Investigation of the Microstructure of Samples of the 08CrNi53MoNbTiAl Nickel-Base Alloy Obtained by Selective Laser Melting

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Abstract. Nickel-base alloys are the most common high-temperature materials. They are widely used for the manufacture of parts operating under high temperatures and oxidizing environments. However, in the practical application of these high-strength alloys, there is a problem of obtaining from them products of complex shape. This problem is caused by their low processability due to the extremely narrow temperature-velocity range of plastic deformation, and also due to the complexity of the machining. One of the effective ways to produce metal products without machining is the use of additive technologies. In this work, the structure and mechanical properties of samples from a high-temperature nickel-based 08CrNi53MoNbTiAl (Russian analogue of Inconel 718) obtained by selective laser melting. The influence of parameters of the selective laser melting process on the formation of products was studied, samples for mechanical tests and metallographic studies were grown. Images of the microstructures of the samples are obtained and defects in the samples are determined. Measurements of microhardness, roughness of samples, mechanical tensile tests and impact strength were also carried out.

1. Introduction
The Inconel 718 alloy is one of the most common high-temperature alloys. Complex parts from the Inconel 718 alloy can be efficiently produced using selective laser melting (SLM) technology. To date, in Russia this technology is not as developed as in other countries, so foreign SLM installations are used in production. Almost all manufacturers supply modes for working with recommended, often of their own production[1]. With the aim of import substitution, many powder metallurgy plants in Russia produce analogues of foreign powders, but obtaining high-quality products by the method of selective laser melting is an individual task for each material, requiring the study of their microstructure and properties.

2. Materials and methods of research
The structure and mechanical properties of the samples were carried out in the Institute of Laser and Welding Technologies SPbPU (ILWT). The experiment on selective laser melting technology was carried out in the Center of Additive Technologies “Osnova” using the Concept Laser M2 Cusing (Germany) (see figure 1). The unit is equipped with a 400W diode-pumped ytterbium fiber laser and a wavelength of 1069 nm, with a 220 × 220 × 280 mm construction zone.
Nitrogen was used as an inert medium for growing the samples. The oxygen content during the construction process was < 0.15%. Cultivation was carried out with a heating platform construction in 200°C.

When constructing a part layer, the "skin and core" processing strategy was used (see figure 2). The laser spot passes a layer of material with two processing strategies[2]. First, there is the "Skin" strategy - bypassing the outer contour of the part, equal to the diameter of the laser spot. Then the core layer is filled - the "Core" strategy. Each subsequent layer of the hatching direction was rotated by 90 °.

The chemical composition, the morphology of the powder particles and the fracture after mechanical tests were studied using a Phenom ProX scanning electron microscope. The preparation of the polished sections for metallographic studies was carried out on a Mecatech 234 grinding and polishing machine. The optical microscope Metam LV-31 (LOMO) was used to determine the defects in the samples and also to study the microstructure of the etched samples. Mechanical tests of tensile samples were carried out on a Zwick / Roell Z100 tensile machine in accordance with GOST 1497-84. The test temperature is 23 °C. Impact specimens were tested on a Novatronic Impact 450U falling-tup machine. The roughness of the samples was determined using a Hommel-Etamic W10 roughness tester. The microhardness was measured on a FM-310 microhardnesser.

Figure 3 shows the surface of the particle of the powder being studied and the points at which the chemical analysis was performed are marked. The powder has a particle size of up to 40 μm and a spherical particle shape. Some particles are covered with small satellites. Table 1 shows the results of measurements of the chemical composition of the powder.
Figure 3. The surface of the powder of 08CrNi53MoNbTiAl

Table 1. The chemical composition of the powder

|   | Fe  | Ni  | Cr  | Mo  | Nb  | Ti  | Al  | Mn  | Co  | Si  |
|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 1 | 150 | 580 | 2   | 830 |     |     |     |     |     |     |
| 2 | 700 | 1,7 | 900 | 340 | 2   |     |     |     |     |     |
| 3 | 810 | 1,5 | 970 | 370 | 2,9 |     |     |     |     |     |
| 4 | 580 | 2,5 | 3,1 |     |     |     |     |     |     |     |
| 5 | 700 | 2   | 80  | 25  | 370 | 90  | 25  |     |     |     |
| 6 | 810 | 1,8 | 970 | 370 |     |     |     |     |     |     |
| 7 | 580 | 2,9 |     | 830 | 3,4 |     |     |     |     |     |
| 8 | 700 | 2,4 |     | 900 | 3,1 |     |     |     |     |     |
| 9 | 810 | 2   |     | 970 | 2,9 |     |     |     |     |     |

The main parameters of the selective laser melting process are the laser power $P$, the scanning speed $v$ and the radius of the laser beam $r$, then to select the regimes we used the formula $[3]$

$$E_p = \frac{p}{\pi r^2} \cdot \frac{2r}{v} \quad [J/mm^2]$$

Table 2 shows the experimental regimes.

Table 2. The experimental regimes

|   | P, W | $V$, mm/sec | $E_p$, J/mm$^2$ | $r$, micron | $h$, micron | P, W | $V$, mm/sec | $E_p$, J/mm$^2$ | $r$, micron | $h$, micron |
|---|------|-------------|-----------------|-------------|-------------|------|-------------|-----------------|-------------|-------------|
| 1 | 580  | 2           | 830             | 2           | 90          | 2,9  | 830         | 2,9            | 90          | 25          |
| 2 | 700  | 1,7         | 900             | 2           | 970         | 2,7  | 970         | 2,7            | 90          | 25          |
| 3 | 810  | 1,5         | 970             | 2,9         | 370         | 2,4  | 370         | 2,4            | 970         | 2,9         |
| 4 | 580  | 2,5         | 3,1             | 90          | 370         | 3,4  | 370         | 3,4            | 970         | 3,4         |
| 5 | 700  | 2           | 80              | 25          | 2           | 80   | 25          | 25             | 2           | 2           |
| 6 | 810  | 1,8         | 970             | 2,9         | 370         | 3,1  | 370         | 3,1            | 970         | 3,1         |
| 7 | 580  | 2,9         | 3,4             | 90          | 370         | 2,7  | 370         | 2,7            | 970         | 2,7         |
| 8 | 700  | 2,4         | 900             | 3,1         | 370         | 2,9  | 370         | 2,9            | 970         | 2,9         |
| 9 | 810  | 2           | 970             | 2,9         | 370         | 3,4  | 370         | 3,4            | 970         | 3,4         |

3D models of tensile test specimens (GOST 11701-84) and impact strength (GOST 9454-78) were made in the CAD software SolidWorks. As samples for metallographic studies, cubes with a side of 5 mm were chosen. Further in the Materialize Magics program, models were prepared for cultivation: the location of the samples on the platform, arrangement of support structures and slicing.

Figure 4 shows the location of the samples on the platform.
3. Results and discussion
The results of some of the samples studied contain defects such as pores (see figure 5(a)) and non-melting (see figure 5(b)). The appearance of non-melting is associated with low power and a sufficiently high scanning speed, which had a bad effect on the formation of the fused surface. The appearance of pores is due to overheating of the material above the boiling point[4].

![Figure 4](image_url)

**Figure 4.** The samples on the platform (a) tensile and impact strength specimens (b) samples for metallography

The microstructure of all samples is similar and typical for the SLM process. On the sections, directly crystallized dendritic cells are visible, which propagate over several layers (see figure 6(a)). Melt baths have a convex shape, their depth depends on the scanning speed and is 50-70 microns, which indicates the repeated re-melting of the previous layer. This is necessary to ensure high bond strength and leads to the fact that most of the grains in the product have a shape elongated along the growing axis (see figure 6(b)).

![Figure 6](image_url)

**Figure 6.** The microstructure of all samples (a) dendritic cells (b) elongated grains

Table 3 shows the results of the tensile tests. Table 4 shows the results of the impact test.
Table 3. The results of the tensile tests

|    | $\sigma_{0.2}$, MPa | $\sigma_b$, MPa | $\delta$, % |
|----|-------------------|----------------|-------------|
| 1  | 739.5214          | 979.0536       | 7.142589    |
| 2  | 736.1847          | 992.9805       | 9.034075    |
| 3  | 727.1811          | 964.7872       | 7.549917    |
| 4  | 695.0501          | 964.713        | 9.420031    |
| 5  | 723.2711          | 1022.859       | 16.16914    |
| 6  | 718.9966          | 1004.161       | 14.01592    |
| 7  | 667.4073          | 953.0903       | 15.76844    |
| 8  | 688.174           | 976.8159       | 14.69701    |
| 9  | 692.3296          | 976.2918       | 15.51601    |

Table 4. The results of the impact test

| KCU, J/cm$^2$ | vertical construction | horizontal construction |
|---------------|------------------------|-------------------------|
| 1             | 40,1                   | 39,0                    |
| 2             | 40,4                   | 33,2                    |
| 3             | 42,2                   | 26,8                    |
| 4             | 37,2                   | 48,5                    |
| 5             | 40,9                   | 36,4                    |
| 6             | 43,0                   | 38,0                    |
| 7             | 34,1                   | 27,4                    |
| 8             | 39,2                   | 39,0                    |
| 9             | 40,5                   | 41,6                    |

The results of the impact tests showed that anisotropy is present in the samples, the average impact strength of vertical samples is higher than that of horizontal ones. The difference in the strength properties of the samples can be related to the direction of grain growth during SLM and the propagation of defects along the grain boundaries.

To further investigate the mechanism of failure, images of fracture patterns after impact tests were obtained (see figure 7). In some samples there are pores and cracks, which affected the magnitude of the toughness. A common feature of all samples is the presence of pits on the entire surface of the fracture, as well as the absence of metallic luster, which indicates a viscous fracture[5].

![Figure 7. Images of fracture patterns after impact tests (a) x2500 (b) x1000](image)

Table 5 shows the results of microhardness measurements. Table 6 shows the results of measurements of the roughness.
Table 5. The results of microhardness measurements

|   | HV   |
|---|------|
| 1 | 333  |
| 2 | 314  |
| 3 | 282  |
| 4 | 308  |
| 5 | 293  |
| 6 | 321  |
| 7 | 294  |
| 8 | 297  |
| 9 | 309  |

Table 6. The results of measurements of the roughness.

|   | Ra   |
|---|------|
| 1 | 1.864|
| 2 | 1.540|
| 3 | 1.473|
| 4 | 2.871|
| 5 | 2.293|
| 6 | 2.132|
| 7 | 3.986|
| 8 | 3.838|
| 9 | 3.198|

The roughness values decrease with decreasing power and increasing the speed, since in this mode the spraying of the material is minimal.

4. Conclusions

The results of metallographic studies have shown that in some samples there are pores and fusions. The appearance of pores is associated with the evaporation of the powder material due to overheating above the boiling point. Low power and high scanning speed are poorly reflected in the formation of the fused surface. For all samples, a similar microstructure is typical, which is a directed crystallized dendritic cell, which propagates to several layers. Melt baths have a convex shape. The depth of the melt baths is 50-70 μm, which indicates the remelting of the previous layer. This led to the fact that most of the grains in the samples have an elongated shape along the axis of cultivation. The morphology of the melt baths depends mainly on the speed of laser scanning.

The yield point decreases with increasing laser radiation power, and the change in scanning speed is practically unaffected. The increase in power leads first to an increase in the tensile strength of the samples, and then to a decrease. The same is the effect of speed. The lowest elongation is found in specimens grown at minimum power. At a power of 400 W, the samples have a high elongation value, which practically does not vary with speed. The best mechanical characteristics are samples № 5 and 6. The average value of the impact strength of vertical samples is higher than that of horizontal ones. The difference in the strength properties of the samples can be related to the direction of grain growth in SLM and the propagation of defects along the grain boundaries. To eliminate the anisotropy of properties, heat treatment is required. A common feature of all samples is the presence of pits on the entire surface of the fracture, as well as the absence of metallic luster, which indicates a viscous fracture. The highest values of microhardness are samples № 1 and 6. The minimum roughness values are those grown at the minimum power and maximum speed, since under this regime the spraying of the material was minimal. The increase in power leads to an increase in roughness.
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