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# Electrochemical sorption of hydrogen in exfoliated graphite/nickel/palladium composite

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## ABSTRACT

The aim of the present work was to examine the processes of hydrogen sorption/desorption occurring in alkaline solution for electrodes made of EG/Ni/Pd (exfoliated graphite/nickel/palladium) composite. The electrochemical investigations were carried out using cyclic voltammetry technique accompanied by the potentiostatic polarization of electrode material at negative potential. To saturate the electrode with hydrogen, when an electrode reached the potential of  $-1.2$  V, the potential was stopped for a chosen time. After that the potential scanning was continued. Anodic charge insignificantly increases with increasing the hydrogen sorption duration, whereas the changes in shape as well as in location of anodic peaks noted in response to the cathodic reactions of hydrogen sorption are much more pronounced. It indicates that mechanism and kinetics of the oxidation/desorption processes are altered due to increase in sorption time. The SEM (scanning electron microscopy) equipped with EDS (energy dispersive spectrometer) detector and XRD (X-ray diffraction) analysis were used to characterize the investigated composite material.

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## Introduction

Hydrogen is still recognized as an important energy source for many practical applications, therefore many investigations are focused on the issue of hydrogen storage. Hydrogen sorption can be performed on the way of physical, chemical and electrochemical processes [1,2]. In the latter case there is no need to use high pressure and the process occurs directly at electrode material under ambient conditions. The mechanism as well as capacity of hydrogen storage are strongly influenced by the properties of the electrode material used as hydrogen reservoir. Among the materials which have been investigated for the purpose of hydrogen storage one can find various forms of carbon, e.g.

activated carbons [1,3,4], carbon nanofibers [5–7], carbon nanotubes [7,8] and other nanostructured carbon materials [9,10]. From the literature data it is known that graphite based materials such as graphite intercalation compounds [11], graphene [12] and graphene oxide [13] can be also recognized as a promising material for hydrogen storage. It was found that the hydrogen storage capacity for carbon materials can be significantly improved by the incorporation into their structures some transition metals such as Ni, Pt, Pd and interstitial metal alloys exhibiting a low overpotential in the hydrogen adsorption/absorption reaction [5,6,14–16]. Our previous works have revealed that the products of chemical as well as thermal exfoliation of graphite intercalation compound with nickel chloride (NiCl<sub>2</sub>-GIC) [17,18] and with nickel chloride, iron chloride and palladium chloride

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(NiCl<sub>2</sub>–FeCl<sub>3</sub>–PdCl<sub>2</sub>–GIC) [19] show enhanced activity in the electrochemical processes of long lasting hydrogen sorption and its subsequent desorption.

The purpose of this work was to study the electrochemical activity of electrode made of EG/Ni/Pd composite demonstrated before and after potentiostatic electrosorption of hydrogen. Electrochemical investigations were carried out using cyclic voltammetry technique. For better understanding the influence of composite components on electrochemical activity, the results obtained for EG/Ni/Pd composite were discussed in relation to the results acquired for EG/Ni composite. Electrochemical results were supported by the SEM observations related with the EDS analysis and information gathered from XRD analysis.

## Experimental

### Preparation of electrode material

The investigated EG/Ni/Pd composite was prepared according to a three-step synthesis. During the first step, exfoliated graphite (EG) being a matrix of the examined composite was prepared by thermal exfoliation of a stage-5 graphite intercalation compound with sulfuric acid (H<sub>2</sub>SO<sub>4</sub>-GIC). Process of exfoliation was carried out at the temperature of 800 °C for 4 min, in air atmosphere. In order to obtain EG/Ni composite, the electrodeposition of Ni from the bath composed of NiSO<sub>4</sub> × 7H<sub>2</sub>O (140 g dm<sup>-3</sup>), NiCl<sub>2</sub> × 6H<sub>2</sub>O (5 g dm<sup>-3</sup>), and H<sub>3</sub>BO<sub>3</sub> (20 g dm<sup>-3</sup>) was performed. Galvanostatic deposition of Ni was carried out by passing through the electrode made of EG constant current density equal to 20 mA g<sup>-1</sup>. This process was performed in electrochemical cell in which EG was a working electrode, Hg/Hg<sub>2</sub>SO<sub>4</sub>/1 M H<sub>2</sub>SO<sub>4</sub> was a reference electrode, whereas Ni rod was used as the counter electrode. Owing to the chemical treatment of EG/Ni composite in PdCl<sub>2</sub>/HCl water solution, the layer of Pd was formed thus yielding final product EG/Ni/Pd composite.

### Electrochemical measurements

Electrochemical measurements were carried out in 6 M KOH at ambient temperature by cyclic voltammetry technique (CV) using a potentiostat-galvanostat PGSTAT 30 AutoLab (EcoChimie B.V, Holland). Three-compartment cell was composed of powder type EG/Ni/Pd electrode as a working electrode, Hg/HgO/6 M KOH electrode as a reference electrode (0.098 V vs. NHE) and a graphite rod (5 mm in diameter) playing a role of counter electrode. The graphite rod of a similar parameters to counter electrode was used as a current collector. In each measurement, the mass of electrode was equal to 30 mg. More details of electrode preparation procedure can be found in our previous papers [17–19]. Voltammetric processes were performed with a scan rate 10 mV s<sup>-1</sup> within the potential range of –1.2 ↔ 0.6 V, starting from the rest potential of electrode (E<sub>R</sub>) towards the negative potentials. The potentiostatic process of hydrogen saturation was performed as follows. During a selected cycle, when the electrode reached the potential of –1.2 V, the potential scanning was automatically stopped for 15, 30, 45, 180 and

900 min. After finishing the potentiostatic treatment, potential scanning was continued in the positive direction.

### Material characterization

Morphology of EG/Ni and EG/Ni/Pd was studied by scanning electron microscopy (SEM) (S-3400N, Hitachi microscope) using a voltage acceleration of electron beam equal to 15 keV. The information confirming the EG/Ni/Pd composition were acquired from the EDS analysis (Energy Dispersive Spectrometry).

The information on the crystalline structure as well as composition of the synthesized material were gathered from X-ray diffraction (XRD) measurements (Philips PW-1710 diffractometer) performed using CuK<sub>α</sub> radiation with nickel filtering.

## Results and discussion

### SEM observations and XRD analysis

On SEM images shown in Fig. 1a one can observe distribution of Ni on the surface of EG/Ni composite whereas Fig. 1b–d display the images of Pd existing on the EG/Ni/Pd surface.

The surface of the investigated material is irregularly coated with spherical structures representing Ni (Fig. 1a). Taking into account the distribution of Ni, it should be emphasized that the surface defects as well as edges are the most favorable location of Ni during its electrodeposition on the surface EG. From Fig. 1b–d it is known, that the surface of EG/Ni/Pd is almost entirely covered with layer mainly composed of Pd particles of a different size.

The particular analysis of SEM images made by SEM software enabling the estimation of thickness of the observed particles have confirmed that the layers of metal deposits both onto the EG and EG/Ni surface are not uniform. The above mentioned calculations revealed that the thickness of Ni deposits on exfoliated graphite ranged from 0.130 to 0.850 μm. In case of Pd layer, it is difficult to separate the Pd deposit from Ni, therefore, the thickness of Pd layer can be calculated from the sum of Ni and Pd deposit. The thickness of Ni/Pd layer existed on exfoliated graphite surface ranged from 0.240 to 1.94 μm. On the other hand, basing on the particular comparison of Ni and Pd particles, it can be assumed that Pd layer is thicker.

The results of EDS analysis revealed that the average concentration of Pd and Ni on EG/Ni/Pd surface is equal to 7% and 1%, respectively. Carbon (81%) and oxygen (11%) appear to be the main components of the surface of the regarded material.

XRD pattern for EG/Ni/Pd (see Fig. 2) indicates that the components of the examined composite exist as a separated phases. On the regarded pattern there are signals corresponded to graphite matrix and well separated signals arisen from Ni as well as Pd. Obviously, besides the graphitic peak the signals from Pd component are of a highest intensity. From Fig. 2 it is clear that during the chemical deposition of Pd onto EG/Ni surface, the Pd–Ni alloys are not formed.

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