Comparison of samples manufactured from 09CrNi2MoCu grade powder by selective laser melting with plate metal of the same grade

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Abstract. The research data on the composition, structure and properties of samples manufactured by the selective laser melting of steel powder 09CrNi2MoCu, and plate metal samples of the same grade are provided. In the melted specimens, an atypical structure for this grade was found, and also the excess of some mechanical characteristics over similar ones for specimens from plate metal was found.

Introduction

In the early 1980s, additive technologies for the formation of three-dimensional objects [1] began to develop intensively due to a gradual increase in the material or a change in the phase state of matter in a given region of space. Of these, at present, the technology of selective laser melting (SLM) has become widespread, consisting in the layer-by-layer formation of three-dimensional objects according to their computer models. The peculiarity of the process is the effect of the laser on the metal powder, as a result of which a monolithic product is formed.

The SLM technology allows to create several models at a time, while only limiting the size of the working chamber. The construction takes several hours, which is more profitable than the casting process. On the other hand, parts produced by laser melting can achieve the same strength indicators as moulded samples or parts produced by subtractive methods [2, 3].

The aim of this work is a comparative description of the structure and properties of samples made from 09CrNi2MoCu plate metal and obtained by SLM from an atomized powder of identical chemical composition.

Materials and experimental methods

The powder from steel grade 09CrNi2MoCu was obtained by melt spraying (atomization) on a HERMIGA 75/IV unit in an argon inert gas atmosphere. The granulometric composition of the powders was measured by laser diffraction using the Fraunhofer approximation on a Malvern Mastersizer 2000 unit. According to the results of particle size analysis, powder fractions of more than 80 µm were excluded, which are not suitable for use in SLM technology.

Non-destructive testing of the elemental composition was carried out using an Inka X-ray fluorescence device on a TESCAN Vega 5136-LM scanning electron microscope. Structural studies
were carried out on an Axio Observer A1m optical microscope with the Clemex Vision PE program and on a TESCAN Vega 3 electron scanning microscope.

In general, the chemical composition of the obtained powder corresponded to the steel grade 09CrNi2MoCu according to TC 5.961-11618-2010, however, some features were found. It is established that the chemical composition of particles on the surface and inside differs (table 1 and figure 1).

Table 1. Chemical composition of powders.

| Spectrum | Mn  | Si  | Cr  | Ni  | Cu  | Mo  | V  | Al  | Fe  |
|----------|-----|-----|-----|-----|-----|-----|----|-----|-----|
|          |     |     |     |     |     |     |    |     |     |
| On the particle surface |     |     |     |     |     |     |    |     |     |
| 1        | 0.520 | 0.720 | 0.400 | 1.980 | 0.670 | 0.460 | -  | 0.050 | 95.200 |
| 2        | 0.280 | 0.320 | 0.580 | 1.950 | 0.360 | 0.380 | 0.100 | 0.080 | 95.950 |
| 3        | 0.380 | 0.300 | 0.440 | 1.820 | 0.520 | 0.320 | 0.070 | 0.090 | 96.050 |
| 4        | 0.520 | 0.720 | 0.400 | 1.980 | 0.670 | 0.460 | -  | 0.050 | 95.200 |
| Average  | 0.420 | 0.510 | 0.450 | 1.930 | 0.550 | 0.400 | 0.042 | 0.068 | 95.600 |
| Inside the particle |     |     |     |     |     |     |    |     |     |
| Area 1   | 0.410 | 0.770 | 0.440 | 2.040 | 0.550 | 0.310 | 0.070 | 0.080 | 95.320 |

For example, the content of Mn, Si, Mo and V shows that these elements are concentrated in different layers of spherical particles. But the revealed effect is due to the conditions of X-ray imaging: points 2 and 3 in figure 1 correspond to the normal incidence of the electronic zone, and points 1 and 4 are close to the tangential direction. The greatest differences were found in the occurrences of Si (~0.4) and V (~0.09), of which the first is concentrated in the surface layers and the second in the depth of the particles.

Figure 1. Photographs of powder particles with marked areas for chemical composition studies on the surface (left) and thin section (right).

The melting of the obtained powders was carried out using the EOSINT M270 unit located at the experimental site Nanocenter of the NRC "Kurchatov Institute" – CRISM "Prometey". In this unit, a solid-state laser with a power of up to 200 W is capable of continuously scanning the melting zone of the powder at different speeds.

Standard samples for tensile testing (ASTM E8 standard) were manufactured. The sizes of samples corresponded to Type III No. 9. Tensile testing was performed on the Instron 3367 unit. The critical points of the Ac1, Ac3, and CTE phase transitions during heating were determined on a DIL 402C dilatometer.
Experimental results and discussion

After tensile testing, the properties of specimens from 09CrNi2MoCu plate metal in delivery condition (after quenching and high tempering) and specimens manufactured by the SLM (table 2) were obtained.

Table 2. Uniaxial tensile testing results.

| Samples       | $\sigma_y$, MPa | $\sigma_{UTS}$, MPa | $\sigma_y / \sigma_{UTS}$ | $\delta$, % | $\psi$, % |
|---------------|-----------------|---------------------|---------------------------|-------------|-----------|
| Plate metal   | 630.0           | 705.0               | 0.89                      | 22          | 73.0      |
| SLM           | 896.9           | 909.4               | 0.98                      | 7           | 43.5      |
| TC 5.961-11618-2010 | $\geq 637.0$ | $\geq 18$           | $\geq 55.0$               |             |          |

It has been established that after deformation, the sample manufactured by the SLM has strength properties higher than the temporary resistance and yield strength of 09CrNi2MoCu plate metal (figure 2). The data obtained do not contradict the results of mechanical tests of additive stainless steels of various grades, in which the excess strength parameters (hardness, tensile strength and yield strength) over similar indicators of monolithic samples of identical chemical composition were also repeatedly recorded [4, 5]. At the same time, according to $\sigma_y / \sigma_{UTS}$, no significant reduction in the plasticity margin was found.

Figure 2. Diagram $\sigma$ ($\varepsilon$) of the sample manufactured by the SLM.

The $\alpha \leftrightarrow \gamma$ transition temperatures were obtained for a sample manufactured by the SLM: $A_{c1} = 780$ °C, $A_{c3} = 900$ °C. The values of the critical points of the melted sample are higher than the previously obtained values for the sample of 09CrNi2MoCu plate metal, for which: $A_{c1} = 720$ °C, $A_{c3} = 840$ °C. This fact indicates that in the material after the SLM, fine-grained bainite was formed earlier, but in less time the effects of the laser beam did not have time to coarsen the grain, as confirmed by the microstructure of the samples according to EBSD analysis (figure 3).

Figure 3. The microstructure of this sample.

Studies of the microstructure of metals, performed by the methods of optical metallography (figure 4), show that the structure of plate metal specimens differs from the structure of specimens manufactured by the SLM. Plate metal 09CrNi2MoCu is characterized by a martensitic type structure with crystallites from 7 to 30 microns, and samples manufactured by the SLM are small objects of bainitic morphology, while their bainitic microstructure differs from plate metal specimens primarily by the layered structure.
associated with layer-by-layer powder melting. The structure also differs in dispersion: the sizes of bainitic crystallites near the boundaries of the melted layers are within $1 \div 5 \mu m$, and inside the layers – $13 \mu m$. The width of the structural layers reaches 200 microns. Such a small difference is very noticeable in terms of deformation.

![Figure 4](image1.png) ![Figure 4](image2.png)

**Figure 4.** The microstructure of 09CrNi2MoCu plate metal specimens (left) and specimen manufactured by the SLM (right) at comparable magnifications.

Photographs of fractures of 09CrNi2MoCu plate metal specimens and specimen manufactured by the SLM (figure 5) showed that the destruction was viscous through the body of grains with the formation of a dimple relief. In the sample manufactured by the SLM, the depth and diameter of the pits are an order of magnitude smaller than in the original. In the fracture of this specimen, wells of $\Omega 10 \mu m$ were found, the number of which is comparable with large pits on the fracture of the plate metal specimen. The remaining pits on the fracture are several times smaller. This fact does not contradict the results of mechanical tests and provides high values of metal strength.

![Figure 5](image3.png) ![Figure 5](image4.png)

**Figure 5.** Electronic photographs of fractures of the test specimens from 09CrNi2MoCu plate metal specimens (left) and specimen manufactured by the SLM (right) at different optical magnifications.

**Conclusions**

On the basis of the present studies of the structure and properties of the samples made from the original 09CrNi2MoCu steel sheet and obtained by SLM from an atomized powder material of the same grade, the following can be concluded:

When the powder particles are melt in the sample, a fine-grained bainitic structure is formed.
The temperatures of the beginning and end of the α→γ transition in the sample built by the SLM are higher than in the same steel sheet.

In comparison with martensite of 09CrNi2MoCu sheet steel, the fine-grained bainite structure of the material obtained by the SLM is characterized by higher strength, but lower ductility.

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References
[1] Shishkovsky I V 2009 Laser synthesis of functional-gradient mesostructures and bulk products (Moscow: Fizmatlit) p 424
[2] Kuznetcov P, Zhukov A, Deev A et al. 2018 Structure and Properties of the Bulk Standard Samples and Cellular Energy Absorbers (Rieka: IntechOpen) p 22
[3] Sedlaka J, Rican D, Piska M and Rozkosny L 2015 Procedia Engineering 100 1232-41
[4] Zhukov A, Barakhtin B and Kuznetsov P 2017 Physics Procedia 89 179-86
[5] Kuznetcov P A, Zisman A A, Petrov S N and Goncharov I V 2016 Deformation and Destruction of Materials 4 9-13