



# FEATURES OF THE EVOLUTION OF VT6 TITANIUM ALLOY STRUCTURE DURING ROLLING OF THE STRIPS IN THE HELICAL ROLLS AND ON THE LONGITUDINAL-WEDGE MILL

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## ABSTRACT

In the article the research results of the effect of the number of passes during rolling of the strips in the helical rolls, as well as the effect of the draft during rolling on the longitudinal wedge mill on the parameters of the microstructure of VT6 titanium alloy have been shown. A comparative evaluation of the grain size of fine-grained, submicrocrystalline and nanocrystalline structures after rolling strips in the helical rolls by the various passages, and after rolling on the longitudinal wedge mill at temperatures of deformation 960, 1050, 650 and 550°C have been conducted. Common parameters of the grain and defect structures, of the crystal lattice curvature, of the local internal stresses and their gradients have been presented. The mechanisms of the plastic deformation and reorientation of the crystal lattice have been studied depending on the number of rolling passes in the helical rolls.

**Key words:** VT6 Titanium alloy, Rolling, Helical rolls, Severe plastic deformation, Longitudinal wedge mill, Electron microscopy, Submicrocrystalline nanostructured state, Microstructure formation, Microstructure evolution, Grain size, A draft.

## INTRODUCTION

Currently known and widely used titanium alloys, which are due to the low energy losses (small loss factor) and good specific strength provide stable operation of mechanical engineering details and shipbuilding products, etc. At the same time titanium alloys with an initial coarse-grained structure under the large loads do not have enough service life.

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However, the enhancement of the physical and mechanical properties of titanium alloys would allow significantly expand their technical capabilities and range of applications. Preliminary estimates made by the authors<sup>1-3</sup> have suggested that the formation of ultrafine structure (nanostructures) in titanium alloys with improved physical and mechanical properties can significantly increase the service life (load-cycle) of such products.

Now to produce high-quality materials with ultrafine grained (UFG) structure (nanostructure) without significant changes in their size, the methods of severe plastic deformation (SPD) are used, mainly by realizing macro shares strain with the total degree of more than 2-3<sup>1,4-7</sup>: torsion under high hydrostatic pressure, equal channel angular pressing, comprehensive isothermal forging and radial-shear rolling, etc. Macro shares lead to the changes in the structure of the metal due to inter crystalline slip, which does not depend on the orientation of the crystal grains. As a result, the degree and uniformity of the mechanical properties of metal increase, and the anisotropy decreases. In the above work and other studies of the last decade<sup>8-11</sup> have been shown that the materials, nanostructured by SPD methods, inherent high physical and mechanical properties. The metals and alloys with submicron and nanocrystalline structure exhibit unusually high and useful strength and ductility.

During sheet rolling process intensive macro shares can be provided by different technological and constructive ways<sup>12</sup>: by using preforms and rolls with a wavy or corrugated surface, by asymmetric rolling, or by uneven cooling-down of the strip plate along its thickness and width by using crossed rolls and the rolls with a projection on the surface, etc. The authors of<sup>12</sup> note that in all these cases, intensive macro shares achieved as a result of local deformation effects on the rolled metal.

The Japanese firm «JFE Steel» suggested the method of multiple sequential alternating bending of the steel strip after hot rolling (deformation method of accumulation of bend)<sup>13</sup>. From the research it can be seen that the use of reverse bending allows to roll slabs without changing its thickness in contrast to conventional rolling. Consequently, this method allows deforming the slab any number of times by using bending cyclic. This allows producing the hot-rolled strips with UFG structure. According to the authors<sup>13</sup>, such kind of methods of rolling can be used in the industry to improve the quality of rolled metal products. This is due to the fact that rolling by cyclic bending leads to the production of hot rolled strips with the size of ferrite grains 1  $\mu\text{m}$  and less.

Thus, many new designs of rollers proposed to improve the quality of sheet products. However, many rollers are not found their wide application in the production for the

following reasons: the complexity of their manufacture; the difficulty of installing them on the rolling mills.

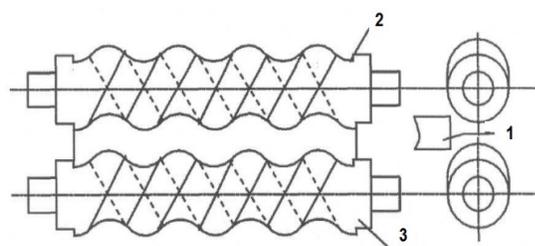
The aim of the research is to study the structure of the VT6 titanium alloy during rolling the strips in the rolls with helical surface and on the longitudinal-wedge mill (LWM).

### Equipment, materials and method of experiment

Among the known SPD methods the foil rolling is widely used in practice. However, because of the small cross-section of the foil, it is of little use for subsequent forming operations. Therefore, a tool containing the rolls with helical working surfaces was developed (Fig. 1). This tool generally intended to produce semi-finished products with UFG structure<sup>14</sup> and it implements the SPD without significant changes of work piece original shape and size. In addition, a 5-stand LWM was designed to roll the strips from the work piece, that has an UFG structure.



(a) Mill



(b) Rolling scheme

**Fig. 1: Duo rolling mill involving the rolls with corrugated working surfaces:  
1 – Workpiece; 2 – Drag-over mill; 3 – Bottom roll**

Continuous mill comprises working stands, motor, coupling, bearing npowered rolls, driven working rolls, base frame, base plate.<sup>15</sup> Cage, which has an AC motor drive, contains working and supporting rollers of constant diameter, while the diameter of working rolls reduces in sequentially located stands, and the diameter of the backup rolls increases in the rolling direction. At the same time the rotation of the rolls is carried out through individual coupling, reducer, gear cage and spindle.

It should be noted that the predetermined distance between the working rolls from one stand to another increases by the magnitude of advance.

The tool for hot rolling of the steel and alloys comprises upper and lower rolls with helical working surfaces. The projections and depressions of the upper roll made by the helical line, and they are opposite to the depressions and protrusions of the lower roll. Moreover, the angle between the tangent to the spiral lines and the line passing through the point of contact for forming perpendicular to the base roll is from  $45^\circ$  to  $60^\circ$ . It should be noted that the projections and depressions of the upper and lower rolls have the same width and consequently height or depth.

During rolling of the workpiece in this tool, the protrusions of the work surface on one side of the roll arranged opposite to the depressions of the working surface on the other side of the roll. Rolling in the first pass and the subsequent compression unit is carried out with draft equal to  $\varepsilon = \Delta h_B/H_0$ ,  $\varepsilon = 2 \Delta h_B/H_0$  (where  $\Delta h_B$  - the height of the projection or the depth of the corrugated roll;  $H_0$  - height of the workpiece before rolling). Such rolling ensures efficient structure refinement along the entire cross-section of the workpiece due to alternating bending deformation in longitudinal and transverse cross-sections of the workpiece. Moreover, along the width of the rolled strip the disalignment of projections and depressions occur, which is arise as a result of rolling. This creates additional macro shares along the cross-section of the workpiece, that lead to an additional grinding of metal structures and alloys, another words, additional conditions are generated for obtaining high-quality material.

The VT6 titanium alloy was selected as the workpiece material with the sizes of  $6 \times 150 \times 400$  mm. Rolling was performed according to Table 1.

As it can be seen from Table 1, heating of the work piece was conducted at the temperature of  $960^\circ\text{C}$  then it was soaked for 3 hrs and after that it was rolled by 2 passes in the helical rolls till the width of 5.7 mm. Thereafter, the same workpiece was warmed up at  $1050^\circ\text{C}$ , soaked for 30 min and rolled on the LWM while the width reaches 3.2 mm. Whereupon, the work piece was again warmed up at the temperature of  $650^\circ\text{C}$ , then soaked for 30 min and rolled in the helical rolls by 2 passes till the width of 3.0 mm. Finally, it was warmed up at  $550^\circ\text{C}$ , then soaked for 30 minutes and rolled on the LWM up the width of 1.5 mm.

The same procedure was followed for the modes 2.1-2.4, 3.1-3.4 and 4.1-4.4. The only difference is in the number of the passes the work piece was processed in the helical rolls. For the mode 2 there are 4 passes, for the mode 3 there are 6 passes, and for the mode 4 there are 8 passes in the helical rolls.

Rolling in the rolls with helical work surfaces performed as follows. The perform

was fed into the gap between the upper and lower rollers and it was deformed by the projections and depressions of the rolls with a draft equal to  $\varepsilon = \Delta h_B/H_0$  in the first pass and with the draft equal to  $\varepsilon = 2 \Delta h_B/H_0$  in the subsequent passes.

**Table 1: Rolling modes**

Work piece	Mode No.	Heating (°C)	Warming (°C)	Soaking (hrs)	Rolling		Width of the work piece after rolling (mm)	Note
					In the helical rolls, No. of pass	On the LWM		
Work piece 1	1.1	960	-	3	2	-	5.7	The work piece was processed through the every stage of mode 1
	1.2	-	1050	0.5	-	+	3.2	
	1.3	-	650	0.5	2	-	3.0	
	1.4	-	550	0.5	-	+	1.5	
Work piece 2	2.1	960	-	3	4	-	5.7	The work piece was processed through the every stage of mode 2
	2.2	-	1050	0.5	-	+	3.2	
	2.3	-	650	0.5	4	-	3.0	
	2.4	-	550	0.5	-	+	1.5	
Work piece 3	3.1	960	-	3	6	-	5.7	The work piece was processed through the every stage of mode 3
	3.2	-	1050	0.5	-	+	3.2	
	3.3	-	650	0.5	6	-	3.0	
	3.4	-	550	0.5	-	+	1.5	
Work piece 4	4.1	960	-	3	8	-	5.7	The work piece was processed through the every stage of mode 4
	4.2	-	1050	0.5	-	+	3.2	
	4.3	-	650	0.5	8	-	3.0	
	4.4	-	550	0.5	-	+	1.5	

Metallographic analysis was performed using energy dispersive spectrometer JNC ENERGY (England), mounted on electron probe microanalyzer JEOL (Dzheol) at an accelerating voltage of 25 kV. JEOL instrument magnification range equal to 40 to 40000 times. The principle of the microprobe is high-energy (25 keV) narrow (1  $\mu$ m) beam of electrons is directed onto the sample, which is set in the screen (frame) by scanning the sample and recording the secondary electrons emitted by the sample. The resulting picture is very similar to the optical photo, but due to the fact that the electron beam is very thin

( $\approx 1\text{-}2\ \mu\text{m}$ ), the depth of focus is much higher than that of optical photo and used magnification is much higher, respectively, it is possible to distinguish smaller structural components of the workpiece. Structural studies of the deformed samples were also performed by transmission electron microscopy methods of thin foils by using JEM-2100CX electron microscope with the accelerating voltage of 200 kV.

Standard techniques of electrodeposited necking of flat workpieces and inkjet electropolishing were used to prepare the samples for electron microscopy studies and using an electrolyte composition: 10% of perchloric acid and 90% of glacial acetic acid.

Before blasting, mechanically polished samples was ground to a thickness of 0.2 mm, and then these discs were cut with a diameter of 3 mm, and then they were polished by electrochemical method in order to remove the defective layer, and only then spray polishing was performed until the formation of holes.

Quantitative analysis of the defect substructure parameters was carried out by standard methods<sup>16</sup>. Microsections for metallographic study were prepared according to traditional methods by using grinding and polishing circles. Concentrated nitric acid solution in ethanol was used for etching samples.

Special methods of dark-field analysis of discrete and continuous misorientation were used to determine the microstructure parameters<sup>17-19</sup>, which can detect and quantitatively certify such important features of high defective structural states as: curvature of the crystal lattice and continuum density of defects (dislocations and (or) disclinations), nonequilibrium boundaries of grains with a high continuum density of disclinations, local (nanoscale level) internal stresses and gradients of these stresses.

Analysis of the crystal lattice curvature was conducted by using the bending-torsion tensor (Fig. 2), furthermore the parameters of the crystal lattice planes curvature of various types of lattice can be described by the components of bending-torsion tensor ( $\chi_{ij}$ ) and by the rotation vector  $\omega$ <sup>17-19</sup>.

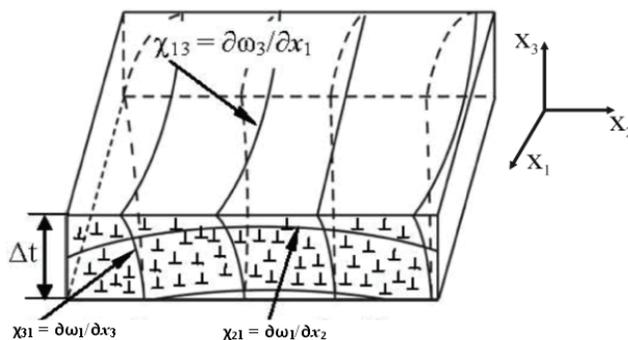
Fig. 2 shows the coordinate system, where the horizontal component of curvature planes (e.g., in  $X_1$  direction) is represented by the component  $\chi_{21}$  in the bending-torsion tensor<sup>17-19</sup>. It requires to note that the components of curvature planes are perpendicular to the wave vector of the electrons (for small values of  $\varphi$ , these are the planes that are perpendicular to the surface of the foil). The same component of the crystallographic planes curvature is  $\chi_{31}$  component of bending-torsion tensor. It forms the diffraction contrast and almost parallel to the wave vector of the electrons (for small values of  $\varphi$ , these are the planes

that are perpendicular to the surface of the foil). The components of the crystal lattice curvature  $\chi_{21}$  and  $\chi_{31}$  (Fig. 2) can be determined by the formulas<sup>17-19</sup>:

$$\chi_{21} = \Delta\varphi \sin\beta / (\Delta r) \quad \dots(1)$$

$$\chi_{31} = (L\chi_{21} - \Delta\gamma_0) / \Delta t \quad \dots(2)$$

where  $\Delta r$  – trace slip of the extinction contours, when changing the tilt angle of the sample in the goniometer by the angle  $\Delta\varphi$ ;  $\beta$  - the angle between the direction of the current reflection and projection of the tilt axis (PTA);  $L$  - the width of extinction circuit;  $\Delta\gamma_0$ - the angular size of the diffraction peak;  $\Delta t$  - the thickness of the foil.



**Fig. 2: The scheme of the structural state of the crystal lattice with a high curvature:  $\Delta t$  - the thickness of the foil;  $\omega_1, \omega_3$  - the projection of the rotation vectors<sup>17</sup>**

On the basis of experimental data about the curvature of the crystal lattice it is possible to estimate the values of local internal stresses ( $\sigma_{lok}$ ) and gradients ( $\partial\sigma_{lok}/\partial r$ ) by the formulas<sup>17-19</sup>.

$$\sigma_{lok} \approx \chi_{ij} E \Delta h / 2 \quad \dots(3)$$

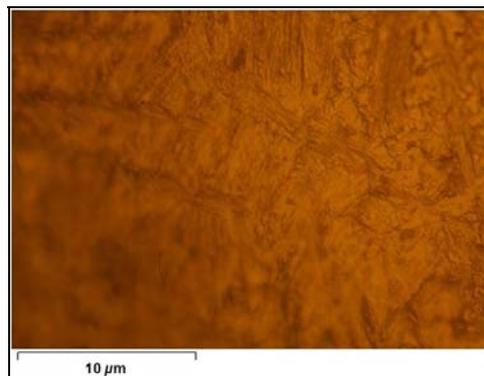
$$\partial\sigma_{lok} / \partial r \approx (E/2\pi) \chi_{ij} \quad \dots(4)$$

where  $E$  - Young's modulus;  $\Delta h$  - the characteristic dimensions of the curvature detection area of the crystal lattice.

## RESULTS AND DISCUSSION

In the initial state of the alloy microstructure has a form of elongated  $\alpha$ - plates inside the relatively equiaxial  $\beta$ -grains. The amount of  $\beta$ -grains corresponds to 6-7 points, and the

value of  $\alpha$ -colonies is 5-6 points. Thickness of  $\alpha$ -plate is 2-5  $\mu\text{m}$ , and  $\beta$ -layers are up to 1  $\mu\text{m}$  (Fig. 3). This microstructure is typical for two-phase alloys with lamellar structure<sup>20</sup>.

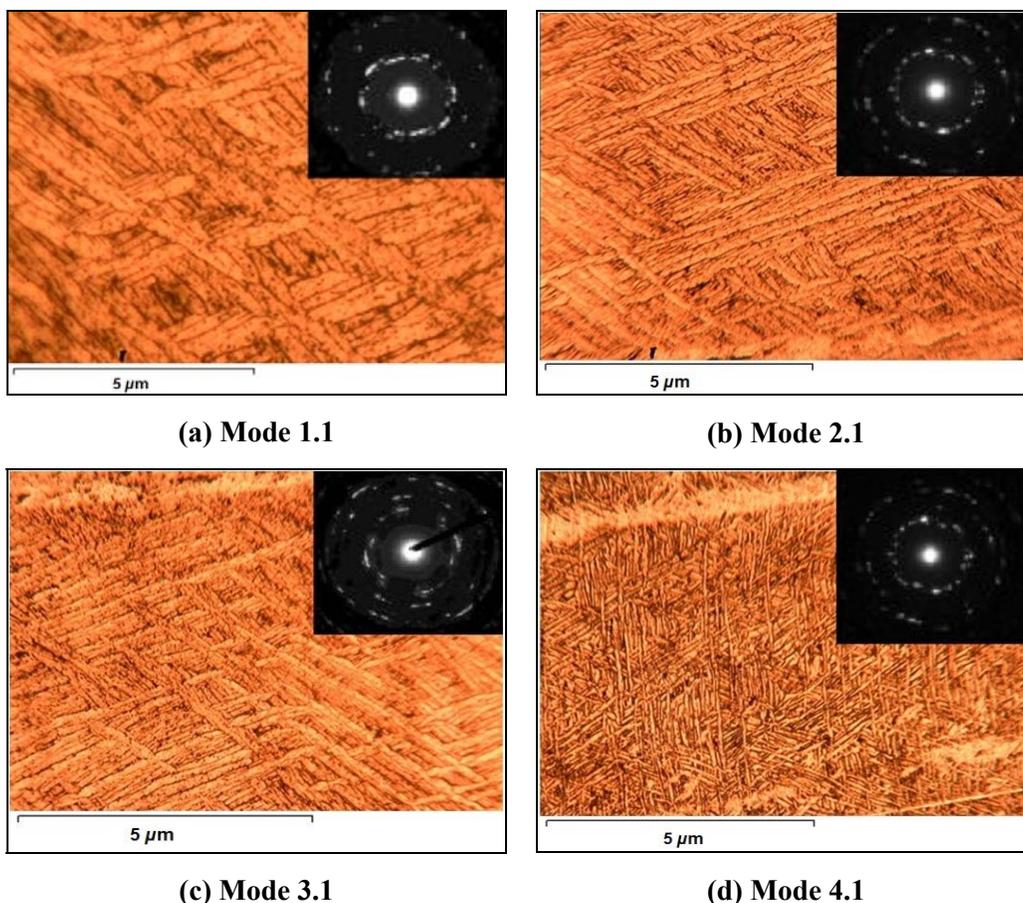


**Fig. 3: The microstructure of the VT6 titanium alloy in the initial state**

The study of the structural state of VT6 titanium alloy, after rolling it according to mode 1.1, showed that a microstrip structural state has formed in the cross-section, which is perpendicular to the rolling plane (Fig. 4, a). In this case, the dislocation density increases and shear band with a wide up to 800 nm forms. The deformation in the form of shear bands takes place mainly within the large grains. The most likely widths of microbands with high-angle boundaries are in the range of 500 to 600 nm at the maximum (very rarely observed) values about 800 nm (Fig. 4, a). The width of micro bands with low-angle boundaries can vary from a few tens of nanometers to 350 nm with a most likely value of about 220 nm. The values of the crystal lattice curvature reach  $\chi_{ij}$  values  $\approx$  20-30 degrees/ $\mu\text{m}$ . The relative volume, occupied by these structural condition, is 45-55%.

Further rolling by modes 2.1 and 3.1 (Fig. 4, b, c) reduces the width of the micro bands, and leads to the formation of thinner strips shift at the boundaries of the original wide microbands. Furthermore, in the cross-section of the strip a pronounced banded structure is formed with a distance not exceeding 350 nm at the most probable values of 250-280 nm between the angle boundaries (Fig. 4, b, c). The curvature of the crystal lattice is  $\chi_{ij} \approx$  15 – 25 degrees/ $\mu\text{m}$ .

An important feature of the structural state is that nearly half of the studied microbands with high-angle boundaries have misorientation vectors equal to  $\theta \approx 45-55^\circ < 110 >$  after rolling by mode 3.1. Perhaps this is due to the alternating bands of deformation in the helicalrolls. A large number of clear electron reflexes through the rings of X-ray diffraction pattern show that the majority of grains in the structure have high-angle boundaries.



**Fig. 4: The microstructure of VT6 titanium alloy after rolling in the helical rolls**

The increase of the number of passes to 8 (Fig. 4, d) leads to a strong fragmentation of the grain into thin shear band with the width of about 110-120 nm. Multiple reflections on the X-ray diffraction pattern along the circles indicate that adjacent grains have the high-angles disorientations. Cross boundaries are formed inside the strips and multiple microtwinning develops, as a result of which the structure strongly refines.

Thus, during rolling process in the helical rolls the action of alternating wavy mechanisms of deformation provides fragmentation and reorientation of the crystal lattice. Meanwhile high-angles boundaries with high density arise in the transverse direction of the workpiece.

A common feature of structural changes during rolling in the helical rolls is the presence of high density boundaries with variable disorientation vectors. Features of such

boundaries have continuous nature of the orientation change of the crystal lattice along the border and, as a consequence, presence of structural states with high values  $\chi_{ij}$  ( $\chi_{ij} = 25-35$  degrees/ $\mu\text{m}$ ) in the border areas of grain. The proportion of such borders is about 65%.

Presence of these borders indicates the implementation of dislocation-disclination mechanism of reorientation of the crystal lattice, which is developed in 2 stages: the formation of a substructure with non-zero components of the tensor density of disclinations; its collective relaxation in the discrete boundaries of disorientation. It is known<sup>17-19</sup> that such a mechanism is one of the most universal mechanism of fragmentation of the crystal, including the formation of submicron and nanocrystalline structural conditions in a wide range of metals and alloys.

To investigate the effect of rolling on LWM on the formation of the microstructure of VT6 titanium alloy, the strips rolled in the helical rolls then was rolled on LWM at the temperature of 1050°C (Fig. 4). It is seen that increasing rolling temperature to 1050°C significantly affect the microstructure of the alloy.

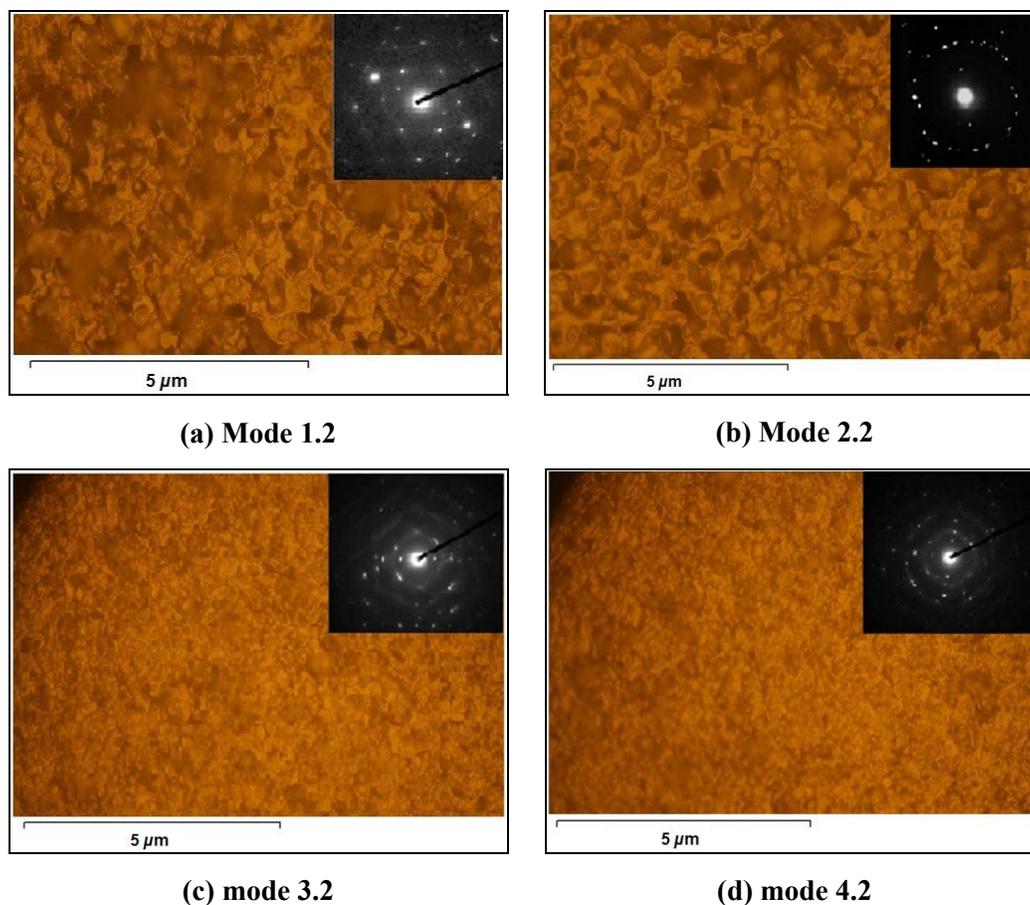
After subsequent rolling of the work pieces according to mode 1.2, its microstructure characterized by the presence of subgrains, which have been formed inside the former strain bands (Fig. 5, a). The average size of subgrains is about 410-430 nm, in addition areas with already formed individual grains are also observed. Electron diffraction pattern confirms the continuation of the crystallographic texture.

Similar changes of the microstructure pictures were observed in the workpiece, which was rolled by mode 2.2. This forms subgrain structure with an average grain size of 370-390 nm (Fig. 5, b).

Rolling of the work piece on LWM, which was deformed according to modes 3.2 and 4.2, leads to the formation of a structure with ultrafine grain size. As a result of primary recrystallization the structure with the ultrafine size of grains equal to 250-270 nm forms along the entire volume of the rolled strips (Fig. 5, c, d). The resulting structure is characterized by uniformity of ultrafine grain size throughout the volume of the material. A large number of clear reflexes along the rings of electron diffraction show that the majority of grains in the structure have a high-angle boundaries. In such boundaries the crystal lattice is continuously changing its orientation and, as a result, the structural states with high values  $\chi_{ij}$  ( $\chi_{ij} = 28-36$  degrees/ $\mu\text{m}$ ) formed in the grain boundary regions. The share of such boundaries is about 75%.

Rolling of the blanks in accordance with the modes 1.3 and 2.3, resulted in a further refinement and development of the grain-subgrain structure in VT6 alloy. The size of some

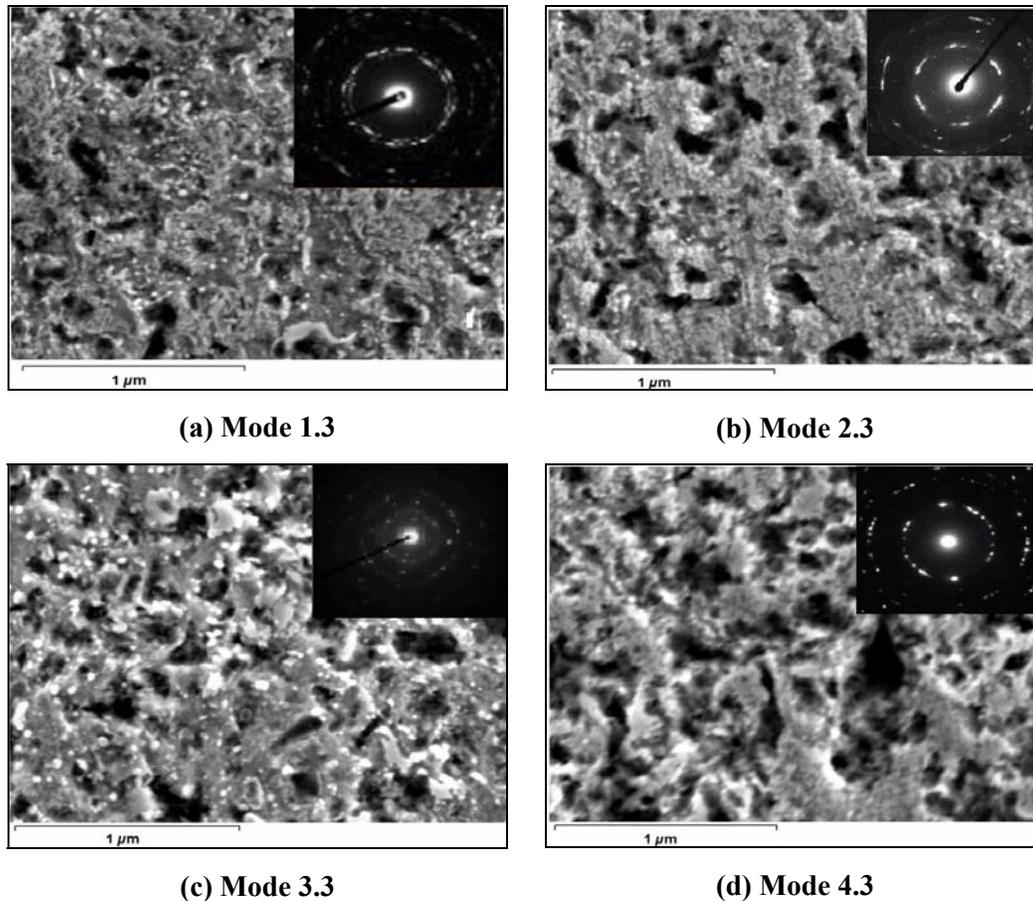
grains reaches 280 nm. An analysis of electron diffraction pattern showed that the structure contains both high and low-angle boundaries, azimuthal blur point reflections indicates an increase in stress (Fig. 6, *a, b*).



**Fig. 5: The microstructure of VT6 titanium alloy after rolling in the helical rolls and on LWM**

After rolling in the helical rolls by mode 3.3 and 4.3, the homogeneous and equiaxial structure form in the longitudinal section of the workpiece, while in the cross-section of the workpiece its elongation retained in the direction of bending (Fig. 6, *c, d*). At the same time it is clear that there is a further grinding of grain-subgrain structure. In the transverse cross-section of the blank, the grains with a pronounced substructure are extended along the bending direction, while in the longitudinal cross-section they have an equiaxed shape with an average size of about 120-140 nm. The diffraction pattern is characterized by the UFG-state with a predominantly high-angle boundaries. In the border areas of the grains structural

states with high values  $\chi_{ij}$  ( $\chi_{ij} = 25\text{-}34$  degrees/ $\mu\text{m}$ ) is formed. The share of such boundaries is about 85%. The dislocation density is very high and it is not possible to calculate the value according to the image of the structure.



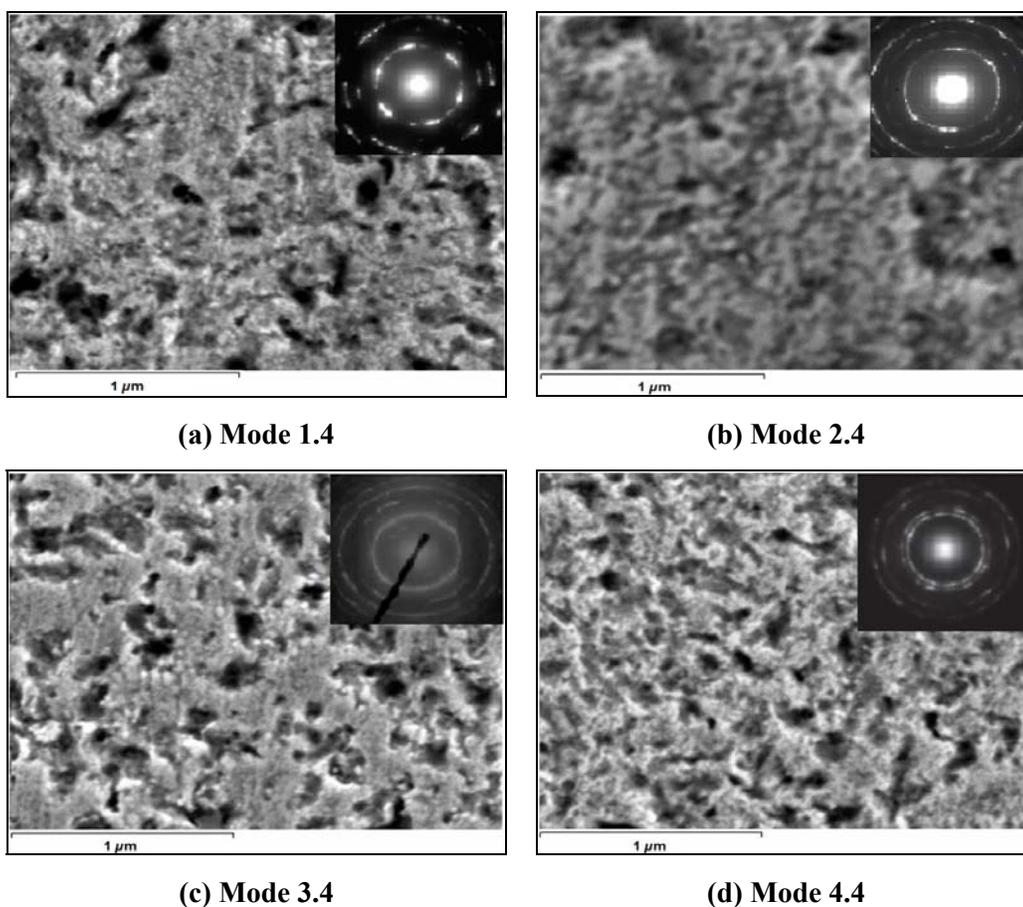
**Fig. 6: The microstructure of VT6 titanium alloy after rolling in the helical rolls, longitude-wedge mill and helical rolls**

It should be noted that with the increase of deformation degree in the subsequent rolling passes in the helical rolls, the grinding structure is not only result of twinning, but it also occurs because of the formation of porous substructures, which in their turn is a result of a slip dislocations processes. At high degrees of accumulated deformation, borders of the former twins and subgrains transfer into the high-angles.

To investigate the effect of rolling on the formation of the microstructure of titanium alloy VT6, strips, which have been rolled in the helical rolls at a temperature of 650°C, then

was rolled on the LWM at the temperature of 550°C (Fig. 7). It is seen that rolling at a temperature of 550°C greatly affectson the alloy microstructure. The microstructuresof VT6 titanium alloy, rolled by modes 1.4 and 2.4, are characterized by a nanostructured grain size (Fig. 7, a, b). The average size of subgrainsare 130-160 nm. After rolling on the LWM the structure is substantially nonequilibrium, boundaries of the structure elements are blurred, there is a large amount of the extinction contours, significant azimuthal blur of the reflections on the electron diffraction pattern. All this sustainthe high level of internal stresses in the grains.

Electron diffraction patterns for these structures have a quasi-ring features (Fig. 7, a, b, inset). On the rings some of the reflexes are well observable, their distribution along the ring indicates the presence of high angle misorientation between the fragments.



**Fig. 7: The microstructure of VT6 titanium alloy after rolling in the helical rolls, on the longitudinal wedge mill, in the helical rolls and on the longitudinal wedge mill**

Rolling on LWM of the workpiece, which was deformed according to modes 3.4 and 4.4, leads to the formation of nanostructures with the size of 80-100 nm (Fig. 7,c, d). The resulting nanosized structure is characterized by the homogeneity of the grain size along the entire volume of the rolled strip. The images of the microstructure after rolling on the LWM show a clear picture of the grain boundaries. Reflexes were uniformly arranged along the concentric circles in the diffraction patterns, indicating the formation of grains predominantly with high-angle boundaries (Fig. 7, c, d). In such boundaries, the crystal lattice is continuously changing orientation and, as a result, the structural states with high values  $\chi_{ij}$  ( $\chi_{ij} = 26-32$  degrees/ $\mu\text{m}$ ) is formed in the grain boundary regions. The share of such boundaries is about 95%.

Thus, the evolution of VT6 titanium alloy structure during rolling process in the helical rolls and on the LWM occurs in the following order:

- Formation of the deformation substructure (dislocation and twin) with a band width of 500-600 nm;
- Forming of cross borders inside the strips, increase of internal stresses and distortions of the original crystal lattice;
- Development of the primary recrystallization with the formation of ultrafine grain structure with the size of 250-270 nm;
- Development of microstrip substructure with constant thinning of structural elements up to 80-100 nm.

## CONCLUSION

Results of the study of evolution of the microstructure of long work pieces at various stages of their production have shown the possibility of obtaining bands with nanoscale structures using severe plastic deformation. The main role in the technological scheme of processing flow sheet belongs to rolling in the helical rolls, as a result of which an intensive grinding structure is reached. Saving enough plasticity of ultrafine grain titanium after rolling in the helical rolls allows to carry out shaping operations on the LWM, which contribute to a further refinement of the grain structure of the work piece, and the formation of the high dislocation density. As a result, in the sheet material of titanium alloy ensures a uniform formation of the nano-sized structure with a grain size of about 40-60 nm, which will increase the strength properties of the alloy and will lead to preservation of good ductility.

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