Sievert-type measurement and acquisition system for the study of hydrogen storage in solids

Jorge Runco, Marcos Meyer
IFLP – Departamento de Física – Facultad de Ciencias Exactas – UNLP
CONICET
{ runco, meyer }@fisica .unlp.edu.ar

Abstract
This paper shows the development and implementation of a system to determine and analyze parameters of interest in the study of hydrogen absorption and desorption mechanisms in solids using the Sievert volumetric method. The experiment is controlled through a PC-type computing system by automatically measuring, controlling, recording and graphing the evolution of variables (pressures and temperatures) according to a set of previously programmed parameters. The manual monitoring, adjustment and operation option is also available to tune the experiment. The software was developed in a high-level programming language (Delphi) which offers the user a graphical interface typical of visual languages. In addition, results for applying the present system to typical ternary hydrides are presented.

Key words: hydrogen storage, processes, absorption and desorption, data acquisition.

1. Introduction

One of the most important challenges for the development of hydrogen utilization as an energy vector is the possibility of storing it in a safe and effective way [1].
Hydrogen in a gaseous state occupies a very large volume and requires very high pressures in storage reservoirs whereas, in a liquid state, it needs reservoirs at very low temperatures.
Since hydrogen is highly reactive, there is a significant number of elements capable of reacting with it to form hydrides under appropriate pressure and temperature conditions. Hence, hydrogen storage in a solid state appears as a more effective alternative in terms of volume with respect to the other methods mentioned above.
Absorption in metals, which forms a hydride phase, has many advantages over current systems (compression and liquefaction), since it does not require compressing and liquefying tasks or cryogenic temperatures.
An important aspect in experimental research is the analysis of hydrogen absorption-desorption properties of new materials, as well as the study of absorption and desorption kinetics.
As considerable pressure changes involve relatively small mass quantities, using volumetric techniques is especially suitable for this kind of research (mainly considering that hydrogen is the lightest element of all).

2. Measurement system description

The equipment is based on a Sievert-type system [2], which allows studying hydrogen absorption-desorption kinetics at different temperatures, keeping pressure level constant in the reaction chamber, in a wide range of temperatures (from 300 K to 1000 K) and pressures (from 1 mbar to 50 bar).

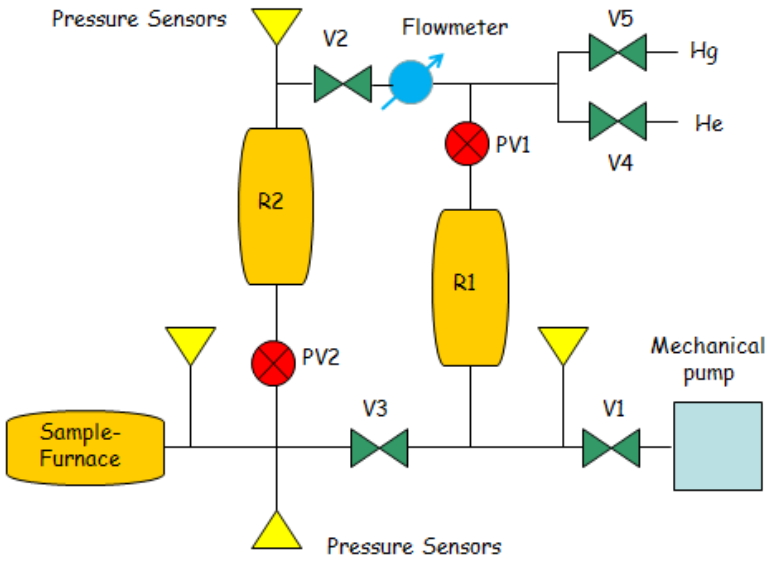

Figure 1. Schematic diagram of the Sievert apparatus.

The measuring instrument was developed based on a PC-type computing system which incorporates a 12-bit general purpose A/D interface [3], 5 inputs of which are used to measure analog magnitudes –4 for pressures and 1 for temperature. It also has digital outputs for the automatic opening and closing of the 7 solenoid valves (V1, V2, V3, V4, V5, PV1 and PV2) of the measurement system. The schematic diagram above illustrates the location of pressure sensors [4] in the experiment as well as valves automatically actuated by the control system.

The measurement system has a furnace and a temperature controller to conduct the experiment at different sample temperatures. This controller communicates with the computer by means of an RS-232 interface and MODBUS protocol [5].

A flowmeter was added to the system to control and measure gas flow during the experiment. This device communicates with the computer also through an RS-232 interface and the MODBUS protocol.

The software developed makes it possible to open and close solenoid valves at any desired time, to acquire and store parameters of interest –pressures, temperature and flow– and to preset temperature and flow values (setpoints) before starting the experiment. In addition, routines necessary to communicate with furnace and gas flow controllers through the MODBUS protocol were implemented.

During the course of the experiment, the abovementioned parameters are acquired and stored, valves open and close when pressure conditions set before starting the measurement are met or for controlling gas flow with the flowmeter, and the system is capable of performing the experiment at a constant temperature, with linearly increasing temperature (temperature ramp), when cooling, or with a combination of these stages.

Figure 2 shows the interconnection between the different modules and components. The Signal Adaptation Electronics module is in charge of conditioning (instrumentation amplifiers) signals coming from sensors at the input of the A/D converter. Moreover, this module is equipped with the circuits needed to actuate the valves with digital signals since they operate with 220 V. Isolation between this voltage and the computing system is required; this is accomplished with optically-coupled circuits.

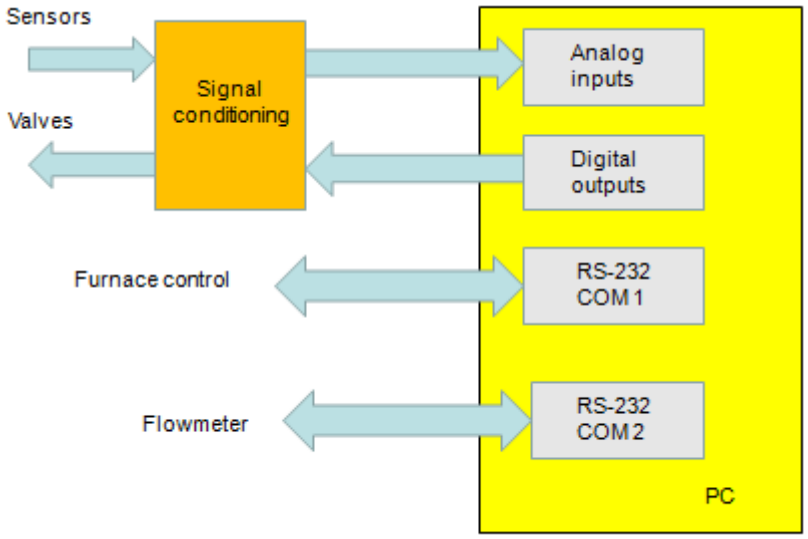

Figure 2. Acquisition and control diagram.

3. Results

The equipment described above was used in several experiments for studying the kinetics of absorption and desorption to form typical ternary hydrides.

Figure 3 displays temperature and evolution of the material's pressure when releasing hydrogen according to time. When preset pressure conditions are met, valves automatically open and close between these two values. When hydrogen is released, pressure increases up to a certain value, hydrogen is discharged, pressure decreases, and the cycle is repeated until the sample releases all the hydrogen. Figure 4 illustrates the analytical treatment of measured data.

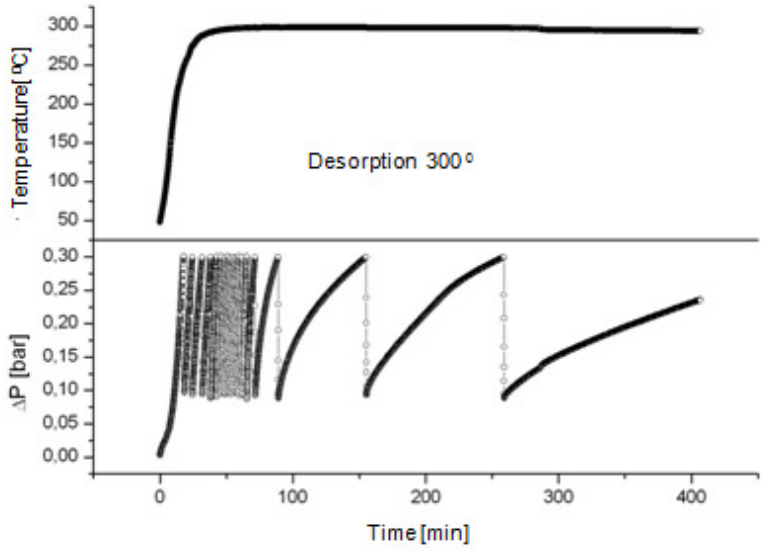

Figure 3. Temperature according to time. Sample desorption. Pressure variation according to time.

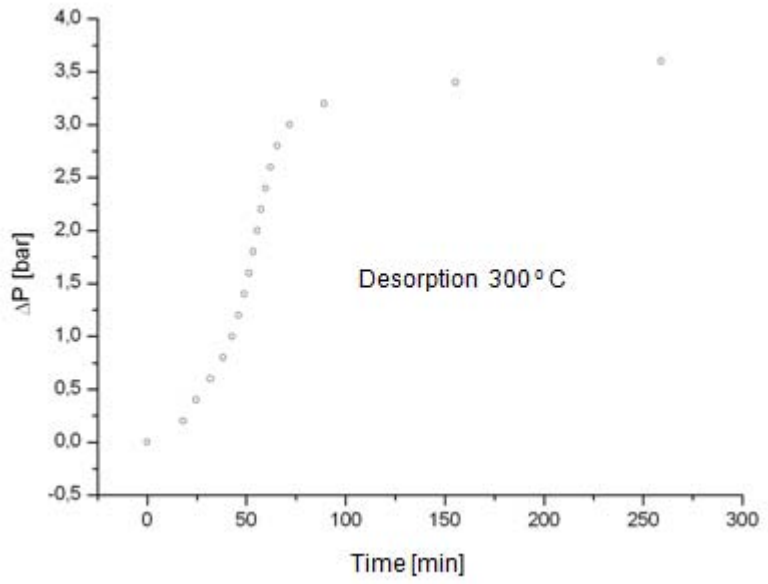

Figure 4. Desorption data treatment.

4. Conclusions

This paper describes the development and implementation of a piece of equipment for studying hydrogen absorption and desorption in metals. Having used commercially available components, the importance of this proposal lies in its low cost.

The design complies with the requirements specified by a research group from the IFLP (Instituto de Física de La Plata [***La Plata Physics Institute***] - CONICET [***for its Spanish acronym, National Council of Scientific and Tehnical Research***), Departamento de Física, Facultad de Ciencias Exactas, UNLP [***Physics Department, Faculty of Exact Sciences, National University of La Plata***]) that is part of the project "Materiales nanoestructurados de aplicación en energías alternativas: síntesis, caracterización y modelado" [***"Nanostructured materials applicable to alternative energies: synthesis, characterization and modeling"***].

The design currently in operation went through several stages: from the development and implementation of the signal adaptation module, to the software that controls and automates the experiment, up to the present situation with variations from the original experiments: constant temperature test, with (linear) increase, acquisition during cooling and gas flow measurement and control.

5. References

[1] Optimización de un hidruro complejo para almacenamiento de hidrógeno. Ph.D. thesis. June, 2009 – Cardozo, César Luis - Centro Atómico Bariloche [***Bariloche Atomic Center***].

[2] R. Checchetto, G. Trettel and A. Miotello. 2003 – Sievert-type apparatus for the study of hydrogen storage in solids. Measurement Science and Technology.

[3] Conversor A/D- ADQ-12 – Microaxial

[4] Druck-PDCR 4000 Series – High Performance Millivolt Output Pressure Transducers. Motorola - MPX2200 Series – On-Chip Temperature Compensated & Calibrated Pressure Sensors.

[5] MODBUS Protocol Specification - http://www.modbus.org.