Development of a High Temperature Hybrid CMNS Reactor: Status Report, at March 2010, of INFN-LNF (Italy) Experiments.

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INTRODUCTION

***** **S**ince 2005 we developed, at Frascati National Laboratory of National Institute of Nuclear Physics (Italy), a procedure that "collects" all together the advantages of methods that were proved (by other Scientist and ourselves) to be beneficial to induce "anomalous effects" due to the close interaction of **Hydrogen [H]** and its isotope **Deuterium [D]** (**Tritium** can't be tested at high concentrations because safety problems) with specific materials (mainly **Palladium, Pd**).

***** Moreover, we experienced that the ***anomalous effects*** happen at macroscopic level **only when** the system is under some ***non-equilibrium condition*** (spontaneous and/or forced). Such specific condition was discovered by us since 1993 and remarked continuously: during Conferences and/or by written reports. It was addressed both to specialist readers (by Science journals) and general people (by reports in magazine and/or news paper); even given talks to Politicians, Industrials, Students, membership of cultural organizations (like Lions or Rotary club).

***** Again, we recall that the non-equilibrium status is one of the necessary conditions but, by itself, doesn't guarantee the generation/detection of anomalous effects that we are looking for (i.e. thermal and/or nuclear): they are **need**, ***at the same time***, **large amounts** of H and or D **inside** the "**lattice**" of active material and/or at its **surface**.

***** The key point, that is the "integral" of our experimental, long lasting time (now exactly 21 years) work, is the following (in the case of D-Pd system):

It is necessary to "move" the Deuterium, at an *amount as large as possible*

and ***as fast as possible*, through the Pd lattice.**

It is not important the direction of the atomic flux:

loading or de-loading cycles gave similar effects.

Forced de-loading, intrinsically faster, gave larger values of anomalous heat.

Origin of new INFN-LNF procedure.

In short, in order to achieve the “fast moving” (at high D/Pd ratio), in situations as reproducible as possible, we developed a **hybrid** procedure based on the following effects:

a) **Electromigration** (EM), at quite large current density (longitudinal up to 45 kA/cm^2), along long (l=50-100cm) and thin (Φ=50μm) Pd wires (R=0.5 Ohm*cm). The EM effect in Pd-H system was developed, since 1929, in **Germany** by several **Scientist**. Starting from 1993 **we** largely **improved** the original EM procedure by introducing ***pulsed regimes*** (few μs duration, peak current up to 150A): it was possible to increase the peak current density through the wires (since 1995) from 50 kA/cm^2, in DC conditions, up to values as large as **300 kA/cm^2**, without increasing the mean power applied. Using proper repetition rates (5-33 kHz) the self de-loading of D_2, between pulses, was kept under control (PLA, 1996).

b) **Large voltage drops** (50-100 V) along the Pd wire. They are consequence of: large EM currents, high resistivity of deuterated Pd, high temperatures (like 500^{o}C or higher in our most recent gas experiments). According to **Giuliano Preparata**'s (Milan University-Italy, 1995) theory, large voltage drops along Pd wires, together with other specific experimental conditions, have the peculiarity to increase the D/Pd ratio because "coherence effects". Preparata studied only electrolytic cells and not pressurised (Pt anode, electrolyte LiOD): maximum temperature of cathode Pd wire <<100^{o}C.

c) Use of, Pd based, **nanoparticles**. The first Researcher that exploited in details the effect of nanoparticles, in CMNS experiments, from the point of view of increased Deuterium loading and generation of anomalous effects, was **Yoshiaki Arata** (Osaka University, 1993). Recently (since 2005, using an innovative Pd-ZrO_2 alloy) Arata get anomalous heat, in a fully reproducible way, for time as long as 2 days: D_2 gas up to 60 Atm.

d) Experiments of Arata were independently reconfirmed (2008) by **Akito Takahashi-Akira Kitamura** group (Technova Inc.-Kobe University collaboration, Japan): even used "commercial" material produced by a Company (Santoku Corporation, Kobe) experts in precious metals and RE purification. **Such experiment was a milestone**: **broke**, once forever, the **historical problem** of ***transferred irreproducibility*** in CMNS experiments.

e) Use of **multi-layer**: two elements (CaO-Pd) were deposited (like sandwich), at nano-metric size, over a bulk Pd plate (procedure developed by **Yasuhiro Iwamura**, since 2000, at Mitsubishi Heavy Industries, Yokohama-Japan). The aim of the experiment was to demonstrate the "transmutation" of selected elements (from Sr, Cs, Ba), deposited in the outermost side of the multilayer, to new one (respectively Mo, Pr, Sm), due to the effect of flowing D_2 gas in a slightly (max 2 Atm) pressurised chamber at mild temperatures (up to 80^oC). Iwamura used (advanced) ion beam technology for the multi-layers construction.

f) We experienced, since 1998, in electrolytic environments and using innovative electrolytes (like salts of Ca, Sr, Ba) at very low concentration (<0.1mM) and mild acidic pH, that some times, specially after several cathodic-anodic cycles, the Pd wire was covered by a sub-micrometric layer of several elements, Pd included. Because the ***spontaneous developing*** 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) even, and specially, at low current density of electrolysis (about 10-20 mA/cm^2). We observed correlation between increasing the amounts of thermal effects and the arising of compositional anomalies (detected by SEM-EDAX and, later, by ICP-MS). **We realised that the origin, or 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, on demand, the layers failed. In short, it was "spontaneous", very delicate and easy to self-destruction.

The new approach to fractal and nano-layer construction

***** Because of the spontaneous self-assembling/growing of sponge materials at the surface of Pd wire (due to electrolysis with their cathode-anode cycling) ***was out of a real control***, we **moved** to another **completely different direction**.

***** In short, we moved from **liquid electrolysis to gas** environment. One of the main aims was to avoid uncontrolled self-dissolution, due to the electrolytes and local pH changing (because of the cathode-anode different regimes) in the volume closest to the wire surface, of the peculiar sponge material. We **prepared the sponge material** over the wire, **in controlled way**, **before** the insertion of it in the reactor.

***** We adopted the **innovative procedure** of **coating intrinsically *active* supports** (like Pd at the moment, planned the use of Ni and Ti, with their alloys, under H_2 and/or D_2 gas) with **nano-materials** (used mainly as anti-sintering agent) **containing** also Pd at very low dimensions.

***** The nominal dimension of such nano-material, in most recent experiments, was 6-9 nm.

***** The ***active*** support is a Pd wire thin (ϕ=50μm) and long (60-90 cm). We chose to get full advantages of both the, previous quoted, electro-migration and Preparata effects.

***** The total **number of layers, made by (simple) physical-chemical deposition procedures,** is quite large (about **50**) so that the apparent increase of thickness of main wire is 1-2μm.

***** The coated wires were reacted/conditioned/stabilised by several (**high) temperatures cycling** (20->900->600->20°C), using Joule heating in air (J up to 35 kA/cm^2), with specific both time patterns and current ramps.

***** The wire was inserted inside, electrically insulated, porous glass (recently quartz) sheaths. In such a way were avoided both short circuitry and was possible to do, in-situ, calibrations (by another similar, in principle inert, Pt wire of same geometrical dimensions and quite similar specific resistance).


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Key characteristics of nanocoated Pd wires

1) The **loading time** (to R/Ro=2, i.e. D/Pd=0.75), using a pressure of H_2 or D_2 gas of about 6.5bar, was of the order of **only 6-30 seconds**. We used the ***loading time*** as a ***marker of quality*** of the sponge we build: ***as shorter as better***.

2) The de-loading time, using vacuum, is quite long and depends on the "quality" of nano-materials deposited on the Pd surface: ***as a general behaviour, as short was the loading time as long, and difficult, will be the de-loading procedure***. Several times it was necessary to reach, under vacuum, by Joule heating, temperature as high as 350-400°C for time as long as 1-2 hours, to get a "full" de-loading. **Anyway, even after such heavy de-loading conditions, we can't guarantee that the 100% of previous H_2 or D_2 absorbed left out from the bulk Pd and/or the specific sponge surface.**

3) The effect of 2), important to our specific purposes, was observed since 2002 even in electrolytic experiments: we named it the ***"Diode Effect"*** (i.e. unidirectional). In the electrolytic experiment, with our big surprise, the Pd wire get self-loading just absorbing the small amounts of Hydrogen and/or Deuterium previously dissolved in the water (and/or in mixed water-ethyl alcohol solution that was adopted at that time). Such phenomenon happened after the anodic stripping procedure ended. For sake of calculation, the value of D_2 dissolved in water was about $1.46*10^{-5}$ moles/mole of water at 298 K. The electrolytic cell contained about 25-50 moles of solution and the Pd wire weight was 15 mg, equivalent to about $14.1*10^{-5}$ moles. Resuming, the amount of D_2 dissolved was 5-10 times larger of that needed to get a value of D/Pd of as large as 1.

4) **As conclusions of (1, 2, 3) the "good" wires/*sponge* adsorb very easily the H_2 (or D_2). Once absorbed, such gases are very difficult to be fully de-absorbed.**

Description of the new experimental set-up 190110_#3

The new experimental set-up is a further developing (#3) of previous apparatus presented at ICCF14 (August 2008, Washington D.C.; #1) and ICCF15 (October 2009, Rome; #2).

Basically, they are similar each other because the power measurements (and calibrations) are based on the comparison of results (temperature detected by an independent thermometer, before another long Pt wire, now an usual Thermocouple-Tc) between: Pt wire (assumed to be inert under D_2 atmosphere; put very close to Pd, 1mm), the "active" Pd wire.

The procedure is based giving power alternatively to Pd and Pt wires, before in inert gas (^4He) and later in active (D_2, H_2). Equilibrium temperatures were recorded versus applied power.

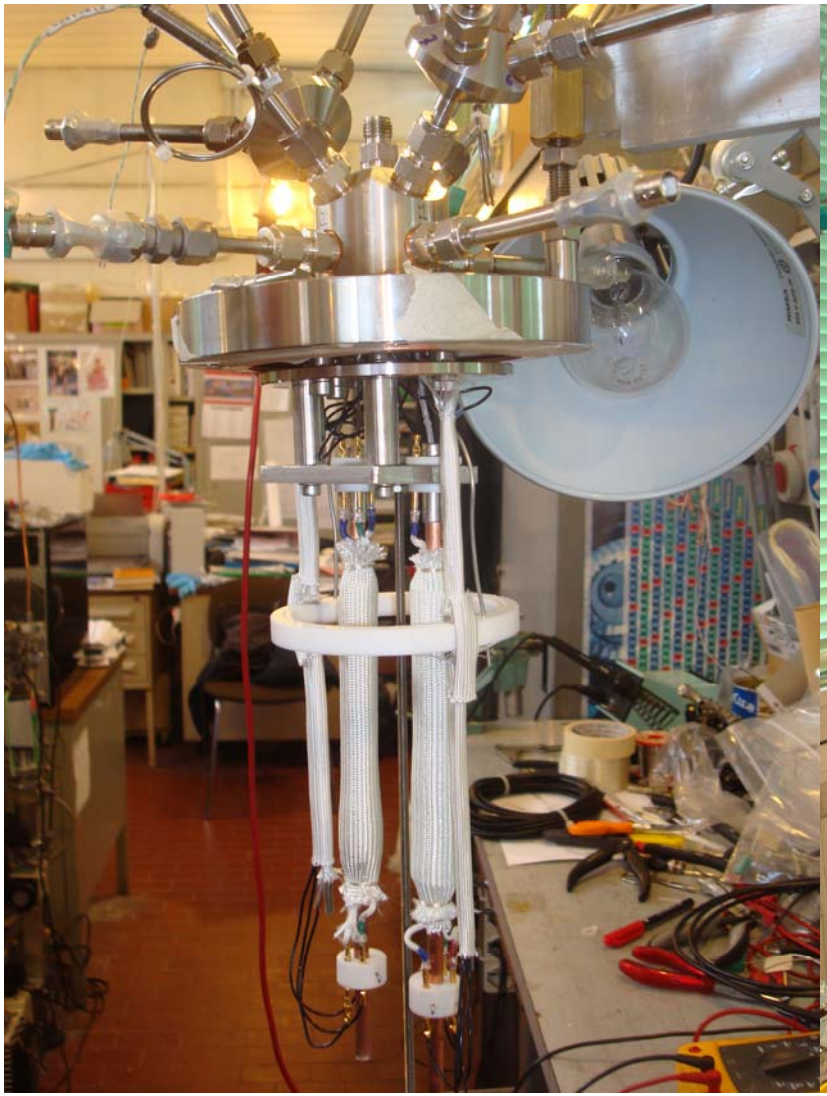
The wires were inserted inside two independents glassy sheath (in #2, 3 quartz), braided each other and thermally insulated from the SS internal wall by HT wool. The internal temperature of the SS reactor wall reached, at maximum, ***110°C*** (***no S problem, as pointed by T. Hioki***).

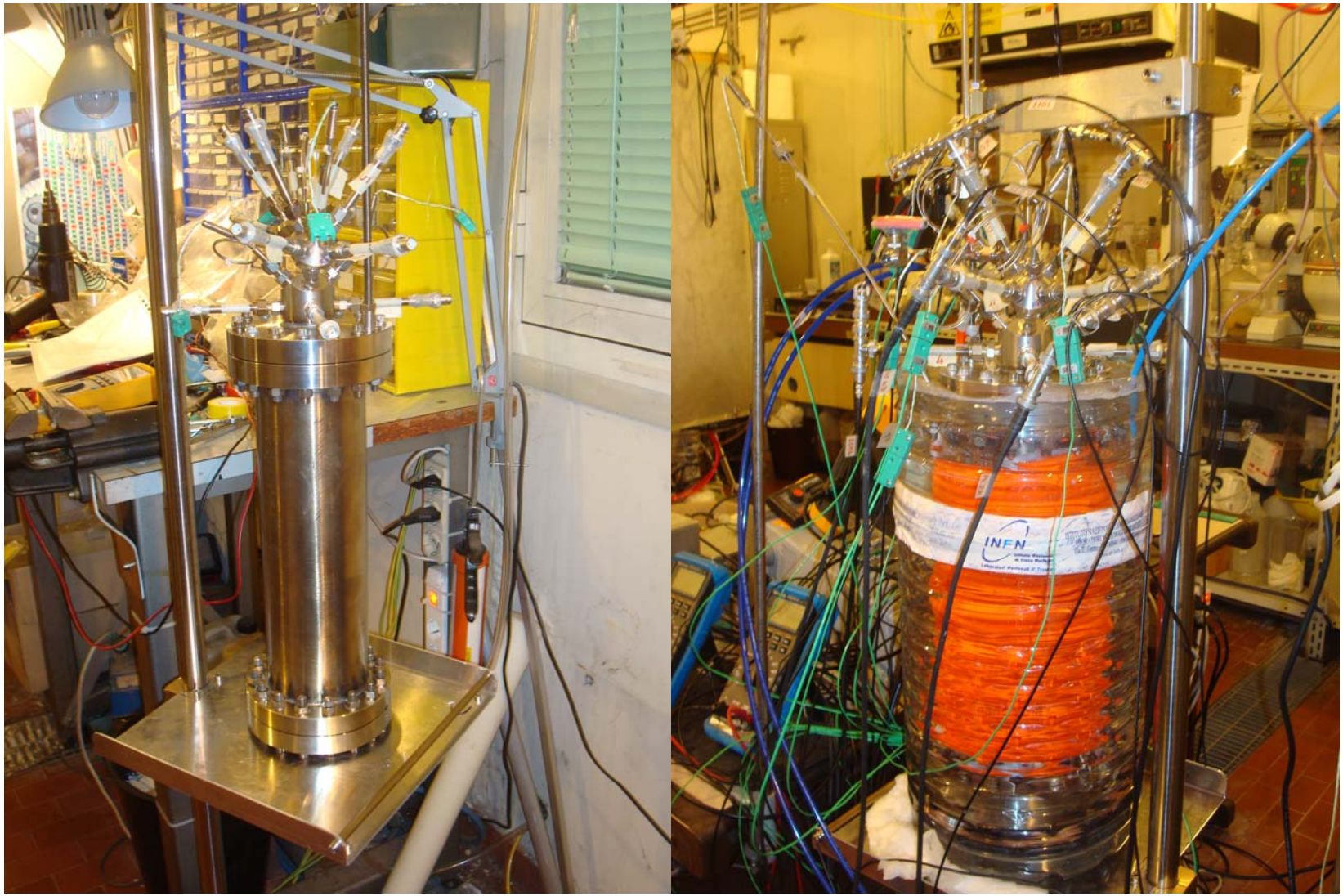
Main improvements were:

a) The braids, made by the quartz sheath allocating the Pd and Pt wires, were rolled up around a Cu 3N tube (4-6 mm inner-outer diameter; length 24 cm) in order to make homogeneous the temperatures. In the inner side of the tube, in the middle, was put a, high resolution, type K Tc with insulated body (SS 316N again gas; MgO as electrical insulator): build a sort of furnace were heaters are, alternatively, the Pd and Pt wires.

b) It was build another “couple” of braids and Cu tube, but with inserted two Pt wires instead of Pd-Pt. Such second set of wires was used as, in situ and on-line, cross-comparison of the behaviours of the reactor changing the atmospheres (^{4}He, D_2, D_2-Ar mix, Ar, Air, Vac.).

c) Both the couples of braids were individually thermal insulated and put in the same pressurised SS cell with the external wall put in a large water bath continuously cooled.

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Methodology of measurements

According to our measurement procedures, were made experiments in ^4He (calibrations) and subsequently D_2 gas atmospheres (both at 6.5bar). Tests made by Ar, Air, Vac. Moreover, experiments were made also with a ***mixture of D_2 (P=4.62bar) and Ar (P=1,68bar).*** The rational was to study if the effect of larger temperature (the thermal conductivity of Ar is over 7 times lower in respect to D_2 at temperature between 35 and 90°C) of Pd wire (at constant power) could compensate for lower pressure of D_2 (i.e. from 6.5 to down 4.6 bar) or even improve the over-all amount of anomalous excess heat production. About gas thermal conductivity, adding 24% of Ar to pure D_2, it is reduced (at 95°C) of 56% (from 151 to 96 $mW*m^{-1}*K^{-1}$). **Such important test, made in reactor #2, was repeated also in reactor #3: obtained similar results.**

The answer was that the increase of temperature improved, in a large way, the anomalous heat production and overcame the deleterious effect of D_2 partial pressure reduction.

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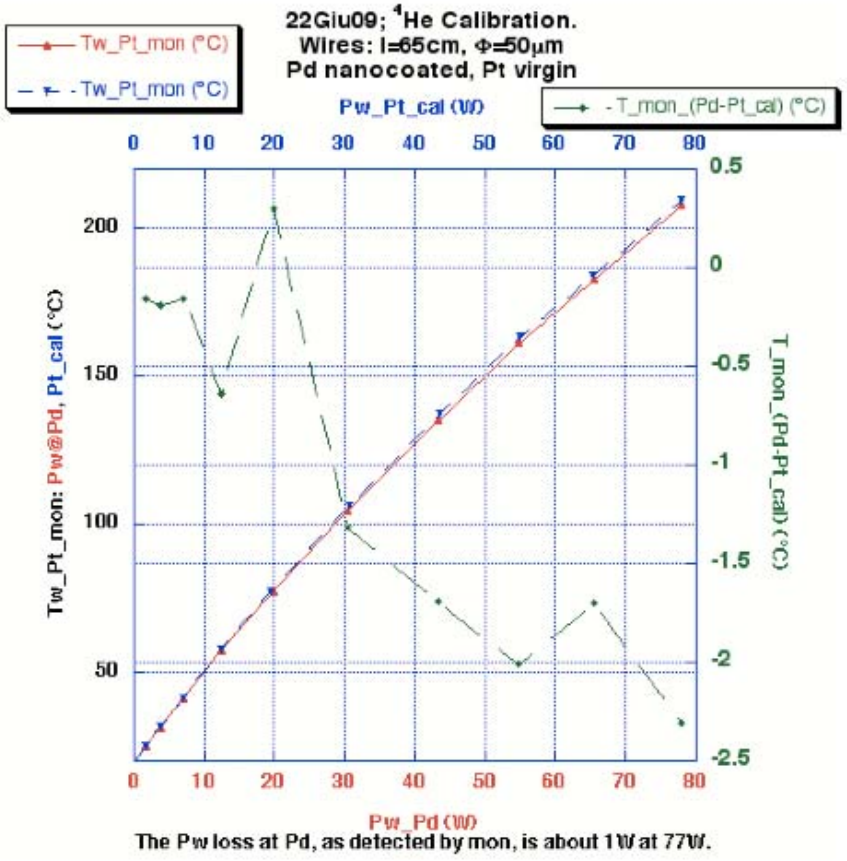

The Pw loss at Pd, as detected by mon, is about 1W at 77W.

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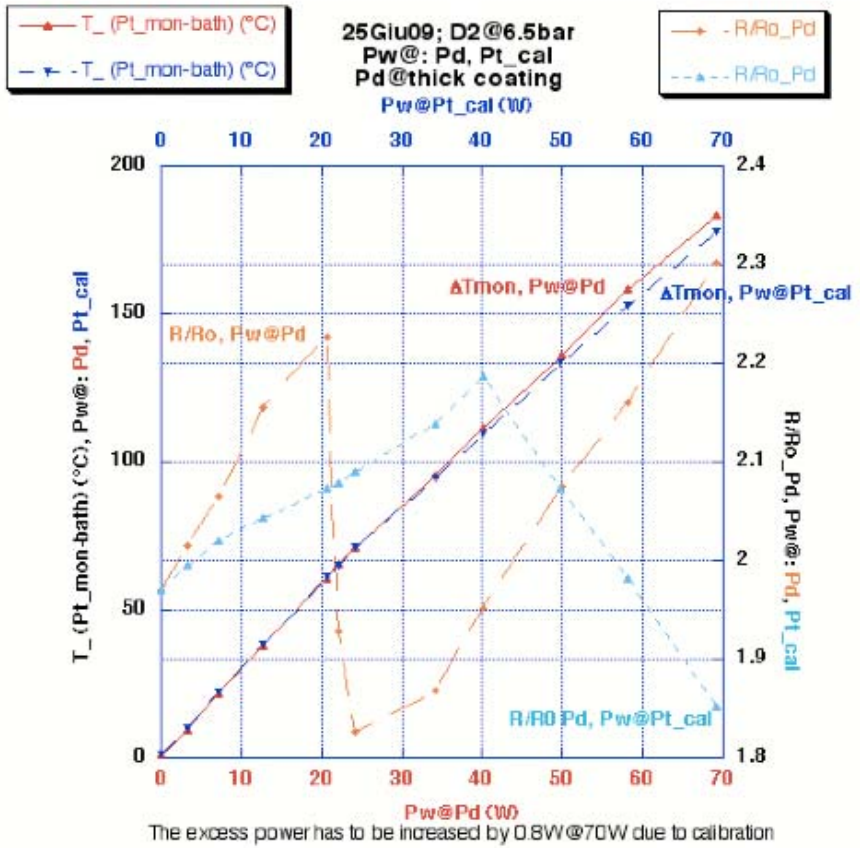
T_ (Pt_mon-bath) (°C)
- T_ (Pt_mon-bath) (°C)
25Giu09; D2@6.5bar
Pw@: Pd, Pt_cal
Pd@thick coating
- R/Ro_Pd
- - R/Ro_Pd
Pw@Pt_cal (W)
0 10 20 30 40 50 60 70
200 150 100 50 0
2.4 2.3 2.2 2.1 2 1.9 1.8
ΔTmon, Pw@Pd
ΔTmon, Pw@Pt_cal
R/Ro, Pw@Pd
R/R0 Pd, Pw@Pt_cal
T_ (Pt_mon-bath) (°C), Pw@: Pd, Pt_cal
R/Ro_Pd, Pw@: Pd, Pt_cal
Pw@Pd (W)
0 10 20 30 40 50 60 70
The excess power has to be increased by 0.8W@70W due to calibration

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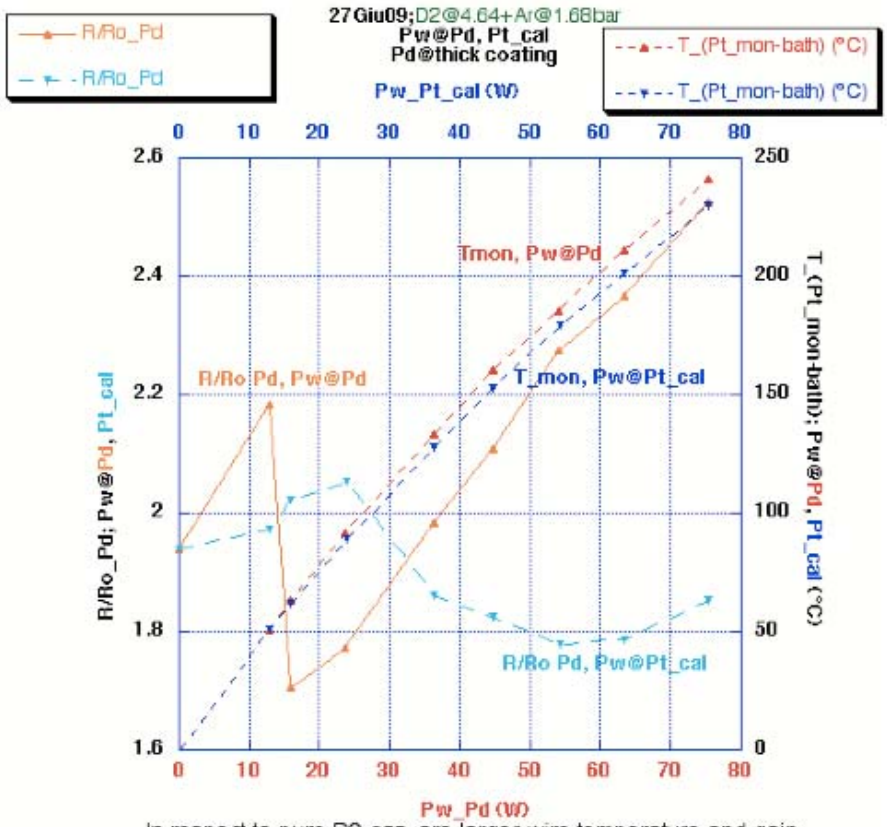


In respect to pure D2 gas, are larger wire temperature and gain.

Main results obtained with reactor #3 and theoretical interpretation

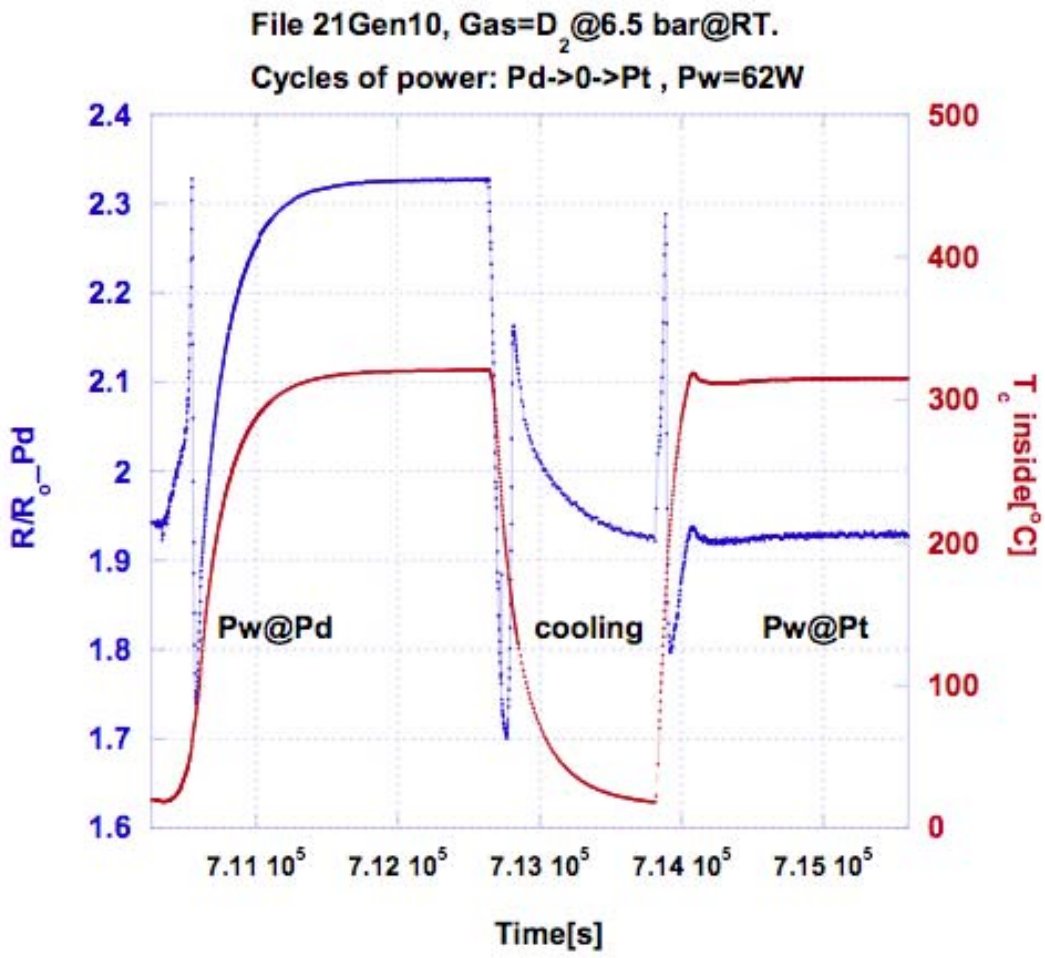
Main results obtained during last two months of experiments were as following:

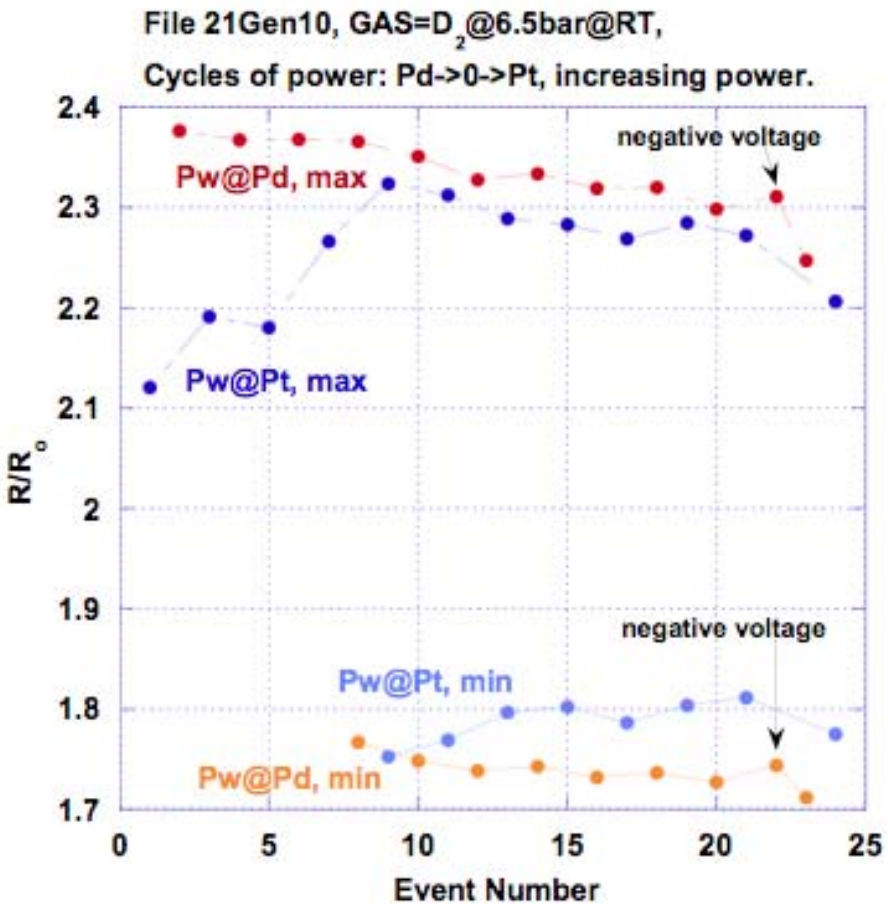
a) We get further confirmation, at high time resolution (1s), that the **maximum of R/Ro of Pd, under direct heating (electro-migration) is ALWAYS larger of any possible maximum due to the temperature** (by indirect heating of the Pt wire, close to Pd one, and used as heater).

Only our experimental set-up is able, at the moment, to study such key effect,

in real time and high resolution,

ruling-out any possible error due to some systematic reasons.


File 21Gen10, Gas=D2@6.5 bar@RT.
Cycles of power: Pd->0->Pt , Pw=62W
R/R_o_Pd
T_c inside[°C]
Pw@Pd
cooling
Pw@Pt
Time[s]
2.4
2.3
2.2
2.1
2
1.9
1.8
1.7
1.6
500
400
300
200
100
0
7.11 10^5
7.12 10^5
7.13 10^5
7.14 10^5
7.15 10^5


File 21Gen10, GAS=D2@6.5bar@RT,
Cycles of power: Pd->0->Pt, increasing power.
negative voltage
Pw@Pd, max
Pw@Pt, max
negative voltage
Pw@Pt, min
Pw@Pd, min
R/R°
Event Number
2.4
2.3
2.2
2.1
2
1.9
1.8
1.7
0
5
10
15
20
25

* Such effect, if we assume that the value of R/Ro is directly related to the D/Pd ratio, can be interpreted as experimental evidence of some kind of "**confinement**" of Deuterium inside Pd due to voltage drop along the thin and long Pd wire (according to **Preparata** Model).

* Such **maximum** was interpreted, by Prof. **Yeong E. Kim** (Purdue University, USA), as evidence **Bose-Einstein Condensation Nuclear Fusion** (BECNF, Naturwissenschaften, 2009): our ICCF14 experiments were quoted as ***first experimental proof of such model.***

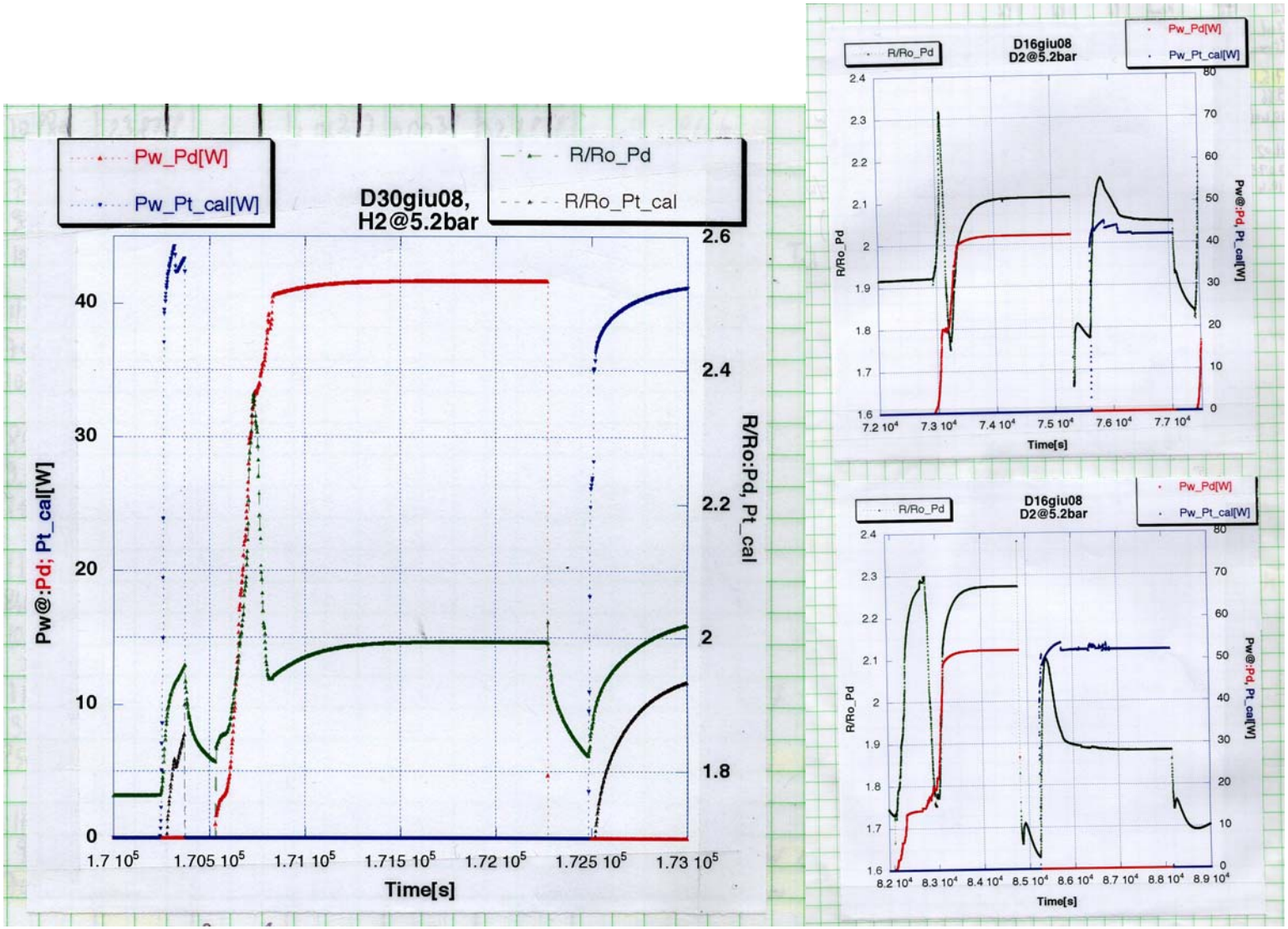
* Prof. **Akito Takahashi** (Technova Inc.) thinks BEC thoughtful concept to start to study the anomalous CMNS effects: his **TSC** model is a special case of dynamic (transient) BEC.

***** Prof. **Francesco Premuda** (Bologna University, Bologna-Italy), since 1993 (Fusion Tech., 1998), predicted that several of CMNS effects could be explained as a special case of **BEC**.

b) The **minimum of R/Ro, in Pd/D** system, is always **LOWER** of any minimum that can be achieved using indirect heating. Such effect was predicted and interpreted by Prof. **Premuda** (now deceased) and by his collaborator Eng**. Emanuele Costa** (Bologna University-Italy, unpublished) as proof of ***"island" of SUPERCONDUCTIVITY*** in D/Pd system (supposing the system in plasma regime).

c) We observed that, using **Pd-H** systems, in similar conditions of direct and indirect heating, in our experimental conditions (i.e. Pressure =6 bar at RT), the maximum of R/Ro is VERY SIMILAR to that of Pd-D system (i.e. 2.35) while ***the minimum is larger in respect to Pd-D experiments.***

Again, the effect of minimum can be explained according to the model of b). In this case, because there are H (Fermion) and not D (Boson), we can' t get islands of superconductivity.


Pw_Pd[W]
Pw_Pt_cal[W]
R/Ro_Pd
R/Ro_Pt_cal
D30giu08,
H2@5.2bar
Pw@:Pd; Pt_cal[W]
R/Ro:Pd, Pt_cal
Time[s]
D16giu08
D2@5.2bar
R/Ro_Pd
Pw_Pd[W]
Pw_Pt_cal[W]
Pw@:Pd, Pt_cal[W]
Time[s]

d) We observed that if we put a **NEGATIVE** voltage polarization to the Pd wire, in respect to almost grounded Pt wire (distance 1mm), **there is a measurable increase of both R/Ro_max and anomalous heat production (+12%)** when compared to same operating condition were the voltage is POSITIVE. Moreover, the temperatures detected inside the Cu tube, at the maximum and minimum of R/Ro are remarkably larger. We observed such effect since 1996 in electrolytic systems (Pd wire rolled around a Cu wire, kept at as same high voltage and insulated by PTFE), and discussed in Conferences, but the high voltage discharge problems forced us to quite such way to increase gain. We are developing a new version of reactor (#4): it is dedicated to study more deeply such phenomenon. It can be quite powerful to increase the amount of anomalous reactions with minimal increase of the electric input power (capacitive effect). In addition, we can speculate that, changing at proper (low) frequency the sign of voltage, could be possible to stimulate the movement of Deuterium from and into Pd bulk and/or sponge surface.

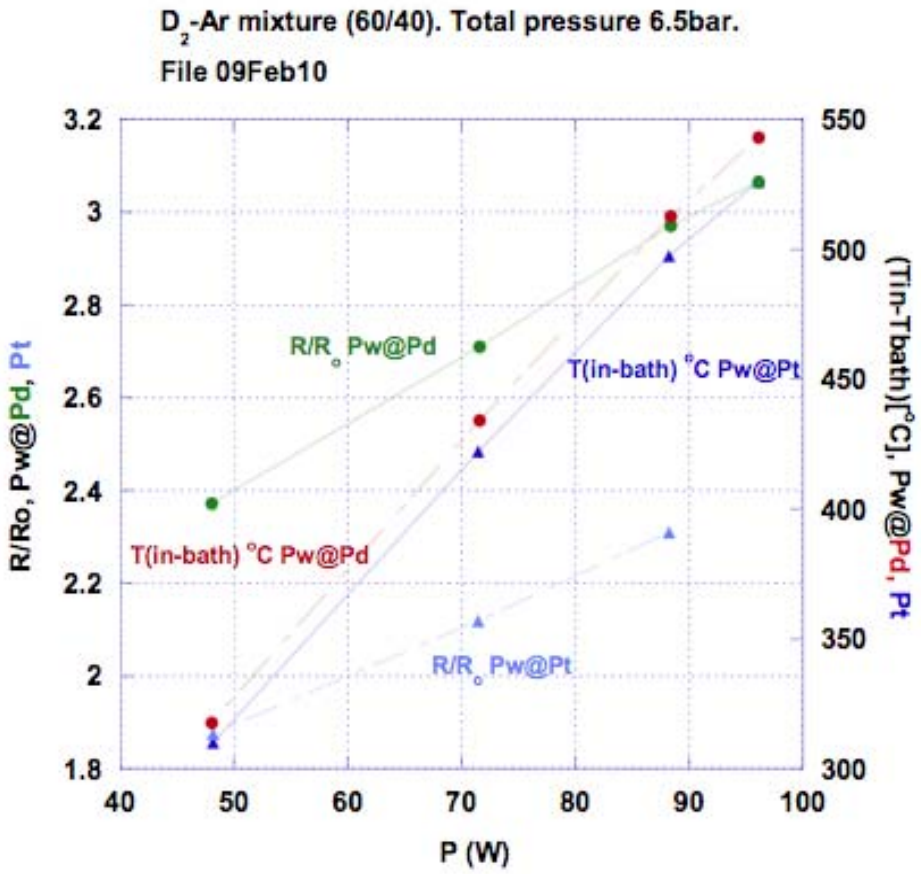
e) We observed that even Pt absorbs some Deuterium (at the level of 1-2%) when there is electro-migration. Estimated by R/Ro measurements.

f) We observed that Pd, under strong electro-migration (J=40-50 kA/cm^2) absorbs He at 1-2% level. Estimated by R/Ro measurements.

g) We observed that also Pt, under strong electromigration (J=40-50kA/cm^2), absorbs He at 0.3-1% level. Estimated by R/Ro measurements.

h) We observed that addition of Ar reduce strongly all such effects in Pt and Pd (about He).

i) We get CONFIRMATION that the **high temperature** is one of the **key parameter** to increase the anomalous heat (another experiment with D_2-Ar mixture) in D/Pd systems.


D2-Ar mixture (60/40). Total pressure 6.5bar.
File 09Feb10
R/Ro, Pw@Pd, Pt
(Tin-Tbath)[°C], Pw@Pd, Pt
P (W)
R/R° Pw@Pd
T(in-bath) °C Pw@Pt
T(in-bath) °C Pw@Pd
R/R° Pw@Pt
3.2
3
2.8
2.6
2.4
2.2
2
1.8
550
500
450
400
350
300
40
50
60
70
80
90
100

CONCLUSIONS

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

2) According to our experiments, in the Pd-Deuterium system,
the key parameter that controls the amount of anomalous heat generated
is the "moving" of Deuterium into or from the Pd bulk and/or nano-coating.
In other words, it is necessary to
maximize the **amount** of Deuterium moving and its **speed**.
Such effect, although discovered (by us) since 1993,
was quite difficult to be obtained in practical experiments**.**

3) The method of coating some specific nano-materials (including Pd at very low dimensionality) over a long and thin **active** (from the point of view of Deuterium absorption) Pd **wire** seems a reliable procedure, at least at Laboratory level, to fulfil the request of reproducible experiments.

4) We experienced that the high temperatures (of the order of 300^{o}C and over), even under mild pressures (4-8Atm), are pre-conditions to get some macroscopic effect.

5) We experienced that high temperatures, by itself, increase the effects, especially **over 700^{o}C**. **Such effect was by us predicted and experimentally found.**

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

7) 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.

8) The power density (up to 400W/g of Pd) and time duration (in principle no time limit, experienced by us 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 the numbers of ON-OFF cycles, probably because were increased the numbers of fractals at the surface (then further reduced dimensionality). It is the, well-known, phenomenon of both **embrittlement** of Pd (and of several other metals, alloys) and large **changing of dimensionality** (see specific our report at ICCF15) due to Hydrogen/Deuterium loading de-loading cycles. Drawback of such (simple) procedure is the possibility that the wire broke, especially if it is thin. So, we made some specific “developments” to reduce such catastrophic final result.

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

10) 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, at macroscopic levels, the input power.

11) If all the still remaining **doubts/questions/developments** will be (soon) fully **clarified/answered/performed**, the phenomenon under study can be the starting point for a **new source of energy for practical purposes** (like engines). We recall that, in our specific experiments, we can operate at quite large temperatures (about 700^{o}C, i.e. at high intrinsic Carnot efficiency) and that the effect increases when the temperature of the Pd wire increases (**positive feedback, obviously to be further exploited and kept under full control).**