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Experimental Investigations of Hydrogen Purification by Purging Through Metal Hydride

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In an experimental stand [1] for investigation of properties of hydrogen accumulating the materials investigated a new type of reactor cleaning and storage of hydrogen.

The applicability of hydrogen purging through metal hydride beds for the purification from non-poisoning admixtures is studied experimentally. The main characteristics of the process together with the main technical barriers of the proposed technology are defined.

Specially designed stainless steel continuous flow reactor filled with $LaFe_{0.1}Mn_{0.3}Ni_{4.8}$ intermetallic compound is tested at variable inlet hydrogen/inert gas composition with measuring mass flow, pressure, temperature and hydrogen content at the outlet both for charging and discharging mode. The estimations of hydrogen losses and purification capacity show certain advantages of the studied technology in comparison with PSA-like mode [1], especially from the point of view of operation regime simplification. The evident process slow-down observed in the experiment is connected with saturation of metal hydride porous bed by hydrogen and with temperature increase due to high thermal effect at sorption (~ 40 kJ/mole H2). The ways for heat and mass transfer optimization together with the range of applicability of the method for fine hydrogen purification are described and discussed.

Keywords: Hydrogen, Hydrogen storage, Purification, Metal hydrides, Intermetallide.

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1. INTRODUCTION

Traditional for hydrogen storage (compressed gas and liquid) have numerous disadvantages making their use impossible in some cases [2].

Metal hydride hydrogen storage is free of these disadvantages [3].

For effective treatment of hydrogen by the usy of cyclic reactors [1] is a complicated problem having some disadvantages:

- the need for big (bulky) valve assemblies;
- the need to regulate the process of discharge of impurities from a free volume metal hydride reactors;
- great loss of hydrogen (20%), depending on the required purity of the product [1];
- thermal losses due to cycling heating/cooling of the reactors.

The method presented helps to simplify the technology of hydrogen purification, get rid of the cyclic removal of impurities from the free volume of reactor, thus reducing the amount of valves in the system and thereby reduce the capital cost of metal-hydride storage and purification systems.

2. THE EXPERIMENTAL TECHNIQUE AND THE OBJECT OF INVESTIGATION

2.1 The experimental reactor

Experimental reactor (Fig. **1**) was charged with 1629,5 g hydrogen absorbing material $LaFe_{0.1}Mn_{0.3}Ni_{4.8}$. The choice of $LaFe_{0.1}Mn_{0.3}Ni_{4.8}$ alloy is due to comparatively high mass content of in hydride (~ 1,3 mass.%), high desorption pressure (Pdes= 0,45 ÷ 0,60 MPa) in the working temperature interval (80 ÷ 100 °C) and resistance to CO_2 admixtures [2-5].

The reactor is a vertical cylinder which is placed inside the alloy $LaFe_{0.1}Mn_{0.3}Ni_{4.8}$, welded covers with two sides. Gas inlet at the top, output - from below of the reactor. Intermetallic compound of a multilayer filters is limited to steel grid with a typical size of 3 mm to avoid the metal hydrides particles entrainment to the piping.

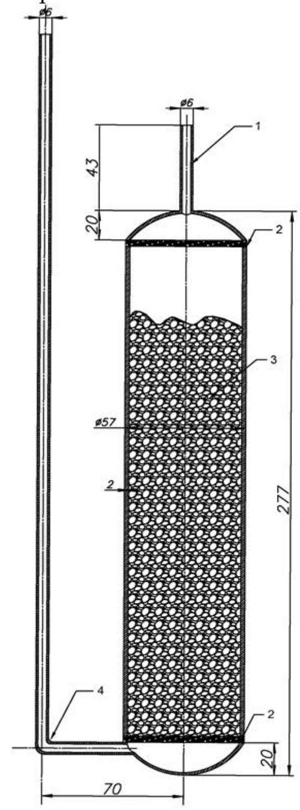

Fig. 1 – Reactor of hydrogen purification by purging through metal hydride. 1 – inlet pipe, 2 – micron grid, 3 – metal hydride bed, 4 – outlet pipe

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2.2 The experimental apparatus

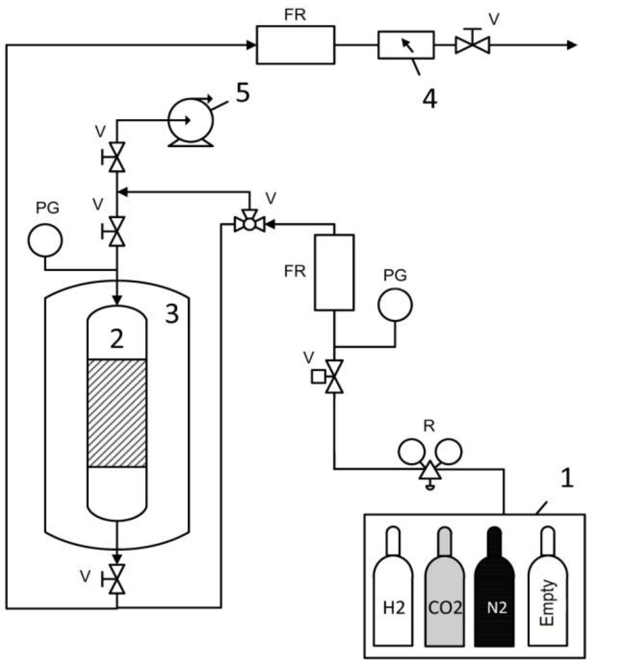

Fig. 2 – Scheme of the experimental section: 1 - ramp for the preparation of the gas mixture, 2 - Experimental Reactor, 3 - liquid thermostat, 4 - flow analyzer, 5 - vacuum pump, R - reducer, PG - pressure gauge, V - valve, FR - regulator of the gas flow rate

Experimental investigations of gaseous hydrogen [4] purification with a flow-type reactor were carried out at the experimental section (Fig. 2), a complex experimental stand [1] at JIHT RAS, which consists of a ramp for the preparation of the gas mixture (1), of the investigated experimental reactor RHOP-1 (2) placed in a liquid thermostat (3), the flow analyzer module (4), a vacuum pump (5), gas flow controllers (FR), valves (V), reducer (R) and pressure sensors (PG).

2.3 The experimental technique

The nitrogen-hydrogen mixtures of various concentration were prepared for the experimental investigations. The mixture was fed through the upper pipe (P_{in} = 0,5 MPa, see Fig. 3) while reactor was placed in thermal bath Julabo FPW50-HE which maintained 0˚C, the sorption reaction temperature. The flow at the inlet and outlet was limited by flow controller (Fig. 4). The outlet flow was organized in the following way that the maximal amount was absorbed in metal hydride bed and depleted mixture was obtained at the outlet. The mixture was analyzed in gas analyzer to monitor the hydrogen content at the outlet with flow rate 1.1 st. l/min . At reaching high outlet hydrogen content values (~ 90% for mixture 1.5% N_2, ~ 60% for 13% N_2 mixture and 27% N_2 mixture, which corresponds to hydrogen mass content in metal hydride ~ 1.2% mass.) the value of bath temperature was changed to 100°C for the start of reactor discharge process with hydrogen exhaust to the atmosphere. The discharge was conducted till the full stop of desorption.

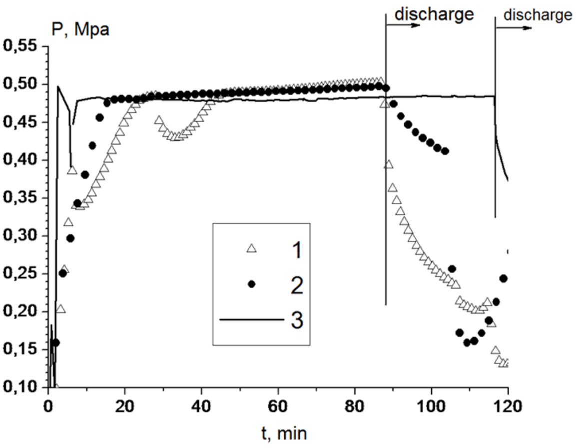

Fig. 3 – The absolute input pressure in a reactor while charging. 1 - 1,5%N_2, 2 – 13%N_2, 3 – 27%N_2

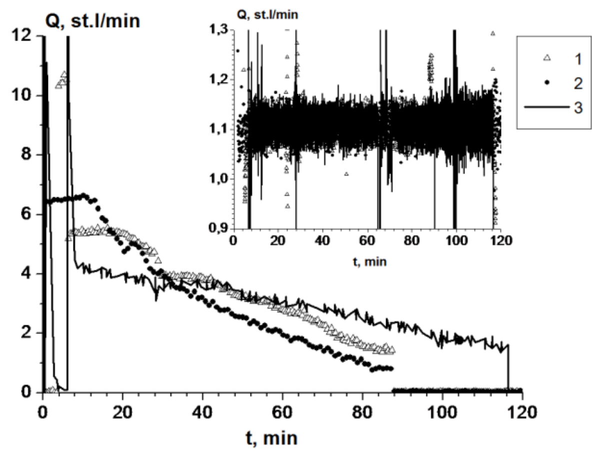

Fig. 4 – Flow rate of mixture at the reactor inlet and outlet, depending on the impurity content in the initial mixture while charging. 1 – 1,5%N_2, 2 – 13%N_2, 3 – 27%N_2

3. RESULTS AND DISCUSSION

One of the main characteristics of MH devices is the dynamics of hydrogen sorption i.e., the dependence of the mass content of hydrogen in metal hydrides on time (Fig. 5). The data show that the velocity sorption by increasing impurities in the initial gas is falling.

The initial period of purification is characterized by zero hydrogen content at the outlet of reactor. This fact is connected with sorption of the entire hydrogen fed to the reactor with admixture removed from reactor. For all the compositions of initial mixtures the interval of zero outlet hydrogen content (Fig. 6) is different: for mixture 1.5% N_2 the interval is 0.05% mass. H_2, for 13% N_2 – 0,5% mass, for 27% N_2 – 0,65% mass.

Experimental investigations of purging method showed the good comparative efficiency of purification at lower costs for exhaust organization (Fig. 7). The relative losses of hydrogen at the considered method account for 36%, 19% and 4% for mixtures 1.5% N_2, 13% N_2 и 27% N_2 correspondingly (for the charge of reactor hydrogen mass content was ~ 1,2% mass.).

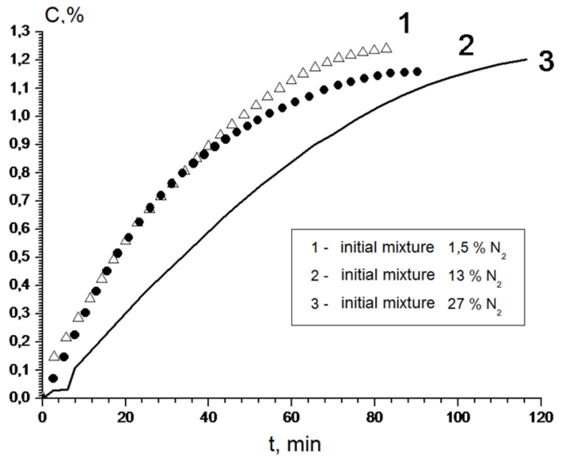

Fig. 5 – Dynamics of hydrogen sorption at various purity of hydrogen 1 – 1,5%N_2, 2 – 13%N_2, 3 – 27%N_2

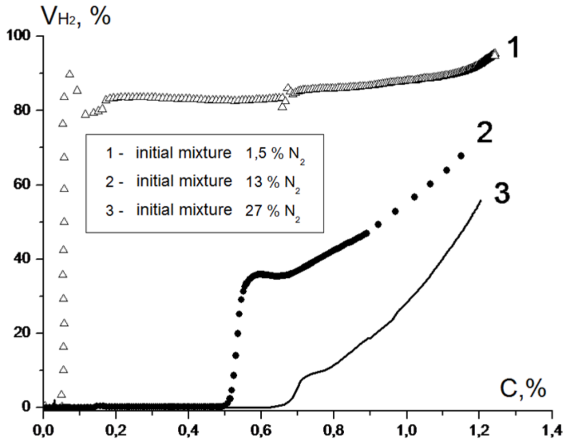

Fig. 6 – The dependence of volumetric concentration of hydrogen at the outlet of the reactor and the mass concentration of hydrogen in a solid phase for various degrees purity of hydrogen 1 – 1,5%N_2, 2 – 13%N_2, 3 – 27%N_2

4. CONCLUSION

1. The developed experimental reactor for purging method RHOP - 1 showed good performance and perspective of the method usage for hydrogen purification by purging through the metal hydride bed at relatively low hydrogen losses. The cascade of reactors could lower the hydrogen losses at the selected technology.

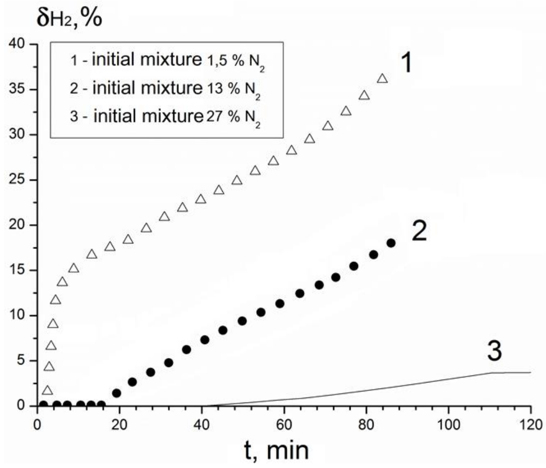

Fig. 7 – Integral losses of hydrogen relatively summary amount of purified hydrogen (accumulated) in charge the reactor for various degrees purity of hydrogen 1 – 1,5%N_2, 2 – 13%N_2, 3 – 27%N_2

2. The conducted experimental investigations demonstrated advanced efficiencies for higher values of admixture content (>10% vol.), than at pressure cycling method.

3. In spite of evident advantages of purging method, there are many scientific and technical problems to solve, especially connected with the arranging heat and mass transfer inside the reactor. Due to low effective thermal conductivity of hydrogen absorbing material (lower than 1 W/m K) and big volumetric thermal effect of the reaction (35 kJ/mole H2) the modernization of the design is needed with respect to heat transfer intensification. The optimization could include the minimization of the metal hydride layer and the increase of the area of the heat transfer between the intermetallic alloy and cooling agent.

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REFERENCES

1. V.I. Borzenko, D.V. Blinov, D.O. Dunikov, S.P. Malyshenko, *18th World Hydrogen Energy Conference* 2010, CD, Essen, Germany, May 16-21, 2010.
2. *Atomic – hydrogen energy and technology*. Vol.8. Energoatomizdat, 1988.-272 pp. (In Russian).
3. B.P. Tarasov, M.V. Lototsky, V.A. Yartys, *Russ. Chem. Journ.* L No6, 5 (2006) (in Russian).
4. D.V. Blinov, V.I. Borzenko, D.O. Dunikov, S.P. Malyshenko, *Vestnik MPEI*, №2, 44 (2012).