

**HEAT MEASUREMENT DURING THE ELECTROLYSIS
USING MODIFIED PALLADIUM CATHODE**

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ABSTRACT

The heat balances during the electrolysis using 2 types of Pd cathodes (B controlled Pd and Ni coated Pd) in 1M LiOD heavy water solution have been measured using the flow calorimeter system. The excess heat was observed at 6 runs out of 14 experiments for B controlled specimen. Among them, the heat burst was observed at 2 runs. For Ni coated specimen the small excess was observed at 4 runs out of 9 experiments. Further study is necessary to improve the reproducibility and to confirm the phenomena.

1. INTRODUCTION

Many positive or negative reports have been published since the announcement of M.Fleischman and B.S.Pons about the excess enthalpy production(the cold fusion) during the electrolysis in LiOD heavy water solution using palladium cathode¹⁾. However, the phenomena is not clear and many people could not believe it mostly from the theoretical point of view. At least a scientifically precise experiments and reliable data are necessary in order to confirm the phenomena

As far as the heat measurement concern, a direct measurement or an absolute measurement should be applied considering the scientific importance of the results. From that point we are applying the flow calorimeter using the thermochemically closed cell from the start of this study in 1989.

The reproducibility is crucial and most important for this. Several other groups also reported the excess heat using Pd cathode²⁻⁴⁾. However, their reproducibility is not enough to confirm the phenomena. The experimental

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recipe to get the excess enthalpy for everyone who wants to do should be presented. There are many factors to think of to get the reproducible results; materials, the electrolysis conditions and so on. Among them we think the Pd material is most important. From this standpoint we have reported the effect of the mechanical treatment, the heat treatment and the effect of some additives⁵⁻⁷.

In this paper we will report our recent results of heat measurements during the electrolysis in LiOD heavy water solution using 2 types of Pd electrodes; boron controlled Pd and Ni coated Pd.

2. EXPERIMENTAL

The electrolysis cell was made by acrylic resin in order to avoid the effect of alkaline solution. Figure 1 shows the schematic drawing of the cell. The recombination catalyst was placed on the upper part of the cell where the recombination reaction (D_2+O_2 or H_2+O_2) proceeded completely up to 4A. The recombination catalyst was Teflon treated fine Pd powder on alumina. The flow calorimetry was applied to the measurement of heat balance during the electrolysis. The copper tubing was surrounded the electrolysis cell and picked up the generated heat by the electrolysis and the recombination reaction. The increase of the temperature of cooling water was measured by CA thermocouple or Pt resistor. The temperature of cooling water was 296 K or 288 K.

2 types of the heat measuring system have been constructed. One is the electrolysis cell in a box of ceramic wool insulator where the heat recovery was up to 99 %. The other is the cell in the newly developed fine temperature controlled bath where inside temperature was controlled $\pm 0.01K$ at the temperature range from 283 to 333 K and the heat recovery was 96 %.

The cathodes were Pd rods(4 mm ϕ x 15 mm) containing B (mostly 500 ppm) and Ni coated Pd rod(2 mm ϕ x 15 mm) where Ni was electrically plated using sulfamate solution. These materials are obtained from IMRA Material(IM), Tanaka Kikinzoku Kogyo(TNK) and Tokuriki Corp.. The anode was Pt wire(1 mm ϕ) which was surrounded the Pd cathode.. The electrolyte was mostly 1 M LiOD heavy water solution which was made by LiOD- D_2O (99 at% D) powder.

The constant power electrolysis was applied in order to get clear heat balance using a constant power generator. The temperature of the electrolyte was between 293 and 323 K during the electrolysis.

3. RESULTS AND DISCUSSION

Table 1 shows the heat balance of B controlled Pd specimens. N1 and N2

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in the first column means the heat measurement using the fine temperature controlled bath. The electrolyses were conducted mostly at the fixed power of 5 W and the current density ranged from 300 to 1100 mA/cm². The average heat balance means the ratio of (total output heat)/(total input power) throughout the electrolysis. The maximum excess heat means the maximum excess power during the electrolysis when the excess heat was observed.

Among 14 runs we observed excess heat 6 times. During the run N1-03 we observed the heat burst 2 times using 500 ppm B containing Pd cathode. The result is shown in Fig.2. The heat burst took place at 225 h and 1020 h and we got maximum excess of 1.8 W at 1020 h for a half hour. The temperature of electrolyte and the cell voltage(or current) changed owing to the heat burst. This kind of abrupt heat burst did not observed at other runs and could not be reproduced even using the same Pd specimen.

Table 2 shows the result of the heat balance during the electrolysis using the Ni coated Pd cathode. In these experiments we want to see the effect of Ni/Pd interface, although the amount of absorbed deuterium(hydrogen) decreased. We observed small excess heat for 4 runs out of 9 runs. These excess heat appeared continuously during the electrolysis. Figure 3 shows the typical excess heat using the Ni plated Pd cathode. Since these excess are so small and very close to the error limit, further study is necessary to confirm this.

4. CONCLUSION

We observed excess heat several times using B controlled Pd and Ni coated Pd. However, the excess heat is mostly very small and the reproducibility has not been improved with applying these treatments. The essential factor to produce excess heat is still not clear. Further study is necessary to improve the reproducibility and confirm the phenomena.

Acknowledgment

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Table 1. Results of electrolysis using B containing Pd.

Run	Pd sample	Current density (mA/cm ²)	Win (W)	Heat balance Ave.(%)	Excess heat (W) Max	Temperature (°C)
23	B 500ppm (IM)	750~300	5	103.5	0.29	
25	B 127ppm (TNK)	850~440	5	99	-	
29	B 500ppm (IM)	750~360	5	101	0.22	
31	B 267ppm (TNK)	900~540	5	101	0.14	
33	B 500ppm (IM)	740~660	5	102	0.14	23
34	B 500ppm (IM)	1180~500	5.1	100	-	
37	B 1000ppm (TNK)	1040~950	5	100	-	
48	B 500ppm (IM)	910~840	5	101	0.16	
49	B 500ppm (IM)	1020~900	5	99	-	
N1-03	B 500ppm (IM)	590~560	5	101	1.8	15
N2-01	B 267ppm (TNK)	810~780	5	99	-	15
N2-02	B 500ppm (IM)	640~320	5	100	-	23
N2-03	B 500ppm (IM)	750~700	5	100	-	15
N2-04	B 500ppm (IM)	700~530	5	99	-	15

Table 2. Results of electrolysis using Ni coated Pd.

Run	Pd sample	Current density (mA/cm ²)	Win (W)	Heat balance Ave.(%)	Excess heat (W)	Temperature (°C)
46	Ni-coated (10 μ m, D ₂ O)	1230~490	5	103	(0.28)	
47	Ni-coated (10 μ m, D ₂ O)	1090~700	5	100	0.23	
50	Ni-coated (10 μ m, H ₂ O)	1530~1310	5	100	-	
51	Ni-coated (10 μ m, D ₂ O)	1380~530	5	99	-	23
52	Ni-coated (10 μ m, D ₂ O)	1210~460	5	100	-	
53	Ni-coated (10 μ m, H ₂ O)	1660~1340	5.8,6	102	(0.19)	
54	Ni-coated (10 μ m, H ₂ O)	Testing now	5	100	-	
55	Ni-coated (1 μ m, D ₂ O)	Testing now	5	100	-	
N1-06	Ni-coated (10 μ m, D ₂ O)	1040~700	5	101	(0.1)	23

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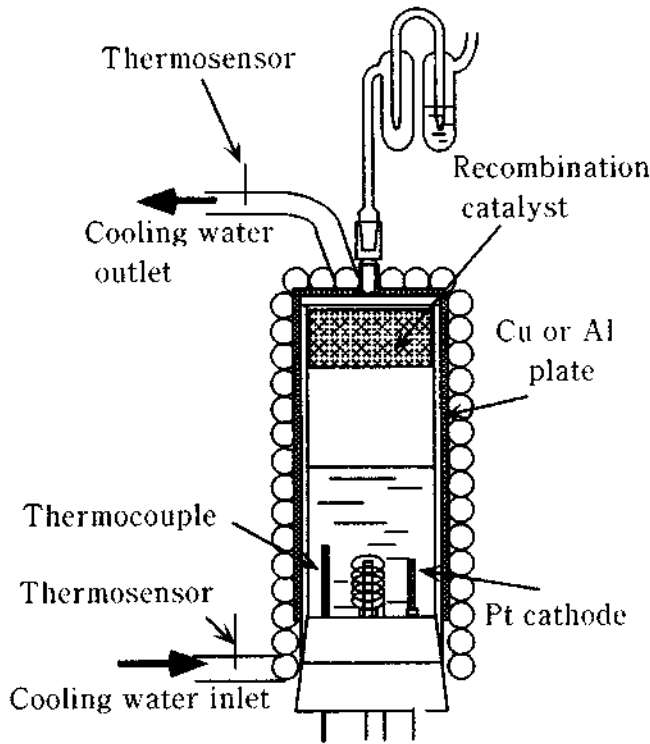


Fig.1. Electrolysis cell.

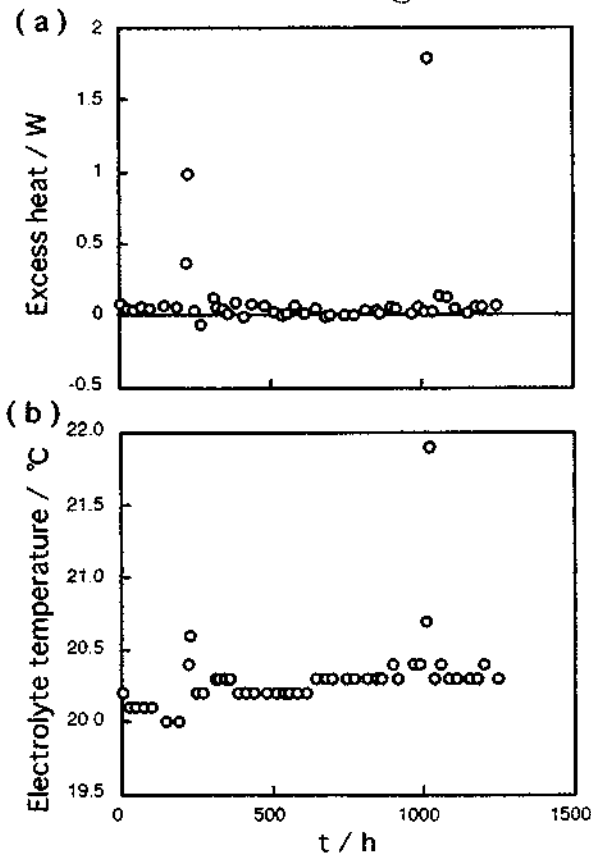


Fig.2. (a) Excess heat (b) Electrolyte temperature of N1-03 (B 500ppm)

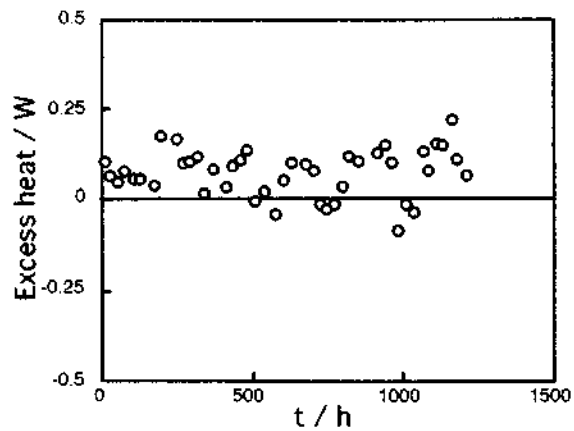


Fig.3. Excess heat of Run-46 (Ni-coated 10 μ m).