



Carbon nanostructure growth: new application of magnetron discharge

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ABSTRACT

Purpose: The application of a common magnetron discharge to the growth of carbon nanostructures is studied. The simplicity of the proposed technique can be beneficial for the development of new plasma reactors for large-scale production of carbon nanostructures.

Design/methodology/approach: Graphite cathode was treated by carbon-containing powder accelerated by use of nozzle, and then aged in hydrogen. Superposition of glow and arc discharges was obtained, when putting the cathode under the negative biasing with respect to the walls of a vacuum chamber. The pulsed discharge was preserved through the whole time of treatment. This process was explained in terms of interaction of glow discharge plasma with a surface of the cathode made of non-melting material.

Findings: The plasma treatment resulted in generation of the diverse nanostructures confirmed by SEM and TEM images. Spruce-like nanostructures and nanofibers are observed near the cathode edge where the plasma was less dense; a grass-like structure was grown in the area of “race-track”; net-like nanostructures are found among the nanofibers. These findings allow concluding about the possible implementation of the proposed method in industry.

Research limitations/implications: The main limitation is conditioned by an explosive nature of nanostructure generation in arcs; thus, more elaborate design of the setup should be developed in order to collect the nanospecies in the following study.

Practical implications: High-productivity plasma process of nanosynthesis was confirmed in this research. It can be used for possible manufacturing of field emitters, gas sensors, and supercapacitors.

Originality/value: Synthesis of carbon nanostructures is conducted by use of a simple and well-known technique of magnetron sputtering deposition where a preliminary surface treatment is added to expand the production yield and diversity of the obtained nanostructures.

Keywords: Nanotechnology, Plasma synthesis, Carbon nanostructures, Magnetron discharge, Arc spots

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MANUFACTURING AND PROCESSING

1. Introduction

Carbon nanostructures of a various dimensionality are widely recognized as advanced materials for application in biology and medicine [1], for biosensing and biomechanics [2], and early diagnosis of neurodegenerative diseases [3]; in electronics [4], high-frequency supercapacitors [5], anode materials [6], electromagnetic wave shielding, optics [7], piezoresistive and gas sensors [8]; in energy storage devices [9]; for nanofluids [10], superlubricity [11], abrasive-wear [12] and corrosion-resistant coatings [13,14], to mention but a few.

Different techniques are developed to grow 0D (fullerenes), 1D (nanoribbons, nanotubes), 2D (graphene), and complex 3D carbon nanostructures, and the technologies of their formation are still a challenge [15]. A review of the main synthetic methods of onion-like nanostructures describes application of at least five approaches including thermal annealing and electron-beam irradiation [16]. Fajardo-Díaz *et al.* carried out a research for synthesis, characterization and cyclic voltammetry studies of helical carbon nanostructures produced by thermal decomposition of ethanol on Cu-foils [17], while Ballotin *et al.* used bio-oil as a precursor to produce carbon nanostructures in liquid phase [18]. Chemical vapor deposition (CVD) growth of carbon nanostructures from nanoparticles was conducted by Kudo who reported a difference between the growth mechanisms at the dependence on the nanoparticle material [19]. A thermal CVD at temperatures of 250 to 350°C was utilized by Ma *et al.* with the aid of Cu catalyst nanoparticles with acetylene used as the reaction gas, where the interaction of hydrocarbon species with the catalyst particles is the key reaction to control the shape of the carbon nanostructures [20]. At the same time Kumar *et al.* emphasized on the reaction temperature and heating-rate in the morphology of carbon nanostructures, when the low and high heating-rates favor formation of carbon globules and nanotubes, respectively [21]. A review of growth mechanisms of bamboo-like carbon nanotubes highlighted the quasi-liquid state of metal nanoparticles as the key factor during the growth process [22].

Not surprisingly, the variety of plasma-based methods has been expanding during the last decade to grow oriented carbon nanostructures [23,24]. The key advantages of these technologies are associated with the presence of a large number of active species in plasmas, such as ions, electrons, radicals and excited states, and possibility to control the plasma parameters by not only creating the pressure gradients and sustaining a certain temperature, but also use

of the magnetic and electric fields [25]. When specifying the plasma interactions with a surface, the plasma density and electron temperature are considered as the most important parameters, which are dependent on the plasma generation source [26]. Synthesis of carbon nanotubes, nanoholes, nanofibers and nanosponges near room temperature using microwave (MW) PECVD was reported by Carvalho *et al.* [27], while carbon nanospheres and nanotubes with different morphologies were synthesized without any catalyst in microwave-assisted pyrolysis of methane [28]. Radiofrequency (RF) discharge was successfully applied to grow carbon nanowalls at low pressure [29], free standing carbon nanostructures were grown at atmospheric pressure conditions in MW plasma [30], while vertically aligned and tree-like carbon nanostructures were obtained also at low pressure in electron cyclotron resonance (ECR) plasma [31]. However, with respect to the plasma methods, arc plasma synthesis of carbon nanostructures allows obtaining the most diverse yield of the nanostructures [32], which is highly zone-dependent and enhanced by the magnetic field [33] and application of anodic arc in pulsed mode [34].

At the same time, use of the glow discharge for the carbon nanostructure growth [35], as well as implementation of the glow engaged by the magnetic field, namely, the magnetron discharge, is not so widespread in spite of the possible benefits [36]. The simplest technique to ignite plasma is based on a two-electrode setup where a negatively biased one (cathode) serves a source of electrons which ionize molecules of a background gas after being accelerated by an electric field created between the electrodes [37]. At that, for low-pressure mode the glow discharge is spread evenly along the whole surface of the electrodes thus creating the perfect conditions for a high-yield uniform growth of nanostructures. However, this configuration suffers from two main drawbacks:

- (i) absence of independent control of ion energies and ion currents, since both parameters are related in a voltage-current characteristic of the glow discharge;
- (ii) lack of density of the ion current to a particular area of an electrode to generate the carbon nanostructures.

That is why additional modifications of the glow discharge should be implemented in order to remedy the drawbacks. The first modification is connected with the necessity to split the current-voltage relations for the particular pressure of the background gas to obtain the independent control of the ion energy and current density. A magnetic field arched above the cathode surface allows reaching the purpose; thus, the common glow is turned to a magnetron discharge [37]. At that, the plasma density can be drastically

increased by more than two orders of magnitude. To overcome the second drawback of the low current density, the ignition of local arcs above the cathode surface should be ensured. As it is known, arc spots above the graphite cathode are the powerful tool to generate various carbon nanostructures, such as nanoclusters, nanotubes, and nanosheets [32]. Unfortunately, use of direct current allows obtaining the arc generation only at the initial stage of the ion cleaning. Therefore, a preliminary treatment of the magnetron cathode surface was proposed to stabilize the arcing.

2. Research methodology

The graphite cathode was treated for 5 min with nanocomposite B₄C powder (200 nm in diameter) accelerated up to velocity of 900 m/s by use of a gun intended for abrasive treatment of surfaces. Prior to the ion treatment, the cathode was chemically cleaned in an ultrasonic agitator using acetone and isopropyl alcohol. A base pressure of 10⁻³ Pa was maintained in the vacuum chamber using a turbomolecular pump backed by a forevacuum pump. Then the cathode was stayed for 24 hours in a vacuum chamber filled with hydrogen under the pressure of 100 Pa. Since hydrogen is known for the ability to saturate the surface layers with H₂ molecules, a layer of graphite with hydrogen clusters was supposedly formed. After the hydrogen treatment, the cathode was mounted on a magnetron device. A schematic of the experimental setup for nanostructure growth is shown in Figure 1.

The planar magnetron provided with the modified graphite cathode with the diameter of 236 mm was installed in the cylindrical vacuum chamber with the outer diameter of 320 mm, inner diameter of 300 mm, and height of 350 mm.

The chamber was filled with oxygen, and pressure of 25 Pa was maintained. As was observed in experiments, graphite surface exhibits a rough topography with nanometre-sized hillocks after oxygen etching [38], which is favourable for the arc initiation. This result is explained by the chemical selectivity of the etching, when the defect areas and perfect sites of basal planes are etched non-uniformly. It is assumed that the effect is dominated by chemical reactions between neutral oxygen atoms from the plasma and carbon atoms from the sample, while the ballistic effects caused by the ion bombardment are not significant in the etching. The discharge characteristics of the system were obtained by use of the customary-designed data acquisition system, which is able to write the discharge parameters such as voltage, current, and gas pressure at the frequency of 25 Hz. Unlike the typical behaviour of the magnetron discharge in similar geometry, a stable plasma torus was not reached for the whole time of experiments for 1 h duration.

The plasma discharge was observed as a superposition of a stable glow with arcs. The glow exhibited a pulsed mode, when the discharge brightness gradually increased until the arc spot is formed above the cathode surface, which was followed by the generation of clusters of the cathode material emitted due to the action of thermoelastic stress. Three photographs of the discharge, which are separated by the time interval of 40 ms, are shown in Figure 2. Faint plasma torus associated with a typical magnetron discharge is shown in Figure 2(a); the glow gradually increases in Figure 2(b) and the arc spot is initiated in Figure 2(c). The emission of the graphite clusters is associated with the bright beams protruding from the arc spot, which can be distinguished in Figure 2(c). Thus, the formation of active spots for the nanostructure growth followed by the plasma etching occurs.

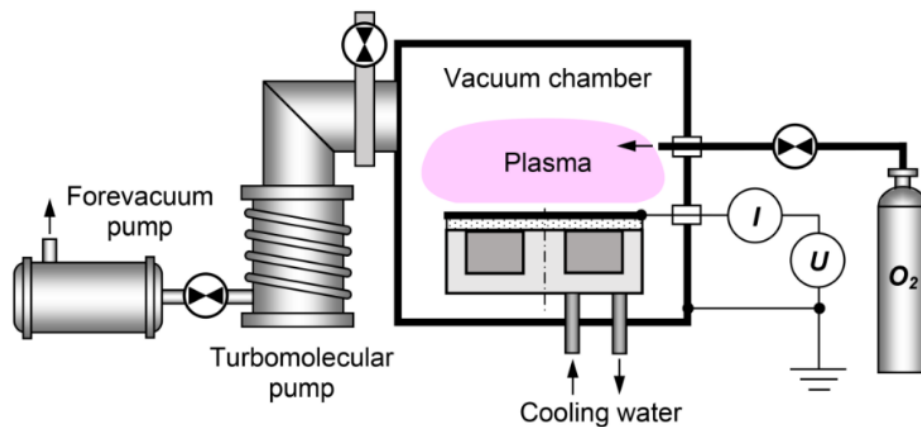


Fig. 1. A schematic of the experimental setup

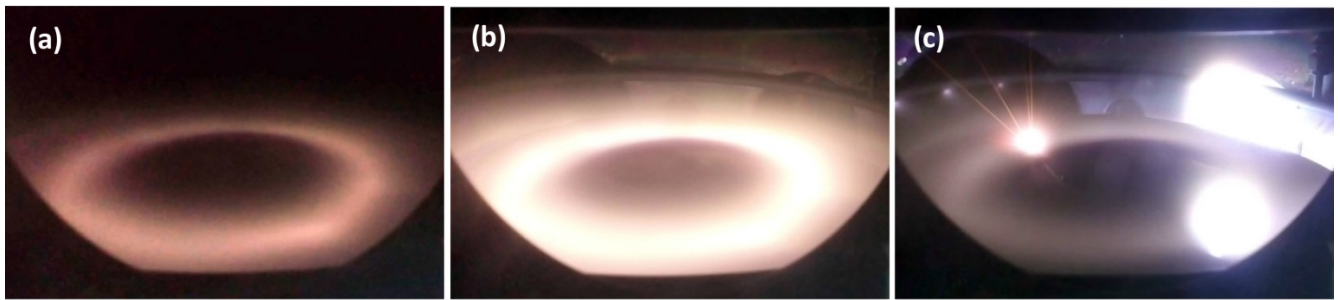


Fig. 2. Photograph of the process of the formation of the clusters of the cathode material in the arc discharge spot: (a) faint glow that (b) becomes brighter before (c) the formation of cluster in the arc spot

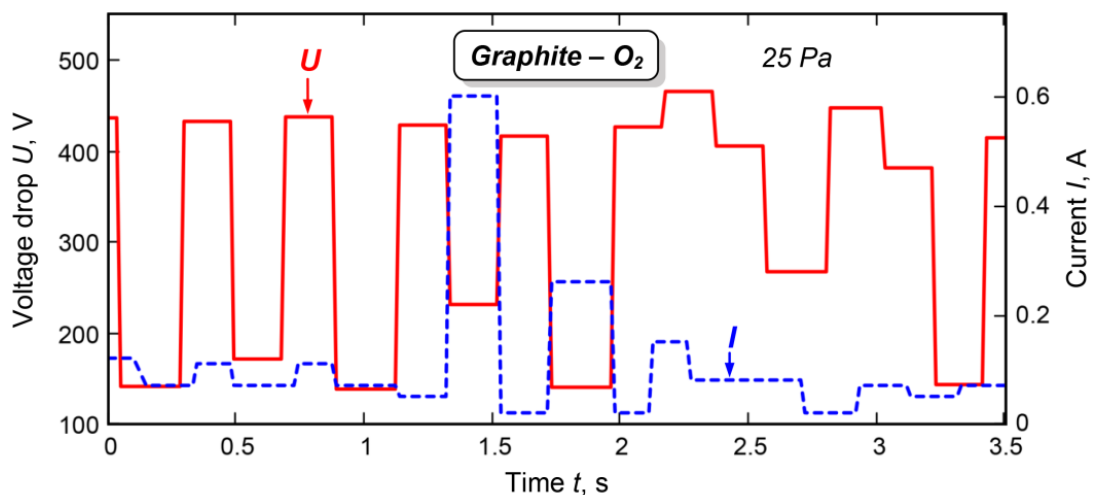


Fig. 3. Characteristics of the discharge at oxygen pressure of 25 Pa

A typical fragment of the dependence of the discharge characteristics such as voltage drop U and discharge current I on time t is shown in Figure 3. A qualitative description of the processes in the arc spot is different for the arcs above the metal and graphite cathodes. In both cases, surface microprotrusions play an important role in the arc generation, since the electric field is greatly enhanced by them, which in turn enhances the electron emission to ignite the spot. When the arc is initiated above a metal surface, the protrusion is melted by the arc current; the local electric field weakens and cannot sustain the arc. After the arc termination, the metal surface becomes smoother, i.e. the number of protrusions decreases, and they are changed to craters. In contrast, the arc above the cathode made of graphite results in generation of solid clusters formed due to the large thermoelastic stress, and ejected from the surface. Thus, the graphite surface is covered with craters with sharp edges that promote formation of new arcs followed by the

cluster generation. This process is supposedly sustained by the presence of the hydrogen gas clusters in the undersurface graphite layers. Hence, the arc ignition above the graphite cathode during the whole treatment modifies the surface and stimulates the growth of various carbon nanostructures.

These speculations were confirmed in experiments. After the ion treatment, the surface layer of the cathode made of the modified graphite was investigated by use of SEM and TEM techniques.

3. Research results and their analysis

SEM images of different areas of the surface are shown in Figure 4. The initial surface after the abrasive treatment by use of nanocomposite carbon-containing powder accelerated in hypersonic nozzle is shown in Figure 4(a), where large number of microscopic defects can be distinguished yet the nanostructures are absent.

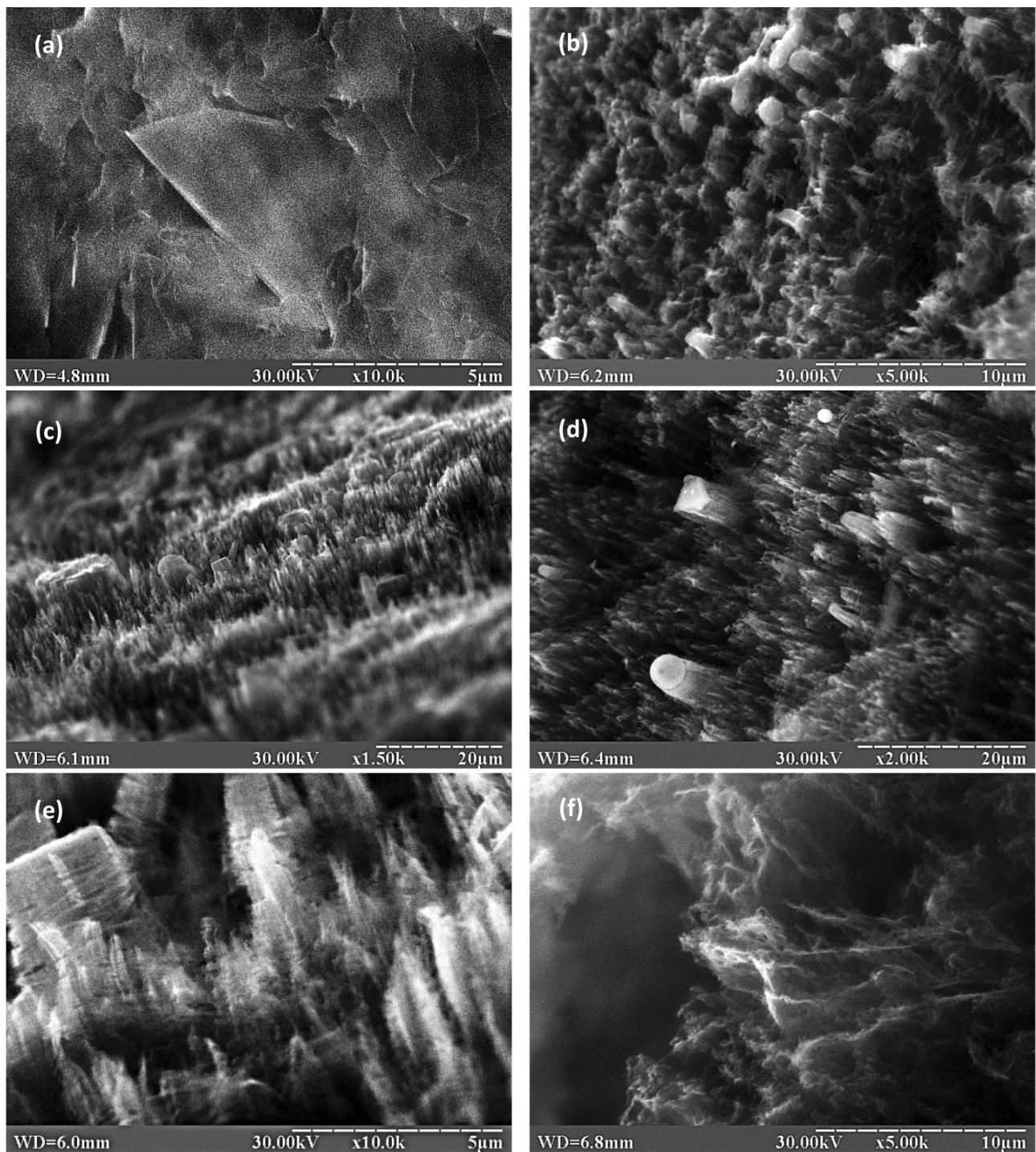


Fig. 4. SEM images of modified graphite surface: (a) after the treatment with the carbon-containing powder accelerated in a hypersonic nozzle, and before the ion treatment; (b) region near the cathode edge where the magnetic field lines are perpendicular to the cathode surface (most of the arcs were generated in that region); (c)–(f) are the regions with the most intense plasma treatment were (c) grass-like, (d) patterned, (e) columnar with nanosheets, and (f) 2D net-like nanostructures were found

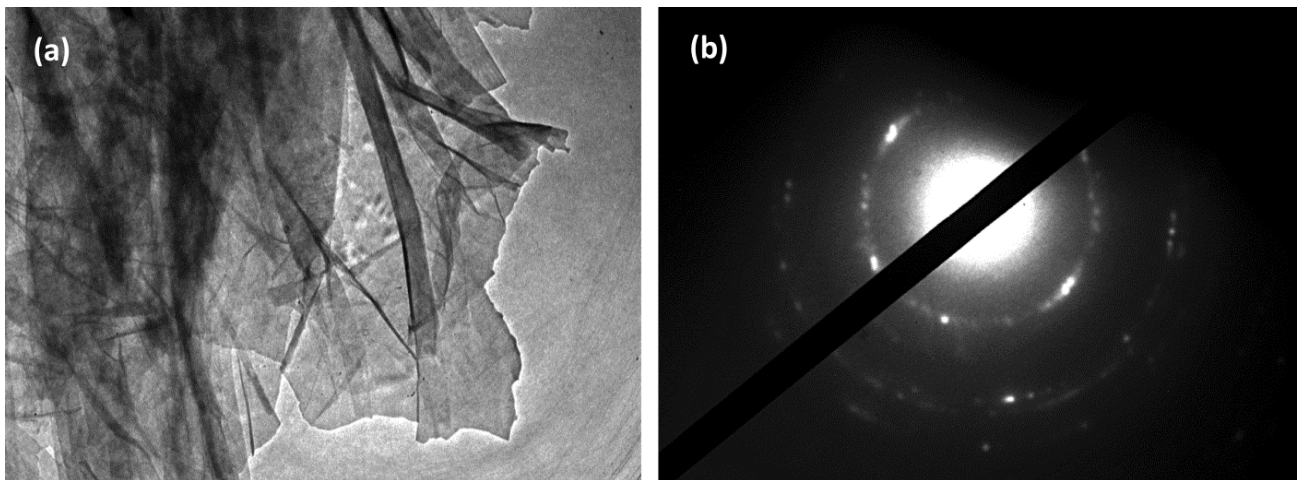


Fig. 5. TEM images of 2D net-like nanostructures: (a) general structure; (b) image of the electron diffraction confirming the carbon nanostructure

After the plasma treatment, the separate areas with spruce-like nanostructures and nanofibers with a thickness of about 100 nm are observed near the cathode edge where the plasma influence was insufficient, as it is shown in Figure 4(b). The spruce-like nanostructures found at the distance of 1-2 mm from the cathode edge are angled toward the edge, which can be explained by the topography of the electric fields there. The plasma influence is more prominent in the area of the most intensive ion sputtering (“race-track”). Thus, a grass-like structure of the nanofibers with a height of 2 μm and a diameter of 200 nm and a possible application as the field emitters can be seen in Figure 4(c). One more interesting effect can also be implemented in practice, namely, the metal particles with a diameter up to 3 μm deposited on the cathode surface during the arcing, which acted like a pattern with respect to the surface sputtered by the ion flux, Ref. Figure 4(d). Generally, the region of the intense sputtering in plasma glow starts at the distance of 3 cm from the cathode edge, and it contains a lot of craters and their number decreases toward the cathode centre. The areas with the densely-packed and twisted nanofibers are observed through the whole region; the nanofibers are bundled and feature sharp edges thus resembling a grass cover. Columns with nanowalls of less than 100 nm in thickness are distinguished among the nanofibers, as it is shown in Figure 4(e), thus forming the areas with high surface-to-volume ratio, which can be beneficial in development of highly sensitive analysers. As for the craters, their shape resembles the groove after the discharge action; the bundles of the nanofibers are found at the crater edges.

In addition, 2D net-like nanostructures (nanosheets) were observed, Ref. Figure 4(f). The nanosheets studied by use of TEM are shown in Figure 5(a) with magnification of $\times 80000$; the image of the electron diffraction of the carbon nanostructure is shown in Figure 5(b). The structure thickness does not exceed 15 nm, as was concluded after the measurements.

4. Conclusions

The results of the experiment confirmed that the magnetron discharge can be a powerful tool to generate the carbon nanostructures in the magnetically-enhanced glow discharge by use of the proposed technique of the preliminary preparation of the cathode surface. The continuous arcing superimposed with the magnetron glow above the cathode leads to abundant yield of the carbon nanostructures with different morphology. 1D, 2D, and complex net-like 2D nanostructures are grown at the dependence on the position along the cathode surface. Nanopatterning caused by the presence of droplets emitted during the arc stage was also observed, and can be used to create patterns for the selective ion etching. The next stage of the research should involve the collection of the graphite samples emitted from the surface by the arcs.

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