Low Temperature Gas Loading of Deuterium in Palladium

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Abstract. One of the most established features of the phenomenon known with the name of “Cold Fusion”, with reference to the system palladium (Pd) – deuterium (D), is that a condition necessary (even though not sufficient) to be satisfied in order for these phenomena to take place is that the content of D in Pd, called also the D/Pd ratio X, approaches the value of 1 (understanding by this quantity the atomic ratio between the two species in the Pd lattice). In order to reach such an high value of X, extensive use of electrolysis of heavy water with a Pd cathode has been made.

The present experiment is aimed at obtaining high loading ratios of deuterium in palladium without using electrolysis. The idea is to have deuterium gas in contact with palladium. The use of low temperatures has the purpose of increasing the equilibrium loading ratio for a given gas pressure.

A first test experiment, performed at ENEA Frascati in 2002, showed that it was possible to have D/Pd ratios as high as 1 at 150 K with a pressure lower than 1 bar [1]. The experiment has been rebuilt at LNF/INFN and the first results are reported here.

An anomaly in the loading dynamics will be also reported.

1. Introduction

The idea is to realize a conceptually simple experiment, reproducible, and with straightforward answers:
- To start with, measuring the D/Pd ratio, aiming to high values.
- Possibly detecting excess heat.
- Analyzing the gas, looking for \(^4\)He.
- Studying the loading dynamics.

We refer to Ref. [1] for the description of the apparatus and the report of the first results. Here we will limit ourselves to describe the calorimetric technique and to report preliminary results on the loading dynamics.

2. The calorimeter

In Fig. 1 is reported the general scheme of the apparatus. The cell containing the sample is surrounded by a shield: both are made out of copper, and can be considered isothermal. With the help of a Cryodyne closed-cycle refrigerator [2], both can be kept at a constant temperature, from a minimum of 20 K to almost room temperature. The thermoregulator [3] assures a temperature constant within \(\pm 1\) mK.

The cell is held at a temperature higher than the shield: thus, heat power has to be dissipated in the cell heater in order to keep the assigned temperature. This power is measured and recorded with the help of a LabVIEW data acquisition system. Should an independent heat source become active within the cell, the thermoregulator has to reduce the amount of energy fed to the cell heater. This decrease, with the opposite sign, gives the value of the heat produced by the independent source.
In order to validate this method, tests have been performed by mounting within the cell a dummy heater (an evanohm wire in which to create Joule heating), and the power sent into the dummy heater is compared with that detected by the calorimeter. A typical example is shown in Fig. 2. Note the oscillation of the signal of $W_c$ (the power sent by the thermoregulator into the cell heater), both when the heat starts and when it stops. However, when the oscillation is quenched, the agreement between the calculated power ($W_{\text{calc}}$) and the experimental one ($W_{\text{exp}}$) is quite satisfactory, on the order of 1 %.

Fig. 2 - Validation of the calorimeter.
It is also possible to measure the total heat produced by the independent heater, by integrating the $W_c$ graph versus time.

3. Preliminary tests of loading dynamics

The geometry of the first samples used for this experiment – the same used in [1] - is shown schematically in Fig. 3a.

The real sample is constituted by a Pd film, of thickness 0.5 to 3.5 μm, 80 μm large, and 2.5 m long, deposited on a glass support, 1 mm-thick, 25 x 35 mm² area. Its resistance can vary, as a function of thickness, temperature and loading ratio, from a few hundreds to many thousands ohms.

The geometry is such that the total resistance is divided in 5 sections, all in series. In Fig. 3b is reported the scheme of the circuit used to measure the five resistances $R_i$. The voltmeter in the figure permits measuring the current circulating in the sample, and thus the measurement of the $\Delta V_i$’s, made by the LabVIEW system, gives the resistances. They are measured and recorded as a function of time, at the rate of one value every minute.

Bearing in mind also the suggestion by Giuliano Preparata, the “Preparata Effect” [4], which envisages the possibility that the loading ratio can be influenced by the value of the electric potential on a Pd cathode (in an electrolysis experiment), we have looked at the behavior of the 5 resistances described above during the loading of Deuterium gas. In our case the “electric potential” was that used to measure the resistances. The results are at present quite poor, because of the difficulty of having good Pd samples (“bad” ones tend to detach themselves from the glass support when absorption starts), but nevertheless they are quite suggestive. We will report them here.

The first run we consider is that performed in 2002 [1], at the temperature of 150 K. In order to keep track of the changes of loading ratio, the value of the resistance is considered: it is well known that the resistance grows linearly with increasing $X$ [5]. At room temperature, when $X \approx 0.75$, the resistance is doubled (it increases by a factor 2.6 in the same interval at 150 K).

In Fig. 4 the behavior of the ratio between the value of the resistance while absorbing deuterium ($R$) and the value of the unloaded sample ($R_0$) is shown as a function of time for the various sections of the sample during the first phase of the absorption process. Note that $R_5$ was broken after about one day, and then short-circuited, so that this measurement is reported only for the remaining four sections of the sample.
Table 1. The loading ratio (X) reached by the four sections of the sample.

<table>
<thead>
<tr>
<th>R/R₀</th>
<th>X</th>
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<tbody>
<tr>
<td>R₁</td>
<td>1.18</td>
</tr>
<tr>
<td>R₂</td>
<td>1.20</td>
</tr>
<tr>
<td>R₃</td>
<td>1.42</td>
</tr>
<tr>
<td>R₄</td>
<td>1.75</td>
</tr>
</tbody>
</table>

Fig. 4 - R/R₀ vs t (2002, 150 K)

The behavior of the four resistances almost two days after starting loading is quite dramatic. In Table 1 the final values of the R/R₀ are listed, together with the corresponding values of X = D/Pd. In this run R₄ was the most negative and R₁ the most positive among the four sections. The absolute value of the voltage used was 0.2 V.

Fig. 5a

The different behavior among the sections becomes much smaller when approaching the maximum value of the loading. This is shown in a Fig. 5a, were another run, this time at room temperature, is considered. In
Fig 5b the same results are reported, by emphasizing the effect at the beginning of the loading. Here $R_1$ is the most negative, $R_5$ is the most positive. The absolute value of the voltage used was 8 V.

Thus, it seems that a negative potential favors the absorption, at least at the beginning of the loading process.

The data are scarce, even though suggestive. More tests are needed and will hopefully be performed. The main problem is having good quality samples, in the sense that they do not detach from the support when they absorb deuterium. A collaboration is under way with this purpose with David Knies of the NRL of Washington DC, USA.

Acknowledgements

Since 2006 this experiment was hosted at the LNF of INFN; thanks are due to the Direction of the Laboratory and to Commission 5 of INFN for its support.

The Author is deeply grateful to his colleague and friend Ivo Modena for continuous scientific support in conducting the experiment.

He thanks warmly Arturo Moleti, Alessio Rocchi and Filippo Sanjust, who took care of the LabVIEW data acquisition system.

Thanks are due to Antonio Frattolillo and Antonella De Ninno of ENEA, and to Alberto Clozza and Angelo Viticchié of INFN, for permitting the use of valuable scientific instruments.

The experiment reported here would not have been possible without the skillful help of Franco Campolungo, Mauro Iannarelli, Livio Bettinali and Lorenzo Martinis: also to them goes the Author’s gratitude.

4. References