

Principle conclusions

It was shown that in the studied metaloceramic alloys on the basis of tungsten monocarbide the coefficients of elasticity decrease steadily at increase of cobalt concentration. Alloy model in the form of isotropic mixture of anisotropic phases describes qualitatively correctly dependence of elastic characteristics of metal ceramics on composition. The computed values of Young modulus and shift coincide well with the measured ones. Gamma irradiation by portions to 10^5 Gy decreases the

level of internal friction in metaloceramic alloys, increases boundary amplitudes of periodical deformation starting with which ultrasonic decay obtains amplitude-dependence character. The reason of this may be both formation of reflections of new carbide phase under the influence of irradiation and structural changes in alloy impeding motion of grain-boundary dislocations. Such character of internal friction change allows supposing that γ -irradiation changes conditions of plastic flow in materials at initial stages of deformation.

REFERENCES

- Zuev L.B., Ulyanov V.L., Botaki A.A. Physical acoustics of solids. – Tomsk: TPU Press, 2006. – 166 p.
- Nikanorov S.P., Kardashev B.K. Elasticity and dislocation inelasticity of crystals. – Moscow: Nauka, 1985. – 250 p.
- Botaki A.A., Ulyanov V.L., Sharko A.V. Ultrasonic check of strength properties of structural materials. – Moscow: Mashinostroenie, 1983. – 80 p.
- Belomestnykh V.N., Pokholkov Yu.P., Ulyanov V.L. et al. Elastic and acoustic properties of ionic, ceramic dielectrics and high temperature superconductors. – Tomsk: STT Press, 2001. – 224 p.
- Tretyakov V.I. Grounds of metallurgy and technology of sintered hard alloy production. – Moscow: Metallurgiya, 1976. – 532 p.
- Chernov I.P., Timoshikov Yu.A., Mamontov A.P. et al. Change of structure of VK alloy under the influence of small portions of γ -radiation // Atomic energy. – 1984. – V. 57. – № 1. – P. 58–60.
- Pozdeeva E.V., Botaki A.A., Ulyanov V.L. Low-temperature measurements TKLR of electroceramic materials // Glass and ceramics. – 1978. – № 4. – P. 30–31.
- Ulyanov V.L., Botaki A.A., Pozdeeva E.V. Elastic and acoustic properties of ceramic dielectrics // Bulletin of the Tomsk Polytechnic University. – 2006. – V. 309. – № 2. – P. 30–31.
- Frantsevich I.I., Voronov F.F., Bakuta S.A. Elastic constants and coefficients of elasticity of metals and nonmetals: reference book. – Kiev: Naukova dumka, 1982. – 286 p.
- Van Buren. Crystal defects. – Moscow: Izd-vo inostrannoy literatury, 1962. – 534 p.

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STRUCTURAL-PHASE STATE AND MECHANICAL PROPERTIES OF SUBMICROCRYSTALLINE TITANIUM ALLOY Ti-6Al-4V OBTAINED WITH USE OF REVERSIBLE HYDROGEN ALLOYING

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Features of evolution of structural-phase state of titanium alloy Ti-6Al-4V at the process of submicrocrystalline structure formation using reversible hydrogen alloying have been investigated by methods of electron microscopic and X-ray diffraction analyses. Influence of hydrogen alloying on mechanical properties at stretching of submicrocrystalline titanium alloy Ti-6Al-4V in temperature interval of 293...1023 K was studied. Possible reasons of increase in ultimate and yield strength and reduction of deformation to destruction of submicrocrystalline alloy Ti-6Al-4V in temperature interval 873...1023 K at hydrogen alloying in quantity 0,08...0,33 mas. % were discussed.

1. Introduction

Formation of submicrocrystalline (SMC) structure (grain size is less than $0,5 \text{ }\mu\text{m}$) in titanium and its alloys improves significantly their operating characteristics [1]. One of the ways of obtaining SMC structure in titanium alloys is the method combining preliminary hydrogen treatment and hot plastic deformation by pressing [2]. In scientific literature there data according to which use of this method allows obtaining homogeneous SMC structure with grain size $d < 0,3 \text{ }\mu\text{m}$ in titanium alloys [2, 3]. It is known that hydrogen may result in fragility of titanium al-

loys at maintenance therefore, it is practically fully removed from alloy after hot working by pressing by vacuum annealing at temperatures 873...973 K. However, such annealing may cause the change of phase composition, recrystallization and growth of grains of SMC structure formed in alloy doped with hydrogen at hot pressing [4, 5] and thereby result in decrease of its strength and plastic properties. In this connection it is of interest to study evolution of structural phase state at degassing by annealing and influence of hydrogen residual concentration on mechanical properties of titanium alloys in submicrocrystalline state obtained by use of reversible hydrogen alloying.

2. Experimental technique

Ti-6Al-4V alloy hydrogen concentration in which amounted to 0,008 wt. % was used in this work as source material for investigation. To obtain SMC structure blanks with sizes $15 \times 15 \times 30$ mm³ were saturated with hydrogen in the device of Siverst type in drained hydrogen medium at temperature 1023 K to concentration 0,33 wt. %. Then at temperature 1023 K the blanks were deformed by compression by 80 %. Samples were degassed by annealing at pressure $5 \cdot 10^{-3}$ Ra and temperature 873 K. Hydrogen concentration in samples was measured by coulometric method of determining hydrogen mass fraction with $\pm 0,0001$ wt. % and by the method of precision weighting. The latter was used at hydrogen concentrations more than 0,1 wt. %.

Electron-microscopic examinations of thin foils were carried out in transmission electron microscope EM-125K. Dimensions of structural elements were measured by proper micrographs by secant method. Phase lattice parameters were determined by the method of X-ray diffraction analysis at diffractometer Shimadzu XRD6000 with accuracy 0,0001 nm.

To study mechanical properties the samples with working section sizes $5 \times 1,7 \times 0,7$ mm were cut of blanks by electric-spark method. Tensile tests of samples with different hydrogen content were carried out at the device PV-3012M with initial rate of deformation $6,7 \cdot 10^{-3}$ s⁻¹ in temperature range 293...1023 K. Before

testing a layer of thickness about 100 mkm was removed from sample surface by mechanical polishing and further electrolytic buffing.

3. Results and their discussion

Electron-microscopic image of structure and pattern of microdiffraction of Ti-6Al-4V-0,33H alloy after deformation by compression by 80 % is given in Fig. 1. At bright-field image (Fig. 1, a) complex deformative contrast does not allow detecting microstructure features. At dark-field image (Fig. 1, б) elements of grain-subgrain structure average size d_{cp} of which amounts to 0,085 m μ m are seen. Distribution of grain-subgrain structure by sizes is submitted to normal-logarithmic law (at histogram N/N_0 is the part of grains with the given grain size to general amount of grains) (Fig. 1, в). At electron diffraction patterns of SMC structures of Ti-6Al-4V-0,33H alloy (Fig. 1, а) taken of the area 1,2 m μ m² almost solid diffraction rings formed by reflections of single crystals are observed. In this case almost all reflections have azimuthal blurring. Such kind of electronograms indicates the presence of large angle disorientations between structure elements and presence of elastic stress in single grains.

Annealing of wrought Ti-6Al-4V-0,33H alloy in vacuum at temperature 873 K, 30 min results in partial degassing of alloy. Hydrogen concentration in alloy decreases to 0,08 wt. %. Electron-microscopic image of mic-

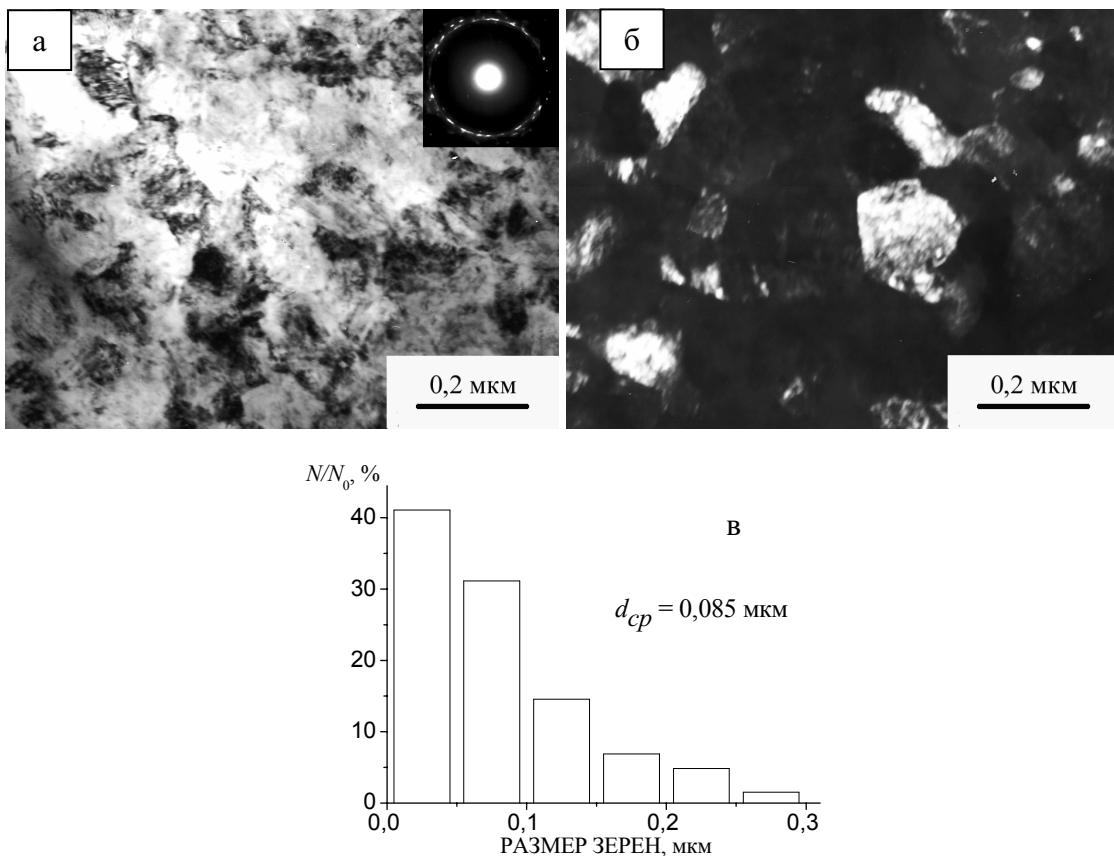
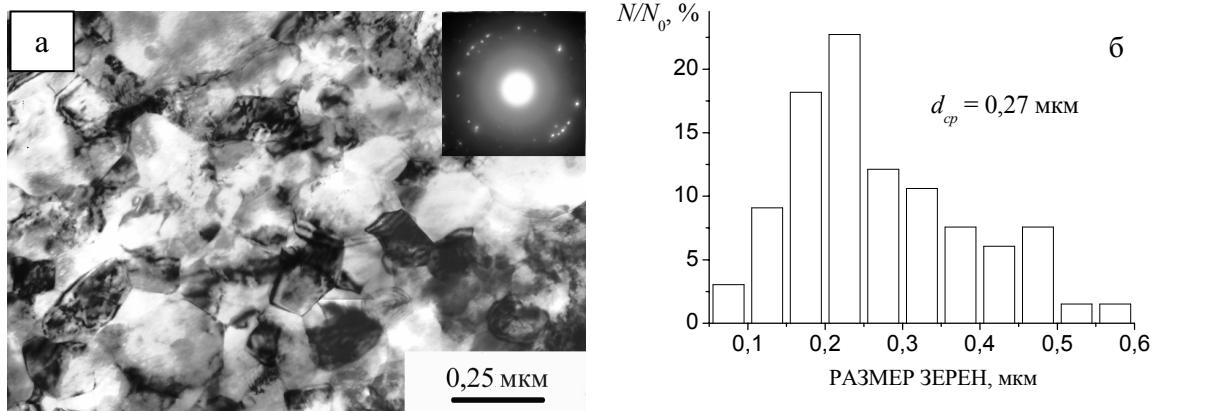
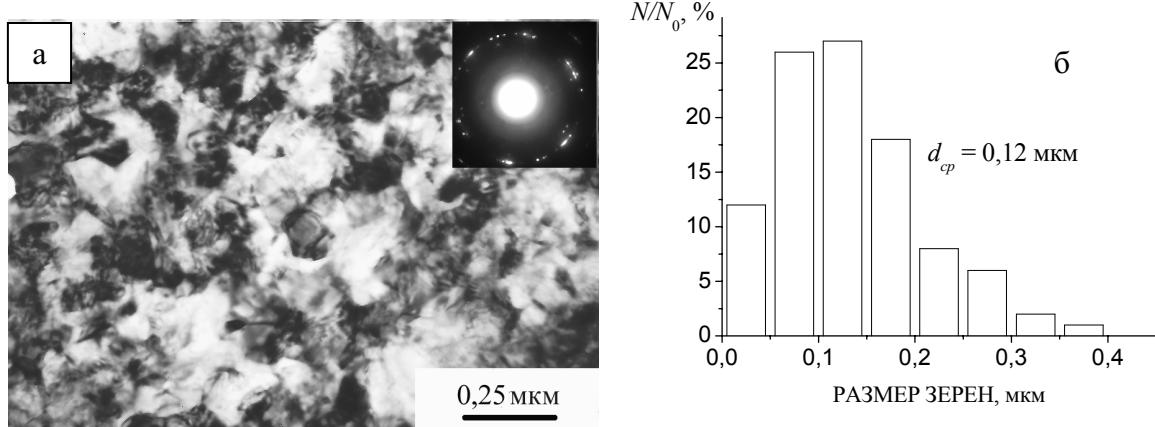


Fig. 1. Electron-microscopic image of structure of alloy Ti-6Al-4V-0,33H after deformation by compression by 80 % at $T=1023$ K: a) bright-field image, б) dark-field image and в) histogram of grain distribution by size. Размер зерен – Grain size



rostructure of Ti-6Al-4V-0,08H alloy is given in Fig. 2. It is seen that in this case alloy has homogeneous SMC structure. At electronograms of such structure taken of the area of $1,2 \text{ мкм}^2$ a large number of reflections situated round a circle is observed; only some of them have azimuthal blurring (Fig. 2, a). It is seen from histogram of size distribution of grain-subgrain structure of Ti-6Al-4V-0,08H alloy (Fig. 2, б) that the majority of elements of grain-subgrain structure has sizes less than $0,15 \text{ мкм}$. Average size of elements of grain-subgrain SMC structure determined by dark-field image amounts to $\sim 0,12 \text{ мкм}$.

Increase of alloy degassing time to 1 h results in decrease of hydrogen concentration in alloy up to $\sim 0,005$ wt. %. Submicrocrystalline structure in alloy remains after stated annealing (Fig. 3) however, its character and size of elements of grain-subgrain structure change. Considerable amount of reflections arranged uniformly round a circle is observed on electronograms of SMC structures of Ti-6Al-4V-0,005H alloy as well as Ti-6Al-4V-0,08H alloy. At the same time azimuthal blurring is almost absent in reflections that indicates a decrease of internal elastic stress. Band contrast typical for equilibrium state of grain boundary attends there. Histogram of size distribution of elements of grain-subgrain structure of Ti-6Al-4V-0,005H alloy has a charac-

ter close to bimodal one. The majority of elements of grain-subgrain structure has sizes $0,15\ldots 0,25 \text{ мкм}$. Average size of elements of grain-subgrain structure amounts to $\sim 0,27 \text{ мкм}$.

X-ray diffraction study showed that not only structure of Ti-6Al-4V-H alloy but phase composition as well change at degassing. At diffraction patterns of alloy Ti-6Al-4V-0,33H (Fig. 4, curve 2) there are reflections of only α phase with hexagonal lattice and parameters larger than proper parameters of alloy lattice in initial state (Table 1). Hydride extraction by X-ray spectrum analysis in alloy is not observed. It indicates the fact that after the stated treatment almost the whole hydrogen in Ti-6Al-4V-0,33H alloy is in solid solution. At diffraction pattern of Ti-6Al-4V-0,08H alloy (Fig. 4, curve 3) there are reflections of α and β -phases the parameters of lattice of which are larger than proper parameters of lattices α and β -phases of alloy in initial state (Table 1). Besides, the changes of ratio of reflection intensities (100), (002) and (101) of α -phase are observed at diffraction pattern. Change of given reflection intensities on which reflections (101), (110), (111) and (220) of hydrides $\text{TiH}_{1,2}$ may be overlapped indicates the presence of hydride extraction in volume of Ti-6Al-4V-0,08H alloy [6]. Diffraction pattern of Ti-6Al-4V-0,005H alloy (Fig. 4, curve 4) is practically identical to diffraction pattern of original al-

loy Ti-6Al-4V-0,008H and parameters of lattices of α and β -phases are close to proper parameters of initial alloy lattice (Table).

Table. Parameters of phase lattice in alloys Ti-6Al-4V-H

Hydrogen concentration, wt. %	Phase lattice parameters, nm		
	a_α	c_α	a_β
0,008	0,2921	0,4665	0,3208
0,005	0,2920	0,4664	0,3210
0,08	0,2925	0,4670	0,3273
0,33	0,2928	0,4676	—
0,33*	0,2924	0,4672	0,3297

*hardening from 973 K

Temperature dependences of ultimate strength σ_B , yield point $\sigma_{0,2}$ and range of deformation to failure δ for SMC alloys Ti-6Al-4V-H with different hydrogen content are given in Fig. 5. It is seen that for all studied alloys values σ_B and $\sigma_{0,2}$ change by a curve with minimum at temperature growth. Similarly, dependence $\delta\sigma_{0,2}$ on temperature is observed for coarse-grained titanium hydrogen doped alloys [7, 8]. However, for titanium alloys in coarse-grained state minimum on dependence curves σ_B and $\sigma_{0,2}$ on temperature is observed at temperatures 250...300 K higher than for SMC state. In paper [7] presence and position of minimum on dependence curve $\sigma_{0,2}$ on temperature in coarse-grained titanium hydrogen doped alloys is connected with strength balance of α and β -phases at specified hydrogen concentration. For SMC titanium alloys increase of σ_B and $\sigma_{0,2}$ at tem-

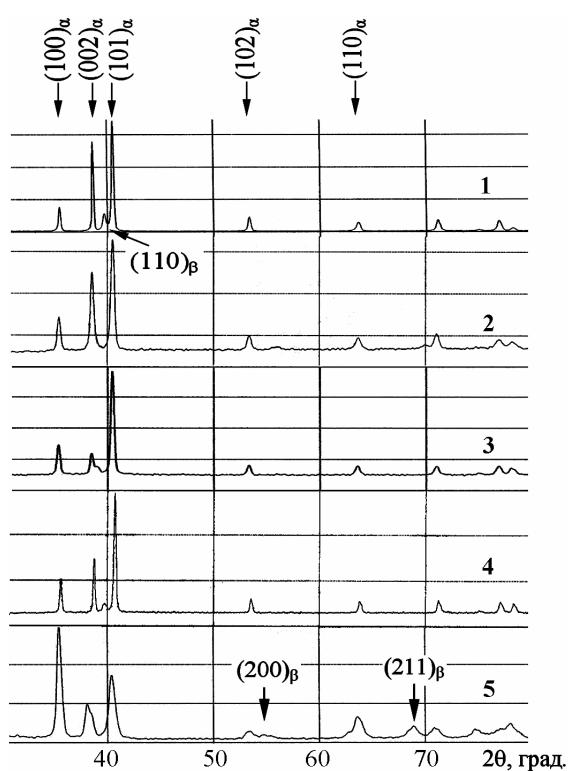


Fig. 4. Sections of diffraction patterns of samples of alloys Ti-6Al-4V-H with different hydrogen content (wt. %): 1) 0,008, fine-grained state; 2–5) SMC state – 2) 0,33; 3) 0,08; 4) 0,005; 5) 0,33, after hardening from temperature 973 K

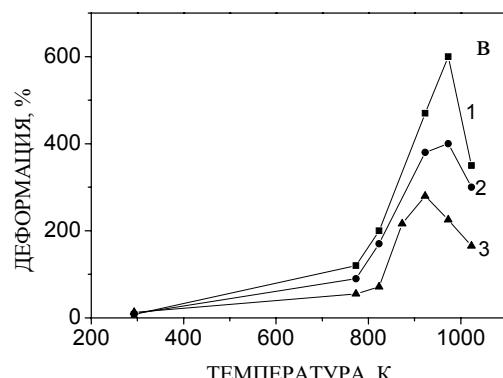
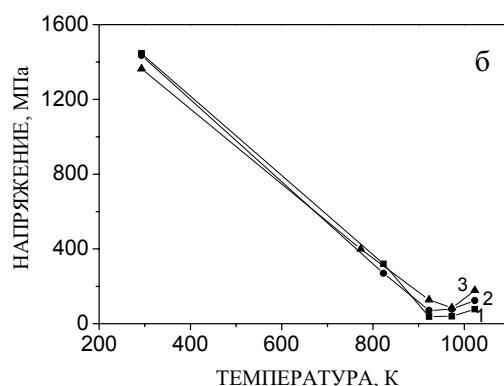
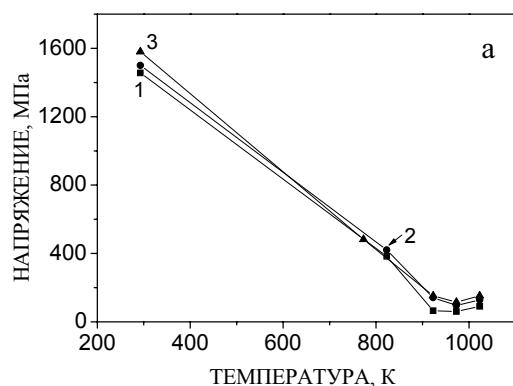


Fig. 5. Dependence of ultimate strength (a) yield point (6) and range of deformation to deconstruction (b) on temperature of testing alloys Ti-6Al-4V-0,005H (1), Ti-6Al-4V-0,08H (2) and Ti-6Al-4V-0,33H (3)
Напряжение – Stress; Деформация – Deformation; Температура – Temperature

perature 973...1023 K is conditioned, obviously, by the beginning of SMC grain growth. The fact that after two hours of annealing at temperature 973 K grain growth is observed even in the alloy with the most equilibrium structure – Ti-6Al-4V-0,005H makes in favor of such suggestion.

It follows from the analysis of hydrogen influence on strength properties of SMC alloys Ti-6Al-4V-H that at temperatures lower than 873 K hydrogen alloying in the range of studied concentration influences insignificantly on values σ_B and $\sigma_{0.2}$ (the observed increase or decrease of values σ_B and $\sigma_{0.2}$ is not more than 10...20 %). In temperature range 923...1023 K increase of hydrogen concentration in SMC alloy from 0,005 to 0,33 wt. % results in growth of values σ_B and $\sigma_{0.2}$ in 2...3 times. For example, at temperature 923 K value $\sigma_{0.2}$ for Ti-6Al-4V-0,005H, Ti-6Al-4V-0,08H and Ti-6Al-4V-0,33H alloys amount to 38,71 and 128 MPa respectively. Increase of values σ_B and $\sigma_{0.2}$ at growth of hydrogen concentration in alloy may be connected with the increase of strength of β -phase owing to hydrogen dissolution in it [9].

Temperature dependence of deformation to failure δ of the studied SMC alloys Ti-6Al-4V-H as well as dependence of σ_B and $\sigma_{0.2}$ on temperature has nonmonotonic character. In temperature range 293...773 K value δ increases with temperature from 7...13 to 60...110 % and depends slightly on hydrogen concentration in alloy. At further temperature growth to 1023 K firstly, the abrupt increase of value δ is observed, and then – decrease. In this case value δ in temperature range 873...1023 K is higher when hydrogen concentration in alloy is lower.

Effect of decreasing plasticity of SMC Ti-6Al-4V-H alloys in temperature range 873...1023 K at hydrogen concentration increase may be connected with occurrence of inhomogeneity of hydrogen distribution on sample volume at testing. Inhomogeneity of hydrogen distribution on sample volume may be caused by hydride dissolution at stated temperatures [10] as well as hydrogen capability of concentrating in the most stressed areas [9]. It is known that hydrogen alloying of alloy Ti-6Al-4V to concentration 1,0 wt. % results in temperature decrease of $\alpha \rightarrow \beta$ transformation to 973 K and formation of β -phase enriched with hydrogen [10]. Formation of β -phase enriched with hydrogen in local areas of a sample results in development of inhomogeneous plastic deformation. The result is deformation localization at microlevel and decrease of deformation value to destruction. The data obtained at studying of deformation distribution on sample test portion length after tension and phase composition of studied alloys after hardening from 973 K make in favour of such supposition.

X-ray diffraction analysis of alloy Ti-6Al-4V-0,33H after hardening from temperature 973 K showed that at specified temperature volume fraction of β phase really increases in alloy; increase of intensity of β phase reflections on diffraction pattern indicates this (Fig. 4, curve 5). In this case parameter of β phase lattice is consi-

derably larger than in original alloy (Table 1). At the same time hardening from temperature 973 K of alloy Ti-6Al-4V-0,005H does not change the sort of its diffraction pattern and parameters of phase lattice.

Studying deformation distribution on sample test portion length it was stated that at testing temperatures higher than 873 K deformation localization at microlevel in studied alloys occurs in development of weakly (alloy Ti-6Al-4V-0,005H) or sharply (Ti-6Al-4V-0,08H and Ti-6Al-4V-0,33H) defined collar (Fig. 6).

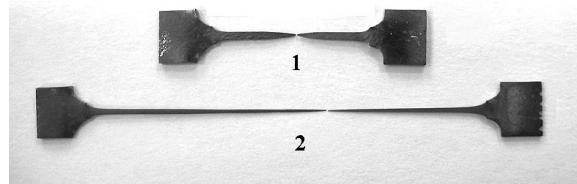


Fig. 6. Samples of alloys: 1) Ti-6Al-4V-0,08H and 2) Ti-6Al-4V-0,005H after tension at 973 K

The degree of development of deformation localization processes at microlevel may be estimated by the value of deformation localization value which is determined by the formula [11]:

$$\eta = 1 / [(1 - \psi)(1 + \delta)],$$

where ψ is the contraction in a collar, δ is the deformation to destruction.

Coefficient of alloy deformation localization for Ti 6Al-4V-0,33H alloys, Ti-6Al-4V-0,08H and Ti-6Al-4V-0,005H at temperature 923 K amounts to 13, 9,6 and 6,8 respectively. At temperature 973 K its value changes and becomes equal to 21, 10,4 and 5,1.

It is seen from comparison of values η of studied alloys (Table 2) that alloys with higher hydrogen content – Ti-6Al-4V-0,08H and Ti-6Al-4V-0,33H show the tendency to a large extent to deformation localization at microlevel in comparison with alloy Ti-6Al-4V-0,005H.

4. Conclusion

Using the method combining reversible hydrogen alloying and hot pressing allows forming in Ti-6Al-4V alloy submicrocrystalline structure with average grain size $<0,3 \text{ } \mu\text{m}$. Alloying of submicrocrystalline alloy Ti-6Al-4V with hydrogen to 0,33 wt. % influences insignificantly the ultimate strength and yield point of alloy at temperatures lower than 873 K. At temperatures higher than 873 K increase of hydrogen concentration in submicrocrystalline alloy from 0,005 to 0,33 wt. % results in growth of ultimate strength and yield point in 2...3 times and decrease of deformation value to destruction. It is supposed that it is connected with formation of β -phase hardened with hydrogen in the most stressed areas of a sample and as a result development of inhomogeneous plastic deformation.

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REFERENCES

1. Kolobov Yu.R., Valiev R.Z., Grabovetskaya G.P. et al. Grain boundary diffusion and properties of nanostructured materials. – Novosibirsk: Nauka, 2001. – 232 p.
2. Mazurovskii M.I., Murzinova M.A., Salischev G.A., Afonichev D.D. Use of hydrogen alloying for formation of submicrocrystalline structure in two-phase titanium alloys // Metals. – 1995. – № 6. – P. 83–88.
3. Yoshimura H., Nakahigashi J. Ultra-fine grain refinement, superplasticity and its application of titanium alloys obtained through protium treatment // Mat. Sci. Forum. – 2003. – V. 426–432. – P. 673–680.
4. Grabovetskaya G.P., Melnikova E.N., Kolobov Yu.R. et al. Evolution of structure-phase state of alloy Ti-6Al-4V at formation of submicrocrystal structure using reversible hydrogen alloying // Izvestiya Vuzov. Fizika. – 2006. – V. 49. – № 4. – P. 86–91.
5. Skvortsov S.V., Ilyin A.A., Gurkovaya G.V. et al. Phase and structure transformations occurring in titanium alloy VT20L under hydrogen // Metals. – 2005. – № 2. – P. 45–53.
6. Nosov V.K., Kolachev B.A. Hydrogen plasticization at hot plastic deformation of titanium alloys. – Moscow: Metallurgiya, 1986. – 118 p.
7. Kolachev B.A., Nosov V.K. Hydrogen plasticization and superplasticity of titanium alloys // Fizika Metallov i Metallovedenie. – 1984. – V. 57, Issue 2. – P. 288–297.
8. Kolachev B.A., Livanov V.A., Bukhanov A.A. Mechanical properties of titanium and its alloys. – Moscow: Metallurgiya, 1974. – 554 p.
9. Ilyin A.A., Mamontov A.M. Temperature-concentration diagrams of phase composition of hydrogen containing multicomponent alloys on the basis of titanium // Metals. – 1994. – № 5. – P. 71–78.
10. Presnyakov A.A. Localization of plastic deformation. – Moscow: Mashinostroenie. – 1983. – 56 p.

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FORMATION OF PHASE STRUCTURE IN THE SYSTEM Ti-3Al AT THE STAGE OF SECONDARY STRUCTURIZATION AT SYNTHESIS IN THE MODE OF THERMAL EXPLOSION

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Features of phase formation processes in the system Ti-3Al at realization of self-extending synthesis in the mode of thermal explosion have been established with use of technological reactor enabling instant switching-off of the heating source. The analysis of synthesis finished products allows to draw a conclusion that phase structure of charge is abnormally depends on sizes of titanium particles. The single-phase product corresponding to initial stoichiometry is synthesized on fine and large fractions for the induction period. On intermediate fraction the product of synthesis is multiphase.

Introduction

Recently, interest of researchers in the field of fundamental and applied material science to compounds on the basis of titanium aluminide increased considerably. Use of intermetallic compounds on the basis of titanium and aluminum in various branches of engineering has wide perspectives owing to combination of a number of mechanical and physical-chemical properties. Titanium aluminides are characterized by low specific weight, high heat resistance, thermal stability, resistance to corrosive media action at high temperatures that makes it possible to apply them in aircraft construction, shipbuilding [1].

At the same time, rather small amount of publications are devoted to studying self-propagating high-temperature (SH) synthesis in this system in spite of the fact that the main field of application of specified compounds is detonation-gas or plasma spraying for obtaining protective coatings. From this point of view the main task of experimenter is to obtain monophase powder materials of certain composition. It should be kept in mind here that the process of phase formation may be nonequilibrium.

In the work [2] the criteria determining two ultimate mechanisms of occurring SH-synthesis processes depending on the ratio of characteristic burning time t_c and structure formation t_s were stated. In the case $t_s/t_c < 1$ equilibrium mechanism of structure formation takes place. In the process of burning all phases known at state diagram (Merzhanov mechanism) are formed. In opposite case $t_s/t_c > 1$ during burning reaction products being in metastable state are formed. At termination of chemical reaction phase and chemical transformations which are determined by diffusion processes (Borovinskaya mechanism) occur in these products. At this very stage the processes of structure formation may depend on environment, first of all, on heat dissipation condition and size of refractory component particles. Change of ratio of indicated times may give an opportunity to control the end product composition in the mode of layerwise burning or thermal explosion. In this case it should be kept in mind that times of structure formation are determined by diffusion coefficients at phase formation in the processes of reactionary diffusion or dissolution. If typical time of heat extraction is rather lower than structure formation time the metastable phases may be obtained [3].