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CARBON NANOSTRUCTURES SYNTHESIS IN MICROWAVE PLASMA TORCH AT ATMOSPHERIC PRESSURE

O. Jašek[a], M. Eliáš[a], L. Zajíčková[a], Z. Kučerová[a], J. Matějková[b], A. Rek.[b], J. Buršík[c]

[a]Dept. Physical Electronics, Masaryk University, Kotlářská 2, Brno 611 37, Czech Republic
[b]Institute of Scientific Instruments, ASCR, Královopolská 147, Brno 612 64, Czech Republic
[c]Institute of Physics of Materials, ASCR, Žižkova 513/22, Brno 61662, Czech Republic

Although the most important form of bonded carbon seems to be sp^3 hybridization in case of hard materials the "graphite" sp^2 hybridization can be found in many interesting carbon nanostructures. Carbon nanotubes (CNTs) [1] can be described as set of one or more concentric cylinders created by rolled up graphene sheet. Plasma enhanced chemical vapor deposition (PECVD) [2] belongs to the most promising methods for synthesis of CNTs. We have successfully synthesized CNTs in microwave plasma torch at atmospheric pressure from the mixture of methane, hydrogen and argon [3]. The plasma torch was generated at the frequency of 2.45 GHz using an iron hollow electrode. Argon (1000 sccm) was flowing through the electrode whereas methane (10-40 sccm) and hydrogen (100-400 sccm) were added to the expanding torch from outside. The CNTs were grown on substrates placed at various distances from the torch electrode. The substrates were silicon wafers (10 x 15 mm) with deposited 200 nm thick silicon oxide layer and thin layer (5-20 nm) of iron catalyst on the top. The samples were imaged by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The processes in the discharge were monitored by optical emission spectroscopy (OES). The SEM analysis showed presence of well aligned 50 μm long multi-wall CNTs with narrow diameter distribution centered around 20 nm. Except the carbon nanotubes the layers often contained various carbon and carbon/iron structures. This deposition inhomogeneity is in our opinion caused by temperature gradients on the substrate and local influence of the discharge itself. The plasma column diameter is only around 5 mm depending on the exact gas flow values. It also can not be excluded that high energy ions and UV radiation has a negative influence on the CNTs growth at the certain substrate parts. The OES revealed the presence of excited Ar and H atoms and CH, C_2 and CN radicals in the discharge. Especially the high C_2 band intensity lead us to the conclusion that high carbon amount could lead to CNTs growth process termination due to so called catalyst poisoning i.e. state, when the catalytic particle is completely covered with amorphous carbon and surface catalytic reaction is stopped. This process lead in our experiments to complete overlaying of CNTs with carbon/metal overlayer. We were partially able to eliminate this negative effect by shortening the deposition time from 15 minutes to 1 minute or less. Gas flows, metal catalyst layer thickness and temperature were also optimized to achieve cleaner CNTs layers, which could be directly used in various applications e.g. CNTs/polymer composites.

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References

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