

Research Article

Development of a High Temperature Hybrid CMNS Reactor^{†,‡,§}

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Abstract

We presents some improvements on the reactor presented at ICCF14 (Washington DC, August 2008): use of long-thin Pd wires with nano-coated surfaces by multi-layers of several elements, loading with D₂ at pressure <10 bar; wires temperatures >500°C; Stainless Steel (SS) reactor wall temperature <100°C; current density along Pd up to 45 kA/cm²; voltage drop along the Pd wire up to 70 V. Mainly, the Pd wire temperature was increased up to 750°C and was improved the temperature detection of anomalous excess heat, if any, using a SS shielded type-K thermocouple: it was put inside a small Cu tube, used as thermal equalizer, where, at the outer surface, both the “active” Pd wire and the “reference” Pt were twisted. The overall results were in agreement with that obtained in 2008 experiments and they confirm the positive effect of high temperatures in increasing the amount of anomalous energy gain. In both the experiments the fast and simple isoperibolic calorimetry was used. Main gas adopted were: He and He (60%)–Ar(40%) mixture, both for calibration purposes; D₂ and D₂(60%)–Ar(40%) as potentially active gas.

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1. Introduction

In the framework of Condensed Matter Nuclear Science (CMNS), starting from 2004, we developed and used a specific procedure that sums together different methods proved to be beneficial to induce “anomalous heat release” in Palladium–Deuterium (Pd–D) systems. Recently, we started experiments also with Nickel–Hydrogen (Ni–H) systems.

Such procedure, that we called “hybrid”, couples the use of:

- a Nano-particles.
- b Multilayer nano-coating onto D (or H) absorbing materials, like Pd.
- c Flowing of large current into materials of b), shaped as thin and long wires, in order to exploit the *Preparata Effect* (voltage drop along wire up to 100 V/m in our experimental set up)..

We remind that the first Researcher, in the field of CMNS, who used concentrated Pd nano-particles, to increase the D/Pd ratio, was Prof. *Yoshiaki Arata* at Osaka University since 1993 [1]; the first one who developed multiple nano-layers of Pd–CaO was Dr. *Yasuhiro Iwamura* at Mitsubishi Heavy Industries, in Yokohama in 1999 [2]. Both used, de-facto, Deuterium gas loading.

As we have been experiencing since 1993, the so-called “anomalous effects” (thermal anomalies and/or some, few, nuclear particles generation) are detectable only when the system is under some *non-equilibrium condition*, spontaneous or forced. This seems to be a *necessary condition* to achieve some macroscopic evidence of the reactions but it’s absolutely not a sufficient one.

It has to be guaranteed, at the same time, a large amount of H or D inside the lattice of the active material or, at least, on its surface. It is therefore necessary to “flow” as much as possible gas (or, better to say, D^+) through the Pd (or Ni) lattice at a rate as fast as possible. It’s not important the direction of the flux: as consequence, fast loading and de-loading cycles have similar effects. Our forced loading procedure takes about 5–10 s using Pd (reaching a D/Pd ratio of around 0.75, obtained by R/R₀ measurement); putting enough gas in the lattice and the faster is the loading, the larger will be the results of anomalous heat production.

The specificity of excess heat, flowing the Deuterium *in any direction* (at 383°C, 13 atm), was first discovered at NASA (*Gustav C. Fralick* and others) on 1989. Because they were concentrated at that time only on neutron emission, they didn’t make deeper studies about, but just reported the effect in the framework of a Technical Memorandum [3] among others, very poorly diffused worldwide.

2. The Hybrid Procedure

In order to achieve both a high D/Pd ratio (or H/Ni ratio) and a “fast flowing” gas in the lattice, as stated above, we developed a hybrid procedure mainly based on the following effects:

(1) Electromigration (EM)

The EM effect was discovered and developed around 1929, in Germany, by several scientists. We started to use it feeding thin Pd wires (diameter $\Phi = 50 \mu\text{m}$, length = 50–100 cm) with large DC (i.e. steady state) current density (up to 40–50 kA/cm²) under electrolytic conditions and, in the more recent years, under gas loading. Previously (in electrolytic regimes), we even improved it since 1996 [4], introducing pulsed regimes of few μs of duration and peak current up to 150 A. The pulsing procedure allowed reaching a peak current density through the wires of up to 300 kA/cm² without largely increasing the mean power applied. Moreover, using proper repetition rates (5–33 kHz), the self de-loading of D₂ between pulses was kept under control [4].

The disadvantages of the pulsing procedure were: very complicate circuitry; complex evaluation of excess heat; quite frequent catastrophic failure of the electronics due to extremely large values of peak power.

(2) Large voltage drop



Figure 1. Wires braid inside the thermal insulation.

The large EM currents, the high resistivity of deuterated Pd and the high temperatures (around 500°C or higher in our most recent gas experiments) induce 50–100 V voltage drop along the Pd wire. According to *Giuliano Preparata's* (Milan University, 1995) theory [5], large voltage drops along Pd wires, together with other specific experimental conditions, have the peculiarity to increase the D/Pd ratio because of “coherence effects”. Preparata studied only electrolytic, not pressurized cells (with Pt anode and LiOD as electrolyte): maximum temperature of cathode Pd wire was $\ll 100^\circ\text{C}$. According to the theory, higher temperatures facilitate the growing of Pd–D γ phase (which formation is an endothermic process, while the formation of α and β phases are exothermic); this leads to an enhanced state of reaction due to a higher D/Pd loading.

(3) Use of Pd-based nano-particles

The effects of micro and nano-particles in CMNS experiments, from the point of view of increased Deuterium loading and generation of anomalous effects, were pioneered by *Yoshiaki Arata* (Osaka University, 1993) [1]. More recently (since 2005) Arata got anomalous heat in a fully reproducible way, for time as long as two days, using an innovative Pd–ZrO₂ system and D₂ gas pressurised at up to 60 atm [6].

Arata's experiments were independently reconfirmed (2008) by *Akito Takahashi* and *Akira Kitamura* group (Technova Inc., Kobe University collaboration, Japan) [7] even using “commercial” material: produced by Santoku Corporation (Kobe), a Company (since 1935) expert in Precious Metals and Rare Earths production/purification. This experiment was a milestone: it overcame the historical problem of *transferred irreproducibility* in CMNS experiments.

(4) Use of multi-layers

Since 2000 *Yasuhiro Iwamura* (Mitsubishi Heavy Industries, Yokohama, Japan) developed a suitable procedure to deposit two substances (CaO and Pd) in nano-metric layers on a bulk Pd plate [2].

The aim of the experiment was to demonstrate the transmutation of selected elements deposited in the outermost side of the multilayer into new ones: recently, from Sr, Cs and Ba they got respectively Mo, Pr and Sm. The effect is induced by the flowing of D₂ gas in a slightly pressurised chamber (max 2 atm) and by mild temperatures (up to 80°C). Iwamura (and Collaborators) used advanced ion beam technology for the multi-layer structure construction.

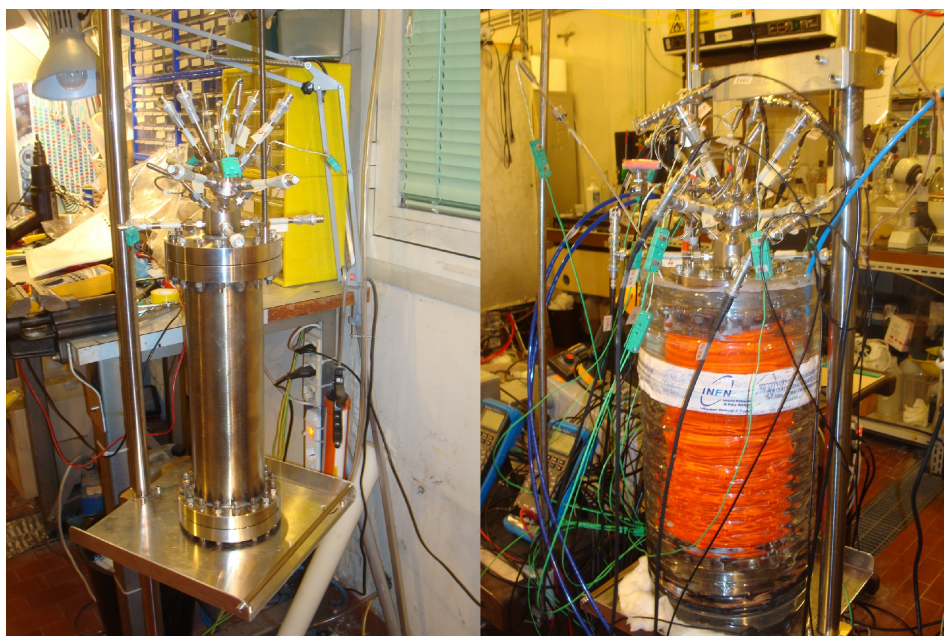


Figure 2. The apparatus mounted without and with the water bath.

(5) *Fractal nano-layer*

Since 1998 [8], we experienced that in electrolytic environment, using a mildly acid solution with innovative electrolytes (like salts of Ca, Sr, and Ba) at very low concentration (<0.1 mM), the Pd wire (typical diameter $100\mu\text{m}$) was sometimes covered by a sub-micrometric layer of several elements, including Pd itself, especially after several cathodic–anodic cycles.

After this spontaneous development of such porous and very thin layer (like a sponge), the characteristics of D_2 absorption changed dramatically and the loading time was reduced of several order of magnitude (from hours to minutes) especially at low current density of electrolysis (about $10\text{--}20$ mA/cm²). Moreover, we observed that Pt deposition (present at anode) was usually deleterious to Deuterium absorption.

We observed correlations between the increasing of the amount of thermal effects and the arising of compositional anomalies (detected by SEM-EDAX and, later, by ICP-MS). We realised that the cause of anomalies arose from such “fractal/sponge” layers. We published several reports about such observations although the situation was quite frustrating: any attempt to build the layers on demand failed. In short words, the system was very delicate and easy to self-destruction.

Because of the uncontrolled growth of this spontaneous layer and its instability, we moved (since about 2004, as ancillary experiments at the beginning) from liquid electrolysis to gas environment where the fractal layer is more stable and can be used with larger success.

2.1. Fractal nano-layer construction

Using a gas environment avoids uncontrolled self-dissolution of the sponge layer due to electrolytes and large local pH changes due to cathodic and short time (if needed) anodic electrolytic regimes at the Pd surface.

We prepared the sponge material over the wire in an almost controlled way before inserting it in the reactor. Anyway, we point out that further experimental work is necessary to improve the quality/reproducibility of the coating.

The wire is made by an active support like Pd (or recently Ni) and it is coated with nano-materials (some used as anti-sintering agents) containing also Pd. The nominal dimension of the main nano-material is 6–9 nm.

The active support is a thin wire, $\Phi = 50 \mu\text{m}$ and length = 60–90 cm, coated by a total number of about 50 layers: the apparent increase of thickness of the wire is 1–2 μm . The procedure involves several high temperature cycling between 20 and 900°C, using Joule heating in air with several specific patterns in the timing of both the steady-state regime and transient one: we adopted current-controlled power supply with low values of output capacitors to avoid blowing of wire.

2.2. Characteristics of nano-coated Pd wires

As general behaviour, the loading time is a nice and simple indication of the coating quality. Usually it takes from 6 to 30 s to reach $R/R_0 = 2$, i.e. $D/Pd = 0.75$ using H_2 or D_2 gas at a pressure of 6.5 atm. The shorter is the loading time the better is the coating and the longer will be the deloading time. A couple of hours can be necessary for a full deloading of the loaded wire by putting the system under dynamic vacuum (usually 10^{-3} – 10^{-4} atm) at 350–400°C.

This is an important effect that we observed since 2002, even in electrolytic experiments. We named it the “Diode

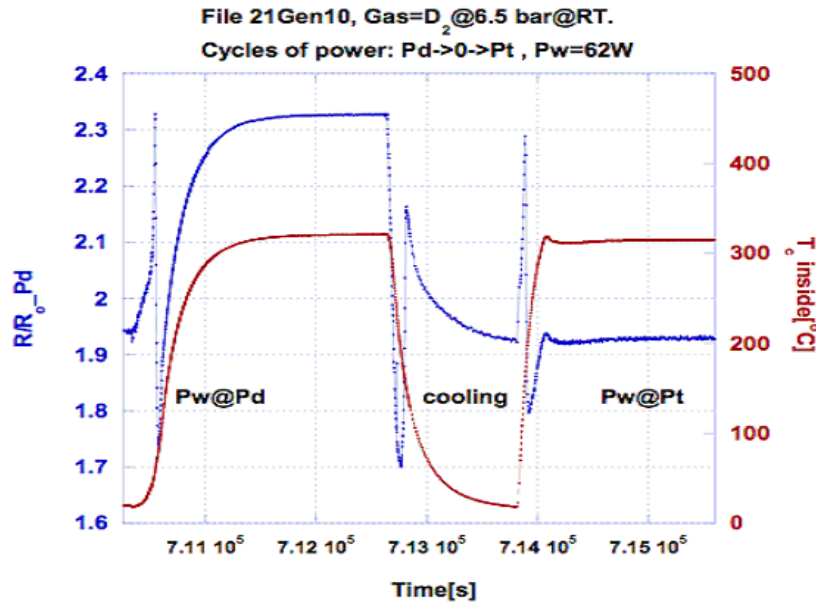


Figure 3. Behaviour of R/R_0 of Pd, changing the Power (Pw) from Pd to Pt wire. At the same temperature (red line) inside the Cu tube, the value of R/R_0 (blue line) is higher if the Pd wire is heated directly (Pw@Pd).

Effect”: the H_2 or D_2 gas can flow-in very easily and rapidly and on the contrary the flowing-out is very difficult.

3. Description of the New Experimental Set-up

The new experimental set-up is a development of the previous apparatus, presented at ICCF14 (August 2008, Washington D.C.) and at ICCF15 (October 2009, Rome). Basically, they are similar each other because the power measurements (and calibrations) are based on the comparison of results between a normal Pt wire, assumed to be inert under D_2 atmosphere, and an “active” Pd wire, i.e. a Pd wire with the above mentioned coating.

These wires are put singly inside an insulating sheath made of glass or quartz fiber and then twisted together to be very close each other. They are thermally insulated, from the stainless steel (SS) internal wall, by a high temperature (fibrous ceramic) wool, named SW607HT, (Fig. 1).

The procedure is based on feeding several different electric powers alternatively to Pd and Pt wires, before in “inert” gas (He) and later in “active” gas (D_2 , H_2). Equilibrium temperatures were recorded versus the different powers applied. The maximum internal temperature reached by the SS reactor wall is $110^\circ C$, avoiding the sulphur degassing problem from the wall, as pointed out by Prof. *Tatsumi Hioki* (Toyota Research Centre, Aichi-Japan) since 2007 at ICCF13 [9].

Main improvements were:

- The braids, made by the quartz sheaths allocating the Pd and Pt wires, were rolled up around a copper (3N purity) tube (4–6 mm inner-outer diameter; length 24 cm) in order to homogenize the temperatures. In the inner side of the tube, in the middle, was put a, high resolution, type K thermocouple (Tc) with insulated body (SS 316N against penetrating of the gas inside the thermocouple lattice; MgO as electrical insulator). In this way we realised a sort of furnace (the Cu is an excellent IR reflector), which is heated, alternatively, by the Pd and Pt wires.
- A second braid made of a couple of wires and a copper tube was prepared, but in this case both wires were made of Pt instead of Pd and Pt. This second set of wires was used as in situ and on-line cross-comparison of the behaviours of the reactor when changing the atmosphere (4He , D_2 , D_2 -Ar mixtures, Ar, air, and vacuum).
- Both couples of braids were individually thermally insulated and put in the same pressurised SS cell. Finally the cell was put in a large water bath continuously and largely cooled (Fig. 2).

4. Methodology of Measurements

The procedure foresaw first of all a calibration test, made by using pure 4He at 6.5 atm, and different wire temperatures due to proper powers applied along it. After that, the experiment was replicated in D_2 , Ar and air atmosphere and in vacuum. Using different atmosphere composition, it was possible not only to evaluate the effect of different gas on the anomalous heat release, if present, but also to change the regime temperature, due to the different thermal conductivity.

Each session included a series of runs; in each run an input power was selected and maintained until a thermally stationary regime was reached. At that time the key parameters (input power, temperature, R/R_0) were measured and labelled as *equilibrium values* for further analysis.

An additional session was performed in a mixture of D_2 (4.62 bar) and Ar (1.68 bar) to study the effect of a higher wire temperature due to the approximately seven times lower thermal conductivity of pure Argon in respect to pure Deuterium. The rationale for this test was that we thought that higher temperatures could be helpful for these specific reactions and that could compensate the lower pressure of active gas or may even improve the overall amount of anomalous excess heat production. A percentage of 24% of Ar in D_2 reduces the D_2 gas thermal conductivity of about 56%, i.e. from 151 to 96 $mW \cdot m^{-1} \cdot K^{-1}$ at $95^\circ C$.

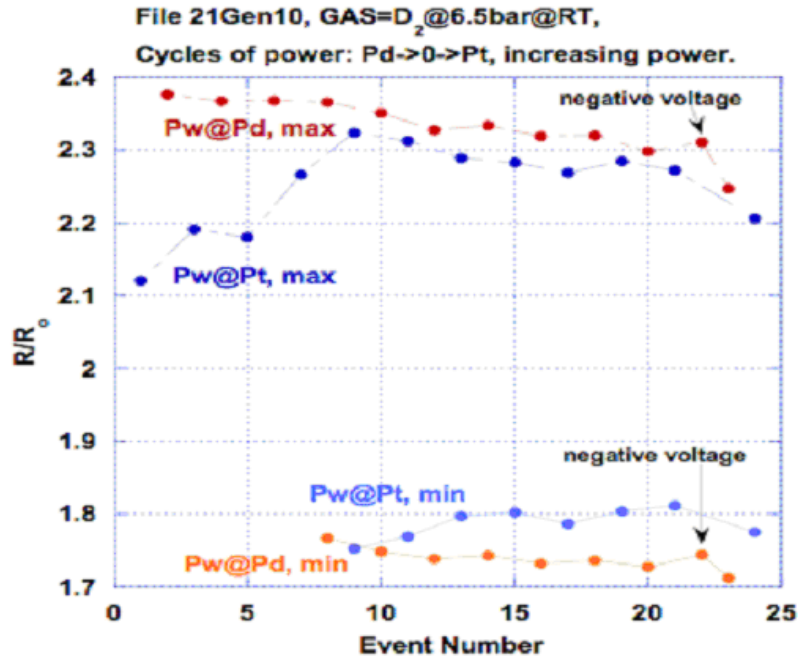


Figure 4. By feeding directly the Pd wire (Pw@Pd) the R/R_0 reaches both higher maximum and lower minimum in respect to indirect heating (Pw@Pt).

Eventually the test gave a positive answer to our expectations and the increase of temperature improved largely the anomalous heat production, overcoming the D_2 pressure reduction.

5. Main Results

This chapter presents the main results obtained with our experimental system between January and February 2010. Also some theoretical interpretations are formulated, but we want to underline that this is not the main purpose of the paper, which is mainly addressed to make our experimental results available for who wants to build a valid explaining theory.

Figure 3 is related to an experiment in atmosphere of pure Deuterium (6.5 bar at room temperature), with an external input power of 62 W. We can identify three different phases: in the first one the power is given directly to the Pd wire (by Joule effect), in the second one there is not external power (to allow system to cool down), in the third one the power is provided to the Pt wire. The figure shows that, even if the final (stationary) temperature in the system is the same, the ratio R/R_0 in the Pd wire is much higher in the first phase, leading to the conclusion that the presence of an electrical current plays a fundamental role in increasing the wire loading by Deuterium.

We consider this as a confirmation that the maximum R/R_0 of Palladium under direct heating (electro-migration) is always larger of any maximum that can be obtained with only high temperatures.

Some authors have interpreted this behaviour as an evidence of some kind of confinement of Deuterium inside Palladium due to the voltage drop along the thin and long Pd Wire (according to the Preparata Model).

Prof. Yeong E. Kim (Purdue University, USA) thinks this is as an evidence of the *Bose-Einstein Condensation Nuclear Fusion* (BECNF, Naturwissenschaften, 2009); he actually mentioned our ICCF-14 experiments as the first experimental proof of such model. Prof. Francesco Premuda (Bologna University, Italy) in 1993 [11] firstly formulated the hypothesis that several of CMNS effects could be explained as a special case of Bose–Einstein Condensation (BEC). Also Akito Takahashi (now at Technova Inc.) thinks BEC is a thoughtful concept to start to study the anomalous CMNS effects, and his *TSC* (Tetrahedral Symmetric Condensate) model is a special case of dynamic (transient) BEC [private communications during 239^oACS, March 2010].

Figure 4 shows the minimum and maximum values of R/R_0 achieved in a series of tests during which different amounts of power were provided alternatively to the Pd (Pw@Pd) and to the Pt (Pw@Pt) wires.

A series of observations are drawn in the following:

- I. The minimum values of R/R_0 in Pd/D system are always lower using direct feeding of the Pd wire instead of indirect heating. Such an effect was predicted by Prof. Premuda as a proof of presence of “islands” of superconductivity in D/Pd system (supposing the system in plasma regime).
- II. Using the Palladium–Hydrogen system in similar condition and with both direct and indirect heating we observed that the maximum values of R/R_0 are very similar to the Pd–D ones, but the minimum values are larger. This effect can also be explained according to the Premuda model, in this case we can’t get islands of superconductivity because there are fermions (H) and not bosons (D).
- III. Putting a negative voltage polarization on the Pd wire, respect to the grounded Pt wire at a 1 mm distance, shows a measurable increase of both R/R_0 maximum values and anomalous heat production (about +12%) when compared to the same operating condition but using a positive voltage. Moreover, the temperatures detected inside the copper tube at maximum and minimum R/R_0 are remarkably higher. We observed this effect since 1996 in electrolytic systems and discussed it in various conferences, but the high voltage discharge problems forced us to quit this method to increase the gain. We are developing a new version of reactor (#4) dedicated to study more deeply such phenomenon. We may speculate that the changing, at the proper low frequency, of the polarization of the voltage could stimulate the movement of deuterium between the surface and the Palladium bulk in both directions enhancing the anomalous reactions.
- IV. We observed that even Platinum absorbed some Deuterium at the level of 1–2% (estimated by R/R_0 measurements) when there is large (20–40 kA/cm²) electro-migration.
- V. We observed that Palladium can absorb He at 1–2% level under very large electro-migration ($J = 40 - 50$ kA/cm²).
- VI. We observed that an addition of Argon reduces strongly the possibility of He absorption by both Pd and Pt.
- VII. We had confirmation that the high temperature is one of the key parameter influencing the amount of anomalous heat generated in D/Pd systems. After Deuterium absorption inside Pd lattice was performed, adding about 40% of Ar in the cell atmosphere rises the temperature that can be reached by the wires due to the lower thermal conductivity of Ar in respect to D₂. In this case we noticed an increased heat surplus made by the Pd wire in respect to the Pt one.

6. Conclusions

It was experimentally proved that, under specific conditions, it is possible to generate anomalous excess heat at macroscopic levels (several Watt or up to 400 W/g of Pd, expressed as excess-power/g).

According to our experiments, in the Pd–D system, the key parameter controlling the amount of anomalous heat generated is the “motion” of deuterium between Pd bulk and/or the nano-coating (i.e. at the surface). In other words,

it is necessary to maximize the amount of moving Deuterium and its speed. Such an effect, although discovered by us since 1993 and roughly discussed [12], is quite difficult to be obtained in practical experiments.

The method of coating with some specific nano-materials (including Pd) over a long and thin palladium wire, to make it active from the point of view of deuterium absorption, seems a reliable procedure, at least at laboratory level, to fulfil the request for reproducible experiments.

We experienced that high temperature (of the order of 300°C and over) is a pre-condition to get some macroscopic effect even under mild pressures (4–8 atm).

We experienced that high temperature by itself increases the anomalous heat release, especially above 700°C. Such an effect was by us predicted and experimentally found.

We experienced that the electro-migration, especially at high current density ($>20 \text{ kA/cm}^2$) improves in the whole the anomalous effects.

Even if this subject is not specifically addressed in this paper, we experienced also that the coating of nano-materials is a procedure that allows, in an easier way, the detection of the anomalous effects, thermal included.

The power density (up to 400 W/g of Pd) and time duration (experienced by us of over 1 week) of anomalous heat generated pushed us to think that the origin of such anomalous heat is out of any known chemical reaction. Remarkably, the anomalous heat increased over a number of ON–OFF cycles, probably because in this way the quantity of fractals at the surface were increased with further reduction of dimensionality. This is due to the well-known phenomenon of both *embrittlement* of Pd (and of several other metals and alloys) and large changing of dimensionality (see our report at ICCF15) due to Hydrogen/Deuterium loading de-loading cycles. Drawback of such (simple) procedure is the possibility of the wire breakage, especially if it is thin. For this reason we made some specific “developments” to reduce such catastrophic final result. Another weak point of Pd is that it changes his dimensions after several loading–deloading cycles. In short, in the case of wire geometry, the length decreases and wide increases.

Further work, and sophisticated/expensive instrumentation, is necessary to identify clearly the supposed non-chemical origin of the phenomenon: e.g. detection of ^4He as nuclear ash.

The positive effect of a transversal electric field at Pd surface (i.e. negative voltage) was reconfirmed and opens the way to an experimental set-up that, in principle, can boost the anomalous heat production without increasing the input power.

If all the still remaining doubts will be (hopefully soon) fully clarified, the phenomenon under study can be the starting point for a new source of energy for practical purposes (like water boilers and even engines). We recall that, in our specific experiments, we can operate at quite large temperatures (about 700°C, i.e. at high intrinsic Carnot efficiency) and that the increasing of the temperature generally lead to an increase of the anomalous effects. In other words, we are in the lucky situation, from technological point of views, of *positive feedback*.

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