TEMPERATURE CHANGES OF TOPOGRAPHY AND MORPHOLOGY OF C-PD FILMS DEPOSITED ON FUSED SILICA

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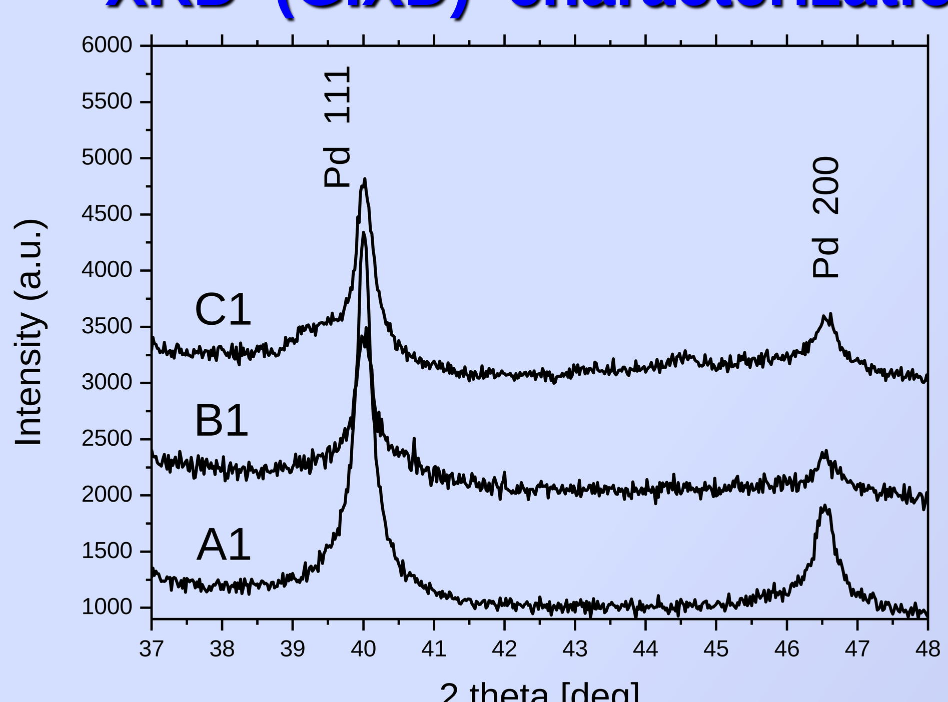
INTRODUCTION

The structure and the composition of carbonaceous – palladium (C-Pd) films that are proposed as hydrogen sensor or hydrogen storing material affect their sensing or storing properties. This type of materials can appear in the various structural forms of both components: palladium and carbon matrix. Palladium can be in the form of nanoparticles, nanowires, thin metal films, whereas carbon matrix can arise as porous, amorphous carbon or higher ordered structures. The structure of palladium is of a great importance taking into account the hydrogen sensing properties of C-Pd films.

SYNTHESIS METHOD

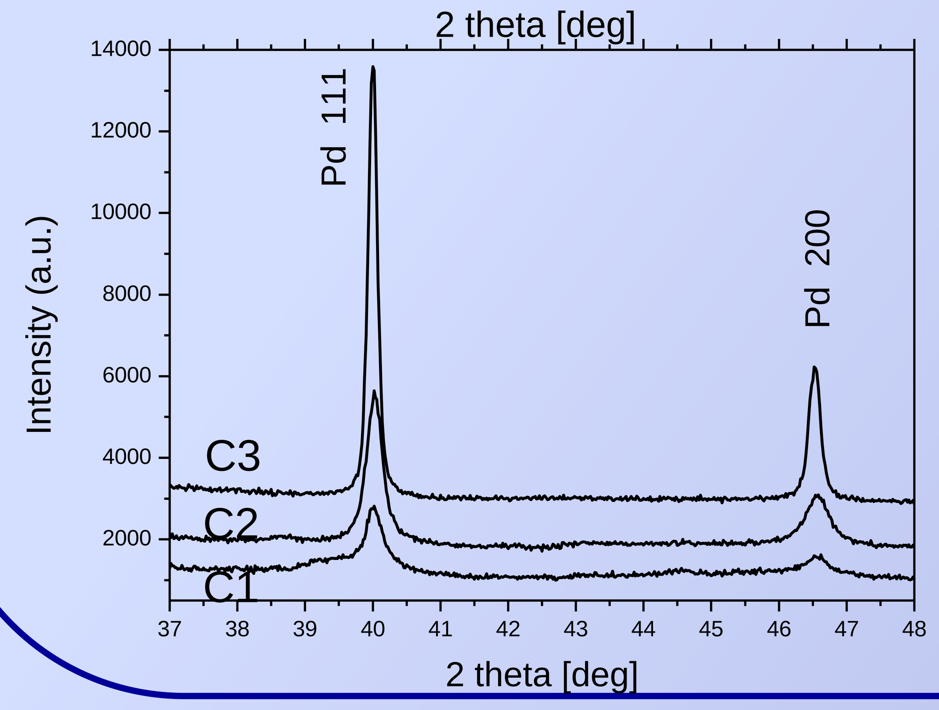
Carbonaceous – palladium films were obtained under dynamic vacuum of 10-5 mbar by PVD process. As precursors of these films fullerene C60 (99.98% pure, Aldrich Chemical Co) and palladium(II) acetate Pd(OAc)2 (pure 47%Pd, Fluka) were applied. Both compounds were evaporated from separate sources heating by electrically resistive. As the substrate an unpolished fused silica was used. The initial PVD films were called here as the films A, B and C according to the different weight content of Pd that was 25 36,4 and 46,2 wt.% respectively. PVD films subjected to modifications by annealing we call as A1, B1 and C(1-3) depending on the applied temperature. Duration time of annealing for all samples was 30 minutes.

XRD (GIXD) characterization



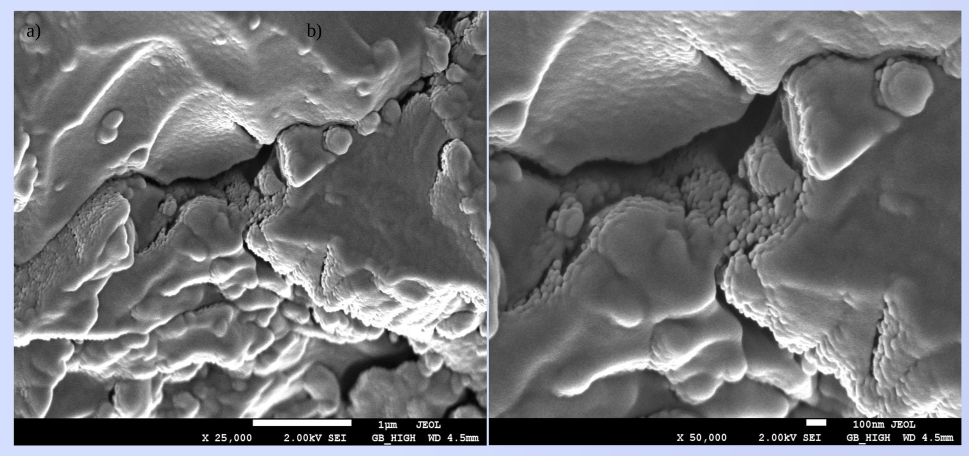
GIXD diffraction patterns of films A1, B1 and C1 (from annealing process

at 500 °C)

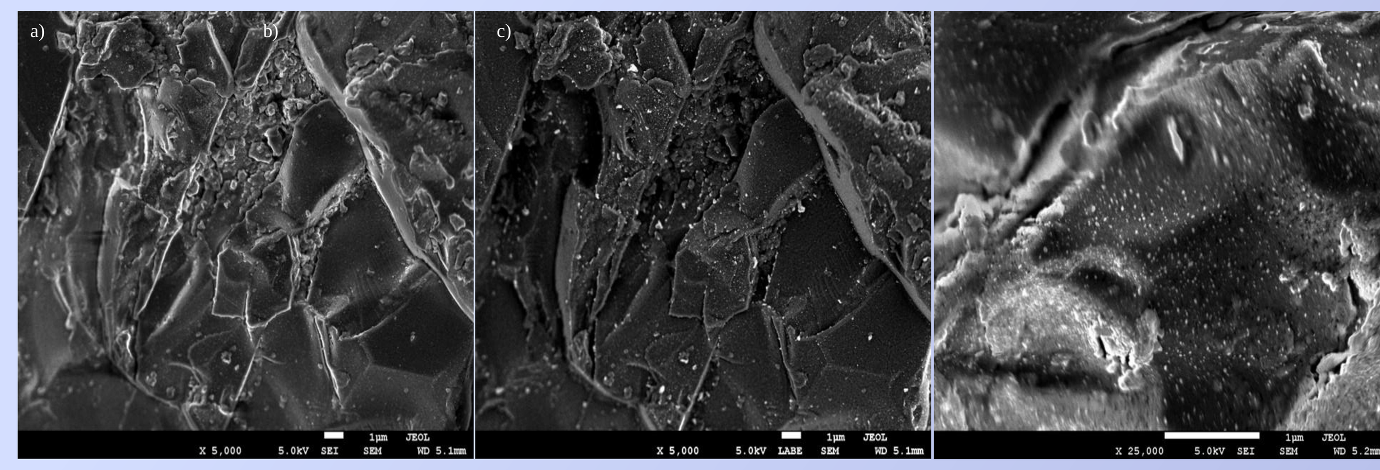


GIXD diffraction patterns of films C(1-3) annealed at 500, 600 and 700 °C respectively.

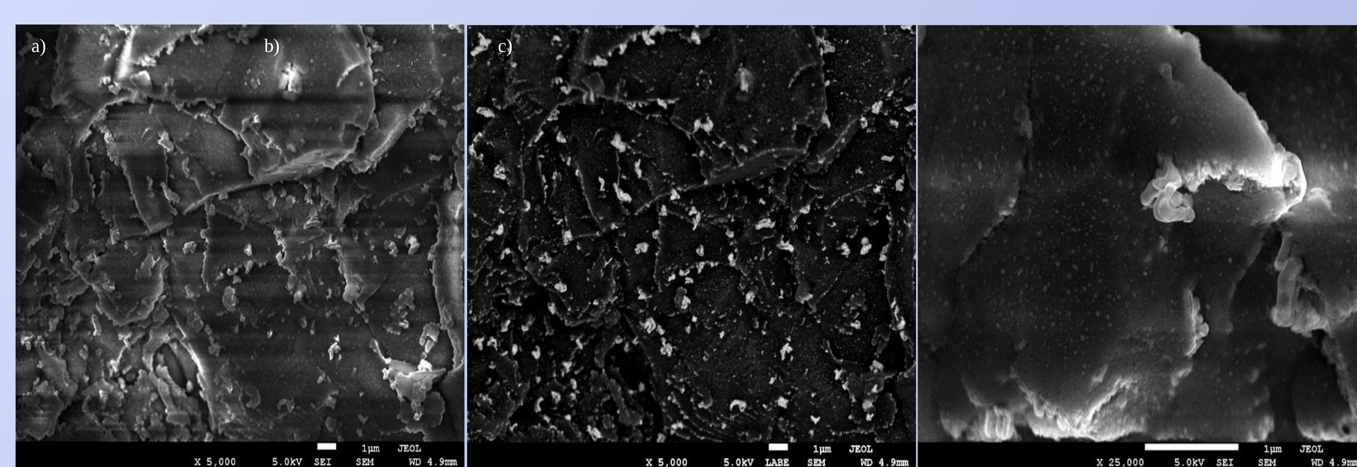
SEM Characterization



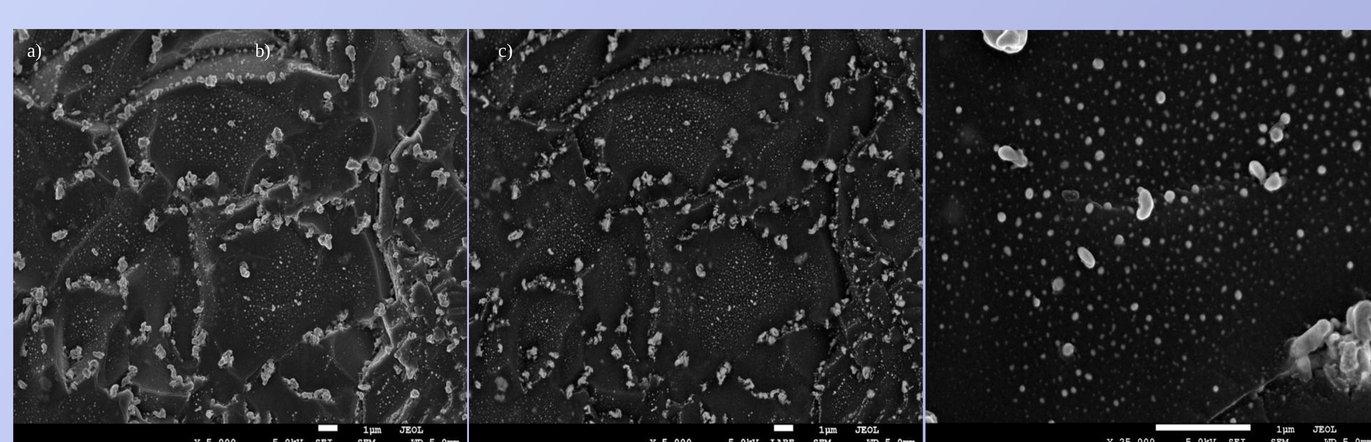
SEM images of the initial film C from SE detector with magnifications:
a) x 25 000
b) x 50000.



SEM images of the film C1 from a) SE detector, b) LABE detector and c) SE detector (a and b with x 5 000 magnification, c with x 25 000 magnification)



SEM images of the film C2 from a) SE detector, b) LABE detector and c) SE detector (a and b with x 5 000 magnification, c with x 25 000 magnification)



SEM images of the film C3 from a) SE detector, b) LABE detector and c) SE detector (a and b with x 5000 magnification, c with x 25 000 magnification).

RESULTS

Lattice constant of Pd crystallites, obtained from diffraction patterns is similar: *a* = 0.3900 nm and is slightly higher than for the bulk Pd structure. The average size of Pd nanocrystallites determined by the Scherrer formula are 34 nm, 32 nm and 31 nm for A1, B1 and C1 films respectively. In a case of films C(1-3) the average size of Pd nanograins increases with increasing the annealing temperature and is 31 nm, 32 nm and 54 nm respectively. It should be noted that the crystallite size is not equivalent to the dimensions of nanograins observed in SEM images. Grains often consist of many nanocrystallites.

CONCLUSION

C-Pd films especially these after heat treatment in an inert atmosphere can be used as active layers in hydrogen sensors. On the annealed films' surface big Pd nanograins appeared and carbon matrix was also partly changed into porous material. Such surface changes are responsible for faster response to hydrogen annealed films compared to films not heat treated.

In the future authors plane to perform *in-situ* diffraction measurements with different H_2 content of gas atmosphere.

This project is co-financed by the European Regional Development Fund within the Innovative Economy Operational Programme 2007-2013 (title of the project "Development of technology for a new generation of the hydrogen and hydrogen compounds sensor for applications in over normative conditions" No UDA-POIG.01.03.01-14-071/08-07) as wall as from COST Action ((number 577/N-COST/20009/0).

INNOVATIVE ECONOMY
NATIONAL COHESION STRATEGY
EUROPEAN UNION
EUROPEAN REGIONAL DEVELOPMENT FUND