

# ON NEW ELEMENTS ON CATHODE SURFACE AFTER HYDROGEN ISOTOPES ABSORPTION

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## Abstract

Several experiments of loading Hydrogen and Deuterium into different metals foils and thin film metal layers by using them as cathodes in prolonged electrolysis experiments were performed. Before and after experiments the cathode surface was analyzed by the Scanning Electron Microscopy technique. Results reveal that different elements that were not present in the surface layer before the experiment could be identified in the system after the experiment, with an accuracy considerable above the experimental errors.

## 1. Introduction

During the last years experimental work has been done in many laboratories to verify and to reproduce the Fleischman - Ponce type experiments [1]. Besides the effort was focused on excess energy detection, systematic work was carried on to thoroughly analyze element concentration changes during the experiment as well. Many papers reveal significant isotope concentration changes during H or D loading experiments in different metals involving different techniques. Among them, [2 - 15] are briefly referenced in this paper.

An electrolyses type experiment was designed and performed to search for element concentration changes in the external layers of H or D loaded metals. The experimental setup, operating conditions and results are presented in [16].

## 2. Experiments with Ni cathode

For the first set of experiments the cathode consisted of a Nickel slab 99.9% purity, having dimensions of 80 x 30 mm (in electrolyte. thickness was 0.0127 mm. The anode was a Pt wire, purity 99.997 %. The electrolyte consisted of  $\text{Li}_2\text{SO}_4$ , 99.98 % purity in  $\text{H}_2\text{O}$ , 1M. The cathode electric current density was approximately  $1.225 \text{ mA/cm}^2$  for 66.25 hours and after that the current was adjusted at  $5.678 \text{ mA/cm}^2$  for another period of 64.5 hours.

Scanning Electron Microscopy analysis, with an accelerating voltage of 20.00 KV and therefore an energy resolution of 61 eV, was employed on two different locations on the Ni foil before and after electrolyses to identify the elements on and under the surface. The same parameters were used for the analysis of all the samples described in this paper. Table I presents the results of the SEM analysis. The composition of the two locations

before the experiment does not present any significant difference, therefore only the results of the analysis on location 1 are presented.

Examining these results we notice that traces of Phosphorous and a small amount of Copper were present in the sample before the experiment. The Phosphorous concentration increased significantly above three standard deviations and the Copper concentration increased to 7% on location 1 and to 8.7 % on location 2 during the electrolysis. Special care was taken to prepare the electrolyte. During the entire time span of the experiment preparation, while the cell was open, none of the parts that were put inside were in contact with bare hands or any possible contaminating tool. The electric contacts of the electrodes with the Teflon insulated conductors entering the cell were carefully insulated with Silicon rubber, to prevent electrolyte contact with them and therefore contamination with different metals. This experiment was repeated with another cathode taken from the same sample, in the same electrolysis cell, but was conducted with a smaller cathode current density, that is 0.48 mA/cm<sup>2</sup> and lasted for 142.4 hours. SEM analysis was performed before and after the experiment. No trace of Phosphorous or Copper was found on the Nickel foil cathode this time.

Sample	Element	Sigma, %	Atomic concentration, %
Location 1, before Electrolysis	P	0.05	0.00, < 2 Sigma
	Ni	0.23	99.98
	Cu	0.23	0.11, < 2 Sigma
Location 1, after electrolysis	P	0.05	0.30
	Ni	0.28	92.71
	Cu	0.27	7.00
Location 2, after electrolysis	P	0.05	0.35
	Ni	0.29	90.93
	Cu	0.29	8.73

Table I – Results of the SEM analysis on Ni foil cathode.

A few things are worth noting from these two experiments. First, Copper was not dissolved in the electrolyte from the possibly imperfect insulation of the electric contacts or from any other possible contamination source. If this were the Copper source, it appeared after both experiments, but only the first one produced it. The second conclusion is that Phosphorous is not the result of any contamination either. Exactly the same cell was used for both experiments and if P were the result of a contamination process, it was found in both experiments but it was found only after the first one.

SEM images of the cathodes before and after experiments were recorded. The surface of the cathode employed in the second experiment, looks very much like before the experiment, while the surface of the first cathode looks embrittled, as a result of the mechanical stress produced by the high loading ratio achieved in the outer layers. This suggests that the second cathode did not absorb Hydrogen while the first one did, possibly because the cathode current density was not big enough to prevent recombination on the cathode surface, thus preventing loading [17], while the first cathode absorbed Hydrogen till the exterior layers got saturated.

### 3. Experiments with multilayer cathodes

Another set of electrolysis experiments was completed using the same experimental setup. The cathode consisted of quartz slab, having the surface very well cleaned. Over

both sides a thin titanium layer, 1200 Å thick and over it a thin layer of Pd were deposited by cold sputtering. Both targets used for sputtering had a purity above 99.97%. The error in measuring the thickness is less than 30%. The anode was a Pt wire, purity 99.997 %.

The electrolyte consisted of 1M  $\text{Li}_2\text{SO}_4$  in  $\text{D}_2\text{O}$ . The current density was 4.8  $\text{mA}/\text{cm}^2$ . This experiment lasted for 80 hours. SEM images of the cathode after electrolysis reveal that the surface presents a grainy aspect. This cathode absorbed Hydrogen in a good amount. The mechanical stress produced by H absorption and by the different expansion of the two metals caused by D absorption produced micro-cracks on the surface. The micro-cracks and the grainy aspect of the cathode surface confirm that the multilayer cathode absorbed Deuterium in big amount.

The results of SEM quantitative analysis performed on two different locations on the cathode surface after the experiment are presented in Table II.

Sample	Element	Sigma, %	Atomic concentration, %
Location 1	C	2.65	22.96
	Si	0.96	37.82
	Ti	0.97	21.84
	Cu	0.8	14.90
	Pd	0.60	2.78
Location 2	C	2.51	29.18
	Si	1.03	38.56
	Ti	1.02	21.69
	Cu	0.81	8.07
	Pd	0.60	2.50

Table II – Results of the SEM analysis on Pd/Ti thin film cathode.

Silicon is found in the composition of the quartz substrate, Titanium and Palladium were sputtered on the substrate. There is no conventional explanation though for the amount of Carbon that was found on the cathode surface. The same electrolysis cell, connectors, cap and insulators were used as in the previous experiments. Unlike after the first experiment described here, no elements with Z around the elements the cathode was made of, that is Titanium and Palladium, were found. There is no conventional explanation for the amount of Copper found on the cathode after the loading, as well.

#### 4. Experiments with Palladium foil

Another experiment was completed using the same experimental setup presented in [16]. A Palladium foil 99.9% purity, with the thickness of 0.025 mm was used as cathode and the anode was a Pt wire, purity 99.997 %.

The electrolyte consisted of  $\text{Li}_2\text{SO}_4$  in  $\text{D}_2\text{O}$ , 0.5M. The experiment performed with this cathode was conducted with a higher current density, 7.5  $\text{mA}/\text{cm}^2$ . The results of SEM quantitative analysis performed on the cathode after the experiment are presented in Table III. Examining SEM images of the cathode after experiment we notice profound micro-cracks that reveal the existence of a strong mechanical stress that produced them. This cathode absorbed Deuterium in big amount. The SEM results presented in Table IV reveal that no element with Z around Palladium was found. Again, Carbon was found in considerable amount on the surface after the cathode achieved a high Deuterium loading ratio.

Element	Atomic concentration, %
C	23.68
Pd	76.32

Table III – Results of the SEM analysis on Pd thin foil cathode.

## 5. Discussion

With these results and observations in mind the appearance of new and unexpected elements on the cathode surface can be associated with achieving a high H isotopes loading ratio in certain metals. These results, taken alone, appear to be surprising, but they should be viewed in the context of other papers written on this subject. In reference [2], after a similar experiment, Calcium was found in electrolyte and after repeating the experiment Tritium was found in the electrolyte as well [3]. A different type of experiment that is by arcing between two carbon rods in water produced Iron, Silicon, Nickel, Aluminum, and Chromium in the Carbon residue in [4] and only Iron in [5]. Tritium in electrolyte after electrolyses is reported in [6] and [7] and Iron on the Gold electrode in [8]. In [9] elements with A between 231 and 234 were found. References [10] and [11] deal with Palladium loaded with Deuterium. Titanium and Calcium was found in [10]. Energy dispersive X-ray analysis (EDAX) revealed the formation of Zn in the Pd cathode that was subject to a 2 atm D<sub>2</sub> gas loading experiment, described in [11]. In [12] both a Titanium and a Palladium cathode were loaded with Deuterium. SEM and Energy Dispersive Spectrometry (EDS) were employed in analyzing the samples and elements like V, Cr, Fe, Ni and Zn were found to be produced during the experiments.

A Pd rod was electrolytically charged with Deuterium in [13]. The gas within the Pd was analyzed afterwards by Mass Spectroscopy and masses of 2, 3, 4, 6, 17, 18, 19 and 20 were found. SEM and EDX revealed the presence of Ruthenium and Indium. Within the grain structure, Si, S and Ca were also found. A similar experiment is described in [14] and surface impurities after electrolysis were Pt, Ni, Zn, Cu Cr, Fe, Ag, and, on one occasion Pb. Reference [15] describes a more detailed analysis. When the cell temperature was raised to 90°C, deposits on cathode surface formed very quickly, especially Silicon and after long electrolysis there was (besides Pd) Pt, Si and Zn.

The experiments described in this work reveal that different elements, both close (Cu) and far (P), in respect to Z, of the host metal lattice appeared on the Nickel surface after it was loaded with Hydrogen and Deuterium. A different result was obtained when Titanium, Palladium or a combination of them was loaded with Deuterium. When the experiment parameters were adjusted in such a manner that the cathode absorbed Deuterium a considerable amount of Carbon was found on the surface. These results suggest that there is a strong correlation of achieving a high loading ratio with the occurrence of new elements on the cathode.

In references as [18] it is suggested that proton – metal reactions might occur in special conditions, that might be fulfilled during electrolysis. The nuclear reaction  $p+Ni \rightarrow Cu$ , undergoing in a novel, unconventional and unexplained yet way might stand for the amount of Copper that was detected after the experiment on the cathode. The nuclear reaction might carry on in a different way when the protons are confined in a lattice. Another factor that makes the low energy nuclear reactions evolve in a different way is the “free” electron concentration that can be doubled when Ni is loaded with H at a loading ratio close to one, simply because the sample keeps being electrically neutral. The

increased “free” electron concentration can act like a strong screening factor for the Coulomb barrier, as it was calculated in papers like [19] but the above mentioned increase can not stand as the only explanation for the low energy nuclear transmutations. The appearance of Carbon on Pd after being loaded with Deuterium might be the result of Catalyzed Nuclear Reactions undergoing in a novel way. The multi-body fusion of D-s is a process that is very improbable in vacuum but can become significant in the presence of an increased electron concentration inside a loaded lattice and in a strong confinement.

The H and D loading experiments and concentration analysis described in this paper fit well in the experimental “landscape” consisting of the results briefly reviewed here and of many others as important as these, but not mentioned in this paper. Element concentration changes after H isotope loading experiments in different metals are intriguing, by the lack of a satisfactory conventional explanation, but this makes them worth being investigated. Future work is planned to extend the experiments and to improve the accuracy of the results.

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