Investigation of the properties of heat-resistant spherical powders

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Abstract. Nowadays, additive manufacturing (AM) become very perspective method for fast fabrication of any details, parts or products. One of the main methods of AM is selective laser melting (SLM), which allows to obtain complex shape parts from various metal powders. However, this process requires high demands on the initial materials. To determine the quality of the initial EP648, EP741I, and Hastelloy X powders, such methods as scanning electron microscopy, laser diffraction, reductive melting and X-ray were used.

1. Introduction
Spherical metal powders are the main raw material to produce products by various additive methods. One of the most promising AM methods is selective laser melting, which is based on layer-by-layer sequential fusion of metal powder by laser. This method allows you to get complex shape parts, which is technologically difficult or impossible to obtain by traditional methods [1]. SLM products, are widely used in various fields.

One of the most important advantages of this method is the ability to create products with desired properties by selecting a powder with appropriate characteristics [2, 3]. In this paper the results of impurity content, fractional and X-ray phase analysis of spherical heat-resistant powders EP648, EP741 and Hastelloy X are presented.

2. Materials and methods
All spherical powders were obtained by inert gas atomization method. The fractional composition of the powders was determined using a Fritsch Analysette 22 NanoTec laser diffraction particle size analyzer and consist up to 60 microns for EP648, from 20 to 80 microns for EP741, and from 20-120 microns for Hastelloy X. The composition of the initial powders is presented in table 1

| Steel grade powder | Ni  | Cr   | Mo   | Fe  | Co  | W  | C   | Si  | Al  | Nb  | Ti  |
|--------------------|-----|------|------|-----|-----|----|-----|-----|-----|-----|-----|
| EP648              |     |      |      |     |     |    |     |     |     |     |     |
| Bal.               | 32-35 | 2.3-3.3 | 4 max |     | 4.3-5.3 | 0.1 max | 0.4 max | 0.5- | 0.5- | 0.5- |
| EP741              |     |      |      |     |     |    |     |     |     |     |     |
| Bal.               | 8-10 | 3.5-4.2 | 0.5 max | 15-16.5 | 5.2-5.9 | 0.02-0.06 max | 0.5 max | 4.8-5.3 | 2.4-2.8 | 1.6-2 |
| Hastelloy X        |     |      |      |     |     |    |     |     |     |     |     |
| Bal.               | 22  | 9    | 18   | 1.5 | 0.6 | 0.1 | 1 max | 0.5 max | 0.5 max | 0.15 max |
The principle of operation of a laser diffractometer is based on measuring the angular distribution of the intensity of scattered light during the passage of a laser beam through a dispersed sample. Large particles mainly scatter light at small angles to the laser beam, while small particles - at large angles. The Mi theory of light scattering is able to determine the size of the particles that form the scattering indicatrix, which coincides with the measured data on the angular dependence of the intensity of scattered light. Particle size is expressed as the sphere diameter of equivalent volume. The study of the morphology of the powder particles was carried out using a Tescan Vega II SBU scanning electron microscope.

![Figure 1](image)

**Figure 1.** The granulometric composition of the powders: a - steel EP648, b - steel EP741, c - Hastelloy X alloy.

Figure 1 shows the particle size distribution of EP648, EP741 and Hastelloy X high-temperature alloy powders. All the studied powders have a rather narrow peak of particle size distribution, which is important for further use.
Figure 2. SEM images of powders: a - EP648, b - EP741, c - Hastelloy X.

Figure 2 shows the SEM images of powder particles. As you can see, most particles have a regular spherical shape. However, there are defects in the form of satellites on the surface of some particles, especially at Hastelloy X surface. Satellites are the most characteristic and inevitable defect of powder particles obtained by gas atomization. At the exit from the flow, the particles collide with each other and leave traces of impacts (Figure 2a) with full crystallization or sticking of particles occurs (Figure 2) with temperature differences. This process usually occurs during the collision of large particles (from the liquid phase) with small particles (from the vapor phase).

3. Results and discussion

The content of oxygen and nitrogen in the samples was determined by the method of reductive melting in a stream of inert gas with subsequent detection of oxygen in the infrared cell and nitrogen in the conductometric cell of the Leco TC-600 gas analyzer. Helium was used as a inert gas. To determine the hydrogen content in the samples, the method of reductive melting was also used, but only with the high purity argon as the inert gas. The detection of hydrogen was carried out in a conductometric cell of a Leco RHEN-602 gas analyzer. Determination of carbon content was carried out by the method of reductive melting in a ceramic crucible in an induction furnace and detected by the amount of evolved gaseous CO2 in the infrared cell of the Leco CS-600 gas analyzer. The results of the study are shown in table 2.
Table 2. Mass fraction of oxygen, nitrogen, carbon and hydrogen in the samples of powders.

| Steel grade powder | O, %   | N, %     | C, %  | H, %    |
|--------------------|--------|----------|-------|---------|
| EP648              | 0,065±0,005 | 0,11±0,001 | 0,07  | 0,00027 |
| EP741              | 0,024±0,003 | 0,0005 max | 0,05  | 0,00096 |
| Hastelloy X        | 0,016±0,003 | 0,022±0,001 | 0,075±0,001 | 0,0004±0,0002 |

As can be seen from the table, powders from steels EP648 and EP741 have a high oxygen content, which is caused, apparently, by the technology for their preparation. In the EP648 sample the nitrogen content is quite exceeded, which can lead to the formation of various defects (cavities, cracks, etc.) with the further fabrication of products by the method of selective laser melting. The content of carbon and hydrogen in all samples corresponds to the content in the monolithic material.

To obtain the phase composition of the powders, X-ray structural phase analysis was used. The diffraction spectra of the powders are presented in Figure 3. In all cases, the powder has a single-phase austenitic composition having an fcc lattice with a large grain size (> 1000 Å)
4. Conclusion
All studied powders have a single-phase austenitic composition with an fcc lattice and a large grain size. These powders also have a rather narrow peak in the particle size distribution and high sphericity. However, due to the peculiarities of the technology (gas atomization method), the satellites are found
on the surface of some particles (especially in Hastelloy X powder), as well as EP648 and EP741 powders have an increased oxygen content, which can provoke cracking during SLM.

Thus, the studied powders can be recommended for fabricating products using the SLM method.

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