Columnar fine grained structure and chemical composition features of austenitic stainless steels 316L and 321 produced by the laser beam powder bed fusion

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Abstract. One of the promising ways of producing metal parts with the required properties and geometry is the technology of fusing powder layers by the using of laser beam. In the present work, the specific structure of samples obtained by the laser powder layer bed fusion is investigated. A comparative analysis of the structure of austenitic steels 316L and 321 with slightly different chemical composition are carried out. The epitaxial mechanism of the formation of elongated grains whose dimensions in the building direction is often several times higher than the powder layer thickness is considered. The reason for the formation of the droplet-shaped boundaries between the fusion layers is explained. The formation of ultrafine columnar grain substructure (the characteristic dimensions of the elements are less than 1 mcm) for both of this steels is explained

1. Introduction

In the last decade, additive technologies have developed rapidly, allowing to grow complex and shaped parts from organic and metallic materials without practically the use of mechanical processing. Despite the relatively high cost of equipment and consumables, as well as the remaining dimensional limitations, additive technologies have proved to be very effective in the manufacturing of unique products.

Promising from the point of view of the formation of metal products with the required parameters is a set of methods in which the creation of the product occurs by successively fusing the powder layers with the using of directed energy flows. In particular, great interest is shown in additive technologies based on selective laser melting (SLM) of metallic powders. Over the past decade the SLM method of various powders based on alloys and steels has been mastered. However, not so long ago many studies were directed to the study of the properties of stainless steels produced by the SLM method.

Due to the involvement of specific effects in SLM processes, it is of great interest to investigate the structural features of the obtained austenitic stainless steels. A particular feature of austenitic steels is the absence of γ-α transition. In our opinion, austenitic stainless steels are currently used most actively in the oil, gas, oil refining, chemical and petrochemical industries. It is also known that austenitic stainless steels are used for the manufacture of automobile components and it is used for the manufacture of certain parts of nuclear reactors and other products.

It is important to note that there are theoretical and experimental attempts to explain the features in the structure of stainless steels prepared by the SLM method. For example, in [1] the formation of a columnar fine-grained structure in 316L stainless steel SLM samples were explained by thermodynamic effects, as well as molybdenum segregation along the columnar grain boundaries.

One can see that in the process of laser melting of a powder on a substrate a very large temperature gradient arises, which also determines the structural peculiarities of products manufactured by the SLM method [1-5].
In this connection, the aim of the work is to compare the structure of two similar types of 316L and 321 austenitic stainless steels manufactured by the SLM powder method, as well as in an additional comparison with the standard structure of steel 321 and laser treated.

2. Experimental methods and materials

As an initial material powders of stainless steels were used both 316L (Höganäs JSC, Sweden) and 321 (Polema OJSC, Russia).

The chemical composition of the powders was determined using the Niton XL3t and LECO CS744 units (Table 1). The particle size distribution was measured at the Malvern Mastersizer 2000. The data obtained during the powder studies are given in [6] and satisfy the requirements of the SLM installation.

To create samples using the SLM method the EOSint M270 was used. The cube with an edge of 1 cm was chosen as the model given in the sample building program. The formation of volumetric production is carried out in stages by successive formation of 40 μm layers with a periodic feed in the laser fusion zone on the platform. Samples were built by using the following process parameters: laser power 190 W, scanning speed 800 mm/s, laser spot diameter 80 mcm.

To scan the surface of a 321 sample with a classic austenitic structure, the LENS-750 was used. Sample tracks were built by using the following process parameters: laser power 190, 225, 259 and 293 W, scanning speed 16 mm/s, laser spot diameter 470 mcm.

Investigation of the structure of the obtained samples was carried out on sections of their cross section, which after polishing were electrochemically etched at room temperature in a 10% aqueous solution of oxalic acid. Then, the sections were examined with the Tescan Vega 3 SBH scanning electron microscope (SEM) equipped with the INCA X-Act X-ray microanalysis system, as well as in the FEI Quanta 3D SEM with the EDAX TSL diffraction analysis system.

Table 1. Chemical composition of the stainless steel powders.

| Powder | C  | Si | Mn | Cr  | Ni  | Mo | Ti |
|--------|----|----|----|-----|-----|----|----|
| 316L   | 0.03 | – | 1.5 | 16.8 | 13.3 | 2.6 | – |
| 321    | 0.12 | 0.8 | 0.8 | 17.2 | 10.6 | – | 0.8 |

3. Results and their discussion

The images in figure 1 show the difference in the structure of steel after SLM (Figure 1 on the right) from the structure of classical austenitic steel obtained by the thermomechanical treatment method (Figure 1 on the left). The additive structure is distinguished by the presence of layers of drop-shaped regions, the thickness of which slightly exceeds the dimensions of the initial powder material. It is also noticeable that there is a layer of elongated grains growing through the drop-shaped boundary.

Figure 1. SEM images of austenitic steels structures obtained by thermomechanical processing (left) and SLM (right).

As it was shown in [7], irradiation of the surface of a powder by a laser beam leads to the formation of a spreading melt, which displaces adjacent, still unmelted, powders. Further, the rapid growth of the melt temperature results in the evaporation of the surface layer, which leads to the formation of a depression above which a high vapor pressure can persist over time. The pressure, the vector of which is directed normally to the walls of the formed depression, increases exponentially as the melt approaches the boiling point (3000K), which pushes the formed melt outward [7]. This effect is clearly visible when observing
the SLM process, when the motion of the laser beam is accompanied by a splash of molten metal. As the beam moves, the surface traversed by it cools and the vapor pressure over the depression decreases, yielding to the melt that fills it. The filled cavities are clearly visible on the cut of the sample, and they form droplet-shaped regions (Figure 2). When each successive layer of powder is fused, the scanning direction changes, which is reflected in the radius of the droplet-shaped areas visible in the plane of the section [8, 9].

![Figure 2. SEM-photos of the 316L steel sample prepared by the SLM method.](image)

![Figure 3. SEM-photos of the 321 steel sample prepared by the SLM method.](image)

Observation of the sections of a sample of 316L stainless steel that had undergone an etching procedure made it possible to reveal within the regions of a droplet-shaped form, grains of a nonclassical elongated shape whose boundaries in some cases do not coincide with the boundaries of the droplet-shaped regions (Figure 2). In addition, the effect of grain crushing on smaller constituent elements was observed, a complex columnar subgrain microstructure with characteristic dimensions of the order of 0.5 microns was observed.

The figure 3 shows photographs of the structure of austenitic steel grade 321. Obviously, the similarity of the given structure to the already given in the article of the austenitic class 316L. This seems interesting, since molybdenum is absent in steel 321.

It can be assumed that the observed inhomogeneity and crystalline anisotropy can be attributed to the high cooling rates of a thin layer of liquid metal on the surface of the precursor layer. A directional heat sink in the liquid layer leads to the appearance of an oriented cellular-dendritic and columnar microstructure near the boundary of the melt layer. However, in a number of works [1, 7], it was shown that the cooling rate in advanced methods, such as SLM, reaches $10^5 - 10^7$ K/s. This cooling rate is sufficiently high to avoid the formation of any dendritic structure, since the time for the formation of a secondary dendritic sleeve is not sufficient, and thus only columnar morphology is observed.

In order to reliably detect the polycrystalline structure on the FEI Quanta 200 3D SEM maps of the crystallographic orientations obtained by diffraction of backscattered electrons (EBSD) were additionally constructed. Figure 4 shows the corresponding orientation map, where different colors correspond to different rotations of the crystallographic basis relative to the normal to the plane of the section.
As can be seen, the detected grains are somewhat smaller in size than their drop-shaped regions. In this case, the grains have a distinctly elongated shape (the average aspect ratio of the thus obtained grains is 0.25) in the direction of building the sample (Building Direction), which is due to the oriented crystallization of the grains of one layer on the surface of the previous one as a primer for directional crystallization. The large chords of the resulting grains are oriented predominantly ± 10° relative to the building direction of the sample, and their length can significantly exceed the value of the layer of the structure. Characteristic Ferre diameter is 68 mcm. In addition, it can be seen that the columnar subgrains have almost the same crystallographic orientation within each large grain. The phase analysis carried out using the EBSD method showed that the steel retained a single-phase structure (austenitic), since the alpha-phase (ferrite) particle was not detected. The crystal structure is represented by a single $\gamma$-Fe phase.

Analysis of the map of crystallographic orientations makes it possible to draw a speculative conclusion that the subgrains located within each large block (crystallite, grain) have roughly equal crystallographic orientations, and therefore the boundaries identified in the course of the metallographic study are supposedly liquation or dislocation nature [1].

It can been suggested that insufficient time for the diffusion of alloying elements with the rapid growth of grains led to the appearance of composite inhomogeneities and, as a consequence, to the identification of subgrain boundaries in the etching process, as well as the boundaries of the drop-shaped form between the layers. Such chemical inhomogeneities have, as a rule, a harmful effect on the quality of the metal, because they lead to unevenness of its properties.

The images in figure 5 show the most obvious inhomogeneities in the distribution of the composition elements in the interlayer boundary. Thus, for example, inhomogeneities in the distribution of silicon, carbon, and molybdenum are seen. It was possible to establish that the region of the interlayer boundary is enriched in carbon. Unfortunately, the problem of resolving composition inhomogeneities at the boundaries of columnar subgrains turned out to be beyond the capability of the method of energy-dispersive micro-X-ray analysis in SEM.

### 3.1. Classical 321 stainless steel

Also, an experiment was performed in which a sample of austenitic steel obtained by thermomechanical treatment was laser treated. For this, the steel sample was placed in the focal plane of the laser system and...
subjected to laser radiation at various power levels of the optical generator. On the basis of the observed results, as a result of this experiment, the assumption is made of the appearance of a dendritic cellular-columnar structure as a result of a combination of thermodynamic conditions.

Figure 6. SEM images of the surface of the 321 steel section after treatment by laser radiation with different power (from left to the right 190, 225, 259 and 293 W) at 16 mm/s scanning speed.

The analysis of the obtained sample revealed a similar cellular-dendritic structure, considered above, at certain values of the energy of laser radiation introduced into the sample surface (Figure 6).

It is interesting to note that knowing the geometrical parameters of the laser beam, the power and the scanning speed is possible to calculate the specific energy input for each of the processing methods. In case of the SLM process, the specific energy input is $4.73 \times 10^{10}$ Ws/m$^3$. It should be noted that this structure remains up to speeds of 600 mm/s [10], which corresponds to a specific energy input of $6.30 \times 10^{10}$ Ws/m$^3$. In case of the LENS process, the specific energy input ranges from 6.85 to $10.6 \times 10^{10}$ Ws/m$^3$. From the data obtained, it can be seen that at an energy input of $9.5 \times 10^{10}$ or more, the formation of a columnar structure may not occur. Thus, it can be concluded that one of the features of the SLM process is that large cooling rates are achieved during fusion, leading to the formation of a dendritic columnar structure. In this regard, samples of austenitic stainless steels, manufactured by SLM, have increased mechanical properties.

4. Conclusion

A nonclassical subgrain structure was revealed, the appearance of which supposedly depended on the difference in the kinetics of the alloying elements under conditions of rapid cooling.

The orientational heredity of crystallites is noted with successive build-up of layers. The crystallite size in the building direction is several times higher than the depth of penetration of the layer.

The features of the crystal structure for austenitic stainless steels 316L and 321 built using the technology of selective laser melting are similar to each other and determined by the production technological parameters.

The study of samples, manufactured with the help of SLM technology, by the EBSD method showed the preservation of the initial $\gamma$-phase structure inherent in steels of this class.

Acknowledgements

This work was supported by the grant of Russian Scientific Fund №15-19-00210.

5. References

[1] Saeidi K, Gao X, Zhong Y and Shen Z J 2015 Hardened austenite steel with columnar sub-grain structure formed by laser melting Materials Science & Engineering A 625, 221–29
[2] Khalfallah I Y, Rahoma M N, Abboud J H and Benyounis K Y 2011 Microstructure and corrosion behavior of austenitic stainless steel treated with laser Optics & Laser Technology 43, 806–13
[3] Kwok C T, Lo K H, Chan W K, Cheng F T and Man H C 2011 Effect of laser surface melting on intergranular corrosion behaviour of aged austenitic and duplex stainless steels. Corrosion Science 53, 1581 – 91
[4] de Lima M S F and Sankaré S 2013 Microstructure and mechanical behavior of laser additive manufactured AISI316 stainless steel stringers Materials and Design doi: http://dx.doi.org/10.1016/j.matdes.2013.10.016
[5] Peifeng Li 2015 Constitutive and failure behaviour in selective laser melted stainless steel for microlattice structures Materials Science&Engineering A 622 114–20

[6] Deev A, Kuznetcov P and Petrov S 2016 Anisotropy of mechanical properties and its correlation with the structure of the stainless steel 316L produced by the SLM method Phys. Proc. 83 789-96

[7] Saad A. Khairallah, Anderson Andrew T, Rubenchik Alexander and Wayne E King 2016 Laser powder-bed fusion additive manufacturing: Physics of complex melt flow and formation mechanisms of pores, spatter, and denudation zones Acta Materialia 108 36-45

[8] Xin Zhou, Dianzheng Wang, Xihe Liu, DanDan Zhang, Shilian Qu, Jing Ma, London Gary, Zhijian Shen and Wei Liu 2015 3D-imaging of selective laser melting defects in a Co–Cr–Mo alloy by synchrotron radiation micro-CT Acta Materialia 98 1–16

[9] Murr L E, Martinez E, Hernandez J, Collins Sh, Amato K N, Gaytan S M and Shindo P W 2012 Microstructures and Properties of 17-4 PH Stainless Steel Fabricated by Selective Laser Melting J. Mater. Res. Technol 1(3) 167-77

[10] Zhukov A, Deev A and Kuznetsov P 2017 Effect of alloying on the structure parameters and mechanical properties of steels 316L and 321 samples obtained by selective laser melting Phys. Proc. 89 172-78