

SYNTERED TITANIUM ALLOYS FOR NUCLEAR INDUSTRY

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The possibility of using sintered titanium alloys as structural materials in nuclear power engineering is considered; brands of titanium powders, which are manufactured industrially in Ukraine, suitable for obtaining products for nuclear power. The principal possibility of obtaining semi-finished titanium alloys by the method of powder metallurgy is shown, with the necessary concentration of elements. The technology of manufacturing semi-finished products and finished products from sintered titanium alloys is shown. The high quality of semi-finished titanium alloys obtained using powder metallurgy and welding technologies was confirmed. It is shown that the application of the developed resource-saving integrated technology will allow reducing the cost of final products without reducing mechanical characteristics, as well as Ukraine's dependence on external suppliers of semi-finished products and finished products from titanium and its alloys.

INTRODUCTION

In Ukraine 55% of electric power are produced by 4 nuclear power plants [1] with 15 hydrogen-water power reactors (HWPR) Ukrainian nuclear power strategy envisages construction and work commencement of 11 new reactors before 2030 [2]. Titanium and its alloys are used as high quality nuclear power structural materials. They are employed for manufacture of condensers, steam turbine blades [3, 4], heat exchangers [5–8], strength fasteners, flanges for connection and connectors used in the reactor systems [9]. Advantages of titanium equipment are high specific strength, corrosion resistance in different media, stability of mechanical properties, manufacturability, nuclear properties, and weld ability using various methods. Guaranteed work time for the constructions is up to 60 years [10, 11]. Total titanium consumption for construction of one HWPR unit is at least 1350 tons of titanium semi-products and welding materials [12].

Currently the main part of Ukrainian titanium consumption is covered by VSMPO-AVISMA corporation (Russia). As result Russian Federation introduces constant Ukrainian dependence on imported semi- and final titanium products. Consequently local manufacturers are financially suppressed by the unequal competition. Meanwhile Ukraine has the largest European titanium resources and the only titanium sponge manufacturing plant with production capacity up to 20000 tpy. The latter number is more than enough for covering of domestic titanium demand.

Further successful applications of titanium in nuclear machine building depend on quality of the ingots and production cost reduction [13]. Resource-saving methods especially related to high-tech industries are a modern tendency of the world economic development. This also applies to nuclear machine building.

Consequently development and introduction of effective technological solutions in to industrial production is the major route for metal and power saving at each technological step.

Thus conducting of theoretical and experimental research in the above area will yield the solution of a

serious applied to nuclear industry problem: forecasting and providing required quality for the nuclear reactor parts.

Significant production cost decrease for the articles applied in nuclear industry is feasible using manufacture of work pieces by powder metallurgy (PM) methods.

Such method shave currently been widely employed for ferrite and ferrite-marten site steels. This includes compacting of fast hardened powders with spherical or scale forms [14, 15]. Studies of crushed titanium hydride and its compacting for applications in transport nuclear units are also known [16].

The main disadvantages for direct broad usage of pressed powders are:

- High residual porosity and consequently low level of physical, mechanical, and special properties compared to wrought work pieces
- State of titanium surface layer which affects atomic fatigue failure and cyclic durability [17]
- Problems with manufacture of complicated shape articles and also long semi-products

Titanium surface layer and its properties can be improved by usage of various concentrated heat energy sources [18, 19], thermal diffusion treatment.

Porosity is a related part of all the alloys which are synthesised from powders. It affects their mechanical and exploitation properties. The major method for minimization of a number of pores and their sizes is work piece treatment under high pressure. In this case the pores might be fully eliminated. Here the following methods are used: hot stamping in closed and/or open dies [20], hot isostatic pressure (HIP), intensive plastic deformation (IPD). HIP can only be used for spherical alloy particles. IPD methods require complicated equipment and can only be employed for small size articles.

The question whether usage of forging is efficient or not depends on the powders. The most cost saving materials seem to be manufactured directly from titanium sponge.

Modern welding technologies allow manufacture of the articles with complicated geometrical shape and also produce long semi-products with reduced number of threaded and/or soldered joints.

Summarising the above the objectives of the current work are as follows:

- Development and implementation of cost saving technology for titanium alloy semi- and final products used in nuclear power industry

- These articles must have definite chemical composition and manufactured by PM and welding methods using Ukrainian raw materials

- Applications of a forging method for production of compacted semi-products from titanium powders

- Evaluation of mechanical strength and corrosion resistance for the samples synthesised by offered technology

- Practical usage of the developed production method as the industrial technological process

MATERIALS AND RESEARCH METHODS

Titanium powders are the raw materials for the development of prospective semi-product manufacture technology. The latter might be used for production of responsible nuclear power reactor parts [21]. The powders are the main technological component which defines properties of the final product and their production cost. Thus these powders should fit complex technical and economic requirements. For example, the price for the spherical powders manufactured by molten metal dispersion could be 10 fold larger compared to those produced by grinding of the solids. Consequently, considering cost saving for a novel method of wrought titanium semi-product manufacture by powder metallurgy [Ошибка! Залка не определена.] thermal-mechanical industrial powders have been chosen for the experiments. These powders of PT type are the cheapest ones. They are produced by State Enterprise "Zaporizhzhia metallurgical research & production plant", PJSC "Titanium institute". Around 28 kinds of PT powders are manufactured. Among them there are 13 types with fractional composition $-0.5+0.16$ mm which fits VT1-0 alloy (GOST 19807-91)

In accordance to modern requirements, structural materials for nuclear reactors must be pure. Impurities significantly increase mechanical corrosion, lessen radiation resistance, and also cause restrictions in design and exploitation of the units. Elevated content of impurities influences mechanical properties and, as a result, decreases service characteristics for majority of titanium alloys. Titanium fully loses ability of plastic deformation and gets brittle when hydrogen, nitrogen, and oxygen concentrations become 0.003, 0.02, and 0.7 weight % respectively [22].

Considering the above requirements for the nuclear grade alloys fractional compositions of $-5.00 +0.45$ mm were selected from PT powders. In fact only 3 kinds of the powders can be used straightaway: PT-1-1, PT-2-1, PT-3-1 (Tabl. 1). The rest of the powders will be raw materials for further refining and obtaining of high purity metals [23]. Then these materials can be used for manufacture of final articles.

Table 1
Chemical composition of PT powders (TU 14-10-026-98) and VT1-0 titanium alloy (GOST 19807-91), weight %

Material (fraction, mm)	Ti	Si	Fe	O	H	N	C	Others
	max							
PT-1-1 (-5.0+1.0) PT-2-1 (-1.0+0.63)	base	0.005	0.02	0.06	NA	0.02	0.01	NA
PT-3-1 (-0.63+0.18)	base	0.015	0.03	0.06	NA	0.02	0.01	NA
VT1-0	base	0.100	0.25	0.20	0.010	0.04	0.07	0.3

Type PT-2-1 is similar to PT-1-1 according to its chemical composition, but a grain size for the former is up to 80 fold smaller compared to the latter (see Tabl. 1). This will yield lower porosity for compacted PT-2-1. PT-1-1 type is recommended for synthesis of sintered powders with maximum porosity, and PT-3-1 for minimum one.

Deformations and breaking of VT1-0 titanium have sufficiently been studied [24, 25]. Data of its radiation grow that the imitated reactor conditions are also available [26]. Therefore, with respect to reducing of a number for the experiments this metal was chosen in further work. It has been considered that all metal parameters and proposed technology must guarantee the standard characteristics of wrought VT1-0 titanium.

Experimental sample geometry should fit the following. Firstly, the samples must have minimum raw material consumption. Secondly, the size of the samples must be easily scaled up to the industrial dimensions. Thirdly, equipment for the sample production should fit technological capabilities of the research laboratory. Taking into account the above two types of the samples were investigated. Type 1: parallelepiped with the size $11 \times 11 \times 55$ mm for mechanical tests and metallographic analysis. Type 2: cylinder with diameter 40 mm and height 60 mm for production of the final article semi-products. The latter samples were used for definition of a size factor on titanium alloy properties.

For two types of the samples the following was developed: technological scheme and equipment, stamping methods and shrinkages, design and capacity calculations, geometrical parameters, and kinematic calculation of the matrix. Offered all-purpose equipment allows production of rod and tube work pieces (Fig. 1).

Following experimental schemes were used:

1) Synthesis + welding:

- Production of long size rod work pieces according to the industrial standards (diameter 40...60 mm);
- Obtaining of tube semi-products for shut-off valves and other hollow articles.

2) Synthesis+welding+plastic deformation: manufacture of rod work pieces for further forging (import replacement program work).

120 pc soft he experimental work piece shave been produced. Then they were welded by a solid phase

method and machined to the final article shape.

Machining ability was evaluated for rod semi-products by cutting of lathe thread.

Pipe work pieces were used for production of:

- Casings of disk (GOST 21345-2005) and ball (TUU 29.1-32638366-001:2005) taps
- Collar of a sleeve shaft pump 28/2 (pressed into titanium ring of the pump and welded together); sizes, mm: diameters – external 53, internal 32, height 37. This articles we used for corrosion resistance tests.



Fig 1. Sintered work pieces for rod a) and pipe b)

Taps were tested by research and industrial methods:

- Leakage at 5 MP a pressure;
- In aggressive media: 32% HNO_3 , 10% H_2SO_4 , 20% HCl . Tests were conducted in flowing liquids at room temperature during 170 hours;
- At gas treatment facilities: as sample taking tap of a scrubber tank. This was done in calcium hypochlorite $\text{Ca}(\text{ClO})_2$ medium (used bleach liquid) under practical pressure and temperature during 1008 hours.

After the experiments the taps were dismantled and observed with respect to corrosion damage.

After 500 work hours the pump was removed and dismantled. The parts were investigated for the trails of corrosion.

Economic feasibility for production of the parts was evaluated by standard research methods and two schemes:

1) Sponge → crushing and screening → electrode production including material losses → (VAR melting + machining of ingot) x 3 times → preliminary forging → cutting into pieces → final forging → machining → rod;

2) Sponge → crushing → screening → stamping → sintering → welding → machining → forging → machining → rod → final treatment → article.

Prior to article manufacture by a powder metallurgy method PT-3-1 powders were dried at 300° C during 30...50 min to remove humidity.

Then the materials was pressed and sintered.

After that powders were shaped on DB 2432 Apress under 700 MPa force. This load has been chosen as to be sufficient for the formation of metal structure and obtaining of small size pores. Also a number of the latter will be minimised here. Large force does not affect porosity much. However it significantly decreases auxiliary gear exploitation time.

Drying and sintering was conducted in a laboratory vacuum electric furnace SNVE – 1.3.1/16 at 13.3 Pa. Sintering parameters: heating rate $V_{\text{heat}} = 20$ °C/min, isothermal temperature (1250 ± 10) °C during 180 min.

Sintered work pieces were sorted and calibrated. Sorting of the samples was based on visual observation of possible metal surface defects. Surface defects were as follows: various curvatures, shape distortions, grooves, cracks, crumbling of the surface. Faulty samples were rejected.

Deformation treatment for sintered work pieces defining diminishing structural components and reduction of excessive porosity was conducted after forging in open dies (total plastic deformation 15-20 %). This has been done in accordance with the method for cast titanium: heat treatment for 25 min – from 870...950 to 780...800 °C [27]. After forging sample surfaces were cleaned from surface films and oxides by sand blasting.

Morphology of powder particles was investing at educing stereo graphic microscope Leica PM L82 and raster electron microscope REM-106I. The powders were fixed on the table holder by carbon conductive adhesive tape. This allowed defining chemical composition and measuring of the particle linear sizes under deep vacuum. Particle morphology, evaluation of micro-structural components and porosity of the samples were analised using microphotographs in Imagepro Plus system according to GOST 1778-70, DSTUISO2738:2009 methods.

Tests of the samples at static loads and calculations of tensile strength were done according to GOST 1497-84. The samples for investing at ion of mechanical properties were cut from work pieces using mechanical methods. Structure and graphic analysis of broken samples were conducted using NEOPHOT optical microscope and rast erelectron microscope JEOL {in the secondary electron beam}.

Break tests were done using servo-hydraulic machine “INSTRON” 8802 at room temperature. Exo-tensile meter base was 25 mm. During the tests deformations for the work part of the sample were checked with precision of $\pm 1\mu\text{m}$. Load measuring precision for the work part of the sample was ± 3 MPa. Exo-tensile meter and spring dynamometer data were digitally displayed at 0.01 sec intervals.

Impact strength was measured according to GOST 9454-78 from the experiments conducted using IMP-460J pendulum pile driver (INSTRON Ltd) which has a digital system for information collection. The samples of type one (U-shape stress concentrator) were used. The trials were accomplished for two lots of the samples prepared in the similar conditions.

EXPERIMENTAL RESULTS AND DISCUSSION

Morphology of sintered powder particles shows well developed surface (Fig. 2) similar to titanium sponge. This positively influences further material compacting for sintering. The powder has irregular, angular and fragmentary form.

After compacting and sintering the samples display complex porous surface of irregular shape in majority. This is related to the shape of the used powder. Shape of the pores defines the application area for produced articles: structural materials for nuclear reactors which operate at temperature below 300°C (cold zones) with absence of impact and cyclical stresses.

Mechanical tests show (Tabl. 2) that strength characteristics of the produced samples are similar to cast VT1-0 titanium [28]. All the samples have low plasticity. The tests displayed absence of elongation neck. Analysis of sample breaking images (Fig. 3) shows that destruction of the samples was brittle.

Table 2
Mechanical properties for sintered samples in different states

State of the sample	Tensile strength, σ_B , MPa	Yield strength, $\sigma_{0.2}$, MPa	Relative		Impact strength, KCU, J/cm ²
			Elongation, δ , %	Area reduction ψ , %	
Sintered	340.3	285.5	8.4	19.1	107.8
Sintered + deformation	410.8	289	14.8	27.3	131.0
Cast	350	300	17.0	37.0	120.2
Hot rolled GOST 26492-85	≥ 345	NA	≥ 15.0	≥ 40.0	≥ 70.0

Assuming that porosity of non-compact articles defines their strength it is feasible to conclude that the mechanical parameters above have good correlation with the method of particle consolidation. There were significant changes in strength and plasticity of the cylindrical samples after deformation. It has been observed during stretching of the central part related to prism shape semi-product after stamping. It is a consequence of particle consolidation degree for this area.

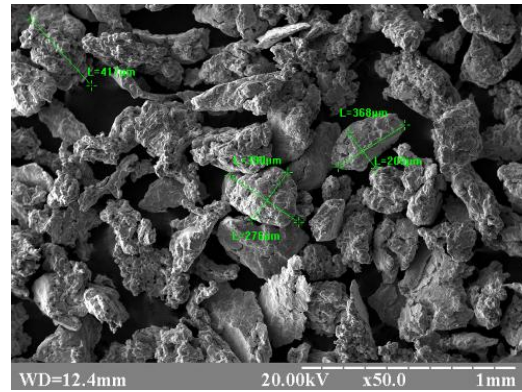
External observation of the deformed semi-product (Fig. 4) did not show any trails of cracks.

Cross-section of stamped work pieces displays consolidation of all sintered powder particles. Also average size and quantity of pores is reduced including full elimination of the latter. Porosity level dropped down to 1%. Level of mechanical properties for the

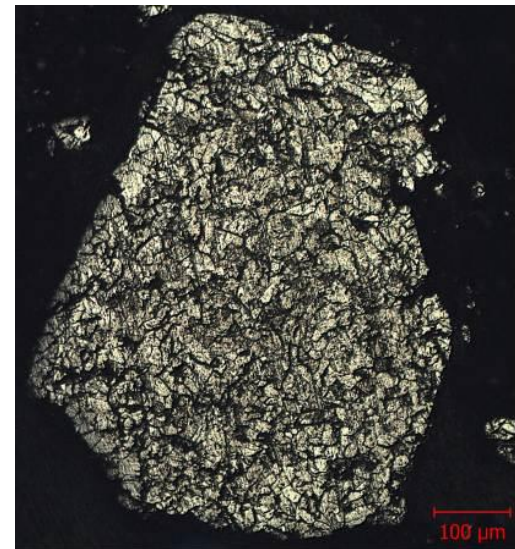
samples cut from the stamped work pieces is similar to conventional VT1-0 rods (GOST 26492-85). Reduced plasticity parameters for the bars are due to the absence of annealing. If annealing will be applied the level of the plastic properties will correspond to GOST 26492-85.



a

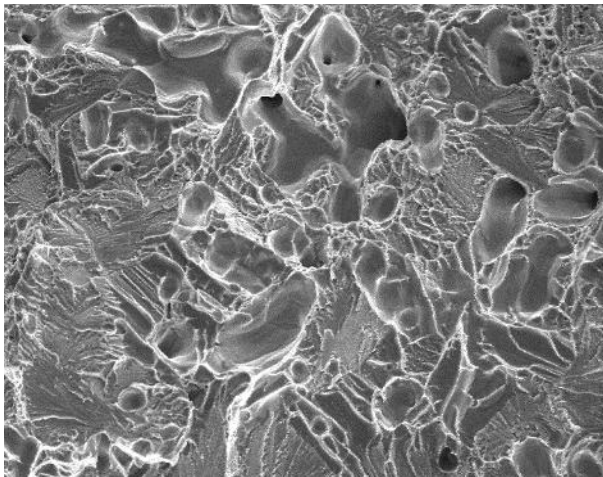


b

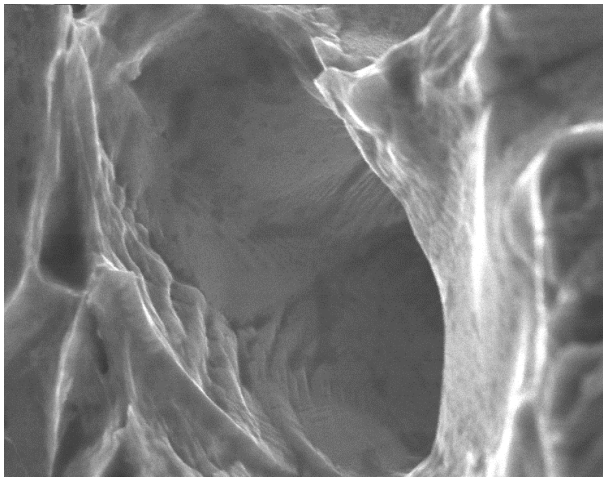


c

Fig. 2. Materials for synthesis of sintered work pieces: titanium sponge (a), general view of titanium powder (b) imicrostructure of a titanium powder particle (c)



x1000



x5000

Fig. 3. Images of broken sintered samples



Fig. 4. Sintered samples after forging

It is feasible to conclude that deformation is an effective method for processing of the articles produced by PM method from CP titanium.

Machining of the experimental rod (Fig. 5) is similar to that for conventional bar semi-products. Quality of threading and formation of turnings were also the same. Work pieces were easily machined using turning and milling (Fig. 6).

Thus feasibility of titanium alloy semi-product manufacture at the industrial scale has been shown. This presents a good base for wider usage of sintered titanium alloys including their welding into long articles. It also increases a number of technical applications for such materials.



Fig. 5. Rod work piece ($\varnothing 40$ mm, length 700 mm) produced using PM and welding methods

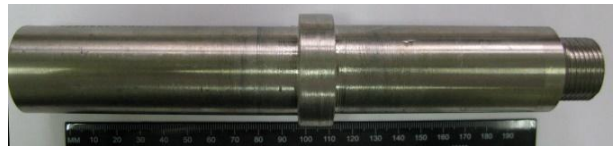
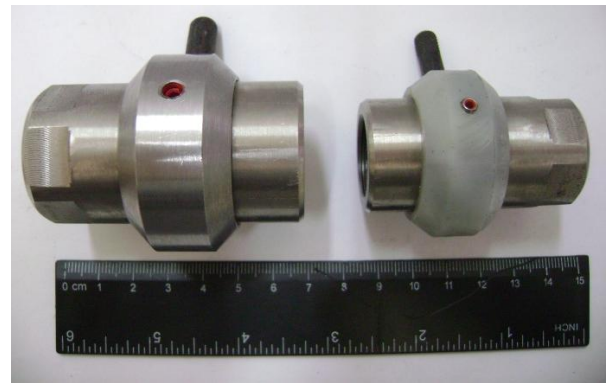


Fig. 6. Rod work piece with $\varnothing 40$ mm, produced by powder metallurgy and welding after machining

During experimental and industrial tests in aggressive media for titanium taps (Fig. 7) manufactured from experimental materials it has been found the following:

- Leakagetests have been fully satisfactory
- Corrosion resistance of the articles was practically the same as for wrought VT1-0 alloy (GOST 19807-91)



a



b

Fig. 7. Casings of taps: disk (a), ball (b)

Production analysis for the conventional articles (see the above) has displayed large raw material consumption. In contrast, PM method reduces it significantly. Production cost saving for the replacement of standard technology by our method reaches 200...400 UAH/tap. This economic effect is due to material and energy savings, reduction in additional manufacturing steps for semi-products, usage of the powders produced directly from titanium sponge, and also by work piece shapes close to the final articles.

As a result, technological and economic effectiveness of sintered titanium alloy article manufacture combined with their welding has been shown for the usage of these materials in nuclear power industry.

CONCLUSIONS

1. Manufacture of work pieces for nuclear power plants using powder metallurgy is a prospective direction for Ukrainian titanium raw material industry and potential customers.

2. Titanium powders manufactured from the sponge of Zaporizhzhia metallurgical experimental plant can be used for synthesis of high quality titanium alloy semi-products.

3. Possibility of obtaining titanium alloy semi-products with definite chemical composition according to GOST 19807-91 using powder metallurgy has been shown.

4. Mechanical properties of sintered experimental VT1-0 alloy were close to those of cast metal. Meanwhile the experimental alloy has up to 7...9 % of excessive porosity and has not been treated by deformation methods.

5. Article production from sintered titanium alloy work pieces is feasible. However porosity could decrease work time for the materials. Thus these articles should be used in the positions where impact and cyclic stresses are absent. Usage of stamped work pieces is suggested for elimination of this shortcoming.

6. Plastic deformation allows production of work pieces with mechanical properties similar to conventional wrought rod samples.

7. Technology for manufacture of sintered VT1-0 titanium (GOST 19807-91) semi-products using welding has been developed. These materials were used for manufacture of casings for disk (GOST 21345-2005) and ball (TUU 29.1-32638366-001: 2005) taps.

8. Results of corrosion resistance tests for VT1-0 metal (GOST 19807-91) produced by PM method with welding of long articles and manufacture of complicated shape semi-products show full viability of this technology. It is planned to make it as an addition to TUU 27.4-00201081-001-2003 "Titanium filtering elements".

9. Rod semi-products synthesised by powder metallurgy and welding have been recommended for import replacement and also for tap casing manufacture.

10. Further research should be directed towards investigation of fractional composition influence on structure and properties of metals. Resistance to irradiation and residual radioactivity for nuclear semi-products should also be studied.

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СПЕЧЕННЫЕ ТИТАНОВЫЕ СПЛАВЫ ДЛЯ ЯДЕРНОЙ ЭНЕРГЕТИКИ

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Рассмотрены возможности использования спеченных титановых сплавов в качестве конструкционных материалов в атомной энергетике; марки порошков титана, которые промышленно выпускаются в Украине и пригодны для получения изделий для атомной энергетике. Показаны принципиальная возможность получения полуфабрикатов титановых сплавов методом порошковой металлургии с обеспечением необходимой концентрации элементов и технология изготовления полуфабрикатов и готовых изделий из спеченных титановых сплавов. Подтверждено высокое качество полуфабрикатов титановых сплавов, полученных с использованием технологий порошковой металлургии и сварки. Показано, что применение разработанной ресурсосберегающей интегрированной технологии позволит снизить стоимость конечных изделий без снижения механических характеристик, а также зависимость Украины от внешних поставщиков полуфабрикатов и готовой продукции из титана и его сплавов.

СПЕЧЕНІ ТИТАНОВІ СПЛАВИ ДЛЯ ЯДЕРНОЇ ЕНЕРГЕТИКИ

О.Є. Капустян, О.В. Овчинников, Т.Б. Янко

Розглянуто можливості використання спечених титанових сплавів як конструкційних матеріалів в атомній енергетиці; марки порошків титану, що промислово випускаються в Україні і придатні для отримання виробів для атомної енергетики. Показано принципова можливість отримання напівфабрикатів титанових сплавів методом порошкової металургії із забезпеченням необхідної концентрації елементів і технологія виготовлення напівфабрикатів і готових виробів із спечених титанових сплавів. Підтверджено високу якість напівфабрикатів титанових сплавів, отриманих з використанням технологій порошкової металургії і зварювання. Показано, що застосування розробленої ресурсозберігаючої інтегрованої технології дозволить знизити вартість кінцевих виробів без зниження механічних характеристик, а також залежність України від зовнішніх постачальників напівфабрикатів і готової продукції з титану і його сплавів.