

## MDE (Metal Deuterium Energy) Project 2009 Results Explanation File

Using abstracts for JCF9 and ACS2009 and ppt slides for ACS2009, results of MDE 2008 fiscal year  
are explained in the following.

[JCF9 abstracts 1-3]

### CMNS Research Progressing in Kobe University -Deuterium Permeation and Absorption-

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Experimental studies on condensed matter nuclear science (CMNS) ongoing in Kobe University are reviewed. One is the subject of nuclear transmutation during forced permeation of deuterium (D) through a multi-layered film of X/CaO/Pd, with X being an element to be transmuted, *e.g.*, <sup>133</sup>Cs transmuted to <sup>141</sup>Pr, <sup>88</sup>Sr to <sup>96</sup>Mo, *etc.*, which was originally claimed by Iwamura *et al.*[1]. In addition to the modified version of the sample exposure system with reversed flow direction installed at a beam line of a tandem electrostatic accelerator SSDH-2, a stand-alone D permeation system was used to examine the phenomenon by *in-situ* or *ex-situ* PIXE/ERD analysis, respectively.

Using Sr as the element X, more than ten samples having a variety of multi-layer structure have been examined. The PIXE spectra have shown that three out of fourteen samples had increased areal densities of Mo in exchange for decreased those of Sr after D permeation with integrated flow rates up to 2 - 20×10<sup>22</sup> cm<sup>-2</sup>. However, the K<sub>α</sub>-K<sub>β</sub> X-ray peaks in the spectra were too small to give sufficiently high reliability to the identification of the peaks, and to deduce definite conclusion of transmutation. It should be noted, nonetheless, that almost all samples with exception of only one had decreased areal densities of Sr after D permeation.

Tungsten was also tested as the element X. Assuming the regularity in transmutation that (atomic number, mass number) increase by (4, 8), we expected production of radioactive nuclei <sup>191</sup>Pt (*T*<sub>1/2</sub> = 2.86 d) from <sup>183</sup>W. An NaI(Tl) scintillation probe and a HPGe detector were employed to detect 0.538-MeV gamma rays from <sup>191</sup>Pt. The result of the measurement for the first sample has shown that the areal

density of  $^{191}\text{Pt}$  is smaller than  $10^7 \text{ cm}^{-2}$ , if any.

The second subject is to confirm heat and  $^4\text{He}$  generation by D absorption in nano-sized Pd powders reported by Arata and Zhang [2], and to investigate the underlying physics. We have installed a twin system to perform calorimetry during  $\text{D}_2$  or  $\text{H}_2$  absorption by micronized powders of Si, Pd, Pd-black, and Pd-Zr oxide compounds. The research is performed as a joint research program with Technova Inc., and is described in detail in the following two presentations [3, 4].

[1] Y. Iwamura, M. Sakano and T. Itoh; *Jpn. J. Appl. Phys.* 41 (2002) 4642-4650.

[2] Y. Arata and Y. Zhang; *The special report on research project for creation of new energy, J. High Temperature Soc., No. 1*, 2008.

[3] Y. Sasaki, A. Kitamura, T. Nohmi, T. Yamaguchi, A. Taniike, A. Takahashi, R. Seto, and Y. Fujita; *Deuterium Gas Charging Experiments with Pd Powders for Excess Heat Evolution, (I) Results of absorption experiments using Pd powders, (this meeting)*.

[4] A. Takahashi, A. Kitamura, T. Nohmi, Y. Sasaki, Y. Miyoshi, A. Taniike, R. Seto, and Y. Fujita; *Deuterium Gas Charging Experiments with Pd Powders for Excess Heat Evolution, (II) Discussions on Experimental Results and Underlying Physics, (this meeting)*.

## Deuterium Gas Charging Experiments with Pd Powders for Excess Heat Evolution

### (I) Results of absorption experiments using Pd powders

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To confirm heat and  $^4\text{He}$  generation by deuterium (D) absorption in nano-sized Pd powders reported by Arata and Zhang [1], and to investigate the underlying physics, we have installed a twin system of double structured vessels to perform flow calorimetry during  $\text{D}_2$  or  $\text{H}_2$  absorption by a variety of micronized Pd samples. The inner reaction vessels contain sample powders of Si, Pd, Pd-black, or Pd-Zr oxide compounds, and have calorimetry arrangement, *i.e.*, coolant pipes and thermocouples on the outer surfaces. The inner vessels are located in the outer chambers which are evacuated for thermal insulation. We can perform  $\text{D}_2$  and  $\text{H}_2$  absorption runs for two samples at the same time under the same environmental condition. Nuclear diagnostic tools, a 2"×2" NaI(Tl) scintillation probe and a neutron dose-rate meter, are also provided around the system.

The first-stage experiments are described in detail in ref. [2]. The evolution of pressure and temperature after introduction of 1-MPa  $\text{D}_2/\text{H}_2$  gas was divided into two phases. In the first phase, during which the pressure was kept well below 10 kPa, the D (H) loading ratio of D/Pd (H/Pd) and the specific output energy reached 0.85 (0.78) and  $0.54 \pm 0.10$  ( $0.45 \pm 0.08$ ) kJ/g-Pd, respectively, for the Pd-black, which were about 2 – 3 times greater than those for the  $0.1\text{-}\mu\text{m}\phi$ -Pd powder. Moreover, when

D<sub>2</sub> gas was used with Pd-black, apparent excess heat production in the second phase was implied, although temperature oscillations and drift were too large to confirm the result.

Then in the second stage, the system was modified to improve the accuracy: The heat capacity of the reaction vessel was decreased, while increasing the mass of the test sample, to minimize the time constant of the calorimeter and maximize the sensitivity.

First, repeated use of the Pd-black samples after one or more cycles of D (H) absorption-degassing has revealed the Pd-black powder stuck with each other to become larger micro-particles without nanoscale structure. Next, nano-sized powders of mixed Pd and Zr oxides fabricated by Santoku Cooperation, Kobe, Japan, have been used to reveal their interesting and exciting characteristics, which is discussed in detail in the succeeding presentation [3].

[1] Y. Arata, et al.: *The special report on research project for creation of new energy, J. High Temperature Society, No. 1. 2008.*

[2] T. Nohmi, Y. Sasaki, T. Yamaguchi, Taniike, A. Kitamura, A. Takahashi, R. Seto, and Y. Fujita: *Basic research on condensed matter nuclear reaction using Pd powders charged with high density deuterium, Proc. ICCF14 (Washington DC, Aug. 10-15, 2008).*

[3] A.Takahashi, A. Kitamura, T. Nohmi, Y. Sasaki, Y. Miyoshi, A. Taniike, R. Seto, and Y. Fujita: *Deuterium Gas Charging Experiments with Pd Powders for Excess Heat Evolution (II) Discussions on Experimental Results and Underlying Physics, (this meeting).*

## Deuterium Gas Charging Experiments with Pd Powders for Excess Heat Evolution

### (II) Discussions on Experimental Results and Underlying Physics

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Our experimental results [Ref-1] obtained for Pd/PdO/ZrO<sub>2</sub> nano-composite samples under the joint research of Kobe University and Technova Inc. in the 2008 fiscal year are summarized as:

- 1) Arata-Zhang's excess heat result demonstrated in May 2008 was replicated quantitatively.
- 2) For Pd/PdO/ZrO<sub>2</sub> powders (produced by Santoku Co. , Kobe Japan):
  - 2-1) D-gas charge in the 1<sup>st</sup> phase (zero pressure interval) gave 20~90 % excess heat than H-gas charge.
  - 2-2) In the 2<sup>nd</sup> phase of pressure rise (finally up to 1MPa), significant excess heat (about 2 kJ/g-Pd) for D-gas charge was observed, while near zero level excess heat for H-gas charge was observed.
- 3) No increase of neutron counts was seen, nor increase of gamma-ray counts.
- 4) D/Pd loading ratio in the end of 1<sup>st</sup> phase was 1.0, while H/Pd ratio was about 0.8-0.9.
- 5) Released energy per D or H-atom in the 1<sup>st</sup> phase was 1.7-1.8eV or 1.0-1.6eV respectively for D or H. These specific values are anomalously large, compared with known values of 0.5eV and 0.2eV per D or H respectively for surface-adsorption and lattice-absorption of hydrogen gas into bulk Pd

metal.

- 6) After evacuation, used Santoku sample retains 100 times more deuterium than Pd-black sample, seen by degassing data by baking up to 574°K.
- 7) In repeated run with used sample after #1, we can expect further steady excess heat. We observed similar effect as “heat after death” after evacuation of reaction chamber in the end of run.

For the 1<sup>st</sup> phase, we discuss the underlying surface and nano-particle physics in views of the enhanced surface adsorption potential by fractal sub-nano-scale trapping points on nano-Pd particle, the diffusion to inner shallower Bloch potential of regular Pd lattice, and the drastic mesoscopic and isotopic effect of surface and lattice rearrangement of nano-Pd particle by full D(H)-absorption to make deeper D(H) trapping potentials of surface adsorption (about 2eV for D) and intermediate surface state trapping.

We add discussions on the 2<sup>nd</sup> phase where we observed long lasting significant excess heat only for D-charging, in the view of possible 4D/TSC model type “condensed cluster fusion reaction” to produce excess heat and <sup>4</sup>He particles by preferred 4D fusion without neutrons and gamma rays.

*Ref-1) Y. Sasaki, A. Kitamura, T. Nohmi, Y. Miyoshi, A. Taniike, A. Takahashi, R. Seto, and Y. Fujita: Deuterium Gas Charging*

*Experiments with Pd Powders for Excess Heat Evolution, (I) Results of absorption experiments using Pd powders, JCF9,*

*Shizuoka March 28-29 2009 (this meeting)*

#### [ACS 2009 NET Session Abstract]

#### **Deuterium gas charging experiments with Pd powders for excess heat evolution**

##### **ENVR 51**

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We have started a series of deuterium (and hydrogen) gas charging experiments with Pd nano-powders to study possible heat evolution and D (or H)-loading characteristics by using a revised Arata-type twin system. The twin system is made of identically designed A1 and A2 systems, in each of which an inner gas-charging cell with flow calorimeter and an outer vacuum chamber are set up. The A1 system is used for D-gas foreground run, and the A2 system is for the H-gas blank run. Our first data with two commercially available Pd powders (0.1 micron Pd particles and Pd-black) are already meaningful. Experiments with Pd-black sample gave 2.6 kJ/g-Pd excess heat for the second phase of 1,300 minutes operation and D/Pd=0.85 for the first phase (about 100 min interval from start) with zero D-gas pressure. No excess heat with H-gas charging was seen with H/Pd=0.78. Experiments with 0.1 micron Pd powders gave D/Pd =0.45 for the first phase and much less excess heat for the second phase. We are extending experiments for nano-fabricated Pd samples to be reported at the meeting. *In situ* radiation monitors are for neutron and gamma-ray. Elemental analysis of “before/after” samples is done by PIXE. <sup>4</sup>He detection will be also tried.

# Deuterium Gas Charging Experiments with Pd Powders for Excess Heat Evolution

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to be presented at ACS2009 NET Session  
Salt Lake City  
March 22-26, 2009

It has been recently reported in ref. [1] that charging of **highly pure D<sub>2</sub> gas** into **Pd nano-powders** in the form of Pd/ZrO<sub>2</sub> nano-composite contained in a stainless-steel vacuum vessel has induced significant excess heat and <sup>4</sup>He generation.

We have constructed an experimental system to replicate the phenomenon of **excess-heat** (and <sup>4</sup>He generation) and **investigate the underlying physics for D(H)-charged Pd powders**.

[1] Y. Arata, et al.; The special report on research project for creation of new energy, J. High Temperature Society, No. 1. 2008.

## Outline of the Present Work

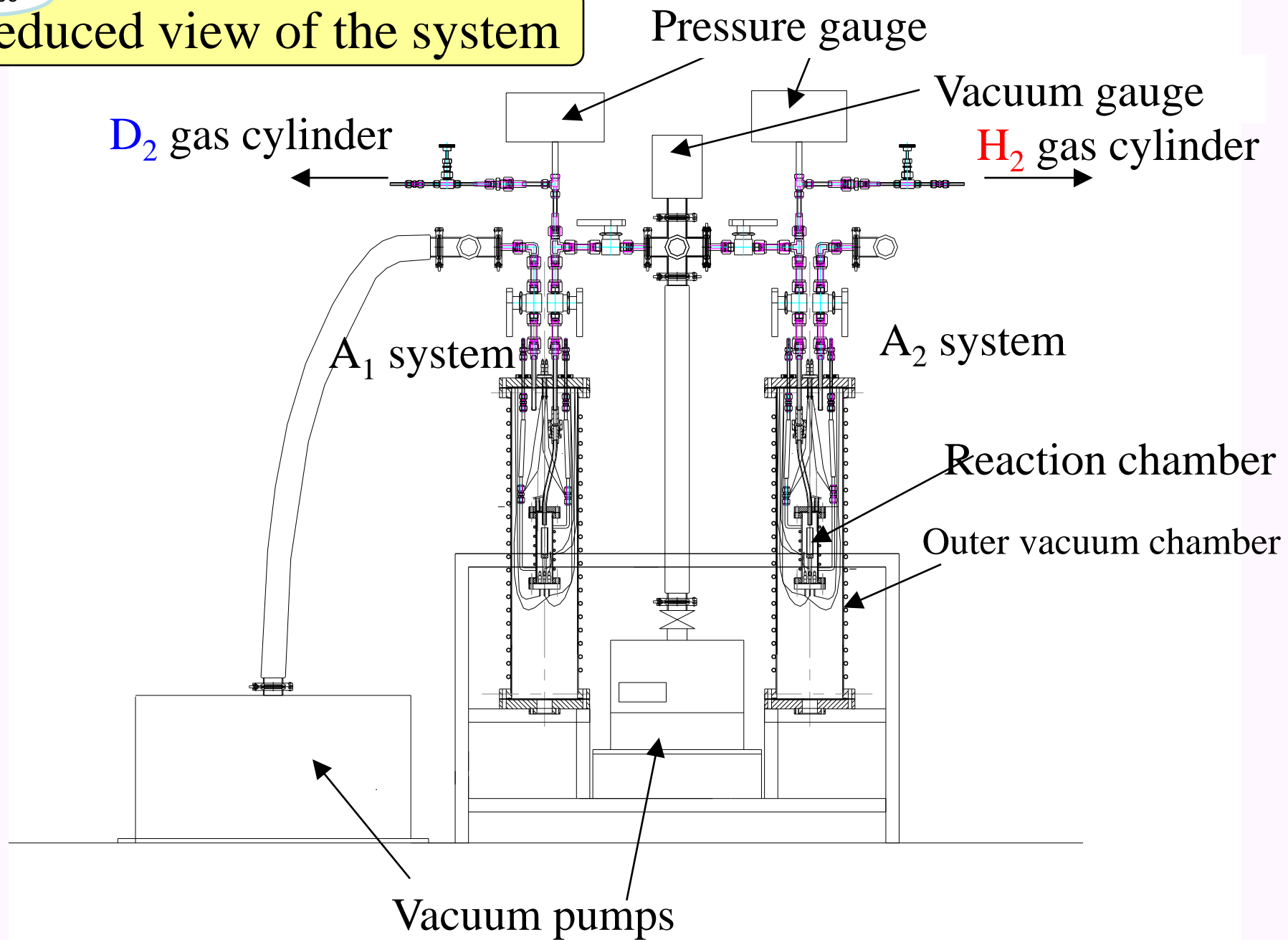
We constructed two identical chambers (**twin system**); one for  $D_2$  gas foreground run and the other for  $H_2$  gas background run. Each system has an inner reaction chamber containing **Pd powders** (**Pd-black** and **nano-Pd/ZrO<sub>2</sub>**) and the outer chambers are evacuated for thermal insulation.

A water-cooling system is provided for **flow calorimetry to estimate heat production rates for two phases.**

**D(H)/Pd ratios** were measured for the 1<sup>st</sup> phase (“zero pressure interval”).

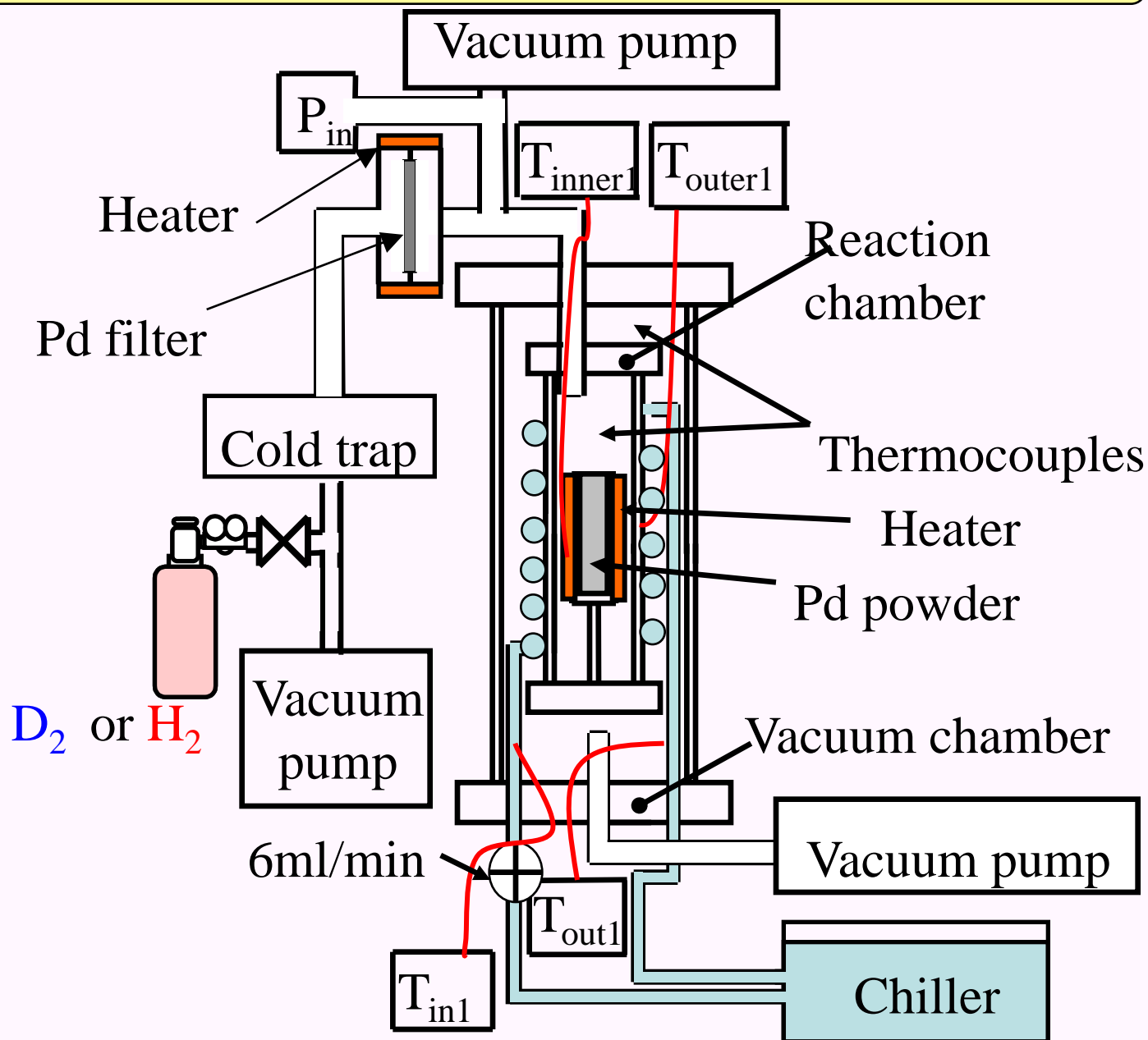
Moreover, after the gas charging, elemental analysis of the **Pd powder** was performed by PIXE (Particle Induced X-ray Emission) analysis. Radioactivity was measured with a Hp-Ge detector. A REM counter was used for monitoring neutron emission. NaI scintillator was used for gamma-ray. Furthermore,  $^4\text{He}$  analysis will be performed in the future.

# Reduced view of the system





## Functional view of the A system (Old)





Neutron Det.

Scintillation probe

A1 System

2008 7 18

# 1. Calorimetry Calibration

## Outline

The temperature of the reaction chamber is thermally isolated by evacuating the outer vacuum chamber.

Flow calorimetry is provided to measure heat output from the sample cup in the reaction chamber.

## Calibration of the calorimetry system

- Input power: 0.5W, 1W, 3W, 6W and 10W.
- D<sub>2</sub> gas pressure in the reaction chamber: 0, 0.1, 0.3, and 1.0 MPa.
- Flow velocity of coolant water: 6 ml/min.

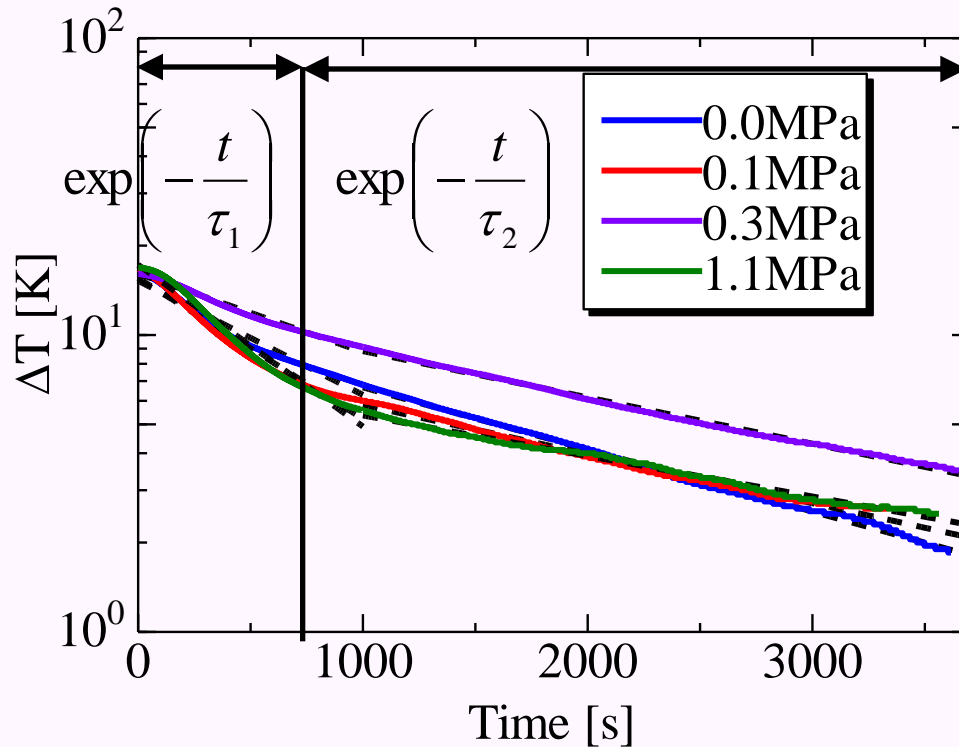
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## Results of calibration (Old A)

Pressure[MPa]	Input power [W]	Output power [W]	Heat recovery rate
0	10.0	5.9	59.3%
0	6.1	4.1	68.2%
0	3.0	2.2	72.5%
0	1.0	0.7	67.5%
3	10.0	7.0	70.2%
3	6.1	4.3	69.7%
3	3.0	2.2	71.8%
3	1.0	0.5	51.9%
1.1	10.0	6.8	68.1%
1.1	6.1	4.3	70.7%
1.1	3.0	2.0	68.3%
1.1	1.1	0.6	53.4%

Heat recovery rate is independent of pressure and input power, and is about 70% ( $\pm 2\%$ ).

Thermal time constant: temperature change following the change in the input power.

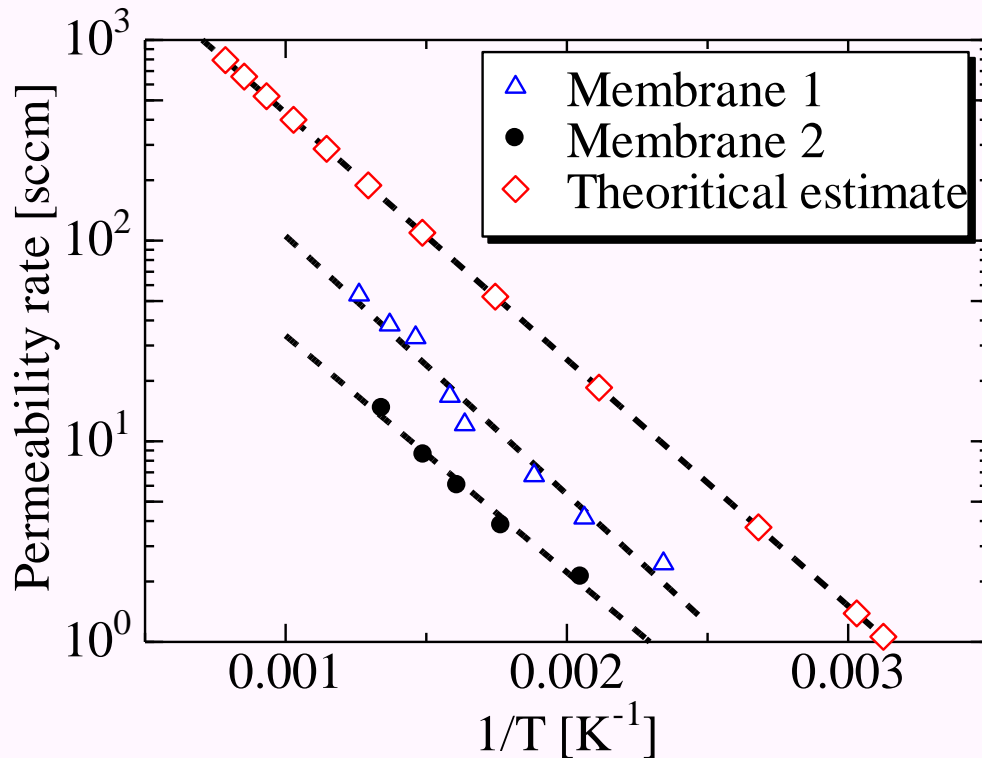


MPa	$\tau_1$ [s]	$\tau_2$ [s]
0	1500	2700
0.1	690	3000
0.3	750	2600
1.1	930	2000

Thermal time constants will be used to determine the phases in absorption runs.



# Performance test of Pd membrane filter

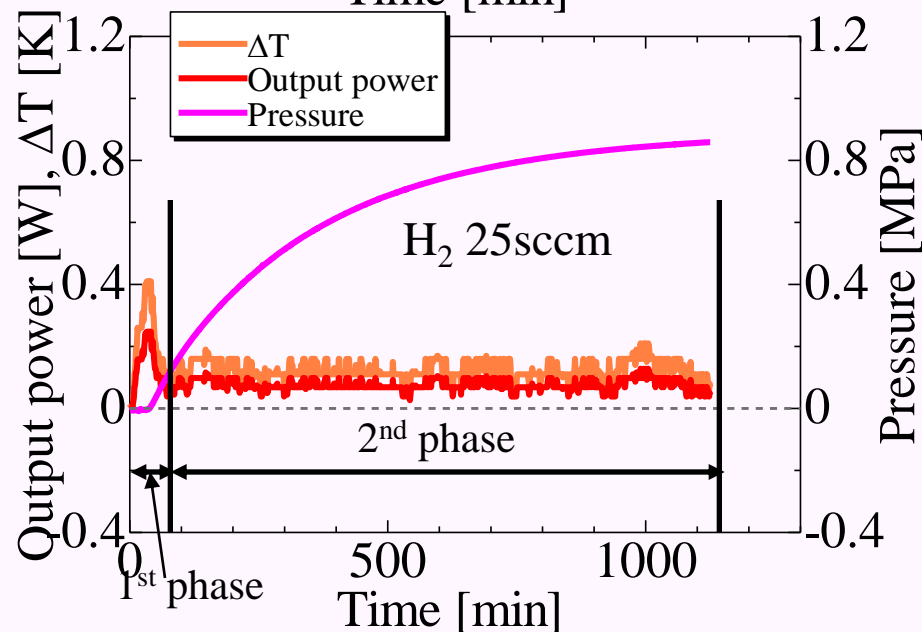
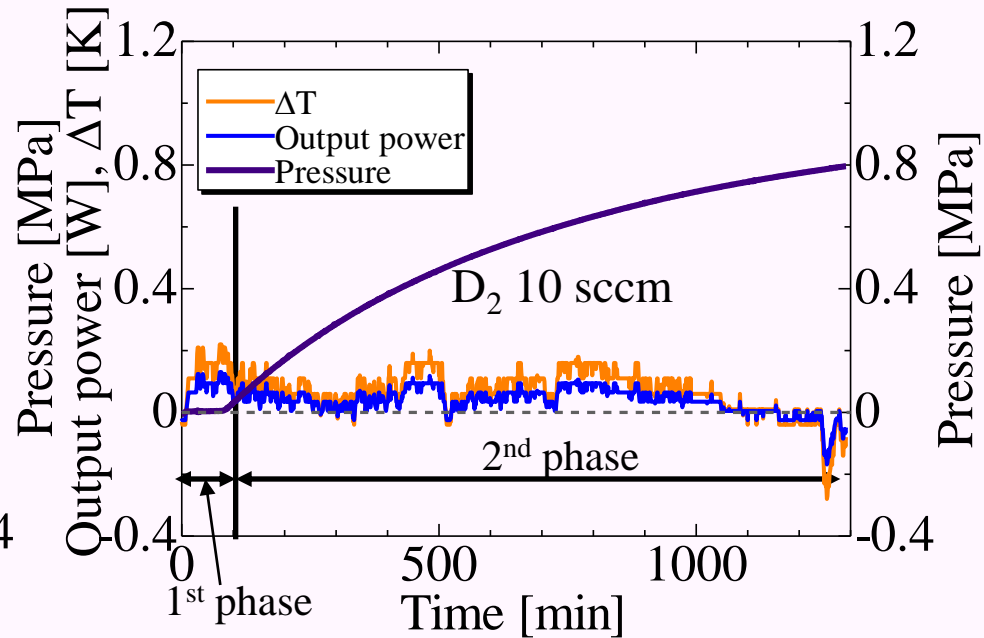
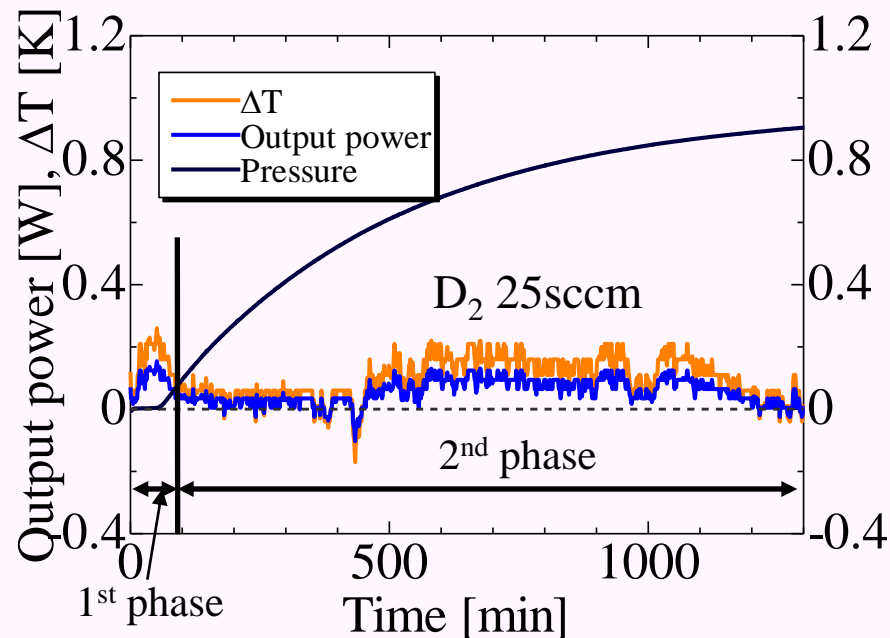


The **Pd membrane** (0.2 mm-t, 99.95%) separates the evacuated reaction chamber (1.6ℓ) and the gas reservoir filled with **D<sub>2</sub>** at 1 MPa.

Permeation rate of **D<sub>2</sub>** gas into the reaction chamber was derived as a function of membrane temperature by measuring the rate of pressure increase after stopping evacuation.

D<sub>2</sub> gas flow rate is controllable between 0.1 and **25 sccm** by varying the temperature from 298 K to **900 K**.

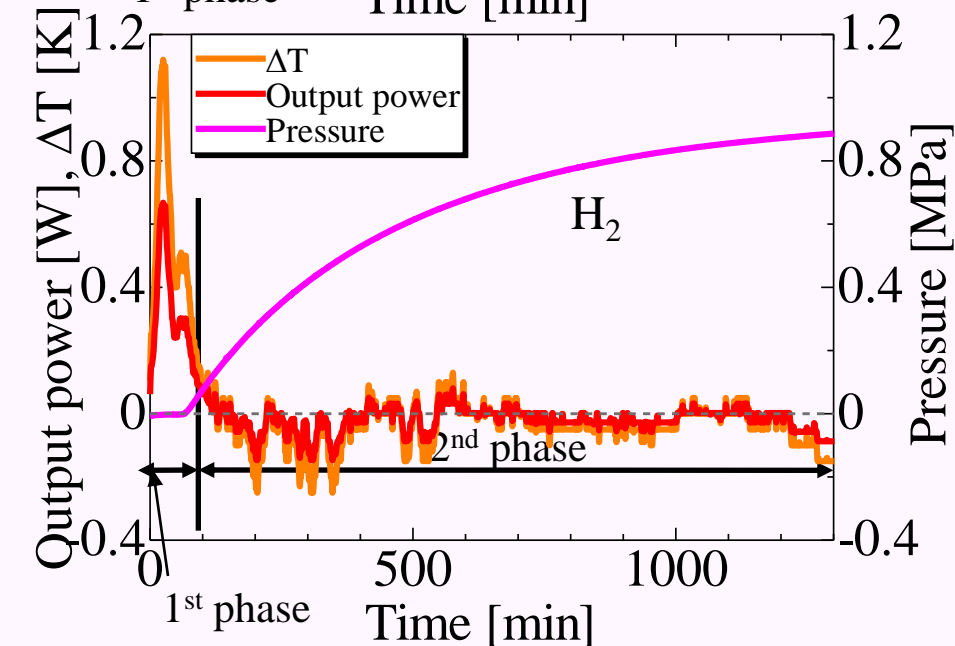
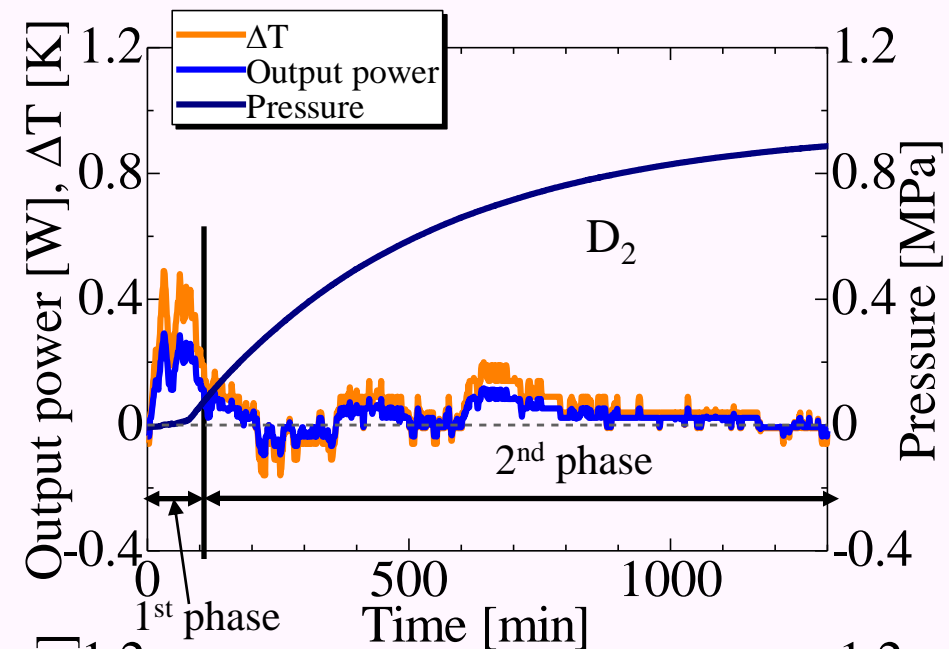
# Pd powders ( $\phi 0.1\mu\text{m}$ , 99.5%) – results



The evolution is divided into 2 phases by the time 30 minutes after the beginning of the pressure rise.

The release of heat of hydride formation should be completed in the 1<sup>st</sup> phase.

# Pd black (300mesh, 99.9%) – results



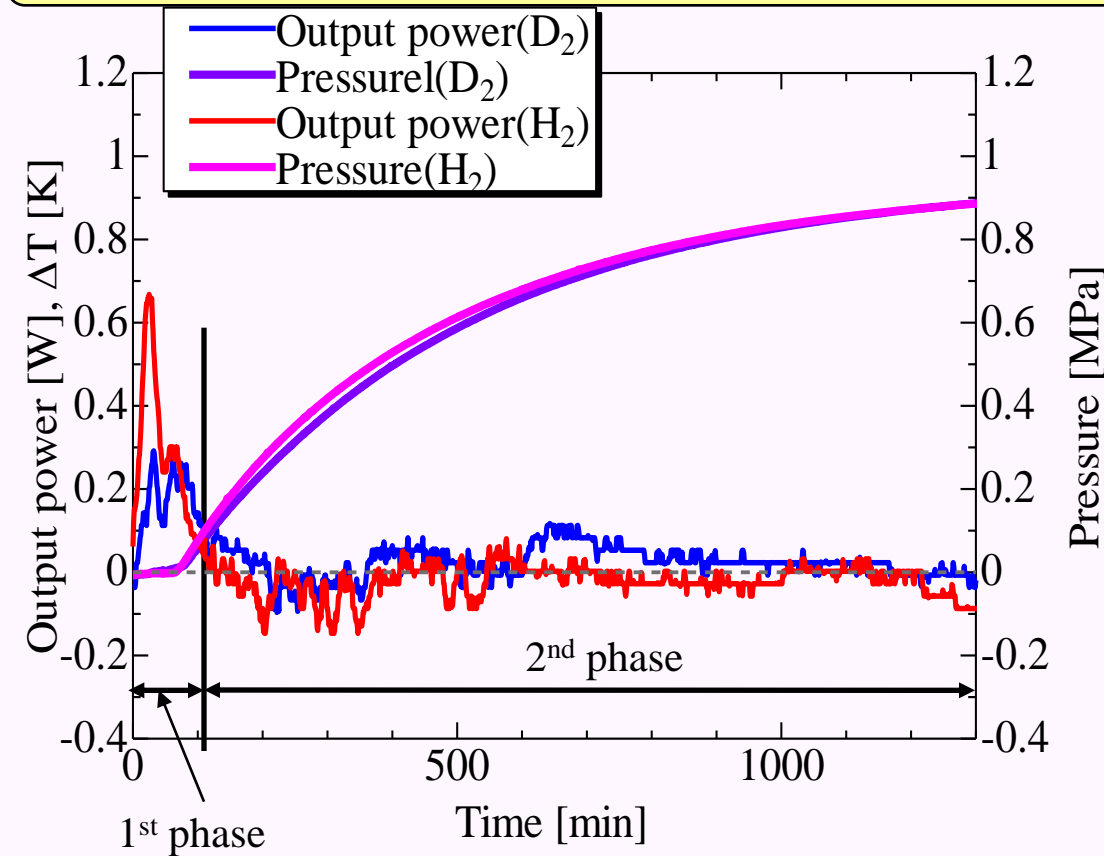
Likely to the case of Pd  $\phi 0.1\mu m$  sample, the evolution looks similar.

The evolution can be divided into 2 phases by the time 30 minutes after the beginning of the pressure rise.

The release of heat of hydride formation should be completed in the 1<sup>st</sup> phase.



## B. Pd black (300 mesh, 99.9%) – comparison of Heat balance



Heat balance

	D <sub>2</sub>	H <sub>2</sub>
1st phase	5.4E+2 J/g	4.5E+2 J/g
2nd phase	2.6E+3 J/g	-6.2E+2 J/g

- The output energies in the 1st phase are almost the same for both cases, but seem to be somewhat larger than the nominal values of 100 – 405 J/g ( $H_2$ ) and 80 - 330 J/g ( $D_2$ )
- On the contrary, the output energy in the 2nd phase appears to be larger for  $D_2$  than  $H_2$ .

## Summary of experimental results for the 1<sup>st</sup> Series

Sample	weight [g]	Gas	Nominal flow rate [sccm]	Measured flow rate [sccm]	1st phase [J/g]	2nd phase [J/g]	Total [J/g]	D/Pd or H/Pd	Absorption [scc]
0.1 $\mu\phi$ -Pd	5	D <sub>2</sub>	10	2.7	1.0E+02	5.2E+02	6.2E+02	0.46	245 $\pm$ 8
0.1 $\mu\phi$ -Pd	5	D <sub>2</sub>	25	3.5	9.7E+01	7.9E+02	8.9E+02	0.43	224 $\pm$ 8
0.1 $\mu\phi$ -Pd	5	H <sub>2</sub>	25	5.4	1.2E+02	1.1E+03	1.2E+03	0.45	236 $\pm$ 8
Pd black	3.2	D <sub>2</sub>	25	3.3	5.4E+02	2.6E+03	3.1E+03	0.85	288 $\pm$ 8
Pd black	3.6	H <sub>2</sub>	25	3.9	4.5E+02	-6.2E+02	-6.5E+02	0.78	298 $\pm$ 8

(1)D (H) absorption rates -D/Pd or H/Pd- for Pd black were about twice of those for Pd  $\phi$ 0.1 $\mu$ m for the phase 1 charging.

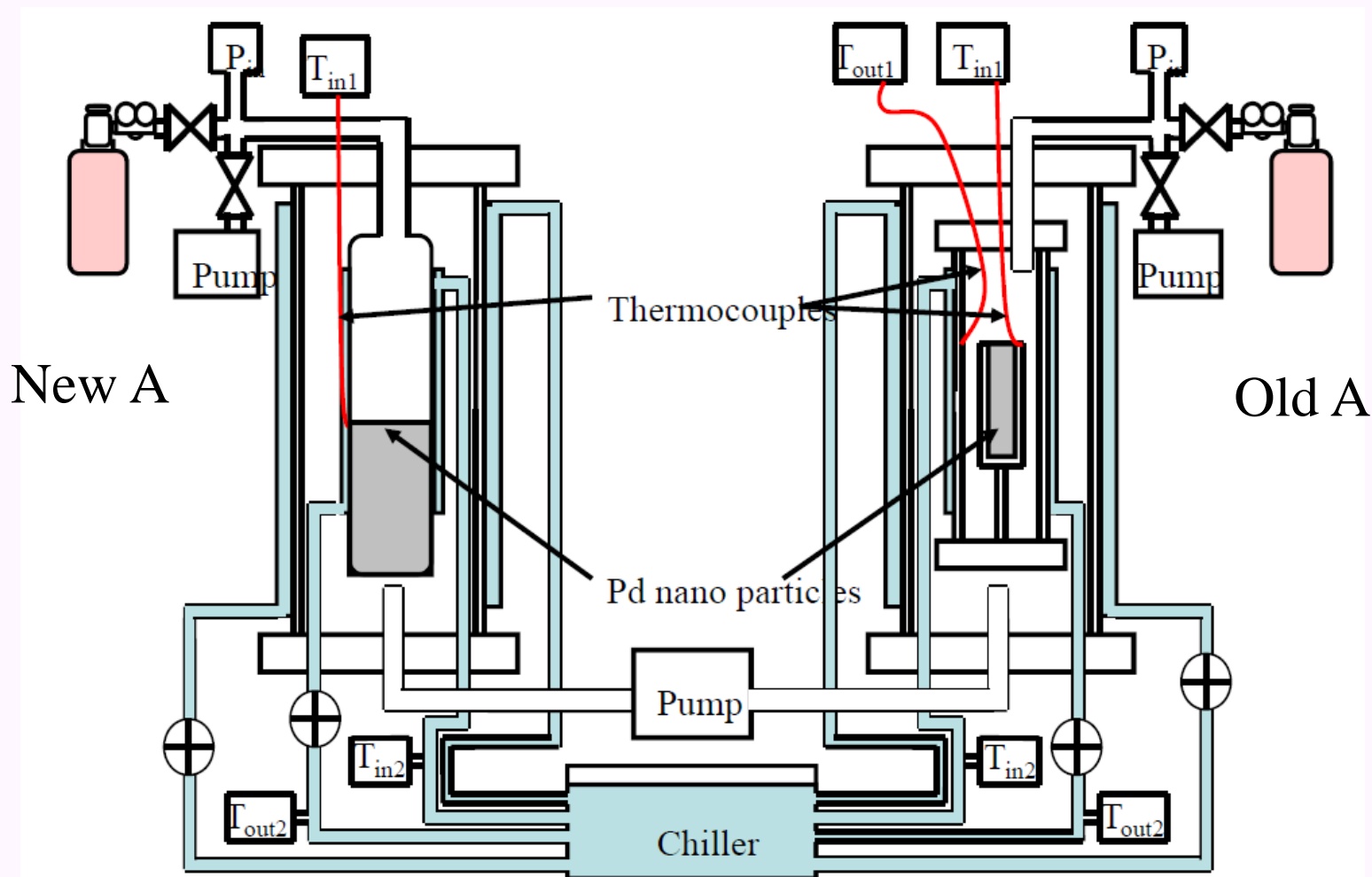
(2)Apparent excess heat about 2.6 kJ/g was observed for Pd black with D<sub>2</sub> gas charging.

## Summary Remarks for the 1<sup>st</sup> Series Experiments

- The Pd- $\text{H}_2$  /  $\text{D}_2$  gas absorption system using commercial Pd powder has been installed which enables a flow calorimetry and nuclear diagnosis.
- The calibration examination and the first experimental results have been described.
- When  $\text{D}_2$  gas was used for the Pd-black sample, trend of excess heat was observed.
- Calorimetry time constant is yet to be made small by making heat capacity of the reaction chamber small.
- Clearer results of heat evolution will be obtained also by increasing the amount of samples.
- Nano-sized Pd powders will also be examined.

# The 2<sup>nd</sup> Series Experiments

**New A:** Shorter FC Time-Constant (5min) and Larger Cell Volume



## New A: Stability of Flow Calorimeter with Zero Input Power

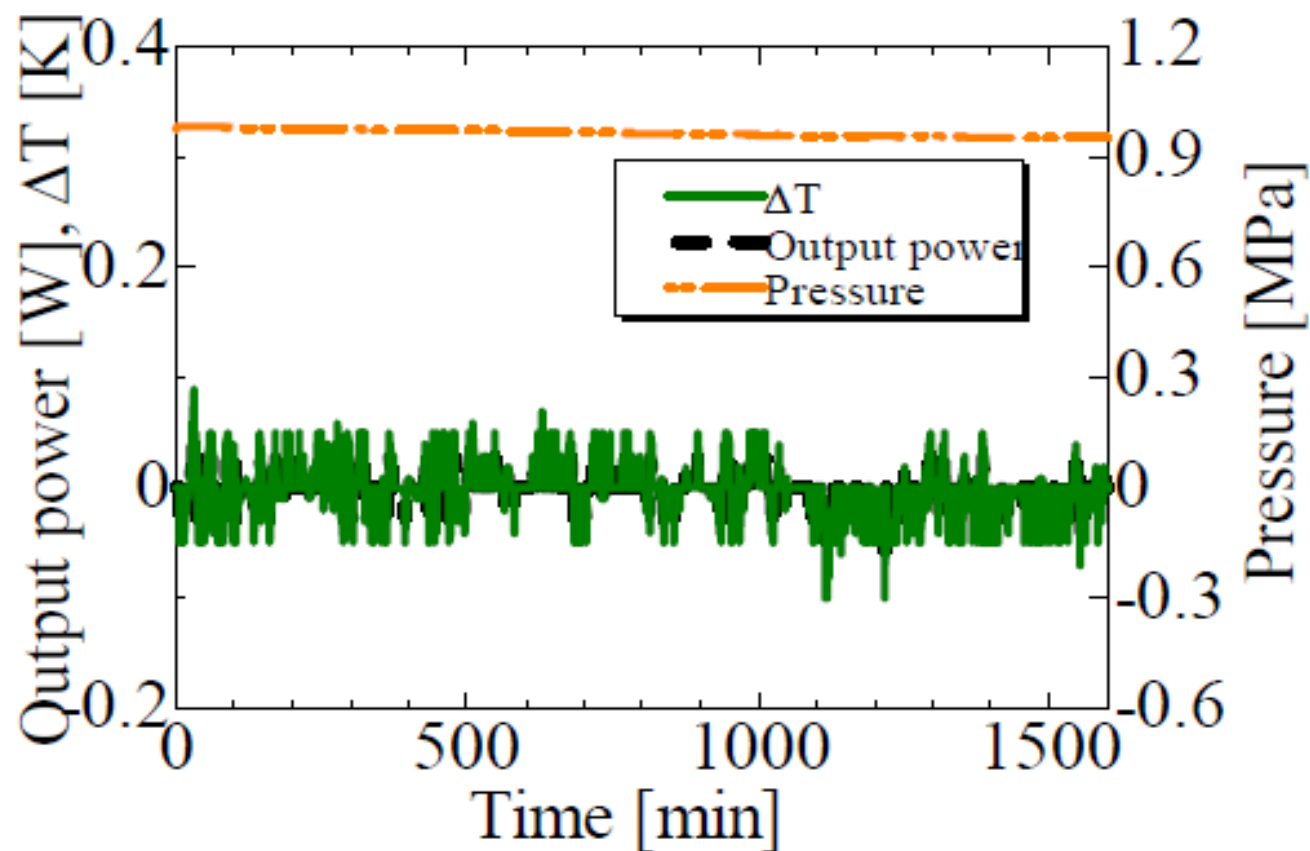
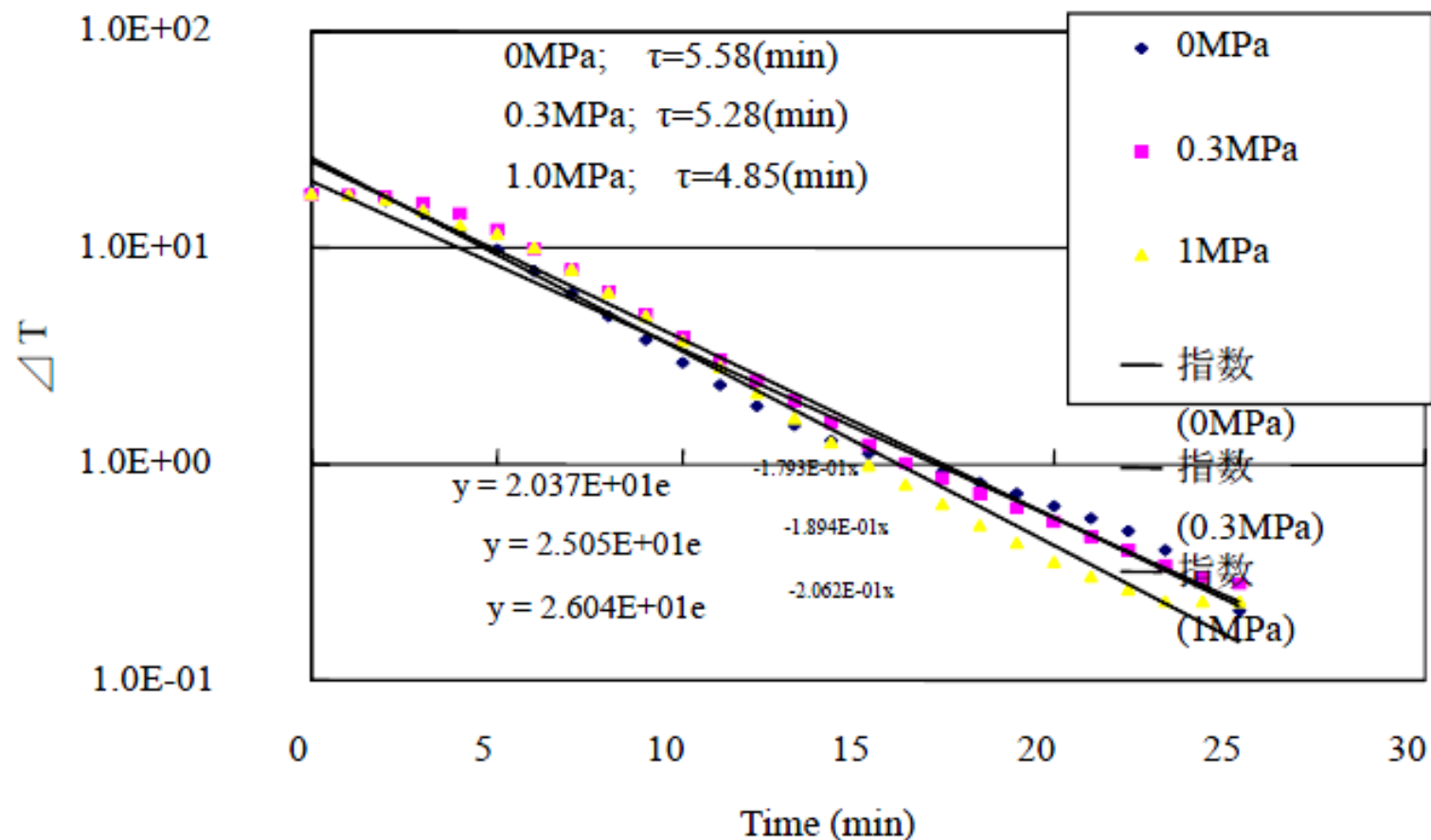


Fig. 4-2. Evolution of temperature;  $D_2$  gas contained with 1MPa in the reaction chamber.

# Time Constant for Heat Removal (75.5 %)

近似式より熱時定数



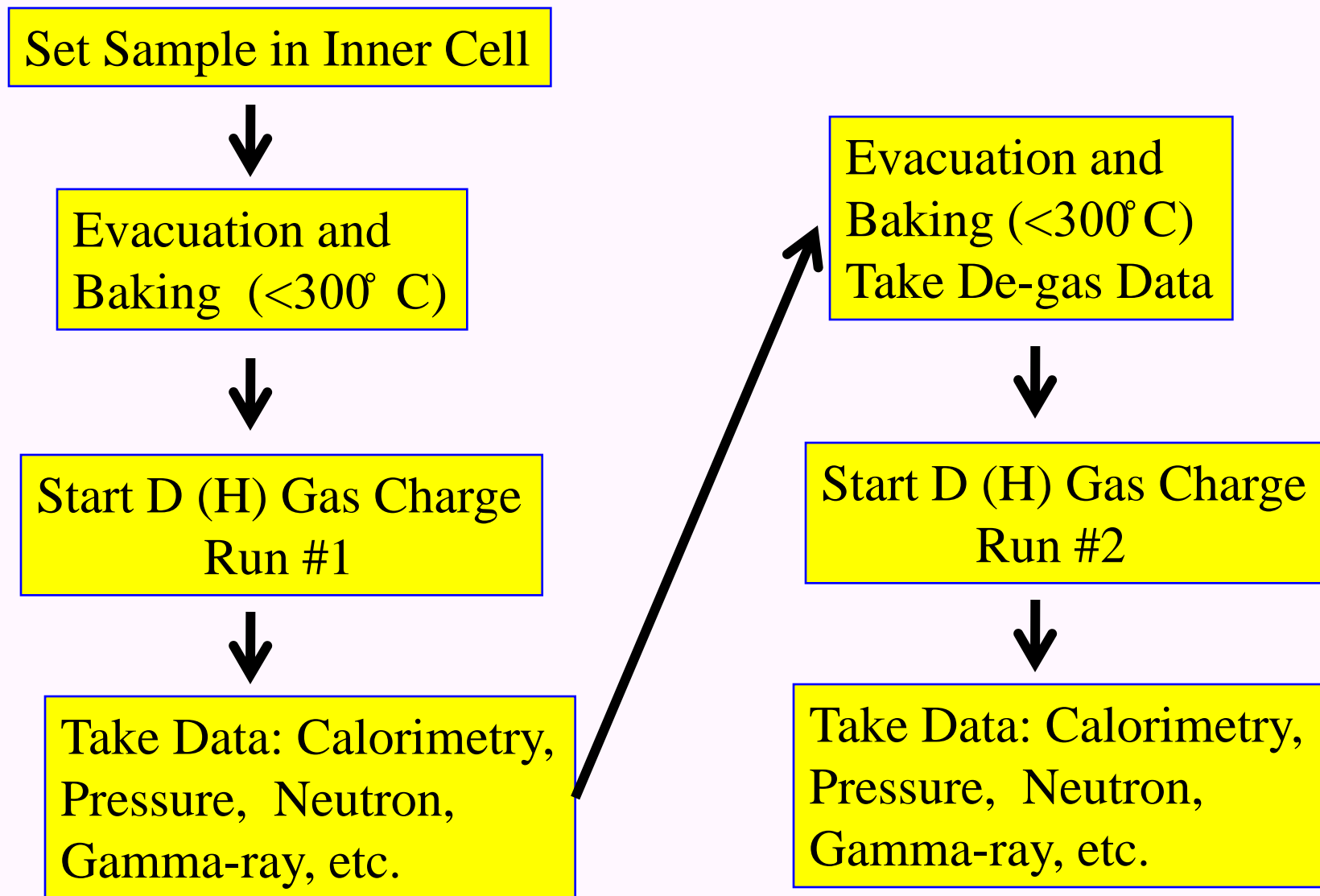
Old A-System

MPa	Thermal time constant $\tau_1$ [s]	Thermal time constant $\tau_2$ [s]
0.0	1463	2731
0.1	686	3016
0.3	746	2566
1.1	927	2048

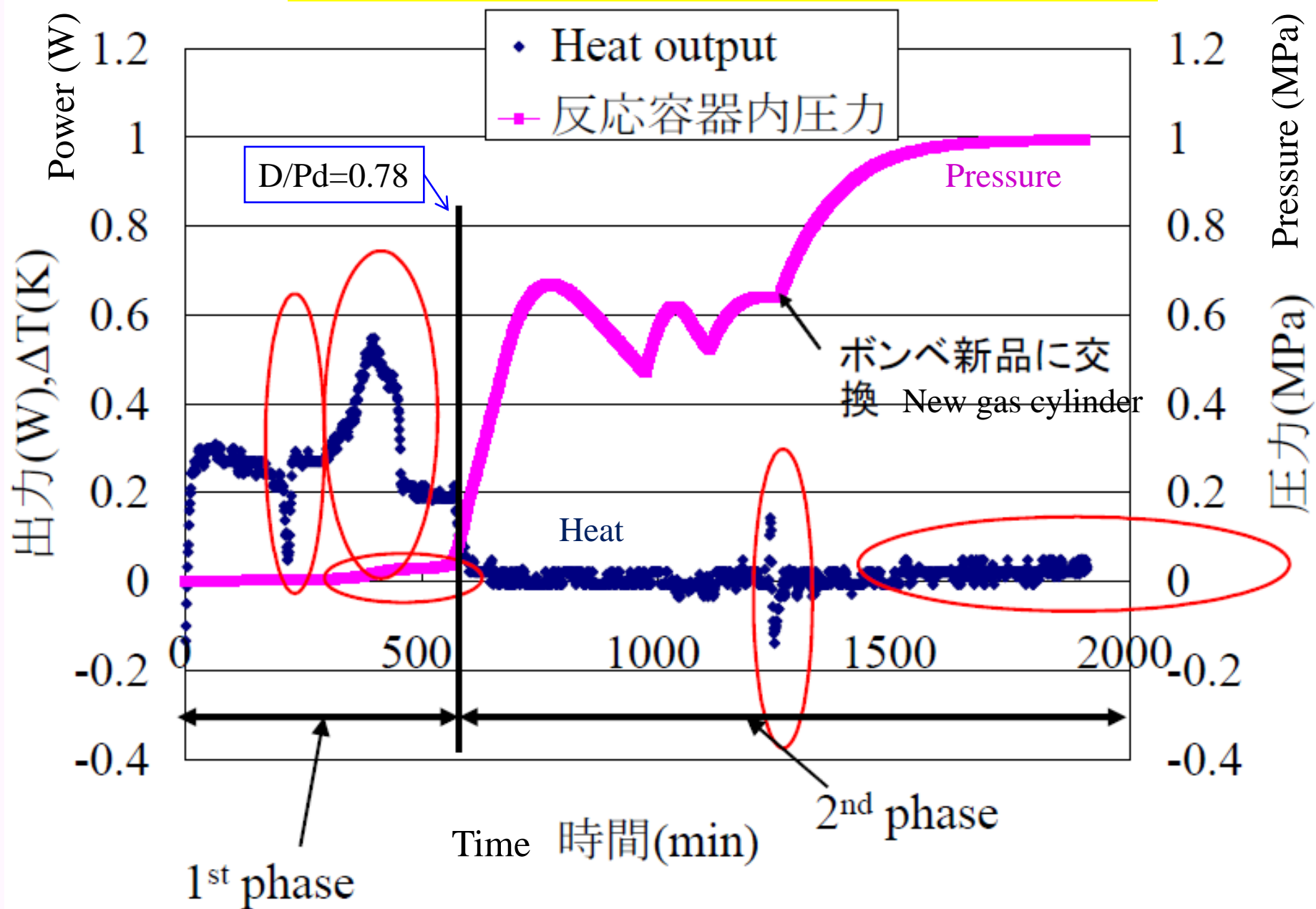
New A-System

MPa	Thermal time constant $\tau$ [s]
0.0	335
0.3	317
1.0	291

## 2. Experimental Procedure

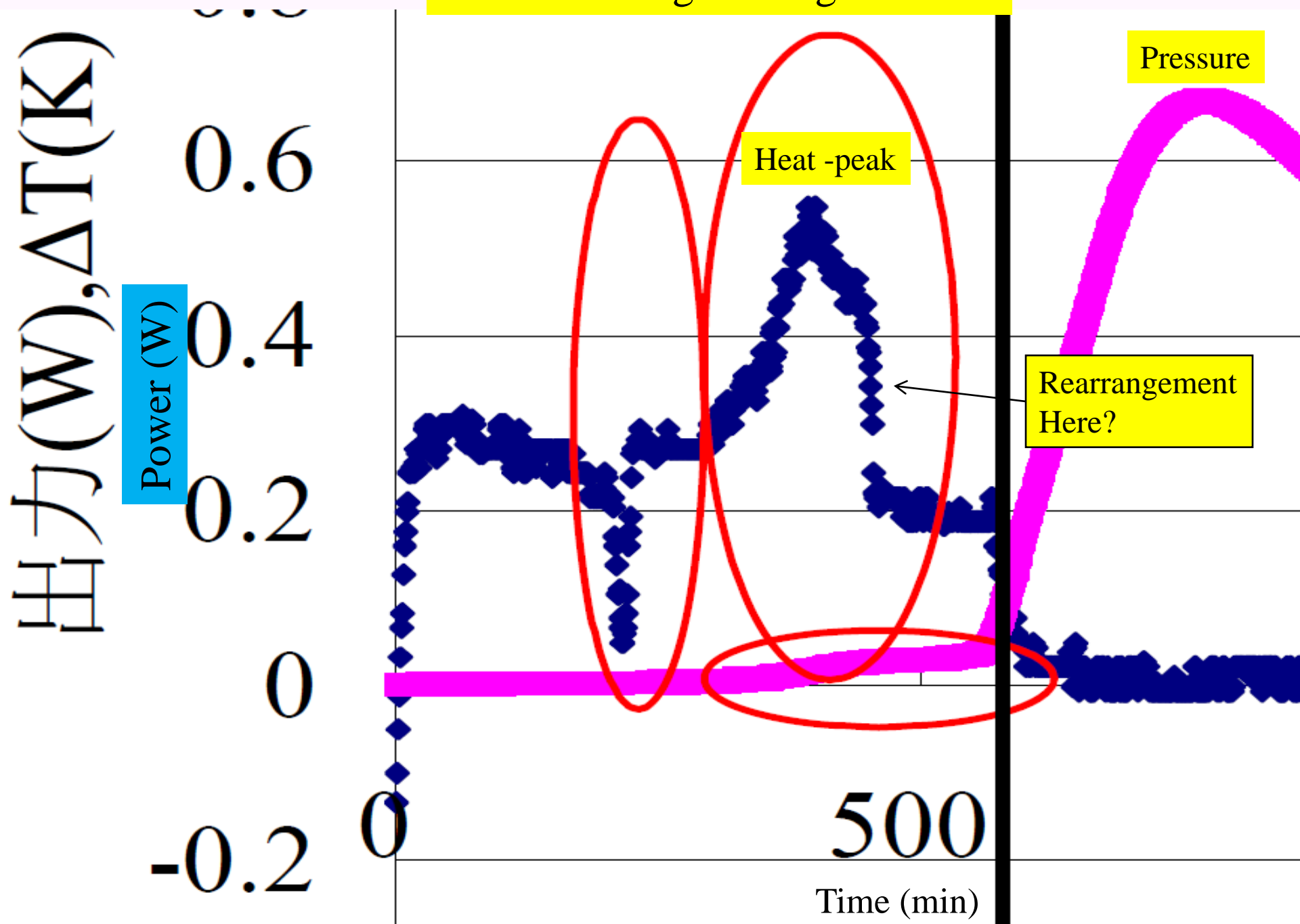


## Pd-black (20g) / D-gas charge #1: Nov. 2009



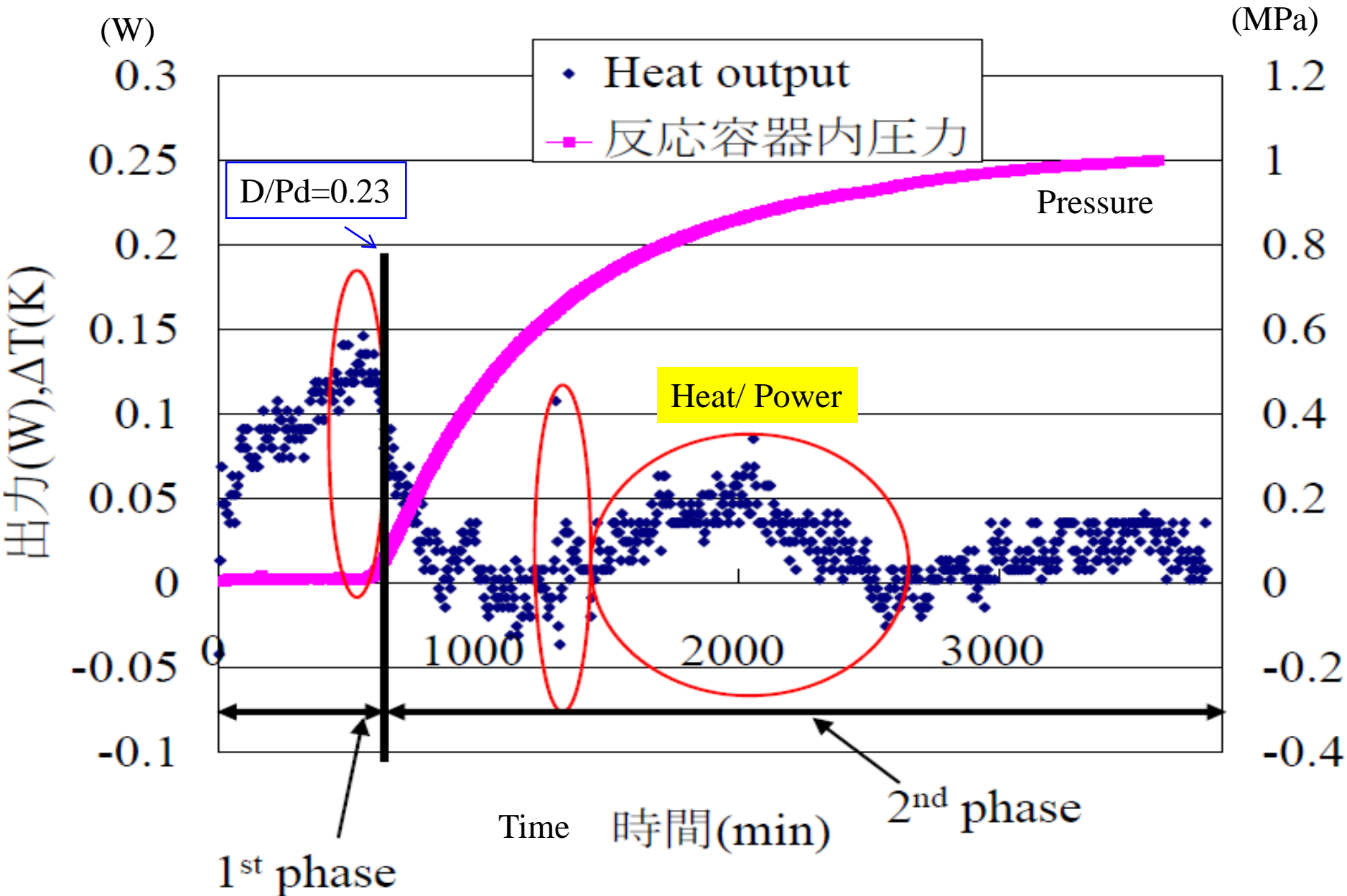


## Pd-black / D-gas charge #1 data

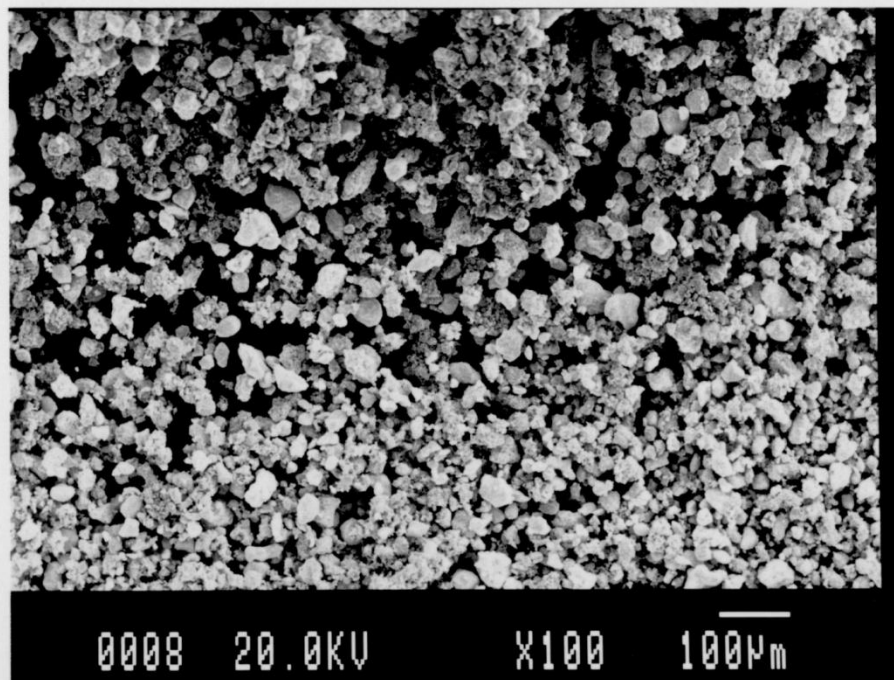


Decreased absorption and heat level for #2

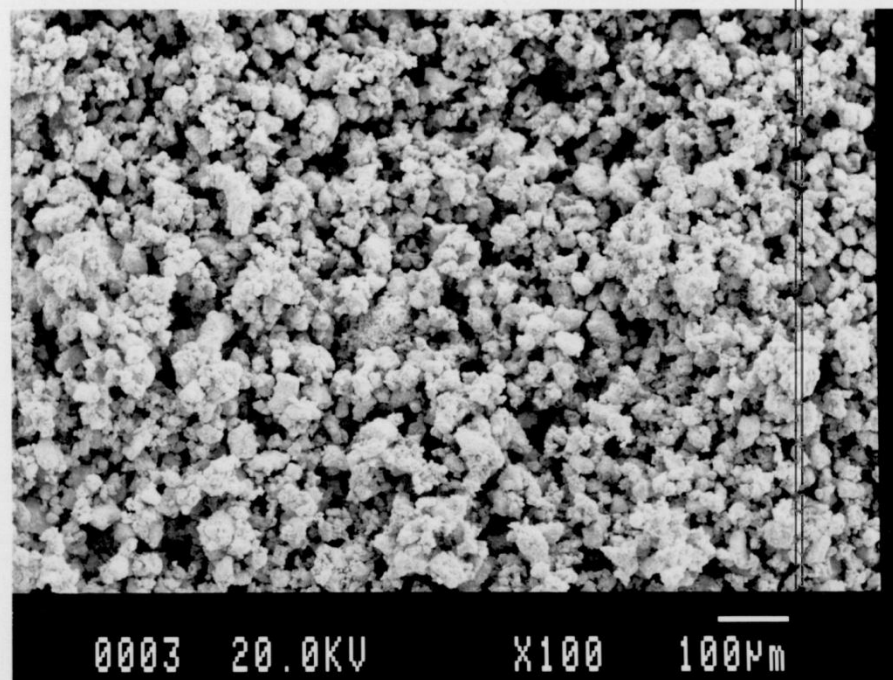
Pd-black (20g)/ D-gas #2



Before Runs



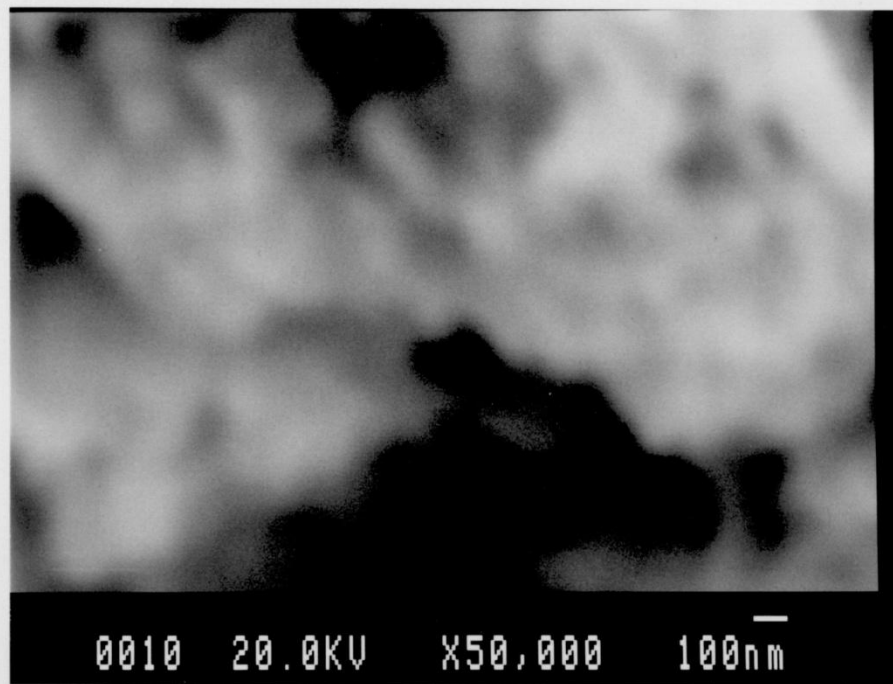
After Runs



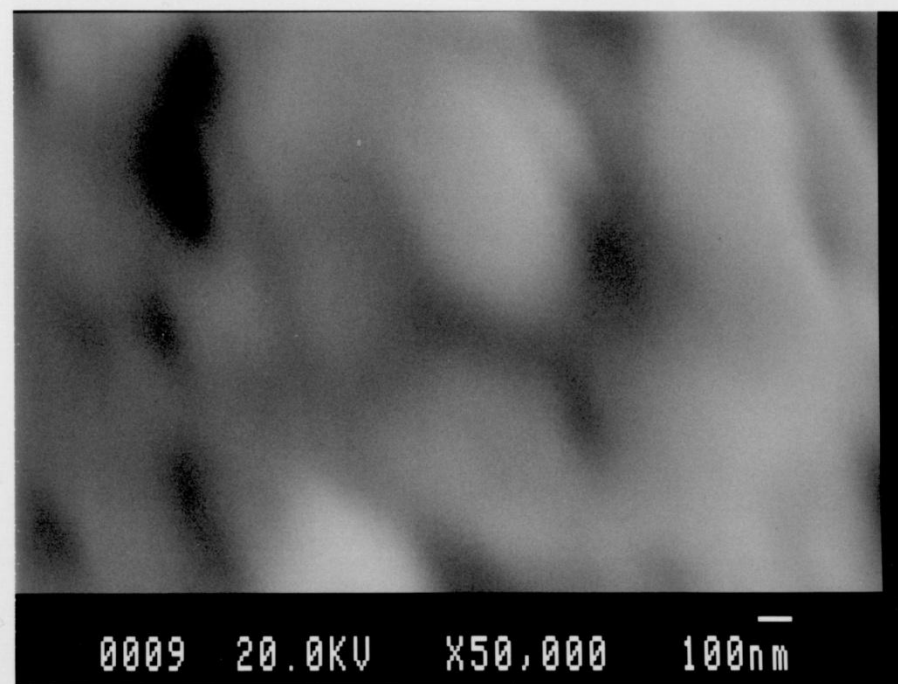
Magnification:  $\times 100$  SEM Image

After experiments, Pd black powders **stuck** to be bigger sizes  $\rightarrow$  Decrease of active surface area

Before Runs



After Runs



Magnification:  $\times 50,000$  SEM Image

Before experiment, surface was fractal in nano-scale.

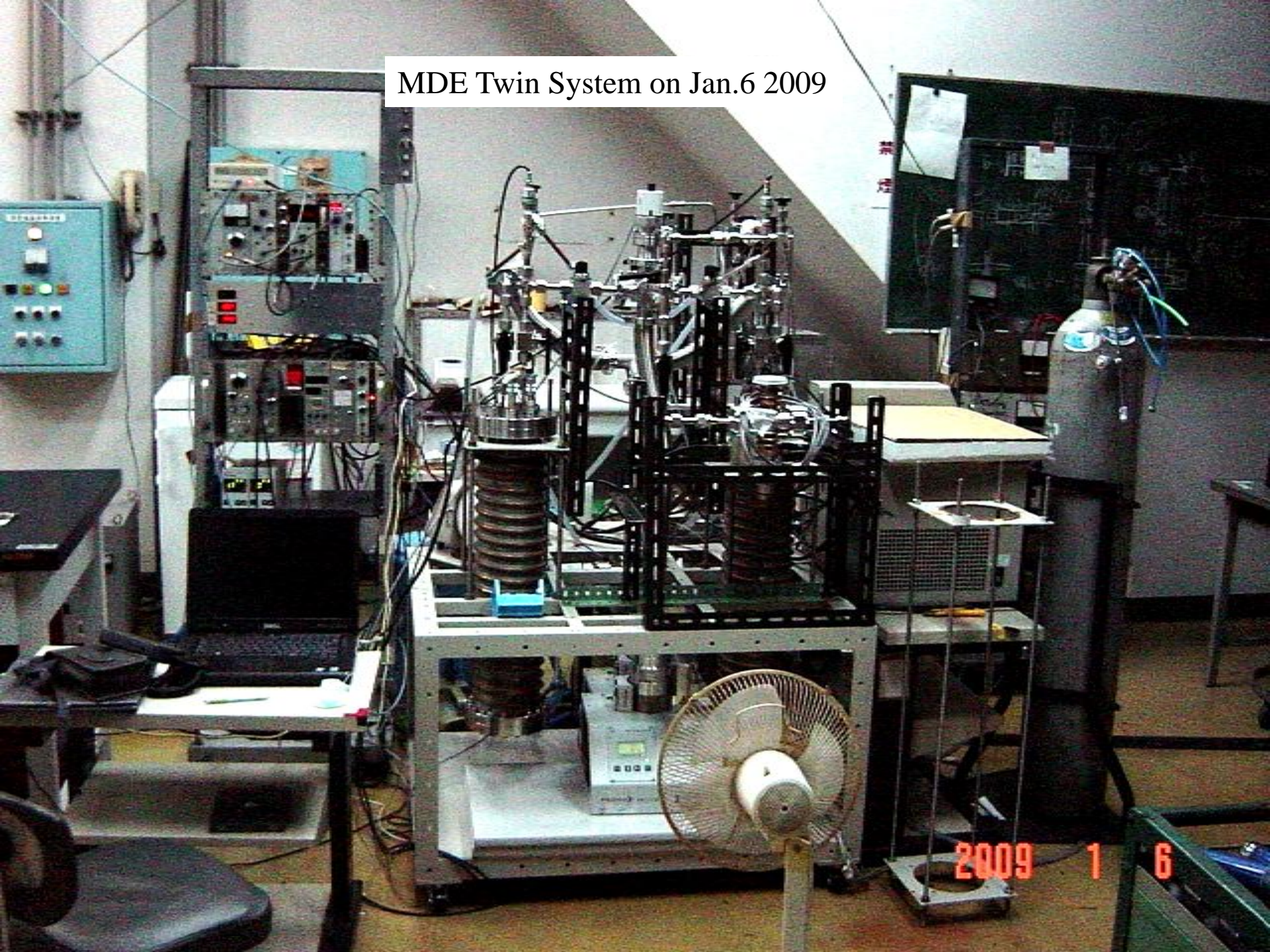
After experiment, surface became flatter in about 10 times larger scale  $\rightarrow$  Decrease of active surface area

# Summary for Pd-black

- There were seen excess heat in the 1<sup>st</sup> and 2<sup>nd</sup> phases, but smaller than those by Pd/ZrO<sub>2</sub>
- Pd-black powders **stuck to be bigger sizes** (about 10 times diameter by SEM observation), after the #1 run.
- In the #2 run and following runs, no significant excess heat was seen.
- D/Pd ratio for #1 was 0.78 in the pressure zero condition under D-charge.
- D/Pd ratios for #2 and later runs, D/Pd was about 0.23 (much smaller).
- No transmuted elements were seen by PIXE.



MDE Twin System on Jan.6 2009



2009 1 6



Pd-membrane A2

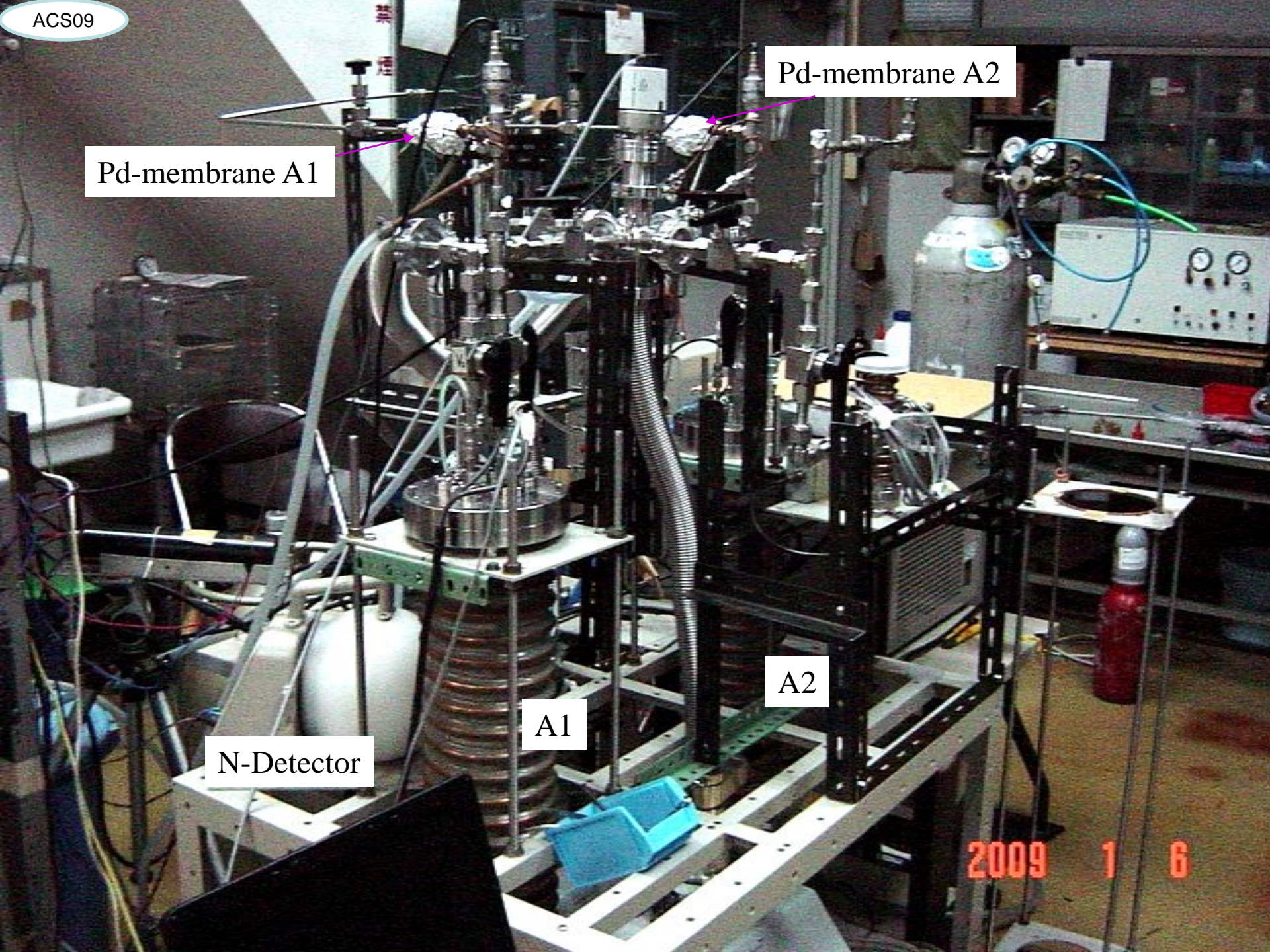
Pd-membrane A1

N-Detector

A1

A2

2009 1 6





Cold Trap

Const. Temp. Water Bath  
Chiller Unit

Constant Flow Pump

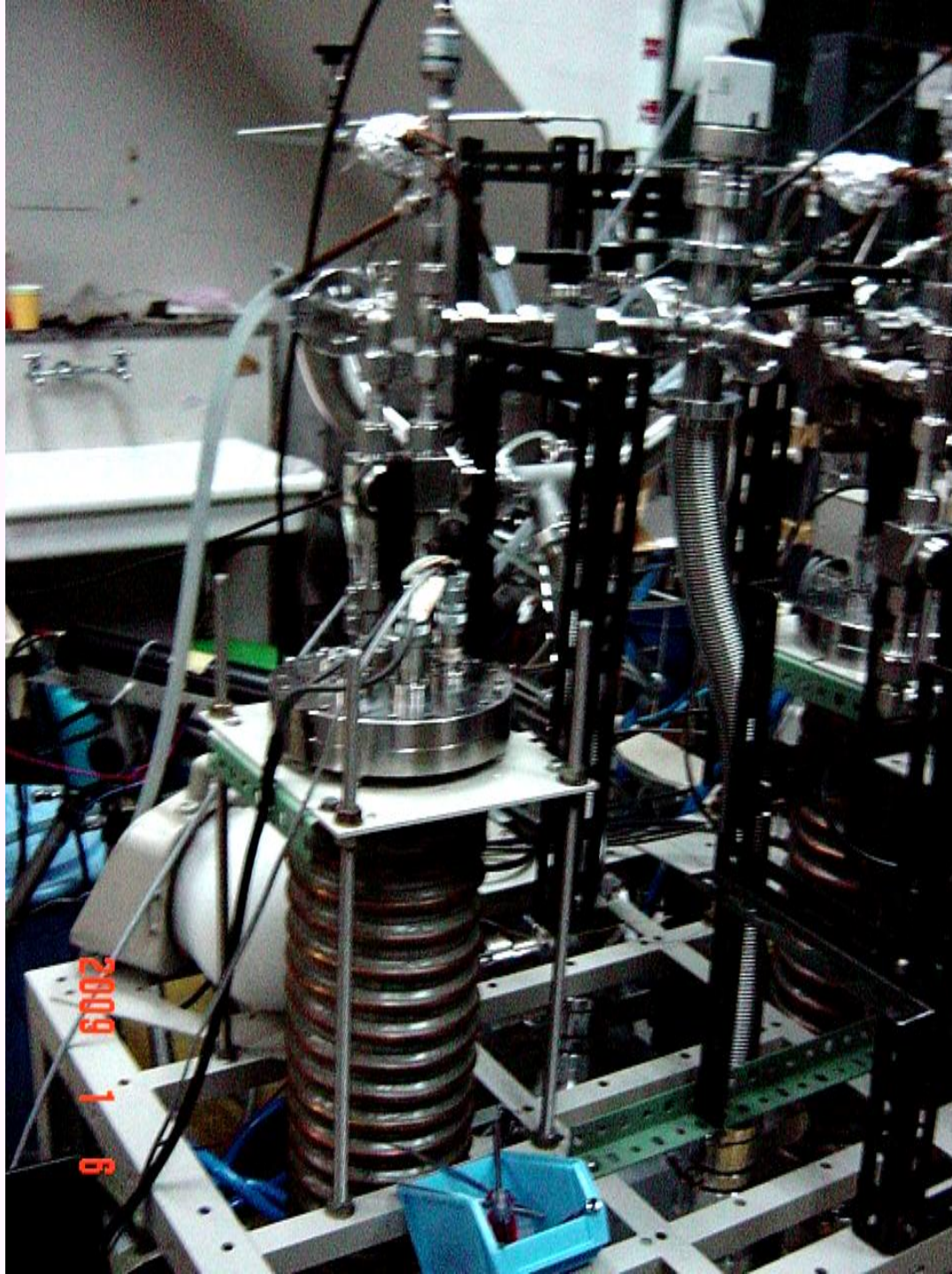
D<sub>2</sub> gas

H<sub>2</sub> gas

2009 1 6



## A1 System





B System  
Under  
Construction



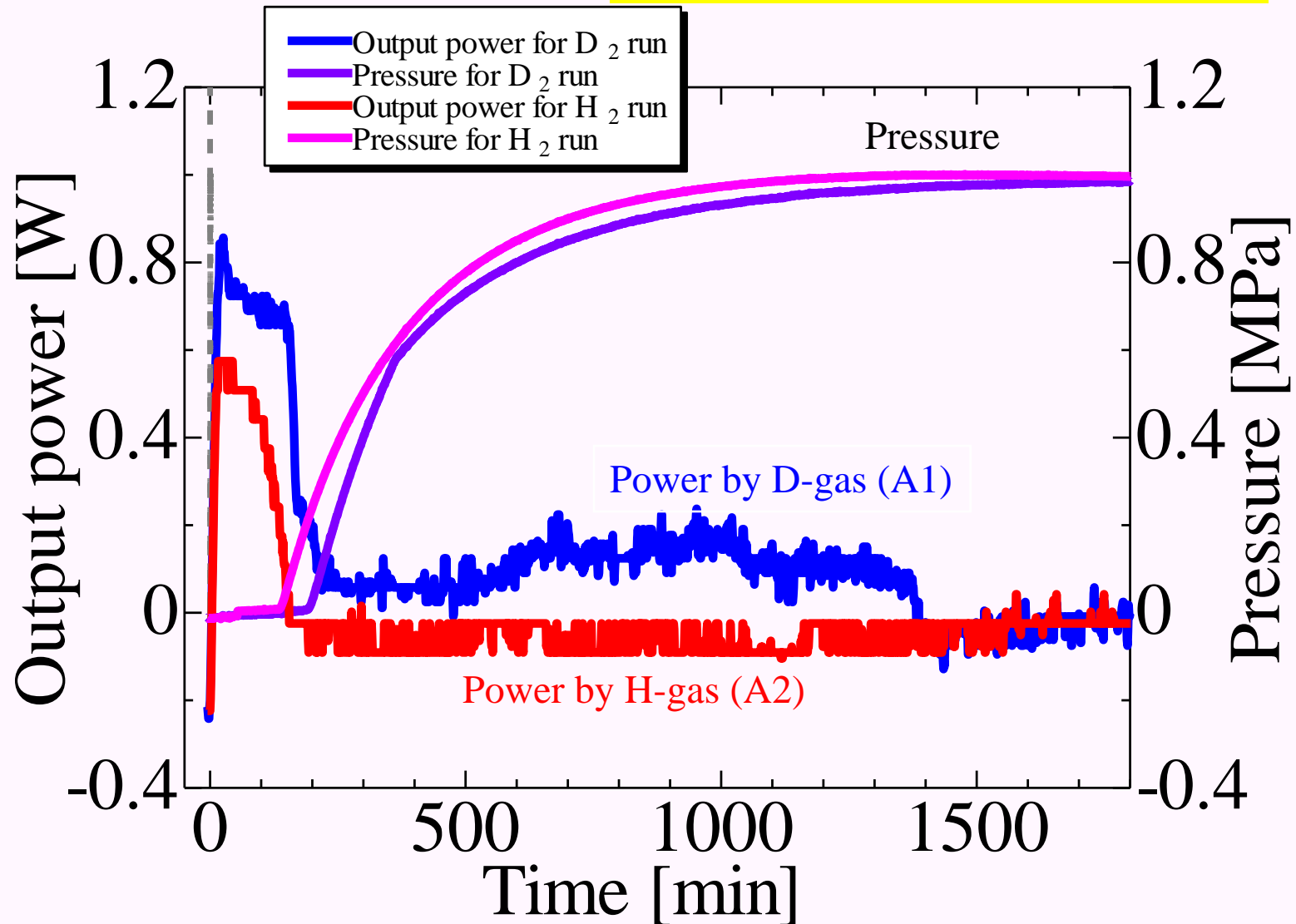
## Experiments with Pd/PdO/ZrO<sub>2</sub> Dispersed Samples

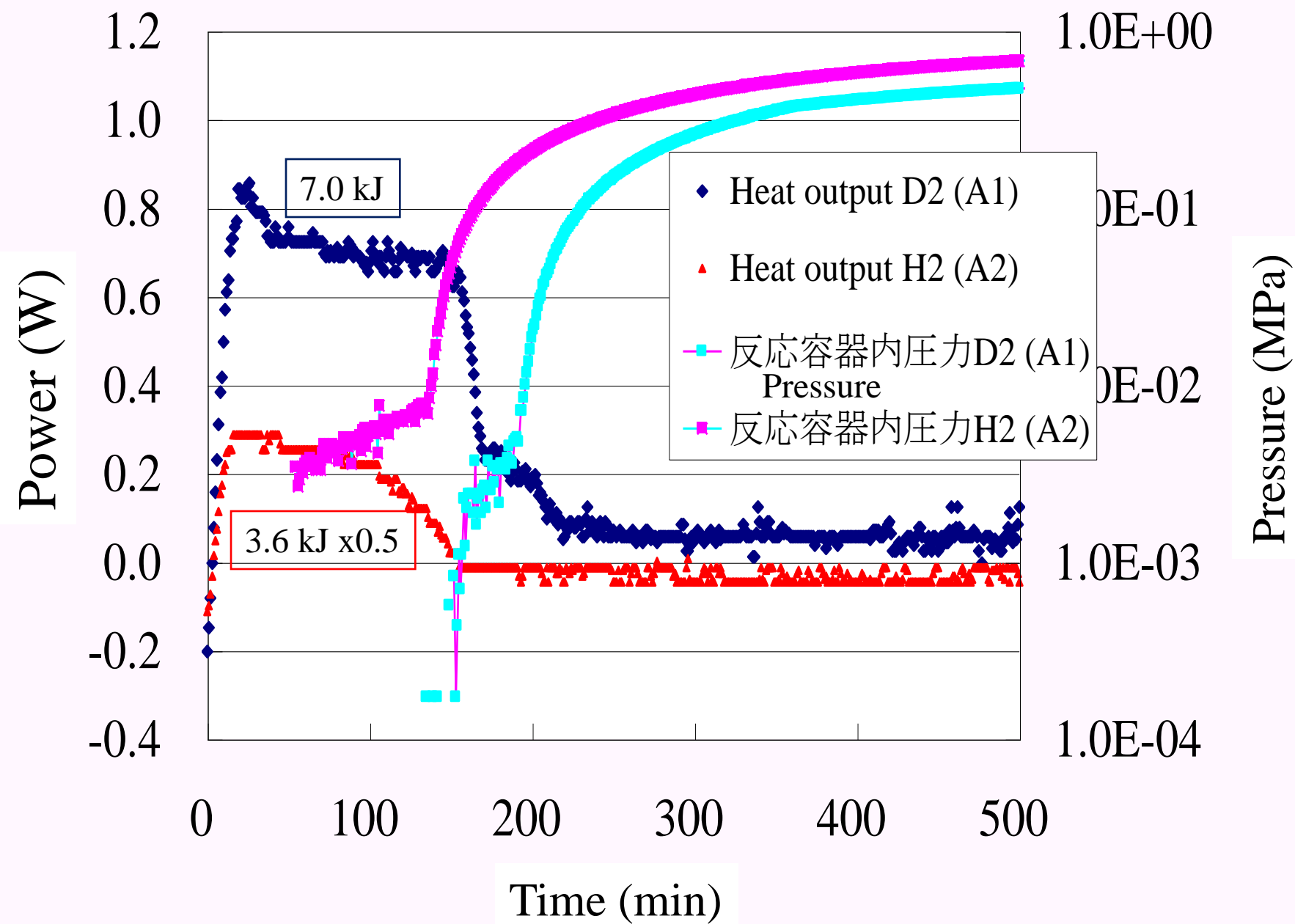
10g (Net Pd weight : 4.3 g): Three trials for Santoku 1, Santoku 2 and Santoku 3 samples, #1 and #2 runs for each sample

- Nano-Pd/ZrO<sub>2</sub> sample was produced by Santoku Co. Japan, based on different protocol from Inoue-Yamaura (Arata-Zhang).
- X-ray diffraction analysis showed composite of Pd/PdO/ZrO<sub>2</sub>.
- Pd particle size is less than 10nm.
- A1 system: 10g for D-gas charging (2-3sccm)
- A2 system: 10g for H-gas charging (2-4sccm)
- Water-flow calorimetry: 6cc/min flow rate

# Santoku 1#1 Results of Heat Evolution

Gas flow rate: 2.8 (D) and 2.6(H) sccm

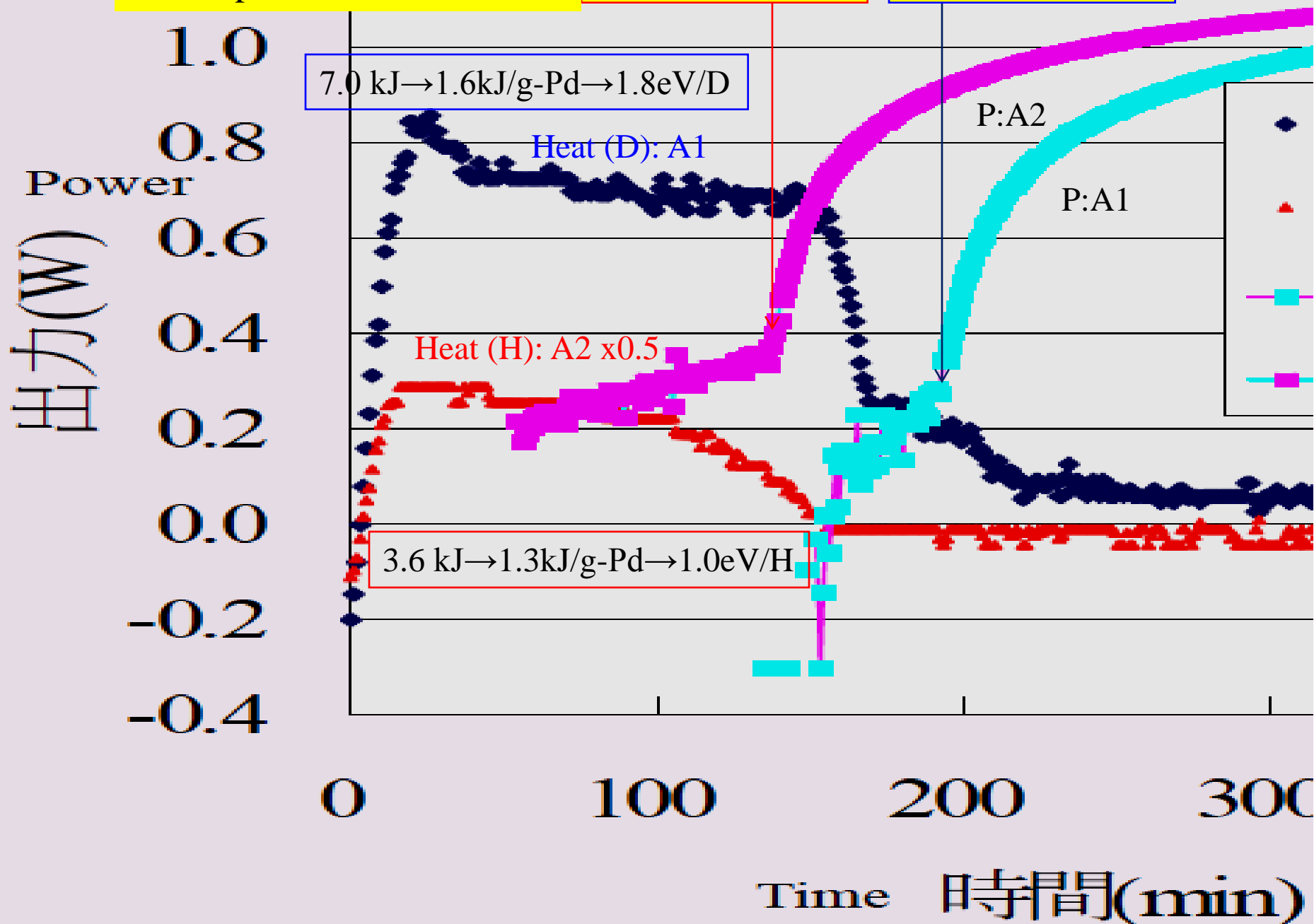


Santoku1 #1 Run: Expanded view for 1<sup>st</sup> phase

Santoku 1#1 Expanded View  
For 1<sup>st</sup> phase

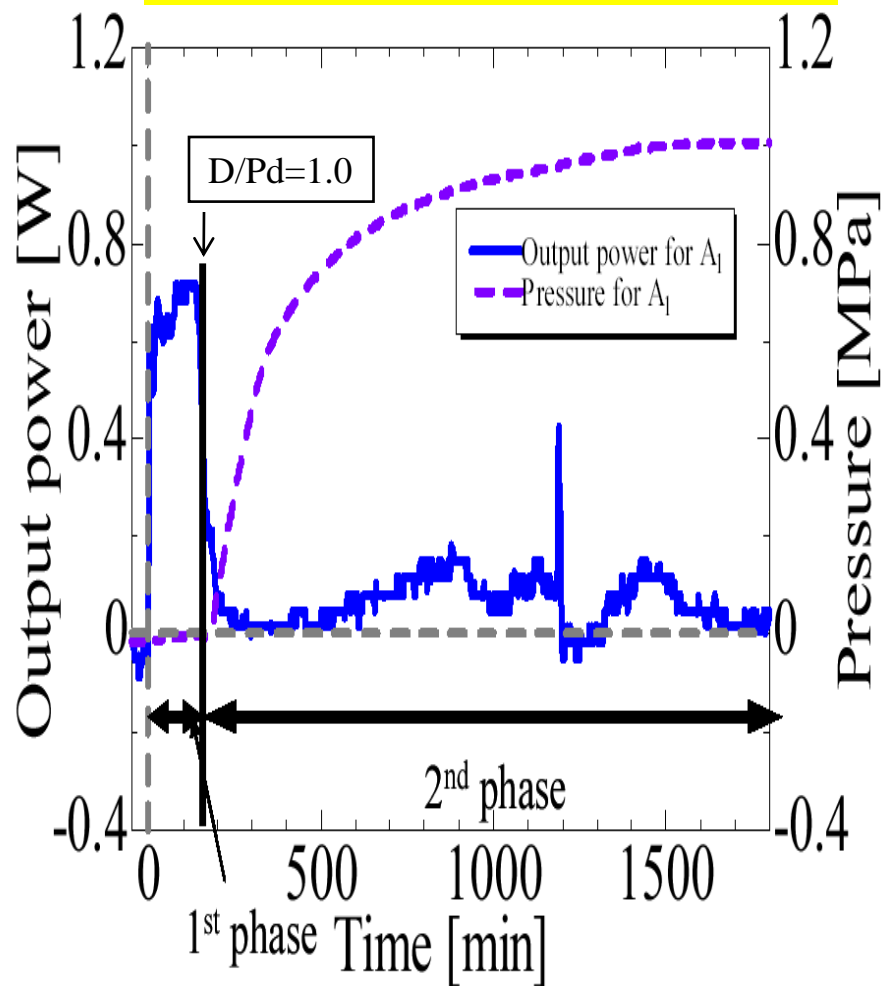
A2 first phase end:  
H/Pd=0.9

A1 first phase end:  
D/Pd=1.0



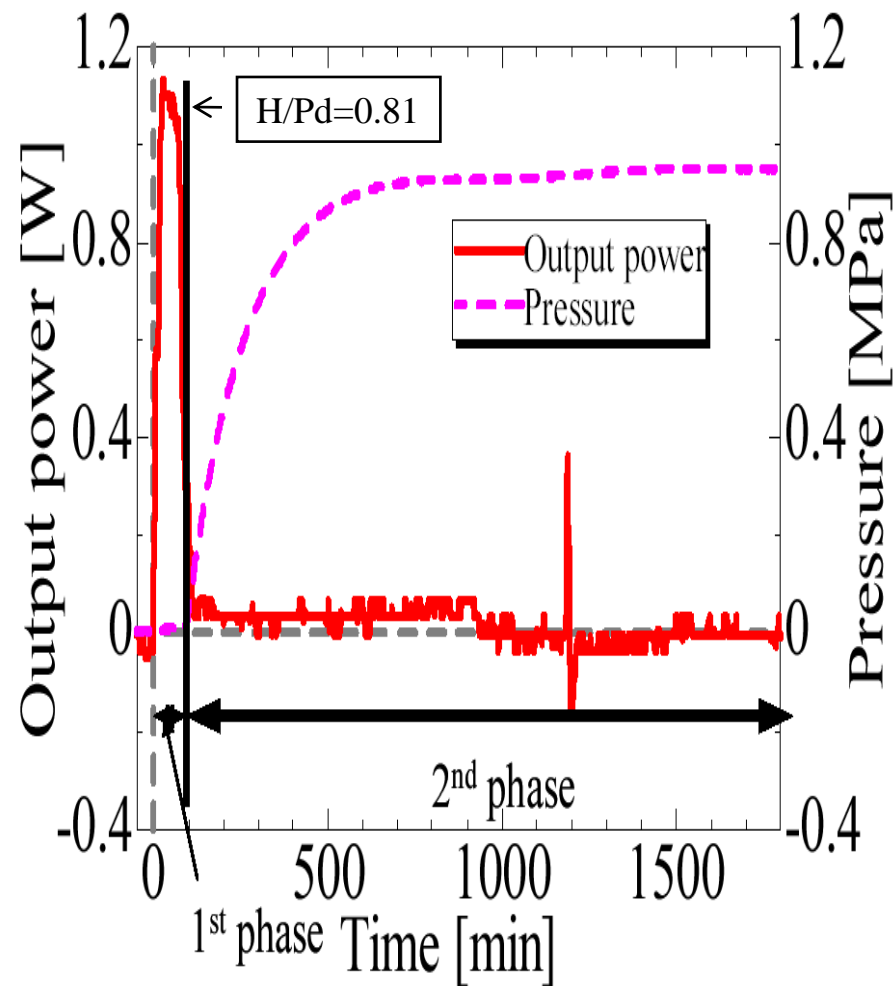
# Results of Santoku2#1 run

A1: D-gas Charge with 2.9sccm



D-gas Charging to Santoku2#1

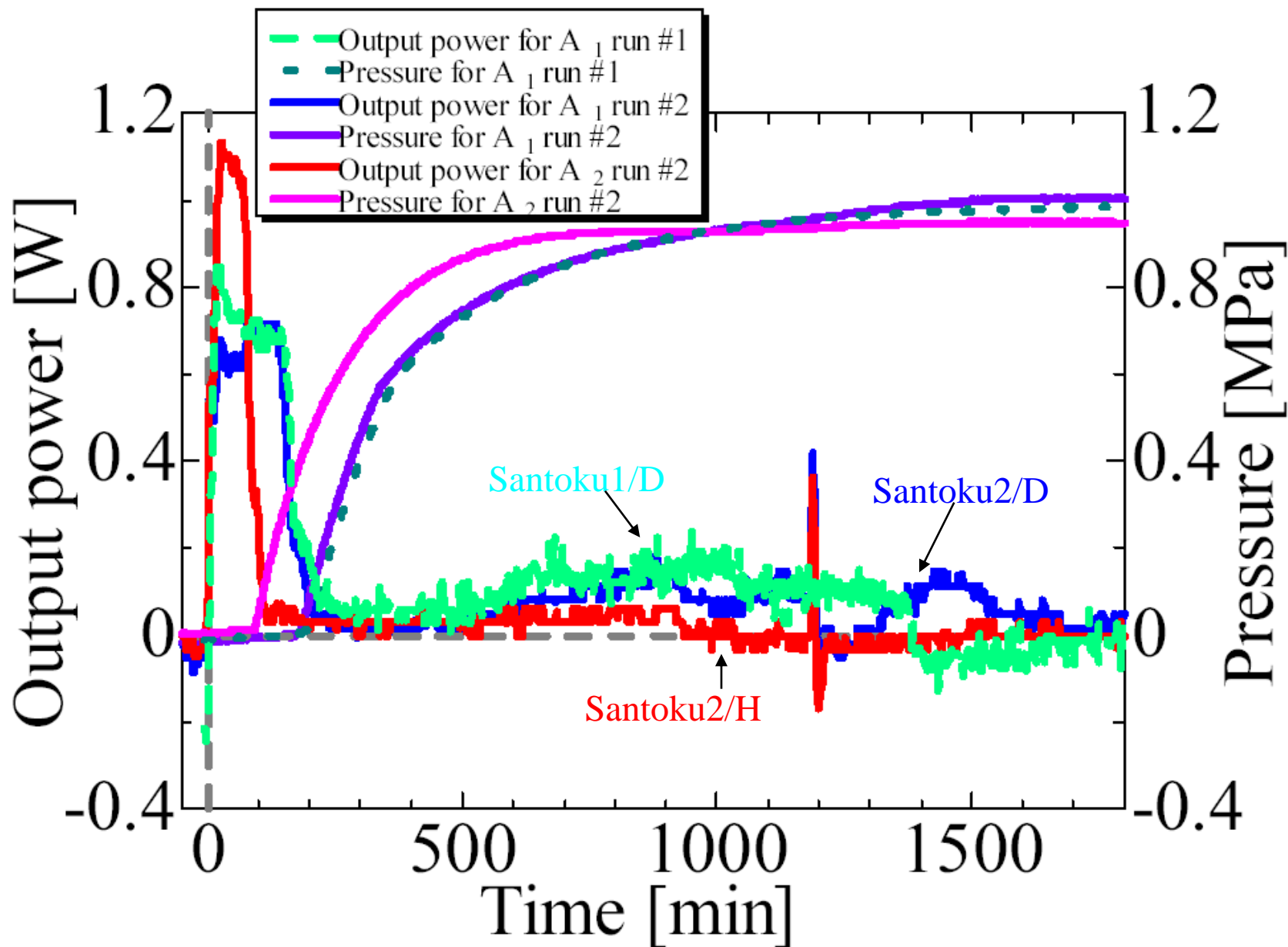
A2: H-gas Charge with 3.5sccm



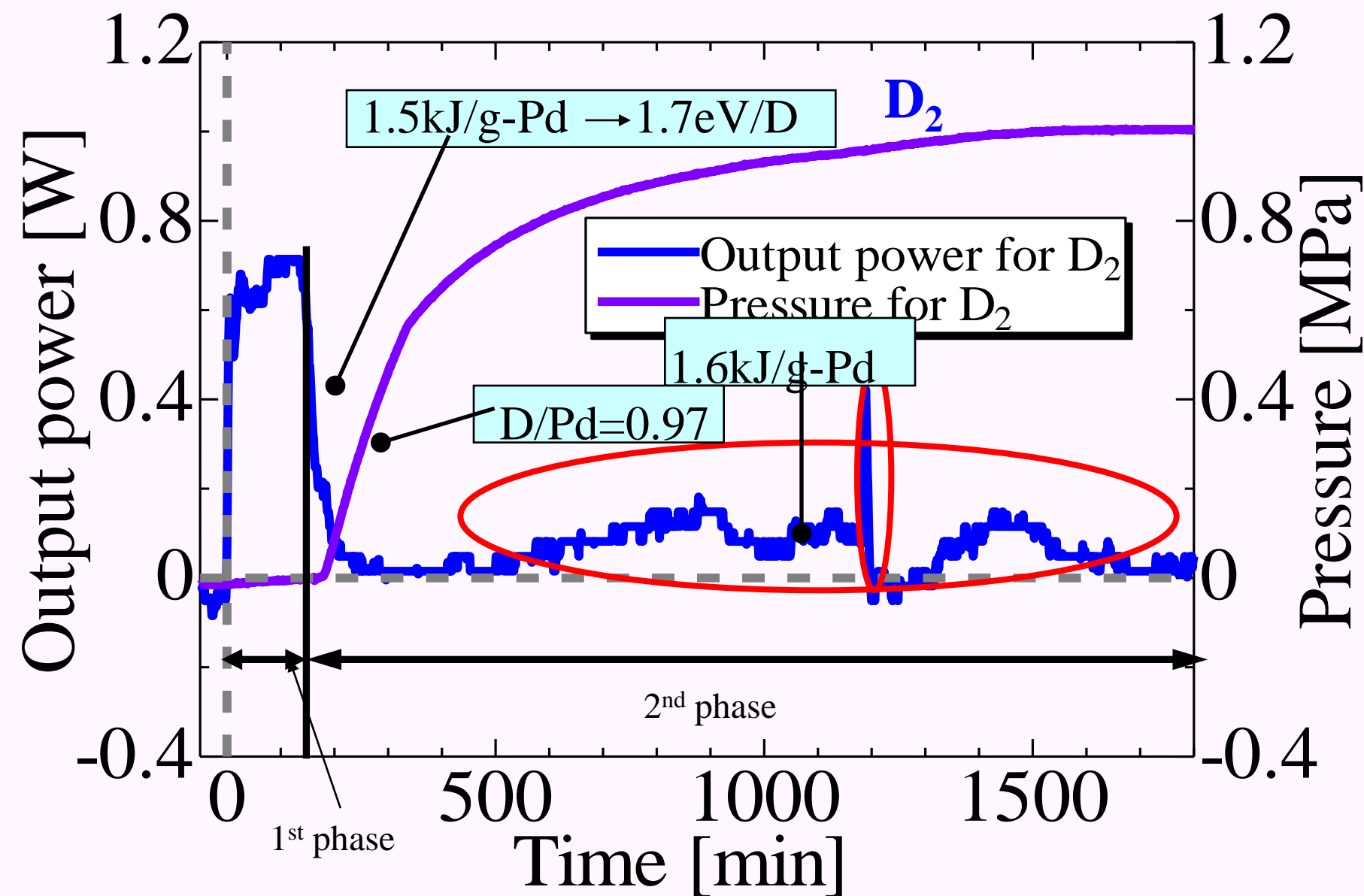
H-gas Charging to Santoku2#1

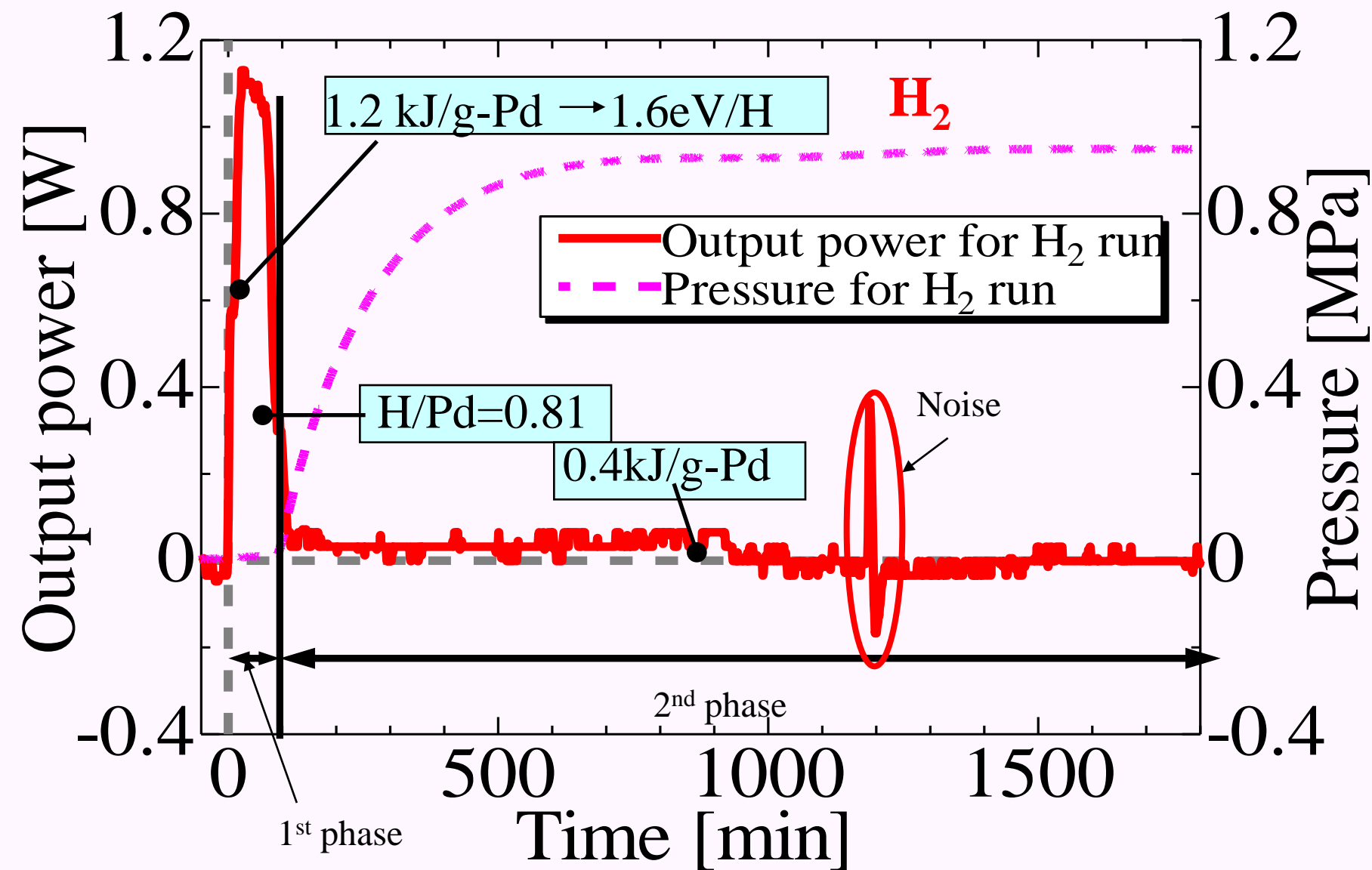


## Comparison of Results for Santoku1#1 and Santoku2#1





**Pd·ZrO<sub>2</sub> Santoku 2 #1 Run (A1) for D-gas Charge**

**Pd·ZrO<sub>2</sub> Santoku 2 #1 Run (A2) for H<sub>2</sub>-gas Charge**

# Integrated Data for Pd/ZrO<sub>2</sub> Santoku #1 Runs

Significant **isotopic effects** are seen for D(H)/Pd =x loading ratios, and out-put energies per Pd (and D(H) atom), for the 1<sup>st</sup> phase. Significant **excess heat** by D-gas charge in the 2<sup>nd</sup> phase.

## Santoku 2#1

Run number	Specific output energy[kJ/g]		Composition; x for	Output energy per D/H
	1st phase	2nd phase		
D <sub>2</sub>	1.5±0.0	1.6±0.2	0.97	1.71± 0.05
H <sub>2</sub>	1.2±0.0	0.4±0.2	0.81	1.67 ± 0.03

## Santoku 1#1

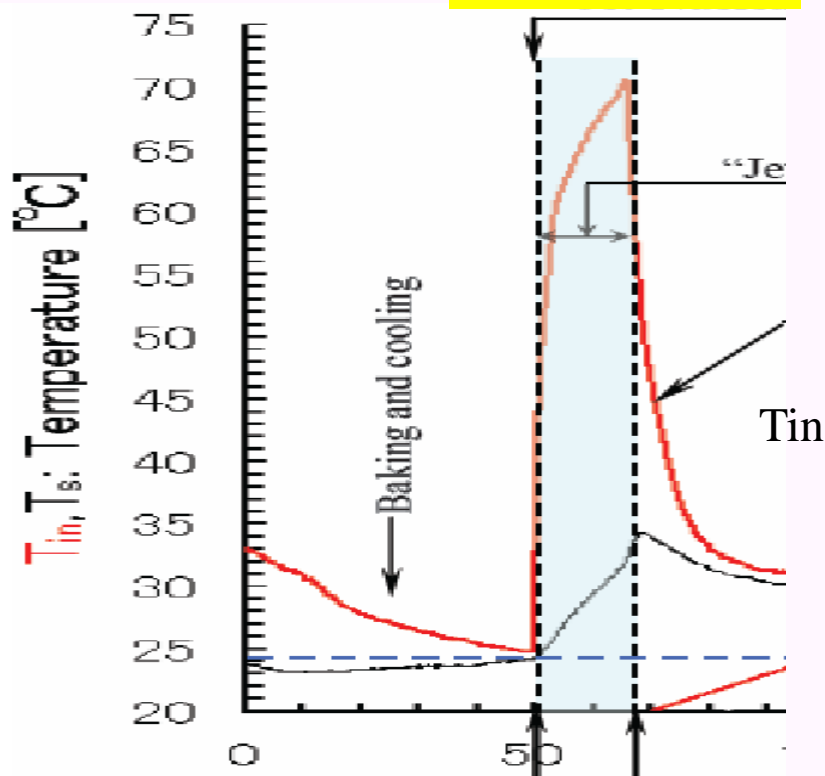
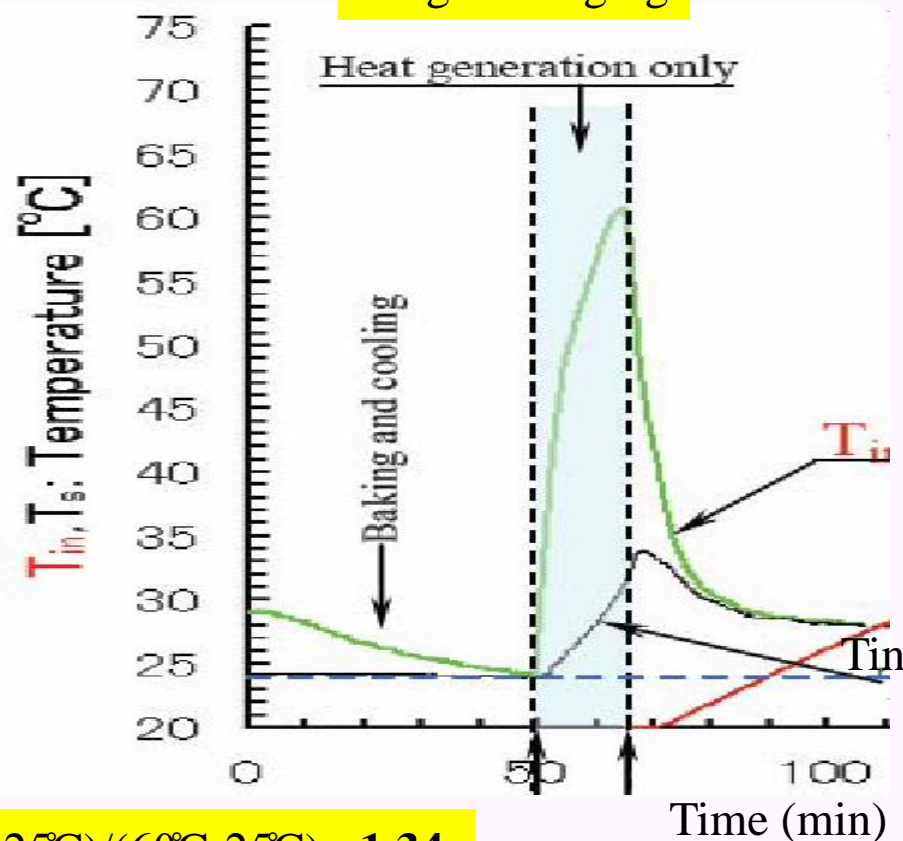
D <sub>2</sub>	1.6±0.0	1.7±0.2	1.0	1.8±0.05
H <sub>2</sub>	0.8±0.0	(-)	0.89	1.0±0.03

# Summary Table of Integrated Data for #1 Runs With Santoku Pd/PdO/Zr<sub>2</sub>O nano-composite samples

Time-Interval: 0-1,800 min

Run number	flow rate [sccm]	Output energy [kJ]		Specific output energy[kJ/g]		Composition; x for PdD <sub>x</sub> /PdH <sub>x</sub>	Output energy per D/H in the 1st phase [eV]
		1st phase	2nd phase	1st phase	2nd phase		
D-PZ1#1	2.8	7.0±0.2	6.8±1.3	1.6±0.0	1.6±0.3	1.0	1.80 ± 0.05
Corrected H-PZ1#1	2.6	3.6±0.1	-5.1±1.4	0.8±0.0	-1.2±0.3	0.89	1.02 ± 0.03
D-PZ2#1	2.9	6.4±0.2	5.5±0.8	1.5±0.0	1.2±0.2	0.97	1.71± 0.05
H-PZ2#1	3.5	5.1±0.1	1.7±0.9	1.2±0.0	0.4±0.2	0.81	1.67 ± 0.03

# Arata-Zhang's 1<sup>st</sup> Phase Data for Pd/ZrO<sub>2</sub> (7g)

D<sub>2</sub>-gas chargingH<sub>2</sub>-gas charging

**Arata-Zhang:**  $[\text{Heat(D)}/\text{Heat(H)}] \sim (72\text{C}-25\text{C})/(60\text{C}-25\text{C}) = 1.34$

**Our Exp. Santoku1:**  $[\text{Heat(D)}/\text{Heat(H)}] = (7.0\text{kJ})/(3.6\text{kJ}) = 1.94$

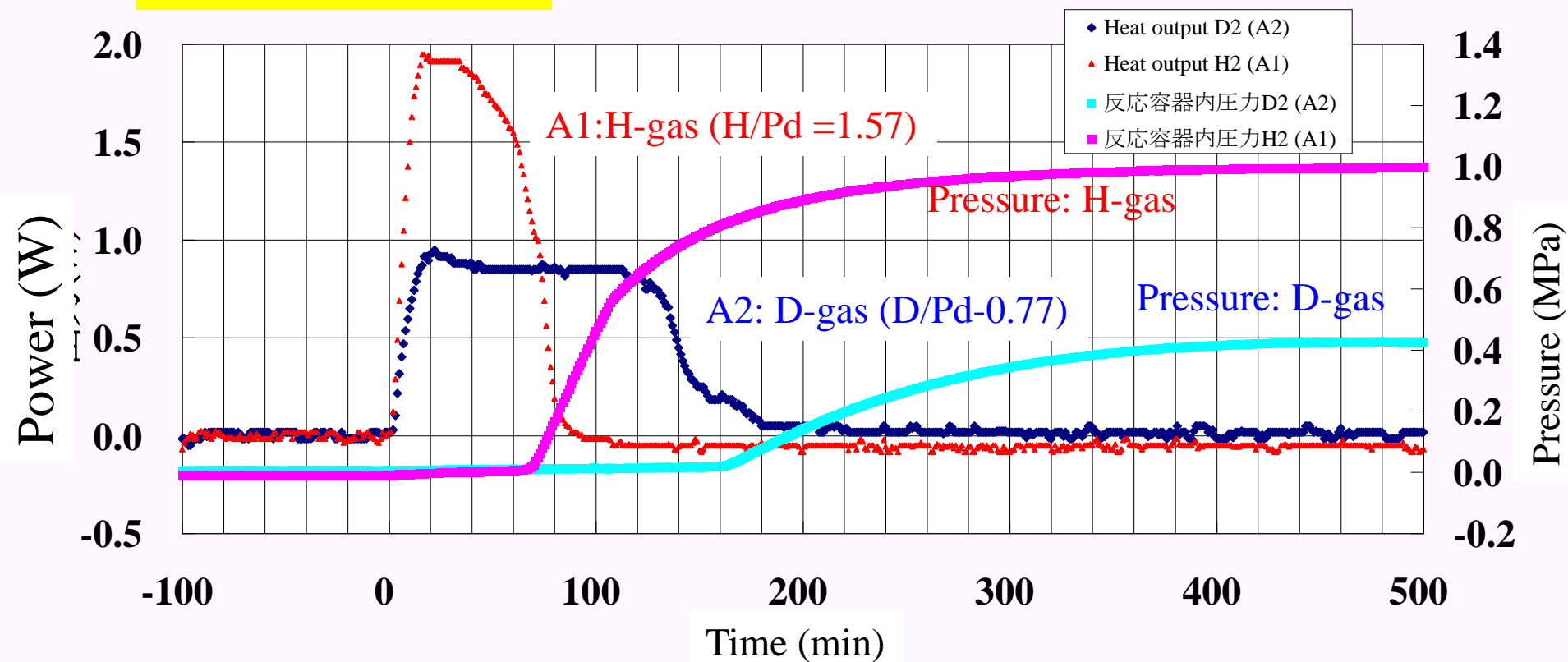
**Our Exp. Santoku2:**  $[\text{Heat(D)}/\text{Heat(H)}] = (6.4\text{kJ})/(5.1\text{kJ}) = 1.26$

**Agreed!**

# Results by Large H-Flow Rate And Low D-Gas Pressure

A1: H-gas, Flow Rate = **9.96** sccm  
A2: D-gas, Flow Rate = **2.1** sccm

## Santoku 3#1 Run



run	1st phase [kJ]	2nd phase [kJ]	1st phase [J/g]	2nd phase [J/g]
D <sub>2</sub> -3-1	7.14±0.15	1.26±1.36	1661±35	293±316
H <sub>2</sub> -3-1	7.07±0.07	-0.23±1.44	1644±18	-54±334

**Table. : Integrated Data for the First Phase (“zero” pressure interval)**

Run	gas	Heat (kJ/g-Pd)	D/Pd or H/Pd	Eout/Pd (eV)	Eout/D(H) (eV)	comment
0.1 $\mu$ Pd#2	D	0.097 $\pm$ 0.05	0.43	0.11	0.25 $\pm$ 0.13	Old A
ibid	H	0.12 $\pm$ 0.03	0.45	0.13	0.29 $\pm$ 0.07	Old A
Pd-black(I)	D	0.54 $\pm$ 0.10	0.85	0.59	0.70 $\pm$ 0.13	Old A
ibid	H	0.45 $\pm$ 0.08	0.78	0.50	0.63 $\pm$ 0.11	Old A
Pd-black(II)#1	D	0.47 $\pm$ 0.06	0.78	0.52	0.66 $\pm$ 0.08	Baking $T \leq 170^{\circ}\text{C}$
Pd-black(II)#2	D	0.17 $\pm$ 0.03	0.23	0.19	0.81 $\pm$ 0.14	stuck
Pd-black(II)#4	D	0.16 $\pm$ 0.02	0.24	0.18	0.75 $\pm$ 0.09	stuck
Pd-black(II)#2	H	0.16 $\pm$ 0.01	0.22	0.18	0.80 $\pm$ 0.05	stuck
Santoku 1 #1 Pd/PdO/ZrO <sub>2</sub>	D	1.63 $\pm$ 0.01	1.0	1.80	<u>1.80</u> $\pm$ 0.01	Baking $T \leq 3000^{\circ}\text{C}$
ibid	H	0.80 $\pm$ 0.01	0.89	0.91	1.02 $\pm$ 0.02	ibid
Santoku 2 #1	D	1.49 $\pm$ 0.01	0.97	1.70	<u>1.71</u> $\pm$ 0.02	Baking $T \leq 170^{\circ}\text{C}$
ibid	H	1.30 $\pm$ 0.01	0.81	1.48	1.67 $\pm$ 0.02	ibid
Santoku 3 #1	D	1.66 $\pm$ 0.03	0.77	1.89	<u>2.38</u> $\pm$ 0.02	A2, 2.1sccm
ibid	H	1.64 $\pm$ 0.02	1.57	1.87	1.15 $\pm$ 0.02	A1, 10sccm

Note-1:  $\text{H}_2\text{O} \rightarrow 2\text{H} + \text{O} - 1.48\text{eV}$ , and  $\text{D}_2\text{O} \rightarrow 2\text{D} + \text{O} - 1.52\text{eV}$  (0.75 eV per H(D))

Note-2: Eout/D(H) values for chemical heat are 0.6 to 0.8 eV per D (or H), for nano-powders of Pd-black

Note-3: Eout/D value 1.7-2.3eV for Santoku-nano-Pd powder anomalously large. And Eout/H values are also anomalously large compared to known chemical values.

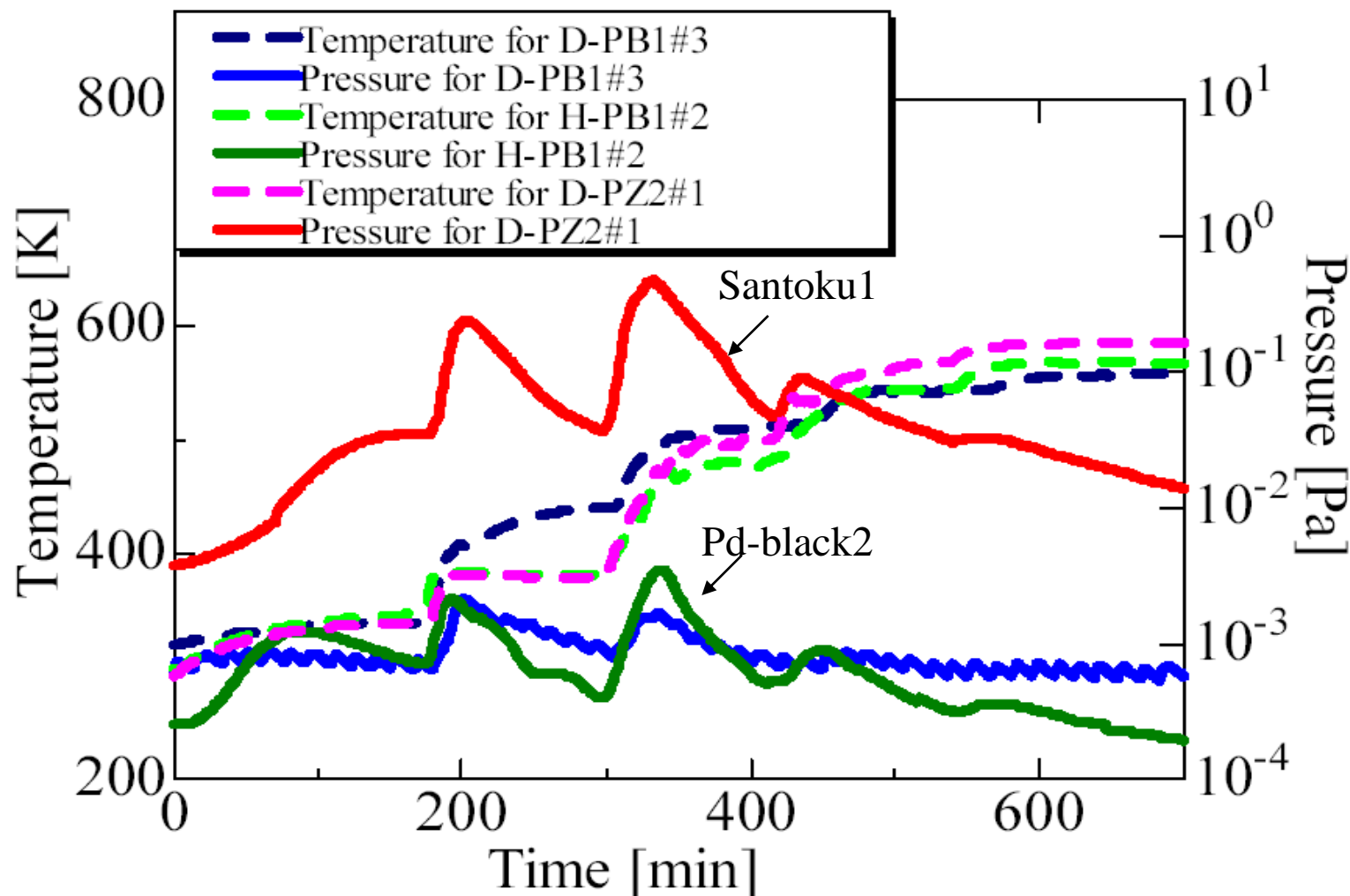
Note-4: Pd-black powder stuck to be bigger sizes after #1 run, but heat/D(H) is same to show some surface reaction active.

# Discussions for the 1<sup>st</sup> Phase

- Pd-black:  $(\text{Heat/D})_{\text{av}} = 0.70 (\pm 0.15) \text{ eV}$   
 $(\text{Heat/H})_{\text{av}} = 0.69 (\pm 0.1) \text{ eV}$
- Santoku1:  $(\text{Heat/D}) = 1.80 (\pm 0.02) \text{ eV}$  (2.38 for Santoku3#1)  
 $(\text{Heat/H}) = 1.02 (\pm 0.02) \text{ eV}$  (1.15 for Santoku3#1)
- Santoku2:  $(\text{Heat/D}) = 1.71 (\pm 0.02) \text{ eV}$   
 $(\text{Heat/H}) = 1.67 (\pm 0.02) \text{ eV}$  (\* flow rate larger for H-gas)
- After Fukai book: 0.2eV/H for bulk H absorption.  
100kJ/mol-H<sub>2</sub> : 0.5eV/H for surface adsorption.
- Reaction may be Surface Mesoscopic Phenomenon for the 1<sup>st</sup> Phase (“zero pressure” interval). Isotopic effect is visible.
- Pd nano-particle makes deep trapping potential of D(H), probably in fractal defects of its surface!?
- This is the reason of high loading “in vacuum” for Pd-nano.

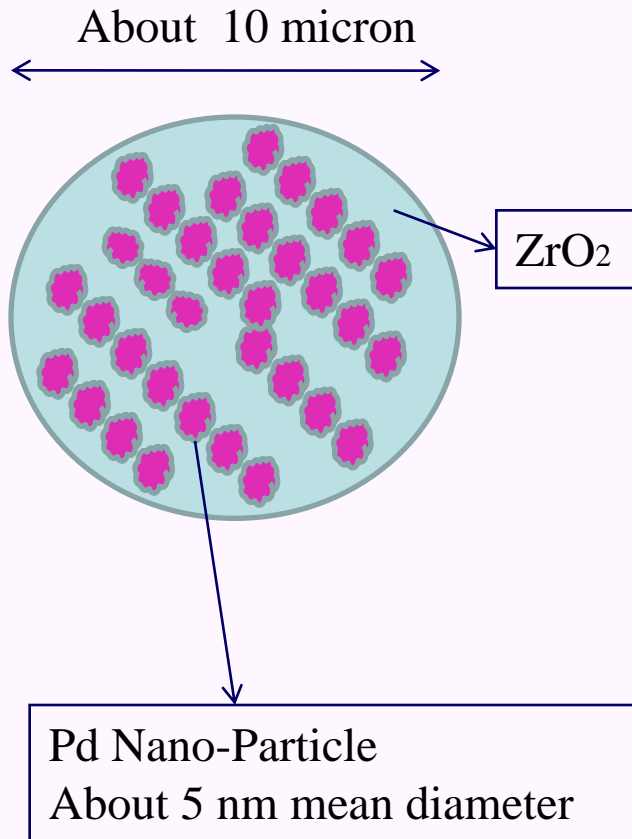


After evacuation, Santoku sample retains much more (100 times) D(H) than Pd-black: due to mesoscopic effect (rearrangement of surface and lattice) of Pd nano-particles.



4-3. Variation of pressure during sample baking for outgassing.

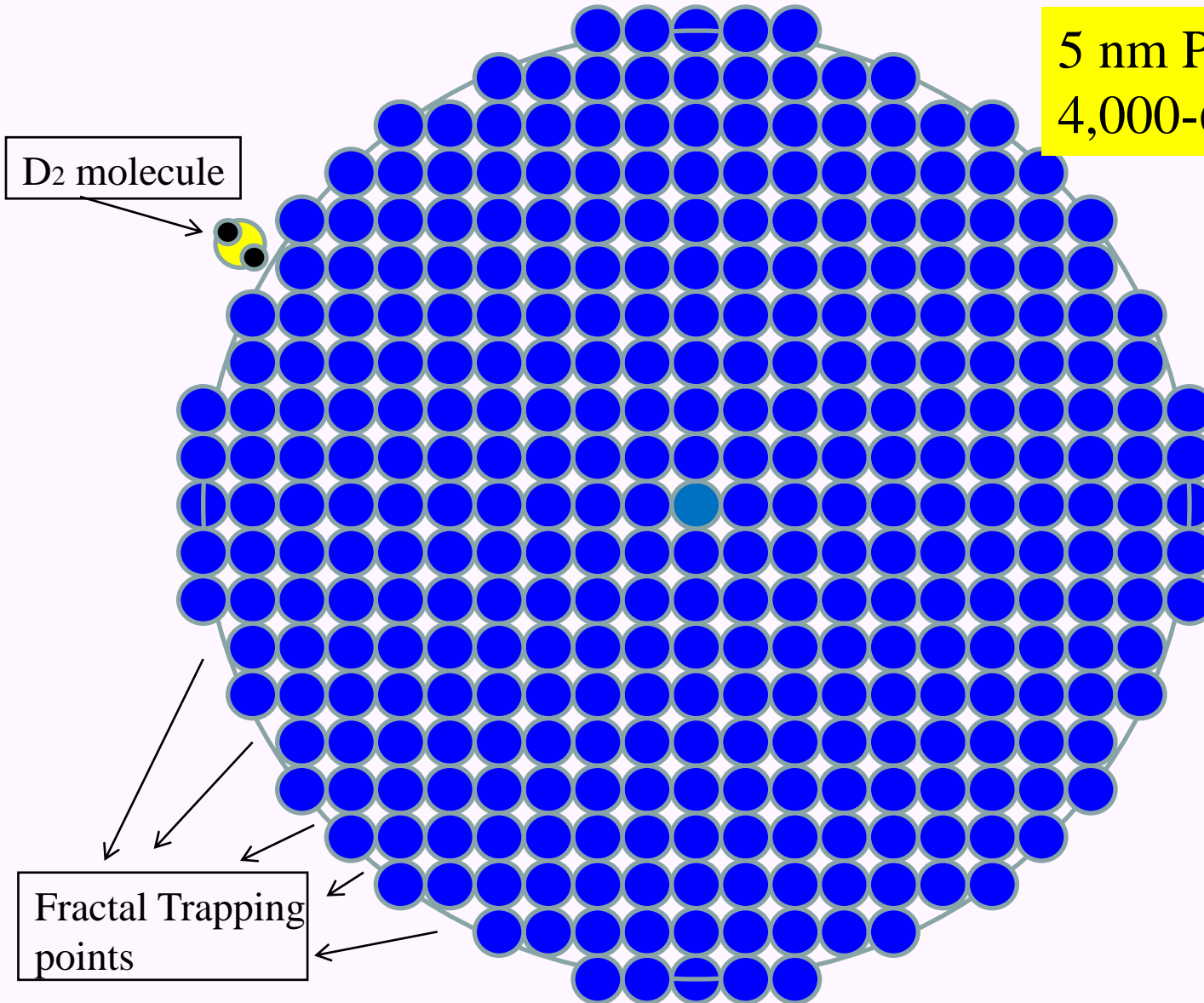
# Metal-Oxide-Nano-Pd Composite



- PdD<sub>x</sub>
- **X=1.0** by Arachi et al.;  
D-Absorption in O-sites of Pd-Lattice  
(We support this data)
- X=2.0 by Yamaura et al; deuterons at Pd-ZrO<sub>2</sub> interface  
(Voids?)
- Arata claimed: **x=2.5**

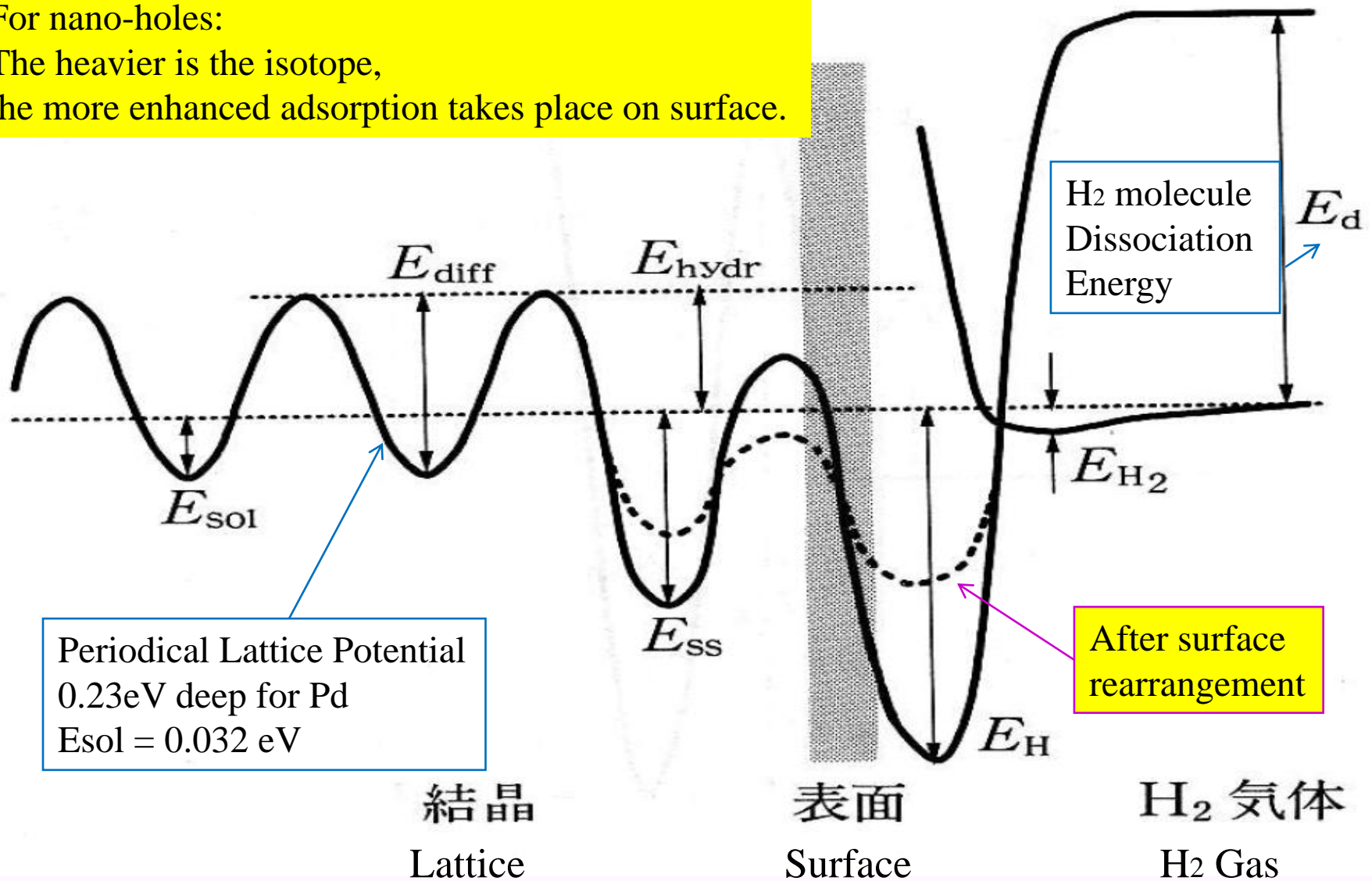
# Irregular and Fractal Sites should form on surface of nano-particle

5 nm Pd particle:  
4,000-6,000 atoms

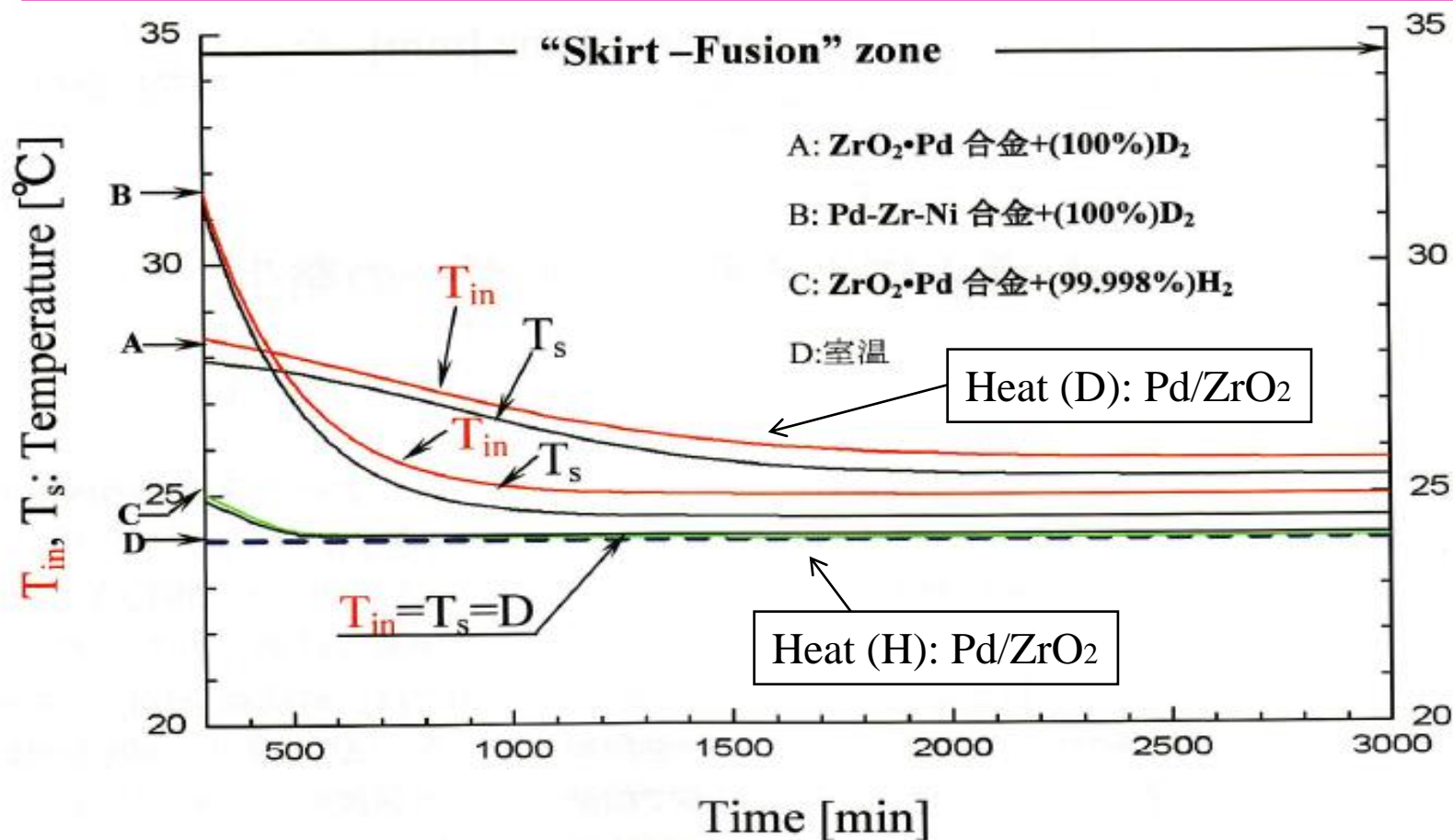


# Potential form of hydrogen adsorption and absorption near surface

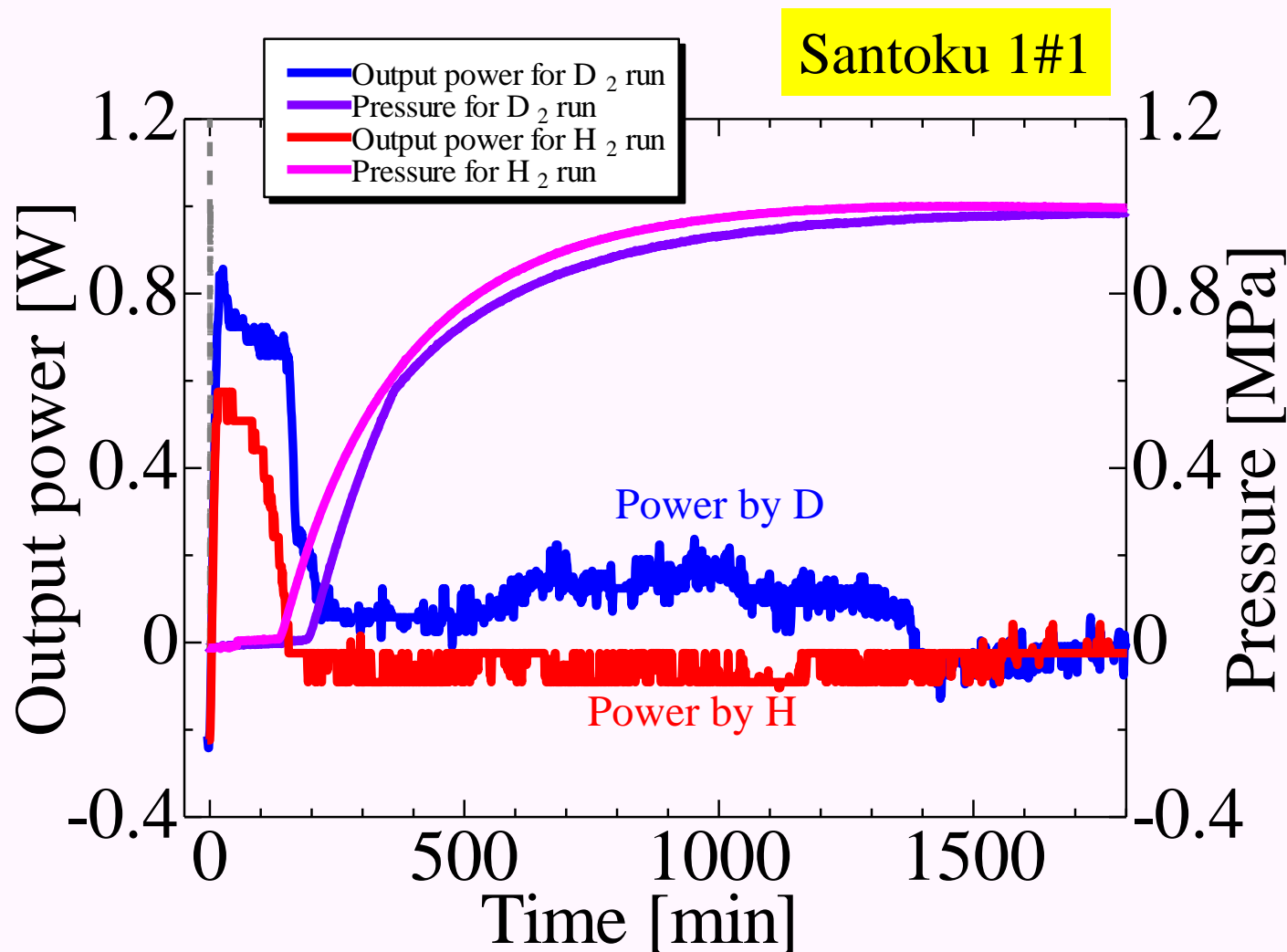
For nano-holes:  
The heavier is the isotope,  
the more enhanced adsorption takes place on surface.



**Long time lasting heat by D-charge without input power:  
29.2kJ Total by 24.4g Pd/ZrO<sub>2</sub> sample**



**Fig.5B “Skirt-Fusion” zone における各燃料にたいする  
Nuclear fusion の発生特性の比較(after 300min)**



Run number	flow rate [sccm]	Output energy [kJ]		Specific output energy[kJ/g]		Composition; x for PdD <sub>x</sub> /PdH <sub>x</sub>	Output energy per D/H in the 1st phase [eV]
		1st phase	2nd phase	1st phase	2nd phase		
D-PZ1#1	2.8	7.0±0.2	6.8±1.3	1.6±0.0	1.6±0.3	1.0	1.80 ± 0.05
Corrected H-PZ1#1	2.6	3.6±0.1	-5.1±1.4	0.8±0.0	-1.2±0.3	0.89	1.02 ± 0.03

## Discussions on Total Excess Heat Rates for #1 Runs: Our results are comparable to Arata's.

- **Arata-Zhang Exp.**

- Sample Pd/ZrO<sub>2</sub> by Fukuda P. M. Co.

weight: 24.4 g

net Pd weight: 10.5 g

- Observed Excess Heat  
(0-3000min): 29.2 kJ

- Excess Heat Rate:

**2.77 (kJ/g-Pd)**

**(Anomalous heat by H**

**about 1 kJ/g inclusive)**

**→ Net Value ~ 1.8(kJ/g-Pd)**

- **Our Exp.**

- Sample Pd/PdO/ZrO<sub>2</sub> by Santoku Co.

weight: 10 g

net Pd weight: 4.3 g

- Observed Excess Heat  
(Example for S2, 0-3000min)

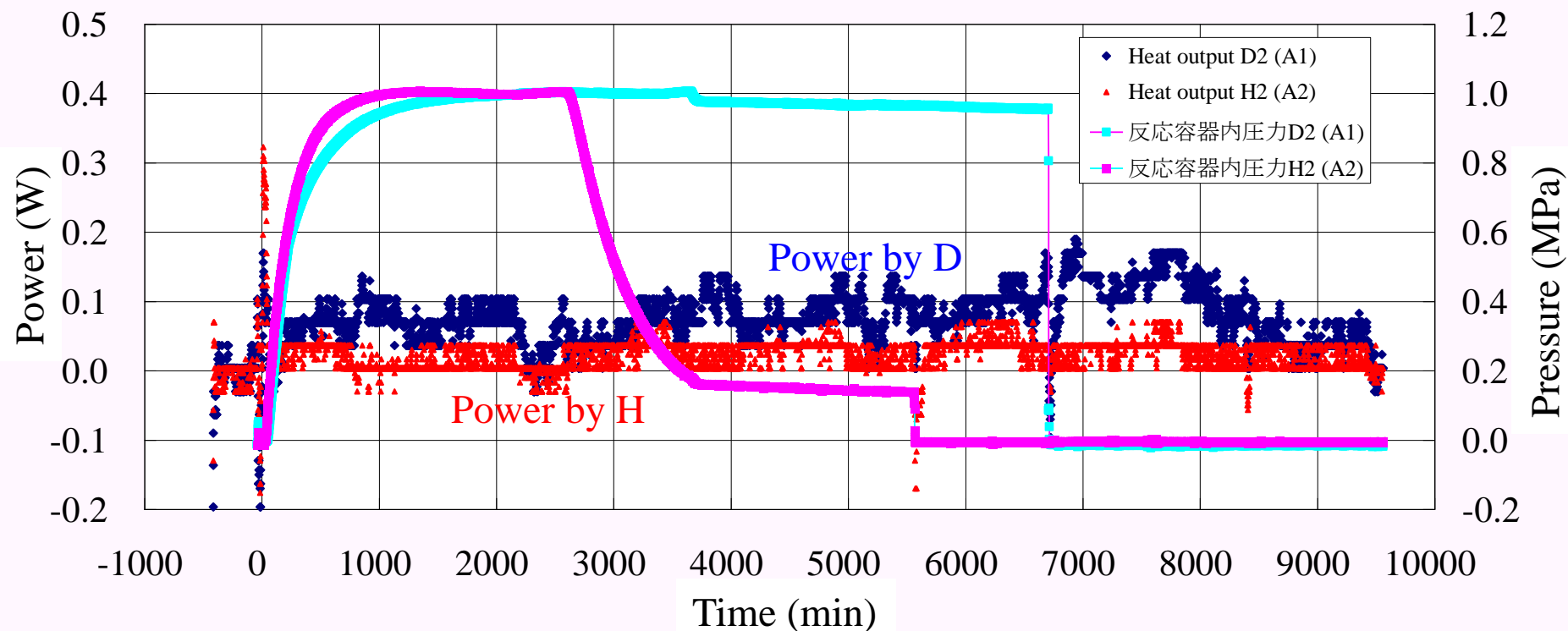
S2:  $(6.4-5.1)+7.0 = 8.3 \pm 1.2$  kJ

- Excess Heat Rate:

**S1:  $2.64 \pm 0.30$  (kJ/g-Pd)**

**S2:  $1.93 \pm 0.30$**

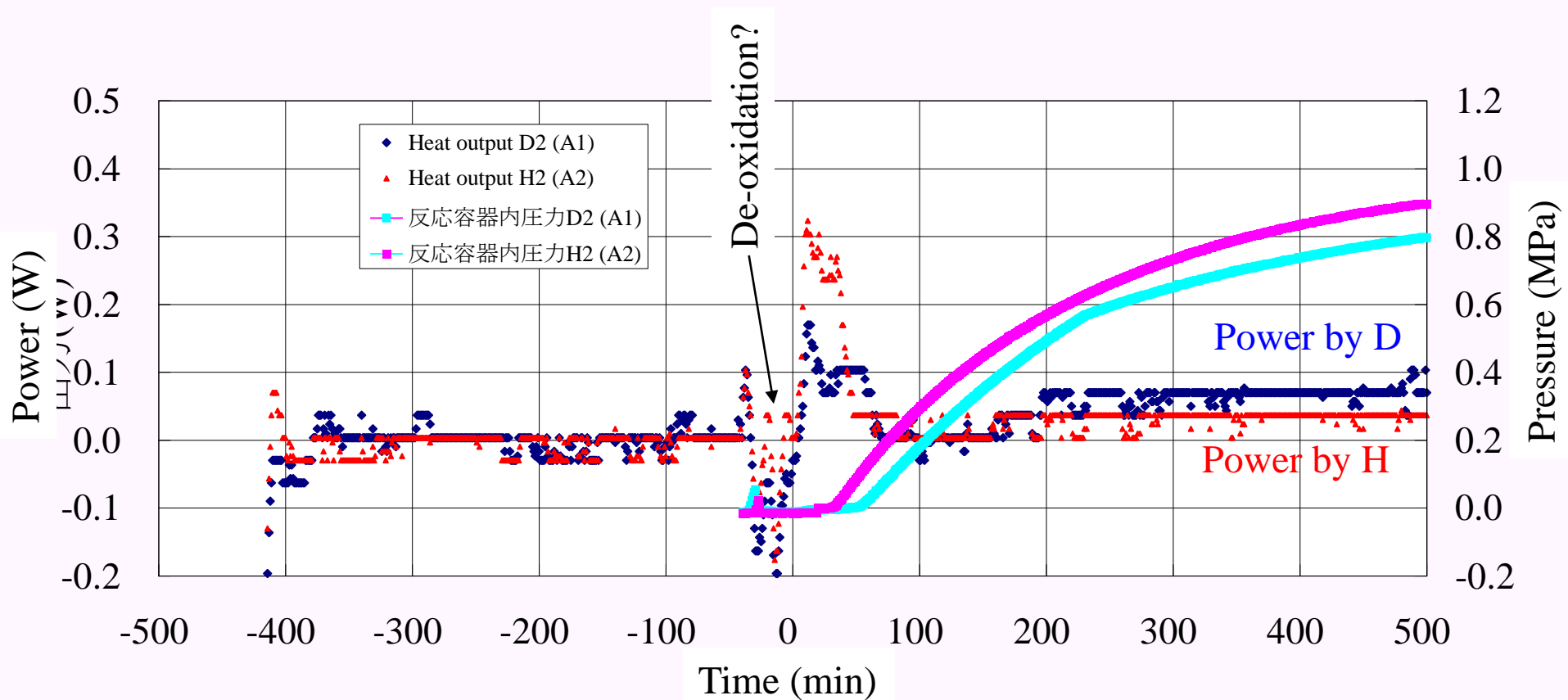
# Long Time Lasting Excess Heat by Run: Santoku 2#2



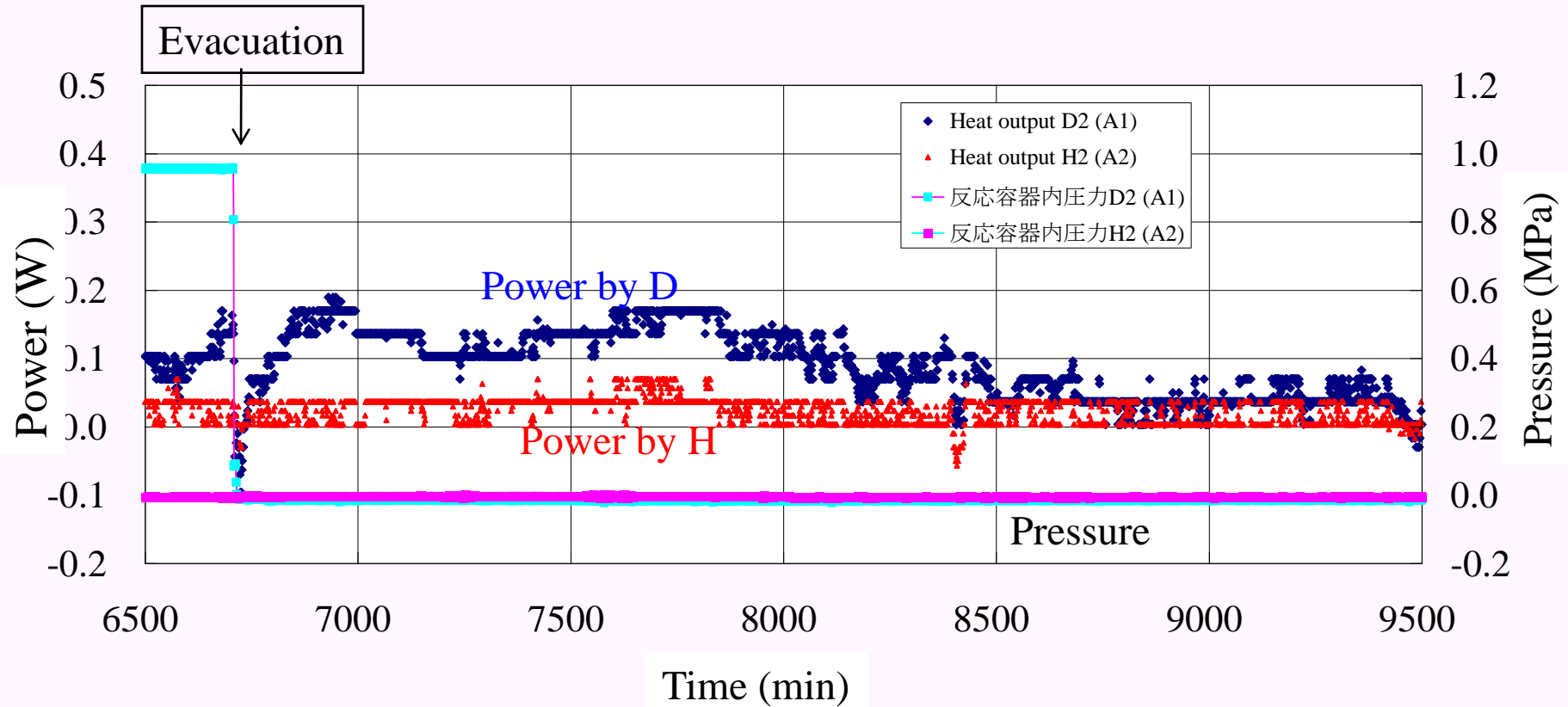
run	Gas	Measured flow rate [sccm]	1st phase [kJ]	2nd phase [kJ]	1st phase [J/g]	2nd phase [J/g]	D/Pd or H/Pd
D <sub>2</sub> -2-2	D	3.87	0.17±0.03	9.89±1.48	40±7.0	2300±345	0.47
H <sub>2</sub> -2-2	H	3.62	0.58±0.05	1.68±1.46	136±10.9	391±341	0.28



# Run: Santoku 2#2: Expanded View of Early –Time Evolution

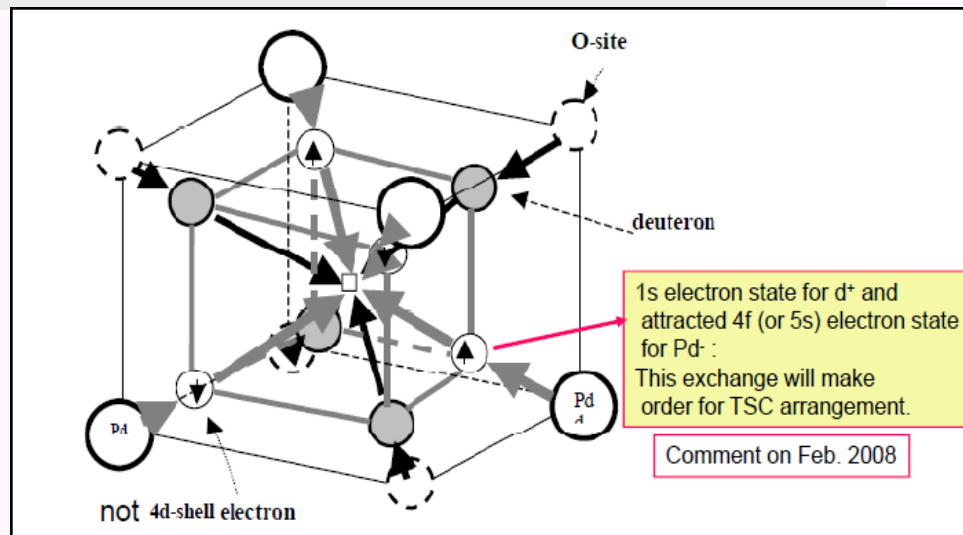
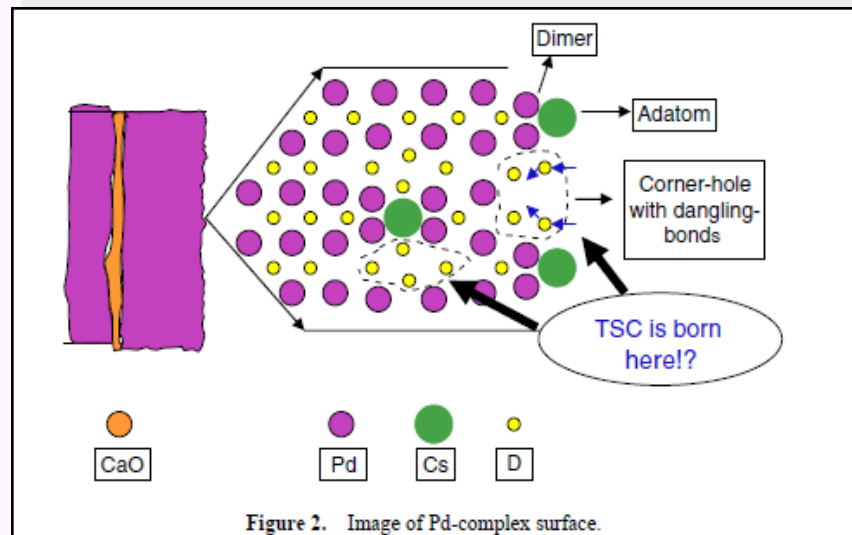
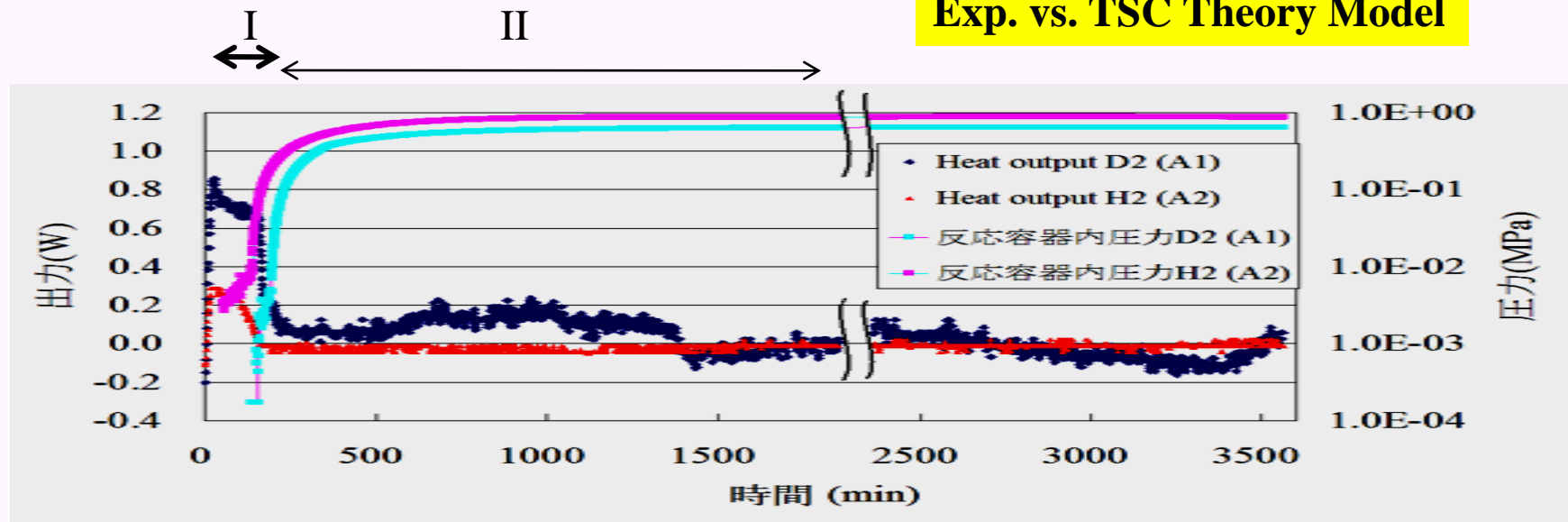


# Run: Santoku 2#2: Expanded View of Heat Evolution after Evacuation- “Heat after Death”



Is Evacuation STIMULUS?

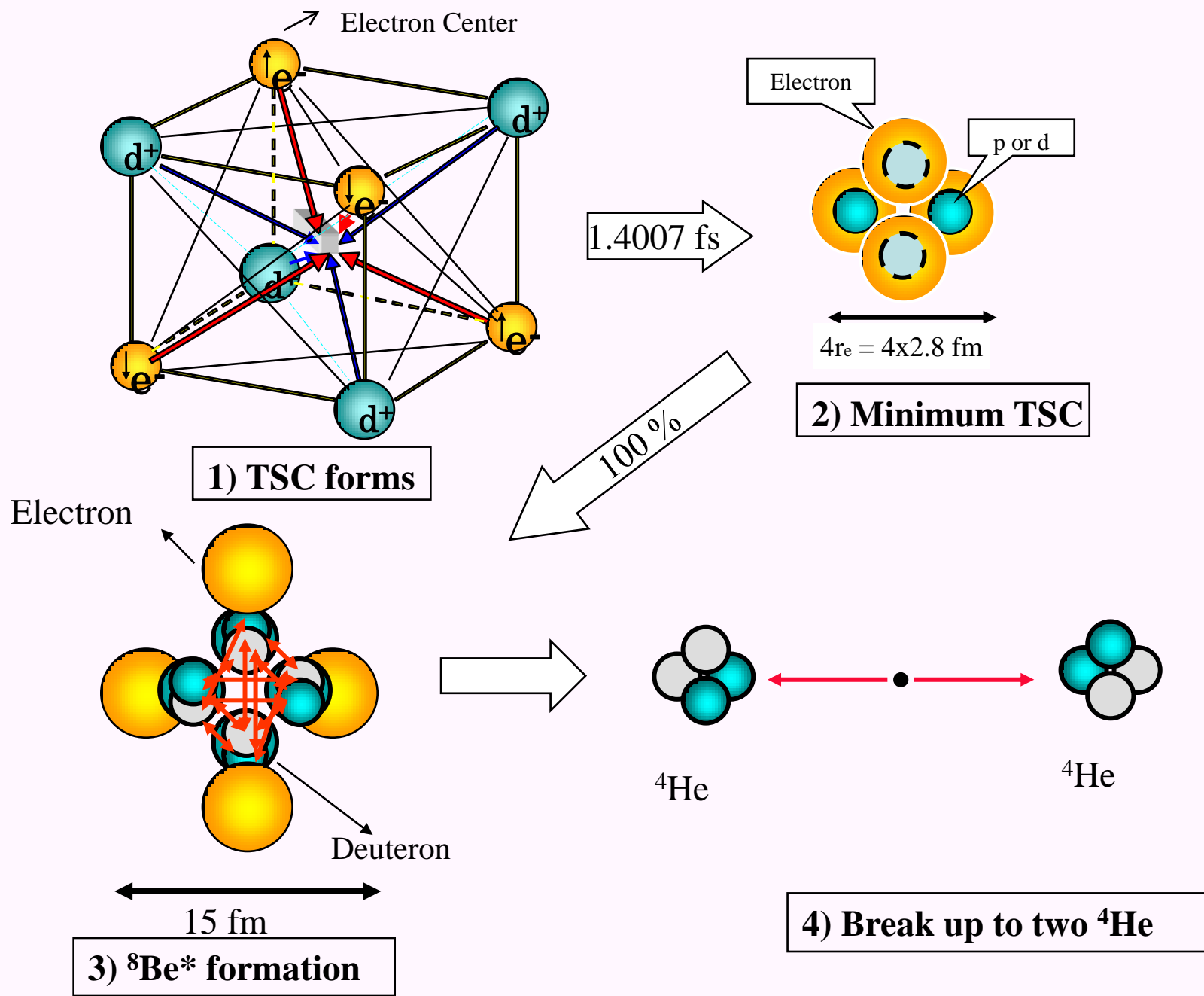
## Exp. vs. TSC Theory Model



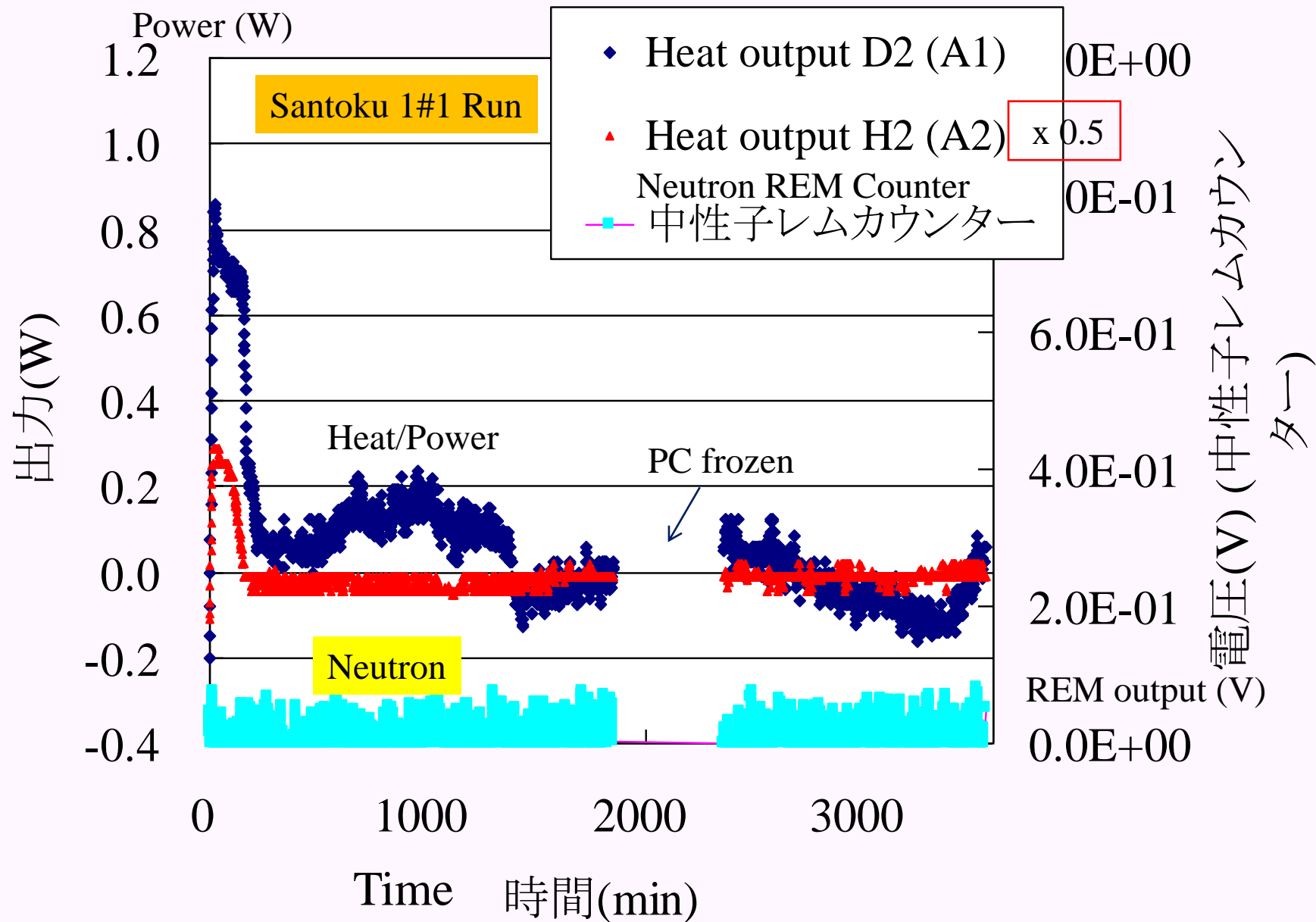
Model b) may correspond to I.

Model a) may correspond to II.





No increase of neutron count rate by REM counter was seen, gamma-ray either.





# Conclusions-1

- Arata-Zhang's Excess Heat Result was replicated quantitatively.
- For Pd/PdO/ZrO<sub>2</sub> powder (Santoku):
  - 1) D-gas charge in the 1<sup>st</sup> phase (zero pressure) gave 20-90% excess heat than H-gas charge.
  - 2) In the 2<sup>nd</sup> phase, significant excess heat (about 2 kJ/g-Pd) for D-gas charge, while zero level for H-gas charge.
- No increase of neutron counts was seen.
- D/Pd ratio in the end of 1<sup>st</sup> phase was 1.0, while H/Pd was about 0.8 - 0.9. Flow rate dependence.
- Further experiments changing conditions will be fruitful for developing clean energy devices.

## Conclusions-2

- Nano-Pd dispersed sample (Santoku, Pd/ZrO<sub>2</sub>) retained 100 times more D(H) atoms after evacuation, than the Pd-black case.
- Mesoscopic effect by Pd-nano-particle, namely surface and lattice rearrangement probably makes deep D(H) trapping potentials (1.0-2.3eV). We need study for D(H)-gas flow-rate dependence.
- Stable excess heat production is expected for #2 and later runs. We need further study for this.
- Detection of nuclear products by the B-system is expected.
- Replication by other groups is important.