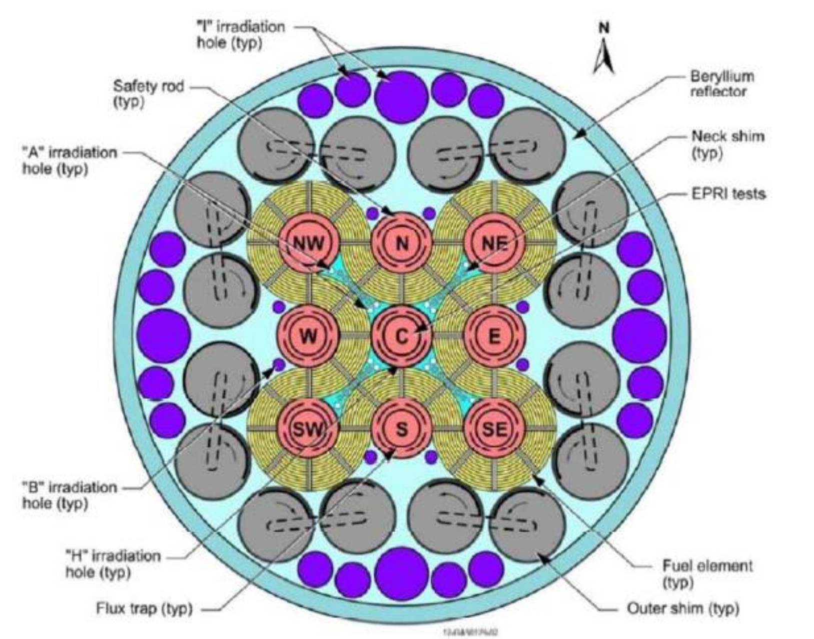
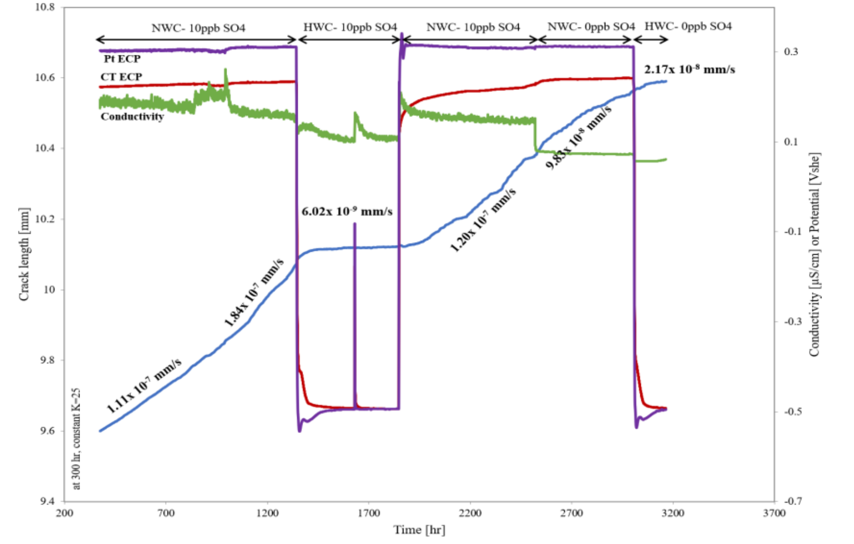




Drew C. Johnson

NSUF Annual Program Review

Monday April 15th

Irradiation Testing of Additively Manufactured Materials for LWR Applications: Alloy 718 and 316L Stainless Steel

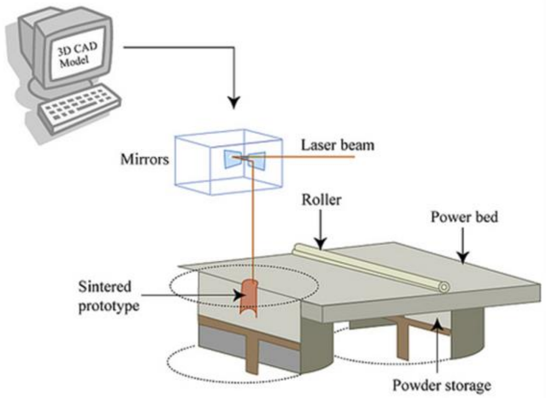
Idaho National Laboratory

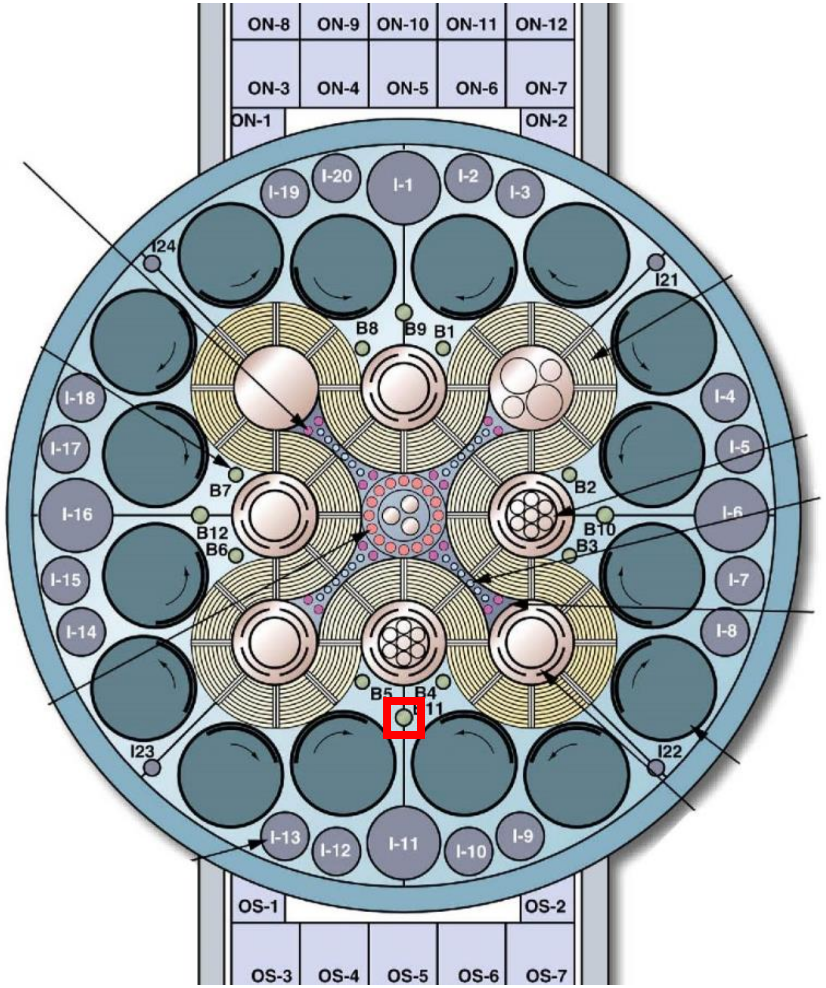
Co-authors

- John H. Jackson, Michael McMurtrey, and Michael Heighes, INL
- Peter L. Andresen, Andresen Consulting
- Myles Connor, General Electric

Project Overview

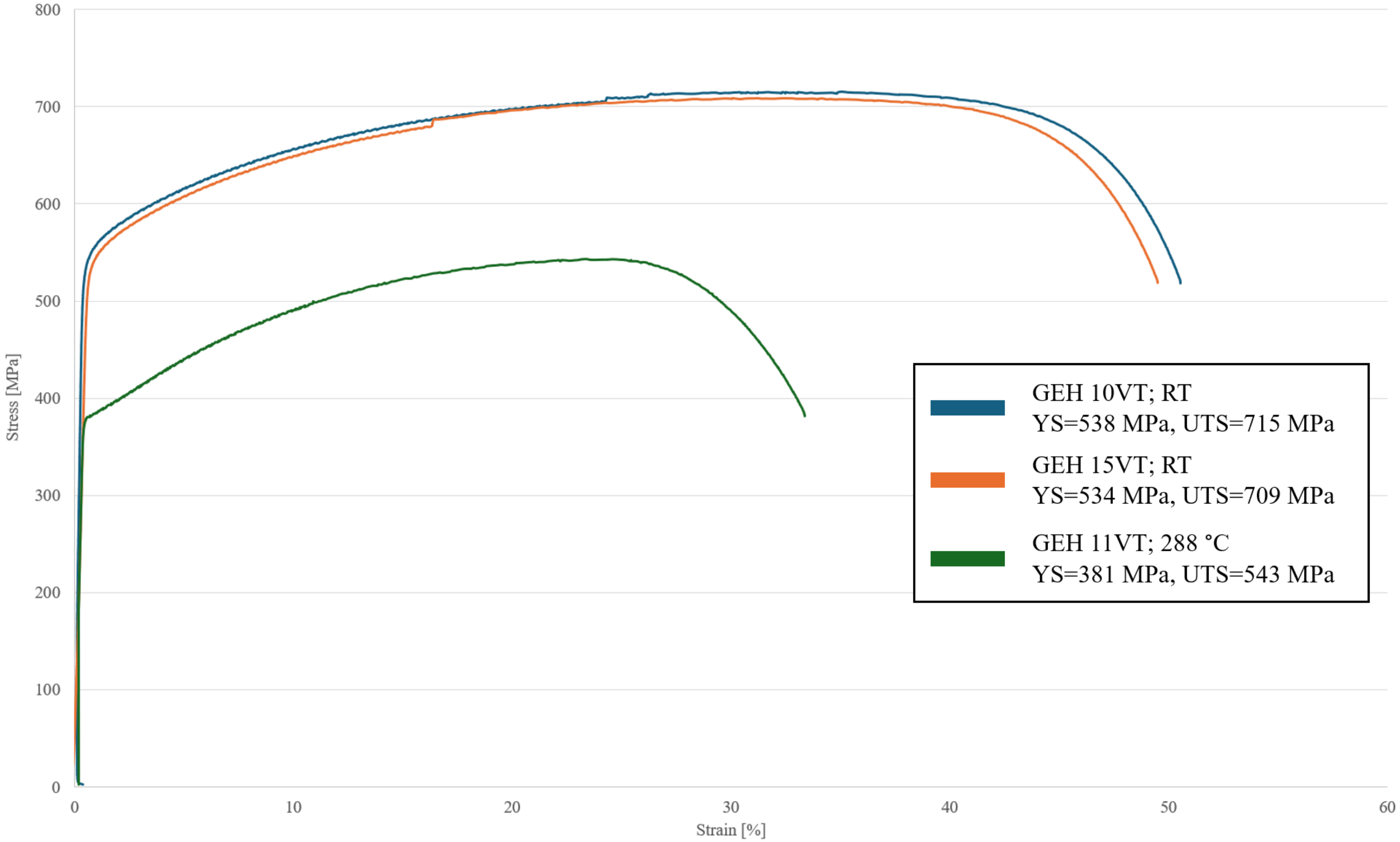
- Direct Metal Laser Melting AM316L and AM718, HIP'ed and solution annealed
- Irradiations conducted at the ATR, utilizing a "Large B" position (B-11)
- ~1 dpa samples produced to investigate end-of-life behavior
- Target temperature of 280-300 °C
- Uniaxial tensile, fracture toughness, crack growth rate testing



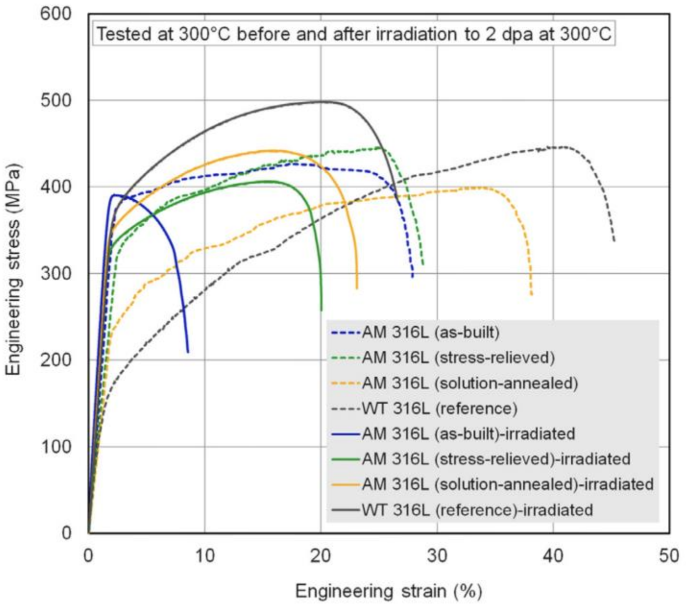


AM 316L Uniaxial Tensile Results

Specimen ID	Test Temp [°C]	0.2% Offset YP [MPa]	UTS [MPa]	Total Elongation [%]
GEH 10VT	27	538	715	50
GEH 15VT	27	534	709	49
GEH 11VT	288	381	543	33
Non-Rad AM 316L	27	270	590	80
Non-Rad AM 316L	288	150	450	49
Wrought 316L	27	270	545	70
Wrought 316L	288	165	450	45



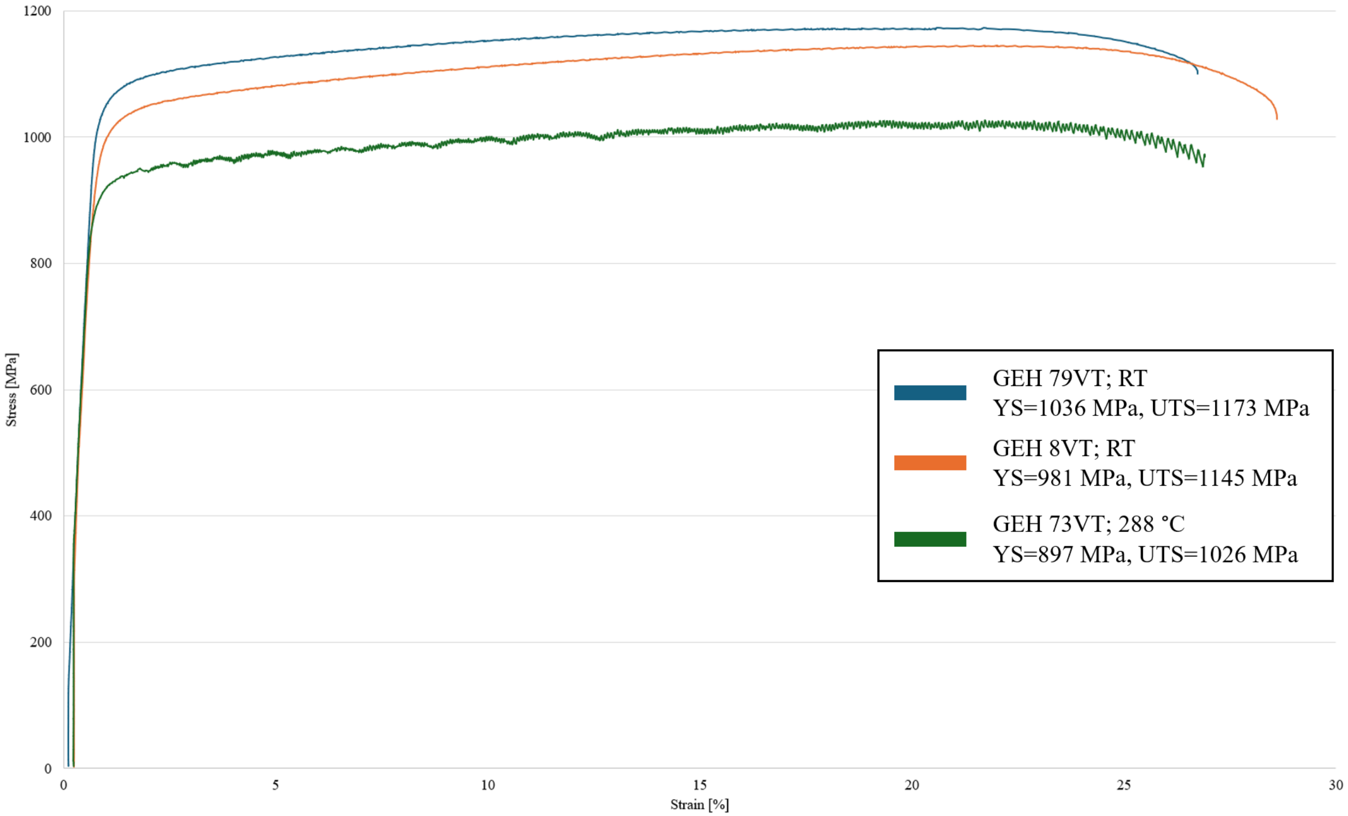
- Anticipated increase in yield, UTS after irradiation at both RT and 288 °C
- Results similar to AM and wrought 316L irradiated to 2 dpa in HFIR



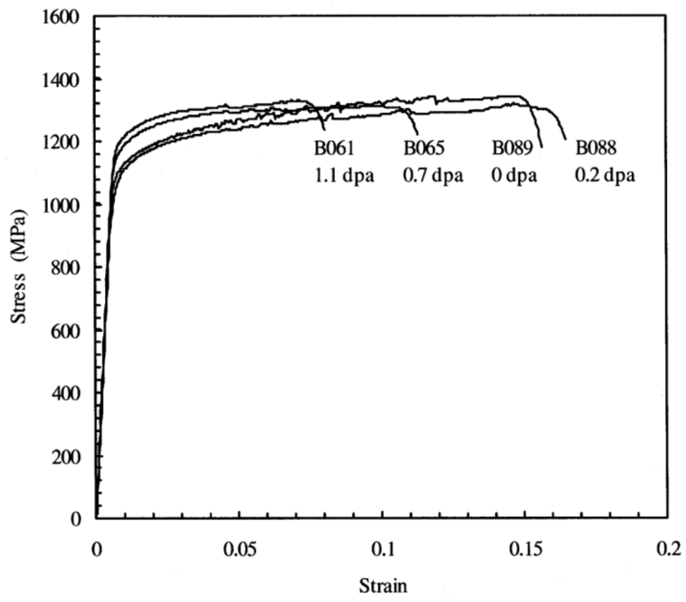
JNM 548 (2021), T.S. Byun et al.

AM 718 Uniaxial Tensile Results

Specimen ID	Test Temp [°C]	0.2% Offset YP [MPa]	UTS [MPa]	Total Elongation [%]
GEH 79VT	27	1036	1173	27
GEH 8VT	27	981	1145	29
GEH 73VT	288	897	1026	27
Non-Rad AM 718	27	915	1280	34
Non-Rad AM 718	288	825	1070	35
Wrought 718	27	840	1140	34
Wrought 718	288	790	1000	35



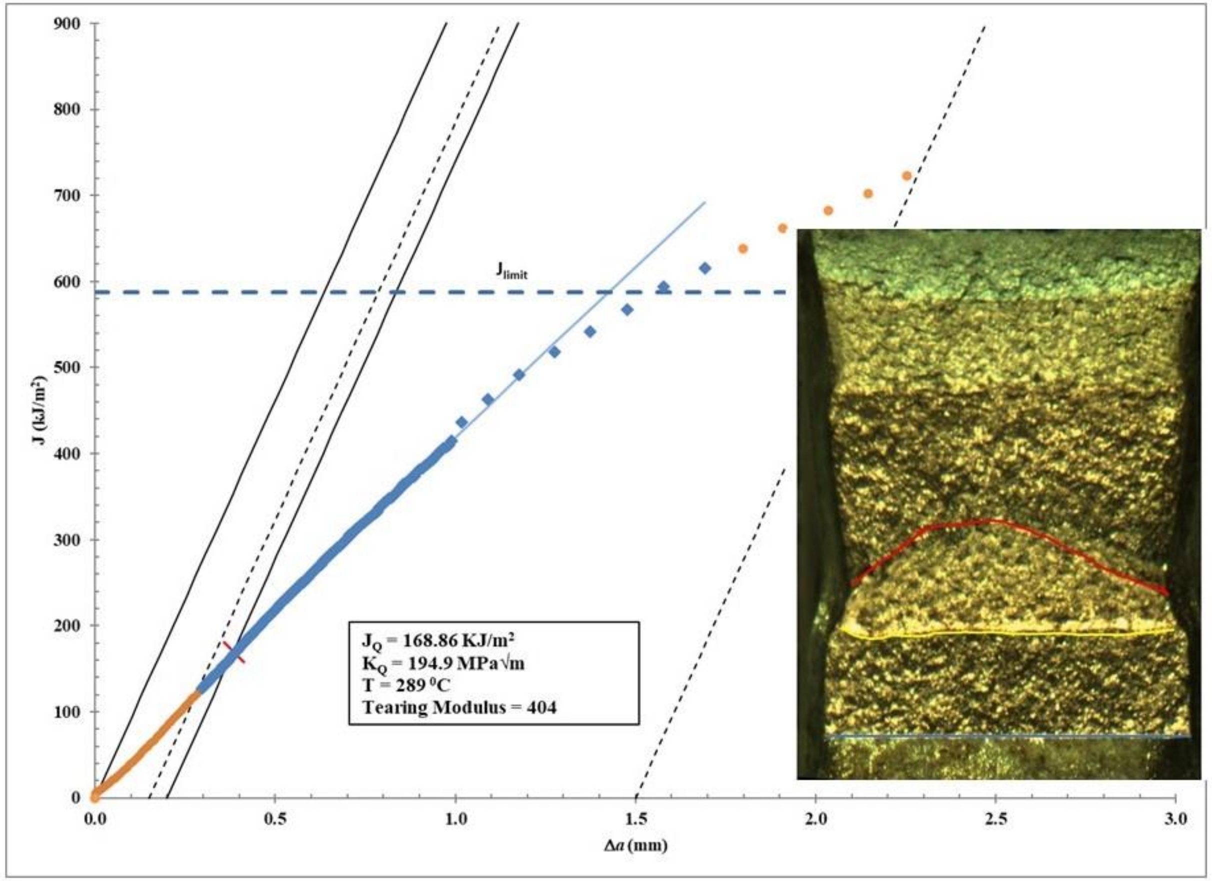
- Anticipated minor increase in yield, UTS after irradiation at both RT and 288 °C
- Results similar to wrought 718 irradiated to ~1 dpa in High Flux Reactor, Netherlands

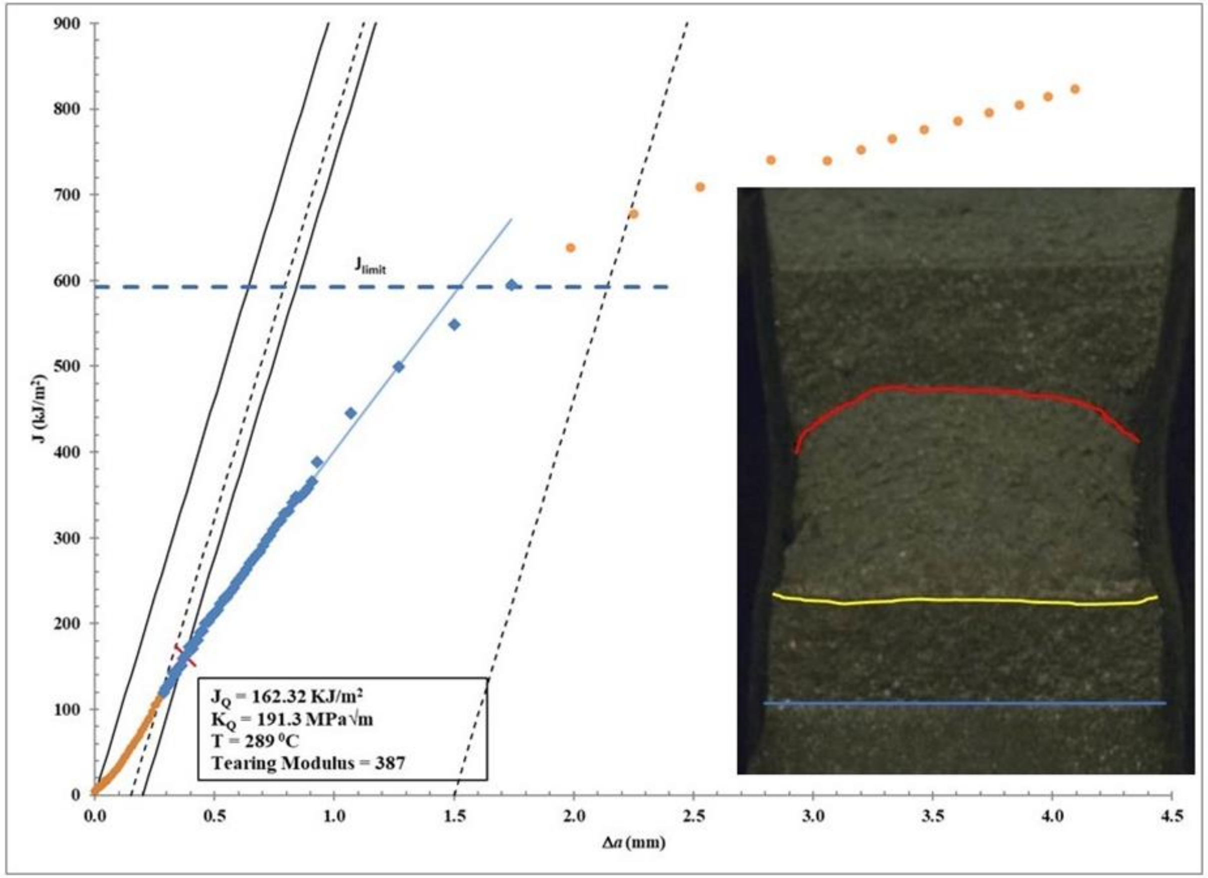


Fusion Engineering and Design 58-59 (2001), P.G. de Heij et al.

AM 316L High Temperature Fracture Toughness Results

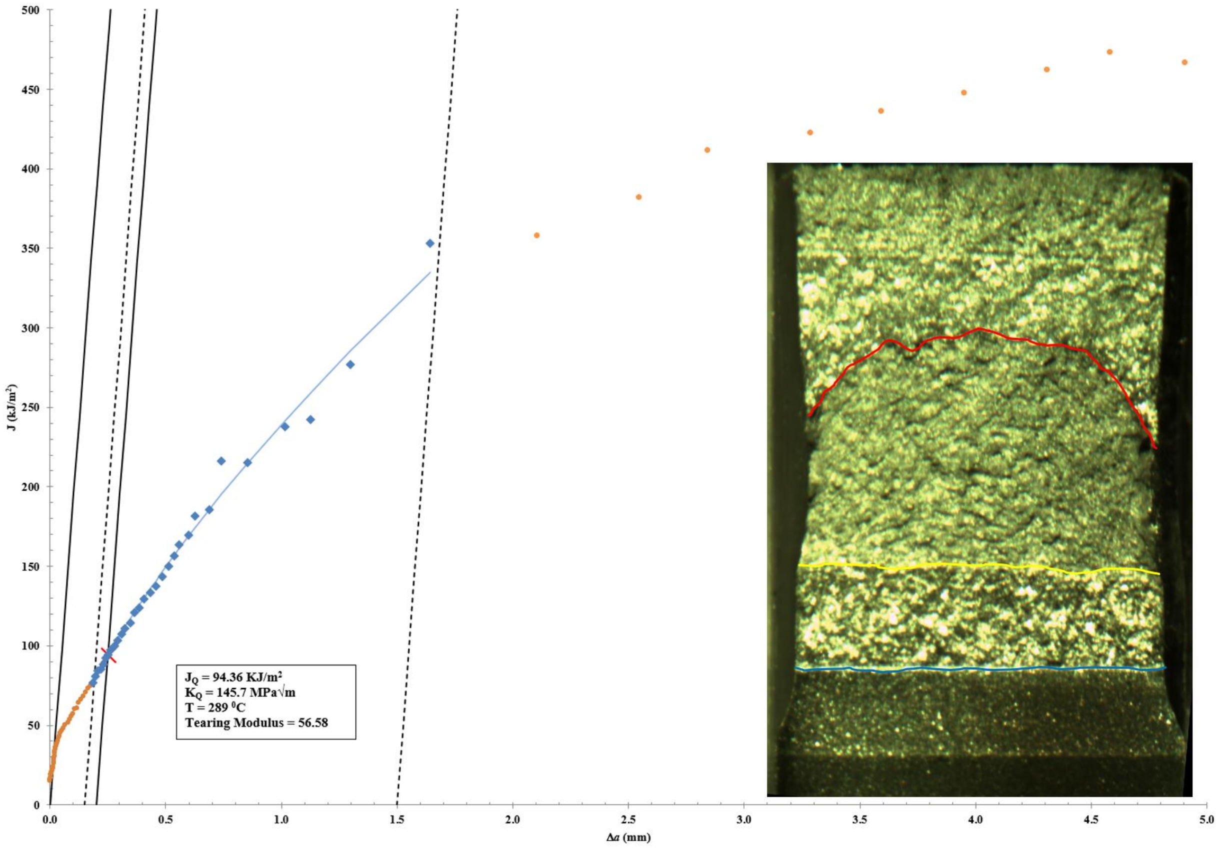
Specimen ID	Material	Temperature [°C]	Dose [dpa]	J_Q [KJ/m2]	K_{JQ} [MPa√m]
GEH 1-1B	AM 316L	288	0.86	162.32	191.3
GEH 5-1D	AM 316L	288	0.99	168.86	194.9

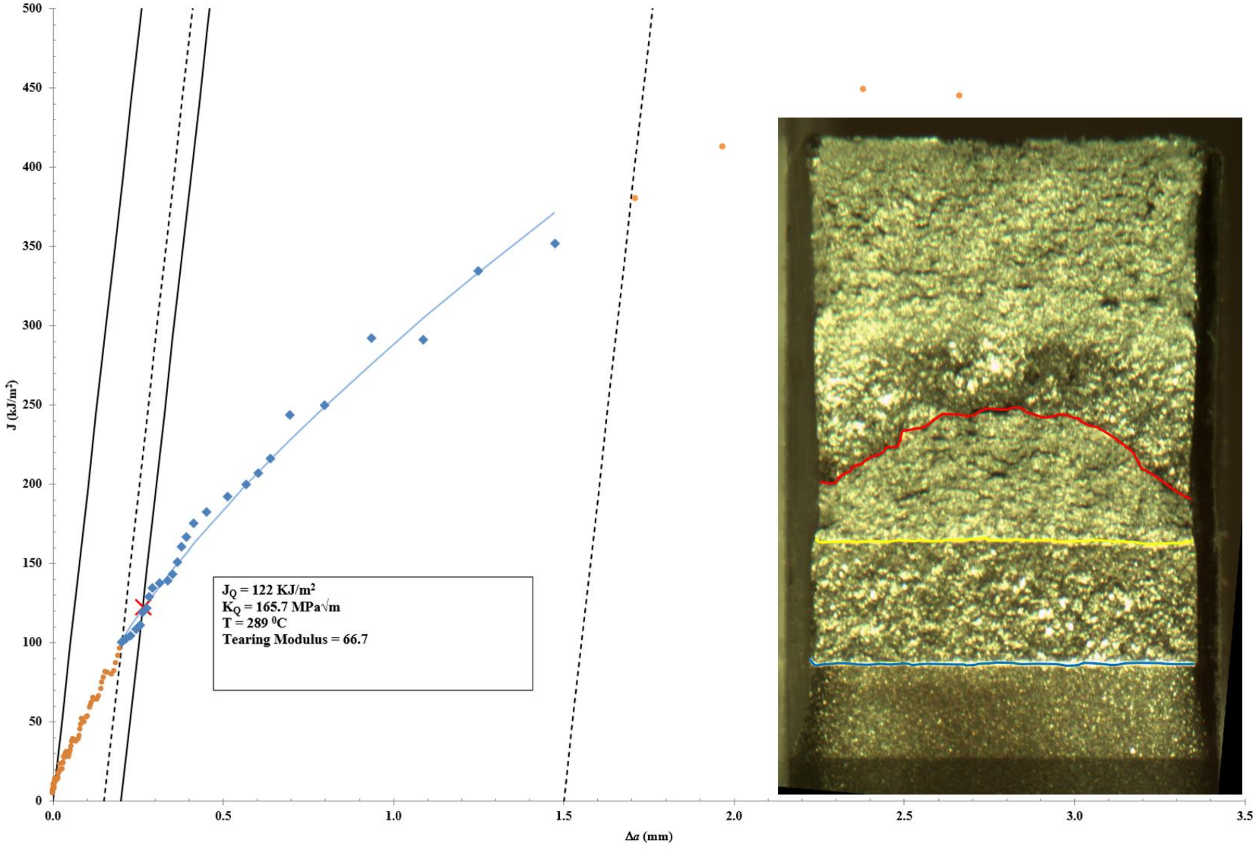




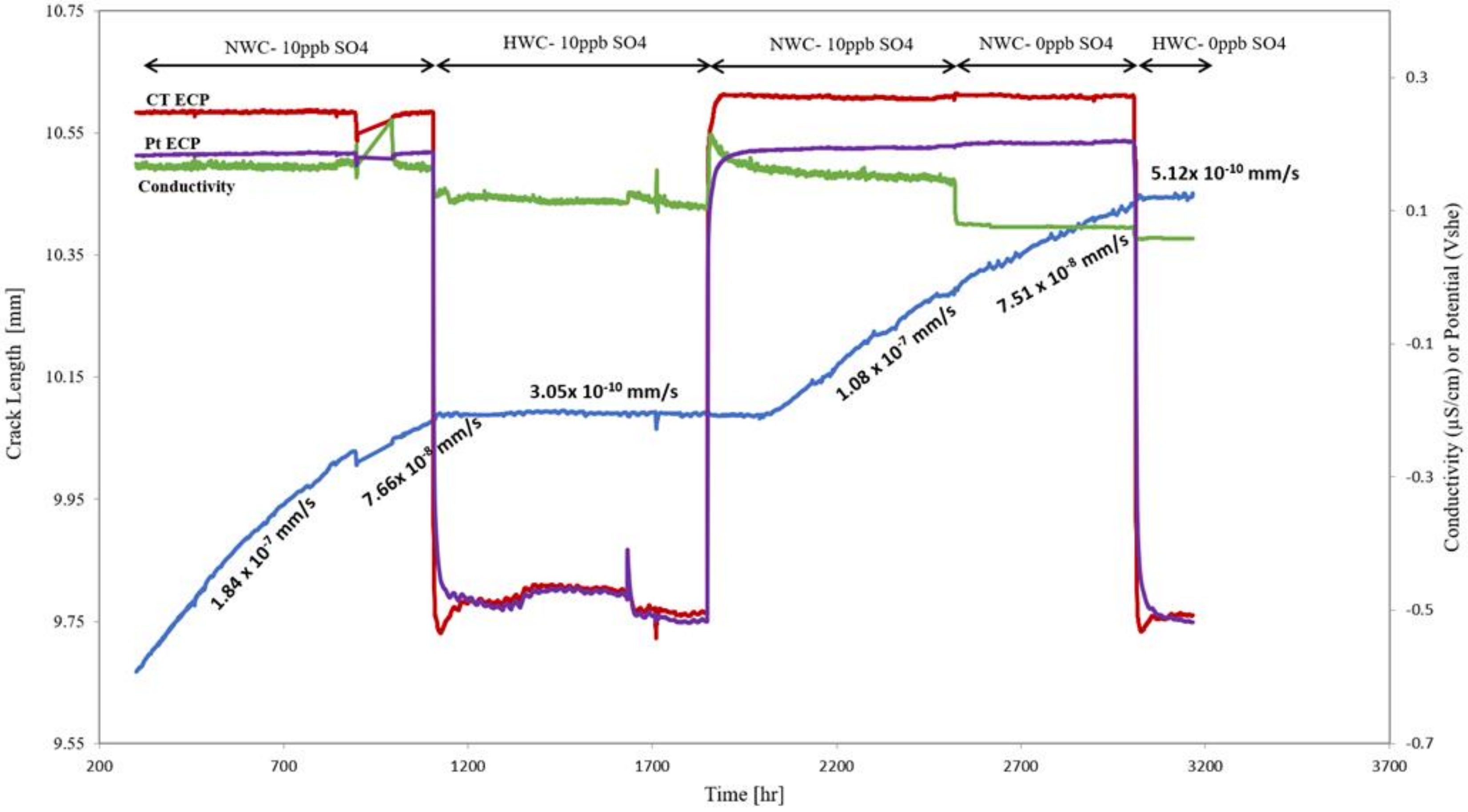
AM 718 High Temperature Fracture Toughness Results

Specimen ID	Material	Temperature [°C]	Dose [dpa]	J_Q [KJ/m2]	K_{JQ} [MPa√m]
GE 46-2B	AM 718	288	1.05	94.36	145.7
GE 51-2A	AM 718	288	0.91	122	165.7





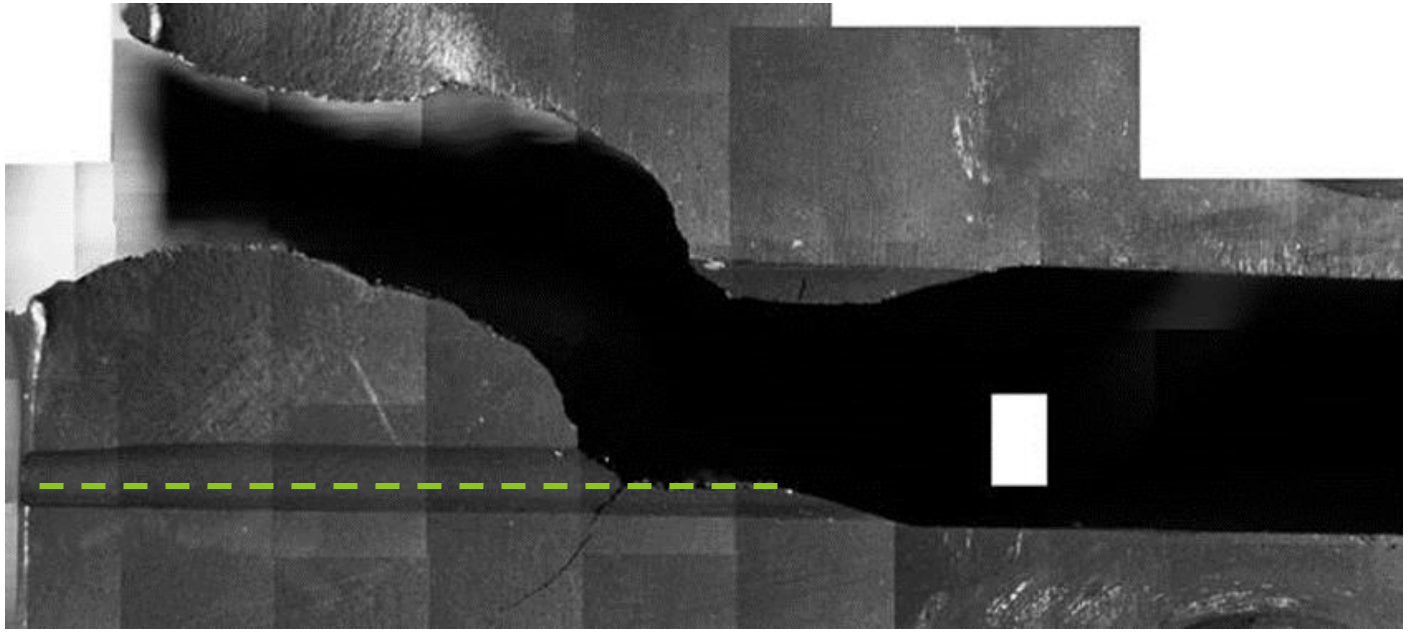
Crack Growth Rate Testing Overview

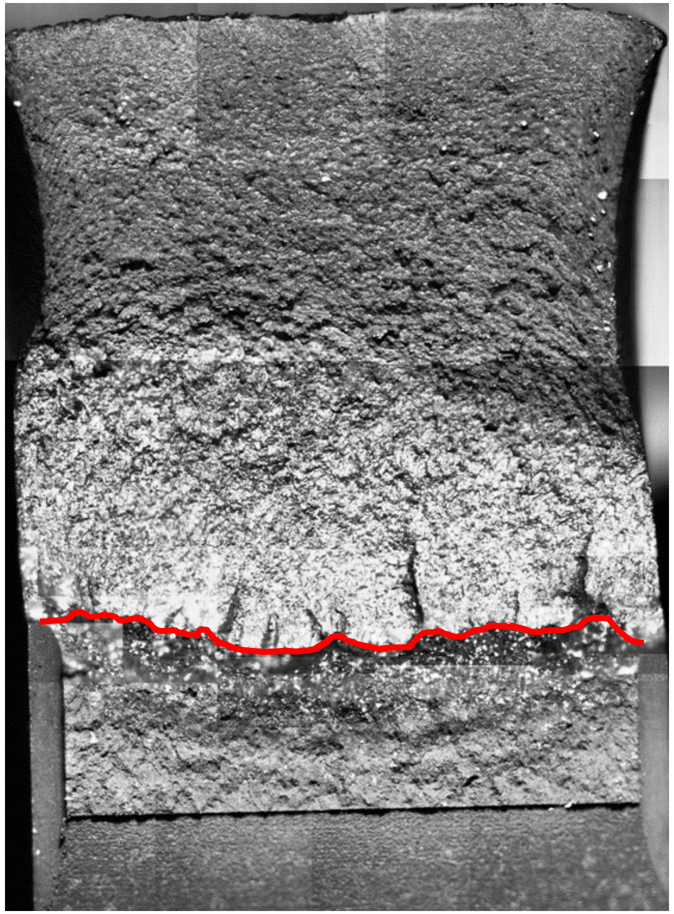


- All testing at 288 °C
- Sulfate additions managed on the fly
- Two samples tested for each alloy (AM316L, AM718)
- AM build directions oriented perpendicular to the anticipated cracking plane

316L 0.89 dpa CGR (GEH31-D)

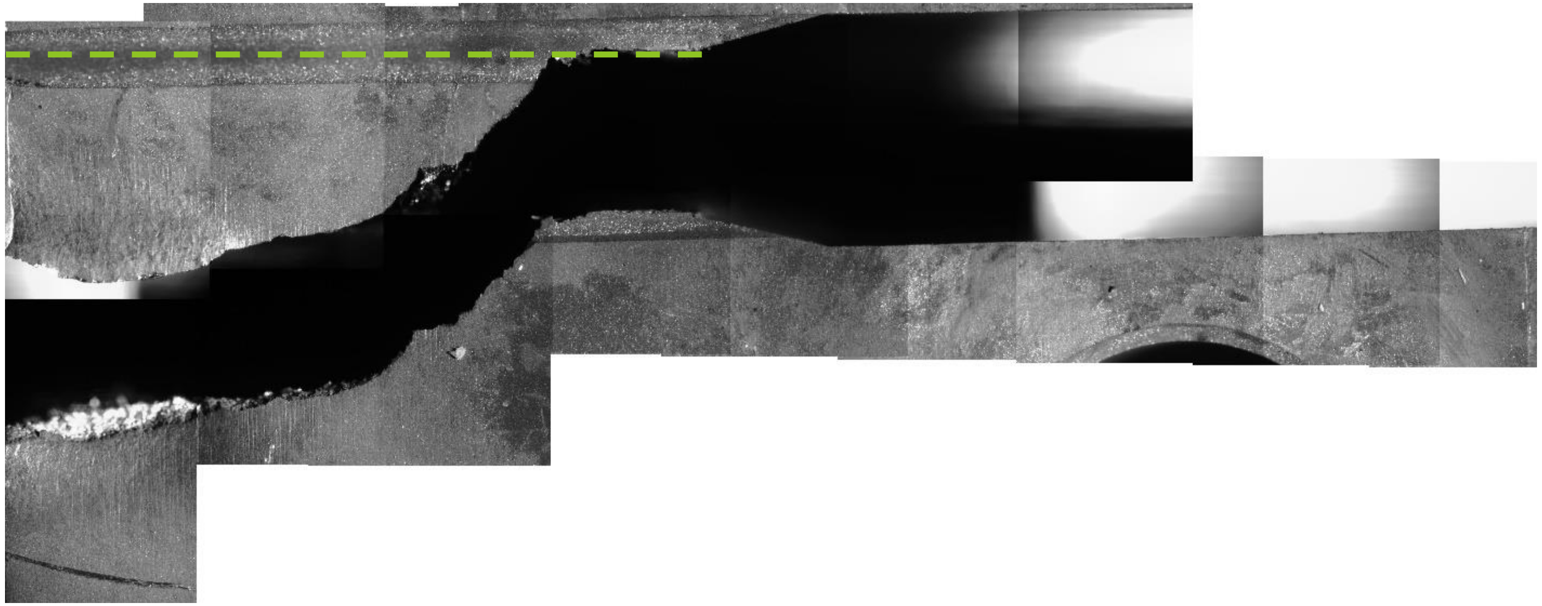
Test Hours	K (MPa√m)	Chemistry	Sulfate	Conductivity (µS/cm)	Average CGR (mm/s)
300	25	NWC	10	0.14	1.84E-07
994	25	NWC	10	0.14	7.66E-08
1144	25	HWC	10	0.1	3.05E-10
1921	25	NWC	10	0.13	1.08E-07
2635	25	NWC	0	0.09	7.51E-08
3024	25	HWC	0	0.07	5.12E-10

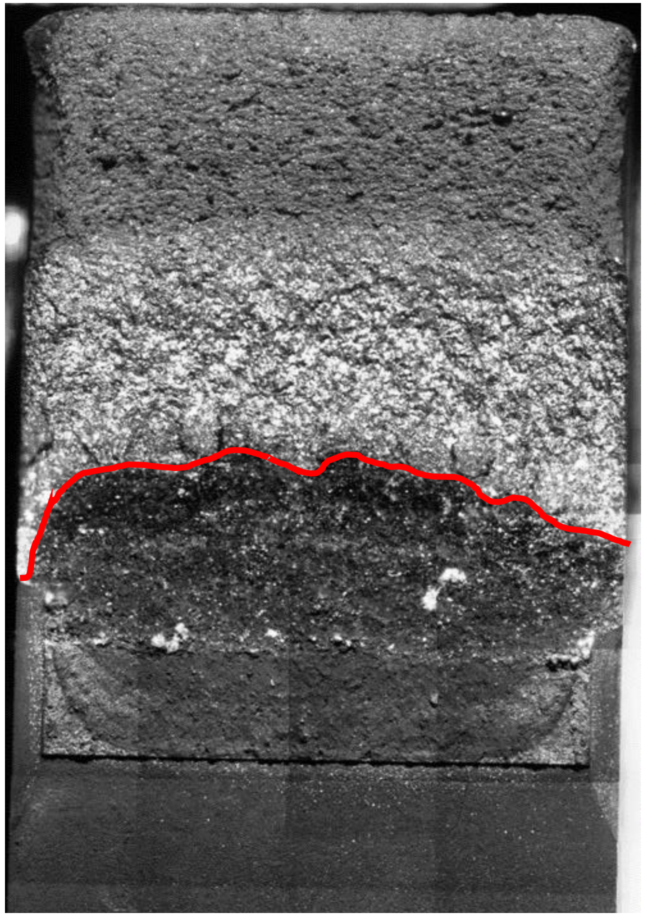




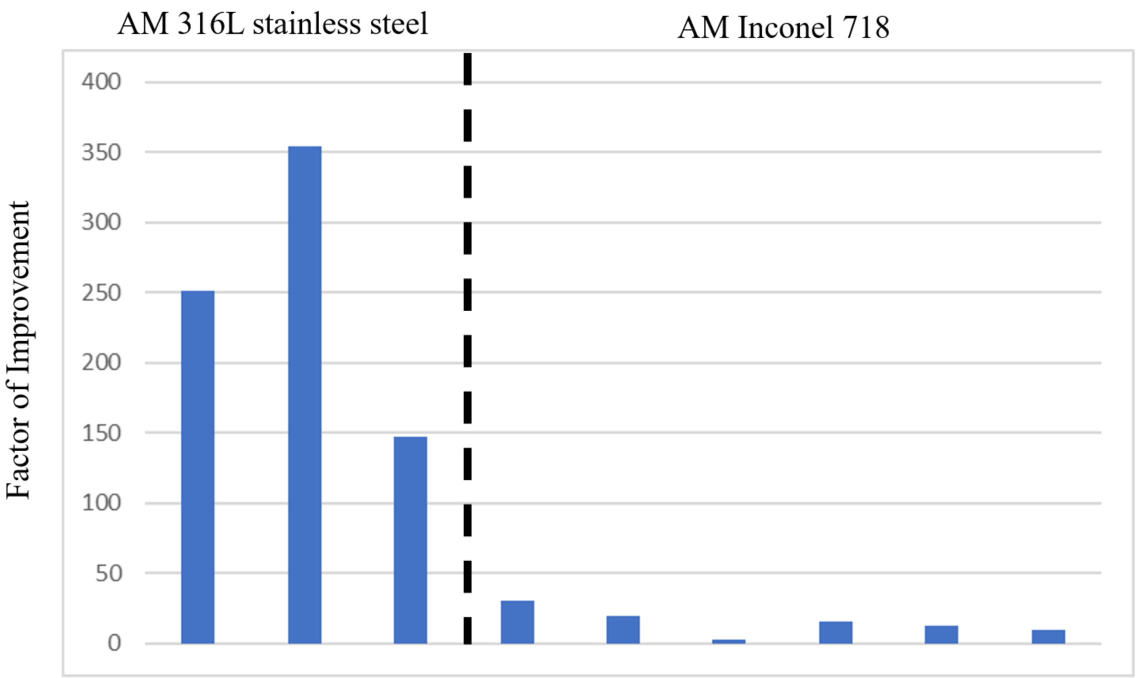
718 1.01 dpa CGR (GE41-2D) – Results

Test Hours	K (MPa√m)	Chemistry	Sulfate	Conductivity (µS/cm)	Average CGR (mm/s)
300	25	NWC	10	0.17	1.11E-07
994	25	NWC	10	0.16	1.84E-07
1312	25	HWC	10	0.11	6.02E-09
1848	25	NWC	10	0.15	1.20E-07
2520	25	NWC	0	0.09	9.83E-08
2998	25	HWC	0	0.07	2.17E-08

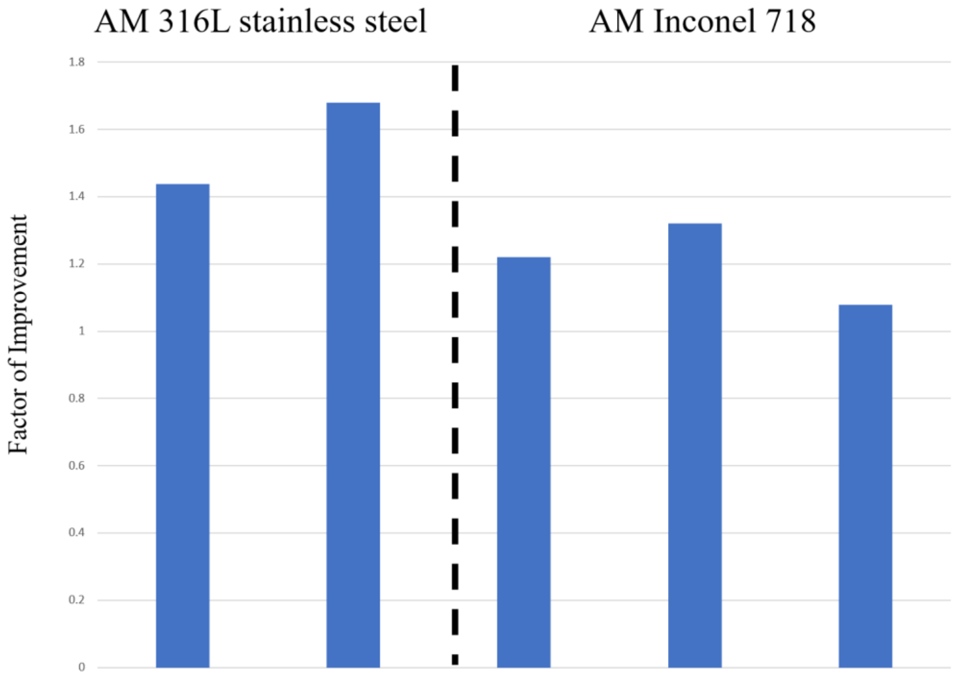




Crack Growth Rate Testing Results



Effect of ECP on CGR



Effect of sulfate additions on CGR

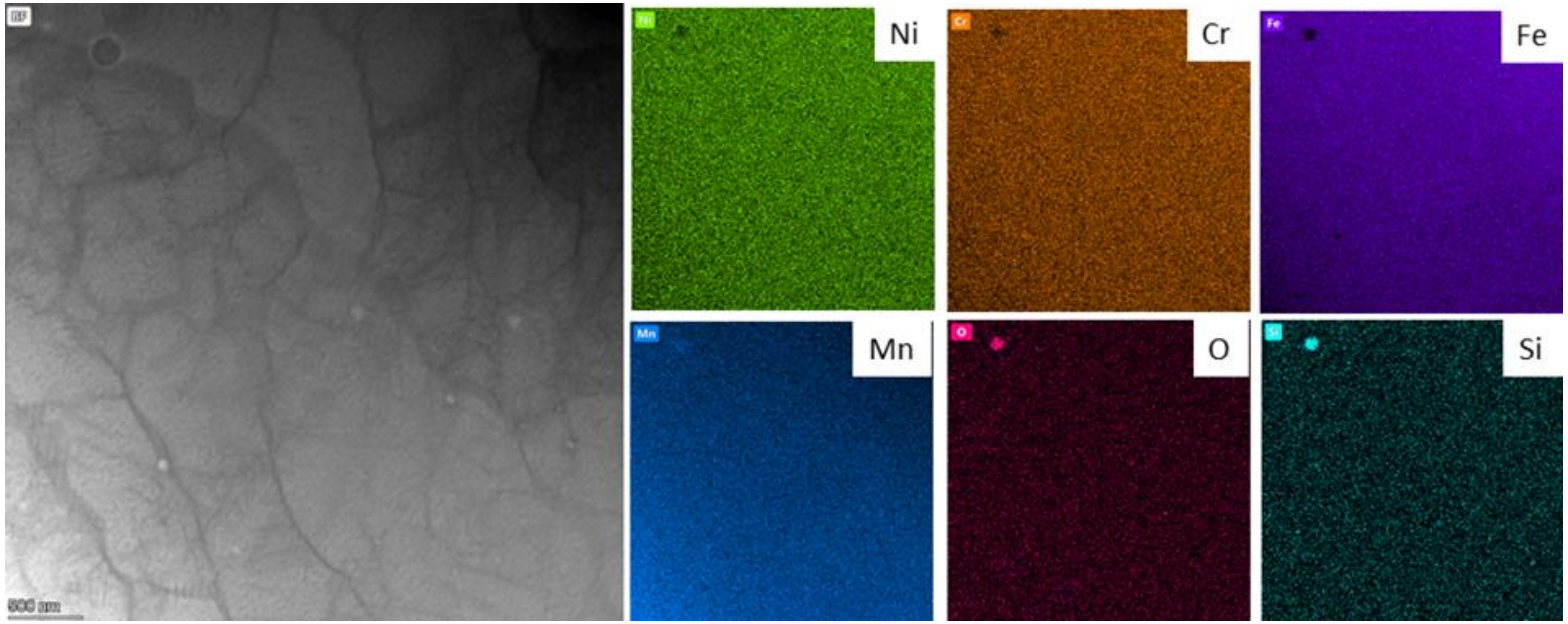
Average CGR reduction at low ECP for AM316L = 251x

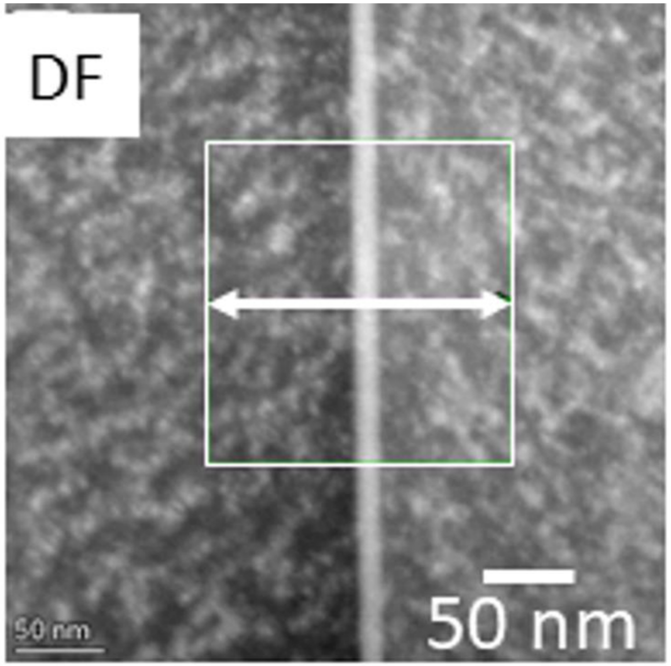
Average CGR reduction at low ECP for AM718 = 18x

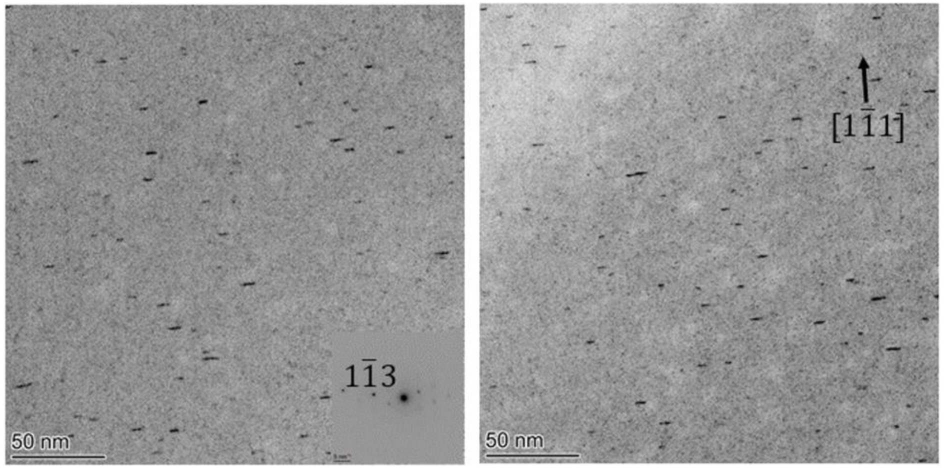
Average reduction in CGR due to changing sulfate additions for 316L=1.56x

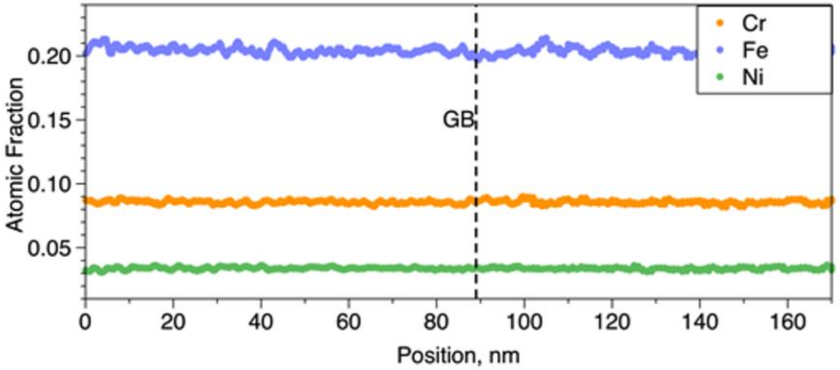
Average reduction in CGR due to changing sulfate additions for 316L=1.21x

AM316L Irradiated Microstructure

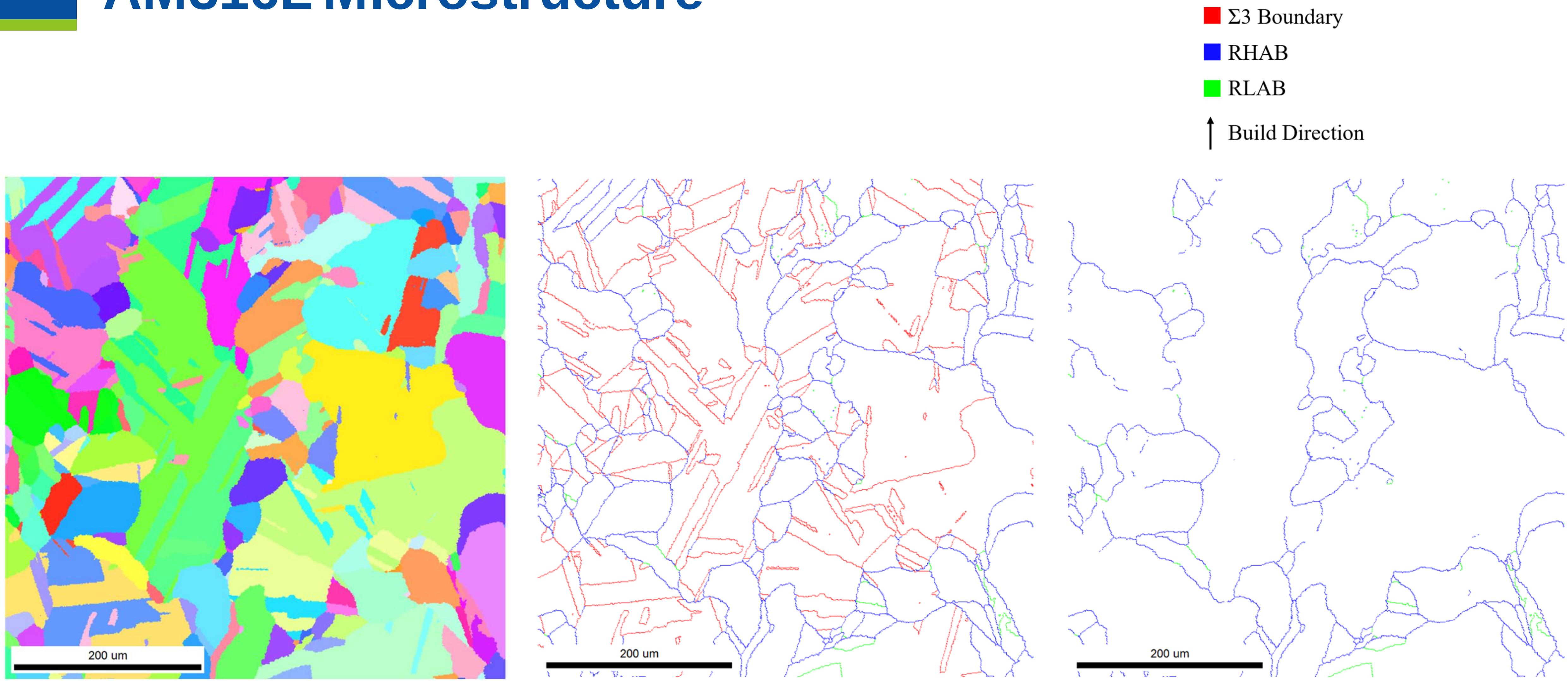




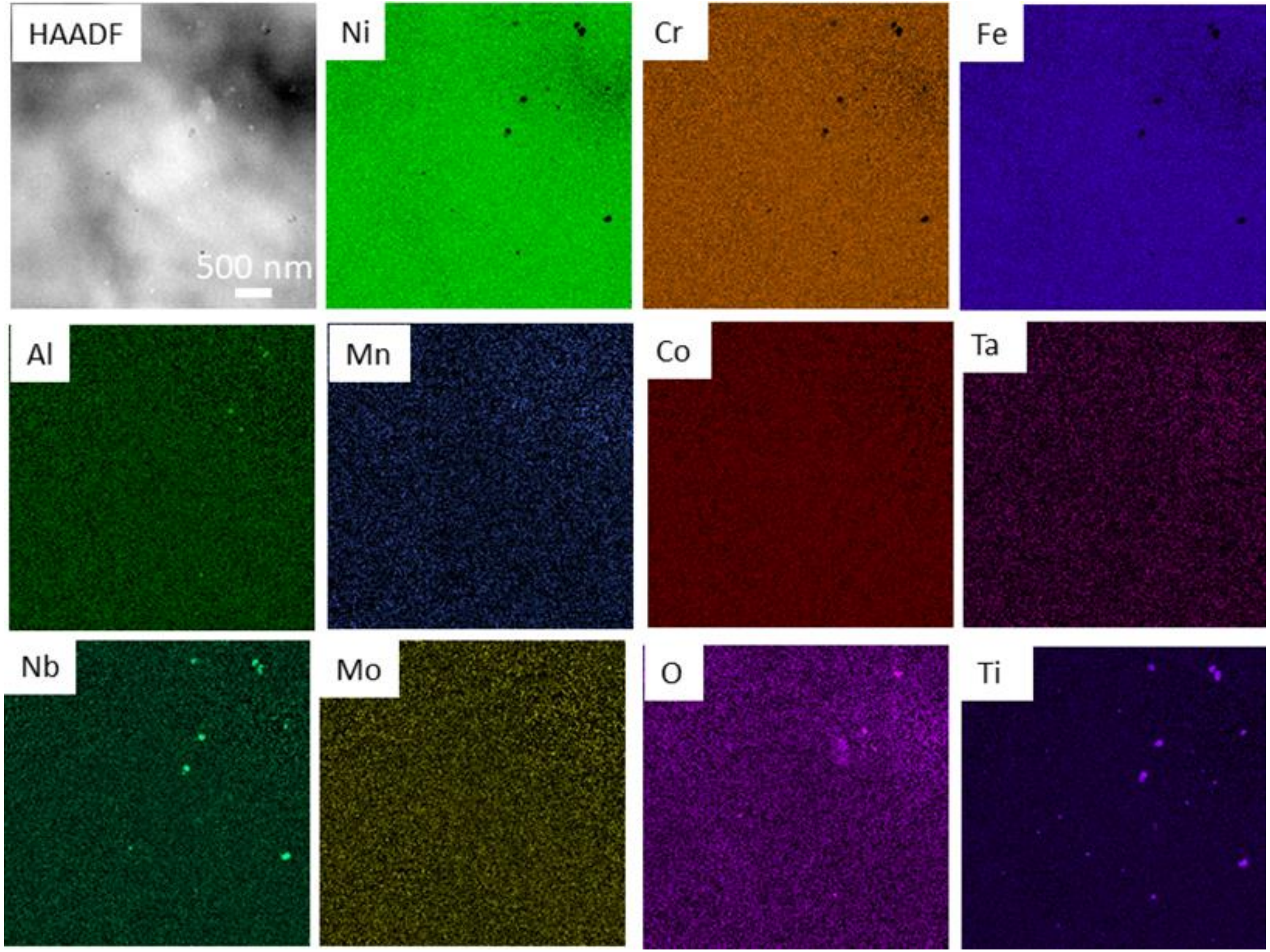


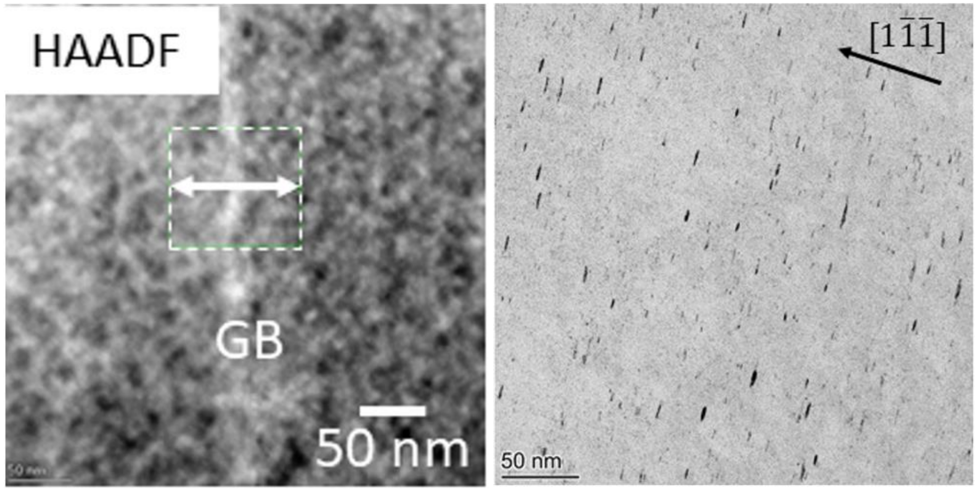


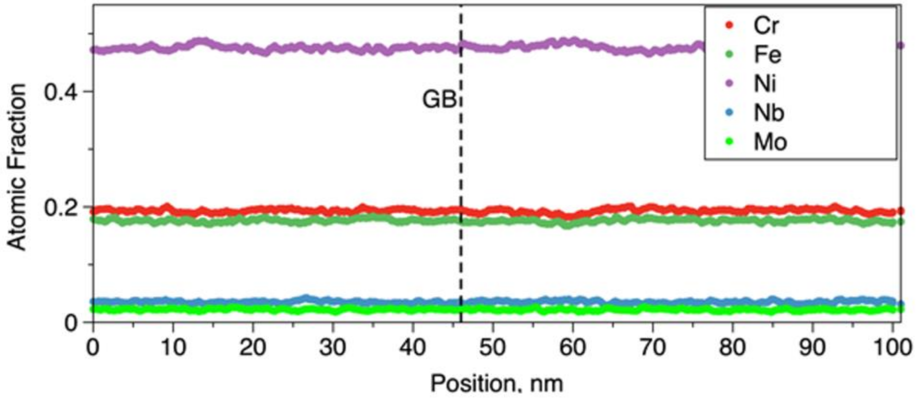
AM316L Microstructure



AM 718 Microstructure







Conclusion

- Anticipated changes in uniaxial tensile behavior observed for both alloys, with yield stress and ultimate tensile strength results matching irradiated wrought material performance
- Based on the fracture toughness results, ample load carrying capacity still remains for both alloys at this dpa level. Reproducible J_Q values observed
- Significant off-plane cracking observed in all samples after completion of the pre-cracking procedure and the start of constant K loading conditions. Evidence of residual dislocation structures and significant twinning grain boundary fraction potentially explain cracking behavior.
- Significant changes in CGR were observed for neutron irradiated AM316L and AM718 after switching between NWC and HWC environments (251x for AM316L, 18x for AM718)
- Slight effect (~1.4x) on CGR observed after changing between 10 ppb sulfate and pure water

Questions?

Backup Slides

AM316L and AM718 Composition

316L Powder Composition

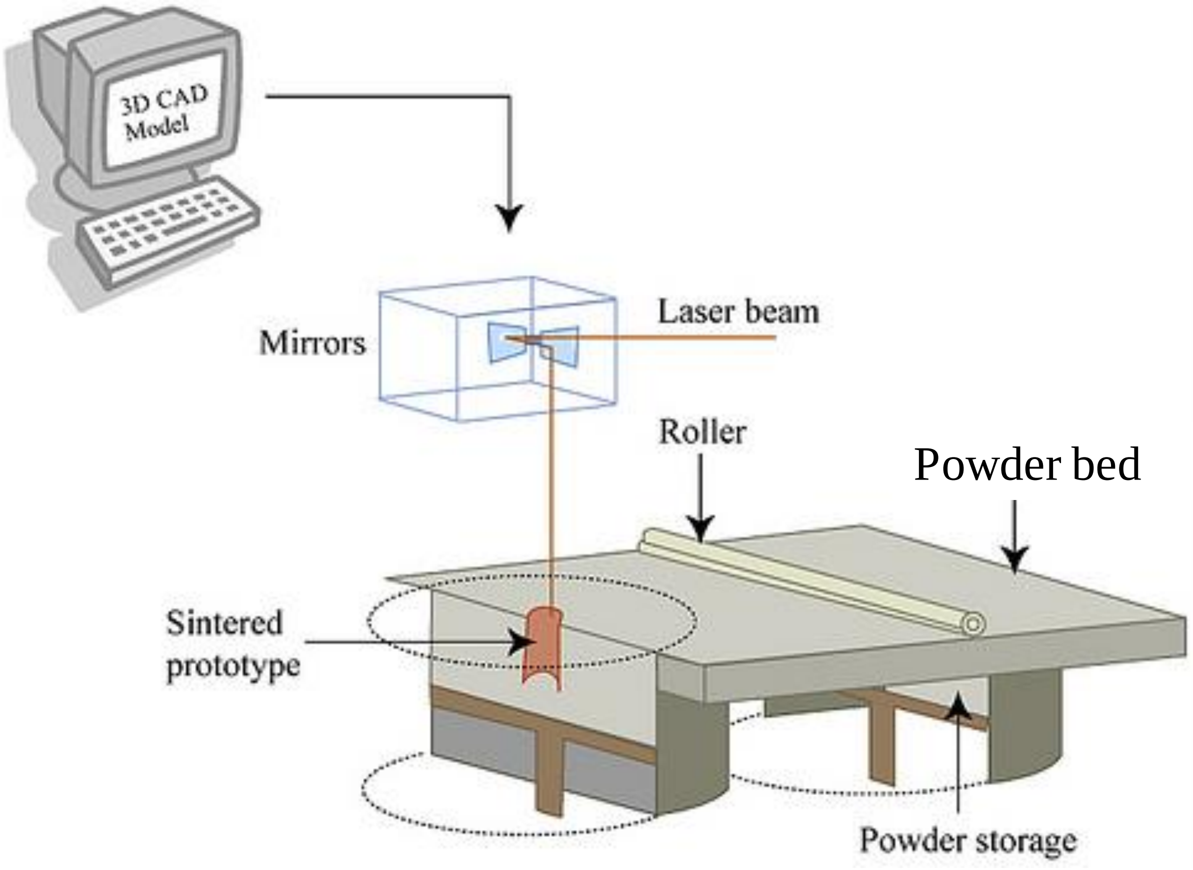
Element	Cr	Ni	C	Si	Mn	P	S	Mo	Fe
Weight %	16.8	10.8	0.02	0.47	1.35	0.015	0.03	2.13	Bal.

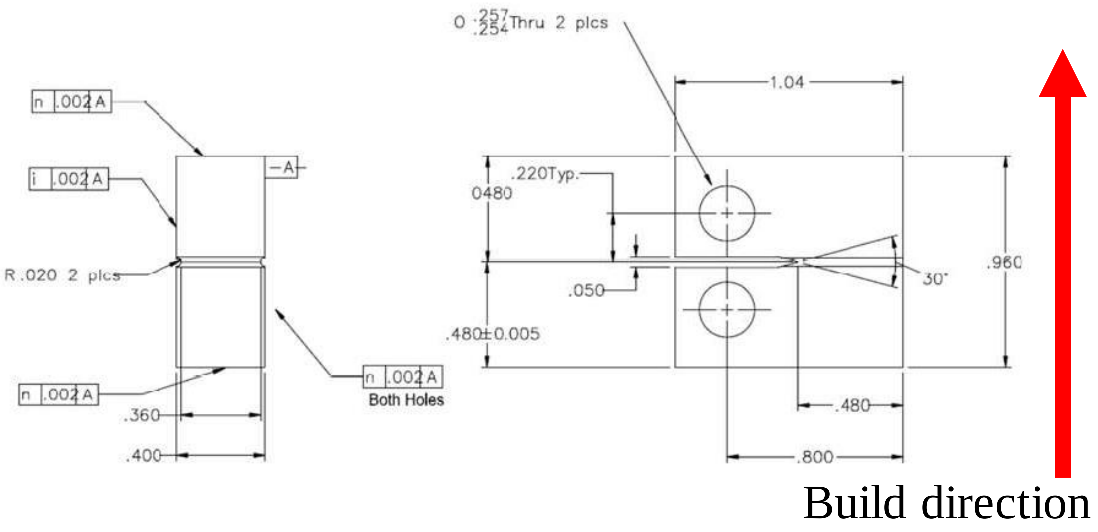
718 Powder Composition

Element	Cr	Ni	Nb	Ti	C	Si	Mn	P
Weight %	19.4	52.4	4.97	0.98	0.04	0.1	0.06	0.004

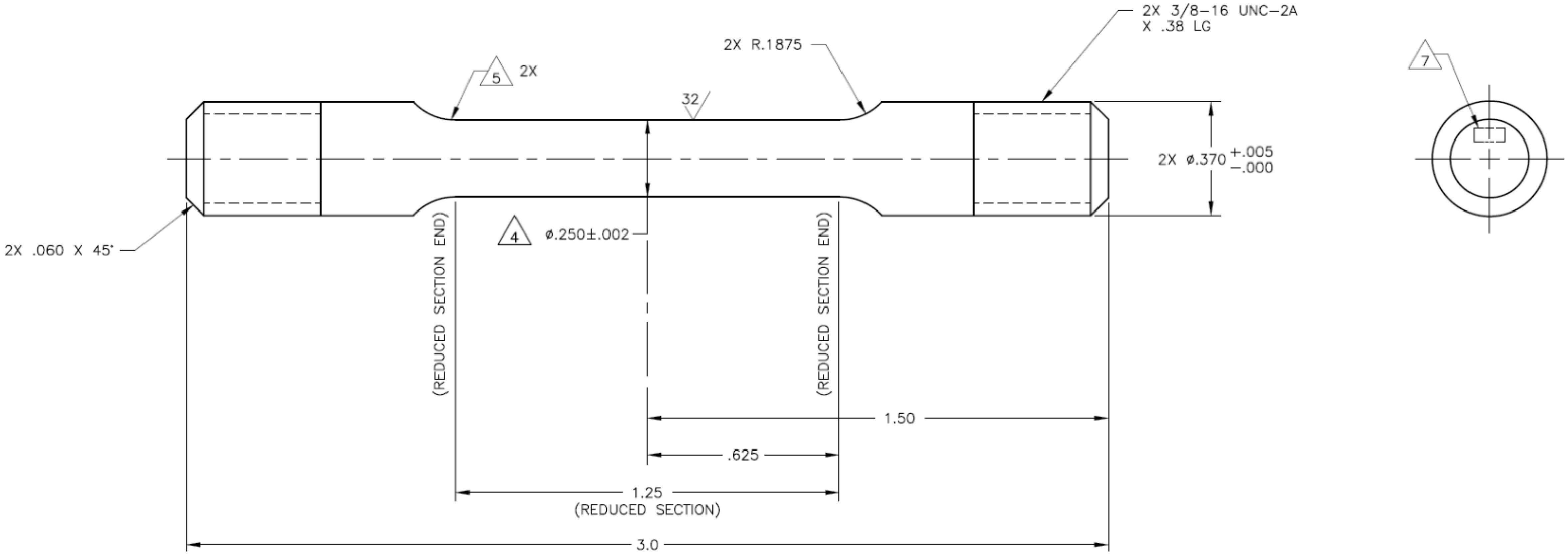
Element	Mo	Al	B	Cu	Co	Ta	Fe
Weight %	2.96	0.41	0	0.09	0.09	0.03	Bal

Direct Metal Laser Melting and Heat Treatment

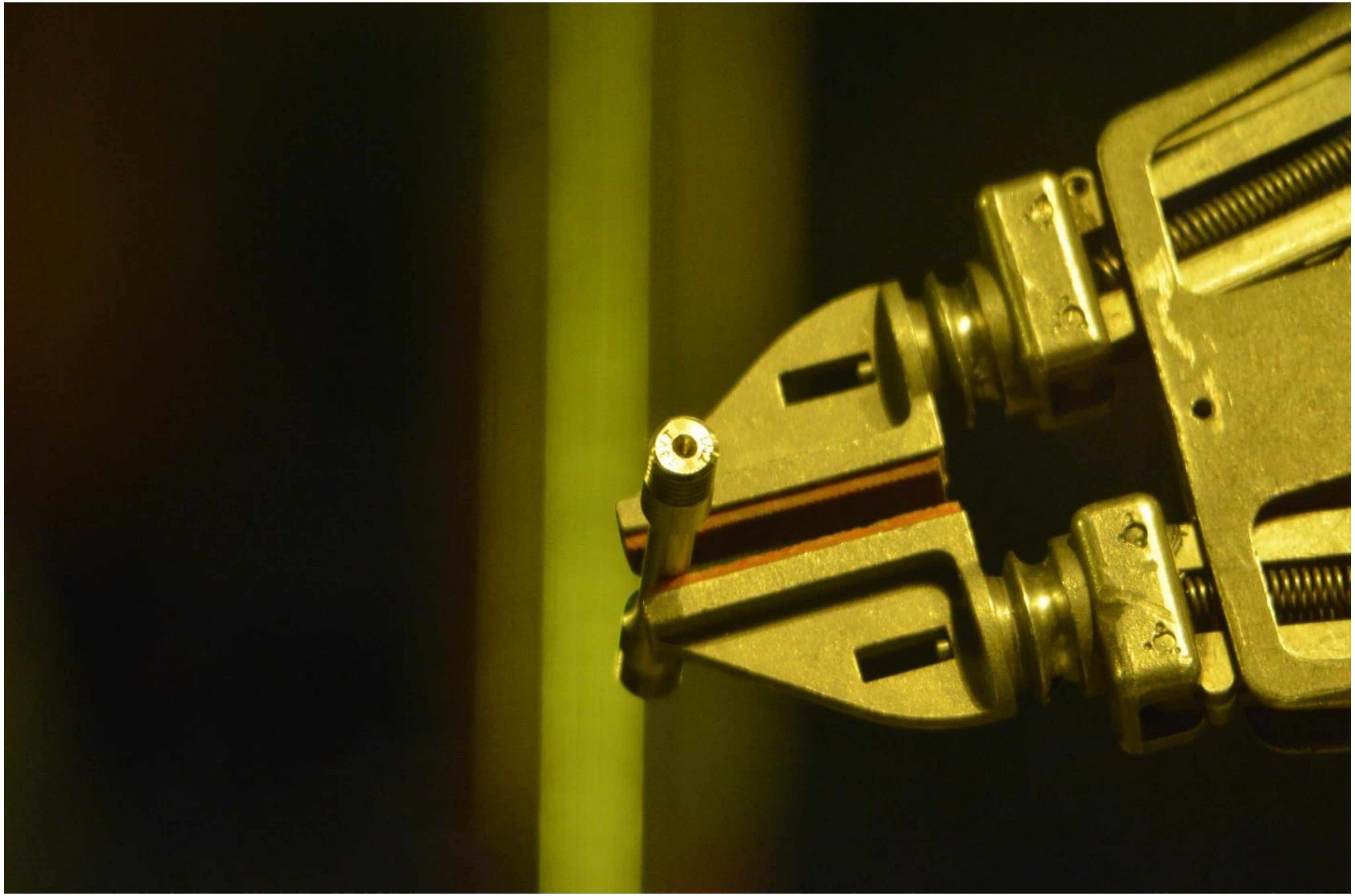




Tensile Specimen Geometry



Tensile Specimen Geometry



Direct Metal Laser Melting and Heat Treatment

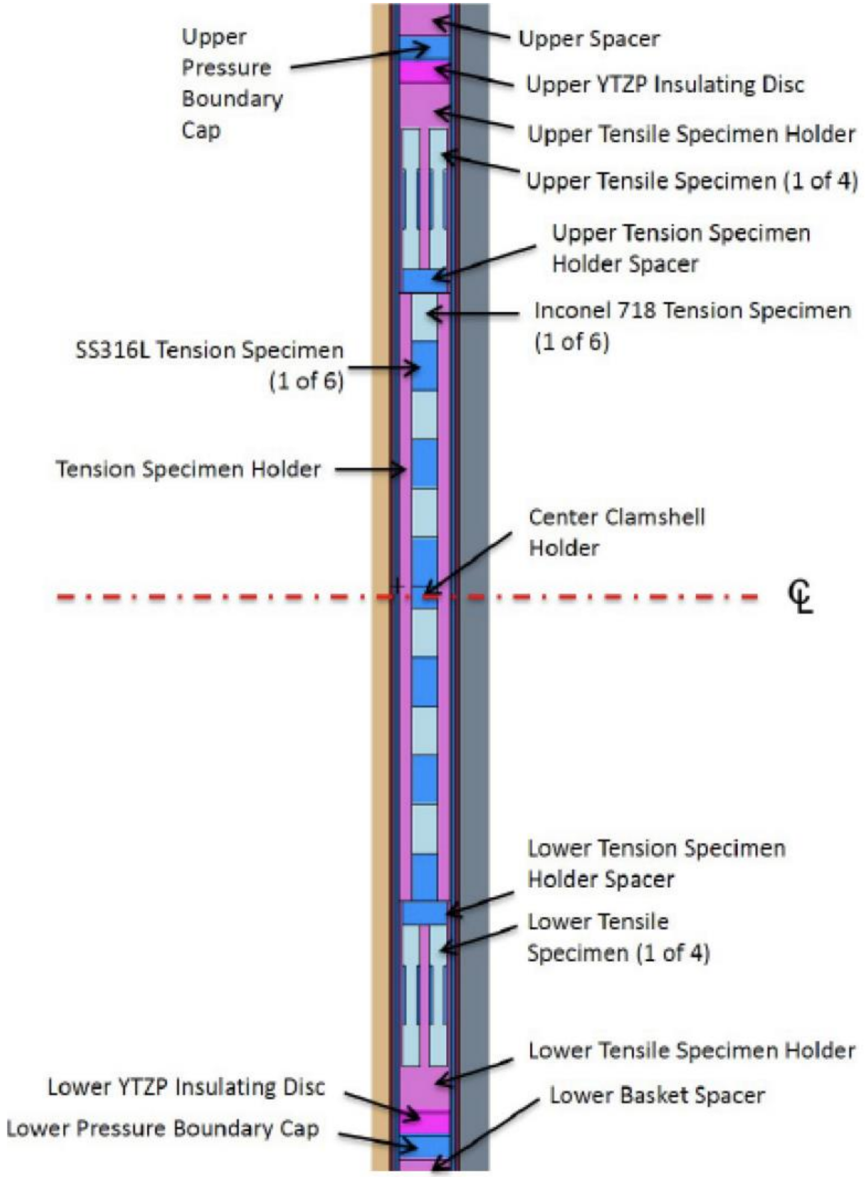
AM316L

Heat Treatment	Temperature [°C]	Time	Atmosphere
Stress Relief	1038 min	2 hrs	inert
HIP	1121-1163	4±1 hrs	inert, min 100 MPa
Solution Anneal	1066 min	1-2 hrs	inert

AM718

Heat Treatment	Temperature [°C]	Time	Atmosphere
Stress Relief	1065±15	1.5 hrs	inert
HIP	1120-1185	4±1 hrs	inert, min 100 MPa
Solution Anneal	1010-1050	1-2 hrs	inert
Precipitation Hardening	704±8	6 hrs	inert

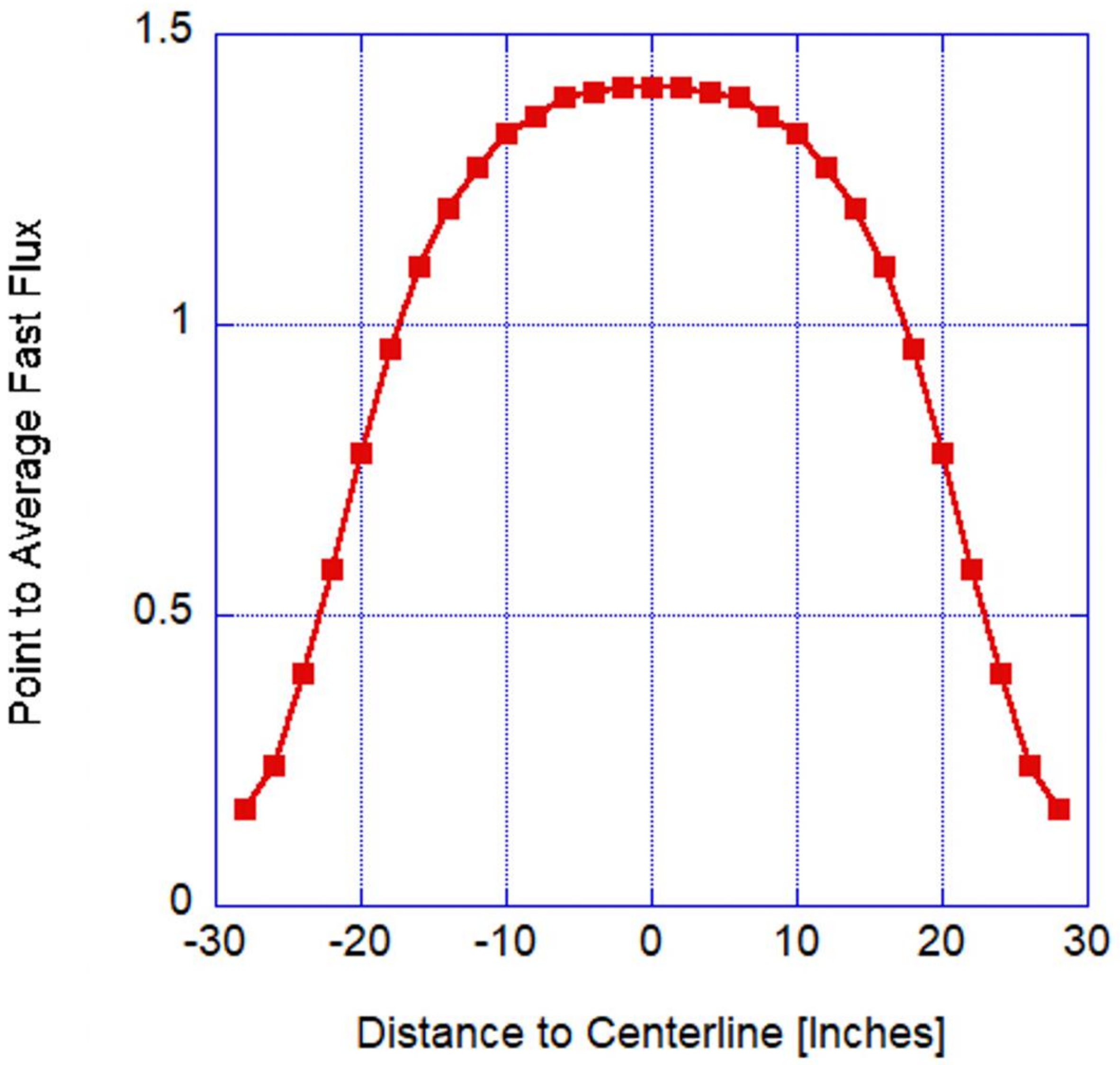
Capsule Loading of Tensile and 0.4T-CT Specimens



ATR Cycle 162A-164B Calculated Dose

Material	Cycle:	162A	162B	164A	164B	Total
	EFPDs:	61.90	38.51	54.91	64.06	219.38
316L samples	10VT (Tensile)	0.22	0.13	0.18	0.22	0.74
	11VT (Tensile)	0.25	0.14	0.21	0.25	0.85
	GEH 1-1B	0.26	0.15	0.21	0.25	0.86
	GEH 3-1D	0.26	0.15	0.22	0.25	0.88
	GEH 4-1A	0.28	0.15	0.23	0.26	0.92
	GEH 4-1B	0.31	0.18	0.26	0.30	1.05
	GEH 5-1C	0.31	0.17	0.25	0.30	1.04
	GEH 5-1D	0.30	0.17	0.24	0.28	0.99
	15VT (Tensile)	0.26	0.15	0.22	0.25	0.88
	12VT-1 (Tensile	0.23	0.13	0.19	0.23	0.78
718 samples	8VT (Tensile)	0.29	0.17	0.24	0.29	0.99
	12VT-2 (Tensile)	0.26	0.14	0.21	0.25	0.86
	GE 41-2A	0.30	0.17	0.24	0.29	0.99
	GE 41-2D	0.30	0.17	0.25	0.29	1.01
	GE 46-2B	0.31	0.18	0.26	0.30	1.05
	GE 46-2C	0.27	0.15	0.22	0.26	0.92
	GE 51-2A	0.27	0.15	0.22	0.26	0.91
	GE 60-2C	0.26	0.15	0.22	0.25	0.88
	73VT (Tensile)	0.31	0.17	0.25	0.30	1.03
	79VT (Tensile)	0.27	0.15	0.22	0.26	0.90

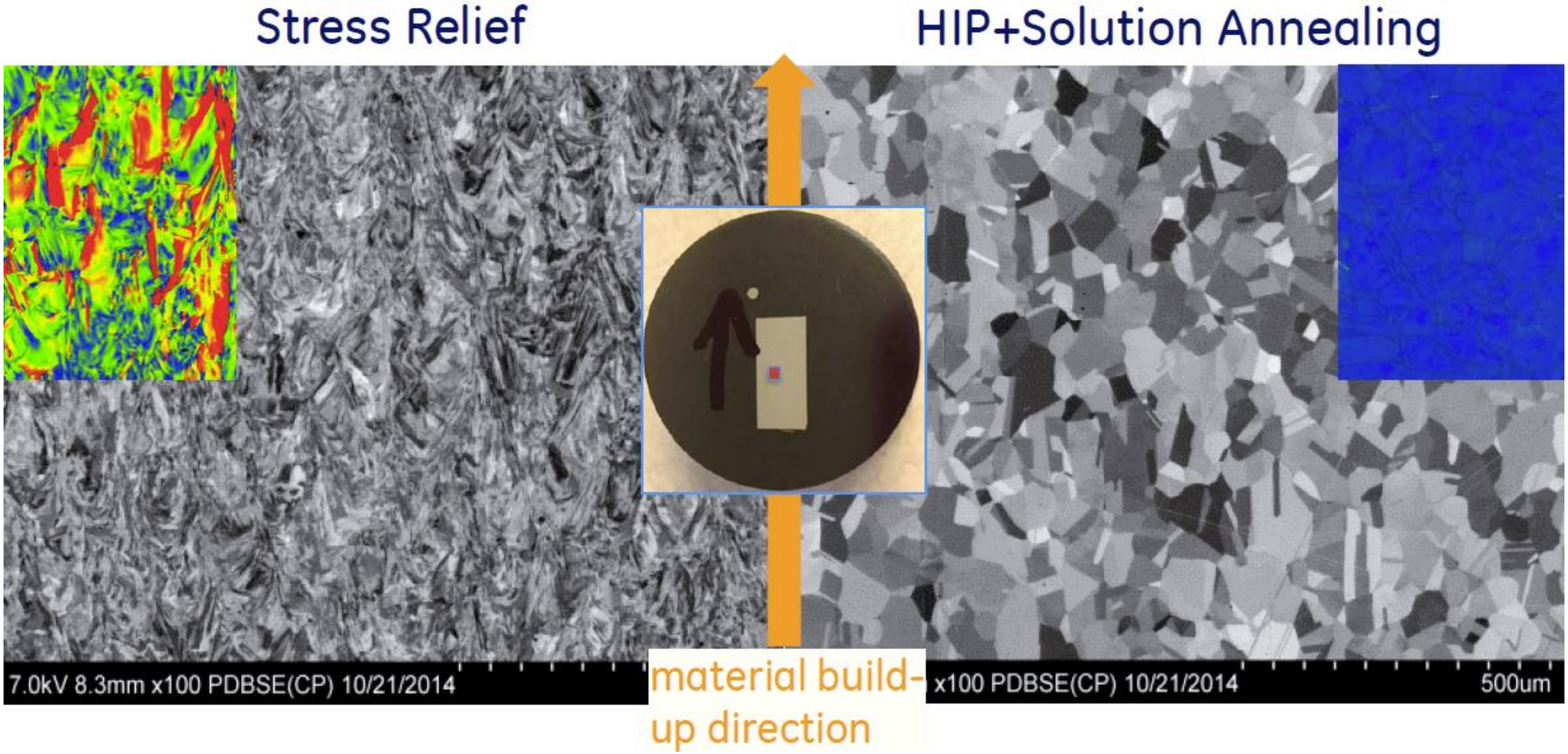
ATR Normalized Axial Flux Distribution



ATR Cycle 162A-164B Calculated Temperatures

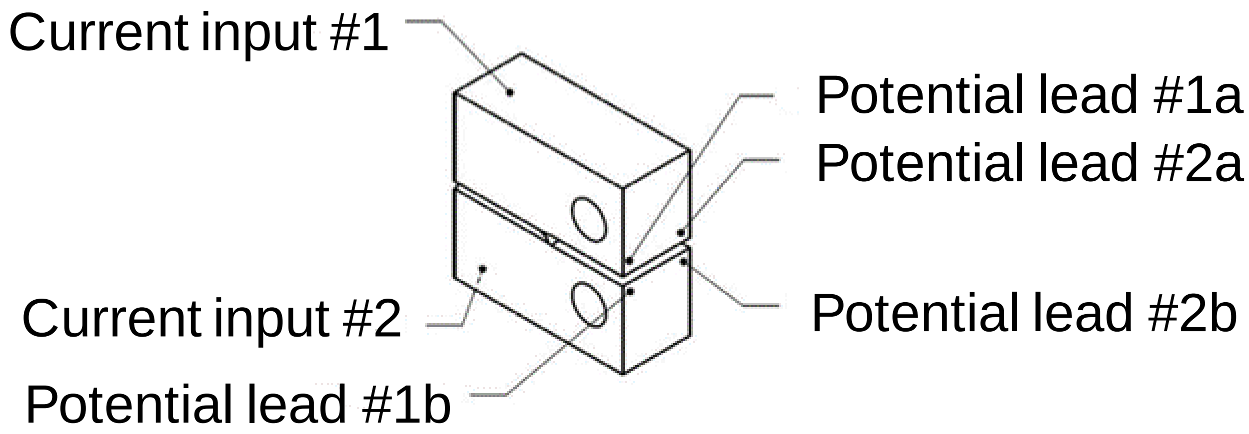
Specimen Material	Specimen ID	Specimen Temperature [°C]			
		162A	162B	164A	164B
316L Samples	10VT (Tensile)	291	274	276	280
	11VT (Tensile)	286	269	271	274
	GEH 1-1B	299	277	284	282
	GEH 3-1D	296	276	281	280
	GEH 4-1A	295	278	280	283
	GEH 4-1B	300	281	284	286
	GEH 5-1C	300	282	285	287
	GEH 5-1D	306	285	289	291
	15VT (Tensile)	292	274	277	279
	12VT-1 (Tensile	288	268	272	274
718 Samples	8VT (Tensile)	290	269	275	274
	12VT-2 (Tensile)	296	273	281	279
	GE 41-2A	309	288	293	294
	GE 41-2D	303	283	287	288
	GE 46-2B	304	283	288	288
	GE 46-2C	305	286	290	292
	GE 51-2A	306	287	290	292
	GE 60-2C	310	290	294	296
	73VT (Tensile)	296	273	281	279
	79VT (Tensile)	290	269	275	274

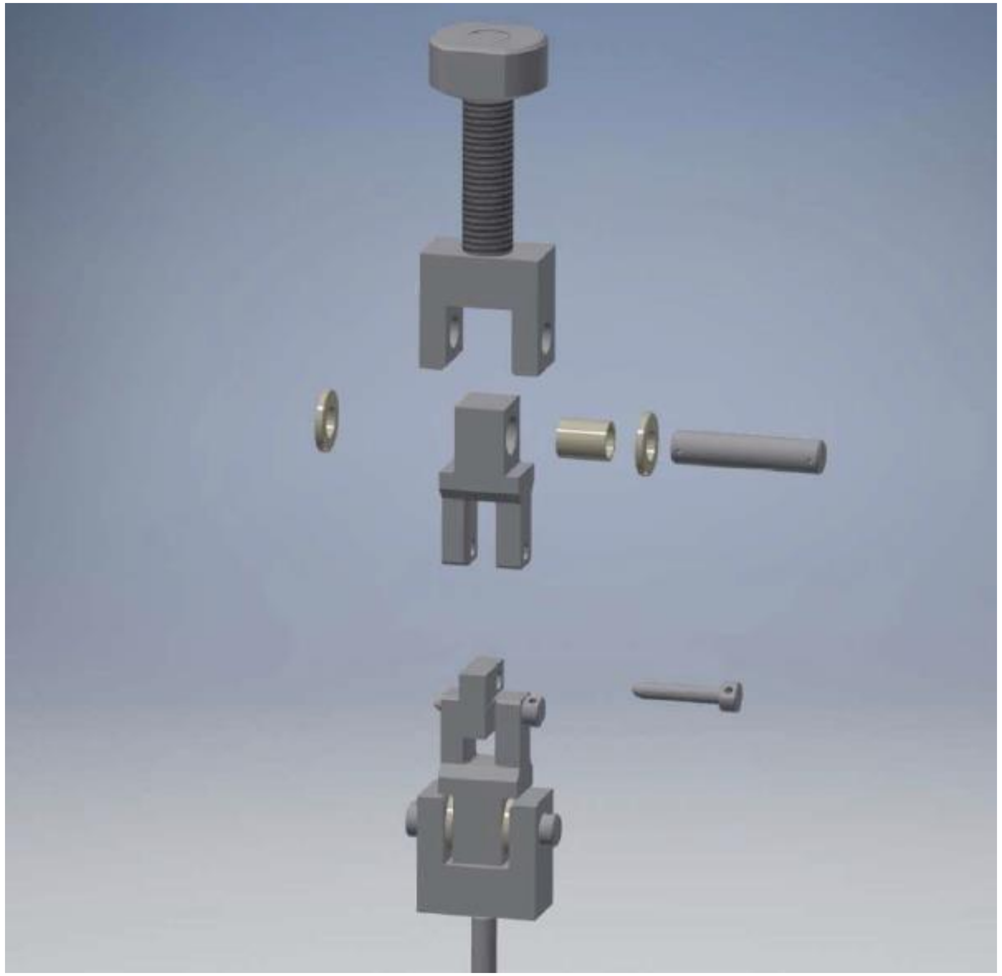
AM316L Microstructure Post Heat Treatment



CGR Testing of Irradiated Samples using DCPD

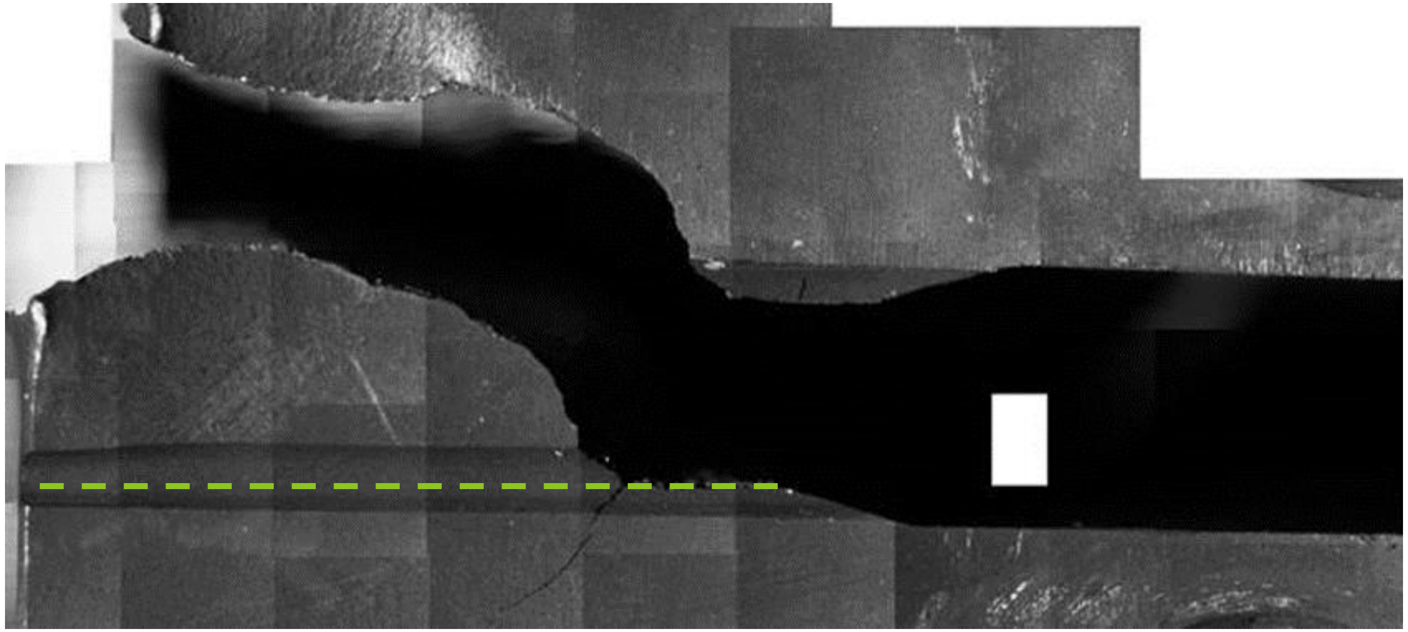
- 3 A direct current used for DCPD crack monitoring
- Water in autoclave during experiment kept at 288 °C, 1500 psig
- 10 ppb sulfate (as H_2SO_4) added to enhance reproducibility and allow for comparison with experiments using non-irradiated samples
- NWC: 2.5 ppm dissolved oxygen
- HWC: 60-90 ppb dissolved hydrogen

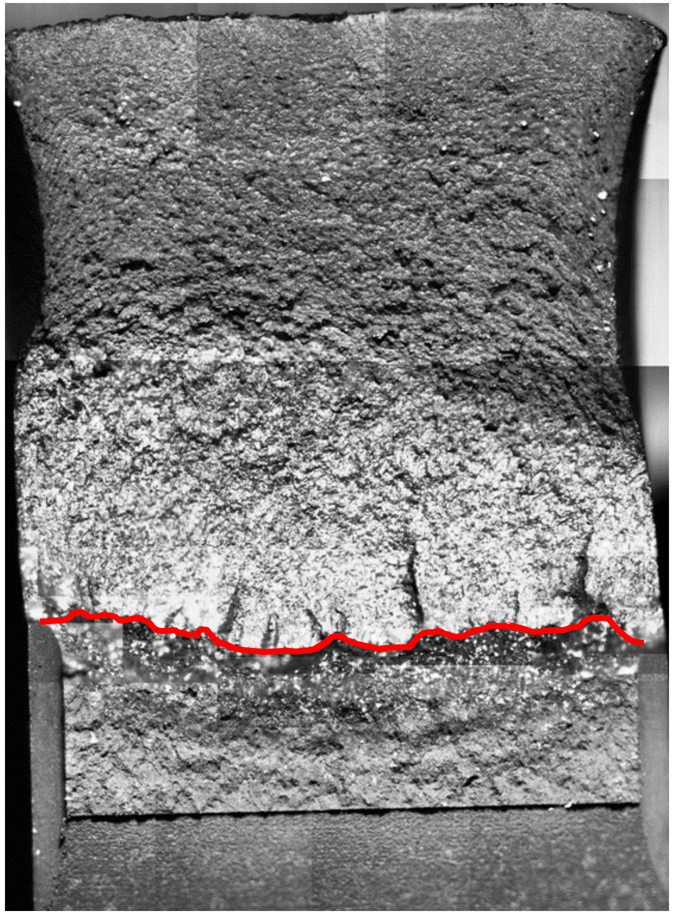




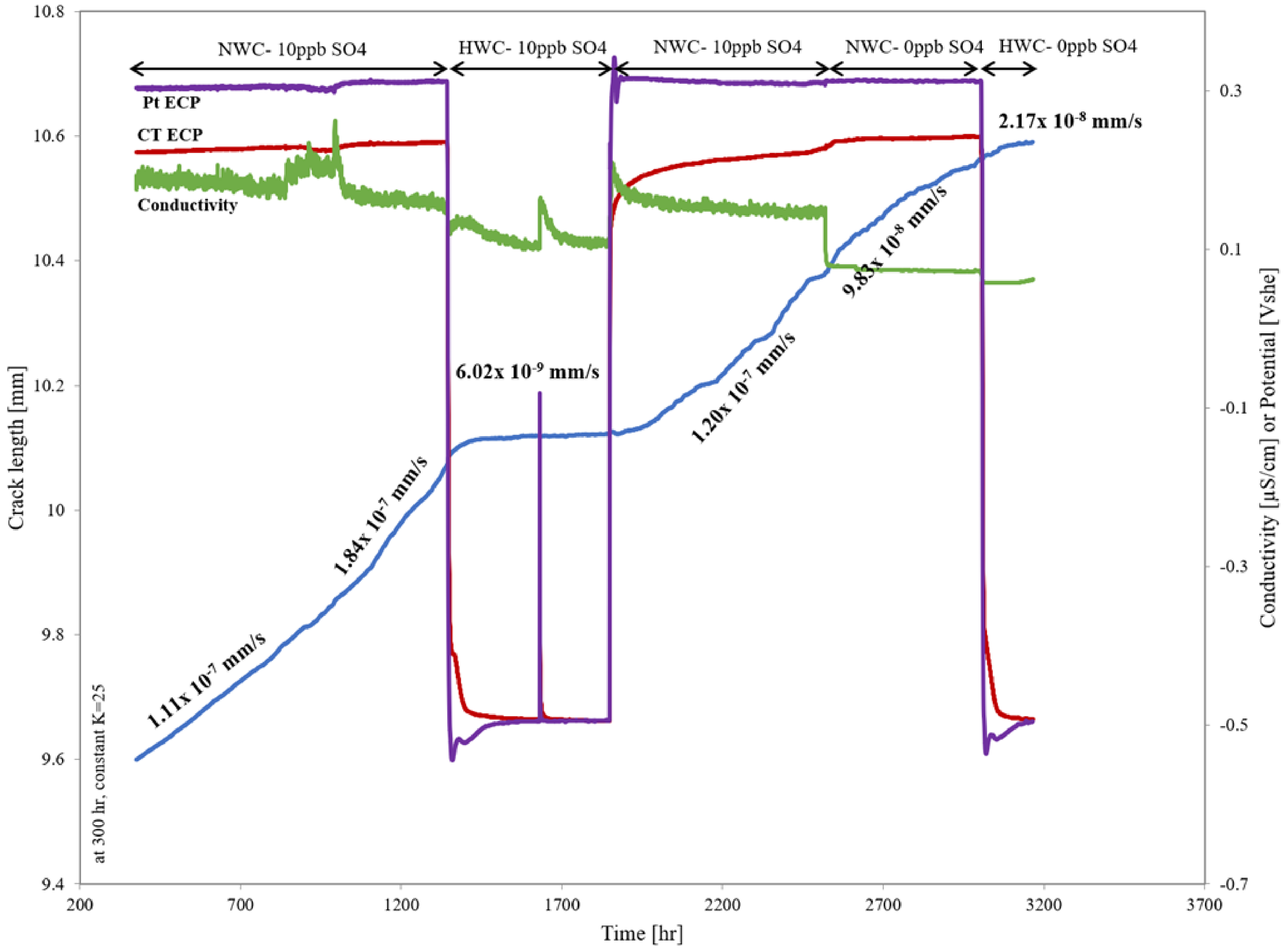
316L 0.89 dpa CGR (GEH31-D) - Results

Test Hours	K (MPa√m)	Chemistry	Sulfate	Conductivity (µS/cm)	Average CGR (mm/s)
300	25	NWC	10	0.14	1.84E-07
994	25	NWC	10	0.14	7.66E-08
1144	25	HWC	10	0.1	3.05E-10
1921	25	NWC	10	0.13	1.08E-07
2635	25	NWC	0	0.09	7.51E-08
3024	25	HWC	0	0.07	5.12E-10

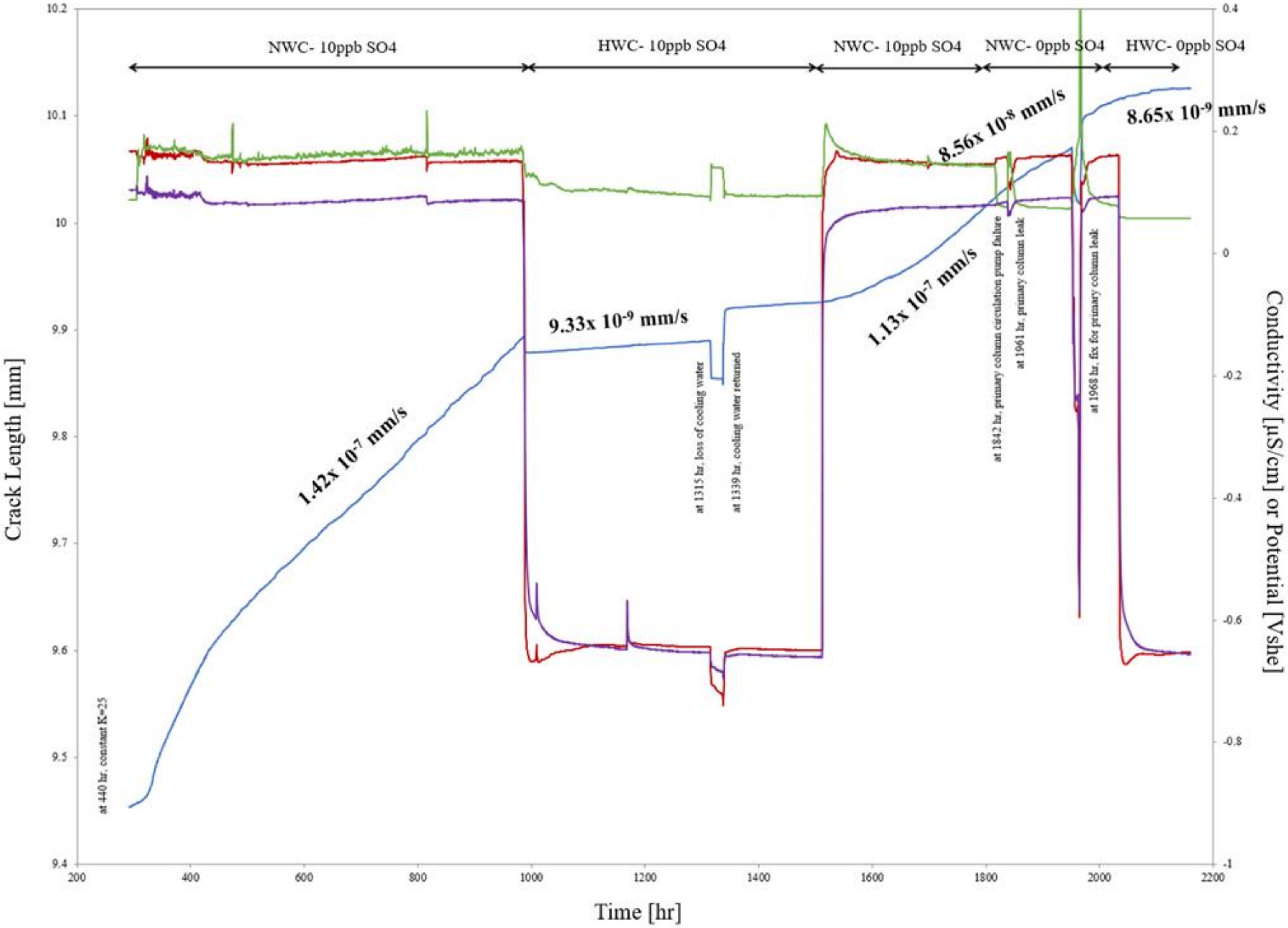




718 1.01 dpa CGR (GE41-2D) – Overview of CGR Test



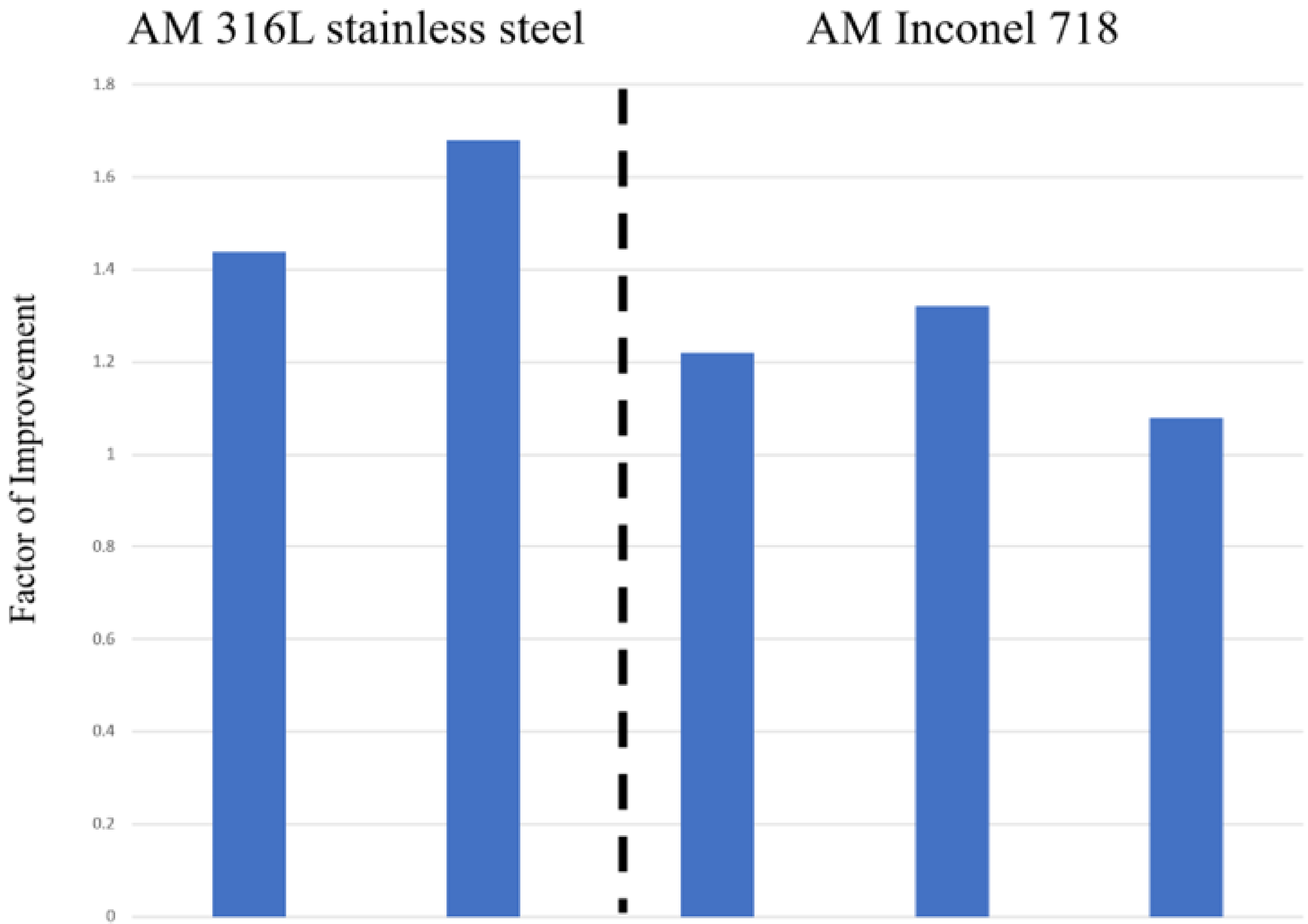
718 0.92 dpa CGR (GE46-2C) – Overview of CGR Test



718 0.92 dpa CGR (GE46-2C) - Results

Test Hours	K (MPa√m)	Chemistry	Sulfate	Conductivity (μS/cm)	Average CGR (mm/s)
440	25	NWC	10	0.16	1.42E-07
984	25	HWC	10	0.10	9.33E-09
1511	25	NWC	10	0.15	1.13E-07
1816	25	NWC	0	0.15	8.56E-08
2033	25	HWC	0	0.06	8.65E-09

Effects of Sulfate Additions on CGR

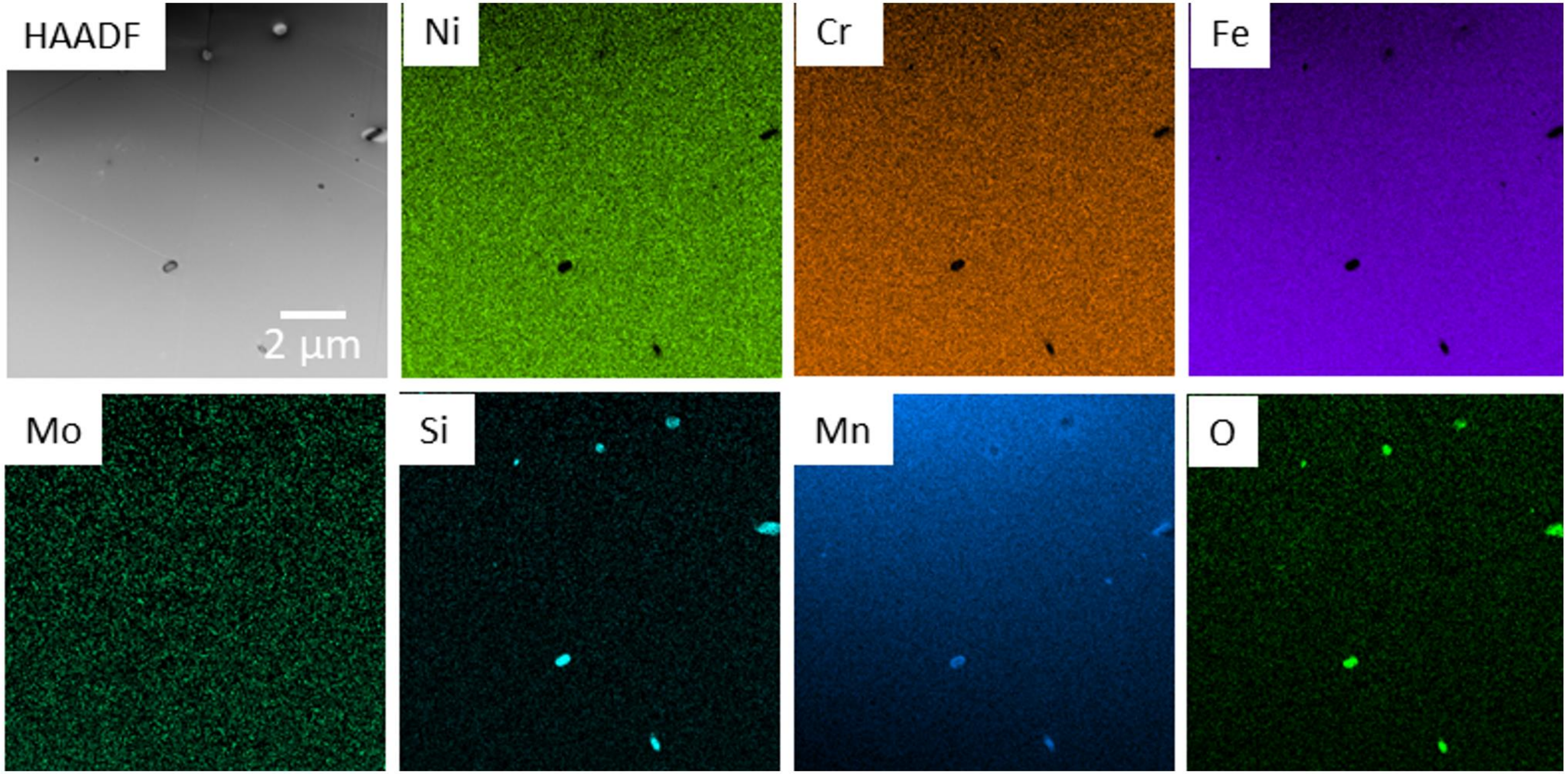


- Data represents results from both 316L and 718 samples
- Average reduction in CGR due to changing sulfate additions for 316L=1.56x
- Average reduction in CGR due to changing sulfate additions for 718=1.21x

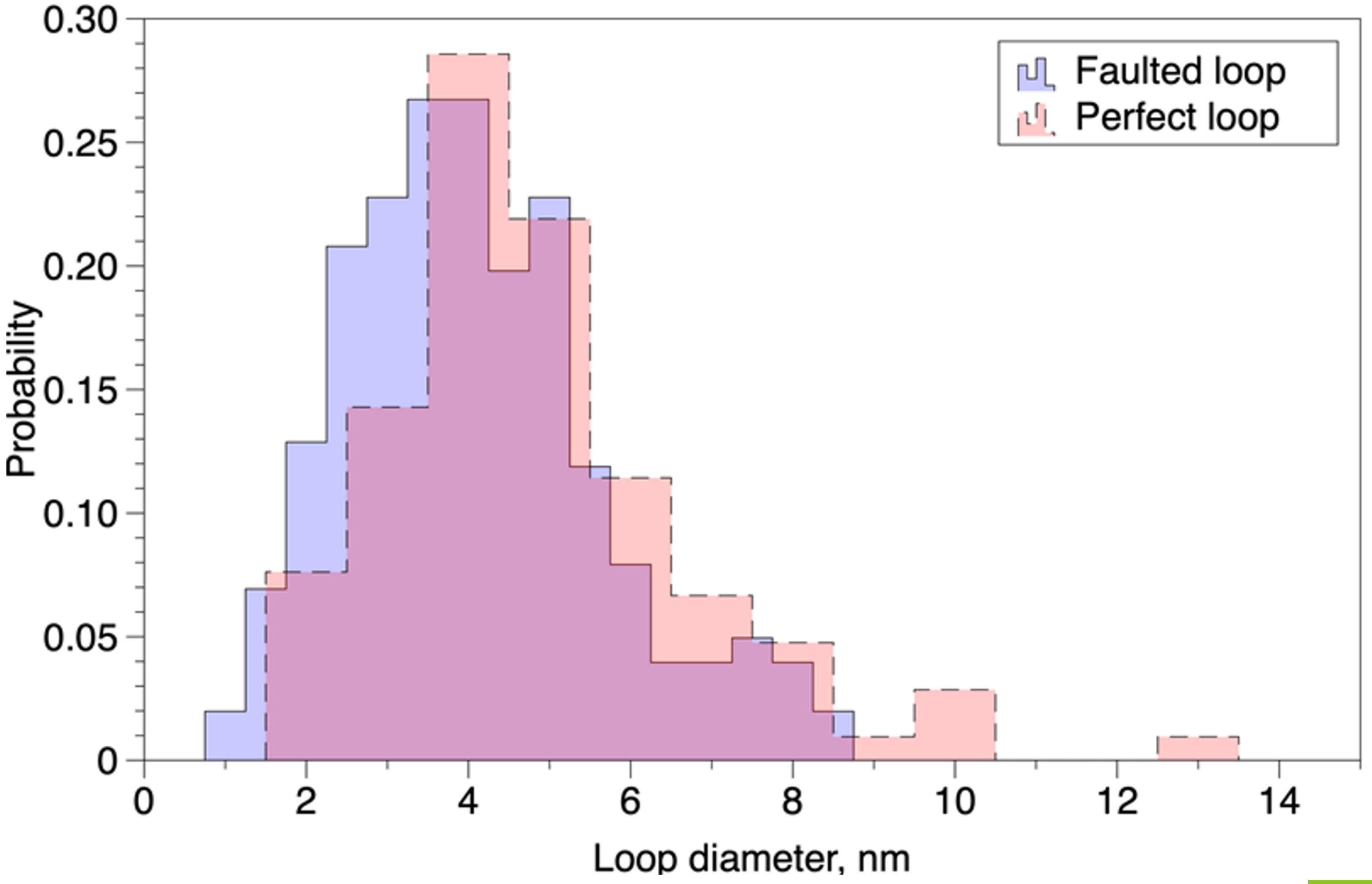
ASTM Fracture Toughness Validity Checks

Specimen ID	Material	Dose [dpa]	Crack Size		Crack Extension		J-R Curve		Qualification
			9.1.4.1	9.1.4.2	9.1.5.1	9.1.5.2	A9.6.4	A9.6.6.6	A9.10
GEH 1-1B	AM 316L	0.86	Yes	No	Yes	No	Yes	Yes	Yes
GEH 5-1D	AM 316L	0.99	Yes	No	Yes	No	Yes	Yes	Yes
GE 46-2B	AM 718	1.05	Yes	No	Yes	No	Yes	Yes	Yes
GE 51-2A	AM 718	0.91	Yes	No	Yes	No	Yes	Yes	Yes

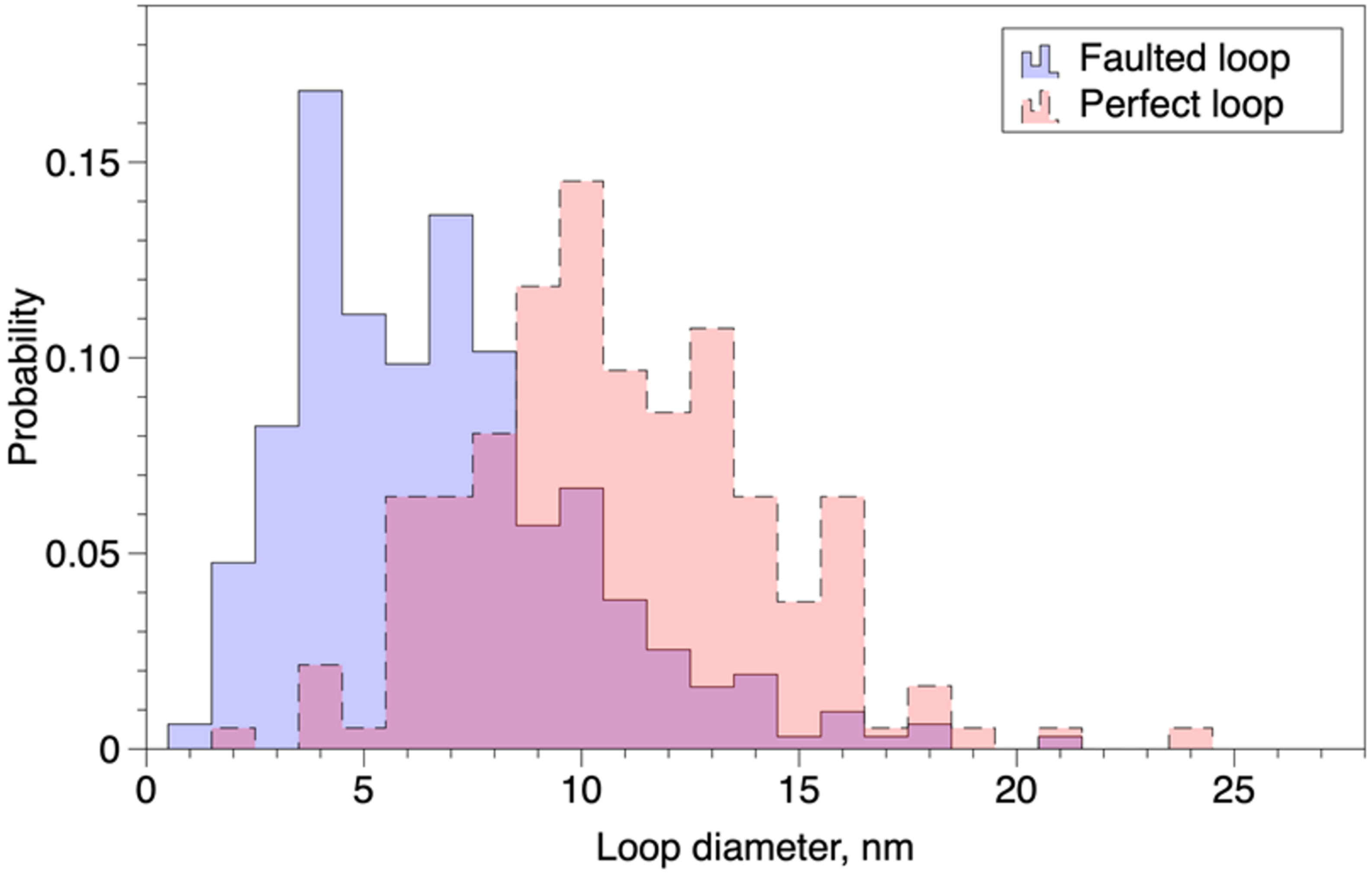
HAADF AM316L Showing Oxide Precipitates



Irradiated AM 316L Loop Distribution



Irradiated AM 718 Loop Distribution



Void Formation in Neutron Irradiated AM 718