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Investigation of the Influence of Modes of Intensive Plastic Deformation on the Process of Grain Refinement of Titanium Alloy Ti-13Nb-13Zr at Equal-Channel Angular Pressing and Subsequent Rotary Forging Compression

Ultrafine-grained materials are currently of great interest due to their excellent mechanical and functional properties. One of the most effective methods to obtain such materials with a unique combination of microstructure and properties is intense plastic deformation (IPD). This paper deals with the development of an efficient IPD method for Ti-13Nb-13Zr titanium alloy based on a combined approach involving equal channel angular pressing (ECAP) and subsequent rotational forging compression (“RFC”). Ti-13Nb-13Zr titanium alloy was pressed at different temperatures using an equal channel angular pressing (ECAP) process through a channel angle of 130° for several passes, followed by rotational forging compression (“RFC”). Microstructural analysis showed that the application of combined processing (RCUP + RFC) transformed the coarse-grained (CG) structure into an ultrafine-grained structure (UFGS). In addition, the results of mechanical tests indicate that the application of combined processing method significantly increases the hardness and modulus of elasticity of titanium alloy Ti-13Nb-13Zr. These changes in the complex of properties allow us to consider this alloy as a highly effective alternative to traditional metallic materials used in biomedical implantology.

Keywords: Intense plastic deformation, ultrafine-grained structure, equal-channel angular pressing, rotary forging compression, titanium alloys.

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Introduction

In recent decades, there has been a steady increase in interest in the use of titanium alloys in medicine, particularly in orthopedics, traumatology, dentistry and cardiac surgery. This is due to the unique properties of titanium and its alloys, such as high corrosion resistance, excellent biocompatibility, low specific weight, and a favorable combination of strength and elastic properties [1]. However, with the ever-increasing requirements for the durability and reliability of medical devices, especially implants, the need to improve the mechanical properties of materials without compromising their biocompatibility becomes obvious. Therefore, it is highly desirable to develop new materials for implants made of titanium and titanium alloys with higher tensile strength and elasticity modulus equivalent to the bone elasticity modulus.

One of the most urgent and promising directions in the field of improving the performance characteristics of titanium alloys is the formation of ultrafine grain structure (“UFGS”). Reduction of the average grain size to submicron level (less than 1 micron) provides a significant increase in strength, hardness and fatigue resistance due to intensification of the grain boundary hardening mechanism [2]. The strength of metallic materials increases with decreasing grain size, which is well known as the Hall-Petch relationship [3]. Grain refinement can induce hardening without the addition of any alloying elements and can potentially achieve the desired strength. Intense plastic deformation (IPD) is known as a new method to produce UMP structures and a large number of studies have been conducted on IPD and UMP structures. Equi-channel angular pressing, multilayer torsion, rotary forging and others are commonly used to fabricate IPD [4].

Nevertheless, most of the existing IPD methods require further optimization in terms of manufacturability, reproducibility and scalability for practical application in the medical industry. In addition, it is important to take into account the influence of deformation parameters on the phase composition, texture and, ultimately, on the biomechanical properties of the finished products. Thus, the actual scientific task is the development of such a method of IPD, which will make it possible to obtain titanium alloys with UMP structure, possessing high strength, stability and suitability for the manufacture of medical implants.

Equal channel angular pressing (ECAP) is one of the most effective methods of severe plastic deformation, which is used to obtain ultrafine-grained and nanostructured materials. This process involves repeatedly pushing a sample through a system of channels with equal cross-sections connected at a certain angle. The main advantage of ECAP is the ability to achieve significant plastic deformation without changing the shape of the specimen, which makes this method attractive for improving the mechanical properties of metals and alloys such as strength, ductility and hardness. And also equal channel angular pressing is one of the most effective methods of intense plastic deformation (IPD), designed to produce ultrafine grained or nanostructured structure in metals and alloys without changing the external shape of the specimen. One of the key factors affecting the efficiency of the ECAP process is the geometry of the matrix channels, including the joint angle and corner rounding radius. Optimization of these parameters allows minimizing deformation inhomogeneities, reducing friction and lowering pressing forces, which is especially important for ensuring microstructure uniformity and improving the quality of the processed material.

Currently, scientists have proposed various variants of mechanical and thermomechanical treatments of Ti-13Nb-13Zr alloy [5]. For example, Majumdar et al. [6] have tried several combinations of hot working, ST and cooling conditions to optimize the mechanical performance. Park et al. [7] introduced warm cross rolling to obtain ultrafine grain structure in Ti-13Nb-13Zr alloy. Li et al. [8–12] first proposed to improve the mechanical properties of this alloy by multi-pass gauge rolling. Recently, he was able to improve this process and obtained the lowest Young's modulus ever reported for Ti-13Nb-13Zr [13]. In [14], the effect of thermomechanical treatment by equal-channel angular flattening on the structures and phase composition of Ti-13Nb-13Zr (TNZ) was investigated. In [15] the peculiarities of microstructure evolution and properties of Ti-13Nb-13Zr alloy under combined processing including ECAP and subsequent rotational forging compression (RFC) were investigated. Due to RCUP + RFC, the tensile strength increased to 1167.7 MPa and the elongation was 8.6 %. The excellent mechanical properties were mainly due to hardening by α-phase precipitation, dislocation hardening and grain refinement.

In this regard, the aim of this work is to develop an efficient IPD method for Ti-13Nb-13Zr titanium alloy based on a combined approach involving equal-channel angular pressing (ECAP) and subsequent rotational forging compression (RFC).

Materials and methods of experiments

Ti-13Nb-13Zr titanium alloy was chosen as the material for the study. The chemical composition of the main alloying elements is: 13.0 wt.% Nb, 13 wt.% Zr, 0.086 wt.% O, 0.009 wt.% N, 0.0012 wt.% H, the rest — Ti. The choice of this alloy is due to its wide application in the production of medical implants due to its high biocompatibility, corrosion resistance and low modulus of elasticity.

The study of structure and properties of samples after different treatment modes was carried out using optical and electron microscopy. Surface microstructure and cross-sectional morphology of the coatings were studied by scanning electron microscopy (SEM) on Vega 4 (Tescan, Czech Republic). Hardness and modulus of elasticity of the samples were measured by the Martens method according to ASTM E 2546 on a hardness tester FISCHERSCOPE HM2000S ("Fischerscope", Germany), at indenter load F = 245.2mN and dwell time of 20s. Surface roughness was determined according to GOST 25142-82 using a profilometer model 130 [16].

A combined approach involving equal-channel angular pressing (ECAP) followed by rotational forging compression ("RFC") was used to obtain an ultrafine grained (UFG) structure.

Figure 1 shows the complete simulation of the equal-channel angular pressing process, including the punch path (*a*), channel geometry (*b*, *c*), finite element mesh (*d*), Mises stress distribution (*e*), and force versus displacement plot (*f*). The punch motion exhibits linear descent and return, which sets the initial conditions for deformation. The channel geometry and grid structure provide high detail of the calculation in the stress concentration zones. The stress distribution shows that the maximum stresses are concentrated in the corners of the channel, confirming the need for shape optimization to reduce peak values ($\sim 14\times10^{8}$ N/m^2). The force-displacement plot illustrates the stability of the process with a peak force of $\sim 9\times10^{6}$ N, which is associated with overcoming the material resistance. This model demonstrates a comprehensive approach to the analysis of ECAP, which allows us to evaluate the influence of process parameters and suggest optimal conditions for uniform formation of ultrafine-grained structure.

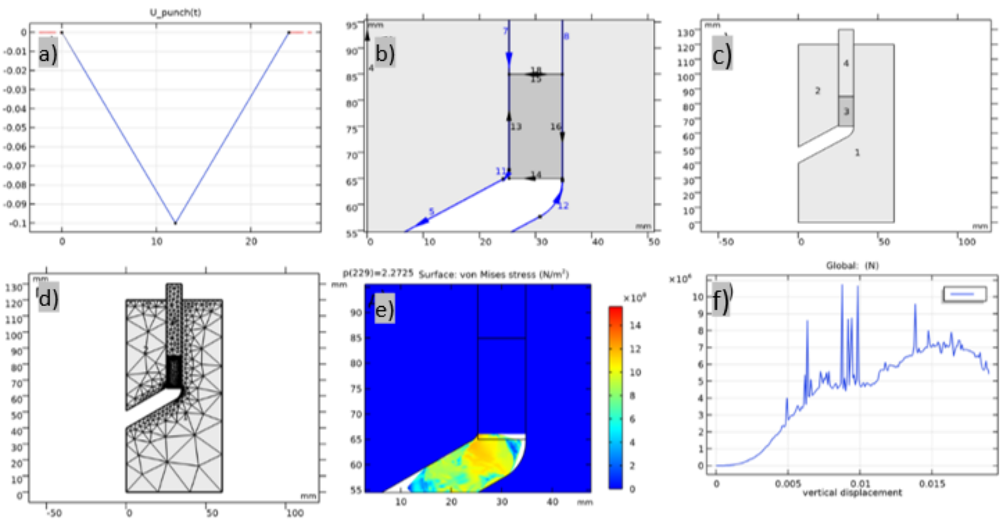


Figure 1. Modeling of ECAP process: (*a*) punch trajectory, (*b, c*) channel geometry, (*d*) finite element mesh, (*e*) Mises stress distribution, (*f*) force dependence on vertical displacement

The computational mesh used is characterized by high density in critical areas, which ensures the accuracy of the analysis and allows for a detailed study of the plastic behavior of the material. Thus, this model serves as an important tool for the study and optimization of the ECAP process, allowing the consideration of various geometrical and technological parameters. This, in turn, contributes to the improvement of material characteristics, reduction of production costs and expansion of the application of the ECAP method in industry.

Based on the theoretical calculations carried out using the finite element method, the design of the matrix for ECAP was developed and optimized. The main design criterion was to ensure uniform distribution of plastic deformation of the material while minimizing stress concentrations and pressing forces. Calculations showed that the optimal angle of channel connection is 130 degrees, which provides effective strain redistribution without significant increase in force.

High-strength tool steel of 9ХГСА grade, which has high wear resistance and deformation resistance, was used for manufacturing the matrix. The design includes an internal channel with an angle of 130 degrees, which allows minimizing the friction of the material against the walls and preventing local fractures during the passage of the sample. The actual design was fabricated with the calculated loads and contact conditions [17, 18].

This matrix (Fig. 2) is designed for experiments to improve the mechanical properties of the material by intense plastic deformation.

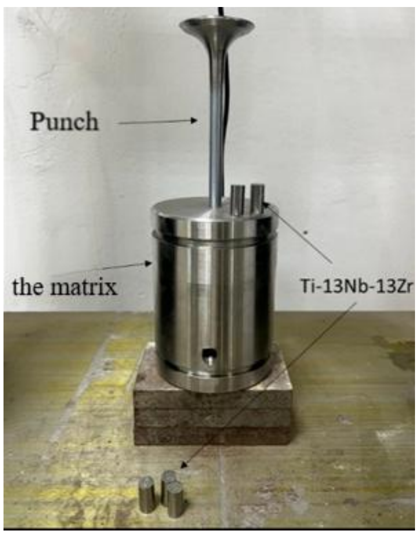
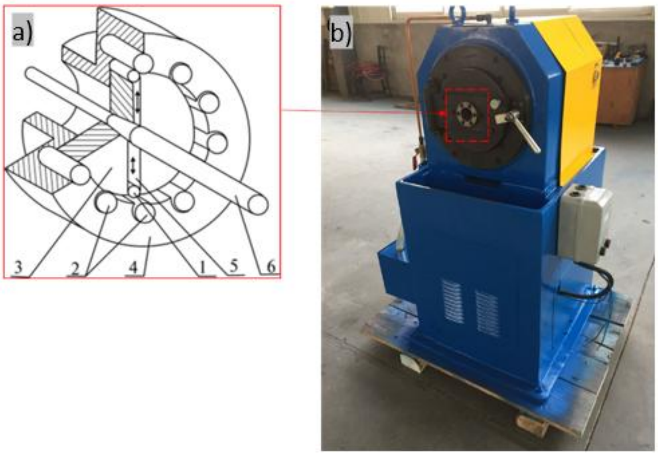


Figure 2. Matrix for ECAP

The samples were prepared in the form of cylinders with a diameter of 10 mm and a height of 20 mm, which meets the requirements of a matrix fabricated with a channel angle of 130 degrees.

After equal channel angle pressing (ECAP), the material is subjected to an intermediate heat treatment to relieve residual stresses and stabilize the structure. This step is critical to prepare the material for the next processing step, rotational forging compression (RFC) (Fig. 3).



1, 2 — rollers; *3* — spindle; *4* — cage; *5* — strikers; *6* — workpiece

Figure 3. Rotary forging scheme (*a*) and rotary forging machine (*b*)

RFS complements ECAP by providing additional grain refinement through a complex combination of rotational and axial deformations, which contributes to microstructure equalization and increased uniformity of mechanical properties. Figure 4 shows a scheme of stages of combined ECAP and RFS processing with intermediate heat treatment, where each stage plays a key role in achieving ultrafine grain structure. The first stage of ECAP provides an intense plastic deformation initiating grain refinement and substructure formation. The material is then subjected to intermediate heat treatment, which relieves residual stresses, activates recrystallization processes and prepares the material for the next stage. The final stage of RFS (rotary forging compression) completes the process by creating complex deformation modes to eliminate structural inhomogeneities and additional grain refinement.

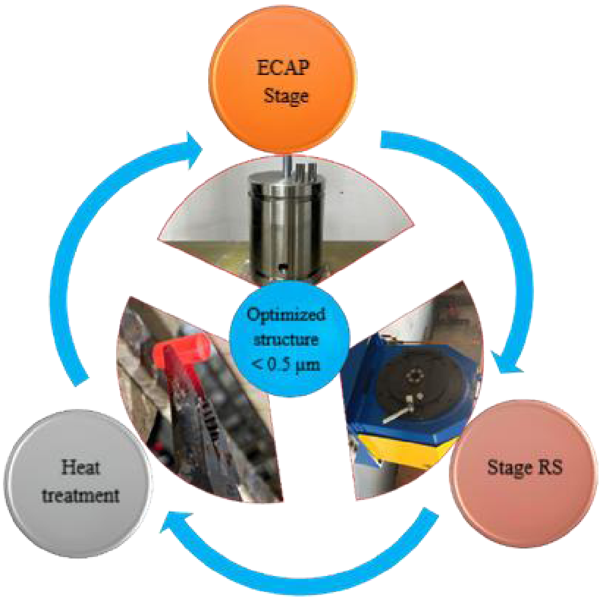


Figure 4. Schematic diagram of stages of combined processing of ECAP and RFS with intermediate heat treatment

Table summarizes the combined severe plastic deformation (SPD) regimes applied to titanium, involving equal-channel angular pressing (ECAP) and rotary forging compression (RFC), with varying numbers of cycles and processing temperatures. All processing was conducted in air.

T a b l e

Modes of combined severe plastic deformation

Mode	Processing method	Processing temperature (°C)	Number of cycles	Processing method	Processing temperature (°C)	Number of cycles
Ti_1	ECAP	700	1	–	–	–
Ti_2	ECAP	700	1	RFC	800	2
Ti_3	ECAP	700	2	RFC	800	3
Ti_4	ECAP	Room temperature (25)	1	–	–	–

Results and Discussions

Room temperature the study of titanium microstructure after intense plastic deformation (IPD) with different number of cycles and application of heat treatment showed a significant influence of processing modes on the material structure (Fig. 5). In the initial state (*a*) titanium had a coarse-grained structure without defects. After 2 cycles with heat treatment (*b*), grain refinement and the beginning of recrystallization were observed, but the structure remained heterogeneous. Three cycles with heat treatment (*c*) promoted the formation of a fine-grained and homogeneous structure, practically devoid of defects. The maximum uniform ultrafine-grained structure was achieved after 5 cycles with heat treatment (*d*), which provided material stability and improved mechanical properties. In the case of 1 cycle without heat treatment (*e*), significant defects such as microcracks and inhomogeneous structure were found, which limits the strength properties. Thus, heat treatment after IPD plays a key role in improving the structure and properties of titanium.

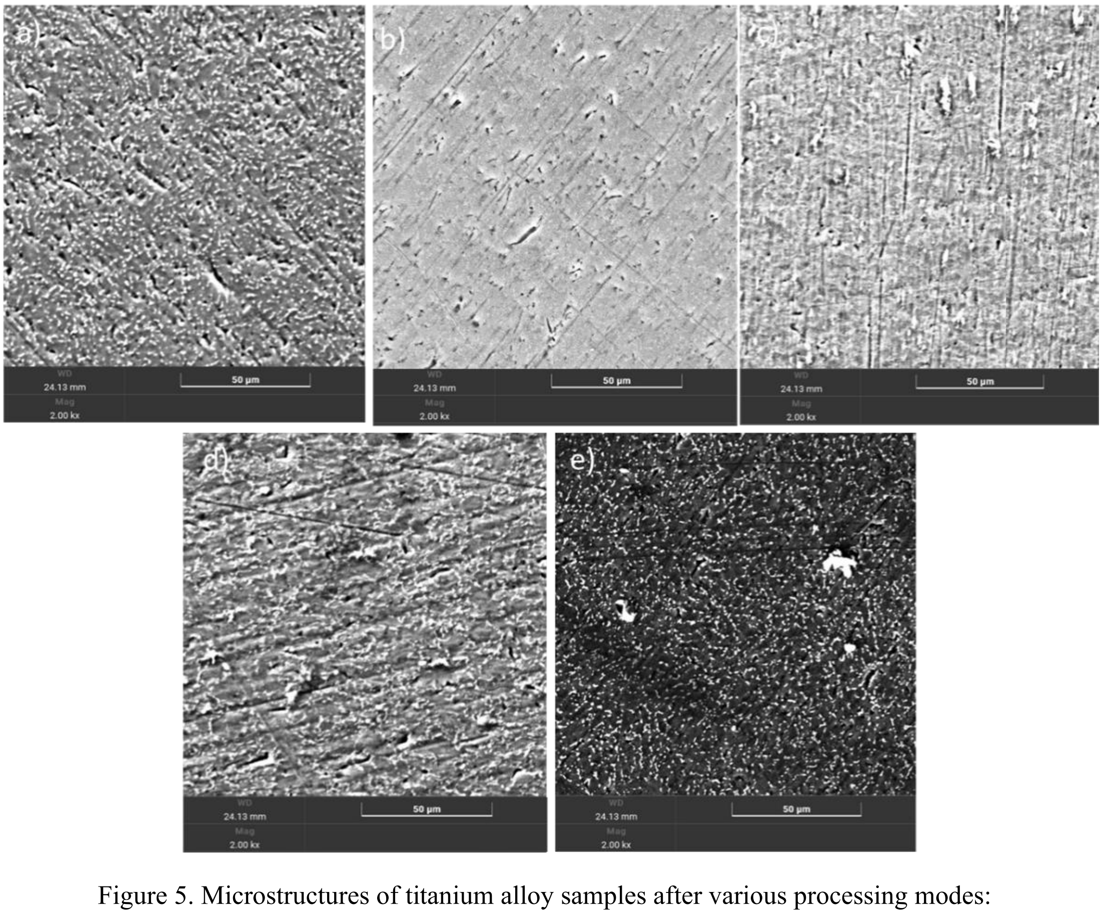


Figure 5. Microstructures of titanium alloy samples after various processing modes:
Ti_orig. (*a*); Ti_1 (*b*); Ti_2 (*c*); Ti_3 (*d*); Ti_4 (*e*)

Figure 6 shows the dependence of hardness (H) and modulus of elasticity (E) of titanium alloy samples on processing modes. The source material has low hardness and modulus of elasticity. After ECAP processing in Ti_1 mode, there is a slight increase in these parameters. A significant increase in hardness and elastic modulus is observed in the Ti_2 and Ti_3 modes, where combined ECAP + RFC treatment was applied. The maximum values are reached in the Ti_3 mode, which is associated with an increase in the number of cycles and more intensive grinding of grains. However, in the Ti_4 mode, when using ECAP at room temperature, the hardness and modulus of elasticity are significantly reduced, due to insufficient deformation of the material and a low degree of grain grinding.

Thus, combined ECAP + RFC treatment with an optimal number of cycles demonstrates the greatest efficiency in improving the mechanical characteristics of a titanium alloy.

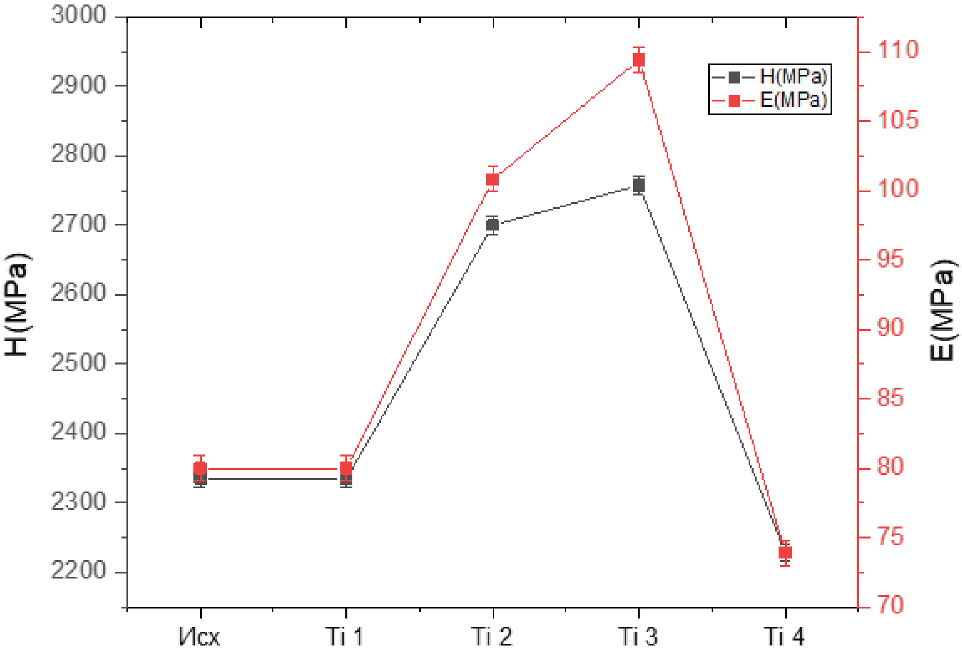


Figure 6. Hardness (H) and modulus of elasticity (E) of the samples

Conclusion

In the course of the study, an effective technology for intensive plastic deformation of Ti-13Nb-13Zr titanium alloy for medical purposes using equal-channel angular compression (ECAP) and rotational forging compression (RFC) was developed and experimentally substantiated.

The results of numerical finite element modeling have confirmed the importance of optimizing matrix geometry for ECAP. It has been shown that the channel connection angle of 130° ensures uniform distribution of plastic deformation and minimizes peak stresses in the material, which helps to prevent the destruction of samples during pressing.

The combined treatment, including the sequential use of ECAP and RFC with intermediate heat treatment, made it possible to achieve the formation of an ultra-fine-grained structure of the material and a significant increase in its mechanical characteristics. Experimental studies of the structure and properties of the titanium alloy have shown that the best results are achieved after five cycles of combined ECAP + RFC treatment at a temperature of 700–800 °C. At the same time, the formation of a uniform ultrafine-grained structure with a minimum number of defects was observed, as well as a significant increase in the hardness and modulus of elasticity of the material compared with the initial state.

The developed technology of plastic deformation intensification opens up new prospects for the creation of a new generation of medical implants with improved performance, increased reliability and durability. The results obtained can be used to scale the process into industrial production and for further research aimed at optimizing deformation and heat treatment modes depending on the requirements for specific medical devices.

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Қарқынды пластикалық деформация режимдерінің Ti-13Nb-13Zr титан қорытпасының дәнін тең арналы бұрыштық престеу және одан кейінгі айналмалы-соғу сығымдау кезінде ұнтақтау процесіне әсерін зерттеу

Ультраұсақтүйіршікті (УҰТ) материалдар қазіргі кезде өздерінің жоғары механикалық және функционалдық қасиеттеріне байланысты үлкен қызығушылық тудыруда. Мұндай материалдарды ерекше микроқұрылымы мен қасиеттер үйлесімінде алудың ең тиімді әдістерінің бірі — интенсивті пластикалық деформация (ИПД). Осы жұмыста Ti-13Nb-13Zr титан қоспасына арналған тиімді ИПД әдісін әзірлеу қарастырылған. Бұл әдіс теңарналы бұрыштық престеу (ТБП) және одан кейінгі айналмалы-қысу соққысын (АҚС) қамтитын біріктірілген тәсілді қолдануға негізделген. Ti-13Nb-13Zr титан қоспасына бірнеше өту арқылы 130° бұрышты арнадан өтетін теңарналы бұрыштық престеу (ТБП) процесі әртүрлі температураларда жүргізілді, одан кейін айналмалы-қысу соққысы (АҚС) қолданылды. Микроқұрылымдық талдау нәтижелері көрсеткендей, ТБП мен АҚС-ты біріктіріп өңдеу арқылы бастапқы ірі түйіршікті (ІТ) құрылым ультраұсақтүйіршікке айналады. Сонымен қатар, механикалық сынақ нәтижелері аталған біріктірілген өңдеу әдісінің Ti-13Nb-13Zr титан қоспасының қаттылығы мен серпімділік модулін едәуір арттыратынын көрсетеді. Қалыптасқан қасиеттер кешеніндегі бұл өзгерістер бұл қоспаны биомедициналық имплантологияда қолданылатын дәстүрлі металл материалдарына жоғары тиімді балама ретінде қарастыруға мүмкіндік береді.

Кілт сөздер: интенсивті пластикалық деформация, ультраұсақтүйіршікті құрылым, теңарналы бұрыштық престеу, айналмалы-қысу соққысы, титан қортпалары

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Исследование влияния режимов интенсивной пластической деформации на процесс измельчения зерна титанового сплава Ti-13Nb-13Zr при равноканальном угловом прессовании и последующем вращательно-ковочном сжатии

Ультрамелкозернистые материалы в настоящее время представляют большой интерес благодаря своим превосходным механическим и функциональным свойствам. Одним из наиболее эффективных методов получения таких материалов с уникальным сочетанием микроструктуры и свойств является интенсивная пластическая деформация (ИПД). В настоящей работе рассматривается разработка эффективного метода ИПД для титанового сплава Ti-13Nb-13Zr, основанного на применении комбинированного подхода, включающего равноканальное угловое прессование (РКУП) и последующее вращательно-ковочное сжатие (ВКС). Титановый сплав Ti-13Nb-13Zr подвергался прессованию при различных температурах с использованием процесса равноканального углового прессования (РКУП) через угол канала 130° в течение нескольких проходов, с последующим вращательно-ковочным сжатием (ВКС). Микроструктурный анализ показал, что при применении комбинированной обработки (РКУП + ВКС) крупнозернистая (КЗ) структура трансформируется в ультрамелкозернистую (УМЗ) структуру. Кроме того, результаты механических испытаний свидетельствуют о том, что применение комбинированного метода обработки существенно повышает показатели твёрдости и модуля упругости титанового сплава Ti-13Nb-13Zr. Указанные изменения в комплексе свойств позволяют рассматривать данный сплав в качестве высокоэффективной альтернативы традиционным металлическим материалам, используемым в биомедицинской имплантологии.

Ключевые слова: интенсивная пластическая деформация, ультрамелкозернистая структура, равноканальное угловое прессование, вращательное ковочное сжатие, титановые сплавы

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