Structure and thermal stability of austenitic steel Fe-17%Cr-12%Ni-2%Mo-0.02%C synthesized by selective laser melting

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Abstract. The structural features of the Fe-17%Cr-12%Ni-2%Mo-0.02%C alloy manufactured by selective laser melting (SLM) and the thermal stability of the formed structures are investigated. It is shown that crystallization cells form at SLM, the boundaries of which are volume plexes of dislocations, fixed by segregation of impurity atoms. The microhardness of such a structure is one and a half times higher than that of an alloy of the same composition in a standard hardened state. During annealing up to 800 °C, this cellular structure maintains stability while maintaining hardness indices.

1. Introduction

Selective laser melting (SLM) is one of laser additive technologies. The new principle of part synthesis is realized in this method: a thin layer (several tens of micrometers) of powder material is fed to a massive metal substrate, smoothed with a roller and re-melted by a laser beam, forming the first profile of a part. The form of the part profile is determined by the laser path on the substrate surface. Further, the operations of feeding the powder, smoothing it, and re-melting are repeated until the entire volumetric detail is grown layer-by-layer [1, 2]. The continuity of the part is ensured by the re-melting depth of about 1.5 times the thickness of the powder layer. The massive substrate and the local area of laser exposure (a diameter of the molten pool is about 100 μm) provide high heating and cooling rates during re-melting of the powder material. According to published data, the cooling rate of the material after melting is approximately $10^5 \div 10^6$ K/s.

Thus, the formation of structure during SLM occurs under conditions of superfast cooling of material from liquid state and further thermal cycling upon re-melting of neighboring sections of powder material. As a result, a nonequilibrium material structure with an excess of crystalline structure defects is formed and further stabilized by thermal cycling.

It is known that during SLM process the cellular structure appears in materials (Fig.1) [3, 4]: cooling rates from liquid state about $10^5 \div 10^6$ K/s correspond to the appearance of crystallization cells [5]. It was shown in [6–9] that the cell boundaries are volume plexes of dislocations, and these boundaries are fixed by segregations of alloying elements or by dispersed particles [10, 11].

In this paper, we study the features of the cellular structure of the austenitic alloy Fe-17%Cr-12%Ni-2%Mo-0.02%C (316L) and its thermal stability.
2. Material and experimental methods
The gas atomized spherical powder of austenitic alloy Fe-17%Cr-12%Ni-2%Mo-0.02%C with average size of 25 μm was subjected to SLM. The powder was in a dual (α+γ) state; the amount of α phase was 8%. SLM was performed with the following technological parameters: the laser power was 50 W, the scanning speed was 100 mm / s, and the laser beam diameter was 70 μm [12]. The process was carried out in a protective atmosphere of nitrogen, a cross melting strategy was used, austenitic steel was used as a substrate. The steel structure was studied in the initial state after the SLM, as well as after annealing for 1 h, which was carried out at range of temperatures from 100 to 1200 °C with a step of 100 °C. The phase composition of the alloy in all structural states was characterized using X-ray diffraction (XRD) in Co Kα radiation. The microstructure of the alloy was studied by metallographic analysis, scanning and transmission electron microscopy (SEM, TEM). Distribution of alloying elements was studied on foils by X-ray microspectral analysis. The concentration of gas impurities in the alloy was determined by the method of reducing combustion. The steel microstructure was revealed by electrolytic etching in a 10% alcohol solution of oxalic acid at a voltage of 20 V and a current density of 0.1 A / cm². TEM samples were prepared by mechanical grinding of the sample to approximately 100 μm thickness and then further electrolytic thinning in a 10% alcohol solution of perchloric acid at a voltage of 35 V. The final thinning was carried out by ion polishing. Microhardness has been measured using standard microhardness tester with the load of 50 g; the relative measurement error was 5%.

3. Results and discussion
The XRD observations revealed that only a single-phase γ-structure forms in the austenitic alloy Fe-17%Cr-12%Ni-2%Mo-0.02%C after SLM. The microstructure of the austenitic alloy presented in Figure 1. Figures 1 (a, b) show that the structure after SLM has a certain hierarchy: the melt pools that were formed by laser irradiation are divided into many crystallization cells with a size about 0.5 μm. Inside the melt pools, fragments of a few tens of micrometers in size can be distinguished, within which the columnar crystallization cells have the same orientation. The cell orientation changes when crossing the fragment boundary. It can be assumed that the fragments are the initial powder particles, the substance of which did not manage to change its initial orientation due to the high temperature gradient during re-melting. This assumption is confirmed by the coincidence of fragment and powder sizes.

The microstructure obtained from a single fragment by the TEM method and the selective area electron diffraction (SAED) pattern from this field of view are presented in Figure 1(c). It can be seen from the figure that the cell boundaries are volumetric plexuses of dislocations, while there are a small amount of free dislocations inside the cells. Sometimes it is possible to observe a partition of the crystallization cell into smaller regions of about 100 nm in size with similar boundaries, which are dislocation plexuses. Thus, the dislocation structure arising during SLM is similar to the structure observing after plastic deformation [13]. It is believed that in the process of laser re-melting high stresses arise in the material with cell structure due to high temperature gradient, which leads to the nucleation of new dislocations and their rearrangement. In [14–15], it was experimentally shown that residual stresses comparable with the yield strength of the material can be present in objects obtained by SLM method. This dislocation configuration allows to reduce the strain energy in the material [16]. The point character of the electron diffraction pattern in Figure 1 (c) reveals that the column cells of one fragment have both the same spatial and crystallographic orientation: the zone axis of the electron diffraction pattern is (123).
The results of X-ray spectral analysis of the cellular structure of austenitic alloy are presented in Table 1. The distribution of alloying elements and impurities in the center of the crystallization cells and its boundaries is studied. From the table, it can be found that the concentration of Cr and Mo at the cell boundaries is slightly higher than that at its center. This result allows us to make an assumption that the plexuses of dislocations at the cell boundaries are stabilized by Cr and Mo segregations.

**Table 1.** Microspectral X-ray analysis of the cellular structure

| Concentration of elements, wt.% | Ni   | Cr   | Mo   | Mn   | Si   |
|---------------------------------|------|------|------|------|------|
| center                          | 11.5±0.1 | 16.8±0.2 | 2.0±0.1 | 1.1±0.1 | 0.6±0.1 |
| boundaries                      | 11.8±0.2 | 17.6±0.4 | 2.7±0.2 | 1.0±0.1 | 0.8±0.1 |

In addition, the concentration of gas impurities in the steel was determined by the method of reductive burning. It was found that the concentrations of oxygen and nitrogen in the re-melted alloy are an order of magnitude higher than their concentrations in the powder material. After the SLM, the concentrations of oxygen and nitrogen are 0.10 and 0.16 wt.%, respectively. It is likely that these impurities which are from the oxide film on the powder surface and the protective atmosphere were dissolved in the melt during the process of re-melting. It was assumed that oxygen and nitrogen atoms also settle on dislocation plexuses at the cell boundaries result in its additional stabilization.

To study the thermal stability of the cellular structure, the changes of the alloy structure were investigated with increasing of annealing temperature. Annealing at all experimental temperatures result in retaining of a single-phase state of the alloy. The structure of the austenitic alloy after SLM and additional annealing are presented in the Figure 2. It can be seen from the figure that up to the...
annealing temperature of 800 °C, the cellular structure is preserved in the alloy. In the sample heat treated at 800 °C, the contrast at the cell boundaries becomes weaker than the one at the fragment boundaries. Annealing at 1000 °C results in disappearing of cells and only the fragment boundaries are preserved in the structure. The further increasing of the annealing temperature does not lead to the structure to change.

Dislocation structure changing upon annealing are presented in Figure 3. It can be seen that at 500 and 600 °C the cellular structure remains almost unchanged compared to the initial state after SLM (Fig. 3 (a, b)). After annealing at 700 °C, the volumetric plexuses of dislocations at the cell boundaries become flatter and its density apparently decreases (Fig. 3 (c)). After annealing at 900 °C, the dislocation cellular structure completely disappears, the distribution of dislocations becomes chaotic,

**Figure 2.** Changing of cellular structure upon annealing 400 (a), 600 (b), 800 (c), 1000 (d) and 1200 °C (e).
however, the dislocation density remains quite high (Fig. 3 (d)). In addition, in Figure 3 (e, f), which correspond to annealing at 900 and 1000 °C, a mosaic structure observes. Such a structure corresponds to the process of polygonization [13]. Structure changes attributed to the primary recrystallization process, were not noticed at these annealing temperatures.

![Figure 3](image)

**Figure 3.** Changing of cellular structure of austenitic alloy upon annealing: a – 500, b – 600, c – 700, d, e – 900 and f – 1000 °C.
Thus, the cellular structure formed during SLM has high thermal stability, being preserved up to annealing at 800 °C. Presumably, such a high structural stability is explained by the stabilization of the dislocations plexus by the segregation of alloying elements and impurity atoms. The dislocation structure restructuring, presumably, begins when increased diffusion mobility of atoms leads to the dissolution of segregations at dislocation boundaries. High thermal stability results in observing of polygonization process at the temperatures of more than $0.55 \cdot T_m$ ($T_m$ – the melt temperature) [K]. As a rule, the process of primary recrystallization finishes at such temperatures.

Figure 4 shows the dependence of the austenitic alloy microhardness on the annealing temperature. The austenitic alloy subjected to SLM with the process parameters used in this study has a hardness one and a half times higher than the alloy after standard quenching in water. The microhardness of alloy after SLM is 265 HV0.05, and the one after quenching is 190 HV0.05. At annealing temperatures up to 800 °C, the alloy hardness does not change within the measurement error. With a further increase of the annealing temperature, a smooth decrease of hardness begins. Even after annealing at 1000 °C, the hardness of the alloy remains at 225 HV0.05, which is 20% higher than the hardness of the quenched alloy. Annealing at 1160 °C results in the same microhardness of the experimental alloy and the quenched one.

![Figure 4. Dependence of the austenitic alloy microhardness on the annealing temperature](image)
Thus, after SLM, an alloy with a cellular structure with enhanced strength properties forms. Structural analysis showed that there are several hardening mechanisms acting in the alloy at once. Dislocation mechanism—a high density of dislocations arose in the alloy due to high thermal stresses during cooling and formed a dislocation cellular structure; solid-solution mechanism—alloying elements and gas impurities (O and N) dissolved in austenite at re-melting, presumably, are located not only in the $\gamma$-phase lattice in the form of single atoms, but also form segregations on dislocation plexuses; grain boundary hardening—crystallization cells with a size of about 0.5 μm act as grain here.

As long as the cellular structure stabilized by segregation of impurity atoms is preserved in the alloy, its microhardness remains practically unchanged. A drop of hardness starts with dislocation repartitioning. But as after the cellular structure repartitioning a high dislocation density is retained (Fig. 3 (d)), the hardness value also remains high. A further drop of microhardness with increasing annealing temperature is associated with the dislocation density to decrease.

Conclusions

1. During the SLM process of the austenitic alloy Fe-17% Cr-12% Ni-2% Mo-0.02% C, a cellular structure similar to the deformation cellular structure is formed: the cell boundaries are volume plexes of dislocations, while the dislocation density inside the cells is significantly lower.
2. Using the method of X-ray microspectral analysis, it was found that the concentration of Cr and Mo at the boundaries of dislocation cells is higher than in their center. Presumably, these alloying elements form segregations on dislocation plexuses.
3. Using the method of reductive burning, it was found that the concentration of oxygen and nitrogen in the re-melted alloy is an order of magnitude higher than in the initial powder: 0.10% O and 0.16% N.
4. Using structural analysis methods, it was shown that the cellular structure is preserved in the alloy up to annealing at 800 °C. At 900 °C, dislocations are randomly distributed in the alloy, and the process of polygonization is observed.
5. The microhardness of the alloy remains elevated till the annealing temperature of 800 °C, further it begins to decline smoothly.

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