

HELIUM ANALYSIS OF PALLADIUM ELECTRODES AFTER MOLTEN SALT ELECTROLYSIS

COLD FUSION

TECHNICAL NOTE

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A case of ^4He enrichment in a spent palladium deuteride electrode is reported. The issue of helium isotope production related to the Fleischmann-Pons effect is still much in debate. In light of this important issue, two palladium samples, used in deuteride- and hydride-conducting molten salt electrolytes, and their corresponding blanks were analyzed for both ^3He and ^4He content. Four specimens from the deuteride sample, which has produced excess heat, showed significant ^4He signals (enrichment) above the blank background level, while the hydride sample, used as a control with no excess heat measured, exhibited an opposite (depletion) effect. The ^3He content remained unchanged, within the limits of the instrument's sensitivity. The amount of ^4He detected was not commensurate with the excess heat according to known reaction mechanisms. The interpretation of the results was complicated by a substantial morphology difference among the samples. Because the level of helium content was small in magnitude, the possibility of atmospheric contamination cannot be dismissed completely.

INTRODUCTION

The generation of ^4He in experiments designed to reproduce the Fleischmann-Pons effect has been much in debate since the first announcement in 1989 (Refs. 1 through 11). In this technical note, we report the results of helium analysis on our spent palladium electrodes, used in a novel elevated-temperature molten salt electrolysis process, in which excess heat has been measured in the deuterium-based system.^{12,13}

We have demonstrated a novel elevated-temperature molten salt technique for generating high-level excess heat. More than 4 MJ/mol D_2 of excess heat, at least 600% over the input power, was measured in two incidents using a torched palladium anode and aluminum alloy cathode in a eutectic LiCl-KCl mixture saturated with excess LiD at $\sim 400^\circ\text{C}$. Measurements on the hydrogen-based system showed normal endothermic behavior. No thermochemical explanation can account for this excess heat.

The palladium electrodes were later examined for their morphological changes and for the concentration of ^3He and

^4He . A very porous surface microstructure of the samples was found¹³ as a result of the electrolysis and deuteriding process. In this technical note, we provide additional bulk microstructure information as a complement to our previous report.¹³ Slightly enriched ^4He in the deuteride sample was detected from all four specimens (in one case 14σ above the background), while the hydride sample showed an opposite effect. Therefore, the enrichment of ^4He in the deuteride system is considered anomalous. However, the amount of ^4He in the deuteride sample was not commensurate with the measured excess heat. We postulate that the majority of the ^4He escaped with the effluent D_2 gas, as reported by Bush et al.¹¹ Atmospheric contamination could be a possible source of error, although we believe this to be unlikely because of the isolation of the sample from the atmosphere during electrolysis.

EXPERIMENTAL ASPECTS

Details of the molten salt technique and the electrochemical cell configuration were discussed in our previous papers.^{12,13} Two palladium samples were made from Engelhard wire (4-mm diam, 99.995% pure) for the deuteride experiments; one served as the anode (PdD-a) and the other as a blank (Pd-b). The samples used in the hydrogen-based experiments were obtained from Alfa Products, Johnson-Matthey (batch 10960-BW, 1-mm diam, 99.997% pure). The sample used in the LiH-based electrolysis and the blank are named PdH-c and Pd-d, respectively. All palladium samples were torched with a propane/oxygen flame through which the palladium was completely melted and solidified into an irregular shape, resulting in a porous surface microstructure.¹³

The electrolysis was carried out in an argon-filled glove box, with oxygen and moisture contents maintained below 1 ppm. The palladium samples were examined by a scanning electron microscope (SEM) (International Scientific Instruments, model SX-40A) equipped with a Princeton gamma technology energy-dispersive X-ray elemental analysis system, before they were sent to Energy Technology Engineering Center (ETEC), Rockwell International Corporation (RI) (Rocketdyne Division, Canoga Park, California), for helium analyses. ETEC performed additional SEM examination on the sectioned palladium samples to determine the bulk microstructure, which is reported below.

The helium analyses were performed by ETEC with a unique high-sensitivity mass spectrometer that operates in the static mode and is capable of measuring as low as 10^{-11} atom fraction of ^4He and ^3He in solid metals. Farrar and Oliver¹⁴ have described the sample handling procedure and the system in detail. Absolute helium measurements in the range of 10^8 to 10^{18} atoms are allowed.

All palladium specimens for helium analyses were prepared by subdividing the sample with a wire cutter. Each specimen was then placed in a tungsten crucible and subsequently vaporized in a resistance heating vacuum furnace attached to the mass spectrometer system. Unwanted species, including hydrogen, deuterium, and tritium, were removed from the gas stream by a series of traps prior to the sample analysis.

Table I shows that the analyses on the deuteride samples were conducted by two completely separate sequences, each of which used two specimens. The sequence comprises the following:

1. the instrumentation background (cold blank) readings, in which ambient intrusion of ^4He contaminant through glass or metal joints was measured first to de-

tect the amount of leakage and the attainable sensitivity level—typically in the range of 10^8 atoms

2. one to three readings of a Rocketdyne-prepared control blank sample (RI control or RI-blk), in which no helium was expected, for calibration of the background level and to identify possible contamination, if any, from the crucible and heating process
3. one reading of a specimen of the deuteride sample to measure the total helium release, including the background
4. another reading of the RI control blank sample to ensure consistency in the background level
5. a reading of the second deuteride specimen
6. a reading of another RI control blank sample to provide another measure of the background signal. The procedure for the Engelhard blank was similar, as shown in Table I.

The readings from the RI control samples were averaged and referred to as the average control background level. The difference between each measured helium release of the

TABLE I
Helium Analysis Results for the Deuteride and the Blank Samples*

Specimen Name	Analysis Specimen Source	Sequence Mass ^a (mg)	³ He (10^9 atoms)			⁴ He (10^9 atoms)		
			Atoms Measured	Average Control ^b	Net Atoms Released ^c	Atoms Measured	Average Control ^b	Net Atoms Released ^c
---	Cold blank ^d		0.4			1.0		
RI-blk-1	RI control ^e	20.72	0.5	0.5 ± 0.1	0.0 ± 0.2	1.3	1.4 ± 0.1	-0.1 ± 0.2
PdD-a-1	Deuterated	21.49	0.6		0.1 ± 0.2	2.0		0.6 ± 0.2
RI-blk-2	RI control	19.12	0.4		-0.1 ± 0.2	1.5		0.1 ± 0.2
PdD-a-2	Deuterated	23.09	0.7		0.2 ± 0.2	4.2		2.8 ± 0.2
RI-blk-3	RI control	11.83	0.6		0.1 ± 0.2	1.5		0.1 ± 0.2
---	Cold blank		0.8			4.3		
---	Cold blank		0.5			4.5		
RI-blk-4	RI control	17.63	0.6	0.7 ± 0.2	-0.1 ± 0.3	3.8	4.0 ± 0.3	-0.2 ± 0.5
---	Cold blank		0.5			4.2		
PdD-a-3	Deuterated	17.07	1.0		0.3 ± 0.3	4.8		0.8 ± 0.5
RI-blk-5	RI control	20.58	0.6		-0.1 ± 0.3	4.3		0.3 ± 0.5
PdD-a-4	Deuterated	21.38	1.4		0.7 ± 0.3	4.6		0.6 ± 0.5
RI-blk-6	RI control	19.22	0.9		0.2 ± 0.3	3.9		-0.1 ± 0.5
---	Cold blank		0.2			1.7		
RI-blk-7	RI control	13.39	3.0 ^f	0.8 ± 0.2		1.6	1.4 ± 0.2	0.2 ± 0.3
Pd-b-1	Blank	11.94	1.0		0.2 ± 0.3	1.6		0.2 ± 0.3
RI-blk-8	RI control	11.50	0.6		-0.2 ± 0.3	1.3		-0.1 ± 0.3
Pd-b-2	Blank	14.30	0.2		-0.6 ± 0.3	1.7		0.3 ± 0.3
RI-blk-9	RI control	10.11	0.9		0.1 ± 0.3	1.3		-0.1 ± 0.3

*The samples were prepared from the Engelhard lot.

^aMass uncertainty is ± 0.01 mg.

^bAverage value of helium signal from the RI control specimens.

^cObtained by subtracting the average control release from the atoms measured. Estimated total uncertainty (1σ) is given following each value.

^dAnalyses of the cold background signal without any release from palladium samples.

^eThe control specimens were provided by RI to evaluate controlled background signal.

^fSuspect result, possibly from spurious helium background in furnace. Datum is not included in the calculation of the mean.

TABLE II
Helium Analysis Results for the Hydride and Blank Samples*

Specimen Name	Analysis Specimen Source	Sequence Mass ^a (mg)	³ He (10 ⁹ atoms)			⁴ He (10 ⁹ atoms)		
			Atoms Measured	Average Blank ^b	Net Atoms Released ^c	Atoms Measured	Average Blank ^b	Net Atoms Released ^c
---	Cold blank ^d		0.6	0.7 ± 0.2		1.9	1.9 ± 0.2	
Pd-d-1	Blank	16.50	0.9		0.2 ± 0.3	16.9		15.0 ± 0.3
---	Cold blank		0.7			2.1		
PdH-c-1	Hydrided	19.90	0.8		0.1 ± 0.3	2.6		0.7 ± 0.3
Pd-d-2	Blank	24.75	0.9		0.2 ± 0.3	197.2		195.3 ± 3.9
PdH-c-2	Hydrided	23.98	1.0		0.3 ± 0.3	8.1		6.2 ± 0.3
Pd-d-3	Blank	20.83	1.5		0.8 ± 0.3	14.4		12.5 ± 0.3
---	Cold blank		0.9			1.7		

*The samples were prepared from the Johnson-Matthey lot.

^aMass uncertainty is ±0.01 mg.

^bAverage value of helium signal from the cold blanks.

^cObtained by subtracting the average blank release from the atoms measured. Estimated total uncertainty 1σ is given following each value.

^dAnalyses of the cold background signal without any release from palladium samples.

palladium specimen of interest and the average control level is considered the net atoms released for each specimen.

The analysis for the hydride system proceeded differently, as indicated in Table II. The RI control samples were not used, as was the case for the deuteride samples; instead, readings of cold blanks were taken among specimens intermittently. The average of the cold blank readings was then assigned as the average control background level and used in the net atoms released calculation for each specimen of the hydride and the Johnson-Matthey blank. This procedure was warranted because the previous measurements of the cold blanks and the RI control samples in the deuteride systems showed no difference in background reading.

RESULTS

In this technical note, we report results from four palladium samples: the deuteride palladium (PdD-a) and its blank (Pd-b), both from Engelhard; the hydride palladium (PdH-c) and its blank (Pd-d), both from Johnson-Matthey. Four specimens from the deuteride sample (PdD-a-1 through 4) and two specimens from the Engelhard blank (Pd-b-1 and 2) were analyzed and are shown in Table I. Two specimens from the hydride sample (PdH-c-1 and 2) and three specimens from the Johnson-Matthey blank (Pd-d-1, 2, and 3) were analyzed and are shown in Table II. The mass for the four specimens in the deuteride sample ranged from 17.07 to 23.09 mg, with an average of 20.76 mg. The specimens from the Engelhard blanks were 11.94 and 14.30 mg. The specimens in the hydride system were comparable in size to the deuteride specimens. The hydride specimens were 19.90 and 23.98 mg. The blank specimens were 16.50, 24.75, and 20.83 mg. No size dependence in the helium reading was observed among any of the specimens.

The ⁴He results for both the deuteride and hydride samples are presented in Figs. 1 and 2, which show the number of events compared with the net atoms released.

Figure 1 shows the following:

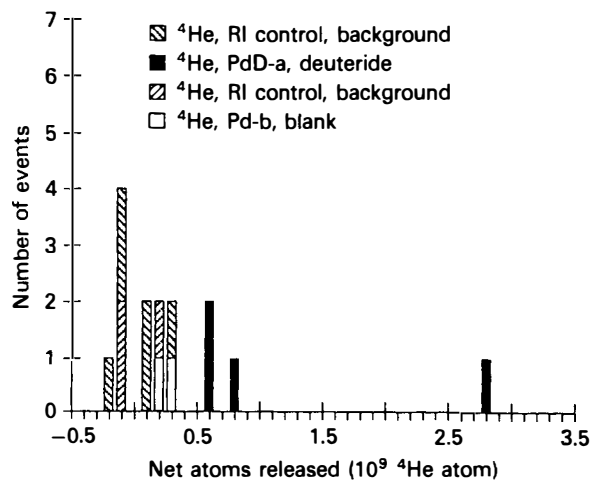


Fig. 1. Helium analysis results of the deuteride palladium and the Engelhard blank.

1. The signals of the four deuteride specimens are all substantially above those of the cold blanks, the RI controls, and the Engelhard blanks.

2. The results did not come from any spurious background signals.

3. The statistical significance of the results, namely, the probability of all positive readings for four specimens, is (1/2)⁴ or one part in 16. If the whole sequence is considered, the probability of all positive readings for four specimens in the sequence of 14 events (Table I) is (1/2)¹⁴, or one part in 16384. Considering the relatively large magnitude of the signals compared with those of the background and in one particular case 14σ above background, we believe the chance of an error is even smaller than 1 part in 16384.

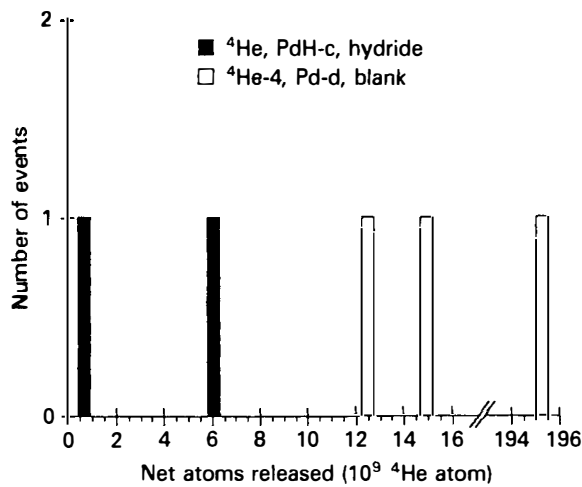


Fig. 2. Helium analysis results of the hydride palladium and the Johnson-Matthey blank.

4. the localization and inhomogeneity of the ^4He distribution in the deuteride sample.

Therefore, the enrichment of ^4He in the deuteride sample is most likely due to an as yet unidentified physical process and not experimental error.

Figure 2, however, reveals a high initial ^4He concentration in the Johnson-Matthey blank, which was unexpected. Apparently, the torching process did not remove the initial ^4He concentration completely, while the subsequent electrolysis in the molten salt substantially reduced the ^4He concentration. Again, the ^4He distribution was not uniform throughout the sample, implying the sporadic distribution is due to the inherent nature of the manufacturing process and not by adsorption and contamination from the ambient or experimental error.

The specific helium concentration for each specimen is summarized in Tables III and IV. The scattered readings and large standard deviations as noted in the reported values are the result of localized, nonuniform distribution of the helium atoms, not instrumentation errors.

Further SEM examination of the sectioned deuteride and Engelhard blank samples is shown in Figs. 3 and 4, respectively, revealing several interesting microstructural features of the bulk. A high content of spherical voids within the deuteride bulk PdD-a is shown in Fig. 3. The size of the voids ranges from tens of micrometres to several hundred micrometres in diameter. The blank Pd-b sample, however, shows only a few much smaller, scattered voids (Fig. 4), which are typically $<100\ \mu\text{m}$ in diameter. In both cases, some voids show a much darker image, indicating that they are possibly hollow channels. The nearly spherical voids are not cracks but may contain either deuterium or helium as a result of electrolysis.

DISCUSSION

From Tables I and III, we can conclude the following: the four deuteride specimens from PdD-a all show significant ^4He signals with an average of 4σ and, in one particular case, PdD-a-2, 12σ above the mean background signal in terms of specific concentration, or 14σ if interpreted from the

TABLE III

Summary of Helium Analysis Results for the Deuteride and Blank Samples*

Specimen	Specimen Mass ^a (mg)	Helium Concentration (10^9 atom/mg)	
		^3He	^4He
PdD-a-1	21.49	0.005 ± 0.01	0.03 ± 0.01
PdD-a-2	23.09	0.01 ± 0.01	0.12 ± 0.01
PdD-a-3	17.07	0.02 ± 0.02	0.05 ± 0.03
PdD-a-4	21.38	0.03 ± 0.01	0.03 ± 0.02
Mean $\pm 1\sigma$		0.02 ± 0.01	0.06 ± 0.04
Pd-b-1	11.94	0.02 ± 0.03	0.02 ± 0.03
Pd-b-2	14.30	-0.04 ± 0.02	0.02 ± 0.02
Mean $\pm 1\sigma$		-0.01 ± 0.04	0.02 ± 0.00

*Samples were prepared from the Engelhard lot.

^aMass uncertainty is ± 0.01 mg.

TABLE IV

Summary of Helium Analysis Results for the Hydride and Blank Samples*

Specimen	Specimen Mass ^a (mg)	Helium Concentration (10^9 atom/mg)	
		^3He	^4He
PdH-c-1	19.90	0.01 ± 0.02	0.04 ± 0.02
PdH-c-2	23.98	0.01 ± 0.01	0.26 ± 0.01
Mean $\pm 1\sigma$		0.01 ± 0.00	0.15 ± 0.16
Pd-d-1	16.50	0.01 ± 0.02	0.91 ± 0.02
Pd-d-2	24.75	0.01 ± 0.01	7.89 ± 0.16
Pd-d-3	20.83	0.04 ± 0.01	0.60 ± 0.01
Mean $\pm 1\sigma$		0.02 ± 0.02	3.13 ± 4.12

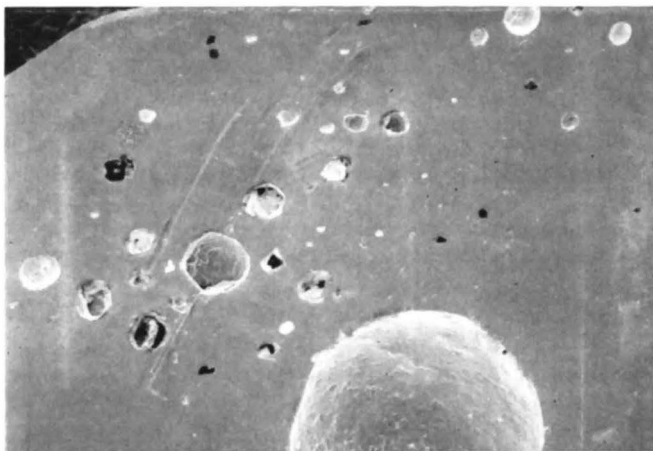
*Samples were prepared from the Johnson-Matthey lot.

^aMass uncertainty is ± 0.01 mg.

net atoms released. The Engelhard blank Pd-b does not contain any measurable amount of ^3He or ^4He . The deuteride sample indicates a ^4He enrichment after electrolysis.

Tables II and IV lead to different results: the Johnson-Matthey blank Pd-d shows a high initial ^4He content and in one specimen, Pd-d-2, about two orders of magnitude higher than those measured in the hydride specimens PdH-c. Apparently, the electrolysis reduced the ^4He content in the PdH-c sample; therefore, a ^4He depleting effect was found in the hydrogen-based system.

These ^4He analysis data are very intriguing when compared with calorimetric data.^{12,13} The excess heat reported in the deuteride system amounted to ~ 6 MJ from two experiments. The origin of the excess ^4He found in the deuteride sample is unknown at this time. A recent report by Bush



64.0× 20.0 kV 100 μm University of Hawaii Pd #0006*

Fig. 3. Section of the deuteride palladium bulk in which a relatively high density of voids was found.



106× 25.0 kV 100 μm AMRAY #0012

Fig. 4. Section of the Engelhard blank, where only few small voids were observed.

et al.¹¹ indicated a semiquantitative ^4He production in the effluent gases, which was correlated with the excess power generated during heavy water electrolysis. Assuming the excess heat and ^4He production originated from an as yet unidentified nuclear process, for example by



where d is the deuteron in the lattice, as suggested by Walling and Simons² and others, we would expect the 6 MJ excess heat would produce 1.6×10^{18} atoms of ^4He . From Table III, we can estimate a mean ^4He release of the whole deuteride palladium to be 2.9×10^{10} atoms. Therefore, there is a discrepancy in magnitude of $\sim 1.8 \times 10^{-8}$ between the retained ^4He in the solid and that expected from the excess heat.

We can explain the discrepancy as a result of most of the ^4He escaping into the effluent D_2 gas, as reported by Bush et al.¹¹ Abell et al.¹⁵ have reported that the ^4He , which en-

tered the palladium matrix, remains in the lattice at room temperature. However, the tendency for ^4He escaping increases with temperature. At our working temperature of $\sim 400^\circ\text{C}$, the amount of ^4He that escaped from the lattice would be expected to be much larger than at near room temperature. In addition, the situation could be more complex when macroscopic and microscopic defects, such as dislocations and cracks, are present in the lattice. If the majority of ^4He escaped into the effluent gas, leaving behind only a trace of ^4He in the sample, this would be consistent with ^4He production near the surface accompanied by a large number of defects and a large temperature gradient from the excess heat. There would be a drastic reduction in the expected concentration of ^4He , as in our case. This scenario has to be experimentally verified by quantitative measurements of the distribution of ^4He in the effluent gas and solid during excess heat production.

The amount of ^3He in all samples remains almost constant throughout the analyses. The variation of ^3He content is therefore either nil or, because it is so small in magnitude, below the range of instrumentation sensitivity. Nonetheless, the lack of a change in ^3He content implies that $p + d \rightarrow ^3\text{He}$, where p is the proton in the lattice, or $d + d \rightarrow ^3\text{He} + n$ reactions occur at a low rate, if at all.

Since a relatively comparable amount of ^4He was detected in both the deuteride and hydride samples after electrolysis, the possibility of atmospheric contamination cannot be disregarded. Nevertheless, we believe that this is unlikely to occur in our case based on the following assumptions:

1. The absorption of ^4He by palladium is generally negligible, although the adsorption on the surface is possible.

2. The detection of a very localized, scattered ^4He distribution, as shown in Figs. 1 and 2, implies that the introduction of ^4He into the lattice is also highly nonuniform and anisotropic.

Therefore, the observed enrichment and depletion effect were probably not caused by contamination from the ambient atmosphere, which would be expected to result in a uniform distribution. Atmospheric contamination would also be expected to give a natural isotopic distribution. Given the natural abundance of ^3He compared with ^4He to be 1.3×10^{-6} (Ref. 16), it is difficult to verify this scenario with the limited sensitivity of the instrument.

The substantial morphological difference between Figs. 3 and 4 was unexpected. Because of the similarity in void shape in both samples, we believe the creation of the voids is probably the result of sample preparation (torching), in spite of the difference in size, due to the relatively uncontrolled torching process.

It is difficult to understand how voids could be formed as a result of electrolysis. One possible mechanism may be the correlation of the region of high density of voids with the location of excess heat production. It has been suggested that excess heat production could be sporadic and localized, resulting in concentrated, but scattered, hot spots. These hot spots could result in high temperatures within a short time span, which could facilitate the deformation of the lattice to a great extent. This deformation could provide a favorable environment for substantially increasing the packing density of deuterons in the lattice (local loading), which would then result in the production of excess heat and the evolution and accumulation of helium without substantial lattice deformation and cracking.

CONCLUSION

The helium analysis of palladium samples subjected to molten salt electrolysis in deuteride- or hydride-conducting electrolytes at elevated temperatures is reported. Noticeable ^4He enrichment in the deuteride sample was found, while the opposite effect was measured in the hydride sample. No ^3He variation was noticed. Atmospheric contamination could, however, be a source of error but is considered unlikely. The bulk morphological studies revealed a much higher void density in the deuteride sample than in the blank.

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