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Synthesis of Carbon nanotubes by catalytic decomposition of Ethyne using Co supported on CaO

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Carbon nanotubes were synthesized by catalytic chemical vapor deposition (CCVD) over cobalt catalysts on calcium oxide support. The catalyst was synthesized by co-precipitation of the metal nitrates. Scanning Electron Microscopy (SEM), thermo gravimetric (TGA) and energy dispersive spectroscopy (EDS) indicate the presence of carbon nanotubes.

Introduction

Carbon nanotubes, long thin cylinders of carbon, were discovered in 1991 by Iijima's. Carbon nanotubes (CNTs) are allotropes of carbon which are members of the fullerene structural family[1]. Carbon nanotubes are one of the most important materials of nanotechnology. With one hundred times the tensile strength of steel, thermal conductivity better than all but the purest diamond, and electrical conductivity similar to copper, but with the ability to carry much higher currents, they are considered as a superior material.

Synthesis of carbon nanotubes can be done by different methods such as Arc discharge method Laser ablation method ,Chemical vapor deposition method ,plasma enhanced chemical vapor deposition ,Thermal chemical vapor deposition ,Vapor phase growth[2]. The CVD process is easy to scale up and also the growth can be controlled[3]. Among the parameters affecting the growth of the carbon nanotubes is the support, as the type of the support affect the final morphology of the obtained carbon nanotubes.[4]. Different supports have been used in preparation of the catalysts such as alumina, zeolite, magnesium oxide and calcium oxide.[5]. However Calcium containing supports have been shown to increase the yield of the obtained carbon nanotube[6].The other fact which should be considered is the gas as well, different carbonous gas has been reported and the most common are methane, ethylene , acetylene [7]and also carbon dioxide[8] and carbon monoxide[9] as well. Depending on the all mentioned factors carbon nanotubes from cobalt transition metals catalysts can be single walled [10],multi walled [11] or even using tri metallic forms of cobalt and its transition metals groups it has formed

bamboo shaped carbon nanotubes[12].So in this study Cobalt supported on CaO has been reported to produce Carbon nanotubes.

Experimental

All the materials were bought from Sigma Aldrich and used as received. The synthesis methodology was modified method of Allaedini et al [13, 14] 20 mmol cobalt salt (Co $(NO_3)_2 \cdot 6H_2O$) and 20 mmol CaO was dissolved into deionized water containing dispersant polyethylene glycol (PEG), then drops of NH_4OH was added with electromagnetic stirring at 50 ◦C . A certain volume of 30% H_2O_2 was dropped into the above suspension. Finally, these solutions were transferred into a centrifuge. Then they were put into an oven to be heated at 100◦ C and finally Calcined at 600 C. The Calcination was done as its reported affecting the morphology of the obtained Carbon nanotubes[15].

In order to obtain Carbon nanotubes the prepared catalyst boat was placed in the centre of the quartz tube. The reaction was carried at atmospheric pressure with ethyne gas as the carbon source. Decomposition of the hydrocarbon source was at 800 C. The Nitrogen gas flowing at 100 ml min^{-1} was passed through the reactor for approximately 45 min., and after it was .The Ethyne gas with a flow rate of 150 mL min^{-1} ,controlled by mass flow meter ,was then passed through the reactor for 60 min.Then the reactor was cooled down with nitrogen flowing at 20 mL min^{-1} for 1hour. The samples were collected and characterized.

Characterization

The SEM micrograph of the obtained cobalt catalyst is shown in figure 1. The catalyst has spherical morphology. The image shows homogeneous dispersity of the catalyst with large surface area suitable for catalytic reaction.

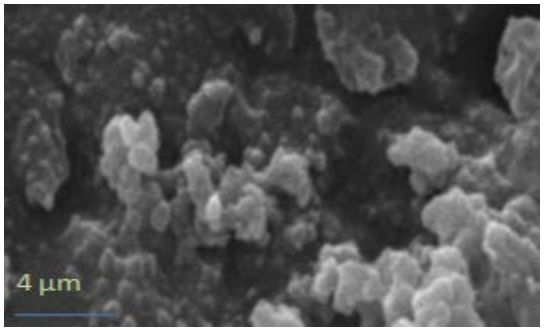

Figure 1- SEM image of the obtained Co/CaO

Figure 2 shows the EDS spectra of the obtained sample after the reaction .As cab be seen the Peak for the carbon is sharp. The element weight percentage has also been shown in table 1 illustrating the weight percent of the 41.67% indicating quite a high yield of carbon deposition .

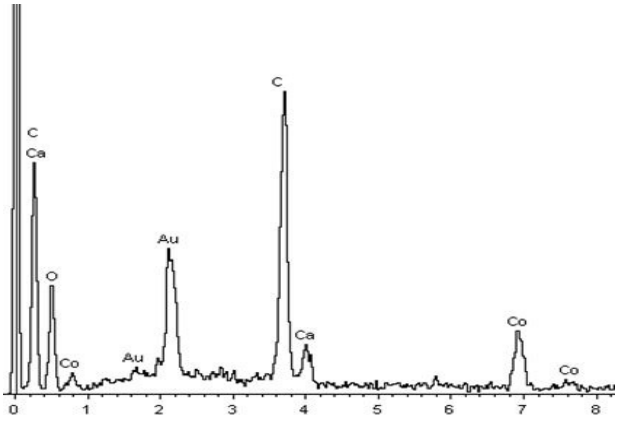

Figure 2- EDS spectra of the obtained sample after reaction.

Element	Weight%	Atomic%
C K	41.67	59.86
O K	26.49	28.56
Ca K	16.37	7.05
Co K	15.48	4.53
Totals	100.00	

Table 1- Weight and atomic % of the obtained sample after reaction.

Figure 3 shows the SEM micrograph of the obtained carbon nanotubes. The tube- like structures which are grown amidst the catalysts is the carbon nanotubes. The TGA of the obtained carbon nanotubes is illustrated in figure 4. Oxidative temperatures more than 450 C were reported to be mainly attributed to crystalline carbon with a high degree of graphitization[16] .So no weight loss is noted below 450 C. The TGA Graph shows about 12% of the weight loss for the obtained CNT and about 88% for the metals and the support.

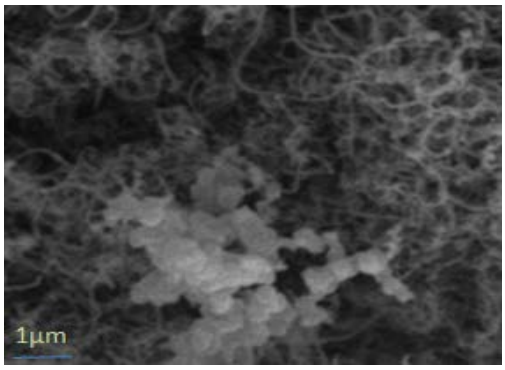

Figure 3- SEM image of the grown Carbon nanotubes amidst the Co/CaO catalysts.

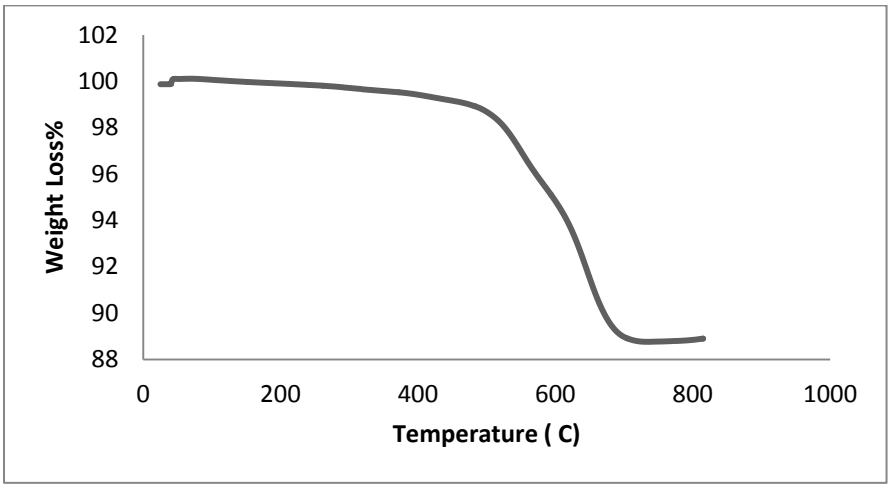

Figure 4- TGA of the obtained sample after reaction

Conclusion

In this work Co supported on CaO catalyst was synthesized, and employed for carbon nanotube production. The carbon nanotubes have been synthesized by the decomposition of ethyne over Co/CaO catalyst. The SEM, EDS and TGA results confirmed the structural properties and the presence of the carbon nanotubes showing that Co/Cao can be used successfully for production of carbon nanotubes by decomposition of ethyne.

References

[1] J.-M. Bonard, H. Kind, T. Stöckli, and L.-O. Nilsson, "Field emission from carbon nanotubes: the first five years," *Solid-State Electronics,* vol. 45, pp. 893-914, 2001.

[2] A. Chatterjee and B. Deopura, "Carbon nanotubes and nanofibre: an overview," *Fibers and Polymers,* vol. 3, pp. 134-139, 2002.

[3] R. Xiang, Z. Yang, Q. Zhang, G. Luo, W. Qian, F. Wei*, et al.*, "Growth deceleration of vertically aligned carbon nanotube arrays: Catalyst deactivation or feedstock diffusion controlled?," *The Journal of Physical Chemistry C,* vol. 112, pp. 4892-4896, 2008.

[4] G. Allaedini, P. Aminayi, and S. M. Tasirin, "The Effect of Alumina and Magnesia Supported Germanium Nanoparticles on the Growth of Carbon Nanotubes in the Chemical Vapor Deposition Method," *Journal of Nanomaterials,* vol. 501, p. 961231, 2015.

[5] K. MacKenzie, O. Dunens, and A. T. Harris, "A review of carbon nanotube purification by microwave assisted acid digestion," *Separation and Purification Technology,* vol. 66, pp. 209-222, 2009.

[6] H. Kathyayini, N. Nagaraju, A. Fonseca, and J. B. Nagy, "Catalytic activity of Fe, Co and Fe/Co supported on Ca and Mg oxides, hydroxides and carbonates in the synthesis of carbon nanotubes," *Journal of Molecular Catalysis A: Chemical,* vol. 223, pp. 129-136, 12/1/ 2004.

[7] Q. Li, H. Yan, J. Zhang, and Z. Liu, "Effect of hydrocarbons precursors on the formation of carbon nanotubes in chemical vapor deposition," *Carbon,* vol. 42, pp. 829-835, 2004.

[8] G. Allaedini, S. M. Tasirin, and P. Aminayi, "Synthesis of CNTs via Chemical Vapor Deposition of Carbon Dioxide as a Carbon Source in the Presence of NiMgO," *Journal of Alloys and Compounds,* 2015.

[9] M. J. Bronikowski, P. A. Willis, D. T. Colbert, K. Smith, and R. E. Smalley, "Gas-phase production of carbon single-walled nanotubes from carbon monoxide via the HiPco process: A parametric study," *Journal of Vacuum Science & Technology A,* vol. 19, pp. 1800-1805, 2001.

[10] A. Moisala, A. G. Nasibulin, and E. I. Kauppinen, "The role of metal nanoparticles in the catalytic production of single-walled carbon nanotubes—a review," *Journal of Physics: condensed matter,* vol. 15, p. S3011, 2003.

[11] A. Fonseca, K. Hernadi, P. Piedigrosso, J.-F. Colomer, K. Mukhopadhyay, R. Doome*, et al.*, "Synthesis of single-and multi-wall carbon nanotubes over supported catalysts," *Applied Physics A,* vol. 67, pp. 11-22, 1998.

[12] G. Allaedini, S. M. Tasirin, P. Aminayi, Z. Yaakob, and M. Z. M. Talib, "Bulk production of bamboo-shaped multi-walled carbon nanotubes via catalytic decomposition of methane over tri-metallic Ni–Co–Fe catalyst," *Reaction Kinetics, Mechanisms and Catalysis,* pp. 1-12.

[13] G. Allaedini, S. M. Tasirin, and M. Pudukudy, "Effect of PH on Cobalt Oxide Nano Particles Prepared by Co-Precipitation Method," 2014.

[14] G. Allaedini, S. M. Tasirin, and P. Aminayi, "Magnetic properties of cobalt ferrite synthesized by hydrothermal method," *International Nano Letters,* pp. 1-4.

[15] G. Allaedini, S. M. Tasirin, J. Sahari, M. Talib, and M. Zainal, "The Effect of Co/Pd MgO Supported Catalyst Calcination Temperature on the Yield and Morphology of CNTs via Methane Decomposition," in *Advanced Materials Research*, 2014, pp. 148-151.

[16] A.-H. Lu, W.-C. Li, E.-L. Salabas, B. Spliethoff, and F. Schüth, "Low temperature catalytic pyrolysis for the synthesis of high surface area, nanostructured graphitic carbon," *Chemistry of materials,* vol. 18, pp. 2086-2094, 2006.