Investigation of the effect of HIP processing on the physical and mechanical characteristics of parts obtained by the SLM method

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Abstract. In this paper presents the results of studying the powder material AISI 321. Selective laser melting (SLM) of the samples was carried out in modes with a change in the radiation power. The subsequent processing of the samples by the method of hot isostatic pressing (HIP) was carried out. The roughness of the surfaces and the microhardness of the samples before and after the HIP were studied.

1. Introduction
The increasing pace of introduction of additive technologies leads to the use of additional methods of processing manufactured products [1,2]. Selective laser melting, as a method of additive manufacturing, provides wide opportunities for the production of products from various purposes from a wide range of materials.

Among other factors, the properties and defects of the initial powder affect the characteristics of the final material. The presence of such defects as satellites and ellipsoid, oval shape negatively affect the transfer of the powder with a squeegee and the density of the package [3-5].

At the same time, hot isostatic pressing is used as one of the methods of subsequent processing of SLM products. The essence of the method consists in the prolonged exposure of elevated temperature and pressure to products located in a chamber filled with an inert gas. There are works [6-10] on hot isostatic pressing of products after SLM, in which a decrease in the porosity of the material is noted. There is also evidence that in some modes of the HIP there is a change in the structure of the material, leading to a change in the strength characteristics.

Also, in many works, the dependence of the internal and surface characteristics on the volumetric energy density is indicated [11-13]. In this work, we investigated the surface roughness after SLM and after HIP, as well as the microhardness of the side surfaces in 3 different regions.

2. Materials and methods
2.1 Study of the shape, surface of granules and internal porosity of granules of the starting material
The shape, as well as the surface of the granules of the initial powder, affects the fluidity and, as a consequence, the uniformity of the deposition of layers during selective laser melting. On the other hand, the same characteristics of the starting material affect the bulk density of the powder in each applied layer, which ultimately affects the porosity, as well as the presence of pores within the powder granules.
When studying the shape and surface of the granules of the AISI 321 powder material, images obtained from a scanning electron microscope were used. Based on the images obtained, the sphericity of the powder granules was assessed, as well as the identification of satellites.

The study of the internal porosity of the granules of the powder material was carried out on the basis of micrographs of a previously prepared thin section using optical microscopy. The preparation of the section was carried out as follows: the initial powder was poured with epoxy resin, then the material was ground and polished, after which the remnants of the polishing paste were removed from the surface of the section using a solvent.

The study of defects in the original powder material was carried out based on information from GOST R 58418-2019 [14].

2.2 Obtaining samples by selective laser melting. Post-processing by hot isostatic pressing.

Selective laser melting was carried out on a Concept Laser 2M complex. The equipment is equipped with an ytterbium fiber laser emitting at a wavelength of 1.07 µm, the spot diameter in the treatment zone was 100 µm. Other characteristics of the equipment are shown below in table 1.

| Table 1. Equipment parameters |
|-------------------------------|
| Dimensions of the printable area | 250x250x300 mm |
| Maximum radiation power | 400 W |
| Maximum surface scanning speed | 4000 mm/s |
| Layer thickness range | 20-80 micron |

The samples were made in the form of a cube with sides 15x15x15 mm. The samples were synthesized in an inert nitrogen atmosphere. The oxygen percentage in the chamber was 0.6-0.8%. The layer thickness during synthesis was 30 µm, the shading step was 150 µm. With the parameters described above, a number of samples were obtained with a fixed scanning speed and different values of the laser radiation power (Table 2) without additional processing along the cross-sectional contour. Additional heat treatment in the furnace was not performed.

| Table 2. Powder material processing parameters |
|-----------------------------------------------|
| Sample number | Processing parameters | EDv, J/mm³ |
|----------------|----------------------|----------|
| 1              | 150 W, 1500 mm/s     | 22.23    |
| 2              | 200 W, 1500 mm/s     | 29.63    |
| 3              | 250 W, 1500 mm/s     | 37.04    |
| 4              | 300 W, 1500 mm/s     | 44.45    |
| 5              | 350 W, 1500 mm/s     | 51.85    |

The processing parameters were recalculated according to the formula in the volumetric energy density EDv [15] (J/mm³):

$$EDa = \frac{P}{v*h*t}$$  \hspace{1cm} (1)

where P is the radiation power, v is the laser scanning speed, h is the shading step, t is the layer thickness.

A similar number of samples obtained with the parameters indicated in Table 2 were post-processed by the HIP method. The HIP parameters were as follows: temperature - 950 °C, pressure - 1500 bar, holding time - 20 hours.
2.3 Measurement of surface roughness of samples before and after HIP

The surface roughness Ra was measured using a Mitutoyo Surftest SJ-201 P profilometer. The measurements were carried out for a surface parallel to the construction plane (XY) and a surface perpendicular to the construction plane (YZ). The measurement scheme is shown in Figure 1.

![Figure 1. Scheme for measuring surface roughness](image)

On the obtained samples, at least 10 measurements were carried out for one side and a similar number for the other. Then the average value of the surface roughness XY and YZ was calculated. The same measurements were carried out for samples after HIP.

2.4 Measurement of microhardness of samples before and after HIP

The measurement was carried out on a PMT-3 microhardness tester according to the Vickers method by pressing a diamond tip in the form of a tetrahedral pyramid with a square base. The applied load was 0.4903325 Newtons. Determination of microhardness was carried out for the YZ plane corresponding to the lateral side of the samples (Figure 2).

![Figure 2. Scheme for determining microhardness](image)

The measured side was divided into 3 areas: at the base of the sample, in the middle and at the top of the sample. For each area, 5 measurements were carried out. Similar actions were performed for samples after HIP.
3. Results and Discussion

3.1 Study of the shape, surface of granules and internal porosity of granules of the starting material

Particle images obtained with a scanning electron microscope are shown in Figure 3.

Figure 3. SEM images of granules of powder material

A small number of ellipsoidal particles are noticeable in the images, the main part of which is close to spherical. In addition, satellites and deformed powder particles are observed.

A section of granules of powder material is shown in Figure 4. On micrographs inside the particles, cracks and a small number of pores not exceeding 1 µm in diameter can be found.

Figure 4 - Micrographs of a thin section of powder granules

3.2 Sample Roughness Measurement Results

Figure 5 shows a diagram of the dependence of the lateral surface roughness (XY plane) on the volumetric processing energy for samples after SLM and after HIP.
Figure 5. Diagram of the dependence of the lateral surface roughness on the volumetric processing energy

When considering the roughness of the upper surface only after SLM, the frequency of increasing and decreasing of the indicator is noticeable with an increase in the volume energy density. This may be due to the heat transfer from the sample layers to the surrounding particles. The smallest obtained roughness value is equal to 13.02 microns on the mode 29.63 J/mm$^3$.

When comparing the data before and after HIP, a slight overall decrease in the roughness of the lateral surfaces of the samples is noticeable. Presumably, this could be due to the deposition of carbon from the tooling, which is used during the HIP on the surface of the samples.

Figure 6 shows a diagram of the dependence of the roughness of the upper surface (YZ plane) on the volumetric processing energy for samples after SLM and after HIP.

Figure 6 - Diagram of the dependence of the roughness of the upper surface on the volumetric processing energy

When considering the roughness of the upper surface only after SLM, reducing the value is noticeable at 29.63 J/mm$^3$, followed by a further increase in the measured indicator. The next reducing
the value is noticeable on the mode 51.85 J/mm³. The smallest obtained roughness value is equal to 12.51 microns on the mode 29.63 J/mm³.

Figure 6 shows a significant decrease in the roughness of the upper surface on a specimen with an energy of 22.22 J/mm³ after HIP, further the roughness increases. At the same time, practically no changes were observed on the sample with an energy of 29.63 J/mm³.

3.3 Results of measuring the microhardness of samples

The measured values of microhardness were entered into a table and averaged, after which a diagram was drawn up for modes and regions on the surface of the samples. The diagram is shown in Figure 7.

![Figure 7. Diagram of the dependence of microhardness on the volumetric energy density (SLM)](image)

Figure 7 shows the microhardness data in the range of modes for the lower, middle and upper parts of the samples. In the lower part of the samples, the microhardness decreases with increasing energy, and then the index increases. In the middle part, the readings are more stable and homogeneous in all modes. In the upper part, a slight drop was noticed, then an increase in the regime with an energy of 44.44 J/mm³ and an alignment with the indicators of the middle. The highest value was recorded for the mode with an EDv of 44.44 J/mm³ in the upper segment of the sample and was 257 HV.

The diagram for samples after HIP is shown in Figure 8.

![Figure 8. Diagram of the dependence of microhardness on the volumetric energy density (HIP)](image)
An increase in microhardness is observed at an energy of 44.44 J/mm³ over the entire area of the sample, followed by a further decrease in the index. At the same time, a general increase in microhardness was observed for the samples in each of the modes. The highest value of the microhardness value was recorded at EDv 44.44 J/mm³ for the middle region of the sample and was 333 HV.

4. Conclusions

The study of the powder material AISI 321 was carried out, the shape of the particles, the presence of a small number of satellites and deformed granules of the powder powder were revealed. The shape of the particles is predominantly close to the reference, there are ellipsoidal granules.

Samples of AISI 321 steel were obtained by selective laser melting. The synthesis of the samples was carried out at a constant laser scanning speed and a variable radiation power.

Data were obtained on the roughness of the surfaces of the samples depending on the volumetric energy density before and after the HIP. The data show that as the energy increases, the surface roughness decreases, which may be due to different amounts of energy transferred. In addition, a slight decrease in roughness after HIP is recorded, this could be due to the deposition of carbon from the tooling, which is used during HIP on the surface of the samples.

Measurements of the microhardness of the samples on the obtained samples were carried out before and after the HIP. An increase in microhardness after HIP is noted for all modes of sample synthesis.

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