The structure of the alloy Ti - (20-30) Nb - 5Zr after smelting and homogenizing annealing

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Abstract. The formation of the Ti- (20–30) Nb-5Zr alloys (at.%) structure was studied. The structure of the samples was determined using a light microscope. It is shown that after melting by the method of vacuum arc melting, the alloys have a developed dendritic structure. Homogenizing annealing at a temperature of 900 °C does not lead to the destruction of the dendritic structure and recrystallization. Annealing at a temperature of 1000 °C completely destroys the dendritic structure in the alloys.

1. Introduction

Currently, Ti-based alloys not containing Ni exhibiting the shape memory effect and superelasticity are actively studied. These alloys are considered as possible candidates for the replacement of the nitinol widely used in medicine, since nickel carcinogenic properties limit further medical use of this alloy [1-5]. The solution to this problem may be to create biocompatible corrosion-resistant surface layers on it [5-14]. However, in this case, the question of the mutual adhesion of the components arises, especially under alternating dynamic loads.

Alloys based on the Ti-Nb-Zr system are of interest for orthopedics and endovascular surgery. Alloy properties, incl. the magnitude of the shape memory effect and superelasticity, depend on the chemical composition, so for the accuracy of the research it is important to ensure the uniformity of the chemical composition of the alloy. For this it was important to work out the technology of smelting and homogenizing annealing of the selected alloys ingots [15-18].

In this paper, we investigated the structure of ingots of Ti-Nb-Zr system alloys after smelting and homogenizing annealing.

2. Materials and methods

The alloys of the Ti-Nb-Zr systems of the following compositions were studied: Ti-20Nb-5Zr, Ti-23Zr-5Zr, Ti-25Nb-5Zr, Ti-28Nb-5Zr, Ti-30Nb-5Zr (at.%). The alloys were smelted by the method of vacuum electric arc melting from pure charge materials. Ingots were obtained in the form of a biconvex lens with a diameter of 40 mm and a thickness in the center of 10-12 mm, weighing 30 g. The ingots were annealed in vacuum at a temperature of 900-1000 °C for 12 hours in a vacuum of 10−3 millimeters of mercury and cooled with the furnace. The temperature range of the homogenizing annealing was chosen, assuming that at 900 °C the processes of dendrite dissolution and recrystallization will begin, and after annealing at a temperature of 1000 °C a fully recrystallized β-phase will form.
Preparation of samples for metallographic studies was carried out by grinding on P320 abrasive paper, polishing on a diamond disk with a suspension with a size of polycrystalline diamond particles of 3 and 1 μm, and polishing on a felt disk with a suspension of colloidal quartz with particle sizes of 0.1 μm. The microstructure was detected by etching in an HF: 3HNO3: 5H2O solution and examined using a Neophot 2 light microscope.

3. Results and discussion
As shown by a light microscopy study in a state after smelting the structure of Ti-Nb-Zr alloys has 3 distinct zones over the ingot cross section (figure 1). The zone adjacent to the bottom of the copper crystallizer during smelting is consist of large homogeneous grains. The width of this zone was about 1 mm in the center of the ingot and decreased to its ends. Then there is a zone of dendritic grains, mainly oriented perpendicular to the crucible bottom. The grain size is about 1-1.5 mm wide and up to 5 mm long. Next the zone with smaller and multidirectional dendrites comes. There dendrites axis of the 1st and 2nd orders are visible. The microstructure of these ingot zones is presented in figure 2.

Figure 1. The microstructure of the Ti-Nb-Zr alloy ingot after smelting: a - the upper part of the ingot; b - the middle part of the ingot; c - the lower part of the ingot.
Figure 2. The microstructure of the ingot after smelting: (a) - in the lower part of the ingot; (b) - transition zone between the lower and middle parts of the ingot; (c) - in the middle part of the ingot; (d) - transition zone between the middle and upper parts of the ingot; (e) - in the upper part of the ingot. 1 - codirectional branches of dendrites, 2 - multidirectional branches of dendrites

Figure 2a shows the grain structure of the lower zone of the ingot. Grains have a size of up to 1 mm and are directed perpendicular to the bottom of the mold. Next, comes the transition zone between the lower and middle parts of the ingot (figure 2b), in which the continuous, uniform structure is replaced by the dendritic one. The middle part of the ingot consists of large grains of co-directed dendrites elongated along the height of the ingot (figure 1, figure 2c). Figure 2d shows the transition zone between the middle and upper parts of the ingot, where the codirectional branches of the dendrites are replaced.
The microstructure of ingots of different Ti-Nb-Zr compositions is shown in figure 3. As can be seen from the presented images, the microstructure of Ti-Nb-Zr alloys does not have significant differences in the shape and size of its elements. Characteristic features for all alloys are a developed dendritic structure in the upper part of the ingot and a coarse-grained structure in the lower part of the ingot.

The dendritic structure of the melted ingot is a marriage because is a chemical liquation. To eliminate the dendritic structure, long-term homogenizing annealing is used to activate the diffusion processes and equalize the chemical composition. The structure of the ingots after annealing at a temperature of 900 °C for 12 hours is shown in figure 4.

As can be seen from the microstructure images annealing at a temperature of 900 °C led to a significant dissolution of dendrites and recrystallization in the Ti-20Nb-5Zr alloy. The boundaries of equiaxed grains began to be etched. The grains of dendrites in the ingots of the remaining alloys are still well etched.

For further dissolution of dendrites and recrystallization, alloy ingots were annealed at a temperature of 1000 °C for 12 hours. The structure of the ingots is shown in figure 5. As can be seen from it annealing at a temperature of 1000 °C led to the growth of grains in the Ti-20Nb-5Zr alloy and complete dissolution of the dendritic structure. In the Ti-23Nb-5Zr, Ti-25Nb-5Zr, Ti-28Nb-5Zr, and Ti-30Nb-5Zr alloys, the boundaries of polyhedral grains and subgrains also began to be etched.

Grains reached a size of 200-400 microns, which is quite suitable for further plastic deformation of melted ingots.
Figure 3. Dendritic structure of Ti-Nb-Zr alloy ingots after smelting: (a) - Ti-20Nb-5Zr; (b) - Ti-23Nb-5Zr; (c) - Ti-25Nb-5Zr; (d) - Ti-28Nb-5Zr; (e) - Ti-30Nb-5Zr.
Figure 4. The structure of ingots of Ti-Nb-Zr alloys after annealing at 900 °C: (a), (b) - Ti-20Nb-5Zr; (c), (d) - Ti-23Nb-5Zr; (e), (f) - Ti-25Nb-5Zr; (g), (h) - Ti-28Nb-5Zr; (i), (j) - Ti-30Nb-5Zr
Figure 5. The structure of the ingots of alloys Ti-Nb-Zr after annealing at 1000 °C: (a), (b) - Ti-20Nb-5Zr; (c), (d) - Ti-23Nb-5Zr; (e), (f) - Ti-25Nb-5Zr; (g), (h) - Ti-28Nb-5Zr; (i), (j) - Ti-30Nb-5Zr

4. Conclusions
1. After vacuum electric arc melting the alloys of the Ti-Nb-Zr system have a developed dendritic structure, including elongated dendrite grains in the middle of the ingots and multidirectional grains in the upper part.
   2. Homogenizing annealing at a temperature of 900 °C for 12 hours does not lead to the destruction of the dendritic structure and recrystallization. Annealing at a temperature of 1000 °C for 12 h completely destroys the dendritic structure in alloys Ti-20Nb-5Zr, Ti-23Zr-5Zr, Ti-25Nb-5Zr, Ti-28Nb-5Zr, Ti-30Nb-5Zr.

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