

PROBLEM ON FULLERENE PRODUCTION BY ELECTRIC ARC METHOD

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INTRODUCTION

According to literature data the fullerene synthesis from graphite in the arc discharge is the most successful method. However, mechanism of the fullerene formation from the graphite fragments is still unclear. Therefore potential opportunities of this method are not revealed completely.

It was found that effectiveness of fullerene synthesis depends on many parameters of the process: current density and voltage, buffer gas pressure and its nature, holding time for the fullerene containing carbon plasma at optimum temperature, the construction and geometry of the evaporation chamber, the shape and dimensions of graphite electrodes, the rate of electrode evaporation, the gap between them etc. Changing in the chamber construction and the process parameters leads not only to the change in the process efficiency but the change in the composition of the fullerene fraction.

Fullerene properties, which distinguish them from usual organic substances, appear in the fact that purity of fullerenes and their chemical stability depend on crystallization conditions when fullerenes entrap the solvent molecules. Little amounts of impurities affect different fullerene properties considerably. It was found [1], that the solvent molecules are fixed not only at the crystal surface but in the crystal bulk (especially at raised temperatures). According to [2], fullerene properties depend on the crystal size. At fast crystallization highly dispersed brown-colored powders are formed. In contrast to the large black-colored crystals they contain more impurities due to the developed surface.

EXPERIMENTAL

In this work the fullerene containing soot has been produced on the plant consisting of two communicating chambers for arc evaporation of graphite anodes. In the synthesis every 20 min the voltage fed on the graphite electrodes, the current intensity in the circuit for each electrode pair, the helium pressure have been measured. As synthesis chambers are connected with a pipe, the pressure in both chambers were the same.

This work is aimed at discussion of synthesis condition effect on the fullerene concentration (Q) in the product synthesized.

Effect of the helium pressure (P, Torr) kept in the chamber in synthesis, the current intensity (I, A) and

the synthesis duration ($\Delta\tau$, min) on a value of Q has been studied. When the parameter studied was changed, the rest of them were kept constant. Dependences $Q=f(\Delta\tau)$; $Q=f(P)$ and $Q=f(I)$ have been obtained.

X-ray structure analysis of the fullerenes produced under different temperature conditions has been conducted.

Method for express analysis has been developed. It enables us to determine the fullerene content in soot fairly fast, to a high precision and on minimum amounts of an aliquot part.

The fullerene containing soot in amounts of 0.05 g has been dissolved in 100 ml of toluene at the continuous mixing on the vibrator. Complete fullerene extraction occurs during 1-1.5 hr. After complete extraction from the soot the fullerene concentration in toluene is far from saturated. Therefore dissolving occurs at constant, close to the maximum rate. Dynamics of fullerene extraction has been determined in the individual experiments.

After completion of extraction the concentration of solutions has been identified visually comparing their color with the color of standard solutions in calorimeter.

The concentration of standard solutions has been chosen in such a way that express analysis allows to determine the fullerene yield correct to 0.5 %.

RESULTS AND DISCUSSION

It has been determined that changing in helium pressure by ~ 20 Torr in the reactor during synthesis (relative to an optimum value) leads to the change in the fullerene yield by $2 \div 2.5$ %.

The fullerene yield changes by 2-3 % on changing in the current intensity by $\sim 2 \div 3$ A/cm².

Experimental data have shown that the fullerene concentration depends on the synthesis duration.

It has been noticed that in some cases (at optimum values of current density and pressure kept in synthesis) the fullerene yield has been found low. Authors connect this fact with insufficient purity of initial graphite. Apparently, some impurities escaping from graphite inhibit the fullerene formation what results in decreasing their content in soot.

The found effect consisting in decreasing helium pressure during the fullerene synthesis is of

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interest. It begins in 20-40 min after the arc ignition. This effect has been observed in all experiments without exception.

It has been found that the purity of crystalline fullerenes produced depends on the temperature conditions under which the fullerene extraction from the fullerene containing soot and evaporation of the fullerene solutions have been conducted. Four types of fullerene crystals have been produced:

Crystals I: a) extraction has been carried out at 12 °C, b) evaporation had been conducted on the rotary evaporator under vacuum at 35-40 °C.

Crystals II: a) extraction - at boiling point in the flask with a reflux condenser, b) evaporation of the solution - at boiling point.

Crystals III: a) extraction - in the Soxhlet apparatus, b) evaporation - at boiling point.

Crystals IV: a) extraction - in the Soxhlet apparatus, b) evaporation - on the rotary evaporator at 35÷40 °C.

The fullerene purity has been estimated by results obtained by X-ray structure analysis of the fullerene crystals on ДРОН-3 X-ray unit.

The crystals of fullerene I has been found the purest. Crystals II contain the largest amounts of impurities. It may be explained by the fact that at high temperatures, especially at boiling, under atmosphere pressure the fullerene adduct formation occurs with the solvent molecules and probably with some impurities. In toluene evaporation they are adsorbed not only at the crystal surface but in the intercrystallite space and the lattice volume.

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