The effect of homogenizing annealing on the microstructure and microhardness of Ti-20Nb-(7.5-10)Ta alloys (at.%)
Figure 1. Diagram of Ti-Nb-Ta alloys [1]
1 - Ti-20Nb-7.5Ta alloy; 2 - Ti-20Nb-10Ta alloy.

The alloy ingots of the selected compositions were smelted by the vacuum-arc remelting method in a water-cooled copper crystallizer in an argon medium. The ingots had the shape of “boats” 20 mm long, 10-12 mm wide, 8-10 mm high, weighing 15 g. After smelting, the ingots were cut into pieces 4-5 mm thick across the length and annealed in vacuum at a temperature of 900-950°C for 12 hours at a pressure of 3*10⁻⁵ mm Hg. The temperature range was chosen on the basis of published data based on their task to align the chemical composition along the cross section of the ingot, while not allowing excessive grain growth [3].

Samples were prepared for metallographic studies by sequential grinding after pressing on a Piatto diamond disk with a grain size of P120 for 3 minutes, P320 for 5 minutes, P800 for 5 minutes, on an Aka-Allegran-3 fine grinding diamond disk with DiaMaxx Poly suspension with sizes of diamond particles 6 and 3 microns, on Akasel DARAN woven acetate with a DiaMaxx Poly suspension with a diamond particle size of 1 μm; on Akasel CHEMAL foamed neoprene with an aluminum oxide suspension with a particle size of 50 nm. The microstructure was revealed by etching in a solution of hydrofluoric and nitric acids with distilled water in a volume ratio of HF: 3H₂SO₄: 16H₂O. Sections were wiped with a solution for 30-60 s, washed with water and ethyl alcohol. The study was performed using a Neophot 2 light microscope manufactured by Carl Zeiss Jena. After etching and analysis of the microstructure, the microhardness of the samples was investigated. To study the microhardness, the Vickers method was used. The load was 300 g, exposure 15 sec.

3. Results and discussion
3.1 The study of the microstructure
As can be seen from Figure 2, the microstructure of the ingots after smelting is grains of oppositely directed dendrites. The axes of dendrites with increased content of refractory elements (Ta and Nb) (light areas of the microstructure - Figure 2a), the more etched inter-axis dark zones (Figure 2a) with larger content of Ti, and the etched grain boundaries of the dendrites (Figure 2b) are visible in the photographs.
Figure 2. The microstructure of the ingot Ti-20Nb-7.5Ta (a) and Ti-20Nb-10Ta (b) after smelting 1 - dendrite, 2 - the area between the "branches" of the dendrite, 3 - the grain boundaries of the dendrite.

The dendritic structure is formed as a result of uneven cooling of the molten ingot due to more rapid cooling of the lower part of the ingot in contact with the copper mold compared to the upper part. Initially, at the point of contact of the ingot with the copper tray “branches” containing more refractory elements (Ta and Nb) crystallize and begin to grow along the temperature gradient. Between the “branches” there remain areas containing less refractory elements (Ti), which solidify later. The resulting dendritic structure is a defect that is corrected by homogenizing annealing.

The structure of the ingots of the selected alloys after annealing at 900 °C for 12 hours is shown in Figure 3a, b.

Figure 3. The microstructure of the ingot Ti-20Nb-7.5Ta (a) and Ti-20Nb-10Ta (b) after annealing at a temperature of 900 °C for 12 hours, 1 - needle discharge, 2 - point discharge.

As can be seen from the photographs of the microstructure presented in Figure 3, grains recrystallized and dendrites dissolved. Samples consist of rather large equiaxed grains with sizes of 0.2-0.4 mm with boundaries of 120 °.

The structure of the ingots of the selected alloys after annealing at 900 °C for 12 hours is shown in Figure 4.
Figure 4. The microstructure of the ingot Ti-20Nb-7.5Ta (a) and Ti-20Nb-10Ta (b) after annealing at a temperature of 950 °C for 12 hours: 1 - needle discharge, 2 - point discharge.

As can be seen from the photographs of the microstructure presented in Figure 4, there was a further growth of grains to sizes of 0.5-0.7 mm. The boundaries remained equal to 120°.

Micrographs of the samples show the separation of needle-shaped (Ti-20Nb-7.5Ta alloy, Figure 3a, 4a) and point (Ti-20Nb-10Ta alloy, Figure 3b, 4b) structural elements inside the main grains. Perhaps this is the α-phase, precipitated as a result of decay after annealing and slow cooling to room temperature of the main β phase, which is characteristic of high alloy titanium alloys. To confirm or refute this statement, an XRF of melted and annealed samples will be carried out in the future.

3.2 Microstructure study

The alloy samples microhardness test results are presented in Figure 5.

Figure 5. Microhardness of Ti-20Nb-7.5Ta (a) and Ti-20Nb-10Ta (b) alloys samples after smelting and homogenizing annealing at temperatures of 900°C and 950°C, respectively.

As can be seen from the research results, the microhardness of the alloys increases significantly after annealing for both alloys. The microhardness of the Ti-20Nb-7.5Ta alloy after annealing at 900°C increases by 20.9%, and after 950 ° C - by 32.5%. The microhardness of the Ti-20Nb-10Ta alloy after annealing at 900°C increases by 22.3%, and after 950°C - by 24.3%. We can note a general decrease in the effect of annealing on the microhardness of the Ti-20Nb-10Ta alloy compared to the Ti-20Nb-7.5Ta alloy. After smelting (by 8%) and after annealing (by 7% during annealing at 900°C and by 14% during annealing at 950°C), this also indicates the plasticizing effect of Ta on these alloys. In general, an
increase in the microhardness of all samples after annealing, possibly indicates the release of additional phases, which is partially confirmed by the microstructural studies described in the previous paragraph. The expected decrease in the microhardness of the alloys due to the removal of thermal stresses arising during the cooling of the ingot after smelting was not found [4].

4. Conclusions
1. After smelting using vacuum-arc remelting, Ti-20Nb-(7.5-10)Ta (at. %) alloys have a dendritic structure.
2. Homogenizing annealing at 900°C and 950°C leads to the dissolution of dendrites and recrystallization of the alloy.
3. Homogenizing annealing leads to the release of additional phases in the grains.
4. The microhardness of alloys after homogenizing annealing increases significantly.
5. The plasticizing effect of Ta on the microhardness of Ti-20Nb-(7.5-10)Ta alloys is shown.

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