ARC DISCHARGE INSTALATION FOR FULLERENE PRODUCTION*

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The electric arc is one of the most used methods to produce fullerenes. This work describes an electric arc installation based able to produce carbon nanostructures with large values of the fullerene yield (fullerene mass proportion in the soot) and productivity.

Key words: fullerene, arc discharge, carbon nanostructure.

1. INTRODUCTION

Fullerenes, a new form of carbon, were discovered in 1985 [1] in graphite vaporization under inert gas at low pressure. Fullerenes have many properties different from either diamond or graphite. Potential applications include superconductors, sensors, catalysts, optical and electronic devices, polymer composites, high-energy fuels and biological and medical materials. But wide application of fullerenes is undetermined by an inability to produce large and inexpensively quantities. The arc discharge method developed by Kratschmer's group [2] in 1990 remains until now the major tool for synthesizing fullerenes. Several works has been done for the macroscopic production of the fullerenes by using other different techniques, laser ablation [1], electron beam evaporation [3], heat resistive method [4], diffusion flame [5] and ion beam sputtering [6].

It is generally accepted that the fullerenes and carbon nanotubes are formatted in special conditions: at a very high temperature (a laser beam or an electric arc discharge evaporation of graphite) and in an inert gas atmosphere at a reduced pressure [7]. On the other hand, fullerenes, fullerene derivatives and fullerene-like structures have been detected in the soot produced for technical applications and also in the diesel motor soot [7].

The electric arc is still one of the nest methods to produce fullerenes, which is why we decided to carry out an installation in order to produce carbon nanostructures with large values of the fullerene yield (fullerene mass proportion in the soot) and productivity. The paper presents the installation components and the obtained results.

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2. EXPERIMENTAL SETUP

The fullerenes were prepared by evaporation of carbon electrodes in an electric arc discharge process in helium atmosphere. The experimental setup is shown in Fig. 1.

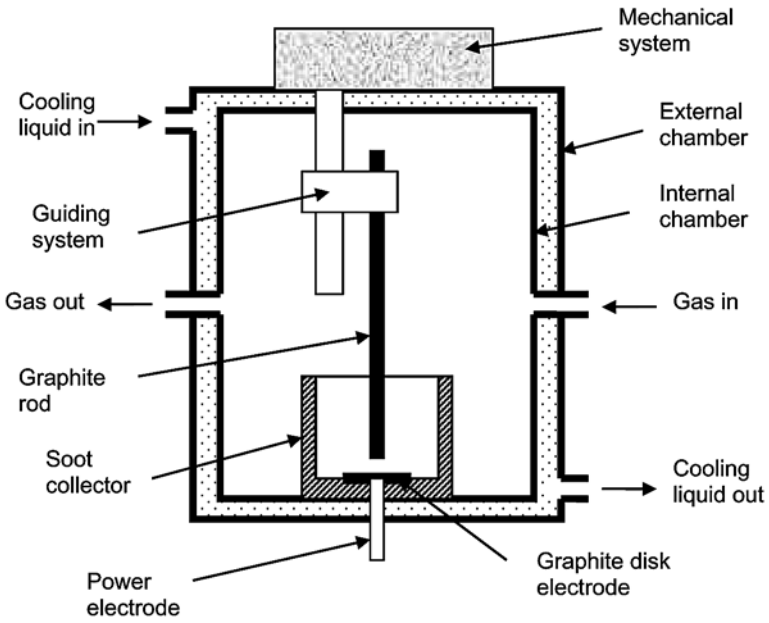

Fig. 1 – Schematic diagram of fullerene production apparatus.

The main part of the deposition system consists in a stainless steel, double walled, cylindrical chamber. Between the two chamber walls is flowing the cooling agent, the temperature being automatically controlled. The two electrodes were horizontally mounted near the bottom of a reaction chamber. For the anode electrode we have used pure graphite electrodes of a diameter of 6 mm and the length of 140 mm. The second electrode consists in a pure graphite disk, mounted at the bottom of the reaction chamber. The reaction chamber acts also like a soot collector. The anode is mounted in a guiding system, controlled by a mechanical system in order to assure a constant distance between the two electrodes during arc discharge. DC power supply unit was used and operated at the voltage of 10–20 V and the current of 0–250 A. In order to avoid the heating of the connection connectors, the electrical contact was made directly between the two electrodes and the discharge chamber walls.

To obtain the carbon soot, the chamber is evacuated of the air until a pressure of 10^{-3}–10^{-4} Torr. The arc discharge is start up for 2–3 minutes, in order to heat the electrodes at the working temperature. After that, the chamber

was filled in with a noble gas (argon or helium) at the pressure between 50 and 200 Torr. The arc intensity is controlled by the distance between the electrodes. During the process, the temperature of the chamber walls is maintained at $30 \pm 2°C$.

The carbon soot was investigated using X-ray diffractometry with a "DRON 2.0" apparatus. Fullerene C_{60} was extracted from de soot with toluene and by vacuum evaporation.

3. RESULTS AND DISCUSSION

The main technological parameters that control the process efficiency are: the discharge current, the distance between the electrodes, the pressure and nature of the working gas, the electrodes composition, shape and dimensions.

First, we present the results from the structural analysis of the graphite electrodes and carbon soot. In Fig. 2 is presented the X-ray diffractrogram for the electrodes material.

It shows the characteristic peaks corresponding to the polycrystalline graphite: a ($2\theta = 12.55$), b ($2\theta = 26.35$), c ($2\theta = 42.64$), d ($2\theta = 44.86$), e ($2\theta = 54.27$), f ($2\theta = 60.0$), g ($2\theta = 77.84$), h ($2\theta = 84.18$), i ($2\theta = 86.99$), j ($2\theta = 94.59$), k ($2\theta = 102.14$) [8]. The diffractogram obtained for a carbon soot probe, presented in Fig. 3, shows traces of graphitic carbon (peaks b and c) and a broad diffuse hump in the diffraction pattern around $2\theta = 20$ that is indicative for the presence

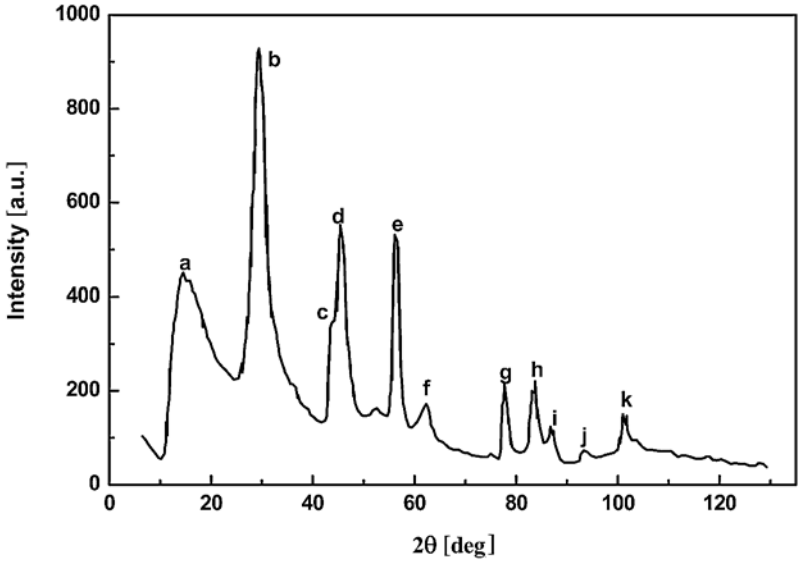

Fig. 2 – X-ray diffractogram of graphite electrodes.

of amorphous carbon [9]. The A ($2\theta = 17.5$) and B ($2\theta = 21$) peaks are characteristics for C60 crystal indicating the presence of the fullerene molecules in the discharge soot.

In Fig. 4 are presented two I–V discharge characteristics, for two distances between the electrodes, 1 mm and 4 mm. The working gas was helium, at 100 Torr pressure.

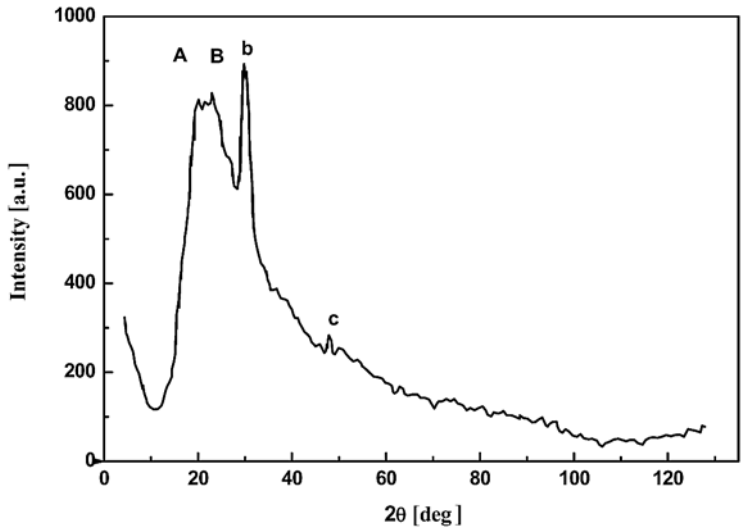

Fig. 3 – X-ray diffractogram of carbon soot.

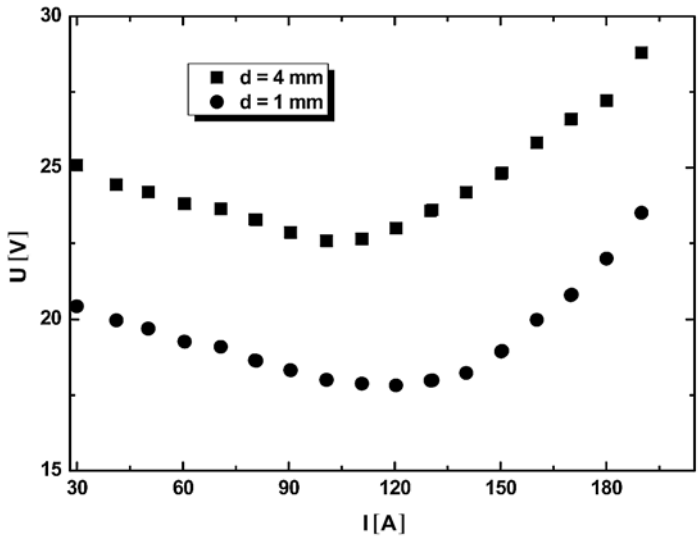

Fig. 4 – I–V discharge characteristics, for two distances between the electrodes.

As we can observe, the discharge power present a minimum for discharge currents in the range 90–150 A. The initial decrease of the power, when the current increases, can be the result of the increase of the graphite fragments concentration in the discharge plasma. When the discharge current increase, the graphite fragments are ionizated and the discharge power increase. The increases of the gap between the two electrodes don't change the shape of the characteristic, the only effect being a translation of the curve to higher voltages.

It is also interesting to see how is influenced the anode burn velocity by the discharge current. In Fig. 5 are presented two dependencies: the anode burn velocity and, respectively, the soot generation velocity, as a function of discharge current.

For small values of the discharge current, the two velocities are practically equals, indicating a small erosion rate from the cathode. When the discharge current increases, the anode burn velocity increases. The soot generation velocity increases faster, because of a greater erosion rate from the cathode. The increase of the discharge current, correlated with an increase of the fullerenes proportion in the soot, lead to an optimized discharge.

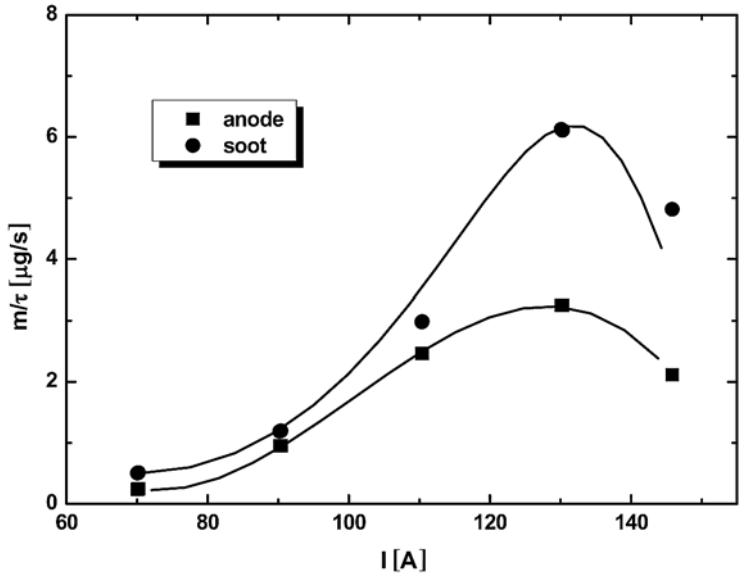

Fig. 5 – The anode burn velocity and, respectively, the soot generation velocity, as a function of discharge current.

4. CONCLUSIONS

This paper describes an electric arc based installation able to produce carbon nanostructures with large values of the fullerene yield (fullerene mass

proportion in the soot) and productivity. The electrodes material and the produced soot were analyzed by X-ray diffraction in order to confirm the presence of the fullerene molecules in generated material. When the discharge current increase, the power first decrease and begin to increase for currents around 120 A. In this region, the increase of the discharge power is correlated with an increase of the soot generation velocity and an increase in the discharge efficiency.

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