INL/MIS-23-72834-Revision-0

ART Advanced Materials Program- Grain Coarsening Behavior of Alloy 709 Heats

June 2023

Grace Burke, Artur Ferreira, C.I. Garcia, Mauricio Viali, Yanli Wang, Pedro Ciacco, Marcos Mortiugui

Changing the World's Energy Future

INL is a U.S. Department of Energy National Laboratory operated by Battelle Energy Alliance, LLC

INL/MIS-23-72834-Revision-0

ART Advanced Materials Program- Grain Coarsening Behavior of Alloy 709 Heats

Grace Burke, Artur Ferreira, C.I. Garcia, Mauricio Viali, Yanli Wang, Pedro Ciacco, Marcos Mortiugui

June 2023

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

ART Advanced Materials Program

Grain Coarsening Behavior of Alloy 709 Heats

Grace Burke (INL), Yanli Wang (ORNL)

Marcos Moritugui, Mauricio Viali, Pedro Ciacco, Artur Ferreira and C. I. Garcia
(University of Pittsburgh)

June 6, 2023

Outline

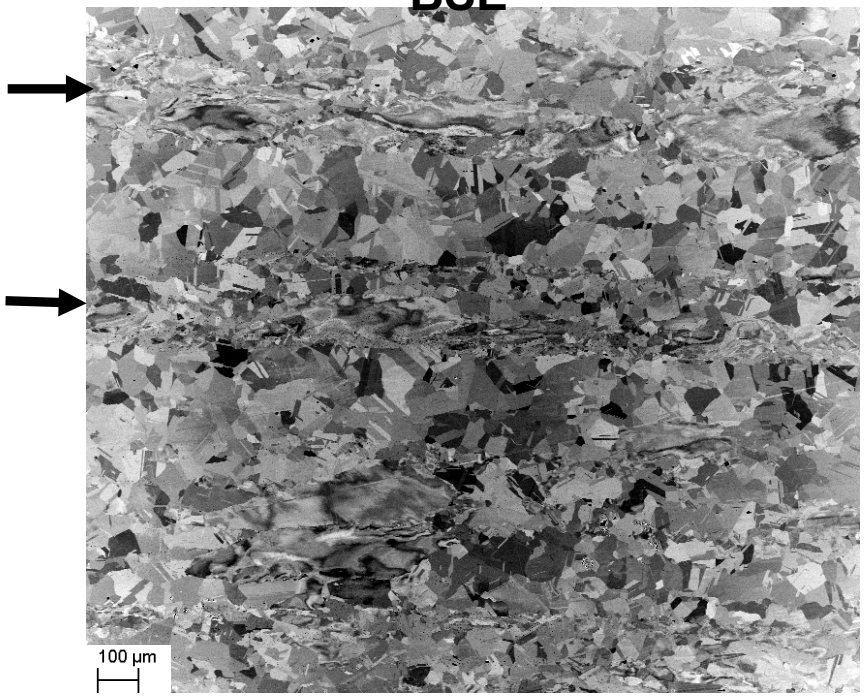
- Background – Grain Coarsening Temperature
- Material and Experimental Approach
- Results
- Summary
- Future Work

Importance of Grain Coarsening Temperature (T_{GC}) for TMP (1)

- As-cast alloys will have significant microsegregation associated with solute partitioning during solidification
  - Microsegregation of solutes
  - Formation of inclusions in the melt and during cooling (submicron to several microns in size)
- **Homogenization coupled with hot-working/hot-rolling required to break down the as-cast structure.**
- Understanding the grain coarsening behavior is critical to ensuring that all precipitates are dissolved so that a **uniform final microstructure** is obtained in the **as-hot-worked condition**. This is important for **both as-rolled as well as as-forged product forms**.

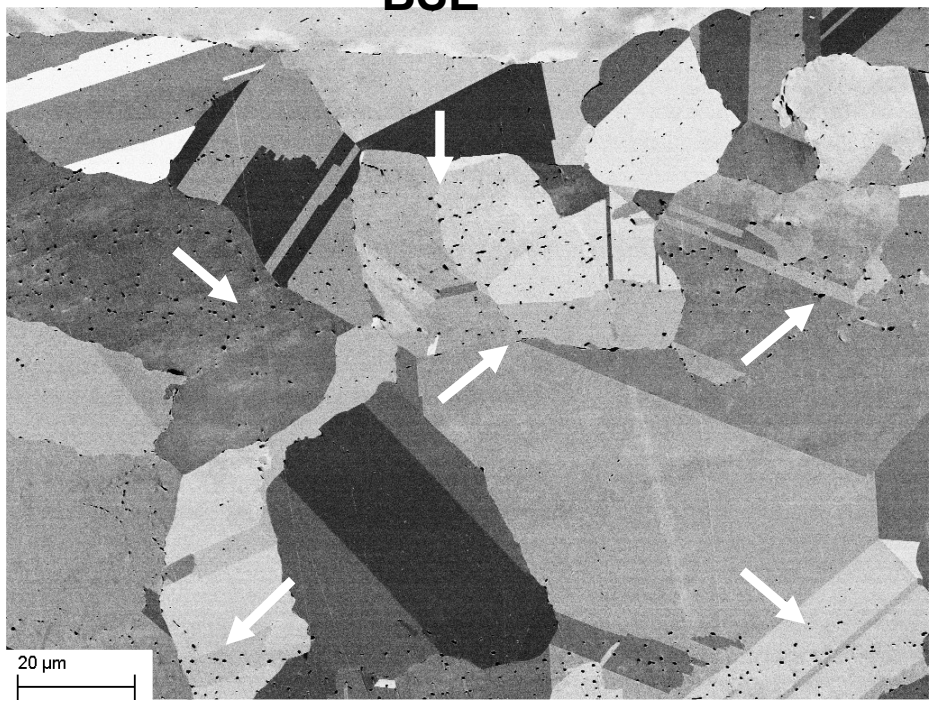
EXAMPLES: Microstructures resulting from inadequate solution-annealing followed by non-optimized hot-rolling

BSE

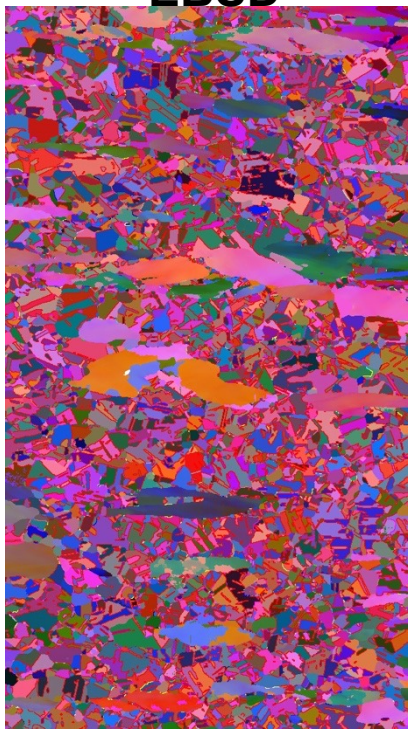


Rolling direction ⟷

BSE



EBSD



- Note the significant variation in grain size within the alloy
- Note the presence of “Ghost Boundaries”(white arrows) that are visible due to the **undissolved carbides** formed earlier during initial TMP

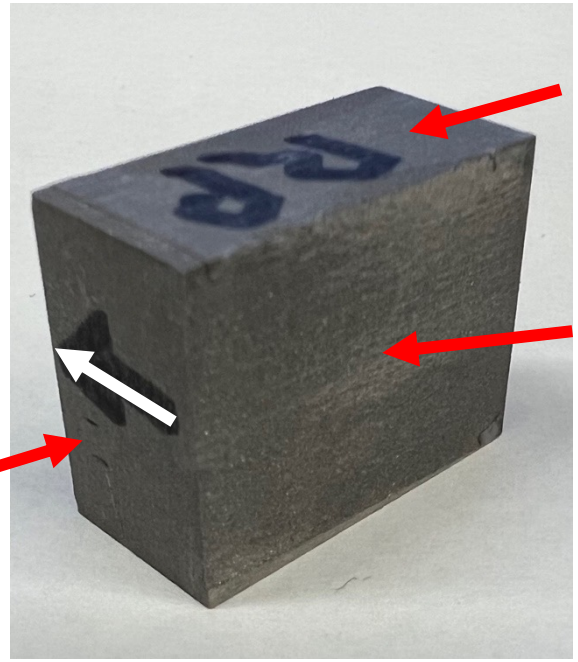
First Step in Determining Appropriate TMP Parameters for Alloy 709

T_{GC} provides the 'starting point' for TMP parameter development

- Uniform microstructure for determination of appropriate TMP processing "space": ε, $\dot{\varepsilon}$, and % recrystallized as f(T_{deform})
- Measurement of elevated T σ and ε for forging and hot-rolling
- Needed for appropriate **solution anneal temperature** for use in experimental **Continuous Cooling Precipitation diagram** generation

Alloy 709 Composition (wt.%)

Sample ID	Heat	C	Mn	P	S	Si	Cr	Ni	Al	Mo	Cu	Nb	Ti	N	Co	B	Fe
Carlson	58776-3R-BC1	0.066	0.9	0.014	0.001	0.38	20.05	25.14	0.02	1.51	0.06	0.26	0.01	0.152	0.02	0.003	Bal.
ATI	529900-02	0.08	0.9	0.004	< 0.001	0.35	20.0	24.6	0.01	1.5	0.07	0.17	< 0.01	0.16	0.02	0.005	Bal.



ROLLING PLANE

TRANSVERSE PLANE NORMAL TO ROLLING DIRECTION

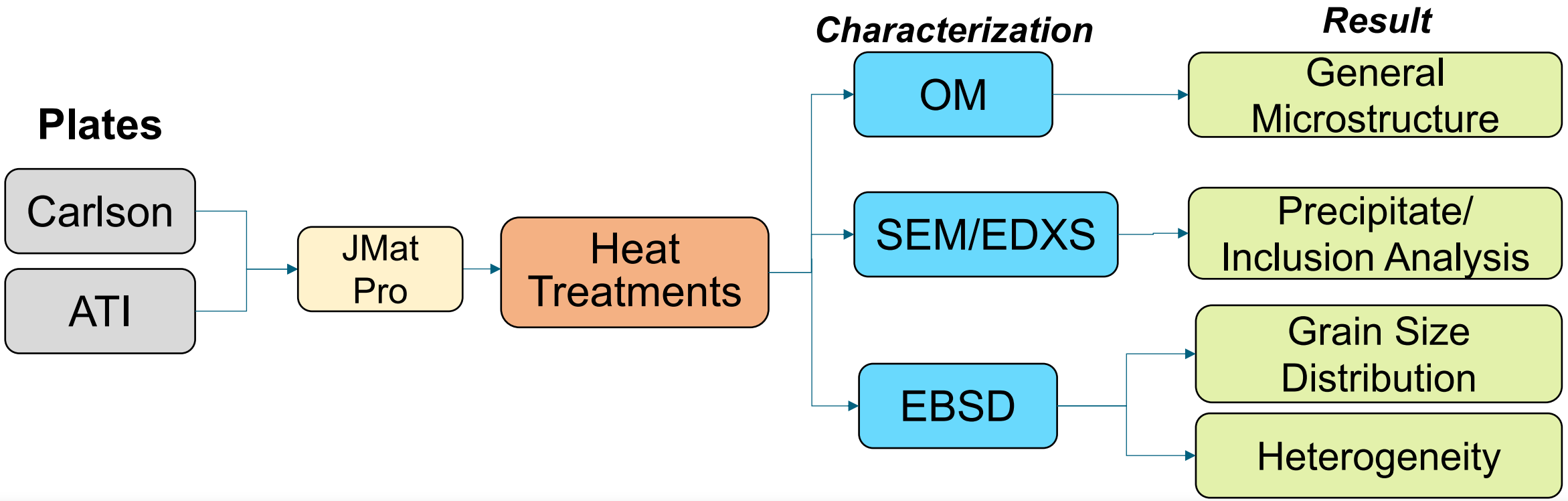
TRANSVERSE PLANE CONTAINING ROLLING DIRECTION

As-Rolled A709: 2nd Commercial Heat from ATI

Experimental Procedure

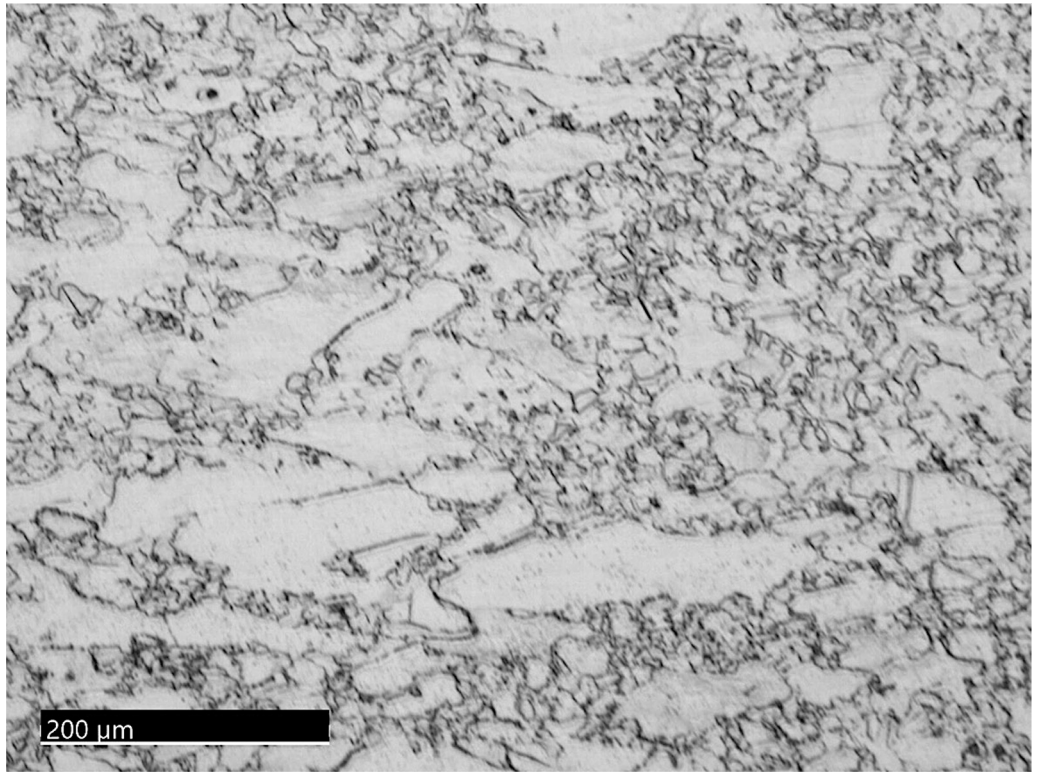
Objectives:

- Assess grain coarsening behavior of two A709 commercial as-rolled plates from Carlson and ATI
- Experimentally define the grain coarsening temperature, T_{GC}, to dissolve carbide/carbonitride precipitates in the as-hot-rolled plates

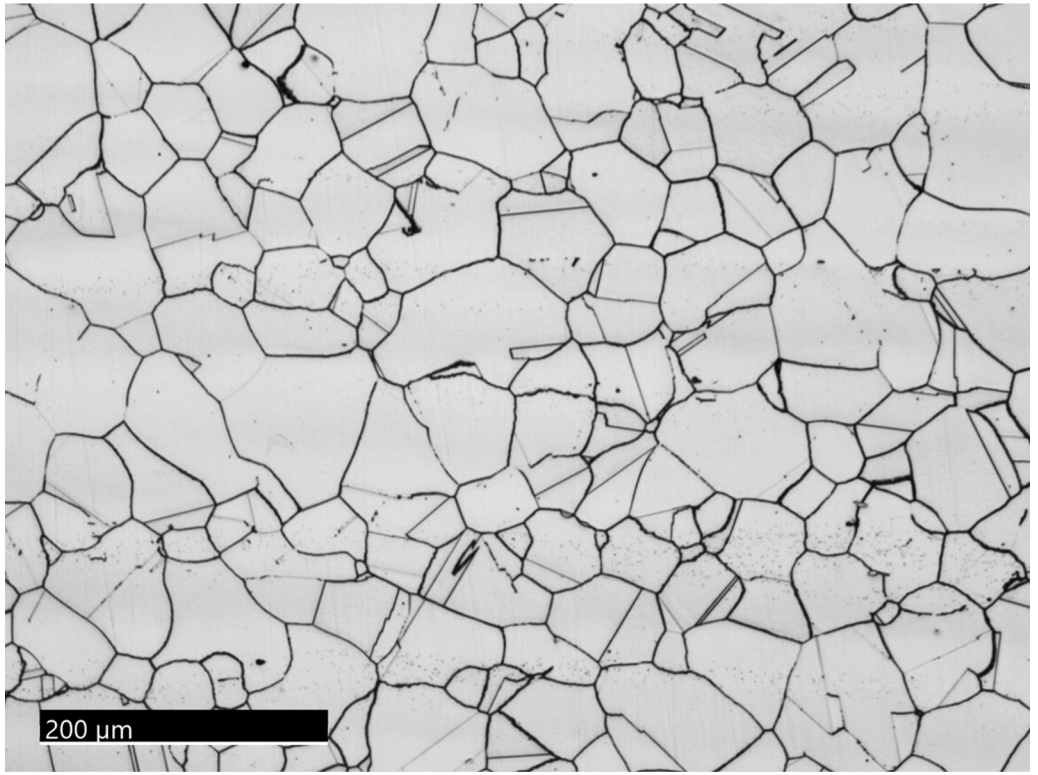


Microstructure of the As-Received (As-Rolled) Alloy 709 Plates

Carlson



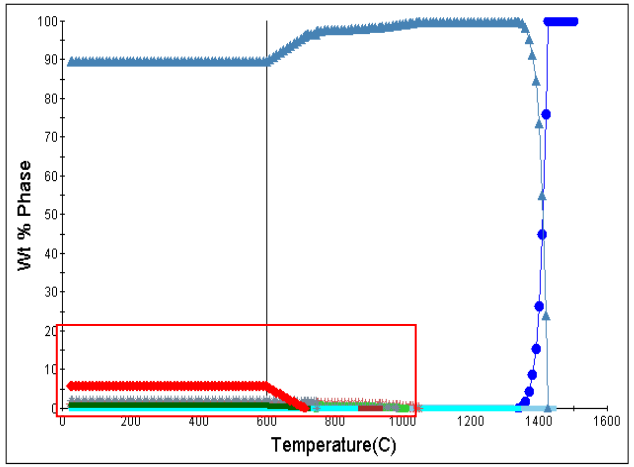
ATI

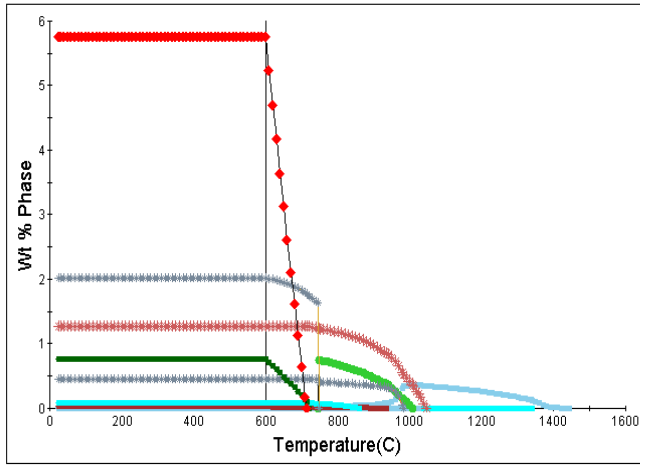


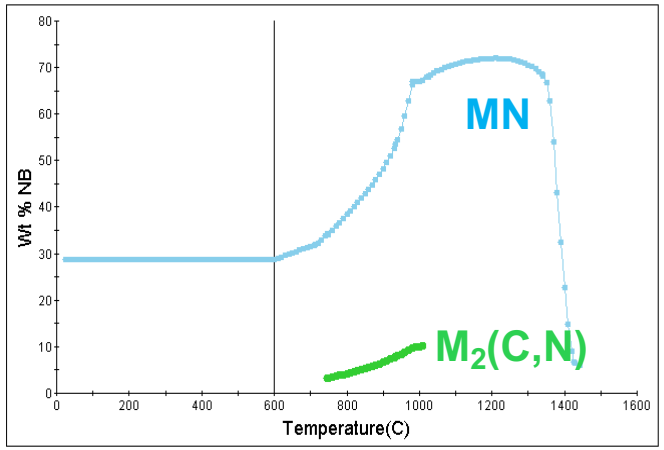
- **Carlson:** Note the inhomogeneous, partially-recrystallized grain structure: Coarse elongated grains "decorated" with ultrafine recrystallized grains ("Necklace" structure)
- **ATI:** Relatively uniform, equiaxed recrystallized grain structure

JMatPro – Thermodynamic Analysis

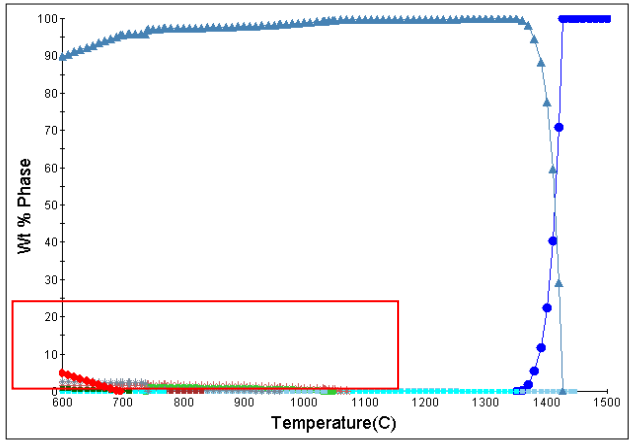
Carlson Heat

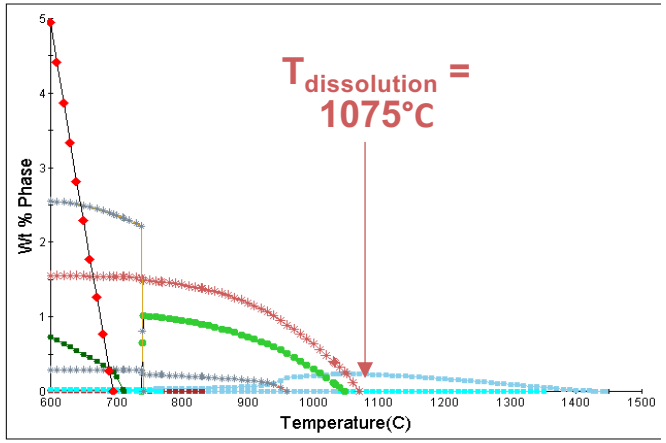


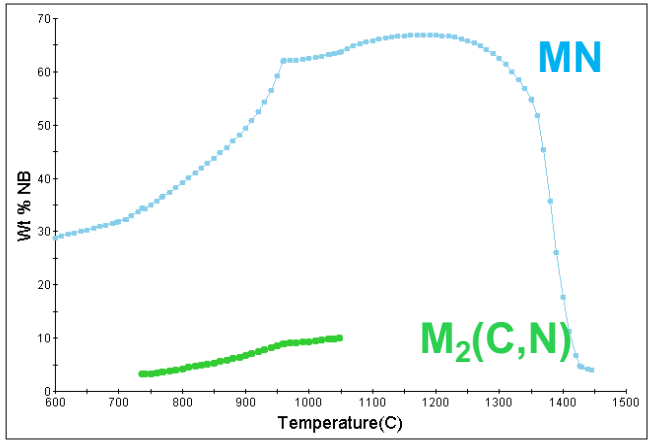




ATI Heat







Proposed Heat Treatments

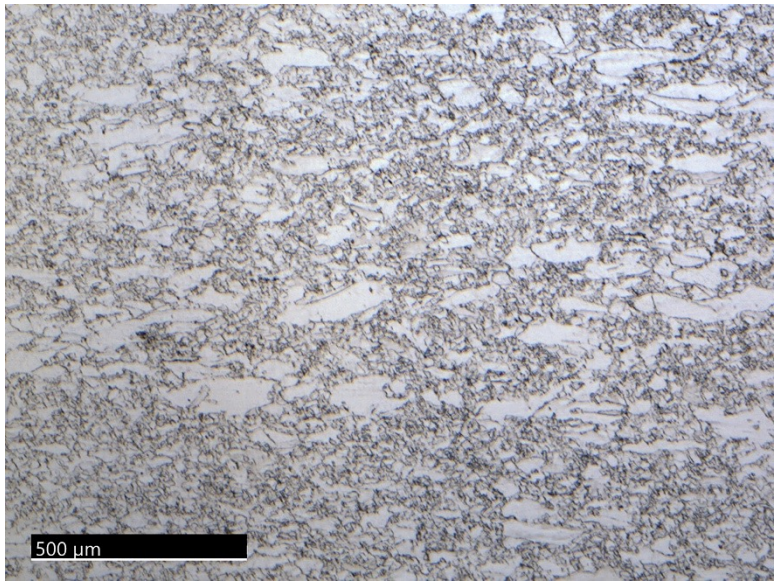
Heat 58776 - 3RB (Carlson heat)	----	1000°C, 1h	1050°C, 1h	1100°C, 1h	1150°C, 1h	1200°C, 1h	1250°C, 1h	1250°C, 3h
Heat 529900 -02 (ATI heat)	950°C, 1h	1000°C, 1h	1050°C, 1h	1100°C, 1h	1150°C, 1h	1200°C, 1h	---	---

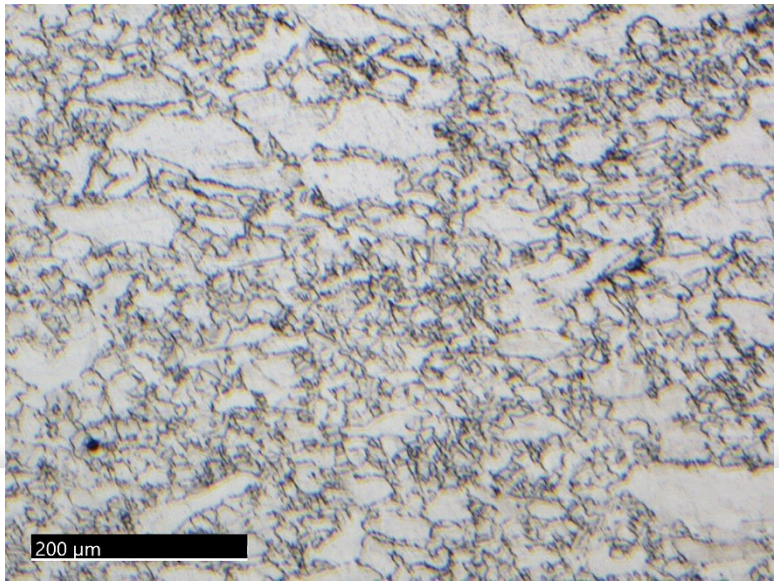
RESULTS:

Light Optical Metallography and SEM Evaluations

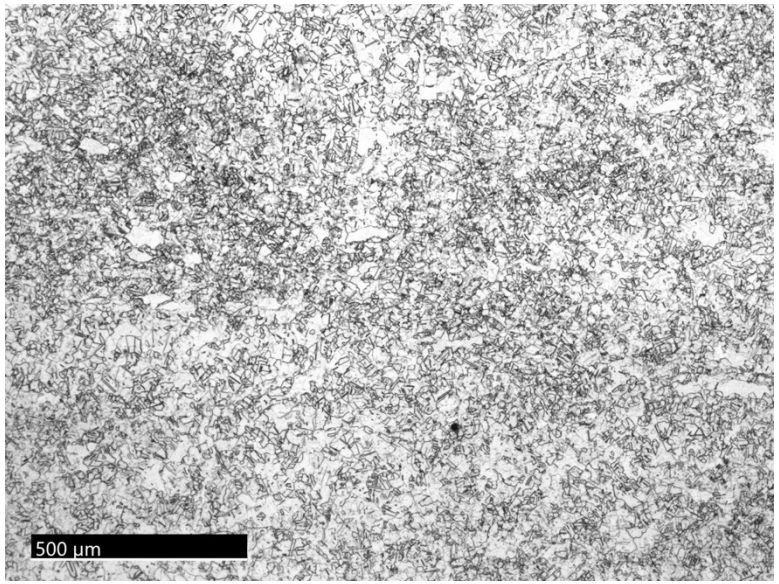
CARLSON:

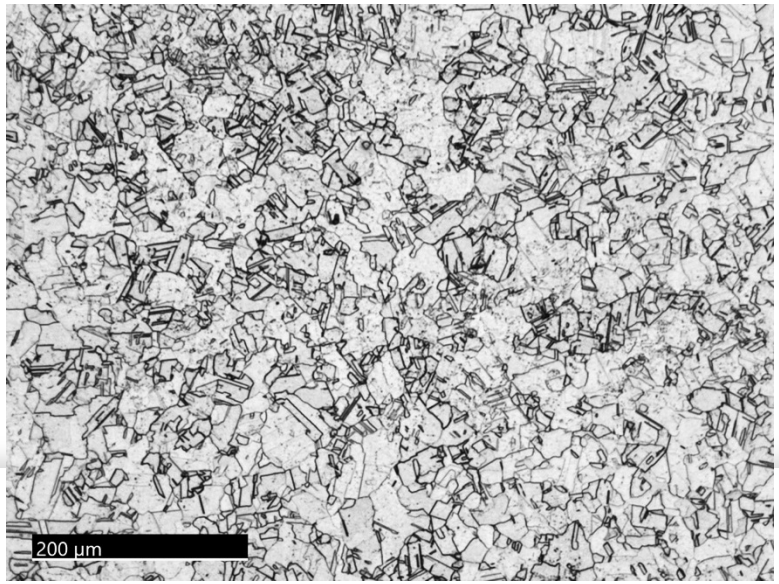
As-received (AR)



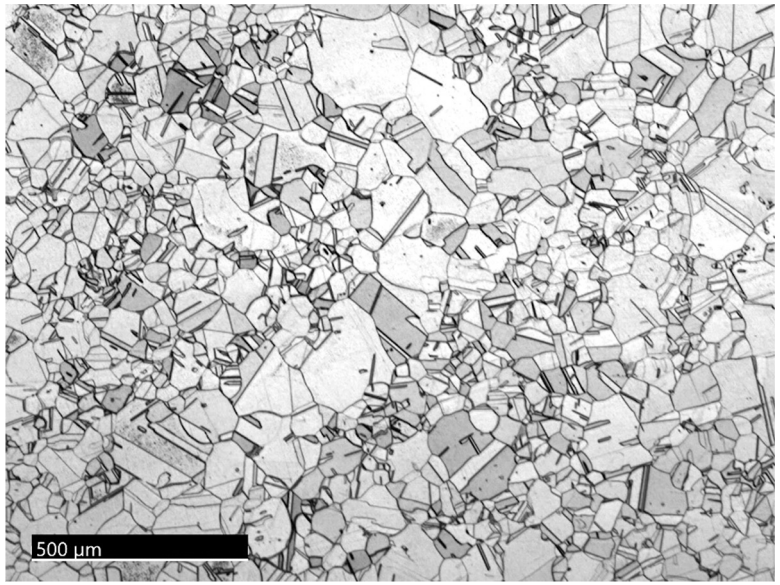


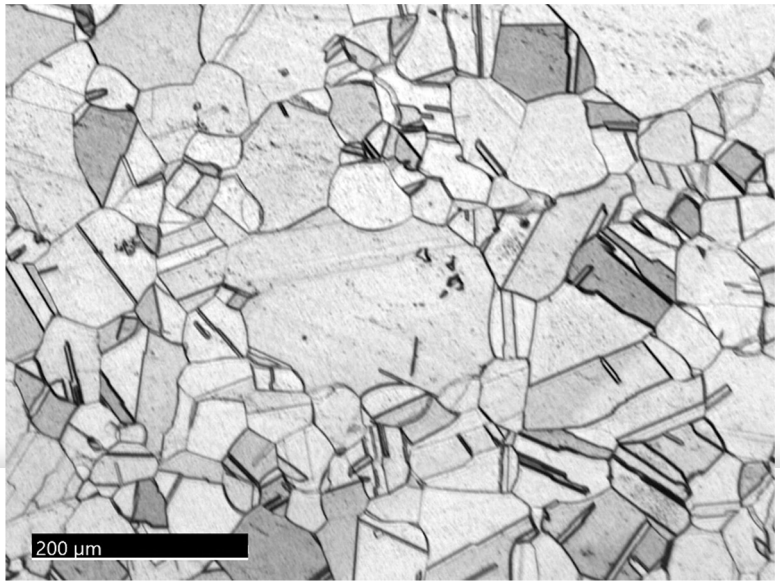
1000°C (1h)





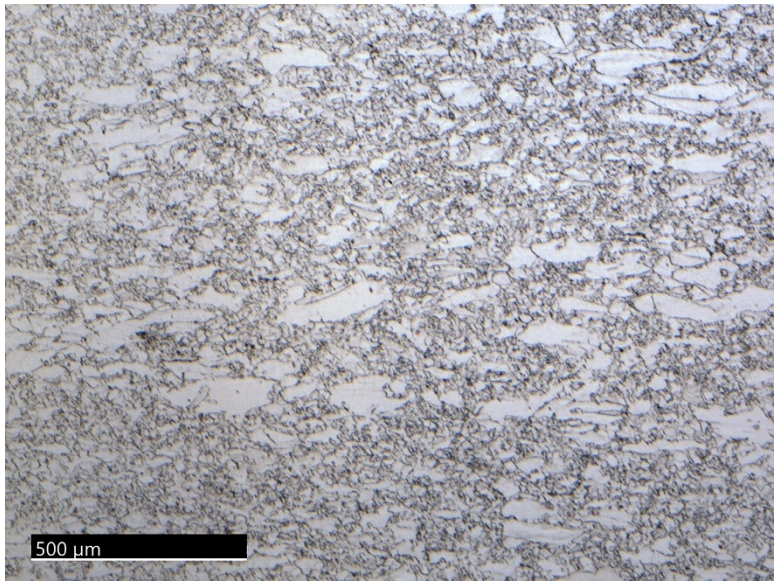
1150°C (1h)

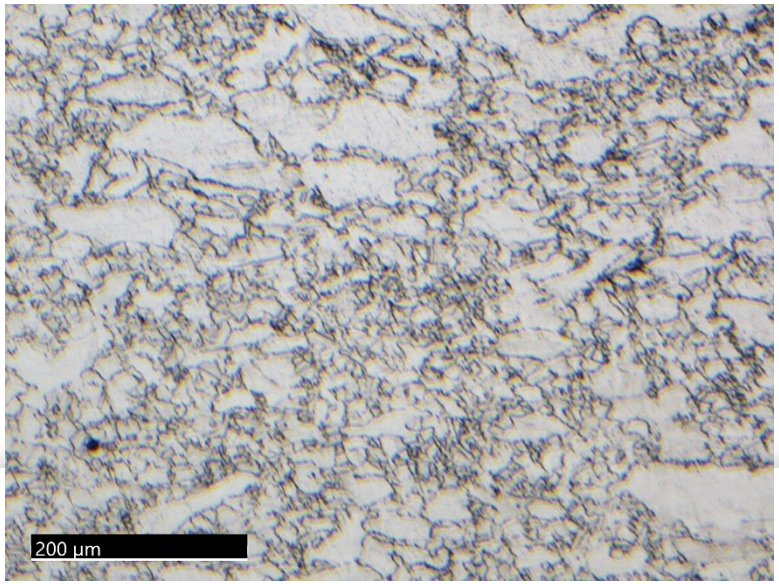




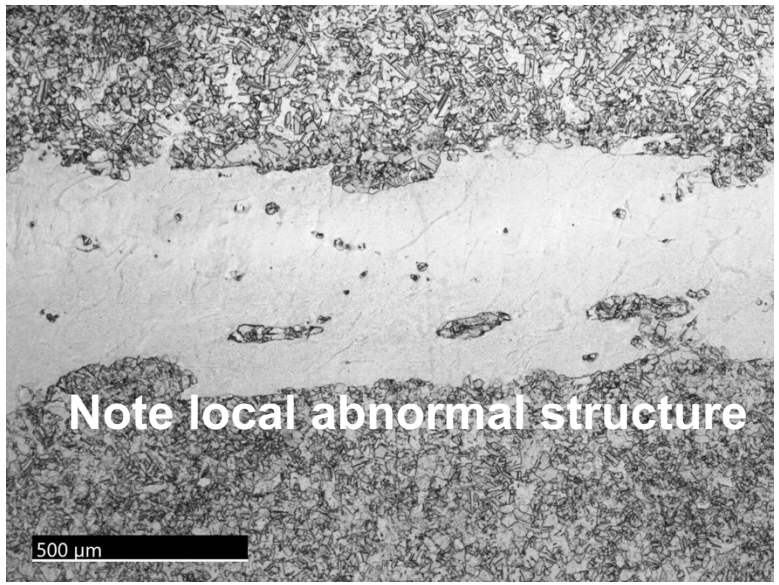
CARLSON:

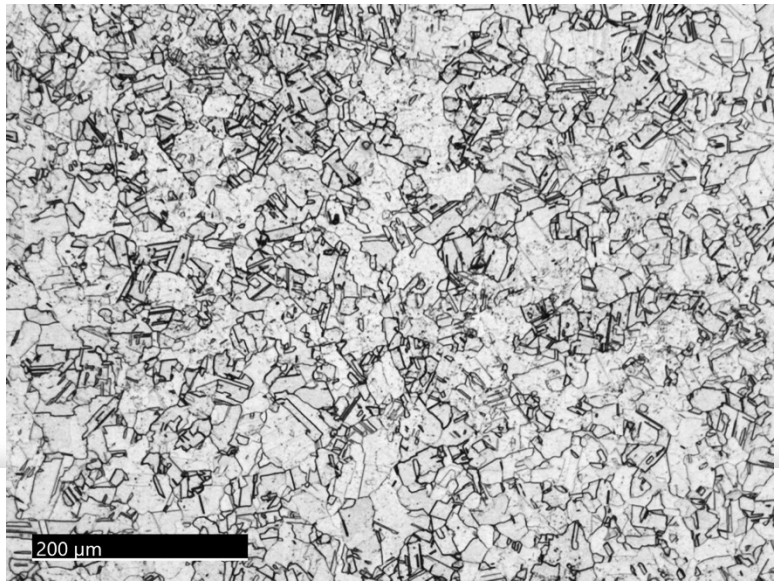
As-received (AR)



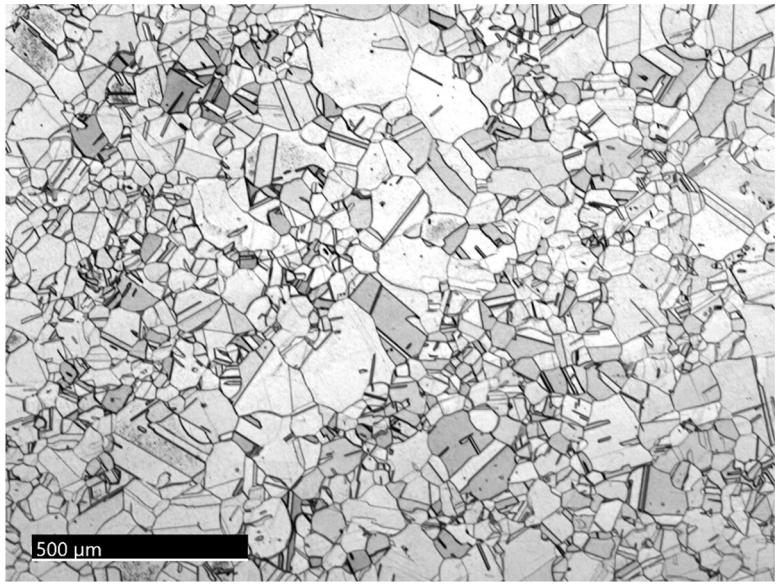


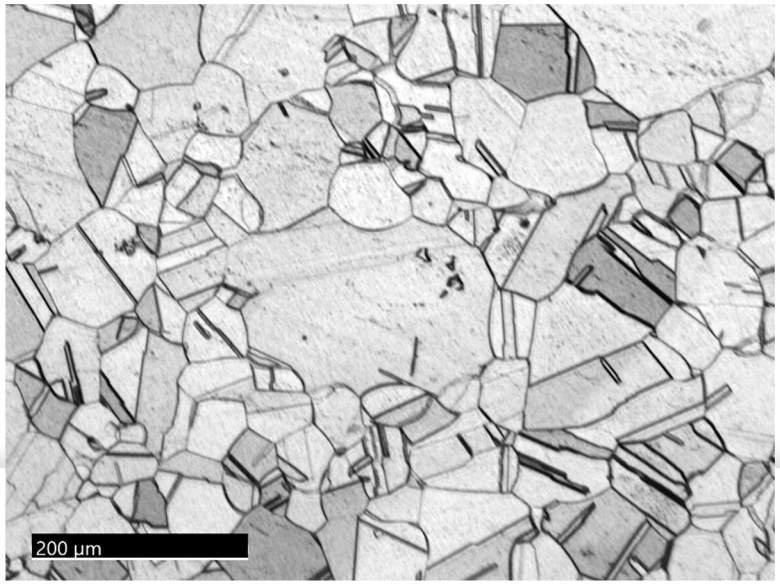
1000°C (1h)





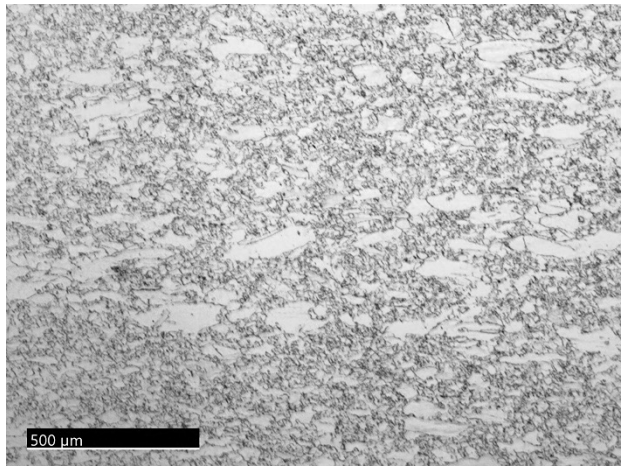
1150°C (1h)



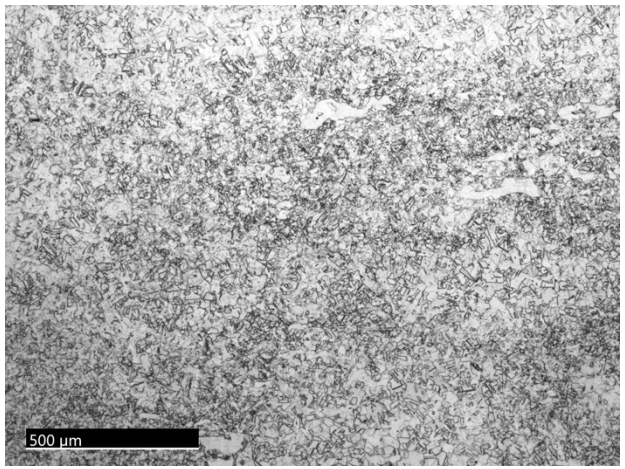


Comparison of Grain Coarsening Treatments – Carlson Heat

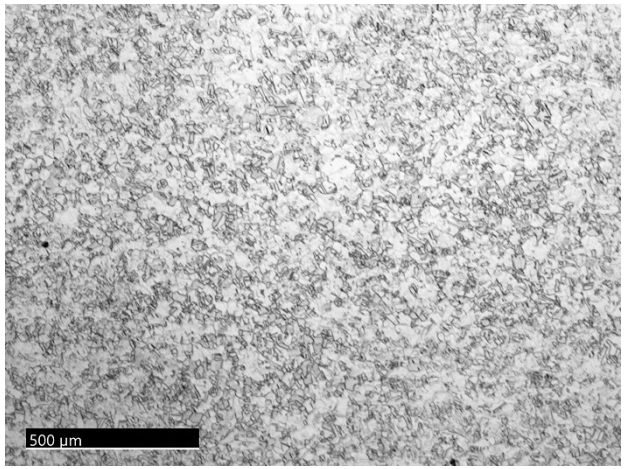
AR



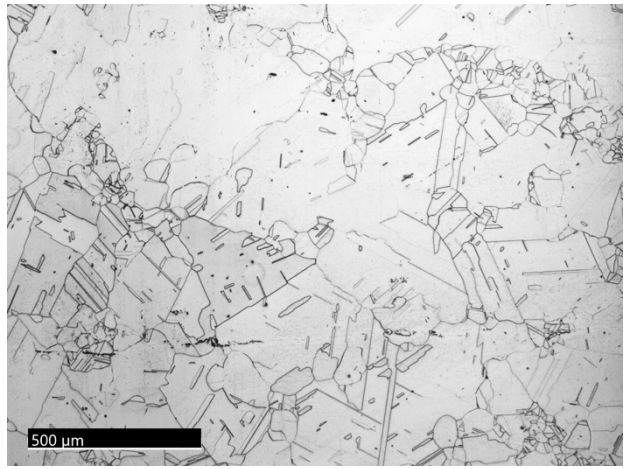
1000°C – 1h



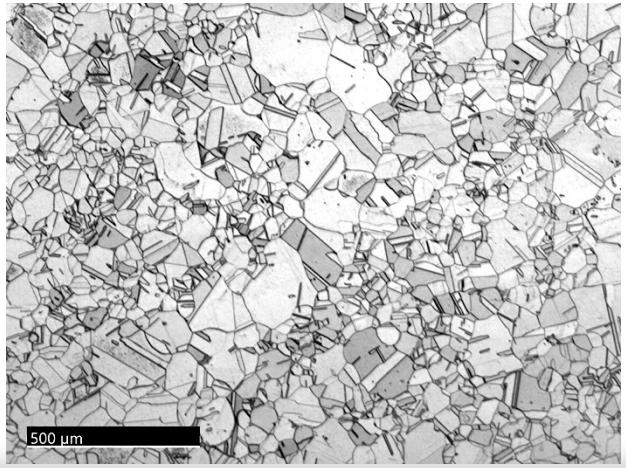
1050°C – 1h



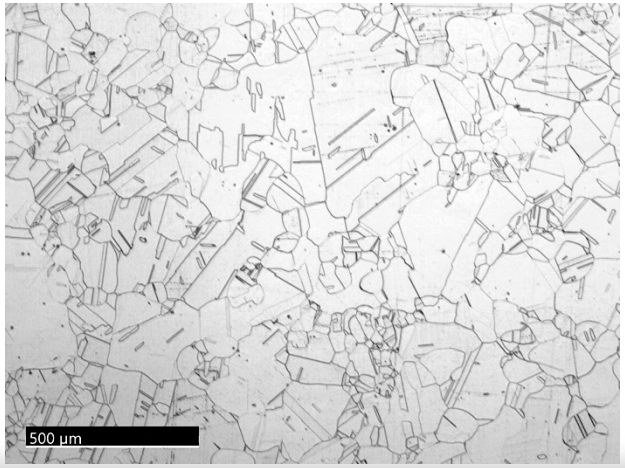
1100°C – 1h



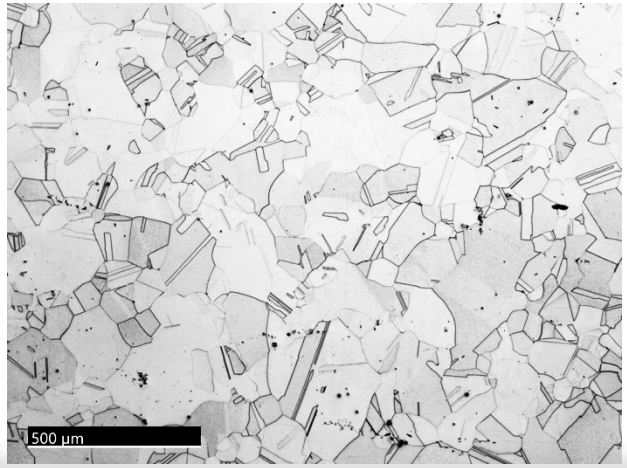
1150°C – 1h



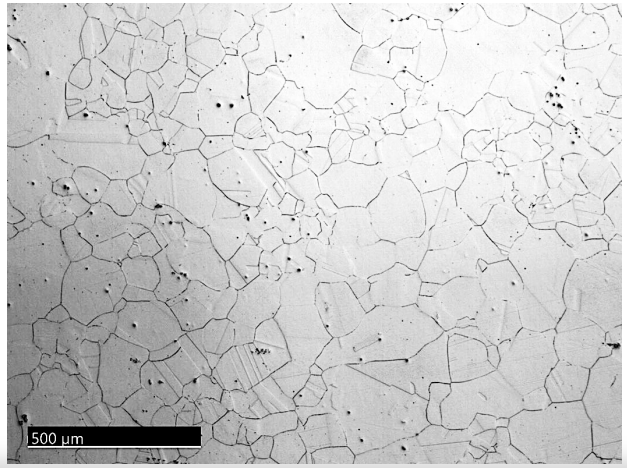
1200°C – 1h



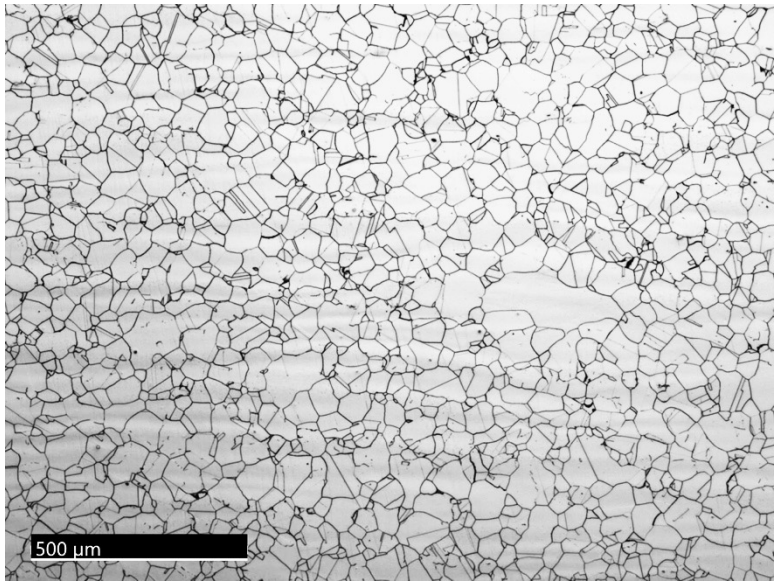
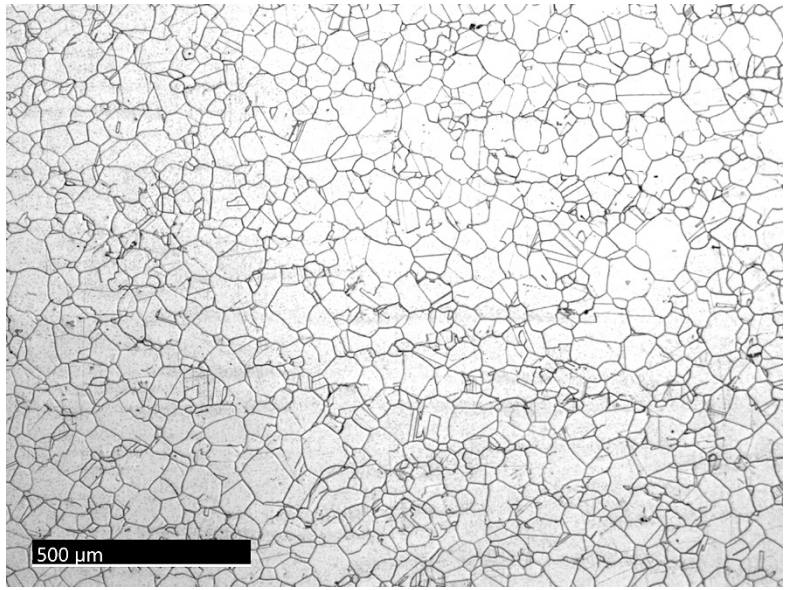
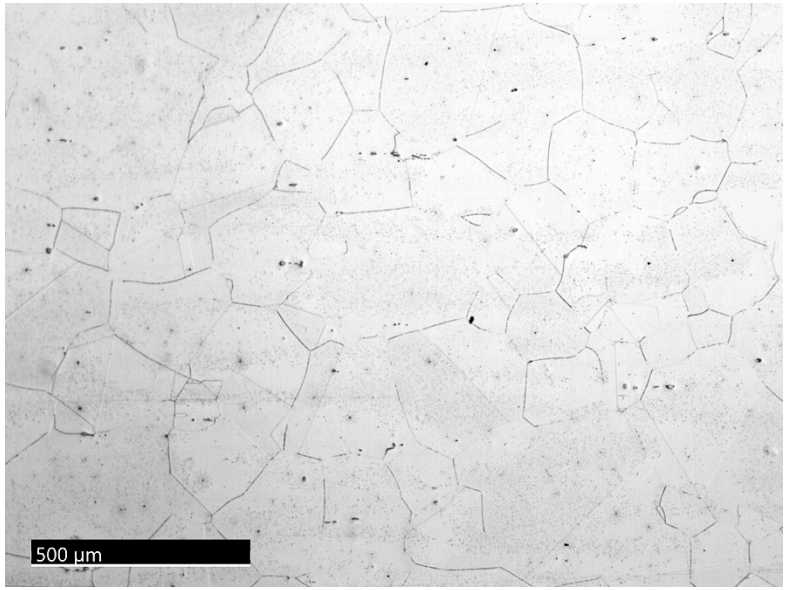
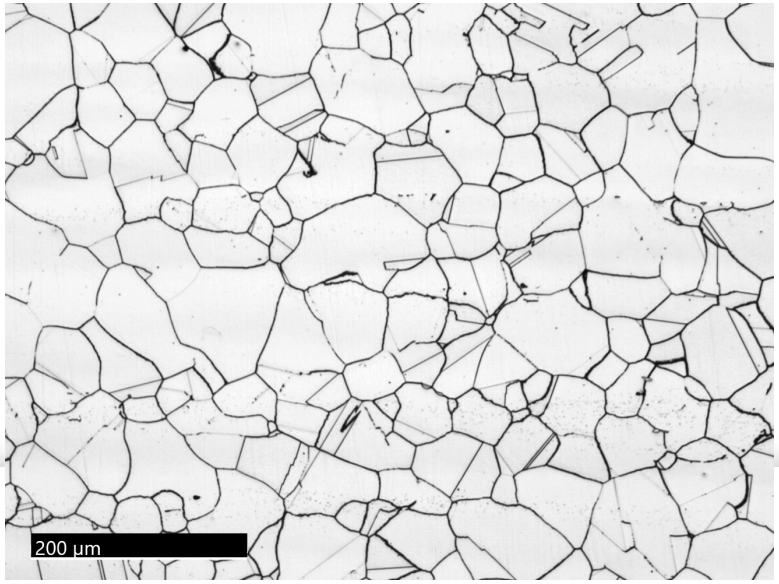
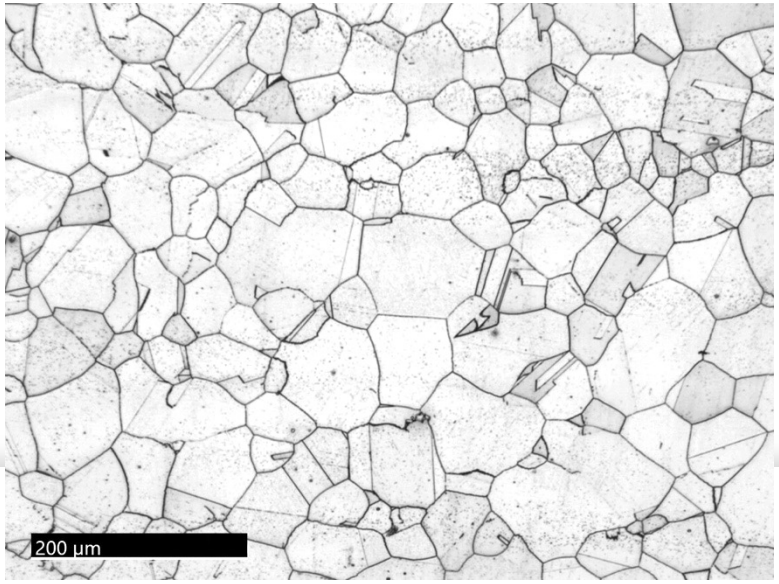
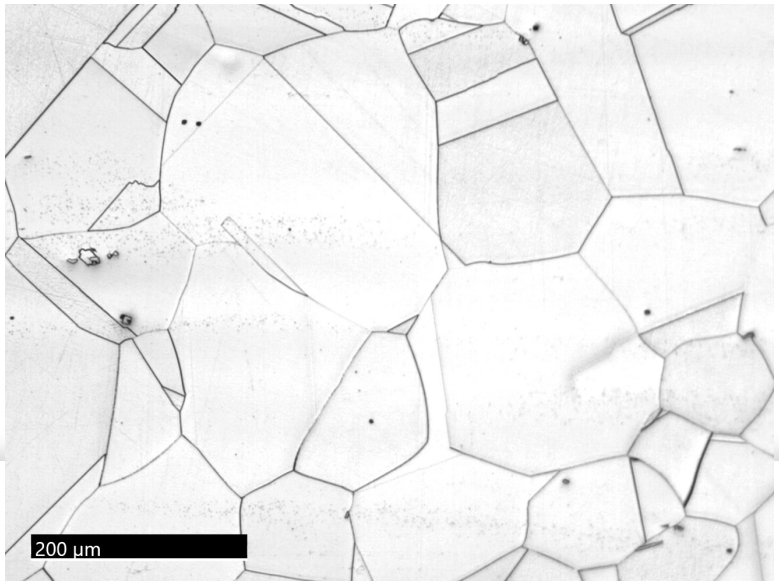
1250°C – 1h



1250°C – 3h

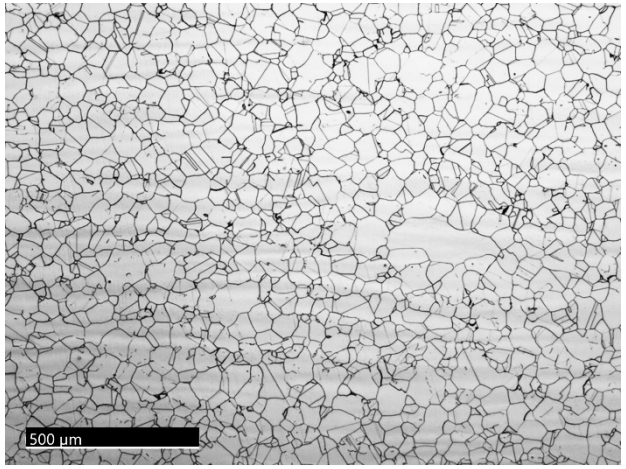


ATI

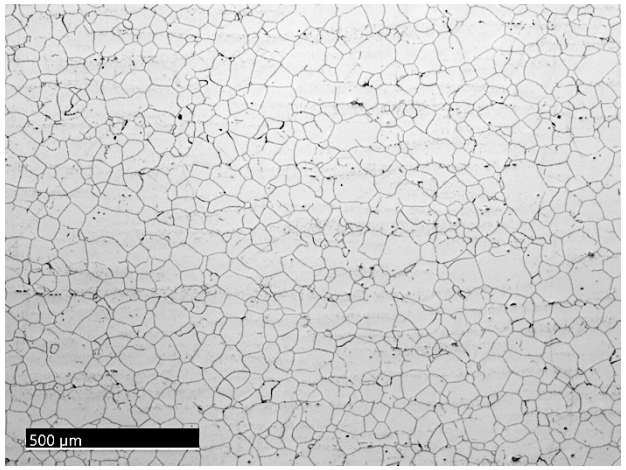
As-Received	1000°C (1h)	1200°C (1h)
		
		

Comparison of Grain Coarsening Treatments – ATI Heat

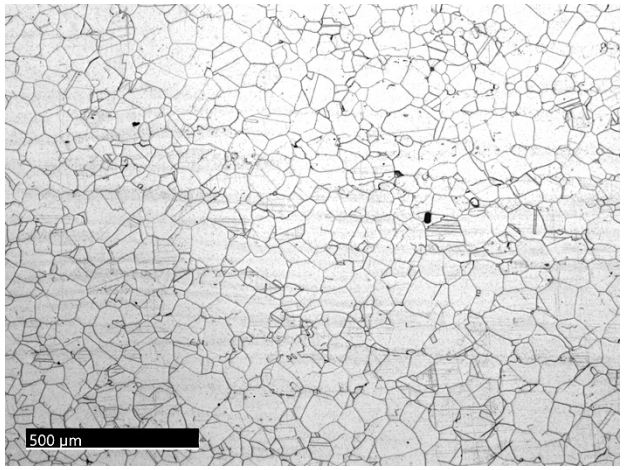
As-Received



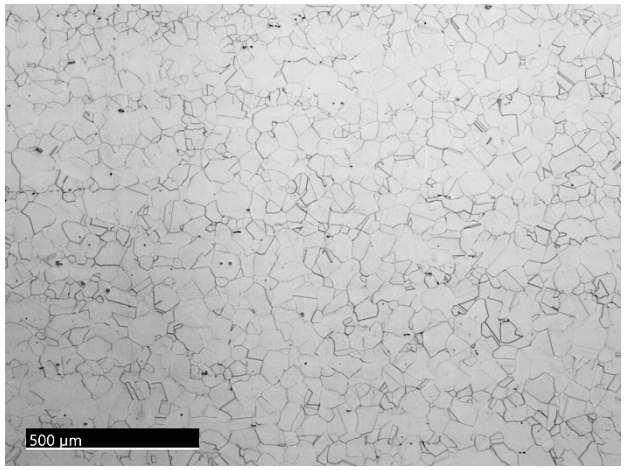
950°C – 1h



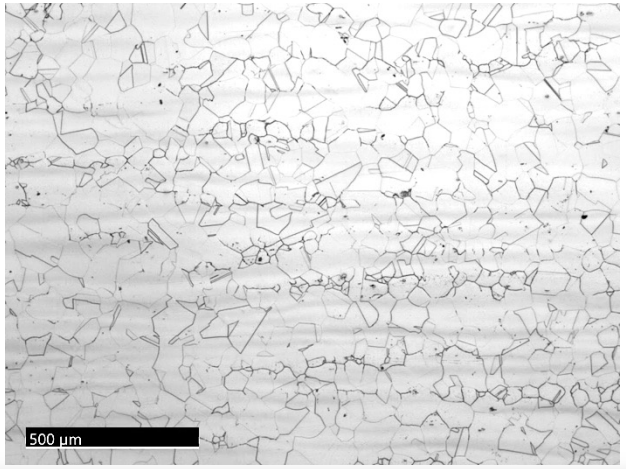
1000°C – 1h



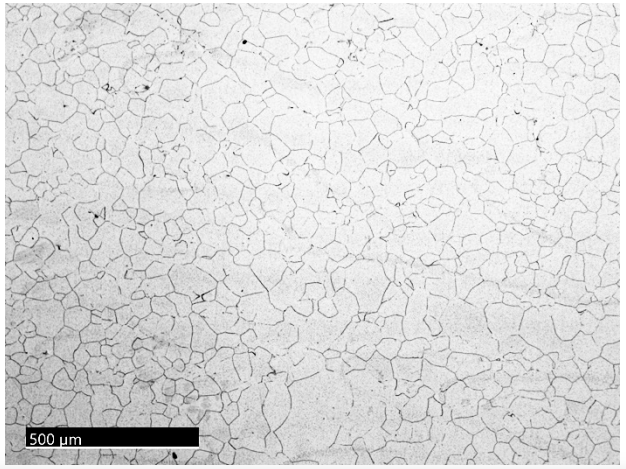
1050°C – 1h



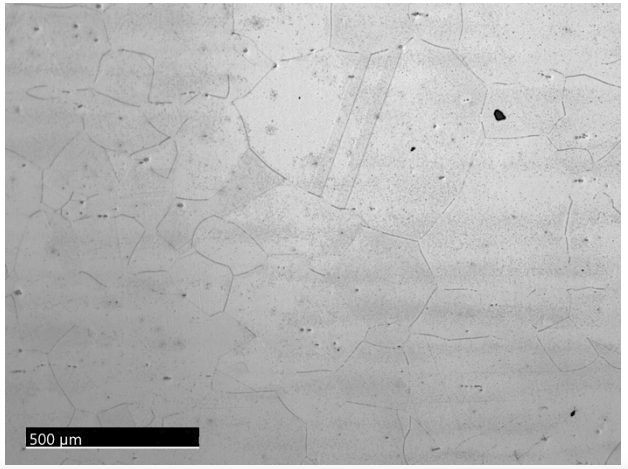
1100°C – 1h



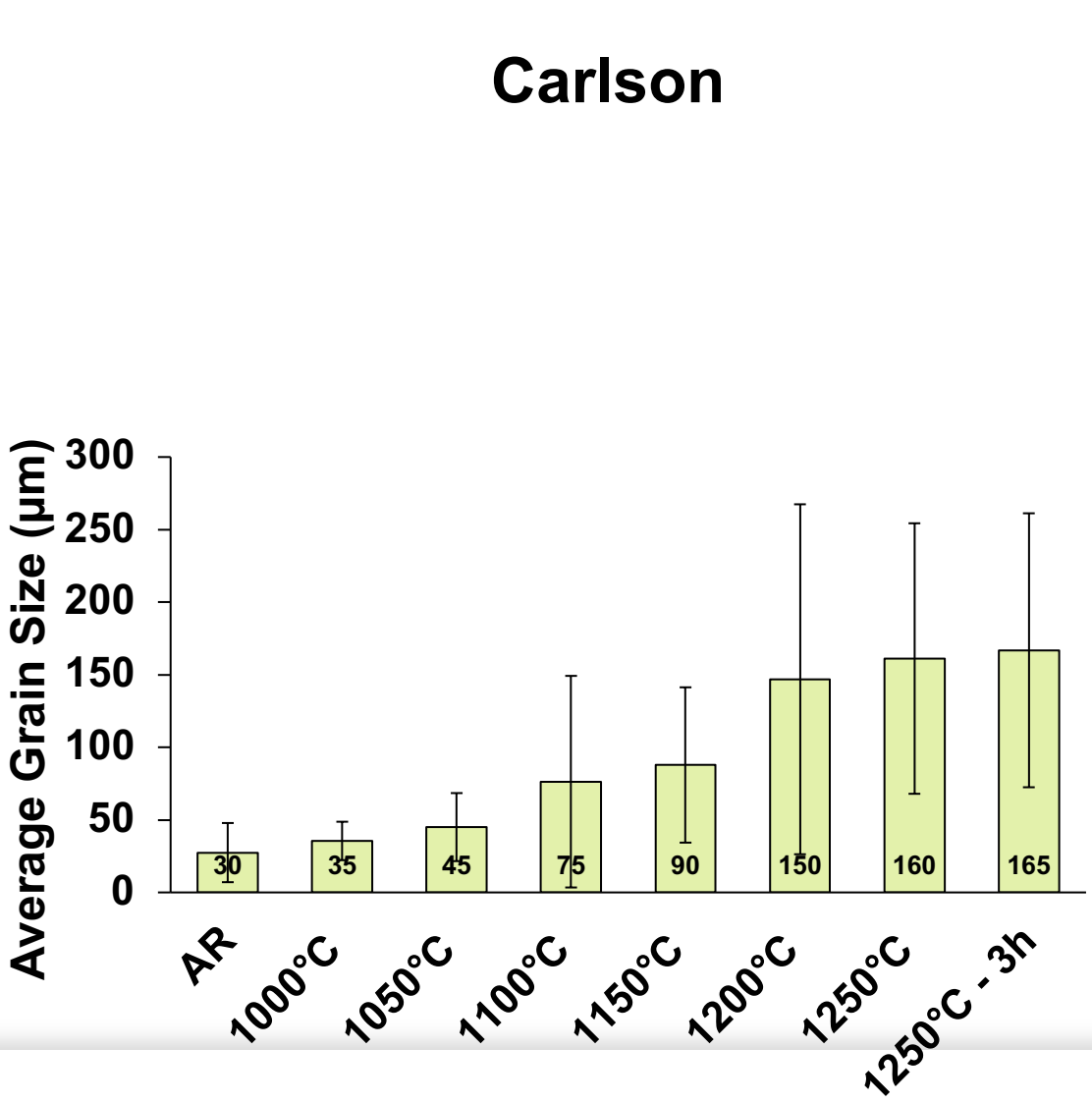
1150°C – 1h

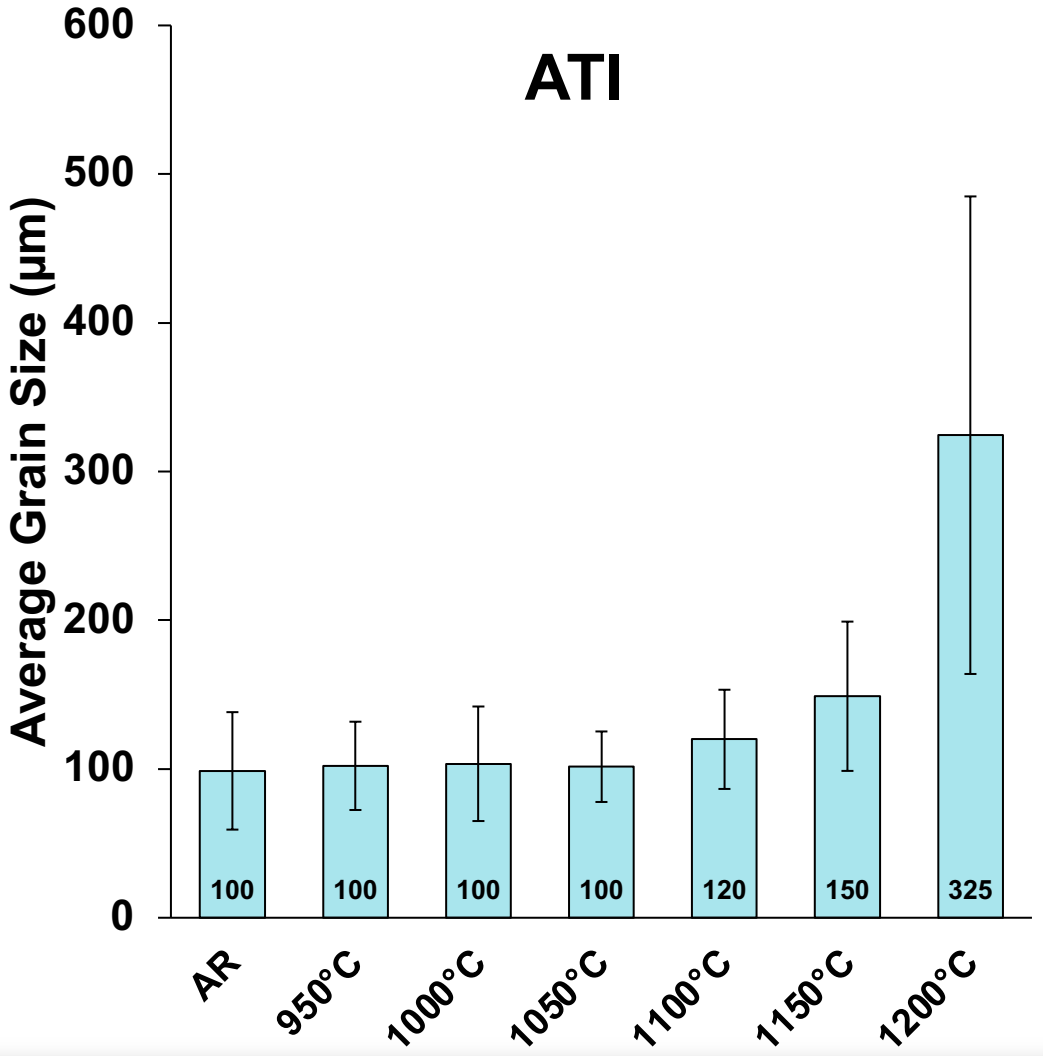


1200°C – 1h



Comparison of Grain Coarsening Behavior: Average Grain Size

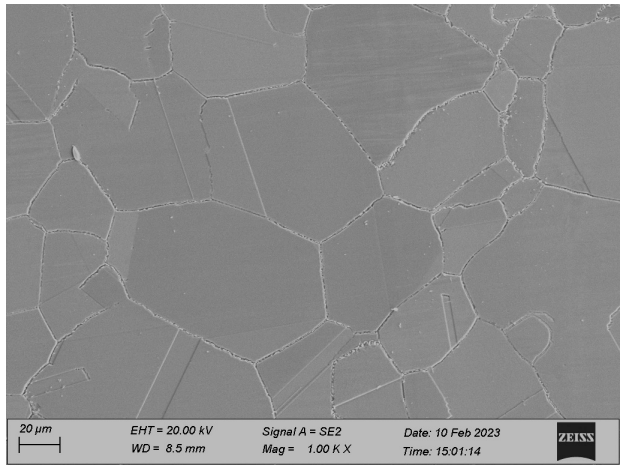


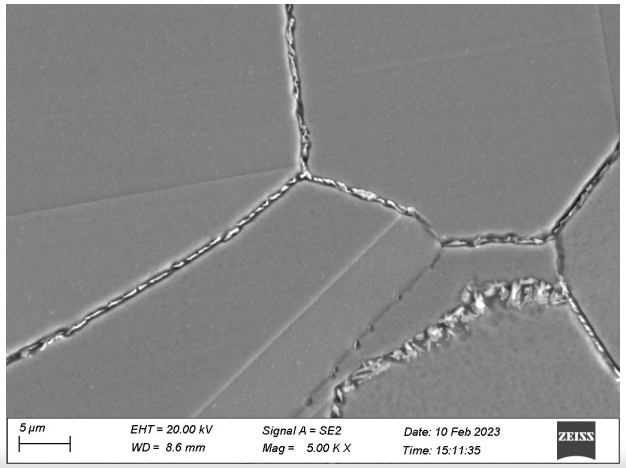


SEM Evaluation of T_{CG} Specimens – Precipitate Dissolution

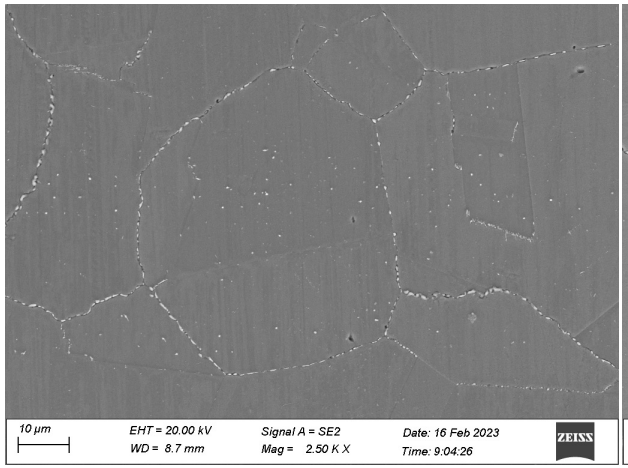
ATI

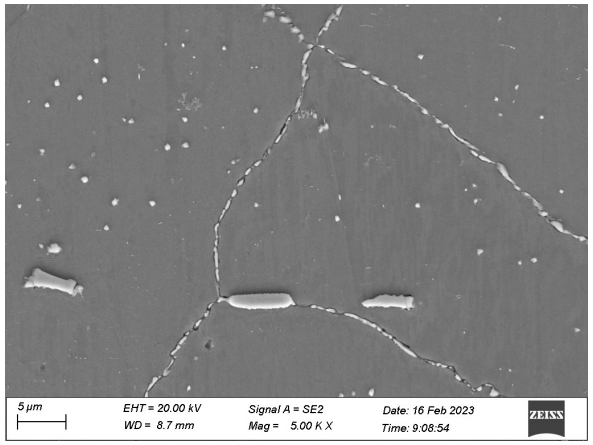
As-received



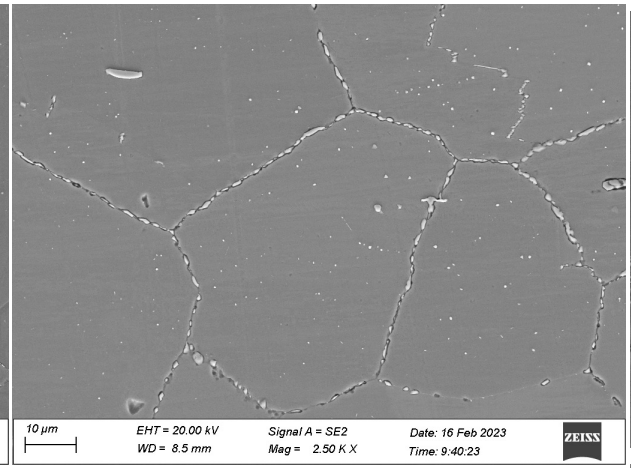


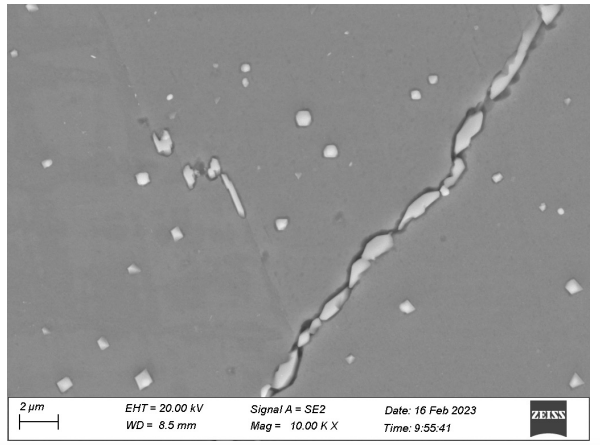
1050°C



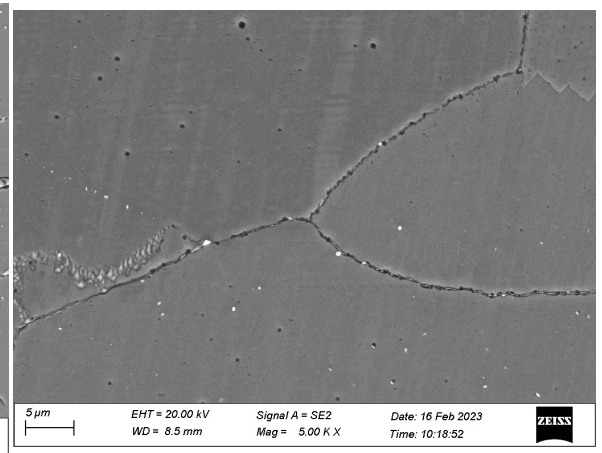


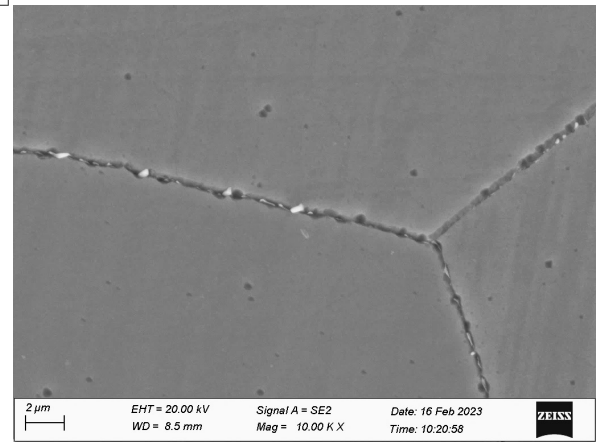
1100°C



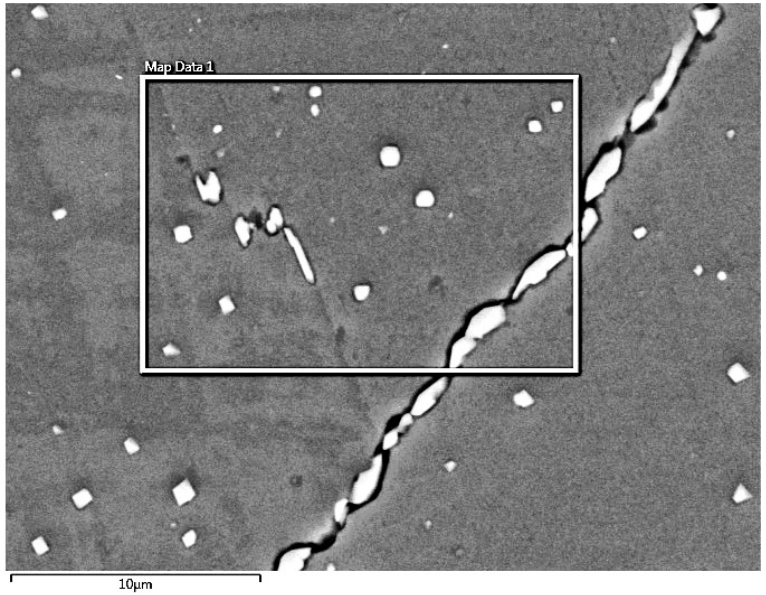


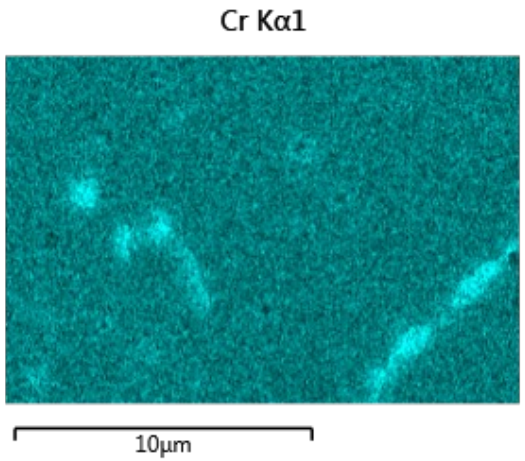
1150°C

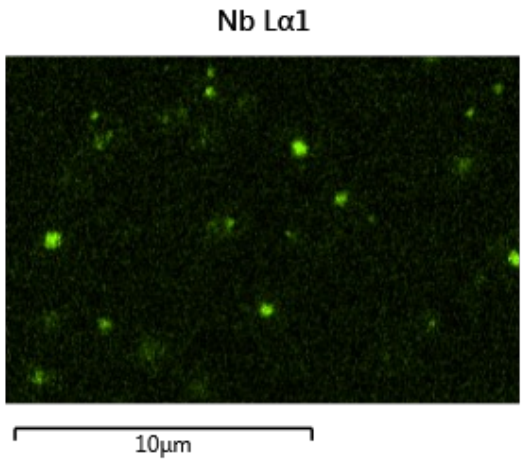


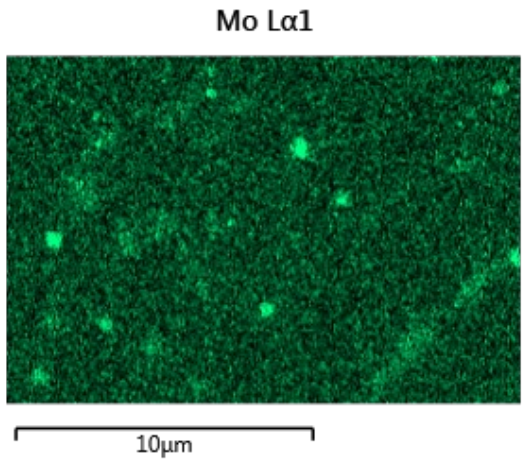


ATI: SEM-EDXS Analysis – 1h @ 1100°C





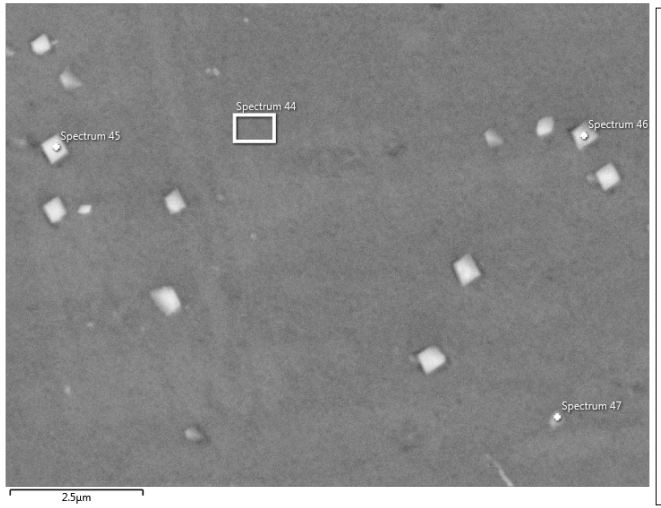


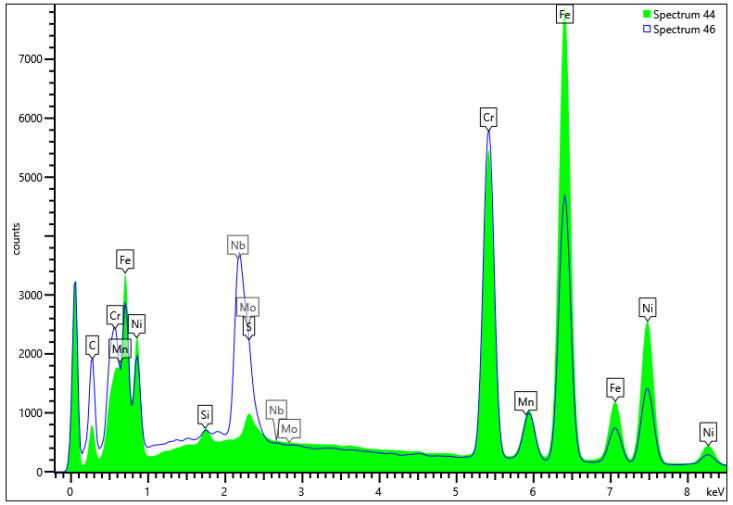


- Intergranular precipitates enriched in Cr and Mo
- Intragranular precipitates enriched in Nb and Mo

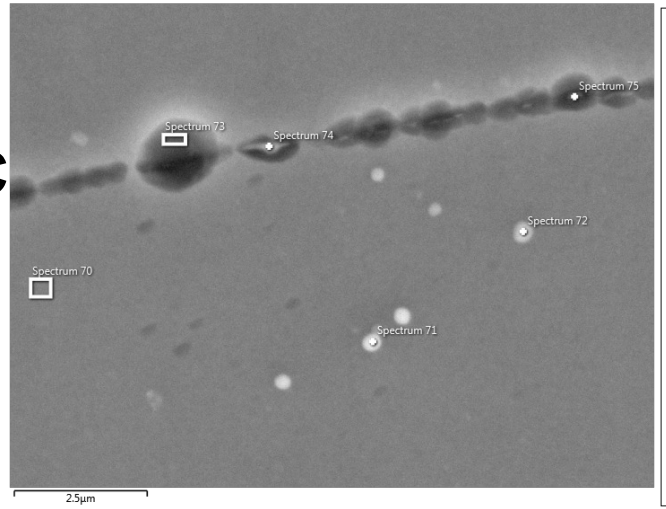
ATI: SEM-EDX Analysis

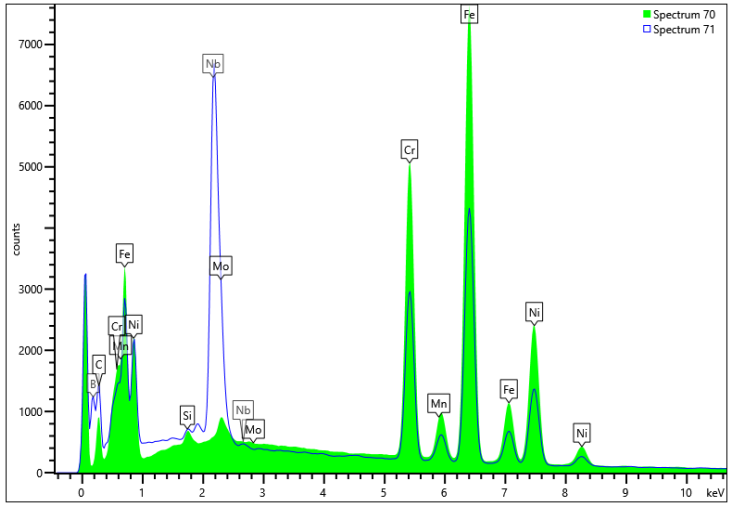
1h @ 1100°C





1h @ 1200°C

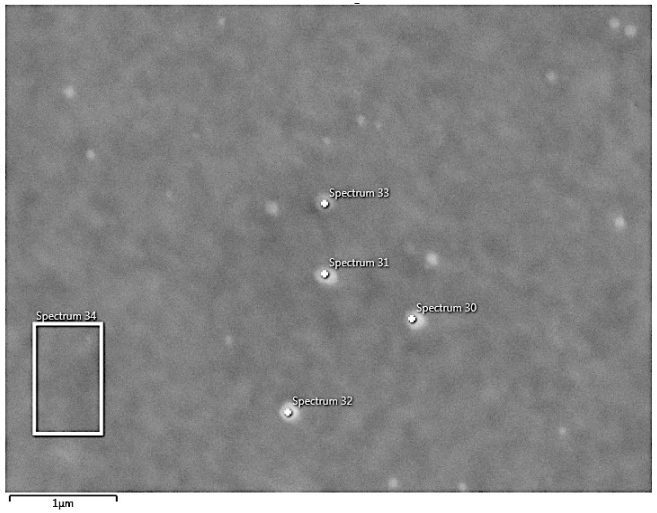


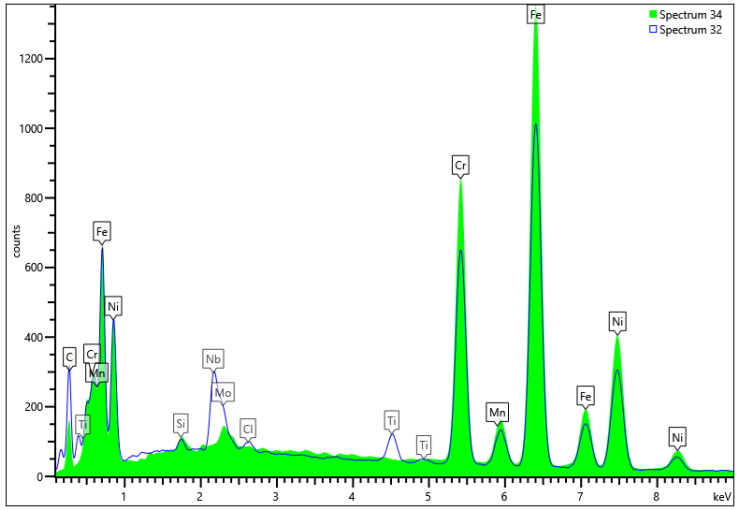


- Numerous intragranular submicron precipitates enriched in Nb-C-N (MCN)
- TEM required for detailed identification and distribution

Carlson: SEM-EDX Analysis

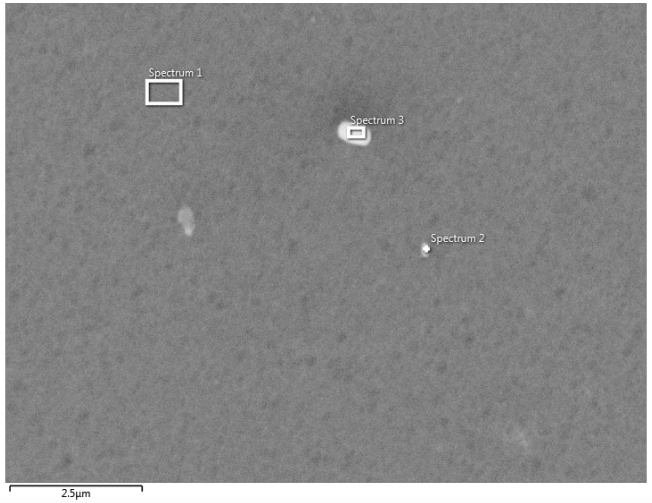
1h @ 1100°C

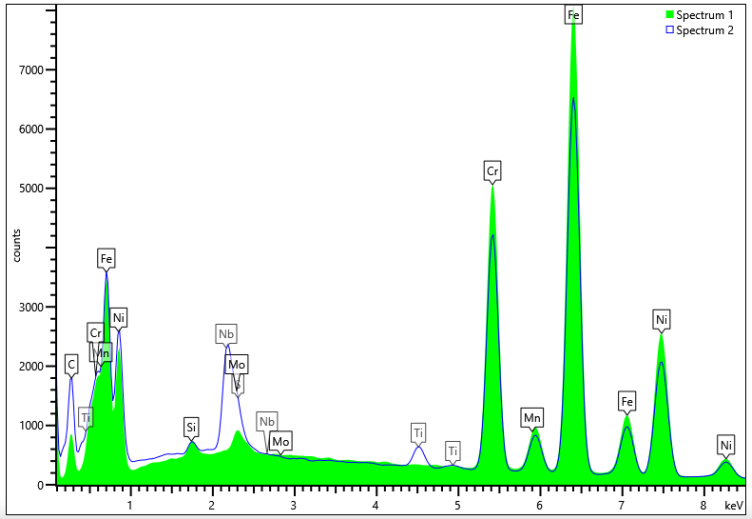




- Numerous intragranular submicron precipitates enriched in Nb-Ti-C-N (MCN)

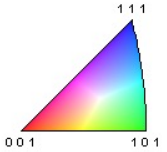
3h @ 1250°C



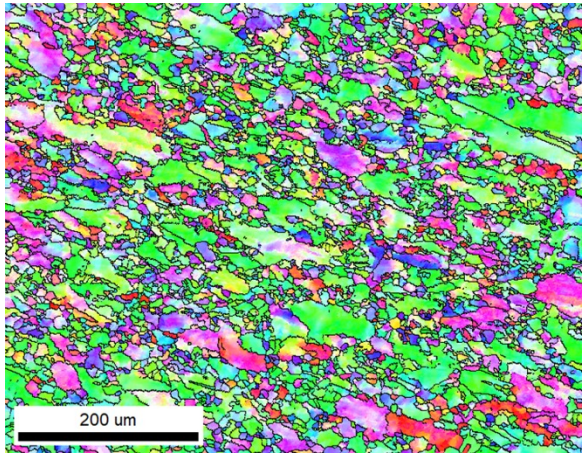


- TEM required for detailed identification and distribution

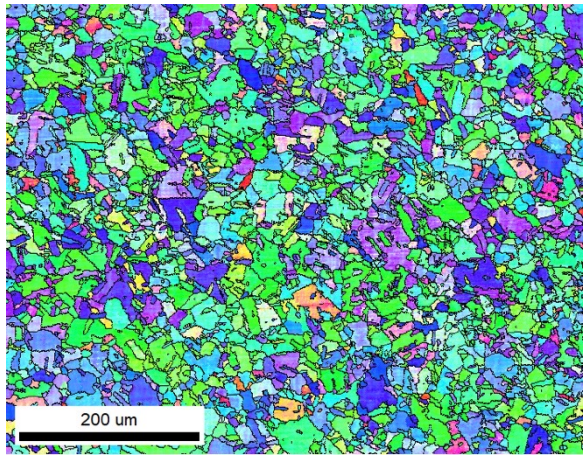
Carlson: EBSD Analysis (Grain Size)



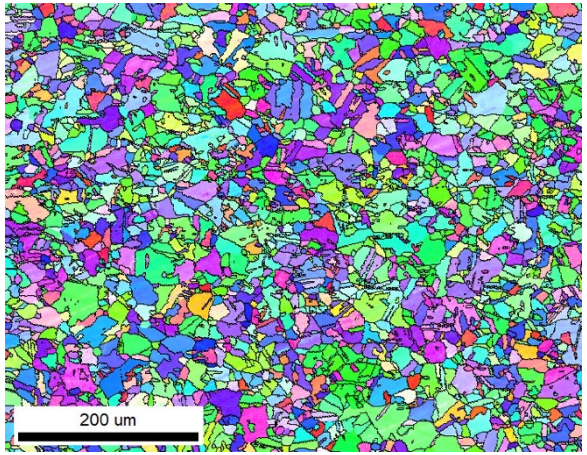
As-received



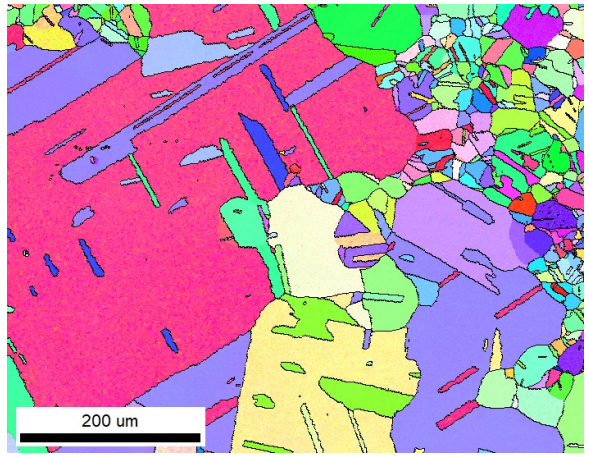
1000°C



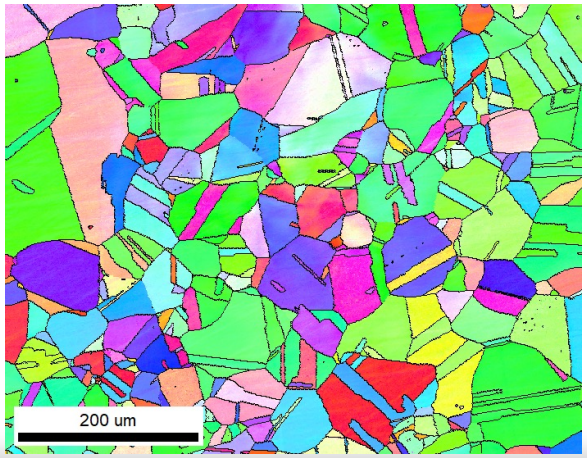
1050°C



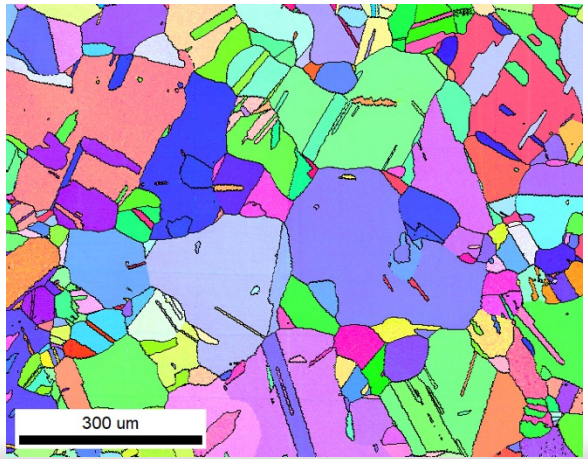
1100°C



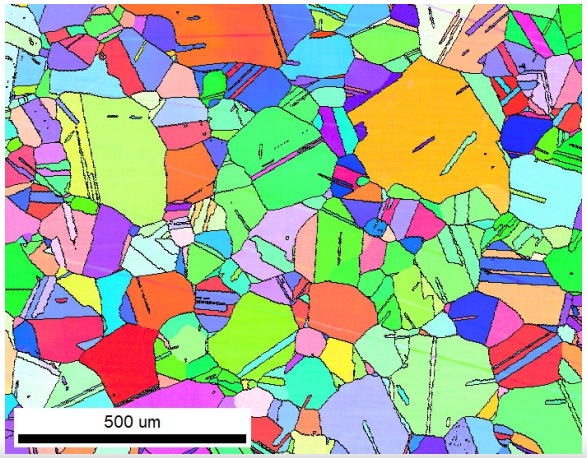
1150°C



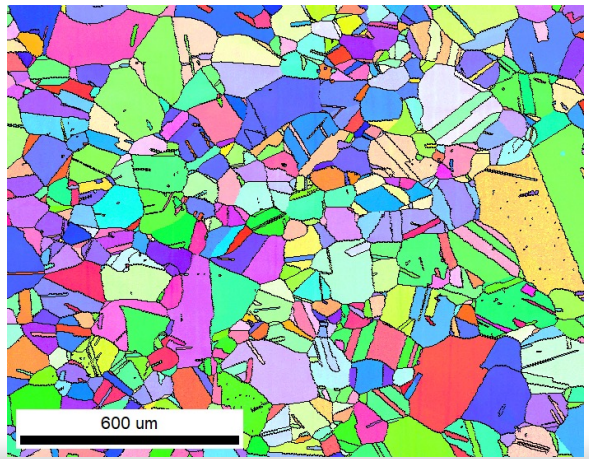
1200°C



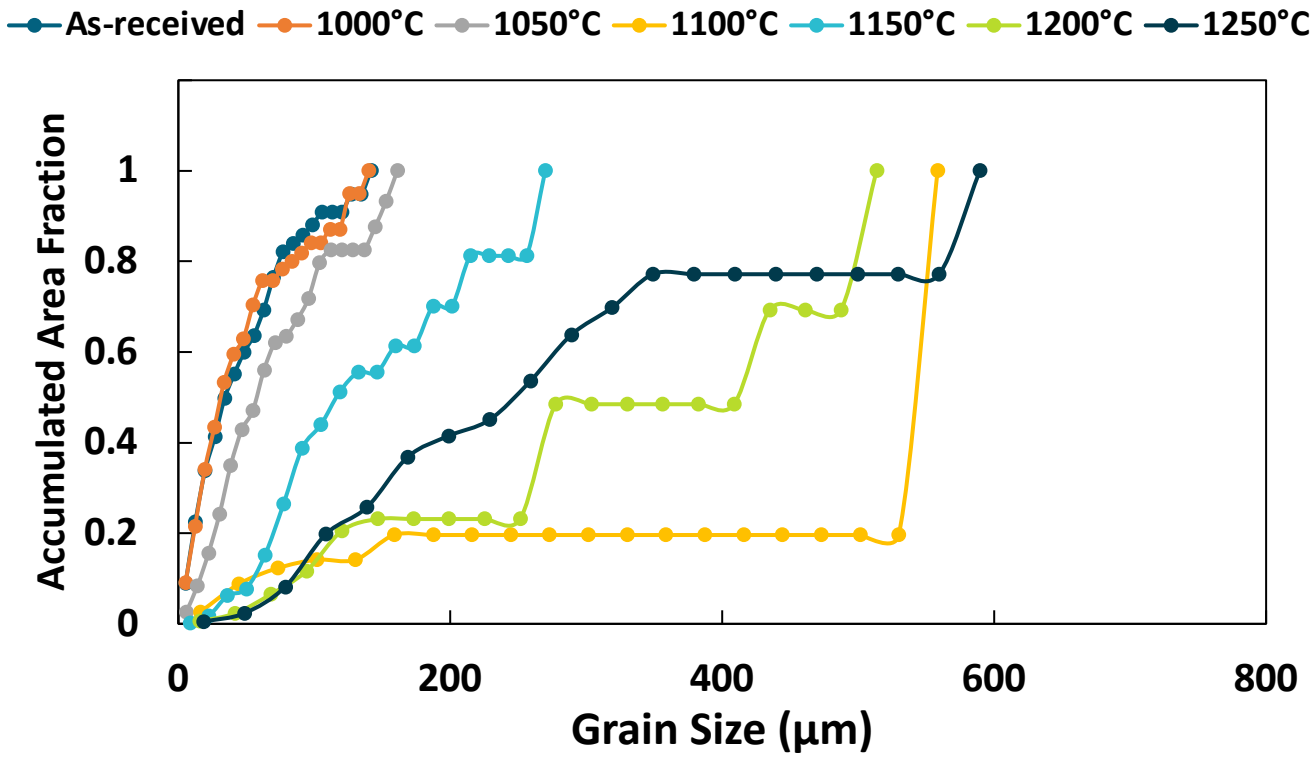
1250°C

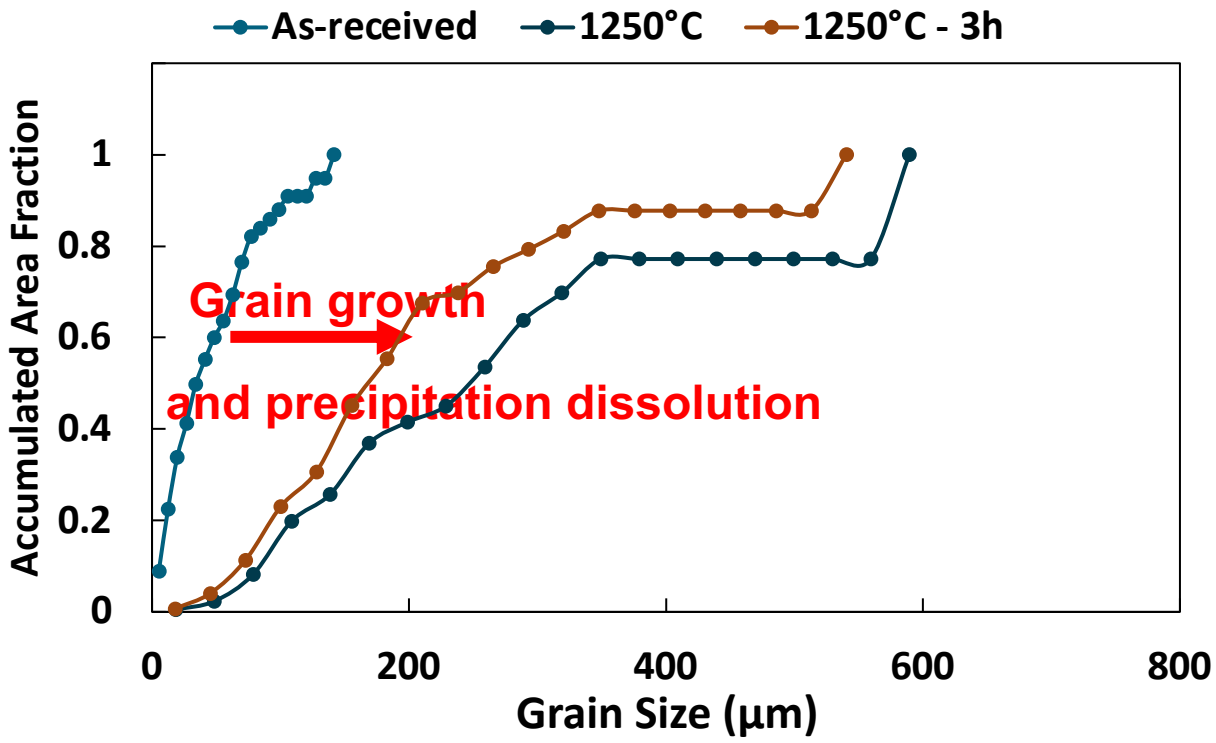


1250°C – 3 hours

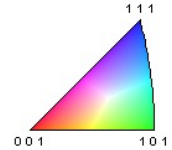


Carlson – Grain Coarsening Behavior

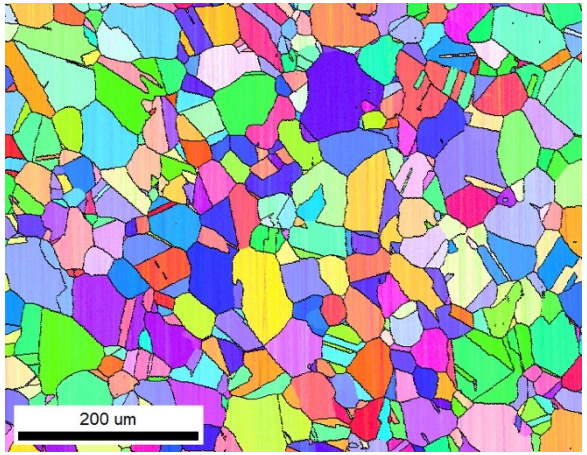




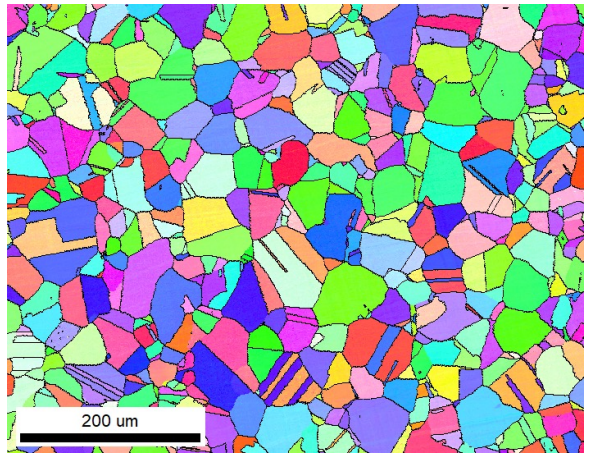
ATI: EBSD Analysis (Grain Size)



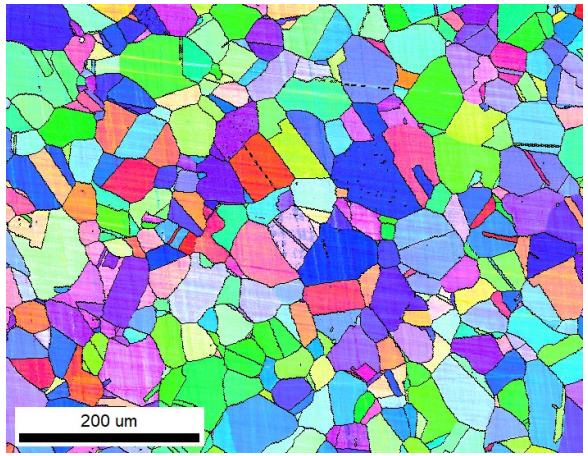
As-received



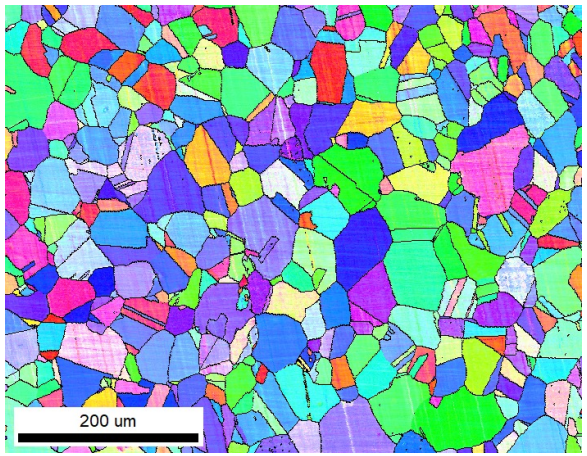
950°C



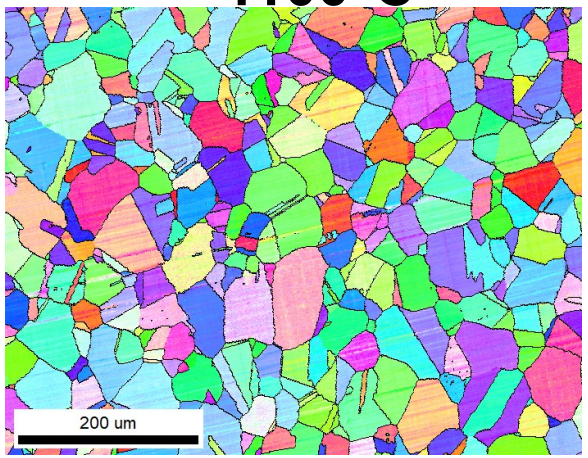
1000°C



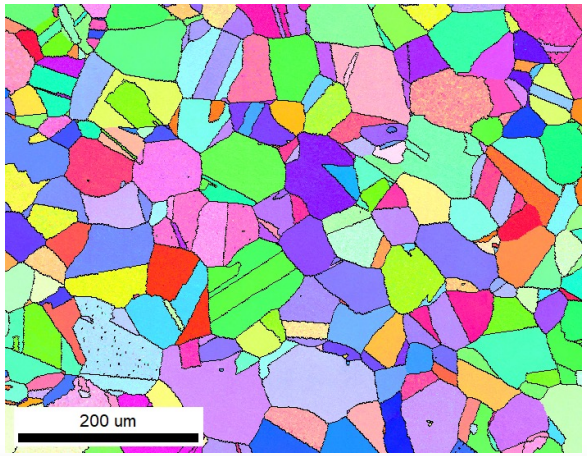
1050°C



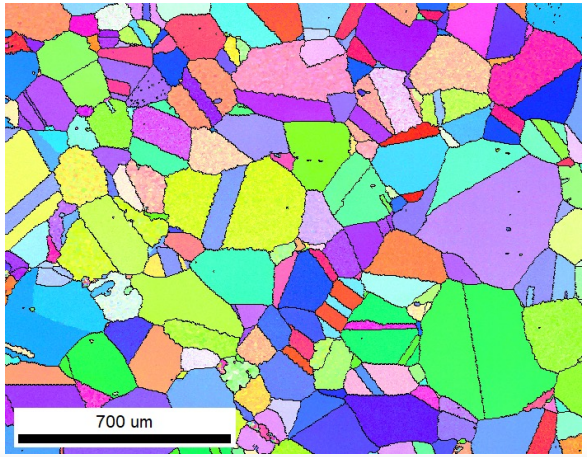
1100°C



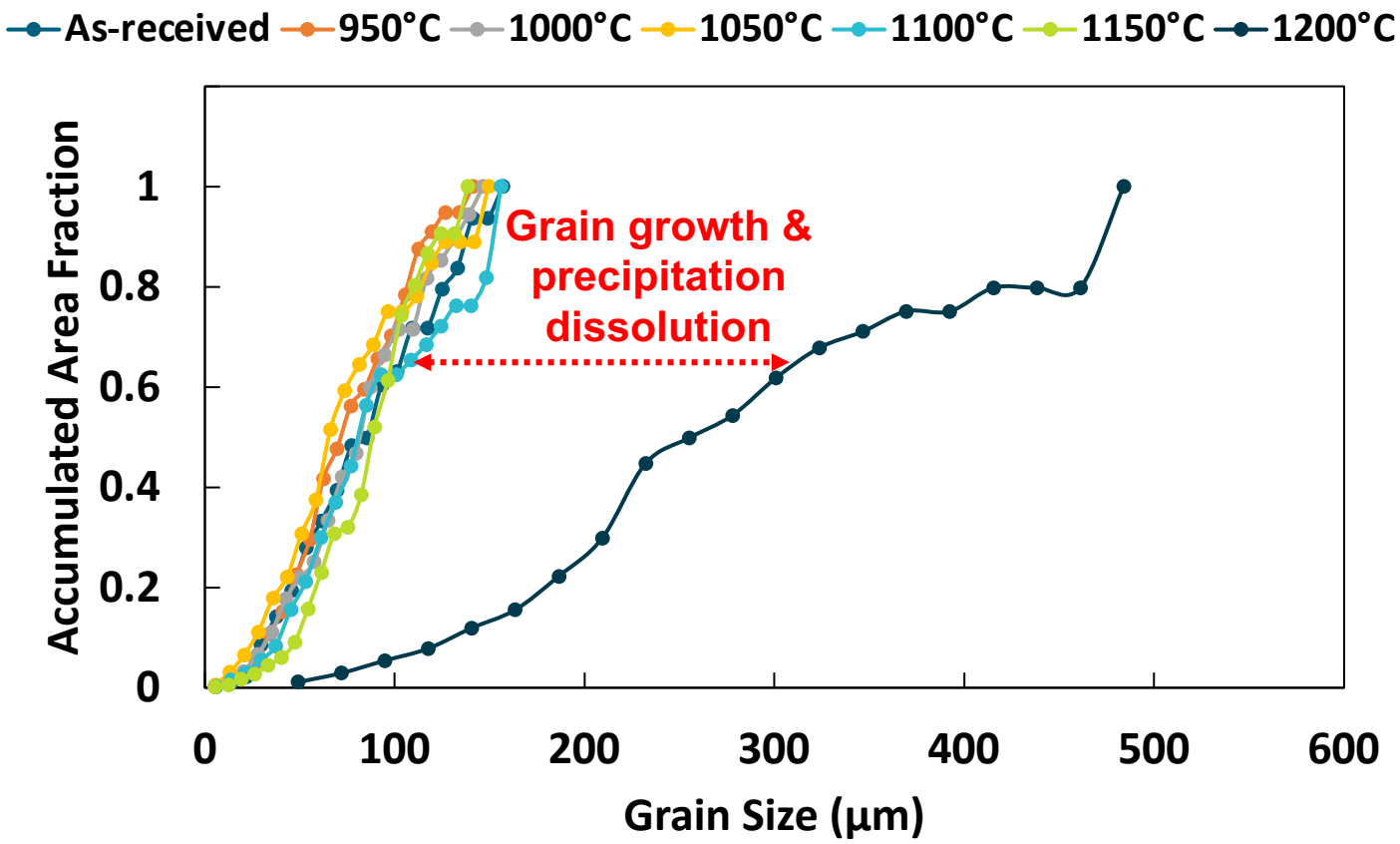
1150°C



1200°C



ATI – Grain Coarsening Behavior



Conclusions

- **Carlson** rolled plate T_{GC} **was 1250°C** based on annealing results.
- Numerous submicron **Nb-Ti-C-N-enriched intragranular precipitates** were present in specimens annealed at **1100°C** (1h) and fewer precipitates were detected at **1250°C (3h). Coarse Nb-Ti-C-N-enriched inclusions (~5 -10 μm)**

- **ATI** rolled plate T_{GC} **was 1200°C** based on annealing results.
- Numerous submicron **Nb-C(N?)** intragranular precipitates were present in specimens annealed at **1100°C,** with fewer intragranular precipitates after **1h at 1200°C. Coarse (~20+ μm) Nb-C inclusions** were non-uniformly distributed throughout the specimen.
- **Cr-Mo**-enriched intergranular and intragranular precipitates were detected in specimens annealed at 1100°C and 1150°C, in addition to the **Nb-C** precipitates.

Next Steps

- Generate **Continuous Cooling Precipitation Diagrams** for the Carlson and ATI Heats to cover the cooling rates from **~10C/sec to 0.01C/sec** to cover **plate, bar and forging** cooling rates using high speed dilatometry with characterization by **SEM and analytical electron microscopy (AEM) – TEM/electron diffraction/STEM-EDXS**.
- Perform interrupted cooling tests to confirm Precipitation-Start Temperatures.

- Opportunities for high T single and multi-pass deformation experiments **(T_{def}, strain, strain-rate,% recrystallized)** to identify appropriate thermomechanical processing windows for bars and forgings to generate uniform microstructures.

Thank you for your attention

U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY