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

Summer 2020 Videoconference

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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, 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.

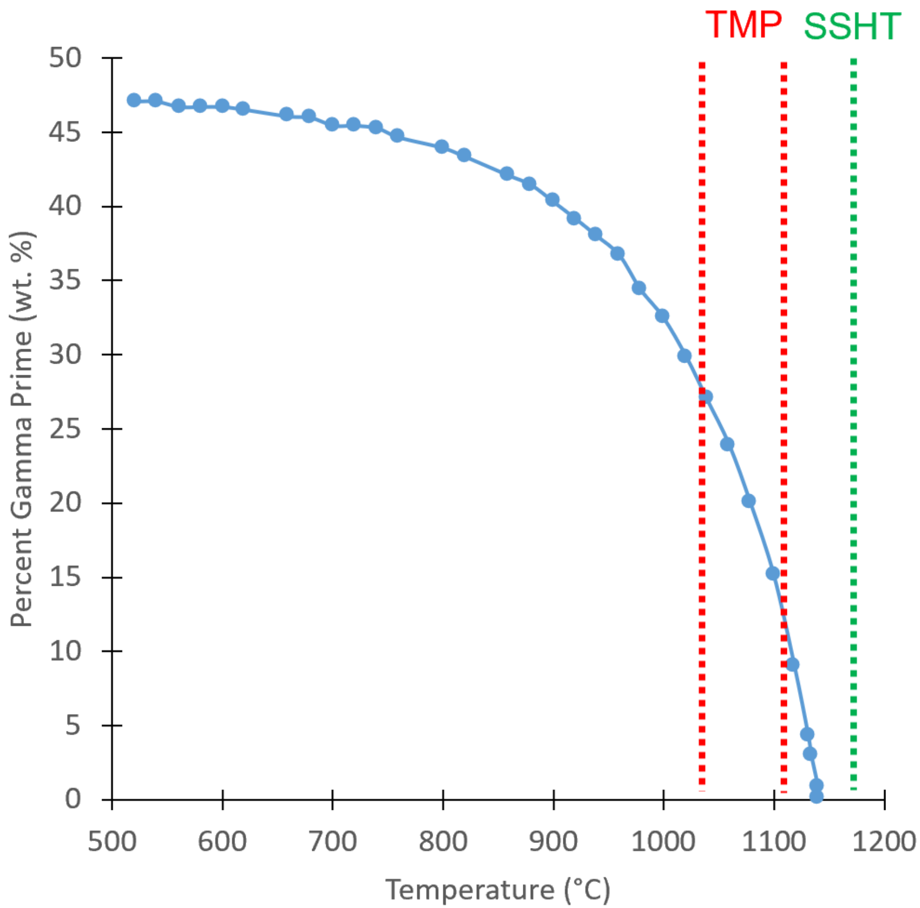
Recent Progress

- Developing mechanistic theory for AGG
- 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	65%	●
4. Develop and test theory to explain abnormal grain growth phenomena	75%	●
5. Perform model testing to observe mechanisms	50%	●

Material: RR1000, γ-γ’

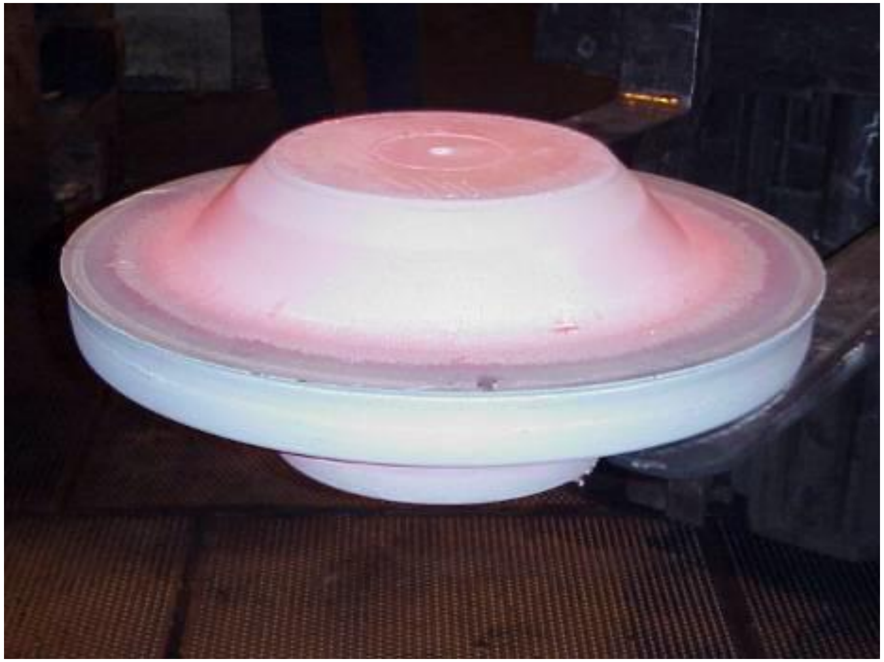
- Processing:
  - Powder metallurgy
  - Hot isostatic pressure compaction
  - Extruded at 5:1 ratio
  - Isothermal forging: 1035-1110°C
    - Performed in Gleeble®
  - Super solvus heat treatment (SSHT)
    - 1150-1170°C
    - Performed in dilatometer
- Critical AGG parameters:
  - Strain
  - Strain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

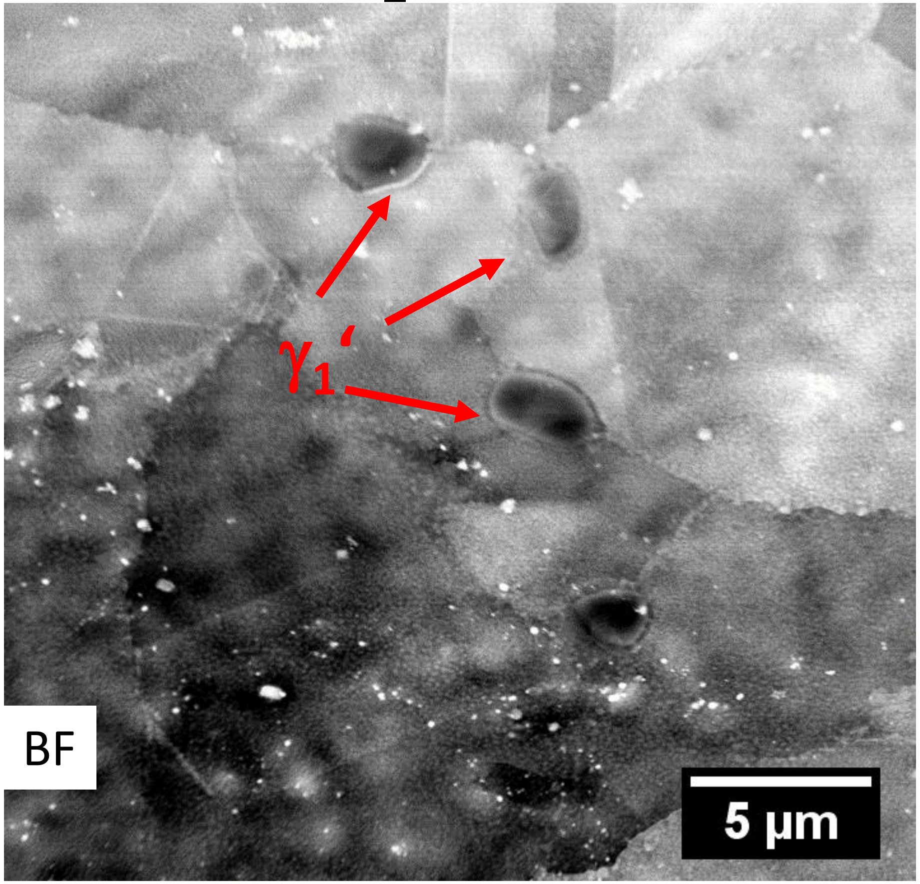


Forging ~1 Meter Diameter

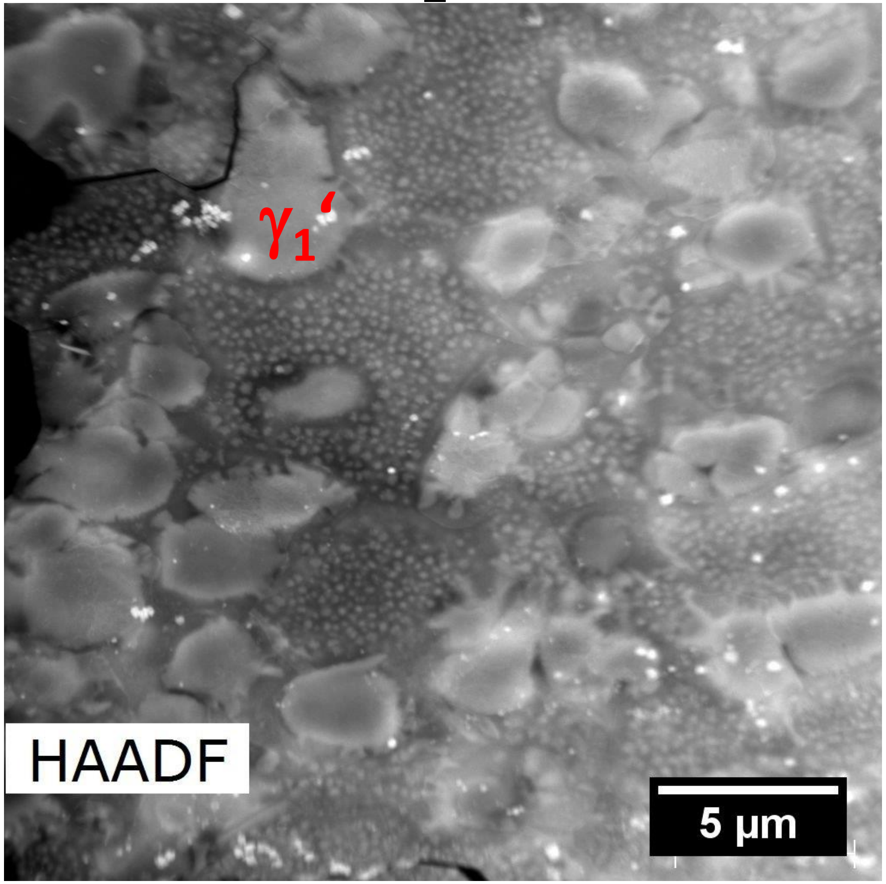
Mitchell, R. J., Lemsky, J. A., Ramanathan, R., Li, H. Y., Perkins, K. M., & Connor, L. D. *Superalloys 2008, pp.* 347–356.

As-received Material

Low γ_1' Fraction



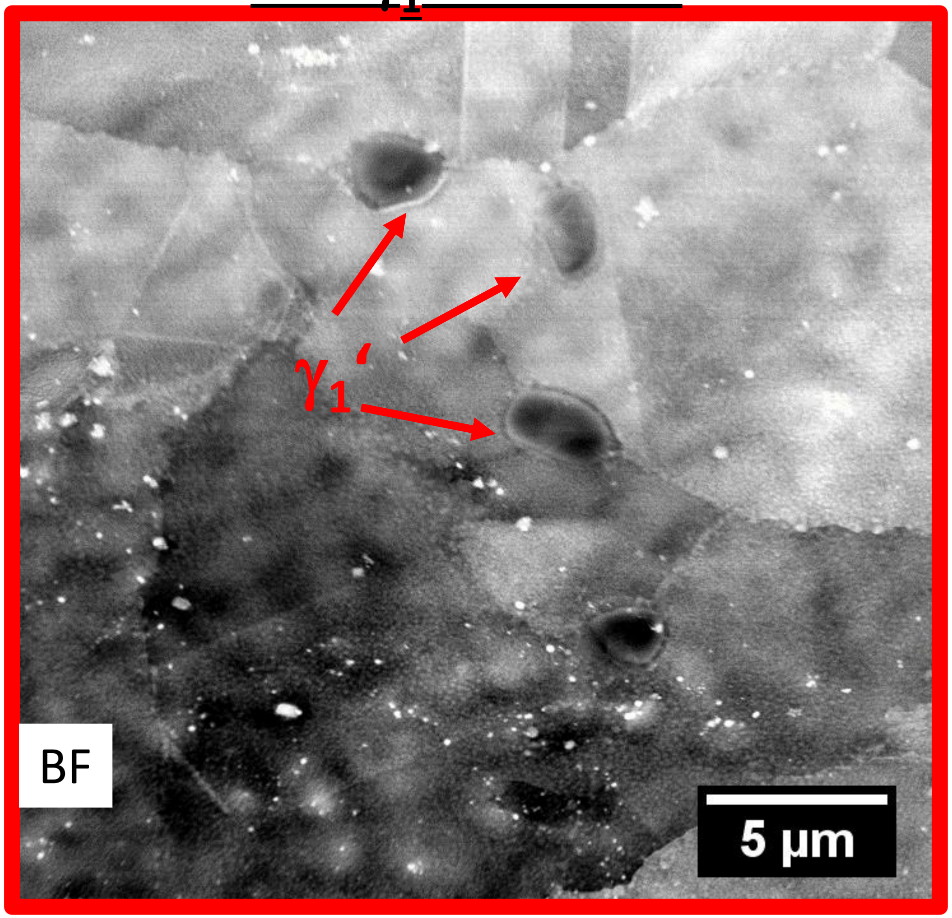
High γ_1' Fraction



Thanks to Yaofeng Guo for TEM imaging

As-received Material

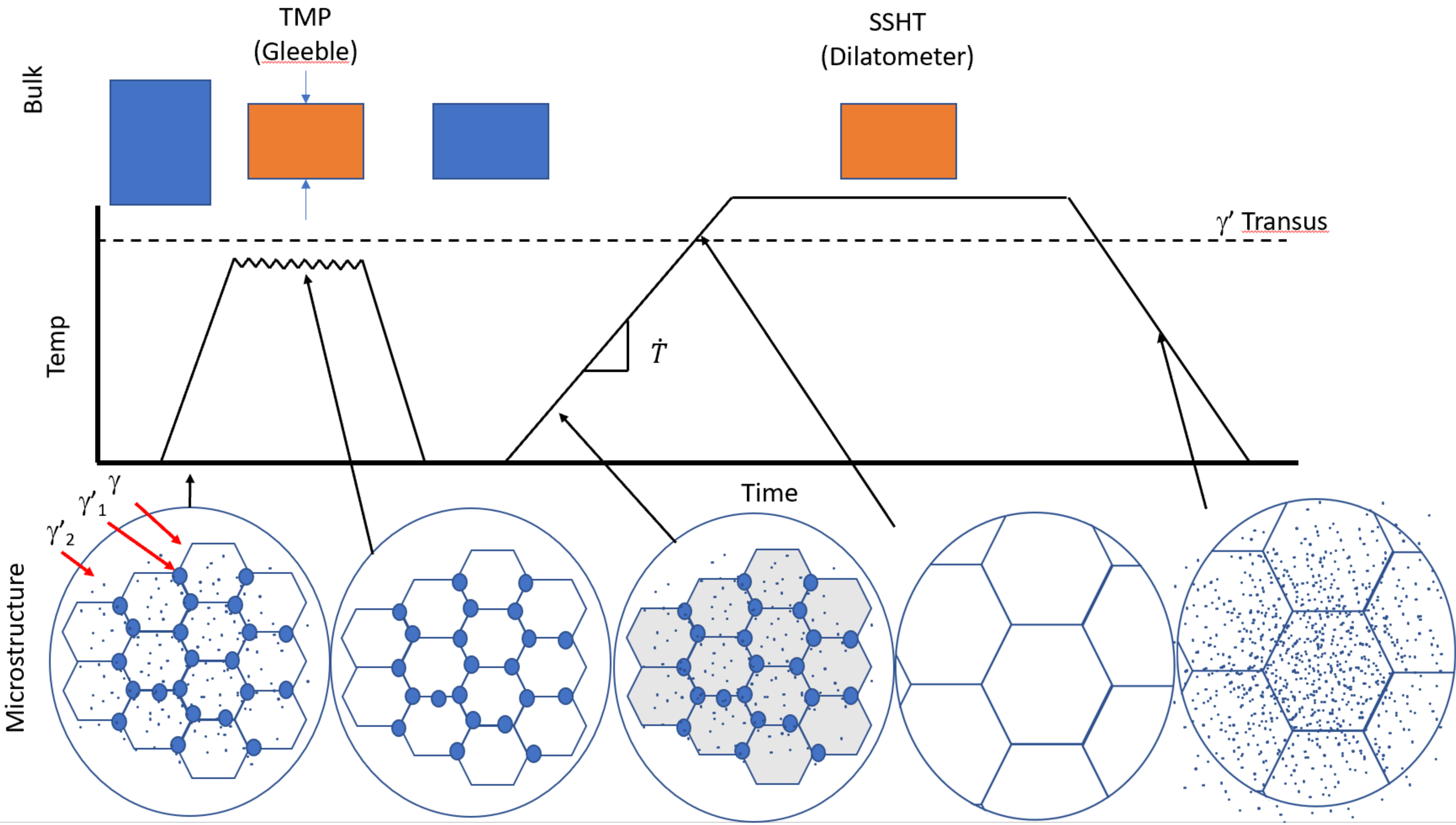
Low γ_1' Fraction



- 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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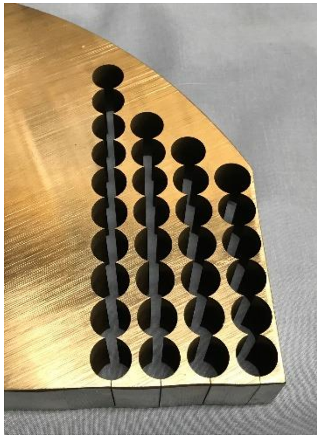
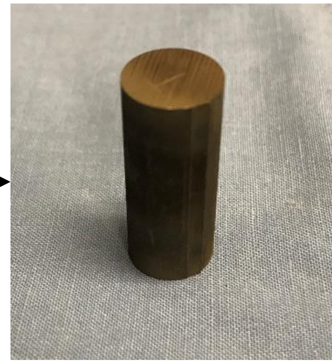
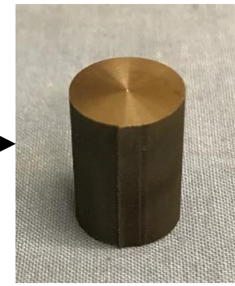
Wire-EDM

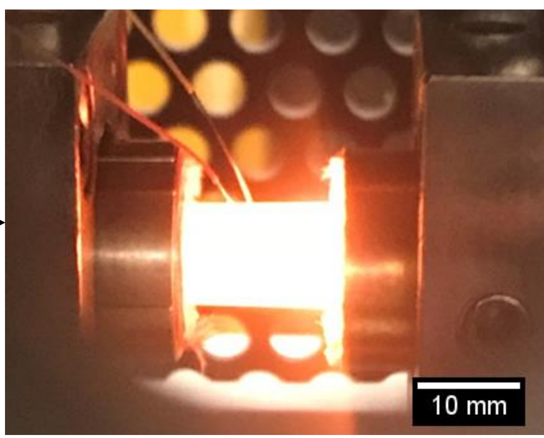




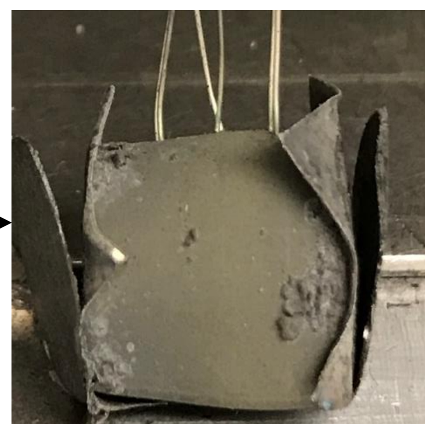
Machine to length with parallel faces



Isothermal forging in Gleeble®

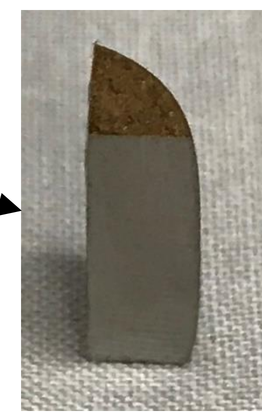


Thermocouple locations



As-deformed Specimen

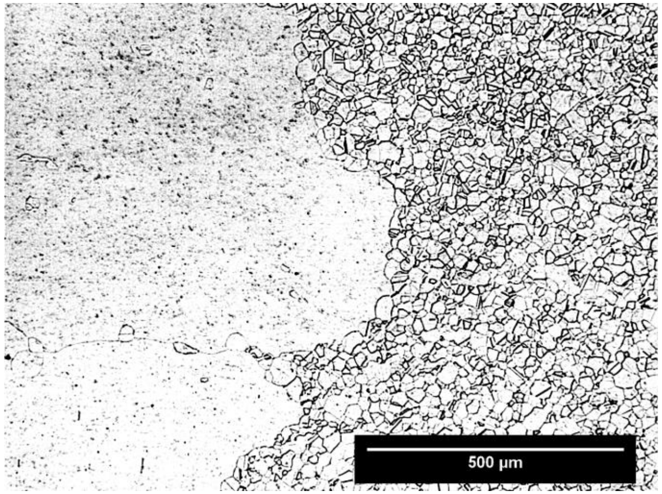
Dilatometer Specimen for SSHT

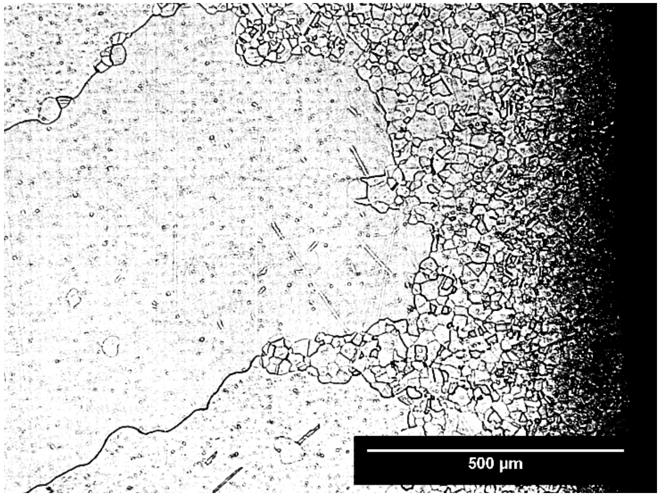


SSHT Specimen

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
- 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

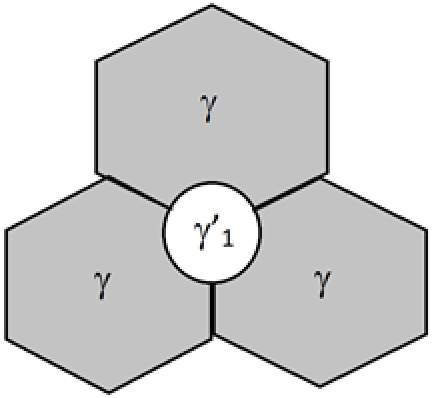




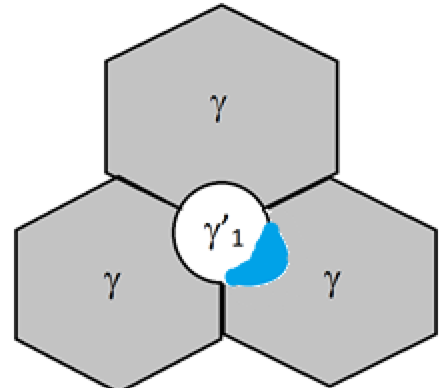
Theory of Abnormal Grain Growth

Coherent γ Recrystallization

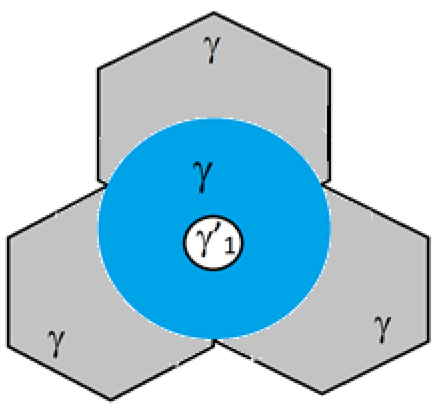
Hetero-epitaxial Recrystallization (HERX) (Charpagne)



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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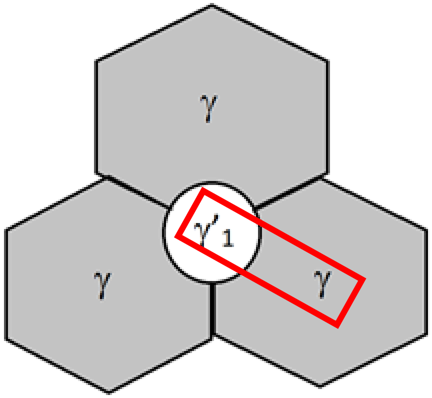

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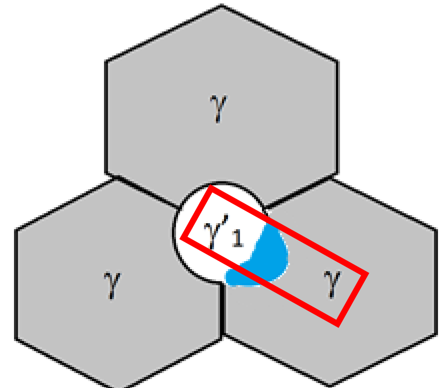

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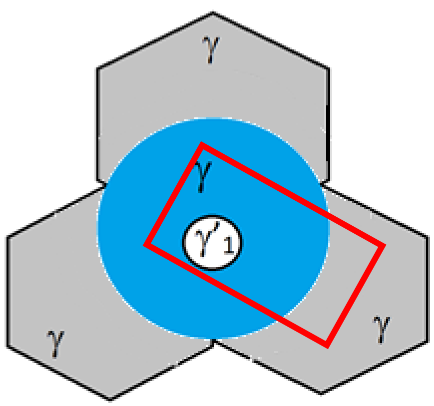
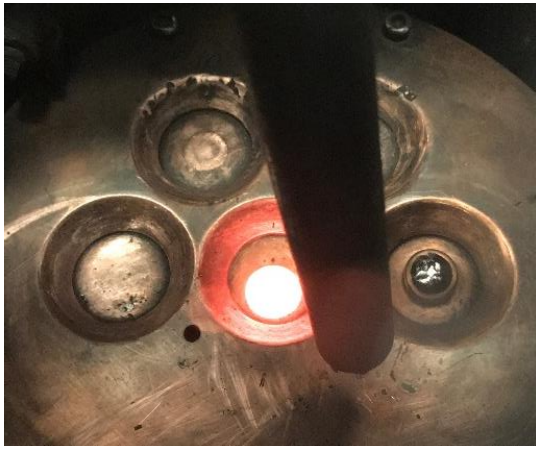


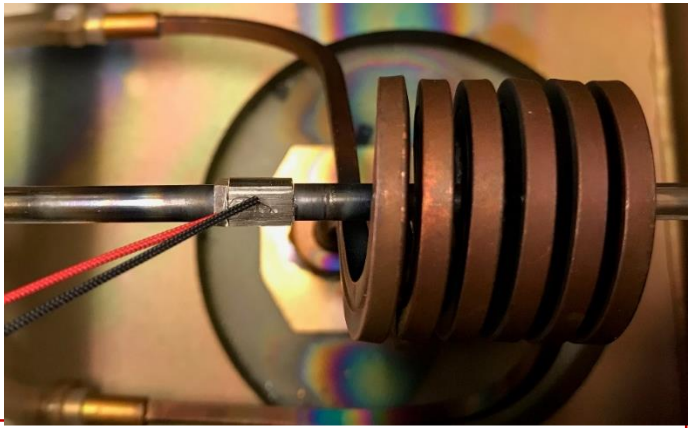
Growth to consume stored energy in γ

- 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

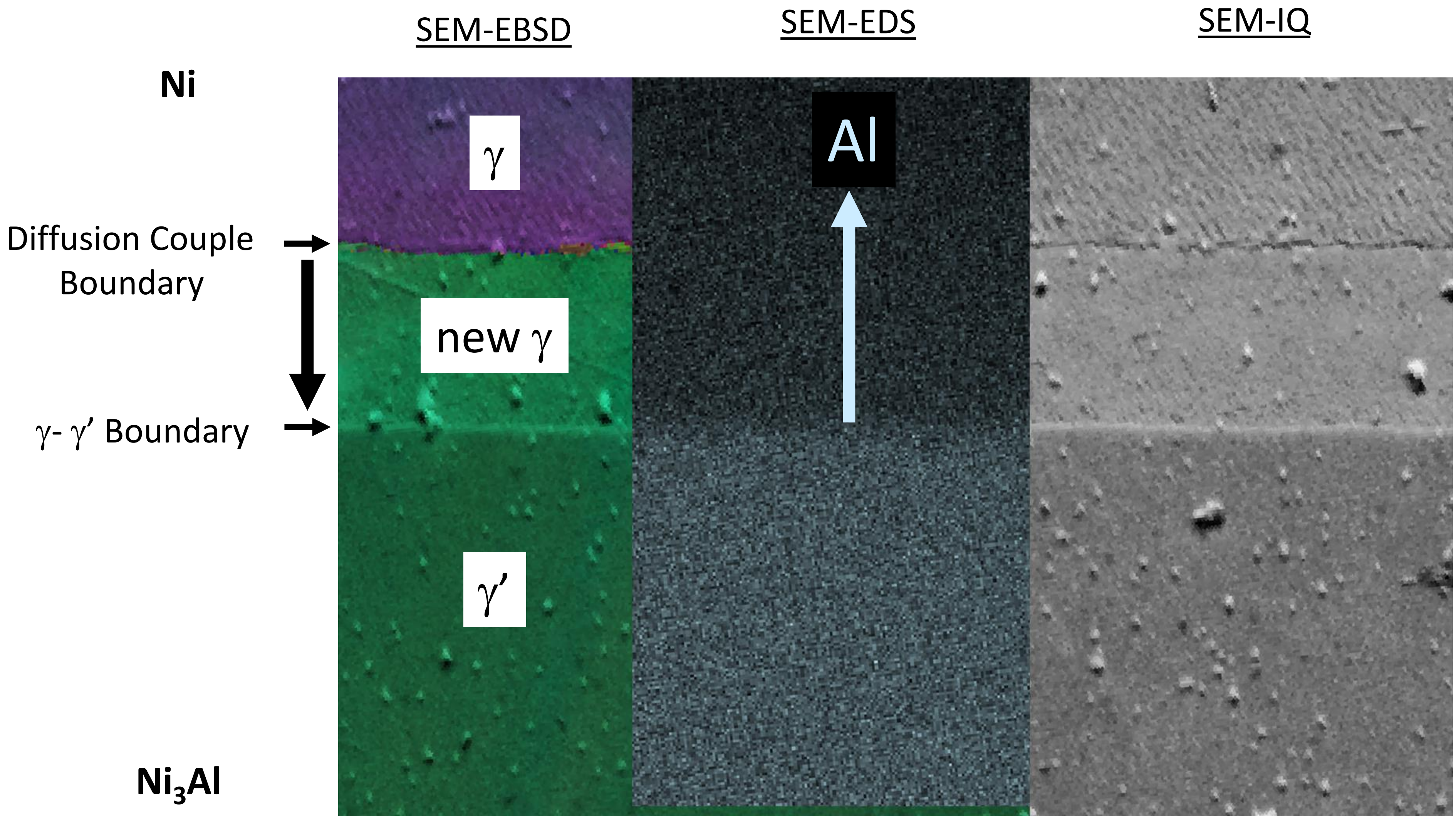
Mesoscopic AGG Study

- 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'



Attempts to Add Deformation

- Ni and IN-625 as γ side
  - Added stored energy through 5-15% deformation (tension)
  - Bonding temperatures (1000°C) allowed for annealing to reduce stored energy
- Super solvus heat treated RR1000 as γ side
  - Added stored energy through 5-15% deformation (compression)
  - Microstructure stable at bonding temperature (1000°C)
  - Issues with oxide preventing bonding (Cr, Ti, Al content)
    - Interface temperature control difficult
  - Demonstrated AGG

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  - Demonstrated AGG **???**

Microstructure Stability Testing
RR1000 (Not As Diffusion Couple)

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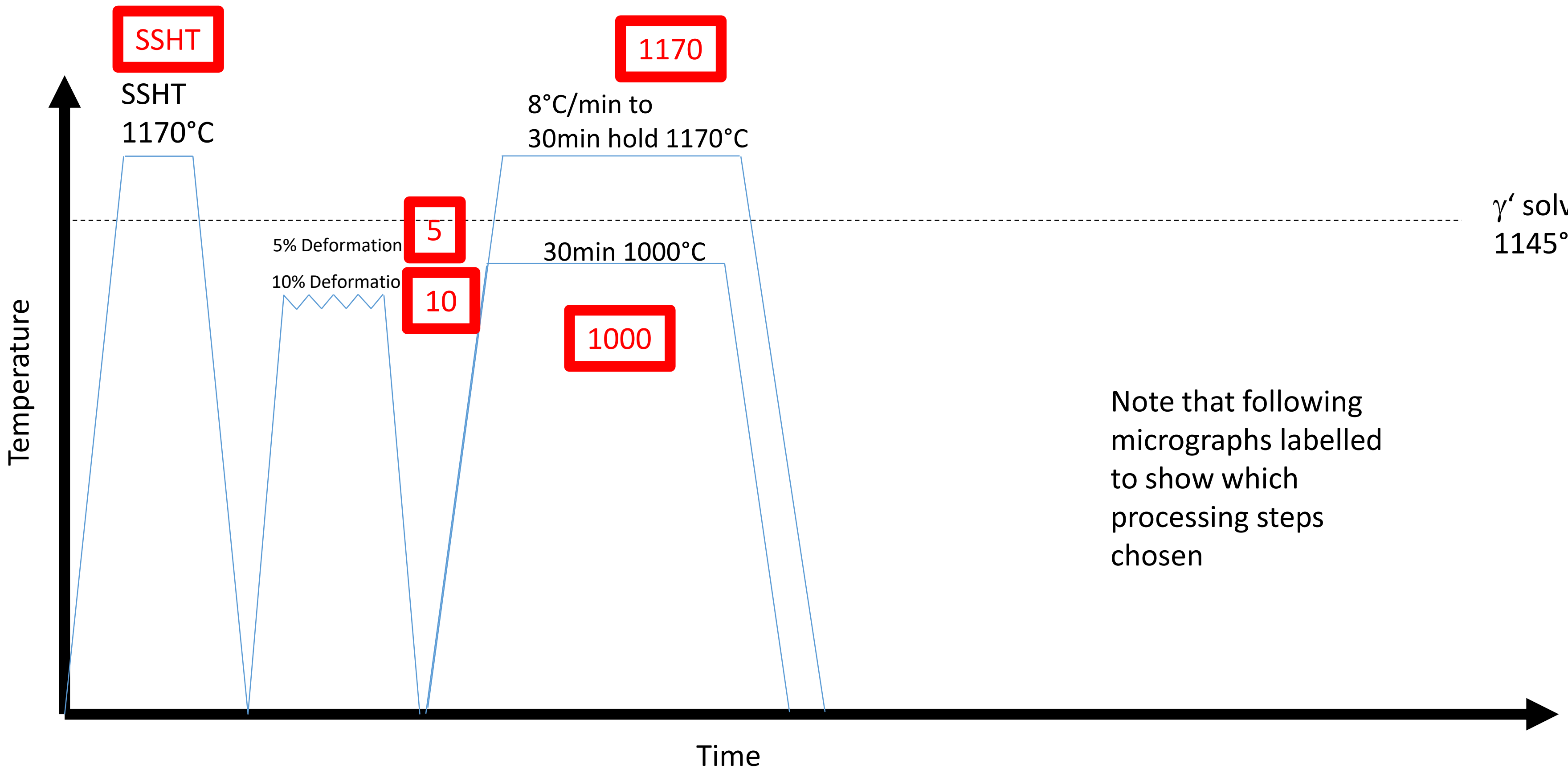
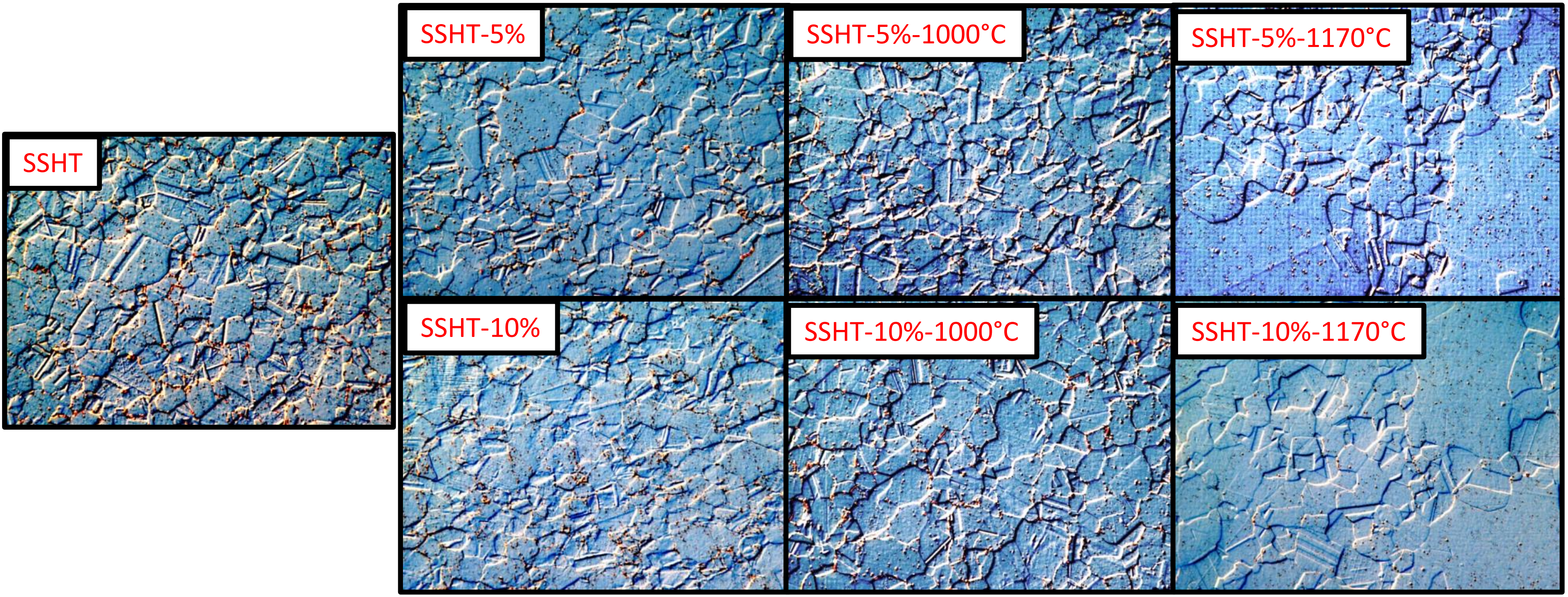
Processing Schematic – RR1000



Note that following micrographs labelled to show which processing steps chosen

Microstructure Stability Testing

RR1000 (Not As Diffusion Couple)



- SSHT-5/10 demonstrate slip bands
- SSHT-5/10-1000 show no recovery, RX, GG. Slip bands still present
- SSHT-5/10-1170 show AGG in regions of lower deformation
  - Likely indicates that large coherent γ' can seed AGG, like incoherent γ'

LOM, etched w/ Mod. Kal

Microstructure Stability Testing

RR1000 (Not As Diffusion Couple)

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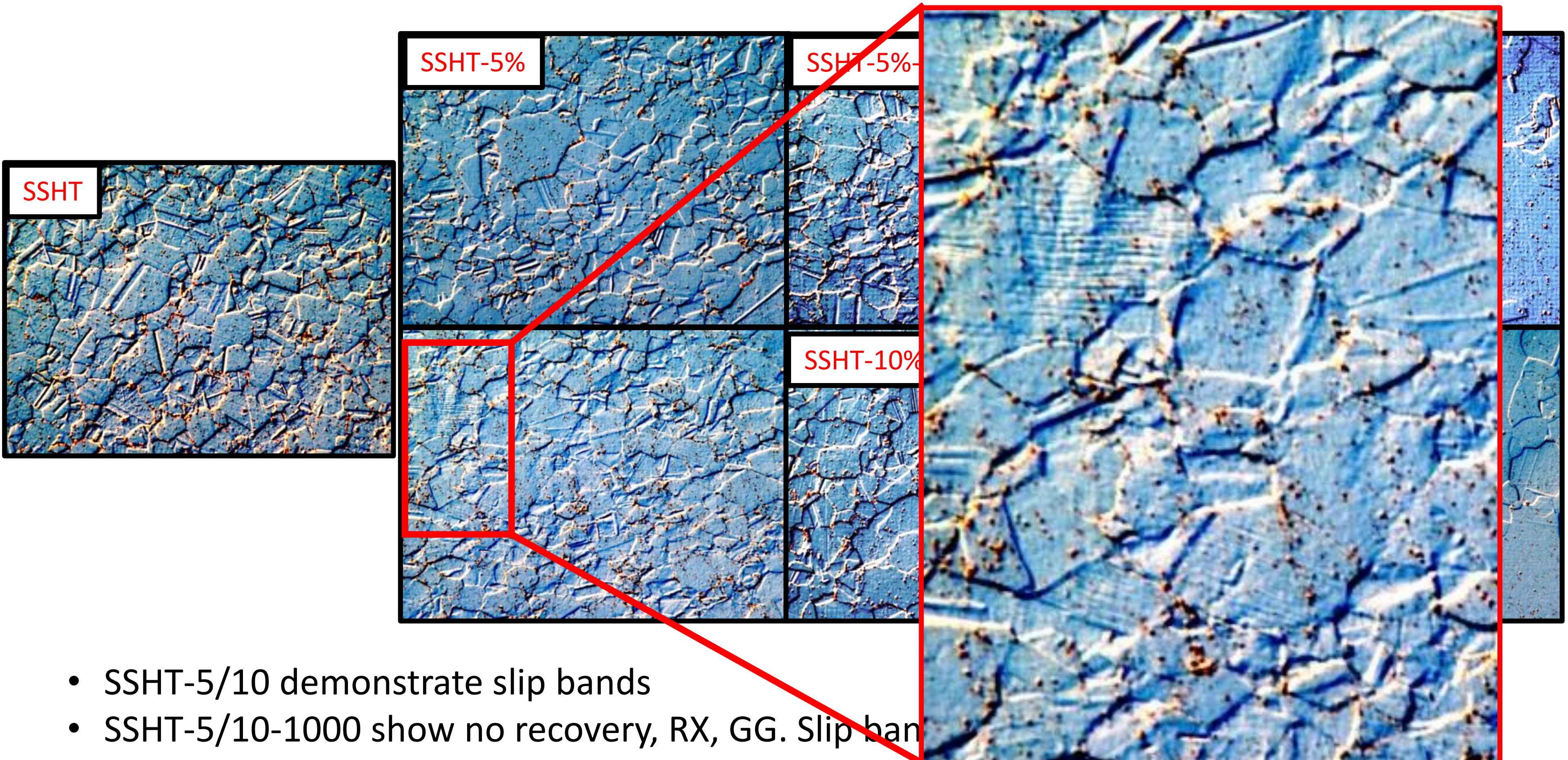

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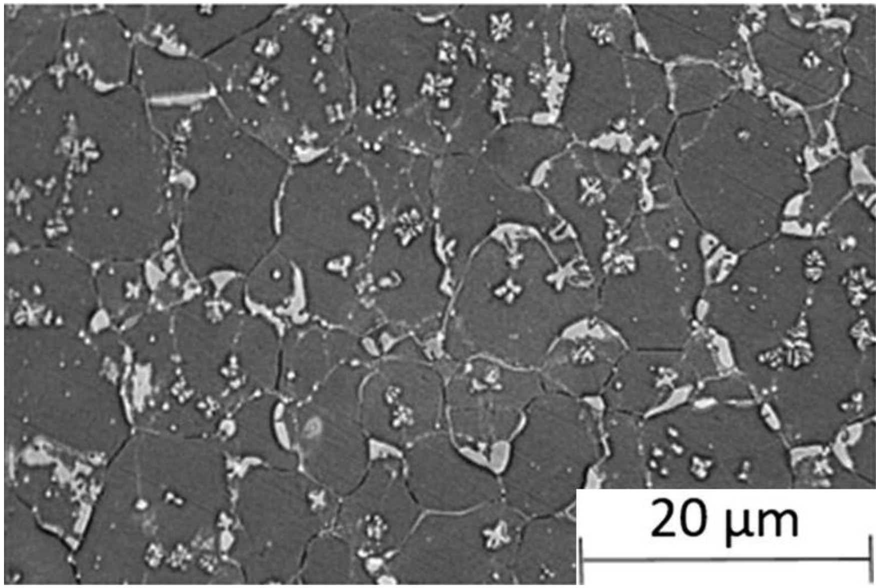
- SSHT-5/10-1170 show AGG in regions of lower deformation
  - Likely indicates that large coherent γ’ can seed AGG, like incoherent γ’

LOM, etched w/ Mod. Kal

AGG in SSHT RR1000

- Unexpectedly observed AGG in SSHT'd RR1000
- Slow cooled RR1000 able to produce large coherent γ'
- Will repeat test to confirm rapid cooling from SSHT does not produce AGG
  - Requires large γ' for nuclei?
  - Coherency not important?

SSHT Slow Cool RR1000



[1] N. D'Souza, W. Li, C. Argyrakis, G.D. West, C.D. Slater, Metall. Mater. Trans. A Phys. Metall. Mater. Sci. 50 (2019) 4205–4222. doi:10.1007/s11661-019-05330-w.

In Progress Testing
Growth of Stable γ AGG Nuclei

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- Nickel and IN-625 specimens are not promising model systems due to lower temperature RX
  - However, still testing Ni-Ni_3Al specimen bonded prior to deformation
- RR1000-Ni_3Al diffusion couple possibly promising if bonded
  - Adding creep deformation to disrupt oxide
  - Reducing gas (H and Ar/N) not currently feasible
- Bonding and deformation issues possibly avoided through Ni-Al button-melt containing 40-70% volume fraction Ni_3Al

1. Slow cooled to control large γ’ size & fraction

2. Deformed to add stored energy

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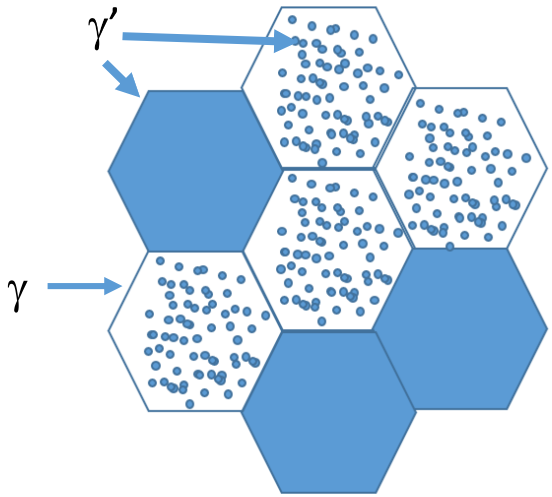
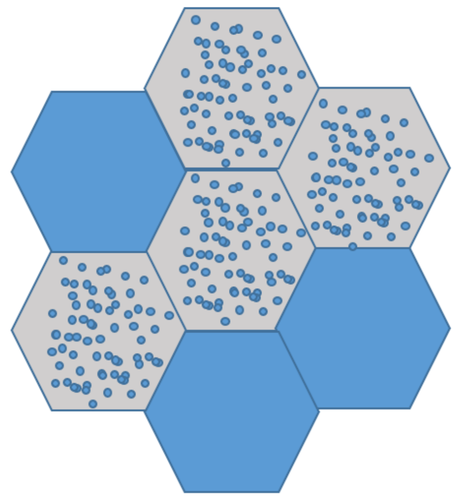
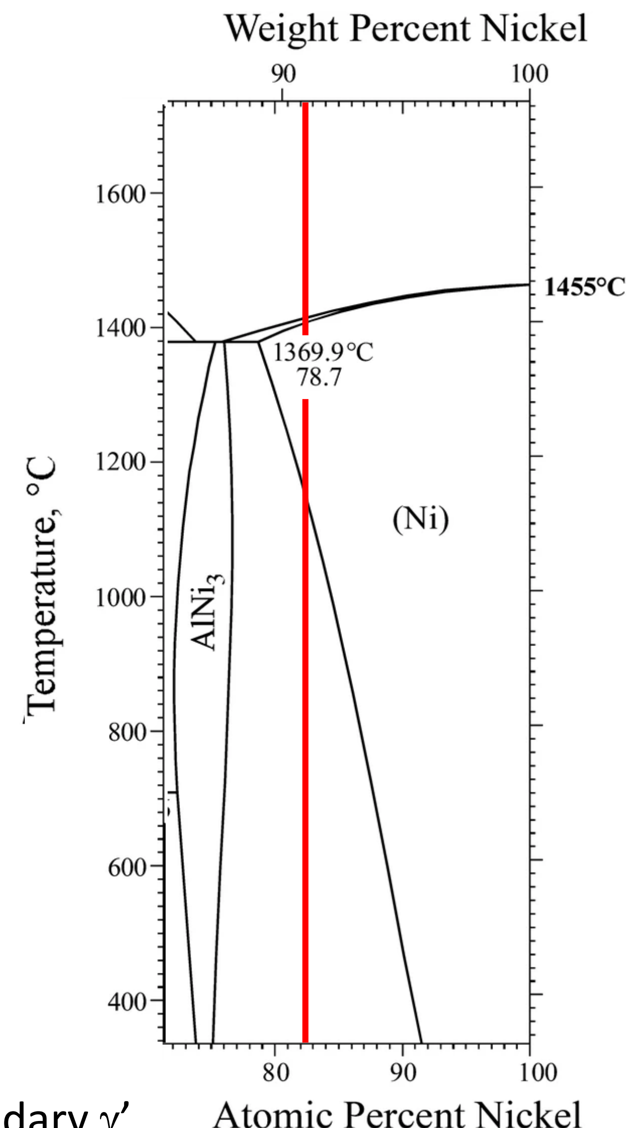
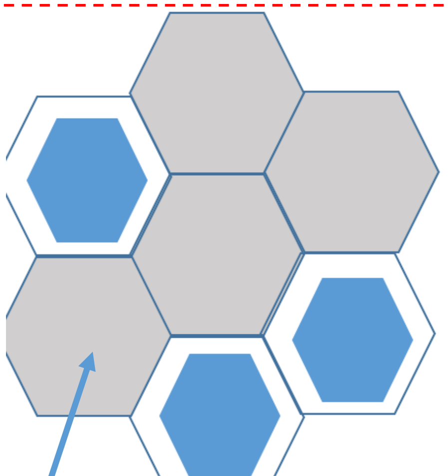




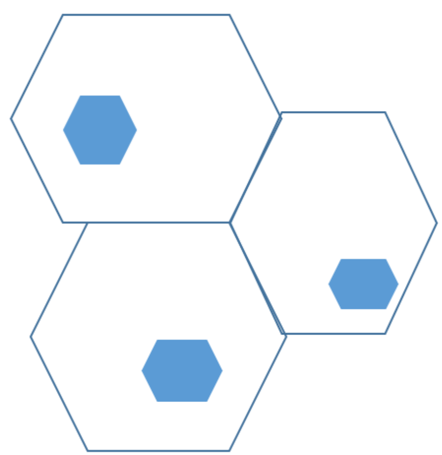


3. Heated to dissolve γ’

4. Heated to grow nuclei



Stored energy



Note: repeat without secondary γ’

Future Work

Diffusion Couple Experiments

- Demonstrate growth of strain free γ nuclei to consume deformed γ
- Able to perform in single Ni-Ni_3Al specimen?

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

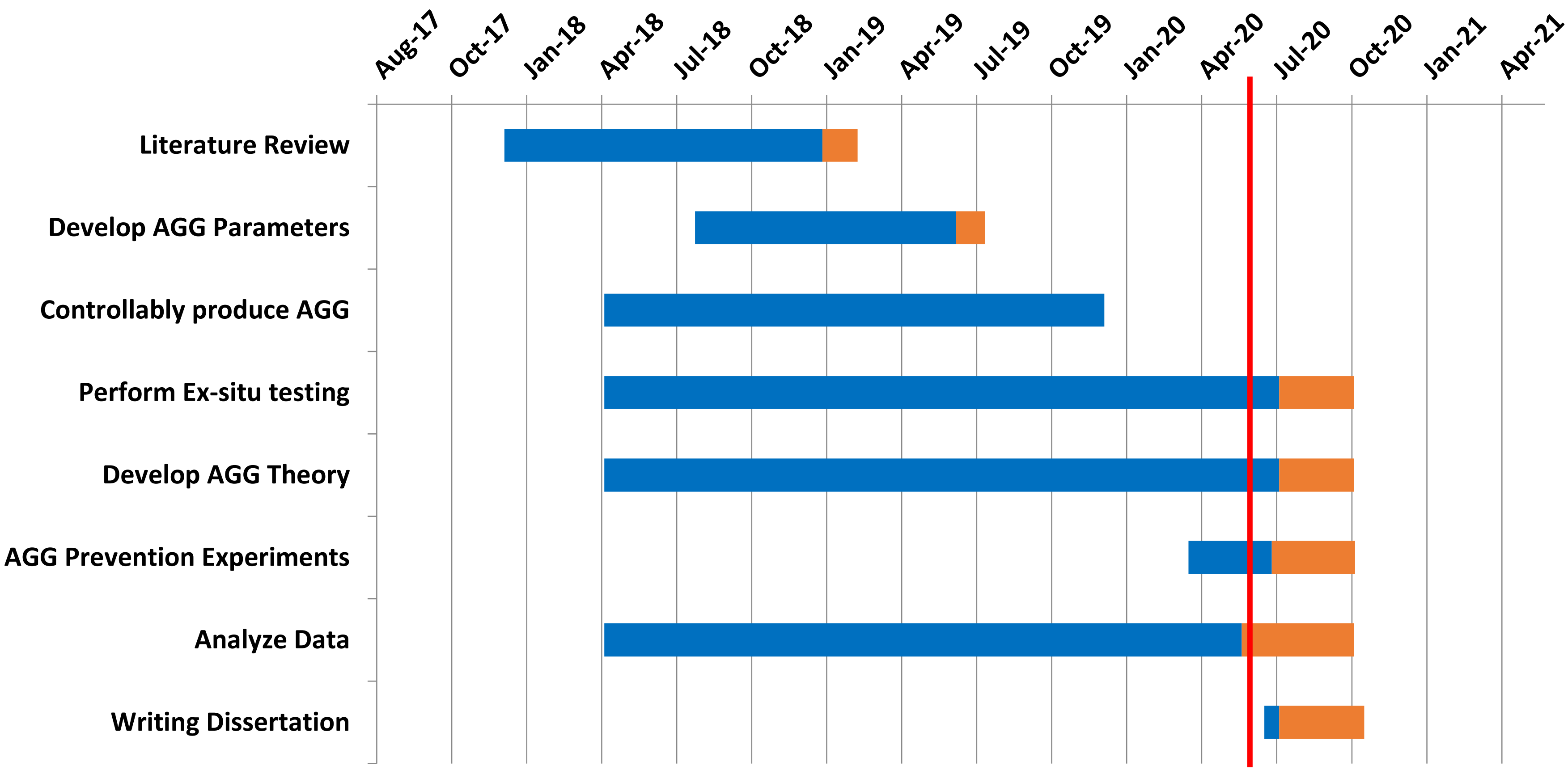
Future Work

<u>Purpose of Experiments</u>

- Demonstrate AGG is a direct result of large γ' dissolving to provide strain free γ nuclei to grow in a matrix of stored energy γ
  - Mesoscopic γ'-γ interface showing stable γ forming in prior γ'

- Demonstrate AGG is <u>not</u> a result of spatially selective RX, reliant upon inhomogeneous deformation and secondary γ' and/or carbides/borides/oxides
  - Rapidly cooled & aged SSHT specimens with prior AGG processing

Progress


Aug-17
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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

Challenges & Opportunities

- Additional insight into isothermal forging of turbine discs may be helpful for providing processing routes to avoid AGG
- Help or advice on diffusion bonding oxidized surfaces
- Coarsening γ' methods
  - Planning slow cooling from solvus with hold

Thank you!

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