

Chapter 7

Replications

7.1 Fleischmann

In 1989, Martin Fleischmann and Stanley Pons reported experimental evidence suggesting that nuclear fusion could occur at room temperature, which they referred to as ‘cold fusion’ [232]. Their work, conducted via electrolysis of heavy water (D_2O) on palladium electrodes, reported what they described as anomalous energy production, later referred to as ‘excess heat’, beyond what conventional chemical reactions could explain. Fleischmann and Pons employed calorimetric methods to measure energy output, observing bursts of energy under specific conditions of prolonged electrochemical polarization. Notably, some of their experiments claimed heat outputs several times larger than the energy input, which they hypothesized as evidence of a nuclear reaction pathway distinct from traditional fusion processes involving neutron emission and tritium production.

Although the initial announcement generated significant excitement, subsequent replication efforts by other laboratories often failed, leading to skepticism about the validity of their findings [233]. Fleischmann and Pons argued that subtle experimental variables – such as electrode preparation, electrolyte purity, and precise control over electrolysis parameters – were critical to producing excess heat. They noted that only after prolonged polarization periods and substantial deuterium loading into the palladium lattice did excess heat manifest, suggesting that high deuterium-to-palladium loading ratios, often exceeding 0.85, were essential [234, 235]. However, their limited reporting on critical preparation steps, such as heat treatments and cleaning of palladium electrodes, further complicated replication efforts.

The original experiments used relatively large palladium samples and high current densities, often exceeding 500 mA/cm^2 , which presented several challenges for calorimetry. Their system consisted of a Pd cathode in sheet, rod, or cube form factor in 0.1 M LiOD in 99.5% D_2O + 0.5% H_2O solutions with a Pd-D reference electrode loaded to the α - β -phase equilibrium. A Pd sheet cathode (2 mm x 8 cm x 8 cm) surrounded by a large Pt sheet counter electrode, or alternatively a Pd rod (1, 2 and 4 mm diameter x 10 cm long) surrounded by a Pt wire anode wound on a cage of glass rods, was used in the low (1.6 mA/cm^2) and high (8 to 512 mA/cm^2) current density experiments, respectively. Fleischmann and Pons employed thermodynamically open Dewar-type calorimeters, which allowed for the electrolyte to evaporate during electrolysis. However, this evaporation altered the thermal environment, complicating heat measurements. In isoperibolic calorimetry, where temperature changes are referenced to a stable environment, any shifts in the electrolyte level could disrupt heat accounting. To mitigate these issues, they sealed the experimental

setups with Parafilm and periodically replenished the electrolyte, but these efforts highlighted the difficulty of maintaining precise thermal conditions under open-system operation.

Other concerns levied against these inaugural LENR experiments include over-interpretation of their data. Critics argued that the bursts of excess heat reported could be explained by classical chemical processes. Specifically, as the electrolyte level dropped and exposed the electrodes, recombination of oxygen and deuterium gas could result in increased temperatures which would be interpreted as excess heat.

7.1.1 Our Replication Efforts

While the scientific community largely moved away from cold fusion research due to challenges in reproducibility, the work of Fleischmann and Pons continues to influence the field of LENR. Any serious effort to determine the physical reality of LENR must revisit the Fleischmann and Pons experiments and evaluate the critical parameters identified in their work. Over the course of our replication attempts, we explored multiple strategies to maintain stable thermal environments and tested several modifications to the original setup. In addition to designing stable thermal enclosures, we implemented two different types of electrolyte refill mechanisms including a gravity-fed water leveler and syringe pump dosing. In total, we performed more than 40 formal and modified replications of the Fleischmann and Pons experiments. Despite better control over electrolyte levels and calorimeter thermal stability, no excess heat was observed. These experiments fall into the following groupings:

7.1.2 Nominal Replications

For these experiments, we applied high currents (>500 mA) to large palladium samples (>10 g) for various durations. These tests were done in open beakers with 35 ml of 1 M LiOD electrolyte, with the heat being measured in an isoperibolic calorimeter with an RTD temperature sensor placed in the electrolyte near the cathode. Additional RTDs were placed in the water bath surrounding the electrolytic cell and in the thermal enclosure. Control experiments were performed with light water. Some experiments lasted less than an hour, while others ran for over 100 hours with the aid of electrolyte replenishment systems. Despite consistent application of current, we never observed any thermal signatures or temperature responses indicative of excess heat. Calibration and control experiments that were performed had temperature profiles similar to the ‘active’ samples. We did, however, note an increase in input power over time at constant current. We hypothesize this effect was likely due to the reaction of atmospheric carbon dioxide with lithium hydroxide in the electrolyte, forming lithium carbonate, a non-conductive, insoluble salt, a reaction which is well documented in literature [236]. As the salt precipitated, the electrolyte’s conductivity decreased, causing the cell voltage to rise when operating at constant current. An example of a long-duration test is shown in figure 7.1. From the figure, we observed an initial input power of 2 W, which increased to 3 W over ~125 hours. Periodic fluctuations in temperature correlated with the syringe pump delivery of heavy water to maintain the electrolyte volume.

7.1.3 Modified with Additives

We hypothesized that the samples may have been prepared in a manner which influenced the replication. Given the large sample sizes, we speculated that they may have been machined, and poorly cleaned prior to use. We identified cutting fluids that were used in the late 1980s and early 1990s. We both soaked the samples and dosed the electrolytes with Rapid Tap cutting fluid. However, no excess heat was ever observed.

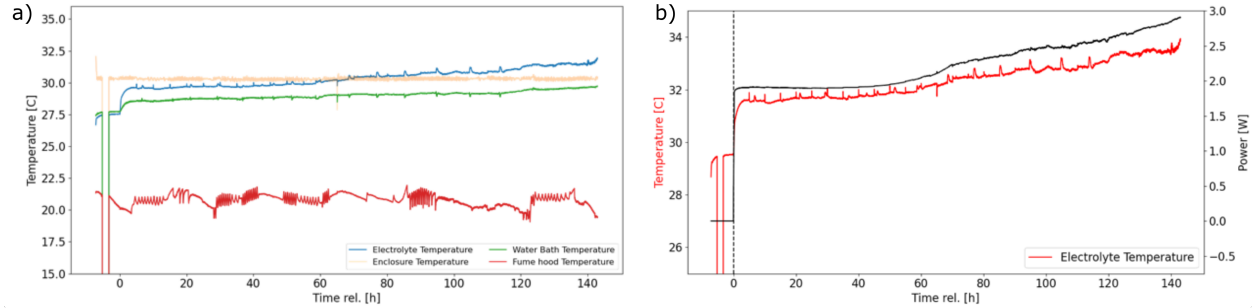


Fig. 7.1: Example of temperature (a) and input power (b, right axis) versus time for a long-duration test.

While nothing noteworthy came from these experiments, an example of the temperature and power profile versus time is shown in figure 7.2.

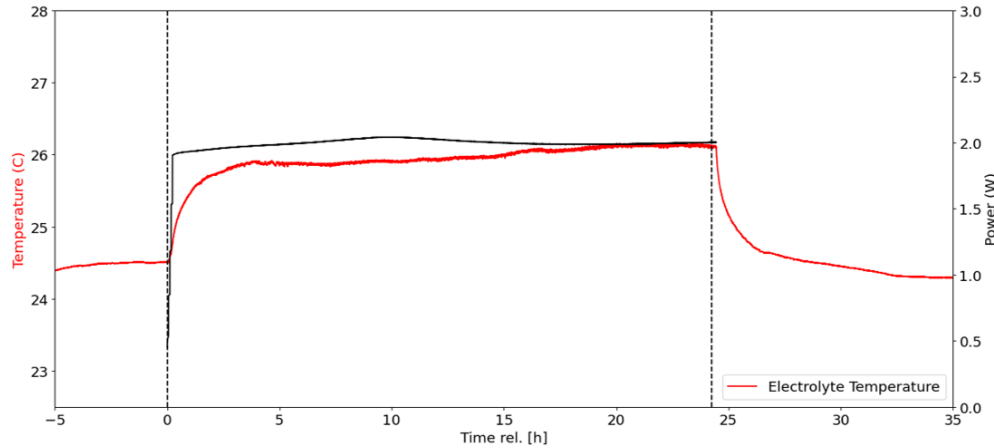


Fig. 7.2: Temperature and power profile for tests with cutting fluid additive.

7.1.4 Modified with Oxygen

We explored the possibility that orphaned oxygen produced during deuterium loading could have contributed to the reported anomalies. To test this, we bubbled gaseous oxygen directly beneath the cathode during electrolysis. While the introduction of oxygen initially caused an increase in current noise and a small temperature rise (several hundred millikelvin), these effects quickly subsided. We attributed this behavior to increased convective heat transfer, which facilitated more efficient heat transfer from the electrodes to the RTDs (this convection effect is commonly observed where bubbling is present in electrochemical cells), rather than any nuclear phenomenon. An example of this temperature rise is shown in figure 7.3.

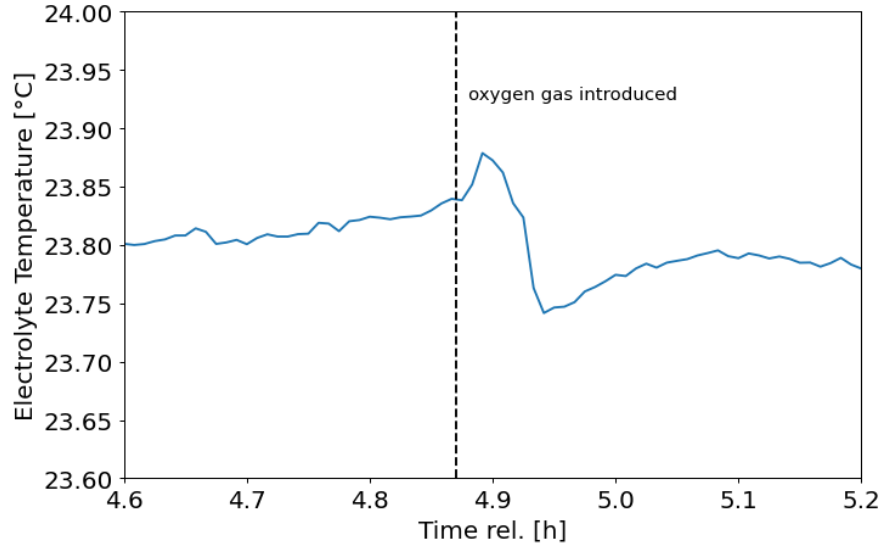


Fig. 7.3: Temperature transient observed during tests with oxygen bubbling beneath cathode.

7.1.5 Modified with High Power

In a separate set of experiments, we deviated from the original design by applying high input power between 100 W and several kilowatts (up to 15 A, 300 V) for short durations. This caused rapid heating of the electrolyte to boiling. At boiling, we observed a striking effect: the electrode emitted light and sparked sporadically. In some cases, when the electrolyte level dropped, the electrode glowed red hot. However, we concluded that this effect was classical in nature for two reasons:

- The phenomenon occurred in light water with protonated salts, which are thought incompatible with LENR, or in any event would not be expected to behave similarly to the deuterated systems if nuclear processes were responsible for the effect.
- The effect was replicated with stainless steel, nickel, and titanium cathodes, which are not generally associated with LENR.

Once convinced that the effect was non-nuclear, we further explored the underlying mechanism. We hypothesized that the observed light emission resulted from a liquid plasma discharge. As gas bubbles formed during boiling, they created conditions conducive to ionization. Using a photospectrometer, we identified sharp emission lines corresponding to excited Pd, Li, and OH species. All four cathodes show sharp Li emission near 670 nm, metal specific emission between 325 and 550 nm, and UV emission between 250 and 350 nm. These findings aligned with classical physics, confirming that the phenomenon was unrelated to LENR. A photograph of the effect and optical emission spectra are shown in figure 7.4. When the electrode becomes exposed such as in figure 7.4 c), the cathode began to glow red hot, likely due to insufficient cooling from the surrounding fluid.

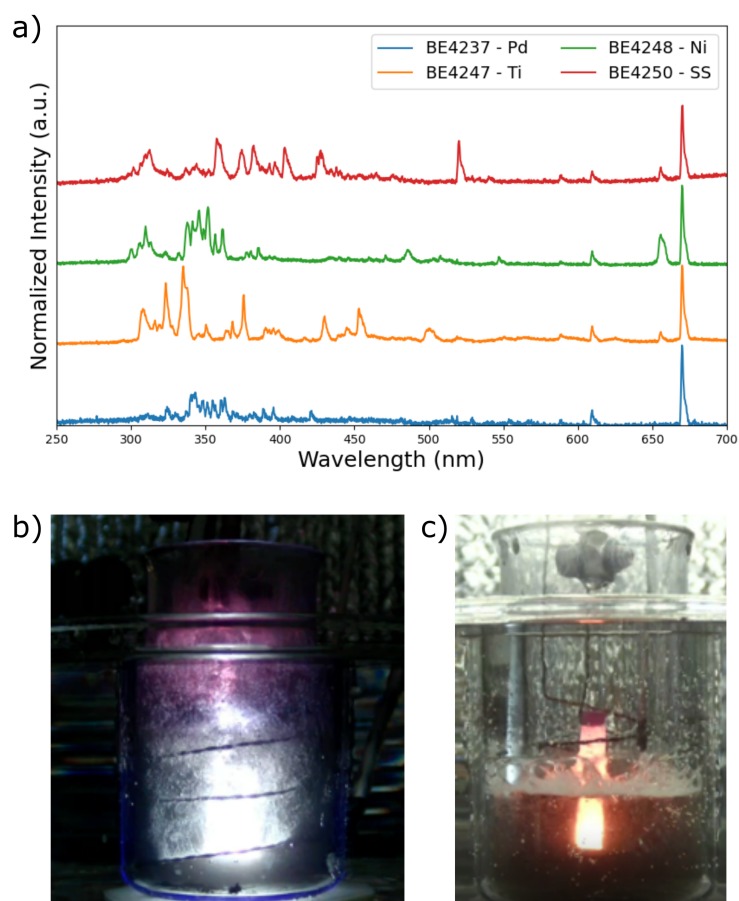


Fig. 7.4: High power experiments: a) Optical emission spectra. b) Plasma discharge glow when electrode is submerged. c) Sustained glow when electrode is exposed.

7.1.6 Conclusion

At no point during our replication efforts of the original Fleischmann and Pons experiments did we find compelling evidence for LENR. Whether this is due to insufficient details provided in the original studies or because the effect itself is not grounded in physical reality remains technically inconclusive. With the very limited data provided in Fleischmann's paper [26] – notably absent of any electrochemical or calorimetry figures – it is impossible to uncover the root cause of the discrepancies between their experimental outcome and our experimental outcomes. Although scientific rigor allows only for proof of a phenomenon's existence, not disproof, our extensive testing – including hundreds of LENR experiments and more than 20 sample-years – strongly suggests that no LENR effect can be reliably observed under any reasonable and experimentally obtainable conditions. These Fleischmann and Pons replications make up a small portion of the conditions we tested; the majority of our experimental efforts are documented in subsequent sections.

7.2 McKubre

7.2.1 Introduction

Michael McKubre studied cold fusion in palladium hydrides from 1989 to the early 2000s [74]. He was a researcher at SRI International (Menlo Park, CA), with a major portion of his research funding coming from EPRI (Electric Power Research Institute, Palo Alto, CA). Contrary to most other researchers in the LENR field, his work was funded by mainstream funding sources and carried out at mainstream institutions, enabling him to perform scientific work to higher standards than most contemporaries. Overall, his work is well documented. Most details are provided in two EPRI Technical Reports from 1994 and 1998, each of them measuring several hundred pages [101, 110]. Contrary to other researchers in the LENR field, he took a systematic approach, studying loading enhancers to maximize electrochemical loading, designed various open and closed calorimeters, characterized his calorimeters, and also employed various techniques to characterize materials and processes, as is common in the mainstream materials research community (e.g., mass spectroscopy).

McKubre reported excess heat was repeatedly observed if the palladium hydride was highly loaded (>0.9), the electrochemical loading current was sufficient ($>100\text{ mA/cm}^2$) and an incubation time of one week had passed [101].

7.2.2 Experiments

Cathode: In most experiments, McKubre used 3 mm diameter palladium rods, 30 to 50 mm long. Palladium (99.9%) was supplied by Engelhard and machined to 3 mm diameter. After cleaning, they were annealed in vacuum for 2 hours at 850°C [110, p. 3-27] or 3 hours at 800°C [110, p. 3-8]. They note that other suppliers' palladium also worked well [101, p. 27].

Electrolytic Cell: In most experiments, a 0.5 mm diameter platinum wire (Engelhard) was helically wound around a cage made of silica rods and PTFE discs, surrounding the cathode (Fig. 7.5 a).

Electrolyte: 1 M LiOH or LiOD was used, obtained by dissolution of lithium (Aesar) in light water or heavy water [110].

Additives: McKubre added aluminum, silicon, boron or copper in concentrations of 200 ppm to many of his experiments, either in elemental or in oxide form [101, pp. vi, 2-2, 2-13]. He reported that silicon increases the loading and aluminum increases the longevity of loading [110, p. 3-48].

Electrochemical Conditions: Typical electrochemical current densities were 300 to 600 mA/cm², but occasionally up to 6.4 A/cm² [110, p.1-3]. Current densities below 200 mA/cm² were found to yield the highest loadings [101, p.2-2]. Sometimes, lower currents (10 mA/cm²) were used [110, p.3-8] and various types of current ramps were applied [110, pp. 2-15, 3A-5, 3A-8]. Typical durations were a few hundred hours, as excess heat was reported to occur no earlier than a week into the experiment [110]. Some experiments lasted thousands of hours.

Electromigration Conditions: The early studies did not contain electromigration / axial currents. In the later studies, axial currents of up to 7 A were applied [101, p.3-13].

Loading: McKubre reported that electrolytic deuterium loadings up to 0.9 and protium loadings up to 0.95 were achieved [110, p.2-5]. 61 % of their cells reached loadings > 90 % [101, p.2-1]. He performed loading optimization studies in open system electrolytic cells, with up to ten cells running in parallel [101, p.v] and a total of over 100 loading experiments [110, p.2-18]. Loading was inferred from measuring the resistance ratio, using a four-terminal AC measurement [110, p.3-9]. It is not clear if the resistance was renormalized when several load-de-load cycles were applied. Additives studied as potential loading enhancers included As₂O₃, sulfides, iodides, aluminum, zinc, bismuth, mercury and silica [110, p.2-18]. As noted above, silicates were found to enhance loading [101, p.2-12]. This could explain the observed higher loadings in quartz vessels compared to PTFE [101, p.2-12]. Furthermore, pre-annealing of palladium at 850 °C for 3 h was found to increase loading [101, p.2-2]. Contrary to Callisto's loading studies, McKubre found that arsenic(III) yielded very slow loading rates and no elevated loading [110, p.2-24]. In agreement with other researchers [105], McKubre noted that slow loading can minimize crack formation and elevate loading [110, p.2-16]. He also postulated that platinum may serve as loading inhibitor [110, p.2-16] and that uniform electric fields increase the loading [101, p.2-11].

Calorimetry: At the center of McKubre's studies were closed system mass flow calorimeters [110, p.iii]. The electrochemical cell was surrounded by heat transfer fluid (silicone oil), which was pumped at a constant rate and whose inlet as well as outlet temperatures were measured (Fig. 7.5 b) [110, p.3-14]. The accuracy was specified as the greater of 50 mW or 0.5 % and the precision was the greater of 10 mW or 0.1 % [110, p.1-3]. They also used other calorimeters in addition to mass flow calorimeters [101, p.1-2], e.g., a Seebeck calorimeter [101, p.3-3]. They used a compensation heater to correct for changes in electrochemical power so that the sum of the heater and electrochemical power input to the calorimeter was held constant, so the system remains isothermal regardless of EC power [110, p.1-4]. COPs were typically reported with respect to the electrochemical input power only, so skepticism of the quoted COPs is warranted, as the total input power was up to 45 W regardless of EC power [110, pp.1-3, 3-55], and a small percentage error in calorimetry could nullify the claimed excess heat.

7.2.3 Results and Assessment

In his 1994 report, the reported excess heat was up to 1.1 MJ and COPs were up to 3.5 [110]. Fig. 7.5c) shows a plot of excess powers compiled from Table 3-1 [110, p.3-3]. The table shows that the COP of 340 % (in P19) corresponded to an excess power of 850 mW on top of an EC input power of 250 mW. The total input power (including the heater) was presumably 12 W. However, there were several hardware failures, some close to the reported excess heat events [110, p.3A-63]. The axial current was 2 A. While no problems with the axial current in this experiment were reported, McKubre notes in a later experiment "During times when axial current is passing both loading information and calorimetry were seriously inaccurate: Calorimetry was poor because the axial input power was not measured at the calorimeter boundary." [101, p.3-11] In our own research we have encountered inaccurate calorimetry when high axial currents were applied due to the

relatively large voltage loss (and thus power dissipation) in the lead wires compared to the sample. Even when the voltage is measured at the thermal system boundary, the location of heat dissipation is different from the location of electrochemical input power dissipation and can thus affect calorimetry. Calibration of these effects is further complicated by the fact that the sample resistance and thus the relative contribution of heat dissipation change as a function of loading.

While the claimed COP amounts to 350 % (3.5) compared to the EC input power, it is much less compared to the overall input power, when including the heater. McKubre specifies that the calorimeter has an accuracy of 0.5 % [110, p. 1-3], but the noise in the heat flow measurement is already 0.4 % [110, p. 3A-6]. This leaves very little error to input power measurement errors, heat source location dependent errors (heater vs. EC power vs. EM power) and errors due to pressure variations / recombination fluctuations in the headspace. We posit that a conservative estimate would raise the uncertainty of the excess power substantially. The fact that the noise in the heat flow is much larger than the uncertainty, exemplifies that the uncertainty was underestimated by McKubre [110, p. 3-24].

Other excess heat events reported by McKubre were well below 10 %, and could be due to the errors listed above. However, in contrast to P19, very limited data is available for those experiments.

The 1998 report fails to support or extend the claims made in the 1994 report [101, 110]. 30 isoperibolic calorimetry experiments and 24 mass flow calorimetry experiments were conducted, but only two of them showed excess heat. In experiment M1, the maximum excess power observed was 100 ± 40 mW or 0.6 % of the total input power. The integrated energy was 27 ± 12 kJ. This is close to the noise of the calorimeter. In experiment M4, two periods of excess heat, each about a day long, were observed. The maximum excess power was ~ 400 mW at 12 W input power, which corresponds to 3.3 %. While this is low, it should be above the noise of the calorimeter. However, during the same period, the sample resistance exhibits a much higher noise than usual. This could be due to a bad contact, resulting in imperfect input power measurements or increased input power from the resistance measurement. Notably, when the resistance measurement noise subsides, the excess heat subsides.

The absence of strong evidence of excess heat after several years of research and numerous experiments with various calorimeters leads McKubre to the conclusion “With hindsight, we may now conclude that the presumption of repeatable excess heat production was premature, and that this has limited the progress achieved in this study.” [101, p. 4-3]. Curiously, despite this, McKubre is regarded as a major scientific figure in the LENR field and as a proponent of the anomalous heat effect. Moreover, the frequently cited “conditions [...] characteristic of all cells yielding episodes of excess heat” [101, p. vi] (incubation time, loading >90 %, current density > 0.1 A/cm²) were based only on those two above mentioned experiments.

7.2.4 Replication Experiments

McKubre’s main achievement was to pose a list of conditions that are allegedly required, though not sufficient, to generate excess heat. These conditions include high loading (>0.9), sufficient EC current (> 100 mA/cm²) and long incubation times (>1 week). He obtained the high loadings using additives. For that reason, we decided to focus our replication attempts on fulfilling these conditions and employing the additives he used. We did not attempt to replicate the flow calorimeters, as we had our own well-characterized closed system calorimeters available (section 2). Performing the replications inside the HTcal calorimeters necessitated scaling the sample down to a 0.5 mm diameter, 28 mm long wire and adapting the currents in order to obtain comparable current densities. We added boron, silicon, aluminum, copper and silica at a concentration of 200 ppm (by mass) to the electrolyte (one additive per experiment). However, we did not find a noticeable increase in loading with any of these additives (Fig. 7.6). Each additive was used in three repeat cells

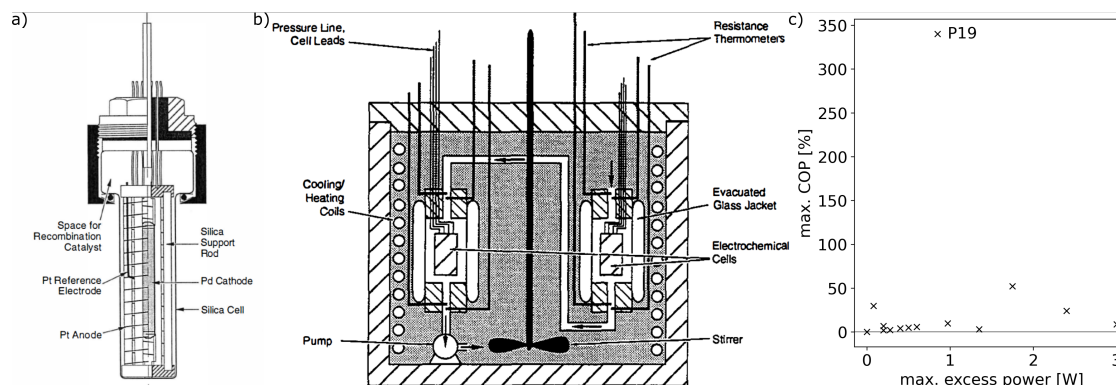


Fig. 7.5: a) Electrochemical cell for Pd resistance measurements [110, p. 3-25]. b) Schematic diagram of *differential calorimeter*. Note that McKubre used only one cell [110, p. 3-14]. c) Maximum COP (relative to EC power, disregarding heater power) and maximum excess power of 14 experiments. Plot generated by Callisto based on data listed in Table 3-1 [110, p. 3-3].

(except six for silica, two for silicon) and one reference cell (with a Pt cathode). EC was applied in power control mode at levels of $[-0.2, -0.5, -1.0, -2.0]$ W for 10 h each with 10 h ramps to increase power (similar to McKubre). These power levels correspond to roughly $[-0.1, -0.25, -0.5, -0.8]$ A/cm². No axial currents were applied (except for small currents to measure the loading). The total experiment durations were several hundred hours (similar to McKubre). Note that the spikes in COP are caused by the changes in input power and cancel each other out, as evidenced in the smooth COP_e plot. (See section 2.5 for definition of COP_e.) The constant offset in some of the COPs can be attributed to drift in the calorimeter calibration. Some experiments stopped prematurely due to recombiner failure, causing pressure buildup and ultimately causing the system to open. We did not observe any measurable excess heat (Fig. 7.7).

7.2.5 Conclusion

We could not replicate the excess power reported by McKubre in his 1994 report [110]. First, we would like to note that even his ‘most spectacular’ excess heat was less than 10 % of the total input power (including the heater input power). In his later report [101], after four years of research, the largest reported excess heat was even lower, at 3 %. Luckily, McKubre provided a wealth of data in his reports. The datasets for both these experiments show hardware malfunction during or near the reported excess heat period. McKubre admits that during application of electromigration currents, the calorimetry was “seriously inaccurate”. This applies to the first experiment with the ‘most spectacular’ excess heat. As noted above, during the second experiment (with 3 % excess heat), the loading measurement malfunctioned. We have observed various mechanisms which could cause false excess heat signals of similar magnitude (section 9.2). We therefore

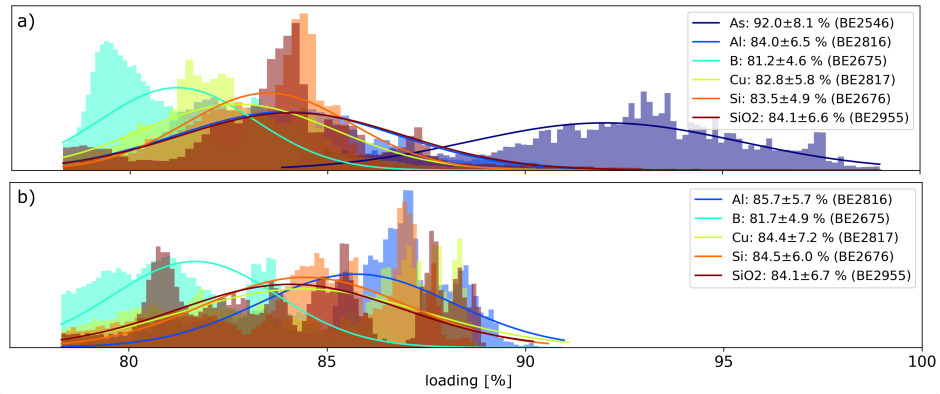


Fig. 7.6: Effect of additives on loading in heavy water. Arsenic was added in the form of 25 mM NaAsO₂ in 0.5 M LiOD electrolyte. All other additives were added at a concentration of 200 ppm by mass in 1 M LiOD electrolyte. a) EC current -100 mA/cm², b) EC current -300 mA/cm².

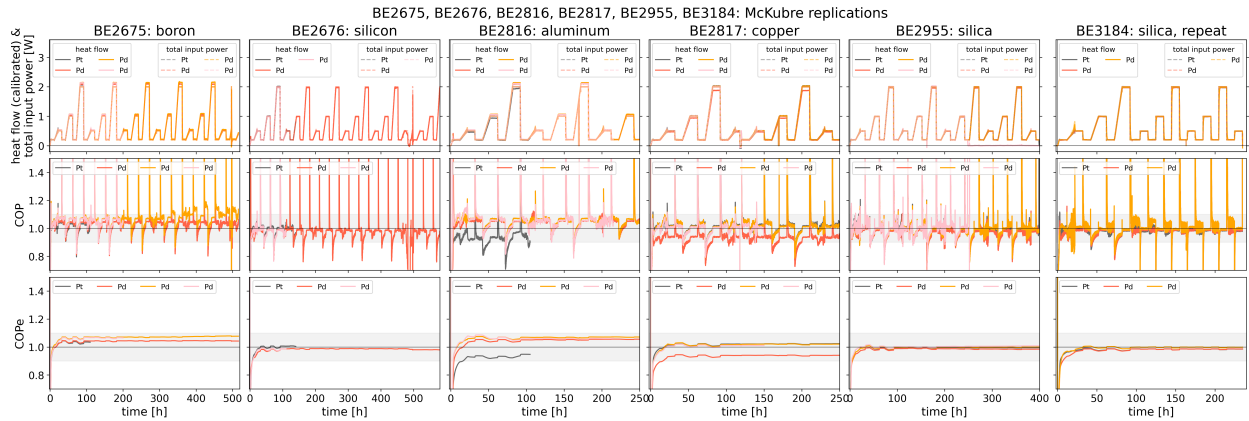


Fig. 7.7: Overview of McKubre replication attempts performed by Callisto, using various additives.

concur with McKubre that “With hindsight, we may now conclude that the presumption of repeatable excess heat production was premature” [101, p. 4-3].

7.3 Staker

7.3.1 Literature Review

Michael Staker published several works [237–239] claiming the successful observation of excess heat, similar to the original Fleischmann-Pons experiment that reported excess heat output compared to the expected heat from input power [240]. In his 2019 work ‘Coupled Calorimetry and Resistivity Measurements, in Conjunction with an Emended and More Complete Phase Diagram of the Palladium-Isotopic Hydrogen System’, Staker reports 100 % excess power (2.4 W of output with 1.2 W of input) after rapidly increasing the input current in two separate instances [237]. He also reports that aside from these two ‘run away’ events, that excess energy out of the system was 150 MJ/cm³ of Pd for 46 days. The author cites the presence of three new, thermodynamically stable phases marked by super-abundant vacancies (SAV) as key factors leading to this excess heat event. Staker postulates that these SAV-phases (γ (Pd₇ VacD₆₋₈), δ (Pd₃VacD₄–octahedral), δ' (Pd₃VacD₄ – tetrahedral)) should be added to the Pd-H phase diagram to properly represent structural changes as a result of H (D) loading $\geq 70\%$ in the Pd structure. In this review we use ‘H’ to represent all of the isotopes of hydrogen and unless explicitly called out, all loadings refer to D. It was theorized that once enough of these SAV-phases were formed (specifically the δ and δ' phases) they form a lattice arrangement of tubes that allowed ultra-high mobility of the intercalated H⁺ ions. It is important to note that the author has presented no experimental evidence for these claims and are all based on his own theory.

Samples used in this experiment were unalloyed 0.5 mm diameter, 25.4 mm long Pd wires from Johnson-Matthey with Cu leads that were braze-bonded around the end of the Pd specimen and connected to a current-controlled DC power supply. The electrolyte was 18.0 ml of 0.5 mol LiOD in 99.8 % deuterium oxide from Cambridge Isotope Laboratory. The cells were constructed by nesting four slightly different size Pyrex test tubes, each separated by two O-rings and a thin air space. A Teflon top, sealed with an O-ring, excluded ambient atmosphere by allowing the positive pressure of O₂ and D₂ gas to exit through a capillary tube into a reservoir of vacuum pump oil. Electrolyte level in cells was maintained using a Harvard Apparatus Model 22 Digital Syringe Pump outfitted with two syringes, one with heavy water from Cambridge Isotope with 99.8 % D and one with double distilled light water. The input current for the experiments ranged from 442 to 535 mA. During these current applications, 10 to 18 °C temperature increases were observed when compared to the control cells [237].

Staker proposes that an anomalous heat event (AHE) occurs only after the formation of SAV-phases, Pd samples are highly loaded (H/Pd >1.14 according to his proposed phase diagram), and H⁺ ions occupy the octahedral sites in the Pd-H lattice. Change in resistivity is used as a measure of H loading in Pd as well as phase changes. The combination of excess heat being accompanied by a decrease in resistance, instead of the expected increase due to their directly proportional relationship, is used as evidence that both the Pd-H δ – octahedral and δ' – tetrahedral phases were formed (change in resistance and occupation by H ions) and δ – octahedral was occupied by H ions (yielding the production of excess heat).

In later works in 2023, Staker reports similar excess heat and power between 0.01 - 1.25 W but quenches the reaction in an ice bath [238, 241]. After equilibration, the reaction does not return to its previously elevated temperature and by extension does not exhibit any excess power or heat. The author uses this lack of continuation of the purported LENR event after returning to room temperature post-quench as evidence

supporting his theory of the intercalated H^+ ions occupying the Pd octahedral interstitial site (δ) instead of the tetrahedral site (δ') during AHE. He states that upon the quench, highly mobile H^+ ions hopped from the octahedral site to the δ' – tetrahedral site which is reportedly more stable at lower temperatures. It is noteworthy that the author published a corrigendum to this work in 2024 stating that the value of the highest excess power density was 361 W/cm^3 , not 240 W/cm^3 , but the effective COP was significantly reduced from 2.0 (1.2 W input, 2.4 W output) to 1.67 (2.66 W input, 4.44 W output) [241].

In this work we assess the claims of AHE made by Staker through our own replication experiments. Additionally, we present a brief literature analysis of Stakers’s published works addressing interesting points made, questions that arose, and discrepancies or missing data that hamper the work’s scientific value.

7.3.2 Literature Analysis

Despite some interesting theories Staker presents in these works, there are many shortcomings in his studies that must be addressed for the good of the scientific community, specifically those in the LENR field interested in replicating these works. Chief among the areas of weakness in these publications is the lack of relevant, clear data supporting the claims of AHE. In the original 2019 publication [237], several key figures (i.e. calibrations and power plots) had hard to decipher data and unlabeled axes. All figures with time as the x-axis value were hard to decipher as the unit of time was ‘data taken every 15 minutes’. Even after calculating several scenarios using the given time values, none of the time data aligned to total the reported 46 days of experiment duration. This leaves the reader and anyone trying to replicate the work to guess the incubation period before these ‘runaway’ events occur, as an incubation period has been theorized to be vital to AHE events [240]. A major shortcoming of the main figure is that the excess heat plots presented displayed very different heats for similar input powers. The author also uses a decrease in resistance as an indicator of both H/Pd loading and phase changes to the SAV-phases; however, there is no precedent for correlating a change in resistance to the formation of these phases. This is especially true considering that all references used validating the formation of SAV-phases were carried out under high-pressure and high temperature conditions (e.g. up to 6 GPa and 900°C) [242–244].

In Staker’s 2023 work ‘The role of superabundant vacancy delta to delta prime transformation in stopping Fleischmann-Pons heat effect in palladium-deuterium’ no new or relevant data was presented outside of a plot (figure 4 in [238]) illustrating deuteron oscillation in the Pd lattice [238]. Neither how these calculations were done nor the nature of the sample used are discussed in this work. This work also fails to present any resistance, temperature, XRD, or other relevant electrochemical and structural data to support the claims of AHE or crystal structure changes as a function of H^+ ion location in the Pd lattice. As an electrochemical paper claiming a phenomenon that releases excess power out compared to power in, the omission of any electrochemical data from the entire work is a cause for concern to any reader. The failure to produce these experimentally relevant pieces of data severely weaken the author’s claims on the mechanism by which the purported excess heat event occurs and the effect lowering temperature has to stop it (i.e., H^+ transition from the octahedral site to the tetrahedral site at lower temperatures). Inclusion of a plot showing Pd resistance, H/Pd loading, and electrolyte and ambient temperature as a function of time would benefit this work immensely. Another weakness of this work is the author’s continual self-citing of previous works [237, 239] to support his claims in this work, especially as it relates to the existence of SAV-phases at room temperature and their role in AHE. Finally, considering that this work also had a significant corrigendum amending the original excess power claim (which was never presented in the original work), it calls the validity of the work as a whole into question.

7.3.3 Replication Experiments

In our studies we carried out multiple replications, both exact replications of Stakers original thermodynamically-open calorimeter (open-system) and also ‘Staker-like’ replications performed in thermodynamically-closed high throughput calorimeters (closed-system).

7.3.3.1 Experimental Setup: Open-System Calorimetry

Our open-system was isoperibolic, whereas the closed-system had an independent heat flow measurement through a thermal bar (for details see section 2.9.1). Our open-system set-up is shown in Figure 7.8. This isoperibolic calorimeter has an RTD close to the electrodes for temperature monitoring. The cell consists of a test tube nested inside another test tube, similar to the original Staker experiment. Temperature is measured using RTDs connected to a NI cDAQ. Power was supplied using Keithley supplies and independent voltage measurement used Keysight 34661A DMMs. We used 30 mm length, 0.5 mm diameter Pd wire from various wire manufacturers (described in the list below) and Pt counter electrodes wrapped around the sample retention. Resistance was monitored using 2-wire and 4-wire NI square wave measurements for the open- and closed-cell testing, respectively. These samples were exposed to a gentle (≤ 50 mA/cm²) 3-cycle loading-unloading pre-treatment in either 18 or 44 mL of 0.5 M LiOH depending on open or closed cell testing. The temperature of the enclosure was maintained at ~ 30 °C. Temperatures inside the cell were measured using a NI cDAQ with Pt100 RTDs. A typical experiment’s duration was one week and hundreds of days (300+) for open-system and closed-system tests respectively. Over the course of these studies we measured the heat of Pd and Pt cathodes exposed to electromigration (EM) current and control cells exposed to standard electrochemical (EC) current. We also carried out post-mortem spectroscopic analysis (i.e., scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS)) to observe macroscopic structural changes and surface chemical composition. We did not investigate Staker’s claims of the existence of SAV-phases and their purported necessity in LENR reactions in this work.

In our open-system testing we used conditions matching the details reported by Staker as closely as possible in addition to many tests with variations for completeness. These conditions are described in the list below:

- Pd Vendor: Johnson Matthey, Goodfellow, Sigma Aldrich
- Pd Form Factor: 0.5 mm diameter wire, 0.5 mm thick foil
- Electromigration Field: 0 to 35 mV/cm (~ 15 to 35 mV/cm)
- Lithium Isotope: ⁶LiOD (95 % ⁶Li), ⁷LiOD (92.5 % ⁷Li)
- EC and EM Inputs: EC up to 100 μ Hz – 100 Hz, DC ~ 95 % DC, and AC EM (up to 1 kHz)
- Pd Pre-treatment: Annealing, w/ and w/o load-unload cycling before ‘active’ cycle, aqua regia etch
- Vessel Material: Pyrex (borosilicate) glass, Perfluoroalkoxy alkane (PFA), Quartz

7.3.3.2 Experimental Setup: Closed-System Calorimetry

The closed-cell experiments were carried out in similar conditions with small variations such as: 4-wire resistance measurements, 1.8 to 2.4 W active cycle, and EM constant power 0.01 W (pre-cycle) and 0.1 W (active cycle). Regular duration experiments were performed over 80 to 90 days and ultra-long duration

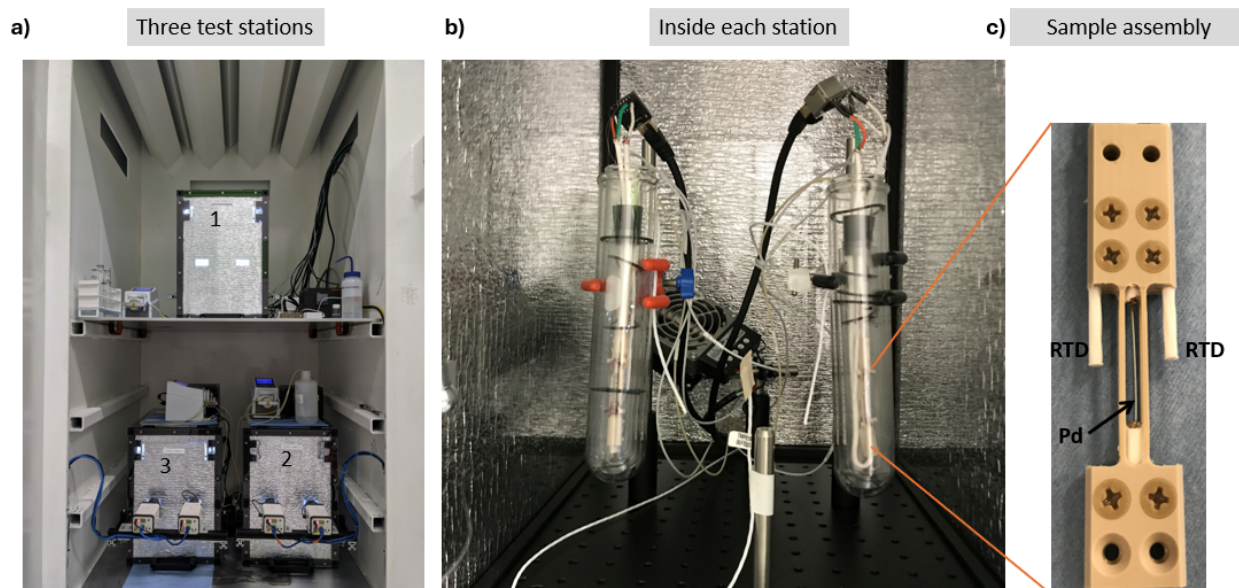


Fig. 7.8: Image of our Staker replication apparatus. The thermal enclosures with connections for the resistance temperature detectors (RTDs) and electrical sensors.(a) A visual depiction of our cells containing Pd cathode, Pt anode, 2 RTDs, electrochemical current controlled make-up water inlet, and 4-wire resistance ratio measurement on cathode. (b) A representative image of the sample assembly with the Pd cathode and RTDs visible. Pt counter electrode and 4-wire resistance sense leads not shown.(c)

experiments were carried out over 250 days. In one of the ultra-long duration experiments, the temperature (of the enclosure and the cold plate) was increased from 30 °C to 40 °C after ~5150 h in order to achieve an electrolyte temperature that is closer to the electrolyte temperatures recorded by Staker [93].

7.3.3.3 Results

Figure 7.9 summarizes the COP (previously described) results for both long duration open-cell (Fig. 7.9a) and closed-cell calorimetry experiments (Fig. 7.9b). As shown in Fig. 7.9a) we include an upper bound of the COP’s error resulting from foaming and gradual electrolyte level change. We corroborated this by including cameras in our open-cell systems which recorded the samples at all times. In our studies we found that foaming and electrolyte level change was almost always accompanied by a large heat flow effect that could be interpreted as excess heat. It is suspected that this combination of changes to the electrolyte eventually exposes ~30 to 40 % of the cathode to the headspace and foam, and is responsible for the spikes in COP. The several green traces depicting the long-duration tests exhibit this behavior, defining the upper error bounds near a COP of 1.6. Our direct replications of Staker’s conditions (Figure 7.9a, blue trace) did not show any elevated heat that could not be explained by these effects. Our longest direct replication of Staker’s work, surpassing 46 days (the duration of Staker’s purportedly successful experiment showing large excess heat) is shown in red, and does not indicate any excess heat. From the onset of the experiment we actually observed a suppressed COP before returning to baseline briefly at 600 h and then being suppressed until equilibration at ~1000 h. We believe the reason for this suppression is likely changes in the electrolyte level.

Notwithstanding our attempts to replicate the original Staker experiments as closely as possible, it is clear that many improvements can be made by transitioning to closed-cell calorimetry testing. These improvements include: elimination of COP error due to electrolyte level changes, lower calorimetry errors due to foaming, PEEK vessel liners enabling longer duration experiments compared to borosilicate vessels, constant power EM in order to eliminate transient input power signals compared to Staker’s current controlled EM, and the use of integrated recombiners such that the heat of recombination of H₂–O₂ remains in the thermal envelope of the vessel. We have observed the formation of an overlayer – coined ‘Coating X’ – on the Pd cathode surface typically comprised of Cu, Fe, Zn and other elements in varying concentrations depending on the electrochemical environment. We theorize that this overlayer acts a ‘Tafel poison’, preventing the deloading process via the Tafel pathway and leading to higher loadings. It is of note that Staker does not discuss any surface depositions on his samples.

Figure 7.9b shows a summary of our results from a three-month long closed-system Staker-like experiment without interruption, with Pd cathodes under various stimulation conditions and one Pt control. All cell COPs remain near unity for the majority of the testing period, outside of some suppressed heat in Cell 2 starting at ~83 days (which can occur when a closed cell becomes open following over-pressure activation of a protective check valve). The spikes in COP observed are transient effects of input power changes.

The results of the ultra-long duration experiments are shown in Fig. 7.10. The full dataset is available in the Appendix (section A.1). The EC power was cycled between [0.8, 2.4, 2.0] W (loading polarity) every 10 h, but some cells hit the compliance voltage of the SMU, thus limiting the current. The increased cell resistance was presumably due to buildup of coating on the electrode and due to loss of electrolyte conductivity as salt deposits formed in the headspace. The spike in excess heat at 6400 h is an artifact due to a server outage lasting a few hours. Due to faulty communication between the NI SMU and the gRPC server, no data was recorded (interpreted as 0 power), but the SMU kept on applying the most recent set current before the outage occurred. The electromigration current failed to be applied in the platinum control cell (cell0) after ~900 h, presumably due to a bad contact. Non-steady state recombination (possibly due to foaming or salt

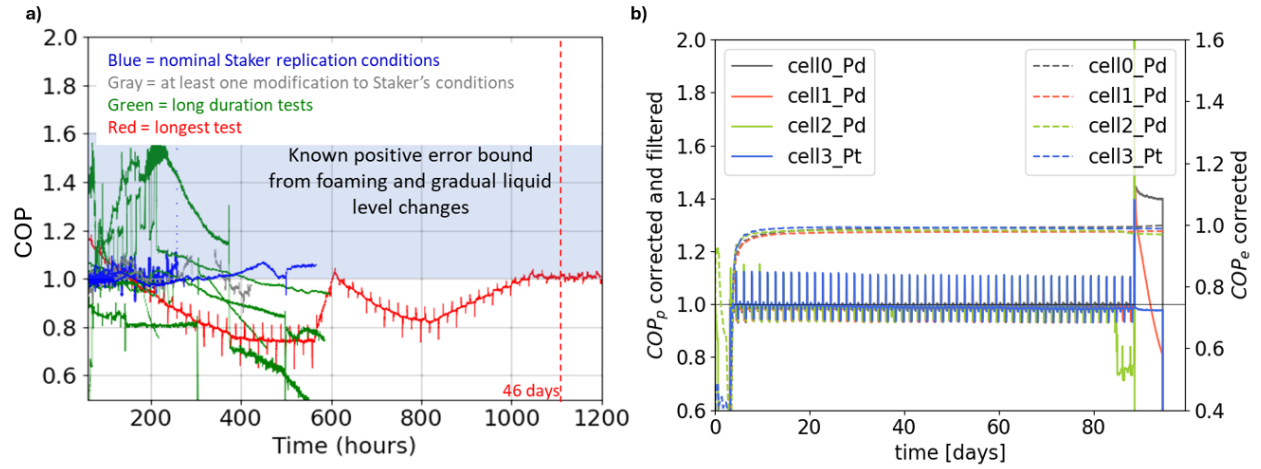


Fig. 7.9: Summary figures of thermodynamically-open calorimetry (a) and thermodynamically-closed calorimetry (b) Staker and Staker-like replications. Figures show the reported COP as a function of time for various experimental conditions described in the key of each figure. In the thermodynamically-closed calorimetry the conditions of the cells are as follows: Cells 0 and 1: nominal Staker replication conditions (i.e., Pd with EM), Cell 2: Pd without EM, and Cell 3: Pt with EM. COP_p = COP power. COP_e = COP energy (integral of power out over integral of power in). The spikes in panel b) are due to changes in input power.

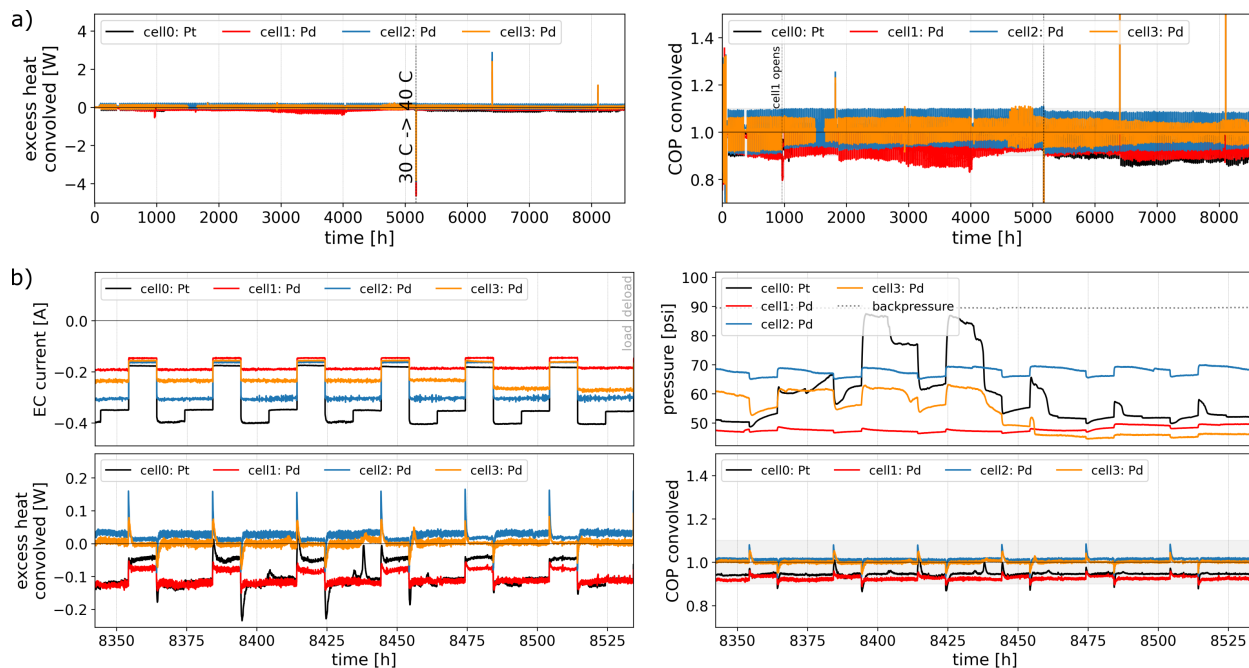


Fig. 7.10: Ultra-long duration Staker-like replication in an HTcal. a) Overview of excess heat and COP during the full experiment. b) Zoom into an arbitrary week towards the end of the ultra-long duration experiment.

buildup on the recombiner) led to build-up and release of pressure in the headspace, resulting in transient suppressions and peaks in heat flow due to the lack or excess of heat of recombination. This is particularly prevalent in the platinum control cell (cell0). Cell3 likely experienced a drift in calorimeter efficiency since the last calibration at the beginning of this experiment, leading to a suppressed heat flow. No unexplained excess heat signals were observed over the entire duration of the 250 day experiment.

We observed in a closed-system Staker-like replication (experiment BE1476 shown in Figure 7.9b and Figure 7.11) cells (Cells 0 and 1) that when EC stopped, the heatflows did not decay in the same manner, possibly due to different deloading kinetics. In our control cell with Pd that received no EM current (Cell 2) behavior was standard when EC was stopped, i.e., heatflow fell to 0 as expected. Similarly in our Pt control (Cell 3) when EC stopped, heat flow falls to the expected amount for the applied EM power. While no instances of AHE were observed over the nearly 90 day period of testing, we did observe nearly 30 excursions that have all been proven to be consistent with temporary recombiner failure. These recombination events highlight the importance of accurate calorimetry and energy accounting to avoid mis-attributing energy released from chemical events to small, localized LENR events. Additionally, the results show the benefit of closed-cell calorimetry especially relating to the quality of data that can be achieved in the absence of excessive foaming, electrolyte level change, chemical energy of oxyhydrogen leaving the vessel, and the other challenges observed in open cells.

Figure 7.11 depicts post-mortem SEM and EDS sample analysis for the long-duration Staker-like replication in the thermodynamically-closed calorimeter. The SEM images show that all cells had surface depo-

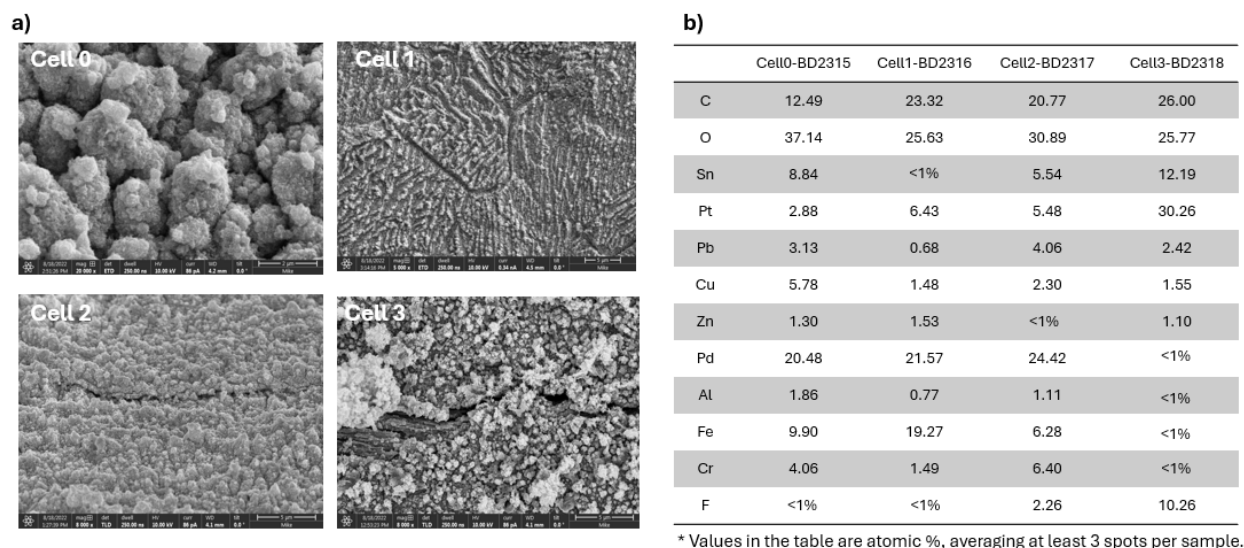


Fig. 7.11: Scanning electron microscopy (SEM) images of each cell after the long duration Staker-like replication in the thermodynamically-closed calorimeter (BE1476). Scale bar for Cell 0 is 2 μm and 5 μm for Cells 1-3.(a) Cells 0 and 1: nominal Staker replication conditions, Cell 2: Pd without EM, and Cell 3: Pt with EM. A table summarizing the average atomic percent (from three different sample locations) of elements found on the surface of each cell's cathode determined by energy dispersive X-ray spectroscopy (EDS).(b)

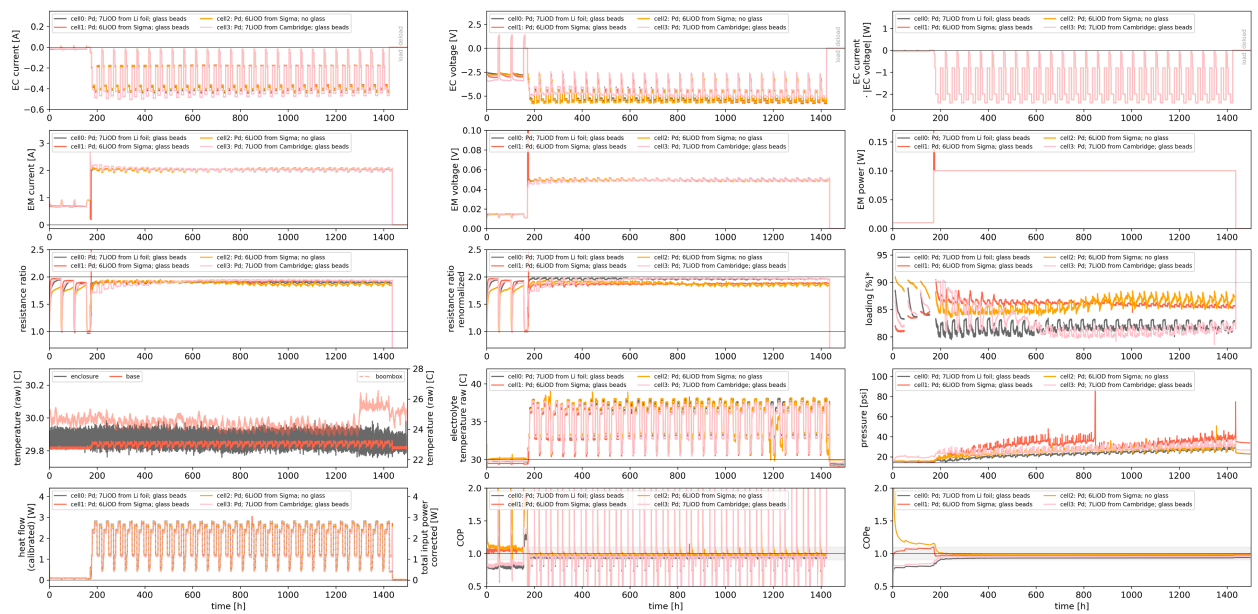


Fig. 7.12: Summary of all relevant experimental components of an approximately 60-day Staker-like experiment with glass bead additives. (From left to right) The first row shows the EC current, voltage, and power. The second row shows the EM current, voltage, and power. The third row shows the Pd resistance ratios, the resistance ratios renormalized to a baseline period with no input power, and the H loading percentage calculated from the Zhang formula. The fourth row shows temperature of system's enclosure, base plate, and external 'boombox', electrolyte temperature, and the pressure for each cell. The final row shows the calibrated heat flow, COP (power), and COP_e.

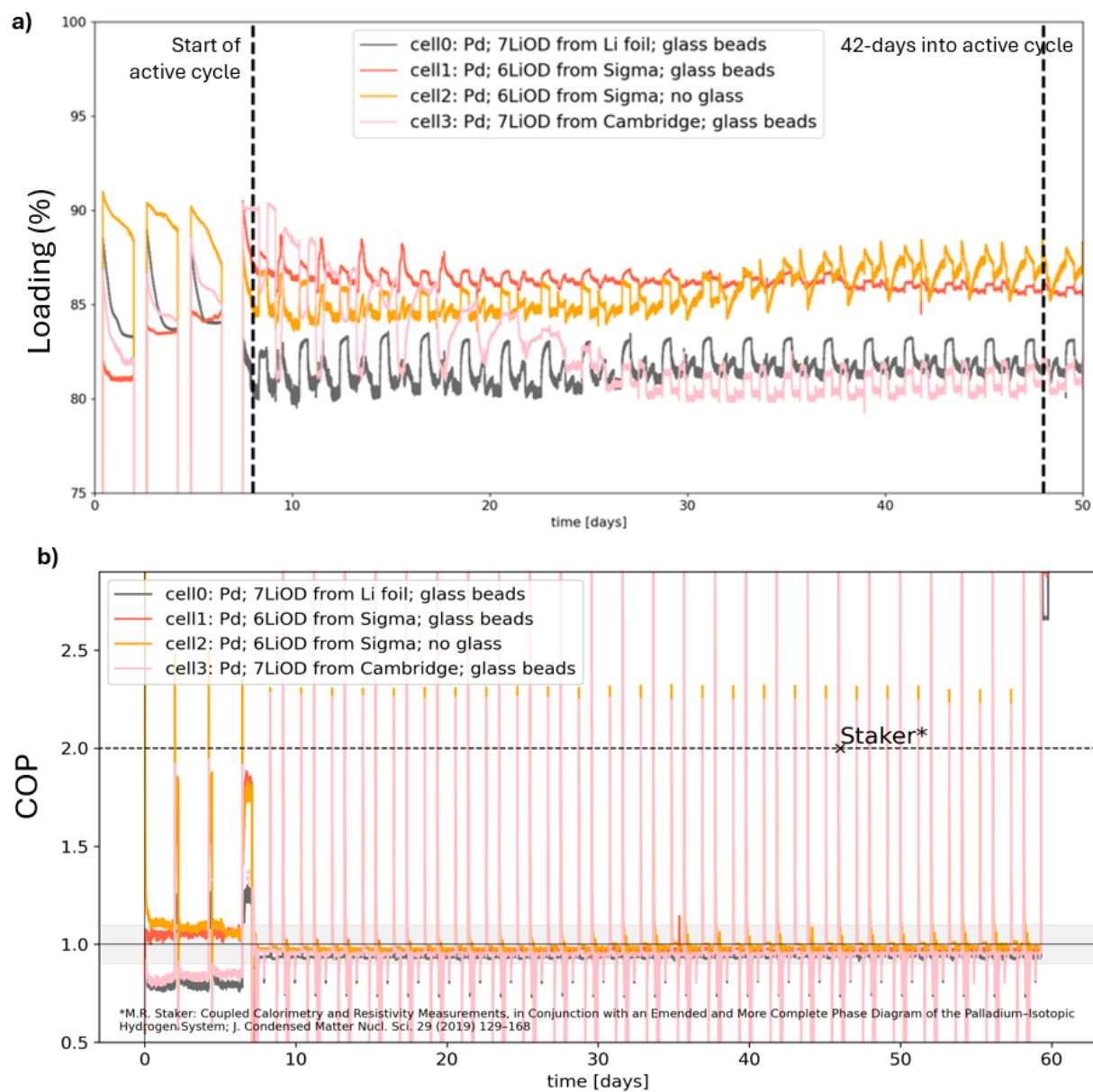


Fig. 7.13: Loading of all cells from 60-day long-duration Staker-like test with glass bead additive and various electrolytes. Loadings remained above 80 % for nearly all cells for the entire experiment. (a) COP for each cell with error band (grey). (b) All steady-state COPs were near unity for the duration of the experiment. All COP outliers (vertical lines) are due to heat flow transients when input power was changed as prescribed. A dashed horizontal indicator showing Staker's originally reported excess power COP is included for reference.

sitions regardless of their electrochemical treatment. The EDS summary table shows that direct replications (Cells 0 and 1) and controls (Cells 2 and 3) all had elements of ‘Coating-X’ (Cu, Fe, and Zn) present on their surfaces. While these elements are to be expected to be present after our experiments due to known electrolyte impurities, other elements such as Sn, Pb, and Cr are likely due to hardware degradation resulting from the foaming electrolyte contacting soldered leads. These soldered connections were shielded with PTFE shrink wrap and Microstop (Brand of Tolber) but over the course of long duration experiments electrolyte can work its way past these protective measures. These elements from the degradation of this solder are not observed in Cell 1, likely because there was no intermittent recombiner failure as occurred in other cells (Figure 7.11b).

Figure 7.12 is a summary figure of a 60 day Staker-like replication in closed-cell calorimeters with glass beads additives. These glass beads were introduced to mimic the electrochemical environment of the original experiment which Staker carried out in a quartz vessel. Considering the fact that alkaline electrolytes – especially at high temperatures – have been observed to attack quartz, [245, 246] in conjunction with many LENR reports being carried out in quartz vessels, it is feasible that some unintended Si additive could play a role in triggering AHE. Figure 7.13 shows the loading (7.13a) and COP (7.13b) of all cells in the the 60 day Staker-like replication experiment. The loading for each cell is shown to be above 80 % for duration of the experiment with vertical markers showing the start of EM stimulation after pre-cycling and 42 day mark, respectively. This 42 day mark is to mark a comparable time of experimentation to Staker’s experiment. As previously discussed, maintaining high loadings is reportedly a prerequisite for an AHE event to be observed, though we concede our loadings did not remain as high as reported by Staker in his works (however owing to Staker’s use of only a 2-point resistance measurement, rather than 4-point, the accuracy of his loading claims are subject to skepticism). In Figure 7.13b we show the COP for all cells over the duration of the experiment with COP error bars highlighted in grey and a horizontal marker depicting Staker’s original claim of COP 2.0 (later retracted to 1.67) excess power for reference. The COP outliers (vertical lines) are due to heat flow transients when input power changes as prescribed. They cancel out with those outliers induced by the changes in input power in the opposite direction. During the duration of this experiment we did not observe any AHE events or unaccounted excess heat. Any excursions were readily explained by energy accounting and determined to be chemical in nature (i.e., recombination events) and not nuclear.

7.3.3.4 Experimental Conditions: UHTP

The UHTP calorimetry system was designed to replicate experiments inspired by Staker’s report. Most critically, it has the option to apply an electromigration current along the wire sample. See section 2.10 for calorimeter details. The experimental campaign detailed in this section is our first long duration experiment (BE4445) with 80 experimental cells (sectors A - D) as the last 20 cells in sector E were still being brought online. In addition to Staker-like conditions, we also found inspiration in other reports of AHE, in particular in the selection of additives [101] and we designed the experiments accordingly.

Figure 7.14 shows a summary of the conditions for each of the experimental cells in the UHTP system. Each cell contained a 0.5 mm diameter and 30 mm long Pd (or Pt control) cathode. The Pd cathodes were either used as received, annealed in argon, annealed in forming gas, or etched in acid. Each cell had 48 ml of 0.5 M LiOD for active cells or 0.5 M LiOH for control cells. The EC treatment for each sector was roughly similar for every sector except Sector B, which did not receive electromigration (EM) current. The EC treatment started with a pre-cycle of the samples undergoing the following steps:

- EC was ramped to a cathodic 130 mW over 2 hours and held at that negative bias for 7 hours.

- EC was ramped to 0 mW over 1 hour and held at open circuit voltage for 2 hours.
- Samples were actively deloaded at 1.2 V and 1.4 V for 3 and 9 hours, respectively.
- Samples allowed to dwell for 5 hours.

This pre-cycling process was repeated three times. For the EC/EM loading cycles a 3 A EM current was applied to sectors A, C, and D for the duration of the experiment, with sector B receiving no EM current. While EM power was constantly being applied, the samples were ramped to 130 mW (negative bias) over a two hour period. The power was held constant at a cathodic 130 mW, 300 mW, and 1 W for 12 hours each. This is shown in the input power plot in Figure 7.15. This constant power application loop was repeated continually for 50 days. Considering we ran 80 samples in parallel, the cumulative time of calorimetry amounted to 11 years in this single experiment.

SECTOR A									
Position	Sample Type	Electrolyte	# Recombiners	Recombiner type		Position	Sample Type	Electrolyte	# Recombiners
09	Pd	LiOH	4	Legacy		19	Pd	LiOH	4
08	Pd	LiOD	7	Legacy		18	Pd	LiOD	7
07	Pd	LiOD	7	Cloth		17	Pd	LiOD	4
06	Pd	LiOD	4	Cloth		16	Pd	LiOD	7
05	Pd	LiOD	7	Legacy		15	Pt	LiOH	7
04	Pd	LiOH	7	Legacy		14	Pd	LiOD	7
03	Pd	LiOD	4	Cloth		13	Pd	LiOD	4
02	Pt	LiOD	7	Cloth		12	Pt	LiOD	7
01	Pd	LiOD	7	Legacy		11	Pd	LiOD	7
00	Pd	LiOD	7	Legacy		10	Pd	LiOD	7

SECTOR B									
Position	Sample Type	Electrolyte	# Recombiners	Recombiner type		Position	Sample Type	Electrolyte	# Recombiners
29	Pd	LiOD	4	Cloth		39	Pd	LiOD	4
28	Pd	LiOD	4	Cloth		38	Pd	LiOD	4
27	Pd	LiOH	4	Cloth		37	Pd	LiOD	4
26	Pd	LiOD	4	Cloth		36	Pt	LiOD	4
25	Pd	LiOD	4	Cloth		35	Pd	LiOD	4
24	Pt	LiOH	4	Cloth		34	Pd	LiOD	4
23	Pd	LiOD	4	Cloth		33	Pd	LiOH	4
22	Pd	LiOD	4	Cloth		32	Pd	LiOD	4
21	Pd	LiOD	4	Cloth		31	Pd	LiOD	4
20	Pd	LiOH	4	Cloth		30	Pd	LiOD	4

SECTOR C									
Position	Sample Type	Electrolyte	# Recombiners	Recombiner type		Position	Sample Type	Electrolyte	# Recombiners
49	Pd	LiOD	4	Cloth		59	Pd-An-Ar	LiOD	4
48	Pd-An-Ar	LiOD	4	Cloth		58	Pd-E	LiOD	4
47	Pd-E	LiOD	4	Cloth		57	Pd-An-FG	LiOD	4
46	Pt	LiOH	4	Cloth		56	Pd	LiOD	4
45	Pd-E	LiOD	4	Cloth		55	Pd-An-Ar	LiOD	4
44	Pd-An-FG	LiOD	4	Cloth		54	Pd	LiOH	4
43	Pd	LiOH	4	Cloth		53	Pt	LiOD	4
42	Pd-An-Ar	LiOD	4	Cloth		52	Pd-E	LiOD	4
41	Pd	LiOD	4	Cloth		51	Pd-An-FG	LiOD	4
40	Pd-An-FG	LiOD	4	Cloth		50	Pd	LiOD	4

SECTOR D									
Position	Sample Type	Electrolyte	Additive Type	Additive Qty (mg)		Position	Sample Type	Electrolyte	Additive Type
69	Pd-An-FG-E	LiOD	0	0		79	Pd	LiOH	0
68	Pd-E	LiOD	Al	10		78	Pd	LiOD	Al
67	Pd	LiOH	SiO2	100		77	Pd-An-FG-E	LiOD	SiO2
66	Pd-E	LiOD	SiO2	10		76	Pd	LiOH	Al + SiO2
65	Pd-An-FG-E	LiOD	0	0		75	Pt	LiOD	Al + SiO2
64	Pd-E	LiOD	Al	10		74	Pd-E	LiOD	Al + SiO2
63	Pd	LiOD	Al	100		73	Pd-An-FG-E	LiOD	Al
62	Pd-E	LiOD	SiO2	10		72	Pd-E	LiOD	(hidden)
61	Pt	LiOH	0	0		71	Pd	LiOD	SiO2
60	Pd-E	LiOD	Al	10		70	Pd-E	LiOD	(hidden)

Fig. 7.14: Summary plots of each of the 80 experimental cells composition including cell number, sample type, electrolyte, number of recombiners and their type, and additive type and quantity. All electrolyte concentrations are 0.5 M. The key for the Pd samples is as follows: Pd (Palladium), Pt (Platinum), Pd-Ar (Palladium - As received), Pd-An-Ar (Palladium annealed in argon), Pd-An-FG (Palladium annealed in forming gas), and Pd-E (Palladium etched).

7.3.3.5 Results: UHTP

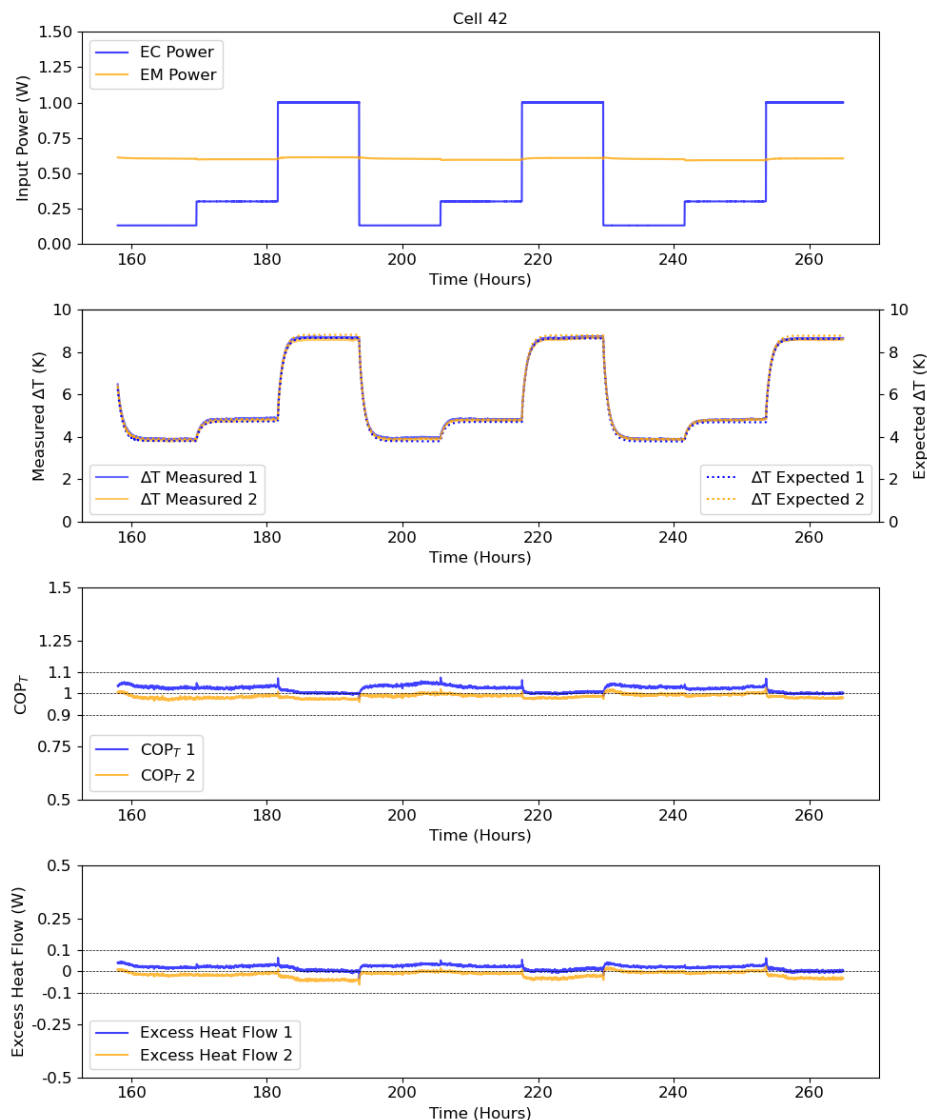


Fig. 7.15: A representative example of an experimental cell (Cell 42, Pd in LiOD) and the standard data streams monitored in the UHTP system for an 100 hour window during the experiment. Data include (from top to bottom) input power (both EC and EM Power), measured ΔT , COP_T , and excess heat flow.

Figure 7.15 is a representative example of an experimental cell and the data streams we typically monitor in the UHTP system. These include input power (W), measured ΔT (K), COP_T , and excess heat flow (W).

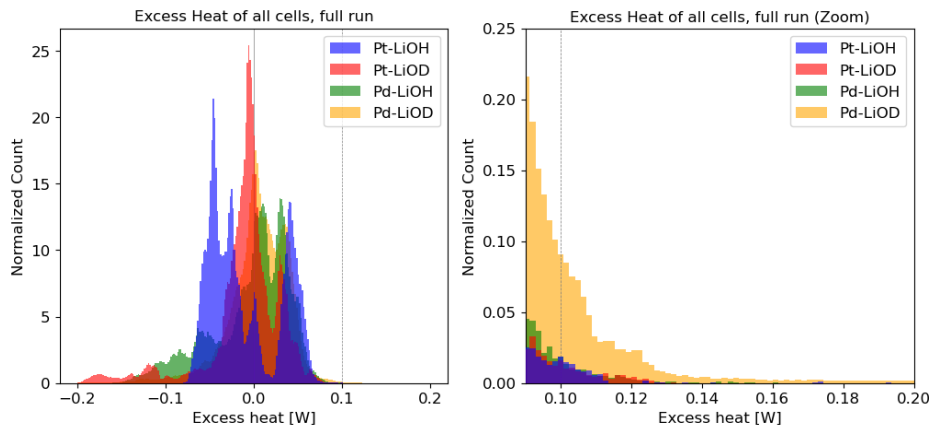


Fig. 7.16: Histogram comparing excess heat for all 80 experimental cells. The left panel shows excess heat of control cells (Pt in LiOH) in blue, Pt in LiOD in red, Pd in LiOH in green, and Pd in LiOD in yellow. The right panel of the figure is a zoom in on any excess heat above 0.1 W. Counts are normalized to cause the total area under the histogram to equal to one. This allows comparison between different sized data sets (i. e., 18 control cells compared to 62 active cells).

Measured ΔT is calculated by subtracting the expected temperature from the measured temperature. In this example cell the electrochemical stimulation scheme is shown in the input power plot (7.15) where input power is held constant for ~ 10 hours before increasing to the next power step. The effect of these power increases can clearly be seen in the ΔT plot. Small changes in the COP_T and excess heat flow plots can be seen when changes in input power occur. These changes are well within the error bands shown and thus can not be ruled out as an excess heat event, but are likely caused by transients due to the change in input power change. These data streams combined give us reliable readings across all cells in the system and allow us to view relevant conditions in the event of any AHE. They also allow us to rule out any false positives due to various failure modes we have observed in previous experiments (i. e., hardware failure, electrode contact error, and spontaneous oxyhydrogen recombination after a failing recombiner recovered).

The results of our first UHTP experimental campaign are shown in Figures 7.15 (COP_T and excess heat) and 7.16. While we did not observe a significant excess heat, we were able to reliably track heat flow of 80 individual cells and generate meaningful statistics, a testament to both the highly parallel and sensitive nature of the UHTP system. Figure 7.16 shows a comparison of excess heat with control cells (Pt in LiOH) in blue, Pt in LiOD in red, Pd in LiOH in green, and Pd in LiOD in yellow. The right panel of both figures shows a zoomed in plot of the excess heat flows greater than 100 mW, a minimum threshold we would expect for any real anomalous heat event. It appears that we have excess heat above 100 mW in the right panel of Figure 7.16, but upon closer inspection we found that a faulty RTD in one of the cells contained 64% of all samples ≥ 100 mW. This error finding is a prime example of how the UHTP's data visualization would clearly allow us to see an excess heat event in comparison to the control cells and investigate which cells were responsible.

While no excess heat was found, this first long duration test in the UHTP system proved its utility as a highly parallel system that vastly improves experimental throughput while boasting a reasonable sensitivity to detect AHE events. After proving out the first long duration UHTP test, we were ready to implement

multiple different conditions we hypothesized could lead to AHE whether from previous works or our own research. These tests are ongoing.

7.3.4 Conclusions

Callisto put tremendous effort into the replication of Staker's experiment producing excess heat. After more than 200 experiments, we have not found definite excess heat. However, we encountered various circumstances which produced false excess heat signals with an apparent COP up to 1.6. Most notable sources of false excess heat signals were changes in electrolyte level and difficulties in accounting for electromigration input power into the thermal system boundary owing to the high currents and small voltages. These small voltages can change drastically in the event of a bad contact.

Due to these shortcomings, we transferred the experiment into a closed-system calorimeter, while maintaining the general design of the electrolytic cell. We saw much fewer false excess heat signals in the closed system, most of them caused by spontaneous bursts in recombination after a buildup of oxyhydrogen, which could be easily diagnosed from pressure changes.

We would like to highlight that Staker's excess heat event reportedly was a one-off, was not published in a peer-reviewed reputable journal and that the report contained many shortcomings, including unintelligible axis labels on the central COP plot. Moreover, the central outcome of the report was later redacted in a corrigendum. In addition to excess heat reports, Staker's papers contain lengthy speculations about the mechanism producing the alleged excess heat. However, no experimental evidence is provided, calling into question his scientific rigor. We conclude that most likely Staker encountered a similar false COP as we did.

7.4 Hubler

7.4.1 Literature Review

Graham Hubler is a nuclear physicist that worked for the Naval Research Laboratory for 40 years, is a professor at the University of Missouri, and was the leader of the Sydney Kimmel Institute for Nuclear Resonance (SKINR). Over the years he has published multiple works spanning topics of nuclear energy, material science, and even brain related research. Amongst these works, he collaboratively published works focused on investigating and reproducing low-energy nuclear reactions (LENR) [247–249] as well as presented LENR experiments at the International Conference for Cold Fusion (ICCF) [250]. Collaborators include the European Nuclear Energy Agency (ENEA). In the 2015 work entitled 'Progress towards understanding anomalous heat effect in metal deuterides', he reported that an anomalous heat event (AHE) yielding hundreds of kilojoules of energy and an average COP of more than 15 was observed. These experiments were carried out through electrolysis in heavy water (D_2O) with either pure palladium (Pd), palladium alloys, or composite Pd cathodes coated in carbon nanotubes as the working electrodes. Pt counter electrodes were used in all experiments. Three of his experiments – experiment #64, Pd under ultrasonic wave and 'SuperWave' stimulation, and Pd coated with single-walled carbon nanotubes (SWCNTs) – which reportedly show excess heat are described in detail.

Experiment #64 was carried out using a pure Pd cathode in 0.1 M LiOD/ D_2O . The Pd cathode was cold-rolled to a thickness of 50 μm , annealed at 850 $^{\circ}C$ for an hour, and finally quenched in 50 % aqua regia for 1 minute. The electrochemical stimulation is called 'SuperWaves' which consist of a superposition of time-dependent regulated currents having several different amplitudes and frequencies [251]. They are designed to dynamically modify the interface between the electrodes and electrolyte so as to increase the flux of deuterium

in and out of a Pd cathode [247]. The heat of the cell was measured using an isoperibolic calorimeter which was placed in a temperature controlled water bath with a temperature measurement accuracy of ± 0.01 °C. The results of the application of SuperWave stimulation to the Pd sample was reported to be an event exhibiting excess power for a 17 hour period, starting 5 hours into the experiment. A maximum of 34 W and average of 21 W of output power on just 0.81 W of input power was observed. This translated to a COP of 2.5 (21 W out on 0.81 W in). It was also observed that during this event, the cell voltage increased from 3 V to nearly 15 V with about 20 % fluctuation around the newly established average voltage. At the end of the reported event voltage fluctuations ceased and the voltage returned back to 3 V. Post-mortem sample analysis was carried out on the purportedly AHE active Pd samples using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). The SEM and EDS showed that the surface of the AHE active cathode was covered in black spots compared to a non-AHE active sample made from the same parent Pd. However, these black spots were mainly comprised of C and O and were the only detectable difference between the electrodes.

A similar experiment was carried out with the pure Pd cathodes being stimulated in LiOD with the SuperWave technique, however, this experiment also exposed samples to ultrasonic (US) waves during the course of electrolysis. This was "...expected to excite cathodes through mechano-chemical cleaning of cathode surfaces and by causing surface pits which are created by shockwave-generated cavitation jets. US-induced pits are expected to be especially significant relative to promoting AHE because they cause dynamic mechanical deformation of the near-surface layer of the cathode resulting in defects (e. g. dislocations and vacancies) that can potentially trap deuterium." [247] This surface pitting was confirmed via SEM. The authors claim that this pitting/ultrasonic excitation led to the observation of D/Pd loadings ~95% using minimal current densities (6-10 mA/cm²) and the long duration of the Pd β -phase (~100 days). Ultimately, this experiment was reported to have exhibited excess heat over the duration of the experiment with two major 'burst' events releasing 1) 1.32 MJ of excess heat over 18 days with a maximum COP of 5.25 and 2) 185 kJ over the course of 23 days with a maximum COP of 1.40.

The final experiment reported to have exhibited excess heat used a composite cathode made from Pd foils coated with SWCNTs. Samples were created by drop casting a 0.1 mg/cm² SWCNT dispersion onto pre-treated Pd foils, followed by electrodeposition of 0.1 M PdCl₂ at 2.5 mA, and finally constant current application in 0.1 M LiOD. Two occurrences of excess heat were observed during this experiment: 1) a 3 hour period releasing 75 kJ of excess heat with peak and average COPs of 55 and 35 respectively and 2) a 1.5 hour period releasing 13 kJ of excess heat with a peak and average COPs of 27 and 16.6 respectively. Researchers observed that for a constant input power, the voltage increased from the original steady state near 2.5 V to fluctuations between 7 - 10 V during the excess heat event. Resistance also was reportedly 2.1 times the baseline value with no input power during the events. The authors report D/Pd loading of ~75%, though the resistance data in the plots is hard to decipher to corroborate. It is important to note that the data does not show resistance data returning to the original baseline resistance until after the second event.

Most recently, Hubler gave an oral presentation of reportedly excess heat reactions at the ICCF 23 conference in Xiamen, China in 2021 [250]. He presented data describing an anomalous heat event producing ~90 mW of power on 42 mW of input power (authors report COP of ~1.9, though the raw calculation of COP is 2.14) over a 58 day period, yielding an estimated 453 kJ during this time. These results are shown in Figures 7.17a and 7.17b which have been reproduced here for convenience. This experiment had a Pd cathode (100 mm x 8 mm x 0.1 mm) under tension to prevent buckling which purportedly releases deposited nanoparticles. However, we draw attention to the fact that there is no data presented showing that buckling releases nanoparticles nor is there data that shows excess heat without nanoparticles present. These samples were placed in a mixed isotope electrolyte of 0.1 M LiOD in H₂O, Pt wire anodes wrapped about the

support pillars, and 4-point resistance measurements (Figure 7.17c). 9 nm Pd nanoparticles were added to the electrolyte which turned the solution black until 2 days after the start of electrolysis where the solution became clear, indicating that all of the nanoparticles had plated out on the cathode. The experimental vessel was an open cell calorimeter and the sample apparatus is pictured in Figure 7.17c. The experiment exhibited excess power within the first 10 hours of the experiments and persisted for nearly 100 hours after input power was terminated. They report that their sample loaded to D/Pd of ~ 0.7 and was fully loaded after 30 minutes of stimulation current. It is important to highlight some shortcomings of the data presented such as: R/R_0 value being higher than expected in the presence of significant amounts of protium, quick loading times of 30 minutes at low currents (1.5 mA/cm^2) compared to our sample where we observed loadings taking ~ 5 hours, lack of temperature stabilization before the test (based on the scale in the red trace 7.17b), and the quite small change of the electrolyte temperature (0.1°C) upon which the claim of AHE is based. We attempted a replication of this most recent experiment, as it exceeded our replication threshold COP and it was published during our project runtime. The shortcomings of the work were improved upon in our subsequent experiments to provide the highest accuracy possible.

7.4.2 Replication Experiments

Our samples were synthesized from Pd foils $25 \times 4 \times 0.1 \text{ mm}$ from Alfa Aesar, and the nanoparticle solution was created according to the same patent referenced by Hubler that he published in 2013 [252]. The nanoparticle deposition was comprised of an $1.6 \text{ mM Na}_2\text{PdCl}_4$ and 0.8 mM Epigallocatechin gallate (EGCG) mixture in 25 mL of DI water for 3 days. 0.1 M LiOD was added to create the final electrolyte and deposition on the cathode was done at a current density of 1.5 mA/cm^2 . Nanoparticles were deposited for 30 hours for which Hubler reported the electrolyte to have returned to completely clear from black, indicating the full deposition of the suspended Pd nanoparticles. In Figure 7.18 we show SEM images comparing our in-situ deposited Pd nanoparticle samples on Pd foils in 0.1 M LiOD in H_2O (7.18a) and D_2O (7.18b) to Hubler's in-situ depositions in 0.1 M LiOD in H_2O (7.18c) and in D_2O (7.18d). It is clear that our surface morphologies closely match those of his samples after scaling concentration for our sample size. After ensuring our samples were prepared in the same manner, we replicated (i.e., EC current density (1.5 mA/cm^2), duration (30 hours), electrochemical environment (0.1 M LiOD in H_2O), etc.) the most recent Hubler work claiming excess power of 90 mW . Figure 7.19 shows representative results of two variations of the Hubler experiment. Figure 7.19a shows a Hubler-like replication with a Pd nanoparticle-deposited Pd cathode in 0.1 M LiOD in D_2O exposed to 20 mW of input current. Figure 7.19b shows a direct replication of Hubler's experiment with the Pd nanoparticle-deposited Pd cathode in 0.1 M LiOD in H_2O with 20 mW of input current. The top plots in both Figures 7.19a and 7.19b show the EC power and current for both the Pd cathode and the Pt control cell. In the last 25 hours of the experiment, input power was changed in an effort to trigger an AHE event. 10 mW negative heat flow at the beginning of the experiment can be ascribed to the difference in the heat of recombination as Pd loads. The baseline data before EC is applied is shown in 7.19b between 0 and 15 hours. A few hours of baseline data are present in 7.19a but appear small due to scaling. As shown in the bottom plots in both figures, no excess heat was observed over the duration of the 200 hour experiment. Considering that Hubler observed an event after the first 10 hours of power application, we would have expected to observe some excess power over a 200 hour window if the event was reproducible.

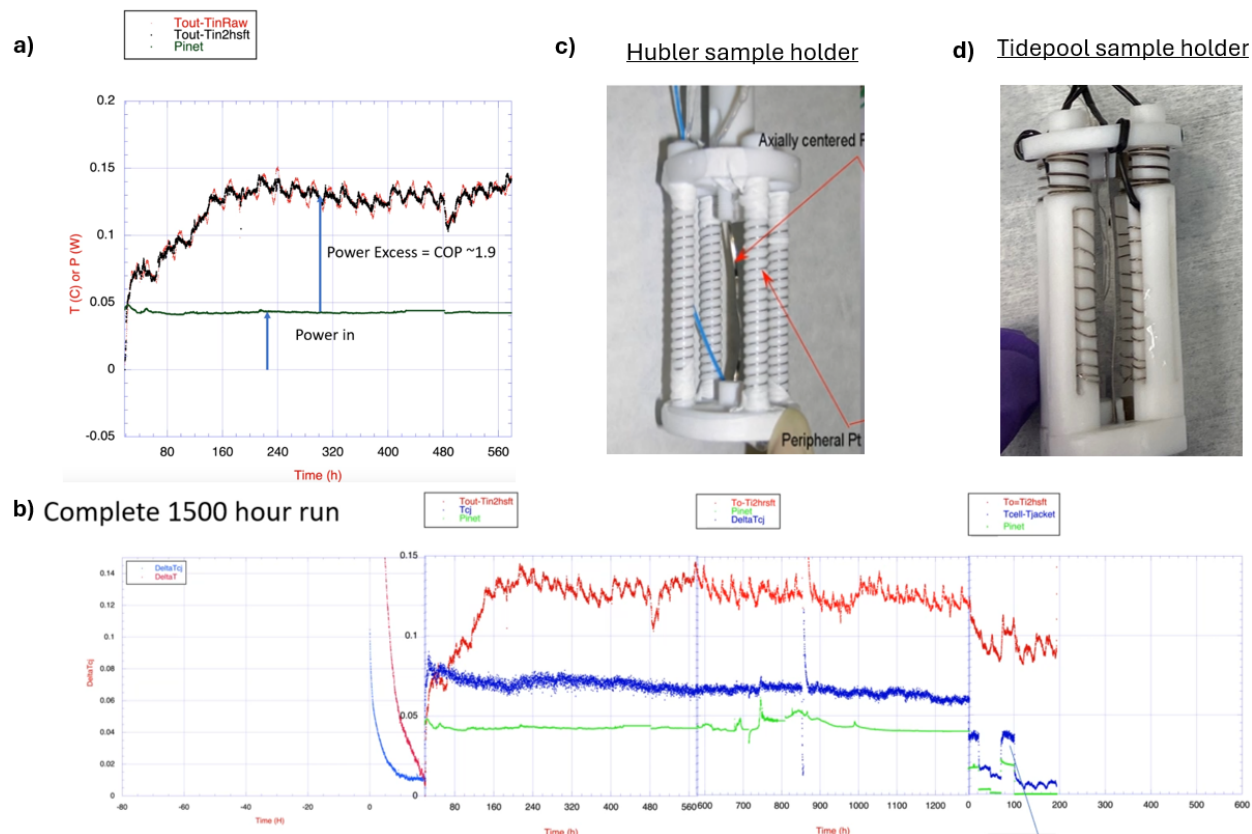


Fig. 7.17: a) to c) Summary of Hubler's major findings (replicated from [250]): a) Excess power of 90 mW (red) on 42 mW of input power (green), yielding a COP of 1.9. b) Full duration of the experiment showing input power (green), output power (red), and the temperature of the outer vessel (blue). Examples of the experimental retention housing the Pd cathode under tension and the wound Pt anodes are shown for both c) Original Hubler sample holder and sample. [250]. d) Callisto sample holder and sample.

7.4.3 Conclusions

We could not replicate the excess heat events that Hubler reported. Our replication efforts focused on his most recent report at ICCF23 [250], assuming that his latest work represents the culmination of his research and is thus most worthwhile to replicate. While the exact cause for the discrepancy between the replication and the report remains unclear, we would like to highlight that his work was not published in a peer-reviewed reputable journal and that it contained some inconsistencies. Most notably, in the central heat flow plot illustrating the excess heat event, the outlet temperature of the mass flow calorimeter exceeded the inlet temperature of the mass flow calorimeter (indicating excess heat) even at times when the cell temperature did not significantly exceed the jacket temperature, suggesting that the cell was not the heat source.

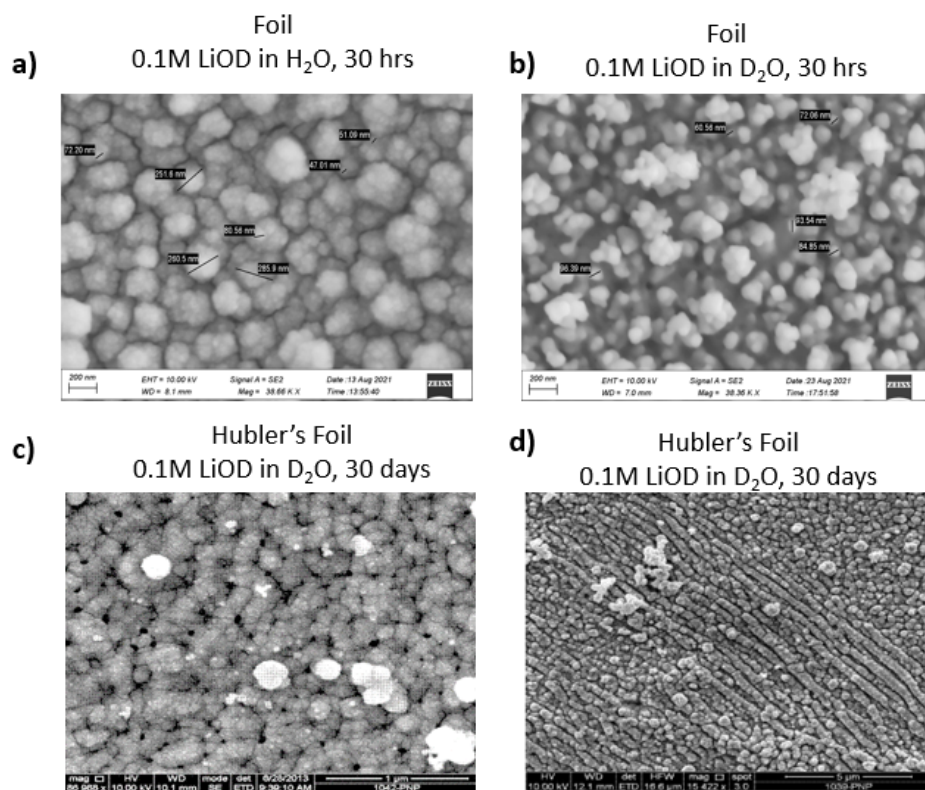


Fig. 7.18: Scanning electron microscopy (SEM) images of in-situ deposited Pd nanoparticles on a Pd cathode. a) Shows the results of our in-situ deposition following Hubler's original electrolyte composition of 0.1 M LiOD in H₂O for 30 hours. b) Shows the result of the same in-situ deposition in a different electrolyte mixture of 0.1 M LiOD in D₂O for 30 hours. These images are compared to Hubler's original 2013 work where this method was developed and patented. c) Shows Hubler's Pd foil after in-situ deposition in 0.1 M LiOD in H₂O and d) 0.1 M LiOD in D₂O for 30 hours. Scale bars for a) and b) are 200 nm and 1 μ m and 5 μ m for c) and d), respectively [250].

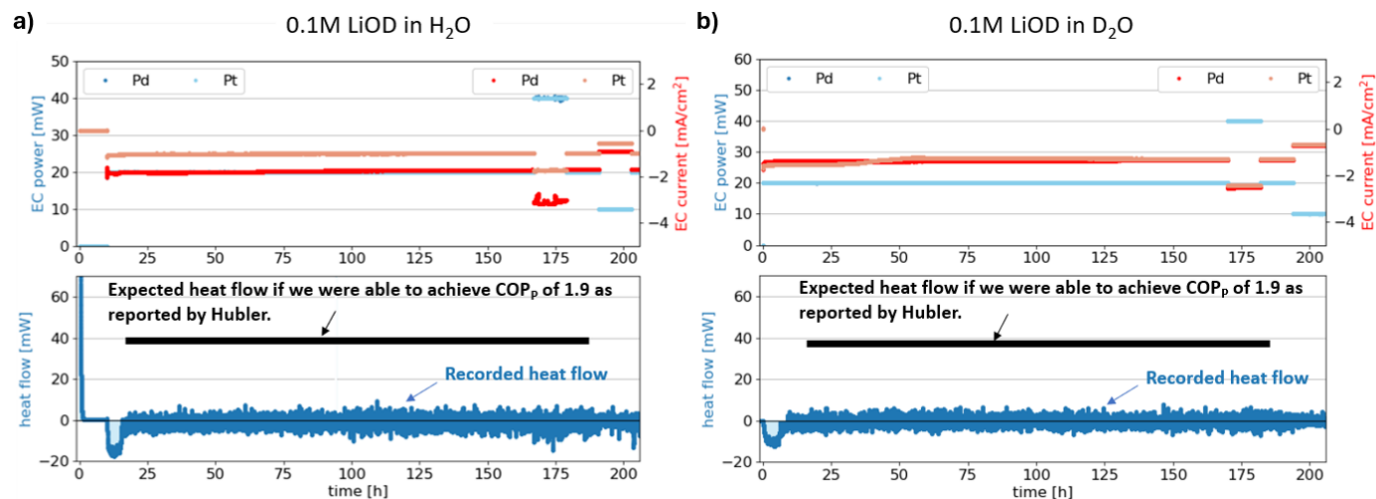


Fig. 7.19: Shows summary data for two variations of the Hubler experiment replicated in the high-precision MS80 calorimeter. a) A direct replication of Hubler's experiment with the Pd nanoparticle-deposited Pd cathode in 0.1 M LiOD in H₂O with 20 mW of input current and b) a Hubler-like replication with a Pd nanoparticle-deposited Pd cathode in 0.1 M LiOD in D₂O exposed to 20 mW of input current. There is a horizontal bar indicating Hubler's reported COP of 1.9. The heat flow is from the probed cell (Pd) to the reference cell (Pt), as the MS80 calorimeter only provides relative heat measurements.

7.5 Storms

7.5.1 Literature Review

Edmund Storms has been a prominent figure in the LENR community, conducting experiments since the early 1990s. He has been a strong advocate for the use of Seebeck-style calorimeters, arguing that they address the shortcomings of many isoperibolic designs. Storms has disseminated his findings through various channels, including books, scientific articles, and conference proceedings. Despite the breadth of his work, he has not provided incontrovertible evidence of LENR. This review focuses on Callisto's attempt to replicate the experimental conditions described in a series of six reports and conference proceedings from the International Conference for Cold Fusion (ICCF) by Storms, which outline his experimental findings, methodologies, and theoretical insights into reliably replicating and understanding LENR. Storms' research primarily involved palladium (Pd) and palladium-silver (Pd-Ag) alloys loaded with deuterium (D) to explore their potential for producing excess heat.

In his first two progress reports, Storms described the construction of a Seebeck calorimeter designed to measure small amounts of excess heat with high precision [253,254]. This calorimeter was built to facilitate electrochemical experiments similar to those proposed by Fleischmann and Pons. It was calibrated using various heat sources, including resistors and quartz light bulbs, and had a time constant of approximately 40 minutes to reach equilibrium. This setup allowed for the precise detection of excess power levels as low as ± 0.05 W (50 mW). In subsequent reports, he made changes to the hardware that brought the claimed detection limit to ± 0.013 W (13 mW).

Progress reports three and four shifted focus to examining the behavior of pure palladium when loaded with deuterium [255,256]. Storms treated palladium samples with heat to maximize their deuterium loading capacity, reporting high loading ratios, with D/Pd values reaching 0.98. Based on Callisto's replication efforts and extensive experience with electrochemical loadings, such high loading results should be approached with extreme skepticism. It is likely that Storms overestimated his loadings by as much as 10 to 15 %. Storms claimed that the high loading was achieved by treating the Pd with concentrated HNO_3 , followed by heating at 900 °C and slow cooling, which resulted in a reactive PdO surface. He argued that this surface oxide led to superior deuterium uptake. However, it is possible that the reduction of the oxide affected his ability to accurately calculate loading. If the sample was more heavily oxidized than Storms estimated, this could lead to significant overestimation of the true loading. A simple calculation using his reported palladium masses indicates that for 1 %, 5 %, and 10 % oxidation, the true loading would be approximately 96 %, 88 %, and 78 % D/Pd, respectively.

During these experiments, Storms observed that the samples exhibited expansion behavior inconsistent with changes in lattice parameters alone, suggesting internal stress and crack formation. This excess volume observation has been corroborated by other researchers and appears to be a valid finding. However, Storms hypothesized that these cracks were essential for LENR, proposing that they could create nuclear active environments (NAEs) within the material. It is unclear that his samples are in fact nuclear active, and therefore the cracking may be inconsequential.

In his fifth progress report, Storms focused on the role of temperature in excess heat production [257]. He claimed that once the palladium was loaded with deuterium and activated, increasing the temperature significantly enhanced excess power production. This challenged the previously held belief that high applied current was necessary to initiate LENR, suggesting instead that thermal conditions at the nuclear active sites were more critical. This claim should be scrutinized due to a lack of evidence showing that the calibration coefficients of his Seebeck calorimeter were insensitive to temperature variations.

Storms noted a distinct pattern when varying the temperature of the cathode, claiming that excess power production increased with temperature, reaching between 0.3 and 0.5 W when the cell temperature was raised to around 60 °C. He correlated this increase with a reduction in the open circuit voltage (OCV), which he attributed to changes in surface deuterium activity. However, it is important to note that electrolyte resistance typically decreases as temperature rises, which can lead to a substantial reduction in voltage. In fact, the electrolyte resistance of LiOH solutions at similar concentrations can decrease by up to half an order of magnitude, explaining the observed drop in OCV with temperature. Storms also claimed that his calorimeter detected excess heat production after the applied current was turned off, a phenomenon he termed 'heat-after-death'. When electrochemical biases are removed from palladium deuteride, the sample enters a chemically dynamic state of deloading, and it is unclear that his calorimetric analysis is sophisticated enough to account for this.

In his final report, Storms sought to correlate excess heat production with radiation emission, a common expectation in nuclear reactions [258]. Although initial results suggested a correlation, further analysis showed that this was an experimental artifact. The apparent radiation was actually due to the temperature sensitivity of the Geiger-Müller (GM) detector used in the calorimeter. Storms demonstrated that temperature changes alone, without deuterium loading or nuclear reactions, produced similar apparent radiation signals, highlighting the importance of eliminating artifacts in LENR research.

More recently, Storms presented his work at ICCF 23, where he discussed his latest protocol for fabricating oxidized, nanoporous palladium [259]. Callisto adopted that fabrication protocol, as it represented the most up-to-date conditions provided by Storms. Rather than relying on crack formation to create NAEs, Storms intentionally incorporated nanoporosity into the sample design to eliminate the need for extensive sample conditioning.

Throughout his investigations, Storms claimed to identify key factors influencing LENR in palladium-deuterium systems. He observed that temperature was the primary factor affecting excess heat production, more so than the applied electrolytic current. Storms also noted that the physical state of the palladium, including impurity content and structural characteristics, played a critical role in LENR. He suggested that samples showing minimal excess volume changes and maintaining high deuterium loading ratios were more likely to produce excess heat. These conclusions supported his theory that LENR requires specific conditions of crack formation and deuterium concentration, aligning with his earlier theoretical work on nuclear active environments.

7.5.2 Replication Experiments

Callisto chose to replicate what we believed to be the critical aspects of Storms' reported conditions for LENR, primarily the sample fabrication and electrochemical conditions. As is often the case in the LENR literature, incomplete details are provided and therefore some replication details require an educated guess. With that said, we replicated such conditions in our MS80 differential calorimeter due to this system having the lowest detection limits and highest sensitivity of any calorimeter in our arsenal (section 2.8.1). Necessary details are discussed herein.

As with many of our replications, we set out to replicate the alleged nuclear active material first. For our Storms replications, we decided to follow the details outlined in his most recent public communication, i.e., his conference proceedings from ICCF-23. In this conference paper, Storms describes the sample fabrication as follows:

"The samples were prepared by pressing the powders in a steel die to form a disk shape about 12 mm in diameter and about 2 mm thick. This disk was then subjected to various treatments including heating in a

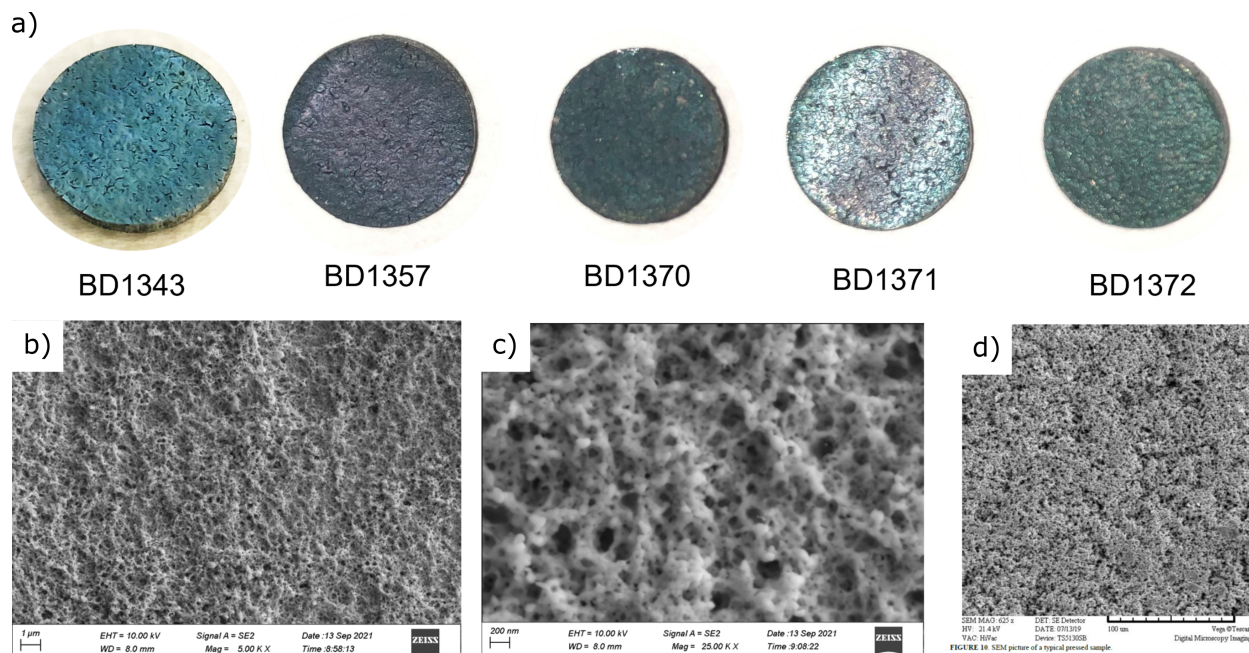


Fig. 7.20: a) Photograph of some replicated Storms samples, with varying degrees of oxidation. b) SEM micrograph of BD1343 (scale bar 1 μm). c) High magnification SEM micrograph of BD1343 (scale bar 200 nm). d) SEM micrograph of Storms sample in ICCF-23 report (scale bar 100 μm).

vacuum at 900 to 1000 °C and/or heating in air at various temperatures and times. The amount of physical pressure used to compact the powder was not explored but is expected to affect the amount of generated power. The formation of an oxide layer and the addition of inert material in the form of a fine powder are also found to affect success. Even though the conditions were not optimized, active samples could be made with reliability. Although many variables and treatments were explored during this study, this paper is focused only on a few behaviors produced by a few samples to show examples of typical behavior.”

From the above excerpt, we have very little information to rely on. However, we decided to attempt to make the samples, as Storms states the conditions were not optimized and still yielded results, indicating an insensitivity to processing conditions. In our attempts to replicate the material, we sourced palladium micropowder with a mean diameter of 3 μm from Goodfellow. The samples were pressed at a constant displacement rate to a thickness corresponding to 50 % full density using an Instron-3369 tensile testing machine. During our first attempt at fabrication, an error in the pressing protocol led to the sample being compacted to full density. The sample was removed, placed in a vitreous carbon dish, and sintered in high vacuum and at 1000 °C for 19 hours. However, upon removal from the pressing-die and prior to sintering, the sample fractured into several large pieces. We suspected the 'addition of inert material' in the report was alluding to a powder-lubricant which is common practice in powder metallurgy. In subsequent attempts to replicate the material and after optimization, we included 1.5 wt.% polymeric lubricant N,N'-ethylenebis(stearamide) in the powder mixture. This allowed for removal of the pellet from the pressing-die without catastrophically compromising the form factor. In subsequent fabrications, the pressed pellet was

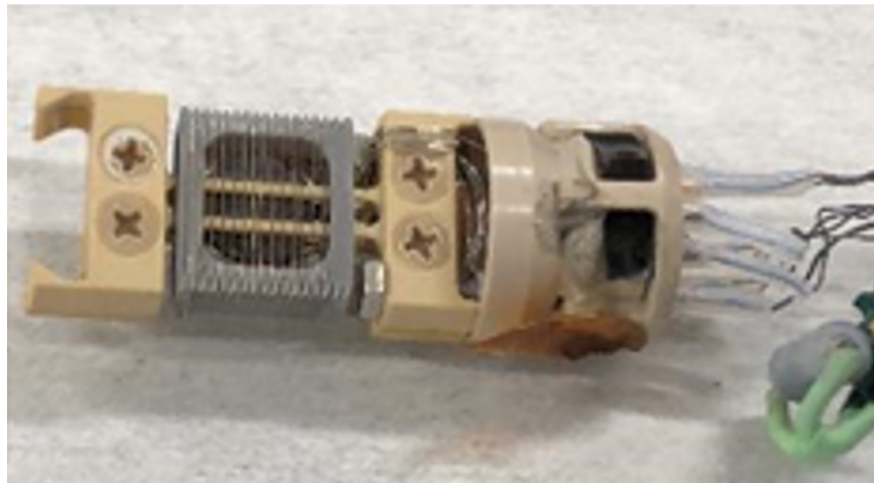


Fig. 7.21: Modified sample retention with 3D printed cage.

then annealed in argon by ramping up to 800 °C in 100 °C increments in order to reduce cracking. We settled on temperatures below the 900 to 1000 °C range due to embrittlement of the samples. Once samples were sintered, they were oxidized by bringing them up to 800 °C, this time in air, and then allowed to cool to room temperature. Mass was measured prior to and following oxidation to approximate the stoichiometry of the samples. In general, we were aiming for approximately 5 %_{at} oxygen, or Pd_{0.95}O_{0.05}. Additionally, a significant color change from gray to green was noted when the samples were oxidized, indicating the protocol's success. This green color is apparent in Fig. 7.20.

The result of the optimized process is shown in a SEM micrograph in Fig. 7.20b,c) and compared to one provided by Storms in his ICCF-23 report in Fig. 7.20d). As we can see, there is generally good agreement in microstructures, and we felt justified that our fabrication protocol was a reasonable replication of what Storms described. Specifically, we noted the length scales of the roughness of the samples were in good agreement, as well as the size of the pores themselves.

Once we optimized the sample fabrication, we proceeded to perform replication experiments in our MS80 differential calorimeter. The first experiment was documented in BE1025. For these replication experiments, we modified our typical sample retention in order to accommodate the large, pellet form factor. An example of this modification is shown in Fig. 7.21, where we show a 3D printed cage that surrounds the cathode and has channels for the wrap-around platinum anode. This ensured that there was no shorting between the electrodes. The electrolyte used was a 0.5 M LiOD solution, and the test was performed near room-temperature at 30 °C. The sample was loaded at a constant current of -100 mA and held for 90 hours. After 90 hours, we explored other currents in order to determine if there was an effect on our ability to produce excess heat. Recall that in differential calorimetry we are applying a nominally identical input power to a thermally connected control cell so that there is zero heat flow in the bridging thermopile. In circumstances where the test cell produces more heat, a non-zero heat flow will be measured by the thermopile. If for whatever reason the control cell is outputting more heat, a negative heat signal is recorded. The former case will result in a coefficient of performance (COP) greater than one, while the latter will result in a COP less than one. In the case of this experiment and follow-up experiments, a platinum cathode in identical heavy

water electrolyte was used as a control.

We can see an overview of the relevant parameters, telemetry, and calorimetry analysis in Fig. 7.22. Each of these plots contains four subplots, which overview the electrochemical (EC) power and current, the cell pressure and relevant temperatures, the measured, expected, unaccounted for heat flow, and COP, as well as the resistance of the palladium cathode. The resistance of the palladium cathode serves as an indicator that the sample is loaded with deuterium and also allowed us to look for any correlations between palladium deuteride stoichiometry and excess heat production (although none was ever observed). It is important to note that the EC power is the measured power, not the set power, as using the set power could lead to gross miscalculations of the expected heat flow in the calorimeter. Additionally, we found it critical to measure the cell pressure in these thermodynamically closed systems, as we would see spurious recombination of oxyhydrogen gas throughout testing which resulted in apparent heat flows. Accurately accounting for the pressure and other state variables allowed us to rule out any anomalous mechanisms.

For the most part, cell pair 3-4 remained quiescent throughout the 160-hour period with two apparent heat flows, one near 90 hours and one towards the end of the test (Fig. A.3). The second of these two events as we understand it, resulted from this spurious and transient recombination in the head space which released chemical energy abruptly into the thermal environment.

More notably, we observed a very large apparent heat flow in cell pair 1-2 around 89 hours into the test, with a measured and briefly sustained differential heat flow of 500 mW and a calculated coefficient of performance of around 2.67. This heat flow eventually decayed to baseline after one to two hours. A zoom-in of this behavior is shown in Fig. (Fig. A.4). Prior to this major heat event, we observed a peculiar ± 100 mW oscillation in differential heat flow that seemed to time correlate with a small change in pressure. However, the delta in pressure did not seem to correspond to what we would expect from recombination of oxyhydrogen gas. Note that the heat flow in the MS80 saturates around 500 mW and so the event likely produced more heat than we measured. While there was an associated 15 psi pressure drop, the calculation we typically use to justify a heat flow as transient recombination returned much less heat than was measured. This was initially a very interesting result, prior to being able to perform postmortem on the samples and the assembly.

It should be noted that postmortem inspections of the test assembly and samples were often critical in understanding test results. Oftentimes our diagnosis of wiring issues, chemical reactions, and component failure was limited while the calorimeter was buttoned up. Following the test, it became very important to carefully inspect various aspects of the assembly and try to rationalize any peculiar behavior that occurred during the test. This proved particularly important for BE1025. Upon disassembly, we uncovered an interesting charring of the PEEK sample retention, which is highlighted in Fig. 7.23. Given the order of magnitude higher enthalpy of reaction for hydrocarbon oxidation (PEEK $\sim 8.9\text{E3}$ kJ/mol) compared to the deuterium oxidation reaction (2.9E2 kJ/mol), and in combination with forensic evidence for charring, we concluded that we observed a classical chemical reaction. While we routinely used PEEK in our experiments, not just this replication, this particularly chemical reaction was quite rare and was not observed during any other of our tests throughout our campaigns. The likely reason behind this lied in the mass of the Pd sample. The Pd samples used in this replication campaign were notably larger than typical wire samples, by approximately 100x. Additionally, they were intentionally oxidized prior to the experiment, and then reduced upon deuterium loading. The result of this is an excessive amount of free oxygen in the head space, which served as a reactant. Beyond this apparent excess heat flow from PEEK oxidation, we did not observe any anomalous results from BE1025.

Storms hypothesized that LENR follows an Arrhenius law, and therefore the amount of excess heat should increase with temperature. In order to test this, we performed 3 more replications at 80 °C. These

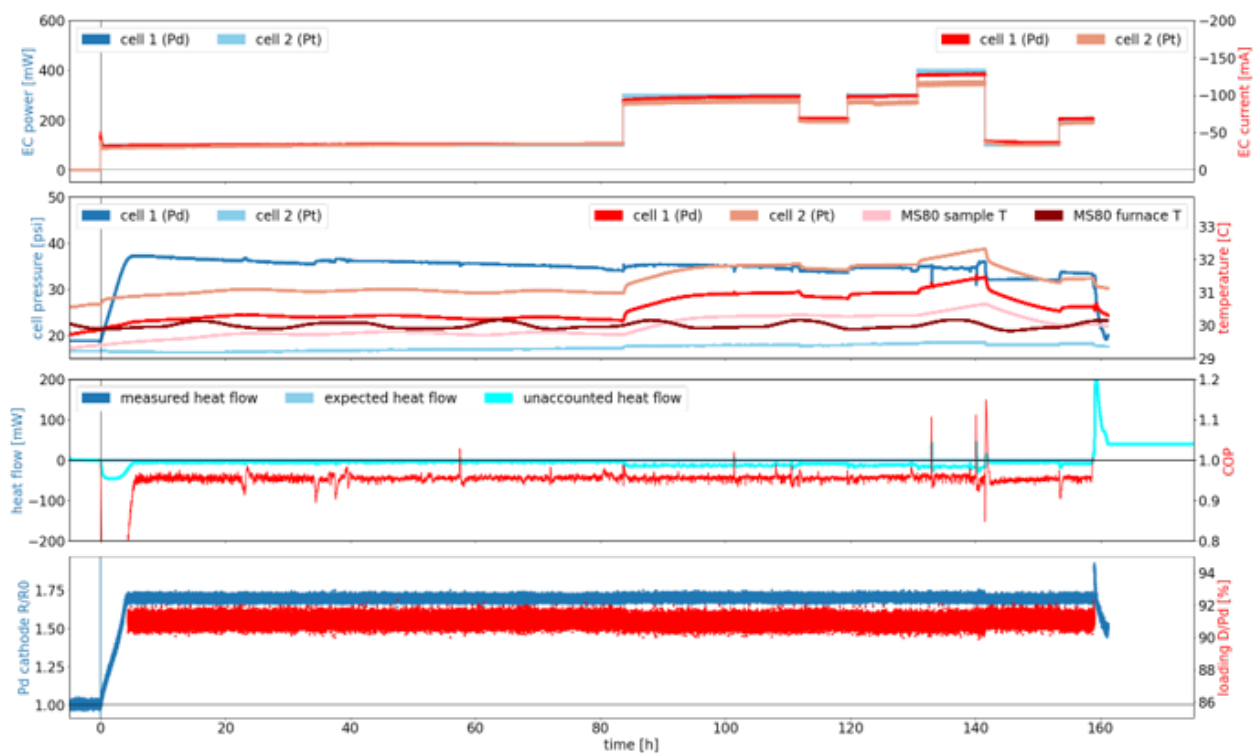


Fig. 7.22: Overview of BE1025 showing experimental parameters, telemetry, and heat flow for cell pair 1,2. Data for a similar experiment is shown in the Appendix (Fig. A.3).

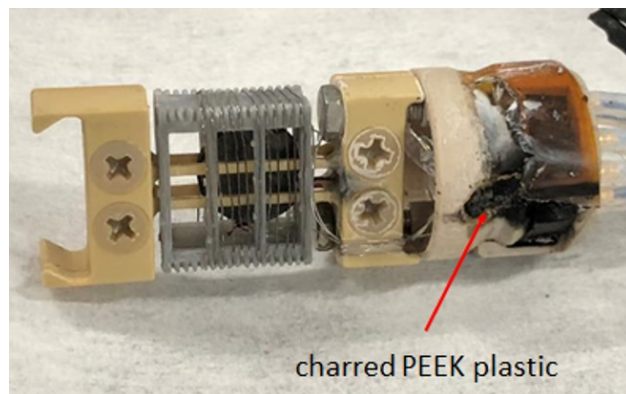


Fig. 7.23: Charred sample assembly from BE1025.

experiments are described in BE1137, BE1199, and BE1226. In all three cases, we did not observe any anomalous heat, although we did observe examples of apparent excess heat caused by classical phenomena. In BE1137 we increased the EC power from 100 mW to 400 mW over 100 hours and then decreased it to 100 mW between 100 and 120 hours (Figs. A.5, A.6). During this period, cell pair 1-2 showed a suppressed heat flow and oscillations that time-correlated with changes in the head space pressure for the Pt control cell. Otherwise, there are no major excursions in heat flow that were observed in cell pair 1-2. Upon disassembly, a leak was noted which would provide a plausible explanation for the suppression in heat flow. When oxyhydrogen gas leaves the system, it can lead to a loss in chemical energy that would normally be present due to deuterium oxidation in the recombiner. The amount of unaccounted heat flow does increase with increasing input power, maximizing at ~ 35 mW and resulting in a COP of around 0.925. Cell pair 3-4 shows better heat flow accounting than pair 1-2. There were no anomalous excursions in measured heat for this test.

The test was repeated in BE1199 (Figs. A.7, A.8). This test did not result in any anomalous heat, although we observed small oscillations in differential heat flow that time correlated with changes in pressure in the platinum control cell. A zoom in on these oscillations is shown in Fig. A.9. From this data, we observed that approximately 250 mpsi fluctuations in pressure resulted in anywhere from 5 mW to 20 mW in measured heat flows. Over time we learned to engineer better recombiners, and at this time we were beginning to understand that water was condensing on the catalytic surface and then evaporating. Eventually, we encapsulated our recombiners in porous PTFE jackets which prevented the condensation of vapor into macroscopic water droplets, ultimately leading to lower failure rates and better overall recombiner performance. Again, however, these improved recombiners were not being used at the time of this replication campaign.

Our final attempt to replicate Storms was documented in BE1226 (Figs. A.10, A.11). In this final test, we observed a fairly large heat flow of 150 mW. However, again it corresponded to a pressure drop in the head space which not only time correlated well with the heat pulse, but also returned the expected amount of energy for hydrogen oxidation. A zoom-in of this event is shown in Fig. A.12. In this plot, we see the heat flow in cell pair 3,4 plotted against both the head space pressure and temperature. A roughly 12 psi pressure drop occurred over a one-minute time frame. A 150 mW spike in measured heat was observed, as well as an increase in the head space temperature by 200 mK. Using a simple ideal gas model, we can calculate the moles

of oxyhydrogen recombined and convert the pressure drop to total energy released. Comparing this value to the integrated area under the heat flow curve, we get one-to-one correspondence. This formalism allowed us to rule out any anomalous sources of heat and attribute this particularly large spike to the deuterium oxidation reaction.

Two years following this investigation, we received samples from Storms himself. These samples, he claimed, were nuclear active and should give us the result that we were looking for. After analyzing and running these samples extensively in our HTCal calorimeter, we never observed any excess heat. There was no commentary provided by Storms as to why we did not see heat with the samples he claimed would give us the result. At the conclusion of our investigations, we remain unconvinced that his results were robust and repeatable in any capacity.

7.5.3 Conclusions

In all our attempts to replicate Storms' samples and apply similar electrochemical conditions, we did not observe any excess heat. We did observe conventional chemical reactions which resulted in major heat flow transients. These, however, were unlike the observations made by Storms using his Seebeck calorimeter. It is unfortunately inconclusive that either Storms made some unwitting mistake in his calorimetry (possible calibration drift or other user errors) or he accidentally stumbled on a working recipe for LENR but could not provide sufficient details for replication.

We would like to note that Storm's report was not published in a reputable peer-reviewed journal. We highlight that he did not provide proper control experiments. To the contrary, he reported that light water experiments also produced excess heat [113]. Furthermore, it is suspicious that he observed excess heat only at elevated temperatures (obtained by heating the calorimeter). While he points to an Arrhenius behavior, we consider it likely that a miscalibration of his calorimeter resulted in apparent excess heat at elevated temperatures and no excess heat at ambient temperature. Finally, he did not use a strictly closed system and the reported excess powers are small compared to the overall input power (including the heater power).

7.6 Miles

7.6.1 Literature Review

Shortly after the 1989 announcement of cold fusion by Martin Fleischmann and Stanley Pons, Melvin Miles began his experiments at the China Lake Naval Weapons Center. He aimed to replicate their findings of excess heat, focusing on improved material preparation, more precise calorimetry, and nuclear product detection. In his 1994 paper 'Calorimetric Principles and Problems in Measurements of Excess Power during Pd-D₂O Electrolysis' Miles described the shortcomings and challenges associated with isoperibolic calorimeters used in electrochemical experiments [260]. He highlighted issues arising from changes in electrolyte levels and heat transport pathways, arguing that many reported experiments assumed steady-state conditions and failed to account for all terms in the differential equation governing calorimetry, leading to significant errors. According to Miles, accurate measurements required careful control of external conditions and consideration of time-dependent factors. Controversially, he claimed that early detractors of the LENR effect made faulty calorimetry measurements, rather than accepting their null results. He re-analyzed a study by Lewis et al., claiming that interpreting the beginning stages of PdD loading as a calibration would suggest around 75 mW of excess heat from their experiment after reaching steady state [261].

Miles' interpretation is weakly supported due to the questionable use of a PdD sample as a calibration standard. A more robust approach is to use either a platinum cathode in heavy water, a palladium cathode in light water, or preferably both, to characterize the thermal response of the isoperibolic calorimeter. Relying on a PdD sample as a control raises a critical question: how can one ensure there is no excess heat during the initial calibration? While discrepancies between input power and measured heat flow may emerge later in a test – an intriguing result – such findings would still require protium and platinum controls for validation. Callisto addressed this issue by employing isotopic contrast and/or platinum controls in our experiments and apparatus characterizations to enhance confidence in any observed excess heat. While simplifying assumptions are often necessary in scientific analysis, they carry risks.

In the same year, Miles, along with Bush and Lagowski, reported in the paper 'Anomalous Effects Involving Excess Power, Radiation, and Helium Production During D₂O Electrolysis Using Palladium Cathodes' observations of excess power, helium production, and anomalous radiation [262]. They claimed measurable helium was detected in gas samples during excess power production episodes, with control samples showing no helium. They established a minimum helium detection limit of 6E10 atoms/ml and reported helium production rates ranging from 10E11 to 10E12 atoms per second per watt of excess power, consistent with typical fusion reactions. Despite occasionally recording high radiation counts correlating with excess power and helium production, many experiments yielded normal radiation counts with no significant excess power. Measuring helium-4 above background levels in a mixture predominantly containing gaseous deuterium was acknowledged to be challenging. In the 1996 report 'Anomalous Effects in Deuterated Systems' Miles and his co-authors discussed further observations of excess power, helium-4 production, and anomalous radiation during D₂O electrolysis using palladium cathodes [263]. They reported excess power in 28 out of 94 experiments, primarily using palladium-boron cathodes. The rationale for using the alloyed cathodes was that the interstitial boron improved the mechanical properties and removed oxygen impurities from the cathode, mitigating electrode cracking during the charging and discharging of deuterium into and out of the lattice and reducing volumetric expansion. Although there is no known mechanism for LENR, the authors believed these factors were critical for observing the effect. Despite these claims, significant reproducibility issues were encountered, attributed to unknown variables in the palladium metal. They continued to report anomalous helium-4 production rates. However, these findings were met with skepticism due to potential experimental errors, including atmospheric helium contamination and calorimetric inaccuracies. The lack of reproducibility continued to challenge the acceptance of these anomalous effects within the broader scientific community.

In the study 'Calorimetric Studies of Pd/D₂O+LiOD Electrolysis Cells' published in 2000, Miles conducted experiments using an improved isoperibolic calorimeter to investigate excess power production during Pd/D₂O+LiOD electrolysis [264]. Excess power was reported in one cell, ranging from 50 to 100 mW, despite a gradually decreasing input power, while a second cell exhibited normal behavior. The excess power effect was identified by an anomalous increase in cell temperature, with two thermistors in each cell showing nearly identical temperature changes. Various chemical processes were considered to explain the excess power, but none satisfactorily accounted for the observations (according to the authors), and Faradaic efficiencies were assumed to be near unity to rule out recombination effects of the electrolysis gases, which is a questionable assumption based on Callisto's own measurements. Certainly, at high current densities the Faradaic efficiency is close to one, but this is not necessarily the case at lower currents. Additionally, over time the palladium surface becomes fouled with other elements which can also contribute to a drop in Faradaic efficiency. Despite the claims made in these reports, the gradual onset of excess power and inconsistent reproducibility highlighted ongoing challenges and skepticism of Miles' work. The claims of nuclear reactions required extraordinary evidence, which these studies did not conclusively provide.

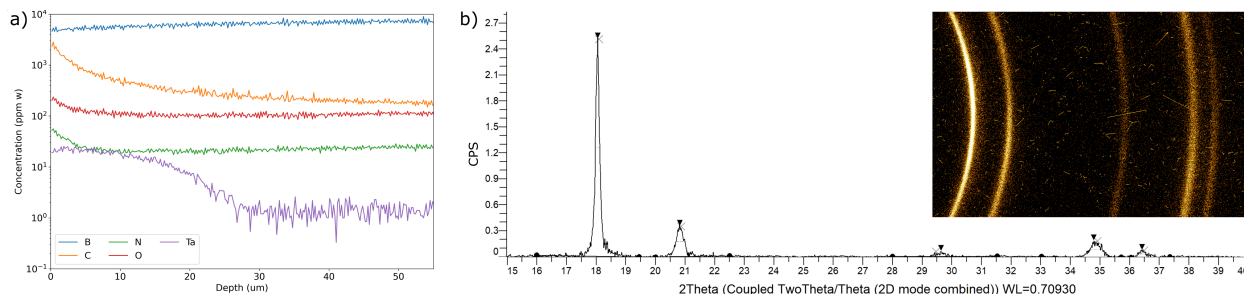


Fig. 7.24: a) Elements with composition gradient by GDMS. b) XRD: Diffraction intensity in CPS (counts per second) vs. diffraction angle 2θ . The inset shows the 2D diffractogram.

7.6.2 Replication Experiments

The first step in our replication was to reproduce the alloyed cathodes as described by Miles and his co-authors. In their investigations, they claimed that boron compositions between 0.50 and 0.75 wt% were required to observe the anomalous heat effect under electrochemical conditions. As such, we worked with ACI Alloys to fabricate PdB cathodes with the relevant compositions. Rods with a length of 30 mm and diameters of 3 and 6 mm were fabricated, however, we found that the 6 mm rods produced too much orphaned oxygen during the loading process. As a result, the 6 mm diameter rods resulted in over-pressurization of our HTCAl vessels and only the 3 mm rods realized completed replication tests. Nevertheless, we independently confirmed the composition of the cathodes using depth profiling glow discharge mass spectrometry (GDMS) and x-ray diffraction, shown in figures 7.24. We noted a few other impurities in the alloy, as indicated by the GDMS depth profile, including nitrogen, carbon, oxygen and tantalum. These impurities seemed to be concentrated at the first 20 microns of the surface, but ultimately they were at very low absolute concentrations in the bulk. While GDMS provides a direct composition measurement, x-ray diffraction (XRD) allowed us to look at the lattice expansion caused by interstitial boron. According to the literature, we can calculate the fraction of boron in the lattice as a function of the lattice parameter:

$$x_B = \frac{a - 0.3892}{0.06882} \quad (7.1)$$

where x_B is the fraction of boron and a is the lattice parameter measured by XRD [265]. Both methods give good agreement, with a measured alloy composition of Pd_{0.9484}B_{0.0516} or ~0.55 wt.% B. These results indicated that we satisfactorily replicated the reported cathode composition and structure.

In the initial replication experiment BE2280, we tested three PdB cathodes and one Pt cathode as a control. In all cases, 44 mL of 0.1 M LiOD was used as the electrolyte, and a Pt wire was used as an anode. The construction of a typical sample holder is shown in figure 7.25a). As discussed in earlier chapters, important features of the holder include the mechanical clamping of the sample to achieve reliable electrical contact, a platinized carbon recombiner, a pressure-sensor (not shown), and an RTD. The electrochemical conditions, telemetry, and calorimetric analysis are shown in figure 7.26. Over a 120-hour period, the EC power was increased to a maximum of 1 W and then decreased back to 0 W. During this initial test, we observed a detonation in cell 3, which resulted in the catastrophic failure of the calorimeter channel and adversely affected the integrity of the calorimetry in adjacent channels. This detonation can be seen around 80 hours into the test, where the pressure builds rapidly, and the EC power in cell 3 suddenly drops to

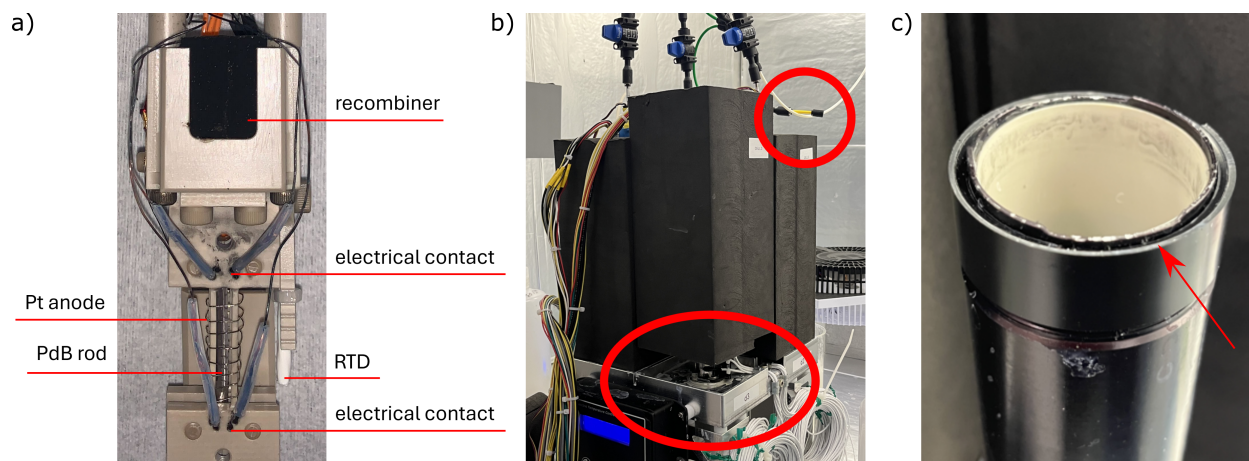


Fig. 7.25: a) HTcal sample holder with typical sample used in Miles replication experiments. b) Four-cell HTcal calorimeter with foam insulation lifted from cap failure (large red marking) and detached backpress line (small red marking). c) Top of the electrolytic vessel with remainder of plastic retaining ring, which used to hold down the top cap, sealing off the vessel. The fracture surface is indicated with a red arrow.

zero, accompanied by an abrupt drop in the electrolyte and thermal enclosure temperatures. A zoom-in on this event is shown in figure 7.27. Upon inspection of the setup following the completion of the test, catastrophic failure of the pressure vessel was observed, as shown in figure 7.25b), d). Here, we see the cap to cell 3 was ripped off and ejected. The back-pressure line supplying gas to the check-valve was dangling inside the enclosure and directing airflow at channel 2 of the HTCal cluster. Additionally, the foam on cell 3 was raised, exposing the baseplate to the thermal environment. With the termination of power in cell 3 and its exposed baseplate, the cross-talk to the adjacent cells is also expected to be impacted. As a result of these combined effects, the heat transfer of the adjacent channel was fundamentally altered, resulting in an apparent suppression of the measured heat flow and calculated coefficient of performance (COP) in that cell. Although this failure mode was not arrived at intentionally, it does provide a strong illustrative example of how a change to the heat transfer environment can result in significant calorimetric errors.

Otherwise, no excess heat was observed in this initial replication experiment. As discussed in the previous chapter on the HTCal, spikes upward and downward in COP are observed whenever the EC power is changed. This mathematical artifact is due to the contrast between the finite thermal time response of the calorimeter vs. the instantaneous change in EC input power. Later in our research program, we implemented a convolution function which uses the calorimeter's instrument response function to better account for these transients. However, this methodology was not implemented at the time of this experiment.

As part of our program's rigor, we consistently inspected the samples with electron microscopy following tests to look for additional information, whether to improve the experimental details, understand a failure mechanism more fully, or to look for any physical manifestations of excess heat production, e.g., sample melting. An example of the SEM and EDS results is shown in figure 7.28. Notably, the samples are all covered by a layer of copper, which is a result of the corrosion of exposed contacts inside the calorimeter. Although contacts were covered with shrink-tubing and Microstop lacquer, over long durations they become compromised and leach impurities into solution. Given routine observation of impurities on the cathode

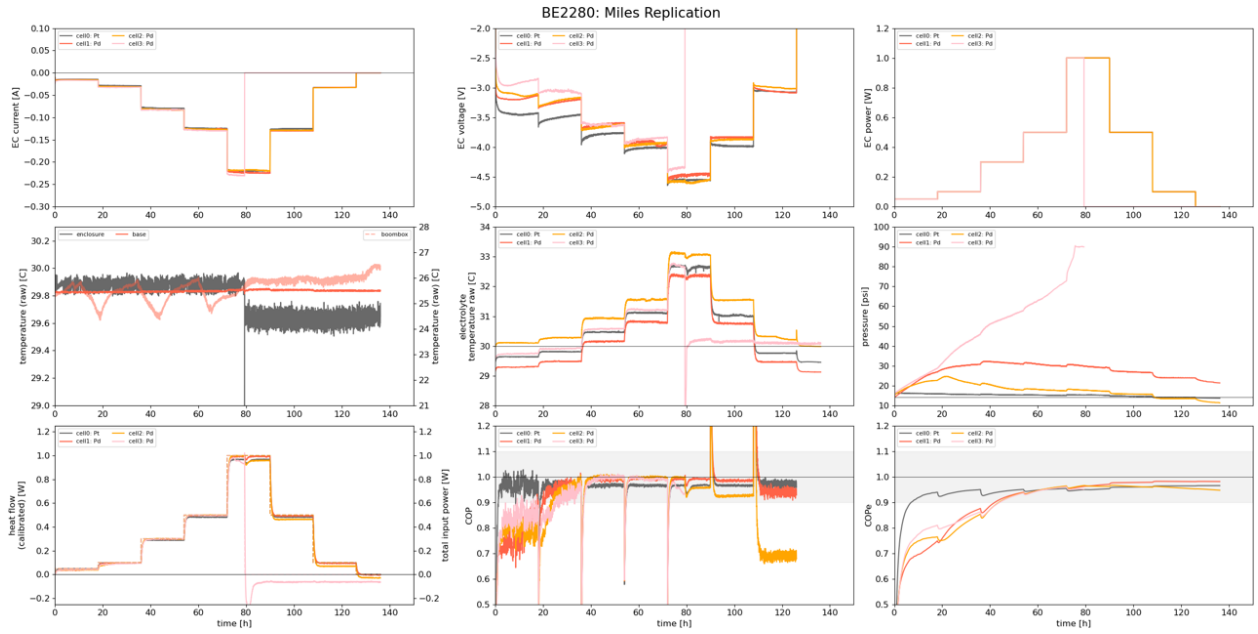


Fig. 7.26: Overview of electrochemical conditions, telemetry, and heat flow analysis for BE2280.

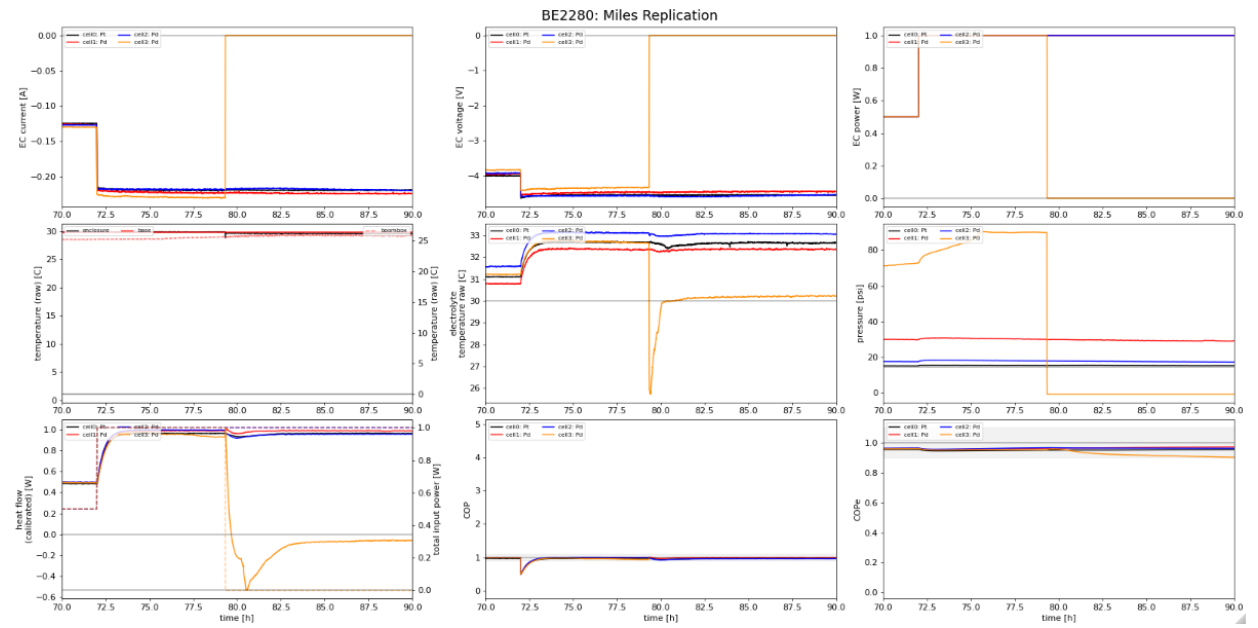


Fig. 7.27: Zoom in on detonation.

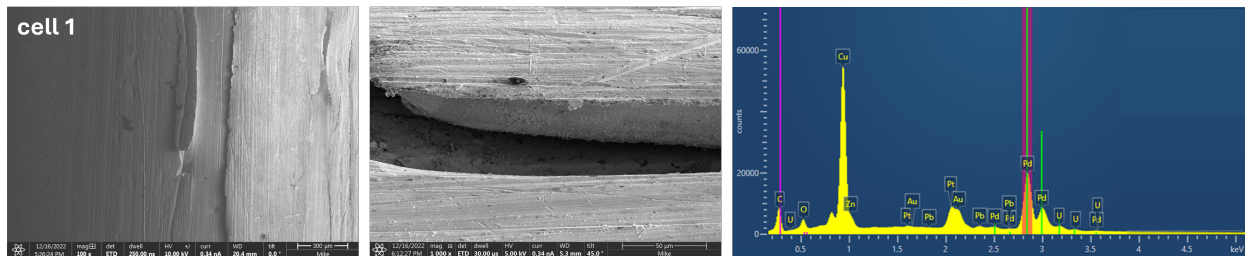


Fig. 7.28: SEM and EDS analysis of post-experiment PdB cathode.

surface after long-duration tests, in this replication campaign and others, we concluded that it is incredibly challenging to maintain a high purity surface for long periods of time. Electrode fouling is a commonly observed phenomena in non-LENR electrolysis experiments and is fairly well documented. Another notable observation was the prevalence of cracking in the cathodes, contradicting Miles' claims about the mechanical integrity of PdB alloys. These cracks were typically longitudinal, extending along the long axis of the rod, and could have an opening width of many microns.

The Miles experiments were replicated several more times in BE2280, BE2469, BE2634, BE2918, BE3067, and BE3152, which are summarized in figure 7.1. In some cases, various failure modes and false positives were observed. In these experiments, failure modes and false positives were primarily associated with inconsistent recombiner performance. Whenever recombiner efficiency degrades and then recovers abruptly, this leads to a sharp decrease in the calorimeter's headspace pressure. This delta in pressure leads to a substantial heat burst, which is registered in the electrolyte temperature and COP analysis. An illustrative example of this failure as it manifested in the Miles experiments is shown in figure 7.29. Here, we see that a sharp drop in pressure correlated in time with a rise in measured heat flow. Assuming an ideal gas, the amount of energy calculated from the pressure drop corresponds very well to the heat measured by the calorimeter.

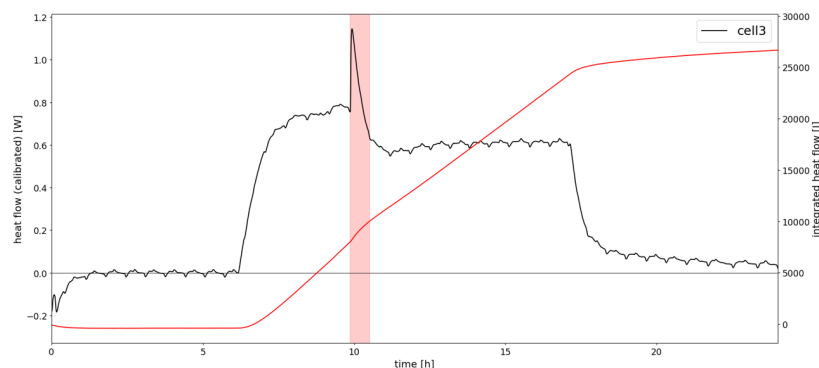


Fig. 7.29: Gaseous oxyhydrogen recombination resulting in a heat burst.

Experiment ID	PdB samples tested	Test Status	Excess Heat	Reason for Elevated or Suppressed Excess Heat
BE2280	3	Completed	Yes	Detonation
BE2469	3	Contact failure	N	Not applicable
BE2634	3	Completed	Yes	Recombination
BE2918	3	Completed	No	Not applicable
BE3067	3	Completed	Yes	Recombination
BE3152	3	Completed	No	Not Applicable

Table 7.1: Overview of Miles replication experiments.

7.6.3 Conclusions

Although we observed apparent excess heat in our investigations of PdB alloys, we were always able to ascribe these to classical phenomena. Our replication of the experimental details provided by Melvin Miles failed to produce any anomalous heat. We would like to highlight that Miles' report lacks light water control experiments.

7.7 Navy Codeposition

7.7.1 Literature Review

In response to the excitement generated by Fleischmann and Pons in 1989, a team from the Naval Laboratory in San Diego developed the Pd/D codeposition process to replicate observations of excess heat. Led by Szpak and Mosier-Boss, the Space and Naval Warfare (SPAWAR) team sought to improve Fleischmann and Pons' methods by increasing deuterium loading in the palladium lattice. Their approach used a solution of palladium chloride (PdCl_2) and lithium chloride (LiCl) in deuterated water (D_2O) to deposit palladium onto a working electrode. They claimed that this process rapidly achieved high deuterium loadings, with simultaneous reduction of palladium salt and hydrogen evolution. Various substrates, including copper (Cu), gold (Au), platinum (Pt), and nickel (Ni), were used to assess the influence of different materials on the co-deposited palladium.

Throughout the 1990s, SPAWAR continued exploring the co-deposition method [266]. Using Dewar-type calorimeters, they reported cases where Pd/D codeposited electrodes appeared to generate more heat than expected from the electrical input. Infrared imaging indicated this excess heat was localized to specific points on the electrode surface. Additionally, they observed phenomena such as positive feedback and "heat-after-death", where heat production persisted after the electrical input ceased. By the late 1990s, research shifted from calorimetry to identifying potential nuclear byproducts [266]. The team employed CR-39 solid-state nuclear track detectors, which reveal tracks from high-energy particles after chemical etching. Based on track morphology, they reported possible signs of alpha particles, protons, and neutrons.

Surface analysis via SEM revealed complex features, such as pseudo-fractal patterns and micro-volcanoes, resulting from Pd/D co-deposition. These formations can be explained by the hydrogen evolution reaction (HER) during electrodeposition, where hydrogen gas bubbles disrupt mass diffusion, causing material buildup around the bubble perimeter. SPAWAR also experimented with external electric and magnetic fields during

co-deposition, which significantly altered the electrode's surface morphology. The authors speculated that these fields might affect the palladium deuteride system.

Szpak's team also collaborated with organizations like Energetics Technologies and researchers such as Dr. Mitchell Swartz to refine the co-deposition process. They focused on optimizing reaction rates and control through methods like Superwave charging (see section 7.8), as well as studying the effects of chemical additives and varied cathode materials. Despite over two decades of experimentation, the Pd/D co-deposition method has not produced irrefutable evidence of LENR, and the results must be interpreted with caution.

There are some weaknesses in the claims of the co-deposition work that we observed. Among these, the micro-volcanoes on the Pd surface observed by SPAWAR due to hydrogen gas bubbles building up and escaping to the surface were also observed in our work. These crater-like structures have been observed in our own control experiments with protium as well as deuterium, suggesting no link to nuclear activity. The simplicity of this explanation seems to have been overlooked by the SPAWAR team. As stated previously, the SPAWAR team speculated that external electric and magnetic fields during co-deposition may have affected the palladium deuteride system. However, classical physics, specifically magnetohydrodynamic (MHD) effects, provides a well-established explanation. Magnetic fields influence ion motion, affecting both heat transfer (via MHD convection) and the electric double layer (EDL) near the electrode surface. These effects alter the structure and dynamics of the EDL, impacting electrochemical reactions (i.e., electro-reduction and HER), but the SPAWAR team did not fully account for these classical phenomena in the interpretation of their results.

Most recently, research by the Naval Surface Warfare Center at Indian Head led by Oliver Barham, built on SPAWAR's work, aimed to resolve lingering questions in LENR. Published in APL-Energy in 2024 [267], Barham's team reported thermal anomalies in some of their 33 experiments, where energy output exceeded input at statistically significant levels (up to 5 W of excess output power). These experiments were carried out with 0.25 mm Pt electrodes (anodes and cathodes) in D₂O with DC current application of up to 900 mA in the presence of chlorine (Cl) based salts of lithium (Li), palladium (Pd), and/or rhodium (Rh) metals. The experimental values were monitored using temperature sensors, solid state nuclear track detectors (CR-39), and radio frequency (RF) sensors. The calorimeter employed Seebeck sensors tiled around all sides of the experimental vessel to measure a voltage difference created from a temperature gradient across the sensors, which with proper calibration is ultimately translated into a heat flow. A photograph of our experimental vessel based upon Barham's design can be seen in Figure 7.32a).

The findings of Barham's experiments do not conclusively indicate nuclear reactions, as alternative explanations like measurement errors or chemical effects remain viable. Barham's detection of radio-frequency emissions and particle tracks using CR-39 detectors mirrors earlier reports of nuclear byproducts but does not confirm nuclear reactions. False signals from chemical origins and inadequate background testing often complicate results with these detectors. Despite their low cost, CR-39 detectors are poorly suited to such experiments, though more reliable solid-state detectors have emerged over the past two decades. Critically, CR-39 (composed of polyallyl diglycol carbonate) is known to be unstable in alkaline environments. KOH and NaOH are routinely used to etch it [268]. Likely, similar effects occur when CR-39 is exposed to corrosive electrolyte. In particular, the hydroxide concentration is expected to be elevated near the cathode, which could explain the differences in CR-39 etching between the electrodes observed by Barham. The team also monitored for evidence of tritium production, but found none (such claims have been widely disputed in the past). In our view, the lack of proper protium controls significantly weakens Barham's claims of excess heat production. In fact, he reported excess heat in platinum control cells, but ascribed it to palladium contamination.

Most notably, the reported excess heat strongly correlated with the anode temperature (see [106] Sup-

plemental Information). Repeatedly, elevated anode temperatures occurred during periods of excess heat (runs #19, #21 to #31) and suppressed anode temperatures occurred during periods of suppressed heat (run # 15). If the temperature difference of almost 10 K within the electrolyte is real, it means that the heat source is the anode, not the cathode. Suspiciously, in two other experiments (runs #12, #15), the electrolyte temperature dropped by ~20 K while the input power was constant (or even increasing). This clearly calls into question the reliability of their calorimetry.

In the experiment with the highest excess heat (run #19), the excess heat is fairly constant at 5 W from the beginning of the experiment, as evidenced by the quick rise in temperature at 0 h. This is curious, as palladium is expected to deposit on the cathode over time, forming a coating, which then gets hydrided and potentially serves as the host lattice for the hypothesized nuclear reactions. For that reason, one would not expect constant excess heat from the very beginning.

Although Callisto was skeptical of the co-deposition body of work, the experiments contained enough detail for replication. Callisto has conducted two iterations of replication experiments covering more than 100 individual experiments at the time of this report – one set replicating the experiments of the SPAWAR work led by Mosier-Boss, and another set replicating the recent experiments from Barham. The outcomes of these replication attempts are described below.

7.7.2 Boss Replication

Callisto performed 13 experiments to replicate the work of Mosier Boss, based on a patent filed. To briefly summarize experimental conditions: 0.25 mm diameter Pt electrodes were placed in 0.03 - 3 M PdCl₂ in D₂O with a magnetic field being applied to the vessel by two magnets fixtured to the external walls. The following current profile was applied for the indicated time: 1 mA for 2 hours, 2 mA for 6 hours, 5 mA for 24 hours, 10 mA for 24 hours, 25 mA for 24 hours, 50 mA for 24 hours, 75 mA for 24 hours, 100 mA for 24 hours. Boss et al. reported a reaction that produced excess heat (enough to form melt spots on the Pd cathode) and nuclear emission of high energy particles, suggesting LENR. Several of our experiments were exact replication attempts based on the Navy patent [269], while others aimed to deepen understanding of the system, including modifications for improved conditions and calorimetry. Notably, we modified our HTCAl setup to fixture N55 magnets, resulting in magnetic fields of 100 to 200 mT inside the cell.

7.7.2.1 Experimental Results

We initially tested palladium thin films (100 nm) in 0.5 M LiCl in D₂O and observed corrosion of the palladium. This was concluded to be an issue for thin films but likely not for bulk palladium cathodes. We then tested arsenic enhancement of Pd wire cathodes in 0.5 M LiCl in D₂O. However, this chemical enhancement strategy was incompatible with the electrolyte, possibly due to the corrosive properties counteracting the formation of the surface Pd-As layer. The resistance ratio is shown in Figure 7.30a), showing no enhanced loading. Pd loading during and after Pd electrodeposition on a Pt wire was measured using 4-point resistance measurements (Fig. 7.30b). An initial drop in resistance likely corresponded with wire diameter growth from electroplating. Following that, when the EC voltage exceeded water-splitting potentials, resistance began to increase again. Electroreduction and HER did not appear to occur simultaneously, challenging the co-deposition framework. The Pd shell loadings did not exceed 75 to 80 % (based on 4-point resistance measurement of the entire sample, subtracting the contribution of the core), lower than SPAWAR's claimed stoichiometric loadings.

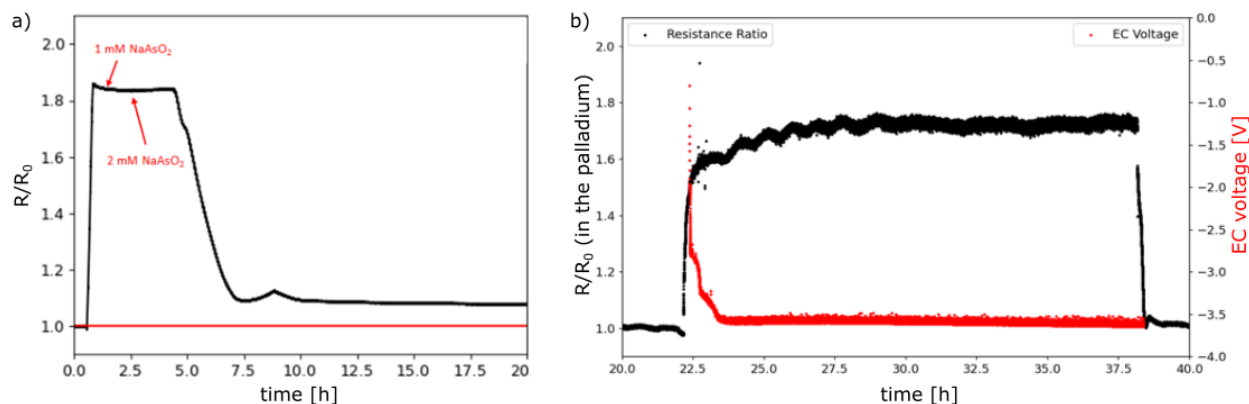


Fig. 7.30: a) Resistance ratio during arsenic-enhanced Pd wire cathode in LiCl electrolyte. Arsenic dosings are indicated by arrows. No loading enhancement is observed. b) Estimated Pd resistance ratio during codeposition of Pt and loading of Pd core.

In the initial closed HTCAl experiments (BE2152, BE2368), the generation of chlorine gas during electrolysis compromised our platinized carbon recombiners, leading to vessel pressurization. This prompted us to improve our recombiner design for the rest of the closed-system calorimetry experiments. Using an improved recombiner design, we encapsulated the platinized carbon paper in a PTFE porous membrane to prevent condensation of corrosive fluids. Despite this improvement, over-pressurization occurred at high current densities due to non-recombinable chlorine gas. The heat flow analysis showed suppressed heat flow at high currents, requiring a major correction to the analysis. The heat balance of the system was indeterminate due to the inability to quantify the relative amounts of chlorine and hydrogen evolution.

In the final replication experiments, we modified the electrolyte from 0.3 M LiCl to 0.3 M LiOD. We hypothesized that this would avoid the formation of intermediate salts such as LiOCl and LiOH/D, which may have influenced prior results. Although clean test results were obtained, no excess heat was observed in these experiments. Postmortem SEM inspections of samples from this replication are shown in Figure 7.31. We replicated crater-like formations and nanostructuring of the surface (Figure 7.31), but the lack of isotopic contrast between protonated and deuterated electrolytes suggests these features are non-nuclear in origin. Blister formations with nanoscopic voids were observed (Figure 7.31 d) and e), likely caused by gas bubble nucleation within lattice defects, and again were found present in systems loaded with either hydrogen isotope (H or D).

Given the lack of coherent scientific evidence in the literature and our experimental results, including minimal simultaneous electroreduction and HER, moderate Pd loadings, and the absence of excess heat, we concluded there was little merit in pursuing further replication of the Boss experiments.

7.7.3 Barham Replication

Callisto performed well over 50 Barham and Barham-like experiments where we either exactly or closely replicated the experimental conditions that were reported to show excess heat [106]. To achieve results similar to Barham we designed a custom experimental vessel tiled with Seebeck sensors (Fig. 7.32a), closely matching the dimensions of the calorimeter reported by Barham. Our standard experimental setup consisted of two 0.5

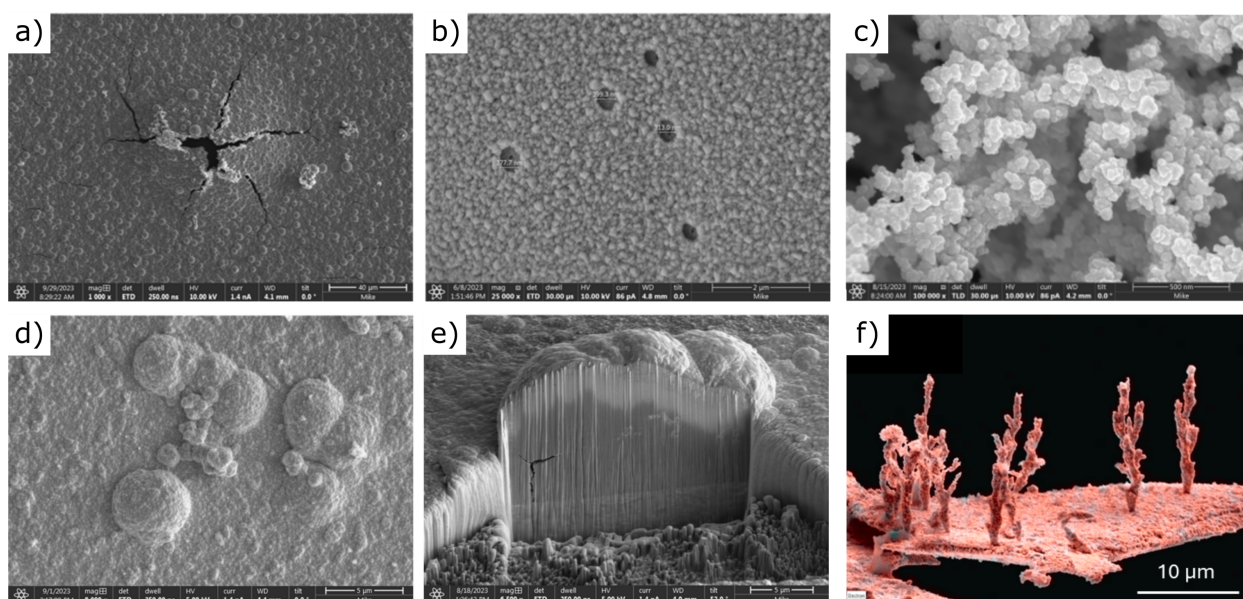
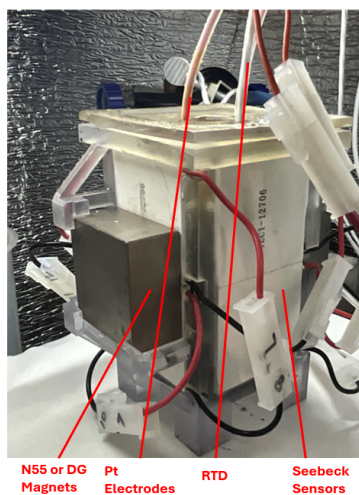


Fig. 7.31: SEM images depicting a) volcano-like (scale bar 40 μm) and b) crater formations (scale bar 2 μm) on palladium cathode surfaces. c) High magnification (scale bar 500 nm). d) Blister formations with subterranean nanoscopic voids (scale bar 5 μm) and e) a representative blister sample which was cut with focused ion beam (FIB) to expose the cross-section (scale bar 5 μm). f) EDS false color map of dendritic growth (red: Pd, blue: Cl).

a)



b)

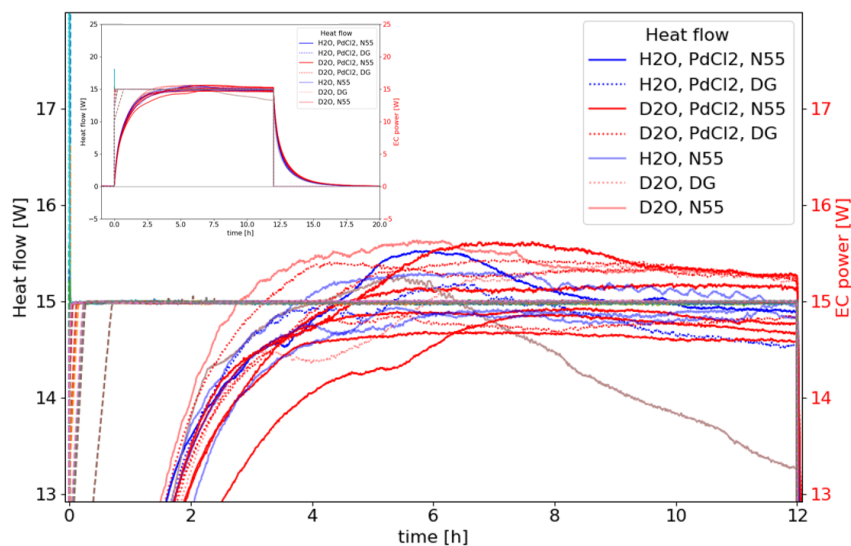


Fig. 7.32: a) Photograph of the experimental vessel used in our Barham replication experiments with Seebeck sensors tiled around the vessel and other components labeled. b) Shows a summary figure of the results of the Barham and Barham-like replication experiments (solid red and dotted red traces) as well as various control experiments. Input power of 15 W are shown in the dashed traces. There was one outlier experiment (brown trace) that had excessively suppressed heat flows due to the vessel lid being ajar halfway through the experimental run. The smaller inset shows the full heat flow data showing the heat flow of all experiments returns to baseline.

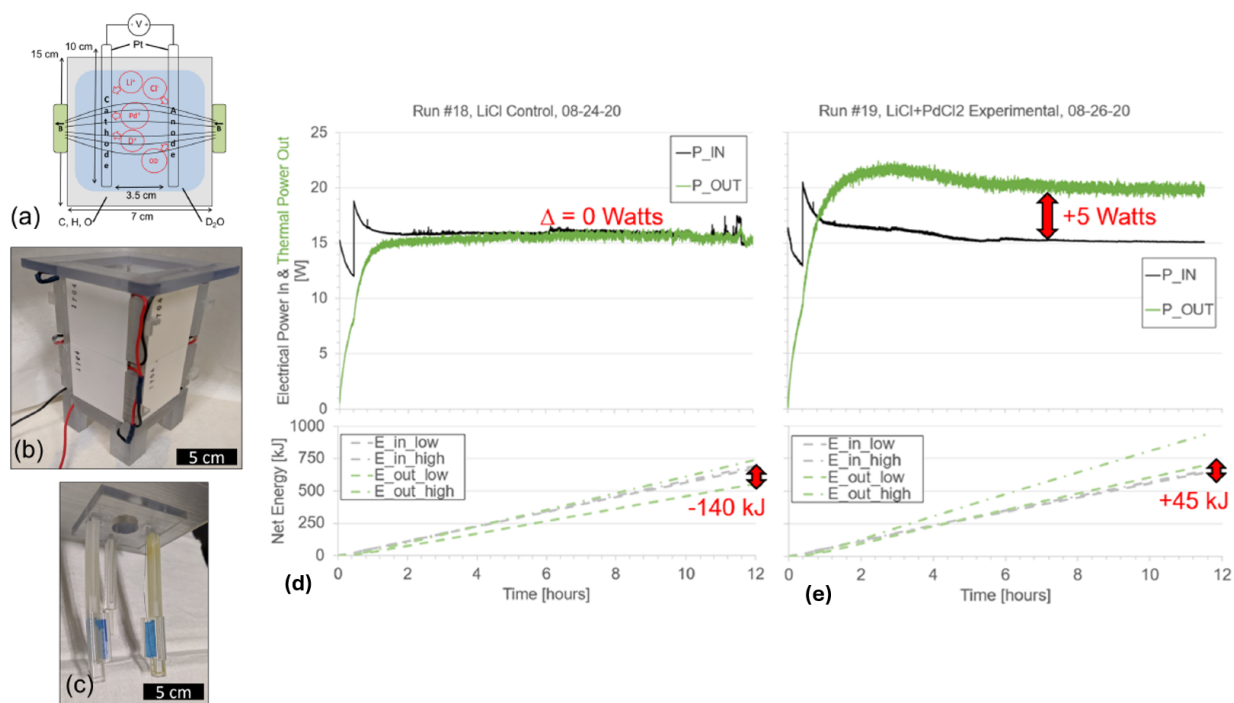


Fig. 7.33: a) Shows a 2D mock up of the Barham vessel with its dimensions and components, b) shows the Barham open-cell calorimeter with Seebeck sensors used in their experiments, and c) shows the lid of the calorimeter with the RTDs placed in sleeves. d) Shows the heat flow and net energy results of the control experiment (#18) run by Barham with Pt electrodes and 150 mM LiCl in D_2O . There is no excess heat reported in the control experiment. e) Shows the heat flow and net energy results of the experiment (#19) run under the exact conditions as experiment #18 but with 1.25 mM $PdCl_2$ added. The figure reports a nominal 5 W of excess heat, although after the authors subtracted their estimated "6-sigma" noise margin, they are effectively claiming only about 1 W (45 kJ over 12 hours) of statistically significant excess heat based on their 6-sigma requirement.

mm diameter and 30 mm long Pt electrodes laser welded to copper wire and housed in shrink-tube shielding to protect the connections from corrosion from the electrolyte. The electrolyte was typically 150 mM LiCl in D₂O (H₂O for control experiments) with 1.25 mM PdCl₂ and/or 0.68 mM RhCl₃ additives. We applied powers of 15 W for 12 hours based on data shown for the main positive result of excess power (experiment 19) reported by Barham (Figure 7.33)e. We specifically focused on experiment 19 which purportedly exhibited 5 W of excess heat compared to the input power [267]. This experiment had Pt electrodes (anode and cathode), an electrolyte containing a LiCl salt in D₂O creating a 150 mM solution, and a PdCl₂ additive that ends in 1.25 mM concentration in the solution. Barham also added a RhCl₃ additive in addition to the PdCl₂ in nearly half of the reported experiments, which we also replicated in our work. In some of the Barham-like experiments we also exchanged the N55 magnets for degaussed (DG) magnets, to probe the effects of magnetic field.

7.7.3.1 Experimental Results

The summary results of the Barham and Barham-like replications are shown in Figure 7.32b. These experiments used a constant 15 W of power applied across the Pt electrodes (anode and cathode) in 150 mL of an 150 mM LiCl electrolyte made with D₂O and 1.25 mM PdCl₂ additive. N55 magnets were interchanged for DG magnets in some of the Barham-like experiments. Other control experiments (i. e., H₂O, no PdCl₂ additive) are also shown in this plot. Overall, no anomalous heat events were observed in any of our direct replications as well as our Barham-like replications. Our control experiments showed a maximum of 15.5 W of output power on 15 W of input power and thus that became our minimum limit to indicate an anomalous event. No experiments showed heat flow beyond ± 0.5 W except for an outlier (Fig. 7.32b, brown trace, BE4477) where the vessel lid came ajar during the test and led to a severely suppressed heat flow. One observation that we noticed during this experimental campaign is that electrolyte composition seemed to change between experiments and even when it was refilled with fresh electrolyte, it exhibited suppressed heat flow behavior compared to a completely fresh electrolyte. This is likely due to the depletion of the electrolyte and precipitation of lithium salts during electrolysis.

Furthermore, we monitored the loss of electrolyte by placing the Seebeck calorimeter containing the electrolytic cell onto a scale during experiments. We continuously recorded the mass and typically observed an electrolyte loss of ~ 30 ml over 12 h at 15 W. It is curious that Barham et al. did not discuss this issue despite their longer durations of typically 40 h. Likely, the calorimeter calibration is sensitive to the electrolyte level. Additionally, any open system calorimeter calibration is not only sensitive to the input power, but also to the electrochemical current, which determines the chemical heat leaving the system in the form of oxyhydrogen gas. This introduces additional error in the heat flow calculation in runs with varying input power despite constant current (runs #5, #15, #17, #28).

7.7.4 Conclusions

In almost fifty Barham replication experiments, we have been unable to reproduce the reported excess heat of 5 W at 15 W input power (or any excess heat beyond the error of the control experiments). While Barham's paper [106] is one of the few papers published in a mainstream peer-reviewed journal, we point out various issues. Most importantly, all reports of excess heat show an elevated temperature near the anode, suggesting the anode as the heat source (as opposed to the palladium deuteride on the cathode). In addition, several runs show temperature instabilities of ~ 20 K at constant input power, calling into question the quality of the calorimetry.

7.8 Energetics: *Superwaves* and Other Variable Electrochemical Currents

7.8.1 Literature Review

Energetics Technologies was an Israel-based company that claimed spectacular cold fusion results with COPs in excess of 30 [102]. At the 2004 International Conference on Cold Fusion, they claimed 20 W output on less than 1 W input. The secret sauce to these spectacular results was ostensibly the application of a specific current history to the electrodes, for which they coined the term *Superwave*. *Superwaves* are self-similar waves that increase in frequency and amplitude as the amplitude of the kernel increases. In theory, there is no limitation in frequency, but in practice the lower frequency is determined by the length of the loading cycle and the higher frequency is determined by the instrument (typically 100 Hz, Fig. 7.34). The mechanism through which the *Superwaves* affect the sample and the heat production is unknown. Their data suggests that loading is not affected by the shape of the *Superwaves*. Two instances of excess heat include sample 56, which yielded a fairly constant COP of ~ 2 for 300 h, without spikes or runaways. Sample 64a showed a COP of 25, with 20 W out over 0.74 W in, with many spikes and peaks. They used an isoperibolic calorimeter to measure heat. While the cell did contain a recombiner, it is suspiciously placed outside of the calorimeter. It is unclear what the vessel pressure was and if it was monitored. The samples they used were palladium foils prepared by Violante (ENEA [Italian National Agency for New Technologies, Energy and Sustainable Economic Development], Italy), measuring $60 \times 7 \times 0.05 \text{ mm}^3$ (preparation protocol unpublished).

In general the experiments are poorly documented, with many graphs missing labels or units. Output powers appear very noisy, often at frequencies impossibly high when considering the thermal time constant of the system. In several instances (samples 64a and 64b), high COPs coincide with the onset of a faulty resistance measurement, which could hint at a bad contact in the resistance measurement and hence unaccounted electrical power from ohmic heating in the resistance measurement entering the calorimeter.

In 2011, McKubre et al. at SRI reported a replication of the Energetics experiments, using the electrolytic cell and calorimeter provided by Energetics [270]. Another replication took place at ENEA [270]. While this report was published in a Conference Series of the American Chemical Society, it does not seem to have undergone peer review, as many of the plots are missing axis labels or units. It is unclear what led to the design of placing the recombiner outside of the calorimeter, such that most of the heat of recombination is not accounted for in the calorimetry, but some of it does re-enter the calorimeter in the form of warm water droplets. Disappointingly, some of the excess heat plots lack the input power traces ([102] Fig. 6). For example, a COP of 5 was reported without the application of *Superwaves* (Fig. 7.35) [270]. It is reported that the input power went down at $\sim 65 \text{ h}$ due to reduced electrolyte resistance as the "Electrolyte Temperature" (sic!) went up, even though it is difficult to construct an effect of this magnitude. It is also surprising to see an almost perfect mirror-symmetric effect of input power on output power (apparently convolved with an instrument function). In the absence of a poorly calibrated compensation heater, this relationship suggests that errors in accounting of input power are a likely cause for the elevated COP.

7.8.2 Replication Experiments

Despite the shortcomings of the Energetics reports, we implemented *Superwaves* into the electrochemical current applied to the electrodes in the MS80 calorimeter (section 2.8.1). In light of the questionable design of the original Energetics calorimeter, we opted to use our own well-characterized closed system calorimeter. Owing to the undocumented preparation procedure of the palladium samples in the original

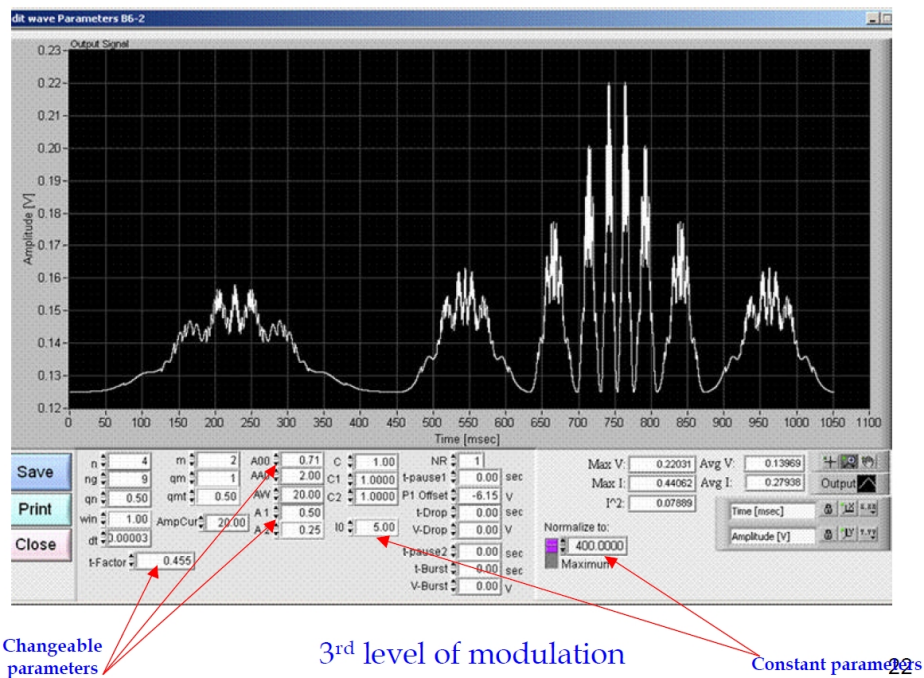


Fig. 7.34: Illustration of a *Superwave* signal applied to the electrodes. Source: [102]

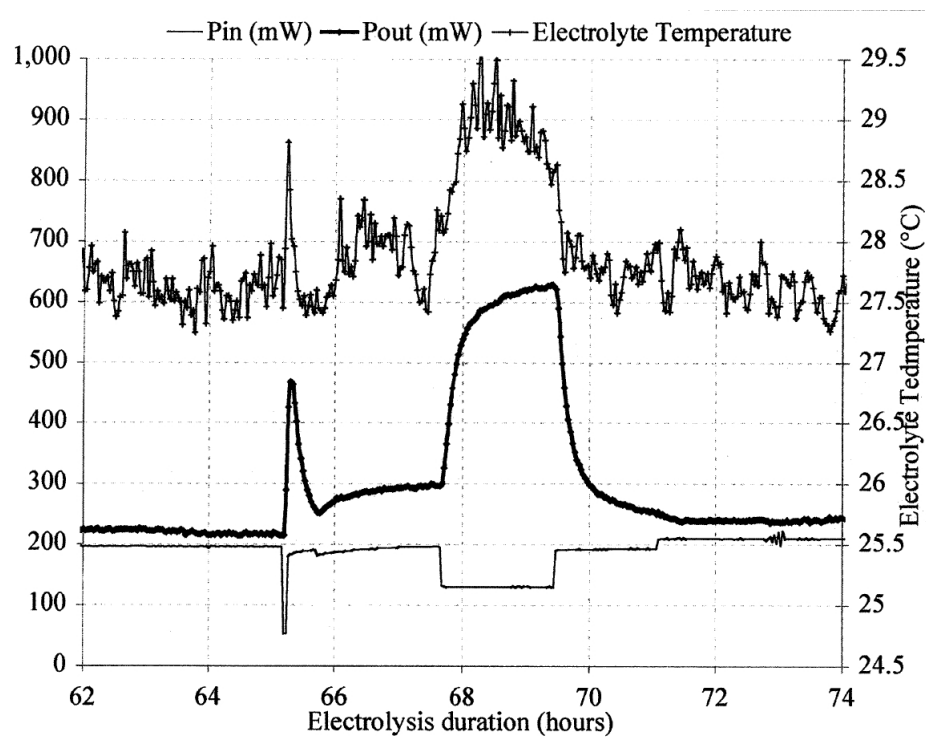


Fig. 7.35: Example of excess heat (COP up to 5) reported by Energetics [270, fig. 13].

experiment, we used as-received palladium wires (Johnson Matthey, 0.5 mm diameter, 28 mm length, in 0.5 M LiOD in D₂O). *Superwaves* were implemented using National Instruments source measure units (as standard electrochemistry potentiostats cannot supply these waves). Specifically, we applied a five-fold nested harmonic wave as illustrated in Fig. 7.36a, with the highest frequency being 700 Hz and the lowest frequency 0.1 Hz. The average current density was 8 mA/cm². The resulting EC voltage, resistance ratio and heat flow are shown in Fig. 7.36 b. No excess heat was detected. We repeated the experiment and we also performed modified replications with increased current density to achieve higher loading and with simple harmonic waves at various frequencies (frequency sweeps). No excess heat was detected.

7.8.3 Conclusions

Neither the Energetics replication experiments (employing *Superwaves*), nor derived experiments with alternating electrochemical currents, showed excess heat. We would like to iterate that the original reports were not published in reputable peer-reviewed journals and are of exceedingly poor quality (omission of critical details, missing units, missing labels, erratic heat flows). In several instances, high reported COPs coincided with the onset of faulty resistance measurements, pointing to hardware malfunction as potential source of apparent excess heat.

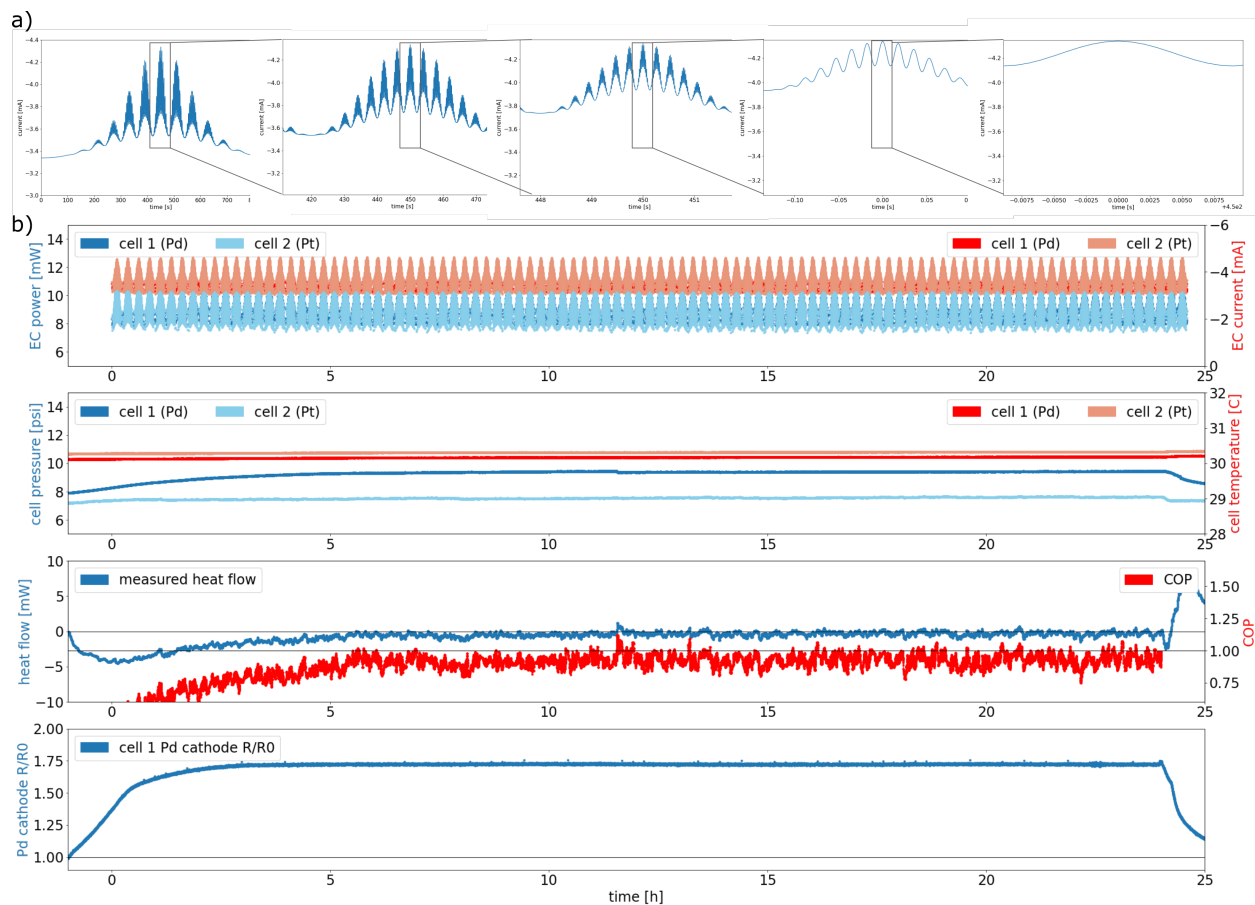


Fig. 7.36: Replication of Energetics *Superwave* experiments in Setaram MS80 calorimeter. a) Applied electrochemical current in the form of a five-fold nested wave (highest frequency 700 Hz, lowest frequency 0.1 Hz); b) In-situ calorimetry data showing EC power and current (top row), cell pressure and cell temperature (second row), heat flow and COP (third row) and cathode resistance ratio, R/R_0 (bottom row). Note the suppressed heat flow during the initial loading as it causes a non-stoichiometric head space gas composition and a reduction in recombination. Upon cessation of EC current, the reverse can be observed.

7.9 Letts: Laser stimulation

7.9.1 Literature Review

In 2003, Letts and Cravens reported excess heat when exposing a palladium deuteride sample located in a magnetic field to laser light [271]. The sample was prepared following a recipe involving repeated polishing, annealing, etching and cold rolling. After electrolytic loading at a moderate current for several days, the current was increased to 1 A and after another day, a layer of gold was electrochemically deposited onto the sample and the laser was turned on. The frequency of the laser light was reported to be relevant, but only a limited number of frequencies was investigated. Among the suitable frequencies was red laser light (682 nm and 662 nm). A crude model was suggested to determine suitable frequencies, however Letts noted "there is no physical reason why they should work, other than luck" [271]. The reported excess heat was typically a few hundred mW up to 1 W for several hours, with a laser power of 30 mW. The electrochemical input power is not specified, but given the current of 1 A, it is likely a few watts. The calorimeter was isoperibolic with two temperature sensors inside the electrolyte. The glass beaker was closed with a lid containing a recombiner and a seal, but it is not clear if the cell was actually gas tight. Performing electrolysis inside a gas tight glass vessel would present a substantial hazard.

The data Letts presented lacks scientific rigor and quality. Figures are missing axis labels, units, and scale bars. In some cases, incorrect units are listed. No baseline heat flow is provided prior to the laser exposure, and in some cases the heat flow does not return to baseline after the laser is turned off.

A few years later, Letts collaborated with Hagelstein, using an additional laser with a slightly offset frequency [104]. The lasers interfere at the sample surface, creating a beat wave in the THz regime, overlapping with optical phonon frequencies in palladium hydride at 8 THz and at 15 THz [189]. Hagelstein's theory proposed that phonon resonances are involved in low energy nuclear reactions. The sample preparation, loading protocol and calorimetry were very similar to the previous work [271]. The authors reported excess heat when the beat frequency coincides with the phonon resonance (Fig. 7.37a). In addition to the compiled figure, they also provided the excess heat measurements in tabular form and figures of individual experiments at a certain frequency range. Our analysis of these datasets is shown in Fig. 7.37b). Interestingly, no trend of excess heat *vs.* frequency is apparent from our analysis.

Suspiciously, there are numerous inconsistencies in the data provided in the paper [104]:

- Figure 9, at a beat frequency of 15.4 THz, shows a huge variation in excess heat from +180 mW to -50 mW. This is confirmed in the text, but not reflected in the table on p. 8.
- In figure 10, the scan rate of excitation frequencies is much too fast for the calorimeter to respond. The calorimeter takes about three hours to equilibrate after turning on power input (p. 5).
- Figure 11 shows excess heat of 200 mW for several hours after the laser is turned off.
- They claim very good reproducibility, but figure 13 shows a repeat experiment and does not support the claim.

In 2015, Letts published a similar paper based on the same dataset [272].

Guffey et al. from ReResearch LLC reported an attempted, but failed replication of the Letts experiments [273]. They directly corresponded with Letts and also tested some of his samples. Because the gold deposition duration was not specified in the original papers, they attempted 10 min to 14 h gold plating durations. As opposed to the original experiment, they used lasers with a lower power of 10 mW instead of

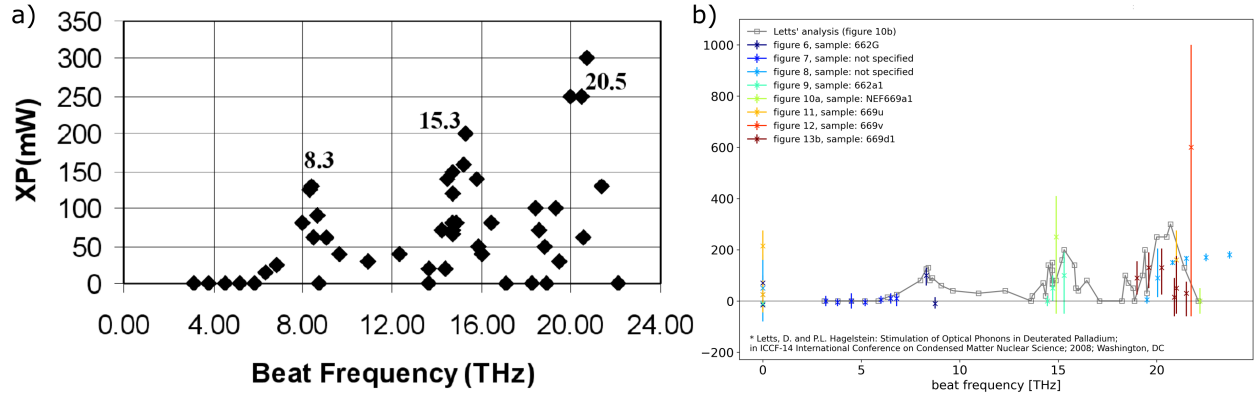


Fig. 7.37: Excess heat *vs.* beat frequency plots of Letts' experiment [104]. a) Plot reported by Letts. Excess heat maxima coincide with the edges of the optical phonon band at 8 THz and at 15 THz Source: [104]. b) Our analysis of the data provided by Letts [104]. No systematic dependence of excess heat on beat frequency is visible. Large variations in excess heat were observed with the laser off (shown at a beat frequency of 0 THz).

30 mW. Ultimately, they provide a histogram of 231 replication experiments, showing an average excess heat of 6 mW (compared to a few W input), concluding that there is no significant excess power. They provide a list of improvement suggestions:

- The heat of recombination has to be included in the heat balance. Apparently, according to their communications, this was not the case in the original papers.
- Variations in the recombiner efficiency can cause heat spikes, which are difficult to diagnose if the pressure is not measured.
- They found >2 K temperature non-uniformity in the electrolyte, which can lead to huge calorimetry errors, especially if the sensor placement is not carefully controlled.
- They encountered several temperature sensor failures, which leads to calorimetry errors.

7.9.2 Replication Experiments

Notwithstanding the severe lack of scientific quality of the original Letts papers and the unsuccessful replication attempts by ReResearch, we performed our own replication attempts, attracted by Hagelstein's phonon resonance model, which provided a more physically sound explanation of excess heat than most other stimulations (e.g., *superwaves* or electromigration). Though fundamentally, the concept of lattice assisted fusion is still at odds with known nuclear physics.

Due to the shortcomings of Letts' calorimeter (semi-open, isoperibolic), we designed a device based on the thermal bar heat flow measurement principle of the HTcal. Our system is thermodynamically closed. A quartz window provides optical access for the two laser beams (Fig. 7.38 a). We followed Letts' sample

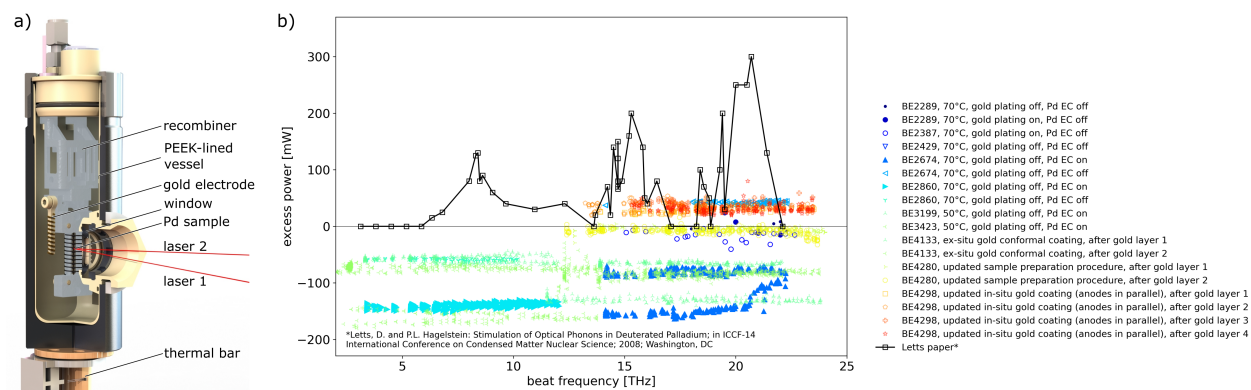


Fig. 7.38: In-house replication of Letts' experiments. a) Schematic of experimental setup showing the HTcal style vessel with a quartz window for optical access. Not shown are the magnets, foam insulation and laser optics including laser sources, optical power meters, spectrometer, polarizers and focusing mirrors. b) Compilation plot of excess heat *vs.* beat frequency of replication experiments.

preparation protocol [104,271] as closely as possible, and were aided by direct communications with Letts. After loading the sample at 0.1 A for 120 h, the EC current was increased to 0.6 A, resulting in an EC power of ~ 3 W. 24 h after that, gold was deposited incrementally. To this end, either the bias was applied between the gold and palladium electrodes (with the platinum counterelectrode disconnected) or the gold electrode was connected in parallel with the platinum counterelectrode. The gold plating duration per deposition increment was varied between 10 s and 1 min with typically five to ten depositions. After each deposition, the laser frequencies were scanned such that the beat frequency was scanned up and down across one of the phonon resonance peaks (2 to 14 THz or 12 to 24 THz). The range of the laser frequencies was not sufficient to cover both phonon resonances; instead one of the lasers had to be swapped to cover the second phonon resonance peak. The duration of each beat frequency scan was typically 50 h. An example dataset of one of the replication experiments is shown in Fig. 7.39. None of the replication experiments showed a clear dependency of excess heat on beat frequency (Fig. 7.38 b). In the initial replication experiments, the excess heat was suppressed by ~ 100 mW, which is $\sim 3\%$ of the input power. We ascribe this reduced accuracy to effects of the window on heat transfer, which were exacerbated by the elevated enclosure temperature of 50°C . While the accuracy was initially suppressed, the precision of the calorimeter is $<1\%$.

7.9.3 Conclusions

The excess heat reported by Letts [104] as a function of laser-stimulated phonon resonances is not supported by the data he provides in the same paper (see Fig. 7.37b), and is therefore an unsubstantiated claim lacking scientific evidence. Previous replication attempts failed as well [273].

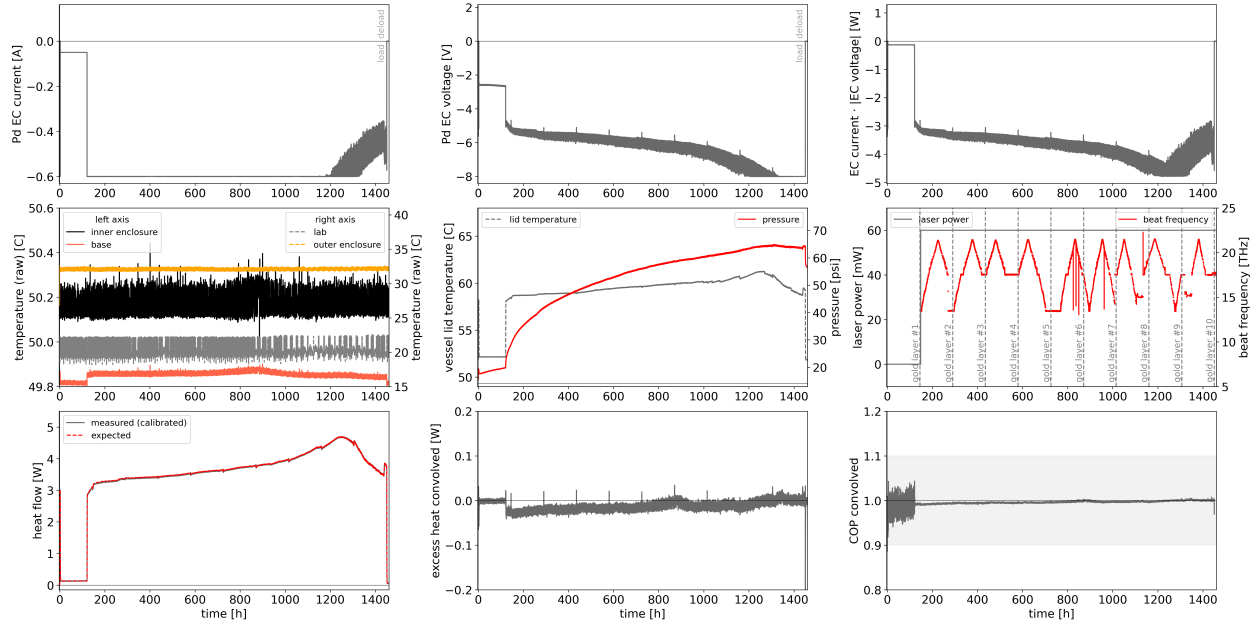


Fig. 7.39: Example dataset of in-house replication of the Letts experiment. Ten layers of gold were deposited. No anomalous heat was detected.

7.10 Conclusions

Callisto performed several hundred replication experiments. Some of them were replicated exactly, down to sourcing vintage components based on correspondence with the original scientists. In other cases, critical elements were replicated, but existing infrastructure (e.g. sample holders, calorimeters) was utilized. We found no signs of excess heat. To summarize:

- Fleischmann [26]: The paper is lacking electrochemical or calorimetry data to support their claims. Insufficient details for an accurate replication were provided.
- McKubre [101, 110]: After reporting excess heat in his 1994 paper, in his 1998 paper he admits that the calorimetry was “seriously inaccurate” under the given electromigration conditions and concludes “that the presumption of repeatable excess heat production was premature”.
- Staker [105]: Staker reported a one-off excess heat event with a COP of 2.0 using open-system calorimetry. In a corrigendum, he retracted the claim and reported a COP of 1.67 [241]. Retracting the central outcome of a paper sheds a bad light onto his scientific rigor. We observed false COPs of similar magnitude using exact replications of his setup.
- Hubler [250]: Hubler’s paper was not published in a peer-reviewed reputable journal and contained inconsistencies in the data, in particular a low cell temperature during periods of reported excess heat, pointing to alternative heat sources.

- Storms [113]: Storms reported relatively small amounts of excess heat using an open-system calorimeter. He also reported excess heat in light water experiments, pointing at a miscalibrated calorimeter or mechanisms other than D-D fusion.
- Barham [106]: Barham reported large amounts of excess heat (5 W at 15 W input) when in-situ depositing palladium onto the cathode in an open-system calorimeter. Curiously, the excess heat is constant and starts before substantial deposition of palladium. Localized temperature measurements indicate that the heat source is the anode, not the cathode.
- Energetics [102]: Energetics' reports were not published in reputable peer-reviewed journals and were of exceedingly poor quality (omission of critical details, missing units, missing labels, erratic heat flows). In several instances, high reported COPs coincided with the onset of hardware malfunctions.
- Letts [104]: The excess heat as a function of laser-stimulated phonon resonances is not supported by the data provided and is therefore an unsubstantiated claim. Previous replication attempts failed as well [273].