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Equipment for Measurement of Hydrogen Desorption Isotherms of Different Scale Samples

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This research is devoted to experimental investigation of influence of a system scale on hydrogen desorption characteristics in metal hydride (scale effect) by new high accuracy set-up US150. We suppose that changing of the alloy cell volume during hydrogen sorption or desorption lead to stress state of the alloy/hydride lattice. Inside a metal hydride bed a reciprocal influence of particles inhibits the lattice relaxation. These processes change thermodynamic characteristics of hydrogen sorption and desorption.

Hydrogen desorption characteristics of $LaFe_{0.1}Mn_{0.3}Ni_{4.8}$ alloy are measured for two samples (100 and 500 g) by the Sieverts method. Evident influence of sample's size on desorption characteristics is discovered. The experimental data allows us to suppose that sample scale has influence on properties of hydrogen solid solution phase.

Keywords: Scale effect, Desorption isotherms, Hydrogen.

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1. INTRODUCTION

Recently most of the research activities in field of hydrogen technologies are directed to improvement of hydrogen storage material characteristics (capacity, operation temperature, pressure, etc.). Much less research and papers are devoted to problems appear during design of hydrogen energy systems. Most of widely-distributed techniques of hydrogen storage materials properties investigation based on testing of small-scale samples (less than 10 g) but device for hydrogen energy system must contain kilograms of storage material. Authors suppose that properties of LaNi5-based storage materials depend on it scale and geometry. This research is devoted to experimental investigation of hydrogen-alloy system scale influence on hydrogen sorption characteristics (scale effect) by new high accuracy set-up US150.

2. EXPERIMENTAL DETAILS

The research of hydrogen sorption characteristics was conducted by measurement of hydrogen desorption isotherms of different scale samples (100 and 500 g) according to the Sieverts method. Unique experimental set-up US150 for isotherm measurements was designed for the research (Fig.1). This equipment allows to investigate hydrogen sorption and desorption with high-accuracy for samples since 10 up to 800 g mass in wide condition range (temperature: -30°C – 400°C, maximal pressure: 150 atm).

It is necessary to wait for a long time to set up equilibrium in alloy-hydrogen system for big samples. For 100 g sample point equilibrium time is 4 hours per point for 500 g sample it is 24 and 48 hours per point. Such long times are chosen to fully eliminate influence of kinetic factors on desorption process and inspect existence of leakage in hydrogen infrastructure. US 150 allows making uninterrupted experiment during 60 days. As it is becomes obvious that 24 h/p is enough for equilibrium setting up, so at 150 °C desorption isotherm for 500 g sample was measured with 24 hours point equilibrium time. Fractional uncertainty of the isotherm measurements are about 1% for 500 g sample and 5% for 100 g sample.

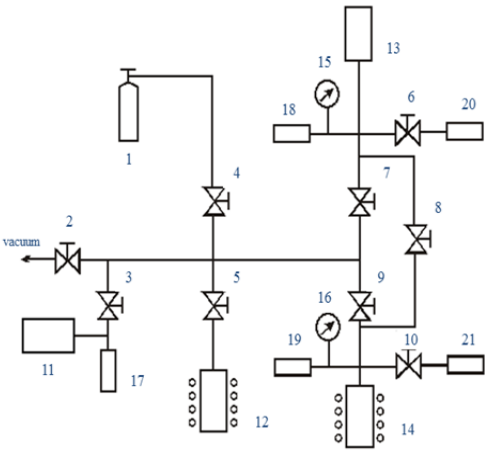

Fig. 1 – Facility for hydrogen desorption isotherms measurement. 1 - balloon with hydrogen; 2-10 - valves; 11 - vacuum tank; 12 - hydrogen accumulator with LaNi5; 13 - buffer tank, 14 – work autoclave; 17 - pressure sensor (0-1 bar); 18, 19 - pressure sensors (0-150 bar); 20, 21 - pressure sensors (0-10 bar)

3. RESULTS AND DISCUSSION

Desorption isotherms of 100 g sample are shown in Fig.2. Desorption isotherms of 500 g sample are shown in Fig.3

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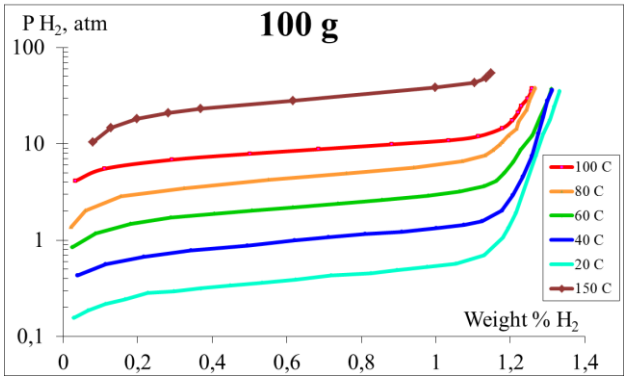


Fig. 2 – Desorption isotherms of 100 g sample of LaFe0.1Mn0.3Ni4.8 alloy at 20, 40, 60, 80, 100 and 150 °C

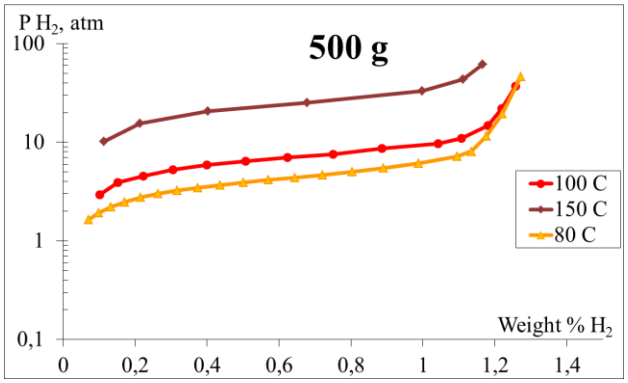


Fig. 3 – Desorption isotherms of 500 g sample of LaFe0.1Mn0.3Ni4.8 alloy at 80, 100 and 150 °C

From the experimental data enthalpy and entropy of the desorption process are calculated by the van't Hoff equation. For 100 g sample they are: $\Delta H_{des.}$= 34,3 kJ/mol, $\Delta S_{des.}$= 109,3 J/mol·K

For 500 g sample they are: $\Delta H_{des.}$= 31,0 kJ/mol, $\Delta S_{des.}$= 99,5 J/mol·K.

Comparison between desorption isotherms of 100 g and 500 g sample at 100 °C is shown in fig. 4. a). Fig. 4. b) demonstrate difference in equilibrium desorption pressure of hydrogen. It is seen that desorption of hydrogen by 500 g sample occurs at lower equilibrium pressure of hydrogen. Difference in hydrogen pressure weakly depends on weight % of hydrogen in the desorption plateau range. Isotherms with different point equilibrium time are the same. Thus we can conclude that kinetic factors do not influence on alloy behavior and there was no considerable leakage in hydrogen infrastructure during experiment.

Fig. 5. shows difference in desorption behavior of 100 g and 500 g samples at 150 °C.

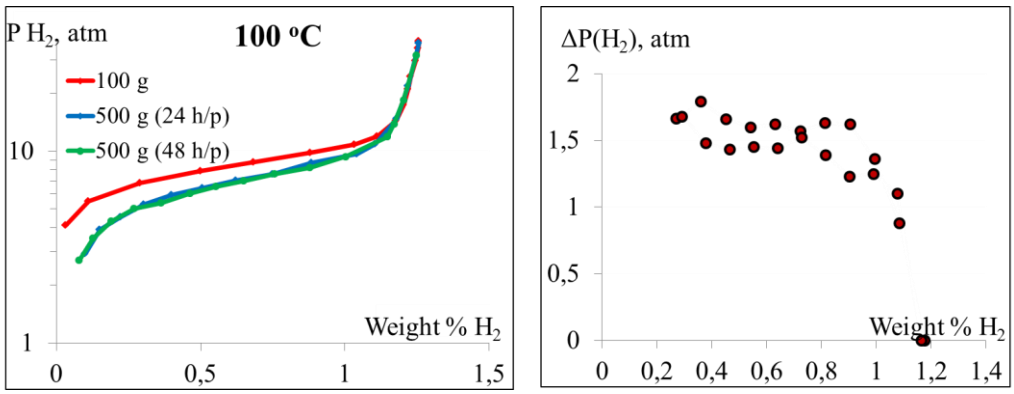
It is necessary to note that in hydride phase area isotherms of different samples consider with each other. Thus scale effect occurs during hydrogen solid state solution phase existence.

4. CONCLUSIONS

Scale effect in LaFe0.1Mn0.3Ni4.8 is experimentally discovered. Hydrogen desorption equilibrium pressure for the 500 g sample is lower than for the 100 g sample. Furthermore van't Hoff's $\Delta H_{des.}$ and $\Delta S_{des.}$ decrease with increasing of sample scale.

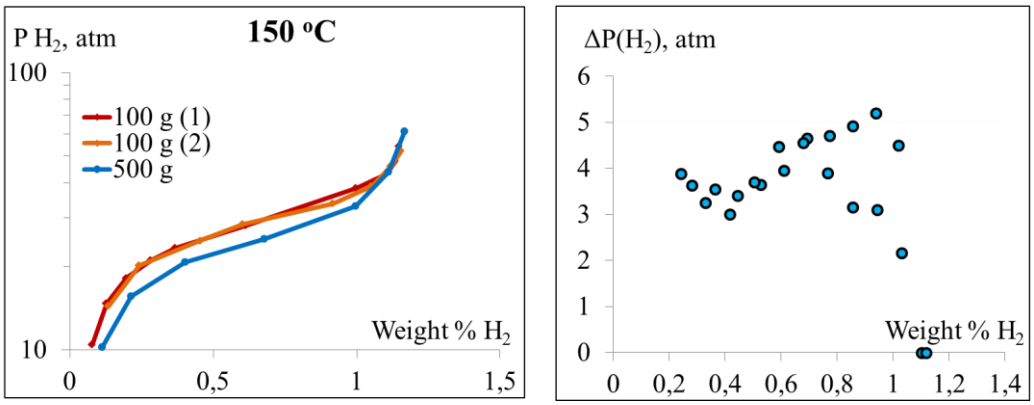
Measurement of desorption isotherms with different point equilibrium time (24 and 48 hours per point) shows that kinetic factors do not effect on 500 g sample behavior in our experiments.

The experimental data allows us to suppose that changing of sample scale has influence on properties of hydrogen solid solution phase.



a b

Fig. 4 – Comparison between desorption isotherms of 100 g and 500 g sample at 100 °C (a); difference in equilibrium desorption pressure of hydrogen (b)



a b

Fig. 5 – Comparison between desorption isotherms of 100 g and 500 g sample at 150 °C (a); difference in equilibrium desorption pressure of hydrogen (b)