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Research Article

Are Oxide Interfaces Necessary in Fleischmann–Pons-type Experiments?

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Abstract

The generation of excess power in a palladium deuteride electrochemical system is often difficult and time consuming. Long incubation times, on the order of weeks or months, are necessary presumably for the basic electrolyte to dissolve and redeposit essential impurities onto the cathode surface. To accelerate this process, we added chemical additives to the electrolyte once the palladium was loaded with deuterium. Chemicals that produce oxide interfaces on the palladium surface seemed to occasionally produce apparent excess power. A single $Pd_{90}Rh_{10}$ cathode generated a total apparent excess energy on the order of 10 kJ after a series of additions in an experiment that only took one week. The results are encouraging and may lead to an understanding of what triggers excess power production in Fleischmann–Pons-type experiments. A hypothetical model describing the possible role of oxide interfaces is described..
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Keywords: Chemical additives, Deuterated palladium cathodes, Electrochemical, Excess power

1. Introduction

Since the initial publication by Fleischmann and Pons [1], numerous accounts of excess power production in palladium cathodes electrochemically loaded with deuterium have been reported [2]. Research at SRI International (SRI) initially identified three criteria necessary for the excess power generation: (1) high cathode loading with a D/Pd ratio near one, (2) maintenance of high loading for considerable periods of time and (3) an interfacial current density above a critical threshold [3]. Later, other conditions were also thought to be important for the successful initiation of excess power

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production such as the presence or absence of certain impurities and the application of external stimulation to produce non-equilibrium conditions [4,5]. Although these factors were recognized by the community, it remained difficult to achieve high loading in Pd cathodes and irreproducibility of excess power production in Fleischmann–Pons-type electrochemical experiments continued to plague the field.

A number of technical advances occurred during the last decade. In 2003, Dardik (Energetics Technologies (ET), Israel) reported on the use of SuperWaves^{TM} excitation to accelerate the rate and extent of electrochemical loading of deuterium into Pd cathodes [6]. When SuperWaves^{TM} were used in lieu of D.C. current to drive electrolysis experiments, a significant amount of excess heat was generated in three ENEA-produced palladium cathodes [7]. These results, presented at the 11th International Conference on Cold Fusion (ICCF11) in 2004, remain among the largest reported to date.

Violante's group (ENEA, Italy) focused on the metallurgical aspects of Pd that led to high deuterium loading. For a particular batch of commercial material, they initially defined a process consisting of cold rolling and annealing that produced an optimized Pd metallurgical structure that reduced the internal stresses and concentration gradients generated in the material during deuterium loading. With the appropriate metallurgy, it was shown that the extent of deuterium loading increased, loading to high D/Pd ratios at relatively low current densities was easier, the reproducibility of excess heat production and the magnitude of excess heat generated were enhanced [8]. At ICCF 14 in 2008, further results on the systematic characterization of the metallurgical properties of Pd cathodes in experiments at ENEA, SRI and ET were presented [9–11]. From the more recent studies, the ENEA group identified additional features necessary for successful excess heat production. These included (a) a spectrum of Pd contaminants that give rise to grains with a nominal size on the order of 100 μm with predominately $\langle 100 \rangle$ crystal orientation after annealing and well defined grain boundaries following a chemical etch in aqua regia, and (b) a textured Pd electrode surface morphology showing pseudo-periodic patterns with a period in the range of tenths of microns.

Because of these developments, NRL embarked on an experimental program in collaboration with ENEA, ET, and SRI. The objective of the effort was to further explore excess power generation in deuterated palladium electrochemical systems with the goal of trying to understand the nature of the palladium interface and its role in the production of excess power. In this paper, the results from over 100 calorimetric experiments are presented on Pd foil cathodes electrochemically loaded with deuterium in thermodynamically closed electrochemical cells under either constant input power or constant current conditions. The experiments were driven by both D.C. current and superwave excitation.

Generally, long incubation times, on the order of weeks or months, are thought to be necessary to observe excess power, although loading of thin electrodes to high levels of deuterium takes hours. During this extended time, the electrochemistry presumably is concentrating some essential impurities onto the cathode surface. We chose to speed-up this process by making intentional chemical additions while the cells were running and after deuterium loading had occurred. Excess power was not observed in the majority of experiments described until elements known to form oxides (and possibly magnetic interfaces) in highly basic solutions were added to the electrolytes. The effect of the additions on excess power production in Pd cathodes is reported.

2. Experimental

Two types of calorimeters were used: flow calorimeters designed by ET and SRI and a four-position heat-conduction calorimeter (Hart Scientific) described previously [12]. In both, closed electrochemical cells were employed with 50 μm thick palladium foil cathodes obtained from different commercial sources and two 99.9% platinum foil anodes in a symmetric "sandwich" configuration. Most Pd cathodes were processed (rolled, annealed and chemically etched) using procedures optimized at ENEA for high loading and excess power production [8,11]. Additional Pd cathodes were custom-fabricated to produce morphologically interesting surfaces. The heavy water electrolyte was either 0.1 M or 1 M LiOD freshly prepared in a glove bag prior to each new electrochemical experiment by dissolving lithium

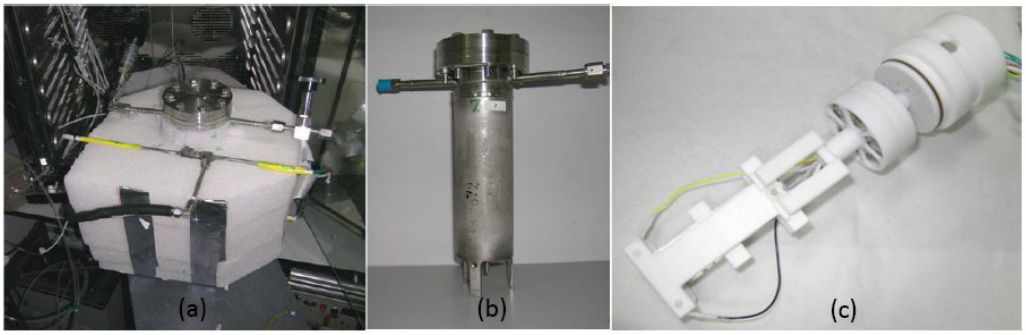

Figure 1. Photographs showing (a) ET flow calorimeter, (b) Teflon-lined stainless steel cell, and (c) all-Teflon interior cell parts.

foil (Johnson Matthey, 99.9%) or lithium deuteride (Aldrich, 98 atom % D) in deuterium oxide (CIL, Aldrich, Alfa Aesar). Light water electrolyte (0.1 M LiOH) was made from lithium deuteride and distilled water. High surface area catalysts, 0.5% Pd on alumina (Sud-Chemie) and Pt electrode on carbon fiber paper (Johnson Matthey), were used to recombine the evolved deuterium/hydrogen and oxygen gases. Chemical additives were reagent grade and used without purification. The chemicals were added to the electrolyte (24 mL volume) in the Hart cells in 5–10 mg quantities. Electrochemical experiments were usually operated in the galvanostatic mode, but some experiments were conducted with SuperWaves^{TM} excitation and some with constant input power. Experiments in the ET calorimeters were run for 1–3 months while those in the Hart calorimeters were carried out over approximately a 1 week period. No measurement of D(H)/Pd loading was made in any of the experiments because a four point probe measurement is a possible source of extra power.

Teflon-lined stainless steel cells (5 cm internal diameter, 23 cm length), with spiral cooling water pipes embedded in the walls, were used in the ET flow calorimeters. A flow calorimeter, cell and all-Teflon interior cell parts are shown in Fig. 1. Two opposite outlets with VCR connectors are located at the top of the cell. A pressure transducer and a pressure relief valve are attached to the cell via one of the connectors. The second connector, for coupling to a mass spectrometer for He detection, was plugged in our work. The sealed stainless steel cell was covered with Rohacell foam for thermal insulation and two tubes that connected to the cooling water pipes passing around the exterior of the cell were inserted through the foam. The inlet and outlet temperatures of the water were each measured with two PT-100 platinum resistance temperature devices (RTDs) inserted into the tubes attached to the cooling water pipes. The cooling water temperature was maintained at 5.00 or 20.00 $\pm$ 0.01°C using an external chiller and the entire calorimeter assembly was held at a constant temperature (8 or 22°C) in an incubator with a thermal stability of $\pm$0.1°C. The mass flow rate of the cooling water was determined using a Bronkhorst mass flow controller. Experiments carried out in three ET cells/calorimeters used a Pd cathode (80 mm $\times$ 10 mm $\times$ 50 μm) between two Pt foils (80 mm $\times$ 20 mm $\times$ 110 μm) and a cathode–anode spacing of 5 mm. Teflon-coated Pt wires were spot-welded to the electrodes and electrical connections inside the cell and to external instrumentation were made via multi-pin connectors located on the sealing flange.

Teflon cells (25 mm internal diameter, 13.5 cm length) based on the ENEA/Violante design were used in three of the four available positions in the Hart calorimeter. Photos of the Hart calorimeter, the anodized aluminum cell holder, internal cell parts and Teflon cell are presented in Fig. 2. The electrode dimensions for the Hart cells were: cathode (40 mm $\times$ 10 mm $\times$ 50 μm), anode (45 mm $\times$ 20 mm $\times$ 180 μm) and cathode–anode spacing of 5 mm. Teflon-coated Pt wires were attached by spot-welding to the electrodes and to Pt wires extending through the PEEK cell top. Two SS

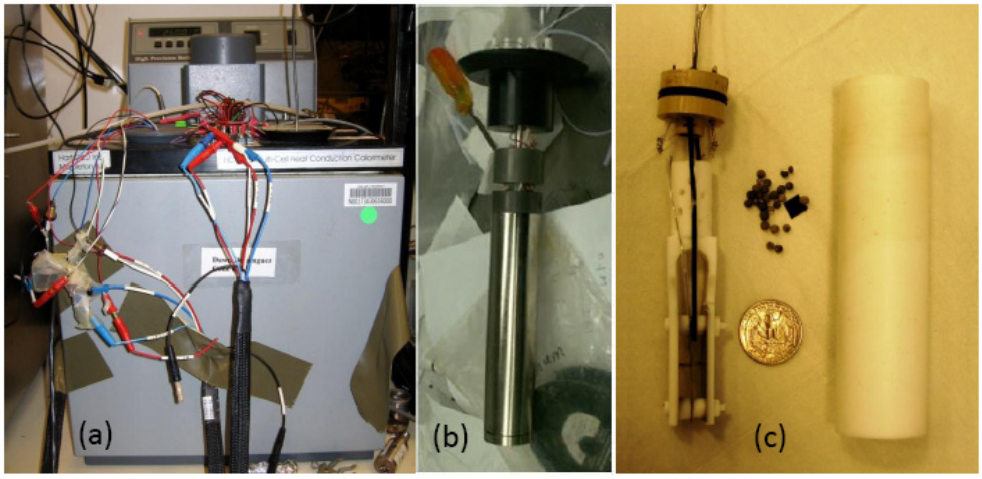

Figure 2. Photographs showing (a) Hart calorimeter, (b) anodized aluminum cell holder, and (c) internal cell parts and Teflon cell.

syringe needles were inserted through the PEEK top to allow chemical additions to the electrolyte. Black polyethylene tubing was attached to one of the syringe needles so that electrolyte could be drawn into the syringe from the bottom of the cell. The Hart calorimeter was typically maintained at temperatures of 20.00, 21.10, 25.00 or 40.00°C in a water bath with a thermal stability of ±0.001°C.

Scanning electron microscopic images were acquired on a Carl Zeiss SMT (formerly LEO) Supra 55 instrument. It is a Schottky thermal field emitting microscope capable of high-resolution imaging at primary beam voltages from 200 V–30 kV. X-ray diffraction measurements were made on a high-resolution 250 mm Rigaku diffractometer using Cu-Kα radiation from a 12-kW rotating anode generator. X-ray photoelectron spectroscopy (XPS) analysis was run on a Thermo-Scientific K-Alpha XPS instrument.

3. Results and Discussion

Table 1 shows a listing of the different cathode materials investigated in both the ET and Hart calorimeters and the number of electrochemical experiments performed in each. As seen from the Table, 63 cathodes produced from eight commercial sources of Pd were examined in heavy water electrochemical experiments through the end of 2010 and 15 cathodes from five commercial sources of Pd were used in light water experiments. In addition, 34 heavy water experiments included some custom Pd foils produced in our laboratory as well as some other metal foil cathodes. Finally, one Ni/LiOH experiment and two experiments with Pd foil cathodes in KOD and H_2SO_4 electrolytes were carried out.

Pd from three, Engelhardt, Platexis and Holland Moran, of the eight commercial sources of Pd used in the heavy water experiments, were acquired prior to 2005. The impurities present in these materials differed significantly from those obtained more recently. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analysis showed that the older materials contained substantial quantities of rhodium (12–31 ppm) and platinum (30–190–ppm) whereas the

Table 1. A listing of cathode materials used in electrochemical experiments performed in the ET and Hart calorimeters from January 2009 to December 2010. The foils highlighted in bold faces were used in the initial experiments where no chemical additive was added to the electrolyte

	ET	HART	Total
Pd/LiOD			
Engelhardt	**2**	**8**	
Platexis	**9**	**7** + 2	
Holland Moran	**1**		
ESPI	**7**	**1** + 5	
Johnson Matthey	**1**		
Goodfellow	2	**2** + 14	
Alfa Aesar		**1**	
G&S		1	
Total	22	41	63
Pd/LiOH			
ESPI	**2**	**3** + 1	
Johnson Matthey	**2**		
Goodfellow		3	
Alfa Aesar		**2**	
G&S		2	
Total	4	11	15
x/LiOD			
Pd/0.25% B		**1** + 1	
Pd/0.75% B		2	
Pd–C nanofoam		**1**	
Pd nanoparticles innanoporous Au		**1**	
Nanoporous Pd–Co		**1**	
Pd/5% Ru		2	
Pd 98%/Pt 1%/Rh 1%		2	
Pd 90%/Rh10%		1	
Ni/Pd		2	
Ni/Pd/Ni		1	
Ni		3	
Nb		3	
Ta		2	
Pt	**6**	5	
Total	6	28	34
Misc			
Ni/LiOH		1	
Goodfellow Pd/KOD		1	
Platexis Pd/H2SO4		**1**	
Total		3	3
Grand Total			115

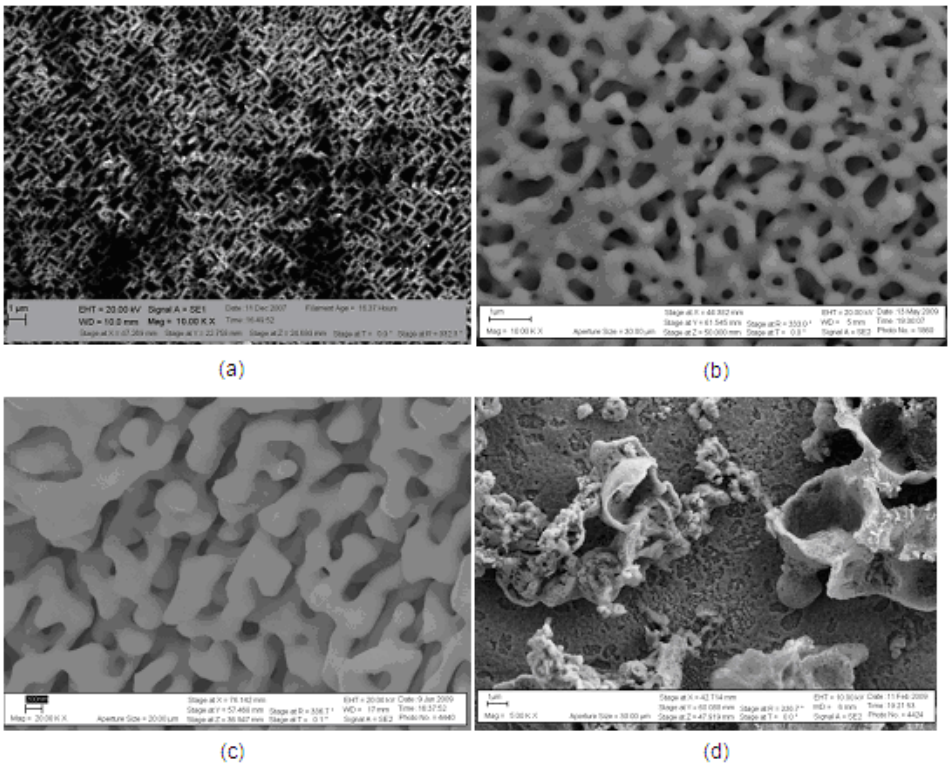

Figure 3. SEM images showing surface morphology of selected cathode foils as follows: (a) ET experiment 64a Pd at 10 k× (1 μm scale bar), (b) electrochemically de-alloyed $Pd_{20}Co_{80}$ at 10 k× (1 μm scale bar), (c) chemically de-alloyed $Au_{30}Ag_{70}$ at 20 k× (200 nm scale bar) and (d) surface oxidized/reduced Pd at 5 k× (1 μm scale bar).

newer materials had less of these elements, but included higher amounts of zirconium, yttrium and hafnium [13]. The different impurity profiles of the materials likely reflect alterations in Pd processing outcomes that occurred after 2005.

The Pd foil cathode used in ET experiment 64a is the "gold standard" for the production of excess power (25 ×) in Pd foil cathodes [7]. The crystallographic orientation of the foil was mostly ⟨100⟩. The morphology of the foil was characterized by grains 100s of μm in size and a well-defined "2D grating" surface structure after etching. Attempts to make cathodes with similar surface morphology were undertaken during the course of our work. Examples of some of the cathode surfaces produced are shown in Fig. 3. Presented in the figure (and included in Table 1) are an electrochemically de-alloyed Pd-Co material, a chemically de-alloyed Au–Ag material and a surface oxidized/reduced Platexis Pd foil. Other techniques used for cathode development include surface templating and ion implantation (not shown).

Highlighted in Table 1 are 59 cathodes that failed to produce excess power in our earliest calorimetric experiments. These experiments were carried out without the addition of chemical additives to the electrolyte. Of these, 15 cathodes (nine Pd cathodes run in light water electrolyte and six Pt cathodes run in heavy water) were run as controls and were not expected to generate excess power. Experiments with 43 of the other 44 early Pd cathodes were carried out in LiOD

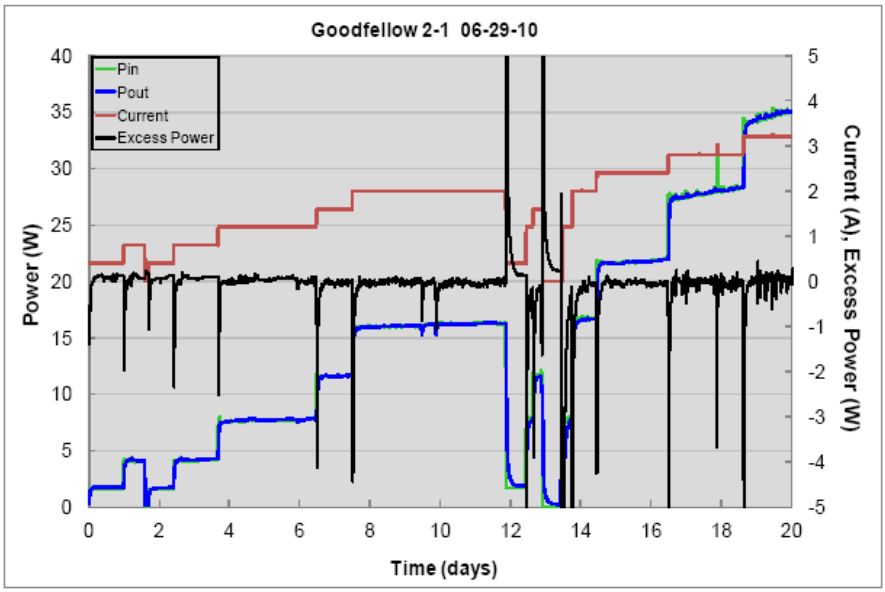

Figure 4. Plots of input power, output power, current and excess power as a function of time for the first 20 days of a Goodfellow Pd cathode run in 0.1 M LiOD at 40°C in the ET flow calorimeter without any chemical additions. During this time, the current and input powers were increased to 3.2 A (400 mA cm^{-2}) and 35 W, respectively.

and 1 Pd cathode was run with sulfuric acid electrolyte. The custom Pd cathodes shown in Fig. 3 were included among the unproductive materials examined.

The early experiments demonstrated the behavior of the ET and Hart calorimeters. In most experiments, the electrochemical cell was operated in the constant current mode with the current increased stepwise from 0.01 to 2000 mA as the experiment progressed. The input and output powers to the calorimeters were calculated at each step and the data were fitted using the initial steps as calibration assuming that no excess power was being produced. The Hart fitting coefficients each varied by less than 1% over a several month period. The consistency of the fitting coefficients confirmed the stable performance of the Hart calorimeter.

Examples of data obtained during electrolysis experiments in the ET and Hart calorimeters when no chemical additions were made to the 0.1 M LiOD electrolyte solution are displayed in Figs. 4 and 5, respectively. The figures show plots of the electrochemical input and output powers to the cell that were fit during the initial current steps, the current and the excess power ($P_{out} - P_{in}$). The data clearly show that no excess power was produced in either of the cells during the times shown.

After running nearly 60 calorimetric experiments over a year and a half with no excess power observed, we began to make chemical additions to the electrolyte in the Hart cells. The purpose of the additions was to accelerate the initiation of the excess power by forming oxides or other reactive species on the cathode surface. Chemicals were added to the electrolyte at high current (power) levels so as not to hinder the loading of the cathode. The additions were made by placing a small amount (few mg) of solid additive in a syringe, drawing up some electrolyte solution into the syringe and re-injecting the solution + additive into the cell. In this way, the additions were made without exposing the electrolyte to the ambient atmosphere. Some of the additions occasionally produced a small, nearly constant apparent excess power

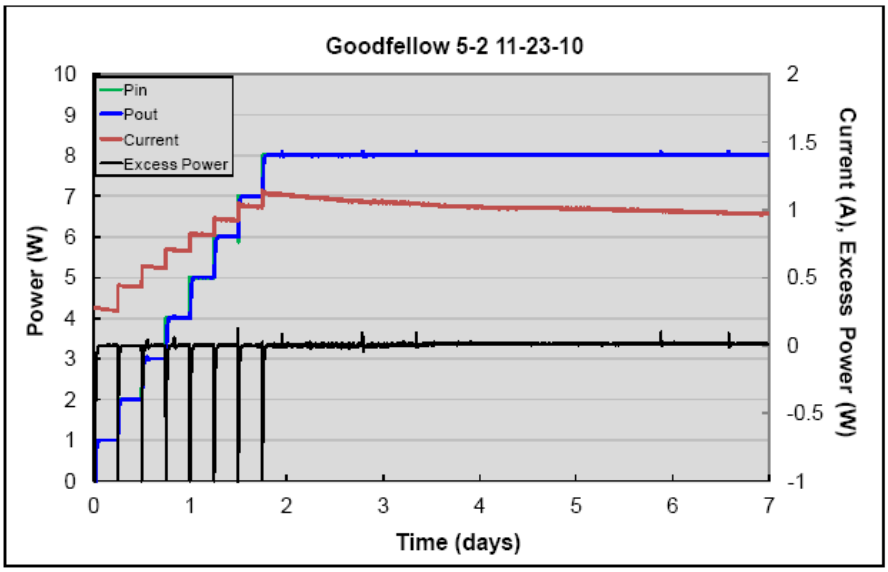

Figure 5. Plots of input power, output power, current and excess power as a function of time since the start of the experiment for a Goodfellow Pd cathode run in 0.1 M LiOD at 40°C in the Hart calorimeter without any chemical additions. During this time, the current and input powers were increased to 1 A (250 mA cm^{-2}) and 8 W, respectively.

(20–100 mW) event following the addition. The generation of an effect by the chemical addition was intriguing, but its small magnitude and constant offset from the baseline caused us to question the source of the heat. One possibility was that the chemical addition changed the resistance of the electrolyte so that the power distribution in the cell was shifted between the electrode and the catalyst regions. Experiments with resistors in the cell indicated that the point of power generation did not greatly affect the heat measurement by the Hart, so the observations of long-term, small amounts of excess power still are unexplained.

In many calorimetric experiments, more than one chemical addition was made when no response from an individual additive was apparent. One such experiment with a $Pd_{90}Rh_{10}$ cathode in 1 M LiOD is shown in Fig. 6. At about 3.7 days, an instability occurred due to a loose connection on the Hart calorimeter that persisted until the system was temporarily stopped, repaired and restarted at a current of 2 A. Once the system regained stability several chemicals were added sequentially to a Hart cell. No apparent excess power was produced after the first two additions. However, a large spike of apparent excess power was generated following the third addition. The apparent excess power spike peaked at a maximum of almost 4 W and persisted for approximately 2 h. Thus, the total apparent excess energy produced was on the order of 10 kJ. This was the largest apparent excess power event seen in our laboratory to date. The chemical energy produced by the additions of a few mg of metal salts cannot account for the10 kJ excess observed in this experiment. A second identical addition to the electrolyte solution following the appearance of the spike and recovery of the baseline had no effect on the excess power production. At this point, it is not known whether the third chemical addition alone was responsible for the power spike or whether some synergism among the different additives occurred. This is under investigation.

Shown in Fig. 7 are some SEM images taken of the $Pd_{90}Rh_{10}$ cathode surface after removal from the Hart cell and

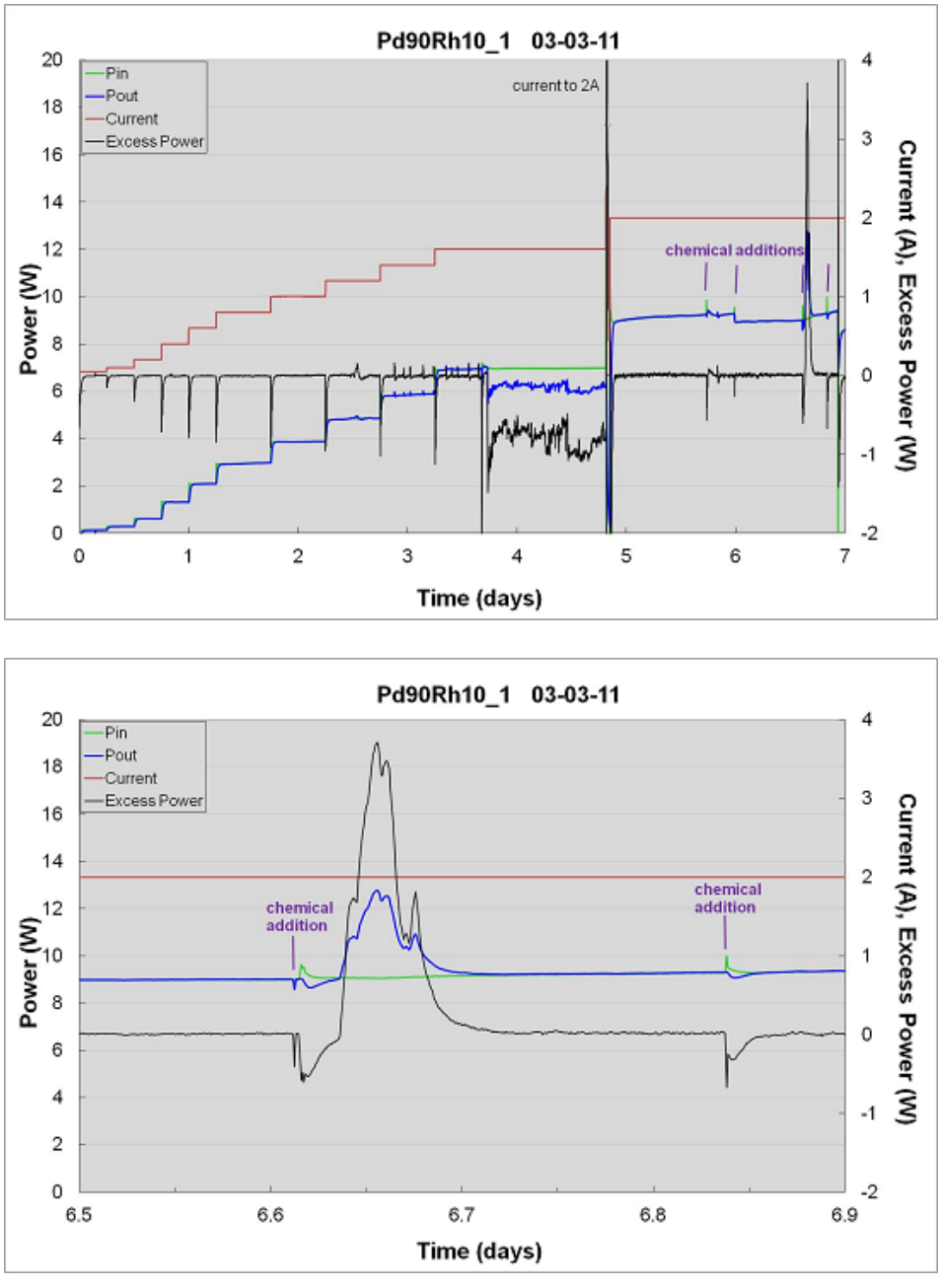

Figure 6. (a) Plots of input power, output power, current and excess power as a function of time for a $Pd_{90}Rh_{10}$ cathode run at 25°C in the Hart calorimeter with chemical additions as indicated and (b) an enlargement of the plots shown above from 6.5 to 6.9 days. Prior to the apparent excess power event, the input current and power were 2 A and 9 W, respectively.

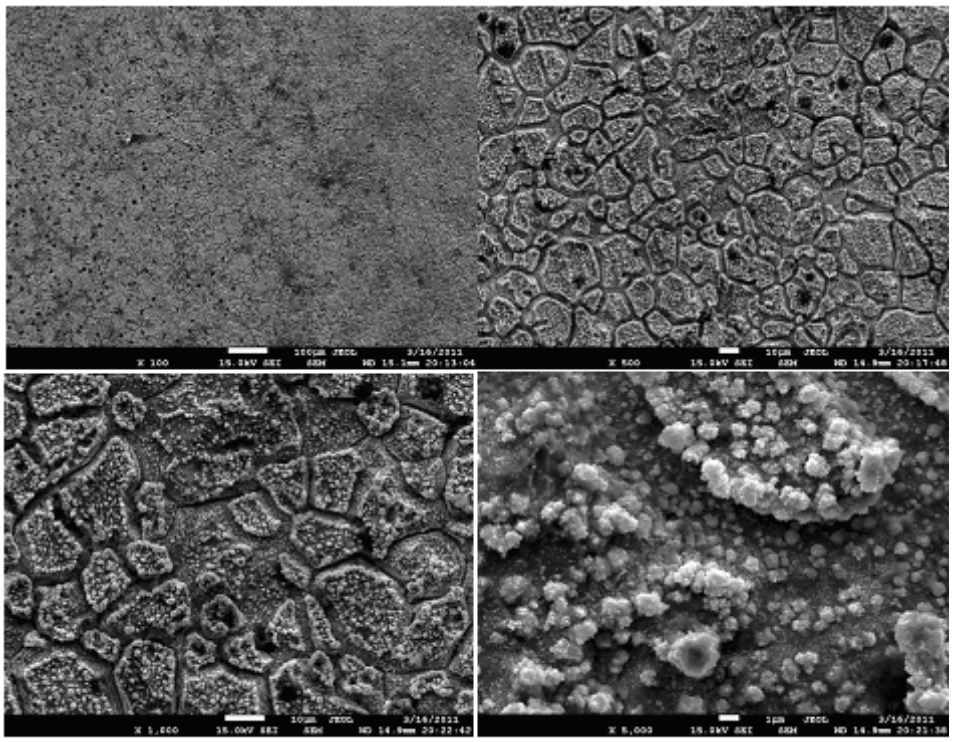

Figure 7. SEM images at 100 (100 μm scale bar), 500 (10 μm scale bar), 1000 (10 μm scale bar) and 5000 $\times$ (1 μm scale bar) magnification of the $Pd_{90}Rh_{10}$ cathode surface after 7 days in the Hart calorimeter. Chemical additions were made to the cell at 2 A/ 9 W input power and an apparent excess power event occurred after the third addition. The images show a white, crystalline material deposited around the grains and in the grain boundaries of the cathode. The white material appears to be a lithium aluminate.

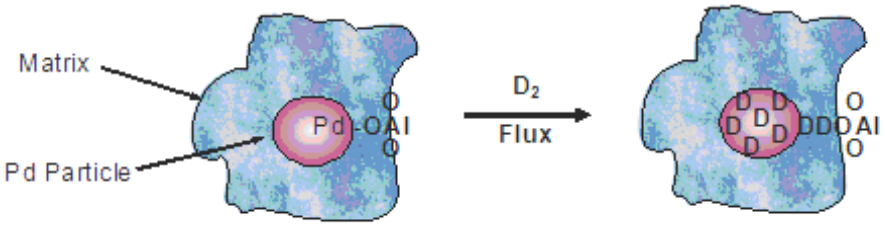

Figure 8. A hypothetical model illustrating the possible role of oxides on the palladium surface in the generation of excess power. The oxides are known to produce strong electric fields up to 9.5 V/nm which might enable the deuterium atoms to approach closely enough to undergo an unknown reaction.

rinsing with distilled water. The images show a white, crystalline material deposited around the grain edges and in the grain boundaries of the cathode. The deposited material contained aluminum and oxygen by XPS and energy dispersive X-ray (EDX) analysis. X-ray diffraction analysis of another cathode foil identified the white material found on that foil as lithium aluminate. The white material usually forms in the cell as a large deposit at the base of the catalyst cage and is presumed to originate as the electrolyte leaches alumina from the catalyst. It is not often observed on the surface of cathodes taken out of the Hart cells.

The surface of the $Pd_{90}Rh_{10}$ cathode was examined by XPS after removal from the Hart cell. Besides carbon and oxygen, a number of elements were identified in the analysis. These included aluminum (supposedly from the catalyst), uranium, copper, nickel, cobalt, sulfur and platinum as well as possibly zinc, lead and tin. The stainless steel syringe needles were thought to be a likely source of most of the metallic impurities. Uranium may have been introduced inadvertently as cross contamination from an earlier experiment. It is not known whether any of these impurities played a role in the excess power generation.

Figure 8 shows a hypothetical model illustrating the possible role of oxides on the palladium surface in the generation of excess power. According to the literature, oxides produce strong electric fields up to 9.5 V/nm [14]. The high electric fields might enable two deuterium atoms to approach each other close enough to form D_2 molecules in the oxide interface that formed on the surface of the electrode during electrolysis. Thus, the presence of a strong electric field might be a requirement for excess power generation in Fleischmann–Pons-type electrochemical experiments.

4. Conclusions

The results of over 100 calorimetric experiments on palladium, palladium alloys and other materials electrochemically loaded with both deuterium and hydrogen were presented. Nearly 60 experiments run without the addition of chemical additives produced no excess power. These experiments were considered as controls showing that the calorimetry is being performed properly. Some chemical additions that could form an oxide interface on the palladium resulted in yet unexplained small, long-term apparent excess power. One palladium–rhodium cathode produced a strong, positive response that could be excess power after a series of additions. Attempts to reproduce and understand this result are continuing in our laboratory. Finally, a model describing the possible role of oxides interfaces in generating high electric fields that might be a requirement for excess power production is proposed.

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References

[1] M. Fleischmann and S. Pons, *J. Electroanal.l Chem.* **261** (1989) 301–308.
[2] *Proceedings of the International Conference on Cold Fusion from ICCF1* (1990) – ICCF14 (2008)
[3] M.C.H. McKubre, R.C. Rocha-Filho, S.I. Smedley, F.L. Tanzella, S. Crouch-Baker, T.O. Passell and J. Santucci, in *The Science of Cold Fusion*, T. Bressani, E. Del Giudice and G. Preparata (Eds.), Conference Proceedings Vol. 33, Italian Physical Society, Bologna, Italy (1991), pp. 419–443

[4] M.C.H. McKubre, S. Crouch-Baker, A.M. Riley, S.I. Smedley and F.L. Tanzella, Proceedings of the 3rd International Conference on Cold Fusion, "*Frontiers of Cold Fusion"*, H. Ikegami (Ed.), Universal Academy Press Inc., Tokyo, Japan, (1992), pp. 139–150
[5] E. Storms, *J. Fusion Technol.* textbf29 (1996) 261–268.
[6] I. Dardik, H. Branover, A. El-Boher, D. Gazit, E. Golbreich, E. Greenspan, A. Kapusta, B. Khachatorov, V. Krakov, S. Lesin, B. Michailovitch, G. Shani and T. Zilov, *Proceedings of the 10th International Conference on Cold Fusion*, ICCF-10, Cambridge, MA (2003).
[7] I. Dardik, T. Zilov, H. Branover, A. El-Boher, E. Greenspan, B. Khachatorov, V. Krakov, S. Lesin, and M. Tsirlin, Presented at the *11th International Conference on Cold Fusion*, ICCF-11, Marseilles, France (2004).
[8] V. Violante, F. Sarto, E. Castagna, M. McKubre, F. Tanzella, G. Hubler, D. Knies, K. Grabowski, T. Zilov, I. Dardik and C. Sibilia, in *8th International Workshop on Anomalies in Hydrogen / Deuterium Loaded Metals*, Sicily, Italy (2007).
[9] V. Violante, F. Sarto, E. Castagna, M. Sansovini, S. Lecci, D.L. Knies, K.S. Grabowski and G. Hubler, *Proceedings of the 14th International Conference on Cold Fusion*, ICCF-14, Washington, DC (2008).
[10] F. Sarto, E. Castagna, M. Sansovini, S. Lecci, V. Violante, D.L. Knies, K.S. Grabowski and G. Hubler, *Proceedings of the 14th International Conference on Cold Fusion*, ICCF-14, Washington, DC (2008).
[11] E. Castagna, M. Sansovini, S. Lecci, A. Rufoloni, F. Sarto, V. Violante, D.L. Knies, K.S. Grabowski, G. Hubler, M. McKubre and F. Tanzella, *Proceedings of the 14th International Conference on Cold Fusion*, ICCF-14, Washington, DC (2008).
[12] S. Barrowes and H. Bergeson, *Fusion Technol.* **26** (1994) 365–368.
[13] V. Violante, F. Sarto, E. Castagna and S. Lecci, Presented at the *15th International Conference on Cold Fusion*, ICCF-15, Rome, Italy (2009).
[14] C. Otero Arean, *Chem. Phys. Lett.* **370** (2003) 631–635.