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Influence of Deformation Processing Modes on the Structure and Mechanical Properties of a High-Temperature Titanium Alloy of the Ti–Al–Zr–Si–Mo–Nb–Sn System

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To determine the kinetics of phase transformations, a calculated CCT-diagram for a titanium alloy of the Ti–Al–Zr–Si–Mo–Nb–Sn alloying system is obtained. The study of the structure and mechanical properties of the heat-resistant alloy of the Ti–Al–Zr–Si–Mo–Nb–Sn alloying system, which are obtained at different temperatures after thermodeformation treatment, is carried out. As established, the deformation treatment carried out in the upper part of the area of the existence of (α+β)-phases made it possible to increase the strength of the material at the room and operating temperatures and, that is especially important, to increase significantly the plasticity of the material, allowing only a slight decrease on average in its yield strength during short-term tests. As also found, a greater degree of deformation destroys hard silicide layers, distributes silicides more uniformly, increases both the strength and plasticity of the alloy, and slightly reduces the heat-resistant properties at 600°C.

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Key words: heat-resistant titanium alloy, phase transformation, deformation treatment, structure, phase, mechanical properties.

З метою визначення кінетики фазових перетворень одержано розрахункову CCT-діяграму для титанового стопу системи леґування Ti–Al–Zr–Si–Mo–Nb–Sn. Проведено дослідження структури та механічних властивостей, одержаних за різних температур, жароміцного титанового стопу системи леґування Ti–Al–Zr–Si–Mo–Nb–Sn після термодеформаційного оброблення. Встановлено, що деформаційне оброблення, яке було проведено у верхній частині области існування (α+β)-фаз, уможливило збільшити міцність матеріялу за кімнатної та робочої температур, а найголовніше, істотно збільшити пластичність матеріялу, водночас допустивши лише незначне середнє пониження межі плинности матеріялу під час короткотривалих випробувань. Встановлено також, що більший ступінь деформування руйнує суцільні силіцидні прошарки, більш рівномірно розподіляє силіциди, підвищує як міцність, так і пластичність стопу, але дещо понижує жароміцні властивості за 600°C.

Ключові слова: жароміцний титановий стоп, фазове перетворення, деформаційне оброблення, структура, фаза, механічні властивості.

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1. INTRODUCTION

Titanium alloys combine high specific strength, characteristics of resistance to fatigue and crack propagation, *etc.* [1, 2]. However, the development of new heat-resistant construction materials is an urgent task in connection with the growing requirements for engine assemblies being designed. Promising alloying elements, which increase the heat resistance of α-titanium, include such a β-stabilizer as silicon [3–5]. The solubility of silicon in α-titanium is rather limited and is of 0.31–0.54% in the temperature range of 750–860°C [6]. Silicide-strengthened titanium-based alloys are attractive promising materials with high structural efficiency for widespread use due to their significant specific strength and stiffness, good high-temperature properties, and fracture resistance. The main problem of such alloys is the near-zero plasticity in the cast state at room temperature, due to which their practical application is significantly limited [7].

An effective way to increase the complex of physical and mechanical properties of semi-finished products and products made of titanium alloys, along with alloying and heat treatment is thermodeformation treatment (TDT). It is known that such alloys deformed in the β-area have a lamellar microstructure and exhibit higher resistance to high-temperature creep and higher impact toughness. However, this advantage comes at the expense of lower ductility and thermal stability, leading to 'beta brittleness' and 'structural heritability'. During (α+β)-

deformation, the material is usually heated and processed by 30–50°C below the β-transition temperature [8–11]. Therefore, when choosing the thermomechanical mode of deformation, it is necessary to determine the average values of the permissible degrees of one-time deformation of cast ingots at different temperatures, as well as the influence of the degree of deformation on the main structural components and mechanical properties of the material. Setting the optimal thermomechanical parameters of the deformation process consists in choosing the initial and final temperature and determining the maximum permissible degree of deformation in the given temperature range. However, it is essential to know the $\alpha \leftrightarrow \beta$-titanium transformation temperature, which for multicomponent titanium alloys can vary depending on the content of the alloying elements [1].

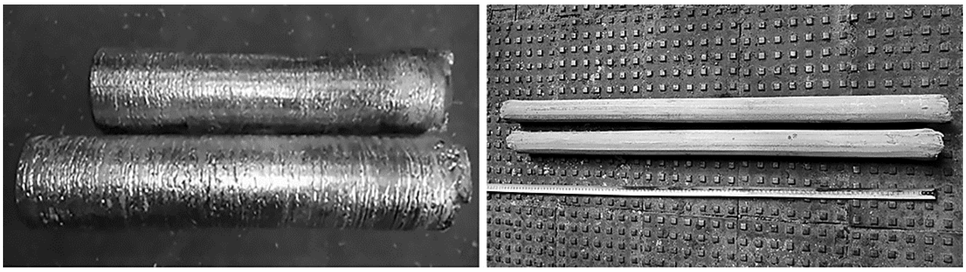
One of the main approaches for calculating equilibrium state diagrams is the CALPHAD (Calculation of Phase Diagrams) method [12], which is based on a comparative analysis of calculated data with experimental information about phase equilibriums in the system and thermodynamic properties of phases. The thermodynamic properties of each phase are described by a mathematical model, the parameters of which are calculated by minimizing the difference between the calculated value and its experimental value, taking into account all coexisting phases. After that, it is possible to recalculate the phase diagram and thermodynamic properties of the phases included in the system. Using the known rules of additivity [13–15], the isothermal transformation diagram constructed in this way can be easily converted into a thermokinetic transformation diagram for continuous cooling (CCT).

2. RESEARCH MATERIALS AND METHODS

E. O. Paton Electric Welding Institute, N.A.S. of Ukraine already has extensive experience in obtaining ingots of both industrial and the latest heat-resistant titanium alloys by the method of electron beam remelting (EBR) [16–18]. Pre-mechanically processed EBR ingots ∅110 mm were used for further deformation treatment (Fig. 1).

To investigate the presence of internal defects such as non-metallic inclusions, as well as pores and leaks in titanium ingots, the method of ultrasonic defectoscopy was used. The study was carried out with an ultrasonic flaw detector UD4-76 (Ukraine) using the echo-impulse method with a contact version of control.

To analyse the content of alloying elements in the obtained ingots, optical emission spectrometry (ICP-OES) was used on an ICP 6500 DUO inductively coupled plasma spectrometer from Thermo-Electron Corporation (USA). The results of chemical analysis (Table 1) show that the distribution of alloying elements along the length of the ingots is uniform.



a *b*

Fig. 1. EBR ingots ∅110 mm (*a*) and manufactured deformed semi-finished products (*b*) from the heat-resistant alloy of the Ti–Al–Zr–Si–Mo–Nb–Sn system.

TABLE 1. Chemical composition of the alloy of the Ti–Al–Zr–Sn–Si–Mo–Nb system.

Average chemical composition, wt. %						
Al	Zr	Si	Mo	Nb	Sn	Ti
6.2–6.9	5.0–5.5	0.5–0.85	0.5–0.8	0.5–0.8	1.5–2.5	base

Cast ingots were heated for rolling in an electric resistance furnace to a temperature of 1050°C with the time required for heating. The ingots and semi-finished products were rolled on a Skoda 355/500 rolling mill.

Samples were cut from the obtained deformed semi-finished products to study the microstructure and mechanical properties of the material at different temperatures. To reveal the microstructure of the samples, etching was carried out in a reagent consisting of a mixture of hydrofluoric and nitric acids in the following ratio: 10% hydrofluoric acid (HF), 30% nitric acid (HNO_3) and distilled water. Metallographic studies were performed by optical and scanning electron microscopy methods using a Neophot-32 optical microscope and a JEOL Superprobe-733 raster microanalyzer. For mechanical tensile tests at 20 and 600°C after deformation treatment, a universal electromechanical machine UTM-100 with a maximum load of 100 kN and a deformation speed of 2.5 mm/min was used.

3. RESULTS AND DISCUSSION

According to the given CALPHAD method, a calculated CCT-diagram was obtained for the heat-resistant titanium alloy of the Ti–Al–Zr–Sn–Mo–Nb–Si alloying system (Fig. 2). The diagram shows the initial

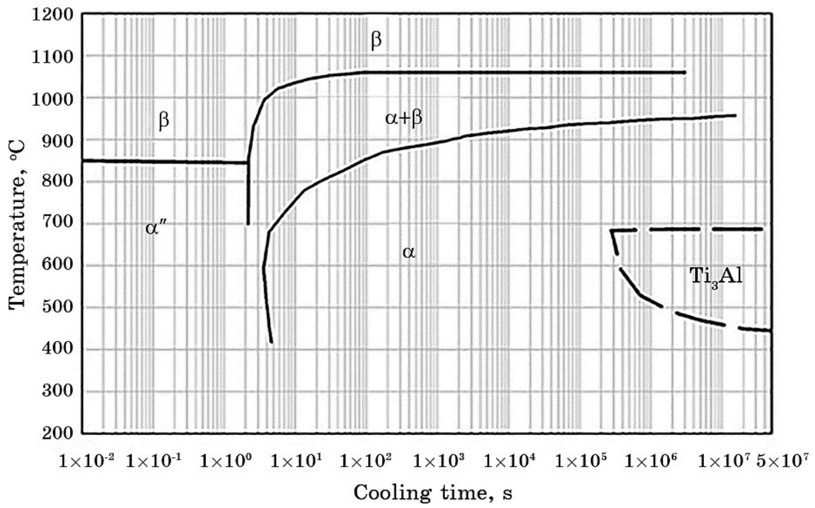


Fig. 2. Calculated CCT-diagram of the heat-resistant titanium alloy of the Ti–Al–Zr–Sn–Mo–Nb–Si system.

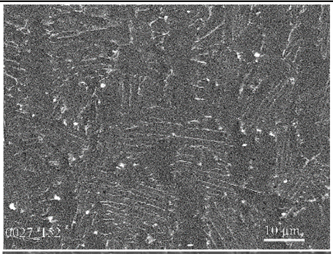
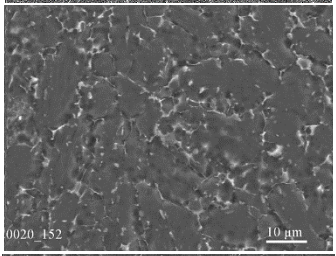
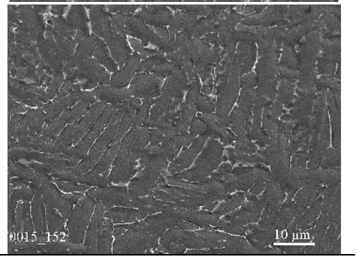
temperature of β→(α+β) transformation (≅1050°C) and the final temperature of β→α-transformation (900...880°C) at cooling rates of 100...0.01°C/s.

The experimental cast alloy of the Ti–Al–Zr–Mo–Nb–Sn–Si alloying system belongs to the near-α alloys, the main structural components of which are lamellar α-phase and a small amount of residual β-phase [19]. However, the final structure of near-α-titanium alloys is formed in the process of hot deformation treatment and the type of structure does not undergo significant changes during the subsequent heat treatment.

It was established that the CCT-diagram of a heat-resistant alloy of the Ti–Al–Zr–Si–Mo–Nb–Sn alloying system, carried out under different regimes, quite significantly changes the morphology of the metal structure. The rolling process was the same in all cases, so the difference in structural parameters is caused only by the temperatures at the end of the deformation process (Table 2).

Titanium alloys deformed in the lower part of the (α+β) area have a mostly equiaxed microstructure [1]. This type of microstructure shows higher ductility and fatigue properties, but lower high-temperature properties and lower impact toughness. Increasing the heating temperature in the area of (α+β) phase reduces the volume fraction of the equiaxed α-phase and improves the high-temperature properties, but creates the risk of reduced plasticity and insufficient thermal stability. Therefore, the question of how to develop a material with a good combination of strength, heat resistance, and plasticity remains an un-

TABLE 2. Temperatures of TDT and thus obtained structures of the alloy of the Ti–Al–Zr–Sn–Si–Mo–Nb system.

No	Final temperature of TDT	The obtained structure	
1	$T_{(\beta\to(\alpha+\beta))-100°C}$	globular–lamellar	
2	$T_{(\beta\to(\alpha+\beta))-150°C}$	close to equiaxed	
3	$T_{((\alpha+\beta)\to\alpha)-50°C}$	of the 'basket weaving' type	

solved problem for a long time.

A study of the mechanical properties at room and elevated temperatures of the cast and deformed material, the rolling of which was carried out in several passes with intermediate heating in the temperature range from 1050°C to the temperature $T_{(\beta\to(\alpha+\beta))}$–100°C and a total final compression of ≅ 70%, that, in our opinion, ensured the formation of an optimal microstructural composition (Table 2). Since the use of this group of alloys is expected at operating temperatures up to 600°C, tests at elevated temperatures were also performed at 600°C.

The data of mechanical tests are shown in Fig. 3. Deformation treatment, which was carried out at a temperature of $T_{(\beta\to(\alpha+\beta))}$–100°C, made it possible to increase the strength of the material both at room and working temperatures, and most importantly, to significantly increase the plasticity (by almost 25 times), while allowing only a slight average decrease in the materials' yield strength during short-term tests.

If we compare the mechanical characteristics of Ti–(6–7)Al–(2–

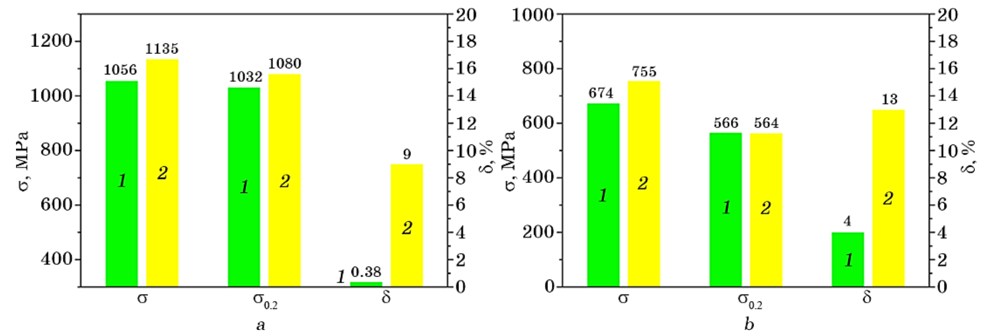


Fig. 3. Tensile mechanical properties of the cast (*1*) and deformed (*2*) alloy of the Ti–Al–Zr–Sn–Si–Mo–Nb system at different temperatures: 20°C (*a*) and 600°C (*b*).

3)Zr–(1–1.5)Si and Ti–(6–7)Al–(3–5)Zr–(1–1.5)Si–(2–4)Sn alloys of the basic Ti–Al–Zr–Si alloying system, which obtained in the previous work of the authors [11], it is possible to conclude that additional alloying of Mo, Nb, and Sn with a decrease in Si content leads to an increase in plasticity at room temperature, but to a significant decrease in heat resistance at operating temperatures of 600–700°C. Therefore, unlike the previously developed alloys intended for gas turbine engine blades, experimental alloys of the Ti–Al–Zr–Sn–Si–Mo–Nb alloying system can be recommended for the manufacture of gas turbine engine compressor discs.

Experiments were also conducted to reveal the influence of the degree of deformation on the structure and properties of the deformed alloy. Rolling of the alloy of the Ti–Al–Zr–Sn–Si–Mo–Nb system was carried out from 1050°C to the temperature $T_{(\beta\to(\alpha+\beta))}$–100°C in several passes with intermediate heating's and a total compression from 50 to 90%. Thus, in the microstructure of the deformed alloy, the α-phase is present in both globular and lamellar forms, but the share of the equiaxed α-phase prevails (Fig. 4).

A greater degree of deformation increases both the strength and plasticity of the alloy at 20°C, but slightly reduces the heat-resistant properties at 600°C (Fig. 5). The deformation texture is formed and is especially noticeable at a lower degree of compression ≅ 50% (Fig. 4, *a*, *b*).

At 90% deformation, the structure of the alloy becomes more fine and homogeneous (Fig. 4, *c*, *d*). Greater deformation also allows the destruction of continuous silicide layers and provides a more uniform distribution of silicides (Fig. 6).

At a temperature corresponding to $T_{(\beta\to(\alpha+\beta))}$–100°C, both the β-phase and the remaining α-phase take part in deformation. The shape of the α-plates changes and they are located along the direction of the metal flow. Further deformation of the α-phase is accompanied by globulari-

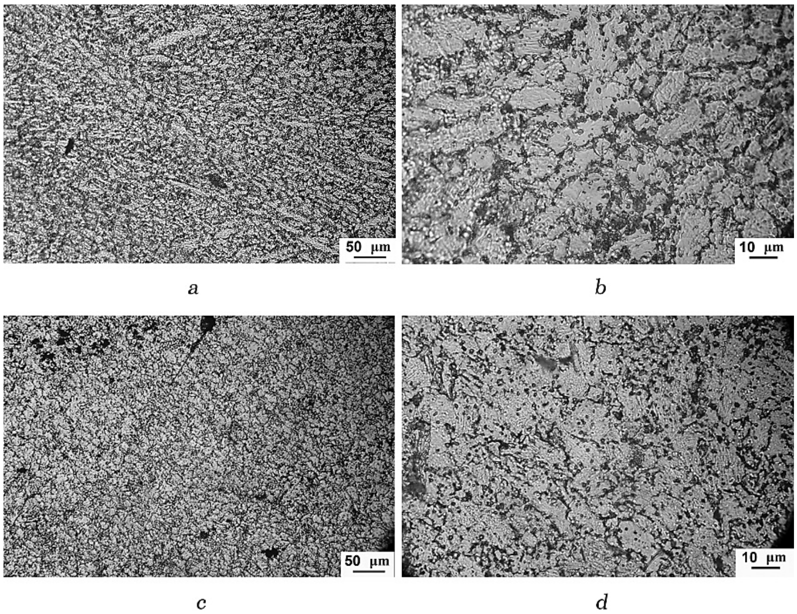


Fig. 4. The structure of the heat-resistant alloy of the Ti–Al–Zr–Sn–Si–Mo–Nb system, deformed at a temperature of $T_{(\beta\to(\alpha+\beta))}$–100°C (light microscopy): *a*, *b*—$\varepsilon = 50\%$; *c*, *d*—$\varepsilon = 90\%$.

zation—the separation of α-plates into individual particles—globules, as a result of which the share of the lamellar α-phase in the alloy deformed by 90% is decreased (Fig. 4, *c*, *d*).

The electron-microscopy study showed (Fig. 6) that, in the structure of the rolled alloy, there are separate large silicides distributed along the boundaries, between α-plates, and there is a fairly large amount of

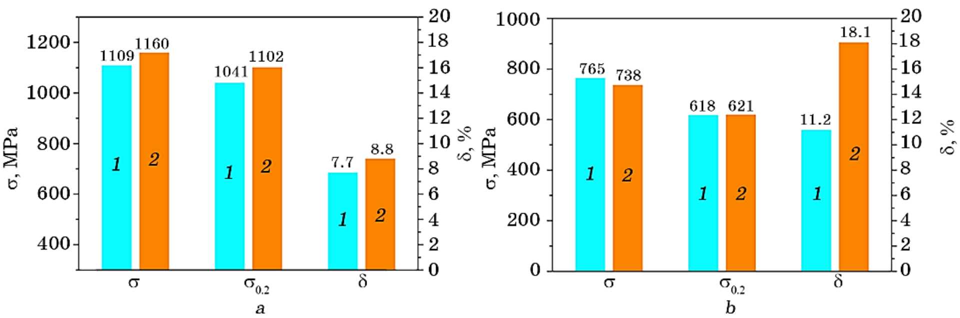


Fig. 5. The influence of the degree of deformation (*1*—50%, *2*—90%) on the structure and mechanical properties at different temperatures of 20°C (*a*) and 600°C (*b*) of the heat-resistant alloy of the Ti–Al–Zr–Sn–Si–Mo–Nb system.

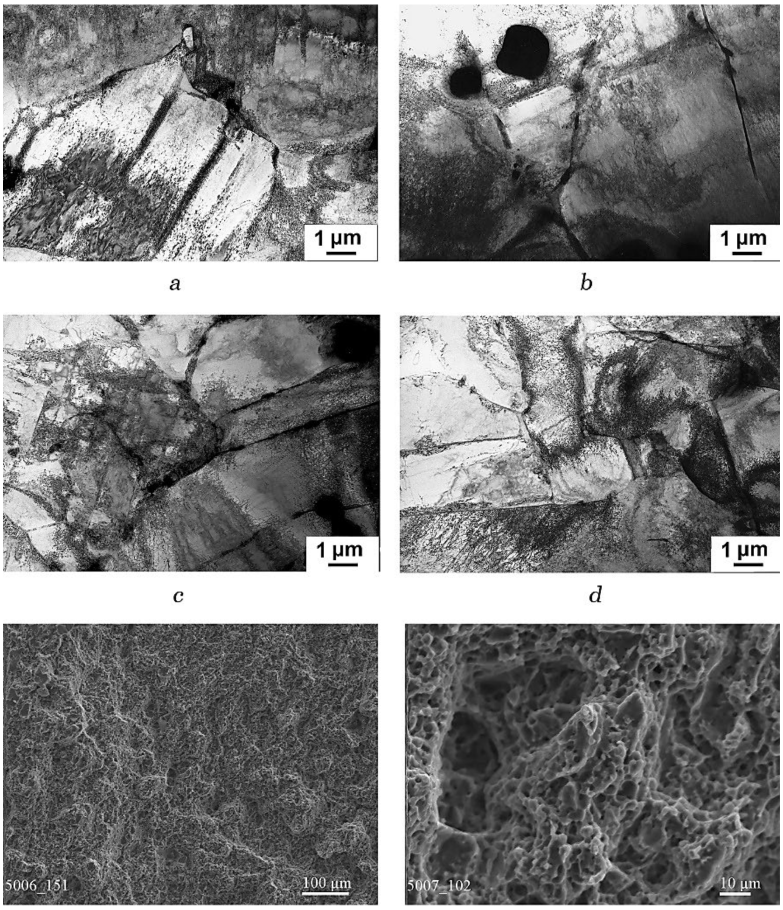


Fig. 6. Electron microscopies of the deformed alloy: transmission (*a*, *b*—$\varepsilon = 50\%$; *c*, *d*—$\varepsilon = 90\%$) and scanning (*e*, *f*—$\varepsilon = 90\%$, tensile fracture surface) ones.

dispersed silicides in β-layers too, in the middle of the α-phase grains, on dislocations. The segregation of silicides at the boundaries of grains and α-plates restrains their growth, while dispersed silicide particles in the grains and plates themselves increase strength. At a higher degree of deformation $\cong 90\%$, the Ti–Al–Zr–Sn–Si–Mo–Nb alloy has smaller α-grains and plates, while dispersing and a more uniform distribution of silicides occurs (Fig. 6, *c*, *d*).

It was earlier established that additional doping with zirconium in the experimental alloys [11, 18, 20] affects significantly the solubility of silicon in titanium and, accordingly, the release of silicides that changes the size of the grains and plates of the α-phase, as well as both mechanical and heat-resistant properties. The dislocation substructure formed in the deformed state intensifies the disintegration of the solid solution with the release and uniform distribution of dispersed silicides. This contributes to obtaining high strength and fluidity at 600°C.

The fracture during stretching of the deformed alloy is mainly intragranular and pitted (Fig. 6, *e*, *f*). The tensile fracture surface of the Ti–Al–Zr–Sn–Si–Mo–Nb alloy with a mixed globular–lamellar structure also contains a small part of intergranular fracture along the α-grain boundaries, and chipped facets in areas of the lamellar α-phase. Most of the fracture surface consists of small pits, at the bottom of which many dispersed silicides < 1 μm in size remain. The deformation ridges pass along the boundaries of α-grains or plates, where β-layers are located.

4. CONCLUSION

A calculated CCT-diagram of the heat-resistant titanium alloy of the Ti–Al–Zr–Sn–Mo–Nb–Si alloying system was constructed, which made it possible to determine the temperature of the α↔β-phase transformation for thermal deformation.

It was found that the best ratio of mechanical properties, namely, 9% relative elongation and a high level of strength both at room temperature (1135 MPa) and at an operating temperature of 600°C (750 MPa), is demonstrated by TDT, which was carried out at a temperature of $T_{(\beta\to(\alpha+\beta))}$–100°C.

The globular-plate structure with dispersed silicides formed in the Ti–Al–Zr–Sn–Si–Mo–Nb alloys after rolling at a temperature of $T_{(\beta\to(\alpha+\beta))}$–100°C with a degree of deformation of 90% ensures high strength and plastic properties at 20°C and 600°C. Therefore, hot deformation processing of these materials is recommended to be carried out with a degree of deformation of at least 70%.

It was established that the high heat-resistant properties of experimental alloys of the Ti–Al–Zr–Sn–Si–Mo–Nb alloying system are provided both by solid solution strengthening based on the α-phase and by dispersed silicide particles. Thus, the mechanical properties of these alloys, obtained during short-term tests at different temperatures, allow us to recommend the Ti–Al–Zr–Sn–Si–Mo–Nb alloy system for the manufacture of experimental disks of gas turbine compressors.

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