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Research of Hydrogen Absorption-Desorption by Ti-Al-Nb Alloy

Kurbanbekov ShR[1], SkakovMK[1], KarakozovBK[1] and BakhtibayevAN[2]

[1]*Institute of Atomic Energy, Republic of Kazakhstan*

[2]*Kh.A. Yassawi International Kazakh-Turkish University, Republic of Kazakhstan*

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***Corresponding author:** Kurbanbekov ShR, Institute of Atomic Energy, Kurchatov, Republic of Kazakhstan, Email: sherzod05_88@mail.ru

Abstract

The paper presents kinetics of hydrogen sorption of Ti-23.5at%Al-21.5at%Nb alloys in isothermal conditions underthe temperature of 450, 500 and 550 °C. It was determined that maximum quantity of absorbed hydrogen is observed at material of sorbed alloy under the temperature of 550 °C. That presents approximately 0.289mass%. It had been found that hydrogen is released under the temperatures within 700 ...790 °C. In addition, it was revealed that maximum release of hydrogen composes 85% of samples saturated under the temperature of 550 °C.

Keywords: Intermetallic compound; Absorption-desorption; Hydrogen; Plasticity; Crucible

Introduction

As is known, to find a safe method for reversible hydrogen storage is currently one of the important issues in the field of hydrogen energetics. Storage of the hydrogen in various hydrides of metals and alloys is one of the advanced methods to solve this issue [1]. Application of alloys for hydrogen storage and its use depends on several tasks, which focused on increasing of sorption properties and cyclic stability of alloys. Ti-Al alloys are one of the efficient materials for storage of hydrogen [2,3]. Using of alloys to storage hydrogen and its use depends on several tasks, which are to increase the sorption properties and cyclic stability of alloys. Alloys based on Ti-Al are one of the most effective materials for hydrogen storage [2,3]. It is known that, additional introduction of niobium into the Ti-Al system significantly increases the plasticity of Ti_3Al, intermetallic which can be explained by a decrease in the degree of ordering and decrease in the share of covalent bond [4]. Also, additional introduction of niobium into the Ti-Al system [5,6] leads to an increase in its absorption-desorption properties of hydrogen due to the formation of nanoscale phases having less dense packaging compared to the face centered close-packed lattice of Ti_3Al.

The purpose of this paper is to determine the optimal absorption-desorption temperatures of hydrogen to the sample materials based on Ti-Al-Nb system and to study the changes in its structural-phase state.

Materials and Methods of Research

Ti (99.9%), Nb (99.96%) and Al (99.98) powders were used as initial raw materials for producing Ti-Al-Nb-composite.

Technology of sparkplasma sintering (SPS-technology) of powder mixtures was used to create compact samples based on intermetallic Ti-Al-Nb system. Sintering of powder mixtures was conducted on a special facility Labox-1575. Research of hydrogen sorption kinetics by intermetallic compounds of Ti-23.5at%Al-21at%Nb system was conducted on an experimental facility VIKA [7] under the temperatures of 450, 500 and 550°C. The facility consists of a working chamber, pumping system and information-measuring system (IMS). Differential pumping system including forevacuum pump NVR-5DM with a nitrogen trap and two magnetic discharge pumps NORD-100 and NORD-250 was used to ensure the required pressure in the working chamber of the facility. Forevacuum pump is used to pre-pumping of gases from the working chamber after loading the sample into the crucible, magnetic discharge pump NORD-250 is used for pumping the working chamber and the measuring path in the annealing process after loading samples, the pump NORD-100 is used to create high vacuum in the chamber and the measuring part of the experimental facility during the experiment. The experiments consisted the following: Ti-23.5at%Al-21at. %Nb sample was loaded in a special ampoule device (AD). After loading of the sample, the high–temperature decontamination of AD cell with the samples was conducted for 30 minutes at a temperature of 800-850°C and a constant pumping of the AD volume by a turbomolecular pump were conducted. Then the body of the AD experimental cell was cooled down to the studied temperature (the temperature of hydrogen saturation) and spectrally pure hydrogen was injected with samples to a given pressure in the volume of AD. Further, pressure change in the AD volume with studied samples was recorded under the preset saturation temperature using a deformation pressure sensor. After that, the heating of ampoule device with samples was stopped, and the samples were cooled in the hydrogen atmosphere to room temperature. After 12 hours, samples were heated again to a preset saturation temperature and kept under this temperature shelf for 15-20 minutes, after which samples were cooled to room temperature, and remaining hydrogen was pumped from the volume of the ampoule device.

Research Results and Discussion

The main criteria that determine the prospects of application of those or other materials for storing hydrogen, typically consider the amount of their sorption capacity, operating temperature and pressure, kinetics of the interaction [8]. Curves of hydrogen sorption by Ti-23.5at.%Al-21at%Nb alloy under temperatures of 450, 500 and 550°C and a pressure of 41 Torr (Figure 1) were presented to compare processes of sorption isotherms. Figure shows that under the increase of temperature from 450°C to 550 °C, an increase in the rate of hydrogen sorption occurs and respectively, the change in pressure of the ampoule (Figure 1a) is observed. Figure 1b shows the mass fraction of hydrogen absorbed by the sorbent at temperatures of 450-550 °C.Figure 1b shows that there is an intensive absorption of hydrogen at a temperature of 550 °C, the proportion of hydrogen absorption reaches up to 0.289 mass.%. Probably, the interaction of Ti_2AlNb phases with hydrogen occurs firstly, traces of which are present in the samples, thus the activation barrier of the reaction of the material main phases is decreased.

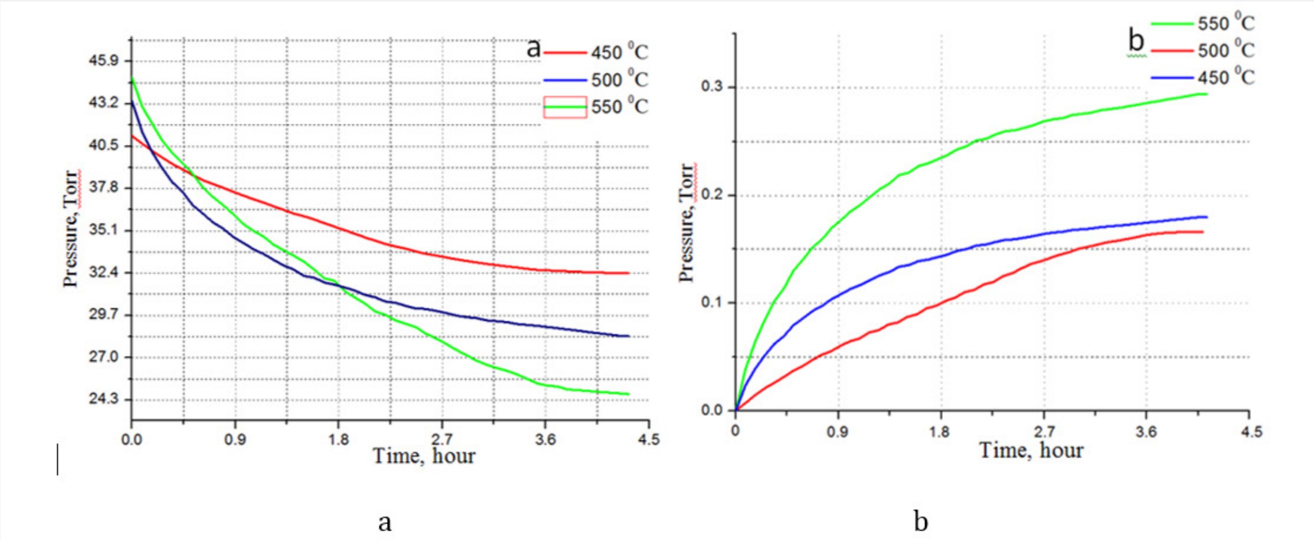

Figure1: Hydrogen adsorption isotherm with Ti-23,5at.%Al-21at.%Nb alloy, saturated under the temperatures of 450, 500 and 550 °C) a) pressure dependence from temperature b) change in the mass fraction of hydrogen in the Ti-23,5at.%Al-21at.%Nb alloy.

The ampoule device was annealed at a temperature of 900 °C for 30 minutes, before the experiment of desorption with an empty ampoule device. Argon was injected to one atmosphere after walls of the ampoule device cooled to a temperature of 20 °C in the volume of the ampoule device, then the ampoule device was closed, and the ampoule volume was pumped to a pressure of 10-4Torr, after which the ampoule device was tested for tightness using RGA-100 quadrupole mass spectrometer and the helium. Further, the desorption process of hydrogen by Ti-23.5at%Al-21at%Nb alloy was conducted. The heating was from 20 to 790 °C.

In the result of conducted experiments, the dependence of the hydrogen pressure from the sample temperature at an increase up to 790 °C (Figure 2) was obtained. Results of the study of Ti-23.5at%Al-21at%Nb alloy desorption showed that hydrogen release was observed in the temperature range of 700 ... 790 °C. Maximum hydrogen content in the sample saturated at 550 °C was 0.289 mass.%. Figure 2b shows that the hydrogen release from a sample saturated at a temperature of 550 °C reaches up to 85%.The active yield of hydrogen is observed at a temperature of 750 °C.

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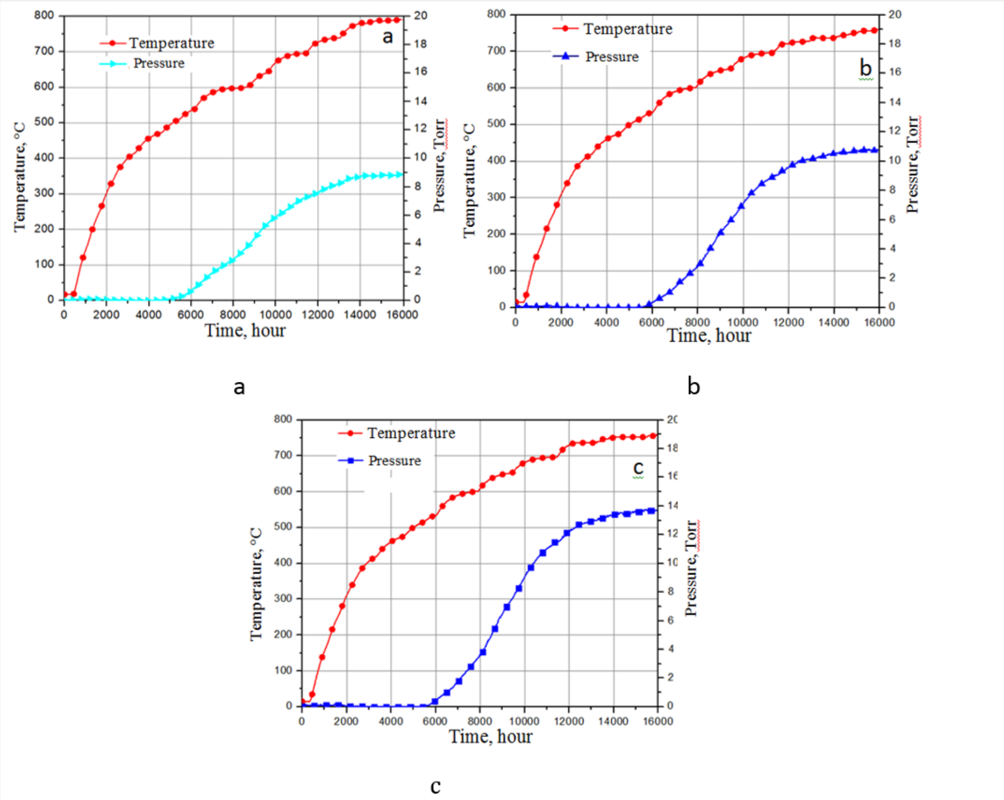

Figure 2: Curves of hydrogen desorption with Ti-23.5at.%Al-21at.%Nb alloy under heating temperatures of 750-790 °C, absorbed at temperatures: a) 450°C, b) 500°C, c) 550 °C.

The paper [9] presents the dependence of the desorption pressure for some systems, which shows that hydrides based on alloys of intermetallic compounds can be used to accumulate hydrogen in a fairly wide range of temperatures and pressures. The main factor limiting the rate of hydrogen release and absorption by the accumulator, in most practically important cases, is the heat and mass transfer in the layers of intermetallic particles, and not the sorption-desorption kinetics on individual particles [10].

The results of the study of hydrogen-adsorption properties showed, that the pressure of hydrogen desorption increases sharply at 500 °C. Thus, Ti-23.5at%Al-21at%Nb alloy is a high temperature getter. Results of the study of hydrogen desorption are presented in Table 1.

Table 1: Results of hydrogen sorption/desorption of Ti-23,5at%Al-21at%Nb alloy.

No	Temperature °C of Sorption	Mass.% Sorption of Hydrogen	Temperature °C of Desorption	Mass.% Desorption of Hydrogen
1	450	0.133	790	0.112
2	500	0.162	750	0.137
3	550	0.289	750	0.261

Thus, it was found that the rate of sorption/desorption of hydrogen depends on the heating temperature. It is also important to note that the orthorhombic phase of Ti_2AlNb is a well hydrogen absorber. This is confirmed by the absorption of hydrogen at a sufficiently low pressure (about 45 Torr.), and can be explained by the acceleration of diffusion in the Ti-Al system by doped Nb.

Conclusion

Based on the analysis of the results, following conclusions can be made:

a) The kinetics of hydrogen sorption by Ti-23.5at%Al-21.5at%Nb at isothermal conditions under temperatures of 450, 500 and 550 °C is studied. The dependence of the mass fraction of hydrogen in the material samples from temperature was obtained.It is determined that the maximum amount of about 0.289 mass% of absorbed hydrogen is observed in the material sorbed at a temperature of 550 °C;

b) It had been found that hydrogen is released under the temperatures within 700 ...790 °C. At the same time, the chemical composition of the material samples practically

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does not affect the temperature modes of hydrogen release. It was found that the maximum 85% of hydrogen release is observed in saturated at a temperature of 550 °C.

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