Influence of technological process temperature on the structure of C-Pd nanocomposite thin films

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The technology of obtaining C-Pd thin films by a two-phase method (PVD/CVD) was developed in the Tele & Radio Research Institute in Warsaw (Poland). Details of technological process are described in [1 – 3]. Depending on the CVD process temperature the form and structure of film changes, especially taking into account carbonaceous matrix that becomes porous for temperatures higher than 600° C, while palladium nanograins embedded in such matrix remain similar (of size of few tens up too few hundred nm). Typical form of such porous carbonaceous matrix in which Pd nanograins are placed is shown in Fig.1.

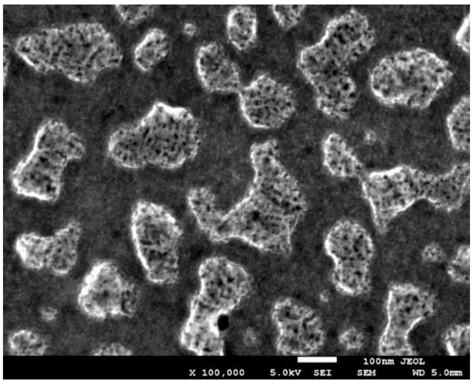

Fig.1. SEM image of porous carbonaceous matrix (for CVD process temperature of 650° C)

The work presents results of Raman spectroscopy (RS) and transmission spectroscopy studies of C-Pd films, obtained at different temperatures of the CVD process. This temperature was changed between 500 and 750° C.

The changes of molecular structure of films, prepared by two steps method with temperature of second CVD process changing from 500 to 750° C , was studied using Raman spectroscopy (Nicolet Almega XR spectrometer, excited with 532nm line). The spectra were recorded in the range from 100 to 4000cm^{-1}. The beam power (0.25mW), was focused in the area of 1µm x 1µm using microscopic lens x50. The transmission spectra measurement was carried out by means of Cary 5000 dispersion spectrophotometer in the range from 200 – 3200nm, with the resolution of 1nm. Surface images for samples were taken using an optical microscope (Olympus B51) with x10 and x 50 lens.

The film topography analysis, based on the microscopic images of samples obtained at various temperatures, confirms that the film surface changes. Grains with various diameters on the surface were observed. In the case of optical microscopical observation no influence of technological process temperature on changes of grains' sizes was observed (Fig. 2).

a) A1 b) A2 c) A3

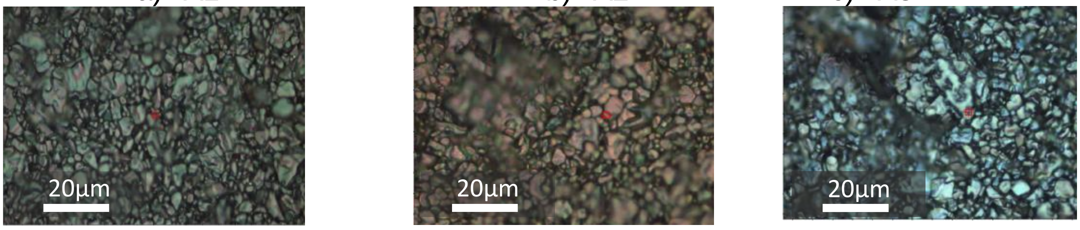

Fig.2. Microscopic images of samples' surface:: a) A1 (T_2 =500°C), b) A1 (T_2 =600°C), c) A3(T_2 =750°C)

Raman spectra for all the samples, taking into consideration the modified process parameter, are presented in Fig. 3.

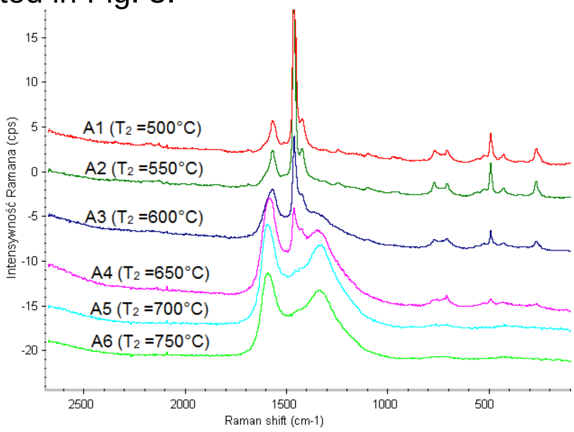

Fig. 3. Raman spectra of C-Pd films obtained in CVD process (various temperatures T_2).

In the spectra of samples obtained at lower process temperatures (500, 550 and 600^{o}C), characteristic C_{60} fullerene spectra can be found [4]. In the spectrum of the sample obtained in 600^{o}C the additional G and D bandwidths are observed, characteristic for amorphous graphite [5]. This can prove existence of both fullerene and amorphous carbon in the carbonaceous matrix.

In the spectra of samples obtained at higher temperatures of CVD process (> 600^{o}C), bandwidths with peaks at about 1350 cm^{-1} and 1585 cm^{-1} are observed. These bandwidths are identified to be D and G.

The measurements carried out reveal that high process temperature influences on the type of carbon phase in the samples. The increase of temperature (T_2) facilitates degradation of C_{60} fullerene.

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References

1.) E. Kowalska, E. Czerwosz, J. Radomska: Metoda syntezy nanoporowatych materiałów węglowo-palladowych. Elektronika 1, 32-35 (2009)
2.) E. Czerwosz, E. Kowalska, H. Wronka, J. Radomska, Patent Notification 2008 nr P384 591
3.) E. Czerwosz, R. Diduszko, P. Dłużewski P, J. Keczkowska, M. Suchanska, Vacuum; 82:372 (2008)
4.) H. Kuzmany, R. Pfeiffer, M. Hulman, Ch. Kramberger; Phil. Trans. R. Soc. Lond. A 362 2375 -2406 (2004)
5.) A. C. Ferrari, J. Robertson; Physical Review B vol. 61, 14095–14107 (2000)