Mechanical properties and thermal stability of an ultrafine-grained near β titanium alloy

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Abstract. Comparative studies of the structure, mechanical properties and thermal stability of an ultrafine-grained near β titanium alloy of the transition class Ti-5Al-5Mo-5V-1Cr-1Fe obtained by the methods of multiple pressing and radial-shear rolling with subsequent aging have been carried out. It is shown that as a result of multiple pressing, the formation of a homogeneous micro-duplex ultrafine-grained structure with an average grain (phase) size of ~0.2 μm is observed. Radial shear rolling followed by aging leads to the formation of an inhomogeneous structure with precipitates of the α phase particles and a subgrain size of the β phase of about 0.5 μm inside which thin-acicular precipitates of the metastable α" phase are observed. Despite the smaller imposed plastic deformation and the larger size of grain-subgrain structure, alloy samples after radial-shear rolling and aging demonstrate higher strength properties at room temperature compared to the alloy obtained by multiple pressing (the ultimate strength of the alloy after these treatments is 1695 and 1600 MPa, respectively.) At the same time, the alloy obtained by the method of multiple pressing shows higher ductility and thermal stability of the mechanical properties due to the micro-duplex ultrafine-grained structure. The drop in strength as a result of annealing is due to the development of recrystallization in the case of the alloy after pressing and the dissolution of the strengthening α" phase in the case of the alloy after radial-shear rolling and aging.

1. Introduction

In the past two decades, bulk ultrafine-grained (submicro- and nanostructured) metallic materials obtained by severe plastic deformation (SPD) [1-3] have been actively developed and investigated. By the SPD effect, a grain-subgrain structure with an element size of less than 1 micron is formed in metals and alloys. Obtaining this structure can significantly change the mechanical and physical properties of such materials [1-4]. Due to this, they can find wide application in various industrial conditions. At the same time, ultrafine-grained (UFG) metals and alloys, obtained by the SPD methods have a high density of deformation defects, a large length of highly non-equilibrium interfaces and increased diffusion coefficients [1,2,5,6]. As a result, such materials show, as a rule, low thermal stability that requires additional research.

To obtain an UFG structure in metallic materials, several methods of SPD treatment have been developed, such as: high-pressure torsion, equal-channel angular pressing, multiple (or abc) pressing and others [1–4]. These methods provide the accumulation in the material of high degrees of plastic strain (usually ε > 4-6) for refining the structure. In recent years, along with the aforementioned, new methods of deformation influence appear, which allow processing to be carried out in conditions close...
to industrial ones. Such methods include radial-shear rolling, through which it is possible to carry out deformation processing of cylindrical rods [7,8]. As was shown in [7,8], the combination of radial-shear rolling followed by aging allows the structure to be significantly reduced and the mechanical characteristics of the near β titanium alloy to be significantly improved. At the same time, it should be noted that comparative studies of the structure and mechanical characteristics of samples obtained by different SPD methods were not previously carried out using the example of near β titanium alloys. In this regard, in the present work, a comparative study of the structure, mechanical properties, and thermal stability of ultrafine-grained near β titanium alloy Ti-5Al-5Mo-5V-1Cr-1Fe obtained by the methods of multiple pressing and radial-shear rolling with subsequent aging is carried out.

2. Material and methods

A commercial near β titanium alloy (Russian alloy VT22) with a chemical composition: Ti-4.74 wt.% Al-5.57 wt.% Mo-5.04 wt.% V-0.81 wt.% Cr-0.98 wt.% Fe was used for the investigations. An ultrafine-grained structure in the alloy was obtained by multiple (abc) pressing with a change in the deformation axis [9] on an IP-2000 press in the temperature range 800-450 °C in billets with the dimensions of 25×25×40 mm³. The strain for one pressing was about 0.5 and the total accumulated strain \( e \) was about 8. Radial-shear rolling was carried out on a rolling mill "14-40" in the temperature range 850-750 °C. The initial and final diameter of the bars before and after the rolling was 40 and 17 mm, respectively. Thus, the extraction ratio (strain) of the bars by radial-shear rolling \( e \) was 1.7.

Aging of the alloy after radial-shear rolling was carried out in a SNOL-16251/11 air furnace at a temperature of 480 °C for 5 hours. Annealing of the samples of the alloy with an ultrafine-grained structure was carried out in the same furnace in the temperature range 500-800 °C for one hour.

Tensile tests of samples in the form of a double blade with a gage size of 5×1.7×0.8 mm³ were carried out on a PV-3012M machine equipped with a strain gauge load measurement system \( s \) with an initial strain rate \( 6.9 \times 10^{-3} \text{ s}^{-1} \) at room temperature. Samples for tensile tests were cut by the electric-spark method. Before testing, a layer about 100 μm thick was removed from the surface of the samples by mechanical grinding and subsequent electrolytic polishing with an electrolyte with the composition: 20% HClO4+80% CH3CO2H. Transmission electron microscopy studies of thin foils were performed using a Jeol JEM-2100 microscope at the Shared Use Center “NANOTEH” of ISPMS SB RAS. Foils for electron microscopy were prepared by the standard method on the “Micron-103” jet polishing unit using an electrolyte of the following composition: 20% HClO4+80% CH3CO2H. The study of fractographic raptures after tensile tests was performed by scanning electron microscopy using a Quanta 200 3D microscope. The phase composition of the investigated titanium alloy after different SPD treatments was studied on a Shimadzu XRD-6000 diffractometer using CuKα radiation, equipped with a monochromator.

3. Results and discussions

3.1. Microstructural investigations

As shown on figure 1, in the result of severe plastic deformation by the method of multiple pressing a homogeneous ultrafine-grained structure with an average grain size of 0.2 μm was formed in the near β alloy. A complex deformation contrast inside the grains (figure 1a) indicates high internal stresses. Microdiffraction patterns obtained using a selector diaphragm with an area of ~ 1.6 μm² show a large number of individual reflexes located around the circumference, which indicates predominantly high angle misorientations of the neighboring grains (figure 1a).
Radial-shear rolling under the above mentioned regime followed by aging at a temperature of 480 °C for 5 hours leads to the formation of a heterogeneous hierarchical microstructure in the Ti-5Al-5V-5Mo-1Cr-1Fe alloy (figure 2). In the volume of the initial large β-phase grains (~150 μm) separated as a result of deformation into sub-grains about 0.5 microns in size precipitates of α-phase particles with sizes of 0.2-0.7 μm formed during the process of radial-shear rolling at temperatures below the temperature of complete polymorphic transformation (transus) are observed [10]. Inside the β subgrains a fine-acicular structure is formed (figure 3b), which is a consequence of the formation of the metastable α" phase during the decomposition of the solid β solution at annealing (aging). Figure 2b shows an example of α"-phase precipitation in β subgrains.

An X-ray studying showed that after both treatments, the UFG alloy is a mixture of two phases α / β. The ratio of the phases in the alloy produced by multiple pressing and radial-shear rolling with subsequent aging is 62/38 and 69/31, respectively. Such a difference in the phase composition of the alloy samples obtained by the two SPD methods indicated above may be due to the difference in their mechano-thermal processing.

Thus, as a result of treatment of the Ti-5Al-5Mo-5V-1Cr-1Fe alloy using the SPD methods indicated above, a UFG structure is formed in it, which differs significantly in homogeneity and size of the structural elements in the considered states.
3.2. Mechanical behaviour and fracture

Despite the larger size of the elements of the grain-subgrain structure detected by the TEM method, alloy samples after radial-shear rolling and subsequent aging demonstrate higher strength properties at room temperature compared with the alloy obtained by multiple pressing (figure 3). The tensile strength in the first case reaches 1695 MPa, while in the second - 1600 MPa. These properties significantly exceed the corresponding properties obtained in the UFG Ti-6Al-4V alloy produced by the method of multiple pressing [11] and ECAP [12]. At the same time, the character of the tensile curves of the alloy after these treatments varies considerably. Therefore, the alloy after multiple pressing demonstrates the deformation behavior typical for UFG materials obtained by SPD methods with a short hardening stage (<1%) and a long (~9%) softening stage (figure 3) [2, 3]. In the case of samples of an alloy obtained by radial-shear rolling with subsequent aging, the tensile curve demonstrates a longer stage of work hardening (up to 2%) with a slightly lower coefficient and a shorter stage of softening (figure 3). At the same time, the ductility of the alloy after multiple pressing is more than twice higher than the corresponding value in the alloy after rolling and subsequent aging (figure 3).

The study of the fracture surfaces of alloy samples after tensile tests showed that in both cases there is a quasi-viscous rapture with a smaller dimple size in the case of the alloy obtained by the method of multiple pressing, which corresponds to the size ratio of the structural elements (figure 4). The character of the rapture patterns suggests that in the case of the alloy obtained by the method of multiple pressing there is a more viscous fracture than in the case of the alloy after rolling and aging (figure 4), which agrees well with the flow curves of these materials (figure 3).

Figure 3. Tensile curves of UFG Ti alloy produced by abc pressing (1) and radial-shear rolling with subsequent aging (2).

Figure 4. Fractographic raptures of the near β titanium alloy produced by multiple pressing (a) and radial-shear rolling with subsequent aging (b) after tensile tests.
3.3. The effect of anneals

Studying the effect of hourly annealing in the temperature range of 500-800 °C on the mechanical properties of the UFG alloy obtained by various SPD methods showed that the alloy obtained by the method of multiple pressing exhibits higher thermal stability compared to the alloy obtained by the method of radial-shear rolling with subsequent aging (figure 5). So, after annealing at a temperature of 500 °C, the ultimate strength of the alloy obtained by the method of multiple pressing is practically unchanged, whereas in the alloy after rolling and aging it decreases noticeably. With a further increase in the annealing temperature, a significant drop in the strength properties of the alloy in both structural states is observed (figure 5). At that, if in the titanium alloy obtained by radial-shear rolling with subsequent aging, an increase in ductility with decreasing strength is observed, then in the alloy after multiple pressing, the ductility after anneals changes in that or in the other direction (figure 5).

Structural studies of the alloy showed (figure 6) that the reasons for the decrease in its strength properties as a result of annealing in different structural states are different. So in the case of the alloy obtained by the method of multiple pressing after annealing at a temperature of 700 °C, the development of the recrystallization process is observed (figure 6a). At the same time, a globular structure with an average grain size of about 0.5 μm is retained. Annealing at the same temperature of the alloy obtained by radial-shear rolling with subsequent aging does not lead to a noticeable growth of the elements of the grain-subgrain structure (figure 6b). At the same time, the thin-acicular precipitations of the metastable α"-phase, which observed in the alloy after aging, are not detected after the annealing, which apparently, leads to a significant drop in its strength properties under tension at room temperature.

![Figure 5](image5.png)

**Figure 5.** Dependence of ultimate strength (1,2) and ductility (3,4) on annealing temperature for pressed (1,3) and rolled (2,4) alloy.

![Figure 6](image6.png)

**Figure 6.** Microstructure of the near β titanium alloy produced by multiple pressing (a) and radial-shear rolling with subsequent aging (b) after annealing at 700 °C for 1 hour.
4. Conclusions
Comparative studies of the structure, mechanical properties, and thermal stability during annealing in the temperature range of 500-800 °C of the ultrafine-grained near β titanium alloy Ti-5Al-5Mo-5V-1Cr-1Fe obtained by the methods of multiple pressing and radial-shear rolling with subsequent aging were carried out. It was shown that as a result of SPD by the method of multiple pressing, the formation of a homogeneous micro-duplex UFG structure with an average grain (phase) size of ~0.2 μm was observed. Radial shear rolling followed by aging led to the formation of an inhomogeneous structure with precipitates of particles of the α phase and a size of subgrains of the β phase of about 0.5 μm, inside which thin-acicular precipitations of the metastable α” phase were observed. Despite the significantly smaller imposed strain and the larger size of the grain-subgrain structure elements, the alloy samples obtained by radial-shear rolling and subsequent aging demonstrated higher strength properties at room temperature compared to the alloy obtained by multiple pressing (the ultimate strength of the alloy after these treatments was 1695 and 1600 MPa, respectively). At the same time, the alloy obtained by multiple pressing showed a higher ductility and thermal stability of the mechanical characteristics due to the micro-duplex UFG structure. At the same time, the drop in strength as a result of annealing in the alloy obtained by pressing was due to the development of recrystallization with conservation of the globular structure, whereas in the alloy after rolling and aging it was associated with the dissolution of strengthening α”-phase precipitates.

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