

ABOUT SOME SPECIAL FEATURES OF FULLERIT MANUFACTURE BY MEANS OF AN ARGON ARC DISCHARGE

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Introduction

The most widespread method of fullerenes and carbon nanotubes manufacture is an arc discharge in a buffer gas. Helium is usually applied as the gas. Argon is the more widespread and cheap gas. But the fullerene output is less than 2 % with that gas whereas in a helium arc discharge it was about ten times greater. We have worked out the effective method of fullerene preparation in a small quantity by means of an argon arc discharge [1-3]. This report is informed about the further investigation in this direction.

Results and discussions

The fullerene structures synthesis is realized into the water-cooling discharge chamber [1] in the regime of direct current $I = 50$ A and voltage $U = 20 \dots 25$ V. A graphite plate with dimensions $30 \times 15 \times 5$ mm was as a cathode, and a graphite rod with a diameter 6 mm was as an anode. Experiments are conducted with the argon pressure varying from $1 \cdot 10^4$ to $7 \cdot 10^4$ Pa. The shoot to be formed in an arc discharge and to be covered the walls of the discharge, the cathode surface and the anode holder surface was the investigating object. X-ray diffraction analysis of the samples were performed on a DRON-4-07 apparatus with Cr K_α radiation. As a monochromator, we applied a crystal of pyrolytic graphite.

The transmission spectrums of pressed shoot tablet in KBr are measured on a spectrophotometer Specord M80.

In our experiments we observed visual forming of the cathode deposit in the form of a cylinder at all used argon pressure. That ledge had grown on the cathode surface opposite the anode and its diameter was nearly equal to the anode profile. In a considerable part of events there were the regime at which an increment of the deposit height was not accompanied by an electrode locking. The strongly depended upon a discharge current and a gas pressure. The average specific solidity of a ledge to be measured by dividing its mass to geometric volume was about $1,3 \cdot 10^3$ kg/m³. Maximum length of the grown cylinder was 65 mm.

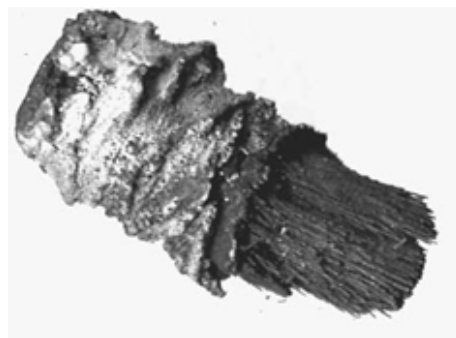


Fig. 1. Photograph of a ledge-deposit fracture.

A photograph of a ledge-deposit fracture is given in Fig. 1. It is seen that the inner part of the formation is the super loose structure to have in large part been generated from the carbon filaments. The outer surface of the ledge-deposit has been formed by the ring-like carbon structure. We suppose the structures of that sort to be the perspective materials for a hydrogen accumulation.

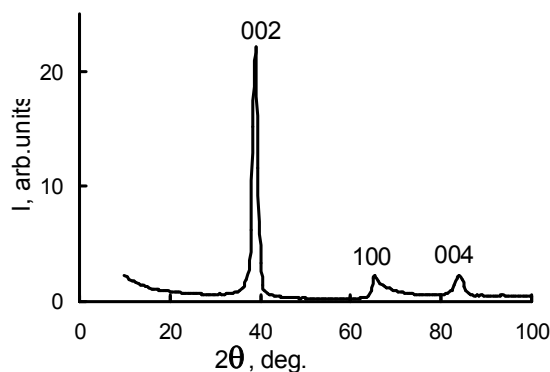


Fig.2. X-ray diffraction a cathode deposit.

In Fig. 2 the X-ray diffraction one of a ledge-deposit is shown. One can see the characteristic X-ray peaks of hexagonal graphite.

The grown ledge is fixed in the special anode holder and it is used for a second dispersal in the arc. Our investigations have been shown that the fullerene yield had been more in the case of the anode from cathode deposit than it had been a graphite [2].

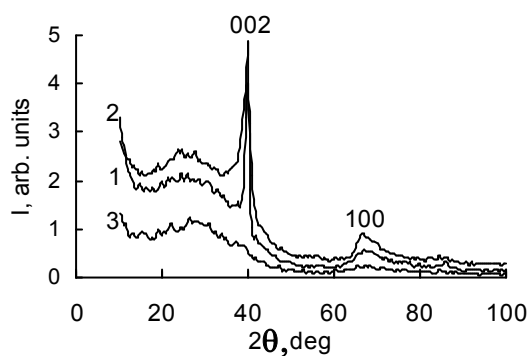


Fig. 3. X-ray diffractions the shoots.

In Fig. 3 the X-ray diffractions of the shoot to be formed on the walls of the discharge chamber (curve 1), on the cathode surface (curve 2) and on the anode holder surface (curve 3) are shown. In all curves it is seen the amorphous halo at the angle region $2\theta \approx 15...40^\circ$ and the characteristic graphite reflex. On a background of the halo it is seen the weak peaks a most part of which coincided with the most intensive peaks of BCC-structure of fullerit C_{60} . It should be emphasized that the toluene tincture of the shoot to be collect from the water-cooling walls of the chamber is been shown alone the characteristic red-brown coloring. We consider these data to testify forming on the electrode's surfaces a toluene insoluble chemical compound of the fullerene molecules.

The fulleren-containing (colored) infusion of toluene are put into the evaporating cell and it is carried the toluene evaporating at the temperature $T = 500 \text{ K}$.

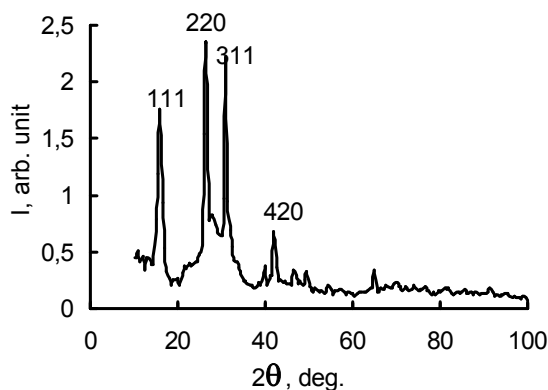


Fig. 4. X-ray diffraction extract.

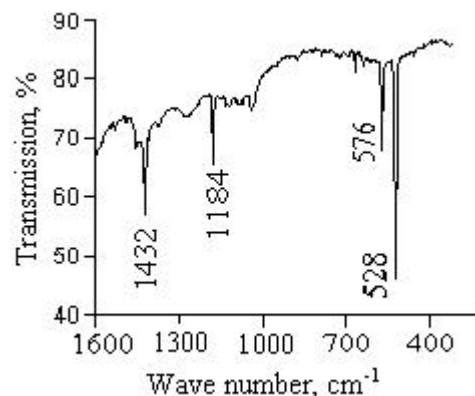


Fig. 5. IR-spectrum extract.

Fig. 4 shows the X-ray diffraction of extract to be obtained and Fig. 5 shows IR-spectrum of this extract. The BCC-lattice reflexes of C_{60} fullerit are identified on the diffraction. The IR-spectrum contains the peaks to be corresponded to the C_{60} and C_{70} crystals.

Conclusions

- (1) The cathode deposit forming in the form of cylinder has been obtained.
- (2) Fulleren-similar structures to be not dissolved in toluene are shown to be formed on the several parts in the discharge chamber.

References

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