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Mechanical Characterization of Additively Manufactured Inconel 625 and AlSi10Mg via High-Throughput Tensile Testing

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Need for Study

- Benefits of Additive Manufacturing
  - Intricate designs
  - Manufacturing flexibility
  - Low cost
  - Fast turnaround

- Limitations
  - Residual stresses
  - Part to part variability
  - Meeting tolerances
  - Identification of outliers

- Precipitation kinetics of heat treated Inconel 625

- AlSi10Mg porosity susceptibility

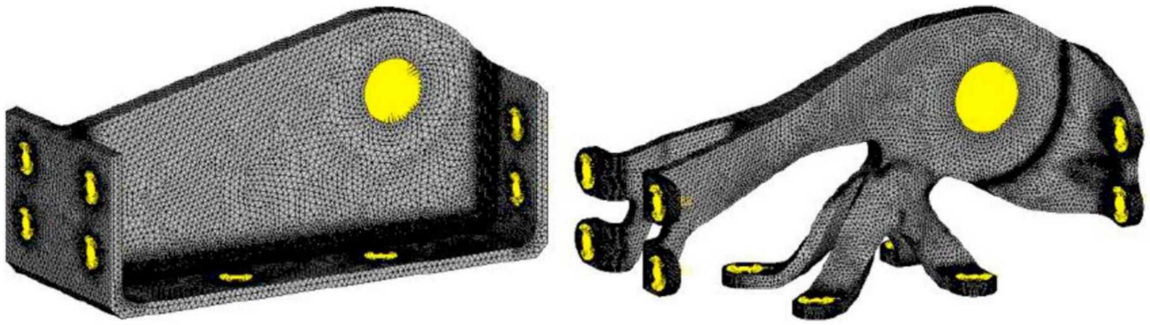
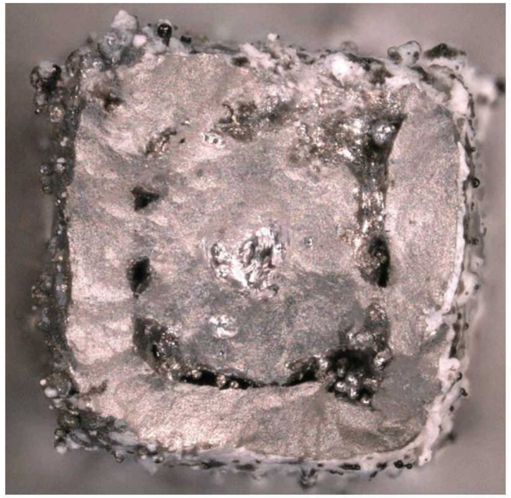


Image courtesy of: Aboulkhair NT, Simonelli M, Parry L, Ashcroft I, Tuck C, Hague R: **3D printing of Aluminium alloys: Additive Manufacturing of Aluminium alloys using selective laser melting.** *Progress in Materials Science* 2019, **106**.

Focus of Study & Outline

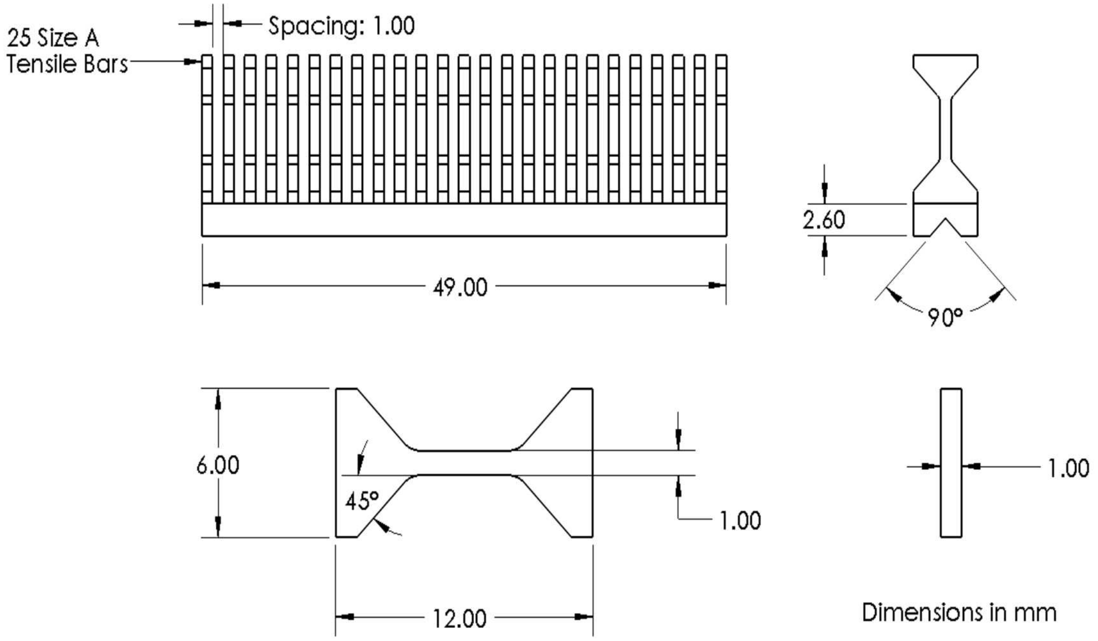
- High-throughput tensile testing (rapid characterization)
  - Inconel 625 & AlSi10Mg
- AM acceptance testing procedure
  - AlSi10Mg
- Influence of heat treatment on Inconel 625 mechanical properties
- Porosity effects on AlSi10Mg

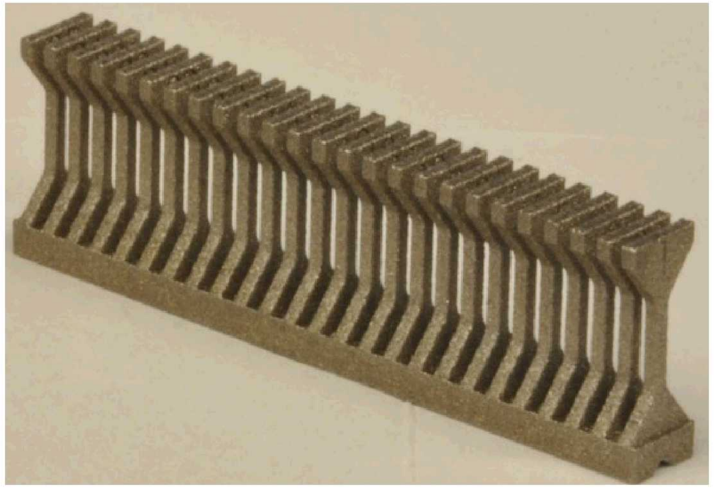


Powder-bed additive manufacturing

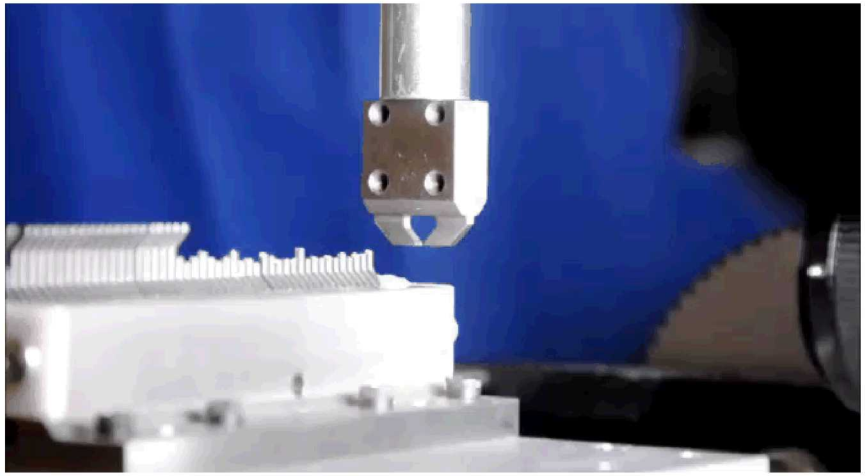
High-throughput specimen design

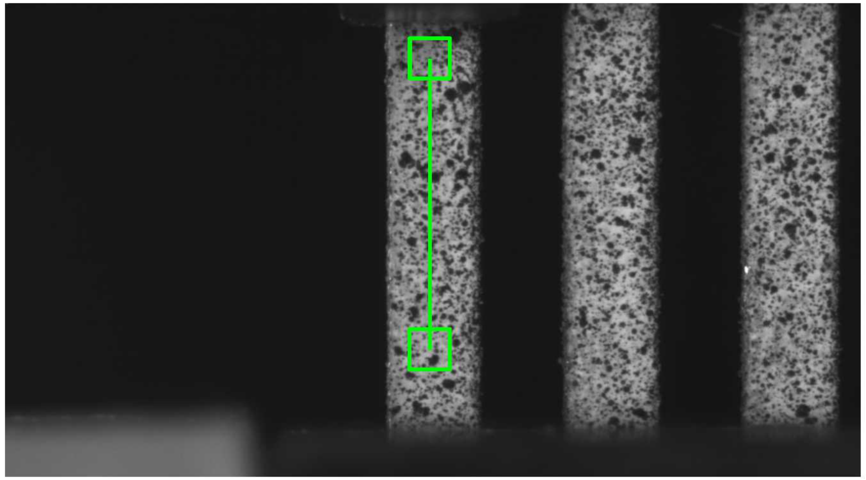
- 1mm x 1mm nominal cross section
- 4-to-1 gauge length ratio to reduce premature necking
- 45° grip head to reduce overhang

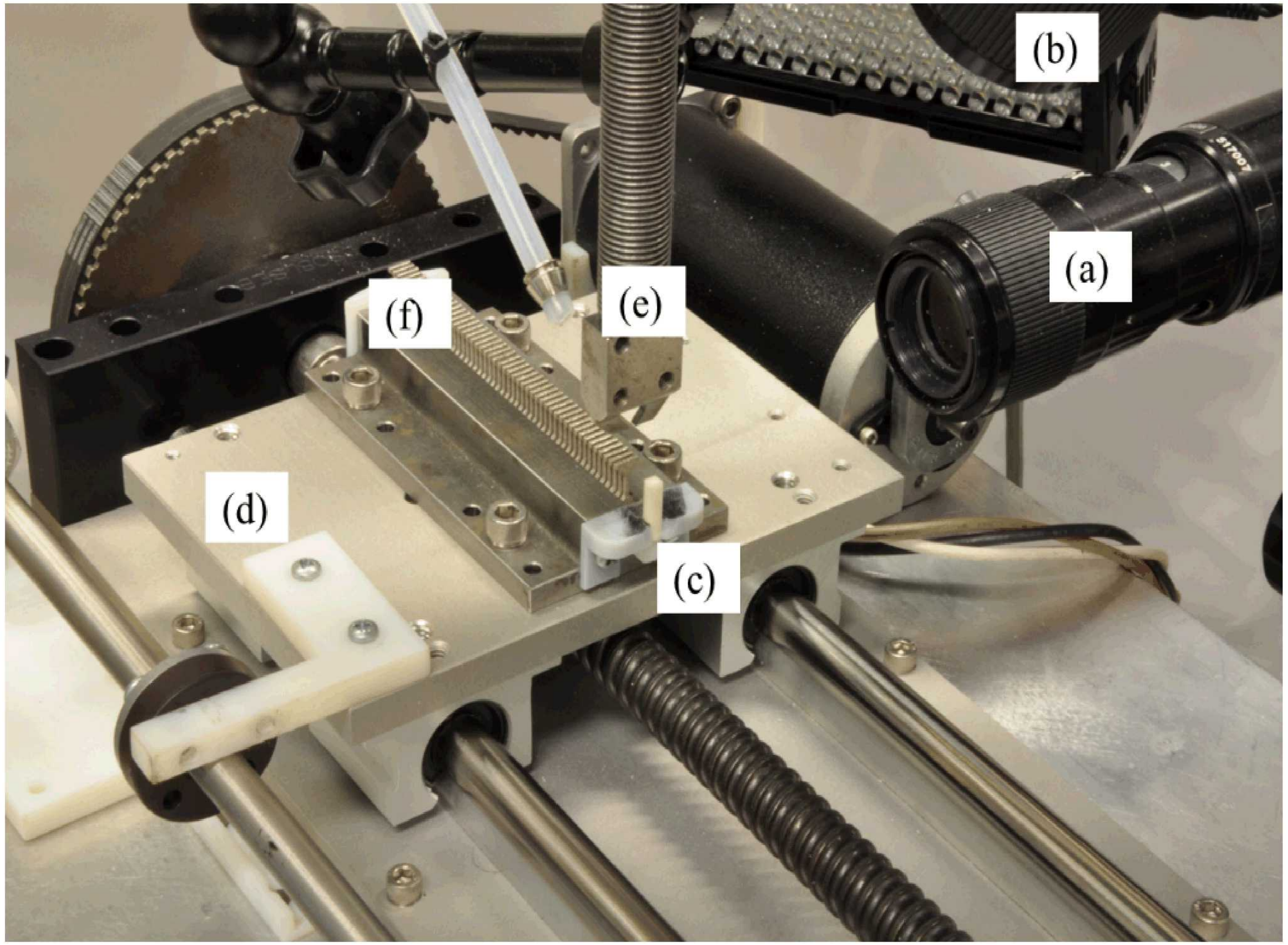




High-throughput tensile test setup



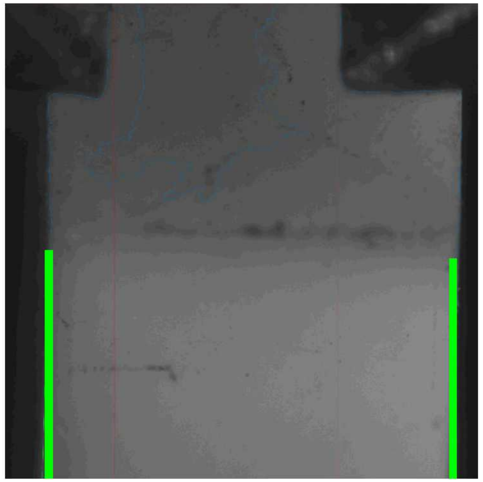
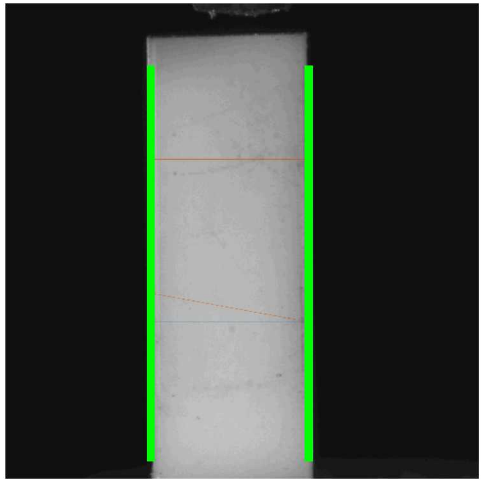
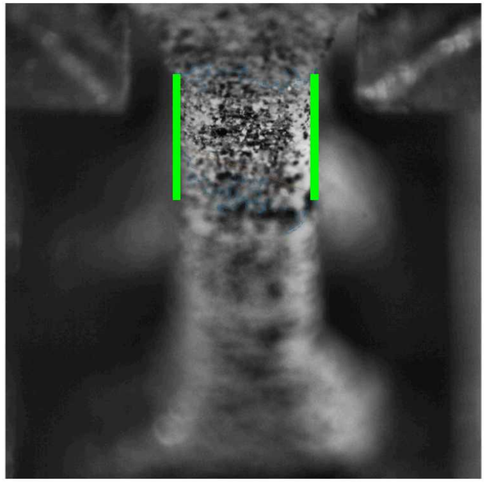
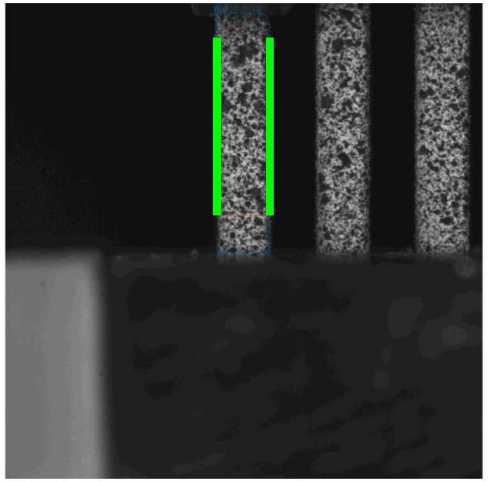




Salzbrenner BC, Rodelas JM, Madison JD, Jared BH, Swiler LP, Shen Y-L, Boyce BL: **High-throughput stochastic tensile performance of additively manufactured stainless steel.** *Journal of Materials Processing Technology* 2017, **241:**1-12.

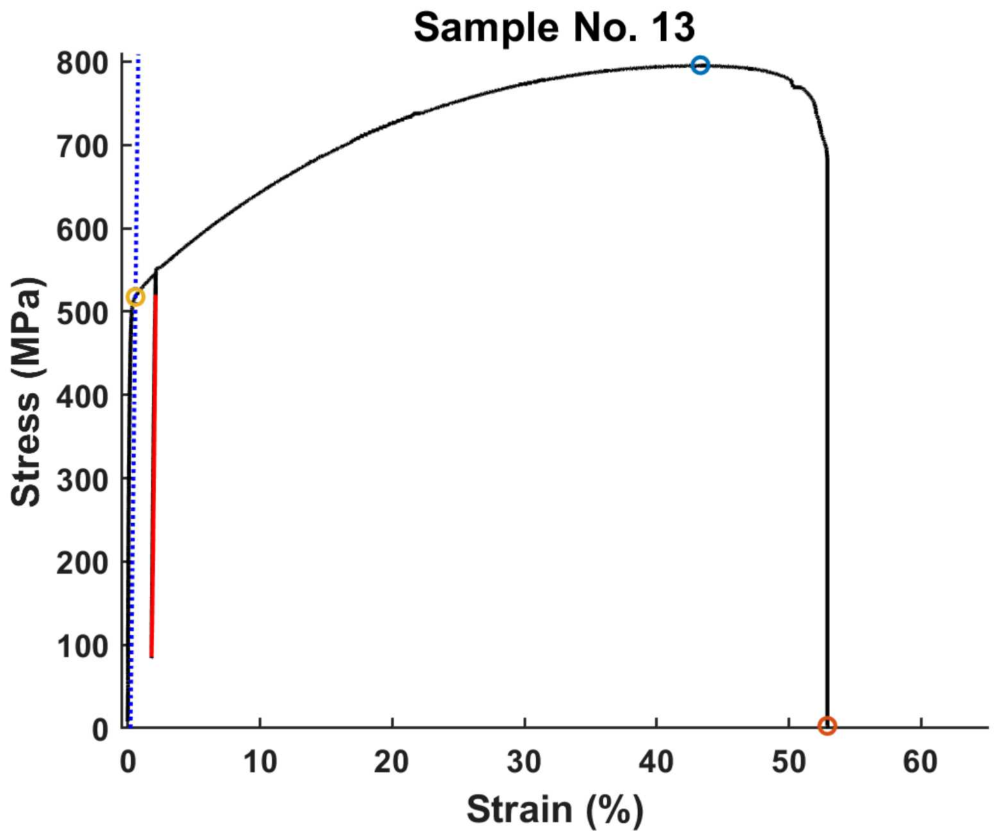
MatLab Analysis – AVID: Area Values for Image Dimensions ©

- Edge detection via MatLab image processing toolbox
- 1^{st} degree polynomial line fit
- Contact measurement vs digital measurement error: 3% ± 2% over-estimation

MatLab Analysis – BATS: Batch Analysis of Tensile Specimens ©

- Application of strain scalar to produce strain values
- Output Results
  - Unloading Modulus
  - 0.2% Yield Stress
  - 0.2% Yield Strain
  - Ultimate Tensile Strength
  - Uniform Elongation
  - Elongation at Failure
- Additional Results
  - Yield Stress & Strain from published modulus
  - Toughness (area under curve)



AM acceptance testing used to predict AM build performance

- Density
- Hardness
- Charpy impact toughness

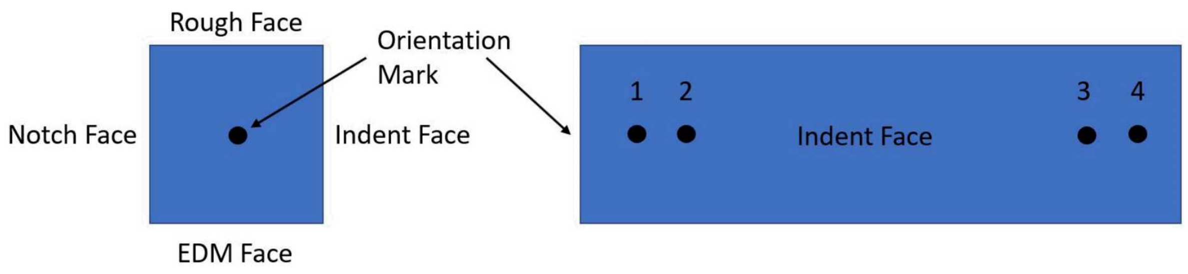


Image Courtesy of Mettler Toledo

Inconel 625 powder characterization

- Minimal variation in powder chemical composition
- Builds 1 & 2 had higher oxygen levels and increased pressure
- Filter cleaned prior to Build 3

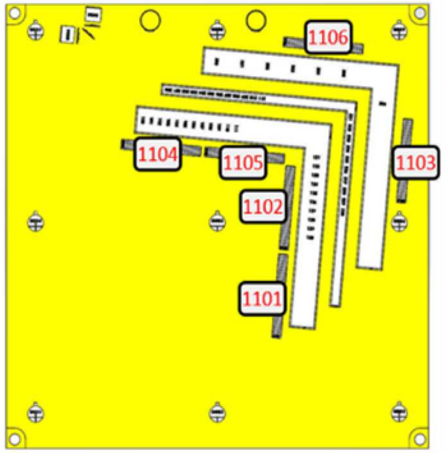
Composition	Units	ASTM F3056	Build 1 (virgin)	Build 2 (virgin)	Build 3 (reuse)
Al	Wt %	0.4 max	0.16	0.16	0.15
B	Wt %	-	<0.0005	<0.0005	<0.0005
C	Wt %	0.10 max	0.035	0.034	0.036
Co	Wt %	1.00 max	0.215	0.21	0.205
Cr	Wt %	20.00-23.00	21.39	21.22	21.23
Fe	Wt %	5.00 max	0.044	0.39	0.063
Mn	Wt %	0.50 max	0.0061	0.0064	0.0076
Mo	Wt %	8.00-10.00	8.81	8.68	8.75
Nb	Wt %	3.15-4.15	3.67	3.62	3.67
Ni	Wt %	Bal	65.3	65.8	65.6
P	Wt %	0.015 max	0.004	0.004	0.003
S	Wt %	0.015 max	0.0013	0.0012	0.0011
Si	Wt %	0.50 max	0.043	0.044	0.049
Ta	Wt %	-	<0.002	<0.002	<0.002
Ti	Wt %	0.40 max	0.12	0.12	0.13
O	Wt %	-	0.012	0.01	0.007
N	Wt %	-	0.006	0.006	0.007
H	Wt %	-	0.0007	0.0007	0.0009

	Build 1	Build 2	Build 3
Max Oxygen Level (ppm)	8.0	4.5	2.8
Pressure (mbar)	84	93	36

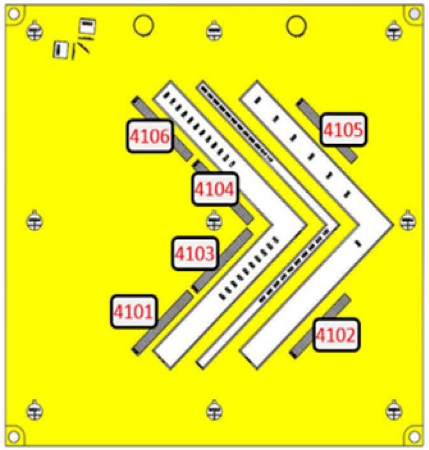
Inconel 625 heat treatment schedule and orientation

- Stress relieved with all samples attached to build plate
- Portion of virgin build solution annealed
- Portion of virgin build HIPed
- All reused powder stress relieved

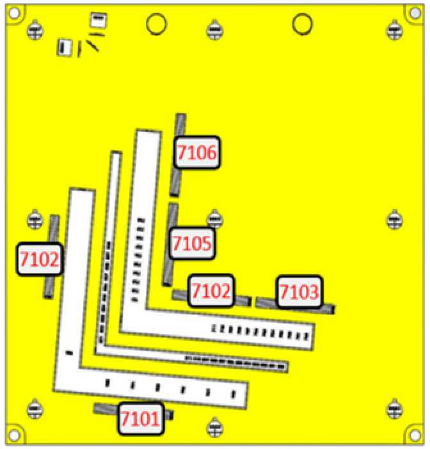
Heat Treatment	Schedule	Standard
Stress Relief (SR)	1040°C ± 15°C x 60 min ± 6 min	AMS 2774 E



Build 1

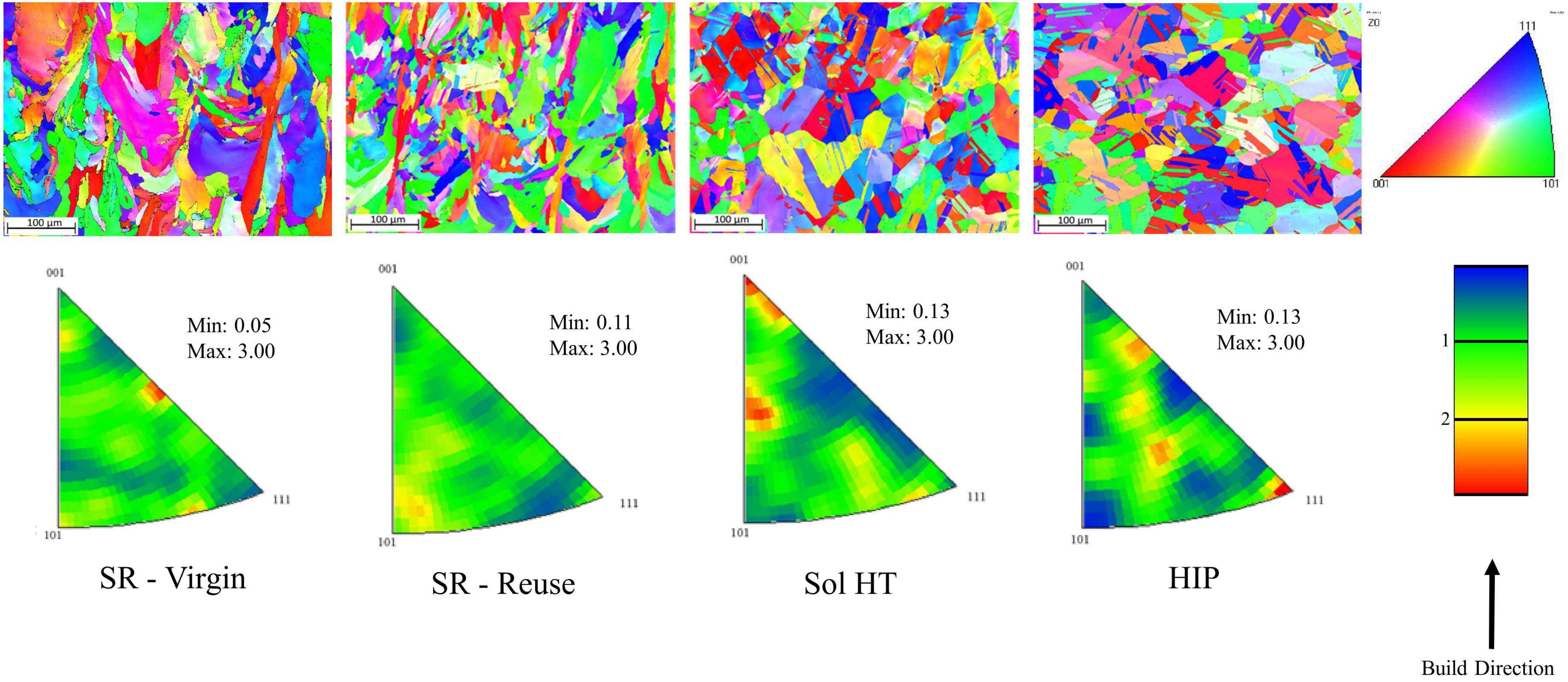


Build 2

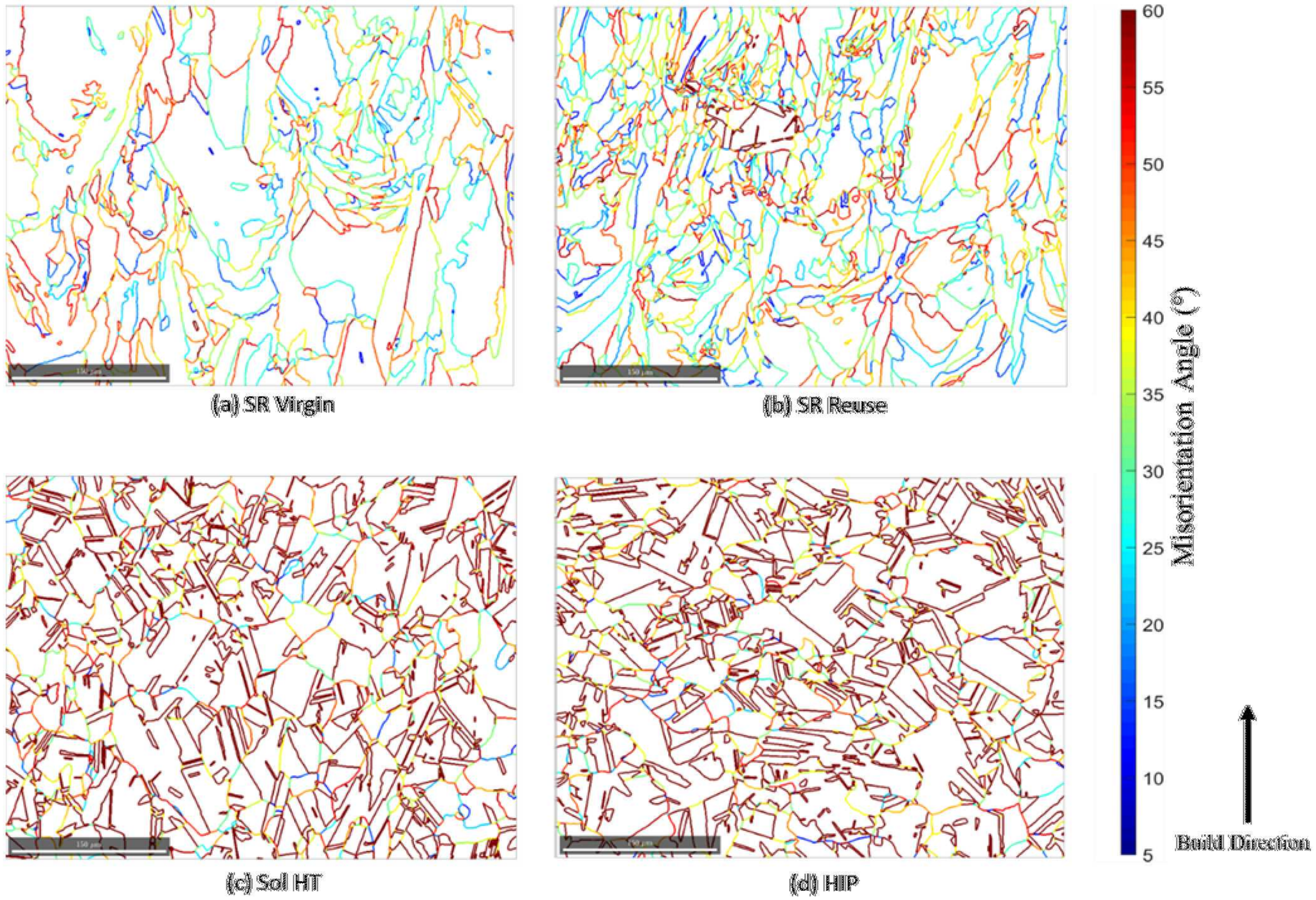


Build 3

Determination of Inconel 625 microstructure



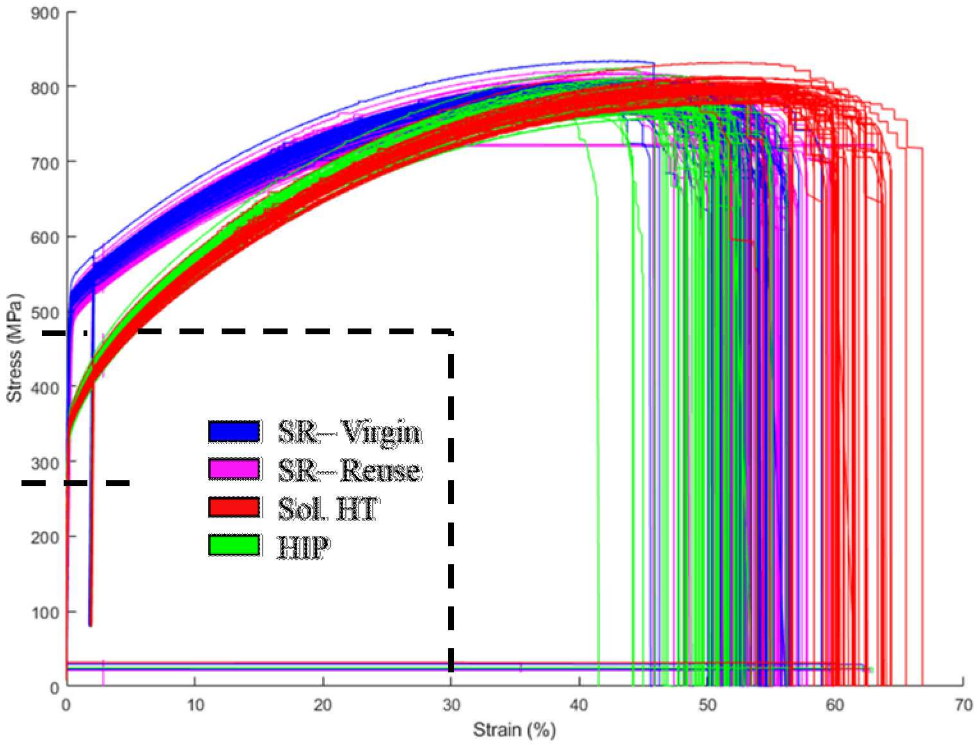
Grain boundary misorientation



Inconel 625 mechanical properties

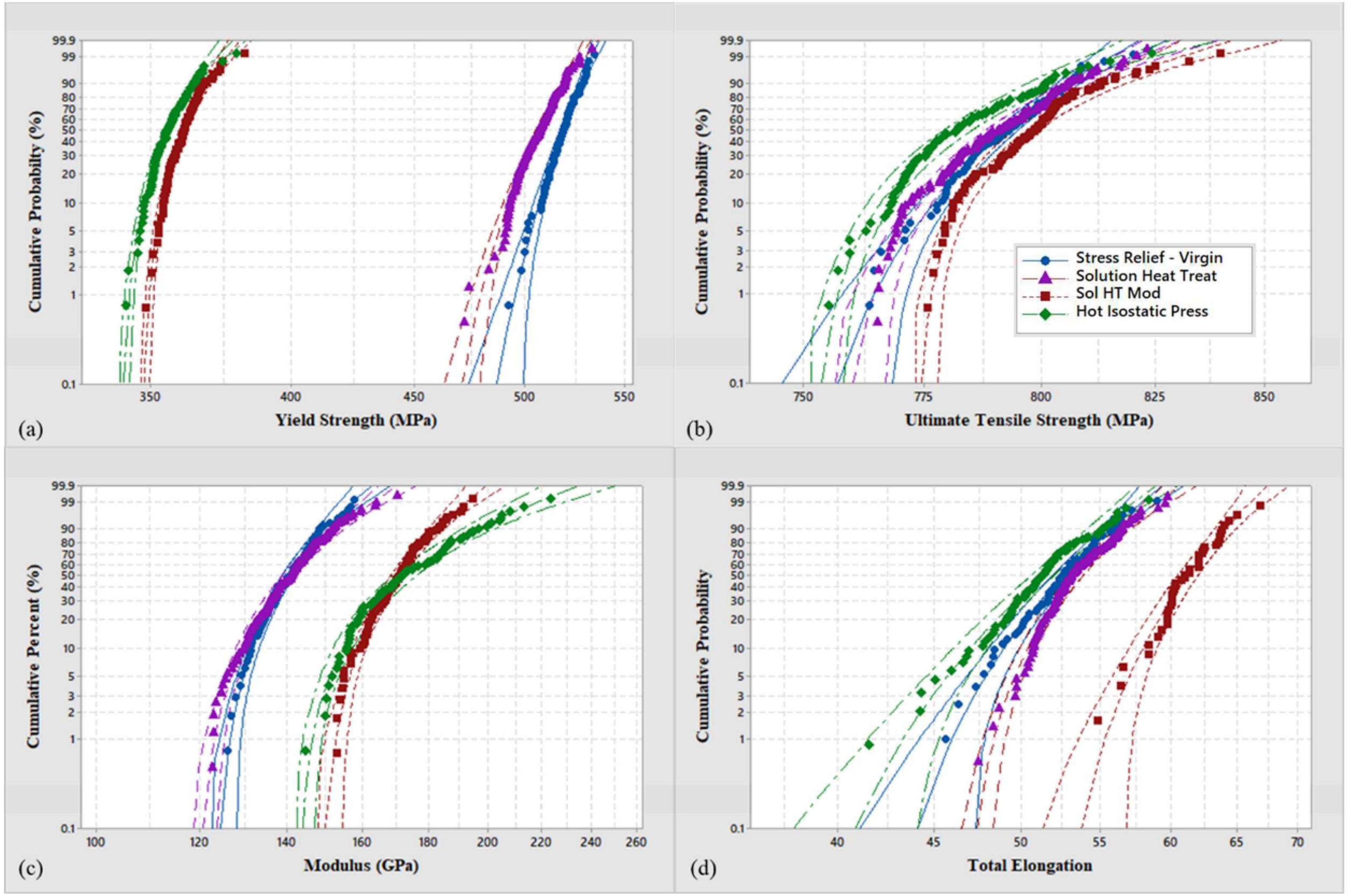
- ASTM F3056 requirements:
  - Yield Strength – 275 MPa
  - Tensile Strength – 485 MPa
  - Elongation to Failure – 30%

Sample	No. of Specimens	Area (mm²)	Modulus (GPa)	Yield Strength (MPa)	Ult. Tensile Strength (MPa)	Elongation at Failure (%)
SR – Virgin	93	1.17	141 ± 7	518 ± 9	792 ± 12	52 ± 3
SR - Reuse	149	1.16	141 ± 9	506 ± 10	790 ± 13	53 ± 2
Sol HT	99	1.16	171 ± 9	361 ± 6	798 ± 12	61 ± 3
HIP	94	1.18	174 ± 12	356 ± 7	782 ± 13	51 ± 3

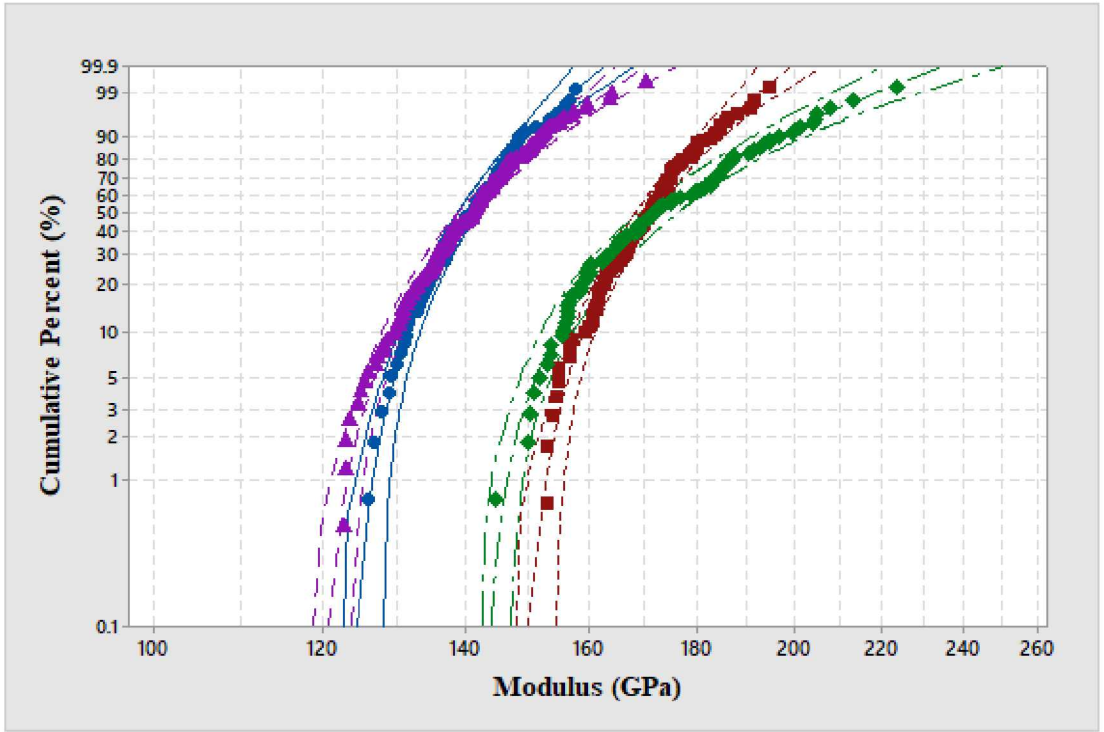


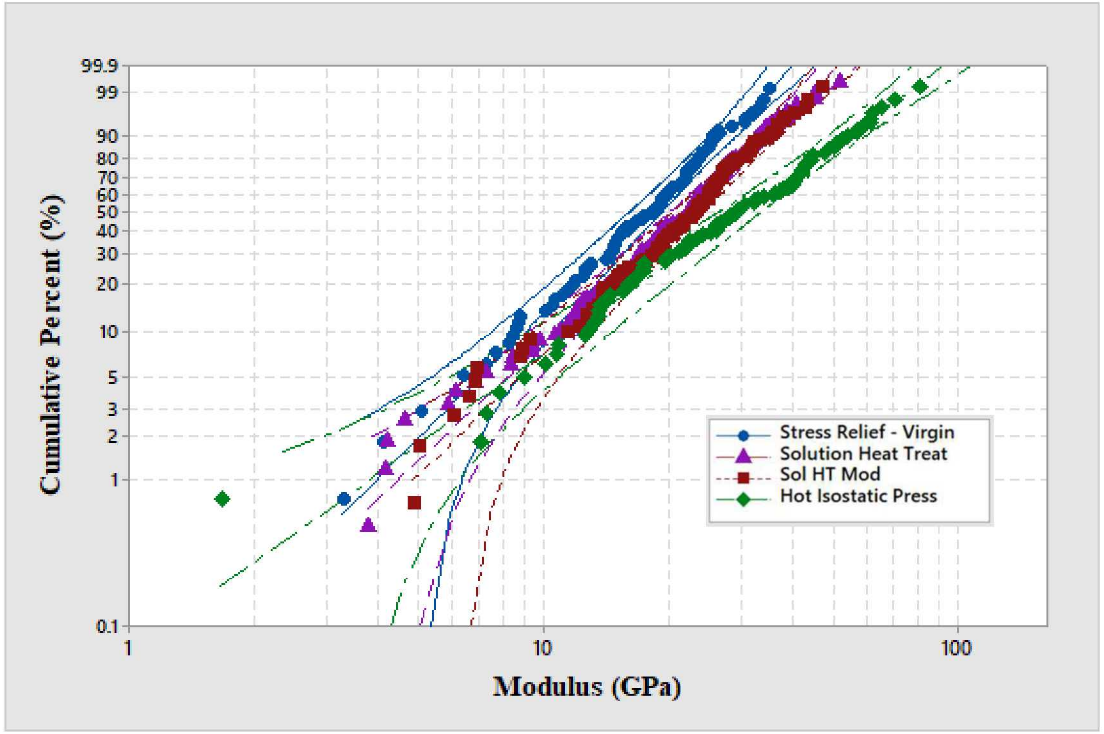
3-parameter Weibull plots of Yield Strength, UTS, Modulus, and Ductility

- Minitab Statistical Software
- $P = 1 - e^{\left[-\left(\frac{\alpha-\gamma}{\eta-\gamma}\right)^{\beta}\right]}$
  - P = probability
  - β = shape
  - η = scale
  - γ = location
- Anderson-Darling statistic
  - Goodness-of-fit @ 95% confidence



Linear x-axis is used to show asymptotic behavior





Inconel Weibull parameters

	Sample	Shape	Scale	Threshold	N	AD	P-value
Yield Stress (MPa)	SR - Virgin	9.159	65.19	456	93	0.259	>0.500
	SR - Reuse	5.957	57.33	453.1	148	0.314	0.473
	SolHT	2.614	16.34	346.7	99	0.356	0.435
	HIP	2.571	18.15	339.9	94	0.320	>0.500
Tensile Strength (MPa)	SR - Virgin	4.848	51.64	744.5	93	0.202	>0.500
	SR - Reuse	2.928	38.42	756.4	149	0.566	0.111
	SolHT	2.125	28.92	773.2	99	0.830	0.032
	HIP	2.487	35.37	751.5	94	0.282	>0.500
Modulus (GPa)	SR - Virgin	2.815	20.07	122.7	93	0.216	>0.500
	SR - Reuse	2.659	24.91	118.8	147	0.299	>0.500
	SolHT	2.764	25.5	147.9	99	0.296	>0.500
	HIP	2.041	35.71	142.8	94	0.281	>0.500
Elongation (%)	SR - Virgin	5.097	12.68	40.79	70	0.180	>0.500
	SR - Reuse	3.267	7.857	46.43	126	0.485	0.18
	SolHT	4.727	10.69	51.33	43	0.432	0.228
	HIP	5.687	16.3	36.01	82	0.525	0.117

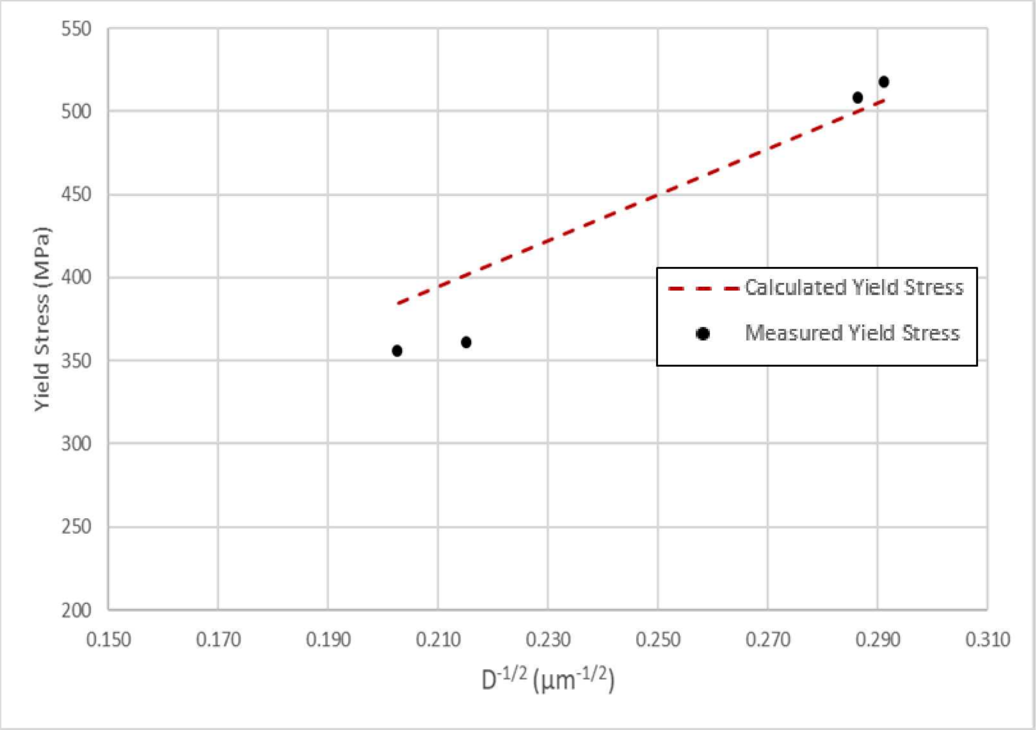
Hall-Petch relation suggests a correlation between yield strength and grain size

- $\sigma_y = \sigma_0 + kD^{-\frac{1}{2}}$

- k = 1380 MPa $\sqrt{\mu m}$
- σ_0 = 105 MPa

Gao Y, Zhou M: **Superior Mechanical Behavior and Fretting Wear Resistance of 3D-Printed Inconel 625 Superalloy.** *Applied Sciences* 2018, **8**.

Sample	Avg. Grain Size (µm)	$D^{-\frac{1}{2}}$ $(\mu m^{-\frac{1}{2}})$	Yield Stress (MPa)
SR – Virgin	11.8	0.29	518
SR – Reuse	12.2	0.28	508
Sol HT	21.6	0.21	361
HIP	24.4	0.20	356



Texture and anisotropy *may* have effects on Young's modulus

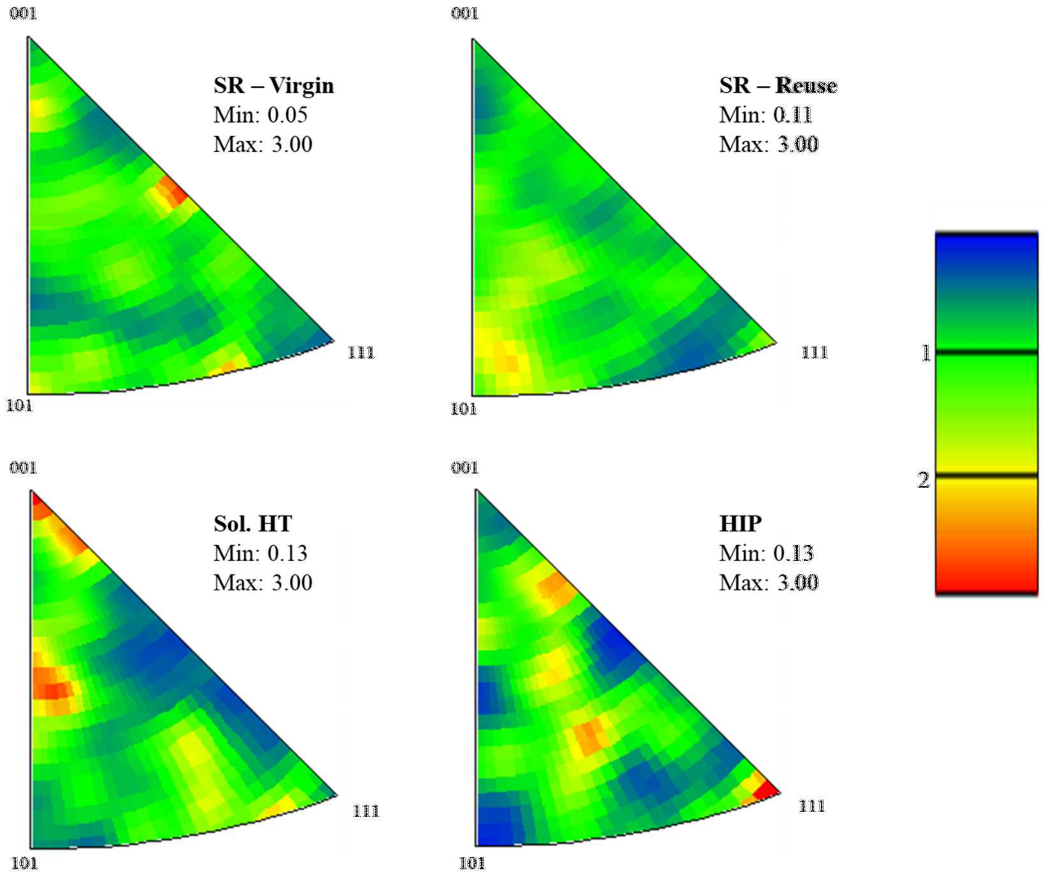
Zener ratio of nickel, Z = 2.54

- $Z = \frac{2c_{44}}{c_{11} - c_{12}}$

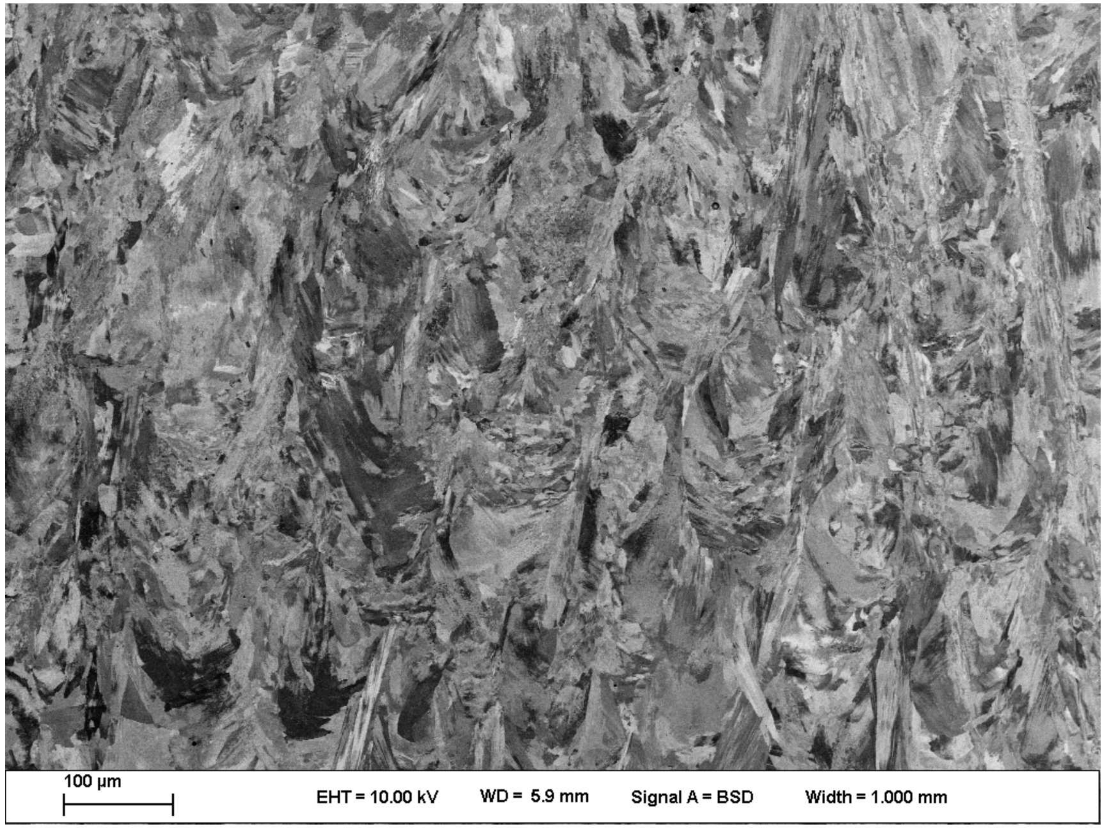
Heat Treatment	SR	Sol HT	HIP
Modulus (GPa)	140	170	174

- $\frac{1}{E_{ijk}} = S_{11} - 2\left(S_{11} - S_{12} - \frac{1}{2}S_{44}\right) \times \left(I_{i1}^2 I_{j2}^2 + I_{j2}^2 I_{k3}^2 + I_{i1}^2 I_{k3}^2\right)$

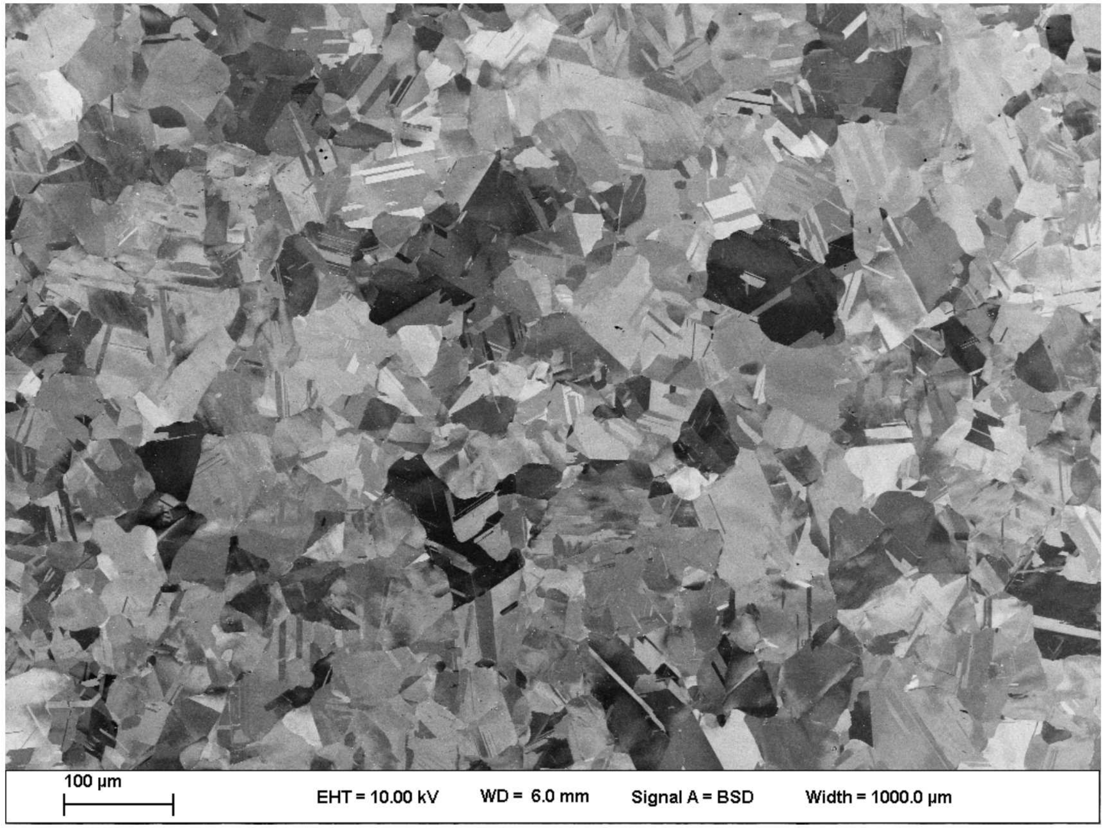
Direction Cosine	[100]	[101]	[111]
Modulus (GPa)	164	234	271



Columnar grain growth *may* exaggerate the anisotropic behavior



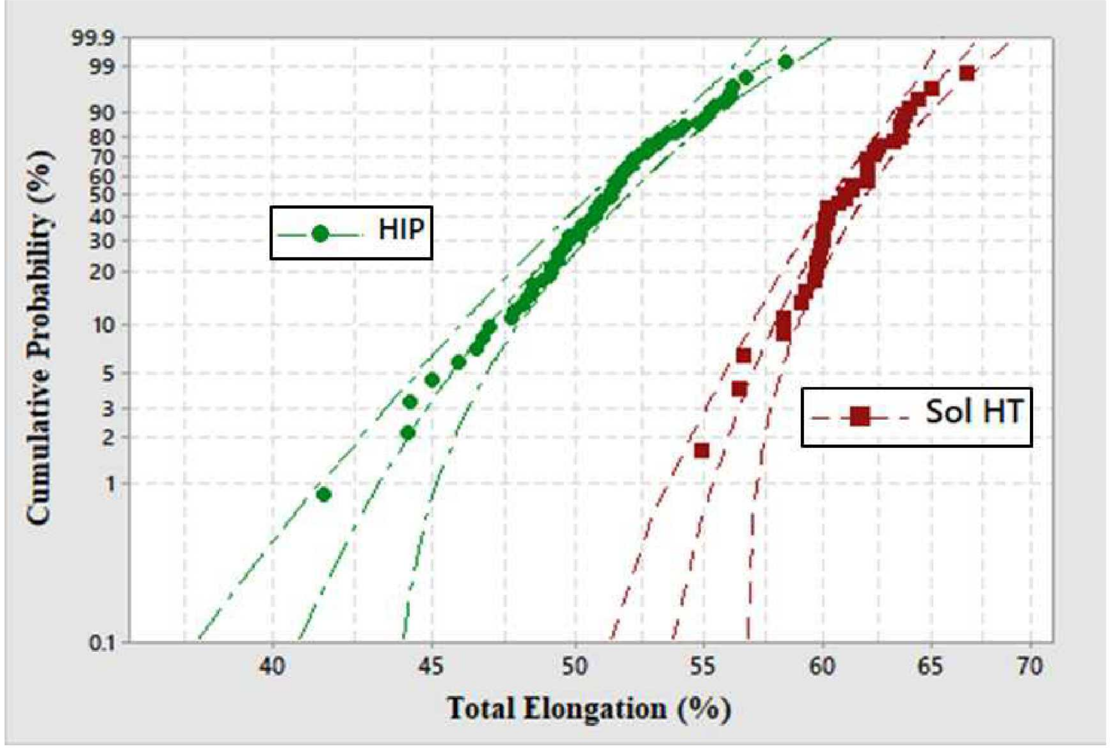
SR – Virgin



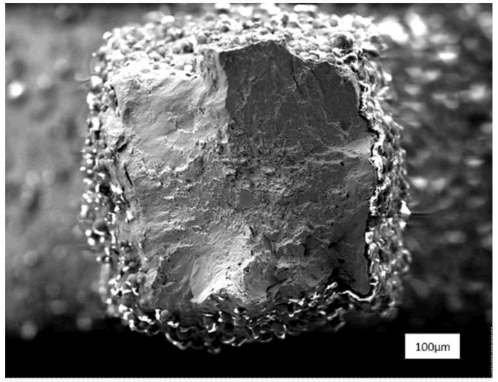
HIP

The HIP samples show *lowest* ductility

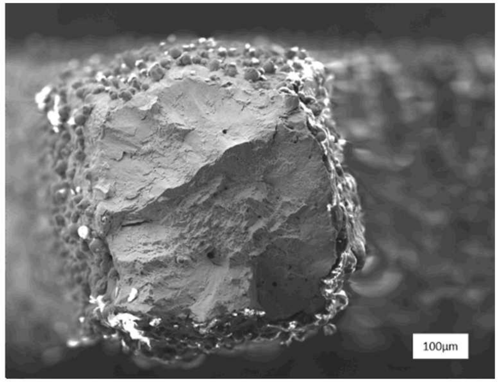
- Analysis methods:
  - Determination of porosity
  - Microstructure analysis



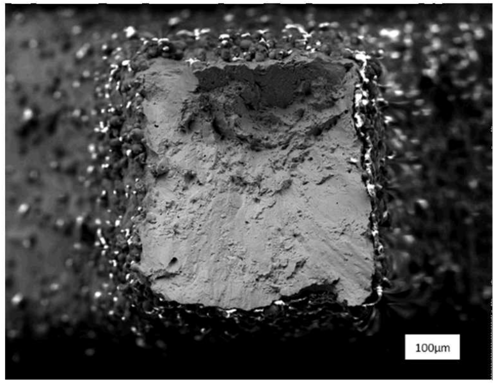
Inconel 625 – Fracture Surfaces



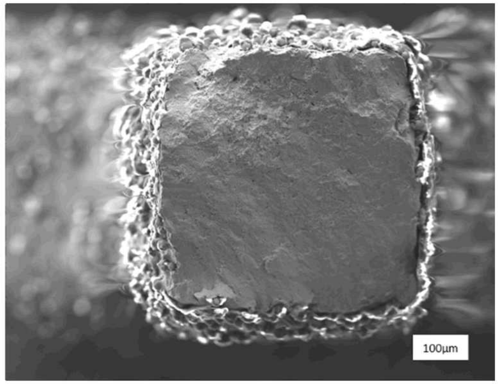
(a) SR Virgin



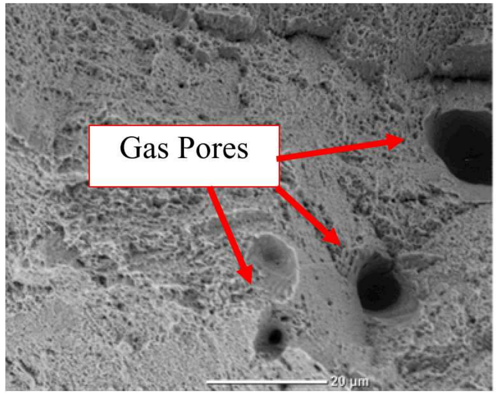
(b) SR Reuse



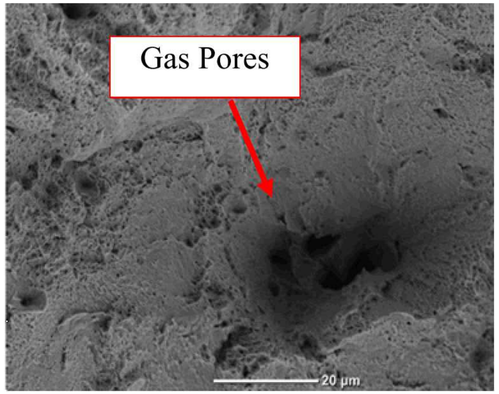
(c) Sol HT



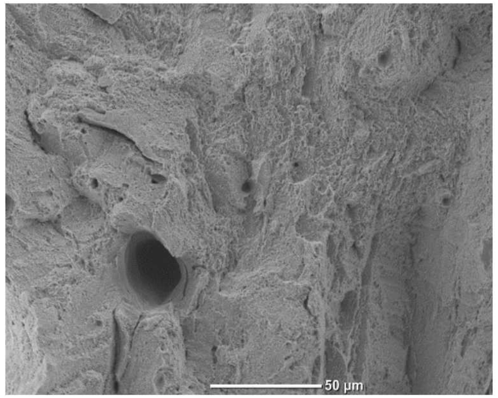
(d) HIP



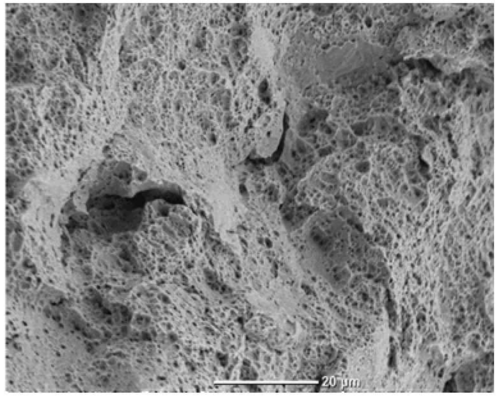
(a) SR Virgin



(b) SR Reuse



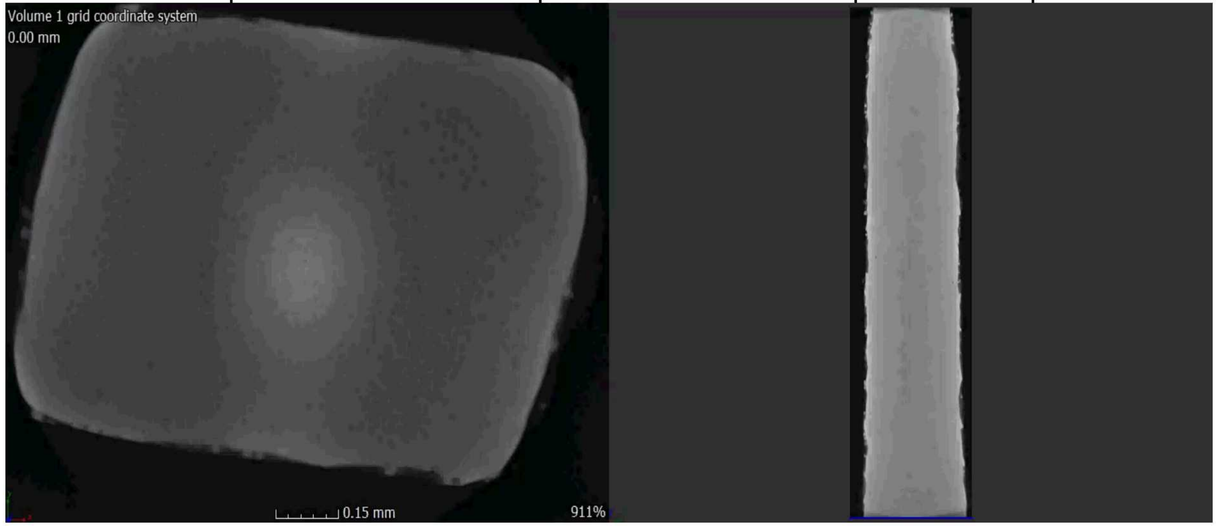
(c) Sol HT



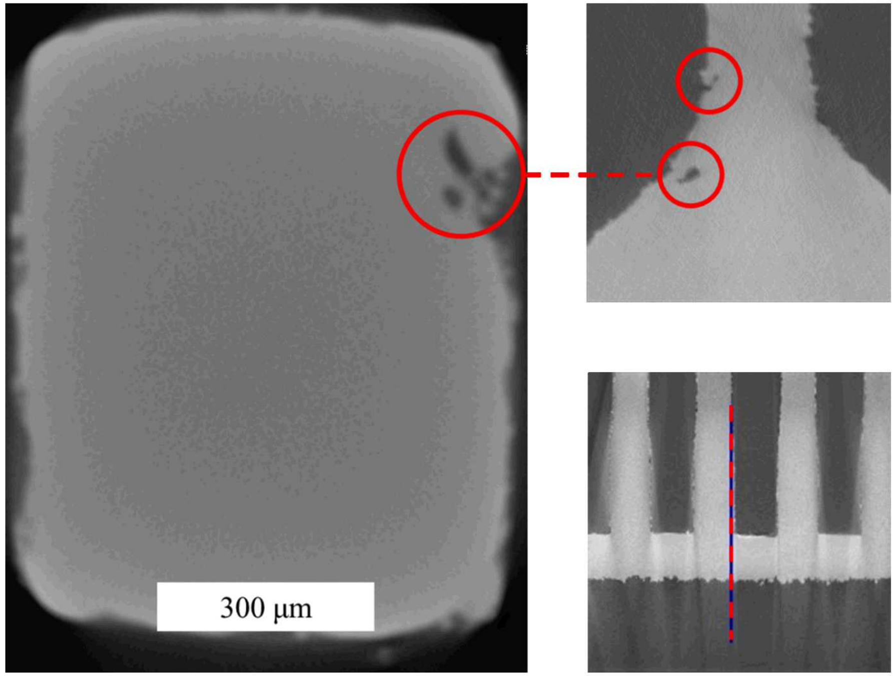
(d) HIP

Density and CT results show porosity *may* play a role

Sample	Density (g/mL)	% Dense
SR – Virgin	8.444	98.1 %
SR – Reuse	8.463	98.3 %
Sol HT	8.460	98.3 %
HIP	8.427	97.9 %

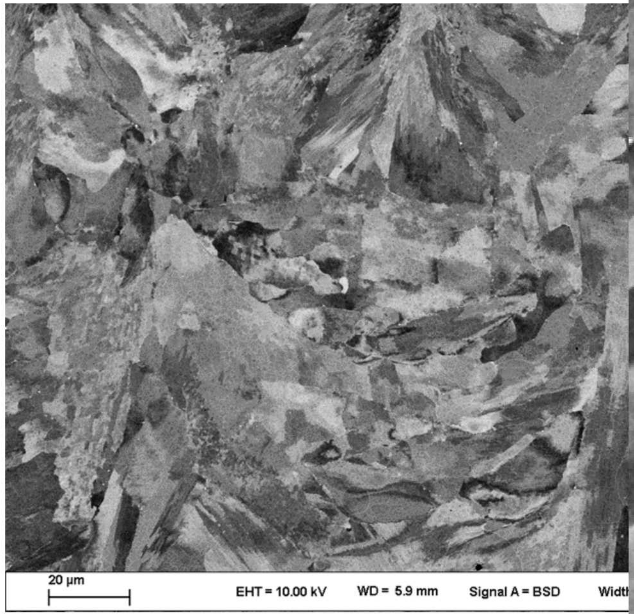


Sol HT

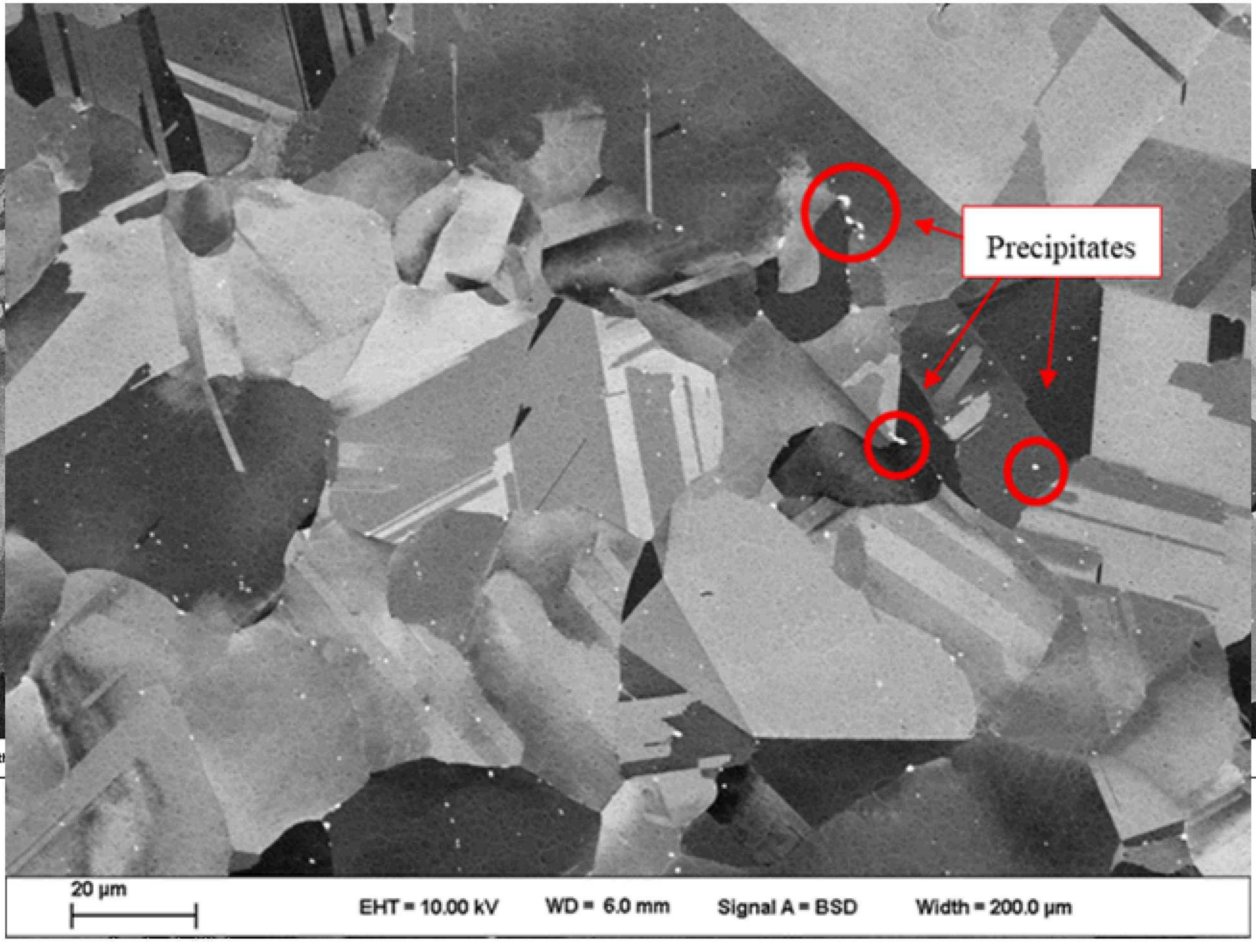


HIP

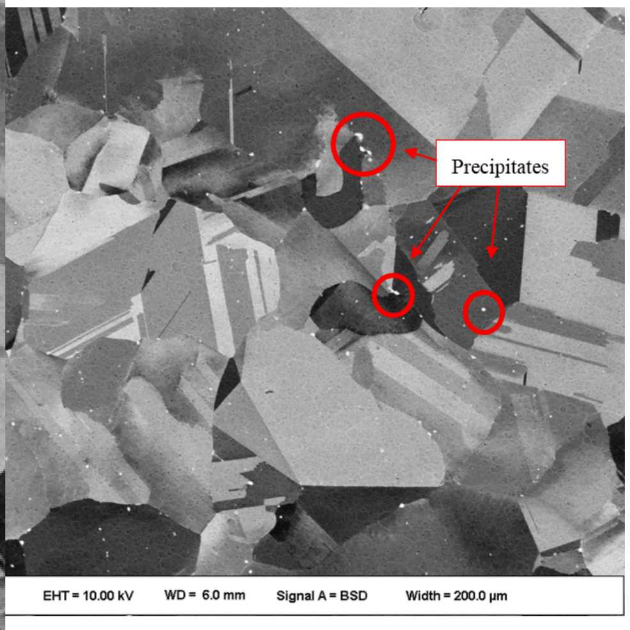
SEM images of the stress relieved, solution annealed, and HIP samples show an increase in population and size of precipitates in the HIP material



Stress Relief

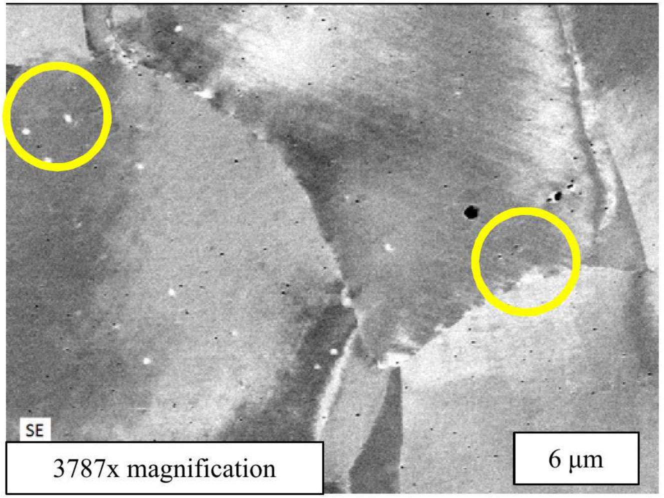


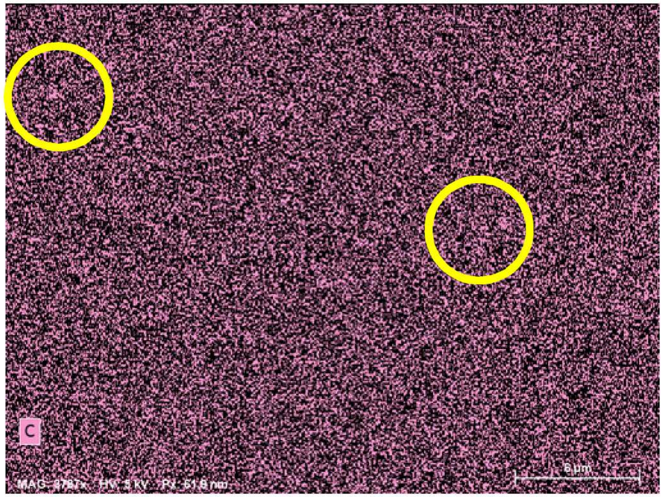
HIP

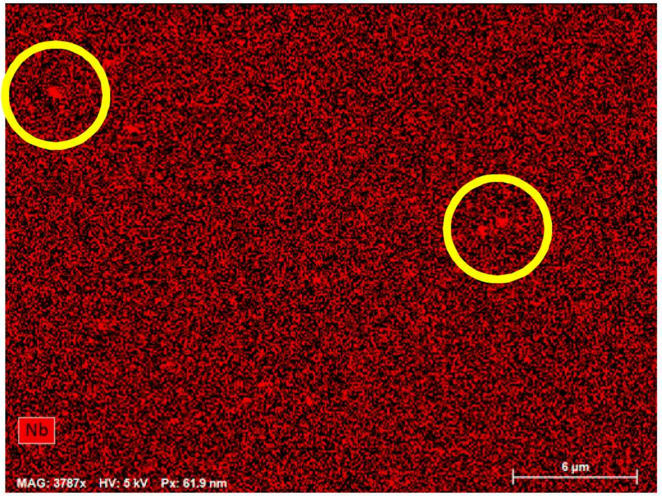


HIP

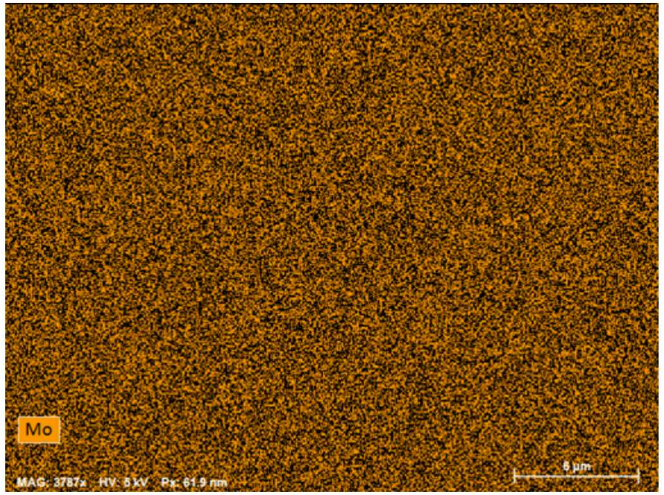
Inconel 625 – Solution Annealed Precipitates

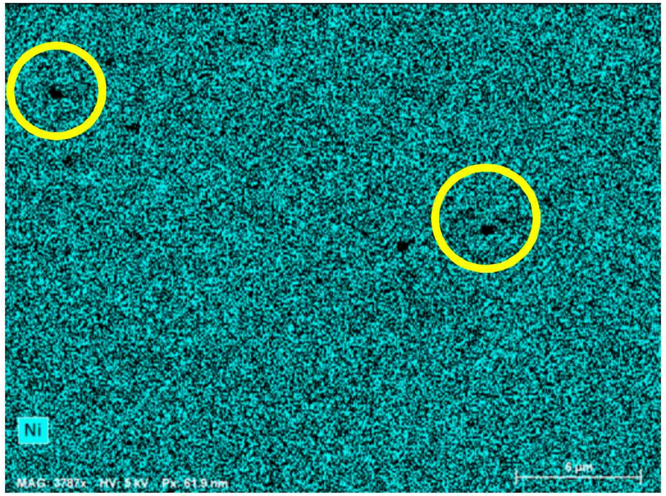




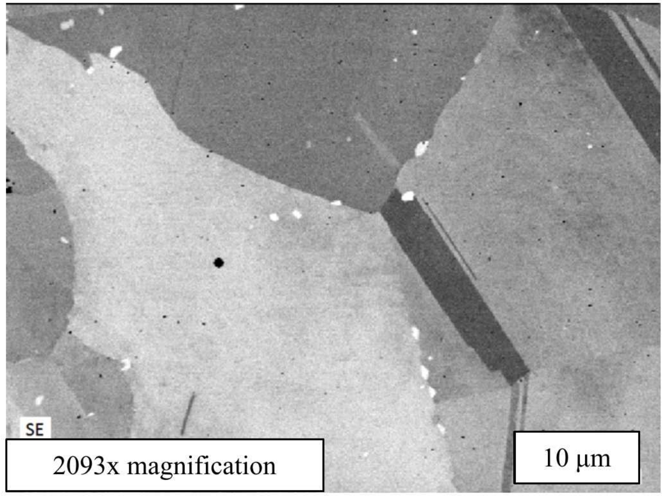


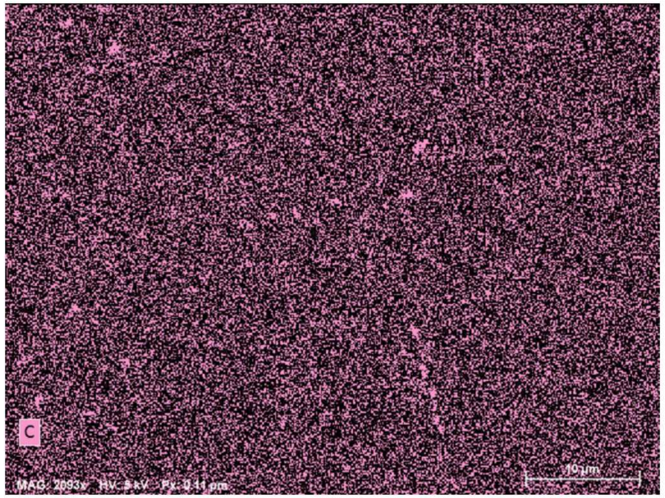
Precipitate size:
~340 nm

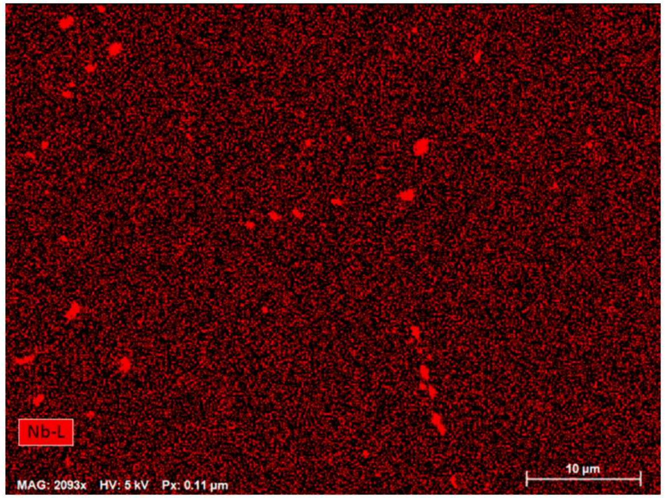




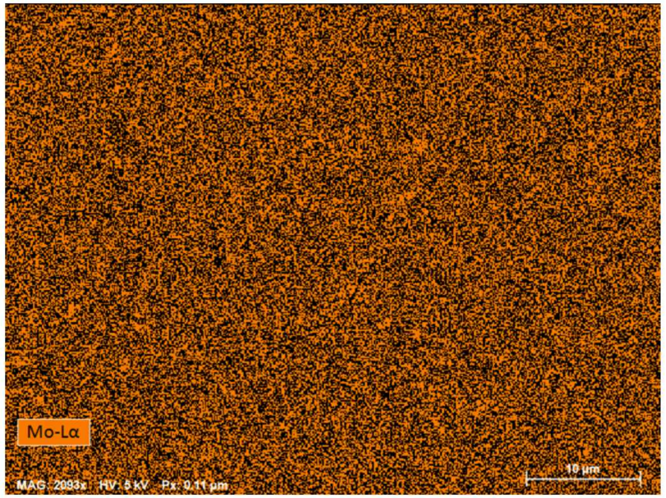
Inconel 625 – HIP Precipitates

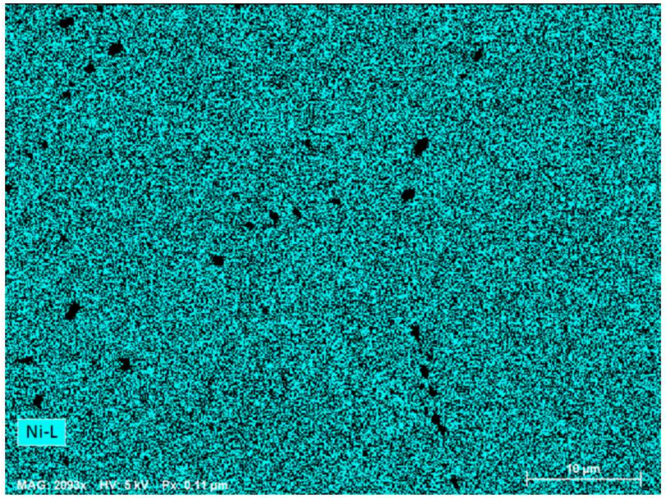




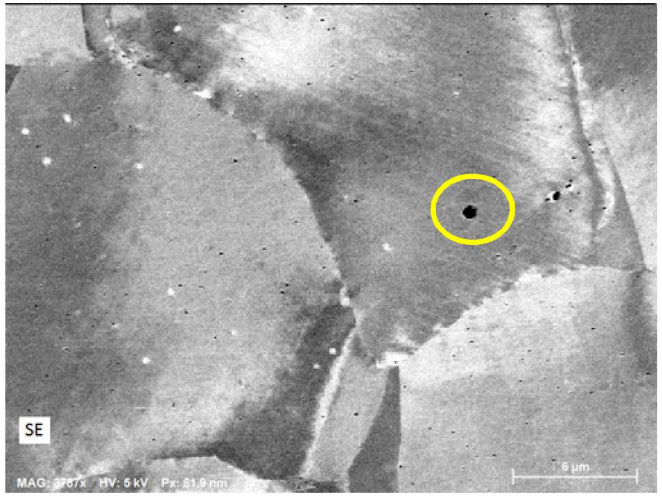


Precipitate size:
~900 nm

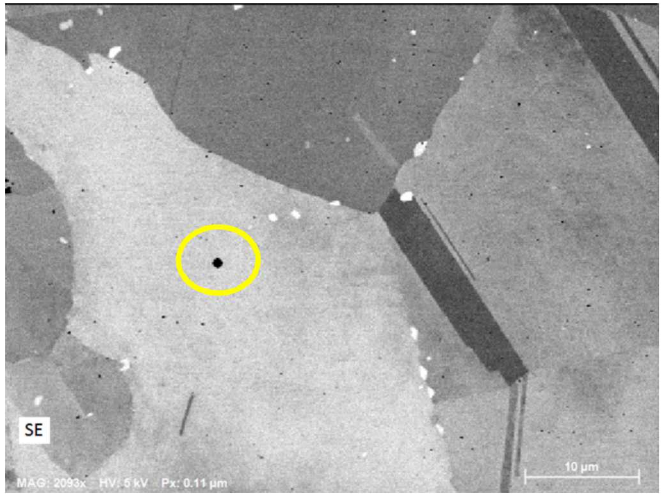




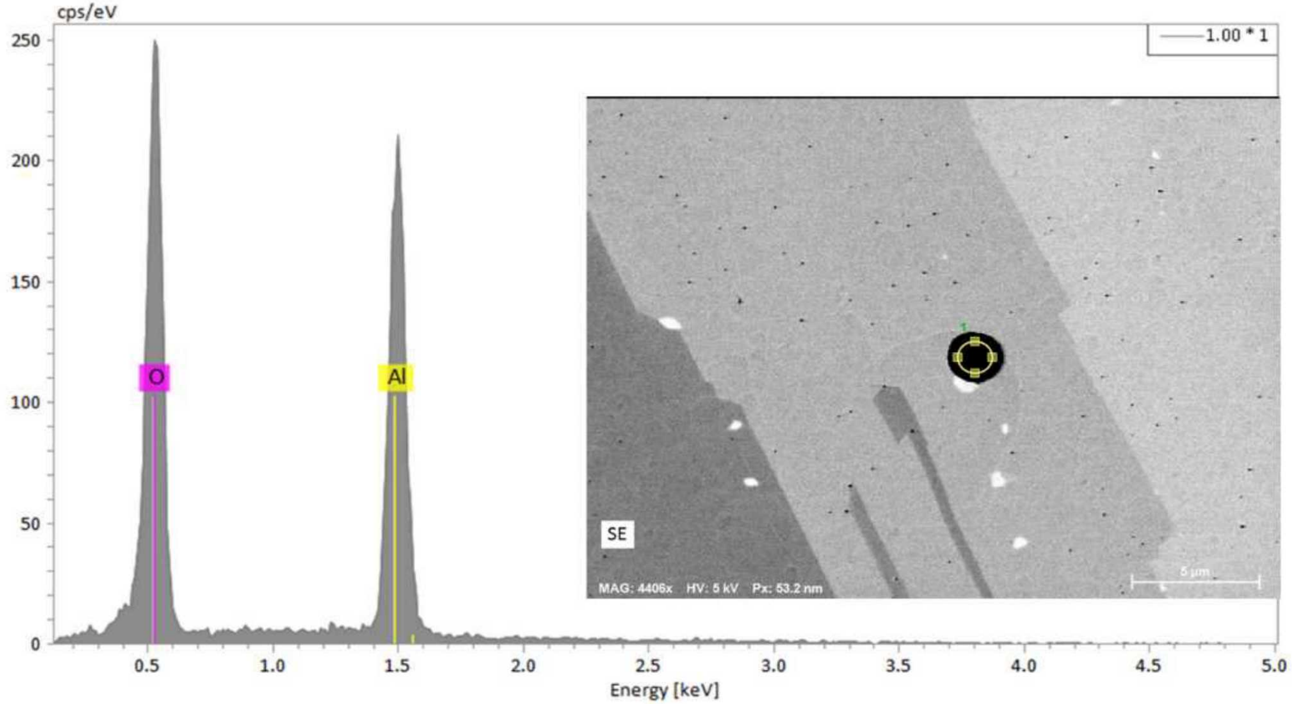
Oxides detected in the Inconel 625 samples, likely the Al_2O_3 oxide presented in literature



Sol HT



HIP



Inconel 625 – Sub-Size Effect

- $\sigma_{corrected} = \frac{F}{(w-2R_p)*(t-2R_p)}$

Sample	R_a (µm)	R_p (µm)
SR – Virgin	7.79	50.44
SR – Reuse	8.42	73.00
Sol. HT	7.65	64.59
HIP	8.52	51.89

Sample	Sub-Size UTS (MPa)	Corrected Sub-Size UTS (MPa)	Standard Size UTS
Stress Relief	791	927	932
Sol HT	798	930	929
HIP	783	918	922

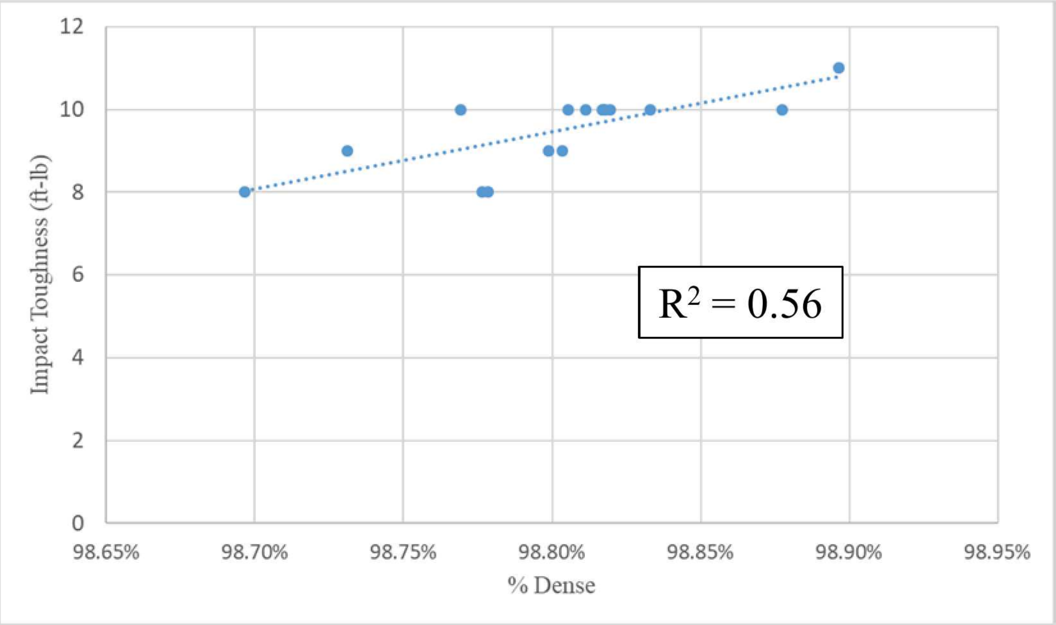
Salzbrenner BC, Rodelas JM, Madison JD, Jared BH, Swiler LP, Shen Y-L, Boyce BL: **High-throughput stochastic tensile performance of additively manufactured stainless steel.** *Journal of Materials Processing Technology* 2017, **241:**1-12.

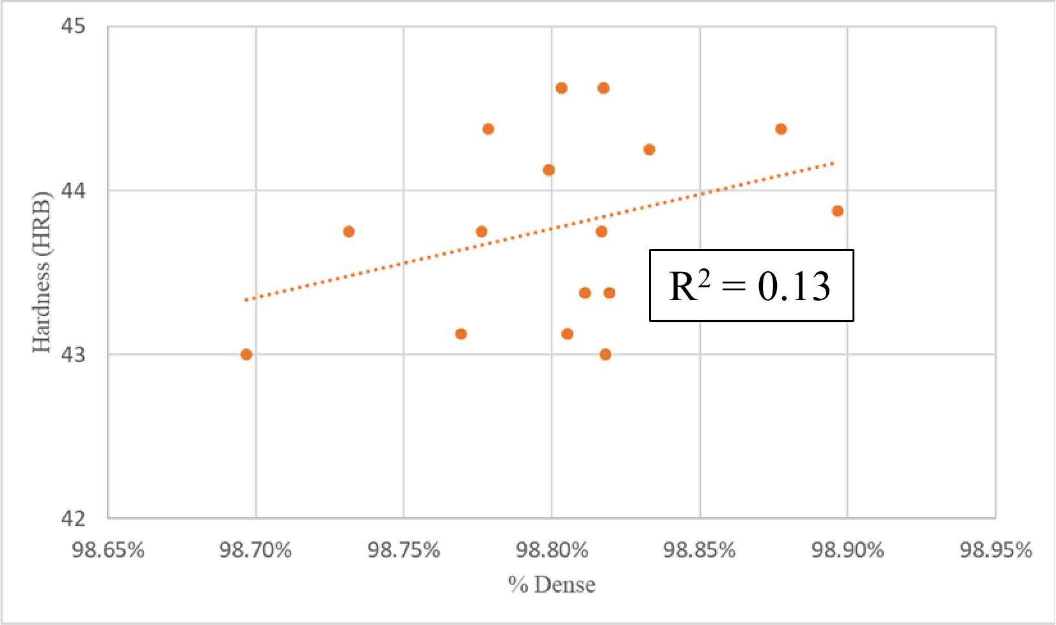
AlSi10Mg – Material Characterization

Composition	Units	ASTM F3318	AM 103C Spec	As Received
Al	Wt %	Bal	Bal	Bal
C	Wt %	0.05 max	0.05 max	-
Ca	Wt %	0.05 max	0.05 max	-
Cr	Wt %	0.05 max	0.05 max	-
Cu	Wt %	0.05 max	0.03 max	<0.01
Fe	Wt %	0.55 max	0.40 max	0.07
Li	Wt %	0.05 max	0.05 max	-
Mg	Wt %	0.20-0.45	0.25-0.45	0.3
Mn	Wt %	0.45 max	0.15 max	0.01
N	Wt %	0.05 max	0.05 max	-
Ni	Wt %	0.05 max	0.05 max	<0.01
Pb	Wt %	0.05 max	0.05 max	<0.01
S	Wt %	0.05 max	0.05 max	-
Si	Wt %	9.0-11.0	9.0-11.0	9.78
Sn	Wt %	0.05 max	0.05 max	<0.01
Ti	Wt %	0.15 max	0.15 max	0.01
V	Wt %	0.05 max	0.05 max	-
Zn	Wt %	0.10 max	0.10 max	<0.01

AlSi10Mg – Acceptance Testing Results

- 98.8% dense relative to 2.68 g/mL in literature
- Average hardness: 44 HRB
- Average toughness: 10 ft-lbf

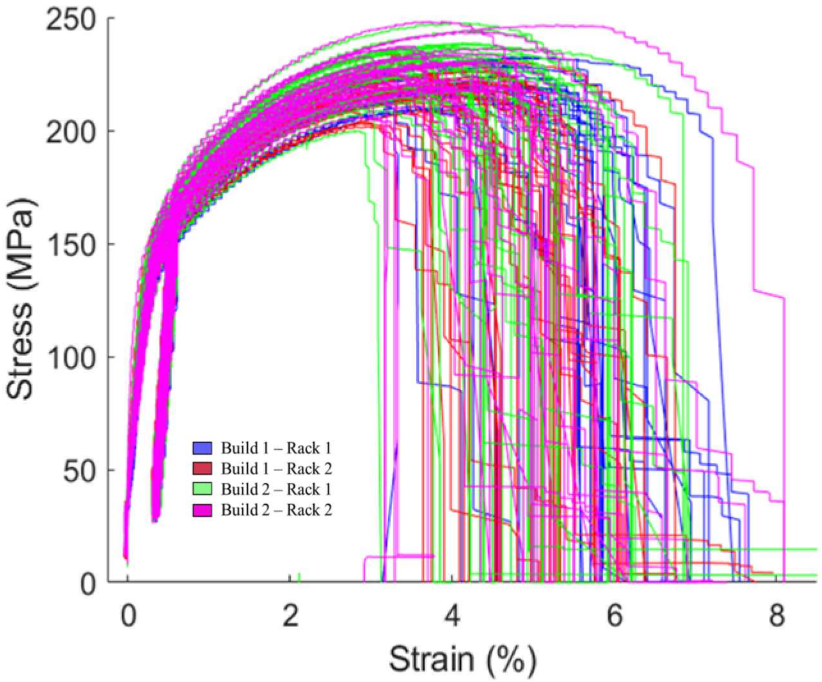




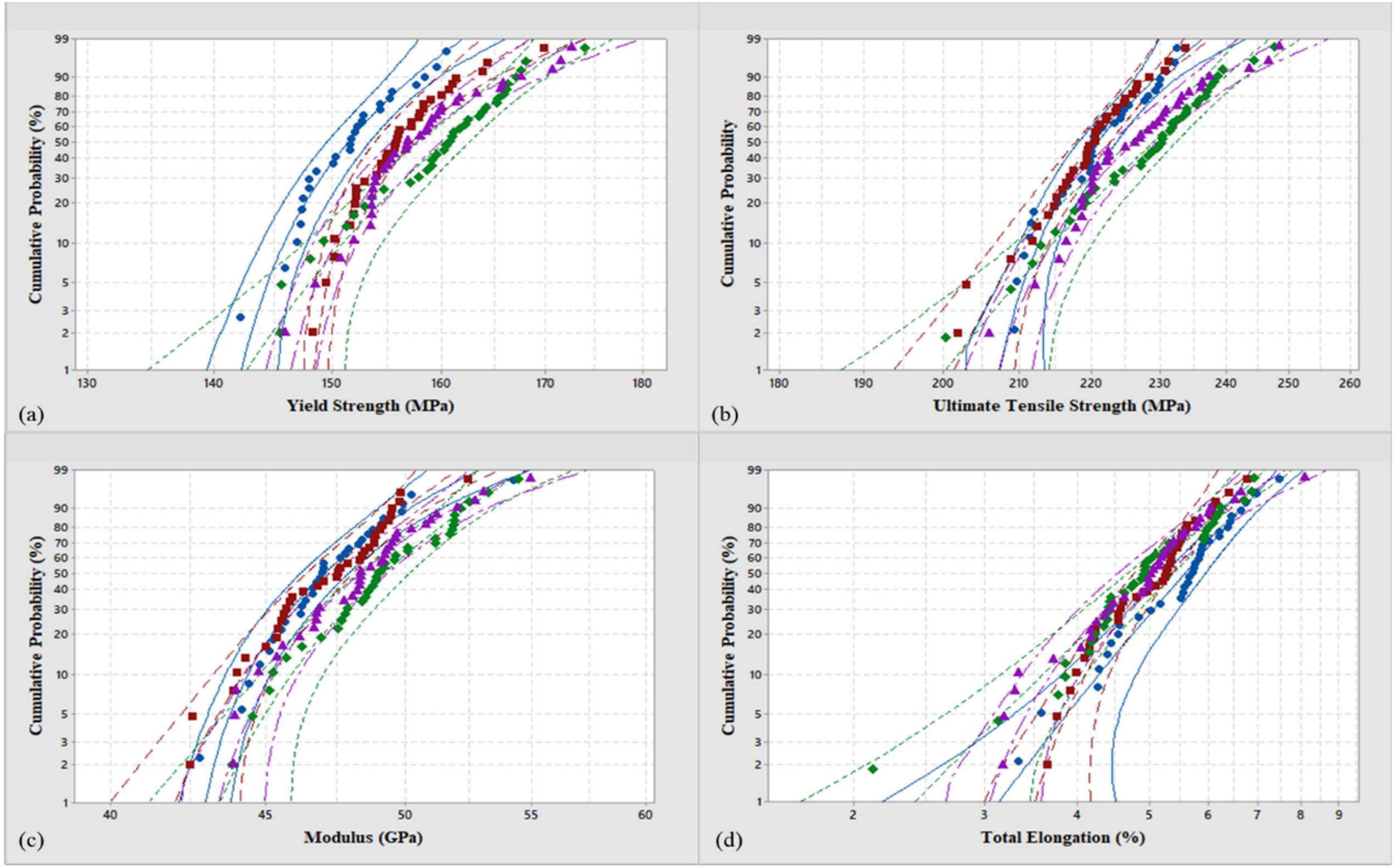
AlSi10Mg – Mechanical Properties

- ASTM F3318 requirements:
  - Yield Strength – 138 MPa
  - Tensile Strength – 241 MPa
  - Elongation to Failure – 10%

Sample	Num. of Specimens	Area (mm^2)	Modulus (GPa)	Yield Strength (MPa)	Ult. Tensile Strength (MPa)	Elongation at Failure (%)
Build 1 – R1	33	0.80	47 ± 2	152 ± 4	221 ± 7	5.5 ± 1.0
Build 1 – R2	35	0.79	47 ± 2	156 ± 5	220 ± 7	5.1 ± 0.8
Build 2 -R1	38	0.77	49 ± 3	160 ± 7	228 ± 10	5.0 ± 1.0
Build 2 -R2	36	0.77	49 ± 3	158 ± 6	227 ± 9	5.0 ± 1.0



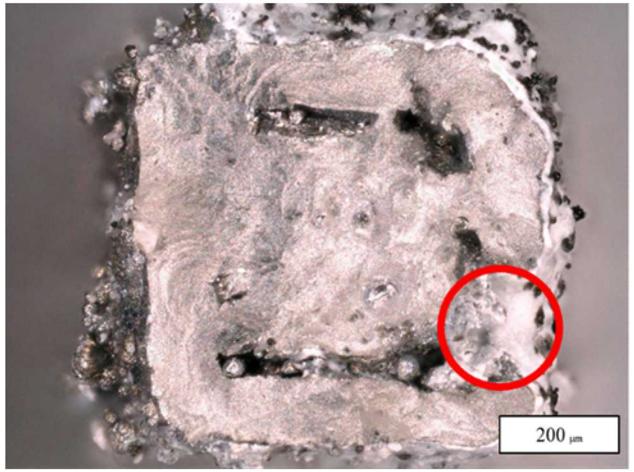
3-parameter Weibull plots of Yield Strength, UTS, Modulus, and Ductility



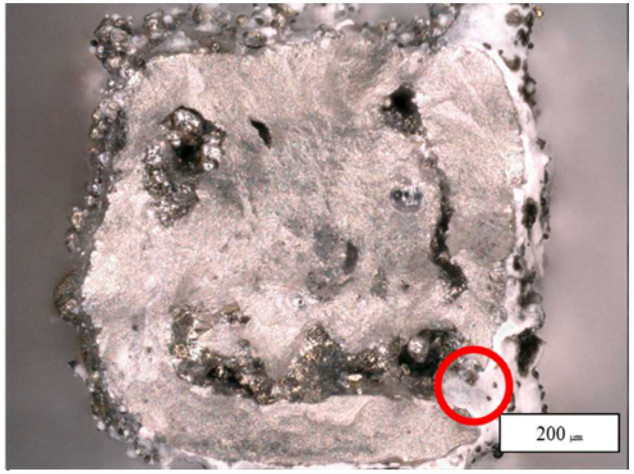
AlSi10Mg – Statistics Cont.

	Sample	Shape	Scale	Thresh	N	AD	P
Yield Stress (MPa)	Build 1 - R1	3.067	13.81	138.2	26	0.268	>0.500
	Build 1 - R2	1.934	9.642	147.5	34	0.18	>0.500
	Build 2 - R1	6.29	37.96	124.4	35	0.311	0.479
	Build 2 - R2	2.374	15.66	144.2	35	0.512	0.183
Tensile Strength (MPa)	Build 1 - R1	3.068	20.35	202.9	33	0.516	0.148
	Build 1 - R2	6.608	42.27	180.3	35	0.253	>0.500
	Build 2 - R1	9.536	83.58	148.7	38	0.215	>0.500
	Build 2 - R2	2.897	28.04	201.5	36	0.315	>0.500
Modulus (GPa)	Build 1 - R1	2.435	5.743	42.11	31	0.315	>0.500
	Build 1 - R2	4.069	8.813	39.17	35	0.462	0.196
	Build 2 - R1	3.782	9.392		35	0.298	>0.500
	Build 2 - R2	2.609	7.037	42.23	35	0.357	0.432
Elongation (%)	Build 1 - R1	5.688	4.98	0.9204	33	0.342	0.41
	Build 1 - R2	3.015	2.324	3.004	35	0.513	0.152
	Build 2 - R1	4.943	4.879	0.4958	38	0.572	0.089
	Build 2 - R2	2.393	2.612	2.663	36	0.534	0.154

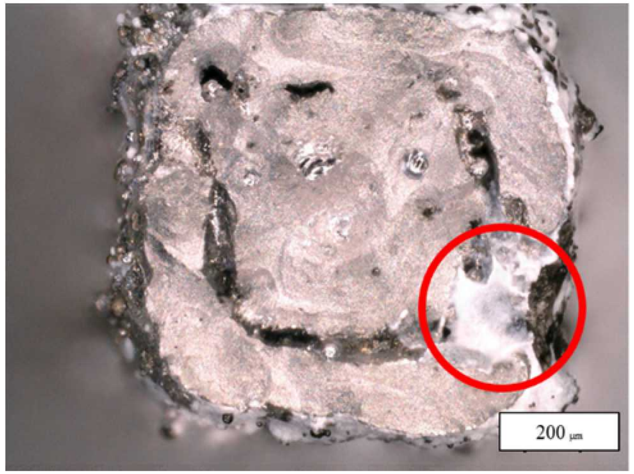
Fractography of AlSi10Mg samples show porosity due to perimeter scans



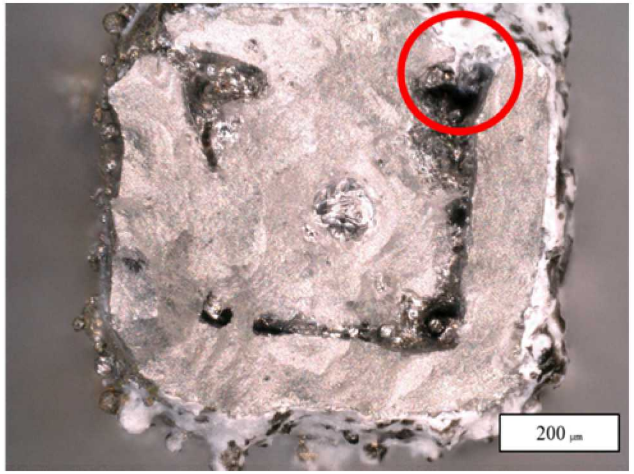
(a) Build 1 – R1



(b) Build 1 – R2

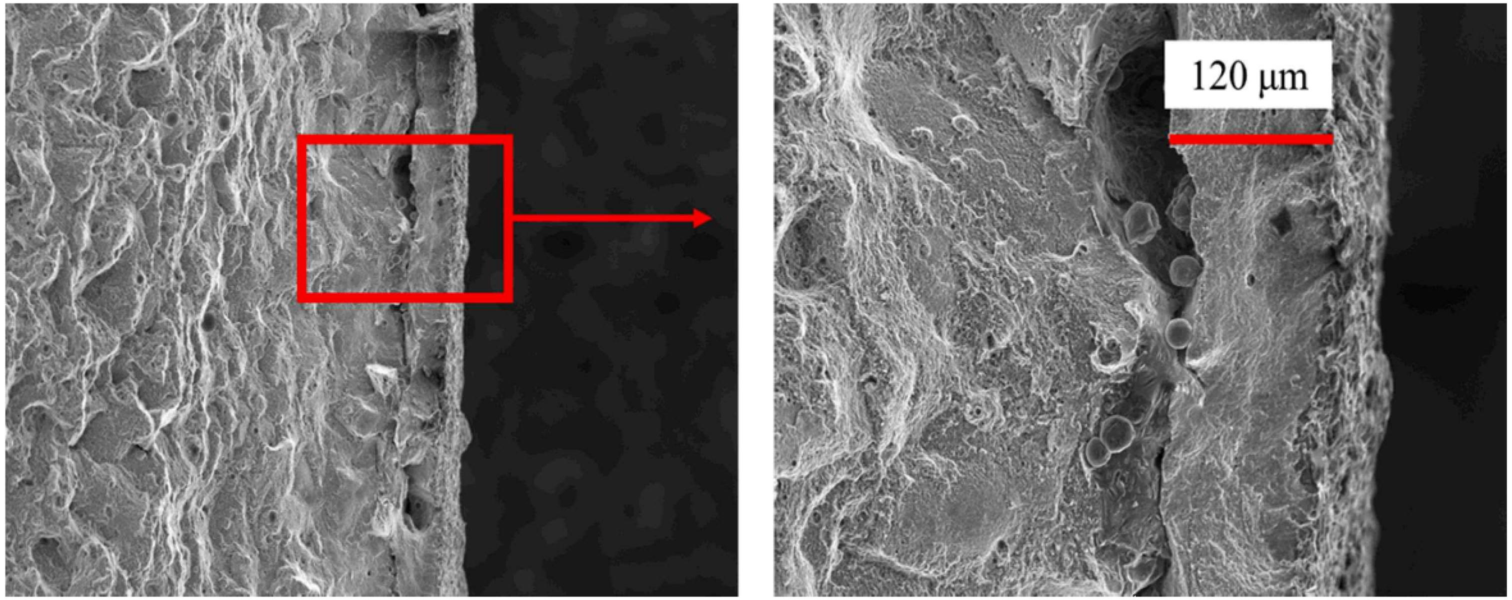


(c) Build 2 – R1



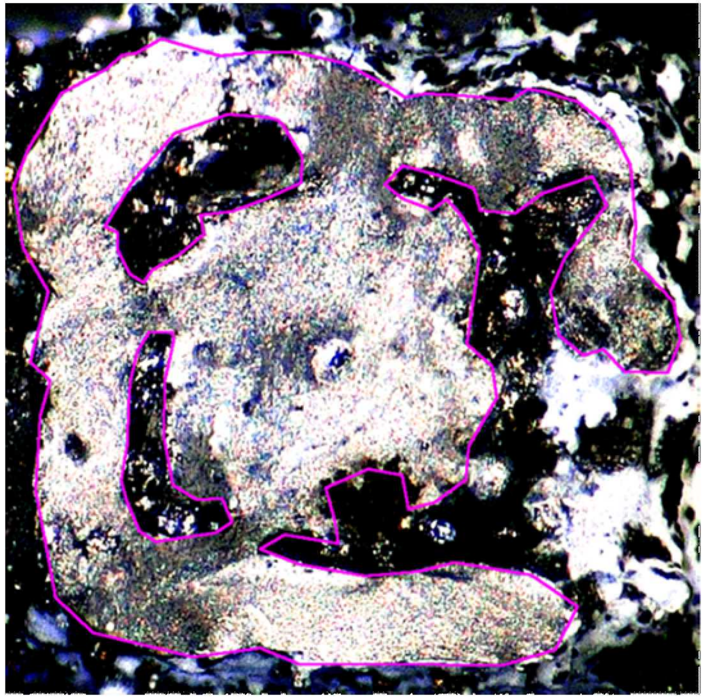
(d) Build 2 – R2

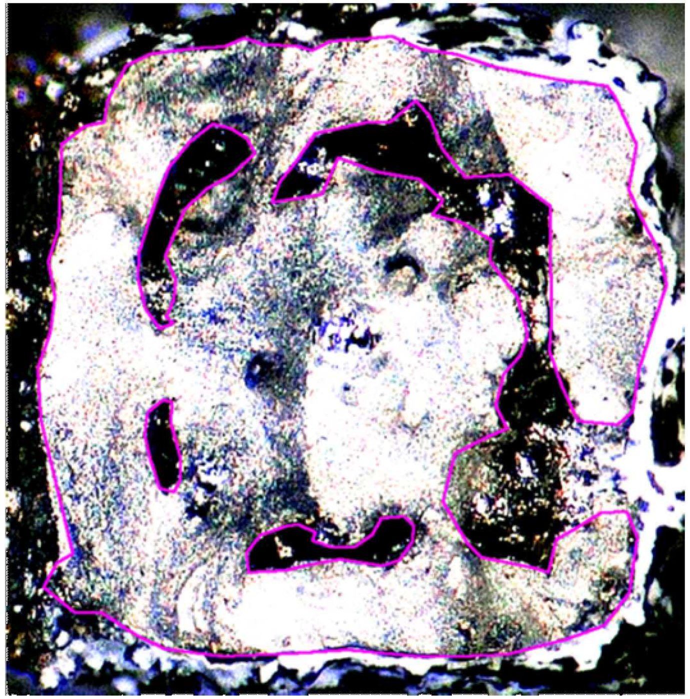
Fractography of Charpy samples show similar lack-of-fusion voids



(a) (b)

AlSi10Mg – Effective Cross-Section





Effective Area: 60%

Tensile Strength: 220 MPa

Elongation at Failure: 2.8%

Corrected Strength: 308 MPa

Effective Area: 74%

Tensile Strength: 247 MPa

Elongation at Failure: 8.1%

Corrected Strength: 311 MPa

Inconel 625 Conclusions

- Statistically determined low-probability outliers all above ASTM minimum requirements
- Reusing powder for a subsequent build had negligible effects on mechanical properties
- Columnar grains and untextured microstructure may have had negative effects on elastic modulus values
- Solution annealing and HIPing resulted in larger grain sizes, potentially reducing the yield strength as explained by the Hall-Petch relation
- A large number of precipitates found in the HIP samples significantly reduced the elongation at failure

AlSi10Mg Conclusions

- AM acceptance testing procedure showed a strong linear trend in the density – toughness relationship
- The decrease in density is due to lack-of-fusion voids caused by incorrect perimeter scans
- Porosity was a catalyst for premature failure at low strength and elongation
- Correction for effective load bearing area increases strength values above ASTM requirements

Future Work

- Adjustments to high-throughput tensile testing to allow for larger samples
- Enhance acceptance testing procedure for producing more accurate results in less time
- Characterization of precipitate formation during HIPing of Inconel 625

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