Effect of particle size distribution on the structure and mechanical properties in the process of laser powder bed fusion

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Abstract. The paper studies effects of particle size distribution on the structure and mechanical properties of monolithic samples obtained by L-PBF. A powder of 321 austenitic stainless steel of one batch was divided into three fractions 0–20, 20–40, and 0–40 μm. It was established that for narrow fractional powder composition, hardness anisotropy is observed that depends on the building direction, whereas for wide fractional powder composition, hardness anisotropy is practically absent. It was found that the particle size composition of AISI 321 steel powder does not fundamentally affect the morphology of the grain structure. Despite the general preferred orientation of the {101} planes, a weak effect of the powder composition on the crystallites orientation is observed.

1. Introduction

Laser powder bed fusion (L-PBF) is a process of consolidating powder materials for the manufacture of complex objects of arbitrary shape directly from models obtained by computer modeling (CAD) without expensive tools and additional processing.

The powder used in L-PBF technology meets certain requirements for particle size distribution and particle shape. It is believed that the most suitable range for most installations is 20–40 or 20–80 μm in particle size distribution, and the particle shape should be close to spherical. These requirements are considered to be the most basic and depend on the thickness of the powder layer to be fused. However, the powder materials are produced by gas melt spraying and the resulting powder contains particles with dimensions of less than 20 microns and substantially more than 40 or 80 microns, up to 200 microns. Moreover, the shape of the particles is far from perfectly spherical, and the content of the fraction of 20–40 or 20–80 microns in the resulting powder is less than 50%, which significantly affects its cost. To reduce the cost of the powder, research is being conducted on the spheroidization of such powders. When fusing powders of various particle size distribution, it may also be necessary to select fusion modes that is discussed in a number of papers [1–8]. For example, [9] and [10] analyzed the effect of particle size on the absorption of laser radiation by the surface of a powder layer: relationship between the absorption, distribution of absorbed irradiance within the powder layers, and surface morphology and geometric characteristics (e.g., contact angle, width and height of tracks, and remelted depth) of the laser scanning tracks. The simulation estimation shows that the absorption capacity of the powder layers significantly exceeds the value of single powder particles or the value of a dense solid material. With increasing particle size, the powder layer absorbs less laser energy [9]. Therefore, it is of interest to
experimentally study the effect particle size distribution of powder material on the quality of the L-PBF samples.

A number of authors [11] suppose that mixing particles of different sizes and shapes can be used to significantly increase the packing density, but can also lead to separation or segregation of the powder layer.

Thus, the influence of the particle size distribution of the powder on the structure and properties of monolithic L-PBF samples is not fully disclosed.

The aim of this work is to study the effect of the particle size density distribution of the powder material on the structure and properties of L-PBF samples made of AISI 321 steel.

2. Materials and methods

The raw powder material of AISI 321 austenitic steel was manufactured at Polema JSC by gas atomisation and sieved through a sieve with 40 μm mesh. Then the raw powder was sieved through a sieve system into two fractions with a particle composition: 0–20 μm and 20–40 μm, which were used for L-PBF. For further investigation, powders of 0–20 μm, 0–40 μm and 20–40 μm were used.

Cylindrical samples with a diameter of 6 mm and a height of 10 mm were made by the L-PBF method at the EOSint M270 installation. The laser melting parameters were as follows: laser power 190 W, laser spot diameter d(1/e²) = 70 μm, track spacing 100 μm, scanning speed 800 mm/s and layer thickness 40 μm. Protective gas of the building chamber was nitrogen.

To study the structure, sections were made whose plane was oriented in two directions: horizontal — perpendicular to the building direction and vertical — located in the building direction. To reveal structure in AISI 321 steel, electrochemical etching was used at room temperature in a 10% aqueous solution of oxalic acid. Metallographic analysis was performed using a Leica DM3 light microscope. Structural studies were carried out using a scanning electron microscope (SEM) VEGA 3 TESCAN. EBSD cards were obtained on a Tescan LYRA 3 microscope with a step of 3 μm (a grid of 400×300 pixels). Hardness measurements were performed on a Zwick / Roell ZHU 750 top with a load of 10N. For each sample, 10 measurements were performed.

3. Results and discussions

3.1. Microstructure of the L-PBF samples

The microstructure of the L-PBF samples before and after etching in two planes relative to the building direction, the horizontal plane and the vertical plane, for samples obtained from AISI 321 powders with three different particle size distributions (0–20 μm, 0–40 μm, and 20–40 μm), are represented in Figure 1. The microstructure of all three L-PBF samples after etching consists of a dendritic and cellular structure with subgrains smaller than 0.5 μm, which is typical for austenitic steels produced by the L-PBF method [12, 13].

The melt pools revealed by etching look almost the same. Their width and depth is about 100 μm. This suggests that the time of exposure of laser radiation was sufficient to melt both the powder layer and the previous already fused layer.

It should be noted that in the sample of 20–40 μm, defects are present to a greater degree than in the sample of 0–40 μm to an average degree, and the smallest volume of defects is in the sample of 0–20 μm (see Table 1). That is, the minimal porosity was achieved using a relatively fine powder.

| Type of sample | Integrated porosity, % |
|---------------|------------------------|
| 0-20 μm       | 0.23                   |
| 0-40 μm       | 0.57                   |
| 20-40 μm      | 1.09                   |
Such a difference in porosity as function of the particle size, apparently, can be associated on the one hand with different absorbing ability of laser radiation by powders [9, 10]. With a decrease in particle size, the surface area interacting with laser radiation increases, which should lead to a more rapid melting of the powder. On the other hand, with a decrease in particle size, powder bulk density should increase because of the presence of particle size distribution and, consequently, larger mass of metal powder melts during laser irradiation. It turns out that we have two competing processes and it is most optimal, from the porosity point of view, to use a multidisperse powder, in which both small and large particles are present.

Figure 1. Microstructure of the L-PBF samples before and after etching: (a), (d) 0–20 µm; (b), (e) 0–40 µm; (c), (f) 20–40 µm; (a) – (c) horizontal plane; (d) – (f) vertical plane.
The EBSD maps of crystallite orientations relative to the normal to the plane of the sample are represented in Figure 2. It should be noted that the layer thickness by building is 40 μm, and the grain length in the building direction in some cases exceeds 500 μm (Figure 2 b, d, f). Thus, with the addition of metal on each layer, grains grow through several layers of metal, which corresponds to the generally accepted position on the epitaxial growth of crystallites.

In order to quantitatively analyze the EBSD structure, pole figures of orientational densities (Figure 3) and inverse pole figures (Figure 4) of crystallographic planes were constructed.

Figure 3 shows the pole figures of the orientational densities of the crystallographic planes \{100\}, \{110\} and \{111\} for the horizontal plane of the sample (Figure 2 a, c, e). This type of pole figures was noted for all three L-PBF samples and, accordingly, does not practically depend on the powder composition.
Figure 3. Typical pole figures of orientational densities of crystallographic planes.

It can be seen from the figure that in the L-PBF process a directional grain structure is created. The crystal lattice of individual grains is characterized by a preferred orientation. Since the pole figures show the set of orientational densities of all crystallographic planes in the studied area, the observed circular shape of these area is associated with a set of crystallites rotated at different angles around the <110> axis, which coincides with the building direction. Thus, the structure in the XY plane is isotropic.

The highest densities roughly correspond to the preferred position of the planes at an angle from the normal to the sample plane: \{100\} – 75° and 30°, \{110\} – 0° and 60°, \{111\} – 35°. Such angles of arrangement of crystallographic planes (according to Figure 3) correspond to the angles between the \{100\}, \{110\} and \{111\} planes in the cubic lattice. Based on this, we can say that the preferred orientation of the fcc lattice in the building direction is as follows (see Figure 3 for an additional image on the left). We can say that for all three powder compositions of the powders, crystallites are oriented mainly in the direction <110>, which coincides with the building direction. In this case, a precession of crystallographic planes relative to the building direction is observed.

The inverse pole figures of the orientational densities in the Z direction relative to the studied plane of the thin section — the horizontal plane perpendicular to the building direction of the sample are presented in Figure 4.

Figure 4. Inverse pole figures of orientations of crystallographic planes along the z axis for L-PBF samples 0–20 μm, 0–40 μm, and 20–40 μm.

It can be seen that the crystallographic planes \{101\} are predominantly oriented normal to the horizontal plane of the sample, which also confirms the predominant orientation of the crystallographic lattice. It can be noted that the inverse pole figures for the samples differ in the form of the density distribution. This indicates a slope relative to the building direction of the preferred orientations of the crystallographic planes. Based on this, it can be assumed that, despite the general preferential orientation
of the \{101\} planes, a weak effect of the powder composition of the powder on the orientation of crystallites is observed. This presumably is associated with thermokinetic and crystallization conditions of the metal [14], which may turn out to be different during the L-PBF process for powders of various compositions. Due to the different bulk density of the powder material, laser radiation is absorbed differently by the powder layer [9, 10], therefore, the geometry of the melt pool changes, which leads to the slope of the preferential heat removal vector [14].

3.2. Vickers hardness
The differences in the structure of different sections of the sample could lead to a variation in hardness values. The average values, as well as the max and min hardness values of the L-PBF samples from three powder fractions, are represented in Figure 5. The size of the Vickers indentation imprint is much larger than the grain sizes of the individual structural components (Figure 5 contains an additional image on the right), so we can talk about the average hardness of all available structural components, the size of which was calculated on the basis of the obtained EBSD maps using the AZtec 3.4 software package. The results of counting the grain size are presented in Table 2, where \( D_{\text{min}} \) is the minimum grain size, \( D_{\text{max}} \) is the maximum grain size, \( D_{\text{mean}} \) is the average value, \( D_{\sigma} \) is the standard deviation.

| Type of sample | \( D_{\text{min}}, \mu m \) | \( D_{\text{max}}, \mu m \) | \( D_{\text{mean}}, \mu m \) | \( D_{\sigma}, \mu m \) |
|----------------|-----------------|-----------------|-----------------|-----------------|
| 0-20 \( \mu m \) | 10.7            | 119.5           | 30.6            | 19.44           |
| 0-40 \( \mu m \) | 10.7            | 127.5           | 31.7            | 19.42           |
| 20-40 \( \mu m \) | 10.7            | 160.5           | 29.4            | 19.44           |

Figure 5. Vickers hardness of the L-PBF samples. H-horizontal plane, V-vertical plane.

Thus, the average grain size is almost identical for samples obtained from the three studied powder fractions, and differences in hardness are not caused by the grain boundary factor.

Hardness is directly dependent on changes in the structure of the metal. The samples obtained are characterized by a pronounced anisotropy of the structure depending on the building direction (see Figures 1 and 2); therefore, hardness anisotropy is also observed depending on the building direction. Such anisotropy of hardness is characteristic of all the powder compositions (see Figure 5). The highest anisotropy of hardness values is characteristic of an L-PBF sample from a powder of 0–20 \( \mu m \) — the measured values on the horizontal and vertical planes differ most significantly (on the vertical plane, the hardness value is 16 HV higher). An L-PBF sample of 0–40 \( \mu m \) powder showed an average hardness anisotropy (difference of 5 HV). For a sample of 20–40 \( \mu m \), hardness anisotropy is practically absent. Such regularities can be attributed to small differences in the structure (Figure 4), a slight change in the
preferred orientation of the crystallographic planes relative to the building direction depending on the powder composition of the powder, which is reflected in the integral hardness.

Based on this, it can be assumed that the powder composition of the powder material does not significantly affect the structure and properties.

4. Conclusion

1. The structure is represented by a set of non-equiaxial crystallites, elongated in the building direction of the sample, rotated at different angles around the axis <110>, which coincides with the building direction. Thus, the structure in the XY plane is isotropic.

   At the same technological parameters of L-PBF, samples of powder 0–20 and 0–40 μm showed better metal quality, with lower porosity, while L-PBF samples of powder 20–40 μm are characterized by a large number of pores.

   It was found that the powder composition of AISI 321 steel powder does not fundamentally affect the morphology of the grain structure. Despite the general preferential orientation of the {101} planes, a weak effect of the powder composition on the orientation of the crystallites is observed.

   2. The effect of the powder composition of AISI 321 steel powder on the hardness of L-PBF samples is considered. The hardness anisotropy of the L-PBF samples increases when using a powder composition of 0–20 μm. The use of a powder material with a wide composition (0–40 μm in this study) allows one to achieve the lowest hardness anisotropy. Screening of a large (20–40 μm) or small (0–20 μm) compositions does not contribute to the production of an isotropic L-PBF sample.

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