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## The Effect of TiH<sub>2</sub> Particle Size on its Properties

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Titanium-based composites have the potential to replace traditional materials in the automotive, aerospace and other industries. This is facilitated by their excellent mechanical, physical, thermal properties, and their strength-to-weight ratio.

In the presented work, the influence of the particle size of hydrogenated titanium on its conductive properties was investigated. Hydrogen saturation was carried out using the Stevenson apparatus, the hydrogen content was 3.8%. After that, the initial sample was subjected to high-energy grinding. 3 main particle sizes were identified for further research: 100 μm, 100-200 μm, over 200 μm.

Resistometric studies have shown that particles of 100-200 μm are characterized by an order of magnitude higher electrical conductivity than other particle sizes (8 (Ohm·cm)<sup>-1</sup>), which is due to the maximum contact between neighboring particles and their optimal geometry when packing the powder sample. Based on this powder size, a nanocomposite with carbon nanostructures was synthesized using mechanosynthesis, which is characterized by an order of magnitude higher electrical conductivity compared to the original components, which indicates the possibility of using such materials in the electrical engineering industry.

**Key words:** TiH, powder, electric conductivity, composite.

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## Introduction

The energy industry is one of the fundamental factors of economic development and shaping the standard of living of society and, at the same time, is closely related to global environmental problems caused by energy production and consumption. Despite the fact that the world's energy sector is traditionally based on non-renewable sources, in recent decades there has been a steady trend toward a transition to alternative, practically inexhaustible, and environmentally safer energy sources, in particular solar, wind, and hydrogen [1,2]. At the same time, existing energy conversion technologies have significant limitations: photovoltaic systems are characterized by environmental problems associated with production and utilization, as well as the need for large areas, while thermionic converters (TECs) are promising in terms of efficiency and environmental friendliness, their practical application, however, is constrained by high

operating temperatures, which necessitates the development of new materials for low-temperature operating modes [3,4].

Hydrogen technologies occupy a special place in modern energy, since hydrogen, owing to its unique physicochemical properties, high diffusion capacity, and reversible interaction with materials, is a promising energy carrier and an effective alloying element [5,6]. The safest and most promising materials for storing and transporting hydrogen are metal hydrides, which provide a high degree of hydrogen binding and the possibility of controlled release [7–12]. In this regard, an urgent scientific and technical task is the development of new hydrogen-accumulating nanocomposite materials with increased hydrogen capacity and accelerated kinetics of hydrogen absorption and desorption processes. Such materials are critically important not only for hydrogen energy but also for creating effective cathodes of low-temperature thermal power plants, since atomic hydrogen

helps clean metal surfaces from oxide films and reduces the work function of electrons from carbon components [13–15].

Among hydride materials, titanium hydride (TiH<sub>2</sub>) powder attracts special attention, as it combines promising structural, thermal, and electrophysical properties [16]. Its ability to be subjected to controlled dehydrogenation, forming active metallic titanium and releasing hydrogen, creates unique conditions for regulating the electrical conductivity, microstructure, and phase composition of composite materials [17]. At the same time, the limited electrical conductivity of TiH<sub>2</sub> necessitates its modification to expand its practical applicability.

An effective approach to controlling the properties of titanium hydride is to create composites based on it with carbon nanotubes (CNTs), which are characterized by high electrical conductivity, large specific surface area, and the ability to form percolation conductive networks at low concentrations [18,19]. The introduction of CNTs into the metal matrix ensures the formation of effective conductive channels, stabilization of the microstructure, and reduction of particle aggregation [20]. During the heat treatment of such composites, dehydrogenation of TiH<sub>2</sub> occurs with the formation of metallic titanium, and in the presence of a carbon phase, the formation of nanosized carbide phases of the TiC type is possible, which further increases the thermal stability and mechanical characteristics of the material.

Given the widespread use of metal powders in powder metallurgy, additive manufacturing technologies, and 3D printing, it is particularly relevant to study the influence of the dispersion composition and particle size on the electrophysical properties of materials. In this context, TiH<sub>2</sub>-CNT-based systems are a convenient model for studying percolation effects, interfacial interactions, and the influence of nanoscale carbon additives and titanium hydride particle size on electrical conductivity and structural transformations. That is why the study of the electrical conductivity of TiH<sub>2</sub> powders with different particle sizes and their composites with carbon nanotubes is an urgent and scientifically substantiated task, which forms the focus of this work.

## I. Experiment

In this work, the effect of the particle size of hydrogenated titanium on its conductive properties was investigated. For this purpose, the titanium sponge was previously saturated with hydrogen. The interaction of titanium with hydrogen was studied using the Sieverts method on the IVGM-2M setup at a temperature of 450°C and a pressure of 0.6 MPa, according to the method described in detail in [21,22]. The content of absorbed hydrogen was determined by the volumetric method based on the difference in pressure in a closed volume before and after heating the sample, and was also controlled gravimetrically—by the change in the mass of the sample before and after hydrogenation. The use of thermosorption-purified hydrogen and labyrinth crucibles made it possible to minimize the effect of oxidation on the change in the mass of the samples. According to the measurement results, the hydrogen content in the material

was 3.8%. The obtained titanium hydride was subjected to high-energy grinding in a ball mill, after which three characteristic particle fractions were isolated for further studies: about 100 μm, 100–200 μm, and over 200 μm.

The phase composition and lattice parameters of the alloy were determined by X-ray phase analysis at a DRON-3M diffractometer.

Nanocomposite materials were obtained by mechanosynthesis, i.e., mechanical mixing of the initial powder components in different ratios for 8 hours. The study of the electrical conductivity properties was carried out by the resistometric method, according to the method described in detail in [23,24]. According to this method, the powder sample was placed in a dielectric cylinder between the electrodes and subjected to stepwise compression with simultaneous measurement of electrical conductivity and density. After achieving the maximum possible compaction, the reverse unloading process was carried out with parallel registration of electrophysical characteristics. The specific electrical conductivity  $\sigma$  was determined by the ratio:

$$\sigma = \frac{m}{R\rho S^2}, \quad (1)$$

where  $\rho$  is the density of the compressed powder sample,  $S$  and  $h$  are the area and thickness of the powder sample layer,  $R$  is the electrical resistance of the sample, and  $m$  is its mass.

The applied approach allows accounting for the anisotropy of the particle shape and changes in their mutual arrangement during loading–unloading cycles, which is important for the correct determination of the transverse electrical conductivity of powder materials.

## II. Results and discussion

The advantage of using materials in powder form lies in the possibility of creating products of a desired shape with specific properties, and this type of production is almost waste-free. The size of the powder fraction significantly influences the properties, so it is important to understand precisely how it affects them. The electrical conductivity of titanium hydride powder with particle sizes of 100 μm, 100–200 μm, and over 200 μm was studied. The X-ray diffraction pattern of the studied powder is presented in Fig. 1, where the corresponding peaks confirm that the material is indeed titanium hydride.

Fig. 2 shows the dependence of electrical conductivity on the degree of compression of a TiH<sub>2</sub> powder sample with a particle size of 100 μm during compressive deformation. The initial value of electrical conductivity is recorded at a density of  $\rho_0 = 0.161 \text{ g/cm}^3$  and is  $\sigma = 10^{-3} (\text{Ohm}\cdot\text{cm})^{-1}$ . Subsequent compression to a density of  $1.85 \text{ g/cm}^3$  (the maximum achievable compression with the apparatus) is accompanied by a gradual increase in electrical conductivity to  $0.48 (\text{Ohm}\cdot\text{cm})^{-1}$ . During compression, the powder becomes more compact and fills the voids between neighboring particles, which increases the degree of contact between particles and contributes to the rise in electrical conductivity [25,26].

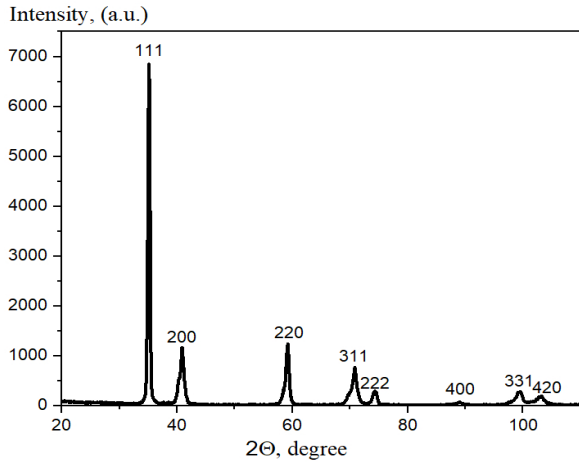


Fig. 1. XRD of powder TiH<sub>2</sub>.

Graphs showing the dependence of specific electrical conductivity on the degree of compression (density) for particles with sizes of 100–200 μm and over 200 μm are presented in Fig. 3a and 3b, respectively. For particles with a size of 100–200 μm, the transition density to the conductive state remains unchanged, but an increase in the initial electrical conductivity by a factor of four is observed (Fig. 3a). An increase in particle size to 200 μm is accompanied by a 25% decrease in the transition density to the conductive state, while the initial value of electrical conductivity remains unchanged.

For all samples, the maximum electrical conductivity is observed at the maximum hardware-possible compression in this experiment and is 0.48, 8.1, and 0.7 (Ohm·cm)<sup>-1</sup> for particles with sizes of 100, 100–200, and 200 μm, respectively. This discrepancy in conductivity values is due not only to particle size but also to the packing characteristics of the samples during compression and the contact area between neighboring particles.

Titanium by itself is not a good conductor, but there are certain applications where the use of metal matrix composites based on titanium offers distinct advantages [30]. Therefore, a composite based on titanium hydride with 20 wt.% CNTs was created. The general view of this composite is presented in Fig. 4a, where it can be seen that the carbon component forms a thin layer around the titanium hydride particles. Multi-walled carbon nanotubes

(MWCNTs) with a diameter of 18 ± 6 nm were used, synthesized via chemical vapor deposition (CCVD) on an Al<sub>2</sub>O<sub>3</sub>–Fe<sub>2</sub>O<sub>3</sub>–MoO<sub>3</sub> catalyst in propylene vapor at 923 K. The diameters of the tubes ranged from 10 to 25 nm. Dark catalyst particles are visible inside some CNTs (Fig. 4b). According to spectroscopic analysis, the CNT sample contains 98.1 wt.% carbon, 1.51 wt.% oxygen, and 0.39 wt.% iron, confirming the presence of catalyst particles inside the CNTs.

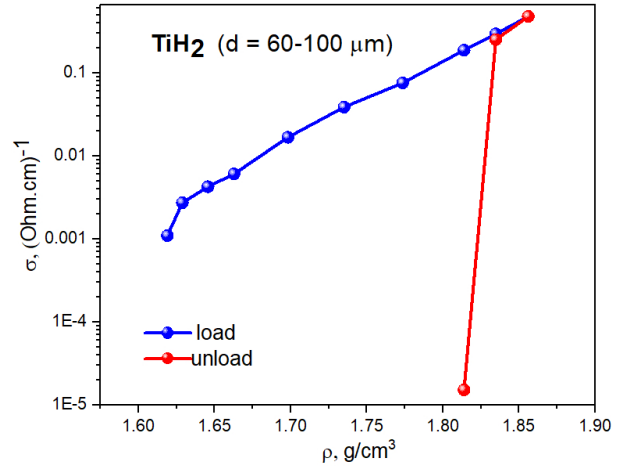


Fig. 2. Dependence of electrical conductivity on the degree of compression for powdered TiH<sub>2</sub> with a particle size of 100 μm.

Resistometric studies of the "TiH<sub>2</sub> +20 wt.% CNTs" sample obtained by mechanosynthesis are presented in Fig. 5a, where the dependence of electrical conductivity on the degree of compression of the pristine CNTs is also shown (Fig. 5b). The general shape of the curves is convex, indicating the influence of CNTs on the electrical conductivity of the resulting composite.

As can be seen from Fig. 5, the addition of carbon nanotubes to TiH<sub>2</sub> leads to a slight increase in electrical conductivity compared to the individual components, but this value differs from that of the mechanical mixture, indicating the formation of a true composite [31]. Such composites are promising for use as cold cathodes in thermionic converters [32,33], and therefore these samples were selected for further studies under alternative synthesis conditions.

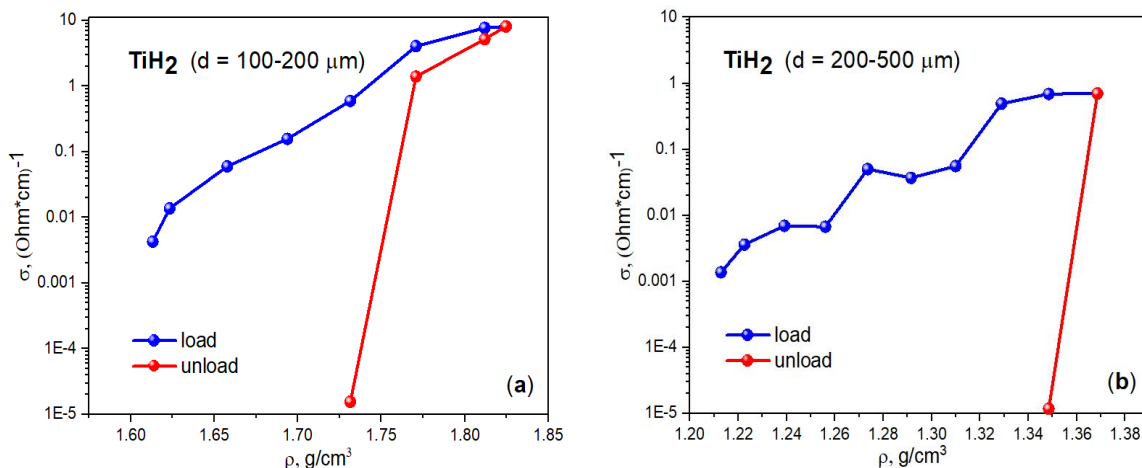
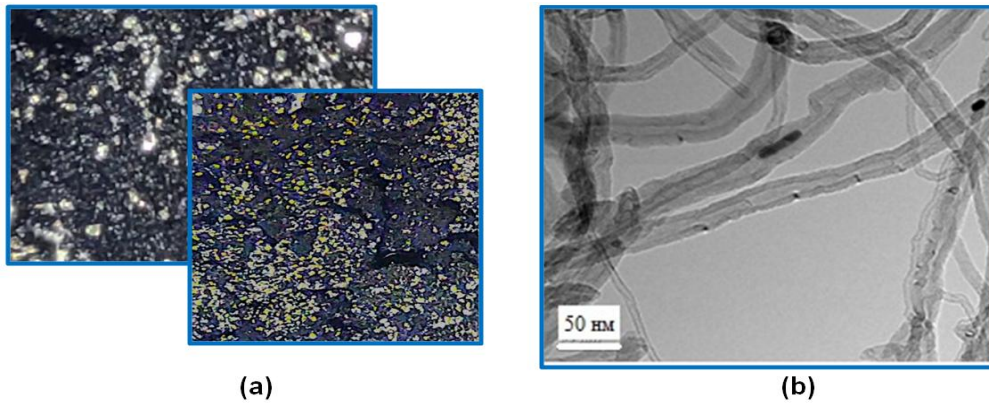
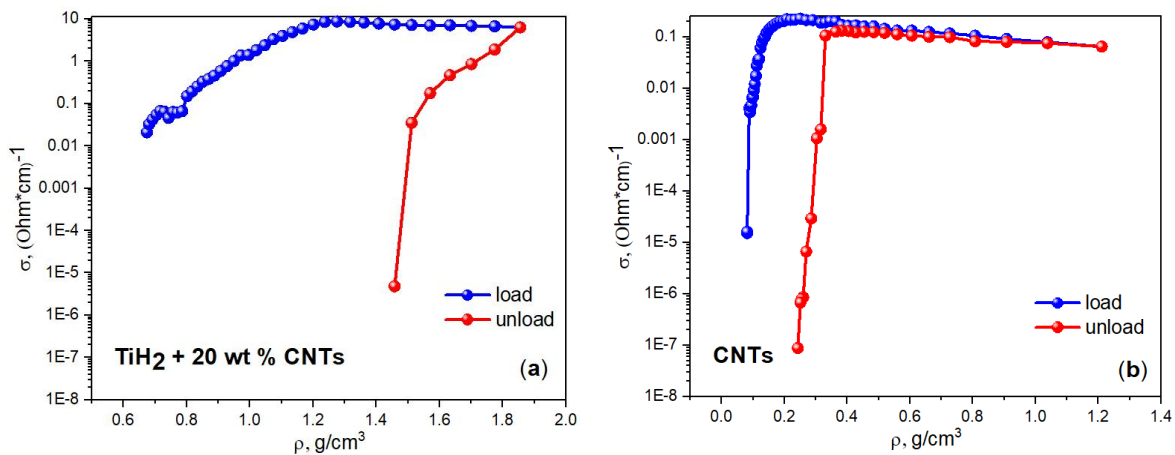


Fig. 3. Dependence of electrical conductivity on the degree of compression for powdered TiH<sub>2</sub> with particle sizes of 100–200 μm (a) and over 200 μm (b).



**Fig.4.** Image of "TiH<sub>2</sub> + CNTs" composite obtained by mechanosynthesis (a) and Electron microscopic image of multilayer CNTs containing catalyst nanoparticles inside (b).



**Fig.5.** Dependence of electrical conductivity on the degree of compression for "TiH<sub>2</sub>+20 wt.% CNTs" (metal particle size 100-200 μm) (a) and the original CNTs sample (b).

Consequently, this work demonstrates that for such systems it is more effective to use the initial titanium hydride powder with a particle size of 100–200 μm, which provides an optimal combination of the properties of the carbon and metal components. The creation of a composite based on titanium hydride and carbon nanotubes is advisable to obtain a material with controlled electrical conductivity, enhanced mechanical and thermal properties, and the possibility of targeted formation of new phases. This approach allows the expansion of the functional capabilities of titanium hydride and opens up prospects for its application in electrically conductive composites, reactive powder materials, sensor systems, and technologies aimed at controlling the phase composition and structure of materials.

## Conclusion

The results show that the conductive properties of titanium hydride powder depend strongly on particle size and the degree of compaction. The most effective fraction for forming conductive contacts and achieving high electrical conductivity is 100–200 μm, owing to optimal packing density and the maximum area of interparticle contacts. Both smaller and larger particles exhibit lower

conductivity efficiency due to the influence of oxide films or insufficient contact density.

Adding 20 wt.% carbon nanotubes to TiH<sub>2</sub> results in the formation of a composite with controlled electrical conductivity, which differs from that of a simple mechanical mixture of components, indicating interfacial interactions. The obtained results confirm the feasibility of creating "TiH<sub>2</sub> + CNTs" composites and their potential use in functional conductive materials, particularly for cathodes of thermionic converters and reactive powder systems.

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## Вплив розмірного ефекту на властивості TiH<sub>2</sub>

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Композити на основі титану мають потенціал замінити традиційні матеріали в автомобільній, аерокосмічній та інших галузях промисловості. Цьому сприяють їхні чудові механічні, фізичні, теплові властивості, а також співвідношення міцності до ваги.

У представленій роботі досліджувався вплив розміру частинок гідрогенізованого титану на його провідні властивості. Насичення воднем проводилося за допомогою апарату Стівенсона, вміст водню становив 3,8%. Після цього вихідний зразок піддавався високоенергетичному подрібненню. Для подальших досліджень було визначено 3 основні розміри частинок: 100 мкм, 100-200 мкм, більше 200 мкм.

Резистометричні дослідження показали, що частинки розміром 100-200 мкм характеризуються на порядок вищою електропровідністю, ніж інші розміри частинок ( $8 \text{ (Om}\cdot\text{cm)}^{-1}$ ), що зумовлено максимальним контактом між сусідніми частинками та їх оптимальною геометрією при упаковці порошкового зразка. На основі цього розміру порошку методом механосинтезу було синтезовано наноккомпозит з вуглецевими наноструктурами, який характеризується на порядок вищою електропровідністю порівняно з вихідними компонентами, що вказує на можливість використання таких матеріалів в електротехнічній промисловості.

**Ключові слова:** TiH, порошок, електропровідність, композит.