Investigation of the parameters of direct laser growing and subsequent processing to obtain a defect-free structure of a material made of a heat-resistant EP648 alloy

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Abstract. This paper presents the development of stable modes of additive technology of direct laser growing, using the starting material - a metal powder made of heat-resistant EP648 alloy of Russian production. The subsequent heat treatment of the manufactured samples was tested in order to avoid the formation of cracks in the structure of the material formed as a result of the presence of internal stresses after surfacing. Recommendations for further research are given.

1. Introduction

Additive technologies are steadily developing at the present time. Modern equipment removes restrictions on the geometry of production, expands the catalog of materials and allows you to set almost any characteristics of the resulting product. The introduction of printed parts into the design documentation is increasingly taking place, the certification of products and technical processes is more active [1].

The technology of direct laser growing (DLG) or direct supply of energy and material is also actively developing. In the DLG technology, the material is fed directly to the place of energy supply and the construction of a part fragment at the moment [2, 3].

The characteristic features of the technology are:

- high utilization rate of metal powder ($\approx$ 95 \%);
- the restriction on the size of the product (minimum wall thickness, overhanging structures, etc.) is determined by the possibility of focusing the laser beam;
- in the process of manufacturing the product, it is possible to change the powder material (titanium, aluminum, stainless steel, copper alloys, etc.);
- high productivity of the process;
- the dimensions of the manufactured product are limited only by the size of the working chamber of the construction and reach a diameter of up to 2-3 meters [4, 5].

The following industries can act as consumers of this technology: engine building, rocket and space, transport, ship and power engineering [6].
This paper presents studies of direct laser growing modes that are necessary for the subsequent development of the technology for manufacturing large – sized blanks using a domestic material - a metal-powder composition (MPC) made of a heat-resistant EP648 alloy.

2. Research methodology

In this work, a metal-powder composition made of a heat-resistant EP648 alloy, obtained by the technology of centrifugal spraying (PREP), was used. The manufacturer of the MPC is JSC "Composite", Korolev.

The following methods were used during the research:

- the chemical composition and shape control of the MPC particles were determined using a Tescan (Czech Republic) VEGA 3 LM electron microscope with the Oxford instruments X-Max module and using the analytical chemistry method - using an optical emission spectrometer;
- the size of the MPC particles was determined by dry sieving on a set of Retsch sieves (Germany) with cell sizes of 40 and 150 microns, with a lid and a tray on a Retsch AS200 control vibrating screen (Germany);
- the fluidity and bulk density of the MPC were determined using a calibrated funnel (Hall device) made of steel grade 12H18N10T with an output diameter of 2.5 mm. The mass of the portion (sample) for one flow test was 50±0.1 g;
- the humidity of the MPC was determined by the gravimetric method for determining water, based on drying in a furnace at a temperature of 105-110 °C of a sample of powder taken in an air-dry state to a constant mass. Drying was carried out in the Binder ED115 drying cabinet (Germany). The sample was weighed on a scale AND HR-100 AZG (Japan) with a division price of 0.0001 g;
- the absence of foreign inclusions in the studied MPC was determined visually during the sieve analysis by examining the residue on the grid;
- the DLG was carried out at the technological installation of direct laser growing produced by ILIST SPbGMTU (Russia, St. Petersburg). The selection of stable modes was carried out experimentally, by growing cylindrical blanks Ø60mm. For this purpose, a control program was written with 30 layers in one roller. When the program was started, the surfacing speed and the MPC consumption on the feeder were set randomly. After working out the program, the distance from the nozzle cutoff to the working point was measured and the speed and flow of the MPC were adjusted in order to eliminate the difference in distances before and after the program was started. When the distance difference of 0...-1 mm was reached, the mode was considered stable. When working out the DLG mode on a cylindrical billet, the MPC consumption on the feeder should not exceed 70%. This is required for the subsequent adjustment of the feed upward at the PLV of the workpiece, due to faster cooling of the grown part compared to the cylindrical workpiece;

![Figure 1. Cylindrical workpieces. a) stable mode; b) unstable mode.](image-url)
metallographic study of the grown samples and their microspectral analysis were carried out on the Tescan (Czech Republic) VEGA3 LM electron microscope with the Oxford instruments X-Max module, on the sections made along the direction of growth of the samples in three sections: at a distance of 10 mm from the edges and along the central section relative to the length of the sample (symbol x-1, x-2, x-c, where x is the sample number). Etching of the grinds was carried out in Vasiliev reagent (CuSO4-5g., H2SO4-1.4 ml., HCl-50 ml., H2O-40 ml.);

- measurement of the microhardness of the sample material was carried out on the PMT-3 device with a load of P=100g.

The selected modes are shown in table 1.

| Parameter of the laser growing mode | The value of the laser growth parameter |          |          |          |          |          |          |
|------------------------------------|--------------------------------------|----------|----------|----------|----------|----------|----------|
| Laser power P, W                   | No1 1000                             | No2 1200 | No3 1400 | No4 1600 | No5 1000 | No6 1200 | No7 1400 | No8 1600 |
| Laser spot diameter Ø, mm          | 2.7                                   | 2.7      | 2.7      | 2.7      | 2.7      | 2.7      | 2.7      | 2.7      |
| The flow rate of the transporting gas S, l/min | 10 | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| Rotation of the feeder disc D, %    | 55                                     | 55       | 50       | 50       | 65       | 60       | 55       | 50       |
| Rotation of the mixing device of the feeder S, % | 40 | 40 | 40 | 40 | 40 | 40 | 40 | 40 |
| Surfacing speed V, mm/sec           | 20                                     | 20       | 20       | 20       | 20       | 20       | 20       | 20       |
| Layer pitch h, mm                   | 0.4                                    | 0.4      | 0.4      | 0.4      | 0.4      | 0.4      | 0.4      | 0.4      |
| Distance between the centers of the rollers, mm | 1.6 | 1.6 | 1.6 | 1.6 | 1.6 | 1.6 | 1.6 | 1.6 |

3. Research results
The results of the study the shape of the MPC particles is spherical (figure 2). A homogeneous structure is observed on the surface of all particles, by which it is possible to judge with high probability the uniformity of their internal structure.

![MPC particles](image1)

**Figure 2.** The shape of MPC particles made of heat-resistant EP648 alloy.

The results of determining the particle size, fluidity, bulk density and humidity of MPC from the EP648 alloy of the main fraction of 40-150 microns are presented in table 2.
Table 2. Particle sizes, fluidity, density and humidity of MPC made of EP648 alloy.

| Granulometric composition, % | Fluidity, sec | Bulk density, g / cm³ | Humidity, % |
|-----------------------------|---------------|-----------------------|-------------|
| plus fraction (more than 150 microns) | negative fraction (less than 40 microns) | | |
| MPC EP648 | 0.5 | 3.9 | 14.5 | 4.91 | 0.04 |
| TU 136-225-2019 | no more than 5 % | no more than 10 % | ≤27.5 | 4.96±10% | ≤0.1 |

No foreign inclusions were found in the MPC.

The results of microspectral analysis of the initial MPC from the heat-resistant EP648 alloy are presented in table 3.

Table 3. Chemical composition of the initial MPC from the EP648 alloy (in mass percentages).

| Measuremen t number | Mn | Cr | Si | Ni | Fe | Al | B | Ti | Mo | Nb | Ce |
|---------------------|----|----|----|----|----|----|----|----|----|----|----|
| Average value | 0.12 | 32.99 | 0.00 | remain der | 0.32 | 1.07 | 0.00 | 0.86 | 2.79 | 0.64 | 0.02 |
| TU 136-225-2019 | ≤0.5 | 32.0-35.0 | ≤0.4 | remain der | ≤4.0 | 0.5-1.1 | ≤0.008 | 0.5-1.1 | 2.3-3.3 | 0.5-1.1 | ≤0.03 |

Figure 3 shows a map of the distribution of the main elements in the MPC particles.

Figure 3. Map of the distribution of the main elements in the MPC particles.

The remaining chemical elements of the heat-resistant alloy powder were determined according to the methods of analytical chemistry. The results are presented in table 4.
Table 4. Chemical composition of residual elements of the initial MPC from the EP648 alloy (methods of analytical chemistry).

|          | C     | S     | P     | W     |
|----------|-------|-------|-------|-------|
| MPC EP648| 0.0482| 0.0047| 0.012 | 4.60  |
| TU 136-225-2019 | ≤0.1 | ≤0.01 | ≤0.015| 4.3–5.3|

When working out the DLG modes, in comparison with previous studies, the surfacing speed was reduced from 45 mm/s to 20 mm/s.

The decrease in the surfacing speed was due to the fact that at high speed the contour of the curved surface did not have time to form properly, and as a result, at the corners, when the direction of movement changed, it was disrupted and it was impossible to perform subsequent surfacing.

Reducing the layer step allowed us to obtain a more “smooth” surface of the grown sample, which has less roughness.

The appearance of the samples is shown in figure 4:

![Figure 4. The appearance of the samples (top view), x0.5.](image)

The upper surface of the samples differs in color: on samples No. 1 and No. 5, light golden color; on samples No. 2 and No. 6, golden color with blue-purple edging on the edges of the samples; on samples No. 3 and No. 7, yellow-brown color with blue-purple edging on the edges of the samples; on samples No. 4 and No. 8, blue-purple color. The fused rollers are clearly visible.

When macro analysis of polished sections without etching in sections 1-1 and 1-c of sample No. 1, a fine porosity is observed, lined up in lines (figure 5 (a)), no defects were found on the remaining images.

![Figure 5. Appearance of the polished surface of the samples before etching: a) sample No. 1; b) sample No. 3, x1.](image)

The microstructure of the samples is shown in figure 6.
Figure 6. Microstructure of samples obtained by the DLG method: a) sample No. 1; b) sample No. 2; c) sample No. 3; d) sample No. 4; e) sample No. 5; f) sample No. 6; g) sample No. 7; h) sample No. 8, x2.

During microanalysis of the grinds without etching, it was found that there are small discontinuities in the material of all samples in the form of looseness, pores and non-melting (figure 7).
In sample No. 1, a fine porosity is observed in the form of lines up to 0.2 mm long, located along the width of the sample (figure 8).

The maximum size of single defects detected in the sample material is shown in table 5.
Table 5. Dimensions of defects detected in samples.

| Sample no. | Cross section | Maximum defect size, mm | Microhardness, HV0.15 |
|------------|---------------|-------------------------|-----------------------|
|            |               | Looseness, Porosity, Ø  | Non-melting (length)  |
| 1          | 1-1           | -                       | 0.04                  | 301…304               |
|            | 1-c           | -                       | 0.05                  | 0.04; 0.13            |
|            | 1-2           | 0.06x0.07               | 0.03; 0.045           | -                     |
| 2          | 2-1           | -                       | 0.028; 0.05           | 0.1                   |
|            | 2-c           | -                       | 0.023                 | 0.09                  |
|            | 2-2           | -                       | 0.028                 | 0.4                   |
| 3          | 3-1           | -                       | 0.045                 | -                     |
|            | 3-c           | -                       | 0.015                 | -                     |
|            | 3-2           | 0.006x0.029             | 0.015                 | -                     |
| 4          | 4-1           | -                       | 0.015                 | 0.03…0.08            |
|            | 4-c           | 0.04x0.016              | 0.015                 | -                     |
| 5          | 5-1           | -                       | 0.025                 | 0.02                  |
|            | 5-c           | -                       | 0.03…0.04            | 0.03                  |
|            | 5-2           | -                       | 0.02…0.05            | 0.02…0.08            |
| 6          | 6-1           | -                       | 0.019                 | 0.045                 |
|            | 6-c           | -                       | 0.018                 | 0.053                 |
|            | 6-2           | 0.046…0.051             | 0.04                  | 0.085                 | 286…325               |
| 7          | 7-1           | -                       | 0.012                 | 0.04                  |
|            | 7-c           | -                       | 0.03                  | 0.02…0.17            |
|            | 7-2           | -                       | 0.014                 | 0.04                  |
| 8a         |               | -                       | 0.03                  | -                     |

*a only single pores were found in all the studied sections of sample No. 8.

The total porosity of the sample material is: in samples No. 1 and 2 up to 0.02 mm, in samples No. 3,4,5 up to 0.015 mm, in samples No. 6,7,8 up to 0.012 mm.

After etching, cracks with a length of 0.05…1.1 m were found in all samples, located along the boundaries of individual grains at a distance of 0.6…2.0 mm from the side surfaces. In the samples 2,4,5,7,8 there are single cracks in the central part (figure 9-13).

Figure 9. Cracks in sample No. 1.
Figure 10. Cracks in sample No. 2.

Figure 11. Cracks in sample No. 4 (cross-section).

Figure 12. Cracks in sample No. 6. a) the central zone; b) the edge zone.

Figure 13. Cracks in sample No. 8. a) without etching; b) after etching.

Before etching, only one crack was detected in sample No. 8 (figure 13).

The micro hardness of the samples is almost at the same level. There is a slight decrease in hardness on the samples obtained by DLG with a power of 1400 W and 1600 W.
Figure 14. The structure of the material according to the width of the samples: a) sample No. 1; b) sample No. 2; c) sample No. 3; d) sample No. 4; e) sample No. 5; f) sample No. 6; g) sample No. 7; h) sample No. 8.
The obtained samples were examined without performing heat treatment after the DLG, when they were in a stressed state. As a result of the impact of the cutting tool, when preparing the grinds for performing metallographic research, their cracking occurred.

To verify this theory, it was decided to conduct heat treatment of samples with the least number of defects, such as pores and friability, and re-examine them.

For the subsequent development of heat treatment, the modes used for samples No. 5, 6, 7, 8 were selected.

The heat treatment of the samples was carried out according to the regime recommended for the VH4L alloy: quenching (1180±10) °C, exposure time of 4 hours, air cooling.

No defects were detected during the macro analysis of the polished sections.

During microanalysis of the grinds without etching, it was found that there are small discontinuities in the material of all samples in the form of pores and non-melts. The pore diameter in samples No. 5 and 6 is up to 0.015 mm, in samples No. 7 and 8 up to 0.012 mm. The maximum size of single defects detected in the sample material is shown in table 6.

| Sample no. | Maximum defect size, mm | Microhardness, HV<sub>0.15</sub> | Average value |
|------------|-------------------------|----------------------------------|---------------|
|            | Looseness                | Porosity, Ø                      |               |
| 5          | 0.024x0.06               | 0.040                            | 334…373       |

Table 6. Maximum sizes of single defects.
After etching in one section, thin cracks with a length of 0.39 mm, 0.4 mm and 0.98 mm were found in the central zone of sample No. 8, located along the grain boundaries along the direction of sample growth (figure 17; 18 d).

| Sample | Length (mm) | Width (mm) | Position |
|--------|-------------|------------|----------|
| 6      | 0.070       | 0.040      | 343…363  |
| 7      | 0.013       | 0.060      | 353…363  |
| 8      | 0.070       | 0.025      | 353…373  |

Figure 17. Crack in sample No. 4.

Figure 18. Microstructure of the material along the width of the samples: a) sample No. 5; b) sample No. 6; c) sample No. 7; d) sample No. 8.
The track boundaries in the microstructure of the sample material are not detected after heat treatment, grain boundaries are formed in the microstructure with the preservation of the dendritic-columnar structure of the fractional orientation (figure 18).

On the surface of the samples after heat treatment, there is oxidation up to 0.13 mm deep (figure 18).

The microhardness of all samples is almost at the same level. There is a tendency to increase the microhardness with an increase in the laser power at DLG. The reverse trend was observed on the samples before heat treatment.

The increase in the hardness of the samples may be associated with the formation of a strengthening $\gamma'$ phase formed after heat treatment.

The performed heat treatment confirmed the version of the formation of cracks in the samples due to internal stresses.

4. Conclusions
According to the results of testing the stable modes of DLG, 8 stable modes were obtained. After etching, cracks with a length of 0.05...1.1 mm were found in all samples. It is possible that the formation of cracks was due to the fact that the obtained samples were examined without performing heat treatment after DLG, when they were in a stressed state. As a result of the impact of the cutting tool during the preparation of the grinds for performing a metallographic study, their cracking occurred.

After performing the heat treatment and re-examination, there were no cracks on all the selected samples, except for one that was made at the maximum surfacing power.

To obtain the most complete picture, it is necessary to conduct further research of the mechanical properties of the samples, both short-term and long-term.

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