Supplemental Information on Increased Excess Heat from Palladium Deposited on Nickel

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Introduction

This document has supplemental information on the paper:

Mizuno, T. and J. Rothwell. *Increased Excess Heat from Palladium Deposited on Nickel (Preprint)*. in *The 22nd International Conference for Condensed Matter Nuclear Science ICCF-22. 2019*. Assisi, Italy.

https://www.lenr-canr.org/acrobat/MizunoTincreasede.pdf

Palladium May Need to be Annealed

Some of the people trying to replicate this experiment have reported that their palladium is harder than nickel, so when it is rubbed on the mesh, little or no palladium adheres to the mesh. The nickel rubs off, instead. Ashraf Imam suggested that annealing Pd for two hours at 650 C will soften it. An inert atmosphere, like argon, ought to be used for the annealing to avoid oxidation.

Nickel Toxicity

Note that the following warning has been added to p. 12 of the report:

WARNING: The steps described in this paper can produce fine nickel powder, which can be toxic. The procedures in this paper should be performed in properly equipped laboratory, in a glove box or other enclosure. Disposable gloves and masks should be worn when handling these materials. They should also be worn to avoid contaminating the materials. **Only people skilled in the art should attempt to replicate this experiment.**

Q&A Responses by Mizuno

A note by Jed Rothwell, June 25, 2019

A person who is thinking of replicating Mizuno's experiment asked series of questions in English. I wrote some responses myself. Then I translated everything into Japanese. Mizuno confirmed my responses and wrote some additional comments. Here are some of the questions with Mizuno's answers, translated, condensed and rewritten by me.

Q: Does the mass of reactant matter?

A: The more reactant you have, the better, so please put in as much as possible.

Q: Is there an optimum number of layers?

A: Probably, the more layers the better, without an upper limit.

Q: Is the choice of Pd important?

A: Probably not. But you should rub it hard. The hypothesis is that by strongly rubbing the Pd into the Ni, you break up the oxide layer and create complex phases of metal on metal. That's what I think is happening, but I do not understand the mechanism yet.

Q: What about the choice of detergent, or the choice of the scrubbing pad?

A: I do not suppose the choice of detergent has an effect on excess heat generation. It probably has nothing to do with it. Just use whatever type you like. However, be sure to rinse thoroughly. This is necessary. I also doubt the choice of scrub pad has anything to do with heat generation. However, be sure to scrub the mesh thoroughly and then wash with alcohol.

Additional comments by Jed Rothwell:

I doubt the source of meshes matters, as long as they are Nickel-200, 180 mesh. My intuitive feeling is that any source of Ni or Pd will work, as long as they are of sufficient purity and you avoid contamination, or you clean off contamination. This method of applying Pd is so crude, there must be large variations in the resulting material. It seems unlikely the details matter. I think that the source of material is probably less important than material preparation. You have to clean the mesh and roughen the surface as instructed. Probably the mass of reactants and the geometry are important.

Materials and Methods

This is an expanded and corrected recipe from two sections from the paper:

Materials: Reactant Nickel Mesh, and R20 Reactor

Methods

Nickel Mesh Preparation

Materials

Sheet Nickel Mesh: Nickel 99% purity, 0.055 mm wire × 180 mesh, dimensions 8 inch × 12 inch.

Palladium 99.95%

Silicon Carbide Wet-Dry Abrasive: grits: 500CC, 800CC, 1200CC, 1500CC.

Cleaning

All handling is done with gloves in hood.

1. Wash sheet with a mild detergent in ordinary tap water, and scrub with a plastic dish scrubbing pad.
2. Sand with water resistant sandpaper, starting with 400 grit, then 800, 1200, and 1500.
3. Wash again with mild detergent using plastic scrubbing pad, and rinse with tap water.
4. Soak in tap water at about 90°C for 1 hour.
5. Wash with ethyl alcohol.
6. Weight each mesh and the palladium.
7. Scrub each mesh strip with the Pd. Rub the nickel screen with a palladium in all four directions on both sides. The entire surface is vigorously rubbed.
8. Periodically weigh the nickel strip under treatment. When the mass of the strip of mesh increases by 15 mg to 20 mg, weigh the palladium and record. Note: with hardened palladium, this method may not work. You may need to keep track of the palladium weight instead. See the next section.
9. Repeat step 8 for the remaining meshes.
10. Place the meshes on top of each other and roll together and slide them in reactor against the wall of the reactor around the sheath heater.
11. Close reactor and install in calorimeter. Evacuate the reactor to 7.5×10^{-5} torr (0.01 Pa). Hold for 2 hours.
12. While pumping, heat with sheath heater to 100° - 120°C for 5 - 20 hours. Pressure must reach 7.5×10^{-5} Torr (0.01 Pa).
13. Increase temperature to 200°C with sheath heater while pumping for 1 - 2 hours. Most of the water should be out of the sample and reactor at this point.

14. Cool down reactor for 1 - 2 hours.

Heat production

1. Valve off the vacuum pumps and set deuterium pressure to 0.75 - 2.25 Torr (100-300 Pa). Do not exceed 45 Torr (6000 Pa). Record pressure history.
2. Raise the temperature 100°C with the sheath heater. This should generate some excess heat.
3. If there is no excess heat, raise the temperature higher

Hardened Palladium may Reduce Weight of Nickel and Create Dust

A note by Jed Rothwell, July 22, 2019

A person who is replicating the experiment told me that after vigorous rubbing, the weight of the mesh decreased. I asked Mizuno to comment. He said that some forms of Pd are hardened, to the point where they are harder than typical Ni. If you are using one, instead of measuring the weight gain of the mesh, you should measure the decrease in weight from the Pd sample. He added, "when the palladium is harder, that means there will be a lot nickel powder, which is extremely dangerous to inhale." He provided these numbers for hardness:

Palladium
Mohs hardness 4.75
Vickers hardness 461 MPa
Brinell hardness 37.3 MPa

Nickel
Mohs hardness 4.0
Vickers hardness 638 MPa
Brinell hardness 700 MPa

The person who reported the weight of the mesh decreased later found fragments of the screen from the edges on the workbench. They must have broken off when the sample was rubbed. A few fragments might weigh 50 mg.

Tap Water may Play a Role

Some people suspect the tap water in Sapporo may make a difference. They are investigating this. There is a detailed description of this water in Japanese which you can Google translate. It is here:

https://www.city.sapporo.jp/suido/overview/suishitu/result/documents/suishitsukekkah31-04.pdf

Preparing Meshes Underwater

Several people have recommended sanding and preparing meshes underwater, in a tray full of water. This prevents nickel nanoparticles from escaping into the air. It is probably safer. However, it is not clear whether this will affect the outcome of the experiment.

Video of Replication in Progress

Here is video of a replication in progress. Unfortunately, in the first attempt, this did not produce excess heat. This could be because the researchers do not have a mass spectrometer to check for contamination. This equipment is highly recommended.

https://www.youtube.com/watch?time_continue=1&v=TWZ-uXQTF_s

Instructions Sent with Prepared Meshes

Mizuno is preparing some meshes and sending them to researchers. Here is a copy of the instructions he sent with a set of 3 meshes. Note that "furnace" here means the stainless steel reactor.

I am sending you the reactant nickel mesh. The net is made by Inada Kanaami Co., Ltd., of nickel 200, twill wire mesh of 0.055 × 180 mesh, and 300 × 300 mm. Pd is attached to both sides. As it is handmade, it is uneven.

It may have been opened on the way for overseas flights. I think that it is dirty for that. Wash with hot water (distilled water, preferably at 80°C, ultrasonic cleaning, 10 minutes) before use. Then wash down with ethyl alcohol and place in the furnace. At that time, please use rubber gloves etc. and do not touch directly with bare hands.

After installing in the furnace, tighten the bolts and evacuate. When the pressure is less than several Pa, heat up to 100°C while evacuating and degas further. Continue processing for 24 hours. After that, the heating was stopped, and when the temperature dropped to the room temperature, then evacuation is stopped and 50 Pa D_2 gas was supplied. I will leave it for 24 hours. Then evacuate and heat to 100°C. After sufficient degassing, cool and supply 50 Pa of D_2 gas.

If there is no vacuum leak, excessive heat will be generated if heating is performed from this condition. And then you will just take the data. The temperature can be heated up to 400°C. This is in the case of a stainless steel furnace.

All the best,

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Replication at Hokkaido University of Science

December 2019

Researchers at the Hokkaido University of Science (https://www.hus.ac.jp/eng/) have replicated the experiment. In one result, they input 500 W of heating and the reactor output ~650 W (150 W excess).

The experiment was performed by Dr. S. Saito, with assistance from two senior undergraduates and an expert from the Hokkai PEEM Co. Ltd., an industry consortium at Hokkaido University that specializes in manufacturing Photo-Electron Emission Microscopes (PEEM) and TOF SIMS machines (https://hpeem1.jimdo.com/). This company apparently intends to manufacture cold fusion devices. See:

https://hpeem1.jimdo.com/%E6%96%B0%E8%A6%8F%E4%BA%8B%E6%A5%AD-1/

Brochure:
https://hpeem1.jimdo.com/app/download/11114946074/%E3%83%91%E3%83%B3%E3%83%95.pdf?t=1516582965

This is not a fully independent replication. Mizuno supplied the reactor and mesh, and advised them on how to construct the calorimeter. They are now working on a more independent replication.

Here is a preliminary look at one of their results. This is a first approximation approach to the calorimetry. They have done more calibrations and more detailed analyses.

Figure 1 is the air flow calorimeter with the faceplate removed to show the inside. This is similar to Mizuno's calorimeter, but the ambient temperature controls in the lab are better, so there is less noise in the data.

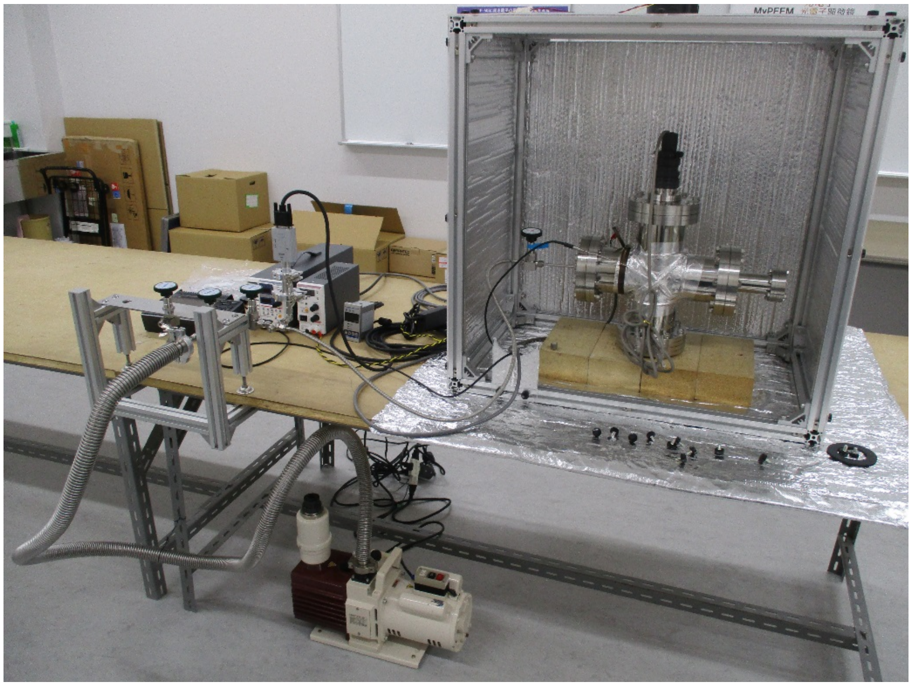


Figure 1. Air flow calorimeter and reactor at Hokkaido University of Science.

Figure 2 is a schematic of the equipment.

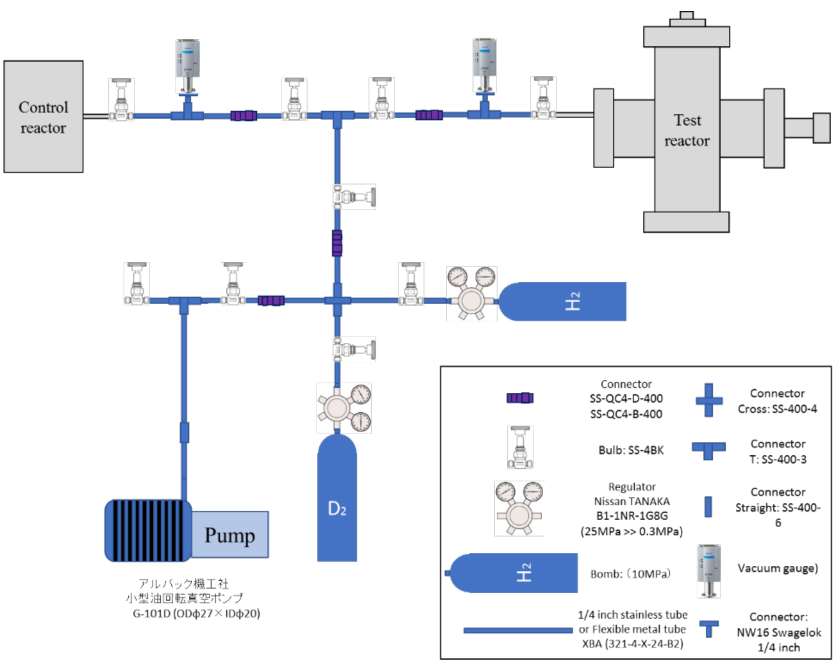


Figure 2. Schematic of equipment.

Figure 3 is a 500 W calibration performed on Dec. 2, 2019, including most of the major parameters measured during the test; blower power, pressure, input heating, reactor temperature, and the inlet and outlet air temperatures. The blower airspeed (not shown here) is 4.2 to 4.3 m/s.

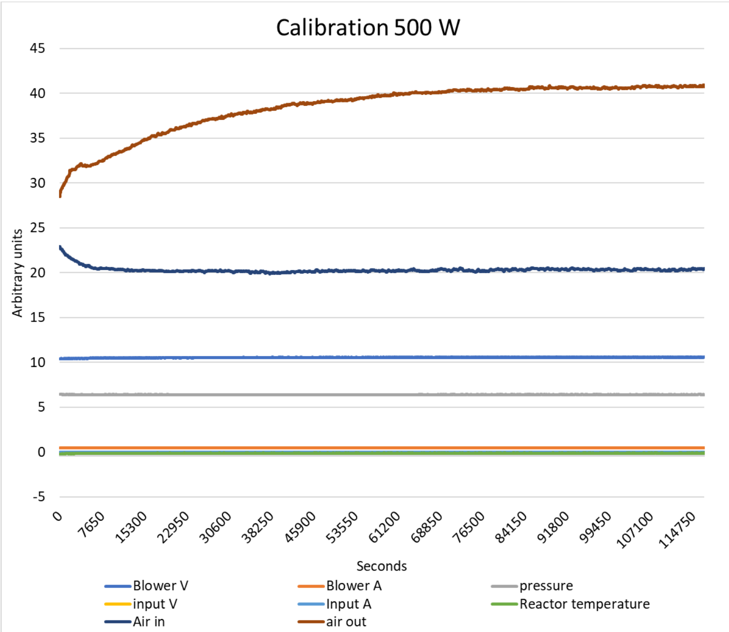


Figure 3. Most of the parameters measured during a 500 W calibration.

Figure 3 is the same calibration data, input and output power, converted to watts. At 500 W, ~70% of the heat is recovered in the flow of air; 30% is lost from the calorimeter chamber walls. In other words, ~350 W is captured in the air flow and ~150 radiates from the reactor walls. This has been confirmed with other methods and calibrations.

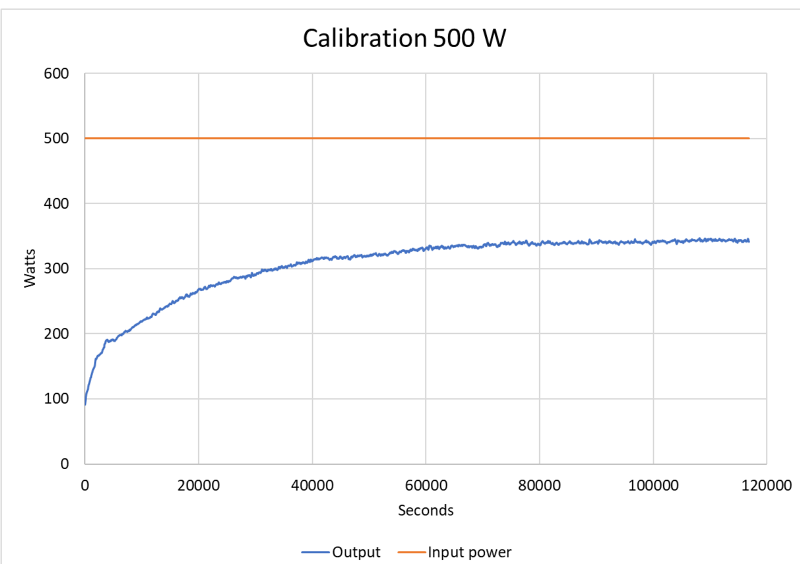


Figure 4. The same 500 W calibration shown in Fig. 2, input and output power converted to Watts.

Figure 4 shows the results of an excess heat test. By coincidence, the total heat measured in the air flow is about the same as input power; around 500 W. (In other words, the blue line happens to reach the orange line.) However, as shown in the calibration, if there were no excess heat, the blue line would be lower, at 350 W instead, so the reaction is producing about 150 W.

At 8,000 s, the heat increases abruptly, even though input power is stable. At 35,000 s heat increases to ~510 W, above the orange line, and then falls back at 40,000 s, even though there is no change in input power. Such perturbations never occur during stable calibrations, so the increased power comes from a reaction, rather than input power.

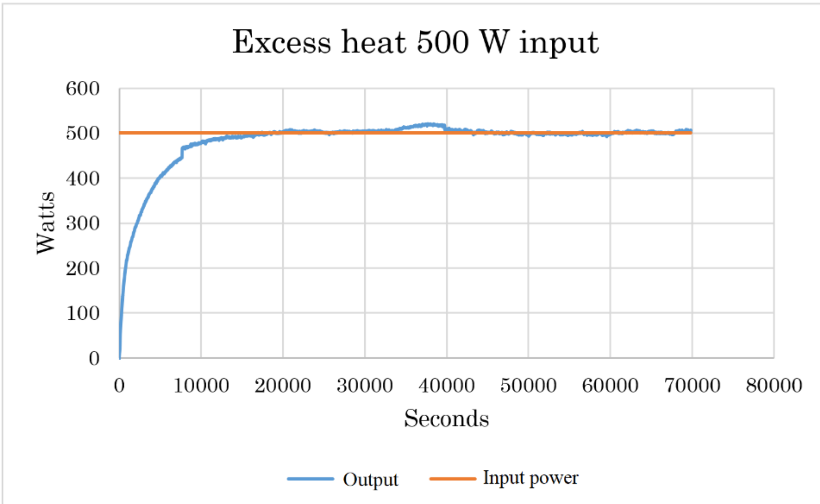
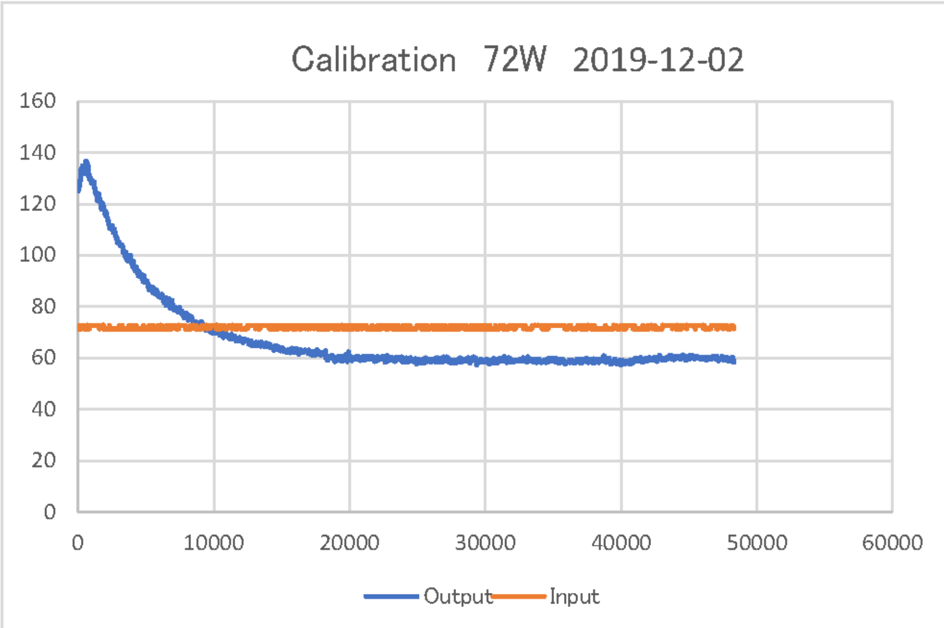


Figure 5. Input and output power during an excess heat test at 500 W.

Tests at other power levels have been reported, and they show a similar pattern. Heat recovered during calibration is below input power, whereas with excess heat, the output is close to input, or it exceeds it slightly. Saito et al. have provided calibration and excess heat graphs for tests at:

72 W
150 W
200 W
300 W
345 W
400 W
500 W (Figs. 3, 4 and 5 above)
600 W
700 W
750 W

Results are shown below for 72 W (Fig. 6), 345 W (Fig. 7) and 750 W (Fig. 8).



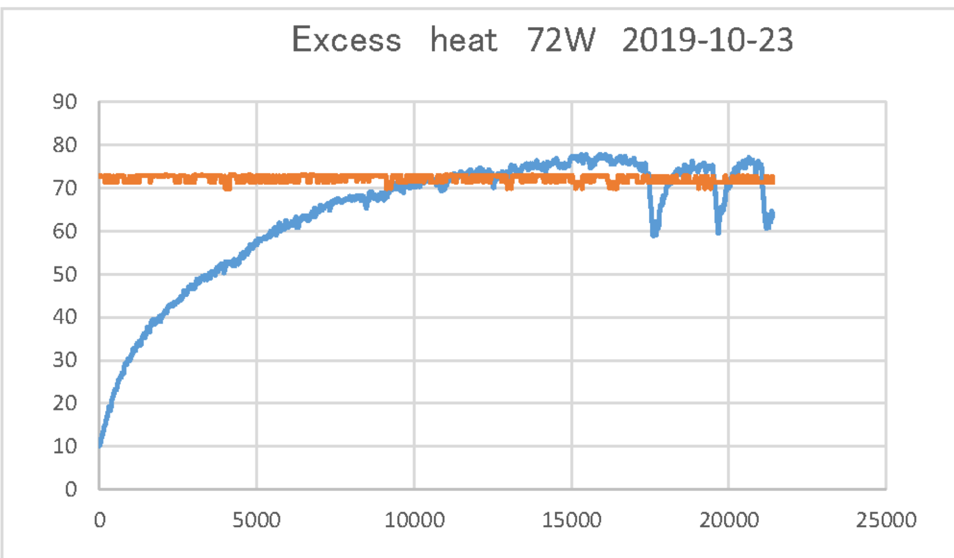
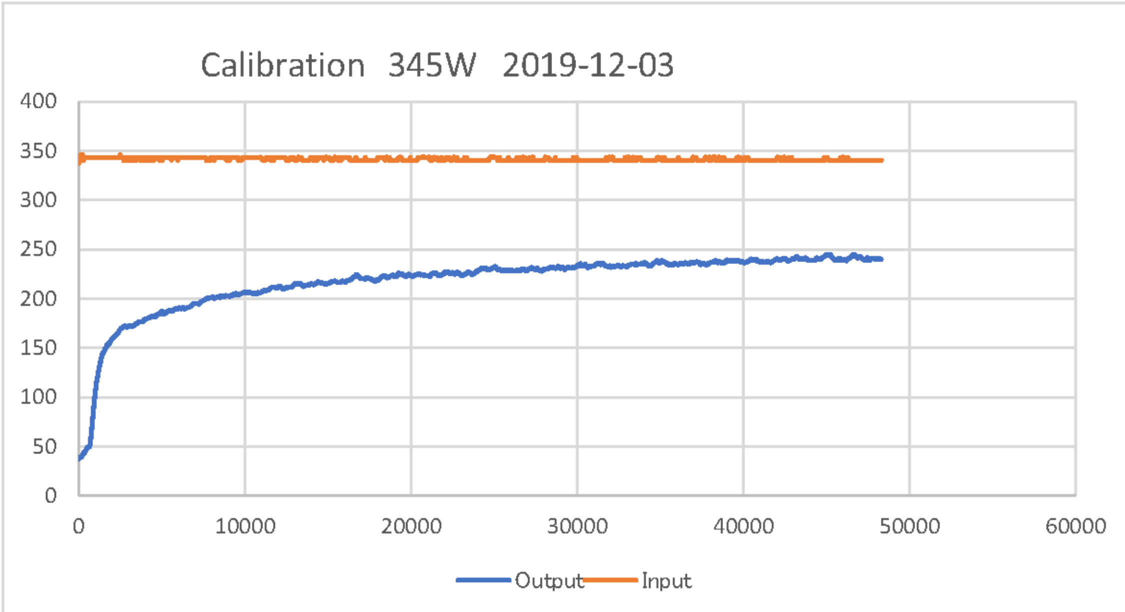


Figure 6. Calibration and excess heat at 72 W.



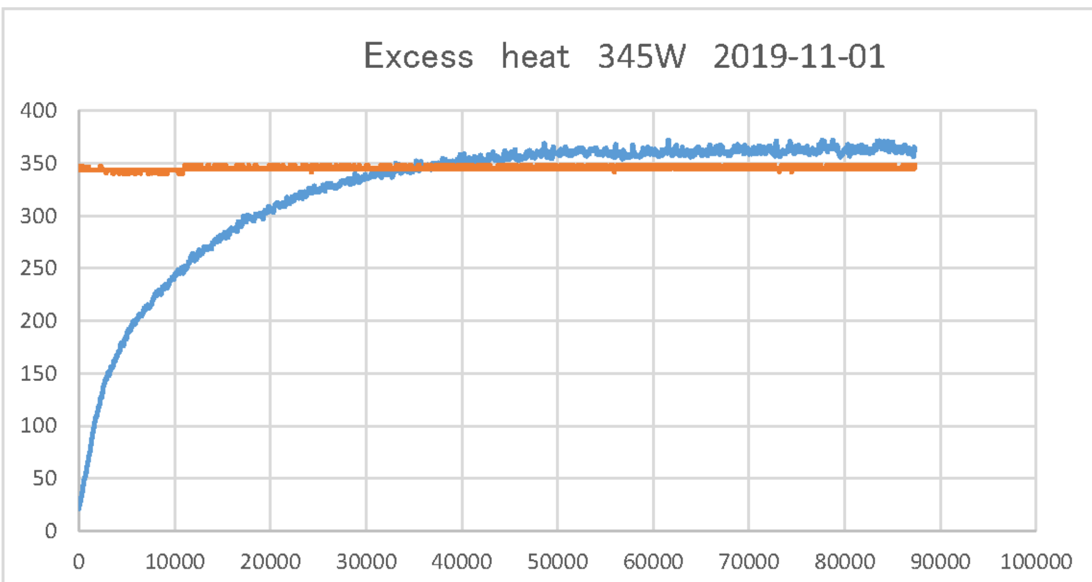
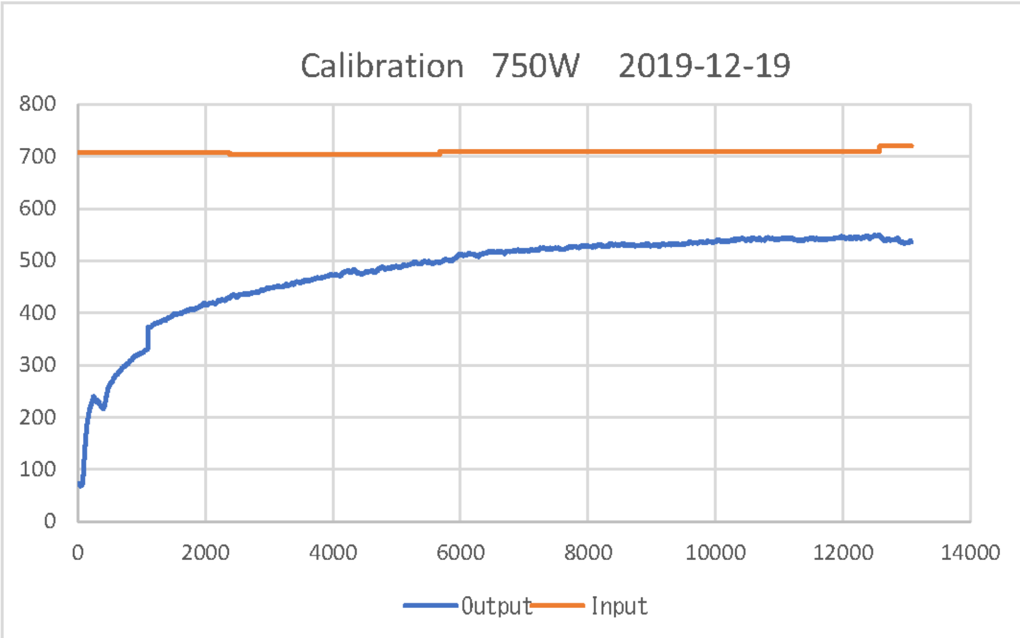


Figure 7. Calibration and excess heat at 345 W.



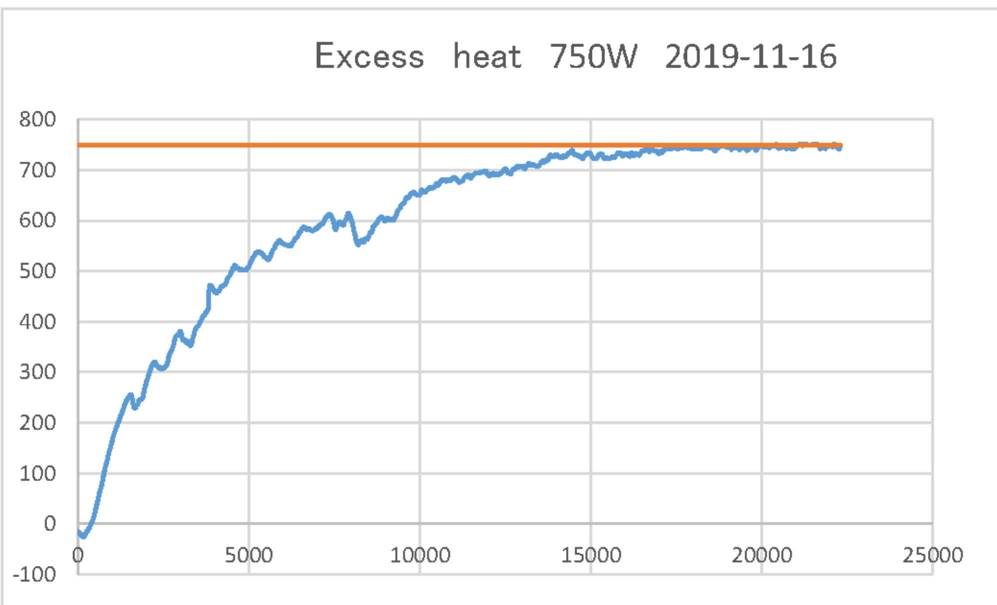


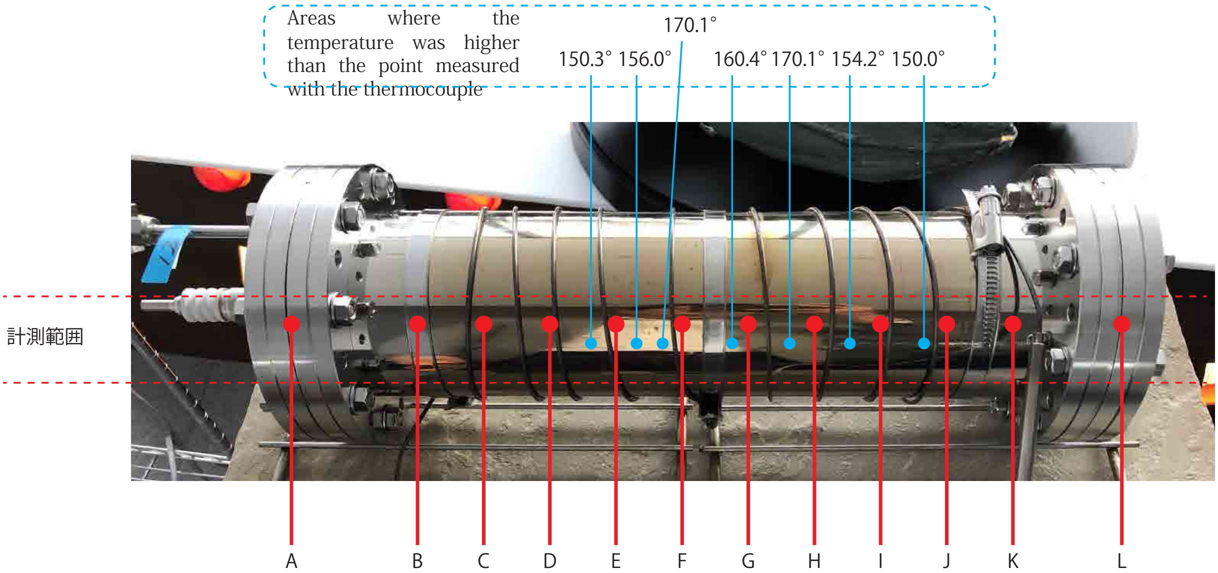
Figure 8. Calibration and excess heat at 750 W.

Temperature Distribution Study

The image and graph below are from a study of the surface temperature of a reactor. The reactor is producing approximately 300 W, including ~30% excess heat. This is an approximate estimation because the reactor is in open air rather than in a calorimeter.

The thermocouple is mounted on the surface at the point marked K. Some other points on the surface marked with blue dots were at a significantly higher temperature than point K. The mesh is under these points, which supports the conclusion it is the source of excess heat.

2019/2/4 13:00 ~ 13:30 Input 100 V, 300 W. Room temperature 25.6°



Measurement point	A	B	C	D	E	F	G	H	I	J	K	L
Measurement 1	46.8°	71.6°	101.3°	114.2°	121.9°	112.0°	112.5°	105.2°	105.1°	126.1°	75.6°	42.7°
Measurement 2	48.8°	104.8°	136.1°	126.9°	110.2°	110.1°	113.5°	125.1°	131.7°	104.1°	97.9°	50.5°
Measurement 3	48.6°	73.5°	112.3°	128.4°	132.5°	138.5°	104.4°	108.6°	105.4°	104.9°	87.9°	48.8°
Measurement 4	61.3°	112.4°	111.6°	158.9°	156.0°	126.4°	131.4°	170.1°	120.7°	145.3°	77.9°	48.3°
Average	51.3°	90.5°	115.3°	132.1°	130.15°	121.7°	131.4°	127.2°	115.7°	120.1°	84.8°	47.5°

Graph of average values