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# Improving the Strength of Steels Produced Using a New Type of Laser Melting Technology

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**Annotation**: The purpose of this work was to establish the causes of the inhomogeneity of the chemical compos ition of the metal obtained by the SLM technology. A powder raw material was produced from a monolithic alloy, followed by its fusion by the SLM method, the metal of laboratory melting of a low-carbon chromium-manganese-nickel composition based on iron served as the initial raw material. To determine the nature of the distribution of alloying chemical elements in the manufactured powder, electron microscopic images of thin sections were combined with X-ray spectral analysis data on cross sections of powder particles. As a result, it was found that the transition (Mn, Ni) and heavy (Mo) metals are uniformly distributed over the cross sections of the powders, and the mass fraction of silicon (Si) is uneven: in the center of the particles, in some cases, it is several times larger. The revealed feature in the distribution of silicon is presumably due to the formation of various forms of SiO4 during cooling of the resulting particles.

The internal structure of the manufactured powder is represented by a martensitic structure of packet morphology. After laser fusion, traces of segregation inhomogeneity in the form of a grid with cells of ~200 μm were revealed on etched sections. Segregation boundaries and fine-grained structure were the dominant mechanisms of steel hardening in the SLM process.

Under the conditions of compression of the samples obtained, the yield strength was 720 MPa, and the highest value deformation resistance reached 1050 MPa, which exceeds the performance of a monolithic material of a similar chemical composition. On the diagrams σ(ε) in the section of parabolic hardening, randomly located extrema of local hardening, followed by abrupt load drops. The shape of the diagrams indicates the inhomogeneous internal structure of the samples.

**Keywords:** additive technologies; powder materials; fusion; hardening; chemical heterogeneity.

**Introduction.** The manufacture of products of complex shape by laser selective melting (LSM* technology) of layer-by-layer applied powder raw materials is of practical and scientific interest [4, 6, 9-11, 13, 14]. In experiments on the fusion of powders of similar granularity industrial production (JSC Polema, JSC Höganäs) with the chemical composition of common steel grades (07Kh16N4D4B, 03Kh16N15M3, 12Kh18N10T, etc. [1, 2, 8, 12, 15]), an increase in the strength of final samples in comparison with the monolithic state of the metal of the same chemical composition. One of the reasons for the increase in strength was the chemical inhomogeneity of the alloyed composition. However, it remained unclear at what technological stage the chemical inhomogeneity arose?

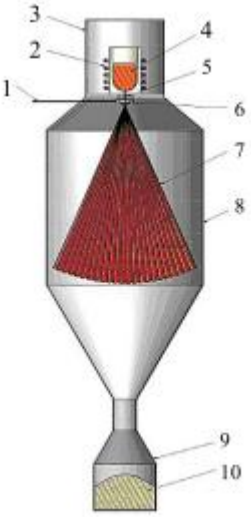
The aim of the work was to establish the root causes of the inhomogeneity of the chemical composition of the metal in the SLM technology. To achieve this goal, it was necessary to manufacture powder raw materials from a monolithic alloy of a given chemical composition in laboratory conditions, followed by its fusion by the SLM method.[23.,24]

**Method of work.** The metal of experimental laboratory melting of low-carbon (C~0.09 wt.%) iron-based chromium-manganese-nickel composition was used as the feedstock. The laboratory ingot and the manufactured powder have the following chemical composition (wt.%), respectively: Mn (0.38; 0.35), Si (0.26; 0.3), Cr (0.41; 0.45), Ni (1.93; 1.88), Mo (0.25; 0.24).[20-22]

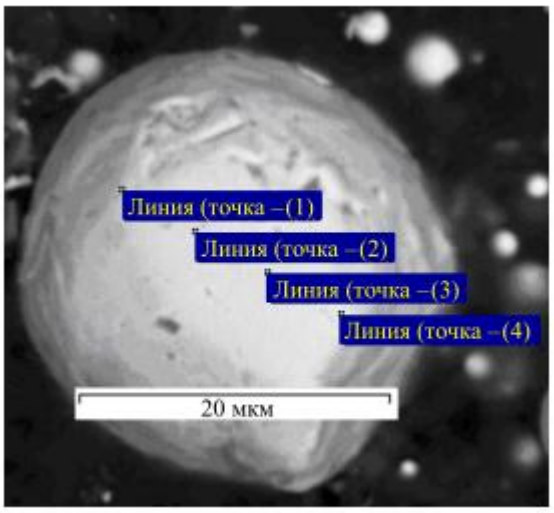
At all stages of manufacturing and subsequent testing of samples, the chemical composition of the metal was controlled by X-ray spectral analysis. According to the averaged data, the chemical composition of the powder practically coincided with the chemical composition of the laboratory ingot.

The powder intended for selective laser melting is made on a HERMIGA 75/IV facility (Fig. 1) by melt sputtering (atomization) at a temperature of 1650 $^{0}$ C in an argon atmosphere followed by cooling at rates from $10^{5}$ to $10^{8}$ $^{0}$ C/s.

The resulting powder was characterized by a spherical shape and a particle size of up to 200 μm. Next, the powder fraction suitable for use in SLM technology (< 80 μm) was screened out, the proportion of suitable powder was 70%. The SLM process was implemented on the EOSINT M270 installation (Fig. 2), which, according to its technical parameters, ensured the complete melting of powders in sequentially applied layers of manufactured raw materials.




a )




b)

Fig.1. Installation HERMIGA 75/IV for atomization of the melt (a) and a representative powder with indication of the points for determining the chemical composition of the metal (b)

1 – spray gas supply; 2 - inductor rings; 3 - induction furnace; 4 - molten metal; 5 - metal outlet tube;

6 - spray nozzle; 7 - fan of sprayed metal; 8 - spray chamber; 9 - receiving container; 10 - metal powder

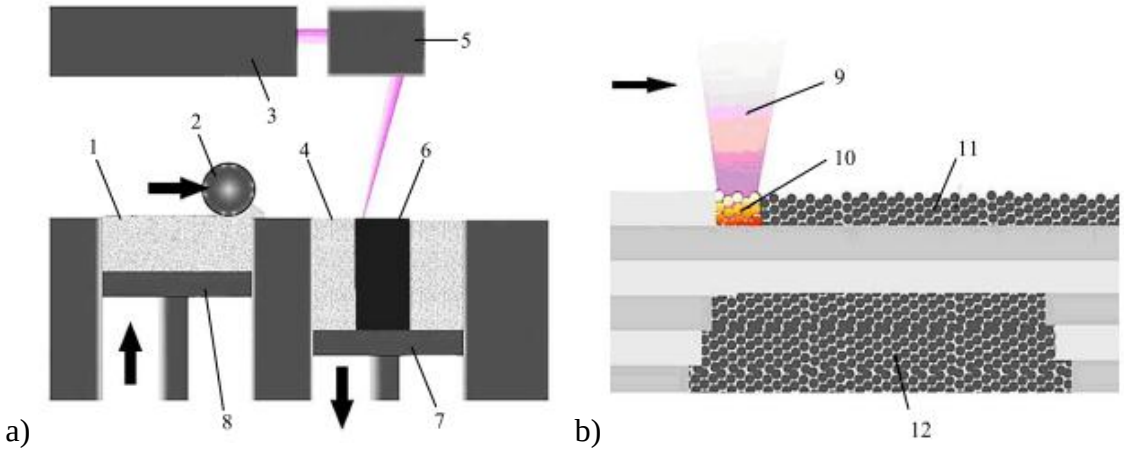



Fig.2. Scheme of implementation of the SLM technology on the EOSINT M270 facility

1 – initial powder; 2 - feed roller; 3 – laser; 4 - powder without fusion; 5 – scanning system; 6 – model after powder fusion; 7 - control system; 8 – power supply system; 9 – laser beam; 10 - zone of selective melting;

11 - Previous layer of powder; 12 – SLS model material

The spherical shape of the particles indicated that the sputtered metal condensed according to the vapor → liquid scenario [3]. Depending on the cooling conditions, the liquid could crystallize into a crystal or remain in a supercooled (partially amorphous) state.

In the experiment, samples in the form of cylinders 6 mm in diameter and 10 mm high were grown from the obtained powder. The construction of the samples was provided by scanning a laser beam with a specific energy of 0.2-0.3 J/mm of the construction area with the introduction into the powder layers. The construction of samples was carried out in layers.

The fabricated samples were tested in the chamber of a Gleeble 3800 power simulator by mechanical compression at room temperature to a true strain $\varepsilon = 0.3$ at a strain rate of 10–3 s–1. The compression test made it possible to level the effect of possible lack of penetration and voids on the strength indicators of the samples.

The SLM structure of the metal was studied by metallography on thin sections using light (Axiovert 4MAT) and electron scanning (Teskan Vega 3) microscopes.

**Discussions.** In order to determine the nature of the distribution of alloying chemical elements in the manufactured powder, the electron microscopic images of thin sections were combined with the data of X-ray spectral analysis on the cross sections of the powder particles. The distribution of alloying elements (Cx) was estimated in relative units (mass fractions) with respect to the base - iron: Cx/Fe.

It has been established that transition (Mn, Ni) and heavy (Mo) metals are uniformly distributed on the cross sections of the powders, regardless of the size of the probed area (Fig. 3, 4). The mass fraction of silicon is unevenly distributed: in the center of the particles, the content of silicon in some cases is several times higher than its presence in the peripheral areas. This suggests that silicon, having an affinity for oxygen, under conditions of high temperatures actively diffuses towards the outer surface of the particles and interacts with the environment, forming oxides.

Under high-temperature conditions, several forms of silicon-containing melts are known, which differ in the mutual arrangement of $SiO_4$ tetrahedra [7]:

β-cristobalite (1710 $^0$ C), β-tridymite (1470 $^0$ C), β-quartz (870 $^0$ C), α-quartz (573 $^0$ C), [17-19]etc., with macromolecular covalent bonds in the solid state. It can be assumed that the revealed feature in the distribution of silicon is due to the formation of various forms of $SiO_4$ upon cooling of the formed particles. At the same time, their internal structure can affect the crystallization kinetics and the liquid-solid interaction of contacting powder particles in the laser impact zone.

The noted artifact can explain the excess of the increase in the strength of the alloy due to the concentration factor (Cx) in comparison with the strain hardening [5]: $d\sigma/dCx > d\sigma/d\gamma$, where $\gamma$ is the shear strain.

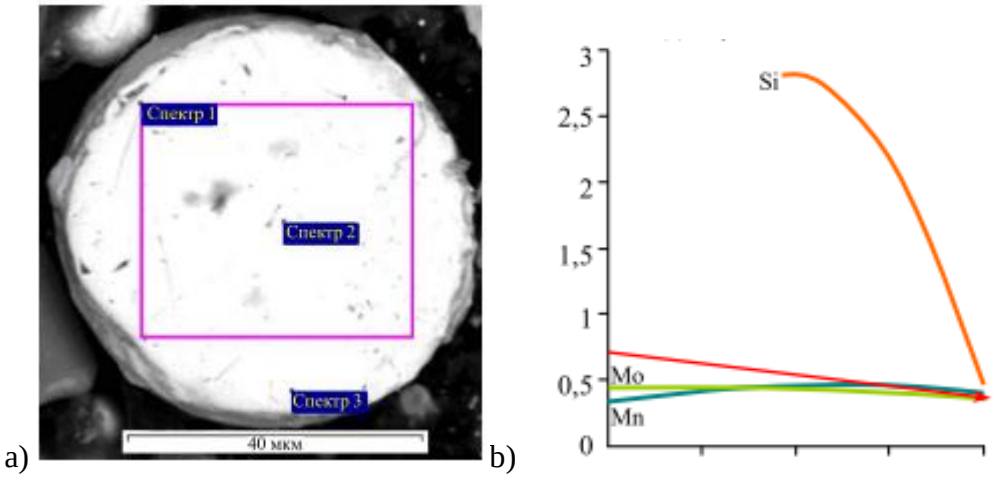



Fig.3. A section of a powder with an indication of the area and local places of X-ray spectral analysis (a), the distribution of Si, Mn and Mo depending on the size and places of X-ray spectral probing (b)

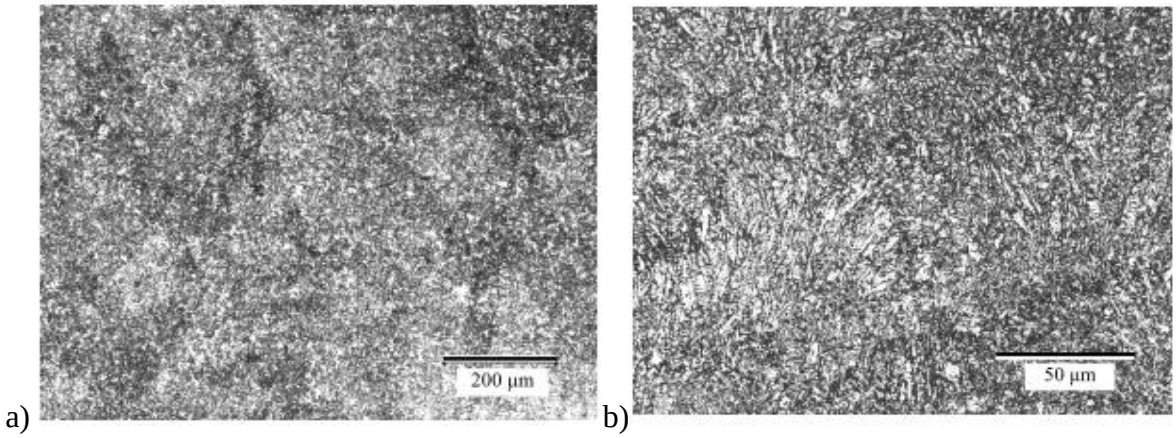



Fig.4. The structure of steel produced by SLM: segregation cells up to 200 μm in size (a) and unequal dendritic type crystals ~5 × 50 μm in size and equiaxed grains of a micrometer scale (b)

The internal structure of the manufactured powder is represented by a martensitic structure of packet morphology. After laser fusion, traces of segregation inhomogeneity in the form of a grid with cells of ~200 μm were revealed on etched sections. Inside the cells there are non-equiaxed (5 × 50 μm) crystals of a dendritic type and different orientations, as well as a mass of grains 3–5 μm in size (Fig. 4). It must be assumed that segregation boundaries and a fine-grained structure were the dominant mechanisms of steel hardening during SLM.

The formulated thesis was confirmed by the results of mechanical tests.

It was established (Fig. 5) that under the conditions of compression of the obtained samples, the yield strength was 720 MPa, and the highest value of the deformation resistance reached 1050 MPa, which exceeds the similar indicators of a monolithic material of a similar chemical composition. On the diagrams σ(ε) in the area of parabolic hardening, chaotically located extrema of local hardening are fixed, after which sharp load drops follow. The appearance of the diagrams testified to the inhomogeneous internal structure of the samples.

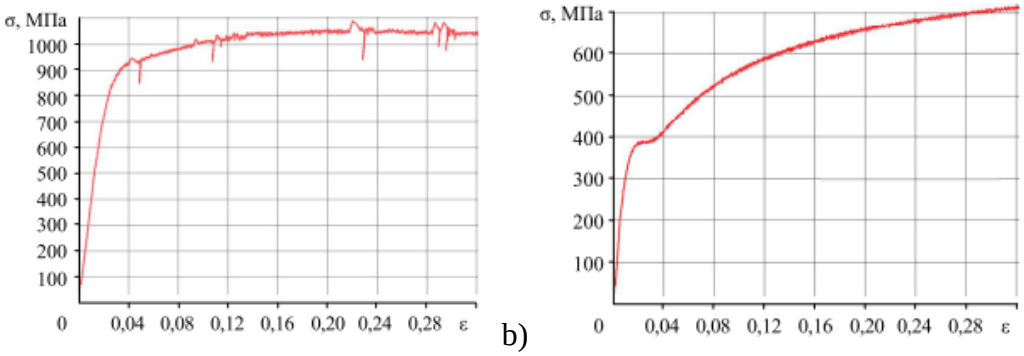



Fig.5. Diagrams of resistance to plastic deformation of steel specimens in the following states: initial after SLM (a); after SLM and subsequent heat treatment at 980 $^0$C for 300 s (b)

Annealing of the obtained samples at a temperature of 980 $^0$C for 300 s in the structural state of a face-centered cube (FCC) followed by cooling at a rate of ~6 $^0$C/s leveled the structural inhomogeneity of the initial state, which caused a decrease in the strength properties of the metal by 1.5 times. In addition, at a level of σ = 400 MPa, a yield plateau appeared, which is typical for steels with a body-centered cube (BCC) structure, due to the effect of strain aging.

**Conclusions.** Based on the results of the work done, the following can be concluded.

1. The chemical inhomogeneity of the powder produced by the spraying method arises at the stage of melt atomization.
2. To implement a technological process with complete melting of powder layers, the particle size should not exceed the thickness of the applied layer, provided for by the technical documentation of the equipment used with a concentrated (laser or other) energy source.
3. For high-quality manufacturing of products using the SLM technology, the technological and morphological parameters of the powder raw materials used must be regulated.
4. During the implementation of the SLM process, the chemical inhomogeneity of powder raw materials can be aggravated due to the diffusion of chemical components towards the outer surface of the particles, followed by the formation of oxides.
5. The chemical inhomogeneity of the alloyed sample can be leveled by additional heat treatment at temperatures and times sufficient for the diffusion of liquates.
6. In comparison with monolithic metal materials, samples of the same chemical composition, manufactured using the SLM technology, are characterized by increased strength characteristics due to chemical heterogeneity and ultrafine crystallization structure.

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