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Desorption of protium and deuterium from different types of titanium beds

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ABSTRACT

When the long term tritium storage is intended, metal hydride materials, particularly the titanium (Ti) beds, seems to be the recommended option, due to its compliance with the criteria of selection (e.g. material cost, stability, storage capacity, loading and unloading conditions, or radioactivity). However few experimental and numerical analyses have been published so far to better support the understanding of the recovery capabilities for different forms of titanium beds. In this work, an investigation on the recovery of different hydrogen isotopes from two types of titanium (Ti) beds, namely Ti powder and Ti sponge, has been performed. Hydrogen isotope release was experimentally verified up to a temperature of 600 °C for both Ti powder and Ti sponge beds. The desorption percentages were determined to be from 24.98 to 20.54 in the case of D_2 on Ti sponge, and from 34.36 to 29.77 in the case of H_2 on Ti sponge. The paper describes in detail the experimental set up, the measurements and the drawn conclusions.

1. Introduction

With a low explosion limit and being the smallest molecule, safe storage of hydrogen gas is not an easy technical issue. In this perspective, the storage of hydrogen and its isotopes as solid metal hydride can be more advantageous over other conventional techniques like gaseous storage in high pressure tanks or liquid storage in cryogenic tanks. Thus, the scientific literature [1,2] highlights a series of specific features and parameters for various materials applicable to tritium storage (e.g. uranium, palladium, titanium and zirconium alloys based $LaNi_5$).

The selection criteria for long term storage, in view of Ti future use are the radioactivity, air stability in the material storage, storage capacity, storage pressure, loading conditions and especially the discharge, retention of helium-3 resulted from the decay of tritium, etc. Both the US Department of Energy (DOE) [1,3] and the Canadian Nuclear Safety Commission (CNCS) [4] recommend titanium for long term storage of tritium.

However, for this purpose, few experimental and numerical analyses have been published to assist us in better understanding of the recovery capabilities for different forms of titanium (Ti) beds or other storage beds [5–8]. In this regard, the present work provide experimental conditions and data on desorption of protium and deuterium, from two forms of Ti beds, namely powder and sponge, under the same working conditions. As the research has been executed in a non-nuclear laboratory, only the release of protium and deuterium was tested.

2. Experiment presentation

The installation set up is shown in Fig. 1, presenting the main mechanical and electrical/automation components of the experimental configuration. The reactor (R), which contains the Ti bed under investigation [9], represents the core of the test assembly. It is inserted in an oven having the capability for temperature controlling, between ambient temperature (approx. 25 °C) and 600 °C.

A vacuum train assures the vacuum conditions throughout the whole experimental setup. The various elements or devices are bound together by stainless steel tubes via valves that are leak tight down until a vacuum of 10^{-5} mbar L/s is reached.

To follow the evolution of the experiments, a data acquisition system was used to track the specific parameters (e.g. temperature, pressure).

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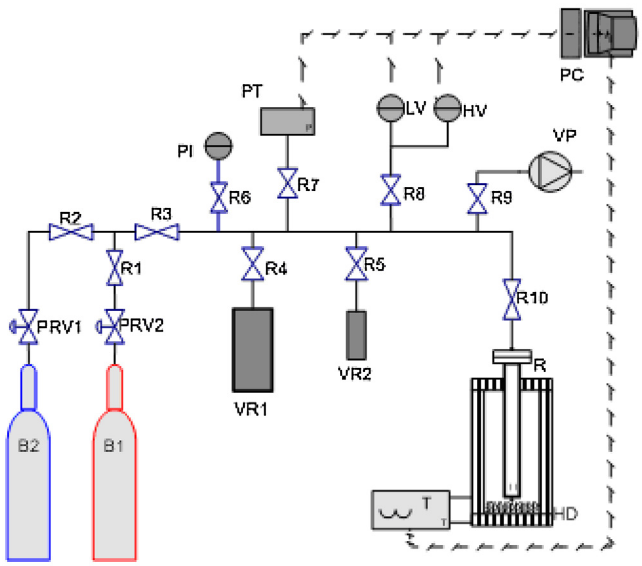

Fig. 1. Hydrogen isotopes storage & recovery experimental installation. Specific components: PRV1-2 – pressure reducing valves, R1-10 – automatically isolating valves, B1 – hydrogen tank, B2 – deuterium tank; VR1 – metering vessel 1, VR2 – metering vessel 2, LV – low vacuum gauge, HV – high vacuum gauge, PT – pressure transducer; PI – pressure indicator; VT – high vacuum train; R – reactor; HD – heating device.

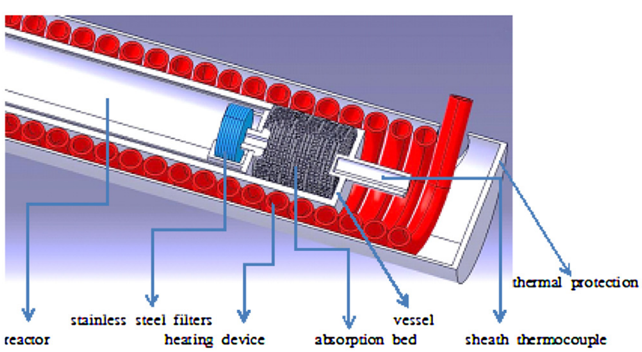

Fig. 2. Section view through the experimental reactor.

2.1. Reactor and heating design

As already mentioned, the main element in the experimental configuration is the reactor that holds the Ti beds under investigation. A detailed cross section of the reactor inserted in the oven is presented in Fig. 2. It is made from stainless steel 316-L and has a cylindrical form with a length of 500 mm. Its length is specifically set so that the Ti bed to be positioned in the middle of heating section of the electrical oven.

The reactor geometrical dimensions (diameter of 26 mm and the height of reserved space for storage bed of 30 mm) were setup based on the necessary amount of metal bed (7.2 g) to be accommodated inside. Inside the reactor a stainless steel filter is installed just above the titanium bed. This is meant to prevent the movement of small metal powdery out of the tank. This powdery could be formed during activation processes or after repeated heating and cooling cycles [10]. A tube with a length of 1 m connects the reactor to the vacuum pumps.

The heating power of the oven was designed to heat up the reactor and its contents from ambient temperature to 600 °C, in less than one hour. A maximum threshold temperature of 600 °C was chosen for the experiments because a stainless steel capable to sustain hydrogen corrosion and diffusion at that temperature is seldom used in industrial nuclear applications since it is very difficult to obtain [11].

Table 1
Data on desorption experiment series.

Pressure/experiment	Protium		Deuterium	
	Powder	Sponge	Powder	Sponge
2 kPa(a)	ExP1-p	ExP1-s	ExD1-p	ExD1-s
20 kPa(a)	ExP2-p	ExP2-s	ExD2-p	ExD2-s
40 kPa(a)	ExP3-p	ExP3-s	ExD3-p	ExD3-s
60 kPa(a)	ExP4-p	ExP4-s	ExD4-p	ExD4-s
80 kPa(a)	ExP5-p	ExP5-s	ExD5-p	ExD5-s

ExP – experiment with Protium, number 1; p – powder; s – sponge; ExD – experiment with Deuterium; 1–5 – experiment number.

2.2. Preparation of the titanium beds

For this experiment two types of titanium beds were selected: titanium sponge and titanium powder. Each of them was evaluated in separate tests, as part of the experiment. The sponge form of Ti metal was chosen due to its large available surface area [12]. Also we considered the Ti powder, as second alternative, despite the safety concerns regarding the easy dispersion of small metal tritide particles, possible to be formed during the loading process. To overcome these concerns a proper filtering system was installed at the reactor interface with the gas supply line.

Titanium sponge of 3–5 mm in size and Ti powder, purchased from Merck, were used for experiments. No subsequent cleaning procedures were applied. The analytical results provided by the supplier showed that the main metal impurities were magnesium, iron, nickel and chrome, even if chlorine is known as a major impurity in titanium sponge [13].

However, prior to their usage both sponge and powder Ti bed were activated.

At room temperature titanium does not react with hydrogen because its surface is covered with a protective layer of oxide, but under vacuum conditions, by heating to a high temperature, it can be activated [12]. The activation procedure causes dissolution of the oxide into bulk of the sponge so that clean titanium surfaces are available for hydriding. Evacuating the resulting gases during heating process, by the use of vacuum train, will remain clean metal surfaces, ready-to adsorb any surrounding gases.

2.3. Loading of the beds

The loading of hydrogen isotopes during the experiment was done after performing the proper activation procedure and cooling the reactor down to room temperature. Protium or Deuterium fills in the vessels VR-1/VR-2, by opening the pressure reducing valves PRV-1/PRV-2, at a pressure depending on the gas quantity desired to be stored. Then, the gas from VR-1/VR-2 enters into the reactor R, after opening a series of other valves (Fig. 1).

Transducers, PI or PT, measure and indicate the pressure obtained inside of vessels VR-1/VR-2. When the Ti bed is very well activated the adsorption reaction is exothermic, almost instantaneous [9,14]. If the adsorption bed is close to saturation, then the remnant pressure is bigger. Table 1 shows the established remnant pressure of the beds (Ti powder and Ti sponge) after various controlled loadings.

3. Results

For desorption process experiments, the storage bed from the reactor (R) was filled until the remnant pressure reached the values presented in Table 1.