Increased Excess Heat from Palladium Deposited on Nickel

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Abstract

We have developed an improved method of producing excess heat with nickel mesh coated with palladium. The new method produces higher power, a larger output to input ratio, and it can be controlled effectively. With 50 W of input, it produces ~250 W of excess heat, and with 300 W it produces ~2 to 3 kW. This paper is a comprehensive description of the apparatus, the reactant, and the method. We hope this paper will allow others to replicate the experiment.

Keywords: Air flow calorimetry, Deuterium gas, Excess heat, Nickel reactant, Pd coating, Simple method

Introduction

Heating a nickel mesh coated with a thin film of palladium produces cold fusion excess heat. When we first reported this method, excess heat was typically 12 W or 12%. We are now getting much better results. Excess heat is much higher; the ratio of output to input is higher; and the heat can be controlled by raising or lowering the reactor temperature. Here are typical results with three reactor versions: R13, used in the ICCF21 paper [1], and R19 and R20, that produced the results described in this paper:

R13: 100 W heat input, 112 W heat output, 12% excess heat
R19: 200 W input, 290 W output, 45% excess
R20: 50 W input, 300 W output, 600% excess (low power result)

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The R20 reactor can produce much more than 250 W excess, but our air-flow calorimeter heat removal capacity is limited to ~1000 W. So, high power results can only be measured outside the calorimeter, as shown in Fig. 1. This has only been done by comparing the performance of the heater to an ordinary 3 kW electric or gas room heater. That method is approximate, but it does show the reactor is producing roughly as much heat as a typical room heater:

R20: 300 W input, ~1.5 to ~3 kW output, at ~600 to ~1000% excess (high power result)

We hope we can test the reactor in a larger calorimeter before the ICCF22 conference, and include these results in the final version of this paper.

The only major change to the experiment since our last report has been the design of the reactor. The reactants and methods have not changed, so we believe the reactor design is the cause of the improved performance.

The air-flow calorimeter used in this study was described in Ref. [1]. The reactor, nickel reactants and methods were also described. They are described here in more detail. We hope this is enough detail to allow persons skilled in the art to independently replicate the results.

Figure 1. An R20 reactor used as a room heater in Sapporo, winter 2018. With 300 W input, it kept the room about as warm as a conventional 3 kW gas or electric room heater. Such room heaters are common in Japan. The reactor is powered by a 500 W, 100 V laboratory power supply, which is not connected in this photo.

Calorimetry

The air-flow calorimeter was used in this study is described in detail in Ref. [1]. It is briefly described here. The instrument has not been changed. As described in Ref. [1], before a new sample is tested, critical parameters are measured. They include heat losses from the walls of the

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reactor chamber, and the air flow rate. The calorimeter performance has been stable. These critical parameter values have not changed significantly since 2017.

Accurate temperature measurements are crucial to calorimetry. The inlet temperature is measured with 1 RTD, and it is the same as room temperature, which is independently measured at locations away from the calorimeter chamber. The outlet temperature is measured with 2 RTDs, which agree closely. All RTDs are calibrated together to ensure they agree, and that outlet minus inlet temperature differences are accurate.

Losses from the walls are estimated by calibration. The air-flow calorimetry recovers 95% of the heat when the reactor vessel is at 40°C, but only 77% when it is at 360°C (Fig. 2). When this heat recovery rate is applied, nearly all of the heat is accounted for (Figs. 3, 7).

The flow rate is measured at several points on the outlet (a traverse test) to ensure the air is well mixed and air temperature is uniform (Fig. 4).

We recommend air-flow calorimetry for this experiment. The reactor walls must be hot for this reaction to occur. In previous experiments we used water-flow calorimeters with cooling coils up against the reactor walls, or cooling coils with insulation between the coil and the wall. Both types removed heat too quickly, reducing or eliminating the reaction. The calorimeter is an integral part of the experiment. It can interfere with the results, or enhance them.
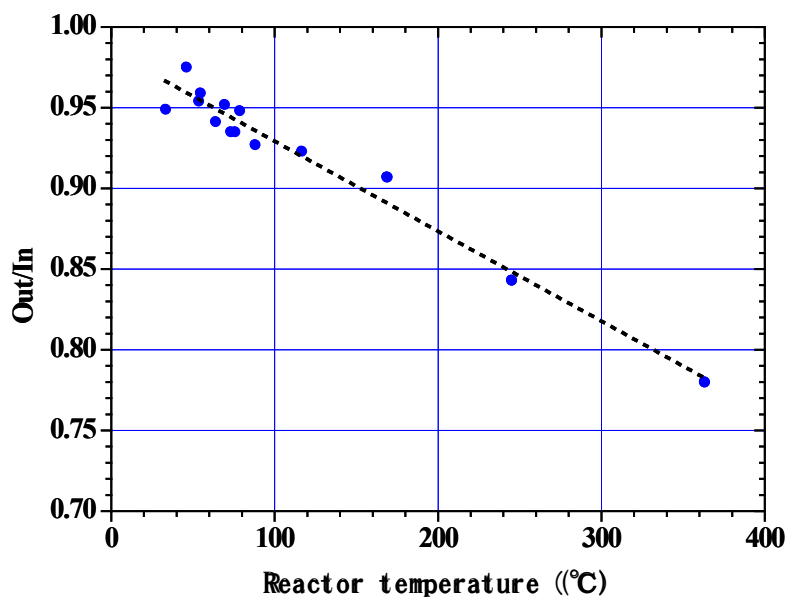

Figure 2. Calibration of reactor temperature and heat recovery rate, from Ref. [1].
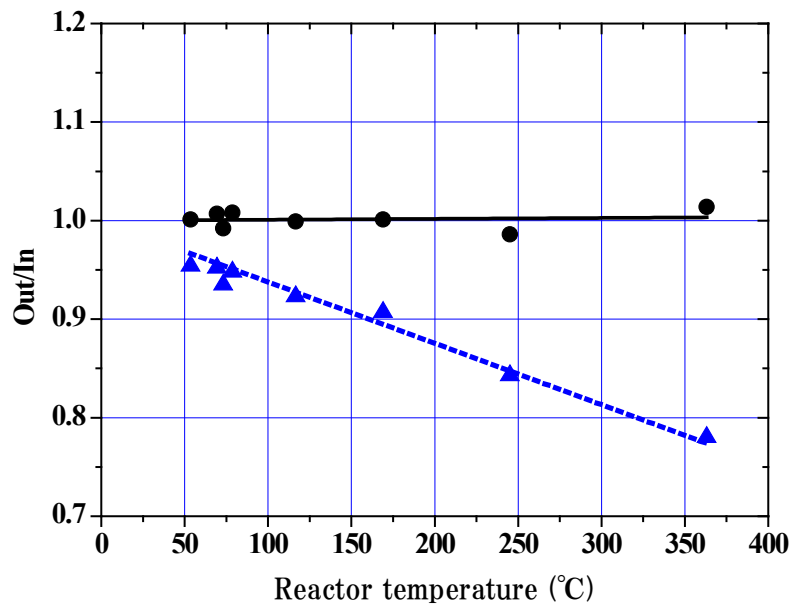
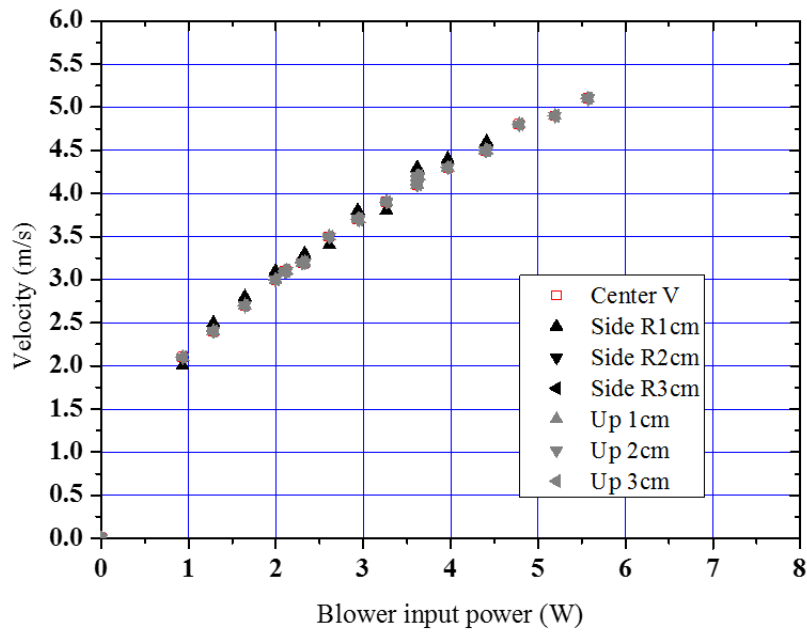Figure 3. Thermal calibration with and without heat recovery. From Ref. [1].

Figure 4. Relationship between blower power and air velocity at the outlet for different locations of the anemometer across the tube section. The velocity profile is almost uniform. From Ref. [1]

A Sample R20 Result

This section describes a typical result with the latest and most effective reactor, version R20. In this test, 40 W of heat was applied initially. This was increased to 50 W. After 3 hours, output heat stabilized at 303 W heat total, or ~250 W of excess heat.

Three methods of analysis are shown in the graphs below:

1. A comparison of the outlet minus inlet temperatures with a 50 W calibration versus the 50 W excess heat test (Fig. 5). This is the raw temperature data from the calorimeter. This is the simplest first approximation. Assuming only that input power and the air flow rate is the same in both tests, this shows that much more heat is produced in the excess heat test. The temperature difference is 10°C higher with excess heat.
2. The temperature difference converted to heat (Fig. 6, gray line). That is to say, the weight of air times the heat capacity of air, times the temperature difference. (See Ref. [1].)
3. The temperature difference converted to heat and then adjusted to include heat losses from the calorimeter chamber walls (Fig. 6, blue line).

You can compute the amount of excess heat by comparison to a calibration (method 1), or by the absolute method of flow calorimetry. Both methods give the same result. This result is refined by taking into account the heat losses from the calorimeter walls (method 3).

Figure 6 shows 250 W of excess heat plus 50 W heating, or 300 W output total. The output stabilizes after 2 hours. It continues at that level indefinitely.

Figure 7 shows two hours from a 50 W calibration. Over 24 hours, average input electric power was 50.6 W. An average of 46.6 W of heat was captured in the stream of air. After taking into account heat losses from the calorimeter walls, the average was 50.5 W.
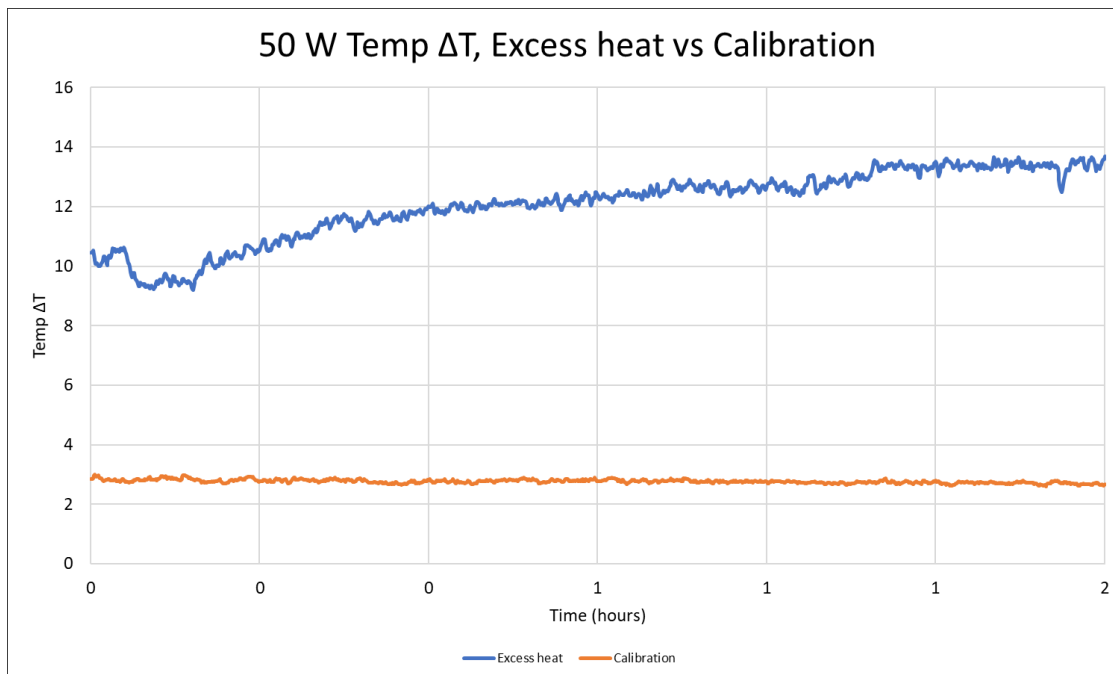
Figure 5. Air flow calorimeter outlet minus inlet temperature, for excess heat test (blue) versus a calibration (orange), both at 50 W. This is the raw data from the calorimeter. The temperatures are subtracted only (outlet air temperature minus the inlet), with no other processing.
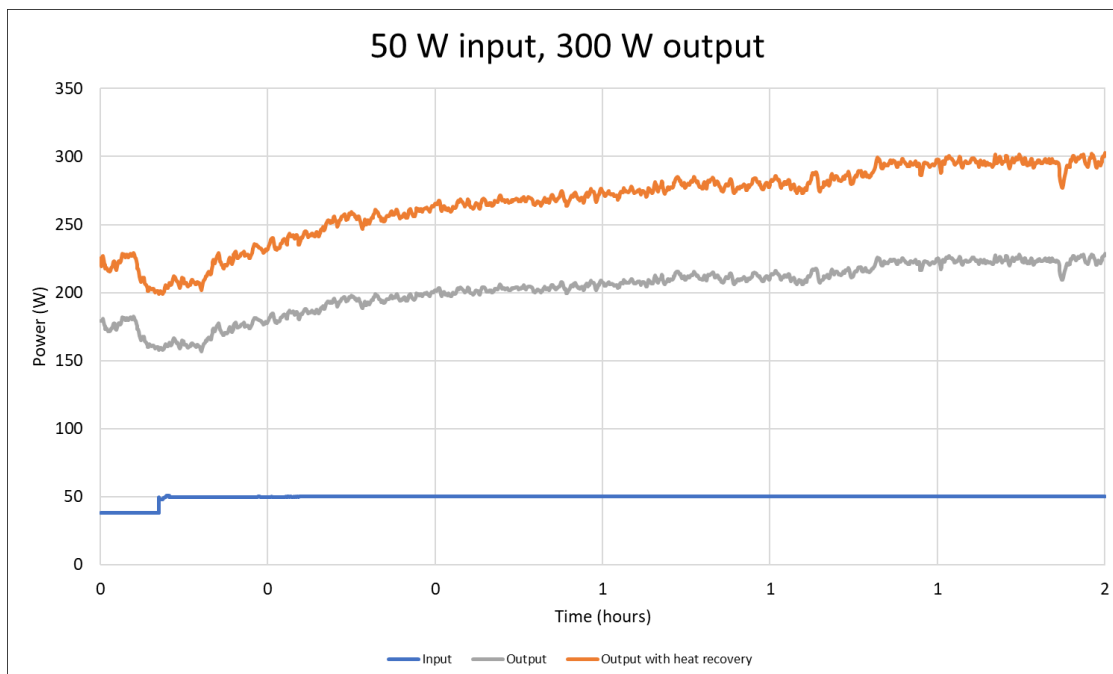

Figure 6. Excess heat test. The temperature difference has been converted to heat (gray), and then adjusted to include heat losses from the calorimeter chamber walls (orange).
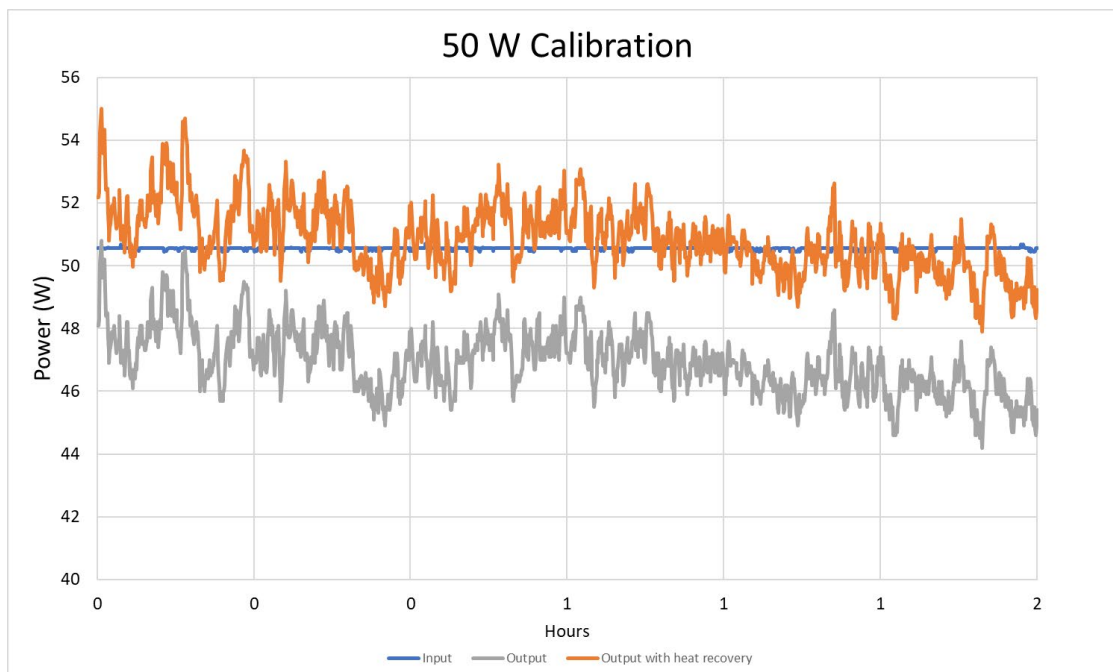
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Figure 7. A calibration shows a close balance between input and output power. The output heat is shown (gray), and then adjusted to account for heat losses from the calorimeter chamber walls (orange).

Control by temperature

The graphs and the conclusions in this section are based on 55 sample results from the R19 reactor shown in Table 1. These were taken with same nickel mesh sample over 111 days, between February 20 and May 23, 2019. They included 22 tests with 100 W input, 31 tests with 200 W input, and 2 calibration tests with no input power. In most cases, the reaction produced 35 to 100 W, or ~45% excess power.

Table 1. Excess heat results from the R19 reactor

Date (Month/Day)	Pressure (Pa)	Input (W)	Out/In	Excess heat (W)	Reactor Temp.	Heat per gram of Ni (W)	H/Ni atom
2/20	5412.0	100.0	1.39	38.63	238.90	0.7154	0.000051
2/21	6320.0	197.4	1.40	78.31	386.00	1.4501	0.000115
2/26	5949.0	50.1	1.21	10.42	145.00	0.1929	0.000128
3/22	5421.0	99.3	1.42	41.84	234.00	0.7749	0.000312
3/23	600.0	98.6	1.42	41.06	229.00	0.7604	0.000269
3/25	120.0	98.2	1.40	39.64	232.00	0.7340	0.000250
3/26	78.4	98.0	1.43	42.30	232.70	0.7834	0.000232
3/27	46.3	98.0	1.41	40.46	232.60	0.7493	0.000222
3/28	35.3	97.9	1.42	40.89	232.00	0.7573	0.000214
3/29	28.0	97.7	1.40	39.20	231.50	0.7260	0.000208
3/30	25.4	97.5	1.39	38.02	230.10	0.7040	0.000201
4/1	17.4	97.4	1.41	40.07	231.96	0.7419	0.000197
4/2	19.5	97.5	1.40	39.43	232.36	0.7301	0.000193
4/3	4644.0	97.3	1.40	39.03	231.83	0.7228	0.000280
4/4	4553.0	97.2	1.42	40.37	233.10	0.7476	0.000310
4/5	4092.0	152.3	1.50	76.17	320.00	1.4105	0.000502
4/6	1932.0	200.9	1.51	102.05	382.70	1.8898	0.001005
4/8	3751.2	200.8	1.49	97.62	383.30	1.8078	0.001670
4/9	3670.0	200.8	1.49	97.94	382.60	1.8137	0.002280
4/10	3268.0	201.0	1.49	97.64	384.50	1.8081	0.002825
4/11	3199.0	200.9	1.48	96.74	383.93	1.7914	0.003373
4/12	3173.0	200.9	1.48	96.04	383.10	1.7784	0.003959
4/13	2581.0	200.9	1.48	96.22	383.40	1.7818	0.004640
4/15	3042.0	200.7	1.51	101.67	381.00	1.8829	0.005211
4/16	3207.0	200.6	1.47	94.24	380.80	1.7451	0.005758
4/17	3058.0	200.6	1.46	91.95	381.90	1.7028	0.006624
4/18	2646.0	200.6	1.47	93.22	382.57	1.7263	0.007206
4/19	2546.0	200.6	1.46	91.97	380.70	1.7031	0.007875
4/20	2676.0	200.5	1.47	93.42	378.16	1.7300	0.008453
4/21	2903.0	200.5	1.47	93.39	377.56	1.7295	0.008973
4/22	2863.0	200.5	1.45	91.09	377.40	1.6868	0.009531
4/23	2771.0	200.5	1.47	94.49	377.00	1.7499	0.010095
4/24	2773.0	200.5	1.47	93.44	377.00	1.7303	0.010680
4/25	2761.0	200.4	1.46	92.74	376.00	1.7173	0.011250
4/26	2831.0	200.5	1.47	94.42	377.00	1.7485	0.011834
4/27	2111.0	200.4	1.46	92.17	375.50	1.7068	0.012600
4/29	1996.0	200.4	1.46	91.15	377.80	1.6879	0.013380
5/1	1156.0	200.5	1.47	94.23	378.30	1.7451	0.013397
5/3	1152.0	200.5	1.45	90.48	377.00	1.6755	0.013400
5/4	2537.0	200.4	1.44	88.44	377.60	1.6378	0.014008
5/5	2129.0	200.4	1.45	89.27	379.00	1.6532	0.014744
5/7	2560.0	200.4	1.45	90.04	378.68	1.6675	0.015345
5/8	2726.0	200.4	1.46	91.57	377.60	1.6956	0.015911
5/9	2800.0	200.4	1.45	90.33	377.60	1.6728	0.016462
5/10	42.4	200.3	1.45	89.61	374.00	1.6594	0.016463
5/11	2.3	200.4	1.44	88.53	373.50	1.6394	0.016464
5/13	5.3	100.3	1.35	35.09	235.00	0.6498	0.016465
5/14	5.6	100.0	1.34	33.97	236.00	0.6291	0.016464
5/15	5.8	99.7	1.37	36.41	236.00	0.6743	0.016464
5/16	6.4	99.5	1.35	34.32	236.25	0.6355	0.016463
5/18	7.4	0.0		2.11	23.64	0.0390	0.016463
5/20	7.5	0.0		2.89	23.07	0.0534	0.016463
5/21	5082.0	98.3	1.36	35.28	232.00	0.6534	0.016591
5/22	4778.0	98.2	1.34	33.82	232.00	0.6262	0.016664
5/23	4571.0	98.1	1.35	34.49	232.20	0.6387	0.016711

Excess heat increases exponentially with the increase in reactor temperature (Fig. 8). This allows good control over the reaction rate, by heating or cooling the reactor. Points are clustered in three groups because the tests were done at 100 W and 200 W input heating power, with 2 calibrations at 0 W.

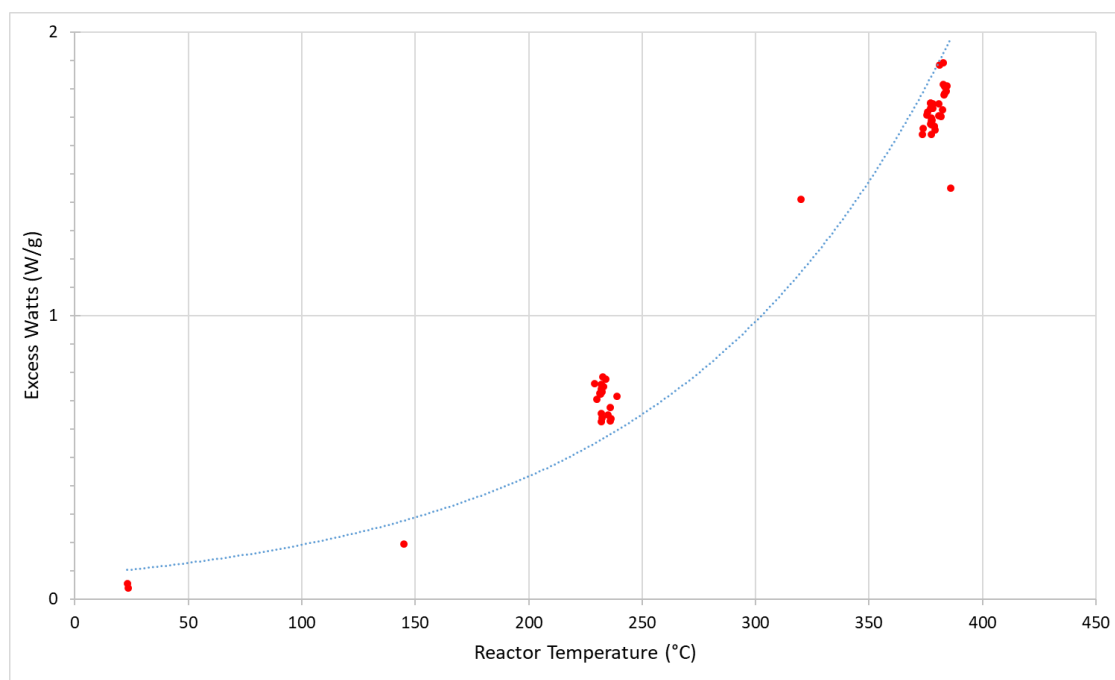The reaction turns on quickly, and quickly responds to an increase in temperature.

Figure 8. Excess heat generation per gram of nickel reactant at different reactor temperatures. $R^2 = 0.959$. Data from Table 1.

Permeability, not high loading, is necessary

The results in Table 1 suggest that high permeability is necessary for excess heat, but high loading is not. On the contrary, high loading apparently reduces excess heat.

Nickel subjected to the treatment described in this paper can be loaded much higher than pure nickel [2]. This appears to be a necessary condition to produce excess heat. However, it also appears that it is not highly loaded deuterium itself, but rather the ability to load (permeability) that is necessary.

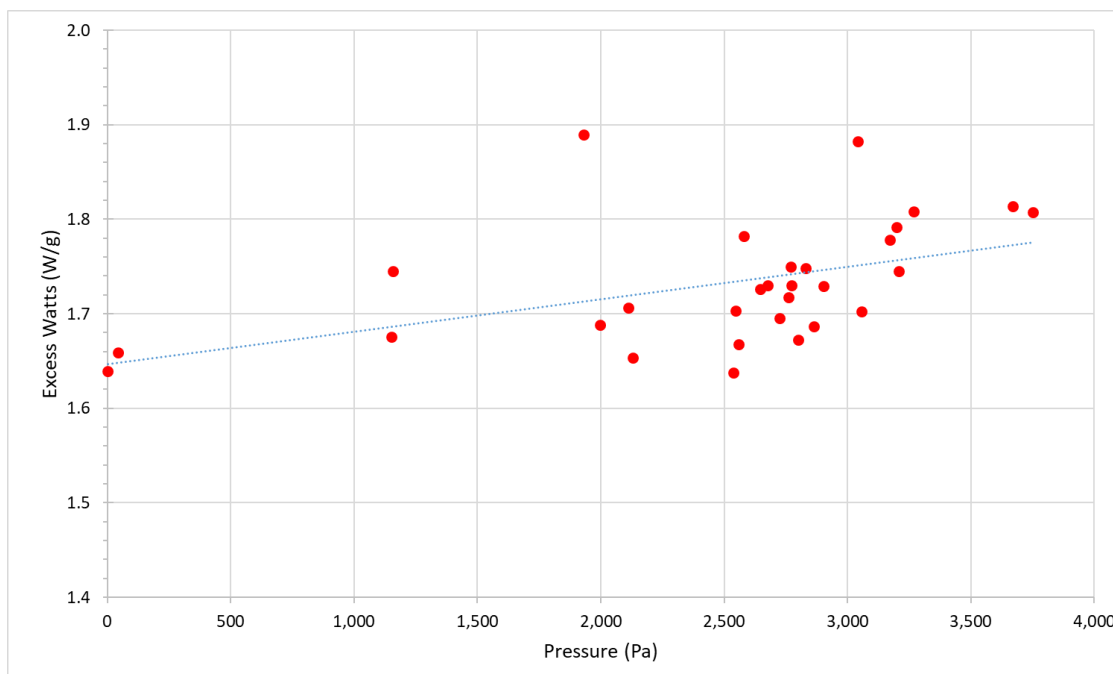Figure 9 shows there is no clear trend from pressure.

Figure 9. Deuterium pressure and excess heat, with 200 W input power. Data from Table 1.

Optimum pressure is between 100 and 300 Pa. It should not exceed 6,000 Pa. The reactant will probably not load at less than 100 Pa. However, as shown in Table 1, once it has loaded, pressure can be pumped down as low as 2.3 Pa and the reaction continues. The gas does not readily come out of from the metal once it loads, even when the gas is pumped out and the temperature is raised to 400 deg C. Table 1 shows that that pressure was reduced to 2.3 Pa on 5/11. It gradually rose to 6.4 Pa by 5/16 as the gas deloaded, but the reaction continued.

Figure 10 shows that the reaction strength is inversely proportional to loading. Loading close to zero produces 1.85 W/g of nickel. As loading increases, heat declines to 1.65 W/g. This is contrary to what has been reported with palladium, which is that high loading correlates with high heat. Perhaps this can be explained if what is needed is high permeability with relatively low loading. This might increase flux, which McKubre says enhances the cold fusion effect. [3]
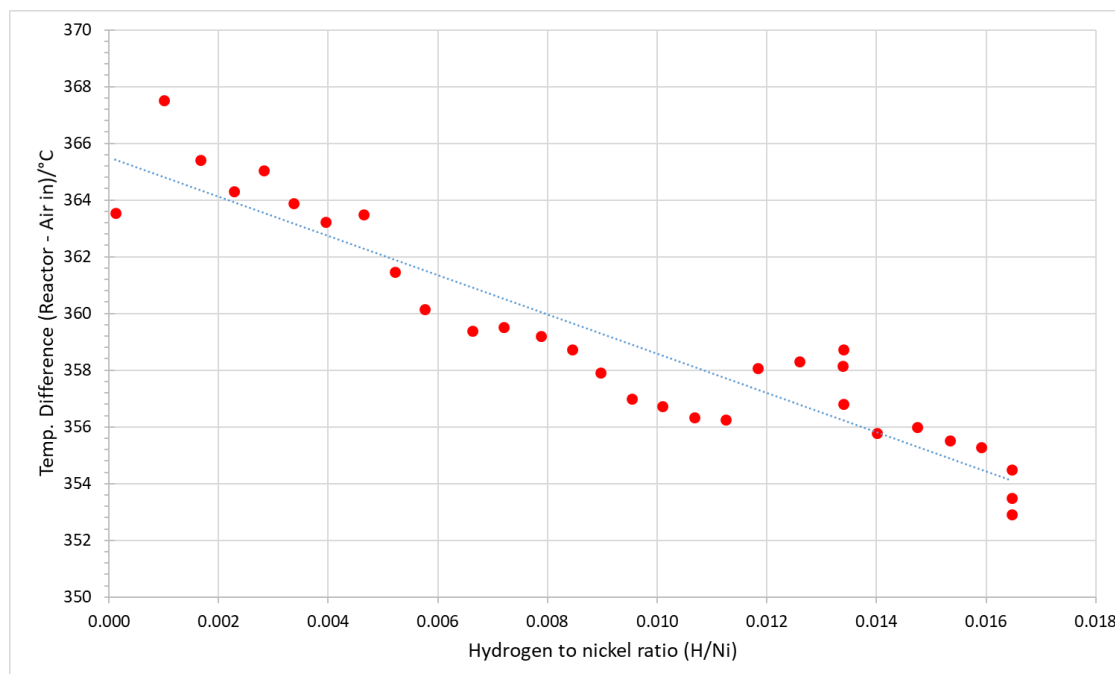
Figure 10. Difference between reactor body temperature and inlet air temperature, versus the D/Ni ratio.

The amount of gas absorbed by the nickel must be monitored to measure loading, to confirm the material is more permeable than ordinary nickel. It will not produce excess heat otherwise. However, once it is determined that it can be highly loaded, it should be de-loaded and run at low gas pressure, as described in the Method section below.

Loading is measured by monitoring gas pressure with a precision meter (ULVAC, GCMT G-Tran ISG-1). When a cell is left with no nickel reactant for several weeks, pressure does not change significantly. If there were a leak, pressure would rise because the cell is far below atmospheric pressure. If the stainless-steel reactor walls absorbed gas, the pressure would fall. During the tests, pressure initially falls every day. The nickel-palladium reactant must be absorbing the gas. Gas present at beginning of test and gas added during the test is inventoried to measure loading.

Then the material absorbs gas, or outgasses, the pressure usually changes a few Pascals per day. During rapid absorption it may change 10 or 20 Pa per day.

In the series of tests in Table 1, loading varied over the 111 days, reaching a peak when 176 cm^3 of deuterium was absorbed. The nickel reactant weighed 54 g. This mass of pure nickel would only absorb 0.9 cm^3 of deuterium according to textbooks. [2] A small amount of the gas is absorbed by the palladium adhered to the nickel, but most of the gas goes into the nickel. The total weight of the applied palladium layer is ~50 mg, or 4.7×10^{-4} mol. The saturation concentration of deuterium at this pressure is PdD0.7, so it absorbs 7.3 cm^3 deuterium gas, only ~4% of the total.

The interface of the nickel surface to which the Pd layer is pressure bonded is different from the nickel bulk, and has a complex structure with many defects in the Pd-Ni metal. It is likely these sites absorb hydrogen.

Materials: Reactant Nickel Mesh, and R20 Reactor

The reactant nickel mesh is the same material, prepared with the same methods, as we previously reported [1].

The nickel mesh is 0.055 mm wire × 180 mesh, dimensions 200 × 300 mm (Inada Kanaami, Inc.) (Figs. 12, 13)

Palladium is applied to the nickel mesh screen, after the mesh has been cleaned and prepared with the methods described below. Two methods of applying palladium have been used. The first is to rub the nickel screen with a palladium rod. The entire surface is vigorously rubbed on both sides (Fig. 11). The second method is to deposit a palladium film on the nickel mesh surface by plating it with an electroless deposition solution of palladium of Pd-10 (High Purity Chemical Co., Ltd.), Pd concentration 10 g/L. The plating conditions are: 40 to 60°C, pH 1.5.

In the tests reported in this paper, the nickel meshes were prepared by rubbing the screen, rather than electroless deposition, to save money. The plating solution is expensive.

To install the meshes in the reactor, the three meshes are stacked on top of one another, rolled up, and then unrolled inside the reactor so that the stack is against the reactor walls (Fig. 12).
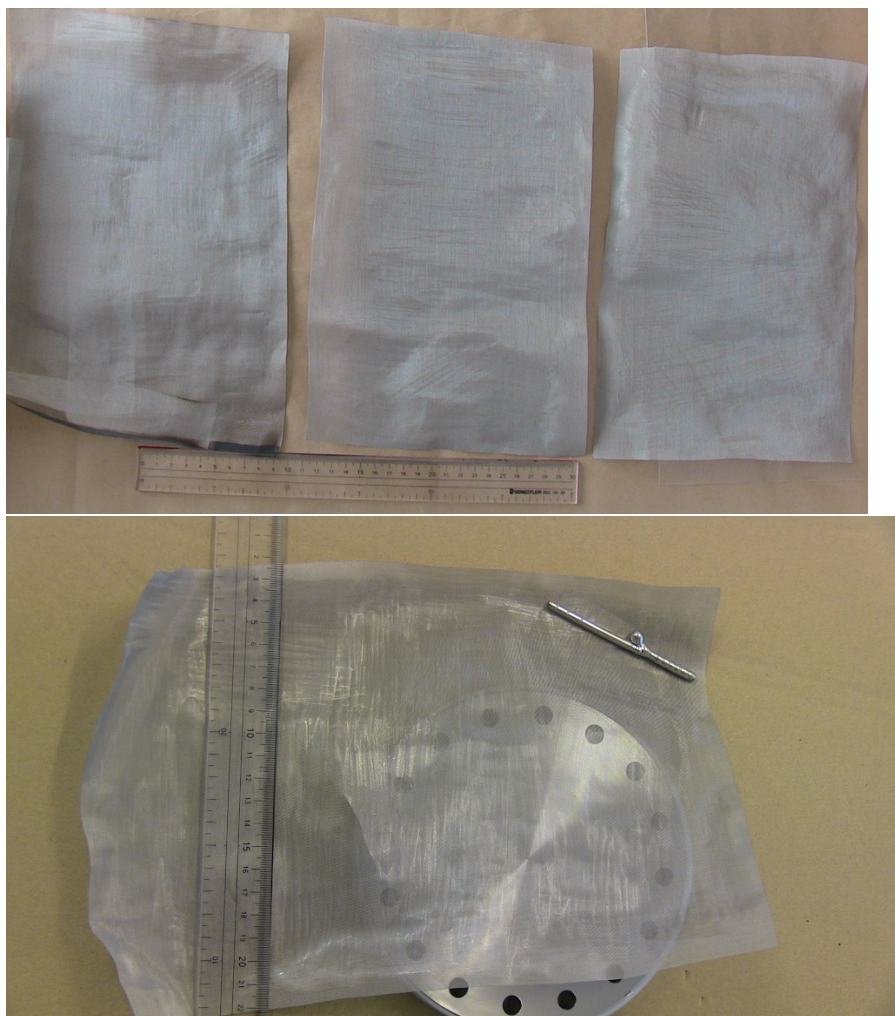
Figure 11. (top) Three nickel meshes are inserted into the reactor. Each is 20 × 30 cm, 180 mesh. Total weight 54 g. (bottom) Mesh with palladium rod. The effect of vigorous rubbing with the rod can be seen. The object under the mesh is the reactor cell flange.

Figure 12. The stack of 3 nickel mesh reactants shown mounted in the reactor, unrolled against the reactor wall.

The R20 reactor is the most effective version we have developed (Figs. 13-17). The major difference between this and previous versions is that it is heated internally with a sheath heater.

This change, along with changes in the methods and pressures, apparently enhanced the reaction, producing the results shown in Fig. 6.

The reactor vessel and flanges are stainless steel. The vessel is 600 mm long, diameter 114 mm, wall thickness 3.2 mm (1/8 inch). The flanges are 150 mm in diameter. Weight 20 kg. A sheath heater is axially mounted in the reactor vessel. The power supply for the heater and the gas pipe passes through one of the flanges (Fig. 16). A thermocouple is mounted on the outside of the vessel.
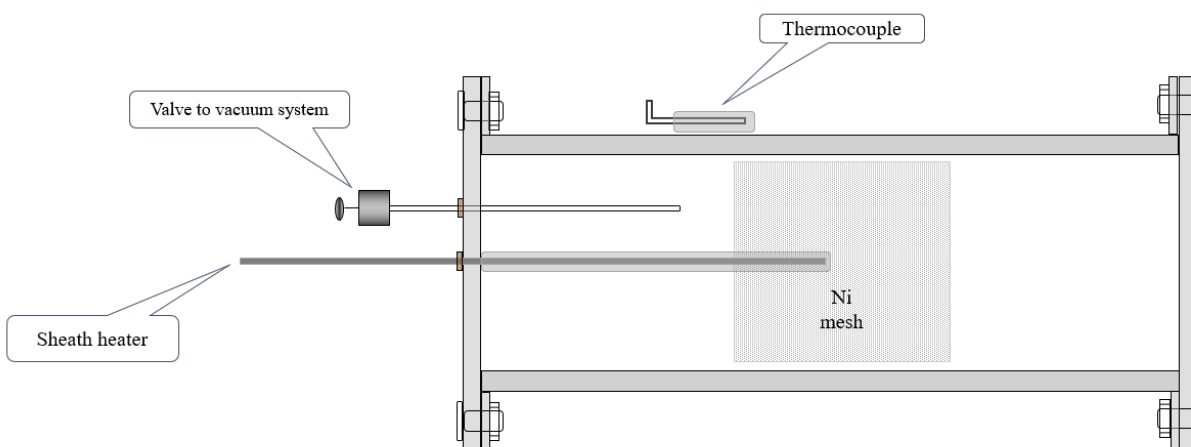
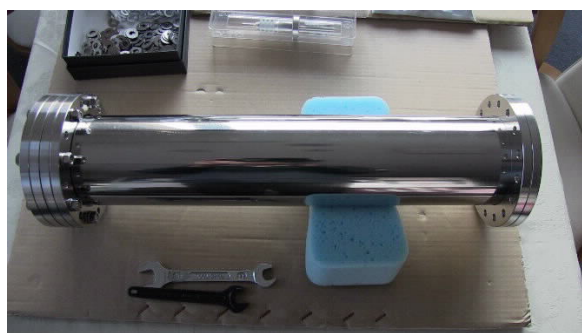Figure 13. Schematic of R20 reactor.

Figure 14. R20 stainless steel reactor vessel.

Figure 15. Reactor vessel stainless steel flange.

Figure 16. Stainless steel flange with electric power and gas connectors.

Figure 17. Swagelok valves.

Methods

The method of preparing the reactor vessel, the reactant mesh material, and the methods of testing and controlling the reaction are described here.

When handling these materials, use disposable gloves to avoid contaminating the reactants.

Three nickel meshes are cleaned, prepared and then coated with palladium. The nickel mesh preparation steps are as follows:

(1)   Wash with a mild detergent (Fig. 18) in ordinary tap water, and scrub with a plastic dish scrubbing pad. (Fig. 19).

(2)   Sand with water resistant sandpaper, starting with 500 grit, then 800, 1200, then 1500 (Fig. 20). Wash again with mild detergent plastic scrubbing pad, and rinse with tap water.

(3)   Soak in tap water at about 90°C for 1 hour.

(4)   Wash with ethyl alcohol. This process removes oil and roughens the metal surface.

Figure 18. Mild detergent used to clean mesh (Kao, Inc., Kyukyuto Orange scented brand). Ingredients: surfactant (37%, higher alcohol (anion), sodium dialkyl sulfosuccinate), stabilizer, disinfectant.
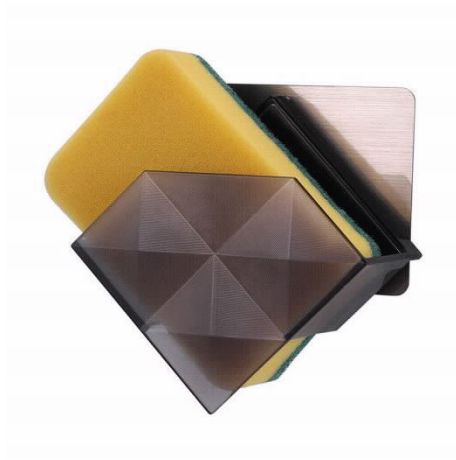

Figure 19. The scrub pad side of this is used to clean the mesh. (Sevenhope brand sponge-scrub pad)
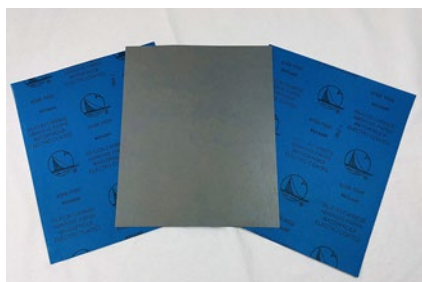
Figure 20. Water resistant sandpaper. Silicon carbide: DCGS: cc-500, cc-800, cc-1200, cc-1500 (Sankyo Rikagaku Co., Ltd.)

After cleaning:

(1)   Stack the three meshes, roll them, and place them in the reactor by unrolling them against the wall. Evacuate to 1 to 2 Pa at room temperature, and hold for about 2 hours.

(2)   Heat treatment, in which pretreatment (removal of impurities and surface refinement) of the reactant metal surface is carried out. The temperature is $100 \sim 120°C$, duration $5 \sim 20$ h.

(3)   Evacuate. Evacuation must be thorough, down to 10^{-2} Pa. To confirm the removal of impurities, the amounts of oxygen, nitrogen, water, and other substances in the evacuated gas should be measured by mass spectrometry. Even when the reactor is evacuated, a slight amount of H2O gas, nitrogen and oxygen remain. 70% of the residual gas is H_2O. In Step 6, below, the reactor is heated to remove the H_2O. The evacuation and heating may need to be repeated until the Q-Mass component peaks for H_2O (16, 17, and 18) are below the ion-current value for the Q-Mass of 10^{-9} A.

(4)   Heat for 1 to 2 h at 200°C with the sheath heater.

(5)   Cool down in the reactor, 1 - 2 h.

(6)   If no excess heat appears in the next step, or if a significant amount of H2O remains in the cell, steps 4 - 6 may need to be repeated.

To produce excess heat:

(1) Set the deuterium gas pressure to between $100 \sim 300$ Pa. The reactant will probably not produce heat at less than 0.1 Pa, and it is better not to exceed 6,000 Pa.

(2) Raise the temperature 100°C with the sheath heater.

(3) The calorimeter should show excess heat. The amount of excess heat depends on the degree to which the nickel surface is activated and also on the temperature. Excess heat should increase at higher temperatures.

(4) If there is no excess heat, raise the temperature higher.

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It is not clear whether the choice of detergent or the type of plastic in the scrubbing brush has an effect on the outcome of this experiment. However, cold fusion is usually sensitive to materials, and the cause of the reaction is not known, so we recommend that people trying to replicate use similar materials, including a detergent with a similar formula. Also, as noted above, we recommend an air-flow calorimeter.

Conclusions

As far as we know, this experiment has the best reproducibility and control, and the highest power output of any cold fusion experiment on record. The output to input ratio is also one of the best on record. The experiment produced 250 W of excess power in the calorimeter, and approximately 3 kW in tests outside the calorimeter.

Comparing these results to what we reported at ICCF21 [1], we have learned the following:

- Performance was improved with a better reactor design. Other materials and methods are unchanged.
- Temperature controls the reaction rate.
- Starting up and modulating the output is rapid, and under good control.
- Pressure should be low, between 100 and 300 Pa.
- High loading is not needed, or desirable. It will inhibit the reaction.
- High permeability is needed, perhaps because it increases flux.

This experiment is remarkably simple. We hope that people skilled in the art can replicate it. If any researcher wishes to replicate and needs more information, please contact us.

Acknowledgements

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