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CHANGES IN THE STRUCTURAL-PHASE STATE AND MECHANICAL PROPERTIES OF VT35 ALLOY AFTER SEVERE PLASTIC DEFORMATION AND SUBSEQUENT ANNEALING

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The influence of severe plastic deformation and subsequent pre-recrystallization annealing on the structural-phase state and mechanical properties of the VT35 titanium alloy is studied. It is shown that the formation of an ultrafine-grained structure leads to a 65−75% increase of the room-temperature mechanical properties of this alloy compared to its initial coarse-grained state. A significant scatter in the mechanical properties of the alloy after such treatment is attributed to the inhomogeneity of the α and β phase distribution in the bulk of the workpieces. A subsequent pre-recrystallization annealing of the VT35 ultrafine-grained alloy at the temperatures of 723 and 773 K leads to an additional increase in its mechanical properties (the yield strength and ultimate tensile strength values can exceed 1700 MPa). Using the samples annealed at 723 K for 0.5 h as an example, it is shown that this increase is accounted for by a larger volume fraction of the α-phase due to the β→α phase transformation occurring inside the β phase regions and, hence, a more uniform α and β phase distribution.

Keywords: titanium alloys, severe plastic deformation, ultrafine-grained structure, phase transformations, annealing, mechanical properties.

INTRODUCTION

The advances of modern engineering are in many respects conditioned by the design of new structural materials and their development and introduction into practice. These materials, in particular titanium alloys extensively used in different industries, should possess higher service and technological properties. Recently, a good promise has been demonstrated by the proposed approaches of deformation-heat treatment of industrial prefabricated parts in forming the target properties of the materials, involving the impact by severe plastic deformation. The bulk materials and alloys produced by this method generally have an ultrafine-grained (submicro- and nanocrystalline) structure (grain size d smaller than 1 and 0.1 μm, respectively) and exhibit a unique combination of physical and mechanical properties. In particular, these materials possess high strength and demonstrate low-temperature and/or high-velocity superplasticity [1−3].

By now, a large number of the studies on the impact of deformation-heat treatment, including severe plastic deformation, on the structure and properties of polycrystalline materials have been performed using the (α+β) titanium alloys as an example [1, 2, 4−7], while the data on the more strongly alloyed transition-class alloys (pseudo-β titanium alloys) are still lacking. These alloys are, however, promising in fabricating critical components and units in the aerospace and automobile engineering in terms of enhancing their service properties [8−11]. There are numerous well-known schemes of deformation-heat treatments designed for transition titanium alloys [12–19]. It should be noted that

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the target service properties in these alloys are achieved by forming various crystal lattice defects via plastic deformation developed in them and as a result of decomposition of metastable phases in the course of aging. Moreover, their mechanical properties can be further improved by forming ultrafine-grained (UFG) structure in these transition alloys by severe plastic deformation followed by heat treatments. However, it is clear that the use of these treatments on the transition-class alloys would have certain peculiarities different from the case of the ordinary (α+β) titanium alloys. In particular, there might be a change in the evolution of some processes taking place in the UFG transition-class titanium alloys during the severe plastic deformation and subsequent heat treatments. Therefore, a comprehensive study of these processes is necessary for the sake of improving the functional characteristics of the UFG transition titanium alloys, including the processes of microstructure formation and evolution, phase transitions, etc. In view of the above, an investigation of common factors of the UFG structure formation in the course of severe plastic deformation of the transition-class titanium alloys and peculiarities of their evolution during different heat treatments is thought to be very important.

EXPERIMENTAL MATERIAL AND TECHNIQUES

The initial material was a commercial wire rod of the VT35 alloy (Ti–2.7Al– 14.5V–2.8Sn–2.8Cr–1.0Mo–0.9Zr). The ultrafine-grained structure of this wire was formed by the method of multidirectional pressing [20] of the ∅25×40 mm workpieces in an IP-2000 pressing machine in a temperature interval of 873−723 K. The strain degree within a single pass was ~0.5 over the workpiece height. The tensile tests of the dumb-bell samples with the gage section size of 5×1.7×0.8 mm were performed in a PV-3012 M testing machine equipped with a load measuring tensometric system with an automated recording of the flow curves in the time – load coordinates at a rate of $6.9 \cdot 10^{-3}$ s^{-1} at room temperature. The samples were formed by the electro-spark cutting. Before the tests, a layer of about 100 μm was removed from the sample surfaces by mechanical grinding followed by electrolytic polishing. The electron microscopy examinations of the thin foils were performed in a JEM-2100 microscope at the SUC NANOTECH center of the ISPMS SB RAS. The foils for TEM studies were prepared by a standard method in a Mikron-103 jet polishing facility using the following electrolyte compositions: 20% $HClO_4$+80% CH_3CO_2H. The sizes of the grain-subgrain structure elements were determined from the dark-field images. The sampling size was no fewer than 200 measurements. The surface microstructure studies were performed at the Tomsk Regional Core Shared Research Facilities Centre of Tomsk State University by the methods of scanning electron microscopy in Quanta 200 3D microscope equipped with tungsten cathodes and a Pegasus system for electron backscatter diffraction (EBSD). The phase composition was investigated with a Shimadzu XRD-6000 in the CuK_α radiation.

RESULTS AND DISCUSSION

Earlier [21] we showed the formation of ultrafine-grained structure in the VT35 alloy after its processing by the method of multidirectional pressing in the temperature interval within 973−773 K, with the average grain-subgrain substructure size of $d \sim 0.12$ μm. It has been noted that an increase of the total strain degree of the workpieces in the interval of 823−773 K improves the homogeneity of the resulting ultrafine-grained structure and mechanical properties of the alloy. The tensile room-temperature tests of the VT35 alloy after the above treatment demonstrated that its yield stress and ultimate tensile strength values are 1450 and 1380 MPa, respectively [21] (Table 1).

In this work we proceed with the studies on optimization of the VT35 alloy pressing regime. The lower bound temperature of the alloy processing was decreased to 723 K. The electron microscopy examinations demonstrated that the average size of the ultrafine-grained structure elements formed in the VT35 alloy after its multidirectional pressing is 0.11 μm (Fig. 1). There is a complex deformation contrast on the TEM images observed in the bulk of the grains. No individual dislocations are resolved. The ring-like microdiffractions, given a small size of the field diaphragm ~1.6 μm^2), suggest a large fraction of the high-angle grain boundaries (Fig. 1*a*). On the other hand, the EBSD studies revealed the presence of inhomogeneities in the form of the β-phase areas (Fig. 2*a*). Inside the latter, there is also a developed grain-subgrain structure with the relatively small-sized α-precipitates (Fig 2*b–f*). According to the XRD

TABLE 1. Mechanical Properties of VT35 Alloy at Room Temperature

Thermomechanical treatment	σ_{UTS}, MPa	$\sigma_{0.2}$, MPa	δ, %
Coarse-grained state	860±20	850±20	19±2
Multidirectional pressing (973−773 K) [21]	1450±20	1380±20	8±1
Multidirectional pressing (873−723 K)	1540±30	1490±30	2.5±0.5
Multidirectional pressing (873−723 K)+annealing at 723 K, 0.5 hr	1720±30	1700±30	1.5±0.5
Multidirectional pressing (873−723 K)+annealing at 723 K, 1 hr	1640±30	1610±30	4±0.5
Multidirectional pressing (873−723 K)+annealing at 773 K, 0.5 hr	1650±40	1610±40	6±1

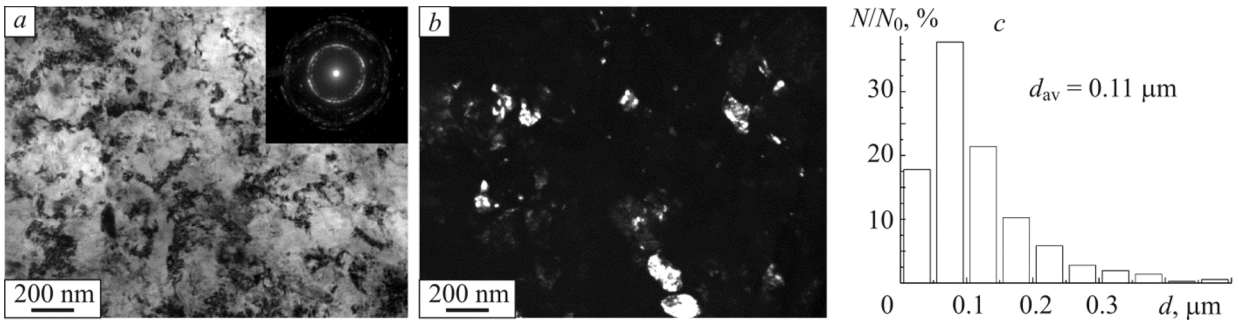

Fig. 1. Microstructure (*a*, *b*) and grain-subgrain size distribution (*c*) of the UFG VT35 alloy after multidirectional pressing.

data, the volume fractions of the α- and β-phases are found to 41 and 59%, respectively. The levels of the lattice microdistortions $\Delta d/d$ in this case are $\sim 1.2 \cdot 10^{-3}$ and $\sim 3 \cdot 10^{-4}$ in the α- and β-phases, respectively (Fig. 3, Curve *1*, Table 2).

The studies of mechanical properties of the VT35 alloy at room temperature demonstrated that the formation of this structural-phase state results in an increase of the yield stress and ultimate tensile strength values up to 1540 and 1490 MPa, respectively, at a simultaneous decrease of the value of elongation to failure (Table 1, Fig. 4, Curve *1*). A comparatively large scatter of the resulting mechanical properties in the treated VT35 alloy seems to be due to the inhomogeneity of the structure formed in it. Earlier [22−24], using titanium alloys subjected to a similar treatment as an example, we demonstrated that an additional heat treatment of the alloy after its multidirectional pressing can homogenize the resulting structure and simultaneously enhance its mechanical properties. Relying on these data, in the present work we studied the influence of additional annealing on the structure and mechanical properties of the VT35 alloy subjected to multidirectional pressing. The presented data (Table 1, Fig. 4) demonstrate that an additional annealing treatment of the VT35 alloy after the severe plastic deformation increases its ultimate tensile strength and yield stress. It should be noted that after annealing at 723 K for 1 hr and at 773 K for 0.5 hr the value of the elongation to failure also increases. The highest increase of the ultimate tensile strength and yield stress is observed after annealing at 723 K for 0.5 hr (Fig. 4).

In order to reveal the reasons for the above-described change of the mechanical properties of the VT35 alloy, we investigated the structural-phase state of the samples annealed at 723 K for 0.5 hr, after which the highest ultimate tensile strength and yield stress values were observed.

The electron microscopy examination of the UFG VT35 alloy structure after an additional annealing at 723 K for 0.5 hr did not reveal any substantial changes. The average size of the grain-subgrain structure elements did not practically change and was found to be 0.1 μm (Fig. 5*b*). It might be noted that the grain boundaries on the TEM images are more distinct (Fig. 5*a*). The latter circumstance could be due to the recovery processes taking place in the boundaries under these annealing conditions. On the other hand, the XRD studies and the EBSD analysis demonstrated an active initiation of phase transformations in the alloy during its annealing. Firstly, according to the XRD data the α-

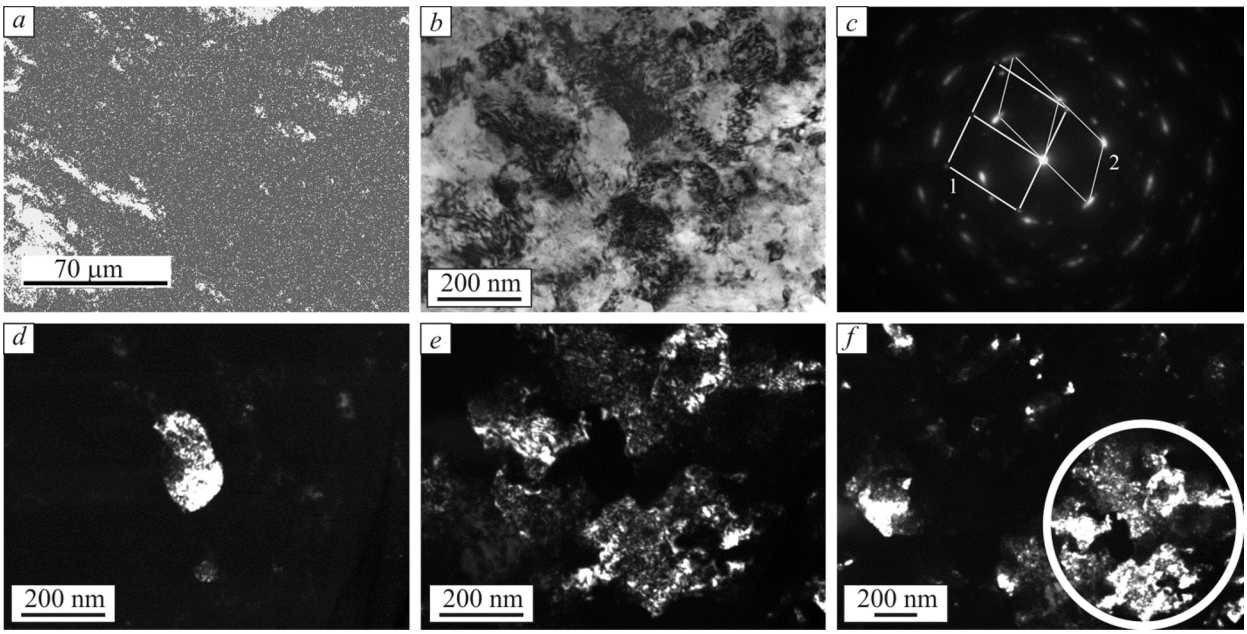

Fig. 2. Microstructure of VT35 alloy after multidirectional pressing: *a* – distribution of α- and β phases taken by EBSD (dark-gray – α-phase, light gray – β-phase); *b* – bright-field image; *c* – microdiffraction patterns and schematics of microdiffractions of (–331) planes of the α-phase (1) and (111) planes of the β-phase (2): *d* – dark-field image of an α-phase grain, *e* – dark-field image of the β-region with an α-phase grain; *f* – dark-field image of microstructure with a magnified β-phase region given in Fig 2*e*.

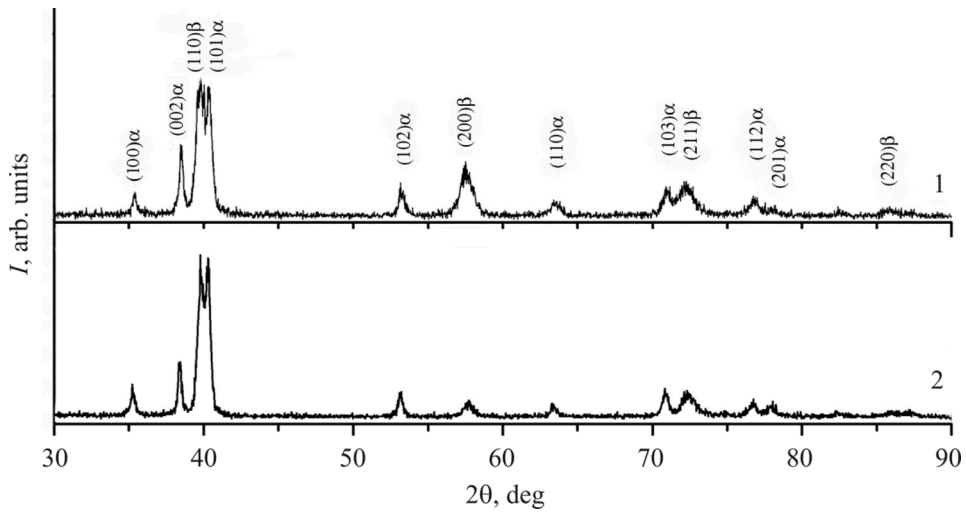

Fig. 3. XRD pattern from VT35 alloy: Curve *1* – multidirectional pressing; Curve *2* – multidirectional pressing + annealing at 723 K for 0.5 hr.

phase volume fraction increases to 48%. As this goes on, the level lattice microdistortions in the α-phase is retained quite high and is found to be ~$3 \cdot 10^{-3}$, while in the β-phase it increases by an order to about $4 \cdot 10^{-3}$ (Table 2). Secondly, the studies involving a EBSD analysis revealed a more homogeneous structure of the alloy in terms of the α- and β-phase distribution (Fig. 5*c*) after its annealing.

TABLE 2. Structure Parameters of VT35 Alloy after Multidirectional Pressing in Temperature Interval of 873−723 K and Subsequent Annealing

Thermomechanical treatment	d, μm	α/β, %	$\Delta d/d$ (α)	$\Delta d/d$ (β)
Multidirectional pressing	0.11	41/59	$1.2 \cdot 10^{-3}$	$3 \cdot 10^{-4}$
Multidirectional pressing + annealing 723 K, 0.5 hr	0.1	48/52	$3 \cdot 10^{-3}$	$4 \cdot 10^{-3}$

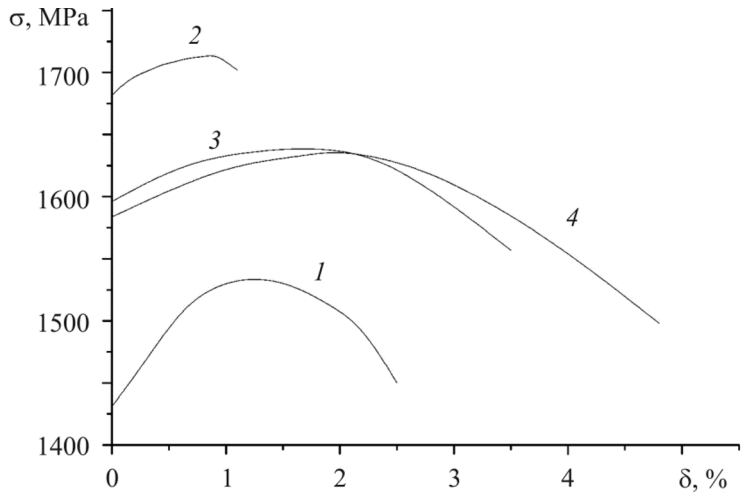

Fig. 4. Engineering stress–strain curves of VT35 alloy: Curve *1* – multidirectional pressing; Curve *2* – additional annealing at 723 K, 0.5 hr; Curve *3* – additional annealing at 723 K, 1 hr; Curve *4* – additional annealing at 773 K, 0.5 hr.

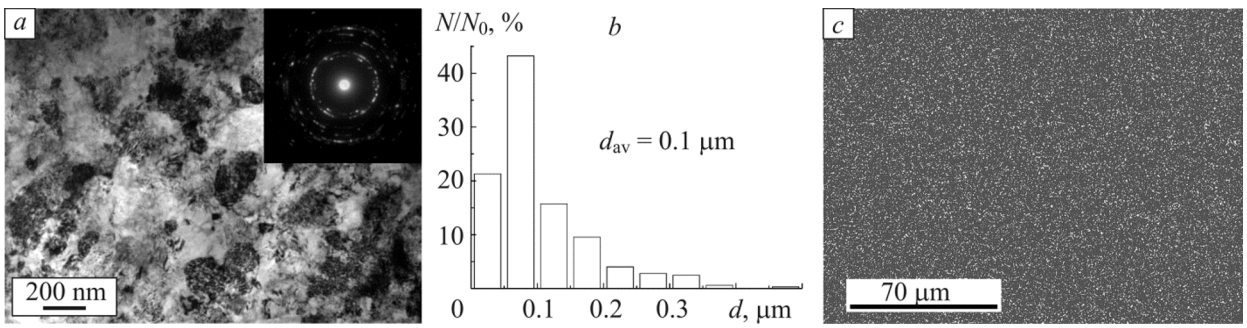

Fig. 5. Microstructure (*a*), grain-subgrain size distribution bar chart (*b*) and α- and β-phase distribution taken using EBSD analysis (dark gray – α-phase, light gray – β-phase) (*c*) of UFG VT35 alloy after its multidirectional pressing.

Briefly, it has been shown that the formation of an ultrafine-grained structure with the average grain-subgrain size of 0.11 μm results in an increase of its ultimate tensile strength and yield stress values to 1540 and 1490 MPa, respectively. However, there is a considerable scatter of mechanical properties depending on the workpiece area from which the samples were cut. Relying on the results, this scatter was attributed to the structure inhomogeneities in the form of the β-regions up to tens of micrometers in size, which were observed in the alloy after its severe plastic

deformation. Inside these regions there is a developed grain-subgrain structure formed by the method of multidirectional pressing. It has been found out that pre-recrystallization annealing of the VT35 alloy at the temperatures of 723 and 773 K additionally enhance its mechanical properties. The values of the yield stress and ultimate tensile strength of the VT35alloy can exceed 1700 MPa. The studies performed on the alloy samples annealed at 723 K, 0.5 hr have shown that the mechanical properties are improved due to an increased volume fraction of the α-phase as a result of the β→α phase transition inside the β-phase regions and, as a consequence, a more homogeneous distribution of the α- and β-phases.

CONCLUSIONS

It has been shown that severe plastic deformation of the VT35 titanium alloy using the method of multidirectional pressing results in the formation of an ultrafine-grained structure with an average grain-subgrain structure element size of 0.11 μm. After this treatment, the room-temperature values of ultimate tensile strength and yield stress of this alloy are found to be about 1540 and 1490 MPa, respectively, which is 65−75% higher than those observed for the coarse-grained state. It has been found out that the scatter of the alloy mechanical properties after this treatment is due to an inhomogeneous distribution of the α- and β-phases in the alloy volume. Additional pre-recrystallization annealing of the UFG VT35 alloy at the temperatures of 723 and 773 K reduces the distribution inhomogeneity by virtue of the β→α transformation and enhances its strength properties to the values higher than 1700 MPa, while maintaining the average size of the grain-subgrain structure elements at the same level.

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