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ANOMALOUS EFFECTS INVOLVING EXCESS POWER, RADIATION, AND HELIUM PRODUCTION DURING D_2O ELECTROLYSIS USING PALLADIUM CATHODES

NUCLEAR REACTIONS IN SOLIDS

KEYWORDS: *calorimetry, diffusion, glass*

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Previous experiments showed that eight electrolysis gas samples collected during episodes of excess power production in two identical cells contained measurable amounts of ^{4}He while six control samples gave no evidence for helium. However, the detection limit for helium could not be defined clearly. This study of helium diffusion into the Pyrex glass sample flasks establishes a minimum helium detection limit of 3×10^{13} atom/500 ml (3 ppb) for these experiments. New D_2O and H_2O control experiments involving helium measurements of electrolysis gas samples collected in metal flasks support this conclusion. This places the ^{4}He production rate at 10^{11} to 10^{12} atom/s per watt of excess power, which is the correct magnitude for typical fusion reactions that yield helium as a product. Simultaneous evidence for excess power, helium production, and anomalous radiation was present in these experiments. Completely new experiments with more precise helium measurements are reported that again show simultaneous evidence for excess power, helium production, and anomalous radiation.

INTRODUCTION

The low intensity of neutrons in cold fusion experiments[1,2] has prompted proposals of nuclear processes that yield only heat and helium as products.[3–7] We previously reported the results of electrochemical calorimetric experiments designed to detect helium in effluent gases during the electrolysis of D_2O and H_2O with palladium cathodes.[8,9] However, the diffusion of atmospheric helium into the Pyrex glass flasks during the time between sample collection and analysis (22 to 61 days) could be significant. Furthermore, our experimental detection limit for helium could not be defined clearly.[9] We have therefore investigated the atmospheric helium diffusion rate for flasks filled with nitrogen, hydrogen, or deuterium-oxygen electrolysis gases. These studies yield revised helium detection limits that place the ^{4}He production rate in the range of 10^{11} to 10^{12} atom/s per watt of excess power.

EXPERIMENTAL SECTION

The eight round-bottom 500-ml Schlenk flasks made of Pyrex that were used for the collection of electrolysis gas samples were vacuum leak tested before use. All glass joints were carefully lapped for a concentric fit and lubricated with silicon grease (Dow Corning, High Vacuum). The average glass wall thickness was volumetrically determined to be $d = 1.8$ mm. The electrolysis gas samples collected at China Lake were sent to the University of Texas for analysis by mass spectrometry. The two calorimetric cells (A and B) were always placed close together in a constant-temperature water bath. The sources of the palladium rod cathode (Johnson-Matthey, 99.96%) and the D_2O (Cambridge Isotope Laboratories, 99.9%) were the same in each experiment. Analyses of our unused palladium showed no detectable amounts of helium.[9] Details of the electrochemical experiments, calorimetric measurements, and helium analyses are reported elsewhere.[8,9] In later experiments, we used a correction term for room temperature changes in the calorimetric calibration and

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measurement cycles, which reduced the experimental error. Two commercial laboratories (Helium Field Operations, Department of the Interior, Amarillo, Texas; and Rockwell International, Canoga Park, California) were used for this study of the rate of atmospheric helium diffusion into these flasks.

The detection of anomalous radiation involved the use of a thin end window Geiger-Mueller (GM) alpha-beta-gamma detector (Ludlum model 44-7) positioned ~20 cm from the tops of the electrochemical cells and connected to a scalar rate meter (Ludlum model 2200) and printer (Casio model HR-8A). In later experiments, the GM detector was positioned within 6 cm of either cell. According to the tube manufacturer (LND, Inc.), the energy response for this detector shows a peak in the relative count rate for photon energies near 60 to 80 keV. There is also a small gradual increase in the relative count rate as the photon energy increases from 0.3 to 1 MeV and higher. Photon energies below 40 keV can enter the detector only through the thin end window and probably would not escape from the electrolysis cells to reach the detector.

RESULTS

Table I presents results from two different laboratories that relate to atmospheric helium diffusion into three flasks filled with nitrogen. These measurements yield a mean diffusion rate of $3.2 \pm 0.6 \times 10^{12}$ atom/day, which is in good agreement with the theoretical value for Pyrex flasks with $A = 314\ \text{cm}^2$ and $d = 1.8$ mm as defined by Dushman[10]:

$$q = \frac{K \cdot P_{He}}{d} = 2.6 \times 10^{12}\ \text{atom/day} \ , \tag{1}$$

where

q = volume of helium flowing through the glass wall per unit area per second ($\text{cm}^3/\text{cm}^2 \cdot \text{s}$)

K = permeability for helium diffusion through glass [$\text{cm}^3 \cdot \text{mm/s} \cdot \text{cm}^2$ (cm Hg)]

P_{He} = difference in partial pressure of helium gas (cm Hg).

This experimental agreement indicates that the three flasks were quite uniform. The measurements by the two outside laboratories were done several months after our helium measurements were completed; hence, any effects due to deuterium or hydrogen saturation of the glass would likely have dissipated.

Measurements of atmospheric helium diffusion into four flasks filled with hydrogen or deuterium-oxygen electrolysis gases are shown in Figs. 1 and 2. These flasks yield a mean helium diffusion rate of $1.9 \pm 0.3 \times 10^{12}$ atom/day, which suggests that the inward diffusion of atmospheric helium is somewhat slower for flasks containing hydrogen or deuterium, as postulated by Miles et al.[9] Flask uniformity is again indicated by the good agreement of the helium diffusion rates for these four flasks. A total of eight flasks were used in our heat/helium studies, but one flask was broken during shipment; hence, Table I and Figs. 1 and 2 present helium diffusion results for the seven remaining flasks. Additional studies by the Amarillo laboratory, covering time intervals of 10 to 20 days, indicated that the rate of atmospheric helium diffusion into the flasks was two to three times slower for flasks that contained deuterium-oxygen electrolysis gases instead of nitrogen.

TABLE I

Experimental Helium Diffusion Rates in Nitrogen-Filled Flasks*

Time (days)	Measured ^{4}He (atoms × 10^{12})	Diffusion Rate (atom/day × 10^{12})
14[a]	47	3.4
44.6[b]	167	3.7
65[a]	170	2.6

*500 ml, Pyrex, $A = 314\ \text{cm}^2$, $d = 1.8$ mm.
[a]Helium Field Operations, Amarillo, Texas.
[b]Rockwell International, Canoga Park, California.

An important result of these helium diffusion studies is that there is absolutely no evidence for any direct leakage of air into any of these flasks and that outside helium enters only by diffusion through the glass. The lowest helium level measured in the electrolysis gas sample (9.7×10^{13} atom/500 ml, Fig. 2) is significantly less than the helium content of high-purity hydrogen (9.8×10^{14} atom/500 ml, Fig. 1) or the helium content of the atmosphere (6.0×10^{16} atom/500 ml). The gradually increasing helium content of the sample flasks with time can be explained totally by the known rate of atmospheric helium diffusion through the glass.

The good agreement between the experimental and theoretical rates for the diffusion of atmospheric helium into the nitrogen-filled flasks allows us to determine a minimum value for the ^{4}He detection limit. As reported by Miles et al.,[9] four of the flasks filled with nitrogen were analyzed for helium by usual procedures after 9 days of storage. One flask showed the presence of ^{4}He at the detection limit, while no helium could be detected in the other three flasks. On the basis of the mean helium diffusion rate of 3.2×10^{12} atom/day established experimentally in Table I, helium analyses for the nitrogen-filled flasks yield a minimum ^{4}He detection limit of 3×10^{13} atom/500 ml or 3 ppb. This detection limit compares favorably with the ±1-ppb error range reported by the Amarillo laboratory for the analysis of the gas samples. Furthermore, five recent Pd/D$_2$O and Pd/H$_2$O control experiments that used metal flasks connected to electrolysis cells yielded a

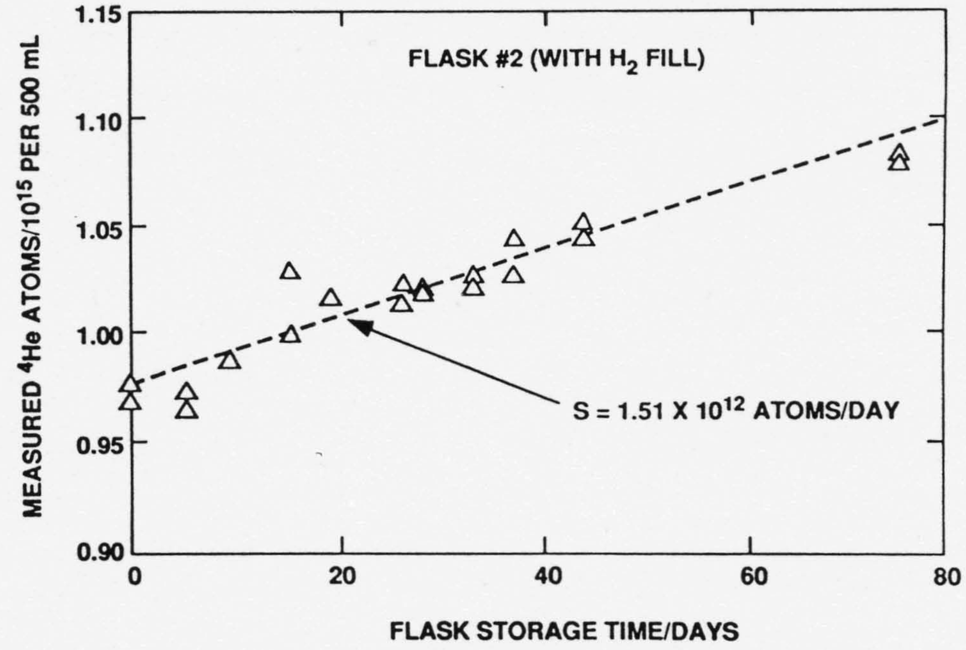

Fig. 1. Experimental rate of atmospheric helium diffusion into a Pyrex flask filled with hydrogen.

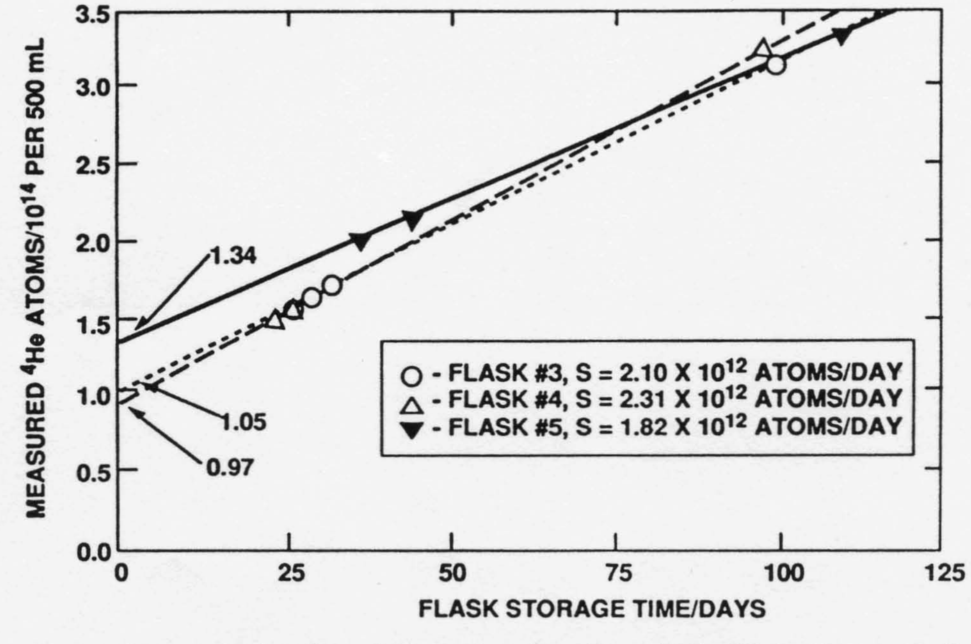

Fig. 2. Experimental rate of atmospheric helium diffusion into three Pyrex flasks filled with $D_2 + O_2$ from Pd/D_2O electrolysis. These experiments yielded excess power, helium production, and anomalous radiation, as shown in Table IV.

background helium concentration of 4.4 ± 0.6 ppb or $5.1 \pm 0.7 \times 10^{13}$ atom/500 ml as measured by the Amarillo laboratory. Helium detection limits of 1000 ppb (1 ppm) reported by Lewis et al.[11] and Albagli et al.[12] are far too insensitive to detect any ^{4}He fusion product in the effluent gases of Pd/D_2O electrolysis cells.

Table II presents measurements of ^{4}He based on the revised detection limit of 3 ppb. Two different electrochemical cells (A and B) were used in these experiments, and the time sequence in which the samples were collected over a 3-month period is indicated by the dates. Since $X = P_{OUT}/P_{IN}$, the input power P_{IN} can

TABLE II

Helium Production During D_2O Electrolysis: Revised Detection Limits

Sample	P_{EX} (W)	X	^{4}He atom/500 ml[a]	Flask Storage Time (days)
December 14(A)	0.52[b]	1.20[b]	10^{15} (large peak)	25
October 21(B)	0.46	1.27	10^{15} (large peak)	58
December 17(A)	0.40[b]	1.19[b]	10^{14} (medium peak)	22
November 25(B)	0.36	1.15	10^{15} (large peak)	24
November 20(A)	0.24	1.10	10^{14} (medium peak)	30
November 27(A)	0.22	1.09	10^{15} (large peak)	22
October 30(B)	0.17	1.12	10^{13} (small peak)	49
October 30(A)	0.14	1.08	10^{13} (small peak)	51
October 17(A)	0.07	1.03	$<10^{13}$ (no peak)	61
December 17(B)	0.29[b,c]	1.11[b,c]	$<10^{13}$ (no peak)	22

[a] No ^{3}He was detected. The mass spectrometer was always set at the highest sensitivity.
[b] $I = 250$ mA/cm^2. All other experiments used $I = 200$ mA/cm^2.
[c] Possible calorimetric errors due to low D_2O solution levels.

be calculated from $P_{IN} = P_{EX}/(X - 1)$. The time required to generate 500 ml of electrolysis gas is 4440 s at 200 mA/cm^2 for the electrode area ($A = 2.6$ cm^2), laboratory temperature (23°C), and pressure (700 Torr). For the experiments yielding 10^{15} atom/500 ml of electrolysis gas, the rate of ^{4}He production is 10^{11} to 10^{12} atom/s per watt of excess power. This is the correct magnitude for typical fusion reactions that yield ^{4}He as a product, listed in Table III.

The amount of helium observed is also compared to the flask storage time for each electrolysis gas sample in Table II. If the diffusion of atmospheric helium into the flasks were a significant factor, the results should yield helium levels that increase linearly with the flask storage time as in Figs. 1 and 2. No such effect is apparent from Table II. For the D_2O/LiOD experiments, high and low helium levels occurred at both short and long storage times, which ranged from 22 to 61 days. In fact, linear regression shows a negative correlation, if any exists at all.[9]

TABLE III

Deuteron Fusion Reactions That Produce Helium

Reaction	Energy (MeV)	^{4}He atom/s per Watt of Excess Power ($\times 10^{11}$)
$^2H + {}^2H \rightarrow {}^4He + \gamma$	23.8	2.6
$^2H + {}^3H \rightarrow {}^4He + n$	17.6	3.6
$^2H + {}^6Li \rightarrow 2{}^4He$	22.4	5.6
$^2H + {}^7Li \rightarrow 2{}^4He + n$	15.1	8.3

Control experiments are essential in ruling out atmospheric contamination in measurements of helium as a nuclear ash. Our controls involved one experiment of palladium in D_2O [October 17(A), Table II] and five experiments of palladium in H_2O; these experiments showed no detectable quantities of helium and are discussed in detail elsewhere.[9] It is difficult to use atmospheric contamination arguments to explain how helium could be present in all eight experiments that showed excess power and not be detected in any of the six control experiments.

The exposure of dental films during episodes of excess power was reported elsewhere.[9] During this same time, the GM detector recorded anomalous high radiation count rates, as shown in Fig. 3. Several subsequent experiments involving Pd/D_2O + LiOD electrolysis with new palladium cathodes yielded near-normal radiation counts. These electrolysis experiments also failed to produce significant excess power and showed no exposure of dental films. Furthermore, the passing of the electrolysis gases directly onto the window of the GM detector did not yield anomalous radiation count rates. Several later electrolysis experiments also failed to produce any anomalous radiation counts, as shown in Fig. 4. This series of studies was used to establish a mean and standard deviation of 31 296 ± 275 counts per 12 h for our GM detector. Radiation monitoring continued for nearly 1 yr, including periods of electrolysis studies as well as periods with no experiments in operation. Normal radiation counts were always observed, and no excess power was detected.

Although film exposure and high GM counts provide no energy information, these results cannot readily be dismissed. After all, radioactivity itself was discovered in 1896 by Henri Becquerel by the fogging of

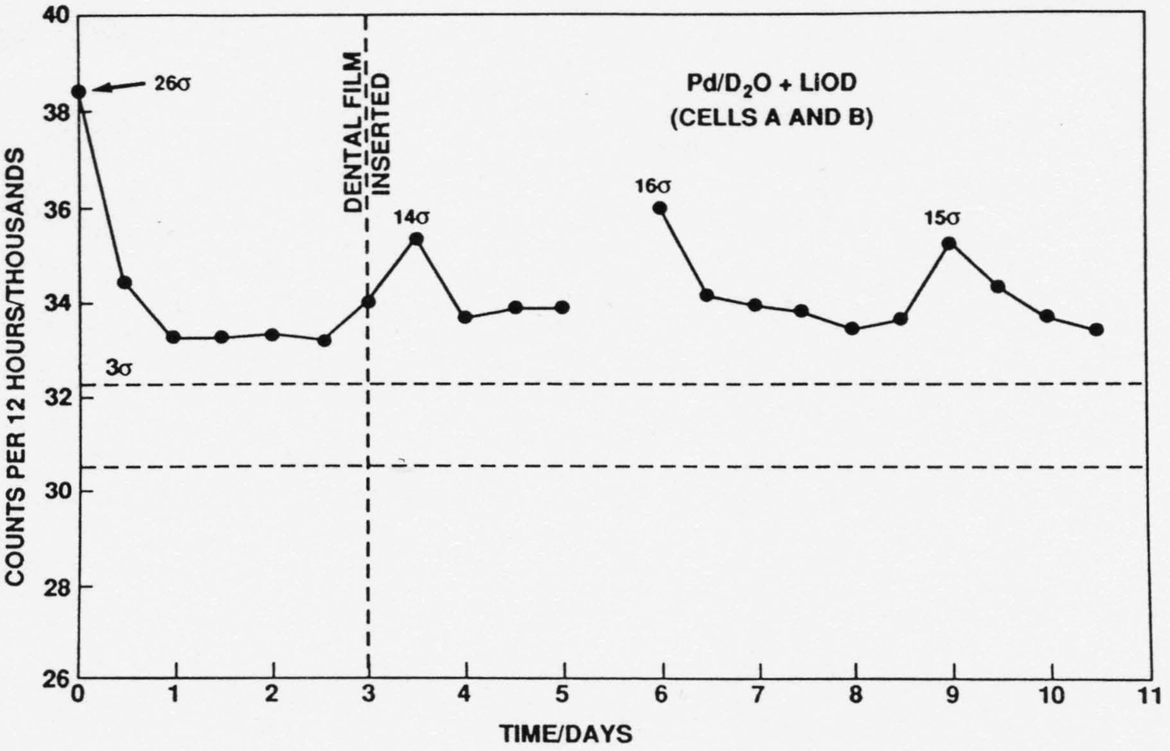

Fig. 3. Anomalous radiation counts observed with the GM detector during the time of the dental film studies. This experiment gave excess power and helium production [December 17(A), Table II] and blackening of the dental films in both cells A and B.

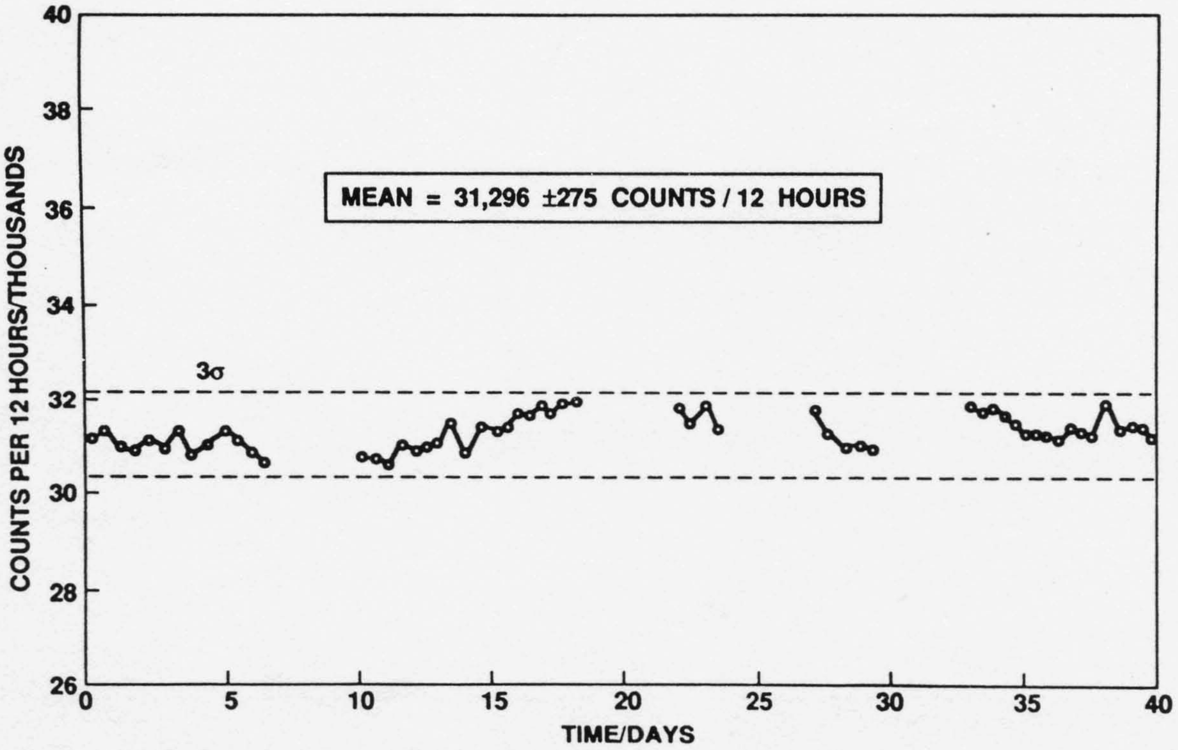

Fig. 4. Radiation counts observed with the GM detector during various experiments that produced no significant excess power. These radiation counts were used to establish the mean background of 31 296 ± 275 counts per 12 h.

photographic plates. Furthermore, the extremely rare observation of high counts by the GM detector during the same time as the dental film exposure and excess power generation occurred defies any simple explanation.

The plateau determination for the GM detector is shown in Fig. 5 for the normal background as well as for ^{55}Fe and ^{137}Cs sources. No unusual behavior was observed. At the instrument setting of 0.9 kV suggested by the manufacturer, small voltage changes would not produce high count rates for this detector. Many $Pd/D_2O + LiOD$ electrolysis experiments gave only normal radiation counts, as shown in Fig. 4. However, several more recent experiments in which the GM detector was placed closer to the $Pd/D_2O + LiOD$ cells (6 cm) showed occasional periods of elevated counts. Furthermore, the variation of the distance between the cell and detector appears to trace the radiation source back to the electrolysis cell. Such studies, however, are difficult because of the weak signal and the sporadic nature of the anomalous radiation. Anomalous radioactive emissions in Pd/D_2O electrolysis experiments, as measured with GM detectors, were also reported by Uchida et al.[13] Evidence for neutrons and gamma rays during Pd/D_2O electrolysis was presented by Pons and Fleischmann.[2]

After nearly 1 yr of many new experiments that failed to generate any significant anomalous effects, a small production of excess power and high GM counts were again detected in experiments involving Pd/D_2O electrolysis. Three gas samples were collected from the two electrolysis cells (A and B) used in this experiment and sent to a commercial laboratory (Rockwell International) for helium analysis. Results for these experiments are presented in Table IV, whereas the helium analyses are shown in Fig. 2.

All three samples yielded helium production rates in the range of 10^{11} to 10^{12} atom/s per watt of excess power, as found in our previous studies. Furthermore, the extrapolation back to the initial time of the gas sample collection eliminates the contribution of helium diffusion through the glass. According to Rockwell International, these extrapolated results are accurate to $\pm 0.01 \times 10^{14}$ atom/500 ml ($\pm 1\sigma$). Therefore, unlike our previous studies, these helium results are much more precise than our calorimetric measurements. In fact, our calorimetric error (± 0.020 W) could be a substantial part of the excess power measurements, especially during the collection of the gas sample in flask 4. Nevertheless, the differences in detected helium and observed power for flasks 3 and 5 or for flasks 4 and 5 both yield a helium production rate of 10^{11} atom/s per watt of excess power. During these experiments, neither Rockwell International nor our laboratory knew the correlation of excess power and helium measurements until after the study was completed and all results were reported to a third party.

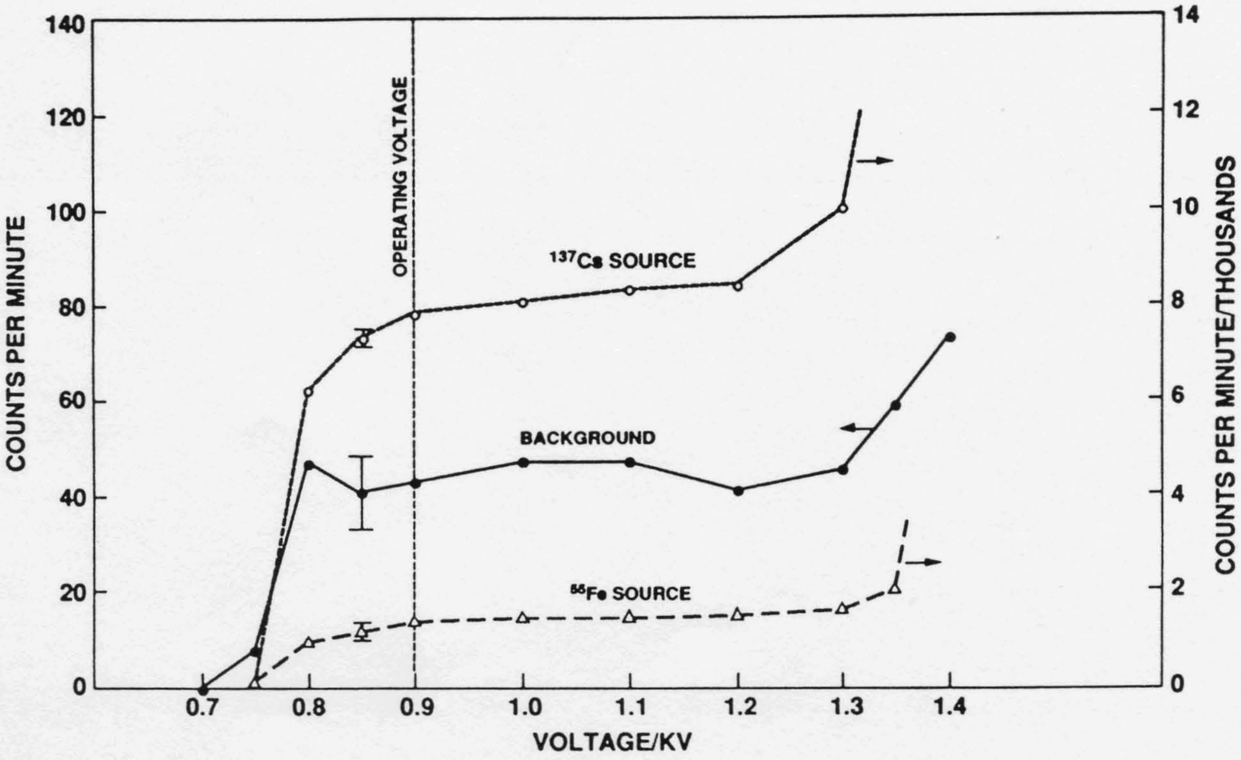

Fig. 5. Plateau determinations for the GM detector with ^{137}Cs and ^{55}Fe sources as well as the background counts. Normal behavior is observed. (The error bars represent $\pm 1\sigma$.)

TABLE IV

New Experiments Yielding Excess Power and Helium Production During D_2O Electrolysis

Sample	P_{EX} (W)	X	^4He atom/500 ml[a] ($\times 10^{14}$)	^4He/s per Watt of Excess Power[b] ($\times 10^{11}$)
December 30, 1991 (B) (flask 5)	0.100[c]	1.08[c]	1.34	2
December 30, 1991 (A) (flask 3)	0.050[c]	1.02[c]	1.05	2
January 3, 1992 (B) (flask 4)	0.020[d]	1.01[d]	0.97	5[e]

[a] Error range reported by Rockwell International was $\pm 0.01 \times 10^{14}$ atom/500 ml ($\pm 1\sigma$).

[b] Corrected for a background level of 5.1×10^{13} atom/500 ml as determined in experiments that used metal flasks.

[c] $I = 525$ mA. An anomalous GM count of 38 668 per 12 h (27σ) was measured during this period.

[d] $I = 500$ mA. The GM count rate was within the normal range.

[e] Less accurate than the other two values because of a larger relative uncertainty in the excess power measurement.

An anomalous GM radiation count of 38 668 per 12 h (27σ) was observed while the gas samples in flasks 3 and 5 were collected. The gas sample in flask 4 was collected 4 days later, during a time of normal radiation counts (31 771 counts per 12 h). Small excess power effects along with elevated GM counts were measured on 14 different days during this study. Although no clear relationship between the amount of excess power and the GM counts existed, one of the highest measurements for both excess power and GM counts occurred on December 30, 1991, during the collection of the gas sample in flask 5. The rate of helium production for that experiment (2×10^{11} atom/s per watt) is close to that expected for the fusion of deuterons to form ^4He (Table III).

More than 40 studies claim electrolytic production of tritium in excess of that expected from the deuterium/tritium separation factors.[14] Although tritium measurements were conducted in each of our experiments, the observed increases in tritium concentrations were within possible electrolytic enrichment factors.[9] It is interesting to note that Hodko and Bockris[14] found no excess tritium in cells that had the same D_2O source as in our experiments (Cambridge Isotope Laboratories). Nevertheless, an unusual increase in tritium was observed for cell A of Table IV following the addition of 0.004 mg of thiourea on January 8, 1992. The tritium level in this cell increased from 47 to 84% above that of the stock D_2O (375 dpm/ml) within 3 days of the thiourea addition. No thiourea was added to the companion cell B,. and no unusual increase in the tritium level was observed.

DISCUSSION

Several reports suggest ^4He production in other cold fusion experiments. Significant levels of ^4He in deuterated palladium systems were reported by Liaw et al.,[15] Chien et al.,[16] Yamaguchi and Nishioka,[17] and Karabut et al.[18] Two of these studies[17,18] involve gas loading rather than electrolysis, and three[15,16,18] involve measurements of helium in the palladium metal. There was a previous preliminary report[3] of ^4He in the electrolysis gas of $Pd/D_2O + LiOD$ electrolysis cells, but complete experimental details have never been made available.

From our electrolysis experiments, it would appear that a surface or near-surface fusion reaction is involved because most of the helium product is found in the gas phase rather than in the bulk metal. However, it is known that PdD_n is significantly different from palladium metal in its properties[19]; thus, it cannot be ruled out that helium may readily escape from bulk PdD_n under conditions of high deuterium loading. In fact, industrial installations that use palladium for the separation of hydrogen from helium and other gases strictly avoid the formation of any β-phase hydride by maintaining the temperature above 300°C (Ref. 19).

The lack of reproducibility of the anomalous effects in Pd/D_2O electrolysis experiments limited the progress of our research effort. Factors that can affect the reproducibility of the excess power effect during $Pd/D_2O + LiOD$ electrolysis include the palladium metallurgy; the handling or conditioning of the palladium; the extent of deuterium loading; the cell configuration and symmetry; the current density profile; the electrolyte; impurities in the cell components, palladium, D_2O or lithium; atmospheric contaminations such as H_2O or CO_2; and the time span of the experiments. Our experience suggests that palladium metallurgy may be critical for excess power. Two palladium rods gave excess power in three experiments. Six out of eight experiments with new palladium rods, in contrast, did not produce excess power or any significant radiation effect. These results suggest that palladium rods that yield excess power may show anomalous effects in a second or third experiment. The uniform loading of the palladium with deuterium without the development of cracks appears to be a key factor.

Experimental processes that could yield excess power effects include the exothermic formation of PdD_n

where $n \leq 0.6$ and the exothermic deloading of the palladium for $n \geq 0.6$, as reported by Balej and Divisek.[20] The exothermic formation of $PdD_{0.6}$ in our experiments would yield an excess power that is below the detection limit of our calorimeter (±0.020 W). The exothermic deloading of the palladium cathode represented by

$$PdD_{0.8} \rightarrow PdD_{0.6} + 0.1D_2 \ , \quad \Delta H = -3050 \text{ J/mol Pd} \ , \tag{2}$$

where H is the thermodynamic enthalpy (in Joules), yields an excess power effect of 0.012 W for our experiments (0.0416 mol Pd) when averaged over the 3-h period required for equilibration by our calorimetry (time constant ≈ 25 min). This exothermic deloading effect for our cells, therefore, yields an excess power that is again below the detection limit of our calorimeter.

Another possible explanation for the excess power observed is the Joule-Thomson effect for deuterium compressed into the palladium. The Joule-Thomson coefficient is expressed thermodynamically as

$$\mu = \left(\frac{\partial T}{\partial P}\right)_H = \frac{T(\partial V/\partial T)_P - V}{C_P} \ , \tag{3}$$

where

T = thermodynamic temperature (K)

P = thermodynamic pressure (atm)

V = volume (ℓ)

C_P = heat capacity at constant pressure ($J/K \cdot mol$).

From the van der Waals's equation of state, the Joule-Thomson coefficient for hydrogen is

$$\mu = \frac{2a/RT - b}{C_P} = -0.02331 \text{ K/atm} \ , \tag{4}$$

where

a = van der Waals's gas constant ($\ell^2 \cdot atm/mol^2$)

b = van der Waals's gas constant (ℓ/mol).

A similar value would be expected for deuterium. Thus, for 1 cm^3 of palladium containing $PdH_{1.0}$ (0.0565 mol H_2), a pressure change for hydrogen of -10^5 atm averaged over a 3-h period would yield an excess power effect of 0.35 W. This is approximately the excess power we observed, as seen in Table II. However, there are several major problems with this Joule-Thomson explanation for excess power. Since a net enthalpy balance is required by the First Law of Thermodynamics, periods of excess power due to the release of deuterium under pressure must be balanced by periods of power shortfalls when deuterium is compressed into the palladium. Furthermore, the Joule-Thomson effect could not sustain excess power over long periods as observed experimentally. In addition, excess power based on the Joule-Thomson effect would be expected for both H_2O and D_2O experiments. Finally, any Joule-Thomson derived excess power would not correlate experimentally with any helium production as reported in Tables II and IV.

The excess power reported in both Tables II and IV was usually fairly steady over time periods of several hours. Approximate excess energies could be calculated from Tables II and IV, by assuming that the excess power remained constant for the day. Major changes in the excess power usually occurred following additions of D_2O at the end of the day. This produces sudden voltage, temperature, and concentration changes within the cell.[21] For the experiments reported in Tables II and IV, the steady change in the cell voltage was generally in the range of -10^{-5} to -10^{-6} V/s. This gradual decrease in the cell voltage with time is due to the increasing concentration of LiOD produced by the D_2O electrolysis. Since there is never any steady state for either the cell voltage or the cell temperature, a differential equation governs the behavior of open, isoperibolic calorimetric cells.[21,22]

CONCLUSIONS

Simultaneous evidence for excess power, helium production, and anomalous radiation in several different experiments suggests that nuclear reactions do in fact occur in Pd/D_2O + LiOD electrolysis experiments. Results from other laboratories provide corroborative evidence for ^4He production in deuterated palladium systems.

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