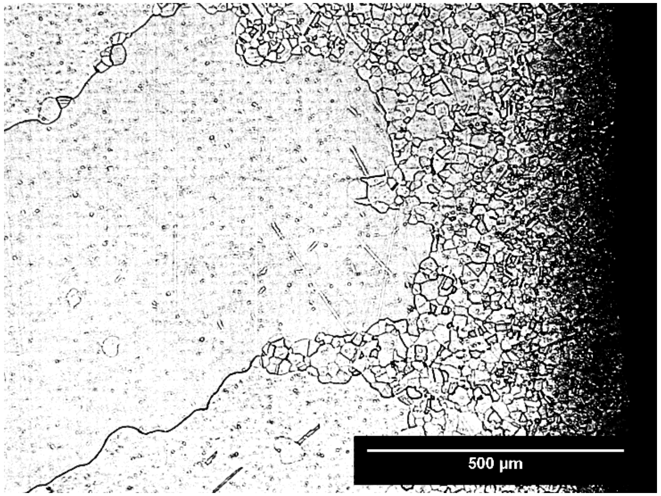


SSHT Specimen

Summary of Prior Work & Results

- Replicate industrial processing that leads to AGG in lab-scale testing
  - High temperature, low strain & strain rate, low SSHT heating rate
  - Forging: 1110-1120°C, 10^{-3} s^{-1}, 0.15 ε
  - Heating Rate: 8°C/min
- Determine influential processing parameters and their roles
  - Varying parameters shift AGG based upon stored energy
- Consistently produce AGG via Gleeble TMP and heat treating
- Exploring experimental techniques to detail phenomena





Theory of Abnormal Grain Growth

Coherent γ Recrystallization

Hetero-epitaxial Recrystallization (HERX) (Charpagne)

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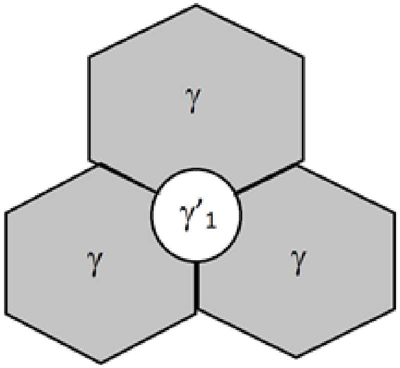
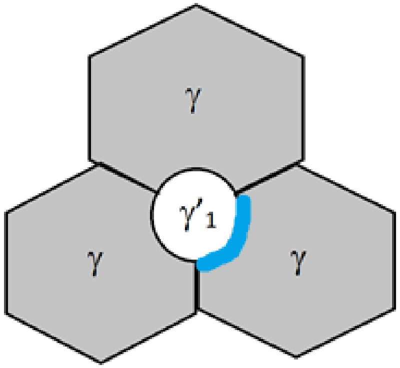
1

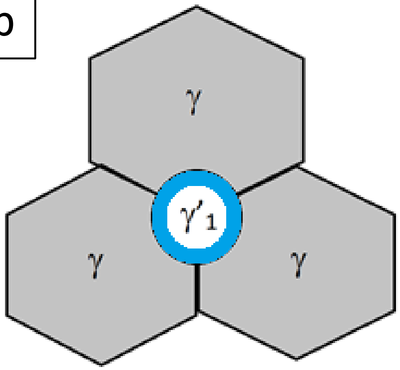


γ'_1 is incoherent with neighboring γ

2a

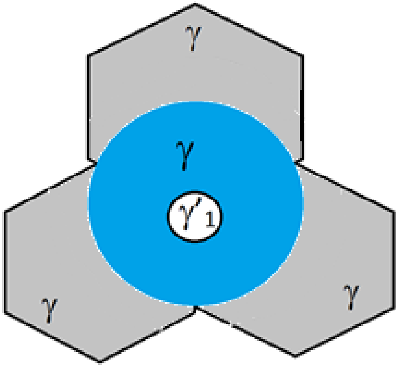


2b



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

3



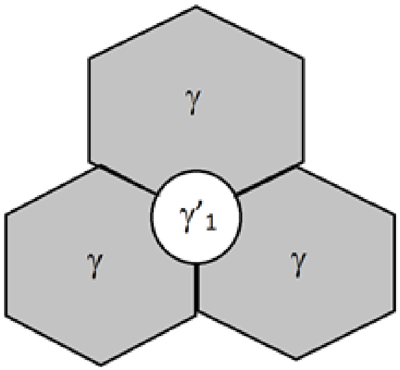
Growth to consume stored energy in γ

- 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

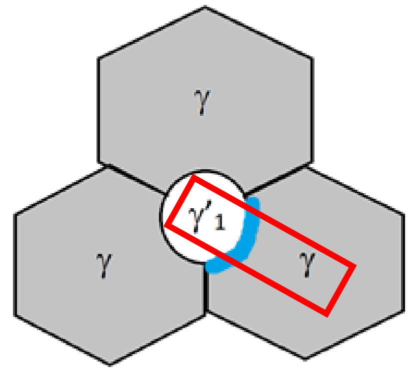
Hetero-epitaxial Recrystallization (HERX) (Charpagne)

1



γ'_1 is incoherent with neighboring γ

2a



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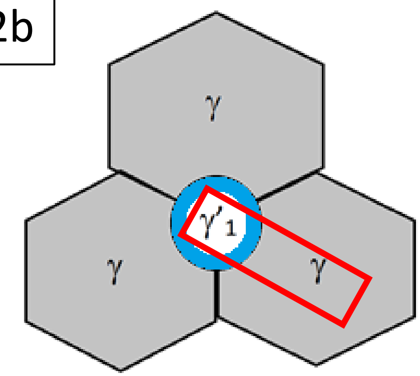
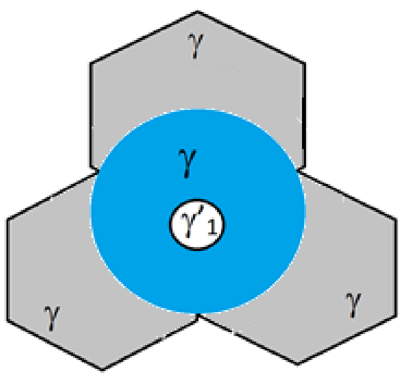


γ nucleates coherently off γ'_1 or during order-disorder transformation

3



Growth to consume stored energy in γ

- Want to observe microstructural development at interface
- Occurs at ~1080-1110°C
- Rapidly in limited instances
- Statistically difficult with 2-D sectioning

Mesoscopic γ-γ' Interface Study

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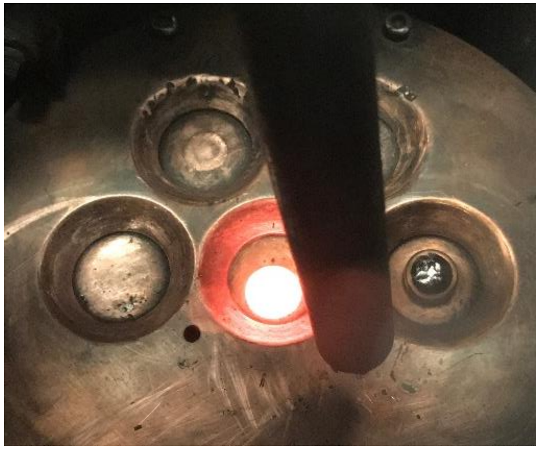
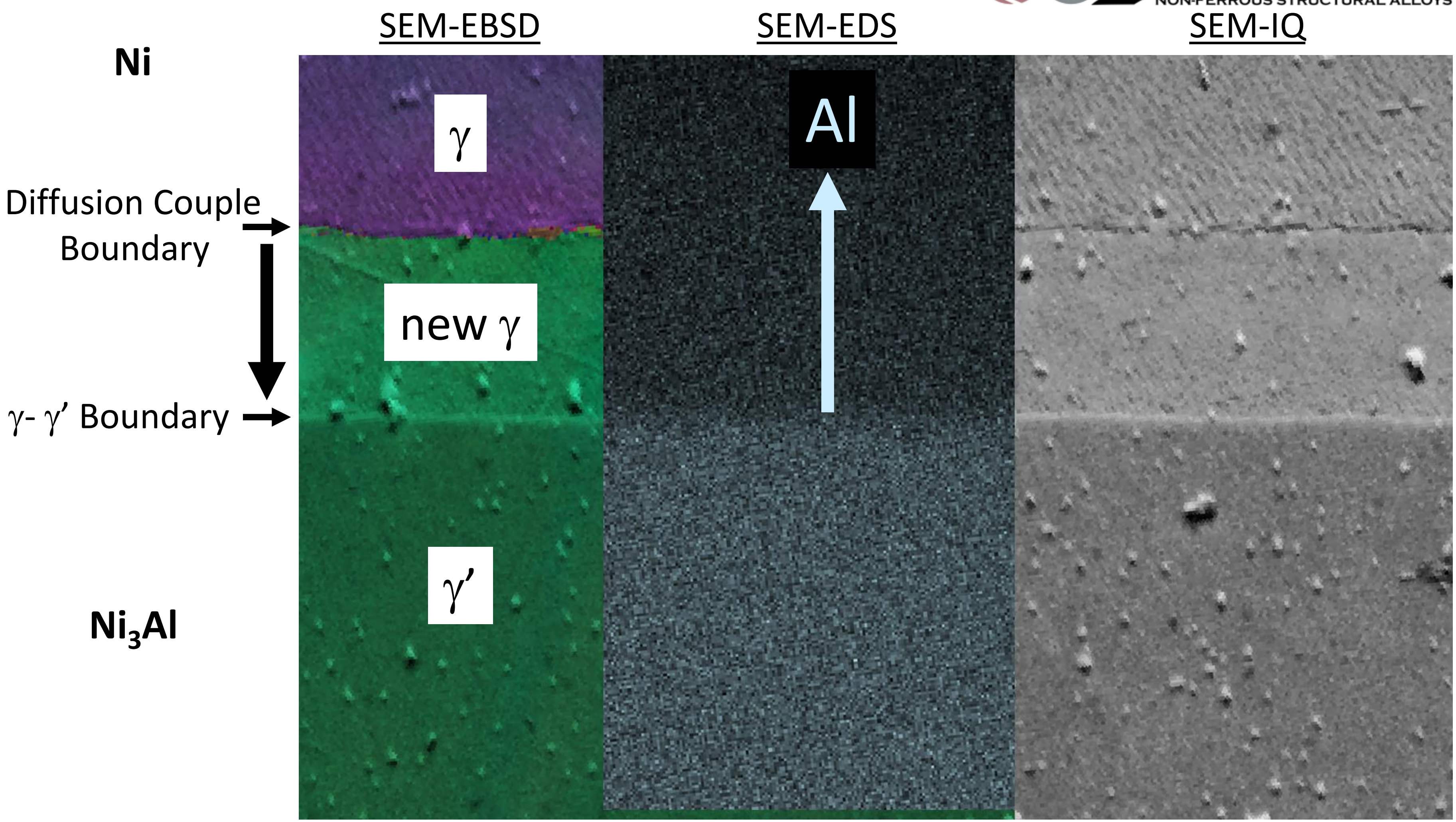


- Diffusion couple to create γ-γ' interface
- γ' created through Ni_3Al button melted specimen
  - Stoichiometric composition
  - Homogenized at 1000°C for 24 hours
- Pure nickel for γ
- Aluminum diffusion driving γ' - γ transformation
- Polished, flat surfaces in dilatometer (10^{-6} torr)
- 1000°C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

Diffusion Couple γ - γ'



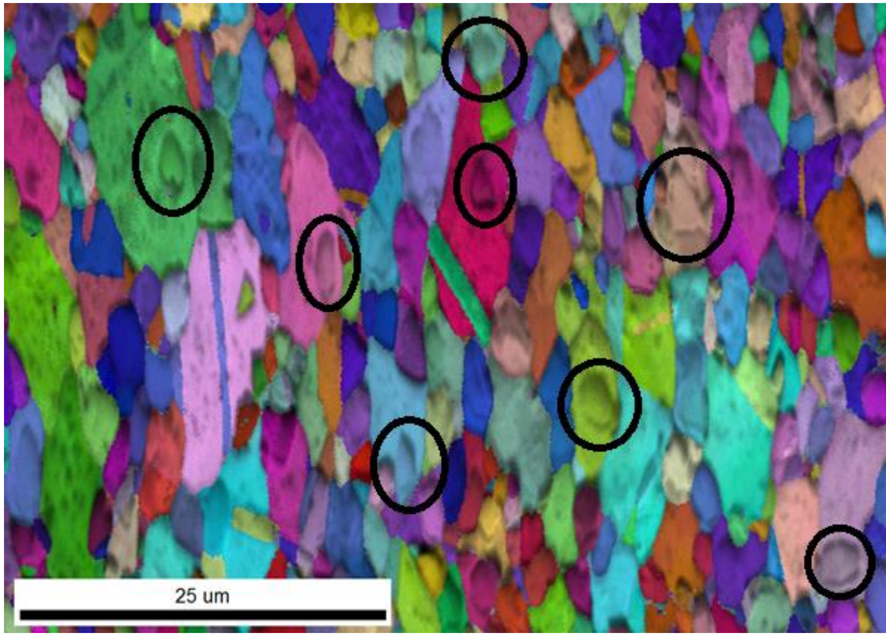
Experiment demonstrates formation of new γ and γ- γ' boundary is energetically favored over movement of existing γ- γ' boundary.

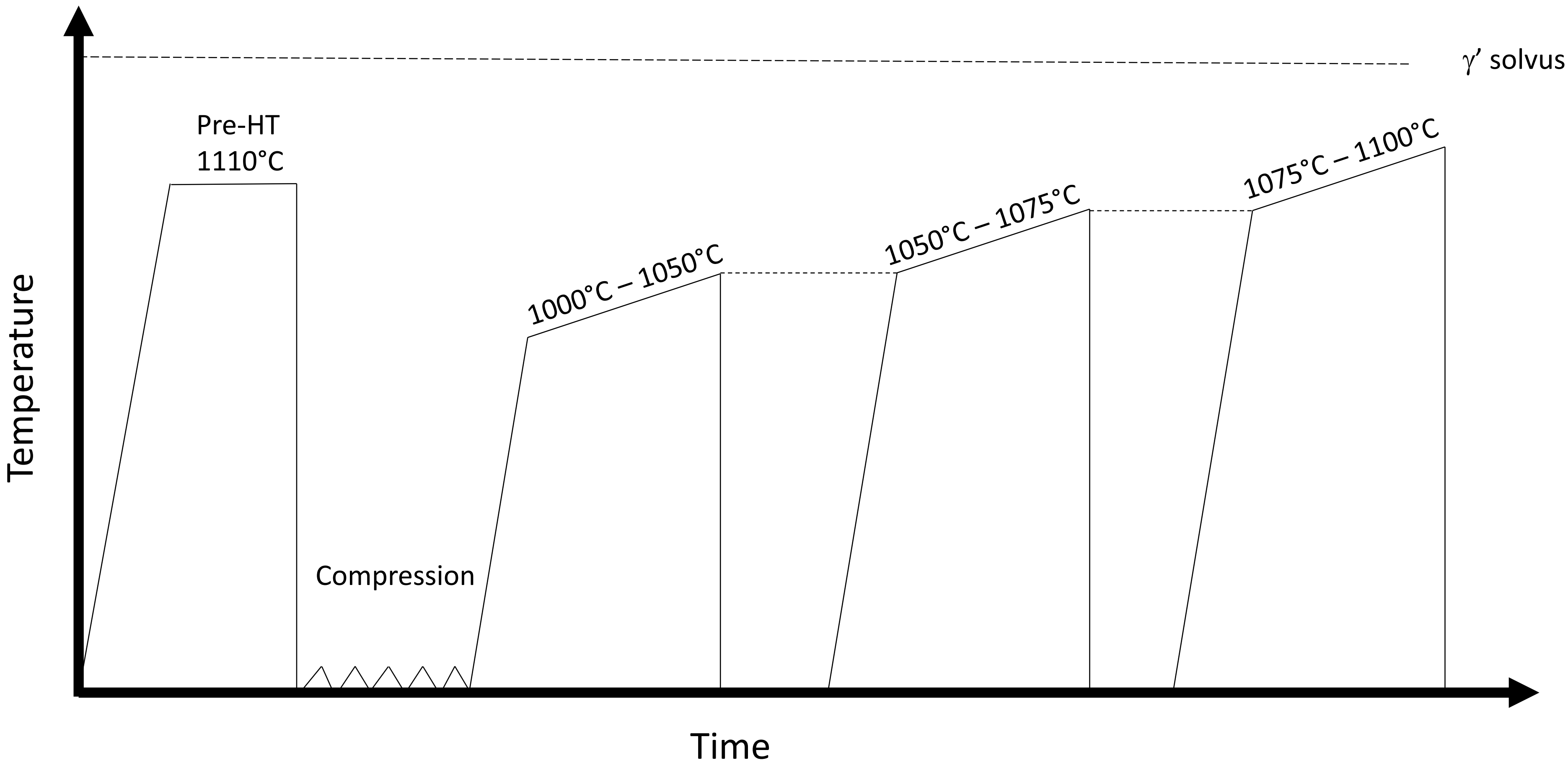
Interrupted Heat Treatments

Methods:

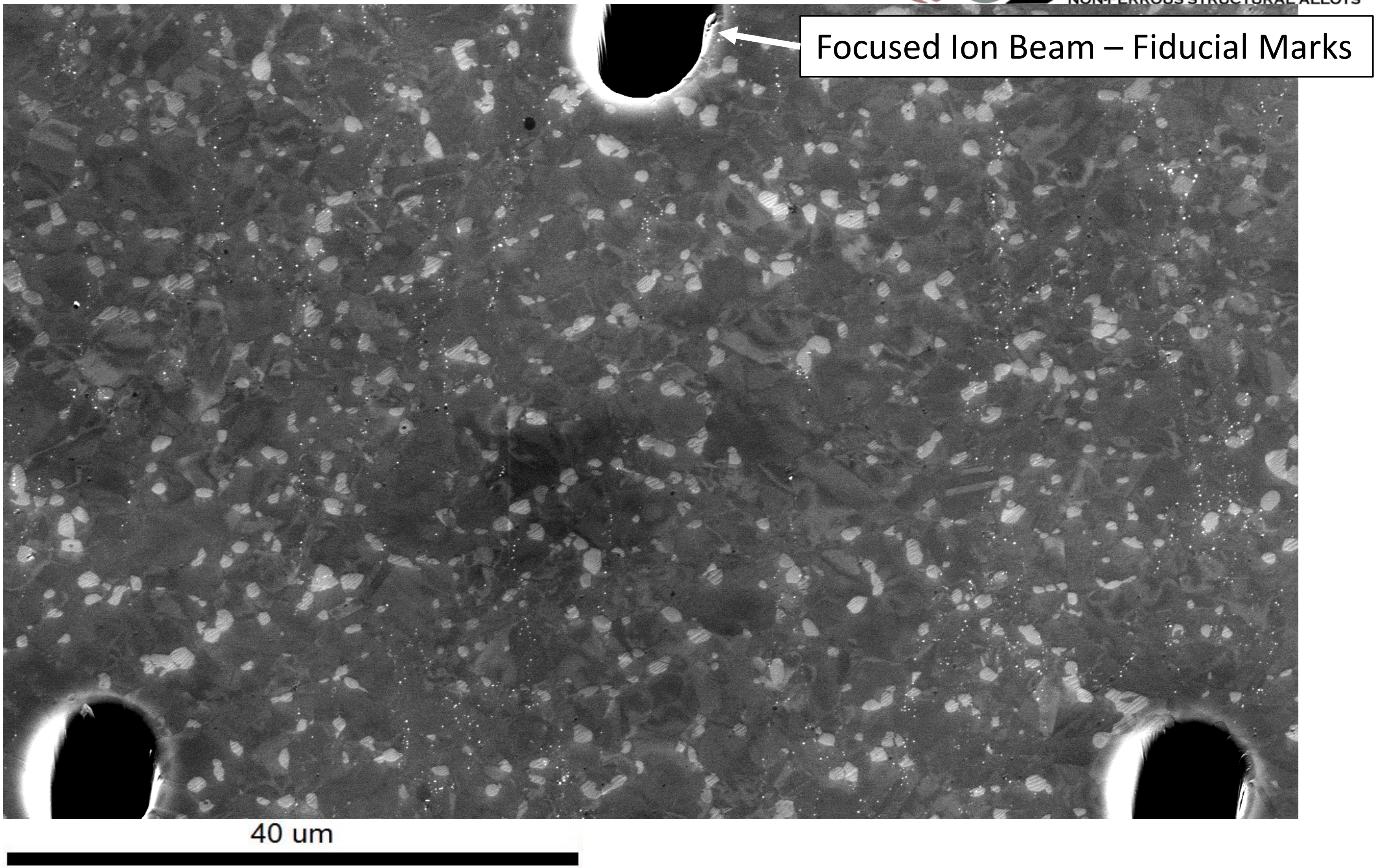
- Utilize room temperature deformation for homogeneous, controlled distribution of stored energy
- Pre-heat treatment to remove existing γ'_1 coherent with matrix γ
  - Sub-solvus heat treatment (1110°C)
  - DRX or MDRX sourced
- Interrupted heating intervals at controlled heating rate
  - **Viewing same region**

As-Received Microstructure

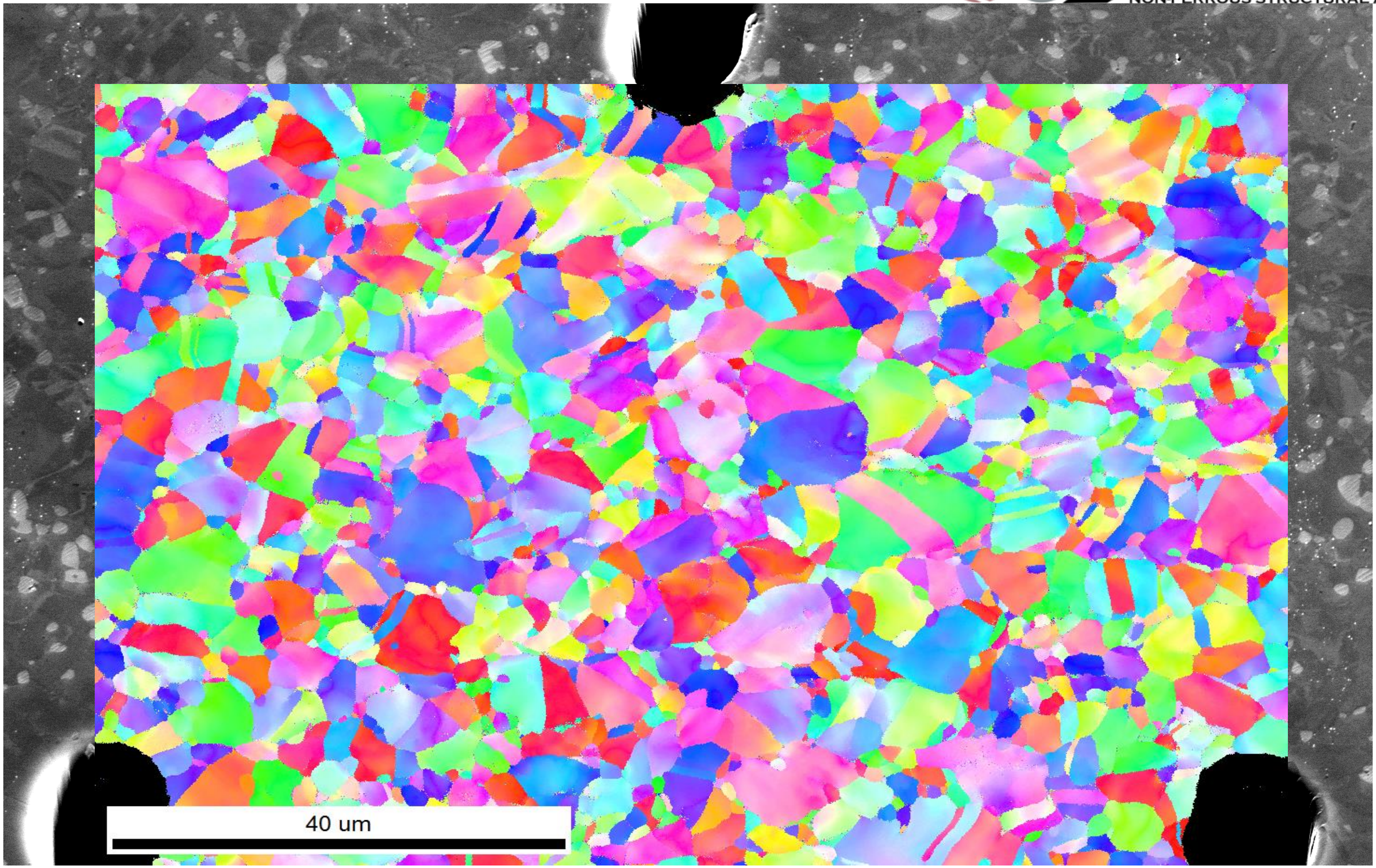



γ' solvus
Pre-HT
1110°C
Compression
1000°C – 1050°C
1050°C – 1075°C
1075°C – 1100°C
Temperature
Time

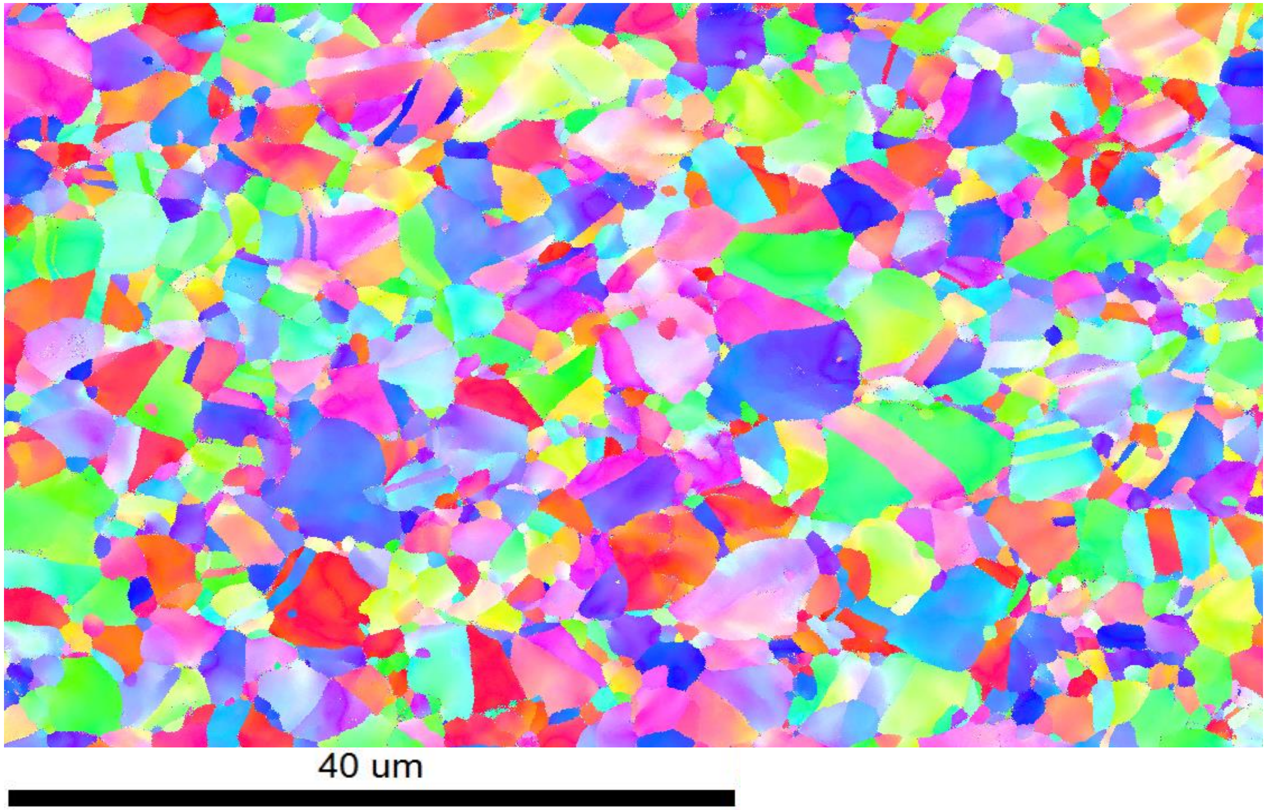
Interrupted Heat Treatments
SEM/SEI – As Deformed



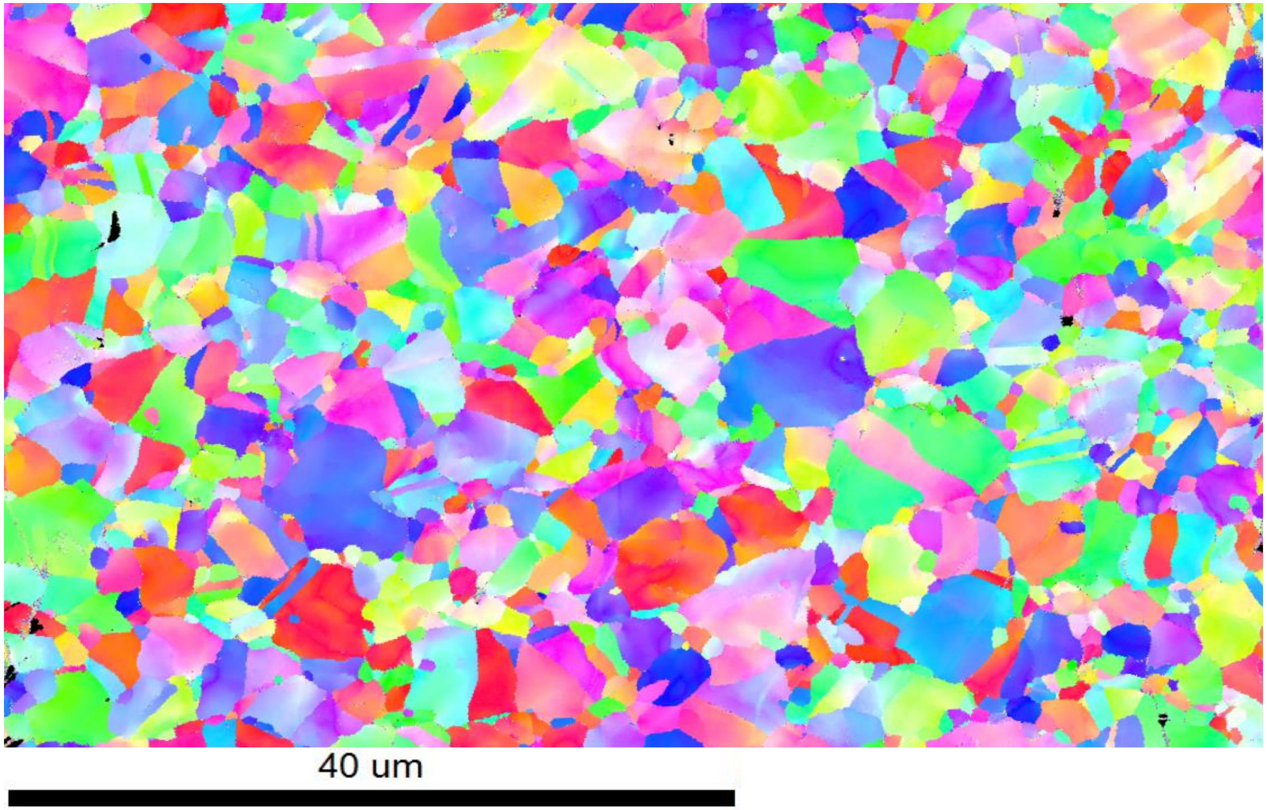
Interrupted Heat Treatments
EBSD – As Deformed



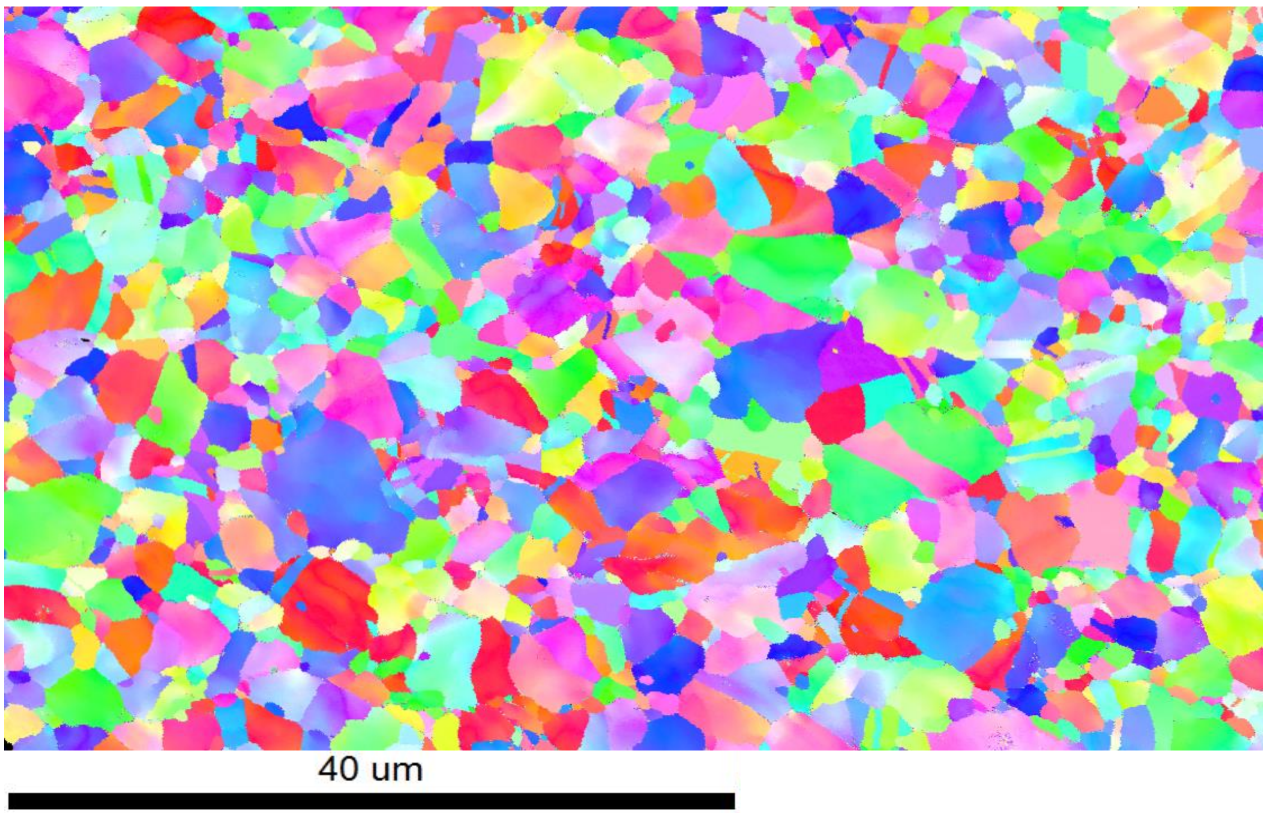
Interrupted Heat Treatments
EBSD – As Deformed



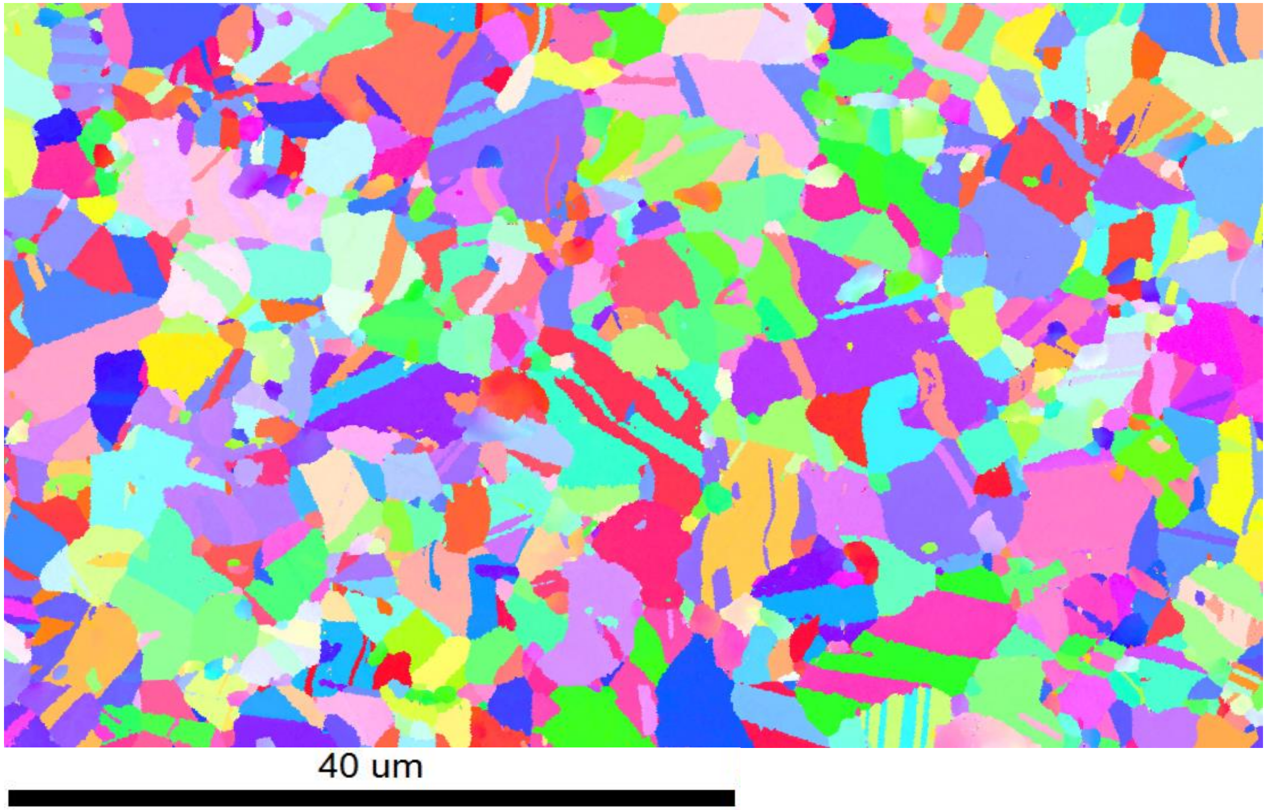
Interrupted Heat Treatments
EBSD – 1050°C



Interrupted Heat Treatments
EBSD – 1075°C



Interrupted Heat Treatments
EBSD – 1100°C



Interrupted Heat Treatment Results

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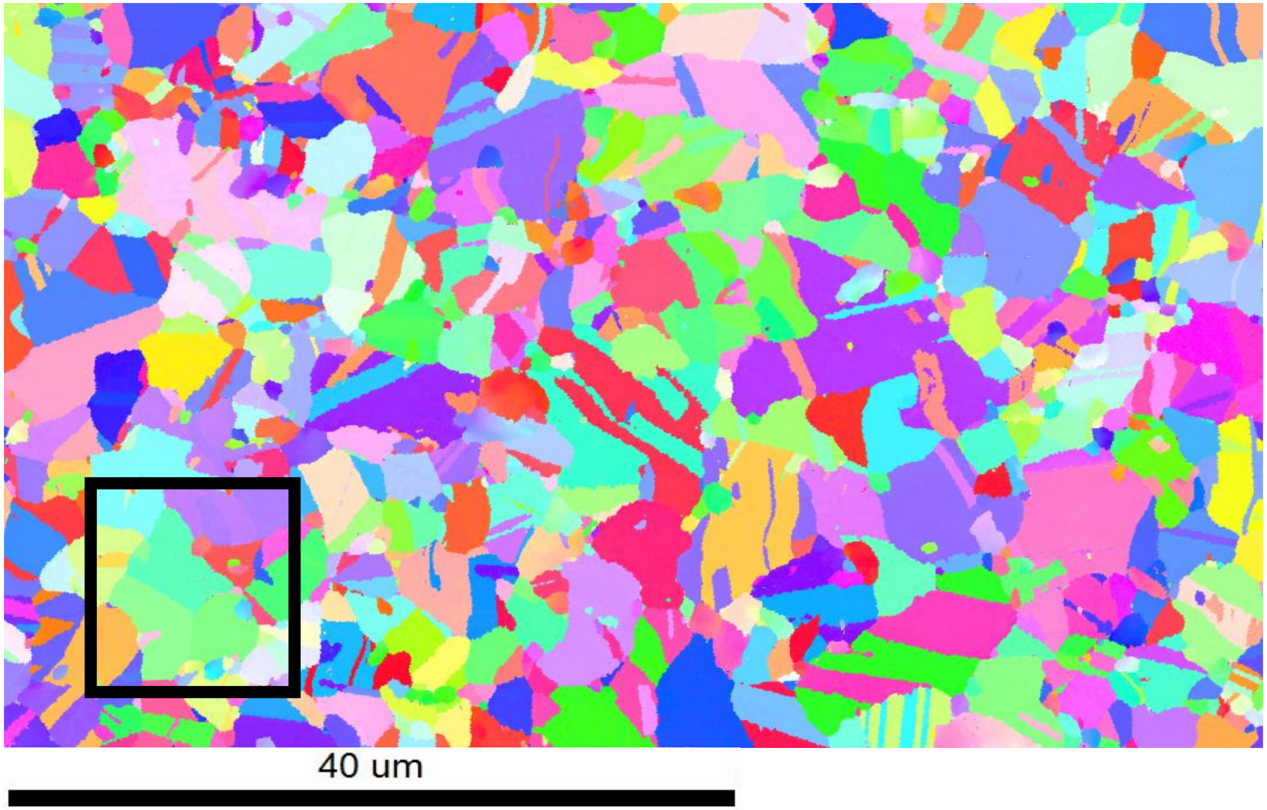
- Temperatures below 1050°C show no major microstructural changes
- Some recrystallization nucleation sites appear in plane, while most are out of plane
- Full recrystallization (determined by GROD) by 1100°C for 1110°C preheat treatment temperature
  - Preliminary 1130°C preheat treatment specimen shows delayed RX growth
- γ boundaries able to pass by γ_1' to create intragranular, incoherent γ_1'
- Lower RX temperature than preheat treatment may suggest γ templating method rather than γ'_1 dissolution (?)

Interrupted Heat Treatments
EBSD – 1100°C



Progression of γ Growth From γ'_1

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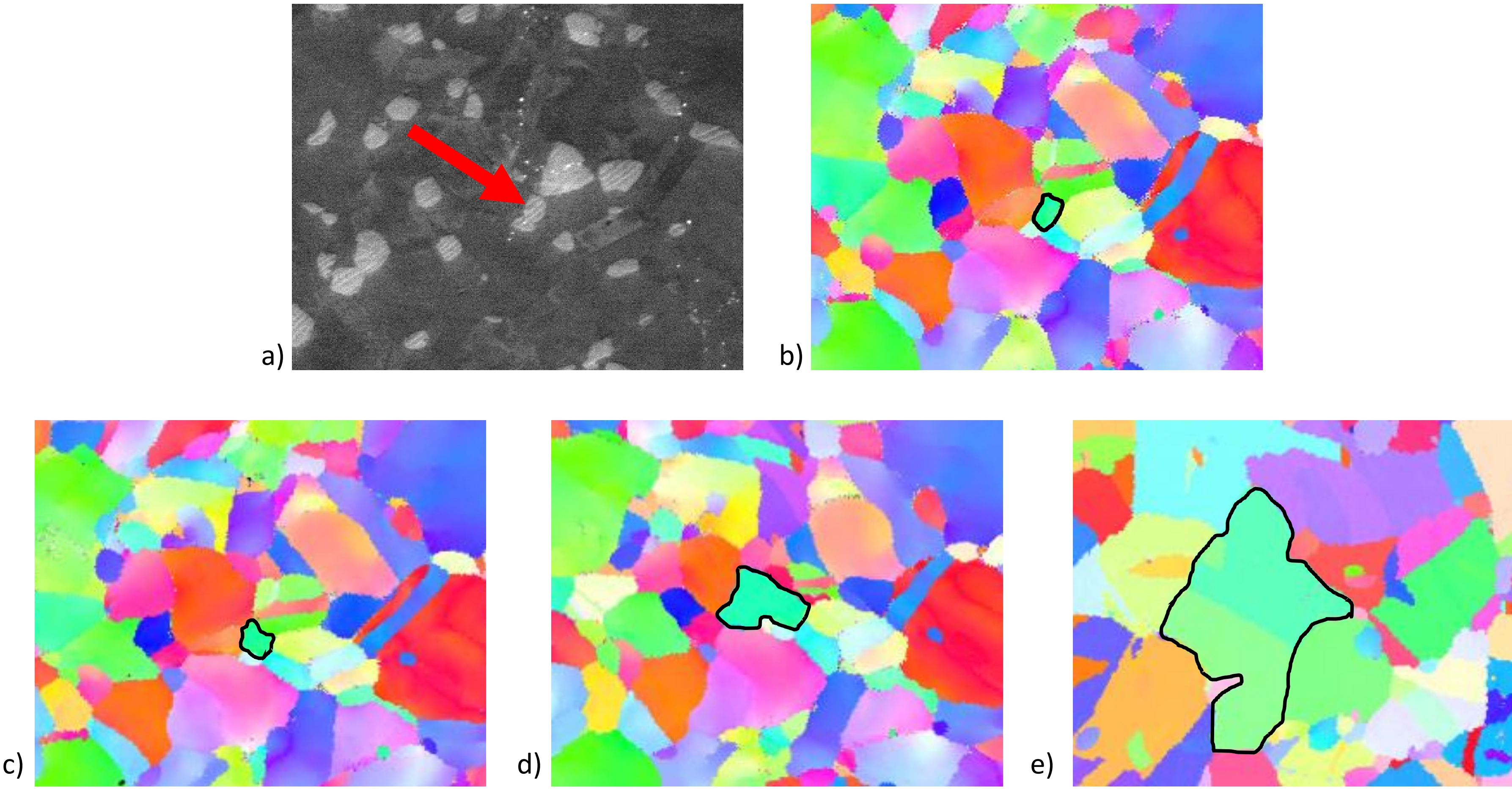
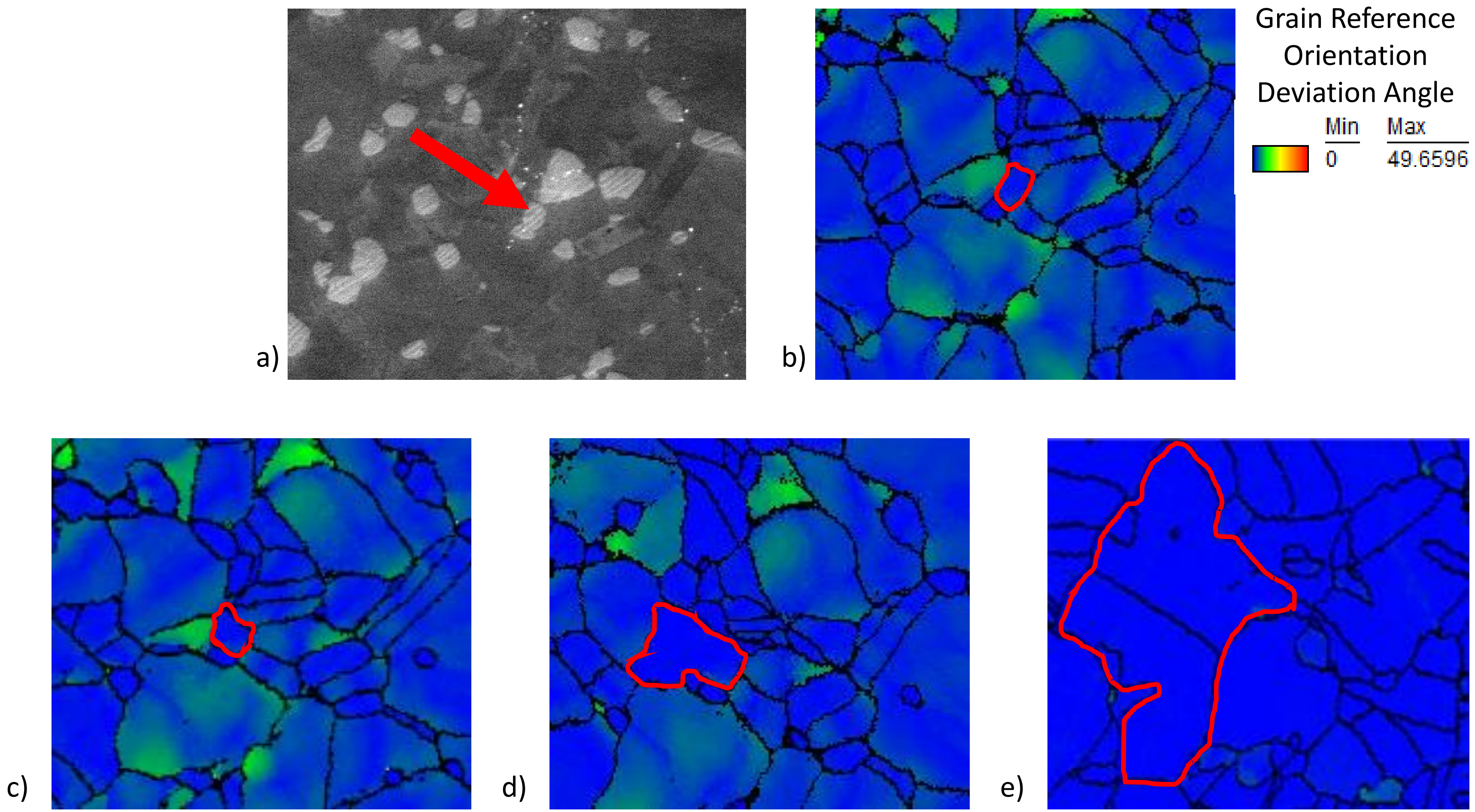
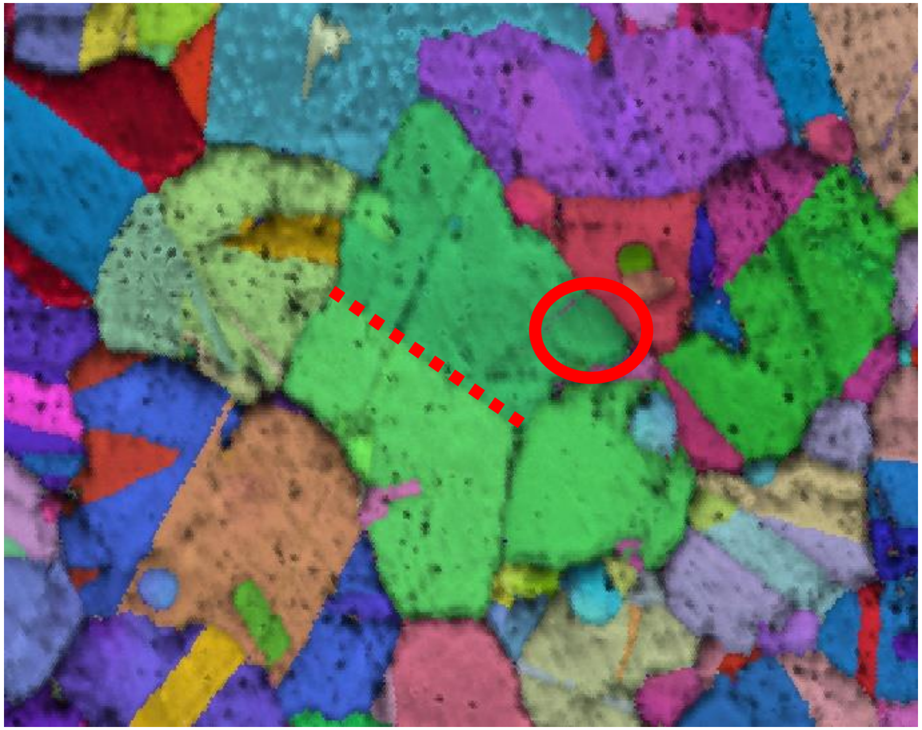


Consumption of Stored Energy



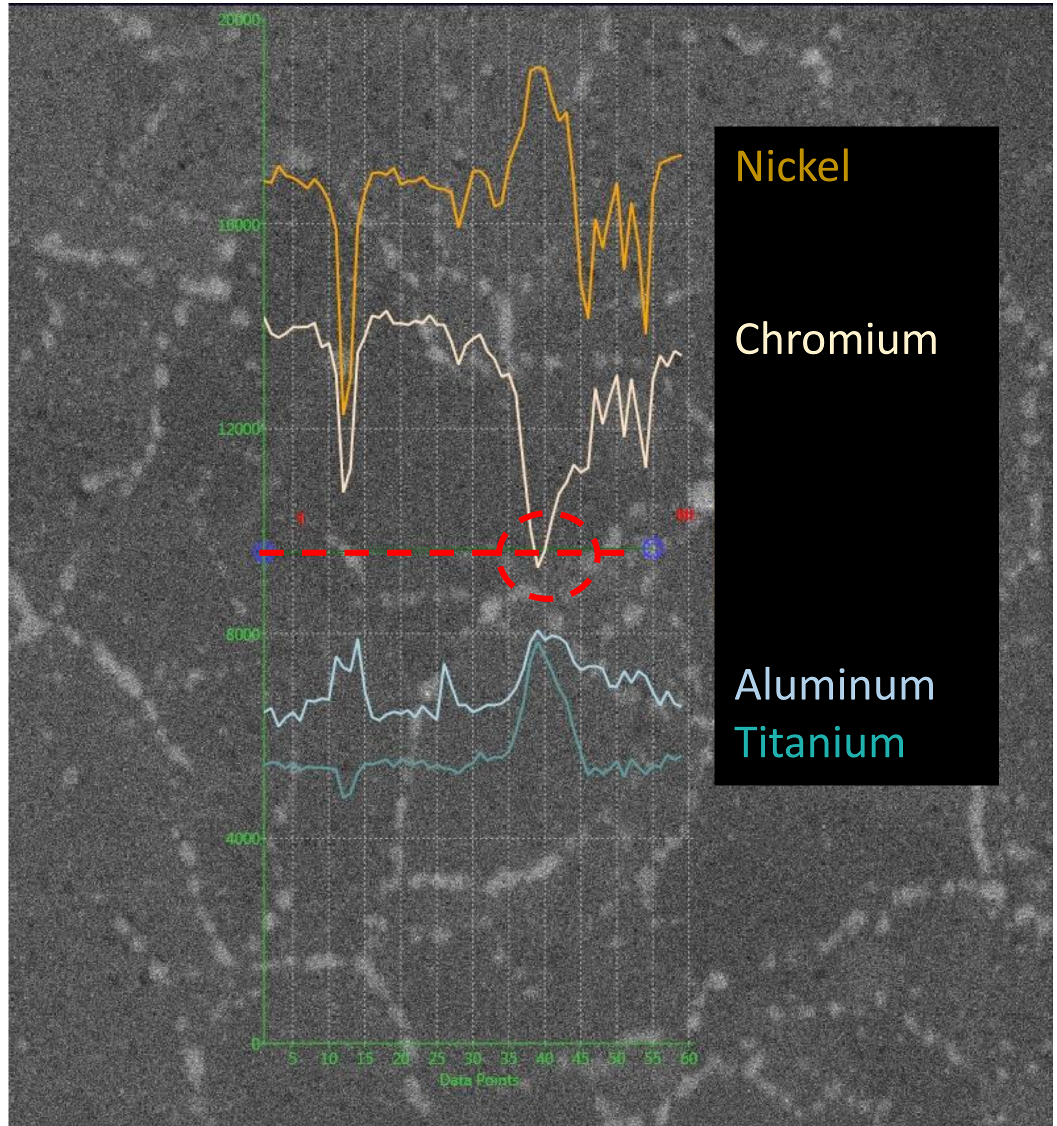
γ'_1- γ Orientation

- SEM-EBSD analysis shows same orientation for γ'_1 precipitate as recrystallized & strain free γ that grew to consume neighboring γ containing stored energy
- γ'_1 precipitate shown in red circle
- γ twin boundary shown by dashed red line



Elemental Analysis (EDS)

- SEM-EDS line scan analysis demonstrates γ' composition for particle
- Neighboring surface oxide present as well
  - Superfluous data



Future Work

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Interrupted Heat Treatments

- Repeat experiment for higher pre-heat treatment temperature
  - Decreased

Coherency Analysis

- Appears that γ_{RX} should be coherent with parent γ'_1
- Orientation alone is insufficient to classify as coherent
- Planning TEM analysis of FIB liftout traversing γ-γ'_1 boundary
  - Misfit dislocations, coherency strain, lattice continuity

Industrial Processing Remedies

- Increased strain rate during late processing possible to impart more strain in critical regions
  - Preliminary results show some promise

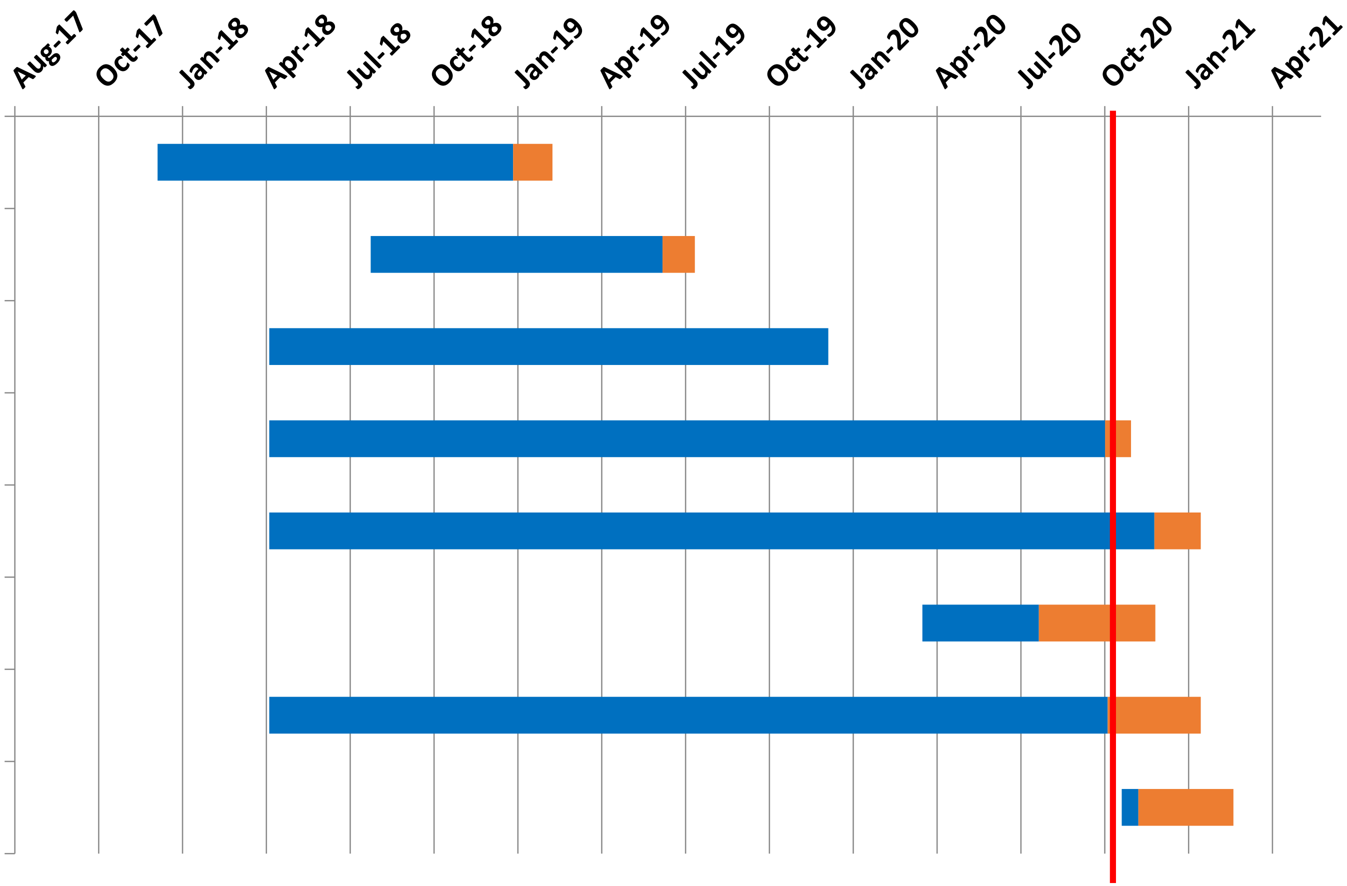
Challenges & Opportunities

- Determining coherency at γ-γ'_1 interface
  - Loss of coherency as γ_{RX} grows doesn't necessarily change hypothesis
- Calculate theoretical size for coherency loss
  - Low lattice parameter misfit ~ 0.05%

Possible future work:

- Interesting cyclic γ'_2 coarsening/splitting behavior observed
  - Previous literature, proposed homogenization process
- Slow cooling appears to promote γ-γ'_1 shelling
  - Locally supersaturated in γ forming elements?
  - Inflection point in γ-γ'_1 solvus line?

Progress



Aug-17
Oct-17
Jan-18
Apr-18
Jul-18
Oct-18
Jan-19
Apr-19
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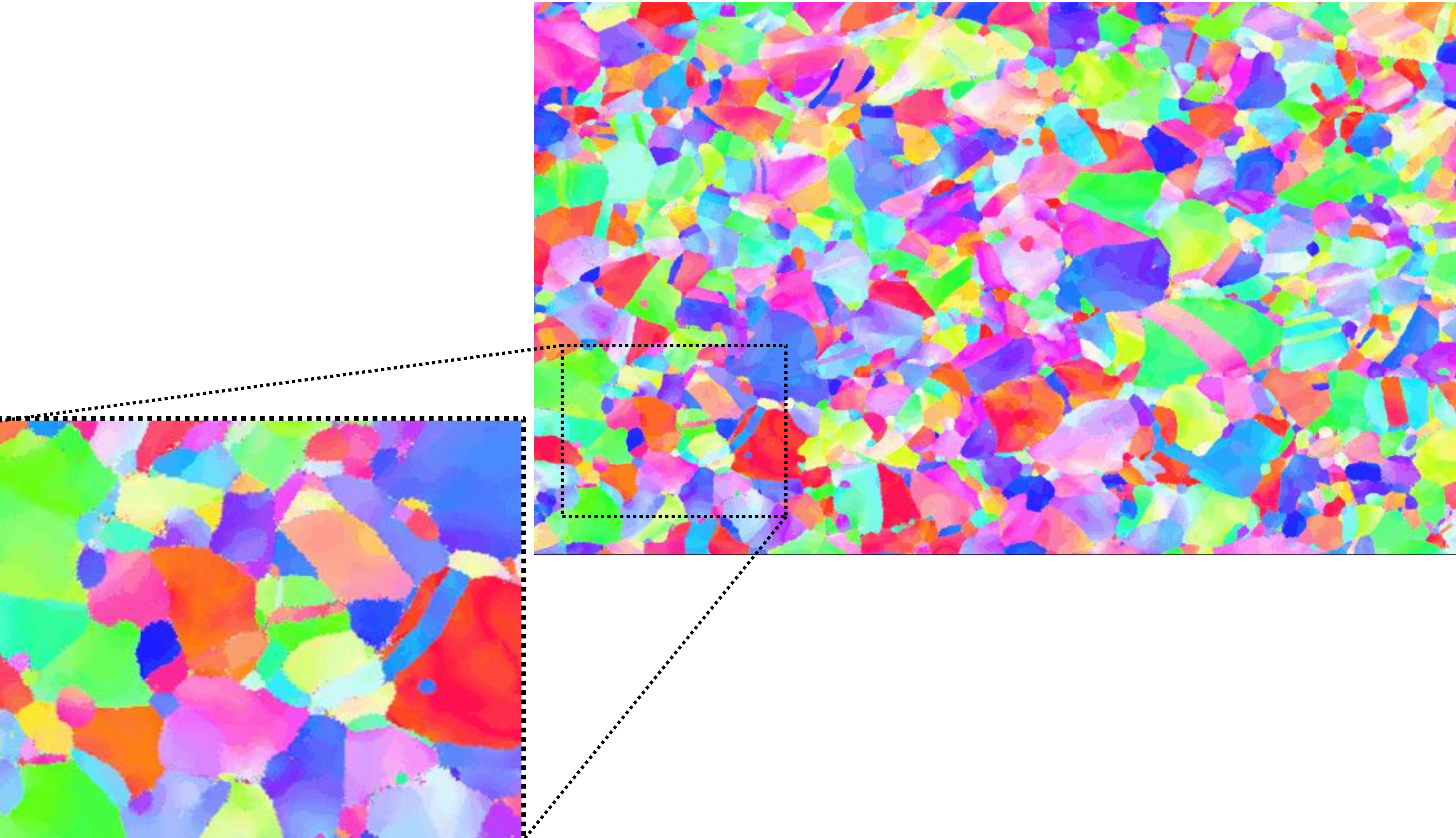
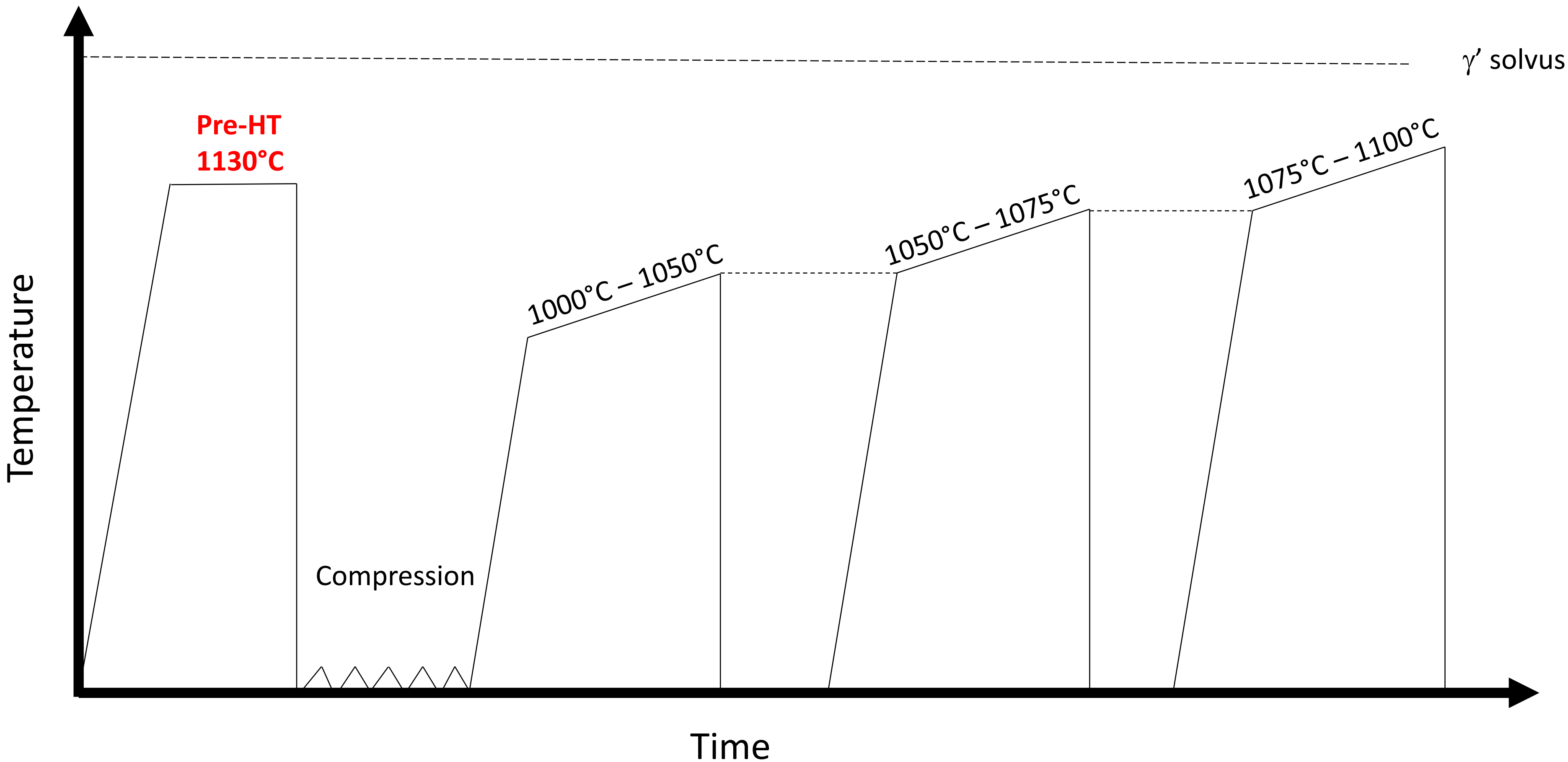
Apr-20
Jul-20
Oct-20
Jan-21
Apr-21
Literature Review
Develop AGG Parameters
Controllably produce AGG
Perform Ex-situ testing
Develop AGG Theory
AGG Prevention Experiments
Analyze Data
Writing Dissertation

Thank you!

Byron McArthur
bmcarthu@mines.edu

1110°C Pre-HT, 15% Compression
Animated




γ' solvus
Pre-HT
1130°C
Compression
1000°C – 1050°C
1050°C – 1075°C
1075°C – 1100°C
Temperature
Time