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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 Superalloys

Spring 2019 Semi-Annual Meeting
Iowa State University, Ames, IA
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Student: Byron McArthur (Mines)

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

Industrial Mentor: Kevin Severs (ATI)

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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
- Quantifying primary γ' percentage vs. temperature
- Improved thermomechanical processing method
- Repeatably produced AGG
- Performing interrupted heat treating
- Proposed mechanism for 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%	●

Industrial Relevance

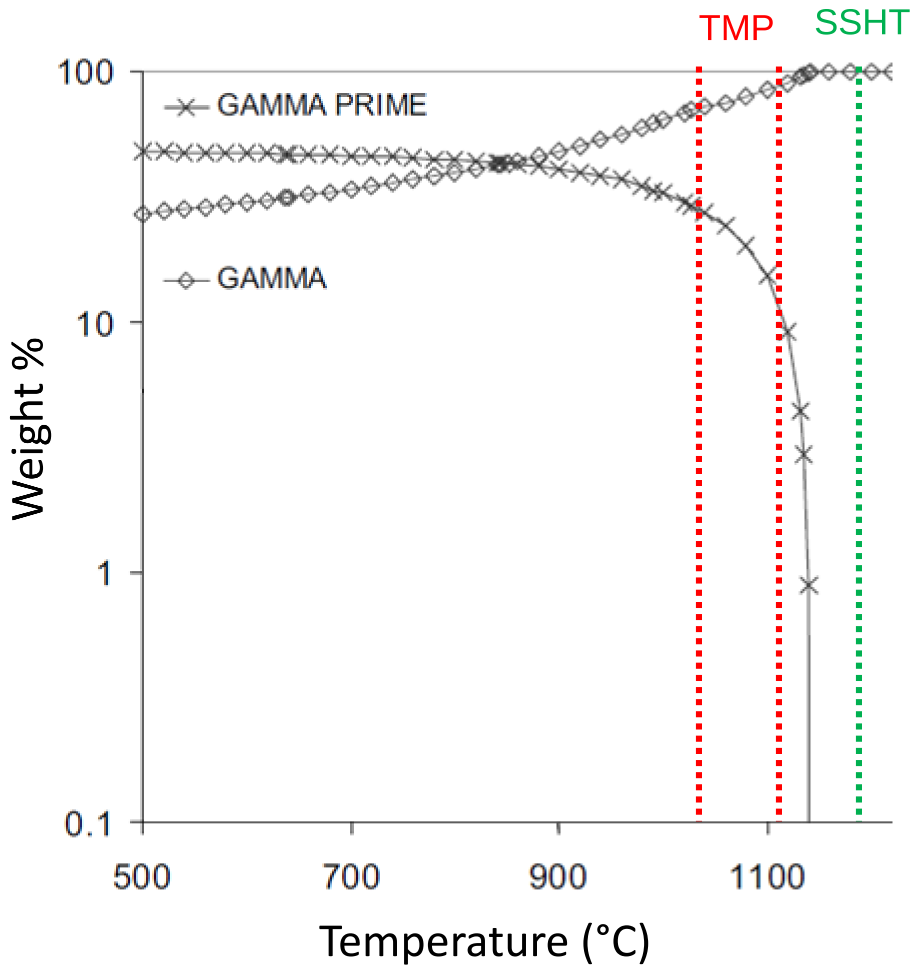
- Ni-based superalloys are used in turbine engine disks
  - Flight-critical components
- Forging parameters influence abnormal grain growth (AGG)
  - Reduction in fatigue life
- Phenomena observed to other superalloys and material systems

High pressure turbine disk
Approximately 50cm diameter

https://jetartaviationshop.co.uk/product/raf-sepecat-jaguar-aircraft-rolls-royce-adour-jet-engine-hp-turbine-disc-aviation-art/

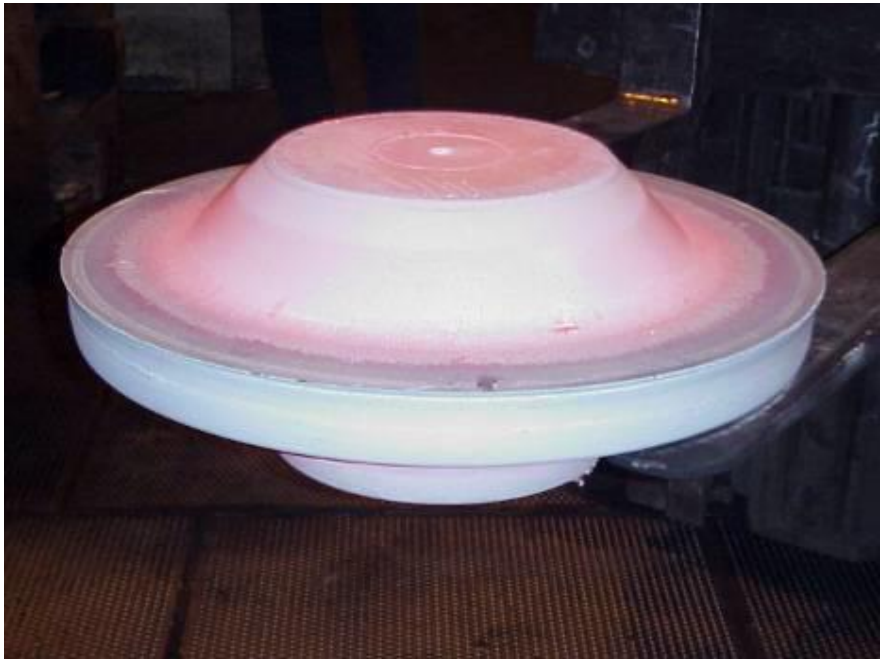
Material: RR-1000, γ-γ’

- 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

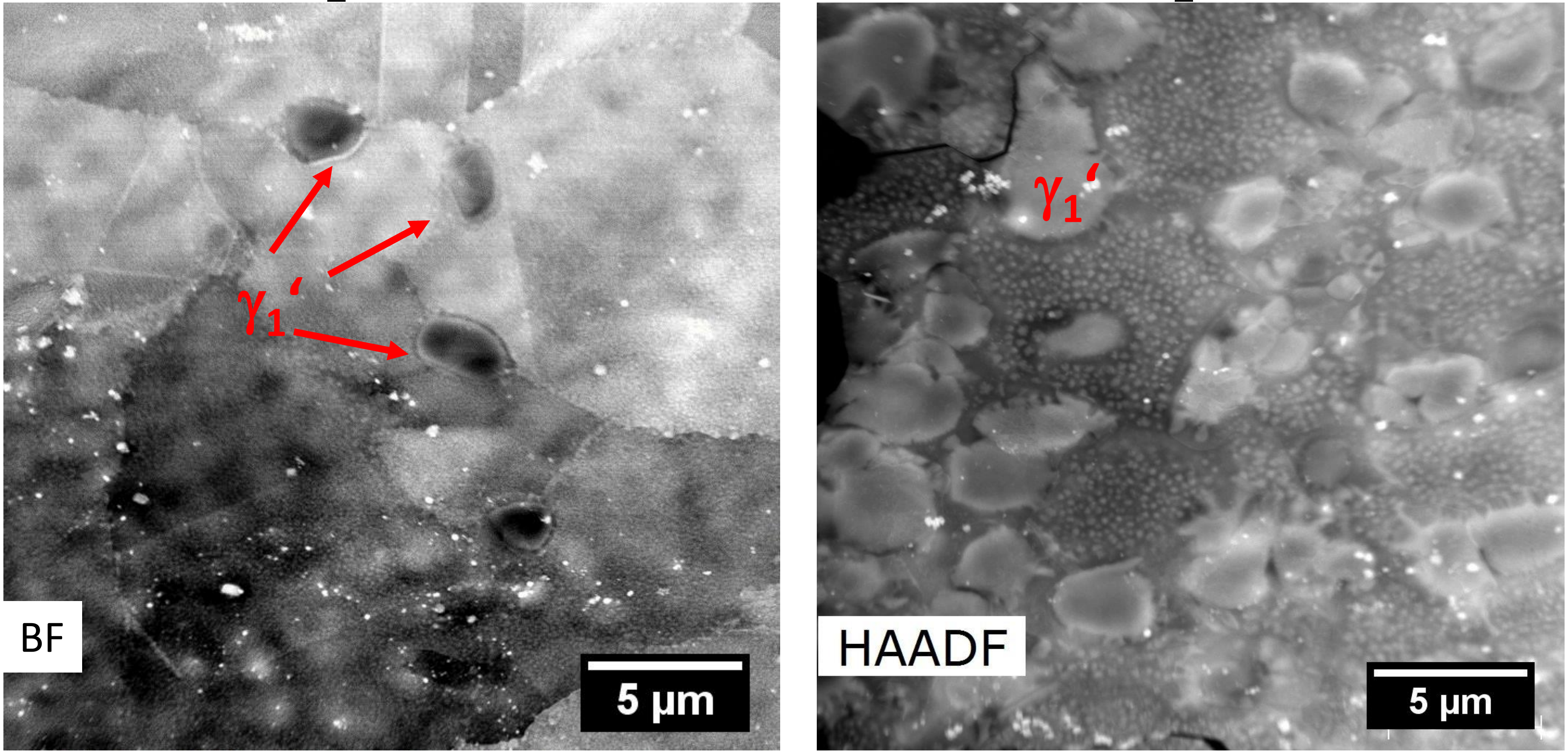


Approximately 1m diameter

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**

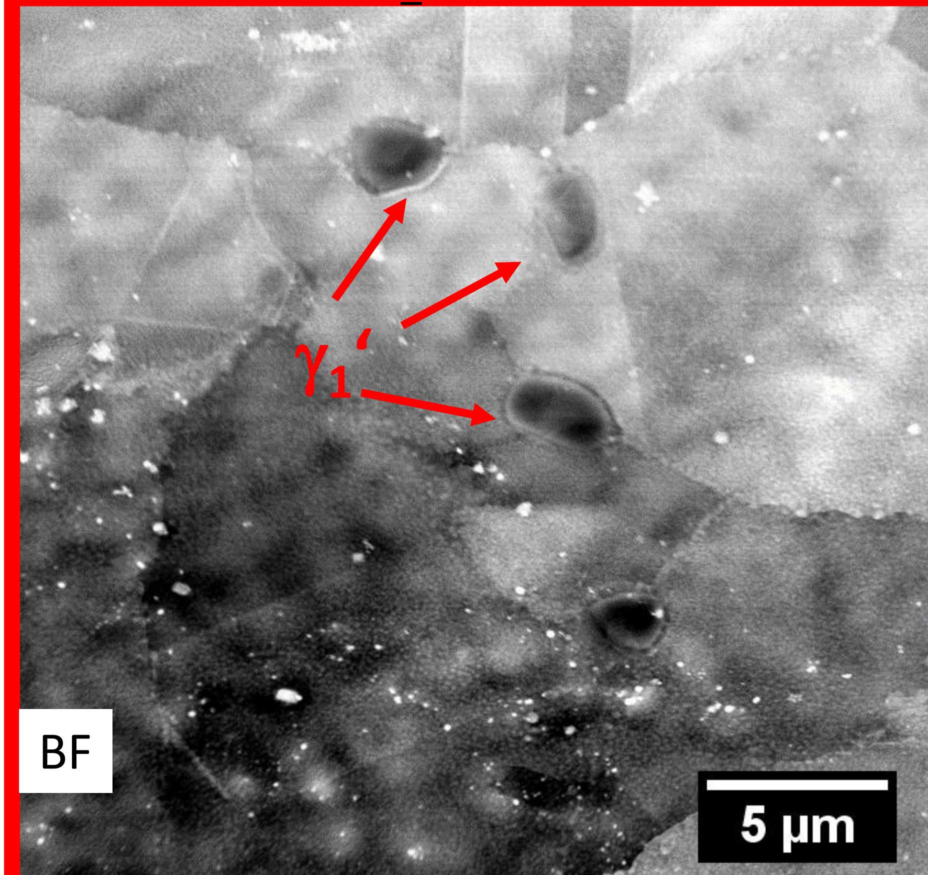


Note larger size of primary and secondary γ' as well as increased primary γ' fraction in Sample 2

Thanks to Yaofeng Guo for TEM imaging

As-received Material

Low γ_1‘ Fraction



High γ_1‘ Fraction

[redacted]

Note larger size of primary and secondary γ‘ as well as increased primary γ‘ fraction in Sample 2

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Experimental Procedure

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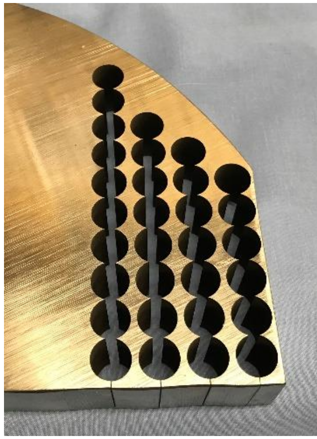
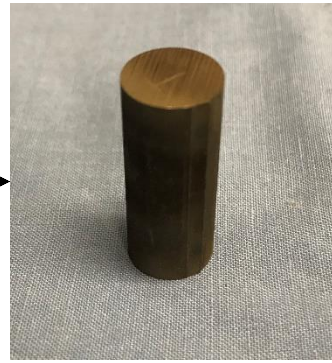
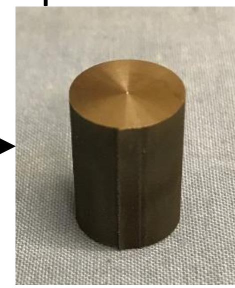
Wire-EDM

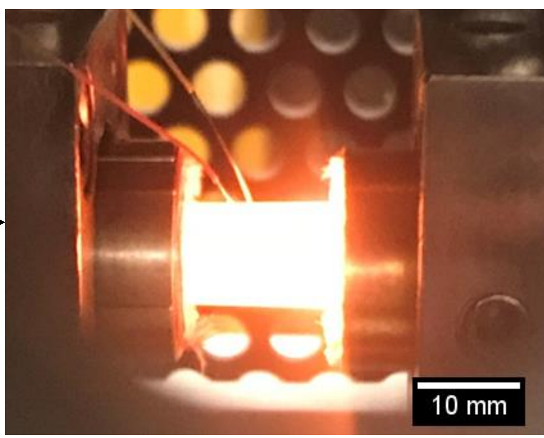




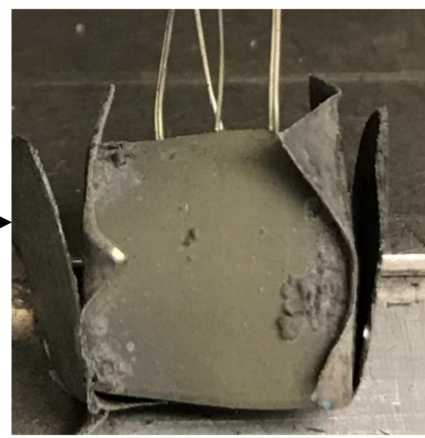
Machine to length with parallel faces



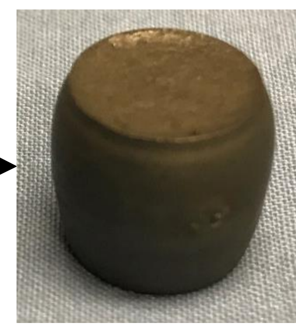
Isothermal forging in Gleeble®



Thermocouple locations

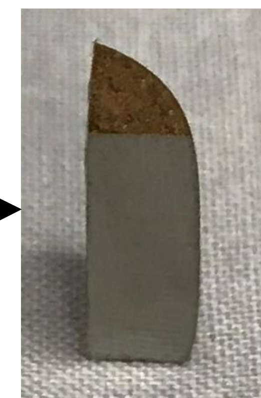


*Deformation gradient



As-deformed Specimen

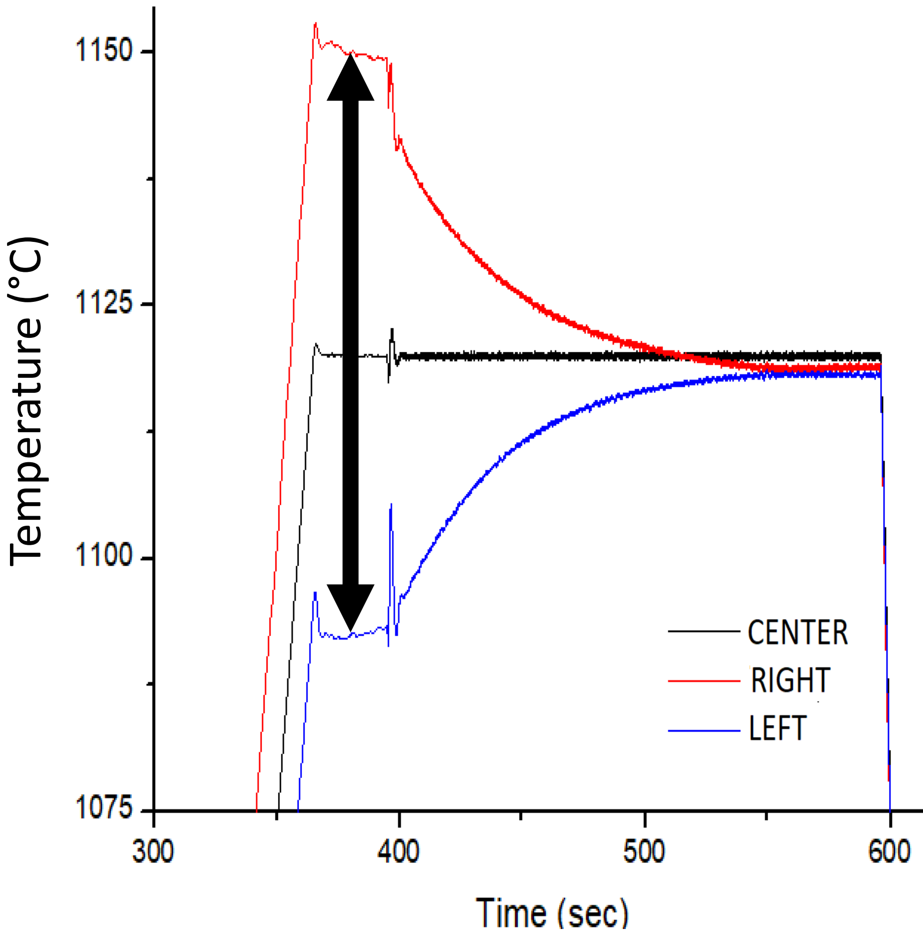
Dilatometer Specimen for SSHT



SSHT Specimen

Prior Gleeble Experiments

- Produced AGG conditions
  - Low strain and strain rate
  - High temperature (low γ_1' fraction)
- Undesirable thermal gradients across specimen
  - Asymmetric
  - Produced varied microstructure
  - Inconsistent between specimens
- Thermal overshooting
  - Load → displacement change
  - Unknown γ_1' at deformation start



Modified Gleeble Experiments

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Changes:

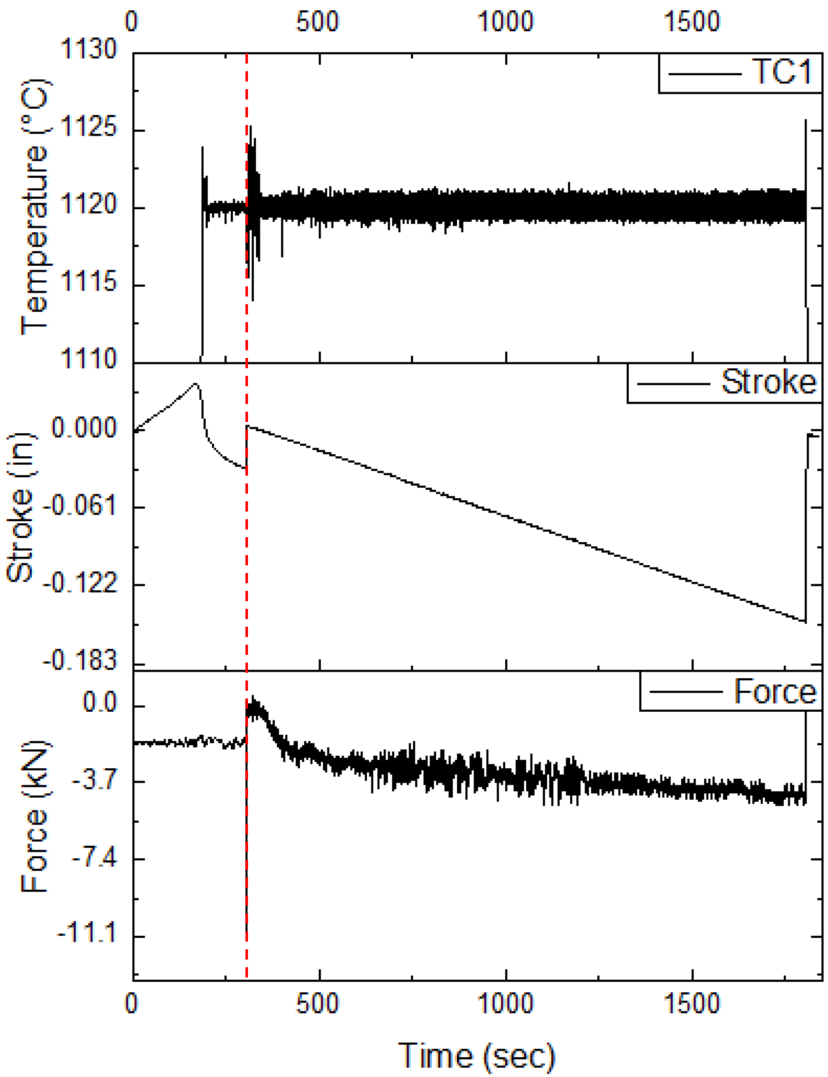
- Larger specimen
- Load-displacement switch at lower temperature
- Increased pre-load

Resolved issues:

- Asymmetric thermal gradients
- Thermal overshoot
- Consistent testing between samples

Results:

- Reproducible AGG
- Consistent with prior results
  - Strain, strain rate, temperature effects on AGG

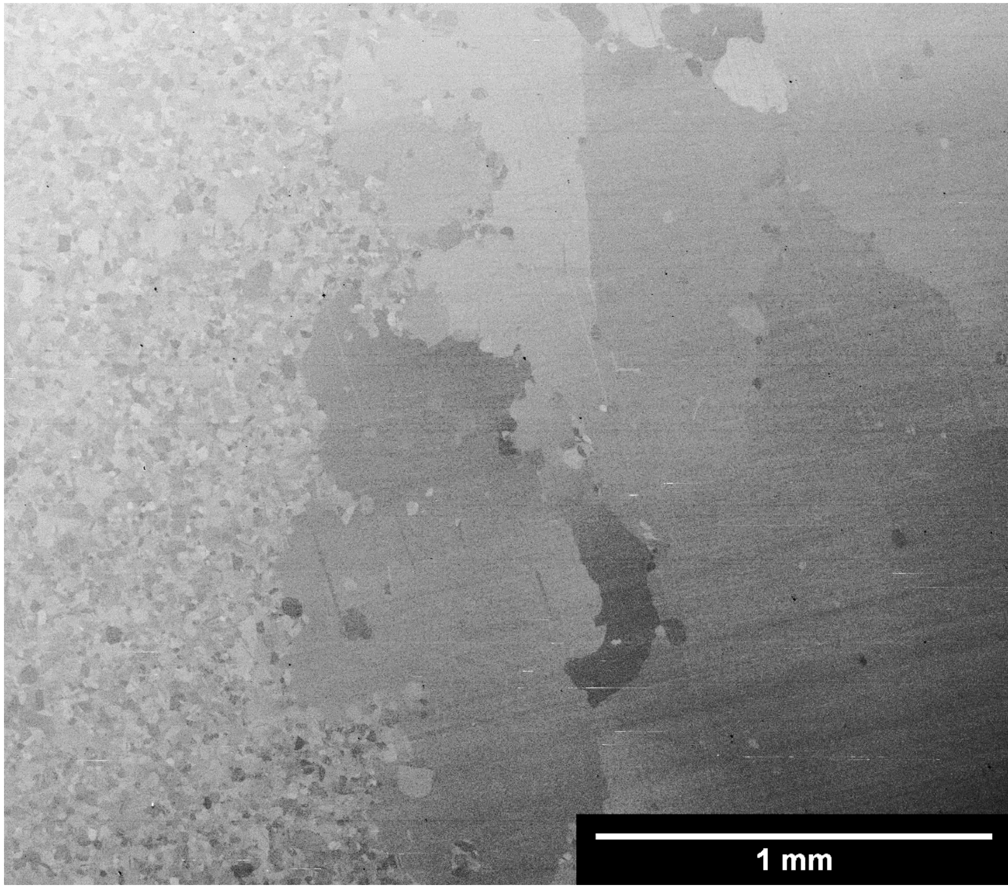


0.1 ε, 1E-5 ε/s

Recent Results

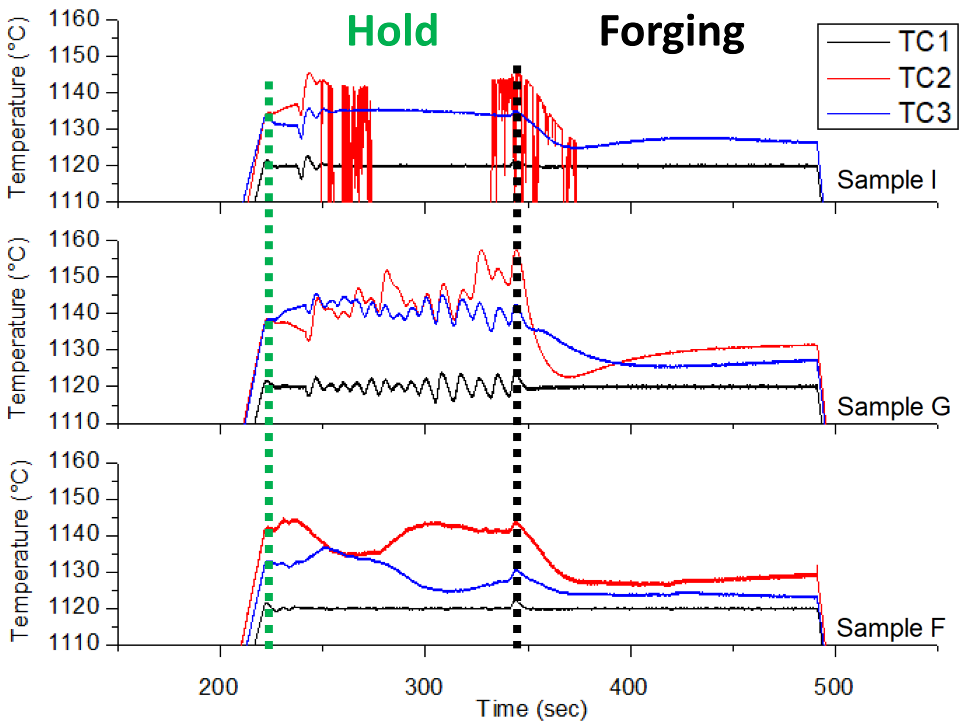
Gleeble Experiments

- Better thermal control
  - 5-20°C gradient
  - Symmetric
- Larger regions to study
- Consistent with prior results
- Producing AGG under conditions of
  - Low strain
  - Low strain rate
  - High temperature (near solvus)



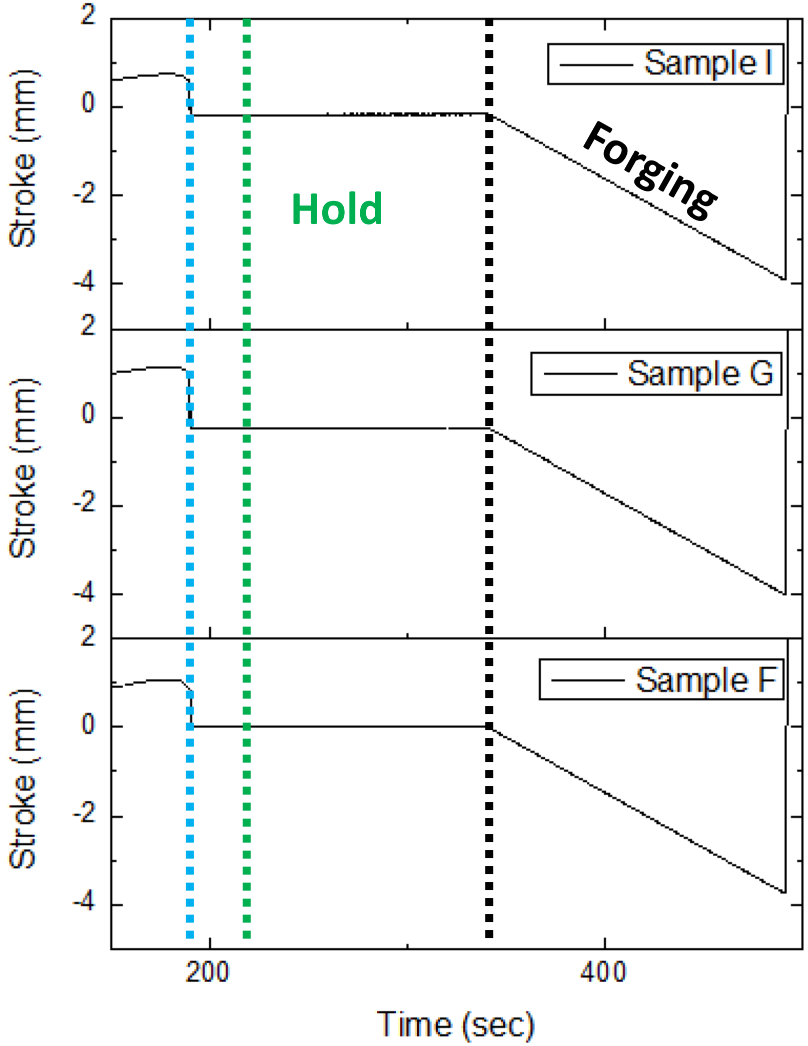
Repeatability Testing

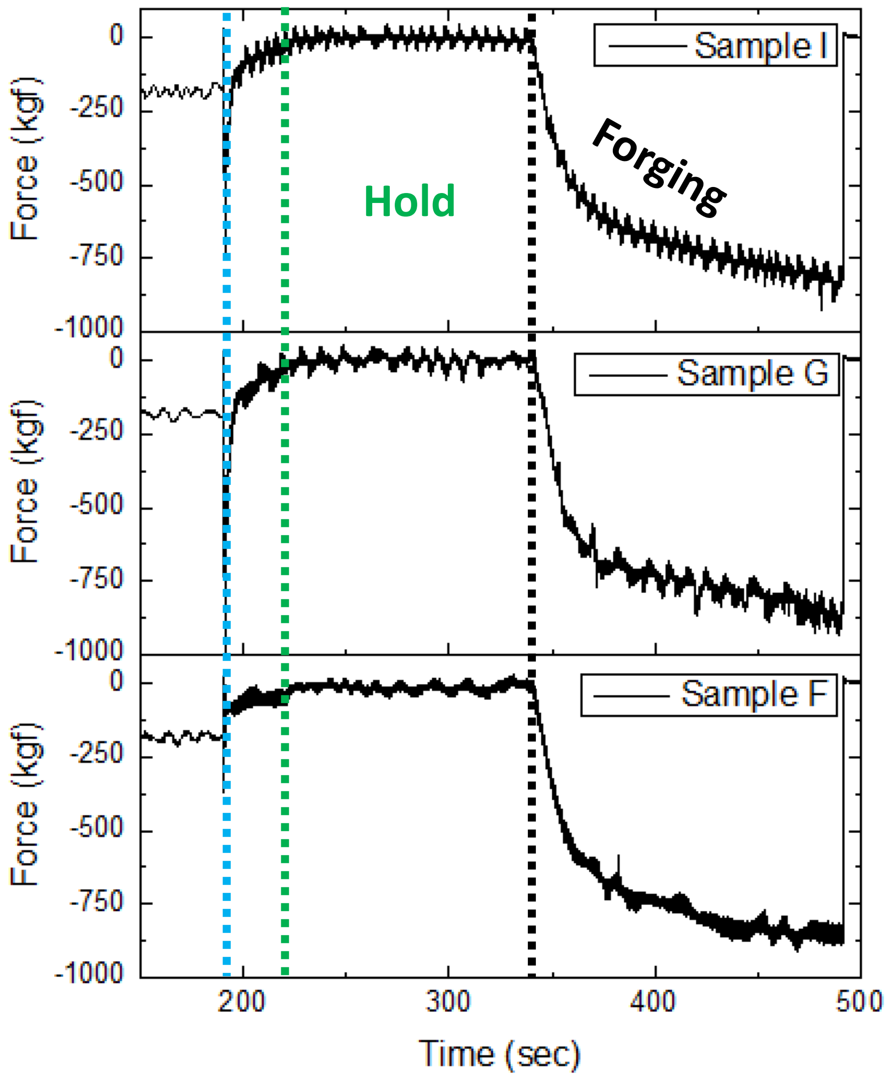
- Testing Gleeble® experimental repeatability
- Ability to consistently create AGG conditions
- Axial thermal gradient remains during hold, reduces during isothermal forging
- Furnace heating (0.13°C/s or 8°C/min) together
  - In bag with titanium 'oxygen getter'



*TC2 connection issues on Sample I

Repeatability Testing

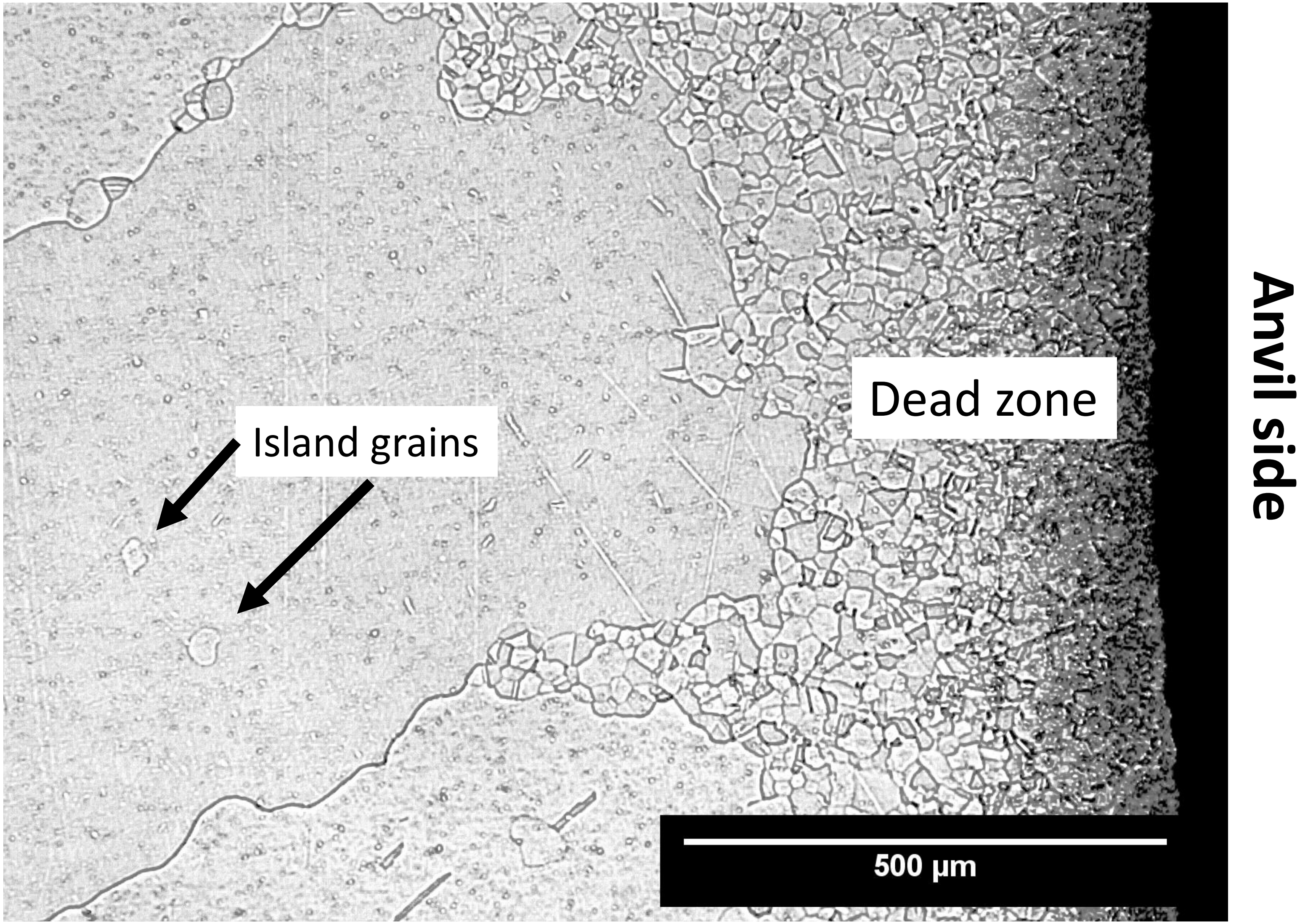




Repeatability Testing

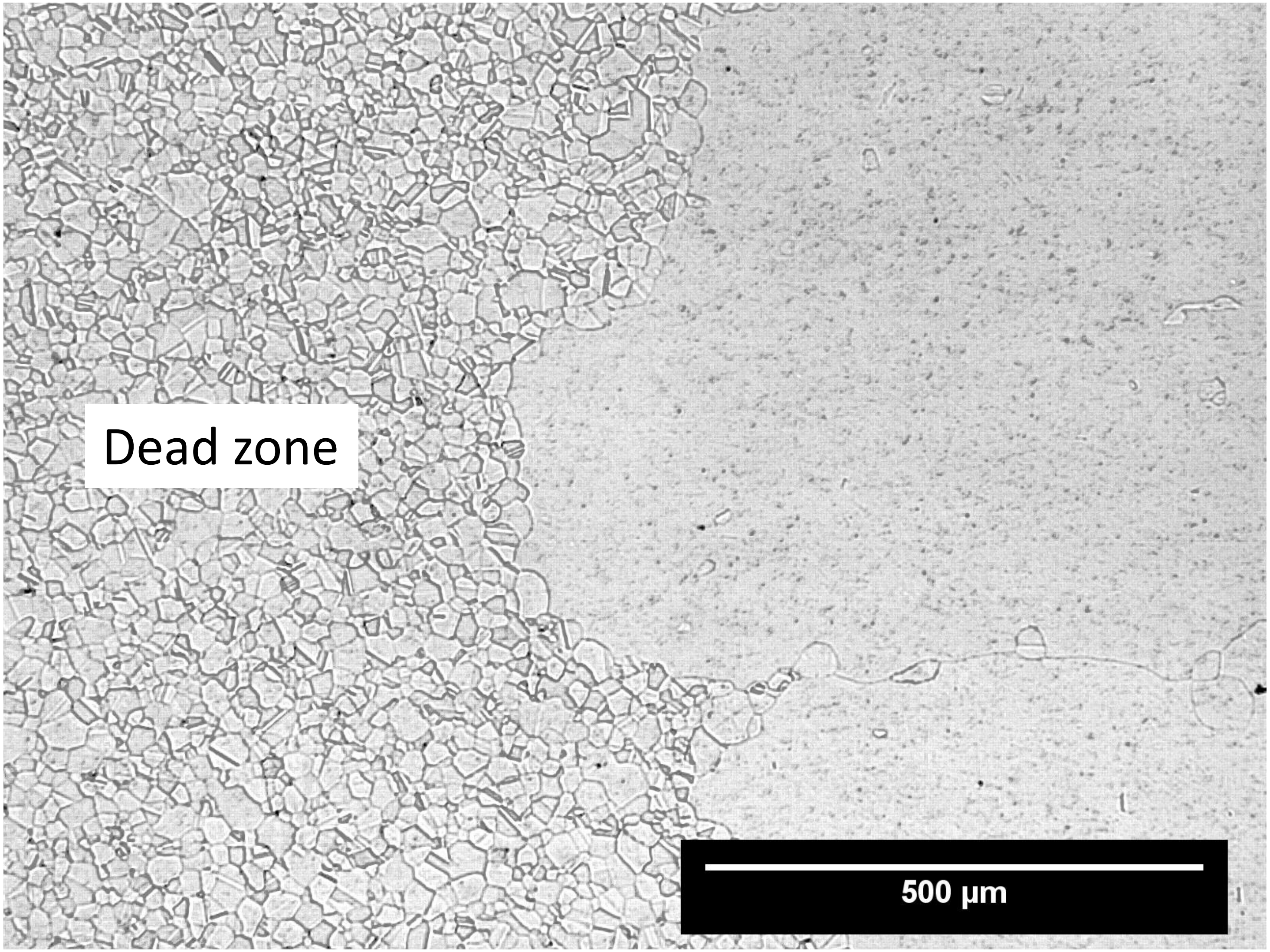
- All 3 Gleeble® specimens sectioned in half and placed in furnace for heat treating
  - Heating to 1170°C at 0.13°C/s
- Analyze produced microstructures and occurrence of AGG
- Intend to produce AGG in consistent regions, and to the same magnitude

Near Anvil – Sample I

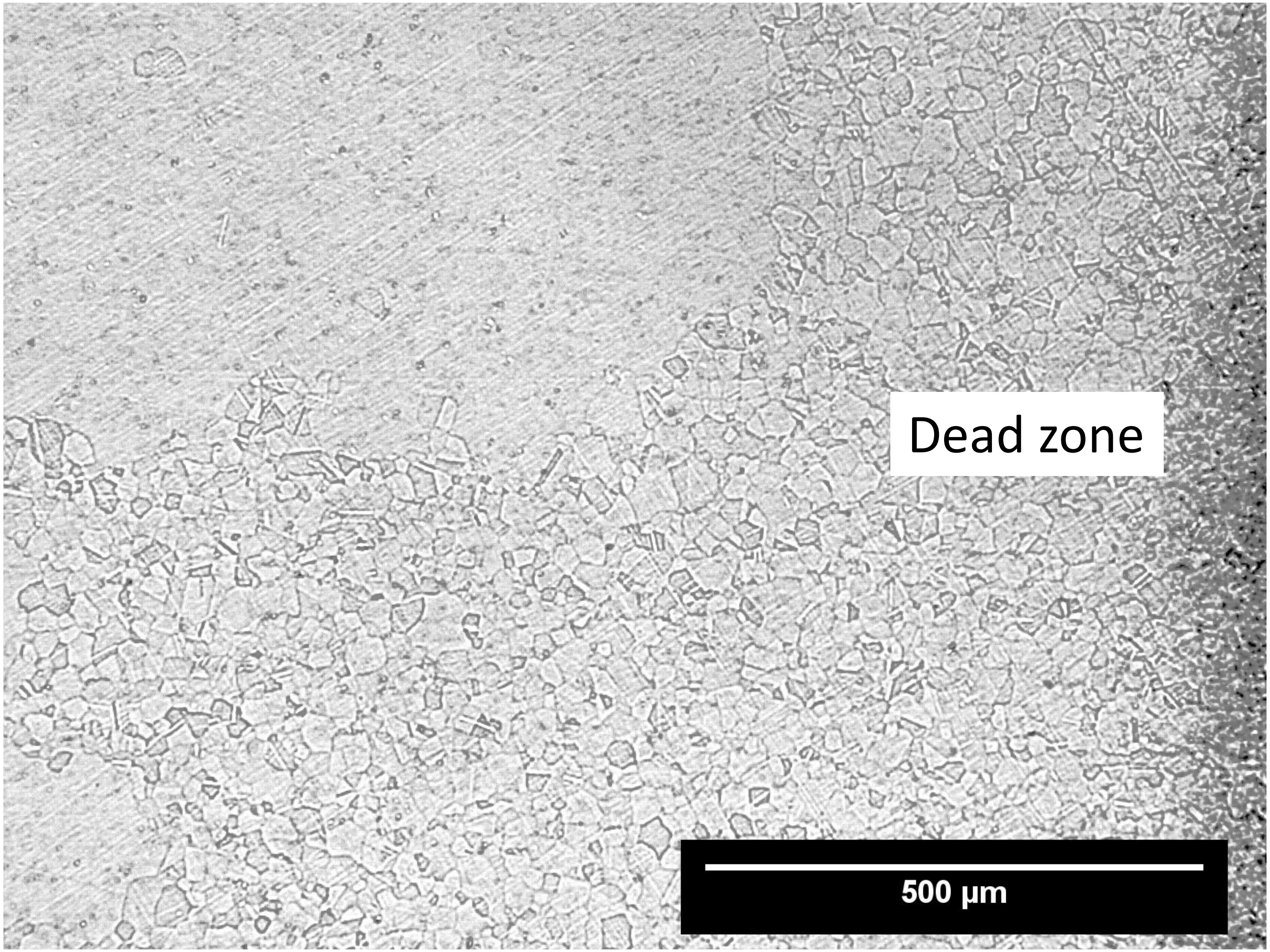


Near Anvil – Sample G

Anvil side

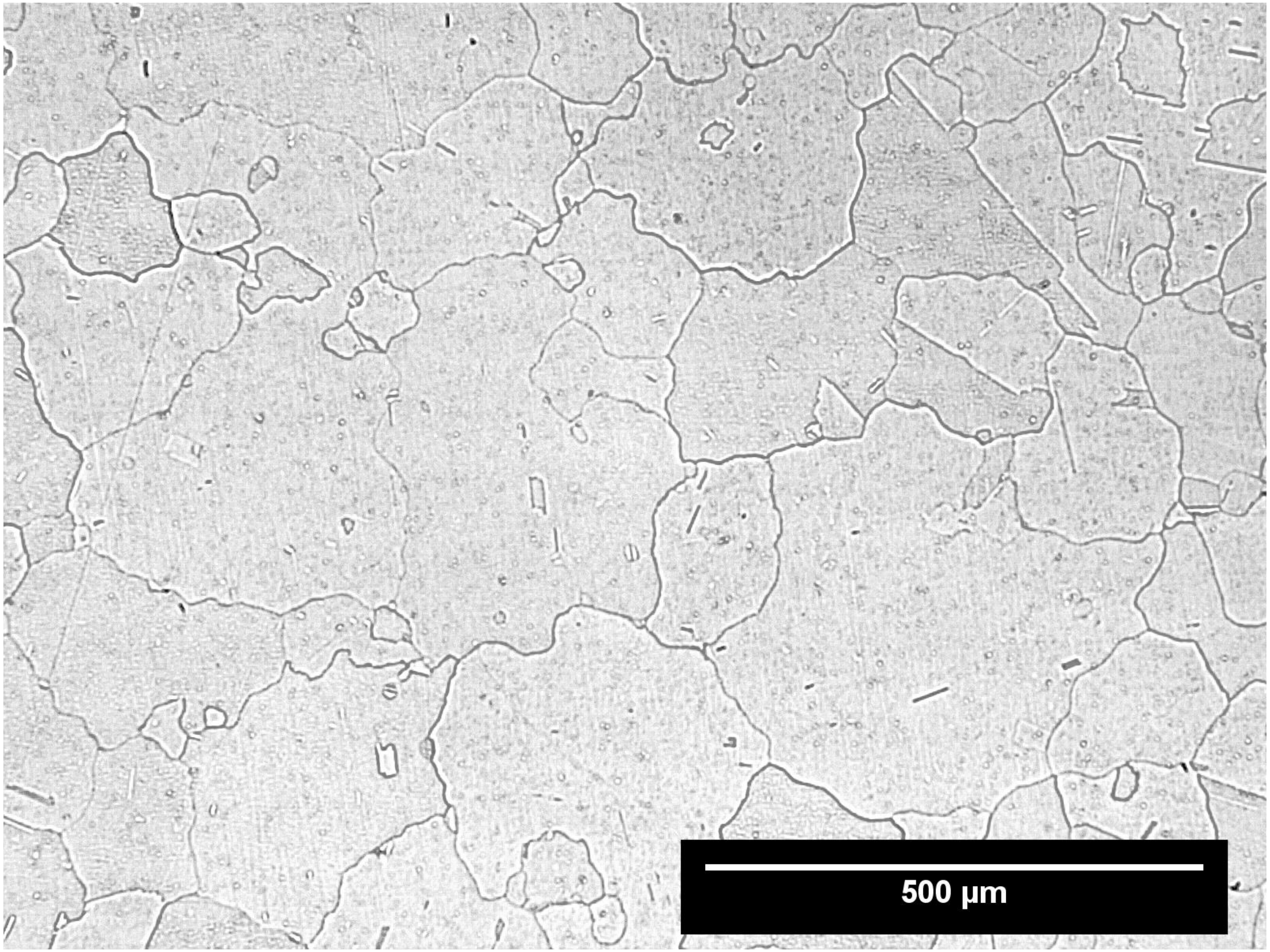


Near Anvil – Sample J

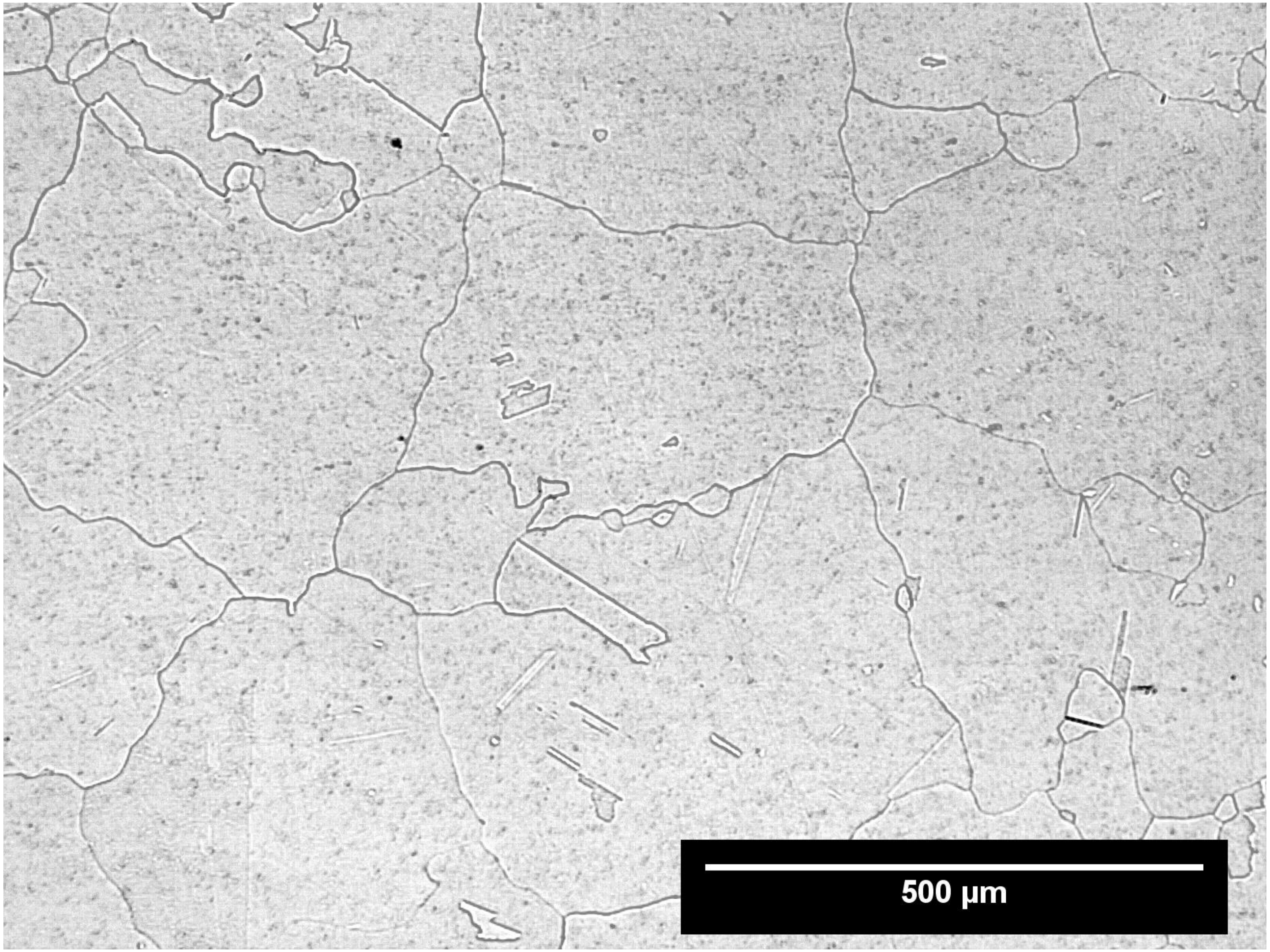


Anvil side

Near Center – Sample I



Near Center – Sample G



Near Center – Sample J

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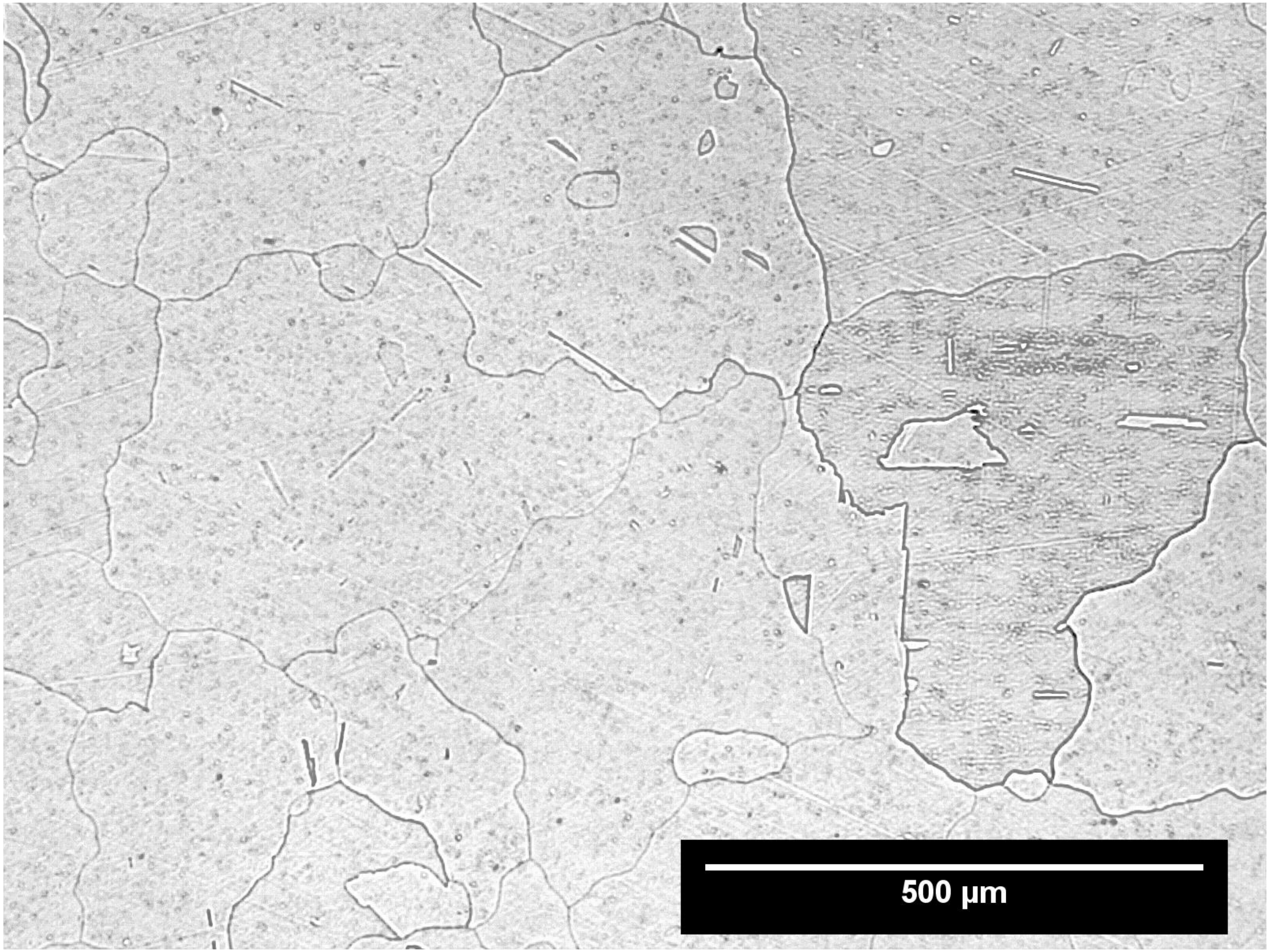


Repeatability Test Summary

- Reduced thermal gradients and thermal asymmetry
- Consistently producing similar:
  - Local deformations
  - Temperatures
  - Flow stresses
  - AGG location & magnitude
- How does AGG progress?...

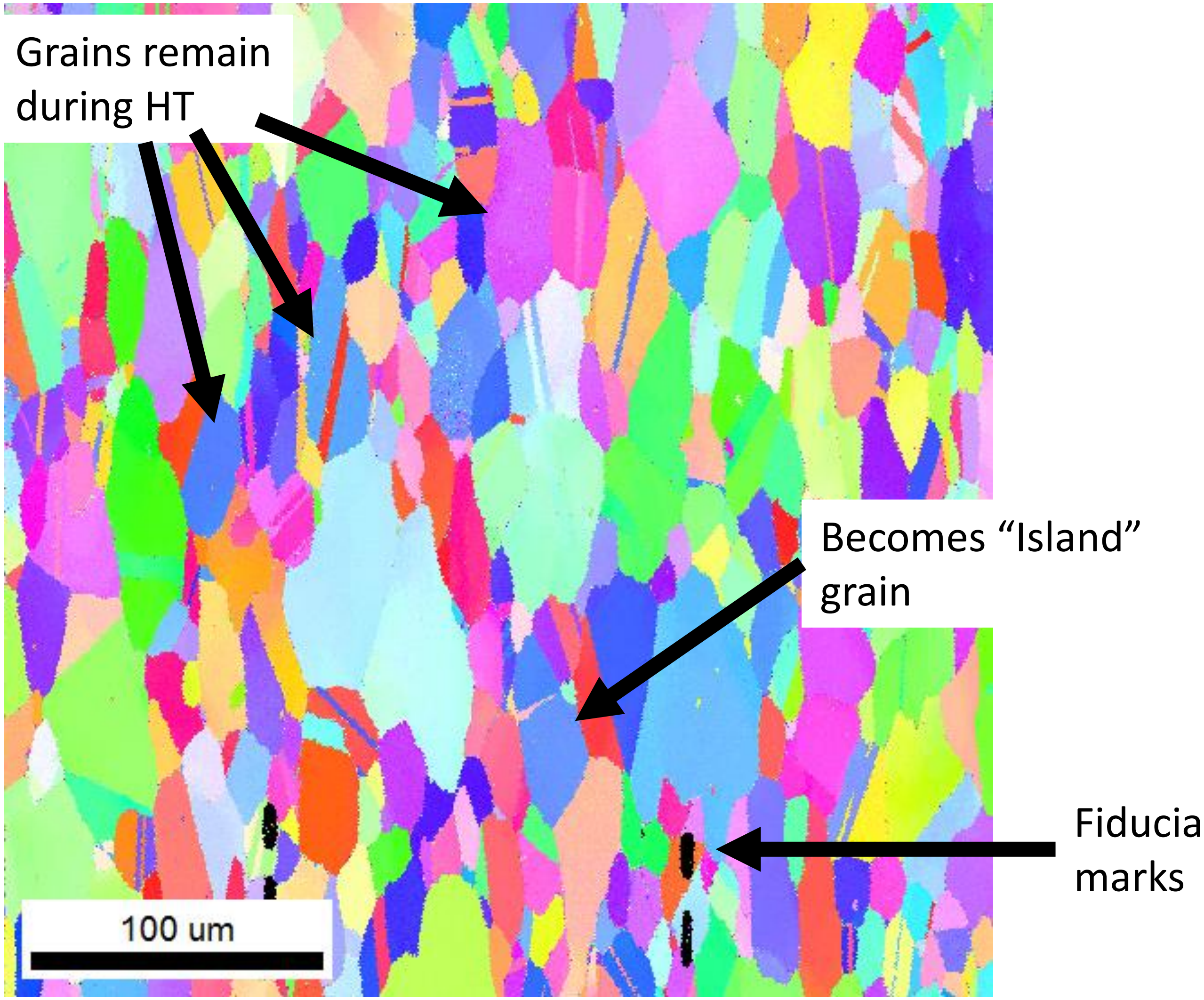
Interrupted Heat Treating

Procedure:

- Gleeble specimen isothermally forged in conditions known to produce AGG
- Small section of Gleeble specimen sectioned out, polished, and imaged
  - Fiducial marks placed with focused ion beam (FIB)
- Specimen iteratively heat treated, polished, and imaged
  - 1130°C, 1150°C, 1170°C
  - Note: 1135°C is γ' solvus
- Micrographs are of the same region
  - Polished with 0.05µm colloidal silica to remove minimal material

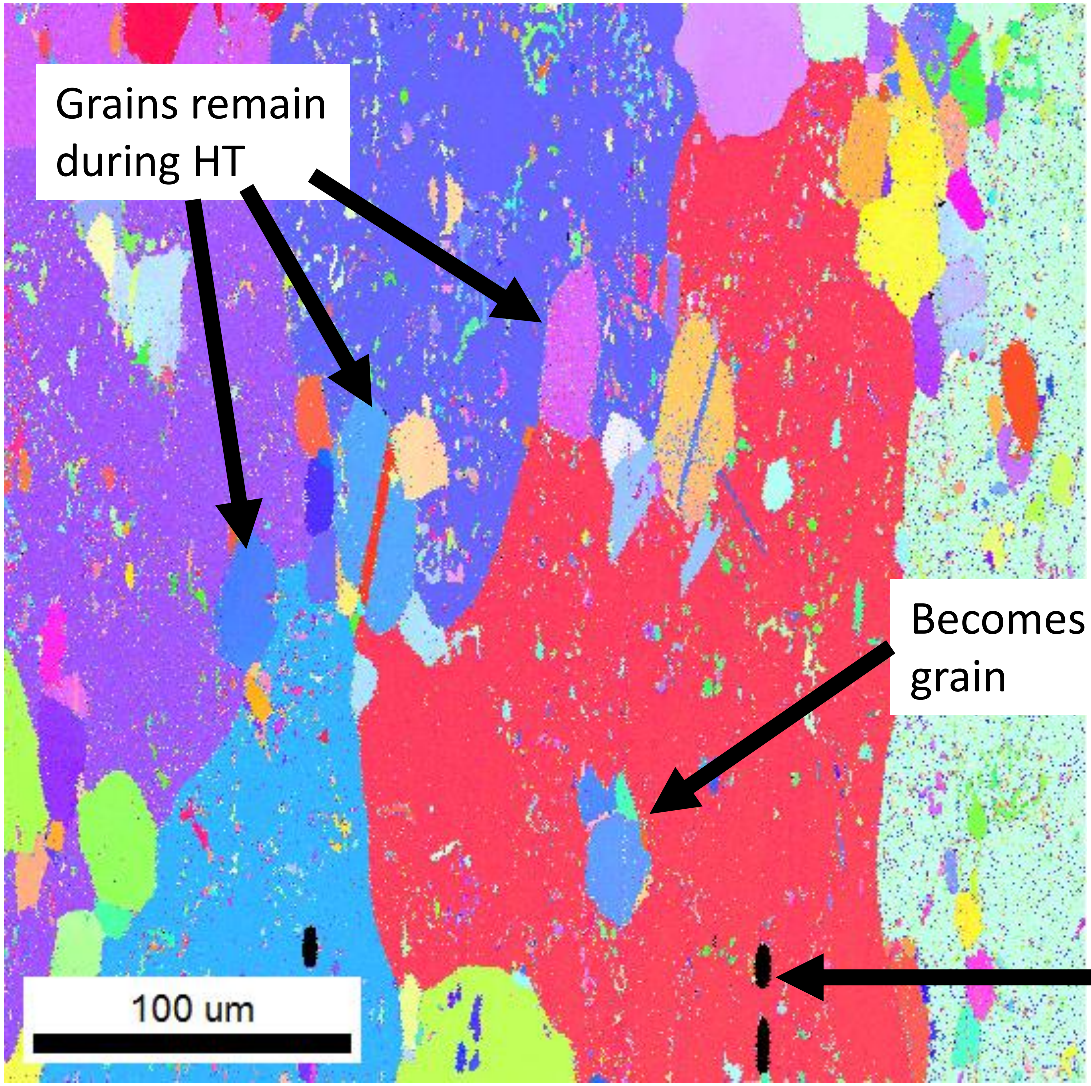
Interrupted Heat Treating

As forged (1120°C, 0.15 strain, 3E-4 strain/s)



Interrupted Heat Treating

Prior + 900°C @ 15°C/s, 1130°C @ 0.13°C/s

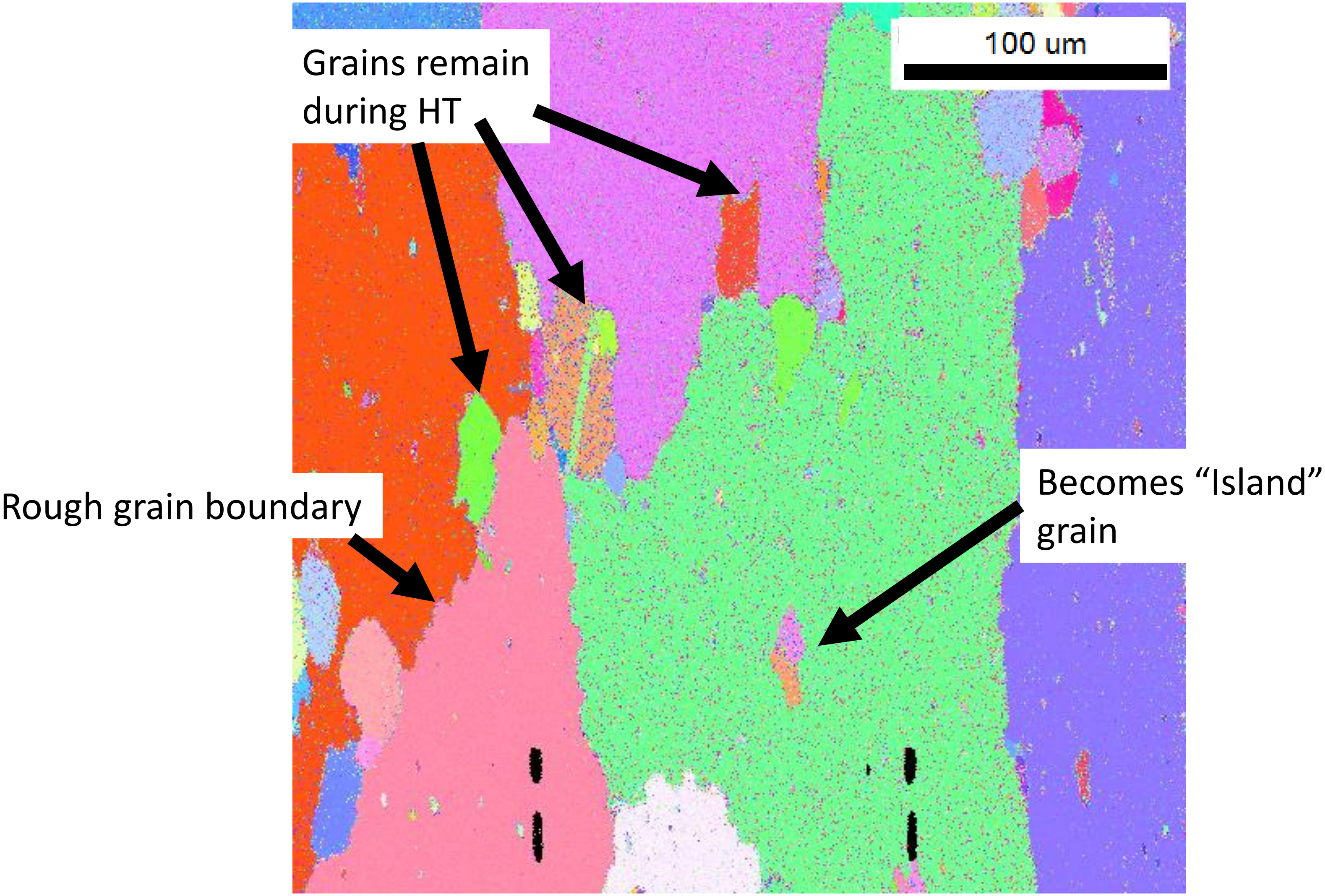


Oxide formation caused noise

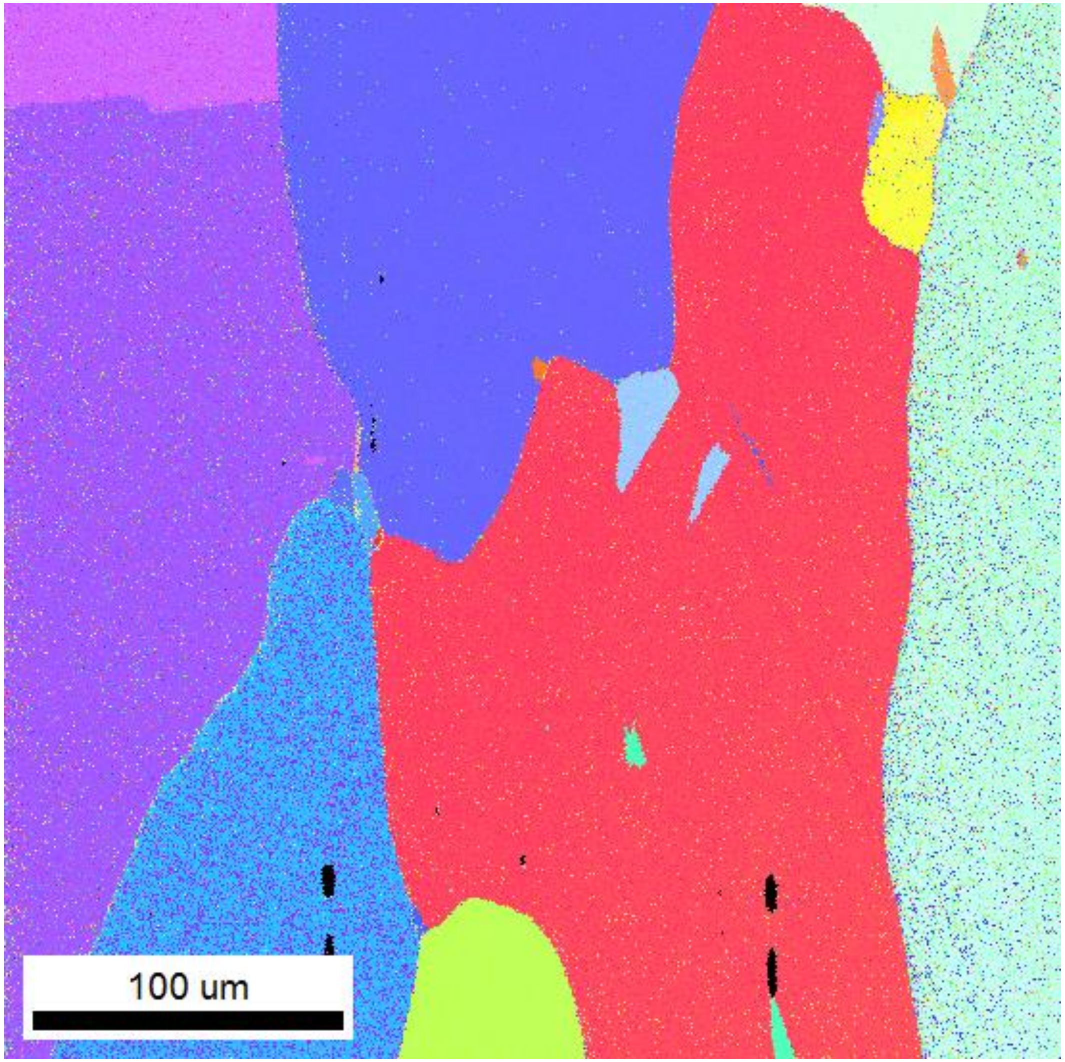
Fiducial marks

Interrupted Heat Treating

Prior + 1130°C @ 15°C/s, 1150°C @ 0.13°C/s



Interrupted Heat Treating
Prior + 1150°C @ 15°C/s, 1170°C @ 0.13°C/s



Interrupted Heat Treating Results

- AGG occurs below γ' solvus
- Interrupting heat treating does not affect AGG
  - Final microstructures comparable
- Unrecrystallized γ grains consumed by abnormal γ grains
- "Island" γ(?) grains appear to be swept over by abnormal γ grain boundary
  - Island grains also appear in specimens without free-surface effects
- Dissolving γ' appears to cause abnormal γ grain roughness
  - Possible curvature effects on boundary mobility/velocity

Quantifying γ'_1 Phase Fraction

- Fraction, size, and distribution of γ'_1 will be influential on HERX and AGG
  - Controls deformation mechanism
  - Provides HERX nuclei
- Quantify phase fraction as a function of time and temperature
  - Dilatometry
  - Temperatures: 1100, 1110, 1120, 1130, 1140°C
    - γ' solvus = 1135°C
  - Times: 2, 5, 30 minutes
- Preparation techniques evaluated
  - Oxalic electropolishing works best (Thanks Jonah Klemm-Toole)
- Software quantifying techniques in progress

Literature Comparison

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Proposed Mechanism:

- Hetero-epitaxial recrystallization (HERX) is a possible explanation
  - γ nucleates coherently off primary γ'_1 [1]
  - Low energy barrier for nucleation [2]
- Current & prior experimental results align with HERX mechanism

Role of Processing Parameters:

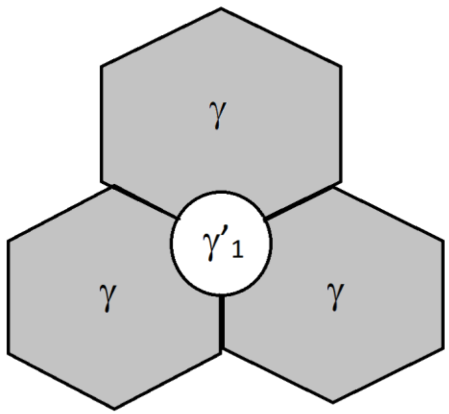
- Temperature
  - Determines γ'_1 phase fraction
- Strain
  - Degree of stored energy
- Strain rate
  - Deformation mechanism

[1] M.A. Charpagne, J.M. Franchet, N. Bozzolo. Mater. Des. 144 (2018) 353–360.

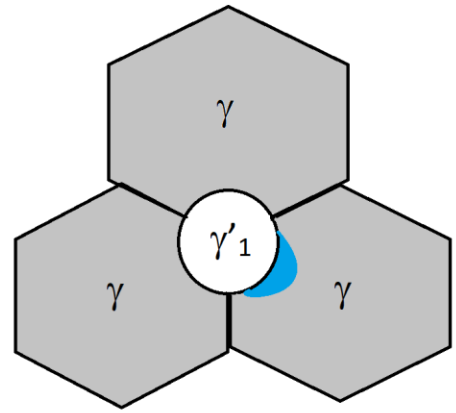
[2] V.M. Miller, A.E. Johnson, C.J. Torbet, T.M. Pollock. Met. Trans. A. 47, 4 (2016) 1566-1574.

HERX Theory

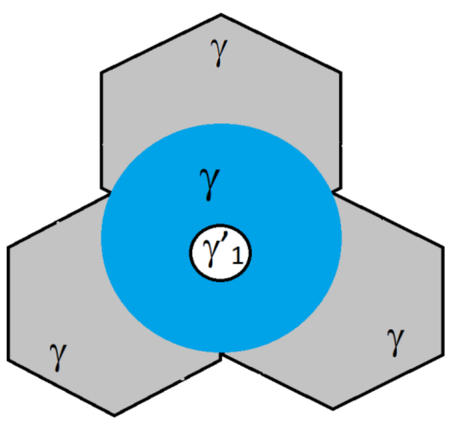
Adapted from Charpagne & Miller



γ'_1 is incoherent with neighboring γ



γ nucleates coherently off γ'_1



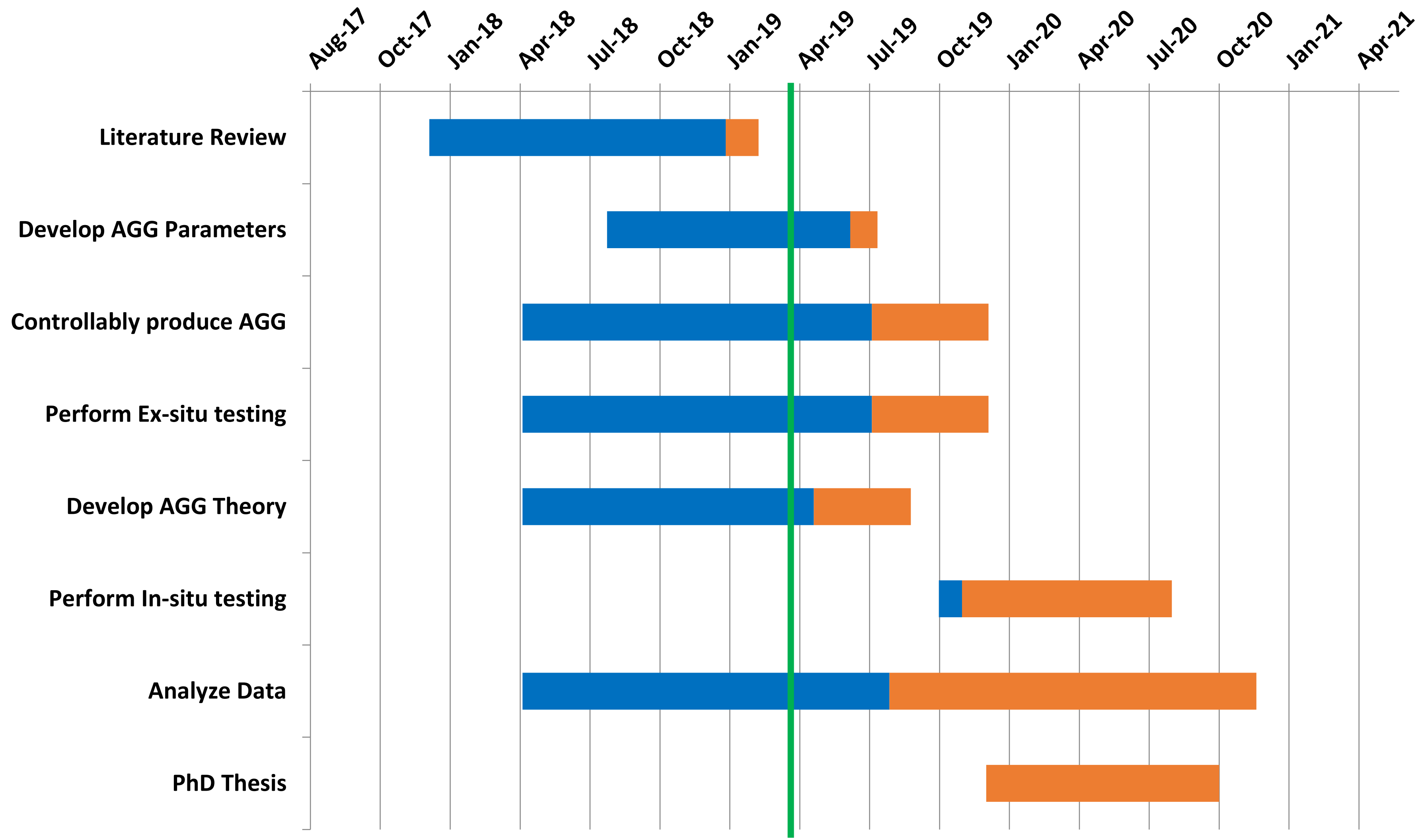
Growth to consume stored energy in γ

- 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

Future Work

- Quantify γ'_1 phase fraction
  - Important for AGG
  - Recreate with strained material
- Continue AGG observation
  - Interrupted heat treating
  - Perform at lower temperatures
  - Use EDX to find γ'_1
    - Local chromium depletion
- Planning in-situ observation of AGG
  - HEDM/HEXD in-situ heating
  - Cornell High Energy Synchrotron Source (CHESS)

Gantt Chart



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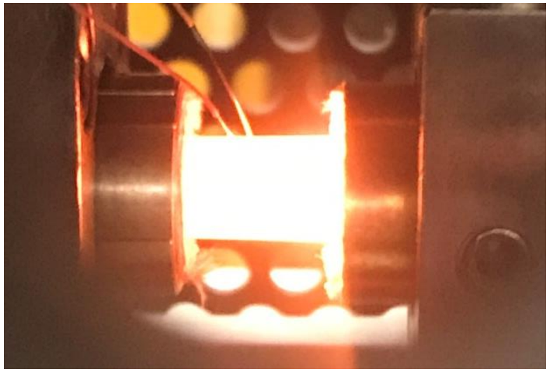
Jul-18
Oct-18
Jan-19
Apr-19
Jul-19
Oct-19
Jan-20
Apr-20
Jul-20
Oct-20
Jan-21
Apr-21
Literature Review
Develop AGG Parameters
Controllably produce AGG
Perform Ex-situ testing
Develop AGG Theory
Perform In-situ testing
Analyze Data
PhD Thesis

Thank you!

Byron McArthur
bmcarthu@mines.edu

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

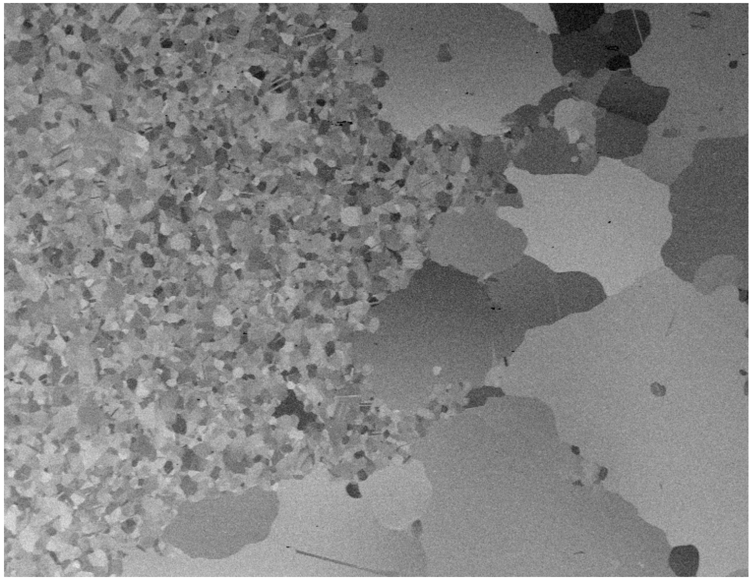


Student: *Byron McArthur*

Faculty: *Amy Clarke, Kester Clarke*

Industrial Partners: *AFRL (Eric Payton), ATI (Kevin Severs)*

Project Duration: *Nov. 2017 – Dec. 2020*



Achievement

- Determine the mechanism of abnormal grain growth in Ni-based superalloys using ex-situ and in-situ characterization techniques.

Significance and Impact

- Provide a mechanistic understanding of abnormal grain growth that will help improve reliability of turbine engine discs.

Research Details

- Ex- and in-situ characterization of material processed through isothermal forging and super solvus heat treatment.

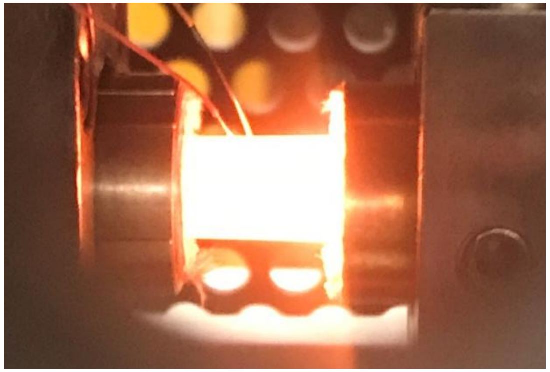
Project 34 – In-situ Observation of Phase and Texture Evolution Preceding Abnormal Grain Growth in Ni-based Aerospace Alloys

Student: *Byron McArthur*

Faculty: *Amy Clarke, Kester Clarke*

Industrial Partners: *AFRL, ATI*

Project Duration: *Nov. 2017 – Dec. 2020*



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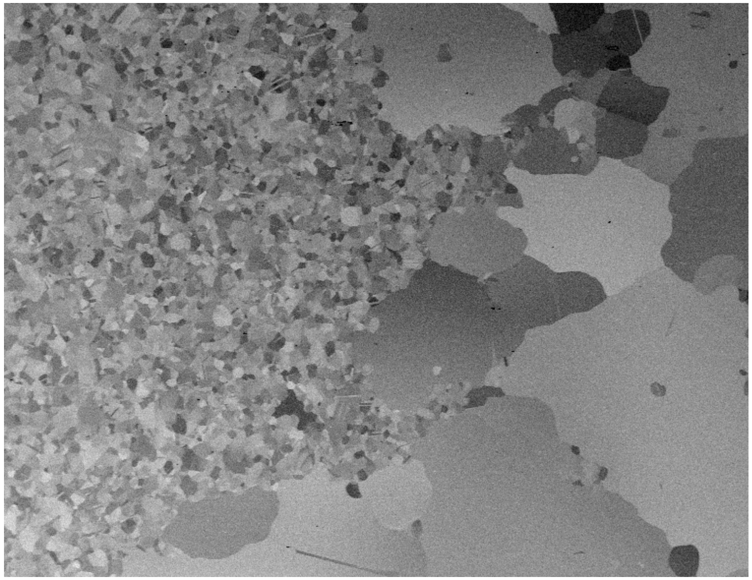


Project 34 – In-situ Observation of Phase and Texture Evolution Preceding Abnormal Grain Growth in Ni-based Aerospace Alloys

Student: *Byron McArthur*

Faculty: *Amy Clarke, Kester Clarke*

Industrial Partners: *AFRL, ATI*

Project Duration: *Nov. 2017 – Dec.*

Program Goal

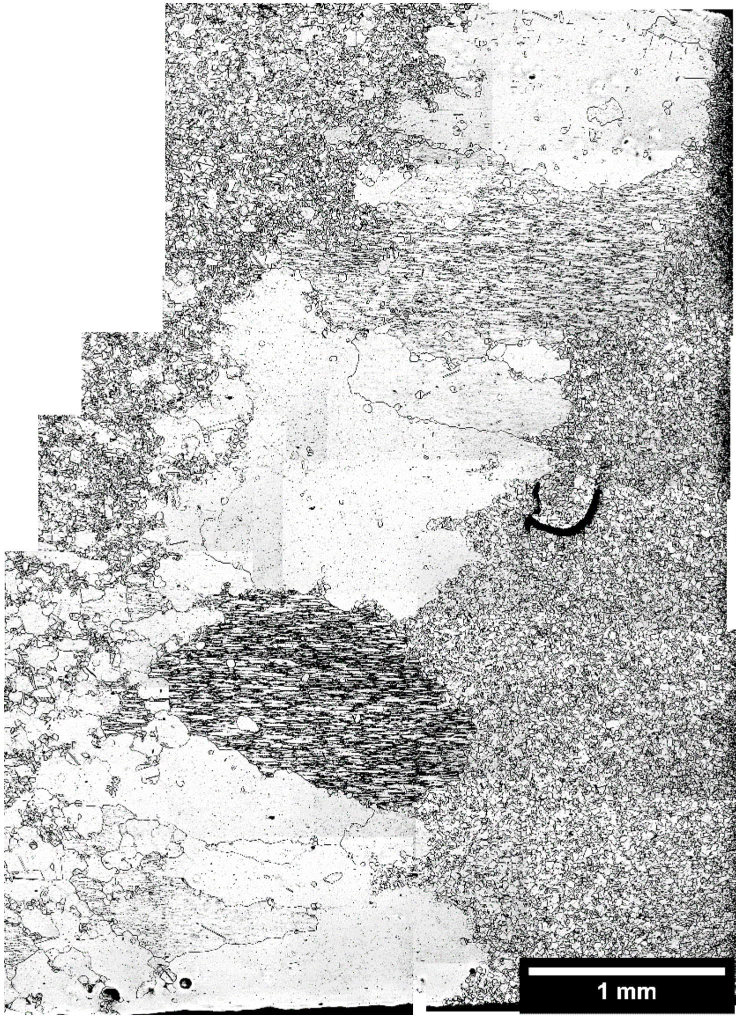
- Determine the microstructural mechanism precursors and process of abnormal grain growth in Ni-based superalloys.

Approach

- Utilize ex- and in-situ characterization techniques to determine the stored energy and texture dynamics due to isothermal forging and heat treatment.

Benefits

- Improved understanding of abnormal grain growth mechanisms and reliability of turbine engine discs



Abnormal grain growth in a Ni-based superalloy