The issue of quality assurance of materials in additive manufacturing of metal products

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Abstract. The article addresses the quality issues of additive manufactured products from the perspective of materials science. Additive manufacturing is multilayered step-by-step deposition of a material in micro-volumes following the trajectory controlled by a CNC program. Such heating sources as electric arc, laser, plasma and electron beam can be used to melt the metallic powder. This article investigates the structure and properties of titanium alloy Ti-6Al-4V ELI produced by selective laser melting (SLM). The samples were produced in pure argon environment in a system with a 3D printer EOS M290 (Electro Optical System GmbH, Germany) and then heat-treated in vacuum at 750…800 °C during 3 hours. It was revealed that the main structural defects reducing mechanical properties of material are pores and stress concentrators in the points of contact between titanium alloy particles and the newly built surface of the previous layer. The ways aimed to avoid the structural defects which reduce fracture toughness were suggested.

1. Introduction
Additive technology provides the ability to produce different complex geometric shapes without necessity to create any complex special equipment. It makes this method one of the most promising and commercially viable for single-piece or small-scale manufacturing of metallic products. In this area, a great practical experience has been gained with the use of laser and electron-beam sources [1]. The technological process can include one-step or multistep processes, when after forming a geometric shape the mechanical properties of materials can be modified. Heat treatment, as the second step of technological process, usually is applying for residual stress relief, and in some cases for additional structural homogenization [1].

Achievements in SLM showed the possibility to reduce the costs for single-piece manufacturing and to decrease the number of details in assembly units due to the complex design of the basic parts. All the materials (pure metals, alloys and composite materials with metallic matrix) applying in machine building theoretically can be used as the "building material" for additive manufacturing details for machines, mechanisms, devices and instruments. According to the world experience, even the most advanced SLM of powders can't always provide the proper quality of material, which is required for some products working as machine details and structural elements for important exploitation. Protective gas environment significantly expands technological capabilities and simplifies the process of 3D-printing, but can't eliminate porosity even when using such high-concentrated energy sources as laser [2], and electron beam [3]. Recent ten years in scientific publications much attention is paid to issues of quality of titanium alloy Ti-6Al-4V as the widely spread material in different areas of manufacturing. Instead of wide use of additive manufactured Ti-
6Al-4V products one of the urgent tasks is eliminating of structure defects as pores in the formed material. Another problem mostly concerns the stability of sintering process and anisotropy control of the structure, which appears in welded layers in local volumes of melted material during electron-beam [3] and laser heating [4].

2. Materials and methods
The structure and mechanical properties of materials obtained by SLM were studied on samples made of Ti-6Al-4V ELI powder alloy. The chemical composition of powder was determined using an ARL X-ray fluorescence spectrometer Optim X. The particle size distribution of the original powder was studied using a Nikon MM-400 instrumental microscope; spherical particles were ranging in size from 20 to 46 µm.

| The content of chemical elements in powder titanium alloy [wt%] |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|----------------|
| V               | Al              | Fe              | W               | Mg              | Ni              | Si              | Mn              | S               |
| 3.76 ± 0.18     | 5.54 ± 0.11     | 0.394 ± 0.02    | 0.098 ± 0.011   | 0.052 ± 0.004   | 0.051 ± 0.003   | 0.012 ± 0.002   | 0.011 ± 0.004   | 0.006 ± 0.001   |
| Ti              | Balance         |

The SLM was carried out in argon environment on a EOS M290 3D-printer (Electro Optical System GmbH, Germany), equipped with an ytterbium fiber-optic laser with a wavelength of 1.065 µm with an output power 400 W. The samples were built up on auxiliary supports on Ti-6Al-4V thick plate (Figure 1), which was attached to the desktop of the printer. Using the massive plate as a substrate prevents the deformation of the samples during their manufacture. Then the plate with the samples was annealed in vacuum at 750 °C for 3 hours with the heating rate not higher than 1 °C per second. Heat treatment of the samples with the plate allows relieving residual stresses and avoiding uncontrolled deformation of the samples after cutting them off the plate.

Samples for tensile tests with the size 150 × 30 × 3 mm and samples for impact bending tests with the size 60 × 12 × 2 mm were printed with an allowance to cut them off from the slab using a wire-spark machine (Fig. 2 a, b). Then the all sides of the samples for testing were grinded. The thickness of the sintered powder layer, the scanning direction of each layer and the scanning speed were controlled using software, which takes into account the product geometry and minimizes the value of residual stresses in the formed product.

To evaluate the level of stresses that appears after sintering, a flat sample 60 × 12 × 2.5 mm was sintered which was not heat treated. After breaking off the supports from the plate, the long side surfaces of the sample lost their flatness (Fig. 2c). To find out the cause of the curvature of the surfaces, the analysis of the stresses by the criterion of the maximum von Mises was carried out using the software package SYSWELD.

Figure 1. SLS samples of Ti-6Al-4V ELI alloy made for mechanical testing and grid-shaped products on supports (upper left part of the plate) built up on titanium alloy Ti-6Al-4V table plate.
Figure 2. Samples for mechanical testing, made of titanium alloy: a - flat samples for tensile testing; b - flat samples for impact strength testing; c - not heat treated sample after sintering.

Tensile tests were conducted on an Instron 3369 testing machine, impact strength testing was carried out on a pendulum copra Metro Com 06103300. The structure was investigated using a Carl Zeiss AxioObserver Alm optical microscope.

3. Results and discussion

The stresses leading to the distortion of the samples and products appear while crystallization of the melted powder layers. Heat treatment of the samples with the plate was preformed to avoid the possible distortion when stress release after cutting the samples from the plate. Not heat-treated sample has a dome-shaped 1.5 mm deflection along the length of the sample and 0.2 mm along the width (Figure 2c). The curvature occurs because of the sequential multilayered crystallization of local volumes when the beam is scanning on the powder layer along the path on the already crystallized (previous) layer.

While modelling, the following assumptions were made to simulate the level of the stresses: the stresses in the sample were formed only during the surfacing of the last layer 0.1 mm thick, and the physic and mechanical properties of the underlying layers corresponded to the properties of Ti-6Al-4V ELI alloy after high-temperature annealing. The laser beam moved from the upper corner of the sample along its short side with the scanning speed 7 m × s⁻¹ and the scanning step 140 µm. The largest calculated tensile stress in the sample with sizes 60 × 12 × 2.5 mm³ was 314 MPa, which is 40% of the yield strength of the Ti-6Al-4V ELI alloy. The propeller-like deformation behavior of the sample matches with the calculated deformation, which indicates that the sequence formation of local volumes of material influences on the generated stresses, on the deviations from the specified surface shape, and on the deviations of their relative position during Bed Deposition. The samples annealed with the massive plate had flat parallel surfaces with the deviation from the flatness within the roughness, which is determined by the fused particles on the surface (Figure 3a) and the surface tension of the melt on the upper deposited layer (Figure 3b).

The surface roughness of the samples was estimated on ZYGO 7300 microinterferometer. The measurements showed that the roughness had a clear periodic profile. The highest Ra parameter (average deviation of the profile) was 5.167 µm and the Rms parameter (standard deviation of the profile) was 6.433 µm. Before mechanical testing the samples with the size 150 × 30 × 3 mm³ were grinded, and the fillets were made. Thus, size of the testing parts was 10 mm. The tensile strength of the formed material was 1182 ±15 MPa, which is slightly higher than the strength values for deformed and annealed alloy (1103 MPa) and significantly higher than the values of the cast alloy (895 MPa). Comparison with the results of tensile strength tests given in [5] showed that the tensile strength of tested samples is at the same level [5] (1115 MPa). Comparison of the tensile test results carried out by the authors of the present study with the results of [5] is quite correct, since they were obtained at similar modes of scanning, heat treatment, and subsequent mechanical treatment to remove welded powder particles that works like stress concentrators. However, in the compared cases, the parameters of the process significantly differed: laser power, the rate of scanning beam relatively to the sintered
layer, the location of the sample being formed relative to the object table (in [5] the samples were formed vertically). Two-stage heat treatment allows to increase the values of tensile strength, for instance, the authors of [6, 7] achieved the values of 1269 ± 10 and 1271 ± 8 MPa.

The tensile strength of produced samples has deviations less than ±1.2%, nevertheless, the values of yield strength, relative elongation and relative narrowing of the simultaneously sintered samples (sintered under the same conditions) have a significant variation (σₚ = 844 ± 261 MPa; ψ = 13.1 ... 20.3%; δ = 8.9 ... 13.3%).

Metallographic studies of the samples showed the presence of pores in the samples and grid-shaped products made by SLM, both before and after heat treatment (Figure 4). The presence of pores after the sintering process has been noted by the most studies dedicated to SLM of Ti-6Al-4V ELI powder alloy [8–12].

Pores have a negative effect on impact resistance. The impact strength (KCV) was 32 ... 36 J × cm⁻², while the impact toughness values for cast alloys of similar composition is 50 ... 100 J × cm⁻². The formation of pores in the formed material is associated with the capture of inert gas atoms and uneven heating of the particles of the powder material by a laser beam. The pores work as stress concentrators under impact action and their shape is well observed in the fracture surface (Figure 5).

The structure of the titanium alloy Ti-6Al-4V directly after SLM, as a rule, is a martensitic α'-phase [13]. The cooling rate can reach 10⁴ °C × s⁻¹, which promotes the formation of a fine needle-shaped morphology. Annealing at 730-780 °C after SLM allows eliminating stresses in the material.
According to the literature, annealing of Ti-6Al-4V ELI at above mentioned temperatures for 2...3 hours leads to a partial decomposition of α'-martensite into more stable α and β phases. In the studied material, annealed at 750 °C for 3 hours, only a trace of the most intense β-phase reflex was detected by X-ray phase analysis, which indicates that only a small amount of the phase can possibly exist.

Metallographic study of the annealed structure clearly reveals the boundaries of the initial β-grains. Due to the fact that the initial martensitic structure of the Ti-6Al-4V ELI alloy is characterized by a high temperature stability [13, 14], the boundaries of the initial β-grains do not undergo changes after annealing. It should be noted that in different directions the morphology and dimensions of the primary β-phase grains are not the same. In the vertical cross-section, the grains have an elongated shape (Figure 4 a), while at the same time in the horizontal cross-section the material has an equiaxial structure (Figure 4 b). This structure feature can be explained by their epitaxial growth during the layer-by-layer melting of the powder material [13].

![Figure 5. Fracture surface of the alloy Ti-6Al-4V, obtained by laser layer-by-layer deposition and heat treatment (of pores in the center of the images).](image_url)

Due to the presence of gaps between particles the laser beam can heat and melt the particles not only by directly hitting on the particle but also because of the reflection of the beam from other particles [9]. The distribution of the power density and the incident angles of the laser beam on the particles lead to uneven heating within the beam spot from temperatures that are quite below the melting point to temperatures of the evaporation. Degassing of the deposited layers can be provided either by additional thermomechanical treatment or by implementing the SLM process in vacuum. Vacuum allows not only to conduct degassing the melt, which has a positive effect on the mechanical properties of the material, but also to apply electron beam as the source of heating. Electron-beam heating makes it possible to obtain products using the “Direct Deposition” method, in which the cladding of the layers is provided by melting filaments of metallic materials. Electron-beam layered surfacing in vacuum has its own benefits and drawbacks. In comparison to “Bed deposition” the main drawback of vacuum electron-beam surfacing is difficulty with the formation of smooth surfaces, thus, it’s hard to control the shape of the products been formed. The main benefit of electron-beam methods of metal additive manufacturing are quality of the formed material, high performance of the process, and the low cost of raw materials, since the filaments are much cheaper than powder materials. Another advantage of electron-beam methods is that the maximum temperature of a metal material bombarded by an electron beam is achieved in the thin surface layer at a distance from nanometers to hundreds of micrometers from the surface. This feature of electron-beam heating allows avoiding uncontrollable transition of the molten metal to gaseous state. Besides, from 75 to 95% of electron beam energy spends into heating, which makes the process more energy efficient in comparison with laser heating. Electron beam heating allows to use metal filaments from a tenth of a millimeter to 5 mm thick when focusing the beam diameter from 200 nm to tens of millimeters, which potentially...
allows for both surfacing and heat treatment of any metallic material: from low-melting metal alloys to tungsten.

4. Conclusion
Selective laser melting of powder materials doesn't allow avoiding the formation of structural defects like pores. Heat treatment of products formed by additive welding makes possible reducing the residual stress that appear while layer-by-layer depositing, but is not sufficient to eliminate pores.

Different sequence of the laser beam moving direction on the deposited powder layer affects the geometry of the resulting surfaces. Heat treatment with alternating layer-by-layer depositing makes possible to reduce the shape deviations of the formed products.

The elimination of such defects in structure as porosity and the improvement of physical and mechanical characteristics of additive manufactured materials can be achieved using vacuum electron-beam heating and filaments as the row material.

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