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Transmission electron microscopy analysis and electrical measurements of carbon thin films

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The paper performs studies on carbon thin films based on electron microscopy data and electrical measurements are reported. The samples were obtained by Thermionic Vacuum Arc (TVA) method. Techniques used to acquire information were BF-TEM (Bright Field Transmission Electron Microscopy), DF-TEM (Dark Field Transmission Electron Microscopy), HRTEM (High Resolution Transmission Electron Microscopy), SAED (Selected Area Electron Diffraction) and Radial Distribution Function (RDF).

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1. Introduction

Progress in the nanosciences during the past decade opened up new pathways for developing materials with enhanced electronic or other properties. Their potential applications are wide-spread, such semiconductor electronics, spintronics, photonic devices, sensors for molecular recognition in chemistry and biology to name a few.

Despite the tremendous progress in recent years, however, substantial challenges remain to be solved before many of these nanotechnologies can reach their anticipated potential. Hereby, one needs to recognize that further advancement inevitably relies on the availability of characterization methodologies that allow for optimization of materials design and processing as well as for gaining a fundamental understanding of underlying scientific phenomena and concepts. As an example of such materials characterization challenges, we have studies issues related to nano-scale granular materials.

Carbon materials are intensively studied for large application in many domains that include chemistry, engineering, medicine and not last, material science. The carbon nanostructures have advantage that can be very easily obtained with a large number of forms. We mention here the very well know form of carbon: graphite, diamond and amorphous carbon.

Starting from graphite we can model and obtain onion like carbon and carbon nanotubes by means of modifying deposition parameters in case of CVD/PVD (Chemical Vapor Deposition/ Physical Vapor Deposition) method or by changing the laser parameters in case of pyrolysis method [1-10]. The structure can be a start point to understand physical properties and physical phenomena that occurs in these materials.

2. Experimental

Carbon thin films were prepared by thermionic vacuum arc (TVA) method. Because this system can heat any material at relevant temperature it is one of the most adequate deposition technologies for evaporate and condensate high melting point materials. It has been already reported to be a very suitable method for deposition of high purity carbon thin films with nanostructured film synthesis [11].

Thermionic Vacuum Arc deposition method consists form an externally heated cathode surrounded by a Wehnelt cylinder. The vapors are obtained by heating the material with thermo electron generated by externally heated filament of a circular form placed above the anode. The anode used was a 2mm diameter graphite rod.

The cathode and the vacuum chamber are grounded so the carbon plasma has a potential against the chamber wall equal with the cathode potential fall. On the substrate are deposited, with the evaporated neutral atoms, the incident energetic ions.

The cathode can be mounted in various positions against the anode. The highest density of plasma vapors is obtained above the anode. Due to the potential differences between the plasma potential and the walls, the ions are accelerated in the chamber walls. Practically the deposition takes part in the vapors of the anode materials, the deposited films containing only the ions of this material and therefore the energy of ions could achieve values up to 500eV. In this way the carbon thin film is bombarded during its deposition by carbon ions with established value of energy.

Due to the high energy dispersed in the most volume plasma, the material is strongly dispersed and completely droplets free. The obtained thin films were very smooth

and in some experimental conditions had a nanoscale structure.

3. Results and discussion

Formvar coated cooper grid designed for TEM investigation are used as support for all samples. Samples are obtained by dispersion in alcohol ~10% vol. Electron micrographs are obtained using a Philips CM120ST transmission electron microscope working at 100kV. Digital micrographs are capture using a Gatan 673 CCD wide angle camera. Microscope is connected with PC using Analysis software that performs image acquisition and processing algorithm.

The application software developed for testing ideal carbon nanostructure is based on graphite crystal design. In this case of nano-carbon the model consists in large sphere formed by small bricks.

The medium resolution TEM image (fig.1) seems to indicate a rather even dispersion of carbon on silicon substrate used. Also the diffraction analysis show diamond structure, so the dark points from figure 1 could be diamonds grains. Right image show Fast Fourier Transformation (FFT) of the marked area in TEM micrograph.

Fig. 1. Select area for diffraction and check astigmatism using Live FFT option. Right image show FFT representation of marked area in TEM micrograph.

The SAED and HRTEM image are used to determine crystalline structure. The graphitic structure of carbon was used for indexing electron diffraction pattern as shown in Fig. 2.

Samples were indexed using hexagonal structure with lattice parameters a=0.206nm and c=0.111nm.

Diffraction data and HRTEM images provide information about carbon structure. In first case we can determine the structure of material and dimension of crystalline region by means of Scherrer relation. Determined dimensions from diffraction data could be compared with those evaluated from electron micrographs. Reading such information we can further develop models of structure that can be applied to calculate other physical or chemical properties of material.

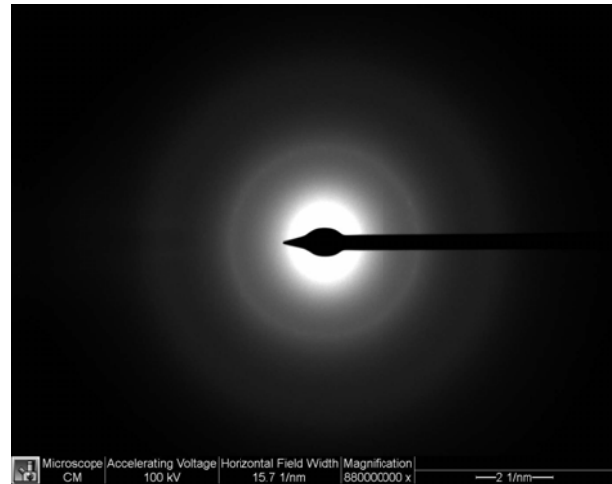


Fig. 2. Electron diffraction pattern obtained for carbon thin film growth on silicon substrate

Fig. 2 displays an electron diffraction pattern obtained with inserted Selected Area aperture and switch to conventional diffraction mode. Spot size 900nm, camera length 880mm, High voltage 100kV (λ=0.0037nm).

Radial Distribution Functions (RDF) presented in Fig. 3 are extracted from Selected Area Electron Diffraction pattern (figure 2) using CRISP2 software package, with ELD for arc and rings.

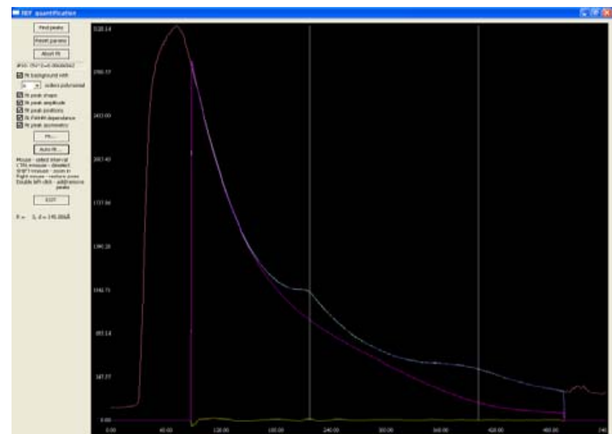


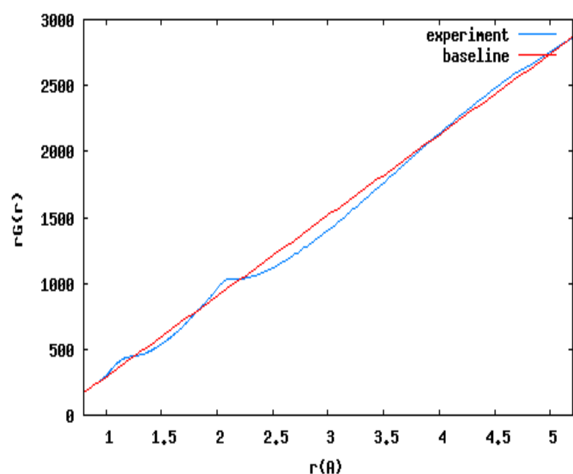
Fig. 3. Radial distribution functions determined for carbon thin films.

The d value for two peak observed in profile are 0.206 and 0.111nm as we can see in followed table.

Table 1. Ring/arc pattern analysis

Peak number	Radius pixels	d-value (Å)	Width pixels	Width (Å)	Int. intens	Scaled
1	216	2.0598	27.37	0.23	9361	2885
2	401	1.1129	84.95	0.19	32440	10000

Determined values could be assigned to diamond cubic structure (for crystalline regions); second d value has a shift because of background contribution.



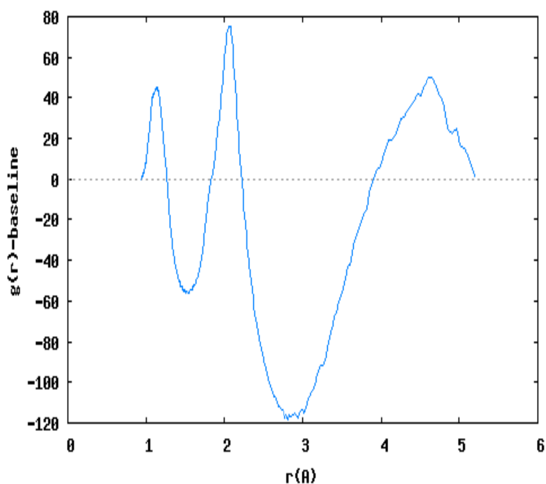


Fig. 4. Radial distribution function and baseline.

In the above graphics is presented experimental rG(r) and fitted baseline that was extracted in second graphic. These improve the visualization of peaks from rG(r) curve.

The first peak resides near 1.2Å and could be assigned to sp^3 connected atom. Second peak is close to 2Å and could be referred to van der Waals connected atom (1.7Å)

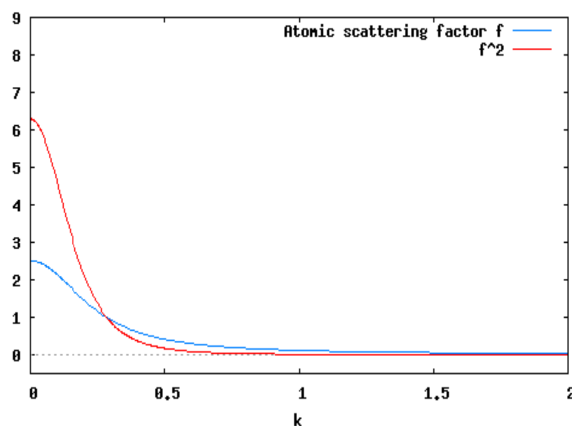


Fig. 5. Electron atomic scattering factor for carbon.

Table 2. Gaussian parameters used to obtain atomic scattering factor

A	B
0.0893	0.2456
0.2563	1.71
0.7570	6.4094
1.0487	18.6113
0.3575	50.2523

Atomic scattering factor (presented in Fig. 5) for electron was evaluated as a sum of five Gaussians with parameters gave in Table 2.

Based on data included in Table 3 and Table 4 electrical behavior patterns were assigned for carbon thin films deposited on silicon and glass substrate (Fig. 6).

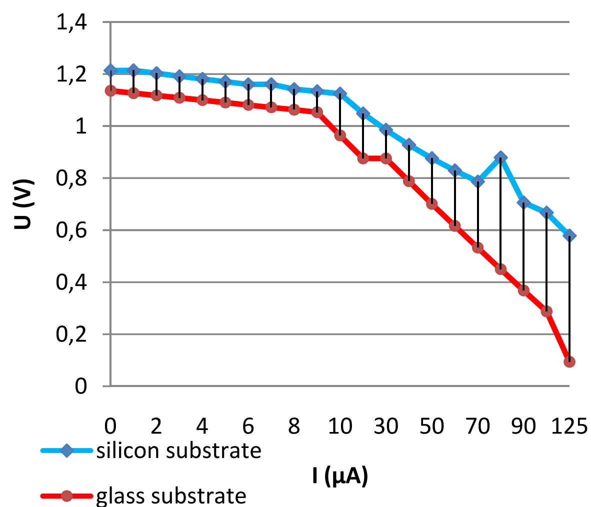


Fig. 6. V-I curve for carbon thin film on glass and silicon substrate

Table 3. Electrical measurements of carbon thin films obtained on silicon substrate.

I(μA)	U (V)
1.000	1.1362
2.000	1.1269
3.000	1.1179
4.000	1.1090
5.000	1.0999
6.000	1.0907
7.000	1.0815
8.000	1.0721
9.000	1.0631
10.000	1.0532
20.000	0.9642
30.000	0.8758
40.000	0.8756
40.000	0.7885
50.000	0.7011
60.000	0.6167
70.000	0.5330
80.000	0.4503
90.000	0.3686
100.000	0.2879
125.000	0.0942
150.000	-0.0836

Table 4. Electrical measurements of carbon thin films obtained on glass substrate

I(μA)	U (V)
0.000	1.2136
1.000	1.2140
2.000	1.2030
3.000	1.1912
4.000	1.1813
5.000	1.1707
6.000	1.1605
7.000	1.1605
8.000	1.1417
9.000	1.1337
10.000	1.1245
20.000	1.0484
30.000	0.9853
40.000	0.9275
50.000	0.8763
60.000	0.8295
70.000	0.7862
80.000	0.8789
90.000	0.7056
100.000	0.6668
125.000	0.5781
150.000	0.4955

4. Conclusions

The size of crystalline regions was determined, electrical measurements were made and morphology was observed using transmission electron microscopy (TEM).

The electron diffraction patterns were used to confirm the crystalline structure of carbon nanostructures. We found that both samples can be described using diamond cubic structure with lattice parameters a = 0.206 nm and c = 0.111 nm.

High resolution transmission electron microscopy reveals interference fringes associated with (002) planes family.

We have presented the results of electron transport studies of several representative carbon thin films. It is plausible that a good electrical contact is established between the metal pads and the ends of the nanostructures. At the present time, a complete understanding of the precise nature of contacts is still being pursued.

Acknowledgment

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