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How to Produce the Pons-Fleischmann Effect

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ABSTRACT

Conditions required for producing excess energy in PdD created in an electrolytic cell are described and reasons for their importance are discussed. This difficult to accept effect can now be produced with a high probability for success using the described procedures.

I. INTRODUCTION

Over five years ago, Drs. Pons and Fleischmann[1] proposed that excess energy could be created by electrolyzing palladium as the cathode in D_2O containing 0.1-1.0 M LiOD. They continued to demonstrate how this amazing effect could be accomplished in subsequent papers [2] and patent applications.[3] Considerable skepticism resulted because the effect could not be easily replicated by a few, well known laboratories[4; 5; 6]; because the effect could not be explained by conventional theory; and because positive results were largely ignored in several popular descriptions of the field[7; 8] as well as by the media in general. Thanks to a few determined scientists, the important variables are now sufficiently well understood to greatly improve reproducibility. In addition, many reasons for past failures are now understood. Skeptics can, if they wish, demonstrate for themselves the reality of the effect.

The claimed effects appear to occur in special environments. For lack of a better designation, these environments will be described as a special condition of matter (SCM), perhaps similar in some respects to the superconducting state. A variety of products have been produced including excess energy, tritium, helium, X-rays, γ-rays, neutrons, high-energy particles of various types and energies, and various elements, both radioactive and stable. Apparently, a variety of nuclear reactions can be made to occur. A better term for the effect might be “Chemically Assisted Nuclear Reactions”[9]. Energy production when deuterium is present appears to be accompanied by the formation of ^{4}He in quantities almost sufficient to account for all of the measured energy [10; 11; 12]. When tritium is occasionally formed, it is accompanied by a few neutrons[13]. With these exceptions, each product seems to be produced independently and by slightly different conditions. This paper will describe only the conditions that produce excess energy in D_2O-based electrolytic cells .

Obviously, the environments in which nuclear reactions can be initiated are unique and difficult to create. One such proposed SCM can be formed after β-PdD (face-centered-cubic structure) has achieved a sufficiently high deuterium content. Once this essential requirement has been achieved, several other conditions are required to form the SCM. These conditions include the presence of certain impurities in the palladium and the application of various forms of energy. Once a stable and sufficiently high D/Pd ratio has been produced, excess energy production starts after many additional hours of electrolysis. The delay time is variable and is proposed to result from slow electrodeposition of the required impurities[14] followed by formation of the SCM[15]. Other changes not yet known may also be required before heat production can start.

The known conditions will be described in sufficient detail so that a careful experimenter would have a good chance of achieving success. However, before attempting to produce the phenomenon, you must realize that considerable skill is required. Information given here can not overcome limitations imposed by poor technique or by inadequate application of the required procedures. Even now, all of the variables are not known. Some must be explored by trial and error. Therefore, do not be disappointed if you are initially unable to produce large amounts of excess energy. Success usually requires patience and several failures. Fortunately, the failure rate can be reduced to acceptable levels.

Of course, a good calorimeter is also required. Suitable designs and required precautions can be obtained from the literature.[13; 16; 17] Although certain precautions are required, success has

been achieved using a variety of designs of the so-called open and closed type[1]. By using procedures described here, sufficient excess heat can be produced to overshadow normal errors in calorimetry.

Excess power levels below 20% are routine while values above 100% are still rare. The highest reported excess power is 1500% [18] using palladium deuteride made in a fused-salt electrolytic cell at 450°C. Generally, the higher the temperature, the higher the heat production

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1. "Closed" cells contain a catalyst that recombines the D_2 and O_2 so that no energy or material leaves the cell. An "open" cell allows the gases to leave, and corrections must be made for this energy loss. Such cells have been criticized because the amount of gas that does not leave the cell may be unknown because some unexpected recombination might have taken place. Most such cells are monitored to correct for this problem if it should occur.

rate. Although the phenomenon can be demonstrated, levels of heat production required for a practical device are still difficult to achieve. More recently, a variety of other methods and materials involving deuterium have been found to produce the effect.[12; 19; 20; 21; 22] These will not be discussed here.

II. CRITICAL DEUTERIUM CONTENT

The first and most important requirement is to achieve the necessary deuterium content in the palladium. An average stoichiometry greater than $PdD_{0.84-0.90}$ is required. The exact value for this critical composition depends to some extent on the method used to measure the deuterium concentration and the nature of the palladium. The average composition is created by competition between gain at the surface, caused by electrolysis, and loss of deuterium caused by several conditions. These two processes combine to produce a concentration gradient within the palladium with the highest concentration at the surface. The magnitude and extent of a critical concentration region, starting at the surface, is proposed to determine the amount of generated energy. Because the critical region is near the surface, a rectilinear-shape (plate) with its higher surface area is a better choice than is a cylindrical-shape (wire).

Gain of deuterium is improved by using a sufficiently high electrolytic current density and thin layers of certain impurities on the surface. The loss rate is reduced by using crack-free[2], high-strength palladium combined with certain surface modifications. These factors interact so that reduction of one can be partially offset by the increased presence of another. Unfortunately, crack-free palladium is rare and difficult to obtain with consistent properties. Failure to use appropriate palladium appears to be the most likely reason for not producing excess energy.

II.1 Uptake Rate

The rate of deuterium uptake is determined by the current density and chemical conditions at the surface. A minimum current current density between 0.10 A/cm^2 and 0.15 A/cm^2 is frequently required[23; 14; 24; 25; 26; 27; 28] to initiate heat production. Occasionally, much higher current densities are necessary. For example, the data of Pons and Fleischmann[3] show an onset near 0.4 A/cm^2, and Storms[29], on one occasion, reports an onset above 0.35A/cm^2.

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[2] A crack is a void space containing D_2 gas that, when it extends to the surface, allows deuterium to move quickly from the interior to the surface as high pressure gas, thereby reducing the interior composition. In contrast, a dislocation is a position in the solid lattice where the atoms do not occupy uniform, periodic positions. These regions tend to trap deuterium atoms, thereby increasing the local composition.

This current should be uniformly distributed over the entire surface of the sample. Failure to apply sufficient current is one of the reasons why excess heat is not observed. Several studies showing this effect are compared in Fig. 1. Differences are caused mainly by uncertainties in cathode area, by nonuniform current distribution, and by different changes in cathode temperature produced by the current. Once production starts, the higher the applied current density above the onset value, the greater the excess power.

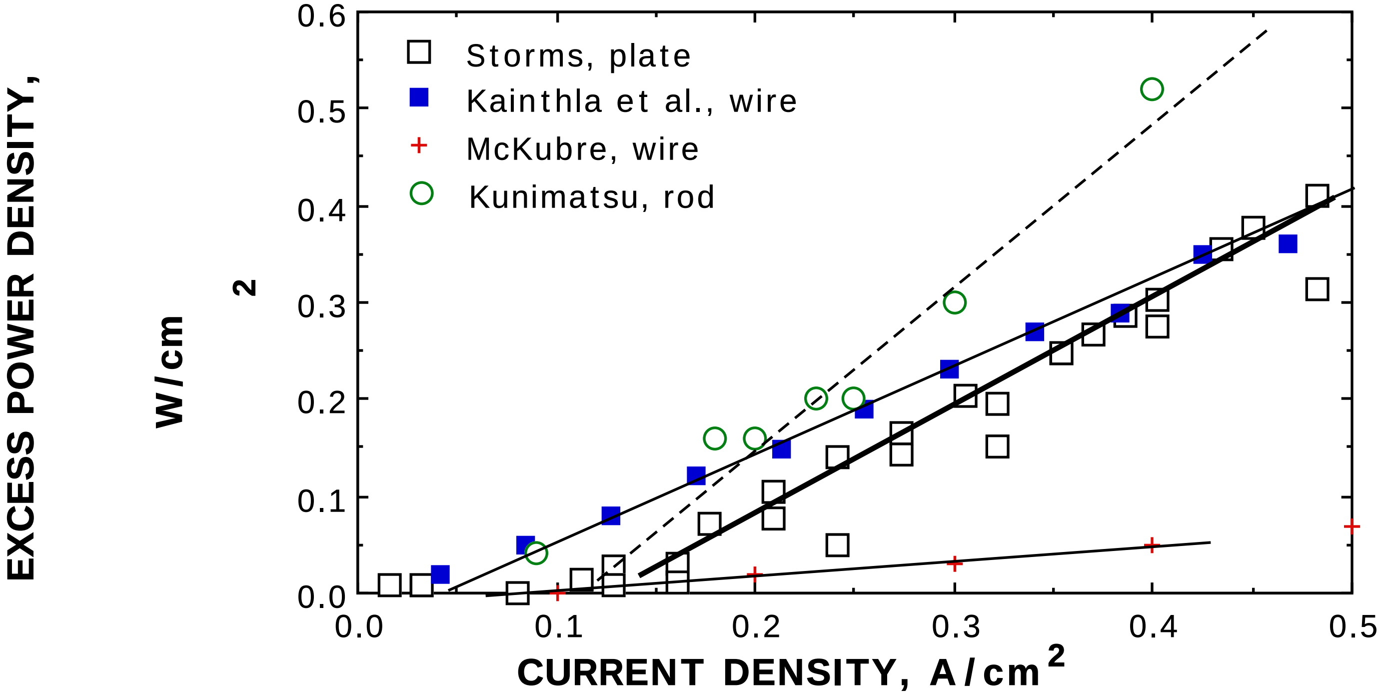

FIGURE 1. Relationship between excess power density and current density.

An average D/Pd ratio between 0.84-0.90 is frequently observed when excess power production first starts.[24; 14; 25; 26] Average values above $PdD_{1.0}$ are occasionally reported.[30; 14] The actual deuterium content of the SCM is still unknown, but clearly above $PdD_{0.84}$ and possibly much higher than $PdD_{1.0}$.

In addition to applying a current density above the onset value, the presence of certain impurities on the palladium surface are required. These effect deuterium uptake and retention. Such impurities can come from the anode, the electrolyte, or the container. Very careful work done at SRI (Stanford Research International)[14] demonstrates how several impurities, including aluminum and silicon, produce beneficial results. These two impurities occur naturally when Pyrex glass is used as the container. Aluminum metal (2-20 ppm) added to the electrolyte after the palladium has achieved its maximum deuterium content is sometimes useful in initiating excess heat production.[14; 27] Thiourea also has been used with limited success.[31] Preoxidizing the surface at 600-750°C in air improves the loading rate[32; 33] because the very thin layer of oxide is quickly reduced to a pure, very chemically active layer of palladium once electrolysis starts. Absence of certain surface impurities such as copper, lead or silver (from solder) is also important. However, a thin film of gold (≈7000 Å) on the palladium surface can increase the limiting D/Pd ratio.[34] The benefits of lithium and platinum, two impurities normally observed on and within the surface region, are still unknown. Other impurities not yet studied may have both good and bad effects.[35]

Because of impurity electrodeposition from the electrolyte and electromigration within the palladium, the chemical purity of the surface is a complex function of time, integrated current, temperature, and purity of the cell environment. Therefore, the chemical composition of the SCM is unknown and probably much different from bulk material.

Regions of stress and dislocations are known to concentrate hydrogen. Dislocations can be created at the surface by polishing and within the bulk material by rolling, drawing, or hammering. Although such methods are not required, their use has produced success. However, initial dislocation and stress are probably overshadowed by that produced during the hydriding process.[36] Unfortunately, stress and dislocation concentration are difficult to measure and are not reported by workers in the field.

Stress can be removed by annealing. Both annealed and unannealed material have produced excess energy. Therefore, initial stress is not required. On the other hand, annealing creates a complicated result. The process can deposit impurities on the surface from the ambient gas, can diffuse surface impurities into the metal, and can cause crystal growth and vapor etching at the surface if done at a sufficiently high temperature. In addition, annealing does not change void and crack concentrations, and may not change the number of dislocations. These consequences make the annealing process unattractive unless considerable care is taken.

II.2 Necessary Characteristic of Palladium

The nature of the palladium cathode is very important. Various types of palladium having purity in the 99.9+% range and obtained from various sources have worked while material of the same purity and obtained from the same source has failed to work. Although the influence of purity is not yet understood, the absence of cracks and voids is critical. Most palladium forms microcracks, voids and dislocations when it is converted to the hydride.[37; 38; 39] Only very unusual palladium is relatively crack-free after hydriding and, consequently, will made excess energy. Pretesting can be used to separate “good” material from “bad”. Because this property is very nonuniform, material from a “bad” batch may occasionally produce heat while some material from a “good” batch may fail. Therefore, testing of each piece is recommended.

Pretesting can be done using two methods as follows:

1. The volume of the palladium is measured using the physical dimensions, and the weight is recorded to ±0.1 mg. The piece is electrolyzed as the cathode in a D_2O-LiOD electrolyte until weight increase indicates an average composition higher than $PdD_{0.75}$. By combining the initial volume (IV) and final volume (FV) with the D/Pd ratio, calculated from the weight change, excess volume fraction can be determined using the equation:

$$\text{Excess volume fraction} =((FV-IV)/IV)-(0.0172+0.14125\ D/Pd).$$

The second term in the equation corrects for the volume change caused by formation of the deuteride. Excess volume is caused by cracks and voids generated within the palladium by hydride formation. If the value is greater than 0.02, the palladium should not be used. Limited experience indicates that a lower excess volume will give greater heat production. More detail is given about this procedure by Storms and Talcott-Storms.[39]

2. Another, less quantitative method is based on observing bubble formation on the surface during electrolysis. “Good” palladium shows no D_2 bubbles until the composition exceeds about $PdD_{0.65}$. Further loading produces a very uniform production of small bubbles.[40] The same pattern continues at a reduced rate should the current be turned off. Production of large bubbles in a few spots indicates “bad” palladium. A successful cathode also shows a higher overvoltage than does “bad” material[41; 42], in part, because a higher surface composition has been achieved.

Crack formation can be reduced if the initial loading rate and temperature are both kept low during the initial loading process. Initial current densities below 20 mA/cm^2 are suggested. Once an average composition above $PdD_{0.75}$ is achieved, the current and temperature may be increased. Many additional hours of charging are normally required to produce excess energy. Various initiating methods (Section III) can reduce this time.

Improved success has been claimed when the palladium has been polished[40], or when it has been cold-worked to achieve an unusually large hardness[43; 44]. Thin films (5-20 µm) of palladium formed on a silver substrate by electrodeposition[45] or an alloy of 0.9 Pd + 0.1 Ag[46; 47]. also gave good success rates. Sintered powder has been used to produce excess energy.[28] In this case, positive results will be very sensitive to the presence of D-D recombination inhibitors coating each particle.

Successful palladium has been found to produce excess energy even after it has been deloaded and reloaded, after it has been kept in liquid nitrogen before being re-electrolyzed, and when heated to 100° C [46; 48; 40] during electrolysis. Bad palladium may occasionally work for brief periods producing low-power, but it can not be relied on to produce significant results regardless of treatment.

II.3 Sources of Unwanted Impurities

Very small amounts of certain impurities can have a very large effect on the results because only a few atomic layers deposited on the cathode surface can significantly affect the limiting D/Pd ratio. Therefore, all sources of chemical contamination in the cell design need to be carefully considered.

Heavy-water sometimes contains impurities that will inhibit the effect.[13; 36] Therefore, the D_2O should be distilled or purified before use. In addition, heavy-water easily picks up normal water from the atmosphere. Excess heat production is significantly reduced when the normal water content is only slightly increased. Therefore, the cell must be isolated from the atmosphere.

The electrolyte is normally made by dissolving lithium metal in heavy-water. Mineral oil used to protect the lithium can be removed by washing the lithium with methanol. Because both reactions make deuterium gas -- although at a slow and controlled rate-- some care must be used to prevent ignition. Electrolyte compositions in the high end of the 0.1-1.0 M LiOD range appear to work better than do compositions in the low end.

Spot welding is sometimes used to attach wires to the cell electrodes. Although this method is effective, it can transfer copper metal from the spot-welding electrodes to the weld. When this metal is redeposited on the cathode surface, heat production is reduced or prevented. This problem can be eliminated by using spot-welding electrodes made of tungsten. Solder of any type, including gold, will dissolve in the electrolyte and be deposited on the cathode.

Use of very pure platinum or palladium for the anode is important because many of the metallic contaminates contained therein will eventually electroplate onto the cathode. Although a slight darkening of the cathode surface will be observed as impurities are deposited, this layer is best kept to a minimum.

A useful method to prepurify the cell is to first electrolyze using a dummy cathode. After several days of electrolysis, this cathode is removed, along with the impurities, and replaced by the cathode to be studied.

The electrolytic current should be uniformly distributed over all regions of the electrode surface. Although a few studies have been successful when electrolyzing only one side of a palladium sheet, this method is not recommended unless the nonelectrolyzed side is coated with a diffusion barrier such as gold.

III. INITIATING METHODS

Application of nonequilibrium conditions can initiate the effect. Very successful results have been reported after using pulsed(1 µsec) high voltages with a resulting high current (up to 100 A)[43], current switched or ramped between two extreme values [40; 29; 49; 50; 51], and MHz frequencies of particular values (81.9 MHz or several other frequencies) superimposed on the electrolysis current or inductively coupled to the cathode.[41] Power levels below 30 mW have been found to initiate heat production. Larger values are expected to be more effective. The reason for this effect is not known with certainty.

Acoustic energy has been used to trigger local, high-energy regions at the cathode surface. These regions result from the collapse of small bubbles created when the acoustic wave passes from the electrolyte to the palladium metal.[12] Excess heat production is reported to be very reproducible .

Excess heat production increases when the temperature is increased.[27] Indeed, significant heat has been produced at 100°C [48] as well as after electrolysis stopped because the electrolyte boiled off.[46] Increasing the temperature after excess heat has been produced can increase the magnitude of the effect. Some caution is needed because this process might become self-feeding.

Bubble formation creates micro regions of current and voltage variations that may produce the required frequencies and local heating in the absence of externally applied energy. This energy source alone appears sufficient to initiate the effect once other necessary conditions have been achieved. However, because the best conditions are seldom present over the entire surface or in a brief time, additional energy is beneficial.

IV. SUGGESTED PROCEDURE

This procedure requires knowledge of the cited literature and some experience constructing and using electrolytic cells of this type.

1. Polish the palladium surface to a mirror finish and wash with acetone. Do not touch with fingers or tissue paper after this step.

Purpose: To remove impurities, to produce a uniform surface, and to create a dislocation layer.

2. Heat in air at 600-700° C until the surface oxidizes to a uniform blue color. This operation is best done in a small furnace. If the color is not uniform, repeat step 1.

Purpose: To remove impurities, to demonstrate uniform chemical activity of the surface, and to produce a layer of pure palladium on the surface.

3. Weigh (±0.0001 g) and measure dimensions (±0.001 cm).

4. Electrolyze as the cathode in 0.3-0.5 M LiOD electrolyte at 20 mA/cm^2 until weight increase indicates a composition greater than $PdD_{0.75}$.

5. Remeasure dimensions and calculate excess volume. Discard if excess volume is greater than 2%. “Good” material may be stored in a sealed container below 0°C indefinitely.

Purpose: To eliminate palladium having excess cracks and voids after hydriding,

6. If excess volume is less than 2%, place the palladium in a prepurified cell and place this cell in a calorimeter. Electrolyze the cell at 200-500 mA/cm^2. If excess energy is not produced within 300 hours, start initiation procedures.

7. The following initiation procedures can be applied in sequence or at the same time:

a. Add 10 ppm of aluminum metal to the electrolyte. Electrolyze for an additional 100 hrs.

Purpose: To create a barrier to reduce deuterium loss.

b. Switch electrolytic current between 20 mA/cm^2 and 500 mA/cm^2. The switch rate can be between 1/sec and 1/min. Continue switching for an additional 100 hrs. Test for excess energy while the current is fixed at 500 mA/cm^2.

Purpose: To create a variable flux of diffusing deuterium and other undefined conditions, some of which are known to initiate SCM formation.

c. Increase the temperature to at least 50°C and continue to electrolyze at 200-500 mA/cm^2. This step is best done after heat production starts.

Purpose: To increase heat production rate.

d. Inductively couple an RF frequency of 81.9 MHz to the cell through a surrounding coil. Power levels below 30 mW have been found to work well but values up to 1 W can be used. If excess energy is to be produced, it will appear immediately.

If none of these techniques work, repeat the process using a new piece of pretested palladium.

V. CONCLUSION

Four major factors are important to achieve excess power using palladium electrolyzed in heavy-water. These factors are: an average D/Pd ratio above a critical value; a cell current density above a critical value; the presence of certain impurities in the surface region of the palladium cathode; and the application of externally generated energy. The closer the chemical conditions are to the ideal condition, the less external energy is required to initiate excess energy production. When a proper mixture of these independent conditions is achieved, a transformation is proposed to take place in the palladium deuteride that produces a special condition of matter (SCM) in which the heat producing reaction occurs. It is unlikely that the resulting SCM is simply another lattice structure in the pure Pd-D system. However, whatever structure the SCM is found to have, its chemical and mechanical properties will be very unique.

Various nuclear reactions can be initiated in the SCM depending on several additional variables. Because these variables are still not understood, some of the nuclear reactions have been more difficult to replicate than others. For example, tritium is produced only occasionally and with difficulty in "normal" cells. Tritium is seldom found in cells producing excess heat.

In contrast to statements made by some skeptics, a person does not have to be a believer to produce positive results. On the other hand, positive results will certainly produce a believer. Anyone reporting negative results should at least show that attempts were made to create and to measure the critical conditions described in this paper before dismissing the phenomenon.

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