The structure and mechanical properties of parts elaborated by direct laser deposition 316L stainless steel powder obtained in various ways

I S Loginova1,*, A N Solonin1, A S Prosviryakov1, S B Adisa1, A M Khalil1, D P Bykovskiy2, V N Petrovskiy2
1 National University of Science and Technology “MISiS”, Moscow, Russia
2 National Research Nuclear University MEPhI (Moscow Engineering Physics Institute), Kashirskoe shosse 31, Moscow 115409, Russia

E-mail: *i-popkova@list.ru

Abstract. In this work the morphology, the size and the chemical composition of the powders of steel 316L received by the two methods was studied: fusion dispersion by a gas stream and reduction of metal chlorides with the subsequent plasma atomization of the received powder particles. The powder particles received by the first method have a spherical shape (aspect ratio 1.0–1.2) with an average size of 77 μm and are characterized by the absence of internal porosity. Particles of the powder received by the second method also have a spherical shape and faultless structure, however, their chemical composition may vary in different particles. The average size of particles is 32 μm. Though the obtained powders had different properties, the experimental samples received by DLD technology demonstrated by equally high durability (Ultimate strength is 623±5 and of 623±18 MPa respectively) and plasticity (38 and 41% respectively). It is established that mechanical properties of DLD samples increase for 7-10% after treatment of the surface.

1. Introduction
Additive technologies are a new type of production of the prototypes and the functional parts of complex shape from powders of aluminum, nickel, titanium, cobalt alloys and alloyed steels [1–10]. The products received by additive technologies can be characterized with high dispersion of structure and enhanced mechanical properties (in comparison with the standard products received by casting, rolling, forging and stamping) [2,5]. The DLD technology is one of the types of additive production, it implies direct supply of powder material by means of a nozzle into the zone of influence of a continuous wave laser beam. By means of DLD technologies, it is possible to produce prototypes and finished products of any shape for arms, medical, aviation and other types of industry.

The initial material in additive manufacturing is a metal powder with special properties: a spherical shape of particles with a narrow particle size distribution ranging from 30 to 70 μm, a homogeneous chemical composition without internal defects (pores). Such powders have better flow rate and compaction than powders with a dendritic particle shape, which ensures continuous supply to the treatment zone through a narrow coaxial nozzle [9]. Formation of a product with a high density and chemical uniformity is possible due to the homogeneous chemical composition of powders and the absence of internal defects. Therefore, development of the fundamentals of technology for the production of powders, which would fully satisfy all the requirements of DLD-process, is a promising
research trend. The most common methods of producing powders for DLD technologies are pulveri of a liquid alloy with a gas jet and plasma atomization of powder particles with an irregular (angular) shape. In the present work, a comparative analysis of the morphology, size, and composition of 316L grade austenitic steel powders, obtained by two methods, was made. The first powder (P1) was obtained by spraying with a gas melt jet, the second (P2) – a reduction of the metal chloride and the subsequent plasma atomization of the resulting solid powder particles. A comparative analysis of the structure and properties of the DLD-manufactured samples, obtained using these powders, was carried out.

2. The objects of research and the methodology of experiments

P1 and P2 powders were used for deposition of the experimental samples. Powder P1 has a spherical particle shape (aspect ratio of 1.0–1.2) with an average size of 77±13.5 μm and can be characterized by dense structure (figure 1a) and a uniform chemical composition (table 1). Powder particles P2 also have a spherical particle shape (aspect ratio of 1.0-1.2) and a defect-free structure (figure 1b). The particle size of the powder P2 is finer than P1 – 32±8.5 μm. Separate powder particles have a non-uniform chemical composition (table 2). However, the average composition of all powder particles P2 corresponds to the 316L steel grade.

![Figure 1. Powder particle structure P1 (a) and P2 (b).](image)

The aspect ratio of powders was determined using SEM images of particles. Image magnification was chosen so as to observe no more than 150 particles. The aspect ratio represents the relation of the maximum linear size of a projection of a particle lmax to its minimum lmin size (lmax/lmin). The form of particles is defined as follows:

- lmax/lmin = 1.0–1.2, the form is defined as spherical;
- lmax/lmin = 1.2–2.0 – rounded shape;
- lmax/lmin = 2.0–5.0 – the form is angular;
- lmax/lmin = 2.0–25.0 – the form is rod;
- lmax/lmin = 25 – needle-shaped.

| Spectrum | Cr   | Ni   | Mo   | Mn   | Si   | C    | Fe   |
|----------|------|------|------|------|------|------|------|
| 1        | 17.5 | 11.8 | 2.4  | 1.8  | 0.7  | <0.03| bal. |
| 2        | 17.9 | 11.1 | 2.7  | 1.5  | 0.8  | <0.03| bal. |
| 3        | 16.8 | 11.5 | 2.6  | 1.7  | 0.85 | <0.03| bal. |
| Average composition | 17.3±1.0 | 11.7±0.6 | 2.5±0.3 | 1.7±0.2 | 0.9±0.25 | <0.03 | bal. |
Table 2. Chemical composition of powder P2.

| Spectrum | Cr   | Ni   | Mo  | Mn  | Si   | C    | Fe    |
|----------|------|------|-----|-----|------|------|-------|
| 1        | 17.2 | 12.5 | 2.8 | 1.5 | 0.6  | <0.03| bal.  |
| 2        | 17.9 | 12.6 | -   | 1.7 | 0.9  |      |       |
| 3        | 17.1 | 12.3 | 2.1 | 1.5 | 0.7  |      |       |
| 4        | 19.0 | 8.6  | -   | -   | -    |      | <0.03 |
| 5        | 24.4 | 1.2  | -   | -   | -    |      |       |
| 6        | 13.1 | 11.5 | -   | -   | 0.3  |      |       |
| 7        | 21.4 | 2.6  | -   | -   | 0.2  |      |       |
| 8        | 19.4 | 8.6  | -   | -   | 0.1  |      |       |
| Average  | 18.6±2.7 | 8.7±3.7 | 2.4±0.4 | 1.6±0.2 | 0.5±0.3 | <0.03 | bal.  |

Multilayer three-dimensional objects were fabricated on a substrate of A284 Steel.

DLD process was carried out on a HUFFMAN HC-205 industrial plant equipped with a fiber laser LS-3.5 with a maximum power of 3.5 kW with a radiation wavelength \( \lambda = 1069 \) nm. The powder mixture was transported by an argon stream and focused on the treatment zone with a coaxial nozzle of diameter 1.9 mm.

In the design of the coaxial head, an axisymmetric beam was formed. It made possible to obtain a thin gas-powder flow resulting in track deposition with a width more than 200 \( \mu \)m and a thickness of 200 \( \mu \)m. In operation, the width of the track was 500 \( \mu \)m. The overlap of the adjacent tracks of one layer was 10–15\%.

Laser surfacing of samples in the form of a brick (width – 12 mm, length – 70 mm, height – 10 mm. figure 2a) and dumb-bells (width of the working part – 4 mm, length – 67 mm, height – 10 mm. figure 2b) using a crosswise strategy of the scanning (figure 3). The laser power was 250 W, the powder flow rate was 0.013 g/s; scanning speed 16 mm/s.

In order to study the structure, the surface of the samples was polished on a Struers Labopol-5. The structure of the alloys was studied using electron scanning microscope TESCAN VEGA LMH with a cathode LaB6 and an X-ray energy dispersive microanalysis system of Oxford Instruments Advanced AZTec Energy X-max 50. The backscattered electron mode (BSE) was used.

![Figure 2](image1.jpg)  
![Figure 2](image2.jpg)  

Figure 2. Form of samples: dumb-bell (a) and brick (b).
Figure 3. Scheme of scanning strategy.

The mechanical properties of the samples were evaluated by the yield strength (YS), and the ultimate strength (UTS) values, which were determined by the uniaxial tensile test on a Zwick Z250 with a test speed of 4 mm/min.

3. Results and discussion
The structure of samples made from P1 and P2 powders was the same. It is shown in figure 4 at different magnifications. On the general image of the structure, tracks distinguish in parallel layers. It was found that crystals of elongated shape were located in these layers facing away from the arc-shaped interface.

The reason for this can be a direct heat removing during crystallization of the melt. The track has the shape of a semicircle. During cooling, the heat is removed in all directions from its surface, so that the crystals grow inward parallel to this direction. (figure 4a). Samples were characterized by high continuity, absence of gas pores and cracks (figure 4a). At high magnifications, it was found that an ultrafine austenite structure with an average crystallite size of 2.5 μm was formed. This crystallite size corresponds to high cooling rates of 10⁵-10⁶ K/s [8].

There are two types of crystals: fine columnar and fine equiaxed grains (figure 4b). It was established by means of EDX that samples made of powder P2 (which have low chemical homogeneity of the particles) do not have a segregation of the chemical composition at their cross section. Apparently powder particles of different composition were melted and form a drop of melt with an average chemical composition during the DLD process. Thus, the chemical composition was leveled after the solidification of layers.

To assess the mechanical properties of the experimental samples, uniaxial tension tests were carried out. The results are represented in table 3. In both samples made from powders P1 and P2, the strength and ductility were high. It should be noted that the strength of the samples was higher than 30–40 MPa in comparison with samples of the same steel obtained by other researchers [8]. Surface treatment of the test samples (EDM of cutting edges) leads to an increase of mechanical properties by 7–10% (table 3).
Figure 4. Microstructure of DLD-samples.

Table 3. Average values of properties.

| Conditions/powder                  | YS. MPa | UTS. MPa | Elongation. % |
|-----------------------------------|---------|----------|---------------|
| DLD/P1                            | 489±20  | 624±4    | 38±14         |
| DLD/P1 (surface treatment)        | 495±7   | 685±21   | 31±2          |
| DLD/P2                            | 400±25  | 632±18   | 41±14         |
| DLD/P2 (surface treatment)        | 447±3   | 675±10   | 46±10         |
| Wrought (cold finished) [8]       | 255–310 | 525–623  | 30            |
| DLD/other studies [8]             | 330–395 | 540–625  | 35–85         |

The fractures were mainly ductile, as evidenced by the presence of pits at the surface of the fractures (figure 5a. b).

Figure 5. Typical fracture image of DLD-samples in the different zones.

4. Conclusions
The morphology, size, and chemical composition of 316L steel powders obtained by fusion dispersion by a gas stream and reduction of metal chlorides with the subsequent plasma atomization were studied. Particles of powder P1 have a spherical shape (aspect ratio of 1.0–1.2) with an average size of 77 μm.
and have a dense structure. Powder particles P2 also have a spherical shape and a defect-free structure but the chemical composition of the individual particles may vary. The average particle size is 32 μm.

It was shown that the experimental samples P1 and P2 obtained by DLD process demonstrated by equally high strength (ultimate strength is 623±5 and 623±18 MPa. respectively) and plasticity (38 and 41%. respectively). despite the different properties of 316L steel powders.

5. Acknowledgments
This work was supported by the Ministry of Education and Science of the Russian Federation in the framework of Increase Competitiveness Program of NUST"MISiS"(No. K1-2014-026) and by National Research Nuclear University MEPhI in the framework of the Russian Academic Excellence Project (contract No. 02.a03.21.0005,27.08.2013).

References
[1] Zaitsev A A. Sentyurina Zh A. Levashov E A. Pogozhev Yu S. Sanin V N. Loginov P A and Petrzhi M I 2017 Structure and properties of NiAl-Cr(Co.Hf) alloys prepared by centrifugal SHS casting. Part 1 – Room temperature investigations Materials Science and Engineering: A 690 (6) 463
[2] Shamsaei N. Yadollahi A. Bian L and Thompson S M 2015 An overview of Direct Laser Deposition for additive manufacturing Part II: Mechanical behavior. process parameter optimization and control Additive Manufacturing 8 12
[3] Zhang B. Dembinski L and Coddet C 2013 The study of the laser parameters and environment variables effect on mechanical properties of high compact parts elaborated by selective laser melting316L powder Materials Science&Engineering A 584 21
[4] Liu Y. Li A. Cheng X. Zhang S Q and Wang H M 2016 Effects of heat treatment on microstructure and tensile properties of laser melting deposited AISI 431 martensitic stainless steel Materials Science&Engineering A 666 27
[5] Yadollahi A. Shamsaei N. Thompson S M and Seely D W 2015 Effects of process time interval and heat treatment on the mechanical and microstructural properties of direct laser deposited 316L stainless steel Materials Science&Engineering A 644 171
[6] Chlebus E. Kuźnicka B. Kurzynowski T and Dybała B 2011 Microstructure and mechanical behaviour of Ti—6Al—7Nb alloy produced by selective laser melting Materials Characterization 62 488
[7] Louvis E. Fox P and Sutcliffe Christopher J 2011 Selective laser melting of aluminium components Journal of Materials Processing Technology 211 275
[8] Ma M. Wang Z and Zeng X 2017 A comparison on metallurgical behaviors of 316L stainless steel by selective laser melting and laser cladding deposition Materials Science&Engineering A 685 265
[9] Cheikh H E. Courant B. Branchu S. Huang X. Hasco J Y and Guillen G 2012 Direct Laser Fabrication process with coaxial powder projection of 316L steel. Geometrical characteristics and microstructure characterization of wall structures. Optics and Lasers in Engineering 50 1779