Calorimetric Studies of Pd/D$_2$O+LiOD Electrolysis Cells

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Abstract

New experiments in sensitive calorimeters displayed the characteristics of the excess power effect during seven different occasions. These measurements clearly show the anomalous increase in the cell temperature despite the steadily decreasing electrical input power during Pd/D$_2$O+LiOD/Pt electrolysis. This strange behavior can be modeled by the use of an anomalous excess power term in the calorimetric equations. Two thermistors used in each calorimetric cell always show nearly identical temperature changes, thus errors due to temperature gradients within the cell are unlikely. The onset of the excess power apparently develops in a gradual manner. There were never any large, abrupt increases in the excess power. The addition of D$_2$O with its sudden cooling of the cell generally dissipated the excess power effect. No clear triggering events for the excess power could be identified. Possible chemical explanations for the excess power are discussed. Normal behavior was always observed for a similar experiment conducted as a control.

Introduction

Ten years have passed since the announcement of the Fleischmann-Pons effect,[1,2] hence it is time to calmly examine the experimental characteristics of the anomalous excess power along with any possible artifacts or chemical explanations. These discussions of new experiments will therefore focus solely on basic principles of calorimetry and physical chemistry. The excess power effect is generally much too small (20-200 mW) to be easily detected or to offer promise as a new energy source. This study involves sensitive heat-conduction, isoperibolic calorimeters that are operated at constant current conditions where the complex calorimetric equations can be greatly simplified. The increase in excess power is then readily apparent by the corresponding increase in the cell temperature. With the use of this sensitive calorimetry, even small power changes of 5 mW can be readily detected by corresponding changes in the cell temperature. The goal of this new study is to characterize the appearance of the excess power effect and to discuss possible explanations for this phenomena.

Experimental Section

The basic calorimetric design has been described in previous publications.[3-5] Heat transfer is mainly by conduction. The small electrochemical cell consists of a long, narrow test tube (1.8 cm diameter and 15.0 cm length) that is filled with 18.0 cm^3 of 0.1 M LiOD. The electrochemical cell is placed in a calorimetric jacket within a secondary compartment that contains two thermistors positioned on opposite sides of the cell wall and at different heights from the bottom of the cell (1.9 cm and 4.5 cm). Figure 1 shows a schematic of the positioning of the two

thermistors relative to the palladium cathode rod (1mm × 20mm) and the platinum anode coil. It is important to note that the thermistors are placed on the outside wall of the cell rather than in the D_2O+LiOD electrolyte. In previous experiments, the secondary compartment was filled with distilled water.[3] In order to increase the sensitivity and to shorten the time constant for these cells, aluminum foil was wrapped around the outside of each cell including the two thermistors. The secondary compartment was then packed with aluminum foil rather than being filled with distilled water. This compartment was insulated at the top and then sealed with silicon rubber. These changes reduced the cell constant by about a factor of three, shortened the time constant to about 15 minutes, and thereby produced relatively rapid responses by the thermistors to any power changes within the cell. Two similar cells were prepared (Cells A and B) and run in series at constant current. Thermistor channels T_1 and T_6 were used for Cell A and T_3 and T_4 for Cell B. The lower numbered thermistor was placed in the lower position for both cells (Fig. 1). The temperature reading for each thermistor channel was numerically displayed to within 0.01°C and printed as a dot on the chart recording every 18 seconds. There were permanent chart recordings of all six temperatures (four cell temperatures, bath temperature, room temperature) as well as both cell voltages for the entire experiment. In addition, measurements were recorded by notebook during working hours. The bath temperature was always maintained at 21.51±0.01°C, and the room temperature was generally 1-2°C higher than the bath temperature. These experiments were conducted in a thermostatted room specially designed for calorimetric studies. Periodic D_2O additions were made using a glass syringe (5.0 cm^3) that was also used to determine the cell electrolyte volume at each D_2O addition.

In previous experiments conducted at China Lake, California, these Johnson-Matthey palladium cathodes produced approximately 200 mW of excess power in one cell, but no measurable excess power effects in a second cell (Figs. 31 and 32 of Ref. 5). These same two palladium cathodes were used again in Cells A and B in Japan. The obvious purpose was the direct comparison of a cell producing excess power (Cell A) with one that produces no anomalous effects (Cell B).

Calorimetric Equations and Modeling

The basic calorimetric equation that describes the heat conduction cells used in this study is

$$P_{EL} + P_x = a + K\Delta T + P_{gas} + P_{calor} \tag{1}$$

where $P_{EL} = [E(t)-\gamma E_H]I$ and P_x is the excess power.[4] The P_{gas} and P_{calor} terms are rather complex and have been described previously.[4] These equations stem from basic principles of physical chemistry and have not been challenged. Similar equations were used by Fleischmann et al.[2] to describe their more complex Dewar calorimetric cells. In this study, Cells A and B were run under steady state conditions where $(P_{gas}+P_{calor})$ «$K\Delta T$ and with sufficient insulation above the cells to render the power loss out of the top of cell (term a) insignificant. Therefore, our calorimetric equation greatly simplifies to

$$P_{EL} + P_x = K\Delta T \tag{2}$$

The time dependent equation for the cell constant for either Cell A or Cell B at any given cell current (I) can be expressed by the equation

$$K(t) = K_0 - (1/0.300)(1.85\times10^{-4})(t-t_0) \tag{3}$$

where K_0 is the cell constant at the time (t_0) expressed in hours when the cell was refilled with D_2O. The change of the cell constant with time is due to the decreasing volume (V) of the electrolyte in an open cell.[4] In fact, it was shown experimentally that $dK/dt=(dK/dV)(dV/dt)$ with values of -1.93×10^{-4} WK^{-1} hr^{-1} for Cell A and -1.78×10^{-4} WK^{-1} hr^{-1} for Cell B at $1=0.300$ A, hence an average of these two values is used in Equation 3.

The change of the cell temperature with time can now be theoretically calculated from Equation 2 where $\Delta T = T_{cell} - T_{bath}$, thus $d\Delta T/dt = dT_{cell}/dT$. Therefore,

$$dT_{cell}/dt = K^{-1} (dP_{EL}/dt+dP_x/dt) - K^{-2}(P_{EL} + P_x)dK/dt \tag{4}$$

assuming 100% faradaic efficiency ($\gamma=1.00$).

If there is no excess power ($P_x=0$) or if the excess power is constant ($dP_x/dt=0$) with $P_x \ll P_{EL}$, this equation becomes

$$dT_{cell}/dt = K^{-1} dP_{EL}/dt - K^{-2} P_{EL} dK/dt \tag{5}$$

Equations 4 and 5 provide the framework for the discussion of our calorimetric measurements.

Theoretically, the change in the cell temperature with time can be either negative, zero, or positive since dP_{EL}/dt is generally negative during electrolysis and dK/dt is always negative. Therefore, the first term in Equation 5 is usually negative while the second term is always positive.

Measurements of exact values for the excess power are more complicated than the detection of power changes since the small size of Cells A and B along with the use of aluminum foil in the secondary compartment makes the cell constants sensitive to the cell electrolyte volume. The relationships of the cell constants to the cell volume were determined experimentally as follows:

$$K_1 = 0.00195V_A + 0.01722 \tag{6}$$
$$K_6 = 0.00189V_A + 0.01686 \tag{7}$$

for Cell A and

$$K_3 = 0.00172V_B + 0.02685 \tag{8}$$
$$K_4 = 0.00170V_B + 0.02699 \tag{9}$$

for Cell B where V_A and V_B represent the volume of electrolyte (cm^3) in each cell at any given time. These cell constants were determined from Equation 2 assuming $P_x=0$ and selecting experimental time periods where this assumption appeared to be valid. The actual cell volume at each D_2O addition could not be determined more accurately than $18.0\pm0.5cm^3$. This introduces an error of $\pm1.9\%$ in the calculation of the cell constants and an uncertainty in the excess power of ±28 mW for a typical cell operating at 50.00°C (AT=28.49°C). These calorimetric cells are much more sensitive in detecting changes in the cell power than in accurately determining the exact amount of excess power.

Results and Discussion

The constant current electrolysis of the D_2O+0.1 M LiOD solution in an open cell slowly increases the conductivity of the solution resulting in a gradual decrease in the cell voltage.[4] Under normal conditions for our cells, this nearly linear decrease in the applied electrochemical power (P_{EL}) with time produces a corresponding linear decrease in the cell temperature with

time.[4] The onset of excess power (P_x) can often be recognized simply by the anomalous increase in the cell temperature despite the decreasing applied power. This raises the fundamental question asking how is it possible for the cell temperature to increase when the applied power is decreasing?

Figure 2 presents the first episode of excess power in Cell A after more than 12 days of electrolysis. Note the contrasting behavior of Cells A and B. Both cells showed the normal gradual decrease in the cell temperature until 292.7 hours when the current was increased from 250.28 mA to 300.21 mA. After reaching a new temperature plateau, the temperature of Cell A continues to increase for the next 10 hours while the temperature of Cell B gradually decreases. After 307 hours, the temperature of Cell A reverses its trend and once again begins to decrease with about the same dT/dt slope as observed for Cell B. The room temperature and the bath temperature are also displayed in Figure 2 using a different temperature scale that ranges from 20°C to 25°C.

Theoretically, the dT/dt slopes calculated from Equation 5 remains negative and nearly constant for both Cells A and B during the entire 287-322 hour time period shown in Figure 2. Several theoretical temperature-time slopes for Cells A and B along with the corresponding electrolysis input powers are presented in Table 1. Cell constant calculations using $K=\Delta P/\Delta(\Delta T)$ involving changes due to the increase in cell current yield $K_A=0.050 \text{ WK}^{-1}$. Therefore, the increase in the temperature of Cell A of 0.9°C in Figure 2 over the expected baseline yields an increase in the excess power of 45 mW.

It is clear from Figure 2 that the two thermistors in a calorimetric cell faithfully track each other despite their quite different locations (Fig. 1). The periodic fluctuations of temperature in Cell B track very closely the sudden changes in the cell voltage (0.1V) observed every 20-60 minutes. The temperature of Cell A is more chaotic than Cell B, but exactly the same behavior is recorded by both thermistors. The temperature differences between the two thermistors in the same cell is largely due to their positioning relative to the cell wall where there is a temperature gradient extending in the radial direction away from the cell. The higher positioned thermistor, however, yields the higher temperature in both cells. Our focus here is on temperature changes, and the two thermistors monitoring the cell always show exactly the same pattern of behavior.

Figure 3 presents another example for an increase in excess power in Cell A and normal behavior in Cell B. The two thermistors in Cell A show the onset of an increase in the temperature beginning at about 477 hours. Following the temperature peak, Cell A shows the normal decrease in temperature and about the same dT/dt slope as observed for Cell B. The two thermistors in each cell once again track the exact same pattern of behavior. Calculations based on Equations 6-9 and Equation 2 show that the excess power increased by 90 mW in Cell A between 474 and 487 hours (Figure 3) whereas there was no significant change in P_x for Cell B. The bath and room temperatures shown in Figure 3 are again for the temperature scale of 20-25°C.

Figure 4 summarizes the average voltage measured on the chart recorder for Cells A and B during the same time period displayed in Figure 3 (472-507 hours). Note that the voltage decreases faster for Cell A (-0.0314 V hr^{-1}) than for Cell B (-0.0205 V hr^{-1}). Theoretical calculations based on Equation 5 are presented in Table 1 and predict significantly more negative dT/dt slopes for Cell A than for Cell B. This is in contradiction to the experimental results shown in Figure 3.

Another episode of excess power in Cell A is presented in Figure 5. Two different temperature scales are used in this figure since Cell A was now running more than 10°C warmer than Cell B. Most of this difference, however, can be explained by the higher electrolysis power for Cell A (Table 1). It is interesting to note that the T_1 thermistor line for Cell A and the T_4 thermistor line for Cell B are merged together until about 600 hours. Both the T_1 and T_6 lines for Cell A then show a substantial increase in temperature that indicates a 60-70 mW gain in the excess power. Once again, Cell B continuously displays only normal behavior.

Figure 6 displays an excess power increase in Cell A along with a calibration based on increasing the cell current from 300.22 mA to 320.28 mA for a period of 2.33 hours. Based on notebook data, the power increase of 0.165 W produced a temperature increase of 3.29°C in Cell A. Therefore, the anomalous rise of cell temperature of 1.6°C above the expected baseline prior to this calibration indicates an increase in the excess power of about 80 mW. Cell B again shows normal behavior. This same increase in current produced a temperature increase of 2.18°C and a power increase of 0.110 W for Cell B. The relationship $K=\Delta P/\Delta(\Delta T)$ suggests that the cell constant is about 0.05 W/K for both Cell A and Cell B. Temperature changes of ±0.1°C are readily detected by these calorimeters, hence power changes of ±5 mW are measurable. Figure 6 shows that the same baseline is followed before and after the calibration by the thermistors in Cell B. In contrast, Cell A shows different baseline patterns with both positive and negatives values of dT/dt. However, both thermistors in Cell A as well as in Cell B always show exactly the same temperature changes. Theoretical calculations based on Equation 5 again predict only negative dT/dt values for both cells (Table 1).

Table 1 presents three time periods from each figure including a time when excess power was observed. These theoretical dT/dt values are always negative and change very slowly with time. The various episodes of positive dT/dt values for Cell A (Figs. 2,3,5,6) require the assumption of an excess power term as in Equation 2 and 4. Table 1 shows that the input power gradually increases during extended electrolysis. This could be caused by some loss of LiOD from the solution after many days of electrolysis as well as by the gradual passivation of the platinum anode and the palladium cathode. The unusual change in dT_A/dt in Table 1 late in the experiment (Figure 6) indicates an increase in passivation for the electrodes in Cell A.

Three other examples of excess power were observed for Cell A during this study that gave anomalous increases in the cell temperature despite the decreasing electrolysis power. These three separate increases in temperature for Cell A began at approximately 355 hours, 660 hours and 835 hours of electrolysis. Cell B, in contrast, always showed only the normal decrease in temperature with time. Cell A shows many examples of somewhat chaotic temperature changes of about ±0.2°C that cannot be explained by changes in the cell voltage. This indicates that many anomalous power changes of about ±10 mW occur in Cell A but not in Cell B. Beginning at about 860 hours of electrolysis, the electrodes in Cell A became quite passivated causing the cell voltage, P_{EL} and T_{cell} to increase with time. The increase in the cell temperature with time basically followed the behavior predicted by Equation 5. The neglect of the P_{gas} and P_{calor} terms in Equation 1 is not valid at cell temperatures higher than about 70°C.[4,5]

This anomalous excess power observed in Figures 2,3,5 and 6 could simply be the chemical recombination of the D_2+O_2 gases ($\gamma<1$), the electrochemical reduction of dissolved O_2 at the cathode ($\gamma<1$), or even some other chemical reaction or the release of stored energy. There must, however, be another power term to explain why the cell temperature increases when the applied electrochemical power is decreasing. Calorimetric errors due to insufficient mixing and

temperature gradients inside the cell can be dismissed since the two thermistors in each cell always show nearly identical temperature changes.

The most obvious explanation for the excess power is, of course, the chemical reaction of the electrolysis gases.[6] If the faradaic efficiency is not 100% ($\gamma<1.00$), then the excess power term is given by

$$P_x = (1.00-\gamma)E_H \cdot I \qquad (10)$$

Based on this equation, an excess power of 50 mW at I = 300 mA can be explained by $\gamma=0.89$; i.e. an 11% reduction in the faradaic efficiency.

Furthermore,

$$dP_x/dt = -E_H \cdot I \, d\gamma/dt \qquad (11)$$

hence the anomalous increase in the cell temperature could be produced by a decrease in the faradaic efficiency with time.

An accurate record of all D_2O additions to Cells A and B do not support explanations of the excess power based on faradaic efficiencies. The experimental rate of D_2O consumption was equivalent to 2.42 cc per day at 300 mA in Cell A and 2.49 cc per day in Cell B. Theoretical calculations based on Faraday's law yield 2.43 cc of D_2O consumption per day at 300 mA. The additions of D_2O were made every two or three days to within ± 0.2 cc based on syringe readings. There is no significant difference in the D_2O consumption for Cells A and B that is outside the range of experimental error.

The effect of the hydrogen+oxygen recombination in undivided electrolysis cells decreases significantly with increasing current density.[7] The heat generated by H_2+O_2 recombination reportedly[7] comprises only 0.03% of the cell input energy at 400 mA/cm^2. For the studies at 300 mA (480 mA/cm^2) shown in Figures 2, 3, 5 and 6, the expected excess power due to recombination would always be less than 1 mW based on the typical input power of 1.9 W for Cell A (Table 1). Therefore, chemical reactions of the electrolysis gases do not readily account for the anomalous increases in temperature for Cell A observed in Figures 2, 3, 5 and 6. Most Pd/D_2O electrolysis experiments at high current densities show faradaic efficiencies near 100%.[8]

It is difficult to propose a chemical process that satisfactorily explains the excess power effect for Cell A. For example, lithium ions may enter the palladium lattice and become reduced to lithium metal. Assuming a composition of PdLi$_{0.1}$, then the 0.18 mmole of lithium reacting with D_2O could sustain 50 mW of excess power for only 12 minutes. Furthermore, proposed reaction mechanisms involving the reduction of lithium ions to form Pd-Li alloys followed by the reaction of the alloy with D_2O to produce deuterium gas would be thermodynamically equivalent to the direct electrolysis of the heavy water. At the present time, it is appropriate to simply refer to the excess power as the Fleischmann-Pons effect. Their experiments show even larger anomalous increases in the cell temperature during Pd/D_2O+LiOD electrolysis.[9] It is important to note that anomalous increases in the cell temperature are also found in some of the Harwell calorimetric data,[10,11] but these interesting effects were not discussed in their publication.[12]

There is possibly some unknown energy storage mechanism in Pd/D_2O+LiOD electrolysis cells. The release of the stored energy could then produce the excess power effect shown in Figures 2, 3, 5 and 6 for Cell A. The mode of energy storage would produce a negative value for P_x (Eqs. 1,4), hence an abnormal decrease in the cell temperature should also be present. There was no

unusual decrease in temperature for either Cell A or Cell B from the start to finish of these experiments. Nevertheless, a very gradual storage of energy would likely be difficult to detect. Any proposed energy storage mechanism, however, would have to explain why such an effect operates in Cell A and not in Cell B. It is indeed a challenge to find an acceptable scientific explanation for the Fleischmann-Pons effect.

The basic characteristics of the excess power effect in Cell A are displayed by Figures 2, 3, 5 and 6. First, there was never any large, abrupt increases in the excess power such as that observed in Figure 6 by suddenly increasing the cell current. The onset of excess power was always somewhat gradual. The most rapid increase in the excess power was observed at 660 hours where there was a step-like increase in temperature of 0.4°C. Due to the time constant of these calorimetric cells, the thermistor readings can only display power changes averaged over about 15 minutes. The addition of D_2O rapidly cooled the cell as seen in Figure 2 and generally seemed to decrease the excess power effect. Each pattern for the onset of excess power shows different characteristics. The time period following the last D_2O addition to the beginning of the excess power effect ranges from 5 hours to 37 hours. There are no obvious triggering actions for the excess power effects in Figures 2, 3, 5 and 6. It could be postulated that the higher cell temperature produced in Figure 2 by increasing the cell current triggered the excess power effect and that the cooling effect of D_2O additions diminished the excess power. However, other cell current increases such as in Figure 6 produced no significant increases in the excess power effect. Positive feedback effects produced by increasing the cell temperature have been previously reported by Pons and Fleischmann.[9] Our experiments typically gave 50-100 mW of excess power for Cell A and no significant excess power for Cell B. The excess power density of 3-6 W per cm^3 of palladium obtained in this study is about the expected value for the applied current density.[2] The simplicity of our calorimeters as well as our calorimetric analysis should make it relatively easy for other laboratories to observe the excess heat effect that has created a lingering scientific controversy.[13]

Summary

The main feature of the Fleischmann-Pons effect is the production of excess power during Pd/D_2O+LiOD electrolysis. These experiments characterize the excess power effect during seven different occasions by the anomalous increase in the cell temperature despite the steadily decreasing input power. There were no clear triggering events for the excess power production. The excess power effect is generally small (50-100 mW) in these experiments, but it is readily detected by the increase in the cell temperature. The onset of the excess power generally develops in a chaotic but gradual manner. Two thermistors were used in each cell, and both always show almost exactly the same temperature changes. Reactions involving the electrolysis gases are the most likely chemical explanation for the excess power. Measurements of the faradaic efficiencies, however, do not support this hypothesis. Normal behavior was always observed for a similar experiment conducted as a control.

Supporting Information Available: Average voltages measured for Cell A and B during the same time periods shown in Figures 2, 3, 5 and 6 are presented in Tables 2S, 3S, 5S and 6S. This material is available free of charge via the Internet at http://pubs.acs.org.

References and Notes

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13. This controversy is due to the difficulty in reproducing the excess heat effect and the dependency of this effect on unknown variables within the palladium metal- Our best cathode materials were palladium-boron alloys prepared by Dr. M.A. Imam of the Naval Research Laboratory (Ref 5).

TABLE 1: Experimental Electrolysis Power and Theoretical Temperature - Time Slopes For Cells A and B

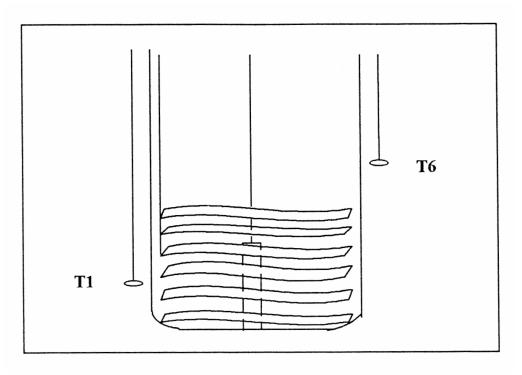| time (hrs)	P_{EL-A}(W)	P_{EL-B}(W)	dTA/dt (Khr--1)	dTB/dt (Khr-1)
Figure 2				
295.28	1.747	1.378	-0.028	-0.016
307.00	1.633	1.318	-0.033	-0.018
318.12	1.555	1.246	-0.026	-0.016
Figure 3				
474.50	1.919	1.543	-0.043	-0.019
487.00	1.762	1.468	-0.051	-0.020
502.78	1.648	1.378	-0.054	-0.022
Figure 5				
594.50	1.978	1.573	-0.060	-0.027
607.28	1.852	1.486	-0.065	-0.030
622.70	1.717	1.390	-0.072	-0.033
Figure 6				
783.25	2.138	1.567	-0.010	-0.030
793.70	2.051	1.498	-0.011	-0.033
814.78	1.868	1.348	-0.011	-0.039

Figures

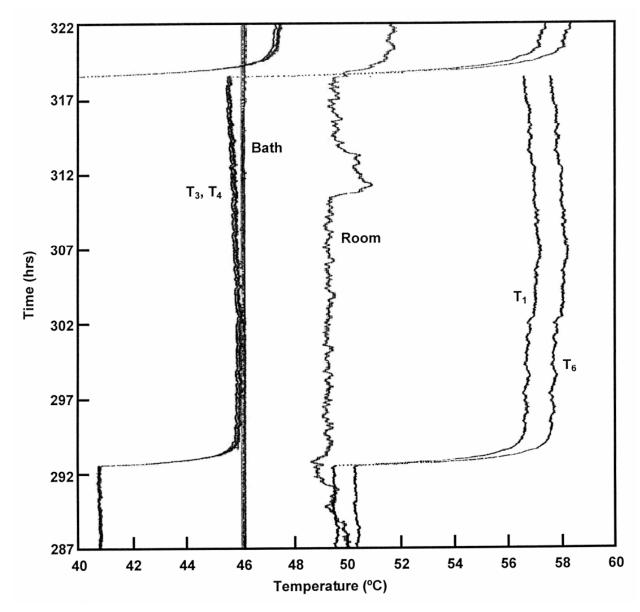**Figure 1. Schematic of positioning for thermistors T_1 and T_6 relative to the palladium**

Figure 2. Cell temperatures versus time showing the first episode of excess power in Cell A (T_1,T_6) and the continuous normal behavior for Cell B (T_3,T_4). The cell current was increased from 0.25028 A to 0.30021 A at 292.7 hours and D_2O was added to both cells at 318.7 hours. The room and bath temperatures are for a 20-25°C temperature scale.
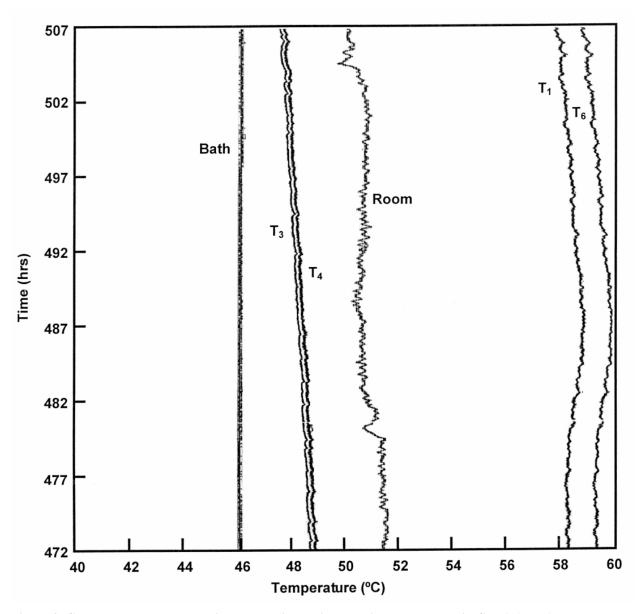
Figure 3. Cell temperatures versus time presenting an increase in excess power in Cell A (T_1,T_6) and normal behavior in Cell B (T_3,T_4). There is an increase in the temperature of Cell A beginning at 477 hours. I = 0.30024 A. The room and bath temperatures are for the 20-25°C scale.
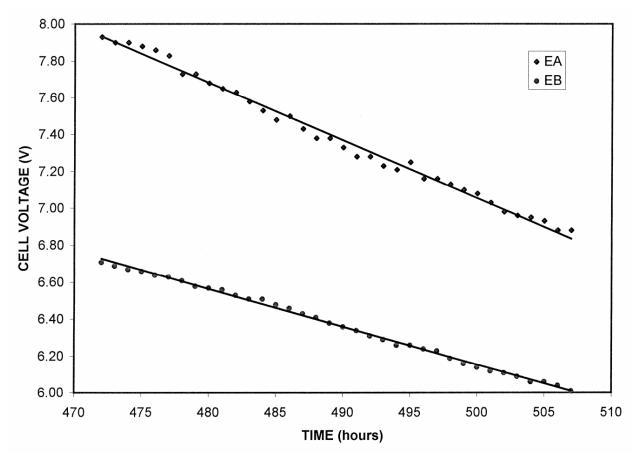
Figure 4. Average voltages measured for Cells A and B during the same time period displayed in Figure 3. The lines have slopes of -0.0314 V/hr (R^2=0.9919) for Cell A and -0.0205 V/hr. (R^2=0.9971) for Cell B.
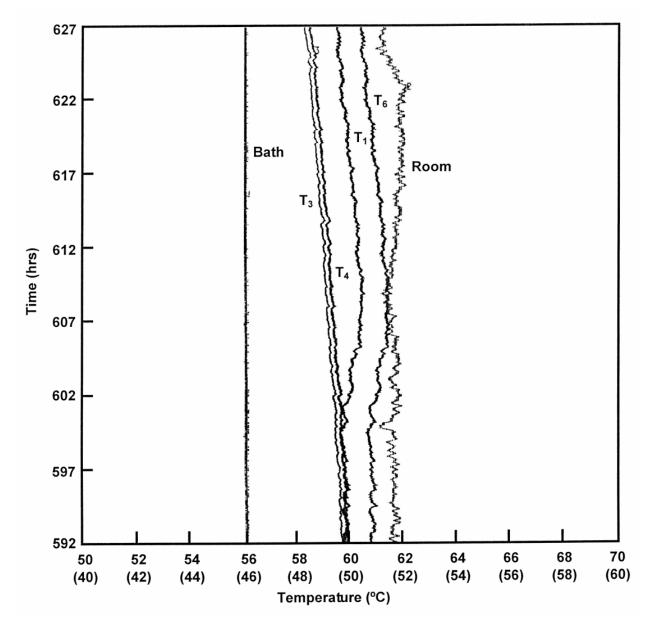
Figure 5. Cell temperatures versus time for Cells A (T_1, T_6) and B (T_3, T_4). There is an anomalous increase in the temperature for Cell A beginning at 600 hours and continuous normal behavior for Cell B. I=0.30021 A. Two temperature scales are used since Cell A was more than 10°C warmer than Cell B. The room and bath temperatures are for a 20-25°C temperature scale.
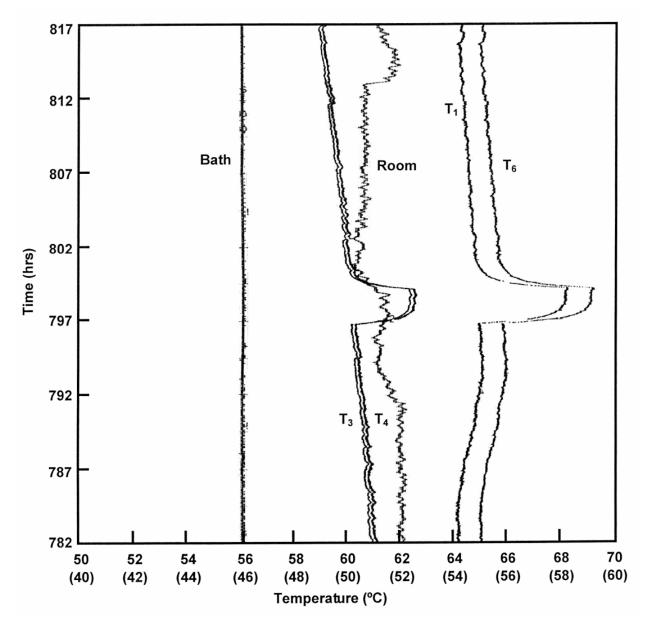
Figure 6. Cell temperatures versus time for Cells A (T_1,T_6) and B (T_3,T_4). There is an increase in the temperature of Cell A beginning at 784 hours and continuous normal behavior for Cell B. There is also a calibration based on increasing the cell current from 0.30022 A to 0.32028 A for a period of 2.33 hours. Two temperature scales are needed since Cell A was more than 10°C warmer than Cell B. The room and bath temperatures scale is 20-25°C.