



Research Article

Role of PdO Surface-coating in CMNE D(H)-Gas Loading Experiments *

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Abstract

The PdO-coated layer of Pd-nano-particle may arrange fractal nano-dips on its surface when D(H)-gas is charged and de-oxidation molecules (D_2O or H_2O) are released. Fractal nano-dips may make local deep adsorption potentials, through which rapid penetration of D-atoms (ions) into deeper Pd-local lattice (Bloch potential) O-sites of nano-particle may be induced, to realize full or over-full D(H) loaded state ($x > 1.0$) of $PdDx$ in a short time of the Phase-I process. Formation of D-clusters, such as 4D/TSC on surface may be enhanced at nano-dips. A phenomenological model of quasi-free D-motion under constraint of the Bloch potential within a global mesoscopic potential well is proposed for the Phase-II phenomena, where nuclear heating by 4D fusion reactions may rather steadily take place. Generation of collective mesoscopic potential well (CMPW) will make a Pd nano-particle with PdO working as “mesoscopic catalyst” which realizes very large D(H)-loading ratios and anomalously large chemical heat releases both for H-gas and D-gas loading.

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1. Introduction

We of the Kobe Group have reported newer results on anomalous D(H) absorption and excess heat by nano-Pd/Metal-Oxide dispersed samples in the JCF10 meeting 1,2, 5–6 March 2010, Tokyo, and also in the ACS-2010 NET Symposium7, 21–22 March 2010, San Francisco.

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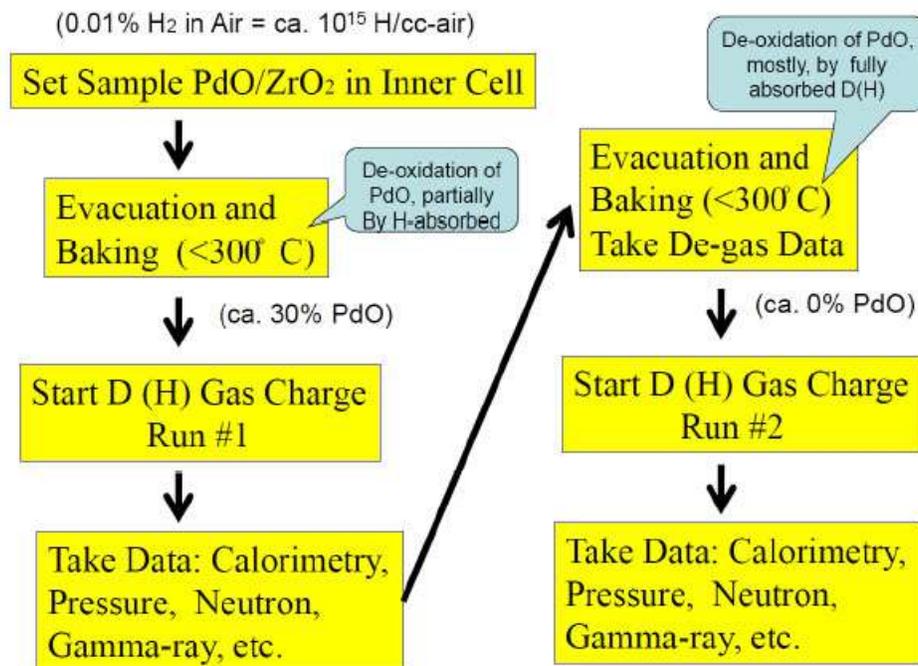


Figure 1. Experimental procedure of D(H)-gas charging to Pd/ZrO₂ powder samples and reduction of PdO.

Two new findings were reported there: (1) Forced oxidation of used Pd/ZrO₂ samples showed remarkable recovery effects on D(H)-loading ratios and heat release rates in the Phase-I interval of D(H)-charging experiments. (2) By time-resolved (time-dependent) measurements of D(H)-loading ratios, we have found the “new second phase” after the Phase-I. At the end of Phase-I, D(H)/Pd ratios became 1.1–1.2 with integrated specific heat-energy 0.83–2.0 eV/atom-Pd. At the end of new second phase (Phase-II) with slower change of D(H)-absorption, additional low level heats were recorded. The third phase (Phase-III) was redefined for the time-period after D(H)/Pd ratios were saturated in Phase-II. We discuss on what kind of underlying physics is there, by proposing a phenomenological model which may relates to the TSC theory [3–5], for the emerging condensed matter nuclear effects (CMNE).

Reduction process of PdO in samples under D(H) charging is first discussed briefly in this paper. Secondly and mainly, the role of PdO surface coating of Pd nano-particle is discussed using a phenomenological (speculative) model on what happens under D(H) charging to induce a mesoscopic catalyst potential and the D-cluster formation and 4D/TSC fusion. We summarize discussions as;

- (1) The de-oxidation of PdO may be made mostly during the baking processes between D(H)-loading runs, because of high level D(H)-density under degassing process at relatively high (473 K) temperature which may enhance D₂O (H₂O) chemical formation.
- (2) The PdO-coated layer of Pd-nano-particle may arrange fractal sub-nano-dips (sub-nano-holes; SNH) on its

- surface when D(H)-gas is charged and de-oxidation molecules (D_2O or H_2O) are released to vacuum.
- (3) Fractal sub-nano-dips may make local deep adsorption potentials, through which rapid penetration of D-atoms (ions) into deeper Pd-local lattice (Bloch potential) O-sites of nano-particle may be induced, to realize full or over-full D(H) loaded state ($x > 1.0$) of PdD_x in a short time of the Phase-I process.
 - (4) Formation of D-clusters, such as predicted by the TSC theory [3–5] on surface may be enhanced at sub-nano-dips already in the beginning of Phase-I D(H)-loading process and 4D/TSC fusion may take place with considerable probability.
 - (5) D-motion in a “mesoscopic deep collective potential” of the nano- PdD_x system may be quasi-free to enhance transient D-cluster (4D/TSC [3–5]) formation probability around tetrahedral sites of local Bloch (periodical-lattice) potential inside a PdD nano-particle. This state can be modeled by the non-linear combination of two oscillations, i.e., one (longer pendulum in approximation) by the deep (about 1.5 eV deep) well-type collective potential and the other (shorter pendulum) by the lattice-type periodical three dimensional Bloch potential (about 0.22 eV deep). The shorter pendulum may have “high excited energy” to realize very mobile D-motion under the constraint of three-dimensional local PdD lattice arrangement, within the mesoscopic global deep potential well.
 - (6) Generation of collective mesoscopic potential well (CMPW) will make a Pd nano-particle working as “meso-

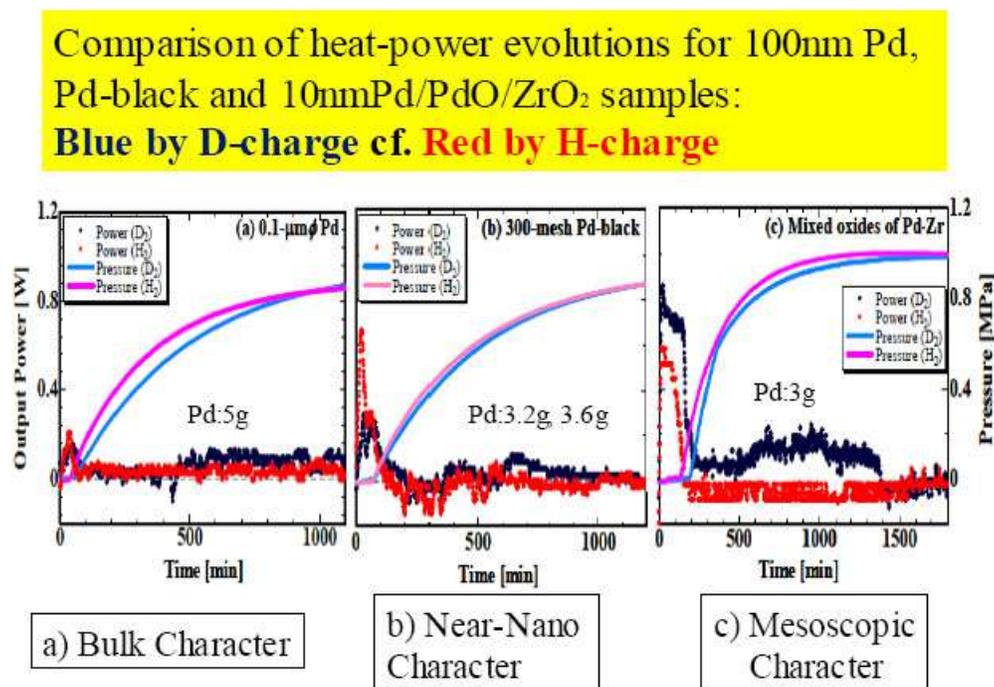


Figure 2. Comparison of typical heat evolution data for PP (0.1 μm diam. Pd powder), PB (300 mesh Pd-black powder) and PZ (10 nm-Pd-nano-powder dispersed in ZrO_2 flakes).

scopic catalyst” which realizes very large D(H)-loading ratios and anomalously large chemical heat releases both for H-gas and D-gas loading.

2. Formation and Reduction of PdO Surface Layer of Pd-nano-particles

Virgin samples of Pd/ZrO₂ (PZ in abbreviation) as provided from the maker (Santoku Co., Kobe, Japan) contained oxygen in the form of PdO/ZrO₂ with 100% PdO and ZrO₂.

Before the first D(H)-charging runs (by the twin system [1,2,7]), we set up PZ samples in reaction chambers of the twin system, evacuated the chambers and baked samples at 200°C to remove “already absorbed” impurity gases. Since samples have been reserved in air for a few months, Pd-particles (PdO) would have absorbed a considerable amount of hydrogen in air (0.01% of air is H₂ gas, 10¹⁵ H₂ molecules per cc air). During a baking process, significant portion of PdO would have been de-oxidized by forming D₂O (H₂O) vapors to be evacuated. PZ samples may be a composite of (PdO)_yPd_x/ZrO₂ with $x + y = 1.0$. We have then made the first D(H)-charging runs to observe D(H)/Pd loading ratios and heat–power evolution in Phases I–III. Before the second D(H)-charging runs for used PZ samples, we have made evacuation and baking. In this second baking stage, we speculate that almost 100% reduction of PdO would have been done by “water-formation” reactions between out-going D(H) atoms and oxygen atoms in PZ sample under the elevated temperature (200°C) which enhanced chemical reaction rates. Such an experimental procedure as above mentioned is flow-charted in Fig. 1.

We refer an essential results of Kitamura et al. PLA 2009 paper [6], for heat–power evolution data under D(H)-gas charging to three kinds of Pd powder samples (PP, PB and PZ), as shown in Fig. 2.

How can we explain the results of anomalously enhanced heat–power evolutions for PZ samples both for D- and

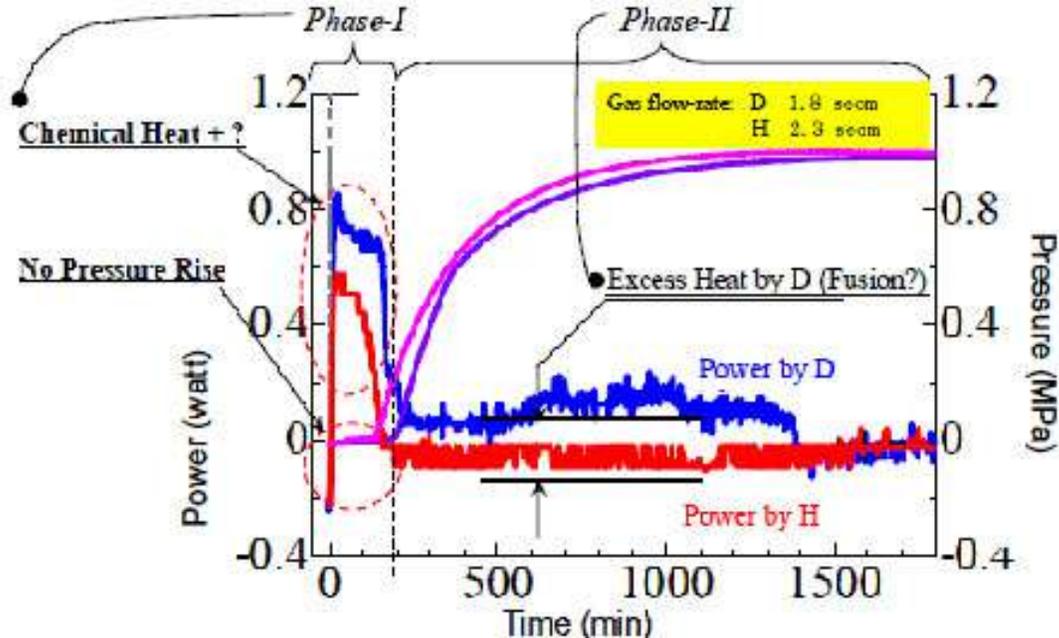


Figure 3. Definition of phases I and II intervals for the D(H)-gas loading experiment [6,7].

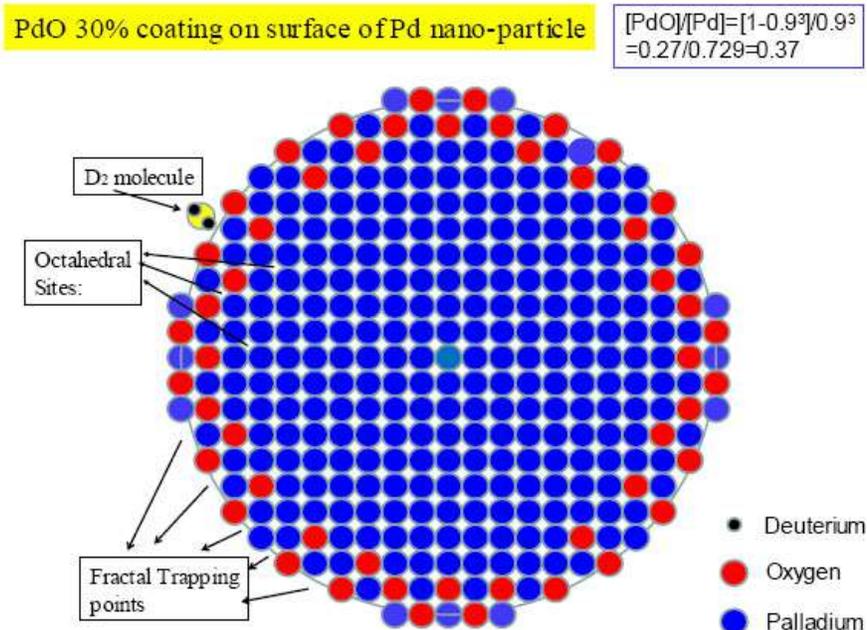


Figure 4. An atomic scale cross sectional image of PdO-layer coated (30%, $y = 0.3$) Pd nano-particle (5 nm diameter); here we used relative sizes of ionic radii and D_2 molecule, and fractal surface structure is supposed to make a role enhancing deeper adsorption potential for D(H)-gas.

H-charging, and why D-charging gave significantly larger heat–power in Phase-I and only gave positive heat–power level in Phase-II? What is a role of PdO component, since the second D(H)-charging runs with used samples (without PdO component) gave very reduced D(H)/Pd-loading ratios and heat? Experiments with forced oxidation [1,2] (4–8% of total Pd amounts) to used PZ and PNZ (Pd/Ni/ZrO₂) samples showed remarkable recovery for D(H)/Pd ratios (up to 1.1–1.8) and heat levels (0.8–2.0 eV) in Phase-I. What is the mechanism for that? Are these phenomena related to the proposed mechanisms of Takahashi’s TSC formation models and D-cluster (4D, typically) fusion reactions?

3. A Phenomenological Model of D(H)-Adsorption/Absorption with PdO Layer

We propose phenomenological models in Sections 3 and 4 for the phase-I and phase-II phenomena as defined in [6]. We show a modified figure for helping this purpose in Fig. 3.

From our analysis of forced-oxidization experiments [7], we have speculated that a portion of PdO at the beginning of first (virgin) D(H)-charging run maybe around 30% ($y = 0.3$ or so). Assuming a formation of PdO surface layer (coating with $y = 0.3$) of Pd nano-particle, an image of atomic scale cross section of PdO/Pd nano-particle (5 nm diameter, for instance) is shown in Fig. 4. By TEM image of used sample (as to be reported by Kitamura ICCF 16 paper, and also we reported [7,17] at ACSNET 2010), 2–10 nm PdO/Pd particles are dispersed in/on ZrO₂ flakes with several micro-meter size. We assume here PdO/Pd particles are kept as isolated each other in a supporter ZrO₂ flake. Position of oxygen-atom can be interstitial or substitute for Pd-atom, but we draw here simply as substitute of Pd-atom.

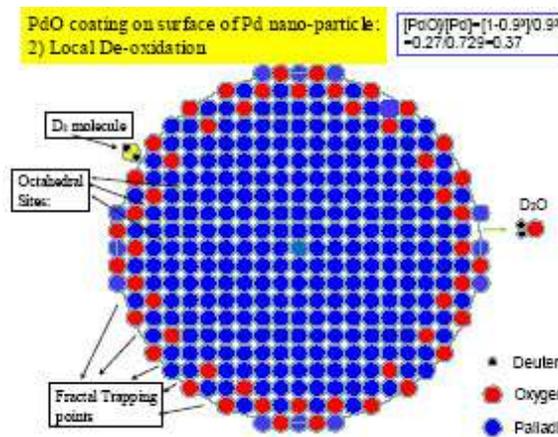
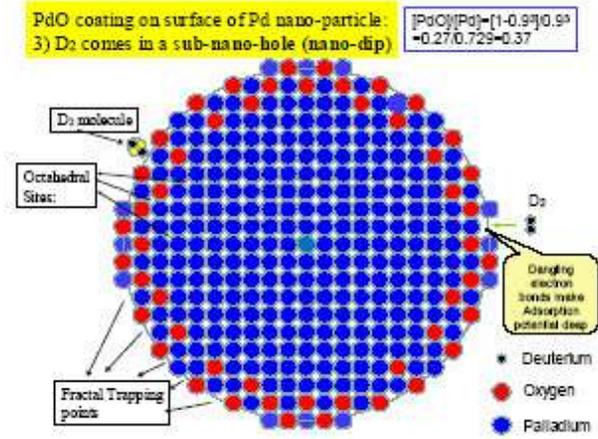
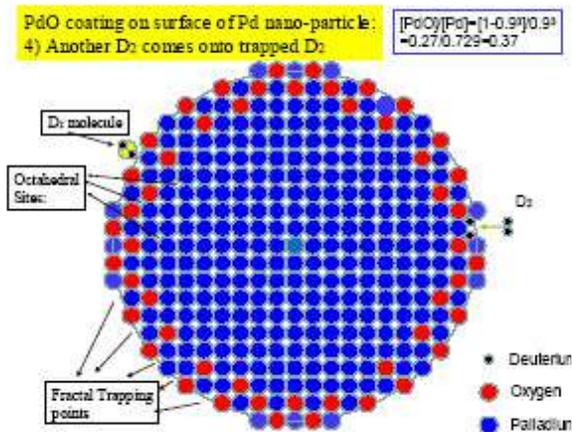
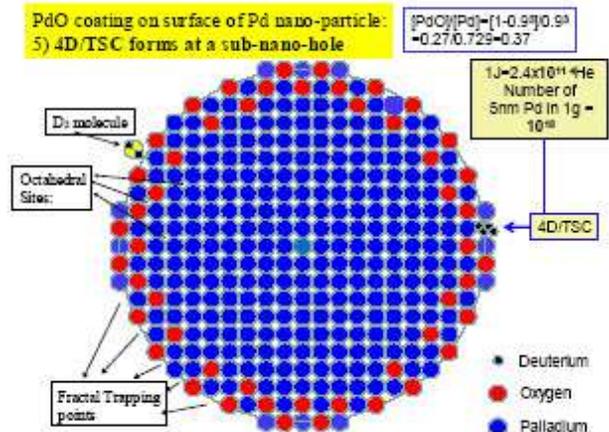
Figure 5. D₂O molecule going out.Figure 6. SNH traps D₂ incoming.Figure 7. Eventual double D₂ trapping.

Figure 8. Eventual 4D/TSC fusion on surface.

We are going to propose a mechanism that oxygen of PdO layer will serve as a “seed” for generating a “sub-nano-hole” (SNH) when D(H)-gas will be started to charge and formation of D₂O (H₂O) by incoming D₂(H₂) gas molecule to combine (de-oxidize PdO) with oxygen. Formed D₂O (H₂O) molecule will go out to vacuum, and a SNH with chemical electron dangling bonds will be generated. As the trapping potential of SNH will be “very” deep (strong sticking force by electron-dangling bonds), incoming D₂ molecules will be doubly (or more) trapped there to form a transient TSC (*transitory Bose–Einstein condensate*) with certain probability. The image of such TSC formation is speculative at this

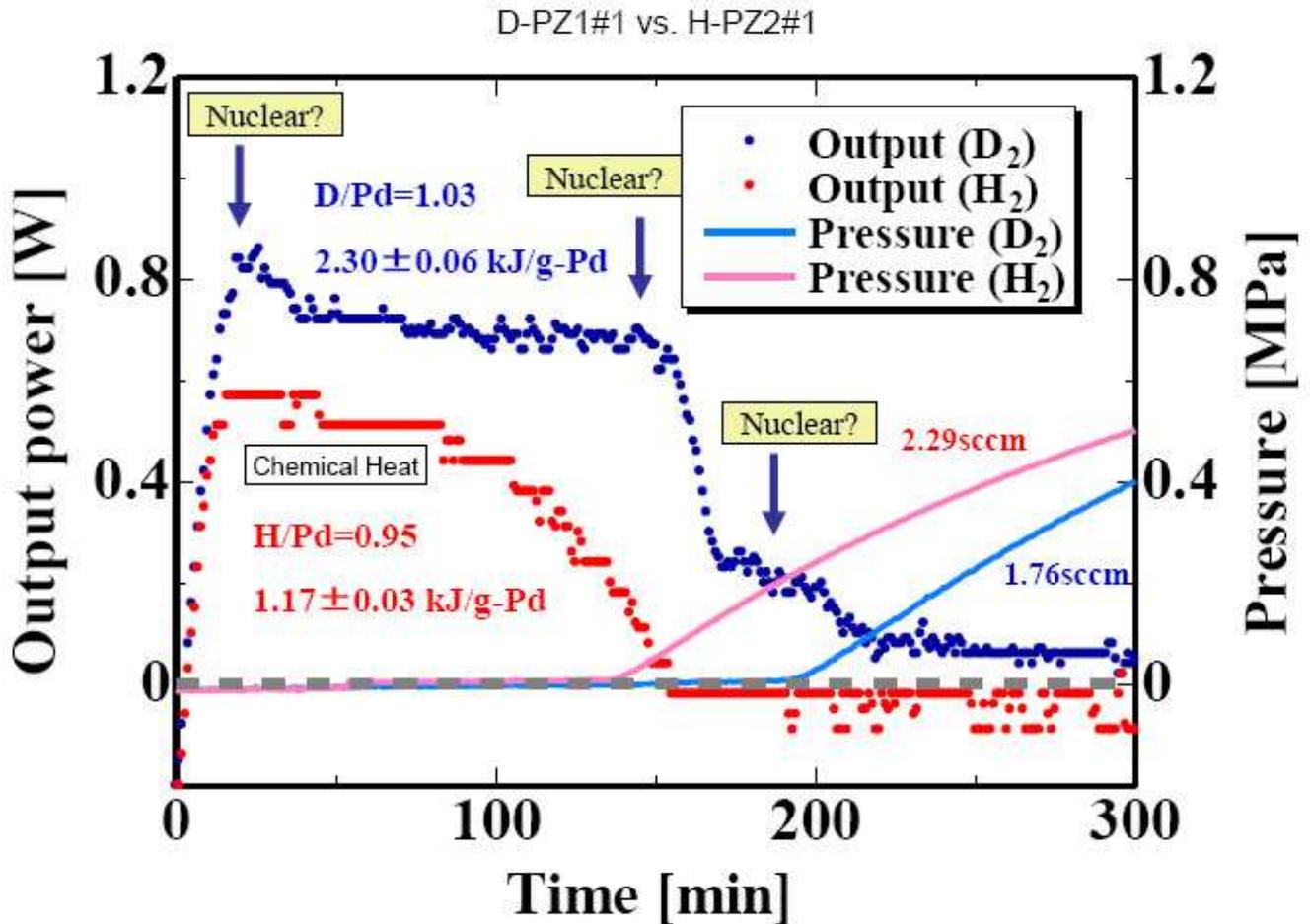
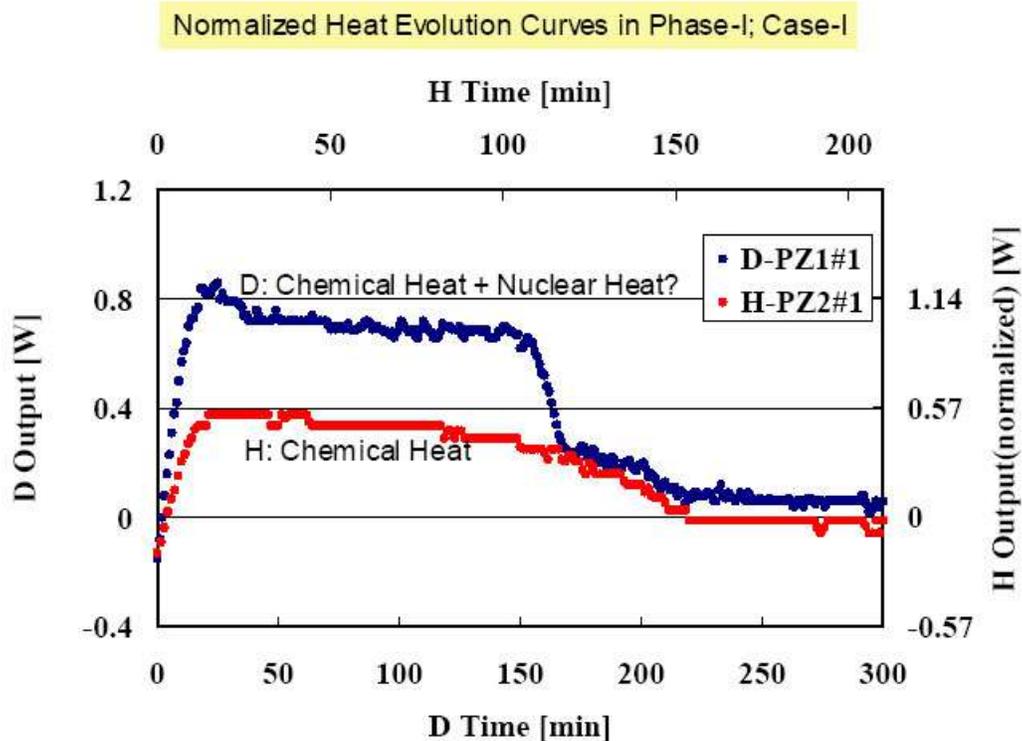


Figure 9. Expanded view of heat–power evolution data by Kitamura et al. [7] for PZ samples.

stage, and we need a further deepened study based on the first principle type quantum-mechanical calculations using time-dependent coupled Schrodinger equations for the SNH composition of three dimensional Pd-atoms arrangement and in-coming D_2 molecules. However, this is not so easy task.

Once a TSC ($t = 0$) is formed, very fast (in 1.4 fs) condensation happens to make a very small (in 10–20 fm diam.) charge neutral entity (TSC-min) which will cause 100% 4d-simultaneous fusion to produce two ^4He products with heat (23.8 MeV/ ^4He) [3–5]. Image of such a process is illustrated in Figs. 5–8. After such an event of D_2 trapping at SNHs, remained surface fractal nano-holes will trap more incoming D(H)-gas and enhance “rapid diffusion” into inner PdDx lattice points (namely O-sites of local PdD lattice), as we speculate existence of “D(H)-pressure” from backside where trapping of D_2 molecules is taking place one after another and accumulating “surface D-clusters”.



As shown in an inset (right top) of Fig. 7, 1 g of Pd nano-powder contains 5 nm-diam. nano-partilces of 10^{18} (of the order). Since 10^{11} 4d/TSC fusion events corresponds to 1 J, one watt heat-level is maintained by a TSC formation rate of “one over 10 million Pd nano-particles per sec” for PZ sample containing 1 g net Pd atoms. This condition looks feasible, in reference to observed heat–power evolution levels by Kitamura et al. [1,2,7].

A typical data reported by Kitamura et al. [7] for Phase-I is expanded and is shown in Fig. 9. We can consider that the heat–power evolution curve for H-charging is purely by chemical heat producing reactions, while the curve for D-charging may have component of some isotope effect, probably “nuclear heating” as shown with specific patterns (see arrows). Data by the twin system [7] gave different end-points of Phase-I for the H-charging and D-charging, respectively, and D(H)/Pd loading ratios are slightly different. We can draw normalized curves by adjusting time-intervals of Phase-I and loading ratios, as shown in Fig. 10.

From Fig. 10, we can have impression that a beginning peak and a later bump of heat-power evolution in the D-charging run look like “nuclear heating components”, while bumps around 200 min are due to a pure chemical heat-power evolution. We have observed and deduced similar normalized heat-power evolution curves, three times, for PZ virgin samples (10 g each) in 2008-2009. Hioki, et al (Toyota Central Research Laboratory) reported8 similar heat-power evolution curve having a very clear peak (sharp spike) in the beginning and a broad bump later for a PZ

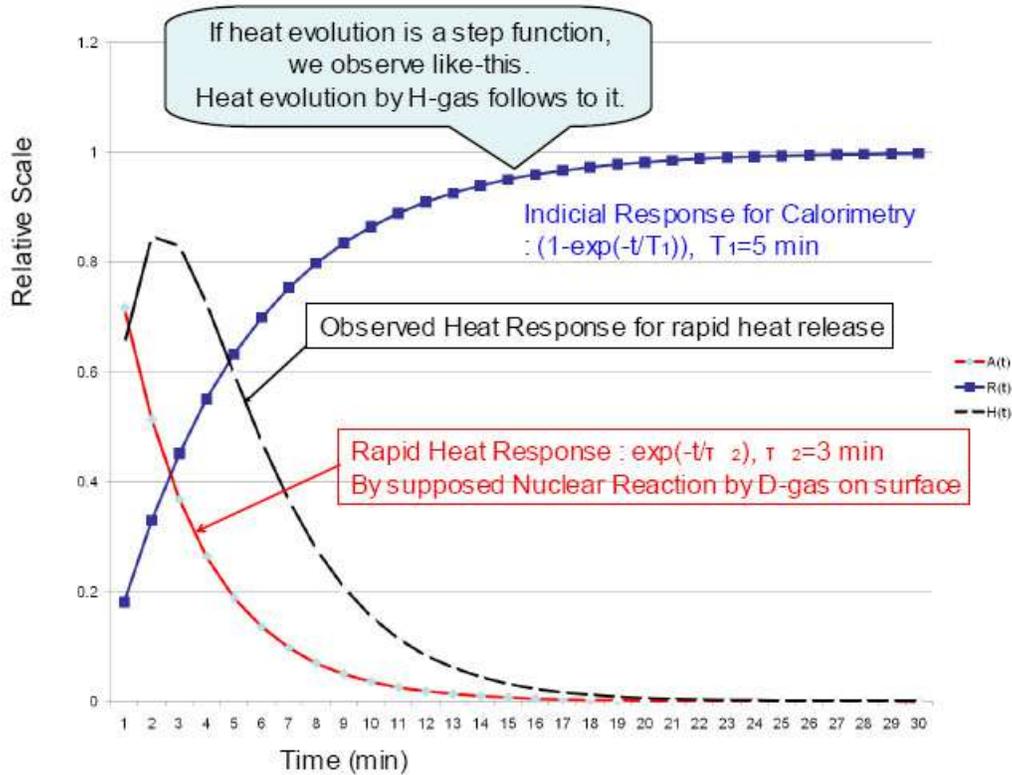


Figure 11. Observed “rapid” heat response is broadened by indicial response of calorimetry, assuming a single exponential “nuclear heat evolution by D-absorption in Phase-I. Such a “nuclear response” may be a superposed component in observed heat evolution data for D-gas, in comparison with rather smooth response by H-gas.

sample (from Santoku Co.) of 54 g. To assure that this is really due to nuclear (fusion) heat, we have of course to detect correlated ash (maybe alpha particles, other minor charged particles, secondary X-rays, EUV and visible lights) on line and/or off-line. This is our future task.

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On the other way of precisely observing time-dependent behaviors of heat evolution in the beginning just after D(H)-charging, we may obtain anomalously large isotopic effect, namely too large difference of heat–power levels

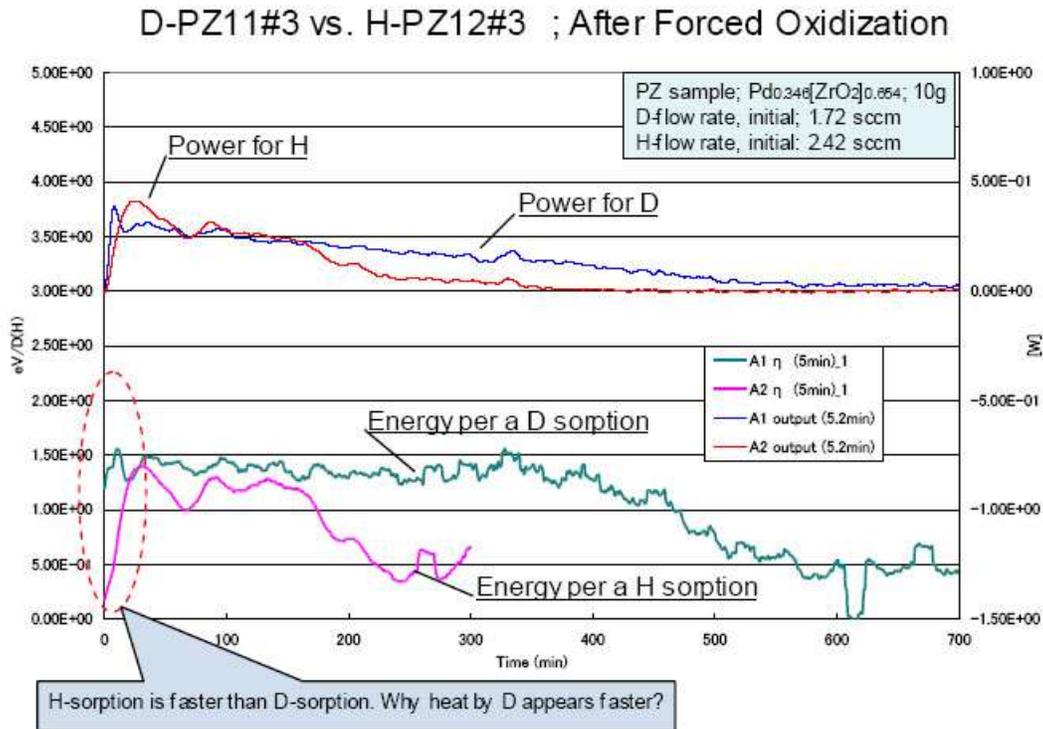


Figure 12. Heat evolution curves and η -values for PZ-11(12)#3 runs; nuclear heating looks appearing in the early stage circled by a red broken elliptic line, for D-gas charging. Amounts of PdO components in PZ-11(12)#3 runs, by forced oxidization, are 8.5 % (5.4 %) for D (H), respectively.

between D-gas and H-gas runs. By exploring such a phenomenon, we may have dynamic information of heat release mechanisms by “new kind of D-related nuclear reactions” such as 4D/TSC cluster fusion.

We can feel now vividly, by such normalized heat–power evolution runs taken by a twin system, that released heat–power by D-charging has a significant component of “some D-related fusion” reactions, due to its time-dependent shapes very different from those for H-charging.

The mechanism of D(H)-absorption in Phase-I is independent of that in Phase-II (the new second phase [2]). The former is a rapid process of absorption with relatively large heat–power level, while the latter looks a slow adsorption/absorption process with relatively weak (about 1/10 of that in Phase-I) heat power level albeit having similar integrated D(H)/Pd loading ratio components (0.5–1.0). Time-dependent D(H)-absorption rate in Phase-I drew decay curves of almost same rates both for D- and H-charging. This fact is another background that the heat–power evolution for D-charging (Fig. 10) looks like containing component by nuclear origin.

Our calorimetry system had a time resolution (time constant) of 5 min and observed heat evolution curve became a convolution with its indicial response and exponential absorption rate. If heat release rate is constant (a step function),

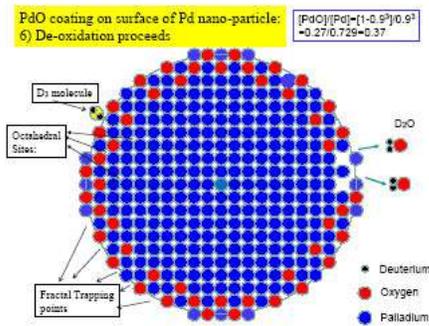


Figure 13. Extended sub-nano dips by D₂O formation.

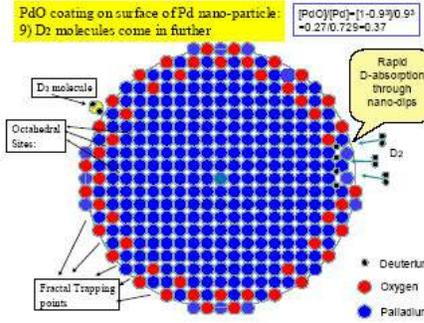


Figure 14. Incoming D₂ molecules are trapped one after another, by dangling bonds of SNH.

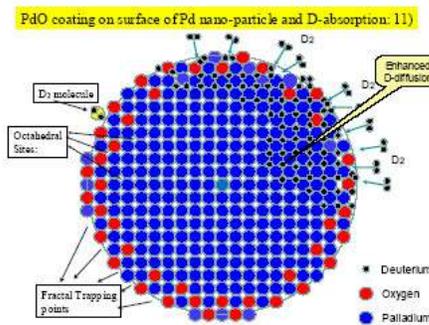


Figure 15. Trapped deuterons diffuse inside with enhanced speed by “D-pressure” from backside.

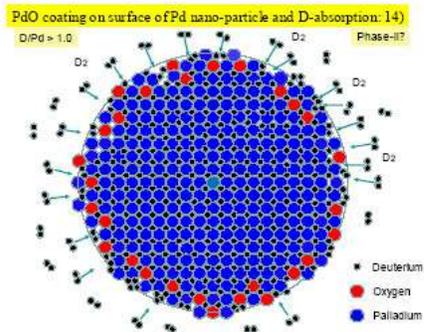


Figure 16. Feature at the end of Phase-I, full D(H)-loading in O-sites plus surface D-clusters component.

we need to wait about 20 min to get saturated (real) power level. However, if we have rapid heat release, as supposed as some surface nuclear reaction, we may have a local peak of heat–power evolution, as shown by simulation in Fig. 11.

Actually observed example of heat responses by H-charging, namely “pure” chemical heat–power evolution curve and energy per an H sorption (adsorption/absorption), η value (13), is shown in Fig. 12, where data by D-charging are drastically different. These runs after the forced oxidization realized significant recovery of heat–power levels and D(H)-loading ratios, in reference to the virgin runs. Heat evolution just after the D-charging has a peak, (see arrows) while that by the H-charging slowly rises without a “corresponding peak”, which is considered (speculated) due to the surface 4D/TSC fusion as shown in Fig. 5. The secondly interesting point is that the D-charging gave more than twice longer-lasting (about 10 hours) heat power than that (about 3 hours) of the H-charging. Evolutions of time-dependent η values are compared between the D-gas charging and the H-gas charging to show “drastic isotopic effect”. Such a big “isotopic effect” is difficult to be explained by usual chemical (electrons-interaction) effects, and needed to explore

Potential form of hydrogen adsorption and absorption near surface

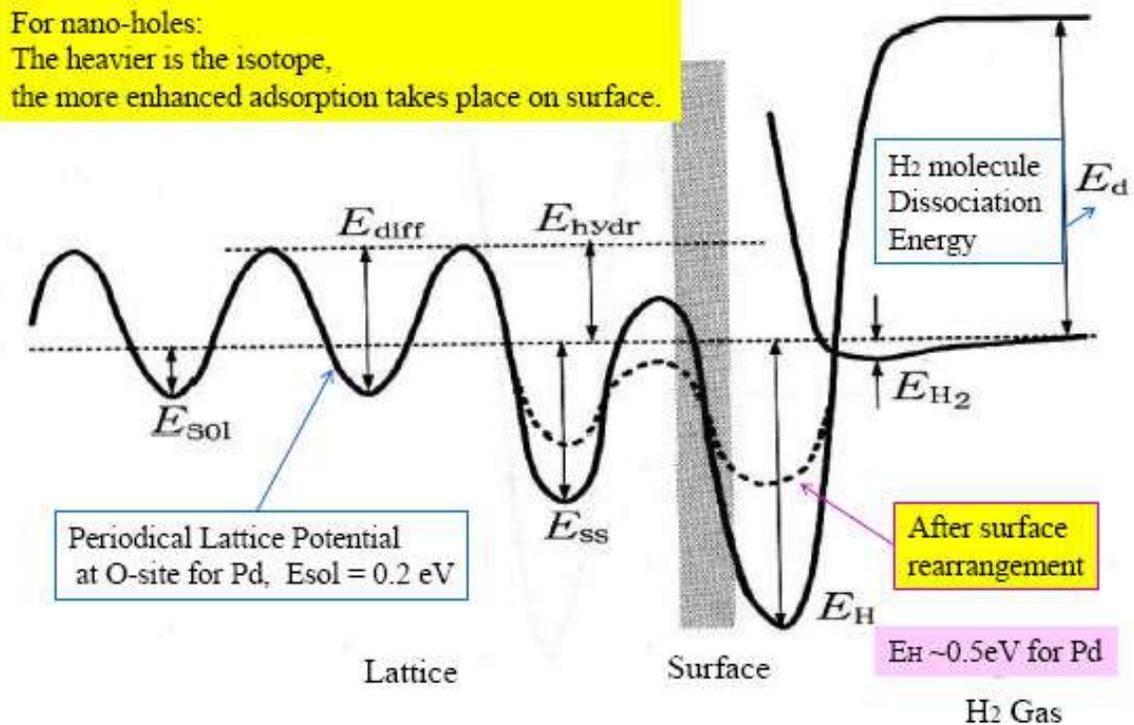


Figure 17. Surface and inner D(H)-trapping potential for a bulk-character Pd metal [9].

some “nuclear origin”. Ratio of η values ($\eta\text{-D}/\eta\text{-H}$) are very large (more than 10) in the beginning few minutes after D(H)-charging. Such a big isotopic effect infers the need of explanation by a rapid heat release mechanism of some nuclear reaction as 4D/TSC fusion on surface.

Now we come back to follow the simulation after Fig. 8 for D(H)-absorption into Pd nano-particle. Double D₂ trapping in an SNH does not always make a TSC, but does make reaction with near-by oxygen to form D₂O molecules and extended SNH as shown in Fig. 13. Through the extended SNH, deuterons trapped diffuse to inner O-sites as shown in Fig. 14.

In Fig. 15, we draw an image of D-diffusion enhanced by pressure of incoming deuterons from behind through the extended SNH. Finally, at the end of Phase-I, Pd nano-particle is fully loaded (PdD, $x = 1.0$), but we have additional trapped deuterons on surface (in SNHs) and apparent x -value becomes more than unity, typically as we can roughly calculate from the drawing, $x = 1.1\text{--}1.5$ which agrees well with observed Phase-I D(H)/Pd ratios [1,2,7]. This feature is drawn in Fig. 16.

4. A Phenomenological Model in Phase-II

As shown in Fig. 16, PdD local lattice may be formed inside a Pd nano-particle in Phase-II. We have in addition trapped deuterons (possibly making D-clusters) on surface SNH. For a known characteristics of bulk Pd metal for D(H) absorption, we have potential forms as shown in Fig. 17 for the surface adsorption and trapping (absorption) in inner lattice O-sites [9].

The surface adsorption energy (depth of potential) is 0.5 eV and lattice absorption energy is 0.2 eV, as evaluated in a standard text book [9]. Therefore heat release level for a bulk Pd sample is around 0.2 eV per D(H)-absorption, since number of trapped deuterons on surface is negligible. In contrast, observed specific absorption energy by PZ (and PNZ) samples are very large, namely around 2.0 eV per D(H), about 10-fold of the bulk value (0.2 eV). To explain observed anomalous values of D(H) loading under as “rapid loading process as observed” and such high specific “chemical” heat-energy level, we need to propose some new mechanism. We speculate and model that surface adsorption potential becomes very deep as 1.5–1.8 eV for a nano-Pd particle and local periodical Bloch potential should be in “shrunk state as shown in right figure of Fig. 18. Here anomalously high chemical heat releases are considered both for D-gas and H-gas charging procedures.

Existence of PdO surface “barrier” and formed SNHs would make very deep D(H) adsorption potential on surface (1.5–1.8 eV deep, speculated by our experiments [9]), which is categorized as an *collective mesoscopic potential well*

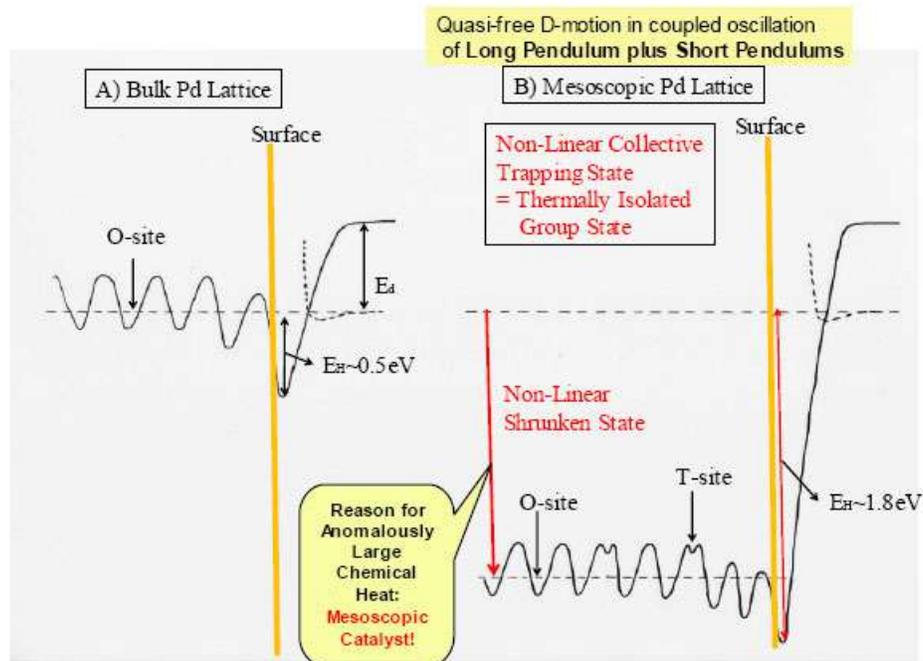


Figure 18. Collective shrunken state potential of Pd nano-particle for D(H) trapping, right figure, compared with a bulk metal trapping potential, left figure.

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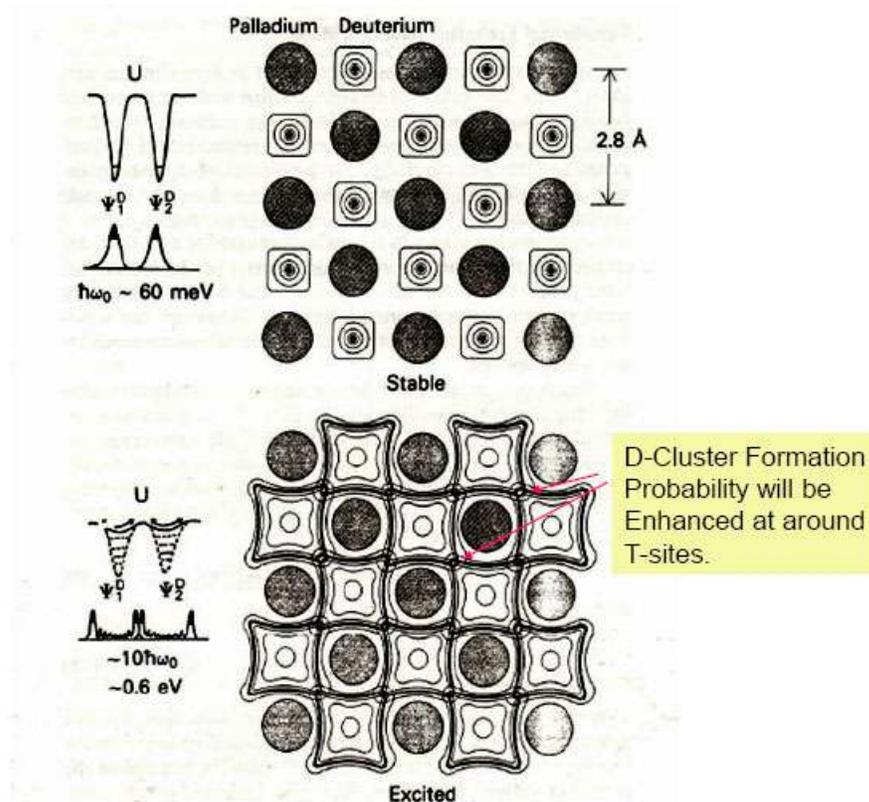


Figure 19. Quasi-free motion of deuterons inside CMPW potential enhances TSC formation around T-sites of local PdD lattice of Pd nano-particle which has formed a collective mesoscopic potential well (CMPW).

(CMPW, or a global potential for a nano-particle). Inside a CMPW confinement, three-dimensional PdD lattice (Bloch) potential exists as local fine structure in “shrunk state”.

The CMPW potential induces a QM non-harmonic oscillation, and the local Bloch potential induces a QM harmonic oscillation (wave function becomes a Hermite function, of which ground state is Gaussian and highly excited state has a U-shape wave-function). Two QM oscillations combine non-linearly [14] to make D-motion in a nano-Pd particle highly free (quasi-free) under the three dimensional constraint of PdD Bloch structure. This quasi-free motion of deuterons inside a mesoscopic nano-particle enhances very much probability of TSC (tetrahedral symmetric condensate [3–5]) formation as we give an image of QM wave superposition in Fig. 19 [10].

Generation of CMPW will basically make a Pd nano-particle working as “mesoscopic catalyst” which realizes very large D(H)-loading ratios and anomalously large chemical heat releases both for H-gas and D-gas loading. If anomalously large heat observed for H-gas run is by this process, some endothermic (slow heat sink) process should exist before we made de-sorption runs for which we observed [13] only about 1/10 levels of heat absorption, compared with released heat levels for the sorption runs, both for D- and H-absorbed samples.

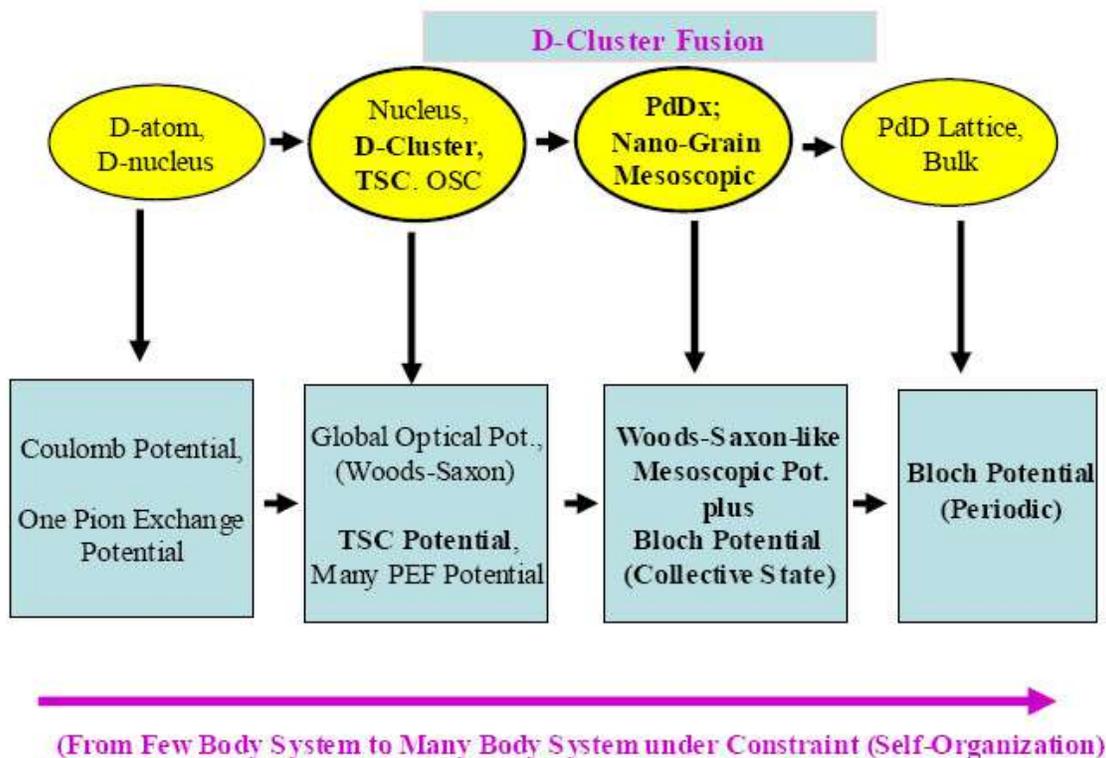


Figure 20. Speculation of useful potential forms to be applied for QM calculations to estimate fusion reaction rates from a simple system to many body, mesoscopic and bulk condensed matter systems.

In Fig. 20, we show a flow-chart drawing of considered useful potential forms in Coulombic (electro-magnetic) and nuclear strong interaction, starting from a simple system of atom and two nucleons to more complex systems as D-cluster, mesoscopic nano-particle and bulk material. By defining useful potentials for interactions in every step, we can extend quantum-mechanical (QM) analysis by using many body Schroedinger equations (or dynamic equations as QM-Langevin equation [3–5]). Fusion reactions in microscopic random systems (gas and plasma) can be treated by using a simple Coulombic potential and a one pion exchange potential (OPEP) [11]. For D-cluster systems, we can apply special trapping potential-forms for Coulombic interactions based on Platonic symmetry (orthogonal coupling) between electron wave functions and deuteron wave functions. For multi-body strong interactions of 4D, 6D, 8D, etc. cluster fusion reactions, we can use an empirical formula of PEF (pion exchange force) [12].

The specific nature of condensed matter is of constraint (ordering or self-organization) motion of particles with lattice regularity or surface fractals. A global shrunken state potential well for trapping D(H) atoms in deep hole will realize a non-linear oscillation mode coupled with harmonic oscillation in PdD lattice, which makes deuteron motion inside a nano-particle quasi-free under ordered constraint to induce enhanced cluster (TSC) formation probability. A bulk Bloch potential (periodical) is useful for established states of D(H) absorption. We speculate that D-cluster fusion is induced in the states of D-cluster and D-mesoscopic systems.

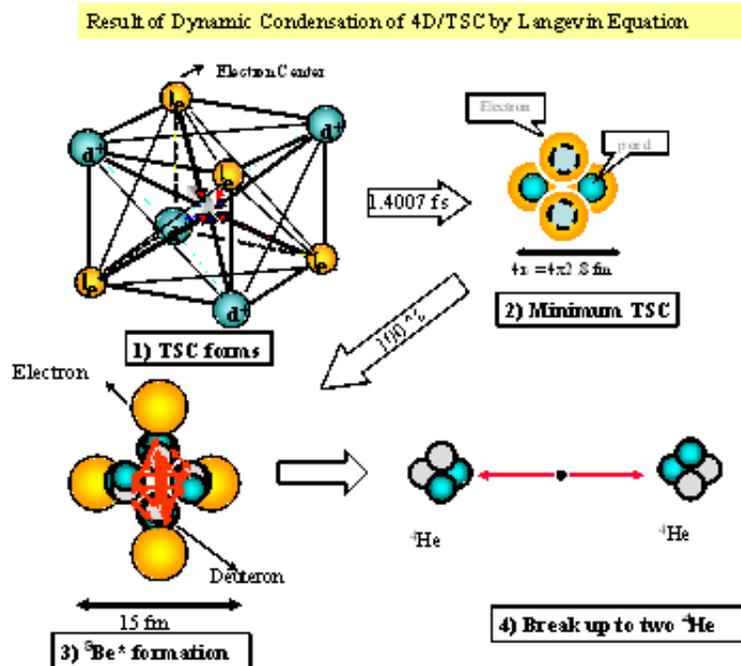


Figure 21. Typical four steps for TSC condensation and simultaneous 4d fusion.

We copy lastly typical four steps of TSC motion and 4d fusion in Fig. 21.

5. Concluding Remarks

- (1) With a PdO surface layer, sub-nano-holes (SNH) are formed in the beginning of D(H)-gas charging. This SNH would become a seed of “anomalous phenomena” in D(H)-loaded Pd nano-particle samples.
- (2) PdO layer also helps to make up a global deep mesoscopic trapping potential well which is a shrunken state of local PdD lattice Bloch potentials inside a nano-particle.
- (3) In SNHs, formation of 4D/TSC is largely enhanced in the Phase-I interval of D(H)-gas loading. 4D fusion thus induced may be a main nuclear component of heat release in Phase-I.
- (4) Within a global mesoscopic potential with local Bloch potentials, quasi-free D-motion is induced by non-linearly coupled oscillations for the global QM state (long pendulum) and a local harmonic oscillator (short pendulum). Anomalous heat by D-charging in Phase-II may be by 4D/TSC fusion of this process.
- (5) Generation of CMPW will basically make a Pd nano-particle working as “mesoscopic catalyst” which realizes very large D(H)-loading ratios and anomalously large chemical heat releases both for H-gas and D-gas loading.

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