Analele Universităţii de Vest din Timişoara
Vol. XLIX, 2006
Seria Fizică

CHARACTERIZATION BY TEM OF THE NANOCARBON STRUCTURES OBTAINED BY PULSED HIGH VOLTAGE DISCHARGE IN FLOWING METHANE GAS

R.Vladoiu[1], V. Ciupina[1], G. Prodan[1], N. Ekem[2], S. Pat[2], V. Dinca[1], G. Musa[1]

[1] *Department of Physics, Ovidius University, Constanța, Romania*
[2] *Osmangazi University, Physics Department, 26480, Eskisehir, Turkey*

Abstract
In this paper, we present the latest results concerning the characterization of the nanocarbon structures obtained by TEM. The growths of amorphous carbons were performed at atmospheric pressure using a high voltage pulsed power supply of 25 kV and 25 kHz, under the methane flux. The pulsed discharge of methane gas was generated between two sharp wolfram electrodes. Two types of carbon were produced by this pulsed plasma: soft carbon and hard carbon. Carbon samples were investigated also by Scanning Electron Microscopy.
Key words: Methane plasma, pulsed discharge, carbon partial graphitize

1. Introduction

Nanocarbon structures represent new type materials with unique mechanical and electrical properties [1]. Among the popular applications, one can mention hydrogen storage, the reinforcement of composites with ceramic and polymer matrices, the development of specialized lamps and flat panels, etc [2-5]. Nanocarbon structures generally are produced by several techniques like Chemical Vapor Deposition (CVD), laser ablation, electric arc methods [6-8].

The aim of this paper is to produce carbon from technical methane gas by using atmospheric pulsed plasma. The conversion of the atmospheric pulsed plasma methane to carbon presented in this paper is very cheap and simple because no any vacuum equipment is necessary. In addition, this method can be used for carbon deposition to any substrates. In this paper, carbon structures were growth on the sharpened wolfram wires and inner surface of the glass tubing surrounding tungsten electrodes.

2. Experimental arrangement

The experimental device consists from a glass tubing (around 22 mm diameter), which has at both ends as electrodes electro polished and sharpened tungsten wires, the diameter of wires being of 1,5 mm. The methane gas can flow at atmospheric pressure through the discharge tube with a flux of 0,3 l/min. A pulsed high voltage of 25 kV was applied between electrodes, the discharge pulses having a frequency of the order of 25 KHz. In series with discharge device is included also a resistor to adjust the voltage pulses.

At the applied discharge pulses, the methane gas pressure decrease and the gas supply must be adjusted (i.e., increased) in order to keep the working parameters constant. The plasma of methane gas generates mainly carbon. In the same time acetylene and other hydrocarbons can be observed too. In the discharge device (inside of the glass tube), a deposition of carbon can be noticed. The analyses have shown the presence of pure carbon.

The tungsten electrodes are sharpened (each of them) to one of the edges, the distance between them was 8 mm. These electrodes are mounted axially as it is shown in fig.1. At the each edges of the glass tubing are mounted insulated discs to close the volume of the discharge device and also to sustain the electrodes on the axis of the glass tubing.

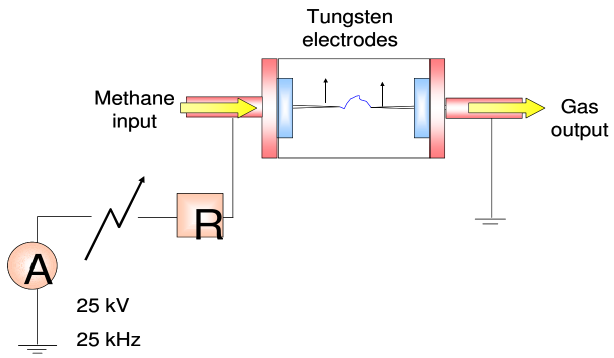

Figure1. *Schematic diagram of the experimental system*

Two small diameter tubing's from glass ($\phi = 4-5mm$) are mounted at the edges of the pulsed discharged device in order to sustain the passage of methane through the discharge space. Two type of sharpened electrodes have been used namely with or without sealing a dielectric material just on sharpened edge of the electrodes. On the right electrode, two different carbon samples (soft carbon and hard carbon) were growth. Hard carbon was growth at the end of the sharpened wolfram electrodes, but in this case, to take this deposited carbon was not so easy. There exists a significant tendency for carbon to condense in globular form, especially in the case of using dielectric electrode. Also, the carbon produced with dielectric electrode presents colonized feature

3. Experimental Results

The photo image and SEM image of the obtained soft carbon on the sharpened wolfram electrodes is shown in figures 2 and 3.

Figure 2. *Photo image of obtained soft carbon*

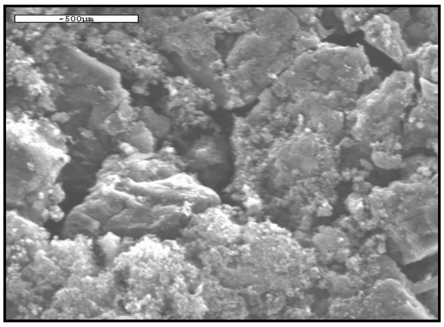

Figure 3. *SEM image of obtained soft carbon*

Hard carbon structures were collected from the centre of the soft carbon structured. The mass (m) of the obtained hard carbon was 0.00207 g/min and the length was about 2 mm. It can be noticed a clear nanoparticle shape of onion-like-carbon and a beginning of film formation.

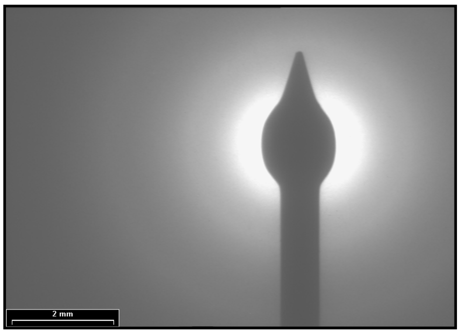

Figure 4. *SAED (Selected Area Electron Diffraction) for soft carbon*

The TEM images of the soft carbon obtained are presented in figure 5.

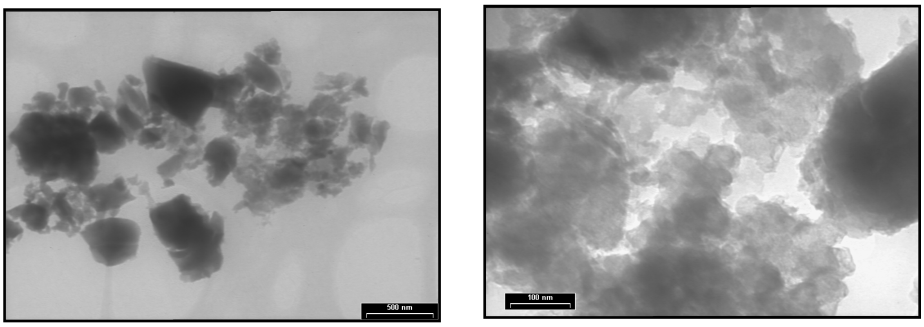

Figure 5. *TEM images of the soft carbon at different scales.*

The TEM images of the hard carbon partial graphitized are presented in figure 6 and SEM images in figure 7. We investigated the hard carbon samples by Scanning Electron Microscopy

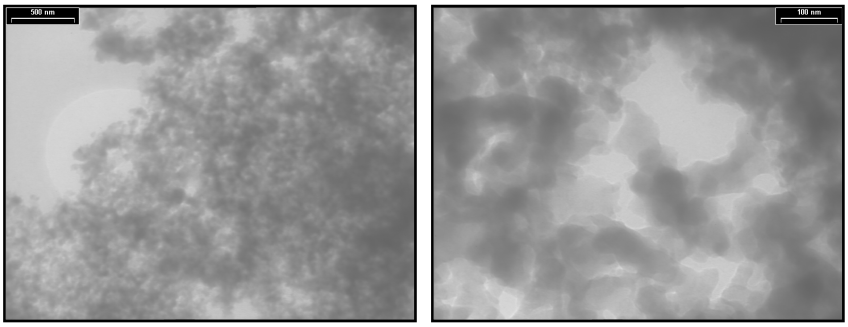

Figure 6. *TEM images of hard carbon at different scales*

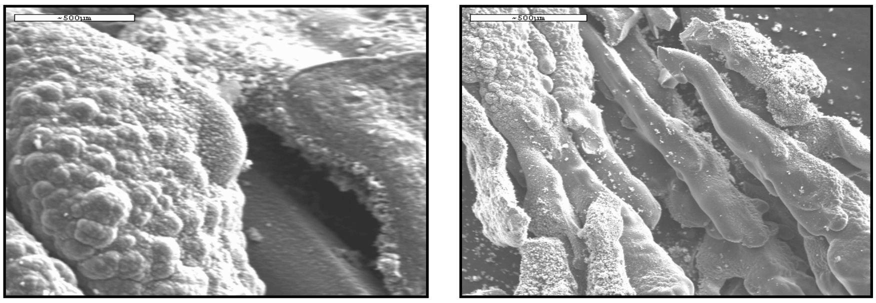

Figure 7. *SEM images of the obtained hard carbon*

4. Conclusions

Characterization of the obtained DLC thin films has been made by Transmission Electron Microscope (TEM) with a magnification of 1, 4 M and a resolution of 1,4 Å. Carbon samples were investigated by Scanning Electron Microscopy, Energy Dispersive X – ray spectroscopy and X-ray diffraction. All two forms of produced carbon have amorphous structure according to XRD results and without contamination according to EDX results

References

[1] Pacheco, M., Pacheco,J., Valdivia,M., Bernal,L., Valdivia R., Huczko A., Lange H., Cruz A., Lopez-Callejas R. Braz.J.Physc., Dec.2004, Vol.34, no.4b, p.1684-1688.

[2] L. Allemand, La Recherche **332**, 55 (2000).

[3] A. Chatelain, J. M Bonnard, Le Temps stratégique 81, Genève, (mai-juin 1998).

[4] P. Collins, P. Avouris, Pour la Science **280**, 76 (2001).

[5] P. Poncharal et al. Science, **283**, 1513 (1999). cité á La recherche **320**, 19 (1999)

[6] T. Guo, P. Nikolaev, A. Rinzler, D. Colbert, R. Smalley, J. Phys. Chem. **99**, 10694(1995).

[7] C. Journet, W. K. Maser, P. Bernier, A. Loiseau M. Lamy de la Chapelle, S. Lefrant, P. Deniard, R. Lee, and J. E. Fisher, Nature, **388**, 756 (1997).

[8] G. Musa, N. Ekem, T. Akan, S. Pat, U.G. Issever, M.Z. Balbag, M.I.Cenik, O. Ozen, E. Killi, 6th Int. Balkan Workshop on Applied Physics, p.106, Constanta, Romania(2005)