

FACTORS AFFECTING THE SUCCESS RATE OF HEAT GENERATION IN CF CELLS

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Abstract

A series of low cost, low precision experiments were conducted to screen for factors which may affect the successful observation of heat from palladium/ heavy water electrolytic cells. Critical factors include the selection of the palladium and the experimental protocol during the initial loading to the beta phase. It was found that bubble patterns, volume expansion, and surface appearance can be used as early predictors of ultimate success. Since large scale defects are detrimental, methods of avoiding cracking are discussed. These include alloying, preparing a uniform surface, loading at a slow rate at low temperatures, delaying use of additives to the electrolyte, and uniform loading techniques. Methods of achieving the later and larger heat releases were found to include: rapid increase in the current density above a threshold value and raising the temperature. A reflux calorimeter design is presented that allows for continuous studies at boiling temperatures of the electrolyte. Unexpected and unexplained occurrences of heat bursts by magnetic fields and radio frequency fields are reported.

Introduction

Over four years of “garage-style experimentation” has resulted in a set of empirical factors which seem to affect the success rate of anomalous heat generation from deuterated metal system. These cells have been relatively small, 2 ml to 30 ml. Most results presented here have come from 3 ml cells. These typically have been run in the constant pressure heat flow mode with constants about 3 degrees C/Watt and with an overall system uncertainty of near 0.2°C or about 0.6 W uncertainty (the error bars on the estimated “excess” are typically 8%). It should be emphasized that this work sought to explore the parameter space affecting cell operation and does not claim rigorous nor accurate calorimetry. A pragmatic approach was taken to see what factors resulted in increases in the temperature differentials between the cell and its bath.

The major factors which affected those differentials can be classified into two general areas. One area encompasses those items which help to achieve high initial loading rates within the metal lattice. The second area involves items which cause dynamic changes of the lattice and deuterium influx and seem to help initiate and control the reactions. Most of the work and information here relates to open Pd/heavy water cells [1]. The work is presented in two stages. The initial loading of the metal should be done at cool temperatures, with low current densities and so as not to disrupt the metal lattice structure. The second stage should be done at higher temperatures and with high current density, and with some dynamic condition imposed on the lattice.

Part 1 - Factors affecting the initial loading ratios

It appears that high loading ratios of deuterium are helpful [2,3], if not required, for the achievement of the anomalous heating events. Although the dynamic conditions discussed in Part 2 may cause local regions of high deuterium ratios, by and large the greatest factor in achieving the heat is to conduct the initial loading of the metal host lattice so as not to cause damage which would prevent a large deuterium ratios in the ultimate heat generation stages. The following steps should be taken to achieve a large initial loading:

- a) Select host lattice materials
- b) Prevent cracking of the host lattice
- c) Load the cathode in a uniform manner
- d) Avoid contamination.

Selecting host lattice materials

The metal host selected to contain the deuterium is important for the observation of anomalous heat effects. Remember that the goal is to load D (D = deuterium isotope of hydrogen) into the host at a rate faster than the D can escape. This means that conditions which increase the egress of the D should be limited and conditions which increase its entrance should be enhanced. We will see that the proper loading techniques and the limitation of cracking are important factors in reproducing the effects.

Reasonable care should be taken in selecting the Pd which will be used for the metal host lattice so that it can hold a high atomic ratio of D to Pd. High purity (>99.5%) Pd is recommended. I use Pd from Aldrich or investment grade Pd from Engelhard. It is especially important that it have low levels of Pt impurities. The Pd should be visually inspected to assure it is free from obvious cracks, voids or Lutter lines. A quick dip in formic acid or an electrolyte to wet the surface sometimes helps to reveal the structure of the material.

It was empirically determined that the bubble patterns on the electrodes are often predictors of excess heat. A “good” sample will initially take up hydrogen (i.e. the deuterium isotope) very readily at low current density. A “bad” sample often exhibits bubbles very early in the initial loading stages. It is often disconcerting to see marked differences from samples cut from adjacent regions of the same wire or rod. Since the initial loading should be done slowly over an extended time, it is recommended that several samples be initially loaded at the same time. If a number of similar samples are loaded simultaneously and in series, their relative hydrogen uptake can be discerned by their bubble patterns, the “best” samples of such an array will be the last to show bubbles at their surface. It is thought that this signals higher loading ratios (i.e. it can take in more hydrogen) and its surface conditions allow less recombination. A “good” sample should load to 0.6 (or even 0.8 with the Rh alloy) before it forms hydrogen recombination bubbles at its surface. This delay in bubble formation is perhaps the single best way to predict a sample’s future success.

It is also possible to screen samples by their volume expansion. First, the Pd is measured and loaded to its beta phase (or 0.6 to 0.7 D/Pd). Then, the samples are measured and their volume change noted. "Good" samples do not increase their volume more than 10%. "Bad" samples have volume changes of 15% or more.

Clear quartz (B&L spectroscopic tubes) or Pyrex containers allow observation of the cell's operation. If the current is momentarily stopped and the D/Pd ratio is over 0.6 to 0.7, deuterium can be seen out gassing. Uniform and very fine bubbles usually indicate a good piece of Pd. On the other hand, localized regions of large bubbles indicate regions where there are pathways for the D to rapidly de-load from the lattice. If you observe such regions, you should try again with another piece of Pd. Microscopic examination usually show voids, cracks, or other defects in such regions. You can eventually come to recognize potentially good pieces of Pd by observing bubble patterns.

A quantitative method was designed to catch the bubbles. The initially loaded samples were placed beneath an array of small test tubes. After the normal runs, the samples were taken out of the cell. As they out gassed, the hydrogen was allowed to displace water in the tubes. This gave a function of bubble volume (i.e. released hydrogen) as a function of length. A large localized bubble yield is reflected in the increase of the standard deviation of the volumes of hydrogen (deuterium) collection as a function of length. The samples with largest standard deviation of out-gas bubbles as a function of length (that is, most of the gases released from small areas) are seen to be "bad" samples and the ones which gave little or no anomalous heat. One can gain experience to visually predict "good and bad" samples at early loading stages by experience in bubble observation. It is expected that those with very costly and laborious data systems may wish to screen samples by bubble patterns and initial loading characteristics before spending large amounts of time and effort on "bad" samples. It is recommended that such individuals use an array of samples and select only those samples which are most likely to achieve the effect by watching the bubble patterns at early stages and checking their expansion in volume.

Preventing cracking of the host lattice

Care must also be taken to prevent cracking of the metal host lattice after you have selected a good piece of Pd. A larger piece requires greater care in loading and handling. The reason is that the Pd lattice expands as it loads. The loading is analogous to heating a piece of glass. Large pieces cannot withstand rapid expansions without cracking. However, smaller pieces can more easily withstand abrupt expansions. Thus, it is best to slowly load the Pd to avoid internal stresses. Such stress and strain would result in increasing the D pathways out of the Pd. If cracks do develop, it is best to remelt and recast the Pd and start again. It is very rare that Pd which is once cracked is ever successful in demonstrating the anomalous heat effects. Likewise, it is better to scale wire systems by length rather than by thickness.

The cracking difficulty can also be overcome by use of alloys (such as 20% Ag in Pd, 5% Re or Rh in Pd, 5% V and Sn in Ti, etc). The 10 to 25% silver alloy of Pd resists cracking on Deuterium absorption. However, it does lengthen the loading time due to decreasing the D diffusion rates. Additions of Li can shorten loading times. The 5% Rh in Pd seems to be best suited for achieving anomalous heat effects. However, such alloys are extremely difficult to form and are quite costly, as a result, very little experimentation was done on such alloys. Another tactic is to plate Pd on Ag or another substrate such as Ni or Cu [4]. The advantages are two-fold. First, the plating can be performed so as to partially form Pd D. This avoids cracking that results

from expansions during rapid loading. Second, it provides better thermal conductivity, [note – It is recommended that the plating be done in an ammoniated solution and the film should be at least 5 to 20 microns thick.]

It is also often beneficial to anneal the sample to relieve internal stress and strains. However, remember that as the sample is annealing, impurities such as Pt, Cd, etc can migrate to the surface. If you anneal, be sure to grind and polish the surface to remove such impurities after annealing. Otherwise, the sample will not “take up” hydrogen. (Pt catalyzes hydrogen recombination). Instead, the hydrogen will simply recombine at the surface and you will notice bubbles early in the loading process. In other words, you must always follow any annealing with polishing and surface preparation.

Loading the cathode in a uniform manner

The shape and surface texture of the cathode is also important. The cathode should be shaped to avoid sharp or jagged corners. Such points do not allow proper loading to be obtained. The idea is to remember to minimize E-field gradients across the surface of the host lattice. Such variations would lead to unequal loading. The D would then tend to diffuse through the Pd and then escape from the lattice from areas with the least surface E-field directed to accelerate the D⁺ flux into the lattice. This means your loading ratio will be limited, in part, by that portion of the surface area of the Pd which has the minimum over-potential (more exactly, the smallest absolute value of a negative potential).

Although rough surfaces may hasten early loading, they seem to decrease the ultimate loading obtained. This is one of the major causes of confusion at understanding variations in success of different samples. Such rough samples contain deep grooves or valleys which are normally the regions with the least surface potential. I polish the Pd to avoid such difficulties. This is done by first polishing on a buffer wheel. The purpose is to round any burrs, rough areas or sharp corners. The Pd is then polished by a series of finer aluminum oxide powders and finally with cerium oxide (optical grade) on a cotton cloth.

A very revealing observation was made on a sample that was polished only on one side. The other side was rough (from a hacksaw). The experiment resulted in a distinct warping of the sample with the greater volumetric expansion on the rough side (approximately 15% greater expansion over the smooth side was estimated from the thickness and curvature). Storms [5] has established such large volume expansions are counter productive for the observation of the heat effects. A series of experiments were conducted with several different surface preparations on thin strip samples. Figure 1 depicts the results of those few experiments.

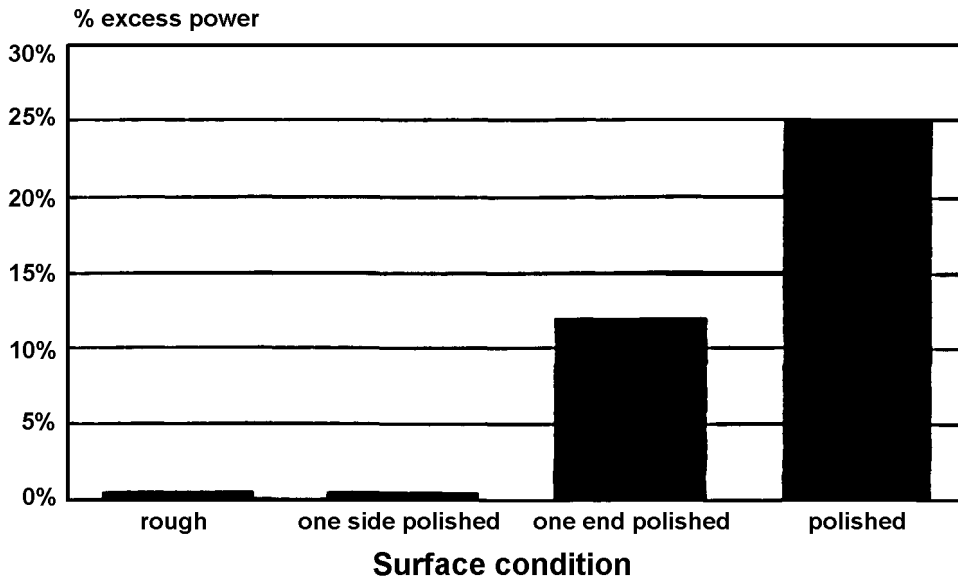


Figure 1. Surface conditions – use a smooth surface

Rough surfaces on the anode should also be avoided. Burrs and points seem to be sites of oxidation and corrosion (this has been seen with nickel anodes). I sometimes use an entire “spiral loop” as an anode with both ends outside of the cell. If you have a free end, you may wish to place it in glue (silicon rubber, epoxy...) to prevent corrosion from the point when it is run for extended times. The best results seem to come with Pt anodes but if you use Ni it should be oxidized first (flame heating or with hydrogen peroxide solution).

It is important that the initial loading of the Pd be done slowly and carefully. The object is to use a low current density (30 to 60 mA/cm²) so that there will not be unequal expansion and the development of large internal stresses. **IMPORTANT** - Do not be tempted to raise the current above 100 mA/cm² until the ratio within the lattice is at least above 0.6 to 0.7 range. If you use pulsing techniques, do not raise the peak current levels above the 100 to 200 mA/cm² levels until the Pd is loaded to at least 0.65. You can calculate the amp seconds needed to load the lattice to 0.7 or better. I prefer to load much longer, until about 150 Amp*hours have passed for each cubic centimeter of Pd (with at least one dimension <1mm). Be patient. It is better to spend too much time in the initial loading stage than too little. Any sudden application of large currents before it reaches the beta phase is likely to crack the host lattice. This could render the Pd useless until it is recast. Some early studies did not produce anomalous heat because researchers used obviously inappropriate samples, or they tried to load too rapidly or too early, and then used the cracked and stressed Pd over and over again. Be cautious of experimentation using the same piece of Pd over and over.

A series of experiments were conducted to see the effect of various initial loading rates on the % excess power produced (all compared when running at 50°C and 500 mA/cm²). Notice (Figure 2) the cathodes which were slowly loaded outperformed those which were initially at a high current density. It should be noted that rapidly loaded wires often had a greater volume expansion.

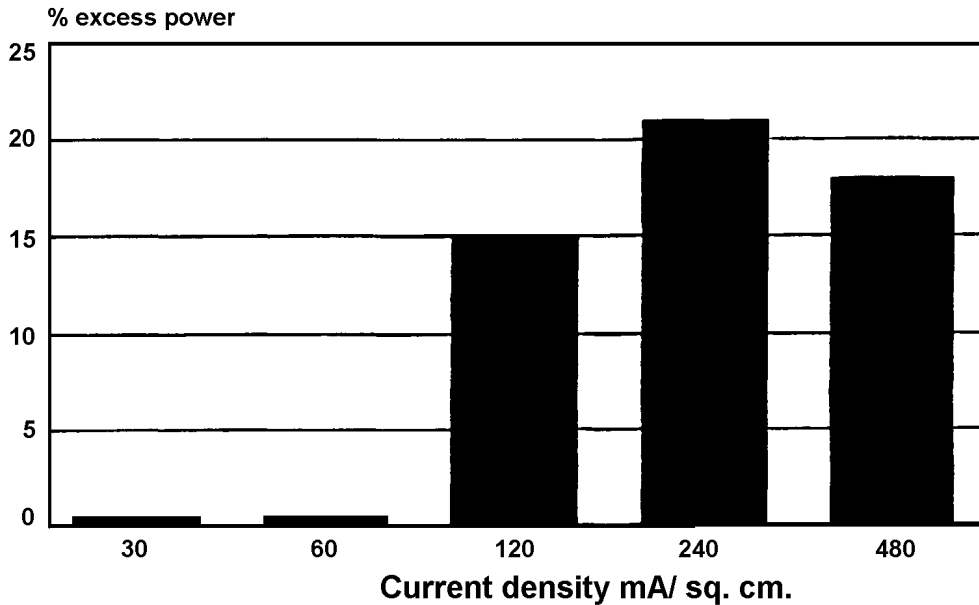


Figure 2. Initial loading – use a slow initial loading.

It would seem that one role of the additives is to increase the over-potential and the internal fugacity within the lattice and select certain electrochemical pathways. Thus, do not add the poisons [2] or other additives (AT, thiourea, Si, B, Mo...) until you are above 0.6 to 0.7 D/Pd. Adding such materials too quickly could cause a creeping or spreading of any preexisting cracks or voids since the gasses build up within such voids. However, if you slowly load the lattice before the additions, the expansion of the lattice on absorbing the D may help to seal such voids. Thus, the goal is to first load slowly to allow the lattice to heal any voids (see Figure 3). Materials which can increase the internal fugacity should be added only after most internal cracks and voids are self corrected.

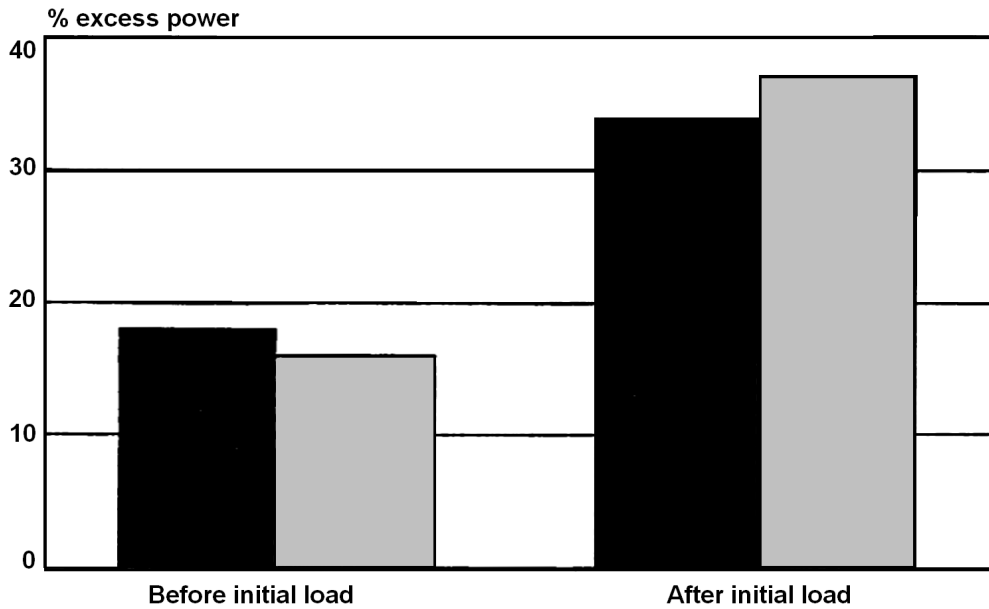


Figure 3. Time of additives – use additives after loading.

The shape of the anode can also affect the results. The anode should have the proper geometry to load the metal host in a uniform manner. Figure 4 shows the results of changing the number of turns of a spiral wound Pt anode. The idea is to have enough turns to supply a uniform field at the metal lattice. This is similar to trying to increase the internal pressure of a balloon. A few points of pressure do not suffice. It takes uniform pressure from all sides. Likewise, since D has a high diffusion rate, large internal D ratios should be attempted by using a uniform potential across the entire surface of the metal host lattice. Otherwise your ratios will be limited by that portion of the electrode with the smallest (absolute) potential.

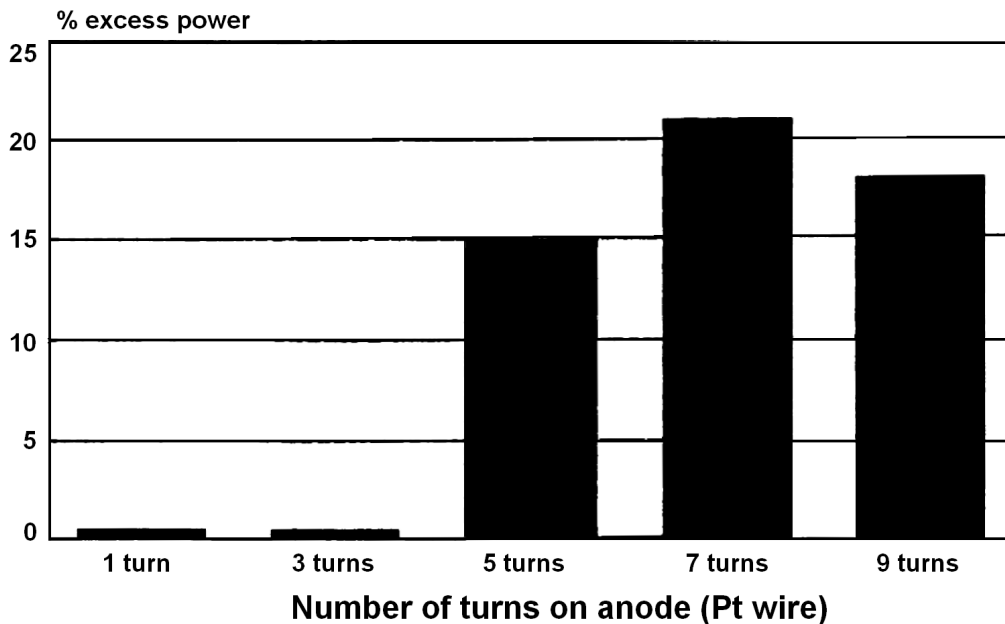


Figure 4. Anode Configuration – use uniform anode spacing

Avoiding some kinds of contamination

Impurities can be harmful to heat generation. I clean the anode and the electrolyte by running it in a separate system with a dummy electrode. This removes any reactive species and most impurities from the platinum and plates them onto the dummy cathode. It also removes some impurities that may be in the electrolyte. The dummy electrode is then removed and with it any such impurities. If Ni is used as an anode, it should be first oxidized to render its surface passive.

Some experimenters have placed their electrical connections to the anode inside their cells. This can cause materials from the electrical lead to enter the cell and be plated onto the cathode. The foam and spray within the cell, over an extended period of time, will wet the connections and contaminate the cathode. It is advisable to keep the anode connections well out of the electrolyte and preferably out of the cell.

On the other hand, the connections to the cathode should be well below the surface of the electrolyte. Remember that the highest loading ratio achieved by your cathode is limited, in part, on the portion of the surface of the cathode which has the least inward deuterium flux. Thus, the metal host lattice should be well under the surface of the electrolyte and the anode should be positioned to extend above and below a cylindrical cathode. The idea is to have all surfaces on the cathode at approximately the same potential with respect to the incoming deuterium ions. I use platinum, silver or nickel for the connection to the Pd. If you use silver or another more active material, be careful not to turn off the current or reverse current for extended periods unless it is electrically and chemically insulated. This can, in some cases, cause oxidation of the connections and the transmission of ions from the connections into the electrolyte.

Heavy water is hygroscopic and will uptake normal water from the atmosphere. When working with large open water baths, you should take steps to prevent the cell from taking in normal water via the atmosphere. One approach is to connect a tube to the cell and run it to a U tube with heavy water or oil. Another approach is to keep an over-pressure of deuterium in the region above the cell. This limits water uptake and also keeps the electrolytic pathways favoring those which lead to deuterium. You can bubble deuterium in at the bottom of the cell. This complicates the calorimetry but is not too bad if you reach the point of seeing high levels of anomalous heat. It also lengthens the time of usefulness of some additives such as thiourea. It should also be mentioned, that a small amount of water (about 1%) seems to level out heat bursts but that high levels (in excess of 10%) seem to prevent the reactions. Such mixtures (10% H₂O) can be used as controls.

Part 2 - factors creating dynamic conditions

Only after the initial loading stages should the system be subjected to large dynamic conditions. Subjecting the system to dynamic or non-equilibrium conditions (after the initial loading) can cause regions of larger deuterium ratios than the global average over the cathode [6]. Such dynamic conditions often initiate or alter the heat generation from within the metal lattice. The act of changing the temperature (large rate of heat gain as a function of time), going to a higher current density, imposing RF signals on the cell, imposing a magnetic field in the region of the cathode, or altering the geometry of the cathode often cause the initiation of greater heat flux (bursts or sustained bursts) from the cathode.

Changes in temperature

The greatest anomalous heat yields were from cells operating at temperatures above 75°C and when used in conjunction with large current densities. The heat effects often seem to be triggered by loading slowly at 20 to 30 degrees and then running in a heated bath at 60 or more degrees.

The anomalous heat yields seem to be more noticeable at higher temperatures (see Figure 5). Due to the large possible errors, the trend line in the figure (as well as others in this article) should only be used to indicate an increasing function and not necessarily a linear one. The greatest sustained heat yields to date by the author have been from cells running at near 100°C [7]. These were outfitted with a reflux column so that prolonged boiling could be sustained. The device is diagrammed in Figure 6. It should be noted that the condensation is returned off-axis to prevent returning cold and non-conductive water directly to the region of the electrode. After calibration, the cell is run with a constant inlet temperature (about 40°C). The flow rate is adjusted manually so that the bottom of the reflux condenser stays at a nearly constant temperature (60 to 80°C). The idea is not to quench the reaction by returning overly cool water, or water devoid of electrolyte, to the region of the metal host lattice. The off-axis return and the elevated temperature at the return portion of the reflux column are subtle but important features.

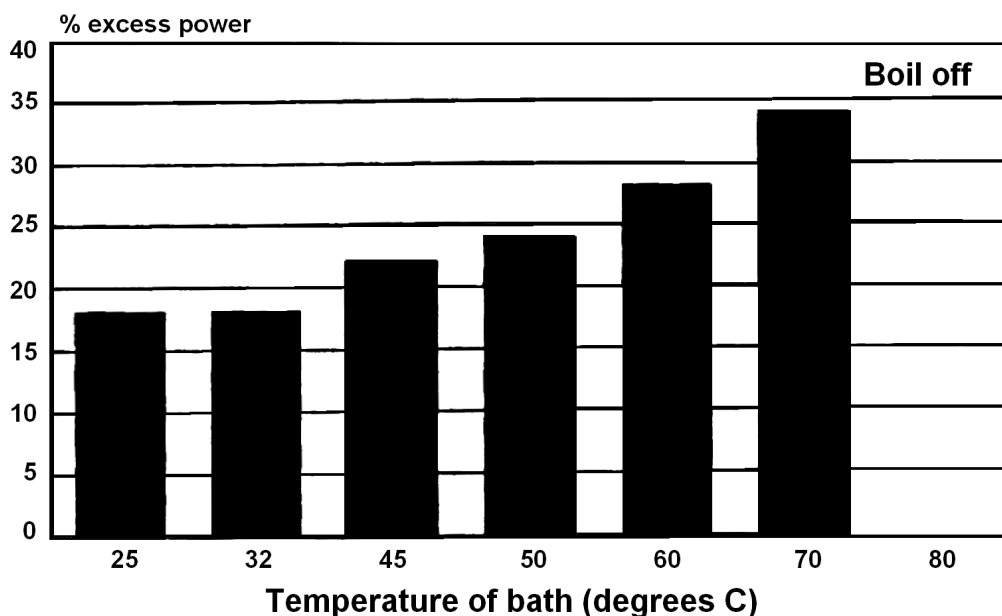


Figure 5. Temperature – run at elevated temperatures

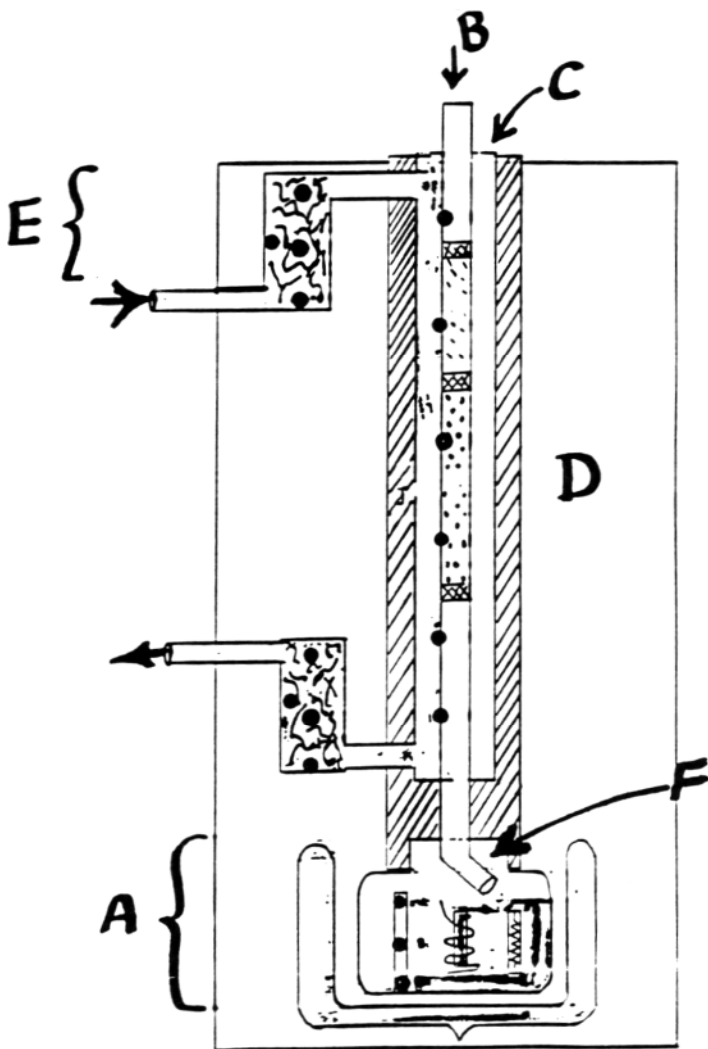


Figure 6. Diagram of calorimeter

The calorimeter is made up of: A) the electrolytic cell contained within a Dewar flask, B) a central reflux column containing crushed aluminum oxide for a vapor condensation surface and recombiner material (not shown is a Tygon tubing connected to top and turning downward to prevent rapid escape of hydrogen and convection currents), C) an outer water jacket to provide cooling to the reflux column, D) a series of insulating layers, E) inlet from a constant temperature bath fitted with copper strip packing to prevent laminar flow in the region of temperature sensors, and F) an condensate return directed away from the active metal host. The temperature differential between the inlet and outlet was held relatively constant. The upper inlet temperature was held constant by the bath temperature. The lower outlet temperature was held approximately constant by adjusting the flow rate.

Increases in current density

Although it is best to load at a low current density (30-60 mA/cm²), the anomalous heat effects are not seen until the current density is raised [8] (usually at least 200 mA/cm² and normally near

500 mA/cm²). Recall that a low initial loading rate is required to prevent cracking of the lattice and ultimately the escape of the deuterium from the lattice. Deuterium ratios above 0.8 can usually be reached only when there is a large influx of deuterium from high current densities. There does not seem to be a universal threshold for the current density for all cathodes. Instead, it appears that there is a range somewhere near 200 to 700 mA/cm² for most “good cathodes.” The best cathodes have a tendency to have a lower threshold than do the marginal cathodes. It would seem that the good cathodes leak less (perhaps due to fewer cracks for deuterium to leak out) and can reach higher ratios at lower current densities. It also seems that the initial slow loading is best at lower temperatures and low current densities. However, the heat production should be done at an elevated temperature and at higher current densities.

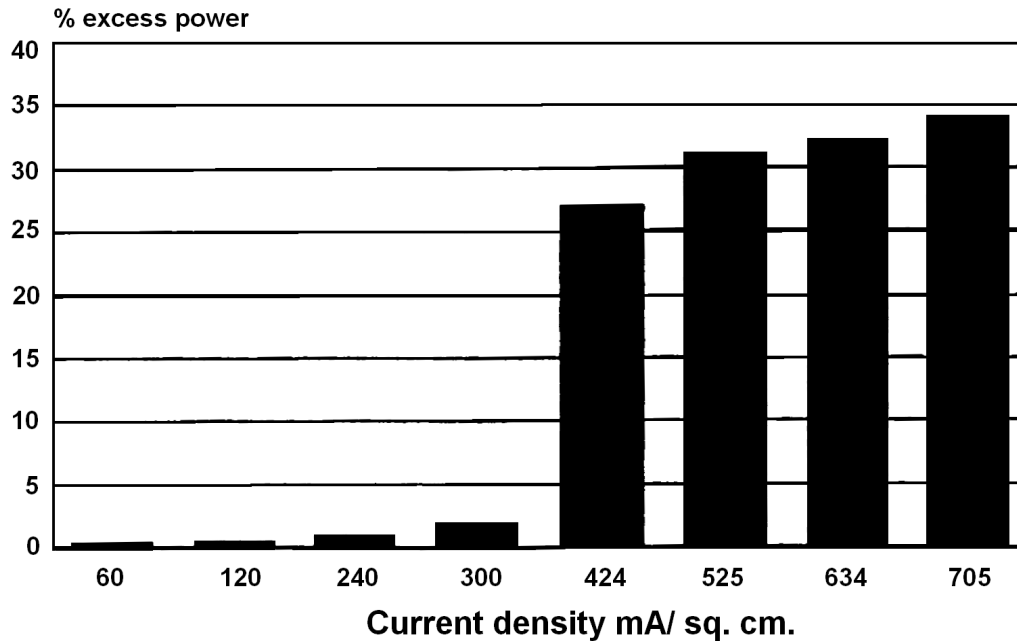


Figure 7. Power versus current density – run at high current

One can make the analogy to filling a bucket having a hole. If the bucket has only a small hole, it can be filled with a small inflow. If the bucket has a large hole or several small holes, it can only be filled with a very large flow. Using higher current densities works, in part, by increasing the inflow above the out flow of the deuterium. Holding the current low during the initial stages works by decreasing the number of “holes” from which the deuterium will leak when one goes to the high current densities after the initial loading.

A few rapid pulses of current may also set up localized regions of high deuterium ratios within the cathode. The use of dynamic currents that are more rapid than the deuterium diffusion times can lead to high deuterium ratios in some regions of the cathode [9]. Changing the electrolysis current can set up dynamic variations and non-equilibrium ratios of the deuterium within the cathode. One way to do this is pulse the electrolytic current [10]. The goal is to set up conditions that will cause a crowding of the deuterium within local regions of the cathode. The pulsing is usually done while keeping a D.C. bias on the current. In other words, the pulses should add on top of a D.C. bias. The bias is selected (for example 60 mA/cm²) so that the cathode is not in a reverse bias condition. This prevents any de-loading that could occur if it was rendered anodic.

For those new to CF cells, it is recommended that they first slowly loaded (30 mA/cm² surface are for 150 Amp*hours per cc of Pd), then slowly ramp to 60 mA/cm² (over 2 hours), and finally start pulsing between the 60 mA/cm² and 500 mA/cm² levels. The pulses should have a fast rise time and a period between a few seconds to a few hours long. I use a period of 30 min and a 50% duty cycle.

It is also possible to pulse the current through 0 volts and reverse the current. If this protocol is used, it is important that the Pd never be de-loaded past the beta phase and into the alpha phase. This method may be a somewhat better than the above one. (However, the D.C. bias method is recommended until experience is gained at recognizing loading ratios.) The mechanism is currently unclear. However, cycling of the cathode often seems beneficial. Higher current densities can be reached by decreasing the anode to cathode separation and adding electrolytic agents, such as lithium sulfate, to increase the conductivity.

Imposing a magnetic field on the metal lattice

It should be emphasized that this section (magnetic effects) is only preliminary. It deals with inexplicable, but tantalizing, observations. They are mentioned here only in hopes that others may find them interesting. Again, the reader is cautioned that due to the complexity of the calorimetry, the effect due to magnetic fields may be spurious and certainly needs more work before instrumental errors are ruled out. Basically it appears that a magnetic field seems to alter the heat flux from cells. Due to the complexities of calculating the magnetic field in the region of a paramagnetic material (and surface dendrites), it is hard to characterize. When a cell is placed in a magnetic field which has approximately 2000 gauss (as measured in air between the poles before placement of a small cell), it often changes its heat flux. On several separate occasions the author has witnessed a change in heat flux as a cell was placed within a magnetic field (above 200 gauss) or when an electromagnet was switched on. The effect is more pronounced when the magnetic field is non-homogeneous. This is now being investigated. This was observed in a small cell (3 ml) by bringing it to a magnet with a pointed iron bar on the opposing pole. Since it is a simple thing to try and nondestructive, others are invited to try it on their cells. So far the effect has only been seen to change the flux on cells that are already exhibiting some degree of anomalous heat.

The imposition of RF fields in the range of 80 to 84 MHz likewise seem to trigger some anomalous heat reactions. This is done by pulsing (both sine and square seem to work) the magnetic field at the cell by wrapping the cell with a coil of wire and connecting it to a RF unit. The excess heat is enhanced by the application of the RF magnetic field. The apparent excess was roughly proportional the power level of the applied field. The effect was most pronounced when the RF coils are impedance matched at about 81.9 MHz (impedance matching units and Sanding Wave Meters are recommended) for optimum RF power transfer. This work was done at 200 mW of power and 5 to 10 turns on the field coils. Others have reported [11] larger effects when 1 to 2 watts of RF power were used. Assuming a reasonably working cell is used (already at about 30%), the effect usually is quickly seen (within seconds or minutes). If the cell is run at above 70 or 80°C, the additional power levels are often large enough to cause rapid boiling. The increases are typically from the initial 30% to the 100-200% range and remain as long as the RF field is on. Often, cells receiving this treatment later seem to “run better” (>than the initial 30%). Again, this magnetic effect is preliminary and exploratory but is dramatic and others may wish to investigate the effect.

Conclusion

Experimental protocol is especially important for successful observation of the anomalous heat. Due to the subtle and complex interactions going on, special care must be taken during the initial loading of the metal lattice. Unfortunately, the multi-disciplinary skills required for proper exercise of the effect is not always available to the new or specialized experimenter. It is hoped that this crude empirical investigation may help others who do not have the time or range of background to fully characterize the experimental parameters and that they may avoid undo experimentation.

For the greatest likelihood of observation of anomalous heat effect:

1. Select Pd or alloys free from visual cracks voids, etc.
2. Prepare surfaces by finely polishing or other consistent methods.
3. Select samples for extensive studies only from those most likely to give the effect.
4. Use bubble pattern observations to narrow selections.
5. Load uniformly.
6. Wait until initially loaded to use any additives.
7. Quickly raise current, but only after the initial loading.
8. Work at elevated temperatures for greater efficiencies.
9. Flow calorimetry used on reflux condensers is especially recommended for those seeking extended studies on boiling systems.

In brief: load cool and slow then run hot and fast.

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