

# Modification of Pd-H<sub>2</sub> and Pd-D<sub>2</sub> thin films processed by He-Ne laser

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## ABSTRACT

In this work we have performed experiments of absorption of hydrogen and deuterium gas by Pd thin films and we have studied the behavior of these samples compared with non processed ones. We have also employed, during the treatment, a continuous wave He-Ne laser to irradiate the samples inside the chamber in order to increase the gas absorption. By a scanning electron microscope (SEM) and an electron probe micro analyser (EDX) we have observed, on the surface of the treated samples, the presence of structures like spots and inside them different elements from Pd were found. By these results we can say that the gas loading has been a very important condition to produce “transmutation” elements and the laser action has been very interesting in order to increase the morphological modifications of the treated samples.

## INTRODUCTION

In March 1989 M. Fleischmann and S. Pons [1] reported that a great excess of enthalpy, recorded with a weak amount of radioactivity, had been detected in electrolytic cells with Palladium (Pd) cathodes during the electrolysis of D<sub>2</sub>O, achieving a critical threshold of the stoichiometric ratio  $x=[D]/[Pd]$  of Deuterium in Pd. The main point at issue was that the claimed excesses of enthalpy were consistent only with a nuclear process (fusion of Deuterons) and that such a process at ambient temperature and without the emission of a adequate number of neutrons was considered to be inconsistent with modern nuclear science. In the following years many works about the production of excess heat and Helium have been published [2, 3], and many devices to improve the stoichiometric ratio  $x$  have been studied [4]. Possible theoretical explanations of the reported phenomena were proposed [5, 6] during these years but we are far to justify completely all obtained results jet.

Recently the gas loading method became a very interesting tool to obtain adequate absorption of D or H gases inside Pd metallic lattice accompanied with the possibility to keep

low the contamination grade during the experiment [7]. Important results were achieved by this method and particular attention was made to reproduce transmutation effects [8].

In this work our attention is voted to study the transmutation phenomenon utilizing Pd film samples treated by gas loading. We have also implanted B in these samples in order to try to have control of the eventually nuclear processes such as Iwamura et al. have done. The treatment of the samples was combined with a continuous wave He-Ne laser light in order to improve the gas loading inside Pd thin films as in previous works was studied [9].

## EXPERIMENTAL SET-UP AND RESULTS

We have realized, by thermal evaporation technique, Pd thin films of 500 nm of thickness deposited on Si wafers of about 1 cm<sup>2</sup> in surface. A 50 nm Ti layer was used to improve the adhesion between the substrate and the Pd layer. These samples were implanted with B ions; a 150 keV accelerating voltage allowed to reach the maximum ion concentration at 158 nm depth in the palladium layer. Fig. 1 shows the distribution of B ions versus target depth.

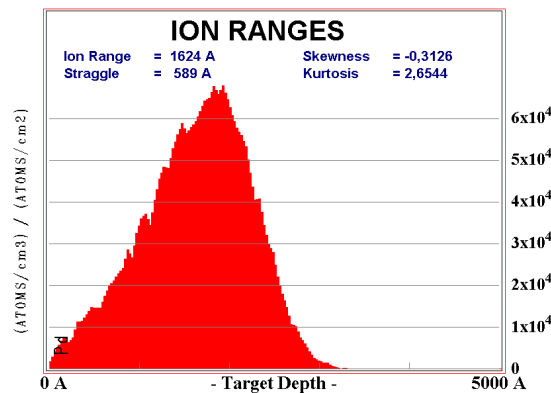


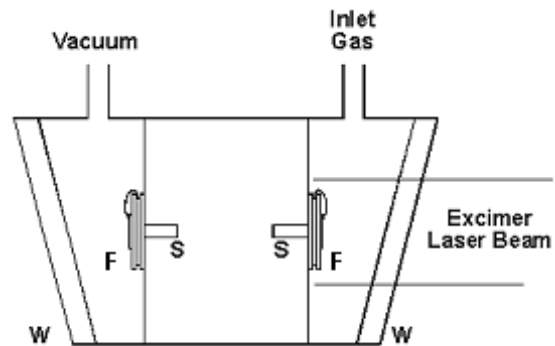
Fig. 1: Boron ions distributions vs. target depth

They were placed in cylindrical stainless steel chambers of about 250 cm<sup>3</sup> in volume. Fig. 2 shows a photo of the experimental set-up.



**Fig. 2:** Photo of experimental set-up

The chambers were equipped at least of a quartz window in order to allow the laser beam to go until the samples. To avoid contaminations, the chambers were carefully cleaned with acetone and dried in nitrogen flux before the experiment beginning. Subsequently a pair of Pd/Si samples has been placed inside the chambers filled with H<sub>2</sub> or D<sub>2</sub> gas to a maximum pressure of 4 bar. In Fig. 3 we have a schematic drawing of a chamber.

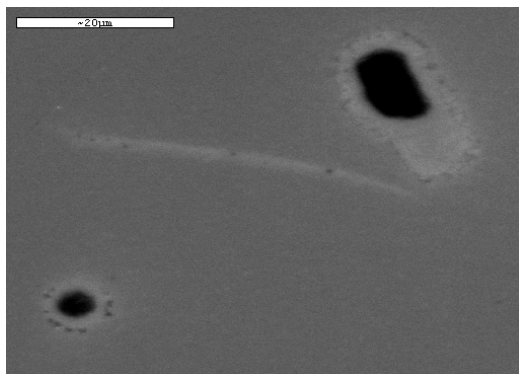


**Fig. 3:** Schematic drawing of a chamber. W: quartz windows; S: supports where the samples are fixed; F: film samples

The treatment of the samples was employed with gas loading and only one sample for each chamber was irradiated by a cw He-Ne laser ( $\lambda=648$  nm) from July 16th to September 29th 2004 at a laser power density of about 2 mW/cm<sup>2</sup>.

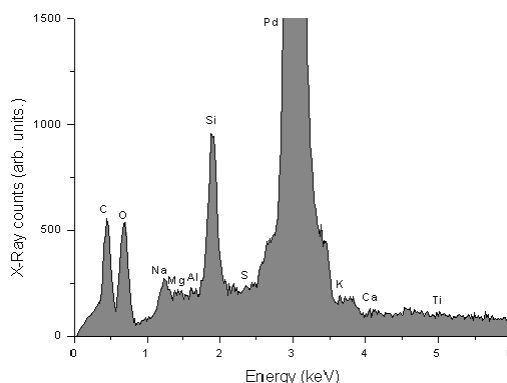
After the treatment ending, the samples were analysed by a Scanning Electron Microscope (SEM) and an EDX micro-analyser. Different behaviors were revealed for samples kept in air, laser treated and no-laser treated: so, about the samples kept in air, the

film surface was smooth, it looked like a mirror; instead, the samples treated and no-treated by laser showed morphological modifications of the Pd-film due to the gas absorption. The morphological modifications consisted in formation of spots with dimension of 1-50  $\mu\text{m}$  after gas loading. Fig. 4 shows an example of spots on the surface of a sample of palladium implanted with boron, loaded by  $\text{D}_2$  gas and not irradiated.



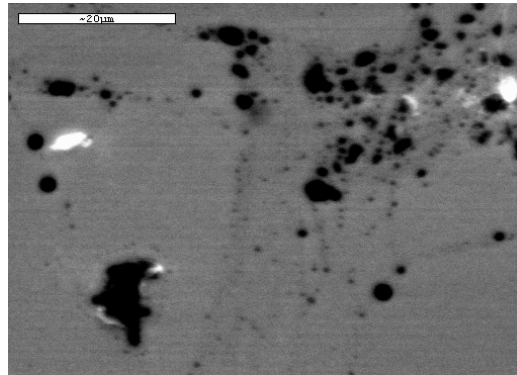
**Fig. 4:** Spots on the surface of a sample with 76 days of treatment (by  $\text{D}_2$  gas only)

By EDX analyser, we have investigated inside the spots and we have found the presence of new elements such as C, O, Ca, Fe, Al, S, Mg, K and Na. In Fig. 5 an example of EDX spectrum of a Pd sample with 76 days of treatment is reported. It is possible to observe the presence of many “new” elements which were inexistent before the treatment.

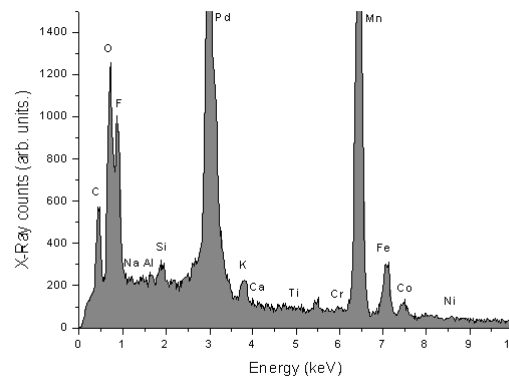


**Fig. 5:** EDX spectrum of a sample with 76 days of treatment (by  $\text{D}_2$  gas only). We can observe the presence of the following elements: C, O, Ca, Al, S, Mg, K, Na

In addition, by He-Ne laser action, we have found a larger number of spots and a larger number of new elements. Fig. 6 shows a SEM micrograph of a sample processed by  $\text{H}_2$  gas and laser; Fig. 7 shows EDX spectrum obtained from one spots of the sample: the new elements were: C, O, Ca, Fe, Al, S, Mg, K, Na, F, Cr, Mn, Fe, Co, Ni.



**Fig. 6:** Spots on the surface of a sample with 76 days of treatment (by H<sub>2</sub> gas and by He-Ne laser action)



**Fig. 7:** EDX spectrum of a sample with 76 days of treatment (by H<sub>2</sub> gas and He-Ne laser). We can observe the presence of the following elements: C, O, Ca, Fe, Al, S, Mg, K, Na, F, Cr, Mn, Co, Ni

In Tab.1 the list of the new elements is reported for every experimental case of the sample treatment. We can observe that the combination between H<sub>2</sub> gas loading and laser action on the treatment of the samples is very interesting in order to produce many transmutation elements; nevertheless the results with D<sub>2</sub> gas loading are also not negligible about the production of new elements, but there are no evident differences between laser and no laser treated samples. The laser action is also very important to increase the spot density on the surface of the treated samples. All new elements were found inside the spots systematically but none of these seems to be generated from a particular nuclear reaction between B and D<sub>2</sub> and H<sub>2</sub>. These experiments confirm the reproducibility of the transmutation phenomenon but we are still far to make clarifications about the mechanisms which happened inside the crystalline lattice of Pd samples.

<b>H<sub>2</sub></b>		<b>D<sub>2</sub></b>	
<b>laser</b>	<b>No-laser</b>	<b>laser</b>	<b>No-laser</b>
Si	Si	Si	Si
Pd	Pd	Pd	Pd
Ti	Ti	Ti	Ti
C		C	C
O		O	O
Ca		Ca	Ca
K		K	K
Na		Na	Na
Al		Al	Al
Cr		Mg	Mg
Fe			S
Co			
Ni			
Mn			
S			
F			

**Fig. 8:** The main detected elements in every experimental case

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