Investigation of Pitting Corrosion Initiation and Propagation of a Type 316L Stainless Steel Manufactured by the Direct Metal Laser Sintering Process

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Direct metal laser sintering (DMLS) is an additive manufacturing process that utilizes a laser to sinter powdered metal to make geometrically complex parts. However, components made by DMLS are believed to be more vulnerable to corrosion due to the presence of residual porosity, as well as laser-induced microstructural deformations. This research focuses on the evaluation of the pitting corrosion resistance of Type 316L stainless steel manufactured using DMLS. The microstructure of the DMLS samples was also compared to specimens annealed to eliminate laser induced scan tracks. Profilometry, compositional analysis, and quantification of the corrosion resistance were performed, before and after the corrosion pitting resistance test, per ASTM G48 Method A (ferric chloride pitting test).

KEY WORDS: ASTM G48, ASTM G5, direct metal laser sintering (DMLS), pitting corrosion, stainless steel

INTRODUCTION

Additive manufacturing using selective laser melting is a relatively new and advanced process for making geometrically elaborate metallic parts, utilizing output from computer-aided design (CAD) programs.[1-2] Each variant of this manufacturing process has, at its heart, a layer-by-layer forming approach; each layer is ca. 20 μm in thickness. The layers are composed of powdered metal sintered using powder bed fusion (PBF) methods, such as by direct metal laser sintering (DMLS).[1] The DMLS technique uses a laser to increase the temperature of the metal powder and induce sintering.[1] The DMLS process has significant advantages for production, such as reducing the amount of base material required to produce a piece, decreasing the time of manufacturing, and increasing precision relating to part creation.[1-2] However, these advantages can potentially be lost if the mechanical properties and corrosion resistance of the final product are compromised. Porosity, scan tracks, and an inhomogeneous microstructure can be present in the final product,[1-3] as the manufacturing process involves the coalescence of the metal powder by heating and rapid cooling,[1] as shown in Figure 1. The resistance to corrosion is a crucial factor for potential application of products manufactured by additive manufacturing processes.[4-5] Consequently, the current research is focused on assessing the pitting corrosion resistance of a Type 316L stainless steel (SS; UNS S31603[(1)]) made by DMLS.

EXPERIMENTAL PROCEDURES

DMLS 316L SS specimens (Fe-65.3, C-0.007, Cr 17.61, Mo 2.68, Ni 12.1, wt%) were tested and compared to an as-received commercial cold-rolled 316L SS (Fe-68.6, C-0.01, Cr 16.96, Mo 2.02, Ni 10.22, wt%). Some specimens were solution annealed at 1,100°C in an Ar atmosphere to study the effect of scan tracks,[1-3,6-7] which are microstructural defects inherent to the DMLS process. Anodic corrosion behavior of the specimens was studied by performing anodic potentiodynamic polarizations in a typical three-electrode glass cell with a solution of 1N H_2SO_4 per ASTM G5[8] (scan rate of 0.1667 mV/s from −20 mV with respect to the open-circuit potential up to 1.2 $V_{Ag/AgCl}$ reference electrode). Pitting corrosion resistance was evaluated per ASTM G48.[9] A 6 wt% $FeCl_3$ solution was utilized to simulate an aggressive environment to which each sample type (rolled, rolled with heat treatment, DMLS, and DMLS with heat treatment) was exposed.

RESULTS AND DISCUSSION

3.1 | Electrochemical Measurements

Figure 2 shows anodic potentiodynamic polarization curves for the rolled and DMLS 316L SS specimens for various conditions; namely as-received (a) and heat-treated (HT) (b). In the first plot (Figure 2[a]), the anodic behavior of the DMLS specimen is compared to the anodic behavior of the rolled

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(1) UNS numbers are listed in *Metals and Alloys in the Unified Numbering System*, published by the Society of Automotive Engineers (SAE International) and cosponsored by ASTM International.

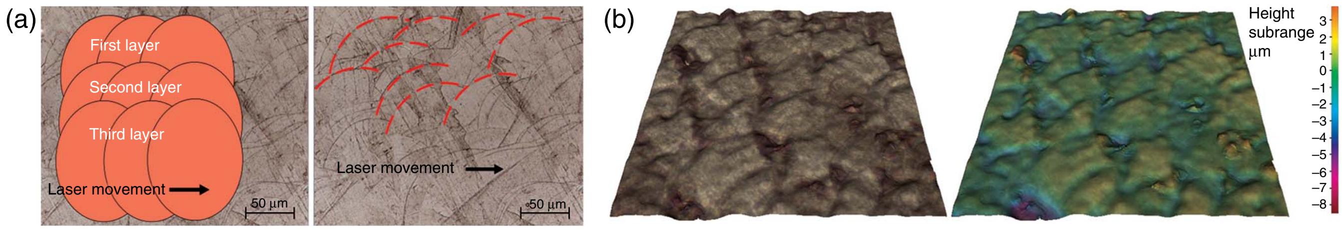


FIGURE 1. *(a) Scan track formation during manufacturing process. (b) 3D profilometry of the scan tracks.*

material. Despite the passivation zone observed in the same potential region for both as-received (DMLS and rolled 316L before HT) specimens, the passive current density of the DMLS 316L SS is almost 8 times higher than for the rolled material. This result suggests that even though the DMLS 316L SS displays a similar passive behavior compared to the rolled material, the corrosion resistance of its passive layer is much weaker. In the second plot (Figure 2[b]), the effect of the heat treatment is shown. The passive current density of the rolled 316L SS specimen decreased, while it did not exhibit a significant change for the DMLS material. Moreover, Figure 3 shows that the heat treatment annihilated scan tracks, suggesting that such defects may not have a significant effect on the corrosion properties of the passive film.

3.2 | Corrosion Initiation Mechanisms (Ferric Chloride Corrosion Test)

In order to obtain a qualitative analysis of the corrosion initiation mechanism for the DMLS 316L SS, a ferric chloride corrosion test was performed in 6 wt% aqueous $FeCl_3$ at 55°C with a specimen exposure duration of 2 d. Microscopic characterization of the 316L SS specimens after the corrosion test provided key information about the mechanisms of corrosion and the detected pitting morphology. Figure 4 shows the surfaces of the DMLS 316L SS specimens after the corrosion test. The images show that there was not a well-defined pattern for corrosion in the rolled material, whereas the corrosion seems to be preferentially initiated on microstructural defects (scan tracks) in the DMLS specimens. Moreover, the pits in the DMLS

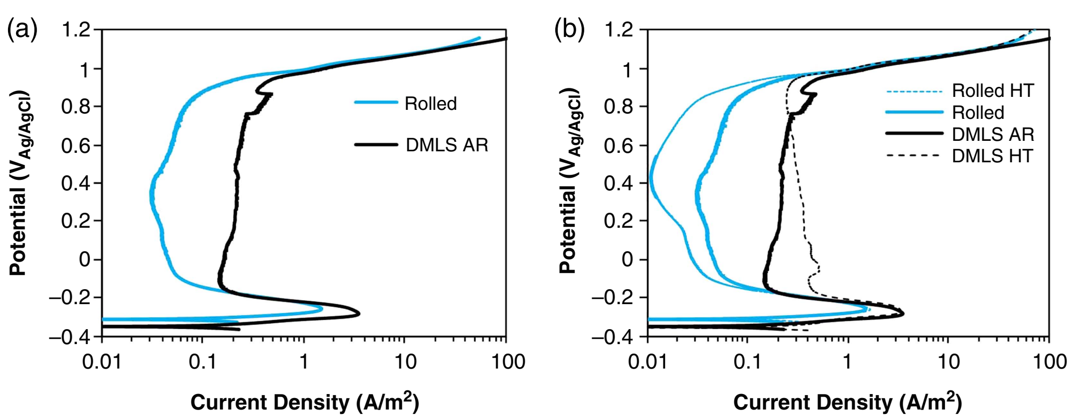


FIGURE 2. *Anodic behavior of the 316L SS specimens. (a) Rolled as-received (AR) and DMLS AR. (b) Rolled AR and heat-treated (HT), DMLS AR and DMLS HT.*

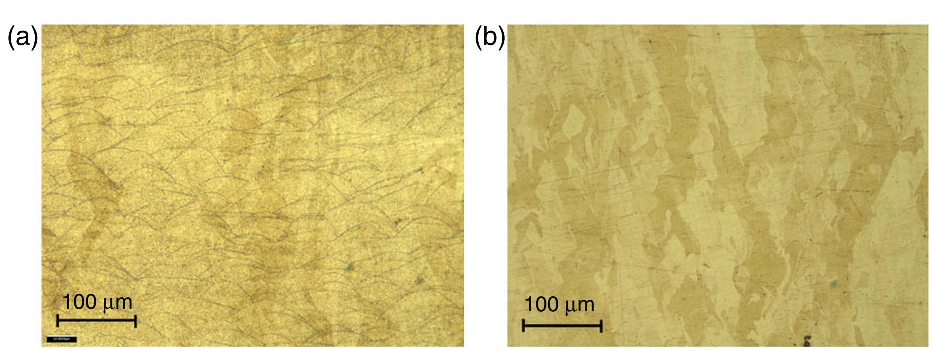


FIGURE 3. *Heat treatment effect on a DMLS 316L SS specimen: (a) as-received material, and (b) heat-treated material. In the second microstructure, the scan tracks disappeared as an effect of the heat treatment.*

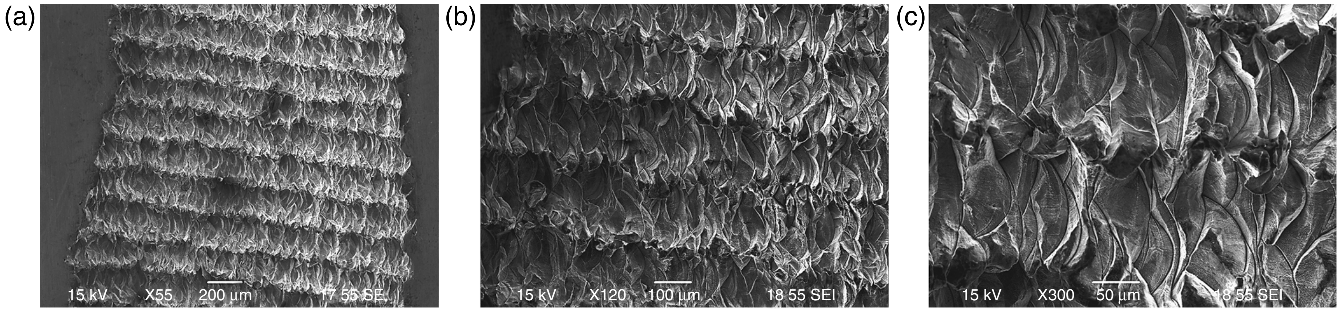


FIGURE 4. *General corrosion of the DMLS 316L stainless steel material: (a) general appearance; (b) and (c) magnification of the affected zones.*

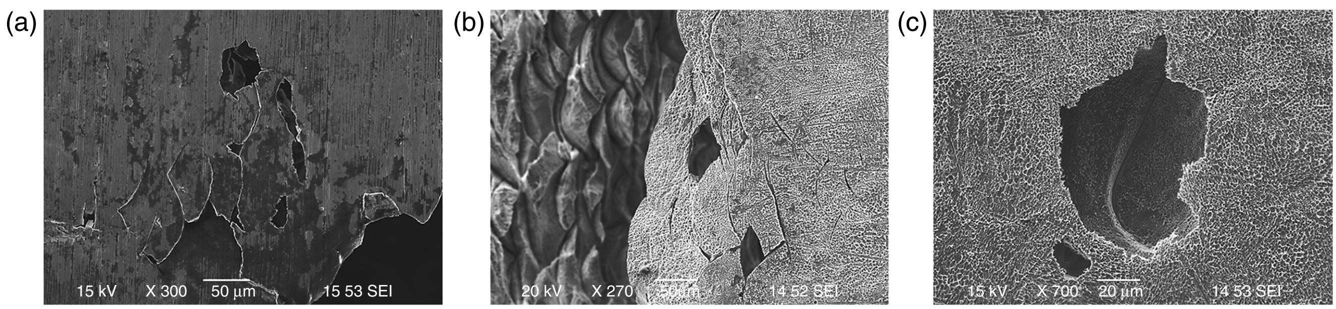


FIGURE 5. *General corrosion and pit morphology of the DMLS 316L stainless steel material: (a) general appearance; (b) and (c) pit morphology.*

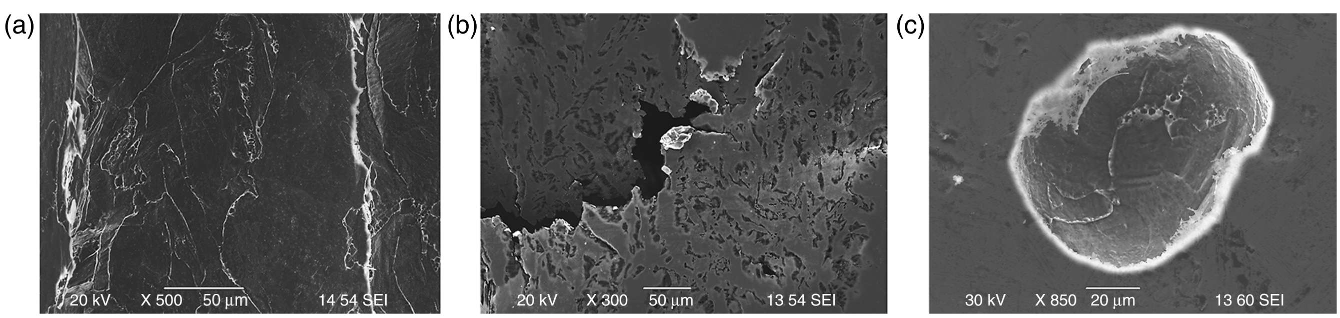
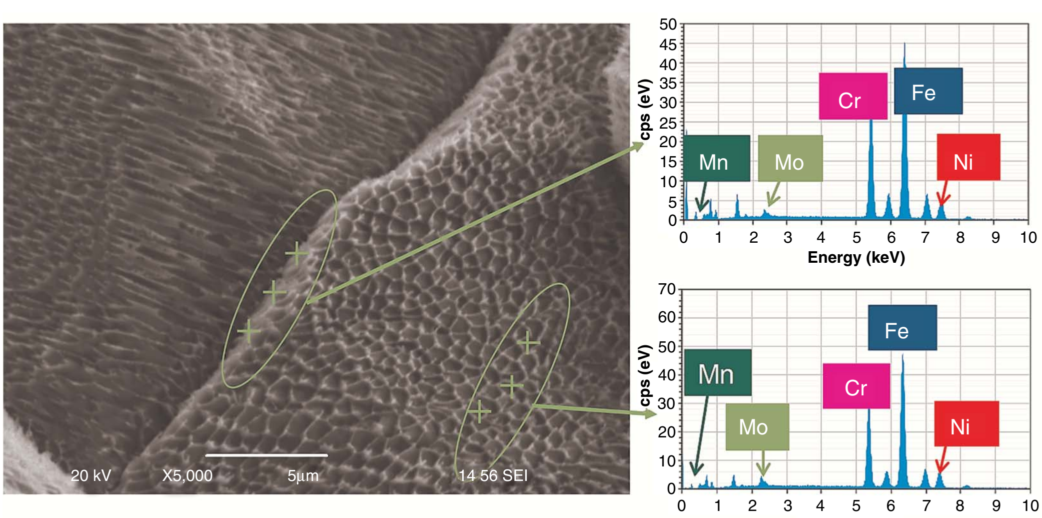


FIGURE 6. *General corrosion of the DMLS 316L stainless steel specimens following heat treatment: (a) general appearance, (b) corrosion initiation, and (c) pit morphology.*



Element	Atom (%)
Oxygen	1.64 ± 0.84
Chromium	28.96 ± 2.86
Manganese	5.83 ± 0.86
Iron	50.77 ± 5.13
Nickel	1.57 ± 0.52
Molybdenum	1.71 ± 0.43

Element	Atom (%)
Oxygen	1.43 ± 0.21
Chromium	22.38 ± 1.38
Manganese	4.98 ± 0.47
Iron	44.40 ± 2.78
Nickel	3.96 ± 0.56
Molybdenum	2.78 ± 0.81

FIGURE 7. *EDS analyses of dark and light phases adjacent to a scan track boundary.*

316L SS specimens showed an irregular pattern associated with scan tracks of the material as shown in Figure 5.

3.3 | Effect of Heat Treatment on Corrosion Behavior of DMLS Specimens

Heat treatment was hypothesized to restore the corrosion behavior of the DMLS steel. Such hypothesis was tested by performing the ferric chloride corrosion test on heat-treated specimens. The general morphology of the corroded surface of the DMLS 316L SS specimens after heat treatment was characterized by scanning electron microscope (SEM), as shown in Figure 6(a). Figure 6(b) shows that the corrosion did not follow any obvious pattern associated with scan tracks. This also suggests that the heat treatment diminished the susceptibility of DMLS material to suffer from preferential corrosion on scan tracks, as compared to the as-received material. Finally, the pit morphology of the heat-treated DMLS 316L SS specimen seems to follow a spherical pattern, confirming that the corrosion is not preferentially initiated on scan tracks as shown in Figure 6(c).

3.4 | Chemical Segregation of Alloying Elements

Another feasible explanation for the preferential corrosion of the DMLS is the microsegregation of alloying elements. Microsegregation of alloying elements, in particular molybdenum, has been reported as a problem relating to the corrosion resistance of superaustenitic stainless steel welds.[10-13] Because the DMLS manufacturing process bears a certain similarity to welding (rapid melting and solidification of metal used to coalesce material), and the fact that the SEM shown in Figure 7 shows the presence of dark and light areas, the presence of microsegregation of elements can be postulated to occur within the interdendritic structure.[10] Therefore, microsegregation of elements can be hypothesized to occur within the dendritic structure of the DMLS specimens, causing preferential localized corrosion in DMLS specimens. The energy dispersive x-ray spectroscopy (EDS) data shown in Figure 7 correspond to a DMLS 316L SS etched sample. EDS analysis was performed on the dark and light phases of a zone contiguous to a scan track. These analyses show that some elements are present at different concentrations within the phases. Thereby, the microsegregation of elements might play a significant role in the preferential corrosion of the DMLS specimens.

CONCLUSIONS

➢ Type 316L stainless steel specimens made by direct metal laser sintering (DMLS) corroded preferentially through the microstructural defects inherent to the manufacturing process (scan tracks).

➢ The preferential corrosion can be attributed to voids and porosity on the surface due to the sintering process as well as chemical segregation within the boundaries of the scan tracks.

➢ Heat treatment did not significantly affect the corrosion behavior of the DMLS 316L stainless steel. However, it reduced the presence of microstructural defects (scan tracks) in DMLS specimens. Such a condition changed the corrosion damage patterns and the morphology of pits formed.

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