

Research Article

LENR Anomalies in Pd–H₂ Systems Submitted to Laser Stimulation

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Abstract

In a continuation of the research started in the late 1990s at the University of Lecce, and then repeated in 2004, recently in the second half of 2014 in the A.R.G.A.L. lab., it was decided to thoroughly investigate the LENR anomalies of Pd–H₂ system (in gaseous environment), having as reference the preparation techniques and surface analysis used in the microelectronics industry, to demonstrate convincingly that material contamination was not affecting the transmutation findings. As previously, the activation of the anomalies occurred during irradiation by low power lasers (633 nm, 1 mW, or 403 nm, 3 mW) on 250 nm thick palladium layers deposited by high vacuum e-beam evaporation on silicon oxide substrates, and with 30 nm of Cr as adhesion layer. In summary we did the following: in a stainless steel chamber filled with hydrogen, through a glass viewport, the sample surface was irradiated for two weeks and then accurately inspected in a SEM equipped with an EDX microprobe. A reference sample was also accurately analyzed before irradiation.

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1. Introduction

In order to better understand the results of the present experiments on the interactions between transition metals and hydrogen, assisted by with laser light excitation, it is a good idea to examine some data from the scientific literature that have strong similarities with said type of interactions.

The first reference is [1]. This paper describes the behavior of a pure nickel rod in a hydrogen environment heated from room temperature to over 400°C, and the subsequent examination under the electron microscope and contextual EDX analysis of the surface of said sample, after this has been shown to react with the generation of excess heat in a quantity not attributable to reactions of the chemical type. For our purposes, what is interesting is the EDX result shown in Fig. 1.

A second reference comes from a private communication to me by Dr. Luca Gamberale, referring to experiments he conducted at the Pirelli advanced research laboratories in the late 1990s. In this communication there are a couple of findings concerning transmutation useful for a deeper comprehension of these peculiar aspects of LENR anomalies. The first one comes from the analysis of a palladium cathode observed by electronic microscope and analyzed by

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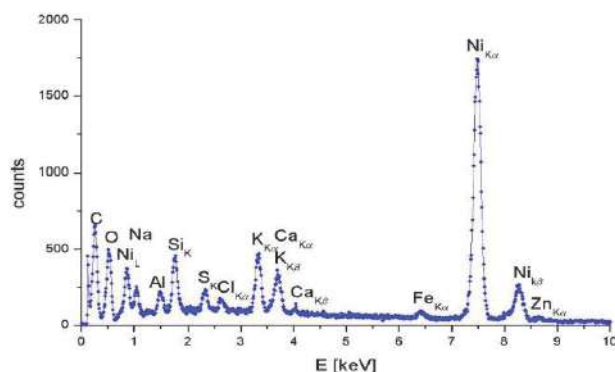


Figure 1. The list of elements from the EDX excluding nickel include: C, O, Na, Al, Si, S, Cl, K, Ca, Fe, and Zn.

EDX micro probe after an experiment of heavy water electrolysis that had shown excess heat: Fig. 2 shows the EDX spectrum.

A second interesting transmutation finding from Gamberale's experiments concerns a copper electrode submitted to glow discharge in a hydrogen environment. The EDX analysis of the electrode after the experiment showed a spectrum with many elements extraneous to the starting material, all lighter than copper (Fig. 3).

Another background reference about transmutation is [2]. In this paper the material submitted to ultrasound stimulation is stainless steel. The shape of the sample was a small rod on which, after one hour of ultrasound stimulation (16 W), several spots appeared on the surface. The results of an EDX analysis of the spots are reported in Fig. 4, Table 1.

Another reference with interesting findings on transmutation concerns experiments on constantan wires is [3]. Several experiments highlighted small excess heat effects and, even though the post experiment SEM analysis was not easy, several spots were detected on the wire surface where an EDX analysis was possible: Fig. 5 shows a typical spectrum.

An interesting quite recent paper about a palladium/deuterium system in an electrochemical cell describes the presence of anomalous contaminations on the cathode surface, the origin of which was not clearly identified [4].

The circumstances of the findings described in the paper led us to think that there is some analogy with the mentioned experiments showing transmutation of palladium to lighter elements. Some common circumstances are as

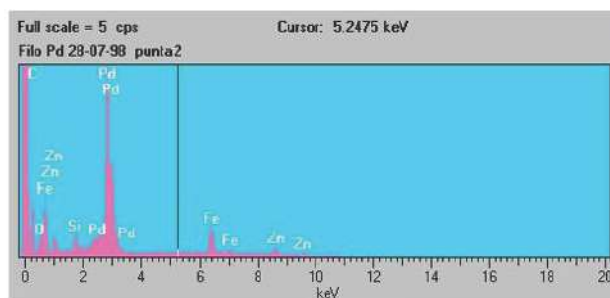


Figure 2. Excluding palladium the new elements are: C, O, Si, Fe, and Zn.

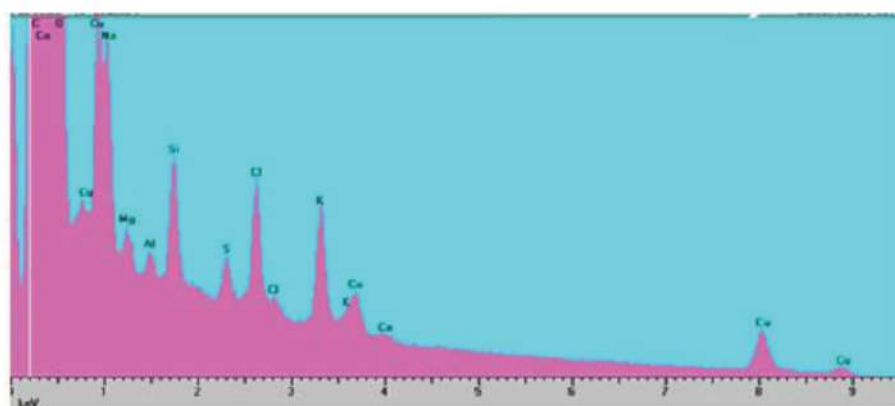


Figure 3. EDX after glow discharge experiment, elements in the spectrum: C, O, Na, Mg, Al, Si, S, Cl, K, and Ca.

Table 1. The results of an EDX analysis.

Element		Weight
C	Carbon	19.80
O	Oxygen	29.27
Na	Sodium	1.20
Mg	Magnesium	0.19
Al	Aluminium	0.53
Si	Silicon	0.49
S	Sulfur	0.27
Cl	Chlorine	1.61
K	Potassium	0.54
Ca	Calcium	0.68
Mn	Manganese	0.47

follows:

- RF was detected at the cathode during the test,
- anomalous heat was produced,
- then, EDX on three suspected contaminated areas of the palladium cathode after electrolysis highlighted the following elements: C, O, F, Mg, Al, Si, Ca, Cr, Fe, and Ni.

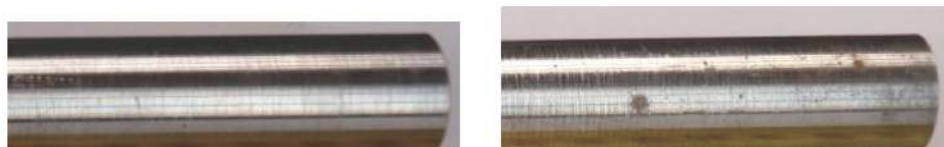


Figure 4. Pictures of the rod before and after the treatment. Table 1 shows in non-bold characters the new elements detected in the spots and the increase of C and O, plus a decrease of the amount of iron.

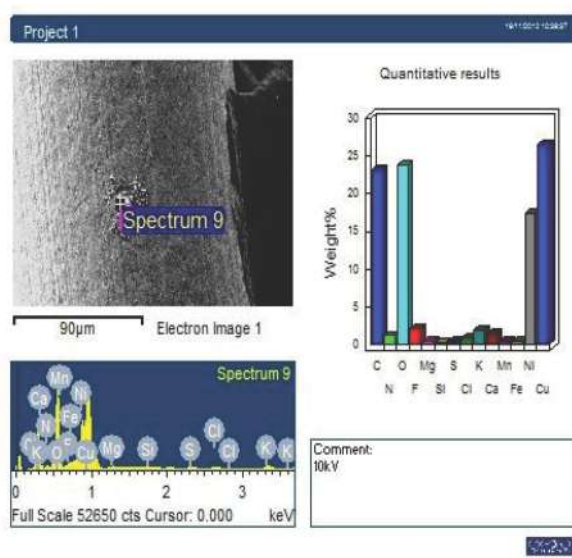


Figure 5. EDX on one of several hot spots detected on the constantan wire. Elements not in the wire composition are: C, N, O, F, Mg, S, K, Cl, and Ca .

The most important background reference about transmutation findings related to our experiments on palladium/hydrogen systems and laser stimulation is Ref. [5].

The experiments reported in this paper, done in 2004 at the University of Lecce, show the effects of laser stimulation of palladium thin layers in hydrogen gas. After several weeks of low-energy irradiation, several hot spots were observed on the surface of the palladium. Careful observation of the spots clearly showed the local melting of the material and

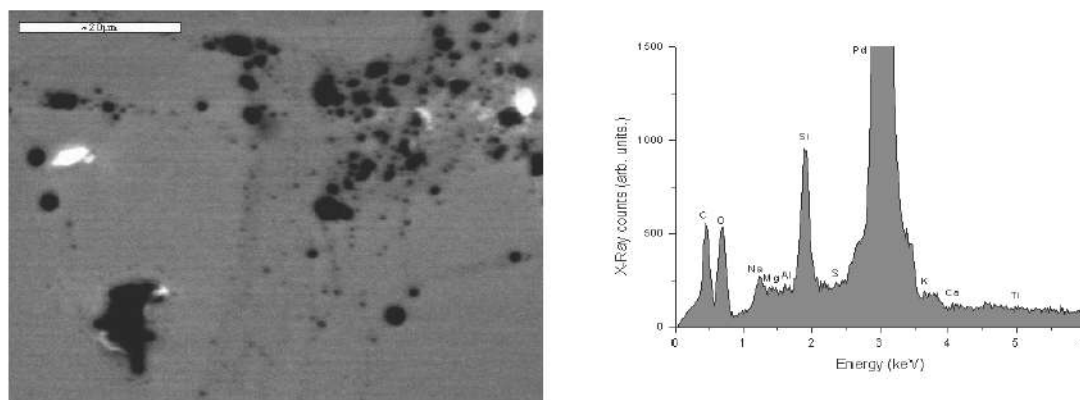


Figure 6. EDX on hot spots after laser stimulation of Pd thin film in H₂ gas. The list of the elements excluding Pd, Si, and Ti (present in the virgin sample) is the following: C, O, Na, Mg, Al, S, K, and Ca.



Figure 7. Photos of the reactor and the bottom part where the sample with the palladium film will be placed.

the EDX analysis highlighted the presence of elements foreign to the composition of the material before the experiment, or the material outside the hot spots.

In the above-mentioned paper, we also reported on experiments with identical palladium thin film samples in deuterium gas; in this case also the reference sample that not irradiated, placed together the irradiated one in the reaction chamber, was affected by hot spots on which EDX analysis highlighted possible transmutation elements. Figure 6 shows a typical picture of a hot spot and the EDX spectrum.

2. New Experiments on Palladium Thin Films and Laser Stimulation

The purpose of the experiments described in what follows was mainly to confirm the activation of LENR “anomalies” in the interaction between palladium and hydrogen (or deuterium) in the presence of coherent electromagnetic radiation (laser or other), in order to understand the detailed mechanism that activates this phenomenon. The choice of this type of embodiment is suggested by the simplicity of the experimental conditions and by the ease of results analysis on small size samples and therefore suitable to a careful examination by SEM microscopy.

Moreover, operating at ambient temperature and without energy input from the outside in addition to that extremely low powered laser, allows easy portability of the entire experimental setup.

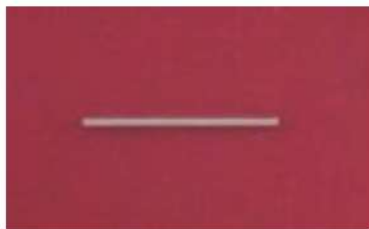


Figure 8. Photo of the sample.

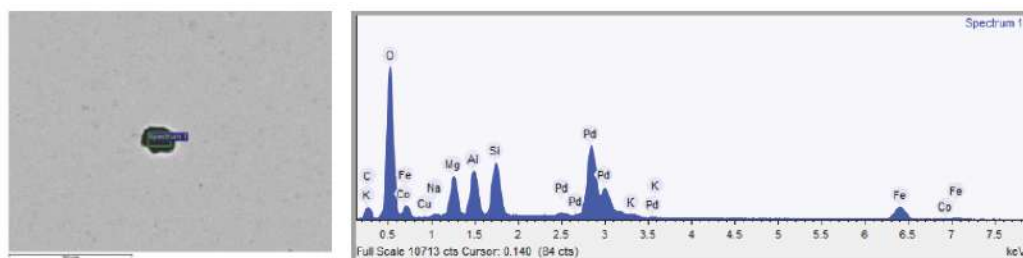


Figure 9. EDX inside the cavity, spectrum element list: C, O, Na, Mg, Al, Si, K, Fe, Co, and Cu.

3. He–Ne Laser Stimulation Experiments

Figure 7 shows the reactor. The sample was an oxidized silicon chip on which it was deposited a layer of palladium 250 nm thick. The size of the chip is: width 1 mm, length 28 mm. See Fig. 8.

4. Test Description

The sample shown in Fig. 8 was 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 cm² size. The palladium surface was then accurately explored with a SEM to

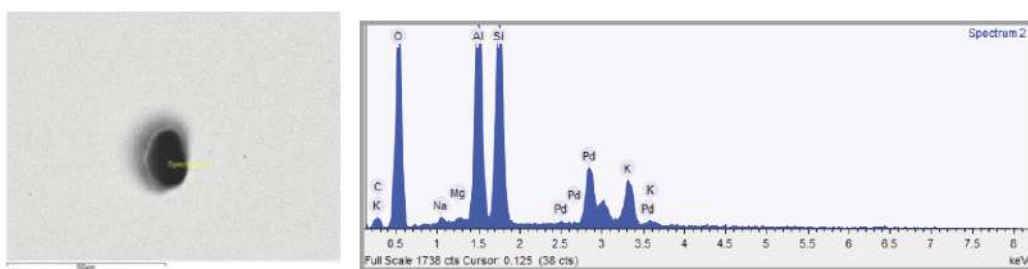


Figure 10. EDX inside the cavity, spectrum element list: C, O, Na, Mg, Al, Si, and K .

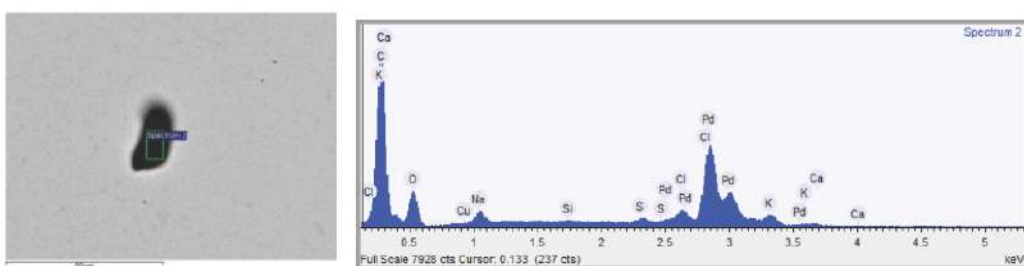


Figure 11. EDX inside the cavity, spectrum element list: C, O, Na, Si, S, Cl, K, Ca, and Cu.

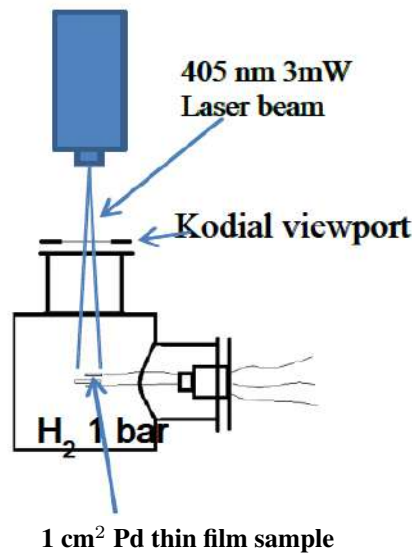


Figure 12. Sketch of the experimental set up.

find possible traces of morphological changes: several cavities were found, looking like similar findings of previous experiments. Images and EDX analysis follow.

5. Irradiated Palladium Film Cavities EDX Analysis

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 the samples were manufactured inside a very clean environment of a microelectronics facility (STMicroelectronics), with maximum care to avoid contamination issues.

Figures 9–11 are the pictures and the spectra of some EDX analysis of cavities.

Cavity pictures and EDX analysis spectra

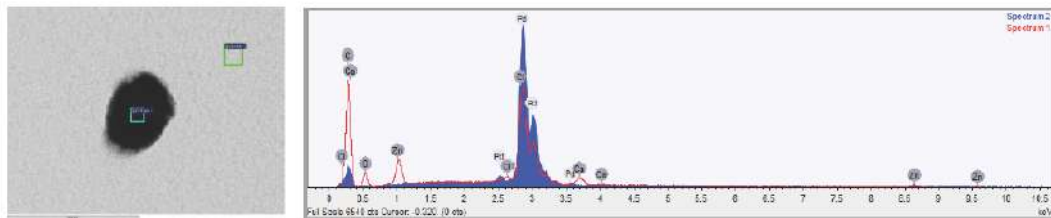


Figure 13. EDX spectrum element list: C, O, Cl, Ca, and Zn (blue spectrum outside the cavity).

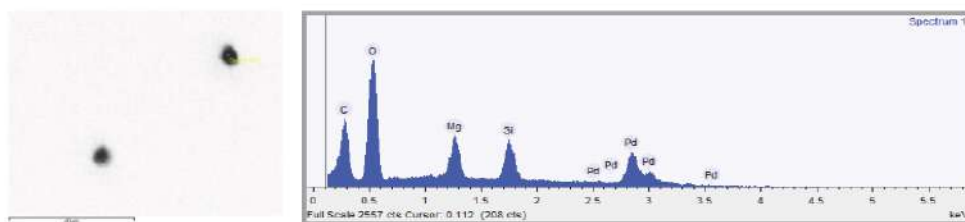


Figure 14. EDX spectrum element list: C, O, Mg, and Si.

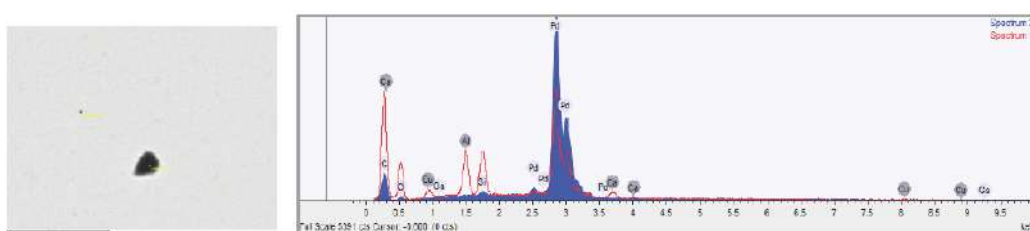


Figure 15. EDX spectrum element list: C, O, Al, Si, Ca, and Cu (blue spectrum outside the cavity) .

6. 405 nm Wavelength Laser Experiments

Another experiment was conducted on a square silicon chip (1 cm^2) covered with the same thin palladium film (250 nm) as the previous tests, but now irradiated with a solid state laser, 405 nm, 3 mW, widening the spot of the beam to about 1 cm^2 . After two weeks of irradiation, the sample was extracted and examined by SEM and EDX analysis using a Hitachi TM 3030 system. Also in this case, carefully exploring the palladium surface revealed several cavities on which EDX analysis was conducted.

With the reactor used in these experiments, it is possible to heat the sample up to 450°C . In spite of that, the experiments were conducted at ambient temperature, the same as the previous ones. Some heating was used, but only to out-gas the sample before the test started in order to best clean at the reactor inside.

Another feature of this reactor, due to the presence of several electrical feedthroughs, allowed us to measure the

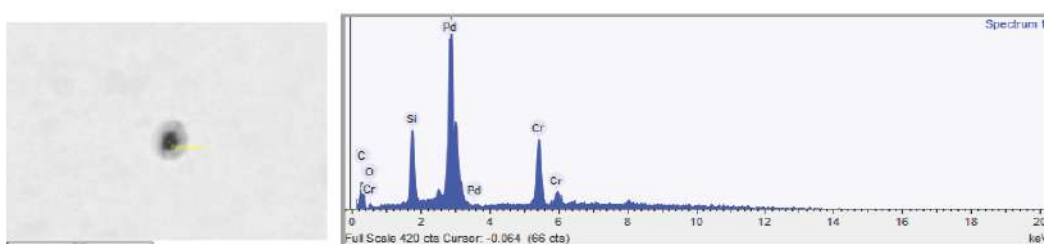


Figure 16. EDX spectrum of a cavity on the sample before the experiments shows elements in the layer as deposited.

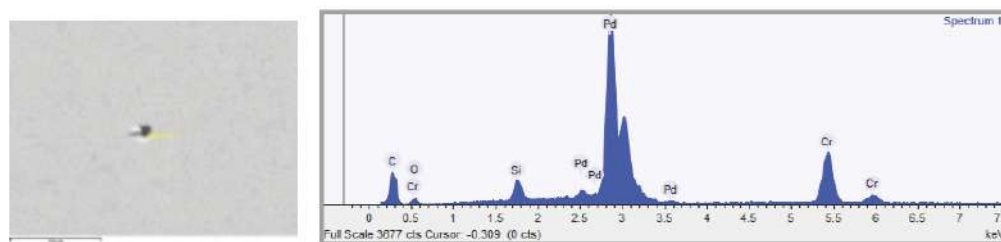


Figure 17. EDX spectrum of a cavity on the sample before the experiments shows elements in the layer as deposited.

electrical resistance of the layer during hydrogen filling. It was observed that the absorption of the hydrogen in the thin film is extremely rapid and reaches equilibrium before the pressure is close to one bar. So, when the experiment starts the environment is at thermodynamic equilibrium.

Cavity pictures and EDX analysis spectra are shown in Figs. 13–15.

7. Reference Sample Analysis

In order to verify that the cavities found on the irradiated palladium surface of the tested samples were due to some kind of new nuclear reactions (LENR) in a laser stimulated H–Pd interaction, the surface of an untested sample has been very accurately explored using the SEM system (Hitachi TM 3030).

What was revealed by the analysis is the almost complete absence of holes, apart from some very small palladium layer imperfections which likely occurred during the layer e-beam evaporation (Pd and Cr adhesion layer on oxidized Si).

Imperfections of the palladium film on the reference sample analysis are shown in Figs. 16–18. These are the related pictures and EDX spectra.

8. Final Remarks

- The experiments shown confirm previous findings, although to a lesser extent (because the duration of the tests was two weeks instead of 10 weeks).
- The 405 nm solid state laser appears to be less effective than the 633 nm He–Ne one.
- In spite of in our continuous monitoring for neutrons and gamma emission, our instruments never registered deviations from the background spectrum, which is the same negative result we saw with previous experiments

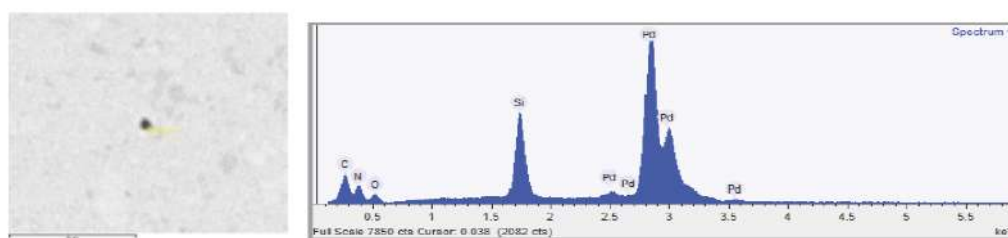


Figure 18. Figure 18. EDX spectrum of a cavity on the sample before the experiments shows elements in the layer as deposited.

done at Lecce University with the Pd/H₂ system. We did observe some neutron emission from the Pd/D₂ system. Moreover, as described above in the introduction, neutron bursts were also detected in the experiments with stainless steel rod and ultrasound [2]. This suggests that different evidences of nuclear activity are strictly connected with the materials used in the experimental set up.

- An accurate analysis of samples not irradiated did not show the presence of cavities with the elements found in the irradiated samples.
- All the elements attributable to transmutation are lighter than palladium.

9. Conclusions

- The experiments cited as background indicate the presence of transmutations similar to those shown in these replication experiments described above. Often the transmutation findings were associated with other LENR anomalies, such as emission of neutrons and excess heat generation.
- Induced oscillations in the material, by ultrasound, radiofrequency, or laser radiation, sometimes appears essential for the occurrence of the anomalies.
- Since some elements attributable to transmutations can easily come from environmental contamination (Na, Si, Al, Mg, Ca), we plan to replicate controls on the samples with accurate SEM analysis before the laser treatment in the reactor.
- Finally, 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 strongly suggested.

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