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Method of determination of palladium concentration for C–Pd nanostructural films as a function of film thickness, roughness and topography

JOANNA RYMARCZYK*, MIROSŁAW KOZŁOWSKI

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

In this paper a method of determination of Pd in a carbon-palladium film (C–Pd film) deposited on a quartz substrate is presented. This method is based on energy dispersive X-ray spectroscopy (EDX) and all experiments were performed using a scanning electron microscope (SEM) equipped with EDX system. Qualitative and quantitative analyses were carried out for C–Pd films prepared by PVD method in different technological conditions. It was shown that results of the experiments depended on the structural model, film thickness and electron beam energy used for Pd content calculation.

This method enabled us to conclude on the homogeneity of palladium distribution in the whole volume of carbonaceous matrix, depending on the parameters of PVD process. Additionally, these studies showed that a different palladium concentration in C–Pd films had a significant impact on their topography and morphology.

Keywords: *palladium; carbon; SEM; EDX*

1. Introduction

Palladium is an element which exhibits strong sorption properties compared to hydrogen. Hydrogen absorption by palladium leads to formation of palladium hydride in gaseous environment. The resulting compounds are characterised by poor stability and good reversibility [1–4]. Different carbon nanomaterials also react to gas containing hydrogen with different concentrations. For carbon-palladium films it can be assumed that C–Pd films exhibit combined sorption properties of palladium and carbon in various allotropic forms in hydrogen-containing gaseous environment. Studies conducted so far indicate that the reactivity of C–Pd films to hydrogen and its compounds depends on palladium content in the film which mostly varies depending on the PVD process by which such films are obtained. The results of our study suggest that the reactivity of C–Pd films to hydrogen and its compounds depends on palladium content in the film as a result of the technological conditions during the PVD process by which the films have been obtained [5–7].

Quantitative and qualitative analyses of the composition of C–Pd films obtained under different process technological conditions were carried out using scanning electron microscopy and X-ray spectrometry. Scanning electron microscope with field emission is one of the most popular instruments used for determining parameters of thin films and layers. The great advantage of this type of microscope is the capability of operating at very low accelerating voltages (less than 2 kV) with a high resolution of 1.5 nm. Thus, it is possible to study a surface morphology with low roughness which is not possible in the case of conventional scanning electron microscope with tungsten cathode [8].

In our previous studies, the analysis of the Pd content was performed by atomic absorption spectroscopy (AAS) with a monochromatic lamp at 232 nm. The films were dissolved in toluene and then dried. The dry powder was used to pre-

*E-mail: joanna.rymarczyk@itr.org.pl