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GROWTH AND CHARACTERIZATION OF CARBON NANOTUBES USING DIFFERENT CATALYSTS

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ABSTRACT

The paper presents results related to the growth of carbon nanotubes (CNTs) on different catalyst by chemical vapour deposition under atmospheric pressure using ethylene as the carbon source. This study investigates the influence of different types of metallic catalysts and the process parameters (catalyst annealing, process temperature and time of growing process) on the CNT formation. The morphology and microstructures of the products were investigated by atomic force microscopy, field emission scanning electron microscopy, high-resolution transmission electron microscopy.

EXPERIMENTS

CNTs were synthesized by CVD growth on silicon substrate (100) type n and silicon (100), type n with SiO_2 (500 nm) with catalyst layer based on iron or nickel, obtained through e-beam and magnetron sputtering methods and also on iron ribbons obtained by melt spinning, according to the table below. Prior to the CNT growth process, catalyst annealing was carried out at a temperature of 750 C for 15 minutes in an inert atmosphere inside the quartz tube of the CVD equipment (table 1). Within the CNT growth experiment by CVD method, C_2H_4 was used as gas source of carbon. General parameters of CVD process for nanotubes growth are presented in table 2.
The obtained samples were characterized by different microscopy techniques (AFM, SEM and HRTEM).

Table 1. Catalysts annealing process parameters

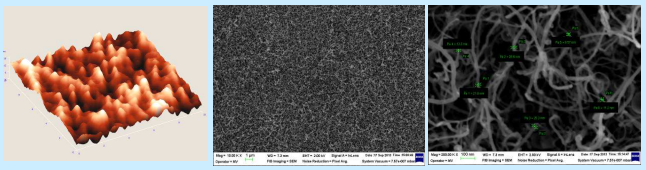
Temperature [C]	Time [minutes]	Pressure [torr]	H_2 flow [cm^3/min]
750	15	760	500

Table 2. CNT samples and CVD process parameters

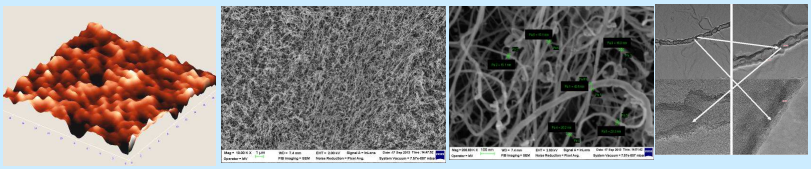
Sample	Substrate	Catalyst	Obtaining method	Temperature [°C]	Time [minutes]	Pressure [torr]	Ar flow [l/min]	H_2 flow [cm^3/min]	C_2H_4 flow [cm^3/min]
A	Silicon (100), type n	Fe	e-beam	750	300	760	1	400	300
B	Silicon (100), type n with SiO_2 layer (500 nm)			750	300	760	1	400	300
C	Silicon (100), type n with Ti layer	Ni	e-beam	750	300	760	1	400	300
D	Silicon (100), type n with SiO_2 layer (500 nm)			750	300	760	1	400	300
E	Iron ribbon	Fe	melt spinning	750	300	760	1	400	300
F	Silicon (100), type n	Fe	magnetron sputtering	750	300	760	1	400	300

RESULTS

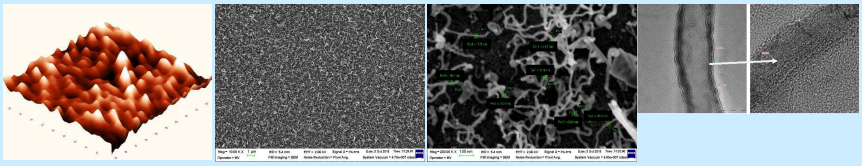
➢ **Random oriented MWCNTs**



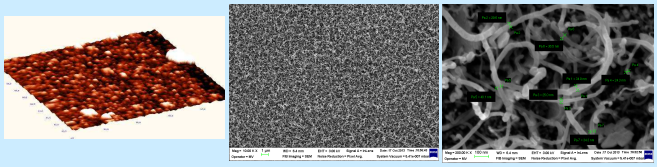
AFM and SEM images of MWCNT (Fe catalyst annealed at 750°C on Si substrate) (sample A)



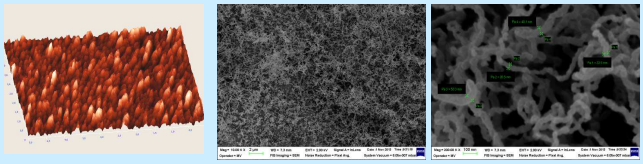
AFM, SEM and HRTEM images of MWCNT (Fe catalyst annealed at 750°C on Si / SiO_2 substrate) (sample B)



AFM, SEM and HRTEM images of MWCNT (Ni catalyst annealed at 750°C on Si substrate) (sample C)

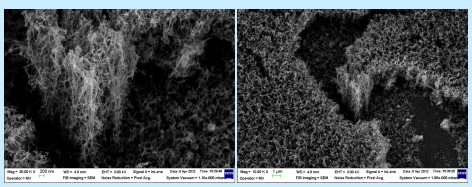


AFM and SEM images of MWCNT (Ni catalyst annealed at 750°C on Si / SiO_2 substrate) (sample D)

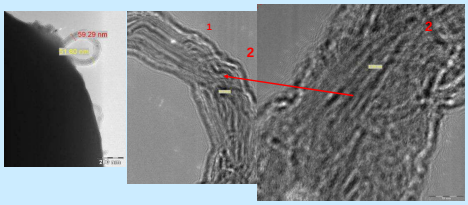


AFM and SEM images of MWCNT (Ni catalyst annealed at 750°C on Si / SiO2 substrate) (sample E)

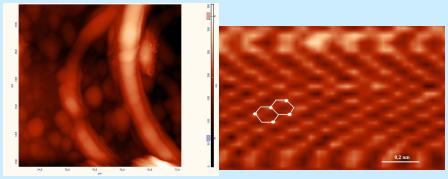
➢ **Vertically Aligned Carbon Nanotubes (VACNTs)**



SEM images of VACNTs (Fe catalyst on Si substrate) (sample F)



HRTEM images of a single CNTs (Fe catalyst on Si substrate)



AFM/STM images of CNTs (Fe catalyst on Si substrate)

CONCLUSIONS

- **The coating silicon substrate with a protective layer of SiO_2 favors the growth of multi-walled CNT at 750°C using C_2H_4 as carbon source;**
- **CNT obtained with Fe catalyst obtained by magnetron sputtering tend to align;**
- **The growth of CNT on Ni catalyst is favored by a catalytic layer thickness of 11 nm;**
- **The distance between two adjacent graphene planes, determined by measuring the thickness of the 10 adjacent planes in CNT wall by HRTEM, was 3.48 Å for the sample B and 3.45 Å for the sample C, versus 3.35 Å - the typical distance between two adjacent planes in graphite.**

ACKNOWLEDGEMENT

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