Deuteron Electromigration in Thin Pd Wires Coated With Nano-Particles: Evidence for Ultra-Fast Deuterium Loading and Anomalous, Large Thermal Effects

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Abstract

Large excess heat is measured in a Pd wire coated with nano-particles. A long (65 cm) and thin (50 μm) Pd wire is coated with thin layers of Pd nano-particles, stabilized against self-sintering by the addition of selected chemical elements: the coating is adhered to the wire surface by heating it in air up to over 800°C. The wire is then heated with up to 1 A of direct current in a pressurized D_2 gas atmosphere. The D^+ deuterons in the Pd lattice are forced to move toward the cathodic end of the wire because of the voltage drop along the wire (the Cöhn effect). Large excess power density (about over 400 W/g of Pd), at high temperatures (up to 400-500°C), is then measured using isoperibolic calorimetry. The reference experiment is made, in situ and without opening the cell, using a Pt wire of same dimensions as the Pd wire, to which was applied the same electrical power. The onset of excess heat occurs in during a phase change from an $\alpha + \beta$ combined phase of the Pd-D to the α phase, and is proportional to the current density, and to the corresponding voltage drop or input power applied, i.e. the Pd temperature. In the range of temperatures explored up to now, the excess power has exhibited "*positive feedback*" behaviour versus temperature. This may prove useful to developing future *self-sustaining* devices for *practical applications*. No anomalous effects were found using ^{4}He (or Ar, or dry-air) gases.

1. INTRODUCTION

1.1. Nano-particles in Cold Fusion

The use of nano-materials in cold fusion studies started with the systematic studies of Yoshiaki Arata (Osaka University, Japan), and his Collaborators, which have been conducted

since 1993 [1]. They first used Pd-black, with particle sizes varying from several micrometers down to a fraction of a micrometer. Later, in 2002, they developed an innovative material by embedding Pd particles with a diameter of about 10 nm in a matrix of ZrO_2 [2].

In May 2008, Arata measured excess heat in the presence of pressurized (60 bar) D_2, using the ZrO_2-nano_Pd material without applying any input power. The excess heat lasted over 50 hours. The chemical heat of formation for this system was observed in a control run using H_2 with the same material: it did not produce persistent, long lasting heat. Large amounts of [4]He, as ashes from the D+D reaction, were also found. The [4]He gas, a known product of cold fusion reactions, was detected on line using a High Resolution Quadrupole Mass Spectrometer [3].

Another procedure to produce nano-materials was developed by Yasuhiro Iwamura and Co-workers (Mitsubishi Heavy Industries, Yokohama, Japan, 2000). The procedure is similar, in some ways, to the fabrication of silicon semiconductors. In the Iwamura experiment, a Pd wafer is fabricated which contains several thin Pd-CaO nano-layers, with a small amount of Sr, Cs or Ba on the surface. When a flux of slightly pressurized D_2 at a temperature of about 80°C is forced to pass through the wafer, the surface materials are transmuted into Mo, Pr and Sm respectively [4, 5].

Most of the other experiments in this field, using gas loading and/or nano-materials (even surface-oxidized Pd tubes), were summarized by Jean-Paul Biberian at this Conference [6].

1.2. Main results of our previous experiments with Pd nano-particles

The maximum excess power achieved in our previous experiments with Pd nano-particles was only about 0.5 W/g of Pd at 300°C and 60 bar of D_2 [7, 8]. These particles were tightly packed together, not coated onto a metal substrate as they are in the experiments described in the present paper. They were produced by filling nano-porous (Φ=5.8 nm) materials (such as γAlumina) with soluble salts of Pd and Sr. They were then decomposed to oxides through high temperatures cycles of calcination and controlled sintering under a controlled atmosphere in a quartz furnace.

The methodology of "incipient wetting impregnation" was adopted for the preparation of the nano-porous material. This standard procedure was modified in order to maximize the amount of Pd in the nano-porous material (up to 10-15% w/w).

A twin-chamber differential reactor (Fig. 1) was developed at INFN-LNF to evaluate the behaviour of the Pd nano-particles under a deuterium or hydrogen atmosphere.

The Pd nano-particles were placed in the stainless steel crucible inside the working chamber. Combinations of inert materials and gas such as Ag- γAlumina and D_2, or supposedly inert gas such as H_2 with active Pd-Sr- γAlumina, were placed into the crucible of the reference chamber for calibration purposes.

Figure 1. The twin chamber differential reactor developed at INFN-LNF. Pressure: up to 100 bar. Temperature: up to 320°C. Useful volume of the crucibles: 15 cm^3.

1.3. Electromigration and the main result of our previous experiments

Since 1994, a series of experiments using the so-called HPPE technique ("High Power Pulsed Electrolysis") was performed by our research team at Frascati National Laboratory of the National Institute of Nuclear Physics. The geometric set-up of the cell was similar to that previously employed by Akito Takahashi (Osaka University, Japan) in 1992 [9]. From 1992 to 1993, we followed Takahashi's procedure using up to 5 A of direct current to electrolytically load a Pd plate cathode (25×25×1 mm). Starting in 1994, we moved from DC to pulsed power: 300 V maximum, 150 A maximum, typical pulse duration 1-2 μs, repetition rate up to 30 kHz.

The HPPE procedure was later applied to long (50-300 cm) and thin (Φ=100-250 μm) Pd wires. In this case, in contrast to the Pd plate, we employed both electrolysis and strong electromigration, at the same time. The D^+ deuterons in the Pd lattice are forced to move along the Pd wire (the Cöhn effect) because of the voltage drop between the most cathodic and less cathodic ends of the wire. We decided to adopt this technique after profitable discussions with Giuliano Preparata (Milan University, Italy) and his collaborators. The electromigration effect applied to cold fusion experiments was pioneered by Martin Fleischman and his collaborators since 1993 [10].

During electromigration, the equilibrium state for the deuterium concentration at a fixed point x of the Pd wire is given by:

$$C_x = C_o \exp \left[-eZ^* V(x)/K_B T\right] \qquad (1)$$

Where:

e = electron charge ($1.602 \cdot 10^{-19}$C);

K_B = Boltzmann constant ($1.38 \cdot 10^{-23}$J/K);

T= temperature (K);

Z^* = effective charge number of H (or D) in Pd. The charge number is equal to 1 at low concentrations (H/Pd <<0.6). It decreases down to 0.1 at higher concentrations (H/Pd>0.8). According to some authors (G. Preparata, 1996, unpublished), it increases again (Z*→1) at very high loading ratios (H/Pd >1).

The performance of pulsed electrolysis (current density along the wire up to 300,000 A/cm^2), in a specific experiment is described in one of our paper published by Physics Letters A [11].

In that series of experiments we found that the surface of the Pd wire was progressively covered with thin, uncontrolled layers made of different materials. The materials came from: impurities present in the electrolyte; the main chemical elements of the electrolyte itself; and partial anode dissolution (Ni in this experiment). We also found that such layers played an important role in the loading rate ($\delta D/\delta t$) and ratio (D/Pd) of the Pd cathode, which are key points for anomalous heat production.

In the present work we decided to perform the Pd loading in a D_2 atmosphere, by using (as in the HPPE technique) both electromigration and, in a substantial improvement over our previous studies, a *controlled coating* on the surface of the Pd wires, which is made without electrolysis.

Unlike the electrolytic technique, the gas loading technique allows us to easily explore the effect of temperatures well above 100°C. The Pd consists of a complex mixture of soluble Pd salts and other elements deposited on the surface of thin Pd wires. The wires are then heat treated by Joule heating in air, up to over 800°C. This temperature is high enough to decompose the PdO, which forms during heating the Pd in air at 500-750°C. Specific chemical compounds were added to the Pd soluble salts in order to improve both the thermal stability of the resulting Pd nano-layer and the mechanical strength of the Pd wire itself. Moreover, the other specific elements added to the Pd surface have the property of largely reducing the nano-Pd self-sintering caused by high temperatures and reduced dimensionality.

The method of preparation described above is also known as "Nanoscale Surface Modification" and is based on the "Atomic Layer Deposition" process.

2. Experimental Reactor

We modified an experimental reactor previously used by us, in 2004, for Pd experiments in hydrogen without fibre-glass sheaths. We tested D_2 loading of Pd wires (and inert gases for comparison). The reactor acts as a differential isoperibolic calorimeter. Inside it are three wires braided together: a Pd wire coated with nanoparticles (that can produces excess heat), and two Pt wires, designated Pt_{cal} and Pt_{mon}. Pt_{cal} serves as a calibration Joule heater and Pt_{mon} serves as

a distributed thermometer; that is, an RTD (resistance temperature detector) that is placed alongside the full length of the Pd and Pt_{cal} wires.

All three wires are of the same diameter (50 μm) and length (usually 65 cm). They are put inside thin, flexible fiberglass sleeves, which withstand a maximum temperature of 550°C, to ensure that they are electrically insulated from one another. The three fiberglass-covered wires are braided together, to insure that the positions of the three wires are symmetrically exposed to the gas and to the other two wires (Fig. 2).

Figure 2. Three wires braided together: 1 coated Pd wire and 2 Pt wires (Ptcal, Ptmon). Each wire is 50μm in diameter. The diameter of the fibre-glass sheaths is 2 mm. The braid is completely covered inside a larger sheath (6 mm) shown below. Ultra-pure (>99.99%) Pt was used for Ptcal and Ptmon. Ptcal serves as a calibration Joule heater and Ptmon serves as a distributed thermometer.

The braid is thermally insulated as shown in Fig. 3. Each wire can be individually supplied with constant direct current, on a rotating basis (one wire at a time). The electrical resistance of each wire is continuously computed by the V/I ratio. The braid covered by thermal insulation is put into stainless steel tube (36 cm length, 10 cm OD, 9.5 cm ID) (Fig. 4) capable of bearing pressurized D_2 (10 bar maximum). The tube is evacuated, and then D_2 gas is admitted into it.

Figure 3. The braid placed inside thermal insulation.

Figure 4. The pressurized experimental cell surrounded by a large water bath.

Typical operating pressure is 6 bar absolute. The stainless steel tube is placed inside a large water bath in order to make the temperature homogeneous; to dampen the effects of room temperature variations; and to dissipate the heat produced inside the tube. The large, thick flange covering the cell is made of an Al alloy to increase thermal conductivity. The internal wall of the cell is fully covered by a 1 mm thick Cu foil to improve thermal homogeneity. The water bath temperature is continuously measured.

The loading rate and loading ratio of the Pd wire is monitored by measuring its resistance ratio (R/Ro), with Ro normalized at 20°C (details below). All gas is removed from the Pd by long-time vacuum degassing at high temperatures (about 450°C).

Once equilibrium is reached – meaning that the loading ratio is stable, the selected D_2 pressure is reached, and the cell is at room temperature – constant direct current power is fed to the Pd wire, which causes electromigration and Joule heating of the wire. Power is typically 5 W at the beginning of the experiment. The temperature of the system rises up until the electrical resistances of the three wires becomes constant (but different for each of the three wires). At this point thermal equilibrium is reached (isoperibolic condition) characterized by the temperature reached by the thermoresistance Pt_{mon}. The power supply is then turned off and the temperature of the system is allowed to fall to room temperature, or – to save time – the temperature is allowed to fall to a fixed value of Pt_{mon} above room temperature (usually 50°C).

The same power level (5 W) that was previously fed to the Pd wire is now fed to the Pt_{cal} wire. The new thermal equilibrium is again characterized through the measurement of the Pt_{mon} temperature. This is differential isoperibolic calorimetry.

Obviously, if the equilibrium temperatures of Pt_{mon} are the same in both cases, no excess heat is being produced. If, on the contrary, a temperature difference is measured, the excess power generated by the Pd wire can be estimated by simple means: e.g. by the extra power which has to be fed to the Pt_{cal} to close the gap between the two Pt_{mon} measured temperatures. Another simple and fast procedure adopted was graphic interpolation; e.g., Fig 5 with ^{4}He gas shows no excess heat, whereas Fig. 8, with D_2, shows large excess heat.

Further values of direct current power (e.g. 10, 15, 20, 25…50, 55 W) are then applied first to the Pd wire and then to the Pt_{cal} wire to measure excess heat as a function of the Pd temperature. This is evaluated by comparing the effects of the power fed to the Pt_{cal} wire on the temperature reached by Pt_{mon}.

The braid arrangement of the three wires should assure good symmetry, so that heat is distributed in the cell the same way regardless of which wire is heated. Thus, in the absence of excess heat, the temperature of Pt_{mon} should be the same when the same level of direct current power is fed to either the Pd wire or to the Pt_{cal} wire.

To verify the symmetry of the system, we begin the experiment by performing several blank tests in a helium atmosphere. The thermal conductivity of He is quite close to that of D_2 [12, 13].

To better understand the performance of the system and confirm the results as a whole, some tests were made in a vacuum (typically 10^{-5} to 10^{-6} bar), and in dry-air, and argon at different pressures and power levels.

3. Experimental Results

3.1. Blank test.

Before each experiment in a D_2 atmosphere, a preliminary blank test is always performed in a helium atmosphere to assess the geometrical and thermal symmetry of the braid. The helium test is repeated, if possible, at the end of each series of experiments.

The results of a blank experiment, with helium absolute pressure at 6.2 bar, are shown in Fig. 5. When the same power is applied to either the Pd wire (red line) or to the Pt_{cal} wire (dark blue line), the Pt_{mon} temperatures are practically the same. The two lines almost completely overlap. At the applied power of 42 W the measured temperature difference is less than 1%, a level of uncertainty of less than 0.4 W.

As far as the R/Ro ratio of the Pd wire is concerned: when the power is applied to the Pd wire (direct heating), the R/Ro ratio values are obviously higher than those observed when the Pd wire is indirectly heated by the Pt_{cal}. For example, at a pressure of 6 bar, direct heating of the Pd at 42 W produces a R/Ro ratio (with Ro measured at 20°C) of 2.03 (orange line) equivalent to a temperature of about 340°C. The indirect heat of the Pd with 42 W through the Pt_{cal} wire produces an R/Ro ratio of 1.45 (light blue line) equivalent to a temperature of about 140°C. In the latter case the Pd wire only reaches the temperature of Pt_{mon}.

The difference in temperatures between the direct and indirect heat depends on the geometry of the pressurized chamber and the specific configuration of the wires, together with the performance of thermal insulation. In this case, we designed chamber geometry to make the temperature changes large in order to easily study the effect of temperature. In a chamber designed to optimize the efficiency, i.e. to reduce input power and get at the maximum amount of anomalous excess heat, the geometry will be drastically changed. We have built a new cell that should reduce the amount of input energy needed to sustain the reaction. Not only is it more efficient, it is equipped with additional thermometers outside the stainless steel chamber in order to eliminate any possible influence due to slight differences in the thermal conductivity of deuterium and helium.

As noted the Pt_{mon} wire acts as an RTD (resistance temperature detector); temperature evaluations are made by a fourth order fit of resistivity data for Pd and Pt, according to CRC data-book (84th edition, 2004), in the temperature range of 273-900 K.
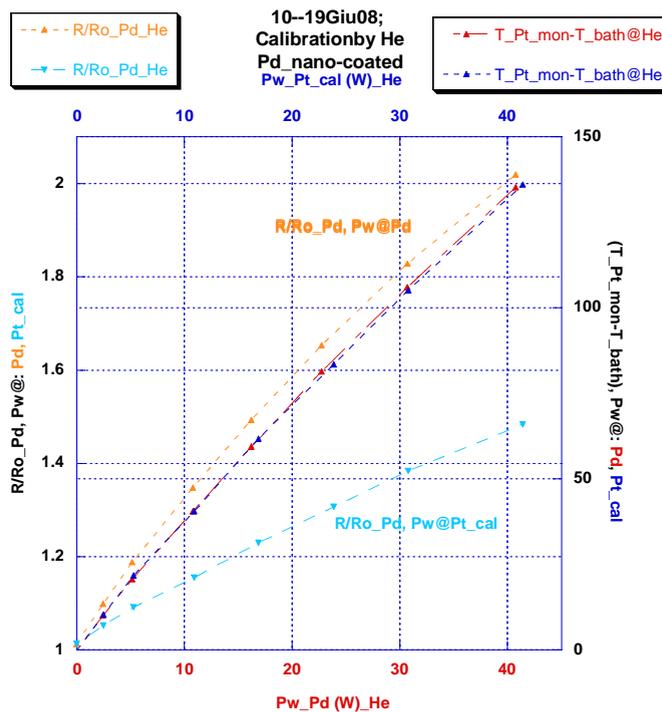
Figure 5. Calibration of the system with helium at 6.2 bar absolute.

3.2. Deuterium atmosphere

Once the blank test/experiments finished, the reactor temperature is allowed to drop down to room temperature for a final cross-check. The helium is then completely removed with a rotary pump equipped with a liquid nitrogen trap. The trap ensures that hydrocarbons from the oil pump vapor are not absorbed by the highly reactive nano-particles. To fully remove the helium from the Pd and Pt wires, the degassing is usually performed at high temperatures (about 450°C) under vacuum conditions. After final cooling to room temperature, a pressure valve is opened, allowing D_2 to rapidly enter the cell. In a few seconds the working D_2 pressure (6.2 bar absolute) is reached and the D loading of the coated Pd wire begins.

A loading rate up to R/Ro = 1.9 is achieved astonishingly quickly: typically in 10 seconds (Fig. 6, blue line). This is probably because of the chemical heat released by the absorption process, which increases the temperature of the wire (Fig. 6, red line). The higher temperature, in turn, increases the loading rate. The net importance of the nano-particles coating with respect to the Pd loading rate is highlighted in Fig. 7. This shows that with long treatments of the Pd wire without any coating, heated up to over 800°C in air, the required loading time is as long as 1000 seconds.
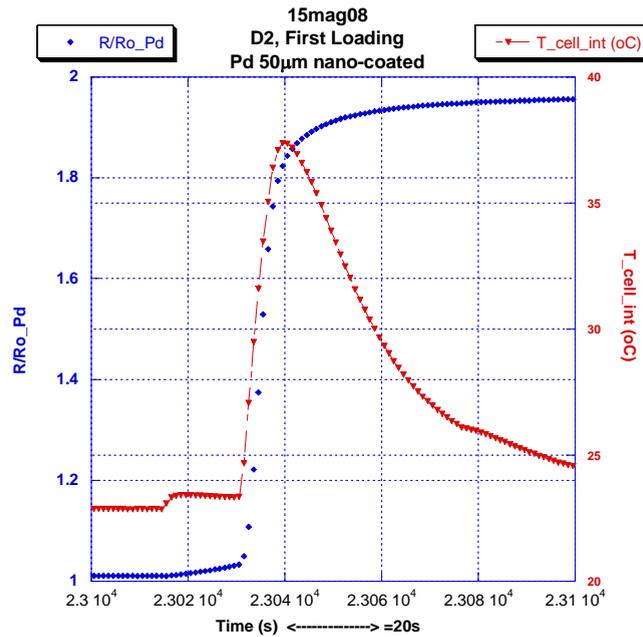
Figure 6. 50 µm nano-coated Pd exposed to deuterium gas at 6.2 bar loads quickly, in 10 – 30 s. In the plot shown in Fig. 6 the "T_cell int" label means the temperature measured at the top of the three fibre-glass sheaths, thermally insulated (as shown in Fig. 3) The temperature is measured by a K type thermocouple installed in the stainless steel tube (Φ=1.5mm).
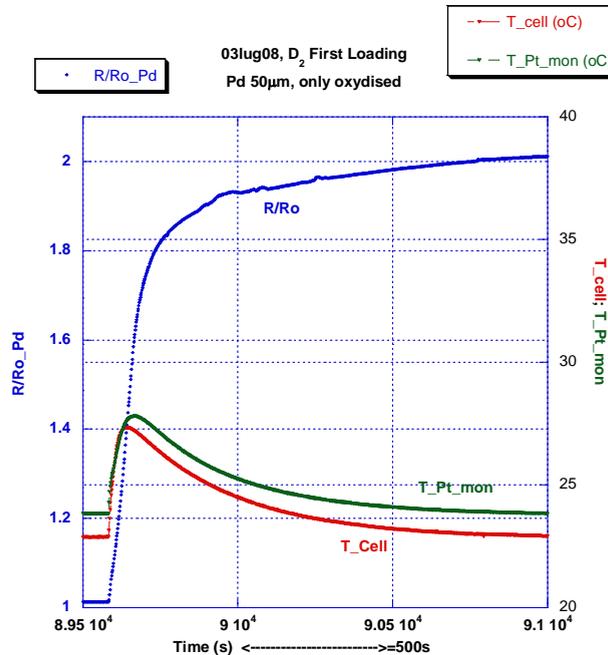

Figure 7. 50 µm Pd without nanoparticles. It has been treated only by being oxidized at temperatures between 600 and 700°C, reduced at over 800°C, in several cycles for a total time of 30 minutes. Final cooling from the high temperatures was slow (two minutes) to avoid quenching, which causes an apparent increase in Pd resistivity because of thermal stress.

Once equilibrium is reached, with D/Pd = 0.75; R/Ro = 2.0; D_2 pressure 6.2 bar; and room temperature, the Pd wire and Pt_{cal} are alternatively fed with nearly equal direct current power. The power is then stepped up and again fed to each wire, until it reaches the maximum value of 52 W in the experiment shown at Fig. 8.

The evolution of the Pd wire loading ratio with temperature (R/Ro values), when directly heating (rather than being heated by Pt_{cal}), exhibits peculiar behaviour:

Region A - low power (0→13 W): the Pd-D system is in the α+β phase; the R/Ro ratio increases almost exponentially from 2 up to a maximum of about 2.35 (orange line, Fig. 8, 9). This is in spite of the concomitant decrease of the D/Pd ratio, due to the degassing process, which naturally accompanies the temperature increase of the Pd wire. This phenomenon is not observed when the Pd wire is indirectly heated to the same temperature with the Pt_{cal} wire instead of with current in the Pd wire itself (light blue line, Fig. 8).

At present, we cannot fully explain the exponential increase of the R/Ro value when direct current is flowing. We do have some candidate hypotheses, such as the "*Preparata effect*" (2000) that predicts a sort of confinement of Deuterium inside the Pd wires due to large voltage drops along it.

In region A, no detectable excess power is observed.

Region B - power 13→20 W: massive degassing of the Pd wire suddenly occurs and the R/Ro value abruptly drops; the wire is now almost completely in the Pd-D α phase. In concomitance with the phase transition the excess power starts (Figs. 8, 9). Even though reliable values of the D diffusivity in the Pd-D α and β phases are not available, there is nearly general agreement on assigning to the α phase the higher value of deuterium diffusivity. Moreover, the effective Z* charge in the electromigration formula (1) is higher in the α phase. Accordingly, the intensity of electromigration should be significantly enhanced in this phase.

Region C - power 20→52 W: the wire is fully in the α phase; the R/Ro ratio increases because of the increase of the Pd wire temperature. Excess power is observed, increasing with the direct current power fed to the Pd wire. Excess power reaches ≥5 W when the current is highest, at 52 W (Figs. 8, 9).

The results shown in Figs 8 and 9 are typical of this series of experiments. A short summary of the main experiments carried out with 50 μm Pd wire is shown in Table A.
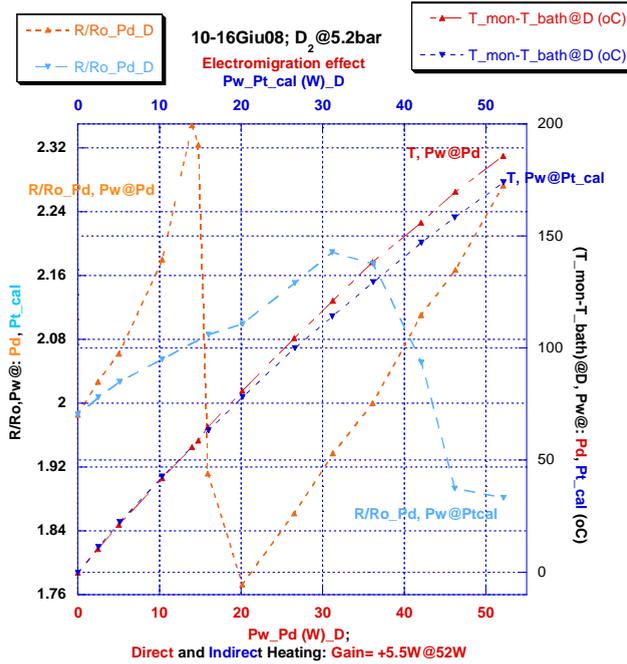
Figure 8. Pd wire nano-coated: deuterium at 6.2 bar abs. Temperature of (Pt$_{mon}$ –bath) and Pd R/Ro, versus power applied to Pd and to Pt$_{cal}$.
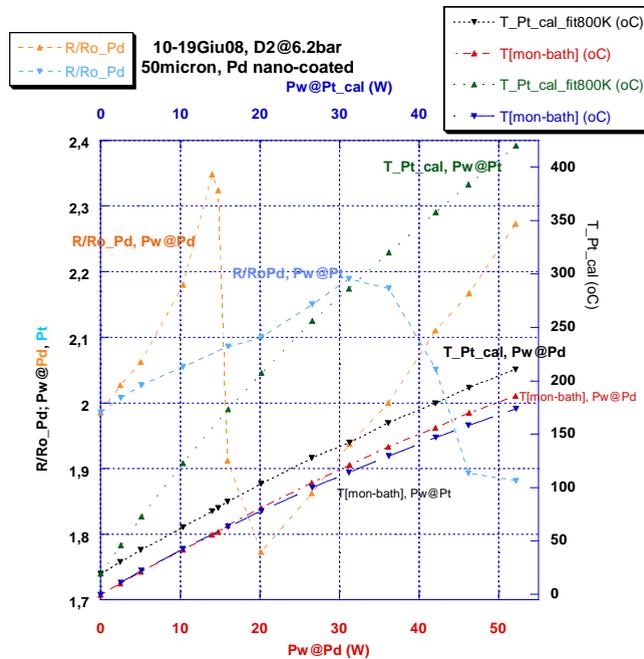

Figure 9. Pd nano-coated, deuterium at 6.2 bar abs. The curve of the Pt$_{cal}$ temperature against power (green line) is also shown. While the massive degassing (Pd wire Joule heated - orange line) happens at 170°C, determined by the relationship to the Pt$_{cal}$ green curve, the degassing of the indirectly heated Pd wire (sky-blue line) is computed based on the relationship to Pt$_{mon}$ (black line).

Table A

Summary of the main 3-wire braid experiments
Pd (99.9% purity) by J/M, Φ=50 μm, l=60-65 cm, S=0.94-1.02 cm^2, weight=14-15.3 mg; coating weight ~1 mg.

Date	Pd wire treatments	Gas type, Pressure (bar)	Input Power (W)	Excess power (W)	Notes
10 Jun 08	nano-coated Air 800°C	He, 5.2	–	No excess power (blank test)	Wire prepared on 5 May 08 and used in previous experiments.
12 Jun 08	nano-coated Air 800°C	He, 1.2	–	No excess power (blank test)	Same wire
13 Jun 08	nano-coated Air 800°C	D_2, 5.2	52	5.2 (370 W/g Pd)	Same wire
19 Jun 08	nano-coated Air 800°C	D_2, 1.5	48	3.1	Wire operative for 50 days. Wire broken after 2 hours of experiments with H_2.
30 Jun 08	nano-coated Air 800°C	H_2, 5.2	41.6	1.4	New wire. Wire broken after 1 day with H_2
02 Jul 08	Virgin	D_2, 5.2	–		Broken at loading
03 Jul 08	Air 800°C no nano-coating	D_2, 5.2	50 / 55	3.3 / 4.0	Broken after 4 days

4. Conclusions

A subtle, well-hidden systematic error is always possible but, up to now, no error has been found, by either our group or by several researchers who carefully examined our experiments and preprint. Moreover, during the ICCF-14 Conference we engaged in long discussions with several attendees: the aim was to find a mistake in the experiment. No such mistake was found.

We have shown that large excess power (about 400 W/g of Pd), at high temperatures (400-500°C), is produced when direct current is flowing in a long (60 – 65 cm) and thin (50 μm) Pd wire immersed in a pressurized deuterium atmosphere. The excess heat occurs when, because of the Joule heating of the Pd wire, a phase transition seems to happen from α+β to α phase of the Pd-D system and the interrelated diffusion coefficient of deuterium increases due to the

combined effect of both high temperature and low-deuterium concentration inside the Pd lattice.

When a thin layer of Pd nano-particles, mixed with other elements, is applied to a Pd wire surface, the wire is stabilized, preventing self-sintering (due to high temperatures) and/or self-destruction (due to localized hot-spot, anomalous thermal effects). The amount of the excess heat increases, and overall stability and reliability significantly improve.

We would welcome the opportunity to do studies of ^{4}He production as ashes of the D+D reactions, such as the measurements performed by Y. Arata [1, 3]. Unfortunately, we have not been able to do this so far, because we do not have access to the HR-QMS at the Frascati Research Center. The people operating this instrument report that it cannot be made available because of complex maintenance issues.

No anomalous thermal effects were detected in several long duration blank experiments with helium gas.

Moreover, there are indications in our other experiments (not described in this paper and only briefly discussed during the Conference) that the absolute temperature of Pd wire, by itself, has a significant effect on anomalous temperature generation. The next experimental apparatus will be optimized to detect also such effect.

Finally, the effects of the *fluxes* of active gas (e.g. deuterium or even hydrogen), through the nano-coated Pd surface and/or along the Pd bulk itself, has to be quantified by specific experiments.

Further work, especially at the engineering level, is needed to apply this new effect to a practical reactor for energy generation. The aim is to transfer the anomalous excess heat from *inside* the reactor (like in the experiment described), to *outside* for a practical use.

5. Acknowledgements

6. References

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