Additive technology for manufacturing structurally-graded materials from the Inconel 625 nickel-based superalloy

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Abstract. This paper describes the results of direct gas-powder laser deposition. Modes of obtaining samples of the Inconel 625 nickel-based superalloy were practically determined. The study displayed the possibility of creating structurally graded materials. Microhardness of areas with different structures was studied. The obtained results allowed to determine the influence of heat on mechanical properties.

1. Introduction

The technologies of additive manufacturing (AM) and layered synthesis are currently among the most dynamically developing trends in the world [1]. They offer engineers broader approaches to designing and further manufacturing of parts as compared with the traditional methods of casting and machining [1].

Recently, there have been numerous studies of the attempts to determine the dependence of the features of structure from the parameters of the additive manufacturing processes [4]. Thus, for example, for the Inconel 718 alloy reducing the grain size facilitates the increase in short-term and fatigue properties while increasing grain size facilitates the increase in long-term creep rupture strength [5].

Conditionally, metallic material AM can be divided into two basic categories: Powder Bed Fusion (processes where products are manufactured within the powder mass), and Directed Energy Deposition (DED) (supposing the feed of the construction material (powder, wire) immediately into the melting pool). The DED method (Fig. 1) is more suitable to create a structural gradient due to more flexibility in the ability to change the melt micro-pool and, consequently, the rate of crystallization.
Figure 1. General layout of the DED method (of direct gas-powder laser deposition) [6].

2. Materials and methods
The first stage of the research was concerned with designing a facility for direct gas-powder laser deposition (Fig. 2).

![Facility for direct gas-powder laser deposition](image)

The main components were:
- a FANUC industrial articulated robot;
- an LS-3 solid-state fiber laser by IRE-Polyus (Fryazino, Moscow Oblast), a subsidiary of IPG Photonics, with the maximum power of 3 kW 1070 nm longwave;
- a cladding head with triple-nozzle powder feed to the melt pool by Fraunhofer;
- two-hopper powder feeder by Plakart.

It should be noted that the main end effector that ensures the process of direct gas-powder laser deposition is the cladding head. It includes a manually controlled collimator for dynamical adjustment of laser beam divergence during operation. Changing the beam divergence will change the focal position of the objective lens (Fig. 3).
This way, there appears the possibility to measure the diameter of the laser spot and, consequently, of the local melting and the volume of the micro-pool.

The second stage consisted of the practical development of the modes of direct gas-powder laser deposition with different diameters of the laser spot. The Inconel 625 nickel-based superalloy was used as the initial powder material. Its chemical composition as declared by the manufacturer and the grain size composition identified by laser diffraction using the Analysette 22 particle size measuring unit are presented in Table 1 and Table 2 respectively.

Table 1. Chemical composition of the Inconel 625 nickel-based superalloy, wt. %.

| Element | Ni  | Cr  | Mo  | Nb  | Mn  | Co  | Si  | Fe  | Al  | Ti  |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Rest    | 20-23 | 8-10 | 3-4 | <0,50 | <1,0 | <0,5 | <5  | <0,4 | <0,4 |

Table 2. Grain-size composition of the Inconel 625 nickel-based superalloy.

| Size   | Measurement | Average |
|--------|-------------|---------|
|       | 1           | 2       | 3       |          |
| $d_{10}$ μm | 47.8       | 47.5    | 47.5    | 47.5     |
| $d_{50}$ μm | 85.4       | 85.1    | 85.2    | 85.2     |
| $d_{90}$ μm | 141.1      | 140.6   | 140.8   | 140.8    |

The study of hardness was carried out using the Zwick Roell ZHU universal hardness testing machine.

3. Results of the research and their discussion.

Within the course of the practical development of the modes of direct gas-powder laser deposition, the following parameters turned out to be variable: laser power, robot manipulator movement speed, amount of the powder fed. The distance from the base to the cladding head was 12-13 mm (the area of the material's "overstretching"). In the course of processing, the two maximum and minimum diameters possible for the given cladding head were used: 0.8 and 3 mm respectively.

Table 3 below shows the modes that ensure the stable process of cladding the Inconel 625 nickel-based superalloy on a single-pass wall.
Table 3. Mode of melting Inconel 625 ensuring the stability of the process.

| Laser spot diameter, mm | Laser power, W | Movement velocity, mm/s | Powder volume, g/min | Elevation, mm |
|-------------------------|----------------|-------------------------|----------------------|---------------|
| 0.8                     | 300            | 10                      | 7                    | 0.2           |
| 3                       | 1200           | 12                      | 18.3                 | 0.6           |

Figure 4 shows the samples obtained with different laser spot diameters. In the case of a large spot, the process is stable in all layers.

![Figure 4](image1)

**Figure 4.** Single-pass walls where a is the large laser spot diameter and b is the small laser spot diameter.

In the case of small diameter, the process used to be stable for the first 30 layers and then started to display longitudinal waviness. Obviously, this is linked to the broad grain-size composition that occasionally does not fall within the focus of the laser spot, thus disturbing the stability of the process.

The structural gradient was created in the following way: first, the base layer was deposited with the larger laser spot (around 6 mm wide and 6 mm high). Further, the grown sample was deposited with the small laser spot (around 5 mm wide and 8 mm high). One half of the remaining sample was sawed off and separated from the platform: this makes the sample No.1. After that, the remaining sample was once more subjected to cladding with a large laser spot (around 5 mm wide and 8 mm high): this makes the sample No.2. Figure 5 shows the process of obtaining the gradient structure.

![Figure 5](image2)

**Figure 5.** The process of obtaining gradient structure from the Inconel 625 nickel-based superalloy in the course of separating the sample No.1.

Then, thin sections were prepared out of the samples. Structural study was carried out by means of etching in the solution of aqua regia with the addition of hydrofluoric acid. Figure 6 shows the panoramic view of the sample No.2.
direction of deposition

**Figure 6.** Panoramic view of the sample No.2 after etching of the Inconel 625 nickel-based superalloy.

The panoramic view shows the micro-pools of the melt, whose size corresponds to the cross-section of the cladding track. A separate shot increased 50 times (Figure 7, a) shows the patterns of the grains consisting of dendritic cells. It has been established that the grain size when using the smaller laser point is two to five times less than when using the larger laser spot. The larger spot is characterized by protruded grains matching the direction of crystallization (along with the sample's height), while at the smaller spot the grains are conditionally equiaxed. Larger magnifications (Figure 7, b) make it clear that the larger laser spot mostly identifies the dendrite branches of the 3rd order, while the smaller spot mostly show the 1st and the 2nd ones.

**Figure 7.** The microstructure of the transition layer of the sample with different laser spot sizes at different approximations where a is the 50 times increase and b is 2000 times increase.

The microhardness test was carried out at the load of 10 kg/s during 10s using a diamond pyramid (according to the Vickers hardness test). Table 4 shows the results of the calculations.

**Table 4.** Microhardness of the samples.

| Measurement | Sample No.1 | Sample No.2 |
|-------------|-------------|-------------|
|             | Large laser spot |             |             |
| 1           | 293.1       | 282.4       |
| 2           | 287.2       | 280.1       |
| 3           | 284.9       | 290.3       |
| Average     | 291.1       | 285.2       |
|             | Small laser spot |             |             |
| 1           | 345.9       | 324.4       |
| 2           | 344.2       | 316.6       |
| 3           | 347.5       | 317.9       |
| Average     | 345.9       | 319.6       |
|             | Large laser spot |             |             |
|   |   |   |
|---|---|---|
| 1 | - | 283.3 |
| 2 | - | 293.8 |
| 3 | - | 280.7 |
| Average | - | 285.9 |

As can be seen in the results produced, the microhardness of the metal obtained using the larger laser spot is approximately repeated regardless of the position within the sample. The microhardness in the smaller spot is, as expected, higher than in the larger one, but this is substantially different in the samples. It is known that in the case of direct gas-powder laser deposition, as well as in all additive technologies, there are high hardening stresses in the synthesized products related to the quick cooling of particular layers (tracks). Thus, cladding of a large spot upon the smaller one launched the process of relaxing the hardening stresses, which, in the end, influenced the mechanical properties of the particular areas. This way, when producing a structurally-graded product, it is necessary to factor in the thermal influence of the neighboring layers (zones) to obtain the desired characteristics.

4. Conclusion
This research has led to designing a facility for direct gas-powder laser deposition. The technological modes of obtaining samples of the Inconel 625 nickel-based superalloy at various diameters of the laser spot have been developed. The following structural features of using different laser spot sizes have been identified: the larger spot results in larger prolonged grains with dendrites of the third order, while the smaller spot results in grains that are 2-5 times smaller and have a more equiaxed structure. The microhardness of the materials obtained using the smaller laser spot significantly surpasses that obtained at the larger spot but is different in samples due to the influence of temperature that causes the relaxation of hardening stresses.

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