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Preparation of a ternary alloy of the Ti-Nb-Ta system for further use in medicine

Titanium alloys are widely used in orthopedics for several decades due to their excellent mechanical properties, excellent corrosion resistance and favorable biocompatibility [1,2]. However, the use of alloys, such as Ti-6Al-4V with a high Young's modulus (E), is not the best solution in load-bearing cells, as stress-shielding occurs due to a mismatch between the rigidity of the implant and the surrounding bone (about 115 GPa for Ti- 6Al-4V), which is significantly higher than the modulus of elasticity of the cortical bone (10-30 GPa) [1.10]. Moreover, these alloys have carcinogenic inclusions such as Al, V. To date, there is a need to obtain titanium-based alloys that do not contain carcinogenic, cytotoxic components and meet biomechanical requirements for the implant. In this work, a superelastic alloy of the Ti-Nb-Ta system is obtained by double remeltingin the vacuum arc furnace. The microstructure was investigated on optical microscope, mechanical properties were measured by universal testmachine Shimadzu AGX and hardness was investigated on microhardness tester Durascan 20. The authors concluded that the resulting alloy of the Ti-Nb-Ta system is suitable for use in medicine.

Keywords: medical titanium alloys, implants, method of obtaining the tantal alloy, biocompatible alloys, low module alloys.

1. Introduction

The most important biomaterials in orthopedics are metal alloys. Ti alloys due to high biocompatibility and mechanical properties are widely used materials for the manufacture of implants for reconstructive arthroplasty and osteoplasty in orthopedics, traumatology and neurosurgery [1-5].

The possibility of using the material in orthopedics and traumatology is determined by two main indicators, biocompatibility and biomechanical compatibility.

Biocompatibility according to [6] is the ability of the material to be implanted into the patient's body, not to cause adverse clinical processes and induce the cellular or tissue response necessary to achieve the optimal therapeutic effect. The most biocompatible metals are Ta, Nb, Ti and Zr.

According to studies [7], most of the materials currently used in the production of implants contain; V, Al, Co, Cu, Cr, Mo and Ni, which have weak and sometimes negative biocompatibility characteristics. So in works [1-7] it is shown that V has cytotoxic properties, and Al has negative effects on the central nervous system.

Biomechanical properties necessary for biomaterials used in orthopedics, in particular for the manufacture of implants, are well described in [8, 9]. Great importance in the development of new orthopedic biomaterials is the permissible deformation (δ) -the ratio of yield stress (σYS) to the elastic modulus E [1, 8, 9]. Admissible deformation of bone is from 0.43 % to 0.55 %. Thus, the best material for the manufacture of implants in orthopedics and traumatology is the material with the closest exponent of permissible deformation (δ) of the bone.

Titanium alloys are widely used in orthopedics for several decades due to their excellent mechanical properties, excellent corrosion resistance and favorable biocompatibility [1, 2]. Numerous studies have been devoted to the study of the properties of these materials for use in medicine. The excellent biocompatibility of titanium has been proved by many authors, both in vitro and in vivo [1, 3, 4]. Commercially pure titanium is used in some orthopedic and dental applications. Nevertheless, the limited strength of titanium (up to 500 MPa) prohibits its use as a material for an orthopedic endoprosthesis, which is an implant form factor [1, 2].

The use of titanium and conventional titanium alloys, such as Ti-6Al-4V with a high Young's modulus (E), is not the best solution in load-bearing cells, as stress-shielding occurs due to a discrepancy between implant stiffness and the surrounding bone (about 115 GPa for Ti-6Al-4V alloy), which is significantly higher than the modulus of elasticity of the cortical bone (10-30 GPa) [1, 10], and as a consequence,

osteoporosis occurs leading to fractures of the surrounding bone or weakening of the implant attachment. For any of these reasons, the lifetime of an o

In the works, the problems of reducing the toughness of the implant due to the increase in porosity were investigated, however, this simultaneously leads to a sharp reduction in the mechanical strength of the implant itself [11-15].

From the above considerations, there is a need to create new titanium-based alloys that do not contain carcinogenic, cytotoxic components and meet the biomechanical requirements for the implant.rthopedic implant of the Ti-6Al-4V alloy is usually limited to 10-15 years [1, 16, 17].

Such alloys are three and four component alloys based on titanium with the addition of Nb, Ta and Zr.

2. Materials and methods of the experiment

As an initial material for the ingot production of Ti73Nb21Ta6 alloy (titanium-niobium-tantalum)TiNb47 alloy and titanium alloy Grade 1 , were chosen. The melting of the starting material was carried out by two remelting in the furnace of an arc vacuum furnace (FAVF). For this purpose, the consumable electrode for the first melting was prepared as follows: titanium bars (Grade1, Ø 50 mm) were welded to the central rod (TiNb47, Ø 120 mm, batch 200-09 / 1 and 200-09 / 2) , in the amount of 10 pieces per electrode, 340 +/- 5 mm in length); Titanium wire was welded with tantalum wire (Ø4 mm), or cut out from the tantalum plate fragments of the following sizes 5-10x5-10x150-300 mm (Fig. 1). To perform the melting process, the TiNb47NT-47 skull was taken. The microstructure of the resulting alloy was investigated using an Altami optical microscope. The hardness test was carried out using the Brinell method, according to ISO 6506-1: 2005 at a load of 49.03 H.

Experimental studies were carried out in the laboratories of the East Kazakhstan State Technical University. D. Serikbaev and the central factory laboratory of JSC «UMP».

Figure 1. A photograph of the consumable electrode for FGWP

3. Results and discussion

The mass of welded tantalum was calculated on the basis of the content of titanium in the ingot TiNb47to obtain the required chemical composition of the main elements (titanium, niobium, tantalum). (Table 1).

Table 1

Requirements for the chemical composition of ingots Ti73Nb21Ta6

Alloy	Mass fraction of main components, %			Mass fraction of impurities, %, not more than									
	Ta	Nb	Ti	Al	Cr	Cu	Fe	Ni	Si	C	H	N	O
Ti73Nb21Ta6	6 +3/-2	21±2	Rest	0,01	0,01	0,01	0,02	0,01	0,01	0,02	0,0045	0,015	0,1

The material was loaded in the form of titanium bars, pieces of compact tantalum metal and niobium. The amount of material was calculated taking into account the obtaining of the required alloy by the basic elements (titanium, niobium, tantalum). As a loadable metal (tantalum and niobium), a material of 5 to 10 mm thickness was used on the first melting, but as practice showed, tantalum and niobium in the loading did not have time to completely dissolve during the time of complete fusion of the electrode. In order to obtain the second ingot of the first remelting, a tantalum and niobium band was used as a loading, which largely managed to dissolve (Fig. 2).

Figure 2. Photo of a skull after draining with annon-dissolved load

The discharge of the resulting melt mass (when melting the first remelting) was produced in an uncooled mold. A chemical analysis of the ingot was carried out, which showed an increased content of niobium (from the required). To reduce the content of niobium, a second remelting was carried out, corrected for the main elements with a discharge into an uncooled mold. After the chemical analysis of the second remelting ingot, which fully met the requirements (Table 2).

On the second ingot (batch 261-16) it was decided to get it by three remelting (to reproduce the process), but taking into account the loaded metal and the melting parameters.

The melting of the first remelting was carried out with the following parameters: a reference voltage of 38 V, ignition at 10 kA and a subsequent stepwise rise to 18 kA, a solenoid current when the bath was 4 A, followed by a decrease to 1.5 A. The first melting passed (the electrode melted) 4.5 minutes. After opening the furnace, it was noted that the loaded refractory material (niobium, tantalum) did not completely dissolve in the melt.

On the received ingot, the macrostructure analysis was carried out, the hardness in the longitudinal and cross sections was measured (Fig. 3-7). In the preparation of macrosections and microsections, the optimal etching solution (2HF: 1HNO3: 17H2O2) was selected by rubbing with a cotton swab. the solutions indicated in the literature sources did not give the proper effect.

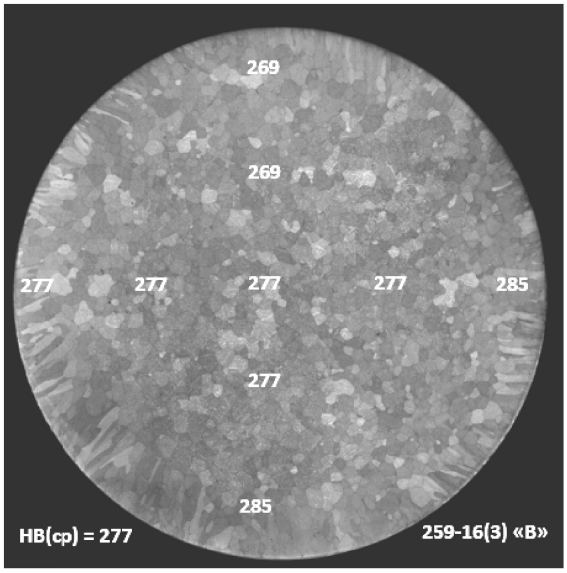


Figure 3. Macro structure and hardness of the ingot in cross section
(the temp from the top of the ingot)

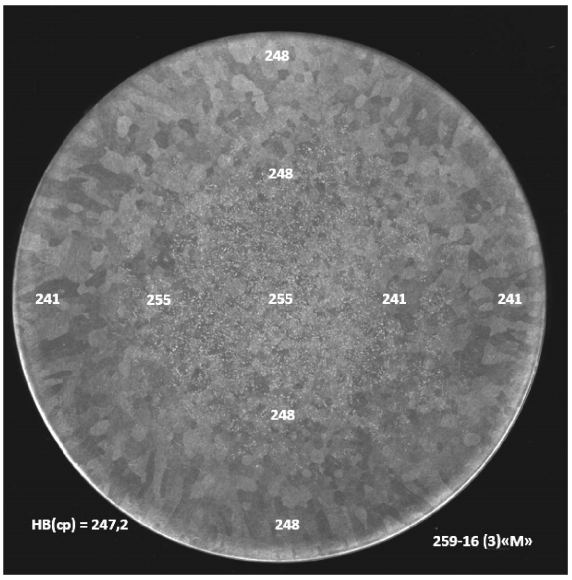


Figure 4.The macrostructure and hardness of the ingot in the cross-section
(the tempo from the middle part of the ingot)

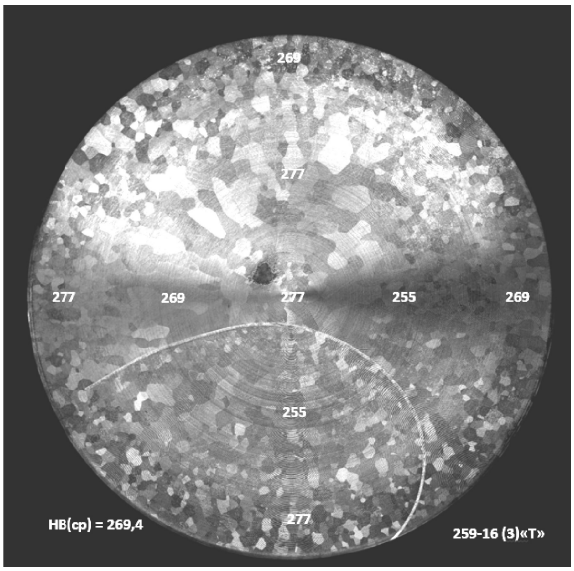
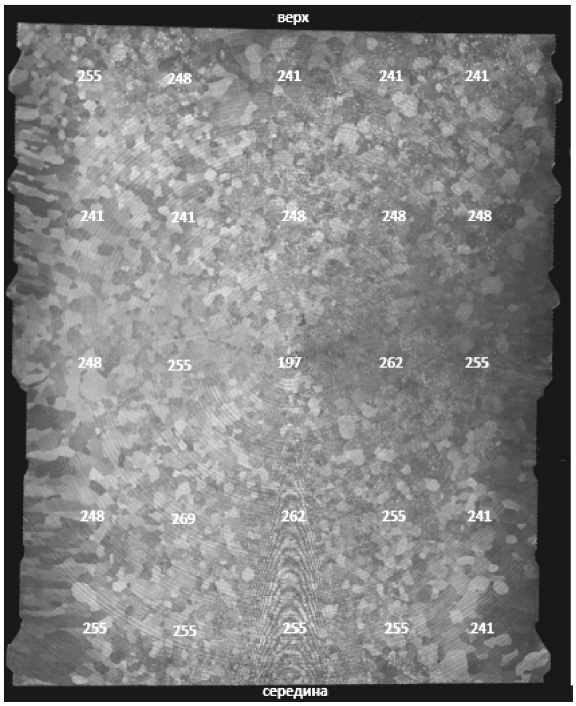
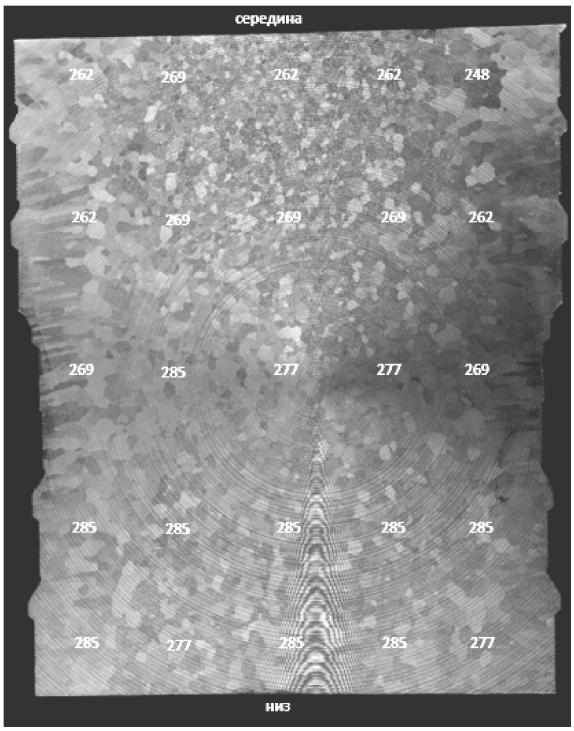


Figure 5. Macro structure and hardness of the ingot in cross section
(templet from the bottom of the ingot)



HB (min) = 197, HB (max) = 269, HB (cp) = 248,2

Figure 6. Macro structure and hardness of the ingot in the longitudinal section
(templet: top — middle of the ingot)



HB (min) = 248, HB (max) = 285, HB (cp) = 272.2

Figure 7. Macrostructure and hardness of the ingot in the longitudinal section
(templet: middle — bottom of the ingot)

From each template (cross section of the ingot), after the macrostructure was studied by hydro abrasive cutting, brushes measuring 15x15x (80-90) mm - 3 samples from each temperture were cut out. The results of measuring mechanical properties are given in Table 2.

Table 2

Mechanical properties

	From the top of the ingot		From the middle of the ingot		From the bottom of the ingot	
	Without annealing	Annealing at 700 °C for 1 hour	Without annealing	Annealing at 700 °C for 1 hour	Without annealing	Annealing at 700 °C for 1 hour
δ, %	3,8	2,1	5,21	2,8	2,97	5,1
σv, kg / mm2 (MPa)	72 (710)	77 (760)	73 (720)	79 (775)	75 (730)	77 (760)
σ0.2, kg / mm2 (MPa)	68 (660)	71 (690)	69 (670)	74 (720)	71 (700)	71 (690)

There are slight differences in mechanical properties along the length of the ingot. This feature can be explained by the fact that in the course of crystallization the process of heat removal proceeds most intensively in the middle part of the ingot, which is in contact with a copper water-cooled crystallizer. Heat sink from the top and bottom (the bottom of the ingot does not cool) part of the ingot passes less intensively, mainly due to radiation (the upper part of the ingot). A low cooling rate creates favorable conditions for the growth of dendrites, so a much more developed columnar structure can be observed in the upper part of the ingot.

The rolling of one of the longitudinal section templates was tested. The template was rolled from a thickness of 12.5 mm to a thickness of 11 mm (deformation rate of 12 %).

The sediment of the alloy sample was tested on a PO-54B press. The sample was deformed by 10 mm (degree of deformation 10 %).

4. Conclusion

The alloy in the cold cast state is not subject to cold pressure treatment (rolling, draft) - the blanks are destroyed. It is planned to carry out further work to determine the possibility of hot processing of the alloy by pressure (pressing heated billets to produce flat baits or round bars).

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Медицинада одан әрі пайдалану үшін Ti-Nb-Ta жүйесіне негізделген үш компонентті қорытпаны дайындау

Титан қорытпалары ортопедияда кеңінен қолданылады, себебі олардың механикалық қасиеттері, коррозияға төзімділігі және қолайлы биоүйлесімдік қасиеттері айырықша жоғары. Алайда Юнг-модулі (E) жоғары Ti-6Al-4V секілді қорытпаларды ортопедиялық жүктемелі элементтерде пайдалану, стресс экрандалу салдарынан үздік шешім болып табылмайды, қоршаған сүйек пен қорытпа арасындағы серпімділік сәйкессіздігі (Ti — үшін шамамен 115 ГПа, сүйектің беткі қыртысы — 10-30 ГПа) себебінен. Сонымен қатар осы қорытпалар Al, V сияқты канцерогенді кірмелері бар. Бүгінгі күні биомеханикалық талаптарына сәйкес канцерогенді және цитотоксикалық компоненттер құрамында емес, титан негізіндегі қорытпаларды алу қажеттілігі туындайды. Бұл мақалада Ti-Nb-Ta жүйесіндегі жоғарысерпімді қорытпа вакуумды доғалық пеште екі мәрте балқыту әдісімен алынды. Микроқұрылым оптикалық микроскопта, механикалық қасиеттер әмбебап Shimadzu AGX сынау қондырғысында өлшенді және қаттылық Durascan 20 қондырғысында зерттелді. Алынған нәтижелер Ti-Nb-Ta қорытпасы медицинада пайдалануға жарамды деп қорытынды жасалды.

Кілт сөздер: медициналық титан негізді қорытпалар, имплантаттар, танталды қорытпаларын өндіру тәсілі, биоүйлесімді материалдар, серпімділік модулі төмен қорытпалар.

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Получение трехкомпонентного сплава системы Ti-Nb-Ta для дальнейшего использования в медицине

Титановые сплавы широко применяются в ортопедии несколько десятилетий из-за их превосходных механических свойств, отличающихся коррозионной стойкостью и благоприятной биосовместимостью [1,2]. Однако применение сплавов, таких как Ti-6Al-4V с высоким модулем Юнга (E) не является лучшим решением в элементах, несущих нагрузку, так как возникает стресс-экранирование, вызванное несоответствием жесткости имплантата с окружающей костью (около 115 ГПа для сплава Ti-6Al-4V), что значительно выше, чем модуль упругости кортикальной кости (10-30 ГПа) [1,10]. Более того, данные сплавы обладают канцерогенными включениями, такими как Al, V. На сегодняшний день существует необходимость получения сплавов на основе титана, не содержащих канцерогенных, цитотоксичных компонентов и отвечающих биомеханическим требованиям к имплантату. В этой работе показано, что сверхупругий сплав системы Ti-Nb-Ta получен путем двойного переплава в вакуумной дуговой печи. Микроструктура была исследована на оптическом микроскопе, механические свойства были измерены на универсальной испытательной машине Shimadzu AGX, а твердость исследована на Durascan 20. По результатам исследования сделано заключение, что полученный сплав системы Ti-Nb-Ta годен для использования в медицине.

Ключевые слова: медицинские титановые сплавы, имплантаты, метод получения титанового сплава, биосовместимые сплавы, сплавы с низким модулем упругости.

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