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Research Article

Anomalous Energy Produced by PdD

Edmund Storms*

LENRGY LLC, Santa Fe, NM 87501, USA

Abstract

Two samples of commercial Pd from the same batch were reacted with D using the electrolytic method and found to produce sustained excess power and energy. The effects of temperature, applied current, and D/Pd ratio on the amount of excess power were studied.
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Keywords: Cold fusion, Enthalpy of reaction, Excess energy, PdD, Volume expansion

1. Introduction

When a material is involved in a chemical reaction, energy is frequently released. The amount of energy has been measured for most chemical reactions and the values are, in general, very accurate. After all, the entire field of chemistry is based on such measurements. When careful measurements reveal release of extra energy over that expected, either the claim is rejected as error or something very strange must be taking place. Once the possibility of error is eliminated, finding the source becomes a worthy effort.

In 1989, Profs. Fleischmann and Pons [1] observed extra energy being produced when they reacted deuterium with palladium using electrolysis. This reaction is very simple and well known, involving splitting D_2O into D_2 and O_2, with the D_2 reacting with the palladium to form the compound PdD. On occasion, more energy was made than could be explained by any chemical reaction. Over the years, this effect was replicated many times [2] and considerable evidence shows the extra energy results from formation of helium [3,4] and sometimes a little tritium, both of which can only be produced by nuclear reactions. A variety of other nuclear products have been observed on rare occasions [5]. Consequently, the process has been called Low Energy Nuclear Reaction (LENR) because it takes place at a much lower energy than is required to initiate hot fusion, the conventional version of the fusion process [6].

Explaining how LENR can occur in a common material has confounded science [7] while the supporting evidence just keeps growing. This and other nuclear reactions caused by LENR in several kinds of materials have been described in scientific journals, many peer reviewed. Nevertheless, the general rejection by the scientific community has forced publication of most information in unconventional places that, fortunately, are available on the web.[a]

*E-mail: storms2@ix.netcom.com.
[a]www.LENR-CANR,org, www.ColdFusionNow.org, www.LENRexplained.com.

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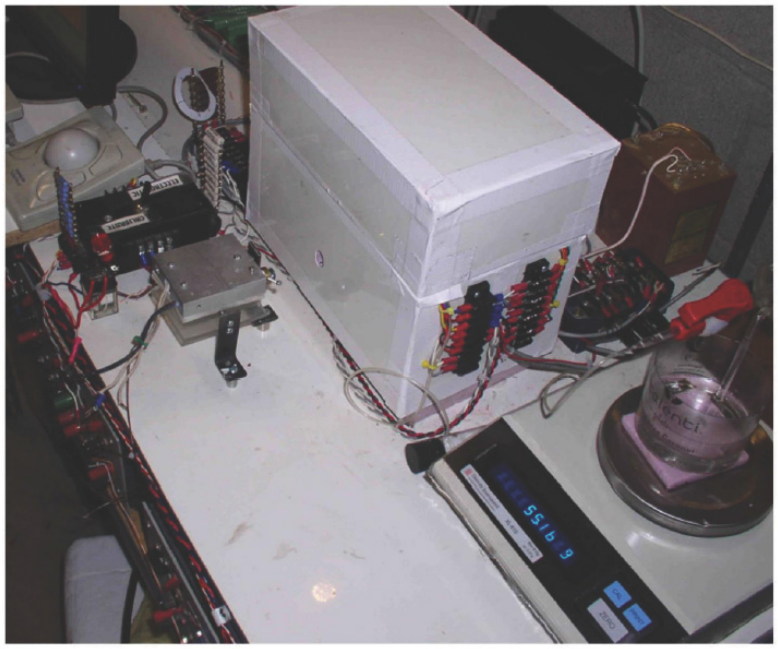

Figure 1. Picture of the closed Seebeck calorimeter. The balance used to measure the deuterium content of the cathode using the orphaned oxygen method is seen in the foreground. A laser is positioned to expose the cathode to laser light though a hole in the side of the calorimeter. This feature is not discussed in this paper.

This study demonstrates production of anomalous energy using two different samples and the behavior of this energy when temperature, deuterium content of the material, and applied current are changed. The observed response gives additional insight into the possible mechanism and corrects some previously incorrect conclusions about this behavior.

The problem is compounded by the difficulty in finding palladium able to cause the effect, i.e. be activated. Most palladium shows no ability to host such a reaction. This difficulty has frustrated replication, hampered research, and fueled rejection. Persistent effort has improved reproducibility by finding treatments that make the palladium more susceptible to activation [8,9]. Various alloys containing palladium and other metals have also been found to improve success [10]. In other words, the effect is not unique to palladium and can now be initiated when suitable skill is used. The common mantra that the effect has not been nor can be reproduced is simply not true, as this and hundreds of published studies have demonstrated [11].

Palladium found to produce extra energy has a few properties different from ordinary palladium. The palladium can achieve a very high D/Pd ratio without forming large cracks in the surface or voids [12–14] in the interior when it reacts with the isotopes of hydrogen. This feature provides a possible test of potentially active material. In addition, the surface frequently shows an array of crystals having a size of less than 100 μm with a preferred orientation [8]. However, these conditions alone do not result in extra energy although they are consistent with the requirements to achieve success identified by the author in previous papers [7,15,16].

This paper describes a successful effort to activate samples of palladium obtained from a commercial source. The conditions required to initiate energy production and the effect of various variables on its behavior are explored.

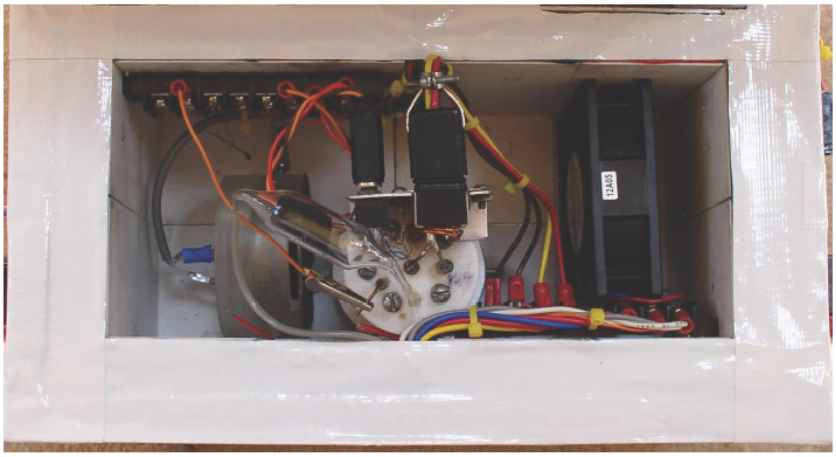

Figure 2. View inside the calorimeter enclosure. The GM detector is on the left, the top of the electrolytic cell is seen in the center, and a fan is on the right. A catalyst is contained in a tube located in the gas line to the orphaned oxygen measurement in order to eliminate any D_2 that failed to react with O_2 in the cell.

2. Results

2.1. Energy measurement

Because the effect is based on a claim for excess energy, accurate measurement of this energy becomes important. A Seebeck-type calorimeter is used, shown in Fig. 1, consisting of a water-cooled aluminum box with thermoelectric

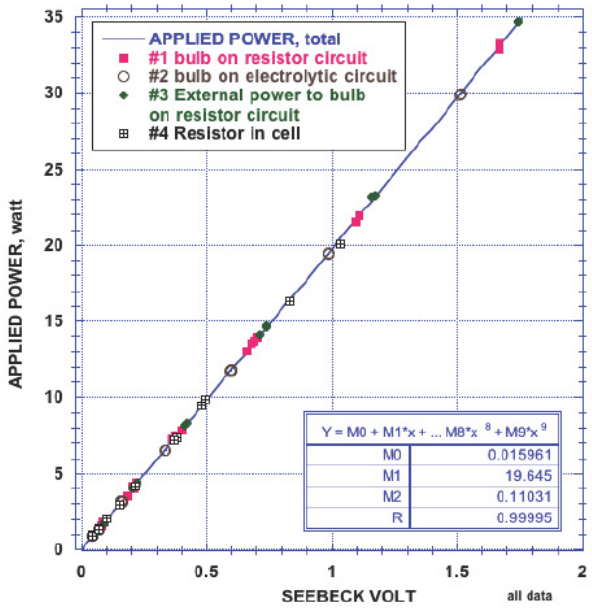

Figure 3. Typical calibration using several methods to apply heat energy to the calorimeter.

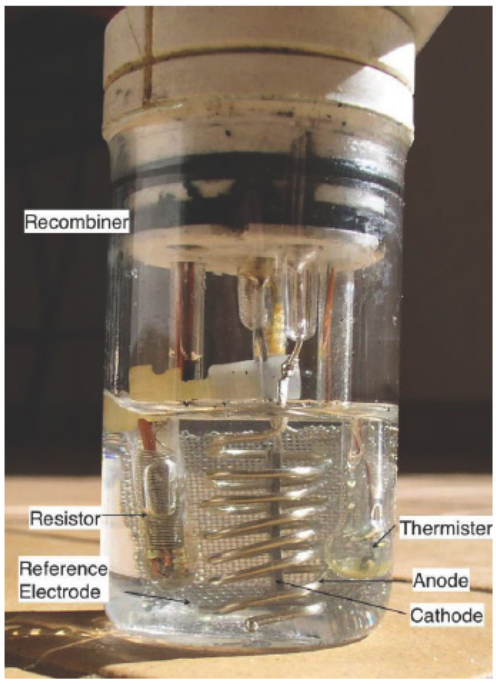

Figure 4. Pyrex electrolytic cell. The anode consists of Pt wire encircling the cathode. The cell is sealed using O-rings.

panels covering the inside surface. Excess power is defined as all power in excess of that applied to the cell as electric power based on a calibration using applied electric power.

The arrangement of the electrolytic cell, GM detector, and fan within the box can be seen in Fig. 2. Energy generated inside the box produces a voltage generated by the thermoelectric converters proportional to the magnitude of applied power. The amount of power is based on a calibration using electrical power applied to a resistor inside the electrolytic cell, as electrolytic current using an inert platinum cathode, or as current applied to a 50 W Quartz–iodine light bulb in place of the electrolytic cell. As can be seen in Fig. 3, each method for generating heat results in the same generated voltage when using the same amount of applied power.

The stability of the calibration is tested regularly by applying current using one of these methods. The uncertainty is ± 0.02 W from 0 to 30 W of total applied power based on fitting a quadratic equation to at least 10 data points measured over the range of power applied during the excess power measurement.

The electrolytic cell containing the Pd cathode is shown in Fig. 4. The cathode is attached by a Teflon clamp to a Pt wire that exits the cell through a glass-metal seal. The anode is made of platinum wire. The reference electrode is made of platinum mesh and is used to measure the open circuit voltage (OCV) relative to the cathode. A thermistor contained in a Pyrex tube is used to determine the electrolyte temperature. The electrolyte temperature can be changed independent of electrolytic power by applying electric power an internal resistor contained in a Pyrex tube. A catalyst consisting of Pt on Al_2O_3 is present in the cell to recombine any excess D_2 and O_2. Consequently, the cell is chemically closed and physically sealed to the atmosphere. A tube connects the cell to an oil reservoir used to measure the deuterium content of the cathode by the orphaned oxygen method. Pictures and a detailed description can be found at

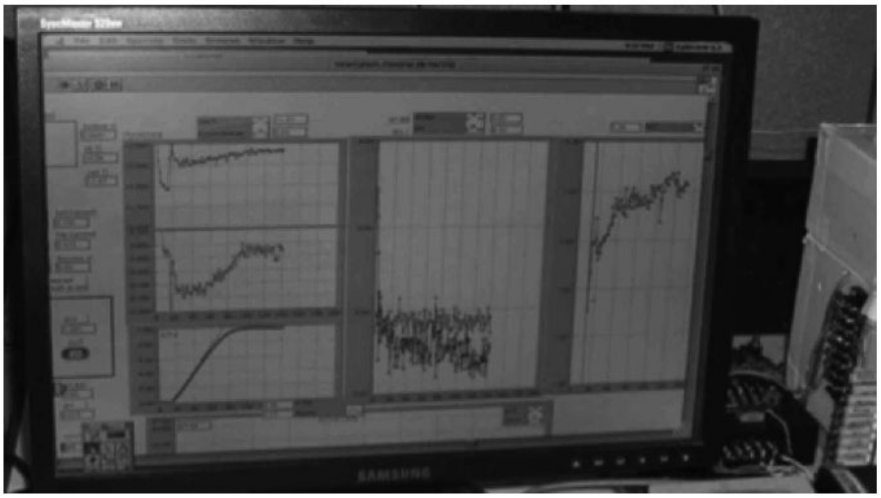

Figure 5. Computer screen showing real time display of cell temperature, excess power, D/Pd ratio, GM counting rate and OCV. The center graph compares the GM flux measured by the detector in the calorimeter to the background flux outside the calorimeter. This measurement will be discussed in a future paper.

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Values are taken every 6 min and plotted on a computer screen (Fig. 5) as well as being saved to an Excel file. Measurements are made of the current and voltage applied to the cell, temperature inside the cell, temperature inside the Seebeck box, the OCV, the radiation flux measured by the detector inside the box and by another identical detector located 3 m away from the calorimeter, the current and voltage delivered to the fan, the voltage produced by the thermoelectric converters in the box, and the weight of oil delivered from the reservoir as result of orphaned oxygen formation. All voltages are measured at the calorimeter boundary.

The electrolyte is 99.8% D as D_2O containing LiOD that is formed by adding about 0.1 g of Li metal to 30 ml of D_2O. The cell is made of Pyrex, the top is Teflon, and, other than the cathode, only platinum is exposed to the electrolyte.

The average D/Pd ratio is measured using the orphaned oxygen method. This method involves measuring the amount of oxygen released when deuterium is absorbed in the Pd. Oil is displaced by the extra O_2 from a reservoir onto a balance, seen on the right in Fig. 1. The accuracy of the D/Pd ratio is ± 0.01.

2.2. Loading behavior

The first step in initiating excess power production starts by creating a chemically active surface on the palladium cathode. In this study, an efficient reaction with D results when the surface has been subjected to cleaning using fuming HNO_3 and heating to 900°C in air for several hours, followed by slow cooling over several hours. The treatment at 900°C cleans the surface by either vaporizing the impurities or causing them to diffuse into the interior where they do no harm. A very thin layer of PdO forms during cooling, which is easily reduced by deuterium to produce chemically active Pd. Typically, the surface used in this study initially contains only Pd, O, and C, based on EDX analysis.

The loading process was studied extensively as part of this investigation, an example of which is shown in Fig. 6. Typically, each sample has a different upper limit to the D/Pd atom ratio while showing a similar behavior of energy

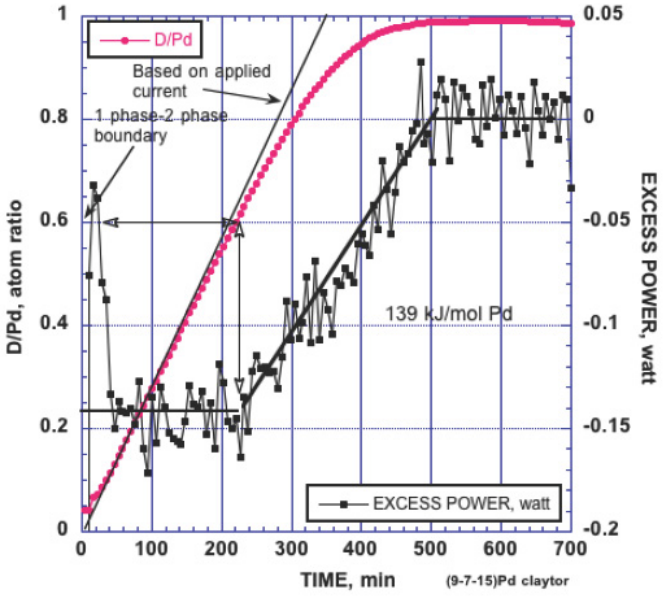

Figure 6. Typical behavior of palladium when reacted with deuterium at 11°C using Pd-#A. The average D/Pd ratio and the power associated with the reaction are shown. The initial behavior of the excess power results because sudden application of power causes the calorimeter to shift from the equilibrium condition when 0.1 A is applied. The negative numbers indicate loading is an endothermic process.

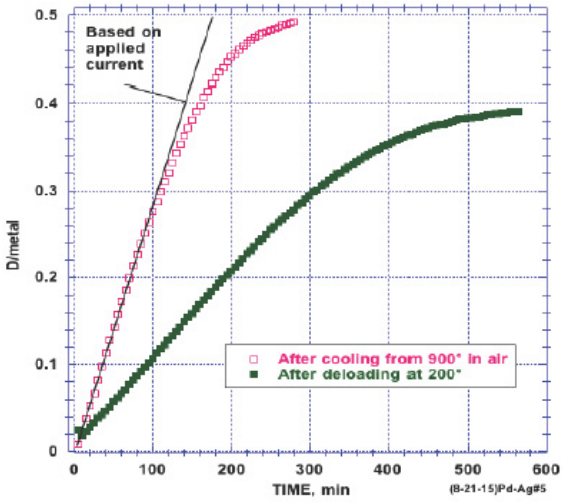

Figure 7. Reaction of Pd-#B with D after being previously loaded to D/Pd = 0.86 followed by partial removal of D without the surface being cleaned.

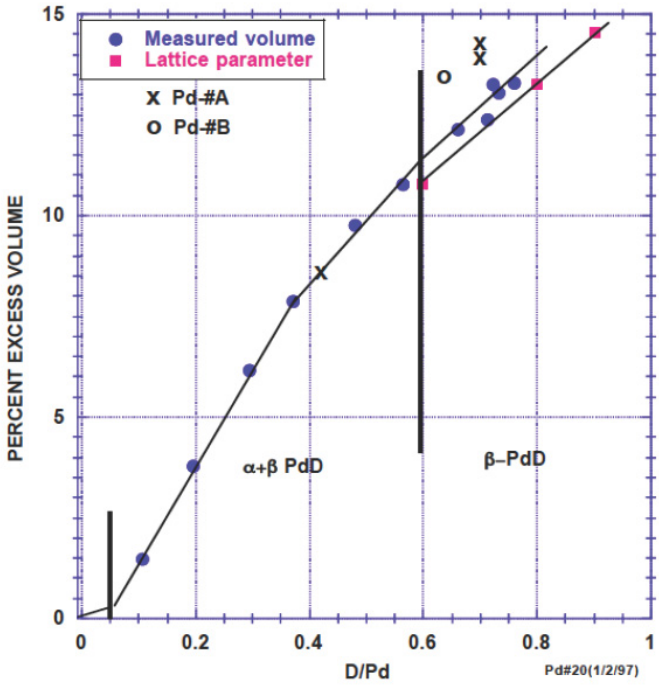

Figure 8. Physical expansion of the volume relative to the initial volume as D is added to typical Pd. The expansion based on the reported behavior of the lattice parameter is compared to the physical measurement. The behavior of Pd-#A and Pd-#B are also plotted.

consumed as deuterium is reacted. Energy is consumed because more energy is required to remove the D from D_2O than is recovered when the D reacts with Pd. This absorbed energy can be recovered when the D is reacted with O_2 to produce D_2O and pure Pd, which can take place within the calorimeter when deuterium is lost from the PdD after electrolysis is stopped or can cause self-heating when unloading occurs in air. The amount of stored energy is trivial

Table 1. Physical changes in Pd samples

Treatment	Width (mm)	Length (mm)	Thickness (mm)	Volume ($mm^{3)}$	Volume expansion (%)	D/Pd
Pd-#A						
Initial, uniform blue surface	10.91	18.43	1.008	202.7	0.00	0
Loaded-partially deloaded	11.17	18.70	1.107	231.2	14.09	0.70
Deloaded–loaded	11.22	18.79	1.100	231.9	14.42	0.70
Deloaded @ 150°C	10.93	18.57	1.084	220.0	8.56	0.42
Pd-#B						
Initial, uniform blue Surface	12.73	12.09	0.976	150.2	0.00	0
Loaded	12.93	12.26	1.077	170.7	13.65	0.64
Deloaded @ 900°C	12.60	11.97	0.998	150.5	0.21	0

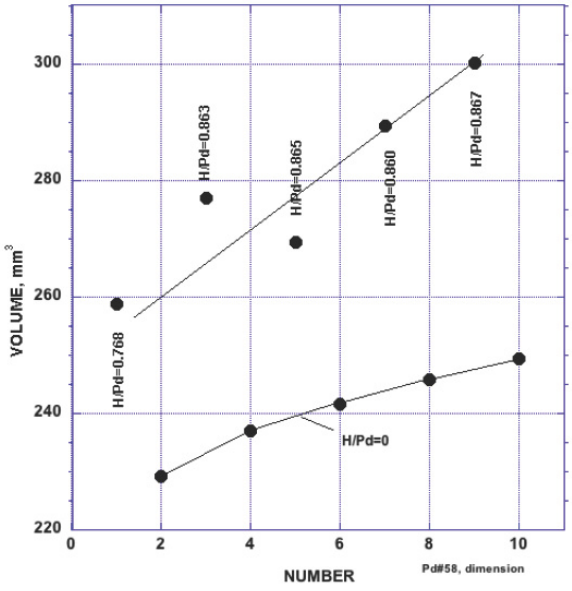

Figure 9. Changes in volume produced when H is repeatedly reacted with Pd and then removed at 150°C.

compared to the amount of excess energy created by the LENR process.

The behavior observed when Pd reacts with D is important because it helps to identify potentially active Pd and reveals information about the nature of the reacting surface. A metal having a surface free of metallic deposits and activated by the method used here will react initially with most D presented to the surface. Gradually, the amount of D being reacted decreases until all the D created at the surface forms D_2 gas at the composition limit. The sample shown in Fig. 6 is unusual in reaching a D/Pd very near the upper limit of the beta phase composition at D/Pd = 1.

The loading process deposits impurities on the surface, which generally consists of Fe, Cr, Si, and Pt, with the first two being supplied by the stainless steel lead attached to the Pd sample. The effect of these impurities on the loading behavior can be made clear by partially unloading a sample of previously reacted Pd and reloading without removing the acquired surface impurity. Figure 7 shows the loading behavior of a sample after being first loaded to D/Pd = 0.86 and then partially deloaded by heating in air at 150°C . In this case, the impurities deposited on the surface during the first loading allowed only 1 in 10 of the D presented to the surface by the electrolytic current to react. This rejected fraction remained constant until the average bulk material reaches about D/Pd = 0.6, similar to the behavior seen in Fig. 6.

This deposited impurity is not uniform on the surface; hence it affects some regions more than others. Consequently, the local D/Pd is not uniform and non-uniform stress is produced between the different compositions. In general, the greater the electrolytic current, the faster this deposit forms on the surface and the sooner the loading behavior deviates from the ideal. Once the surface deposit is removed by concentrated HNO_3, a repeat of the loading process causes the behavior to match that shown by a clean surface. Apparently, the fraction of D reacted initially depends only on the presence of surface impurities.

In summary, a clean Pd surface reacts initially with every D presented to the surface by electrolysis. Accumulation of deposited impurities interferes with this process more in some regions of the surface than in others. The final D/Pd

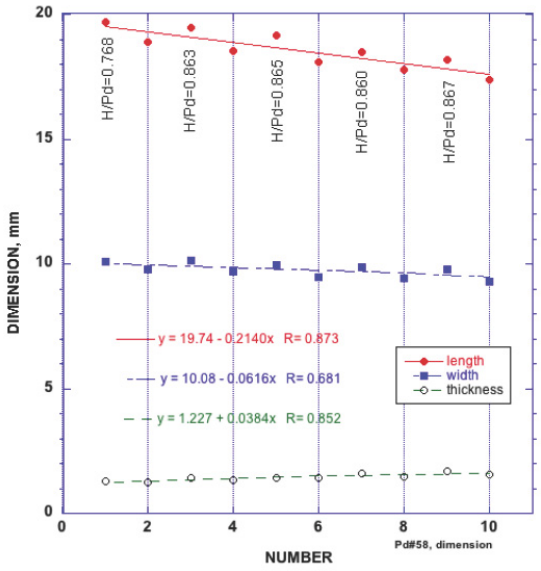

Figure 10. Changes in the physical dimensions of the sample shown in Fig. 9 after reacting with H and after the H is removed, with the values obtained after H is removed plotted on the vertical lines.

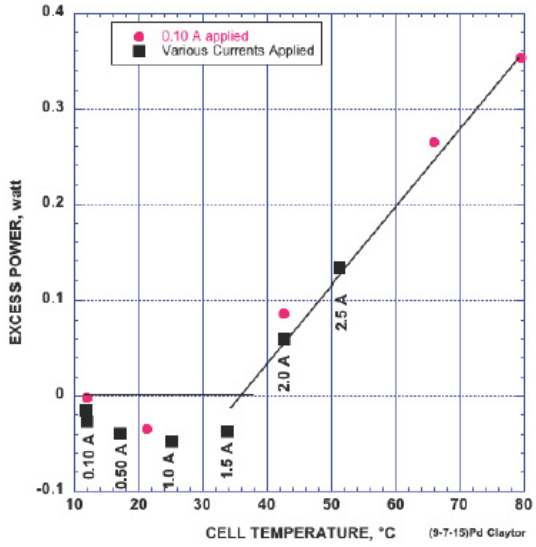

Figure 11. Comparison between excess power produced by applying electrolytic current and by changing the temperature at fixed current.

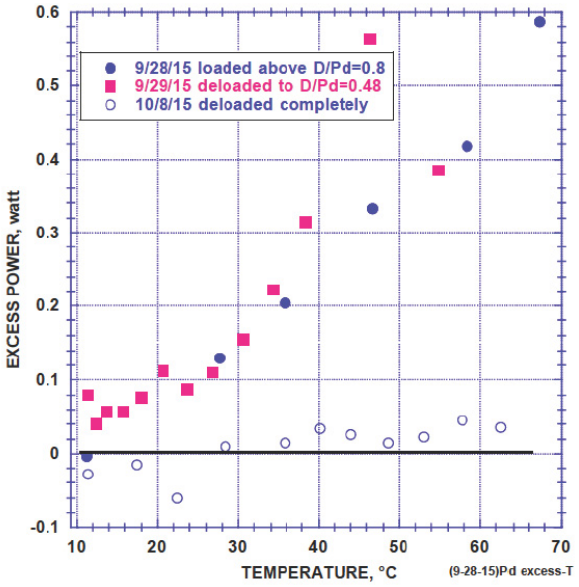

Figure 12. Effect of temperature on excess power when the average D/Pd ratio is changed.

ratio achieved is determined largely by the initial properties of the Pd, which are different for each sample.

2.3. Excess volume

As PdD forms in the material, the shape and volume of the sample change [13]. In general, the material expands in response to the increase of the lattice parameter, as can be seen in Fig. 8. In many cases, the expansion exceeds the amount expected based on the lattice parameter and this extra volume remains after the D is removed. This extra volume is called excess volume and the amount is different for each sample.

In addition, the length and width of the sample shrink while the thickness increases. Palladium that produces more than a few percent of excess volume tends to form large cracks and is limited to a lesser limit to the D/Pd ratio [14]. Consequently, potentially active material is sought among palladium having very little excess volume [17].

Table 1 compares the dimension and volume for the samples used in this study. This expansion is important because it produces stress in the surface region and causes cracks to form. These cracks, depending on their gap width, can cause excessive loss of D_2 or creation of the NAE, in which, according to Storms [2,18], the fusion process takes place. The rate of loading will affect the rate at which this stress is generated and, consequently, the response of the structure to crack formation. The sample used in this study showed production of only 0.21% excess volume, which identified this material as being potentially nuclear active, as later experience demonstrated.

A study was undertaken to examine the volume change in more detail using light hydrogen. A sample was loaded with H and caused to reach the H/Pd ratio, as shown in Fig. 9. The volume increased as expected when H was added, but each time the H was removed and then added again, the volume increased further. The dimensions also changed, with the length and width becoming smaller while the thickness increased, as shown in Fig. 10. Numerous people have previously studied this well-known effect [13,19–21] and reported similar behavior. Repeated loading followed by unloading causes the shape to change with the result that a flat plate tends to form a cube having greater volume

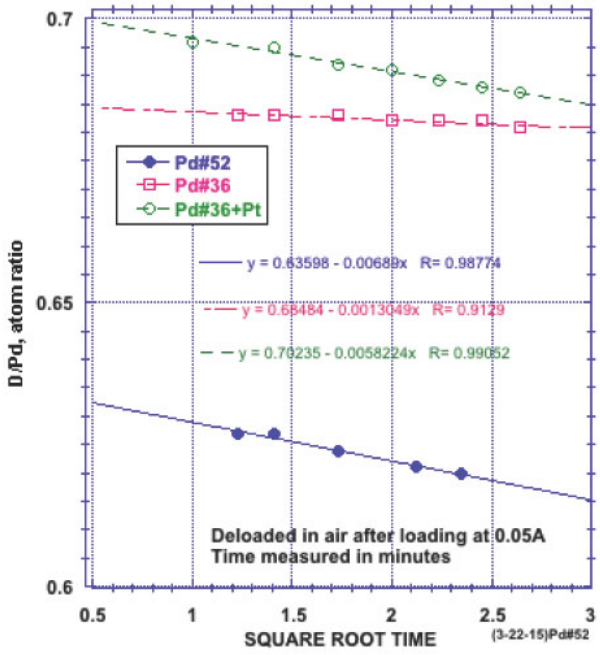

Figure 13. Examples of the change in the average D/Pd ratio as deuterium is lost from various samples of PdD in air at room temperature after electrolytic current is stopped. The least-squares line is extrapolated to zero time to determine the initial D/Pd ratio. The time is measured in minutes.

than the original palladium.

2.4. Excess power

Potentially active palladium does not produce excess power when it is initially reacted with D no matter how high the achieved D/Pd ratio. A process of partial unloading and reloading is required. Even this treatment will not always result in excess power production. Once excess power is produced, it tends to be very reproducible even after some of the D is removed by allowing the D to leak out when electrolytic current is turned off or by heating in air at 150°C. Both samples used in this study were subjected to a loading–unloading treatment before excess power was detected.

Separating the variables thought to affect energy production is the next task. The effect of temperature can be seen in Fig. 11, where the temperature is changed by applying power two different ways. In both cases, the resulting excess power shows the same relationship to temperature. This behavior strongly indicates that temperature alone is the controlling variable.

Figure 12 compares the excess power measured when the average D/Pd is fixed above 0.8 by applying electrolytic current and at a value near 0.48 after some deuterium had been lost by heating at 150°C in air. Excess power production no longer occurs when deuterium is completely removed by heating at 150°C in vacuum.

The D/Pd ratio is determined after each study by weighing the contained deuterium. This is done by measuring the weight change as a function of the square root of time, as shown in Fig. 13, and extrapolating the values to the time electrolytic current was stopped. The total deuterium content is also determined by measuring the weight change after

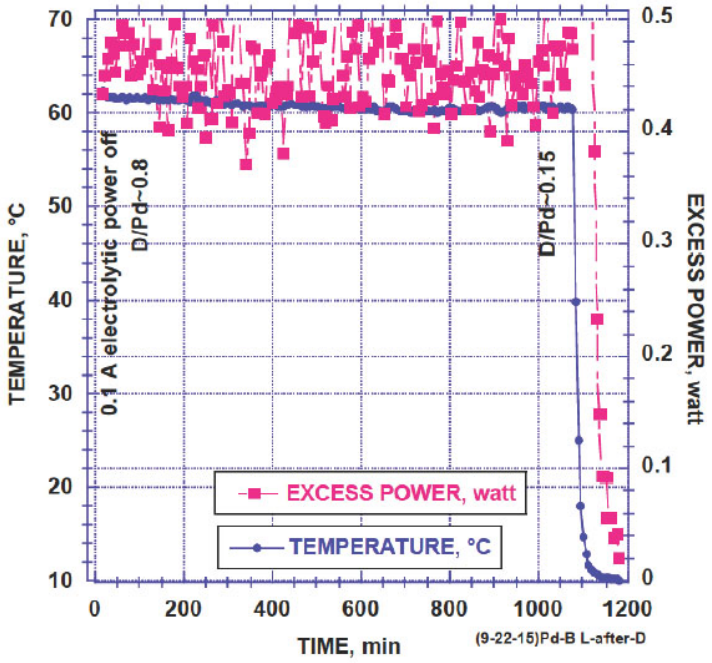

Figure 14. Excess power as a function of time while a sample lost deuterium at about 60°C after applied current was stopped. The excess power rapidly dropped to a value too small to measure when the temperature was reduced to 10°C.

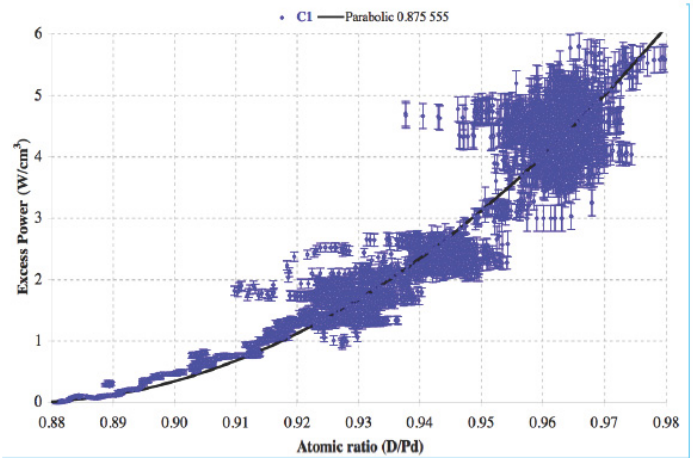

Figure 15. Example of the apparent relationship between the average D/Pd ratio and excess power production [25].

all deuterium is removed. These values represent the average deuterium content; not the deuterium content close to the NAE, which is the composition that would affect excess power production. In view of the non-uniform composition known to be present in such samples, the composition at the NAE is unknown and cannot be assumed equal to the average measured composition.

Turning off the electrolytic current while a sample is making energy produces the so-called "heat after death" behavior, as can be seen in Fig. 14. In the past, Fleischmann and Pons observed energy production to continue for extended times [22]. The various reported runaway heating events [23,24] are consistent with this behavior. In the present study, the temperature is held constant at about 60°C by an internal heater after the electrolytic power is turned off. Excess power remains constant as the average D/Pd ratio changes from above 0.8 to 0.15 as the sample deloads. The excess power continues and drops to an undetectable value only after the temperature falls to 10°C because the internal heater was turned off. A total of 30 kJ excess energy was produced, 2.5 kJ of which resulted from the unloading process.

The deuterium content after the study is determined by weighing the total amount of deuterium lost by heating at 150°C in air.

3. Discussion

Conventional belief says that excess power is increased when either the D/Pd ratio or the applied current are increased. Evidence for these conclusions is shown in Figs. 15 and 16. This study is in direct conflict with the conclusions based on the information in these figures. The conflict is proposed to result because the effect of temperature on excess power production was ignored during the previous studies. In general, applying different amounts of electrolytic current, which also changes the temperature, will change the D/Pd ratio. Consequently, these three variables are interrelated and must be separated in order to find the effect of each.

3.1. Effect of applied electrolytic current

McKubre et al. [25] were unique in attempting to keep the temperature of the electrolyte constant during their measurement of excess power production. Based on the design of the calorimeter they used, this goal would have been only partly successful. Consequently, the effect of applied electrolytic current measured by McKubre, as shown in

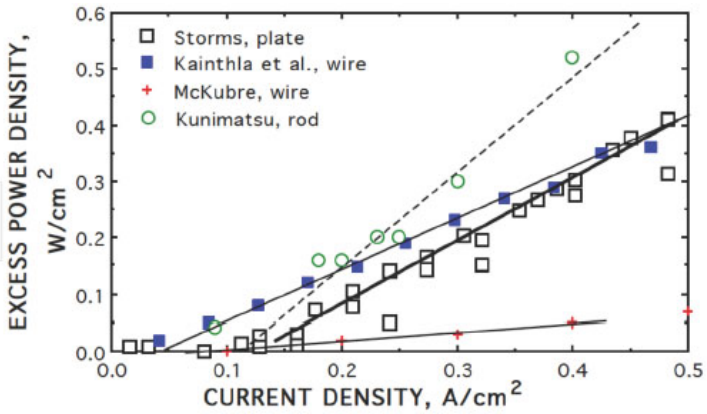

Figure 16. Comparison between several studies showing the apparent effect of applied electrolytic current on excess power production [26].

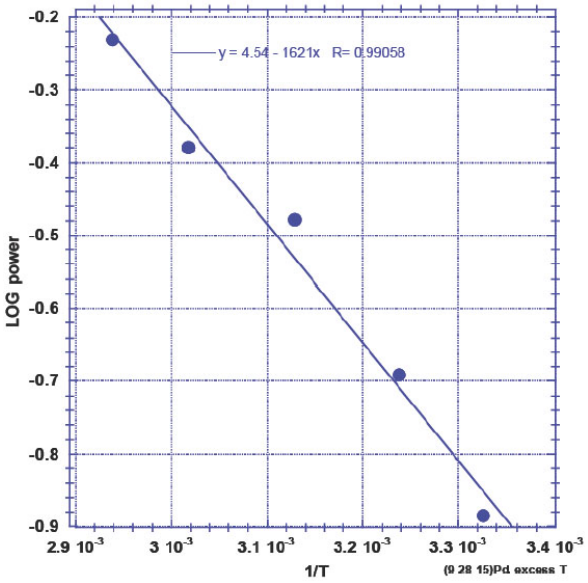

Figure 17. Effect of temperature when plotted as $1/T$ vs log watt and has a slope of 1621.

Fig. 16, is smaller compared to measurements made when the temperature is allowed to change during other studies,

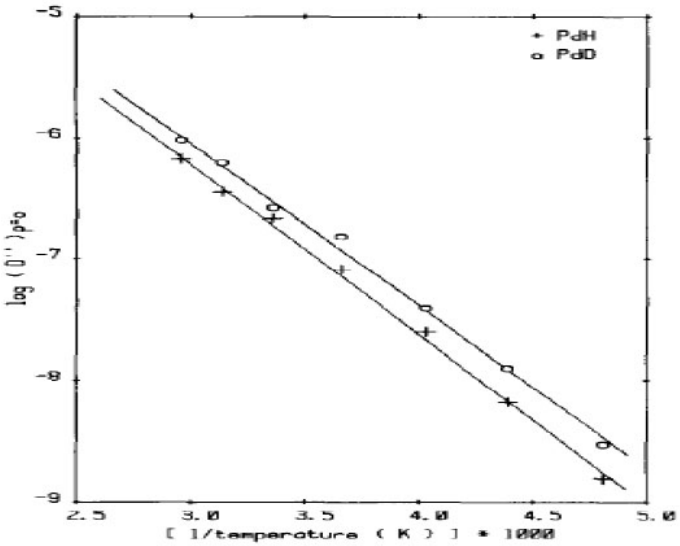

Figure 18. Effect of temperature on the Fick's diffusion constant for PdD and PdH [27]. The slope describing the diffusion of D in PdD is 1760.

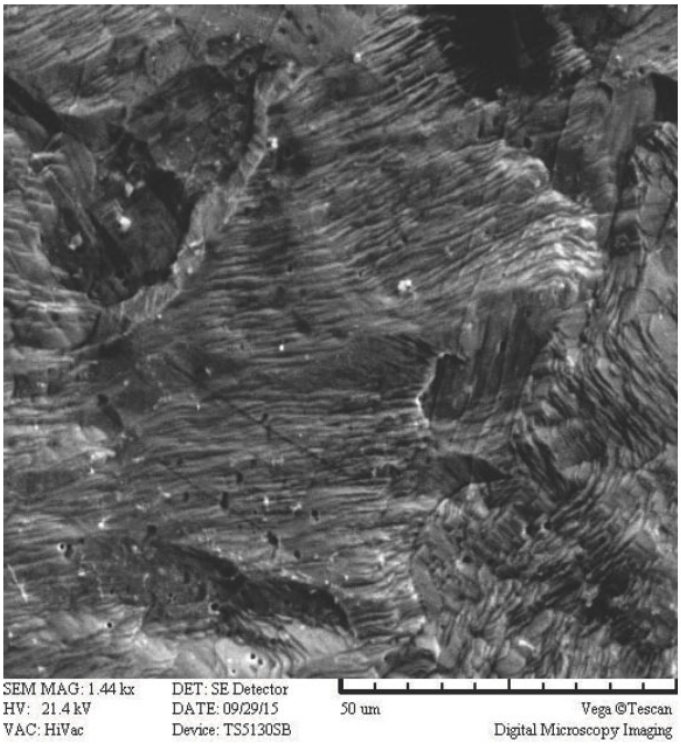

Figure 19. Surface of the active material after excess energy was made.

as would be expected.

3.2. Effect of temperature

The effect of temperature can be presented as an Arrhenius plot (Fig. 17) from which an activation energy of 1.8 kJ/mol can be obtained. The activation energy is very close to the activation energy of 1.9 kJ/mol based on the diffusion of D in the PdD lattice, as shown in Fig. 18. This amount of energy is much too small to directly affect a nuclear reaction. Consequently, the temperature is proposed to influence how fast D can diffuse in the PdD and reach the NAE where the nuclear fusion process takes place.

Diffusion would have a greater effect on power production than would the average composition when the concentration of energy-generating sites is relatively small and the distance over which D needs to diffuse from its source in the lattice is relatively large. These conditions are consistent with the observed non-uniform nature of the surface region where LENR apparently takes place.

3.3. Effect of D/Pd ratio

When the temperature is isolated as a variable, excess power is independent of the D/Pd ratio and applied electrolytic current, being only sensitive to temperature. If the effect of temperature is taken into account, the threshold effect

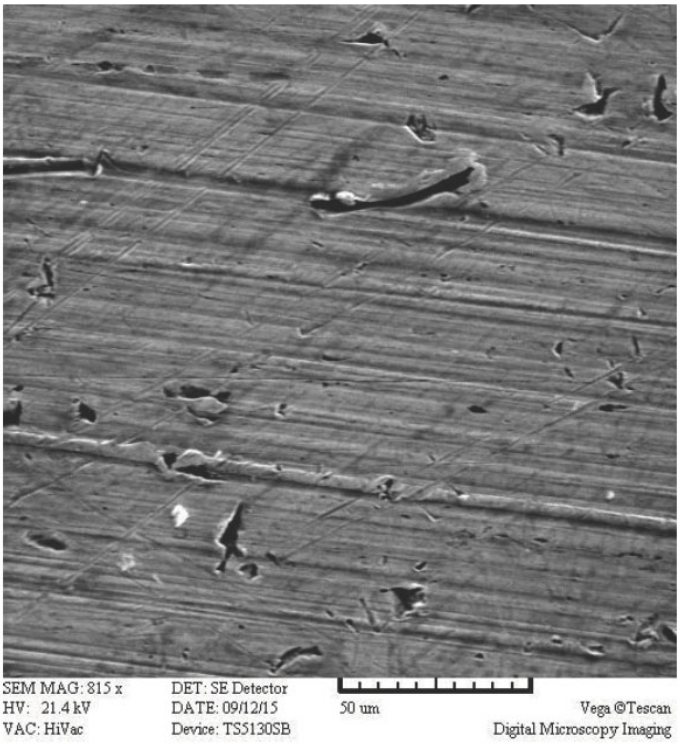

Figure 20. Surface of sample before reacting with D_2.

proposed by McKubre et al. [28] (Fig. 15) does not appear to be present. Clearly, excess power can be produced when the D/Pd ratio is well below 0.88, in contrast to conclusions drawn from the McKubre data. The data obtained in this study show that once the unique conditions required to produce excess energy have been created, power production can be detected after the temperature is increased above a critical value (Fig. 11). This conclusion is consistent with the reports of excess power continuing after the electrolytic current was turned off and even increasing as the temperature increased enough to destroy the apparatus [1,29,30].

In view of the above conclusion, presence of vacancies formed at high deuterium content, as proposed by Haglestein and Chaughary [31], would appear not to be involved in the nuclear process. Apparently, a unique condition continues to support the nuclear process as the average D/Pd ratio changes even when the alpha phase forms in the bulk material. We can assume the deuterium concentration near the NAE is unaffected by the average concentration until nearly all the deuterium is removed. Once all deuterium is removed, excess power production stops and does not start again even after the sample has again reacted with D until an activation process is used.

3.4. Effect of stress

Storms [16] proposes the nuclear-active sites are nano-cracks with a gap near 1 nm, which are created by stress relief. This idea is consistent with the role of volume expansion in influencing the ability to initiate the nuclear process. The potentially active material is proposed to form many very small cracks rather than a few large ones, as is common

behavior when a great deal of stress is applied to the surface by uneven expansion. Most important to the formation of a suitable gap is the morphology of the Pd before it reacts with deuterium. Without this critical morphology being present, no amount of later treatment is able to cause the LENR process. Based on the model of Storms combined with this and similar studies, the potentially active surface is one containing many weak regions having nearly identical strength. Since the initial stress was removed by annealing at 900°C without harming the ability to cause LENR, initial stress is not important. High concentrations of oxygen and carbon in the surface region are also not important.

Regardless of how the initial surface looks, the electrolytic process causes significant changes with few common features seen at the available magnification. The surface of the active sample Pd-#A is shown in Fig. 19 where layers of material have grown. The initial surface, shown in Fig. 20, shows no indication of being any different from normal inactive Pd at the available magnification. Many different variations in the surface condition have been reported to host LENR. A common feature is not obvious at the magnification used. Use of a greater magnification is suggested by this work.

3.5. Role of initial material

Two samples cut form the same piece of Pd were studied and both were found to have similar behavior with respect to excess volume and excess power. Other samples and alloys obtained from other sources produced no excess energy during this study. This experience is similar to the experience reported in the past when the entire batch of Pd containing the active sample is found to be nuclear active [32]. Apparently, the LENR effect is sensitive to conditions created in the material, which are replicated throughout the batch. These conditions then allow a special and unique structure to form in which the nuclear process can occur. The nature of this condition is not yet known although much effort has been devoted to the search by Violante et al. [33].

4. Conclusions

The applied electrolytic current and the resulting average D/Pd ratio have very little if any effect on the amount of excess power produced by a nuclear-active sample of PdD. The only variable of importance is temperature of the PdD. The temperature effect shows an activation energy similar to that involved in the diffusion of D in PdD. This behavior suggests the heat energy is generated in isolated regions, such as the proposed NAE, to which the deuterium fuel has to diffuse.

Electrolysis apparently produces significant deuterium concentration gradients in the material. In addition, reaction with D causes significant expansion, distortion in shape, and changes in surface morphology. Repeated partial loading and unloading are found necessary to initiate excess power production. This process, combined with the composition gradients and shape changes, would produce stress that is proposed to create the NAE.

A critical average D/Pd ratio does not appear to be required to initiate excess power production. Once initiated, excess power will continue at average compositions as low as D/Pd = 0.15.

In this study, as much as 27.5 kJ (1719 kJ/mol Pd) of excess energy was produced without any electrolytic power being applied and this energy production showed no sign of stopping for 1100 min. In other words, the sample produced a (power out)/(power in) ratio equal to infinity for an extended time.

If a sample is found to produce excess energy, most other samples in the batch of Pd will show the same ability, which suggests the critical features are present throughout the batch. The ability of Pd to become nuclear active is not affected by it being heated at 900°C in air for as long as 5 h or by removing about 1 μm from the surface using HNO_3. Apparently, the potentially nuclear-active region is a stable variation of the normal palladium structure located below the surface that needs to be modified in some way to become active.

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