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Research Article

Exothermic Phenomena in Hydrogen Desorption Experiments Using Pd-Ni Samples

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Abstract

In this study, we conducted a hydrogen desorption experiment using a Pd foil sample coated with a Ni membrane to investigate its thermal behavior and hydrogen diffusion characteristics. Two heating methods were employed: direct heating and indirect heating. In the direct heating experiments, transient heat generation was observed 0.5–1.0 hours after applying constant power, accompanied by temperature changes associated with hydrogen release. Comparison with unabsorbed samples suggested that the excess heat was estimated to be up to several hundred milliwatts. On the other hand, in the indirect heating experiments, heat generation was rarely observed, but a heat generation phenomenon was confirmed when a constant power of 1 W was applied. These results suggest that anomalous diffusion during the hydrogen desorption process may induce heat generation, indicating the need for further confirmation of reproducibility and higher-precision evaluation of excess heat.
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Keywords: H2-gas, Cold fusion, Metal complex, Nano-structure, Thin film.

1. Introduction

Several previous studies on hydrogen/deuterium absorption and desorption in Pd-Ni and Cu-Ni systems have reported the generation of excess heat [1]–[9]. These studies highlighted the following conditions as necessary for such anomalous phenomena:

- Formation of metal complexes (e.g., composite particles, multi-layer structures)
- Presence of nano-structures (e.g., nano particles, thin membranes)

Considering these conditions, we conducted a hydrogen desorption experiment using a Pd foil sample coated with a Ni membrane to investigate its thermal behavior and hydrogen diffusion characteristics. The sample was heated to promote hydrogen desorption, employing two heating methods:

- Direct heating: The heater was in direct contact with the sample.
- Indirect heating method: The sample was fixed to a metal plate, and a heater was used to heat the metal plate. Heat was then transferred from the plate to the sample.

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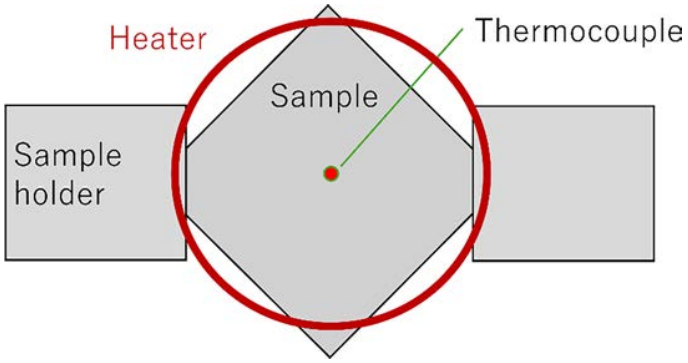


Figure 1. Schematic of direct heating.

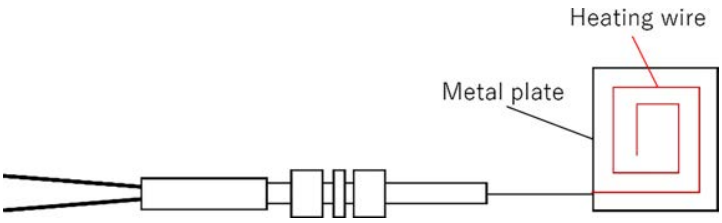


Figure 2. Schematic diagram of indirect heating.

2. Experiment

2.1. Sample

The sample material consisted of a Pd foil (10 × 10 × 0.1 mm) and a Ni membrane (t = ∼100 nm). First, the Pd foil was washed with acetone and annealed at 900 °C for 10 h. Subsequently, the annealed Pd foil was washed with aqua regia to remove any metallic impurities from its surface. A fine structure was formed on the surface of the Pd foil by etching with an Ar+ beam. Subsequently, an Ni membrane was deposited on the etched surface via Ar+ sputtering.

2.2. Hydrogen Loading

A schematic of the experimental setup is shown in Fig. 4.

The Pd-Ni sample was placed in a stainless-steel cell, which was filled with hydrogen gas (purity: 99.99999% or higher) at 5 atm and pressurized for approximately 20 h. The weight of each sample was measured before and after loading, and the loading ratio (H/Pd) was calculated from the weight difference. The electronic balance used in this experiment was a GH-202(A&D Co., Ltd.), with a minimum display of 0.01 mg and a standard deviation of 0.02 mg.

When calculating the loading ratio from the weight, the thin film was considered to have a negligible amount of absorbed hydrogen because its thickness (100 nm) is much smaller than that of the substrate (0.1 mm).

In addition, the gas released between removing the sample from the absorption apparatus and weighing it is not taken into account in the calculation of the loading ratio.

The absorption experiments were repeated 17 times, yielding an average H/Pd ratio of 0.72.

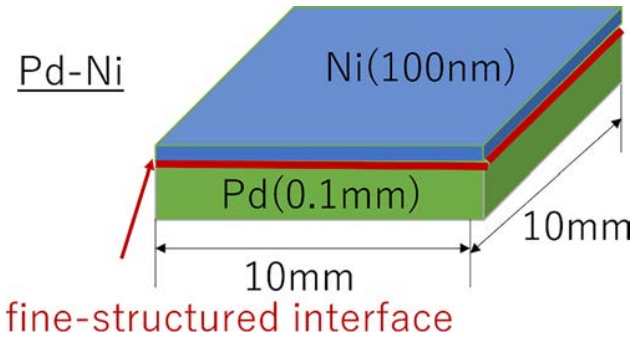


Figure 3. Schematic diagram of sample.

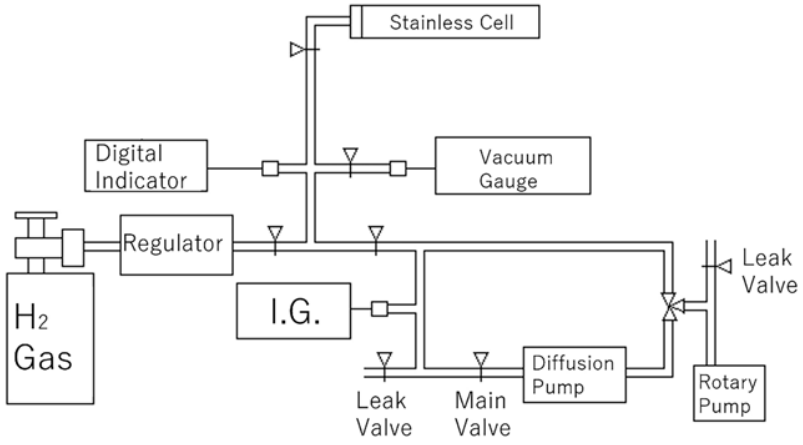


Figure 4. Schematic of the absorption experimental setup.

2.3. Hydrogen Desorption

A schematic of the experimental setup is shown in Fig. 5. The volume of the vacuum chamber in the desorption apparatus is approximately 1.098 L. The sample was placed into a chamber equipped with a TMP, which could be evacuated to $\sim 10^{-4}$ Pa. Inside the chamber, the sample was heated by applying constant power to the heater to stimulate hydrogen diffusion. The sample temperature and chamber pressure were continuously monitored for approximately 24 h. A thermocouple was used to measure the temperature, and an ionization gauge was used to measure the chamber pressure. The voltages and currents applied to the heater were recorded during the experiments.

2.4. Blank Experiment

After the desorption experiment, the samples were treated as hydrogen-free for subsequent experiments. The experiment was conducted under the same conditions as the desorption experiment, and the sample was heated for 20 h using the heater.

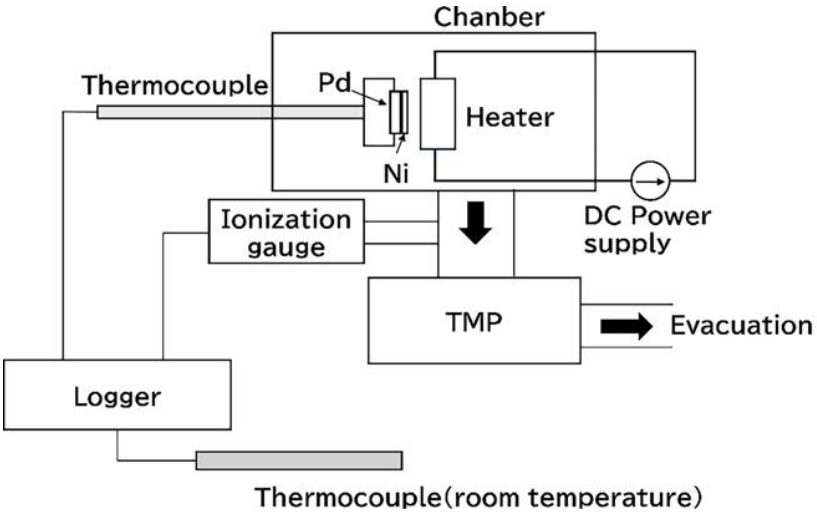


Figure 5. Schematic of the experimental setup.

2.5. Calibration

The power supplied to the heater varied every two hours during the experiment. For example, when 1.2 W was supplied to the heater in the desorption and blank experiments, the output was increased incrementally to 1.3 W, 1.4 W, 1.5 W, and 1.6 W, using 1.2 W as the reference. The corresponding temperature was measured for each power setting, and a calibration curve of temperature versus supplied power was constructed.

3. Results

3.1. Experimental Result 1: Hydrogen Desorption By Direct Heating

The experiments were conducted using a direct heating system. Fig. 6(a) shows the time dependence of the sample temperature and chamber pressure during the desorption experiment (Pd(Ni)-2024-09). Fig. 6(b) presents an enlarged plot of the initial 24 h, and Fig. 6(c) provides an enlarged plot of the initial 4 h of the experiment. Similarly, Fig. 7(a) shows the time dependences of the sample temperature and chamber pressure during the desorption experiment (Pd(Ni)-2024-10), and Fig. 7(b) depicts an enlarged plot of the initial 24 h. Furthermore, Fig. 7(c) shows an enlarged plot of the initial 4 h.

The data obtained from 0 to 24 h corresponded to the desorption experiment, from 28 to 48 h to the blank experiment, and from 52 to 74 h to the calibration experiment. In the desorption phase, transient heat generation was occasionally observed between 0.5 and 1.0 h following the application of constant power. Concurrent pressure variations correlated with the temperature changes were also recorded. These results suggested a possible increase in the surface temperature of the sample during hydrogen release. The unique conditions of the sample may have induced specific hydrogen diffusion, causing an exothermic reaction. This phenomenon was observed in approximately 4 out of 6 trials.

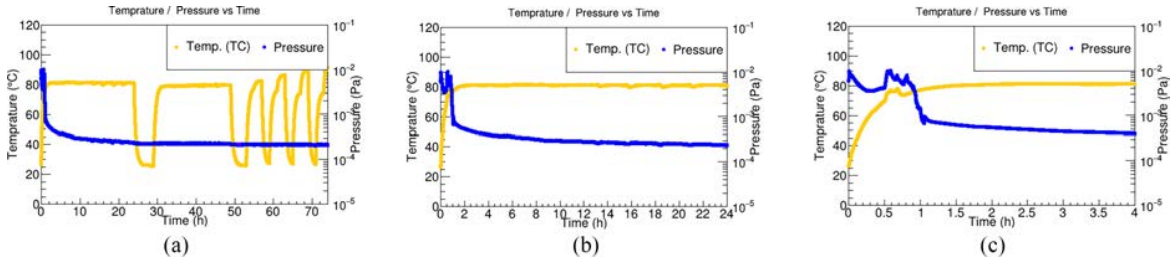


Figure 6. (a) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-09) over 74 h. (b) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-09) over 24 h. (c) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-09) over 4 h.

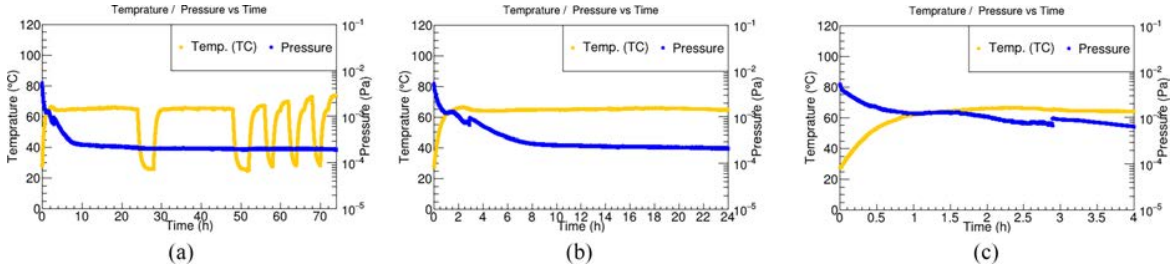


Figure 7. (a) Time dependence of sample temperature and chamber pressure within the chamber for the experiment (Pd(Ni)-2024-10) over 74 h. (b) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-10) over 24 h. (c) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-10) over 4 h.

3.2. Experimental Result 2: Hydrogen Desorption by Indirect Heating

The experiments were conducted using an indirect heating system. Fig. 8(a) shows the time dependences of the sample temperature and chamber pressure during the desorption experiment (Pd(Ni)-2024-13). Fig. 8(b) presents an enlarged plot of the initial 24 h, and Fig. 8(c) shows an enlarged plot of the initial 4 h of the experiment. Similarly, Fig. 9(a) shows the time dependence of the sample temperature and chamber pressure during the desorption experiment (Pd(Ni)-2025-03). Moreover, Fig. 9(b) depicts an enlarged plot of the initial 4 h, Fig. 9(c) shows an enlarged plot of the sample temperature during the initial 4 h of the experiment.

The data from 0 to 24 h correspond to the desorption experiment, from 28 to 48 h to the blank experiment, and from 52 to 74 h to the calibration experiment. In the experiments using an indirect heating system, heat generation phenomena were rarely observed. Pressure fluctuations associated with hydrogen release were recorded. Notably, heat generation associated with hydrogen release was observed when a constant power of 1.0 W was applied. Further investigations are required to clarify the specific conditions under which hydrogen-related heat generation occurs. This phenomenon was observed in approximately 3 out of 11 trials.

4. Summary

Hydrogen desorption experiments were conducted using a sample consisting of a Pd substrate coated with a nickel layer. Two heating methods were employed: direct and indirect. In the using direct heating experiments, heat generation was observed between 0.5 and 1.0 h after the application of constant power. This heat generation was potentially associated with hydrogen desorption. Furthermore, the specific conditions of the sample induced anomalous diffusion

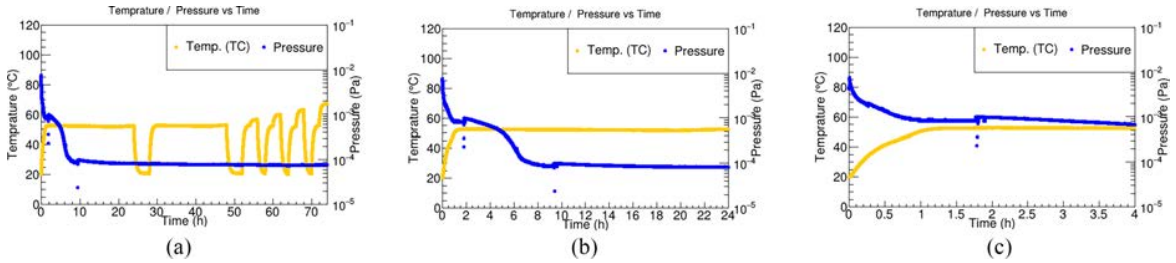


Figure 8. (a) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-13) over 74 h. (b) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-13) over 24 h. (c) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2024-13) over 4 h.

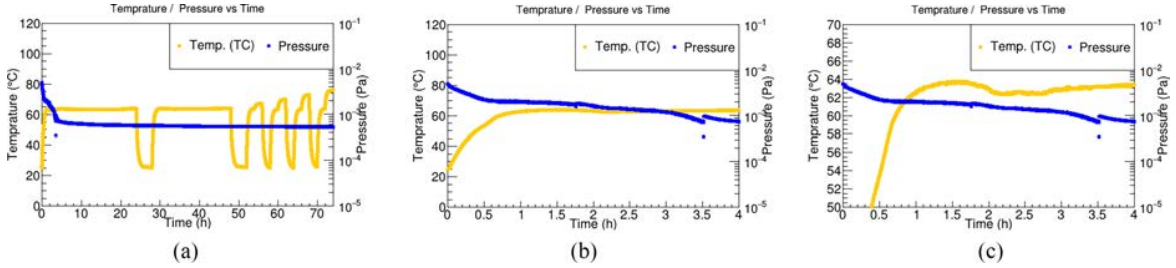


Figure 9. (a) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2025-03) over 74 h. (b) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2025-03) over 4 h. (C) Time dependence of sample temperature and chamber pressure for the experiment (Pd(Ni)-2025-03) over 4 h (enlarged view of the sample temperature).

behavior of hydrogen, which in turn triggered an exothermic reaction. Excess power was estimated by a comparison with the temperature measured for the unloaded sample, yielding preliminarily values up to several hundred milliwatts. In contrast, in the indirect heating experiments, heat generation observed in the case of direct heating was rarely detected. However, when a constant power of 1 W was supplied, heat generation phenomena, possibly related to hydrogen desorption, were occasionally observed. Experiments are ongoing to confirm heat generation and improve the accuracy of excess heat evaluation.

Acknowledgement

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