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Pitting and Crevice Corrosion Resistance of a Direct Metal Laser Sintered (DMLS) 316L Stainless Steel in Artificial Seawater

Claudia Prieto, Marc Singer, David Young

Institute for Corrosion and Multiphase Technology
Department of Chemical and Biomolecular Engineering
Ohio University, Athens, OH 45701

ABSTRACT

The use of 2%-Mo containing austenitic stainless steels is a common practice for marine applications, such as for the fabrication of fuel nozzles and impellers. Such geometrically complex parts can be manufactured more efficiently using additive manufacturing techniques, such as the direct metal laser sintering process (DMLS). However, research has revealed that 316-type stainless steels are not entirely exempt from undergoing localized attack. Environmental factors, such as chloride content, temperature and oxygen levels are key governing factors limiting the application of 2%-Mo containing austenitic stainless steels. Moreover, the susceptibility to localized attack for additively manufactured products, such as 316L DMLS, has been postulated to significantly increase due to residual porosity, surface asperity and microstructural defects inherent to the additive manufacturing process. Since the additive manufacturing of geometrically complex parts confers advantages in terms of design, it is essential to determine if their performance against corrosion would compromise their real-world applicability. By using aerated artificial seawater per ASTM D1141 and cyclic potentiodynamic polarization (CPP), the metastable pitting characteristics of a 316L stainless steel manufactured by the DMLS process was characterized. Moreover, the effect of an argon quenched heat treatment was explored. A cold-rolled 316L stainless steel, as-received and heat-treated, was used as a reference for this study. Results indicated that the heat treatment increased the resistance to pitting initiation of the 316L stainless steel made by DMLS as inherent microstructural defects were healed.

Key words: 316L, pitting corrosion, EBSD

INTRODUCTION

Selective laser melting is an additive manufacturing (AM) process capable of making geometrically complex metallic parts utilizing computer-aided design (CAD) programs^{1,2}. The basic principle is the use of a 'layer-by-layer' forming approach, with powdered metal sintered using powder bed fusion methods (PBF)^{1,3}. The direct metal laser sintering (DMLS) technique uses a laser to increase the temperature of the metal powder and induce sintering^{1,4}.

DMLS, as well as other AM processes, has the advantage of precision in manufacturing¹⁻³, which can make it attractive for marine and biomedical applications. Among stainless steels, low carbon 300-series alloys are typical for this type of manufacturing process^{1,3,5-7}; with 316L stainless steel (UNS S31603) the most commonly used^{1,3,5-7}. One significant advantage of 316L stainless steel is its resistance to pitting due to the presence of molybdenum (Mo).⁸ Consequently, this resistance to corrosion is a crucial factor for potential application of products manufactured as DMLS 316L stainless steel. As a general rule, AM relies on stainless steel to avoid corrosion issues.^{1,7}

Nonetheless, the absolute corrosion resistance of DMLS 316L stainless steel cannot be assumed. There are factors such as metallurgy, dislocation density and surface morphology can have an impact on the corrosion resistance of a stainless steel. For example, Peguet, *et al.*⁹, reported that the addition of alloy elements might affect the stability of the austenite thereby affecting the pitting resistance of a stainless steel; Geng, *et al.*¹⁰, reported that the microstructure of the surface layer of a passivate stainless steel can be affected by the increase of the formation of an α' -martensite phase as well as the increase of dislocation density. Regarding the heat treatment, it is well understood that depletion of elements (such as chromium) and the formation of some deleterious phases can happen if the diminish the performance of the corrosion resistance of stainless steels, such as carbides and sigma ferrite (σ -Fe)¹¹⁻¹³.

Therefore, the inherent defects of the parts due to the manufacturing process need to be considered. Porosity, microscopic surface defects (scan tracks and melting pools) and dendritic microstructure can be present in the final product^{1-4,14}, and play a significant role in corrosion resistance. Therefore, the current research is focused on assessing the pitting and crevice corrosion resistance of a DMLS 316L stainless steel in artificial seawater.

EXPERIMENTAL PROCEDURE

Electrochemical Measurements

The experimental apparatus was a typical 3-electrode setup in a 2L glass cell, as shown in Figure 1. The 316L stainless steel cold-rolled specimens were used as working electrodes. The specimens were cut to squares with a working area of 1 cm². The 316L DMLS specimens were manufactured with a 400 W laser at a volume rate of 2 mm³/s, a

spot size of 50 μm of radius, and a layer thickness of 20 μm . Samples were flush-mounted in epoxy and lacquer was used on the interface between edges to avoid crevice initiation.^{15,16} Table 1 shows the chemical composition of the specimens tested.

Table 1
Chemical composition (wt.%) of the 316L SS samples (as-rolled and DMLS)

	Al	C	Cr	Cu	Mn	Mo	N	Nb	Ni	P	S	Si	Ti	W	Fe
Rolled	0.011	0.01	16.96	0.41	1.23	2.02	0.04	0.014	10.22	0.032	0.006	0.36	0.013	0.05	balance
DMLS	0.007	0.007	17.61	0.21	1.64	2.68	0.07	0.01	12.1	0.017	0.008	0.26	0.035	0.024	balance

The steel specimens were sequentially polished with 150, 400, and 600 grit silicon carbide paper. They were then rinsed with isopropanol and sonicated for 5 minutes to remove any debris from polishing. A platinum-coated titanium mesh was used as the counter electrode. A saturated Ag/AgCl electrode was used as the reference electrode connected via a Luggin capillary. The working electrolyte was an artificial seawater brine¹⁷ with the composition shown in Table 2.

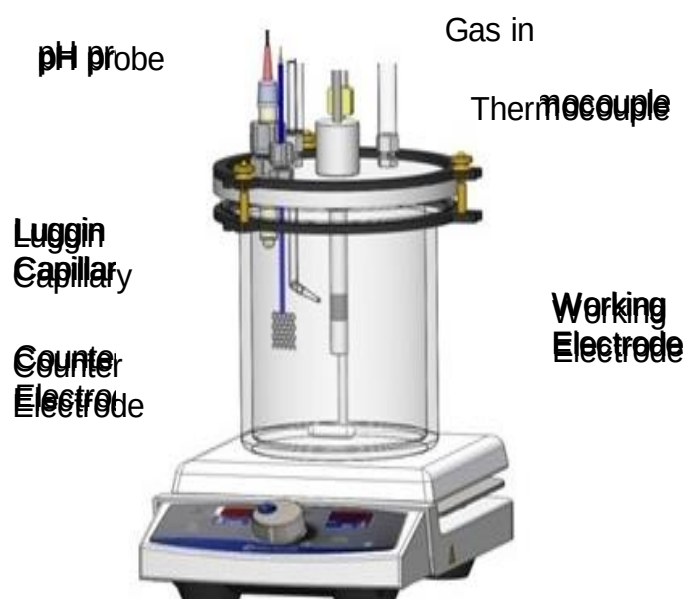


Figure 1: Three electrode glass cell setup to perform electrochemical measurements.

Table 2
Artificial seawater composition

Compound	Concentration, g/L
NaCl	24.53
MgCl ₂	5.2
Na ₂ SO ₄	4.09
CaCl ₂	1.16
KCl	0.695
NaHCO ₃	0.201
KBr	0.101
H ₃ BO ₃	0.027
SrCl ₂	0.025
NaF	0.003

Before each experiment, the working solution was deoxygenated by sparging with nitrogen for 2 hours, pH was then adjusted to 8.2 with the addition of a solution of 0.1 M NaOH. After specimen immersion, the open circuit potential (OCP) was monitored for one hour. Cyclic potentiodynamic polarization¹⁵ was performed starting from -100 mV with respect to the OCP at a forward and backward scan rate of 0.166 mV/s, 50 A/m² (5 mA/cm²) was the apex current set to start the reverse scan. Each experiment was repeated three times.

Heat Treatment

Selected specimens were solution annealed in an argon atmosphere to study the effect of scan tracks^{1-4,14}, which are microstructural defects inherent to the DMLS process. Table 3 shows the conditions of the heat treatment.

Table 3
Heat treatment for annealing

Heat Treatment	Conditions
Annealing in an argon atmosphere	1100°C Soaking time: 45 min in an argon bed at 10 psig (0.68 bar). Argon quenched increasing up to 100 psig (6.89 bar) to achieve a cooling rate of 50 °C/min

Optical microscopy was utilized to show the effect of the heat treatment of the stainless steel in both the DMLS and as-received specimens. Such images can be found in a past paper¹⁸.

Surface Characterization

SEM was used to characterize the crevices and pitting after the electrochemical experiments. Electron backscatter diffraction (EBSD) was performed to determine the effect of heat treatment on the grain size¹⁹. The samples were sequentially polished with 150, 400, and 600 grit silicon carbide abrasive paper. After that, the specimens were polished with 9, 6, 3, and 1 μm diamond suspension. For EBSD, the polishing continued with 0.05 μm alumina suspension then electropolishing was performed to remove the Beilby layer and enhance map acquisition²⁰.

RESULTS AND DISCUSSION

Corrosion Test

Figure 2 shows the cyclic potentiodynamic polarization of the 316L cold-rolled and DMLS as-received specimens. Initially, it was observed that the corrosion potential (E_{corr}) of the DMLS was, on average, 183 mV lower (standard deviation of 12.2 mV) than its cold-rolled counterpart. It was also observed that the transpassive potential (E_T) of the DMLS was reduced by 194 mV on average (standard deviation of 5.7 mV), whereas the repassivation potential was virtually the same with an average difference of 18 mV.

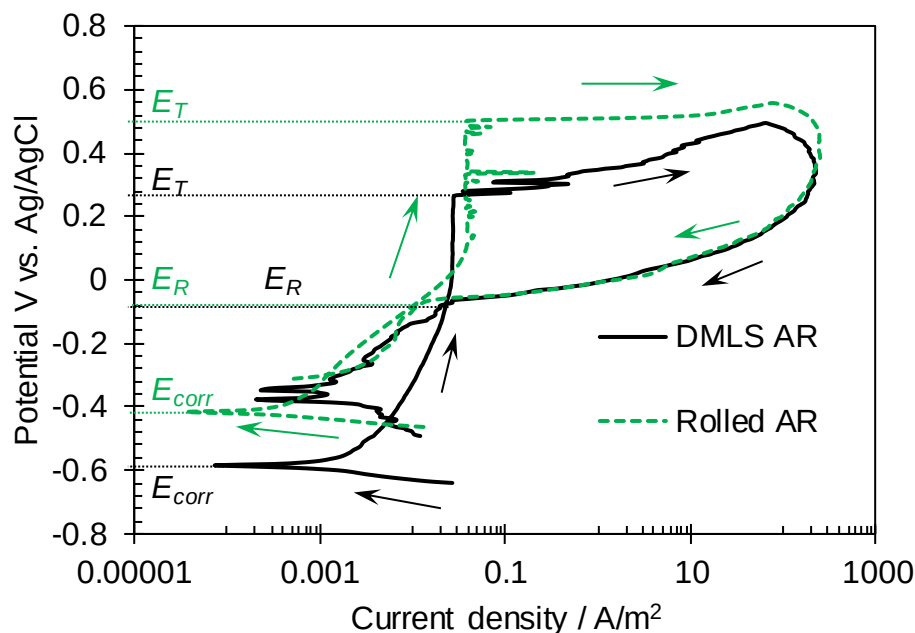


Figure 2: Cyclic potentiodynamic polarization curves for the DMLS and cold-rolled 316L SS. E_{corr} is the corrosion potential; E_T is the transpassive potential, and E_R is the repassivation potential.