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HYDROGEN ADSORPTION ON NANOPOROUS Pd-C FILMS

EWA KOWALSKA[1] , MAŁGORZATA SUCHAŃSKA[3], ROBERT NOWAKOWSKI[2], ELŻBIETA CZERWOSZ[1]

[1] TELE AND RADIO RESEARCH INSTITUTE, RATUSZOWA 11, 03-450 WARSAW, POLAND
[2] INSTITUTE OF PHYSICAL CHEMISTRY PAN, KASPRZAKA 44/52, 01-224 WARSAW, POLAND
[3] KIELCE UNIVERSITY OF TECHNOLOGY, Al.1000-LECIA PP 7, 25-314 KIELCE, POLAND

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, catalyst, 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

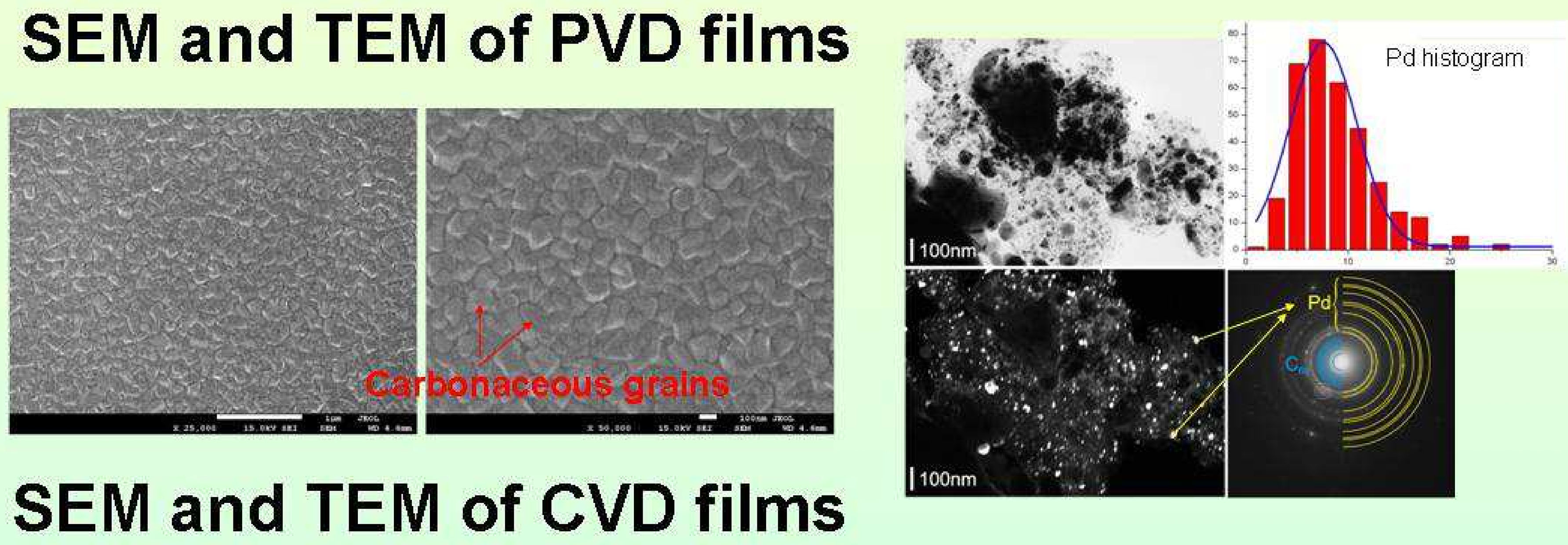
Nanostructural Pd-C materials are obtained in two steps method using PVD (Physical Vapor Deposition) and CVD (Chemical Vapor Deposition) techniques. It is possible to obtain films with different morphology, topography and composition depending on parameters of these technological processes.

In PVD step, fullerene C_{60} 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.

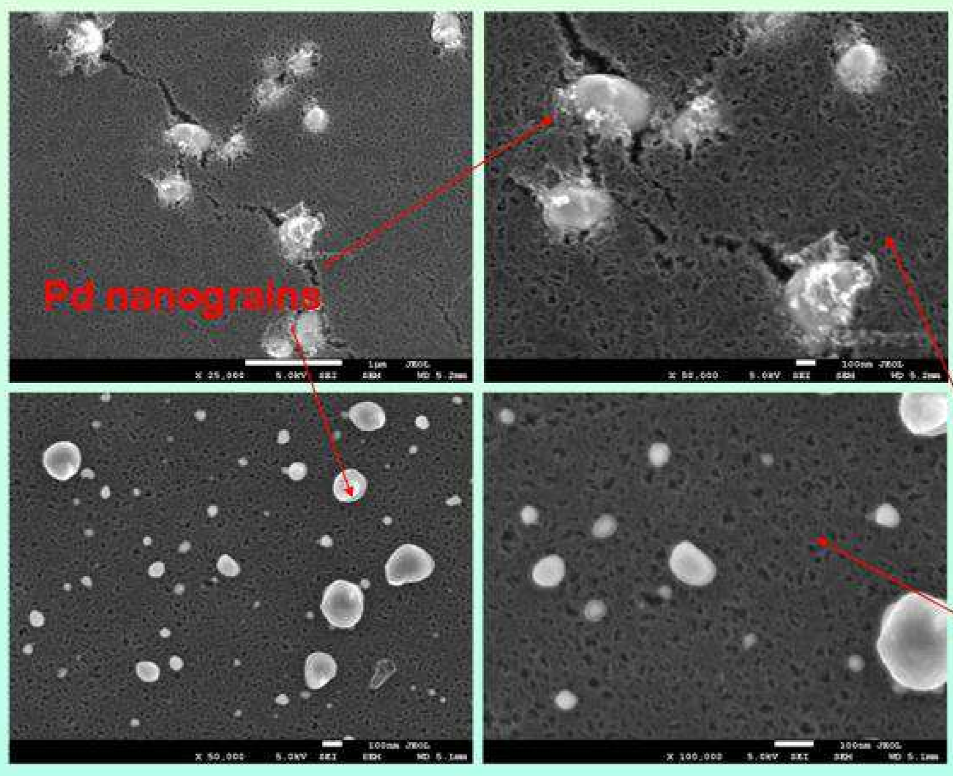
Next PVD films are modified at the temperature of 650°C in CVD process using xylene as an additional carbon source.

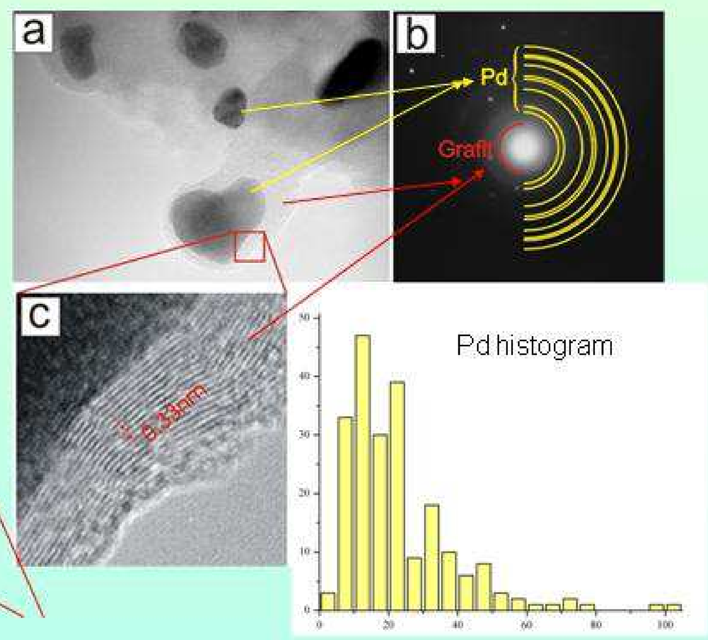
Initial carbon matrix is changed into porous one not only due to decomposition of C_{60} and Pd acetate present in PVD films but also Pd nanorgains migration trough the films volume. During this process an agglomeration phenomenon occurs and Pd nanograins size increases up to 100nm. Some of these nanograins are surrounded with graphitic shells.

SEM and TEM of PVD films



SEM and TEM of CVD films



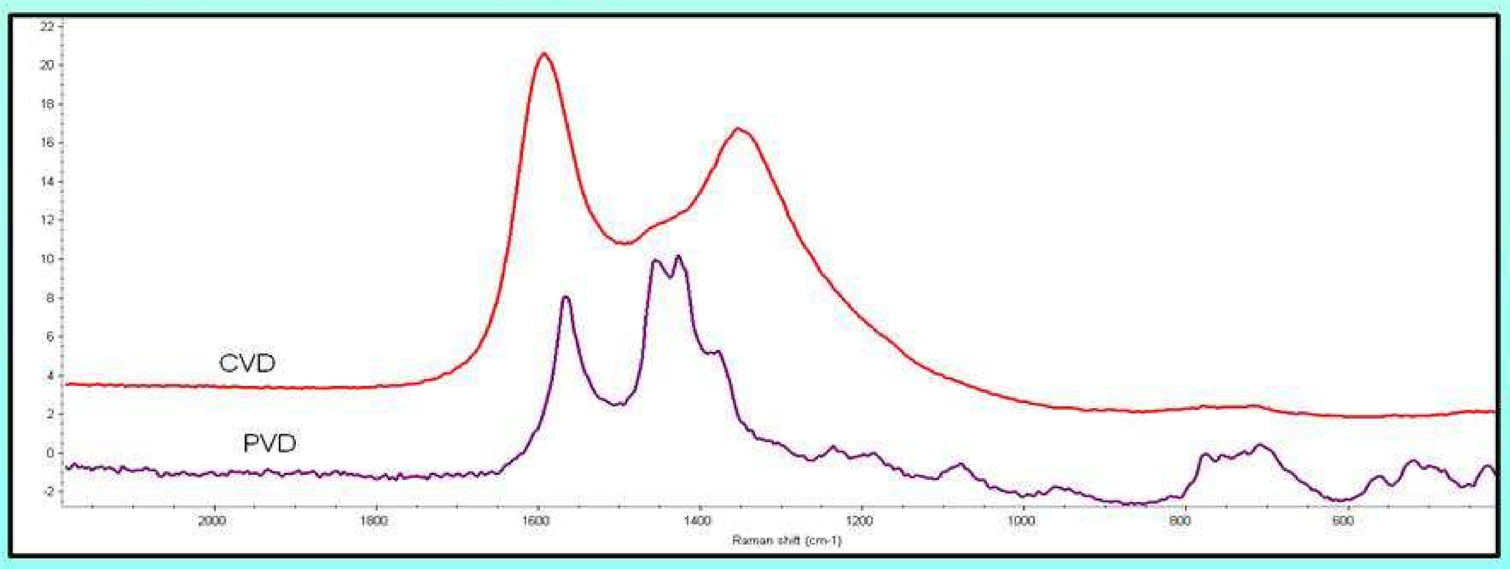


nanoporous structures

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 amorphic graphite one.

Raman spectroscopy of PVD and CVD films



BET measurement

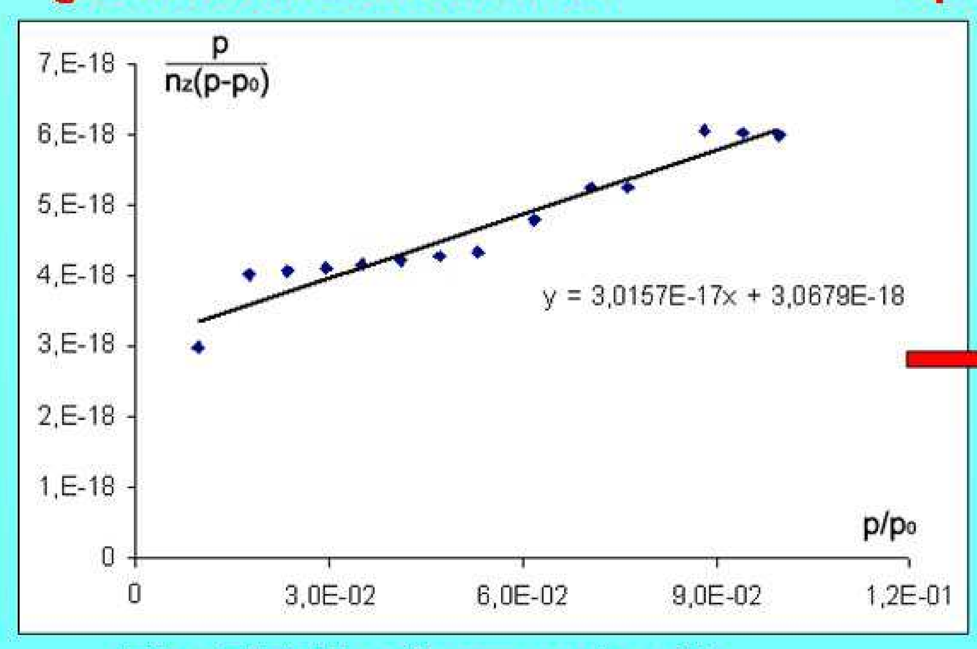
An adsorption isotherm of xenon on CVD films at 78K was recorded and analyzed with the Brunauer – Emmett – Teller (BET) algorithm to determine their specific surface area. The linearity of the isotherm represents multilayer adsorption of Xe on the films' surface. The measurement indicates that the real surface area of the samples is 20 times bigger than the geometrical surface, thus CVD films should adsorb gases.

In case of H_2 we registered its absorption at 78K. Hydrogen thermodesorption (to 478K) from CVD film (with the specific surface area of 130 cm^2) was $1{,}52\times10^{-7}$ mol. It seems that this absorption can be connected only to the porosity of the film. Absorption on Pd nanograins can be detected in case when Pd nanograins are pure and not embedded in carbonaceous shells.

CVD film with porous structure on quartz substrate

7cm² geometrical surface area

130cm² specific surface area



The BET isotherm using Xe

CVD film surface area is 20 times bigger than geometrical surface area

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 H_2 absorption is connected to the porosity of CVD films rather than with the presence of Pd nanograins

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DEVELOPMENT OF TECHNOLOGY FOR A NEW GENERATION OF THE HYDROGEN AND HYDROGEN COMPOUNDS SENSOR FOR APPLICATIONS IN ABOVE NORMATIVE CONDITIONS

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