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Protocol for Controlled and rapid Loading/ Unloading of H_2/D_2 Gas in Self Heated Pd Wires to Trigger Nuclear Events

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Introduction

It has now been established that during electrolysis of LiOD using Pd cathodes a threshold loading ratio of at least 0.85 needs to be achieved before excess heat production can be expected [1]. However for emission of neutrons and tritium and possibly charged particles and transmutation products too much lower D/Pd ratios, in the region of 0.4 to 0.7 appears to be adequate. This has been independently corroborated in a variety of electrolytic and gas loading experiments. It is not so much the magnitudes of the steady state loading ratio, but rather creation of non - equilibrium conditions which facilitates rapid migration/transport of deuterons within the Pd lattice that seems to be required. With this in view a systematic study has been undertaken using electrically self-heated 0.125 mm dia Pd wires in H_2/D_2 atmospheres to optimize the conditions under which rapid loading/unloading of H_2 or D_2 can be achieved. Pd wire was of 99.9% purity and was procured from Lieco Industries, USA.

Procedure for Loading

After a number of trial experiments the following protocol was established for optimum and rapid loading:

1. Measure initial wire resistance, namely R_0^* of the unannealed wire.
2. Heat wire to glow hot condition in air by ohmic heating. R_t/R_0, the ratio of resistance at glow hot condition to resistance at room temperature would be ~3.
3. Lower the current to < 100 mA and measure resistance in atmospheric air after wire cools down. This is the R_0 value to be used for further studies. This may differ slightly from R_0^*.
4. Evacuate system to ~10^{-2}mb and heat wire to red hot condition. Current for red hot condition in vacuum would be much lower.
5. Introduce air under red hot condition of pd wire. This provides a thin oxide layer on the wire surface.
6. Evacuate once again.
7. Introduce H_2 or D_2 as required when wire is red hot. It reacts with oxide layer and activates surface. Measure resistance (R_{max}) after cooling.
8. A loading of approximately 0.6 corresponding to R_{max}/R_0 ~ 1.7 for H_2 or 1.85 for D_2 may be achieved within 2-3 minutes, provided activation of wire surface has taken place. Loading increases further to ~0.7 in another 30 minutes. For rapid loading the wire should be as hot as possible during step No.7. However increasing wire tempera-

ture unduly has the risk of accidentally burning out the wire.

Some typical parameters recorded in our experiments are:

Wire diameter	:0.125 mm
Wire length	:~ 100 mm
Annealed wire resistance R_0	:0.8 ohms
Resistance at red hot condition R_t	:2.5 ohms
Vacuum employed in chamber better than	:10^{-2} mb
H_2/D_2 gas filling pressures	:1-2 bars

Verification of (H(D)/Pd) Loading Ratio

On routine basis (a) Resistance Ratio [1] is used to infer the degree of loading. However to cross check the loading value, (b) Oil manometer technique and (c) Inert gas fusion technique were used in a few cases. All the three techniques were found to give comparable loading values.

Summary of main Results

1. We are able to load up to a H(D)/Pd value of 0.7 both in case of H_2 as well as D_2.
2. We do not find any difference in loading rate whether it is D_2 or H_2.
3. Loading is fast if the gas (H_2 or D_2) is introduced in the system when the wire is brilliantly hot. Because of good conductivity of hydrogen, wire temperature automatically falls below the threshold temperature for absorption in Pd, as soon as gas enters the chamber.
4. Since temperature at which wire gets unloaded is less in D_2 (110°C) than in H_2 (150°C), at a given filling pressure, current for deloading is lower for D_2 than for H_2 [3].
5. Loading rate is not particularly influenced by H_2 or D_2 gas pressure (at least in region of few tens of mb to 4 bars, which we have studied), but it is very sensitive to the pretreatment given to the wire.
6. Rate of unloading of a loaded Pd wire at room temperature in atmospheric air is found to be surprisingly slow .
7. If the wire surface is activated properly loading up to 0.6 or 0.7 can be attained with 100% reproducibility.

Hysteresis Studies

To get the hysteresis curve [2], following procedure is adopted:
1. Load the wire at particular gas pressure (1 to 2 bars) to the maximum extent possible (0.7 in our case).
2. Slowly increase the current in steps. Measure the resistance at each current step after wire temperature stabilizes.
3. At some value of current, resistance suddenly drops.
4. On increasing current further, at a particular current, wire resistance starts going up again. In this state wire is fully unloaded.
5. To obtain hysteresis curve now slowly decrease the current; observe that resistance

starts decreasing; at some value of current resistance begins to increase. This implies that wire has begun to load. The current at which wire starts loading in reverse part of cycle is less than in the forward half cycle.

6. Typical hysteresis curve obtained by us is shown in Fig 1.

7. Effect of temperature on loaded Pd wire at different hydrogen gas pressures is shown in Fig 2.

Procedure for Repeated Cycles of Loading/ Unloading

From the hysteresis curves corresponding to a given pressure select the lower and upper "safe" current values at which absorption and desorption will be rapid and assured. For the case of D_2 gas at 1.8 bars for example the lower and upper current values are 0.9 amps and 1.7 amps for loading and unloading respectively (See Fig.1). A simple and automatic switching device can be designed which will flip the current between these two values by shorting a precalculated resistance placed in series with the Pd wire. Since loading takes a longer time , it is recommended that more time be allowed in absorption mode than in desorption mode.

Applications and Conclusion

Having learnt to absorb/desorb H_2 or D_2 in a 100% reproducible and controlled manner in Pd wire, creating non-equilibrium conditions, following experiments have been carried out/are proposed to be carried out in our centre in search of anomalous nuclear effects:

1 Autoradiography: After loading the wire with H_2 or D_2 it was autoradiographed using medical X - ray photographic film. Every loaded wire is found to fog X - ray film. Typical autoradiographs are shown in Fig 3 & 4. Even a low loading of 0.1 fogs the film. This is the phenomena of PdH_x luminescence first reported by our group in 1991 [2]. Efforts to understand the cause for mechanism of fogging are continuing.

2. Charged particle emission: Solid State Nuclear Track Detectors (SSNTD) and surface barrier detector were employed to search for emission of charged particles if any from PdD_x wires subjected to repeated cycles of loading/unloading of deuterium. So far we have not detected any charged particles from self-heated Pd wires.

3 Tritium and neutron detection experiments are under way.

References

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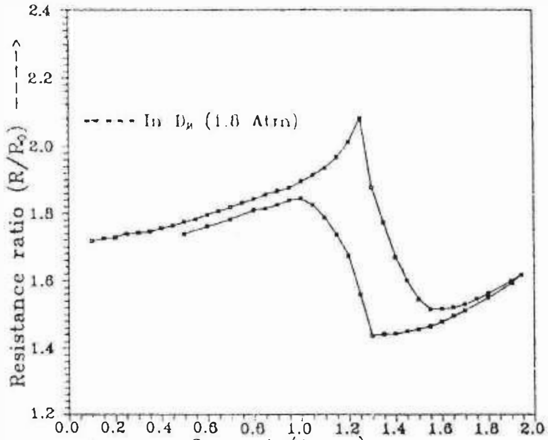

Fig 1. Hysteresis curve for Pd wire

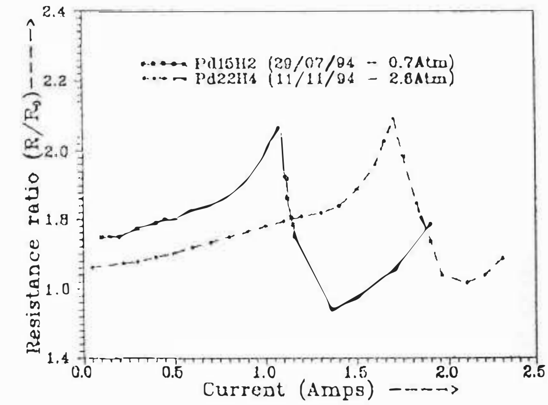

Fig.2 Temperature effect on a loaded Pd wire at different H_2 gas pressures

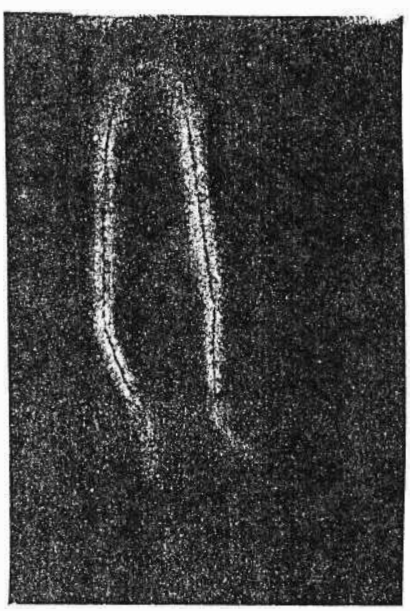

Fig 3. Autoradigraph of Pd hydride wire H/Pd~0.65

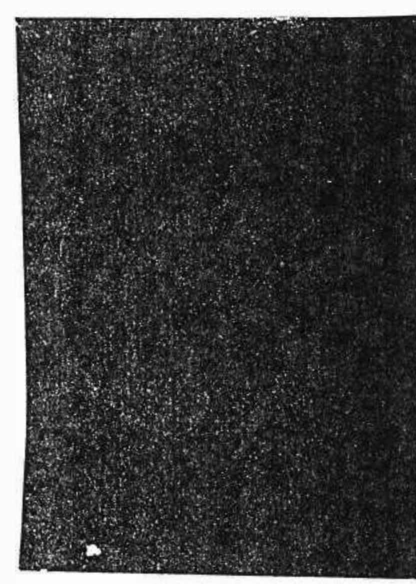

Fig 4. Autoradiograph of Pd hydride wire H/Pd ~0.6