Sandia National Laboratories

Evaluating Corrosion Properties and Controlling Factors in Emergent Materials

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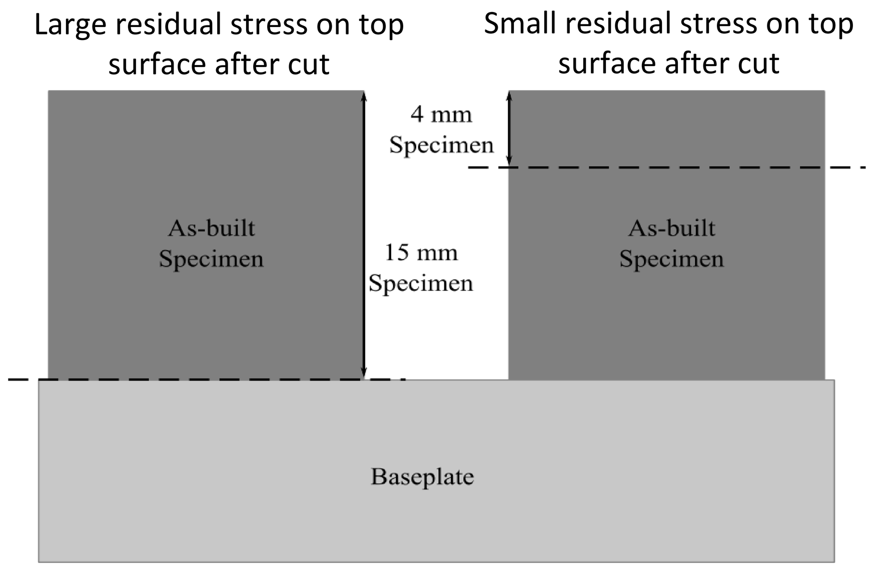
Objectives

Evaluate corrosion susceptibility in accelerated ASTM chloride environments of Laser Beam-Powder Bed Fusion (LB-PBF) 316L stainless steel & cold-sprayed nickel-based alloys on 304 stainless steel. **What factors influence the corrosion behavior?**

Laser Beam-Powder Bed Fusion 316L

Background

Several studies have observed the beneficial effects of heat treatments on alleviating tensile residual stresses of LB-PBF 316L and their subsequent reduction of stress corrosion cracking susceptibility.[1,2] Heat treatments affect both the residual stress and the microstructure. Cutting LB-PBF samples at different heights from the build plate results in different residual stresses without modifying the microstructure.[3]



Materials

316L LB-PBF (3D Systems ProX DMP 200)

As-built cut to different heights:
- 4, 6, 8, 10, 15 mm

Heat-treated:
- 600, 800, 1200 °C for 1 hour

Methods

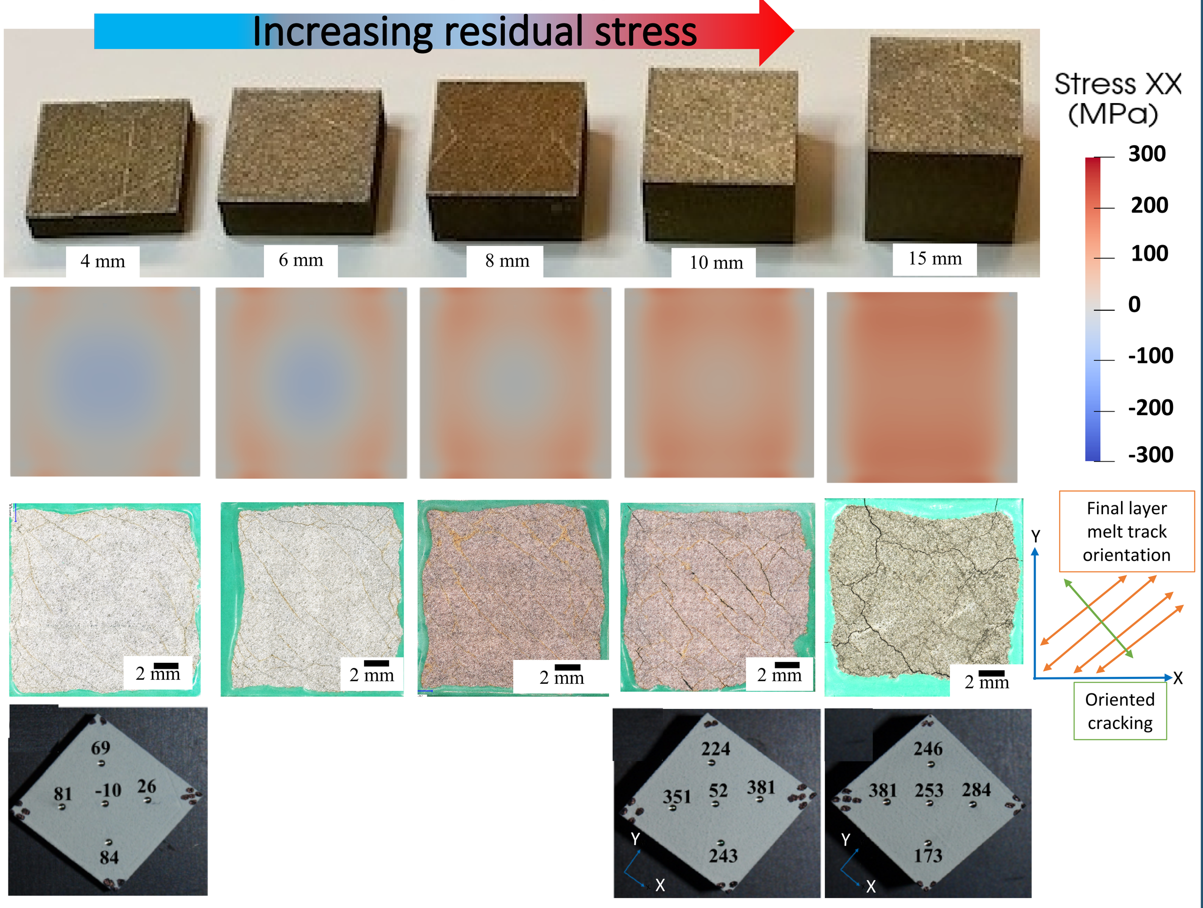
Boiling $MgCl_2$ Test
- 155 °C
- Saturated $MgCl_2$
- Test until cracked or ~350 hours

Hole-Drilling Stress Measurement
- 0.8 mm diameter drill
- 0.6 mm depth, 25 µm increments

Residual Stress Simulations
- Finite element analysis
- Sierra mechanics module

Microstructure & Dislocations
- Cross-sections
- Polished & etched
- Electron backscatter diffraction
- Geometrically necessary dislocation density

As-Built

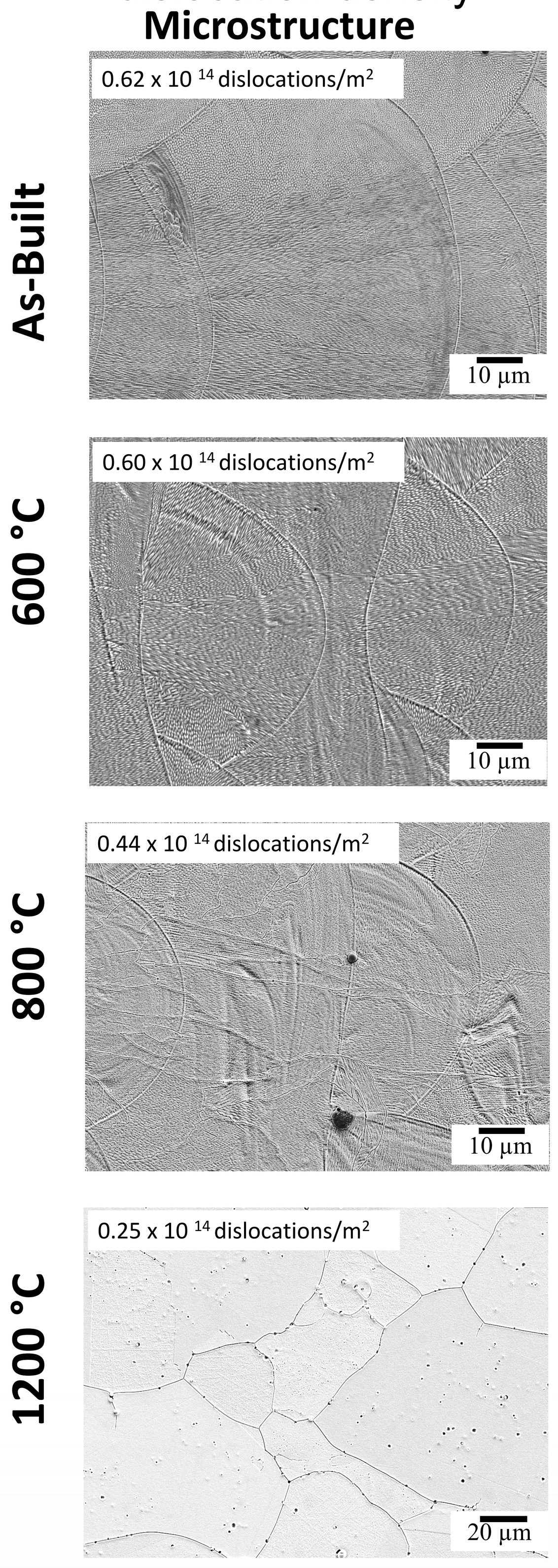


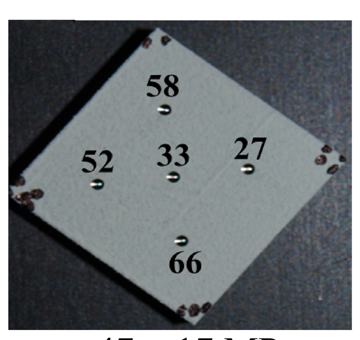
Results

- Height & heat treatment affect residual stress
- All as-built samples cracked in 24 hours
- < 10 mm as-built show oriented cracking
- Oriented cracking: perpendicular to melt tracks
- More cracking in higher tensile residual stress areas (based on modeling results)
- 600 °C, 4 mm sample still showed cracking
- 800 °C eliminated SCC even in 15 mm sample
- 800 °C shows recovery of some dislocations, formation of new grain boundaries
- 1200 °C shows recrystallization

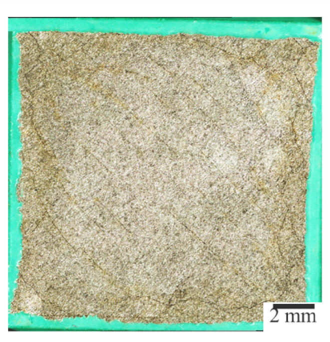
Heat-Treated

Microstructure



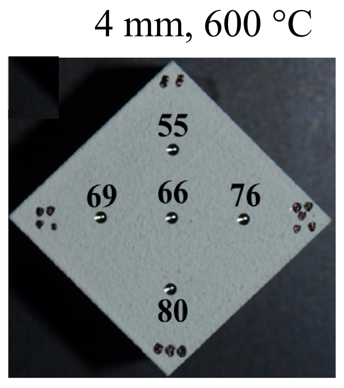


47 ± 17 MPa
4 mm, 600 °C

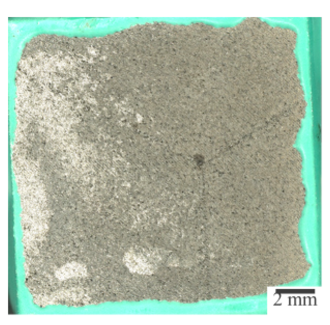


4 mm, 600 °C

Still shows oriented cracking. Microstructure (sub-grain cellular structure and melt pool boundaries) maintained. No real change in dislocation density.



69 ± 10 MPa
15 mm, 800 °C



15 mm, 800 °C

No observed cracking (visible lines are artifacts of ProX quilt-like print pattern). Microstructure still present, but evidence of dislocation recovery.

Conclusions

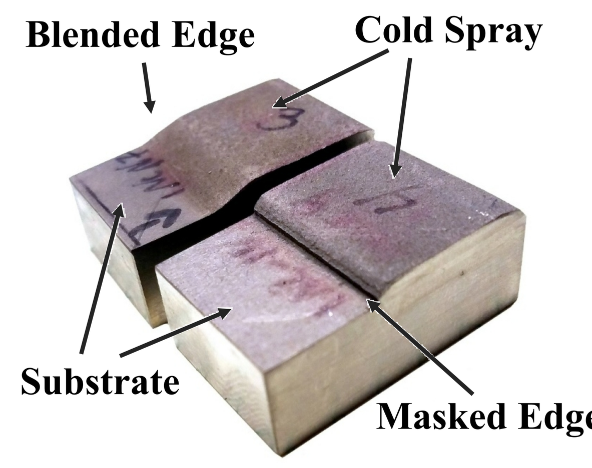
- Residual stresses from melt tracks can contribute to SCC
- Don't need to eliminate LB-PBF microstructure to eliminate SCC
- SCC susceptibility not exclusively related to residual stress magnitude
- SCC susceptibility influenced by dislocation density

Karasz, E. et al. Measuring the residual stress and stress-corrosion cracking susceptibility of additively manufactured 316L by ASTM G36-94. Corrosion, (2021).

Cold Sprayed, Nickel-Based Alloy Coatings

Background

Cold spray is a technique where metal particles are accelerated into a substrate material, propelled by a stream of inert gas and adhere to the substrate through a kinetic deformation process (unlike LB-PBF which relies on melting). Cold spray also induces compressive residual stresses[4] which are often considered beneficial for preventing stress corrosion cracking[5]. One application space for cold spray is as a corrosion prevention/mitigation coating. If it is applied as a patch, a junction between the cold spray and substrate is left exposed and could be at higher risk for corrosion.



Materials

Commercially pure nickel & two nickel-based alloys were cold sprayed onto a substrate material of 304 Stainless Steel. Nitrogen or Helium gas was used in spraying. Samples were sprayed and the porosity and surface roughness measured at Pacific Northwest National Laboratory.

Sample	Porosity (%)	Surface Roughness S_a (µm)
SC-N	5.51 ± 0.44	16.7 ± 0.5
Inc-He	1.21 ± 0.20	15.7 ± 0.5
Inc-N	5.79 ± 0.18	17.2 ± 0.6
Ni-N	3.78 ± 0.59	18.5 ± 0.6

	Inconel 625 (Inc)	Super C (SC)	Nickel (Ni)
C	0.02%	0.02%	≤0.01%
Co	0.09%	0.2%	-
Cr	21.47%	23.2%	-
Ni	Balance	Balance	>99.9%
Mo	9.06%	17.7%	-
Mn	0.04%	0.7%	-
P	0.0%	0.002%	-
S	0.0%	0.004%	≤0.001
Si	0.07%	0.5%	-
Fe	4.62%	0.6%	≤0.14%
Al	0.04%	-	-
B	0.001%	0.003%	-
Nb	3.65%	-	-
O	0.024%	-	≤0.4%
V	-	0.30%	-
W	-	0.26%	-

Methods

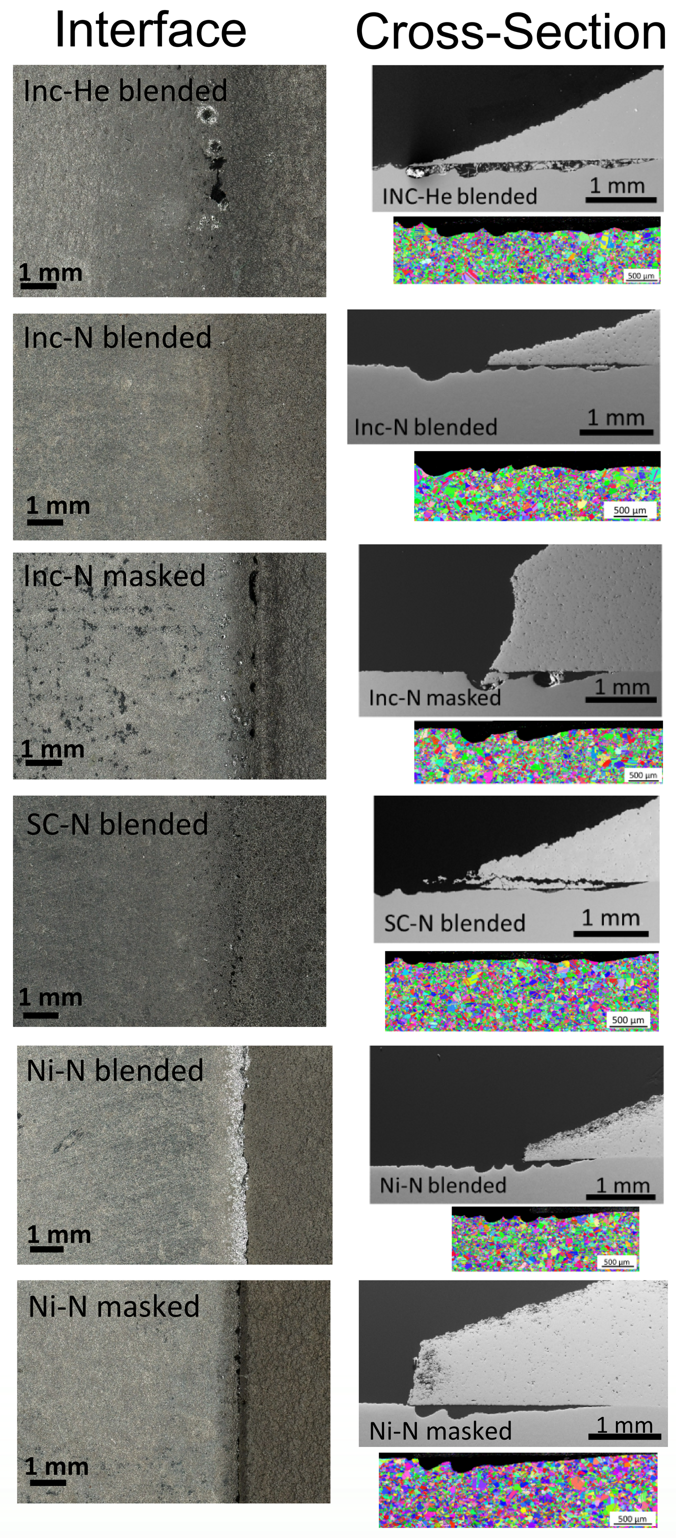
Ferric Chloride Test: ASTM G48 Method A
- 6% by mass ferric chloride solution
- 72 hours, 22 °C
- sides & bottom coated in epoxy

Electrochemical Scans
- only tested cold spray region
  - as sprayed and polished conditions
- 0.6 M NaCl, 0.167 mV/s

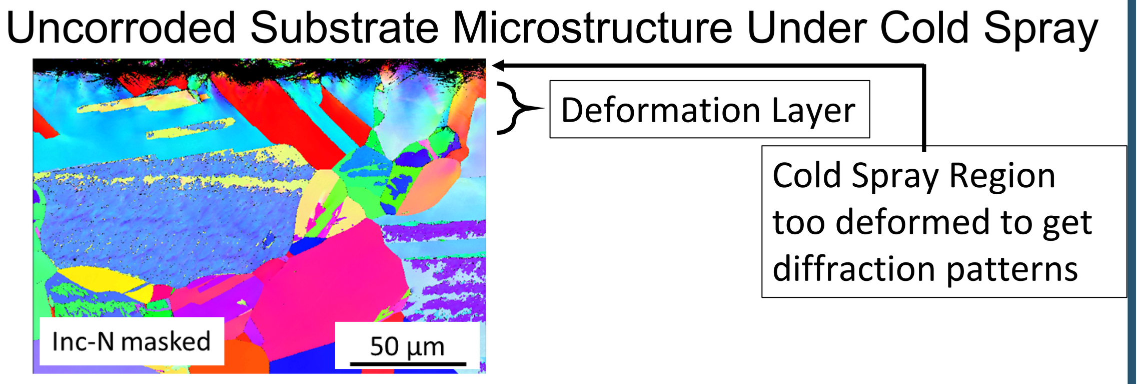
Results

- Top-down, nickel samples show more damage in cold spray and at the interface
- Amount of damage seen top-down doesn't correlate with the amount of damage seen in cross-section
- In cross-section, all samples have damage in substrate, some have damage in cold-spray (porosity dependent?)
- Reducing roughness minimizes metastable pitting
- He processed has less metastable pitting (porosity related?)

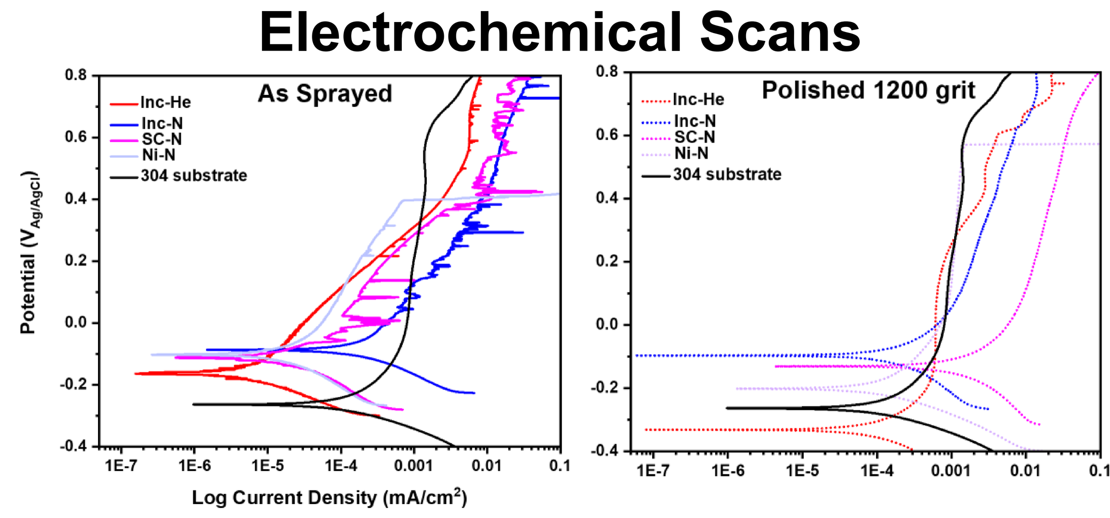
Ferric Chloride Test



Uncorroded Substrate Microstructure Under Cold Spray



Electrochemical Scans



Conclusions

- Gas selection impacts porosity, location of damage in cross-section, and metastable pitting
- Interface changes how crevice corrosion presents (top-down)
- Material selection influences breakdown potential, corrosion morphology
- Top-down damage not indicative of damage between substrate and cold spray (cross-section)
- Electron backscatter diffraction shows deformation layer in substrate is thinner than corrosion damage layer

References

1. E. De Bruycker, M.L.M. Sistiaga, F. Thielemans, K. Vanmeensel, Materials Sciences and Applications 08, 03 (2017): p. 223- 233.
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4. V. Luzin, S. Kuroda, S. Yin, A.S.M. Ang, Journal of Thermal Spray Technology, 29 (2020): p. 1211-1217.
5. J. Toribio, Journal of Materials Engineering and Performance, 7 (1998): p. 173-182.

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Cold spray samples were sprayed by Ken Ross at Pacific Northwest National Laboratory.

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