

TEM AND CL INVESTIGATIONS OF Pd NANOGRAINS INCLUDED IN CARBONACEOUS FILM

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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. So we need sensors that can continuously and effectively detect gases. In Tele - & Radio Research Institute such films, containing palladium nanograins were synthesized and can be applied as active layer in hydrogen detector.

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 $Pd(C_2H_3O_2)_2$. During the synthesis process the temperature of the substrates was $\sim 100^\circ C$ and growing time was 8 min. The film originating from the PVD process was modified in the CVD method due to temperature and xylene decomposition over the film surface. Finally a carbon nanoporous film like foam structure containing Pd nanograins was obtained. More information about this process [1] [2]. The CVD film are presented in Fig.1.

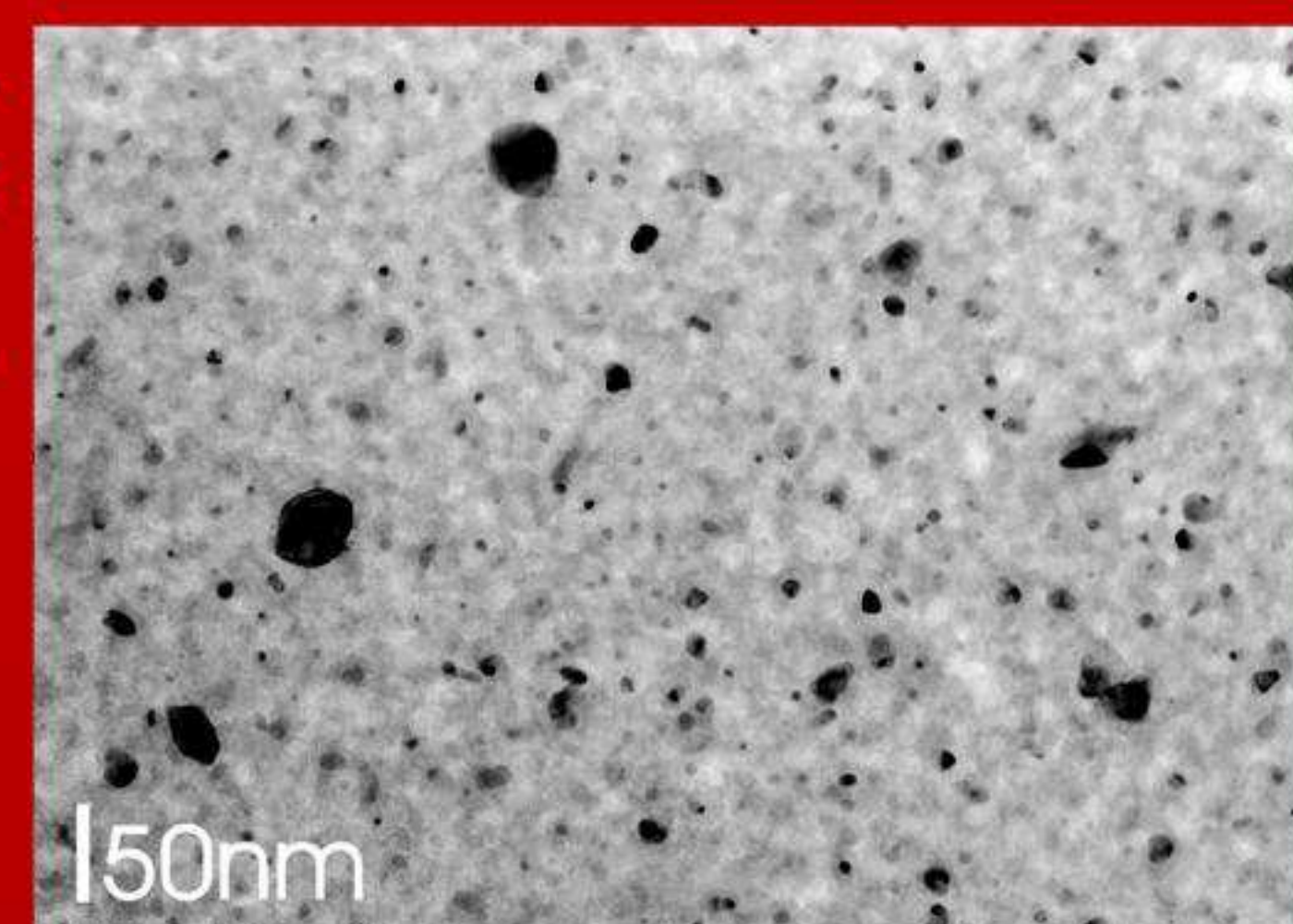


Fig.1. TEM image of the CVD sample.

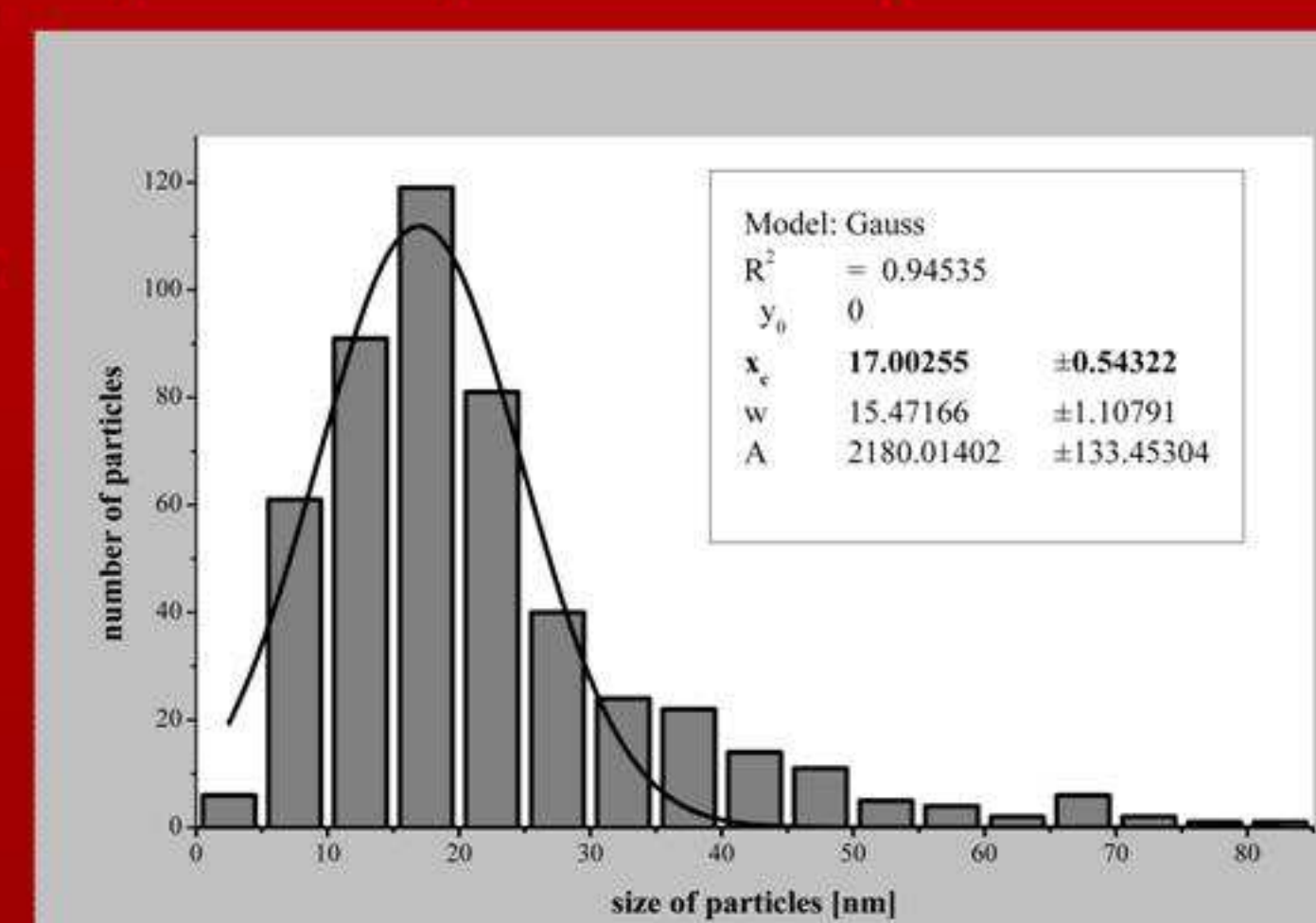
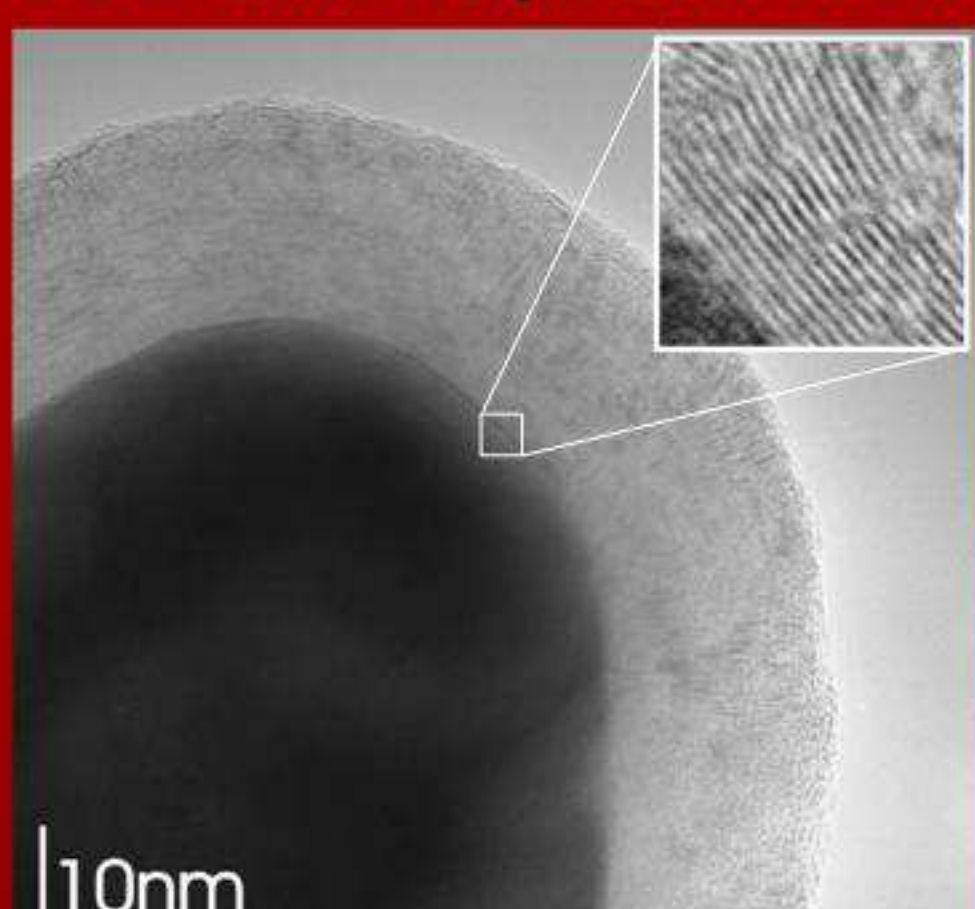


Fig.2. Histogram of the size distribution of the Pd nanoparticles.



RESULTS

Carbonaceous films obtained in the CVD process and containing Pd nanocrystallites were studied by TEM and CL. The size distribution of Pd nanocrystallites for CVD film is presented in Fig.2. Our observations showed that big (more than 40 nm) nanoparticles had a graphite shell. The shell thickness dependent on dimensions of the Pd nanograin and grow with the size of the Pd nanograins. The shell on Pd nanograin is showed in Fig.3. and on on the fig 4. selected area electron diffraction pattern from this structure with a marked ring of graphite. We carried out connected examinations by TEM and CL methods. For our research we chose two kind particles: with and without a graphite shell. The particles were marked after TEM investigation and passed on to CL for further research. Noted that particle with shell were optically active. In Fig.5. we presents a series of images for a naked particle. On the first image there is shown a TEM image with a particle that was chosen for further investigations. Second picture – the same particle in scanning microscopy, next, image in CL and the last one – spectrum collected from that particle. We do not observe any significant optical activity. In the second case we investigate a particle with a graphite shell. In Fig.6. we see images in a similar situation from Fig.5. This time a single particle was optically active – image in CL with strong visible particle and we observed strong peaks in CL spectrum at 525 nm.

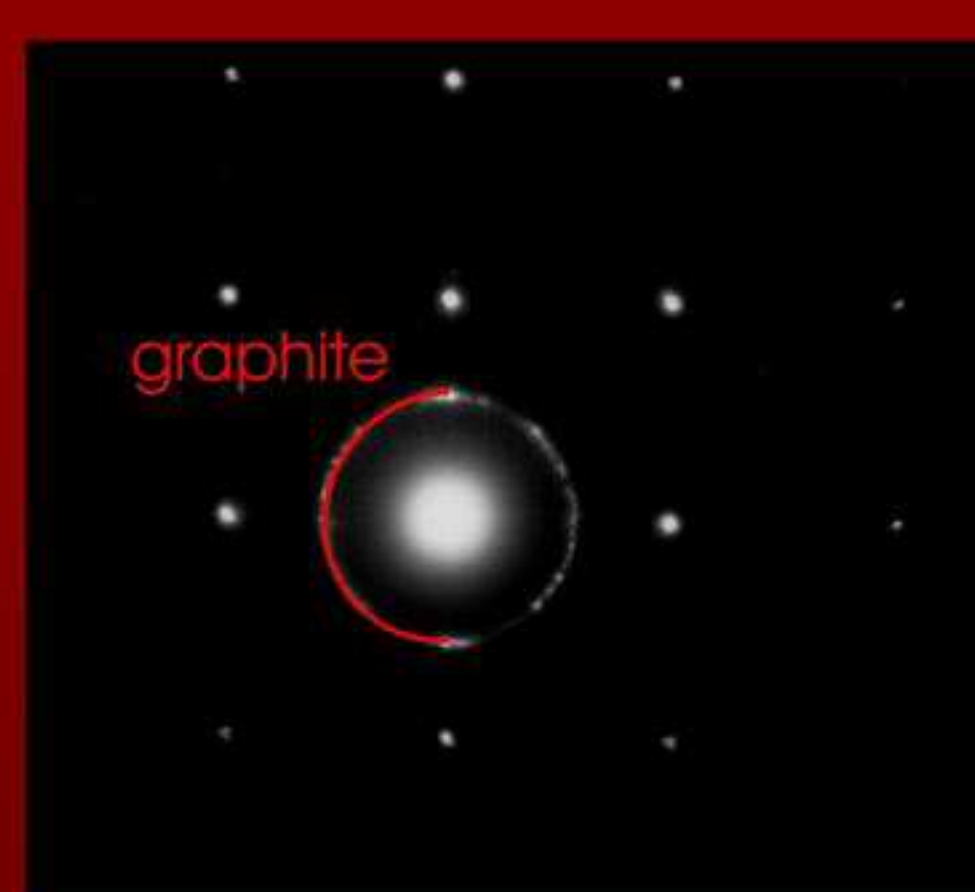
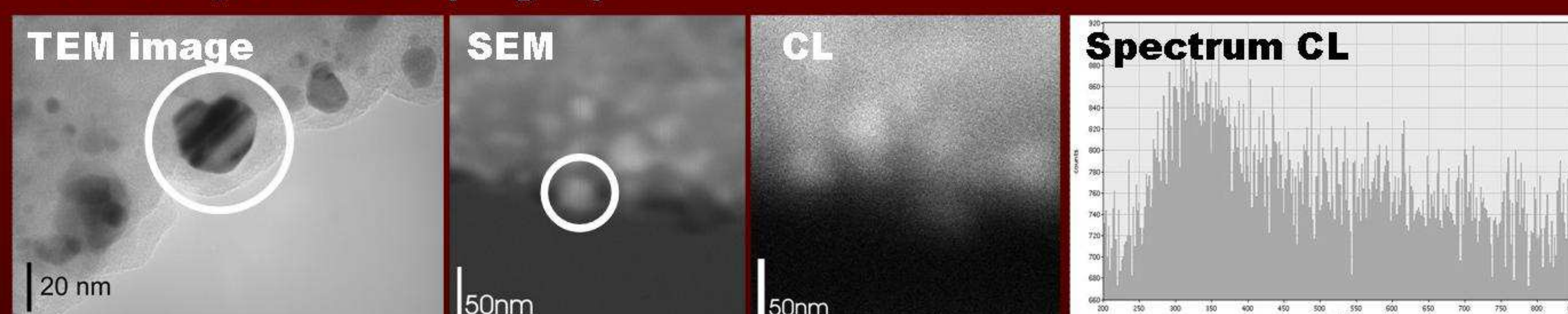


Fig.4. SAED from Pd particle with shell

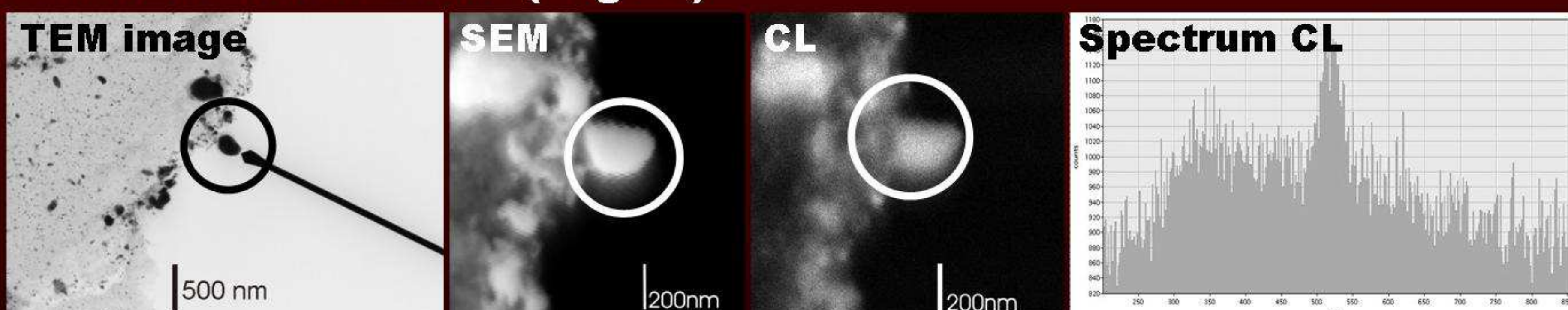
PLANS FOR THE FUTURE

The physical effects of lighting Pd nanograins with a shell are not well known. It seems that Pd nanograins need a full graphite shell to be active optically. In the future we want to check whether the energy of emission depends on the size of the Pd nanograins and the thickness of the shells. It will be helpful to get to know the phenomena of lighting. Probably surface plasmons (SPR) are the best mechanism to explain such phenomena.

Naked particle (Fig.5)



Particle with shell (Fig. 6)



CONCLUSIONS

TEM and CL connected investigation of the single nanoparticles showed an interesting phenomenon in which optical activity is only observed in nanograins with a graphite shell. Energy emission is 2.36 eV and it is part of the visible light spectrum (green). So far in this range, spectra were observed only for nanorods and Ag nanospheres [3]. Spectral range for particles with shells (so far) ends on a value of 590 nm.

[1] E. Kowalska, E. Czerwosz, J. Radomska „METODA SYNTEZY NANOPOROWATYCH MATERIAŁÓW WĘGLOWO - PALLADOWYCH” Elektronika 1/2009

[2] Sposób otrzymywania nanopianki węglowej zawierającej nanokrystaliny metalu E. Czerwosz, E. Kowalska, J. Radomska, H. Wronka zgłoszenie patentowe nr P384591 z dnia 03.03.2008r.

[3] S. Lal et al. Nature Photonics 1 2007 641-648