SYNTHESIS METODS OF CARBON NANOTUBES (review article)

A.M. ALIYEVA
Institute of Physics Ministry of Science and Education of Azerbaijan Republic, AZ 1073, Baku, H. Javid Ave., 131

Carbon nanotubes are discussed in this review synthesis methods. Carbon nanotubes synthesis by variety methods and each of the mechanisms have precise features that can be specify properties of carbon nanotubes. Laser ablation and chemical vapor deposition are most common methods for synthesis carbon nanotubes.

Keywords: laser ablation, carbon nanotubes, physical vapor deposition, pulsed laser vaporization (PLV), Nd:YAG.
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INTRODUCTION

Carbon nanotubes have attracted considerable interest since their discovery by Iijima in 1991 [1]. Carbon nanotubes have aroused great interest recently because of their unique physical properties, which span a wide range from structural to electronic. For example, nanotubes have a low weight and high elastic modulus, and thus they are predicted to be the strongest fibers and widely touted as attractive candidates for use as fillers in composite materials due to their extremely high Young's modulus, stiffness, and flexibility [2–6]. These latter applications will require vast quantities of nanotubes at competitive prices to be economically feasible. Moreover, reinforcing applications may not require ultrahigh purity nanotubes. On the other hand, functionalization of nanotubes to facilitate interfacial bonding within composites will naturally introduce defects into the tube walls, lessening morphologies are needed for specified applications of CNTs. For instance, usually it is needed to prepare knotted CNTs to improve in rich interfacial adhesion, which can lead to nanotube aggregation within the matrix composites. So, there are many methods of producing these nanomaterials, including electric arc discharge [1,7], laser evaporation [8], chemical vapor deposition (CVD), catalytic CVD [9–19], hydrothermal treatment [20] and mechanothermal process in which graphite powders were first mechanically ground at room temperature and then annealed at 1400 8C [21], but little is known about the possibilities of mechanothermal processing aimed to the synthesis of carbon nanostructures in different conditions. Although significant research progress has been made to synthesize carbon nanomaterials, but developing an easy approach to large-scale production of carbon nanotubes has still been limited to date. On the other hand, most of the synthesizing methods are complicated and uncontrollable. More recently, we have suggested that using washable supported catalysts is accompanied by valuable advantages and with an extraordinary structure [22,23]. Herein, we use an efficient method for the controlled synthesis of spring-like multiwall carbon nanotubes by mechanical activation assisted annealed process [14].

In their paper authors investigated the synthesis of carbon nanotubes.

Laser ablation method

Physical vapor deposition (PVD): PVD is a technique by which a material canbe vaporized into gaseous form and then deposited on the surface of a substrate.

Target source: The most common carbon source target used is solid graphitewhich is irradiated by laser source and vaporized into vapor carbon atoms.

Laser source: Laser source used for vaporization of target material into target vapor atoms can be continuous laser source such as CO_2 laser or pulsed laser source such as Nd:YAG laser (Neodymium doped Yttrium Aluminum Garnet, Nd:$Y_3Al_5O_{12}$) .

Substrate used: The substrate used in this method is the water cooled coppercollector on which the vaporized carbon atoms deposit and grow as CNTs.

Inert gas atmosphere: Argon gas is commonly used as inert gas which flows at aconstant flow rate towards the water cooled copper collector.

Procedure for synthesis of CNTs by Laser Ablation method

Laser Ablation method is a Physical Vapor Deposition method in which graphitetarget is vaporized by laser source Figure 1. In this method the graphite target is placed at the center of quartz chamber filled with argon gas and maintained at 1200°C.The graphite target is vaporized by either continuous laser source or pulsed laser source. The vaporized target atoms (carbon) are sweeped toward cooled cop- per collector by the flow of argon gas. The carbon atoms are deposited and grown as CNTs on cooled copper collector. In case of continuous laser beam, the carbon atoms are continuously vaporized whereas in case of pulsed laser beam the amountof CNTs produced can be monitored as each shot of pulsed laser beam is directly proportional to the amount of carbon atoms vaporized [11]. By this method multi- walled carbon nanotubes are synthesized and to synthesize single-walled carbon nanotubes catalyst nanoparticles of Fe, Co, Ni are used.

The obtained CNTs arefurther purified to get the pure form of CNTs.

131, H.Javid ave, AZ-1073, Baku
Institute of Physics
E-mail: jophphysics@gmail.com

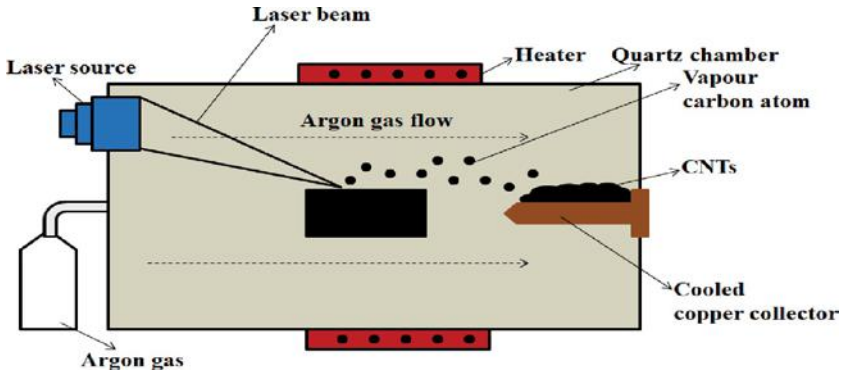

Fig. 1. Laser ablation method-schematic representation.

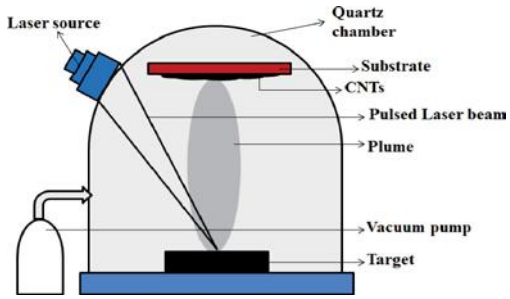

Fig. 2. Pulsed laser ablation method-Schematic representation.

Procedure for pulsed laser deposition method

Pulsed Laser deposition is a thin film deposition technique in which the targetmaterial is vaporized by pulsed laser beam and vaporized target atoms are made to deposit on substrates Figure 2. The furnace contains a target at bottom and substrate mounted on the top. A pulsed laser beam from Nd:YAG laser source is made to strike the target to produce vaporized target atoms called the plume (plume is vaporized atoms at high temperature) [12]. The plume moves towards the substrate and it is deposited and grown as CNTs. Each shot of laser is directlyrelated to the amount of material ablated, thus deposition rate can be controlled and calibrated.

The synthesis methods were investigated by Seyed Oveis Mirabootalebi, Gholam-Hosein Akbari Fakhrabadi. Authors (13).

First synthesis of CNT was done accidentally by arc discharge (1) and now synthesis methods of carbon nanotubes are different and various. Generally, produce type can specified properties of CNT (15); precursor (which can be solid, liquid, or gas), heat source, time, temperature, atmosphere of reactions and commonly; mechanism, determinate traits of CNT. Most common methods for synthesis of carbon nanotube are arc discharge, laser ablation and chemical vapor deposition (15-19). Some of other methods for synthesis of carbon nanotubes include: dipping graphite in cold water (21), mechano-thermal (20), synthesis with decomposition Sic, torsion of graphene layers, with solar energy, synthesis with heat treatment of polymer, pyrolysis, with liquid phase and electrolysis.

Electrolysis method

This method based on liquid phase which was invention in 1995. with electrowinning of alkali or alkaline-earth metals from their chloride salts, CNT deposited on substrate (22).

By applying DC voltage between two electrodes in chamber of molten alkali-alkaline earth metals, could be produced multi walled carbon nanotubes]. Relation (1) show formation lithium carbide (23).

$$2Li^{+}+2e^{+}+2C\ (graphite) = Li_2C_2 \qquad (1)$$

By forming lithium carbide (Li2C2), synthesize of CNT can be started in liquid phase. Generally; diameter of cnt in this method was 2-10 nm and length of them is 0/5 micrometer or more was reported and amorphous carbon, carbon nanofibers, nanographites and encapsulate CNT are byproducts of this method (24). Obtained CNT usually are multi walled, but also in some researches, produced single walled CNT (26) Different salts, applied for producing cnt such as NaCl, LiCl, KCl, and LiBr (25).Current density, electrolysis regimes, time, molten salt and temperature are the controller parameters of reaction. By optimizing condition of process, the reaction yields up to 20- 40% increase for producing multi walled carbon nanotubes].

Electrolysis can be done in low temperature, don't need to advanced equipment, possibility to controlling process of synthesize, have high quality, having low energy consumption and also don’t suitable for mass production.

Laser Ablation method

In Laser ablation/vaporization, by strike pulsed laser or continuous wave laser on graphite target; nucleation and growth of carbon nanotubes to be started (31,23). First formation a hot evaporation and subsequently quickly cooled.

During cooling of the samples, small molecules and carbon atoms quickly condense and form larger clusters and synthesized carbon nanotubes by van der waals forces stay together. For producing multi wall carbon nanotubes use pure graphite rods and for single wall use composite block of graphite. For producing single walled, Graphite composited with metal catalyst, such as Fe, Ni and Co and He-H_2 and Ar use as ambient gas.

In pulsed laser, needs more intensity laser's light than continuous laser. Nd:YAG and CO_2 are most common lasers used for laser ablation (31).

Diameters of CNT which produced by this method 4-30 nm and length of them is about 1 micrometer.Byproducts and impurities are amorphous carbon, catalyst particles, fullerene etc. Catalyzers also help to growth of carbon nanotubes. Most of catalyst were used in laser ablation are: Co, Cu, Nb, Ni, Pt, Co/Ni, Co/Pt, Co/Cu, Ni/Pt. quality of production related to target composition, powerof laser beam and laser properties, catalyst type, type of ambient gas, temperature of reactions and distance between substrate and target (31). Produced carbon nanotubes by this method have high purity, high yield and most produced single wall CNT; but not suitable for mass production and also need expensive and especial equipments.

Flame Synthesis

In this method, CNT synthesizing by direct combustion of carbon source in presence of an oxidizing gas (23). Generally; flame synthesis contain of three steps. First producing carbon source by hydrolysis of hydrocarbon. Second; diffusion of carbon's atoms to metallic catalyst and third; nucleation carbon nanotubes on surface of catalyst and it's gradual growth.

Oxidation gas can be oxygen or nitrogen and carbon feedstock are acetylene, methane, ethanol and ethylene. Type of flame created, have essential role on quality of produced carbon nanotubes and determinate amount of amorphous carbon in final producing. For optimizing condition, must be control temperature, composition of fuel gas and catalyst of reactions. This way is economic, suitable for mass production and most synthesizing single walled CNT, but rate of growth is relatively low.

Laser vaporization method

The CNTs were first synthesized using a laser in 1995, and the pulsed laser vaporization (PLV) technique is used to extract a large variety of carbon allotropes from graphite, one of which is CNTs. A laser beam (Yttrium Aluminum Garnet or CO_2 laser) was used in this process, and it will be put within a reactor, where it will be focused on the graphite rods. Until beginning the process, argon buffer gas and a catalyst mixture of Co and Ni in a 50:50 ratio was passed through the rod at 1200°C. The metal was added to the process to help catalyze the creation of SWCNTs, but it also produced a slew of other byproducts. As a result of the high temperature of the argon buffer steam, the rod will vaporize. As the vaporization was cooled, it was deposited in the copper collector, and nanotubes were formed (33,34). The schematic diagrams for the laser ablation procedure are shown in Fig. 3.

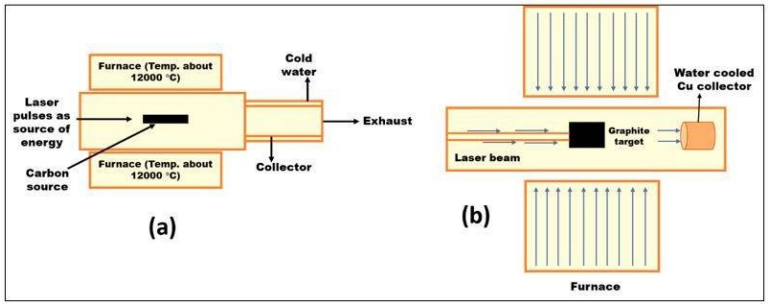

Fig. 3. Schematic diagram for laser ablation technique.

One of the benefits of this method is that high-quality SWCNTs with limited defects can be achieved in a reasonably high yield. Since the metallic atoms appear to vaporize when the tube's end is closed, this was possible. SWCNTs generated have excellent structural integrity as well. Furthermore, diameter modulation of the samples can be achieved easily by adjusting the method's parameters, i.e. catalytic metal, flow rate, and temperature. Apart from that, SWCNTs can be synthesized without requiring the production of MWCNTs.

However, there are several drawbacks to this approach. The CNTs generated may have some branching and may not be perfectly straight. Despite using higher purity graphite rods and laser forces, the amount of CNTs generated is lower than when the arc discharge approach is used (35). Typically, a significant amount of SWCNTs can be generated with good crystallization using both arc discharge and laser ablation methods. However, owing to the unique basic equipment requirements and high energy demand, these two strategies are less advantageous than CVD. In terms of yield and purity, CVD are superior to arc

and laser methods. This is because the size of the carbon source, i.e., anode for arc discharge and target for laser ablation, determines the amount of the sample formed in both arc and laser processes. There is also a need for intensive purification of CNTs developed using these two techniques, which led to the production of gas phase methods like CVD.

Chemical vapor deposition (CVD): CVD is a technique in which the vaporized reactants react chemically and forms a nanomaterial product that is deposited on the substrate **Figure 4**.

Sources for carbon: The precursor for carbon nanotubes are hydrocarbon gasessuch as acetylene, ethylene, methane, etc.

Substrate used: Substrates are materials on which the CNTS are grown. The commonly used substrates in CVD method are zeolite, silica, silicon plate coated with iron particles, etc.

Catalyst used: To produce single-walled carbon nanotubes metal catalyst nanoparticles such as iron, cobalt, nickel, molybdenum, iron-molybdenum alloys, etc. are used.

Sources for CVD used: Based on the heating source, the CVD can be:

- Thermal activated CVD which is heated by IR radiation, RF heater, etc.
- Photo assisted CVD which is heated by Arc lamps, CO_2 laser, Argon ion laser,Nd:YAG laser, etc.
- Plasma assisted CVD which is heated by microwave radiation, etc.

Conditions maintained: The following conditions are maintained inside thefurnace.

- Temperature: 500–900°C.
- Inert gas atmosphere: Argon gas.

Procedure for synthesis of CNTs by thermal CVD method

CNTs are synthesized by thermal CVD method by using hydrocarbon gas as car- bon source. In this method, a quartz tube is placed inside a furnace maintained at hightemperature (500–900°C) heated by RF heater. A crucible containing the substrate coated with catalyst nanoparticles is placed inside quartz tube filled with inert gas such as argon gas.

The hydrocarbon gas (carbon source) is pumped into the quartz tube which undergoes pyrolysis reaction and forms vapor carbon atoms. These carbonatoms bind to the substrate and join to eachother by Vanderwaal force of attraction and grow as multi-walled carbon nanotubes (MWCNTs) on the substrate [23]. To synthesize single-walled carbon nanotubes catalyst nanoparticles of Fe, Co, Ni are used. The obtained CNTs are further purified to get the pure form of CNTs.

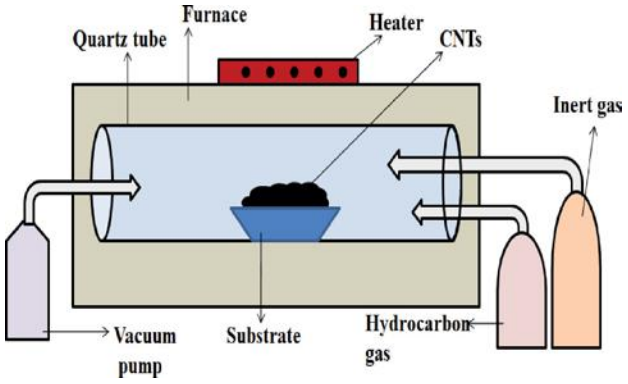

Fig. 4. CVD method.

Electric arc discharge method

Carbon nanotubes are synthesized by electric arc discharge method which is alsocalled Plasma Arcing method.

Electrodes: Pure graphite rods (both positive and negative electrode). The positive electrode is adjustable from outside to maintain the gap between the two electrodes.

Diameter of electrodes: 5–20 μm. **Gap between electrodes:** 1 mm. **Current:** 50–120 amperes.

Voltage: 20–25 V.

Inert gas pressure: 100–500 torr (No CNT formed below 100 torr). Inert gas is used for cooling and condensation of atoms to form the CNTs. Inert gas deter- mines the structure of carbons to be present in CNTS. Commonly used inert gas ishelium gas.

Temperature: 3000–3500°C.

Reactor: It contains a quartz chamber which is connected to vacuum pump and a diffusion pump to inert gas supply. Initially the chamber is made vacuum bythe vacuum pump and then the chamber is filled with helium gas by the diffusion pump [9].

Procedure for synthesis of CNTs by Electric arc discharge method

In this method, a potential of 20–25 V is applied across the pure graphite elec- trodes separated by 1 mm distance 4and maintained at 500 torr pressure of flowing helium gas filled inside the quartz chamber **Figure5**. When the electrodes are madeto strike each other under these conditions it produces an electric arc. The energy produced in the arc is transferred to the anode which ionizes the carbon atoms of pure graphite anode and produces C^+ ions and forms plasma (Plasma is atoms or molecules in vapor state at high temperature). These positively charged carbon ions

moves towards cathode, gets reduced and deposited and grow as CNTs on the cathode. As the CNTs grow, the length of the anode decreases, but the electrodes are adjusted and always maintain a gap of 1 mm between the two electrodes. If proper cooling of electrodes are achieved uniform deposition of CNTs are formed on the cathode which is achieved by inert gas maintained at proper pressure [10]. By this method multi-walled carbon nanotubes are synthesized and to synthesize single- walled carbon nanotubes catalyst nanoparticles of Fe, Co, and Ni are incorporated in the central portion of the positive electrode. The obtained CNTs are further purified to get the pure form of CNTs.

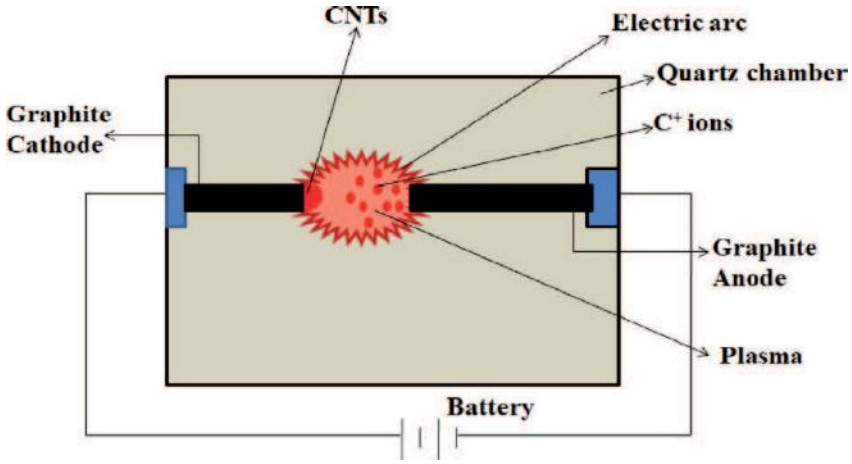

Fig. 5. Electric arc method.

Chemical vapor deposition (CVD) method

CVD is showing high potential to be used further as a CNTs processing tool for future industrial applications due to advantages such as relatively low growth temperature, high yields, and high purities with many explicit properties that can be obtained during its manufacture While this process was first used in the 1960s and 1970s to produce carbon fibers and carbon nanofibers, it was not until 1996 that it was used on a wide scale for the production and synthesis of CNTs. Strong, liquid, and gaseous precursors were used to aid in the development of CNTs at relatively low temperatures, i.e., 500 to 1000°C, and at atmospheric pressure. Metal or metal oxide catalyst particles are used as "seeds" to aid in the development of certain precursors. In addition, the gaseous or volatile carbon compound can decompose with the aid of metallic nanoparticles as a catalyst in this step. The catalyst also serves as a nucleation site for the development of CNTs. The reaction chamber is filled with a combination of nitrogen, ethylene, and acetylene during the procedure. This approach allows CNTs to expand in a variety of macroscopic morphologies, including powders and films (i.e., thick or thin), as well as various microscopic architectures (i.e., aligned, coiled, intertwined, or straight). The simplicity of the process is due to the use of such precursors, as well as different substrates and catalysts. As a result, producers will get the desired type of CNT. The four key parameters that specify the form of CNTs formed (SWCNT or MWCNT) in this process are the reactor's atmosphere, the source of hydrocarbon, the catalyst, and the growth temperature. MWCNTs are typically formed at lower temperatures (i.e. 600 to 900°C), while SWCNT development occurs at temperatures greater than 900°C. Not just that, but the commodity obtained is purer and has a higher yield. Regardless, defects can be detected in the composition of the samples collected, and these defects are normally in significant numbers. The general and basic schematic diagrams for the CVD technique are shown in Fig.6.

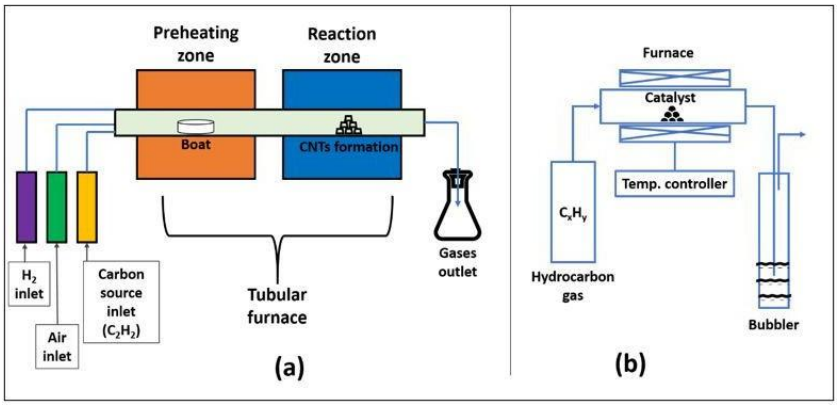

Fig. 6. Schematic diagrams of CVD process.

Alcohol CVD (ACCVD)

Since the temperature of the process is relatively low as low as 550°C, the ACCVD system can produce SWCNTs in large quantities at a low cost. Evaporated methanol and ethanol are applied to zeolite-supported iron and cobalt catalytic metal particles in this process. Alcohol reacting with catalytic metal particles created hydroxyl radicals, which removed carbon atoms with hanging bonds, preventing the formation of high-purity SWCNTs.

Plasma-enhanced CVD (PECVD)

PECVD is a broad concept that encompasses a variety of synthesis methods which may be either direct or remote. Direct PECVD was used to make MWCNT field emitter towers and some SWCNTs, while remote PECVD can be used to develop all types of CNTs. Instead of using thermal energy, this method uses energy sources that can supply energy for both hydrocarbon decomposition and CNT processing at a low temperature. The plasma energy sources used on the CNT formation are hot filament PECVD, direct current PECVD, radio frequency PECVD, and microwave PECVD. Hydrocarbon gas is used over intermediate metals in an ionized state in this process. Furthermore, the reactive species in the plasma system influenced the development of microscopic diameter tubes, which had implications for both diameter regulation and selective etching of metallic SWCNTs. The PECVD process consists of three major stages: (i) primary reaction takes place between energetic particles and carbon precursor in nonequilibrium plasma which leads to the precursor to be decomposed into carbon atoms and other active radicals; (ii) diffusion of carbon atoms and radicals onto the catalyst surface, as well as secondary reactions between the reactants, and (iii) catalyst interacts with the reactants on its surface, allowing carbon atoms to infiltrate and precipitate, as well as the release of result gases (Ding et al., 2016). This kind of CVD process has the benefit of producing a high yield of matched CNTs at a lower substrate temperature. Furthermore, by adding a voltage to the substrate, the volume of supplied ionized carbon species onto the catalyst surface can be tuned. The chirality distribution and growth rate of SWCNTs can be regulated, for example, by adjusting catalyst size and H_2 flow rate. This kind of CVD process has the benefit of producing a high yield of matched CNTs at a lower substrate temperature. Furthermore, by adding a voltage to the substrate, the volume of supplied ionized carbon species onto the catalyst surface can be tuned. The chirality distribution and growth rate of SWCNTs can be regulated, for example, by adjusting catalyst size and H_2 flow rate.

Thermal catalytic CVD

Decomposition of hydrocarbons or other carbon feedstock will take place at elevated temperatures ranging from 500 to 1200°C in this process. Prior to use, the feed would be dissolved in a steady flow of noble gases in the furnace, which would pass through transition metal catalysts such as iron, nickel, and cobalt, among others. The decomposed carbon would dissolve such metal particles until it reached a point of super saturation. The hydrocarbon is then decomposed and deposited on a substrate, i.e., alumina, glass, or silicon substrate. The carbon precipitate would then form in the shape of fullerene dorms. Depending on the temperature range of the process, different forms of CNT may be produced by using various types of carbon feedstock. Chemical composition and textural properties of the catalyst material(s) used are two parameters that control the length, diameter, orientation, and consistency of produced CNTs. The amounts of defects and the existence of amorphous carbon was also used to test the above properties of the finished product.

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