Growth of Carbon Nano-Tubes (CNT) in Electric-Arc Discharge in Argon

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Electric-arc discharge deposition was used to produce CNT products. Design and electric scheme are discussed in details, as well as the technological features. On the basis of DTA-TGA results there are discussed the outputs of all technological schemes applied. It's established that maximum CNT formation is in the form of compact cathode deposits ("stubs"), but the latter has restricted conditions of formation, that may be some of difficulties in scaling of CNT production.

As a main apparatus for the arc-discharge growth we used a slightly modified installation for the Czochralski-crystal growth. Inside the growth camera of this furnace there was installed a special graphite construction for producing of high-temperature plasma in the direct current discharge arc in the Ar atmosphere at controlled pressure. Some experiments were carried out with dopants that were powders of pure metals (Fe, Ni, Co) mixed with graphite powder to give the dopant/anode concentration of 0,5-3 % at.

As an express-method for the analysis of carbon deposits we used DTA-TGA method. The experiments confirmed known from the literature (see, e. g. [1]) temperature intervals of burning different deposit components: the amorphous carbon nano-particles were burnt before 500 °C, fullerenes – before 650 °C, nano-tubes – before 750 °C, and crystalline graphite was oxidized at 800-900 °C.

The stub deposit was formed in the very narrow range of voltage and current, and at a definite inter-electrode gap. The gap size was strictly bound by the nature of the process and was automatically established and self-supported on the same meaning during the stub growth. All the attempts to lower or to increase the gap led either to short-cut or to ceasing of stub formation. Maximum length of the stub, produced by this process, was 20-35 mm, with ~ 6,1 mm in diameter, whereas mass loss, accounted for the soot and loose cathode deposit, was about 20%. Average stub growth rate was 12.4±0.5 mm/min.

At the Co-doping with concentration ≤ 3 at. % we did not find any noticeable difference in the arc-discharge regimes in comparison with those without dopant. At the Co-concentration of 3 % at, in the core of the stub the small droplets of metal were found, that were the evidence of incomplete metal evaporation and its interaction with carbon vapor. At Ni-doping (0,5 % at.), the length of the stub produced was about 42 mm.

Using of the additional graphite screens permitted us to increase the stub length up to 50 mm at the same arc-discharge parameters. It was also found that the screens geometry influences the deposit composition.

Analysis of carbon deposits by DTA-TGA showed that the maximum nano-tube content was in the stubs, both doped and undoped, though the rate of deposit formation in the form of stubs is next to the lowest (Fig. 1). Nevertheless, the stub growth rate in argon is 10 times higher than that in helium at similar arc discharge parameters. The possibilities of the arc discharge optimization are discussed. It is found that the stub grows in argon only at very narrow set of parameters (inter-electrode gap, current, and gas pressure). The CNT content in the body of the stub may be as high as 80 %.

Reference

1. Zhang H., Wang D., Xue X. et al. The effect of helium gas pressure on the formation and yield of nanotubes in arc discharge. J. Phys. D 1997, 30(3); L1.

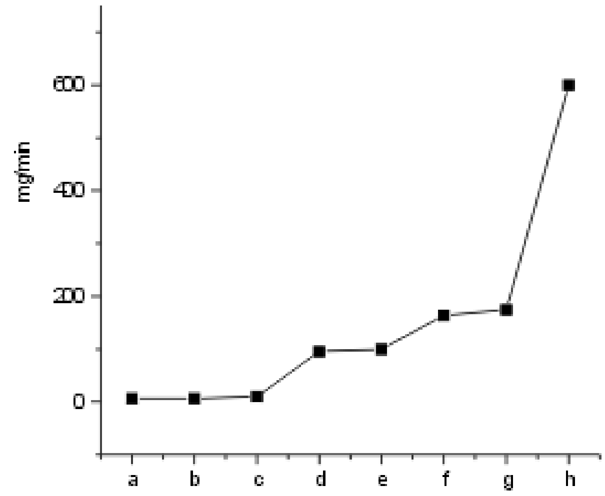

Fig.1. Rate of formation (mg/min) for different arc deposits; I - current: a - cathode deposit undoped (I=60 A); b - cathode deposit doped (I=60 A); c - cathode deposit undoped (I=170 A); d - stub doped; e - stub undoped, f, g - cathode deposit (different screens applied); h- soot from reactor walls, cathode deposit with insulated screen.

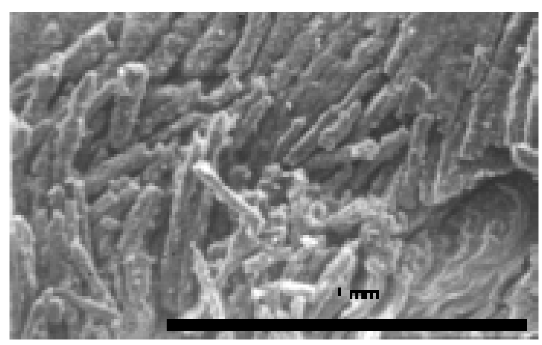

Fig.2. CNT-bundles revealed by SEM.

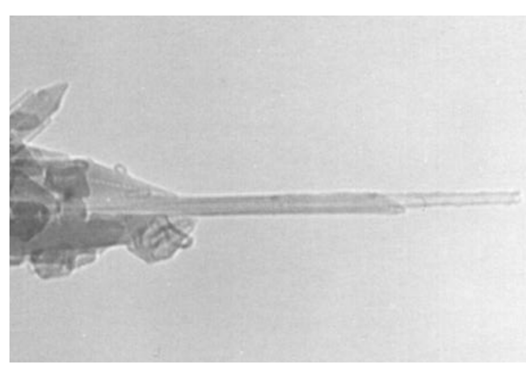

Fig. 3. Multiwall CNT by TEM.