# Material transformations into Pd-H<sub>2</sub> and Pd-D<sub>2</sub> systems induced by laser irradiation

#### U.Mastromatteo

(ubaldo.mastromatteo@libero.it)

A.R.G.A.L. association, via S. Stefano, 27 - 20010 Bareggio (Mi), Italy

In order to explore anomalous transformation in  $Pd-H_2$  and  $Pd-D_2$  systems as claimed by several researchers, samples of Pd thin films were submitted to laser irradiation inside a stainless steel chamber having a suitable quartz window and filled with  $H_2$  or  $D_2$  at a pressure of few bar. The irradiation treatment was done with a continuous wave (CW) He-Ne laser having a beam power of 2 mW sent through an optical system to widen the light beam to uniformly illuminate the 1 cm<sup>2</sup> sample. A scanning electron microscope (SEM) with an electron probe micro analyzer (EDX) were used to analyze the surface of the treated samples, in particular on areas of the film where morphological modifications occurred. In those areas many elements not present in the reference samples not exposed to Hydrogen/Deuterium and laser irradiation were detected.

The samples were implanted with Boron in order to enhance possible transformation processes the way other researchers have done [1], [2].

The treatment with the He–Ne laser beam was applied because supposed to enhance gas loading inside Pd thin films, as in previous studies.

#### Experimental set up and results

We have realized, by thermal evaporation technique, Pd thin films of 500 nm of thickness deposited on silicon wafers then diced in samples of about 1 cm<sup>2</sup> size. 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 at 150 keV accelerating voltage with the maximum ion concentration at 158 nm depth in the palladium layer. Fig. 1 shows the distribution of B ions versus target depth. The samples were placed in cylindrical stainless steel chambers of about 250 cm<sup>3</sup> in volume.



Fig. 1- Boron ions distributions vs. target depth

The chambers were equipped with at least one quartz window to allow the laser beam to irradiate the samples. To avoid contamination, the chambers were carefully cleaned with acetone and dried in nitrogen flux before the experiment. Subsequently a pair of samples has been placed inside the chambers filled with  $H_2$  or  $D_2$  gas to a maximum pressure of 4 bar. In Fig. 3, we have a schematic drawing of the set-up.





*Fig. 2 - Schematic drawing of the experimental set-up. D2: Deuterium gas at 4 bar pressure; T: Target sample; W: Quartz window; L:Convergent lens; on the right a picture of the real chamber during a beam alignment test.* 

The treatment of a couple of samples consisted in spontaneous gas loading and in laser beam irradiation by the CW He-Ne laser ( $\lambda$ =648 nm) having a power density of about 2 mW/cm<sup>2</sup> for the sample faced to the optical window; any test lasted about 70 days.

After the treatment ending, the samples were analyzed by a Scanning Electron Microscope (SEM) equipped with an EDX micro-analyzer. Different behaviors were revealed for samples kept in air, for samples exposed to the laser and for the ones with only gas loading without laser irradiation. With the samples kept in air, the film surface was smooth, with a mirror-finish and the EDX spectrum detected only Pd, Si and Ti; the samples placed in deuterium gas but not treated with lasers showed morphological modifications of the Pd film; the samples placed in hydrogen gas without laser irradiation were similar to those kept in air; those treated with lasers both in hydrogen and in deuterium showed higher density of morphological modifications.

The modifications consisted in the formation of spots with dimension of  $1-50 \ \mu m$  where the palladium film melted an solidified into irregular shapes.

Figure 3 here below, shows an example of spots on the surface of a sample of palladium implanted with boron, loaded by D<sub>2</sub> gas, but not laser irradiated. By EDX analyzer, we investigated inside the spots and found the presence of unexpected elements such as C, O, Ca, Fe, Al, S, Mg, K and Na.



Fig. 3 - On the left, spots on the surface of a sample with 76 days in  $D_2$  gas without laser irradiation; on the right, the EDX spectrum on one of the spots

It is relevant to notice that the spots morphology clearly highlights the Pd melting and random solidification as for the occurrence of a very energetic explosion. On this purpose the Figure 4 shows a SEM micrograph of a sample processed by H<sub>2</sub> gas and laser and also the EDX spectrum obtained from one spots of the sample: the found extra elements were: C, O, Ca, Fe, Al, S, Mg, K, Na, F, Cr, Mn, Fe, Co, and Ni.



Fig. 4: Spots on the surface of a sample with 76 days of treatment (by  $H_2$  gas and by He-Ne laser action) and EDX spectrum in the spots. We can observe the presence of the following elements: C, O, Ca, Fe, Al, S, Mg, K, Na, F, Cr, Mn, Co, Ni

Table 1 shows a summary of the new elements detected in various experiments. We observe that the combination between H2 gas loading and laser action on the samples is an effective method of producing many "transmutation" elements. The results with D2 gas loading alone, without laser irradiation, also produce new elements, and in this case there are no evident differences between the number and type of elements produced with and without the laser. It seems that the laser action has mainly the effect of increasing the spots density on the surface of the treated samples, but it does not change the nature of the reaction. All the "extra" elements were found inside the spots. None of them seems to be generated from a particular nuclear reaction between B and D2 and H2. These experiments confirm the reproducibility of the transmutations as seen in similar experiments, although the cause of this has not yet been clarified.

H <sub>2</sub>		<b>D</b> <sub>2</sub>	
laser	No-laser	laser	No-laser
Si	Si	Si	Si
Pd	Pd	Pd	Pd
Ti	Ti	Ti	Ti
С		С	С
0		0	0
Ca		Ca	Ca
K		K	K
Na		Na	Na
Al		Al	Al
Cr		Mg	Mg
Fe			S
Со			
Ni			
Mn			
S			
F			

Table 1: The main detected elements in every experimental case

### Material transformation anomalies replicated in a different lab.

The experiments described above were carried out at the applied electronics laboratory of the University of Lecce (2004). Those we are going to mention below were done in the A.R.G.A.L. laboratory in Bareggio (MI) (2015).

### **Test description**

Samples very similar of those described so far were placed inside a reactor filled with hydrogen at 1.5 bar for 2 weeks and irradiated through a glass viewport with a low-power He-Ne laser (633 nm, 0.9 mW) at ambient temperature. An optical system was used to enlarge the laser spot up to  $1 \text{ cm}^2$  size.

The palladium surface was then accurately explored with a SEM to find possible traces of morphological changes: several cavities were found, looking like similar findings of previous experiments.

In contrast to the experiments conducted at the University of Lecce in 2004, in these experiments we did not observe hot spots with evident traces of melting of the material, but a certain number of cavities with dimensions around tens of micrometers, inside of which the EDX analysis showed the presence of elements not present in the thin palladium layer as deposited. In reference to this, it is important to consider that even this time the samples were manufactured inside a very clean environment of a microelectronics facility (STMicroelectronics), with maximum care to avoid contamination issues. It is important to underline that in these new experiments the sample were non implanted with Boron.

Below are pictures and spectra of some EDX analysis of cavities.

## Irradiated palladium film cavities EDX analysis



Figure 5. EDX inside the cavity, spectrum element list: C, O, Na, Mg, Al, Si, K, Fe, Co, Cu



Figure 6. EDX inside the cavity, spectrum element list: C, O, Na, Mg, Al, Si, K

### **Conclusions and highlights**

- It is worth to emphasize that the type of experiments described above are relatively easy to replicate.
- In spite of in the ARGAL lab there is continuous monitoring for neutrons and gamma emission, our instruments never registered deviations from the background spectrum, which is the same result we saw with previous experiments done at Lecce University with the Pd/H<sub>2</sub> system.
- Because almost all the experimental evidence reported, new and old, points to transmutation of the reacting material to lighter elements, a deeper investigation of possible fission reactions of the material submitted to the described tests is suggested.
- The matter transformations observed are quite similar to those found by other experimenters, even when different combinations of elements and other types of stress were used. We think it is also worth pointing out that the evidence of the 2004 experiments suggests the importance of certain "impurities" present in the material: Boron in this case, as demonstrated also in recent experiments with electrolytic cells having a Boron doped palladium cathode.
- Finally, considering that in the experiments shown, the melting of material indicates highly concentrated energy bursts, does not seem to be far from the prospect of finding a particular experimental system that can lead to the generation of clean energy, highly concentrated and using materials behaving like Pd, but abundant in nature and therefore low cost.

### References

 V. Nassisi et al., Modification of Pd-H<sub>2</sub> and Pd-D<sub>2</sub> Thin Films Processed by He-Ne Laser; J. Condensed Matter Nucl. Sci. Vol. 5, pp. 1–6, 2011.
V. Nassisi, Fusion Technol. 33, 468 (1998).