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SYNTHESIS OF VARIOUS FORMS OF CARBON NANOTUBES BY ARC DISCHARGE METHODS – COMPREHENSIVE REVIEW

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Abstract - *Carbon Nanotubes since its discovery have always excited the scientists around the world by virtue of its properties. There is no denying the fact that the future of the materials industry lies in the development of newer techniques for nanomaterials. The goal of this review paper is to provide an updated and in-depth review of some of the most promising and important developments in the processing of carbon nanotubes. Nanotubes can be formed in various structures using several different processing methods, for example, arc discharge, the laser ablation and the chemical vapor deposition (CVD) etc. In the recent period of the nanotubes production, the first two methods were utilized mainly for the production of single-wall carbon nanotubes (SWCNT) while the third one produced mainly Multiwall carbon nanotubes (MWCNT). Each of the synthesis methods used to produce specific kinds of nanotubes is discussed and detailed comparison is given.*

Key Words: Nano composites, Multiwall carbon nanotubes, arc discharge, the laser ablation, chemical vapour deposition (CVD)

1. INTRODUCTION

Carbon is known to be the most multipurpose element that exists on the earth. It has many different properties which depend upon how the carbon atoms are arranged. Carbon in the form of graphite was discovered in 1779, and 10 years later on in the form of a diamond. It was then proved that both of these forms belong to a family of chemical elements. In 1985, Kroto, Smalley, and Curl discovered fullerenes (Kroto et al., 1985), recipients of 1996 Nobel Prize in Chemistry for the discovery of fullerenes. A few years later, CNT was discovered by Iijima in 1991 [16]. He analyzed the samples synthesized by the arc discharge in the atmosphere. With TEM microscopy he observed some fascinating hollow tubule-like structures with nano sized diameter. Since that time these structures called carbon nanotubes has made long way, but they are still in the focus of research groups because of their immense future potential. Carbon nanotubes (CNTs) are characterized as a graphene sheet rolled-up to form a tube, for example a single-walled tube (SWNT). When two or more concentric tubes are placed one into another, multi-walled carbon nanotube (MWNT) is formed. Initially, the arc discharge method was used to produce carbon nanotubes. This method was popular enough and was utilized for the synthesis of carbon filaments and fibers. Later on, other techniques such as laser ablation and chemical vapor deposition (CVD) were examined in the synthesis of carbon nanotubes. In fact, these are the three main production methods till date. Some more efforts were also made to look for other possibilities to produce nanotubes but they had little success. The cause may have been the expensive reaction apparatus, the state or the price of the catalyst material, the strange reaction conditions, e.g., high pressure, temperatures of liquid nitrogen. So, as a result, the old technologies were improved, optimized and adapted to new conditions rather than to discover new technologies. Today, the arc discharge and chemical vapor deposition methods are widely in use for the formation of carbon nanotubes. Many studies and researches were made to improve either the quality or the quantity of the produced material by optimizing the synthesis process. As a result, some other types of CVD method were discovered such as plasma-enhanced, microwave-enhanced, radio frequency enhanced CVD.

The aim of this article is to provide an up-to-date and in-depth review of some of the most exciting and important developments in the synthesis of CNTs from the industrial perspective. The article also provides an outlook on future research directions on these materials as well as the techniques employed to produce them. It should be emphasized that the article is not a history of this subject, which is beyond the scope of this article. To obtain a more physics-oriented point of view the reader is referred to the several other review articles on the topic. For a fully comprehensive history of the subject, there are several books on carbon nanotubes.

2. STRUCTURE OF CARBON NANOTUBES

A single-walled nanotube (SWNT) is formed by rolling a sheet of graphene into a cylinder along an (*m*,*n*) lattice vector in the graphene plane (Figure 1). The (*m*,*n*) indices determine the diameter and chirality, which are key parameters of a nanotube. Depending on the chirality (the chiral angle between hexagons and the tube axis), SWNTs can be either metals or semiconductors, with band gaps that are relatively large (_0.5 eV for typical diameter of 1.5 nm) or small (10 meV), even if they have nearly identical diameters.

For same-chirality semiconducting nanotubes, the band gap is inversely proportional to the diameter.[1-8]

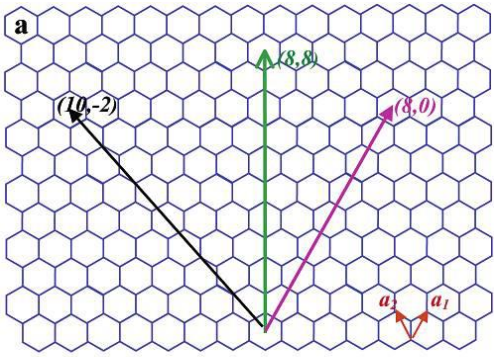

Figure 1. SWNT lattice vector in the graphene plane

Single-walled carbon nanotubes can be formed by folding the sheet along lattice vectors [1]. The two basis vectors **a**1 and **a**2 are shown in figure 1.Folding of the (8,8) will lead to armchair tube structure as shown in figure 2. For single-wall carbon nanotubes with the nanotube axis normal to the chiral vector which, in turn, is along: (**a**) the $\theta = 30^\circ$ direction [an "armchair" (*n, n*) nanotube, as shown in figure 2:

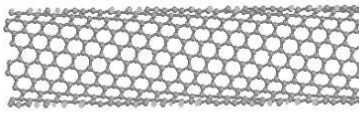

Figure 2. "armchair" (*n, n*) nanotube[1]

Folding of the (8,0) vectors lead to zigzag tube structure as shown in figure 3a . For single-wall carbon nanotubes with the nanotube axis normal to the chiral vector which, in turn, is along the $\theta = 0^\circ$ direction [a "zigzag" (*n,* 0) nanotube].[1-2]

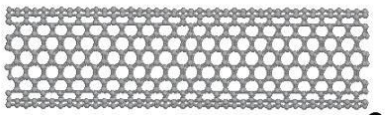

Figure 3 a. "zigzag" (*n,* 0) nanotube [1]

Folding of the (10,-2) vectors lead to chiral tubes as shown in figure 3 b. For single-wall carbon nanotubes with the nanotube axis normal to the chiral vector which, in turn, is along a general θ direction, such as *OB* (see Fig. 2), with $0 < \theta < 30^\circ$ *[1-4}*[a "chiral" (*n,m*) nanotube].

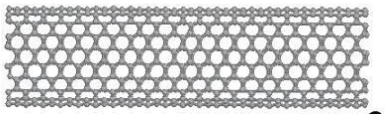

Figure 3 b. a "chiral" (*n,m*) nanotube [1]

Composite materials contain a matrix with one or more physically distinct, distributed phases, known as reinforcement phases or fillers. The reinforcement or filler is added to the matrix to obtain the desired or enhanced properties such as high strength, toughness, thermal and electrical properties, wear and damping resistance. Application of composite materials can be found anywhere especially from planes to cars and sports equipments. Callister (2007) et al [9] metal matrix composites (MMCs) are composite materials in which the basic constituent (the matrix) contribute at least 50 % by volume is a metal and the reinforcements whether one element or more could be a metal, ceramic or an organic compound. The materials used as a matrix in these composites are usually Al, magnesium, copper, titanium, Al-lithium, and super alloys. According to Bustamanate et. al. (2006) describe [10] Al-based (MMC) are widely spreading and required by many industries due to their relatively low density, high specific stiffness, and wear resistance. MMC's can be fabricated by dispersing reinforcing materials that have unique physical properties such as oxides, carbides in the Aluminium matrix. The composite carries the applied load by transferring it [10-14] from the matrix to the reinforcement. The main function of the matrix metal is to bind the reinforcement together and to transmit and distribute the external loads to individual reinforcement. This transfer of load depends upon the bonding interface between the matrix and the reinforcement. Bonding depends on the type of the matrix, reinforcement, and fabrication technique. Matrix can be selected on the basis of oxidation, corrosion resistance and other properties like a response to heat treatment, etc. The continuous fiber reinforced Al MMCs offer the best combination of strength and stiffness. However, based on the studies of Johnson (1987), the cost of these systems is very high, mainly because of high costs of the continuous fibers and of the production. These materials are mainly of interest for aerospace or military industries, where weight savings are very specific properties are of great importance than material cost. Among the attractive properties of these materials the most important one is the elevated temperature strength. The possibility of mechanical working on these materials is small, and they are hardly recyclable. Deuis et al. (1996) [91] showed that Aluminium-Silicon alloys and Aluminium based metal matrix composite hard particles offer superior operating performance and resistance to wear. Friend (1987) had found that the deformation and fracture behaviour of the composite revealed that the importance of particle size. A reduction in particle size observed to increase the proportional limit, yield stress and the ultimate tensile stress as per the studies of Lewandowski et al. (1991). It is well established that large particles are detrimental to fracture toughness due to their tendency towards fracture. It would be highly desirable to have a composite system where the reinforcing particles are relatively fine (4 μm or less) so as to get the stiffness benefits of a composite without significantly lowering fracture toughness. A uniform reinforcement distribution is essential for effective utilization of the load carrying capacity of the reinforcement. From the investigations of McKimpson and Scott (1989), non-uniform distributions of reinforcement in the early stages of processing was observed to persist to the final product in the form of streaks or clusters uninfiltrated reinforcement with their attendant porosity, all of which lowered ductility, strength, and toughness of the material.

Oberlin et al. (1976) had produced hollow tubes of carbon ranging between 2nm and 50 nm in diameter by decomposition of a mixture of benzene and hydrogen and had described the structure as —turbostratic stacks of carbon layers, parallel to the fiber axis and arranged in concentric sheets like the annular rings of a tree. Fabricate Aluminium matrix composite reinforced with CNTs. They fabricate a CNT-Al nanostructure composite by cold isostatic pressing (CIP) and subsequent hot extrusion techniques. The investigation of the microstructure and the measurement of the mechanical properties of composites are reported. It was reported that 1.0 wt. % carbon nanotube (CNT) reinforced 2024Al matrix composite was fabricated by cold isostatic press and subsequent hot extrusion techniques. The mechanical properties of the composite were measured by a tensile test. Meanwhile, the fracture surfaces were examined using field emission scanning electron microscopy. The experimental results show that CNTs are dispersed homogeneously in the composite and that the interfaces of the Al matrix and the CNT bond well. They found out that the hardness, Young's modulus, and tensile strength of the AI-CNT composite were enhanced by 30.8%, 41.3%, and 35.7% relative to the 2024Al matrix, respectively. Meanwhile, the elongation of the composite was maintained at 17.9%, which is equivalent to that of the matrix fabricated under the same process. Recently the same group studied the thermal expansion behaviors of the composites [Deng et al. (2008)]. In their work, aluminum matrix composite reinforced with 1.0 wt. % multi-wall carbon nanotubes (MWNTs) fabricated by cold isostatic pressing and hot squeeze technique. The coefficient of thermal expansion was measured between 25 and 400˚ C with a high-precision Thermo- mechanical analyzer and compared with those of pure aluminum and 2024Al matrix fabricated by the same processing. Finally, they concluded that addition of 1.0 wt. % MWNTs to 2024Al matrix decreases the CTE by as much as 12% and 11% compared with those of pure aluminum and 2024Al matrix at 50˚ C, respectively, which indicates that carbon nanotube reinforced metal matrix composite may be promising materials with low CTE. Deng et al. (2008) investigated the damping behaviors of the composite with frequency of 0.5, 1.0, 5.0, 10, 30 Hz, at a temperature of 25–400˚ C. It was reported that the damping capacity of the composite with a frequency of 0.5 Hz reaches 975×10−3, and the storage modulus is 82.3 GPa when the temperature is 400˚ C, which gives a high damping capabilities at an elevated temperature without sacrificing the mechanical strength and stiffness of a metal matrix. Esawi [92] et al. (2008) fabricate carbon nanotube-reinforced Aluminium strips using powder can rolling technique. In their work the Al-CNT mixtures are blended in either a mixer-shaker at a rotary speed of 46 rpm, or under argon in a planetary mill at a rotary speed of 300 rpm. The strength of the rolled strips is evaluated for various wt. % CNT samples. It is reported from their work that the Al-0.5 wt. % composite strips exhibited enhanced mechanical properties.Improved the strength of the aluminum composite using CNTs as reinforcements. In their research three major mechanisms are used for the strengthening of Al- CNT composites. They synthesize CNTs by arc evaporation method and fabricate the Al- CNT composite by powder metallurgy technique. They prepare the Aluminium composites reinforced by both single walled and multi-walled CNTs. Three mechanisms are employed to find the strength of the composites i.e. thermal mismatch, Orowan, looping and shear lag. The author concludes that the above mechanisms strengthen the Al-CNT composite synergetically. The attractive physical and mechanical properties that can be obtained with metal matrix composites, such as high specific modulus, strength, and thermal stability, have been documented extensively by Danels (1985), McKimpson et al. (1993) and Zhao et al. (1991). The various factors controlling the properties of particulate MMCs and the influence of the manufacturing route on the MMC properties have also been reviewed by several investigators such as Wang and Rack (1991), Margaret (1990), Jain et al. (1993) and Taya et al. (1991). Improvement in modulus, strength, fatigue, creep and wear resistance has already been demonstrated for a variety of reinforcements by Kirit and Mehrabian (1982) and Kelly (1973). Of these properties; the tensile strength is the most convenient and widely quoted measurement and is of central importance in many applications. Studied on finishing process effects in the process of spiral polishing process. In a micro-lapping process, dine in silicon and another micro-manufacturing process, the required surface finish need to be met and these surfaces were observed and studied in this work. Del stark et al of Brussels in 2006 investigated on the development of nano composite materials formed by different methods. Conducting organic polymers and CNTs were focused on their study; considering recent development purpose. HülyaCebeci et al. Studied on multifunctional properties of carbon nanotubes and carbon nanotube polymer composites. The composite was studied to enumerate the major properties getting enhanced on using CNTs as additives. Enrique Lavernia et al. investigated on mechanical properties of nanostructures materials, defined as having a mean grain size that falls in the 50-200 nm ranges, is reviewed and the underlying mechanisms are discussed. Particular emphasis is placed on nanostructures materials that are processed via two synthesis approaches consolidation of Nano-crystalline powders and electrode position. Their view demonstrates that processing history significantly influences mechanical behaviors as revealed by the following observations. Russelmcenzi book on applied composite materials introduced the various property variations possible in composites especially when there is an application oriented-approach done in developing the composite. X D Yang et al studied on synthesis on carbon nanotube reinforcement in Aluminium powder by in situ chemical vapour deposition method. The effect of Ni/Al catalyst on reaction time for CNT/Al composite was studied. FESEM and TEM characterization of the composite was performed and the study emphasizes on a new way to prepare carbon nanotubes. L Girisha et al studied on fabrication of multi walled CNT reinforced aluminium MMC

by stir casting method. The study showed that an 80% increase in hardness was observed with a small percent addition of the composite. Moreover, a 2 wt.% addition of MWCNT made a 20% increase in strength of the MMC.

3. SYNTHSIS OF CARBON NANOTUBE BY ARC DISCHARGE METHODS

a. Preparation of carbon nanotubes by Arc Discharge Evaporation: [15-18]

The apparatus used by them to produce carbon nanotubes was similar to what had been used till then for the production of ultrafine powder of SiC with some changes [15]. The setup consisted of a vacuum chamber of 30cm diameter with two vertical carbon electrodes of which cathode being 10mm diameter, the upper carbon electrode being a movable cathode. The arc current for the setup had been kept constant at 220A at 25V. The carbonaceous soot [16-18] particles were observed to be deposited on the cathode tip after the arc discharge evaporation in the presence of Helium gas. The Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) results of the particles confirmed the existence of the carbon nanotubes. The test was carried out under the pressure conditions in the range of 20-200 torr. The gap between electrodes was kept constant to avoid any geoterical effects on the evaporation throughout the experiment. The test results have shown that the optimum pressure for the Helium and Methane gas is around 50 torrs whereas for Argon it was around 20 torr. There was no carbon nanotubes formed below the 10 torr pressure.

b. Synthesis of various forms of carbon nanotubes by AC arc discharge method [19-25]

In this experimental setup two graphite rods (99.99% pure) of the same diameter (15mm) were used. Each of the electrode was having a drilled hole of 7mm diameter and 80mm depth, packed with a mixture of cobalt and graphite powder (weight percentage of cobalt was 10%). The helium pressure was maintained at 140 Torr [19]. The voltage and the current were kept at 20-30 V and 700 A, respectively. After the arc was carried out, instead of depositing on the electrode, a number of nanotubes were found in the soot deposited on the wall. Some forms of nanotubes different from those which had been reported previously were found in the soot (but not the cathode deposit). These were the carbon nanocapsule, carbon cone-shaped nanotube and the carbon toroidal nanotube, respectively. The soot obtained was purified and sonicated using o-dimethylbenzene, nitric acid and ethanol respectively. Preliminary works showed that there are no nanotubes present in the soot produced by two pure graphite rods of the same diameter without cobalt addition under the same conditions. The experimental results thus showed that since the arc was run between two electrodes of the same diameter, there is no deposit on the face of either of the two graphite rods. In the previous studies in which the DC arc discharge method was used, the cathodes were always larger than the anodes in their diameters. The carbonaceous matter thus used to deposit on the cathode owing to its low current density. In the AC arc discharge method, the plasma was in resonance periodically because of the periodical alternation of the electric current. Owing to the fact that the diameters were of the same value in this experiment and hence the current densities of the two electrodes were equal. Since the temperature of the two carbon rods was very high, the nanotubes were unable to form on either of the electrodes. Only when the reactants moved out of the plasma because of thermal movement, could their reactivity decrease step by step. Therefore, in this experiment, the multi-walled nanotubes were deposited on the reactor wall, but not on the faces of the graphite electrodes. This is a significant difference between the AC and DC arc discharge method.

c. Synthesis of single walled carbon nanotubes by AC arc discharge method [26-31]

Till date the only method known to synthesis the single walled carbon nanotubes was through the DC arc discharge method, which was not very efficient as the single walled carbon nanotubes were not observed in the carbon deposit formed on the cathode. Hence this method was rendered ineffective for efficient synthesis of the SWCNTs. On the other hand the possibility of getting good yield of SWCNT becomes high as all the carbon evaporated deposits as soot, and not as a carbon deposit on the cathode. Thus this possibility was explored in the experiment.[26] The experimental setup used is shown in the figure 5, in which two electrodes are installed vertically at a distance of 2-3 mm. Carbon rods containing catalytic metals, which were mixed with metallic oxides of Ni, Fe, Co, Y, La and Cu were used as electrodes. Each of the carbon rods had 5% of only one kind of metallic oxide. The dimensions of the carbon rod were 6mm in diameter and 50mm in length. Helium gas at 400torr was used in the setup, discharge current being AC 85A [26-29] with evaporation period of 1-2 min. After the evaporation all the carbon in the form of soot had deposited on different parts of the apparatus. The soot adhering to each of these points was collected, and observed by TEM which confirmed the presence of SWCNT. The synthesis efficiency of SWNTs was

found to be the highest when combinations of three kinds of ferromagnetic metals (Ni, Fe, Co) were used [29-31].

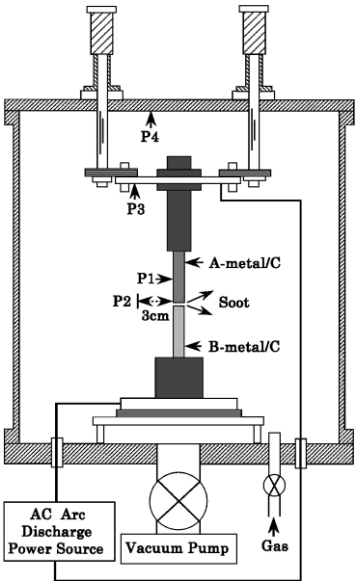

Figure 5 . The apparatus used for carbon nanotube formation [26].

Thus it was concluded that since there was no carbon deposit s formed on cathode in case of AC arc discharge and all the evaporated carbon becomes soot in which SWNTs exist. So this method was far superior for high efficiency SWNT synthesis.

d. Double walled carbon nanotubes fabricated by a hydrogen arc discharge method. [32-37}]

J.L. Hutchisona et al [32] devised a method to obtain double walled carbon nanotubes (DWCNT) through the conventional arc discharge method. This selective production of DWCNT was important for establishing the correlation between different types of physical properties and the obtained nanotubes.For this the arc discharge evaporation of a metal graphite electrode was used. The anode was a graphite rod of 8.2 mm diameter having a drilled channel of 3.2 mm diameter and 140 mm length, stuffed with the catalyst (a mixture of Ni, Co, Fe powders). The process was carried out with an arc current of 75-80A in an atmosphere of Argon and Hydrogen gas mixture (1:1 / v: v) at 350 torr. The voltage was kept at 26-28 V and the gap width was kept constant at 2 mm. After about 40 min of the process there were two kinds of carbon material obtained: (1) The product deposited on the reactor wall and (2) Remaining product deposited on the electrodes. These products were then characterised with the help of TEM, HREM and Raman spectroscopy [32-37]. The HRTEM images confirmed the presence of DWCNT, the outer diameter in the range of 1.9 to 5 nm and the inner diameter being in the range of 1.1 to 4.2 nm. Also, it has been shown that the addition of Sulfur to the starting mixture of Carbon and Cobalt significantly increased the yield of SWCNTs and also upon further investigation it was seen that the presence of sulphur was not critical for the production of DWCNTs. It was also observed that the conditions used in this experiment were optimum and even the slightest deviation led to the domination of SWCNTs in the product mixture.

e. Optimisation of the arc discharge production of the multi walled carbon nanotubes. [38-45]

M. Cadek [38] et al studied the effect of varying current density and pressure during arc generation on the yield and purity of multi walled nanotube containing carbon soot. The results were quite encouraging as well. The setup [38] used is the Kra¨tschmer generator which consists of the cylindrical steel chamber with dimensions of 300mm in length and 100mm in diameter. The chamber filled with Helium gas encloses two graphite electrodes, a stationary anode (6.1mm) made of 99.5% pure graphite and a 99.998 % pure, movable cathode (diameter 4.6mm). The voltage was kept constant at 23V with the current density being varied from 165-195 A/cm2 and the helium pressure being varied from 300-500 Torr [38-42]. During the process the cathode sublimed and the deposit was formed on the anode owing to the low current density of the anode. The purity of the nanotubes produced was measured using EPR and TGA techniques [38-45].The results showed the increase in the yield of the MWCNTs with increase in the current density and pressure of the Helium gas in the chamber. For current densities of 165 A/cm and pressures of 300 Torr low yields are observed in the region of 2.6 mg/min. While current densities and pressures are increased to 180–185 A/cm and 400 Torr, respectively, the yield plateaus at regions of between 6 and 12 mg/min. As these parameters are further increased the yields increase sharply to 22 mg/min for the 195 A/cm2, 500 Torr sample and 24 mg/min for the 190 A/cm2, 500 Torr sample. The high yield samples were analysed using the micrographs, which clearly showed that not only the yield was high for them but also the purity was high (upto 48%) as these samples contained less impurities, which was later confirmed through EPR and TGA [38][43]. This clearly shows that careful choice of current and pressure are critical to the optimisation of nanotubes production.

f. Low pressure synthesis of single walled carbon nanotubes by arc discharge [46-54]

In the earlier journals it was shown that the yield of SWCNTs was a direct function of pressure and was found to increase

with the increase in the gas pressure. But in this research paper Young Soo Park et al showed that high yield of SWCNTs was possible even at lower pressure.

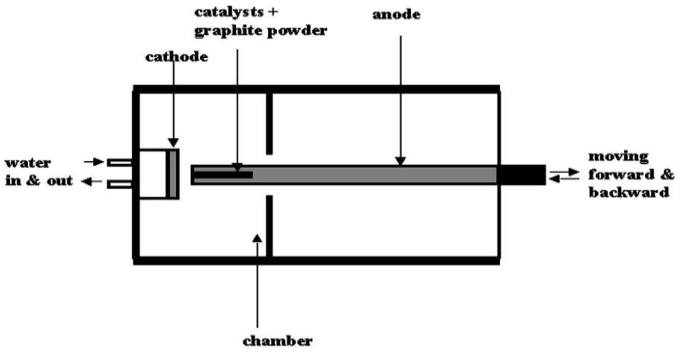

Figure 5. The scheme of arc discharge methods [46]

The apparatus used in this experiment was as shown in figure5 [46]. It consists of a stainless steel chamber of 150mm diameter and reactor of 150mm length. The cathode and the chamber wall being cooled by the water. The graphite electrode of 6mm diameter act as an anode where a hole of 4mm diameter and 90mm in length is drilled which was filled with mixtures of two or more kinds of catalysts. The anode was attached to a step motor which controlled the distance between the two electrodes. The graphite electrode of 25mm diameter was used as a cathode. Helium at a pressure range of 100-500 Torr [46-48] was used in the experiment. The voltage and current was maintained at 25 V and 60-80 A respectively. The soots are collected in the cathode and the chamber walls, which was analysed by using SEM, TEM, Raman and TGA. The results showed that with the rods containing mixture of graphite, Nickel, [46-54]Iron and Sulfur the yield was maximum at the gas pressure of 100 Torr. And also Sulfur was responsible for the existence of large diameters of CNTs.

g. Large scale synthesis of carbon nanotubes by plasma rotating arc discharge technique [55-67]

It is already a known fact that arc discharge is one of the major CNT synthesis process. But the fact that the yield of the CNTs can be further increased was explored by Seung Jong Lee and others, when they went for a setup where instead of keeping the electrodes stationary they rotated the graphite electrode at high speeds. The results obtained were quite encouraging.

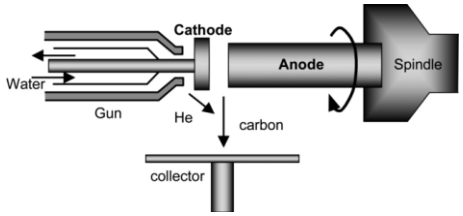

Figure 6. Schematic diagram of plasma rotating electrode process system. [61]

It was a well-known fact that the conventional arc discharge method is a discontinuous and unstable process, as electrode spacing is not constant and hence the current flow being not uniform. As a result the density of carbon vapor and the temperature distribution is non-uniform and carbon nanoparticles and impurities always coexist with nanotubes. These reasons limit the production of high quality CNTs on a large scale. To eliminate this problem Plasma rotating electrode process was introduced. A rotating electrode distributes the micro-discharge uniformly and it makes the plane-to-plane micro-discharge in comparison with the point-to-point discharge in conventional arc discharge. The experimental setup is as shown in figure 6 [61]. The anode was rotated at a speed of 0–10 000 rev/min. Carbon plasma was generated between 12 mm dia pure graphite anode and the 15-mm dia pure graphite cathode. The discharge current was changed from 80 to 120 A [55-61] with a voltage of approximately 20–30 V. The gap between electrodes was maintained at 3 mm. The pressure within the chamber was maintained at 500 torr with inert He gas flow at 5 1/min. The carbon evaporated in the anode was deposited in the collector instead of the cathode due the centrifugal forces. Thus PREP allows continuous production of CNTs. The products obtained were analysed using the XRD, SEM, TEM and Raman [61-67]. It was observed that as the speed of rotation increased the CNT yield also increased. Also, it was seen that as the distance between the collector and the electrode decreased the CNT yield increased. Hence this method was seen to be utilized for mass production of CNTs.

h. High-yield synthesis of multi-walled carbon nanotubes by arc discharge in liquid nitrogen [68-75]

Among all the methods the arc discharge method still remains the most sought after for the large scale synthesis of MWCNTs.[68-71] But the conventional arc discharge method, however, requires a complicated vacuum apparatus with an efficient cooling system. Olk and Ishigami et al. proposed a modified method for the synthesis of CNTs, in which liquid nitrogen was filled in the arc discharge chamber. Liquid

nitrogen prevents the electrodes from contamination with unwanted gases and effectively lowers the temperature of the electrodes. Moreover, since the CNTs do not stick to the walls of the chamber, hence we obtain a clean sample CNTs. In this letter, H. Jung and co report that the content of the CNTs in the reaction products can be as high as 70%with an arc discharge in liquid nitrogen, so that this method can be an economical route for the mass production of highly crystalline CNTs. The apparatus used in the experiment is as shown in fig. The anode is a pure carbon rod of 8-mm diameter and the cathode is a pure carbon rod of 10-mm diameter. The flask was filled with Nitrogen and the electrodes were dipped into it. The voltage was set in the range of 20 V - 27.5V and current at 80A. Due to low current density on the cathode the carbon was deposited on the cathode. The Field Emission SEM [68-75]was used to analyze the morphology of the carbon deposited on the cathode.

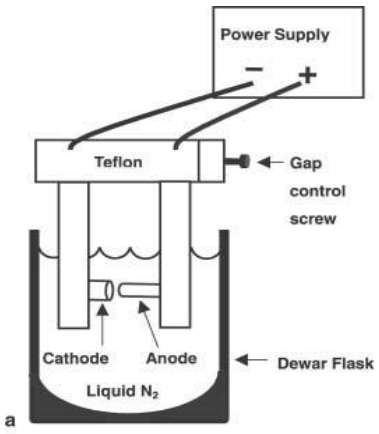

Figure 7. Schematic drawing of the arc discharge apparatus [68].

Through the observed results it was concluded that MWCNTs as high as 70% of the sample size are possible through this method. Also, since the apparatus and its operation are simple, and the scale-up is straightforward, this technique can be a practical option for the large-scale synthesis of MWNTs with high purity.

i. New Synthesis of High-Quality Double-Walled Carbon Nanotubes by High-Temperature Pulsed Arc Discharge [76-83].

From what we see in the current trend and scenario the SWCNTs easily overshadows the MWCNTs in terms of applications due to the low structural uniformity of the MWCNTs. But SWVNTs has been an exception to this case as they are having the thinnest graphite layer structure with excellent graphitization as reported in SWNTs. Usually the DWNTs have been produced by several methods such as the electric steady arc discharge method,6,7 the catalytic chemical vapor deposition (CCVD) method, etc, which usually gives DWCNTs with large diameter distributions (2 to 5 nm) and thus limiting the applications of the DWCNTs. It was soon realized that thinner DWNTs can offer intriguing device applications in nano-electronics.[76-79]This research article reports a high-yield synthetic method and purification of thin DWNTs with an inner and an outer diameter of 0.8-1.2 and 1.6-2.0 nm, respectively. The experimental setup (figure 7) of the high temperature pulsed arc discharge method has been described as follows. The apparatus consists of a furnace (ISUZU KRO-12K), a quartz tube (_ 25 mm), graphite electrodes, a water-cooled trap, and a homemade pulsed HV power supply capable of providing 1.5 kV and 100 A. [76-83] The electrodes of graphite doped with catalytic metal atoms (Ni/Co 0.7/0.7 at. % and Y/Ni 1.0/4.2 at. %: Toyo Tanso Co. Ltd.) were located at the center of the furnace. The pulsed arc discharges (600 ís, 40-60 A, and 50 Hz) were generated between the electrodes, which vaporized the cathode in high-temperature Ar buffer gas (1000-1400 °C).11-13 The vapor from the cathode was annealed in the buffer gas and was converted into soot containing DWNTs. The CNTs produced after the experiment were purified and then characterized using Raman spectroscopy, SEM and TEM.It was observed that all the DWCNTs were produced with Ni/Co catalysts over the whole temperature range and the arc pulse width range studied (300 ís-100 ms and 1000-1400 °C, which is due to the fact that Ni/Co catalysts tend to produce much narrower SWNTs than the Y/Ni case in the pulsed arc discharge.11-13. The preferential formation of SWNTs and DWNTs seems to be related to the diameters of the nanotubes. The results suggest that thicker nanotubes are necessary to form DWNTs. This tendency is supported by the temperature dependence where the average diameters and the abundance of DWNTs increase as the furnace temperature increases. Thus the present DWNTs can thus be viewed as the narrowest “nano-coaxial cable” ever made.

j. Large scale and high purity synthesis of single-walled carbon nanotubes by arc discharge at controlled temperatures [30] [84 -90]

Before going into the research some of the challenges associated with the synthesis of SWCNTs by conventional arc discharge method were low yield and purity. So this work was focused on improving upon those disadvantages. In

order to improve this, a modified the conventional arc discharge apparatus was used. The apparatus (figure 8) could freely control the temperature of the chamber in the range from room temperature to 900 ˚C. Furthermore, the modified arc discharge furnace could prepare SWNTs in large scale (>45 g/h) and high purity (95% after purification) on the wall of the chamber, comparable to the HiPco laser ablation method. The experimental apparatus was designed by Liu et al.The vacuum chamber is Φ300 · 400 mm2. And there is a heating apparatus mounted outside the chamber to control the temperature inside the chamber. There are six Φ 6 · 100 mm2 graphite rode anode with a Φ 4 · 60 mm2 hole drilled in them which is filled with a 19:1 (weight ratio) mixture of Fe-Ni-Mg (2:1:2 wt%) powders. The six anodes are mounted at an equal distance from each other on a wheel which can be rotated in order to change the active anode. In our experiments: an arc was generated between the anode and the pure graphite cathode at a current of 60 A in a 400 Torr of static helium atmosphere. The distance between the electrodes was maintained at around 2 mm by continuously

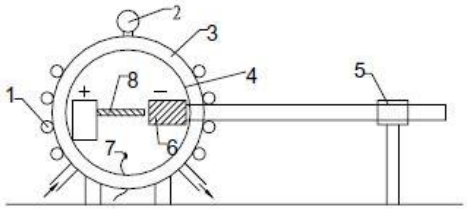

Fig. 1. Above is a schematic drawing of our arc discharge apparatus which is capable of carrying out experiments at different temperatures. 1—Water-cooling system. 2—Vacuum pressure-meter. 3—Vacuum chamber. 4—Temperature controlled apparatus. 5—Electrode feeding system. 6—Moving cathode. 7—Thermal couple. 8—Fixed and rotated anode.

Figure 8. Arc discharge apparatus.[30].

feeding the cathode throughout the arc process. A cloth like soot had formed on the entire inner wall of the chamber and, in general, an 80 mm anode rod was used up in 5 min and 5.3 g of soot could be collected. The SWNTs were studied using SEM, TEM, HRTEM, XRD [30] [84-90] (CuKa) and Raman scattering spectroscopy. The results of as grown SWNTs prepared at temperature of 25, 300, 400, 500, 600 and 700 ˚C were compared.It was seen that the temperature strongly affects not only the yield of SWNTs but also the purity and the bundle diameter distributions. The optimum temperature of 600 ˚C was observed. Beyond this the yield decreased a bit. The reason for such behaviour was explained, as the temperature played an important role in increasing the SWCNT content in the soot, also the catalytic behaviour was assumed to have increased with temperature. The environmental temperatures preheat the catalyst, which leads to the shortening of the time for catalyst to activate and accelerates the growth of carbon nanotubes. When the temperature is 700 ˚C or higher, the yield decreased which may be explained by the thermal decomposition of SWNTs or a decrease of catalytic activity. The SWNT bundles with diameters 7-20 nm and the production of 45.3 g/h were prepared at 600 ˚C. By purification, the purity was found to be greater than 95%. The Raman spectrum showed that the diameter is uniform distributed and is about 1.24 nm.

4. CONCLUSIONS

The paper presents a detailed analysis of the advancements over the past in the field of Carbon Nanotubes synthesis through Arc Discharge method. It was seen that after the discovery of CNTs by Ijima arc discharge emerged as the major syntheis method for both SWCNTs as well as MWCNTs. It was seen that during a DC arc discharge method the carbonaceous matter gets deposited mostly on the electrode having the lower current density i.e having larger diameter. And it is a well known fact that the majority of the SWCNT is present in the soot deposited on the wall of the chamber, hence DC arc discharge method rendered almost ineffective in the production of the SWCNTs. However if we use AC arc discharge method with two equal electrodes we are sure to observe the soot getting deposited on the wall instead of the electrode, hence increasing the yield of SWCNTs. Also, it was shown by J L Hutchisona et al that extremely careful conditions led to the formation of DWCNTs as well in addition to the others, so this method was a tool to produce DWCNTs. Also it is seen that there is an increase in the yield of the MWCNTs in the presence of the liquid Nitrogen as shown by Olk & Ishigami et al and also there is a significant increment in the production of the MWCNTs with the increase in the current density and pressure of the chamber gas to certain extent. There was a major improvement in the yield of the SWCNTs and DWCNTs by rotating the Anode ie. using Plasma rotating technique. Also, it was observed that there was an improvement in the synthesis and purity of SWCNTs with the increase in temperature till 600 ˚C.

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