Diamond & Related Materials 48 (2014) 52–59

Contents lists available at ScienceDirect

Diamond & Related Materials

journal homepage: www.elsevier.com/locate/diamond

Single stage production of carbon nanotubes using microwave technology

N.M. Mubarak [a,b], J.N. Sahu [c,*], E.C. Abdullah [d], N.S. Jayakumar [a], P. Ganesan [e]

[a] Department of Chemical Engineering, Faculty of Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia
[b] Department of Chemical and Petroleum Engineering, Faculty of Engineering, UCSI University, Kuala Lumpur 56000, Malaysia
[c] Petroleum and Chemical Engineering Programme Area, Faculty of Engineering, Institut Teknologi Brunei (A Technology University), Tungku Gadong, P.O. Box 2909, Brunei Darussalam
[d] Malaysia–Japan International Institute of Technology (MJIIT), Universiti Teknologi Malaysia, Jalan Semarak, 54100 Kuala Lumpur, Malaysia
[e] Department of Mechanical Engineering, Faculty of Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia

ARTICLE INFO

Article history:
Received 13 March 2014
Received in revised form 17 April 2014
Accepted 8 July 2014
Available online 15 July 2014

Keywords:
Carbon nanotubes
Chemical vapor deposition
Microwave technology
Ferrocene
Acetylene
Hydrogen

ABSTRACT

Carbon nanotubes (CNTs) were produced by gas phase single stage tubular microwave chemical vapor deposition (TM–CVD) using ferrocene as a catalyst and acetylene (C_2H_2) and hydrogen (H_2) as precursor gasses. The effect of the process parameters such as microwave power, radiation time, and gas ratio of C_2H_2/H_2 was investigated. The CNTs were characterized using scanning and transmission electron microscopy (TEM), and by thermogravimetric analysis (TGA). Results reveal that the optimized conditions for CNT production were 900 W reaction power, 35 min radiation time, and 0.6 gas ratio of C_2H_2/H_2. TEM analyses revealed that the uniformly dispersed vertical alignment of multiwall carbon nanotubes (MWCNTs) have diameters ranging from 16 to 23 nm. The TGA analysis showed that the purity of CNT produced was 98%.

© 2014 Elsevier B.V. All rights reserved.

1. Introduction

Since the accidental discovery of carbon nanotubes (CNTs) during the synthesis of fullerene by Iijima [1], tremendous research has been done on CNTs. The prospect of developing novel carbon-based nanomaterial has excited worldwide interest among researchers. CNTs have been of great interest; both from fundamental point of view and for potential applications because of their amazing mechanical [2,3], thermal [4], electric and magnetic properties [5,6]. They have large number of potential applications, which include flat panel field emission displays [7], nanoelectronic devices [8], chemical sensors [9], hydrogen storage [10,11] and scanning probe tip [12]. Many ways are currently available for the production of CNTs, which include arc-discharge [13], pulsed laser vaporization [14], chemical vapor deposition [15]. However, commercial applications of CNTs have been inhibited by the lack of large-scale production of purified CNTs.

Recently, a microwave-assisted synthesis is enabling technology that has been extensively used in organic synthesis [16–18]. Microwave-assisted modification of CNTs is non-invasive, simple, fast, environmentally friendly, and clean method as compared to traditional methods. Usually, the use of the microwave facilitates and accelerates reactions, often improving relative yields. In case of microwave-assisted functionalization of CNTs, microwave irradiation of CNTs and CNFs reduces the reaction time and gives rise to products with higher degrees of functionalization than those obtained by the conventional thermal methods [19]. Interestingly, a competitive effect of microwave irradiation promotes functionalization and also removes some initially-present functional groups [20]. On the other hand, Vazquez and co-workers show that a solvent-free technique combined with microwave irradiation produces functionalized nanotubes in just 1 h of reaction, paving the way to large scale production of functionalization [18]. It is of current general interest the development of new techniques for the efficient and selective synthesis of CNTs and carbon nanofibers (CNFs) other carbon nanostructures at the cheapest possible cost. One such possibility is the use of microwave radiation, which over the past few years has played an important role as a thermal tool in organic synthesis due to considerable advantages over conventional methods [21]. The use of microwave radiation in the synthesis and functionalization of CNTs or other nanostructures is advantageous because it provides a fast and uniform heating rate that can be selectively directed toward a targeted area. The first report of the production of carbon nanostructures with microwaves was made by Ikeda et al. (1995), [22] who synthesized fullerenes from microwave-induced naphthalene–nitrogen plasma at atmospheric pressure inside a cylindrical coaxial cavity. Kharissova et al. [23] has reported the synthesis

* Corresponding author. Tel.: +673 603 79675295; fax: +673 603 79675319.
E-mail addresses: mubarak.yaseen@gmail.com (N.M. Mubarak), jay_sahu@yahoo.co.in (J.N. Sahu).

http://dx.doi.org/10.1016/j.diamond.2014.07.005
0925-9635/© 2014 Elsevier B.V. All rights reserved.

of vertically aligned CNTs using a domestic microwave oven. Unlike conventional heating, microwave heating has a higher heating rate which results from the intrinsic transition of electromagnetic energy to thermal energy by a molecular interaction with the electromagnetic field, rather than heat transfer by conduction or convection. Although materials are heated differently by microwaves, the maximum temperature is determined primarily by the dielectric properties of the receptor. The microwave heating of carbon-based materials gives rise to hot spots that appear as small sparks or electric arcs, with local temperature higher than 1100 °C. These hot spots have been well established as the thermal sensitizer upon microwave irradiation in the fields of organic synthesis, environmental remediation, preparation of catalysts, and carbon nanostructures.

In this study, CNTs were produced by tubular microwave chemical vapor deposition (TM-CVD) using ferrocene as a catalyst and acetylene (C_2H_2) and hydrogen (H_2) as precursor gasses. The effect of process parameters such as microwave power, radiation time and gas ratio of C_2H_2/H_2 was investigated. The preparation conditions were optimized to produce the best CNT yield in terms of the amount and high purity.

2. Materials and methods

2.1. Gasses

There are three types of gasses used in the production of CNTs namely, H_2 (99.99% purity), C_2H_2 (99.9%) and Ar (99%). All are analytical grade supplied by MOX Sdn. Bhd.

2.2. Catalyst

Ferrocene catalyst for analytical grade was purchased from Merck and used as received.

2.3. Production of CNTs

Fig. 1 shows schematic of horizontal tubular microwave model Synotherm-T1500, China reactor for the production of CNTs. It comprised a quart tube of 55 mm OD, 50 mm ID and 615 mm length. Ferrocene catalyst was placed at the entrance of the chamber and quartz boat size was placed at the middle of reaction chamber. The system was initially flushed with Ar in order to ensure oxygen free environment. The gas flow rate of C_2H_2 and H_2 was free mixed before entering into tubular microwave chamber using gas mixture model KM-20-2, Germany, after the gas mixture was sent to microwave chamber. The reaction was carried on for the desired time period and on completion, the total amount of CNTs produced in the quartz boats was collected and weighed.

2.4. Characterizations of the best quality CNT produced

Chemical and physical characterizations of the best quality CNT were studied. The morphologies of the CNT produced were examined using Field Emission Scanning Electron Microscope (FESEM) model Zeiss Auriga, Japan. The structure of CNT was characterized using Transmission Electron Microscope (TEM) model Hitachi H-7100, Japan and high-resolution TEM (HRTEM) model JEOL JEM 2100F, Japan. The purity of CNT was determined by Thermogravimetric Analysis (TGA) model Pyris diamond TG/DTA, USA. The graphitization of the CNT samples was determined by using Raman spectroscopy model Renishaw inVia, USA at a wavelength of 532 nm with green laser excitation. The XRD patterns were obtained by using model Siemens D-500 X-ray, diffractometer, USA. Fourier Transform Infrared (FTIR) spectroscope (Brand: Bruker, Model: IFS66v/S) was used to analyze the CNTs for the determination of the surface functional groups.

3. Result and discussion

3.1. Effect of process parameters on synthesis of CNT

CNT production requires the knowledge of optimum microwave power, radiation time and gas ratio of C_2H_2/H_2 ratio. The effects of process parameters on the synthesis of CNT were studied to enhance the high yield with high purity. The effects of each process parameter were further discussed in the following section.

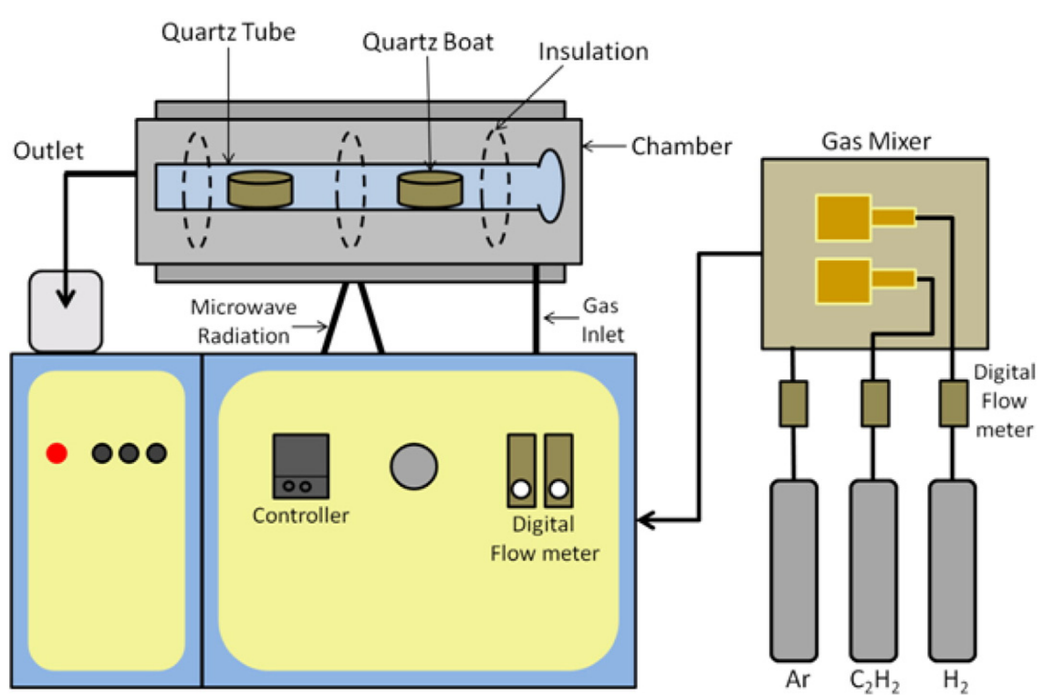

Fig. 1. Schematic of TM–CVD for CNT production.

Download English Version:

https://daneshyari.com/en/article/701983

Download Persian Version:

https://daneshyari.com/article/701983

Daneshyari.com