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THE INFLUENCE OF CHEMICAL-THERMAL TREATMENT ON GRANULOMETRIC CHARACTERISTICS OF TITANIUM SPONGE POWDER

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Abstract. The phase composition and structure of powders of titanium sponge exposed to processes of chemical and thermal processing is investigated.

As a result of X-ray analysis, it is found that after hydrogenation titanium powder consists of $TiH_{2\pm x}$ phase, while with the increase in the degree of phase dehydrogenation the percentage of $TiH_{2\pm x}$ decreases, and under perfect the dehydrogenation the structure consists mainly of α-Ti. Particle size analysis revealed that the partially dehydrated powders have the least degree of polydispersity.

It is established that by means of regulation of technological regimes of hydrogenation-dehydrogenation the two-phase structure with different ratios of α-phase to $TiH_{2\pm x}$ can be synthesized.

It is shown that the presence of titanium hydride contributes to stabilization of the structure of formed powders.

Keywords: structure, titanium, phase hydride, granulometric characteristics, properties, powder.

Introduction

The need to save financial resources and reduce production costs requires producers and processors of titanium and its alloys, improving existing technologies shaping products and introduction of new highly efficient solutions. Recently, high-tech industries such as engineering, aviation and aerospace industry there is a significant intensification of research to develop new technological approaches as expensive as getting parts using additive technologies (3D printing technologies) [1; 2]. This approach is based on the formation of the layered objects when initially each new layer of detail of the future is a powder fraction consisting of the same or different in shape and particle size structural material [3; 4].

Modern laser 3D-printers can work with different materials. Most of the technology is used as a consumable metal powders. Among the most promising are titanium alloy powders that are characterized by a unique combination of high structural strength, low density and high corrosion resistance. However, the complexity of manufacturing operations necessary for the formation of spherical particles leads to a significant increase in the cost of products [4]. In this regard, the development of alternative technological approaches metallic powders suitable additive technology is an extremely urgent task.

Substantiation of the problem

One of the promising methods for metal powder technology with optimal performance is the use of chemical and heat treatment in a hydrogen atmosphere. Established that hydrogenation-dehydrogenation titanium alloy powders receiving promotes changing the shape of its particles [5]. Even small hydrogen content (0.3-3.0 wt. %) Experienced higher density products after sintering by grinding and rounding the particles of powder fraction [6]. However, currently getting dispersed powder of spherical particles has a number of technical difficulties [7], accompanied by the complication of the process of production, which in turn leads to an increase in their value. In this regard, economically and technologically viable objective is to optimize the parameters getting cheaper powders from titanium, which will use them as an alternative raw material for additive technology in aircraft and engine.

This work is devoted to one of these parameters, such as size distribution of the powder and the impact of previous chemical and thermal processing.

Technique of research

Powders obtained by mechanical grinding of titanium sponge in a planetary mill, resulting have an irregular (non-spherical) shape and the surface of the developed structure.

The preferred method of identification phase using X-ray phase analysis (XRD). Powder diffraction filmed on a DRON-3M diffractometer using copper radiation anode (Cu K_α-radiation).

Diffraction unscrambled using software packages PowderCell [8] and FullProf [9] on the basis of crystallographic data from the database ICSD [10].

Powder morphology was studied using optical and electron microscopy. In the case of optical microscopy studies were conducted using an optical microscope MBS-9 equipped with camera glasses eTREK-520. This sample testing thoroughly mixed glass, scattered band of a certain length and divided into about 7 or 8 equal parts. Pair of cast and odd-mixed and re-cut in this way. Next, a small amount of powder was transferred to a glass slide and evenly distributed. Fixing the image performed at least three specific fields of view.

Electron microscopy was performed using a scanning electron microscope EVO 40XVP. This sample powder obtained as described above, was applied to the conductive tape. Research conducted for the increase in 50 and 100 times.

Particle size analysis was performed using specialized material science complex image analysis ImageJ [11].

The degree of heterogeneity (polydispersity) powder, depending on the dominant medium sized particles in some fractions and standard deviation of the particle size of the powder of medium size [12], was defined by Gauss curve based on the histogram distribution of particles in a certain faction [13].

Results and discussion

The process of obtaining titanium powder is titanium sponge crushing blocks and the subsequent scattering of chopped on fractions. Small particles sponges that comprise the study powder, characterized by its own porosity and can contain several grains [14]. Titanium hydride traditionally produced by heating the metal in vacuum furnaces, followed by exposure to a hydrogen atmosphere at 400–600 °C [15]. This hydrogenation process takes several hours and requires significant energy costs and significant duration increases the risk of process contamination hydrides derived impurities. Therefore, in the future conduct dehydrogenation process that not only leads to the withdrawal of material harmful impurities, but also to fining the microstructure of the material.

According to [16] it is known that the sharp decrease in the concentration of hydrogen in the hydride is in the temperature range 500–850 °C. However, the high-temperature heat is not sufficient to completely remove hydrogen from the powder, since hydrogen is not completed even at 1350 °C.

According to RFA revealed that the source powder contains only α-Ti (Fig. 1, Table 1). Found that in the process of hydrogenation, hydride formed $TiH_{2\pm x}$ (Fig. 1, Table 1). In [16] the results showing that adjusting the content of titanium hydride can correlate the mechanical properties of extruded samples are presented. It is known that with increasing content $TiH_{2\pm x}$ increasing hydrogen content and also reduces the oxygen content, which improves the mechanical properties of the powder. However, there is no data in the literature regarding the influence of chemical and heat treatment in hydrogen atmosphere at granulometric characteristics of titanium powders.

After partial dehydrogenation (Fig. 1, Table 1) is a two-phase structure powder (a mixture of α-Ti and $TiH_{2\pm x}$). In [17, 18] showed that the presence of a mixture of titanium powder 15–25 wt. % Titanium hydride helps particles spheroidation, which in turn improves workability and reduces sintering. As a result, the full source dehydrogenation phase composition of the powder is fully restored (Fig. 1d, Table 1).

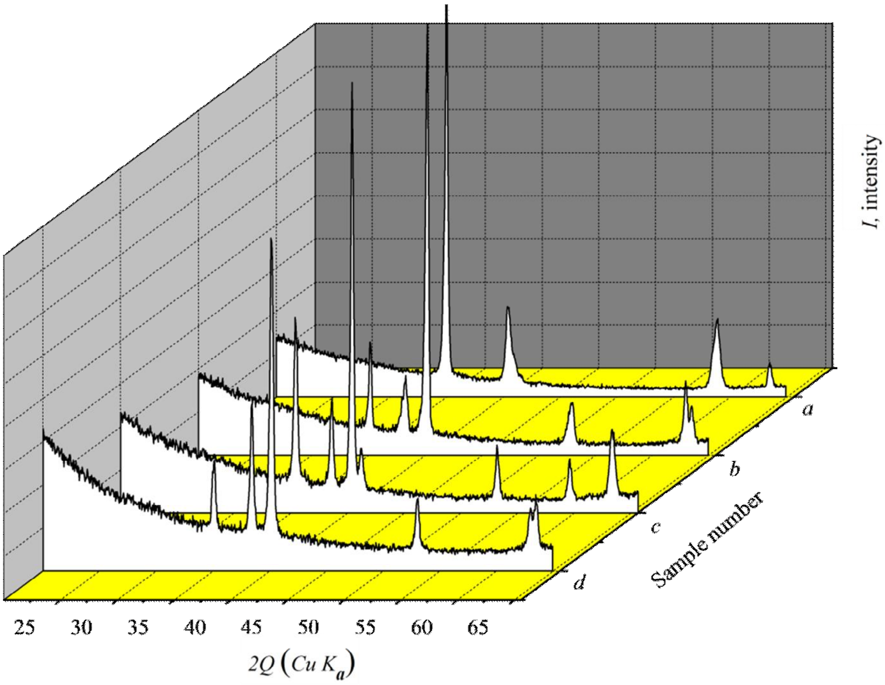


Fig. 1. *Powder diffraction pattern of titanium sponge after thermo-chemical treatment: a – hydrogenation; b – hydrogenation and dehydrogenation complete, c – partial hydrogenation and dehydrogenation; d – not subjected to chemical and thermal processing*

Table 1

Phase composition of samples of titanium sponge powder

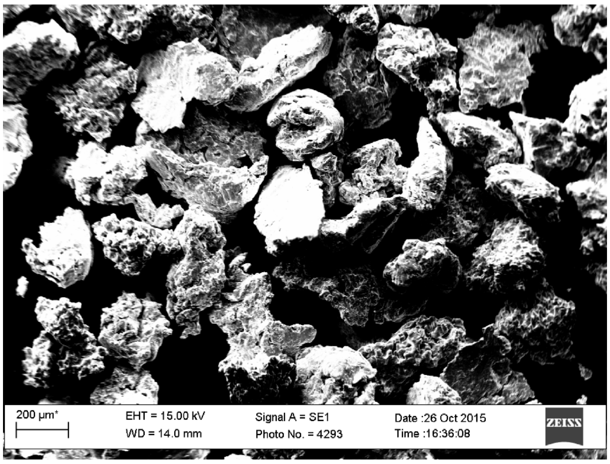
Sample	Phase composition	Content about, %	Grid settings, Å	
			a	c
Hydrogenated	TiH_2	100	4,431(1)	-
Wholly dehydrated	α-Ti	100	2,962(6)	4,739(3)
Partly dehydrated	α-Ti	77	2,942(1)	4,6810
	TiH_2	23	4,391(9)	-
No hydrogenated	α-Ti	100	2,941(9)	4,667(4)

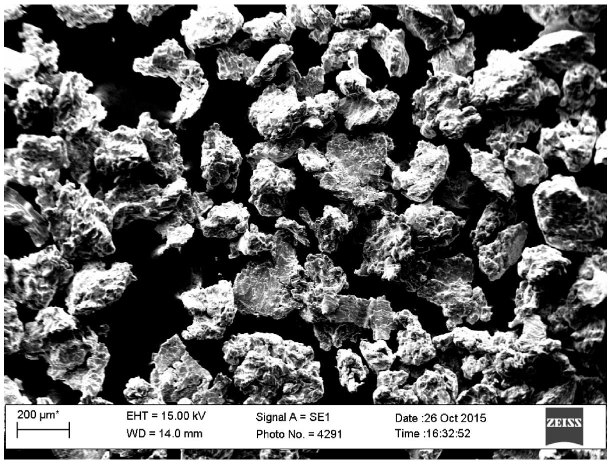
Analysis of the morphology of powders of different fractional composition (Fig. 2) showed that a reduction of the size fractions is uniformity of powder form and size. Thus, there is an almost complete absence of predisposition to conglomeration. However, the particle size of the powder is too large, so it used the grinding process of hydrogenation-dehydrogenation.

The powder that has passed only hydrogenation process (Fig. 3), has a more even distribution of powder particle size and particle conglomerates less specific, but in spite of this particle size is too large.

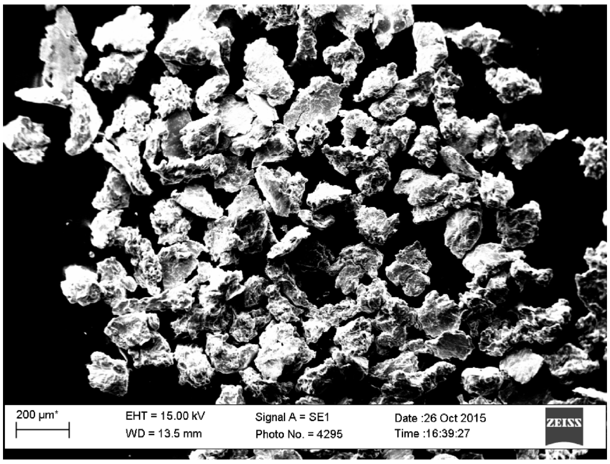
After complete hydrogenation-dehydrogenation (Fig. 4) powder morphology characterized by a significant spread of particle size and the formation of a large number of conglomerates. It can be assumed that the cause agglomeration is their high dispersion and lack of protective environment that would be impossible sintering processes of particles during grinding.

Comparative analysis of Figs. 4 and 5 showed a significant impact phase of the morphology of titanium powders. Yes, completely dehydrated powder, consisting only α-Ti, characterized by significant dispersion of particles of powder size and formation of large conglomerates number of different configurations. The presence of titanium hydride powder is stabilizing structure that is both to uniform powder particle size, and to reduce their tendency to conglomeration.





a *b*



c

Fig. 2. *Morphology of powders of titanium sponge different fractional composition:*
a – 0,315-0,2 mm; b – 0,25-0,1 mm; c – 0,1-0,063 mm

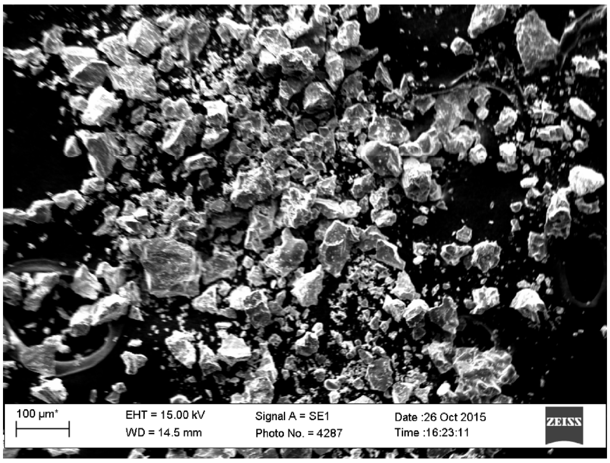


Fig. 3. *Morphology of powders of titanium sponge, after complete hydrogenation*

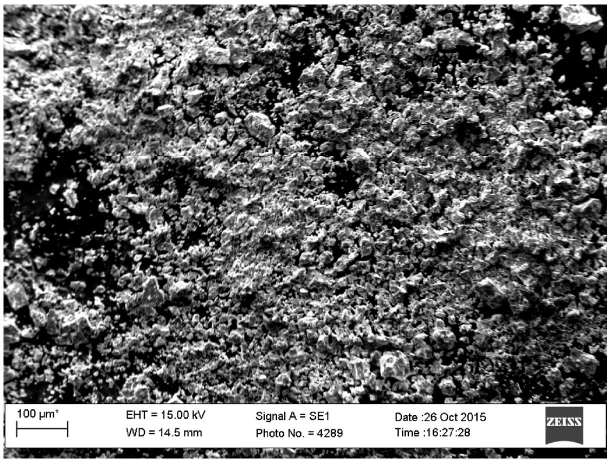


Fig. 4. *Morphology of powders of titanium sponge, after complete hydrogenation-dehydrogenation*

Thus, we can expect that adjusting process parameters regimes hydrogenation-dehydrogenation can adjust the proportion between α-Ti and $TiH_{2\pm x}$ for the best technological properties of the powder (bulk density, fluidity) when using them in additive technology.

To determine the effect of chemical and thermal processing on the particle size distribution was carried out to determine the area of projection section base particles on the plane. Image microstructures of titanium sponge powder (Fig. 5) and treated with digitizing using ImageJ. Agglomerates that could not be divided into individual particles in the calculations were not taken into account.

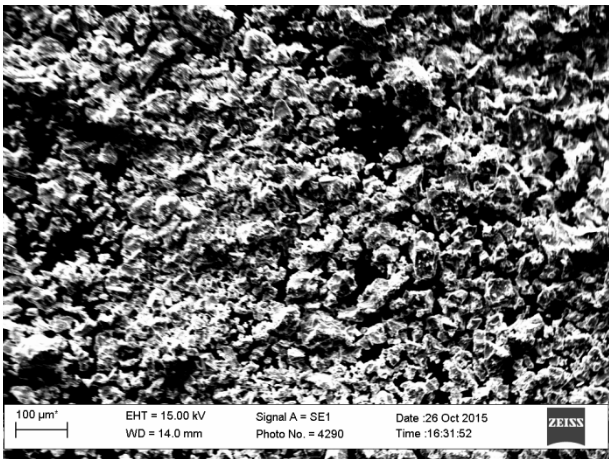
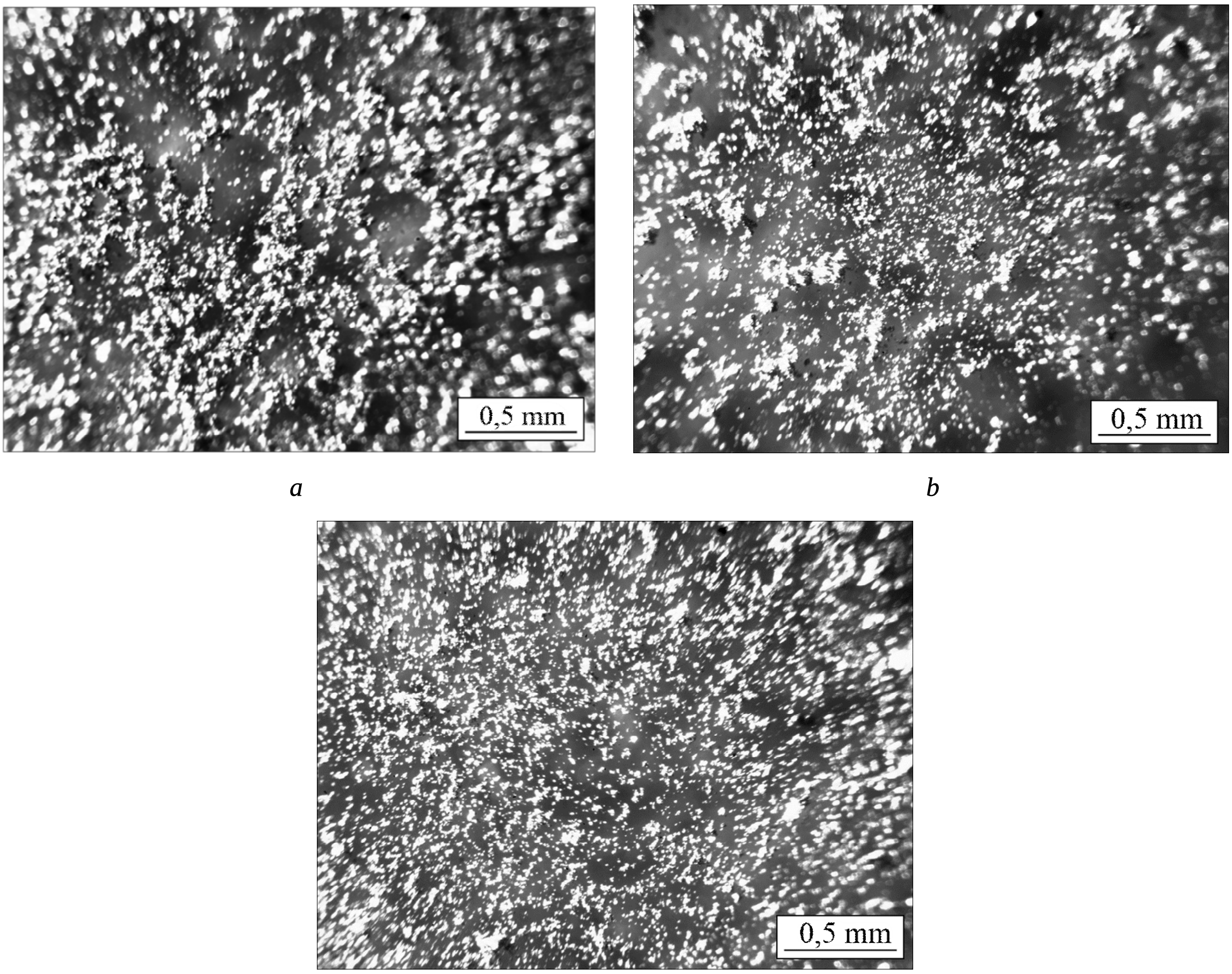


Fig. 5. *Morphology of powders of titanium sponge, after partial hydrogenation and dehydrogenation*

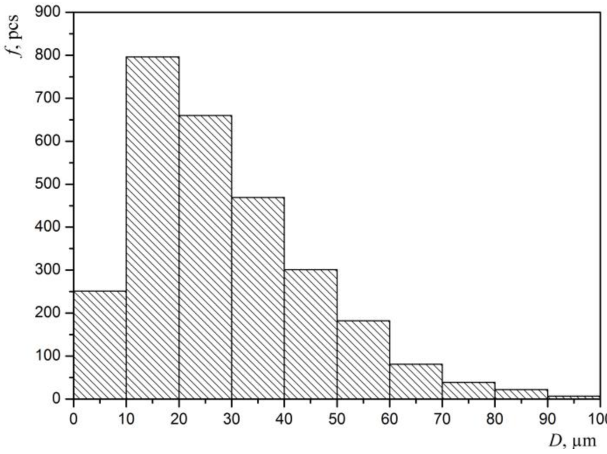


a *b*

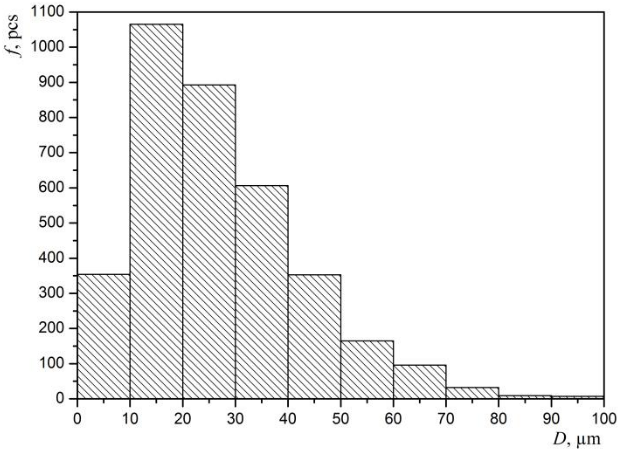
c

Fig. 6. *Morphology of powders of titanium sponge after thermo-chemical treatment:*
a – hydrogenation; b – partial hydrogenation and dehydrogenation; c – hydrogenation and dehydrogenation full

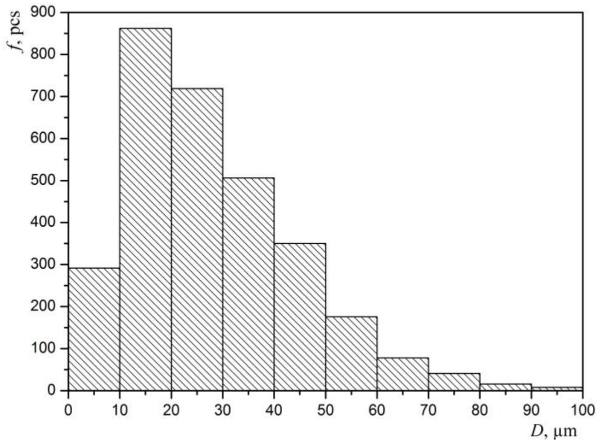
As a result of calculations for each image (Fig. 6) a number of intervals of the size distribution and average diameter Feret (Dser) powder particles (Fig. 7) were received. Also specific share of the dominant particle size for hydrogenated, partially dehydrated and dehydrated powder were set.



a



b



c

Fig. 7. *Histograms of distribution of powder particles of titanium sponge Feret diameter: a – hydrogenated; b – partially dehydrated; c – completely dehydrated*

To determine the dominant particle size powder, integral and differential curve based on the histogram size distribution (Fig. 8).

As a result of construction of differential and integral curves (Fig. 8), found that the predominant amount of powder on the lips for different modes of chemical and heat treatment is in the range from 3.5 to 27 microns. However, after hydrogenation, the dominant share of 60 % after partial dehydrogenation – 78 % and full dehydrogenation – 72 %. Analysis of differential curves shows that the least degree of polydispersity characteristic powder obtained by hydrogenation, dehydrogenation and partial grinding (Fig. 8). This suggests that the process of hydrogenation-dehydrogenation titanium alloy powders can be regarded as a positive process, by which the possible adjustment of fractional powder is done. In particular, the results of comparative microstructural studies indicate that the presence of a phase of titanium hydride powder structure facilitates grinding powder uniformity and their size.

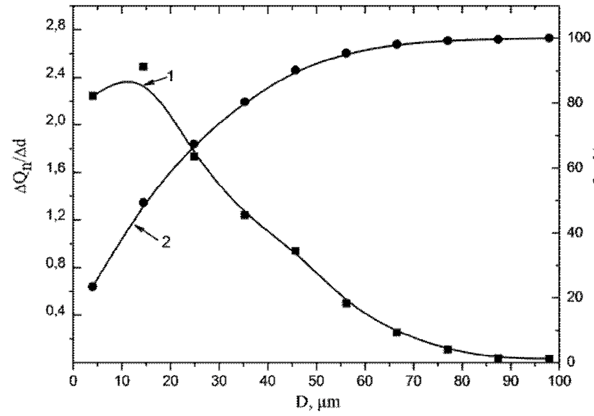
Conclusions

1. The relation between chemical-thermal treatment and phase composition of the powder is revealed.

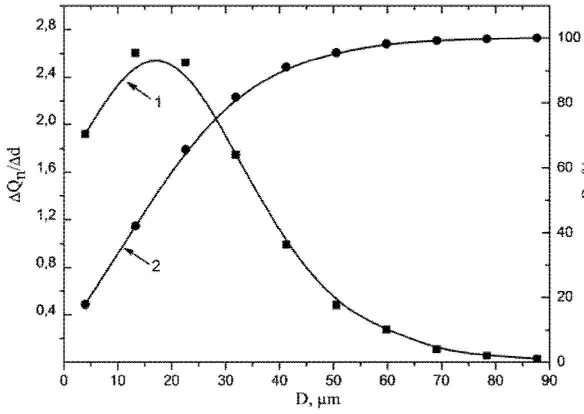
2. It is established that regulation of technological regimes of hydrogen treatment can affect granulometric characteristics of powders of titanium alloys due to purposeful change of the proportion between the structural components.

3. For the optimal, from technological point of view, fractional composition of the powder, prior screening of fractions of powders with the average diameter of particles larger than 25 microns is recommended.

4. It is confirmed that the process of hydrogenation-dehydrogenation of titanium alloy powders can be regarded as a positive factor by which the adjustment of fractional powder is possible.



a



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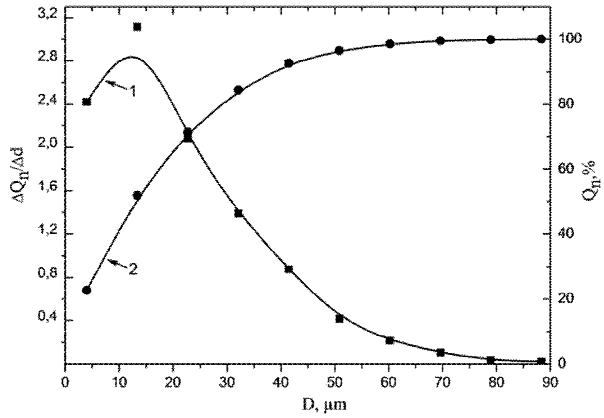

c

Fig. 8. *Differential (1) and integral (2) quantitative distribution curves of titanium sponge powder particle diameter Feret: a – hydrogenated; b – partly dehydrated; c – completely dehydrated.*

References

[1] Александров А. В. Развитие рынка титана в СНГ // материалы конференции Международной конференции Ti2009 в СНГ. – К.: РИО ИМФ им. Г. В. Курдюмова НАН Украины, 2009. – С. 7–10.

[2] Петрик И. А., Овчинников А. В., Селиверстов А. Г. Разработка порошков титановых сплавов для аддитивных технологий применительно к деталям ГТД // Авиационно-космическая техника и технология. – 2015. – № 8. – С. 11–16.

[3] Довбыш В. М. Аддитивные технологии и изделия из металла / В. М. Довбыш, П. В. Забеднов, М. А. Зленко // Библиотечка литейщика. – 2014. – № 9. – С. 14–71.

[4] Зленко М. А. Аддитивные технологии в машиностроении / М. А. Зленко, А. А. Попович, И. Н. Мутылина – СПб. : Издательство политехнического университета, 2013. – 221 с.

[5] Овчинников А. В., Ольшанецкий В. Е., Джуган А. А. Применение несферических гидрированых и дегидрированых порошков титана для получения изделий в аддитивных технологиях // Вестник двигателестроения. – 2015. – № 1. – С. 114–117.

[6] Влияние содержания гидрированного титана в смеси порошков на механические свойства спеченного сплава ВТ1-0 / А. А. Скребцов, А. В. Овчинников, В. Г. Шевченко [и др.] // Строительство, материаловедение, машиностроение: Сб. науч. трудов. – 2014. – Вып. 73. – С. 89–94.

[7] Заявка 2361698 Российская Федерация, МПК B22F 9/10. Способ получения сферических порошков и гранул / А. К. Давыдов, В. И. Миронов и др. – № 2008110117/02; заявл. 19.03.2008; опублик. 20.07.2009, Бюл. № 20.

[8] The Collaborative Computational Projects CCP14 [Electronic resource]. – Access mode: http://www.ccp14.ac.uk.

[9] Rodriguez-Carvajal, Recent. J. Developments of the program FULLPROF // Commission on Powder Diffraction. – 2001. – 26. – pp. 12–19.

[10] Introduction to ICSD Web [Electronic resource]. – Access mode: https://icsd.fiz-karlsruhe.de.

[11] Конюхов А. Л., Руководство к использованию программного комплекса ImageJ для обработки изображений: Учебное методическое пособие. – Томск : кафедра ТУ, ТУСУР, 2012. – 105 с.

[12] Walpole Roland E., Myers Raymond H. Probability and Statistics for Engineers and Scientists. – New York : Macmillan Publishing Company, 1985. – 639 p.

[13] Микроскопические методы определения размеров частиц дисперсних материалов : учеб. пособие / Н. Н. Гаврилова, В. В. Назаров, О. В. Яровая. – М. : РХТУ им. Д. И. Менделеева, 2012. – 52 с.

[14] S. Zhang. Hydrogenation behavior, microstructure and hydrogen treatment for titanium alloys // Progress in Hydrogen Treatment of Materials. – 2001. – pp. 282–298.

[15] Водород в титане : монография / В. А. Ливанов, А. А. Буханова, Б. А. Колачев. – М. : Металлургиздат, 1962. – 245 с.

[16] Ma Qian, Francis H. Froes. Titanium powder metallurgy. – Butterworth-Heinemann, 2015. – 628 p.

[17] Fngelo H. C., Subramanian R. Powder Metalurge: Science, technology and application. – New Dehli, 2008. – 312 p.

[18] Ahsan M. N. et. al. A comparative study of laser direct metal deposition characteristics using gas and plasma-atomized Ti-6Al-4V powders // Materials Science and Engineering. – 2011. – pp. 7648–7657.