Properties of spherical stainless steel powders

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Abstract. The additive technologies are gaining popularity due to the possibility of creating custom products for complex applications in many industries. Spherical metal powders are initial components for metal additive manufacturing. Initial materials are very important part of manufacturing, because their quality has an influence on stability of production process and quality of final product. In this work properties of spherical powders including 304L, 310, 316L and 17-4PH grade were determined using scanning electron microscopy, laser diffraction particle size analysis, reductive melting method and X-ray analysis.

1. Introduction
Additive manufacturing methods are becoming more common as there is a demand for rapid prototyping and production of parts that require design features that cannot be manufactured by the conventional press and sinter process normally used in powder metallurgy (for example biomedical implants) [1-7]. Additive manufacturing is process which builds a part layer by layer out of particulate material, a chemical binder and a digital design.

The powder characteristics most desirable for additive manufacturing are spherical powders with a fine particle size. Having a distribution of particles that flows and forms dense or well packed beds is important in both powders for additive manufacturing methods mainly for selective laser melting [8-9].

Stainless steel, a class of ferrous alloys, is well-known for their resistance to corrosion, creep and high temperature applications. Due to increasing demand of powder metallurgy components made from stainless steel in variety of applications, including aerospace, automotive, chemical processing and biomedical field, it became of a great interest in the research domain. In this work we studied the properties of spherical stainless steel powders of grades 310, 304L, 316L and 17-4PH products from which are widely used in various industries.

2. Methods and materials
Spherical stainless steel powders were obtained by gas atomization method. During the gas atomization process, the molten steel is atomized through the use of inert gas jets into fine metal droplets which cool down during their fall in the atomizing tower. Metal powders obtained by gas-atomization offer a perfectly spherical shape combined with a high cleanliness level. The chemical composition of steels is presented in Table 1.
Table 1. Chemical composition (mass %) of stainless steel.

| Steel grade powder | Cr, % | Ni, % | Cu, % | Nb, % | Mo, % | S, % | P, % | C, % | Si, % | Mn, % | Fe, % |
|--------------------|------|------|------|------|------|------|------|------|------|------|------|
| 310                | 24   | 19   | 0    | 0    | 0    | 0,03 max | 0,04 max | 0,08 max | 1 max | 2 max | Balance |
| 304L               | 18   | 11   | 0    | 0    | 0    | 0,03 max | 0,04 max | 0,03 max | 1 max | 2 max | Balance |
| 316L               | 16   | 15   | 0    | 0    | 3    | 0,03 max | 0,04 max | 0,03 max | 1 max | 2 max | Balance |
| 17-4PH             | 16   | 4    | 4    | 0,3  | 0    | 0,03 max | 0,04 max | 0,030 max | 1 max | 1 max | Balance |

The particle size distributions of powders were determined using an Analysette 22 NanoTec laser diffraction particle size analyzer from Fritsch. The particle size analyzer measuring cell contains the sample particles prepared by the dispersion unit which are irradiated with laser light. By changing the spacing between detector and measuring cell, a different angle range of the scattered light is detected. The particle size distribution is calculated from this data.

The phase composition was determined by an Ultima IV X-ray diffractometer (Rigaku, CuKα radiation) with a graphite monochromator that is intended for the use of different polycrystalline inorganic and organic substances, metals, alloys, films, coatings, and composites with a vertical goniometer and D/teX high-rate semiconductor detector using CuKα radiation according to the Bragg–Bretano method and by the oblique incidence technique with a fixed rotation angle of the X-ray tube.

The Leco TC-600 is a software-controlled instrument that measures both nitrogen and oxygen in a wide variety of metals, refractories, and other inorganic materials. The inert gas fusion principle is employed. A weighed sample, placed in a high-purity graphite crucible, is fused under a flowing helium gas stream at temperatures sufficient to release oxygen, nitrogen, and hydrogen. The oxygen in the sample combines with the carbon from the crucible forming primarily carbon monoxide (CO). In some instances, depending upon sample type and furnace temperature, some oxygen can be released directly as carbon dioxide (CO). The nitrogen present in the sample releases as molecular 2 nitrogen, and any hydrogen present is released as hydrogen gas. Hydrogen determination by inert gas fusion thermal conductivity detection of the Leco RHEN-602 gas analyzer. The Leco CS-600 system is designed for wide-range measurement of carbon and sulfur content of metals, ores, ceramics, and other inorganic materials.

Scanning electron microscope (SEM) Tescan Vega II SBU with an INCA Energy unit for energy dispersive analysis was used to study the surface morphology and to carry out the fractographic investigation of the specimens after the static tests.

3. Results and discussion
The particle size distributions of spherical stainless steel powders 304L, 310, 316L and 17-4PH is shown in Figure 1. 304L steel and 310 steel powders consist 10-30 μm size particles, 316L and 17-4PH – 40 and 120 μm respectively.
Figure 1. Particle size distribution of the powders: a - steel 304L, b - steel 310, c - steel 316L, d - steel 17-4PH.

The phase composition was determined by an Ultima IV X-ray diffractometer. X-ray diffractometer showed that 304L and 17-4PH steel powders consist of both austenitic and ferritic phases, steel 310 and 316L powders – only austenitic phase.

Measuring of oxygen, nitrogen, carbon and hydrogen content was carried out using gas analyzers TC-600 from Leco, RHEN-602 from Leco, CS-600 from Leco. The results of the study are shown in Table 2.

Table 2. Impurity content (mass %).

| Steel grade powder | O, %    | N, %      | C, %      | H, %      |
|--------------------|---------|-----------|-----------|-----------|
| 310                | 0,061±0,001 | 0,14±0,01 | 0,372±0,003 | 0,0016±0,0003 |
| 304L               | 0,066±0,003 | 0,21±0,01 | 0,030±0,001 | 0,0016±0,0004 |
| 316L               | 0,061±0,005 | 0,077±0,001 | 0,017±0,001 | 0,0008±0,0001 |
| 17-4PH             | 0,037±0,003 | 0,11±0,01 | 0,030±0,001 | 0,0008±0,0002 |

The carbon and hydrogen content in all powders is the same as in bulk stainless steel. Increased oxygen content in all samples may reduce the corrosion resistance of final product. In spherical stainless steel powders 304L nitrogen content also exceeded, that can cause the formation of defects (holes, cracks, etc.).

The study of the powder morphology was carried out using a Tescan Vega II SBU scanning electron microscope. SEM images (Figure 2) show that particles have a spherical shape. However, on the surface of some particles there are defects in the form of satellites. Satellites are the most characteristic and inevitable defect of powders obtained by gas atomization.
Figure 2. SEM images of powder: (a) - steel 304L, (b) - steel 310, (c) - steel 316L, (d) - steel 17-4PH

4. Conclusion
The spherical powder obtained by gas atomization has defects in the form of satellites, which can affect the fluidity of the powder. Increased oxygen and nitrogen content in all samples may reduce the corrosion resistance and formation of various defects in the final product. Application of spherical stainless powder will require determination of the correlations between the properties of the powders and the properties of final products.

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References
[1] Nasakina E O, Baikin A S, Sergiienko K V, Leonov A V, Kaplan M A, Seryogin A V, Konushkin S V, Myasnikova N V, Sevostyanova M A, Kolmakov A G, Simakov S V 2017 Formation and Investigation of Composite Material Silver–Nitinol for Medical Purposes Inorganic materials: applied research 8 №1 pp 112–117
[2] Nasakina E O, Baikin A S, Sevostyanov M A, Kolmakov A G, Zabolotnyi V T, Solntsev K A 2014 Properties of Nanostructured Titanium Nickelideand Composite Based on It Theoretical Foundations of Chemical Engineering 48 №4 pp 477–486
[3] Kaplan M A, Sevost’yanov M A, Nasakina E O, Baikin A S, Sergienko K V, Konushkin S V, Kolmakov A G 2018 Influence of the Surface Modification on the Mechanical Properties of
NiTi (55.8 wt % Ni) Alloy Wire for Medical Purposes Inorganic Materials: Applied Research 9 №4 pp 751–756

[4] Yap C Y, Chua C K, Dong Z L, Liu Z H, Zhang D Q, Loh LE, Sing S L 2015 Review of selective laser melting: Materials and applications Appl. Phys. Rev. 2 041101

[5] Kruth J P, Froyen L, Vaerenbergh J V, Mercelis P, Rombouts M, Lauwers B 2004 Selective laser melting of iron-based powder J. Mater. Process. Technol. 149 pp 616-622

[6] Schleifenbaum H, Meiners W, Wissenbach K, Hinke C 2010 Individualized production by means of high power Selective Laser Melting CIRP-JMST 2 №3 pp 161-169

[7] Yadroitsev I, Gusarov A, Yadroitsava I, Smurov I 2010 Single track formation in selective laser melting of metal powders J. Mater. Process. Technol 210, pp 1624–1631

[8] Wohlers T 2014 Additive Manufacturing and 3D Printing State of the Industry Annual Worldwide Progress Report. Wohlers AssociatesInc 275 p

[9] Smirnov M A, Kaplan M A, Sevostyanov M A 2018 Receiving finely divided metal powder by inert gas atomization IOP Conf Ser Mater Sci Eng. 347 pp 1-5