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ScienceDirect

Physics Procedia 84 (2016) 407 – 414

Physics Procedia

International Conference "Synchrotron and Free electron laser Radiation: generation and application", SFR-2016, 4-8 July 2016, Novosibirsk, Russia

Ni nanostructures in porous anodic alumina matrices: structure and cathodic properties in hydrogen release reactions

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Abstract

A synthesis has been performed of Ni nanostructures by magnetron deposition on the matrices of porous alumina obtained by the two-stage anodic oxidation. The scanning electron microscopy method is used to investigate alumina films before and after nickel deposition. Investigations by X-ray diffraction, Raman spectroscopy and X-ray photoelectron spectroscopy and EXAFS show that the nickel deposited on porous alumina is in an oxidized state, in contrast to nickel deposited on a substrate with a smooth surface. No influence of the porous structure of matrices on the structure of nickel and the composition of near-surface layers has been detected. The catalytic properties of the nanostructures in hydrogen release reactions in a 5 M NaOH solution are analyzed. It is shown that the nickel films deposited on porous alumina exhibit a greater catalytic activity in hydrogen release reactions than the nickel film deposited on a smooth aluminum substrate.

Peer-review under responsibility of the organizing committee of SFR-2016.

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Peer-review under responsibility of the organizing committee of SFR-2016.
doi:10.1016/j.phpro.2016.11.069

Keywords: Ni nanostructures; porous alumina; magnetron deposition; SEM; XRD; Raman spectroscopy; XPS; EXAFS, hydrogen release reaction

1. Introduction

Interest in hydrogen as an alternative source of energy is currently motivated by two factors [1-3]: first, by environmental pollution due to the use of fossil fuels, which are the main sources of energy at present [2], second, by the fact that fossil fuel reserves are finite [3]. Hydrogen as an alternative to fossile fuels is a solution to the above-mentioned challenges [2].

At present there exist many methods for obtaining hydrogen [4,5]. One of them is the electrolytic reaction of hydrogen release from aqueous solutions of acids and alkalis [5]. In this reaction, a lot of attention is paid to cathodes, which were formerly manufactured mainly of platinum metals. Recently they have been succeeded by cathodes containing various metals, among which nickel is the most widely used [6]. It should be noted that the hydrogen release reaction proceeds the more effectively the larger the area of the cathode surface [7]. In this paper, nickel deposited on the highly developed surface of porous anodic alumina matrices by magnetron deposition is proposed to be used as cathode material for hydrogen release reactions.

Thus, the goal of this work is to perform a synthesis of nickel nanostructures into porous alumina matrices and to investigate their catalytic activity in hydrogen release reactions in an alkaline medium.

2. Materials and methods

The synthesis of porous alumina films was performed in a two-electrode electrochemical cell. A stainless steel electrode was used as cathode material. The current was supplied from a power unit, which allowed us to set anode voltage and current within the range of 0 to 299 V and 0 to 299 mA, respectively. The average pore diameter was estimated by statistical methods using the image processing software ImageJ [8].

The crystal structure and the phase composition of the samples were analyzed by X-ray diffraction (XRD) using the diffractometer RugakuMiniflex 600 with Co – K_α excitation (Physical-Technical Institute of UB RAS) and by Raman light scattering by means of the Raman spectrometer/microscope RenishawInVia (Department of Material Sciences, Lomonosov Moscow State University) using an Ar^+ laser as an excitation source with a wave length of 514 nm.

The chemical composition and the electronic structure were analyzed by X-ray photoelectron spectroscopy (XPS) using the X-ray electron spectrometer SPECS (Physical-Technical Institute of UB RAS). The photoelectron spectrum was excited using AlK_α radiation. The depth analysis of the chemical composition of the samples was carried out by ionic etching with an ion gun voltage of 1 keV within 4 cycles, each cycle lasting 1 minute.

Analysis of the microstructure of the samples was performed using the scanning electron microscope FEI Inspect S50 (Udmurt State University). The voltage on the accelerating electrode was 20 kV; magnification were from 100x to 150000x.

The EXAFS spectra (Ni-K edges) of the studied samples were obtained at the “EXAFS spectroscopy” station, Beamline No.8 of VEPP-3 storage ring at the Siberian Synchrotron and Terahertz Radiation Center (SSTRC), Novosibirsk, Russia. using Si (111) crystal monochromator.

The hydrogen release reaction was performed in a three-electrode electrochemical cell in a 5 M NaOH solution. An alumina film with deposited nickel with a visible surface of 0.28 cm^2 was used as the main electrode, and a platinum electrode submerged into a 5 M aqueous NaOH solution was used as an auxiliary electrode. A silver-silver chloride electrode submerged into a 3.5 M KCl solution served as a reference electrode.

3. Experimental

3.1. Preparation of porous anodic alumina films

To obtain porous Al_2O_3 films, 0.2 mm aluminum foil was used. Before anodization, the samples were