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# ARC Discharge with a Directional Gas Flow: Synthesis and Properties of Fullerenes

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**Abstract** In the discharge with a hollow carbon electrode with the gas flow direction of formation duration of the trajectory forming in the space with a high plasma density and temperature longer. In the sediment synthesis product is not marked amorphous carbon, but the increased essentially output of heavy fullerenes. In this condition a trajectory of fullerene forming, as shows calculation, is about a helix and more then in an usual arc discharge There were made calculations of the fundamental characteristics of heavy fullerenes. It noted the dependence of the spectrum and the uncertainty of the stability of the fullerene ion depending on the sign of the charge of fullerenes, and the preferred use of ions fullerenes.

**Keywords** Adiabatic electron affinity, amorphous component, formation duration, gas-plasma stream, hollow electrode, turbulence

## Introduction

In the 1970s, the possibility of the existence of hollow $C_{20}$, $C_{60}$ clusters and their electronic structures were shown theoretically [1]. These clusters of carbon with a characteristic size ≈1nm were called fullerenes. These structures were obtained experimentally in a supersonic helium stream by the action of a laser beam on the graphite surface [2]. Investigations showed that fullerenes are effective electron acceptors and form compounds with new properties with atoms of other elements. The discovered peculiarities show fullerenes and their derivatives to be a new class of molecules-compounds; the trends in physics, chemistry, biology, medicine and technology, which are originating on their basis, are also new. Big hopes are tied with water-soluble fullerenes' form, which would allow you to enter the sphere of the use of different salt solutions including the lymph and blood person, and directly related to determining the person' health. In ongoing studies [3, 4], it is assumed to develop method for fullerene modifying surface and to obtain more stable output of product.

Interest in fullerene structures increased when based on composite organic semiconductors with fullerenes $C_{60}$ was established effective photoelectric converter. In the device first was given going out of conversion efficiency more than 1%. It has been achieved with the process adsorption of a quantum radiation in the organic semiconductor and with a high amount of fullerene electron affinity [5]. The last parameter defines the time of forming and life-time of fullerene negative ions (NI). In this type of solar cells efficiency of converting solar energy into electrical energy reaches 6,7 % [6]. (Overall effectiveness – taking into account the cost of materials and environmentally friendly production process – plastic organic solar cells reaches silicon batteries, the effectiveness of which is 2-5 times less than achieved in conventional sources TES or nuclear power plants UES, and is expected to obtain greater efficiency in the organic semiconductor batteries, if you reduce the cost of fullerene materials[7]). The photocell Electro-donor material is selected from a variety of organic materials [8], but the acceptor material must contain fullerenes ($C_{60}$,$C_{84}$) or their connection. Grab a free e-fullerene and prevention of recombination of charges for the certain

period of time determined by the lifetime **τ** of NI fullerene. On ecological and economical solar cells made of organic semiconductors with fullerenes surpass the known elements of the Si, GaAs, ZnO,…[9].

**General Provisions**

Therefore it was intended to carry out a technological revolution by modernizing the main spheres of activity through the use of fullerenes, their derivatives and composites based on them as the main material. To realize this idea, a search for and development of fullerene synthesis methods were initiated. At the present time, the most commonly used fullerene synthesis methods are: laser [2], chemical [10] and arc discharge [11, 12] methods. The common peculiarity of the methods is that fullerene synthesis takes place in an inert gaseous medium at the temperature $1500 < T < 7000$ K (low - temperature plasma conditions). The formation of nanostructures in a space with high temperature takes place by collision of carbon clusters. The process is affected by the Coulomb interaction of particles with opposite sign. The synthesis methods differ in fullerene formation mechanism, but the results of the last few decades have shown that methods within the measurement error have a limit value output $\eta = (19 \pm 4)$ %. New methods for the synthesis of fullerenes have relatively smaller history, experimental basis, and have defects. For laser method disadvantages are: low percentage conversion of pump energy into the energy of the beam, the opacity of carbon vapor laser beam and the problem of radiation entering the cell fusion. For the chemical method is characterized by a low percentage of conversion of carbon clusters in view of the complete structure of the fullerene conversion features polycyclic aromatic hydrocarbons (PAHs) [13] at a low value of optimal synthesis temperature ($T \leq 2000$ K). Further development of laser and chemical methods probably will eliminate the deficiencies noted. But arc plasma medium on the basis of carbon vapor is an indispensable element to maintain the continuity of the arc and the synthesis process. Discharge control elements are: the geometry of the electrode discharge, gas atmosphere and arc discharge parameters. An additional advantage of the method is the experience of creating and using a wide range of bit devices and the equipment for arc discharge.

The aim of the work is a more detailed discussion of the features of the new method of arc synthesis of fullerenes [14], which showed a significant advantage in comparison with the approved methods, and to study insufficiently known the fundamental characteristics of heavy fullerenes, to explain the feature of the mass spectrum of second processes. The content of this article at the urging the wseas.org the proposal is in addition to the advanced [14], therefore presented in the materials obtained primarily from the same experimental facilities, some parts are abridged and some new parts added.

**Problem Solution**

**1. General Picture of Fullerene Formation Process in Arc Discharge**

Fullerene arc synthesis is a many stage event process, the theory of which has not been carried to completion. According to general concepts, the $C_j$ fullerene formation process takes place in the discharge space at 1500 – 5000 K during diffusion as a result of collisions followed by coalescence of nanosized particles. Fullerene synthesis is conventionally represented as series of acts of attachment of small $C_k$, $C_i$ clusters of carbon vapor from the heated portions of the electrodes and the subsequent kinetic excess energy relaxation of coalescing particles in inverse processes, annealing-away of $n_f \cdot C_f$ clusters. Conventional synthesis scheme:

$$C_j = C_к + \sum|_i n_i \cdot C_i - \sum|_f n_f \cdot C_f, \qquad (1)$$

where $C_k$ is the initial fullerene cluster formed in the interelectrode space, the coefficients $n_i$, $n_f$ are integers. The clusters in the scheme (1) are summed with respect to the subscripts i and f. According to the scheme (1), the formation parameters of each $C_j$ fullerene also develop on the conventional path. The fullerene formation parameters are formed from the sum of individual segments: duration of the formation process, $T_j = \sum|_j t_{Cj}$, and formation path length $H_j = \sum|_j h_{Cj}$.

During the formation stability of the structure is controlled by the annealing process, which is under the influence of atomic bonds at conventional scheme (1) correspond to particles $\sum|_f n_f \cdot C_f$. Structure formed with a number of carbon atoms believe resonance ($C_{60}$, $C_{70}$,…), because the mechanism of formation of these structures is unknown. In arc

discharge, the diffusion nature of spatial motion with temperature decrease from radius gives a spread of clusters in path parameters, in mass and quality of the product. In arc discharge, amorphous carbon black accounts for a larger fraction of the product (> 50%). From the analysis of the existing concepts it may be assumed that the product of other quality can be obtained in a space with high temperature, in which longer duration and length of fullerene formation path with turbulent portions are ensured. The space with such peculiarities has a circular symmetry at the minimal energy consumption.

**2. Arc Discharge in Hollow Electrode with Working Gas Flow (ADHE-WGF)**

**Discharge Device**

To ensure continuous synthesis process, a stock-produced TDM-317 as welding power source with dropping current-voltage characteristic and a pulsed arcing stabilizer (PAS) were used jointly. The TDM-317 + PAS circuit continuously holds plasma in the space between electrodes, also at the moments of passage of current and voltage through zero [15]. Presented here design of the discharge device was created based on the original completed devices [16, 17], which was develop a design discharge chamber on the possibility to combine the advantages of the marked above bit devices. In the works [18, 19], fullerene synthesis was carried out in a chamber of 68 mm diameter (h = 150 mm) on the discharge device shown in Fig.1: graphite electrode 1 for emission spectrum analysis (diameter 6 mm), graphite electrode 2 (cavity diameter 15 mm, h = 15 mm) with slits through which working gas (He) enters the electrode cavity. The cuts (slots) in transverse and longitudinal dimensions provide education directed jet passing gas. In the design, the part 3 is a graphite cover. Electrical insulation between the electrodes 1 and 2 is provided by a high-temperature ceramic, an alundum crucible 4. An extra function of crucible is heating the working gas that enters the discharge space. The working gas coming from the free space heats on the passage of gas-plasma flow and discharge radiation over the surface of the crucible, which heats up on thermal contact with the heated electrode portion. The parts 5 and 6 of common steel apply potential to the electrode 2 and fasten the construction together into a whole. The part 7 is the chamber wall.

**3. Product of Synthesis by Arc Discharge in ADHE-WGF**

The composition of the product of synthesis by ADHE-WGF fullerite was determined on an AutoFlex apparatus (Bruker, Germany) from a time-of-flight mass spectrum (MS), Fig. 2. This is a MS of fullerenes with negative charge for a product obtained in ADHE-WGF under optimal conditions. As can be seen, the lines in this series follow at the interval $\Delta$ (m/z) = 24 amu = $2m_c$, which is usual for fullerene spectrum. It is seen from Fig. 2 that the MS consists of background bands, which differ in height and width, bands of medium hydrogenated clusters ($C_{26}$, $C_{27}$, $C_{28}$), $C_{60}$, $C_{70}$, $C_{72}$ and a series of $C_{74+2n}$ lines, where n = 0-40. In the spectrum, the lines of stable nanostructures, which accumulated during discharge on the cooled walls of the chamber: $C_{60}^-$, $C_{70}^-$,…$C_{78}^-$, $C_{84}^-$,.., $C_{90}^-$, $C_{96}^-$, $C_{112}^-$,… $C_{150}^-$,…stand out in the value of the peaks. The peak values in the MS of fullerenes in Fig 2 are in agreement with the formation mechanism of scheme (1) and another detailed presentation proposed: the peak values of fullerenes decrease with increasing mass, but the decrease in peak values in ADHE-WGF is smaller as compared with ordinary-arc discharge spectrum. An important peculiarity of the spectrum of ADHE-WGF, which contradicts the above one, is that the peak value of $C_{84}$ fullerene exceeds that of the $C_{70}$ fullerene. The spectrum of fullerenes with positive charge ends with the $C_{84}^+$ fullerene peak (see below), but the background spectrum has an extension at m/z > 1800 u.

**4. Accurate Mechanism of Fullerene Formation in ADHE-WGF**

As was pointed out above (1), fullerenes begin to form on collisions of carbon atoms, ions and small $C_2$, $C_3$, $C_4$ clusters by the action of a turbulent evaporation flow from the surface of the electrodes. But in a hollow electrode, the turbulence under the action of working gas flow is higher, and the process occurs with the participation of helium atoms via forward and back reactions [20]. Another distinction of the MS of ADHE-WGF from ordinary-discharge MS is background. The structures that create a background in the MS of ADHE-WGF don't affect the transparency of the benzene solution of fullerene carbon black. The background is created by nanostructures formed as a result of "re-formation" of fullerenes and annealing away of fragments, which become just a "nanobackground". The nanobackground peaks in the mass spectrum 24 ≤ m/z < 2000 u have a value of up to 2 – 4

% of the peak value of $C_{60}$. The MS of the product of ADHE-WGF at the discharge current I = 70 A without filtration coincides with the spectrum obtained after double filtration. The result is confirmed by the MS of fullerene carbon black synthesized at I = 80 A. There is a background in the spectrum of positive and negative ions, and the background is larger for the product obtained at higher discharge current.

**5. Geometrical Factor in Fullerene Formation Shows the Nature of the Limit Value Outputs**

In arc discharge, the length of fullerene formation path is estimated from the equation:

$$L_0 = (1 \div 3) \times \Delta, \quad (2)$$

where Δ is working interelectrode gap Δ =1 –7 mm [19], and the length of fullerene formation path $L_0 \approx 15$ mm. An examination of the formation space in the device for ADHE-WGF is given on the basis of the size of the electrodes and gas plasma thermodynamics, gives the path:

$$L_1^* \geq 100 \text{ mm}. \quad (3)$$

In this case the trajectory of the fullerene formation shaped like a helix, which has 2-3 turns. Comparison of the values in (2) and (3) shows an advantage of the method with hollow electrode over that with ordinary discharge: *the fullerene yield is higher at the longer length of nonlaminar formation path.*

**6. The Measurement Results**

The synthesized in ADHE-WGF product was analyzed in a solid fullerite phase on an AUTOFLEX $^{R}$IILRF 20 time-of-flight mass spectrometer with pulsed $N_2$ laser (Germany). The parameters of the laser beam: q = $2.6 \cdot 10^{-6}$J, $\tau_0 = 3 \cdot 10^{-9}$sec, s =1mm$^2$ and the estimated power density on the target.: $P = q/(\tau \cdot s) \approx 0{,}1 \text{Mw/cm}^2$. The mass spectrum negatively charged fullerenes presented in Fig. 2. The spectrum was obtained from the fullerene synthesized at the discharge current I = 70 A. The values of the peaks of the fullerenes $C_{60}$, $C_{70}$, $C_{84}$, …, $C_{150}$ in the mass spectrum are as follows: 100, 57, 78, …, 6. The descent of the envelope curve of the peaks of fullerene mass spectrum with increasing number of atoms in the cluster is common. This is in accordance with the generalized scheme (1). In discharge with hollow electrode, however, the decrease in the values of peaks is smaller, and the value peak $C_{84}$ is larger than peak $C_{70}$. The peculiarity apparently shows that the structure of the $C_{84}$ cluster is formed by the optimal number of covalent electron bonds, which have a three-dimensional structure. As a result, the $C_{84}^-$ cluster has been obtained, which is stronger than other fullerenes Fig 2. In mass spectrum is distinguished peaks lines (conditional resonant structures) poorly known fullerene $C_{112}$, $C_{122}$, $C_{146}$, $C_{150}$. At the peak values of $C_{60}$ and $C_{70}$ in Fig.2 held Estimating the surface temperature of fullerite [22]:

$$T = 1032/[0.35+\lg(I_{60}/I_{70})],$$

where $I_{60}$, $I_{70}$ – the magnitude of the peaks of the mass spectrum $C_{60}$ and $C_{70}$, and defined by common logarithm of the ratio of the peaks. Based on the T ≈ 1700 K. But, as can be seen in Fig. 2 shows the mass spectrum of the mass spectrum of cluster are no less $C_{60}$.This shows that the surface temperature of fullerite to 1000 K. On the possible deviations in the temperature calculation surface thin layer fullerite drew attention to the derivation formula [22]. Long term retention of high temperature on the surface of a thin layer of material containing $C_{60}$ and $C_{70}$ having the difference in volatility, changes the ratio of components in the fullerite and respectively (increase) the calculated value of the temperature. The above calculation of the estimated power density in the laser focus for these conditions gives a value of $P \approx 0.1$ MW/cm$^2$. But with such a power density in our experience surface temperature ≤ 1000 K.

**7. The Measurement Results and Discussion of free flow properties of fullerenes**

To determine the values of the electron affinity ($EA_n$) and energy of ionization ($EI_n$) of fullerenes were made calculations. The calculations are based on a rigid model for the structure of the carbon spheres formed from n carbon atoms with an effective radius of the $R_n = n^{0,5}$. The calculations used in a ratio of adjusted:

$$IE_n = \varphi_\infty - K_1/R_n \quad (4)$$

$$\text{and } EA_n = \varphi_\infty + K_2/R_n, \quad (5)$$

where n – number of carbon atoms, $K_1$ and $K_2$ – normalization coefficients, $\varphi_\infty$= 5.37 eV – the work function of graphite sheet. Data for $C_{60}$ were used in the calculations [19]. The results of calculations for noted of heavy fullerenes Fig. 2 are given in the Table.

According Table seen that the ionization energy of clusters decreases with increasing number of carbon atoms, but with the number of carbon atoms increases the electron affinity. Therefore, according to Table between electro-physical parameters of fullerene $C_n$ there is a general relationship:

$$EA_n + IE_n \leq 2\varphi_\infty \quad (6)$$

The values included in the relation (6) is by definition have a different nature, and show that the mass spectra of fullerene differing in sign of charge fullerenes.

The MS of negatively charged fullerenes spectra Fig. 2 obtained at the optimum value of the discharge current is gradually decreases with increasing number of carbon atoms tail, which includes the structure and containing up to 150 and more carbon atoms. In this part of negatively charged ions fullerenes in intensity in 2 to 4 times higher than the background, but for ordinary fullerenes ($C_{60}$, $C_{70}$….) the excess over the background of the peak of the fullerene has a value of 50-60.

MSs recorded from fullerite value obtained when the discharge current I = 80 A, is listed on Fig.3 [23]. The upper part shows the spectrum of fullerene with a negative charge and a positive charge on the lower half. The measurements were performed with the same parameters the laser, but it sets the appropriate polarity of the potential on the extraction electrode. As seen, they have a lower intensity than in Fig. 2 and run these features: intensity peak $C_{84}^-$ greater, than the intensity $C_{70}^-$, and in the spectrum of visible line $C_{150}^-$. But the picture of the spectrum of positively charged fullerenes has a different character. In this spectrum the background intensity is greater than in the spectrum with a negative charge. The range Fig.3 starts with small clusters based on the $C_2^+$ and $C_3^+$. Then, follow consistently lower peaks of $C_{60}^+$, $C_{70}^+$, $C_{78}^+$, $C_{84}^+$ and $C_{100}^+$ near the end. The rest of the spectrum of ions are indistinguishable from background structures. A possible reason for the decline of fullerene ions with a positive charge Fig.3 is to capture free electrons flow evaporating fullerenes in the surface plasma. Therefore, due to the action of the beam evaporation flow of fullerenes is neutral and positively charged fullerenes, but does not contain negatively charged fullerenes. To arrive at this conclusion when analyzing form envelope peaks of the duration of the delay with respect to initial beam and by analogy with a similar picture of the process interaction of a laser beam with a solid [24]. As a result of the capture the part of charged particles is reduced, the flow of ions converted into a stream of neutral fullerenes. Condition charge neutralization fullerene occurs when the energy of a free electron $E_e$ lot less energy of the electron affinity $EA_n$ of the fullerene n carbon atoms: $E_e << EA_n$. Under the terms of the experiment $E_e \approx 0{,}1$ eV and according to Tab. $AE_{84}$=7,23 eV. Table marked with * estimates fullerene ions with a positive charge, the receipt of which is not reliable. Comparison of the spectra shows that the mass spectrum of fullerene negatively charged illustrating more details of the composition of analyte fullerite. Therefore fullerene with a negative charge is preferably used in the processes of electrodynamics.

The calculation NI. For vacuum p =$10^{-5}$ Pa fullerene $C_{150}^-$ on the length L=1•2m and V= 20 KV has a duration of $\tau^{**}$ = 44μs. The work [25] under these conditions was measured the length of the existence of negatively charged fullerene $C_{60}^-$ and fluorine derivatives of the $C_{60}F_{18}^-$ and $C_{60}F_{36}^-$ depending on the energy of ionizing electrons. Measurement have shown that the length of the existence NI is within a few seconds and molecules with a large number of ligands duration longer. Investigated the influence of electrons with energies in the range of 5-20 eV. Therefore, in normal, as in [5, 6], circumstances the duration of existence NI is $\boldsymbol{\tau} > 1$ s.

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