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Phase composition, structure and microhardness of the VT23 titanium alloy after deformation in a Bridgman chamber

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Abstract: The authors have studied for the first time the phase composition, microhardness and fine structure of the VT23 (α+β)-titanium alloy, with stable and metastable β-phase, after torsional deformation in a Bridgman chamber under a pressure of 4 GPa at room temperature. It has been found that the alloy microhardness, depending on the true degree of deformation under high hydrostatic pressure, changes along a curve with a maximum. The role of stress-induced βm→α" martensitic transformation in the formation of alloy structure, and microhardness under high-pressure torsion was revealed. The highest microhardness of the alloy with stable β-phase was 395 HV 0.05, and with metastable – 470 HV 0.05. At the same time, the maximum microhardness of metastable alloy, compared to stable alloy, was shifted to the region of lower true strain e=2.6. Using X-ray diffraction analysis, and transmission electron microscopy methods, made it possible to trace the evolution of alloy structure under high-pressure deformation consisting in grinding of α-, and α"-phase plates compared to the quenched state, as well as in the development of deformation βm→α", and α"→βm martensitic transformations. An increase in the degree of deformation by high-pressure torsion to e=7.7...7.9, regardless of the deformation stability of the β-phase, leads to a decrease in the alloy microhardness to a level of 185...205 HV 0.05. This is associated with the development of the dynamic recrystallisation process, and the formation of equiaxed α-phase nanoparticles with a size of 20...50 nm. The differences in the loading-unloading curves revealed by kinetic indentation, corresponded to the nature of the change in the VT23 alloy microhardness, depending on the quenching temperature and the true deformation degree.

Keywords: VT23 titanium alloy; phase composition; Bridgman chamber; high-pressure torsion; true deformation degree; metastable β-phase; martensitic transformations.

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INTRODUCTION

The strength of titanium alloys for structural purposes can be effectively increased by means of thermomechanical treatment, using methods of severe plastic deformation (SPD), or “megaplastic deformation” (MD) [1–3]. The high level of strength properties of these materials while maintaining a sufficient reserve of plasticity and ductility, is determined by the formation of an ultrafine-grained (UFG) or nanocrystalline microstructure, with predominantly high-angle grain boundaries (HAB) under intense deformation [3]. Among the known SPD methods, such as equal channel angular pressing, multi-axial isothermal forging, accumulative pack rolling with layer bonding (Accumulative Roll Bonding – ARB process), allowing increasing significantly the strength properties of metallic materials, due to the formation of UFG, and nanocrystalline structure while maintaining sufficient plasticity [4], the method of torsion (shear) under pressure has become widespread. Such deformation treatment, with the imposition of high hydrostatic pressure, due to the implementation of the “softest” stress-

strain state, allows achieving very high true deformation degrees ($e \approx 8$) without destroying the samples [5; 6]. According to the data of [4], high-pressure torsion at room temperature allows increasing significantly the tensile strength of VT1-00 pure titanium, and Ti–6Al–4V (VT6) alloy to the level of σ_u=1200 MPa and σ_u=1750 MPa, respectively due to the creation of an UFG and nanocrystalline structure with a grain size of up to 80–100 nm. To achieve the best combination of strength and plastic properties of titanium, and its alloys after SPD torsion under pressure, it is advisable to use additional short-term low-temperature annealing at temperatures of 300–400 °C. In a number of titanium alloys, after high-pressure torsion, as a result of β→ω- and partial α→ω-transitions, the formation of an embrittling ω-phase characteristic of high-pressure deformation in an amount from tenths to tens of percent was recorded, and the possibility of developing a reverse ω→α-transformation during SPD was found [7; 8].

In [9], when studying the features of phase and structural transformations in metastable titanium alloys under SPD conditions, it was found that during high-pressure torsion of the metastable Ti-5553 alloy (Ti–5Al–5V–5Mo–3Cr), the grain microstructure refinement to d<50 nm is achieved, due to the crushing of the initial β-grains by plates of the forming stress α"-martensite. When high (critical) deformation degrees are reached, the β-phase is stabilised, with respect to the formation of deformation martensite and the reverse α"→β-martensite transformation develops. The authors of [10] revealed a change in the shape and dispersion of strengthening precipitates, as well as an increase in the microhardness of the aged metastable Ti–15Mo alloy after high-pressure torsion deformation, compared to the undeformed state. Formation of stress-assisted α"-martensite in titanium alloys with deformation-metastable β-phase, activates the processes of dispersion hardening, and promotes additional strengthening during subsequent annealing (aging) [3].

One should note that a comparative study of the effect of SPD in Bridgman anvils, on the structure and mechanical properties of domestic two-phase titanium alloys in a stable and metastable state has not been previously conducted.

The aim of the work is to study the effect of the accumulated degree of deformation by compression and torsion under pressure on the microhardness, phase composition and fine microstructure of the domestic two-phase VT23 alloy in a stable and metastable state with respect to plastic deformation.

METHODS

The initial material was two-phase VT23 titanium alloy (Ti–5Al–5V–2Mo–Cr) produced by PJSC VSMPO-AVISMA Corporation (Russia). The chemical composition of the VT23 titanium alloy was determined using a NITON XL2 980 GOLDD X-ray fluorescence spectrometer (Table 1), and complied with OST1 90013-81.

The blanks of VT23 titanium alloy in the as-delivered condition after annealing at 750 °C, were quenched from temperatures of 800 and 860 °C in water, in order to form different β-phase stability, since according to the data [11; 12], after quenching from 800 °C, the β-phase is in a state stable with respect to mechanical loading, and after quenching from 860 °C, it is in a metastable state.

The process of intensive plastic (megaplastic) deformation of monolithic samples of VT23 titanium alloy with a height of 0.5 mm and a diameter of 10 mm, was carried out at room temperature in a Bridgman chamber by compression, under a pressure of 4 GPa followed by torsion. Steel anvils with a contact pad diameter of 10 mm were used in the work. Torsion under a pressure of 4 GPa was carried out with the lower anvil rotating at a speed of ω=0.3 rpm. The rotation angle φ varied within the range from 0 to 1080° (0...3 revolutions). The true accumulated deformation was estimated using the relationship given in [13]:

$$e = \ln\left(1 + \frac{\varphi^2 r^2}{h_0^2}\right)^{1/2} + \ln\left(\frac{h_0}{h_K}\right),$$

where φ is the rotation angle during torsion;
r is the disk radius;
h_0 is the initial disk thickness;
h_K is the disk thickness after deformation.

Kinetic microindentation was performed on a Fischerscope HM2000 XYm measuring system (Germany), using a Vickers indenter and WIN-HCU software at a maximum load of 0.005 N. The measurement error did not exceed 2 %. The measurements were taken at the middle of the sample radius. X-ray phase analysis of the samples was performed on a DRON-3 diffractometer in Co-Kα radiation, in the angle range of 25–105° with a step of 0.05°. Electron microscopic analysis of the microstructure of VT23 titanium alloy was carried out by transmission electron microscopy (TEM) on a JEOL JEM-2100 plus microscope (Japan) at an accelerating voltage of 80 keV. Preparation of thin foils for research included cutting out blanks 300...500 μm

Table 1. *Chemical composition of the VT23 alloy, wt. %*
Таблица 1. *Химический состав сплава ВТ23, мас. %*

Element	Ti	V	Al	Mo	Cr	Fe	Si	Zr
Content	85.870	4.780	4.855	1.865	1.305	0.800	0.150	0.025

thick, with a thin abrasive disk under water cooling conditions, mechanical processing to a thickness of 100 μm, and subsequent electropolishing in a methyl electrolyte at a temperature no higher than −50 °C.

RESULTS

The microhardness of the VT23 alloy after quenching from temperatures of 800 and 860 °C was 132 HV 0.05 and 48 HV 0.05, respectively (Fig. 1). As can be seen from Fig. 1, the microhardness of the VT23 alloy samples, depending on the degree of true plastic deformation *e* in Bridgman anvils, changes along a curve with a maximum. A significant increase in the alloy microhardness, compared to the initial state, occurs already at the initial stage of deformation at a value of *e*=1.5 under compression, under a pressure of 4 GPa without torsion. In this case, the microhardness of the sample of the alloy with a metastable β-phase, quenched from a temperature of 860 °C, grows more intensively compared to the alloy with a stable β-phase after quenching from 800 °C and reaches a maximum (470 HV 0.05), at a true deformation of *e*=2.6. The maximum microhardness of the alloy with a stable β-phase (395 HV 0.05) is observed after torsion under pressure upon reaching a higher degree of true deformation *e*=5.4. The microhardness of the alloy, regardless of the quenching temperature, and accordingly, the deformation stability of the β-phase at the maximum degree of true deformation *e*=7.7…7.9, is approximately at the same level – 185…205 HV 0.05.

The loading – unloading curves of the samples of the alloy with a stable and metastable β-phase, in the initial state, have characteristic differences (Fig. 2). At deformation degrees from *e*=1.6 to 4.5…4.7, the loading curve of the alloy with a metastable β-phase, has a smoother increase in stress with deformation, which is associated with the martensitic transformation. Further, with an increase in the degree of true deformation from *e*=5.2...5.4 to 7.7...7.9, the difference in the position of the curves gradually decreases, and at a degree of deformation of 7.7...7.9 they practically coincide, which is associated with the stabilisation of the β-phase.

The quantitative phase composition of the studied samples is given in Table 2. X-ray phase analysis showed that after deformation the alloy has a three-phase (α+β+α") state (α-phase with a hcp lattice, β-phase with a bcc lattice and α"-phase with orthorhombic lattice), and the ω-phase, the formation of which is possible at pressures above 2 GPa, was not recorded in the diffraction patterns in the angle range of 20…105° (Fig. 3). As can be seen from Fig. 3, the lines of the α-phase (100), (110), (112) broaden as the deformation degree increases, and their intensity decreases, which indicates the refinement of α-crystallites, and the presence of internal microstresses as a result of plastic deformation. A redistribution of the integral intensities between the X-ray peaks of the α/α"- and β-phase is also observed, which indicates both the texture formation, and the phase transformation of the metastable phase βm→α".

The study of the fine microstructure of the alloy, quenched from 800 °C by the TEM method (Fig. 4), revealed the presence of α-plates with a thickness of 150...250 nm located in the β-matrix, corresponding to the crystallographic relationship [110]β || [001]α. Reflections from the α"-phase formed in the β-matrix during quenching were also revealed in the microdiffraction patterns of the quenched samples. Thin and distinct interphase boundaries indicate a high degree of coherence of these phases. After quenching from a temperature of 860 °C, a complex tweed contrast was found when studying the β-matrix (Fig. 4 b), which indicates a reduced stability of the β-phase with respect to martensitic transformations caused by stress.

TEM study of the microstructure of the VT23 alloy, quenched from 800 °C after SPD with a degree of *e*=1.6, showed the presence of fragments of the initial α-phase plates of varying thickness from 50 to 250 nm with β-phase interlayers (Fig. 5 a). The plates have an irregular shape,

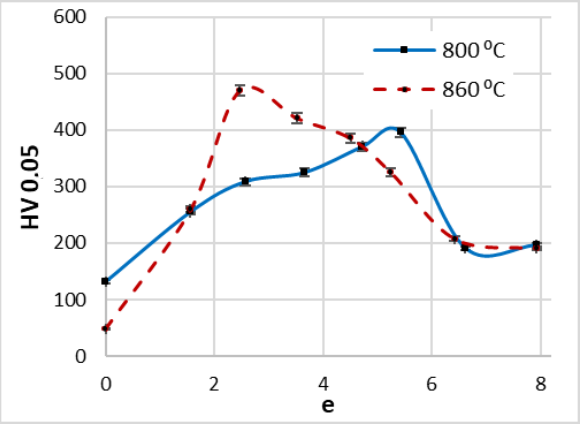


Fig. 1. *Effect of true deformation degree e during torsion under pressure on the VT23 alloy microhardness*

Рис. 1. *Влияние истинной степени деформации e в процессе кручения под давлением на микротвердость сплава ВТ23*

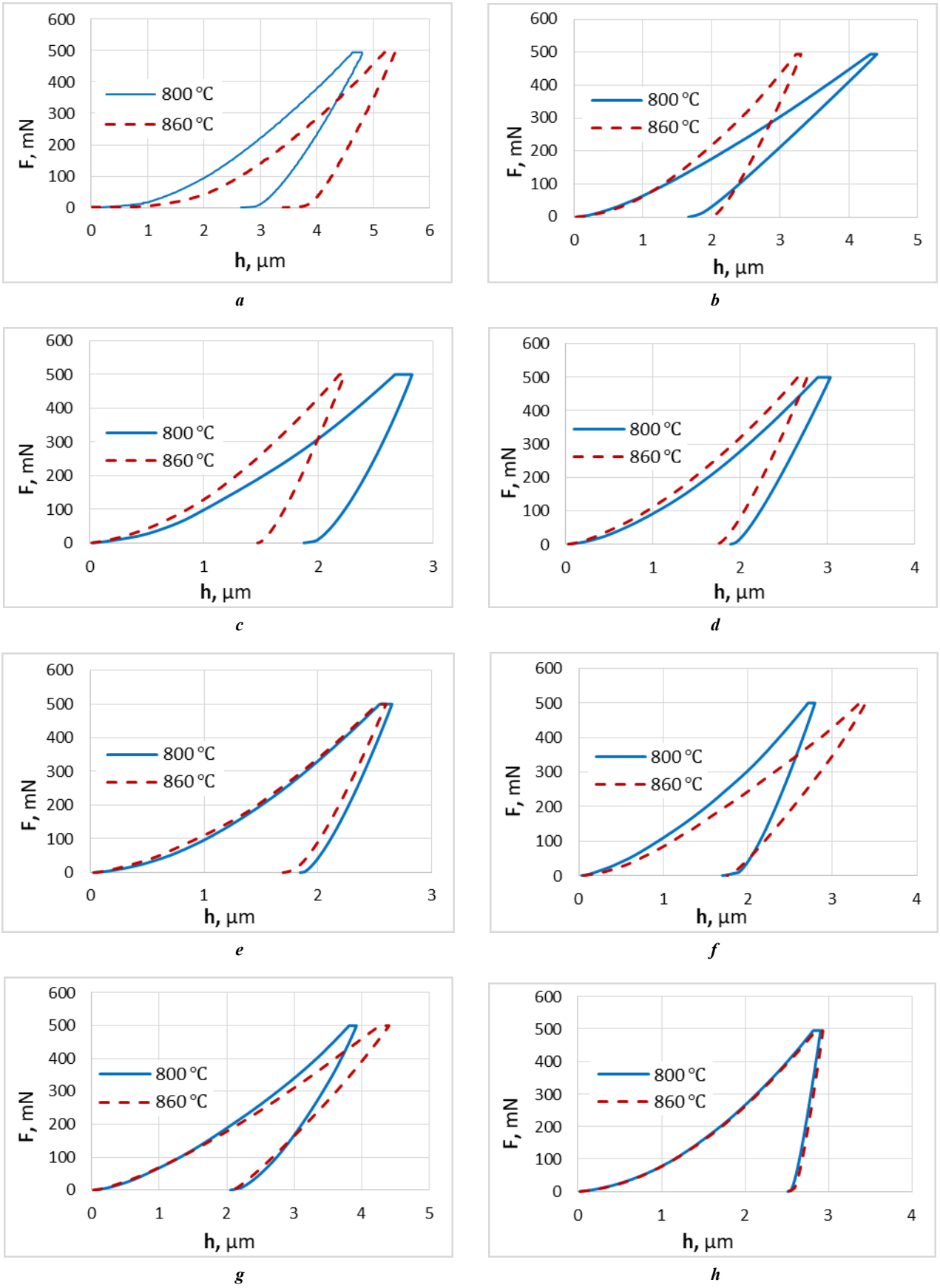


Fig. 2. *Loading – unloading curves of VT23 alloy samples after various degrees of deformation under pressure:* ***a*** *– without deformation;* ***b*** *– e=1.6;* ***c*** *– e=2.5…2.6;* ***d*** *– e=3.5…3.7;* ***e*** *– e=4.5…4.7;* ***f*** *– e=5.2…5.4;* ***g*** *– e=6.4…6.6;* ***h*** *– e=7.7…7.9*

Рис. 2. *Кривые нагружения – разгружения образцов сплава ВТ23 при различных степенях деформации под давлением:* ***a*** *– без деформации;* ***b*** *– e=1,6;* ***c*** *– e=2,5…2,6;* ***d*** *– e=3,5…3,7;* ***e*** *– e=4,5…4,7;* ***f*** *– e=5,2…5,4;* ***g*** *– e=6,4…6,6;* ***h*** *– e=7,7…7,9*

Table 2. *Phase composition of the VT23 titanium alloy [11]*
Таблица 2. *Фазовый состав титанового сплава ВТ23 [11]*

Heat treatment conditions	α, %	β/βm, %	α", %
Quenching at 800 °C	32	50	18
Quenching at 860 °C	7	8	85

strongly distorted areas and a high density of the dislocation structure. This indicates their deformation origin, resulting from deformation fragmentation of structural elements. In the sample quenched from a temperature of 860 °C with a metastable β-phase, the processes of deformation by compression under pressure, with a degree of e=1.6 are characterised mainly by phase transformations, caused by deformation of the metastable phase βм→α" (Fig. 5 b). The TEM structure is represented by fragments of plates of the initial α-phase with a thickness of 50...250 nm, as well as by more dispersed particles of α"-martensite (stress) (Fig. 5 b).

In the electron diffraction patterns of the samples, deformed with a degree of e=7.7...7.9, a significant number of reflections located along the circumference are observed (Fig. 6, 7), which indicates the presence of multiple crystalline orientations associated with grain refinement. The presence of an insignificant amount (up to 5 %) of the high-pressure ω-phase, which was not detected by the X-ray phase analysis method, was recorded. A TEM study of the microstructure of the alloy quenched from 800 °C after deformation, with a degree of e=7.7, revealed the presence of equiaxed grains with a weak dislocation contrast, with a diameter of 20…30 nm and smaller grains up to 20 nm of irregular shape, with a characteristic banded contrast (Fig. 6). In the TEM images of the alloy quenched from 860 °C, larger homogeneous particles, and particles with a banded contrast with a size of 30…50 nm were recorded (Fig. 7).

DISCUSSION

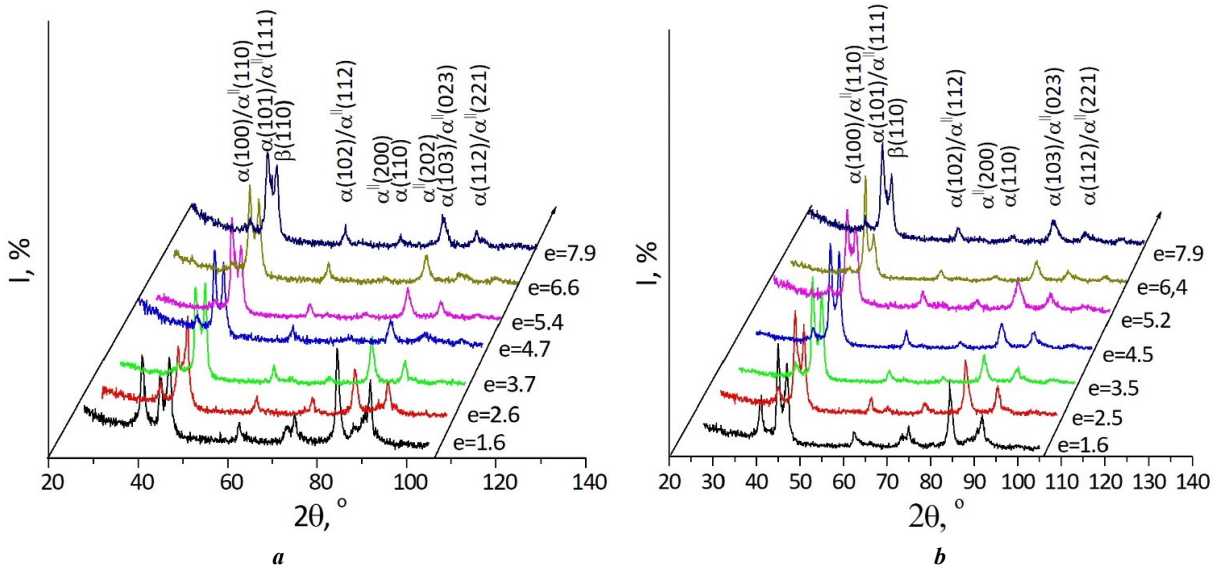
The difference in the nature of the curves of microhardness change, depending on the degree of deformation of the samples after quenching, from temperatures of 800 and 860 °C (Fig. 1 a), can be explained by their phase composition (Table 2). Thus, the smoother nature of the growth of microhardness of the sample quenched from 800 °C is associated with a gradual increase in the density of dislocations in the alloy crystalline structure, the refinement of the α-phase plates and athermal α"-martensite. In accordance with the Hall–Petch dependence, smaller grains contribute to an increase in microhardness [14]. An increase in the amount of athermal α"-martensite, which has a more dispersed microstructure compared to the initial α-phase [11], in the sample quenched from a temperature of 860 °C, contributes to a more active growth of microhardness, since the large surface area of the martensite plates compared to large α-phase plates, allows them to interact with a large number of dislocations, creating obstacles to their movement, and increasing the strengthening effect [15]. Moreover, the β-phase in the alloy quenched from a temperature of 860 °C, which is in a metastable state, with respect to mechanical loading [12], undergoes a martensitic transformation β→α" during torsional deformation with the formation of stress-induced martensite, which increases the alloy microhardness. It should also be noted that the crystalline structure of athermal martensite, as well as stress-induced martensite, is strongly distorted compared to the initial α- and β-phases, which leads to a high dislocation density and internal stresses (Fig. 4).

The softening of the alloys in a stable and metastable state after quenching from 800 and 860 °C is associated with the process of low-temperature dynamic, recrystallisation occurring at high degrees of plastic deformation, described in [2]. The Vickers microhardness decreases due to a decrease in the dislocation density, and the formation of equiaxed grains.

The sharp difference in the loading-unloading curves of the initial samples (Fig. 2 a) can be explained by the presence of a metastable βm-phase. Metastable phases can have a high resistance to initial deformation, which leads to a longer loading curve before the onset of significant plastic flow [16] (Fig. 2 a). The coincidence of the loading-unloading curves at a deformation degree of 7.7...7.9, indicates that the processes of decomposition of metastable phases and dynamic recrystallisation were fully realised (Fig. 2 h).

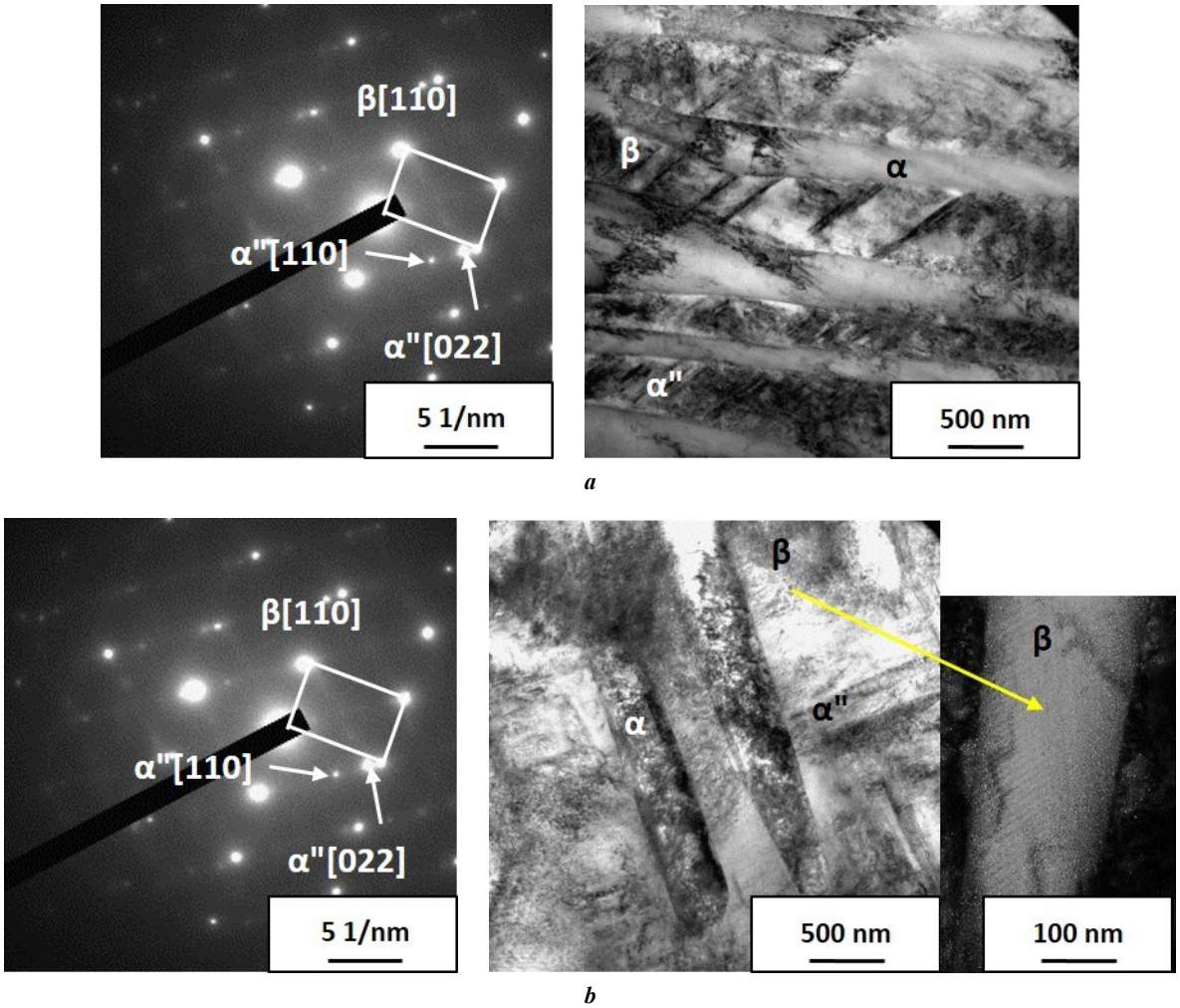
Analysis of TEM images at deformation degrees of 7.7...7.9 (Fig. 6, 7) showed that the formation of equiaxed grains without deformation contrast, is associated with low-temperature dynamic recrystallisation of the α phase, described in [2; 17]: the formation of high-angle boundaries during deformation, leads to the appearance of new grains by the continuous recrystallisation mechanism, i. e. due to the increase in the misorientation of sub-boundaries. Particles with banded contrast, according to [5], are deformation fragments, formed during the subsequent deformation of recrystallised grains. The larger size of recrystallised grains in the alloy quenched from 860 °C is associated with the fact that the processes of low-temperature dynamic recrystallisation of the alloy in the deformation-metastable state, occurred more completely during torsional deformation under pressure.

Therefore, based on the conducted study, the authors revealed the extreme nature of the change in the VT23 alloy microhardness, with an increase in the true degree of torsional deformation under pressure associated with the gradual development of the processes of the structure dispersion, and subsequent dynamic recrystallisation, as well as differences in the level of microhardness maximum, and the corresponding degrees of deformation for the alloy with a stable and deformation-metastable β-phase.



a *b*

Fig. 3. *Diffraction patterns of VT23 alloy samples:*
a *– after quenching from 800 °C and torsional deformation under pressure;*
b *– after quenching from 860 °C and torsional deformation under pressure*
Рис. 3. *Дифрактограммы образцов сплава ВТ23:*
а *– после закалки от 800 °С и деформации кручением под давлением;*
b *– после закалки от 860 °С и деформации кручением под давлением*



a

b

Fig. 4. *TEM image of the microstructure of VT23 alloy after quenching:* ***a*** *– from 800 °C;* ***b*** *– from 860 °C*
Рис. 4. *ПЭМ-изображения микроструктуры сплава ВТ23 после закалки:* ***а*** *– от 800 °С;* ***b*** *– от 860 °С*

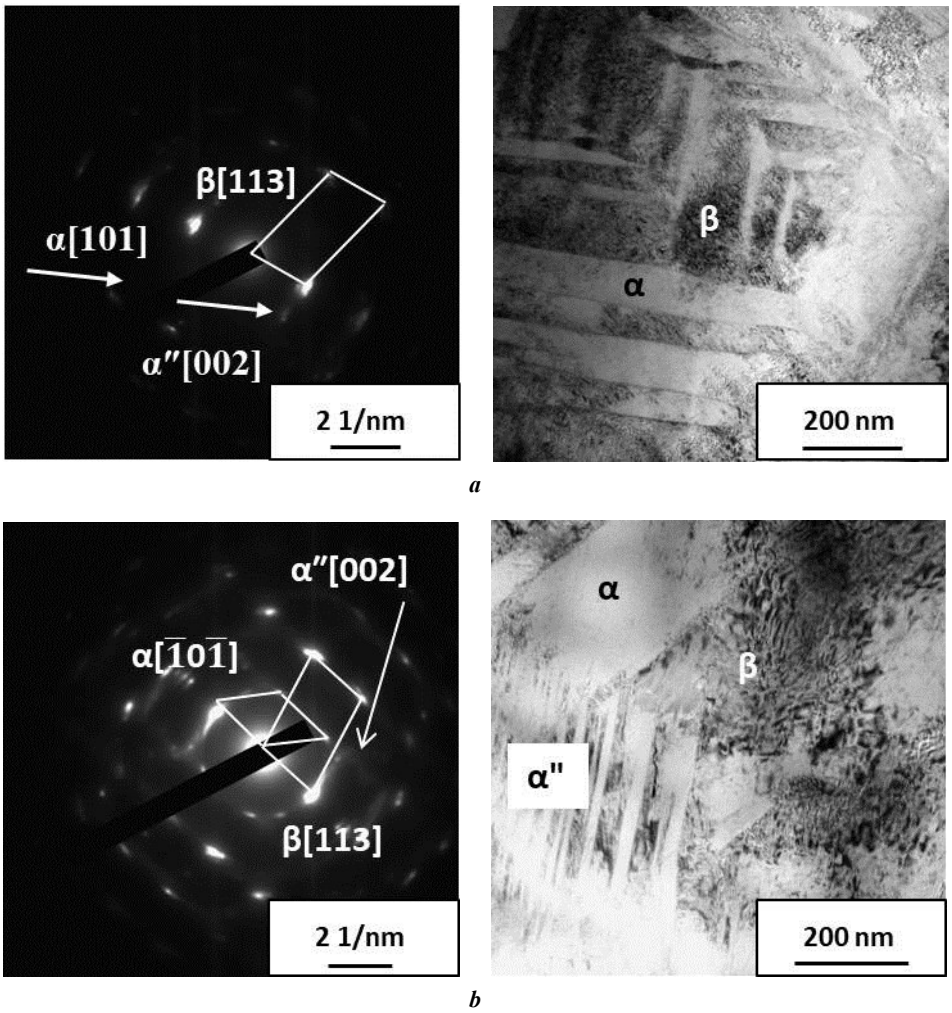


Fig. 5. *TEM images of VT23 titanium alloy:*
a *– after quenching from 800 °C and SPD, e=1.6;* ***b*** *– after quenching from 860 °C and SPD, e=1.6*
Рис. 5. *ПЭМ-изображения титанового сплава ВТ23:*
а *– после закалки от 800 °С и ИПД, е=1,6;* ***б*** *– после закалки от 860 °С и ИПД, е=1,6*

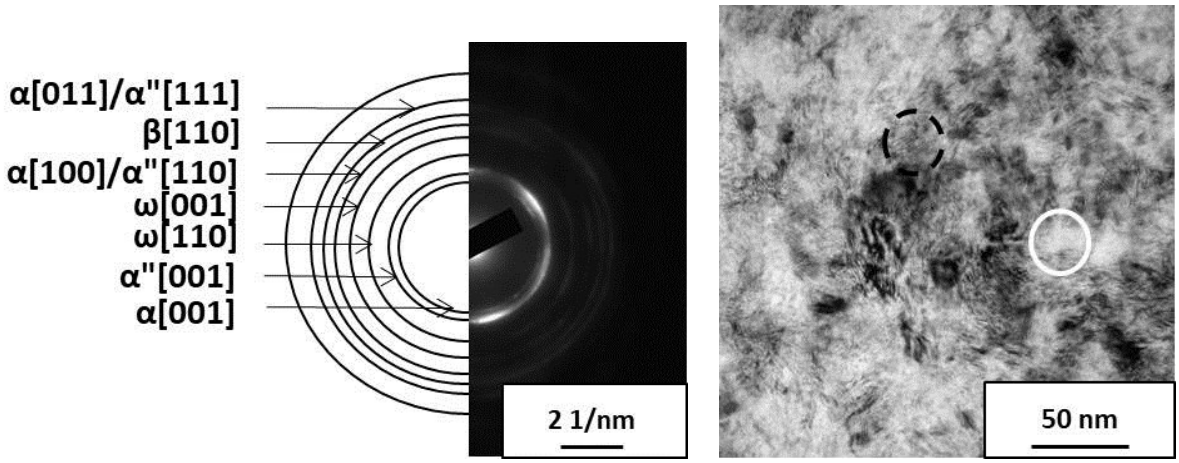
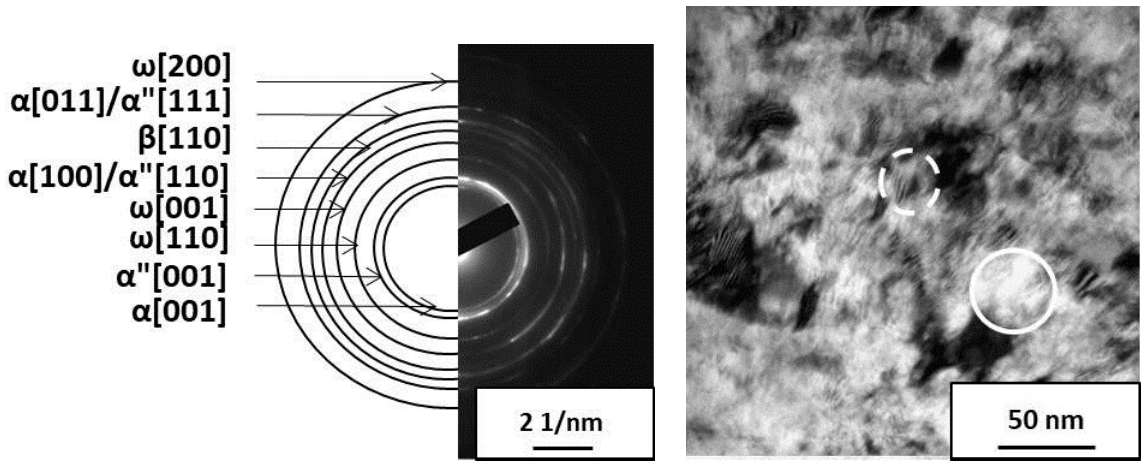


Fig. 6. *TEM images of VT23 titanium alloy after quenching from 800 °C and torsion under pressure, e=7.7.*
The solid line highlights α-phase equiaxed particles with weak dislocation contrast,
and the dotted line highlights those with banded contrast
Рис. 6. *ПЭМ-изображения титанового сплава ВТ23 после закалки от 800 °С и кручения под давлением, е=7,7.*
Сплошной линией выделены равноосные частицы α-фазы со слабым дислокационным контрастом,
пунктирной – с полосчатым контрастом



b

Fig. 7. *TEM images of VT23 titanium alloy after quenching from 860 °C and torsion under pressure, e=7.9. The solid line highlights α-phase equiaxed particles with weak dislocation contrast, and the dotted line highlights those with banded contrast*

Рис. 7. *ПЭМ-изображения титанового сплава ВТ23 после закалки от 860 °С и кручения под давлением, e=7,9. Сплошной линией выделены равноосные частицы α-фазы со слабым дислокационным контрастом, пунктирной – с полосчатым контрастом*

CONCLUSIONS

A nonlinear dependence of the alloy microhardness on the degree of true high-pressure torsional deformation was found. The maximum level of microhardness of samples quenched from temperatures of 800 and 860 °C was 395 HV 0.05 and 470 HV 0.05 at a deformation of 5.3 and 2.6, respectively. The increase in the microhardness of the sample quenched from 800 °C is associated with an increase in the dislocation density in the initial α- and β-phases, and refinement of the structural components. The greatest increase in the microhardness of the sample quenched from a temperature of 860 °C is associated with the intense strengthening of athermal α"-martensite and the phase transformation of metastable phases according to the schemes βm→α" and α"→βm. The subsequent softening of the alloy in the deformation range of e>5.4 for the sample quenched from 800 °C, and e>2.6 for the sample after quenching from 860 °C, estimated by the decrease in microhardness, occurs as a result of low-temperature dynamic recrystallisation of the α-phase. At the maximum value of the true deformation degree e=7.7...7.9, regardless of the quenching temperature, the alloy microhardness differed insignificantly, and corresponded to the level of 185...205 HV 0.05.

Using X-ray phase analysis, it was shown that during deformation in a Bridgman chamber, the alloy retains a three-phase (α+β+α") composition, at the degree of deformation from e=1.6 and more, there is a refinement of α- and α"-plates, as well as a phase transformation – the βm-phase decomposition during quenching from 860 °C according to the scheme βm→α".

At the deformation degree of e=1.6, a fragmentation of the α and α" plates occurs, as well as an increase in dislocation contrast. In a sample with a metastable βm-phase (quenching from 860 °C), it decomposes with the formation of dispersed needles of α"-stress martensite. An increase in the deformation degree to e=7.7…7.9 leads to the formation of equiaxed α-particles with a size of 20…30 nm (quenching from 800 °C), and 30…50 nm (quenching from 860 °C) due to the processes of low-temperature dynamic recrystallisation and deformation fragments with a banded contrast.

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Фазовый состав, структура и микротвердость титанового сплава ВТ23 после деформации в камере Бриджмена

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Аннотация: Впервые изучены фазовый состав, микротвердость и тонкая структура (α+β)-титанового сплава ВТ23 со стабильной и метастабильной β-фазой после деформации в камере Бриджмена кручением под давлением 4 ГПа при комнатной температуре. Установлено, что микротвердость сплава в зависимости от истинной степени деформации в условиях высокого гидростатического давления меняется по кривой с максимумом. Выявлена роль инициированного напряжением βм→α" мартенситного превращения в формировании структуры и микротвердости сплава при кручении под давлением. Наибольшая микротвердость сплава со стабильной β-фазой составила 395 HV 0,05, а с метастабильной – 470 HV 0,05. При этом максимум микротвердости метастабильного сплава по сравнению со стабильным был смещен в область меньшей истинной деформации e=2,6. Использование методов рентгенофазового анализа и просвечивающей электронной микроскопии позволило проследить эволюцию структуры сплава при деформации под давлением, заключающуюся в измельчении по сравнению с закаленным состоянием пластин α- и α"-фаз, а также в развитии деформационных βм→α" и α"→βм мартенситных превращений. Увеличение степени деформации кручением под давлением до e=7,7...7,9 независимо от деформационной стабильности β-фазы приводит к снижению микротвердости сплава до уровня 185…205 HV 0,05, что связано с развитием процесса динамической рекристаллизации и формированием равноосных наночастиц α-фазы размером 20…50 нм. Выявленные при кинетическом индентировании различия в кривых нагружения – разгружения соответствовали характеру изменения микротвердости сплава ВТ23 в зависимости от температуры закалки и степени истинной деформации.

Ключевые слова: титановый сплав ВТ23; фазовый состав; камера Бриджмена; кручение под давлением; истинная степень деформации; метастабильная β-фаза; мартенситные превращения.

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