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VISUALIZATION AND CONTROL THE INTERNAL STRUCTURE NANOCRYSTALLIC TITANIUM AND ARTICLES THEREOF

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At present more attention is given to development of the methods of severe plastic deformation providing formation of submicrocrystalline or nanostructured states in the bulk as well as the investigation of structure and properties of such materials. Today a set of the methods of severe plastic deformation have been developed such as high pressure torsion, equal channel angular pressing, multiple uniaxial pressing, forging (abc-pressing) and so on [1-8]. There are a lot of experimental data illustrating a great improvement of mechanical characteristics such as tensile ultimate strength, tensile yield strength, fatigue strength, wear resistance, microhardness and so on as a result of the severe plastic deformation [3-6]. Many researchers give particular attention to commercial pure titanium because of developing technologies of the nanostructured state formation discover a new prospect for its medical application.

A well-known method of severe plastic deformation [7, 8] has been modified to obtain the bulk nanostructured titanium and provides its high mechanical properties. The commercial pure coarse-grained titanium (99,58 Ti, 0,12 O, 0.18 Fe, 0,07 C, 0,04 N, 0,01 H wt. %) was used as an initial material. At the first stage the titanium billets were subjected to the multiple uniaxial pressing by using press-mold at 10^{-3}-10^{-2} s^{-1}speed range under stepwise temperature decreasing from 1023 K to 623 K. Uniaxial pressing at a fixed temperature was carried out along three deformation axis gradually. At the next stage the plastic deformation was applied with grooved or flat rollers at room temperature to get further grain refinement and to reach the nanostructured state. The total value of rolling deformation was 90%. Then the rolled sticks and plates were subjected to the prior-to-recrystalline annealing at 523 K.

The TEM studies have shown that the multiple uniaxial pressing allows formation of nanostructured state in the titanium billets already at the first stage, but it is heterogeneous in the longitudinal and the transverse sections. The grains and the subgrains have not equiaxial and elongated shape. The average size of grains and the subgrains is 90 nm in the transversal section (Fig. 1a). However, in longitudinal section the average size of grains and subgrains is much higher and it can reach 300 nm (Fig. 1b). The microhardness studies have shown, the multiple uniaxial pressing gives rise to a microhardness values in titanium billets, but it has also different values in the longitudinal and transversal sections. The maximal value of microhardness is found in the central part and it is equal to 3000 MPa (Fig. 2, curve 1). The microhardness decreases monotonously from 3000 to 2400 MPa at moving from the billet center to its periphery (Fig. 2, curve 2). By the way the average microhardness value in the coarse grained state is 1800 MPa.

The problem of heterogeneity of the titanium microstructure has been solved with the use of plastic deformation by rolling. By using TEM it was shown (Fig. 1c), that homogenous microstructure the grain – subgrain size of which is less than 100 nm was reached in titanium billets whereby rolling deformation reaches 90%. In addition, the microhardness values in titanium become almost constant at moving from the central part of a billet to its periphery and the average microhardness value increases up to 3400 MPa (Fig. 2, curves 3). However, the plasticity of nanostructured titanium formed under rolling deformation is less than 2 %. At the same time the plasticity of the coarse grained titanium is 24 % (Fig. 3, curves 1).

In order to improve a plasticity the prior-to-recrystallization annealing at 523 K is realized. It was demonstrated that the annealing in the temperature interval 523 K does not lead to visible changes in the microstructure and does not raise the size of elements (grains, subsgrains) (Fig. 1d). As a result of microhardness insignificant decreases up to 3200 MPa (Fig. 2, curve 3), but increases plasticity of nanostructured titanium from 2 to 6 % (Fig. 3, curve 2).

The uniaxial tension testing have demonstrated that formed nanostructure ensures the high mechanical characteristics of the nanostructured titanium. The tensile ultimate strength is 1160 MPa and tensile yield strength is 1100 MPa, (Fig. 3, curve 2). At the same time in the case of coarse-grained titanium these values are 500 MPa and 320 MPa respectively (Fig. 3, curve 1).

The mechanical characteristics such as tensile ultimate strength, tensile yield strength, fatigue strength, microhardness for coarse-grained and nanostructured titanium, titanium alloys Ti-6Al-4V and Ti-2Al-5Mo-5V are presented in Table 1. It can be seen that mechanical characteristics of the developed nanostructured titanium are similar to those of high-strength titanium alloys (Ti-6Al-4V and Ti-2Al-5Mo-5V).

An important characteristic of the resulting material is also the homogeneity of the elastic properties of the cross section and the possibility of cracking during machining. Investigation of the transverse distribution of mechanical characteristics, in particular the density of the material was carried out on the tomograph "Eagle", a tomograph 4-th generation with the collection of information through a conical beam of x-ray radiation. The radiation source is X-ray tube voltage of 140 kilovolts and the size of the focal beam of 15 microns. Through the lens radiation is recorded by the detector matrix 400x400 elements, each of which represents a combination of scintillator cesium-iodine-thallium and photodiode. Scanning mechanism provides: moving object in a plane transverse beam cross section, within the 300x300 mm.; Movement detector matrix in a plane transverse to the beam, within the limits of 300x300 mm.; Vertical displacement of the X-ray tubes of up to 150 mm. That lets you change the scale of the resulting images; rotation of the object at 180 degrees. In step 0,5 deg. The accuracy of object positioning control better than 1 micron. The software allows the imager to implement direct ray regimes, laminografii and tomographic reconstruction of the density distribution across the section. Studied the rods cross section 6x6 mm. In the rod was made to the disclosure of flaws on the surface of 10 microns. In Fig.4 b Fig.5 shows images of cracks in two mutually perpendicular directions. It is seen that the crack to the disclosure of the order of 1 micron. The system can detect. In Fig.6 are two cross-section with a thickness of each layer of 25 microns. Layered reconstruction allows a more detailed investigation of the shape of a crack in the various sections Analysis of the transverse density distribution over the cross section bar Figure 7 shows that the density is greatest at the surface exposed to compaction. In the figure 7 shows two sections of round bar, which show that the plane on the edge of a maximum and decreases towards the center. To the center density decreases, indicating the heterogeneity of the distribution efforts, and, consequently, the elastic properties of the cross section bar.

The structure of the material at the level of a micron with the help of "Eagle" could not be investigated, because of the radiator should have a focal spot to within fractions of a micron.

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Table 1. The mechanical characteristics for coarse-grained titanium, nanostructured titanium and titanium alloys

Material	$\sigma_{0,2}$ MPa	$\sigma_в$, MPa	σ_0, MPa	δ, %	H_{μ}, MPa
coarse-grained titanium	270	400	300	23	1700
nanostructured titanium	1100	1160	580	6	3300
Ti-6Al-4V	1010	1100	570	>6	3500
Ti-2Al-5Mo-5V	1000	1050	-	>10	3400

$\sigma_{0,2}$ – tensile yield strength; $\sigma_в$ – tensile ultimate strength; δ – plasticity; σ_0 – fatigue strength at $3 \cdot 10^6$ cycle base; H_{μ} – microhardness

Fig. 1. TEM bright field image and corresponding SAD of microstructure of nanostuctured titanium produced by multiple uniaxial pressing (a, b), subsequent rolling (c) and prior-recrystalline annealing (d): a - transversal section**,** billet center; b – longitudinal section**,** billet center, c - longitudinal section in relation to rolling, d - longitudinal section in relation to rolling, annealing at 523 K.

Fig. 2**.** Microhardness distribution of nanostructured titanium in section: 1 - nanostuctured titanium produced by multiple uniaxial pressing, longitudinal section; 2 - nanostuctured titanium produced by multiple uniaxial pressing, transversal section; 3 - nanostructured titanium produced by multiple uniaxial pressing after rolling deformation up to 90%; 4 – nanostructured titanium after annealing at 523 K; the arrow points the microhardness of coarse grained state.

Fig. 3. Stress-strain curves under tension testing: 1 – coarse grained titanium; 2 – nanostructured titanium after annealing at 523 K.

Fig.4. The image of a crack in a plane perpendicular to the beam axis.

Fig.5. Image of a crack in the plane of the beam axis.

Fig.6. Two-section rod in a plane perpendicular to the beam axis. The thickness of each section of 25 microns.

Fig.7. Layered reconstruction of the transverse distribution of density in round bars

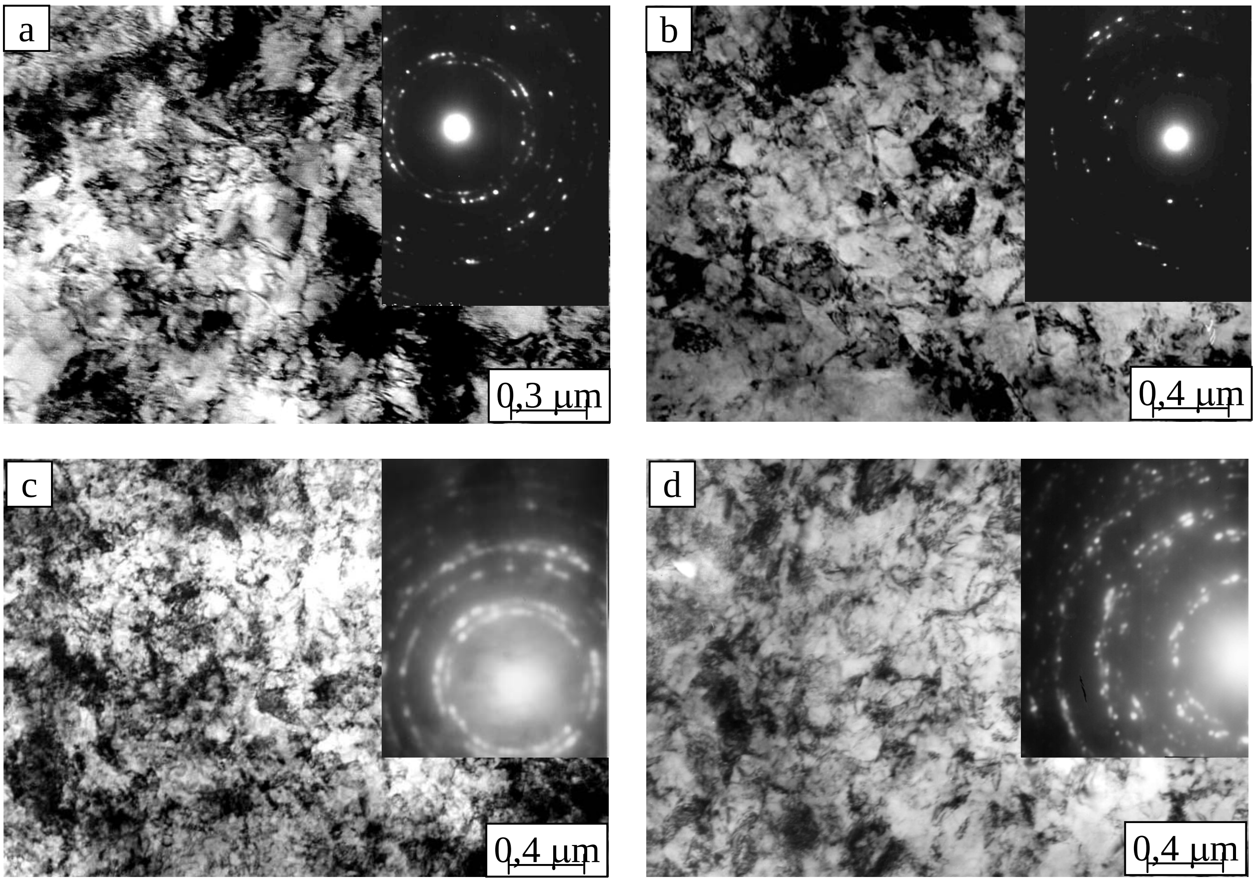
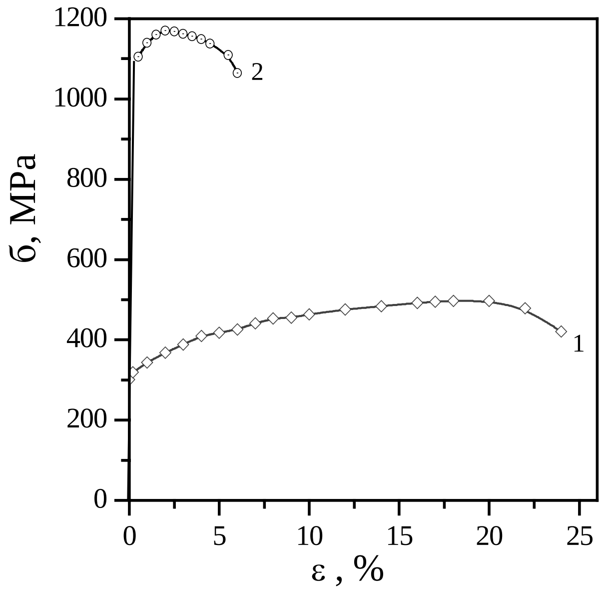
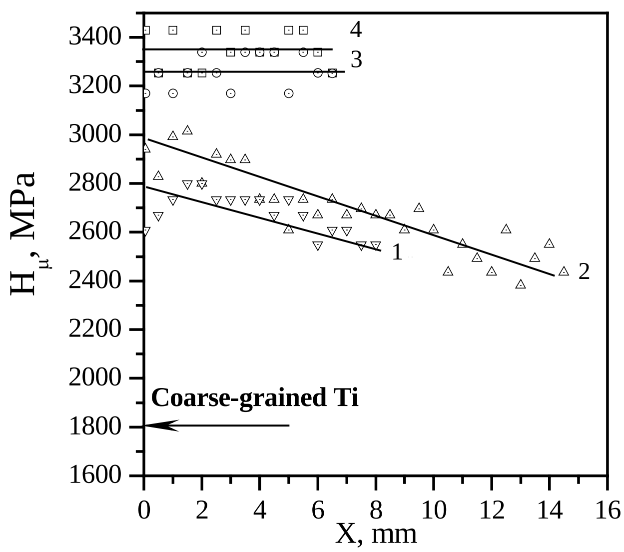


Fig.1



1200
1000
800
600
400
200
0
б, MPa
0
5
10
15
20
25
ε , %
1
2

Fig.2



3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
Hμ, MPa
0
2
4
6
8
10
12
14
16
X, mm
4
3
1
2
Coarse-grained Ti

Fig.3

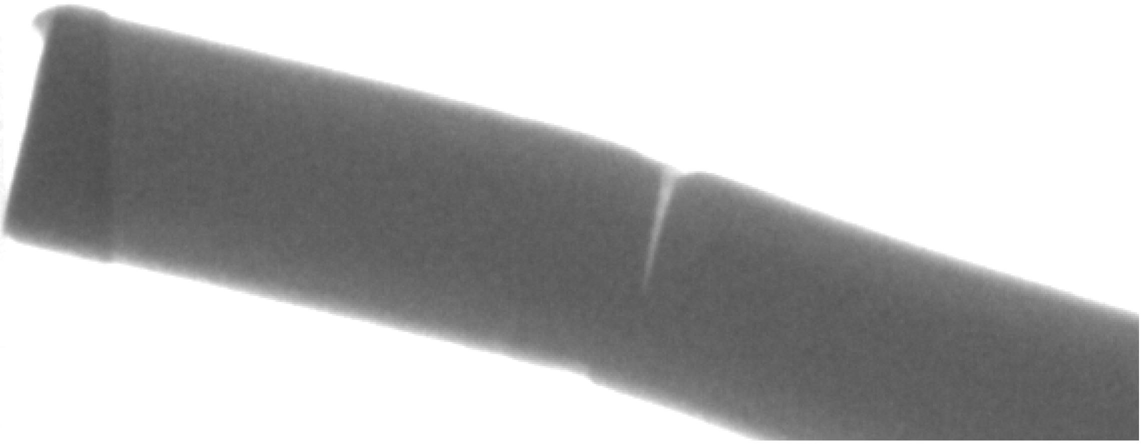


Fig.4.

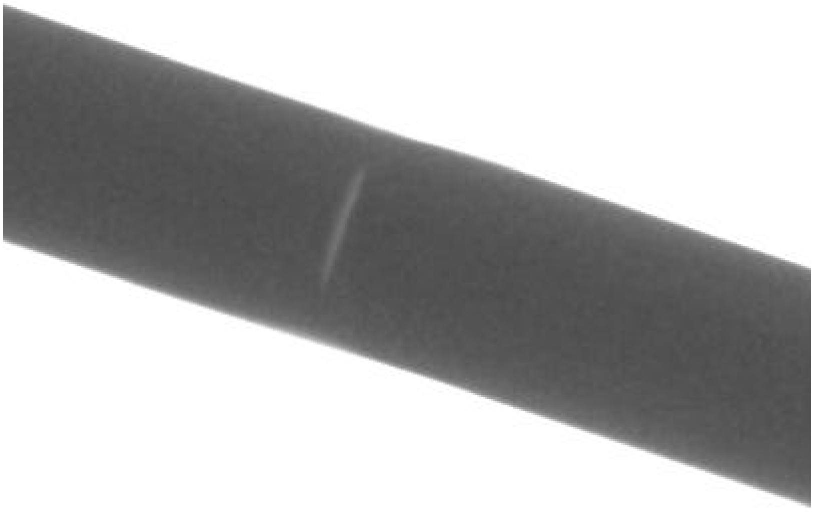


Fig.5.

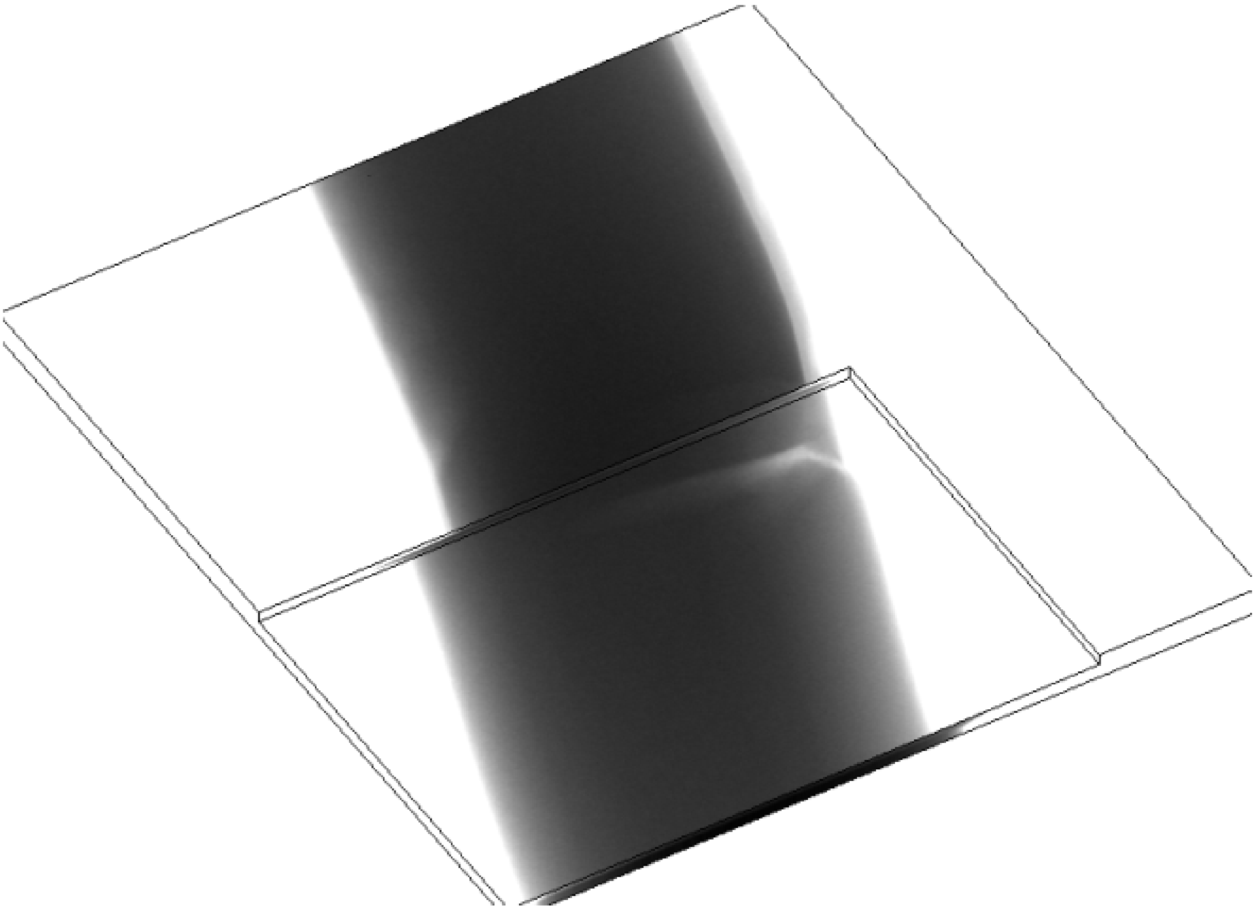


Fig.6.

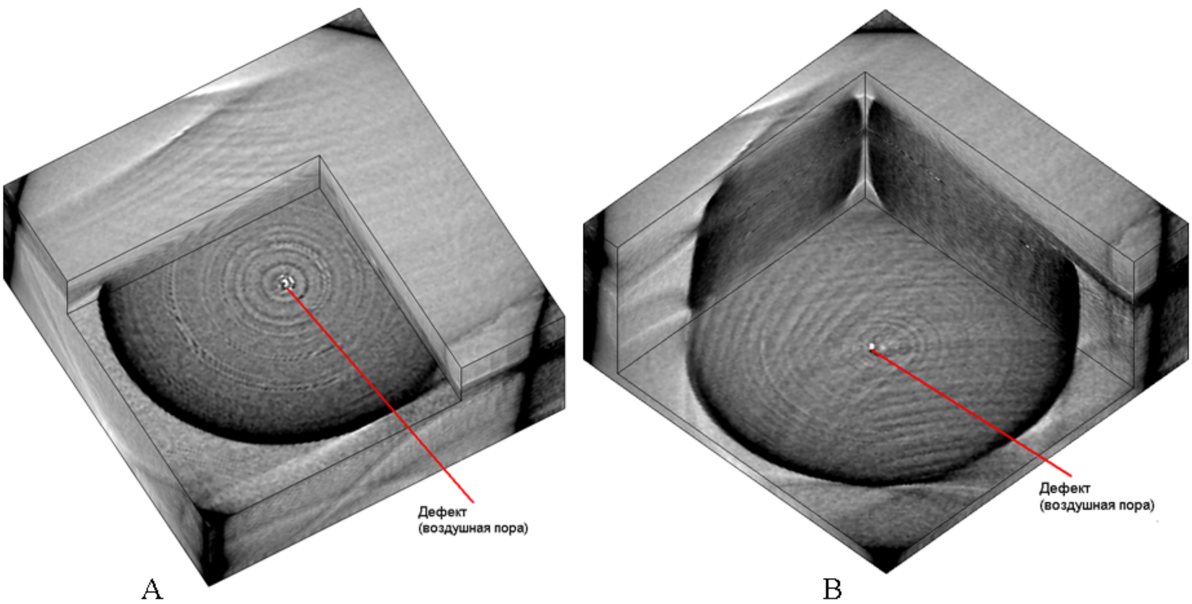


Fig.7