Evidence for Surface Heat Release Reaction over Nano-sized Multilayer Metal Composite with Hydrogen Gas

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Abstract

Excess energy generation using a nano-sized multilayer metal composite with hydrogen gas has been investigated. Two nano-sized metal multilayer composite samples, which were composed of Ni, Cu, CaO thin films on bulk Ni (25 mm×25 mm×0.1 mm), were used. These samples were fabricated by Ar ion beam or magnetron sputtering method. Heat burst and excess energy generation were observed during the experiments using nano-sized metal multilayer composite on Ni substrate and hydrogen gas. Maximum average released energy per absorbed total hydrogen was 21 keV/H or 2.0 GJ/H-mol up to now.

Heat burst phenomena were simultaneously detected by two radiation thermometers looking at both surfaces (A and B) of the multilayer thin films and a thermocouple (TC) located near the metal composite. An experimental example showed that heat burst reaction occurred at the surface A at first and propagated to the TC and afterwards heat burst reaction occurred at the surface B. This type of clear evidence that heat release reactions occur in the near surface region of the nano-sized multilayer metal composite with hydrogen gas was often obtained during recent experiments. It also demonstrates that observed excess energy in our experiments was due to real heat generation and cannot be attributed to the artifact like variation of a calibration curve on the relationship between electrical input and TC. These experimental results cannot be explained by any known chemical process and implies that the observed heat generation must be of nuclear origin.

1. Introduction

New type of excess heat experiments using a nano-sized metal multilayer composite and hydrogen gas have been performed based on the permeation-induced transmutation experiments with multilayer thin film and excess heat experiments with nano particles. Figure 1 shows the background of the present experimental method.

The permeation-induced transmutation phenomenon, which is completely different from conventional transmutation by nuclear reactors or accelerators, was first reported in 2002 [1]. D_2 gas permeation through a nano-structured multilayer thin film composed of Pd and CaO thin film and Pd substrate with a target element induces nuclear transmutation reactions [2]-[4]. Permeation-induced nuclear transmutation reactions were firstly observed at Mitsubishi Heavy Industries and were successfully replicated by other institutes such as Toyota R&D center [5]. Typical target element is Cs and produced element is Pr. Transmutation reactions of Sr, Ba, W into Mo, Sm, Pt were also observed

by this method. In this research, deuterium diffusion through nano-sized multilayer thin film was a key factor and the elemental analysis was important technique.

Collaborative Research Project between six Japanese organizations funded by NEDO (New Energy and Industrial Technology Development Organization) on anomalous heat effects was done from Oct. 2015 to Oct. 2017 using Ni, Pd, Cu, Zr nano particles. Anomalous heat generation, which is too much to be explained by any known chemical process, was observed. Qualitative reproducibility was confirmed between the Kobe University and Tohoku University [6]-[9]. The authors replicated the experiments using nano Pd/Ni fabricated by glow discharge with D_2 gas developed by Dr. Mizuno [10]. In these experiments, nano-sized particles and diffusion of hydrogen and deuterium were one of key factors to observe the heat effects and precise heat estimation was crucial.

Combining above important factors and methods to induce transmutation and heat generation reactions, we developed a present method using a nano-sized metal multilayer composite and hydrogen gas. Larger excess energy per H was obtained using the present experimental method.

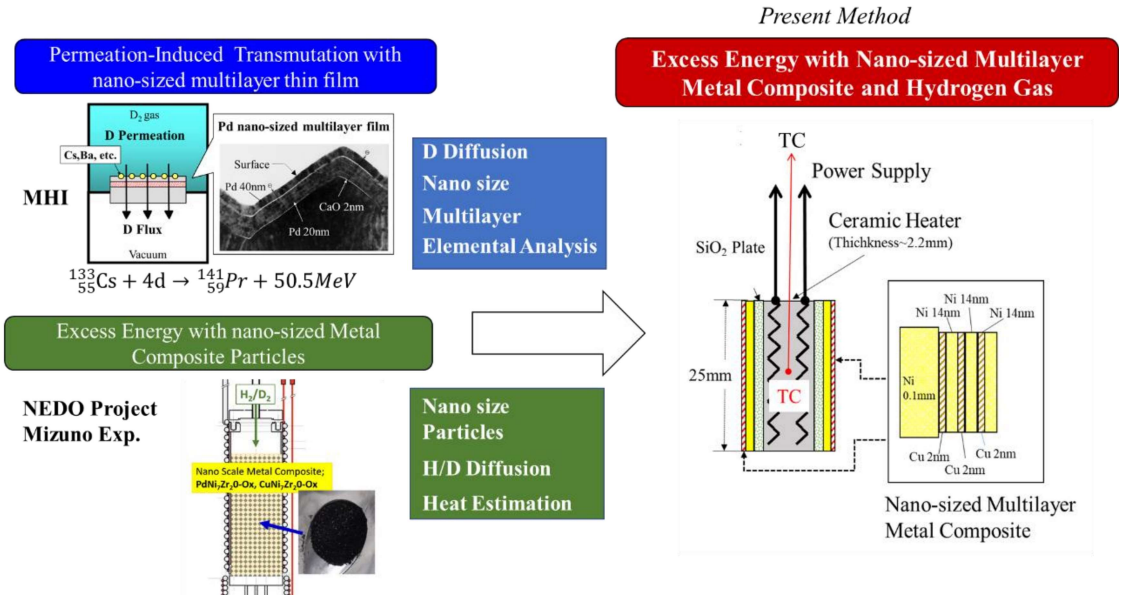

Figure.1 Background and Motivation of the Present Experimental Method.

2. Experimental

Schematic of our experimental apparatus is shown in Fig.2(a). It is basically the same with the paper [11]-[14] and improved in some points. Two nano-sized metal multilayer composite samples, which were composed of Ni, Cu, CaO thin films on bulk Ni (25 mm×25 mm×0.1 mm), were used. H_2 gas and its pressure were monitored by a Pirani gauge. The chamber could be evacuated by a turbo molecular pump. The multilayer samples could be heated up by a ceramic heater in which a thermocouple (TC; Pt-PtRh13 %) was embedded.

In the paper [11], surface temperature of a sample was evaluated by an infrared radiation thermometer (IR-CAQ3CS; Chino Corp.). Recently, we have introduced additional radiation thermometers and surface temperature measurement for the two nano-sized metal multilayer composite samples became possible. The detector was made of InGaAs and two wavelengths, 1.55 μm and 1.35 μm, were used in this work. Heater input power was supplied by a DC power source with constant voltage mode. The input voltage and current were measured both by voltage and current monitors provided by the power supply and an independent voltmeter and amperemeter, respectively.

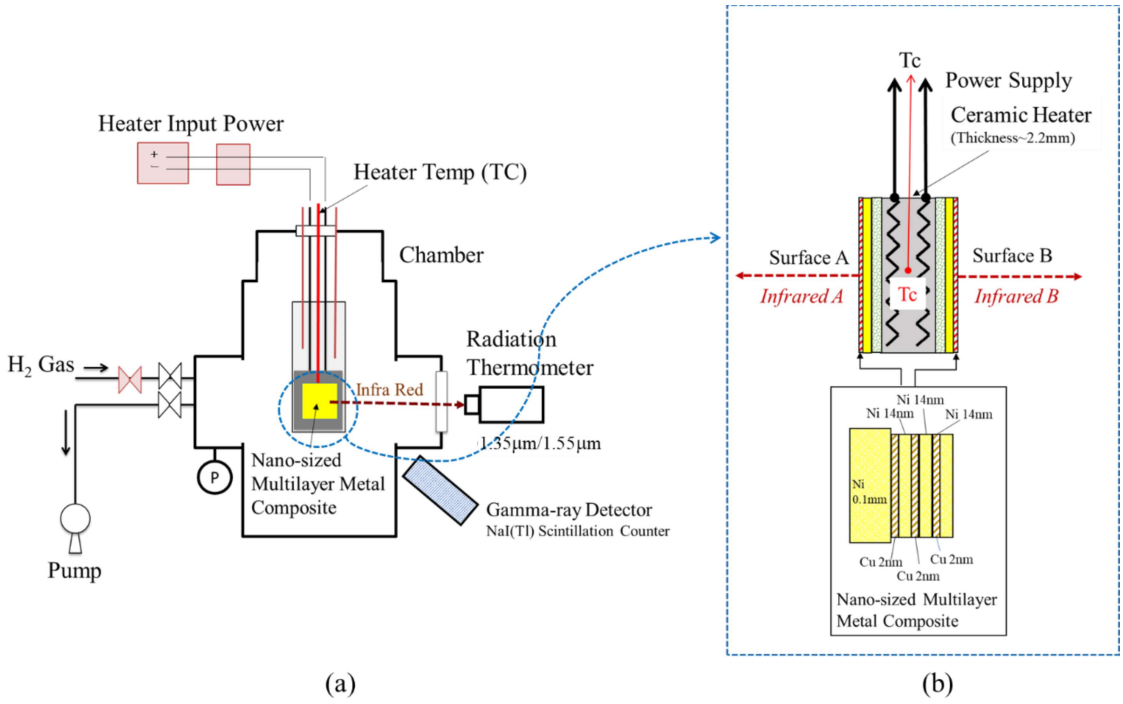

Figure 2. Experimental Set-up; (a) Schematic of Experimental Apparatus, (b) Detail Drawing around Nano-sized Multilayer Metal Composite.

A detailed drawing of the Ni based nano-sized metal multilayer composite is shown in Fig.2 (b). It was composed of a Ni Plate (25 mm square and 0.1 mm thickness) and Cu-Ni multilayer thin film (20 mm diameter circle and about 100 nm thickness). These samples were fabricated by Ar ion beam or magnetron sputtering method. Two nano-sized metal multilayer composite samples were heated by the ceramic heater (25 mm square and 2.2 mm thickness) through SiO_2 plates (0.3 mm thickness). If certain energy generation reactions would happen on the surface of samples, the temperature of the embedded thermocouple (Tc) would rise. Simultaneously, infrared emission detected by the radiation thermometer, which corresponds to surface temperature of the sample, would increase.

The experimental procedure is as follows. Two nano-sized metal multilayer composites were placed in the chamber and baked for 1-2 days at temperature more than 200 °C to remove H_2O on the surface under vacuum condition. After baking, H_2 gas was introduced into the chamber up to about 200 Pa at 250 °C. Usually H_2 gas was loaded for

about 16 hours. Then, H_2 gas was evacuate by the turbo molecular pump and simultaneously the samples were heated up by the ceramic heater up to 600-950 °C. These process triggers heat generation reactions and observed excess heat. Typically, after 8 hours, the heater input was turned down and the samples were made cool down to 250 °C. These processes (H_2 loading, heating up and cooling down samples) were repeated several times changing heating temperature.

During the above experimental procedure, hydrogen atoms are supposed to diffuse from the Ni plate through the nano-sized metal multilayer to the surface. The diffusion mechanism of hydrogen atoms is well known as "quantum diffusion" at low temperature [15]. Hydrogen atoms are hopping from a site to another site in metal. We assume that hydrogen flux is one of the key factors to induce condensed matter nuclear reactions and the hydrogen flux is intentionally arranged by the present experimental method. Hydrogen flux J from the nano-sized metal multilayer composite to the chamber is caused by gradient of hydrogen concentration and gradient of temperature as shown in eq. (1)[16].

$$\boldsymbol{J} = -\mathrm{nD}\left(\nabla \mathrm{c} + \frac{cQ^*\nabla T}{k_B T^2}\right), \tag{1}$$

where n is the number of lattice atoms per unit volume, c is hydrogen concentration defined as the hydrogen/host-metal atom ratio, D is diffusion coefficient and Q* is the heat of transport.

3. Results and Discussion

3.1 Excess Heat Evaluation

Excess Heat is evaluated based on the model described in Fig.3(a) and the following equation (2).

$$k_{eff}\frac{T_C - T_W}{L_{eff}}A_{eff} + A_S\sigma\{\varepsilon_A(T_{SA}^4 - T_W^4) + \varepsilon_B(T_{SB}^4 - T_W^4)\}$$
$$+ A_{Rloss}\varepsilon_{Rloss}\sigma(T_{Rloss}^4 - T_W^4) = P_{in} + H_{ex} \ , \tag{2}$$

where k_{eff} is equivalent thermal conductivity, Tc is the thermocouple temperature embedded in the ceramic heater, Tw is wall temperature of the chamber, L_{eff} and A_{eff} are effective length and effective surface area between the sample holder and wall, respectively. As is surface area of the sample, T_S is the surface temperature, ε is the emissivity of the sample, σ is the Stefan–Boltzmann constant. subscript A and B means surface A and B, respectively. A_{Rloss}, ε_{Rloss} and T_{Rloss} are effective surface area; effective emissivity and effective surface temperature for radiation loss except from the sample surface, which is mainly derived from the sample holder. P_{in} is the electrical heater input and H_{ex} is excess power. This equation is obtained under the following assumptions.

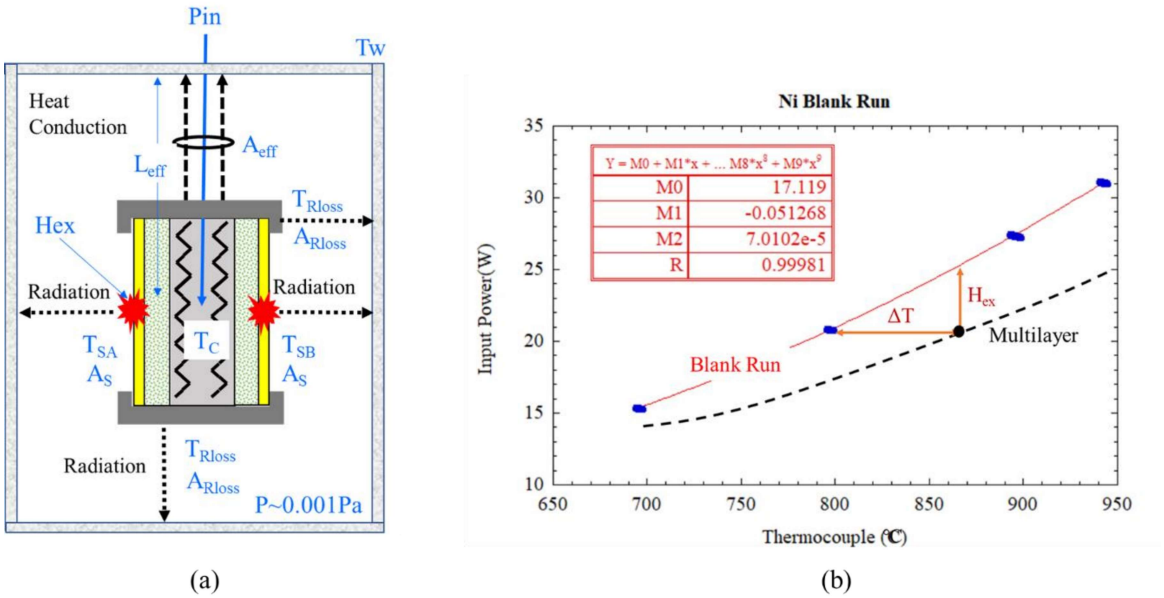

Figure 3. Excess Heat Evaluation; (a) Model of Excess Heat Evaluation, (b) Relationship between Input Power(W) and Thermocouple Temperature (℃) for a Blank Run and a Multilayer Run.

1) Thermal conduction via H_2 gas is negligible as H_2 pressure is low enough.
2) Radiation from chamber wall is negligible because Tw is room temperature.
3) The electrical input power is constant.

A blank run, in which same sized Ni bulk samples without multilayer thin films were used, was performed with the same procedure described above. Figure 3(b) shows the relationship between input power given to the ceramic heater and heater temperature detected by the thermocouple. As the radiation loss term from the sample holder is the same for Ni bulk and multilayer samples for the same temperature, generated excess heat power is supposed to be evaluated based on the blank run result as shown in Fig.3(b). Equation (2) for Ni bulk (subscript "0") is written as

$$k_{eff}\frac{T_{C0}-T_W}{L_{eff}}A_{eff} + A_S\sigma\{\varepsilon_{A0}(T_{SA0}^4 - T_W^4) + \varepsilon_{B0}(T_{SB0}^4 - T_W^4)\}$$

$$+ A_{Rloss}\varepsilon_{Rloss}\sigma(T_{Rloss0}^4 - T_W^4) = P_{in}. \quad (3)$$

In the papers [11]-[14], excess heat analysis was done based on the assumption that ε is constant for Ni based nano-sized metal multilayer composite and Ni bulk as a first step of data analysis.

Emissivity ε can be measured by switching two wavelengths mode and single wavelength mode. Actual measured emissivity was in the range of 0.1-0.2 at surface

temperature 700-750 ℃, depending on the condition of the sample such as oxidation of surface or surface roughness, the vacuum of the experimental apparatus. However, the difference in emissivity between Ni bulk and Ni multilayer composite samples is within 0.05 (< 5 %), and the assumed condition is satisfied. It would be possible to consider that emissivity was almost the same for the Ni bulk and multilayer composite samples.

Excess heat Hex is written based on the equations (2) for multilayer composite and Ni bulk (subscript "0") samples.

$$H_{ex} = k_{eff}\frac{T_C - T_{C0}}{L_{eff}}A_{eff} + A_S\sigma\{\varepsilon_A T_{SA}^4 - \varepsilon_{A0}T_{SA0}^4 + \varepsilon_B T_{SB}^4 - \varepsilon_{B0}T_{SB0}^4 - T_W^4(\varepsilon_A - \varepsilon_{A0})\} + A_{Rloss}\varepsilon_{Rloss}\sigma(T_{Rloss}^4 - T_{Rloss0}^4)$$

$$\approx k_{eff}\frac{T_C - T_{C0}}{L_{eff}}A_{eff} + A_S\sigma\{\varepsilon_A T_{SA}^4 - \varepsilon_{A0}T_{SA0}^4 + \varepsilon_B T_{SB}^4 - \varepsilon_{B0}T_{SB0}^4\} + A_{Rloss}\varepsilon_{Rloss}\sigma(T_{Rloss}^4 - T_{Ploss0}^4) \quad \because\ T_{SA,B}^4 \gg T_W^4 \tag{4}$$

Now, we assume the following relations based on our experimental data. It is possible that T_{SA} and T_{SB} can be expressed as liner function of Tc within the experimental parameters.

$\varepsilon_A \sim \varepsilon_{A0},\ \varepsilon_B \sim \varepsilon_{B0},\ T_{SA} \sim \alpha_A T_C + \beta_A,\ T_{SB} \sim \alpha_B T_C + \beta_B,\ T_{Rloss} \sim \alpha_{Rloss} T_C + \beta_{Rloss}.$

ΔT is defined as

$$\Delta T = T_c - T_{c0}. \tag{5}$$

Therefore, the following expression is obtained.

$$H_{ex} \approx \Delta T\left\{\frac{k_{eff}}{L_{eff}}A_{eff} + A_S\varepsilon_{A0}\sigma\alpha_A(T_{SA} + T_{SA0})(T_{SA}^2 + T_{SA0}^2) + A_S\varepsilon_{B0}\sigma\alpha_B(T_{SB} + T_{SB0})(T_{SB}^2 + T_{SB0}^2) + A_{Rloss}\varepsilon_{Rloss}\sigma(T_{Rloss} + T_{Rloss0})(T_{Rloss}^2 + T_{Rloss0}^2)\right\}. \tag{6}$$

This equation shows that excess heat can be written as a function of ΔT. Therefore, excess heat evaluation by the Ni bulk calibration curve shown in Fig.3(b) is valid under the assumptions described above.

3.2 Experimental Results

Heat burst phenomena were observed by a single radiation thermometer and the thermocouple in the heater simultaneously as shown in Fig.4 published already in [11]-[13]. A significant simultaneous increase of surface and heater temperature were observed twice, while no significant changes for input electrical power and room temperature were detected during these burst events.

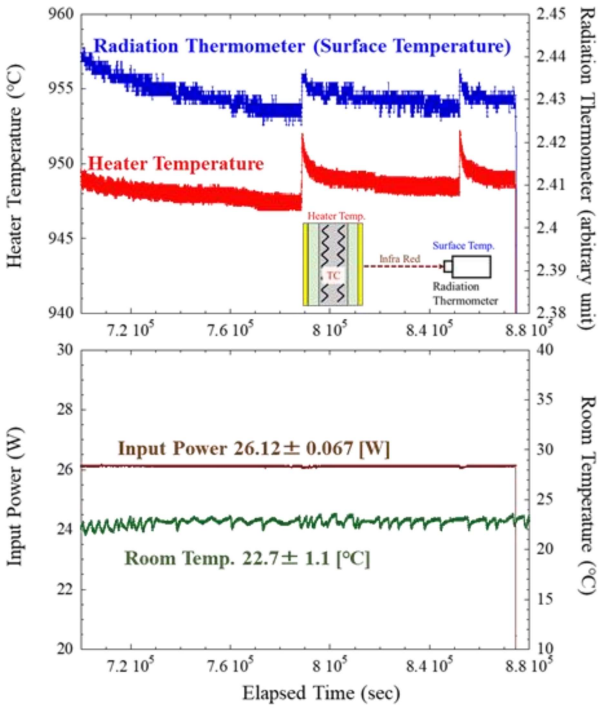

Figure 4. Simultaneously detected by a radiation thermometer looking at the surface of the multilayer thin film and a thermocouple located in the center of heater [11].

In this study, we have introduced additional radiation thermometers and surface temperature measurement for the two nano-sized metal multilayer composite samples became possible. By using the two independent radiation thermometers, interesting phenomena could be obtained.

Figure 5 shows temperature time variations for Tc, surface A and B. It is possible to see that heat release reaction occurred at the surface A at first and afterwards at the surface B. Their heat bursts propagated to the thermocouple. Input power and room temperature were constant during these events. Therefore, these events did not be caused by the change of electrical input power or heat income from environment. The samples consisted of 6 layers of Cu (2 nm) and Ni (14 nm) fabricated by magnetron sputtering method on Ni bulk same as in [11]-[14].

Delay time from the burst-like heat release at surface A to the thermocouple is about 33 sec as shown in Fig.5. Delay time from the heat burst at surface B is supposed to be about 40 sec, as inflection point of Tc can be recognized as shown in Fig.5. Although 3-dimentional heat analysis would be desirable, rough estimation of time constant for the observed burst phenomena. Figure 6 shows numerical model for one dimensional non-steady state heat conduction analysis. Time constant τ is given as the following equation.

$$\tau = \rho C_p V \frac{L}{k_{Al_2O_3+SiO_2} A} \tag{7}$$

where ρ is the density, C_p is the specific heat, and V is the volume for the sample holder. And k is the thermal conductivity for the mixture of Al_2O_3 and SiO_2, A is the surface area and L is the distance between the surface and center of the heater. As Ni is thin and thermal conductivity is larger than Al_2O_3 and SiO_2, contribution of Ni for τ is negligible. Based on the rough estimation, τ is calculated as 36 sec. It agrees with the experimental results, therefore the obtained delay times are supposed to be reasonable. This example gives us clear evidence that heat release reactions occurred in the near surface region of the nano-sized multilayer metal composite with hydrogen gas.

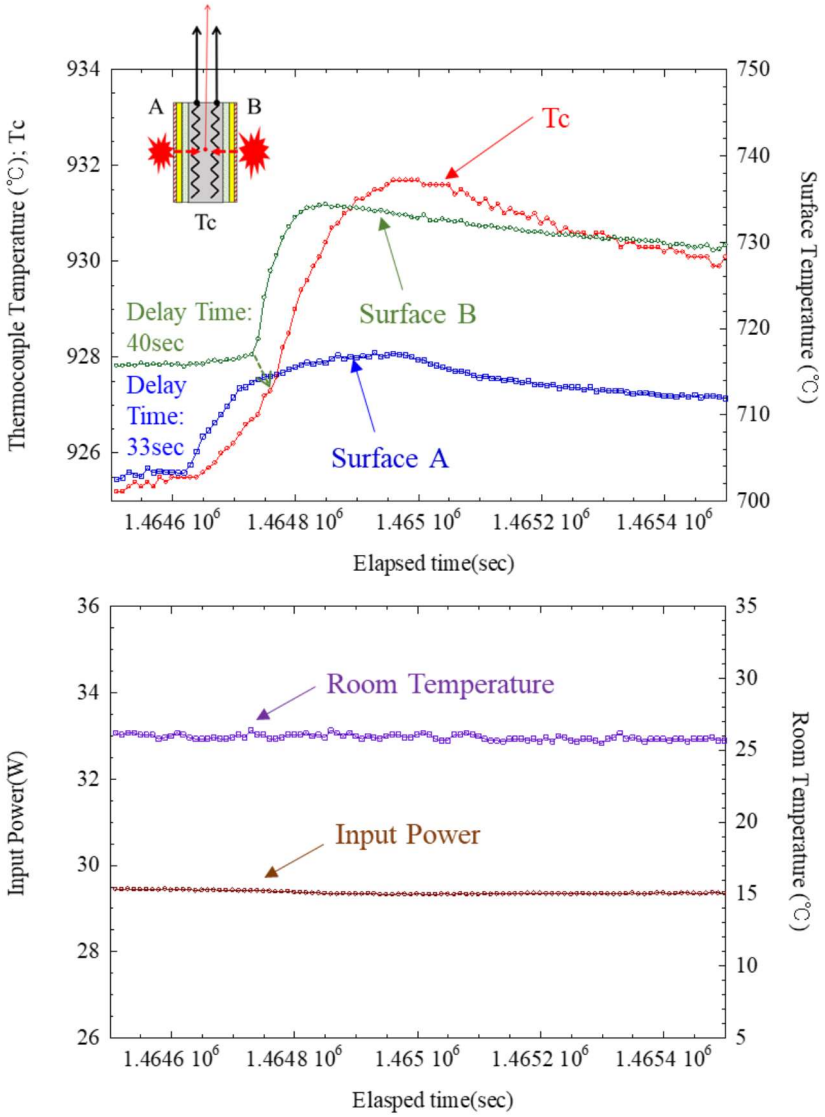

Figure 5. Heat burst reactions occurred at the surface A at first and afterwards at the surface B. Their heat bursts induced the rise of Tc. Input power and room temperature were constant during these events.

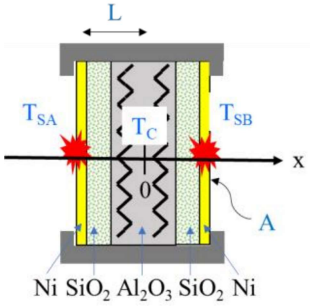

Figure 6. Numerical model for one dimensional non-steady state heat conduction analysis.

The above is an example of heat burst occurring on two surfaces, but it is more common for a heat burst to occur on only one surface. Figure 7 shows an example that a heat burst generated at surface B only. The samples for this experiment consisted of 6 layers of Cu (2 nm) and Ni (14 nm). In this case, the heat burst generated at surface B reached the thermocouple with a delay time of about 24 seconds and was further transmitted to surface A, but the temperature rise at surface A is small. Input power and room temperature were also constant during the heat burst event. These two examples demonstrate that observed excess energy in our experiments was due to real heat generation and cannot be attributed to the artifact like variation of a calibration curve on the relationship between electrical input and Tc.

Excess heat of this example is plotted in Fig.8. At the beginning of these experimental cycles, H_2 gas was evacuated and simultaneously each sample was heated up by the ceramic heater. After that, excess heat more than input power was observed for each experimental cycle as shown in Fig.8. Above each excess power plots, the value of the electrical input and the approximate thermocouple temperature are noted. During each experimental cycle, the input was almost constant. However, since the power supply was operating in constant voltage mode, when the sample temperature decreased, the resistance of the heater decreased: the input electrical power increased. As in the previous experiments, the excess heat tended to decrease gradually compared to immediately after the temperature rise at the beginning of each experimental cycle. We can see that some heat bursts occurred in this experiment as shown in Fig.8.

As a verification of the uncertainty in this excess heat calculation, we describe the effect of emissivity variation on the excess heat evaluation. In Eq. (2) and Fig. 3, the heat conduction loss and the heat loss from the sample holder are constant whether the sample is Ni bulk or Ni multilayer. Measured emissivity values in the temperature region where the heat burst phenomenon occurred shown in Fig. 7 were 0.16 for side A and 0.18 for side B, respectively. Based on these values, the energy released by radiation from surface A and B was about 2.8 W and 3.4 W, while electrical input power was 26.7 W. Thus, the radiant energy from the sample surface is not dominant to the input energy. The excess heat is evaluated to be about 6.7 W, as shown in Figure 9. Even though the error would be about half of the radiant energy from the surface A and B, about 3.6 W of excess energy would be being released from the Ni based nano-sized multilayer metal composite.

Released excess energies per hydrogen for this example is evaluated. The total amount of absorbed hydrogen for all the experimental cycles was 5.9×10^4 mol. The amount of released excess energy was calculated by the time integration of excess powers for each experimental cycle. Total released energy is calculated as 1.2 MJ. Although it

seems highly unlikely that all the absorbed hydrogen atoms reacted, we can still estimate that average released energies per absorbed total hydrogen. Total excess energy per absorbed hydrogen is 2.0 GJ/H-mol or 21 keV/H-atom. Obviously, it is too large to be explained by any known chemical reactions.

Even if the excess heat were 1/10 of this value, it would be 0.2 GJ/H-mol or 2.1 keV/H-atom, which means that energy that could not be explained by known chemical reactions must have been generated. Therefore, the uncertainty of the excess heat evaluation does not significantly affect the conclusion that observed phenomena cannot be explained by known chemical reactions.

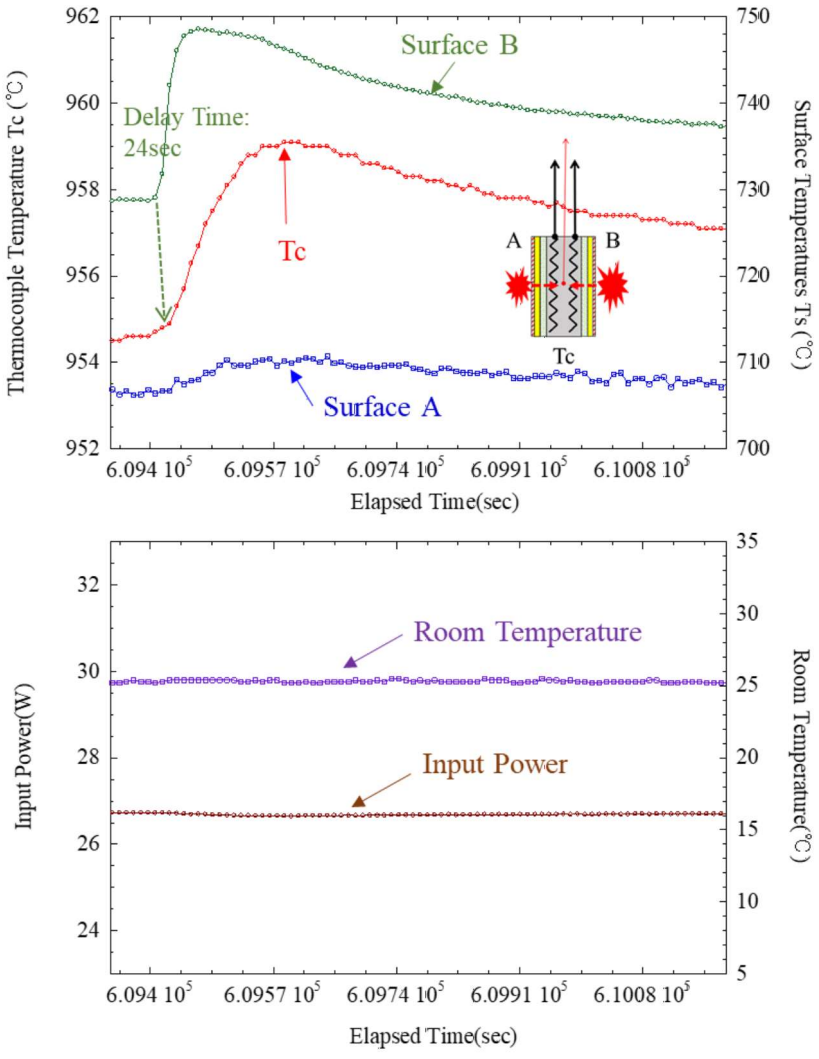

Figure 7. Heat burst reactions occurred at the surface at the surface B only and propagated to the TC. Input power and room temperature were constant during these events.

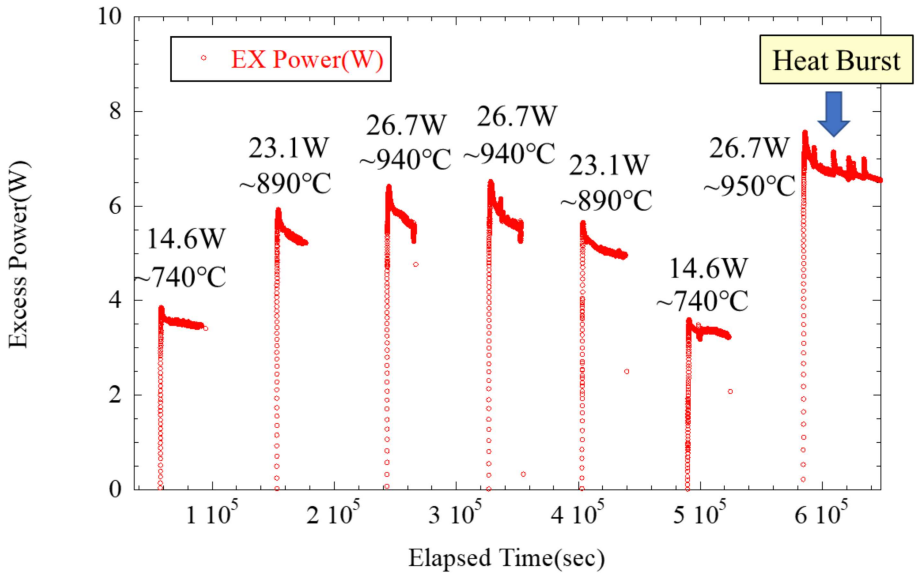

Figure 8. Excess power for this example. The heat burst phenomenon shown in Fig. 7 is an expansion of the time zone indicated by the arrows in this graph.

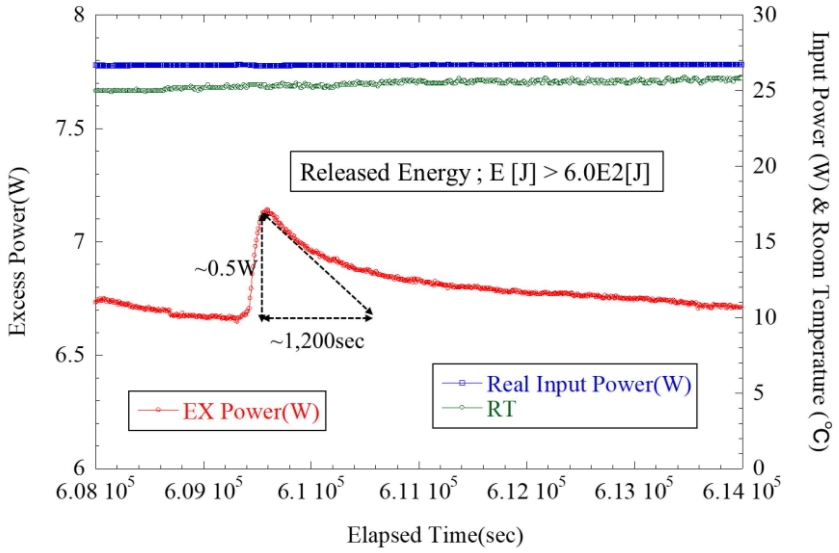

Figure 9. Estimation of Released Energy by the Heat Burst described in Fig.7.

Next, we examine the possibility that the observed heat burst phenomenon was caused by some chemical reactions. Figure 9 plots the change in excess heat during the heat burst shown in Figure 7 of this experimental example. At 6.094×10^5 sec, a heat burst of excess heat occurred. The peak increase of excess power was about 0.5 W and the

excess increase due to the burst lasted for at least 1200 seconds. This means that the excess heat increased by the heat burst is more than 600 J.

Consider the following chemical reactions that may cause a thermal burst for some reason.

(1) If all the residual gas would be oxygen and the absorbed hydrogen would be oxidized and generates heat.

(2) If the residual gas would be oxygen, and the Ni metal would be oxidized by it.

(3) If the residual gas would consist of mixed gas (H_2:O_2 = 2:1), and the combustion energy would give energy to the surface of the two samples.

During the heat release experimental cycle, all the hydrogen introduced for absorption was evacuated, and the degree of vacuum was less than $1x10^{-4}$ Pa when the heat burst phenomenon was observed. Therefore, the number of moles of residual gas was less than $4x10^{-10}$ mol. It is known that the combustion reaction of hydrogen is about 290 kJ/mol and the heat of formation of nickel oxide is about 240 kJ/mol. Based on the number of moles of residual gas in the chamber, the heat generated in each case can be calculated as (1) $5.1x10^{-5}$ J, (2) $1.7x10^{-4}$ J and (3) $6.8x10^{-5}$ J. Comparing these values to the energy increased by the heat burst, 600 J, shows that they are completely negligible. This clearly shows that at least the chemical reactions (1)-(3) cannot explain the heat burst phenomenon observed in this study.

Above experimental results and discussions strongly suggests that some condensed matter nuclear reactions were induced in our experiments. According to the results, about 10^{-3} of the absorbed hydrogen might react if the condensed matter nuclear reactions would release energy order from 1MeV to 10MeV. Identification of reaction products is very important future work, in addition to more preciously excess heat evaluation, respectively.

4. Concluding Remarks

We have developed a new experimental method that combines the experiments of hydrogen permeation through multilayer films with those of hydrogen storage in nano-sized metals and temperature rise. As reported at JCF20 in 2019, we observed heat generation that cannot be explained by chemical reactions using Cu/Ni and Cu/Ni with CaO and Y_2O_3 in multilayer metal composites. Recently, we introduced a radiation thermometer, which enables us to measure the surface temperature of the sample, and we can frequently observe bursts of surface temperature. Examples of burst phenomena observed on only one side and on both sides of the two samples used in the experiment were reported. When the surface temperature of the sample increased, the heat was transmitted to the thermocouple in the center and to the sample on the opposite side, indicating that the exothermic reaction occurred near the surface. The burst phenomenon cannot be explained by known chemical reaction, and strongly suggests that he observed heat generation must be of nuclear origin.

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