Mechanical Properties, Density, and Defect Structure of VT1-0 Titanium after Intense Plastic Deformation Due to Screw and Longitudinal Rollings

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Abstract—The influence of screw rolling combined with standard methods of mechanothermal treatment on the homogeneity of the forming submicrocrystalline structure, density, and mechanical performance of VT1-0 commercial titanium is studied. It is shown that such a treatment carried out within optimal temperature and strain rate intervals (special deformation conditions) causes minor softening of the material and can be effectively used to form a homogeneous submicrocrystalline structure with high strength and elastoplastic properties.

Much interest has been recently shown in submicrocrystalline and nanocrystalline metals and alloys. This is because such materials offer unique physicomechanical properties, which are much superior to the same properties of coarse-grained polycrystals [1–6].

Intense plastic deformation (IPD) is viewed as a promising way for producing metallic materials in submicrocrystalline and nanocrystalline states. Among related methods, the method of equal-channel angular pressing (ECAP) is most popular. However, using this method, it is difficult to produce a wide range of industrial products (sheets, slabs, rods with a given diameter, etc.). There are also other disadvantages of this method in some cases [4].

In this work, we suggest another way of producing a stable submicrocrystalline structure: IPD by screw rolling in combination with longitudinal rolling under different conditions (standard methods of mechanothermal treatment in optimal temperature and strain rate intervals [5]).

The main objective of this work was to see how the above combined way of IPD influences the mechanical properties, density, and defect structure of a metallic material carried to a submicrocrystalline state.

EXPERIMENT

VT1-0 commercial titanium was chosen as a metallic material. Such a choice was due to the fact that titanium and titanium alloys have a wide range of application from aerospace engineering to biomedicine (the biomedical application is of special importance [6]).

As-prepared titanium had the form of rods 40 mm in diameter with a grain mean size of 22 μm. The rods were treated under different conditions with variable degrees of straining. Two conditions turned out to be optimal in regard to structural and mechanical properties. In the first case (conditions 1), the rod was first thinned to a diameter of 16 mm by radial-displacement rolling at 400°C, then was thinned further to a diameter of 12 mm by longitudinal rolling also at 400°C, and finally was subjected to helical rolling at room temperature to a diameter of 8 mm. The second optimal treatment conditions (conditions 2) were the following: radial-displacement rolling at 400°C to a diameter of 20 mm, then longitudinal rolling at 400°C to 9.5 mm, and finally room-temperature helical rolling to 8 mm (for the detailed temperature–rate conditions of rollings, see [5]).

Note that a disadvantage of screw rolling is "loosening" of metallic materials in their central parts at a "critical" degree of rolling [7]. We observed such a case after radial-displacement rolling at 400°C to a diameter of 16 mm followed by helical rolling at room temperature to 8 mm (hereinafter, such a regime will be referred to as the third critical regime).

To relieve internal stresses and improve the stability of the structure, titanium samples were annealed at 350°C for 3 h.

The structure was examined under a Qwanta 600 FEC scanning electron microscope equipped with a

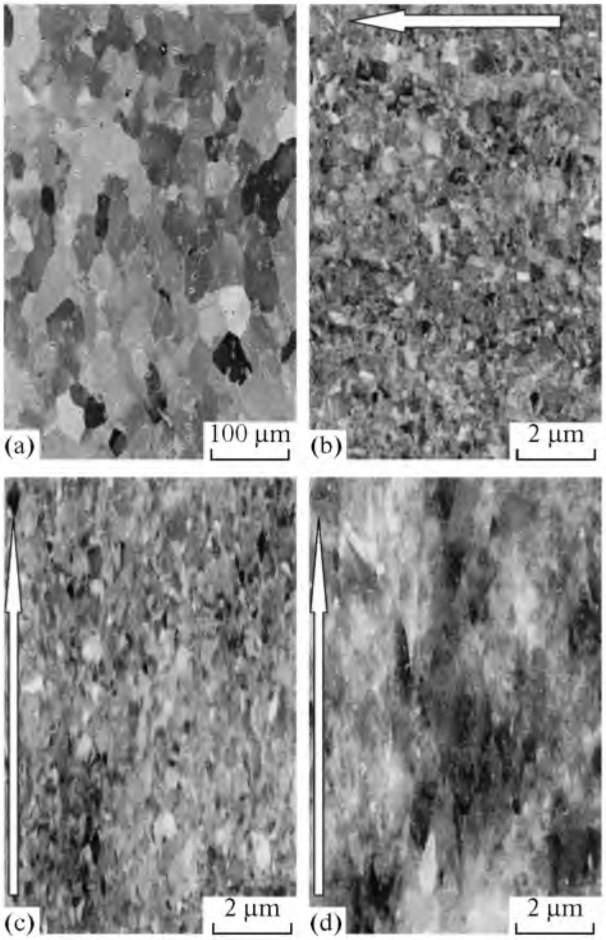

Fig. 1. Microstructure of VT1-0 titanium (a) in the as-prepared (initial) state and after rolling under (b) conditions 1, (c) conditions 2, and (d) critical conditions.

field emission gun. The strength and ductility of the samples were determined with an Instron 5882 machine at room temperature. The density of the titanium samples was measured by the precision method of hydrostatic weighing in air and in distilled water accurate to 0.5%.

Acoustic measurements were used to determine quantities characterizing elastic (Young's modulus E) and reversible microplastic deformations (amplitude-independent damping ratio δ and microplastic flow stress σ), which are related to the vibration motion of dislocations. The feature of the acoustic experiments is that the dislocation structure of the samples does not change at moderate amplitudes: upon an acoustic action on a metal, the dislocation density remains unchanged [8]. Specimens for acoustic measurements were prepared from a work piece made under the two rolling conditions mentioned above. They were 24 mm long rectangular rods with a cross-sectional area of 3.0×0.6 mm. With such a length, resonance frequency f of longitudinal vibrations of the specimen was near 100 kHz. The Young's modulus was found by the formula $E = 4\rho l^2 f^2$, where ρ is the density of the specimen.

The resonance method of a composite vibrator (for details, see [8]) was applied, which not only provides information on the Young's modulus but also makes it possible to study ultrasound absorption (internal friction) and inelastic (microplastic) properties of specimens. The inelastic properties can be estimated by measuring modulus E and elastic vibration damping ratio δ in a wide range of vibrational deformation amplitude ε. At sufficiently large ε, nonlinear amplitude-dependent absorption, $\delta_h = \delta - \delta_i$, and an amplitude-dependent defect of the Young's modulus, $(\Delta E/E)_h = (E - E_i)/E_i$ (E_i and δ_i are the Young's modulus and damping ratio at amplitudes so small that both parameters are still ε-independent) arise.

Acoustic measurements in a wide amplitude range allow researchers to estimate the mechanical (microplastic) properties of materials in stress–inelastic strain coordinates (vibrational stress amplitude $\sigma = E\varepsilon$ is plotted on the vertical axis, and nonlinear inelastic strain $\varepsilon_d = \varepsilon(\Delta E/E)_h$ is plotted on the abscissa axis), which are commonly used in mechanical tests.

The parameters of electron density nonuniformities, which might be induced by IPD, were determined by the modified method of small-angle X-ray scattering (SAXS) [9]. It was shown earlier that IPD due to ECAP results in the formation of nanopores in aluminum [4, 10] and titanium [11]. The method of small-angle scattering was employed before and after the application of a high hydrostatic pressure (which effectively "heals up" porosity-related nonuniformities [4, 10, 12, 13]) to identify nonuniformities, which might arise under different conditions of screw and longitudinal rolling.

RESULTS AND DISCUSSION

The typical microstructure of VT1-0 titanium in the initial (recrystallized) state and upon IPD in two optimal and one critical regimes are shown in Figs. 1–4. Electron microscopy data allowed us to draw the following conclusions. Specimens prepared under conditions 1 (hereinafter, specimens 1) have a grain/subgrain mean size of ≈0.3 μm; however, they also contain coarser grains 0.7–0.8 μm across. The finest grains/subgrains, ≈0.2 μm, were observed in specimens obtained under conditions 2 (specimens 2). The structure of these specimens is uniform, and they are almost free of large grains. The specimens prepared under critical conditions 3 (specimens 3) exhibit a nonuniform structure with a mean grain/subgrain size of 1.2 μm. Specimens 2 have globular grains, unlike specimens 1, which have large grains 0.7–0.8 μm across extended along the rolling direction with a mean size of structural elements of ≈0.3 μm. Specimens 3 have the most nonuniform grain structure apparently because of the highest degree of helical rolling.

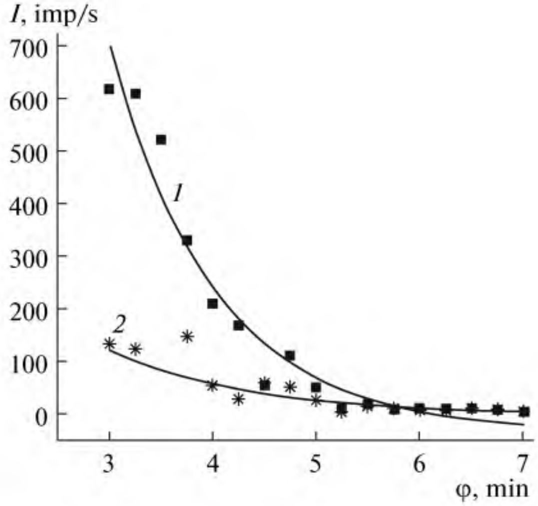

Fig. 2. X-ray scattering intensity I vs. angle φ for the titanium specimens deformed under the critical conditions (*1*) before and (*2*) after the application of a hydrostatic pressure of 1.5 GPa.

The density was systematically measured on no less than 15 initial recrystallized titanium specimens and deformed specimens 1–3. Importantly, the specimens were cut from different parts of the rod subjected to complex rolling. On each specimen, the density was measured no less than twice. Certainly, special attention was paid to specimens obtained under conditions 1 and 2 of intense rolling deformation.

It was found that the density of the rolled VT1-0 titanium decreases upon rolling, the densities of specimens 1 and 2 markedly differing. The densities of the initial recrystallized specimen, specimen 1, and specimen 2 were $\rho = 4.551 \pm 0.006$, 4.508 ± 0.005, and 4.548 ± 0.001 g/cm^3, respectively. It is significant that specimens cut from different parts of the rod had nearly the same densities within the measurement accuracy for both deformation conditions. One can therefore conclude that the density of specimens 1 and 2 is distributed uniformly. This is especially true for specimens 2 with regard to the spread in density (see above).

Another situation is observed for specimens cut from different parts of the rod rolled under the critical conditions: here, the density varies from 4.481 to 4.525–4.550 g/cm^3.

As has been mentioned above, electron density nonuniformities were studied by the SAXS method and the origin of these nonuniformities was determined by comparing SAXS data for the samples before and after the application of a high hydrostatic pressure. Typical data for one of the titanium deformation conditions are shown in Fig. 2. It is seen that the pressure leads to a considerable decrease in the scattering intensity. The processing of the difference curves according to [9] showed that low-density nonuniformities (regions), which can be viewed as regions with an excess free volume, are roughly ≈20 nm in size.

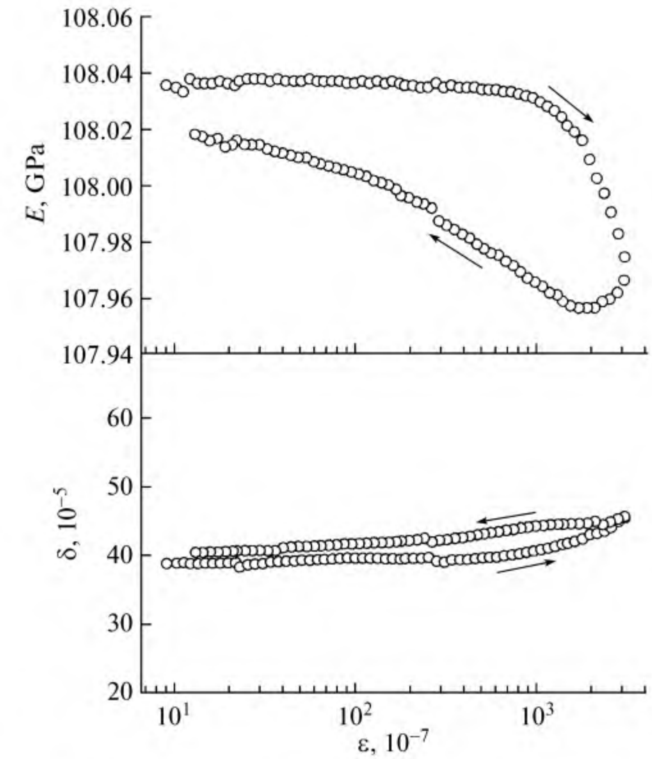

Fig. 3. Amplitude dependences of Young's modulus E and damping ratio δ for the titanium specimens in the initial state that were measured for successively increasing and decreasing amplitude ε (arrows indicate the sign of ε variation). $T = 287$ K.

In specimens 1, the integral volume of low-density regions is estimated as $\approx 9 \times 10^{-3}$, which is close to an excess volume arising in ECAP-deformed aluminum ($\approx 5 \times 10^{-3}$ [10]). In specimens 3 (critical deformation conditions), softening due to deformation-induced porosity may be twice as high (see the table). The softening and, as follows from the SAXS data, the integral volume of low-density regions are minimal in specimens 2. Here, the integral volume is 15 times smaller than in titanium specimens rolled under conditions 1 and also in aluminum and its alloy specimens subjected to ECAP [10].

Consider now the results of mechanical tests. Data for the strength and ductility of initial titanium specimens and specimens rolled under conditions 1–3 are listed in the table. It is seen that titanium specimens 2 offer the highest strength at a high enough ductility.

To conclude, we turn to the elastoplastic properties of the titanium specimens. Figures 3–5 show the amplitude dependences of Young's modulus E and damping ratio δ for initial titanium and titanium specimens subjected to IPD (rolling) under conditions 1 and 2. The curves $E(\delta)$ and $\delta(\varepsilon)$ were taken by successively increasing and decreasing the amplitude. All the

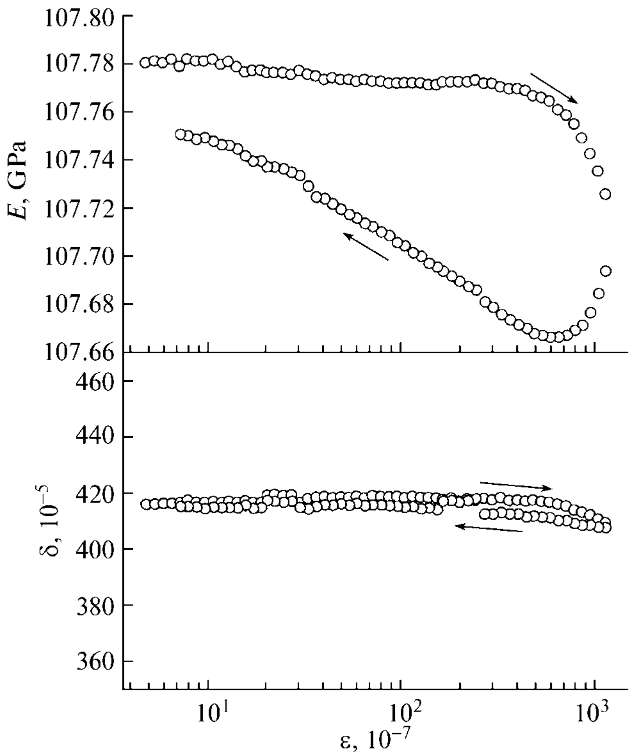

Fig. 4. Amplitude dependences of Young's modulus E and damping ratio δ for the titanium specimens deformed under conditions 1 that were measured for successively increasing and decreasing amplitude ε (arrows indicate the sign of ε variation). $T = 287$ K.

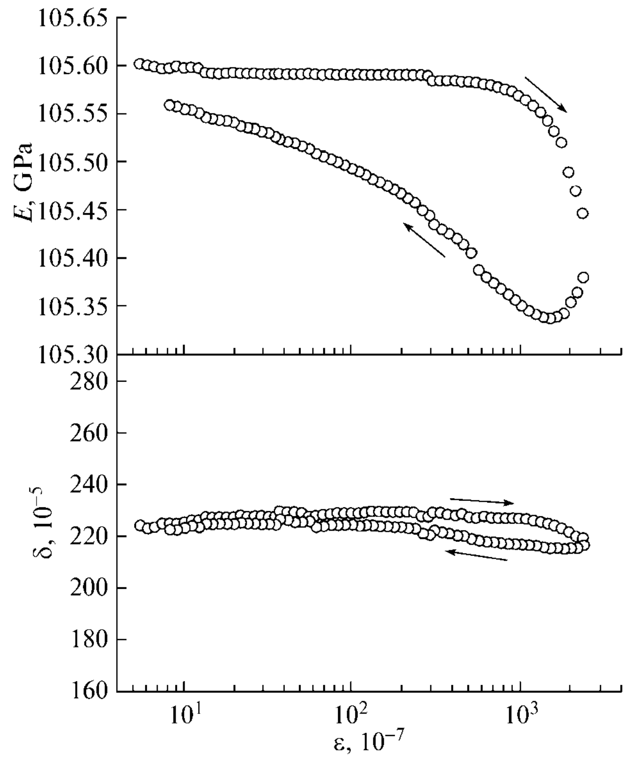

Fig. 5. Amplitude dependences of Young's modulus E and damping ratio δ for the titanium specimens deformed under conditions 2 that were measured for successively increasing and decreasing amplitude ε (arrows indicate the sign of ε variation). $T = 287$ K.

curves for the Young's modulus exhibit a considerable amplitude hysteresis: when taken with the amplitude increasing and decreasing, they do not coincide. In all cases, the shape of hysteresis and the values of E and δ were different. From the curves $E(\varepsilon)$ in Figs. 3–5 that were taken for the increasing amplitude, we constructed acoustic stress–strain diagrams $\sigma(\varepsilon_d)$. The diagrams for the initial titanium and for titanium specimens 1 and 2 are seen to differ markedly. From these diagrams, conventional micro-yield strength $\sigma = \sigma_s$ for inelastic strain ε_d can be estimated at a level of 1×10^{-7} (see the table).

Let us analyze the data for the elastoplastic properties, i.e., the values of E, δ, and σ_s (see the table).

From these data it follows that the formation of an IPD-induced submicrocrystalline structure leads to a decrease in E and a considerable increase in δ. This finding is consistent with theoretical concepts. The elasticity modulus and damping ratio, which should be measured with a high accuracy, are the structure-sensitive parameters of a material. In terms of the theory of interaction between point defects and dislocations [8, 14], the latter decrease the elasticity modulus and increase the damping ratio of a specimen. The elastic-

Mechanical properties and titanium density defect versus the titanium state

State	Relative softening, $\Delta\rho/\rho$	Ultimate tensile strength, MPa	Elongation at rupture, %	E, GPa	$\delta \times 10^{-5}$	σ_s, MPa
Initial	–	460	34	108.036	39	26
After deformation under conditions 1	9×10^{-3}	910	13	105.601	225	16
After deformation under conditions 2	6×10^{-4}	930	16	107.78	416	12
After deformation under critical conditions	$\sim 10^{-1}$–10^{-2}	650	10	~105.1	210	~16

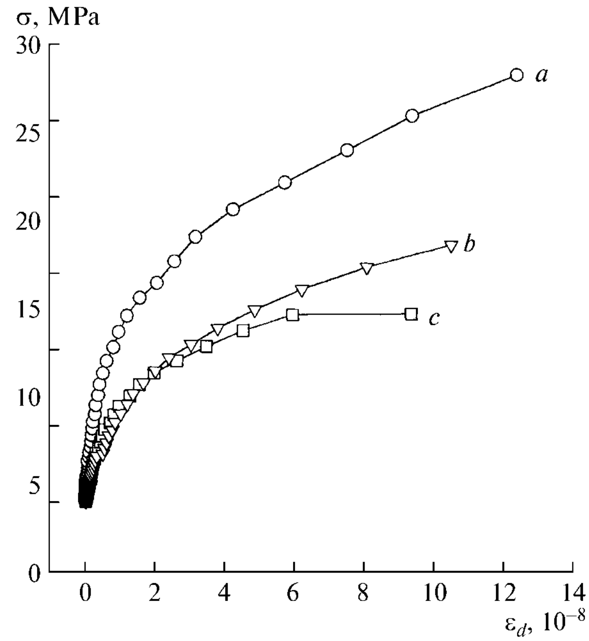

Fig. 6. Stress–microplastic strain curves constructed from the acoustic measurements of the titanium specimens (*a*) in the initial state and deformed under conditions (*b*) 1 and (*c*) 2.

ity modulus also decreases when low-density regions like nanopores arise in a material [15]. From this point of view, the lower value of the elasticity modulus in specimens 1 compared with the elasticity modulus in specimens 2 (see the table) may be associated with a high concentration of low-density regions, which arise upon deformation under conditions 2 (as has been shown above). In titanium specimens 3 (critical deformation conditions), the nanoporosity-related decrease in the elasticity modulus is the most pronounced (see the table).

Amplitude-independent damping ratio δ varies in proportion to the dislocation density [8, 14]. IPD under rolling certainly leads to a considerable rise in the dislocation density, which is reflected on δ. Its value grows from five- to tenfold (see the table). Upon deformation under conditions 2, δ is twice as large as upon deformation under conditions 1. This may be because grain boundaries also contribute to δ: the finer the grains, the larger their surface area and the higher the value of internal friction. In addition, it is likely that the dislocation density in the specimens deformed under conditions 2 is somewhat higher than in those deformed under conditions 1.

When analyzing data for the conventional yield strength, one should bear in mind the following. According to today's concepts, the nonlinear inelastic microplastic strain ε_d is given by $\varepsilon_d = b\lambda_d\psi$, where b is the Burgers vector, λ_d is the density of dislocations responsible for straining, and ψ is the mean displacement of these dislocations from the equilibrium position [8]. Obviously, ε_d depends on external stress σ and dislocation total density λ: the higher λ, the lower σ for strain ε_d. For this reason, the stress in the initial titanium specimens was higher than in specimens 1 and 2.

As has been noted, the dislocation density in specimens 2 seems to be the highest, since the conventional micro-yield strength in them is the lowest (see the table). This finding is consistent with the data for internal friction, as follows from the table. It should be noted that a low level of stress causing microplastic deformation means that the material is easily adapted to variations in the environment. Eventually, this may add to the strength of the material, as was demonstrated for microcrystalline aluminum [16].

CONCLUSIONS

Our experimental data show that one can select optimal conditions for screw and longitudinal rolling of commercial titanium. These conditions provide a uniform submicrocrystalline structure with high strength and elastoplastic properties. With regard to the temperatures of rolling and final annealing, it can be argued that the resulting structure is thermally stable. This fact, as well as a wide range of products that can be obtained by intense plastic deformation during screw and longitudinal rollings, highlights the undeniable advantages of this approach to obtaining a submicrocrystalline structure. One more important factor providing high mechanical properties upon screw rolling under optimal conditions is the almost complete absence of low-density regions, which may result from equal-channel angular pressing and have an adverse effect on the properties of nanocrystalline and submicrocrystalline metals and alloys [4].

ACKNOWLEDGMENTS

This work was supported by the Russian Foundation for Basic Research, project no. 09-02-00596-a.

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