



## Electrochemical characteristics of Pd-P electroless thin films deposited on carbon steel

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**Abstract** – In this study Pd-P electroless alloys with different phosphorous content were electrochemically characterized for hydrogen evolution reaction. The results pointed to different mechanisms related mainly with the phosphorous content which in turn can be related to the different microstructures present in the films. The results also showed that compared to pure palladium, Pd-P alloys are better catalyst for hydrogenation reactions, because their higher ability to hydrogen adsorption.

Palladium and palladium–phosphorous (Pd-P) alloys obtained via electroless deposition are widely used as membranes in many kinds of substrates, mainly due to the palladium based materials ability to adsorb/absorb hydrogen, and its application in hydrogen storage, and to its catalytic properties towards hydrogen evolution reaction (HER). However, data on the deposition mechanism and on the phosphorous content influence on the electrochemical characteristics of the film are scarce in the specialised literature. In this context, the present work investigates the influence of the phosphorous content on the electrochemical characteristics of Pd-P alloys obtained over carbon steel by electroless deposition, by using Tafel plots and electrochemical impedance spectroscopy (EIS).

In the alloys deposition process, the carbon steel (AISI 1045) disks were polished to mirror finishing and submitted to an electrolytic alkaline etching and a sensitization/activation process [1] before the deposition. The samples were immersed in a plating bath described in details elsewhere [2] composed by a palladium-amine complex  $[Pd(NH_3)_4Cl_2]$  and  $NaH_2PO_2$ , used as d agent, at constant temperature ( $50 \pm 0.1$  °C) and agitation, for 120 minutes. Film samples with different P content were prepared by varying the reducing agent concentration. All the solutions were prepared with P.A. grade reactants and type 1 water. The EIS experiments were performed in the potentials corresponding to the electrical double layer potential region (edl) and to the HER, with the alternate signal frequency ranging from 5 Hz to 15 kHz and amplitude of 5 mV (rms). Equivalent circuits were adjusted to the EIS experimental results using EQUIVCRT software[3]. The potentiodynamic polarisation curves were obtained at  $0.1$  mV s<sup>-1</sup>. All the electrochemical experiments were performed in an AUTOLAB PGSTAT20 (EchoChemie) potentiostat in a KOH  $0.1$  mol dm<sup>-3</sup> aqueous working solution, using a platinum sheet as counter electrode and a saturated calomel electrode (SCE) as reference electrode. The working electrodes were Pd bulk, Pd<sub>88</sub>P<sub>12</sub> and Pd<sub>86</sub>P<sub>14</sub> (wt. %). Prior electrochemical measurements, the alloys electrodes were structurally and morphologically characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM).

The results indicate that small phosphorous content variations strongly affect the mechanism of hydrogen evolution reaction and hydrogen adsorption/absorption, mainly due to different morphological and structural characteristics, which for the materials properties are indistinguishably related.

### References

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