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.

Keywords: Pd nano-particle, D-gas loading, Role of PdO, Sub-nano dip, Over-full D/Pd loading, Anomalous heat, Phenomenological model, Collective mesoscopic potential, Mesoscopic catalyst

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.

Two new findings were reported there: 1) Forced oxidation of used Pd/ZrO_2 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 to 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 theory³⁻⁵, 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;

- 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.
- 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 (D₂O or H₂O) 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 PdDx in a short time of the Phase-I process.
- 4) Formation of D-clusters, such as predicted by the TSC theory³⁻⁵ 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-PdDx system may be quasi-free to enhance transient D-cluster (4D/TSC³⁻⁵) 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 1.5eV deep) well-type collective potential and the other (shorter pendulum) by the lattice-type periodical three dimensional Bloch potential (about 0.22eV 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 "*mesoscopic 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, 10^{15} 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 Phase-I and Phase-II & 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 paper⁶, 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**.

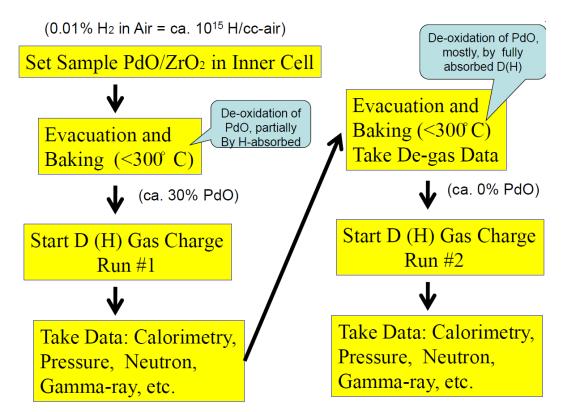


Fig.1: Experimental procedure of D(H)-gas charging to Pd/ZrO2 powder samples and reduction of PdO

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

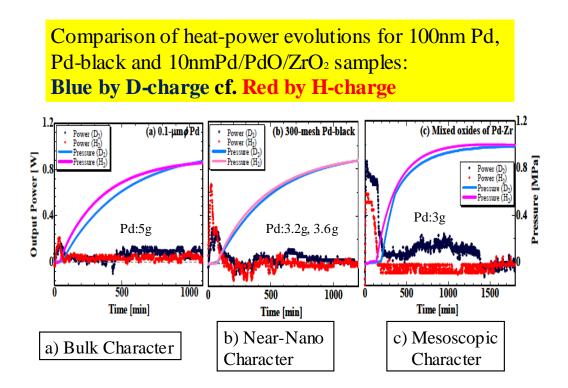


Fig.2: Comparison of typical heat evolution data for PP (0.1 micron diam. Pd powder), PB (300 mesh Pd-black powder) and PZ(10nm-Pd-nano-powder dispersed in ZrO₂ flakes)

3. 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 Kitamura PLA paper⁶. We show a modified figure for helping this purpose in **Fig.3**.

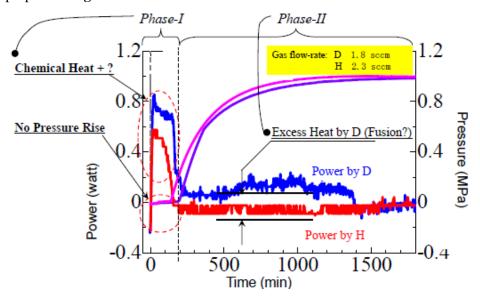


Fig.3: definition of the phase-I and phase-II intervals for the D(H)-gas loading experiment^{6,7}.

From our analysis of forced-oxidization experiments⁷, 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 ICCF16 paper, and also we reported^{7,17} at ACSNET2010), 2-10nm 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.

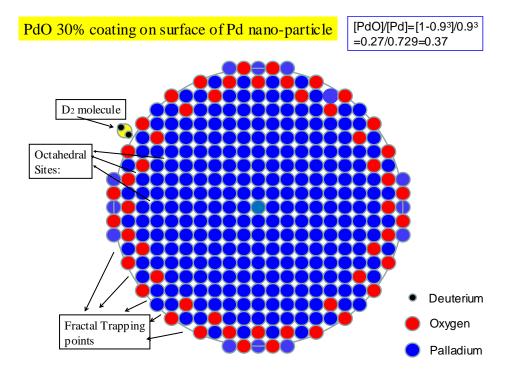


Fig.4: An atomic scale cross sectional image of PdO-layer coated (30%, y=0.3) Pd nano-particle (5 nm diameter); here we used relative sizes of ionic radii and D_2 molecule, and fractal surface structure is supposed to make a role enhancing deeper adsorption potential for D(H)-gas

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 D_2O (H₂O) by incoming $D_2(H_2)$ gas molecule to combine (de-oxidize PdO) with oxygen. Formed D_2O (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 D_2 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 stage, and we need a further deependent coupled Schroedinger 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 $(23.8 \text{MeV}/^4\text{He})^{3-5}$. Image of such a process is illustrated in **Figs.5-8**. After such an event of D₂ 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".

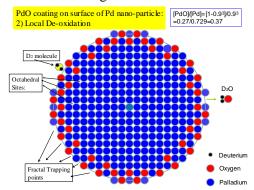


Fig.5: D₂O molecule going out

D2 m

Octahed

Sites

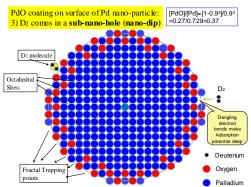
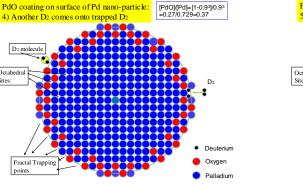


Fig.6: SNH traps D₂ incoming



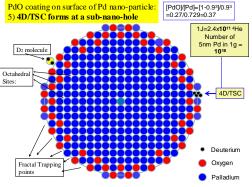


Fig.7: Eventual double D₂ trapping

Fig.8: Eventual 4D/TSC fusion on surface

As shown in an insert (right top) of Fig.7, 1g of Pd nano-powder contains 5nm-diam. nano-partilces of 10^{18} (on the order). Since 10^{11} 4d/TSC fusion events corresponds to one joule, 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 one gram 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 al⁷ 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 system⁷ 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.

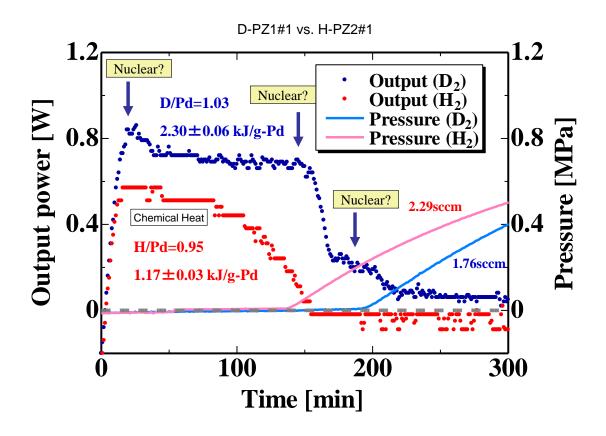


Fig.9 Expanded view of heat-power evolution data by Kitamura et al⁷ for PZ samples

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 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.

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 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.

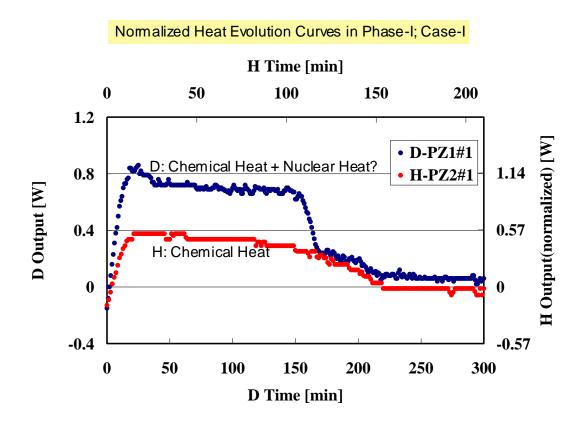


Fig.10: Normalized heat evolution curves in Phase-I for PZ-1(2)#1 runs⁷

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 phase²). 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 to 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), we need to wait about 20 minutes 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**.

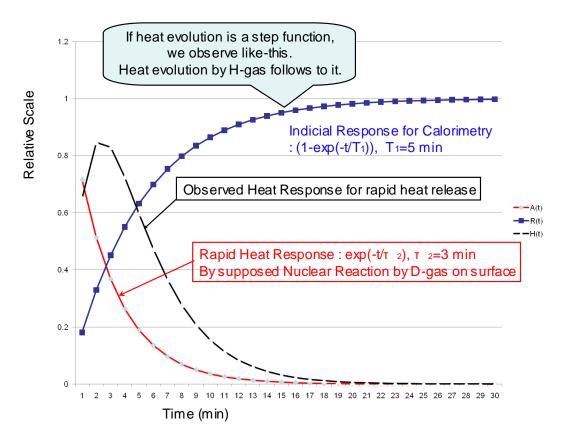
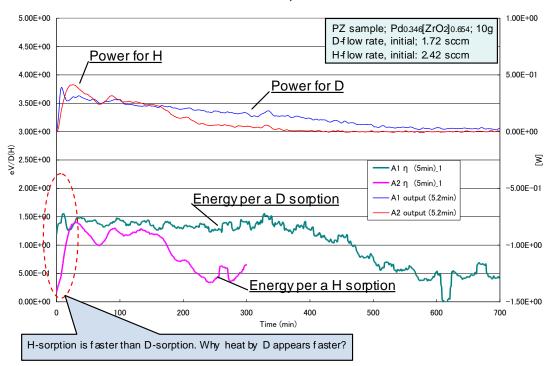


Fig.11: Observed "rapid" heat response is broadened by indicial response of calorimetry, assuming a single exponential "nuclear heat evolution by D-absorption in Phase-I. Such a "nuclear response" may be a superposed component in observed heat evolution data for D-gas, in comparison with rather smooth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

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.6. 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 some "nuclear origin". Ratio of η values (η -D/ η -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.



D-PZ11#3 vs. H-PZ12#3 ; After Forced Oxidization

Fig.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.

Now we come back to follow the simulation after Fig.8 for D(H)-absorption into Pd nano-particle. Double D_2 trapping in a SNH does not always make a TSC, but does make reaction with near-by oxygen to form D_2O 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**.

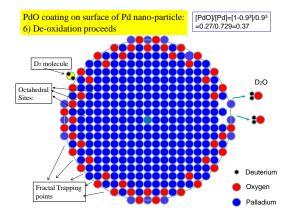


Fig.13: Extended sub-nano dips by D₂O formation

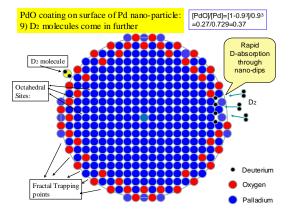


Fig.14: Incoming D_2 molecules are trapped one after another, by dangling bonds of SNH

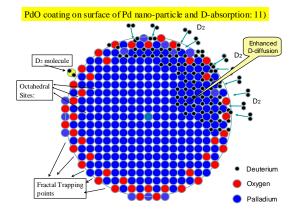


Fig.15: Trapped deuterons diffuse inside with enhanced speed by "D-pressure" from backside

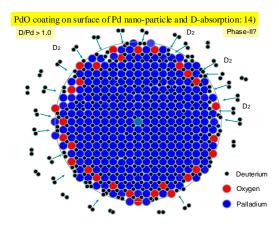


Fig.16: Feature at the end of Phase-I, full D(H)-loading in O-sites plus surface D-clusters component

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=1.1-1.5 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-sites⁹.

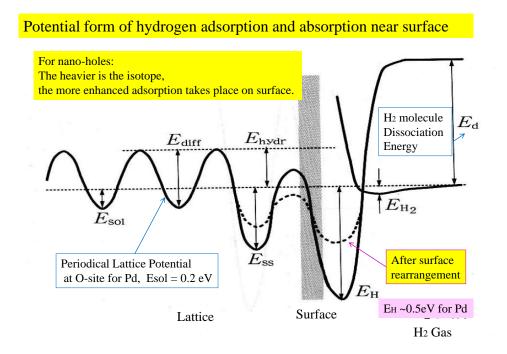


Fig.17: Surface and inner D(H)-trapping potential for a bulk-character Pd metal⁹

The surface adsorption energy (depth of potential) is 0.5eV and lattice absorption energy is 0.2eV, as evaluated in a standard text book⁹. Therefore heat release level for a bulk Pd sample is around 0.2eV 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 "shrunken 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.

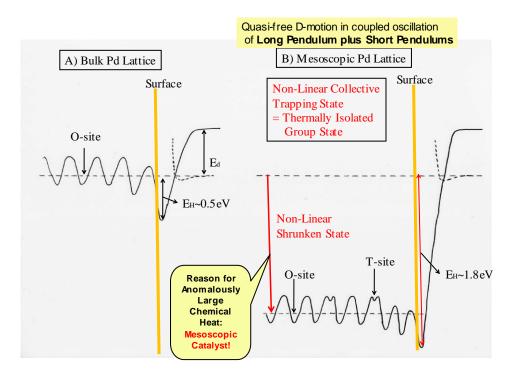


Fig.18: Collective shrunken state potential of Pd nano-particle for D(H) trapping, right figure, compared with a bulk metal trapping potential, left figure.

Existence of PdO surface "barrier" and formed SNHs would make very deep D(H) adsorption potential on surface (1.5-1.8eV deep, speculated by our experiments¹³), which is categorized as an *collective mesoscopic potential well* (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 "shrunken 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-linearly¹⁴ 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 condensate³⁻⁵) 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 observed¹³ only about 1/10 levels of heat absorption, compared with released

heat levels for the sorption runs, both for D- and H-absorbed samples.

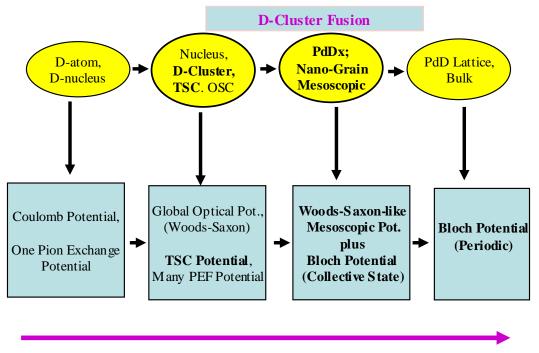
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Fig.19: Quasi-free motion of deuterons inside CMPW potential enhances TSC formation around T-sites of local PdD lattice of Pd nano-particle which has formed a collective mesoscopic potential well (CMPW).

Excited

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 equation³⁻⁵). 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.



(From Few Body System to Many Body System under Constraint (Self-Organization)

Fig.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

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

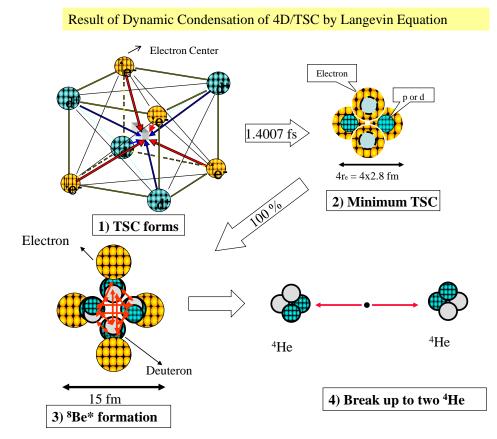


Fig.21: Typical four steps for TSC condensation and simultaneous 4d fusion

5. Concluding Remarks

- 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.
- 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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