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CALORIMETRIC MEASUREMENTS OF EXCESS POWER OUTPUT DURING THE CATHODIC CHARGING OF DEUTERIUM INTO PALLADIUM

R. A. ORIANI, JOHN C. NELSON, SUNG-KYU LEE, and J. H. BROADHURST
University of Minnesota, Minneapolis, Minnesota 55455

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A Seebeck-effect calorimeter was used to establish that generation of energy, in excess of the electrical energy input, can occur during the electrolysis of D₂O. The magnitude of the excess power is measured with respect to the electrolysis of H₂O as the baseline. The excess power levels of >60 W/cm³ palladium and excess energies of 74 kJ cannot be understood in terms of recombination of D₂ and O₂ within the calorimeter, other chemical reactions, or a storage-and-relaxation mechanism.

The claim by Fleischmann and Pons¹ of heat generation over and above the power put into the electrolysis of heavy water, D₂O, and its attribution to nuclear fusion have met with much justified skepticism. Although a few investigators, e.g., Huggins² and Appleby et al.,³ have also reported observing the unexpected heat production, many other workers have reported⁴ negative results. This disparity and the potential importance of the subject serve as the motivation for this technical note.

The calorimeter used in this work is of the Seebeck-effect type. It has 1961 copper-constantan junctions in series occupying the annular space between the concentric cylindrical metal walls of the calorimeter, which are electrically and thermally insulated from each other. The heat generated in an electrolysis cell at the center of the cylindrical cavity sets up a radial temperature gradient that produces a Seebeck electromotive force (emf) in the thermopile, which is fed to a sensitive strip-chart recorder. In this type of calorimeter, any temperature inhomogeneity within the energy-producing object is of no consequence. The end faces, perpendicular to the axis of the cylinder, do not have thermoelectric junctions. They are made of plastic, double walled to contain a dead air space, and serve for input of electric power and outlet of gases. The calorimeter measures ~81 cm long with a 25-cm i.d., providing ample space for an electrochemical cell and a long closely fitting spiral tube that serves to extract heat from the evolved gases before they escape from the calorimeter. The electrolysis cell is suspended centrally from the spiral tube, and the gases it produces are passed through a trap to encourage condensation within the calorimeter of water vapor carried by the gases before these are conducted into the spiral tube. The calorimeter is immersed in a thermostatically controlled water bath at 28°C.

The electrolytic cell used a 1-mm-diam × ~4-cm-long palladium wire as a cathode, held axially within a coil of platinum wire as the anode. Connections to the electrodes are made by platinum wires. The electrolyte was either H₂O or D₂O into which enough lithium metal was dissolved to produce a 0.1 M solution of LiOH or LiOD. Additional electrolyte was not put in during a calorimetric run so that the duration of a run was generally limited by the consumption of water by dissociation and by vaporization, if any. The isotopic purity of the heavy water used (from the Aldrich Chemical Company) was 99.9%. Electrical energy was furnished from a dc power supply usually operated in the constant-voltage mode, and the current and voltage were measured by sensitive instruments. The evolved gases were not measured.

To calibrate the calorimeter, electric current is passed through a coil of nichrome wire set into the electrolysis cell envelope in place of the electrodes. Figure 1a displays the results of a calibration run as a xerographic copy of the recorder chart of the calorimeter emf versus time. The mean emf, 3.93 mV, is constant within the noise band of ±0.02 mV over 11 h at steady state. Using the calibration and its uncertainty, as described below, this noise band corresponds

to ± 0.04 W. Figure 2 presents the electrical calibration curve in which the abscissa is the product of the current and voltage at the calorimeter input connections and the ordinate is the steady-state Seebeck emf of the thermopile, which requires ~ 80 min to be established. We estimate the uncertainty in the overall calibration to be $\pm 0.3\%$.

The dissociation of H_2O was measured in separate runs, each with ~ 220 ml of electrolyte containing 0.1 M LiOH in a cell in which the vacuum-annealed palladium electrode (99.9% purity from Johnson-Matthey) was surrounded by two concentric glass cylinders that, below the electrolyte level but not above, were perforated by many small holes to permit ionic conductivity between the electrodes. The purpose of this cell configuration was to decrease the mixing of the hydrogen and oxygen gases as much as possible to reduce explosion hazards. The hydrogen was made to flow through the spiral tube, made of glass in this configuration, before leaving the calorimeter through an exit tube. The oxygen was allowed to fill the calorimeter cavity and then to exit from the calorimeter through another exit tube with flow resistance larger than that of the exit tube for the hydrogen. It was discovered later that the difference in flow resistance caused differences in electrolyte level between the cathode and anode compartments so that the gases mixed within the dissociation cell. Figure 1b is a xerographic copy of the recorder chart, calorimeter emf versus time, of an H_2O dissociation run beginning with a reduction of input power to the cell after a previous run, showing the approach to steady state and displaying the stability of the signal during ~ 12 h at steady state. The signal varies within about ± 0.01 mV, which, with the uncertainty in the calibration factor, translates to an uncertainty in the power within ± 0.02 W. Figure 2 plots the steady-state emf values of various H_2O dissociation runs versus $I(V - 1.48)$, where I is the current, V is the applied voltage at the calorimeter input leads, and 1.48 V is the faradaic equivalent of the enthalpy of dissociation of 1 g-mol of H_2O . The abscissa is the thermal power that must leave through the walls of the calorimeter at steady state, thereby generating the Seebeck emf, and hence must replace the product IV that is appropriate for the electrical calibration. The dissociation runs with H_2O require ~ 3 h to achieve a steady-state calorimeter signal after which the steadiness and the noise in the signal, as well as in the errors in the measurement of the net input power, are such that the scatter of each data point is smaller than the symbols in Fig. 2. The close congruence of the H_2O results and the electrical calibration shows, among other things, that recombination of hydrogen and oxygen was negligible despite the gas mixing that occurred within the cell, and that the H_2O runs can serve as the calibration technique for the calorimeter.

The same cell and calorimeter configuration were employed for the dissociation of D_2O with 0.1 M LiOD, using a new palladium electrode from the same stock as for the H_2O runs, both having been vacuum annealed together. Figure 2 shows that the three data points in this run lie exactly on the line established by the H_2O experiments and the electrical calibration. For the D_2O , the abscissa represents $I(V - 1.53)$ where 1.53 V is the faradaic equivalent of the enthalpy of dissociation of D_2O . Note that the highest point of the D_2O runs, at 16.04 W, corresponds to a current density at the palladium cathode surface of only 0.422 A/cm², and that the cell was operated at that input power level for ~ 31 h. No evidence of anomalous power generation (i.e., no deviation from the H_2O line) is discernible.

The design of this cell made it impossible to attain desirable larger current densities without undesirably large electrolyte temperature levels. Another cell was therefore built in which the attempt to keep the oxygen and hydrogen separate was abandoned, leading to a considerably lower internal cell resistance. The mixed gases were conducted to a liquid trap, to a copper spiral tube in close contact with the inner wall of the calorimeter, and then out of the calorimeter.

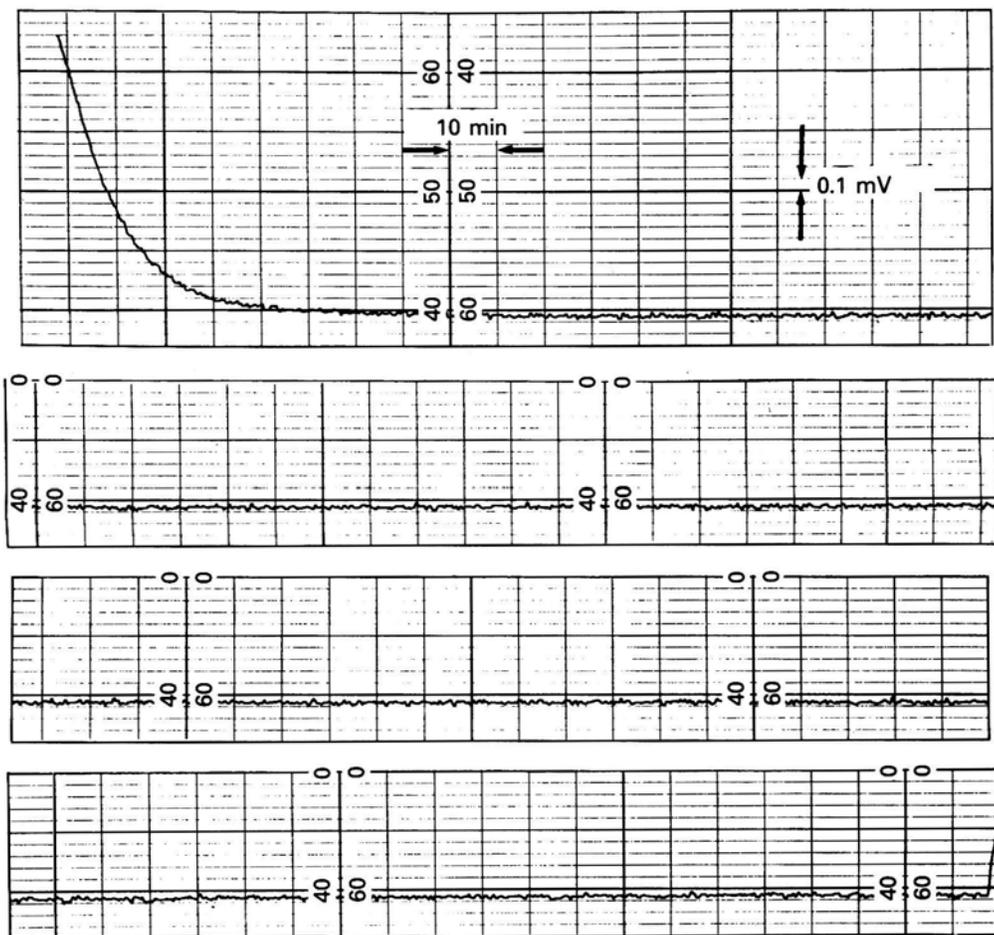


Fig. 1a. Strip-chart records of an electrical calibration run, showing the degree of steadiness of the calorimeter signal. Time advances from left to right and from the upper to the lower segments.

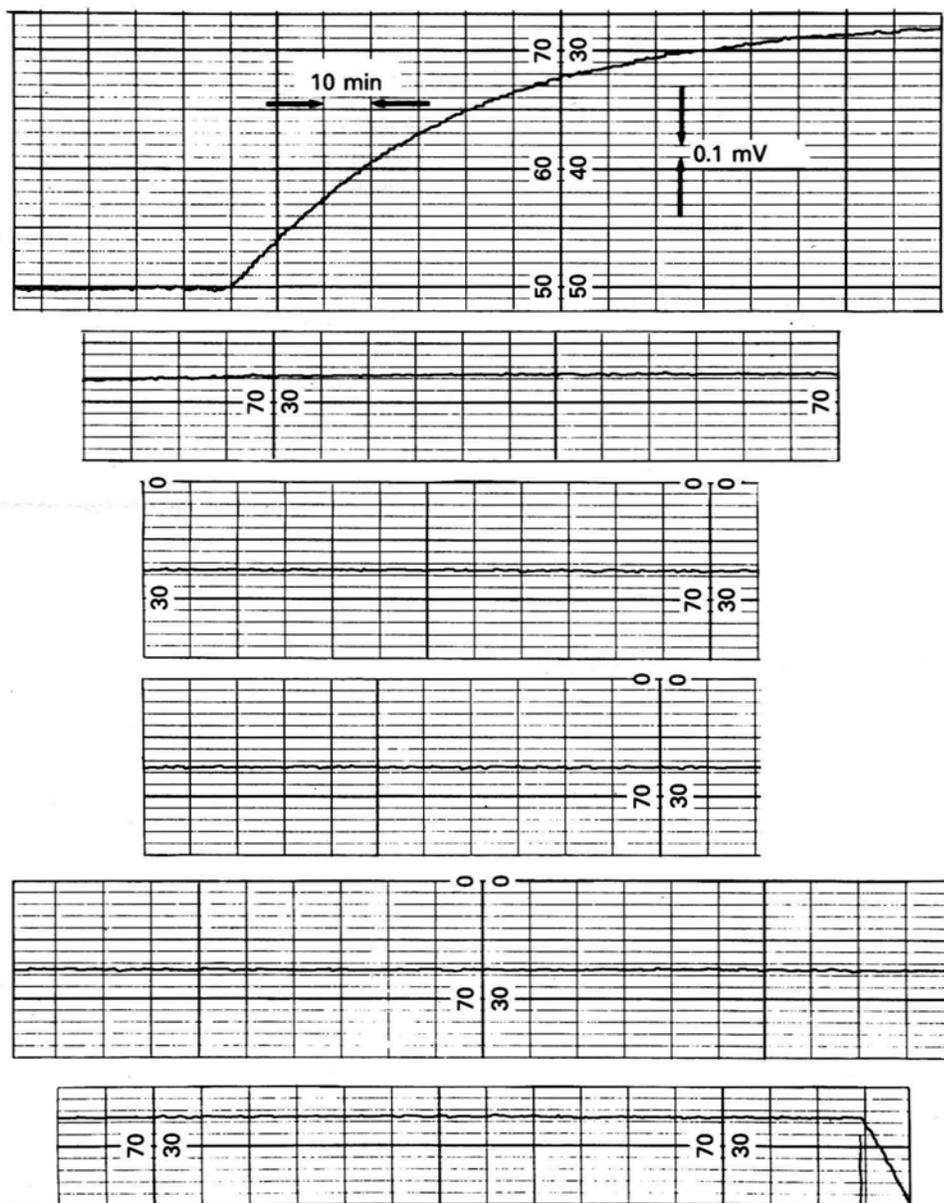


Fig. 1b. Strip-chart records of an H_2O electrolysis run, showing the degree of steadiness of the calorimeter signal. Time advances from left to right and from the upper to the lower segments

The first run conducted with the low-resistance dissociation cell employed D_2O , 0.1 M LiOD, and 0.5 ml of concentrated D_2SO_4 (from the Aldrich Chemical Company) in 146 ml of electrolyte. The latter was added to decrease the ionic resistance of the electrolyte and to increase the input fugacity of the deuterium, in analogy with what is known to happen with ferrous alloys.⁵ The palladium cathode was 1-mm-diam vacuum-annealed wire from the same stock as for earlier experiments. The anode was a spiral of platinum wire, as before, concentric with the cathode. However, a 2.5-cm length of 0.25-mm-diam palladium wire was spot-welded to the platinum anode. The purpose was to produce asperities of palladium upon the cathode by anodic dissolution from the anode and subsequent cathodic deposition on the cathode. The motivation was the desire to produce very large localized electric fields at the minute palladium protruberances, and therefore large highly localized concentrations of dissolved deuterium within the metallic protruberances, and consequently, very large concentration gradients. This should promote the internal cracking of the palladium, as is known to happen in other metals.⁶

We were led to this strategy regarding the possible role of cracks in cold nuclear fusion within metals by the considerations of Mayer et al.⁷

The results of this D₂O run are shown in Fig. 3; the scatter in each point is smaller than the size of the symbol used to represent it. The first two data points in the experimental sequence lie on the H₂O line (determined in subsequent experiments with the low-resistance cell). These points represent periods of constant input power of 16¼ and 22 h. Then we began to obtain data points anomalously, but definitely above the H₂O line. As an example, the data point at 11.3 W showed a constant calorimeter signal of 5.91 ± 0.05 mV for >2 h (after which the input power was decreased); this was preceded by a 4-h rise to steady state. This point corresponds to 1.09 A/cm^2 and to an excess heat flux of $0.70 \pm 0.18 \text{ W}$; i.e., with H₂O, the net input power required to yield the observed calorimeter signal would be 12.0 W. Although the magnitude of the applied current density seems to influence whether anomalous power generation occurs, it is clearly not the only factor. Consider the data point immediately below the above-noted point at 11.3 W. This data point was the last calorimetric steady state measured in this D₂O run and it lies directly on the H₂O line. The lowest current density at which excess power was observed in this series was 0.82 A/cm^2 , corresponding to the data point at 6.86 W in Fig. 3.

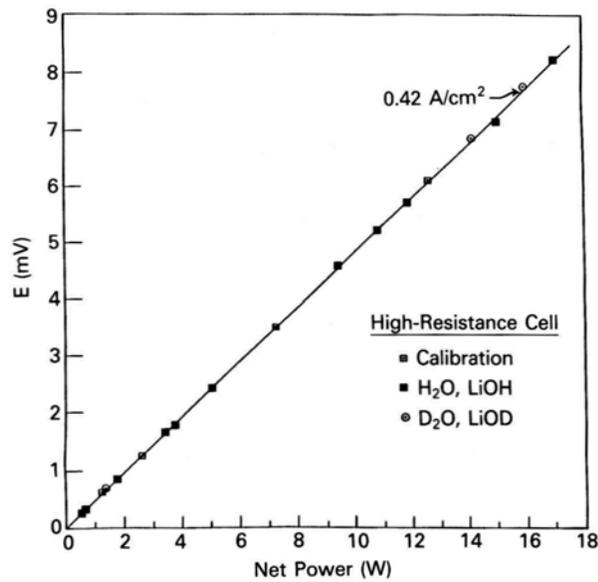


Fig. 2. Calorimeter signal (Seebeck emf) versus net power input for the electrical calibration.

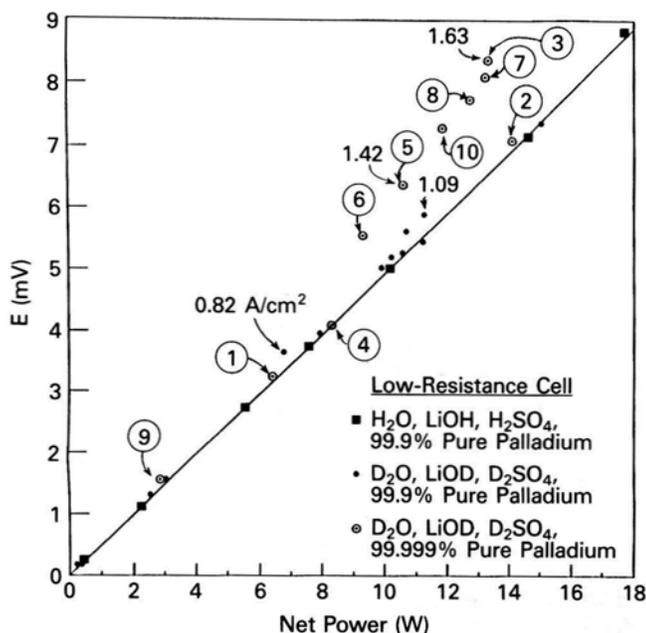


Fig. 3. Calorimeter signal versus net power input for electrolysis in low-resistance cell using acidified electrolytes and palladium wire attached to the platinum anode. Numbers in circles indicate the experimental sequence for the second D_2O series.

The surprising observation of excess power with the low-resistance cell caused us to question the applicability of the line established with the high-resistance cell for the electrolytic dissociation of H_2O , which coincides with the results of electrical calibration. Therefore, the next experimental series performed with the low-resistance cell was H_2O dissociation using 0.1 M LiOH plus 0.5 ml concentrated H_2SO_4 , a short length of palladium wire spot-welded to the platinum anode, and another palladium wire, of the same provenance as the others, as the cathode. The results of these experiments appear in Fig. 3. A comparison of this H_2O line with that in Fig. 2 shows that both lines begin exactly at the origin with the line in Fig. 3 having a higher slope, such that at 14 W the calorimeter signal is nearly 0.2 mV higher. We believe that this small difference arises from the extraction of heat by the copper spiral in the second experimental configuration rather than by the glass spiral.

Another D_2O dissociation run was performed with the low-resistance cell after the series with H_2O . The new electrolyte was again 0.1 M LiOD plus 0.5 ml of concentrated D_2SO_4 in 140 ml of electrolyte, and palladium wire was affixed to the platinum anode. In this run, the cathode was from different stock. It was 1-mm-diam \times 4.4-cm-long 99.999% pure palladium wire and was used without any pre-treatment except cleaning. The results are shown in Fig. 3. The initial point at 6.47 W represents a steady state lasting >16 h and it lies on the H_2O line established with the low-resistance cell. After this, the input power was increased to 14.16 W (at 11.298 V). As the steady state was being approached, it was noted that the current had exceeded the capacity (2 A) of the ammeter used; as the cell temperature increased, the cell resistance decreased so that the current increased at constant applied voltage. The voltage was therefore decreased, and a higher range ammeter was substituted. The point labeled 2 in Fig. 3 represents the calorimeter signal at 1.988 A and before a steady state had been reached, as indicated in Fig. 4. Point 2 is above the H_2O line and shows that excess power began generating at some time during the approach to steady state, which takes ~ 3 h with the low-resistance cell. At steady state, the calorimeter signal would be above point 2 for that input power of 14.16 W.

Figure 4 displays the interesting phenomenon that the reduction of the input power produced a sudden reversal of the incipient expected lowering of the calorimeter signal. The power output continued to increase at the reduced input of 13.37 W (10.491 V) and reached a quasi-steady state $\sim 1\frac{1}{2}$ h later. Point 3 represents an anomalous (excess) power generation of ~ 3.6 W. To put this in perspective, we note that the power that would be generated by the complete recombination of D_2 and O_2 within the calorimeter would be 1.53 I, which at the measured current equals 3.41 W. Reduction of input power yielded point 4 on the H_2O line despite the current density of 1.06 A/cm². This was followed by several anomalously high points, after which the input power was reduced to 2.903 W, yielding point 9, which is above the H_2O line even though the current density is only 0.63 A/cm². At this stage of the investigation, it is not

possible to assert that there exists a threshold current density below which excess power generation does not occur; apparently, the phenomenon is somehow a function of the preceding states.

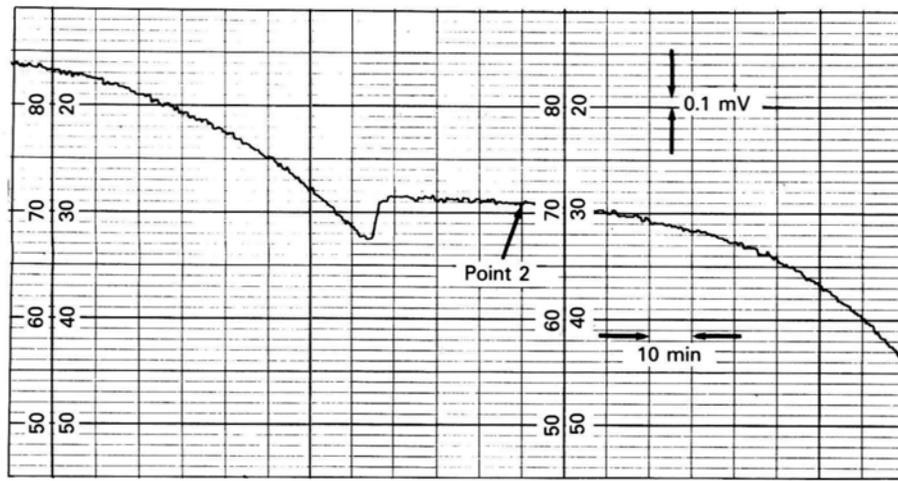


Fig. 4. Section of strip-chart record for the second D₂O run of Fig. 3, showing the anomalous rise of the calorimeter signal a short time after reduction of input power.

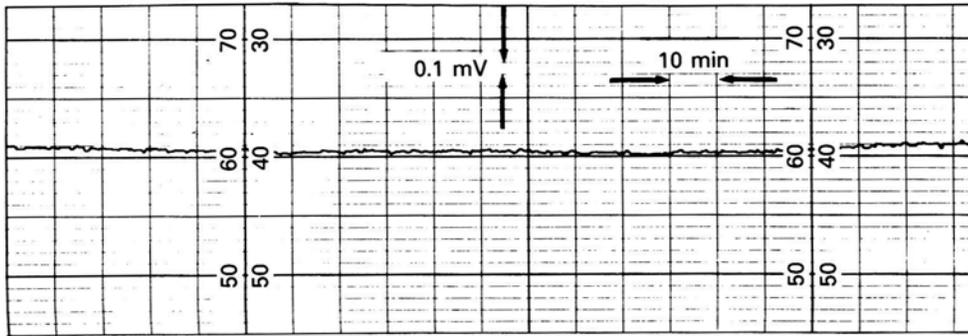


Fig. 5. Section of strip-chart record showing the “waviness” that was often found to accompany excess power production.

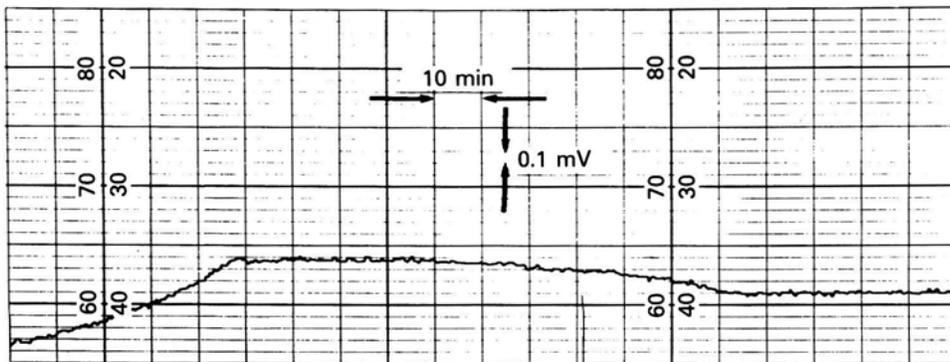


Fig. 6. Section of strip-chart record showing rise of calorimeter signal after a long quasi-steady state, all at constant input power. Time advances from right to left.

TABLE I
Characteristics of the Second D₂O Experiment in the Low-Resistance Cell

Episode	Input Watts		Duration (min)	E^a (mV)	E_0^b (mV)	Input Energy		Excess Quantities	
	Net	1.531				(Net kJ)	(Total kJ)	(W)	(kJ)
1	6.465	1.957	985 ^c	3.25 ^d	3.25	382.1	497.7	0	0
2	14.16	3.04	156 ^c	7.15 ^e	6.95	132.5	160.0	0.4 ± 0.1	---
3	13.37	3.41	90 ^e	7.8 ^f	6.58	72.2	90.6	2.5 ± 0.2	13.5
	13.37	3.41	150 ^d	8.35 ^d	6.58	120.3	151.0	3.6 ± 0.2	32.4
4	8.33	2.24	320 ^e	4.09 ^d	4.09	159.8	202.8	0	0
5	10.68	2.98	30 ^e	---	5.23	19.2	24.6	---	---
	10.68	2.98	120 ^g	5.8 ^f	5.23	76.9	98.4	1.16 ± 0.2	8.4
	10.68	2.98	560	6.1 ^d	5.23	358.8	459.0	1.78 ± 0.2	59.8
	10.68	2.98	103	6.35 ^f	5.23	66.0	84.4	2.29 ± 0.2	14.1
6	9.36	2.80	77 ^h	---	4.60	43.2	56.2	---	---
	9.36	2.80	70 ^d	5.56 ^d	4.60	39.3	51.1	1.96 ± 0.1	8.2
7	13.27	3.44	17 ^e	---	6.52	13.5	17.0	---	---
	13.27	3.44	63 ^g	7.5 ^f	6.52	50.2	63.2	2.00 ± 0.2	7.6
	13.27	3.44	68 ^d	8.1	6.52	54.1	68.2	3.23 ± 0.2	13.2
8	12.76	3.38	42 ^h	---	6.28	32.2	40.7	---	---
	12.76	3.38	33 ^d	7.78 ^d	6.28	25.3	32.0	3.07 ± 0.1	6.1
9	2.903	1.33	167 ^h	---	1.34	29.1	42.4	---	---
	2.903	1.33	770 ^d	1.56 ^d	1.34	134.1	195.6	0.45 ± 0.2	21
10	11.91	3.23	44 ^e	---	5.85	31.4	40.0	---	---
	11.91	3.23	109 ^g	7.0 ^f	5.85	77.9	99.0	2.4 ± 0.2	15.4

^a E = observed calorimeter signal.

^b E_0 = calorimeter emf of the calibration curve (H₂O dissociation line) at same net power input.

^cTotal over the entire episode.

^dSteady state.

^eRising signal portion of the episode.

^fAverage over the indicated duration.

^gRising signal portion when $E > E_0$.

^hDecreasing signal portion of the episode.

Table I lists the raw data and calculated quantities for this D₂O run. The values of excess power were calculated by subtracting from the observed millivolt signal the amount E_0 corresponding to the H₂O line at the same power input, and multiplying the difference by the slope of the H₂O line: 2.03 W/mV ± 0.3%. An interesting characteristic that is often, but not always, observed when there is excess power generation is that the calorimeter signal shows an overall waviness of ~0.05-mV amplitude and a wavelength of several hours; Fig. 5 shows an example. This is in strong contrast with the behavior with H₂O for which the signal is contained within a band of <0.01 mV (Fig. 1b), and it may be caused by sporadic generation of excess power smoothed out by the large relaxation time of the calorimeter. An egregious example of the variability of the signal during anomalous power generation is shown in Fig. 6, which is a record of the signal-time chart a few hours after the record of Fig. 5. We see in Fig. 6 that at constant input power during which the calorimeter signal had been slowly varying about a mean value of 6.1 mV, as described for Fig. 5, for ~560 min, the Seebeck emf rose to 6.38 mV within 30 min, producing point 5 of Fig. 3. Note that 6.1 mV also represents an anomalous power generation of ~1.8 W. Integrating this excess power over 560 min and the excess of ~2.3 W over 103 min gives

an excess energy of 73.9 kJ for the episode ending with point 5. Integration of the excess power of all of the episodes of the second D₂O run with the low-resistance cell yields ~200 kJ for the total excess energy generated. The excess power output represented by point 5, using units employed by Fleischmann and Pons' is 67 W/cm³ palladium; point 3 represents 106 W/cm³ palladium. These numbers are considerably larger than those reported by Fleischmann and Pons¹ and by Appleby et al.³

To examine possible chemical reactions that may be responsible for such magnitudes of energy generation, assume first the exothermic formation of a palladium deuteride that (surprisingly) does not have a corresponding palladium hydride. Assuming that the hypothetical PdD₂ has a ΔH of formation of -200 kJ/mol, one calculates that at 1A the excess power would be 1.04 W if all the D₂O reacted to form hydride. Since our work and that of many others (e.g., Ref. 3) demonstrate that essentially all the D₂ leaves the cell at steady state, the calculated excess power from this source decreases by a factor of at least 50. Furthermore, should such a hypothetical deuteride be formed at high current densities at which a high activity of dissolved deuterium is generated, at low current densities and correspondingly low activities the PdD₂ would decompose endothermically and thereby produce abnormally low calorimetric signals (i.e., below the H₂O line). This is never observed. One can similarly examine the possible exothermic formation of a lithium deuteride for which the corresponding hydride does not form under parallel conditions. Assuming -200 kJ/mol LiD for the enthalpy of formation, and knowing that our cells had an initial lithium ion content of 0.0104 mol, the maximum possible excess energy would be only 2.8 kJ, not the 73.9 kJ of excess energy that we observed in the episode leading to point 5. It is similarly most difficult to conceive of any chemical reactions involving impurities in the metals or in the electrolyte to be responsible for the magnitudes of excess energies in our experiments. Finally, we note that the possibility that the anomalous power is the result of the storage in the system of some energy (such as mechanical strain energy or the formation of an unstable endothermic compound) followed by its subsequent relaxation can be refuted not only by the large magnitudes of excess energies observed here, but also by the realization that during the time of the alleged storage of energy, the calorimeter signal would necessarily fall below the H₂O line for the corresponding input power. This was never observed.

If nuclear fusion is responsible for the observed excess power, it may be considered that one or more of the following reactions is taking place:

1. $d + d \rightarrow {}^3\text{He} + n + 3.269 \text{ MeV}$ (n escape energy = 2.452 MeV)
2. $d + d \rightarrow {}^4\text{He} + \gamma + 21.422 \text{ MeV}$ (γ escape energy = 21.176 MeV)
3. $d + d \rightarrow \text{T} + p + 4.032 \text{ MeV}$
4. ${}^6\text{Li} + d \rightarrow {}^8\text{Be} \rightarrow {}^4\text{He} + {}^4\text{He} + 23.374 \text{ MeV}$

Reactions 1 and 2 produce penetrating radiations with the indicated energies, which would escape from the calorimeter and not contribute to the heat generation. Only the difference between the reaction energy and the escape energy would generate heat. To generate excess power of 1.0 W by reaction 1, it would have to produce 7.6×10^{12} n/s. At 1 m from the calorimeter, the flux would be $\sim 6 \times 10^7 \text{ cm}^{-2}\text{s}^{-1}$, which would correspond to a dose rate of $\sim 7.95 \text{ rad}\cdot\text{s}^{-1}$, assuming absorption in water and an absorption cross section of 1 b. However, during periods of production of excess power, the calorimeter was monitored by means of a Ludlum neutron monitor, and no neutron signal was observed.

Although a detector specifically designed for gamma rays was not used, radiation of energy corresponding to reaction 2 would interact with the BF₃ gas of the detecting element of the neutron monitor to produce a false indication of the presence of a neutron flux. A signal consistent with the gamma flux by reaction 2 was not observed.

Reaction 3 would be demonstrated by finding tritium within the palladium cathode and in the electrolyte after the generation of excess energy. To account by reaction 3 for the excess energy of ~200 kJ observed during the second successful D₂O run (Table I), $\sim 3 \times 10^7$ tritons should have been produced. The sample from this run was heated to ~1000°C for 5 min and the evolved gas (deuterium plus putative tritium) was catalytically converted to the corresponding oxides. These were diluted with 1.5 ml H₂O and 1 ml of the mixture was added to Ecolite Plus scintillator cocktail from the ICM Corporation and assayed with a well-type (Beckman) scintillator counter. The observed count rate was 24.1 count/min, and the background count was 24.7. Similar negative results were obtained for the deuterium gas evolved during cathodic charging and collected outside the calorimeter. The electrolyte used in this run was not investigated for tritium. The electrolyte used in the first D₂O run that exhibited excess power generation was analyzed for tritium. The result gave 81 count/min after the run and 61 count/min for an unused batch of electrolyte serving as a blank; this result is not considered significant. Negative results were also obtained with the deuterium extracted from the palladium cathode used in the first successful D₂O run.

Reaction 4 produces neither penetrating radiation nor radioactive daughter nuclei. After the production of ~200 kJ of excess energy hypothetically by reaction 4, 5.3×10^{16} helium atoms should appear, presumably in the palladium. This is considerably above the natural background for helium in metals [10^{11} to 10^{12} atom/g (Ref. 8)] and should be easily measurable. However, our means of extraction of the deuterium from the palladium precluded analysis for helium.

Since no nuclear manifestations were observed in this work, we do not assert that the excess power and energy that we have measured is the result of nuclear fusion. We intend to continue this investigation, specifically by incorporating an X-ray detector within the calorimeter, since both reactions 3 and 4 produce charged particles that would recoil in the palladium lattice with sufficient energy to generate the characteristic palladium X rays at reasonable intensities.

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