STRUCTURAL CHARACTERISTICS OF DIFFERENT TYPES OF NANOCARBON MATERIALS

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Introduction

Creation of novel functional composite materials based on nanocarbon is one of the directions of modern nanotechnology. One of more perspective nanocarbon materials is carbon nanotubes. As known while the nanotubes are obtained by the arc discharge method, the produced on the electrodes surface deposition contains the besides nanotubes a lot of impurities including as metal particles such particles of amorphous carbon and nanographite. That is why attestation and establishment of structural characteristics of nanocarbon materials obtained by different methods just as the development of purification methods and investigation of influence of these methods on the structure of nanocarbon materials are urgent tasks.
The paper presents the results of structural characteristics investigations of obtained by arc discharge method nanocarbon material and the changes of structural parameters of these materials during purification process.

Experimental

As initial material carbon deposition obtained by arc discharge method on the surface of anode (Sample #1), on the top of cathode (Sample #2) and around of cathode (Sample #3) was used. For purification of raw carbon deposition the following scheme was proposed [1]:

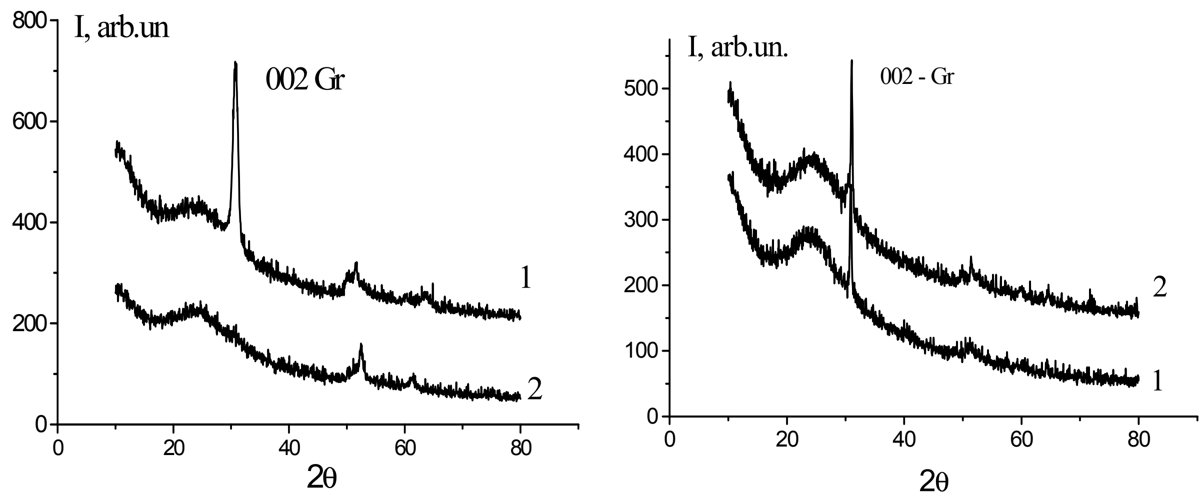
1. Heating of raw material in nitric acid solution (2.5 M) for removing catalyst particles.
2. Sequential material washing with distilled water, caustic soda solution (pH 10-11) and ethanol.
3. Vacuum dehydration at temperature T = 110-1200C during 5.5 hours. This stage is necessary for partial nanotubes structure recovery, which was possible damaged during acid treatment of nanocarbon material.
4. Open air oxidation at T = 5500C during 30 minutes for removing amorphous carbon particles.

The structural investigations of obtained materials were made with diffractometer DRON- 4 07 in filtered Co Kα radiation and by method of SEM (JSM–840, JEOL, Japan) and TEM (EMB-100AK, Ukraine).

Results and Discussion

Obtained raw samples from electrode surface are the dark-gray powders. X-ray phase analysis showed presence in all samples catalyst particles – iron, nickel and cobalt, the largest content of catalyst is in the sample #3, and contain of catalyst in samples #1 and #2 is smaller.

The Fig.1 presents diffractograms for raw and purified in according to above scheme samples #2 and #3. As it is shown from the figures for both raw samples (curves 1) besides lines of catalyst lines corresponding to 002 reflexes of graphite are observed. The angle positions of these lines correspond to the interlayer distances d002 = 3.38A for sample #2 and d002 = 3.36A for sample #3. These interlayer distances can be formed both nanographite and multi-walled carbon nanotubes. After purification 002-line of graphite in the sample from cathode surface is remained, although it becomes asymmetrical. For sample obtained from the top of cathode, lines corresponded to reflexes from graphite layers are disappeared. It can be caused by effect of acid treatment on the initial material that makes to formation of fine amorphous phase or single nanographite shifts.



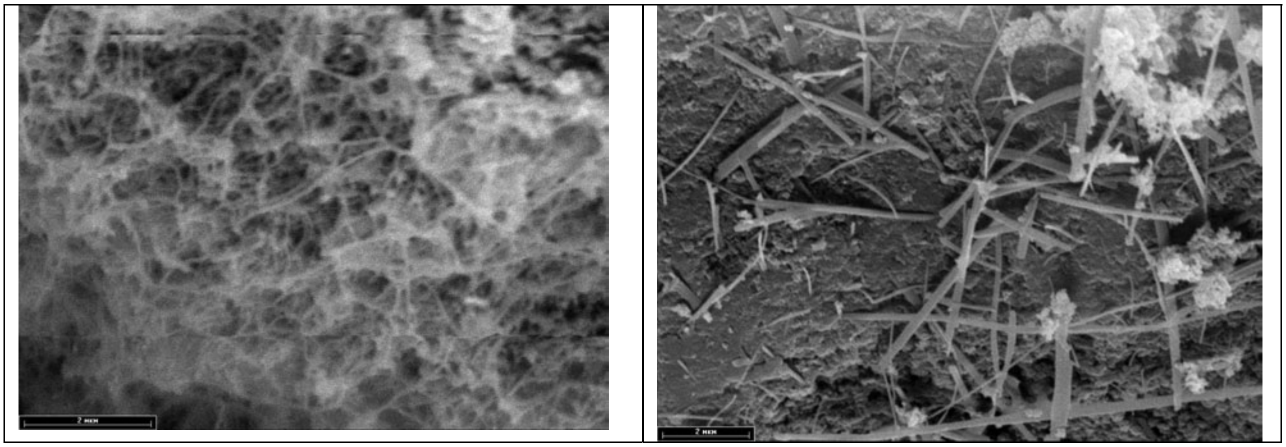
a) b)

Fig.1. Diffractograms of the samples obtained from different regions of cathode surface: a) from the top of cathode; b) from the surface of cathode: 1- the raw samples; 2 – purified samples.

For more careful structure analyses of obtained materials the investigations of samples particles surface were carried out by method of SEM and TEM. The results of investigations are shown on the Figs. 2 –4.

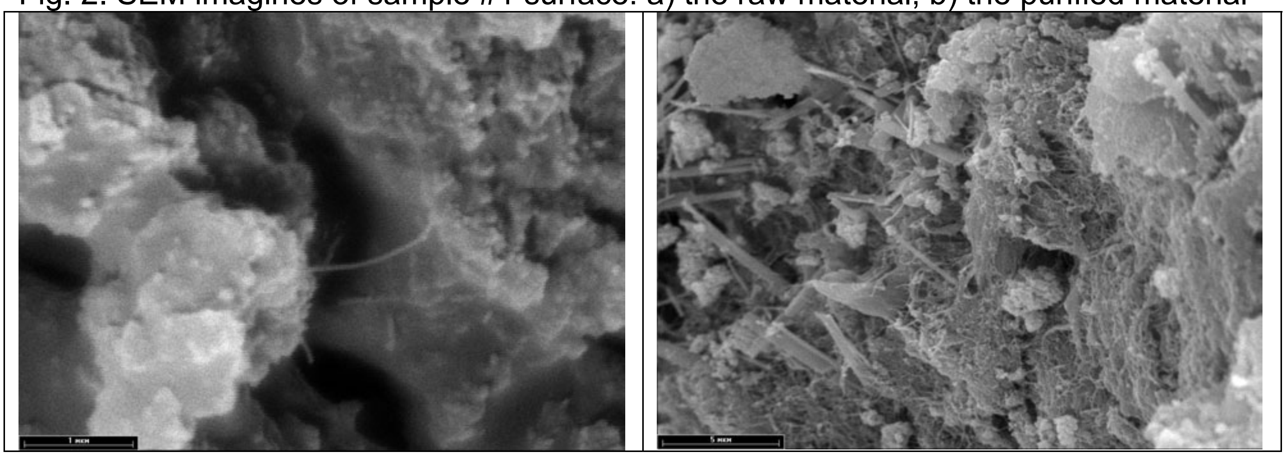
As it is shown from the figures the surface structures of samples obtained from different parts of electrodes are quite different. In the sample #1 it is neatly showing the filiformed structures, which are the bundles of carbon nanotubes. The diameter of these nanotubes is in the range from 15nm to 60 nm, that is these nanotubes are multi-walled nanotubes. Besides with multi-walled nanotubes in sample #1 the nanocarbon particles and the finest particles of catalyst are revealed.

Sample #2 is solid carbon mass with ingrained in it catalyst particles. At the back of carbon particles superfine “runners” are revealed. These “runners” are carbon nanotubes. The diameter of these nanotubes from data of SEM is up to 30 nm. Material obtained from the top of cathode is more “dirty” than material obtained from anode surface and nanotubes concentration is smaller.



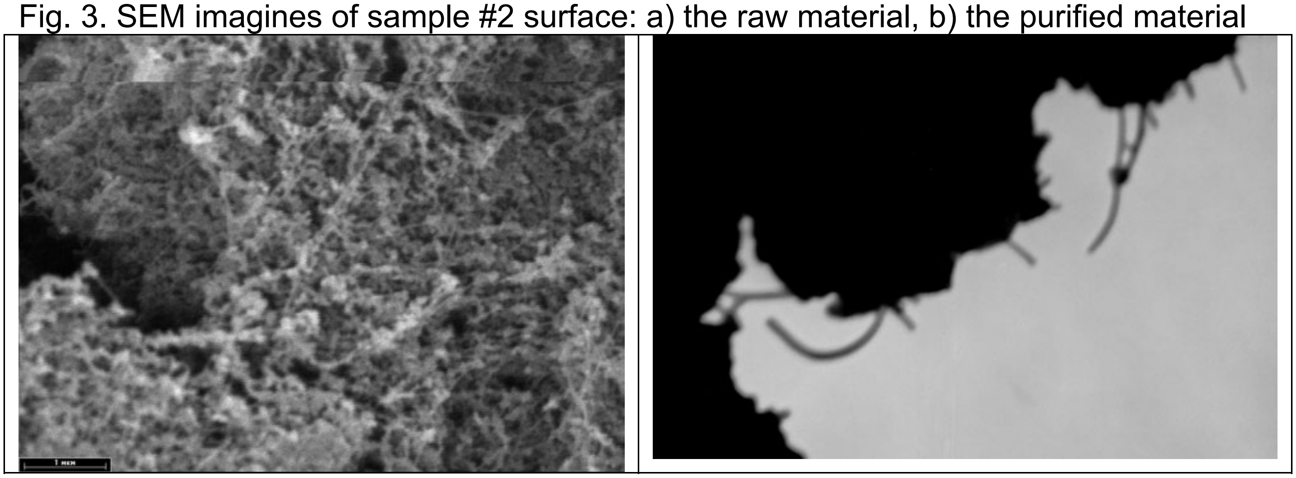
a) b)

Fig. 2. SEM imagines of sample #1 surface: a) the raw material, b) the purified material



a) b)

Fig. 3. SEM imagines of sample #2 surface: a) the raw material, b) the purified material



a) b) magnification $\times$ 120 000

Fig. 4. SEM (a) and TEM (b) images of sample #3.

Sample #3 is porous dendrite structure. The SEM investigations weren't revealed carbon nanotubes in this sample. There aren't also catalyst particles on the SEM imagines. However more precise structure analysis of sample #3 by method TEM (Fig. 4b) allows to reveal the typical "runners" with diameter up to 6 nm, which are evidently singlewall carbon nanotubes or multi-walled nanotubes with small number of carbon layers.

After purification in the sample #1 (Fig.2b) a lot of new structures are formed. These new formations are similar to "nanoscrolls". The average diameter of these "nanoscrolls" (up to 300 nm) is essentially greater than diameter of multi-walled nanotubes presenting in raw material. The sample #2 after purification (Fig. 3b) also presents single "nanoscrolls" with diameter up to 500nm and single graphite nanoshifts. Thus the purification essentially changes the structure and phase contains of raw material. The proposed purification technique results in destruction of single-walled nanotubes structure and formation of new "nanoscrolls" structures

Conclusions

Consequently, carried out investigations obtained by arc discharge method nanocarbon materials have revealed, that composite and structural characteristics of these materials are essentially determined by one's position: material obtained with anode surface contains the greatest number of multi-walled nanotubes and small number of nanocarbon particles. The materials formed at the top of cathode contain mainly carbon deposition with penetrating nanotubes. The material made on the surface of cathode is dendrite structure with single nanotubes with diameter up to 6 nm. The purification of raw nanocarbon material results in the change of content and structural characteristics of initial material. These changes are determined by such purification procedure as position of initial material on the electrode surface.

References

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