This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.

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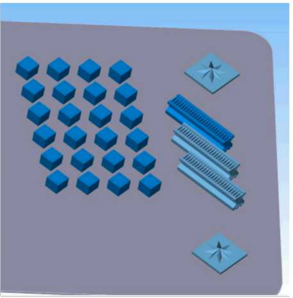

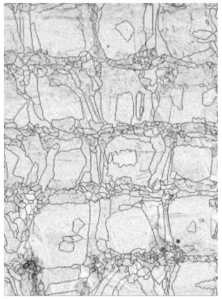

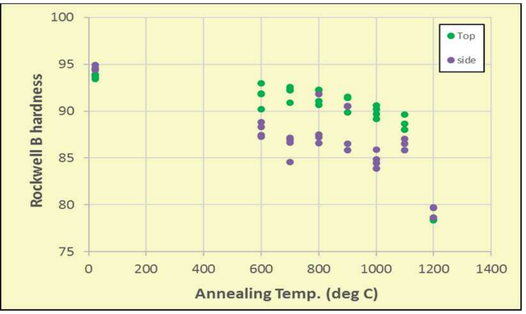

The Effects of Annealing Treatments on AM 316L Stainless Steel

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Sandia National Laboratories

Albuquerque, NM USA

AM JOWOG

October 25, 2018

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Collaborators

Dennis DeSmet: heat treatment

Jay Carroll: tensile testing

James Griego: XRD analysis

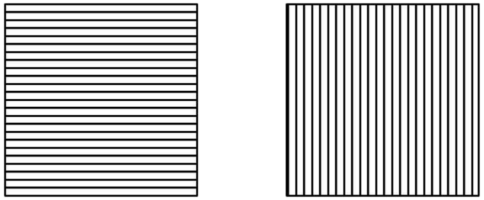
John Lopez and Ryan Andersen: AM support

Approach

- 316L stainless steel was deposited using a laser powder bed system, ProX 300
- Post-deposition annealing performed between 600°C-1200°C with the objectives of decreasing internal residual stress and maximizing ductility and toughness
- The annealing response was compared to examples of wrought stainless steel heat treatment as well as other AM stainless steel examples

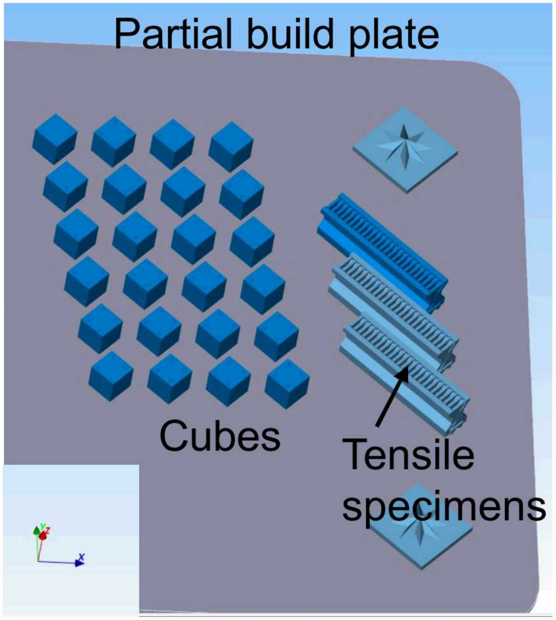
General Theme: AM material is resistant to annealing, relative to wrought material of the same alloy at the same annealing temperature

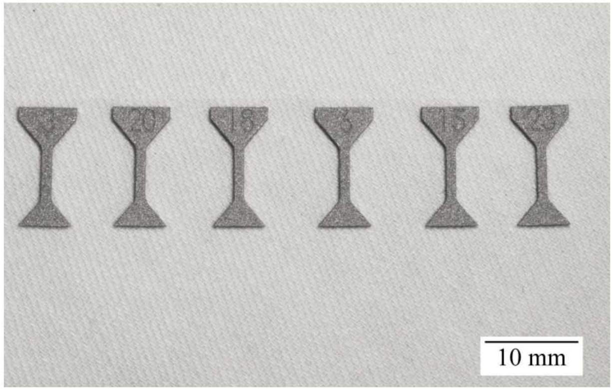
Laser Powder Bed Additive Manufacturing (AM)



ProX 300 laser powder bed machine, 3D Systems Inc.

- 316L stainless steel powder
- Cube shaped samples, 1cm across, built with alternating laser path directions, i.e. cross-hatch pattern. Contour pass applied around the edges of the cube.
- Dogbone tensile bars also produced using same process

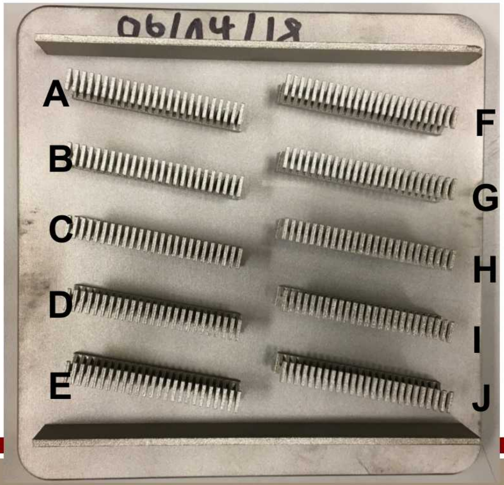


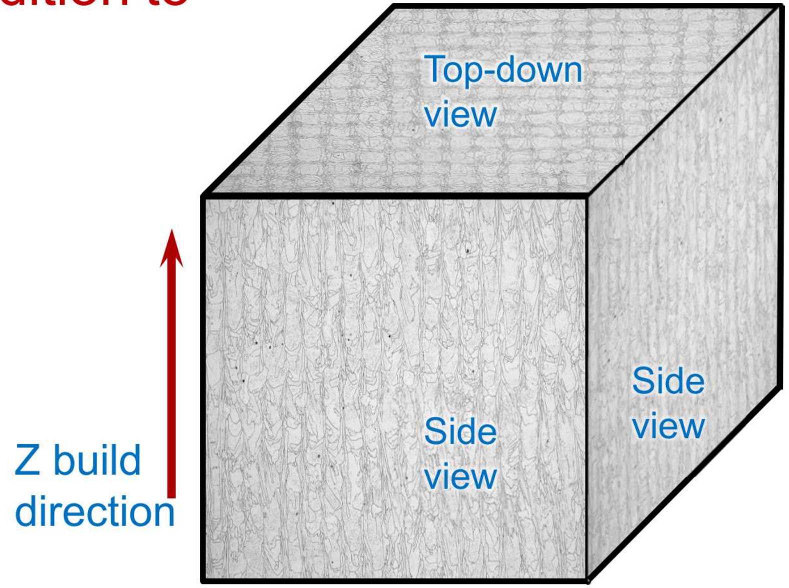


Heat Treatment and Characterization

Annealing treatments applied to investigate recrystallization and grain growth regimes

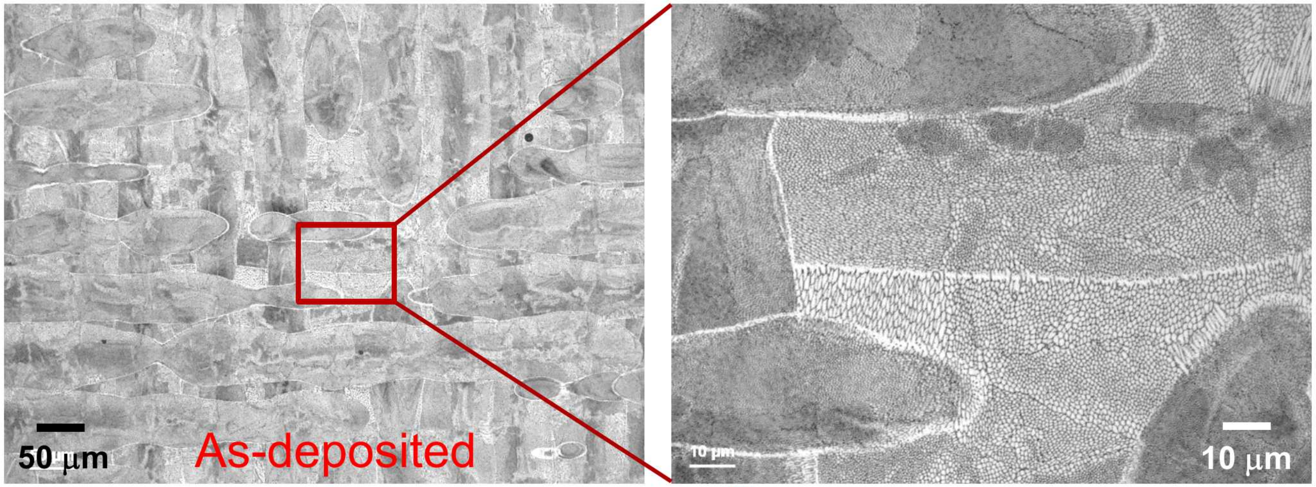
- Temperatures 600-1200°C, argon atmosphere
- Annealing time held constant at 2 hrs.
- Rockwell B hardness indentation performed in the “top-down” and “side” directions. Vickers microhardness maps of entire cube faces.
- 25+ tensile bars tested at each condition to develop statistical data using a “high-throughput” technique



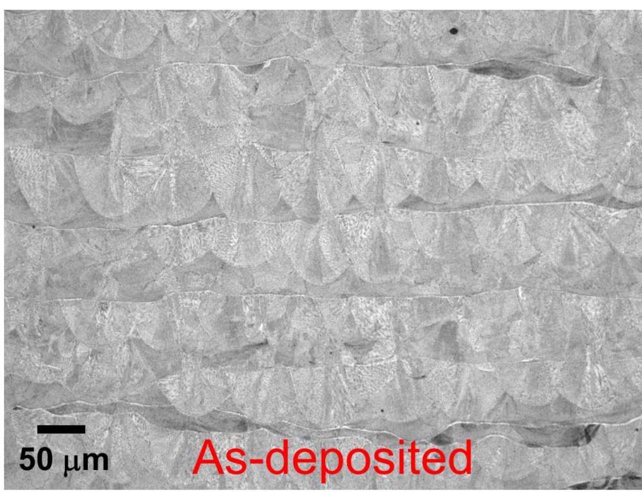


As-deposited Microstructure

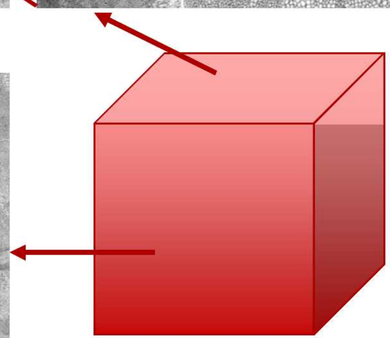
Top-down view



"Basket-weave" pattern visible in the top down view. Fine-scale cellular solidification sub-structure in the as-deposited condition.

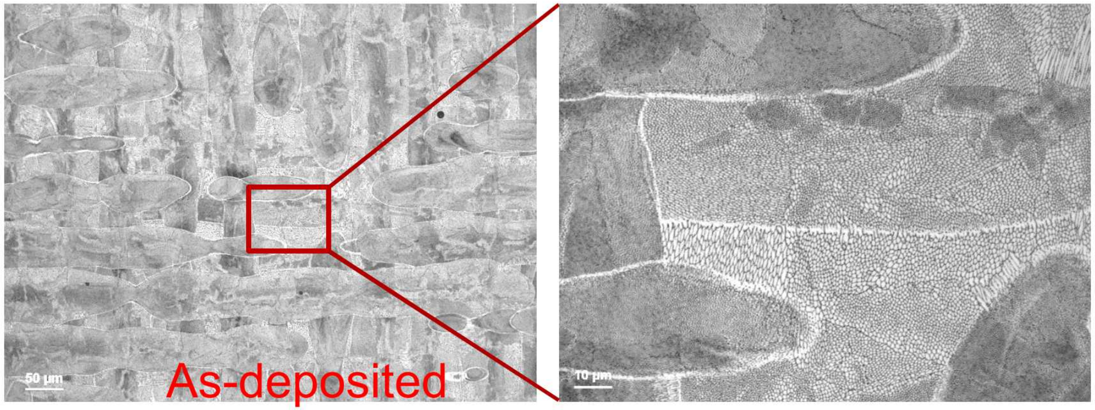


Side view

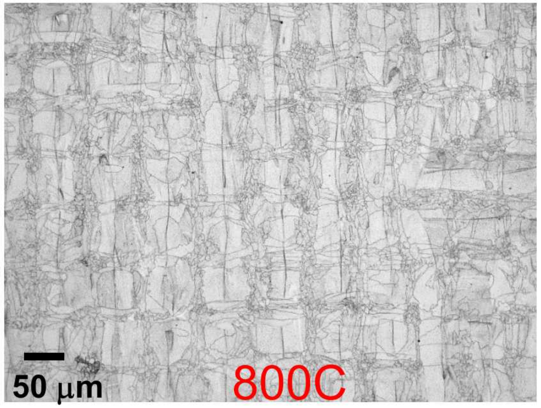


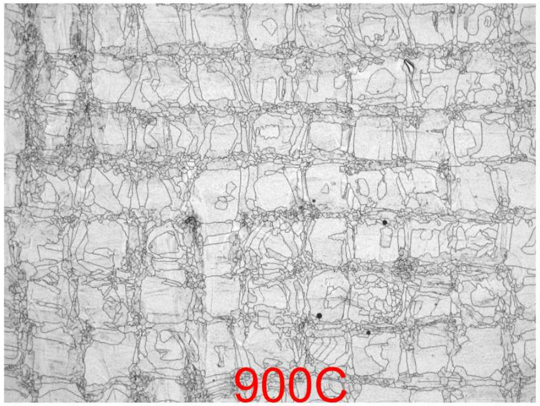
Layers of overlapping laser welds are visible in side-view.

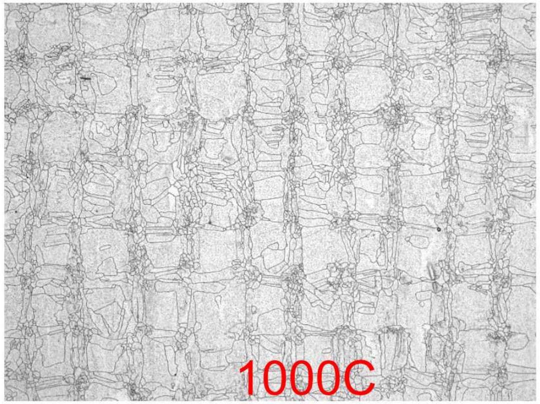
Effects of Annealing: Top-down view

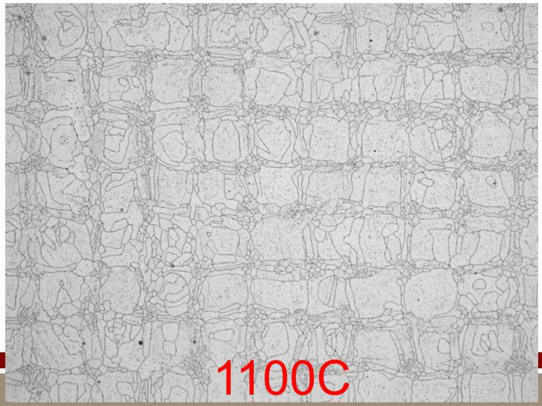


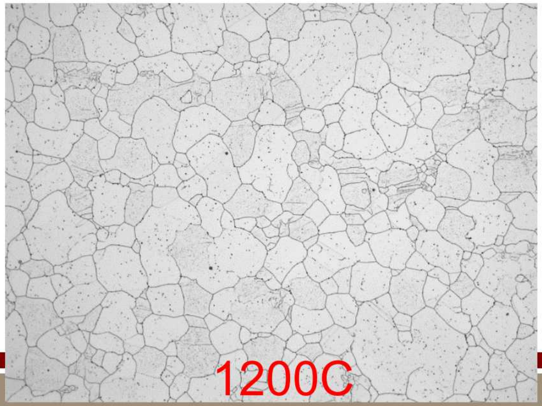
Unusual “square shaped” grains dominate this view, with finer grains in between. Remnant of build scan pattern.





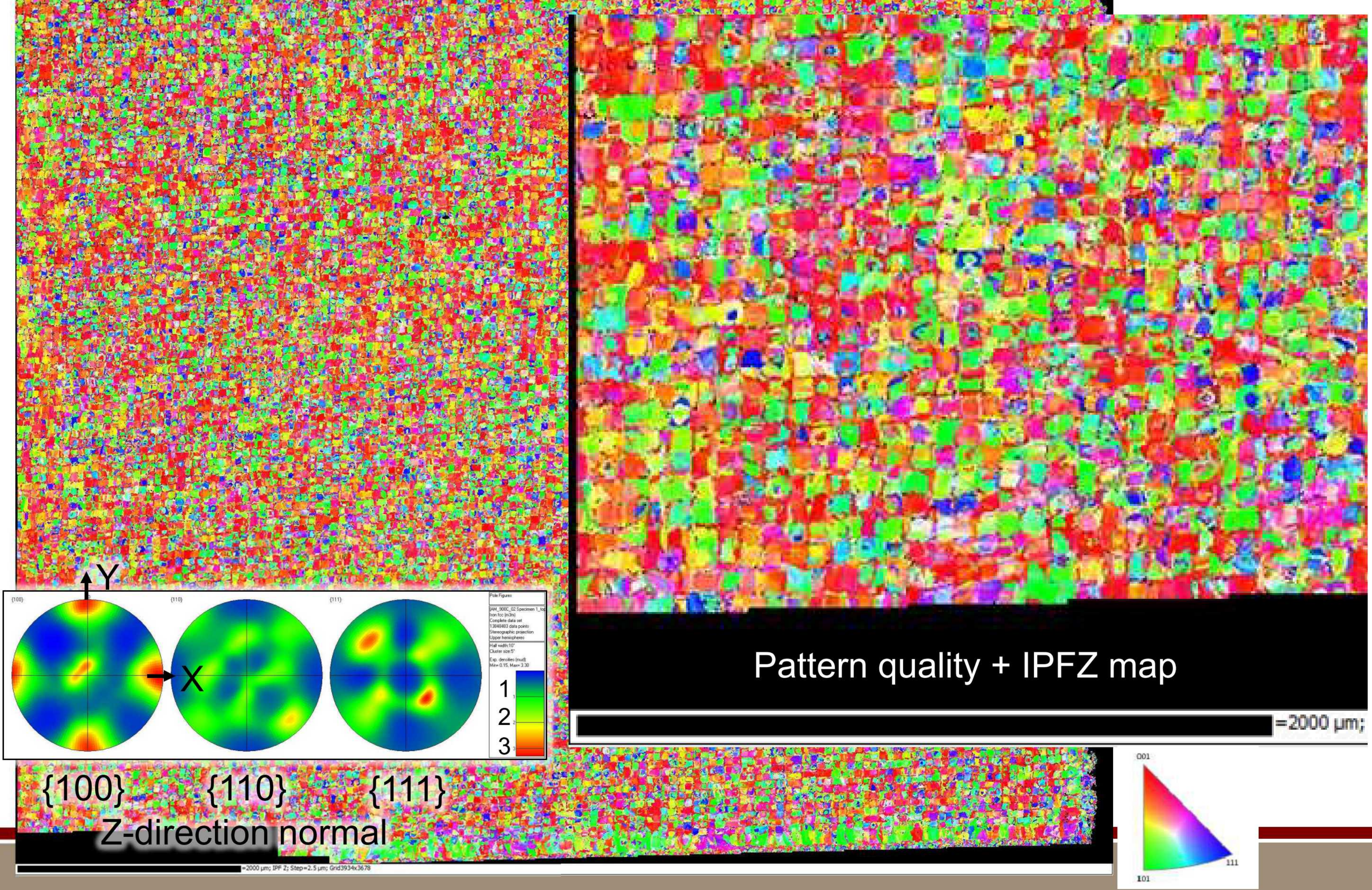






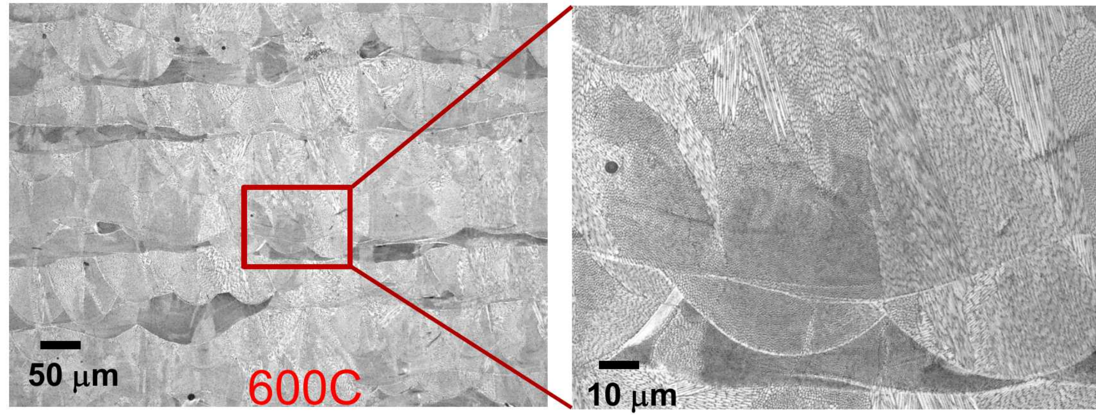
The structure changes only slightly until 1200°C, replaced by relatively large equiaxed grains with no remnant of the deposited structure.

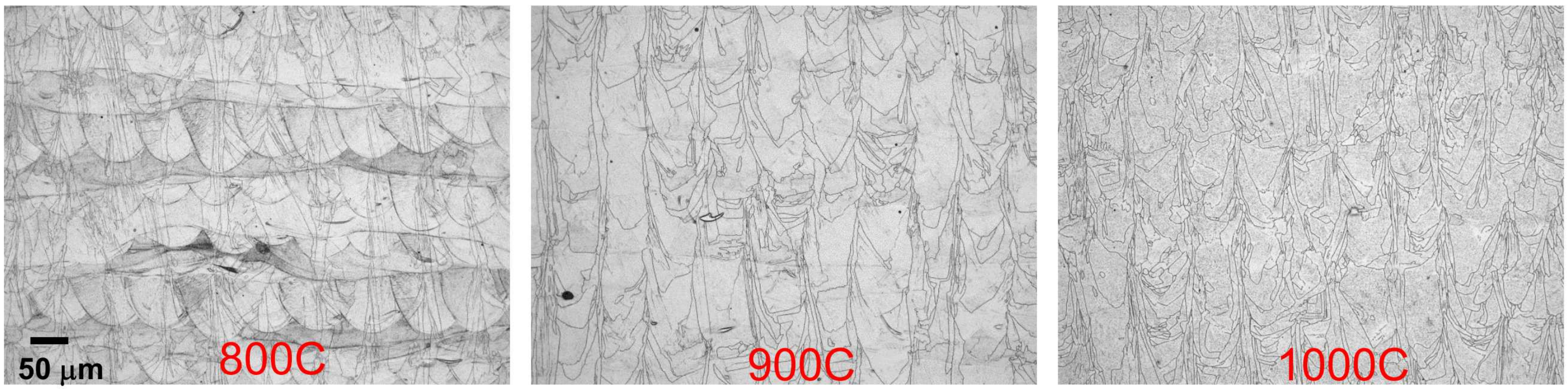
EBSD, 900C anneal: slight (100) texture in build direction



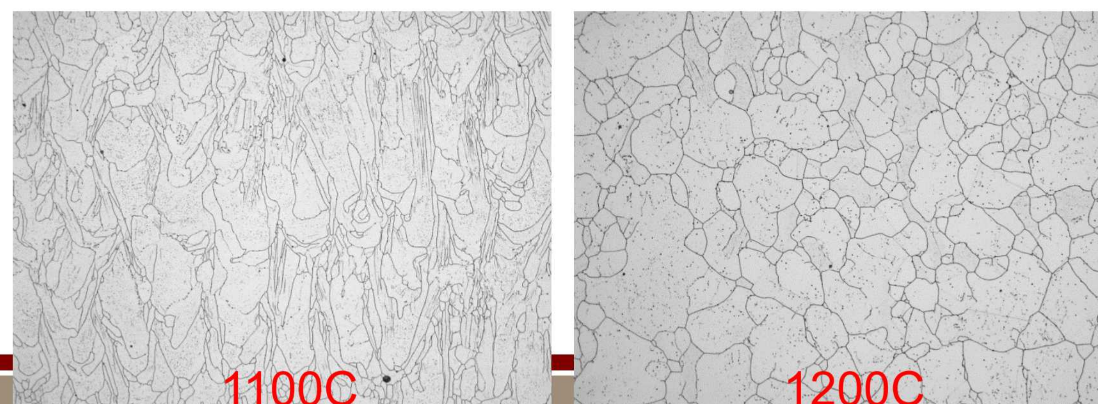
Effects of Annealing: Side View

Fine-scale cellular structure is annealed out between 700 and 800°C. A roughly columnar grain structure develops at intermediate annealing temperatures – spans through multiple build layers.

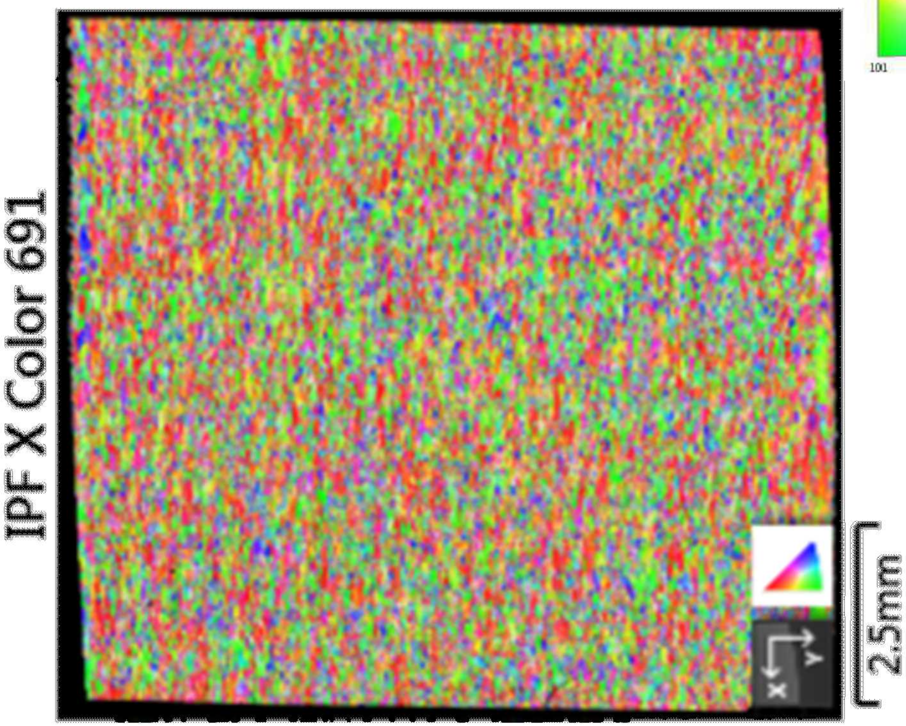


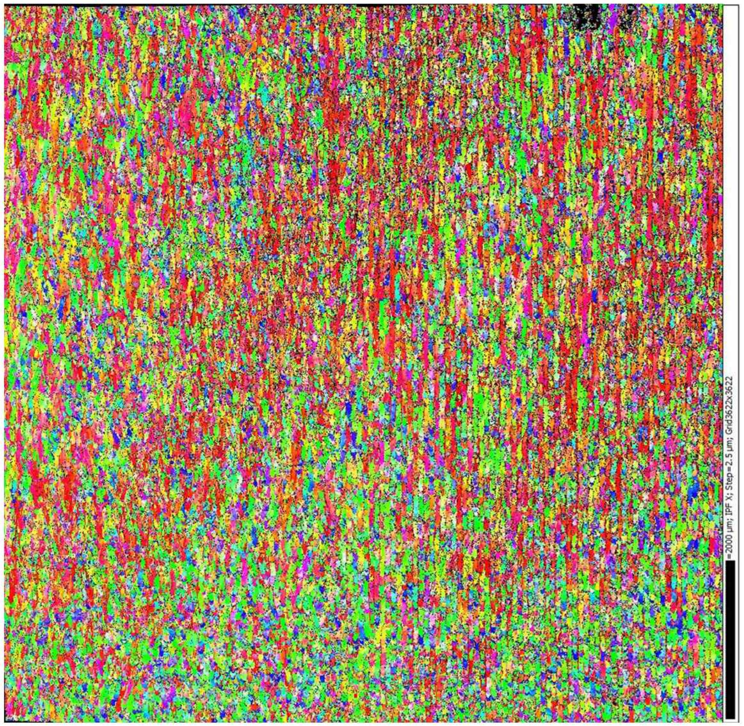


After 1200°C, the microstructure is almost identical to the top-down view. (Note very low amount of porosity in these builds.)



EBSD side view: As-dep. Vs. 900C slight texture changes in build direction





As-deposited

Pattern quality
+ IPFY maps

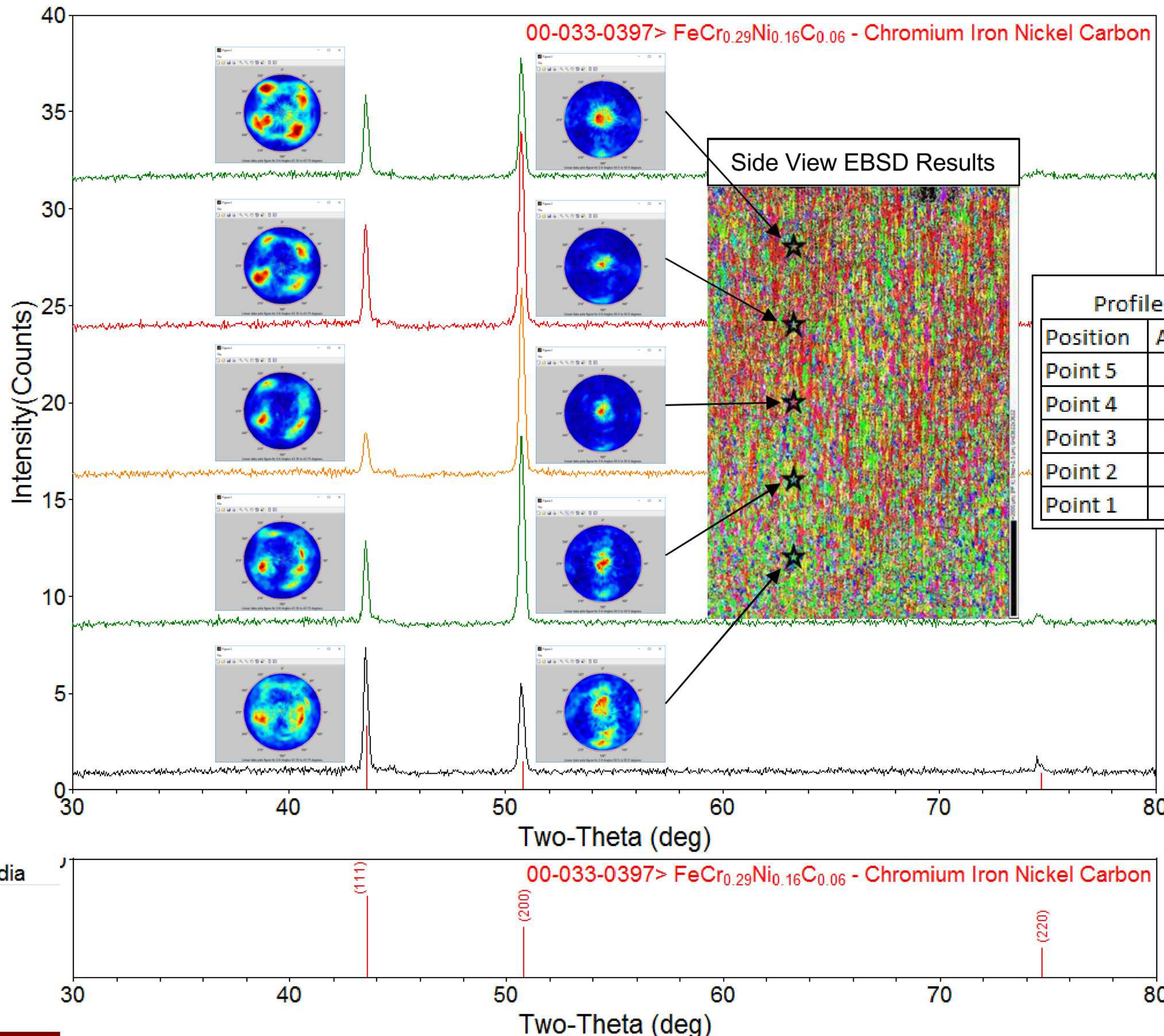
900C anneal

Investigated Further with Pole Figure XRD Analysis with small beam size

AM 316L 900C Side, Points 1 to 5: All XRD patterns are in the standard XRD geometry (i.e. no Chi tilt) with the 60 individual Phi rotations merged into one XRD plot

Scan Information:

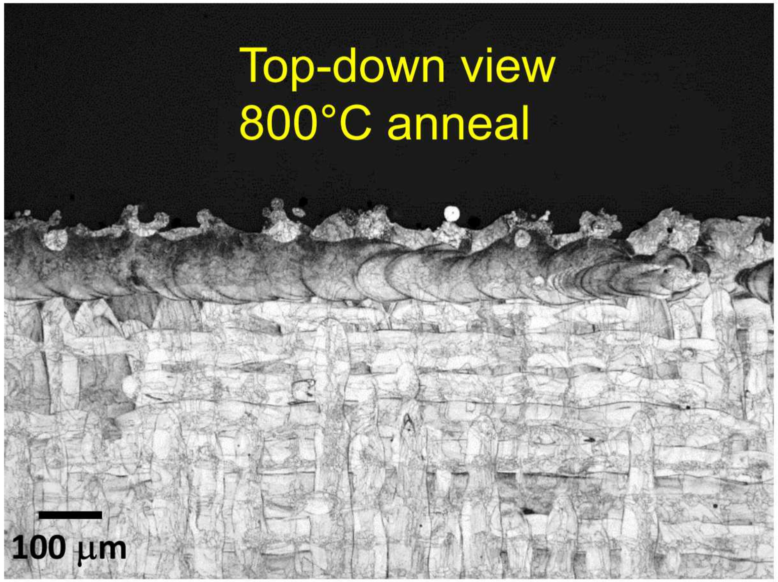
- Instrument Name: Bruker D8 Discover micro-diffractometer equipped with a Vantec-2000 2D detector
- X-ray Tube type: Cu with Nickel foil filtering
- Tube voltage and current (kV , mA): 40 , 40
- X-ray spot size (μm): 500
- # of scans total: 3360 (~20 hr to complete)
- Time/scan (s): 10
- # of ψ-tilts and ϕ-rotations: 14 and 60 (for each ψ-tilt, 60 rotations needed)
- ψ-tilt range: 0° to 78
- ϕ-rotation range: 0° to 354° (there are 6° steps in-between)
- In order to span 15-80° 2θ, 4 separate detector frames are required. This results in 840 XRD scans that can then be used to perform Pole Figure Analysis.

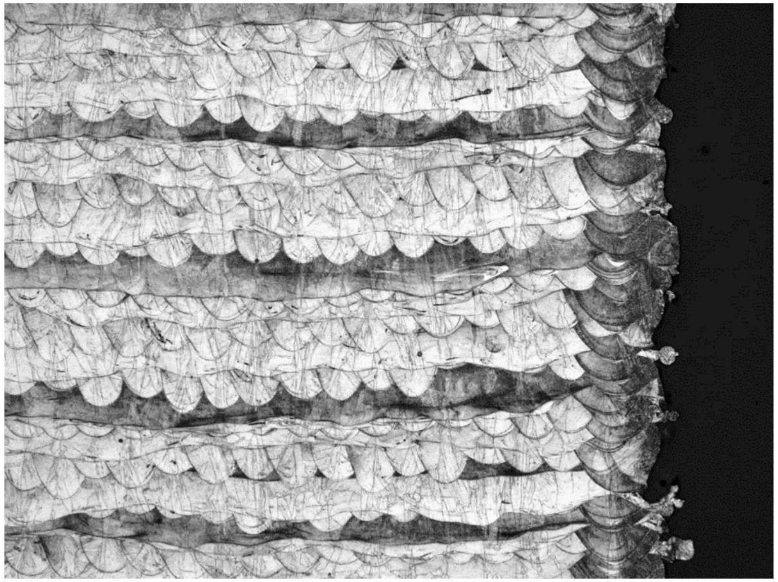


Profile Fit Results	
Position	Area (111)/(200)
Point 5	0.6
Point 4	0.4
Point 3	0.2
Point 2	0.4
Point 1	1.2

Microstructure of Contour Pass (Edge Effect) Regions

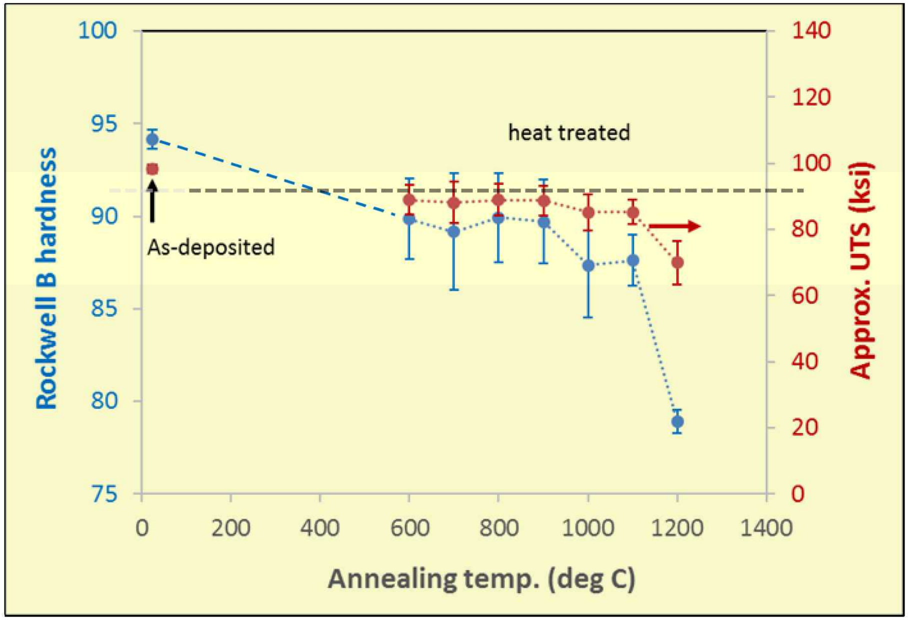
Surface contour passes employed different scan parameters than the interior passes. The contour passes appear wider, with absence of overlap effects. Unmelted/partially melted surface particles are common.





Side view
800°C anneal

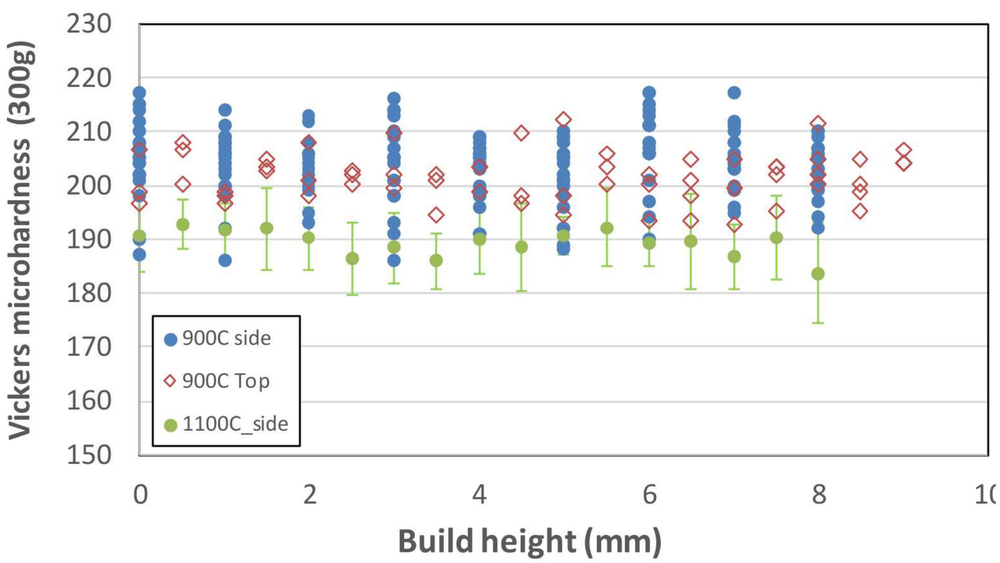
Rockwell B Hardness

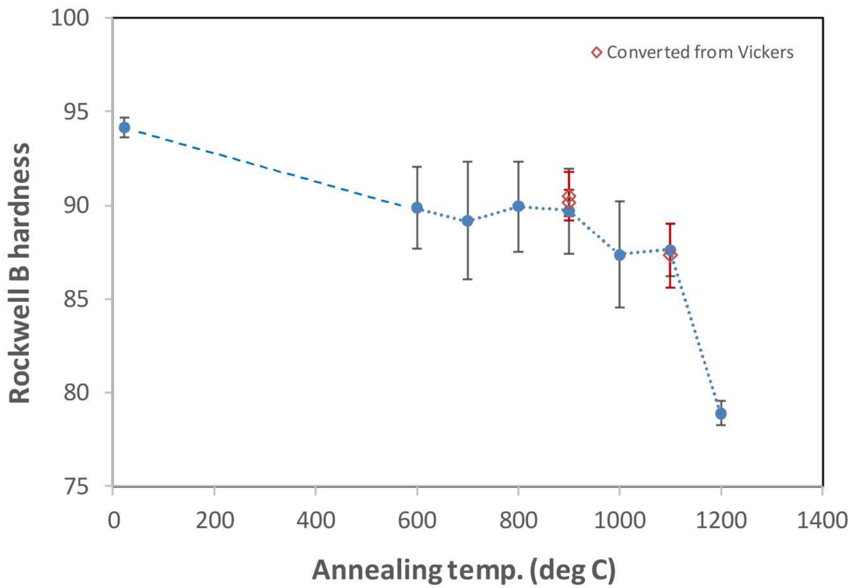


Initial decrease in hardness from ~94 HRB in the as-deposited condition to ~90 HRB with 600°C anneal. Hardness then decreases only moderately until a significant drop at 1200°C anneal.

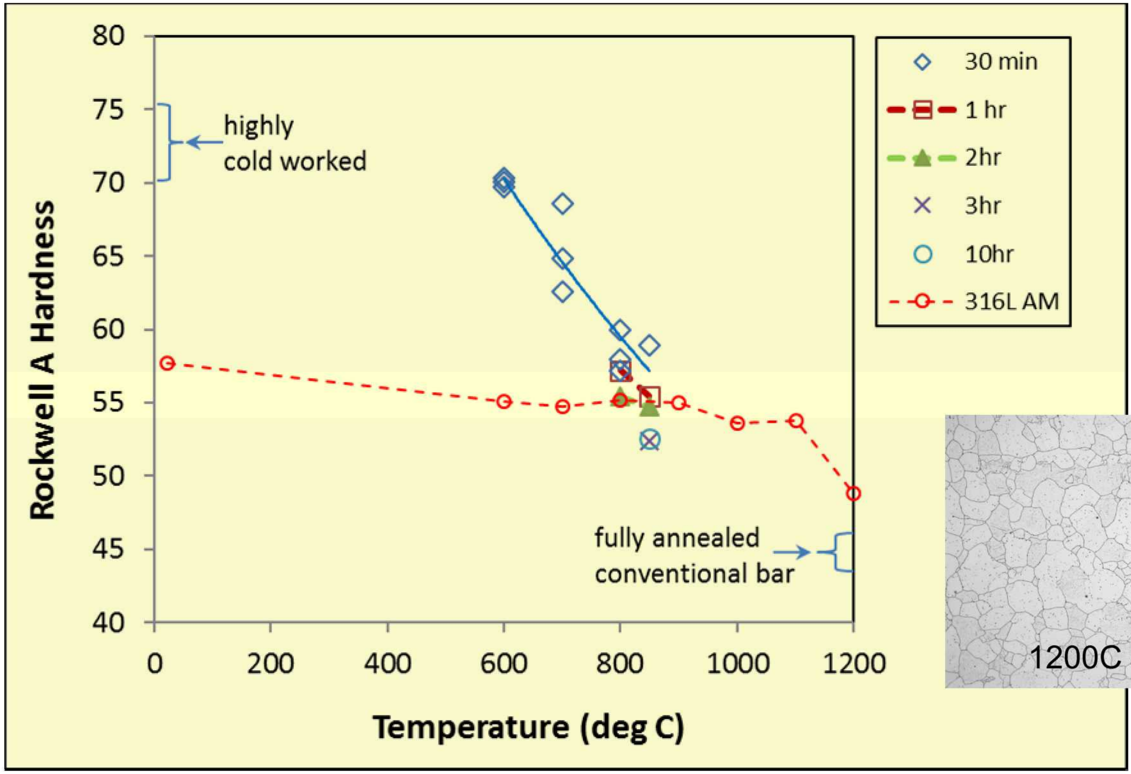
Microhardness mapping

- Approximately 150 Vickers indents (300g load) performed across the entire face of AM cubes at 0.5 mm spacing
- No trend with z build height
- Vickers microhardness measurements (avg. of ~150 indents) confirm previous Rockwell B hardness results (~3 indents per condition)

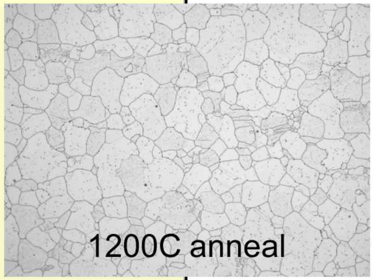




Comparison to Annealing of Wrought Cold-Worked Austenitic Stainless Steel



Overall, the AM 316L hardness decreased only moderately up to 1200°C where a larger drop was exhibited due to recrystallization and growth of equiaxed grains.



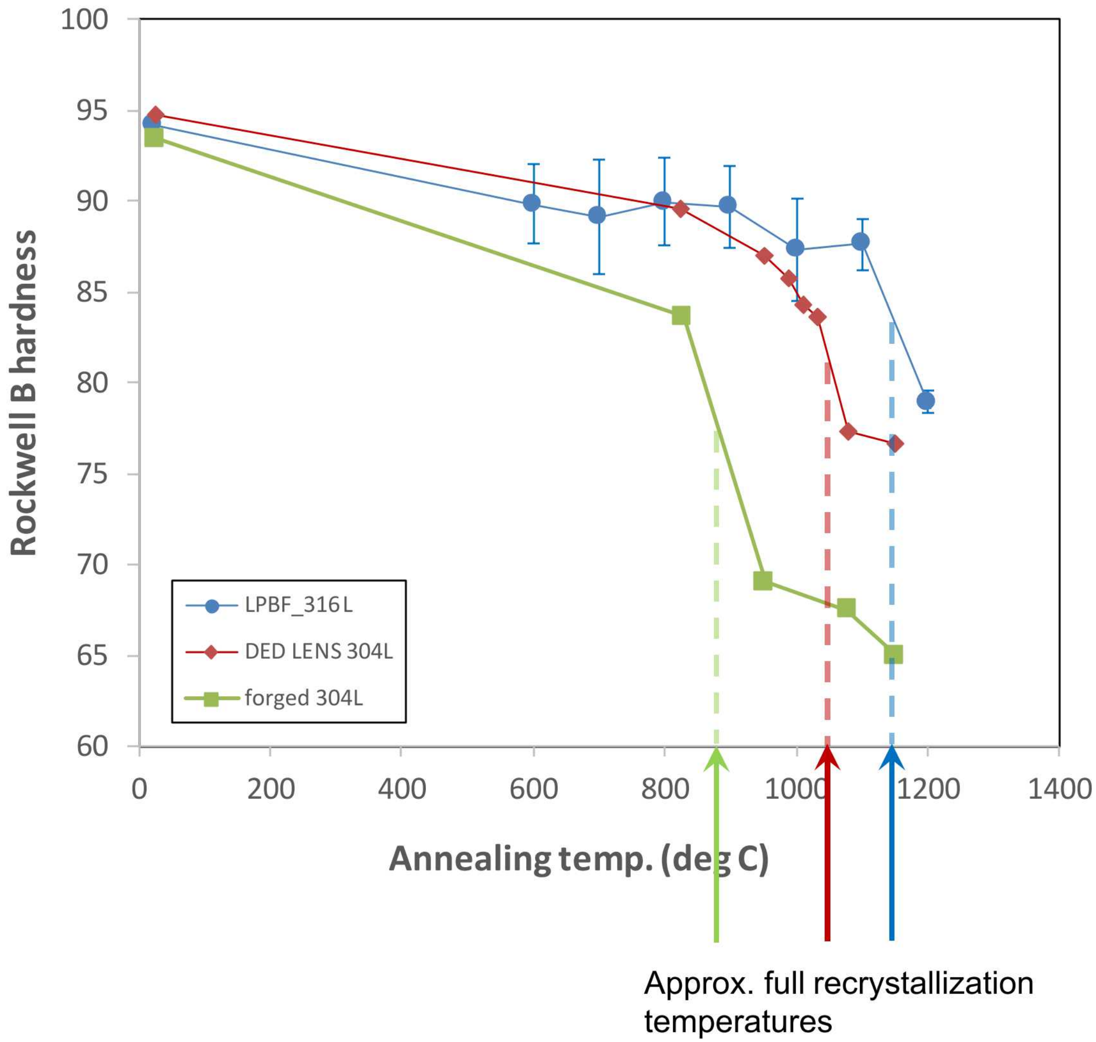
AM 316L and cold-worked 304L properties converge after annealing at about 800C.

Although the as-deposited hardness is somewhat higher than annealed 316L due to fine-scale microstructure, it does not contain high amounts of deformation (dislocation density not very high). Therefore, it does not exhibit a dramatic driving force for annealing (recrx and grain growth). In comparison, highly cold-worked (flowformed) 304L shows a significant annealing response, as low as 700°C, due to a high amount of stored internal energy.

Compare to other AM and wrought material

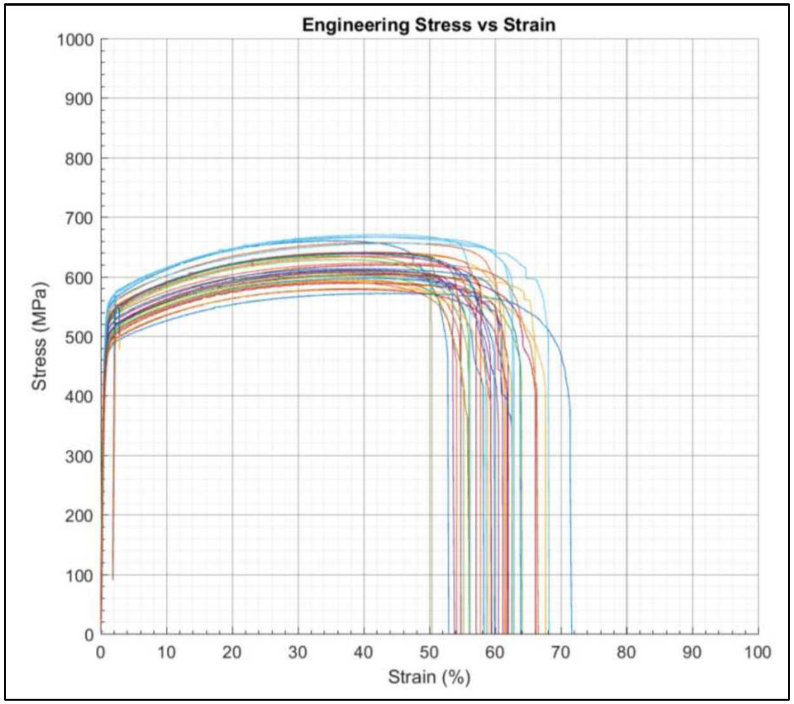
- Smith et al. (JOM, Vol. 70, No 3, 2018) measured hardness on direct energy deposited (LENS) 304L and wrought (forged) 304L.
  Note: converted from Vickers microhardness
- AM material shows more "resistance to annealing" relative to wrought material, *for equivalent starting hardness values*. Smith et al. attributed the behavior to microsegregation (solidification substructure) inhibiting recrystallization in AM materials.

  Powder-bed AM is possibly even more resistant at high temperatures – higher hardness due to finer overall and recrystallized grain size

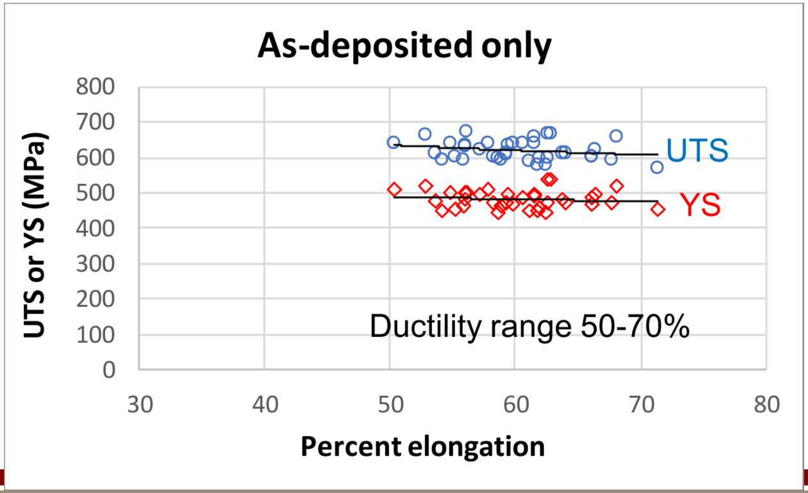


Tensile Test Results: As-Deposited

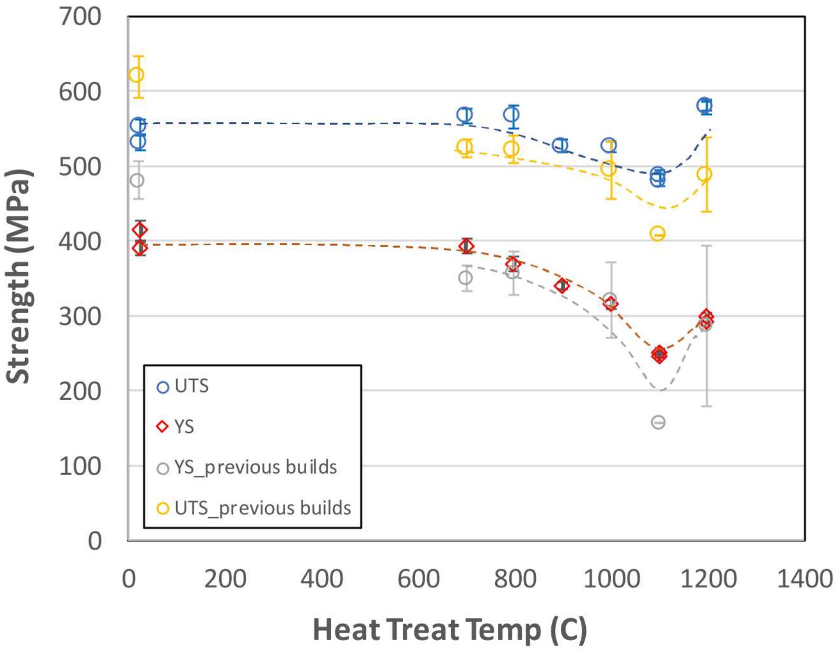
Stress-strain curves from 50 tests

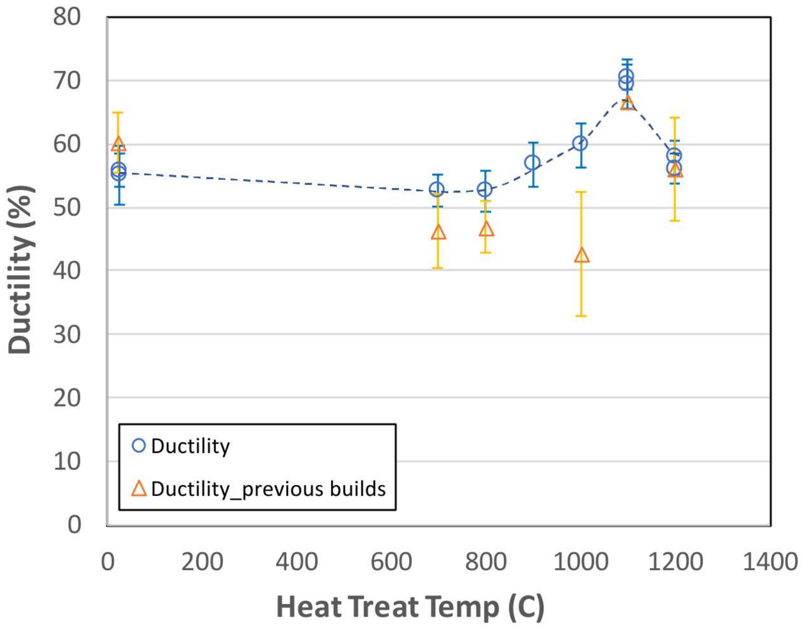


Ductility in the range 50-70% is quite good and highlights the low defect concentration in these AM builds of 316L. Scatter in strength appears nominal for stainless steel.



Effects of Annealing on Tensile Properties





- Error bars indicate standard deviation of >25 tensile tests (some cases 50), with the exception of 1100C "previous build".

- General decrease in strength up to 1100°C (large columnar grains). Slight increase in strength associated with full recrystallization at 1200°C. This strength increase does not correspond to hardness results from "previous build". Hardness not yet measured on latest build specimens.

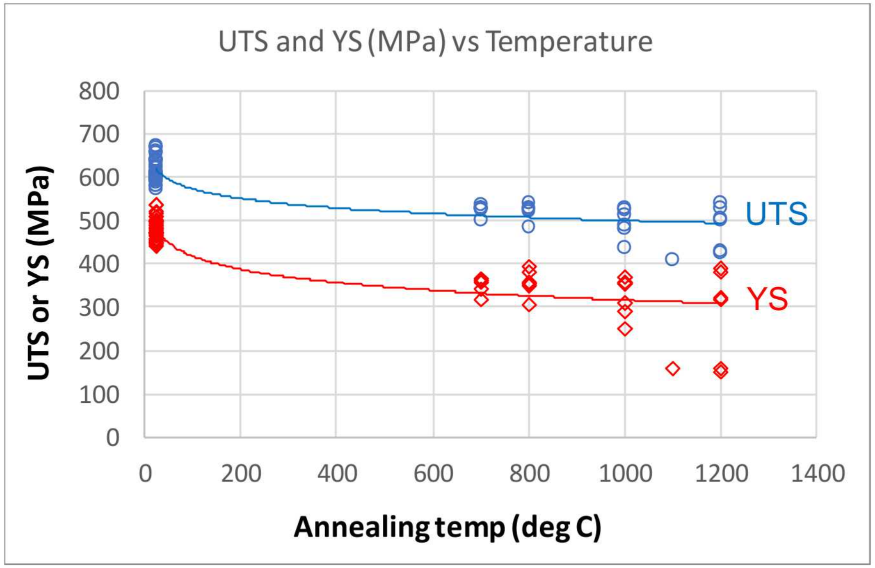
- Ductility peaks with annealing at 1100°C, corresponds to lowest strength condition.

Summary

- With cross-hatch build sequence, the annealed microstructure shows remnants of the scan pattern, i.e. "square shaped" grains. Mild (100) texture was observed in the build direction after annealing, with some change in texture as the sample is built up from the base plate.
- Hardness and strength decrease moderately for anneal temperatures up to 1100°C. Strength decreases to 1100C also, followed by slight increase with 1200C anneal. Need to measure hardness of 1200C tensile specimens.
- AM material is resistant to annealing, relative to wrought material of the same alloy at the same annealing temperature. For this powder-bed AM316L, temperatures >1100°C are necessary for full recrystallization and grain growth. This could be due to persistent solidification substructure (microsegregation) inhibiting recrystallization, as well as generally fine grain size throughout annealing.
- Tensile elongation (ductility) ranged from 50-70% in as-deposited material. Recent tensile tests show peak in ductility corresponding to lowest strength condition with 1100C anneal.

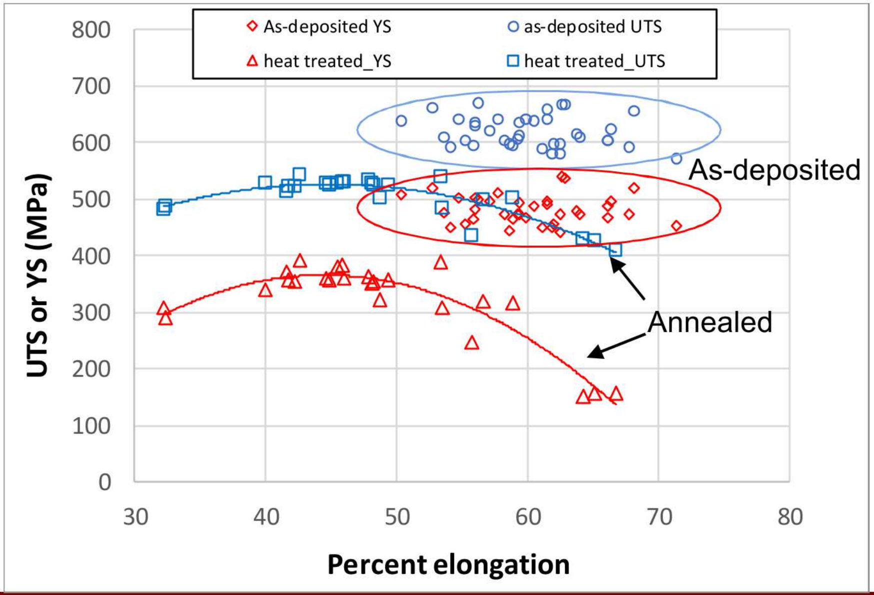
- Back up slides

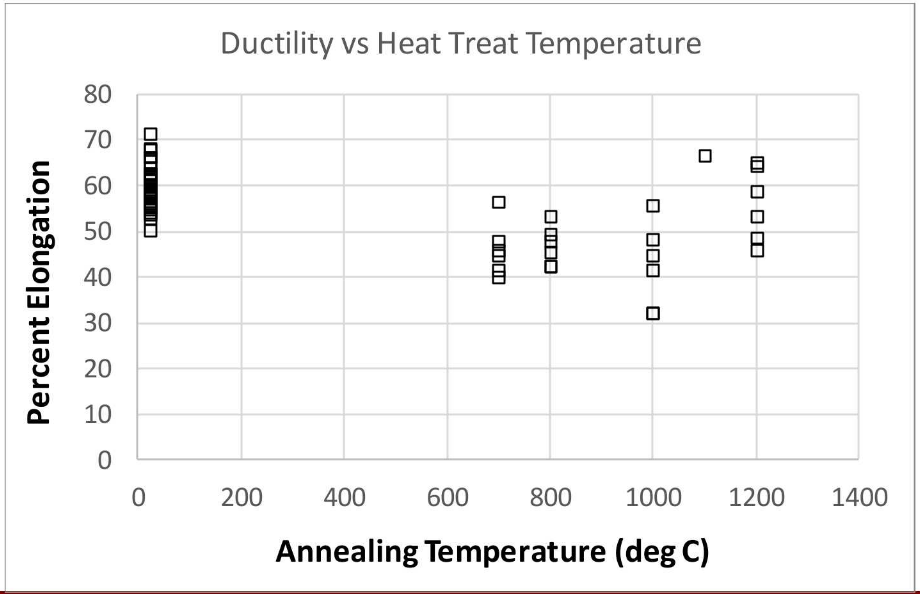
Original Tensile Test Results (1st build)

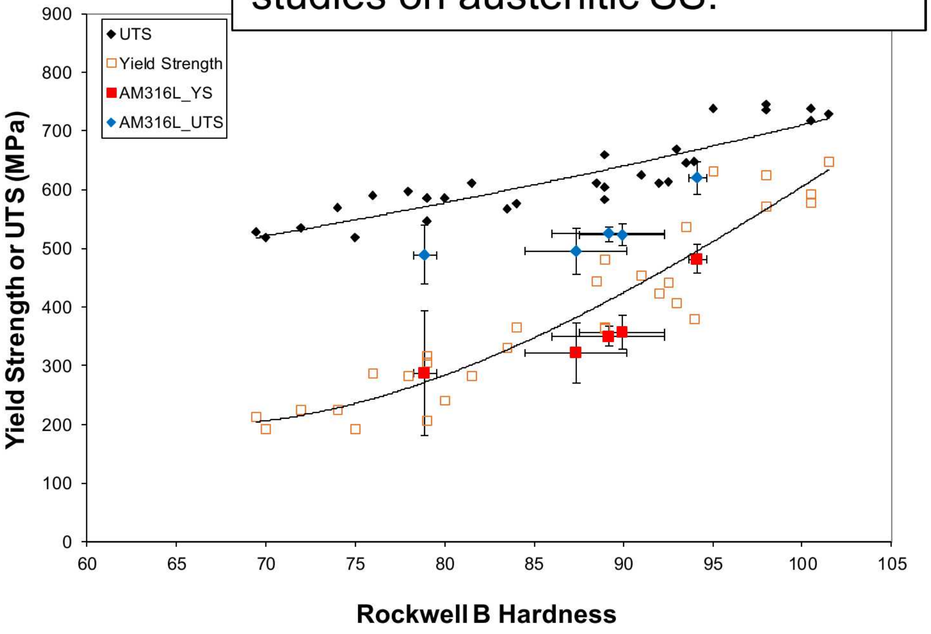


Strength decreases with annealing temperature, as expected. Consistent with hardness results. More scatter in strength properties with higher annealing temperatures.

The ductility does not necessarily increase with annealing temperatures. More work is needed to understand these effects.







UTS and YS correlate well to the hardness results and previous studies on austenitic SS.

Red and blue symbols = AM 316L as deposited, and annealing at 700, 800, 1000, 1200C

Trace Chemical analysis of AM 316Lpowder

ANALYSIS	ppmw	ANALYSIS	ppmw	ANALYSIS	ppmw
~~H~~		Zn	4.9	Pr	<0.1
Li	<0.1	Ga	28	Nd	<0.1
Be	<0.1	Ge	10	Sm	<0.05
B	4.9	As	75	Eu	<0.05
~~C~~		Se	<0.5	Gd	<0.05
~~N~~		Br	<0.5	Tb	<0.05
~~O~~		Rb	<0.1	Dy	<0.05
F	<0.5	Sr	5.8	Ho	<0.05
Na	0.75	Y	<0.3	Er	<0.05
Mg	1.8	Zr	115	Tm	<0.05
Al	65	Nb	155	Yb	<0.05
Si	5200	Mo	Major	Lu	<0.05
P	225	Ru	<0.1	Hf	1.2
S	120	Rh	<0.1	~~Ta~~	
Cl	0.14	Pd	<0.1	W	265
K	0.19	Ag	≤1	Re	2.6
Ca	0.65	Cd	<0.3	Os	<0.1
Sc	<0.1	In	0.92	Ir	<0.1
Ti	0.59	Sn	70	Pt	<0.1
V	390	Sb	6.3	Au	<0.5
Cr	Major	Te	<0.3	Hg	<0.5
Mn	Major	I	<0.1	Tl	<0.05
Fe	Major	Cs	<0.3	Pb	0.22
Co	1010	Ba	<0.5	Bi	<0.03
Ni	Major	La	<0.5	Th	<0.03
Cu	2150	Ce	<0.5	U	<0.03

Trace Chemical analysis of AM 316L build material

ANALYSIS	ppmw	ANALYSIS	ppmw	ANALYSIS	ppmw
~~H~~		Zn	0.82	Pr	<0.05
Li	<0.1	Ga	27	Nd	<0.05
Be	<0.1	Ge	17	Sm	<0.05
B	1.9	As	39	Eu	<0.05
~~C~~		Se	<0.5	Gd	<0.05
~~N~~		Br	<0.5	Tb	<0.05
~~O~~		Rb	<0.1	Dy	<0.05
F	<0.1	Sr	<5	Ho	<0.05
Na	0.20	Y	<1	Er	<0.05
Mg	0.42	Zr	55	Tm	<0.05
Al	100	Nb	140	Yb	<0.05
Si	3600	Mo	Major	Lu	<0.05
P	160	Ru	<1	Hf	<0.1
S	79	Rh	<0.1	Ta	0.44
Cl	0.019	Pd	<0.5	W	190
K	0.11	Ag	<0.5	Re	2.6
Ca	0.42	Cd	<0.2	Os	<0.1
Sc	<0.1	In	<0.1	Ir	<0.1
Ti	1.4	Sn	85	Pt	<0.1
V	690	Sb	6.8	Au	<0.5
Cr	Major	Te	<0.1	Hg	<0.5
Mn	Major	I	<0.1	Tl	<0.05
Fe	Major	Cs	<0.1	Pb	0.16
Co	870	Ba	<0.3	Bi	0.015
Ni	Major	La	<0.1	Th	<0.01
Cu	2300	Ce	0.11	U	<0.01

AM 316Lpowder

Method:	Combustion – Infrared Absorption / Inert Gas Fusion				
Sample ID	C (wt. %)	S (wt. %)	N (wt. %)	O (wt. %)	H (wt. %)
Sample 1: Top of Container	0.0508 ± 0.0007	0.0072 ± 0.0002	0.112 ± 0.001	0.0848 ± 0.0025	0.0007 ± 0.0001
Sample 2: Middle of container	0.0512 ± 0.0003	0.0073 ± 0.0002	0.111 ± 0.001	0.0851 ± 0.0009	0.0007 ± 0.0001
Sample 3: Bottom of container	0.0517 ± 0.0007	0.0069 ± 0.0005	0.112 ± 0.001	0.0835 ± 0.0017	0.0008 ± 0.0001

AM 316L cube builds

METHOD: Inert Gas Fusion					
Sample ID	C(wt%)	S(wt%)	N(wt%)	O(wt%)	H(wt%)
Cubes 2-3	0.0140±0.0002	0.0116±0.0001	0.0924±0.0007	0.0608±0.0002	0.0002±0.0001