PROGRESS REPORT #6
ADDITIONAL BEHAVIOR OF PURE PdD

Subject: Part 5. Additional Behavior of Pure PdD
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INTRODUCTION

As is generally known, all pieces of palladium have properties slightly different from each other for reasons not easy to determine. The interaction between D and Pd is no exception. Consequently, each piece of Pd must be tested to determine its ability to react with D_2 and host LENR. The initial goal of this study was to find an alloy with more reliability in its ability to host LENR than pure Pd. To this end, Tom Claytor provided a batch of Pd to be formed into a Pd-Ag alloy. Testing of this palladium revealed an unusual ability to produce LENR, resulting in a temporary detour. The source of this Pd is unknown other than it was bought from a commercial supplier as a 1 mm thick sheet of the metal.

Over the years, finding such a lucky batch of Pd determined success in causing the F-P effect. In no case is the reason for success understood. Having active Pd now makes possible a search for the reasons and a test of various theories. The study of Pd-Ag alloys will just have to wait until this detour is explored.

Previous reports describing the apparatus, method, and previously observed behavior of the palladium sample are available at www.LENRexplained.com. This study expands on the results described in Progress Report #5 where the temperature of active Pd was found to be the most important variable affecting energy production once LENR starts. A correlation between excess power production and what appeared to be radiation generated by the process was also reported. This observation is shown to be an artifact as described below.

Based on the insights obtained so far, the F-P effect can be described as follows: Electrolytic decomposition of D_2O releases D_2 by an endothermic reaction and the D_2 reacts with the Pd cathode to produce only a small amount of energy. As result, the overall reaction absorbs energy. Conveniently, this reaction can be used to determine the enthalpy of formation of PdD as a function of composition.

Once the Pd is fully reacted with D, further application of current has very little effect other than to deposit impurities on the cathode surface. LENR is not initiated by simply achieving a high D/Pd ratio as some explanations predict. Instead, initiation of LENR requires the material be treated in special ways. Once the material is activated, a steady but small current must be maintained to keep the D in the Pd while the temperature is increased to cause increased power production. This small current plays no other role in causing or amplifying the LENR process. Only the ambient temperature of the cathode determines how much excess power can be made once some D is available and some NAE has formed.

Initially, the Pd is exposed to concentrated HNO_3 for 2 minutes followed by heating in air at 900° C for 5 hours, and slow cooling to room temperature as the furnace cools over about 8 hours. This treatment produced a uniform blue coating of PdO. The treatment is designed to remove the impurities from the surface either by vaporization or

by causing them to diffuse into the Pd were they are rendered harmless. Some of the impurities will be converted to their inert oxide, which has no ability to affect the reaction rate with deuterium. The PdO has been shown by many studies to create a very reactive surface because it is easily reduced to pure PdD.

EDX of the surface shows only Pd, oxygen, and carbon after the initial treatment. However, this treatment does not make a piece of Pd nuclear active. It only allows it to react as best it can with deuterium. Other treatments are required to make the material nuclear active if such activation is possible for a particular sample. Most samples of Pd can not be activated for reasons yet to be determined. Hopefully, these reasons will be made clear by future studies.

RESULTS

The Pd was subjected to two loadings, each of which is described separately. After the first loading, all the D was removed by heating at 150° C in air and the surface was again oxidized. The second reaction with deuterium was done using the same applied current and temperature as the first loading. The goal is to determine whether the loading process is affect by a previous treatment.

After loading was complete, methods were applied to cause the material to become nuclear active. These methods will be discussed in future reports. This report only describes the result of the loading and how the behavior of excess energy responds to various conditions.

The weight and dimension of the sample are listed in Table 1. EDX analysis revealed small amounts of Fe, Pt and Si in addition to the usual O and C after the first study. This deposit consisted of a thin brown-yellow stain, which was removed by concentrated HNO_3 before weighing. A greater weight loss resulted from this treatment compared to when pure Pd was exposed to acid because the Pd-Li alloy that forms on the surface during electrolysis was removed.

The calorimeter was calibrated between the two studies described here and gave the values plotted in Fig. 1 and fit by a quadratic equation. The standard deviation of the difference between this equation and the measured value is ±0.013 watt. When excess power is being made, the random scatter in individual values is greater than this, being near ±0.025 watt. A test of the calibration was made near the end of the study by removing the cell and using a quartz light bulb as a source of electrical heat. The resulting three points are plotted on Fig. 1 and show an average deviation from the initial calibration of no more than ±0.010 watts. Apparently, the calibration remained constant though out the study. The bulb is shown in Progress Report #2 to produce the same effect when compared to any other source of energy in the calorimeter.
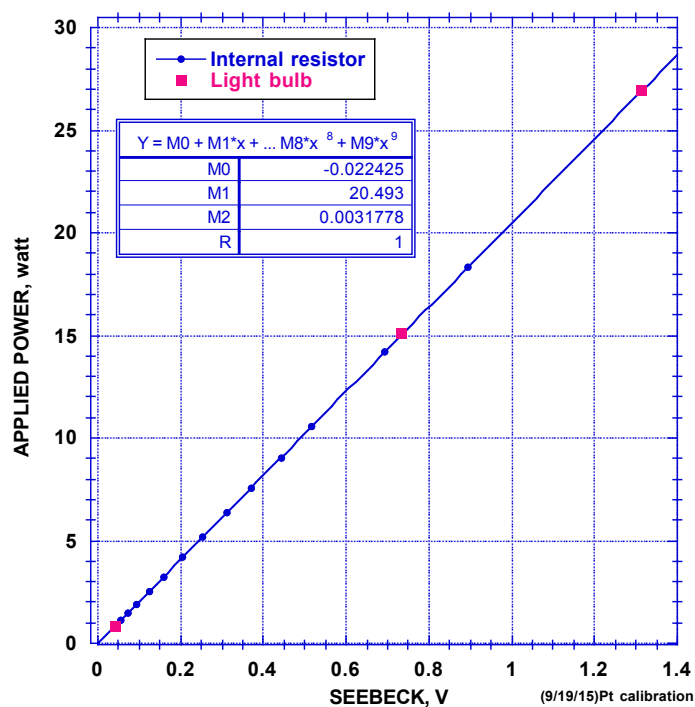
FIGURE 1. Calibration using the internal heater. Values obtained near the end of the study using a quartz light bulb are compared to the calibration done at the start of the study using the internal heater. The Seebeck reference temperature is 10° C.

Loading behavior

Figure 2 compares the D/Pd ratio and the power being used by the loading reaction during the first loading process. This sample loaded to $PdD_{0.88}$ and showed a behavior for the power being used that was somewhat different compared to the sample described in Report #5 even though the treatment was similar and the source of Pd was the same. Clearly, other variables not being controlled play a role. The second loading showed a similar behavior, as plotted in Fig. 3. In general, repeated loading following by the chosen treatment results in reproducible loading behavior.
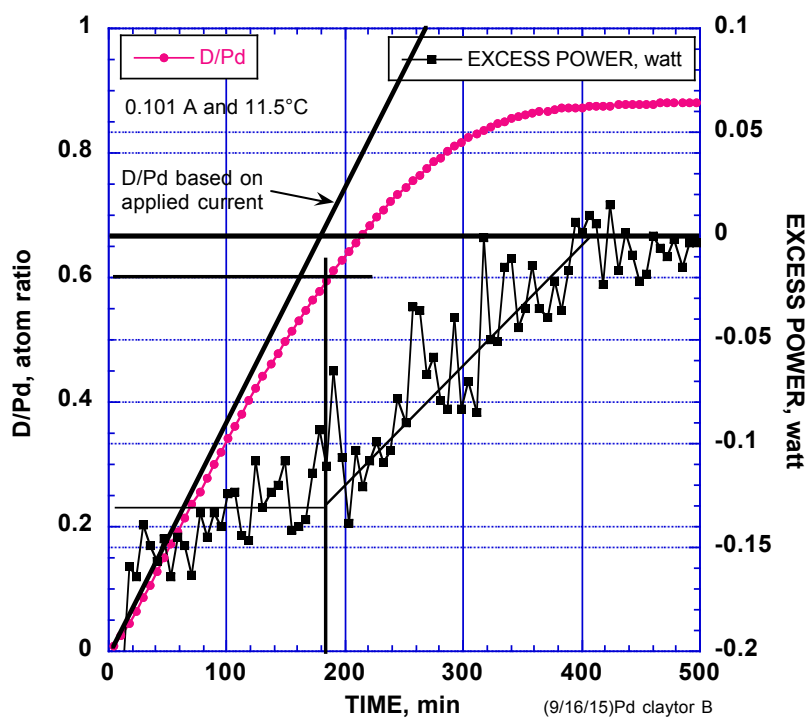
FIGURE 2. D/Pd atom ratio and excess power as a function of time during initial loading of Pd.
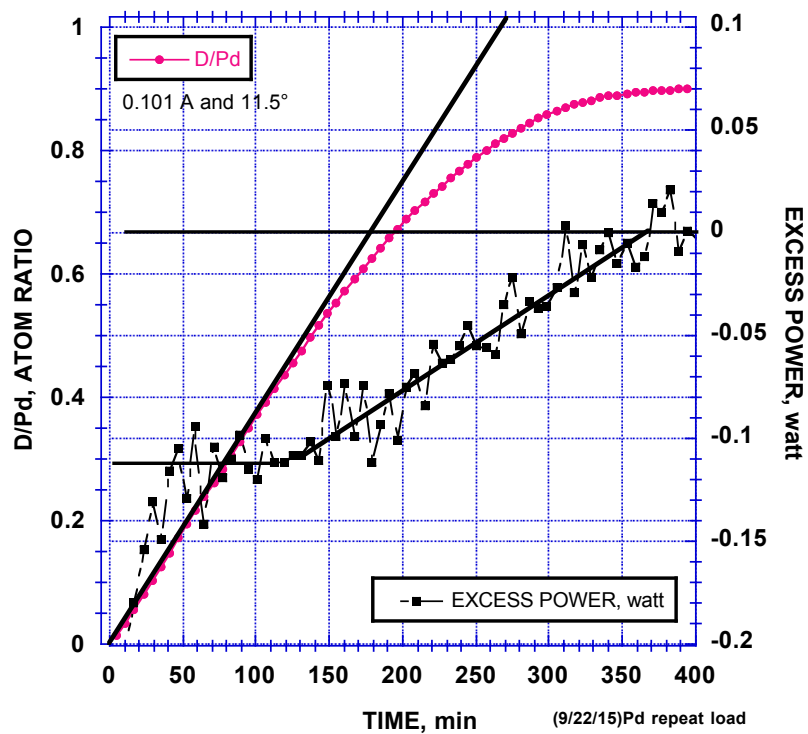

FIGURE 3. D/Pd atom ratio and excess power as a function of time during second loading of Pd.

Radiation flux

Most if not all of the apparent radiation reported in Report #5 is an artifact. This fact was demonstrated when apparent radiation was produced when the temperature was changed before the sample had reacted with deuterium, when a quartz-iodine light bulb was used as the source of heat, and when Pt was used as the cathode. In addition, insertion of a 1/8 thick plate of aluminum had no apparent effect on the count rate.

Apparently, the GM detector is sensitive to temperature, as shown in Fig 4 where the count rate measured every 6 minutes is compared to the temperature inside the calorimeter box. The box temperature changes slightly as result of changes in cell temperature. The vertical extent of the points shows the random variation of the very low background radiation. This temperature effect and the random variation add uncertainty to any measurement of radiation coming from the cell.
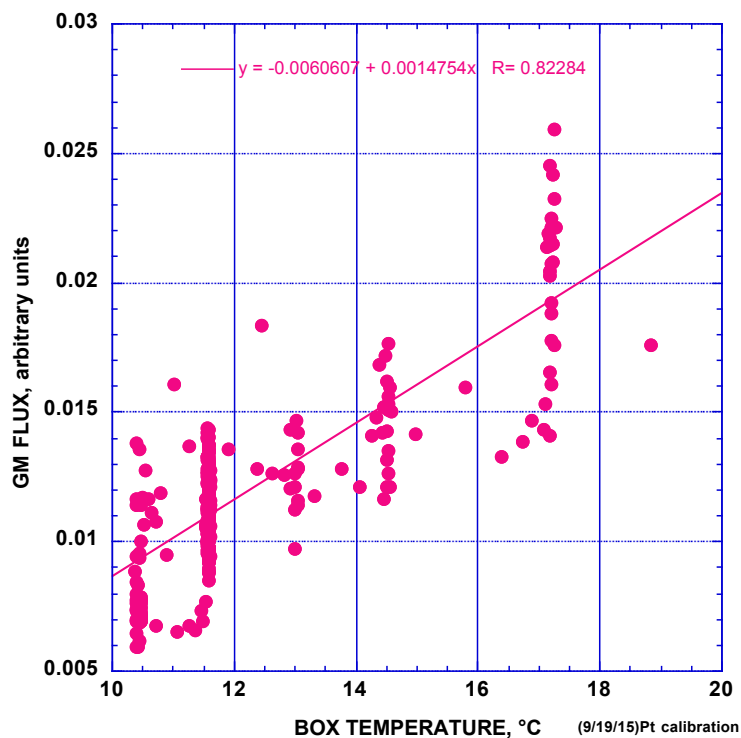

FIGURE 4. Apparent GM flux inside the calorimeter measured every 6 minutes as the temperature of the GM changed while using a Pt cathode during calibration.

Excess power production

Excess power is not produced unless the material is activated. Fleischmann and Pons (F-P)(*1*) observed the activation to require many weeks while electrolytic current was applied. Other people have found the process to be faster than this. Nevertheless, a change must take place in the cathode before excess power is produced. Apparently, some samples can not be activated no matter how they are treated. As described in the next section, the amount of excess volume created during loading predicts the eventual ability to make excess energy, as described first by Storms(*2-4*).

For this study, a constant current of 0.1 A is applied during loading and subsequently to maintain the concentration of D in the cathode. Once the sample is fully

reacted with D, the temperature is increased. Before activation, the increased temperature has no effect. As activation proceeds, the amount of excess power is found to increase, as shown in Fig. 6. Repeated activation causes the amount of excess power to increase.
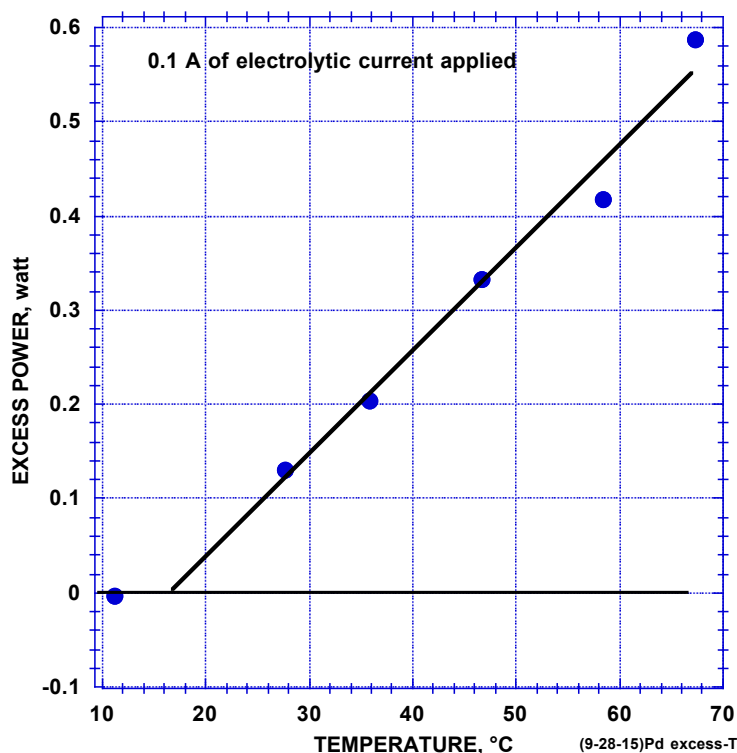
FIGURE 6. Excess power as a function of cell temperature while 0.1 A is applied as electrolytic current.

Creation of life-after-death(5) where the electrolytic current is turned off and excess heat production continues is easy to demonstrate. Figure 7 shows the excess power after the 0.1 A electrolytic current is turned off. As long as the temperature is not reduced, this excess power continues even as the sample slowly loses D.

This loss of D is proportional to the square root of time and essentially stops as the composition approaches $PdD_{0.67}$ at 11° because the equilibrium pressure of D in beta-PdD equals the ambient pressure of D_2 at this composition. In contrast, the temperature of 63° caused almost a complete loss of D to react D/Pd=0.15 over a period of 1000 minutes. The composition reached at the end of this time is obtained by weighing the amount of D lost when the sample is heated at 150° C. The composition at the start of the study is based on the amount of orphaned oxygen created. Although the excess power shows unsteady production, it remained essentially constant in spite of the change in average composition while the temperature slightly decreased. When the internal heater was turned off, excess power rapidly dropped to zero at a rate consistent with the time constant of the calorimeter as the temperature returned to the reference temperature of the calorimeter.
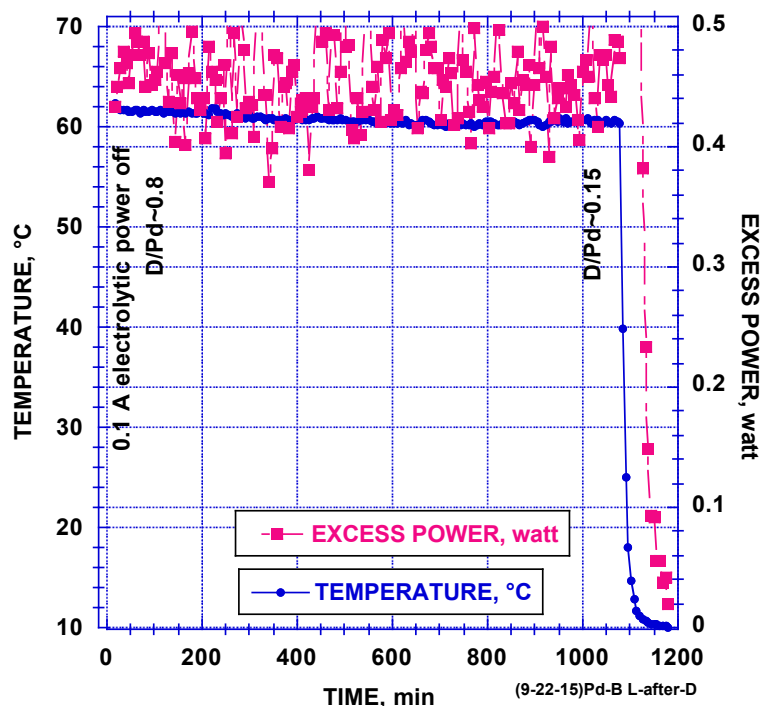
FIGURE 7. Excess power as a function of time after electrolytic current is turned off. The approximate D/Pd values are shown at the start and at the end of the study.

A sample that is being self-heated by power from the LENR process would continue to make energy after the electrolytic current is turned off because once LENR starts, it does not need to be fed by D supplied by the electrolytic process. The D that is already in the lattice supplies D to produce LENR even at low compositions. In this case, too little excess power is produced to maintain the high temperature required to produce a significant rate of LENR. Consequently, the excess power is reduced as the sample cools.

Excess volume

The Pd used for this study apparently produces very little excess volume. As previous work predicted, this behavior is consistent with the ability to achieve a high D/Pd ratio, which reached $PdD_{0.88}$ during the first loading and $PdD_{0.89}$ during the second loading. A second nearly identical daughter sample from the same supply of Pd loaded to $PdD_{0.84}$ and also produced no excess volume. The excess volume achieved by the sample from the same batch described in Progress Report #5 has not been measured.

Table 1 lists the measured weight and dimensions after various treatments. The excess volume is obtained by comparing the volume before reaction with D with the volume after all the D is removed by being heated at 150° for several hours, as emphasized by the red highlight in Table 1. Other samples have produced a volume increases as result of this treatment as high as 15%. Such samples seldom achieve high loading. Whether excess energy can be produced when excess volume is too low is a separate issue that needs further study. After all, the proposed cracks are created by uneven stress, which would not be present when excess volume is too small.

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TABLE 1
Summary of physical measurements after various treatments of Pd Claytor B

First Run	weight, g	D/Pd	width, mm	length, mm	thick, mm	volume,mm^3
Wt Pd	1.73904					
Pd+lead	1.78110		12.78	12.11	0.986	152.6
After HNO$_3$	1.77477					
After 900°	1.77473		12.73	12.09	0.976	150.2
After first loading	1.79553	0.636	12.93	12.26	1.077	170.7
After 150°	1.76600*		12.58	11.95	1.001	150.5

* weight lost as result of loss of D and cleaning with HNO$_3$

Open circuit voltage

As a reminder, the OCV is proportional to the chemical activity of D on the surface of the cathode. This chemical activity is related approximately to the D/Pd ratio. The value is sensitive to temperature, applied current, and the impurity content at the surface. For this particular apparatus, the OCV of the alpha+beta two-phase region is below 0.8 V.

A typical behavior of the OCV during loading is shown in Fig. 8. As the D/Pd ratio in the bulk increases, the OCV also increases along with the cell voltage. However, this increase eventually stops and further electrolysis causes a reduction in OCV. This reduction is proposed caused by the slow deposit of Pt and other impurities that hinder reaction with D, thereby causing a reduction in the surface composition.
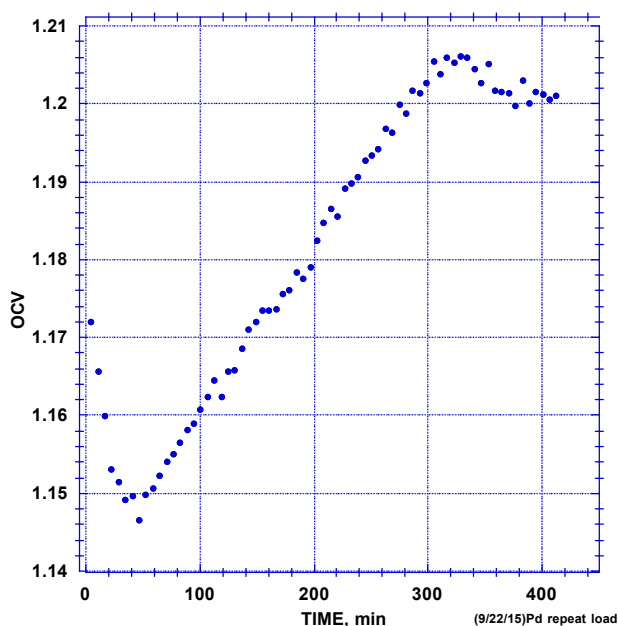
FIGURE 8. Typical change in OCV as the sample initially reacts with D.

Increase in temperature causes an initial reduction in the OCV, with a smaller effect at high temperatures.

Appearence and composition of the surface

After the study, the surface of the sample was found to consist of many parallel sheets of atoms that formed ridges, as showin in Fig. 9. The nanocracks are proposed to form between these sheets. However, their gap would be too small to resolve using the available SEM. The surface composition is: Pd- 74 at %, O-23%, Pt-0.9%, Si-0.8%, Cu-0.7% and C-0.6%, which is typical of the deposit produced by this electrolytic cell. All of the impurities have an identified prosaic source.

The surface before the study can be seen in Fig. 10. Only Pd, O and C were detected on the surface.

DISCUSSION
OCV

The OCV initially increases as Pd reacts with D, with a trend toward lower values once the sample is fully reacted. Part of this reduction is probably caused by various impurities deposited on the surface by electrolysis. In general, the greater the applied current, the faster this deposit forms. Consequently, application of high current is best avoided.
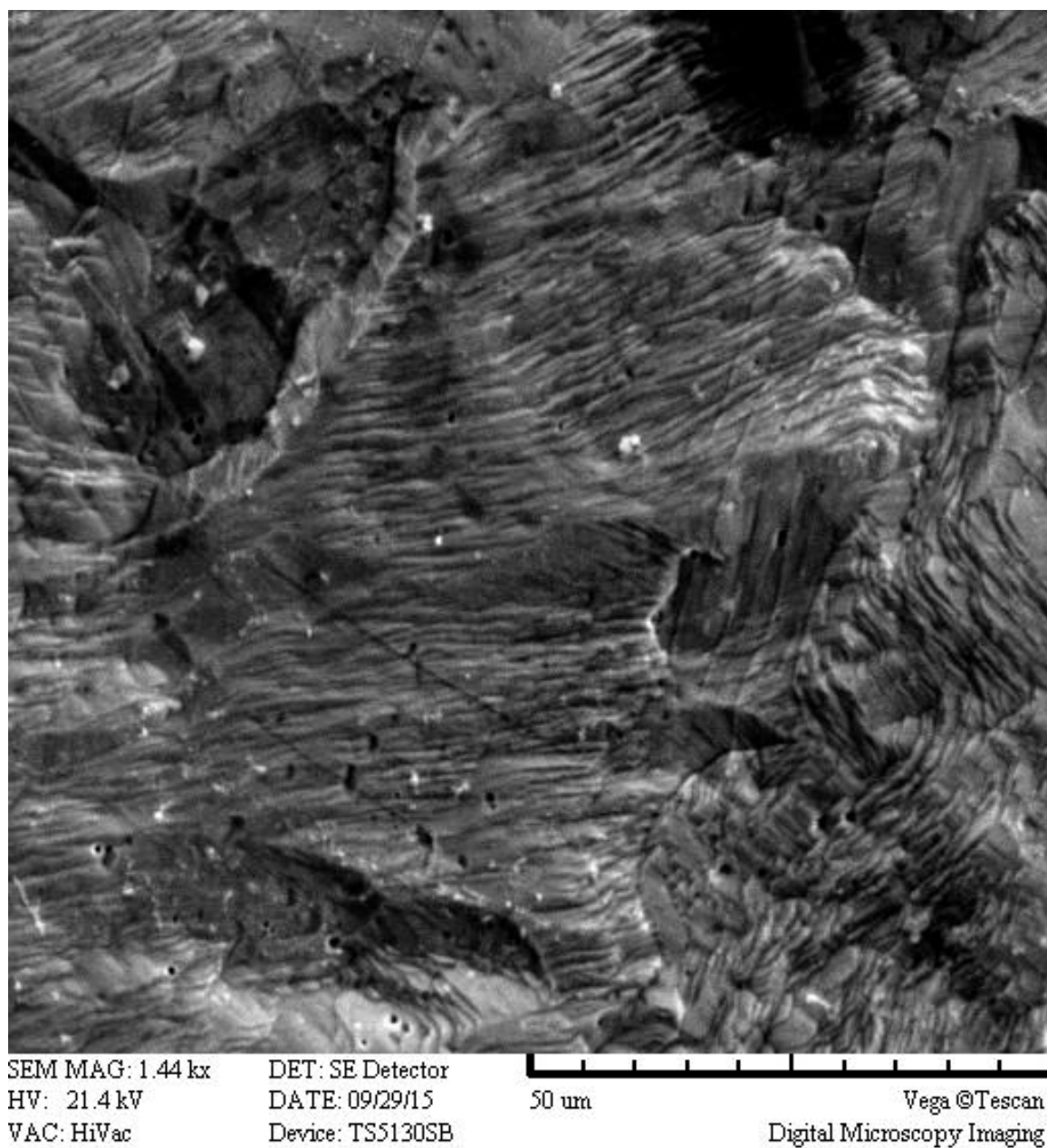
Apparently, the surface maintains a significant composition of D even after the electrolytic curremnt has been stopped and D slowly leaves the bulk through the surface.

Every sample has a slightly different behavior of the OCV and the amount of excess power appears to have no relationship to the OCV.
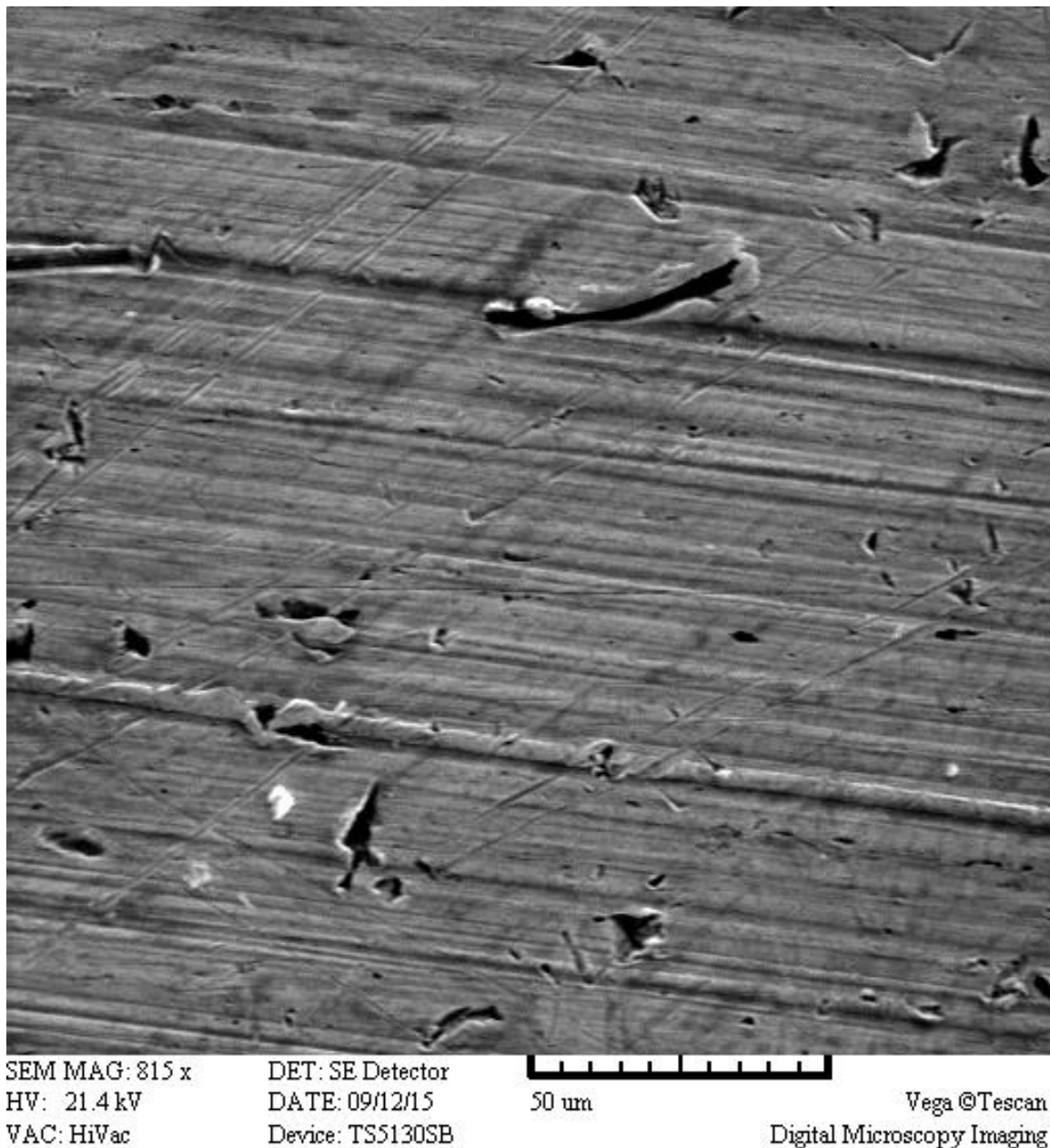
Excess volume

Palladium expands when it reacts with D. This expansion is not uniform in all directions and is not consistent with the increase in lattice parameter. In addition, some samples do not return to their original volume when all the D is removed. Excess volume is used as a measure of how non-ideal the lattice structure has become as result of this process. The excess volume is presented as the change in volume divided by the initial volume. This number can vary between 0 and about 15%, depending on the initial properties of the Pd.

The process of forming the excess volume would create uneven stress and result in crack formation. When large cracks form, D_2 can be released through them to cause a reduction in surface composition and a lower average composition. In contrast, a small amount of excess volume is proposed to produce small cracks without the ability to release D_2 but with the ability to initiate LENR. Consequently, a small excess volume produced by Pd, such as is the case with the sample studied here, is important and relevant to being able to achieve a high D/Pd and generate excess energy. Therefore, finding ways to test for and modify the amount of excess volume is important, as I pointed out over 20 years ago.

SEM MAG: 1.44 kx   DET: SE Detector
HV: 21.4 kV   DATE: 09/29/15   50 um   Vega ©Tescan
VAC: HiVac   Device: TS5130SB   Digital Microscopy Imaging

FIGURE 9. Surface of the Pd cathode after the study.

SEM MAG: 815 x   DET: SE Detector
HV: 21.4 kV   DATE: 09/12/15   50 um
VAC: HiVac   Device: TS5130SB

Vega ©Tescan
Digital Microscopy Imaging

FIGURE 10. Surface of the Pd cathode before the study.

Effect of temperature

A person might wonder how and why a nuclear reaction could be so sensitive to such a low temperature as is the case with LENR. An answer can be found by making an Arrhenius plot using data shown in Fig. 6. The apparent activation energy for the effect of temperature on the rate of power production can be compared to other process potentially related to the LENR process. Figure 11 shows a plot of log power vs $1/T$, where the excess power is assumed to represent the rate of a nuclear reaction being influenced by temperature. The slope of the line in Fig 11, which is 1621, can be compared to the slope of a similar plot in Fig. 12 based on the Fick's diffusion constant, which is 1760. With the slopes of the two behaviors being very similar, we can conclude

that the major influence is determined by the rate at which D can diffuse through PdD to reach the active sites. The comparison is slightly compromised by the diffusion constant being sensitive to the D/Pd ratio, which is variable, and by the different temperature ranges being compared. Nevertheless, the two activation energies are close enough to strongly suggest a relationship. In any case, what other process involving so little energy could change the rate of a nuclear reaction?
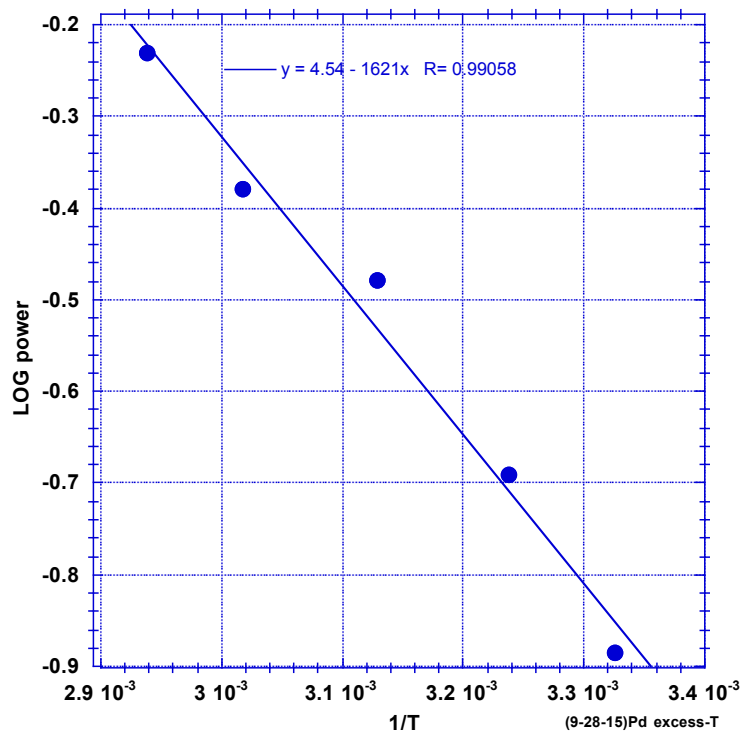

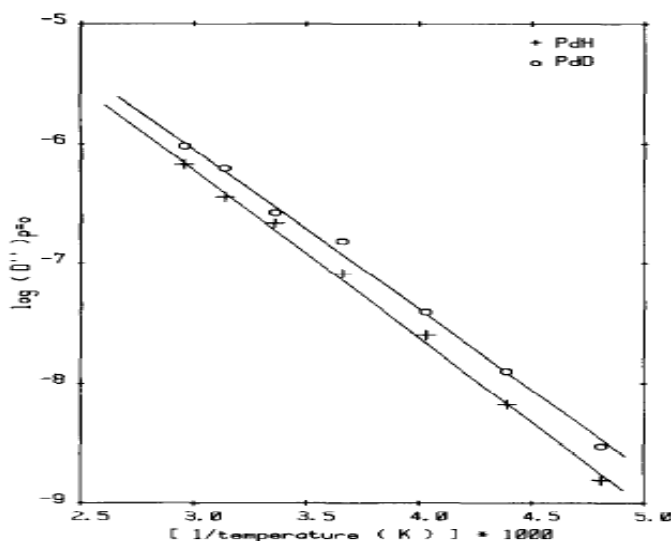**FIGURE 11**. Plot of log (excess power) vs 1/T.

FIGURE 12. Effect of temperature on the Fick's diffusion constant for PdD and PdH. (*6*) The　slope describing the diffusion of D is 1760.

Once the temperature is acknowledged to have the effect shown in Fig. 11, the commonly observed need for an apparent critical applied electrolytic current can be explained. As shown in Progress Report #5, the current has no influence on the amount of power generated by LENR. On the other hand, application of electrolytic current would change the temperature of the cathode and the NAE contained therein. For power to be detected, this source of temperature must create enough power to exceed the sensitivity limit of the calorimeter. For the calorimeter being used here, the temperature must produce at least 0.05 watts for any excess power to be claimed. Lower temperatures might generate excess power, but in too small an amount to be detected. Consequently, a claim for excess power would depend on the reference temperature used in the calorimeter and how sensitive the cathode temperature is to applied current, both of which will affect the temperature of the cathode. If the reference temperature is low, a high electrolytic current would have to be applied to reach a reaction rate able to generate detectable power, provided, of course, the sample is nuclear active. This insight provides another condition requiring consideration when replication is attempted and explanations are proposed.

Apparent Radiation:

Here we have an example of the care required to test for all unexpected behavior. Normally, the false conclusion of radiation detection would have been removed in the final paper. By reading the Progress Report, a better understanding of the potential false leads Nature frequently uses to test the experimenter can be obtained. Replication by other studies is not the only way a conclusion must be tested. Tests must also be done within each individual study. Such tests are part of the routine in this study, which revealed the previous false conclusion.

In the future, the effect of temperature on the apparent radiation flux must be subtracted from the real flux to obtain the flux emitted by the sample. Unfortunately, this requirement adds considerable uncertainty. Hopefully future studies will resolve this uncertainty when much greater excess power is produced.

SUMMARY

A lucky coincidence was created when Tom Claytor supplied some unusual Pd for use in making Pd-Ag alloys. This batch of Pd was found not to produce excess volume when it is loaded and deloaded with D, in contrast to the common behavior of conventional Pd. The sample also showed the ability to achieve an unusually high D/Pd ratio and to eventually produce excess energy after suitable activation. As found over 20 years ago, this sample shows that absence of excess volume is correlated with the ability to achieve a high D/Pd ratio and produce excess power.

Once Pd has become fully reacted with D, the material must be activated before excess power can be produced. This activation process will be described in future Reports.

After excess power is produced, the temperature of the material is a major variable in determining how much energy will be generated. The amount of current applied to maintain the D/Pd ratio is not important. This behavior makes heat-after-death possible.

The amount of excess power is not related to the composition of the surface as determined using the OCV.

Apparently, the GM detector used here produces a pulse that is slightly sensitive to the temperature of the detector. Consequently, the output of the electronic circuit appears to indicate a slightly increased average radiation flux that is not caused by a nuclear reaction. This effect would normally not be observed because a GM tube is generally held at a relatively constant temperature and the small change seen here would not be considered significant. In this case, the effect must be subtracted from the apparent radiation flux to isolate radiation being generated by the sample. So far, the excess power achieved so far is not sufficient to produced detectable radiation.

The results are not consistent with the common belief that a high D/Pd ratio is required to initiate LENR. The results are not consistent with the common belief that the rate of LENR is sensitive to applied electrolyte current. In contrast, all behaviors seen so far are consistent with the theory of Storms(*7-9*), which will be used as a guide for these studies.

Having three samples able to produce reproducible excess power makes a study possible of the other variables having influence on power production. Once again, knowing where to look and having suitable tools available make progress possible – along with some luck.

References

1.   M. Fleischmann, The present status of research in cold fusion, in Second Annual Conference on Cold Fusion, "The Science of Cold Fusion", Ed: T. Bressani, E. Del Giudice, G. Preparata, (Societa Italiana di Fisica, Bologna, Italy, Como, Italy, 1991), 475.
2.   E. K. Storms, C. Talcott-Storms, The effect of hydriding on the physical structure of palladium and on the release of contained tritium. *Fusion Technol.* **20**, 246 (1991).
3.   E. K. Storms, Measurements of excess heat from a Pons-Fleischmann-type electrolytic cell using palladium sheet. *Fusion Technol.* **23**, 230 (1993).
4.   E. K. Storms, My life with cold fusion as a reluctant mistress. *Infinite Energy* **4**, 42 (1999).
5.   M. Fleischmann, S. Pons, Calorimetry of the Pd-D_2O system: from simplicity via complications to simplicity. *Phys. Lett. A* **176**, 118 (1993).
6.   S. Majorowski, B. Baranowski, Diffusion coefficients of hydrogen and deiterium in highly concentrated palladium hydride and deuteride phases. *J. Phys. Chem. Solid.* **43**, 1119 (1982).
7.   E. K. Storms, A Theory of LENR Based on Crack Formation. *Infinite Energy* **19**, 24-27 (2013).
8.   E. K. Storms, *The explanation of low energy nuclear reaction*. (Infinite Energy Press, Concord, NH, 2014), pp. 365 pages, (updated e-version available at Amazon.com).
9.   E. Storms, Explaining Cold Fusion. *J. Cond. Matter Nucl. Sci.* **15**, 295-304 (2015).