



The effect of preliminary treatment with subsequent aging on structural-phase state and mechanical properties of β titanium alloy

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The effect of aging at 723 K on the structural-phase state and mechanical properties of VT35 titanium alloy after β quenching and various treatments using severe plastic deformation has been studied. It is shown that annealing at 1073 K followed by quenching and aging leading to the precipitation of large α -phase lamellae with volume fraction of 20% doesn't have a strengthening effect on the alloy. After processing by radial shear rolling and subsequent aging fine α -phase precipitations in the form of plates and nanometer-sized particles with a volume fraction of 24% are observed in β -phase grains. However, large areas of β -phase without α -phase precipitates are observed. The strength properties of the alloy after the treatment increase by 40–45%. The processing by the method of multiple pressing leads to the formation of a mixed α/β UFG structure with a grain/subgrain size of 0.11 μm . Subsequent aging leads to the decomposition of the residual β -phase and to a homogeneous distribution of α and β phases in the alloy with volume fraction of α -phase 51%. The formation of such an UFG structural-phase state leads to an increase in the strength properties of the alloy by a factor of almost two compared to the initial state.

Keywords: β titanium alloy, severe plastic deformation, phase transformations, strength.

1. Introduction

At present, the use of high-strength products and semi-finished items from promising titanium-based alloys is largely due to the fact that they successfully combine various properties — high impact strength, corrosion resistance, high specific strength and fatigue, and some others. However, the development of modern technology imposes increasingly high requirements on such materials in terms of operational and technological properties. The most promising of the approaches proposed in recent years to solving the problem is the deformation-heat treatment of industrial semi-finished products, including the effect of severe plastic deformation and subsequent heat treatments [1–6]. At the same time, the achievement of specified operational properties of titanium alloys occurs due to the formation of an ultrafine-grained (UFG) structure in them, the creation of various types of crystal lattice defects by plastic deformation, and also as a result of the decomposition of metastable phases during subsequent annealing. At present, a large number of studies on the effect of this treatment on the structure and properties of polycrystalline materials have been carried out on the example of ($\alpha + \beta$) titanium alloys [1, 2, 7–10], while the treatment of more alloyed β titanium alloys has been studied to a much lesser degree. At the same time, such alloys are promising in the manufacture of critical parts and assemblies of aerospace and automotive equipment in order to improve its performance [11–13].

There are a fairly large number of methods of deformation-heat treatment developed for high-alloy titanium alloys [14–20]. Moreover, an additional increase in the mechanical properties

of these alloys is possible due to the formation of an UFG structure using various methods of severe plastic deformation and subsequent heat treatments [21–23]. Obviously, the application of these treatments to β titanium alloys will have its own characteristics compared to conventional ($\alpha + \beta$) alloys. In particular, it is possible to change the development of the processes of formation and evolution of the microstructure, phase transformations, occurring in UFG titanium alloys directly during severe plastic deformation [24, 25] or subsequent heat treatments [21, 23]. In connection with the above, in the present work, comparative studies of the effect of annealing (aging) at a temperature of 723 K on the structural-phase state and mechanical properties of the β titanium alloy VT35 (Ti-15V-3Cr-3Al-3Sn-1Mo-1Zr) were carried out after quenching from the β -phase region (1073 K) and various deformation-thermal treatments using the methods of severe plastic deformation.

2. Material and methods

A commercial titanium alloy VT35 (Ti-14.5V-2.7Al-2.8Sn-2.8Cr-1.0Mo-0.9Zr) in the form of a rod 25 mm in diameter was used as the initial material for the studies. The temperature of complete polymorphic transformation of this alloy is 1033 K [14]. Radial shear (helical) rolling of a billet with dimensions of $\varnothing 25 \times 200 \text{ mm}^3$ was carried out on a screw rolling mill “14-40” at a temperature of 1073 K. The total reduction ratio was 2.1 (true strain ≈ 0.8). Aging was carried out at a temperature of 723 K for 5 hours. The ultrafine-grained structure was obtained by pressing with a change in the deformation axis on an IP-2000 press in

the temperature range of 873–723 K in workpieces with dimensions of $\varnothing 25 \times 40 \text{ mm}^3$ [21]. The strain for one pressing along the sample height was ≈ 0.5 and the total accumulated strain was ≈ 8 . Tensile tests of samples in the form of a double blade with gauge dimensions of $5 \times 1.7 \times 0.8 \text{ mm}$ were conducted on a PV-3012M machine at room temperature and a strain rate $6.9 \cdot 10^{-3} \text{ s}^{-1}$. The samples were cut by the electrospark method. Before testing, a layer about 100 μm thick was removed from the surfaces of the samples by mechanical grinding and subsequent electrolytic polishing.

Transmission electron microscopy studies of thin foils were carried out with a Jeol JEM-2100 microscope at an accelerating voltage of 200 kV at the Shared Use Center NANOTEH of ISPMS SB RAS. The foils for electron microscopy were prepared by the standard method using a Mikron-103 jet polishing machine with an electrolyte of the following composition: 20% HClO_4 – 80% $\text{CH}_3\text{CO}_2\text{H}$. The sizes of the grain-subgrain structure elements were determined from the dark-field images. Not less than 200 measurements were sampled. Structural studies of the alloy samples after different thermo-mechanical treatments were carried out by scanning electron microscopy using a Quanta 200 3D microscope with a tungsten cathode and an attachment for the electron backscatter diffraction (EBSD) analysis by Pegasus. Phase composition studies were performed by X-ray diffraction analysis by means of a Shimadzu XRD-6000 diffractometer using Cu K_α radiation equipped with a monochromator. Metallographic studies of the alloy were performed on an Olympus GX 71 microscope. The dislocation density in the bcc phase (β) was evaluated using the formula suggested in work [26]:

$$\rho = 2\sqrt{3} \langle \varepsilon^2 \rangle^{1/2} / (Db), \quad (1)$$

where $\langle \varepsilon^2 \rangle^{1/2}$ and D the volume-averaged values of microdistortions and size of coherent scattering regions

(CSR), respectively, b is the Burgers vector of an edge dislocation in the β -phase of titanium.

3. Results and discussion

The studies performed have shown that, in the initial state, the VT35 alloy has a coarse-grained structure. In this structure, the grains are elongated along the rolling direction (axis of the rod) by hundreds of microns and are strongly textured (Fig. 1). The grain size in the direction perpendicular to rolling direction is 60 μm on average. Inside the grains, as a rule, a large number of low-angle boundaries are observed (Fig. 1b). X-ray diffraction study shows that the volume fraction of the β -phase in the initial alloy reaches 100% (Table 1). In the initial state, a fairly low dislocation density ($\sim 1 \cdot 10^{11} \text{ cm}^{-2}$) is observed in the β alloy (Table 1) which is characteristic of annealed coarse-grained bcc metals and alloys [12].

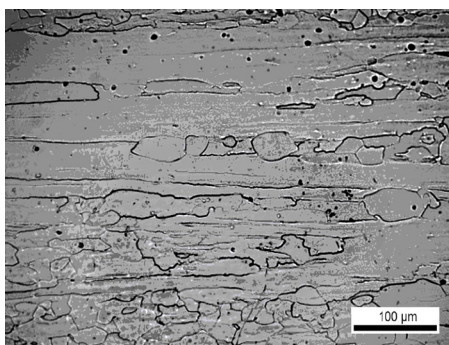
The room temperature tensile tests of the VT35 alloy in the initial state showed that it had relatively low strength properties (Table 2). At the same time, the alloy in this structural-phase state has a high ductility. The table also demonstrates the mechanical properties of the alloy after various thermo-mechanical treatments.

It is known that β titanium alloys are well hardened by heat treatment, which includes quenching from the β or $\alpha + \beta$ region and subsequent aging [14]. In this regard, in this work studies of the effect of treatments of this type on the structure and mechanical properties of the VT35 alloy were carried out.

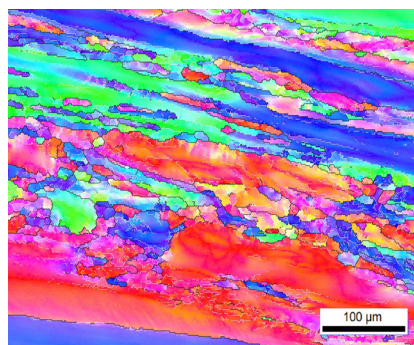
As it was shown above, the VT35 alloy under study in the initial state has an inhomogeneous coarse-grained structure with a pronounced texture. In this regard, in order to obtain a more uniform structure, the alloy was subjected to one-hour annealing in the β region at 1073 K followed by quenching. As a result of such treatment, complete recrystallization

Table 1. Structural-phase characteristics of VT35 alloy revealed by X-ray analysis after different thermo-mechanical treatments.

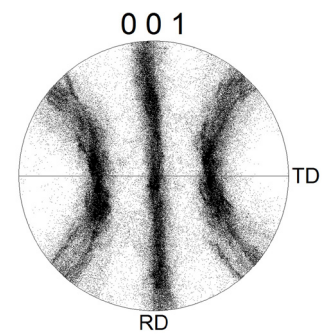
Treatment	α/β , %	CSR $_{\beta}$, nm	a_{β} , Å	$\langle \varepsilon^2 \rangle_{\beta}$	ρ_{β} , cm^{-2}
Initial state	0/100	127	3.2262	$3 \cdot 10^{-4}$	$1.2 \cdot 10^{11}$
Annealing 1073 K, 1 h	0/100	180	3.2398	$2 \cdot 10^{-4}$	$8.4 \cdot 10^{10}$
Annealing 1073 K, 1 h + quenching + aging 723 K, 5 h	20/80	83	3.2299	$1.9 \cdot 10^{-3}$	$5.6 \cdot 10^{11}$
Radial-shear rolling (RSR)	0/100	68	3.2280	$8 \cdot 10^{-4}$	$4.5 \cdot 10^{11}$
RSR + aging 723 K, 5 h	24/76	48	3.2156	$3.1 \cdot 10^{-3}$	$1.2 \cdot 10^{12}$
Multiple pressing	41/59	18	3.2111	$3 \cdot 10^{-4}$	$1 \cdot 10^{12}$
Multiple pressing + aging 723 K, 1 h	51/49	26	3.2000	$2.5 \cdot 10^{-3}$	$2.1 \cdot 10^{12}$



a



b



c

Fig. 1. (Color online) Microstructure of β titanium alloy in the initial state: metallographic image (a); EBSD map of grain structure (b); direct (001) pole figure (c).

Table 2. Mechanical properties of VT35 alloy at room temperature.

Treatment	UTS, MPa	YS, MPa	El, %
Initial state	860	850	19
Annealing 1073 K, 1 h + quenching + aging 723 K, 5 h	810	790	15
Radial-shear rolling at 1073 K + quenching + aging 723 K, 5 h	1200	1150	8
Multiple pressing + aging 723 K, 1 h	1640	1610	4

occurs in the alloy leading to the formation of globular grains with an average size of $\approx 69 \mu\text{m}$ (Fig. 2a). In this case, the texture characteristic of the initial state practically disappears (Fig. 2b). The volume fraction of the β -phase in the alloy is still 100% (Table 1).

The aging of the VT35 alloy after the above treatment was carried out at a temperature of 723 K for 5 hours. Such a treatment leads to the decomposition of the supersaturated β solid solution with the precipitation of the α phase in the form of large lamellae growing from the boundaries into the bulks of grains (Fig. 2c). In this case, the volume fraction of the α -phase reaches 20% (Table 1). After this treatment, however, the mechanical properties of the investigated alloy even slightly decrease as compared to the initial state (Table 2). Evidently, this fact can be explained by a decrease in the density of deformation-induced defects in the volume of β grains after recrystallization annealing (Table 1) and a strong size inhomogeneity of the formed grains. It should also be noted that there are a large number of β regions where the precipitations of α phase are not observed (Fig. 2c). This inhomogeneity of the precipitation of the α -phase is apparently due to the low density of deformation defects in the volume of β -grains after recrystallization annealing. The dislocation density in the VT35 alloy after annealing and hardening is $8.4 \cdot 10^{10}$ (Table 1). As it is known [27,28], the nucleation of the α -phase during aging begins primarily along the grain boundaries and on deformation defects. It should be noted that the above results are qualitatively similar to the results obtained in [29] for the investigated VT35 alloy.

To overcome the negative consequences of the treatment considered above, the VT35 alloy in the initial state was subjected to radial shear rolling in the β region at a temperature of 1073 K followed by water quenching. After such treatment, the volume fraction of the β -phase is still 100% (Table 1). The average grain size in the direction perpendicular to the rolling direction is about $22 \mu\text{m}$ (Fig. 3a,b). Despite the high rolling temperature, the dislocation density in the alloy increases by

almost an order of magnitude compared to the state after annealing (Table 1). A slight spreading of the initial texture is observed (Fig. 3c), which may indicate the beginning of the development of dynamic recrystallization. However, the structure of the alloy retains grains that are strongly elongated along the axis of the rod and are several hundred microns long, although their transverse size decreases to $20 - 40 \mu\text{m}$ (Fig. 3a,b).

As a result of aging of the VT35 alloy processed by radial shear rolling at a temperature of 723 K for 5 hours, a finely dispersed α -phase is precipitated in the form of lamellae and nanosized particles along the boundaries and in the volume of most β -grains (Fig. 4). The volume fraction of the precipitated α -phase is 24% (Table 1). At the same time, regions that are nearly free from precipitates of the α -phase are still observed in the alloy (Fig. 4a,b). The formation of such a microstructure (highly-disperse of α -phase and its fairly uniform distribution over the grain volume) leads to an increase in the strength properties of the VT35 alloy by approximately 40–45% compared to the initial state (Table 2). The values of ultimate strength and yield stress reach 1200 and 1150 MPa, respectively.

In order to obtain a more uniform distribution of α and β phases in the volume of the alloy under study, workpieces were processed using severe plastic deformation by the method of multiple pressing. It is known that after such processing, a structure with a large number of boundaries and a high density of deformation defects is formed [21,23]. These factors should contribute to a relatively uniform distribution of α and β phases in the bulk of the alloy. The conducted studies have shown that in the VT35 alloy after multi-axial pressing, a uniform grain-subgrain structure with an average element size of $0.11 \mu\text{m}$ is formed in the volume (Fig. 5). According to the data of transmission electron microscopy, a complex deformation contrast is observed in the volume of grains, in which individual dislocations are not detected (Fig. 5). Studies by X-ray diffraction analysis showed that

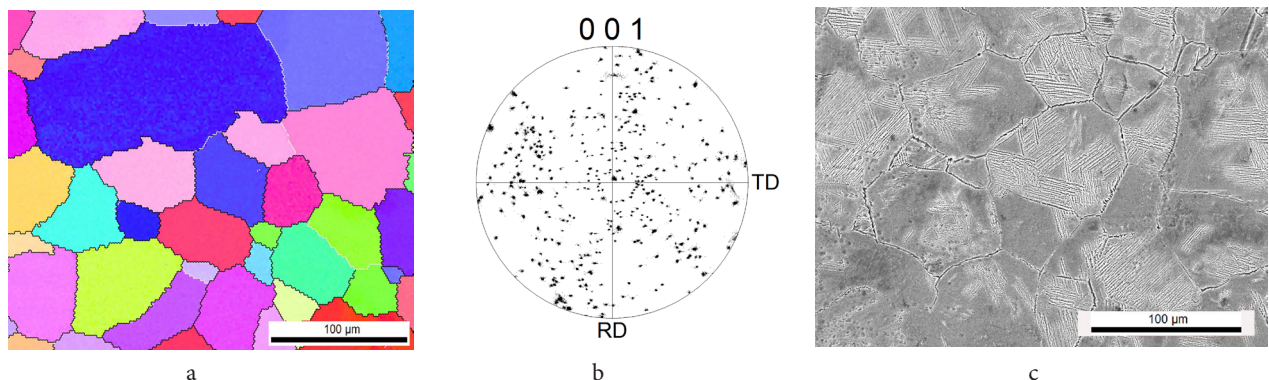


Fig. 2. (Color online) Microstructure of VT35 alloy after annealing at 1073 K, 1 h followed by water quenching: EBSD map of grain structure (a); direct (001) pole figure (b); metallographic image of the structure after annealing at 1073 K, 1 h and aging 723 K 5 h (c).

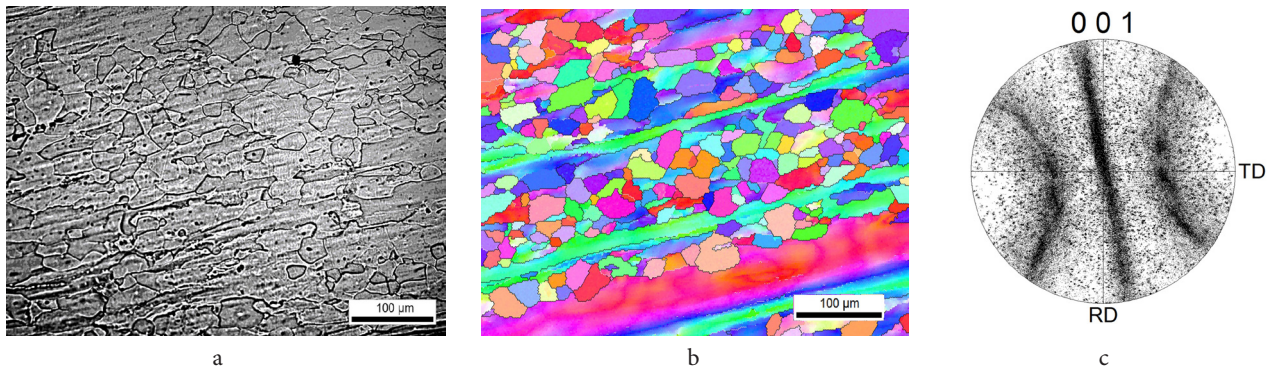


Fig. 3. (Color online) Microstructure of VT35 alloy after radial shear rolling: metallographic image (a); EBSD map of grain structure (b); direct (001) pole figure (c).

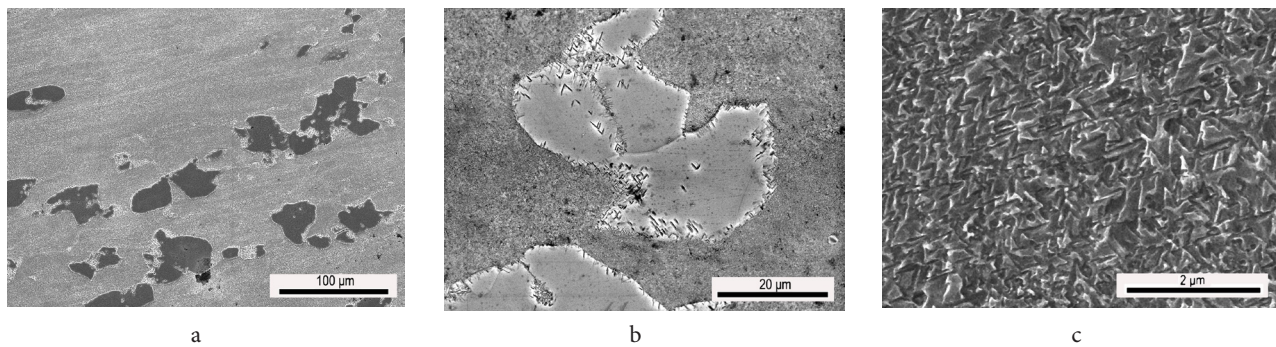


Fig. 4. Microstructure of VT35 alloy after radial shear rolling and aging at 723 K 5 h: image of mixed α/β phases and β phase (bright and dark fields, respectively) (a); internal structure of β field: some lamellae of α -phase located on grain boundaries and defects in β grain are seen (b); microstructure of regions consisting of the mixture of α and β phases (c).

the dislocation density in the alloy was $1 \cdot 10^{12} \text{ cm}^{-2}$ (Table 1). Ring-shaped microdiffraction pattern obtained with a small selector aperture diaphragm (area $\approx 1.6 \mu\text{m}^2$) indicates a large fraction of high-angle grain boundaries (Fig. 5a). However, studies carried out using the EBSD analysis method showed that inhomogeneities also occurred in the alloy structure in the form of large regions of the β -phase (Fig. 5c), within which relatively small precipitates of the α -phase are observed (Fig. 5c). The volume fractions of α and β phases, according to X-ray diffraction analysis, are equal to 41 and 59%, respectively.

Aging of the VT35 alloy samples after multiple pressing at a temperature of 723 K for an hour practically does not change the character of the microstructure (Fig. 6). The value of the average size of the elements of the grain-subgrain structure is $0.12 \mu\text{m}$. At the same time, it can be noted that the grain boundaries on electron microscope images are more distinct (Fig. 6a). The latter may be a consequence of the development of recovery processes within the boundaries during the considered annealing. At the same time, the studies carried out using X-ray diffraction and EBSD analyzes showed that during annealing, active development of phase transformations takes place in the alloy and further decomposition of residual β regions is observed. In this case, the structure of the alloy becomes more homogeneous with respect to the distribution of α and β phases (Fig. 6c). The volume fraction of α -phase increases up to 51%. The dislocation density in the residual β phase is $2.1 \cdot 10^{12} \text{ cm}^{-2}$ (Table 1). The considered processing of the VT35 titanium alloy (multiple pressing + aging) leads to a significant increase in its strength properties at room temperature compared to

the initial state (Table 2). In particular, the values of ultimate strength and yield stress are 1640 and 1610 MPa, respectively, while maintaining satisfactory ductility.

As is known, most hardening heat treatments of metastable titanium alloys include quenching and aging processes [14]. In this case, a different initial dislocation structure, even with an unchanged phase composition, significantly affects the aging kinetics, morphology, dimensions, and character of the distribution of the hardening phase [27,28]. As can be seen from the above experimental results, the formation of a recrystallized structure with a low dislocation density in β titanium alloy VT35 after heat treatment using quenching from 1073 K and aging at 723 K for 5 hours leads to the precipitation of coarse α -phase lamellae by a homogeneous mechanism. The volume fraction of this phase is about 20%. In addition, a large number of β regions free of α -phase precipitates are observed in the alloy. As a result, the considered heat treatment of the VT35 alloy does not lead to an increase in its mechanical (strength) properties.

The deformation processing of workpieces using radial shear rolling before quenching from 1073 K leads to a decrease in the average grain size and an increase in the dislocation density by about an order of magnitude (Table 1). As a result of this treatment during subsequent aging at 723 K, the decomposition of the β -phase in most grains proceeds according to a heterogeneous mechanism with the precipitation of the α -phase in the form of nanoscale plates throughout the volume. Although there are separate β grains in which the precipitates of the α -phase in the form of large plates are observed only at the grain and subgrain boundaries. It can be assumed that a low density of deformation defects

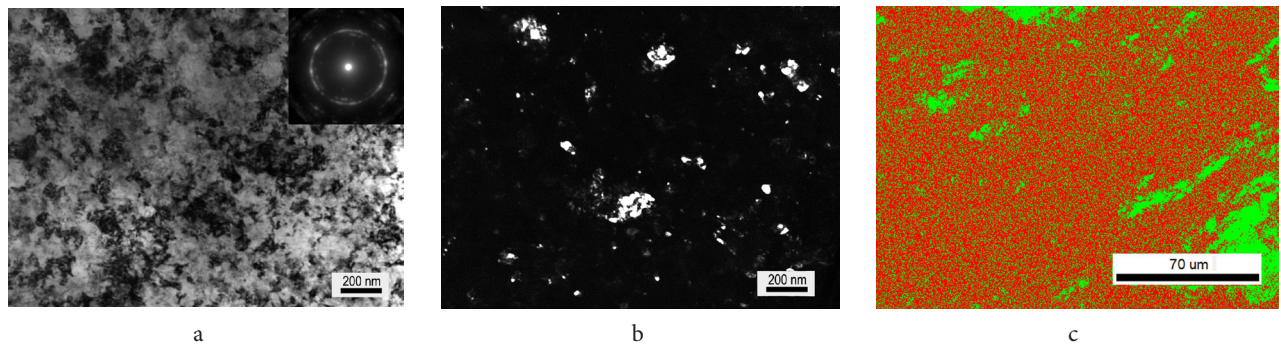


Fig. 5. (Color online) Microstructure of VT35 alloy after multiple pressing: bright (a) and dark (b) field images; EBSD map of α and β phase distribution (β -phase is lighter) (c).

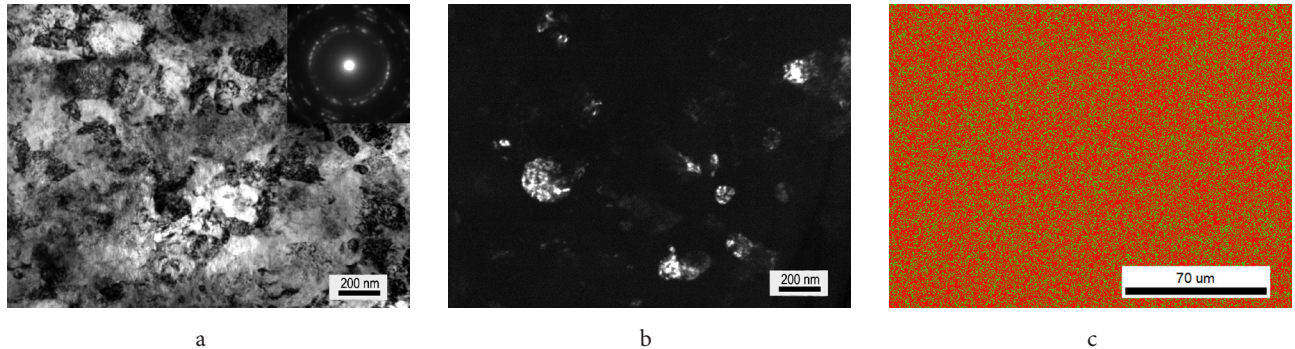


Fig. 6. (Color online) Microstructure of VT35 alloy after multiple pressing and aging 723 K, 1 h: bright (a) and dark (b) field images; EBSD map of α and β phase distribution (β -phase is lighter) (c).

is retained in these grains due to mechanical treatment of the alloy at a high temperature (1073 K). Thus, the formation of a structure with a high density and a small particle size of the α -phase, despite its relatively small volume fraction (24%), leads to an increase in the strength characteristics of the alloy VT35 by 40–45%.

It is known [1–6] that it is possible to significantly increase the dislocation density (up to 10^{12} cm^{-2}) in polycrystalline metals and alloys using the methods of severe plastic deformation. Such a treatment leads not only to an increase in the dislocation density, but also to the formation of a nonequilibrium ultrafine-grained structure with grain sizes less than a micron and, as a consequence, an increase in the density of interfaces. It can be expected that these changes in the structure of the alloy can lead to an acceleration of the development of processes that occur during the decomposition of a solid solution under the conditions of low-temperature annealing (aging) and, as a result, a change in the mechanism of α -phase precipitation. The above experimental results show that the treatment of the VT35 alloy by the method of multiple pressing leads to the formation of an UFG structural-phase state with an average size of grain-subgrain structure elements of about $0.11 \mu\text{m}$ and a volume fraction of α and β phases of 41 and 59%, respectively. These phases are distributed rather uniformly in the bulk of the alloy, although there are relatively large β regions, tens of microns in size.

As noted above, the UFG structural-phase state formed by deformation is strongly nonequilibrium. Therefore, even short-term annealing at a temperature of 723 K leads to further decomposition of the residual β -phase. Its volume fraction decreases to 49% and a complete disappearance of large β regions is observed (Fig. 6). Studies using

transmission electron microscopy have shown that in the volume of the investigated alloy after this thermomechanical treatment, there are no lamellar precipitates of the α -phase. It can be assumed that the decomposition of the β -phase occurs according to one (or both together) of the following mechanisms. The first one is that during the decomposition of the β -phase, nanosized globular particles of the α -phase are formed, which then increase in size during the heat treatment of the alloy. Second, there is a transition of ultrafine-grained grains as a whole from the β to the α -phase due to a change in the concentration of alloying elements in the bulk of the grain. To elucidate this issue, further research is needed.

4. Conclusion

It is shown that quenching of β titanium alloy VT35 after one-hour annealing at 1073 K followed by aging at 723 K for 5 hours leads to the precipitation of the α -phase in the form of large lamellae by a homogeneous mechanism. The volume fraction of the precipitated phase in this case reaches 20%. However, such a treatment does not lead to an increase in the strength properties of the alloy, which is apparently due to the low volume fraction of the α -phase and the low density of deformation defects after recrystallization annealing.

The deformation treatment of the VT35 alloy by the method of radial shear rolling leads to a change in the mechanism of decomposition of the β -phase solid solution during aging from the homogeneous to the heterogeneous one. This may be due to an increase in the density of deformation defects after the deformation treatment and subsequent quenching. Precipitation of the α -phase occurs in the form of nanoscale plates and particles in most β -grains throughout the volume. However, in individual grains, the

precipitations of the α -phase in the form of large plates are observed only at the grain and subgrain boundaries. The volume fraction of the α -phase after aging reaches 24%. The formation of such a structural-phase state leads to an increase in the strength properties of the VT35 alloy by 40 – 45%.

Processing of β titanium alloy VT35 by the method of multiple pressing leads to the formation of an ultrafine-grained structure with a size of structural elements of 0.11 μm and a uniform distribution of α and β phases in the volume. However, in this case, regions of tens of microns in size remain in the alloy, in which β -grains are predominantly observed. Subsequent heat treatment of the alloy at 723 K for an hour leads to further decomposition of the β -phase in the indicated areas and a uniform distribution of the α and β phases over the volume of the alloy. The volume fraction of the α phase in this case reaches 51%. The consequence of the formation of such UFG structural-phase state is an increase in the strength properties of the VT35 alloy by almost in two times compared to the initial state.

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