HYDROGEN ADSORPTION ON NANOPOROUS Pd-C FILMS

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Introduction
Porous materials because of their properties such as a high porosity (the fraction of pore volume to the total volume) can be used in various applications ranging from gas storage to molecular sieves, absorbents, electrodes in batteries, gas sensors, catalysts, supports, filters and electronic devices. For gas sensor applications open pores are required to intensify a gas penetration into these materials.
We present carbon nanoporous materials doped by palladium nanograins (Pd-C films) that could be utilized as active layers in hydrogen/hydrogen compounds detection.

Synthesis method
Nanostructural Pd-C materials are obtained in two steps method using PVD (Physical Vapor Deposition) and CVD (Chemical Vapor Deposition) techniques. It is possible to obtain films with different morphology, topography and composition depending on parameters of these technological processes.
In PVD step, fullerene C₆₀ and palladium acetate are evaporated from two separated sources to deposit the initial films on the substrates.
PVD films are polycrystalline and are built from nanograins of carbonaceous materials (with the size of 100-200nm) and Pd (with the size of 5-10nm). Pd nanograins are hidden under films surface.
Next PVD films are modified at the temperature of 650°C in CVD process using xylene as an additional carbon source. Initial carbon matrix is changed into porous one not only due to decomposition of C₆₀ and Pd acetate present in PVD films but also Pd nanograins migration through the films volume. During this process an agglomeration phenomenon occurs and Pd nanograins size increases up to 100nm. Some of these nanograins are surrounded with graphitic shells.

Raman spectroscopy
Investigation were made using Almega XR (Nicolet) at 532nm excitation. Raman spectroscopy shows changes undergoing due to CVD modification of PVD films. Shapes of spectra are different. For PVD samples the fullerite-like structures is seen, whereas for CVD samples the obtained spectrum is similar to amorphous graphite one.

BET measurement
An adsorption isotherm of xenon on CVD films at 78K was recorded and analyzed with the Brunauer – Emmett – Teller (BET) algorithm to determine their specific surface area. The linearity of the isotherm represents multilayer adsorption of Xe on the films’ surface. The measurement indicates that the real surface area of the samples is 20 times bigger than the geometrical surface, thus CVD films should adsorb gases.
In case of H₂ we registered its absorption at 78K. Hydrogen thermodesorption (to 478K) from CVD film (with the specific surface area of 130 cm²) was 1,52x10⁷ mol. It seems that this absorption can be connected only to the porosity of the film. Adsorption on Pd nanograins can be detected in case when Pd nanograins are pure and not embedded in carbonaceous shells.

Conclusion
- Porosity of the film increases due to CVD modification
- BET results show 20 times increase of specific surface area of CVD films
- It seems that H₂ absorption is connected to the porosity of CVD films rather than with the presence of Pd nanograins

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