TRIGGERING OF HEAT AND SUB-SURFACE CHANGES IN Pd-D SYSTEMS

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INTRODUCTION

More than four years after the first reports of chemically stimulated nuclear reactions, the triggering of heat evolution and the production of associated nuclear debris is still a highly uncertain matter. This is so both as to the duration of the switch-on time and, indeed, whether a given electrode will commence to show nuclear activity within 500 hours of the beginning of electrolysis.

In the present study, 3 methods of triggering anomalous heat are described; the changes in the sub-surface of palladium during the evolution of D₂ or H₂ are described as a function of potential, temperature and time.

Finally, these results are brought up against the present theories of heat production in metals.

1. EXPERIMENTAL

1.1. Electrochemical Stimulation

Hodko and Bockris (1) presented a pulsing study in the 1991 Meeting in Como. In that study, emptying and filling the electrode with D (D/Pd from 0.3 to 0.83) gave rise to the initiation of heat bursts. The present study examines the efficacy of the Takahashi conditions.

Electrolysis Cell: The cell was made of plexiglass of 5 mm thickness. It had outer dimensions of 120 mm x 75 mm x 100 mm. It was provided with a lid of the same material with provisions for introducing the electrodes, cooling coil, etc. The cell housed a glass cooling coil with six spirals through which chilled water maintained at 20±0.01°C in an external cooling system (Haake, Model A81) was circulated at a constant rate of ~5 liters per minute to cool the electrolyte. This arrangement ensured an efficient exchange of heat between the electrolyte (source of heat) and the chilled water coils (sink). Thermal equilibrium was reached within 30 or 40 minutes.

Electrodes: A 1 mm thick, 25 mm square plate of palladium metal served as the cathode. This material was received as a gift from Tanaka Kikinzoku Kogyo, Japan, and was similar to the Pd used by Takahashi (2). The material was 99.97% pure Pd. It was used as such without pretreatment, supported by two small polyethylene blocks on either side, and surrounded by an anode of 0.5 mm thick platinum wire (Johnson Matthey, Puratronic Grade) which was wound round the blocks (6 turns with ~5 mm pitch) keeping the anode-cathode distance at ~10 mm at both sides. The platinum contact wire to the cathode and the platinum anode lead wire were both covered with Teflon tape to ensure electrical isolation. The electrolyte was 0.29 M LiOD, obtained by dissolving lithium metal (Johnson Matthey, USA, 99.9% pure) in D₂O (ISOTEC, Inc., USA, 99.9 atom percent pure) in an atmosphere of argon. Fig.1 depicts the assembly.

Power Supply: A constant current assembly was used.

1 An arbitrary time at which the examination of an electrode is often terminated
Electrolysis Conditions: The cell was filled with 650 ml of electrolyte. The level was kept at 1 cm below the top of the cell. A thermometer was placed near the cooling coil and a thermistor (matched earlier with the thermometer) between the electrode assembly and the cooling coil. A thermistor thermometer (Omega, USA), with both analog and digital readout, was used to record the temperature of the electrolyte during the experiment. The electrolyte level was maintained within 1 cm of the initial level by replenishing periodically with D$_2$O. A 1 ml sample was withdrawn every 2 or 3 days to check for tritium. The temperature in the laboratory remained at 20±1°C, except for a period of about 10 days when there was a breakdown in the temperature control and measurements were not taken.

The experimental procedure involved a 'preloading phase' employing a saw-tooth current mode. The current was cycled linearly from 0.25 A to 4.2 A (0.02 - 0.336 A.cm$^{-2}$) in a 20 minute period. This was continued for 9 days. After this, the measurement phase was carried out in the Low-High (L-H) current mode, in which the current was kept constant at 0.25 A and 4.2 A alternately for 6 hour periods. The current cycling modes are shown in Fig.2. The experiment was carried out over a 61-day duration.

Calibration procedure for excess heat calculation: Calibration by Joule heating was adopted. The experiment was, therefore, started with an initial test operation. Currents of 1A, 3A and 5A were passed through the cell for 40 minutes each and the electrolyte temperature and cell voltages noted. Assuming that there was no excess heat generation at this stage of the experiment, the temperature rise, $\Delta T$, during this period was taken as a measure of the calorimeter calibration. A temperature increase ($\Delta T$) of 4.70°C was recorded for 50W Joule heating. The "zero power" line, corresponding to 20°C, was taken as constant in view of the steady room temperature. A value of 0.094°C was thus obtained for $\Delta T$ per watt input power over the experimental range.

Determination of the D/Pd ratio: The degree of loading of deuterium into palladium was monitored 'in situ' by means of four probe resistivity measurements. Four platinum leads were spot-welded to the cathode at appropriate locations and the resistance was continuously recorded by means of a digital micro-ohmometer (Model DMO-350, Tacrad Inc., Canada) and stored into an IBM-286 compatible computer. The ratio of the initial resistance, $R_0$, before the electrolysis to the resistance, $R$, at any time during the experiment was plotted against time. To calculate the value of D/Pd from this plot, use was made of a previously constructed calibration graph of $R/R_0$ vs. D/Pd (in which the D/Pd determination had been made on the basis of coulometric measurements).

Measurement of the potential of the cathode: A luggin capillary contact tube was placed within 1 mm of the cathode and the cathode potential was measured with respect to a saturated calomel electrode (S.C.E.).

Measurement of tritium: During the experiment, the tritium activity in the electrolyte was being monitored every two or three days. A 0.5 ml sample was withdrawn, mixed with 6.5 ml of Optiphase, "HiSafe-3" scintillation cocktail and the activity of this solution was counted for 10 minutes. A Wallac 1410 Liquid Scintillation Counter was made use of for this purpose.

1.2. Radio-Frequency Stimulation

Electrolysis cell: A Johnson Matthey palladium foil cathode of 99.9% purity and dimensions 11.9 x 12.5 x 1 mm was held between two teflon holders and was surrounded by 7 turns of 20 gauge platinum wire anode (Fig.3.). This electrode assembly was contained in a 10 cm tall cylindrical glass cell of 25.4 mm internal diameter. 15 ml of 0.3 M LiOD in D$_2$O was taken in the cell for electrolysis.

The temperature was measured by means of a thermistor. The RF power was applied to the cell by means of a 20 gauge copper wire that was wound around the cell in 15 turns in a typical NMR configuration.

RF Generator: A Rohde & Schwarz generator, with a frequency range 100-1000 MHz was used in conjunction with a RF amplifier (ENI, Model 603L). The RF power (6 - 100 mW), mentioned in the experiment, is the maximum power delivered assuming 100% coupling efficiency. The efficiency of the RF-coupling to the cell was not measured.

Measurement of heating due to application of RF power: Prior to the stimulation experiment.
different RF power ranging from 100 mW to 1 W were applied to the cell containing 15 ml electrolyte. The rise in temperature was 5.2 °C per Watt.

Before the RF power was coupled to the cell, the palladium cathode was charged with deuterium by carrying out the electrolysis at 0.25 A for 139 hours. The D/Pd ratio was expected to have reached > 0.8 by this procedure. At this stage, maintaining the charging current unchanged, the RF power of 6 to 30 mW was applied to the cell at 365.608 MHz.

1.3. Magnetic Stimulation

The magnetic field was applied by means of two different permanent magnets:

Magnet 1: A horse shoe magnet of 200 Gauss was placed around the cell (Fig. 4.). Magnet 2: Two 1" diameter disc magnets of Neodymium were placed in attractive mode on opposite sides of the cell; the field strength was measured at the Los Alamos National Laboratory as 800 Gauss in an empty cell (Fig. 5.).

This experiment was carried out at 3.5 °C in a small refrigerator. The connecting leads were run through small holes drilled on the sides of the refrigerator which were then sealed with epoxy. The lower temperature was chosen to facilitate deuterium loading.

The cathode was 99.9 % pure Engelhardt palladium which had been cold rolled. Its dimensions were 12.5 x 12.5 x 0.28 mm. It was charged with deuterium at a current of 80 mA [cell voltage was 2.64 V] in 15 ml of 0.3 M LiOD/D₂O for 48 hours before applying the magnetic stimulation. It is probable that this treatment corresponded to a D/Pd ratio of > 0.8.

1.4. Materials Science

A standard three electrode electrochemical system was used in this study with Palladium as the working electrode and Platinum as the counter electrode. Working electrodes were in the form of a foil, 50 μm x 10 mm x 5 mm, purchased from Johnson Matthey as 99.975 % pure Palladium. They were used as cathodes in the electrolysis of 0.1 M KOH or 0.1 M KOD medium with saturated calomel electrode as a reference electrode. Experiments were performed by varying the time of electrolysis and overpotential at room temperature. The potential range studied was from the reversible potential to overpotential of \( \eta = -1.0 \) V. The applied potentials were maintained for different periods of time, varying from 0.5 hour to 6 weeks.

Each experiment was carried out with a fresh electrode. Following electrolysis at certain conditions, the electrode was washed with purified water and etched in 30 % 1:1 HNO₃ + HCl mixture(3). A preliminary investigation had been carried out prior to the experiments to observe the effect of duration of etching on the depth of the surface exposed. Measurements were carried out for durations ranging from 1 to 5 minutes. After etching, the solution was analyzed by ICP and by knowing the area of etched surface it was calculated that one etching procedure corresponds to the depth of 800 Å per minute. An etching time of 2.5 minutes, corresponding to 2,000 Å was chosen for our investigations. The etched surfaces were then subjected to examination by means of Differential Polarization Interference Contrast Microscopy (DPICM) and Scanning Electron Microscopy (SEM).

DPICM was chosen because it is a technique capable of imaging minute surface structures in differentiating color. At magnifications of 100 and 600 that have been used in this study, it affords greater observability of patterns having dimensions of the order of c. 0.5 mm with a resolution of 2 and 0.4 μm. SEM was chosen due to its capability to image the surface in three dimensions (depth of field is large and there is shadow relief effect of the secondary electrons). At magnifications of 1000 and 50,000, the resolution of 0.2 and 0.004 μm affords observations of crystal grains.

The microscopy was repeated after re-etching the surface 5 successive times, up to a depth of 1 μm.
2. RESULTS

2.1. Electrochemical Stimulation

The cell voltage during the L-period was almost steady at about 3.6V. The electrolyte temperature was also constant at 20\(\pm\)0.05°C, indicating no excess heat during these periods. There was an appreciable increase in the cell voltage during the H-period from the initial value of 20V to about 31V towards the conclusion of the experiment and the corresponding electrolyte temperatures also showed a rise with time.

The fall in the electrolyte level due to electrolysis had an effect on the cell voltage during the H-periods and on the corresponding electrolyte temperature. Therefore, only the values taken within a short time -say 1 or 2 hours- after the initial level was restored by replenishment with \(\text{D}_2\text{O}\) were taken into account for the calculation of excess heat. This seemed to be reasonable in view of the fact that the thermal equilibrium between the cell and the circulating water was always attained within 40 minutes.

If the cell voltage was \(V\) and the current \(I\), then the input power for Joule heating, \(Q_{\text{in}}\), would be

\[
Q_{\text{in}} = (V-1.54)\cdot I \text{ watts} \quad (1)
\]

The output power, \(Q_{\text{out}}\), was calculated from the observed rise in the electrolyte temperature, \(\Delta T\), by means of the calibration done earlier, i.e.,

\[
Q_{\text{out}} = \Delta T + (0.094) \text{ W} \quad (2)
\]

The excess power, \(Q_x\), was then arrived at as

\[
Q_x = Q_{\text{out}} - Q_{\text{in}} \quad (3)
\]

That is

\[
Q_x = [\Delta T+0.094] -[(V-1.54)\cdot I]. \quad (4)
\]

During the 50 days of the L-H operation, the rise in the electrolyte temperature in the H-periods was always higher than that calculated for Joule heating.

The present method gave rise to an estimate of the "average" input and output power(4) to within \(\pm5\%\). During the 50 days that the cell was run on L-H current mode, an average excess power of approx.18 watts(i.e., 28.8 Watts/cc of Pd) was observed during the H-periods (Fig.6.) but no excess power during the L-periods; this amounts to 39 MJ of excess energy. The total input energy during the experiment(on the basis of 26.5V for H- and 3.6V for L- periods) amounts to approx.230 MJ(approx.17 \%)

The degree of deuterium loading into palladium, the D/Pd ratio, was being continuously monitored during the experiment and reached 0.83 after 15 hours and remained almost constant thereafter (Fig.7.).

The potential of the palladium cathode was measured to determine the overpotential value. The value of the cathode potential was -1.4 V vs. S.C.E. at low current(0.25 A). The pH of the solution being 13.5, the reversible potential was calculated as -0.81 V. The resistance of the electrolyte was very nearly 1 \(\Omega\), so that the IR drop would contribute 0.25 V. This meant that the overpotential was about -0.34 V. At high current mode operation (4.2A), the potential read -5.93 V vs S.C.E. and the overpotential value was estimated as -0.92 V.

The tritium activity generated during the experiment was counted periodically as described earlier. It was observed that the activity rose to about 3 times above the background and remained constant.

2.2. RF Stimulation

After the palladium has been sufficiently loaded with deuterium, RF power was applied to the cell, maintaining the d.c. The temperature of the cell started rising within 10 minutes after the application of RF. The rate of increase in temperature was proportional to the power of the RF (Fig.8.). The electrolyte returned to room temperature within 10 minutes when the RF signal was turned off.

Two other frequencies, viz., 533.688 MHz and 81.924 MHz were also found to trigger exothermic effect in deuterated palladium. The heating effect was specific to the stated frequencies and disappeared at other frequencies. Also, such a heating effect was not observed in a H\(_2\)O system.

The experiment was carried out in an open cell configuration. The cell was not thermostated. The ambient laboratory temperature, however, was constant to within \(\pm0.5\degree\)C. The excess power is shown in
Fig. 9 shows a function of frequency. Heating by RF itself was precluded by the following observation. The exothermic response in the cell to the application of a 1 Watt amplified signal at 81.924 MHz was that, after an initial rise, the temperature starts declining and finally falls to room temperature after 120 minutes. The RF power remained constant during this period. Application of 1 Watt RF power, by itself, would have raised the temperature of the 15 ml electrolyte by about 5 °C but this increase should have remained constant and not decreased if the effect was due to the application of RF power.

There were two other observations during this experiment: 1) The cell voltage began to fluctuate just before the manifestation of the exothermic effect. The steady applied voltage of 3.6 V fluctuated between 2.65 and 4.10 V for about 1 to 3 minutes and then settled down at 3.6 V. 2) During the time when the fluctuations occurred, the cell temperature initially decreased by 0.5 to 1.5 °C before starting to rise [such observations have been made earlier(1&5)].

2.3. Magnetic Stimulation

After the cathode had been charged with deuterium for 48 hours at a current of 80 mA, the cell was placed in the field of a permanent magnet of 200 Gauss strength. The cell electrolyte temperature rose to 5 °C (Fig. 10.) after 230 seconds. After 576 seconds, the magnet was replaced by two, one inch Neodymium magnets with a 800 Gauss field placed as described earlier. The temperature immediately started increasing and reached 13.5 °C in about 15 minutes and remained constant. The temperature returned to 3.5 °C when the magnet was removed.

2.4. Materials Science

The microscopic investigations revealed some characteristic morphological changes that were brought about in Palladium by electrolysis under different experimental conditions. Throughout all these studies, including electrolysis for six weeks and at overpotentials up to -1.0 V, no changes were observed on the electrode surface by SEM. The changes were manifest on the subsurface, the observations beginning at a depth of 2,000 Å that was obtained after etching. Regular patterns which resembled hexagons (Fig. 11.) appeared. Similar hexagons have been mentioned by Brooks et al (6). Microvoids, in the form of black spots of 2,500 Å dimensions, were seen along the sides of these hexagons. There was a progression in the formation of the hexagons and the microvoids with variations in overpotential, time and temperature. These changes decreased exponentially with depth (Fig. 12.) and were difficult to observe at a depth below 1 μm.

2.4.1. Effect of H₂O in place of D₂O

Examination of sub-surface changes obtained by electrolyzing both in KOH and KOD suggests that the frequency of microvoid formation at η = -0.35, is 3 to 5 times greater in D₂O than in H₂O. However, at η = -0.50 V, the frequency of microvoid formation in H₂O seems about the same as in D₂O.

2.4.2. Effect of overpotential

To study the effect of overpotential on sub-surface structural changes, electrodes were subjected to different overpotentials at 20 °C for 0.5 hours. At overpotentials more positive than -0.20 V, the sub-surface remained unaffected.

The first "visible" changes, detected by means of SEM at a magnification of 10,000 (i.e., microvoids were >100 Å), were observed at overpotential of -0.35 V. At η more negative than -0.35 V, the hexagon patterns, mentioned above, could be observed. The sides were approximately 3 μm in length. However, such patterns were visible only on 2 - 3 % of the total sub-surface and were scattered.

Black spots or microvoids at the nodes of the "hexagons", i.e., at the intersection of grain boundaries were observed (Fig. 13.a.). There were up to 6 microvoids per hexagon cell. With increase of overpotential to η = -0.50 V, the changes on the sub-surface extended to ~5 % of the total surface and the microvoids per hexagon increased in number. from 6 at -0.35 V to 30 at -0.50 V (Fig. 13.b.). At more negative overpotentials, η = -1.00 V, ruptures appeared on the sub-surface of the electrode (Fig. 13.c.).

1-6
Intensive and extensive ruptures appeared on the sub-surface. The intensive ruptures were ~ 20 μm in size and covered approximately 7% of the surface. Extensive ruptures were 5-10 μm in size and were distributed over ~ 20% of the sub-surface. These ruptures were grouped into patches, each having the dimensions of ~0.5 mm.

2.4.3. Effect of time
The effects observed on the palladium electrode upon electrolysis for various periods of time are shown in Figs. 14.a-d. To evaluate the effect of time over the range of 0.5 hour to six weeks, an η of -0.35 V was maintained at a constant temperature of 20 °C.

For the shortest period studied, t = 0.5 hours (Fig. 14.a), hexagons were scattered over 2 to 3% of the total surface. As the time was prolonged to 3 hours, the hexagons increased and tended to group together to form "islands", Fig. 14.b. Each island contained between 20 and 50 unit hexagons and the hexagons spread to 10% of the electrode area at 3 hours (Fig. 14.b) and 20% at 10 hours (Fig. 14.c). Here, islands, each having between 200 and 300 hexagon units that were separated by about 20 to 25 μm were formed and tended to "cluster" into groups. These clusters had dimensions of the order of 500 μm and were seen about 1 mm apart. Fig. 14.d. shows the situation after 100 hours of electrolysis.

When the Palladium electrodes were subjected to electrolysis for 6 weeks (1000 hours) the whole sub-surface was "damaged" and the hexagonal units can no longer be seen.

2.4.4. Effect of temperature
Experiments were conducted at -0.35 V for 0.5 hour at 20 and 50°C (Fig. 15.a. and b.). Island formation at 50 °C resembled the behavior at η = -1.0 V at 0.5 hours.

3. DISCUSSION

3.1. Electrochemical Stimulation
The electrochemical stimulation experiment, performed in a manner similar to that of Takahashi, shows an unmistakable generation of excess energy. As Takahashi has observed, excess heat is manifest within two days after High-Low current pulsing mode is started. There are, however, some observations that differ from those reported by Takahashi:
1. The cell voltage during the L-periods remained constant at 3.6V. The temperature of the electrolyte during these periods was close to 20 °C in this experiment, suggesting no excess energy. The current during the L-periods was 0.25 A which meant that the current density was 0.02 A.cm⁻². Earlier observations (7 to 10) have indicated that this current density is too low to manifest excess heat. Takahashi (2), however, reports "significant temperature increases even for L-periods".
2. There was an excess energy of approx. 18 W during the H-periods throughout this experiment. This is lower than what is quoted by Takahashi. It is to be mentioned here that though Takahashi first reported a relative excess of ~70% (2), this value was much lower (~30%) when he repeated the experiment (4).
3. Though there was a constant excess energy during the H-periods, there was no "surface boiling" that was reported in (2); again, this effect was absent in the subsequent experiment (4).
4. The degree of loading of deuterium into the cathode was continuously monitored and its value was 0.83. Takahashi did not measure but assumed it to be ~0.9. The overpotential was -0.92 V.

3.2. Pulsing and its Mechanism
It has been pointed out by Gittus and Bockris (11) that the solubility of H in Pd is greater than that of D and that, because of the relation between C₂ and local stress, the solubility is particularly high at dislocations. It follows that reactions which may be the origin of the heat production phenomenon will be likely to occur at high concentration points near dislocations within...
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\[ C_0 = C_0 e^{\frac{aV_n}{RF}} \] (5)

Now, there is always some H in the solution and in an unannealed electrode; thus, H may block dislocation positions from being occupied by D. Hence, pulsing, during which the D/Pd is varied up and down, may be seen as dissolving some of the H from its blocking positions on the dislocations - it will be filled increasingly with D (Fig.16.).

This suggests that deep anodic pulsing to bring D/Pd towards zero followed by rapid cathodic pulsing to bring it up again, as shown in Fig.17 [cf.(1)], is the most likely switch-on mechanism.

3.2. Radio-Frequency Stimulation

The Deuterium nucleus in the Pd-D is partly ionic and partly covalent(13). Especially when the D/Pd ratio is greater than 0.8, there are effectively neutral D atoms in the lattice which can 'sense' the presence of the orbital electron. This results in the generation of a fine structure magnetic field at the deuterium nucleus (14).

Calculation of Fine Structure Magnetic Field

The fine structure magnetic field, that is, the field generated at the deuterium nucleus by the orbital motion of the electron was calculated from the following equation (15)

\[ H = \frac{Q}{R^2} \times \frac{V_{el}}{C} \] (6)

where

- \( H \) = Magnetic field in Gauss
- \( Q \) = Electronic charge \( (4.802 \times 10^{-10} \text{ stat.coulomb}) \)
- \( R \) = Bohr radius \( (5.3 \times 10^{-9} \text{ cm}) \)
- \( V_{el} \) = Orbital velocity of the electron \( (2.2 \times 10^8 \text{ cm/Sec}) \)
- \( C \) = velocity of light \( (2.997 \times 10^{10} \text{ cm/Sec}) \)

The magnetic field was calculated as \( 1.2535 \times 10^5 \text{ Gauss} \) or 12.535 Tesla.

Calculation of the Frequency of Stimulation

The frequency \( v \) is required to resonate and induce spin transitions in a neutron, proton or a deuterium nucleus (NMR Frequency) in a magnetic field of \( H \) is given by the equation (16)

\[ v = \frac{\mu \beta H}{h.1} \] (7)

where

- \( v \) = Frequency in Hz
- \( \mu \) = magnetic moment \( (0.8574 \beta \text{ for deuterium, } 2.7927 \beta \text{ for proton and } -1.9132 \beta \text{ for neutron}) \)
- \( \beta \) = Nuclear Magneton \( (5.05 \times 10^{-24} \text{ Erg/Gauss}) \)
- \( H \) = magnetic field \( (1.2535 \times 10^5 \text{ Gauss}) \)
- \( h \) = Planck's constant \( (6.625 \times 10^{-27} \text{ Erg.Sec.}) \)
- \( I \) = Spin Number, 1 for Deuterium and 0.5 for proton and neutron

\( v \) was calculated as \( 81.924 \text{ MHz} \) for deuterium, \( 365.608 \text{ MHz} \) for neutron and \( 533.688 \text{ MHz} \) for proton.

Thus, the exothermic effect observed at these specific frequencies may be related to the spin orientations brought about within the deuterium nucleus in the PdD lattice. It is not clear at the moment as to how and why these spin orientations cause excess heat.

It is interesting that the excess heat, caused by RF stimulation, reaches a maximum value and, after a certain time, falls to zero. A possible explanation is that the RF stimulates only the deuterium nucleus at the near surface of Pd. It is well known that, due to the 'skin effect', high frequency alternating currents will be felt only up to a certain depth (called 'skin depth') which is given by (17)

\[ d = \frac{1}{(\mu \sigma \omega)^{1/2}} \text{ (metres)} \] (8)

where,

\( d \) = skin depth
σ = electrical conductivity of the material (mho/metre)
μ = permeability of the material (Henry/metre)
f = frequency of the a.c. (Hz)

For the frequencies that have been applied in this stimulation experiment, it can be shown that the depth to which the RF would have been effective is only ~ 0.007mm (7μm).

Summary of Observations
1) RF energy at three specific frequencies triggers exothermic effect in deuterated palladium without the presence of an external magnetic field.
2) A similar effect is manifest in the presence of a magnetic field of 200 Gauss or greater.
3) The triggering is effective even at electrolysis current densities below 100 mA/cm².
4) Sufficient deuterium charging of the palladium (D/Pd probably greater than 0.8) is a necessary prerequisite to the triggering effect.

3.3. Materials Science
3.3.1. Initiation of subsurface damage with respect to overpotential and pressure

It is well known that a pressure theory exists for the treatment of the switch-on potential for cracking (18). The D evolved on the electrode surface is assumed to be in equilibrium with D₂ in voids. In so far as the pressure (~ fugacity) in the voids exceeds the spreading pressure, the crack will spread and damage will be detectable.

In this study, damage was found to begin at an overpotential of -0.35 V (Fig. 18.). In addition, the Tafel line measurements show a sharp increase in slope in this region which may be assumed to correspond to the saturation of the electrode surface and a change of mechanism to one in which the coupled discharge-chemical combination

\[ M + D₂O + e \xrightleftharpoons{R.D.S.} R.D.S. \xrightarrow{R.D.S.} M - D + OD^- \]  

\[ M - D + M - D \rightarrow 2M + D₂ \]  

changes to fast discharge slow chemical combination

\[ M + D₂O + e \xrightarrow{R.D.S.} M - D + OD^- \]  

\[ M - D + M - D \xrightarrow{R.D.S.} 2M + D₂ \]  

In the latter case, the fugacity of D₂ is given by

\[ f_{D₂} = e^{\frac{-2\pi F}{kT}} \]

The condition for the beginning of damage can then be shown to be

\[ e^{\frac{-2\pi F}{kT}} \geq \left( \frac{16}{3} \frac{\nu Y}{l} \right)^{\frac{1}{2}} \]  

and

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\[ \eta_{\text{crit}} = \frac{RT}{2F} \ln \left( \frac{16 \gamma Y}{3} \right)^{\frac{1}{2}} \]  \tag{15} 

where \( \gamma \) is the surface tension of Pd, equal to 1.5.10^3 dynes.cm.^2, \( Y \) is the Young's Modulus, equal to 0.45.10^{12} dynes.cm^2 and \( l \) equal to 10^6 cm. is the length of the initial crack, assumed to be lens-like. Therefore, \( \eta_{\text{crit}} = -0.30 \) V which is in excellent agreement with the observed value of the beginning of cracking.

3.3.2. Effect of overpotential

Figs.19 and 20 show the effect of overpotential on the percent of change of the electrode surface after 30 minutes of electrolysis. Taking into account both the extensive and intensive type of ruptures, an exponential behavior of percent change is followed, Fig.19. However, by plotting the log of the percent of change of the electrode surface as a function of overpotential, Fig.20, a linear relationship with the slope of 295 mV/decade is obtained. Thus, for the surface to change by one order of magnitude, it is necessary to increase potential for \( \sim 300 \) mV. This is in agreement with the previously observed change in Tafel slope from \( \sim 150 \) to \( 350 \) mV/decade upon reaching the overpotentials of \( \eta > -0.35 \) V.

3.3.3. Effect of time

In Fig.21, the percent change of the electrode subsurface as a function of time of electrolysis is shown. Two linear relationships are obtained for the time period of 0.5 to 10 hours and from 10 to 1000 hours of electrolysis. Thus the largest subsurface changes occur during the first ten hours of electrolysis.

3.3.4. Hexagon formation

Hexagons appear as a result of plastic deformation and slip. The disappearance of hexagons, at higher overpotentials would follow as a consequence of increased loading and hence fracture(19,20).

3.3.5. Time to reach saturation of the first 10\mu m layer

There is much evidence in Cold Fusion Phenomena(21) which suggests that the essential phenomena occur within the first 10\mu m of the surface. One has, therefore, to calculate the time for saturation at that depth.

The two main shapes of electrodes used are a) rectangular and b) cylindrical. For a planar surface, in which there is an initial surface concentration, \( C_0 \), the concentration \( C_x \), at a distance \( x \) from the surface at time \( t \), is given by

\[ C_x(t) = \frac{2C_0^x}{\sqrt{2Dt}} e^{-\frac{x^2}{4Dt}} \]  \tag{16} 

or

\[ \frac{C_x(t)}{C_0} = \frac{2x}{\sqrt{2D}} e^{-\frac{x^2}{4Dt}} \]  \tag{17} 

This equation can be solved for different \( t \) at \( x = 10^{-7} \) cm., when \( D = 10^{-7}, 10^{-8} \) and \( 10^{-9} \) cm^2 sec.

(22). Correspondingly, for a cylindrical electrode.
where \( J_0 \) and \( J_1 \) are the roots of Bessel's differential equation. The concentration profiles for different times of electrolysis, calculated for both the types of electrodes, are shown in Fig.22a. and b. In fact, the switch on times are generally longer than these times shown here.

3.3.6. Electronic state of dissolved hydrogen

Pure palladium is paramagnetic. A number of experimental studies (23) indicate that upon absorption of hydrogen there is decrease in paramagnetism. Also, results of different authors (23) agree for the value of \( \text{H/Pd} \approx 0.65 \), at which the solids become diamagnetic. Thus, after \( \text{D/Pd} \) ratio of 0.65, electrons are not filling the d-band of the host metal and magnetic susceptibility is zero. Due to this, electrons are now more localized in regard to protons. After reaching the D/Pd ratio of 0.80, all octahedral places in the lattice are occupied and those in tetrahedral positions are now available (Fig.23.). As a consequence, hydrogen is much less mobile and it can be speculated that a covalent bond between Pd and H is starting to form.

Wipf (24) has compiled some experimental data on effective charge number of hydrogen isotopes in polycrystalline Pd (Table 1).

3.3.7. Charge on Deuterium

There are two methods of determining the charge on D\(^+\). One can measure the diffusion coefficient and then (e.g., with tritium marking) obtain the mobility. The Nernst-Einstein Equation enables one to obtain the charge, \( Z \). Alternatively, one may determine \( Z \) from the magnetic susceptibility.

There is a linear decrease of magnetic susceptibility to \( \text{D/Pd} = 0.65 \) after which the d holes are filled and the susceptibility tends to zero.

The value of \( Z \) is clearly near to 0.5 (Fig.24.) at low values of D/Pd and decreases as D/Pd increases. At \( \text{D/Pd} = 0.8 \), the likely value of \( Z \) from magnetic susceptibility measurements is decreasing to zero.

3.4. Theories of Cold Fusion

3.4.1. Difficulties In Cold Fusion seen by classical nuclear physicists

The difficulties are 1) penetrating the coulomb barrier, 2) the fact that nuclear and chemical processes take place at radically different frequencies (e.g., \( 10^{14} \text{ sec}^{-1} \) and \( 10^{22} \text{ sec}^{-1} \)) [referred to as "asymptotic freedom" by Preparata (25)] and 3) the Leggett-Baym (26) point, according to which the deuterium ions in the palladium lattice are further apart than they are in deuterium molecule and therefore, as the authors saw it, must remain stably apart.

As to barrier penetration, it is invalid to apply a simple Gamow formula to calculate the tunneling probability in metals as is done in the dilute plasma. Thus, in the metal the approach to collision is through an electron gas which screens the charges from each other and thus reduces the barrier. A Gamow calculation is inapplicable.

The insulation of the nucleus from chemical forces seems incompatible with the fact that Mossbauer frequencies depend upon the chemical surrounds of the nucleus.

The Leggett-Baym point is a difficulty. Even in the tetrahedral positions, the D-D distances are 1.77 Å too large.

However, if the Gittus-Bockris hypothesis is pursued, i.e., the activity is at dislocations, the solubility is greatly enhanced and D/Pd in those areas must be >> 1. If it is >3, the tetrahedral holes are filled when the D/Pd at the dislocations exceeds 3. The only possibilities are interstitial positions when the D-D distances are smaller.

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3.4.2. Anomalous phenomena which act as criteria for the correctness of models

The essential phenomena of cold fusion—small rate of production of neutrons, large rate of production of tritium, heat more than between 10 and 100 (occasionally 1,000) watts per cc of Pd—are well known.

However, a successful theory must be consistent with the following:
1. The sporadicity of observation of the effects,
2. Switch-on is dependent upon impurities in the solution, or the metal,
3. Switch-on does not occur for times of the order of 100 hours of electrolysis.

3.4.3. Types of Theories

1. The fusion of D + D in the bulk of a metal lattice [Preparata(25); Takahashi(27)].
2. The fusion of D + D at promontories on the surface of the electrode [Bockris(28); Kim(29)].
3. Production of tritium and heat with a mechanism which involves "virtual" neutrons [Hegelstein(30)].
4. Transmutational reactions [Kucherov(31)].

3.4.3.1 Fusion in the bulk

3.4.3.1.a. Preparata Model (25)

The most comprehensive attempt to give a credible theory of this is due to Preparata(25) with an application of his ideas of super radiance (particles behave in a coherent fashion within the lattice).

This theory does take notice of the electronic structure of transition metals. The model of a metal tacitly assumed by most workers is that of a series of individual (non-bonded) cations interspersed by essentially free electrons. But transition metals are bonded (32) and share electron orbits in a three-dimensional manner. Preparata produces a diagram (Fig. 25.) in which he utilizes the properties of the d electron level in palladium to make a rudimentary theory of such screening, and deduces therefrom a reduction in the equivalent barrier height (Jones Fusion). At this point, to increase the rate of fusion to attain the observed heat, Preparata introduces his hypothesis that there are groups of electrons (400 Å) and groups of deuterons (1,000 Å) which move coherently.

Such a model would give rise to extreme heating in the hot spot regions of the coherent groups and therefore destroy their structure which, in this model, is the origin of the enhanced fusion. For this reason, Preparata brings in "electron cooling,". He shows that the excess heat is "taken away" by the coherent electron groups.

Preparata's theory has the virtue of leaning upon the real electronic structure of palladium. However, after having got to the Jones limit quite rationally, it needs faith, i.e., that the coherent groups which he assumes, must be assumed to exist. Further, it gives no interpretation of the facts brought out above as those most characteristic of cold fusion experiments, their sporadicity; their dependence upon specific surface structures and the abnormally long time they take to switch on (longer than that to reach D/Pd = 0.83 near the surface).

3.4.3.2. Surface Promontory Models

The first of these was proposed in 1990 by Bockris et al.(28) on the basis that observations made of successful electrodes showed that their surfaces possessed a dendritic structure.

On this basis, a model was suggested which depended upon the high field developed at tips of low radius of curvature [Bockris and Gonzales-Martin(33)].

Thus, the evolution of deuterium preferentially occurs at these tips and and they become associated sporadically with bubble formation. When these bubbles attain a certain size, the high field at the tip of the dendrites then emits electrons into the deuterium in the gas containing the bubble and ionizes some of the D₂. \( D_2 + e \rightarrow D^- + D \) (Fig. 26.).
A zeroth approximation theory was given by Packham, et al.(34), still assuming a D\(^+\)---D\(^+\) relation and a Gamow barrier. But since that time, it has been realized that the dendrite tips have ADSORBED D, which may have a zero or even a negative charge. This model does not have a Coulomb barrier.

The theory at first seemed to be a hot fusion theory, hence consistent only with \(n/T=1\). However, in the cluster impact work(35), there is the same high reaction rate for the tritium-proton channel and a low rate for the conventional He\(^+\) + neutron level, as is observed in cold fusion. It is found here that the pre-exponential factor of the Gamow equation is \(10^25\) times larger for the low energies involved here rather than for the high ones in classical nuclear physics.

This suggestion of a preferred channel for tritium solves the principal difficulty suffered by the original dendrite theory of Bockris et al (28).

The dendrite theory has advantages over competing theories because it explains the factors which are anomalous in other models. Thus, it depends upon the surface conditions, because only some surfaces will grow dendrites. The dendrites may not consist only of palladium, they may be of other materials grown from the solution. Furthermore, the growth of dendrites would depend upon the impurities in solution, in a characteristic way as developed by Popov(36). Briefly, the rate of growth would depend exponentially upon the impurities in the solution. Irreproducibility obtains an interpretation, as do long switch-on times.

Thus, the dendrite theory not only explains the anomalous \(n/T\) ratios formation but it also gives a quantitative interpretation of the surface sensitivity, the dependence upon impurities, and the long time for delay. Such features are not possessed by any other theory.

3.4.3.3. Theory involving Neutron Transfer

This theory is due to P. L. Hegelstein(30) and has a principal point as its foundation: the Coulomb barrier is difficult to overcome without some special mechanism such as the high field arising at points of minimal curvature, so that it is clear that a mechanism which avoids it is welcome.

In Hegelstein's view, this can be achieved by assuming that neutrons from deuterons transfer to "acceptor" nuclei such as another deuteron, \(^6\)Li, \(^10\)B or to Pd itself, entering their nuclei to produce various products including tritium (cf. Kucherov's model).

On the other hand, Hegelstein, by avoiding the coulomb barrier, involves a difficulty, namely, the distance the neutrons would have to travel to achieve the reaction which he suggests. Thus, only "virtual" neutrons are available to him and it is easy to calculate the life time of such a particle (Fig.27.).

Hagelstein's model is of great interest because it would appear to give a step towards the production of various new nuclei as observed by Kucherov. The major problem is to lengthen the life time of "virtual" neutrons by, say, \(10^5\) times.

3.4.3.4 Transmutational Theories

Karabut, Kucherov and Savvatimova(31) have suggested a theory based upon experiments that they have carried out in glow discharge experiments in the presence of deuterium. They observe characteristics which are similar to those which are observed in aqueous solution, excess heat, weak neutron generation, tritium and He\(^+\) production, together with characteristic X-rays and weak gamma radiation.

The novel feature of their work is that they analyze their cathodes chemically, finding, of course, helium and tritium similar to the findings earlier published by Bockris et al. However, they utilize an X-ray microprobe, high resolution dipole mass spectrometry and secondary high mass spectrometry and find a host of new elements in the palladium as a result of the electrolysis. Thus, they found not only \(^6\)Li, but also several other metals from groups one and two and then also Ca, Cr, Ni, Ge, etc. These elements occur in the upper 1 micron layer of the cathode, in consistency with the concept of the surface reaction as the origin of fusion. The content of the transmuted elements is up to 0.1 atomic %.

Karabut et al. observed that the presence of germanium is wholly unexpected. The maximum concentration is 0.1 atomic %.
The possible reactions suggested by Karabut et al. are given in Fig.28. Very approximate calculations which have been made by Karabut et al. seem to suggest that the right order of magnitude of heat can be obtained on the basis of these suggested reactions.

4. ACKNOWLEDGMENTS

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5. REFERENCES

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9) E. Storms, ibid., p. 21.
30) P. H. Hagelstein, ibid., 23 (1993) 353; also a preprint of the paper to be communicated to Fusion Technology.
<table>
<thead>
<tr>
<th>HYDROGEN ISOTOPE</th>
<th>H/Pd or D/Pd</th>
<th>T (K)</th>
<th>EFF.CHARGE NUMBER, Z*</th>
<th>EXPER. TECHNIQUE</th>
</tr>
</thead>
<tbody>
<tr>
<td>H</td>
<td>High (α-phase)</td>
<td>290-350</td>
<td>positive, +1 for H/Pd=0.6</td>
<td>Drift velocity</td>
</tr>
<tr>
<td>H</td>
<td>≤2.8x10^{-2}</td>
<td>455-513</td>
<td>+0.4 to +0.55</td>
<td>H₂ flux meas.</td>
</tr>
<tr>
<td>H</td>
<td>≤1.6x10^{-2}</td>
<td>520-620</td>
<td>+0.54</td>
<td>H₂ flux meas.</td>
</tr>
<tr>
<td>D</td>
<td>≤1.0x10^{-2}</td>
<td>520-620</td>
<td>+0.51 to +0.59</td>
<td>H₂ flux meas.</td>
</tr>
<tr>
<td>H, D</td>
<td>≤4.0x10^{-2}</td>
<td>420-1070</td>
<td>Between +0.3 and +0.7</td>
<td>H₂ flux meas.</td>
</tr>
<tr>
<td>H</td>
<td>≤8.2x10^{-3}</td>
<td>970</td>
<td>+0.44</td>
<td>Diff. potent.</td>
</tr>
<tr>
<td>H, D</td>
<td>≤8.2x10^{-3}</td>
<td>970</td>
<td>+0.35</td>
<td>Diff. potent.</td>
</tr>
</tbody>
</table>
Experimental Set-up

Chiller

Heat-Exchanger
Water Tank

20°C Water

Pump

Water In 6l/min

Water Out

Thermocouple-2

Cold Fusion Cell

D₂O

Thermocouple-1

Experimental Set-up
Fig. 2a  Saw-tooth-current Mode Operation; Pre-loading

Fig. 2b  L-H Current Mode Operation; Heat Measurement

Current Operation Modes.
CELL ASSEMBLY FOR RF STIMULATION

WORKING ELECTRODE (Pd)
COUNTER ELECTRODE (Pt)
THERMISTOR

H₂ + D₂

ELECTROLYTE LEVEL (0.3 M LiOD)

RF TO GENERATOR

RF FROM GENERATOR
CELL ASSEMBLY FOR MAGNETIC STIMULATION

(IN THE FIELD OF 200 GAUSS HORSE-SHOE MAGNET)

WORKING ELECTRODE (Pt)
COUNTER ELECTRODE (Pt)
THERMISTOR

H2 + D2

ELECTROLYTE LEVEL (0.3 M LiOD)

HORSE-SHOE MAGNET
CELL ASSEMBLY FOR MAGNETIC STIMULATION
(SYMMETRIC 800 GAUSS MAGNETIC FIELD)

WORKING ELECTRODE (Pd)
COUNTER ELECTRODE (Pt)
THERMISTOR

ELECTROLYTE LEVEL
(0.3 M LIOD)

H$_2$ + D$_2$

TEFLON HOLDER FOR ELECTROLYSIS CELL AND MAGNETS

PERMANENT DISC MAGNETS
Excess Power As A Function Of Time
D/Pd MEASUREMENTS
RF STIMULATION AT 365.608 MHz

△ 30 mW  ● 12 mW  ■ 6 mW
EXCESS POWER AS A FUNCTION OF FREQUENCY

Excess Power (W)

RF Frequency (MHz)

71.9 76.9 77.9 78.9 79.9 80.9 81.9 82.9 84.9 91.9 365.6 400.6 533.7 560
MAGNETIC STIMULATION

Temp Increase (°C)

0  2  4  6  8  10  12  14

0  500 1000 1500 2000

Time (sec)

800 GAUSS MAGNET IN

200 GAUSS MAGNET IN
HEXAGONS AND MICROVOIDS
SURFACE COVERAGE WITH HEXAGONS
AS A FUNCTION OF DEPTH

% change

Depth (um)
EFFECT OF OVERPOTENTIAL
EFFECT OF PULSING

RESIDENT HYDROGEN IN DEFECTS

\[ c_\sigma = c_0 e^{-\frac{\sigma V}{RT}} \]

AFTER PULSING HYDROGEN PARTLY FLUSHED WITH DEUTERIUM

AFTER PROLONGED PULSING HYDROGEN FULLY FLUSHED WITH DEUTERIUM
EXCESS HEAT ON CELL 16-P

% EXCESS

D/Pd

TIME (HOURS)

% EXCESS HEAT

D/Pd ratio

CHARGING

PULSING

50 mA/cm²

500 mA/cm²

0.2 Hz (5 s)

200 Hz (5 ms)
CONDITIONS FOR CRACKING

MECHANISM 1. COUPLED DISCHARGE CHEMICAL DESORPTION

OVERPOTENTIAL < -0.35 V

\[
f_{D_2} = 10^{1.5} e^{-\frac{1}{2} \frac{1}{RT}}
\]

MECHANISM 2. FAST DISCHARGE SLOW CHEMICAL COMBINATION

OVERPOTENTIAL > -0.35 V

\[
f_{D_2} = e^{\frac{2nF}{RT}}
\]
SURFACE COVERAGE WITH HEXAGONS AS A FUNCTION OF OVERPOENTIAL
SURFACE COVERAGE WITH HEXAGONS AS A FUNCTION OF OVERPOTENTIAL

![Graph showing surface coverage as a function of overpotential with extensive and intensive changes indicated.]
SURFACE COVERAGE WITH HEXAGONS AS A FUNCTION OF TIME

% change

Time (hours)

0.1  1  10  100  1000
CONCENTRATION CHANGE WITH TIME AT DEPTH OF 10 um IN FOIL 1 mm THICK

C/C₀ vs Time (sec)

- D = 10⁻⁷ cm² s⁻¹
- D = 10⁻⁸ cm² s⁻¹
- D = 10⁻⁹ cm² s⁻¹
CONCENTRATION CHANGE WITH TIME AT DEPTH OF 10 um IN CYLINDER 1 mm RADIUS
PALLADIUM DEUTERIDE LATTICE

**OCTAHEDRAL**

D/Pd < 0.80

- Pd - Pd = 4.08 Å
- D1 - D2 = 2.88 Å
- D2 - D3 = 2.88 Å

**TETRAHEDRAL**

D/Pd > 0.80

- Pd - Pd = 4.08 Å
- D3 - D4 = 1.77 Å
- D4 - D5 = 2.51 Å

- PALLADIUM
- DEUTERIUM IN OCTAHEDRAL SITES
- DEUTERIUM IN TETRAHEDRAL SITES
EFFECTIVE CHARGE NUMBER OF H AND D AS A FUNCTION OF H,D/Pd

MAGNETIC SUSCEPTIBILITY = 0
PREPARATA'S COHERENCE DOMAIN MODEL

d-ELECTRONS' SHELL

\[ \eta \sqrt{2a_o} \]

DISK OF 'STATIC' CHARGE

200 (eV)

SHALLOW HOLES

DEUTERIUM NUCLEI

-2a_o \quad \xi \quad 2a_o
THE DENDRITE ENHANCED TUNNELING MODEL

FIELD \(10^7\) V/cm
DISCHARGE CURRENTS OF \(\sim 10^5\) A

\[D^+ \rightarrow D \rightarrow D^+_2 + D + 2e\]
HAGELSTEIN'S "VIRTUAL" NEUTRON MODEL
KUCHEROV'S REACTION SCHEMES

\[ ^{46}\text{Pd} \rightarrow ^{36}\text{Kr} + ^{10}\text{Ne} \]
\[ \rightarrow ^{34}\text{Se} + ^{12}\text{Mg} \]
\[ \rightarrow ^{30}\text{Zn} + ^{16}\text{Sn} \]
\[ \rightarrow ^{24}\text{Cr} + ^{22}\text{Ti} \]

\[ ^{46}\text{Pd} + \text{D} \rightarrow ^{42}\text{Mo} + ^{5}\text{B} \]
\[ \rightarrow ^{40}\text{Zr} + ^{7}\text{N} \]

\[ ^{46}\text{Pd} + 2\text{D} \rightarrow ^{38}\text{Sr} + ^{10}\text{Ne} \]
\[ \rightarrow ^{42}\text{Mo} + ^{6}\text{C} \]
\[ \rightarrow ^{46}\text{Pd}^* + ^{2}\text{He} \]

\[ ^{46}\text{Pd} + 3\text{D} \rightarrow ^{44}\text{Ru} + ^{5}\text{B} \]
\[ \rightarrow ^{46}\text{Pd}^* + ^{3}\text{Li} \]