THE CROSS SECTIONAL INVESTIGATIONS OF POROUS CARBON FILMS CONTAINING PALLADIUM NANOCRYSTALLITES

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INTRODUCTION

The problem of detection hydrogen and also hydrocarbons with sufficient sensitivity is very important. The future of aerospace, automobile and energy sectors will revolve around hydrogen fuel. It becomes really important to control and monitor these gases, as there is a huge risk of damage to property and loss of human health if a leak occurs. Certain gases can be toxic for humans, corrosive and also explosive. So we need sensors that can continuously and effectively detect the gases. In Tele - Radio Research Institute such films, containing palladium nanograin were synthesized and can be applied as active layer in hydrogen detector.

SAMPLE PRODUCTION

In PVD method multiphase carbonaceous Pd films were deposited on ceramic substrates under the dynamic pressure of 10-5 mbar. Two separated sources were used: one containing fullerene C60 powder (99,9%) and second with palladium acetate Pd(C2H3O2)2. During the synthesis process the temperature of the substrates was ~ 100°C and growing time was 8 min. The film originating from the PVD process was modified in the CVD method due to temperature and xenon decomposition over the film surface. Structure containing Pd nanocrystals was obtained. More information about this process will be published in the near future. The PVD film is presented on Fig.1 and the PVD/CVD film on Fig.2.

SAMPLE PREPARATION

For TEM research, we have prepared cross-sections specimens (lamella) of both samples obtained after PVD and PVD/CVD process using the Form Ion Beam (FIB) from Helios NanoLab 600i. First, the film surface has been protected by the platinum layer. Then the protected layer was cut out using the gallium ions beam. This method was not destructive for the studied samples. Finally, the lamella was sputtered to the standard Omniprobe Lift-Out grids. View of both lamella are presented on Fig.3 PVD and Fig.4 PVD/CVD films. Dimensional individual layers of the lamella.

RESULTS

The carbonaceous films obtained in the PVD and PVD/CVD process were studied by TEM, using TITAN 60-300 and Jeol 2100 EX. Sample from PVD process (Fig.5) showed the existence of large crystallites of palladium (4-20 nm in diameter) which are surrounded by a layer of amorphous carbon containing small Pd crystallites (1-3 nm). In sample PVD/CVD we observed that the particles of the Pd are larger than (Fig.6) some reach the size of 300 nm. Graphitization occurred in the walls of Pd (Fig.7), which is the cause of decomposition of amorphous carbon surrounding the Pd particles. The difference between layer obtained in PVD/CVD is the existence of a carbon foam layer covering the layer formed by PVD (Fig.7). Pd particles have a polycrystalline structure (Fig.8). Are observed, as in the PVD samples, clusters of small nanocrystals of Pd (Fig.8).

CONCLUSIONS

Traditional sample preparation method using epoxy glue and ion milling in the case of such thin films is practically impossible. The use of Helios allows the production cross section while minimizing damage beyond the sample. This method allows for accurate measurement of thickness of layer occurring in the sample.

Acknowledgements: This research was cofinanced by the European Regional Development Fund within the Innovative Economy Operational Programme 2007-2013 (Development of technology for a new generation of the hydrogen and hydrogen compounds sensor for applications in above normative condition). No UDA-POIG. 01.03.01-14-07(68-60).