Synthesis of carbon nanostructures in electric discharge

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Abstract. A synthesis method of carbon nanomaterials in an argon arc is described in this paper. The two separate cases have been considered. In the first case it is carried out by evaporation of a graphite anode followed by deposition of carbon vapor on a tungsten cathode. In the second case the anode material was specially synthesized in the electric arc recessed into liquid hydrocarbon raw material. The fuel oil was used as a hydrocarbon raw material. During that decomposition of this material took place and as a result carbon deposits were formed on the electrodes. Then these carbon deposits were used as the arc discharge anode at the next stage of the process. In both cases the carbon synthesis of nanotubes was carried out on the tungsten cathode surface. There was the intensive growth of carbon nanotubes during the experiments.

Key words: carbon nanotubes (CNT), electric arc, graphite, synthesis of carbon nanomaterials.

1. Introduction

Despite the fact that the technology of growing carbon nanotubes (CNTs) is already well developed [1–4], the search for new ways to synthesize carbon nanotubes still remains extremely important. And the main reason for that is the expansion of carbon nanotubes application fields. Carbon nanotubes are most widely used as additives in polymers, in catalysts, as anodes in lithium batteries, in composite materials, supercapacitors, and in many other areas. Carbon nanotubes are used more and more in electronics, medicine and energetics.

A wide range of CNT application is due to its extraordinary properties, such as high strength, capillarity, superconductivity, etc. They have a molecular structure in the form of a skeleton and their strength is not much different from the strength of a diamond.

Carbon nanotubes could be used in much more spheres, but due to the expensiveness of their production it is not happening. Also depending on the sphere of CNT application nanotubes with various parameters are required. In some cases (medicine, electronics), single-wall nanotubes are required, in other cases (lithium-ion batteries, catalysts) - multi-walled are needed. When creating nanocomposites, nanotubes with non-standard geometry work best.

Currently, nanotubes are produced using an electric arc, by laser evaporation and chemical deposition [1-2]. There are also methods for producing carbon nanotubes from liquid hydrocarbons [3-4]. Electrical and gas-dynamic characteristics of electric discharges, effectively used in the synthesis of carbon nanostructures, are presented in the works [5–9] describing Heat transfer in a longitudinal glow discharge.

In this paper, a technique for the production of such carbon nanostructures as nanotubes and graphene in argon electric arc discharge from graphite, as well as from nanomaterials, preliminarily deposited on the cathode in electric arc decomposition of fuel oil residue, is proposed. A tungsten rod with a diameter of 5 mm was used as the cathode. Tungsten has a hexagonal structure similar to that of
graphite. Based on this, it was assumed that nucleation centers of nanostructures will form on the graphite surface, and tungsten molecules will act as a reaction catalyst.

2. Experiment
The experiment was carried out in a cylindrical vacuum stainless steel chamber with a diameter of 40 cm and height of 25 cm. (see figure 1). Both the walls and the doors of the chamber are water-cooled with double walls. In the chamber there are current leads and technological openings for gas outlet, noble gas inflows and ambient air inflows for pressure equalization. The chamber also has transparent doors from heat-resistant glass to monitor the formation of carbon nanostructures. AC welding transformer with rectified adjustable current of up to 300 A was used as a power source. A tungsten rod served as the cathode, and a 5 mm-dia graphite rod served as the anode.

After the air was pumped out, the chamber was filled with argon up to the pressure of 100 Tor. After lighting the electric arc between the electrodes, growth of deposits on the tungsten cathode was observed. Then the experiment was repeated for pressures of 300, 400, 500, 550, 600 Tor. Carbon deposits growth rate at the cathode was determined. During the experiments we tried to maintain the constant distance between the electrodes in the range of 3-5 mm. Similar experiments were carried out at various current values (from 50 to 100 A). During the experiments a rapid growth of CNTs was observed on the cathode surface. The samples of the highest quality were obtained at a pressure of 550 Tor and a current of 100A, and at a pressure of 85 Tor and a current of 100A.

It is necessary to monitor the experiment through dark glass to avoid the retinal burn. As the material of the anode evaporates and the CNT grows on the tungsten cathode, the distance between the electrodes is controlled by means of regulators for raising and lowering the electrodes. The experiment lasts several minutes, depending on the growth of nanotubes needed. Figure 1 demonstrates electrodes after the completion of CNT synthesis experiment.

It can be seen that there is a growth of carbon nanotubes on the tungsten cathode. The graphite rod is almost completely removed on the cathode surface. It is remarkable that the graphite rod diameter remains the same. Both the diameter of the carbon nanotube deposits and the diameter of the graphite rod are 5 mm. If the same discharge conditions are observed, it can be expected that the length of the synthesized nanotubes will be about a few mm.

The results of studies of samples obtained in an electron microscope are given below.

3. Results
We see from Figures 2 and 3, that about 60-70% of the obtained material are carbon nanotubes. Their length is not less than a few microns.
At relatively low pressures (200 Tor and below), the electric arc section expands and the synthesis of graphene sheets begins on the side surface of the tungsten cathode. According to preliminary
estimates, the thickness of graphene sheets reaches several nanometers.

Figure 4. Photographs of samples obtained in argon at a pressure of 150 Tor and a current of 75A.

Figure 5. Carbon nanotubes synthesized from carbon deposits on the cathode during plasma chemical decomposition of liquid hydrocarbons.

The sample presented in this photograph (figure 4) was grown on the sides of a tungsten substrate.
This image shows that there is flat graphene here. At 35,000x magnification, we can already see that the nanotubes have grown in different directions. The diameter of carbon nanotubes is roughly the same, the length is around several micrometers. The quantity of nanotubes within the field of view in this image (figure 5) is about 97%. Also in this picture there are visible round formations that have a fairly good rounded smooth shape. The diameter of these formations ranges from 20 to 300 nm.

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