

Investigation of C-Pd films in hydrogen atmosphere using molecular spectroscopy

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ABSTRACT

In this paper influence of hydrogen on molecular structure of carbonaceous-palladium (C-Pd) films using Raman and FTIR spectroscopy methods has been studied. The special experimental setup (gas cell) was design and realized. The spectra were measured in H_2/N_2 atmosphere under different gas pressure and flow rate and compared with base spectra measured in air. The noticeable influence of gas flow on intensity of some bands in FTIR spectra were observed. Understanding of role of H_2 in interaction with C-Pd film needs other experimental works. Observed phenomenon can be used in construction of optical hydrogen sensor.

Keywords: C-Pd layer, palladium hydride, hydrogen sensor, FTIR spectroscopy.

1. INTRODUCTION

From many years, palladium has been known as the hydrogen absorber and thus it is often used in the devices and the gas detecting systems [1,2]. A compound called palladium hydride (PdH_x) which is formed during gas-metal interaction has different electrical and optical properties in relation to the pure palladium [3]. Recently, monolithic metallic structures are substituted by composite materials including Pd nanograins due to the relatively high prices of pure palladium. Carbon-palladium nanostructures (C-Pd) are one of the example, which are also considered as promising candidates in detection of hydrogen [4-7]. One of the simplest technology to produce the C-Pd film is Physical Vapour Deposition method (PVD), where C_{60} fullerene and palladium acetate are used as a carbon and as a palladium source respectively [8-9]. In effect, C-Pd nanocomposites are obtained, where palladium nanograins are embedded in a carbon (fullerene) matrix.

Spectroscopic methods, in particular the Raman and FTIR spectroscopy are common methods of the carbon nanomaterials investigation. In this article the experimental results of hydrogen influence on the C-Pd films using molecular spectroscopy methods were presented.

Effects of hydrogenation of the fullerene-palladium nanostructures can be tracked by analyze of the integral intenseness changes (I^{int}) of the absorption bands derived from vibrations of groups C-H ($\nu(CH) = 2800 - 3100 \text{ cm}^{-1}$), that undergo marked changes during hydrogenation process. Additionally, relationships $I^{int} = f(t)$ for the bands with small intensity in range $810-880 \text{ cm}^{-1}$ were analyzed, where bands deriving from vibrations of Pd-H system are expected [10]. The analysis of literature data showed that the dominant product of C_{60} hydrogenation may be $C_{60}H_{36}$ or $C_{60}H_{18}$ systems. The formation of these reaction products was confirmed by analyzing the IR spectra in which the absorption bands were located respectively at 1452 i 1739 cm^{-1} (in range of $1400-1800 \text{ cm}^{-1}$), and at $755, 729 \text{ cm}^{-1}$ for both systems as well as at $2831, 2849$ and 2912 cm^{-1} for $C_{60}H_{36}$ and at $2835, 2854$ and 2917 cm^{-1} for $C_{60}H_{18}$ respectively [11-13]. Processes of hydrogenation of C_{60} fullerene may also lead to the formation of other products that have been reported by empirical formula $C_{60}H_x$, where $44 < x < 52$ [11,14]. The IR spectra show the appearance of bands at $2886, 2952 \text{ cm}^{-1}$, and also in the area of $3000-3100 \text{ cm}^{-1}$. In the last area of an IR spectrum bands appear derived from stretching vibration C-H of aromatic systems. A presence of bands in the spectra originating from the stretching vibration $\nu(C-C)$ in the region $1450-1650 \text{ cm}^{-1}$ and $1250-1300 \text{ cm}^{-1}$ as well as bending vibrations $\delta(C-C)$, $\delta(C-H)$ in a plane in area of $900-1150 \text{ cm}^{-1}$ and out of the plane in the area of $650-900 \text{ cm}^{-1}$ is the confirmation of the establishment of the aromatic system.

Another possibility is the appearance of absorption bands derived from vibrations of the metal-hydrogen functional groups of the $C_{60}Pd_n$ system subjected to hydrogenation process. Systems with an empirical formula $C_{60}M_nH_x$ ($x = 2, 4, 6, 8$) or $C_{60}M_6H_{48}$ ($M = Ti$) may be products of this process [15, 16]. Bands derived from terminal groups M-H appear respectively at $1700-2250 \text{ cm}^{-1}$ ($\nu(M-H)$) and $600-900 \text{ cm}^{-1}$ ($\delta(M-H)$) in the vibrational spectra. In the case of the