Botta, E., et al. *Measurement of 4He Production from D2 Gas-Loaded Pd Samples*. in *Sixth International Conference on Cold Fusion, Progress in New Hydrogen Energy*. 1996. Lake Toya, Hokkaido, Japan: New Energy and Industrial Technology Development Organization, Tokyo Institute of Technology, Tokyo, Japan.

# Measurements of <sup>4</sup>He Production from D<sub>2</sub> gas-loaded Pd Sample

E. Botta(1), T. Bressani (1,2), D. Calvo(1), C. Fanara(1,3), F. lazzi(1,4)

- (1) INFN, Sezione di Torino, I-10125, Torino, Italy
- (2) Dipartimento di Fisica Sperimentale, Universita di Torino, I-10125, Torino Italy
- (3) Fiat Avio, I-10129, Torino, Italy
- (4) Dipartimento di Fisica del Politecnico di Torino, I-10129, Torino Italy

#### Abstract

A Pd sheet gold plated at both ends was loaded with  $D_2$  in a Cold Fusion cell operating on the basis of the Coehn effect. The procedure was the same followed in a previous experiment. The gas has been analyzed by means of a high resolution mass spectrometer before the absorption into Pd and after desorption: a significant presence of  $^4$ He after desorption has been measured, in agreement with the previous results.

#### 1. Introduction

In the frame of the Cold Fusion research, the aim of the Torino group was always to search for signatures of nuclear reactions occurring inside D-Metal systems.

Following this line, after a set of experiments devoted to the detection of 2.45 MeV neutrons [1, 2] with a suitably designed detector [3, 4, 5], the efforts were directed to the measurement of the  $^4$ He content in the  $D_2$  gas after absorption and desorption from a Pd metal lattice. Since in the scientific debate about Cold Fusion it is generally accepted that the atomic ratio  $\alpha = [D]/[Pd]$  plays a quite crucial role for the positive measurement of neutron production,  $^4$ He content and heat excess, particular care has been devoted to the technique of loading the Pd lattice with D over the thermodynamical value  $\alpha = 0.67$ . The Torino group has chosen a technique based on the Coehn effect that allows the  $d^+$  ions to move along a Pd conductor toward the cathode.

A cell has been designed and constructed to take advantage of this effect in order to accumulate the Deuterium in a volume near the cathode, up to values of  $\alpha$  greater than 0.67. The complete apparatus has been already described [6] and results of a first set of measurements (dedicated mainly to explore the best operating parameters) were shown during the ICCF5 (1995) Conference. The following main conclusions were reached in those measurements: a) the electromigration of  $d^+$  ions (Coehn effect) could be applied to gas loaded Pd sheets and seemed to be effective to speed up the loading of  $D_2$  into Pd up to  $\alpha \approx 0.7$ ; b) higher gas pressure and accurate polishing of the Pd surface helped the absorption; c) the temperature of the Pd sample should not exceed 70 °C in order to avoid thermomechanical troubles.

The  $D_2$  gas desorbed from two different Pd samples was analysed by means of a high resolution mass spectrometer and in one case an amount of  $7.5 \cdot 10^{16}$  <sup>4</sup>He atoms was observed, roughly corresponding to a specific power of ~25 W/cm<sup>3</sup>.

In the experiment we are presenting here, the experimental set-up was the same (a part for some small modifications in the vacuum circuit) and the Pd sheet used was shaped and gold plated following the indications obtained in the previous experiment. Four values of the electric field were applied to the Pd sample for different time intervals and after the last two absorption-desorption runs, the gas was analyzed and the <sup>4</sup>He content was measured by the high

### 2. Experimental apparatus and calibrations

The experimental apparatus has been completely described in a previous paper [6]; only the main features are reported here. The cell ( $166\pm1~\text{cm}^3$  volume), containing a Pd sheet clamped by two copper electrodes (see Fig. 1), is connected to a vacuum circuit ( $3900\pm5~\text{cm}^3$ ) equipped with two Turbomolecular pumps and a mass spectrometer (ULVAC HI-RESOM 2SM) with resolution M/ $\Delta$ M=200 (see Fig. 2).

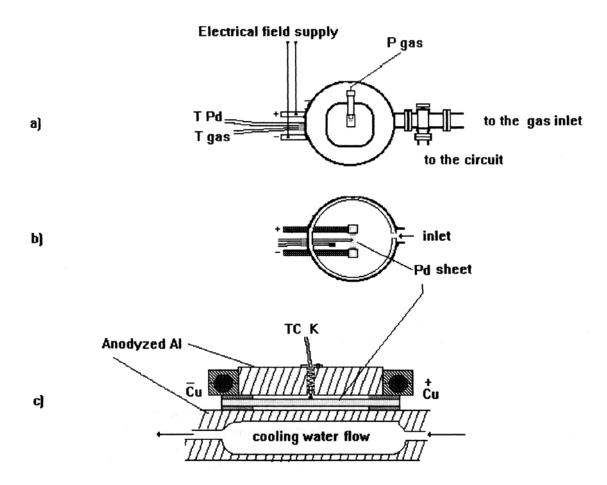


Fig. 1 Cell layout (not in scale); a) External top view, b) Internal top view (upper cover removed), c) lateral view.

To ensure a good quality of the measurements, a particular care has been taken in the vacuum control of the circuit and in the calibration of the instrument. Several days of degassing led to an overall residual pressure of  $2 \cdot 10^{-6}$  mbar. The switching-off of the pumps brought to  $2 \cdot 10^{-5}$  mbar in two hours, a value that remained quite constant over two days, proving then the tightness of the circuit. The calibration of the spectrometer was performed introducing at subsequent times a known amount of  $^4$ He into the analyzing tube and recording the peak heights (i.e. the ionic currents) and the pressure in the region M/e=3.9 - 4.1.

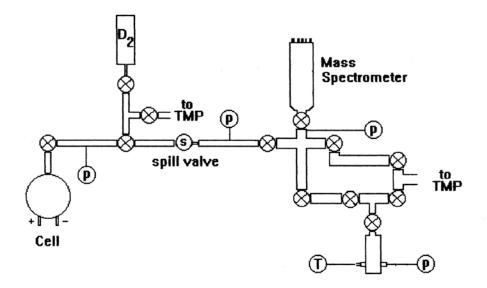


Fig. 2 Experimental apparatus: vacuum circuit, Coehn Cell and mass spectrometer. (TMP = Turbomolecular pump, T = Thermocouple, P=pressure gauges).

The sensitivity, defined as the ratio between current and partial pressure value, calculated from repeated measurements, gave a mean value of  $\varepsilon = (2.20\pm0.17)\cdot10^{-4}$  A/mbar, showing a quite good stability of the instrument. The pressure range variation of these measurements, between  $2\cdot10^{-5}$  and  $1\cdot10^{-4}$  mbar, was the same during the experiment.

#### 3. Results and Discussion

A Pd sheet  $(8 \times 1 \times 1 \cdot 10^{-2} \text{ cm}^3)$ , gold plated at both ends for a length of 1.5 cm (gold thickness  $\approx 15 \mu m$ ), has been clamped by the two Cu electrodes inside the cell.

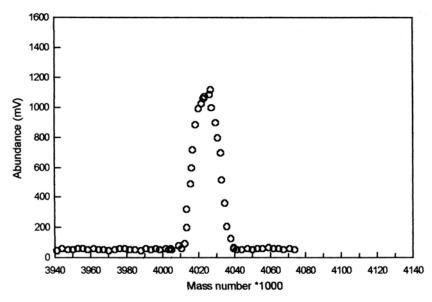


Fig. 3 Mass analysis of the gas immitted in cell in the M/e 3.9 - 4.1 range (blank measurement). Only the  $D_2^+$  peak (M/e=4.028) is visible.

During degassing the cell has been continuously in contact with the vacuum circuit. After an immission of  $D_2$  gas, reaching a final pressure of 2.7 bar, the valve separating the cell from the rest of the circuit was closed and the gas herein was analyzed by the spectrometer. This analysis is considered the blank measurement (see Fig. 3).

After 18 hours from gas immission, the loading of the Pd sample reached a value of  $\alpha \approx 0.5$  due to thermodynamical absorption (without electric stimulation). At the beginning (first 5 hours) of this time interval, the  $\alpha$  ratio increased rapidly, while later on, it rose much slower, as shown in Fig. 4.

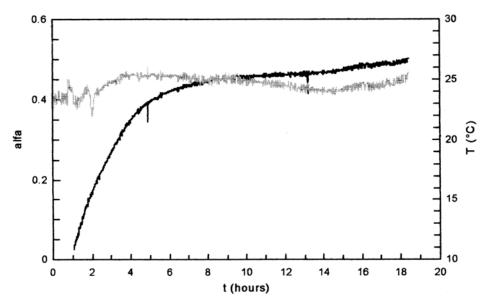


Fig. 4 Variation of the loading ratio  $\alpha$  measured by means of the thermodynamic parameters (black) and sample mean temperature (grey), during the first 18 hours.

The sheet temperature was continuously monitored and looks quite constant (after an initial transient) at the equilibrium (around 25°C) between the exothermic absorption and the external cooling water temperature.

Table 1 summarizes the sequence of the operations performed on the sample. The first column shows the start time of the operation from gas immission; in the second column the mean value of the loading ratio (averaged on the whole sheet volume) before the operation is reported; the mean and maximum values of the applied current appear in the third column, while its duration is listed in the fourth column. The fifth column indicates whether a gas analysis has been performed at the end of the operation involved, and the last one reports whether a <sup>4</sup>He production above background level has been observed.

Table	1 _	Operations	nerformed	on Pd sheet
1 abic	1 -	VIIVI AUVIIS	DCI IVI IIICU	vii i u siicci

Time (hours)	< \a>	I (A)	Duration (hours)	Gas analysis	<sup>4</sup> He
18	$0.48\pm0.02$	1 50 max	0.3	No	No
		125 mean			
65	$0.66\pm0.02$	225 max	2	No	No
		100 mean			
95	$0.83\pm0.02$	300 max	1	Yes	No
		175 mean			
117	$0.80\pm0.02$	440 max	0.4	Yes	Yes
		330 mean			

During the operation with electric field, the thermocouples measuring the sheet temperature were disconnected due to some noise caused by the very high circulating currents. Fig. 5 shows the behaviours of the current and of  $\alpha$  during the application of the electric fields corresponding to the last row of the Table 1.

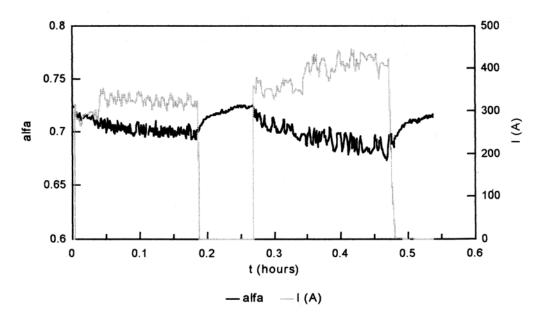


Fig. 5 Behaviour of loading ratio  $\alpha$ , measured by means of the thermodynamic parameters (black), during application of electrical current (grey).

The decrease of  $\alpha$  when the electric field is applied and the increase when the field is switched off, have been interpreted in the following way: the electric field favours electromigration of the  $d^+$  ions toward the cathode raising the concentration in the gold coated zone, but also favours desorption of D from the Pd uncoated region (this last phenomenon increases the pressure of the external  $D_2$  gas); as the current is switched off, probably  $\alpha$  is higher near the cathode, while the uncoated central region is deeply depleted; in this region, without electric field, the Joule effect stops and  $D_2$  is strongly adsorbed, producing an increase of the average a.

The desorbed gas was analysed by the mass spectrometer (see rows 3 and 4 of table 1); the results of the last analysis are shown in Fig. 6, where a peak around the atomic mass of <sup>4</sup>He is evident.

The analysis of the gas was repeated several times, always giving the same clear evidence of <sup>4</sup>He presence.

From the peak height a partial pressure, and thus a percentage of  ${}^{4}$ He in the analyzed gas, was obtained; in this way, a total value of  $(5.3+0.7)\cdot10^{18}$  atoms produced in the cell has been inferred.

Since the <sup>4</sup>He presence in the background measurement (Fig. 3) is not detectable, the production inside the gas loaded Pd sheet is at least equal to this value: in fact, an amount of produced <sup>4</sup>He could have been trapped inside the Pd sheet.

Some comments can be made about the very high value reported above, corresponding to the same number of nuclear reactions

$$d + d \rightarrow {}^{4}He + 23.8 \text{ MeV}$$
 (1).

The heat excess (in the simplified hypothesis that all the energy in (1) is converted into heat), of the order of  $(2.0\pm0.3)\cdot10^7$  J, is compatible with the values (heat or <sup>4</sup>He production) reported by other experiments. Considering nuclear reactions, their occurrence seems quite well established by so huge numbers.

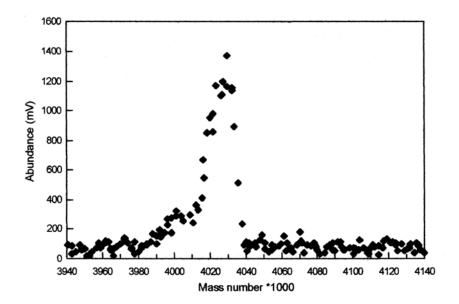


Fig. 6 Mass analysis of the gas desorbed from the Pd sheet after the fourth application of electrical field (see table 1). A  $^4$ He peak (M/e=4.0026) is appreciable near the  $D_2$  one

(M/e=4.028).

Looking at the generated power, this experiment cannot determine the time interval during which the nuclear reactions occurred inside the Palladium. In fact, the total time of the experiment overcome 5 days and the sheet was considerably loaded nearly from the beginning. As extreme hypothesis, the  $^4$ He production could start quite soon; in this case the average production rate should be of  $(1.2\pm0.2)\cdot10^{13}$   $^4$ He/s (corresponding to a generated power of  $46\pm7$  W). But, if we believe that every application of the electric field is sufficient to empty the sheet from the adsorbed gas, the production should be considered as starting from the first field application; in this case, the total time should be  $7.56\cdot10^4$  s, with a production rate of  $(7.0\pm0.1)\cdot10^{14}$   $^4$ He/s and a generated power of the order of  $(2.6\pm0.4)\cdot10^2$  W. So, the corresponding excess heat, is fully compatible with the tens of Watts generally reported by other experiments.

As a final remark, the total Pd sample volume in which the nuclear reactions occurred was of  $8\cdot10^{-2}$  cm<sup>3</sup>: the <sup>4</sup>He production density should then be  $(6.6\pm0.9)\cdot10^{19}$  <sup>4</sup>He/cm<sup>3</sup> and the rate per unit volume, in the shorter production time hypothesis, is  $(8.8\pm0.1)\cdot10^{15}$  <sup>4</sup>He/(s-cm<sup>3</sup>), corresponding to a power production of  $(3.3\pm0.5)\cdot10^{3}$  W/cm<sup>3</sup>

## 4. Conclusions and Perspectives

A new technique, based on the Coehn effect applied to Pd sheets, has been used in order to overload Pd with D over the thermodynamical limit a=0.67. A sequence of four values of electric fields with increasing maximum current intensities, was applied, each separated by periods of field off. In this period a loading value exceeding a= 0.8 has been reached. The mass spectroscopic analysis of the gas desorbed from the Pd sheet shows a clear peak at the atomic mass corresponding to <sup>4</sup>He, while a similar measurement performed on the gas before the absorption shows a background consistent with zero.

The  ${}^{4}$ He production is of the order of  $5 \cdot 10^{18}$  atoms, a strong evidence for the occurrence of nuclear reactions.

Concerning the rates, the sequence of operations in the present experiment, didn't allow the determination of the time interval in which the <sup>4</sup>He was produced, as well as no precise indication of local overloading values (perhaps greater than 0.8 - 0.9) could be inferred from the data.

Future efforts will be directed to explore a sequence of adsorption-desorption cycles with more frequent spectroscopic analysis of the gas (still remaining successful in the <sup>4</sup>He production); a second goal will be to reach a better control of the temperature and voltages along the Pd sheet, in order to deduce more information about the electromigration and local concentration of the deuterium in the metallic lattice.

## **Acknowledgments**

We wish to thank technical staff of INFN - Sez. Torino, and particularly O. Brunasso and G. Dughera for their assistance, even outside their usual duties. We feel also indebted to Mr. U. Ferracin for some useful suggestions.

#### References

- [1] T. Bressani et al., II Nuovo Cimento 104 A (1991) 1413.
- [2] T. Bressani et al., II Nuovo Cimento 105 A (1992) 1663.
- [3] G.C. Bonazzola et al. NIM A299 (1990) 25
- [4] F. Benotto et al. IEEE Trans. Nucl. Sci. 39 (1992) 838
- [5] M. Agnello et al. IEEE Trans. Nucl. Sci. 39 (1992) 1270
- [6] E. Botta et al. "Search for <sup>4</sup>He production from Pd/D2 systems in gas phase", Proc. of the 5<sup>th</sup> Int. Conf. on Cold Fusion (ICCF5), Monte Carlo (1995), p. 233.