INVESTIGATIONS OF THE DEUTERIUM-DEUTERIUM FUSION REACTION IN CAST, ANNEALED, AND COLD-ROLLED PALLADIUM

COLD FUSION

TECHNICAL NOTE

KEYWORDS: *cold D-D fusion, palladium, radiation detectors*

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Received December 29, 1989
Accepted for Publication May 17, 1990

A search was conducted for neutrons, protons, tritons, ^{3}He ions, gamma rays, and ion-induced X rays from deuterium-deuterium (D-D) fusion in cast (36-g), annealed (4-g), and cold-rolled (16-g) palladium specimens and a palladium hydrogen thermal valve (11 g) electrochemically charged with deuterium. The palladium cathodes were charged in an electrolytic cell [0.1 M LiOD (99.8% deuterium), platinum anode] at a current density of 25 mA/cm^2 from 20 to 140 h. One unique aspect of the experiment was the radiation detection system, consisting of a CR-39 track-etch detector, bare for proton detection (sensitivity limit 4.8 × 10^{-2} fusion/s), combined with a polyethylene fast neutron radiator (0.95 fusion/s), a boron thermal neutron radiator (26 fusion/s), a BD-100 bubble damage polymer detector (5.2 fusion/s), an array of six ^{3}He proportional counters (126 fusion/s), a CaF_2 thermoluminescent dosimeter (11.4 fusion/s), and a germanium semiconductor spectrometer (17 fusion/s). The D-D fusion rate in cast, annealed, and cold-rolled palladium is <3 × 10^{-22}, <7.8 × 10^{-21}, and <1.2 × 10^{-21} (D-Dn) fusion/D-D pair·s^{-1}, respectively. In the palladium hydrogen thermal valve, this value was <1.1 × 10^{-23} (D-Dp) fusion/D-D pair·s^{-1} and <2.3 × 10^{-22} (D-Dn) fusion/DD pair·s^{-1}.

I. INTRODUCTION

Recently it has been reported that deuterium loaded in hydride-forming metals (palladium, titanium) can undergo nuclear fusion at room temperature.[1,2] Release of heat, neutrons, tritium, and helium during the electrolytic charging of palladium and titanium in heavy water was reported.[1,2] It was suggested that

$$^2D + {}^2D \rightarrow {}^3T + {}^1H + 4.03\ MeV\ ,$$

$$^2D + {}^2D \rightarrow {}^3He + n + 3.27\ MeV\ ,$$

or

$$^2D + {}^2D \rightarrow {}^4He + 23.85\ MeV$$

takes place at the rate of ~10^{-19} (from neutron flux measurements)[1] or 10^{-12} to 10^{-9} (according to enthalpy generation)[1] and ~10^{-23} (neutron flux measurement)[2] fusion/deuterium-deuterium (D-D) pair·s^{-1}. However, most other laboratories have been unable to reproduce these results.[3-12]

In April 1989, a research program on cold fusion began at the J. Stefan Institute. The cold fusion laboratory was set up in a concrete basement at the Nuclear Training Centre. The laboratory was equipped with

1. proportional counter (an array of six ^{3}He type) for detection of thermal neutrons
2. bubble damage polymer detector (BD-100) for fast neutrons
3. semiconductor gamma-ray spectrometer [high-purity germanium (HPGe)] with a neutron radiator (H_2O) for thermal neutrons
4. track-etch detector (CR-39) for protons
5. track-etch detector (CR-39) with a boron (BN-1) radiator for thermal neutrons
6. track-etch detector (CR-39) with a polyethylene radiator for fast neutrons
7. thermoluminescent dosimeter (CaF_2) for detection of protons, and/or proton-induced X rays.

The neutron background and radon concentration were monitored by a BF_3 proportional counter and radon dosimeter utilizing a CR-39 detector.[3] The characteristics, sensitivity, and response to background radiation and lower limit of detection of the detectors were described in Ref. 3.

The influence of the treatment of the palladium cathode on the reaction $D_{ads} \rightarrow D_{lattice}$ that takes place during electrolysis and the corresponding influence on the fusion rate have not been systematically investigated. The aim of the present work is to analyze the influence of metal state (as-cast, as-worked, and heat-treated) on possible D-D cold fusion in electrochemically charged palladium. For this reason, a search for D-D fusion reaction products was carried out during electrochemical charging of cast (~36-g), annealed (~4-g), and cold-rolled (~16-g) palladium in 0.1 *M* LiOD at a current density of ~25 mA/cm^2.

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II. EXPERIMENTAL DETAILS

The electrochemical cell, radiation detectors, calibration, and measuring procedure were described in detail in Ref. 3; however, the experimental conditions are briefly summarized here. The electrochemical cell with palladium cathode (tube) and platinum anode is shown in Fig. 1. The electrochemical data are summarized in Table I. The electrolyte was made by dissolving chemically pure (99.9 wt%) lithium metal in 99.8% D_2O.

The properties of the palladium specimens are given in Table II. The specimens (1, 2, and 3) were made from pure (99.95 wt%) palladium. The melt for specimen 1 was made in an induction vacuum furnace (130 Pa) using an Al_2O_3 crucible. The melt was chill cast under vacuum from 1650°C into a metal mold. Note that the vacuum conditions were not adequate to completely avoid oxygen and nitrogen pickup. The starting material for specimen 2 was a palladium rod (diameter = 11 mm). The rod was cold rolled, followed by recrystallization annealing at 700°C. The cold-work-anneal cycle was repeated until the diameter of the palladium rod was 4 mm. After the last deformation, annealing was not carried out. The starting material for specimen 3 was an annealed palladium rod (diameter = 4 mm). It was cold drawn to 2 mm in diameter and then annealed at 700°C in a closed outgassed quartz tube for 15 min. The difference in grain size and/or orientation among the different palladium treatments is illustrated in Fig. 2, where optical micrographs are presented. The values of mean grain size and hardness are given in Table II. Some of the data obtained with a palladium hydrogen thermal

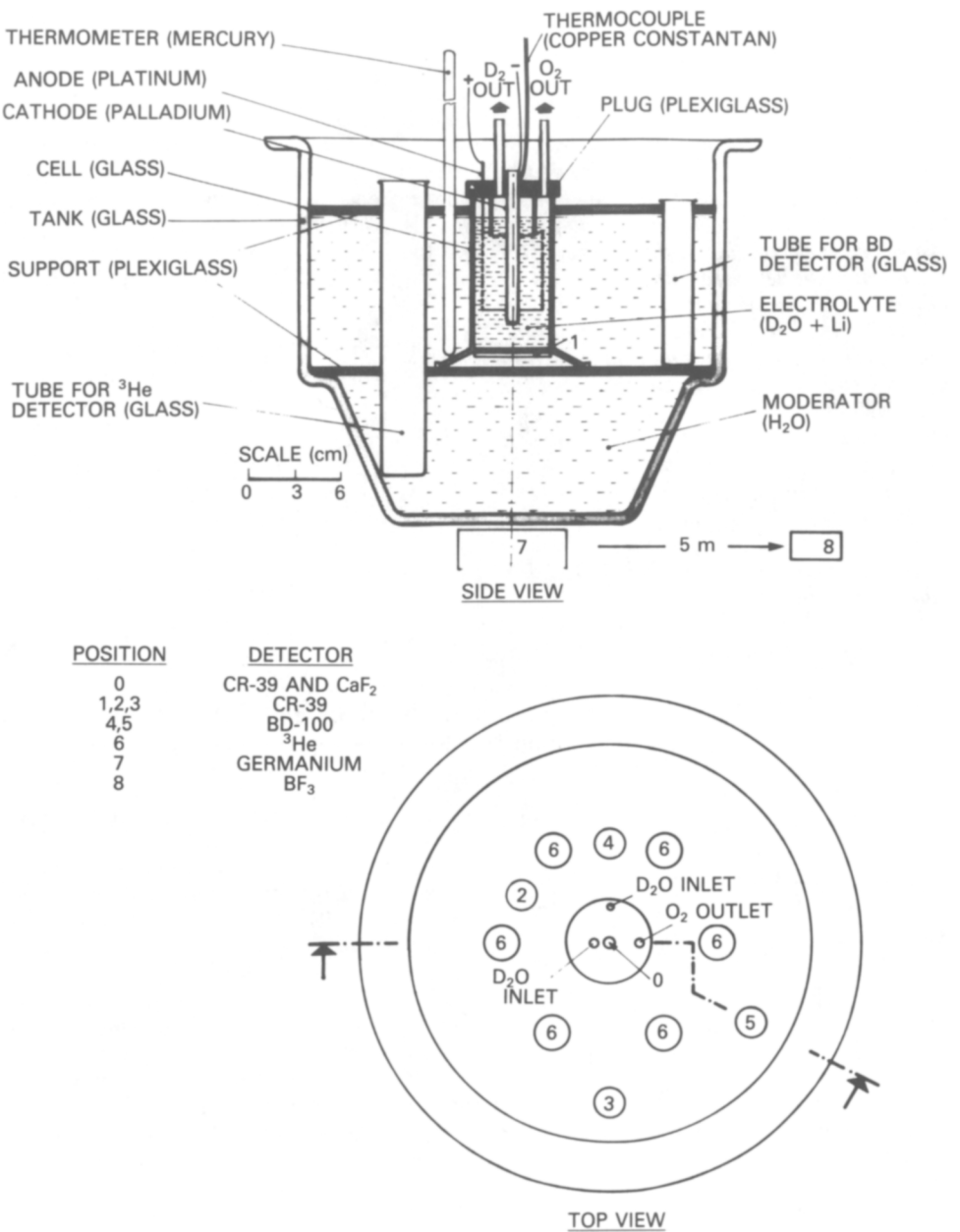

Fig. 1. Layout of the electrochemical cell and radiation detectors. The characteristics of the detectors are given in Ref. 3.

TABLE I

Electrochemical Data

Cathode[a]	Palladium
Anode	Platinum
Shape	Mesh
Size (cm)	diam = 4, length = 5
Weight (g)	21.25
Electrolyte	LiOD (99.8% deuterium)
Volume (ml)	160
Concentration (M)	0.1
Temperature (°C)	21
Current density (mA/cm^2)	25 ± 2
Potential[b] (V)	3 to 4.8

[a]Different shape and treatment (see Table II).
[b]Depending on the shape and size of the specimen.

valve for ion sources (specimen 4) (Ref. 3) are also included for completeness.

Prior to electrolysis, the palladium specimens were outgassed in a vacuum of $\sim 10^{-3}$ Pa at ~450°C for ~3 h and stored in D_2O. The D/Pd atom ratio was determined by accurately weighing the palladium specimens prior to and after electrolysis.

A copper-constantan thermocouple was fixed on the palladium cathode. During the charging time, the temperature of the cathode was recorded with an accuracy of ±0.1°C.

The positions of the radiation detectors (Table III) used in our experiment are marked in Fig. 1. The experimental (for neutrons) and computational (for protons) procedures to determine the total efficiency η of the detectors to relevant fusion products were described in Ref. 3. Values of η are summarized in Table IV.

III. RESULTS

The lowest detectable yield intensity Y_D (defined as the number of fusions per unit time) is given in Table IV. The value of Y_D was calculated by

$$Y_D = \frac{L_D}{\eta b t} , \quad (1)$$

where

L_D = detection limit of a given detector for qualitative determination

b = branching ratio (0.5)

t = charging time.

The definition of L_D is[3]

$$L_D = 2.71 + 3.29\sigma_B , \quad (2)$$

where σ_B is the standard deviation of the blank. The blank is defined as the signal resulting from an experiment in which conditions are identical to the experiment of interest, except that no Pd-D_2O electrolysis occurs. In Ref. 3, for our experimental conditions the following equations were determined:

$$\begin{aligned} \sigma_B &= [\rho_b A + (sA)^2]^{1/2} && \text{for CR-39} \\ &= 0 && \text{for BD-100} \\ &= c\mu_B t && \text{for } ^3\text{He and } BF_3 \\ &= [\sigma_a^2 + (r\mu_B t)^2]^{1/2} && \text{for } CaF_2 \\ &= (\mu_B t)^{1/2} && \text{for germanium detector} , \end{aligned} \quad (3)$$

TABLE II

Properties of Palladium Specimens and Charging Surface/Mass Ratio S_{ef}/m_{ef}

Specimen Number	Shape	Treatment	Hardness HV5	Mean Grain Size (μm)	Size[a] (cm)		Mass (g)		S_{ef}/m_{ef} (cm^2/g)
					Total	Effective	Total	Effective[b]	
1[c]	Cylinder	Cast	48	~400	$\phi = 1.6$ $l = 1.5$		36.834	30.414	0.44
2	Rod	Annealed	80	~70	$\phi = 0.2$ $l = 11$	$l_{ef} = 7.1$	4.285	2.727	1.62
3	Rod	Cold rolled	114	[d]	$\phi = 0.4$ $l = 11$	$l_{ef} = 7$	16.438	10.61	0.85
4[e]	Tube				$\phi_i = 0.55$ $\phi_o = 0.65$ $l = 10$	$l_{ef} = 7$	11.178	7.825	1.9

[a]Inner and outer diameters of specimen 4 are denoted by ϕ_i and ϕ_o, while the diameter of other specimens is denoted by ϕ. Total and effective (submerged in the electrolyte) length are denoted by l and l_{ef}, respectively.
[b]Submerged in the electrolyte.
[c]The cylinder was fixed to a palladium rod (ϕ = 0.2 cm, l = 20 cm) of which 3 cm was submerged in the electrolyte.
[d]Seventy percent reduction in cross-sectional area.
[e]Palladium hydrogen thermal valve for ion sources.[3]

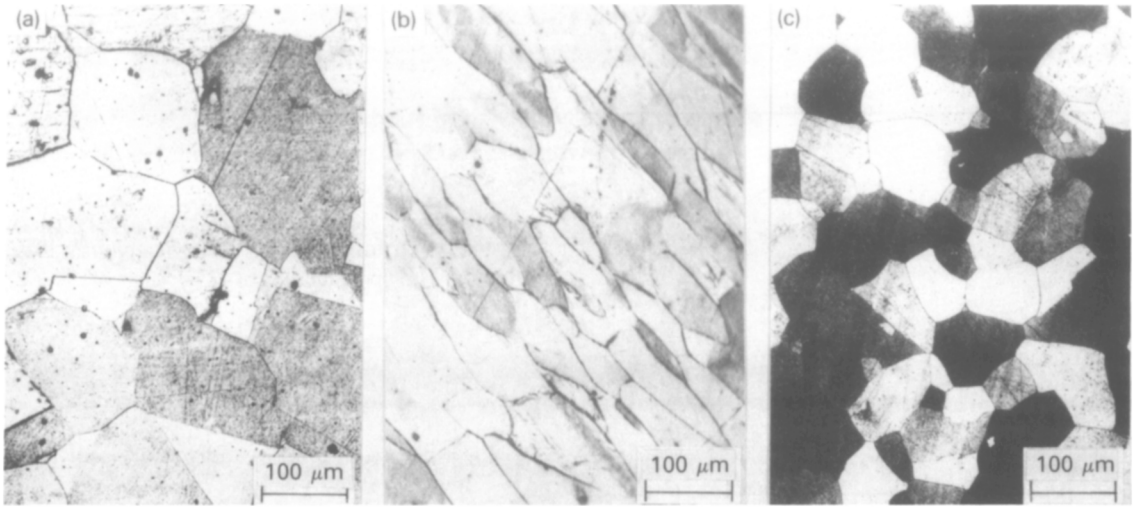

Fig. 2. Optical micrographs of (a) cast, (b) cold-rolled, and (c) annealed palladium.

TABLE III

Characteristics of Radiation Detectors Used in Cold Fusion Experiment

Detector	Type	Producer/Model	Fusion Product Measured	Main Detection Reaction
Track-etch detector	CR-39	Tastrak	p n	[a] $^1H(n_f,p)$[b] $^{10}B(n_{th},\alpha)$[c]
Bubble damage polymer detector	BD-100	Bubble Technology Industry	n	$^1H(n_f,p)$
Thermoluminescent dosimeter	CaF_2:Mn	J. Stefan Institute	p	Protons and p-induced X rays
Proportional counter	^3He	Texlium Cat. No. 9333	n	$^3He(n_{th},p)$
	$^{10}BF_3$	N. Wood Model G-10-5	n	$^{10}B(n_{th},\alpha)$
Semiconductor Spectrometer	Germanium	Canberra HPGe	n	$^1H(n_f,p)$

[a]Direct detection.
[b]With polyethylene fast neutron radiator, produced by Totra, Yugoslavia.
[c]With boron (BN-1) thermal neutron radiator, produced by Kodak Pathe, France.

where

ρ_b = background (prior to irradiation) track density (~ 12 cm^{-2})

A = measured area (0.3 cm^2)

s = constant that describes the variation of the background track density (5 cm^{-2})

c = experimentally determined constant (0.6 and 0.3 for ^{3}He and $^{10}BF_3$ detectors, respectively)

μ_B = mean rate of the blank (4.4×10^{-2} and 1.7×10^{-3} count/s for ^{3}He and $^{10}BF_3$ detectors, respectively)

σ_a = error of the signal of an unexposed fresh pellet (1 ± 1 μGy)

r = relative error of the thermoluminescent signal (3%)

μ_b = mean rate of the gamma-ray background radiation (2.3×10^{-3} count/s) in the energy intervals 2.22 to 2.23 MeV for the germanium detector and rate of the background ionizing radiation (1.5×10^{-4} μGy/s) as recorded by the CaF_2 detector.

Using these data, Y_D was calculated for a charging time of 10 days. The data given in Table IV indicate that the most sensitive detector is proton sensitive bare CR-39 ($Y_D = 4.8 \times 10^{-2}$ s^{-1}), followed by fast neutron sensitive detectors CR-39/polyethylene (0.95 s^{-1}) and BD-100 (5.2 s^{-1}).

The value of the gross signal-to-blank ratio S/B is given in Table IV. The gross signal is defined as the observed value (number of tracks, bubbles, or counts) during electrolysis

TABLE IV

Total Efficiency η to Neutrons ($E_n = 4 \pm 2$ MeV) and Protons (3 MeV), Lowest Detectable Yield Intensity Y_D, and Gross Signal-to-Blank Ratio S/B Obtained in Electrochemically Charged Palladium: Cast Cylinder (Specimen 1), Annealed Rod (2), Cold-Rolled Rod (3), and Hydrogen Thermal Valve (4)

Detector[a] / Radiator	Position[b]	η[c]	Y_D[d] (s^{-1})	S/B[e] Specimen Number[f] 1[g]	2[g]	3[g]	4[h]
CR-39	0	5.1×10^{-4}	0.048[i]				1.3 ± 0.4
CR-39/BN-1	1	9.6×10^{-7}	26	1.0 ± 0.8	1.0 ± 0.8	1.3 ± 0.8	1.0 ± 0.4
	2	2.7×10^{-7}	91	1.8 ± 1.0	1.3 ± 0.8	0.8 ± 0.6	1.4 ± 0.5
CR-39/polyethylene	0	2.6×10^{-5}	0.95				1.3 ± 0.5
	1	7.2×10^{-7}	34	0.8 ± 0.6	0.8 ± 0.7	1.0 ± 0.8	0.8 ± 0.3
	2	7.2×10^{-8}	343	2.0 ± 1.2	2.4 ± 1.4	1.3 ± 0.8	0.7 ± 0.3
BD-100	4	1.2×10^{-6}	5.2	0/0	0/0	0/0	
	5	8.9×10^{-7}	7.0	0/0	0/0	0/0	
CaF_2:Mn	0	2.5×10^{-4}	13.2				1.0 ± 0.05
^{3}He	6	1.4×10^{-3}	126	1.2 ± 1.0	0.8 ± 0.7	0.5 ± 0.4	0.8 ± 0.3
Germanium[j]	7	2.0×10^{-5}	17	1.01 ± 0.07		1.02 ± 0.08	

[a]Details of the detection system are given in Ref. 3.
[b]Position of the detectors is marked in Fig. 1.
[c]Experimental and/or computational procedure is described in Ref. 3.
[d]At proton/neutron emission ratio of 1 and charging time of 10 days.
[e]Signal S is defined as observed value during electrolysis (palladium, LiOD) and blank B as observed value without electrolysis.
[f]The characteristics of the specimens are given in Table II.
[g]Present work.
[h]Recalculated from the data given in Ref. 3.
[i]Only for specimen 4.
[j]For 2.225-MeV gamma rays in the energy interval from 2.22 to 2.23 MeV (see Ref. 3).

TABLE V

Charging Time t, Measured Value of D/Pd Ratio, Number of Deuterium Pairs $N_{D\text{-}D}$, and Lowest Detectable Fusion Rate λ_D

Specimen Number	Treatment	t (h)	D/Pd	$N_{D\text{-}D}$ ($\times 10^{22}$)	λ_D[a] (fusion/D-D pair·s^{-1}) n Branch	p Branch
1[b]	Cast	69.55	0.69	5.94	3.0×10^{-22}	
2[b]	Annealed	24.3	0.85	0.66	7.8×10^{-21}	
3[b]	Cold rolled	44.85	0.75	2.37	1.2×10^{-21}	
4[c]		140.5	0.48	1.06	2.3×10^{-22}	1.1×10^{-23}

[a]For proton/neutron emission ratio of 1. The most sensitive radiation detection systems: BD-100 (position 4) for specimens 1-3 and bare CR-39 (position 0) and CR-39/polyethylene (position 0) for specimen 4 were considered in this calculation.
[b]Present work.
[c]Ref. 3.

(Pd-D_2O) for the charging time given in Table V. The blank is defined as the observed value without electrolysis from experiments in which conditions were identical to the experiment in interest. The blank was measured with D_2O in the cell without electrolysis for 73 h (Ref. 3). From the results given in Table IV, obtained from four specimens with seven radiation detection systems, it is evident that no statistically significant deviation from the background was observed. No

thermal excursion was registered with the copper-constantan thermocouple fixed on the palladium cathodes, and no temperature variation was observed with the mercury thermometer placed in H_2O moderator. This supports the absence of cold fusion in our electrolytic cell.

The lowest fusion rate detectable λ_D is defined as[3]

$$\lambda_D = \frac{Y_D}{N_{D\text{-}D}} , \quad (4)$$

where $N_{D\text{-}D}$ is the number of deuterium pairs:

$$N_{D\text{-}D} = \frac{1}{2} n_{Pd} \frac{D}{Pd} m_{ef} , \quad (5)$$

where

n_{Pd} = palladium atom density (5.66×10^{21} atom/g)

D/Pd = average deuterium/palladium atom ratio

m_{ef} = effective (submerged in the electrolyte) mass of the palladium cathode.

The ratio $D/Pd = (R - 1)M_{Pd}/M_D$ was determined from the measured ratio of the effective mass of the palladium specimen at the end of and prior to electrochemical charging R and the ratio of the atomic weight of palladium and deuterium M_{Pd}/M_D. The measured values of R at different times t_e elapsed after charging are given in Fig. 3. The value of m_{ef} is given in Table II, while values of D/Pd and the corresponding $N_{D\text{-}D}$ are listed in Table V. The deuterium loading in palladium was slightly dependent on the palladium treatment or size of the specimens. Therefore, one can suppose that the fusion rate in the bulk material is independent of the grain size, grain orientation, and number of crystal imperfections.

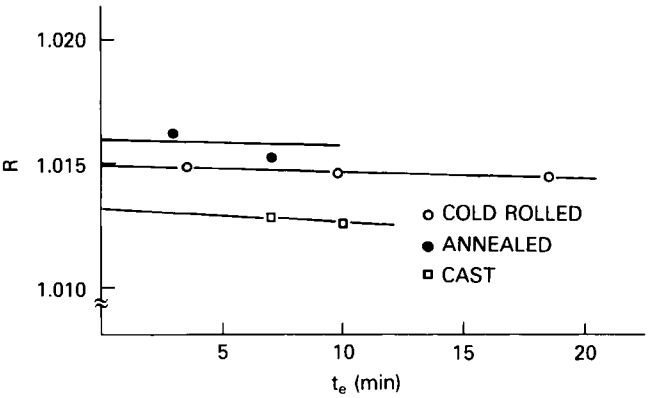

Fig. 3. Measured values of the ratio of effective (submerged in the electrolyte) masses of the palladium cathodes R as a function of time t_e elapsed after electrochemical charging. The value of R extrapolated to the end of charging ($t_e = 0$) was used in the calculation.

TABLE VI

Measured Value of Fusion Rate λ in Electrochemically Charged Palladium with Deuterium

λ (fusion/D-D pair·s^{-1})		Detector (Spectrometer)	Institution	Reference
n Branch	*p* Branch			
$<3.0 \times 10^{-22}$		BD-100	J. Stefan Institute, Ljubljana, Yugoslavia	Present work
$<2.3 \times 10^{-22}$	$<1.1 \times 10^{-23}$	CR-39	J. Stefan Institute, Ljubljana, Yugoslavia	3
$<5.0 \times 10^{-22}$		BF_3	Iowa State University	4
$<2.0 \times 10^{-23}$		^3He	Sandia National Laboratories	5
$<2.2 \times 10^{-24}$		NaI(Tl)	AT&T Bell Laboratory	6
$<2.0 \times 10^{-25}$		NE-213	Yale University and Brookhaven National Laboratory	7
$<1.0 \times 10^{-22}$		Germanium	University of California and Lawrence Berkeley Laboratory	8
$<4.0 \times 10^{-24}$		NE-912	R. Bošković Institute, Zagreb, Yugoslavia	9
$<2.0 \times 10^{-21}$	$<3.0 \times 10^{-16}$	Germanium	Battelle-Pacific Northwest Laboratories	10
$<2.7 \times 10^{-24}$		[a]	Lawrence Berkeley Laboratory	11
$<3.6 \times 10^{-23}$		BF_3	Technische Universität and Atominstitut, Vienna, Austria	12
$\sim 10^{-19}$		NaI	The University of Southampton, United Kingdom, and University of Utah	1
$\sim 10^{-23}$		BC-505	Brigham Young University and University of Arizona	2

[a]Liquid scintillator (type not specified).

Using these data, and data for charging time (Table V), λ_D was calculated for all specimens, taking into account the most sensitive detector/position system [for specimens 1, 2, and 3, BD-100 (*n*-branch), position 4; for specimen 4, bare CR-39 (*p*-branch) and CR-39/PE (*n*-branch), position 0]. The results presented in Table V show that the D-D fusion rate in cast, annealed, and cold-rolled palladium is $<3 \times 10^{-22}$, $<7.8 \times 10^{-21}$, and $<1.2 \times 10^{-21}$ (D-D*n*) fusion/D-D pair·s^{-1}. The λ_D in the palladium hydrogen thermal valve was found[3] to be $<2.3 \times 10^{-22}$ (D-D*n*) fusion/D-D pair·s^{-1} and $<1.1 \times 10^{-23}$ (D-D*p*) fusion/D-D pair·s^{-1}.

Measured values of the fusion rate λ in electrochemically charged palladium with deuterium, obtained in other laboratories under similar experimental conditions, and with various types of radiation detectors, are summarized in Table VI. The results given in Table VI do not support the claims of Fleischmann and Pons[1] and of Jones et al.[2]

IV. CONCLUSIONS

A search for D-D fusion reaction products in electrochemically charged cast (36-g), annealed (4-g), and cold-rolled (16-g) palladium specimens, and a palladium hydrogen thermal valve (11 g) was carried out with seven different radiation detectors.

The lowest detectable yield intensity Y_D of our experiment for a charging time of 10 days was 5.2 (D-D*n*) fusion/s detected by BD-100 for cast, annealed, and cold-rolled palladium specimens. The Y_D for the palladium hydrogen thermal valve was 4.8×10^{-2} (D-D*p*) fusion/s and 0.95 (D-D*n*) fusion/s, obtained by bare CR-39 and CR-39 in combination with polyethylene.

Using these detector systems, we found that the rate of cold fusion λ in cast, annealed, and cold-rolled palladium, electrochemically charged in 0.1 *M* LiOD at a current density of ~25 mA/cm^2 is $<3 \times 10^{-22}$, $<7.8 \times 10^{-21}$, and $<1.2 \times 10^{-21}$ (D-D*n*) fusion/D-D pair·s^{-1}, respectively. The λ in the palladium hydrogen thermal valve was found to be $<2.3 \times 10^{-22}$ (D-D*n*) fusion/D-D pair·s^{-1} and $<1.1 \times 10^{-23}$ (D-D*p*) fusion/D-D pair·s^{-1}. No thermal excursion was observed in our electrolytic cell, supporting the absence of cold fusion.

ACKNOWLEDGMENTS

The authors are grateful to their colleagues from the reactor physics department and M. Pleško from Sincrotrone, Trieste, for helpful discussions during the initial stages of this experiment.

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