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Development of hydrogen absorption–desorption experimental test bench for hydrogen storage material

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ABSTRACT

A volumetric experimental set-up used for measuring hydrogen absorption–desorption characteristics of hydrogen storage material will be presented. Although the experimental set-up is mainly employed to do hydrogen absorption–desorption cycling (including pressure cycling and thermal cycling) measurement automatically, it also can incidentally provide general measurements such as pressure-composition-temperature (P–C–T) curves and kinetics measurements in manual way in the ranges of 0.004–12 MPa and 213–773 K. The experimental set-up can be used to investigate the influence of hydrogen absorption–desorption cycles to hydrogen storage properties of material. The leakage rate of the whole experimental set-up was evaluated systemically. The usability and reliability of the experimental set-up were checked with $LaNi_5$ and Pd/K (kieselguhr).

Introduction

The world problems about energy crisis and environmental pollution have become the most pressing political issues of our time and are urging many countries to develop new cleaner energies to supply our increasing demand [1]. Among the new energy technologies, using fusion energy to generate electricity would have a lot of advantages [2]: (1) No carbon discharge. The only by-products of fusion reactions are inert helium gas, which will not make any pollution. (2) Affluent fuels. Deuterium is made from water and tritium is produced from lithium. The sources are so abundant in the earth that these two fusion materials can continuously supply for millions of years. (3) High energy density. 1 kg fusion fuel can yield the same amount of energy as about 1×10^7 kg fossil fuel. (4) No long lived radioactive waste. The fusion power generation only induces some plant components become radioactive. However, the radioactivity of these components will decrease to below safety margin within about 100 years. (5) Good safety. The large scale nuclear accident is almost impossible because only a few fuels are used in fusion devices.

Hydrogen has three isotopes: protium, deuterium, and tritium. Among these, deuterium and tritium are used in fusion power for electrical generation, which requires the separation of deuterium and tritium in high purity. Because isotopes have very similar chemical properties, isotope separation is, in general, more difficult than chemical separation. Several

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Nomenclature

n	amount of hydrogen atoms, mol
m	sample mass, g
M	molecular weight of sample, g mol^{-1}
ρ	molar density, mol l^{-1}
$p_{a,min}$	minimum hydrogen pressure in absorption, MPa
$p_{d,max}$	maximum hydrogen pressure in desorption, MPa
$x_{a,max}$	maximum H/M ratio in absorption, dimensionless
$x_{d,min}$	minimum H/M ratio in desorption, dimensionless
T_L	the lowest temperature of sample container in absorption, K
T_H	the highest temperature of sample container in desorption, K
$T_{PR,a}$	temperature of pressure reservoir when pressure reaching $p_{a,min}$, K
$T_{PR,d}$	temperature of pressure reservoir when pressure reaching $p_{d,max}$, K
V_{PR}	volume of pressure reservoir, l
V_{SC}	volume of sample container, l
V_S	volume of sample, l

Subscripts

PR	pressure reservoir
S	sample
SC	sample container
eq	equilibrium
a	absorption
d	desorption

technologies have been developed to separate hydrogen isotopes. These technologies include thermal diffusion, cryogenic distillation fractional absorption, elution chromatography, permeation through palladium alloy membrane, and thermal cycling absorption process (TCAP) [3]. Among them, the TCAP, based on metal hydride, is an efficient and reliable process for separating hydrogen isotopes. However, TCAP requires hydrogen absorbing metals or alloys to repeat thermal absorption–desorption process for thousands of times, which may cause pulverization due to the expansion and shrinkage of metal particles during cycles and result in a decrease in hydrogen absorption capacity and rate. Besides, the hydrogen compressor based on reversible metal hydrides also includes absorption at low temperature and desorption at high temperature, however, with fresh hydrogen charging and discharging in every cycle [4,5]. And hence, the efforts to optimize the alloy composition and learn the intrinsic and extrinsic mechanisms of degradation of different kinds of alloys with specific application are necessary. Besides, the pulverization process leaded by hydrogen absorption–desorption cycling also can be used to make fine metal powders with high quality and low cost [6].

In our laboratory, an all-purpose volumetric experimental set-up has been developed to supply services for most application development related to hydrogen storage material. This set-up can automatically submit alloy samples to up to many thousands of hydriding and dehydriding cycles, which can simulate the working conditions similar to hydrogen isotopes separation or hydrogen compression. Besides, measurement of the basic data, i.e. P–C–T curves and kinetics of hydrogen storage material in manual way, as well as production of fine metal powders can carry through on this device.

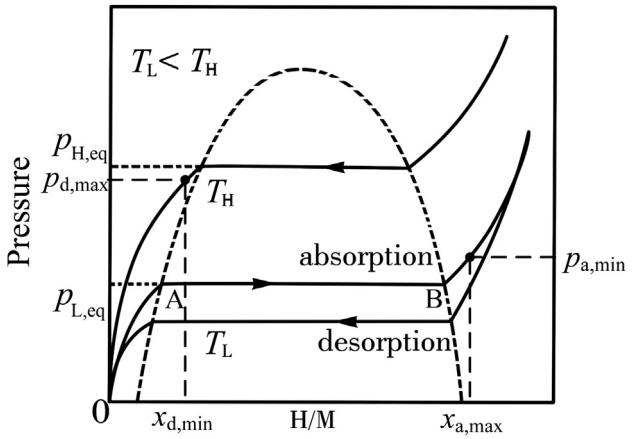

Fig. 1 – P–C–T curves of hydrogen storage alloy.

In two previous papers [7,8], we introduced two similar volumetric apparatuses. However, the first volumetric apparatus [7] was not so compacted and so efficient, and was mainly for measuring P–C–T curves or kinetics, and pressure cycling simultaneously, while the present was mainly for thermal cycling or pressure cycling experiment. The previous apparatus [7] could not complete thermal cycling experiment, because (1) the sample cell was immersed in thermostatic oil bath, (2) no appropriate volume control reservoirs were connected to the second section, and (3) no specific theoretical calculation formulas about volume control and hydrogen content difference between absorbed and desorbed states were provided. The second paper [8] only introduced the method of applying SolidWorks software to do 3D virtual structure design of a very simple P–C–T measuring apparatus with use of manual ball valves and VCO connectors. The point was how to use SolidWorks software to complete designing this kind of apparatus. We thought that 3D virtual structure design was vital to achieve structure optimization for this kind of apparatus, although we did not change this simple design into real form.

Principle of the experimental apparatus

The absorption–desorption P–C–T curves and kinetics are measured by this set-up based on the principle that a closed and sealed system contains hydrogen either in gaseous state or dissolved within the hydrogen storage material [9]. If the volume is calibrated precisely, the gaseous amount can be calculated by measuring the respective temperatures and pressures and using a real gas equation of state. In our related measurement, the newest Helmholtz-type hydrogen gas equation of state (EOS) recently recommended by NIST (National Institute of Standards and Technology, USA) is used [10]. Compared with the MBWR EOS, the present EOS covers wider temperature and pressure range. The methods about volume calibration