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PECULIARITIES OF ULTRAFINE-GRAINED STRUCTURE FORMATION IN Ti GRADE-4 USING ECAP-CONFORM

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Abstract. The paper describes the peculiarities of Ti Grade 4 structure evolution depending on a number of ECAP-Conform passes. Billets with a diameter of 12 mm were subjected to ECAP-Conform processing with a number of passes from 1 to 10 at a temperature of 200 °C. It has been shown that accumulation of strain that happens, when the number of passes increases from 1 to 6 (e~4.2), provides formation of an ultrafine-grained (UFG) structure with an average size of 250 nm in titanium. This leads to strength enhancement from 700 to 1020 MPa with some decrease of ductility. It has been established that further increase of the strain degree to ~7.0 (10 passes) will result in additional enhancement of relative and uniform elongation with retention of the strength level. This happens due to increase of a fraction of high-angle boundaries in the UFG structure, which are capable of grain boundary sliding during plastic deformation. It has been shown that in the course of ECAP-Conform a crystallographic texture forms in a rod, which can be described as a combination of orientations of pyramidal $\{01\bar{1}2\}\langle 10\bar{1}0\rangle$ and prismatic $\{10\bar{1}0\}\langle 01\bar{1}0\rangle$ components. During ECAP-Conform sliding along base planes is hindered, which can be associated with lower processing temperatures of titanium in comparison with a conventional ECAP.

1. INTRODUCTION

Due to high corrosion resistance and biocompatibility titanium and its alloys are widely used for production of medical implants [1]. However, there is observed a significant increase in requirements to products in modern medicine. For example, in addition to biocompatibility it is necessary to provide reduction in product weight with retention of its high strength and fatigue load resistance. Nowadays due to development of severe plastic deformation (SPD) techniques significant success was achieved in increasing of strength in titanium by high pressure torsion (HPT) [2-4], equal-channel angular pressing (ECAP) [5,6], ECAP in combination with extrusion and rolling [7-9].

One of new modifications of the ECAP techniques is ECAP-Conform (ECAP-C), and also combination of ECAP-C with subsequent drawing. This method allows fabricating an ultrafine-grained (UFG) structure in long-length rods and provides high production capacity and high perspectives for industrial application [10-12]. In order to develop this method and to achieve high mechanical properties, it is urgent to study general regularities and peculiarities of UFG structure formation depending on processing regimes and determine the impact of basic parameters of the forming structure (grain size, grain shape, substructure, state of grain boundaries and *etc*.) on the mechanical properties of titanium.

This work is focused on investigation of evolution of structure, texture and mechanical properties

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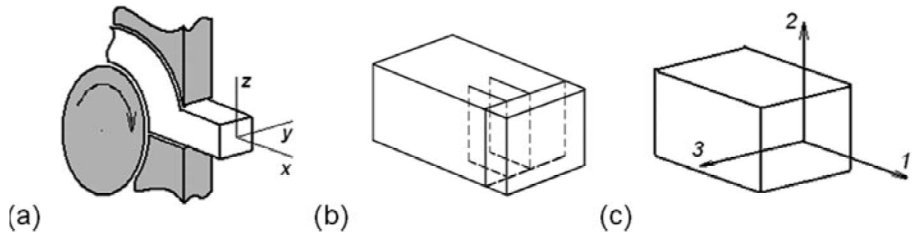

Fig. 1. Orientation of XZ and YZ planes towards a sample during ECAP-C (a) and scheme of samples cutting for foils for TEM studies (b); scheme of the area examined by X-ray (c).

of Ti Grade 4 depending on the strain degree (number of passes) in the course of ECAP-C.

2. MATERIAL AND EXPERIMENTAL PROCEDURE

CP Ti Grade 4 was used as initial material (produced by Dynamet Company) in the form of hot-rolled rods with ∅12 mm and an average grain size of 25 μm. Table 1 presents the chemical composition of the material. The rods were subjected to ECAP-Conform (Bc route) processing (Fig. 1a) with various degrees of accumulated true strain from 0.7 to 7.0 according to the number of passes (1, 2, 4, 6, 8 and 10). It is known that decrease of the ECAP temperature leads to grain size reduction in the UFG structure [10,13]. However, processing of titanium Grade 4 at room temperature usually is accompanied with crack formation. That is why in the present work ECAP-C was performed at 200 °C. A piece of 50 mm long was cut from each processed rod with a length of approximately 300 mm to study microstructure and mechanical properties under static tension.

The analysis of the microstructure was carried by transmission electron microscopy (TEM). The processed rods were examined in two sections, those are cross section plane YZ and longitudinal section plane XZ shown in Fig. 1a. The samples for foils were cut out by electrospark method according to the scheme shown in Fig. 1b. After mechanical thinning to 100 mm they were subjected to electrolytic polishing with the help of «Tenupol 5» set. Polishing was conducted with the help of the solution of perchloric acid - 5%, butanol - 35% and methanol - 60% within the temperature range of -20…-35 °C. The microstructure of foils was investigated by the JEOL JEM 2100 microscope under an accelerating voltage of 200 kV.

Texture formation in Ti was investigated with the help of diffractometer “DRON-3M” equipped with an automatic texture goniometer. Pole figures (PF) were obtained using filtered X-ray beaming of Cu $K_{\alpha 1}$ (0.1540598 nm). The experiment was conducted within the range of the radial angle γ from 0° to 75° and azimuth angle d from 0° to 360°. Consequently, a set of intensities of reflected X-rays $P_{hl}(y)$ was obtained. The diameter of the beamed area is 0.6 mm. The investigations were carried out in the geometric center of the cross section (plane 2-(-3)) (Fig. 1c).

Mechanical tensile tests were conducted at room temperature and with a strain rate 10^{-3} s^{-1} on the «Instron» test machine. There were used cylindrical samples of type 3 according to GOST 1497-84 with an initial gauge length of 15 mm and a diameter of 3 mm.

3. RESULTS OF THE EXPERIMENT AND DISCUSSION

In the present work ECAP-C performed at 200 °C allowed producing intact samples without obvious surface defects, when they were subjected to 10 deformation passes. Titanium Grade 4 and Grade 2 was usually processed by ECAP at a temperature of 400–450 °C in the die set with the angle of channels intersection Φ = 90°, as at lower temperatures surface defects and cracks appeared, which led to failure of a billet [14,15]. In the present work ECAP-C was conducted successfully at a temperature of 200 °C, which is connected with decrease of strain

Table 1. Chemical composition of Ti Grade 4 (according to the certificate, wt.%)

Ti	C	Fe	N_2	H_2	O_2
base	0.04	0.14	0.006	0.0015	0.36

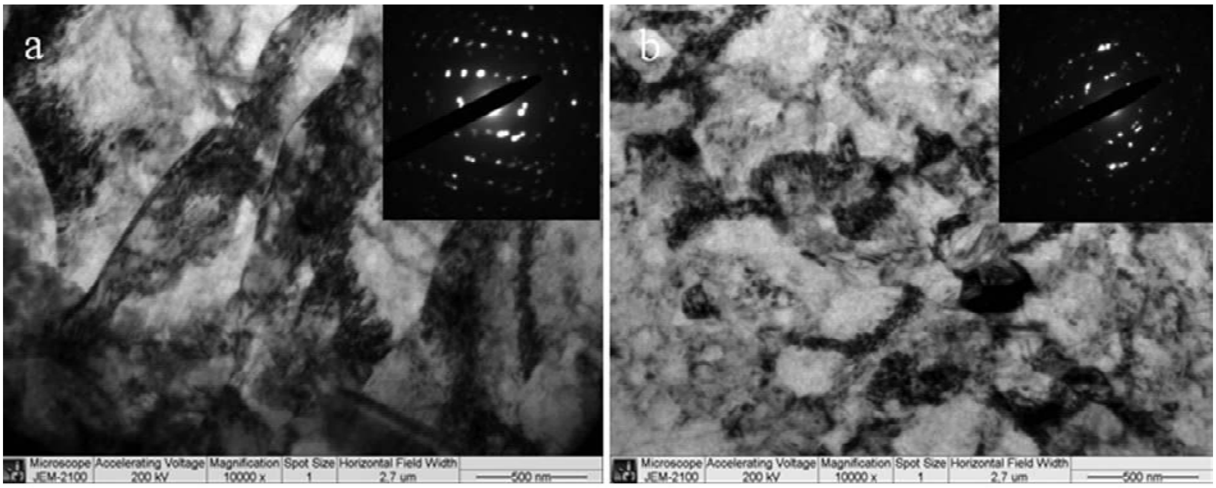

Fig. 2. Bright-field TEM images of Ti Grade 4 at a strain degree of ECAP of 0.7 (1 pass) (a) and of 2.8 (4 passes) (b) in the cross section.

intensity (angle of channels intersection $\Phi = 120°$) and with improvement of the process tribology.

TEM showed that after the first and second ECAP-C passes a subgrain structure forms (Fig. 2a). The subgrain structure is characterized by low-angle boundaries, enhanced dislocation density and deformation twins comparable to a grain size that is typical for low strain degrees and during conventional ECAP [6,15].

Due to crushing and intersection of twins, enhancement of misorientations of subgrains, further increase of the strain degree to e = 2.8 and higher during ECAP-C leads to formation of an ultrafine-grained structure (Fig. 2b). The size of grains/subgrains measured with the help of dark-field images is approximately 300 nm. The microstructure is characterized by inhomogeneity, high level of defectness of grain and subgrain boundaries, high dislocation density because of lower temperature of deformation (200 °C) in comparison with 450 °C used in earlier works for ECAP processing of Ti Grade 4 [15].

When the number of ECAP-C passes was increased to 6 and 8, the average size of grains and subgrains decreased inconsiderably in the microstructure of the cross and longitudinal sections of a rod, and it was 250 nm (Fig. 3a, b). Besides, further evolution of the microstructure was observed, which was characterized by increase of the total dislocation density and formation of larger fraction of high angle boundaries. This can be proved by increase in the number of spots in the electron-diffraction patterns (Fig. 3). When the strain degree was increased to 10 passes, that is e = 7.0 an ultrafine-grained structure of a granular type mostly forms in Ti (Fig. 3c, d). The grains are of equi-axed shape, their boundaries are thin and clearly observed on the bright-field images (Fig. 3).

The study of mechanical properties of samples after tension demonstrated that the highest increment in strength (from 750 to 880 MPa) was observed during the 1st pass (Fig. 4) due to strong fragmentation of the structure and enhancement of dislocation density. When the strain degree achieved ~ 4 (6 passes), the strength increased to 1020 MPa due to formation of a UFG structure with an average size of grains/subgrains of approximately 250 nm. Tendency towards reduction of ductile parameters (relative and uniform elongation) to 13 and 1.5% was observed, which can be associated with accumulation of high dislocation density in an ultrafine grain and presence of a large number of low-angle subgrain boundaries. The strength of the material stabilizes and almost does not change, while the strain degree increases from 5.6 to 7.0, which is explained by retention of grain and subgrain sizes. It should be noted that after 10 passes there is a tendency towards enhancement of ductility, that is relative and uniform elongations (to 14 % and 2.2 %, correspondingly). This can be explained with increase of a fraction of high-angle boundaries in the UFG structure, which are capable of grain boundary sliding during plastic deformation [17,18].

It is known that the formation of crystallographic texture can also influence mechanical properties of metals and alloys. The position of texture maxima on the experimental pole figures (PF) of the initial state of Ti Grade 4 (Fig. 5) is typical of the PF of Ti deformed with the help of rolling. Prismatic $\{10\bar{1}0\}\langle 01\bar{1}0\rangle$ and basic $\{0001\}\langle 01\bar{1}0\rangle$ components of the texture prevail, which are active for coarse-crystalline titanium [19]. It is observed that

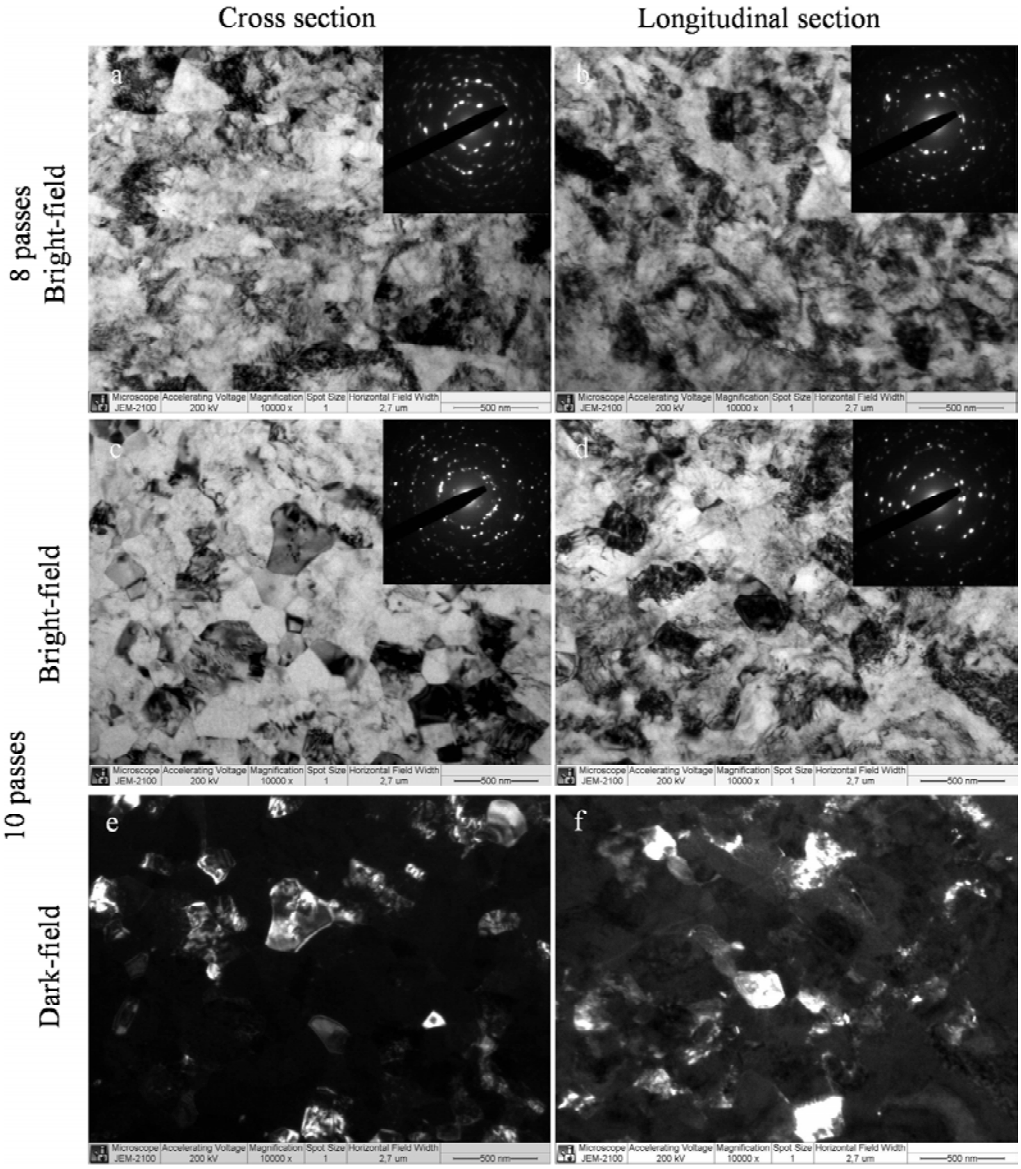

Fig. 3. TEM images of Ti Grade 4 after 8 and 10 ECAP-C passes in cross and longitudinal sections.

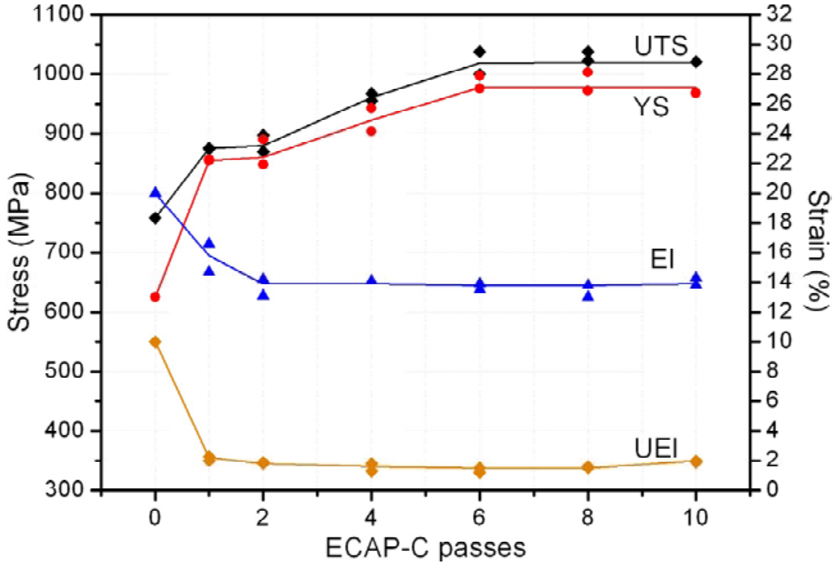

Fig. 4. Influence of the number of ECAP-C passes on the mechanical properties of Ti Grade 4. UTS – ultimate tensile strength; YS – Yield Strength; El – relative elongation; UEl – uniform elongation.

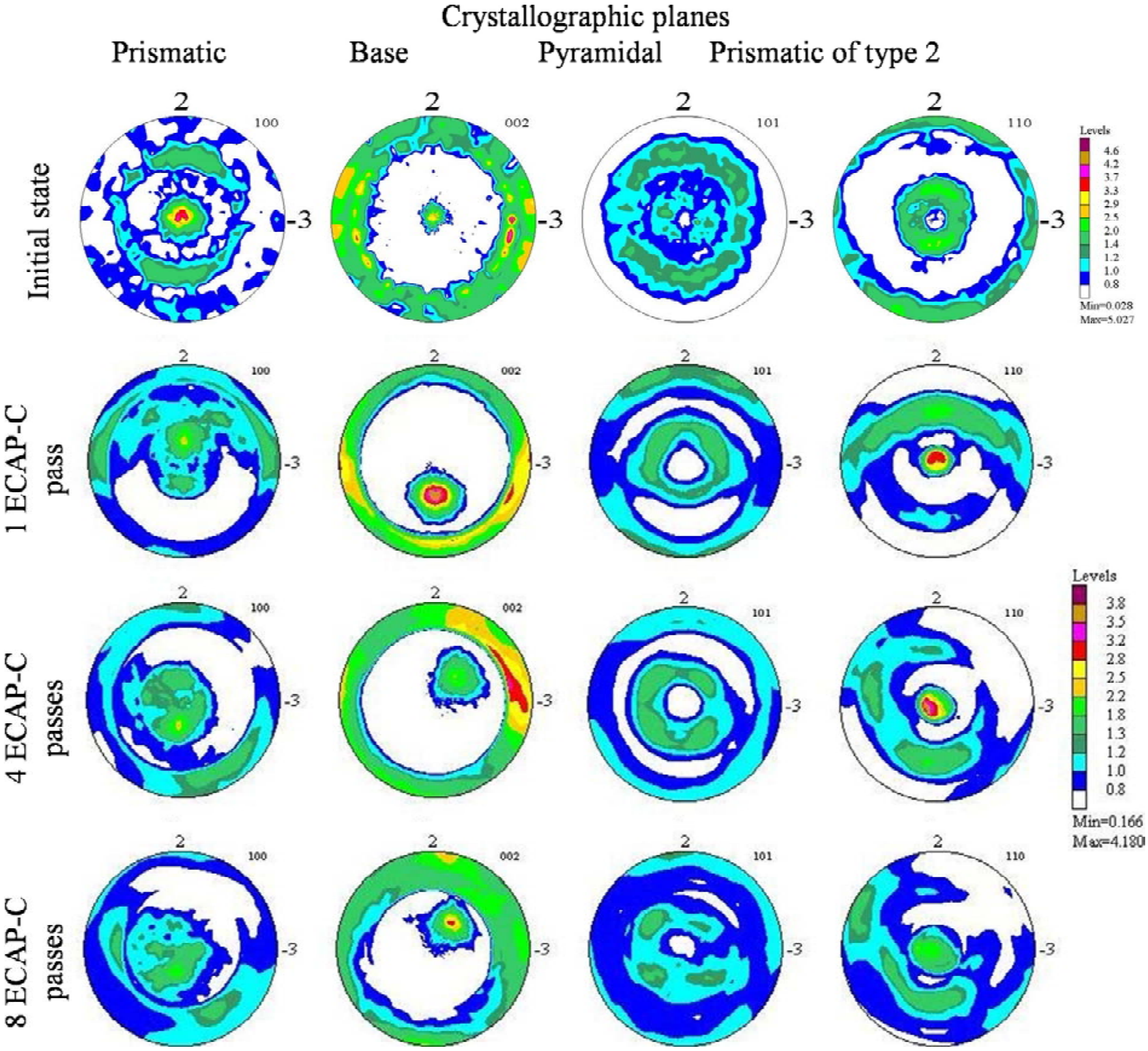

Fig. 5. Experimental pole figures (PF) (10$\bar{1}$0), (0002), (101) and (110) Ti Grade 4 in various structural states.

the texture maxima on the PF are practically located in their center, but at the same time they are characterized with blurring of pole density in the radial directions.

The first ECAP-C pass predetermines the character of further distribution of preferred orientations of crystallites. The normals to the base planes of hexagonal close-packed lattice are located both along the longitudinal axis of a rod with some 45° turn and radially from the center of a billet. This texture can be described as combination of orientations of pyramidal $\{01\bar{1}2\}\langle 10\bar{1}0\rangle$ and prismatic $\{10\bar{1}0\}\langle 01\bar{1}0\rangle$ components of texture.

When the number of ECAP-C passes increases to 4, the prismatic components of the second order $\{11\bar{2}0\}\langle 0001\rangle$, pyramidal ones $\{10\bar{1}1\}\langle 10\bar{1}0\rangle$, and those close to basic ones $\{0001\}\langle\bar{3}\bar{2}50\rangle$ become main components. When the number of ECAP-C passes increases to 8, the crystallographic texture practically does not change (Fig. 5). It should be noted that in case of conventional ECAP sliding along the base planes prevails [16], in case of ECAP-C sliding along the base planes is hindered. It may be associated with lower temperatures of processing of titanium (ECAP at 450 °C, ECAP-C at 200 °C). When the strain degree achieves e = 5.6 in a rod, there is no sharp crystallographic texture, which corresponds with the results of TEM studies, particularly with formation of grains of mainly equiaxed shape both in the cross and longitudinal sections of the rod.

Therefore, the total accumulated strain degree during ECAP-C has a significant influence on increase of strength properties of titanium due to formation of the UFG structure. Achievement of high strain degrees (from e ~5.6 to 7.0) does not result in additional grain size reduction and, as a consequence, in further increase of the achieved level of strength. Besides, the evolution of the UFG structure, in which, probably, transformation of subgrain boundaries into grain ones takes place, and grains of equiaxed shape form, contributes to enhancement of ductile properties (relative and uniform elongation). Such regularities were also observed during SPD by conventional ECAP for Ti and other metals [9,10]. The formation of such a type of UFG structure may result in qualitative changes of structure formation during subsequent deformation process, for example drawing or rolling. In particular, specific mechanisms of deformation are possible to be realized (grain boundary sliding), which contributes to increase of deformability of the material and additional refinement of UFG structure. In our further investigations of development of combined processing regimes using ECAP-C and subsequent drawing, main attention will be focused on such structure parameters as grain size and shape, dislocation structure, state of grain boundaries having influence on mechanical behavior of metals.

4. CONCLUSIONS

According to the results of conducted investigations the following peculiarities of UFG structure formation and mechanical properties in Ti Grade 4 during ECAP-C at T = 200 °C depending on the accumulated strain degree have been stated:

1. It has been demonstrated that at low strain degrees (e = 0.7 ÷ 1.5) one of the mechanisms of structure refinement is considered to be intersection and crushing of twins and also accumulation of enhanced dislocation density that leads to increase of strength from 750 to 880 MPa with some reduction of ductile parameters;

2. The increase of the strain degree up to 7.0 contributes to enhancement of misorientations of grains and subgrains relative to each other and to increase of a fraction of high-angle boundaries and transformations of subgrains into grains of mainly equiaxed shape. The strength achieved 1050 MPa under relative and uniform elongation of 14 and 2.2 %, correspondingly;

3. The crystallographic texture, which forms after e = 4.2 and is characterized by the components of prismatic type of the second order $\{11\bar{2}0\}\langle0001\rangle$ and pyramidal $\{10\bar{1}1\}\langle10\bar{1}0\rangle$ and close to basic type $\{0001\}\langle\bar{3}\,\bar{2}\,50\rangle$, practically does not change, when the strain degree achieves e = 7.

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