

The influence of technological PVD process parameters on the topography, crystal and molecular structure of nanocomposite films containing palladium nanograins

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The paper describes the preparation and characteristics of films composed of Pd nanograins placed in carbonaceous matrix. Films were obtained in PVD (Physical Vapor Deposition) process from two sources containing: the first one – fullerene powder and the second one – palladium acetate. The topographical, morphological and structural changes due to different parameters of PVD process were studied with the use of Atomic Force Microscopy and Scanning Electron Microscopy, whereas the structure was studied with the application of the Transmission Electron Microscopy and Fourier Transform Infrared Spectroscopy methods. It was shown that topographical changes are connected with the decomposition ratio of Pd acetate as well as the form of carbonaceous matrix formed due to this decomposition. Palladium nanograins found in all films exhibit the *fcc* structure type and their diameter changes from 2 nm to 40 nm depending on the PVD process parameters.

Keywords: palladium, carbon, SEM, AFM, TEM, FTIR.

INTRODUCTION

One of the most interesting materials are nanostructure films containing clusters, nanoparticles or nanograins of metal. Metal nanocrystals or nanograins display interesting properties, which provides extensive opportunities for their use. Nanograins with diameter of a few nanometers have more atoms on the surface than inside^{1,2}. It has a significant impact on the sorption properties of such grains. Palladium grains are commonly known as one of the best adsorbents for hydrogen gases^{3,4}. Recent studies have shown that the amount of hydrogen is higher in Pd nanocrystals, compared with a thin layer of palladium⁵.

Carbon is also very good adsorbent for some gases due to its ability to form various structural combinations (e.g. nanopores, nanotubes, nanofibers). Depending on the form of carbon, it can have different electronic properties (from insulating to metallic). By combining such two materials as metallic nanograins and carbonaceous grains it is possible to obtain a material with new properties in comparison with the macroform of these materials.

The nanocomposite carbon – palladium films (C-Pd films) presented in this work are interesting because of their ability to absorb hydrogen and due to the possibility of application of these films as hydrogen and hydrogen containing compounds sensors. The C-Pd films preparation method was elaborated in Tele and Radio Research Institute, where many investigations of film properties were performed⁶⁻⁹. In paper¹⁰ we showed that the sorption properties of films containing Pd nanograins are affected by their resolution, particle size and surface topography. In this paper we present the results of studies of the influence of technological PVD process parameters on the topography, crystalline and molecular structure of C-Pd films. Our films are applied for hydrogen sensing and they can be used as a covering layer for hydrogen storage materials, as well.

EXPERIMENT

Nanocomposite C-Pd films were obtained with the use of physical vapor deposition method (PVD) with precursors evaporated from two separated sources: C₆₀ fullerenes and palladium acetate. PVD process was performed in a vacuum chamber (dynamic vacuum 2×10^{-6} mbar). Film was deposited on a quartz substrate. Figure 1 schematically presents the PVD process.

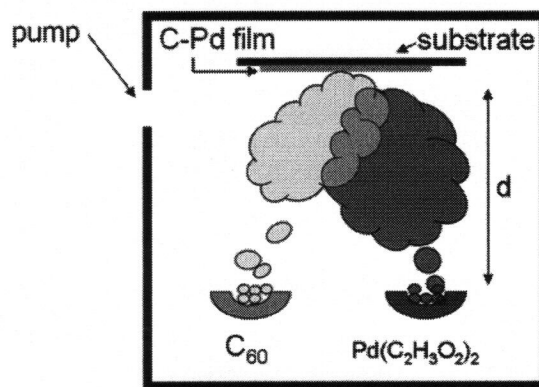


Figure 1. PVD process scheme

Sources-substrate distance (*d*) amounted to 60 mm and deposition time (*t*) was 10 min in all processes. We obtained C-Pd films of various thickness, structure, morphology, topography and Pd content depending on technological parameters of PVD process such as current intensity for two separated sources (*I*_{C₆₀}, *I*_{Pd}). Sources containing C₆₀ and palladium acetate were heated to a high vapor pressure by electrically resistive heating, separate for each source. Table 1 presents the intensity of evaporation of C₆₀ and palladium acetate use for all PVD processes. The temperature to which the sources were warmed up, corresponds to their decomposition temperature, for C₆₀ is 400–620°C (in the air) and for palladium acetate is ~250°C (organic compounds)