



AN EXPERIMENTAL DEVICE FOR EVALUATION OF HYDROGEN SORPTION

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Abstract

Hydrogen storage for the purposes of the automotive industry in a form other than under high pressure or cryo conditions has been under careful investigation by researchers over past decades. One of the arising methods is the usage of powdered/granulated beds that contain metal hydrides and/or carbon materials to take advantage of the “spillover” phenomenon. Handling and characterization of such material can be troublesome, which is why the experimental setup needs careful investigation. The apparatus for the analysis of hydrogen sorption/desorption characteristics has been successfully designed and described based on the constructed unit within the scope of this article. The full functionality of that setup covered fuelling the bed as well as the examination of sorption/desorption potential. Moreover, the proposed experimental device can clarify many uncertainties about further development and optimization of hydrogen storage materials.

Keywords: demonstrator, experimental device, hydrogen storage, sorption capacity, bed sorption.

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1. Introduction

The use of hydrogen in a variety of mobile objects and, related to this, the topic of hydrogen storage, constitutes a subject of strategic global studies. The dominant branch of research focuses on the development of safe lightweight containers for compressed gas (in the range of 350 to 700 bars, and networks as well as logistics of its distribution [1]). An alternative solution is a concept of storing chemically bounded hydrogen under low pressure – mainly in a form of metal hydrides [2, 3]. However, novel solutions that will fulfill a criterion of minimum gravimetric density (which, according to the US Department of Energy, should amount to 6% [4]) are necessary. A group of substrates essential for the development of this idea comprises carbon

materials (such as high porosity carbon, carbon nanotubes, fullerenes, or graphene and graphene-related materials) able to reversibly store large amounts of hydrogen [5–7]. High hydrogen gravimetric density of the systems based on various types of carbon allotropes is achieved through their nano- or micro-scale modifications with metallic catalysts and the phenomenon of “spillover” [8, 9]. In that way, decorated by metallic catalysts carbon materials benefit from simultaneous chemical and physical mechanisms of bonding hydrogen. Apart from the mentioned pros, there are still numerous disadvantages of that type of material for hydrogen storage. In the case of nanotubes, the problematic aspect is their reproducible (under industrial conditions) synthesis, namely, maintaining strictly controlled dimensions. Partial decomposition of fullerenes during hydrogen release and, related to this, volatile hydrocarbon contamination [10], still needs careful investigation by researchers. In this way, it is metal-decorated graphene and graphene oxide that make a rational choice of a carbonic material for reversible sorption of hydrogen.

Storage of hydrogen for purposes of the automotive industry involves mainly the use of high-pressure cylinders. The common examples are 35 MPa containers for heavy-duty vehicles and, in the case of lighter vehicles, also the 700 bar MPa ones. Such approach was used, *i.a.*, in Toyota Mirai [11]. Such high-pressure tanks and the technology or gas compression require both money and energy. The best initial form of application opportunity for low-pressure small hydrogen storage systems of a capacity between 0.2 and 1 kg appears to be in the field of special-purpose electric carts equipped with fuel cell systems. In 2021, the market of forklift trucks in Poland amounted to approximately 34 thousand pieces, while the offer of electric vehicles was comprised of ca. 2.7 thousand models [12]. A wide range of carts that include passenger, golf, and special purpose ones, reaches the annual production of several thousands of pieces. The advantages of hydrogen storage tanks in such applications are related to logistic purposes. The usage of low-pressure sorption containers does not require a distribution system of highly compressed hydrogen; therefore, there is no such proliferation barrier in this case. The supply of hydrogen for such types of vehicles could be simply performed with the use of standard 150 bar hydrogen cylinders produced and distributed by companies already present in the market.

The given article presents an experimental unit designed for the purpose of examining of hydrogen sorption capacity under conditions approaching normal ones. The functionality of the proposed device was proved with the use of a solid-state working bed – granulated spatial graphene with metallic catalysts.

2. Methodology

2.1. Hydrogen sorption/desorption test bench

The main element of the system is an AISI 1.4541 stainless steel container for the working bed material. The schematic drawing of the container chamber is presented in Fig. 1. The system is equipped with two connections allowing the refilling and supplying of hydrogen. The process of dosing gas to the tank is carried out using a mass flow regulator (β -ERG) with a maximum capacity of 10 l/min, while the process of gas supply to the working element is carried out using a mass flow regulator with a maximum capacity of 1 l/min. The hydrogen supply and discharge system is based on ball valves with a maximum operating temperature of 200°C. In addition, a sintered silencer made of stainless steel as a particulate filter is used in the place of gas evacuation. The pressure and temperature measurement system consists of a manometer with a working range of –1–10 bar with the possibility of electronic reading of pressure values and three probes of different heights enabling analysis of the bed temperature gradient at different

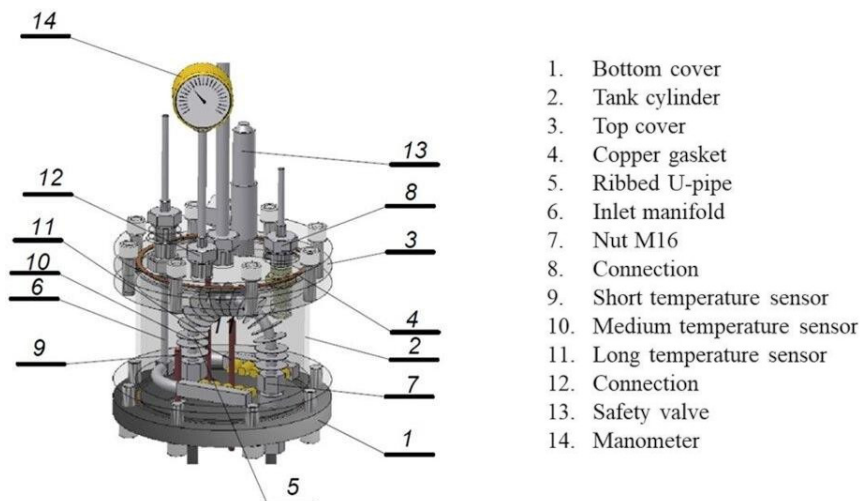


Fig. 1. Schematic view of the working bed container with peripheral devices.

heights. The system is equipped with a safety valve with an opening pressure of 10 bar and the ability to lead the gas outside the working area of the station.

The allowable temperature and pressure range in which it is possible to conduct tests is from 10 to 200°C and 0.9 to 10 bar, respectively. All system components (pressure gauge, mass flow regulators, safety valve) are comply with the requirements for working with explosive gases. The temperature control system is designed on the basis of two laboratory baths with heating and cooling power of 2 kW and 2.2 kW, respectively. The operating diagram of the system is presented in Fig. 2. A spiral made of stainless-steel pipe with an outer diameter of 10 mm with a set of valves allowing remote control of the cooling/heating liquid flow depending on the required bed temperature is mounted inside the working bed container.

The measuring system makes it possible to determine the energy characteristics of the sorption/desorption cycle of the analysed sorbing bed. The system is based on LabView software and a set of sensors, which, coupled with the LCD display, provide real-time values of individual

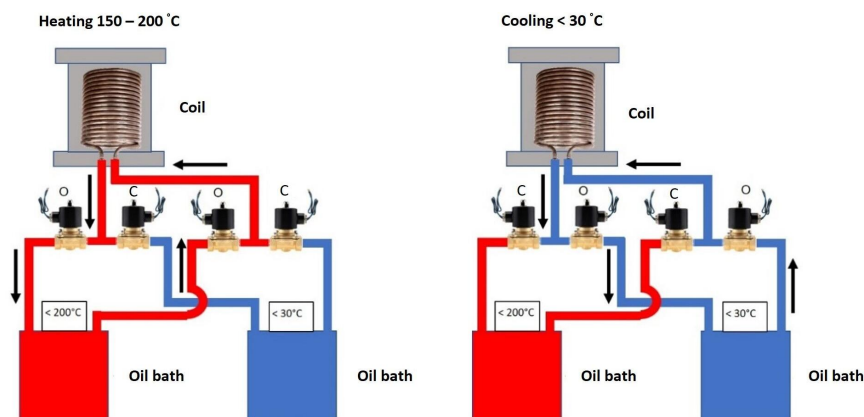


Fig. 2. Operating diagram of heating and cooling of the working medium.

system parameters, and also control its operation based on the recorded parameters. The most important operating parameters include:

- the amount of hydrogen introduced into the tank,
- hydrogen pressure in the tank,
- temperature measured at three points located at different heights of the bed column,
- the amount of hydrogen removed from the tank.

The system is equipped with a process controller enabling control of the devices included in the test bench. In addition to the basic functions of recording operating parameters, the controller will also control the heating and cooling system as a function of gas pressure in the tank and bed temperature. The main tracked and regulated parameter in the tank is pressure. A Shinko PCB1 process controller provides pressure changes or stabilization. It can work in the stabilization or program control mode (10 programs with 10 steps each – to be used depending on the research purpose). The programs allow setting separate work strategies for the hydrogen tank filling phase and for the hydrogen recovery phase. The pressure in the tank is influenced by controlling the temperature in the heating/cooling mode. The controller has the ability to work on-off or PID with the function of auto-tuning. SSR (Anly ASR-10DD-H) external semiconductor relays have been attached to the controller relay systems. It allows to control the two groups of two solenoid valves in the heating and cooling circuit depending on the requested temperature. An additional electric heating system and additional insulation are applied to the tank walls. The additional heating system is based on a 1200 W resistance heater and is activated by the same set of relays that activate the solenoid valves in the main heating/cooling system.

An NI-USB6212 measuring card was used at the stand. 8 signals were connected to it according to Table 1. The card is operated via an application in the LabVIEW environment providing:

- acquisition and registration of all measured signals to a file with a fixed sampling time from 1 s to 10 min,
- graphic presentation of the process on the virtual control panel.

Table 1. Description of signals and sensors used for system control and data acquisition.

Channel number	Registered parameter	Type and range of signal	Sensor
CH0	Pressure in the main tank	4–20 mA (250 Ω /1–5 V)	Wika PGT23/–1–15 bar
CH1	Temperature in the main tank (top)	4–20 mA (250 Ω /1–5 V)	Wika TR34/20–250°C (Pt1000)
CH2	Temperature in the main tank (middle)	4–20 mA (250 Ω /1–5 V)	Wika TR34 / 20–250°C (Pt1000)
CH3	Temperature in the main tank (bottom)	4–20 mA (250 Ω /1–5 V)	Wika TR34 / 20–250°C (Pt1000))
CH4	Hydrogen flow in the main tank inlet	0–10 V	analogue measuring output of the flow controller ERGM (BETA – ERG Electronic Pressure and Gas Flow Regulators)
CH5	Hydrogen flow in the main tank outlet	0–10 V	analogue measuring output of the flow controller ERGM (BETA – ERG Electronic Pressure and Gas Flow Regulators)
CH6	on/off status of the heating system	binary 0–5 V	Direct measurement
CH7	on/off status of the cooling system	binary 0–5 V	Direct measurement

2.2. Measurement methodology

The designed test bench makes it possible to determine both sorption and desorption capacity of the working bed.

Determination of the bed desorption capacity

- In order to initiate the hydrogen desorption process, the temperature of the bed is gradually increased until the hydrogen pressure in the tank increases.
- The shutoff valve is opened and hydrogen from the tank is gradually released by means of the mass flow regulator to maintain a constant pressure of 1 bar in the working chamber. The volumetric flow of hydrogen is recorded and stored in a file.
- Gravimetric hydrogen desorption capacity of the bed is determined on the basis of the following equations:

Mass of hydrogen discharged from the tank:

$$m_1 = \int_0^{tk} q dt, \quad (1)$$

where: m_1 – a mass of hydrogen discharged from the tank [g], q – volumetric flow recorded on the mass flow controller [g/s].

Mass of hydrogen desorbed by the bed:

$$m_{H_2} = m_1 - m_G. \quad (2)$$

Gravimetric density is determined based on the equation:

$$\rho = \frac{m_S}{m_Z} \cdot 100\%, \quad (3)$$

where: ρ – gravimetric density [%], m_s – a mass of hydrogen desorbed by the bed [g], m_Z – a mass of the bed [g].

Determination of bed sorption capacity

- The tank with the bed is pumped down to obtain a base vacuum of 1 Pa.
- Hydrogen is continuously introduced into the cooled tank through the mass flow regulator (recorder) until the flow self-disappears after the time tk (end of refuelling).
- Gravimetric hydrogen sorption capacity of the bed will be determined on the basis of the following equations:

$$m_2 = \int_0^{tk} q dt, \quad (4)$$

where: m_2 – a mass of hydrogen supplied to the tank [g], q – volumetric flow recorded on the mass flow controller [g/s].

Mass of hydrogen sorbed by the bed:

$$m_{H_2} = m_2 - m_G, \quad (5)$$

where: m_{H_2} – a mass of hydrogen sorbed by the bed [g], m_G – a mass of gaseous hydrogen in the tank [g] calculated from Eq. (4).

Gravimetric density is determined based on the equation:

$$\rho = \frac{m_S}{m_Z} \cdot 100\%, \quad (6)$$

where: ρ – gravimetric density [%], m_s – a mass of hydrogen absorbed by the bed [g], m_Z – a mass of the bed [g].

2.3. Working bed composition

Hydrogen sorption/desorption was realized due to the presence of a carbon-based working bed. It was the granulate of spatial graphene with metallic catalysts: lead and LaMischmetal (LaMMNi_{3.63}Al_{0.2}Mn_{0.4}Co_{0.75}La/SE 70%, heat treated – obtained from the Treibacher Industrie AG). Pb was added in the wet method. The granulate was mixed with an aqueous solution of PdCl₂ at acidic pH. After 60 minutes of intensive stirring, the catalyst – metallic Pb, was synthesized with the use of NaBH₄ as the reducing agent. After the drying, the material was mixed with powder LaMischmetal (size of particles was lower than 75 μm). Carbon-based working bed is characterized by the size of the pores in the range of 1.9–2.3 nm and the specific surface area of 340 ± 2 m²/g (the results of conducted measurements based on low-temperature nitrogen adsorption mechanism – not presented in that article). The overall mass of the working bed was 150 g of granulate of bulk density equal to 6.44 g/cm³.

2.4. The procedure of filling the working chamber of test bench with the analysed sorbing/desorbing bed

The analysed working bed is a material sensitive to atmospheric oxygen and prone to contamination. Reactivity for hydrogen of metal hydrides requires free access of that gas to the surface of the bed. All the corrosion processes or other ways of contamination can lead to the formation of firm layers *e.g.*, oxides, that significantly reduce the sorbing potential [13]. Therefore, the working bed was supplied in a form of suspension of the sorbing material in heptane. In the wet form, it was poured into a metal container mounted directly inside the empty space of the spiral coil of the heating/cooling system located in the working chamber of the test bench. After that, the system was sealed with a new copper gasket and the mounting bolts were tightened with the correct torque. Subsequently, the system was pumped down using an oil-free vacuum pump, resistant to chemical agents. The pumping procedure was conducted as long as the vacuum value indicated by the manometer was less than 1 Pa and did not increase with closing the valve of the pump. The sorbing bed was initially annealed at 180°C, under hydrogen overpressure for 1 hour. Then the container with the bed was cooled down to room temperature to enable the hydrogen sorption process. The heating and cooling procedures with releasing and supplying hydrogen were repeated at least 10 times to obtain a fully activated working bed with maximum and repeatable sorption/desorption capacity.

The proper research measurements of hydrogen sorption capacity were conducted three times and the results were averaged.

3. Results and discussion

Before the test, the tank was cooled down to 20°C, the hydrogen pressure was lowered to about 0.2 bar above atmospheric pressure, and heating to 140°C was started. After reaching the set temperature and pressure stabilization (at the level of about 9.5 bar), the cooling process was started. The recorded thermodynamic characteristics of the desorption/sorption cycle are shown in Figs. 3 and 4. Temp 1, Temp 2 and Temp 3 in Fig. 3 refer to temperature sensors in the top, middle and at the bottom of the tank, respectively (see Table 1).

As can be seen in Fig. 3, the pressure starts to increase simultaneously with increasing temperature. Initially, the linear increase is visible up to 1350 s of the test. After that, the pressure increase takes a logarithmic form. This means that the desorption process proceeds quite rapidly when the temperature increases, and after reaching the assumed temperature, the bed

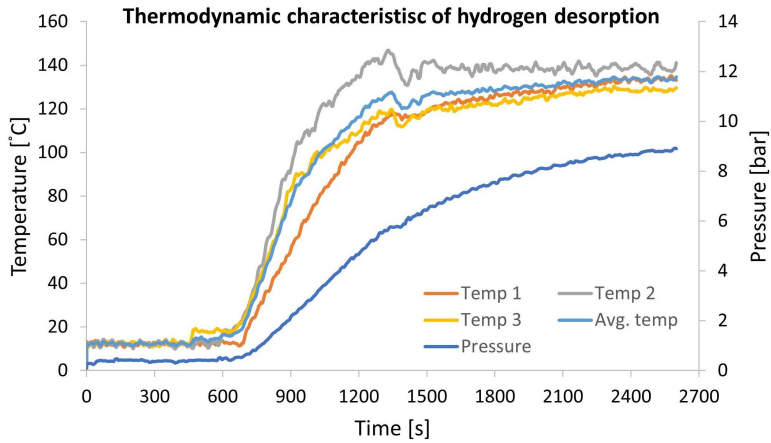


Fig. 3. Thermodynamic characteristics of the desorption process without releasing the hydrogen overpressure.

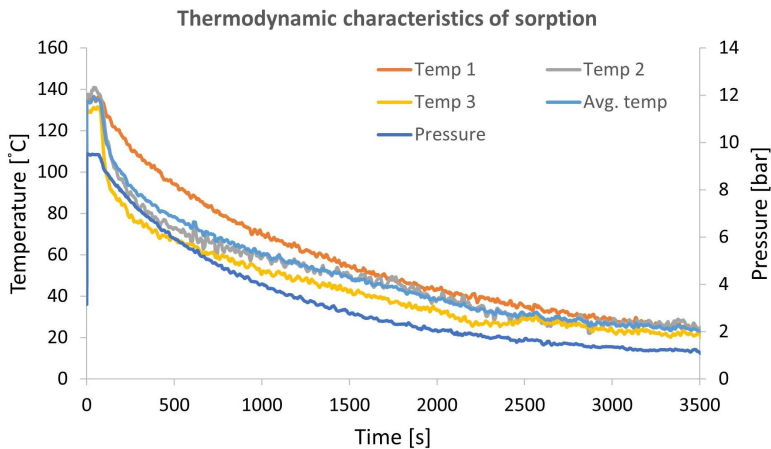


Fig. 4. Thermodynamic characteristics of the sorption process without supplying the hydrogen.

still slowly desorbs as the component of the increase in the pressure of gaseous hydrogen due to the increase in temperature is zero. The temperature distribution in the working chamber shows a slight difference between the particular sensors which is a problematic aspect mentioned in the literature [14]. Nevertheless, the temperature tends to stabilize and homogenize in time. Such slight variations of temperature show that sorption/desorption parameters of the bed are rather homogenous which positively testifies to the prepared working bed. During the cooling process, the pressure gradually decreases and after the test, it is slightly higher as compared to the value registered. This means that the sorption process is time-dependent and still in progress. Moreover, it confirms the tightness of the tank and other apparatus. The results of the final test aimed at the evaluation of the gravimetric density of the bed are presented in Fig. 5. To avoid misleading the reader, only averaged temperature is presented in the graph.

As stated earlier, the test was conducted with simultaneously releasing hydrogen overpressure to maintain the constant value of ca. 1 bar. In the graph there are visible fluctuations of pressure caused by the operation of the mass flow controller. Also, an interesting change can be found in the

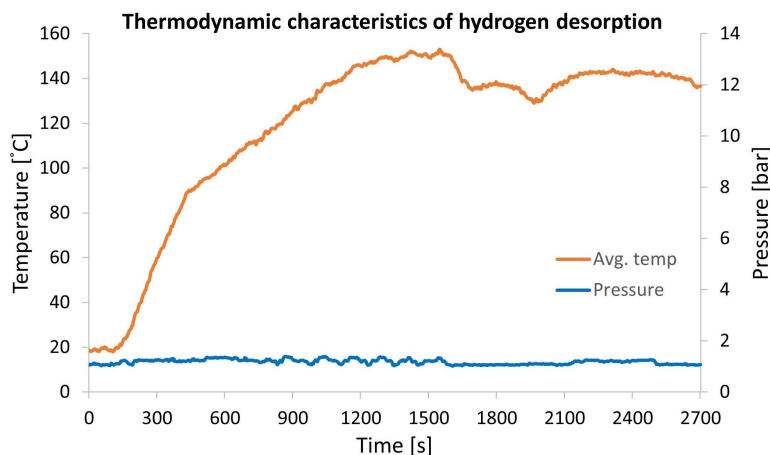


Fig. 5. Thermodynamic characteristics of the desorption process with releasing the hydrogen overpressure.

averaged temperature, which is caused by the constant releasing of hot gas and significantly lower pressure of hydrogen in the tank which undoubtedly affected the heat distribution as compared to the temperature trend in Fig. 3 (and 9.5 bar of hydrogen).

The observed increase in the hydrogen pressure caused by the temperature increase ($\Delta T = 120^{\circ}\text{C}$) in comparison with the heating of the container without the bed was equal to 0.512 bar. Knowing the mass of hydrogen removed from the tank mH and the mass of gaseous hydrogen in the tank mG (1.62 g and 62.3 mg, respectively), the calculated gravimetric density based on the presented equations (Section 2.2 Measurement Methodology) was equal to 1.04%. The obtained value proves the sorption potential of the working bed material and the adequacy of the installation described above. It is also worth mentioning that the proposed loading system involving a powdered bed immersed in liquid hydrocarbon prevents possible deterioration of the bed due to contamination by moisture and oxygen from air, which is one of general problems with metal hydrides [15].

4. Conclusions

Based on the obtained experimental results, it can be concluded that the apparatus for the analysis of hydrogen sorption/desorption characteristics has been successfully designed and constructed. It was mainly developed for the examination of working beds operating at low pressures (up to several bars) and temperatures (up to 200°C). The presented system can be easily modified for direct implementation in the automotive industry of forklift trucks. The designed test bench was validated and functionally tested giving the expected results. It was also shown that the construction of the test bench makes it possible to analyse and easily exchange oxygen-sensitive working beds without the risk of their oxidation or contamination. That feature is of great interest in the case of usage of the powdered form of metal hydrides as a working bed. The conducted experiments confirmed the sorption/desorption ability of the analysed working bed (at the level of gravimetric density equal to about 1%) and reliable and reproducible results have been obtained. The proposed apparatus can be used in further studies on the development and optimization of sorbing/desorbing agents in terms of functional requirements for widely understood hydrogen storage technologies.

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