Features of thermal decomposition of titanium hydride fraction

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Abstract. One of the areas of work on the technology of producing products based on titanium hydride fraction HTK-D is the study of methods and modes of modifying the surface of titanium hydride fraction in order to increase its thermal stability at high temperatures and protect titanium from oxidation. The research was carried out using modern instrumental physical methods and physical and chemical analyses. Differential thermal and thermogravimetric analyses of the initial and modified titanium hydride fractions were performed in the temperature range of 20-1000 °C in the air atmosphere. The curves TG and DTG of the initial fraction of titanium hydride show a slight change in mass in the temperature range of 20-100 °C with a maximum at T – 82 °C, which is associated with the removal of sorbed moisture. The data of the DTG analysis showed that the modification of the titanium hydride fraction with sodium borosilicate leads to a noticeable decrease in the mass loss of the samples: in the temperature range of 600-900 °C, the mass loss for the modified titanium hydride fraction was 0.39 and 1.58% (wt.) for the initial fraction of titanium hydride. In the course of the experiment, it was revealed that a further increase in the temperature leads to an increase in the mass of titanium hydride fraction samples, which is a consequence of titanium oxidation. It was found that the modification of the titanium hydride fraction with sodium borosilicate increases its thermal stability.

1. Introduction
Protection against neutron radiation in nuclear science and technology remains relevant today [1-4]. In order to meet the high requirements for neutron protection, there is a constant search for new materials that can meet them [5-8].

Titanium, like transition metals, interacts with hydrogen to form metal hydrides, which are dark powders characterized by electrical conductivity and magnetic properties typical of metals [9, 10]. The theory of the structure of such metal-like hydrides is still far from perfect. The fraction of titanium (II) hydride has a microporous structure [11]. When titanium (II) hydride is heated above 250 °C, significant internal stresses occur in its TiH₃ crystals due to different specific volumes of titanium metal and its hydride, as a result of which macro-cracks develop during heat treatment, which facilitates the desorption of hydrogen [12]. Currently, there is a growing interest in studying the general laws and properties of the titanium-hydrogen system, as the interaction of hydrogen with titanium in a wide temperature range is a particularly important problem in physical materials science [13, 14].
2. Materials and methods
Titanium hydride in the form of spherical granules (fractions) of 1-4 mm diameter of the HTK-D series, which was synthesized from sponge titanium of the TH-90 brand according to laboratory technology, was used for research. The density of the HTK products is 3800 kg/m³, the bulk density is 2526 kg/m³, the hydrogen content is 3.55 wt.%. Sodium methylsilicate: density is 1190 kg/m³, the silicon (Si) content is 4 wt.%, pH=12, and chemically pure boric acid (H₃BO₃) was used.

Differential thermal (DTA), thermogravimetric (TG) and differential thermogravimetric (DTG) analyses of samples were performed on a STA-449 F1 Jupiter derivatograph at a heating rate of 10°C/min in air, sample weight 0.2–0.9 g, sensitivity DTA-1/5, DTG-1/5, crucible – alundum, reference standard – corundum.

3. Results and discussions
One of the areas of work on the technology of producing products based on titanium hydride fraction HTK-D is the study of methods and modes of modifying the surface of titanium hydride fraction in order to increase its thermal stability at high temperatures and protect titanium from oxidation.

The curves TG and DTG of the initial fraction of titanium hydride show a slight change in mass in the temperature range of 20-100 °C with a maximum at T–82 °C, which is associated with the removal of sorbed moisture (Figure 1). The endothermic effect at 325 °C, both in the modified and in the initial fraction of titanium hydride, is caused by the structural-phase transformation in titanium hydride. This fact is indicated by the absence at a given temperature of the effect on the differential curve (DTG) - the mass loss of the sample. In the initial fraction of titanium hydride in the temperature range of 400-750 °C, an exothermic effect was observed, accompanied by an increase in the mass of the sample. This may be caused by the oxidation of part of the titanium. A similar effect is also observed on the modified fraction of titanium hydride in the specified temperature range. In the temperature range at 500 and 574 °C, endothermic effects were observed, presumably caused by the melting of the modifying cover on the fraction – sodium borosilicate, as no such thermal effects were found on the initial fraction of titanium hydride.

In a modified fraction of titanium hydride at a temperature of 807 °C, an intense endothermic effect was observed, accompanied by a loss of sample mass (Figure 2). A similar thermal effect in an unmodified fraction of titanium hydride manifests itself at a lower temperature of -757 °C, which is caused by the dissociation of titanium hydride. At a temperature of 882 °C, an endothermic effect was observed on the DTA curves, apparently caused by a polymorphic transition of a metal α-Ti with a HTP lattice (hexagonal tightly packed), the KK coefficient = 74% in β-Ti with a VCC lattice (volume-centered cubic) with a smaller atomic packing value (KK = 68%).

The exothermic effect in the temperature range of 890-1000 °C indicates the oxidation of titanium, which was facilitated by the polymorphic transition α-Ti → β-Ti.

The data of the DTG analysis showed that modifying the titanium hydride fraction with sodium borosilicate led to a noticeable decrease in the mass loss of samples in the temperature range of 600-900 °C, the mass loss for the modified titanium hydride fraction was 0.39 and 1.58 % (wt.) for the initial fraction of titanium hydride.

Based on the experimental data, it can be concluded that modifying the titanium hydride fraction with sodium borosilicate increases its thermal stability, and it can also be assumed that this is due to a decrease in the rate of redistribution of hydrogen atoms on the surface of the titanium hydride fraction.
Figure 1. Thermogram of the initial fraction of titanium hydride.
Figure 2. Thermogram of the modified fraction of titanium hydride.

Data on improving the thermal stability of the modified titanium hydride fraction are confirmed by thermogravimetric TG analysis after heat treatment for 100 hours (Figure 3). The process of titanium
oxidation occurred at a temperature above 500 °C, which led to an increase in the mass of samples of the initial fraction, and for samples of the modified fraction – at a temperature above 700 °C. The DTA data showed that a further increase in temperature leads to an increase in the mass of the titanium hydride fraction samples, which is a consequence of titanium oxidation.

![Figure 3](image)

**Figure 3.** Change in the mass of the titanium hydride fraction during heat treatment for 100 h: 1 – initial, 2 – modified.

4. Summary
According to the data of the DTG analysis, it was found that the modification of the titanium hydride fraction with sodium borosilicate led to a noticeable decrease in the mass loss of the samples: in the temperature range of 600-900 °C, the mass loss for the modified titanium hydride fraction was 0.39 and 1.58 % (wt.) for the initial fraction of titanium hydride. It was found that modifying the titanium hydride fraction with sodium borosilicate increases its thermal stability.

5. References
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