8/15/2020
SAND2020-7251C

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

Aron S. Robbins[1], Jay D. Carroll[1], E. W. Reutzel[2]

[1] Sandia National Laboratories, P.O. Box 5800, Albuquerque, NM 87185

[2] Applied Research Laboratory, University Park, PA 16802

Submission number 9674

SEM XIV International Congress

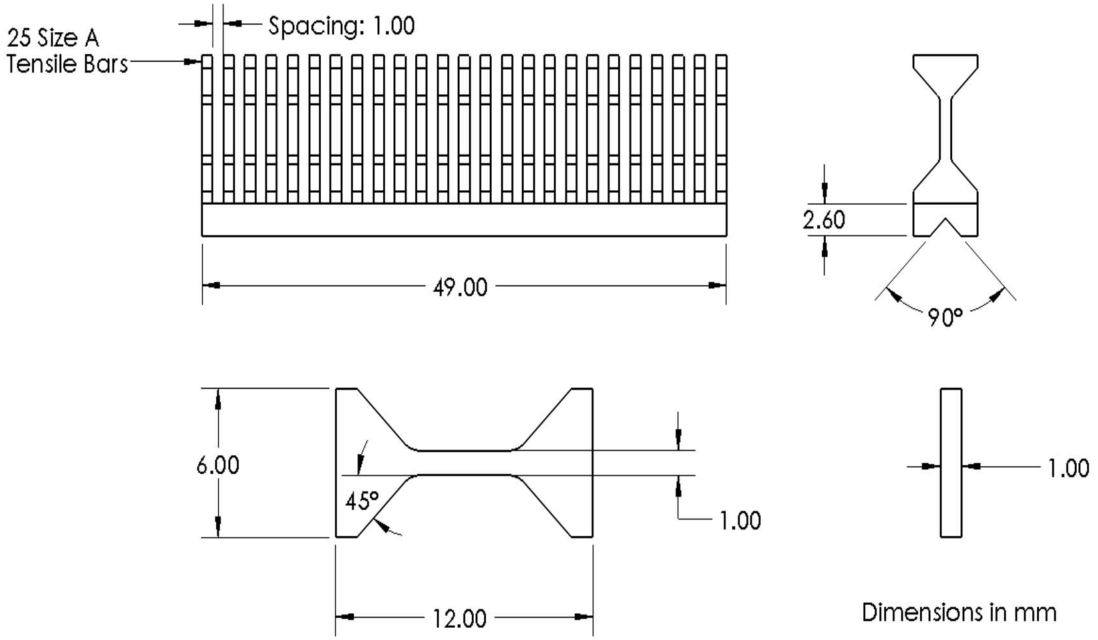
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. ~~SAND2019-13651 PE~~

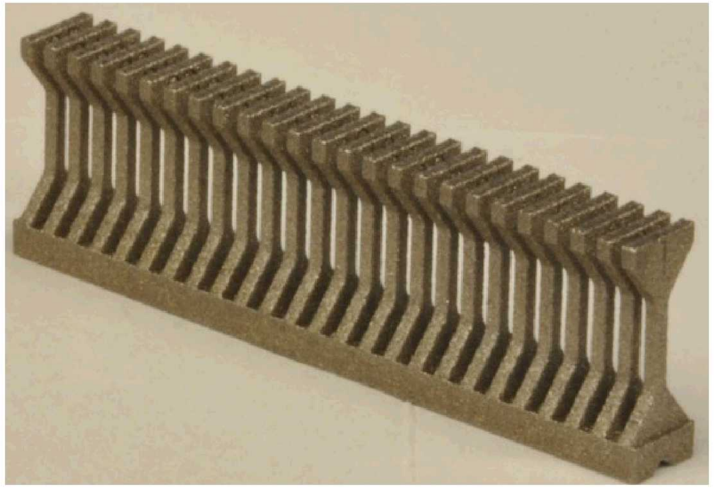
Focus of Study & Outline

- High-throughput tensile testing & batch analysis
- Material characterization
- Statistical analysis of mechanical performance
- Analysis of abnormally low ductility in HIP samples

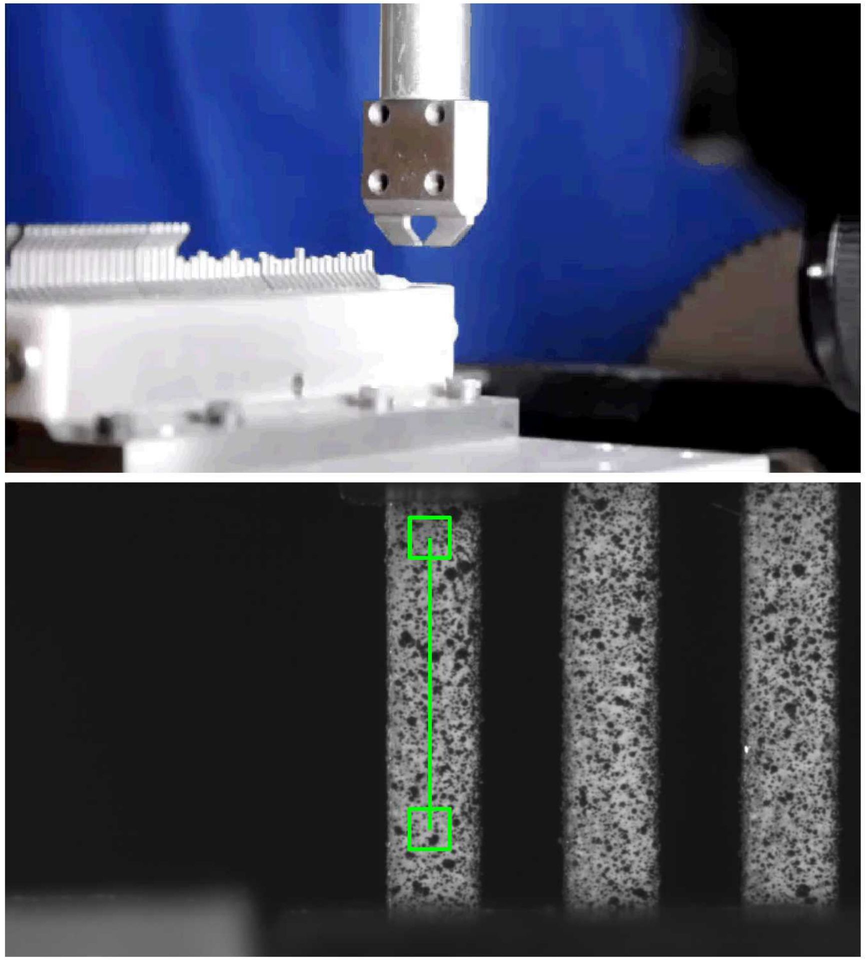
High-throughput specimen design

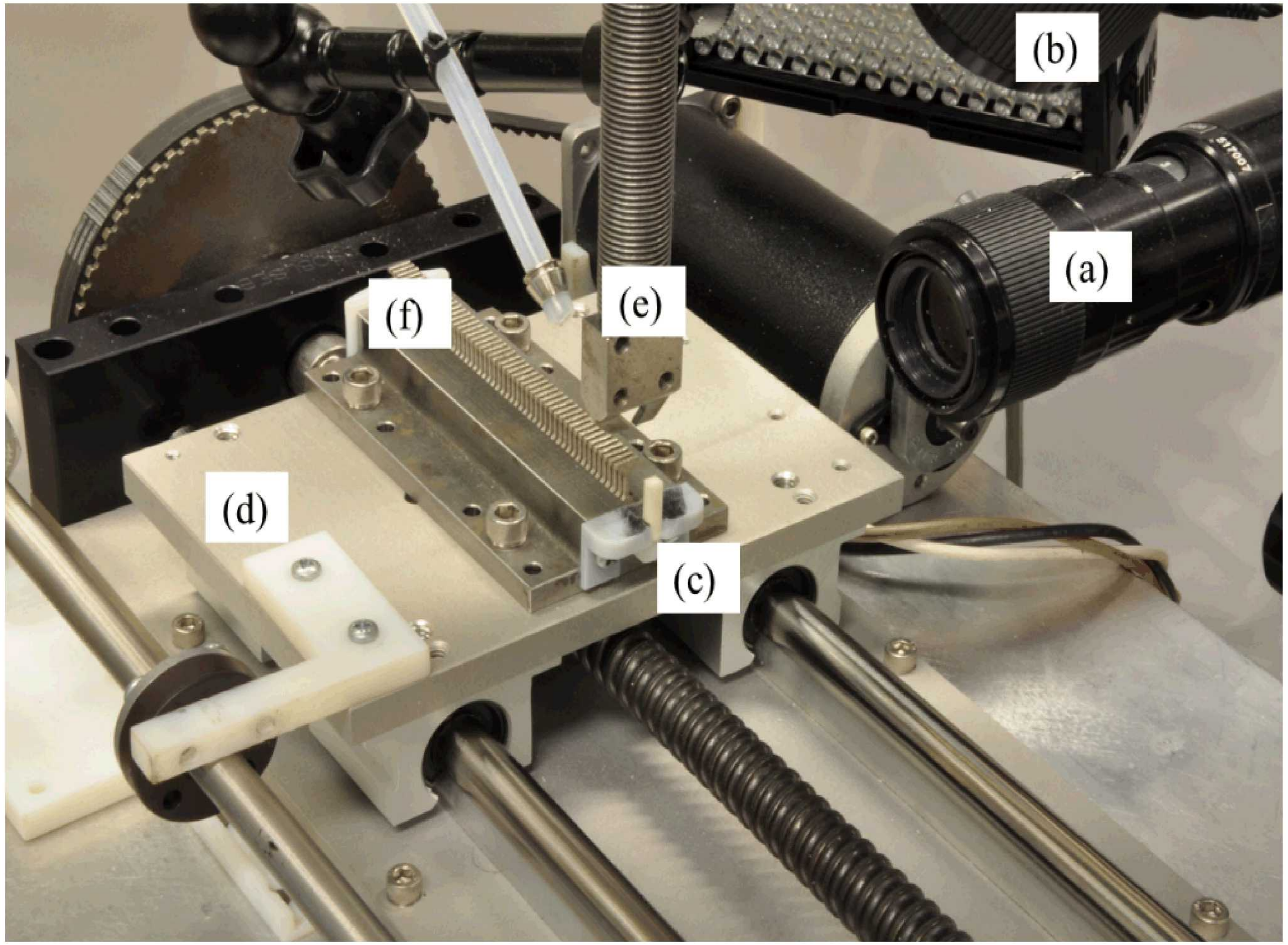
- 1mm x 1mm nominal cross section
- 4-to-1 gauge length ratio to reduce premature necking
- 45° grip head to reduce overhang





High-throughput tensile test setup

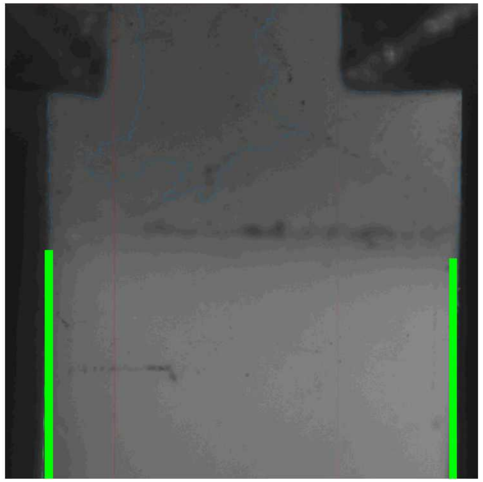
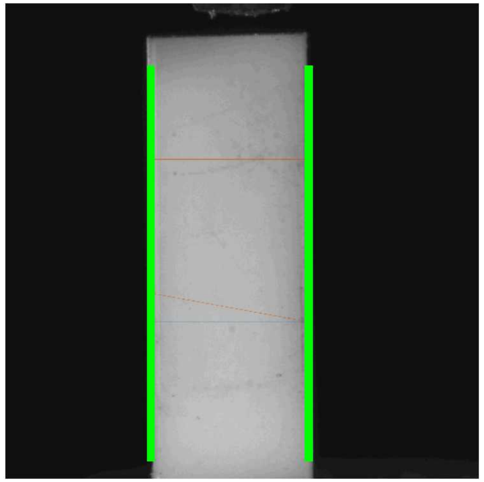
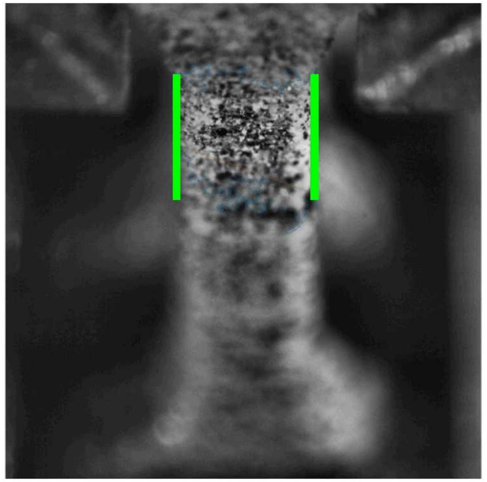
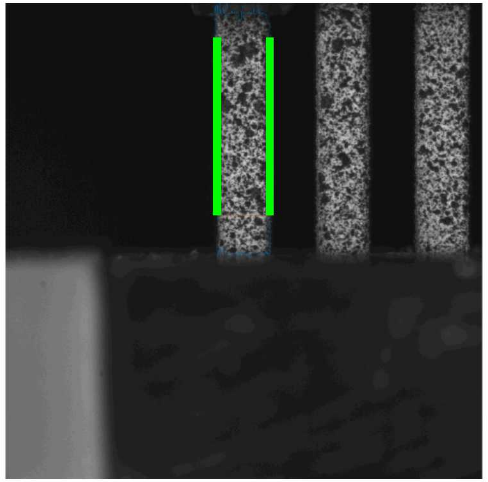




Salzbrenner BC, Rodelas JM, Madison JD, Jared BH, Swiler LP, Shen Y-L, Boyce BL: **High-throughput stochastic tensile performance of additively manufactured stainless steel.** *Journal of Materials Processing Technology* 2017, **241:**1-12.

MatLab Analysis – AVID: Area Values from Image Dimensions ©

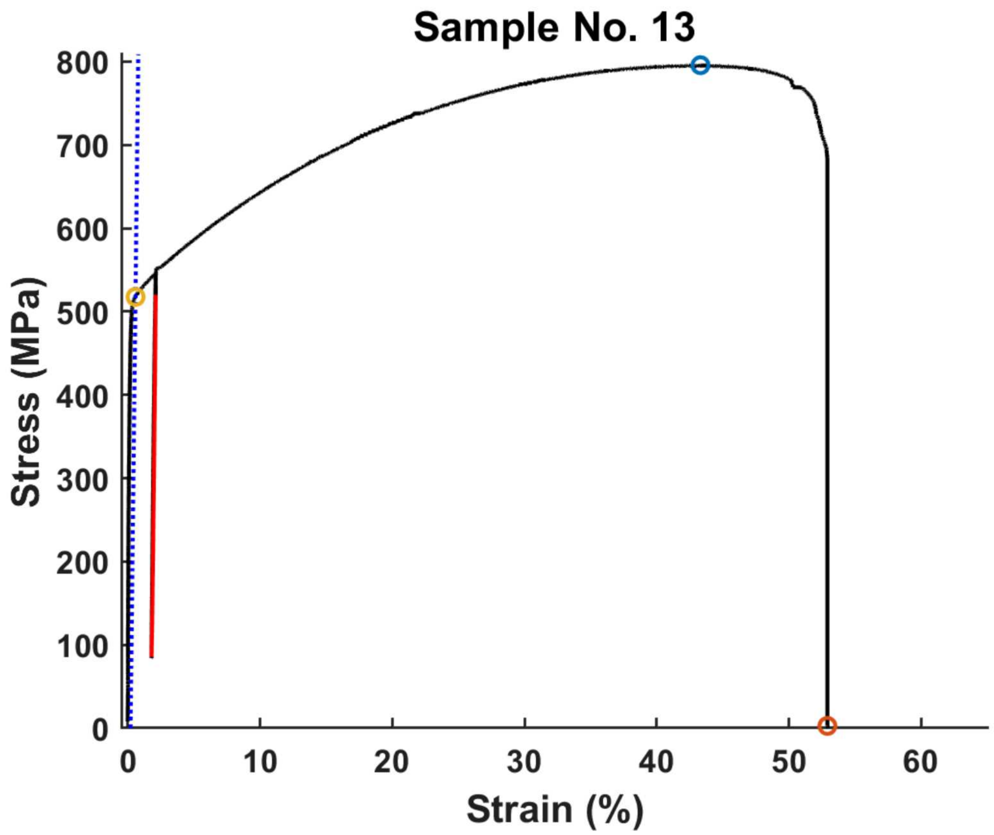
- Edge detection via MatLab image processing toolbox
- 1^{st} degree polynomial line fit
- Contact measurement vs digital measurement error: 3% ± 2% over-estimation

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)



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

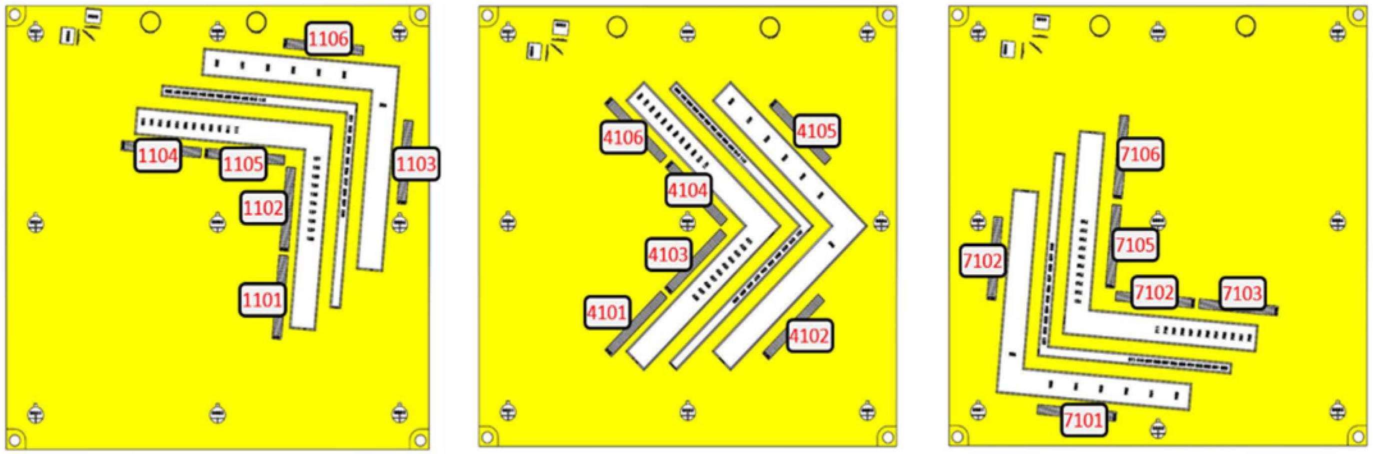
Composition	Units	ASTM F3056	Build 1 (virgin)	Build 2 (virgin)	Build 3 (reuse)
Al	Wt %	0.4 max	0.16	0.16	0.15
B	Wt %	-	<0.0005	<0.0005	<0.0005
C	Wt %	0.10 max	0.035	0.034	0.036
Co	Wt %	1.00 max	0.215	0.21	0.205
Cr	Wt %	20.00-23.00	21.39	21.22	21.23
Fe	Wt %	5.00 max	0.044	0.39	0.063
Mn	Wt %	0.50 max	0.0061	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

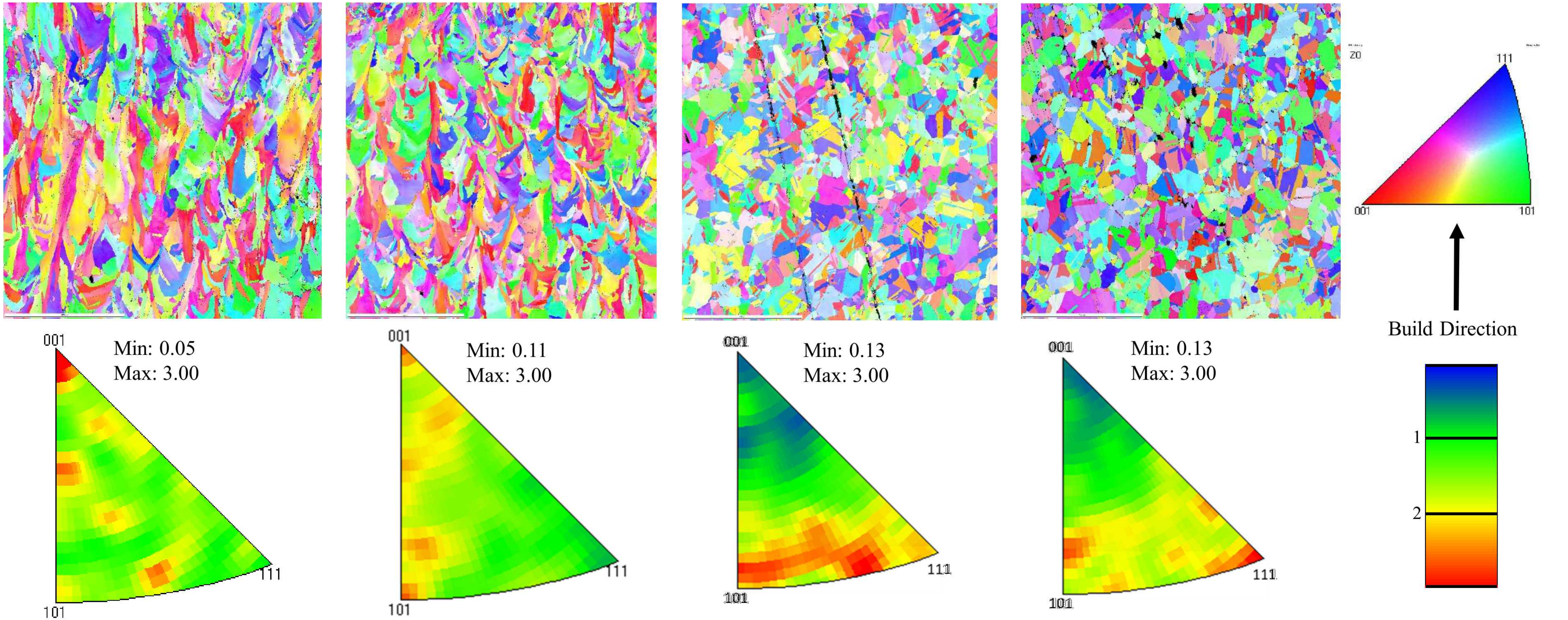
- Stress relieved with all samples attached to build plate
- Portion of virgin build solution annealed
- Portion of virgin build HIPed
- All reused powder stress relieved

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



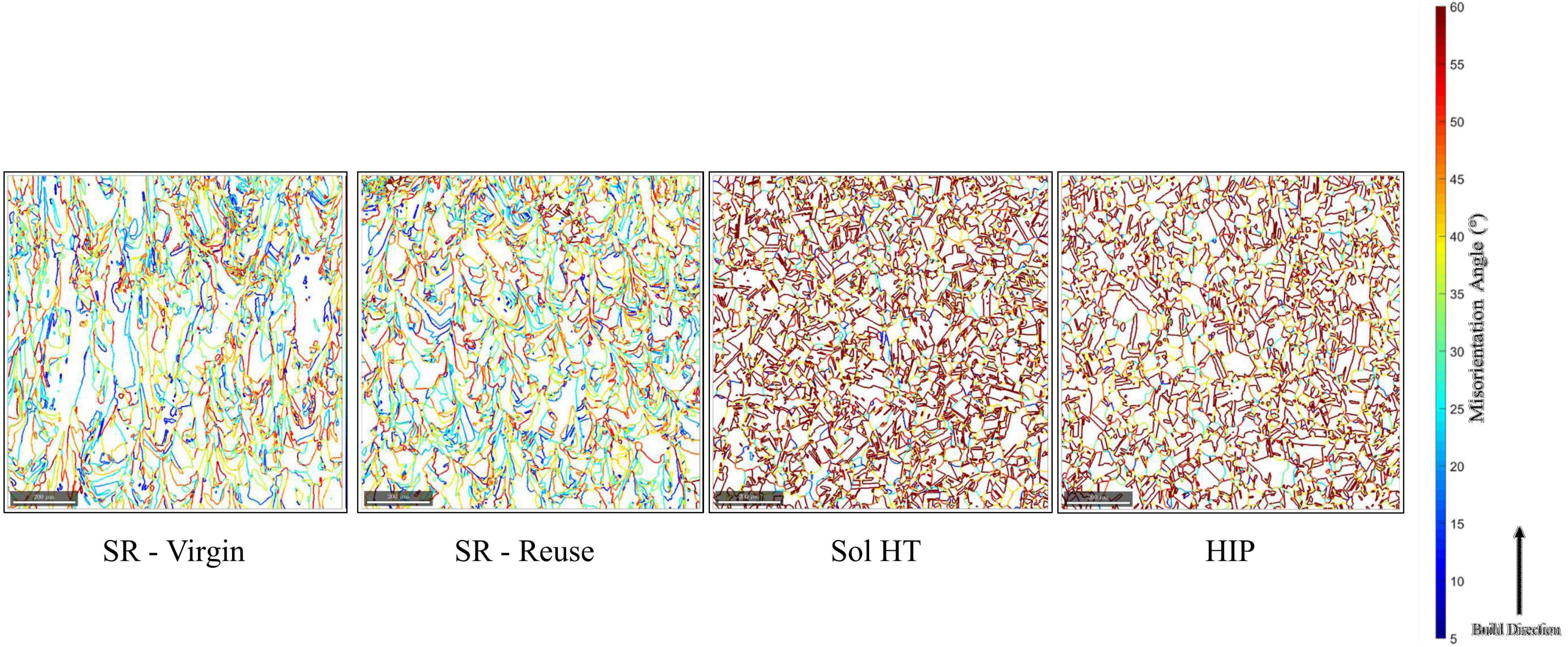
SR - Virgin

SR - Reuse

Sol HT

HIP

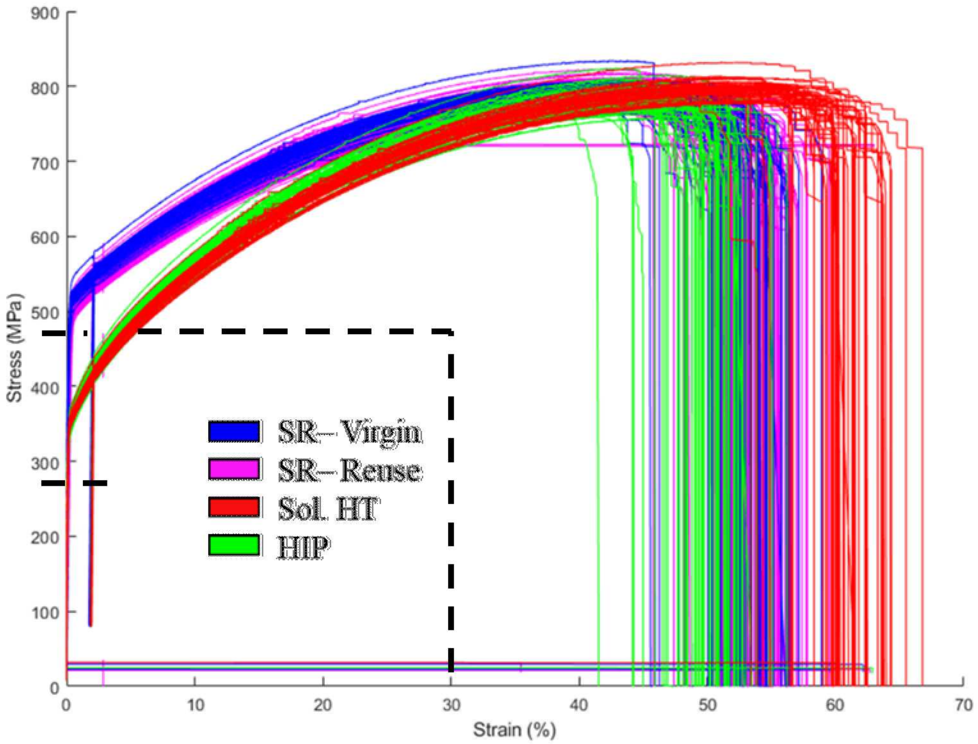
Grain boundary misorientation



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



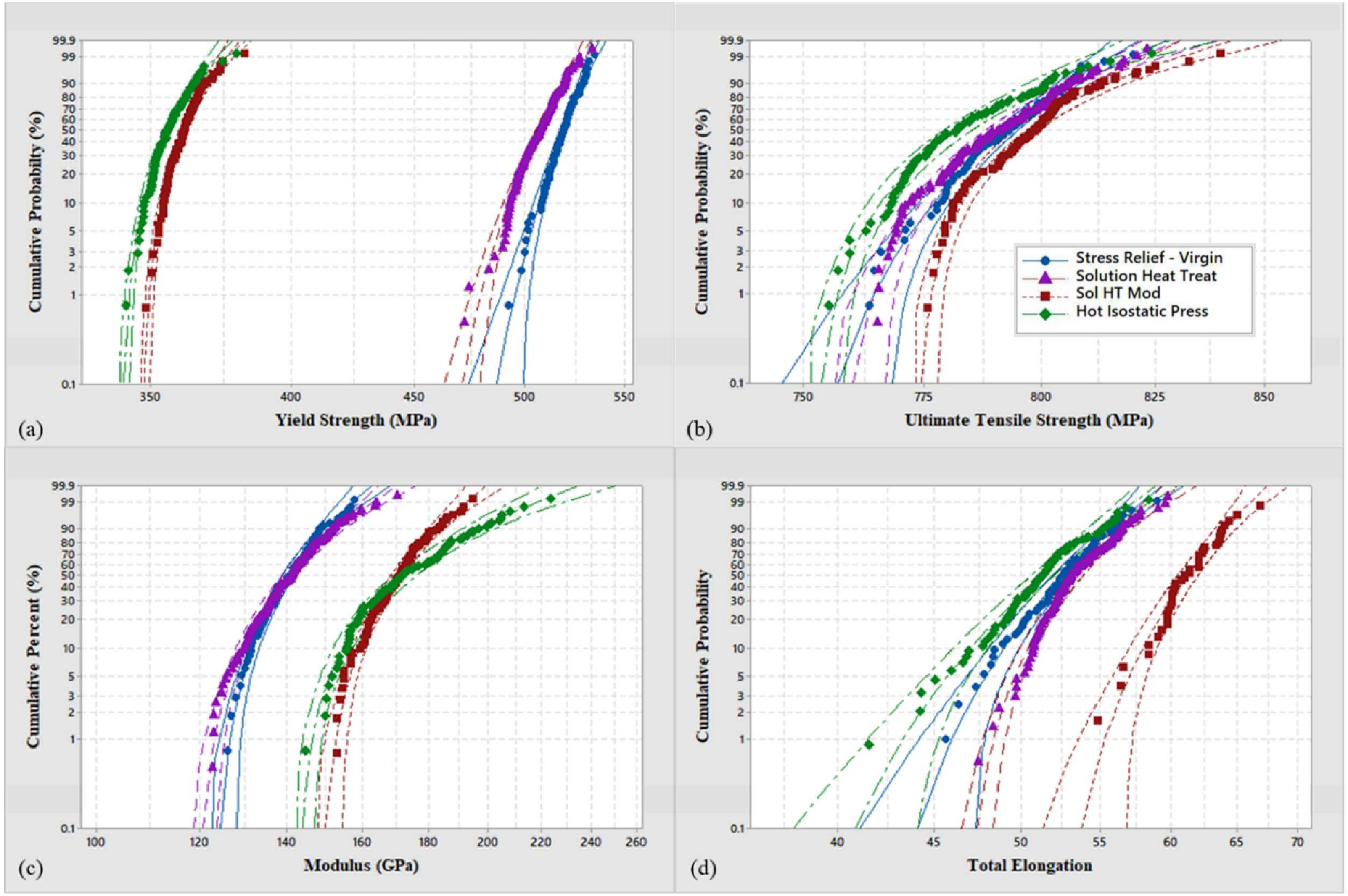
Sandia National Laboratories

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



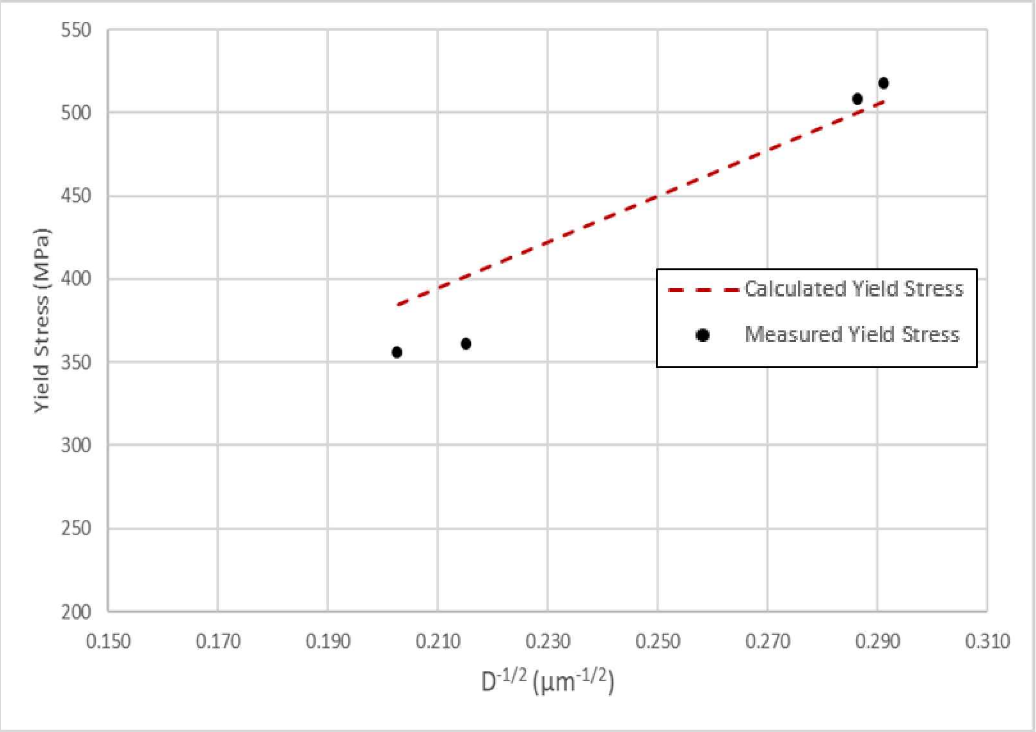
Hall-Petch relation suggests a correlation between yield strength and grain size

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

- 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

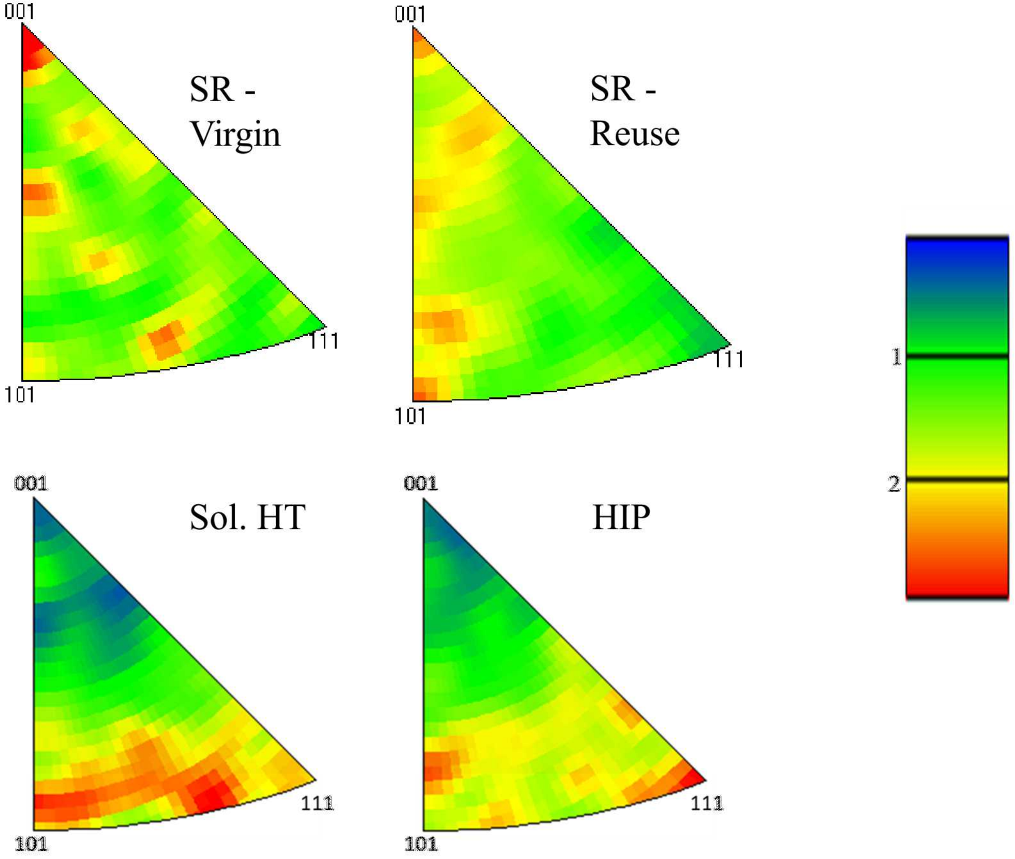
Zener ratio of nickel, Z = 2.54

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

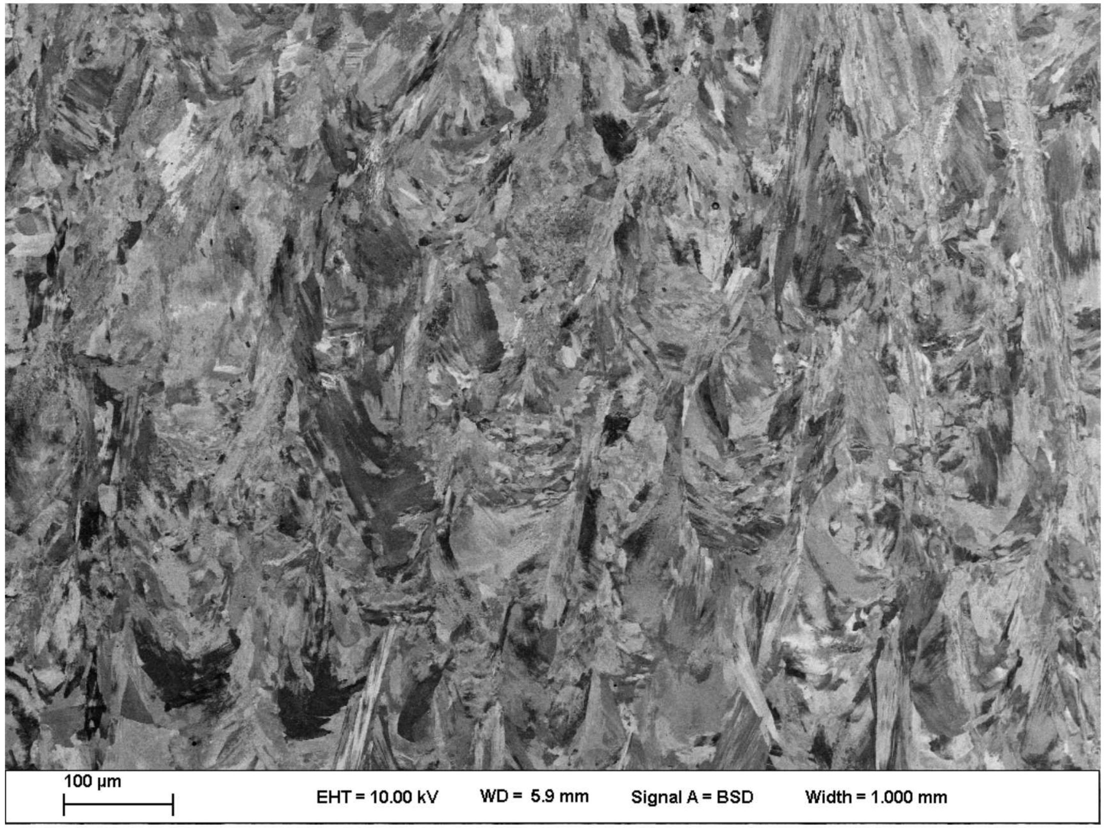
Heat Treatment	SR	Sol HT	HIP
Modulus (GPa)	140	170	174

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

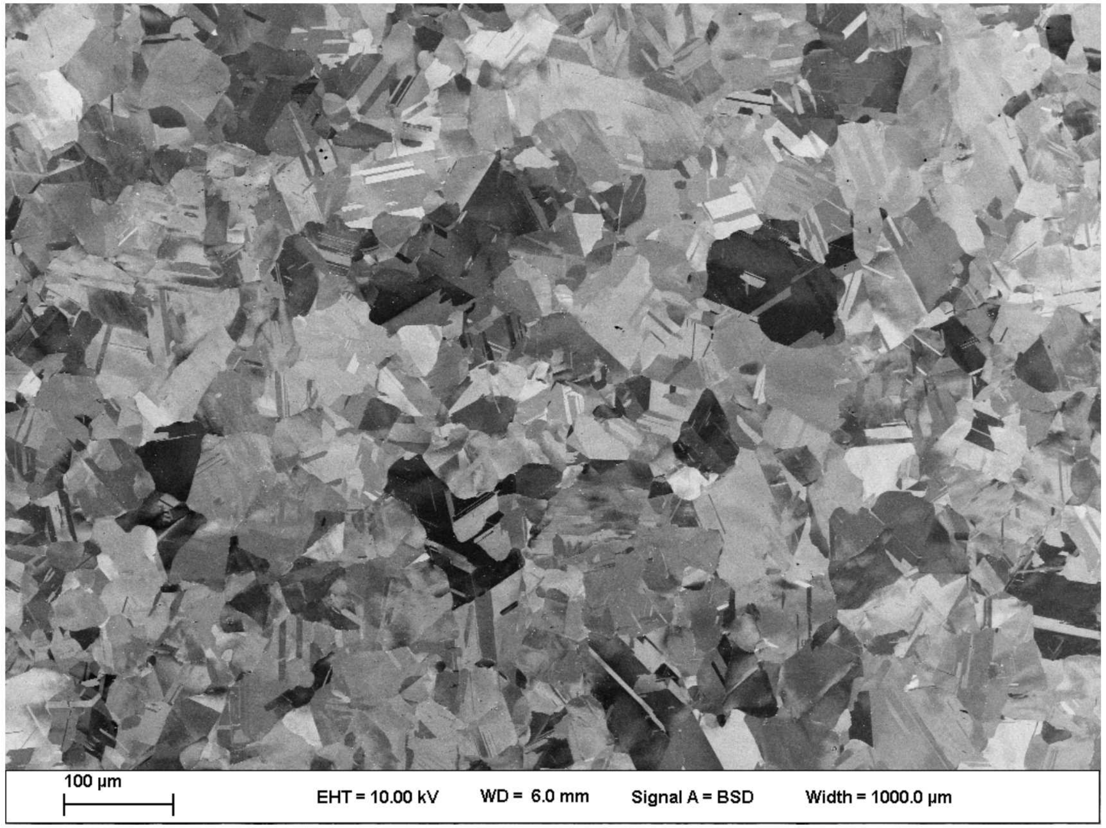
Direction Cosine	[100]	[101]	[111]
Modulus (GPa)	164	234	271



Columnar grain growth *may* exaggerate the anisotropic behavior



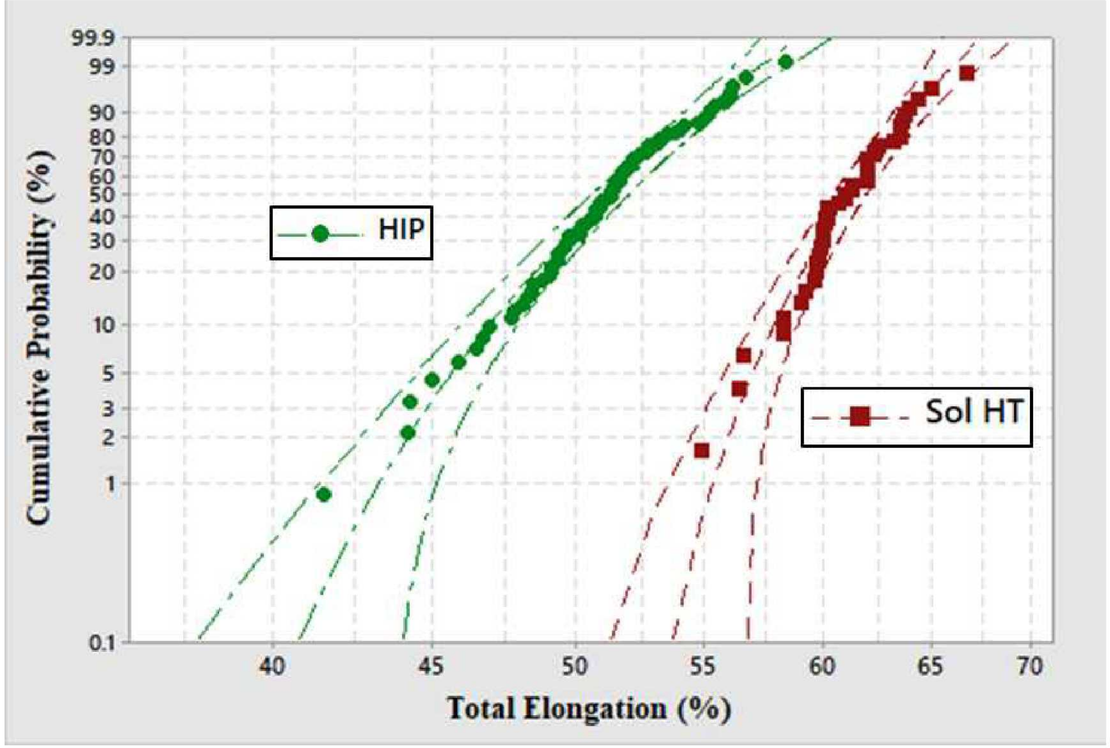
SR – Virgin



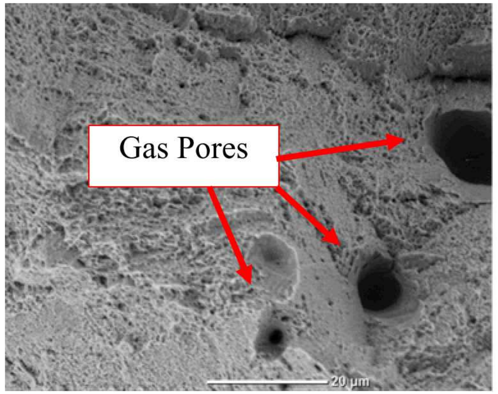
HIP

The HIP samples show *lowest* ductility

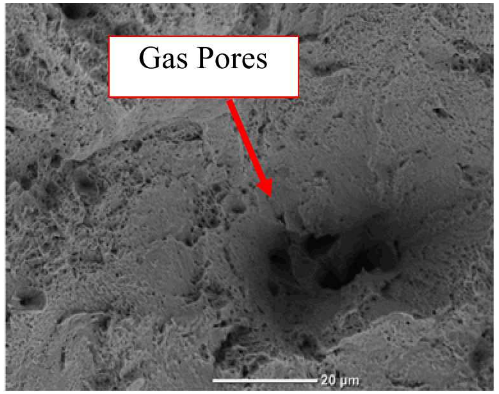
- Analysis methods:
  - Determination of porosity
  - Microstructure analysis



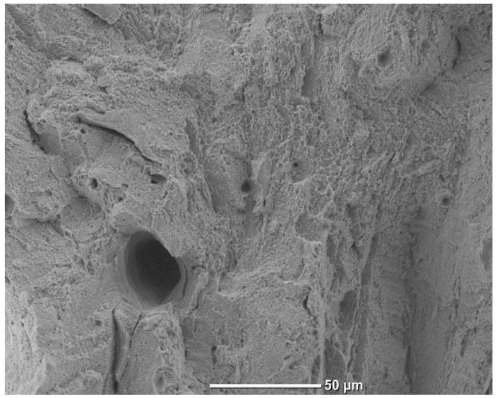
Inconel 625 – Fracture Surfaces and Porosity



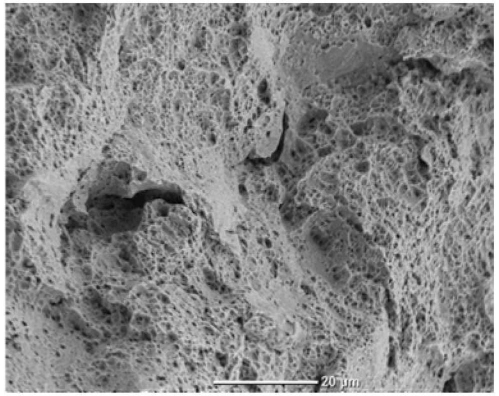
(a) SR Virgin



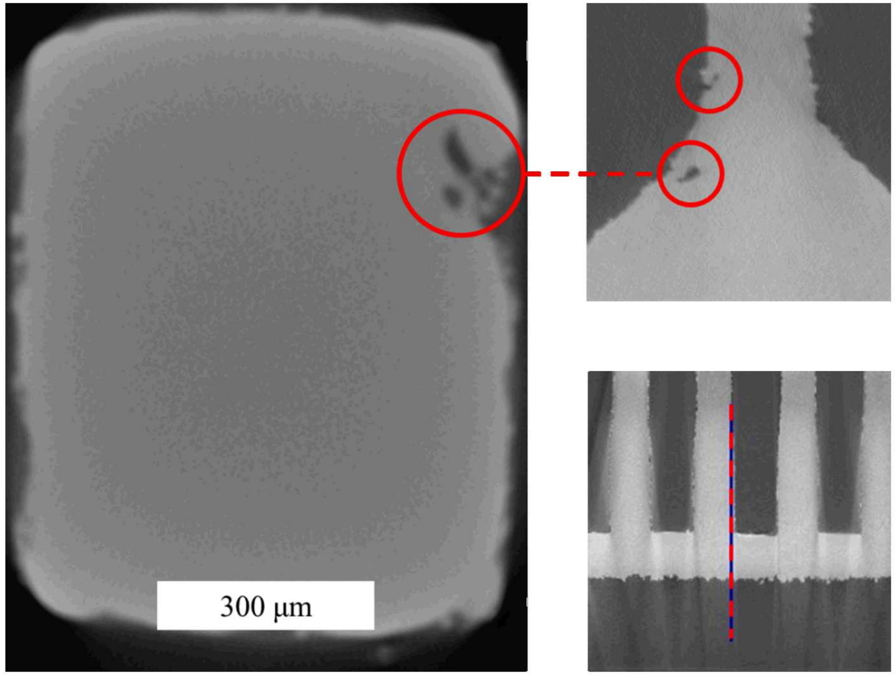
(b) SR Reuse



(c) Sol HT

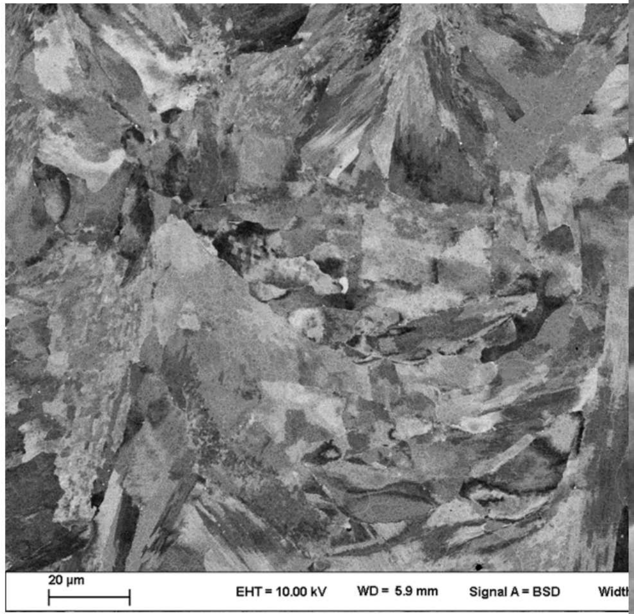


(d) HIP

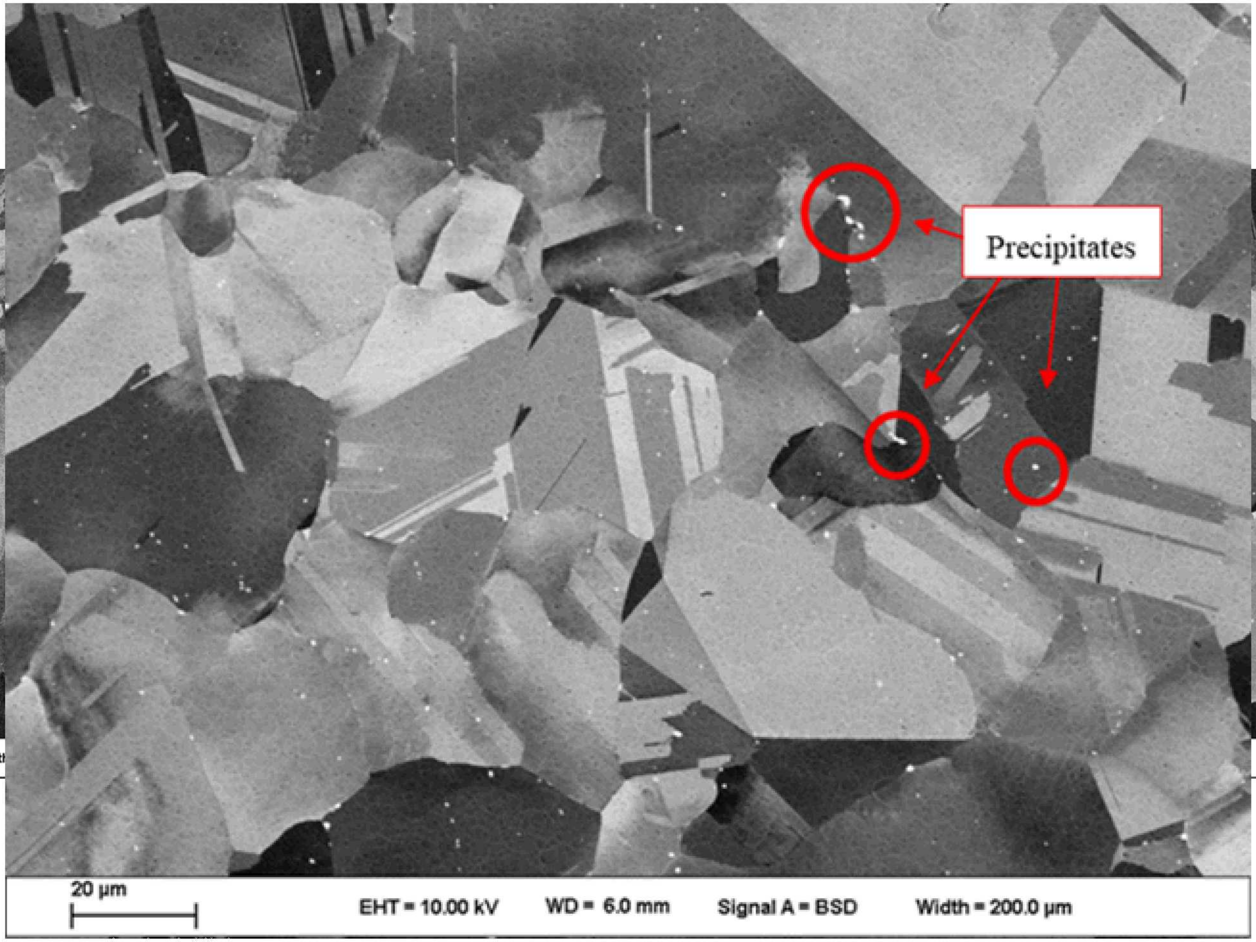


HIP

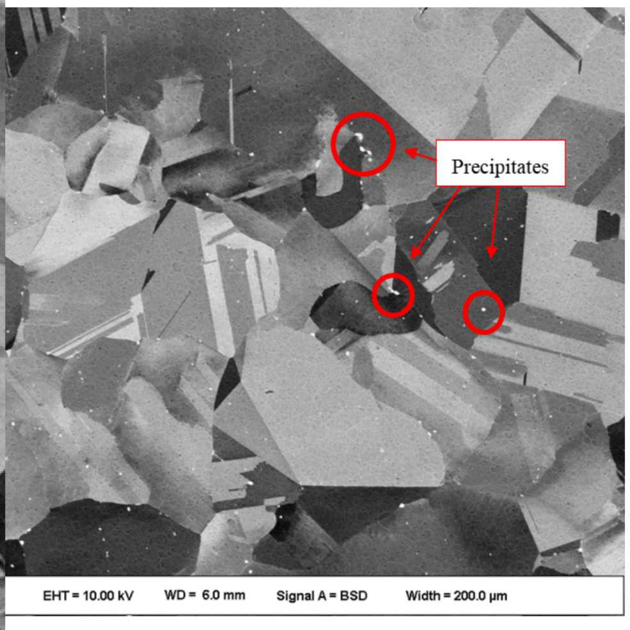
SEM images of the stress relieved, solution annealed, and HIP samples show an increase in population and size of precipitates in the HIP material



Stress Relief

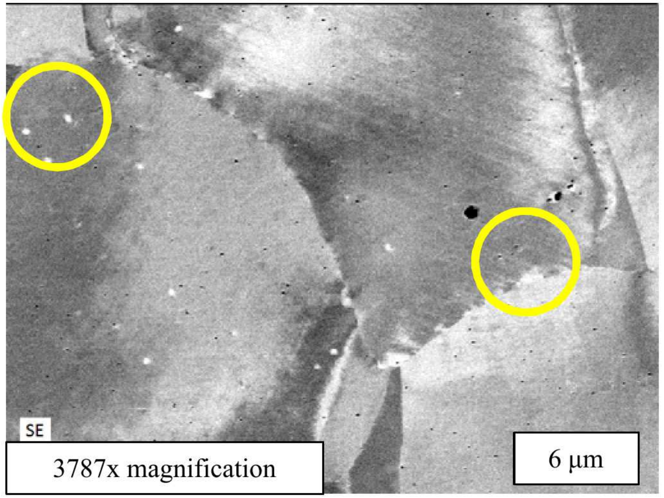


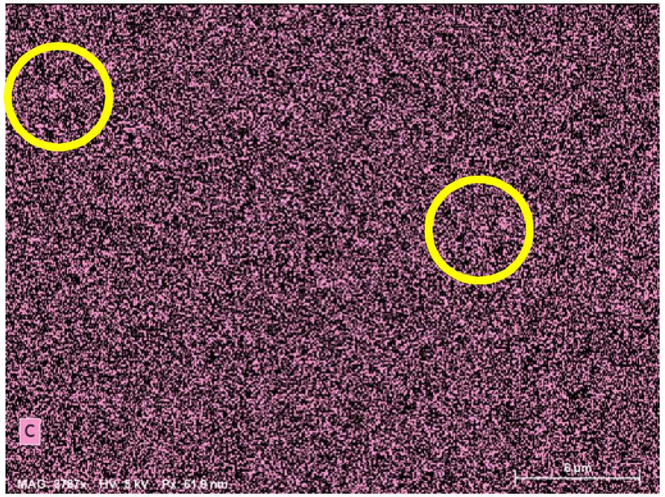
HIP

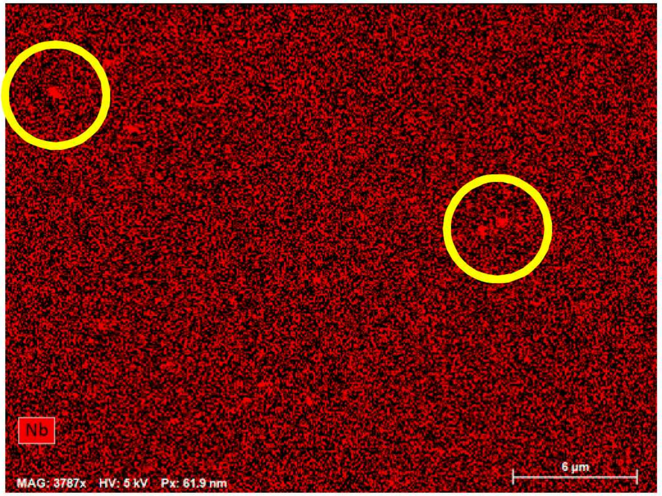


HIP

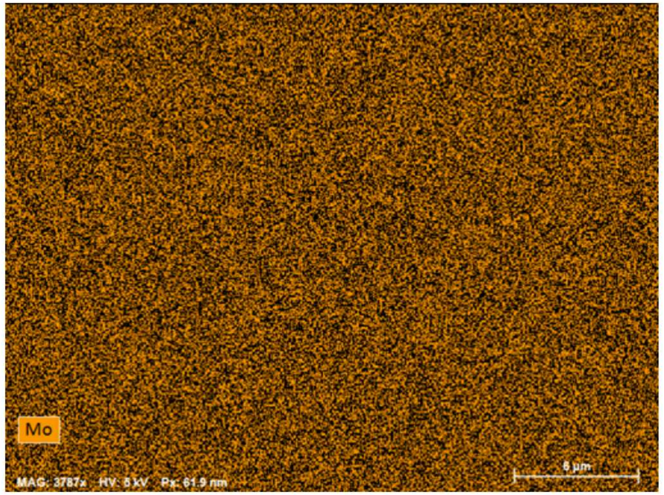
Inconel 625 – Solution Annealed Precipitates

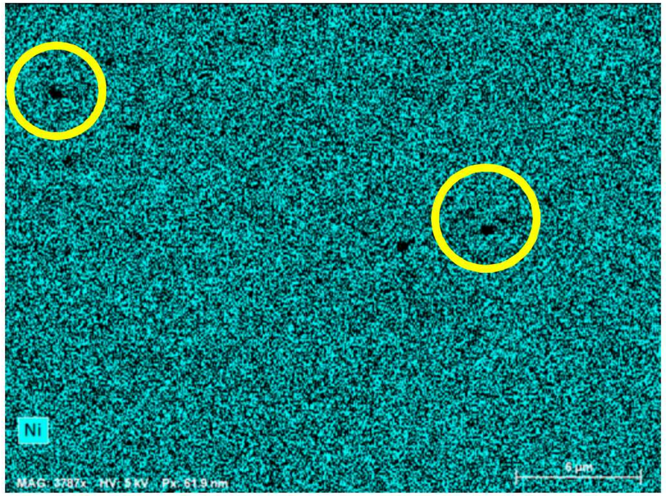




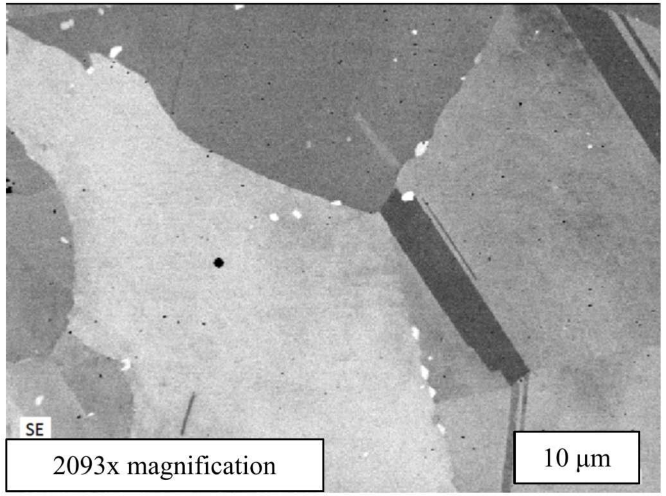


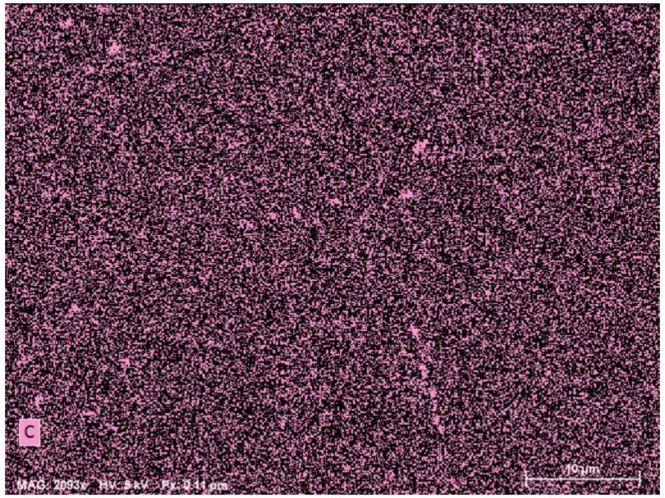
Precipitate size:
~340 nm

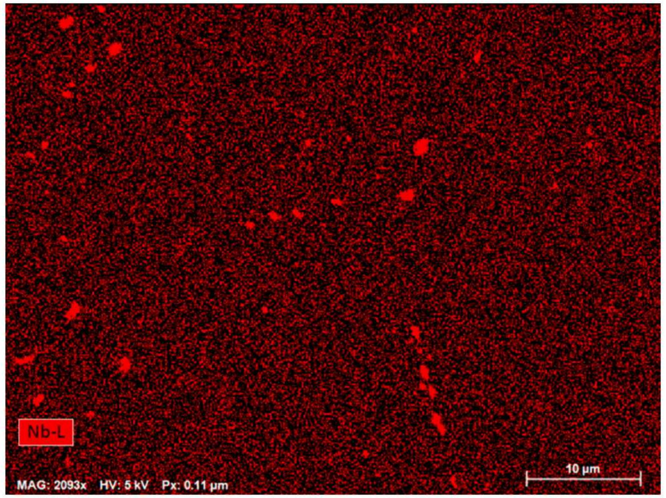




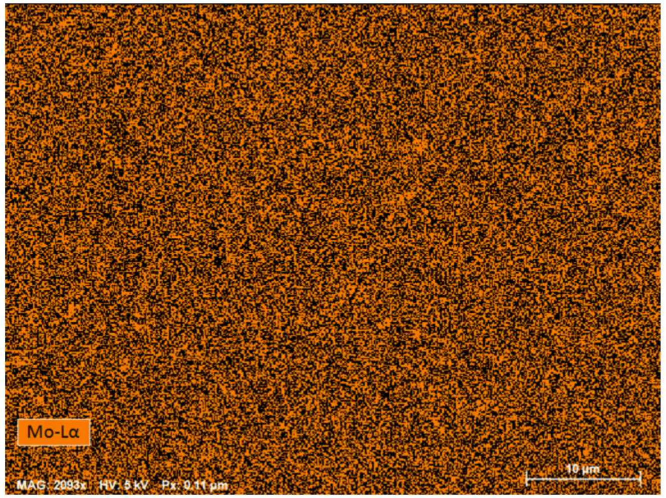
Inconel 625 – HIP Precipitates

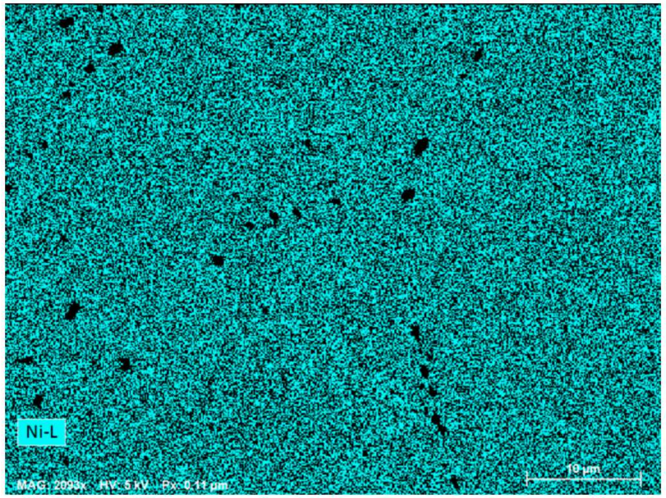






Precipitate size:
~900 nm





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