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Refinement of Microstructure and Mechanical Properties of Titanium Processed by Twist Extrusion and Subsequent Rolling

V. V. Stolyarov*, Ya. E. Beigel'zimer, D. V. Orlov**, and R. Z. Valiev***

** Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, ul. K. Marksa 12, Ufa, 450000 Bashkortostan, Russia*

*** Donetsk Physicotechnical Institute, ul. R. Lyuksemburg 72, Donetsk, 83114 Ukraine*

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Abstract—Microstructure and mechanical properties as well as thermal stability of commercially pure titanium VT1-0 subjected to severe plastic deformation by twist extrusion (TE) were studied before and after low-temperature annealing and cold rolling. TE leads to a refinement of structural fragments to sizes of less than 1 μm, enhancement of strength, and the appearance of strong anisotropy of mechanical properties. Subsequent low-temperature annealing increases plasticity of the deformed titanium, retaining its enhanced strength. A combined treatment (TE and rolling) results in an additional refinement of the microstructure, which remains thermally stable to 300–350°C, and changes the anisotropy of mechanical properties.

INTRODUCTION

It is well-known that the structure and properties that are formed upon deformation of metals are determined to a great extent by the states of stress and strain and the regimes of deformation and that the most strong effects are observed when using severe plastic deformation (SPD) [1–5]. Development of the SPD methods—torsion under quasi-hydrostatic pressure (high-pressure torsion (HPT)), equal-channel angular pressing (ECAP), and multiple forging—allowed one to reach high degrees of deformation without final changes in the shape of workpieces and to obtain massive samples with ultrafine-grained (UFG) structure and unique physical and mechanical properties [1].

Comparatively recently, a new SPD method was suggested—twist extrusion (TE); it differs from the known methods in both the states of stress and strain of a workpiece and in its technological possibilities [6, 7]. In particular, TE can be realized using hydroextrusion setups [6] to obtain long-dimension high-precision shaped profiles (including cylinders with an axial channel), which are most efficient types of metal semiproducts in machine building [8–10]. No other known SPD methods permit one to obtain products of such type.

The TE process ensures accumulation of large deformations in metals, substantially refines their structure, and significantly improves mechanical characteristics [11], but, at the same time, leads to the appearance of strong anisotropy of properties, which is frequently undesirable [12].

It is known that the anisotropy caused by one type of deformation can be eliminated by another type of deformation. In this connection, it is important to investigate the possibility of improvement of mechanical properties of workpieces subjected to TE by means of their subsequent deformation different from TE. For long-dimension profiles this may be, for example, rolling or hydroextrusion. The main type of deformation in these processes is elongation of the profile along its axis, whereas the main type of deformation upon TE is simple shear in the plane perpendicular to the extrusion axis.

The aim of this work is to study the microstructure and anisotropy of mechanical properties of commercially pure titanium VT1-0 upon two-stage cold deformation. At the first stage, the samples were subjected to TE [6, 7, 11, 13], which made it possible to reach high degrees of deformation. After TE, templates cut from these samples were cold-rolled in flat rolls.

Titanium was chosen as the material for the investigation because of its great practical importance and the need for enhancement of its strength properties. Besides, there is already a considerable number of publications on titanium, which allows one to compare its structure and properties after SPD by different methods [2–5, 12].

EXPERIMENTAL

As the material for the investigation, we chose a hot-rolled rod ∅20 × 100 mm of commercially pure VT1-0 titanium containing (wt %) 0.12 O, 0.01 H, 0.04 N, 0.07 C, and 0.18 Fe. According to the material certificate, the presence of aluminum to 0.3 wt % is possible in the heat, but its content in the rod has not been determined.

The twist extrusion is one of new processes of metal forming used at present for obtaining UFG structures in metals by SPD methods [6, 7, 11–13]. In this method, a prismatic workpiece is forced through a die with a helical channel (Fig. 1). As a result, the material experiences deformation that changes its structure and properties but retains the cross-sectional dimensions and shape of the workpiece unaltered, which makes it possible to perform multiple extrusions to accumulate high degrees of deformation.

Among different SPD methods, ECAP is closest to TE in technological features, but the states of stress and strain of metal in these processes differ substantially. It was shown in [6, 11] that, to a first approximation, a simple shear of deformed material in the plane perpendicular to the extrusion axis occurs at the entrance into the helical part of the die. In this case, the character and distribution of deformation over the workpiece's cross section are similar to those that are realized in the scheme of high pressure torsion of disk samples. At the exit from the helical part of the die, the deformed state of metals is analogous to that at the entrance but with the components of the strain-rate tensor that are opposite in sign [6, 11].

According to [11, 14], a simple shear in narrow layers, as compared to other types of deformation, is most efficient for the formation of UFG structures; and cyclic deformation, according to [15], provides a higher plasticity in comparison with monotonic deformation. On the whole, this points to the twist extrusion as a promising method for obtaining UFG materials.

In [11], the following relationships for the greatest $\varepsilon_{i\max}$ and the least $\varepsilon_{i\min}$ values (within the workpiece cross section) of the degree of deformation ε_i after one pass upon TE were derived:

$$\begin{aligned} \varepsilon_{i\,\max} &= \tan\beta_{\max}; \\ \varepsilon_{i\,\min} &= 0.4 + 0.1\tan\beta_{\max}. \end{aligned} \tag{1}$$

The degrees of deformation at the quasi-monotonic (entrance and exit) parts are half as high as those calculated by formulas (1).

Several schemes can be employed to realize TE [6, 11]. In this work, the scheme of hydromechanical twist extrusion (Fig. 2) was used, in which the workpiece together with the die are in the medium of a high-pressure working liquid. This allows one to enhance the technological plasticity of the deformed metal and to increase the intensity of fragmentation [11, 14–18]. An I-20 commercial oil was used as the working liquid. The hydrostatic pressure in the container was equal to 700 MPa.

The deformation was effected at room temperature on a setup for hydromechanical twist extrusion of the vertical type with a container's working chamber 50 mm in diameter that was designed for operative pressures to 1500 MPa and was mounted at the base of a hydraulic press with an operating force of 4000 kN. The helical die had a cross section of 14 × 16 mm, and the inclination of the helix channel to the pressing axis

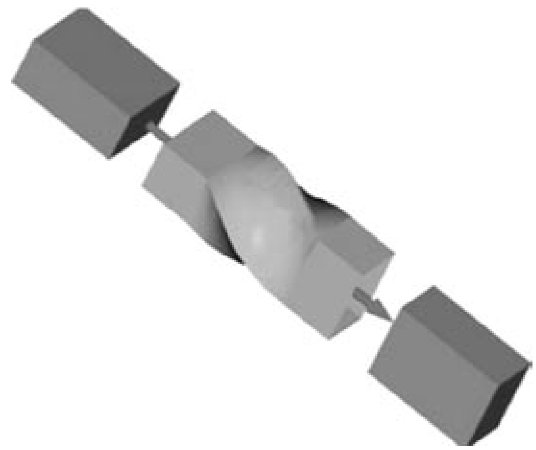

Fig. 1. Scheme of twist extrusion.

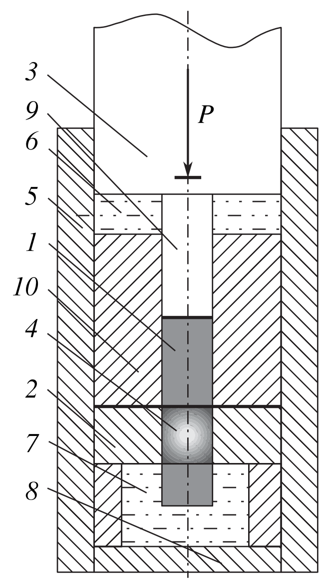

Fig. 2. Setup for hydromechanical twist extrusion (schematic): *1*, workpiece; *2*, helical die; *3*, plunger; *4*, schematic of the helical channel of the die; *5*, container; *6* and *7*, two high-pressure chambers connected together; *8*, valve; *9*, rod; and *10*, auxiliary bushing.

was $\beta_{\max} \approx 60°$. At such parameters of the hardware, according to (1), the deformation per pass that was averaged over the workpiece cross section was $\varepsilon_{i\text{av}} = 0.5(\varepsilon_{i\min} + \varepsilon_{i\max}) = 1.15$, which is equal to the deformation per pass upon ECAP with a right angle between channels [14]. The strain rate in this case was on the order of 0.5 s^{-1}.

The titanium workpieces with dimensions of 70 × 14 × 16 mm were first subjected to a threefold cold twist extrusion, which made it possible to produce a highly refined structure in them [12]. The total degree of strain after TE was equal to $\varepsilon_{i\text{av}} \approx 3.45$.

Plates with dimensions of 30 × 15 × 5 mm were cut in the longitudinal direction from the middle part of the workpiece that underwent deformation by TE. The

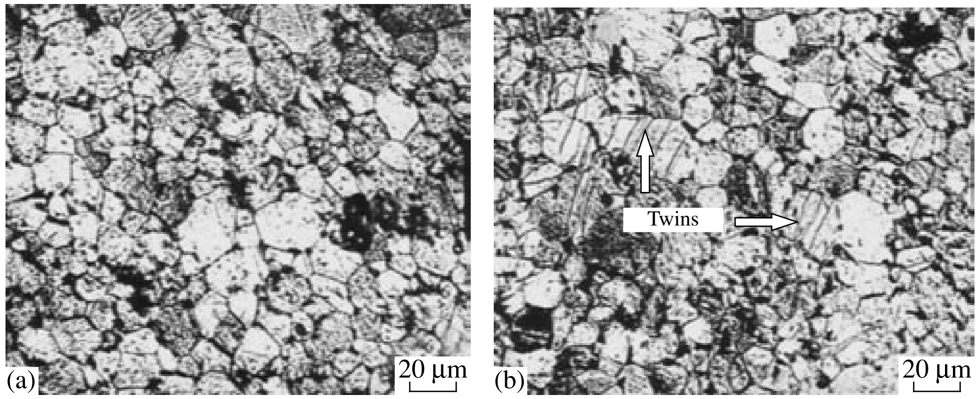

Fig. 3. Microstructure of the initial titanium in (a) transverse and (b) longitudinal sections.

plates were rolled in the extrusion direction in cylindrical rolls 100 mm in diameter in a few passes to a thickness of 2.5 mm. The total degree of deformation upon rolling was $\varepsilon_i \approx 0.70$ (reduction to 50%).

The titanium microstructure was analyzed with the help of a Neophot-32 optical microscope at magnifications to 2000 times (observation of the structure at high magnifications was realized using an immersion objective) and a JEM-100C transmission electron microscope. The electron diffraction patterns were taken from an area of 2 μm^2. The average size of structural elements (grains, fragments, etc.) was calculated by the intercept method from dark- and bright-field images.

To carry out tensile mechanical tests before and after extrusion of the rod, as well as after rolling, plates 0.7 mm thick whose plane coincided with the extrusion direction were cut from the rod. Then, flat dumbbell-shaped specimens 0.7 × 1 mm in cross section with a gage length of 5 mm were spark-cut along and across the rolling direction. The tensile tests were conducted at room temperature on an Instron testing machine at a velocity of the cross arm of 0.5 mm/min (strain rate 1.6×10^{-3} s^{-1}). The average values of mechanical properties were determined using three specimens for each state.

After annealing in a temperature range of 100–700°C for 1 h, microhardness measurements were performed in order to analyze the thermal stability of titanium obtained using a combination of TE and cold rolling. The microhardness was measured on a plate sample (after rolling in the longitudinal and transverse directions relative to the rolling axis) using a PMT-4 microhardness tester at a load of 100 g for 15 s.

RESULTS ND DISCUSSION

Workpieces As-Observed after TE

After TE, defects such as distortions of the shape of the end parts, incomplete filling of the cross section of the die channel, twist of a workpiece about the extrusion axis, and its fracture because of the exhaustion of the reserve of plasticity can be observed. The majority of these defects are related to the insufficient counterpressure [11]. In this work, the level of counterpressure was high enough (on the order of the yield strength of titanium) to eliminate fracture of the metal and distortion of the shape in the middle part of workpieces in the course of three passes through the helical die. The realization of the fourth pass without a preliminary annealing resulted in fracture (recall that the deformation was performed at room temperature).

Since the counterpressure was effected by a liquid rather than a rigid punch, some faceting of the vertices was observed at the end parts of the workpieces [11]. These defects propagated along the workpiece axis for a length of about 5–7 mm from each butt end.

The length of inhomogeneous parts of workpieces upon straight (conventional) extrusion is on the order of the diameter of the cross section of the die channel [8]. For this reason, in spite of the fact that defects of the shape of workpieces propagated for a length of no more than 5–7 mm, segments 15 mm long were cut away from each butt end to ensure elimination of the end effects.

Microstructure

Figures 3 and 4 display micrographs and diffraction patterns of titanium in the initial state as well as after TE with one and three passes. In the recrystallized structure of the initial titanium (Figs. 3a, 3b), equiaxed grains with an average size of 20 μm and a small amount of nonmetallic inclusions are present in the longitudinal and transverse sections. Dislocation density inside the grains is low, and some grains in the longitudinal section contain deformation twins (Fig. 3b).

Analysis of the microstructure of titanium after TE shows that with increasing number of passes and amount of accumulated deformation, the degree of dimensional uniformity of structural elements (grains/subgrains) grows and their characteristic size d in the longitudinal and transverse sections of the speci-

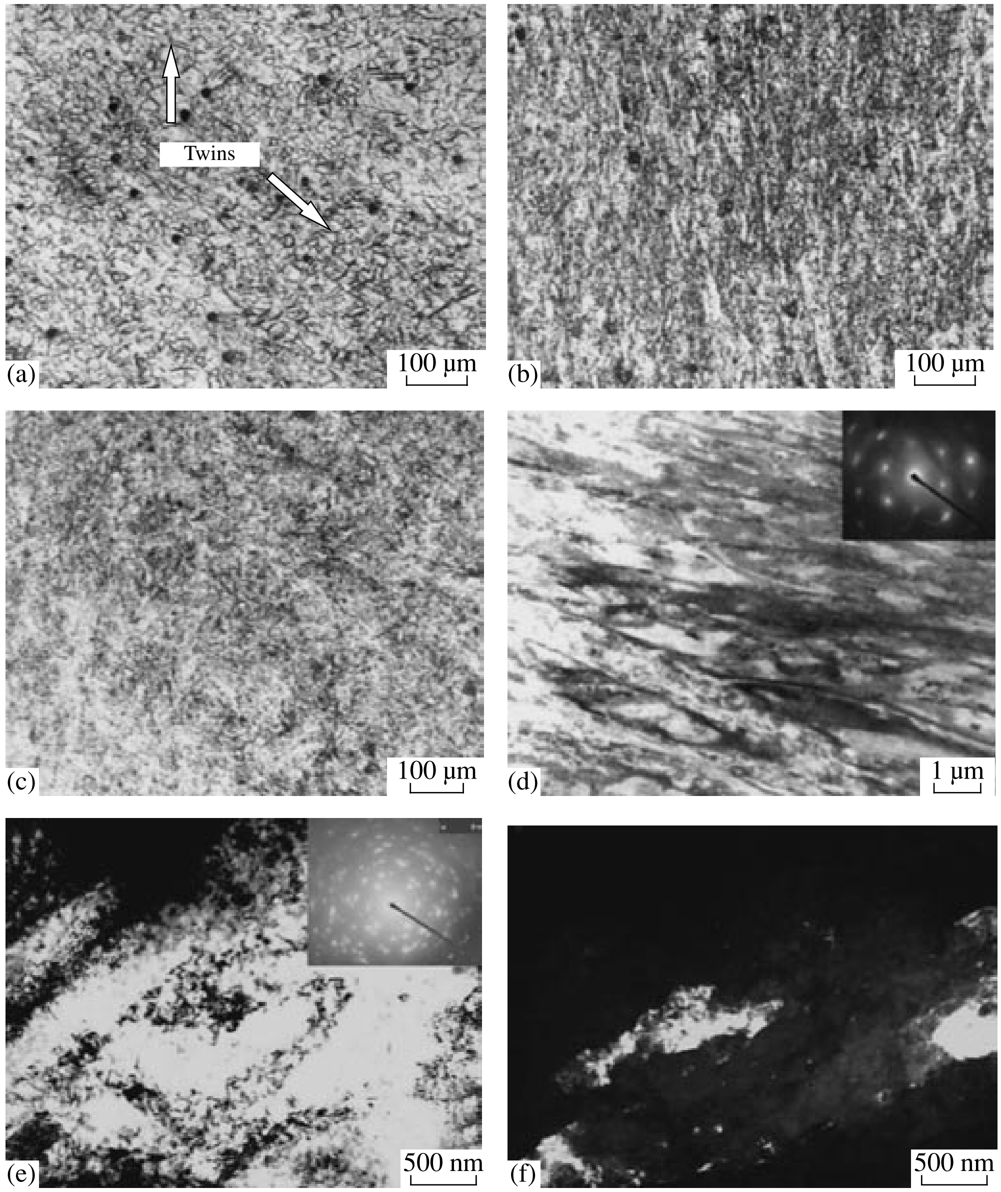

Fig. 4. Microstructures and selected-area diffraction patterns of titanium: (a) TE (one pass); (b–d) TE (three passes); (e) TE (three passes) and rolling; (a, b) transverse section; (c–f) longitudinal section; (e, f) bright and dark fields, respectively.

men decreases sharply. In the initial state, the average size is $d \approx 20$ μm (Fig. 3); after one and three passes, $d \leq 10$ μm (Fig. 4a) and 1 μm (Figs. 4b–4d), respectively.

In the transverse section of the specimen the structure elements are elongated in the radial direction, whereas in the longitudinal section the structure is of a vortical character. Apparently, this is connected with the fact that structural elements in the form of radially arranged "rollers" are formed upon severe alternating-sign shear. This problem calls for an additional investigation.

After TE with three passes a one-dimensional banded structure of the dislocation–disclination type arises in the longitudinal section [19]. Orientation of the bands corresponds to the tangential direction, and the maximum ratio of the length of the band to its width is 10 : 1 (Fig. 4d). The diffraction pattern corresponds to a single-crystal structure and confirms the absence of grained structure. The nonuniform diffraction contrast inside the bands and the diffuseness of the boundaries of structural elements indicate high internal stresses, which is characteristic of severely deformed materials. Note also that after one pass the number of twins that are typical of a coarse-grained titanium in the as-delivered state abruptly decreases (Fig. 4a), and after three passes they are not observed even at a high magnification (Fig. 4d). This may point to the fact that, instead of twinning, dislocation slip becomes the main operative

Table 1. Mechanical properties of titanium after TE, subsequent annealing, and cold rolling

State	Direction of tests relative to the extrusion axis	σ_u, MPa	$\sigma_{0.2}$, MPa	δ, %	H_V, MPa
As-delivered	Longitudinal	505	375	36	1800
	Transverse	500	360	46	1850
TE (three passes)	Longitudinal	505	463	31	2300
	Transverse	835	765	17	2800*
					2400**
TE (three passes) + 1-h annealing	Longitudinal	500	441	37.5	–
	Transverse	865	737	37.5	–
TE (three passes) + cold rolling (reduction 50%)	Longitudinal	780	750	30	2700
	Transverse	795	760	27	2750*
					3300**

* Butt end of plate.
** Plane of plate.

Table 2. Mechanical properties of titanium obtained by different SPD methods

SPD method	T_{SPD}, °C	Degree of deformation	P_{SPD}, GPa	Direction of tension	σ_u, MPa	$\sigma_{0.2}$, MPa
HPT [2]	20	5 rev., $\varepsilon = 5$	5	Transverse to the twist axis	950	790
TE	20	Three cycles, $\varepsilon = 3.45$	0.75	Transverse to the extrusion axis	835	765
				Along the extrusion axis	505	463
ECAP [3]	450–400	Eight cycles, $\varepsilon = 9.2$	1.2	Transverse to the pressing axis	805	765
				Along the pressing axis	710	640

mechanism of plastic deformation in the TE process at the stage of the formation of a UFG structure.

Cold rolling of titanium after TE results in an additional refinement of the alloy structure and the formation of a cellular substructure with an enhanced density of subboundaries as evidenced by a significant increase in the number of reflections and by the formation of concentric rings in the electron diffraction pattern (Fig. 4e) as compared to Fig. 4d.

Microhardness and Mechanical Properties

In order to determine the degree of structural anisotropy in different sections before TE, after TE, and after TE with subsequent rolling, we measured microhardness in the longitudinal and transverse directions relative to the extrusion axis (Table 1). It is seen that no anisotropy of microhardness is observed in the as-delivered state. The anisotropy manifests itself after TE with three passes and is absent after subsequent cold rolling. The anisotropy of microhardness and strength in this case is qualitatively coincident: microhardness in the longitudinal direction is smaller by 20% than in the transverse direction on the butt end of the plate. However, the microhardness in the other transverse direction in the plane of the plate is markedly smaller. Higher values of microhardness in the transverse direction on the butt end of the plate are associated with a greater degree of torsional deformation at the periphery than at the center of the sample.

In the initial state, as follows from Table 2, the strength properties in the longitudinal and transverse directions are virtually the same. This indicates the isotropy of mechanical properties and the absence of crystallographic texture in coarse-grained titanium, which corresponds to the type of structures shown in Fig. 3. A difference in the elongation to failure in both directions can be connected with the anisotropy of the shape of grains that exists in the initial titanium.

After severe plastic deformation by TE, a marked change in the mechanical properties of titanium also occurs along with a noticeable refinement of the microstructure. It is seen that TE leads to a selective enhancement of strength properties and to the appearance of a significant anisotropy of strength and plasticity that does not exist in the as-delivered state. In the longitudinal direction, for example, the ultimate tensile strength remained unaltered, the elongation to failure decreased slightly, and the yield strength increased by 25% as compared to the initial state. In such a situation, a postdeformation low-temperature annealing at 300°C for 1 h affected the strength but increased the elongation to failure. This is consistent with the known fact that such a regime of annealing results only in the relaxation of stresses without recrystallization-related growth of grains [3].

In the direction perpendicular to the workpiece axis, the ultimate strength and the yield strength of titanium increased to a greater extent: by 67 and 112%, respectively. The postdeformation low-temperature annealing led to insignificant (within the measurement error) changes in the strength properties and to an unexpectedly sharp enhancement of the plasticity. Thus, as a result of annealing, the elongation to failure δ in the transverse direction increased by a factor of more than two.

Structural data available to date are insufficient to draw conclusions regarding the factors that are responsible for such a selective change in the strength and ductility of titanium. One cause for an abrupt enhancement of the strength of deformed titanium in the transverse direction may be the formation of sharp crystallographic and metallographic texture and its dependence on the conditions of deformation and subsequent annealing. Thus, it was shown in [3] that in titanium subjected to ECAP there forms a texture in which normals to the basal planes are predominantly located in the direction transverse to the pressing axis and the ultimate strength and the yield strength in this direction are higher than in the longitudinal direction.

It is also undoubtful that the alternating-sign nature of deformation and the simple mode of shear along the planes orthogonal to the workpiece axis make their contributions to the formation of structure and mechanical properties. Indeed, according to [11, Ch. 1], the alternating-sign torsion of a sample only slightly affects the yield strength along the torsion axis. At the same time, an intense simple shear at the insufficiently high level of hydrostatic pressure may give rise to the formation of vacancies and layers of incipient micropores that reduce plasticity in the direction orthogonal to the shear plane. The role of postdeformation annealing in this case is in the elimination of defects and enhancement of plasticity.

It is of interest to compare mechanical properties of titanium processed by different SPD methods (TE, ECAP [3], and HPT [2]) which are based on the deformation scheme of simple shear (see Table 2). This comparison shows that the most high-strength state of titanium is formed in the process of deformation by HPT, which is connected with a stronger grain refinement (to 80 nm). The level of strength properties of titanium obtained by the methods of ECAP and TE is appreciably lower. They are very close in the transverse direction (are equal to 800–835 and 765 MPa for the ultimate strength and the yield strength, respectively). At the same time, the degree of anisotropy in titanium produced by ECAP is considerably smaller than in titanium produced by TE. This is associated with the location of the plane of intense shear upon ECAP (with a right angle between channels) at an angle close to 45° to the workpiece axis. Unfortunately, it is impossible to correctly compare plasticity characteristics because of different dimensions of the samples tested.

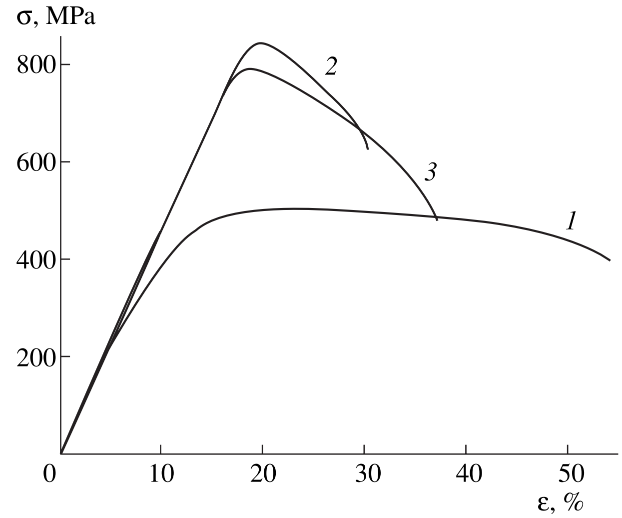

Fig. 5. Engineering stress–strain curves for titanium (*1*) in the as-delivered state, (*2*) after TE, and (*3*) after TE + rolling; transverse direction.

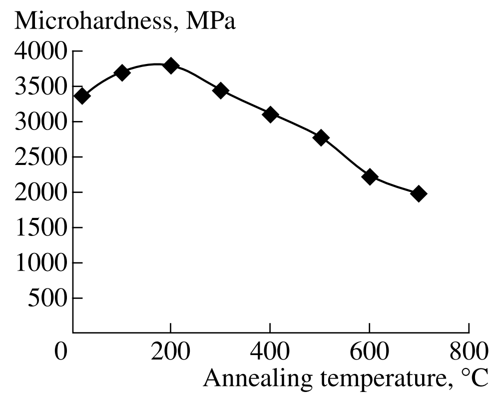

Fig. 6. Dependence of the microhardness of titanium after three passes of twist extrusion and cold rolling to 50% on the annealing temperature at an annealing time of 1 h.

It follows from the analysis performed in [11] that the mechanical properties of metals after TE can be improved by using a comparatively small additional deformation of workpieces differing from torsion about their axes. First, this may lead to an intense strengthening of the material. Second, this may result in a partial healing of incipient micropores and in an enhancement of plasticity (especially if the additional deformation occurs at a sufficiently high level of hydrostatic pressure). The results of tensile tests of the specimens of VT1-0 titanium after TE and subsequent cold rolling confirm this supposition.

It is seen from Table 2 that cold rolling after deformation by TE markedly increases strength characteristics in the longitudinal direction, making them be closer to those in the transverse direction. It is important to note that elongation in the transverse direction likewise increases strongly due to cold rolling and reaches that observed in the longitudinal direction. As a result, the anisotropy of the strength and plasticity characteristics virtually disappears as compared to the state before rolling, which indicates a high potential of the chosen combination of deformation treatment.

Figure 5 displays engineering stress–strain tensile curves of titanium in the direction transverse to the extrusion axis in the initial and deformed states. The basic distinction of the mechanical behavior of titanium in the state after TE (curve *2*) is a shortening of the stage of strain hardening and, correspondingly, a decrease in the uniform elongation as compared to the as-delivered state (curve *1*). In titanium after TE, elongation in the specimen neck is dominant in comparison with the uniform elongation, which reduces the total elongation to failure (see Table 1). This fact is related to specific features of the dislocation mechanism of deformation in UFG materials and is typical of many metals obtained by SPD [1]. However, additional rolling of titanium after TE favors the enhancement of the uniform and total elongations and is accompanied by only a weak softening. In this case, apparently, the determining factors are not only the grain size but also the crystallographic texture, which changes upon the rolling. The enhancement of the texture upon rolling is indirectly evidenced by a sharp increase in the microhardness (from 2400 to 3300 MPa) measured in the plane of the plate in the direction of its thickness (see Table 1).

Thermal Stability

Figure 6 shows the variation of the microhardness of titanium after TE and cold rolling as a function of the annealing temperature. It is seen that with a rise in the annealing temperature to 200°C, the microhardness increases and reaches a maximum value. With a further increase in the annealing temperature, the microhardness gradually decreases to values close to those for the initial undeformed state. The dependence obtained suggests that the structural state of deformed titanium remains thermally stable to temperatures of 300–350°C upon holding for 1 h.

Note that the behavior of the curve obtained differs from the typical temperature dependence upon heating for deformed pure metals characterized by the absence of the stage of strengthening. The existence of strengthening can indicate the process of artificial aging, which is possible in a commercially pure titanium because of the presence of an appreciable amount of impurities in it. In the course of TE and rolling, some interstitial and substitutional impurities may dissolve in α titanium, which was already observed in the VT1-0 samples subjected to SPD by torsion [17]. Subsequent heating to relatively low temperatures (200°C) is able to cause the decomposition of solid solution and precipitation of strengthening dispersed particles. A further elevation of the annealing temperature to 500°C results in relaxation of elastic stresses, coarsening of precipitated particles, and a weak growth of grains, which brings about a decrease in the microhardness, whose level, however, remains high enough. A noticeable drop in the microhardness occurs at temperatures above 500°C, when the processes of recrystallization-induced grain growth become dominant.

CONCLUSIONS

(1) The effect of a combined treatment (severe plastic deformation by twist-extrusion and subsequent cold rolling) on the structure, mechanical properties, and thermal stability of massive workpieces of VT1-0 titanium was studied. Twist extrusion causes a refinement of structural fragments to submicron sizes, an enhancement of strength characteristics, and the appearance of a significant anisotropy of strength properties in the longitudinal and transverse directions of the samples.

(2) Subsequent cold deformation, for example, rolling, can serve as an efficient tool for the elimination of the anisotropy of mechanical properties. The commercially pure titanium that was deformed using cold twist extrusion and rolling manifests an aging effect upon heating. The structural state of titanium after combined treatment is thermally stable to temperatures of 300–350°C.

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