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Research Article

INFLUENCE OF CARBON SOURCE ON SYNTHESIS OF CNT USING

CHEMICAL VAPOR DEPOSITION

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ABSTRACT

Carbon nanotubes were synthesized by chemical vapor deposition using different carbon sources (isopropanol, ethyl alcohol and low density polyethylene (LDPE)) on magnesium oxide supported on Co. Nanotubes synthesized by chemical vapor deposition were characterized by scanning electron microscopy (SEM) and Raman spectroscopy (RS). Owing to these changes, diverse selectivity towards nanotubes, morphologies, purity degrees and crystallinity are obtained, as evidenced by the characterization results.

Keywords: carbon nanotubes, CVD, SEM.

INTRODUCTION

The carbon nanotube (CNTs) has taken its own place in the field of nanotechnology after the discovery by lijima in 1991¹. The unique properties of CNTs like high aspect ratio, good thermal conductivity, high mechanical strength and chemical stability², has explored its application in hydrogen storage³, field emitting device⁴, nanoelectronic devices⁵, lithium battery electrode material⁶. Till today the widely used CNTs synthesis techniques are arc discharge, laser ablation and chemical vapour deposition⁷. The first two methods can produce high quality CNTs but limited to research and not adaptable to the industrial production. But the chemical vapour deposition produces CNTs at low cost, easy to scale up, good control of the synthesis parameters⁸. The quality of CNTs depends on catalyst type, precursor gas, temperature, reaction time, carrier gas, gas flow rates⁹. The active catalyst for the CNTs synthesis contain the transition metals like Fe, Co, Ni, Pt, Mo, silicon or their alloys¹⁰. These transition metals can decompose the carbon source such as methane, acetylene and benzene¹¹.

J.P.Pinherio et al.¹² reported the CNTs synthesis using Co/MgO with carbon monoxide as carbon source and optimized the yield with Co composition. Hui yang et al¹³ used ethanol as carbon source for the synthesis of CNTs in contact with 316-type stainless steel and observed the different morphology of CNTs. G.ortega et la.¹⁴ Studied the CNTs formation using ethanol as carbon source over different substrate like conducting glass, quartz plate, nickel plate with Co and Fe as catalyst. They have reported that the CNTs formation depends on substrate temperature and ethanol exposure time. H. Ryu et al¹⁵ used the various combination of Co-Ni/MgO to see their effect on yield and quality of CNTs with acetylene gas as carbon source. Villas Ganpat Pol¹⁶ used the various plastic wastes to synthesize carbon microsphere.

The objective of this research to compare ethanol, isopropanol and waste plastic as crabon source on Co/MgO catalyst in simple CVD system. The main purpose of using plastic waste is to replace toxic gases which are difficult to handling and to convert plastic waste into an advantageous product. The structure of as synthesized carbon nanotubes are characterized by scanning Electron Microscopy (SEM) and Raman spectroscopy.

Experimental details

The CNTs were synthesized by chemical vapor deposition method (CVD) of a gas mixture evaporated from catalyst powder. The schematic diagram of experimental set up as shown in Figure.1. The main body of the reactor made of quartz glass with ID of 5cm and height of 50 cm. The bottom end of the reactor is attached with silicon crucible and the upper end with gas inlet. The electrical heater was mounted around the bottom part of the reactor and the gas flow rates were measured by mass flow metres.

Catalyst Preparation

The impregnation method was used to prepare the supported catalyst Co/MgO. The MgO of 20 g was suspended in 100ml of ethanol and 5wt% cobalt nitrate was dissolved in 100 ml of ethanol separately. Then the solution of MgO and Cobalt Nitrate were mixed together and sonicated for 20 min. then the solution was kept in an oven at 90°C for 24 h to ensure the catalyst retained no water. Following the heat treatment, the mixture was grounded finely to get fine powder with an average diameter of about 5-6µm. Fig. 2 show the finely distributed catalyst powder.

Synthesis of CNTs

The catalyst was loaded in quartz reactor. Argon was purged into the reactor for 20 min to create an inert atmosphere, and then the reactor was heated to 550°C with hydrogen flow. After attaining 550°C the carbon source was introduce with argon as carrier gas for 60 min. The reaction was terminated by shutting the supply of carbon source and reactor was cooled to room temperature with argon gas. The synthesized product is taken from reactor and analyse the sample.

For the synthesis of CNTs using waste plastic as carbon source, the reactor was loaded with catalyst and the small pieces of waste plastic like waste polyethylene bags. Then the argon gas was purged to induce the inert atmosphere. The reactor was heated with hydrogen gas to 550°C for 60 min. the reaction was terminated by switching the flow of hydrogen and the reactor was cool to room temperature with argon gas. The produced product was characterized using SEM and Raman spectroscopy.

RESULTS AND DISCUSSION Waste plastic as the carbon source

The CNTs synthesized from waste plastic is as shown in fig.3. The SEM image indicates the poor structure of CNTs and high degree of buckling. The Raman spectra analysis indicates the CNTs with high degree of impurities. The formations of CNTs are confirmed with two distinct peaks on the Raman spectra. The graphitization of CNTs was observed by using Raman spectroscopy as shown in Fig.4. The Dband, observed at 1347 cm⁻¹ is known as the disorder induced due to either the wall disorder or the presence of amorphous carbon deposited on the outer surface of nanotubes. The G-band (observed at 1588 cm⁻¹) can be attributed to the degree of crystallinity of carbon nanotubes. The ratio of In/ Ig indirectly indicates the crystalline order of carbon nanotubes¹⁷. The obtained I_D / I_G ratio 1.1 for this carbon sample indicate that the defects level in the nanotubes structure is more. This may be due to the softening point of the LDPE when introduced into the reactor and melt over the catalyst. During the pyrolysis, CNTs grows from the surface due to the degradation of LGPE and release of volatile gases. The high degree of buckling of CNTs may because of nonsufficient pyrolysis process¹⁸.

Isopropanol as carbon source

The SEM image in Fig.5 shows that the CNTs are produced using Isopropanol with cluster of metal catalyst. The diameter of CNTs is in the range of 61 to 132nm. The Raman spectra as shown in Fig.6 reveal the I_D/I_G ratio of 1.3 which indicates that the synthesized products are of high defects containing large amount of impurities which is in good agreement with the SEM results. The larger diameter of CNTs may be due to low temperature of pyrolysis.

Ethanol as the carbon source

The SEM image in the Fig.7 shows the CNTs produced with the diameter in the range of 48 to 80 nm. The graphitization of CNTs was observed by using Raman spectroscopy as shown in Fig.8. The obtained I_D/I_G ratio ≈ 0.75 indicate that the defects level in the nanotubes structure is less.

CONCLUSION

In this work, it has been shown that CNTs can be synthesized from waste plastic, isopropanol and ethanol as carbon source. In order to understand the conversion of CNTs various parameters need to be addressed. The future challenge is to synthesize the high degree CNTs from waste plastic with different operating parameters in order to eliminate the environment pollutant.



Fig. 1: Experimental set up of CVD for CNT synthesis



Fig. 2: SEM image of finely distributed Co/MgO catalyst



Fig. 3: CNTs synthesized from waste plastic as the carbon source



Fig. 4: Raman Spectra of as produced CNTs using waste plastic



Fig. 5: SEM image of produced CNTs from Isopropanol





Fig. 7: SEM image of CNTs produced from ethanol



ethanol as feed stock

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