TECHNICAL ARTICLE



Effect of Microstructure of Additively Manufactured Inconel 625 on Long-Term Corrosion Behaviour in Sulfuric Acid Media

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The aim of this work was to investigate the effect of the microstructure of Inconel 625 additively manufactured by laser powder bed fusion (LPBF) and laser-assisted directed energy deposition (LDED) on the resistance to corrosion in sulfuric acid solution. Corrosion tests lasted 30 days, 90 days and 180 days. Microstructure characterization was performed using light microscopy, scanning and scanning-transmission electron microscopy. Corrosion resistance measurements were conducted using polarization curves and impedance spectroscopy. After immersion in sulfuric acid, the corrosion pits were observed on the surface. The area fraction of corrosion pits increased with prolongation of the test and after 180 days was equal to 9.63% and 4.49% in LPBF and LDED specimens, respectively. The higher density of the pits occurred along the boundaries of the melt pools, grain boundaries and cell walls and next to precipitates. It was determined that higher drop in impedance and increase in corrosion current occurred in LPBF than LDED Inconel 625, which was mainly related to a more intensive propagation of corrosion pits in areas with higher free energy. Furthermore, the weaker corrosion resistance is related to a more refined microstructure with a higher density of the preferential sites of localized corrosion after long-term corrosion in sulfuric acid.

INTRODUCTION

Inconel 625 is a nickel-based superalloy characterized by high strength at high temperature and excellent corrosion resistance in harmful environments.¹⁻⁵ Due to these properties, it is commonly used in automotive, aeronautic and chemical industries. Unfortunately, high hardness, poor machinability and low thermal conductivity make it difficult to fabricate components with a complex shape.^{2,6} These problems can be solved using additive manufacturing (AM), also known as 3D printing. Laser powder bed fusion (LPBF) process, one of the major additive manufacturing processes, which relies on selective fusing or melting of powdered material layer upon layer using one or more lasers, is commonly used to fabricate Inconel 625 parts. In turn, laser-assisted directed energy deposition (LDED) uses powder or wire feed system to additively manufacture large freeform objects or to regenerate parts made of Inconel 625. During the LDED manufacturing operation the whole system is moving on a multi-axis arm and selectively sintering tracks; thus, this technology is similar to welding. $^{7-9}$ Consequently, the microstructure of AM parts produced by LPBF and LDED is similar to that after welding, and the morphology and size of solidification structure features depends on temperature gradient, solidification rate and undercooling.¹⁰ Furthermore, as in the welding process, the laser-assisted AM processes are generating high amounts of the internal residual stresses, which by applying post-build heat treatment are reduced.¹¹⁻¹⁴ Commonly, the LPBF and LDED manufactured Inconel 625 parts are stress-relief annealed at 870 °C and 980 °C for 1 h or solution treated at a temperature up to 1100 °C for 1 or 2 h.^{9,15–19} The level of internal stress in LPBF samples is higher than in LDED. Therefore, for LPBF samples, a higher stress relief annealing temperature of 980 °C is often used. On the other hand, for LDED samples are stress relief annealed at a lower temperature of 870 °C. In

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LPBF-printed superalloys the molten pools are much smaller than in LDED ones, so the solidification structure is characterized by cellular-dendritic or columnar dendritic morphology and fine grains. In turn, in LDED-printed superalloys, the structure is mostly columnar dendritic or even dendritic and characterized by larger grain size. The common feature of the LPBF and LDED Inconel 625 microstructure is precipitation of the Laves phase and MC carbides in the intercellular areas.^{20,21}

Although Inconel 625 exhibits outstanding longterm corrosion resistance in many aggressive environments, including those containing chloride, phosphate and fluoride ions, its AM varieties may be less resistant than the wrought superalloy, which might be caused by microsegregation of chemical elements, less homogeneous microstructure and presence of intermetallic compounds.^{22–24} Recent studies have shown that the presence of a cellular-dendritic or dendritic structure might be the reason for the formation of galvanic micro-cells between dendritic and interdendritic areas or intermetallic phases under high temperature high pressure H_2S/CO_2 .²²

The aim of the present study is to determine the effect of the microstructure of Inconel 625 fabricated by LPBF and LDED on the long-term corrosion in sulfuric acid. In diluted sulfuric acid (< 10%) the aggressiveness of the acid is limited, so stainless steels can be used. At concentrations 10% and higher, more corrosion resistant material, like Inconel 625, may be required. Here, the microstructural changes caused by corrosion are revealed and correlated with the results of electrochemical measurements after 30 days, 90 days and 180 days in an aggressive environment. The findings provide insight into the effect of cellular-dendritic structure refinement on the corrosion mechanism in additively manufactured Inconel 625 superalloy.

MATERIALS AND METHODS

In this study, the microstructural analysis and electrochemical tests of the LPBF and LDED Inconel 625 specimens were performed. The LPBF process was carried out by means of EOS M290 (EOS GmbH, Germany) machine and EOS NickelAlloy IN625 powder (EOS GmbH, Germany). On the other hand, the LDED process was conducted using the RPMI 557 (RPM Innovations, Inc., USA) machine and IN625 45–125 μ m powder (Höganäs Belgium S.A., Belgium). The chemical composition of both types of specimens, determined by spark source mass spectroscopy, using MiniLab 300 spectrometer (GNR s.r.l, Italy), is given in Table I. The main difference in chemical composition of both variants mainly concerns the content of Fe, Mn, Co, Si and Ti. In LPBF Inconel 625, the concentration of Fe is 1.7% higher than in LDED. Also, the concentration of Ti is slightly higher in the LPBF variant. Meanwhile, the concentration of Co and Si is

slightly lower in LPBF than in LDED for 0.15 and 0.34%, respectively. In both specimens, the carbon concentration is < 0.01%.

In the LPBF process a change in the scan strategy of the deposition layers by 67° relative to the previous layers was applied, while in LDED it was 45° . Additively manufactured LPBF and LDED specimens of cuboidal shape, $15 \times 15 \times 15$ mm in size, were subjected to the stress-relief annealing at a temperature of 980 °C and 870 °C for 1 h, respectively. Subsequently, slices with dimensions of 7.5 mm \times 7.5 mm \times 2 mm were cut from the specimens, grinded by using SiC grit paper (P1000– P2000), polished with diamond suspension and finally cleaned in an ultrasonic washer with distilled water. Afterwards, the specimens were subjected to corrosion tests in 10% sulfuric acid (H₂SO₂) for 30, 90 and 180 days at the room temperature.

The microstructural investigation before and after corrosion tests was carried out by means of light microscopy (LM) and scanning electron microscopy (SEM) using MM100 (Opta-Tech, Poland) LM and InspectS500 (FEI, USA) SEM equipped with an energy dispersive X-ray spectrometer (EDS) Octane Elect Plus (EDAX, Japan). SEM analyses were performed using secondary electron (SE) and backscattered electron (BSE) detectors. The microstructure and chemical composition in the cellular and intercellular areas were examined by means of scanning-transmission electron microscopy (STEM) using Tecnai TF 20 X TWIN microscope (FEI, USA) equipped with a STEM high-angle annular dark-field (HAADF) detector and an EDS r-TEM spectrometer of EDAX. The area fraction of the corrosion pits, A_{A CP}, was determined using SEM BSE images with a total area at least equal to $6800 \ \mu m^2$ applying a systematic manual point counting method. The measurement error was < 0.14%. For each specimen the total number of testing points was at least 4488. The tested specimens were characterized by negligible porosity, so there was no risk of classifying the pores as pits.

The measurements of area fraction of particles A_A were carried out using SEM SE images of at least three randomly selected locations of the samples. The area of each image was equal to 550 μ m². The image analysis was carried out by using ImageJ/Fiji (NIH and LOCI, University of Wisconsin, USA).²⁵

Impedance spectroscopy (EIS) and polarization tests were performed by means of potentiostat/galvanostat Autolab PGSTAT 302N (Methrohm, Switzerland) and Nova 2.0 software. Corrosion measurements were carried out in a three-electrode system, where the working electrode, counter electrode and reference electrode were the tested material, platinum plate and saturated Ag/AgCl electrode, respectively. Corrosion measurements were carried out for each sample in order from EIS to open circuit potential (OCP) and linear polarization in 10% sulfuric acid aerated medium. For each material and incubation time, at least two samples were tested.

Table 1. Chemical composition of LPBF and LDED Incohel 625, wt.%													
Specimen	Elements												
	Ni	Cr	Mo	Nb	Fe	Mn	Со	W	Si	Ti	V	С	Al
LPBF LDED	$\begin{array}{c} 62.46\\ 63.34\end{array}$	$22.38 \\ 22.50$	$8.25 \\ 8.22$	$3.66 \\ 3.63$	$2.53 \\ 0.83$	$\begin{array}{c} 0.09 \\ 0.43 \end{array}$	$0.06\\0.21$	$\begin{array}{c} 0.20 \\ 0.23 \end{array}$	$\begin{array}{c} 0.20 \\ 0.54 \end{array}$	$0.13\\0.04$	< 0.01 < 0.01	< 0.01 < 0.01	0.02 < 0.01

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EIS measurements were performed with scan in the frequency range from 10^5 Hz to 0.1 Hz. The OCP was measured by 10 min to determine the range of the scan in linear polarization. Polarization tests were performed using scan speeds of 0.7 mV/s in the range from -0.4 V to 1.0 V versus OCP, but the anodic part of the curve was limited because of the achievement of the maximum current value set within the measurement range. The corrosion potential (E_{corr}) and the corrosion current (i_{corr}) were determined using the Tafel extrapolation method, while the EIS parameters were indicated using Nova 2.0 software. The current density (j_{corr}) was estimated for the measured area of the sample. The area of the samples was limited by the nonconductive varnish to 0.4–0.6 cm². For each sample, the surface area was determined with $a \pm 5\%$ error.

RESULTS AND DISCUSSION

Figure 1 shows LM and SEM images of the LPBF and LDED Inconel 625. In Fig. 1 a,d, the shape of the melt pools reveals the conductive mode of laserassisted AM. The range of the width of melt pools in the plane parallel to the build direction (BD) was in the range of 40–140 μ m and 600–1400 μ m for LPBF and LDED Inconel 625, respectively. It was noticed, therefore, that in LPBF the width of the melt pools was ten times smaller. LPBF Inconel 625 exhibited a fine cellular structure (Fig. 2a), while in the LDED specimen a coarser cellular-dendritic structure (Fig. 2b), typical for the DED processed alloys, was observed.^{26–30} The area of cells was in the range of 0.1-5.6 μ m² in LPBF and 3.2-107.0 μ m² in LDED. Dispersive Al₂O₃ oxide inclusions were also observed by SEM BSE compositional contrast in both variants and by STEM EDS mapping (Figs. 1c and f and 2). The comparison of the area fraction of oxide inclusions in both materials revealed higher value for LPBF Inconel 625 than in LDED (Fig. 3). The literature reports that such oxides also occur in other AM materials.^{31–33} STEM-EDS microanalysis of both specimen variants revealed the segregation of Mo and Nb to the cell walls, while the content of Cr in the cell walls was lower than in the cell interior (Fig. 2). Other researchers have also reported similar segregation of chemical elements in AM Inconel 625.^{19,34-37} In LPBF and LDED Inconel 625, as a result of Nb and Mo enrichment in the intercellular areas, precipitates of secondary phases occurred as precipitates rich in Nb and Mo located in the intercellular areas (Fig. 1f and 2b). In

LPBF Inconel 625 the Laves phase, MC carbides particles were identified. Their cumulative area fraction was 4.28%.²¹ In turn, in LDED Inconel 625 the MC and $M_{23}C_6$ carbides and the Laves phase were detected.20 Their area fraction was significantly lower than in LPBF Inconel 625 and was equal to 2.86%. Some single particles of the δ phase had plate-like morphology, and A_A equal to 1.06%; $(Cr, Nb)_2N$ nitrides were also present. The graph comparing the area fraction of the precipitates and oxide inclusions that occur in the LPBF and LDED Inconel 625 is given in Fig. 3.

Microstructural analysis after corrosion tests allowed us to observe pitting on the surface of the LPBF and LDED specimens and estimate the area fraction of the corrosion pits. After 30 days, the impact of the sulfuric acid solution on the surface of LPBF Inconel 625 was insignificant (Fig. 4). A small number of pits was observed, the $A_{A CP}$ of which was equal to 1.20%. The pitting started to occur along the grain boundaries and cell walls. This could be expected because of the different electrochemical activity caused by the difference in local chemical composition and higher free energy in these areas.³ However, in LDED specimen after 30 days of testing, the $A_A \ _{CP}$ was $\geq 1.92\%$. This result indicates that the corrosion started faster, which can be attributed to the presence of precipitates, the additional corrosion sites beside grain boundaries and cell walls (Fig. 4). This is consistent with the findings of Carini et al., 38 who noticed that the corrosion initiates at the boundaries of niobium rich precipitates, such as carbides and δ phase, as well as the areas with lower chromium content and the edges of the melt pools. Furthermore, in both materials higher density of pits along the melt pool boundaries was observed. According to the research by Revilla et al.³⁹ the main reason for the initiation of corrosion pits in the melt pool boundaries is the large difference in the electrochemical potential between the boundaries and the interiors of melt pools. It is caused by the high heterogeneity in the microstructure created during the solidification, manifested by the formation of finer grains along melt pool boundaries and thus a higher density of precipitates, which occur along the grain boundaries and in the interdendritic areas.

After 90 days, it was noticed that the formation of corrosion pits accelerated distinctly in the LPBF specimen, while in the LDED it was slightly slower (Fig. 4). The $A_{A CP}$ was equal to 5.70% and 3.14% for

Effect of Microstructure of Additively Manufactured Inconel 625 on Long-Term Corrosion Behaviour in Sulfuric Acid Media



Fig. 1. Microstructure of the (a, b, c) LPBF and (d, e, f) LDED Inconel 625 in the stress-relieved condition, LM and SEM images.

LPBF and LDED, respectively. On the surface of both specimens, corrosion occurred mainly in the interdendritic areas, grain boundaries and the vicinity of precipitates.

Observations of the microstructure after 180 days of the test revealed that the corrosion pits in LPBF Inconel 625 appeared on the entire surface of the specimen, but still the higher density of the pits occurred along the boundaries of the melt pools (Fig. 4). The $A_{A \ CP}$ was equal to 9.63%. Meanwhile, in the LDED specimen pitting corrosion progressed steadily and the $A_{A \ CP}$ was 4.49%, thus significantly lower than in the LPBF. Pitting that occur on the grain boundaries started to overlap, which caused an accelerated corrosion at these locations (Fig. 4).

OCP for LDED and LPBF samples after 24 h was equal to 0.009 V and 0.002 V, respectively. A gradual change of OCP for LPBF was observed after 30 days, 90 days and 180 days to -0.071 V, -0.132 V and -0.220 V, respectively. For LDED Inconel 625 the change in OCP was faster and the potential values were close to each other after these times and were equal to -0.183 V, -0.214 V and -0.215 V, respectively. For both materials, the OCP after 180 days was almost the same, which proves the similar corrosion behavior of LPBF and

LDED Inconel 625, but it can be concluded that initial corrosion changes occurred faster on the surface of LDED than LPBF because of the initiation of corrosion at the boundaries of niobium-rich precipitates.

Figure 5 shows the potentiodynamic polarization curves and EIS diagram of the LPBF and LDED specimens after corrosion tests. The electrochemical parameters obtained from each potentiodynamic polarization curve, such as $E_{\rm corr}$ and $j_{\rm corr}$, as well as the equivalent value of the circuit elements are presented in Table II. The proposed circuit replacement model is shown in Fig. 5c and d, for which R_1/CPE_1 (resistance/capacity) corresponds to the passive surface, and R_2/CPE_2 (resistance/capacity of the metal/oxide interface) could be related to the corroded area.

In the polarization curves of both AM Inconel 625 specimens, an $E_{\rm corr}$ in range of -0.147 V to -0.217 V was observed. All $E_{\rm corr}$ values were in similar range, but a there was a small decrease in $E_{\rm corr}$ after 180 days for both LPBF and LDED Inconel 625. The corrosion potential is sensitive to the local chemical composition of material and corrosion solution, but the observations imply that a passive layer and corrosion products are



Fig. 2. SEM and STEM-HAADF images of the microstructure together with STEM-EDS maps of chemical elements distribution in (a) cellular structure of LPBF and (b) the dendritic cellular-dendritic structure of LDED Inconel 625 in the stress-relieved condition.

dynamically forming on the surface, which can cause fluctuation in the potential value. The character of the EIS diagrams of the specimens tested for 30 days was typical of that for corroded metallic materials with the ability to create a passive layer.⁴⁰ The EIS spectra consisted of two semicircles, which were related to the passive layer and the metal corrosion products of Inconel 625. The character of the EIS spectrum for LPBF and LDED specimens after 30 days corresponded to a low defected surface with a compact passive layer, while after 90 days and 180 days the impedance response is due to corrosion products and pits.

The EIS measurements show a decrease in the Inconel 625 impedance with the prolongation of the corrosion time for both LPBF and LDED-printed variants. The change of frequency-dependent impedance character in the medium- and low-frequency regions with a prolongation of time in sulfuric acid



Fig. 3. Area fraction of the precipitates and oxide inclusions that occur in the LPBF²¹ and LDED Inconel 625.

was observed. This can be explained by the increase in the area fraction of the corrosion pits,⁴¹ which was confirmed by SEM investigations.

From the results of the EIS and polarization tests, it can be concluded that Inconel 625 manufactured by LDED exhibits slightly better corrosion resistance than LPBF because of less impedance deterioration and a lower corrosion current after 180 days of immersion in sulfuric acid solution. This indicates that the formation of the more refined microstructure in the LPBF process adversely affects the corrosion resistance of Inconel 625. Although the addition of Mo in nickel-based superalloys improves the resistance to pitting corrosion, its action is synergistic with chromium.⁴² Our observations show that despite the high Mo content, Cr-depleted sites are prone to the formation of corrosion pits.



Fig. 4. SEM SE and SEM BSE images of LPBF and LDED Inconel 625 after the corrosion tests lasting 30 days, 90 days and 180 days; the values of the area fraction of the corrosion pits A_{A CP} are given.



Fig. 5. Polarization curves after exposure in 10% H_2SO_4 solution of (a) LPBF and (b) LDED Inconel 625, together with the Nyquist plots after corrosion test of (c) LPBF and (d) LDED Inconel 625, as well as an equivalent electrochemical circuit (R_1 , R_2 = resistors; CPE₁, CPE₂ = constant phase elements).

Table II. Electrochemical parameters of the LPBF and LDED Inconel 625 after immersion in 10% H_2SO_4 solution; (R_1 = resistance of the passive surface, R_2 = resistance of the metal/oxide interface, CPE₁, n_1 = capacity and phase angle of the constant phase element of the passive surface, CPE₂, n_2 = capacity and phase angle of the metal/oxide interface)

		Parameter									
Specimen		OCP [V]	$E_{ m corr}$ [V]	$j_{ m corr}$ [A cm ⁻²]	$R_1\left[\Omega ight]$	$R_2 \left[\Omega ight]$	$CPE_1 \ [\mu S]$	n_1	$CPE_2 [\mu S]$	n_2	
LPBF	30 days	-0.071	-0.176	$7.94 imes 10^{-5}$	273.70	0.95	22.07	0.89	0.72	0.91	
	90 days 180 davs	$-0.132 \\ -0.220$	-0.147 -0.205	10.23×10^{-5}	$2.65 \\ 5.23$	$24.99 \\ 14.14$	$36.31 \\ 12.21$	0.91	$638.21 \\ 75.88$	0.58	
LDED	30 days	-0.183	-0.142	1.38×10^{-5}	265.59	7.43	1.33	0.60	86.22	0.67	
	90 days 180 days	$^{-} 0.214 \\ - 0.215$	$^{-} 0.217 \\ - 0.193$	$\begin{array}{l} 8.91 \times 10^{-5} \\ 9.33 \times 10^{-5} \end{array}$	$\begin{array}{c} 10.24 \\ 15.26 \end{array}$	$\begin{array}{c} 109.60\\ 62.04 \end{array}$	$10.71 \\ 11,286.03$	$\begin{array}{c} 0.90 \\ 0.92 \end{array}$	$\begin{array}{c} 1.33 \\ 78.24 \end{array}$	$\begin{array}{c} 0.50 \\ 0.66 \end{array}$	

CONCLUSION

The microstructure of Inconel 625 manufactured by LPBF and LDED and its electrochemical corrosion behavior in 10% H₂SO₄ solution in tests lasting 30 days, 90 days and 180 days were studied. In summary, we have demonstrated that the corrosion resistance of the LPBF and LDED Inconel 625 depends on the microstructure refinement. In both cases, the initiation of pitting corrosion occurs at the grain boundaries, melt pool boundaries and Effect of Microstructure of Additively Manufactured Inconel 625 on Long-Term Corrosion Behaviour in Sulfuric Acid Media

interdendritic areas and around precipitates of secondary phases according to the locally higher concentration of Nb and Mo and the reduced concentration of Cr in these sites.

With prolongation of the corrosion test, the area fraction of the pits increases in both variants; however, the intensity of corrosion varies greatly. After 180 days of testing, pits occur on the entire surface of the LPBF Inconel 625, while in LDED they are localized at grain boundaries and interdendritic areas.

The observed mechanism of corrosion initiation and propagation behavior during immersion in H_2SO_4 solution is well correlated with the electrochemical studies of LPBF and LDED Inconel 625. Measurements of EIS and polarization curves confirmed less intense initiation of corrosion pits in LPBF than LDED after 30 days of testing. Meanwhile, after longer periods of tests, the observed higher drop in impedance and increase in j_{corr} in LPBF than LDED Inconel 625 was mainly related to a more intensive propagation of corrosion pits in areas with higher free energy. Consequently, the results confirm that LPBF Inconel exhibits a lower resistance to corrosion in sulfuric acid than LDED because of the higher density of corrosion initiation sites.

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CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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