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THE EFFECTS OF GRAIN SIZE ON MECHANICAL PROPERTIES OF Ti-Al INTERMETALLIC ALLOY

ВПЛИВ РОЗМІРУ ЗЕРНА НА МЕХАНІЧНІ ВЛАСТИВОСТІ ІНТЕРМЕТАЛІЧНОГО Ti-Al СПЛАВУ

During the synthesis of intermetallic alloys, the grains of the material are destroyed and transformed into smaller ones through plastic deformation and rapid cooling. By increasing the level of plastic deformation during high-temperature synthesis of the alloy under pressure, even better results can be achieved. For example, adding niobium to γ -TiAl alloys (in a quantity of 7–8 % by weight) and increasing the level of plastic deformation by extrusion at 1100°C allows for the production of a final product with a grain size of only 10–12 μm and a two-level structure with nanolamellar colonies with a distance of up to 500 nm. Theoretical calculations using the Hall-Petch model have shown that a Ti-Al-Nb alloy with such nanostructures can have a ultimate strength of up to 1800 MPa, which is three times higher than that of a Ti-Al alloy.

Keywords: modeling, thermokinetic analysis, intermetallics, thermochemical reaction, thermochemical pressing, activation energy.

Під час синтезу інтерметалідних сплавів зерна матеріалу руйнуються і перетворюються на менші шляхом пластичної деформації та швидкого охолодження. Якщо збільшити рівень пластичної деформації під час високотемпературного синтезу сплаву під тиском, можна досягти ще кращих результатів. Наприклад, додавання ніобію до γ -TiAl сплавів (в кількості 7—8 % за вагою) і збільшення рівня пластичної деформації шляхом екструзії при 1100 °C дозволяє отримати кінцевий продукт з розміром зерна всього 10—12 мкм та дворівневою структурою з наноламельними колоніями з відстанню до 500 нм. Теоретичні розрахунки з моделі Холла-Петча показали, що сплав Ti-Al-Nb з такими наноструктурами може мати граничну міцність до 1800 МПа, що в 3 рази більше, ніж у сплаві Ti-Al.

Ключові слова: моделювання, термодинамічний аналіз, інтерметаліди, термохімічні реакції, термохімічне пресування, енергія активації.

Problem's Formulation

Thermochemical pressing (TCP) is a new type of pressure metal processing technology, in which hot products of thermochemical synthesis, which have not yet had time to cool down, are compacted by external forces (pressing, extrusion, blasting). TCP is a materials manufacturing process that uses high temperature and pressure to activate a chemical reaction between powder materials that results in the creation of bonds between atoms and the formation of a solid material.

The TCP process is used to manufacture a variety of materials, including ceramics, metal alloys, solid solutions, and composite materials. In the TCP process, material powders are placed according to the composition and desired properties of the final product, after which they are subjected to high pressures and temperatures. Under the influence of these conditions, the powders interact and react, forming a solid matrix with the desired properties.

The application of TCP is broad and includes the production of materials for high-tech industries such as aviation, space, electronics and medicine. This process makes it possible to obtain materials with balanced physical and mechanical properties that meet the needs of modern production.

In the TCP process, powdered materials are placed in a mold, after which it is subjected to compression using a press, which usually works on the principle of hydraulics or electric current. After compression, the mold with the material is heated to high temperatures, which activate a chemical reaction between the materials. This leads to the fact that the powder particles begin to combine, forming a solid material with bonds between atoms.

TCP can be used to fabricate a wide range of materials, including ceramics, metals, cermets, carbides, nitrides, oxides, and others. This process makes it possible to produce materials with high density, high strength and high precision of form. Grinding of the intermetallic alloy grain in the process of its synthesis under pressure occurs as a result of plastic deformation of the synthesis product and high cooling rates. A higher efficiency of the process of grinding the grains of the structure of the intermetallic alloy can be achieved during the plastic deformation of the synthesized alloy in the process of forming the grains of the structure during the high-temperature synthesis of the alloy under pressure. For example, by extruding the synthesized intermetallic alloy through a hole (caliber) in a mold directly during high-temperature synthesis under pressure.

Research on TCP is carried out in many scientific institutes, universities and industrial enterprises around the world. Scientific research on TSR is aimed at improving technologies and expanding applications of this process in various fields.

For example, TCP research may include studying the reactions between powder materials, analyzing the mechanical and physical properties of the resulting materials, and optimizing TCP parameters such as temperature, pressure, time, and powder mixture composition.

Researchers from different countries, in particular, the USA, Japan, China, India, Europe and other countries, are working in this direction. The results of their research are used to create new materials with various properties for use in various fields, such as mechanical engineering, energy, medicine, and others.

Analysis of recent research and publications

Depending on the technology of preparation of blanks, modes of hot deformation and heat treatment, it is possible to obtain three main types of TiAl intermetallic structures: lamellar (lamellar),

recrystallized and mixed (duplex) [1]. The mechanical properties of intermetallic TiAl at room temperature with these types of structures are shown in Tabl. 1.

Table 1. Influence of the type of TiAl intermetallic structure on mechanical properties

Structure type	Mechanical properties		
	σ_s , MPa	δ , %	σ_{100}^{700} , MPa
Lamelna	350...400	0,5	480
Recrystallized	580	0,8	310
Duplex	550	1,5	380

Fine-grained intermetallic TiAl shows a certain tendency to superplasticity: at a temperature of 800 °C and a deformation rate of $8.3 \times 10^{-4} \text{ s}^{-1}$, the relative elongation is more than 225 % [2].

Therefore, due to low density and sufficiently high strength characteristics, intermetallic alloys based on TiAl are superior to actual heat-resistant alloys based on titanium, iron, and nickel in terms of specific values of elastic moduli and heat resistance indicators in the temperature range up to 850...900 °C and can be selected as the basis for the development of multicomponent intermetallic alloys based on titanium aluminides.

It is possible to increase the plasticity and strength of NiAl and TiAl intermetallics by grinding the grain and increasing the purity of the starting materials. At the same time, alloying is one of the most effective ways to increase the complex of physical and mechanical properties.

Formulation of the study purpose

The purpose of the work is to establish the regularities of the mechanism of obtaining a compact intermetallic alloy with a highly dispersed structure.

Presenting main material

The fine-dispersed two-phase duplex structure of γ -TiAl alloys (Fig. 1) has the best plasticity, but at the same time another equally important characteristic — the viscosity of the alloy — decreases. The best option is to obtain alloys with a completely lamellar two-phase (γ/α_2) structure with a certain amount of γ - and α_2 -phase in the alloy [3].

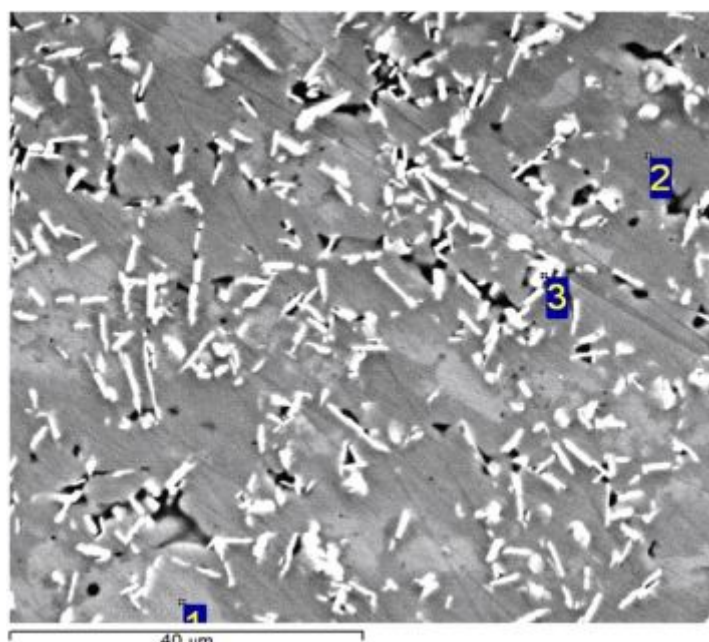


Fig. 1. Microstructure of the obtained two-phase (γ/α_2) intermetallic TiAl alloy

In order to achieve the maximum possible high-temperature strength, resistance to oxidation, and room temperature plasticity in products, it is crucial to alloy with a refractory impurity of Nb [4]. The addition of Nb has a significant impact on the volume fraction of the α_2 phase and the average interlamellar distance [5]. Therefore, by doping γ -TiAl alloys with 7—8 % niobium by mass and increasing the degree of deformation of the intermetallic product synthesized under pressure at a deformation temperature of 1100 °C, the grain size in the final product can be reduced by an order of magnitude (to 10—12 μm).

Analysis of the microstructure of the synthesized titanium aluminides showed that a feature of the formation of finer structures during the high-temperature synthesis of compositions based on Ti-Al-Nb is the increased content of the β -stabilizing element. As a result, a thin composite texture is formed, consisting of alternating parallel lamellae of two different crystal phases: tetragonal γ -phase (TiAl) and hexagonal α_2 -phase (Ti_3Al) (Fig. 2). Thus, a two-level structure is formed: each polycrystalline α -grain forms a limited lamellar colony, which consists of thin lamellae with an interlamella distance of 500 nm [6].

The theory of Hall-Petch gives the following dependence of the value of the short-term yield strength of the material $\sigma_{0.2}$ on the size of the grain d and the thickness of the lamellae λ during phase segregation [7]:

$$\sigma = \sigma_0 + \frac{k_d}{\sqrt{d}} + \frac{k_\lambda}{\sqrt{\lambda}}, \quad (1)$$

where σ_0 is the yield strength of the textured material; k_d, k_λ — material constants.

The mechanical properties of the two-level structure can be improved when the structural-phase segregation parameters d and λ change from the micron to the nanoscale level (Fig. 2). In addition to thermal conditions, the segregation parameters are optimized by introducing modifying alloying impurities that affect the values of the k_d and k_λ coefficients. The best result is achieved when these factors work together.

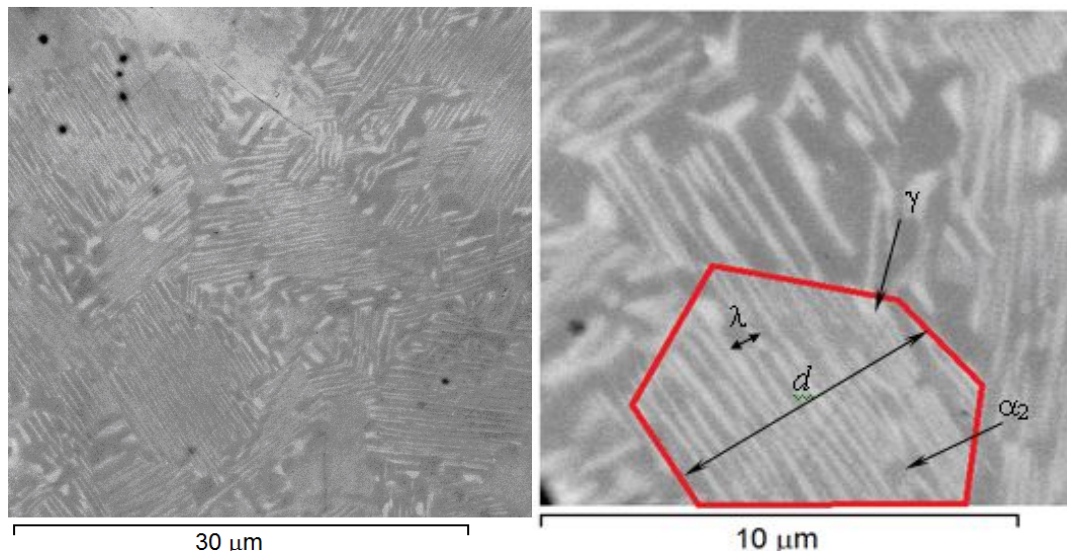


Fig. 2. Grain size d and lamellar distance of colonies λ in intermetallic Ti-Al-Nb alloy

It was established that doping γ -TiAl alloys with niobium (7...8 % by weight) and increasing the degree of plastic deformation under extrusion conditions at a deformation temperature of 1100 °C allows to significantly reduce the grain size in the final product (to 10...12 μm) and form a two-level structure with nanolamellar colonies with a distance of up to 500 nm. Theoretical calculations carried out using the Hall-Petch model showed that the Ti-Al-Nb alloy with nanostructures of lamellae has an ultimate strength of up to 1800 MPa, which is 3 times greater than that of the Ti-Al alloy (Fig. 3).

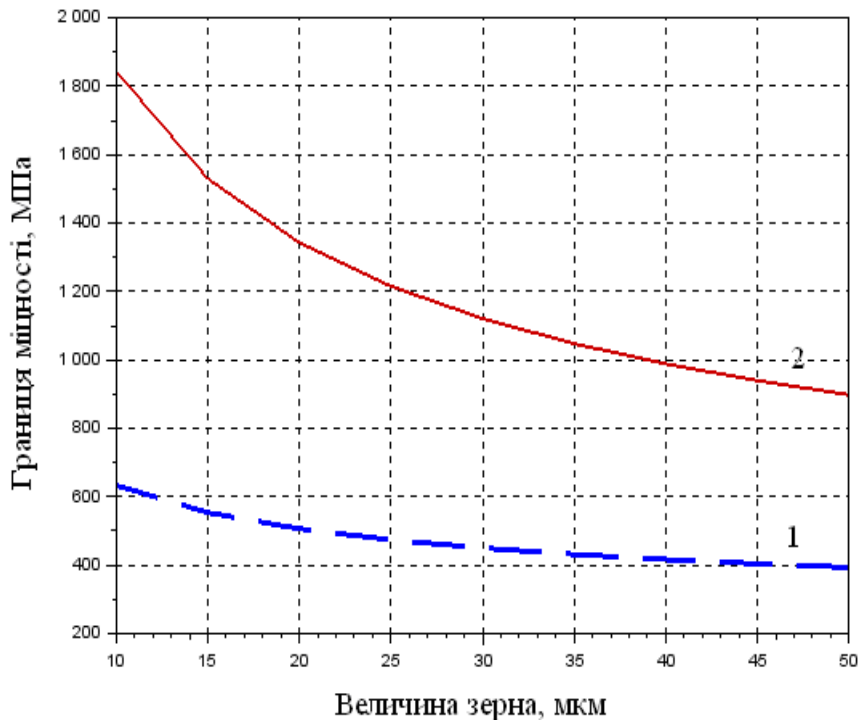


Fig. 3. Dependence of strength limit σ on grain size d : 1 — *Ti-Al*; 2 — *Ti-Al-Nb*

The mechanical properties of the two-level structure can be improved by changing the parameters of the structural-phase segregation d_f from the micron to the nanoscale level. In addition to thermal conditions, the segregation parameters are optimized by the introduction of modifying impurities that affect the value of the K_f coefficient. The best result is achieved when these factors work together. Therefore, the strength limit of intermetallic alloys:

For cast intermetallic *TiAl* alloy:

$$\sigma_s = 344 + 2,25 \cdot 0,00025^{-\frac{1}{2}} = 486 \text{ MPa};$$

for deformed *TiAl* alloy:

$$\sigma_s = 368 + 1,60 \cdot 0,00052^{-\frac{1}{2}} = 590 \text{ MPa};$$

for the intermetallic *Ti₃Al* alloy:

$$\sigma_s = 250 + 1,26 \cdot 0,00036^{-\frac{1}{2}} = 460 \text{ MPa};$$

for the intermetallic *TiAl₃* alloy:

$$\sigma_s = \sigma_i + K_f d_f^{-\frac{1}{2}} = 200 + 1,37 \cdot 0,0003^{-\frac{1}{2}} = 450 \text{ MPa}.$$

The resulting microstructure and mechanical properties of intermetallic phases of the *Ti-Al* system are presented in *Tabl. 2*.

Table 2. Microstructure and mechanical properties

	<i>TiAl</i> (cast)	<i>TiAl</i> (deform)	<i>Ti₃Al</i>	<i>TiAl₃</i>
Microstructure, μm	250	52	36	30
Yield strength σ_y , MPa	344	368	250	200
Strength limit σ_s , MPa	486	590	460	450
Relative elongation δ , %	1,5	3	0,5	2

An increase in strength is usually accompanied by a decrease in ductility and fracture toughness. This happens because high-strength materials have little energy absorbed during destruction. The level of this energy during brittle failure is determined by the size of the plastic zone in front of the crack front. The reduction of the plastically deformed volume (and therefore the work of plastic deformation) when testing for hardness and fracture toughness is a consequence of the same phenomenon — a decrease in the mobility of dislocations [8, 9]. If a residual dent occurs when the indenter is inserted into the surface of the counterbody in the contact zone, then there is always a plastically deformed area around it, which extends to a certain depth h_s . This region is limited by a closed surface on which the plasticity condition ($\sigma_i = \sigma_y$) is satisfied.

The magnitude of the stress intensity at the contact center σ_i is proposed to be determined by the formula [10]:

$$\sigma_i = \frac{1,5 \cdot A \cdot p_0 \cdot \varepsilon_i}{3\varepsilon_i + \exp[-1,5\varepsilon_i + C(z - R)]}, \quad (2)$$

where p_0 is the pressure in the contact center; R — indenter radius; ε_i — intensity of elastic-plastic deformation; A and C — constant, defined as:

$$A = \frac{h_s^2}{\ln \frac{h_s + R}{R} \sqrt{(h_s^2 + a^2)^3}}, \quad (3)$$

$$C = \frac{1}{h_s} \left[-1,5\varepsilon_i + \ln 3\varepsilon \left(\frac{A \cdot p_0}{2\sigma_T} - 1 \right) \right], \quad (4)$$

where h_s is the depth of propagation of plastic deformation; a — the radius of the contact area.

The depth of the expansion of the plastic zone when pressing the spherical indenter h_s was found using the formula [10]:

$$h_s = \sqrt{\frac{P}{2\sigma_T} - 1,2 \left(\frac{d}{2} \right)^2}, \quad (5)$$

where P is the applied load; σ_T — yield strength of the material; d — value of the diameters of the prints obtained.

To establish the effect of microstructure on fracture toughness, it is necessary to have full knowledge of K_{IC} as a change in microstructure. For the brittle failure of a TiAl-based alloy with controlled stress can be obtained using the classical theory of brittle failure developed by A. Tetelman [11], which was used to describe the fracture behavior of high-strength materials:

$$K_{IC} = 2,89\sigma_T \left[\exp \left(\frac{\sigma_i}{\sigma_T} - 1 \right) \right]^{1/2} \sqrt{P}. \quad (6)$$

To study the depth of the plastic zone, contact deformation was carried out with a spherical indenter with a diameter of $D = 5$ mm under loads P from 147 to 11,875 N at a temperature of 293 K on Brinell and Rockwell devices.

As a result of the study, it was found that the mechanism of failure in the alloy based on the γ -phase is the formation of cracks and microcracks. The highest value of K_{IC} is observed in a completely lamellar microstructure and can be mainly associated with the lamellar connection of phases and the exfoliation of the interface of the γ/α_2 phase. It is obvious that there is a large number of bonds for the lamellar microstructure, on the other hand, small shear bonds can be found in the nearby gamma microstructure. The formation of a shear bond was proposed as the main mechanism of impact toughness in the intermetallic TiAl alloy. The microstructure dependence of the fracture stress σ_i is similar to the ultimate stress σ_s , as shown in Tabl. 3, which means that the inverse relationship between plasticity and viscosity is due to the different response of each microstructure to the stress state, and not to a change in the fracture mechanism [12, 13].

Table 3. Investigation of resistance to brittle fracture of intermetallic Ti-Al alloys

Параметр	TiAl(cast)	TiAl (deform)	Ti ₃ Al	TiAl ₃
$K_{IC}, MPa \sqrt{M}$	23	29	26	21
σ_b, MPa	400	480	440	380
Strength limit σ_s, MPa	486	590	460	450

For a uniaxial tensile test, the stress is distributed uniformly and the elongation mainly depends on the failure stress σ_s . The magnitude of the degree of the fracture process zone, in which the critical stress must exist to cause fracture, is of the same order as the previous colony size or the size of the γ -phases. The formation of fine-grained structures, including the fragmentation of excess phases, will in the long run increase the resistance and increase the work of crack propagation of alloys based on the obtained titanium aluminide alloy. It was established that the strength of the alloy based on the γ -phase is controlled by the microcrack nucleation stage in the grain with the maximum size. The nucleation of a crack in a lamellar microstructure is more difficult than in a grain of a duplex structure, provided the grain size is the same. The fully lamellar microstructure shows the highest fracture toughness K_{IC} and modulus of rupture: on the other hand, the near-gamma structure and duplex microstructure show the lowest K_{IC} fracture toughness and modulus of rupture.

To achieve optimal properties for a TiAl alloy product, it is necessary to subject it to plastic deformation at high temperatures, which results in a lamellar structure. This structure provides a combination of high-temperature properties, such as strength and creep resistance, with room-temperature properties like plasticity and fracture toughness. Plastic deformation is an effective method not only for obtaining fine-grained semi-products, but also for controlling the parameters of the lamellar structure in TiAl alloys, especially when producing lamellar microstructures with small colony sizes and nanocrystalline interlamellar spacing. These structures are of great practical interest, according to the literature. Synthesis processes and dynamic compaction of the synthesis product can be used to obtain a compact intermetallic alloy with a highly dispersed structure, whose grain size is significantly smaller than that of alloys obtained by casting (grain size $d \sim 100 \mu m$), sintering ($d \sim 50 \mu m$), or shock wave action ($d \sim 35 \mu m$) on the synthesized product [14, 15].

Conclusions

Plastic deformation can be effective not only for obtaining fine-grained semi-products, but also for controlling the parameters of the lamellar structure in intermetallic alloys, in particular, when obtaining lamellar microstructures with a small size of colonies and a nanocrystalline interlamellar distance, which represent, according to the literature, the greatest practical interest.

It has been found that by adding niobium to γ -TiAl alloys (at a weight percentage of 7–8 %) and increasing the degree of plastic deformation through extrusion at a temperature of 1100 °C, it is possible to significantly decrease the grain size in the final product (down to 10–12 μm) and form a two-level structure consisting of nanolamellar colonies with a spacing of up to 500 nm. Theoretical calculations based on the Hall-Petch model have demonstrated that producing a Ti-Al-Nb alloy with nanostructures of lamellae can result in ultimate strength of up to 1800 MPa, which is three times higher than that of a Ti-Al alloy.

The intermetallic alloy grain is ground during its synthesis under pressure due to plastic deformation of the synthesis product and high cooling rates. To make the process of grinding the intermetallic alloy grain more efficient, it is necessary to apply intensive plastic deformation during the high-temperature synthesis of the alloy under pressure to form the grain structure. Recently, it has been discovered that by deforming the alloy through thermochemical pressing, it is possible to produce a material based on TiAl with a grain size of 20 to 30 μm and a relative elongation of 4 % at room temperature. Additionally, the formation of fine-grained structures, including the fragmentation of excess phases, can enhance the resistance and increase the work of crack propagation of alloys based on the titanium aluminide alloy produced.

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