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Structure and properties of titanium hydride powder obtained from titanium sponge by SHS hydrogenation

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Abstract: The results of the study of the structure and properties of titanium hydride powders obtained from titanium sponge by SHS hydrogenation and mechanical grinding are presented. Hydrogenation was carried out in a reactor at a constant hydrogen pressure of 3 MPa. After passing the combustion wave, the hot titanium sponge was cooled to room temperature in a hydrogen atmosphere. As a result, titanium hydride spongy granules with a hydrogen content of 4.2 wt.% were obtained. Titanium hydride was ground in a ball mill and divided into 4 fractions corresponding to the fractional composition of titanium powder PTK, PTS, PTM and PTOM. Particle size analysis showed that the samples of the PTK and PTOM powders have a narrower particle distribution in comparison with the PTS and PTM ones. Further, obtained powders chemical composition and surface morphology studies were carried out and bulk density, compaction, pycnometric density and specific surface area were determined. According to the chemical analysis results the content of carbon and oxygen impurities decreases during SHS-hydrogenation and the iron content slightly increases during mechanical grinding depending on the grinding time. The study of morphology showed that the hydride titanium particles have an irregular fragmentary shape, such morphology is characteristic of powders obtained by this technology. The surface structure has partially preserved structure of the initial titanium sponge and consists of elongated oriented grains. It is established that with a decrease in the particle size, the bulk density decreases, and the compaction increases. Pycnometric density and specific surface area values are approximately equal for all powder samples.

Keywords: titanium hydride, powder metallurgy, self-propagating high-temperature synthesis (SHS), hydrogenation, morphology, technological properties.

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Структура и свойства порошка гидрида титана, полученного из титановой губки методом СВС-гидрирования

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Аннотация: Представлены результаты исследования структуры и свойств порошков гидрида титана, полученных из титановой губки СВС-гидрированием и механическим измельчением. Гидрирование осуществляли в реакторе при постоянном давлении водорода 3 МПа. После прохождения волны горения горячую титановую губку охлаждали до комнатной температуры в среде водорода. В результате были получены губчатые гранулы гидрида титана с содержанием водорода 4,2 мас.%. Их измельчали в шаровой мельнице и разделяли на 4 фракции, соответствующие фракционному составу порошка титана: ПТК, ПТС, ПТМ и ПТОМ. Анализ размера частиц показал, что образцы порошков ПТК и ПТОМ имеют более узкое распределение частиц в сравнении с ПТС и ПТМ. Далее для полученных порошков были проведены исследования химического состава, морфологии поверхности и определены насыпная плотность, уплотняемость, пикнометрическая плотность и удельная поверхность. Из результатов химического анализа было установлено, что в ходе СВС-гидрирования происходит снижение содержания примеси углерода и кислорода, а при механическом измельчении, в зависимости от его времени, незначительно увеличивается содержание железа. Исследование морфологии показало, что частицы гидрида титана имеют неправильную осколочную форму, — такая морфология характерна для порошков, полученных по данной технологии. Структура поверхности частично сохранила структуру исходной титановой губки и состоит из вытянутых ориентированных зерен. Установлено, что с уменьшением размера частиц насыпная плотность снижается, а уплотняемость возрастает. Значения пикнометрической плотности и удельной поверхности приблизительно равны для всех образцов порошка.

Ключевые слова: гидрид титана, порошковая металлургия, самораспространяющийся высокотемпературный синтез (СВС), гидрирование, морфология, технологические свойства.

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Introduction

Titanium is one of the metals, capable of actively absorbing hydrogen and forming hydrides. Titanium hydride (TiH_2) is known as a titanium with hydrogen chemical compound where hydrogen atoms are randomly distributed in the cavities of the titanium tetrahedral lattice [1, 2]. Titanium hydride has a fairly wide practical application in aerospace, aviation, the chemical industry and nuclear power engineering. It serves for porous titanium and titanium filters production. Blowing agent technique is used for aluminum foam production. Recently, due to the high hydrogen capacity (4.04 wt.%), titanium hydrides have been used as a hydrogen storage material [3— 9].

Hydrogen is also known to cause embrittlement of metals and alloys. The brittleness of titanium hydride is due to the fact that hydrogen reduces the stress required for the movement of dislocations, increases their speed of movement and promotes the formation of microcracks, as well as their growth and the propagation of avalanches. Hydrogenation of titanium also increases the volume of the space cell — approximately in 2.5 times [10].

This characteristic is taken into account in the production of titanium powders using titanium sponge or scrap and by implementing hydrogenation-dehydrogenation method. While hydrogenating, the initial titanium is saturated with hydrogen during the isothermal heat treatment in hydrogen atmosphere. The resulting titanium hydride is quite brittle and can be micro ground within a short period of time implementing mechanical grinding. Afterwards titanium hydride can be dehydrogenated for producing a finely-dispersed titanium powder, which serves to create corrosion-resistant filters, medical implants, and fabricating products by using powder metallurgy methods [11—14]. The powder particles obtained by this method have an irregular splinter shape and the content of impurities depends on the content of impurities in the source raw material. Obtained titanium powder has the same granulometric size composition as the initial titanium hydride. Hydrogenation-dehydrogenation method is of moderate cost and has small impact on the final powder price. Furthermore, finely-dispersed titanium hydride powder can be used for the synthesis of binary and multicomponent alloys and intermetallics [15, 16].

Another way to obtain titanium hydride is with the help of self-propagating high-temperature synthesis (SHS) method. It consists in using the heat of exothermic reaction after the local activation of a combustion one. High temperatures develop in the combustion front, that moves along the source titanium from layer to layer due to thermal transmission. No additional energy inputs are required, the process develops due to the heat of the chemical reaction $Ti + H_2 \rightarrow TiH_2 + Q$ (39 kcal/mol.) [17].

Production of titanium powders by SHS sponge hydrogenation with posterior dehydrogenation is cost-effective. Application of such powders as a primary component allows reducing significantly the cost price of titanium products [18].

Technological properties (bulk density, compressibility) of initial powders are of significant importance for products manufactured by means of the titanium powder metallurgy techniques [19, 20]. According to consumer requirements such powders should possess certain properties and characteristics. Therefore, the study of properties and particle structure of powders used in manufacturing products implementing solid-phase sintering method is a relevant objective for the development of the titanium powder metallurgy techniques. The quality parameters of powders should be stable and not change during the shelf time [21—23].

The object of the given research is to study the structure and determine the technological and chemical properties of titanium hydride powders produced by SHS titanium sponge hydrogenation in the reactor and subsequent mechanical grinding.

Research data and methods

Titanium hydride powders with the density composition corresponding to the granulometric size composition of powders PTK, PTS, PTM, PTOM types (Specifications TU 14-22-57-92), prepared by titanium sponge TG-100 hydrogenation (GOST Russian National Standard 17746-96). The particle size of the initial titanium sponge was from 5 to 20 mm. Hydrogen gas, type «A» grade (GOST (Russian National Standard) 3022-80) was used as the hydrogen source.

Hydrogenation of the sponge was carried out in a sealed reactor of 2 L (Fig. 1, *a*). Titanium sponge 0.5 kg in weight was loaded into a gas-permeable shell installed in the reactor. Further finely-dispersed titanium powder was placed in a paper envelope on top of the titanium sponge to ignite the sponge. The reaction started due to the nickel chrome spiral heating and by means of electricity flow passing through it. Before synthesis, the reactor was sealed and purged with hydrogen to remove

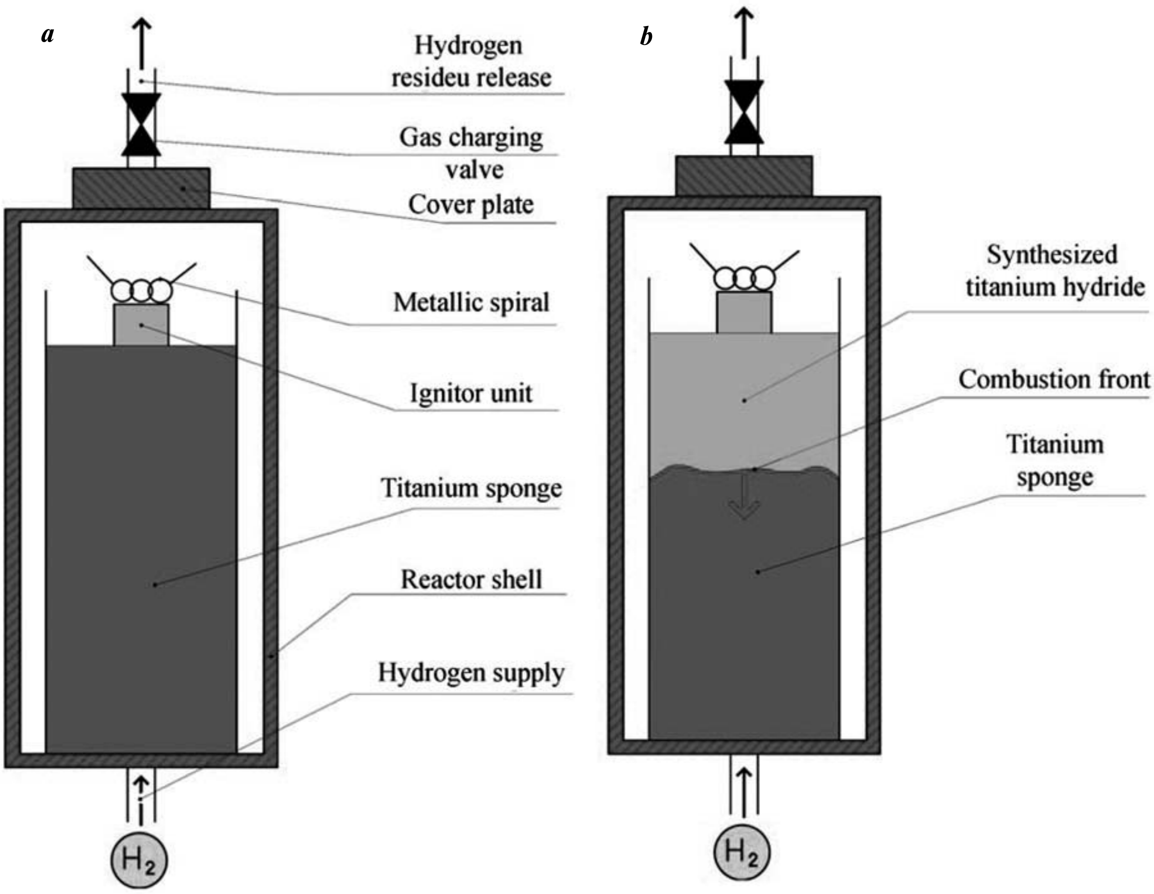

Fig. 1. High-pressure reactor schematic illustration (***a***) and SHS hydrogenation process (***b***)

Рис. 1. Схематическое изображение реактора высокого давления (***a***) и процесса СВС-гидрирования (***b***)

Table 1. **Hydrogenated titanium sponge grinding modes**

Таблица 1. Режимы измельчения гидрированной титановой губки

Grinding mode	Weight of the grinding sponge, kg	Drum rotation speed, rpm	Grinding time, min	Ratio of grind-ing bodies to titanium sponge
1	0.5	90	15	5 : 1
2	0.5	90	20	5 : 1
3	0.5	90	25	5 : 1

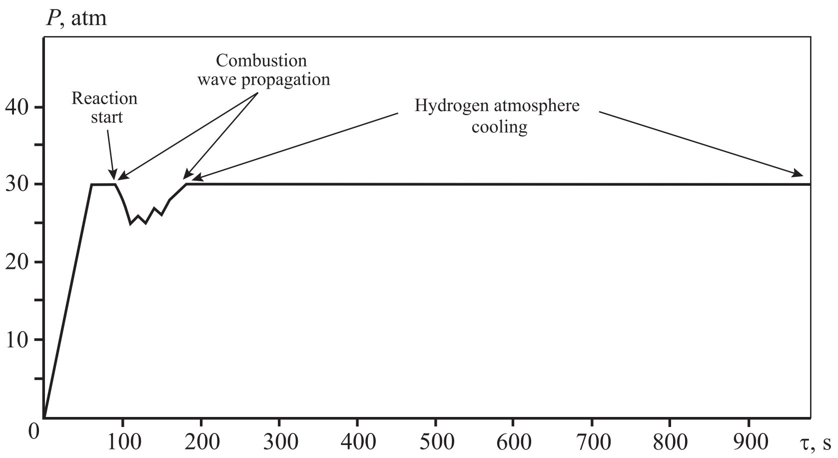

Fig. 2. Hydrogen pressure variation in the reactor during SHS hydrogenation

Рис. 2. Изменение давления водорода в реакторе в процессе СВС-гидрирования

air. Then it was filled with hydrogen until a hydrogen pressure of 30 atm was reached. While combusting, the pressure was maintained by the periodic supply of hydrogen in the direction opposite to the propagation of the combustion front (Fig. 1, *b*). The synthesis time was about 75 s. Then the heated titanium sponge was cooled to room temperature for 1 h in a hydrogen atmosphere (Fig. 2).

Derived titanium hydride sponge was mechanically ground in a steel ball mill with steel grinding bodies according to the modes given in Table 1.

Varying the grinding time resulted in obtaining titanium hydride powders with the particle distribution shown in Fig. 3.

For further study the ground powders were sieved (according to GOST Russian National Standard 18318-94) in order to obtain titanium hydride powders with the density composition corresponding to PTK, PTS, PTM, PTOM types. As a result, powders of 4 fractions were obtained: PTK (<40 μm — 10 wt.%; 40—280 μm — remaining wt.%), PTS (40—100 μm — 35 wt.%; <40 μm — remaining wt.%), PTM (40—100 μm — 25 wt.%; <40 μm — remaining wt.%), PTOM (40—100 μm — 5 wt.%; <40 μm — remaining wt.%).

Following technological characteristics of powders were determined for these fractions: bulk density, compressability, pycnometric density and specific surface area. In addition, the morphology was studied, particle-size distribution by means of laser diffraction was carried out, and the main impurity content was determined for the obtained powders.

Powders bulk density was defined according to GOST Russian National Standard 19440-94, while their compressibility (compaction) — to GOST Russian National Standard 25280-90. Powder pycnometric density was determined according to GOST Russian National Standard 2211-2020: the method in use is based on determining the analytical sample weight and its true volume followed by density calculation. The true volume of the sample was estimated using a pycnometer with saturating liquid (toluene). Specific surface area was measured using the method of low-temperature nitrogen adsorption on Sorbi-M device designed for determining the specific surface area of porous materials.

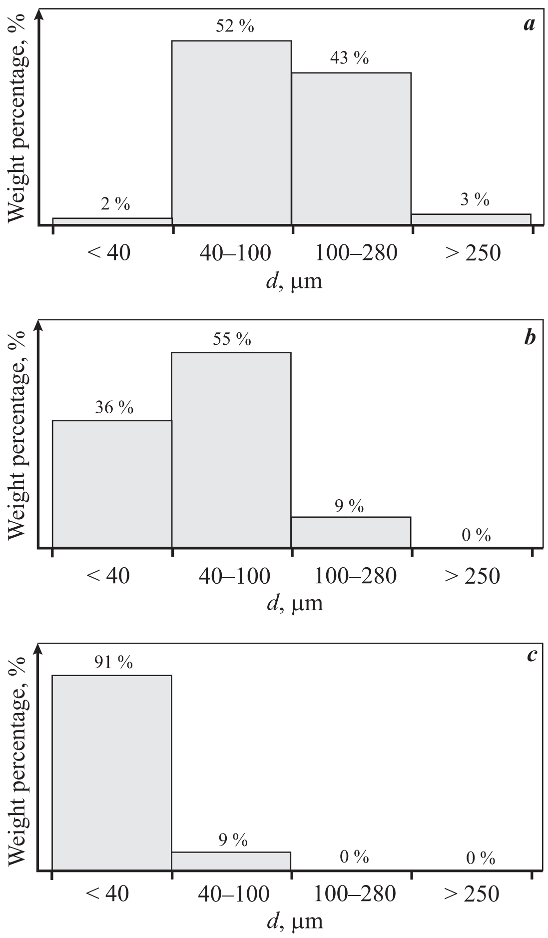

Fig. 3. Particles titanium hydride distribution at grinding modes *1* (*a*), *2* (***b***), *3* (***c***)

Рис. 3. Распределение частиц гидрида титана при режимах измельчения *1* (***a***), *2* (***b***), *3* (***c***)

Obtained titanium hydride powder particle morphology was studied with an electron-scan microscope LEO 1450. The particle size was studied with a laser particle analyzer MicroSizer 201. Impurity elements content of carbon, oxygen, nitrogen and hydrogen was determined using analyzers Leco CS-600, AK-7716P, TC-600 and RHEN-602 respectively. Powder iron content was estimated using Concentration photoelectric photometer-3-01.

Results and discussion

As a result of the SHS hydrogenation of the titanium sponge titanium hydride with hydrogen content of 4.2 wt.% was obtained. It is assumed that the high hydrogen content is recorded due to the concentration diffusion caused by high hydrogen reactor pressure (30 atm) during synthesis. The large amount of dissolved hydrogen in the crystal lattice of titanium causes embrittlement of the latter and makes it possible to grind a large sponge with a particle size from 20 mm to 40 μm.

After grinding powders were divided into 4 density compositions, that correspond to the particle size distribution of PTK, PTS, PTM and PTOM types. Fig. 4 shows histograms of the investigated titanium hydride powders particle distribution. PTK and PTOM samples have narrower particle distribution in comparison with PTS and PTM.

Impurities play an important role in the quality of products made of the titanium powders. Final product presence of small amounts of metallic impurities, such as iron or aluminum, does not significantly affect its properties. The presence of non-metallic impurities such as oxygen, nitrogen and carbon should be strictly limited, as with the introduction of titanium they form solid solutions and chemical compounds that significantly reduce its moldability. A chemical analysis of the starting material and the synthesized powder was therefore carried out (Table 2). As a result, it was found that during SHS hydrogenation the carbon and oxygen content decreased, which indicates a self-cleaning of the titanium sponge. Supposedly, the impurities reduction occurs during the combustion process when the initial titanium sponge is sharply heated. It causes a significant increase in the diffusion coefficient and promotes the diffusion mass transfer of impurity atoms to the particle surface. Already on the surface, nitrogen and carbon impurities atoms can form molecules, which are subsequently desorbed into the gas state [24]. During mechanical grinding the iron content increases insignificantly due to the short grinding time.

A shape of the titanium powder particles largely determines its behavior at all stages of the technological process of obtaining products. It also significantly influences its technological properties. Fig. 5 (SEM-images) shows the general appearance of the obtained titanium hydride powders. The images clearly show that the hydride particles are of irregular splinter shape. This morphology characterizes the powders obtained by means of this technology [14].

Titanium hydride surface micro structure (Fig. 6) partially preserves the lamellar structure of the titanium sponge. As seen (Fig. 6, *a*, *c*), the surface structure is similar to that after annealing and consists of large elongated oriented grains. Traces of stress cracking caused by various titanium and titanium hydride specific volumes are noticeable on titani-

Table 2. **Impurity content, wt.%, in the synthesized titanium hydride powder**

Таблица 2. Содержание примесей, мас.%, в синтезированном порошке гидрида титана

Sample	C	N	O	Fe
Initial titanium sponge TG-100	0.52 ± 0.03	0.11 ± 0.01	0.51 ± 0.01	0.02 ± 0.001
PTK	0.18 ± 0.01	0.17 ± 0.01	0.26 ± 0.01	0.03 ± 0.001
PTS	0.11 ± 0.01	0.17 ± 0.01	0.21 ± 0.01	0.03 ± 0.001
PTM	0.13 ± 0.01	0.28 ± 0.01	0.32 ± 0.01	0.04 ± 0.001
PTOM	0.17 ± 0.01	0.27 ± 0.02	0.25 ± 0.01	0.04 ± 0.001

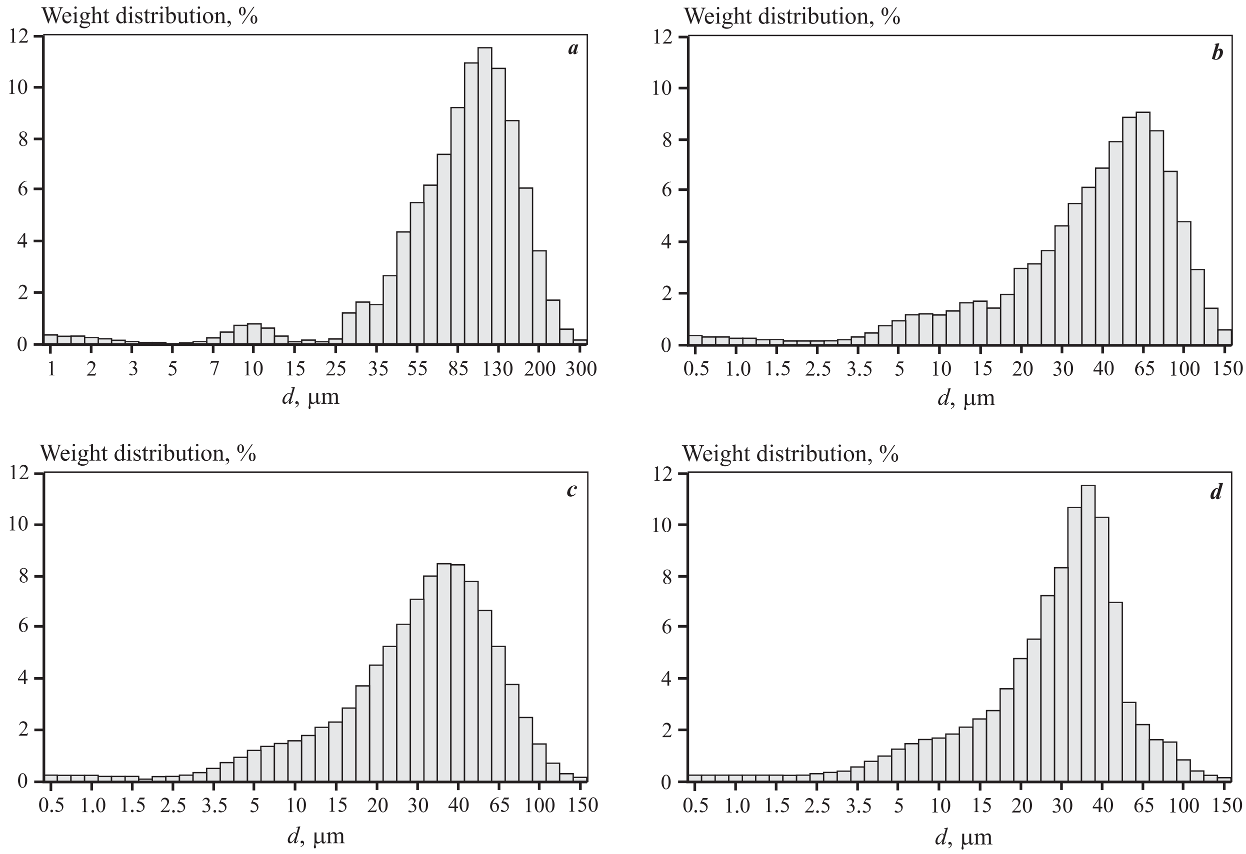

Fig. 4. Histograms of the synthesized titanium hydride powder particle distribution by size
PTK (***a***), PTS (***b***), PTM (***c***), PTOM (***d***)

Рис. 4. Гистограммы распределения частиц синтезированного порошка гидрида титана по размерам
Изучены образцы ПТК (***a***), ПТС (***b***), ПТМ (***c***), ПТОМ (***d***)

um hydride surface layers (Fig. 6, *b*). Fig. 6, *d* shows a surface of an intergranular brittle fracture. Its appearance is characterized by relatively smooth surfaces.

Powders of the same chemical composition but with different physical characteristics can have different technological properties affecting the conditions of the further transformation of powders into products.

The technological properties of the synthesized titanium hydride powder are shown in Table 3.

Bulk density is a powder volumetric characteristic which represents a ratio of the powder mass to its volume in free-fall. Its value depends on the powder particles packing density considering certain freely filling volume. The greater the packing density, the coarser and more regular the powder particles and the greater their

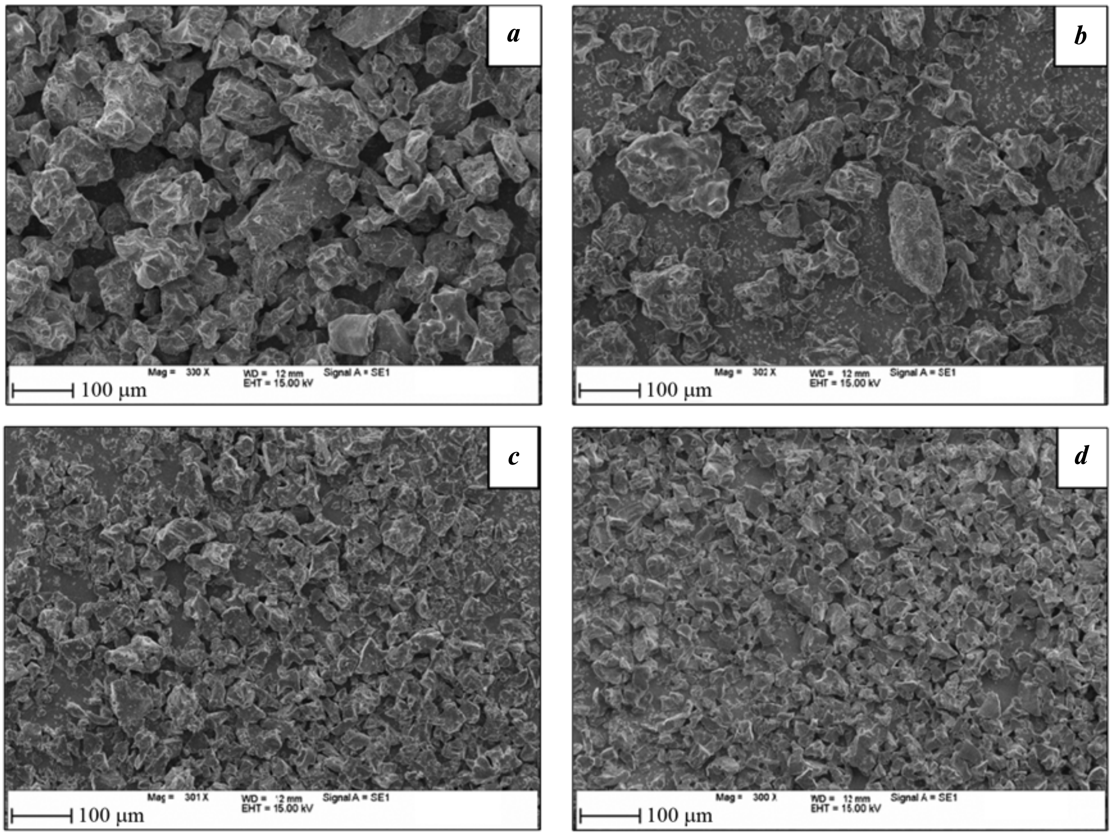

Fig. 5. Synthesized titanium hydride powder general appearance
a — PTK, ***b*** — PTS, ***c*** — PTM, ***d*** — PTOM

Рис. 5. Общий внешний вид синтезированного порошка гидрида титана
a — ПТК, ***b*** — ПТС, ***c*** — ПТМ, ***d*** — ПТОМ

pycnometric density. Obtained titanium hydride powders bulk density value reduces with decreasing particle size. The PTK sample has a wider fractional composition, which leads to a denser packing of particles: small particles fill the cavities formed by the packing of larger ones. The PTOM sample has a greater specific surface area due to the decreased particle size, which contributes to friction between particles, making it difficult for them to move relative to each other and leading to powders bulk density value reduction.

Powder compaction is the ability of a powder, under the influence of an external force, to acquire and retain a certain shape and size. Good compressibility makes the powder forming process easier and cheaper. The measured compaction values of the powders obtained are almost identical — this is because the titanium hydride particles are highly brittle and break apart during pressing to fill the voids.

Due to peculiarities of the production process, the particles of the metal powders may be characterized by significant internal porosity and the presence of a large number of cavities in the lattice nodes. As a result, the actual density of the particles may differ significantly from the one calculated by using radiographic lattice-parameter determination data. Obtained titanium hydride particles density correlates with the theoretical one of 3.75 g/cm^3, which indicates the practically total absence of cavities in the particle material.

The specific surface area of disperse bodies is the surface area of a powder unit mass or volume. The specific surface area depends not only on the particles size but also on the degree of the development of their surface, which is determined by the obtaining of powders conditions. The specific surface area is a very important characteristic of powders: determines the content of adsorbed gases in powders, their corrosion resistance, sintering ability, and a number of other characteristics. The obtained values for the specific surface area of titanium hydride powders are close to the values for spheri-

Table 3. **Titanium hydride synthesized powder technological properties**

Таблица 3. Технологические свойства синтезированного порошка гидрида титана

Sample	Bulk density, g/cm^3	Compaction, g/cm^3, at 200 MPa	Pycnometric density, g/cm^3	Specific surface area, m^2/g
PTK	1.38 ± 0.04	2.83 ± 0.04	3.79 ± 0.01	0.6 ± 0.01
PTS	1.31 ± 0.03	2.85 ± 0.03	3.81 ± 0.01	0.6 ± 0.01
PTM	1.30 ± 0.02	2.86 ± 0.03	3.80 ± 0.01	0.6 ± 0.01
PTOM	1.16 ± 0.02	2.88 ± 0.03	3.72 ± 0.01	0.7 ± 0.01

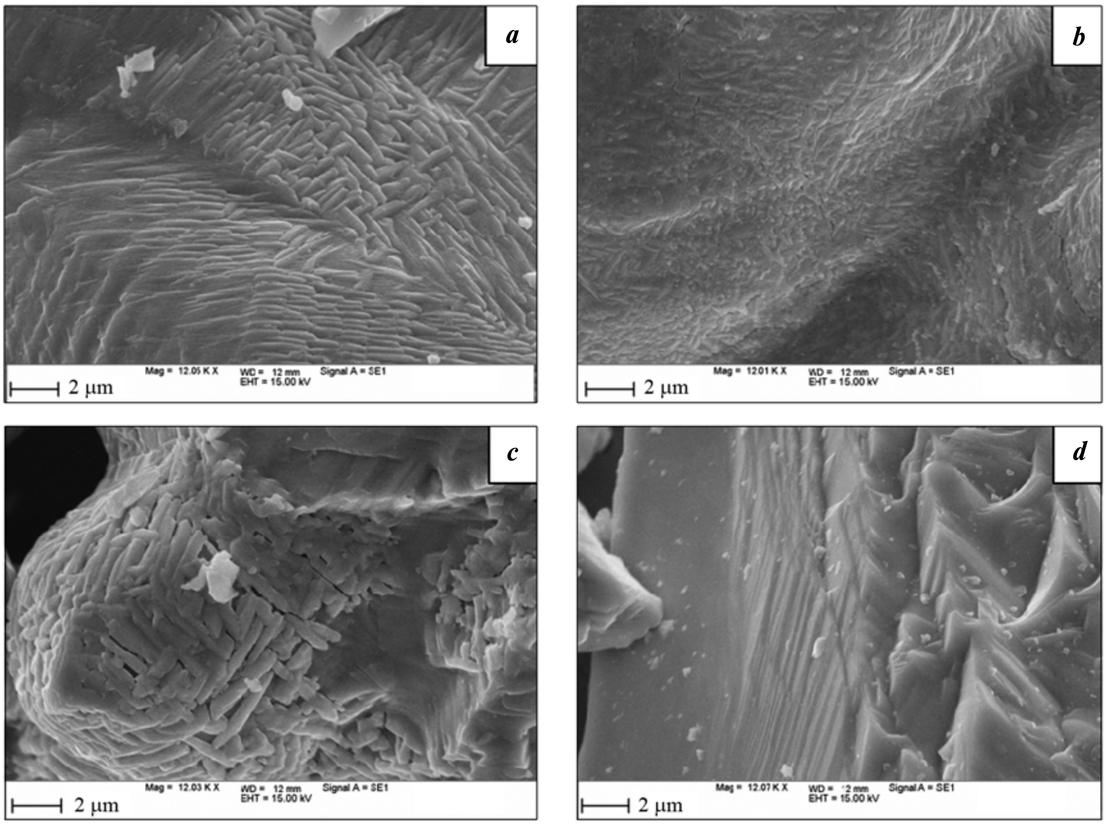

Fig. 6. Synthesized titanium hydride powder micro structure
a — PTK, ***b*** — PTS, ***c*** — PTM, ***d*** — PTOM

Рис. 6. Микроструктура синтезированного порошка гидрида титана
a — ПТК, ***b*** — ПТС, ***c*** — ПТМ, ***d*** — ПТОМ

cal powders (0.14 m^2/g), therefore, the obtained hydride powder does not have a developed porous surface which coherent with the received data on the morphology of the particles.

Conclusion

Experimental studies of the structure and properties of titanium hydride powders obtained from titanium sponge by the SHS method in a high-pressure reactor have been conducted.

It is found that due to high embrittlement of titanium hydride the hydrogenated titanium sponge is quickly and easily broken to a particle size of less than 40 μm. Iron impurity content increases insignificantly during the mechanical grinding process. The SHS hydrogenation leads to a product self-cleaning and also to carbon and oxygen impurities reduction.

Titanium hydride particles have an irregular splinter shape. Generally speaking, the shape and defects of particles are typical of the powders obtained by this method. Particles surface structure consists of elongated oriented grains. Due to stresses resulting from various specific volumes of titanium and titanium hydride the titanium hydride surface layers show traces of cracking.

A study of the technological properties demonstrated that the obtained powders possessed necessary parameters for application in powder metallurgy.

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