Microstructure and properties of Ti-6.5Al-3.5Mo-1.5Zr-0.3Si parts produced by electron beam melting

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Abstract. The aim of this work is to study the influence of electron beam current on the microstructure, structural-phase state and the change in microhardness of Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy samples. The samples were fabricated by electron beam melting (EBM) at different beam currents (3-6 mA). The phase composition and microstructure were analyzed by X-ray diffraction and scanning electron microscopy, respectively. The peculiarities of structure and properties change of samples made by electron beam melting from Ti-6.5Al-3.5Mo-1.5Zr-0.3Si powder have been established, depending on the manufacturing parameters.

1. Introduction
Additive manufacturing (AM) are being actively implemented in many industries due to high rate of production and possibility of manufacturing products with unique shape and geometry over traditional methods. Moreover, AM allows creating a new generation of materials with unique set of properties. Electron beam melting is one of the most perspective methods for creation three-dimensional products [1]. This method has the following advantages: high resolution in the horizontal plane, high performance; this method does not require subsequent heat treatment to achieve high mechanical strength of manufactured materials. The features of microstructure formation in metallic materials produced by EBM depends on the thickness of samples [2], beam power [3], beam speed, scanning strategy, line offset [3-5], etc. Titanium alloys are widely used in the automotive, aerospace and chemical industries as well as in medicine due to a combination of such properties as high strength, low specific weight, high corrosion resistance, good biocompatibility [6]. The most studied titanium alloy produced by additive manufacturing is Ti-6Al-4V alloy [1, 3, 5]. Ti-6.5Al-3.5Mo-1.5Zr-0.3Si (TC11) titanium alloy is a heat-resistant martensitic alloy and is widely used in the aerospace industry [7]. At the same time, there is no data about the structure and properties of products obtained from the powder of Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy by AMEBM method. There is a large number of works about selective laser melting of Ti-6.5Al-3.5Mo-1.5Zr-0.3Si titanium alloy [8-10]. However, the influence of electron beam melting parameters on the structure and properties of the Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy is still unclear. The aim of this work is to study the beam current
effect on microstructure and hardness of Ti-6.5Al-3.5Mo-1.5Zr-0.3Si parts produced by electron beam melting.

2. Materials and Experimental Procedure

2.1. Samples Preparation

The samples were produced from Ti-6.5Al-3.5Mo-1.5Zr-0.3Si powder using electron beam melting machine designed in Tomsk Polytechnic University. The sizes of the samples were 20×20×2 mm³. During manufacturing, the following parameters were used: the operating vacuum 5·10⁻³ Pa, build area 150×150 mm², beam power 6 kW. The substrate was stainless steel. The diameter of the electron beam during heating was 4 mm; electron beam speed during heating was 16000 mm/s; beam current (I) was 3, 4 and 6 mA. The spherical Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy powder was produced by Beijing AMC Powders Co Ltd Company (Chinese). The SEM images of the metal powder and the particle size distribution of the powder are shown in figure 1. The composition of the powder, according to the energy-dispersive analysis, is given in table 1.

![Figure 1. The SEM images of the powder Ti-6.5Al-3.5Mo-1.5Zr-0.3Si (a) and the particle size distribution of the powder (b).](image)

**Table 1.** The EDA of the powder Ti-6.5Al-3.5Mo-1.5Zr-0.3Si.

| Element | Ti     | Al     | Mo    | Zr   | Si    |
|---------|--------|--------|-------|------|-------|
| at.%    | 87.76  | 6.49   | 3.8   | 1.59 | 0.36  |

2.2. Experimental Procedure

The microstructure of the samples was analyzed by scanning electron microscopy (SEM) using a SEM 515 microscope (Philips, Eindhoven, The Netherlands). Before microstructural studies, the samples were etched in Kroll's reagent (1.5%HF, 2.5% HNO₃, and 96% H₂O). The phase identification and structural investigations were performed by X-ray diffraction. The X-ray diffraction studies were performed with CuKα radiation (1.5410 Å wavelength) using XRD-7000S diffractometer (Shimadzu, Kyoto, Japan) in Bragg-Brentano geometry from 30° to 80° with the scan speed of 10.0°/min, the sampling pitch of 0.0143°, the present time of 42.972 s at 40 kV and 30 mA. Microhardness testing was done on KB30S (Pruftechnik, Ismaning, Germany) Vickers hardness testing machine with 0.5 kg load.

3. Results and Discussion

Figure 2 shows SEM images of the surface of titanium Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy produced by electron beam melting. The structure of samples contains the alpha phase in the form of plates; the beta phase is represented in the form of rods between the alpha plates (figure 2). The dimensions of
the alpha plates increase from 0.33 to 0.8 μm with rising the beam current from 3 to 6 mA, respectively. The formation of such microstructure is probably related to the cooling rates of the melt pool during manufacturing. The cooling rate affects the microstructure of titanium alloys under thermal treatment [10]. The change in the cooling rate regulates not only the dispersion of the secondary α-phase plates, but also the final phase composition of the Ti alloys. Under slow cooling the secondary α-phase is preferably precipitates on the plates of primary α-phase. At higher cooling rates, the α phase can precipitate in the β-phase interface in the form of plates, which size decreases with increasing cooling rate. In the works of Jamshidinia [11] it is noticed, that the beam current increasing leads to the enlargement of the melt pool size and cooling rate decreasing.

![Figure 2. Surface SEM images of Ti-6.5Al-3.5Mo-1.5Zr-0.3Si titanium alloy produced by electron beam melting at different beam currents: a – 3 mA, b – 4 mA, c – 6 mA.](image)

| Beam current, mA | Phase          | Phase content, vol.% | Lattice parameters, Å | The size of crystallites, nm | Microstrain, 10^-3 |
|-----------------|----------------|----------------------|-----------------------|----------------------------|-------------------|
| 3               | Ti_hexagonal   | 88.0                 | a=2.9267              | 30                         | 0.609             |
|                 | Ti_cubic      | 12.0                 | a=3.2384              | 18                         | 1.079             |
| 4               | Ti_hexagonal   | 85.6                 | a=2.9261              | 34                         | 0.148             |
|                 | Ti_cubic      | 14.4                 | a=3.2475              | 20                         | 2.075             |
| 6               | Ti_hexagonal   | 83.9                 | a=2.9280              | 31                         | 0.317             |
|                 | Ti_cubic      | 16.1                 | a=3.2391              | 14                         | 3.302             |

Table 2. XRD of titanium Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy produced by EBM.
Table 2 shows the structural-phase state of Ti-6.5Al-3.5Mo-1.5Zr-0.3Si titanium alloy produced by EBM at different beam current. Microstructure of samples contains α phase with hexagonal close-packed crystal structure and β phase with volume-centered crystal modification (table 2).

It is established that when the beam current changes, the microstrain in the β phase of titanium increase. In additional, a change in the volume content of the β phase is observed depending on the electron beam melting parameters. The highest content of β phase (≈16% by volume) is observed in samples produced at beam current of 6 mA; the smallest content of β phase (≈12% by volume) is observed in samples produced at beam current of 3 mA.

The results of microhardness measurements are shown in figure 3. It is established that the microhardness decreases with increasing beam current. The decrease in the microhardness of the samples is due to an increase in the volume fraction of the β phase in the material accompanying raising of beam current (table 2). The increase in the dimensions of the alpha-plates can also have an effect on the microhardness.

![Figure 3](image)

Figure 3. The microhardness of the samples produced by EBM at beam current: a – 3 mA, b – 4 mA, c – 6 mA.

4. Conclusion
The microstructure and hardness of samples manufactured by electron beam melting from Ti-6.5Al-3.5Mo-1.5Zr-0.3Si powder was investigated. The increase in the beam current from 3 to 6 mA leads to enlargement of the alpha-plates in the structure of the manufactured samples. The samples prepared at beam current 6 mA have greatest beta phase content as well as the highest values of microstrain. The hardest samples of titanium Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy are manufactured at beam current of 3 mA, which is related to lower content of the beta phase and the finer microstructure of material.

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