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Timothy Montoya

mvr9uh@virginia.edu

University of Virginia

Duane Macatangay

University of Virginia

Robert G. Kelly

University of Virginia

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Impact of Elemental Segregation on Active and Transpassive Corrosion Phenomenology of Additively Manufactured 316L

Timothy Montoya¹, Duane Macatangay¹, Robert G. Kelly¹

University of Virginia, Materials Science and Engineering, Charlottesville, Virginia

Abstract

The active and transpassive dissolution behaviors of additively manufactured (AM) laser powder bed fusion (LPBF) and direct energy deposition (DED) 316L stainless steels were evaluated using ASTM standard methods as well as electrochemical, and microscopy characterization techniques. Conventional wrought 316L and wrought 317L was used as baselines for chemical, microstructure, and electrochemical behavior. It was discovered that both LPBF and DED induce non-equilibrium solidification conditions that lead to elemental segregation of Cr, Ni, and Mo. The segregation occurs within the cellular dendritic walls in LPBF and within the ferrite phase in DED whereas wrought materials exhibit a homogenous microstructure and elemental distribution. Under active dissolution in the presence of acidic (H_2SO_4) and depassivating species (KSCN), cellular dendritic interiors and melt pool boundaries undergo selective attack in LPBF while austenite dissolves faster than ferrite in DED. Conversely, when transpassive dissolution occurs, cellular dendritic walls are attack in LPBF and ferrite is attacked in DED. When LPBF processing parameters are optimized for density, the corrosion rate (ASTM A262 Practice B) was the lowest in comparison to wrought 316L and 317L. Samples with the highest corrosion rate correlated to the presence of lack of fusion porosity (1.8% porosity) or large grain structure (DED). From these findings, it is evident that elemental segregation drives the dissolution in AM materials but the presence of dislocations and other microstructure are not the reason for dissolution in LPBF or DED materials.

1. Introduction

The use of additive manufacturing (AM) has become increasingly popular over the last decade due to the ability to construct complex geometries while controlling microstructure, mechanical, and corrosion properties with techniques such as laser powder bed fusion (LPBF) and direct energy deposition (DED) ¹⁻⁵. LPBF and DED are both rapid solidification techniques that use a high-energy laser or electron beam to melt powder (LPBF/DED) or wire (DED) to manufacture material ^{1,5}. However, LPBF solidification rates are orders of magnitude faster (10^3 – 10^8 K/s⁴) in comparison to DED (10^2 – 10^5 K/s^{6,7}) resulting in the formation of complex, non-equilibrium microstructure which is different than its cast or wrought counterparts. Because of faster solidification rates in LPBF, the formation of a finer grain structure and cellular dendritic microstructure occurs ^(3,4,8,9) while the formation of larger grains and dendritic microstructure occurs in DED due to slower cooling rates ⁴. Many alloys have become good candidates for LPBF and DED with alloys such as austenitic 316L stainless steel being one of the first and most-studied alloys for AM ^{4,8,9}.

For an alloy like 316L, numerous studies have focused on understanding how processing parameters impact the ability to achieve high density parts with similar or improved mechanical properties versus traditional material¹⁰⁻¹². However, the connection of the influence of processing parameters on the corrosion behavior of AM 316L has not been fully explored¹³. Most work on AM corrosion of 316L has been focused on pitting corrosion and usually focus on the impact of process-induced features such as lack-of-fusion porosity and residual stress^{1,14,15}. These microstructural features often involve compositional differences which may control the electrochemical behavior of these structures as well as how corrosion phenomenology varies with electrochemical conditions. Previous work on LPBF 316L has shown how preferential dissolution takes place along melt pool boundaries and the interior of the cellular dendritic structures in ammonium persulfate, hydrochloric acid, and sulfuric acid environments¹⁵⁻²⁴. Recent work has explored the use of ASTM standard corrosion tests to evaluate the sensitization of AM 316L with the use of ASTM A262 and ASTM G108^{13,21,25-28}.

Stainless steels like 316L are known for their excellent corrosion resistance due elements such as Cr and Mo²⁹⁻⁴¹. Cr has been shown to create a protective Cr₂O₃ passive oxide film (for Cr content greater than ~13 wt%)^{29,30}, while Mo has been associated with improving pitting potentials, reducing current densities, and improving the stability of Cr₂O₃ passive oxide film^{34,37-39}. At lower anodic overpotentials in acidic, non-chloride containing solutions the surfaces of 316L will become activated causing anodic dissolution of the surface due to the absence of a protective passive oxide film. At modest anodic overpotentials, the passive film forms, causing a significant decrease in dissolution and protection of the surface. When higher anodic overpotentials are applied, transpassive dissolution occurs at the passive oxide film interface causes Cr³⁺ to oxidize to Cr⁶⁺⁴²⁻⁴⁴ and Mo⁴⁺ to oxidize to Mo⁶⁺^{42,45-47}. Given the importance of Cr and Mo in 316L, the non-equilibrium solidification that occurs in AM drives the formation of complex microstructure and heterogenous elemental distribution in LPBF⁴⁸⁻⁶⁰ and DED^{6,7} for critical alloying elements such as Cr and Mo which impact the localized corrosion behavior.

The purpose of this work is to understand how AM techniques such as LPBF and DED impact the localized corrosion behavior of 316L under acidic and depassivating environments in accordance to ASTM A262 Practice B (ASTM A262-B)⁶¹ and ASTM G108⁶². Whether it is through LPBF or DED, AM solidification processes result in complex microstructural features which can become critical to determine how these features dictate selective corrosion behavior, particularly in the test environments that are used as defaults for testing SS. Further, with the use of various characterization techniques, the goal is to better understand how to employ ASTM standards to evaluate complex microstructures produced by AM processes especially when comparing to its conventional cast and wrought counterparts. Proposed hypotheses involving the role of composition of critical alloying elements (Cr, Mo, and Ni) and dislocation structures are further tested by using high-resolution characterization methods and validated through comparison of electrochemical behavior between wrought steels that have varying, but spatially uniform, compositions and different degrees of dislocation density to simulate AM microstructural features. By performing these tests, the main goal of this study is to propose holistic mechanisms of selective corrosion in AM 316L while comparing behavior to the conventionally wrought counterpart with the use of ASTM standardized techniques.

2. Experimental

2.1 Material Information and Preparation for Electrochemical Testing

In this work, three manufacturing methods were studied which include wrought, LPBF, and DED. Two wrought austenitic SSs, wrought AISI 316L and 317L, from North American Stainless (Pendergrass, Georgia) were used in this study. Wrought 316L was used to establish a baseline corrosion behavior in comparison to the LPBF 316L stainless steel samples. However, given the higher composition of critical alloying elements in the manufactured LPBF 316L samples, wrought 317L was also studied. The AISI 316L stainless steel powder used was manufactured by EOS (EOS GmbH – Electro Optical Systems, Krailing, Germany, EOS art. no. 9011-0032) and had a spherical particle size distribution of 10-63 microns (μm). Samples were printed at Johns Hopkins University Applied Physics Labs (JHU/APL), which utilizes a 1064 nm, 200 W continuous Yb fiber laser with a fixed 80 μm spot size and an argon-purged atmosphere. Samples were printed with dimensions of 20 mm x 20 mm x 60 mm. The 20 mm x 20 mm base was oriented perpendicular to the build direction. The default vendor (EOS) parameters are given by the parameters for LPBF A8 SS. Further information can be found in recent work¹³ detailing the processing parameter selection process used for the LPBF material in this current study.

Two sets of DED samples were used in this study. One set of DED samples was printed at the University of Virginia (UVA) and the other at Lincoln Electric (LE). UVA DED 316L was fabricated using a commercial M450 Meltio printer using 316L feedstock wire with a 0.9 mm diameter and high purity argon build atmosphere. LE produced DED samples using a Blue Max® 316LSi wire with a 1.1mm diameter and manufactured with a proprietary DED system.

All build parameters used for both the LPBF and DED samples is shown in Table 1 and Table 2 along with their respective compositions in Table 3. The composition of wrought, LPBF, and UVA DED samples were analyzed by IMR Test Labs (Lansing, New York) via inductively coupled plasma atomic emission spectrometry (ICP-AES) to measure the bulk elements whereas coupled and combustion-infrared absorbance to measure C, S, and O. LE DED samples were characterized using X-ray fluorescence spectroscopy (XRF) and Laboratory Equipment Company (LECO) analysis was done at LE. All samples were prepared sectioning from their as-received state and ground to a 1200 grit silicon carbide (SiC) finish followed by a deionized (DI) water rinse.

Table 1. LPBF AM 316L processing parameters		
LPBF Parameters	LPBF A3	LPBF A8
Power (W)	95	195
Scan Speed (mm/s)	1510	1083

Hatch Distance (mm)	0.06	0.09
Layer thickness (mm)	0.02	0.02
Volumetric Energy Density (J/mm ³)	52	100
Gas Environment	Argon	Argon

Table 2. DED AM 316L processing parameters			
UVA DED Parameters	UVA DED 316L	Lincoln Electric DED Parameters	Lincoln Electric DED 316L
Power (W)	800	Heat Input (kJ/in)	15
Travel Speed (mm/s)	7	Deposition Rate (lb./h)	11
Deposition Rate (mm/s)	16.31	Bead Width (mm)	10
-	-	Layer Height (mm)	2.92
Gas Environment	Argon	Gas Environment	Argon

Table 3. Composition of wrought, LPBF, and UVA DED material determined by (1) ICP-AES, (2) combustion-infrared absorbance, or (3) fusion. Composition of LE DED material determined by XRF (4). All values are in reported in wt. %.

Element	Wrought 316L	Wrought 317L	LPBF Process Powder	LPBF A3 As-Built	LPBF A8 As-Built	UVA DED Process Wire	Lincoln Electric DED Process Wire ⁴
Fe	Balance	Balance	Balance	62.31	64.42	Balance	Balance
Cr ¹	16.7	18.26	18.62	17.95	17.93	16.8	18.3
Ni ¹	10.2	11.5	14.04	14.84	14.82	11.0	11.4
Cu ¹	<0.1	<0.1	< 0.1	<0.1	<0.1	<0.1	<0.1
Mn ¹	1.99	1.85	1.65	1.50	1.35	1.95	1.70
Si ¹	0.86	0.52	0.37	0.30	0.30	0.32	0.80
Mo ¹	1.99	3.01	2.77	2.95	3.03	2.30	2.30

C ²	0.006	0.003	0.007	0.001	0.01	0.002	0.02
S ²	0.006	0.008	0.005	0.004	0.004	0.004	0.01
P ¹	<0.010	<0.010	<0.010	<0.005	<0.005	<0.010	0.02
N ³	0.04	0.08	0.07	-	-	-	0.06

2.2 Microstructure Characterization

Several characterization techniques were performed to evaluate the microstructure and phases in all samples pre- and post-test. Pre- and post-corrosion micrographs were generated using a FEI Quanta 650 scanning electron microscope (SEM) at 10kV accelerating voltage with secondary and backscatter detection modes. Energy dispersive spectroscopy (EDS) was used to assess the LPBF and DED material to identify elemental and phase distribution. A Helios Dual-Beam focused ion beam (FIB) and SEM was used to section and extract transmission electron microscopy (TEM) lamella for examination of the LPBF microstructure. A Themis 60-300 kV TEM was performed in combination with STEM-EDS construct compositional maps of fine LPBF microstructure. High angle annular dark field (HAADF) imaging was utilized to investigate the dislocation structures of LPBF material. Electron backscatter diffraction (EBSD) was performed using a Thermo Scientific Scios DualBeam™ to construct grain and phase maps of LPBF and DED material.

2.3 Electrochemical testing in Sulfuric Acid Environments

Open circuit potential measurements (OCP) and linear potentiodynamic polarization (LPP) tests were carried out to understand the corrosion behavior of the AM material in comparison the wrought material in acidic environments at room temperature. All tests and data were collected using a Biologic SP-200 Potentiostat. A typical three-electrode cell configuration consisting of the sample as the working electrode, platinum-coated niobium mesh as the counter electrode, and an Ag/AgCl reference electrode was utilized. Three electrochemical tests were employed to understand the behavior of wrought, LPBF, and UVA DED in acidic environments which include: (1) monitoring the OCP over 24-hours (hrs), (2) determining the active behavior in 1M H₂SO₄ and 0.01M KSCN, and (3) determining the transpassive behavior in 1M H₂SO₄. For some tests, potassium thiocyanate was added as it induces active corrosion on the surface of Fe-Ni-Cr alloys by destabilizing the air-formed film¹³. The OCP was measured for 30 minutes followed by an LPP scan with a scanning rate of 1.667 mV/s, over a voltage range of -40 mV below OCP to 0.345V_{vs Ag/AgCl}. The transpassive behaviors of LPBF 316L, DED 316L, wrought 316L, and wrought 317L in 1 M H₂SO₄ were investigated similarly to the active behavior by measuring the OCP for 30 minutes followed by a LPP -40 mV below OCP to 1.3V_{vs Ag/AgCl}. All testing surfaces were oriented perpendicular to the build direction (BD), ground to a 1200 grit SiC finish, and tested a minimum of three times. The resultant microstructure following electrochemical tests was examined using SEM and TEM.

2.4 Mass Loss Testing in Boiling Acid Solutions

To measure the mass loss, the use of a boiling acid environment was performed. The experimental setup and chemical environment followed ASTM A262-B. Sample sizes for all material varied but

all met the requirement of having a total surface area of at least 5 cm^2 to 20 cm^2 . All faces were ground to a 1200 grit SiC finish similar to the electrochemical tests. Following the 120 hr exposure, all samples (wrought, LPBF, and LE DED) were rinsed with DI water, sonicated in DI water for 10 minutes, then air dried for mass measuring. Mass loss is expressed as a penetration rate in units of mm/year and calculated by Equation 1.

$\text{Corrosion Rate} = \frac{87660W}{Atd}$	Equation 1
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Corrosion rate is in units of mm/year, W is mass loss in grams, A is area in cm^2 , t is exposure time in hours, and d is the density of 316L in units of g/cm^3 . In the case of chromium nickel-molybdenum steels, a density value of 8 g/cm^3 was used as indicated in ASTM A262-B⁶¹. Similar to the DL-EPR solution,⁶² the addition of H_2SO_4 is to create an aggressive acidic environment while the addition of Fe_2SO_4 is to chemically polarize the metal samples. The redox potential of the ASTM A262-B solution has been reported to be $+0.645 V_{vs SCE}$ ⁶³.

3. Results

To understand the microstructure prior to corrosion testing, pretest characterization using EBSD, TEM/STEM-EDS, and SEM-EDS was utilized and presented. Following pretest characterization, results describing the corrosion phenomenology in sulfuric acid environments with a depassivating species probing the active dissolution for wrought, LPBF, and DED is shown especially at 24 hr OCP measurements and low anodic overpotentials. Post-test microstructure was characterized following polarization to better understand the influence of the microstructure on the corrosion behavior. The transpassive dissolution was investigated without the presence of a depassivating species and higher anodic overpotential. Similar to the active dissolution tests, post-test characterization was conducted and is shown. Lastly, the use of ASTM A262-B determined corrosion rates through accelerated boiling acid tests and showed the influence of microstructure on mass loss in wrought, LPBF, and DED.

3.1 The lower cooling rate in DED resulted in greater ferrite formation and differences in spatial elemental segregation when compared to LPBF

The differences in grain misorientation, grain structure, and formation of secondary phases for LPBF and DED were characterized using EBSD. Representative areas (Figure 1a) show that LPBF have a wide range of misorientation and grain structure along with higher fraction of smaller grain size in comparison to the larger grains of DED (Figure 1b). Both types of AM samples contain two phases: Iron body center cubic (BCC) and Iron face centered cubic (FCC). Iron BCC is associated with δ -ferrite and Iron FCC is associated with γ -austenite. It was determined that LPBF has minimal δ -ferrite at an average 0.2% in comparison to DED which has a higher average of 3.2%. In LPBF, the δ -ferrite signal originates from the grain boundaries whereas δ -ferrite signal originates both within the grain and at grain boundaries in DED. In the LPBF, the δ -ferrite is difficult to see in the original phase map, so a digital zoom was applied to better demonstrate the location of δ -ferrite in comparison to DED as shown in Figure 1c and f.

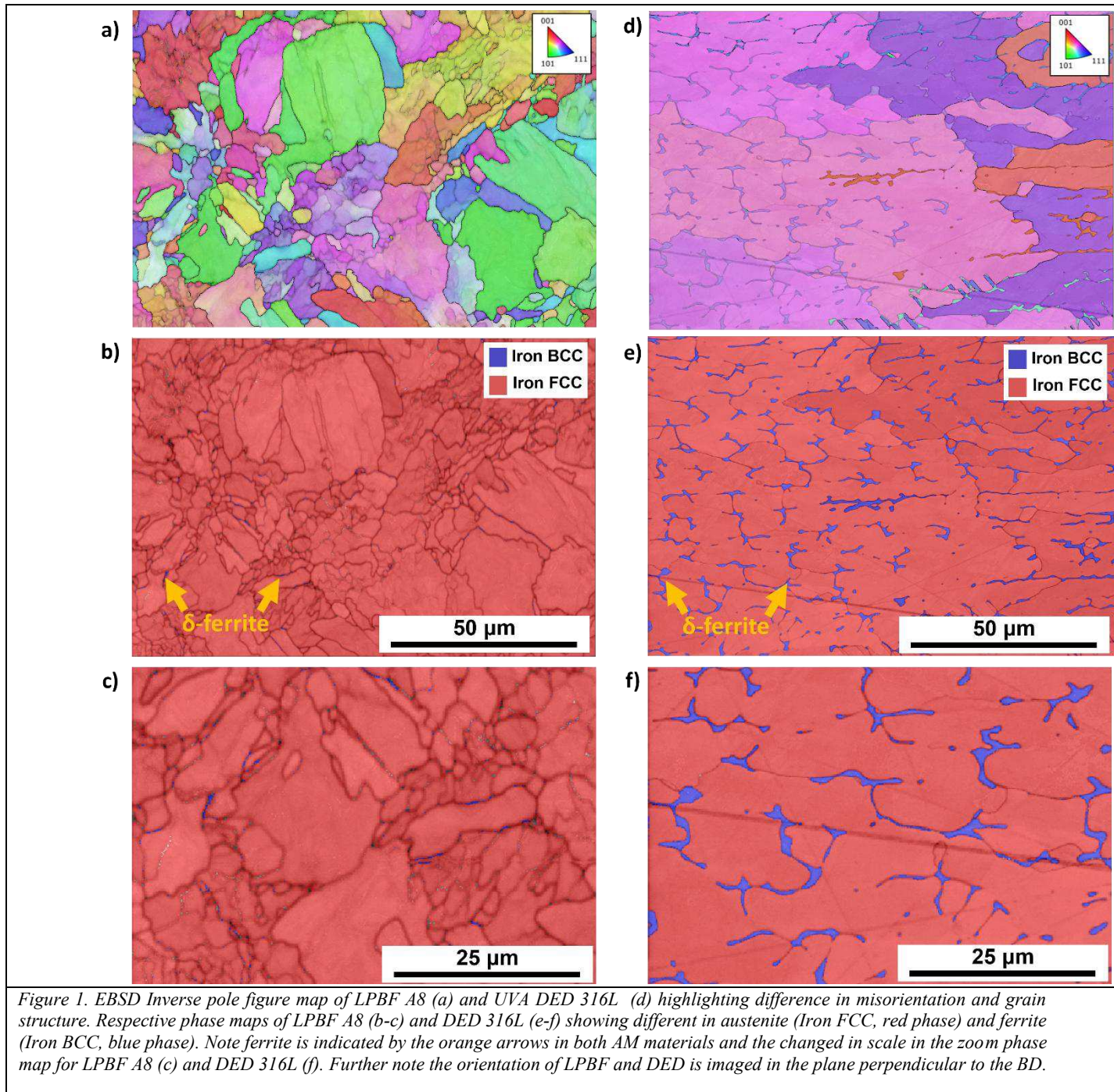


Figure 1. EBSD Inverse pole figure map of LPBF A8 (a) and UVA DED 316L (d) highlighting difference in misorientation and grain structure. Respective phase maps of LPBF A8 (b-c) and DED 316L (e-f) showing different in austenite (Iron FCC, red phase) and ferrite (Iron BCC, blue phase). Note ferrite is indicated by the orange arrows in both AM materials and the changed in scale in the zoom phase map for LPBF A8 (c) and DED 316L (f). Further note the orientation of LPBF and DED is imaged in the plane perpendicular to the BD.

To better understand the elemental distribution within the two detected phases and overall microstructure in LPBF and DED, analysis using SEM, TEM, and EDS was employed. Given the size of the microstructure and elemental segregation in LPBF and DED, TEM/STEM-EDS was employed for LPBF and SEM/SEM-EDS was employed for DED. Figure 2a shows a HAADF-STEM micrograph of a cellular dendrite that solidified parallel to the imaging direction.

The bright contrast at the cellular boundary indicates the high presence of dislocation density in comparison to the cellular interior. Figure 2a also shows STEM-EDS mapping from the same region and shows a depletion of Fe and slight enrichment of Cr, Ni, and Mo at the cellular boundary measured. A STEM-EDS line profile (Figure 2b) was conducted across a cellular boundary as shown in red arrow drawn in Figure 2a to further show the depletion of Fe and ad slight enrichment of Cr, Ni, and Mo which complements the elemental mapping. Figure 2c shows a representative micrograph of the DED microstructure where the orange arrows highlight a larger elemental distribution in DED in which Ni is depleted in regions where Cr and Mo are enriched (Figure 2d). The spatial distributions of Cr and Mo enrichment are notably different in LPBF and DED where LPBF enrichment is on the nanometer scale while the DED enrichment is on the micron scale.

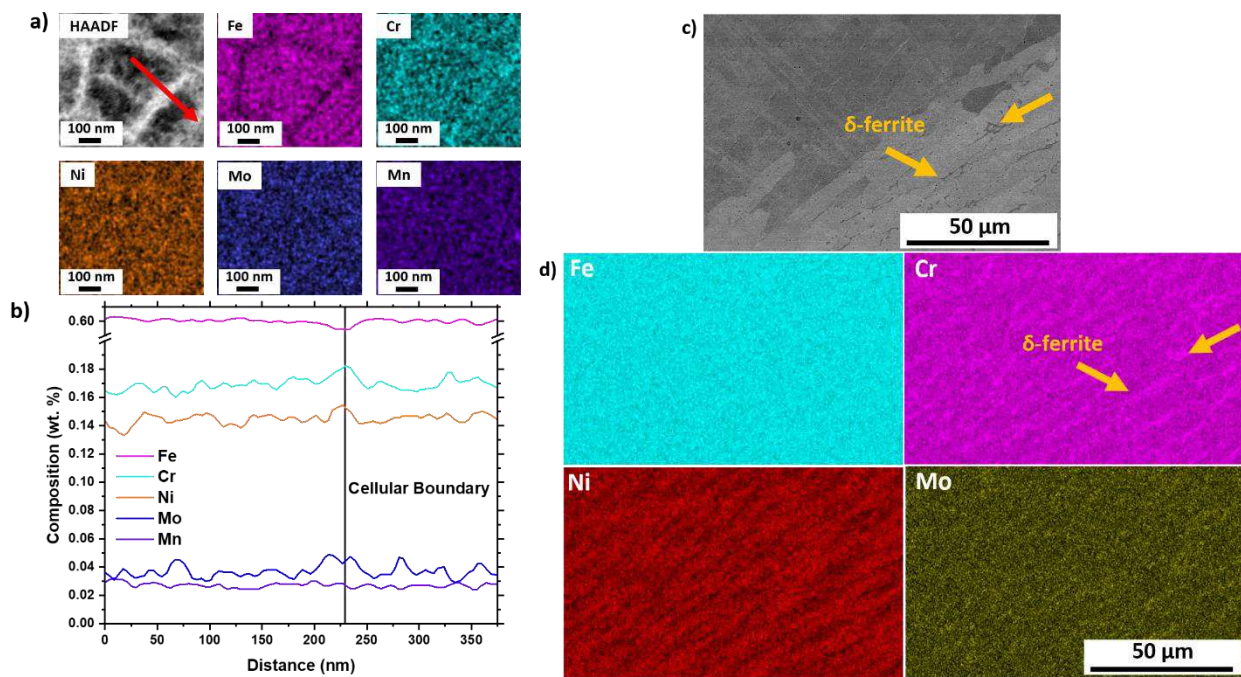


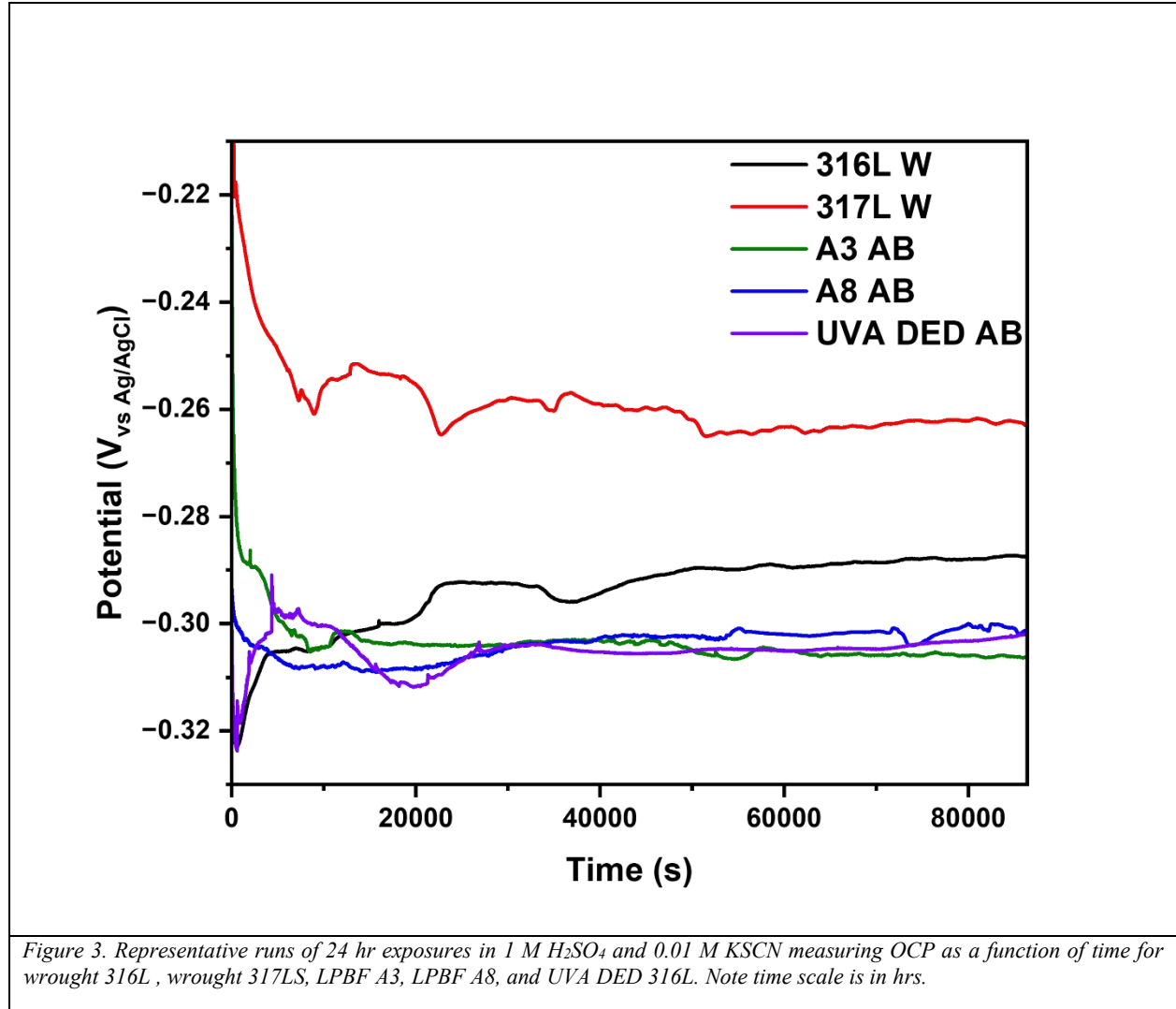
Figure 2. HAADF-STEM micrograph of the cellular microstructure in LPBF A8 with STEM-EDS of the elemental distribution within the cellular dendrite (a). STEM-EDS line profile (b) conducted across a cellular boundary from (a). SEM micrograph of the austenite and ferrite microstructure in UVA DED 316L (c) with SEM-EDS of the elemental distribution at the δ -ferrite phase as indicated by the white arrows (d). Note the orange arrows indicate δ -ferrite, the difference in scale when comparing TEM and STEM, and the difference in the color assignment for elements for STEM-EDS and SEM-EDS. Further note the orientation of LPBF and DED is imaged in the plane perpendicular to the BD.

3.2 Electrochemical potential controls which microstructural feature is dissolved

3.2.1 Active dissolution attacks regions of lower Cr and Mo

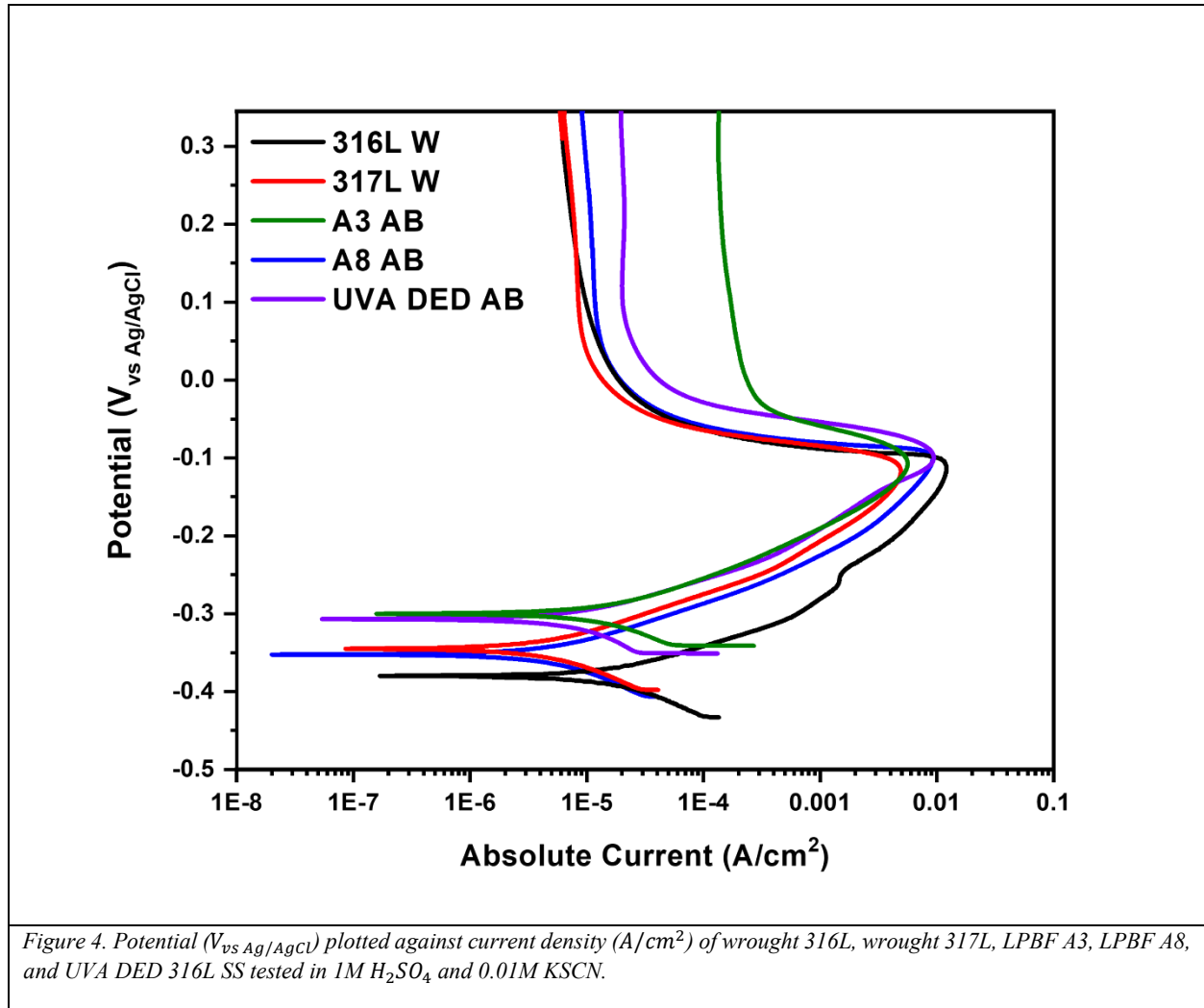
The OCP after 24 hrs of exposure in 1M H₂SO₄ and 0.01 M KSCN, all samples OCP reached steady state, but wrought and AM 316L exhibit different trends in Figure 3. Wrought 316L shows an subtle increase after 5.5 hrs then reaches steady state around 14 hrs $-0.29 V_{vs Ag/AgCl}$. The opposite is observed for wrought 317L which shows a large $-0.09 V_{vs Ag/AgCl}$ decrease in OCP

within the first 3 hrs and then reaches steady state around 14 hrs at $-0.26 V_{vs Ag/AgCl}$. For both LPBF 316L samples A3 and A8, there is a decrease in OCP within the first 3 hrs similar to wrought 317L and each maintains a steady state potential of $-0.30 V_{vs Ag/AgCl}$ and $-0.31 V_{vs Ag/AgCl}$, respectively. For DED 316L, there is an increase of potential at 1.4 hrs and a decrease in potential at 5.5 hrs before it reaches steady state at $-0.30 V_{vs Ag/AgCl}$ similar to LPBF 316L A3.



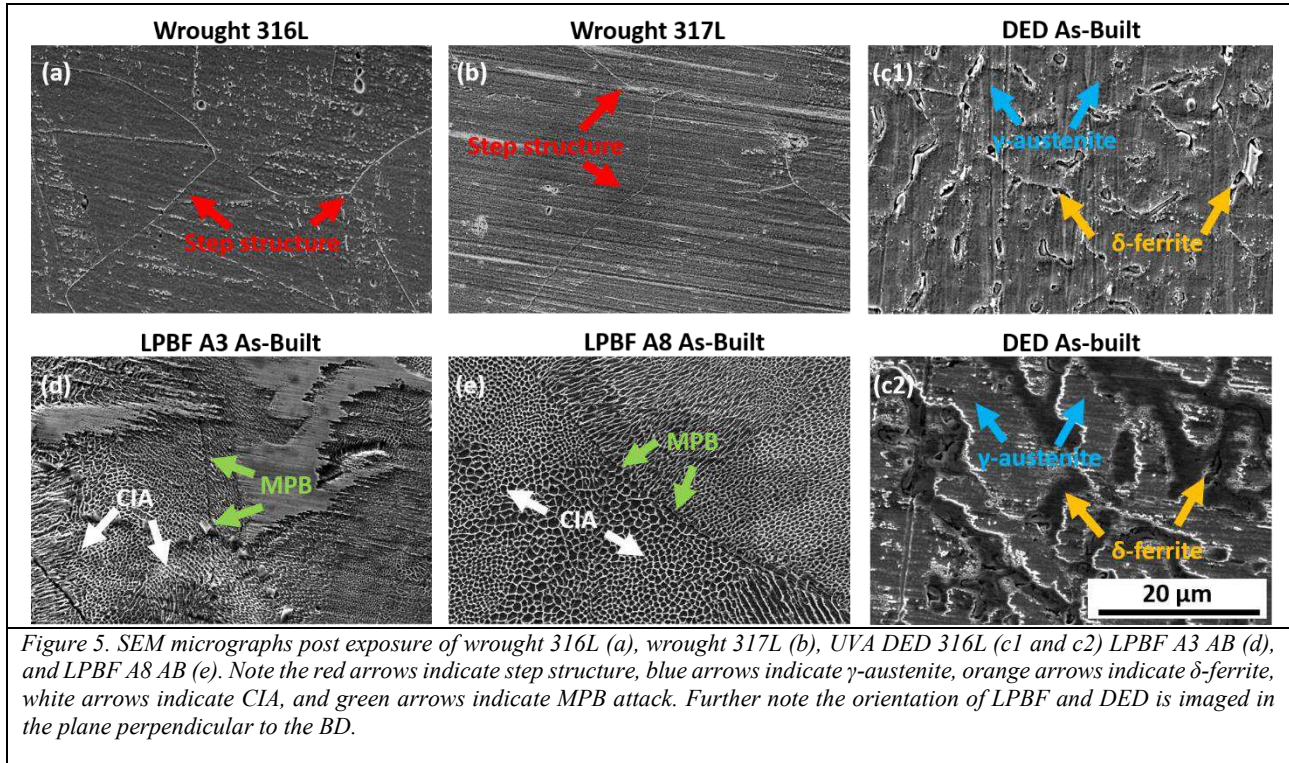
LPP tests conducted in 1M H_2SO_4 and 0.01M KSCN showed differences in anodic dissolution and passivation across all wrought, LPBF, and DED samples. Figure 4 shows that wrought 316L exhibits the highest current density in the active region (i.e., between -0.4 and $-0.1 V_{vs Ag/AgCl}$) in comparison to wrought 317L, both LPBF 316L samples, and DED 316L SS. The high current density measured (i_{max}) achieved was defined as the highest current density measured before passivation begins during the polarization test. Wrought 316L had the highest average i_{max} while wrought 317L had the lowest by nearly a factor of two. All AM 316L samples showed similar

average i_{max} values of $8.27 \times 10^{-3} \text{ A/cm}^2 \pm 0.001$. The primary passivation potential at which the surface transitions from active dissolution to passivation was found to be similar across all samples at $-0.102 V_{vs \text{ Ag/AgCl}} \pm 0.003$. Following complete passivation of the surface, the passivation across all wrought, LPBF, and DED samples led to varying passive current densities (i_{pass}). The average i_{pass} (collected at $+0.2 V_{vs \text{ Ag/AgCl}}$) was calculated for all sample types revealing that wrought 317L had the lowest i_{pass} of $1.66 \times 10^{-5} \text{ A/cm}^2$ whereas LPBF A3 had the highest i_{pass} $3.18 \times 10^{-4} \text{ A/cm}^2$ which was an order of magnitude higher than wrought and all other samples. DED 316L exhibited. LPBF A8 316L showed an average between wrought and DED with a value of $2.31 \times 10^{-5} \text{ A/cm}^2$.

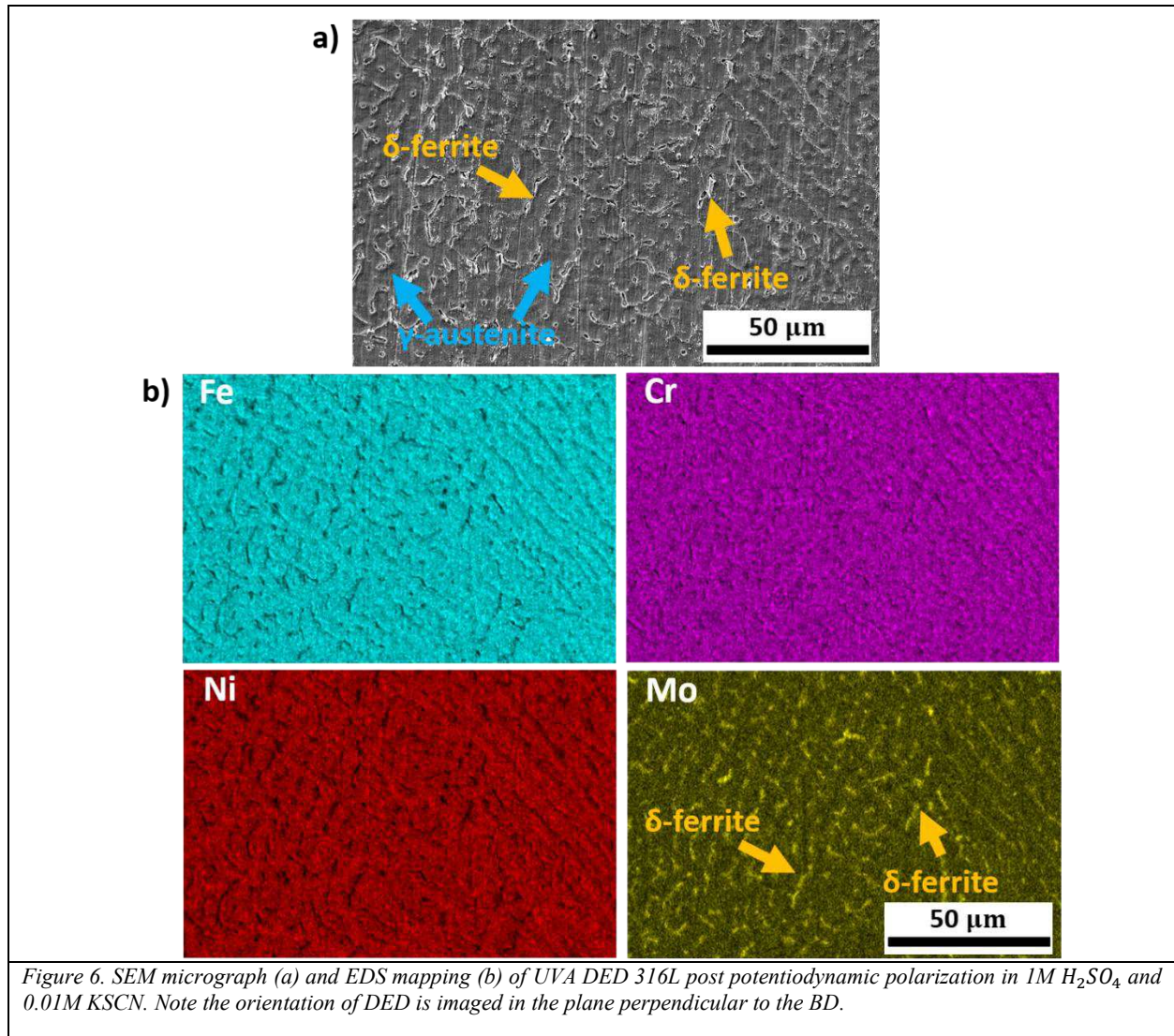


The corroded microstructures were characterized after the polarization in Figure 4 in 1M H_2SO_4 and 0.01M KSCN for all samples and are shown in Figure 5. Wrought 316L and 317L underwent crystallographic attack of the grains which in turn revealed a step-structure as indicated in Figure 5a-b. Conversely, LPBF and DED exhibited preferential attack of certain microstructural features.

DED 316L experiences preferential attack of the γ -austenite leading to regions where the δ -ferrite protrudes from the surface. The extent of attack at the δ -ferrite phase varies area-to-area as highlighted in Figure 5c1-c2. Both LPBF A3 and A8 316L underwent similar modes of attack which include cellular interior attack (CIA) and melt pool boundary (MPB) attack. There were no obvious signs of MPB attack in DED 316L.



Post-corrosion elemental analysis was conducted in LPBF and DED to better understand the influence of microstructure and elemental distribution on the observed corrosion behavior. Figure 6 shows a representative post-corroded microstructure of DED 316L following LPP in 1M H_2SO_4 and 0.01M KSCN. The δ -ferrite islands protrude from the surface while the γ -austenite recess into the surface. When mapping the elements using SEM-EDS (Figure 6b) it is evident that Cr and Mo map to the δ -ferrite islands while there is a depletion of Fe and Ni.



3.2.2 Spontaneous passivity is limited at high potential by transpassive dissolution

Figure 7 shows that when KSCN is removed from the 1M H₂SO₄ solution, all samples undergo a rapid positive increase of potential upon immersion for 24 hrs which slows and reaches a quasi-steady state OCP that is more electropositive in comparison to the 1M H₂SO₄ and 0.1M KSCN solution (Figure 3). The samples with the largest potential increase were wrought 317L (+0.420V_{vs Ag/AgCl}) and UVA DED (+0.420V_{vs Ag/AgCl}) followed by LPBF A3 (+0.303V_{vs Ag/AgCl}), wrought 316L (+0.233V_{vs Ag/AgCl}), and LPBF A8 (+0.217V_{vs Ag/AgCl}). LPBF A3 indicating some slight instability in potential has indicated by the potential spikes but reached a quasi-steady state potential.

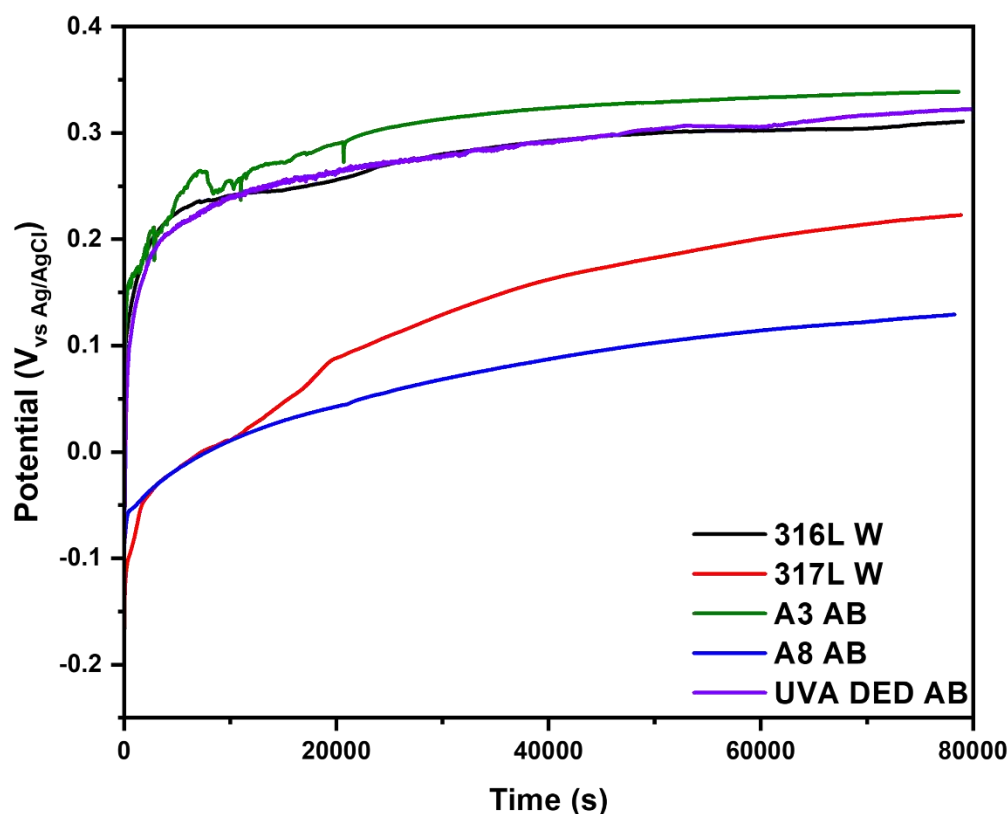


Figure 7. Representative runs of 24 hr exposures in 1M H₂SO₄ measuring OCP as a function of time for wrought 316L, wrought 317L, LPBF A3, LPBF A8, and UVA DED 316L. Note time scale is in hrs.

To better understand the passivation and transpassive behavior of the different materials, potentiodynamic polarization was conducted to a higher overpotential in 1 M H₂SO₄ alone (i.e., without KSCN) in Figure 8. The absence of KSCN allows all samples to spontaneously passivate rather than activating. The OCP measured in wrought 317L and LPBF A3 316L are more noble (electropositive) than wrought 316L, LPBF A8, and DED 316L SS. Besides the positive OCP, the passivation behavior for wrought 317L is similar to wrought 316L, LPBF A8, and DED 316L. As observed in Figure 8, the passive current density of LPBF A3 was higher than all other samples in 1M H₂SO₄. All other samples exhibited similar passive current density throughout the passive region. In the transpassive region, the potential at which samples turned from passive to transpassive was analyzed. The transpassive potential (E_{tp}) was measured but the differences between the lowest and highest sample were only 0.04 $V_{vs Ag/AgCl}$. At approximately +1 $V_{vs Ag/AgCl}$, the current diverges slightly for wrought 317L, LPBF A3, LPBF A8, and DED 316L in comparison to wrought 316L SS.

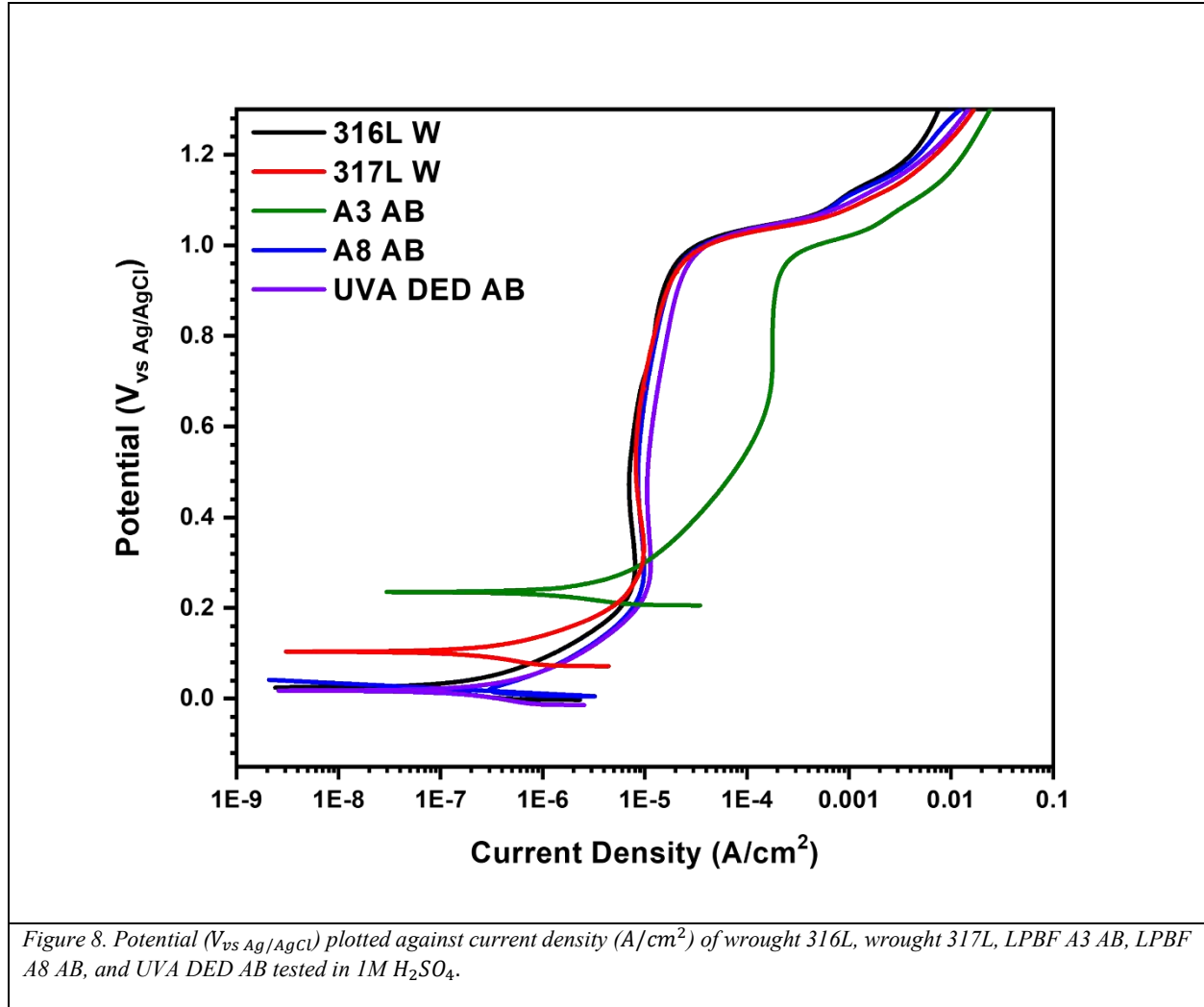
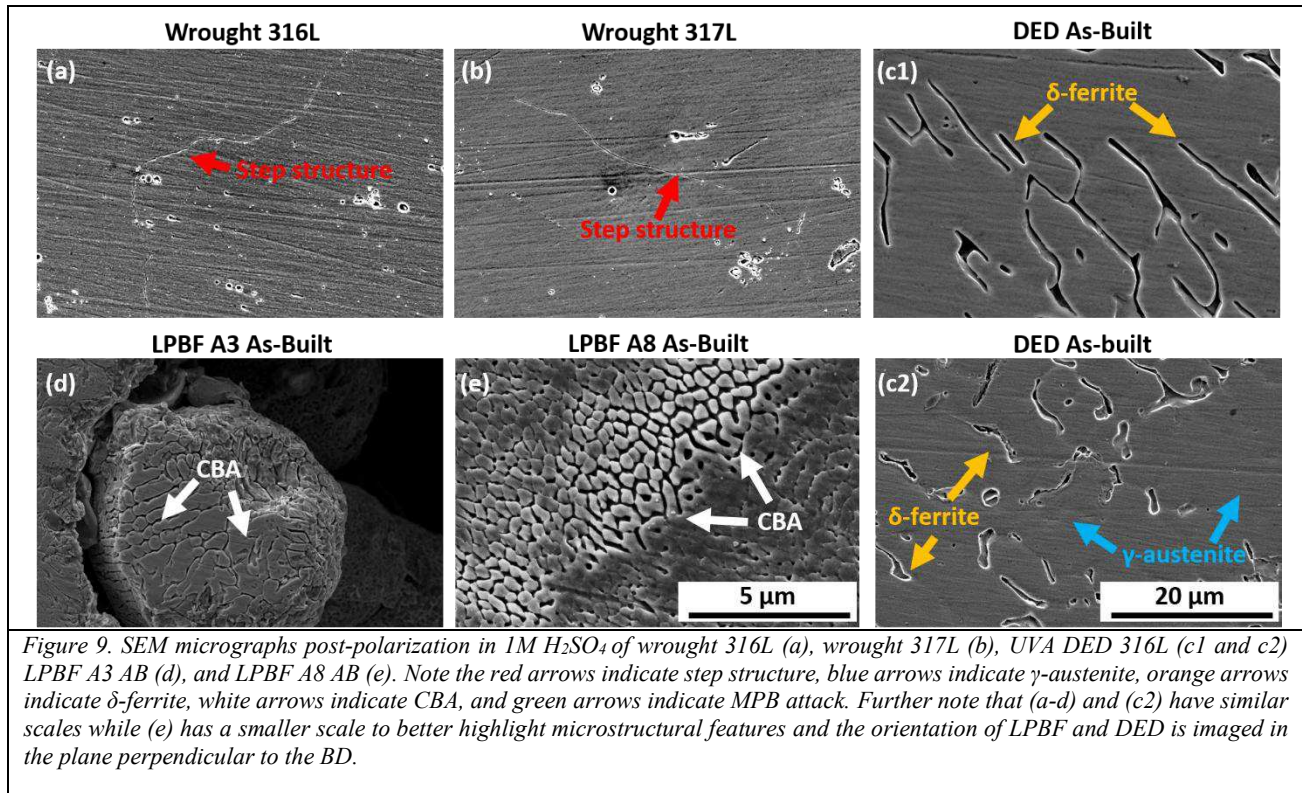


Figure 9 shows the post-polarization corrosion morphologies. Wrought 316L microstructure undergoes crystallographic attack, leading to a step-structure surface. The wrought 317L microstructure undergoes similar attack to 316L (Figure 9a-b), but the degree of grain etching is less than the active polarization test (Figure 9b). DED 316L experiences a change in localized attack of the two-phase microstructure where δ -ferrite is preferentially attacked over the γ -austenite phase (Figure 9c1-c2). Figure 9d shows that the cellular boundaries and columnar dendrites of APL A3 and A8 underwent boundary attack (CBA) while the cellular interiors were not preferentially attacked as observed in the polarization test under active conditions (Figure 9d-e).



Following transpassive dissolution testing, SEM and EDS were conducted to understand the influence of Cr and Mo on the dissolution observed on DED 316L as shown in Figure 10. The δ -ferrite islands now recess into the surface while the γ -austenite remain at the top of the surface. Unlike the DED 316L surfaces from active dissolution (Figure 6). SEM-EDS (Figure 10b) is unable to show the enrichment of Cr and Mo in the δ -ferrite islands. SEM-EDS was conducted on both LPBF samples but the results were also non-conclusive.

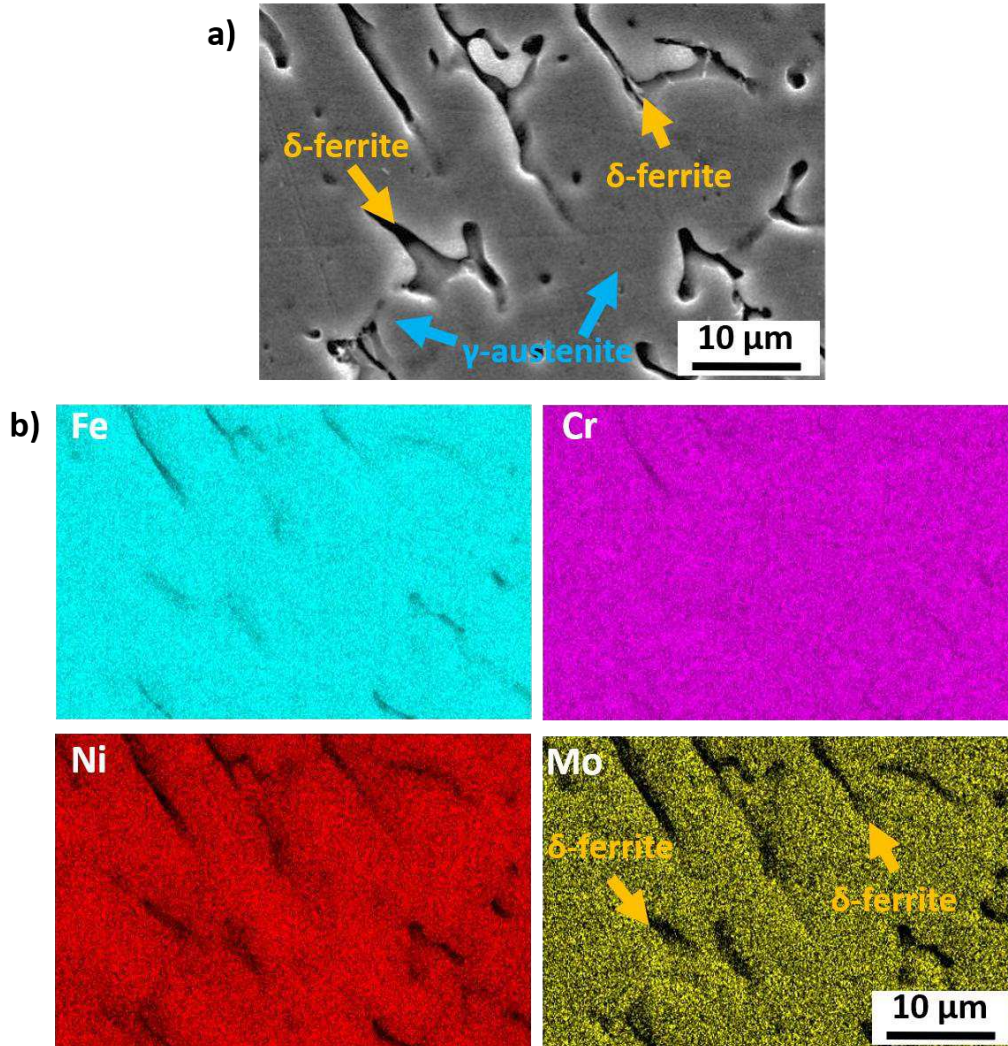
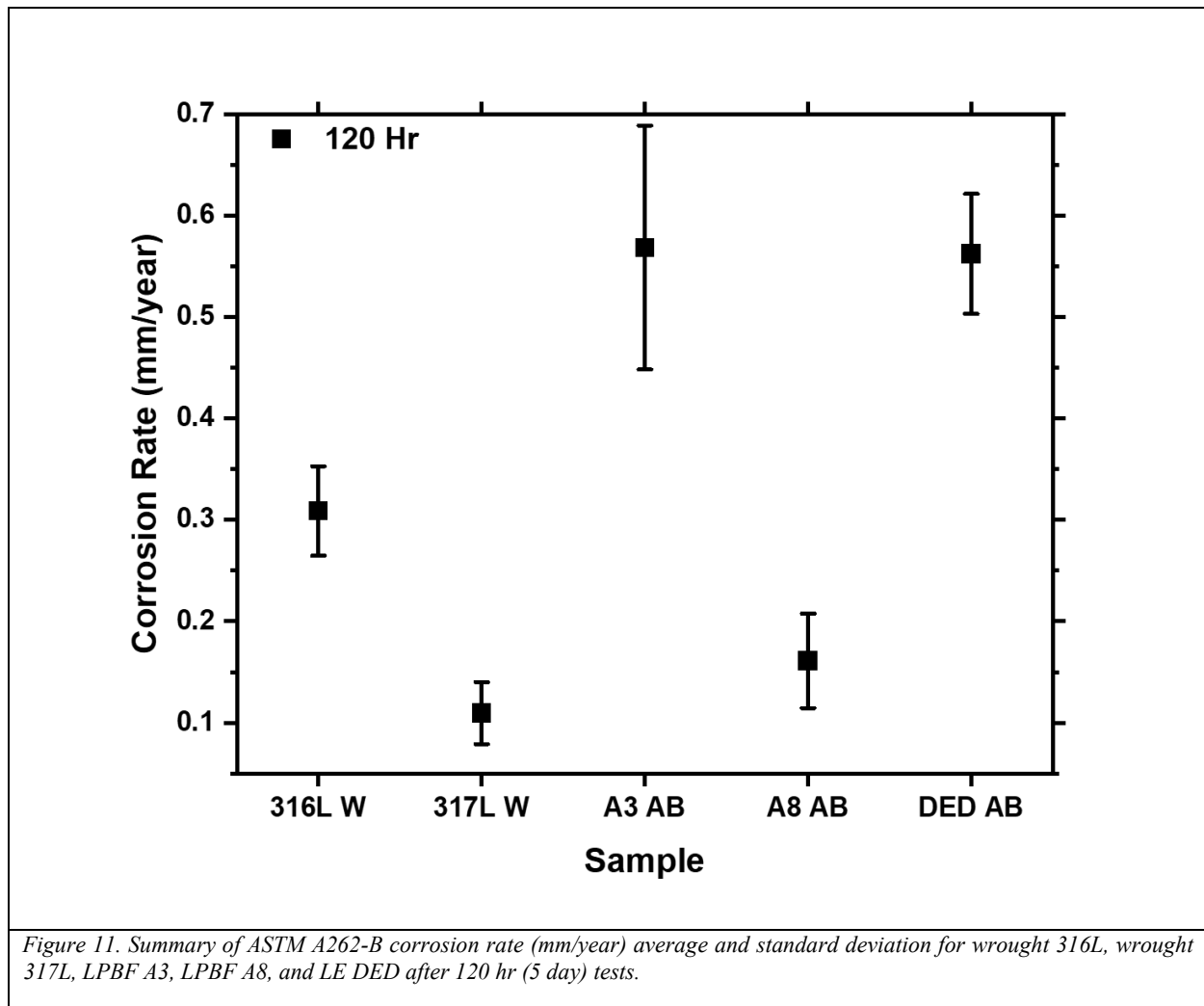


Figure 10. Corresponding SEM micrograph (a) and compositional SEM-EDS maps UVA DED 316L after potentiodynamic testing in 1 M H_2SO_4 . Note the orientation of DED is imaged in the plane perpendicular to the BD.

3.3 Mass loss testing reveals distinct differences in average corrosion rates when comparing wrought, LPBF, and DED

Corrosion rates measured from ASTM-A262-B boiling acid testing show differences in wrought, LPBF, and DED as shown in Figure 11. The lowest average corrosion rates were observed in wrought 317L and LPBF A8. Given the standard deviation of the two samples, the two are considered statistically similar. However, wrought 316L exhibited corrosion rates three times faster than wrought 317L and LPBF A8. LPBF A3 and DED had corrosion rates similar to wrought 316L.



Post-mass loss micrographs in Figure 12 reveal that ASTM A262-B exposures attack grains and susceptible non-equilibrium microstructure in wrought, LPBF, and DED. Wrought 316L and 317L experience extensive attack of the grains that leads to a visible grain boundary around the majority of the grains at 5 days as shown in Figure 12a-b. An interesting finding is the pit-like features in both wrought 316L and 317L where wrought 316L experiences pits that are in a line and follow a particular direction while wrought 317L experiences random pitting throughout the surface. The extent of the grain attack and pitting is observed after 5 days of exposure for both wrought samples as observed in Figure 12a-b. The microstructural attack is different in LPBF and DED due to the presence of non-equilibrium AM microstructure. After 5-day exposures, LPBF experiences grain attack the leads to the revealing of grains and grain boundaries similar to both wrought samples as observed in Figure 12c-d. Both LPBF samples experienced CIA which reveals the cellular dendritic boundaries and melt pool boundaries. DED experiences a somewhat different type of attack in which γ -austenite is preferentially attack relative to the δ -ferrite, but the difference in rates is less than in the polarization studies during transpassive dissolution (Figure 9 and Figure 10). After 5 days of exposure, the extent of attack is greater where large ditches are observed which is nearly on the scale of the grain as shown in Figure 12e.

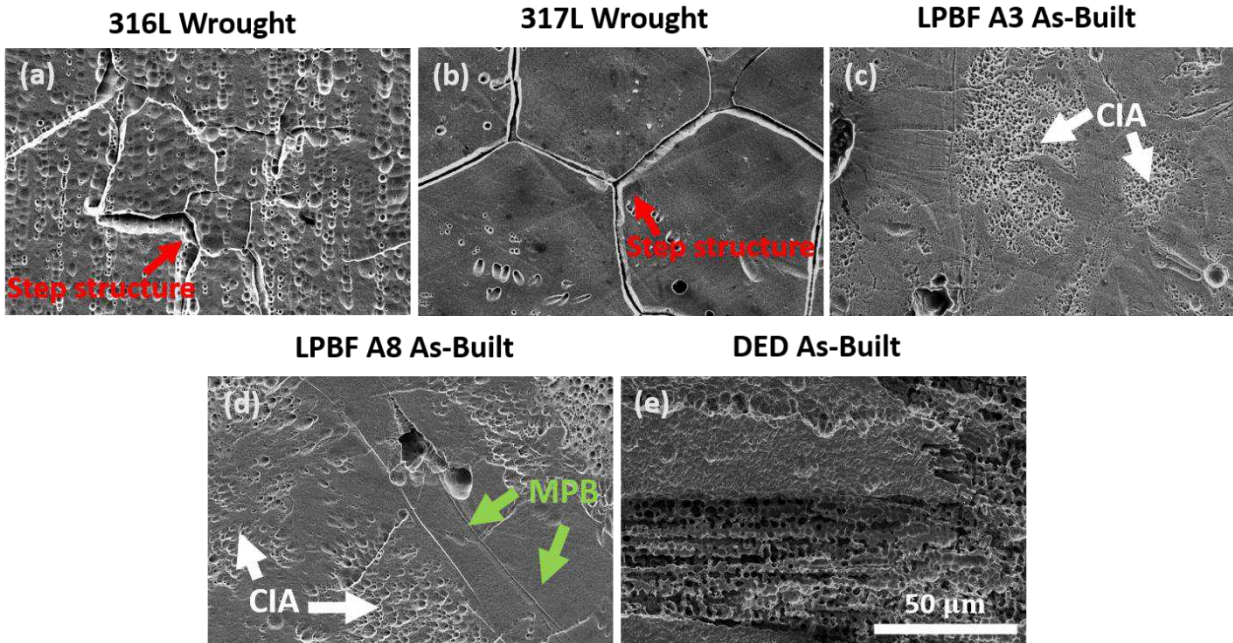


Figure 12. SEM secondary electron micrographs of wrought 316L (a), wrought 317L (b), LPBF A3 (c), LPBF A8 (d), and LE DED 316L post 5 day ASTM A262-B boiling acid testing. Note the orientation of LPBF and DED is imaged in the plane perpendicular to the BD.

4. Discussion

The use of ASTM standardized exposure and electrochemical tests has been a common approach to detecting and measuring the susceptibility to intergranular corrosion for austenitic stainless steels in various environments. The tests preferentially attack areas with lower Cr content to allow differentiation of sensitized and unsensitized materials^{13,21,25–27,64–66}. In this study, two ASTM standards, ASTM A262-15⁶¹ and ASTM G108⁶², were used to help elucidate the influence of AM processing on the corrosion behavior of wrought, LPBF, and DED 316L SS.

ASTM standards were developed for wrought alloys in which the microstructure is chemically homogenous due to heat treatment after casting. Cast and AM alloys undergo non-equilibrium solidification that leads to the formation of heterogenous microstructures. These differences would be expected to impact the corrosion response. For example, ASTM A262-15-B⁶¹ relies on the precipitation of Cr-bearing, second-phase particles preferentially at the grain boundaries which create adjacent Cr-depleted regions that dissolve at much higher rates than the interior of the grains. This grain boundary dissolution leads to grain fall-out and mass loss that is a measure of the amount of sensitization. Given that AM processes generate heterogenous microstructures such as dendrites, melt pool boundaries, porosity, and grain interlocking, the complexity of this microstructure can lead to heterogeneous distributions of Cr and thereby influence the mass loss measured from ASTM A262¹³. ASTM G108⁶² is a cyclic polarization electrochemical method using sulfuric acid with potassium thiocyanate to determine sensitization through measurements of the ease of reactivation after passivation^{13,21,25–27,64–66}. ASTM G108 utilizes the increased

susceptibility of Cr-depleted areas to reactivation after passivation to detect the level of sensitization by the preferential dissolution of these Cr-poor areas adjacent to Cr_{23}C_6 particles. The sensitization is manifested in the reactivation current density during a passive-to-active transition.

Understanding how AM materials respond to standard tests is important to avoid misinterpretation as test samples are generally not examined post-test with microscopy, with only mass loss or measured current density ratio being reported. The AM 316L literature provides evidence that AM processing causes the formation of both non-equilibrium microstructures and the partitioning or elemental segregation of critical alloying elements such as Cr, Ni, and Mo to certain microstructural features such as cell walls and melt pool boundaries^{48–60}. Dislocations at high density form in LPBF material because of the high strain created from rapid thermal shrinkage leading to a cellular dendritic nanostructure. It has been proposed that these dislocations make it energetically favorable for Cr and Mo to remain at the cellular walls during solidification⁶⁷. STEM-EDS conducted in this study (Figure 2) of cellular walls from a LPBF sample shows higher concentrations of Cr, Mo, and slightly higher concentrations of Ni at the cellular walls which aligns with observations made in multiple studies^{48–60}.

The extent to which this segregation leads to preferential attack of at Cr- and Mo-depleted areas other than sensitized grain boundaries during testing could make the interpretation of results from standard tests such as ASTM A262-B⁶¹ and ASTM G108⁶² challenging for AM materials. Thus, there exists a need to evaluate and understand the response of 316L produced by AM processes in ASTM standard testing. Herein we use results from ASTM standardized testing and well-established electrochemical procedures to understand the morphology of corrosion developed in wrought, LPBF, and DED 316L. Post-test microscopy is used to understand the influence of the microstructure on the measured corrosion behavior. The possible impact of these different morphologies on the interpretation of ASTM tests where standard procedures or electrochemical procedures do not require further evaluation of microstructure for interpretation is discussed.

4.1 Acidic, depassivating environments lead to active corrosion conditions that preferentially attack Cr- and Mo-depleted microstructural features in LPBF and DED

The basis for the corrosion resistance of stainless steels is the presence of Cr and Mo^{29–41}. These two elements provide the impressive corrosion protection through their impact on the composition and nature of the passive film that forms^{29–41} and/or their inhibition of dissolution under film-free conditions^{34,39}. The importance of the two elements to the quality of the passive film has been explored in depth, with the critical role of Cr being undisputed^{29–41}, whereas the role of Mo in the oxide continues to be the subject of some debate^{34–39}. For Cr, once the Cr content is above ~13 wt%, a percolating network is enabled that allows oxygen anions to bridge between neighboring Cr atoms, leading to an excellent passive film of Cr_2O_3 oxide^{29,30}. Meanwhile the role of Mo for stainless steels has been observed to raise pitting potentials, reduce anodic current densities, and thus enhance the stability of the Cr_2O_3 passive oxide film by filling in point defects within the oxide^{34,37–39}. Overall, the importance of Cr and Mo is critical for stainless steels to passivate and withstand corrosive environments.

Many accelerated testing solutions for stainless steel involve the use of hot, reducing, acidic solutions to detect intergranular corrosion susceptibility for wrought 304L and 316L^{13,68–70} and LPBF 316L^{13,21,25–28}. The high temperature (e.g., boiling) ensures that there is virtually no dissolved oxygen present that could aid in passivating the surface. ASTM G108 prescribes testing at room temperature but adds KSCN to 1M H₂SO₄ which acts as a depassivator, leading to active, film-free dissolution under open circuit conditions. Alloying with Cr and Mo have been shown to dramatically reduce the corrosion rate of stainless steels under such active, film-free conditions^{29,30}. The low open circuit potentials measured in this work for all three materials studied in the KSCN-bearing solution (~ -0.260 to $-0.310 V_{vs Ag/AgCl}$ as shown in Figure 4) are consistent with the claim that the surfaces are actively corroding in a film-free condition, as the active-passive transition is at $\sim -0.1 V_{vs Ag/AgCl}$.

The impact of Cr and Mo depletion is apparent from anodic polarization through the active-to-passive transition. As shown in Figure 5a-b, anodic polarization in 1M H₂SO₄ and 0.01M KSCN shows a distinct difference between 316L and 317L in the active corrosion region of the polarization curve. Because of the higher Cr, Ni, and Mo in wrought 317L, the active dissolution is slower than that of wrought 316L. That is, the dissolution current density is lower for wrought 317L at all potentials below the active/passive transition of $\sim -0.1 V_{vs Ag/AgCl}$. Post-test examination of the surfaces for both alloys reveals uniform attack of all grains and a step-like microstructure⁶¹ as observed in Figure 5a-b. From the micrographs it also appears that the grain boundaries etched along with the grains. Li et al.⁶⁸ observed similar attack in pure Cr, pure Ni, and wrought 304 in H₂SO₄ solutions containing KSCN. The proposed mechanism was similar to observations made in duplex 430 stainless steel where there is an increase in Cr dissolution at edges or kinks counteracting the formation of Cr-O-Cr passivating networks^{40,41}. Li et al.⁶⁸ showed evidence that thiocyanate disproportionates on the alloy surface into elemental sulfur and cyanate. The sulfur species adsorb at the step edges or ledges, allowing faster dissolution to occur there than at the smooth bulk surfaces such as the grains⁶⁸. When dissolution occurred at the edge of the grains leading to increased dissolution at the grain boundary in wrought 316L and 317L, it is believed that the mechanism is due to the sulfur adsorbing at the edges similar to Li et al.⁶⁸.

A different corrosion morphology is observed in LPBF 316L under active dissolution conditions than that of wrought 316L and 317L. The enrichment of Cr, Mo, and Ni at the cellular walls decreases dissolution and improve passivation leading to the cellular interiors dissolving faster. The result of this attack leaves a straw-like morphology as seen in Figure 5d-e. A similar observation has been made for melt pool boundaries where Cr and Mo are depleted up to ~ 0.5 wt.% leading to preferential attack⁵¹. LPBF A3 exhibited the highest passive current density of all materials tested. The difference is particularly striking when compared to LPBF A8 which had an identical composition and was made at the same time, albeit with different processing parameters. LPBF A3 had a lower laser power and high scan velocity which can cause lack of fusion (LOF) porosity due to incomplete melting and fusion between powder particles during melting and solidification⁷¹. These process parameters caused LPBF A3 to exhibit a large amount of visible LOF porosity (98.2% density) whereas LPBF A8 (99.5% density) did not have any easily visible porosity on the surface¹³. This observation follows similar observations with rough surfaces or porosity exhibiting high passivation current densities and lower break down potentials in both wrought^{19,72–75} and LPBF 304L or 316L^{72–75}.

Choundraj et al.⁵⁷ observed that removal of the passive oxide from cathodic polarization results in the presence of an active-to-passive transition in 1 wt.% HCl similar to results presented in Figure 4. Although that work did not use a depassivating agent such as KSCN, substantial cathodic polarization can disrupt and remove air-formed oxides present on the stainless steel surface prior to anodic polarization. When the cathodically polarized samples (oxide removed) were anodically polarized, an active-to-passive transition was observed resulting in cellular interior attack⁵⁷. This attack is consistent to the attack observed for both LPBF samples in Figure 5d-e. Choundraj et al.⁵⁷ noted that cell walls have a high concentration of dislocations in comparison to the cellular interiors. However, it was concluded that the corrosion attack of cellular interiors or walls was due to the differences in Cr and Mo distribution between the two regions rather than the high dislocation density at cellular walls.

During the DED process for 316L, a two-phase microstructure develops due to the slower cooling rates relative to LPBF process, i.e., 10^2 – 10^5 K/s^{6,7} for DED in comparison to 10^3 – 10^8 K/s for LPBF⁴. This solidification process during DED is considered to follow the ferritic-austenitic mode where delta-ferrite forms from the liquid and transforms to gamma-austenite during cooling^{76,77}. Due to thermal cycling during following deposited layers, ferrite continues to convert to austenite leaving a small amount of skeletal or vermicular delta ferrite in the final microstructure^{76,77}. The ferrite will be higher in Cr and Mo than the austenite as all three form BCC crystal structures. Given that Cr and Mo preferentially partition to ferrite during the solidification process (Figure 1, Figure 5, and Figure 6), the attack of austenite during active dissolution is prominent in the DED material. This result is consistent with the observations discussed above: (1) higher Cr and Mo in wrought 317L leads to slower anodic dissolution and (2) the Cr- and Mo-rich cellular walls in LPBF corrode slower than the cellular interiors. To confirm the dissolution mechanism for DED 316L, SEM-EDS was conducted to understand which regions of the two-phase microstructure were selectively attacked in Figure 6. White arrows show the regions with higher Cr and Mo EDS signals, consistent those regions being ferrite while the lower Cr and Mo EDS signals are consistent with those regions being austenite. The microstructural attack further confirms this mechanism with respect to the SEM-EDS showing that Cr- and Mo-depleted microstructural features are susceptible to preferential attack in LPBF and DED, albeit, on different size scales and different phases. Further it is worth noting that the attack that was observed in the UVA DED samples that were testing during polarization exhibited a similar attack of the microstructure to the LE DED samples.

4.2 Cr and Mo are preferentially oxidized to higher valence states and dissolve at elevated potentials.

Under highly oxidizing conditions, stainless steels undergo transpassive dissolution. During transpassive dissolution there is preferential dissolution of elements in the passive film to higher valence states that are highly soluble, such as Cr (Cr^{3+} to Cr^{6+})^{43,44,78} and Mo (Mo^{4+} to Mo^{6+}) at high anodic potentials and low pH environments^{42,45–47}. As with active dissolution at the much lower potentials, the dissolution appears activation-controlled (i.e., the current density increases exponentially with increasing potential). It is well known^{43,44,78} that under these conditions, Cr in solid solution is no longer protective as it is oxidized to the hexavalent state, forming the highly

soluble chromate ion. A similar argument is made for Mo where tetravalent state is oxidized to the hexavalent state but the evidence for how process occurs in the presence of Cr and Cr_2O_3 is still unclear^{42,45–47}.

To investigate the transpassive behavior and resulting corrosion morphology, testing on both wrought alloys was done in 1 M H_2SO_4 without KSCN. Under these conditions, the stainless steels spontaneously passivate and their OCP remains in the passive region (Figure 4 and Figure 7) and⁶⁸. From the very small differences the passive current density and the transpassive potential, E_{tp} , it would be assumed that the resulting microstructure would be similar across wrought, LPBF, and DED. Wrought 316L and 317L exhibited an initial sharp increase in potential upon immersion followed by a gradual stabilization of potential out to 24 hrs. Recall that in the 1M H_2SO_4 with 0.01M KSCN the OCP fell dramatically after a short time of immersion as the air-formed film was dissolved and the surface activated.

After polarization into the transpassive region (Figure 8), wrought 316L and 317L show dissolution morphologies that are nominally uniform due to their chemically homogeneous microstructures. Figure 9 shows post-test wrought 316L microstructure underwent grain etching. The wrought 317L microstructure undergoes similar attack to 316L, but the degree of crystallographic attack is less than polarization in the depassivating environment (Figure 5).

4.3 Under transpassive conditions cellular walls are attacked in LPBF, whereas ferrite is attacked in DED

Transpassive dissolution morphologies of LPBF and DED materials were consistent with the distribution of Cr and Mo in the microstructural features. Figure 9 shows that the cellular walls and cellular dendrites of LPBF A3 suffered preferential attack of the cellular walls instead of the interiors as observed under active conditions (Figure 5). As observed in Figure 2, Cr and Mo are enriched in the walls of the cellular walls and columnar dendrites. Similar to LPBF A3, LPBF A8 undergoes exterior attack of the dendritic microstructure. Figure 9 highlights the attack of cellular walls at a melt pool boundary showing that the Cr- and Mo-enriched walls are susceptible to transpassive dissolution at higher anodic potentials. This result shows that regardless of process parameters, the microstructure produced from LPBF is susceptible to cellular wall attack under transpassive dissolution. Choundraj et al.⁵⁷ found that when the surface was tested in the as-polished condition in 1 wt.% HCl solution, the undisrupted passive film resulted in no active-to-passive transition but instead spontaneous passivation. This observation is similar to Figure 8 in this current study where all samples exposed to H_2SO_4 alone experienced spontaneous passivation and no active-to-passive transition. When the anodic potential reaches transpassive conditions, attack of the cellular walls was characterized and identified⁵⁷. Partial scans of the as-polished surface at low anodic polarizations relative to the OCP led to very minimal attack of the microstructure due to the presence of a passive oxide. These findings confirm that the enrichment of Cr and Mo observed in previous works⁵⁷ and this current work are more susceptible to dissolution under transpassive conditions.

Figure 9c1-c2 shows the attack of the ferrite phases in the DED material. This attack is due to the higher amount of Cr and Mo within ferrite leading to accelerated transpassive dissolution at higher

anodic potentials. The enrichment of Cr and Mo in the ferrite is not obvious in Figure 10 because it dissolved preferentially and that limited the signal from those areas. UVA DED and LE DED exhibited similar microstructures confirming this behavior is consistent across different DED builds.

4.4 Acidic oxidizing solution at elevated temperature (ASTM A262-B) corrodes lower Cr and Mo microstructure in LPBF and DED.

Similar to the polarization tests conducted in 1M H₂SO₄ with and without 0.01M KSCN, the corrosion during ASTM A262-B testing appears to be influenced by material chemistry and microstructural attack is observed. LPBF A3 corroded 3 times faster than LPBF A8 (Figure 11) where both LPBF materials showed preferential attack of cellular walls and MPB, and grains were revealed as observed in Figure 12c-d. The findings were similar to that of the anodic polarization in 1M H₂SO₄ and 0.01M KSCN where Cr- and Mo-depleted regions were susceptible to attack. Because LPBF A3 and A8 had nearly identical material compositions, it is likely that the presence of LOF porosity in LPBF A3 (98.2% dense) led to more surface area exposed leading to more mass loss than LPBF A8 (99.5% dense) as observed in ¹³. That said, when the porosity is minimized as in LPBF A8, the mass loss was similar to wrought 317L which aligns with the impact of Cr, Mo, and Ni on the dissolution in the acidic oxidizing environment.

DED 316L experienced corrosion rates that were similar to wrought 316L and LPBF A8. After 5 days, the austenite matrix experienced higher attack than ferrite, leaving behind the ferrite microstructure. The surface of the DED sample also shows large ditch areas where accelerated corrosion occurred (Figure 12e). It is unclear why the accelerated attack happened at these regions although the microstructure of the DED was not uniform. Further work needs to be done to better understand this complex microstructure.

In wrought 316L, the corrosion rate is nearly triple the corrosion rate of 317L after 5 days (Figure 11). These findings are similar to that of the anodic polarization in 1M H₂SO₄ and 0.01M KSCN solution however the difference is ASTM A262-B ⁶¹ is a mass loss technique used to determine sensitization through the loss of individual grains ⁶¹. Given that no samples were heat treated to induce grain boundary attack, the increase Cr, Mo, and Ni in wrought 316L is likely one contributing factor for decreased mass loss. Unlike wrought 316L, wrought 317L does not undergo a pit like attack within the microstructure but exhibits similar grain attack that reveals a distinct edge between grains as seen in Figure 12a-b. The distinct edge between grains is consistent with the findings from Li et al. ⁶⁸ where accelerated corrosion was observed at grain boundaries despite the specimen's lacking sensitization. The pit-like features observed in wrought 316L follow a distinct direction and is believed to be due from a mechanical rolling process creating stress that is susceptible to pitting ⁷⁹. The same wrought 316L material was used in Montoya et al. ¹³ where all samples were heat treated to induce sensitization. Findings from this work showed the lack of these very small pit-like features corroded during ASTM A262-B test which proves that a stress relief likely occurred removing the stresses created during the grinding process. ⁷⁹

4.5 Dislocations are not the driving force for dissolution at inter-dendritic regions or cellular walls in LPBF 316L.

Because of the high dislocation density at the cellular walls, one would expect that the preference of attack at the walls should increase higher potentials as the highly defective surface would ease dissolution. Instead, the local elemental composition differences dominate with the applied potential determining whether Cr and Mo are protective or not. Under active dissolution, both elements inhibit dissolution, so Cr- and Mo-rich areas dissolve slower while at high applied potential transpassive dissolution causes Cr- and Mo-rich areas to dissolve faster.

The formation of dislocations has been associated with the rapid solidification process that occurs during the LPBF and DED process for materials where dislocations form at the inter-dendritic regions and contribute to the partitioning or elemental segregation of solute atoms. For 316L, the elemental segregation of critical alloying elements such as Cr and Mo has been observed from many studies^{48,50,58-60}. It has been observed that where the elemental segregation occurs is consistent with the high-density dislocation networks along the cellular walls with the use of high resolution techniques such as TEM and 2D XRF^{48,54,58,59}. Further, there has been observations of dislocation networks in cellular walls in DED with enrichment of Cr and depletion of Fe further confirming the presence of highly dense dislocation networks in LPBF and DED materials⁵⁹. Although many of these studies^{48,50,58-60} have observed large amounts of dislocations and elemental segregation of Cr and Mo at the cellular walls in DED and LPBF, the influence of dislocations on the localized corrosion of DED and LPBF has not been fully investigated in literature. For example, all these studies that used TEM and STEM-EDS to identify dislocation and elemental segregation at the inter-dendritic regions conducted an etching procedure^{48,59,60} or corrosion testing⁵⁸. Etches conducted in a reducing, acidic environment (e.g., distilled water-H₂O:H₂SO₄:HF – 90:8:2⁶⁰ or 1g picric acid, 5mL HCl, and 100 mL ethanol⁵⁸) resulted in attack of the cellular interiors. These findings are consistent with the current study findings and explanations where cellular interior attack was observed when anodically polarizing DED and LPBF in 1M H₂SO₄ and 0.01M KSCN.

However, studies that employed oxalic acid (0.5M-1.2M) and a polarizing environment (3V-7V) greater than 1V, resulted in attack of the cellular walls undergoing transpassive dissolution^{48,59,80}. These findings are consistent with the current study as Cr- and Mo-enriched cellular walls or phases underwent transpassive dissolution in LPBF and DED. An interesting finding from Tian et al.⁵⁴ found that LPBF 316L FIB lamella exposed to 1 wt.% HCl experienced attack at the cellular walls when polarized +460mV to its measured OCP. There was no conclusive evidence of the dissolution mechanism, but two mechanisms were proposed: (1) attack was due to high strain energy associated with the dislocation accumulation or (2) preferential attack due to elemental segregation. Bulk experiments conducted on the same sample revealed similar attack at the cellular walls but the applied potential went -0.2V to 1.2V with respect to OCP likely caused transpassive dissolution to occur. Another interesting observation was made by Sprouster et al.⁵⁸ where cellular interior attack was observed inside a localized corrosion site after cyclic voltammetry in 0.6M NaCl. This observation is consistent with a modern understanding of pitting in which the activated surface inside the localized site was acidic and very high in chloride, leading to the inability of a surface oxide to be stable and thus activation of the surface, as in the 1M H₂SO₄ and 0.01M KSCN.

From the evidence provided by previous work and this current study it is evident that the preferential attack that occurs at the AM microstructure is driven by the depletion and enrichment of Cr and Mo at the inter-dendritic regions or secondary phases in LPBF and DED 316L. Although there is a large presence of dislocations at the inter-dendritic regions, the electrochemical etches and experiments prove that dislocations do not influence attack at the inter-dendritic regions due to the applied potential driving susceptibility for dissolution around or at the inter-dendritic region.

Overall, the current work demonstrates that when using ASTM standardized testing on AM 316L, post-test microscopy is required, even when the standard does not. Failure to do so can result in confusing interpretations, particularly when it comes to testing for sensitization. As noted above, high polarization in oxalic acid leads to preferential attack of the cell walls. ASTM A262 Practice A⁶¹ is the Oxalic Etch test in which the sample is polarized to 1 A/cm² for 1.5 minutes. Similar attack has been observed for ASTM A262 Practice C⁶¹ which uses boiling concentrated nitric acid. Conversely, we have shown that ASTM A262-B causes preferential attack of the cell interiors. These differences in the corrosion morphology for different tests that are used to detect the same phenomenon, i.e., sensitization, could be confusing with regards to interpretation and would be confounded with differences in grain size and interlocking that could impact all of the tests. ASTM G108 testing is also affected as it is shown here that 317L has lower current densities in the active region than 316L due to the former's higher Cr and Mo^{13,70}. Beyond tests for sensitization, it has been shown that in ASTM G48 testing, pits formed on LPBF 316L have surfaces on which the interiors of the cellular structure have been preferentially attacked⁽⁸¹⁾ consistent with the understanding that during pitting of stainless steel, the local solution chemistry is highly acidic and reducing due to the absence of dissolved oxygen.

5. Conclusions

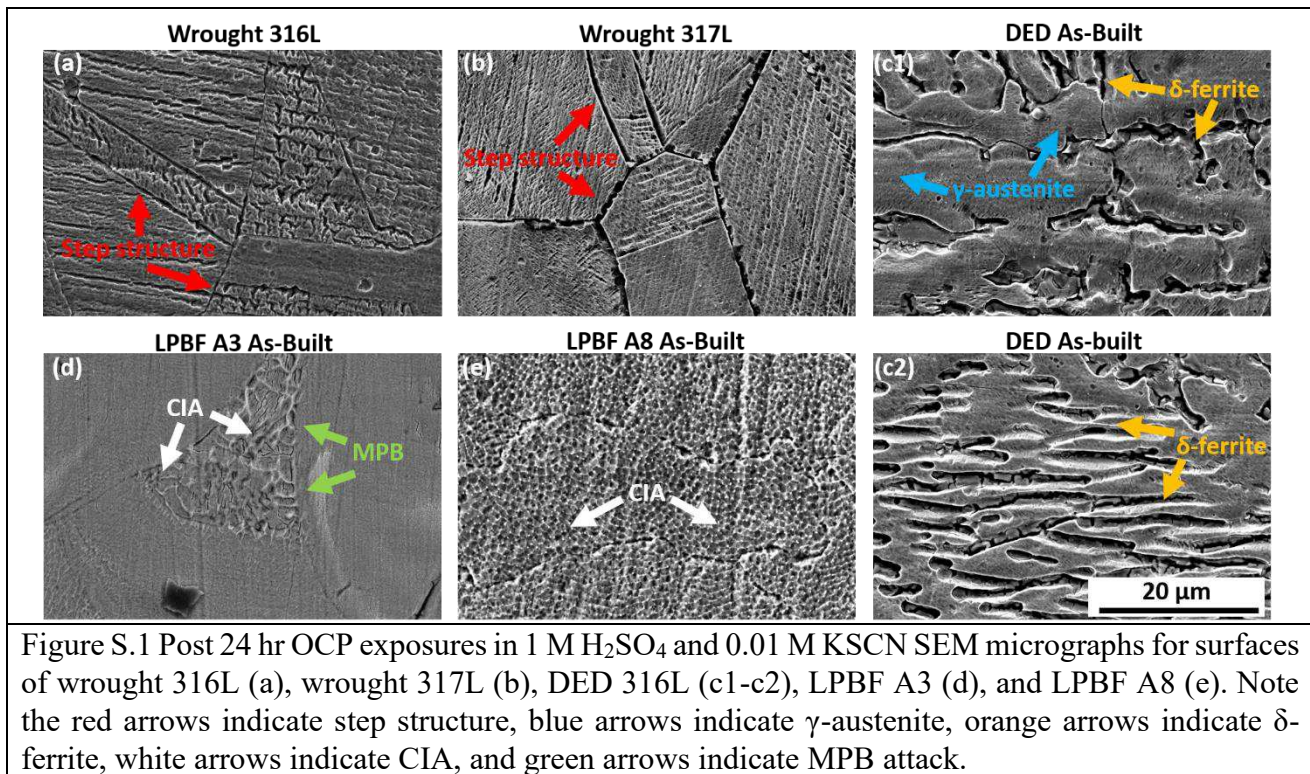
The corrosion morphology of LPBF and DED 316L is determined by the partitioning or segregation of Cr and Mo into certain microstructure features. Under active, film-free dissolution conditions, LPBF preferential attack occurs at the cellular dendrite interiors and melt pool boundaries whereas in DED preferential attack occurs in the austenite phase. Wrought experiences grain etching due to the lack of elemental segregation. High anodic overpotentials without the presence of a depassivator lead to the Cr- and Mo-rich microstructure becoming attacked. For LPBF this preferential attack includes the cellular walls and the ferrite in DED. Wrought experiences no distinct microstructural attack.

ASTM G108 DL-EPR test detects Cr- and Mo-depleted areas of the microstructures in LPBF and DED while showing the influence of increased Cr and Mo in wrought 317L in comparison to wrought 316L. ASTM A262 Practice B also detects the Cr- and Mo-depleted microstructures in LPBF and DED similar to ASTM G108. The corrosion rate was observed to be highest when LPBF processing parameters are not optimized for density (e.g., LPBF A3). DED experiences large amounts of dissolution visually, but the cause needs to be further investigated. From the results produced from ASTM G108 and ASTM A262 Practice B, it is evident that dislocations may be the primary driving force for elemental segregation in AM materials, but they are not the driving force for dissolution under all the conditions tested. Observations made by other studies show similar trends to those presented in this work.

Supplemental Information

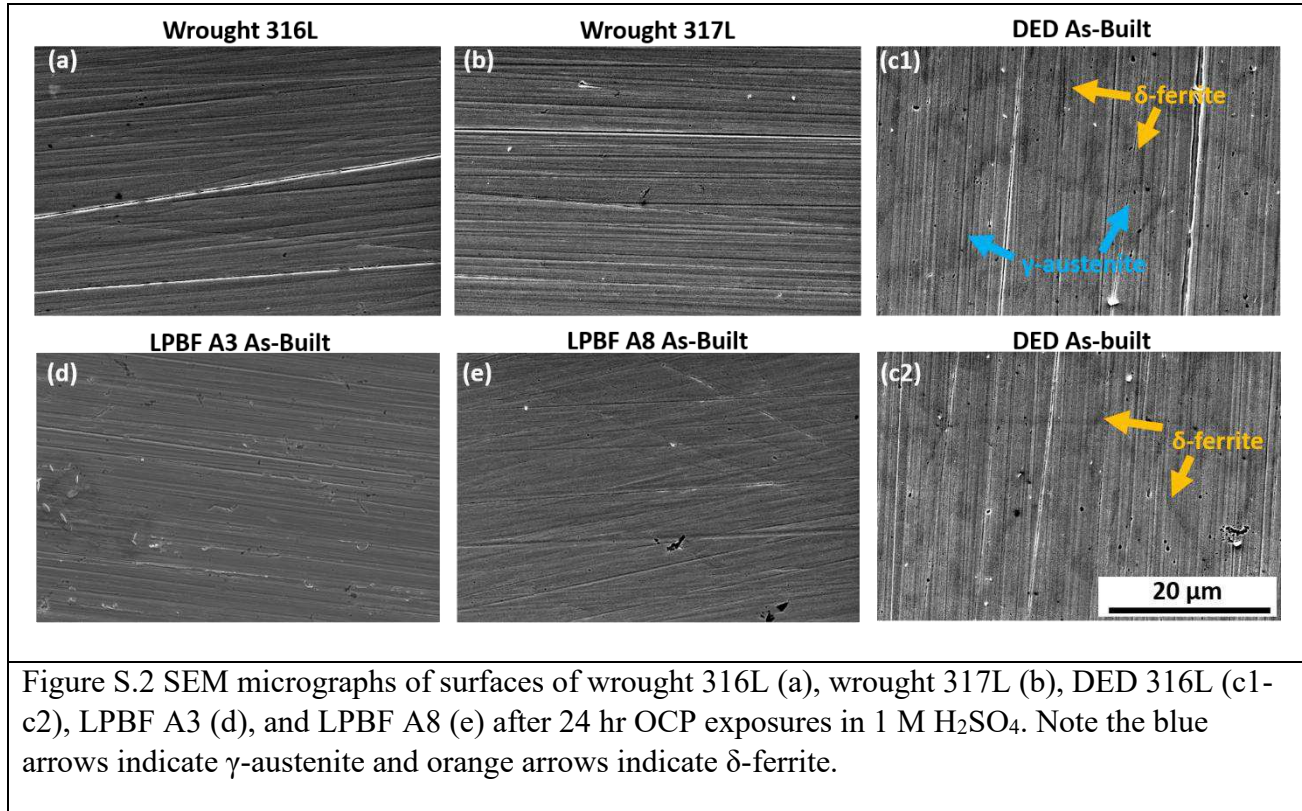
Cr and Mo depleted microstructure is susceptible to selective attack in open circuit conditions.

All samples exhibited attack of the microstructure with all three manufacturing conditions experiencing different types of attack (Figure S.1) following 24 hr OCP exposure in 1 M H₂SO₄ and 0.01 M KSCN. Wrought 316L and 317L undergo crystallographic attack or etching of the grains which reveal a step-like structure or the difference in grain heights as seen in Figure S.1a and Figure S.1b respectively. DED 316L experience attack of the dual phase microstructure where the δ -ferrite phase undergoes selective attack which recedes below the surface, while the γ -austenite phase experiences a uniform like attack. Two representative areas of this attack are shown in Figure S.1c1 and c2. From the representative microstructure no grains or AM microstructural features were distinctly present like all wrought and LPBF samples. Both LPBF samples experience similar attack of the non-equilibrium AM microstructure including attack of the cellular dendrite interior or cellular interior attack (CIA) and melt pool boundary (MPB) attack as observed in Figure S.1d and e. Grains are also attacked similarly to both wrought samples revealing a step-like structure as observed in Figure S.1e.



Without a depassivating species, all samples passivate an oxide that protects the surface from corrosion. After 24 hr exposures in 1M H₂SO₄, the surface revealed little to no microstructurally-specific attack in Figure S.2 in comparison to the exposures with KSCN present in Figure S.1. Surfaces that showed no microstructural attack were wrought 316L (Figure S.2a), wrought 317L (Figure S.2b), LPBF A3 (Figure S.2d), and LPBF A8 (Figure S.2e) where the grind lines from the 1200 grit polish are present and no grain etching, CIA, or MPB attack are observed. Grind

lines were also observed in DED 316L SS, however there were distinct areas where the difference in the dissolution rates of γ -austenite and δ -ferrite revealed the two phases (Figure S.2c1 and c2). However, visually the extent of the attack appears minimal in comparison to the dissolved δ -ferrite observed in Figure S.1c1 and c2.



Wrought 2205 stainless steel shows that dual phase microstructure is susceptible to selective attack similar to DED 316L. Supplemental experiments were performed on wrought 2205 stainless steel to emphasize the difference in behavior between the austenitic and ferritic phases in a stainless steel alloy containing both phases. The active and transpassive behavior of wrought 2205 in 1 M H_2SO_4 with and without KSCN was investigated through potentiodynamic scanning. Similar to wrought 316, wrought 317L, LPBF, and DED samples. Following polarization SEM and EDS was conducted to understand microstructural attack at the two phase microstructure. Figure S.3 shows the polarization behavior of wrought 2205 in 1 M H_2SO_4 and 0.01M KSCN. When compared to wrought 316L, LPBF 316L, and DED UVA 316L it is apparent that the wrought 2205 has the second fastest anodic kinetics in the active region. A notable region is the active-to-passive region where there are two peaks. Given from the polarization alone, it is unclear if the two peaks are related to the passivation of the ferrite and austenite. When passivation is achieved, the passive current density is similar to DED 316L. in wrought 2205 and DED 316L is similar to the Cr and Mo depleted cellular interiors in LPBF 316L.

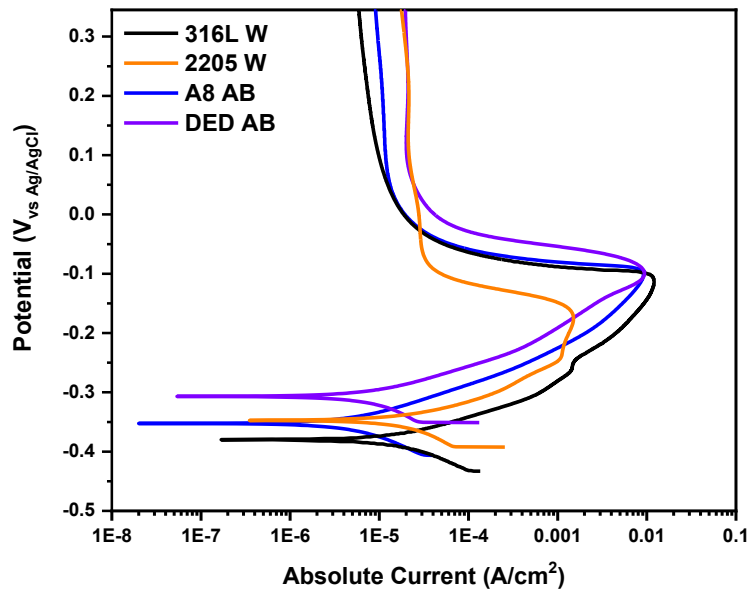


Figure S.3 Polarization scan of wrought 316L, wrought 2205, LPBF A8, and UVA DED in 1 M H₂SO₄ and 0.01 M KSCN.

Wrought 2205 stainless steel austenite phase shows Cr and Mo depletion similar to DED 316L. Post-test SEM micrographs (Figure S.4) confirm preferential attack of one phase over another similar to DED 316L. SEM-EDS confirms Cr and Mo depleted regions underwent preferential attack which would be correlated to the Cr and Mo depleted austenite phase observed in DED 316L. Although LPBF 316L does not have a dual phase microstructure, the preferential attack at the Cr and Mo depleted austenite phase

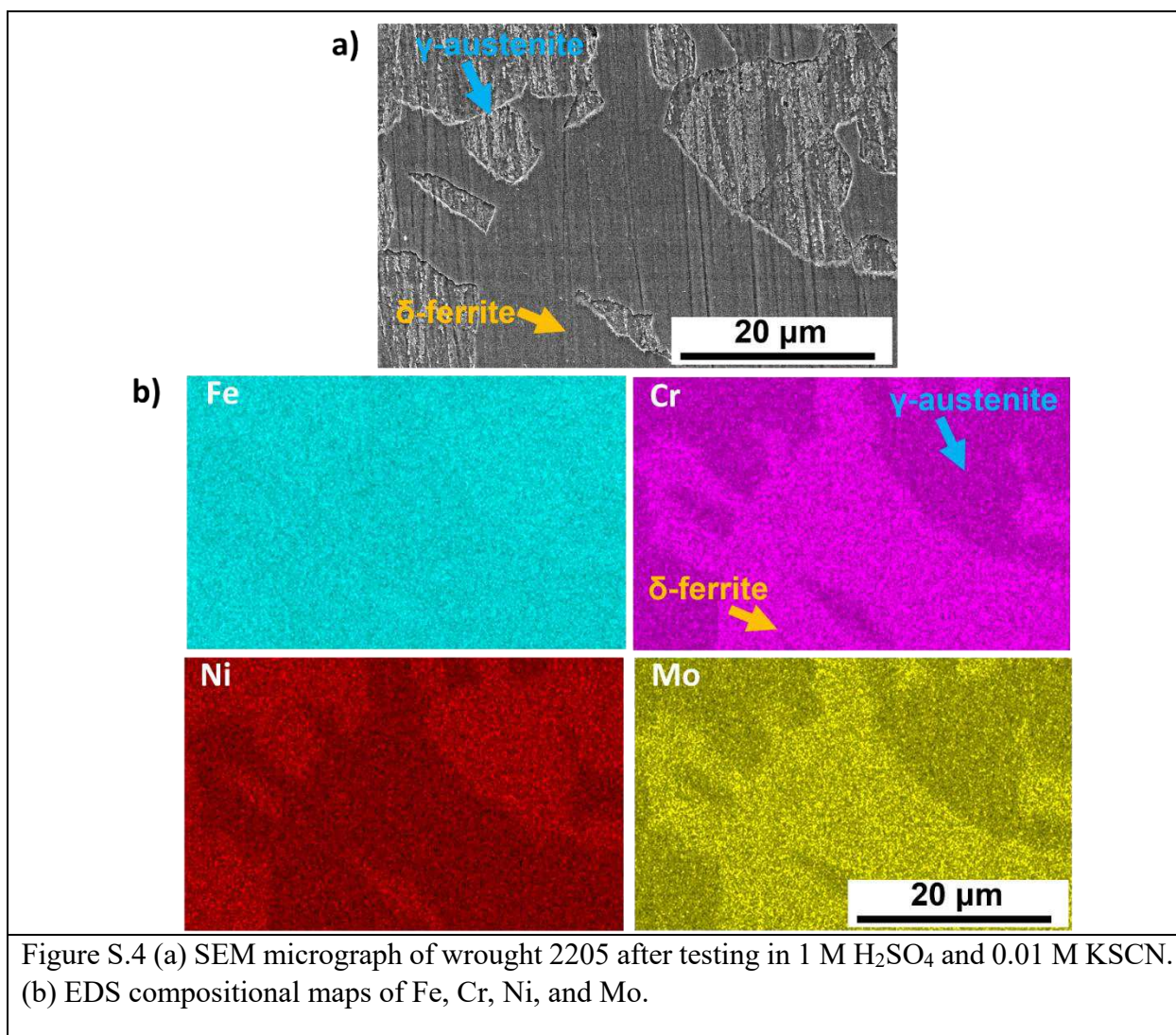


Figure S.4 (a) SEM micrograph of wrought 2205 after testing in 1 M H₂SO₄ and 0.01 M KSCN. (b) EDS compositional maps of Fe, Cr, Ni, and Mo.

High anodic overpotentials demonstrate differences in behavior for wrought 2205 stainless steel than wrought, LPBF, and DED 316L. Figure S.5 shows the transpassive behavior of wrought 2205 in 1M H₂SO₄. When compared to wrought 316L, LPBF 316L, and DED 316L it is apparent that wrought 2205 stainless steel has a higher OCP which impacts the passive current density range ($\sim 0.4V_{vs Ag/AgCl}$ to $\sim 1V_{vs Ag/AgCl}$). However, wrought 2205 stainless steel exhibited spontaneous passivation similar to the other samples but the passive current density was nearly an order of magnitude higher. Because of the increase in the passive current density, the transpassive current densities were greater but this is due to the difference in the passivation observed in the passive region.

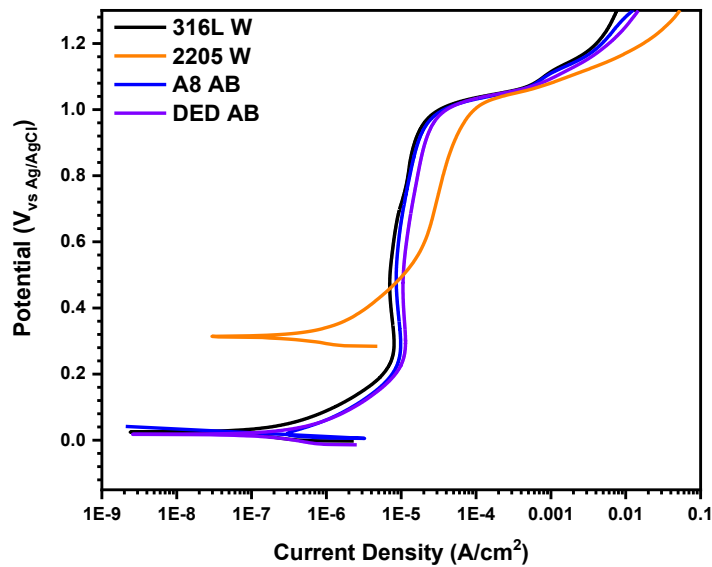


Figure S.5 Polarization scan of wrought 316L, wrought 2205, LPBF A8, and UVA DED in 1 M H₂SO₄.

Transpassive dissolution causes the Cr and Mo rich ferrite phase to dissolve more than austenite in wrought 2205 stainless steel. Post-test SEM micrographs confirm preferential attack of one phase over another similar to DED 316L as shown in Figure S.6. SEM-EDS confirms Cr and Mo depleted regions underwent preferential attack which would be correlated to the Cr and Mo depleted austenite phase observed in DED 316L. Although LPBF 316L does not have a dual phase microstructure, the preferential attack at the Cr and Mo depleted austenite phase is similar to the Cr and Mo depleted cellular interiors.

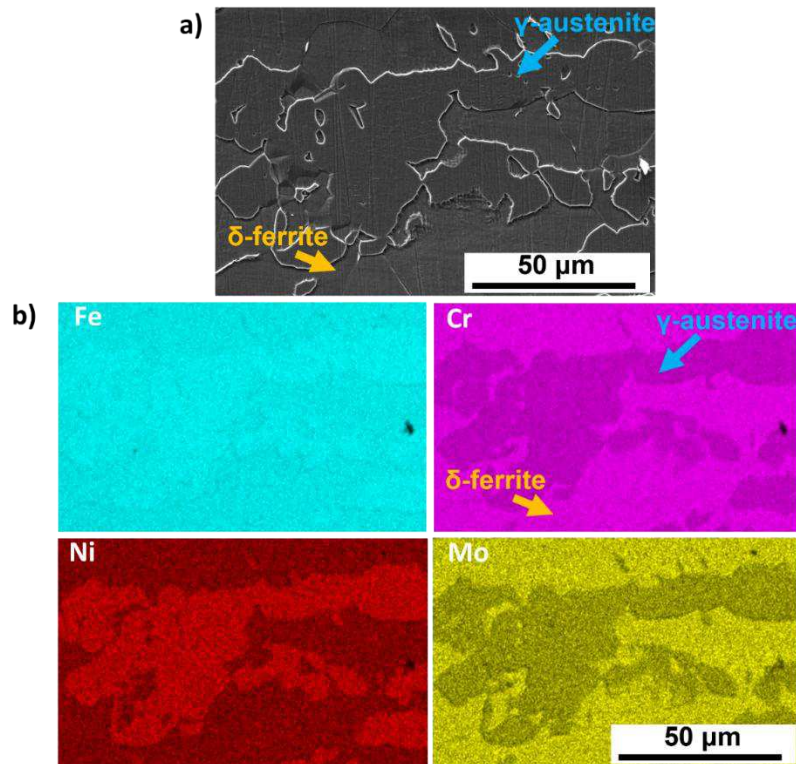


Figure #. (a) SEM micrograph of wrought 2205 after testing in 1 M H₂SO₄. (b) EDS compositional maps of Fe, Cr, Ni, and Mo, .

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