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Preparation and Characterization of Carbon Nanotubes Using Ablation Method for Composite Applications

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Abstract: The synthesis of the single-walled carbon nanotubes (SWCNTs) by laser ablation process (LA) is catalyst-dependent. In this work, two targets techniques were used instead of one target. The two targets used are nickel sheet (as catalyst) and graphite (as carbon source) next to each other. The laser used is Q-switched Nd:YAG of wavelength 1064nm hits the two targets simultaneously at the interface between them. Different parameters of LA process were studied: furnace temperature 750°C -1000°C, Ar flow rates and different laser fluences. The prepared CNTs were then used to prepare polymer/CNTs nano-composite. Scanning electron microscope and Raman spectroscopy were used to characterize the produced CNTs. The results showed that during laser ablation, the diameter of the prepared carbon nanotubes will decrease with increasing temperature from 750°C to 1000°C. The yield of the SWCNTs will increase with temperature from 850°C to 1000°C. The production of SWCNTs and MWCNTs are possible by controlling the furnace temperature. At Ar flow rates of 2 L/min, the SWCNTs yield is maximum. The polymer/CNTs nanocomposite prepared showed some improvement in the mechanical properties i.e. ultimate strength and Young's modulus.

Keywords: Carbon nano-tubes; Laser ablation; Electron microscopy; Raman spectroscopy; Nano-composites.

1. INTRODUCTION

CNTs were first discovered by Sumio Iijima [1]. Since their discovery; CNTs have remained exciting materials ever. CNTs are stronger than steel, harder than diamond, electrical conductivity higher than copper, thermal conductivity higher than diamond. [2]. Because of their extraordinary mechanical, electrical, and optical properties, CNTs have range of technologies applications in electronics, composites, hydrogen storage, sensors, and biological applications [3]. CNTs include both multiwalled carbon nanotubes (MWNTs) and single-walled carbon nanotubes (SWCNTs). A single-wall carbon nanotube is a rolled-up graphene sheet which is made up of benzene-type hexagonal rings of carbon atoms. The MWCNT is a stack of graphene sheets rolled up into concentric cylinders with a layer spacing of 0.3–0.4 nm. [4]. The CNTs have been synthesized by various methods e.g. electric arc discharge, laser evaporation and chemical vapor deposition. [5,6]. In Chemical vapor deposition (CVD), hydrocarbon gas is used as a source for carbon atoms and metal catalyst particles as “seeds” for nanotube growth. The CVD temperature is between (500-1000°C)[7,8]. Chemical vapor deposition (CVD) is most popular and widely used because of its low set-up cost, high production yield, and ease of scale-up [9]. The arc discharge method is the one by which CNTs were first produced and recognized. CNTs were produced by passing a direct current of 50 -100A between two carbon electrodes (cathode and anode) placed end to end, separated by approximately 1mm, in a closed chamber that is usually filled with inert gas at low pressure. The high temperature (>3000°C) provide by the arc carbon atoms in order to vaporize carbon atoms into a Plasma [5,10-11]. The arc discharge process has been used to produce large quantities of CNTs with modest purity since the majority of the impurity being amorphous carbon. [12].

Laser ablation method uses a high-power laser (YAG type) to vaporize pure graphite targets inside a furnace at 1200 ± °C under an Ar atmosphere to produce MWNTs [13]. Electric arc discharge and laser evaporation techniques are both able to produce single-walled and multiwall carbon nanotubes (SWCNTs, MWCNTs) [14]. The laser ablation technique has been able to produce SWCNTs with purity as high as 90%. which are purer than CNTs produced by arc discharge[15].

The pulsed laser-ablation process for the production of single-wall carbon nanotubes was developed by Guo et al. [13, 16] at Rice University. Other improvements were made by Thess et al., [17] and] and Rao et al., [18]. Laser ablation requires a metal catalyst for the production of CNTs. Various combinations of catalysts have been tried, including iron, platinum cobalt, nickel, and yttrium. It has been found that bimetallic catalysts are more productive than single metals [15].

Many reports have focused on the effect of processing parameters of laser vaporization (such as catalyst concentration, furnace temperature or gas flow and pressure) on the production yield and the characteristics of the carbon SWNTs. The higher intensity and high furnace temperature favors the formation of larger SWNTs with diameter at about 1.4 nm. At the optimal laser intensity it is believed to obtain: the highest SWNT yield, the largest SWNT bundles diameter, and the lowest level of amorphous carbon [19]. The yield and characteristics of SWNTs produced by the laser-vaporization of carbon depends on the concentration and the nature of the catalysts [16, 20, 21].

Differing concepts of laser ablation use a single pulsed laser or two pulsed lasers operating at different wavelengths; other concepts use a continuous laser [22, 23]. The effect of

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different catalyst concentration in laser ablation was investigated using Co/Ni and Co/Pt mixtures. The SWCNTs yields are 10-100 times that for the single metals alone. Thus, the use of bimetallic catalysts is more productive than single metals [16]. The atmospheric temperature controls the chemical reaction and the laser intensity affects the process by which the particles species ejected [24].

In general, CNTs produced by laser ablation method via the interaction of the laser beam with graphite/metal composite target which lead to increase the concentration of the metal catalyst at the target surface during the ablation process. Thus, the target surface becomes metal-rich and this lead to decrease the yield of the SWCNTs i.e. the abundance of SWCNTs within the produced soot.

The main objective of this work is to present a new method for synthesizing CNTs using two targets (Ni/graphite) instead of one target (Ni/graphite). The two targets methods will allow manipulation of the amount of graphite and catalyst.

The aim of this work is also to prepare CNTs /polymer nanocomposites using the prepared CNTs by laser ablation method and to study the mechanical properties of the prepared CNTs/polymer nanocomposite.

2. LASER ABLATION SYSTEM

The laser ablation (vaporization) apparatus (Fig. 1) used in the present work is a home built and consist of a quartz glass chamber with an inner diameter of 2.8 cm and a length of 60 cm. Another quartz tube with an inner diameter of about 2 cm and a length of 50 cm was located at the center of the chamber. A target was placed inside, at the center of the inner quartz glass tube. A quartz glass plate was situated in front of the target as a CNTs collector. The quartz tube was surrounded by a tubular furnace that heats a quartz tube to a temperature (700-1200) °C. The targets (graphite and Ni sheets mounted next to each other) were located inside a quartz tube and laser impacted on the boundary points between graphite and nickel sheets causing ablation of the target under an Argon inert gas. The laser ablation system was built at the Institute of Laser for postgraduate studies, university of Baghdad. One of the important parameter that used to optimize the synthesis of carbon nanotubes is the temperature. Different temperatures were selected 750, 800, 850, 900 and 1000°C. Argon gas flowed through the tubes at a rate of 0.5 L/min. A 532nm and 1064nm Q-switched Nd:YAG laser of pulse duration of 10ns with pulse repetition rate of 6Hz irradiated the target surface through a quartz glass window of the chamber. The laser beam power density range was (117.85-235.57) MW/cm² and the beam diameter was 4 mm. Number of pulses for each experiment was 1800 pulses. The as-produced carbonaceous deposits were characterized by Scanning Electron Electron Microscope (SEM) and Raman Spectroscopy. The SEM (Hitachi s-4160) images were used to study the morphology of the produced carbon nano-tubes in addition to the distribution of the carbon nanotubes. The SEM was provided with a program to calculate the nanotubes diameters. The Raman spectroscopy (Horiba Jobin Yvon LabRam HR800) images were used to insure that the produced soot.

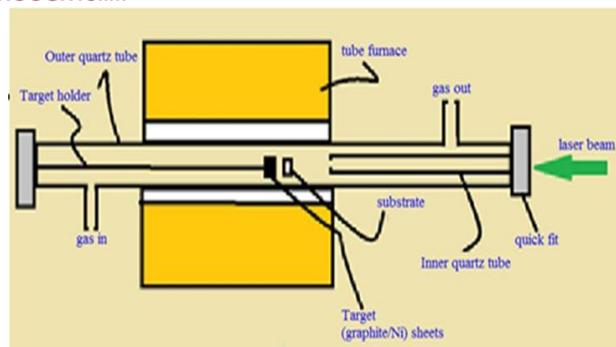


Fig. 1: Schematic diagram of the laser ablation system used in this work.

3. CNT/EPOXY NANO-COMPOSITE

The resin used in the present work was LEYCO –POX 103 (Density at 23°C is 1.05(g/cm³)). This resin has good advantages such as: Extremely low viscosity, Short and long pot lives, High mechanical strength. The prepared MWCNTs by laser ablation (this work) were used as reinforcement with diameter 15nm and lengths (5-7) μm. A 5g of ethanol as solvent was used for dispersion of prepared MWCNTs in epoxy matrix. About (0.5, 1, 1.5, 2 wt %) of MWCNTs was dissolved in ethanol with the epoxy. The epoxy-MWCNTs-ethanol was mixed using magnetic stirrer (FANEM SAO Pulo-Brasil) for 8 hours. It was noticed also that by increasing the MWCNTs wt% the viscosity of the epoxy-MWCNTs increases also.. After all ethanol was evaporated (about 8 hours, mixing time). The hardner was added to mixture of epoxy-MWCNTs with ratio (epoxy: hardener 7:3). The epoxy-MWCNTs-hardener was then stirred for 15 min. The prepared epoxy nanocomposite was then poured into a rectangular glass mold. The sample was then cured in furnace at 80°C for 2 hours. Samples of plain epoxy without the nanotubes were prepared for reference. Samples were then cut according to ASTM-D638 for Tensile test. The samples were subjected to uniaxial tensile test using tensile testing machine (Q-TEST MTS) with a computer for data acquisition. The test speed was set at 1 mm/min.

4. RESULTS AND DISCUSSION

4.1 Effect of Furnace Temperature on the Production of Carbon Nanotubes.

One of the most important parameters that affect the growth of carbon nanotubes is the temperature. The laser ablation experiments were done at temperature (750-1000) °C. The parameters for these experiments are Nd:YAG laser with 1064nm, 10ns, No of Pulses (1800), fluence (2.5)J/cm² with Argon gas flowing inside a quartz tube at flow rate (0.5L/min). Fig. 2 shows SEM image of as-grown carbon nanotubes after laser ablation of (graphite/Nickel) target at (a) 750 °C (b) 800 °C (c) 850°C and (d) 900°C. At temperature between 750-800 °C the structure of the deposit carbon nanotubes has a web-like structure. At temperature 850°C - 1000°C one can notice the transformation of the deposit carbon nanotubes structure from web-like structure to spaghetti-like structure. Fig. 3 shows the number of CNTs distribution carbon nanotubes at different temperature (750-900)°C using a program instilled with SEM.

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The Raman spectra of the above images at different temperatures 750 °C, 800 °C, 850 °C, 900 °C is shown in Fig 4. The Raman spectra for carbon nanotubes consist of three main regions: (1) the low frequency region (Radial Breathing Mode RBM) (120-240) cm^{-1} ; (2) the high frequency region (1530-1650) cm^{-1} due to the vibration of carbon atoms of the graphite (G-band) and; (3) the D-band centered around $\sim 1350\text{cm}^{-1}$ [16,25] is due to the vibrations of carbon atoms with dangling bonds in the disordered plane structure. The spectra are finger print of MWCNTs according to the G-band and decreasing the intensity of the D-band with increasing temperature from 750°C - 800°C. The G-band centered at 1566cm^{-1} and 1580cm^{-1} for 750°C and 800°C respectively. This indicates that with increasing temperature for 750°C to 800°C the diameter of the MWCNTs will decrease. Also the diminishes of the RBM indicate there are no single-wall carbon nanotubes at these temperatures [25]. The appearance of the RBM region between (850-1000) °C which is related to single wall carbon nanotubes, Fig 4. The peaks of the RBM are located approximately between 113cm^{-1} and 221cm^{-1} with increasing the intensity of the peaks located to the left of the RBM region.

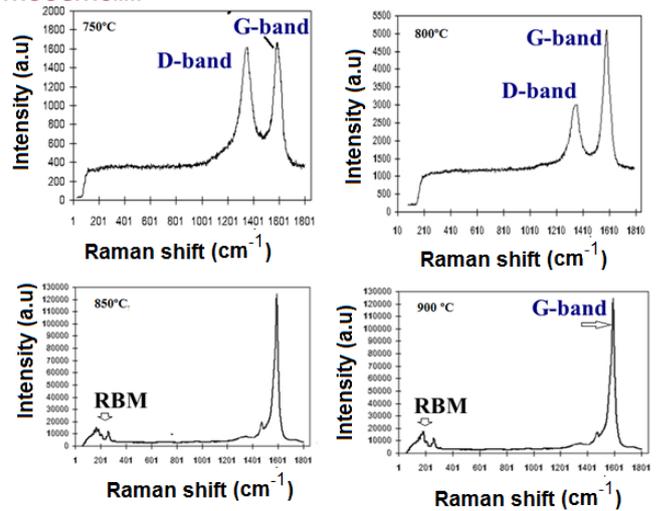


Fig. 4: Raman spectra of carbon nanotubes produced by LA 750°C -900 °C.

These peaks are coincide with the carbon nanotubes diameter $\sim (1-2)\text{nm}$ [26]. With increasing the temperature from (850-1000) °C the prominent peak at $\sim 1470\text{cm}^{-1}$ of C₆₀ [16] decreased. This reveals that the C₆₀ is transformed to SWCNTs with increasing temperature. The temperature dependence of the carbon nanotubes diameter can be explained by changes in the cooling rate of the ablated carbon species. As the carbon vapor plume expands and cools down, the nanotubes structure become kinetically fixed when the temperature is not high enough to allow rearrangement of carbon atoms. Expansion into the colder buffer gas increasing the cooling rate and will lead to nanotube nuclei sized being fixed earlier in time and therefore to smaller nanotubes [21]. The larger diameter is associated with larger chiral angle. As the nuclei size changes the strain energy of the curved grapheme sheet is balanced by strain energy of an open grapheme edge. A decrease in the nucleus diameter for fixed number of carbon atoms. Probably the nanotubes do not reach thermal equilibrium in rapidly expanding and cooling plume of carbon cluster [27].

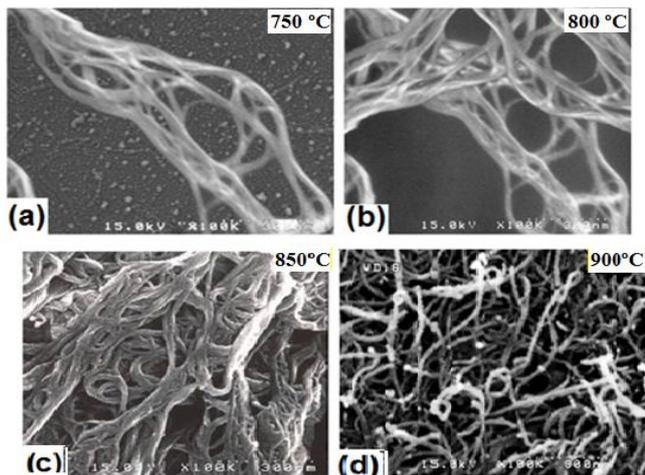


Fig. 2: SEM image of as-grown carbon nanotubes after laser ablation of (graphite/Nickel) target at (a) 750 °C (b) 800 °C (c) 850°C and (d) 900 °C.

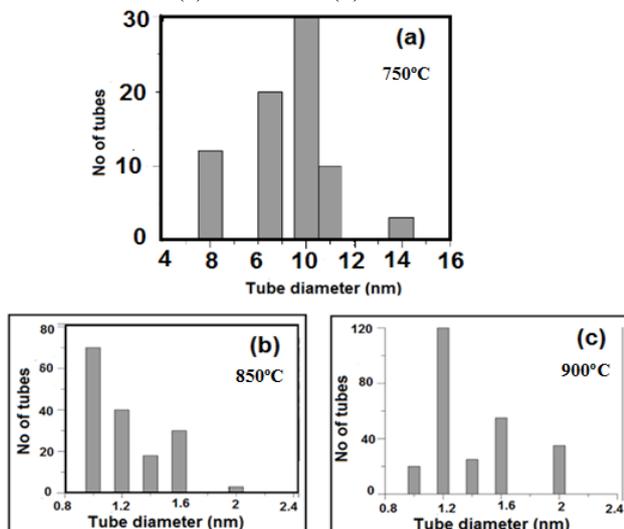


Fig. 3: Histogram of SEM image of CNTs distribution at (a) 750°C ; (b) 850°C ; and (c) 900°C .

4.2 Effect of Gas Flow Rate on the Production of Carbon Nanotubes.

To understand the mechanism of formation carbon nanotubes by laser ablation of two targets under an ambient Argon environment, the effect of the flow rate of the argon gas was studied. These experiments were done under different flow rates of Argon and the other parameter were kept constant. The range of the flow rate is around (0.1-5) l/min. The other parameters are ($\lambda=1064\text{nm}$, pulse duration=10ns, fluence = $1.5\text{J}/\text{cm}^2$, No. of pulses = 1800 pulse and temperature =850°C pressure 0.15MPa).Fig 5 represent the SEM images of grown carbon nanotubes under different flow rates of argon at 850°C. Fig. 5 shows the structure of the carbon deposit is weblike structure with the changing of the abundance of the carbon nanotubes. Fig. 6 shows increase the abundance of carbon nanotubes with increase of flow rate up to 2L/min and noticeable decrease of the abundance of carbon nanotubes at flow rate above 2 L/min. The sharp decrease of carbon nanotubes abundance in

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soot at gas flow rate above 2 L/min leads to the conclusion that the nanotubes growth time is close to the drift time of the reaction product through the hot zone of the furnace [28]. The drift time is on the order of few seconds [19]. Also Raman spectra of carbon nanotubes at different gas flow rate show that there is no change in the position of the peak of the RBM region but the changes occur on intensity of these peaks.

4.3 Effect of Laser Fluence on The Production of Carbon nanotubes

The effect of laser fluence on the on the yield and the structure of carbon nanotubes have been investigated at laser fluence of 1,1.5 , 2, 2.5, 3 J/cm² using two wave lengths ($\lambda=1064$ nm and $\lambda=532$ nm, Temperature = 850°C, pulse duration = 10ns, No. of pulses = 1800 pulses under flowing of Argon gas at 0.5 l/min with different fluence values) using laser fluence of 1,1.5 , 2, 2.5, 3 J/cm² respectively. It was found that the structure of the carbon nanotubes is a web-like structure. Fig 7 shows the RBM region of Raman spectra for two laser configurations. With increasing the fluence from 1 J/cm² to 2.5 J/cm² Raman spectra will be shifted to the right side of the RBM region. The second things that there is no single wall carbon nanotubes formed at fluence greater than 2.5J/cm².The diminishes of RBM region with fluence above 2.5 J/cm² means that there is a fluence threshold for formation of SWCNTs. Such a result is also reported in case of $\lambda=1064$ nm [19] with longer pulse duration (175-575) ns.

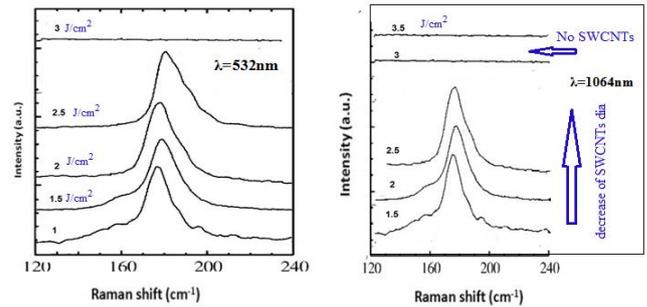


Fig. 7: RBM of Raman Spectra of laser configuration at $\lambda=532$ nm , 1064nm at different fluence.

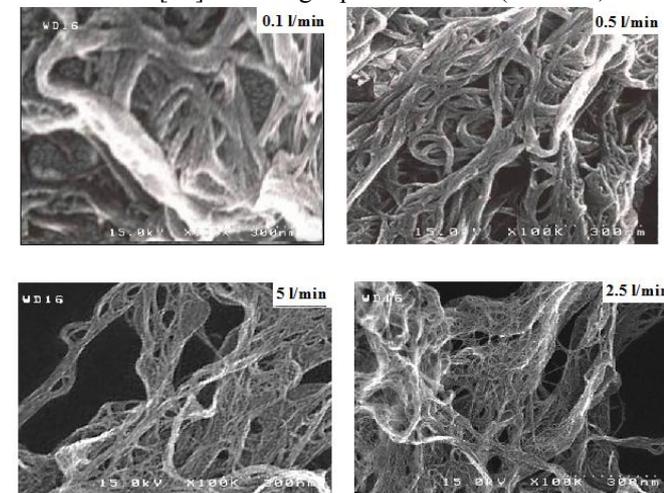


Fig. 5: SEM image of as grown carbon nano-tubes produced at 850°C at different Argon Flow rate

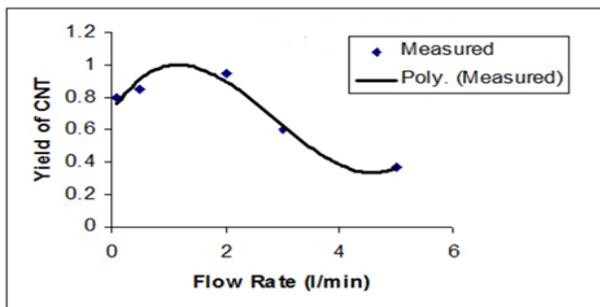


Fig 6: Yield of carbon nano-tubes as a function of gas flow rate at a temperature 850°C.

4.4 Tensile Test

The stress-strain curves for epoxy with various weight percentages of MWCNTs were shown in Fig 8. The results showed significant improvement in modulus and ultimate tensile strength were obtained for prepared epoxy nano composite in addition to pure epoxy material. The stress-strain curve showed an increase in the ultimate tensile strength as well as modulus of elasticity with increase in weight percentage addition of MWCNTs into the epoxy polymer matrix for epoxy. However, it can be seen that as the stiffness of the epoxy increases with increase in reinforcement, the strain to failure reduces. The percentage increase in the strength and Young Modulus has been shown in Fig. 9. The improvement in UTS is 37 %. For modulus, the improvement obtained for epoxy was as high as 125.1 %. Most part of the improvement in modulus and UTS for epoxy polymer is obtained with the addition of 0.5 wt% of MWCNTs. This can be attributed to the ease of dispersion for low concentrations of MWCNTs.

5. CONCLUSIONS

The production of the carbon nanotubes by using laser ablation of two targets can be considered as an effective method for production of SWCNTs. The distribution of the tubes diameter is wider than other laser ablation technique. The quality of the produced SWCNTs is good which is appeared in high ratio of the intensity of G-band to D-band. Changing furnace temperature can give the two types of the carbon nanotubes, namely SWCNTs at temperature between (850-1000) °C and MWCNTs at temperature between (750-800)°C.

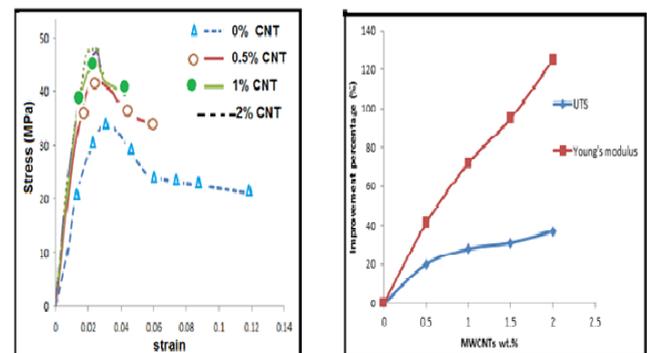


Fig. 8: (a) Stress – strain curve of epoxy with and without carbon nanotubes (b) Percentage improvement in the properties of epoxy nano-composite.

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