Theory and Processes of Formation and Sintering of Powder Materials
Теория и процессы формования и спекания порошковых материалов

UDC 621.762.016
https://doi.org/10.17073/1997-308X-2025-4-28-39

Research article
Научная статья

Features of densification, structure formation, and properties of powder titanium under hot die forging

V. Yu. Dorofeyev[1], A. N. Sviridova[1], S. V. Sviridova[2], L. I. Svistun[3]

[1] **Platov South-Russian State Polytechnic University (NPI)**
132 Prosveshcheniya Str., Rostov region, Novocherkassk 346428, Russia
[2] **Tambov State University named after G.R. Derzhavin**
33 Internatsionalnaya Str., Tambov 392000, Russia
[3] **Kuban State Technological University**
2 Moskovskaya Str., Krasnodar 350072, Russia

dvyu56.56@mail.ru

Abstract. Research in the field of titanium powder metallurgy has been ongoing for more than 60 years. Nevertheless, there are relatively few examples of the practical application of powder titanium, which is associated with insufficient reliability and durability of the manufactured products. The ability of titanium parts to withstand static and dynamic loads is determined by residual porosity, non-metallic inclusions, and microstructural characteristics. At present, the most widely used method for producing powder titanium components is the press–sinter route. However, the porosity of sintered titanium typically ranges from 3 to 15 %, which reduces its load-bearing capacity and highlights the need for effective methods to minimize porosity. Hot working methods, particularly hot die forging of porous preforms, hold considerable potential in addressing this issue. This study presents the results of investigating the features of densification, structure formation, and properties of powder titanium under hot die forging. A technology for producing hot-forged powder titanium is proposed, which includes hydriding–dehydriding of porous preforms. This operation promotes the reduction of oxides localized on the surfaces of open pores by hydrogen and their activation, thereby improving conditions for interparticle bonding during subsequent hot repressing. As a result, the obtained samples demonstrate higher fracture toughness and ductility compared with reference samples. The values of the maximum specific work of hot densification of porous powder titanium, required to achieve monolithic density at different preheating temperatures of the preforms, were determined. It was shown that the non-monotonic temperature dependence of the maximum specific densification work is associated with the formation of a coarse-grained structure and with reduced ductility of the deformable material in the temperature range of the $\alpha \rightarrow \beta$ phase transformation.

Keywords: hot die forging, porous preforms, powder titanium, densification work, fracture toughness, ductility, strength, ductile fracture, interparticle fracture, oxide reduction, hydriding, dehydriding, interparticle bonding, activation

Acknowledgements: The SEM images were obtained using a Quanta 200 i 3D microanalyzer at the Nanotechnology Shared Research Facility of Platov South-Russian State Polytechnic University. The authors express their gratitude to Höganäs Eastern Europe for providing the ABC100.30 iron powder manufactured by Höganäs AB.

For citation: Dorofeyev V.Yu., Sviridova A.N., Sviridova S.V., Svistun L.I. Features of densification, structure formation, and properties of powder titanium under hot die forging. *Powder Metallurgy and Functional Coatings*. 2025;19(4):28–39. https://doi.org/10.17073/1997-308X-2025-4-28-39

Особенности уплотнения, формирования структуры и свойств порошкового титана при горячей штамповке

В. Ю. Дорофеев[1], А. Н. Свиридова[1], С. В. Свиридова[2], Л. И. Свистун[3]

[1] **Южно-Российский государственный политехнический университет (НПИ) имени М.И. Платова**
Россия, 346428, Ростовская обл., г. Новочеркасск, ул. Просвещения, 132
[2] **Тамбовский государственный университет имени Г.Р. Державина**
Россия, 392000, г. Тамбов, ул. Интернациональная, 33
[3] **Кубанский государственный технологический университет**
Россия, 350072, г. Краснодар, ул. Московская, 2

dvyu56.56@mail.ru

Аннотация. Работы в области порошковой металлургии титана проводятся уже более 60 лет. Несмотря на это, примеров практического использования порошкового титана не так много, что связано с неудовлетворительным характером показателей надежности и долговечности получаемых изделий. Способность титановых изделий сопротивляться воздействию статических и динамических нагрузок определяется наличием остаточной пористости, неметаллических включений, а также характеристиками микроструктуры. В настоящее время при изготовлении изделий из порошкового титана наибольшее распространение получила технология прессования–спекания. Однако пористость спеченного титана составляет 3–15 %, что снижает его сопротивляемость действию нагрузок и обусловливает актуальность разработки эффективных методов снижения пористости. Большой потенциал в решении указанной задачи имеют методы горячей обработки давлением, в частности горячая штамповка пористых заготовок. В работе представлены результаты исследования особенностей уплотнения, формирования структуры и свойств порошкового титана при горячей штамповке. Предложена технология получения горячештампованного порошкового титана, включающая выполнение операций гидрирования–дегидрирования пористой заготовки, обеспечивающих восстановление оксидов, локализованных на поверхностях открытых пор, водородом и их активизацию, что способствует улучшению условий формирования межчастичного сращивания при последующей горячей допрессовке и повышению трещиностойкости и пластичности получаемых образцов в сравнении с образцами-свидетелями. Установлены значения величины максимальной приведенной работы горячего уплотнения пористого порошкового титана, необходимой для достижения плотности монолита, при разных температурах преддеформационного нагрева заготовок. Показано, что немонотонность температурной зависимости максимальной приведенной работы уплотнения связана с формированием крупнозернистой структуры и с уменьшением пластичности деформируемого материала в интервале температур фазового α → β-превращения.

Ключевые слова: горячая штамповка, пористые заготовки, порошковый титан, работа уплотнения, трещиностойкость, пластичность, прочность, вязкий излом, межчастичное разрушение, восстановление оксидов, гидрирование, дегидрирование, межчастичное сращивание, активация

Благодарности: Снимки на растровом микроскопе-микроанализаторе Quanta 200 i 3D получены в Центре коллективного пользования «Нанотехнологии» ЮРГПУ (НПИ). Авторы выражают благодарность компании «Хёганес Восточная Европа» за предоставленный железный порошок ABC100.30 производства фирмы Höganäs AB.

Для цитирования: Дорофеев В.Ю., Свиридова А.Н., Свиридова С.В., Свистун Л.И. Особенности уплотнения, формирования структуры и свойств порошкового титана при горячей штамповке. *Известия вузов. Порошковая металлургия и функциональные покрытия*. 2025;19(4):28–39. https://doi.org/10.17073/1997-308X-2025-4-28-39

Introduction

The unique properties of titanium – its high specific strength, corrosion resistance, and good biocompatibility – determine the wide application of this metal and its alloys in aerospace, automotive engineering, medicine, and other industries [1]. The prospects of powder metallurgy technologies for manufacturing titanium and titanium alloy products are largely driven by the high cost of alternative casting technologies [2; 3]. The production of cast titanium is further complicated by significant losses during machining. The average material utilization factor in manufacturing titanium products from wrought stock does not exceed 18 % and is often much lower [4].

Research in titanium powder metallurgy has been ongoing for more than 60 years. Nevertheless, there are still relatively few examples of its practical application. In many cases, consumers prefer cast titanium over powder-based titanium due to unsatisfactory

mechanical properties and/or high cost of the latter [1]. Reducing the cost of powder titanium products requires the development of economically efficient technologies both for producing titanium powder and for fabricating finished parts [5].

One promising approach to lowering the cost of titanium powder is the use of by-products from sponge titanium production as the raw material. Another promising route is the *hydride–dehydride* (HDH) process, which involves mechanical milling of pre-hydrided sponge titanium, turnings, scrap, and other machining waste, followed by dehydrogenation of the milled material [6–8].

Improving the mechanical properties of powder titanium products during processing is also of critical importance, although success in this area often directly depends on the quality of the starting powder [9]. The ability of titanium products to resist static and dynamic loads is determined by the presence of residual porosity, non-metallic inclusions, and microstructural characteristics [10]. The residual porosity of powder titanium depends on the production technology. In recent decades, the press–sinter method has become the most widespread [1; 11; 12]. Porosity in sintered (undeformed) titanium typically ranges from 3 to 15 %. Since pores act as stress concentrators, they reduce the effective cross-section of the sample and diminish its load-bearing capacity. Therefore, improving the physical and mechanical properties of powder titanium requires the development of effective methods to reduce porosity [1].

The use of fine, amorphous, and nanostructured powders, thermocycling in the $\alpha \rightarrow \beta$ phase transformation range (t = 800–1100 °C), thermomechanical treatment, and spark plasma sintering are effective methods of sintering activation [11; 13–15]. In addition, the use of activating additives also provides positive results; these are conventionally divided into two types [16].

Additives of the first type activate self-diffusion of the base element, thereby improving densification conditions during sintering. The diffusion rates of transition metals and phosphorus in α-Ti are 3–5 orders of magnitude higher than its self-diffusion [17]. Self-diffusion of titanium is also enhanced by elements that reduce the solidus temperature of the alloy [16].

The second type of additives includes elements that promote the formation of a transient liquid phase, which disappears during sintering and provides effective diffusion pathways for mass transport, thereby increasing the density of sintered products. Moreover, liquid-phase formation may be related to eutectic reactions between two precipitated phases (solid–liquid sintering). Significant sintering activation is observed during liquid-phase sintering of amorphous titanium powders [13]. Powders of iron, nickel, silicon, cobalt, and copper are used as activators [18–26]. However, liquid-phase sintering technology has several drawbacks: distortion of the shape of preforms, segregation of solid and liquid phases, and rapid grain growth, all of which negatively affect the mechanical and service properties of the products.

High diffusion rates are also observed during sintering of titanium powder produced by the hydride–dehydride process [27]. This is due to the formation of a large number of lattice defects during dehydrogenation, which promote diffusion processes [28].

Among the more recent technologies of sintering activation for powder titanium are induction vacuum sintering methods and FAST (*Field-assisted sintering technology*), which employs strong electromagnetic fields [29; 30]. A major limitation of these methods is the need for expensive specialized equipment, which significantly increases production costs [31].

Limiting factors also include the low ductility and toughness of powder titanium and its alloys, which adversely affect their fracture toughness and fatigue strength [32; 33]. In this regard, it is worth recalling a recent event. At the 2021 Tokyo Olympics, an accident occurred involving the bicycle of an Australian rider, caused by the fatigue failure of handlebars manufactured by additive technology from Ti–6Al–4V powder alloy [34; 35].

Although the specific strength of powder titanium is about four times higher than that of steels, its resistance to cyclic loading is often low due to its limited ductility. In addition to porosity, ductility, fracture toughness, and fatigue strength are negatively affected by interstitial impurities (O, H, N, C) and microstructural characteristics [32]. The presence of impurities is associated with the high chemical affinity of titanium for these elements, necessitating heating operations in vacuum or inert atmospheres.

The dependence of fatigue life on microstructural characteristics is determined by the slip length during plastic shear of the crystal lattice. In titanium-based materials, this corresponds to the α-phase grain diameter in equiaxed structures, the α-phase plate width in basket-weave structures, or the colony size in lamellar structures [33]. A reduction in slip length due to microstructural refinement contributes to improved fatigue strength. It should be noted that prolonged high-temperature exposure during sintering leads to grain growth. This highlights the relevance of employing methods in powder titanium processing that enable

simultaneous reduction of porosity and refinement of structural constituents.

A promising approach to addressing this challenge lies in hot deformation methods. The first studies in this area were carried out in the late 1950s to early 1960s. At Novocherkassk Polytechnic Institute, I.N. Goncharov proposed a method of hammer forging titanium powder particles or crushed titanium sponge to achieve welding [36]. Densification was performed under isothermal conditions in a preheated die at 900–920 °C. The resulting samples exhibited high density (4.46–4.48 g/cm^3), along with high ductility and deformability of forged powder titanium. Since the starting material subjected to hot deformation was powder or sponge particles, this method developed by I.N. Goncharov can, in modern terminology, be classified as *direct powder forging* (DPF).

In industrial practice, hot forging of loose powder prepacked into cans was introduced later, in the 1970s, for the production of high-alloy tool steels [37]. More recently, studies have investigated the feasibility of applying DPF technology to titanium powder processing [31; 38]. A high-density Ti–6Al–4V powder material with a homogeneous lamellar two-phase (α + β) structure was obtained, characterized by high ductility, low impurity content both on the surface and in the bulk of the experimental samples, and only minor deviations from the chemical composition of the starting powder. Using this technology, a femoral implant was fabricated that meets the requirements of ASTM F136-13 (2021) [39].

One advantage of the DPF process is that the high stresses and strains generated during forging at the powder – can interface promote the fracture of intermetallics and oxides formed there, facilitating subsequent can removal. However, this advantage may present a problem: after can removal, the powder material surface exhibits a fracture-like relief, necessitating additional machining.

Research on hot forging of porous titanium preforms (HFPTP) was conducted by S.S. Kiparisov and co-authors in the late 1960s to early 1970s [11, pp. 47, 48]. These studies established the optimal parameters for HFPTP: heating temperature of 900 °C and impact energy of 2000–2500 kJ/m^2. However, the upper limit of the heating temperature range did not exceed 900 °C, and the impact energy values were expressed per unit area rather than per volume of the sample in the compact state, which complicates their practical application for densification.

Subsequently, V.A. Pavlov and co-authors carried out research on hot deformation of titanium powders and other non-ferrous metals [40; 41]. They examined the forging behavior of porous titanium preforms in open and closed dies, heating under various conditions (unprotected, protected with glass coatings, in argon, and in vacuum), as well as deformability characteristics and energy consumption during cold hydrostatic pressing of titanium powder. However, the energy requirements for densifying powder titanium to a pore-free state by hot forging were not determined. Such data are also absent in the works of Yu.G. Dorofeev and his scientific school. The lack of information on the parameter W_{max} (maximum specific densification work, i.e., the work required to reach monolithic density [42; 43]) for HFPTP complicates the selection of technological modes, since densification energy is one of the key control parameters in dynamic consolidation of powder materials [44].

The aim of this work was to study the densification behavior, microstructure formation, and properties of powder titanium during *hot die forging* (HDF).

Experimental procedure

The experimental samples were fabricated from electrolytic titanium powder grade PTES-1 according to TU 48–10–22–85. A powder fraction of –0.63 + 0.18 mm with a bulk density of 1.5·10^3 kg/m^3 was used. The processing flowcharts for sample fabrication are shown in Fig. 1. At the initial stage of the study, the energy characteristics of hot densification were determined using cylindrical samples with dimensions ∅20 × 8 mm (scheme *1*). The ratio of the final height of the sample (h_f) to its diameter (d) was kept constant (h_f/d = 0.4 = const). The porosity of the cold-pressed billets was in the range of 22–25 %. After heating to various temperatures (800–1050 °C), the billets were further compacted on a laboratory drop hammer with a falling weight of 50 kg. The densification work (W) was varied in the range of 30–150 MJ/m^3. Preheating of the billets before further compaction was carried out in a portable heat-resistant steel container into which helium was supplied. The container with the sample was then placed into a laboratory muffle furnace with silicon carbide heating elements, which was also purged with helium.

The value of W_{max} was determined by a graphical–analytical method, processing the plots of $\lg W = f(\lg\beta)$ according to Dorofeev's methodology (β – relative volume) [42; 43]. The density of the samples was measured by hydrostatic weighing in accordance with GOST 18898–89.

At the next stage of the study, prismatic samples with dimensions 130×26×15 mm were fabricated to determine the mechanical properties and conduct microstructural analysis. To reduce the probability

of oxidation, the billets for prismatic samples were made bimetallic. The outer layer (sheath) of the billets was produced from high-pressibility iron powder grade ABC100.30 (Höganäs AB).

After HDF, the billets were subjected to machining. The fracture toughness (K_{1c}) was determined on type 4 samples (GOST 25.506–85) with dimensions 12.5×25×112.5 mm containing a pre-induced crack using the three-point bending method. The crack was initiated under cyclic loading. The values of K_{1c} were calculated according to the procedure [45]. Tensile tests were carried out on type II Gagarin samples (GOST 1497–84) with a working section diameter d_0 = 5 mm and a gauge length l_0 = 25 mm.

Based on the results of the first stage of HDF testing, the mechanical properties and fracture toughness were determined at a specific densification work of W = 195 MJ/m^3 Since fully dense samples were not obtained in the first stage, the pre-deformation heating temperature of the billets was varied in the range of t_{HDF} = 1000÷1200 °C.

During the fabrication of samples according to technological scheme *3*, porous cold-pressed billets were subjected to hydrogenation in hydrogen, followed by dehydrogenation during vacuum sintering. This step was intended to reduce impurity content and activate interparticle surfaces for subsequent hot forging, similar to the previously observed effect of hydrogenation–dehydrogenation of initial titanium powders during sintering [27]. For comparative analysis, control samples were produced according to technological scheme *2*, which did not include the hydrogenation step of cold-pressed billets.

The fracture surfaces of the samples were examined using a Quanta 200 i 3D scanning electron microscope–microanalyzer and an MBS 9 binocular microscope. The fraction of fracture surface components was determined by grid and area methods applied to electron microscopic images [46], with 10–12 fields analyzed. Metallographic studies were performed with an AltamiMET 1M optical microscope (Altami Ltd., Russia) on both etched and unetched polished sections. Kroll's reagent (2 ml HNO_3 + 2 ml HF + 96 ml H_2O) was used as the etchant, with an etching time of 20 s.

Results and discussion

Fig. 2 presents the $\lg W = f(\lg\beta)$ dependences for porous titanium billets subjected to different pre-deformation heating temperatures. The relationships between the relative volume of powder titanium and the specific densification work, plotted in logarithmic coordinates, exhibit a linear form, which is characteris-

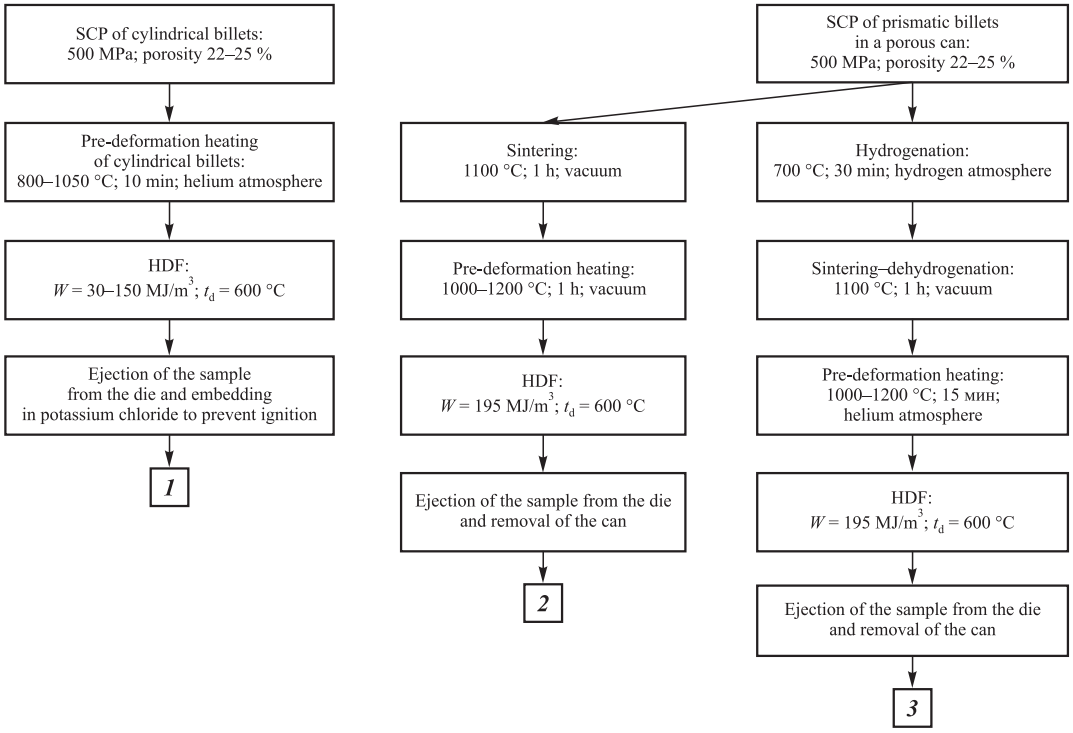

Fig. 1. Processing flowcharts for producing hot die forged powder titanium

SCP – static cold pressing; t_d – preheating temperature of the die matrix for HDF; W – specific densification work

Рис. 1. Технологические схемы получения горячештампованного порошкового титана

SCP – статическое холодное прессование; t_d – температура подогрева матрицы пресс-формы для ГШ; W – приведенная работа уплотнения

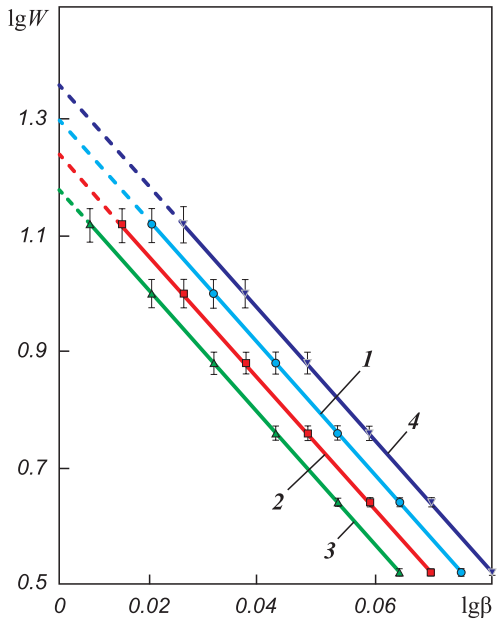

Fig. 2. lg$W = f$(lgβ) dependences during hot die forging of cold-pressed titanium powder billets

t_{HDF}, °C: ***1*** – 800, ***2*** – 900, ***3*** – 950, ***4*** – 1000; $h_f/d = 0.4$

Рис. 2. Зависимости lg$W = f$(lgβ) при горячей штамповке холоднопрессованных заготовок из титанового порошка

t_{HDF}, °C: ***1*** – 800, ***2*** – 900, ***3*** – 950, ***4*** – 1000; $h_f/d = 0{,}4$

tic of ductile materials [42]. The W_{max} were determined by extrapolating curves *1–4* to their intersection with the ordinate axis (dashed lines).

The dependence of the W_{max} values determined in this way on the hot die forging temperature is shown in Fig. 3. It is evident that the dependence is nonmonotonic. Increasing t_{HDF} in the 800–950 °C range leads to a decrease in W_{max} due to the enhanced plasticity of the α-phase. At t_{HDF} = 950–1000 °C, the W_{max} value increases, which is attributed to the α → β phase transformation and the formation of the β-phase, characterized at these temperatures by reduced plasticity

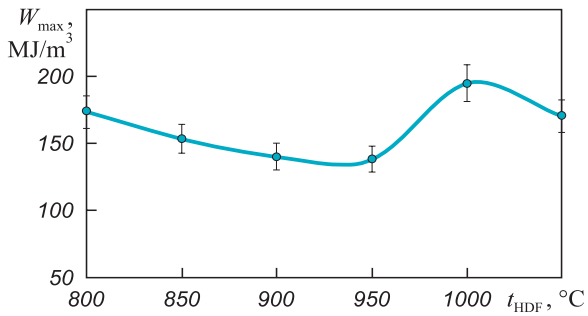

Fig. 3. Dependence of the maximum specific densification work on the heating temperature of porous titanium billets

Рис. 3. Зависимость максимальной приведенной работы уплотнения от температуры нагрева пористых заготовок из титана

and higher strength. At t_{HDF} > 1000 °C, the plasticity of the β-phase increases, resulting in a decrease in W_{max}.

The effect of phase transformations on densification behavior and deformability has been previously reported in studies of dynamic and explosive hot pressing of porous iron-based billets [42; 43]. With regard to titanium and its alloys, the literature data remain contradictory [11]. Some authors indicate a monotonic increase in plasticity with temperature across all titanium alloys [47], whereas others note a nonmonotonic trend [48]. The reduction in plasticity in the α → β transformation range has been linked to the development of a coarse-grained structure [49].

In our experiments, the grain size of samples fabricated at t_{HDF} = 950 °C, was 10–20 μm (Fig. 4, *a*), corresponding to grades 2–3 on the titanium alloy microstructure scale used for metallurgical quality control [49]. At t_{HDF} = 1000 °C, the grain size increased to 25–35 μm (grades 5–6) (Fig. 4, *b*). This provides sufficient grounds to assume that the nonmonotonic

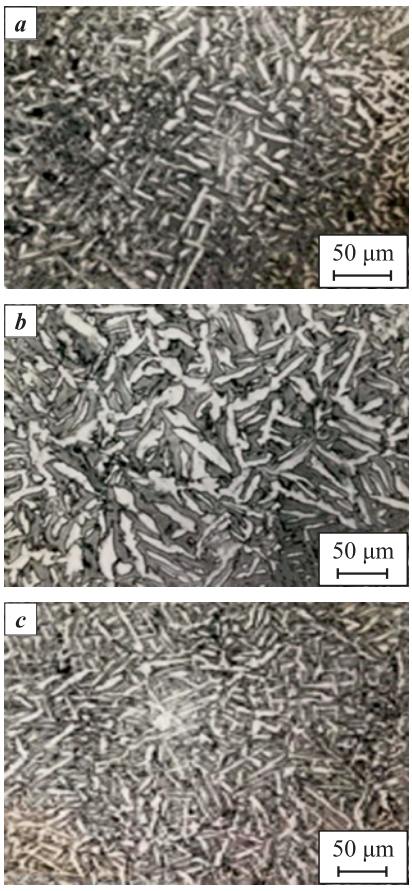

Fig. 4. Microstructure of hot die forged powder titanium

a, ***b*** – scheme *1*, ***c*** – scheme *2*; t_{HDF}, °C: 950 (***a***), 1000 (***b***) and 1200 (***c***)

Рис. 4. Микроструктура горячештампованного порошкового титана

a, ***b*** – схема *1*, ***c*** – схема *2*; t_{HDF}, °C: 950 (***a***), 1000 (***b***) и 1200 (***c***)

$W_{max} = f(t_{HDF})$ dependence is also associated with grain growth during phase transformation. The samples exhibited a lamellar intragranular α-phase structure with areas of basket-weave microstructure. This morphology is related to the temperature distribution within the billet during deformation and subsequent cooling.

A similar lamellar α-phase intragranular structure is observed in titanium castings under slow cooling conditions [48]. In our experiments, cooling was relatively slow: after removal from the die preheated to 600 °C, further cooling proceeded under a layer of potassium chloride. Consequently, the formation of the lamellar structure can be attributed to β → α recrystallization. In this process, α-phase lamellae nucleate at β-grain boundaries and grow inward. The formation of basket-weave regions is apparently associated with cooling of the billet surface layers in contact with the die walls during hot forging. Deformation of these zones begins in the β-region and ends in the α + β region, which provides favorable conditions for basket-weave formation [50].

The W_{max} values obtained in the investigated pre-deformation heating range were 150–200 MJ/m^3, whereas the heat of fusion of titanium in comparable units is significantly higher – 1411 MJ/m^3 [51]. This indicates that in powder titanium processing by hot die forging, as in other metals, the energy expenditure is considerably lower than during melting, since only part of the particle material undergoes plastic deformation, determined by the material's ductility [43]. The measured W_{max} values are also much lower than the reported activation energy of powder titanium sintering (~15,000 MJ/m^3) [52], which approximately corresponds to the activation energy of self-diffusion in β-Ti.

Fig. 5 presents the dependence of fracture toughness and mechanical properties of powder metallurgy titanium on the hot die forging temperature. Samples were fabricated according to technological schemes *2* and *3*. The $K_{1c} = f(t_{HDF})$ dependences are nonmonotonic. Increasing t_{HDF} to 1150 °C leads to higher K_{1c} due to improved deformability of the porous billet material. A further increase in t_{HDF} to 1200 °C results in a decrease in K_{1c}, which is attributed to grain refinement (see Fig. 4, *c*).

Grain refinement in samples fabricated at $t_{HDF} > 1150$ °C, is associated with the higher post-deformation cooling rate caused by the increasing temperature gradient between the heated porous billet and the die. Throughout the studied t_{HDF} range, the fracture toughness of samples fabricated according to scheme *3* was consistently higher than that of control samples fabricated according to scheme *2* (cf. curves *1* and *2* in Fig. 5, *a*). This difference can be explained by distinct fracture mechanisms in the compared types of samples.

Raising the hot die forging temperature also altered the fracture characteristics of the samples. In control samples fabricated at t_{HDF} = 1000–1100 °C, the fracture surface of the initial fatigue crack contained secondary cracks in the failure origin (Fig. 6, *a*). In the final fracture zone, which formed under static loading, the fracture was of a ductile fine-dimple type with areas of interparticle fracture (Fig. 6, *b*, *c*). Dimple sizes ranged from 4 to 6 μm. At $t_{HDF} > 1100$ °C, secondary cracks in the fracture origin were not observed; however, interparticle fracture areas remained present in the final fracture zone. The dimples of ductile fracture in this zone were larger – 10–12 μm – than in control samples fabricated at 1000–1100 °C, indicating higher material ductility (Fig. 6, *d*).

The fracture surfaces of samples fabricated according to scheme *3* were characterized by the absence of secondary cracks in the fracture origin at all studied t_{HDF} values (Fig. 6, *e*). In the final fracture zone, interparticle fracture was not detected, which indicates higher fracture energy compared to control samples.

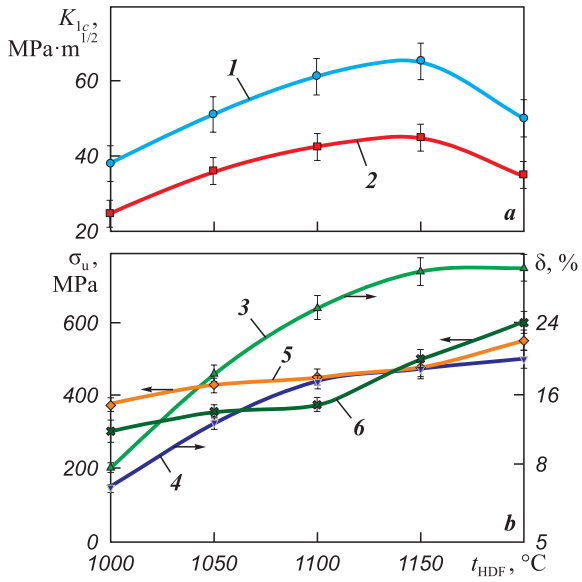

Fig. 5. Effect of pre-deformation heating temperature of porous titanium billets on fracture toughness (***a***) and mechanical properties (***b***)

1 – K_{1c} (scheme *3*), ***2*** – K_{1c} (scheme *2*); ***3*** – δ (scheme *3*); ***4*** – δ (scheme *2*); ***5*** – σ_u (scheme *3*); ***6*** – σ_u (scheme *2*); W = 195 MJ/m^3

Рис. 5. Влияние температуры преддеформационного нагрева пористых заготовок из титана на трещиностойкость (***a***) и механические свойства (***b***)

1 – K_{1c} (схема *3*), ***2*** – K_{1c} (схема *2*); ***3*** – δ (схема *3*), ***4*** – δ (схема *2*), ***5*** – σ_u (схема *3*), ***6*** – σ_u (схема *2*); W = 195 МДж/м^3

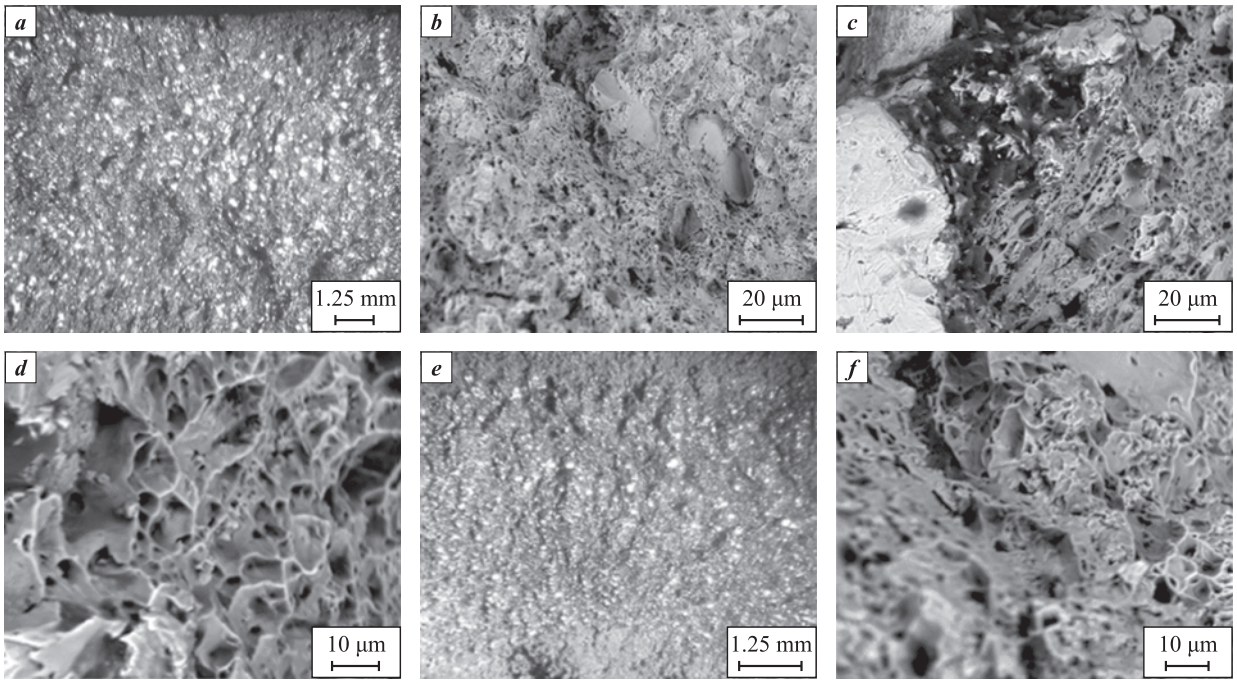

Fig. 6. Fracture surfaces of powder titanium samples fabricated by schemes *2* (***a–d***) and *3* (***e***, ***f***) after fracture toughness tests

a, ***e*** – fracture panorama; ***b***, ***c***, ***d***, ***f*** – final static fracture zone:
b – area of ductile fine-dimple fracture, ***c*** – area of interparticle fracture,
d – large dimples of ductile fracture with interparticle fracture areas, ***f*** – ductile fracture
t_{HDF}, °C: 1000 (***a***, ***b***, ***c***); 1100 (***e***, ***f***); 1200 (***d***)

Рис. 6. Поверхности разрушения образцов порошкового титана, полученных по схемам *2* (***a–d***) и *3* (***e***, ***f***), после испытаний на трещиностойкость

a, ***e*** – панорама излома; ***b***, ***c***, ***d***, ***f*** – зона статического долома:
b – участок вязкого мелкоямочного разрушения, ***c*** – участок межчастичного разрушения,
d – крупные ямки вязкого излома с участками межчастичного разрушения, ***f*** – вязкий излом
t_{HDF}, °C: 1000 (***a***, ***b***, ***c***); 1100 (***e***, ***f***); 1200 (***d***)

The trend in dimple size variation with t_{HDF} was similar to that observed for control samples (see Fig. 6, *f*). Thus, the predominant factor influencing the fracture toughness characteristics of scheme *3* samples is the hydrogenation–dehydrogenation treatment. An analysis of impurity composition in the initial titanium powder and in the fabricated samples showed that under scheme *2*, the oxygen concentration in the resulting material increased by about two fold (see Table).

In samples obtained according to scheme *3*, the oxygen content was significantly reduced, while no substantial changes were observed in the amount of other impurities. This is explained by the fact that hydrogen released from titanium hydrides during vacuum sintering of porous billets after hydrogenation promotes the reduction and activation of oxides localized on the surfaces of open pores. A similar sintering activation effect when using hydrogenated titanium powder was reported earlier [27]. In our experiments, the activation of pore surfaces and their refinement from oxides improved the conditions for interparticle bonding during subsequent hot die forging [53]. In contrast, the relatively high oxygen content in control samples (scheme *2*), associated with the presence of oxides on the collapsing pore surfaces during hot die forging, impaired interparticle contact conditions and

Impurity content in titanium powder and materials produced from it

Содержание примесей в порошке титана и материалах на его основе

Material/ processing scheme	Content, wt. %						
	Fe	Cl_2	N_2	O_2	C	Si	H_2
PTES-1 powder	0.06	0.03	0.020	0.06	0.02	0.02	0.010
Scheme *2*	0.06	0.02	0.011	0.15	0.03	0.02	0.003
Scheme *3*	0.06	0.01	0.008	0.03	0.02	0.02	0.002

led to lower fracture toughness compared to scheme *3* samples (Fig. 5, *a*).

Strength and ductility characteristics were less sensitive to the quality of interparticle bonding and were mainly determined by grain size. Accordingly, the dependences of σ_u and δ on t_{HDF} were monotonic: increasing temperature led to growth of these properties throughout the studied range (see Fig. 5, *b*). In this case, the dominant factor was grain refinement with increasing t_{HDF} (see Fig. 4, *c*). Improved interparticle bonding conditions during hot die forging of billets subjected to hydrogenation–dehydrogenation resulted in higher ductility of scheme *3* samples compared to control samples (cf. curves *3* and *4* in Fig. 5, *b*).

Conclusions

1. The maximum specific densification work required to achieve monolithic density in porous powder titanium was determined for different pre-deformation heating temperatures of billets. The nonmonotonic character of the W_{max} dependence on t_{HDF} is associated with the formation of a coarse-grained structure and reduced ductility of the deforming material in the $\alpha \rightarrow \beta$ phase transformation temperature range.

2. A processing technology was proposed for producing hot die forged powder titanium, involving hydrogenation–dehydrogenation of porous billets. This treatment promotes hydrogen-assisted reduction by hydrogen of oxides localized on the surfaces of open pores and their activation, thereby improving interparticle bonding during subsequent hot die forging and enhancing the fracture toughness and ductility of the resulting samples compared to control samples.

3. Increasing the hot die forging temperature of porous titanium billets contributes to higher fracture energy and improved ductility of the samples. At the same time, the likelihood of secondary crack formation in the origin of the initial fatigue crack is reduced, and the size of dimples in the final ductile fracture zone increases.

References / Список литературы

1. Fang Z.Z., Paramore J.D., Sun P., Chandrana K.S.R., Zhang Y., Xia Y., Cao F., Koopman M.M., Free M.M. Powder metallurgy of titanium – past, present, and future. *International Materials Reviews*. 2018;63(7):407–459. https://doi.org/10.1080/09506608.2017.1366003
2. Froes F.H., Eylon D. Powder metallurgy of titanium alloys. *International Materials Reviews*. 1990;35(1):162–184. https://doi.org/10.1179/095066090790323984
3. Fang Z.Z., Sun P. Pathways to optimize performance/cost ratio of powder metallurgy titanium – a perspective. *Key Engineering Materials*. 2012;520:15–23. https://doi.org/10.4028/www.scientific.net/KEM.520.15
4. Seong S., Younossi O., Goldsmith B.W., Lang Th., Neumann M. Titanium – industrial base, price trends, and technology initiatives. Santa Monica (CA): RAND Corporation; 2009. 128 p. https://doi.org/10.7249/MG789
5. Froes F.H.S., Gungor M.N., Ashraf Imam M. Cost-affordable titanium: The component fabrication perspective. *Journal of the Minerals, Metals and Materials Society*. 2007;59(6):28–31. https://doi.org/10.1007/s11837-007-0074-8
6. Rubtsov A.N., Olesov Yu.G., Antonova M.M. Hydrogenation of titanium materials. Kyiv: Naukova Dumka, 1971. 128 p. (In Russ.).

   Рубцов А.Н., Олесов Ю.Г., Антонова М.М. Гидрирование титановых материалов. Киев: Наукова думка, 1971. 128 с.
7. Zhu L., Kong L., Yang B., Xu B. Production of low-oxygen titanium powder by thermochemical and electrochemical processes: Current state and perspectives. *Journal of Materials Research and Technology*. 2025;36:1522–1535. https://doi.org/10.1016/j.jmrt.2025.03.226
8. Barbis D., Gasior R.M., Walker G.P., Capone J.A., Schaeffer T.S. Titanium powders from the hydride-dehydride process. In: *Titanium powder metallurgy: science, technology and applications*. Oxford: Butterworth-Heinemann, 2015. P. 101–116. https://doi.org/10.1016/B978-0-12-800054-0.00007-1
9. Hidalgo A.A., Frykholm R., Ebel Th., Pyczak F. Powder metallurgy strategies to improve properties and processing of titanium alloys: A review. *Advanced Engineering Materials*. 2017;19(6):1600743. https://doi.org/10.1002/adem.201600743
10. Wang H., Fang Z.Z., Sun P. A critical review of mechanical properties of powder metallurgy titanium. *International Journal of Powder Metallurgy*. 2010;46(5):45–57.
11. Antsiferov V.N., Ustinov V.S., Olesov Yu.G. Sintered titanium-based alloys. Moscow: Metallurgiya, 1984. 168 p. (In Russ.).

    Анциферов В.Н., Устинов В.С., Олесов Ю.Г. Спеченные сплавы на основе титана. М.: Металлургия, 1984. 168 с.
12. Majima K., Hirata T., Shouji K. Effects of purity of titanium powder and porosity on static tensile properties of sintered titanium specimens. *Journal of The Japan Institute of Metals*. 1987;51(12):194–1200. https://doi.org/10.2320/JINSTMET1952.51.12_1194
13. Liu L.H., Yang C., Kang L.M., Qu S.G., Li X.Q., Zhang W. W., Chen W.P., Li Y.Y., Li P.J., Zhang L.C. A new insight into high-strength $Ti_{62}Nb_{12.2}Fe_{13.6}Co_{6.4}Al_{5.8}$ alloys with bimodal microstructure fabricated by semi-solid sintering. *Scientific Reports*. 2016;6(1):23467. https://doi.org/10.1038/srep23467
14. Panigrahi B.B., Godkhindi M.M. Sintering of titanium: Effect of particle size. *International Journal of Powder Metallurgy*. 2006;42(2):35–42.
15. Robertson I., Schaffer G. Some effects of particle size on the sintering of titanium and a master sintering curve model. *Metallurgical and Materials Transactions A*.

2009;40(8):1968–1979.
https://doi.org/10.1007/s11661-009-9894-1

16. Qian M., Yang Y.F., Luo S.D., Tang H.P. Pressureless sintering of titanium and titanium alloys: sintering densification and solute homogenization. In: *Titanium powder metallurgy: science, technology and applications*. Oxford: Butterworth-Heinemann, 2015. P. 201–218. https://doi.org/10.1016/B978-0-12-800054-0.00012-5
17. Pereloma E.V., Savvakin D.G., Carman A., Gazder A., Ivasishin O.M. Microstructure development and alloying elements diffusion during sintering of near-β titanium alloys. In: *Powder Metallurgy of Titanium*. Brisbane, Australia: Trans Tech Publications Ltd, 2012. P. 49–56. https://doi.org/10.4028/b-HGgP7a
18. Panigrahi B.B. Sintering behaviour of Ti–2Ni and Ti–5Ni elemental powders. *Materials Letters*. 2007;61(1):152–155. https://doi.org/10.1016/j.matlet.2006.04.025
19. Xia Y., Schaffer G. B., Qian M. The effect of a small addition of nickel on the sintering, sintered microstructure, and mechanical properties of Ti–45Al–5Nb–0.2C–0.2B alloy. *Journal of Alloys and Compounds*. 2013;578:195–201. https://doi.org/10.1016/j.jallcom.2013.05.104
20. Liu H.-W., Bishop D. P., Plucknett K. P. A comparison of Ti–Ni and Ti–Sn binary alloys processed using powder metallurgy. *Materials Science and Engineering A*. 2015;644:392–404. https://doi.org/10.1016/j.msea.2015.07.085
21. Robertson I.M., Schaffer G.B. Design of titanium alloy for efficient sintering to low porosity. *Powder Metallurgy*. 2009;52(4): 311–315. https://doi.org/10.1179/003258909X12502872942499
22. Liu Y., Chen L.F., Tang H.P., Liu C.T., Liu B., Huang B.Y. Design of powder metallurgy titanium alloys and composites, *Materials Science and Engineering: A*. 2006;418:25–35. https://doi.org/10.1016/j.msea.2005.10.057
23. Esteban P.G., Bolzoni L., Ruiz-Navas E.M., Gordo E. PM processing and characterisation of Ti–7Fe low cost titanium alloys. *Powder Metallurgy*. 2011;54(3):242–252. https://doi.org/10.1179/174329009X457063
24. Yang Y.F., Luo S.D., Bettles C.J., Schaffer G.B., Qian M. The effect of Si additions on the sintering and sintered microstructure and mechanical properties of Ti–3Ni alloy. *Materials Science and Engineering: A*. 2011;528(24):7381–7387. https://doi.org/10.1016/j.msea.2011.06.029
25. Xia Y., Yu P., Schaffer G.B., Qian M. Cobalt-doped Ti–48Al–2Cr–2Nb alloy fabricated by cold compaction and pressureless sintering. *Materials Science and Engineering: A*. 2013;574:176–185. https://doi.org/10.1016/j.msea.2013.03.023
26. Xia Y., Luo S.D., Wu X., Schaffer G.B., Qian M. The sintering densification, microstructure and mechanical properties of gamma Ti–48Al–2Cr–2Nb alloy with a small addition of copper. *Materials Science and Engineering: A*. 2013;559:293–300. https://doi.org/10.1016/j.msea.2012.08.100
27. Savvakin D.H., Humenyak M.M., Matviichuk M.V. Molyar O.H. Role of hydrogen in the process of sintering of titanium powders. *Materials Science*. 2012;47(5):651–661. https://doi.org/10.1007/s11003-012-9440-y
28. Ivasishin O.M., Savvakin D.G., Gumenyak N.M. Dehydrogenation of powder titanium hydride and its role in the activation of sintering. *Metallofizika i Noveishie Tekhnologii*. 2011;33:899–917. (In Russ.).

    Ивасишин О.М., Саввакин Д.Г., Гуменяк Н.М. Дегидрирование порошкового гидрида титана и его роль в активации спекания. *Металлофизика и новейшие технологии*. 2011;33(7):899–917.
29. Raynova S., Yang F., Bolzoni L. Mechanical behaviour of induction sintered blended elemental powder metallurgy Ti alloys. *Materials Science and Engineering: A*. 2020;799:140–157. https://doi.org/10.1016/j.msea.2020.140157
30. Weston N., Jackson M. FAST-forge – A new cost-effective hybrid processing route for consolidating titanium powder into near net shape forged components. *Journal of Materials Processing Technology*. 2017;243:335–346. https://doi.org/10.1016/j.jmatprotec.2016.12.013
31. Careau S.G., Ulate-Kolitsky E., Tougas B. Direct powder forging – A new approach for near net shape processing of titanium. *Powders*. 2023;2(1):21–31. https://doi.org/10.3390/powders2010002
32. Singh A.P., Gabbitas B., Zhang D. Fracture toughness of powder metallurgy and ingot titanium alloys – A review. *Key Engineering Materials*. 2013;551:143–160. https://doi.org/10.4028/www.scientific.net/KEM.551.143
33. Romero C., Yang F., Bolzoni L. Fatigue and fracture properties of Ti alloys from powder-based processes – A review. *International Journal of Fatigue*. 2018;117:407–419. https://doi.org/10.1016/j.ijfatigue.2018.08.029
34. Weston R. Anatomy of an AM part failure: Lessons for managers, designers and producers from 2021's Olympic bike crash. *Additive Manufacturing*. 2022;8(2):115–125. URL: https://www.metal-am.com/articles/anatomy-of-an-am-part-failure-lessons-for-managers-designers-and-producers-from-2021s-olympic-bike-crash/
35. Baker J. An investigation into the handlebar failure that occurred in the Australian men's team pursuit race at the Tokyo 2020 Olympics. *Report*. 2022. 171 p. URL: https://assets.auscycling.org.au/s3fs-public/2022-02/handlebar-9_21-ev-3_5-combined-inc-annexes-appendicies.pdf?utm_source=Metal+AM&utm_medium=magazine (Accessed: 30.04.2025).
36. Goncharov I.N. Method for processing titanium sponge and device for implementing the method: Author's certificate 132409 (USSR). 1960.

    Гончаров И.Н. Способ переработки титановой губки и устройство для осуществления способа: Авт. св-во 132409 (СССР). 1960.
37. Olsson L.R., Lampe V., Fischmeister H. Direct forging of high-alloy steel powders to bar stock. *Powder Metallurgy*. 1974;17(34):347–362. https://doi.org/10.1179/pom.17.34.1974.007
38. Careau S.G., Bernard Tougas B., Ulate-Kolitsky E. Effect of direct powder forging process on the mechanical properties and microstructural of Ti–6Al–4V ELI. *Materials*. 2021;14(16):4499. https://doi.org/10.3390/ma14164499
39. ASTM F136-21. Standard specification for wrought titanium-6aluminium-4vanadium ELI (Extra Low Interstitial)

alloy for surgical implant applications (UNS R56401). West Conshohocken, PA: ASTM International, 2018. 5 p.

40. Pavlov V.A., Kiparisov S.S., Shcherbina V.V. Pressure treatment of non-ferrous metal powders. Moscow: Metallurgiya, 1977. 170 p. (In Russ.).

Павлов В.А., Кипарисов С.С., Щербина В.В. Обработка давлением порошков цветных металлов. М.: Металлургия, 1977. 170 с.

41. Pavlov V.A. Pressure processing of non-ferrous metal powders. Zaporozhye: Motor Sich OJSC, 2010. 347 p. (In Russ.).

Павлов В.А. Обработка давлением порошков цветных металлов. Запорожье: ОАО «Мотор Сич», 2010. 347 с.

42. Dorofeyev Yu.G. Dynamic hot pressing of porous materials. Moscow: Nauka, 1968. 120 p. (In Russ.).

Дорофеев Ю.Г. Динамическое горячее прессование пористых материалов. М.: Наука, 1968. 120 с.

43. Dorofeyev Yu.G. Dynamic hot pressing in metal ceramics. Moscow: Metallurgiya, 1972. 176 p. (In Russ.).

Дорофеев Ю.Г. Динамическое горячее прессование в металлокерамике. М.: Металлургия, 1972. 176 с.

44. Roman O.V., Shuganov A.D., Smirnov G.V. High-energy technologies based on the use of explosives. In: *50 years of powder metallurgy in Belarus. History, achievements, prospects*. Minsk: PM SSPA, 2010. P. 583–601. (In Russ.).

Роман О.В., Шуганов А.Д., Смирнов Г.В. Высокоэнергетические технологии, основанные на применении взрывчатых веществ. В сб.: *50 лет порошковой металлургии Беларуси. История, достижения, перспективы*. Минск: ГНПО порошковой металлургии, 2010. С. 583–601.

45. Brown W.F.Jr., Srawley J.E. Plane strain crack toughness testing of high strength metallic materials. In: *Plane Strain Crack Toughness Testing of High Strength Metallic Materials*. Ed. Committee E-24. West Conshohocken, PA: ASTM International, 1966. 129 p. https://doi.org/https://doi.org/10.1520/STP44663S

46. Murphy Th.F., Lindsley B. Metallographic analysis of PM fracture surfaces. 15 p. URL: https://studylib.net/doc/10702066/metallographic-analysis-of-pm-fracture-surfaces--hoeganae... (Accessed: 30.04.2025).

47. Mazharova G.E., Komanovskii A.Z., Chechulin B.B., Vazhenin S.F. Pressure treatment of titanium alloys. Moscow: Metallurgiya, 1977. 96 p. (In Russ.).

Мажарова Г.Е., Комановский А.З., Чечулин Б.Б., Важенин С.Ф. Обработка титановых сплавов давлением. М.: Металлургия, 1977. 96 с.

48. Illarionov A.G., Popov A.A. Technological and operational properties of titanium alloys. Yekaterinburg: Publishing house of the Ural University, 2014. 137 p. (In Russ.).

Илларионов А.Г., Попов А.А. Технологические и эксплуатационные свойства титановых сплавов. Екатеринбург: Изд-во Уральского ун-та, 2014. 137 с.

49. Salishchev G.A., Kajbyshev O.A., Gordienko E.G., Chernyakov M.I. Influence of initial structure on superplasticity effect of VTZ-1 titanium alloy. *Izvestiya. Non-Ferrous Metallurgy*. 1976;4:104–107. (In Russ.).

Салищев Г.А., Кайбышев О.А., Гордиенко Е.Г., Черняков М.И. Влияние исходной структуры на эффект сверхпластичности титанового сплава ВТ3-1. *Известия вузов. Цветная металлургия*. 1976;4:104–107.

50. Aleksandrov V.K., Anoshkin N.F., Belozerov A.P., Bovchar G.A., Brun M.Ya., Ermanok M.Z. Semi-finished products from titanium alloys. Moscow: ONTI VILS, 1996. 581 p. (In Russ.).

Александров В.К., Аношкин Н.Ф., Белозеров А.П., Бовчар Г.А., Брун М.Я., Ерманок М.З. Полуфабрикаты из титановых сплавов. М.: ОНТИ ВИЛС, 1996. 581 с.

51. Berdonosov S.S. Titan. In: *Physical Encyclopedia*. (Ed. A.M. Prokhorov). Moscow: Bol'shaya rossiiskaya entsiklopediya, 1994. Vol. 4. 704 p.

Бердоносов С.С. Титан. В кн.: *Физическая энциклопедия*. В 5 т. (гл. ред. А.М. Прохоров). М.: Большая российская энциклопедия, 1994. Т. 4. 704 с.

52. Robertson I.M., Schaffer G.B. Some Effects of particle size on the sintering of titanium and a master sintering curve model. *Metallurgical and Materials Transactions A*. 2009;40A: 1968–1979. https://doi.org/10.1007/s11661-009-9894-1

53. Dorofeev Yu.G., Popov S.N. Welding together of metals during dynamic hot pressing. *Soviet Powder Metallurgy and Metal Ceramics*. 1971;10(2):118–124. https://doi.org/10.1007/BF00796409

Дорофеев Ю.Г., Попов С.Н. Исследование сращивания металлов при динамическом горячем прессовании. *Порошковая металлургия*. 1971;98(2):44–51.

Information about the Authors

Vladimir Yu. Dorofeyev – Dr. Sci. (Eng.), Professor of the Department of material science and engineering technology, Platov South-Russian State Polytechnic University (NPI), (PSRSPU (NPI))
ORCID: 0000-0001-6743-1727
E-mail: dvyu56.56@mail.ru

Anna N. Sviridova – Cand. Sci. (Eng.), Associate Prof. of the Department of Automobiles and Transport-Technological Complexes, PSRSPU (NPI)
ORCID: 0000-0002-9851-1073
E-mail: anysviridova@yandex.ru

Сведения об авторах

Владимир Юрьевич Дорофеев – д.т.н., профессор кафедры «Материаловедение и технология машиностроения», Южно-Российский государственный политехнический университет (НПИ) имени М.И. Платова (ЮРГПУ (НПИ))
ORCID: 0000-0001-6743-1727
E-mail: dvyu56.56@mail.ru

Анна Николаевна Свиридова – к.т.н., доцент кафедры «Автомобили и транспортно-технологические комплексы», ЮРГПУ (НПИ)
ORCID: 0000-0002-9851-1073
E-mail: anysviridova@yandex.ru

Svetlana V. Sviridova – Resident of the Institute of Medicine and Health Preservation, Tambov State University named after G.R. Derzhavin
ORCID: 0009-0007-5260-3726
E-mail: dr.sviridova27@inbox.ru

Lev I. Svistun – Dr. Sci. (Eng.), Professor of the Department of Engineering of Control Systems, Materials and Technologies in Mechanical Engineering, Kuban State Technological University
ORCID: 0009-0001-8552-2582
E-mail: levsvistun45@gmail.com

Светлана Владимировна Свиридова – ординатор института медицины и здоровьесбережения, Тамбовский государственный университет имени Г.Р. Державина
ORCID: 0009-0007-5260-3726
E-mail: dr.sviridova27@inbox.ru

Лев Иванович Свистун – д.т.н., профессор кафедры инженерии систем управления, материалов и технологий в машиностроении, Кубанский государственный технологический университет
ORCID: 0009-0001-8552-2582
E-mail: levsvistun45@gmail.com

Contribution of the Authors

V. Yu. Dorofeyev – scientific supervision, formulation of research objectives and tasks, manuscript preparation, and drafting of conclusions.
A. N. Sviridova – mechanical testing and analysis of research results.

S. V. Sviridova – structural and compositional analysis of experimental samples.
L. I. Svistun – preparation and execution of the experiment, supervision of experimental work.

Вклад авторов

В. Ю. Дорофеев – научное руководство, постановка цели и задачи исследования, подготовка текста, формулировка выводов.

А. Н. Свиридова – проведение механических испытаний, анализ результатов исследований.
С. В. Свиридова – проведение исследований структуры и состава опытных образцов.
Л. И. Свистун – подготовка и проведение эксперимента, руководство проведением эксперимента.

Received 11.05.2025
Revised 19.05.2025
Accepted 30.05.2025

Статья поступила 11.05.2025 г.
Доработана 19.05.2025 г.
Принята к публикации 30.05.2025 г.