Performance of containers with hydrogen storage alloys for hydrogen compression in heat treatment facilities

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ABSTRACT

Containers tested in this study constitute the main part of a hydrogen compressor model designed to be used with a furnace for heat treatment. The paper presents the results of hydrogen absorption and desorption for containers with $LaNi_{4.8}Sn_{0.2}$ (45 kg) and $LaNi_{4.25}Al_{0.75}$ (3.5 kg) hydrogen storage alloys. Containers with $LaNi_{4.8}Sn_{0.2}$ and $LaNi_{4.25}Al_{0.75}$ were working within 0.15–3.3 MPa and 0.013–0.22 MPa hydrogen pressure ranges, respectively. We present the data on absorption and desorption kinetics, temperature changes of the alloys during the process and absorbed hydrogen quantities. The tests revealed a decrease in hydrogen storage ability of the containers after 53 ($LaNi_{4.8}Sn_{0.2}$) and 42 ($LaNi_{4.25}Al_{0.75}$) absorption–desorption cycles. The experiment involving hydrogen absorption from hydrogen–nitrogen mixture showed a significant adverse effect of the presence of nitrogen in the mixture on hydrogen storage ability of $LaNi_{4.8}Sn_{0.2}$ alloy.

1. Introduction

Reversible hydrogen storage properties of metal hydrides make them attractive candidates for use in a variety of applications e.g. hydrogen storage or hydrogen compression [1–6]. Hydride compressors presented in the literature are able to compress hydrogen in a wide pressure range where the maximum pressure of several dozen MPa could be achieved. Recent publications present compressors using AB_5 type alloys able to work in different pressure ranges such as: 0.062–3.5 MPa (293–353 K) [3], 0.575–2.1 MPa (298–363 K) [4] and 4–70 MPa (heating to 423 K) [5]. One of the main features of metal hydrides is heat release during absorption of hydrogen and absorbing heat during desorption. This, and the fact that alloys are usually in the form of powder characterized by low value of heat transfer coefficient, cause that their application faces the problems of assuring effective heat and mass transfer to the alloy bed [7–12]. The common solutions for heat transfer improvement inside the containers with alloys include: the use of comprehensive heat exchangers [8], Al and Cu foams [9,10], pressing an alloy together with another material characterized by high thermal conductivity (Al, graphite) [10] or covering the powder with Cu [11]. Porous filter tubes (pore size 1–5 μm) are used to assure fast hydrogen flow to the alloy bed [10]. When a large amount of a metal hydride is used, the problem of fast and

full charging becomes more pronounced – the container with an alloy has to be equipped with more heat exchangers and filter tubes. The design of the container with metal hydride for industrial application should also be simple enough to make the use of a hydride-based compressor economically justified.

There are various reasons for using gas (hydrogen in particular) as a quenchant instead of oil (or another liquid) but the main ones include environmental and product quality benefits [13–15]. In the case of hydrogen, high quenching rate is also an advantage. Steel quenching in hydrogen is characterized by the highest value of heat transfer coefficient in comparison with other gases (almost two-fold higher than for nitrogen) [16,17]. A mixture of hydrogen and nitrogen (80% H_2 and 20% N_2 – optimal concentration) is also known to have even superior heat transfer potential and allow to improve quenching rate by 30–40% (in comparison to pure hydrogen) [16]. Therefore, the industrial hydride compressor should be able to work with hydrogen as well as with hydrogen–nitrogen mixture.

The compressor model used in the experiments presented in this paper is a prototype of the industrial hydride compressor which could be used in a hydrogen recycling system connected to the hardening furnace. In such a furnace, hydrogen will be used for quenching. The hydride compressor is considered to be a device which permits safe hydrogen compression, which could also be interesting from the economical point of view. The study is a continuation of experiments presented in [18]. Its purpose was to test the suitability of two types of containers with two alloys: $LaNi_{4.8}Sn_{0.2}$ and $LaNi_{4.25}Al_{0.75}$ for hydrogen recycling in heat treatment facilities.

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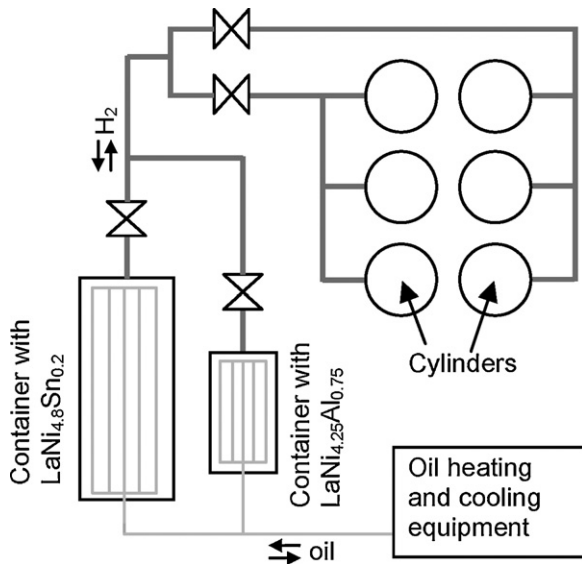
Fig. 1. Schematic view of the experimental installation.

2. Experimental details

The experiments were carried out using the installation whose simplified scheme is presented in Fig. 1. This installation, a model of the industrial compressor, works in 0.02–3.3 MPa pressure range and uses alloys: $LaNi_{4.8}Sn_{0.2}$ and $LaNi_{4.25}Al_{0.75}$ (in powder form) for hydrogen compression. The installation consists of the following parts:

- two containers for alloys: container no. 1 with $LaNi_{4.8}Sn_{0.2}$ and container no. 2 with $LaNi_{4.25}Al_{0.75}$ (Fig. 2 and Table 1),
- six cylinders that imitate the volumes of a furnace chamber and buffer container,
- heating–cooling equipment which uses oil as a heat transfer medium (oil was cooled by water or heated by electrical heaters mounted inside the oil container).

Inside the containers with alloys, heat exchangers and filter tubes (hydrogen inlet/outlet) were mounted. Finned tubes for $LaNi_{4.8}Sn_{0.2}$ (distance between fins: 1.96 mm) and smooth-walled tubes for $LaNi_{4.25}Al_{0.75}$ were used as heat exchangers (Fig. 3). The containers were also equipped with heating/cooling oil mantle. In each container, one porous filter tube with pores diameter not exceeding 0.5 μm was mounted. The thermocouples were mounted inside the alloy bed (one in each container).

Using this installation, 53 cycles of absorption–desorption for $LaNi_{4.8}Sn_{0.2}$ and 42 cycles for $LaNi_{4.25}Al_{0.75}$ were carried out. For $LaNi_{4.8}Sn_{0.2}$, hydrogen was absorbed from 6 cylinders and desorbed to 3 cylinders. During hydrogen desorption from $LaNi_{4.8}Sn_{0.2}$, the heating equipment was set for 433, 438 or 450 K. In the case of $LaNi_{4.25}Al_{0.75}$, only 3 cylinders were used for both absorption and desorption (set point of heaters for desorption: 413 K). It should be mentioned that the quantity of $LaNi_{4.25}Al_{0.75}$ was proportionally too small in comparison with $LaNi_{4.8}Sn_{0.2}$. To keep the mass proportion of alloys, which is needed in a compressor working with a

Fig. 2. Container no. 1 (on the right side) and container no. 2.
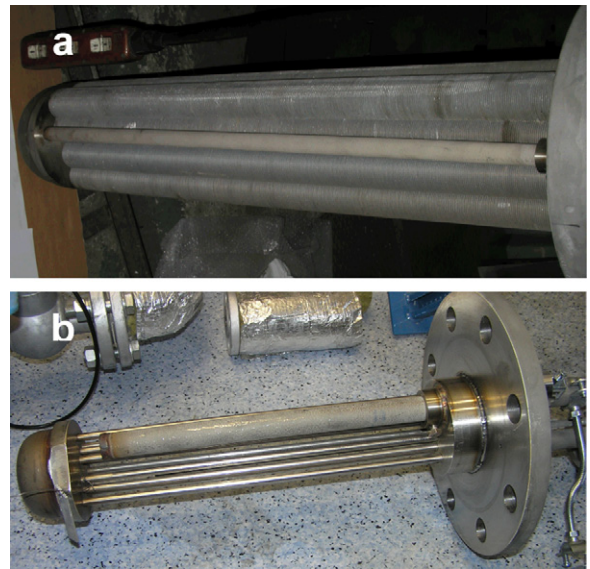

Fig. 3. Internal view of: (a) container no. 1 – finned tubes and a porous filter tube and (b) container no. 2 – smooth-walled tubes and a porous filter tube.

furnace, 7 kg of $LaNi_{4.25}Al_{0.75}$ should be used. Smaller quantity of $LaNi_{4.25}Al_{0.75}$ was the reason for using only 3 cylinders for $LaNi_{4.25}Al_{0.75}$ and it made the function of the experimental installation slightly more complicated.

Desorptions for $LaNi_{4.8}Sn_{0.2}$ and $LaNi_{4.25}Al_{0.75}$ were conducted in different ways. For $LaNi_{4.25}Al_{0.75}$ the whole process was divided into three parts: oil preheating, alloy heating and desorption. In case of $LaNi_{4.8}Sn_{0.2}$, desorption was conducted immediately after oil preheating. In the course of desorptions, the power of the heaters was 6 kW except for desorption no. 25 for $LaNi_{4.25}Al_{0.75}$ when half of the power was used. To change slightly the work conditions of alloys, the hydrogen amount in the installation during $LaNi_{4.25}Al_{0.75}$ cycling was decreased twice by 6%, first time after cycle no. 20 and second time after cycle 26.

The whole compression cycle, using both alloys working together, the main stages of which were: (1) absorption of hydrogen by $LaNi_{4.8}Sn_{0.2}$ (from 6 cylinders), (2) absorption of the rest of hydrogen by $LaNi_{4.25}Al_{0.75}$ (3 cylinders), (3) transfer of hydrogen from $LaNi_{4.25}Al_{0.75}$ to $LaNi_{4.8}Sn_{0.2}$ and (4) desorption from $LaNi_{4.8}Sn_{0.2}$ (to 3 cylinders) was also performed (after the last stage, the pressure of hydrogen was equalized in all cylinders).

After cycle no. 45, container no. 1 (with $LaNi_{4.8}Sn_{0.2}$) was pumped out and the cylinders were filled with the mixture containing 75% of hydrogen and 25% of nitrogen. Then, 4 cycles for $LaNi_{4.8}Sn_{0.2}$ were carried out. Afterwards, $LaNi_{4.8}Sn_{0.2}$ was again pumped out and charged with hydrogen to check if any changes took place as a result of working with hydrogen–nitrogen mixture. Hydrogen purity used in the experiments was 99.99% H_2.

3. Results and discussion

3.1. Experiment with hydrogen

If a hydride compressor is used for the hydrogen recycling from the quenching furnace, the time of the whole compression cycle should be no longer than about 2 h and 30 min. The time of absorption is critical. Absorption should not last longer than 30 min (max. 15 min for each alloy) because during this time no other operation connected with usual furnace work could take place. We have about 2 h for desorption from $LaNi_{4.25}Al_{0.75}$ (transfer of hydrogen between both alloys) and desorption from $LaNi_{4.8}Sn_{0.2}$.

It is assumed that using $LaNi_{4.8}Sn_{0.2}$ and $LaNi_{4.25}Al_{0.75}$ alloys will make it possible to obtain pressure as low as 0.02 MPa in the furnace chamber and at the same time recover approximately 99% of the amount of hydrogen. The compressor should also be able to achieve pressure of 3.3 MPa. This pressure value obtained in the buffer container (a container from which hydrogen flows into the furnace) allows to achieve 1.5 MPa in the furnace during quenching. Quenching rate under 1.5 MPa hydrogen pressure could be compared with quenching rate of hot oil [17] and therefore obtaining

Table 1
Values characterizing the alloy beds size and emplacement.

	Container no. 1 ($LaNi_{4.8}Sn_{0.2}$)	Container no. 2 ($LaNi_{4.25}Al_{0.75}$)
Internal free volume of the container[a] (m^3)	0.024	0.002
Length of internal part of the container (m)	0.990	0.370
Diameter of internal part of the container (m)	0.2	0.1
Mass of the alloy (kg)	45	3.5
Maximum volume of the alloy[b] (m^3)	0.012	0.001

[a] Without alloy.
[b] During absorption increase of 15% of the volume was assumed.

Table 2
Temperature of $LaNi_{4.8}Sn_{0.2}$ and $LaNi_{4.25}Al_{0.75}$ alloys and hydrogen pressure in the cylinders before absorptions and desorptions.

Process	Pressure in the cylinders (MPa)	Temperature of the alloy (K)
Absorption – $LaNi_{4.8}Sn_{0.2}$	1.7–1.8	297–308
Absorption – $LaNi_{4.25}Al_{0.75}$	0.22–0.26	297–308
Desorption – $LaNi_{4.8}Sn_{0.2}$	0.14–0.26	290–300
Desorption – $LaNi_{4.25}Al_{0.75}$	<0.12	298–305

such pressure would allow further replacement of oil quenching with gas quenching.

The foregoing data regarding time limits and required pressure values were provided by Seco/Warwick, Swiebodzin, Poland, the manufacturer of heat treatment equipment.

The temperature of alloys and the pressure of hydrogen in cylinders before every absorption and desorption are shown in Table 2. The representative results obtained after 30 min of hydrogen absorption carried out for $LaNi_{4.8}Sn_{0.2}$ have been presented in Figs. 4–6. It could be noticed that the pressure at the end of absorption increased gradually in every subsequent process. The maximum hydrogen absorption capacity achieved in absorption no. 10 was 3.98 $H/LaNi_{4.8}Sn_{0.2}$. According to the pressure composition isotherm (desorption) obtained by the producer of both alloys (Treibacher Industrie AG, Austria) the alloy should be saturated in a higher degree (Fig. 7). The isotherm presented in [1] also showed higher hydrogen capacity: 5.5–6 $H/LaNi_{4.8}Sn_{0.2}$ (293–298 K). The amount of hydrogen absorbed in subsequent processes was even lower and decreased as cycling number increased, but the degradation rate was slowing down.

The results of absorption for the container no. 2 were more chaotic (Figs. 8–10). For example, in the last absorption, no. 30 the amount of hydrogen absorbed was the highest (and pressure 0.013 MPa the lowest) while the whole amount of hydrogen in the installation was lower than in other cases (with the exception of absorption 27 and 28 when the amount of hydrogen was
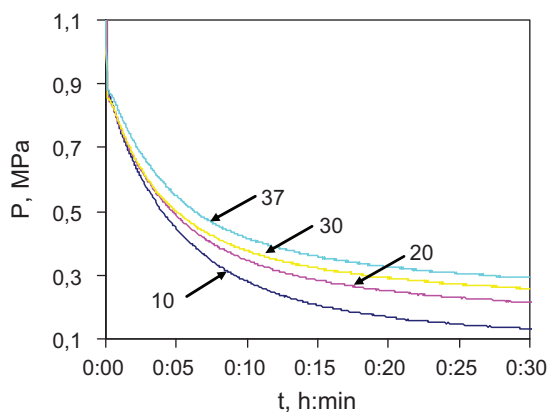
Fig. 5. Temperature of $LaNi_{4.8}Sn_{0.2}$ as a function of time for hydrogen absorption (absorption numbers: 10, 20, 30, 37).

Fig. 6. Saturation of $LaNi_{4.8}Sn_{0.2}$ during hydrogen absorptions after 5, 15 and 30 min.

Fig. 7. Pressure composition isotherm for $LaNi_{4.8}Sn_{0.2}$ obtained by manufacturer of this alloy Treibacher Industrie AG, Austria.

Fig. 4. Hydrogen pressure in the installation as a function of time during hydrogen absorption by $LaNi_{4.8}Sn_{0.2}$ (absorption numbers: 10, 20, 30, 37).
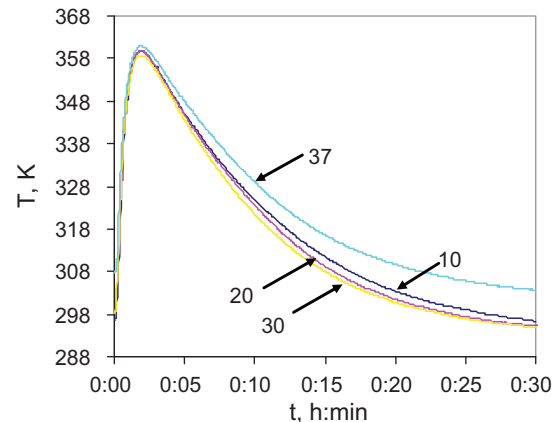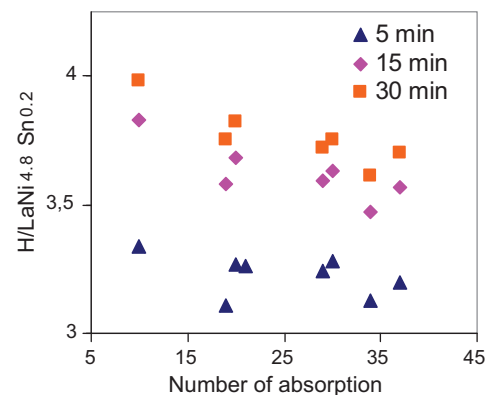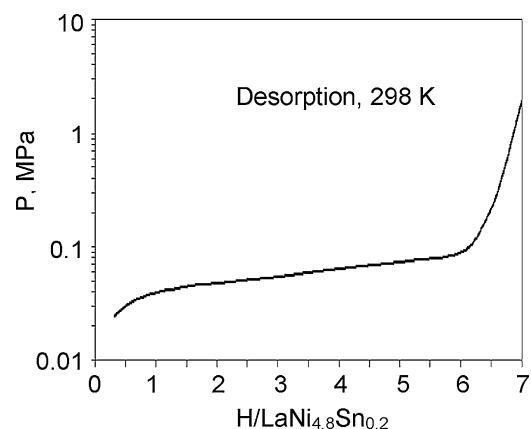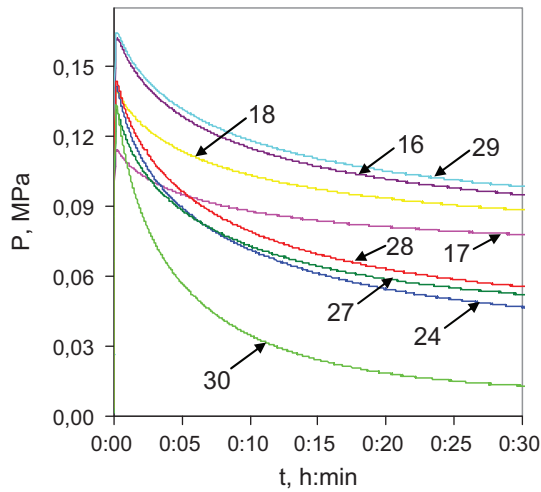
Fig. 8. Hydrogen pressure in the installation as a function of time during hydrogen absorption by LaNi$_{4.25}$Al$_{0.75}$ (absorption numbers: 16–18, 20, 24, 27, 28, 30).
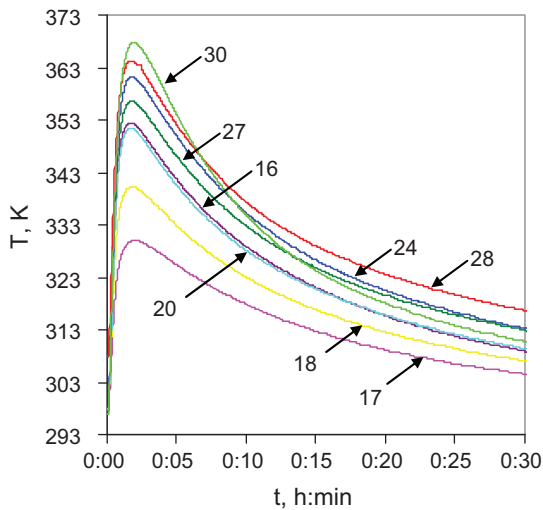

Fig. 9. Temperature of LaNi$_{4.25}$Al$_{0.75}$ as a function of time for hydrogen absorption (absorption numbers: 16–18, 20, 24, 27, 28, 30).
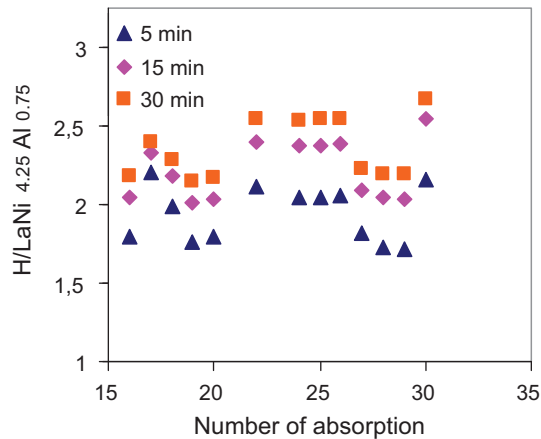

Fig. 10. Saturation of LaNi$_{4.25}$Al$_{0.75}$ during hydrogen absorptions after 5, 15 and 30 min.

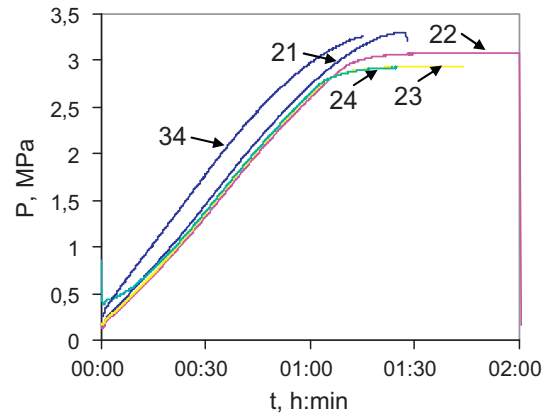**Fig. 11.** Hydrogen pressure in the installation as a function of time during hydrogen desorption by LaNi$_{4.8}$Sn$_{0.2}$. Alloy was heated to the following temperatures: desorption no. 21, 34–450 K, desorption no. 22, 22–438 K, desorption no. 23, 24–433 K.

the same). The hydrogen absorption capacity obtained for absorption no. 30 reached 2.67 H/LaNi$_{4.25}$Al$_{0.75}$ (after 30 min) and it was also lower than that in equilibrium state. The maximum hydrogen desorption capacity obtained for our alloy from *p–c* isotherm measurements was 3.5–3.65 H/LaNi$_{4.25}$Al$_{0.75}$ (0.008–0.01 MPa) in 293 K. The maximum hydrogen desorption capacity reported in [19] was 3.5 H/LaNi$_{4.25}$Al$_{0.75}$ (0.01 MPa, 313 K).

After 15 min of absorption no. 10 for LaNi$_{4.8}$Sn$_{0.2}$ and no. 30 for LaNi$_{4.25}$Al$_{0.75}$, it was possible to obtain low pressure approximating the assumed values. Nevertheless, the amount of LaNi$_{4.25}$Al$_{0.75}$ alloy was substantially lower than the amount of LaNi$_{4.8}$Sn$_{0.2}$ and Figs. 5 and 9 confirm that container no. 1 was equipped with much more efficient heat exchangers. It was impossible to achieve the level of saturation as high as for equilibrium state and the discrepancy was higher in the case of LaNi$_{4.8}$Sn$_{0.2}$. On the other hand, large variations in hydrogen storage capacity were observed for LaNi$_{4.25}$Al$_{0.75}$. It is also known that LaNi$_{4.8}$Sn$_{0.2}$ and LaNi$_{5-x}$Al$_x$ alloys exhibit less pronounced degradation of hydrogen storage potential than that presented here, even if the alloys are submitted to hundreds or thousands of absorption–desorption cycles (also for hydrogen of the same purity) [20–22]. It was concluded that in the presented experiments insufficient mass transfer was responsible for the low saturation level of alloys. We believe that alloy particle decay causing tamping down of the whole alloy bed contributed significantly to the impeded mass transfer. For LaNi$_{4.25}$Al$_{0.75}$, which works with much lower hydrogen pressure, the problem with hydrogen transport should be more visible and could be an explanation for large variations in hydrogen storage capacity that were obtained for this alloy. If a significant mass of alloys is used (45 kg and 3.5 kg), another type of container should be applied.

The results of hydrogen desorption from LaNi$_{4.8}$Sn$_{0.2}$ were more uniform and the required pressure of 3.3 MPa was achieved several times (Fig. 11). The representative saturation level for desorption 21 was: 0.74 H/LaNi$_{4.8}$Sn$_{0.2}$ after 85 min (3.3 MPa, 450 K). This result was the same as for the equilibrium state [1]. It shows that the hydrogen desorption kinetics of LaNi$_{4.8}$Sn$_{0.2}$ does not differ significantly from the data presented in other papers and it confirms the assumption that the impeded hydrogen transfer is the reason for the hydrogen absorption capacity decrease observed in this study.

During desorption of hydrogen from LaNi$_{4.25}$Al$_{0.75}$ it was possible to reach 0.22 MPa (Fig. 12). The difference in duration of processes nos. 23 (24) and 25 was caused by the difference in heating powers used for these processes. Reaching 0.22 MPa enables to transfer hydrogen from LaNi$_{4.25}$Al$_{0.75}$ to LaNi$_{4.8}$Sn$_{0.2}$ since it was possible to achieve lower pressure value for absorption by LaNi$_{4.8}$Sn$_{0.2}$ (0.15 MPa in 30 min). The residual hydrogen capac-
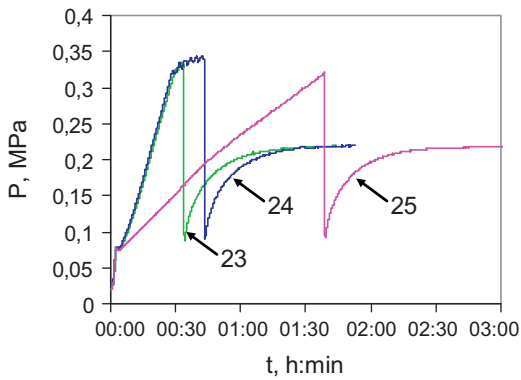
Fig. 12. Hydrogen pressure in the installation as a function of time during hydrogen desorption by LaNi$_{4.25}$Al$_{0.75}$ (desorption numbers: 23–25).

ity for desorption no. 23 after 90 min was 0.25 H/LaNi$_{4.25}$Al$_{0.75}$ (0.22 MPa, 412 K).

Desorptions for both alloys allowed to achieve the required pressure level but the times of the processes were too long if we consider industrial application of the hydride compressor (Figs. 11 and 12 do not take into account the time of preheating which was 20–25 min). Higher efficiency of heat exchangers mounted in container 1 was visible once more. Time of the process could be shortened using higher power heaters or mounting heaters inside the containers with alloys.

Fig. 13 presents the results obtained for the compression cycle when both alloys were used. The work of an industrial compressor

was simulated here. The pressure in six cylinders at the beginning of the cycle was 1.65 MPa. After absorption by LaNi$_{4.8}$Sn$_{0.2}$, the pressure dropped to 0.35 MPa and then after absorption by LaNi$_{4.25}$Al$_{0.75}$ the pressure went down to 0.025 MPa. In this experiment, the most evident finding was the long time (longer than the desorption for LaNi$_{4.8}$Sn$_{0.2}$) of hydrogen transfer between containers caused by an ineffective method of LaNi$_{4.25}$Al$_{0.75}$ alloy heating. The steep decline of the values in Fig. 13 after absorption by LaNi$_{4.25}$Al$_{0.75}$ was caused by a temporary break between this stage and the next one (oil heating) which was not shown in the figure. It could also be noticed that during hydrogen transfer hydrogen pressure was higher in container 1 than in container 2 but to conduct this stage the pressure should be higher in container 2. This discrepancy results from different accuracies of pressure sensors mounted in both containers and it increased when the temperature in the container 2 was high.

3.2. Experiment with hydrogen–nitrogen mixture

At the beginning of the experiment, the installation was filled with hydrogen. After 30 min of absorption, the pressure was 0.37 MPa (299 K, 3.5 H/LaNi$_{4.8}$Sn$_{0.2}$). Then nitrogen was introduced into the installation. This caused an increase of gas pressure in the installation by 0.75 MPa. Afterwards, four cycles of absorption–desorption for hydrogen–nitrogen mixture were carried out. The alloy absorbed less and less hydrogen in the following cycles and this degradation of hydrogen storage capacity was much stronger than that demonstrated earlier (Table 3). After pumping out the installation and filling it with hydrogen again, one more
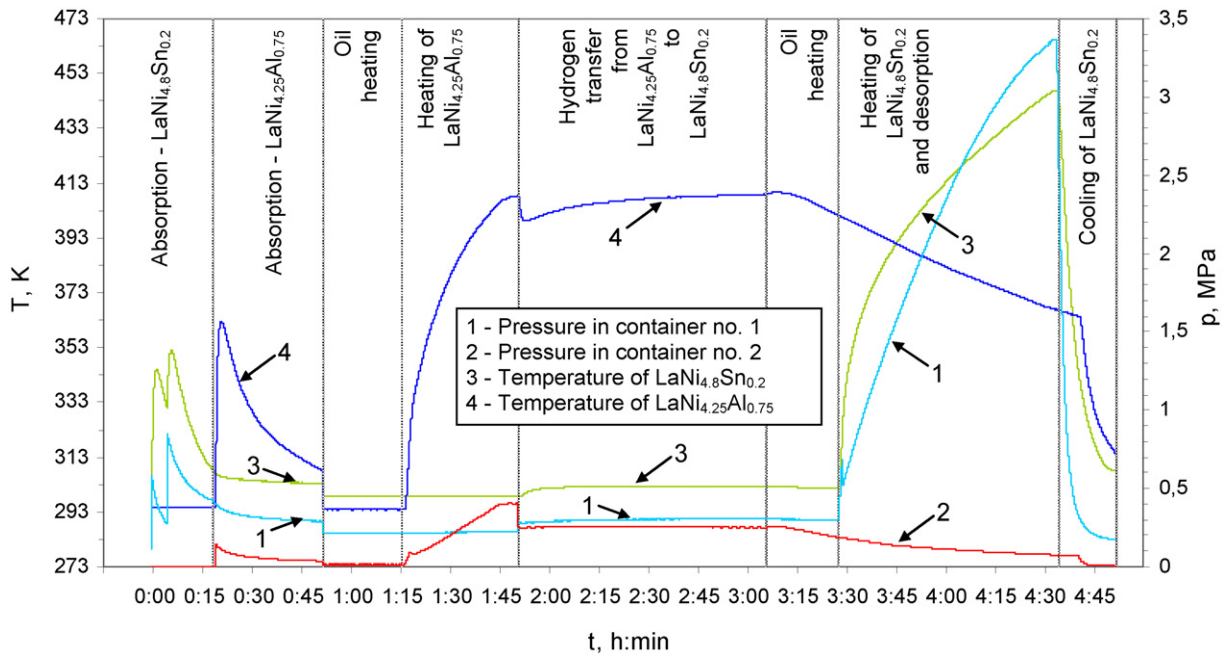

Fig. 13. Hydrogen compression cycle accomplished using containers nos. 1 and 2.

Table 3
Pressures, temperatures and degree of saturation for LaNi$_{4.8}$Sn$_{0.2}$ after 30 min of hydrogen absorption from the mixture of 75% H$_2$ and 25% N$_2$.

Absorption number	Temperature of LaNi$_{4.8}$Sn$_{0.2}$ (K)	Pressure in the installation (MPa)	Hydrogen pressure in the installation (MPa)	Degree of saturation of LaNi$_{4.8}$Sn$_{0.2}$ (H/LaNi$_{4.8}$Sn$_{0.2}$)
1	292.65	1.31	0.56	3.14
2	292.95	1.47	0.72	2.82
3	293.85	1.75	1.0	2.26
4	295.85	1.92	1.17	1.93

absorption was performed. The final pressure reached 0.35 MPa. This means that nitrogen did not have a permanent adverse effect on hydrogen storage capacity of LaNi$_{4.8}$Sn$_{0.2}$.

4. Conclusions

Results of the experiments presented in this paper are a part of the research on a hydrogen compressor. The compressor model utilized hydrides (LaNi$_{4.8}$Sn$_{0.2}$–H$_2$ and LaNi$_{4.25}$Al$_{0.75}$–H$_2$) placed in the containers equipped with heat exchangers. It was noticed that the hydrogen storage ability of this device was lower and was decreasing faster than it should be expected according to the literature concerning the employed alloys. This problem was caused probably by inefficient hydrogen transfer inside the containers. The mass of alloys, especially in the case of LaNi$_{4.8}$Sn$_{0.2}$, was probably too large for this type of containers. The alloy particle decay and tamping down of the alloys is believed to cause the main difficulties in mass transfer. The finned tubes proved to serve well as heat exchangers for cooling the alloy.

The method of heating the alloys was ineffective – the heating elements should be replaced with higher power elements or should be placed inside the containers. The latter solution could also limit heat loss.

All pressure levels required for an industrial compressor working with a quenching furnace were achieved at least once for each process type (absorption or desorption) for both alloys.

The negative influence of nitrogen on hydrogen storage ability of the containers probably involves a disturbance of hydrogen flow to the alloy. Mixing two gases inside the alloy bed is a long-lasting process but the use of a gas mixer connected to the installation will be the simplest solution. Adsorption of nitrogen molecules on the surface of LaNi$_{4.8}$Sn$_{0.2}$ and impeding absorption of hydrogen could also take place. After pumping nitrogen out of the installation, the initial hydrogen storage ability was recovered.

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