Preparation of nanocomposite carbonaceous films containing palladium nanograins and studies of the annealing time influence on their structure and sensing properties

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Abstract. C-Pd films were obtained by Physical Vapor Deposition (PVD) or by annealing of these initial PVD films at 650°C in different time (5, 10 and 30 minutes) in argon flow. These C-Pd films were characterized by Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and FTIR spectroscopy. The sensing properties of the films were studied in a specially prepared chamber allowing for measurements of the changes in resistivity as a function of gas composition changes. It was found that annealing changes the films morphology, topography and structure, and consequently their sensing properties.

1. Introduction

Since Seiyama [1] developed the first solid-state gas sensor, many other research groups have been attempting to improve gas sensors, with the aim of enhancing their sensing performance. Many materials such as: carbon nanotubes, nanoporous carbon, diamond-like carbon, graphene can detect low concentrations, of ppb order, of gases such as NO_2, NH_3, O_2, H_2, CO_2, and CO [2–8]. However, there are carbonaceous materials with very high resistivity 10^7–10^9 $\Omega\cdot$cm [9,10] that can be applied for sensing different gases by such resistivity limit their sensitivity. Introduction of nanoparticles or nanograins of some metals improves sensing properties. For instance, introduction of palladium nanoparticles into a carbonaceous film allows obtaining a very sensitive hydrogen sensor [11-14]. This phenomenon is connected to solubility of hydrogen in palladium lattice [15-17]. It is known that nanostructural Pd materials improve H_2 sensing in comparison with metallic palladium. In the presence of H_2 the resistivity of Pd will change due to the formation of a solid solution of Pd/H [18]. Furthermore, Pd is highly selective to H_2. Several fundamental problems are associated with bulk Pd-based hydrogen sensors. First, the diffusion of the hydrogen into bulk Pd, e.g. thick Pd film, can cause a large internal stress destroying the film irreversibly [19, 20]. Second, at room temperature the hydrogen atoms diffusion in Pd is very slow (the diffusion coefficient is 3.8×10^{-7} cm^2 /s at 298 K [21]) what causes long time of response for hydrogen. Now, palladium nanomaterials development leads to production of new class of H_2 sensors. These sensors could base on single Pd nanowires [18, 22-24] or on thin films containing nanograins of Pd [25-27]. These films can be obtained by different method (atomic layer deposition, physical vapor deposition or chemical vapor deposition). The change