

Properties of Pd–C films for hydrogen storage applications

Elżbieta Czerwosz¹, Piotr Dłużewski², Ewa Kowalska¹, Mirosław Kozłowski¹, and Joanna Rymarczyk^{*1}

¹ Tele & Radio Research Institute, 11 Ratuszowa, 03-450 Warsaw, Poland

² Institute of Physics PAS, 32/46 al. Lotników, 02-668 Warsaw, Poland

Received 13 September 2010, revised 17 December 2010, accepted 17 December 2010

Published online 8 July 2011

Keywords palladium, carbon, AFM, SEM, TEM

* Corresponding author: e-mail joanna.rymarczyk@itr.org.pl

Carbon nanoporous films (Pd–C films) containing Pd nanograins for hydrogen storage devices and hydrogen sensor application were prepared by two steps PVD/CVD method. Development of a carbon surface and size of Pd nanocrystals

were studied for these films. The structure, topography and morphology of Pd–C films were studied by AFM, SEM and TEM methods. SEM and TEM images show that Pd nanograins are enclosed in graphite shells.

© 2011 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim

1 Introduction The high rate of depletion of fossil fuels is the cause of seeking new and better sources of energy. Hydrogen can be stored in special materials and then energy can be retrieved [1]. The problem of hydrogen-based energy economy is still the hydrogen storage and the lack of a safety. Interaction of hydrogen with transition metals to the reversible formation of metal hydrides is of fundamental relevance to practical application in hydrogen storage, hydrogen sensor, catalysis and corrosion [2]. Research of nanocrystalline materials have been shown to have distinct differences in their hydrogen uptake characteristics when compared with their bulk counterparts [3]. Palladium and especially nanopalladium grains are well known as one of the best adsorbents for hydrogen gases. Recent studies have shown that the amount of hydrogen uptake is higher in nanocrystalline PdH_x than that in the bulk Pd metallic surface. H₂ gets dissociated to hydrogen atoms on the Pd surface and diffuses into the Pd to form a solid solution of Pd/H. The hydrogen is sorbed in Pd as dissolved H (α -phase) and as metal hydride (β -phase). The transition between these phases depends on the pressure and temperature at which the isotherm is measured [4, 5].

Carbon is also very good adsorbent for some gases due to its ability to form various structural combinations (e.g. nanoporous, nanoions, nanotubes, nanofibers). Theoretical and experimental studies have shown that the size distribution of the pore in carbonaceous films affects hydrogen storage capacity [6].

In this paper we present the results of studies of morphology, topography and structure of Pd–C films that are

potentially good material for many applications in hydrogen sensitive devices.

2 Experimental Nanoporous Pd–C films were prepared by a two steps method consisting of physical vapor deposition (PVD) and chemical vapor deposition (CVD) processes. In PVD process, a nanocomposite film built of Pd nanocrystals embedded in a carbonaceous matrix was obtained and in the second step this film was modified in CVD process by xylene pyrolysis in a temperature of 650°C. In PVD processes films evaporated from two separated sources – fullerene C₆₀ and palladium acetate. Three types of samples were prepared with various technological parameters (see Table 1). Films were obtained on many substrates such as: molybdenum foil, Al₂O₃ ceramic and Si monocrystalline plates.

Table 1 Technological parameters of PVD processes.

sample	intensity of evaporation		time [min]	substrate
	I_{C60} [A]	I_M [A]		
S1-PVD	2	1.2	10	Al ₂ O ₃ /Mo
S2-PVD	2.1	1.2	10	Al ₂ O ₃ /Mo/Si
S3-PVD	2.1	1.2	8	Si

In PVD chamber the distance from the sources to the substrates was the same for all processes. The temperature of the substrates during the growth processes was ~100 °C.

The content of Pd was determined by atomic absorption spectroscopy and in all samples it was ~15–19 wt%.