

CONDENSED-STATE PHYSICS

THE EFFECT OF SEVERE PLASTIC DEFORMATION AND SUBSEQUENT ANNEALING ON THE STRUCTURE AND MECHANICAL PROPERTIES OF TITANIUM ALLOY PT-3V

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The effect of severe plastic deformation by multi-axial pressing followed by thermal treatment on the structure and mechanical properties of titanium alloy PT-3V (4.66% Al : 1.92% V) has been investigated. Submicrocrystalline structure formation is shown to dramatically improve the mechanical properties of the alloy at room temperature and to decrease the superplastic flow temperature interval by 250–300 K. Low-temperature annealing of the material in a submicrocrystalline state is found to cause further increase in the yield strength and ultimate strength as compared to the preannealing level, with the strain at fracture being nearly doubled.

Keywords: titanium alloys, severe plastic deformation, submicrocrystalline structure, mechanical properties, superplasticity.

INTRODUCTION

Development of advanced materials with target service and technological characteristics is a major challenge of modern materials engineering. Several innovative approaches to solving the problem at hand have been proposed in recent years. Among these is thermomechanical treatment of semifinished products, including severe plastic deformation (SPD) that holds the greatest promise. Bulk metals and alloys thus produced are generally of ultrafine-grained (submicro- and nanocrystalline) structure (grain size d is < 1 and $< 0.1 \mu\text{m}$, respectively). In certain conditions, the materials may possess a unique combination of physical and mechanical properties, such as high strength, low-temperature and/or high-rate superplasticity, etc. [1–4]. By and large, the special features of the behavior of the materials in question are poorly understood, however. For instance, low thermal stability of the ultrafine-grained structure gives no way of making a direct use of many of the thermomechanical treatment regimes developed for coarse-grained materials. Even in the case where the annealing temperature is lower than the recrystallization point, relaxation of internal stresses, recovery of strain-induced defects, and deterioration of the mechanical properties are observed [1–4]. As the annealing temperature is increased, the grain size is increased dramatically, and unique properties of the alloys are lost. At the same time, there is some evidence that efficient thermal treatment regimes can be selected to improve the mechanical properties of submicro- and nanocrystalline alloys produced by means of the SPD technique as compared to their untreated analogues [5–7]. This opens up new opportunities for thermal treatment of this kind of alloys, bringing about controllable changes in their structure and properties and improving their thermal stability. However, the physical nature of processes developing during annealing remains to be explored. It will take a large number of experimental investigations to solve this problem. Thus, the challenge is to perform a comprehensive research into processes involved in different kinds of thermal treatment, including formation and evolution of

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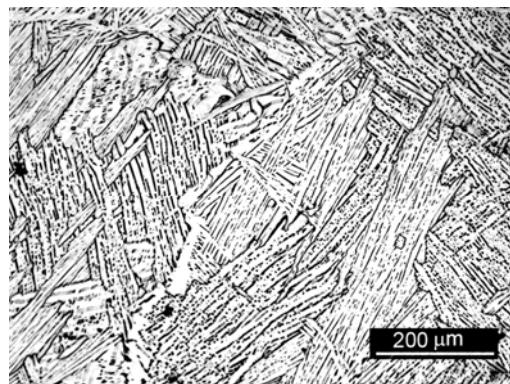


Fig. 1. Metallographic structure of titanium alloy PT-3V (4.66% Al: 1.92% V) in a coarse-grained state.

microstructure, phase transitions, etc., for improving the functional characteristics of currently existing metals and alloys and developing advanced ultrafine-grained materials. In this connection, it is important to investigate the mechanisms of formation of ultrafine-grained structure and special features of structural evolution of the materials under study for different kinds of thermal treatment used.

TEST MATERIALS AND EXPERIMENTAL TECHNIQUES

The initial material was a rod of titanium alloy PT-3V (4.66% Al : 1.92% V). Submicrocrystalline structure was produced by means of multi-axial pressing [8] at $T = 1073\text{--}723\text{ K}$. Dumb-bell specimens measuring $5 \times 1.7 \times 0.8\text{ mm}$ in the gage section were subjected to tensile tests using a testing machine fitted with a strain-measuring system. The latter measured the load, performing an automatic recording of flow curves in a load-time plot in vacuum at 10^{-2} Pa at a rate of $6.9 \cdot 10^{-3}\text{ s}^{-1}$ in the interval from room temperature to 1173 K. Test specimens were prepared by an electric discharge machining technique. Prior to tests, a $\sim 100\text{ }\mu\text{m}$ thick layer was removed from the surface of the specimens by grinding followed by electrolytic polishing. Thin-foil electron microscopy was performed in a transmission electron microscope equipped with a goniometer. The foils were prepared in a polishing machine (20% HClO₄ + 80% CH₃CO₂H) by a standard technique. The size of the grain-subgrain structure elements was determined from dark-field images. The sample contained no less than 200 measurements. Investigations on the phase composition and texture were performed by a CuK_a diffractometer (Shimadzu XRD-6000). An optical microscope (Olympus GX 71) was employed for a metallographic examination.

RESULTS AND DISCUSSION

Test titanium alloy PT-3V (4.66% Al: 1.92% V) in the as-delivered condition was of coarse-grained martensitic structure (Fig. 1). The volume fracture of the β -phase in the alloy made up 1–2%. The titanium alloy under study exhibited moderate yield and ultimate strength. When subjected to multi-axial pressing, the material developed a homogeneous submicrocrystalline structure. Electron microscopy revealed that subsequent to multi-axial pressing, the size of the grain-subgrain structure elements was basically in the range $0.1\text{--}1\text{ }\mu\text{m}$. The microdiffraction patterns demonstrated a large number of reflections arranged in circles (Fig. 2a). Reflection density as high as this for a small selected aperture area ($\sim 1.8\text{ }\mu\text{m}^2$) is evidence of grain-subgrain structure with a submicron grain size. The average grain size of the grain-subgrain structure elements determined from dark-field images was $\sim 0.27\text{ }\mu\text{m}$ (Fig. 3a). Multi-axial pressing caused marked changes in the mechanical properties of the alloy in question at room temperature as compared to the coarse-grained material. For instance, the yield strength $\sigma_{0.2}$ and the ultimate strength σ_b of the specimens were increased by $\sim 60\%$ (see Table 1), with the strain at fracture δ falling down to 7%.

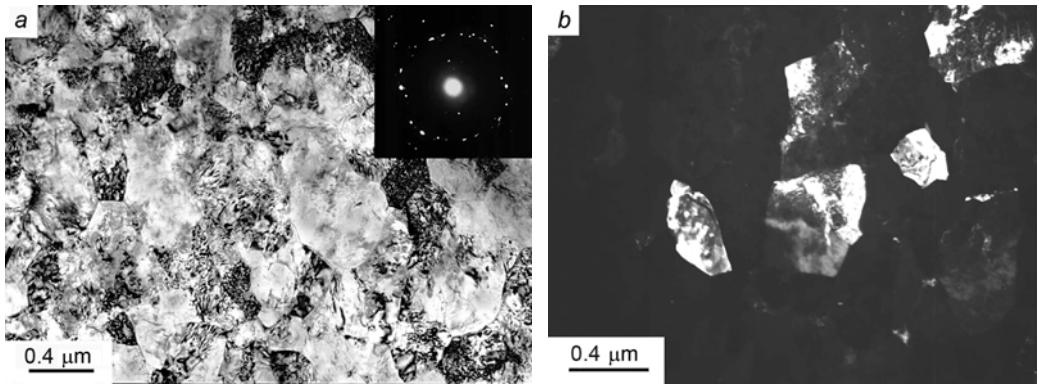


Fig. 2. Structure of alloy PT-3V subjected to multi-axial pressing: bright-field (*a*) and dark-field images (*b*).

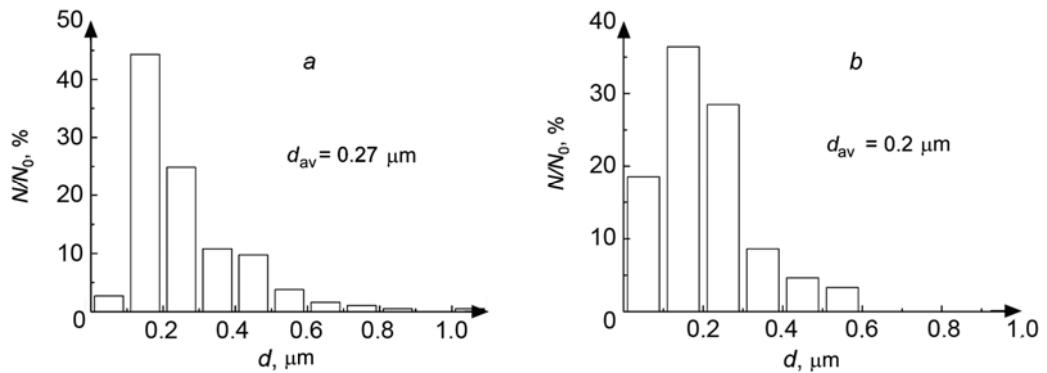


Fig. 3. Histograms of size distribution of the grain-subgrain structure elements of titanium alloy PT-3V: on multi-axial pressing (*a*) and on multi-axial pressing and subsequent annealing at 673 K for 6 h.

The temperature dependence of the yield strength and ultimate strength of the submicrocrystalline alloy was found to be much stronger than for the coarse-grained material up to 673 K (curves 1 and 3 in Figs. 4*a* and *b*). As the test temperature was increased above this point, the submicrocrystalline material exhibited a sharper softening than its coarse-grained cousins. As a result, σ_B and $\sigma_{0.2}$ get much lower in the submicrocrystalline state than in the coarse-grained one. The elongation at failure of the submicrocrystalline alloy was as high as ~400% at 873 K (curve 3 in Fig. 4*b*). The coarse-grained alloy under consideration did not transform into a superplastic state under the experimental conditions used. This is why we have investigated the temperature dependence of the yield strength and ultimate strength of a fine-grained alloy produced by annealing of submicrocrystalline alloy PT-3V at 973 K for 1 h. The average grain size was $d_{av} = 6 \mu\text{m}$. It is evident from Fig. 4*b* (curve 2) that the elongation at failure of the alloy was 300% at 1173 K. Thus, the temperature at which titanium alloy PT-3V in the submicrocrystalline state starts to exhibit superplasticity is 250–300 K lower than in the fine-grained state (curves 2 and 3 in Fig. 4*b*).

The effect of thermal treatment on the structure and mechanical properties of alloy PT-3V in the submicrocrystalline state has been investigated on annealing at 673 K for 6 h. Mechanical tension tests have shown that thermal treatment causes further increase in the yield strength and ultimate strength at room temperature. The elongation at failure of the specimens increases dramatically (Table 1). Electron microscopy has revealed that the strain-induced defect density inside the grains is decreased (Fig. 5). What is more, relatively large grains of size ~1 μm disappear (see the histograms in Fig. 3). A modest reduction in the average size of the grain-subgrain structure elements is observed. Hand in hand with these effects, there is a considerable increase in the number of grains of size

TABLE 1. The Mechanical Properties of Titanium Alloy PT-3V at Room Temperature for Different Kinds of Treatment

Treatment	$\sigma_b \pm 20$, MPa	$\sigma_{0.2} \pm 20$, MPa	$\delta \pm 1$, %
As-delivered condition	720	670	14
Multi-axial pressing	1170	1120	7
Multi-axial pressing + annealing, 673 K, 6 h	1240	1200	13

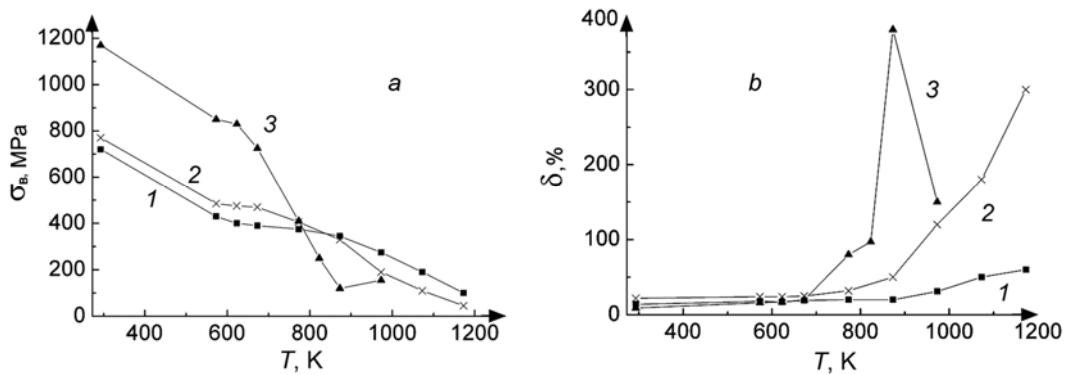


Fig. 4. Temperature dependence of ultimate strength (a) and percentage elongation at failure of titanium alloy PT-3V (b) for $d \sim 700$ (1) and $\sim 6 \mu\text{m}$ (2) and $d_{av} = 0.27 \mu\text{m}$ (3) of grain-subgrain structure elements.

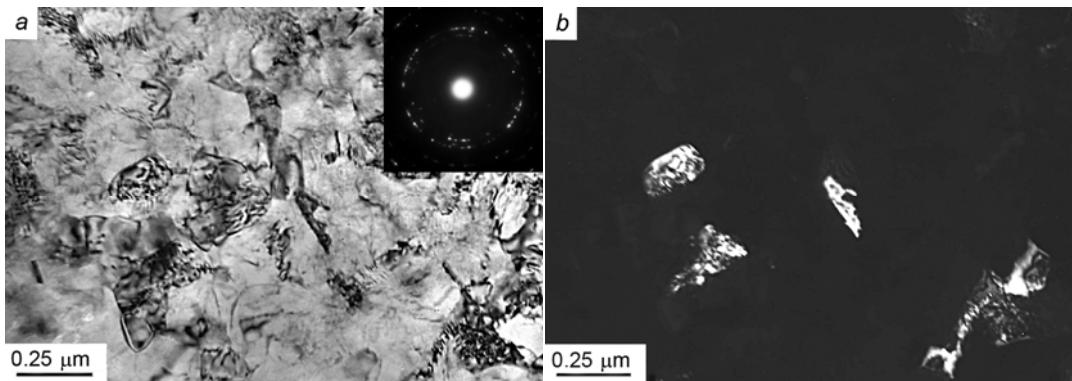


Fig. 5. Structure of titanium alloy PT-3V subjected to multi-axial pressing and subsequent annealing at 673 K for 6 h: bright-field (a) and dark-field images (b).

<0.1 μm (see Fig. 3). Structural changes such as these may be due to grain subboundaries and grains formed during annealing. To gain insight into the structural changes of the material, we have performed an x-ray diffraction analysis of submicrocrystalline alloy PT-3V before and after annealing. As indicated by x-ray diffraction patterns in Fig. 6, annealing of the alloy subjected to multi-axial pressing changes the intensity ratio between virtually all principal maxima.

Literary evidence shows [9] that hardening of α-β-titanium alloys may result from ageing of the materials during annealing in the temperature interval 623–873 K. In this case, the mechanical properties can be improved, say, due to decomposition of a residual β-phase to form a fine-grained α-phase. However, as shown by the x-ray diffraction analysis, the amount of the alloy β-phase is about 1–2%, and its decomposition is unlikely to bring about

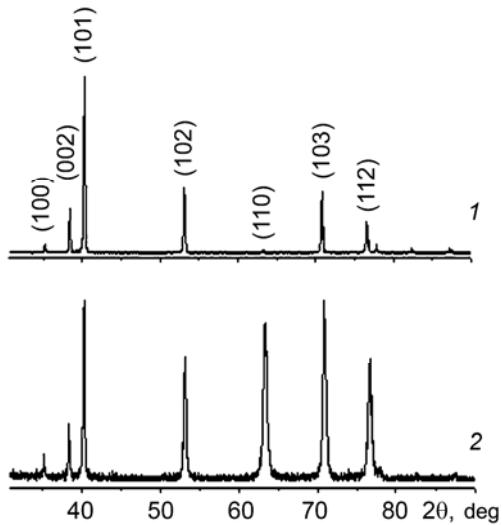


Fig. 6. Diffraction patterns for titanium alloy PT-3V: on multi-axial pressing (1) and on multi-axial pressing and subsequent annealing at 673 K for 6 h (2).

the aforementioned structural changes. On the other hand, metallic materials subjected to SPD are known to exhibit high strain-induced defect density and high internal stresses [1–4]. These factors may be responsible for the development of recrystallization at relatively low temperatures. The experimental data on the changes in the texture of the alloy and a significant increase in the volume fraction of grains of size $<0.1 \mu\text{m}$ favor the fact that recrystallization takes place under the annealing conditions used. At low temperatures, the newly-formed grain growth slows down substantially with subsequent grain refinement under the conditions in question.

Thus, the data obtained from electron microscopy and x-ray diffraction analysis lead us to suggest that annealing of titanium alloy PT-3V at 673 K gives rise to a more homogeneous submicrocrystalline structure, redistribution of dislocations inside the grains, and reduction in the average size of the grain-subgrain structure elements due to grains formed during annealing. It is believed that the foregoing annealing regime produces changes in the internal stress pattern inherent to the alloy under consideration. Yet further investigations need to be performed to check the validity of this assumption. These changes are likely to result in improved strength and plasticity of the alloy specimens subjected to annealing.

CONCLUDING REMARKS

Formation of submicrocrystalline structure of titanium alloy PT-3V (4.66% Al: 1.92% V) is found to bring about significant changes in the mechanical properties of the material. At room temperature, the yield strength and ultimate strength are increased by 60% as compared to the coarse-grained alloy. Note that plasticity is quite satisfactory in this case. In addition, the temperature interval for superplastic flow of the as-received material is decreased by 250–300 K. For temperatures varied between room temperature and 673 K, the mechanical properties of submicrocrystalline alloy PT-3V subjected to tension are much higher than those observed in the coarse-grained state. Annealing of the submicrocrystalline alloy at 673 K is shown to improve its structural homogeneity and reduce the average size of the grain-subgrain structure elements due to new grains formed during annealing. The structural changes in the alloy give rise to further increase in the yield strength and ultimate strength of the material, with the strain at fracture being nearly doubled.

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REFERENCES

1. R. Z. Valiev and I. V. Alexandrov, Bulk Nanostructured Metallic Materials [in Russian], IKTs “Akademkniga”, Moscow (2007).
2. Yu. R. Kolobov, R. Z. Valiev, G. P. Grabovetskaya, *et al.*, Grain Boundary Diffusion and Properties of Nanostructured Materials [in Russian], Nauka, Novosibirsk (2001).
3. O. A. Kaibyshev and F. Z. Utyashev, Superplasticity, Structure Refinement, and Hard-to-Deform Alloy Treatment [in Russian], Nauka, Moscow (2002).
4. N. I. Noskova and R. R. Mulyukov, Submicrocrystalline and Nanocrystalline Metals and Alloys [in Russian], UrB RAS, Ekaterinburg (2003).
5. A. A. Popov, R. Z. Valiev, I. Yu. Pyshmintsev, *et al.*, Fiz. Met. Metalloved., **83**, No. 5, 127–133 (1997).
6. Yu. R. Kolobov, O. A. Kashin, E. E. Sagymbaev, *et al.*, Russ. Phys. J., **43**, No. 1, 77–85 (2000).
7. R. Z. Valiev, A. V. Sergueeva, and A. K. Mukherjee, Scripta Mater., **49**, No. 7, 669–674 (2003).
8. V. A. Vinokurov, I. V. Ratochka, E. V. Naydenkin, *et al.*, RF Patent № 2388566 (May 10, 2010).
9. U. Zwicker, Titan und Titanlegierungen, Springer-Verlag, Berlin-Heidelberg-New York (1974).