Manufacturing of corrosion-resistant steel 316L using additive technologies as a way to increase its corrosion resistance

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Abstract. Austenitic steel 316L is a common corrosion-resistant structural material used in a variety of industries from food to nuclear. There is a well-known tendency of steel 316L to precipitate second phases in the temperature range of 500 ... 800 ° C. During operation in this temperature range, degradation of corrosion properties occurs for a long time. Materials and parts obtained by modern methods of additive technologies are of great interest, since this method of alloys processing affects the thermodynamic equilibrium of the system and the kinetics of the release of second phases in aging alloys. The ability to control the kinetics of second phase formation is one of the key factors for improving the corrosion resistance of alloys. In this work, the effect of the method of steel 316L manufacturing (traditional and selective laser melting) on corrosion resistance at a temperature of 750 ° C for 100 hours in a KCl-NaCl environment was estimated. Corrosion tests were carried out, as a result of which it was found that a sample of 316L steel obtained by the method of selective laser melting has the lowest corrosion rate..

1 Introduction

Selective laser melting (SLM), which belongs to additive technologies or 3D printing methods, is currently an innovative direction in the formation of complex-shaped metal parts. The method of selective laser melting consists in layer-by-layer synthesis of an object due to automatically controlled scanning of microlayers of a powder material with high-energy laser radiation [1-5].

Using the SLM method, it is possible to obtain products of almost any configuration, however, with size restrictions dictated by the technical features of the SLM installation [6-8].

The properties of a part made by the SLM method, as well as its structure, depend on many technological parameters. Currently, there are up to 120 different factors affecting the quality and characteristics of objects obtained by the SLM method [9-13].

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The unique conditions of powder crystallization during SLM, namely, ultrafast cooling rates from the state of a liquid metal, as well as multiple thermal cycling, lead, in most cases, to the formation of nonequilibrium structures [14–20].

Austenitic steel 316L is a common corrosion-resistant structural material used in a variety of industries from food to nuclear. The tendency of steel 316L to precipitate second phases in the temperature range of 500 ... 800 ℃ is well-known. During operation in this temperature range, degradation of corrosion properties occurs for a long time [21, 22]. In one of the previous works [23], it was found that the fraction of precipitates of the second phase (χ-phase) correlates with the energy density of the laser beam during selective laser melting. The ability to control the kinetics of second phase formation is one of the key factors for improving the corrosion resistance of alloys.

Therefore, the purpose of this work is to evaluate the effect of the method of steel 316L manufacturing (traditional and selective laser melting) on corrosion resistance at a temperature of 750 ° C for 100 hours in a KCl-NaCl environment.

2 Material and research methods

Corrosion-resistant steel 316L was used as a material for research. Table 1 shows the chemical composition of the investigated steel.

Table 1. Chemical composition of steel 316L [9]

Content by elements, mass,%									
C	Mn	P	S	Si	Ti	Ni	Mo	Cr	Fe
up to						10,0…14,0	2,0…3,0	16,0…18,0	the rest
0,03	2,0	0,045	0,03	1,0	0,5				

To achieve the set tasks, metal samples from steel 316L obtained both using SLP and using traditional metallurgical technology were investigated.

Table 2 shows the SLM modes of samples made of steel 316L.

Table 2. Parameters of the SLM mode

Laser beam power, W	**Scanning speed, mm / s**	**Shading step, µm**	**Step in the contour, µm**	**Layer thickness, microns**
357	850	80	70	50

For testing, metal samples made of steel 316L were pre-annealed in an air atmosphere in a SNOL muffle furnace at a temperature of 1200 ° C with a holding time of 1 hour, followed by water quenching.

Metallographic analysis was performed using an Epiphot 200 optical microscope at magnifications of 100 ... 1000 times. Photographs of the microstructure were obtained using a Nikon digital camera mounted on a microscope and connected to a computer, and the Nis-Elements Basic Research software.

During the manufacture of thin sections the steel samples were treated on the sandpaper with consequent reduction of the fraction of abrasive paper to a minimum by using StruersLaboPol- 5 installation, and then polishing using the diamond slurry with a decrease in the fraction of 9 to 1 micron. To obtain the orientational-compositional contrast in SEM, it is necessary to remove the surface stresses arising during mechanical processing; therefore, the samples were subjected to final polishing on colloidal silicon for 30 min.

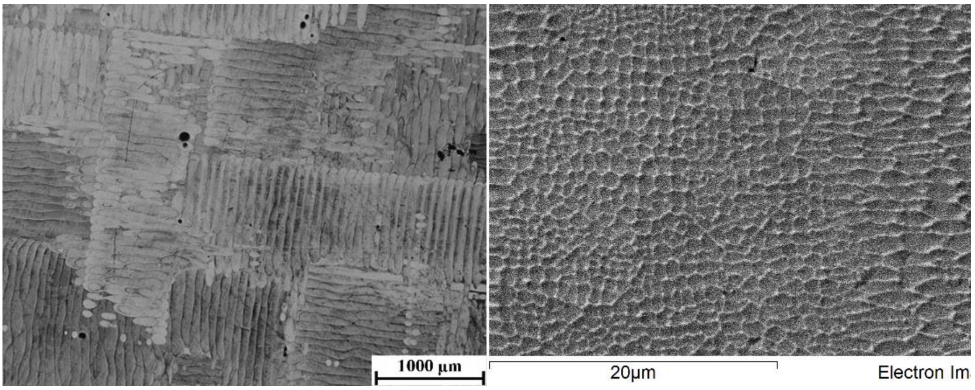
Metallographic analysis was performed using a Jeol JSM-6490LV scanning electron microscope. The chemical composition of different zones of the samples and inclusions was determined by X-ray microanalysis (MRSA), which was carried out on a Jeol JSM-6490LV scanning electron microscope equipped with Oxford Inca Energy 350 energy dispersive microanalyzers at an accelerating voltage of 20 kV.

Corrosion tests were carried out in a KCl-NaCl mixture, which was prepared by mixing individual KCl and NaCl salts in an MBraunUnilab box in an atmosphere of dry and purified argon. The molar ratio of the components KCl / NaCl was 1: 1. The holding of the samples was carried out in a quartz cell, into which an alundum crucible with salt and samples was placed. The melt in the cell was under an atmosphere of high purity argon (actual argon content 99.999%). The exposure of the samples was carried out at 750 ° C. The time of the experiments was 100 hours. After corrosion tests, the melt was cooled to a room temperature, steel samples were removed, thoroughly washed and dried. Corrosion rate was calculated from the change in the mass of each sample - gravimetric method.

The essence of the gravimetric method is to determine the change in the mass of a sample of the material under study, exposed to any medium under certain conditions. When the corrosion products are completely soluble in a saline medium, the loss in the mass of the sample directly characterizes the magnitude of the destruction of the metal. Determination of the change in the loss of samples during exposure makes it possible to establish the kinetic laws of corrosion processes.

3 Results and discussion

Figure 1 shows images of the structure of samples after SLM across the growth direction.

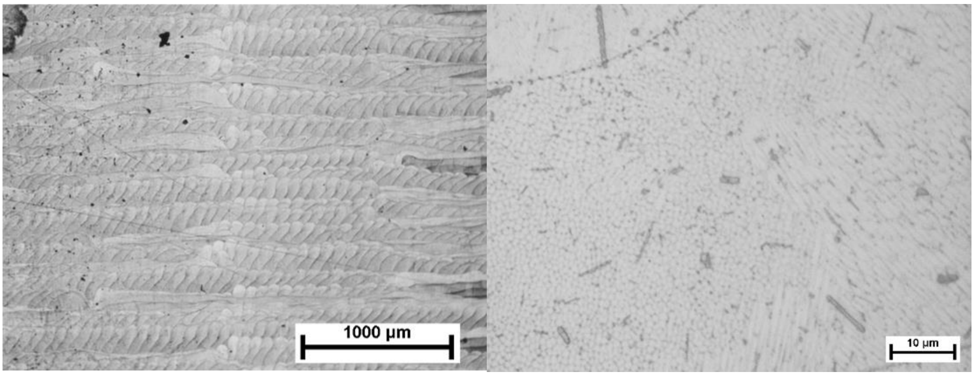


a b

Fig. 1. The microstructure of samples after SLM across the growth direction (a – OM, b-SEM)

The melt baths in this section have the shape of an ellipse, and they are connected into tracks in two mutually perpendicular directions. The track width is on average 80 µm, which corresponds to the “shading step” (from the parameters of the SLM mode).

Along the growth direction, the melt baths have the shape of arcuate segments (Figure 2), the width of which also coincides in size with the shading step and averages about 80 μm. The depth of the baths corresponds to the layer thickness and is about 50 μm.



a b

Fig. 2. OM images showing the sample microstructure after SLM along the growth direction.
a, b - SLM sample × 10 and × 500

Annealing at 1200 ° C initiates the formation of a typical structure of the austenitic alloy 316L: polyhedral grains with annealing twins characteristic of fcc crystals appear, their average diameter is 50 ... 150 μm, while elongated grains inherited from 3d printing are preserved over a larger area of the section (Figure 3).

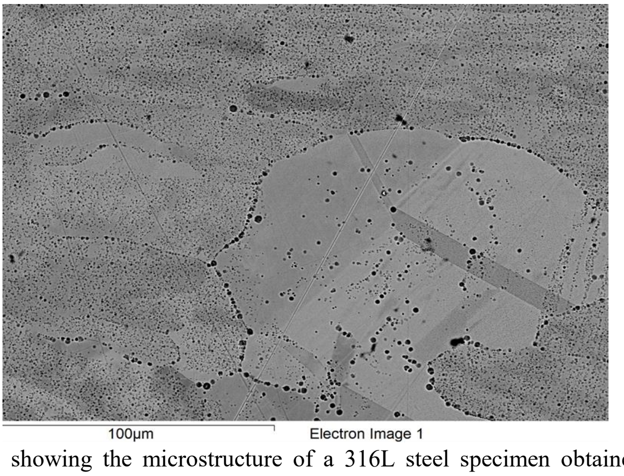


Fig. 3. SEM images showing the microstructure of a 316L steel specimen obtained by the SLM method after heat treatment

Samples from steel 316L obtained by conventional metallurgical technology have an austenitic structure. In the sample without heat treatment, a relatively high density of defects is observed. After annealing at 1200 ° C for 1 hour, polyhedral austenite grains with a size of about 25 ... 30 μm are visible in the structure (Figure 4).

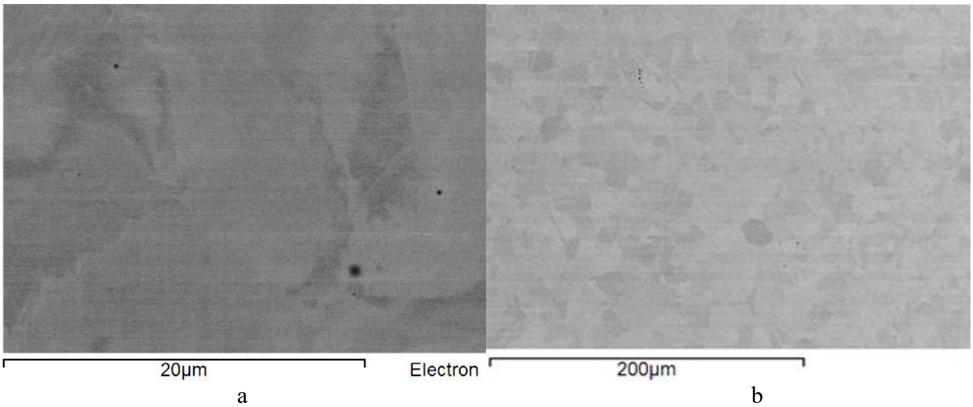


Fig. 4. SEM images showing the microstructure of a 316L steel sample obtained by traditional metallurgical technology: a - without heat treatment, b - after annealing at 1200 ° C, 1 h

Figure 5 shows images of the microstructures of 316L steel obtained by the SLM method after corrosion tests. The preliminary annealing promoted a more uniform corrosion of the sample as compared to the sample without preliminary heat treatment. In both cases, the steel surface after interaction with the KCl-NaCl melt is depleted in chromium: 10 wt. % - on the surface, 17 wt. % - in the depth of the sample. In addition, during holding in the KCl-NaCl melt at 750 ° C, a large amount of precipitates of the second phase enriched in molybdenum (up to 14 wt%) appeared in the sample. They are located along the boundaries and in the body of austenite grains. They have an elongated rounded shape - along the borders; needle-like - in the body of the grain.

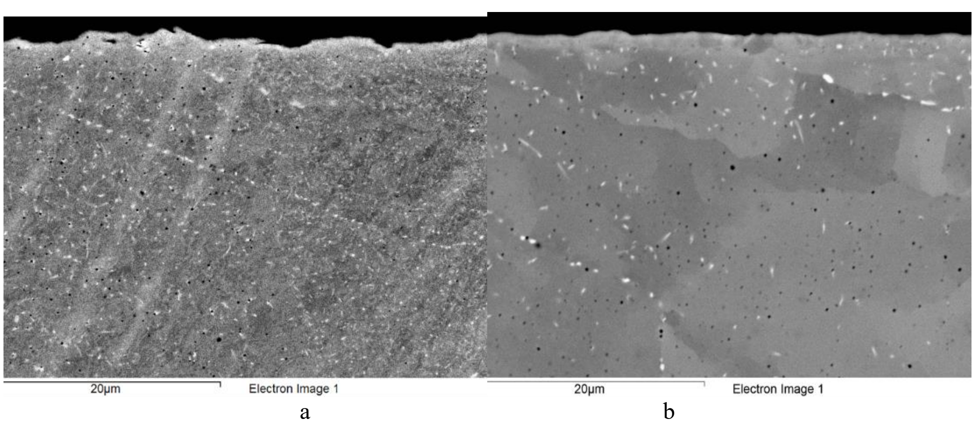
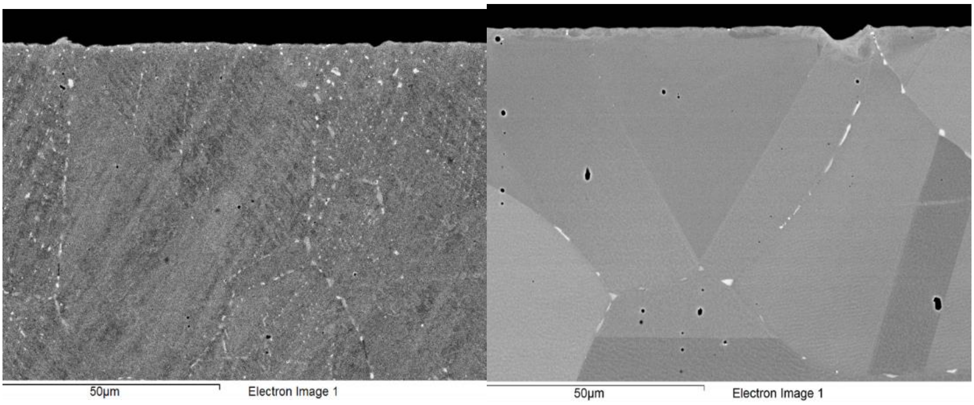


Fig. 5. SEM images showing the microstructure of steel 316L obtained by the SLP method after corrosion tests. a - without preliminary heat treatment, b - after annealing at 1200 ° C, 1 h

Similar results were obtained for steel samples obtained by traditional metallurgical technology. However, the precipitation of the second phase is significantly less than for the samples obtained by the SLM method, which is due to the smaller number of defects.



a b

Fig. 6. SEM images showing the microstructure of steel 316L obtained by traditional metallurgical technology after corrosion tests. a - without preliminary heat treatment, b - after annealing at 1200 ° C, 1 h

Table 3 shows the corrosion rates of 316L steel in the KCl-NaCl melt at 750 ° C for 100 hours. The lowest corrosion rate is observed for a steel sample obtained by selective laser melting after preliminary annealing.

The corrosion rate, as a rule, is the higher, the higher the degree of defectiveness of the alloy. In this case, annealing leads to a decrease in the number of defects; therefore, the performed heat treatment promoted a decrease in the corrosion rate [24, 25].

In turn, the difference in corrosion rates between 316L steel obtained by traditional technology and a similar alloy obtained using the selective laser melting method is due to the peculiarities of the technological process of obtaining a metal alloy. It is possible that the method of selective laser melting allows to obtain an alloy with a more perfect distribution of chemical elements, which allows to reduce the corrosion potential in chloride melts (Table 3).

Table 3. Corrosion rate

	Conventionally produced 316L steel	**Steel 316L, obtained by SLM method**
Corrosion rate, mm / year	Without heat treatment	
	0,029	0,020
	After annealing	
	0,023	0,016

Thus, reducing the corrosion rate due to the use of the SLM method in the manufacture of 316L steel will extend the service life of products by 20%. At the same time, it is possible to estimate the change in the volumes of greenhouse gas emissions ΔGHG due to the change in the service life of products made of 316L steel, manufactured by the SLM method, compared to products made of 316L steel, obtained by traditional metallurgical methods:

$$\Delta GHG_{service\ \ life} = K_s \frac{GHG_{total}^0}{100\%},$$

where 〖GHG〗_total^0 – is the total volume (amount) of CO2 emissions during the basic processes of production and use; K_s - the degree of change in the service life of innovative products in comparison with the standard, % [26].

If we take for 〖GHG〗_total^0 the total CO2 emissions per year attributable to the ferrous metallurgy, equal to 145 million tons (according to data for 2005 [27]), then a decrease in the carbon footprint due to a change in the method of manufacturing steel 316L will be about 29 million tons.

4 Conclusions

1. The nature of the destruction of steel 316L during holding in an equimolar mixture of KCl-NaCl at a temperature of 750 ° C is continuous and uniform. The depth of the layer with the changed chemical composition is about 10 ... 15 microns.
2. The corrosion rate of samples from alloy 316L obtained by the SLM method is lower than that of similar alloys obtained by the traditional method.
3. Reducing the carbon footprint by changing the method of manufacturing steel 316L from traditional metallurgical processing to selective laser melting will amount to about 29 million tons.

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