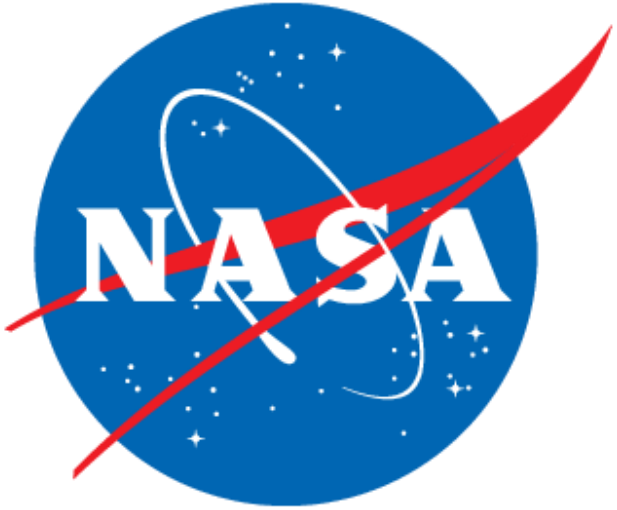
Metallography of Additively Manufactured Materials

Henry Kaplan
Araben Tablizo
Summer 2023



About Us

Henry Kaplan

- Third year physics major at Yale University
- From Brooklyn, NY

Araben Tablizo

- Rising 2nd year ChemE Masters student at Johns Hopkins University
- Earned Bachelors of Science in BME/MatSci at Rensselaer Polytechnic Institute

Project Overview

- Develop metallographic techniques to reveal microstructures of the following additively manufactured metals
  - **GRX 810**
  - Al 6061/6061RAM2
  - Haynes 214
- Optical microscopy and SEM will be employed to analyze microstructures of the above metals

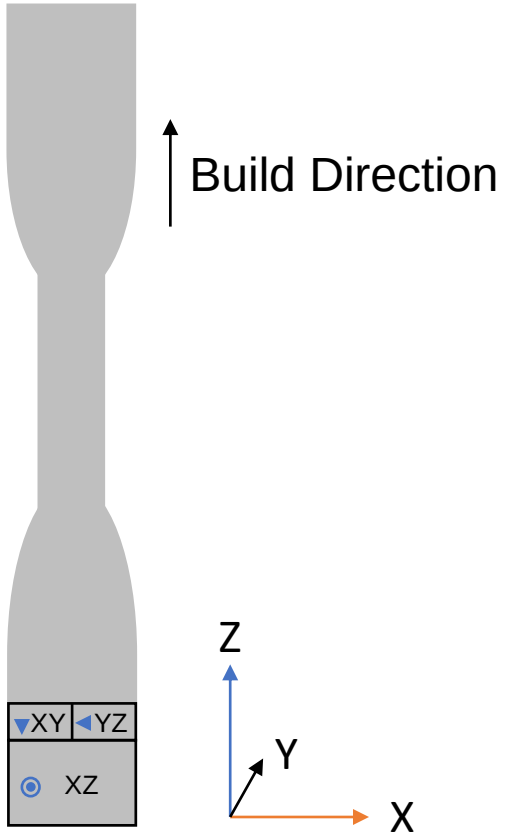
GRX-810

- Experimental nickel-based superalloy developed at GRC
- Y_2O_3 dispersion strengthened to improve mechanical properties
- Nominal composition in wt %

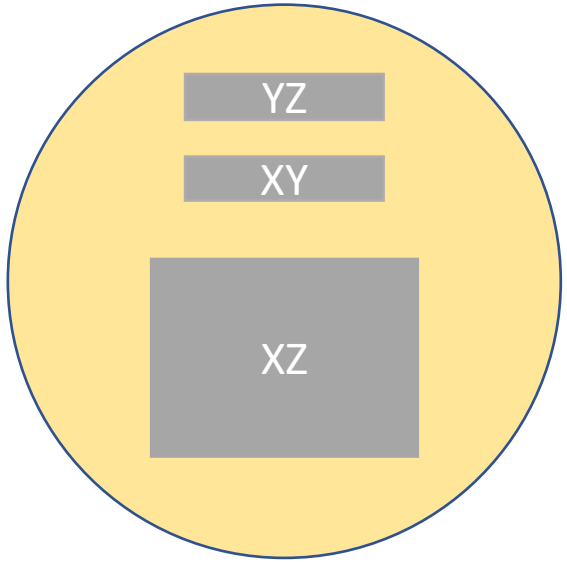
Ni	Co	Cr	Re	Al	Ti	Nb	W	C
bal	33	29	1.5	0.3	0.25	0.75	3.0	0.05

- Can withstand temps > 2000°F
- 2x as strong,1,000x more durable than current AM superalloys, twice as resistant to oxidation

GRX-810 Cut Plan

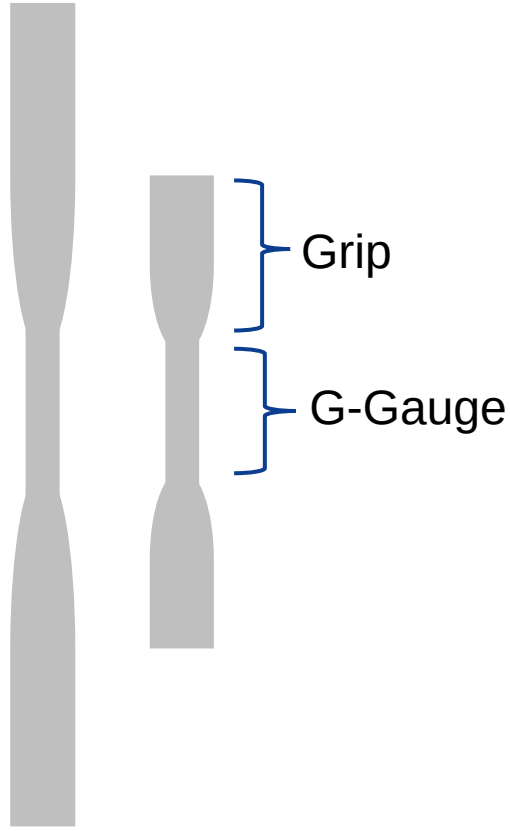


Mounting Scheme



Labeling Scheme

L refers to longer specimens
G refers to gauge section



L-Long

Cutting and Mounting Procedure for GRX-810

- All sections were cut from as-built and heat treated tensile specimens using Struers Secotom-50 machines found in the cutting room with 10A20 SiC blades at ~2000 rpm at feeds no more than 5 mm/s
- All GRX-810 specimens were mounted with LevoFast using the Struers predefined methods

Grinding and Polishing Procedure for GRX-810

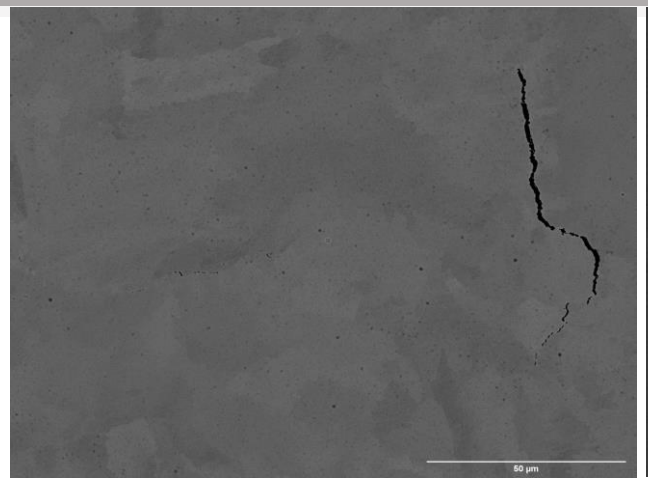
Grinding Procedure						Polishing Procedure							
Grit	Time	Force	Plate Speed	Holder Speed	Rotation Type	Plate	Solution	Time	Force	Plate Speed	Holder Speed	Rotation Type	Dose
220	Until plane	25 N	150 rpm	150 rpm	Co-rotation	MD-Dac	DiaDuo-2 3 µm	5:00	25 N	150 rpm	150 rpm	Co-rotation	15
500	3:00	25 N	150 rpm	150 rpm	Co-rotation	MD-Mol	DiaDuo-2 1 µm	5:00	25 N	150 rpm	150 rpm	Co-rotation	15
1200	3:00	25 N	150 rpm	150 rpm	Co-rotation	MD-Nap	MasterPrep Alumina	5:00	25 N	150 rpm	150 rpm	Counter-rotation	Manual
2000	3:00	25 N	150 rpm	150 rpm	Co-rotation								
4000	3:00	25 N	150 rpm	150 rpm	Co-rotation								

- Adapting the Al 6061 procedure and using data from the ASM Handbook on Ni-based superalloys, this procedure was used

Etching Procedure for GRX-810

- Since GRX-810 has similar composition to Haynes HR-160, etching procedure for Haynes HR-160 was attempted
- Electrolytic etching in 5g Oxalic, 95 mL HCl at 25 °C. Stainless steel cathode, carbon cathode at 6V with 1-2 s contact. Etch until blue film covers specimen surface evenly

SEM As Built and Heat Treated XY-Plane, 1000x Build⊗



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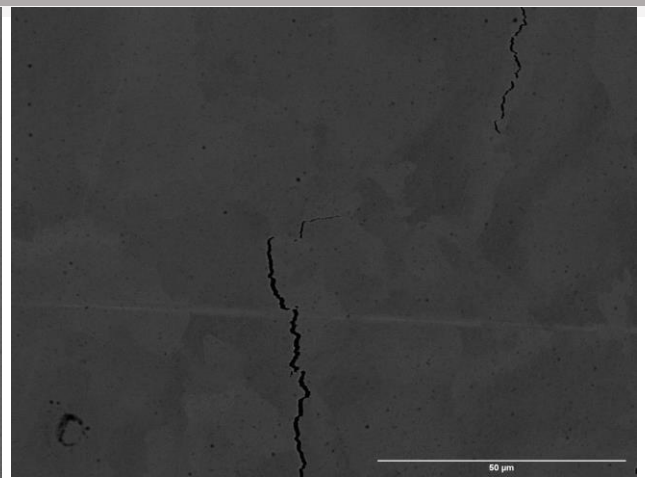
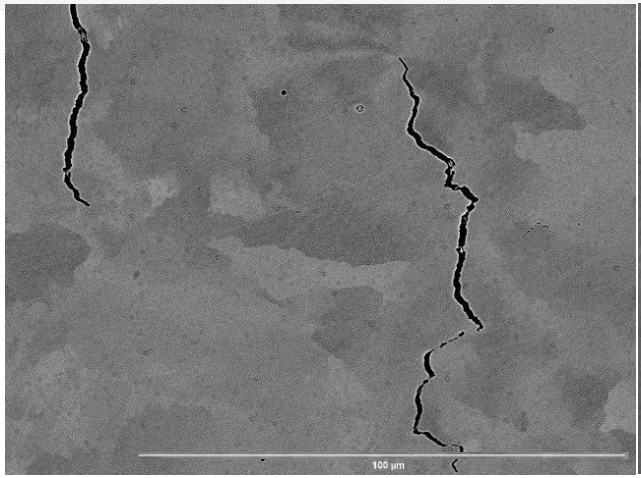


1.5mm AB



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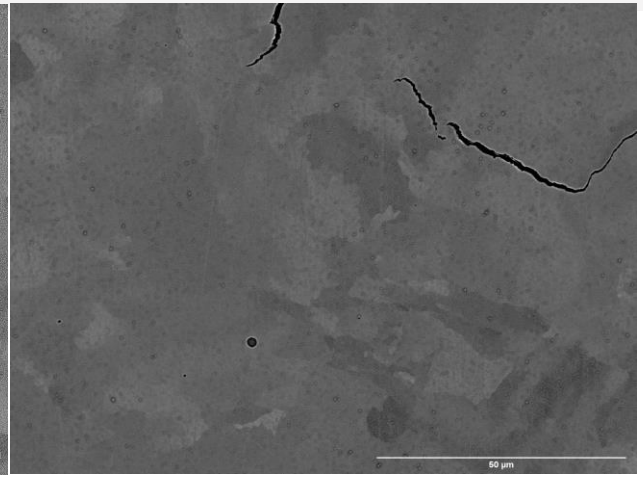

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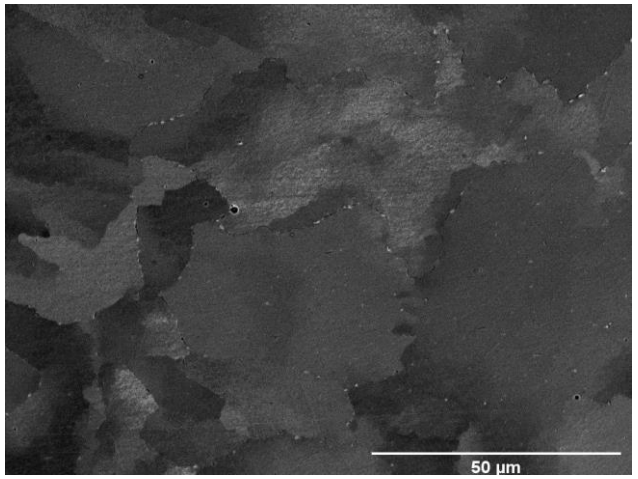

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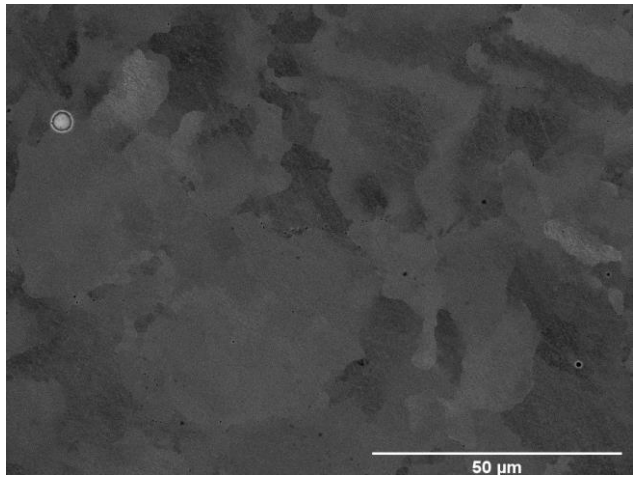

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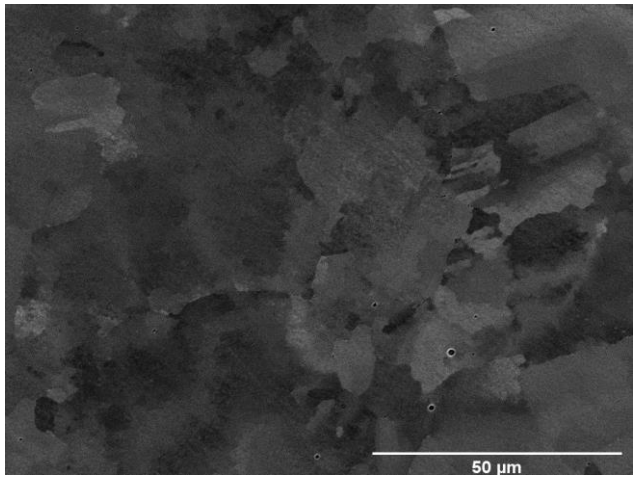

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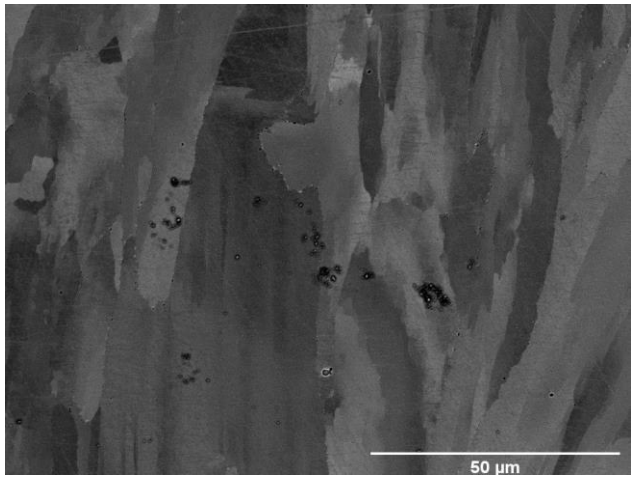

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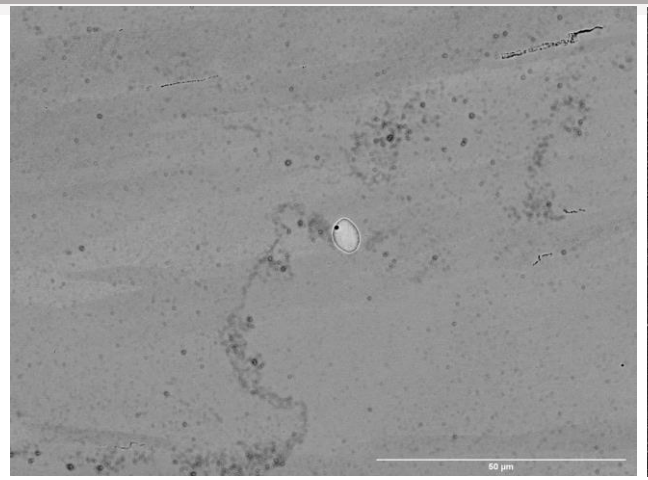
SEM As Built and Heat Treated YZ-Plane, 1000x Build ↑



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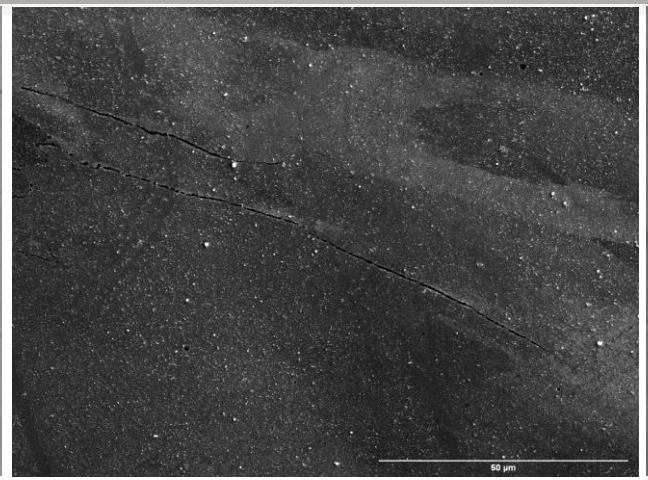

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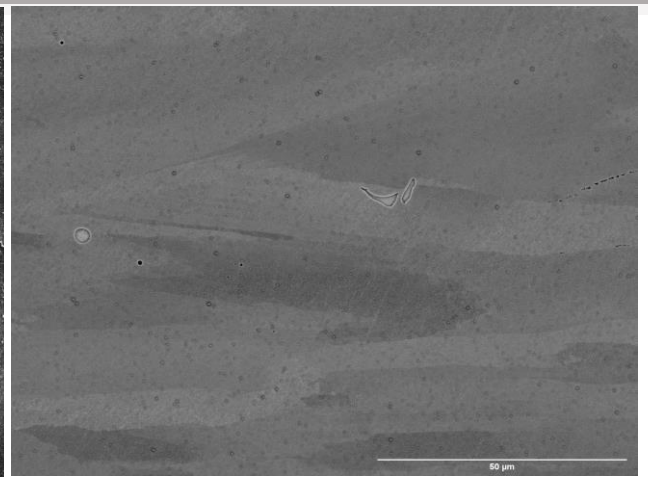

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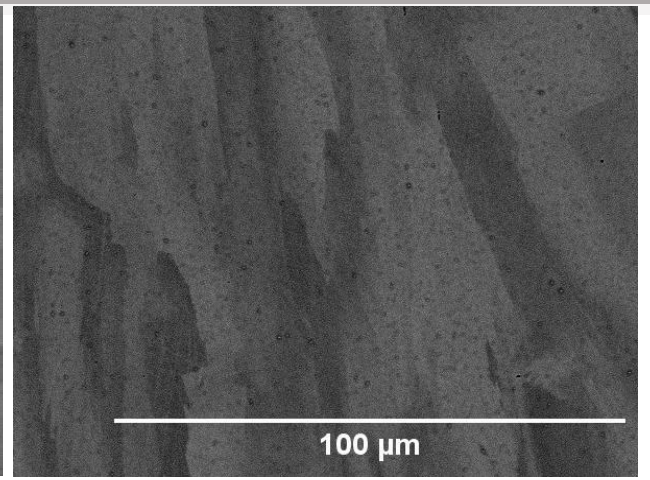

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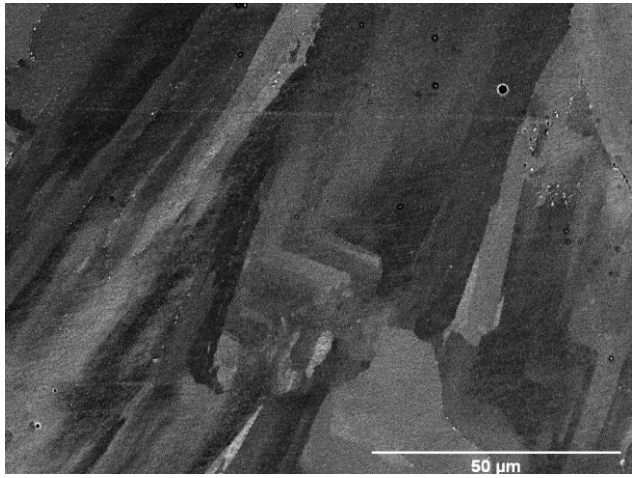

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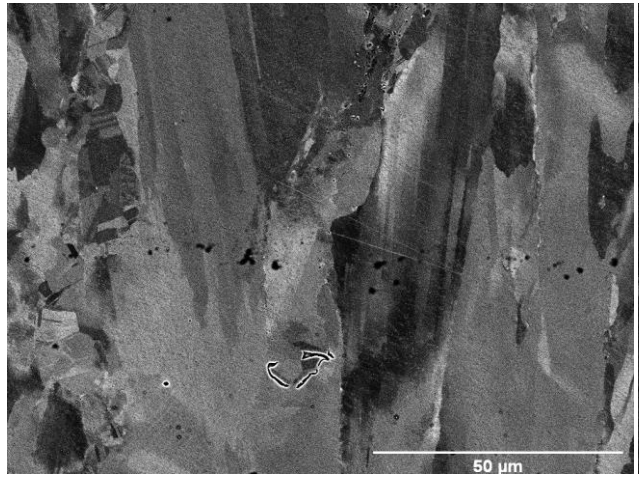

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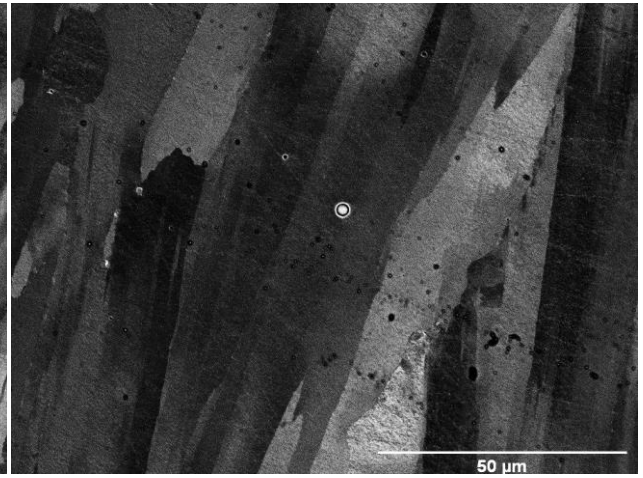

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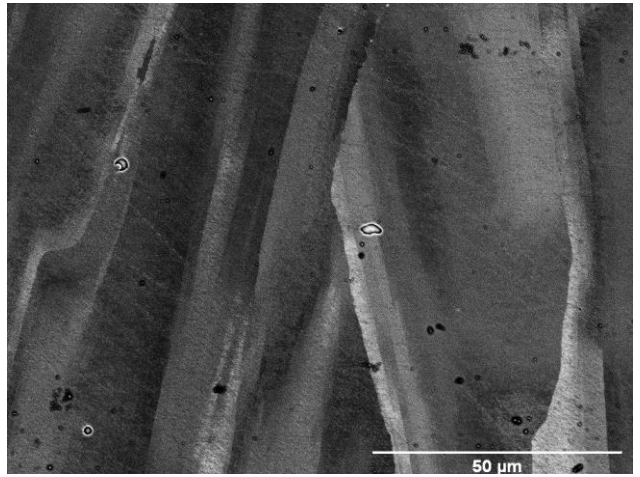
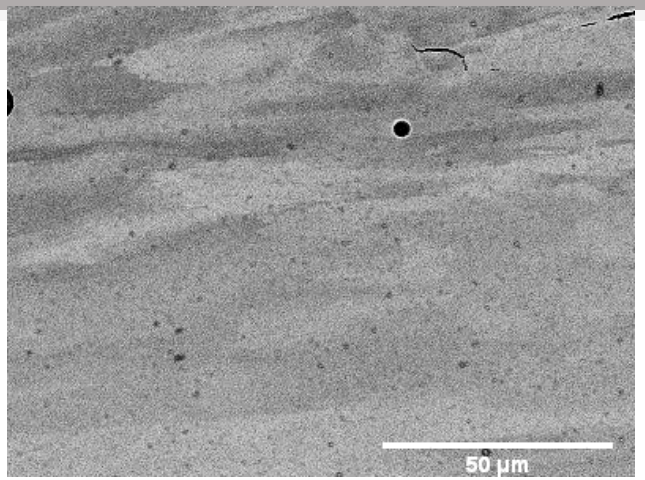


2.5mm HT

SEM As Built and Heat Treated XZ-Plane, 1000x Build ↑



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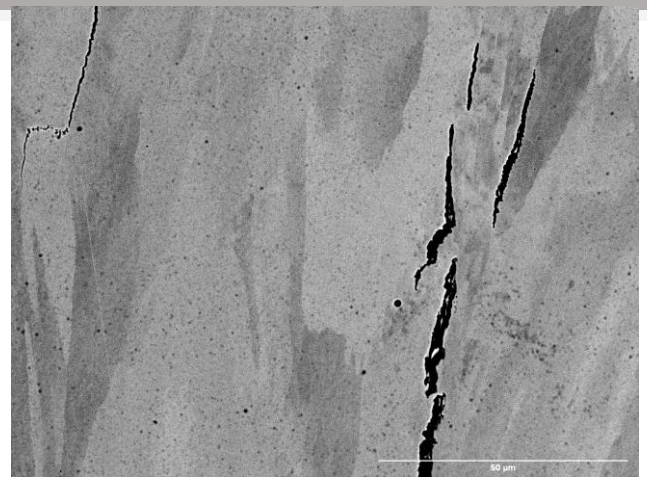

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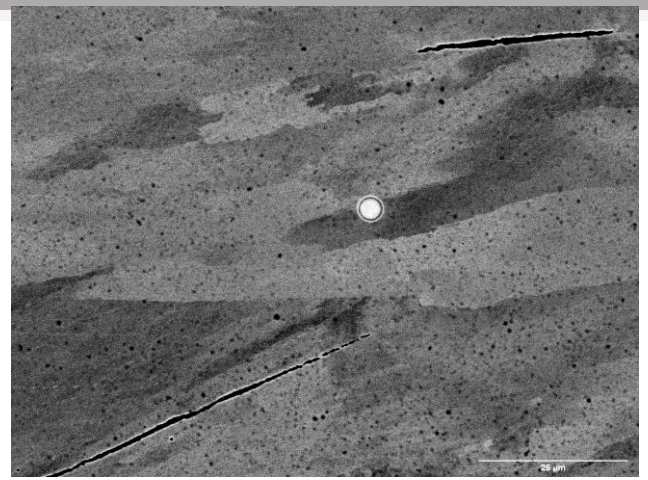

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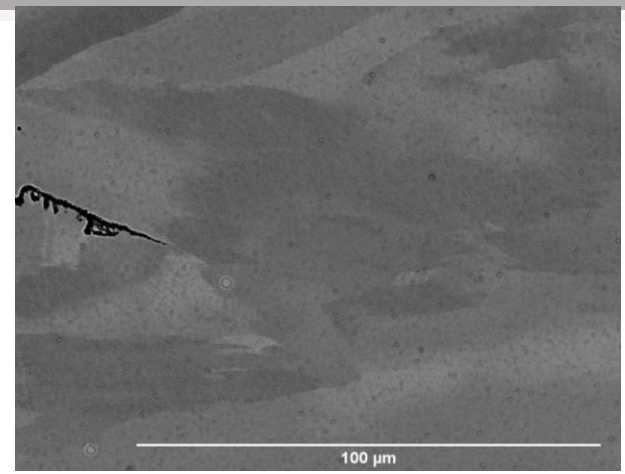

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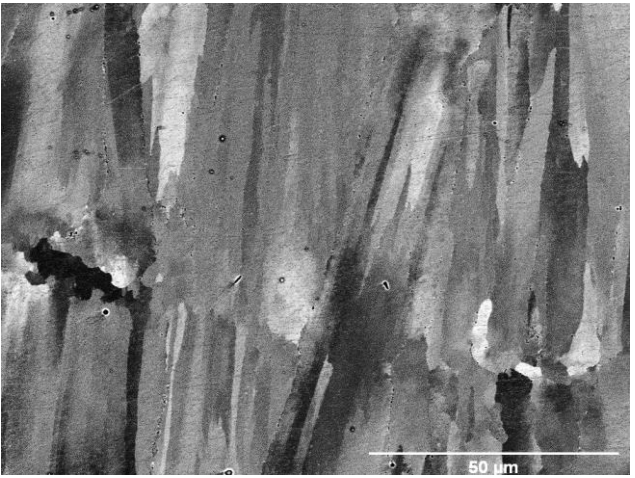

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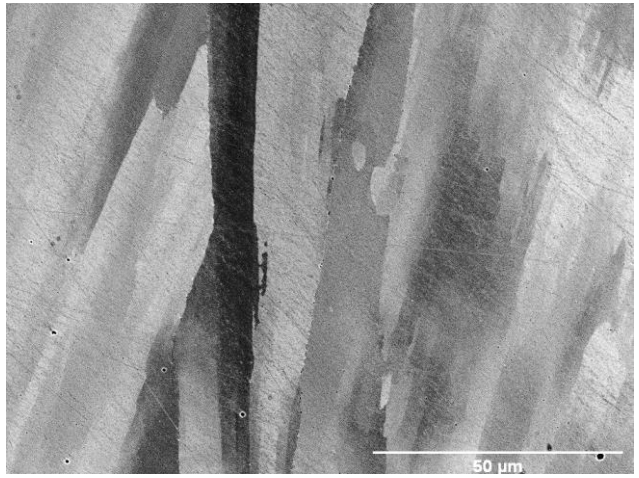

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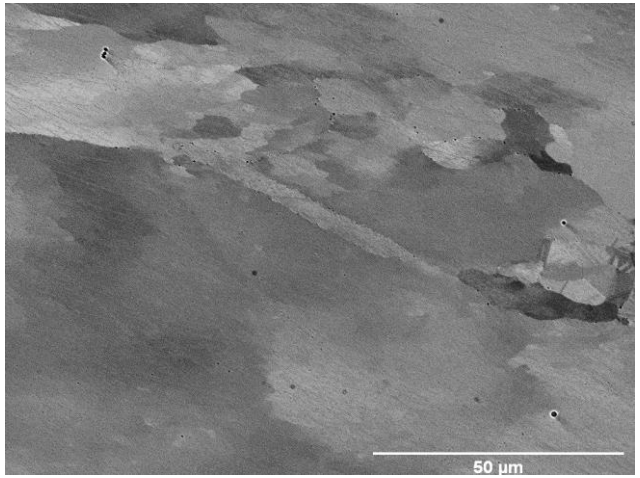

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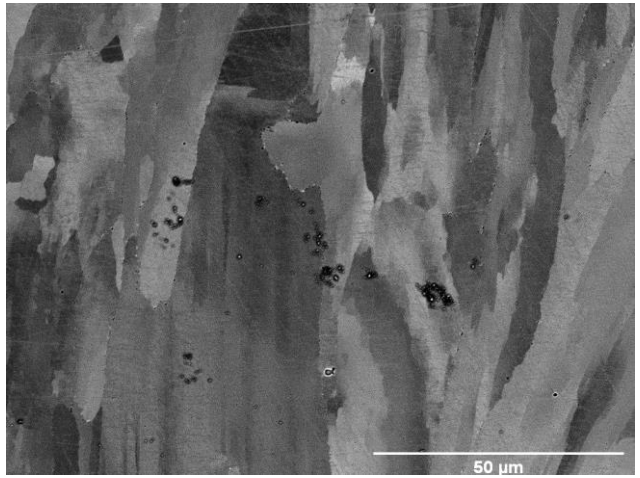
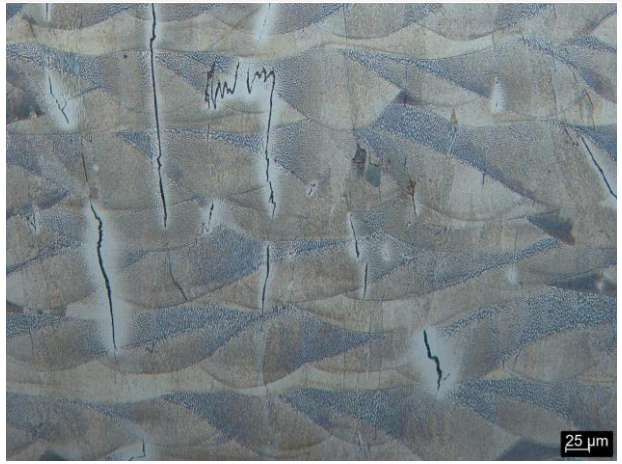


2.5mm HT

Etched As Built and Heat Treated XZ-Plane, 200x Build ↑

1mm AB



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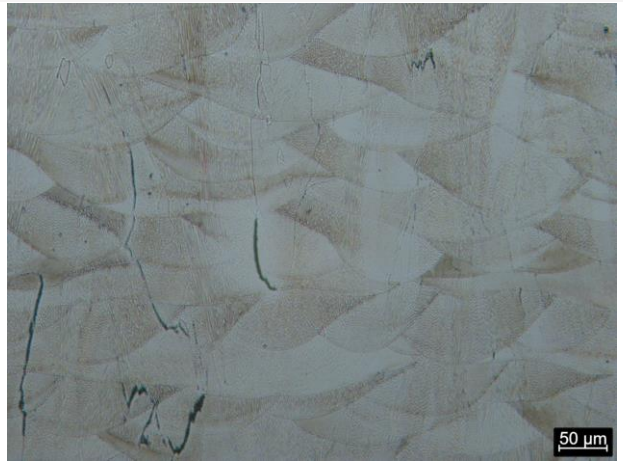
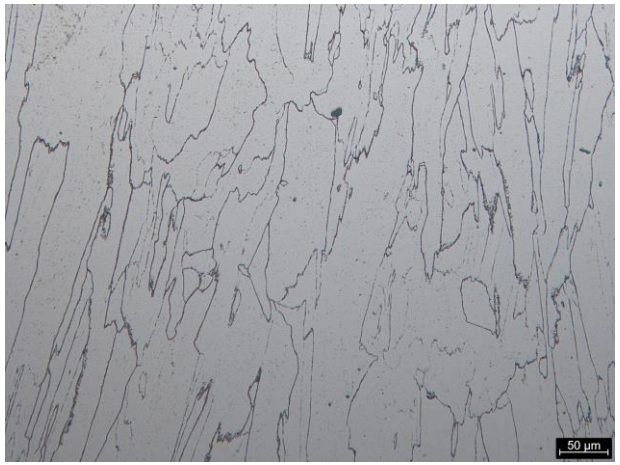


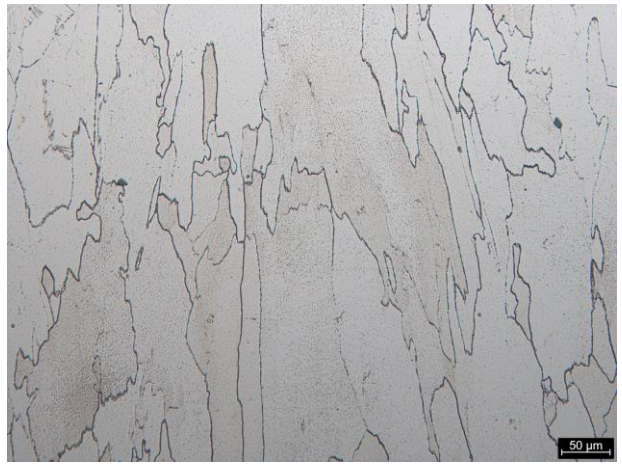
2mm AB

2.5mm AB



1mm HT

1.5mm HT



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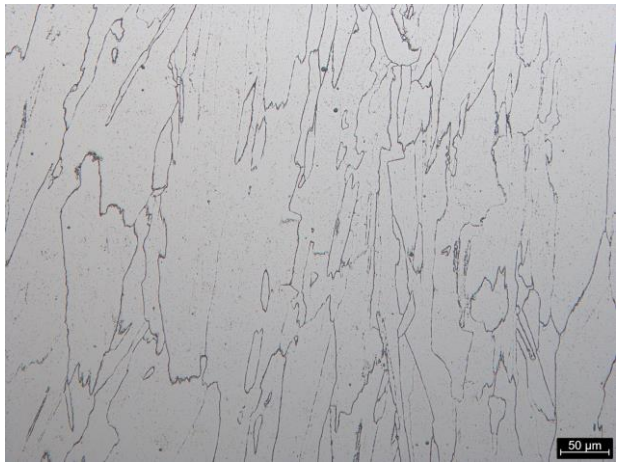

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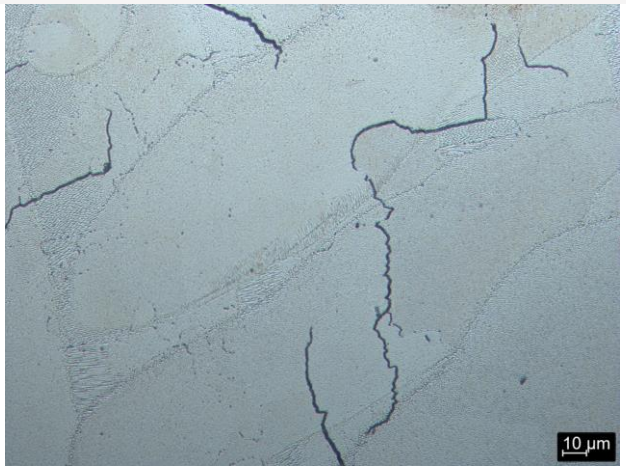
Etched As Built and Heat Treated XY-Plane, 500x Build ⊗



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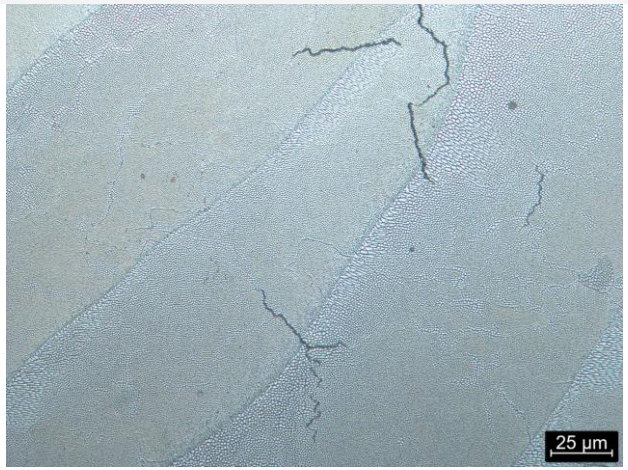
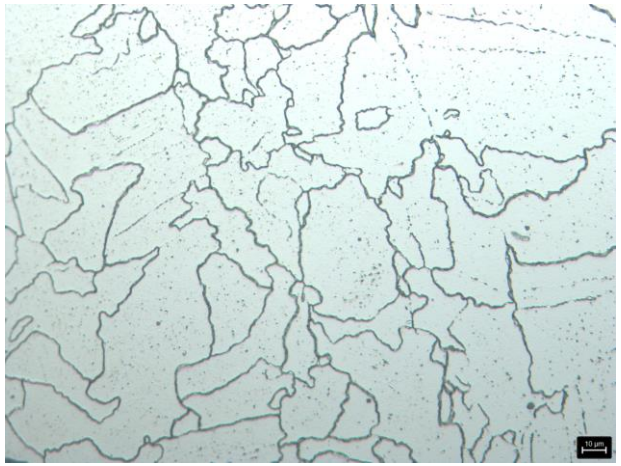


1.5mm AB

2mm AB

2.5mm AB



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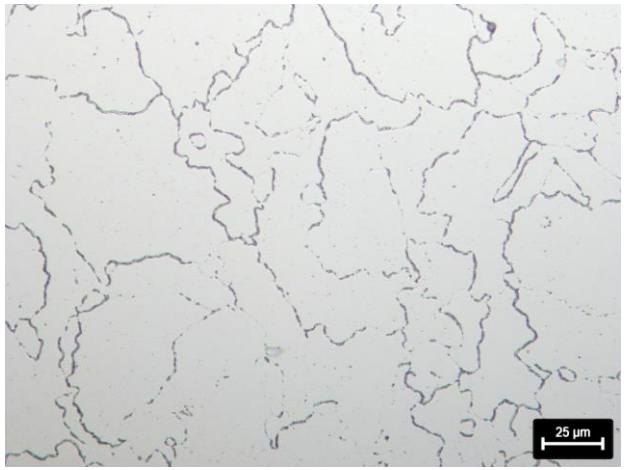

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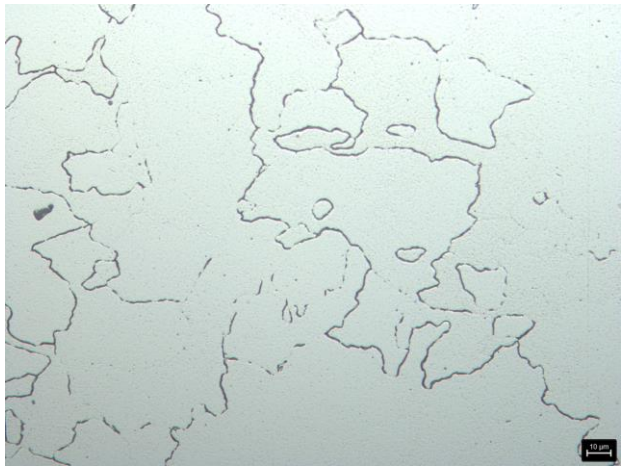

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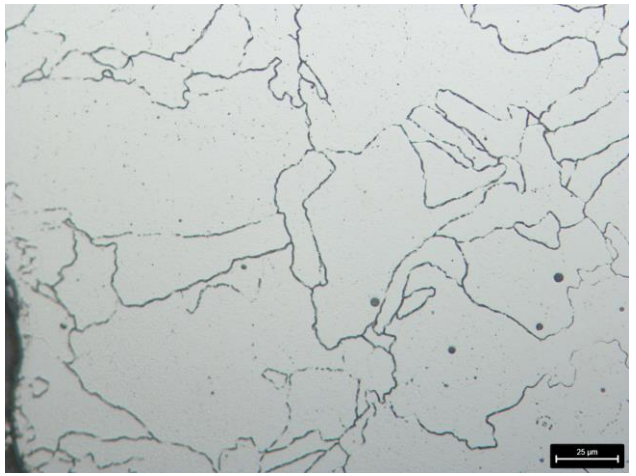
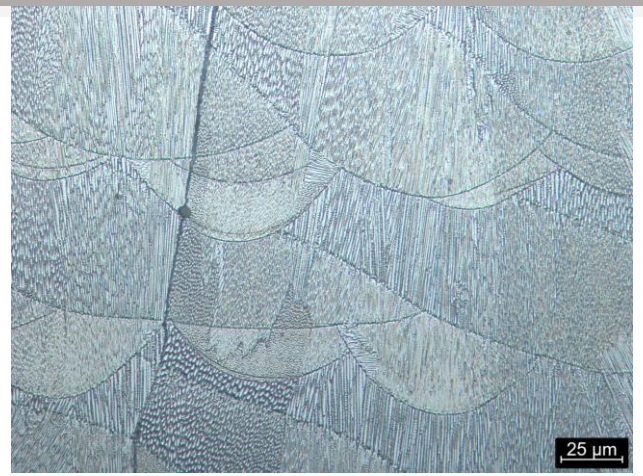


2.5mm HT

Etched As Built and Heat Treated YZ-Plane, 500x Build ↑

1mm AB



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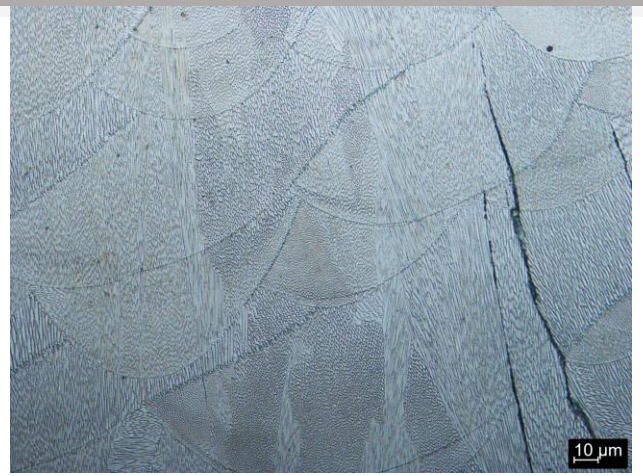
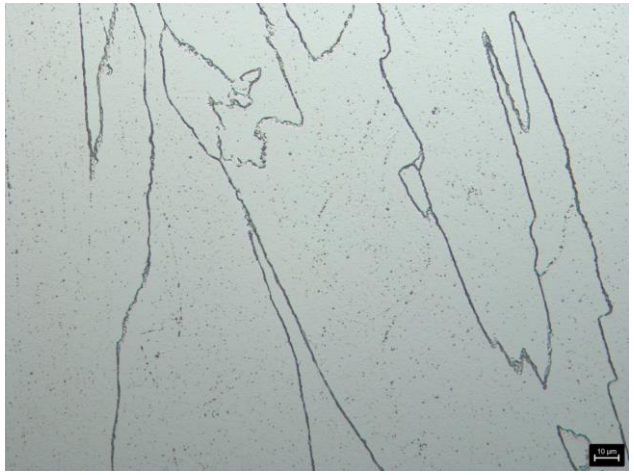


2mm AB

2.5mm AB



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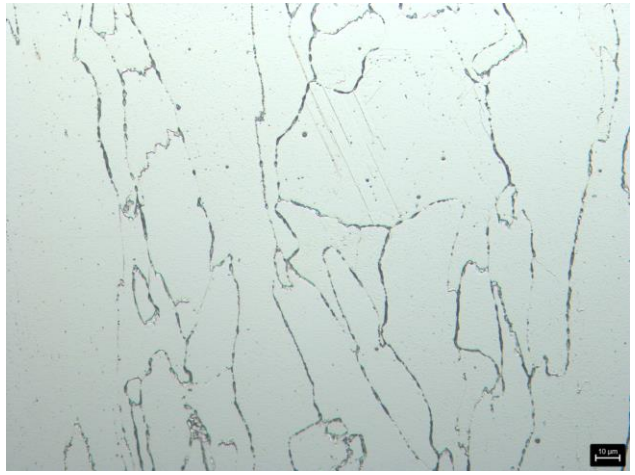

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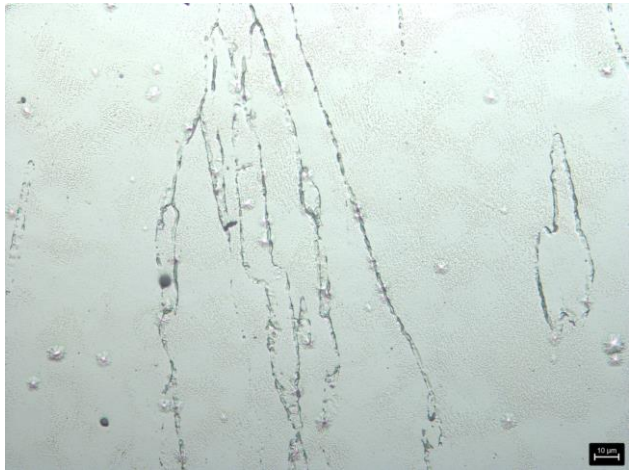

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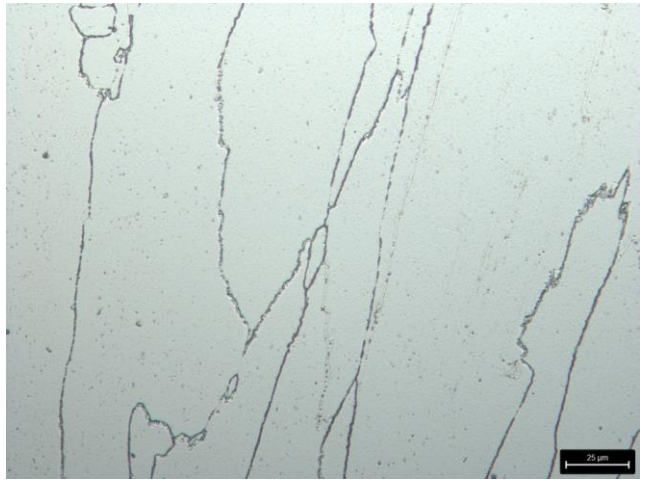
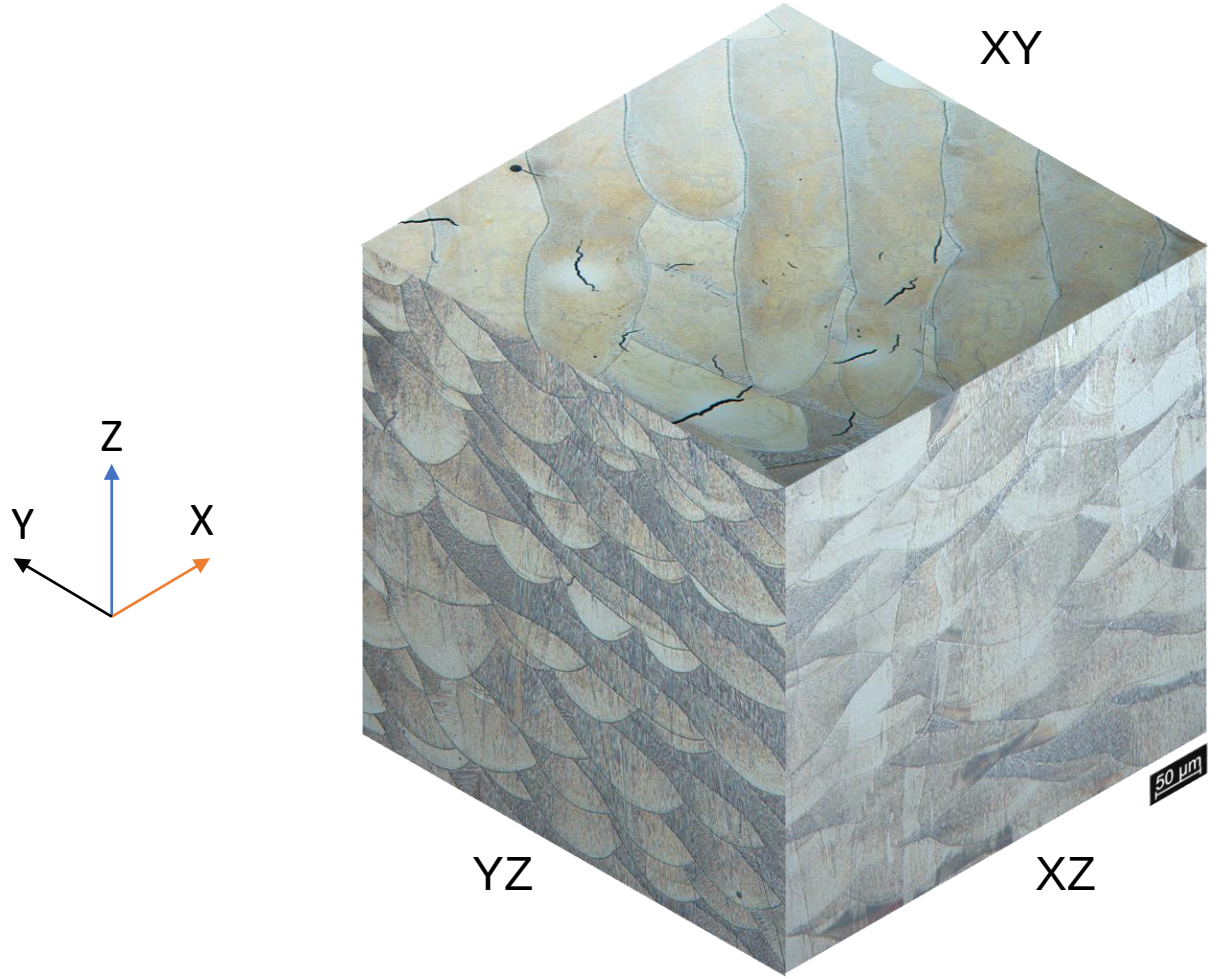
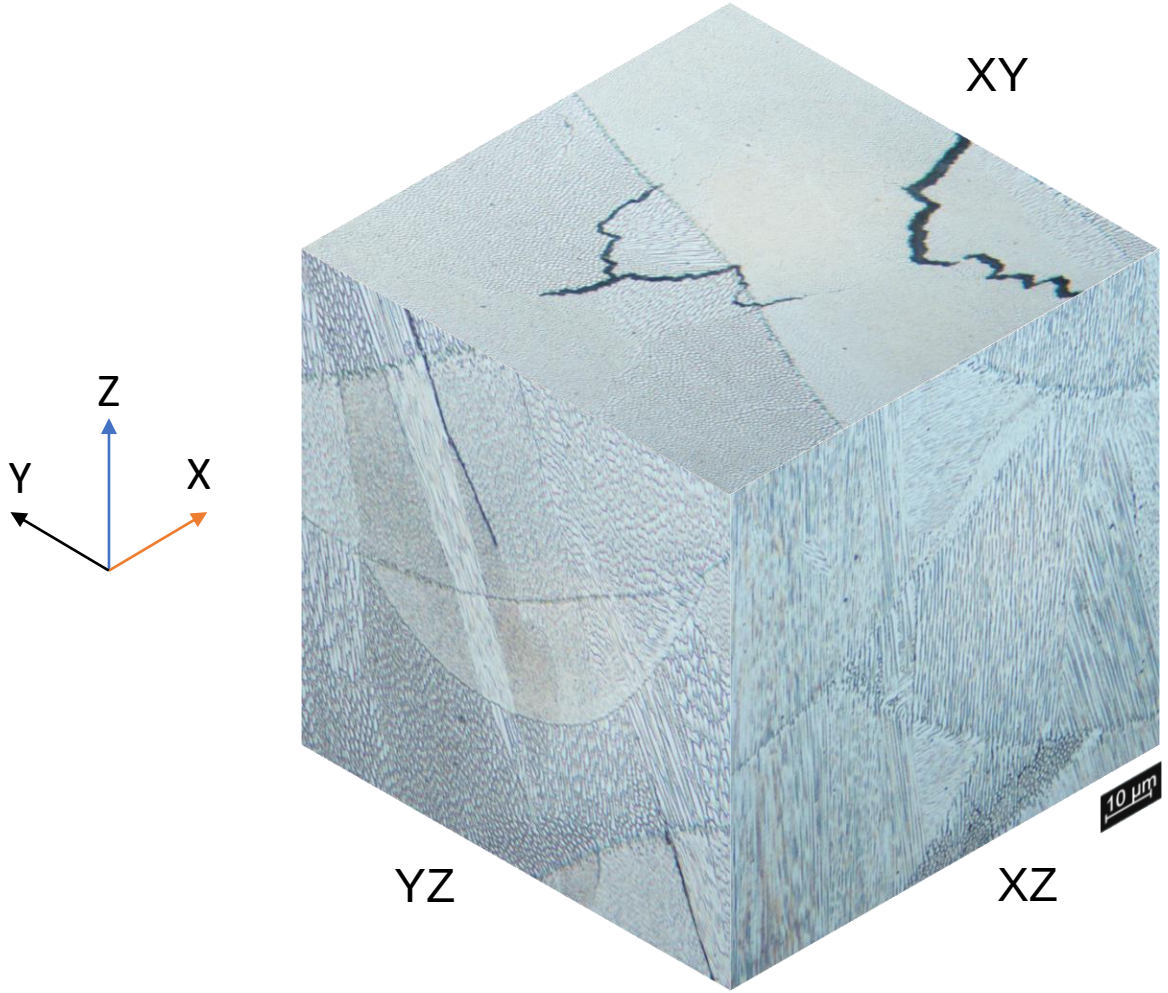


2.5mm HT

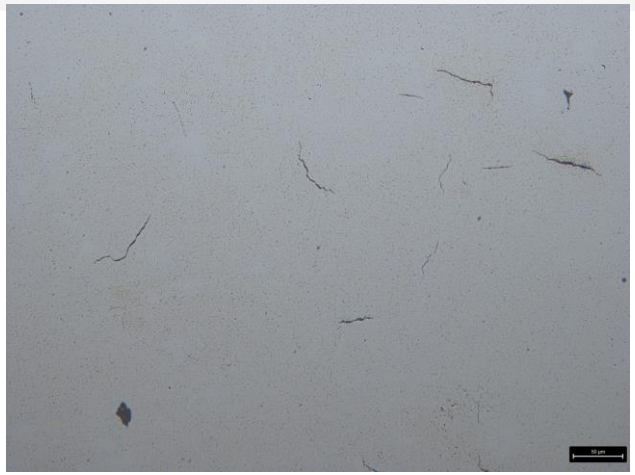
AB Macrostructure, 50x Build↑



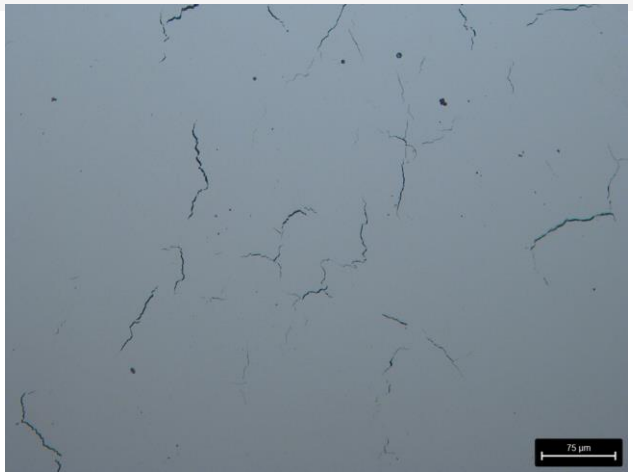
AB Microstructure, 1000x Build↑



Cracking As Built XY-Plane



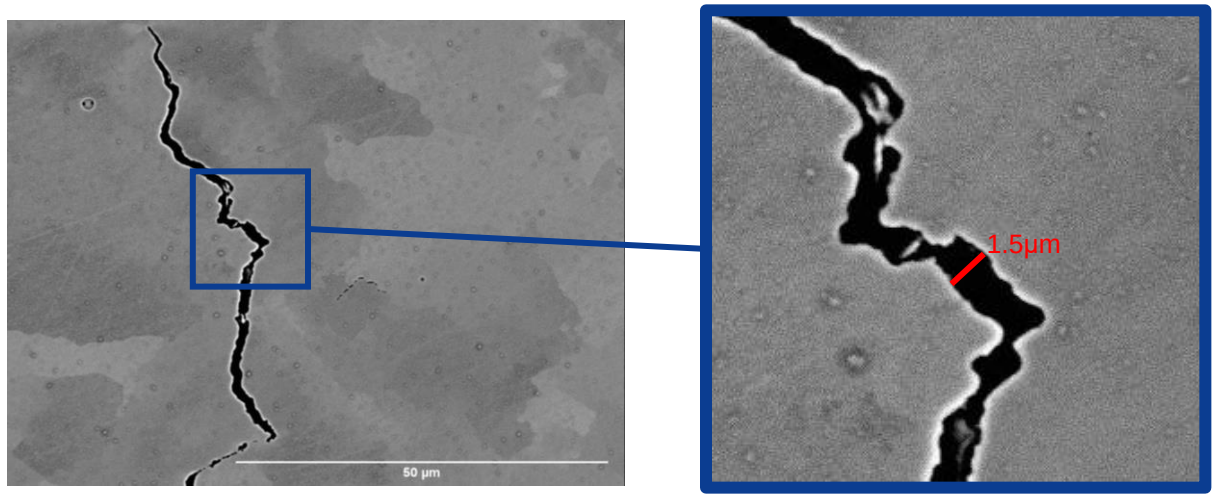
1mm AB



1.5mm AB

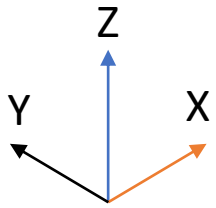
2mm AB

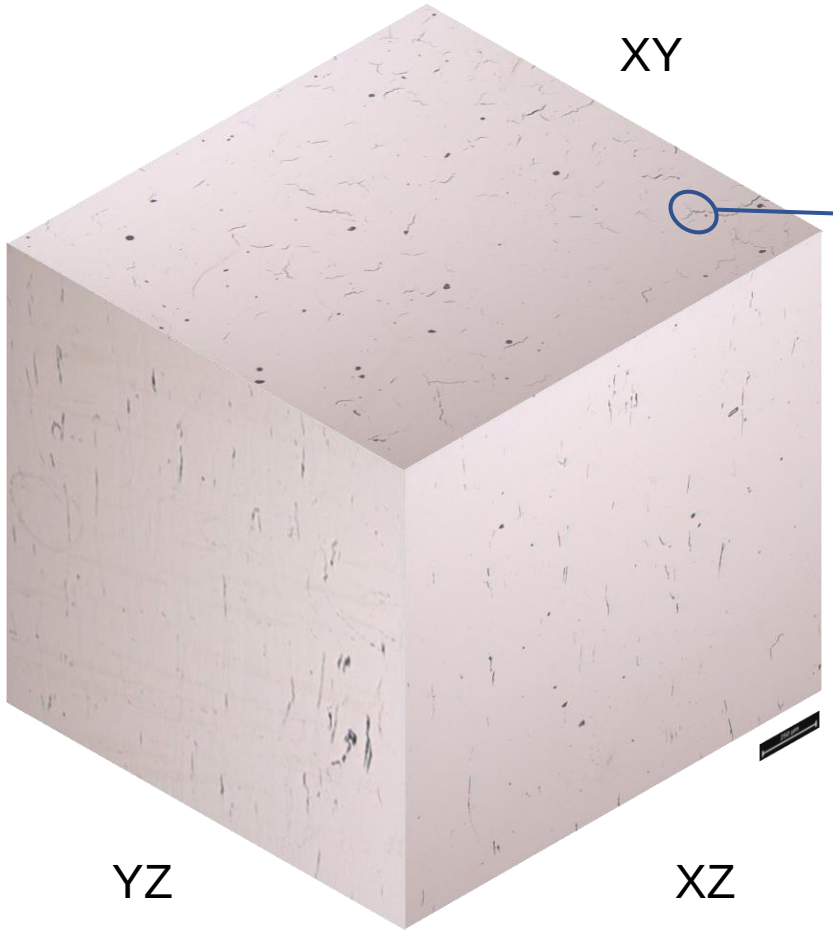
2.5mm AB

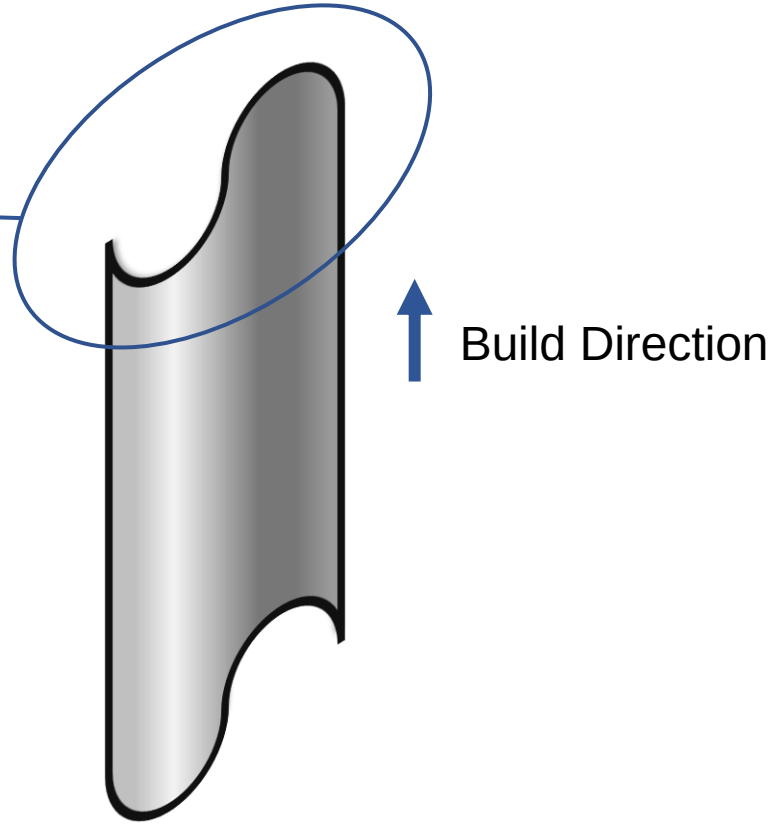


2mm AB XY 1000X

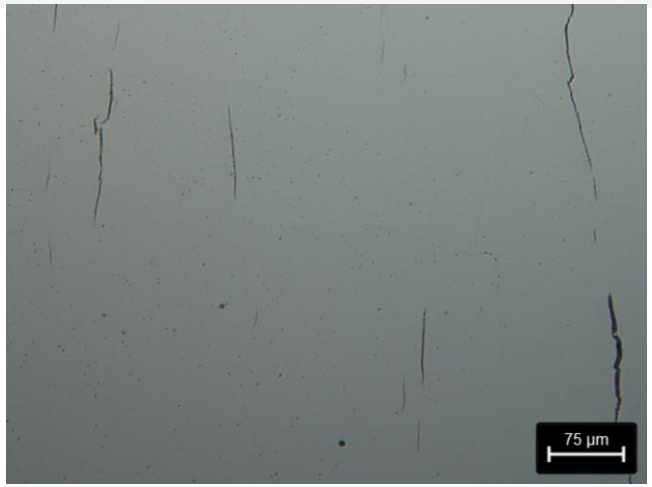
AB Cracking, 50x Build







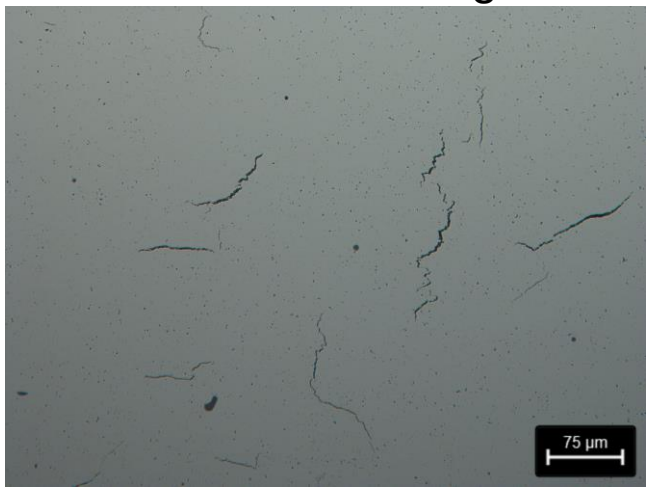
Cracking in Gauge and Grip Sections



1mm AB XZ Gauge

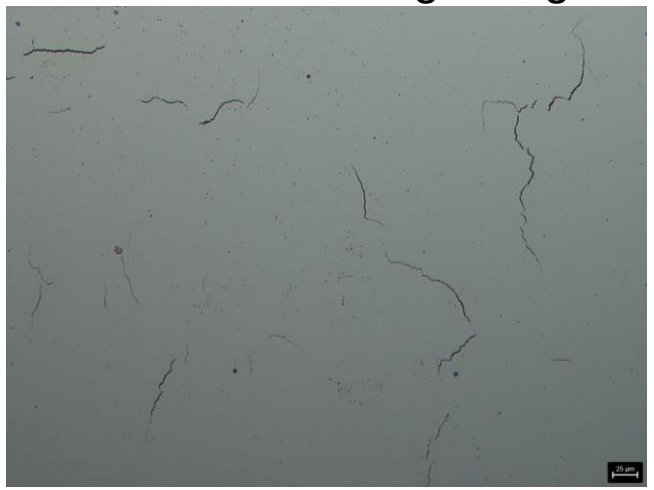
1mm AB XZ Long Grip

1mm AB XZ Long Gauge



1mm AB XY Gauge

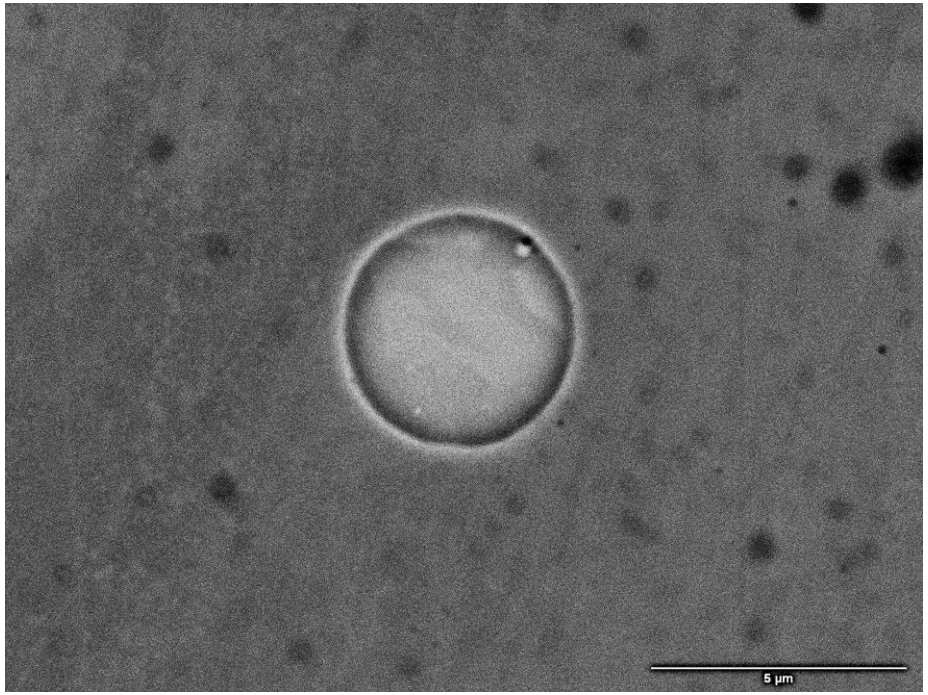
1mm AB XY Long Grip



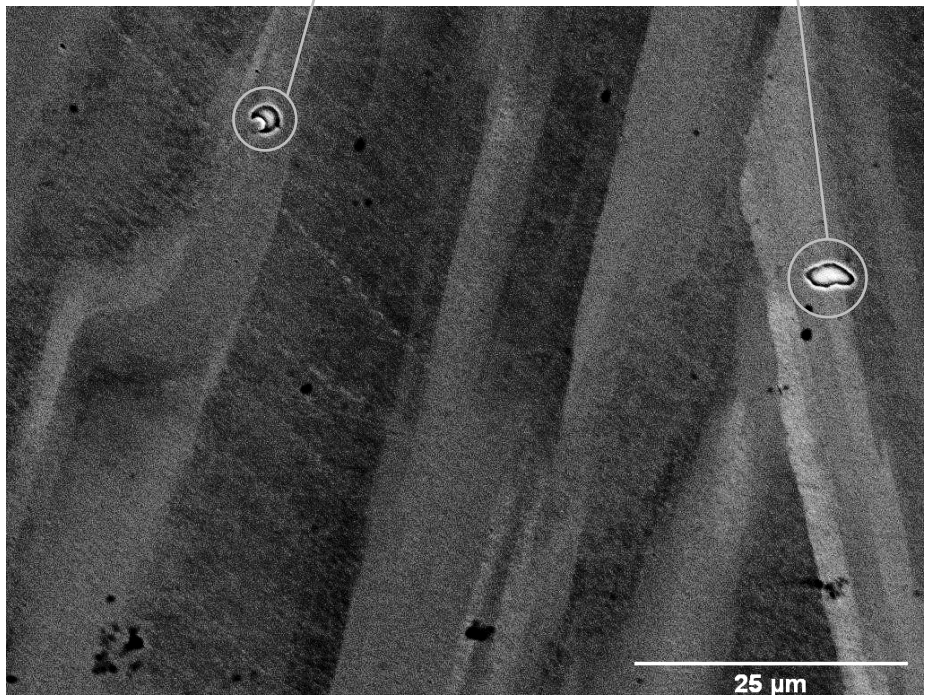
1mm AB XY Long Gauge

Cracks in the XZ and YZ planes tend to run along build direction while the cracks in the XY planes are distributed randomly. Most cracks are present in the XY planes.

Yttrium Oxides in GRX-810



1.5mm AB XZ

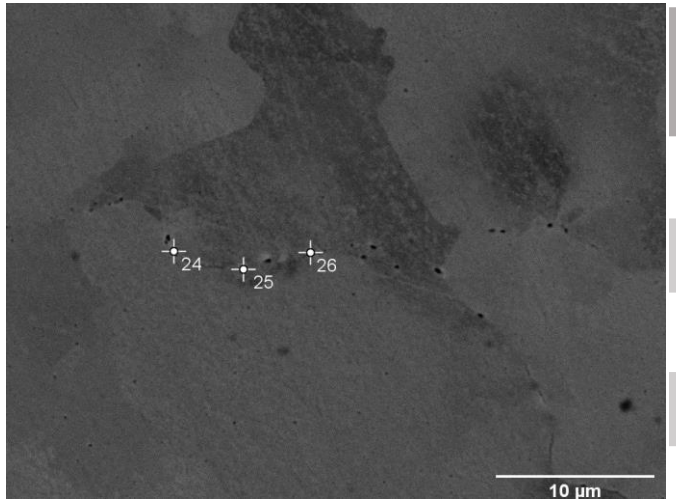


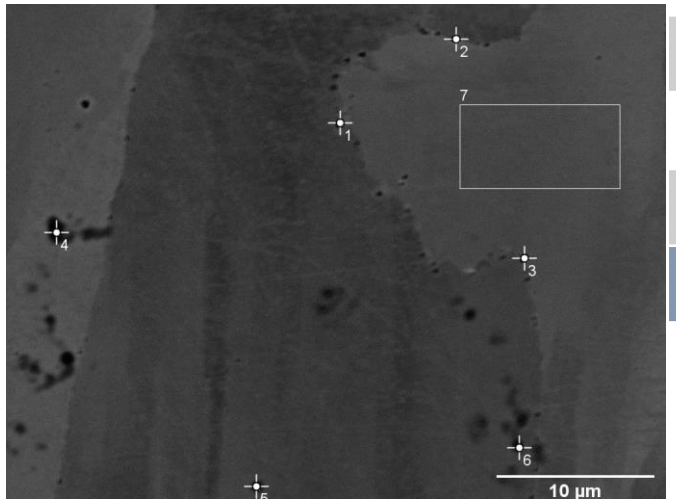
2.5mm HT YZ

Element (at. %)	12	14
B	21.27	-
O	36.42	50.36
Al	1.32	-
Ti	0.57	0.5
Cr	6.37	3.94
Co	4.75	2.24
N	4.45	2.53
Y	25.02	39.87
Br	-	0.57

Carbide Composition in GRX-810

1.5mm HT XZ 3000x





2.5mm HT YZ 3000x

Element (at. %)	1	2	3	4	5	6	7	24	25	26
O	-	-	-	12.51	-	-	-	-	-	-
Al	-	-	-	7.14	-	-	-	-	-	-
C	21.44	-	-	-	-	-	-	29.47	27.14	27.87
Ti	1.8	2.53	0.39	0.27	0.38	0.27	-	6.46	10.51	10.29
Cr	24.99	31.88	33.93	27.06	33.5	32.93	33.47	20	21.1	20.23
Co	24.03	29.97	31.82	25.68	32.25	31.23	31.8	19.21	18.57	17.94
Ni	23.58	29.22	32.33	25.38	32.18	30.59	31.51	18.96	18.07	17.93
Nb	1.94	3.02	-	0.43	-	0.36	-	4.02	3.77	3.88
C/Nb	11.05	-	-	-	-	-	-	7.33	7.20	7.18

- Location 4 could be alumina contaminant leftover from polishing
- Carbide formation along grain boundaries which is to be expected

Microhardness of GRX-810

- Vickers microhardness was performed on samples of GRX-810
  - No statistically significant effect of thickness or heat treatment on hardness values

Specimen Thickness	Plane	Heat Treatment	xxxHV0.1	σ	Specimen Thickness	Plane	Heat Treatment	xxxHV0.1	σ
1 mm	XY	As-Built	268±15.9	21.8	2.5 mm	XY	As-Built	258±10.2	15.6
		HIP	248±3.4	9.7			HIP	260±14.0	21.4
	XZ	As-Built	282±9.8	13.0		XZ	As-Built	272±4.2	16.9
		HIP	240±6.0	8.4			HIP	264±3.3	14.4
	YZ	As-Built	278±2.9	12.5		YZ	As-Built	278±17.8	27.2
		HIP	255±2.8	12.3			HIP	262±10.1	16.2

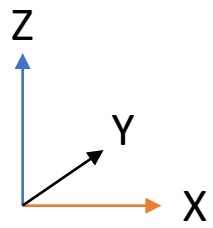
Summary

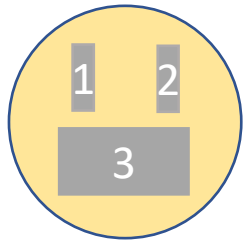
- Procedures were developed for GRX-810 to cut, grind, and polish material for metallographic presentation
- Successful etching procedure was developed for GRX-810
- Significant solidification cracking was found through all specimens
  - Could be a consequence of the build and cooling parameters
- Microhardness of GRX-810 was determined

Al 6061 Sample Overview

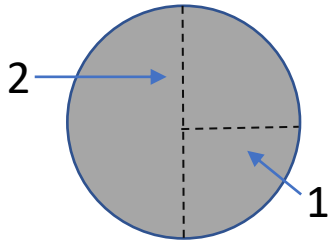
- Weld areas of an additively manufactured nozzle
  - This was broken up into four separate specimens marked 1 through 4

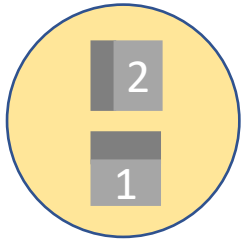
- Anodized additively manufactured sections
  - Broken up into three separate specimens
    - #1 corresponds to ZY plane
    - #2 corresponds to XY plane
    - #3 corresponds to ZX plane



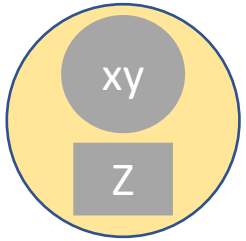


- Explosive Bound Coupon
  - Two Al 6061-Ni bound specimens



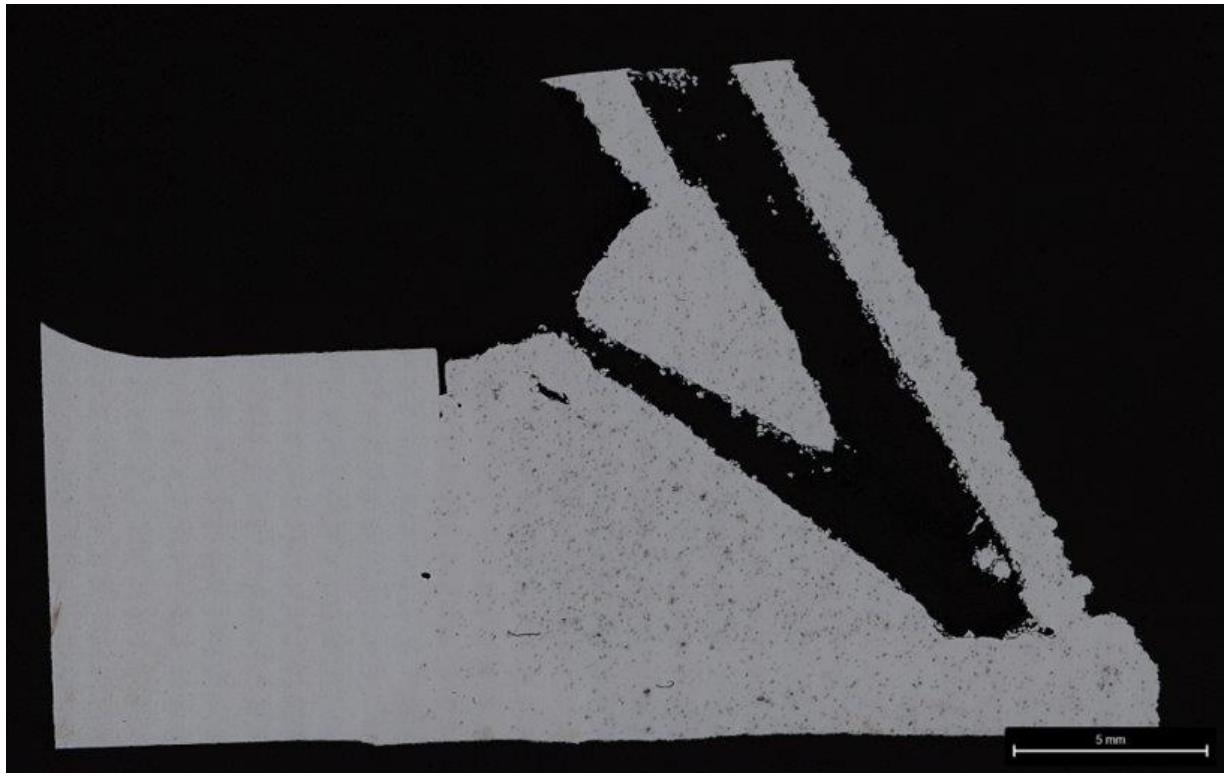


- E-316-JJ-4 Billet
  - Top profile is XY plane
  - Side profile is Z plane



Weld Etching Results

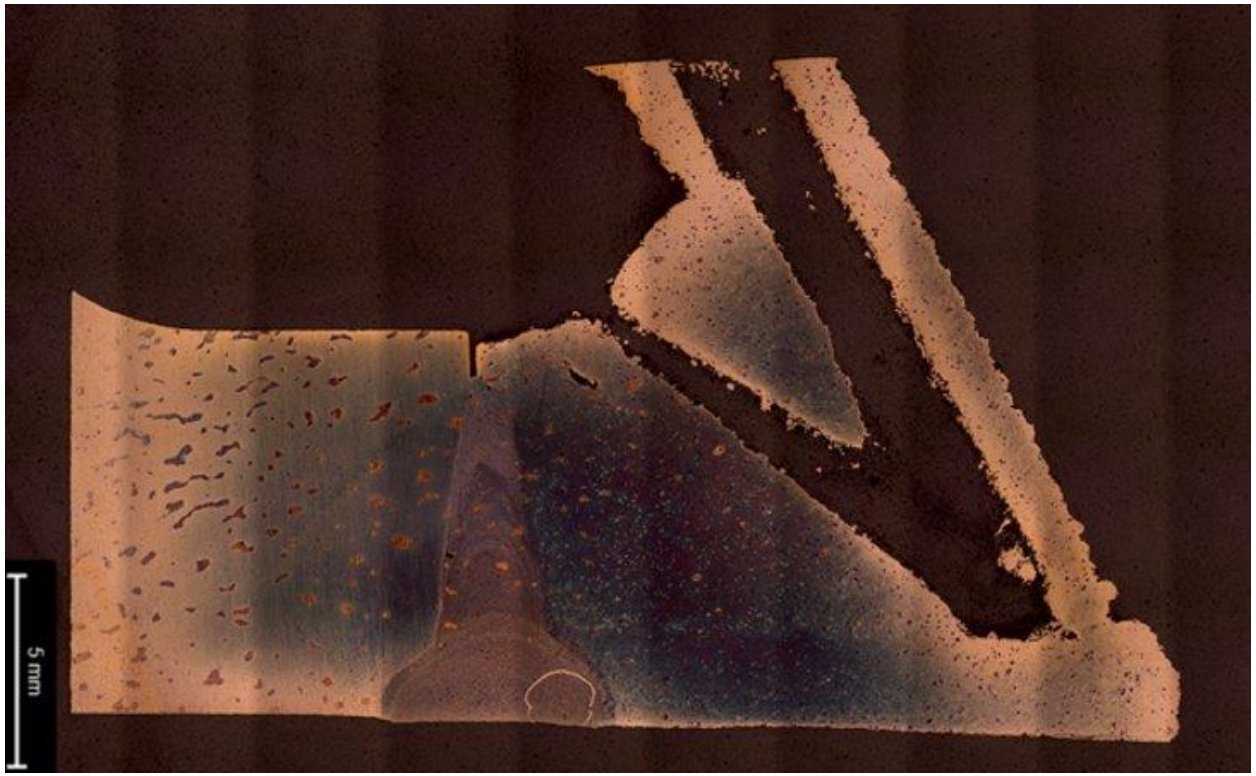
Unetched Tilescan of Weld 1



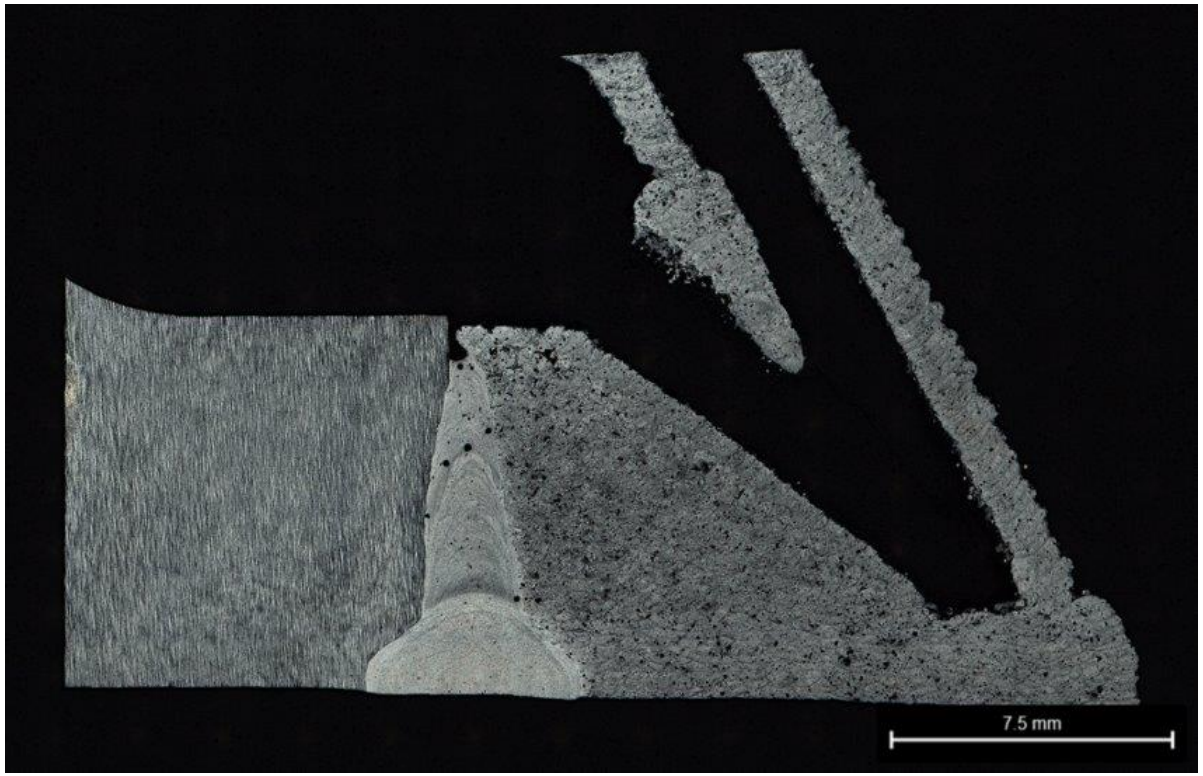
Keller's Etch of Weld 1

Weld Etching Results Cont.

Weck's etch of Weld 1



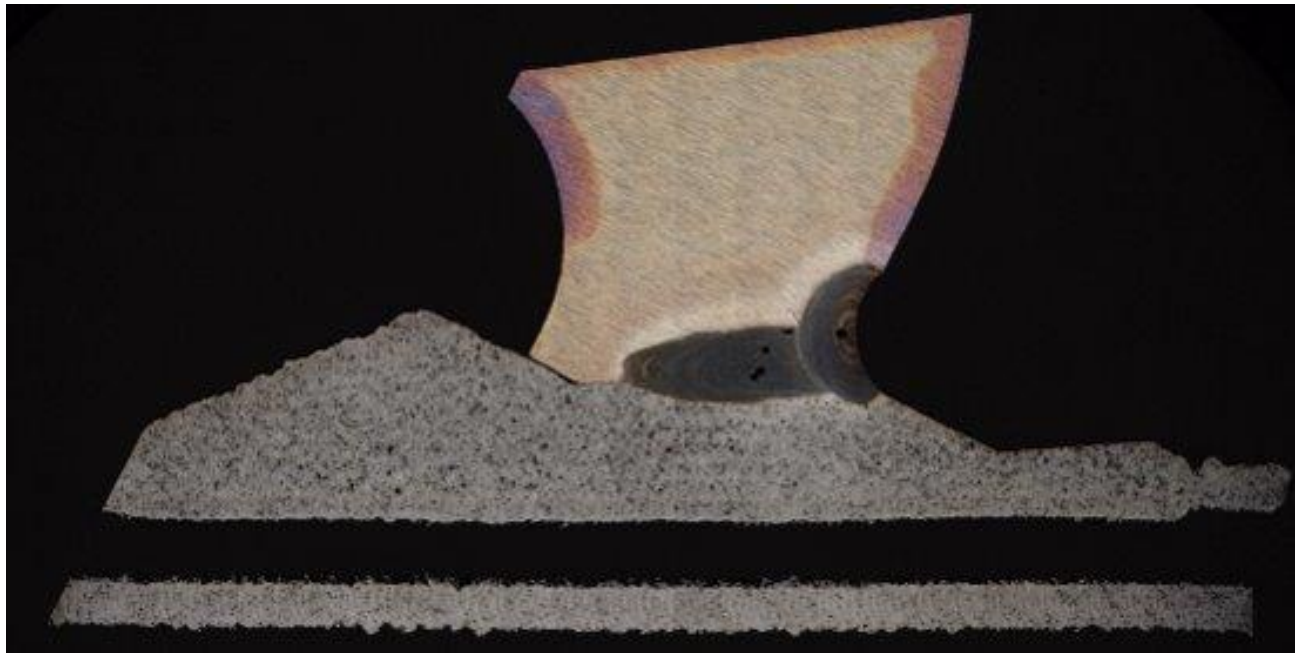
Methyl Regia etch of Weld 1



Weld Etching Results Cont.

NaOH + NaF etch of Weld 1

NaOH + NaF etch of Weld 2

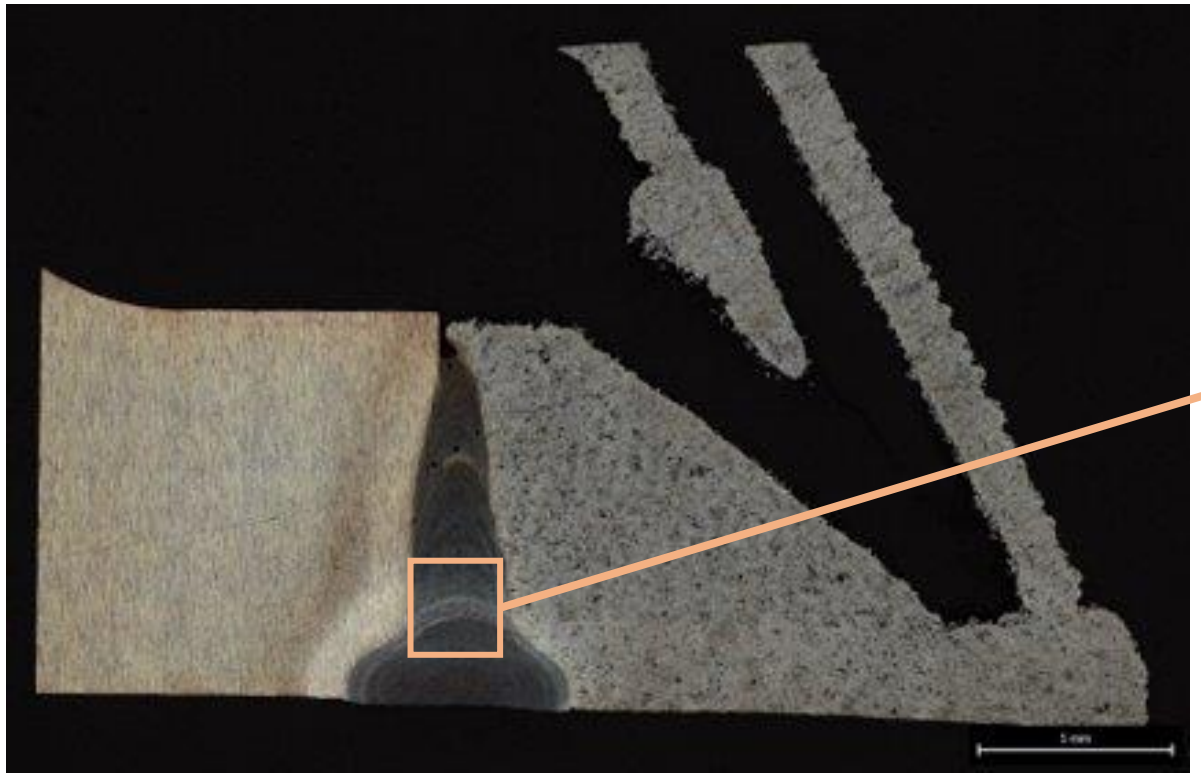


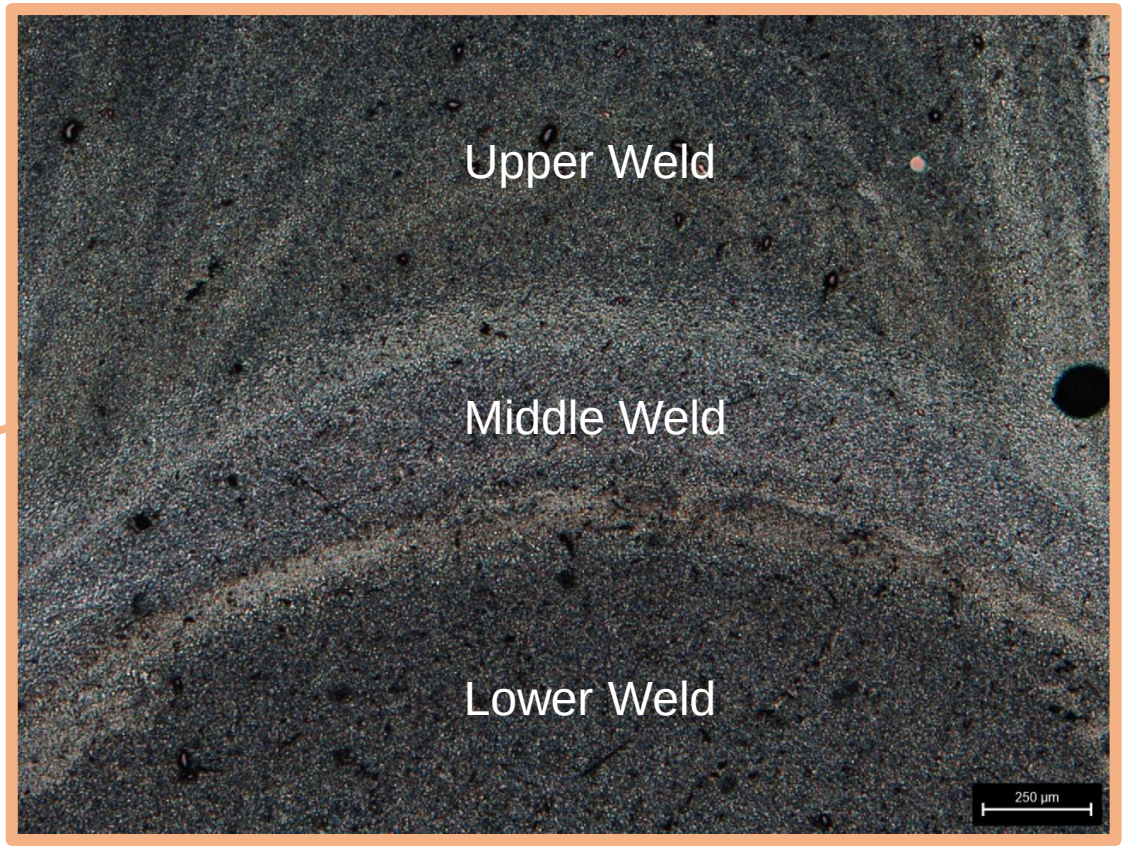
Porosity of Welds

- Porosity was calculated in ImageJ using circular drawing tools after calibrating scale to burned in scale bars

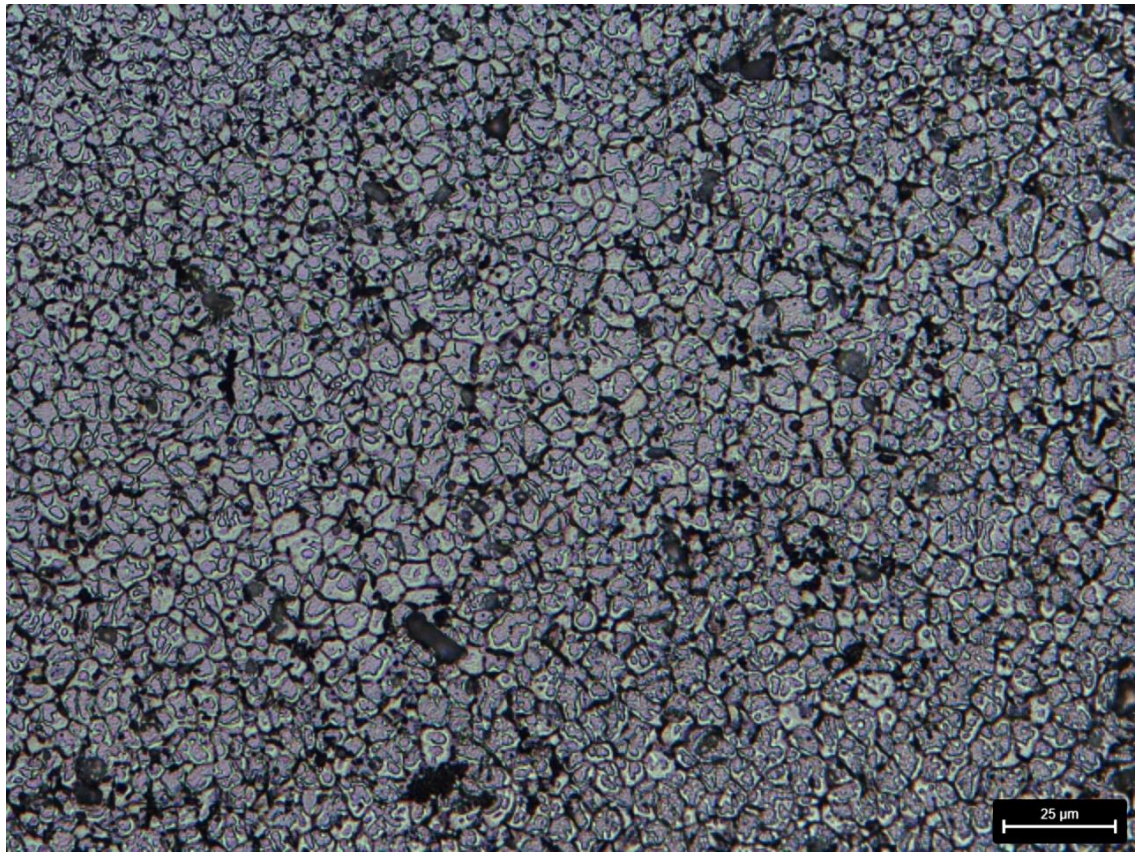
Weld Number	Weld Area (mm^2)	Void Area (mm^2)	Porosity (%)	# Voids < 0.15 mm^2	# Voids > 0.15 mm^2
1	24.183	0.406	1.68	7	5
2	19.766	0.433	2.19	6	0
3	22.618	0.425	1.88	13	0
4	20.194	0.769	3.81	11	1

Weld Microstructure

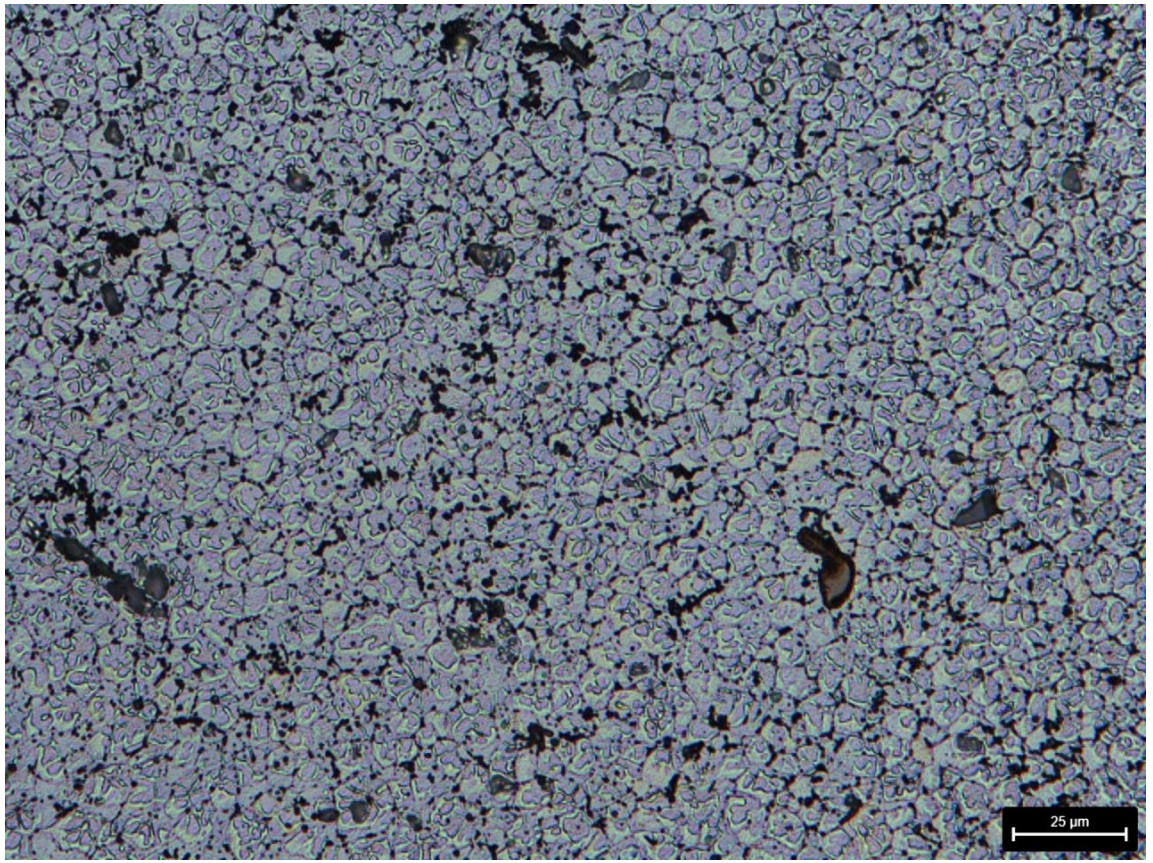




Weld Microstructure Cont.

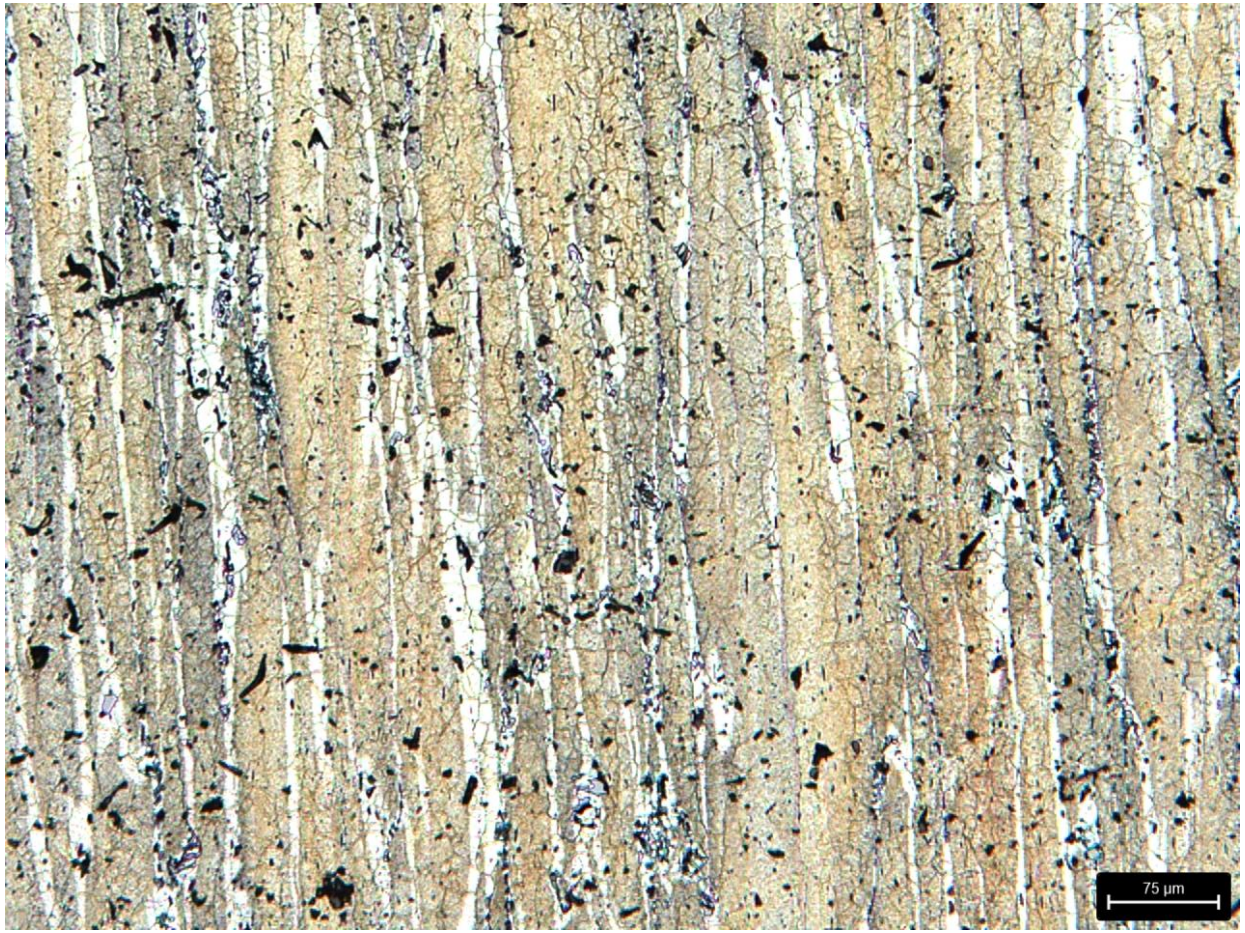


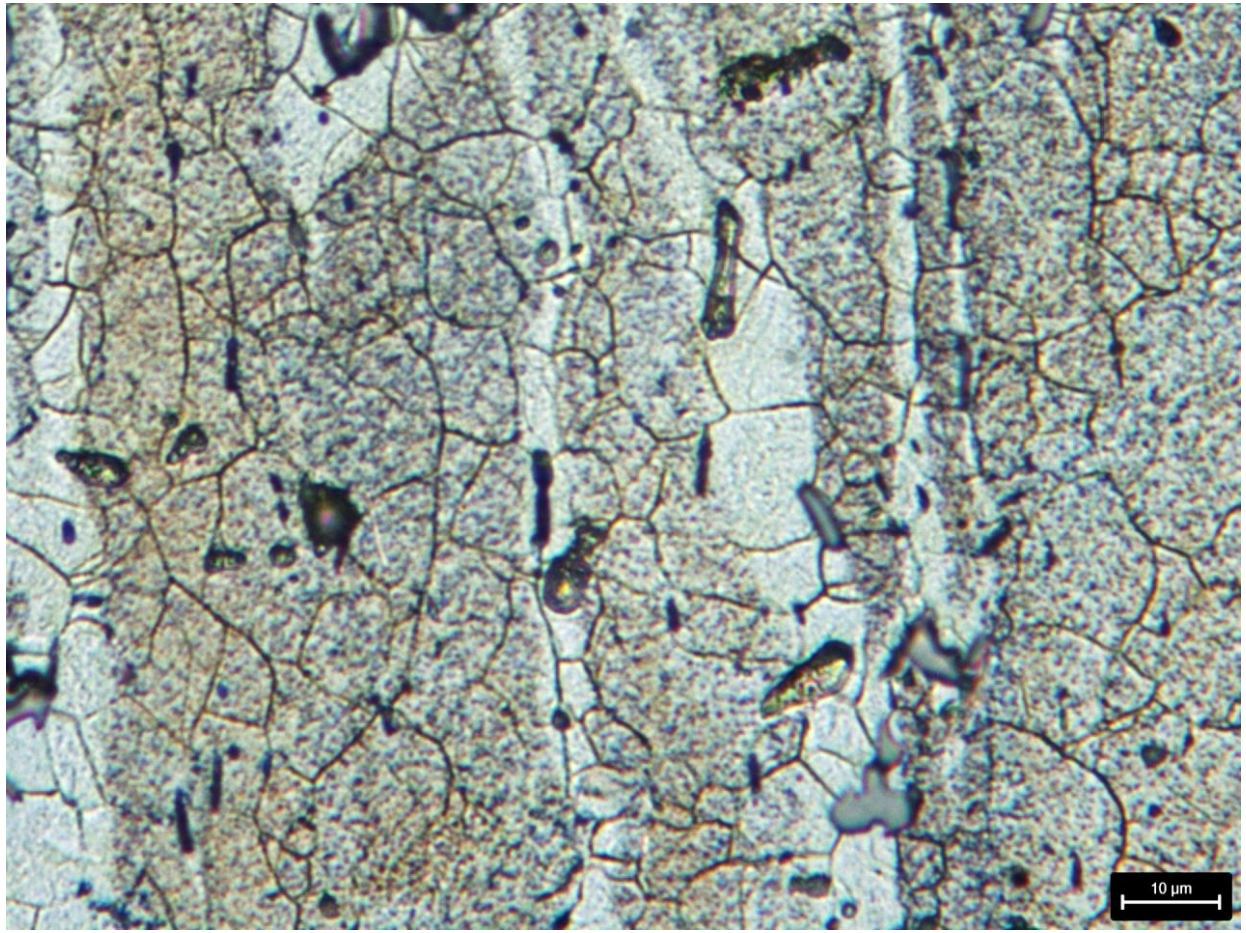
Upper Weld



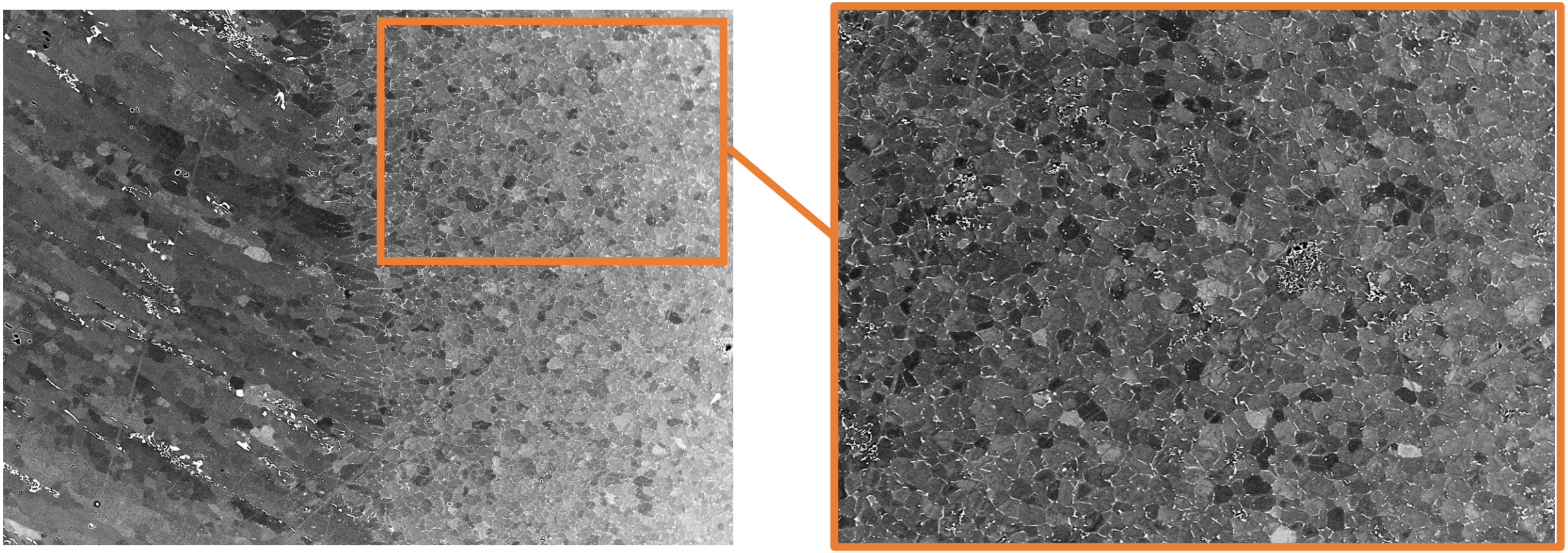
Middle Weld

Nozzle Wrought Microsection





SEM Weld Microstructure



250 µm

100 µm

Haynes 214 Sample Overview

- Tensile specimens of Haynes 214 were made to 4 different thickness using L-PDF
  - Thicknesses were 1, 1.5, 2, 2.5 mm
- Half were mounted as-built, while the other half were mounted after heat treatment
- Of the heat-treated samples, 1 and 2.5mm samples were subjected to an aging step
- SEM imaging was performed to see effects of thickness and heat treatment on precipitate and carbide formation
- Haynes 214 Composition (wt. %)

Ni	Cr	Al	Fe	Co	Mn	Mo	Ti	W	Nb	Si	Zr	C	B	Y
bal	16	4.5	3	2 max	0.5 max	0.5 max	0.5 max	0.5 max	0.15 max	0.2 max	0.1 max	0.04	0.01 max	0.01

Grinding and Polishing Procedure for Haynes 214

Grinding Procedure						Polishing Procedure							
Grit	Time	Force	Plate Speed	Holder Speed	Rotation Type	Plate	Solution	Time	Force	Plate Speed	Holder Speed	Rotation Type	Dose
120	1:00	20 N	300 rpm	150 rpm	Co-rotation	MD-Dac	DiaDuo-2 3 µm	5:00	20 N	150 rpm	150 rpm	Co-rotation	15
220	1:00	20 N	300 rpm	150 rpm	Co-rotation	MD-Mol	DiaDuo-2 1 µm	5:00	20 N	150 rpm	150 rpm	Co-rotation	15
500	1:00	20 N	300 rpm	150 rpm	Co-rotation	MD-Nap	OPA	5:00	15 N	150 rpm	150 rpm	Counter-rotation	Manual
1200	2:00	20 N	300 rpm	150 rpm	Co-rotation								
2000	1:00	20 N	300 rpm	150 rpm	Co-rotation								
4000	1:00	20 N	300 rpm	150 rpm	Co-rotation								

Etching of Haynes 214

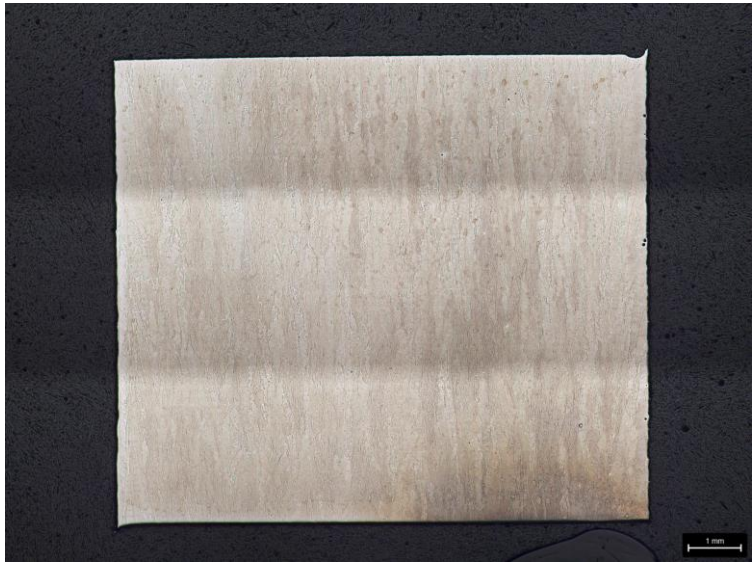
- Etch with waterless Kallings at room temperature
  - 50s for As Built samples
  - 2:30 for Heat Treated samples
  - Worked well to reveal grains of XZ sections, yet XY and YZ sections burned easily, especially for the As Built

As Built – XZ Plane

1mm

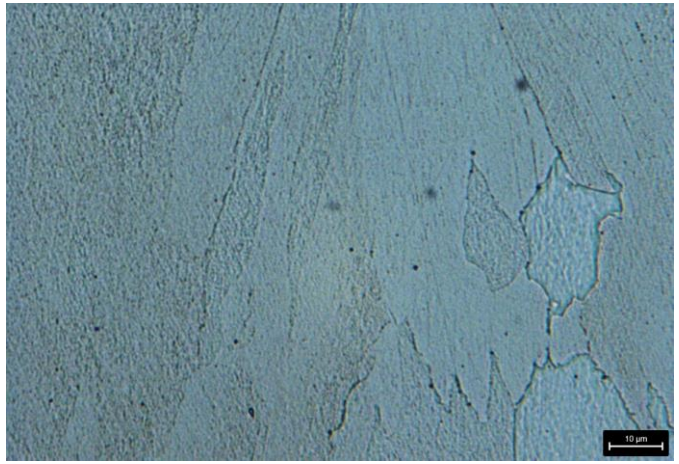
1.5mm

2mm



2.5mm

As Built – XZ Plane Cont.



1mm

1.5mm

2mm

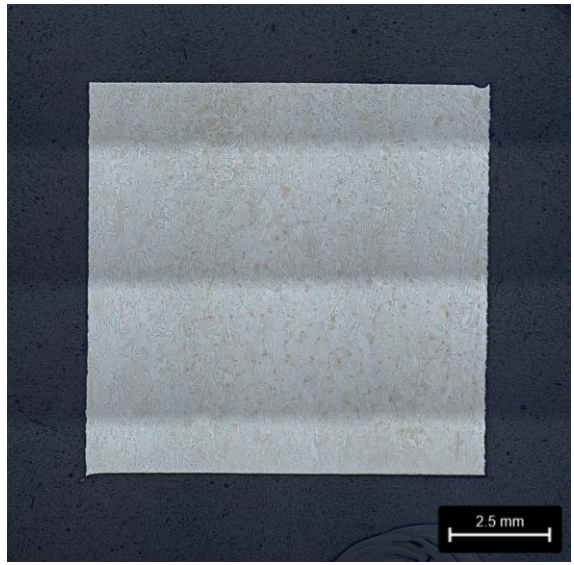
2.5mm

Heat Treated – XZ Plane

1mm

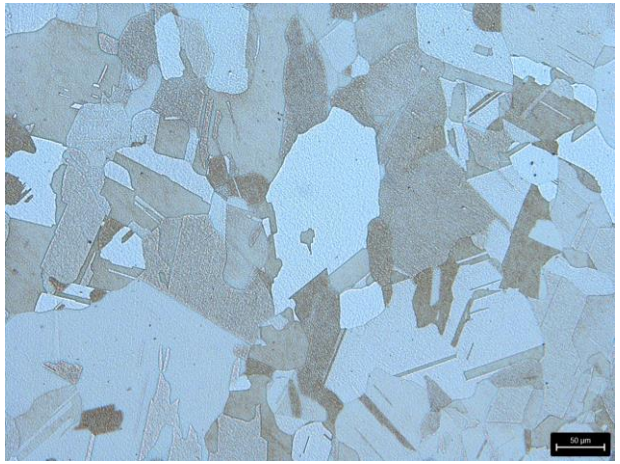
1.5mm

2mm



2.5mm

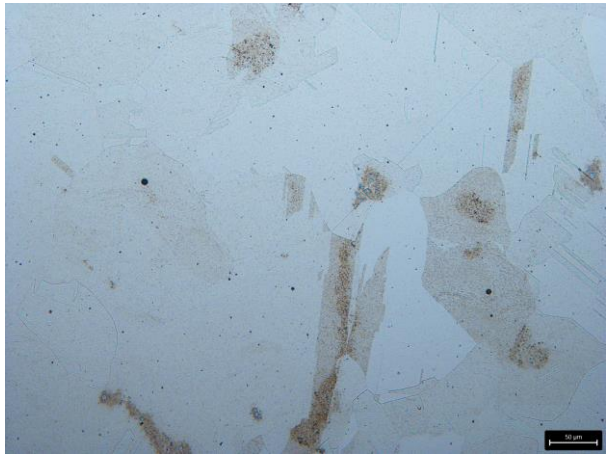
Heat Treated – XZ Plane Cont.



1mm

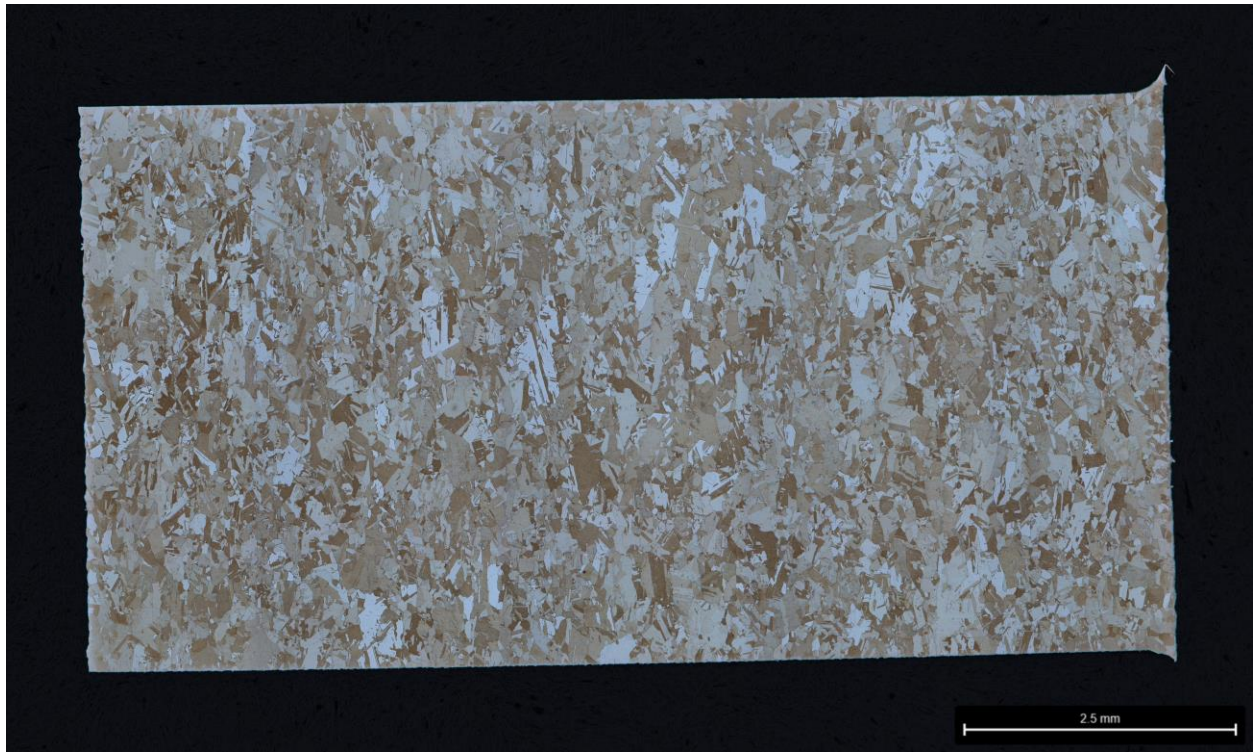
1.5mm

2mm



2.5mm

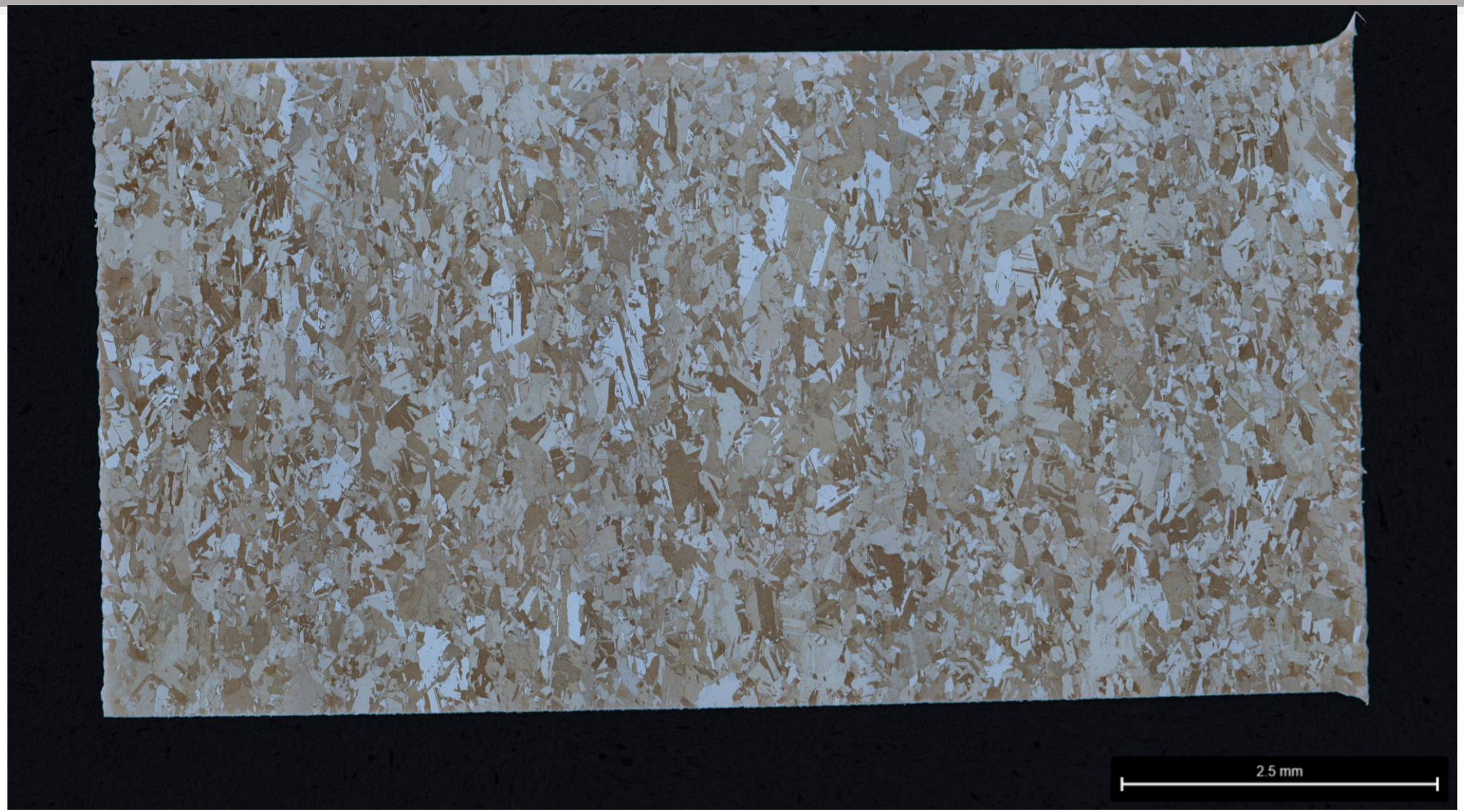
Heat Treated First Run – XZ Plane



1mm

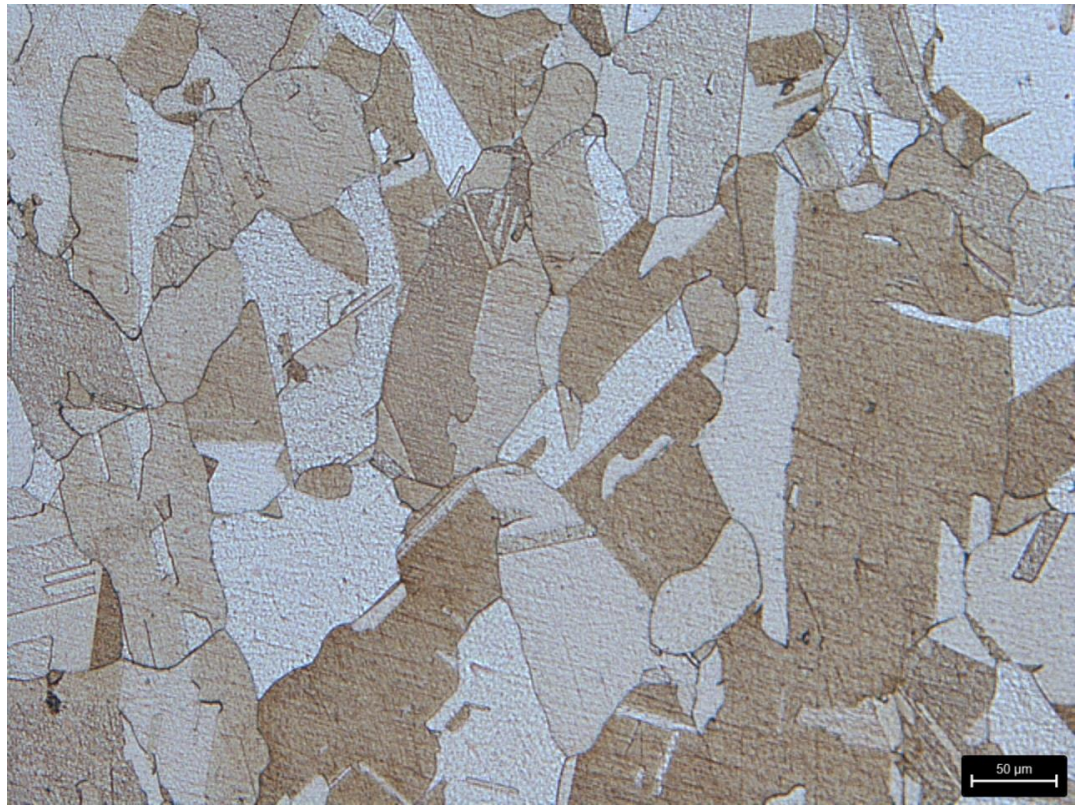
2.5mm

Heat Treated First Run – XZ Plane

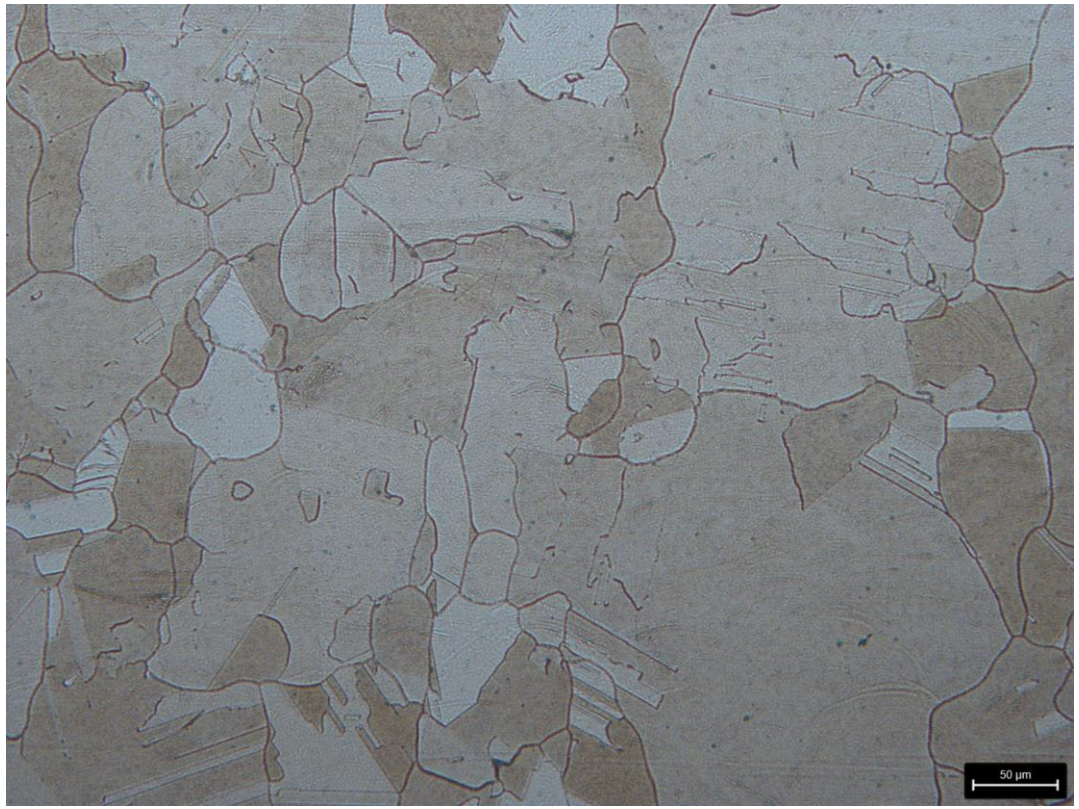


1mm

Heat Treated First Run XZ-Plane Cont.

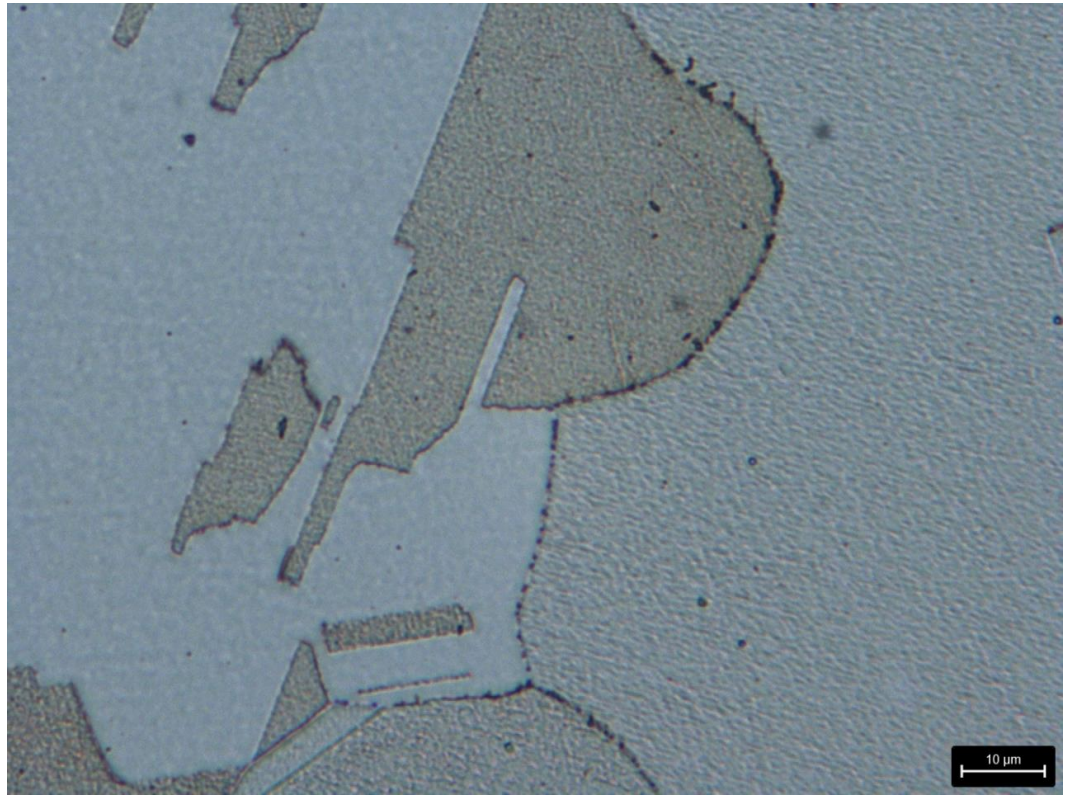


1mm

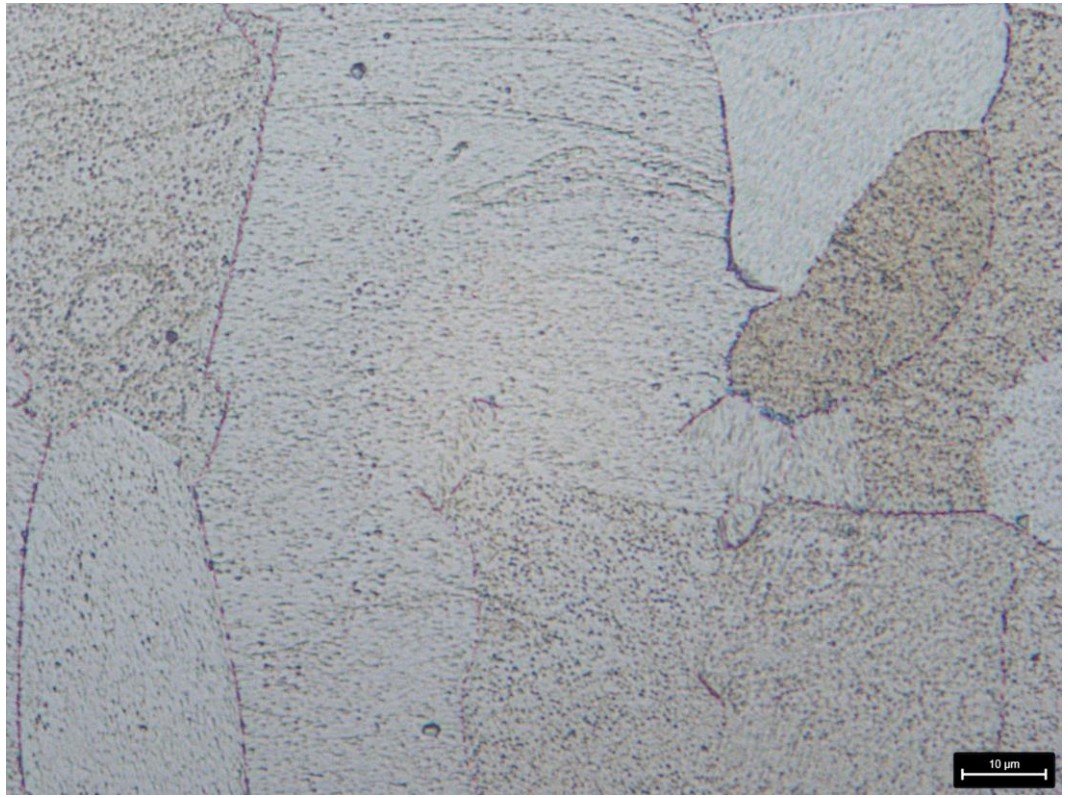


2.5mm

Heat Treated First Run XZ-Plane Cont.

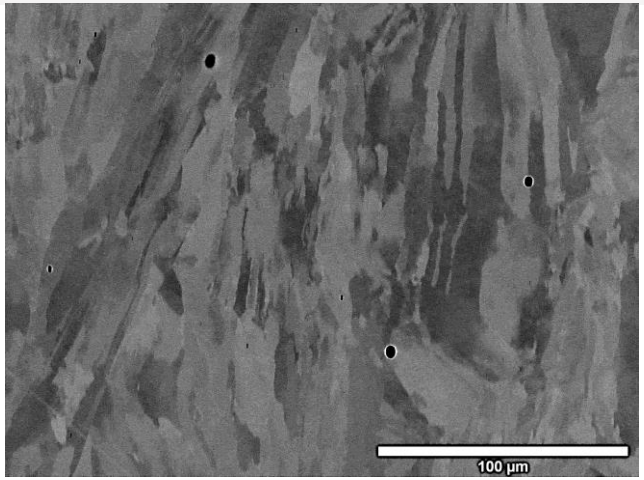


1mm

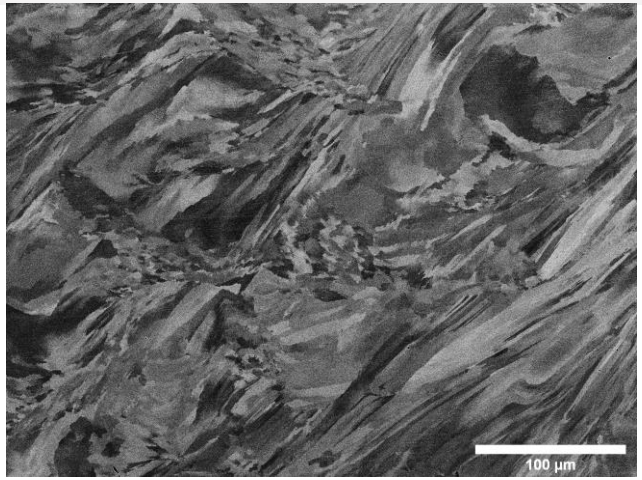


2.5mm

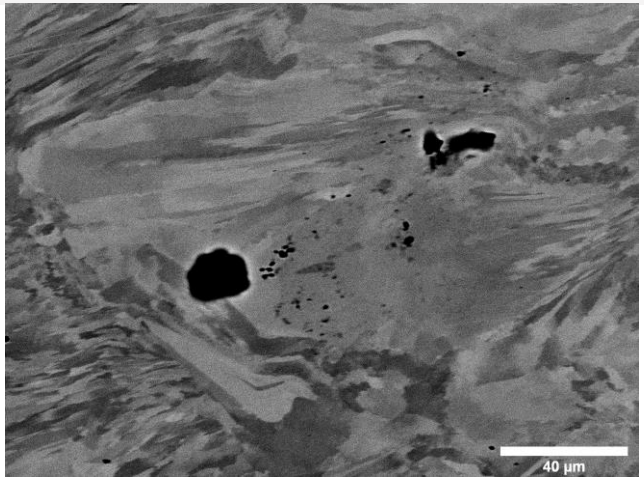
SEM As Built and Heat Treated XZ-Plane



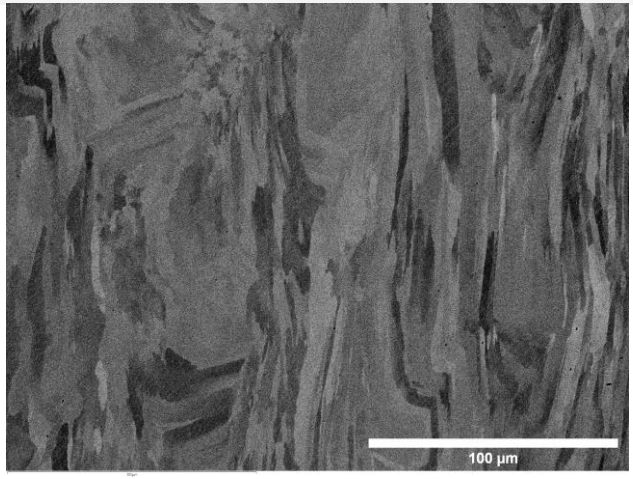
1mm AB



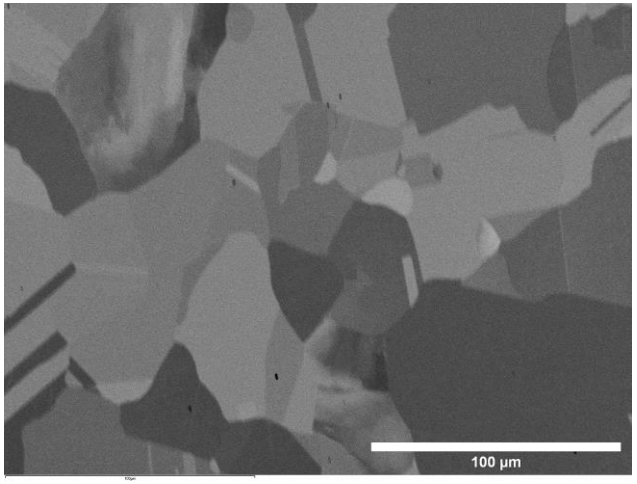
1.5mm AB



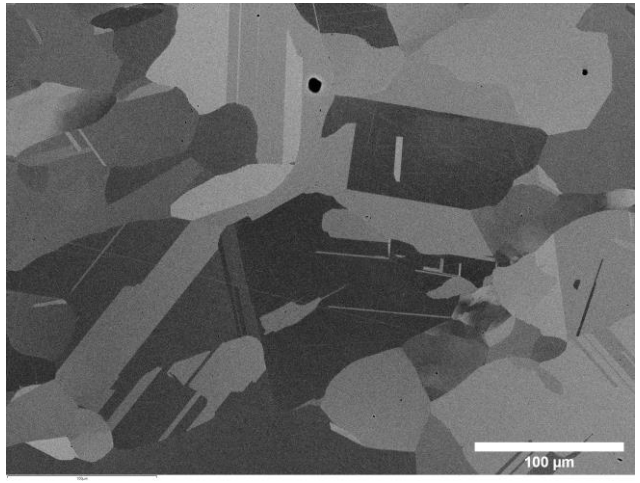
2mm AB



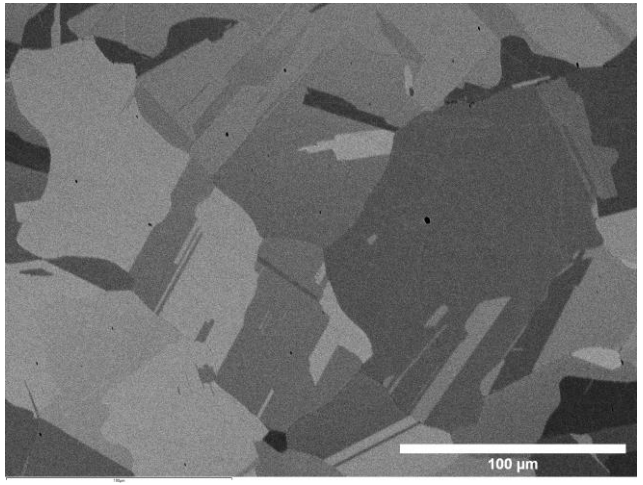
2.5mm AB



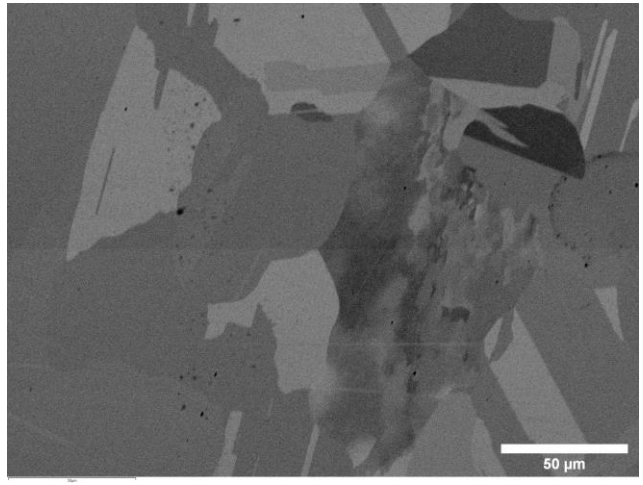
1mm HT



1.5mm HT

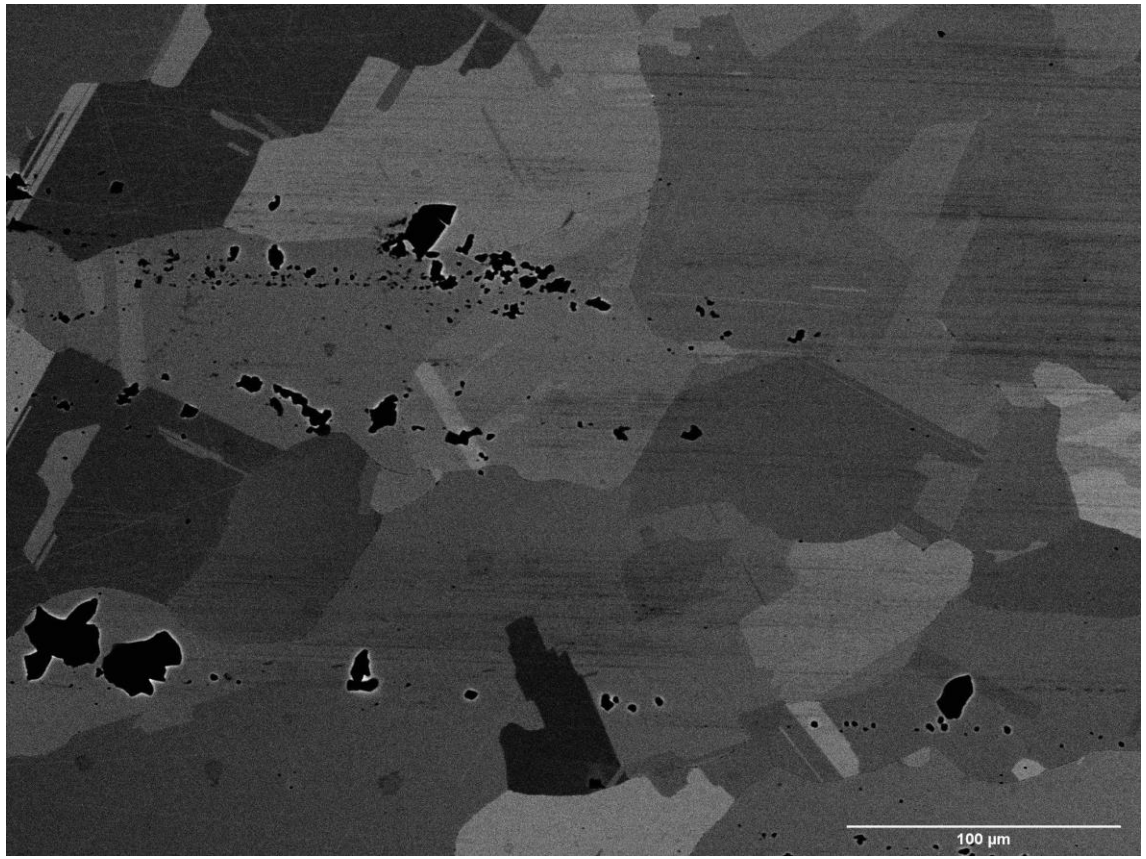


2mm HT

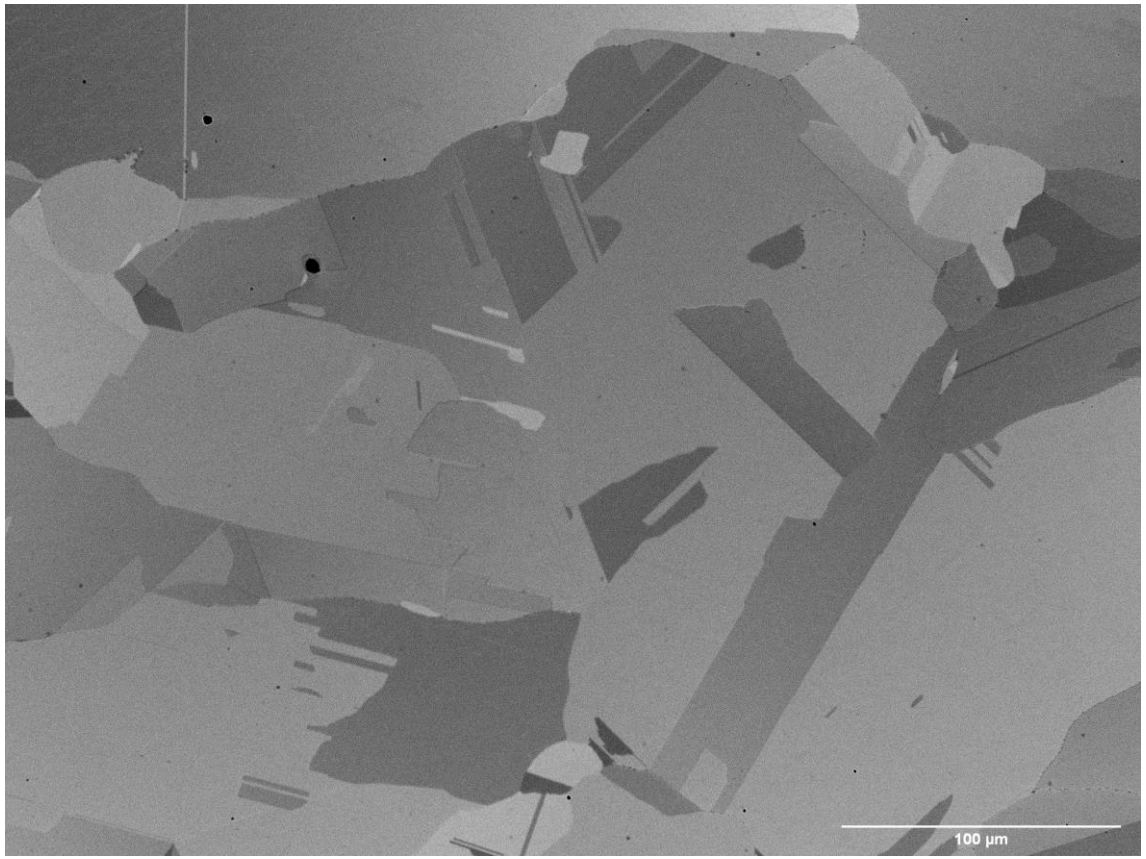


2.5mm HT

SEM Heat Treated First Run XZ-Plane



1mm

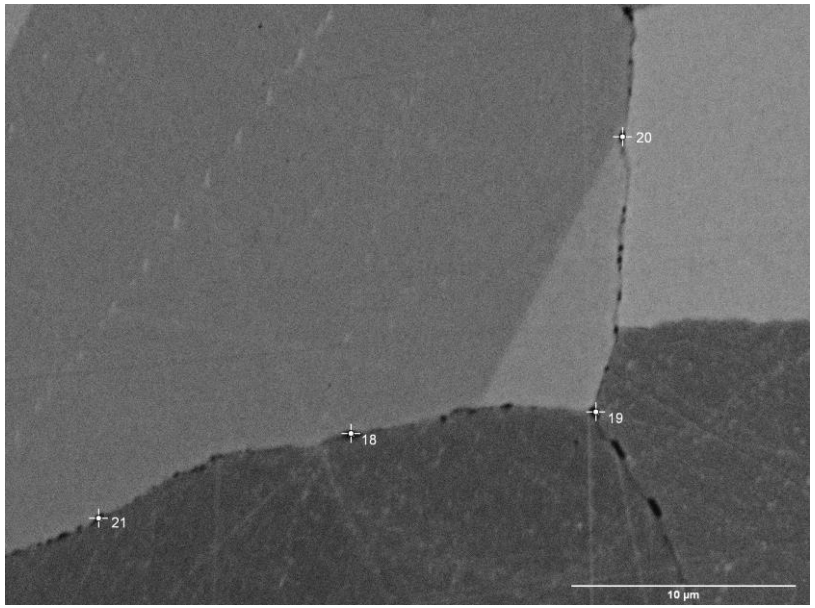


2.5mm

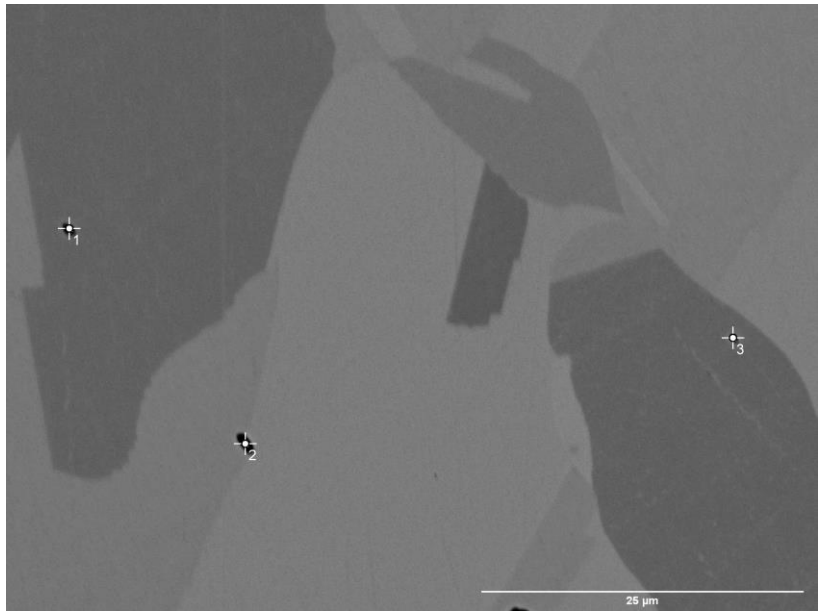
SEM Carbide Formation

Carbides Present

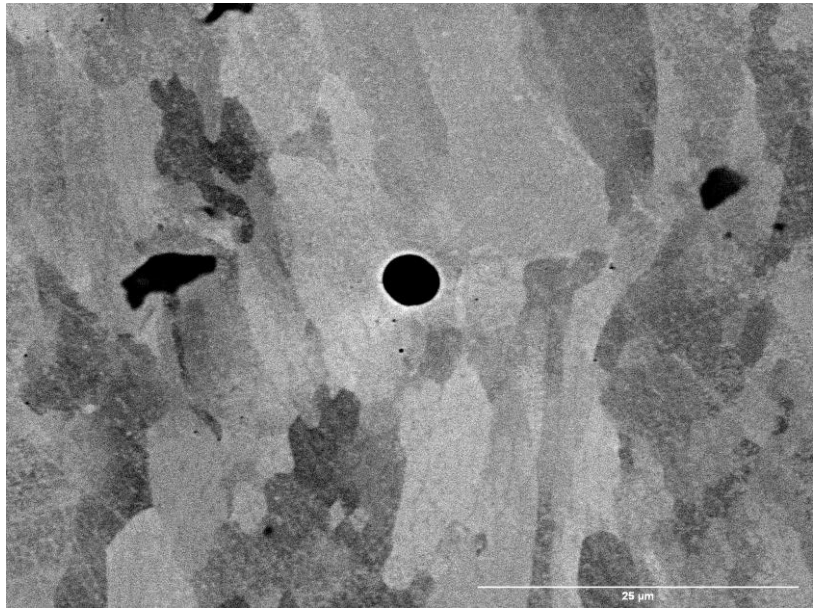
Carbides Not Present



1mm HT FR



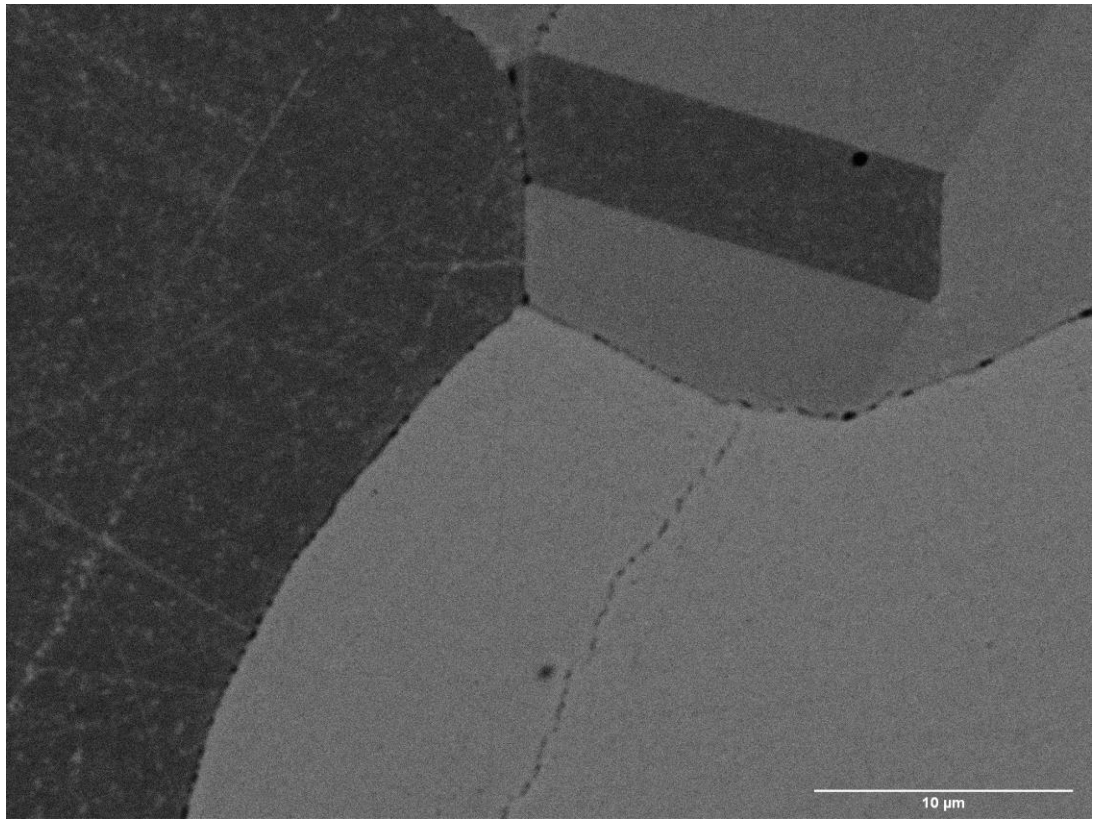
1mm HT



1mm AB

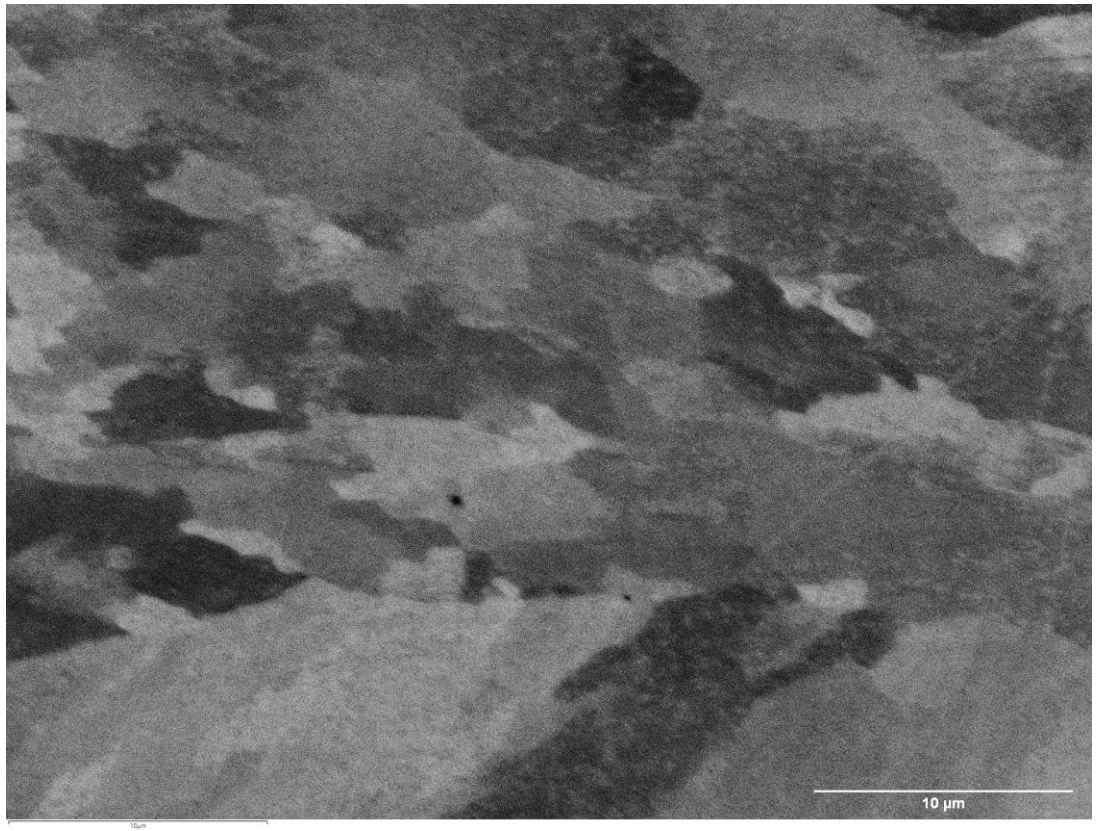
SEM Carbide Formation Cont.

Carbides Present



1.5mm HT

Carbides Not Present

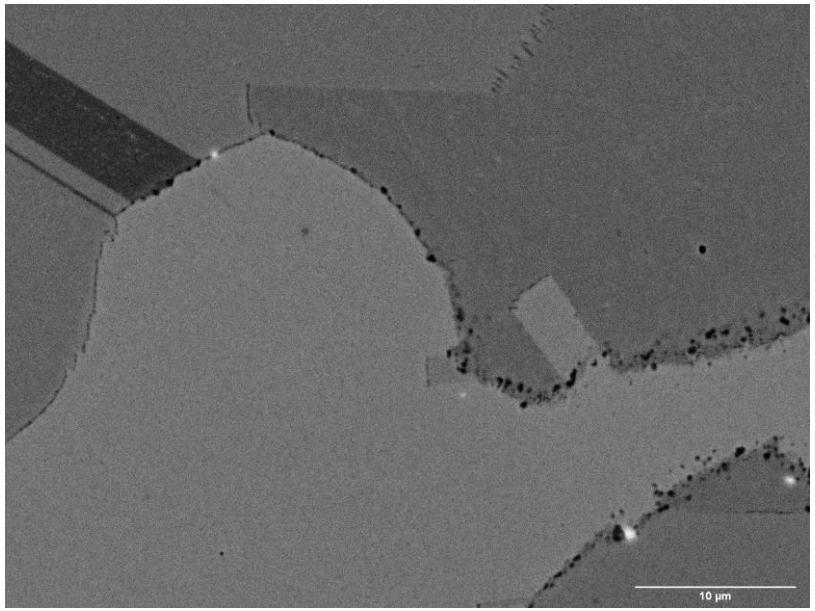


1.5mm AB

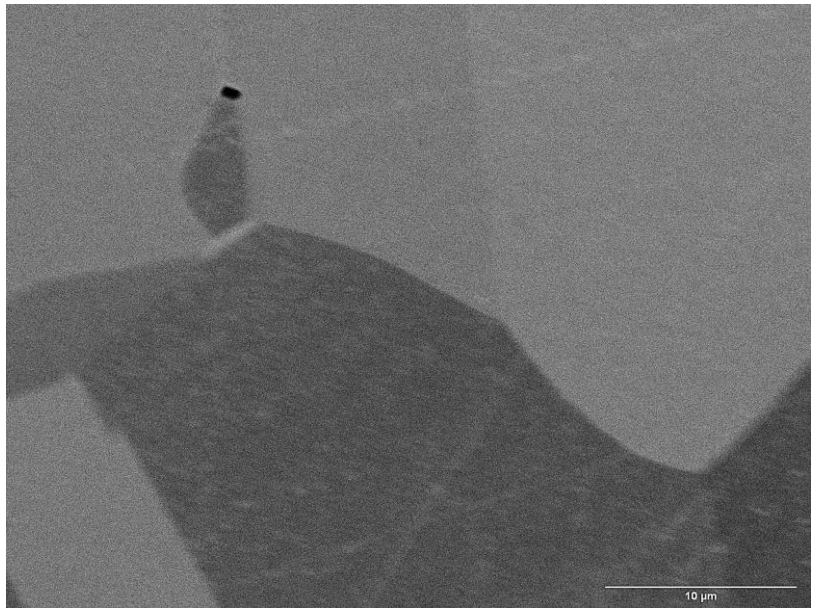
SEM Carbide Formation Cont.

Carbides Present

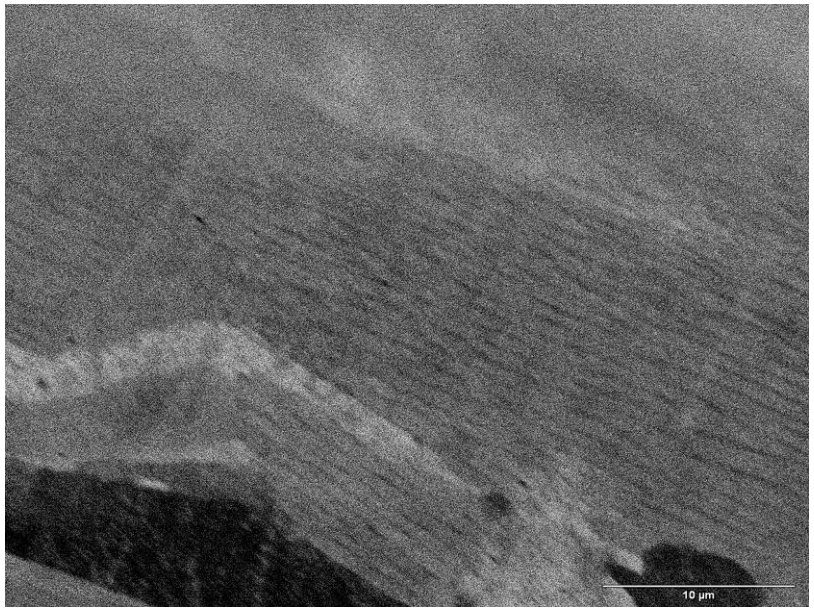
Carbides Not Present



2.5mm HT FR



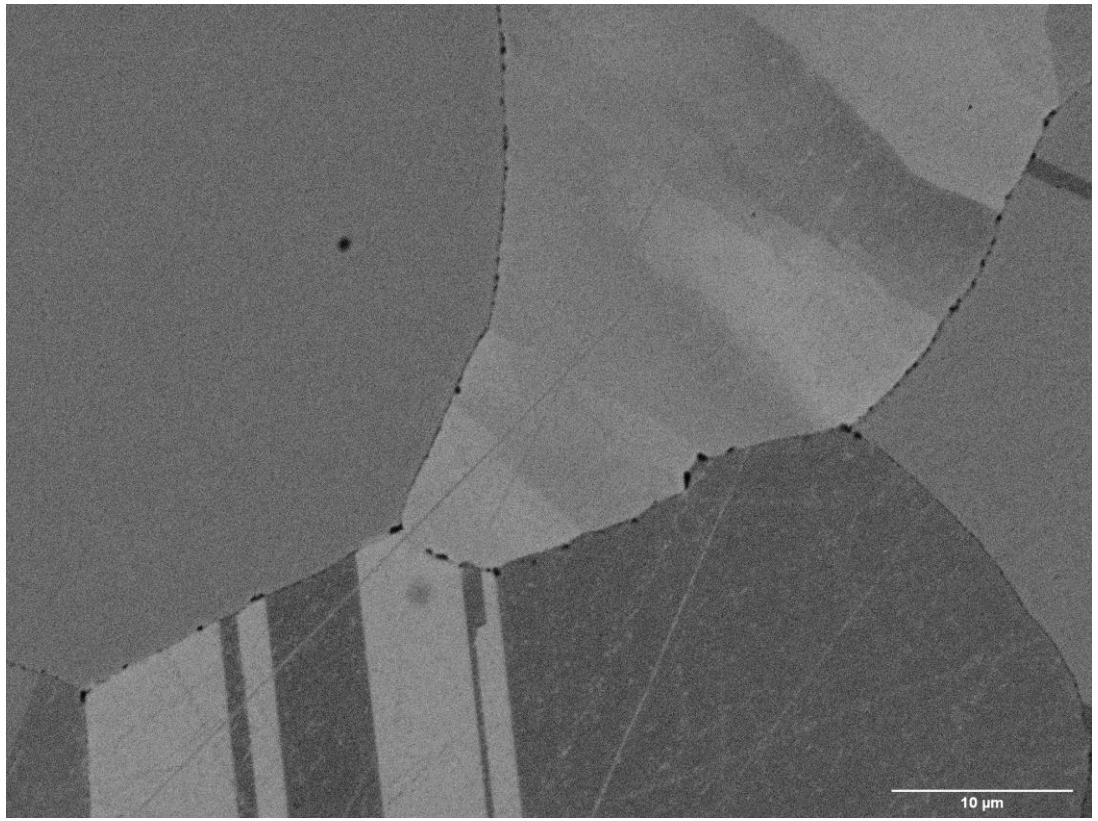
2.5mm HT



2.5mm AB

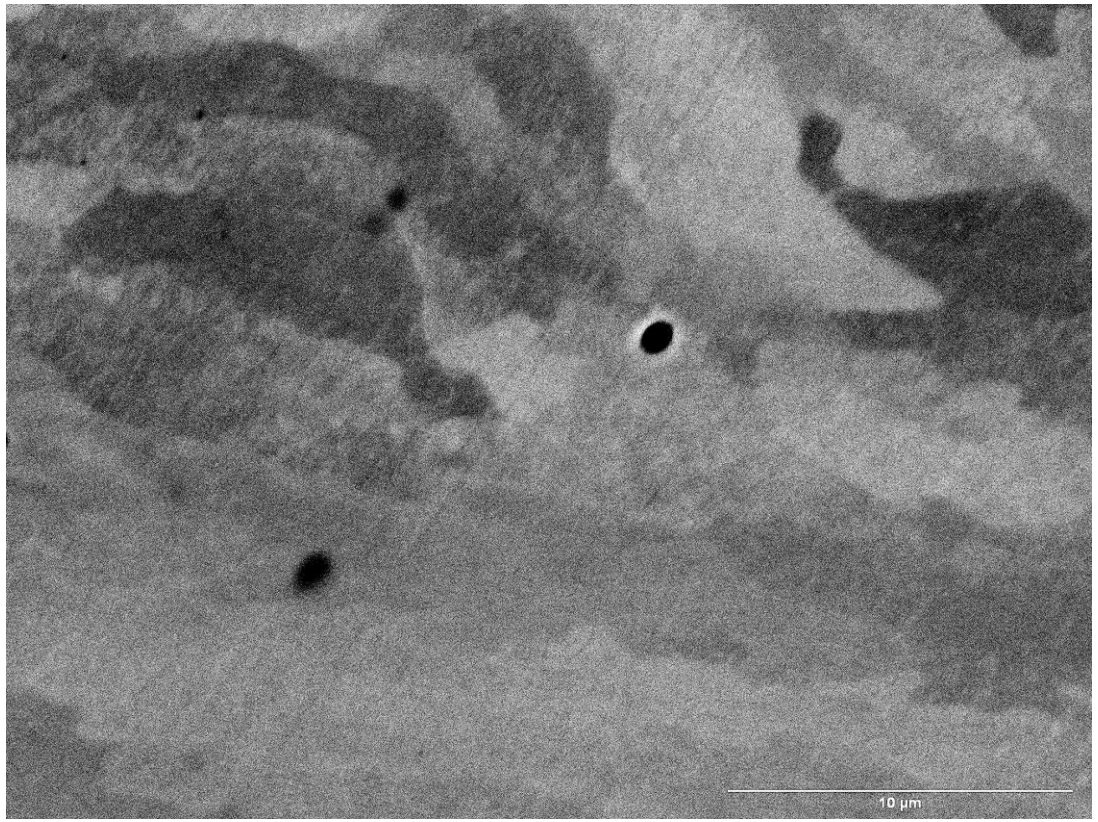
SEM Carbide Formation Cont.

Carbides Present



2mm HT

Carbides Not Present



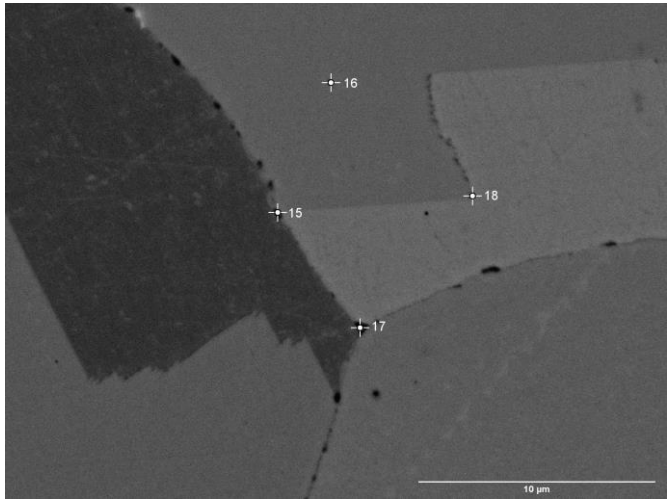
2mm AB

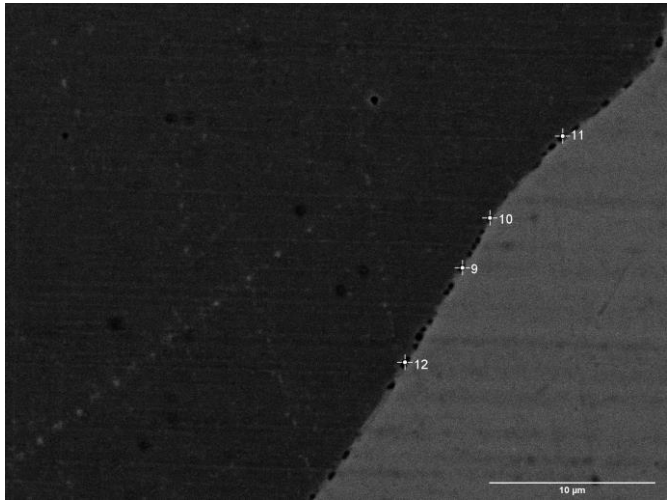
SEM Carbide Formation Findings

- In samples with first run heat treating – 1mm and 2.5mm – carbide formation was observed at the grain boundaries for the HT FR only, not the HT or AB
- For samples with just the heat treating – 1.5mm and 2mm – carbide formation was observed at the HT grain boundaries

Carbide Presence in Haynes 214

2mm HT XY 4500x





1mm HT XZ 3200x

Element (at. %)	9	10	11	12	15	16	17	18
O	-	-	-	-	-	-	-	-
Al	3.67	5.11	4.93	6.93	4.31	5.76	3.11	6.87
C	22.54	20.28	22.46	21.79	26.78	18.48	27.12	20.24
Fe	1.4	1.66	1.66	1.76	1.51	1.41	1.27	1.81
Cr	33.51	22.58	21.65	18.19	25.2	10.52	36.6	13.1
Co	-	-	-	-	-	-	-	-
Ni	31.33	42.68	42.44	45.71	36.66	43.7	26.38	53.08
Cr/C	1.48	1.13	1.04	1.20	0.94	0.57	1.35	0.65

- Carbides could be MC
- Carbide formation along grain boundaries which is to be expected

Grinding and Polishing Procedure for Al 6061/6061RAM2

- Previous intern's procedure was refined for Al 6061/6061RAM2

Grinding Procedure						Polishing Procedure							
Grit	Time	Force	Plate Speed	Holder Speed	Rotation Type	Plate	Solution	Time	Force	Plate Speed	Holder Speed	Rotation Type	Dose
120	1:00	20 N	300 rpm	150 rpm	Co-rotation	MD-Dac	DiaDuo-2 3 µm	10:00	20 N	150 rpm	150 rpm	Co-rotation	15
220	1:00	20 N	300 rpm	150 rpm	Co-rotation	MD-Mol	DiaDuo-2 1 µm	10:00	20 N	150 rpm	150 rpm	Co-rotation	15
500	1:00	20 N	300 rpm	150 rpm	Co-rotation	MD-Nap	OPS	10:00	15 N	150 rpm	150 rpm	Counter-rotation	Manual
1200	2:00	20 N	300 rpm	150 rpm	Co-rotation								
2000	1:00	20 N	300 rpm	150 rpm	Co-rotation								
4000	1:00	20 N	300 rpm	150 rpm	Co-rotation								

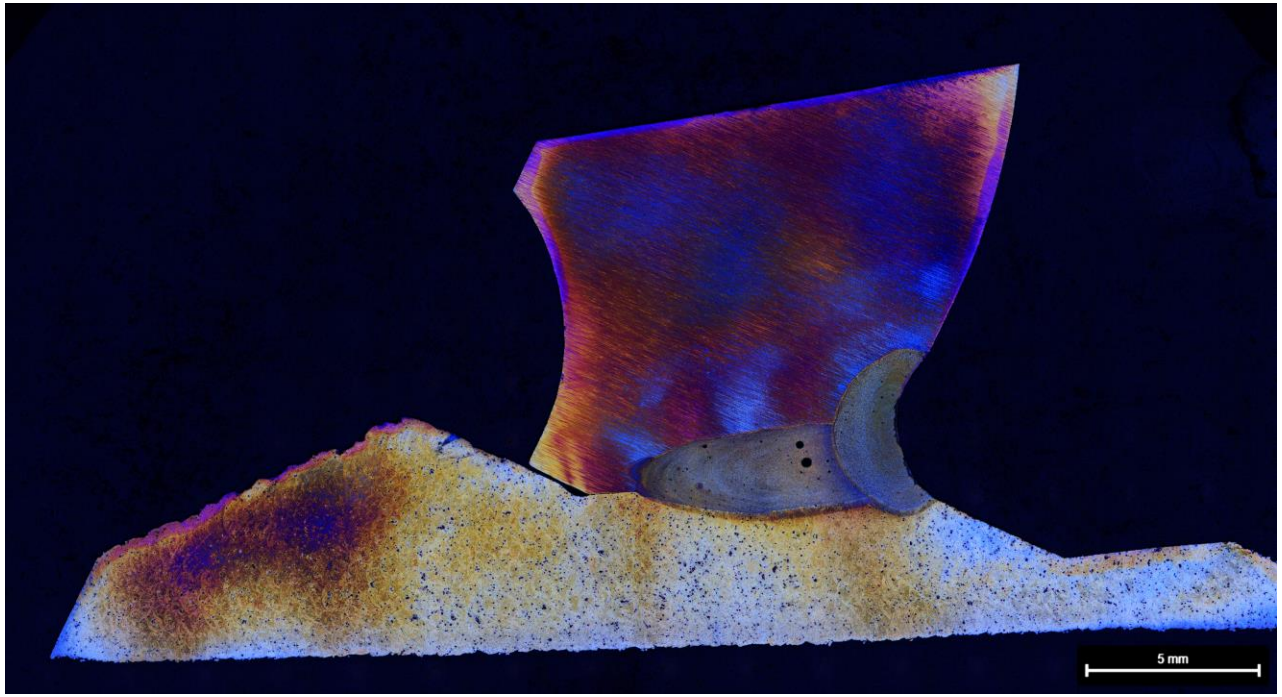
Etching of Al 6061/6061RAM2

- The following etchants were used to varying degrees of success
  - Keller's (20s, then 3 minutes at 25 °C)
    - 20s etch did not reveal microstructure
    - Followed ASM for 3 minutes, but overetched specimen
  - Weck's (8s)
    - Revealed macrostructure but tinted and stained sample
  - Nitric Acid (25% wt., 40s at 70 °C)
    - No grain definition
  - NaOH (9% wt., 5s at 70 °C)
    - Etchant failed to reveal anything
  - Nitric + Hydrochloric + Methanol (1:1:1 vol., 30s - "Methyl Regia")
    - Revealed macrostructure without staining
  - NaOH + NaF + H_2O (2:5:85 wt., 2:30 at 25 °C)
    - Microstructure of weld joints was revealed, but other specimens did not show microstructure

Weld Etching Results Cont.

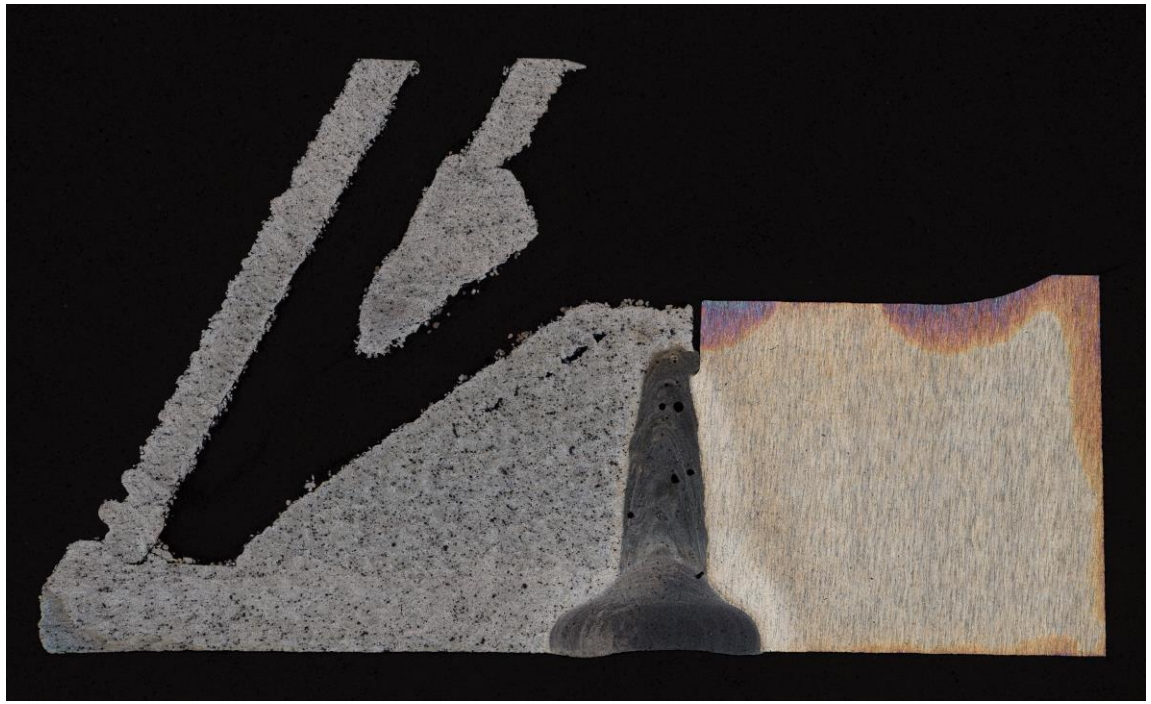
Wecks etch of Weld 3

Wecks etch of Weld 4

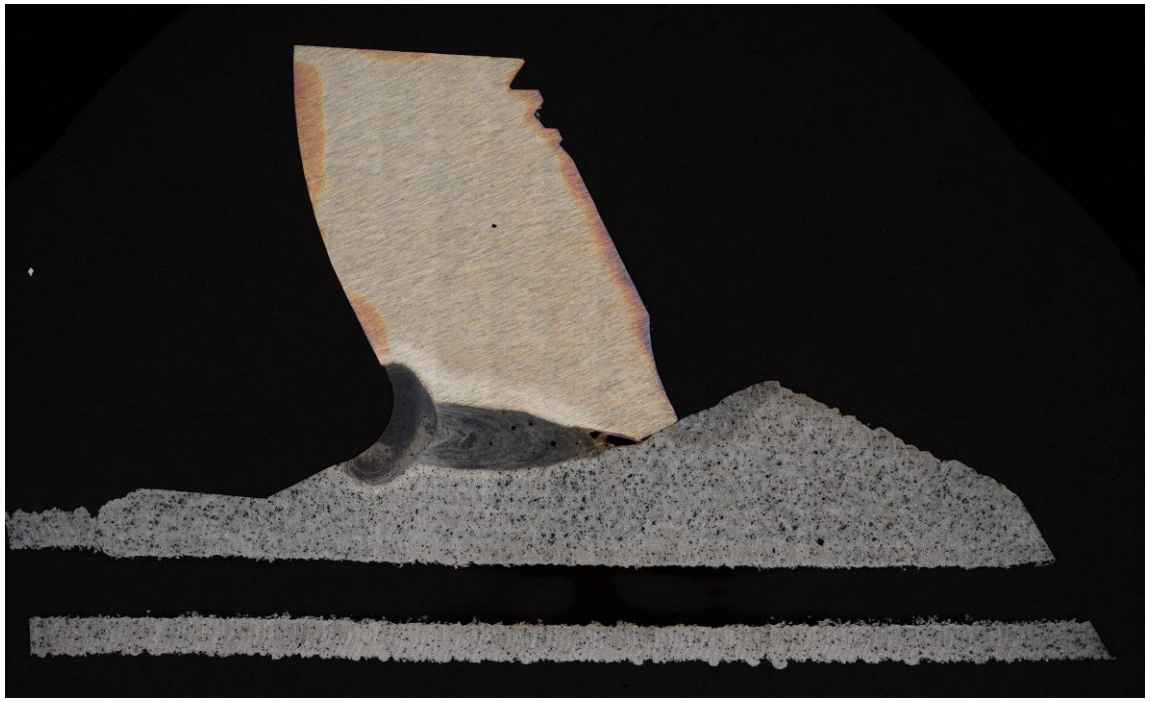


Weld Etching Results Cont.

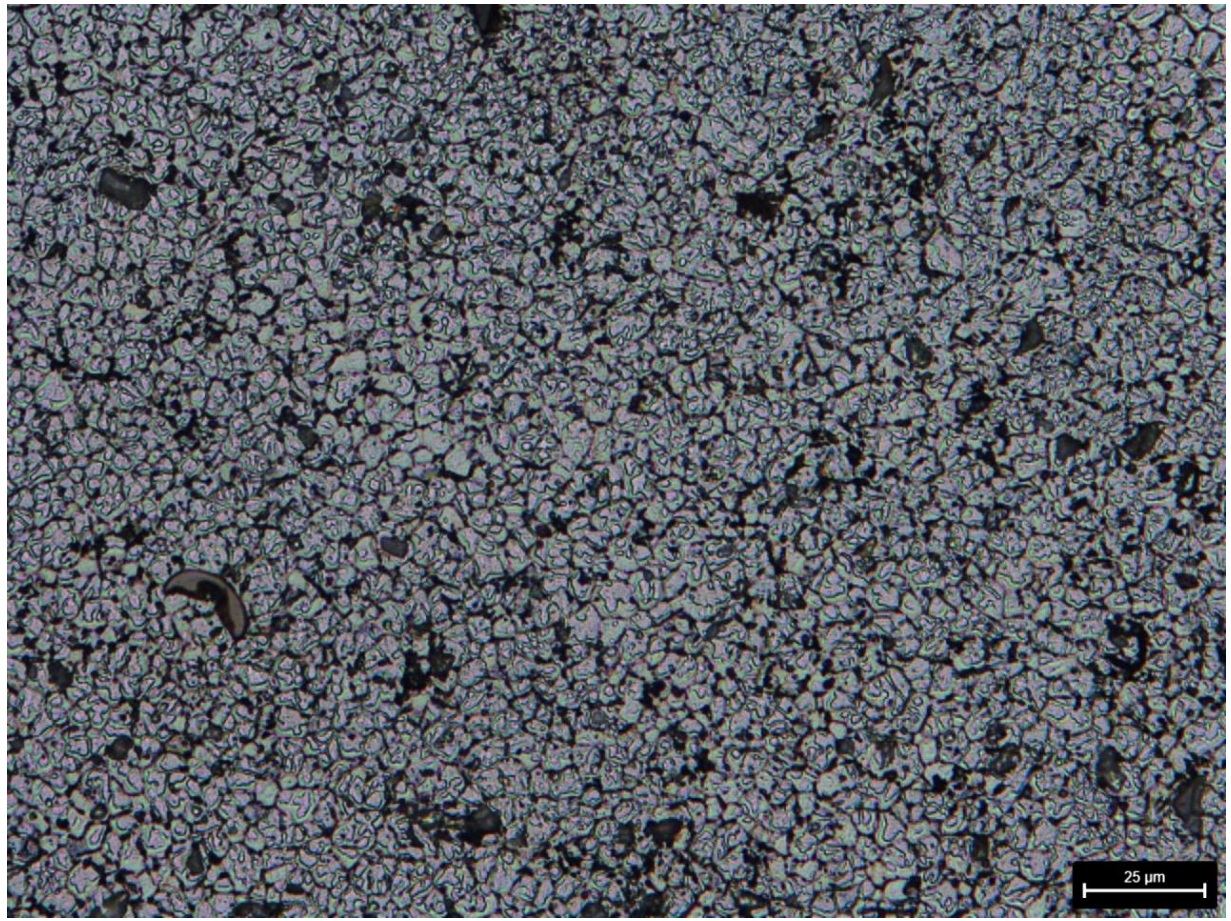
NaOh + NaF etch of Weld 3

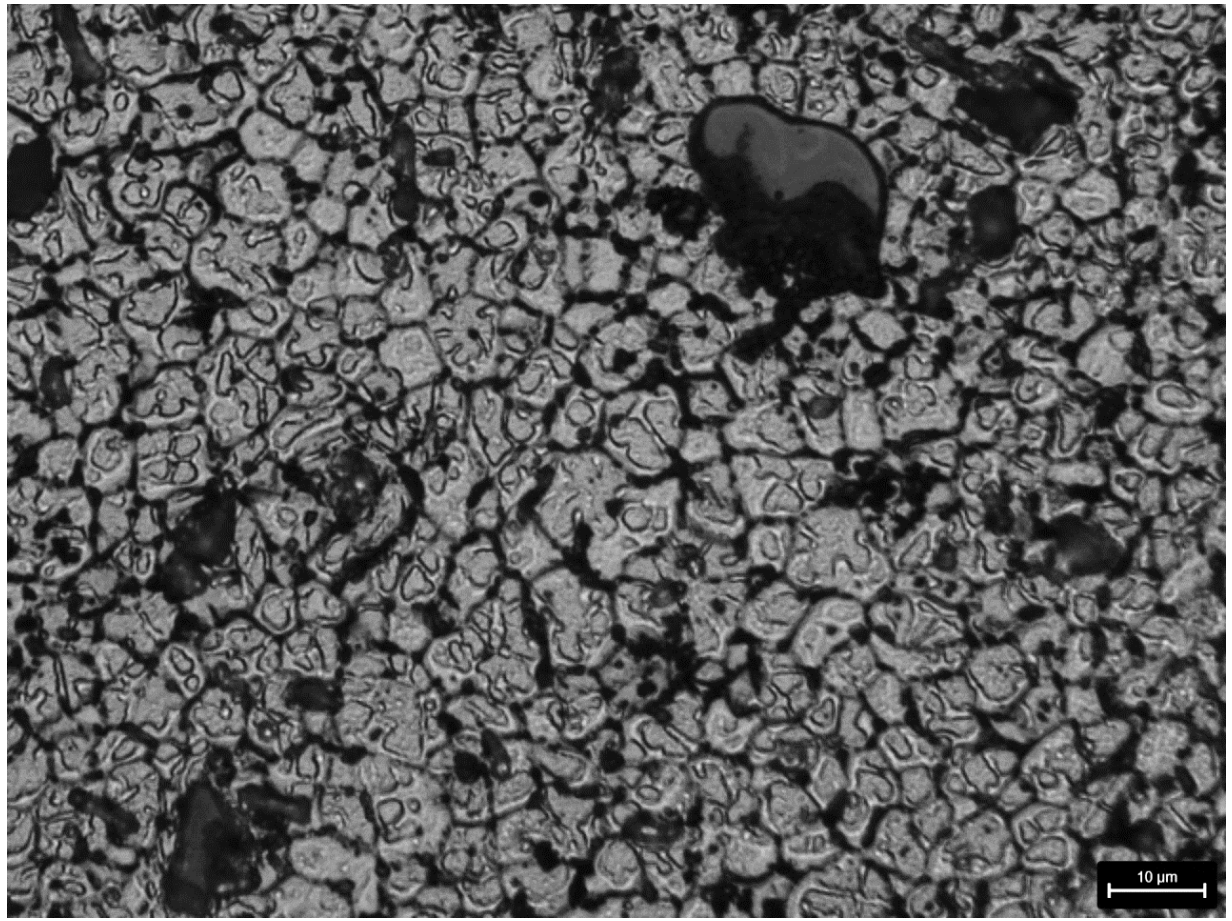


NaOh + NaF etch of Weld 4



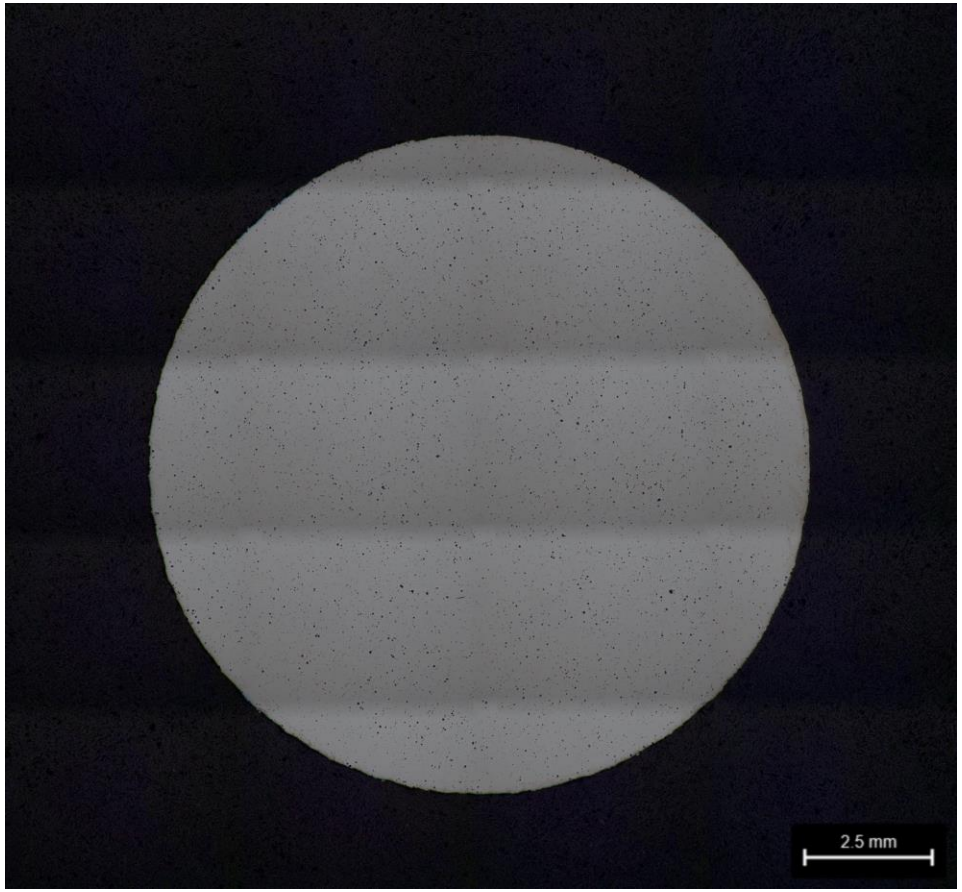
Weld Microstructure Cont.



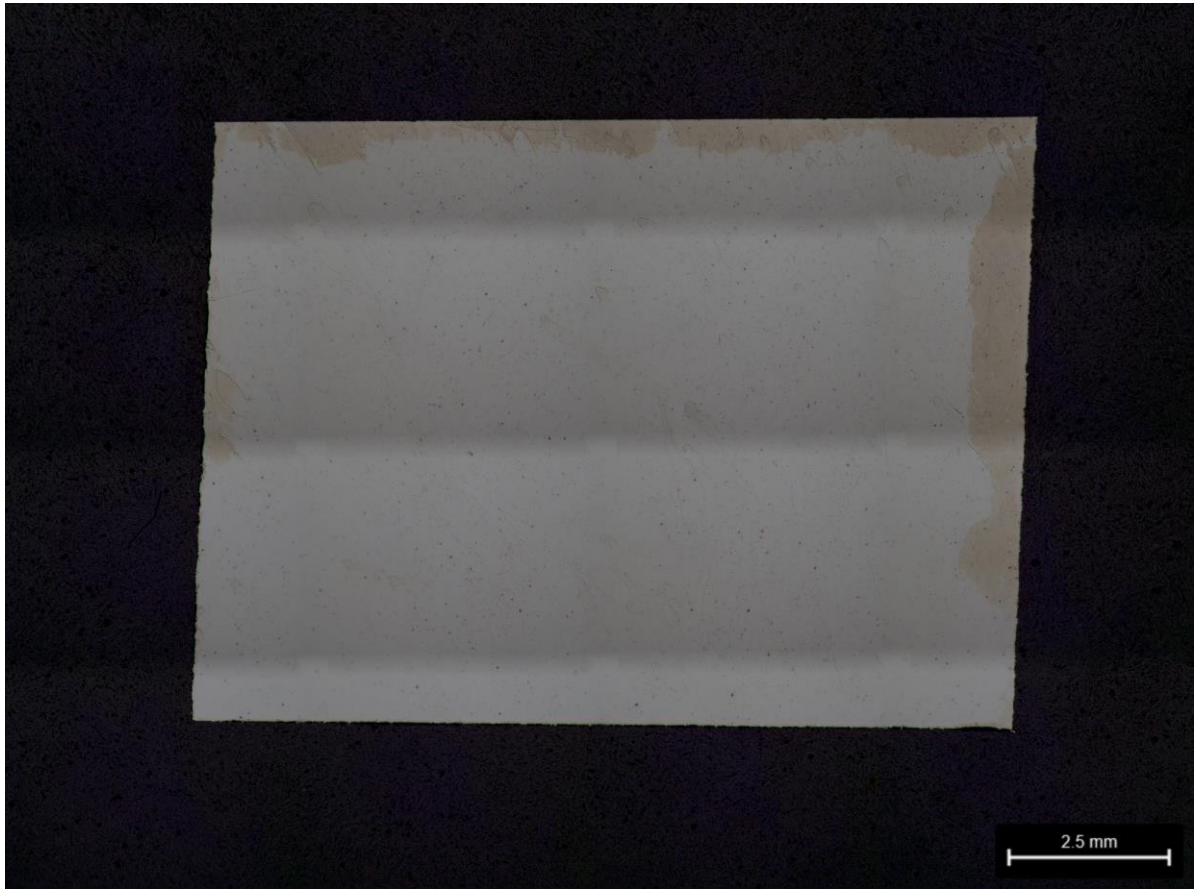


Lower Weld

Unetched Billet (pre-NaOH)

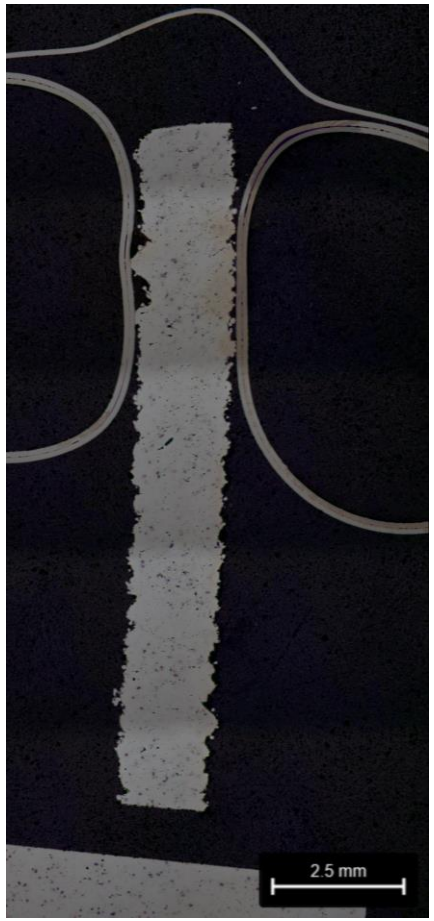


XY Plane



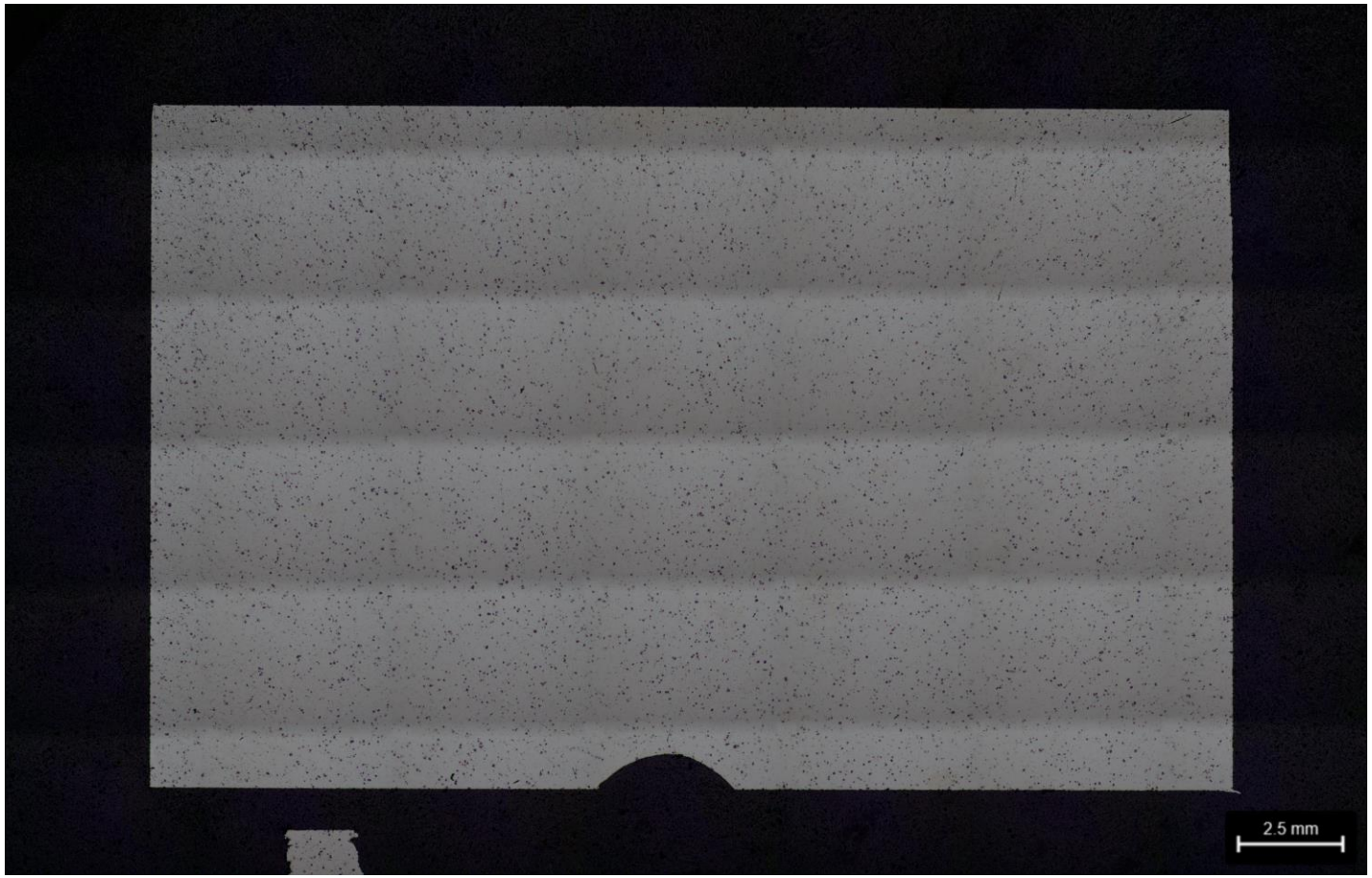
Z Plane

Unetched Anodized Plate (pre-NaOH)



YZ Plane

XY Plane



XZ Plane