Elastic properties of Ti and its alloys nanostructured due to severe plastic deformation

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Abstract. Studies of elastic properties of titanium and its alloys processed by severe plastic deformation are presented. The evolution of the modulus of elasticity resulting from the transformation from a coarse-grained state to an ultrafine-grained one due to severe plastic deformation (SPD) was investigated. The acoustic composite oscillator technique was used to measure the elastic modulus in a wide strain amplitude range. The microstructure was studied in detail by scanning electron microscopy and electron backscatter diffraction methods. Parameters of nanoscale porosity were measured by small-angle X-ray scattering before and after treatment with a high hydrostatic pressure (1.5 GPa). In addition, densities of titanium and its alloys in various structural states were determined by the precision method of hydrostatic weighing. As experiments showed, noticeable changes in the elastic properties resulting from the change in the grain state can be attributed to several factors, such as dislocations, nanoporosity, high internal stresses, and the structure of the materials before SPD processing.

1. Introduction
Practical use of nanostructured materials obtained by severe plastic deformation (SPD) is due to their enhanced mechanical properties as compared to coarse grained polycrystals [1-3]. Of special interest among metallic materials is titanium which can be used in medicine for implants. For this application titanium of high strength with a low modulus of elasticity close to that of bones is required [4-6]. Elastic properties of nanostructured Ti are of great importance but they have been insufficiently studied up to now.

2. Methods and materials
Young’s modulus $E$ and logarithmic decrement $\delta$ (internal friction) of titanium were measured on rectangular rod samples (before and after SPD) in a wide range of vibrational strains $\varepsilon$ by the acoustic composite oscillator technique [7]. We used moderate amplitudes to keep the dislocation structure (density) unchanged after the application of an acoustic load to the sample. The acoustic system (a composite oscillator consisting of a piezoquartz and the sample) was adjusted in resonance at a certain exciting voltage frequency. So we could determine the decrement $\delta$ and natural resonance frequency $f$ of the sample. The Young’s modulus $E$ was calculated as $E = 4\rho f^2$. Here, $\rho$ and $f$ are the sample density and length, respectively.

In order to estimate the integral volume and sizes of nanopores arising due to SPD, the upgraded small-angle X-ray scattering technique with Kratky collimation (MoKα-radiation) and high-precision density measurements (an error less than $2 \times 10^{-4}$) were used. The measurements were carried out before
and after the high hydrostatic pressure (1.5 GPa) processing. It has been found that the use of hydrostatic pressure is an effective way of reducing the excess free volume in the material [8, 9]. The scattering components that are not related to the pores have been shown to remain unchanged after the pressure application to the sample. Thus, it was possible to reveal the X-ray scattering sources and determine the parameters of nanoporosity generated in metals and alloys by SPD. 

The structural investigations were carried out by optical, scanning and transmission electron microscopies. 

A lot of SPD methods were used to obtain various structural characteristics of titanium and its alloys. The samples were investigated after an equal-channel angular pressing [10] and various modes of transverse screw and longitudinal rolling [11].

We used in our experiments two sets of VT1-0 α-titanium. The first one had lamellar grains with sizes of ~150×10 μm in the initial coarse grained state. Another one had a more equiaxed coarse grained structure where grains had initial sizes of ~22 μm or ~40–50 μm. Any of the grain structures was transformed into an almost equiaxed grain state (~250×290 nm) after the SPD processing.

Another set of α-titanium (Grade 4) had an almost equiaxed structure with the mean grain size of about 40 μm in the initial state. Equiaxed grains ~250 nm in size were observed after SPD. A β-titanium alloy (Ti-26Nb-7Mo-12Zr) was also studied after rolling which created plastic strains from 30 to 90% [12].

As an example, Figure 1 shows the transformation of the VT1-0 titanium from the lamellar coarse-grained structure (a) into the ultrafine-grained (b, c) state.

Table 1 lists the materials that have been studied, the grain sizes of the samples, and the SPD methods used to obtain the ultrafine-grained (UFG) structure.

![Figure 1](image)

Figure 1. Microstructure of VT1-0 alloy in the initial coarse-grained (a) and UFG states - longitudinal (b) and cross sections (c).

3. Results and discussion

The main goal of our study was to reveal the factors that affect the evolution of the Young’s modulus during the transition from the coarse-grained to the ultrafine-grained state due to SPD.

According to the internal friction theory that describes the interaction between dislocations and point defects, the Young’s modulus decreases and the decrement increases due to plastic deformation as a result of a high fresh dislocation density [7, 13]. Figure 2 shows the $E(\varepsilon)$ and $\delta(\varepsilon)$ dependences measured for increasing and then decreasing amplitude $\varepsilon$ for the VT1-0 titanium in the initial coarse-grained state (1) and UFG state (2) after SPD. It can be seen that the Young’s modulus is lower and the decrement is higher for the UFG sample as compared to the same sample in its initial state. This is in a good qualitative agreement with the theory.
Table 1. The materials, the SPD methods, and the grain sizes of the samples.

| Material          | SPD                        | Grain size |
|-------------------|----------------------------|------------|
| VT1-0             | ECAP (8 passes)            | 40, 0.25   |
| VT1-0 [11]        | RDR→SR→LR                 | 22, 0.2    |
|                   | RDR→LR                    | 1.2        |
| VT1-0 [14]        | RDR→SR→LR                 | 150×10, 0.25 |
| Grade-4 [14]      | RDR→SR→LR                 | 40, 0.25   |
| Ti-26Nb-7Mo-12Zr  | Deformation by rolling 30% | 280, 20.5  |
|                   | Deformation by rolling 60% | 10.1       |
|                   | Deformation by rolling 90% | 9.1        |

a Radial-displacement rolling.
b Screw rolling.
c Longitudinal rolling.

Figure 2. Amplitude dependences of Young’s modulus $E$ and decrement $\delta$ for VT1-0 titanium: (1) initial coarse grained state, (2) UFG state after SPD; equiaxed (a) and lamellar (b) initial grain structure; arrows indicate the direction in $\varepsilon$ changes.

Figure 2 (a, b) presents experimental data for two sets of VT1-0 titanium samples that have the equiaxed (a) and lamellar (b) initial grain structures. It is evident that the decrease in the Young’s modulus for the lamellar Ti is almost an order of magnitude greater than for the equiaxed one. A low effect (1-3%) of SPD for the equiaxed initial structure was confirmed for another set of Ti, i.e., Grade-4 [14]. This means that initial structures are of great importance for the elastic properties of titanium subjected to the SPD processing. A considerable decrease in $E$ (~20%) for the lamellar initial structure...
may be explained [14] by the texture that is realized due to a prevailing crystallographic orientation of grains after SPD.

It is known that any kind of flaws, such as pores and micro-cracks, leads to a lower Young’s modulus [15, 16]. Table 2 contains the data on density, Young’s modulus, and decrement for the VT1-0 titanium measured before (initial state) and after the SPD processing by three types of rolling which differ by temperatures and rolling rates from each other [11]. The data demonstrate that the density becomes lower after the rolling (the third type of rolling produced inhomogeneous densities along the rod sample). Low-angle X-ray scattering investigations showed that the SPD processing by rolling resulted in the formation of nanopores with the average size of ~20 nm. It can be seen from Table 2 that the UFG structure is characterized by a lower E, which may be due to both a high dislocation density and different quantities of nanopores in the metal that result from various types of rolling. Figure 3 shows that there is an exponential relation between the porosity (changes in density) and the Young’s modulus.

**Table 2.** The data on density (ρ), Young’s modulus (E), decrement (δ) and grain sizes for the VT1-0 titanium.

| State   | ρ, g/cm³ | E, GPa | δ·10⁻⁵ | Grain size, μm |
|---------|----------|--------|---------|----------------|
| CG      | 4.551    | 108.036| 39      | 22             |
| UFG-1   | 4.508    | 105.601| 225     | 0.3            |
| UFG-2   | 4.548    | 107.78 | 416     | 0.2            |
| UFG-3   | ~4.481+4.525 | ~105.1 | 210     | 1.2            |

**Figure 3.** Young’s modulus E of Ti as a function of density variation Δρ/ρ due to SPD.

**Figure 4.** Young’s modulus E of Ti–26Nb–7Mo–12Zr alloy as a function of SPD by rolling.

The amplitude-independent decrement δ measured at low ε is proportional to the dislocation density [13]. It is evident that rolling leads to a higher dislocation density, which leads to higher values of δ (Table 2). In addition, the decrement depends on grain dimensions: the smaller the grains the larger the area of the grain boundaries and the higher the internal friction. In all probability, the highest δ of the samples prepared by the second type of rolling (UFG-2 in Table 2) may be due to the lowest grain size (~0.2 μm).
It was found that SPD could result in a lower and higher Young’s modulus. This effect was revealed when $E$ was investigated as a function of strain in the SPD experiments. Figure 4 shows the data obtained for the β-titanium alloy Ti-26Nb-7Mo-12Zr. One can see that the 30% strain decreases $E$, but a further increase in the strains results in higher $E$. One can explain this effect in the following way. Besides the dislocation density, the Young’s modulus measured in the experiments may be affected by long-range internal stresses [17], which lead to higher values of $E$. High internal stresses in the sample can be caused by both different crystallographic orientations of grains and different structures and states of grain boundaries [18-20]. Structural investigations by electron backscatter diffraction (EBSD) showed that the deformation increase from 30 up to 90% led to a large area of grain boundaries of a high misorientation ($\theta$>15°). According to the EBSD, the high-angle grain boundary fraction of 37, 78 and 82% corresponded to the strains of 30, 60 and 90%, respectively.

4. Conclusions
Titanium and its alloys were studied in the coarse-grained and ultrafine-grained nano-structural states. The most important results obtained are as follows:
1. The decrease in the modulus of elasticity after SPD may be due to an increase in the dislocation density, as well as to the presence of nanoscale porosity.
2. The increase in the modulus of elasticity with increasing deformation can be explained only by the development of high long-range internal stresses. It is clearly seen for β-Ti alloy.
3. It is shown that the initial structure of the metal determines to a large extent its elastic properties after SPD. It is shown for VT1-0 α-Ti.

The data obtained in our study may have a practical use: they show an additional possibility to get desired mechanical (elastic and plastic) properties of UFG metals

Acknowledgments
The study was funded by RFBR, project 18-08-00360.

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