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**NEW PROCEDURES TO MAKE ACTIVE, FRACTAL LIKE, SURFACES
ON THIN PD WIRES**

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Abstract

In the framework of anomalous effects coming out because very close interaction of some specific gas (usually Deuterium-D, some times Hydrogen-H) with some specific solid materials (usually Palladium-Pd, some times Nickel or others) is an emerging evidence that the physical condition at the surface of the host element play a crucial role.

It has been experimentally demonstrated, by Yoshiaki Arata at Osaka University, that nanoparticles of Pd, embedded in a matrix of ZrO₂, are able to absorb extremely large amounts of H and/or D, at even room temperature and pressure.

Because of such results, we re-analyzed some of our previous experiments under the new point of view and were convinced that most of our “positive” results in Condensed Matter Nuclear Science come because of lucky, specific condition of our Pd cathode.

We decided to improve the quality of Pd, from the point of view of production of nanostructure at its surface as large (and stable) as possible, in a controllable way, using both electrolytic procedure and special preparation of Pd before the use.

Some of our efforts seemed to give positive results, although the stability at long time as to be improved.

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1 Introduction

In the framework of Condensed Matter Nuclear Science, it has been recently shown, by Yoshiaki Arata at Osaka University [1], that nano-structures of metallic Palladium, stabilized in a matrix of ZrO₂, can rapidly absorb surprisingly large amount of H₂ or D₂.

According to Arata results and to the Akito Takahashi [2] theoretical model, atomic ratios (H, D)/Pd \gg 1 can be easily accomplished at a pressure of only a few bars into Pd nano-particles, stabilized in a ZrO₂ matrix. In the Arata experiments, together with the abnormally high D/Pd ratios, a remarkable excess heat and production of 4He were detected.

On the basis of such results (and new deeper interpretation of some of our previous experimental results [3] that, although quite interesting from the point of view of experimental effects, didn't get at that time a satisfactory explanation), we are now convinced that most of the high loading ratios (H-D/Pd) and/or anomalous effects, both thermal and nuclear (by using D), obtained (generally in a not reproducible way) by the people involved in cold fusion experiments, can be reasonably attributed to the spontaneous and uncontrolled growing-up of fractal nanostructures on the Pd surface.

In our opinion, even the Sr and Cs transmutations obtained in the experiments carried out by Iwamura et al. [4] at Mitsubishi Heavy Industries, Yokohama, Japan, which occur on the surface of the Pd/Pd-CaO/Pd multilayer, could be due to the formation of fractal-like structures produced during the multilayer fabrication process.

The present work deals with experiments aimed at the production of nano-structures on Pd wires both by anodic oxidation (in situ) and air oxidation (in situ and ex situ).

2. Experimental procedures

2.1. Cells and electrolytes

The cell geometry and the experimental set-up were reported in detail in our previous papers [5,6,7,8].

Along the last year, about usual electrolytic experiments, we make 2 specific one in order to compare and cross check the results about new elements detected by ICP-MS, using light and heavy electrolytes. The cell and experimental set-up were almost the same as described in ref. 5—8. We just changed the composition of cell (from chemical glass to quartz) in the last experiment with Deuterium (b type), as detailed in [8].

Hydrogen loading: main solution 750cc (C₂H₅OH 95%, H₂O 5%); Th(NO₃)₄=5cc (with 1cc=1mg of Th in D₂O); Hg₂SO₄ = 7cc (concentration 10⁻³M, in D₂O) – Chemical glass (Schott Duran like, brand Fortuna) cell.

b) Deuterium loading: main solution: C₂H₅OD=1005cc, D₂O=89cc; Th(NO₃)₄=6cc (with 1cc=1mg of Th in D₂O); Hg₂SO₄=8cc (concentration 10⁻³M, in D₂O); NH₄OD (0.16M, in D₂O)=6cc. - Ultra pure quartz cell.

2.1.1. Procedure for the anodic oxidations *in situ*

The Pd wires were H, D loaded up the maximum of R/R₀ (H,D /Pd \simeq 0.75) by a current density of 5-10 mA/cm². The wires were then anodically de-loaded with a current density of 1-2 mA/cm² and when the de-loading reaches the point R/R₀ \simeq 1.1 or less, the current is raised to 5-20 mA/cm² and kept for a few minutes until full deloading.

2.1.2. Electrolytic Hydrogen loading after the anodic oxidation in an alcohol solution

An example of very good result, using the combined effects of previous Pd anodic oxidation, proper electrolyte composition and effect of cathodic current, is shown in Fig. 1: the loading rate is extremely high and after 300s the maximum of R/Ro is reached and surpassed. After that, the loading proceeds *still at a surprisingly high rates* up to values of R/Ro down to 1.2 (right hand side of the Baranowski curve), within $\simeq 700$ s, and down to 1.15 within 1500s. It should be noticed that without the anodic oxidation the time for loading up to R/Ro $\simeq 1.6$ —1.5 (right hand side of the Baranowski curve) is measured by the tens of hours.

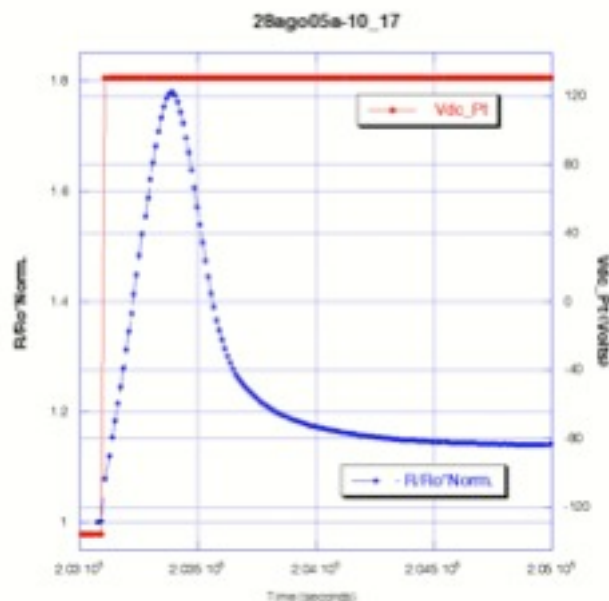


Fig. 1. R/Ro ratio, versus time, using optimized anodic oxidation and electrolyte composition. The solution is quite insulating (main liquid is vacuum-distilled&100nm filtered $C_2H_5OH + H_2O$, few salt are dissolved): the cell Voltage is 135V even at only 30mA with an Anode-Cathode distance of 4cm.

These results indicate that a proper anodic oxidation produces a very active surface. On the basis of Arata experiments, such activity could be explained as due to the formation, just at the surface of the wire, of something similar to Arata's true nano-particles (embedded in a matrix of ZrO_2).

2.1.4. Electrolytic Deuterium loading after the anodic oxidation

Generally, the loading experiments carried out with electrolyte b-type showed lower loading rates and lower maximum (final) loading with respect to the ones with electrolyte a-type. Typically the maximum loading didn't exceed the one correspondent to R/Ro $\simeq 1.6$ (right hand side of the Baranowski curve). Anyway, deuterium based electrolyte some times showed anomalous effects like excess heat and “new” elements as detected by ICP-MS (routinely) and (sometimes) SEM micro analysis.

2.1.5. ICP-MS results

In consideration of the fact that all our previous electrolytic loading experiments in both a) and b) electrolytes, included anodic de-loading cycles, i.e., anodic oxidation, we

routinely examined electrolytes, wire and sediments in order to check whether some transmutations had occurred.

The main motivation of ICP-MS analysis was the results of Sr to Mo transmutation, shown in a very elegant experiment, by Yasuhiro Iwamura team (at Mitsubishi Heavy Industries, Yokohama, Japan), in 2002 [4]. We recall that, at that times and since 1998, we were using Sr as main electrolyte in our heavy water and/or heavy ethyl alcohol mixtures. We detected [9] some Mo in our Sr based electrolyte experiments, confirming the Iwamura results using a very different environment (liquid electrolyte instead of pure and pressurized Deuterium gas).

The latest results about main new elements, detected by a high resolution ICP-MS, are reported in Tab. 1 and were presented at ICCF6 [8]. See Table 1 comments for further details.

Table 1. Main new elements detected in light alcohol-water (Scott Duran type) and heavy alcohol-water (quartz) cell. All the reagents $Th(NO_3)_4$ (at 1mg/ml concentration) and Hg_2SO_4 ($10^{-3}M$) are in D_2O . BKG and reagents subtracted. 1 Count = about $5E10$ Atoms. In bold characters are reported value of new elements, about Deuterium experiments, larger than a factor 10 in respect to Hydrogen one.

Element (isot. ab. %)	H loading Exp. Counts	D loading Exp. Counts	Comments
P	0	6.4E6	BKG=4E3
³⁹ K	0	1.8E7	BKG=1E6
Cu	2.3E6 63/65=2.20	2.5E7 63/65=2.11	Nat. 63/65 =2.25
Zn	6.2E6	4.9E7	
Rb	3.1E4	8.2E4	
¹⁰⁸ Pd (26.5%)	Normal	Depleted of 5%	
Ag	6.5E4	2.6E5	
¹⁴⁰ Ce(88.5%)	1.86E4	1.31E5	
W	1.16E4	4.42E4	
Tl	80	900	
Pb	4.5E5	1.38E7	
U	1.1E3	1.0E4	
¹⁹⁵ Pt(33.8%)	4.3E7	10.8E7	Marker Anode dissolution

2.2. Deuterium self-loading after electrolysis and SEM analysis

In many of the previous electrolytic D loading experiments, after some anodic oxidation cycles had been carried out, we found evidence (although not fully reproducible) of spontaneous deuterium self-loading, i.e. without applied electrolytic current, of the Pd wires immersed in the electrolyte. The deuterium was absorbed from the gas dissolved in the solution due to previous electrolysis. This assertion is demonstrated by the fact that the value of the loading is reversibly controlled by controlling the concentration of the dissolved D_2 through alternatively bubbling Ar / D_2 , as shown in Fig. 2.

In Fig 2, at the time 269500s, the anodic stripping was ended (the current, from $-9mA$ was returned to $0mA$). Almost immediately, the wire started to absorb the deuterium dissolved in the solution and the R/Ro increased from about 1.05 to about 1.405. At the time 273500s large amount of Argon gas was introduced, by bubbling it inside the solution. Immediately, the R/Ro started to decrease. At the time 275500s, after the R/Ro decreasing (because of Argon effect) from 1.405 to 1.375, D_2 gas was added again. The R/Ro value

again increased, showing reversible and controllable effects. At the time 277300s, when the R/Ro increased to 1.45, the intake of D₂ was stopped and Argon was added again. At the end, another anodic de-loading was carried out (at -6mA) in order to fully de-load the wire.

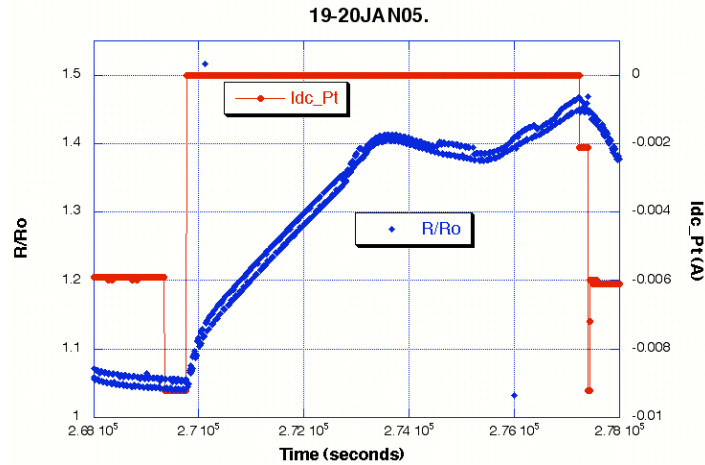


Fig.2. R/Ro versus time (seconds) with the effects of D₂→Ar→D₂ intake and anodic stripping current (A). The values of R/Ro are “twin” because we introduced the procedure to measure also the Resistive Thermal Coefficient (RTC), see later.

Moreover, observing Fig. 3, we would like to stress that there is unquestionable experimental evidence that the spontaneous self-loading following anodic oxidation reaches values of D/Pd over the well-known thermodynamic limit. In other words, we expected a R/Ro ratio less than 1.8 but we observed a value much larger, close to 1.95-2.0. Such value is equivalent to a deuterium pressure of about 8 ÷10 times larger than 1bar i.e. the D₂ pressure used in the cell (e.g. time 287000s--300000s).

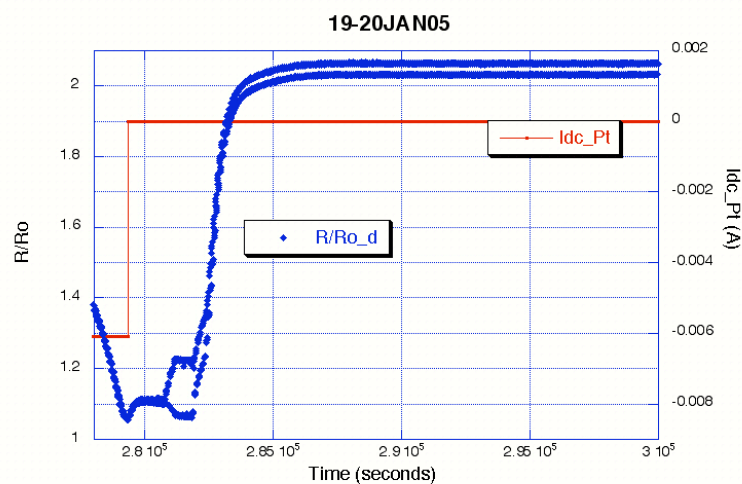


Fig. 3. R/Ro versus time. R/Ro is about 2 instead of 1.8 expected. Other comments like Fig. 2.

In Fig. 3 it is shown the spontaneous loading of deuterium gas dissolved in solution (time 282000s → over 300000s). At the time 279000s the anodic de-loading was ended in an Argon atmosphere. At the end of the anodic cycle a weak self-loading was observed. Later (time 281000s) Argon was again added for calibration purposes. At the time 282000s Deuterium gas (at about NTP, overpressure of only 50mbar) was finally added and self-loading started immediately.

The R/R₀ values are >2.0 (time 290000s) because of the effect of residual stress (R/R₀ at D/Pd=0 is increased to about 1.05 with respect to 1.00 at the time “0” at the beginning of the experiment). In other words, the wire was de-loaded (at the time 282000s) and intentionally we didn’t correct (by mathematic calculation) the value in such experiment because we decided to “monitor” the value of the residual stress vs. time.

2.2.1. SEM micro-photography and microanalysis

In Fig. 4, 5, 6 are shown some pictures, by SEM micro-photography, of Pd wire before (Fig. 4) and after (Fig.5, Fig. 6) D electrolytic loading.

Elemental analysis (by microprobe) of some «white» area of Fig 6 are shown in Fig. 7.

The Figs. 4—7 are the same as presented at JCF6.

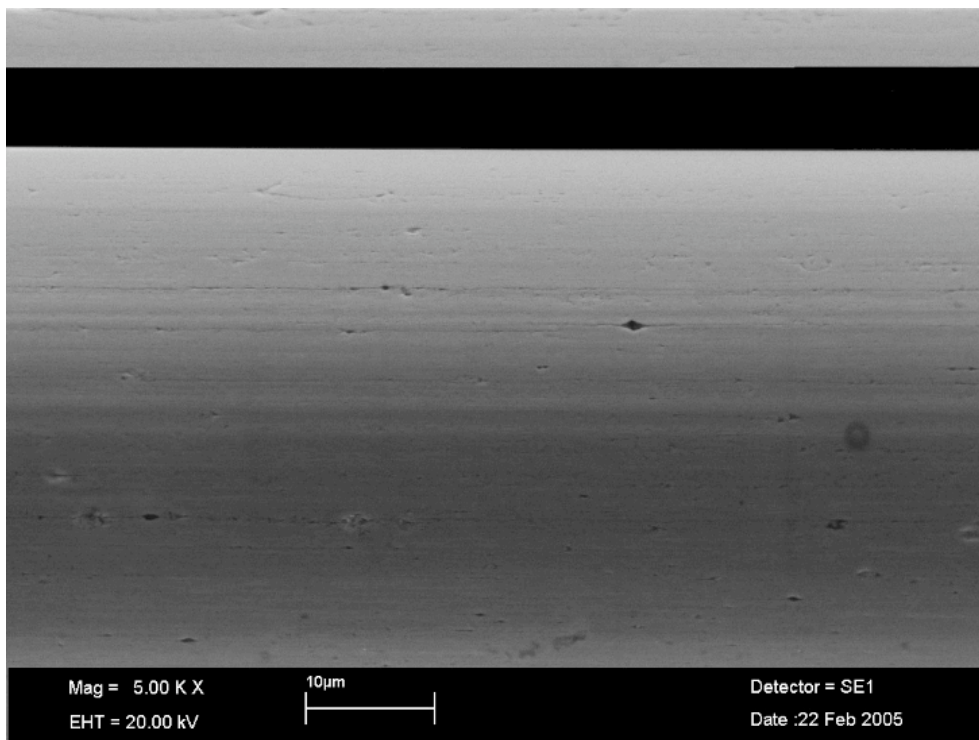


Fig. 4. SEM picture of Pd virgin wire, before electrolysis. The wire is contiguous to used one of Fig. 4.

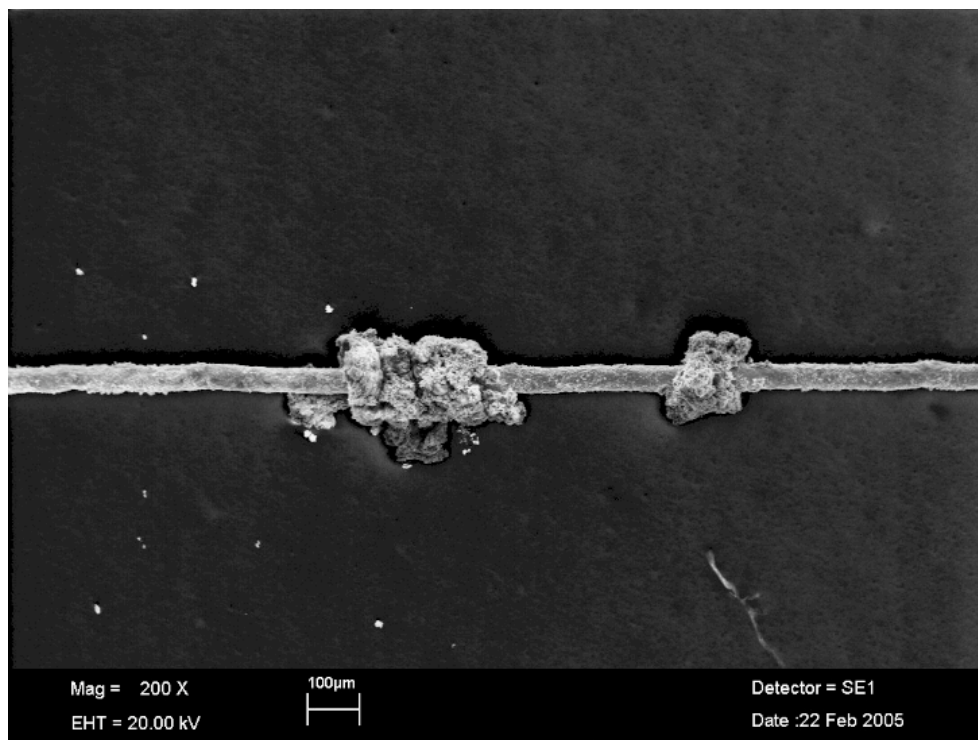


Fig. 5 SEM picture of a used wire, heavy solution electrolysis. The “new” elements and Pt deposit (from anode) are concentrated in few spot area (about 15% of wire length).

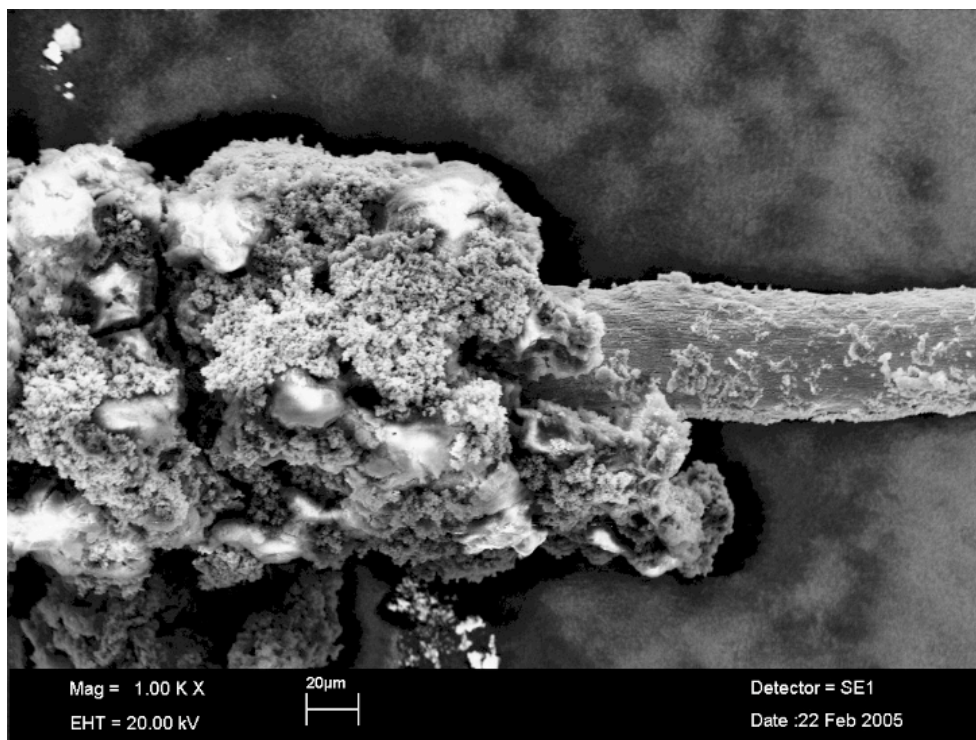


Fig. 6. Details of deposits as reported in Fig.5.

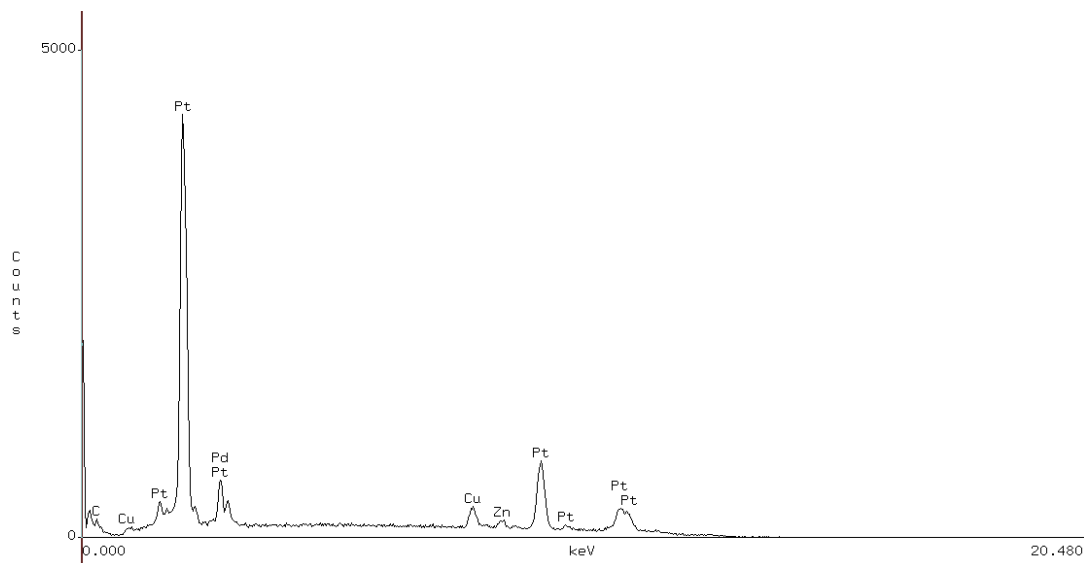


Figure 7. SEM elemental analysis of one of “white” area has shown in Fig. 5. The, unwanted, large deposition of Pt at Pd surface is clearly detected. Moreover, also Zn and Cu, detected by ICP-MS analysis, are re-confirmed.

The wire analysed was about 10cm long out of 60cm total. The new elements are concentrated within about 15% of the total length, in a random like distribution.

It is clearly shown the problem coming out from anode dissolution and its deposit at Pd cathode surface. Such deleterious effect, in respect to H and D absorption in Pd, is not yet solved (despite our deep efforts). We recall that, unfortunately, the over-voltage of H,D at Pt surface is close to zero. We are convinced that one of most important reasons of poor Deuterium loading into Palladium, as reported from several Researchers, is very often the Platinum coating (from the anode) at Palladium cathode surface, specially at large current density (over 500mA/cm² in water solution).

It is interesting to note that, apart sensitivity, the same elements are detected from both SEM and ICP-MS. In other words, the ICP-MS results are “safe” and aren’t instrumental fakes.

2.3. Air oxidation of Pd and coating

As we have shown, in our electrolytic loading experiments, we observed the phenomenon of spontaneous deuterium loading, i.e. without the application of any electrolytic current (self-loading). After several years of observations and specific tests we realized that large self-loading occurred only at the end of certain anodic cycles, in specific electrolytes. In particular, we found that the self-loading effect obtained with water electrolytes was almost negligible ($R/R_o \approx 1.1$), while with hydro-alcoholic electrolytes, both a) and b) types, the effect was higher. By optimizing the anodic cycle it was possible to reach self-loadings up to $R/R_o = 1.75$ (left hand side of the Baranowski curve) with H₂ and $R/R_o = 1.95$ (left hand side of the Baranowski curve) with D₂.

SEM micro-photography of the Pd wires, which presented strong self-loading effects, show the presence of thin porous structures on the surface of the wire. It was also observed that during the anodic cycles in hydro-alcoholic electrolytes a significant diffused pitting corrosion occurs both of Pd and Pt electrodes. ICP-MS revealed a consistent presence of Pt

in the electrolyte, in the sediments and on the Pd surface. These facts suggested that in such electrolytes the anodic cycles produced strong oxidations of the Pd (useful) and Pt (deleterious, see previous discussion about over-voltage) wires.

These considerations led us to try a high temperature air oxidation of Pd wires as an alternative and more practical mean for obtaining active palladium surfaces. Since the first trials we found out that this simple procedure is scarcely effective. As a matter of fact it produced slight and unstable self-loadings and only for once.

We considered that during the anodic cycles, in addition to anions, negatively charged particles are electrophoretically deposited on the Pd electrode and that this effect is enhanced in an alcoholic environment, so we thought that for a significant self-loading to occur, the presence of an impurity layer, intimately adherent to the oxidized surface, might be a necessary condition. So we resorted to artificially apply such impurities to the oxidized Pd wire in the form of colloidal silica.

2.3.1. Procedure for the preparation of the samples

A thin (50 μ m diameter) and long (60cm) Pd wire was Joule heated in air (\cong 600mA for 60 seconds) at about 700°C in order to get a thin layer of PdO.

After such a heat-treatment the wire was immersed into a diluted solution of colloidal silica, and then heated \rightarrow cooled \rightarrow heated \rightarrow cooled again several times.

2.3.2. Measurements

Virgin Pd wires (no treatment) exposed to D₂ gas at 1atm and ambient temperature, usually reach a maximum loading, corresponding to an electric resistance ratio $R/R_0 \approx 1.82$, in over 2 days. (Fig.8).

The wires, treated according to the reported procedure, exposed to Hydrogen or Deuterium in the same condition, instead, showed extremely fast, and quite larger, loading rates.

In Fig. 9 is shown the case of a wire exhibiting an astonishingly high loading rate: the electric resistance ratio (R/R_0) of the wire exposed to a 1atm D₂ gas, at 24 °C, reached the value of 1.90 in about 100 seconds.

The maximum R/R_0 value of 1.98 ($D/Pd \cong 0.75$), was reached two minutes later, and remained stable for a long time. Further details, by the authors of this paper, are shown in the JCF6 proceedings.

It was also found that if during the treatment procedure of the wires, the step involving the immersion in colloidal silica is omitted, the loading rates are significantly lower and after a couple of hours R/R_0 starts to decrease (de-loading).

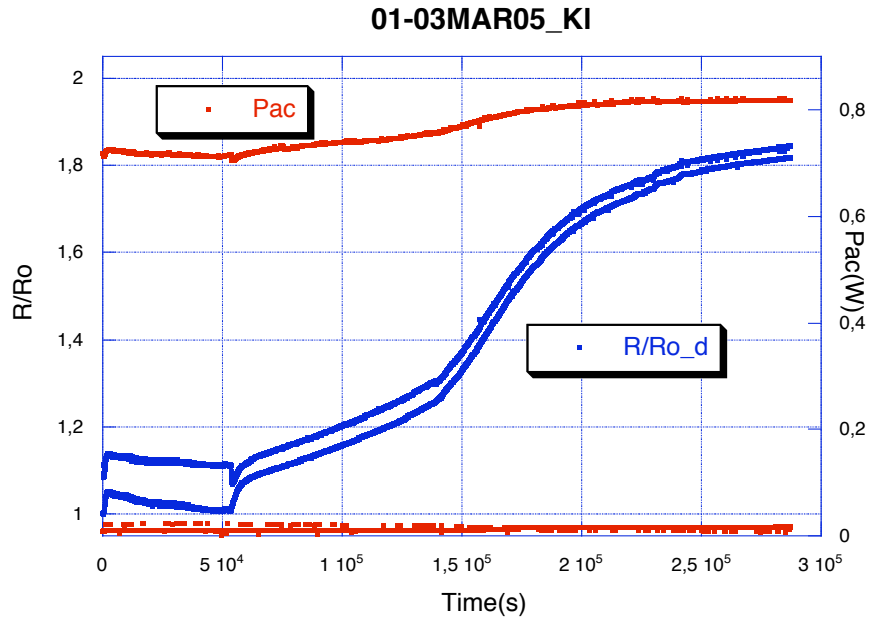


Fig. 8. Loading characteristic of a virgin Palladium wire: loaded at room temperature with 1bar of Deuterium gas. In order to “activate” the Pd surface, it was made a preloading cycle (from time 0 to about 60000s) with D_2 gas, 293K, 1 bar and later allowed to decrease, very slowly, the Deuterium content in the cell. Even neglecting the activation time, the time needed to reach the thermodynamic limit of $R/R_o=1.82$ is as large as 220000s. Such values (i.e. R/R_o and time) are in agreement with data usually reported in the literature. As standard in our procedure from about 2 years, we cyclically inject electromigration ac current (square wave, 10KHz) with low (about 10-15mW) and high power (700-800mW) in order to calculate the Resistive Thermal Coefficient versus D/Pd ratio.

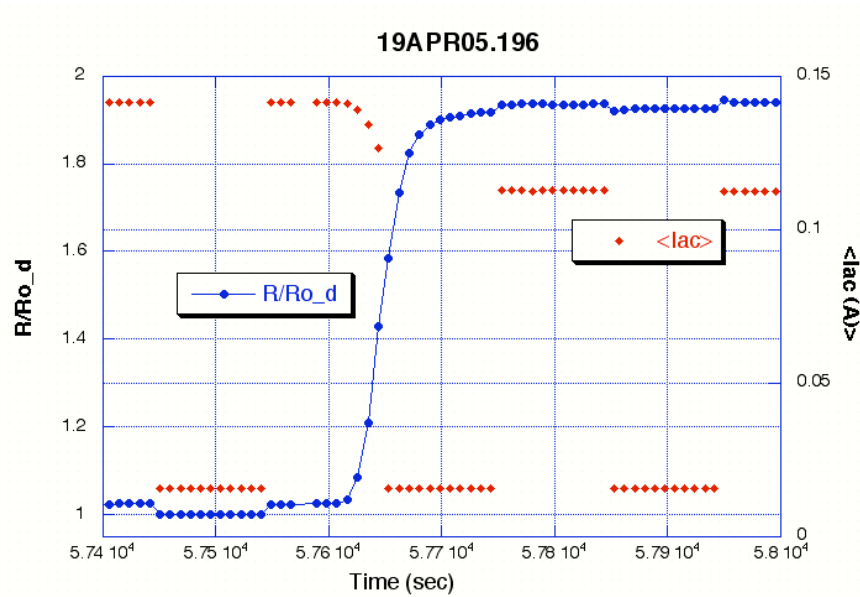


Fig. 9. Loading ratio, versus time of a Pd wire treated by oxidation and silica. D gas loading (1bar).

3 SAFETY rules-procedures

The following procedures have to be *carefully* followed to avoid **EXPLOSIONS** because possible mixing of Hydrogen (and or Deuterium) with Oxygen and highly catalytic effect of Palladium surface, activated because growing of nano-structure at its surface.

We remark the safety procedures are quite easy in our experimental set-up because we take fully advantage of our procedure to measure both the R/Ro value and the RTC (Resistive Thermal Coefficient) of Pd wire versus loading (see our reports at, Asti5, ICCF11 and JCF6 for further details). In such a case, we used the ratio between the R/Ro value made by injecting alternatively low (about 15mA equivalent to about 10mW of power dissipated, called R/Ro_L) and high (about 120mA, equivalent to about 700mW power dissipated, called R/Ro_H) AC current along the thin Pd wire. Because very different thermal conductivity of gas was adsorbed (H₂, D₂, He, Ar, Air, “vacuum”), after proper calibration once for ever, it is very easy to understand the real composition of gas atmosphere inside the cell.

3.1. Typical situation

New Joule heating of the wire (0.05mm diameter) that previously was H and D loaded.

The aim is *in situ* new oxidation by air to get fresh PdO for further tests.

The procedure is detailed, step by step, as following.

1) Intake, and flux, Ar gas.

* Measure continuously R/Ro_L and R/Ro_H.

* End the large intake of Ar only when the ratio between R/Ro_H and R/Ro_L increases largely (and stabilize) due to the lower thermal (at 0°C, 1.013 bar) conductivity of Ar (16.36 mW/m*K) in respect of H₂ (168.35mW/m*K) or D₂ (130.63mW/m*K)

2) Increase, slowly, the DC I_{em} (I_{em}), from 0 to 350mA, keeping Ar flux at low values.

3) Observe deloading.

4) Wait for complete deloading (10-30minutes, depending on wire coating). Decrease I_{em} from 350 to 0mA.

5) Check R/Ro_L value: if close to 1.0, go to step 6, otherwise wait until full deloading by injecting I_{em} again at large value (350mA).

6) Intake of He for test: the ratio of R/Ro_H versus R/Ro_L as to decrease largely in respect to Ar filling, due to the larger thermal conductivity of He (142.64 mW/m*K) in respect to Ar.

7) Make vacuum (e.g. oil-free membrane pump, limit about 10mbar) to pump-out He.

8) Intake of **dry air** (thermal conductivity, at 0°C 1.013 bar, 23.94 mW/m*K) at STP. I_{em}=0mA. Measure R/Ro_L only.

- 9) Increase I_{em} from 0 to about 600mA. Wait 1m at 600mA. Observe the R/Ro instantaneous increasing due to large I_{em} : R/Ro about 3. At R/Ro=3 the wire temperature is about 700°C.
- 10) Decrease slowly (in about 2m) I_{em} from 600mA to 0mA. Repeat the cycle 3 times.
- 11) Check the R/Ro value and recalibrate the value, if necessary.
 - 11a) Reintroduce the R/Ro_L and R-Ro_H cycles.
 - 11b) If all was correct, the values of R/Ro_L and H, in air at NTP, are respectively: 1.00, 1.13 (input current of about 15mA and 120mA).
- 12) Make vacuum and check if the values of R/Ro_L and R/Ro_H have the proper values as expected. They are used the calibration data obtained at the beginning of the experiment.
- 13) Wash the cell with Ar or He. Later on, make vacuum again.
- 14) Intake of H_2 or D_2 and observe loading and cell temperature. By the way, we observed that some residual He gas, at the order of 1%, instead of usual Ar, has a beneficial effect about both maximum value of loading and anomalous effects generation. Such phenomena as to be more deeply investigated.
- 15) Stop immediately any current, and inject/wash with He or Ar, if cell temperature and/or pressure increase in an anomalous way.

4 Conclusions

According to us, almost all of positive experimental results (excess heat, transmutation, particle emissions) in Cold Fusion experiments (Fleischmann, Takahashi, Arata, Iwamura, Mizuno, Celani, Mc. Kubre, Swartz, Storms, Preparata-De Ninno-Del Giudice, Miley, Violante....) can be **RATIONALISED** as the effect of nano-structures at the Pd surface.

It is very difficult to produce such nano-structures (almost all, except Arata, Iwamura, Celani) obtained just by chance.

Moreover, such nano-structures are not stable over time.

We begin to develop a **SIMPLE** procedure to obtain nano-structures, both by electrolysis (routinely produced also during the usual cathodic regime: specific electronic circuit and procedure developed from our Group since 2004, patenting) and by addition of colloidal silica on oxidised Pd surface (since March 2005, gas loading). In some aspects, the latter method is similar to Yoshiaki Arata procedure that was the Pioneer about nano-particles production for Cold Fusion studies. The kind of colloidal silica (5-10nm dimension, low Na content), that up to now gives the best results, was specially developed (after 5 years of efforts), by an Italian chemical company, according to our request.

We think that the nano-structure interpretation of anomalous effect in deuterated metals will bring soon to some technological device: boiler using liquid electrolyte, even some turbine engine using pressurised high temperature gas loading method.

More systematic work is necessary in order to elucidate all the possibilities that come because nano-size materials (specially pure Pd and/or Pd based alloys) properly coated/embedded by a third element.

Finally, as shortly shown also during ICCF12, we get indications that *some anomalous heat* seems coming out, from a new pressurised (4bar) cell (SS), using even the Hydrogen gas when the temperature of the wire, heavy coated by colloidal silica, was forced to reach about 200°C (by Joule heating).

Acknowledgments

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We can't forget that the pioneer work of Prof. Arata about nano-particles (starting with the use of "palladium black" at different grain sizes) which opened our eyes about new interpretation of some of our old data that never get satisfactory explanation.

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