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INVESTIGATION OF CARBONACEOUS - PALLADIUM COMPOSITIES BY TEM AND CL METHODS

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INTRODUCTION

The problem of the hydrogen molecules detection 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 human lives if a leak occurs. Certain gases can be toxic for humans, or corrosive gases or else explosive. It is a need of sensors that can effectively detect this type of gases. In Tele and Radio Research Institute carbonaceous films, containing palladium nanograins were synthesized by PVD/CVD method. These films can be applied as active layer in hydrogen and hydrocarbons detector.

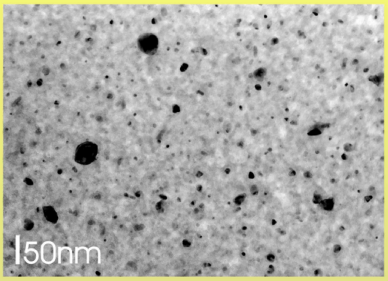


Fig.1. TEM image of the CVD sample.

EXPERIMENTAL

In PVD method multiphase carbonaceous nano-Pd films were deposited on ceramic substrates under the dynamic pressure of 10^{-5} mbar. Two separated sources were used: one containing fullerene C_{60} powder (99,9%) and second with palladium acetate $Pd(C_2H_3O_2)_2$. Current parameters in resistance heater were 2,1 A and 1,2 A for C_{60} and palladium acetate sources respectively. The distance *d* between sources and substrate was 69 mm and deposition time *t* was 8 min. This film was next modified in CVD method with a temperature (650°C) and xylene decomposition over the film surface. Finally, carbonaceous porous films with foam-like structure containing Pd nanograins were obtained. More information about these processes can be found in [1] and [2]. TEM image of the CVD film is presented in Fig.1.

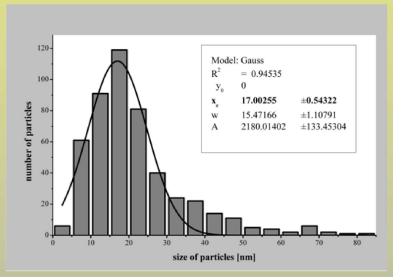


Fig.2. Histogram of the size distribution of the Pd nanoparticles.

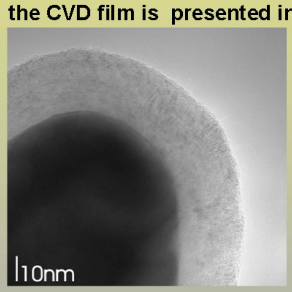
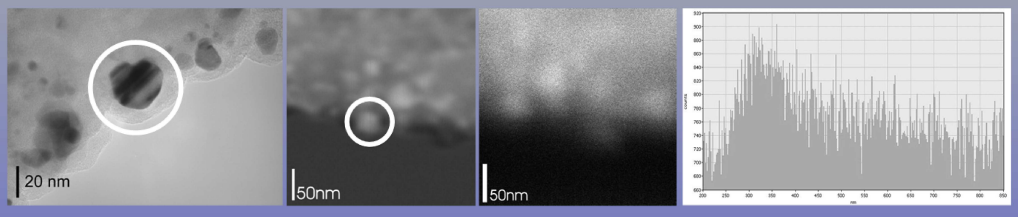


Fig.3. Pd particle with graphite shell

RESULTS

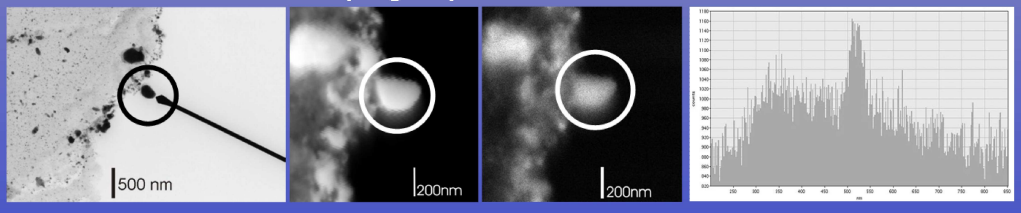
Carbonaceous porous films obtained after CVD process containing Pd nanocrystalites were studied by TEM and CL. The size distribution of Pd nanocrystallites in CVD film is presented in Fig.2. Our observations shows that big (more than 40 nm) nanoparticles have a graphite shell. The shells thickness dependents on dimensions of Pd nanograin and grows with the size of Pd nanograins. The shell around the individual Pd nanograin are shown in Fig.3. We carried out connected examinations by TEM and CL methods. For our research we chose two types of particles: with and without a graphite shells. The particles were marked after TEM investigation and submitted on to CL for further research. We noticed, that particle with shells were optically active. In Fig.4. we present the series of images of naked Pd particle. In the first TEM image the particle chosen to further investigations is shown. The second picture shows the same particle in electron scanning microscopy, next, image in CL and the last one – spectrum collected from that particle are presented. There was not observed any significant optical activity in this case. Similar studies of Pd particle with graphite shell are shown in Fig.5. In this investigation we observed single particle optically active. In CL spectrum strong peak at 525 nm was found.

Naked Pd particle (Fig.4.)



TEM SEM CL Spectrum CL

Particle with shell (Fig.5.)



TEM SEM CL Spectrum CL

PLANS FOR THE FUTURE

The physical effects of cathodoluminescence from Pd nanograins with shells are not well know. It seems that Pd nanograins need full graphite shells to be optically active. Further, we want to check whether the energy of emission depends on the size of the Pd nanograins and the thickness of shells. It will be helpful to get to know the phenomena of luminescence from such nanoobjects. We could explain observed phenomena with a surface plasmons (SPs).

CONCLUSIONS

TEM and CL investigations of the single nanoparticles showed an interesting phenomenon, which is an optical activity of nanograins with a graphite shell. Energy of CL emitted light is 2,36 eV (green light region).

[1] E. Kowalska, E. Czerwosz, J. Radomska „Metoda syntezy nanoporowatych materiałów węglowo - palladowych" Elektronika 1/2009
[2] E. Czerwosz, E. Kowalska, J. Radomska, H. Wronka Sposób otrzymywania nanopianki węglowej zawierającej nanokrystality metalu" zgłoszenie patentowe nr P384591 z dnia 03.03.2008r.

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