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EXPERIMENTAL INVESTIGATION OF THE EFFECTS OF VESSEL DESIGN AND HYDROGEN CHARGE PRESSURE ON METAL HYDRIDE BASED HYDROGEN STORAGE PARAMETERS

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Abstract: The hydrogen charge time in metal hydride vessel (MHV) is strongly influenced by the heat transfer from hydride vessel. In this study, the effect of parameters such as reactor geometry and alloy characteristics on hydrogen charge procedure is studied experimentally. In the experimental setup two different types of hydride vessel are designed and manufactured. Both vessels have the same interior volume; the un-finned type vessel cooled with natural convection and the second one was manufactured with fins around the vessel. The temperature variations of the vessel at four locations were measured charging with a range of pressure from 2 to 8 bar. The finned vessel show the lowest temperature increase with the fastest charging time under all charging pressures investigated. The stored hydrogen mass was measured as 0.82, 0.94, 1.15 wt% on un-finned vessel and 0.91, 1.01, 1.24 wt% on finned vessel at 2, 6 and 10 bar respectively. The experimental results show that the charge time of the vessel is considerably reduced, when the used fins manufacture on vessel. Furthermore, the addition of Al to the $LaNi_5$ alloys has caused to reduce the hydrogen charge time and decreased of the stored hydrogen mass.
Keywords: Hydrogen storage, Metal hydride, Vessel design, charge pressure, $LaNi_{4.75}Al_{0.25}$

METAL HIDRÜR ESASLI HİDROJEN DEPOLAMA PARAMETRELERİNE REAKTÖR DİZAYNI VE HİDROJEN ŞARJ BASINCI ETKİSİNİN DENEYSEL OLARAK İNCELENMESİ

Özet: Metal hidrür reaktörlerden ısı transferi önemli oranda hidrür reaktörlere hidrojen şarj süresini etkilemektedir. Yapılan bu çalışmada, hidrojen şarjına etki eden, reaktör geometrisi ve alaşım özellikleri gibi bazı parametreler deneysel olarak incelenmiştir. Deneysel çalışmalarda iki farklı hidrür reaktör tasarlanmış ve imal edilmiştir. Her iki reaktör aynı iç hacimlerinde; kanatçıksız reaktör doğal konveksiyonla soğutulurken, ikinci reaktör kanatçıklı olarak dizayn edilmiştir. Reaktörlerin dört farklı noktasından, 2-8 bar arasında farklı hidrojen şarj basınçlarında sıcaklık değişimleri ölçülmüştür. Kanatçıklı reaktör ısı transferinin hızlı olmasına bağlı olarak; bütün şarj basınçlarında, daha hızlı şarj sürelerinde, düşük sıcaklık yükselmeleri göstermiştir. Depolanan hidrojen miktarı ise 2, 6 ve 10 bar hidrojen şarj basınçlarında kanatçıksız reaktörde sırasıyla ağırlıkça % 0.82, 0.94, 1.15 olurken, kanatçıklı reaktörde sırasıyla ağırlıkça % 0.91, 1.01, 1.24 olarak ölçülmüştür. Deney sonuçlarına göre reaktör üzerinde kanatçık imal edilmesi reaktöre hidrojen şarj süresini önemli oranda düşürmüştür. Ayrıca, $LaNi_5$ alaşımlarına Al ilavesi hidrojen şarj süresini ve depolanan hidrojen miktarını düşürmüştür.
Anahtar kelimeler: Hidrojen depolama, Metal hidrür, reaktör dizaynı, şarj basıncı, $LaNi_{4.75}Al_{0.25}$

INTRODUCTION

The concerns about negative effects of greenhouse gases and exhaustion of fossil fuels make it important to use alternative energy sources. Due to its high calorific value and cleanness, hydrogen attracts attention as an energy carrier has been studied widely. However, the storage problem of hydrogen prevents its wide usage and commercialization. In recent years there has been increasing interest in using metal hydrides for hydrogen storage due to advantageous characteristics such as high volumetric density and better safety compared to conventional methods. In recent years metal hydride reactors which can store large amounts of hydrogen in small volumes and which allow working at high pressure levels, have attracted considerable attention. For this reasons, hydride reactor design are extremely important for removing the heat from the reactor. Therefore, there have been numerous experimental and theoretical investigations on several aspects of hydrogen storage.

Demircan et al. (2005) experimentally examined the hydrogen absorption in two $LaNi_5$-H_2 reactors. They found that hydriding time mainly depends on the successful heat removal from the bed and the bed geometry; the one that provides more heat transfer area significantly reduces hydriding time. Optimization of hydrogen storage in metal hydride beds was investigated by Eustathios et al.(2006). In this study, cooling design options were investigated by introducing additional heat exchangers with a concentric inner tube and annular ring inside the tank. Kaplan (2009) has studied the effects of heat transfer mechanisms on the charging process in metal hydride reactors under various charging pressures. Three different cylindrical reactors with the same base dimensions were designed and manufactured. The experimental results showed that charging to the MHVs was mainly heat transfer-dependent and the reactor which has better cooling condition exhibited the fastest charging characteristics. Dhoau et al. (2010) investigated the reaction time of hydrogen in metal hydride vessels which was strongly influenced by the heat transfer from/to the hydride bed. They experimentally studied the effect of geometric and the operating parameters of a finned spiral heat exchanger. This was carried out to identify their influence on the performance of the charging process of the metal hydride vessel. The experimental results show that the charging time of the reactor was considerably reduced, when finned spiral heat exchanger is used. In addition, the effect of different parameters (mass flow rate and temperature of the cooling fluid, applied pressure, and hydrogen tank volume) were discussed and the obtained results showed that the optimum choice of these parameters was important. Mellouli et al. (2007) designed and manufactured a novel design of MHV equipped with a spiral heat exchanger and mentioned that the charge and the discharge times were significantly reduced. Jemni et al.(1999) presented an experimental approach to determine the reaction kinetics, the equilibrium conditions and the transport properties in $LaNi_5$–H_2 system. Heat and mass transfer effects were also determined experimentally. Based on Jemni and Ben Nasrallah’s continuum mixture model [Jemni et al. 1999], Mat and co-workers carried out a detailed analysis and studied the parameters affecting the hydriding process, and extended the analysis to three-dimensional cases [Mat et al., 2002]. Muthukumar has analyzed the effects of various operating conditions in a metal hydride-based hydrogen storage device using AB_5 alloys and found that cold fluid temperature has a significant effect on hydrogen storage capacity at lower supply pressures, and higher values of overall heat transfer coefficients yield better rates of absorption and desorption [Muthukumar, 2005]. At a supply pressure of 35 bar and at a cold fluid temperature of 15 °C, $MmNi_{4.6}Fe_{0.4}$ alloy stored about 1.6 wt%, while $MmNi_{4.6}Al_{0.4}$ stored 1.3 wt%. The optimization of hydrogen storage in metal hydride beds was investigated by Kikkinides et al. (2006). In these two references, cooling design options were investigated by introducing additional heat exchangers with a concentric inner tube and with an annular ring inside the tank. The problem was mathematically formulated as a dynamic optimization problem where the selected control variables were the cooling fluids flow rates and/or temperatures as well as the hydrogen charging profile. Design decisions included, among others, the radius of the inner tube and the radial position of the concentric annular ring in the tank. Optimization results indicated that significant improvements in the total storage time could be achieved by a safe and economical. $LaNi_xAl_{5-x}$ is considered to be a very useful material for H_2 absorption because of its low plateau pressure and the resistance to impurities in hydrogen gas. It was also found that the partial replacement of Ni in $LaNi_5$ alloy by a small amount of Al resulted in a prominent increase in the cycle life time without causing much decrease in hydrogen absorption capacity and in minimizing corrosion attack of the hydride electrode [Zhang et al. 1993; Feng et al. 2000; Wang et al. 1993; Kiyonori et al. 2000; Cheng et al. 2006; Giza et al. 2007]. It should be noted that partial substitution amount of Al for Ni is below 0.5.

In this paper two kind of metal hydride vessel called finned and un-finned were designed and manufactured. The effect of heat transfer on the hydriding/dehydriding rate, reaction kinetic, Al addition in $LaNi_5$ alloys and thermal behavior of two metal hydride vessels was investigated experimentally. Results obtained from experimental study were compared for two vessels. In experimental studies each vessel, 2-8 bar of hydrogen charge pressure was adjusted and the thermocouples were positioned at 5, 20, 40 and 60 mm points in the z-axis of the vessels and temperature distributions along the vessel were measured. In addition, the charge pressure was changed between 2-10 bar and the effect of the charge pressure, the temperature and vessel design on the stored hydrogen mass at vessels were determined.

EXPERIMENTAL METHOD

Vessel design and experimental setup

Two different vessels were designed as shown in Figure 1 to determine the effect of heat transfer between hydride vessel and environment, by taking hydrogen pressure and metal hydride vessel geometry into account, which are the process parameters that mainly affect hydrogen charge/discharge procedures on hydrogen storage (hydrogen pressure and metal hydride vessel geometry). Both vessels are made of St 42 steel. Un-finned vessel was the base case having 20 mm inner diameter, 125 mm outer length and 2 mm thickness. Finned vessel has the same dimensions as un-finned vessel but had 23 circular fins spaced by 4 mm distance, 40 mm outer diameter and 1 mm thickness to obtain a better heat removal from the vessel. Experimental setup for hydrogen absorption is schematically presented in Figure 2. The setup mainly consists of a metal hydride vessel, a vacuum pump to evacuate from vessel before the experiment, a ball mill to produce fine $LaNi_{4.75}Al_{0.75}$ particles, manometers, thermocouples, a data acquisition system, a tank containing 99.999% pure hydrogen gas and a nitrogen tank providing operation under inert atmosphere environment. Four temperature points were added at the same height on the vessels.

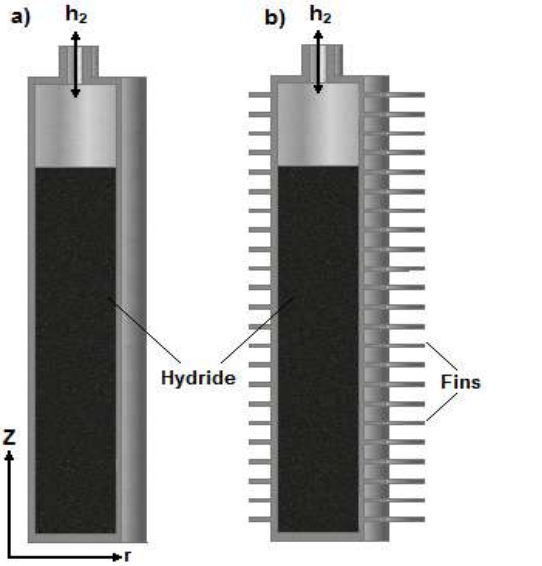


Figure 1. Cross sections of two different vessel designs (a) Un-finned (b) Finned

Experimental procedures

Before the hydriding test, $LaNi_{4.75}Al_{0.25}$ powder was grounded for 4 h. Grinding process was conducted by a ball mill which was located in a glow box of 60 mm diameter and 80 mm height. Nitrogen gas was used during the process to prevent possible oxidation. Grinding speed was set at 515 rpm. After milling, the powders were put into the storage vessel. The packing fraction was approximately 75% of the internal volume (125 g). Activation is a very important problem for practical applications of metal hydrides because the surfaces of metals are usually covered with oxide of various thicknesses, depending on the formation process of particular metal. After grinding, the first hydrogenation was performed at 200 °C in furnace for 2 h under low pressure. The metal powders were activated by repeatedly evacuating and filling the storage tank with high pressure hydrogen (10 bar) for 2 h at room temperature. After the activation, the hydrogen storage capacity reached a saturated value of approximately for finned and un-finned vessel 1.15 and 1.24 wt%. The experimental part of the study started after the activation process was fully completed. Vessels were then charged with hydrogen at the range of 2–8 bar pressure, where the pressure was continuously maintained.

The hydriding process was monitored by temperature measurements obtained at four locations r=0 and z=5–60 mm from the bottom of the vessels for the first 4000 s of the absorption period.

The temperature and storage hydrogen mass variation were recorded on a computer for further processes and for determination of storage rate and the interpretation of hydriding behavior.

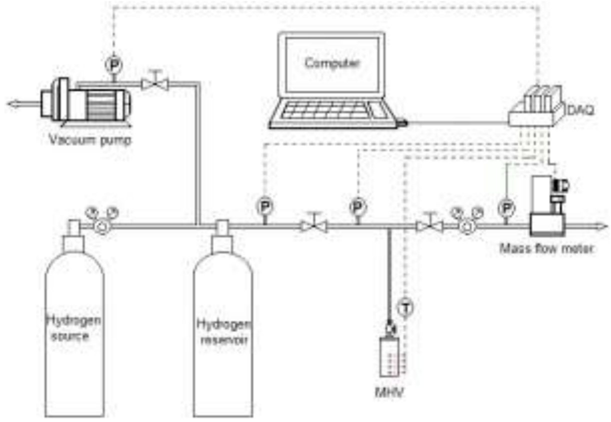


Figure 2. The schematic diagram of the experimental setup

Uncertainty analysis

An uncertainty analysis was performed to assign credible limits to the accuracy of reported values. When the uncertainties are random and multiple trials are performed to obtain the best estimate of a parameter, the standard deviation is an appropriate choice for describing the uncertainty in the measurement (Ceylan et al., 2013).

$$x_m = \frac{1}{N}\sum x_i \quad (1)$$

where x_m arithmetic mean of experimental measurement.

The uncertainty associated with each measurement can be determined from the standard deviation *S* mathematically, the standard deviation can be expressed as;

$$S = \left[\frac{1}{N}\sum_{i=1}^{N}(x_i - x_m)^2\right]^{1/2} \quad (2)$$

In here *N* Number of times the measurements are performed, x_i corresponds to i^{th} measurement of the parameter. The uncertainty can be calculated by following equation.

$$a = \frac{1}{\sqrt{N}} \quad (3)$$

$$U = \sqrt{\sum_{i=1}^{R} a_i^2 . S_i^2} \quad (4)$$

where *U* is uncertainty and *a* sensitivity. The standard deviation provides an estimate of the average uncertainty associated with any one of the *N* measurements that were performed. An uncertainty analysis result was shown Table 1. Measured temperature and stored hydrogen were more stable at 2 bar for the both vessels.

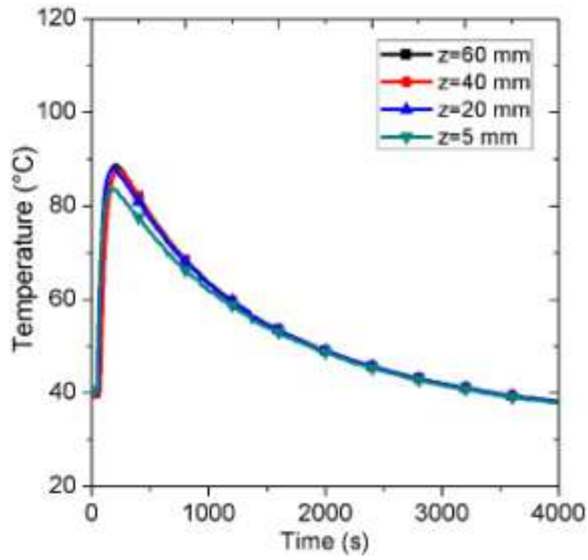
Table 1. Uncertainty analysis of temperature and absorbed hydrogen with Vessel 1 and Vessel 2 at different charging pressure.

Pressure (bar)	Vessel 1		Vessel 2	
	Temperature (°C)	Stored H_2 (g)	Temperature (°C)	Stored H_2 (g)
2	±1.36	±0.016	±1.21	±0.017
6	±1.67	±0.019	±1.61	±0.018
10	±1.97	±0.021	±1.78	±0.023

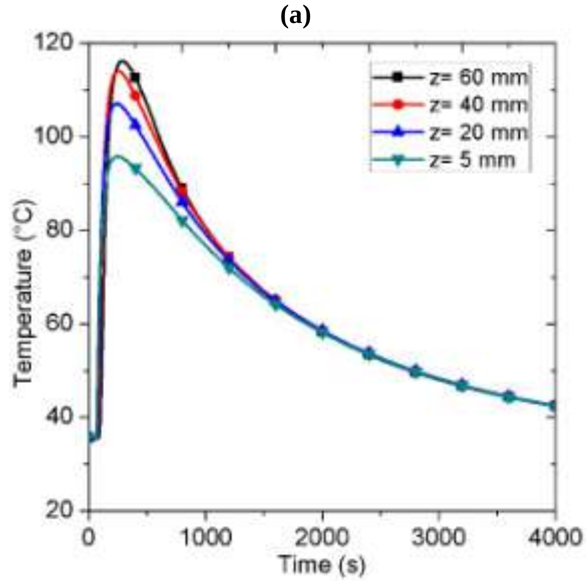
RESULTS AND DISCUSSIONS

In this study the performance of hydrogen storage devices were mainly denoted by the hydrogen storage capacity and the absorption rate. Figure 3a and b shows the temperature variation of the un-finned vessel during the first 4000 s at different axial positions and at 2 and 6 bar charging pressure, respectively. Accordingly, as one moves from the centre to the base of the vessel, the time period for reaching the maximum temperature increases. It seen that region close to inlet of the vessels has highest temperature in both vessels. At all points of the vessels, maximum temperature was reached approximately 300 s. At 2 bar pressure, the maximum temperature values are 88.77, 80.7, 79.7 and 75.71 °C in 209, 292, 206 and 199 s for at z=60, 40, 20 and 5 mm respectively. At 6 bar of pressure, the maximum temperature values are measured as 116.24, 114.11, 107.12 and 95.8 °C in 290, 251, 245, 237 for at z= 60, 40, 20 and 5 mm respectively. Figure 4a and b shows the temperature variation of the finned vessel during the first 4000 s period at 2 and 6 bar, respectively. At 2 bar, the maximum temperature values were measured as 81.33, 80.7, 79.7 and 75.71 °C in 225, 224, 206 and 199 at z= 60, 40, 20 and 5 mm points respectively. At 6 bar of charging pressure the maximum temperature values were measured 113.27, 111.08, 107.45 and 96.92 °C in 202, 201, 193 and 190 s at z=60, 40, 20, and 5 mm points respectively. According to these results, it can be seen that the temperatures measured for the finned vessel were lower than at those for the un-finned vessel.

The temperature variation of the un-finned vessel and the finned at z=60 mm during charging at different hydrogen pressures of 2, 4, 6 and 8 bar is shown in Figure 5a and b. For a higher value of charging pressure, there would be more hydrogen available in the vessel, thus a higher charge of the hydride would be possible in vessels. Different locations on the vessels have a sharp temperature rising at first 400 s; it could see that hydriding occurred over the inlet vessel in the initial phase. The maximum temperature was reached at 8 bar and began to fall down depend on pressure decreased. In addition, the amount of stored hydrogen was increased as the rate of exothermic reactions is increased. At z=60 mm location on the un-finned vessel, the time needed for reaching maximum temperature at 2, 4, 6 and 8 bar were recorded as 229, 398, 292, 356 s respectively and the maximum temperatures were measured at these periods 88.77, 102.92, 116.24 and 128.72 °C respectively. At z=60 mm location of the finned vessel, the maximum temperature at 2, 4, 6 and 8 bar were recorded as 225, 268, 202, 284 s, respectively,



(a)



(b)

Figure 3. The temperature variation of the un-finned vessel along its length during charging of different hydrogen pressures of 2 bar (a) and 6 bar (b)

and the maximum temperatures measured in these periods were 81.33, 98.1, 113.27 and 121.52 °C, respectively.

The stored hydrogen variation of different hydrogen charge pressures of 2, 6 and 10 bar for un-finned and the finned vessel were shown in Figure 6. For both vessels, as hydrogen charge pressure increases, exothermic reaction also increases. Therefore, the amount of stored hydrogen and charge rate also improved. During the first 400 s as reaction occurs quickly hydrogen mass also increases. After this period, the rate of mass increment slows down and remains almost fixed until it reaches the maximum capacity. Hydrogen charge pressure values were set as 2, 6 and 10 bar and the value of stored hydrogen at these pressure levels were measured as 0.82, 0.94 and 1.15 wt% respectively, for the un-finned vessel, while the hydrogen mass was measured as 0.91, 1.01 and 1.24 wt% respectively for the finned vessel. It is clear that for a high pressure, the absorption kinetics was improved.

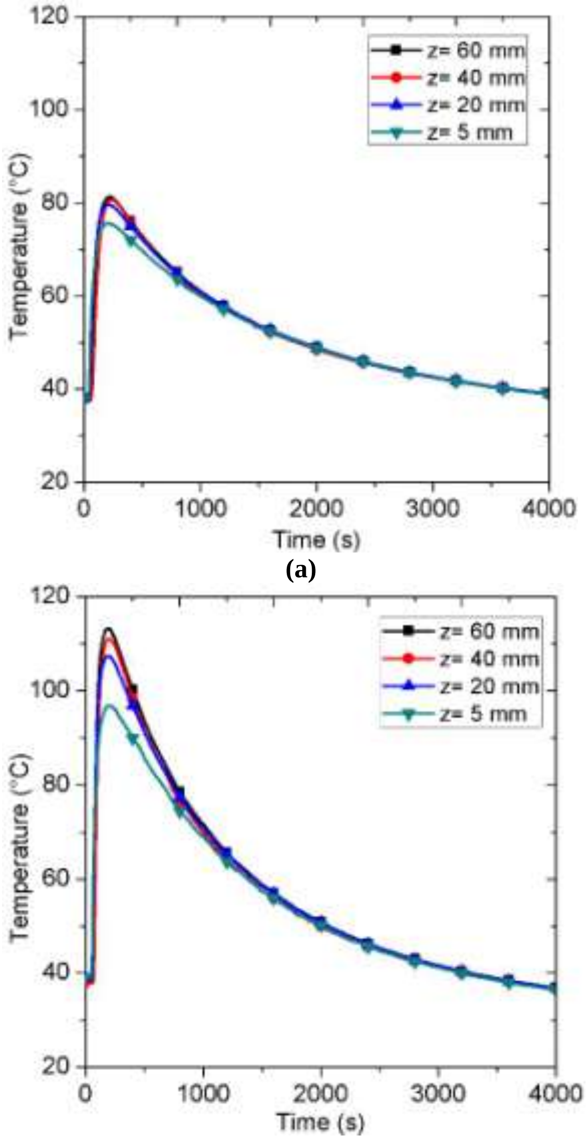


Figure 4. The temperature variation of the finned vessel along its length during charging of different hydrogen pressures of 2 bar (a) and 6 bar (b).

The variation in stored hydrogen mass and reaction temperature for various vessel designs are shown in Table 2. The values of the parameters (such as maximum temperature, time etc.) obtained from our study has been compared with available studies results conducted in literature. It can be clearly seen from table results of this study are good agreement with the given references. The Al addition to the $LaNi_5$ alloy has caused to accelerate charging time while it was decrease the stored amount of the hydrogen mass. Furthermore, according to this table it is presented that the stored hydrogen mass increased when the heat transfer improved by using the different vessels design such as finned, water cooled, heat pipe and spiral heat exchanger.

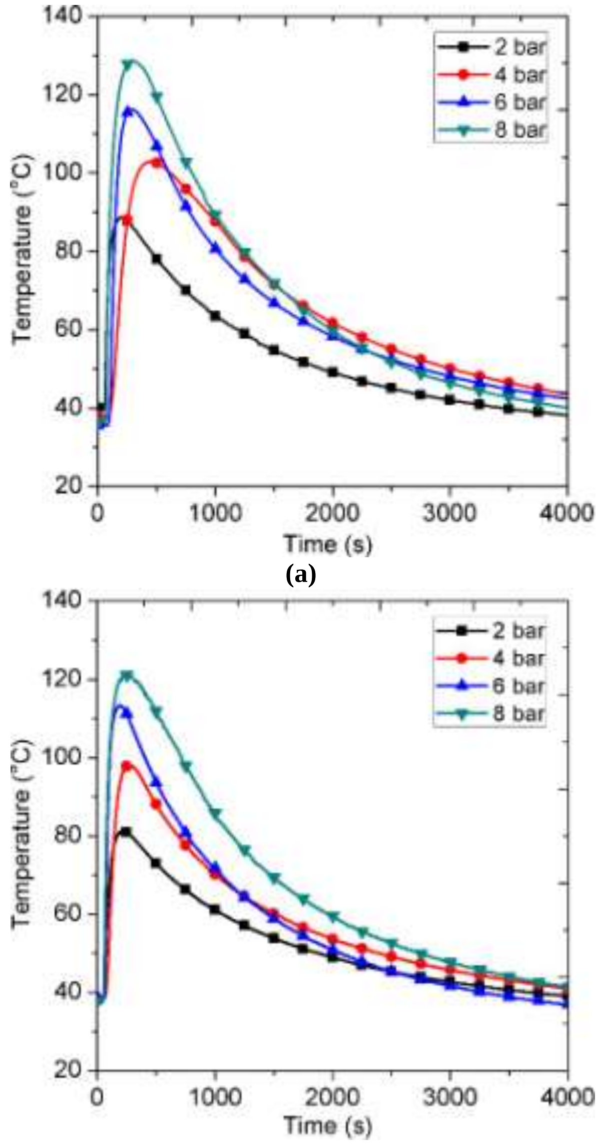


Figure 5. The temperature variation of the Un-finned (a) and Finned (b) vessel at z=60 mm during charging of different hydrogen pressures of 2–8 bar.

The temperature values of the metal hydride vessels measured at high charge pressures are larger than those measured at lower charge pressures. The reason behind this reality is that the increase of charging pressure acts to increase the reaction rate. Therefore the exothermic reaction rate at higher pressures is faster and the reaction rate decreases as charge pressure is reduced. It was observed that the heat evolved as a result of reaction was higher at the center and decreases from the center to the wall. In addition, it was seen that temperature increased as moving along the z-axis and the maximum temperature was measured at z=60 mm.

In addition, along the length of the vessel, there were different periods that had maximum temperatures at different conditions. As the upper section of the vessel had met hydrogen before, less hydrogen was found here compared to the lower section of the vessel.

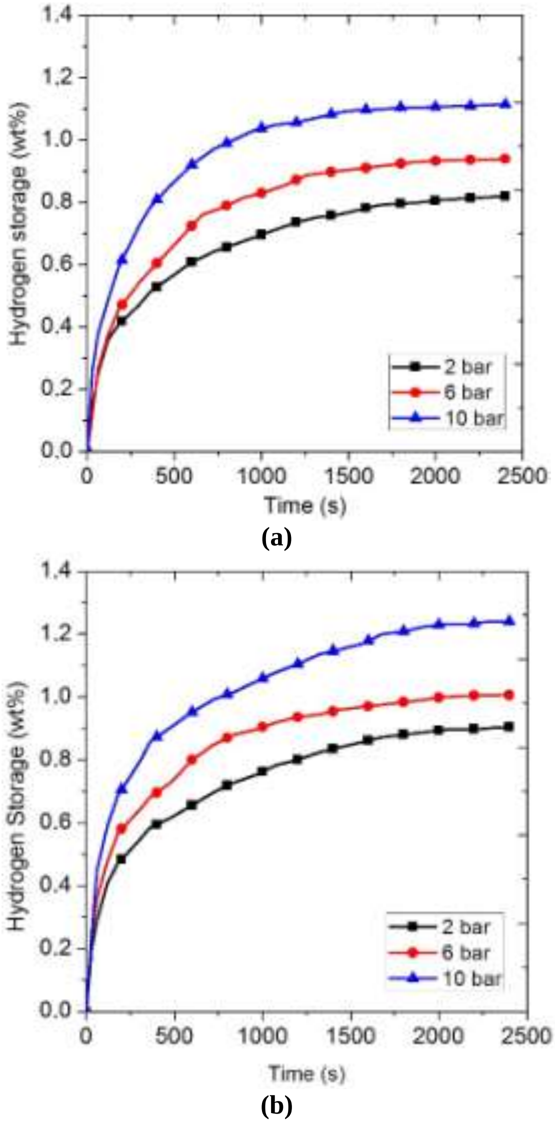


Figure 6. The variations of stored hydrogen mass at 2, 6 and 10 bar for (a) un-finned and (b) finned vessel versus time.

As approaching the entrance area, hydrogen absorption is completed and as one goes towards z-axis, there was more chance of contact with more hydrogen. In addition, by taking the experiments into account which were conducted to the amount of hydrogen stored at vessels versus time, it was determined that hydrogen charge pressure and vessel design affected the amount of hydrogen stored. The hydrogen charge was conducted with the un-finned and the finned vessels at 2, 6 and 10 bar pressure values and the amount of hydrogen stored was measured at each time. As hydrogen charge pressure to vessels increased, the amount of stored hydrogen also increased.

CONCLUSIONS

In this study, two different vessels were designed as finned and un-finned with the same base dimensions and manufactured. The un-finned vessel was the base case.

This study presents comparisons between the tanks with and without fin, and also grinding was performed for 4 hours so as to increase activation, storage rate and improvement of the storage hydrogen amount for $LaNi_{4.75}Al_{0.25}$ alloys. Therefore the powder of the alloy was produced smaller size and it was ensured that surface area, together with the amount of stored hydrogen increases.

The results are:

- During charging of hydrogen to vessels, as metal hydride alloys at contact at first with the areas which are close to the inlet vessel's the temperature increment reaches at maximum values at this point and as one moves along the x-axis of the vessels, we notice the maximum temperature values at 5, 10, 40 and 60 mm points respectively.

Table 2. Comparison of the different vessel geometries.

Vessel type	Max.Temp. (°C)	Time* (s)	Pressure (bar)	Stored H_2 (wt%)	Alloys	Ref.
Un-finned reactor	58	~300	2	-	$LaNi_5$	Kaplan,2009
Finned reactor	55	~250	2	-	$LaNi_5$	Kaplan,2009
water cooled reactor	32	~200	2	-	$LaNi_5$	Kaplan,2009
with heat pipe	75	~200	10	~1.3	$LaNi_5$	Chung et al. 2013
without heat pipe	95	~200	10	~1.3	$LaNi_5$	Chung et al. 2013
With spiral heat exchanger	20	~250	10	~1.3	$LaNi_5$	Melloulli et al. 2007
Un-finned	88	209	2	0.79	$LaNi_{4.75}Al_{0.25}$	this study
Un-finned	102	238	6	0.91	$LaNi_{4.75}Al_{0.25}$	this study
Un-finned	116	292	10	1.07	$LaNi_{4.75}Al_{0.25}$	this study
Finned	81	190	2	0.85	$LaNi_{4.75}Al_{0.25}$	this study
Finned	98	202	6	0.97	$LaNi_{4.75}Al_{0.25}$	this study
Finned	113	268	10	1.19	$LaNi_{4.75}Al_{0.25}$	this study

*Reached maximum temperature time

- Using $LaNi_{4.75}Al_{0.25}$ alloys ensured significant increased at the reaction rate but decreased storage hydrogen mass compared to $LaNi_5$ alloys. The absorption time was increased almost 10%.
- Valuable developments were also observed at reaction kinetics and thermodynamic characteristics by grinding alloys in atritor and activation process. While the hydrogen is charged, an exothermic reaction occurs. For different pressure values, 2, 4, 6 and 8 bar, the temperature increased rapidly during the first 400 s at the vessels. Maximum temperature values for both vessels were reached vary points, depending on the hydrogen charge pressure and depend on the vessel. However, it is possible to say that as pressure increases maximum temperature values increase, too. In addition, depending on the heat transfer, temperature values for the finned vessel were lower than for the un-finned vessel.
- As the hydrogen charge pressure increases, exothermic reaction also increases and therefore the mass of stored hydrogen rises. Mass of stored hydrogen essentially depends on the cooling of the vessel.
- Finned vessel stored more hydrogen compared to un-finned vessel, because of the faster heat transfer rate.

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