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On the enhanced corrosion resistance of a selective laser melted austenitic stainless steel



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ABSTRACT

Corrosion of stainless steels, including the austenitic Fe–Cr–Ni alloys, is associated with MnS inclusions that provide local sites for corrosion pits. Much research effort has been focused on the role of MnS in corrosion of conventional (cast and wrought) stainless steel to date, including microanalysis of MnS and the surrounding microchemistry. Herein, it was elucidated that an austenitic stainless steel (type 316L) when manufactured through selective laser melting was able to be produced without MnS inclusions and hence with no accompanying Cr-depletion in the vicinity of MnS, resulting in superior corrosion resistance relative to wrought form.

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Austenitic stainless steels (SS) are an important class of corrosion resistant alloys with broad industrial use. Corrosion resistance of stainless steels is the result of alloying beyond a critical chromium content ($> \sim 11$ wt%), which stimulates the formation of a thin protective Cr_2O_3 surface film [1]. However, in spite of their name, stainless steels can be susceptible to pitting corrosion in aqueous environment that are either highly corrosive, or that permit microstructurally driven corrosion [2]. In austenitic stainless steels that are nominally single phase such as 316L (Fe–Cr–Ni–Mo) or 304L (Fe–Cr–Ni), pitting corrosion events are found to initiate in the vicinity of second-phase particles present due to alloy impurities, such as manganese sulphide (MnS) [2–8]. Significant research to date has focused on the role of MnS inclusions and their role on corrosion [2–7]. It has been hypothesized that the MnS itself (being void of a Cr-containing passive film) [4–6], or the localized depletion of Cr in the vicinity of MnS (whereby local Cr concentration may be below the critical threshold for the formation of a protective surface film) is responsible for pitting in stainless steels [2].

Sulphur (S) is a persistent impurity in steel making and the formation of low melting point iron sulphide (FeS) inclusions along grain boundaries may cause cracking problems during hot-rolling [9]. As a result, manganese (Mn) is typically added in conventional steelmaking to

counter FeS formation by segregating S as the higher melting point (~ 1655 °C) MnS, which is thermodynamically more stable than FeS [9]. The oxidation of the MnS inclusions during pit initiation will result in the formation of elemental sulphur (S) beside other S-containing species including sulphides (S^{2-}), hydrosulphides (HS^-) and even thiosulphates ($\text{S}_2\text{O}_3^{2-}$) [10–16]. This will eventually result in the formation of a sulphur “crust” at such pit initiation sites [3,17]. A local environment is soon manifest in the sulphur crust which is concentrated in chloride and sulphide species that prevent the re-growth of the passive film, resulting in pit propagation [2,3]. The characteristics of MnS inclusions (e.g. composition, density and size) therefore play a major role in the pit initiation and propagation in stainless steels [3,12]. On this basis, the inhibition of pitting in stainless steels could practically be achieved by eliminating such MnS inclusions or restricting their sizes and distribution.

Austenitic stainless steels are typically prepared for service in either cast or wrought form, however additive manufacturing (AM) has emerged as a technique to manufacture net shape SS components [18–20]. The microstructure of AM austenitic SS has recently been commonly reported [20–25], however corrosion studies are more limited [26–30], in particular corrosion studies that relate performance to detailed micro/nanostructure analysis are yet to be reported. A recent study [30] has revealed that the pitting resistance of 316L manufactured using selective laser melting (SLM) was much higher than that of wrought 316L, independent of the SLM processing parameters. However,

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the origin of such superior corrosion resistance has not been clarified. Considering the inherent rapid solidification rates involved in AM it can be posited the density and size of the MnS inclusions may be reduced. This may be somewhat analogous to laser surface melting where Stewart and Williams [31] and Carnoni et al. [32] have shown significantly improved pitting behavior of wrought 304L and 316L, respectively. They attributed this to the solutionizing of MnS by local melting and rapid quenching. In the present study we seek to clarify the relationship between microstructural features (inclusion chemistry and size, and local elemental segregation) and the pitting behavior in additively manufactured 316L SS.

The microstructure and the electrochemical characteristics of both wrought and SLM 316L SS, with comparable chemical compositions (see Table 1) were studied herein. Wrought 316L SS was supplied as annealed plate (6 mm thick) by Bahru Stainless (Malaysia). The SLM 316L SS was produced using a SLM 125 HL metal printer (SLM Solutions, Germany) equipped with a 400 W IPG fire laser. Specimens (X:Y:Z = 30:30:90 mm) were printed using 10–45 μm 316L metal powder (SLM Solutions, Germany) under a high purity argon atmosphere using a laser power of 175 W, laser scan speed of 730 mm/s, laser diameter of 200 μm , laser focal offset distance of 2 mm, hatch distance of 120 μm and a recoat thickness of 30 μm . Samples of SLM 316L were annealed at 1100°C for 5 min in a muffle furnace under an argon atmosphere to relieve stresses. All characterization and testing was performed on material taken from a plane perpendicular to the SLM build direction (BD), and on the normal plane surface of the wrought material.

Cyclic potentiodynamic polarization (CPP) in 0.6 M NaCl was conducted at 25 °C. Specimens were flush mounted in epoxy and metallographically prepared to a 2000 grit SiC finish. For CPP testing, scans were executed at 1 mV/s from -0.2 V vs the open circuit potential to a current density of 10 mA/cm², after which the scan direction was then reversed. A three electrode flat-cell was used for electrochemical testing, employing a reference saturated calomel electrode (SCE) and a platinum mesh counter electrode. Three replicate tests were performed for each condition.

Microstructural characterization was performed at multiple scales using scanning and transmission electron microscopy (SEM and TEM), atom probe tomography (APT) and X-ray diffraction (XRD). Samples for SEM examination were prepared to a 0.04 μm colloidal silica finish. SEM analysis was conducted at 20 kV using a JEOL JSM 7800F field emission gun (FEG) SEM equipped with an electron backscatter diffraction (EBSD) detector automated by AZtecHKL software (Oxford Instruments). TEM foils were prepared by punching and grinding 3 mm discs to ~ 50 μm thickness and the final thinning to perforation was achieved using a Gatan precision ion polishing system (PIPS, Model 691) at 5 kV. TEM examination was performed at 200 kV on a JEOL JEM 2100F FEG-TEM equipped with a JEOL JED-2300T energy dispersive X-ray Spectrometer (EDS). Specimens for APT analysis were prepared using focused ion beam (FIB) milling and in-situ lift-out method on a dual-beam FIB-SEM instrument (FEI Quanta 3D FEG). The APT needle specimens were analyzed using a Local Electrode Atom Probe (LEAP) 4000 HR instrument (CAMECA Instruments Inc.) operating in voltage mode under ultra-high vacuum at a set-point temperature of 60 K, a pulse fraction of 20%, pulse repetition rate of 200 kHz, and detection rate of 0.005 atoms per pulse. The APT data was topographically reconstructed and visualized using IVAS software. Residual stress was measured by the $\sin^2(\Psi)$ method using a PANalytical X'Pert PRO MRD (XL) X-ray diffractometer with Cu K α radiation, operated at 40 kV and 30 mA.

Stress was derived by measuring the inter-planar spacing of (311) austenite lattice plane as a function of $\sin^2(\Psi)$ [33].

XRD analysis and SEM EBSD imaging (Fig. 1a–c) revealed the as-printed SLM 316L SS to be a fully austenitic single-phase structure free from ferritic δ or σ phases, consistent with prior studies [25,27,30]. The as-printed 316L had a predominance of high angle grain boundaries (HAGBs) and a mean grain size of ~ 8.1 μm , whilst a small proportion of low angle grain boundaries (LAGBs) and 60° $\langle 111 \rangle$ $\Sigma 3$ twin boundaries were also detected (Fig. 1c). High-magnification backscattered electron (BSE) imaging revealed the presence of a cellular sub-structure network (cell size of 200–500 nm) throughout the microstructure (Fig. 1b). Such cell walls have been reported previously and suggested to be dislocation tangles accompanied by Mo/Cr segregation [24,25,27] – a notion that is clarified herein by high resolution characterization. In addition, spherical nano-sized particles (< 50 nm) were also observed (Fig. 1b) to be randomly dispersed through the microstructure, as also reported in [24,25]. A short annealing treatment of as-printed SLM 316L SS at 1100 °C for 5 min resulted in the annihilation of substructure.

The wrought 316L SS displayed a typical recrystallized microstructure composed of equiaxed grains with a mean size of ~ 12.1 μm , however with a higher fraction of $\Sigma 3$ annealing twin boundaries ($\sim 37\%$) and a lower fraction of LAGBs (Fig. 1f–h). The wrought alloy was also free from δ ferrite and σ phase, but comprised relatively coarser spherical or irregular shaped particles in the size range of ~ 0.2 – 2 μm , locally isolated near austenite grain boundaries (Fig. 1f, g). Assessment of residual stress by XRD revealed that the SLM and wrought 316L had compressive normal stress of 248 ± 6 and 120 ± 21 MPa, respectively. The development of compressive residual stress is common in the interior of additively manufactured alloys [19,34,35] and previously postulated to be beneficial for pitting resistance in SS [36,37]. A significant stress relieving of $\sim 90\%$ was achieved by the short annealing of SLM 316L SS.

The CPP responses of as-printed, annealed SLM 316L, and wrought 316L specimens (in 0.6 M NaCl) are shown in Fig. 2. The wrought 316L alloy displayed the characteristic passive window as revealed by potentiodynamic polarization, and an average pitting potential (E_{pit}) of $0.23 \pm 0.03 V_{\text{SCE}}$, in close agreement with studies of wrought 316L SS in similar conditions [30,38]. The pitting potential (E_{pit}) is herein defined as the point of inflection in the anodic polarization curve, where the anodic current density rapidly surges from around 10^{-6} – 10^{-5} A/cm² to $> 10^{-4}$ A/cm² within a few mV of anodic polarization.

In contrast, the as-printed SLM 316L SS displayed a significantly wider passive window than wrought 316L SS, in addition to metastable pitting behavior (i.e. minor current fluctuations) for polarization beyond $\sim 0.5 V_{\text{SCE}}$. The intensity and frequency of such current fluctuations nominally associated with metastable pitting [39,40] are noted to be most prominent at potentials significantly greater than the E_{pit} and breakdown of wrought 316L SS. A comparatively (very) high pitting potential of $0.74 \pm 0.02 V_{\text{SCE}}$ was determined for the as-printed SLM 316L SS, correlating well with recent work by Sander et al. [30] that studied SLM 316L specimens prepared by a different instrument and starting powder. Short-term annealing of the SLM 316L resulted in a minor reduction in the average E_{pit} to $0.56 \pm 0.07 V_{\text{SCE}}$, which was highly reproducible, and possibly due to the relieving of compressive residual stress. It is noted that such unique differences in the measured E_{pit} for 316L SS of the same bulk composition, whilst reported, have not been previously clarified. The present work seeks to focus on the deterministic rationale for the large difference in E_{pit} between wrought and SLM 316L (where bulk chemical composition and grain size are comparable), whilst the

Table 1
Chemical compositions of the bulk as-printed (SLM) and wrought 316L stainless steels

Element (wt%)	C	Si	Mn	P	S	Cr	Mo	Ni	Nb	Ti	Al	Cu	N	O	Fe
As-printed	0.02	0.59	1.23	0.01	0.01	16.9	2.55	11.8	0.01	0.01	0.01	0.08	0.037	0.036	Bal
Wrought	0.02	0.45	1.39	0.03	0.01	16.5	2.01	10.2	0.01	0.01	0.01	0.38	0.053	0.006	Bal

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