Effect of severe plastic deformation on the physical and mechanical properties of the ultrafine-grained β-alloy Ti-15Mo

V V Polyakova, S A Gatina, A A Polyakov and I P Semenova
Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, 12 K. Marx street, Ufa, 450008, Russian Federation

E-mail: vnurik@gmail.com

Abstract. The influence of severe plastic deformation on physical and mechanical properties of the pseudo-β-alloy Ti-15Mo alloy has been investigated.

1. Introduction
The combination of a low elastic modulus, good corrosion resistance and unique biocompatibility determines the wide use of titanium and its alloys in medicine as functional materials for designing implants of a wide range of applications [1]. Lately, much attention has been paid in the world to the development of pseudo-β-titanium alloys, which have the ability to change their elastic modulus depending on the phase composition [2]. Among the commercial Ti alloys certified in medicine, the Ti-15Mo alloy (ASTM F2066) is intended for applications in orthopedics, traumatology, and cardiovascular surgery. The Ti-15Mo alloy has the lowest elastic modulus (about 80 GPa) in the single-phase β-state. However, it is characterized by unsatisfactory strength (UTS≤ 800 MPa, σ_f≤400 MPa) (ASTM F 2066). Conventional methods of improving the strength of pseudo-β-titanium alloys by thermal or thermomechanical treatment, including rolling or drawing in combination with aging, normally lead to a reduction of ductility, an inevitable increase in the elastic modulus due to the precipitation of the second phases (α and β), as well as the formation of a sharp texture and consequently, strong anisotropy of properties [3]. One of the effective methods for increasing fatigue strength while preserving ductility in metals and alloys in the formation of an ultrafine-grained (UFG) structure in them by severe plastic deformation (SPD) that enables very large strains to be achieved at relatively low temperatures under high applied pressures [4]. Therefore, increasing the fatigue strength of the pseudo-β alloy Ti-15Mo while preserving its relatively low elastic modulus is a relevant problem in the area of design of biomedical materials and permanent joint replacements.

In this work, the possibility of increasing the strength and fatigue properties of the Ti-15Mo alloy without a significant increase in the elastic modulus due to the formation of the UFG structure in it using equal-channel angular pressing (ECAP) is demonstrated.

2. Material and experimental procedure
The object of the study was rods with a diameter of 15 mm from the pseudo-β-titanium alloy Ti-15Mo in the two-phase (α+β)-state [5]. The UFG state was produced by thermomechanical treatment that included: β-solution quenching in water from a temperature of 810 °C (heating time 30 minutes) and...
subsequent ECAP processing (4 passes via route Bc with a channels intersection angle of 135°) at a temperature of 250 °C, with a total strain of \( \varepsilon = 1.91 \) [6]. A metallographic study was conducted using a JEOL JEM-2100 microscope (TEM) and a JEOL JSM 6390 microscope (SEM). The stress-strain curves were recorded using an initial strain rate of \( 1.0 \times 10^{-3} \) s\(^{-1} \) with an Instron universal testing machine. The tension-compression fatigue tests were conducted with an Instron 8801 machine at room temperature under a symmetric loading cycle \( R = -1 \) with a frequency of 30 Hz at 10\(^7\) cycles to failure, in compliance with the international standard for cyclic tests ASTM E466, on smooth samples of a round section having a gauge diameter of 3 mm. The elastic modulus of the alloy was determined by nanoindentation using a CETRN universal microtribometer with a load of 100 mN, with a loading-unloading time of 15 s. The nanoindentation procedure was in compliance with ISO 14577.

3. Results and their discussion

3.1. Microstructure

For medical products that require resistance to intensive loads, the Ti-15Mo alloy is used in its stronger (\( \alpha+\beta \)) state with an increased elastic modulus (figure 1, a). To form a single-phase \( \beta \)-structure, the initial material was exposed to a temperature of 810 °C for 30 minutes followed by water quenching. This temperature was selected on the basis of the results of X-ray phase analysis and differential scanning calorimetry [7].

![Figure 1. Coarse-grained Ti-15Mo alloy: a) initial (\( \alpha+\beta \))-structure, SEM; b) \( \beta \)-structure after quenching, SEM; c) microdiffraction pattern taken from a \( \beta \)-grain, TEM.](image)

After quenching, the alloy is characterized by a polycrystalline bimodal structure consisting of recrystallized \( \beta \)-grains with average sizes of 35±2 µm and 15±3 µm (figure 1, b). The volume fraction of grains larger than 35 µm in size is 55%. The microdiffraction pattern of the Ti-15Mo alloy (figure 1, c) taken from the axis \( <110> \) of the bcc lattice of the \( \beta \)-phase, in addition to the \( \beta \)-phase reflections, is also characterized by the presence of secondary reflections of the athermal \( \omega_{ath} \)-phase that correspond to the positions 1/3 and 2/3 of \{112\}\( \beta \). The diffusionless formation of the \( \omega_{ath} \)-phase is typical for pseudo-\( \beta \)-titanium alloys quenched from temperatures above the polymorphic transition temperature [8].

![Figure 2. ECAP-processed Ti-15Mo (250 °C, 4 passes, 135°): a) typical structural features. b) mesobands consisting of microbands, microdiffraction pattern; c) microtwins. TEM.](image)
By analyzing the microstructure of the ECAP-processed samples, several typical structural elements can be distinguished (figure 2, a). In the structure, closed (loop-like) configurations of dislocation boundaries (figure 2, a-3), as well as extensive mesobands (figure 2, a-1), representing a system of parallel dipoles with disrupted dislocation boundaries, are observed. Inside the mesobands, microbands are observed (figure 2, a-2). A weak contrast between them indicates small misorientation angles (figure 2, b). Also, packs of microtwins are observed in the structure, the width of which ranges from 10 to 50 nm (figure 2, a-4, c).

In general, after ECAP, the microstructure is characterized by ultrafine structural elements in the form of grain/subgrain fragments with a cross-sectional size of less than 1 µm, oriented along the axis rod of the rod. In addition, weakly-expressed reflections of the α-phase can be seen in the diffraction patterns (figure 2, b). Due to the low intensity of the reflections, an image of the α-phase could not be obtained.

3.2. Mechanical properties
The tensile curve in the initial (α+β)-state is characterized by the presence of a sharp yield point attributed to a sharp increase in the number of mobile dislocations of the β-grain at the onset of plastic flow, which is typical for metals with a bcc lattice [9]. Quenching led to an increase in the ductility of the alloy from 17±2 to 32±3 % and to a decrease in the ultimate tensile strength (UTS) from 1020±10 to 790±5 MPa due to the growth of the β-grain and the complete dissolution of the strengthening α-phase, i.e. the transition of the alloy from the two-phase state to the single-phase state (figure 3, a).

![Figure 3](image-url)

**Figure 3.** Physical and mechanical properties of the Ti-15Mo alloy: a) tensile curves; b) tension-compression cyclic tests; c) endurance limit vs elastic modulus of biomedical Ti alloys.* [11], ** [12].

The formation of an ultrafine structure in the Ti-15Mo alloy by ECAP resulted in a significant enhancement of the mechanical properties (figure 3, a). The UTS increased from 790 ± 5 to 1450 ± 20 MPa, and the elongation to failure decreased from 32 ± 3 to 10 ± 1 %. At the same time, worth noting is a visible reduction in uniform elongation on the tensile curve of the processed alloy, which indicates an early strain localization, characteristic of many metallic materials produced by SPD and associated with impeded nucleation of dislocations in the structure [4]. Figure 3, b shows the fatigue curves of the Ti-15Mo alloy in different structural states, obtained during tension-compression tests. Two main areas can be distinguished on the fatigue curves: low-cycle and high-cycle fatigue. In the initial (α+β)-state, the endurance limit of the Ti-15Mo alloy is 500 ± 10 MPa at 10^7 cycles to failure. The formation of a single-phase β-state in the alloy led to a decrease in the endurance limit to 410 ± 10 MPa, which is conditioned by the exclusion of the strengthening α-phase from the structure. The ultrafine structure formation by ECAP led to an increase in the endurance limit of the alloy, which increased by more than 50 % as compared to the quenched condition, and amounted to 640 ± 10 MPa at 10^7 cycles to failure (figure 3, b).

3.3. Stress-elasticity modulus ratio
Figure 3 shows the relationship between the endurance limit and elastic modulus for the Ti-15Mo alloy and other alloys widely used as biomaterials, in particular, Grade 4 Ti, Ti-6Al-4V ELI after a
conventional treatment and SPD [10]. In the initial α+β-state, the alloy has an elastic modulus of 105±7 MPa, influenced by the presence of a fraction of the α-phase in the structure, its elastic modulus for Ti-15Mo being 124 GPa [11]. After quenching, the value of E was 87±5 GPa. As it was shown in [9], the elastic modulus of the Ti-15Mo alloy is strongly influenced by the stability of the β-phase and the stability of the ω-phase particles that grow with increasing processing temperature. The athermal ω_{ath} phase, present in the alloy after quenching, is the pre-precipitate of the isothermal ω_{iso} phase that formed via the mechanism of atoms displacement in the lattice of the β-phase [9], and therefore its effect on the elastic modulus value of Ti-15Mo is not so large. A slight increase in the alloy’s elastic modulus to 93±5 GPa was observed after ECAP processing at a temperature of 250 °C. Apparently, the main reason could be a very small amount of the ω_{iso} phase after the processing. Besides, some contribution into the preservation of the low values of Young’s modulus is made by the formation in the structure of a high density of non-equilibrium grain boundaries with introduced dislocations, as it was demonstrated for the nanostructured TNTZ alloy [12].

4. Conclusions
1. This paper reveals the microstructural features of the Ti-15Mo alloy processed by SPD. It is shown that the processing of the alloy by equal-channel angular pressing in the β-state at a temperature of 250 °C leads to the formation of a complex ultrafine structure with a structural element size of less than 1 μm, providing a significant enhancement of the mechanical (UTS from 800 to 1450 MPa) and fatigue properties (σ_{f} from 420 to 640 MPa) of the alloy.
2. The elastic modulus of the Ti-15Mo alloy after ECAP at T=250 °C slightly increases from 87 to 93 MPa as compared to the single-phase β-state after quenching, which could be due to the increase in the density of the non-equilibrium boundaries of the β-phase grains/subgrains and the formation of a relatively small amount of the ω_{iso} phase.
3. Comparison of the study results for the ECAP-processed Ti-15Mo alloy and the similar parameters of Grade 4 Ti and Ti-6Al-4V ELI visibly demonstrates that Ti-15Mo with an ultrafine structure can occupy a worthy place among other biometallic materials, and its application is promising for the manufacture of products for osteosynthesis and orthopedics.

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