

PACS numbers: 61.05.cp, 61.66.Dk, 61.72.Ff, 81.05.Bx, 81.30.Bx, 81.70.Jb, 81.70.Pg

Development of a Titanium Aluminide Alloy of Optimal Composition by Means of Modification

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Alloys based on γ -aluminide are a promising class of heat-resistant materials for the manufacture of parts of modern aircraft engines, the maximum operating temperature of which is in the range of 600–700°C. The main advantages of these alloys are the combination of low density, high structural stability, and heat resistance. However, due to the natural characteristics of intermetallic compounds (structure ordering, strong covalent bonding), γ -alloys are characterized by low plasticity compared to traditional titanium alloys that complicates the industrial application of these alloys and increases the complexity of the process of manufacturing semi-finished products and final parts. For the successful use of γ -alloys and their introduction into

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Citation: O. B. Halienkova, V. S. Yefanov, O. V. Zavgorodniy, I. M. Kirian, S. O. Bulish, and O. D. Rud, Development of a Titanium Aluminide Alloy of Optimal Composition by Means of Modification, *Metallofiz. Noveishie Tekhnol.*, **47**, No. 3: 245–256 (2025), DOI: [10.15407/mfint.47.03.0245](https://doi.org/10.15407/mfint.47.03.0245)

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modern aircraft engines, it is necessary to increase their technological plasticity, while ensuring high strength and heat resistance characteristics. One of the most effective ways to accomplish this task is by means of alloying and modification. The work considers the influence of alloying elements niobium and molybdenum, as well as the modifying element rhenium, on the morphology, phase composition and mechanical characteristics of the γ -alloy in the Ti–Al system. The optimal concentrations of the elements Nb, Mo and Re in the titanium aluminide-based Ti–29Al–7Nb–2Mo alloy are determined, ensuring a two-fold increase in the mechanical properties of the alloy due to the weakening of covalent bonding forces. Using the method of high-temperature thermal analysis, it is established that the introduction of niobium, molybdenum and rhenium into the intermetallic does not change the sequence of transformations and the temperature ranges of phase-regions existence.

Key words: titanium aluminides, modifiers, phase composition, x-ray micro-analysis, x-ray phase analysis.

Стопи на основі γ -алюмініду є перспективним класом жароміцних матеріалів для виготовлення деталей сучасних авіаційних двигунів, максимальна робоча температура яких лежить у діапазоні 600–700°C. Головними перевагами цих стопів є поєднання низької густини, високої структурної стабільності, жароміцності. Однак, у зв'язку з природними особливостями інтерметалідних сполук (впорядкованість структури, сильний ковалентний зв'язок), γ -стопам притаманна низька пластичність порівняно із традиційними титановими стопами, що ускладнює промислове освоєння цих стопів і підвищує трудомісткість процесу виготовлення напівфабрикатів і кінцевих деталей. Для успішного використання γ -стопів і впровадження в сучасні авіаційні двигуни необхідно підвищити їхню технологічну та конструкційну пластичність з одночасним забезпеченням високих характеристик міцності та жароміцності. Один з найбільш ефективних способів виконання цього завдання досягається шляхом легування та модифікування. В роботі розглянуто вплив легувальних елементів Ніобію та Молібдену та модифікувального елементу Ренію на морфологію, фазовий стан і механічні характеристики γ -стопу системи Ti–Al. Визначено оптимальні концентрації елементів Nb, Mo та Re у стопі на основі алюмініду Титану Ti–29Al–7Nb–2Mo, що забезпечують підвищення рівня механічних властивостей стопу у 2 рази за рахунок послаблення сил ковалентного зв'язку із введенням Молібдену та Ніобію та зміни морфології за рахунок поверхнево-активної дії модифікатора. З використанням методи високо-температурної термічної аналізи встановлено, що введення Ніобію, Молібдену та Ренію в інтерметалідний стоп не змінює послідовність перетворень і температурні діапазони існування фазових областей.

Ключові слова: алюмініди Титану, модифікатори, фазовий склад, мікроаналіза, рентгенофазова аналіза.

(Received 2 February, 2025; in final version, 11 March, 2025)

1. INTRODUCTION

Titanium aluminide-based alloys, or γ -alloys, are one of the most promising materials for producing blades for new-generation gas turbine engines. Compared to other structural materials used in the aviation industry, the most important advantages of aluminide-based alloys are low density, high specific strength and modulus of elasticity, and high structural stability. The main forming technology for the production of titanium aluminide parts is casting technology [1–5], which is the most economical production process for obtaining products of complex shape. The microstructure of casting products is characterized by a coarse-grained structure, because of which they will be inferior in properties to products obtained, for example, by deformation or additive technologies [6].

To date, the largest numbers of studies on the influence of parameters and type of structure on mechanical properties are carried out on fourth-generation titanium aluminide alloys [7–9]. These alloys contain modifiers for the formation of an equilibrium lamellar structure with a small colony size, which provides high heat resistance and sufficient values of plasticity and fracture toughness.

The main direction of modification is the use of surface-active elements, the mechanism of action of which is adsorption on the surface of grains, which contributes to the change in the surface activity of crystals and the diffusion rate and leads to a change in the shape and size of crystals [10, 11]. The main criterion for choosing a surface-active modifier is low solubility in the lattice of the base metal, *i.e.*, titanium.

Lanthanides and rare earth elements are used as modifiers in titanium alloys – Sc, Y, B, La, Ce, Nd, Dy, *etc.*, the most effective and widely used of which are B and Y, Re is less commonly used [12–14]. Low boron content significantly changes the microstructure and properties. Since the atomic radius of boron (0.91 Å) differs from that of titanium (1.46 Å), at a boron content of more than 0.10%, compounds TiB, Ti₃B₄, TiB₂ are formed in the form of a coarse-needle framework along the grain boundaries, which reduces plasticity [15]. Therefore, the optimal B content is important, ensuring refinement of the structure and at the same time uniform distribution of boride release.

Yttrium has favourable thermodynamic characteristics, which allows it to influence the structure of titanium alloys [16]. Yttrium does not dissolve in titanium due to the large difference in their atomic radii [17]. Having a greater affinity for oxygen than titanium and a number of other elements, yttrium reacts with elements at grain boundaries.

Rhenium lowers the polymorphic transformation temperature of titanium. At rhenium concentrations up to 0.6%, the hardness of tita-

nium increases more than two-fold [18]. Because the difference in atomic radii with Ti is only 6.6%, rhenium has a higher solubility in titanium than yttrium. However, there is currently insufficient information on the influence of rhenium on the morphology, phase state and mechanical characteristics of γ -alloys.

Therefore, the work investigates the effect of Rhenium concentration at the level of 0.2–0.3% on the structure and properties of the Ti–29Al–7Nb–2Mo alloy based on titanium-aluminide intermetallic compound.

2. MATERIALS AND METHODS

The object of study in this work was titanium aluminide ingots of the composition Ti–29%Al alloyed with molybdenum and niobium, followed by modification of the alloy with rhenium. To study the influence of microadditions of the modifying element on the structure formation of the cast material, ingots of different compositions were melted in a vacuum arc furnace in a copper crystallizer using a non-consumable tungsten electrode. The ingots had the shape of washers with a diameter of 65–70 mm, a thickness of ≈ 12 mm, and a weight of ≈ 200 g.

The limiting concentration of Rhenium was selected based on the analysis of literature data, which indicate that the optimal content of modifying elements in alloys based on titanium aluminide is no more than 0.2...0.4% [10, 12, 13]. At higher content of modifying elements, the efficiency of cast grains refinement in general decreased. Therefore, the limiting concentration of rhenium content was selected at the level of 0.2–0.3%.

The chemical compositions of alloys were studied by the spectral reference-free method on an energy-dispersive x-ray fluorescence analyzer “Expert 3L”. Etching of samples for metallographic studies was performed in the “Titan” reagent with the composition $\text{HF}:\text{HNO}_3:\text{H}_2\text{O} = 1:2:6$. The assessment of the template macrostructure was carried out with the naked eye along the thickness of the ingot. The study of the microstructure of the ingot material was performed using an optical microscope “AxioObserver 5” at magnifications of $\times 25 \dots 200$. The study of the structure and distribution of alloying elements at higher magnifications was performed on a JEOL JSM IT-300 scanning electron microscope equipped with an Oxford Instrument X-Max80 x-ray spectral energy-dispersive microanalysis system.

Mechanical properties for tensile testing were determined according to DSTU ISO 6892 1:2019. Samples for testing were cut in the transverse direction to the ingot axis. Registration of diffractograms was carried out on a DRON-4 diffractometer in the Bragg–Brentano geometry with a MoK_α radiation in the angle range from 5° to 60° with a step

of 0.05° and an exposure of 4 s at each point. Differential thermal analysis was carried out on an automated analyser manufactured in the G. V. Kurdyumov Institute for Metal Physics of the N.A.S. of Ukraine in a helium atmosphere with a heating rate of 20 K/min.

3. RESULTS AND DISCUSSION

The chemical composition and mechanical properties of the obtained ingots are given in Tables 1 and 2, respectively. The alloy of the base composition Ti–29Al (alloy No. 1) belongs to the first generation titanium aluminide based alloys and has low strength and almost zero ductility (Table 2). The production of ingots of the Ti–29Al–7Nb–2Mo alloy (alloy No. 2), which belongs to the fourth generation alloys, involves the use of double remelting. During the first remelting, refractory alloying elements (niobium and molybdenum) are introduced into the alloy. During the second remelting, aluminium is added to the resulting ingot, taking into account evaporation losses. This allows minimizing aluminium losses and ensuring guaranteed dissolution of refractory alloying elements and uniform distribution over the ingot cross-section.

From the analysis of Table 2, it is determined that when niobium and molybdenum are introduced into the alloy, an increase in mechanical properties is achieved by weakening the covalent bond between Titanium and aluminium atoms. The increase in strength and ductility for the alloy with rhenium is likely associated with a change in the morphology of the initial alloy.

Qualitative and quantitative phase analysis of the alloys was performed using x-ray diffractometry (Fig. 1). It was found that regardless of the change in chemical composition, the main phase content is unchanged and corresponds to a mixture of α_2 - and γ -compounds in different ratios (Table 2). The base alloy has a higher percentage of α_2 -phase, and with the introduction of β -stabilizers and a modifier, the content of γ -phase increases, which leads to an increase in plasticity characteristics. This is due to the formation of a slightly distorted lattice of the γ -phase due to alloying with niobium and molybdenum [6, 19], as well as due to a change in the shape and size of the structural

TABLE 1. Chemical composition of alloys.

Alloy No.	System	Element content, wt. %					
		Al	Nb	Mo	O	Ti	Re
1	Ti–29Al	29.4	–	–	0.12	base	–
2	Ti–29Al–7Nb–2Mo	28.7	7.4	2.1	0.13	base	–
3	Ti–29Al–7Nb–2Mo–0.2Re	32.7	7.6	1.31	–	base	0.27

TABLE 2. Phase composition and mechanical properties of alloys.

Alloy No.	Phase composition of the alloy		$\sigma_{0.2}$, MPa	δ , %
	α_2	γ		
1	77	23	230	—*
2	16	84	385	0.4
3	14	86	510	0.6

*not determined due to the high fragility of the sample.

components of the alloy with the introduction of rhenium. The increase in the percentage of γ -phase with the introduction of niobium and molybdenum allows us to assert the action of the above elements as γ -stabilizers.

The macro- and microstructures of the alloys are shown in Figs. 2 and 3. Comparison of the macrostructure of alloys of different compositions clearly demonstrates the influence of modifiers on changing its morphology. Thus, the macrostructure of the original Ti–29Al alloy consists of large grains of irregular shape of different sizes (Fig. 2a), the Ti–29Al–7Nb–2Mo alloy with niobium and molybdenum consists of large shiny grains with a clear boundary, corresponding to 8–9 points on the 10-point scale of macrostructures of titanium alloys [20] (Fig. 2b). The introduction of rhenium in an amount of 0.2% led to the

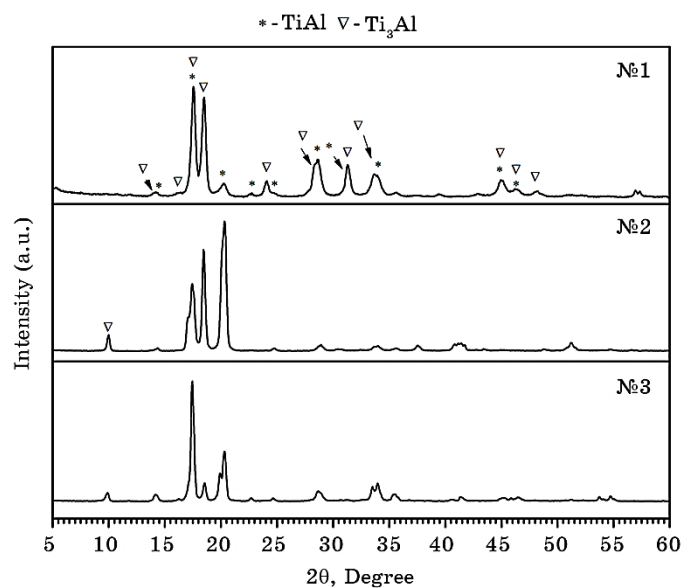


Fig. 1. XRD patterns of alloys: No. 1—Ti–29Al, No. 2—Ti–29Al–7Nb–2Mo, No. 3—Ti–29Al–7Nb–2Mo–0.2Re.



Fig. 2. Macrostructure of alloys after etching: *a*—alloy No. 1, *b*—alloy No. 2, *c*—alloy No. 3.

formation of a macrostructure of 5 points (Fig. 2*c*).

Metallographic study of ingots (Fig. 3) revealed that the microstructure of alloys is characterized by a lamellar structure consisting of a two-phase mixture of ($\alpha_2 + \gamma$) phases. The complex introduction of alloying elements and modifier led to a significant change in the morphology of the structural components of alloys compared to the structure of the initial composition.

The microstructures of Ti–29Al and Ti–29Al–7Nb–2Mo alloys (Fig. 3*a, b*) are typical for the cast state of titanium alloys, *i.e.*, coarse-grained, characterized by large grains, the internal volume of which is represented by colonies of plates (lamellas) of α_2 - and γ -phases, disorientated within one grain. The length of the plates is 100–400 μm , the thickness is 5–15 μm .

When rhenium is introduced into alloy No. 2 in an amount of 0.2% (Fig. 3*c*), the colonies of ($\alpha_2 + \gamma$)-phase plates are oriented in a single direction within one grain. In addition, there is a thinning of the structural components of the ingot to 2–3 μm and a reduction in the length of the plates to 100–250 μm , which in turn contributes to an increase in the strength limit of the alloy by 1.5 times compared to the initial

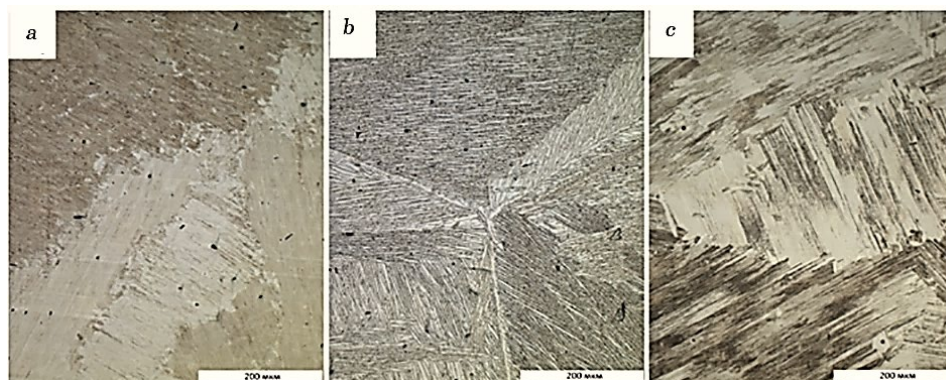


Fig. 3. Microstructure of alloys: *a*—alloy No. 1, *b*—alloy No. 2, *c*—alloy No. 3.

data.

To determine the uniformity of the distribution of the main alloying elements across the cross-section of ingots of the Ti–29Al, Ti–29Al–7Nb–2Mo, and Ti–29Al–7Nb–2Mo–0.2Re alloys, x-ray spectral microanalysis (XSMA) was performed in the mapping mode (Fig. 4).

Thus, the light areas of the structure are depleted in aluminium, while the dark areas, on the contrary, are enriched. According to the stoichiometric composition of the intermetallics Ti_3Al (α_2 -phase) and TiAl (γ -phase) and the solubility limit of aluminium in the correspond-

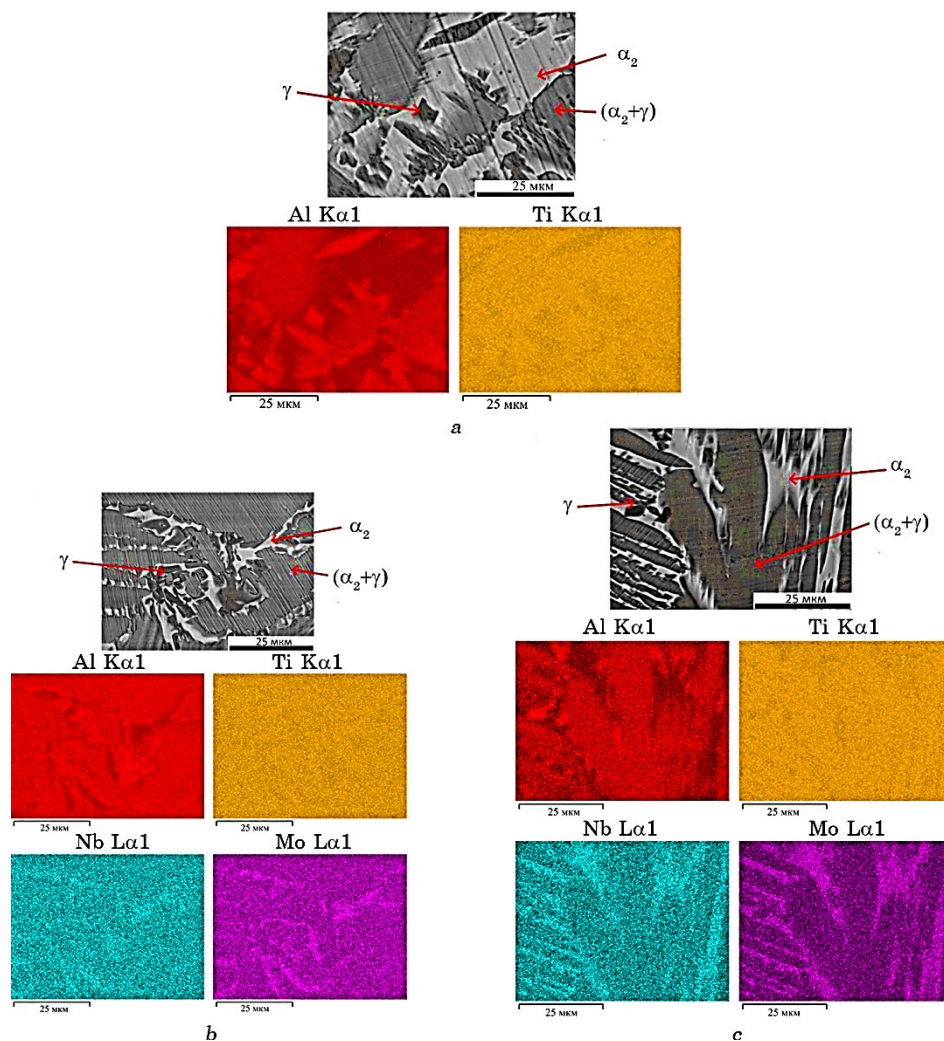


Fig. 4. X-ray spectral microanalysis of alloys in mapping mode: *a*—alloy No. 1, *b*—alloy No. 2, *c*—alloy No. 3.

ing compounds according to the phase diagram [21], the light areas of the structure can be classified as α_2 -phase, the dark areas as γ -phase. The results of XSM correlate with x-ray phase analysis, which shows a higher percentage of γ -phase in the composition of alloy No. 3 (Table 2).

According to the Ti–Al phase diagram (Fig. 5), the phase composition of the alloys at room temperature corresponds to a mixture of compounds α_2 (Ti_3Al)+ γ (TiAl).

In terms of the content of molybdenum and niobium, more intense colours in Fig. 4 correspond to the areas of the α_2 -phase. From the solubility graph of alloying elements in Ti_3Al and TiAl intermetallics [22], it is established that the solubility of β -stabilizers is greater in the γ -phase lattice: the solubility limit of molybdenum in the Ti_3Al lattice is no more than 1.5 at.%, in the TiAl lattice, it is of about 9 at.%; the solubility limit of niobium is of 12 at.% and 28 at.% for α_2 - and γ -phases, respectively. That is, the content of molybdenum and niobium in the alloys is close to the solubility limit of the specified elements in the α_2 -phase.

The high-temperature differential thermal analysis was used to study the thermal effects observed during continuous heating of samples from the experimental alloys (Fig. 6). Comparison of the curves with the Ti–Al diagram allows us to state that the endoeffects in the temperature range of 1110–1200°C are due to the transition of the ordered α_2 -phase into the disordered α -phase with the h.c.p. lattice by the reaction $\alpha_2 + \alpha \rightarrow \alpha + \gamma$ with subsequent transition to the single-phase α -region. Thermal effects in the temperature range of 1200–1300°C are not confirmed in the diagram and may be associated with the oxidation of the samples during heating or the process of separation of secondary

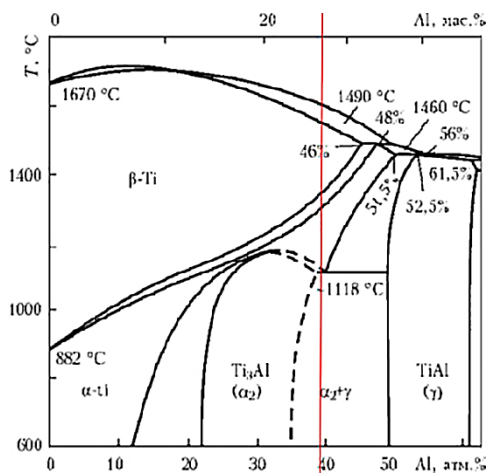


Fig. 5. Ti–Al phase diagram with a plotted line of chemical composition (in terms of aluminium content) of the studied alloys [21].

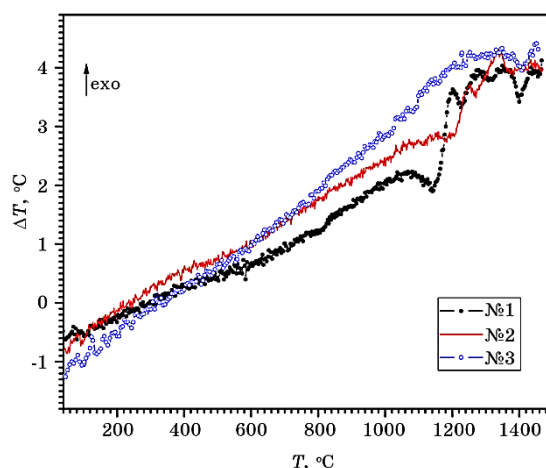


Fig. 6. Thermograms of alloys: No. 1—Ti–29Al, No. 2—Ti–29Al–7Nb–2Mo, No. 3—Ti–29Al–7Nb–2Mo–0.2Re.

phases. The following endoeffects at a temperature of 1350–1400°C are associated with the transition to the β -region, *i.e.*, a polymorphic transformation occurs. Thermal effects observed up to 1100°C, according to [23, 24], can be caused by the ordering-disordering processes occurring in intermetallic phases. Thus, it is determined that the introduction of alloying and modifying elements into an intermetallic alloy does not change the sequence of transformations and the temperature ranges of the existence of phase regions.

Thus, it has been established that alloys based on titanium aluminide of the Ti–29Al system are characterized by low ductility due to the structural features of the intermetallic compound. The introduction of alloying elements, in this case molybdenum and niobium, contributes to the improvement of the mechanical characteristics of the alloy by weakening the covalent bond between titanium and aluminium atoms. The addition of molybdenum and niobium to the Ti–29Al alloy improves the mechanical characteristics of the alloy by weakening the covalent bond between titanium and aluminium atoms. Due to the introduction of the modifying element rhenium into the alloy, the strength is increased to 510 MPa, which is 1.5 times higher compared to the data for the Ti–29Al–7Nb–2Mo system, and the plasticity is increased to 0.6%. The increase in mechanical characteristics due to the modification is achieved by changing the morphology of the structural components of the alloy; the coarse-grained structure of the original alloys with the length of the α_2 - and γ -phase plates of 100–400 μm has changed to thinner lamellae with a thickness of up to 2–3 μm and a length of up to 250 μm .

According to x-ray phase analysis, the phase composition of the al-

loys corresponds to a mixture of α_2 - and γ -phases in different percentage ratios. In the alloy of the basic composition Ti–29Al, the content of the α_2 -phase prevails, with complex alloying and modification in the phase composition of the alloys, the γ -phase with higher values of plastic characteristics prevails. Thus, the introduction of a modifying element does not lead to a change in the phase composition of the alloy, but contributes to an increase in mechanical properties by changing the morphology of the structural components.

4. CONCLUSIONS

1. The optimal concentrations of alloying elements Nb, Mo and modifying element Re in an alloy based on titanium aluminide system have been determined, which ensure an increase in the mechanical properties of the Ti–29Al–7Nb–2Mo–0.2Re alloy.
2. The mechanisms of influence of alloying (molybdenum, niobium) and modifying (rhenium) elements on the structure and properties of the titanium aluminide based alloy have been established: increased plasticity is achieved by weakening the covalent bond forces upon the introduction of molybdenum and niobium and by changing the morphology of the structure due to the surface-active action of the modifiers.
3. Based on the data of x-ray phase analysis, it was established that alloying with molybdenum and niobium leads to a change in the phase ratio in the alloy based on titanium aluminide. In unalloyed aluminide, the content of α_2 -phase prevails (up to $\cong 77\%$). After introducing niobium, molybdenum and rhenium, the alloy mainly consists of γ -phase ($\cong 84\%$) and a small amount of α_2 -phase ($\cong 16\%$). This phase ratio results in increase in mechanical characteristics up to 1.5 times compared to the properties of unalloyed titanium aluminide.
4. By the method of high-temperature thermal analysis, it was established that the introduction of niobium, molybdenum and rhenium into the intermetallic alloy does not change the sequence of transformations and the temperature ranges of existence of phase regions.
5. The obtained level of properties allows us to consider the Ti–29Al–7Nb–2Mo–0.2Re alloy for use as a material for aircraft products.

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