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Research Article

Heat Measurement in Hydrogen Absorption into Metal Composite Powder and Thin Films

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Abstract

Recent studies have reported excess heat generation during hydrogen absorption–desorption cycles in Cu–Ni composite metals. It has also been suggested that laser irradiation of the sample can enhance the reactions of hydrogen within metals. Considering these findings, we investigated the thermal behavior of hydrogen-loaded Cu–Ni alloys supported on zirconia (CNZ). In absorption experiments with a heater input of 50 W, maximum excess heat of 16 W was observed. Furthermore, when laser irradiation at a wavelength of 793 nm with an output power of 1–6325 mW was applied to the CNZ sample, the excess heat calculated from the sample temperature was positive, whereas that derived from the gas temperature was negative. In addition, hydrogen desorption experiments using Pd–Ni multilayers deposited on a zirconia substrate were carried out. The results showed that excess heat occasionally became negative depending on the heater power, indicating poor reproducibility.
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Keywords: H2-gas, Laser irradiation, Cold fusion, Thin films, Powder

1. Introduction

In a study of low-energy nuclear reactions in condensed matter, hydrogen absorption in copper-nickel powder with zirconia as the support (CNZ) has been reported to generate excess heat 100–1000 times higher than that expected from the most energy dense chemical reaction [1]–[4]. This suggests new physical phenomena that cannot be explained by the reaction processes in conventional metal–hydrogen systems. In this study, experiments were conducted to confirm the reproducibility of this heat generation phenomenon in a CNZ sample.

Nuclear transmutation has also been reported to occur under laser irradiation [5], [6]. We performed a hydrogen loading experiment on the sample using a laser with a wavelength of 793 nm and varied its output power within the range of 1–6325 mW.

In addition to powder samples, exothermic phenomena in hydrogen absorption and desorption have also been reported for composite metal thin film samples [7]–[10]. Therefore, we are currently performing experiments using composite thin film samples. In this paper, we report the heat generation results for a palladium-nickel multilayer sample deposited onto a zirconia plate.

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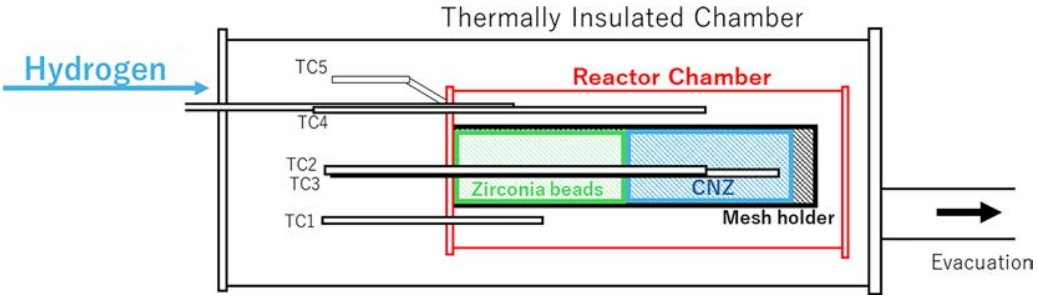


Figure 1. Schematic of experimental setup.

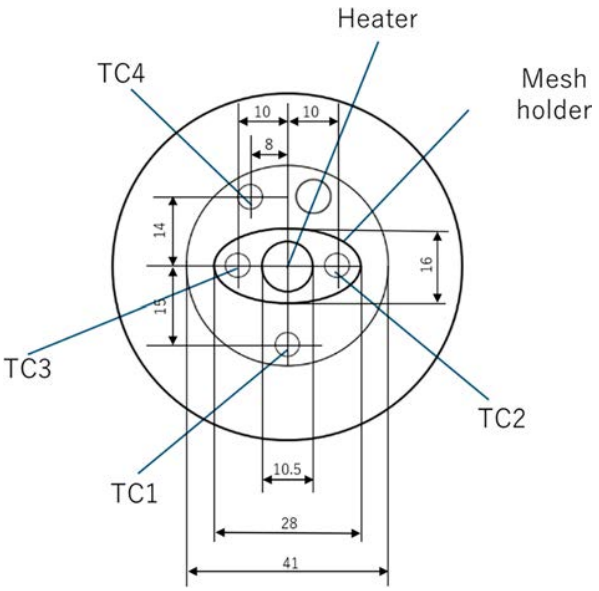


Figure 2. Cross-sectional view of the reactor chamber.

2. Experiment

2.1. Absorption Experiment in CNZ

2.1.1. *Sample*

The sample consisted of a copper–nickel powder supported on zirconia, with a molar ratio of Cu:Ni:ZrO_2 = 1:7:14 with reference to previous studies and a particle size in the range of 75–125 μm. In addition, a reference experiment was conducted using zirconia beads with a particle diameter of 1 mm. In the experiment, 90.5 g of zirconia beads and 61.5 g of the CNZ sample were used, and the lengths when loaded into the mesh holder were approximately 8 cm each.

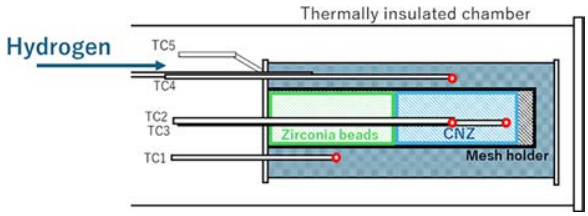


Figure 3. Temperature measurement points.

Table 1. TC positions.

TC1	Hydrogen gas temperature
TC2	Center temperature of CNZ
TC3	Edge area temperature of CNZ
TC4	Hydrogen gas temperature

2.1.2. *Experimental Method*

Figure 1 shows a schematic of experimental setup, and Fig. 2 presents a cross-sectional view of the reactor chamber together with its dimensions. After setting the CNZ and zirconia beads such that the CNZ was positioned at the edge area of the mesh holder (Fig. 2). The outer and reactor chambers were evacuated using a scroll pump to a pressure of a few pascals. The outer chamber served to thermally insulate the system from the surrounding environment Hydrogen was supplied to the reactor chamber at a pressure of 1.6 atm. Subsequently, 50–80 W of constant power was supplied to the heater, and the sample was heated for 6 h. Four thermocouples were installed in the inner chamber to measure the sample and gas temperatures at various locations throughout the experiment (Fig. 3, Table 1). For reference, we performed an experiment using zirconia beads, which do not absorb hydrogen. Subsequently, the heat generated because of hydrogen absorption in the CNZ sample was evaluated by comparing its thermal behavior with that of the zirconia beads.

2.1.3. *Excess Heat Calculation*

Excess heat was estimated from the difference between the temperatures observed in the reference and absorption experiments. The evaluation method was as follows.

1. Plot the temperatures at each heater input in the reference experiment.
2. Fit the data with a quadratic function.
3. Derive the differential coefficient from the fitting curve (y' = dT/dW).
4. Obtain a calibration factor ($k = 1/y'$) from Step 3.
5. Calculate excess heat as $W_{\mathrm{ex}} = k(T_1 - T_0)$.

In Step 5, T_0 denotes the temperature in the reference experiment, and T_1 denotes the temperature in the hydrogen absorption experiment.

2.1.4. *Results and Discussion*

Figure 4 shows the results of the hydrogen absorption experiment conducted with 50 W of heater input. Based on these results, the reduced temperature gradient observed during the initial phase (0.8–2.0 h) was associated with an endother-

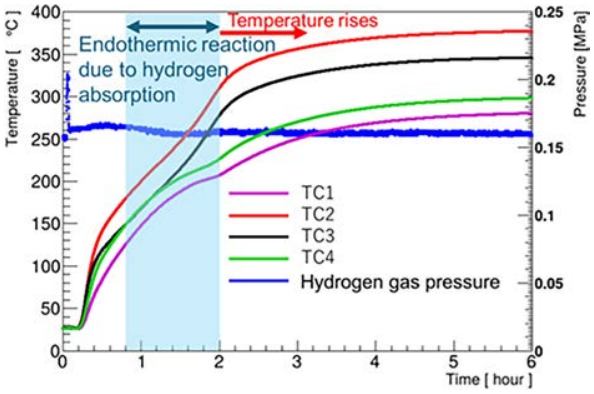


Figure 4. Time dependence of sample temperature and pressure of absorption experiments with a 50 W heater input.

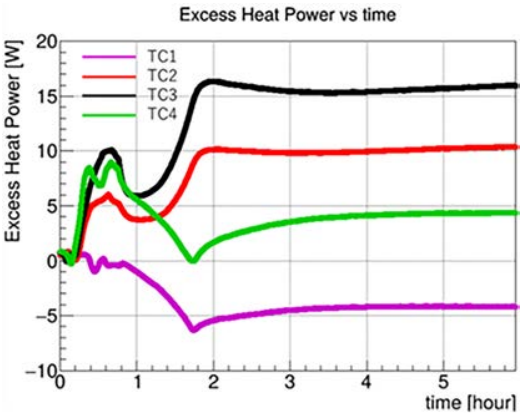


Figure 5. Time dependence of the excess heat with a 50 W heater input.

mic process resulting from the hydrogen absorption into nickel. In contrast, the subsequent temperature increases in the later phase (2.0–6.0 h) suggest the emergence of an anomalous heat effect, indicating a possible transition from hydrogen uptake to heat-generating reactions within the sample.

The results of the absorption and reference experiments with an 80 W heater input are presented in Figures 6 and 7, respectively. The observed temperature differences for each thermocouple were –4.25 °C (TC1), 26.25 °C (TC2), 36.15 °C (TC3), and 8.10 °C (TC4).

The results for excess heat with 50 W of heater input are shown in Fig. 5. The values of excess heat at the end were –4 W for the first thermocouple (TC1), 10 W for TC2, 16 W for TC3, and 4 W for TC4. In this experiment, a negative excess heat signal was observed at TC1, although the cause remains unclear. In contrast, positive excess heat appeared at the other thermocouples. The experiments conducted using the same sample under varying heater inputs over a total of 90 h consistently exhibited excess heat in all cases.

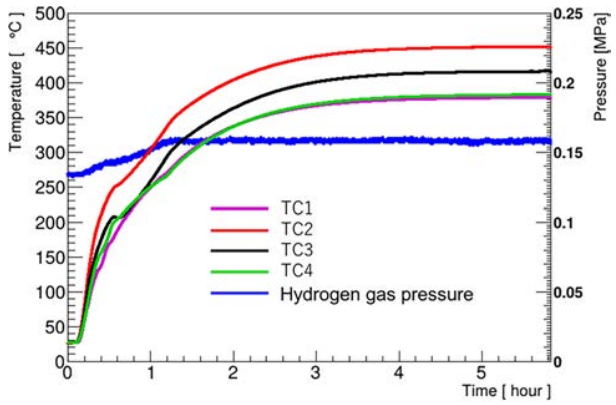


Figure 6. Time dependence of sample temperature and pressure of absorption experiments with an 80 W heater input.

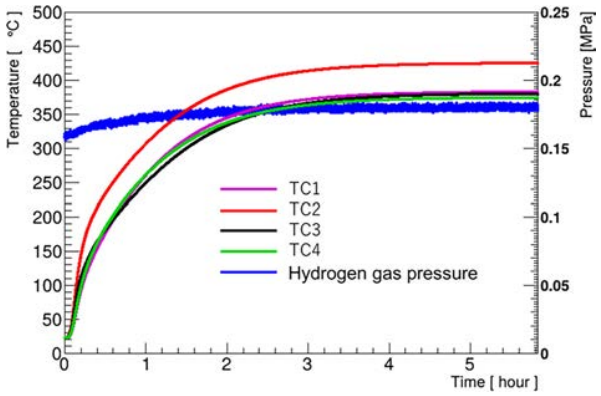


Figure 7. Time dependence of sample temperature and pressure of reference experiments with an 80 W heater input.

Figures 8 and 9 show the relationship between heater input and excess heat at the conclusion of the 6-hour experiment. For the heater input ranges of 50 - 80 W, the excess heat tends to increase with higher input power. This trend suggests that the amount of excess heat may be influenced by the level of energy supplied to the system.

2.2. Laser Irradiation Experiment

2.2.1. *Sample*

As in Section 2.1.1 (Sample), the sample consisted of a copper–nickel powder supported on zirconia, with a molar ratio of Cu:Ni:ZrO_2 = 1:7:14 and particle size in the range of 75 - 125 μm. In addition, a reference experiment was conducted using zirconia beads with a particle diameter of 1 mm.

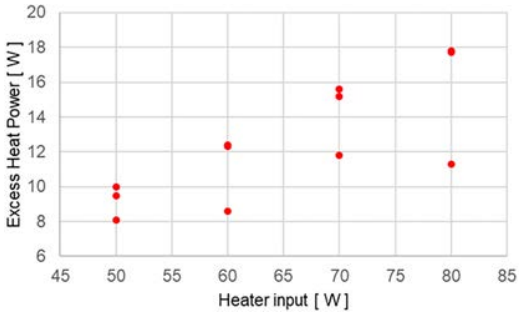


Figure 8. Dependence of excess heat (TC2) on heater input.

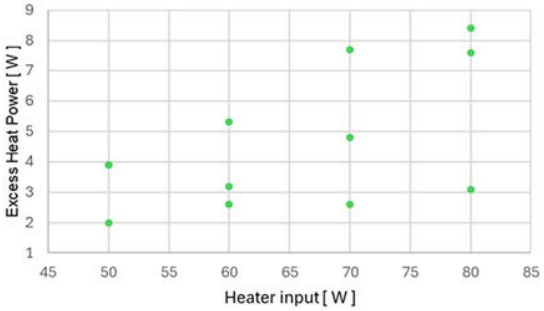


Figure 9. Dependence of excess heat (TC4) on heater input.

2.2.2. *Experimental Method*

A schematic of the laser experiment is shown in Fig. 10. The laser had a wavelength of 793 nm, with the output power set within the range of 1 - 6325 mW. The irradiation was directed toward the vicinity of thermocouple TC2. In this experiment, a 9-hour hydrogen absorption test was conducted under a heater input of 50 W, with laser irradiation applied during the final 3 h.

2.2.3. *Results and Discussion*

Figure 11 shows the result of laser irradiation at 6325 mW. A temperature increase of 28 °C was observed at TC2, which was positioned near the spot irradiated by the laser. The results of the laser irradiation from the reference test are shown in Fig. 12. The laser power was the same as in Fig. 11. However, in this case, it was applied to zirconia beads. Although a temperature increase at TC2 was also observed during laser irradiation in this reference experiment, the rise was approximately 8 °C lower compared to that observed in the CNZ experiment.

Figure 13 shows the resulting excess heat in the 6325 mW laser irradiation experiment. As shown in Fig. 13, excess heat increased by 1.7 W at TC2 and 2.0 W at TC3 during laser irradiation, whereas a decrease of approximately 1 W was observed at TC4. This behavior may be attributed to differences in the physical properties of the CNZ and zirconia beads, particularly in their ability to absorb or reflect the laser light. Similar trends were observed at other laser power levels.

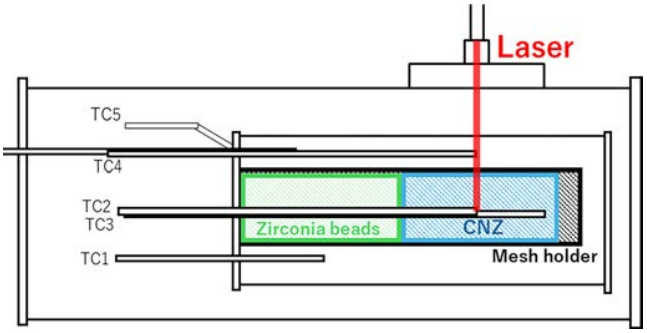


Figure 10. Schematic of laser experiment.

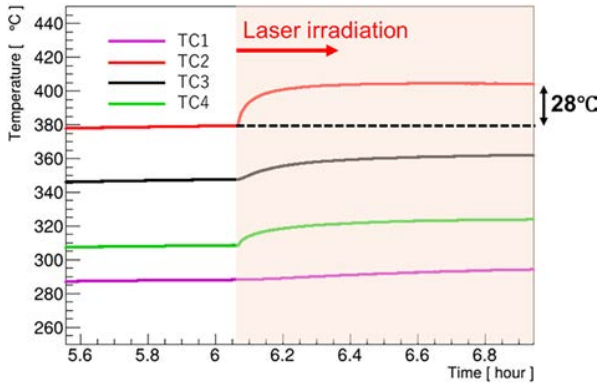


Figure 11. Time dependence of sample temperature with a 50 W heater input and 6325 mW laser irradiation.

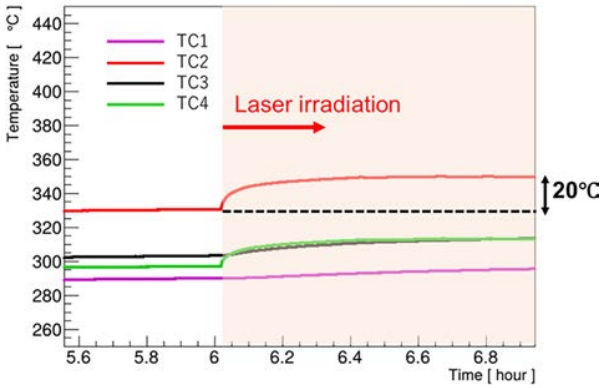


Figure 12. Time dependence of zirconia beads temperature with a 50 W heater input and 6325 mW laser irradiation.

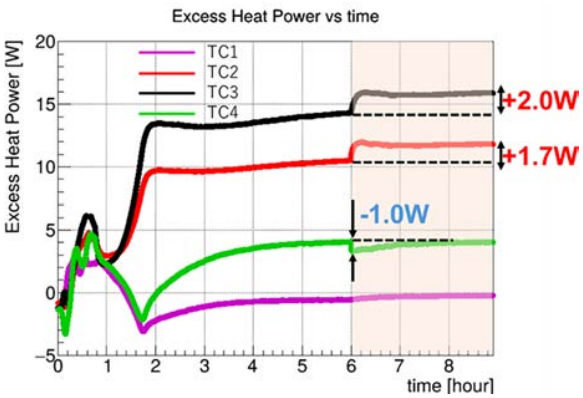


Figure 13. Time dependence of excess heat with a 50 W heater input and 6325 mW laser irradiation.

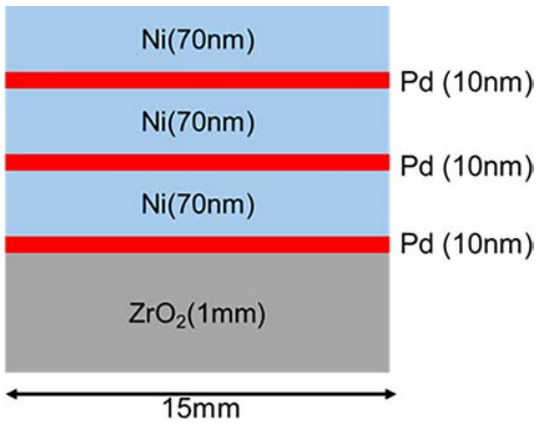


Figure 14. Schematic of sample.

2.3. Hydrogen Desorption Experiment in Multilayer Thin Films

2.3.1. *Sample*

The sample was composed of alternating layers of Pd (∼10 nm) and Ni (∼70 nm) deposited onto a 1 mm-thick ZrO_2 plate ($15 \times 15\ mm^2$) by sputtering (Fig. 14).

2.3.2. *Experimental Method*

A schematic of the experimental setup is shown in Fig. 15. The experimental procedure was as follows. First, the sample was baked after evacuation. Subsequently, approximately 250 Pa of hydrogen gas was introduced, and hydrogen absorption occured for 20 h while heating the sample to approximately 180 °C. The chamber was then evacuated to a pressure of a few pascals. Finally, the sample was heated to a temperature in the range of 600–800 °C. As a reference experiment, a zirconia plate was used.

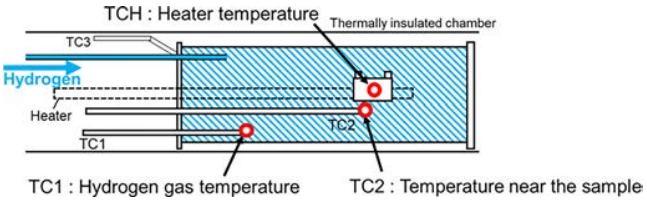


Figure 15. Schematic of the experimental setup.

Table 2. Temperatures reached in each experiment.

Heater Input (W)	Temp at TCH (°C)	Temp at TC1 (°C)	Temp at TC2 (°C)
20	627.90	182.30	216.25
30	722.70	223.00	259.85
40	794.70	257.25	296.15

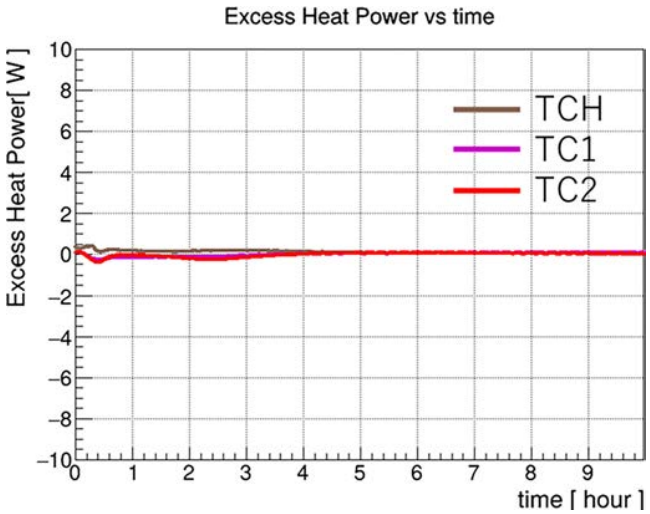


Figure 16. Time dependence of excess heat with a 20 W heater input.

2.3.3. *Results and Discussion*

Table 2 shows the maximum temperatures reached in the experiments at each input power level. Figure 16 presents the excess heat results for the 20 W heater input experiment, Fig. 17 for the 30 W experiment, and Fig. 18 for the 40 W experiment. Focusing on the 30 W experiment, excess heat was observed during the initial 6 h, with a maximum value of 5 W. In contrast, no excess heat was detected in the 20 W experiment, and a heat deficit (negative excess heat) was recorded for the 40 W heater input. Overall, the experiment exhibited low reproducibility, and conclusive evidence for excess heat generation was not obtained.

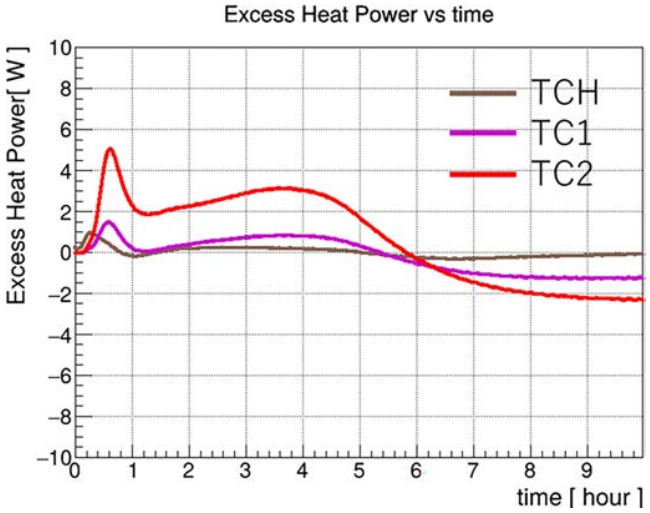


Figure 17. Time dependence of excess heat with a 30 W heater input.

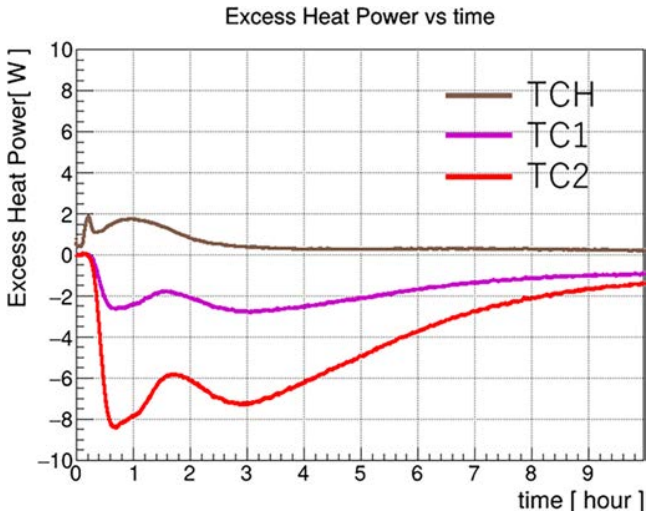


Figure 18. Time dependence of excess heat with a 40W heater input.

3. Summary

In this study, hydrogen absorption and laser irradiation experiments in CNZ and desorption experiments in multilayer thin films were conducted.

In the absorption experiment, excess heat of approximately 4 W, based on hydrogen gas temperature, and 10 W, based on the sample temperature, was observed. Moreover, a certain degree of correlation was identified between the

heater input and measured excess heat. Furthermore, the excess heat generation persisted for approximately 90 h using the same sample.

In the laser irradiation experiment, excess heat increased by 1.7 W at TC2 and 2.0 W at TC3 during laser irradiation, whereas a decrease of approximately 1 W was observed at TC4. The negative excess heat observed at TC4 is considered to be due to the higher reflectivity of the zirconia beads compared to CNZ. Therefore, the excess heat observed at TC2 and TC3 can be attributed to their higher optical absorption, and it cannot be conclusively identified as being induced by an anomalous heat generation phenomenon. This suggests that optimization of laser specifications and irradiation conditions may be required, considering the properties of the CNZ and zirconia beads.

In the desorption experiment in multilayer thin films, heat generation was evaluated during hydrogen desorption. Although one instance of heat generation was observed, reproducibility was limited. Further investigations will be conducted to determine the conditions that reliably induce heat generation.

Acknowledgements

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