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Carbon Nanotube-Polymer Composites for Sensor Applications

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Abstract:
Carbon nanotubes (CNTs) have many distinct and superlative mechanical, electrical and thermal, properties that have been exploited to develop variety of commercial, industrial and scientific applications. In the recent past decade, it has been shown that combining the intrinsically high aspect ratio (micrometer length/nanometer diameter) CNTs as fillers in polymer matrices may have further advantages for various applications. The CNTs besides being widely applied as reinforcement for enhancing the mechanical properties of polymers, their application also can extend the electrical conductance of insulating, or conducting polymers. The conductive CNT-polymer composites can be manipulated for sensing various entities such as gas/vapour, strain, temperature, pressure, and fluid flow. Similarly, in conjunction with optically active, bio sensitive or piezo resistive polymers, the sensing functionality for such CNT-polymers can also be advantageous. Indeed, the CNT-polymer composites for sensor applications have emerged as an active area of research and development for next generation sensors. In this article, a review of recent developments in conductive CNT-polymer composite material systems, their properties and applications for sensors are presented, with emphasis on composite systems for gas- sensors, strain sensors and infra-red sensors

Keywords: *CNT, Polymer, Composites, functionalization, Sensors*

1. Introduction

Carbon nanotubes were discovered [1] in 1991, following to the discovery of carbon Fullerences developed a few years earlier [2]. Fullerenes are geometric cage-like structures of carbon atoms that are composed of hexagonal and pentagonal faces. The first closed, convex structure formed was the C60 molecule. Nanotubes are long, slender fullerenes where the walls of the tubes are hexagonal carbon (graphite structure) and often capped at each end. Carbon nanotubes (CNT) can be visualized as a sheet of graphite that has been rolled into a tube. The CNTs with such cage like symmetric structures have generated huge activity in most areas of science and engineering due to their extraordinary properties. The CNTs are known to be many-fold stronger than steel, harder than diamond, of electrical conductivity higher than copper and thermal conductivity higher than diamond. Also, the individual CNTs can be metallic or semiconducting with electron mobilities far higher than silicon, presently the most important semiconductor for electronic applications. No previous material has displayed the combination of such superlative mechanical, thermal and electronic properties attributed to them. Referred in the next section of this article, the Table1 provides a summary of CNT properties. These properties make carbon nanotubes ideal for a wide range of present and future commercial applications [3] and also for fundamental science research [4]. The number of CNT-related papers on fundamental as well as application research has been increasing, indicating that balanced research between fundamental and applied sciences for CNTs has been developed [5].

The unique mechanical and physical properties of nanotubes combined with their high aspect ratio (micrometer scale length /nanometer diameter) and low density (theoretical $\approx 1.3 \mathrm{gcm}^{-3}$) have brought about extensive research in creating CNT- polymer composite material systems to exploit these properties [6,7]. The carbon nanotube (CNT)-Polymer/composites are expected to have a blend of good processability and other useful characteristics of the polymer with excellent physical and functional properties of the CNTs.

The concept and practice of using *micro* dimensioned filler material such as alumina, silicon carbide and glass or carbon as a reinforcing agent to enhance mechanical properties of matrix materials including polymers has been fairly common. Similarly, using conductive carbon or graphite as fillers in insulating polymer matrix, conducting composite could also be prepared. The composite properties with micro scaled fillers generally could be predicted by the well known rule of mixture by using a weighted mean of the constituents of the composite [8]. However, in such composites, to impart a significant change in the matrix properties, larger volume fraction of fillers were necessary.

The concept of polymer composites changed drastically when the fillers of nano dimensions were used and a new class of polymer composites, the so-called Polymer nanocomposites emerged. As nano fillers are only a few nanometers (~10,000 times finer than a human hair) in dimension, ultra-large interfacial area per volume between the nano-element and polymer matrix is offered, allowing much greater interaction with polymer matrix. For this advantage of nano dimensionsed fillers, according to percolation theory [9], a low percolation threshold for the filler in the composite in tailoring the composite property can be obtained. Compared to different range of nanofillers, carbon nanotubes (CNTs) have emerged as the most promising nanofiller for polymer composites due to their remarkable mechanical and electrical properties.

Considerable interest has focused on utilizing carbon nanotubes as a filler or reinforcement of polymers to tailor mechanical [10-12] electrical [13-16] thermal [17-19], electro-thermal [20-24] electro-mechanical [25-30] and opto-electrical [31-36] properties of CNT-Polymer composites. Indeed, the first CNT-polymer composite report of Ajayan et al [37] appeared as early as in year 1994.

There is already a great potential for exploiting CNT reinforced -polymer composites for structural applications [3,6], the recent past trend has shown an intensive research focusing on their other functional properties, particularly the electrical conductivity. A large motivation comes from remarkable enhancement of electrical conductivity by several orders of magnitude at very low percolation thresholds (<0.1 wt% of filler in the matrix) of composites of CNTs in polymer matrices without compromising other performance aspects of the polymers such as their low weight, optical clarity, low melt viscosities, ease of processibilty [38-41]. The CNTs have demonstrated decisively their capability as fillers in diverse multifunctional nanocomposites where beyond a simple physical combination of the CNTs and polymer properties, some synergistic effects and new features appear that can be developed into applications [42-50]. The synergies between CNTs and conducting polymers have allowed the application of their composites to energy conversion and storage, including rechargeable lithium-ion batteries, supercapacitors, photovoltaic devices, organic electronics/transistors and printable conductors, gas-sensors and bio-sensors.

The research on the topic of CNT-polymer and their applications is indeed very vast and to make a comprehensive overview of all aspects of this large subject in the framework of one article cannot be an object here. Therefore, to keep the task manageable, the present review restricts on issues especially of electrically conductive CNT/polymer nanocomposites using insulating or conductive polymer matrix and their applications to various sensors prominently, the gas/vapor sensors, piezo-resistive or resistance-type strain sensors and infra-red sensors based on bolometric effect.

The properties of carbon nanotube have significant implications on the CNT-polymer composite properties. With this view, the features and properties of CNTs have also been included in this conductive CNT-polymer composite article.

2. Carbon Nanotubes

2.1. Structure and Morphology

Conceptually, single wall nanotubes (SWCNT) can be formed by rolling a graphene sheet (hexagonal structure) into cylinder and a multi-walled nanotube (MWCNT) is composed of concentric graphene cylinders with an interlayer spacing of 0.34 nm. Atomic structure and morphology of Carbon nanotubes can be visualized as a sheet of graphite that has been rolled into a tube. Fig 1 shows the graphite structure as a 2-D sheet of carbon atoms arranged in a hexagonal array. The carbon nanotube can be visualized by cutting the graphite sheet along the dotted lines and rolling the tube so that the tip of the chiral vector $\boldsymbol{C_h}$ touches its tail. The chiral vector, often known as the roll-up vector, can be described by the following equation:

$$\boldsymbol{C_h} = n\,\boldsymbol{a_1} + m\boldsymbol{a_2} \qquad (1)$$

where the integers (n, m) are the number of steps along the ziz-zag carbon bonds of the hexagonal lattice and $\boldsymbol{a_1}$ and $\boldsymbol{a_2}$ are unit vectors, shown in Fig.1.The chiral angle(θ) determines the amount of 'twist' in the tube. There may be two limiting cases for chiral angle; (i)$\theta=0^0$ where C-bond geometry around the circumference of the nanotube is 'zigzag' and (ii) $\theta=30^0$, where the C-bond geometry is like an 'armchair' as may be seen in Fig1. Also, it may be noted that the integers (n, m), for the zigzag case become (n, 0) and for the armchair (n, n). For any other values of n and m the tubes are chiral tubes. The pictorial view of above structural classification of carbon nanotubes is shown in Fig 2.

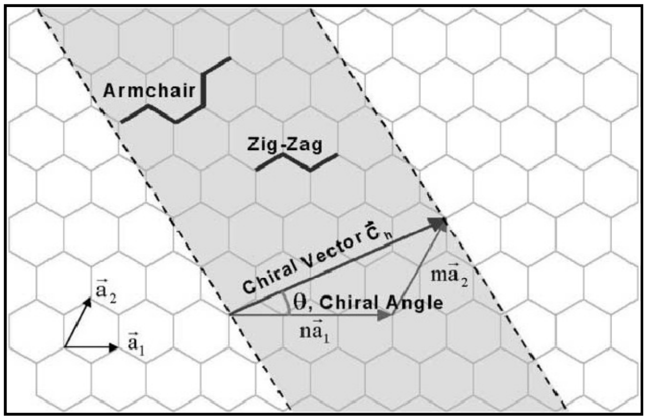

Figure 1: Hexagonal arrangement of C atoms in planar graphite sheet. Construction of the CNT can be achieved by cutting the graphite sheet along the dotted lines and rolling to a tube so that the tip of the chiral vector C_h touches its tail [Thostenson et.al [6]

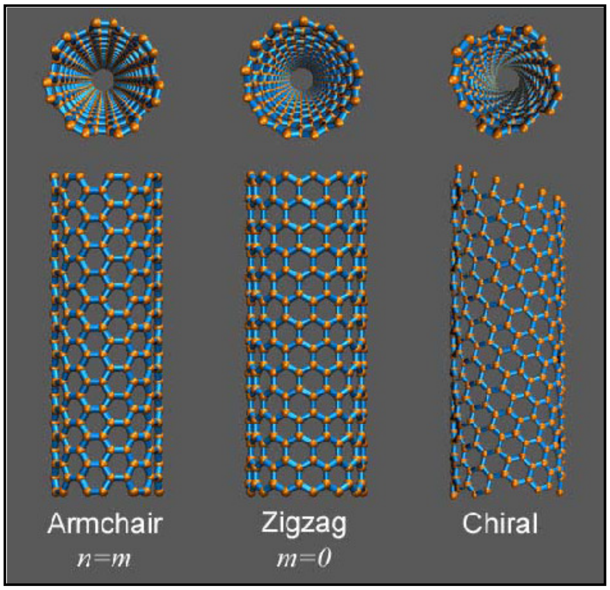

Figure 2: Structural classification of CNT ; the Arm chair(n,n), Zigzag (n,0) and Chiral (n,m)

As mentioned before, fullerenes are closed, convex cages that are composed of pentagons and hexagons. Under stress or tension, the Stone-Wales transformation may introduce a new defect, the heptagon in the nanotube structure [51-52]. The Stone-Wales transformation is a reversible diatomic interchange where the resulting structure is two pentagons and two heptagons in pairs, heptagons allow for concave areas within the nanotube. The heptagonal defects in nanotubes can result in many possible equilibrium shapes. In fact, most nanotubes are not ideal straight cylinders with hemispherical end caps.

2.2. Properties

Nanotubes are formed in a range of diameters and may be singlewalled or multi-walled, depending on their fabrication process and reaction conditions. The single-walled variety exist as a bundle made up of strands, like a rope, in which each strand is a single-walled nanotube ; each bundle contains typically 10 – 50 strands [53]. The diameter of SWCNTs is of the order of a nanometer (~ 1.4 nm), and the inter tube spacing within the bundle has been determined to be ~ 0.3 nm, at closest approach [53]. As said earlier, the MWCNT are comprised of several graphene sheets rolled up, resulting in concentric tubes with successively larger radii. Multi-walled nanotubes also aggregate, due to significant van der Waals forces.

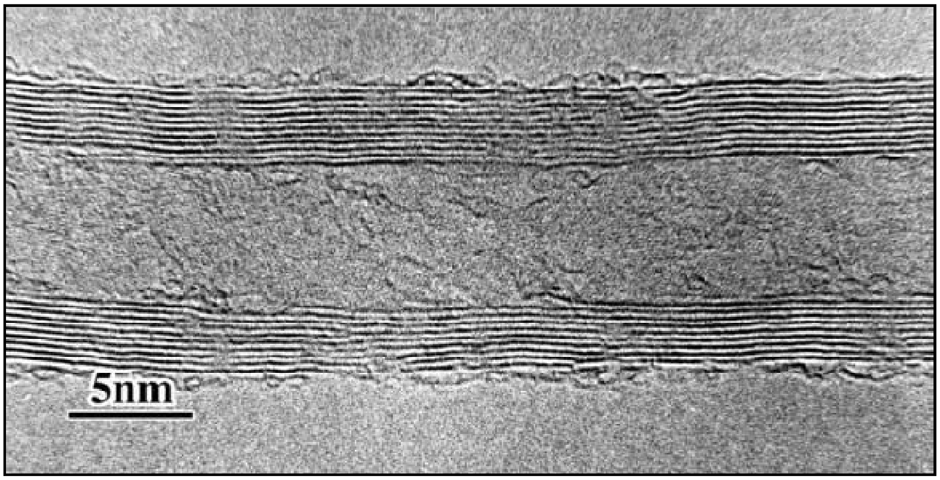

Figure 3: TEM image showing the nanostructure of a typical multi-walled carbon nanotube; several layers of graphitic carbon and a hollow core are evident [Ack: Thostenson et.al [6]

The diameter of MWNTs is typically 5 – 20 nm, and the interlayer spacing has been determined to be ~ 0.34 nm) [53]. Fig 3 shows a transmission electron microscope (TEM) image showing the nanostructure of a multi-walled carbon nanotube where several layers of graphitic carbon and a hollow core are evident.

Due to their nano-scale size, carbon nanotubes possess extremely large surface area; BET (Brunauer-Emmett-Teller) measurements on MWNTs yield surface areas between 100 and 200 m^2/g, which is higher than graphite, [54]. Furthermore, the CNTs possess a very low density, ~ 0.6 g/cm^3 and 1 – 2 g/cm^3 for SWCNT and MWCNT, respectively [54].

The diameter and length of the tubes, the morphology, nano structure and the atomic arrangement or chirality of the carbon nanotube have significant implications on the material properties. In particular, tube chirality is known to have a strong impact on the electronic properties of carbon nanotubes. Depending on chirality, carbon nanotubes can either be conducting or semiconducting. Armchair nanotubes (n=m) are metallic. For all other chiralities of tubes, there are two possibilities. If (n-m)=3N, where N is an integer, the tubes are expected to be metallic; otherwise, the tubes are semiconducting with an energy gap of approximately 0.5 eV [55]. Statistically, given a random distribution of nanotubes, approximately ⅓ are metallic and ⅔ are semiconducting [53]. In the semiconducting case, the energy gap scale inversely with diameter, with increasing diameters leading to decreased energy gaps [55] from approximately 1.8 eV for very small diameter tubes to 0.18 eV for the widest possible stable SWCNT [56].The diameter, d of the tube can be determined from [53]-

$$d = \frac{\boldsymbol{C_h}}{\pi} = \frac{a_{c-c}\sqrt{3}}{\pi}\sqrt{n^2 + m^2 + nm} \qquad (2)$$

Where, a_{c-c} is the nearest neighbour distance in graphite (=1.42 Å).

In general, singlewalled carbon nanotubes (SWCNTs) are a mixture of metallic and semiconducting material, depending sensitively on their geometrical features, while multiwalled carbon nanotubes (MWCNTs) are regarded as metallic conductors. Especially for SWCNTs, the electronic properties vary from metallic to semiconducting depending on their structures. Additionally, as a result of the 1-D nature of CNTs, electrons can be conducted in nanotubes without being scattered. The absence of scattering of the electrons during conduction is known as ballistic transport and allows the nanotube to conduct without dissipating energy as heat so that nanotubes can carry very large current densities of up to 10^8 A/cm^2 [57]. The conductivity of single MWNTs have been evaluated experimentally, and the conductivity range from 10^5-10^7 S/m have been observed [58]. Although it is still smaller than the conductivity of the copper, gold or silver (10^8 S/m), it is reaching the conductivity of the crystalline graphite, ~10^7 S/m. In addition, carrier mobilities as high as 10^5 cm^2/V-sec have been observed in semi-conducting nanotubes [59]. Superconductivity has also been observed in SWNT, though only at very low temperature of 5K [60]. Nanotubes are also very conductive for phonons. Theoretically, a room temperature thermal conductivity of up to 3000-6000 W/m K have been predicted and upto 2000W -3000/mK have been practically achieved [17, 61].

The mechanical properties of CNTs are excellent; theoretically, calculated values of Young's modulus of SWCNT are 1000-1500 GPa, similar to that of graphite [62], significantly, the values are independent of the diameter [63,64]. The experimental values of Young' modulus for SWCNT are measured to be 1 TPa for small diameter SWNT bundles and are fairly close to theoretical values [64,65]. In comparison, the experimental modulus values of MWCNT are 0.27–0.95 TPa. However, the fracture strength of SWCNT has shown a large variation from 50 -500 GPa and so also for the MWCNT in the range 11–63 GPa [63, 64]. Although the chirality has a relatively small influence on the elastic stiffness, it is said to play a key role in the nanotube plastic deformation under tension [51]. It has been theorized that the Stone-Wales transformation results in ductile fracture for armchair nanotubes. [65, 66].

A summary of CNT properties from literature data has been presented in Table 1. In summary, these superior mechanical properties make carbon nanotubes suitable as reinforcing materials in composites.

2.3. Carbon Nanotubes as Fillers

Due to their excellent properties and special morphology namely the low density, high aspect ratio, nano sized diameter, CNTs have established their potential to be used as the perfect filler for polymer composites. Since the early preparation of a CNT/epoxy composite by Ajayan et al. [37] in 1994, very large type of polymer matrices have been investigated with respect to reinforcement by CNTs to improve mechanical-strength, elastic properties, electrical conductivity, thermal conductivity, opto-electric, electro-mechanical properties. However, as said earlier, this review essentially covers the polymer matrix of insulating polymers and conductive polymers where MWCNT /SWCNT are dispersed as fillers to enhance the conductivity of the polymers.

Much effort has gone into the preparation of CNT-polymer composites using various insulating as well as conductive polymers matrices. The insulating polymers, such as polystyrene(PS) [66, 67], epoxy [68,69] polycarbonate(PC) [66,70-72] polypropylene (PP) [73,74], polumethylmethacrylate(PMMA) [75], polydimethlysiloxane(PDMS) [76], polyurethane (PU) [25,77] poly ethylterphthalate (PET) [78] and many more [79,80] have been experimented. The conductivity of these composites follows percolation law where after a certain threshold amount of conducting filler, the insulating polymer attains enhancements in conductivity by several orders. In such composites of CNT-polymer, the conductivity can change significantly under various stimulus for example, on interaction with various gas molecules, stress (pressure), temperature (say, by absorption of infra-red radiation). These behavior of composites can be utilized for various sensors for gas/vapour, [81-84] strain [30,85-88] and infrared radiations [35, 36, 89] besides for other applications such as anti-static [16] and EMI shielding [68,90].

CNT property	Theoretical/Experimental values
Dimensions	SWCNT 1-1.4nm MWCNT 5-20nm
Density	0.8 g/cm^3 for SWCNT; 1.8 g/cm^3 for MWCNT (theoretical)
Specific Surface Area (BET)	10–20 m^2/g
Mechanical	
Elastic Modulus	~1 TPa for SWCNT; ~0.3–1 TPa for MWCNT
Strength	50–500 GPa for SWCNT; 10–60 GPa for MWCNT
Electrical	
Electrical Conductivity	10^4-10^7 S/m (MWCNT)
Maximum Current density	10^{13}A/m^2
Electronic band gap	1.8ev to 0.18 ;Semiconducting SWCNT 0 ev; Metallic
Carrier mobility	10^5 cm^2/V-sec
Magnetic	
Magnetic susceptibility	22 x 10^6 emu/g (perpendicular with plane), 0.5 x10^6 emu/g (parallel with plane)
Thermal	
Thermal conductivity	3000 W/m/ K (theoretical)MWCNT 6000W/m/K (theoretical) SWCNT
Thermal expansion	Negligible (theoretical)
Thermal stability	>700 8C (in air); 2800 8C (in vacuum)
Phonon mean free path	~100 nm
Relaxation time	10^{-11}s

Table 1: Properties of carbon nanotubes

[Adopted from Schadler, L.S. (2004). Polymer-Based and Polymer-Filled Nanocomposites, in Nanocomposite Science and Technology (eds P. M. Ajayan, L. S. chadler and P. V.Braun), Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim].

Another class of conductive CNT-polymer composites, using conductive polymer matrix such as polyaniline (PANI) [91, 92] polypyrrole (PPy) [93,94] polythiophene (PTH) [95] and its derivative poly(3-hexylthiophene (P3HT) [96] polyacetylene (PA) [73] and poly-m-phenylene vinylene (PmPV) [97] have also been prepared. Both conducting polymers and carbon nanotubes possess conjugated π-systems and the nature of any electronic interaction is proposed to occur via π-π stacking. The synergy between multifunctional CNTs and the conducting polymer matrix has been judiciously exploited to create highly desirable smart polymer composite devices. Although not highlighted in this article, a number of applications have been cited for these composites including Schottky diodes [98], supercapacitors [99,100], actuators [101], electrodes for high-energy density batteries [102], nanowires for optoelectronic applications [103] and organic light emitting diodes (OLEDs) [104,105] and photovoltaic devices [106].

In sections to follow, some of above conductive CNT-polymer composite systems will be discussed; theory for their conductivity, properties, processing and the sensing mechanism based on which descriptions of various sensors specifically the gas/vapour sensors, strain sensors and infra-red sensors will be covered.

3. Theory of Conductive CNT-Polymer Composites

3.1. Conductivity in CNT- Polymer Composites

As said earlier, the conductive CNT-polymer composites have been fabricated using both insulating polymer matrix such as polyethylene (PE), polycarbonate (PC), polystyrene (PS), poly methylmethacrylate (PMMA), etc. and the conductive polymers such as polyacetylene (PA) polyanilene (PANI), polypyrrole (PPy), polytiophene (PTH), poly-m-phenylene vinylene (PmPV)etc. The theory of charge conduction in composites using insulating or conductive polymers may differ. In the following, both types of CNT-polymer composites have been considered to describe their conductivity theory.

3.1.1. Insulating Polymer Matrix

In general, dispersing the conductive materials into the nonconductive matrix can form conductive composites. The electrical conductivity σ of a composite is strongly dependent on the volume fraction, ϕ of the conductive phase. For CNT-polymer composite, the Fig 4, as found for PVC/MWCNT composite, shows typical conductivity dependence with the filler volume fraction, ϕ [107]. At low volume fractions, the conductivity remains very close to the conductivity of the pure matrix. At a certain critical volume fraction of the conducting filler, ϕ_c, the conductivity of the composite drastically increases by many orders of magnitude. This behavior for composite systems is well known as percolation phenomenon and can be well explained by percolation theory [9].

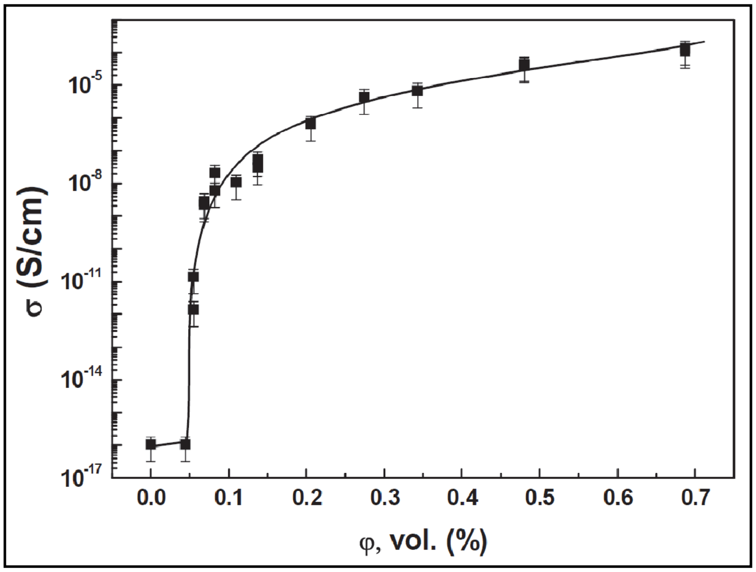

Figure 4: Dependence of electrical conductivity (σ) of PVC/MWCNT composite on the CNT filler content fraction, ϕ. At the percolation threshold, ϕ_c (~0.05 vol% in this study), the conductivity is enhanced by several orders. [Ack : Y. Mamunya [107]]

In CNT-polymer composite when a well ordered conductive network also called as *infinite* conductive network may be formed for an end to end charge transfer, the conductivity can be described by the following equation-

$$\sigma = \sigma_0(\emptyset - \emptyset_c)^t \quad , for\ \emptyset > \emptyset_c \qquad (3)$$

Where, σ_0 is a scaling factor and the exponent *t* reflects the dimensionality of the system. For the theoretical random 3D conductive systems t ≈ 2, for conductive CNT being 2D structures, t<2 are expected, typically the values 1.2-1.5 have been observed [108].

Higher aspect ratio of the filler particles leads to lower value of the percolation threshold [109]. In CNT-polymer composites using metallic MWCNT with high anisotropy of aspect ratio (length in µm /diameter in nm scale) in range of 100-10000, very small electrical threshold can be achieved [13,110]. The Fig 5 shows such an inverse dependence of percolation threshold with aspect ratio [109]. Also note in the figure that the curl (ratio λ) and waviness nature of CNTs is another significant factor that influences the percolation threshold and consequently also the conductivity as shown in Fig 6. For wavy nanotubes, a curl ratio has been defined as $\lambda = L_{CNT}/L_{effective}$, where $L_{effective}$ is an effective nanotube length which is the maximum distance between a pair of arbitrary points on the nanotube of length L_{CNT}. It is shown in the figure that the percolation threshold of wavy nanotubes increases with increasing curl ratio [111].

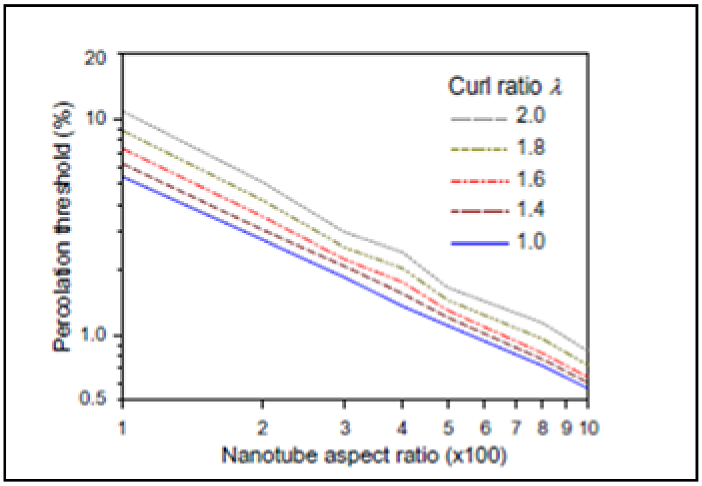

Figure 5: The influence of aspect ratio of CNTs of various curl ratio λ on percolation threshold (%) [Ack:[Li et.al[109]]

During various experiments it has been found that the predicted values of conductivity and percolation threshold differ significantly; Kilbride et al [108] in their studies on the electrical conductivity of carbon nanotube/polymer composite thin films, found the conductivity was significantly lower than expected.

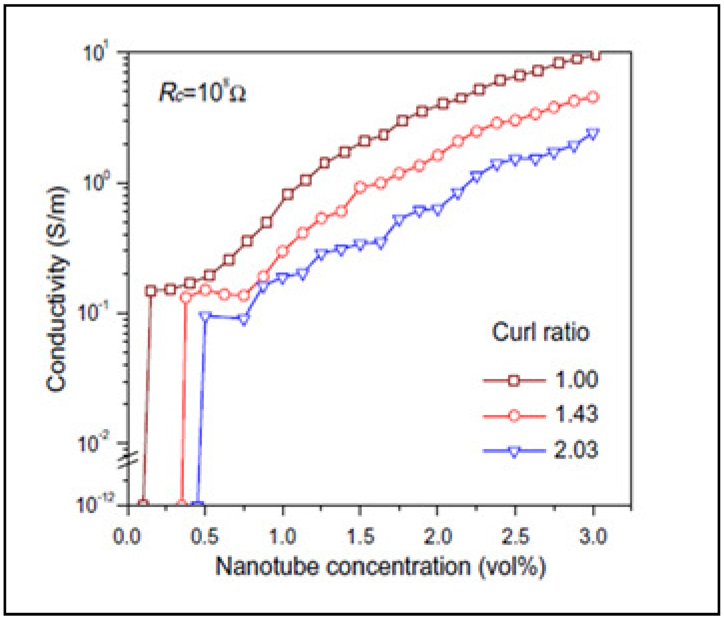

Figure 6: The influence of concentration of CNTs on conductivity and percolation threshold at various curl/waviness parameter λ [ACK:[Li et.al 111]]

One of the reasons attributed to this observation is said to be inter-nanotube contact resistance. When carbon nanotubes are dispersed in a matrix material electrically connected networks of CNTs are formed which may provide three continuous conductive pathways as shown in Fig 7 [112]. However, between the nanotubes and at the contact points of the junction nanotubes, a thin insulating layer may be formed. Now, a nanotube network has two sources of electrical resistance, i.e. the intrinsic resistance along the nanotube itself and the contact resistance at a nanotube junction due to a gap or cross-over between CNTs as shown in Fig 7.

When the thickness of the inter-nanotube matrix region is sufficiently small, the electrons can cross this region by the quantum mechanical tunneling effect for electrical conduction. A thick coating of polymer around nanotubes results in poor electrical conductivity and conduction in the composite films is dominated by the tunneling effect [113].

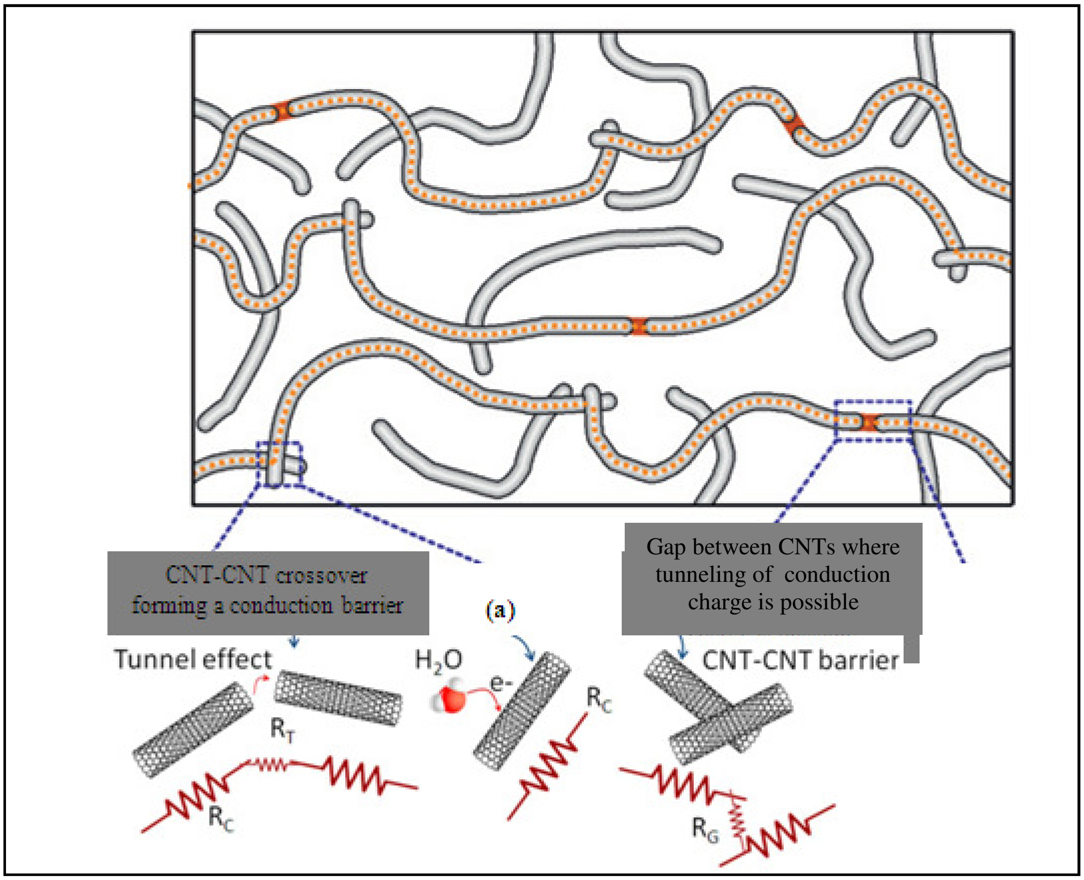

Figure 7: CNT network in (a) of CNT- polymer composite showing three types of conduction path ; path resistance shown in (b), due to tunneling at gap between CNTs (resistance R_T), due to intrinsic conductivity (resistance R_c) of CNT and due to a cross-over barrier of CNTs (resistance R_G) [Adopted from Kuronuma et.al [112]

Simulation results due to Li et.al. [113] shown in Fig 8 indicate that the contact resistance (noted as R_c in the figure) plays a dominant role in nanotube composite films in contrast to the dominant role of the intrinsic resistance of the nano-tube. The critical thickness of insulating polymer film for tunneling conductivity is found theoretically to be <1.8nm. It has also been found that the contact resistance at metallic–semiconducting junctions is three orders of magnitude higher than that at the metallic–metallic junctions [114]. Understandably, the metallic rather than the semiconducting CNTs may be preferred as fillers in conductive CNT-polymer composites.

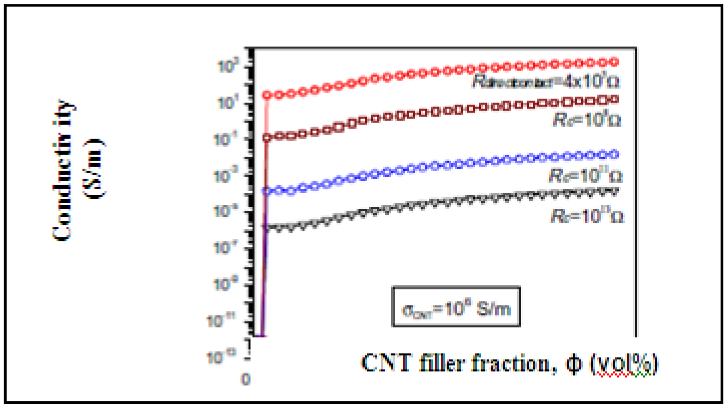

Figure 8: The effect of inter nanotube contact resistance, R_c and CNT filler fraction ϕ on the conductivity of composite film [Ack: Li et al [113]

3.2. Conductivity in CNT- Conductive Polymer Composites

Conducting polymers in their pristine (undoped) states are usually considered as semiconductors or insulators, having band gap energy excessively high for the thermal excitation of a significant number of charge carriers. Therefore, undoped conducting polymers, such as polyacetylene (PA) and polythiophene (PTH), show electrical conductivities of only 10^{-10}–10^{-8} S/ cm. Upon doping of conducting polymers, there is a dramatic increase in the electrical conductivity by several orders of magnitude up to values of approximately 10^{-1}

S/ cm. Such doped (conducting) polymers are conjugated polymers, which consist of alternating single and double bonds along its linear chains (sp^2 hybridized structure). The conductivity of conducting polymers relies on these double bonds, which are sensitive to physical or chemical interactions [115,116]. Similarly, CNTs also have sp^2 hybridized bonds over the structure. When mixed together, the two materials show a strong interfacial coupling via donor–acceptor binding and π-π interaction [42, 43]. Combining CNTs and conducting polymers in a composite has been found to affect their chemical and electronic structures. Beyond a simple physical combination of their properties, some synergistic effects and new features appear and can be developed into applications [48-50]. The capabilities of CNT-conducting polymer extend beyond a simple electrical conductor to functionalities such as charge storage in supercapacitors, photovoltaic devices and organic light emitting devices, to name some of these. Our concern here of course is restricted to their sensor applications involving their conductivity,

In conclusion of this section, it may be stated that the composite conductivity depends on the conductive network formation of CNT in the polymer matrix. The formation of these networks will depend on the type of polymer matrix, processing technology as well as the type of CNT materials used. It is significant to note that the conductivity and percolation are expected to be sensitive to the nature of dispersion/aggregation and degree of alignment of CNTs [38, 40]; thus an important role of processing may be expected for the composite electrical properties. In the section4 to follow the prominent processing methods for CNT-polymer composites are briefly covered. Later, some specific material systems are described with the obtained experimental results of percolation threshold and the conductivity for the experimented composite systems that may help understand the role of various factors on the conductivity of CNT-polymer composite and subsequently to their applications.

4. Processing of Conductive CNT/polymer nanocomposites

The CNTs, as-fabricated are normally mixtures of various chiralities, diameters and lengths, besides the presence of impurities, catalytic residuals, amorphous and spheroidal carbon and other defects. Further, due to their small size, CNTs have strong tendency to form aggregates due to their large surface area. Furthermore, CNTs are normally curled and twisted. Owing to these as grown characteristics, the CNTs embedded in a polymer only exhibit a fraction of their potential. Given that post fabrication purification can be achieved satisfactorily, aggregation of nano dimensioned CNTs is inevitable and their dispersion as fillers in polymer matrices challenging.

Several processing methods available for fabricating CNT/polymer composites based on either thermoplastic or thermosetting matrices have been described in past review articles [39, 40, 117]. Although inherently different processing routes have been attempted, in various studies, all address important issues such as exfoliation of CNT bundles and ropes, homogeneous dispersion of the individual tubes into the matrix, alignment and interfacial bonding that affect composite properties. Some of the most commonly adopted processing methods for preparing the CNT-polymer composites will be covered here namely the solution/ evaporation mixing, melt/ shear blending, in-situ polymerization.

For the as prepared CNTs to be useful for their fullest potential, they need to be purified and chemically functionalized for their good dispersion as well as efficient interaction for load transfer incase of reinforcement of mechanical properties or charge transfer for enhancement in electrical conductivity of polymer matrix. Prior to describing composite processing methods it is considered worthwhile to consider the purification and certain chemical modification processes of as prepared CNTs that are helpful for their dispersion in the polymers.

4.1. Purification/ Activation and Functionalization of CNTs

4.1.1 Purification/ Activation

Purification/ Activation and Fictionalization of CNTs are essentially pre- processing steps for preparing CNT-polymer composites. The CNTs have been purified by various approaches such as by burning in air or oxygen [118], oxidation by acid and oxidizing agent [119], hydrothermal treatment [120], alternating current (AC) electrophoresis [121]] with their respective advantages of simplicity or disadvantages of poor yield or even potentially damaging to intrinsic properties of CNTs. In general, during the oxidation processes by oxygen, air, concentrated sulfuric acid, nitric acid and 30% aqueous hydrogen peroxide that purify the CNTs, the active carboxylic acid and hydroxyl groups could be formed on the surface or open ends of CNTs [122]. In a way, such purifications provide activation or functionalization as well to CNTs. Though, this usually is at a cost of creating defect sites at the lateral ends of CNTs. There have been safer approaches also for purification such as, by Coleman et al. [123] and McCarty et al. [124] who used a functional organic polymer, poly(m-phenylene-co-2,5-dioctoxy-pphenylenevinylene) (PmPV), as a filtration system to purify CNTs. They found that the solution of PmPV is capable of suspending nanotubes indefinitely whilst the accompanying amorphous graphite separates out. Liu et al. [122] developed a purification method that consisted of refluxing in nitric acid and re-suspending CNTs in pH 10 water with a surfactant followed by filtration with a cross-flow filtration system. This may be an efficient method to purify CNTs owing to the combined advantages of the chemical and physical methods.

4.1.2. Functionalization

surface functionalization of CNTs is the most effective means in stabilizing the dispersion, since it can prevent re-aggregation of nanotubes besides, importantly, leading to coupling of CNT with polymeric matrix. Due to the nanoscale confinement in the system, the interaction via interfacial bonding is considered to play an essential role in the CNT-polymer composites [125-128]. The CNTs have been functionalized primarily by covalent and non-covalent bond methods. A schematic of both of these types functionalization of CNT are shown in Fig 9. Morphologically, the interfacial interaction sites on the CNT surface as shown in Fig9 are: (a) defect sites at the tube ends and side-walls; (b) covalent side-wall bindings; (c) non-covalent exohedral side-wall bindings (d) wrap around non-covalent bindings. The activation by oxidation described in previous section leading to grafting of –COOH and -OH functional groups is an example of covalent functionalization.

With these functional groups the dispersion of CNTs in polar solvents such as water can be made stable for several days. Non-covalent functionalized being non-invasive is more relevant in the present context of this article for conductive CNT-polymers. In non-covalent functionalization, the intrinsic electrical properties are likely to be preserved most compared to the covalent functionalization.

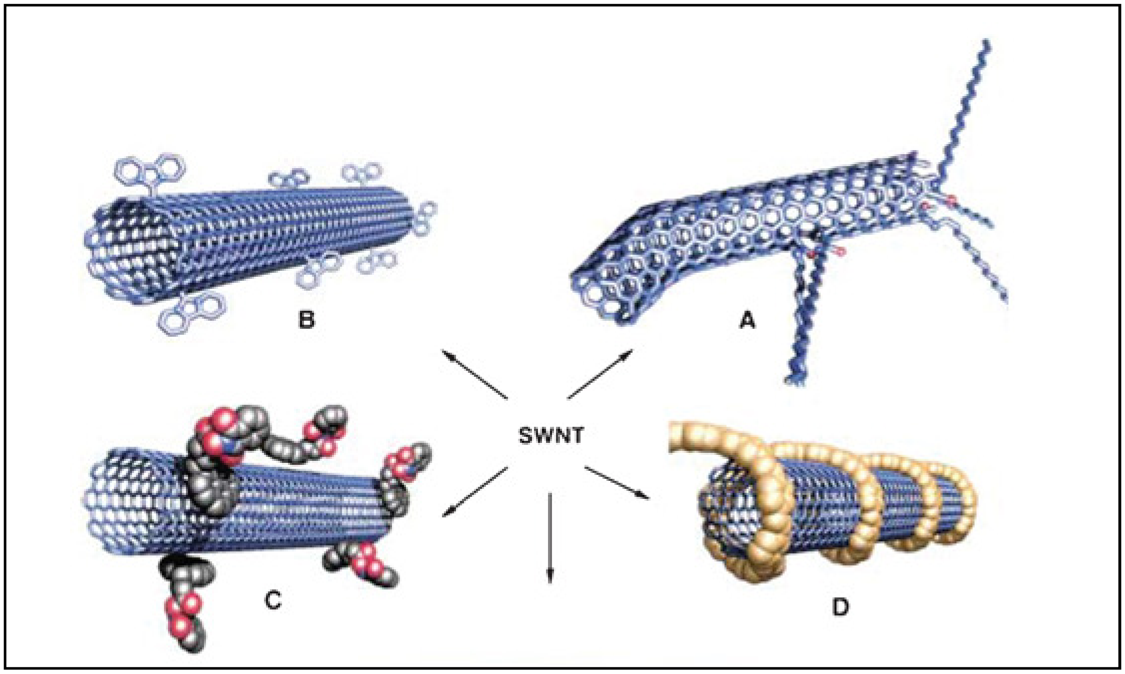

Figure 9: Covalent and non-covalent functionalization of SWCNT. [From. A. Hirsch, Angew. Chem. Int. Ed., 2002, 41, 1853–1859]

4.1.3. Non-Covalent Functionalization

The conjugated conductive polymer and long polar side chain polymer are capable of effectively dispersing SWNTs in terms of π-πstacking between their multi-aromatic moieties and the nanotube surface. The polymers such as poly(*m*-phenylene-*co*- 2,5-dioctoxy-*p*-phenylenevinylene) (PmPV) can be used to wrap around nanotube in organic solvents such as $CHCl_3$ [123] Polar side chain containing polymer, such as poly(vinyl pyrrolidone) [PVP] or poly(styrene sulfonate) [PSS] gave stable solutions of SWCNT/polymer complexes in water [129].

The presence of functional groups on the nanotubes can enhance their processibility [130] and their interaction with a polymeric matrix [131-135]. The functionalization however may also be undesirable particularly where preservation of the electrical properties is required, since it results in disruption of the conjugated π-π system [136] thereby compromising or diminishing the properties that are highly dependent on this conjugation.

In summary, while for good dispersion of CNTs in the polymer matrix, the pre processing steps of CNT purification and functionalization are necessary with a compromise of overall composite conductivity. Thus, an optimization of processing methods may be crucial in preparing conductive CNT-polymer composites.

4.2. Processing Methods of CNT-Polymer Composites

4.2.1. Solution Mixing

The most common method for preparing CNT/polymer nanocomposites involves mixing of CNT and polymer in a suitable solvent. A general approach for all solution processing methods include the dispersion of CNT powder in a liquid medium, mixing the CNT dispersion with a polymer solution and controlled evaporation of the solvent with or without vacuum conditions to recover a casted composite film or a mixed precipitate. For an efficient de-aggregation and dispersion of curled or bundled CNTs, the ultrasonication has been used most commonly, both the high-power sonication by a tip or of relatively low energy in a bath.

Qian et al. [137] made use of a simple solution-evaporation method assisted by high energy sonication to prepare polystyrene (PS)/MWCNT composite films, in which MWCNTs were dispersed homogeneously in the PS matrix. Similarly, Sandler et al. [138] dispersed CNTs in epoxy under high speed stirring (2000 rpm) for 1 h, and proved that intense stirring was an effective process to achieve dispersed CNTs uniformly in epoxy. The method is also very successful in case of PMMA and polyethylene [PE] nanocomposites [139].

In solvent blending, slow evaporation step often leads to CNT re-aggregation. To accelerate the evaporation of solvents, the method of spin casting the CNT/polymer suspension [140] or the method of drop-casting where the mixed suspension is dropped on a hot substrate [141] have been used. An alternative approach to overcome re-aggregation problem was proposed by Winey and co-workers

[142] for fabricating composites with individually dispersed CNTs, the so called coagulation. In this method, after the solution mixing step, a CNT/PMMA suspension was dripped into a large excess of non solvent such as water in order to induce instant precipitation of the polymer chains. The precipitating chains entrapped the carbon nanostructures and prevented them from bundling again. After filtration and drying in vacuum, nanocomposites were obtained with homogeneously distributed CNTs.

Another critical issue in the solution mixing method can be the use of high-power ultrasonication for a long period of time necessary for dispersion. This can shorten the nanotube length and so also reduce the aspect ratio. The large aspect ratios of intrinsic CNTs is one of their excellent properties, clearly, the smaller aspect ratios may limit the potential of CNTS for the composite properties. To minimize this problem, surfactants such as sodium dodecylbenzene sulfonate have been used to disperse higher loadings of nanotubes [143,144] However, the surfactants are retained in the composites affecting adversely the electrical conductivity of the composites. Industrial applications, melt processing is a preferred choice because of its low cost and scalability to large scale fabrication.

To improve CNT dispersion leading to efficient conductive network formation, the melt blend method also has been combined with other processing techniques. The melt blending can be combined with processes such extrusion [145], compression under pressure using a hydraulic press to yield composite films. Haggenmuller et al. [146] prepared PMMA/SWCNT composite films and fibers by melt processing. They found the composite films showed higher conductivity along the flow direction than perpendicular to it; Potschke et al. [71] have fabricated large batch sizes, 1 kg batches of MWCNTs/polycarbonate composites by melt extrusion and compression molding.

For good dispersion of CNTs, the critical issues in the method are an optimum viscosity vis-a-vis melt temperature and the amount of CNT loading in the polymer matrix; the temperature of melt reduces while CNT loading enhances it. The viscosity increases significantly with increasing CNT concentration, especially for CNT weight fractions above 2% [71] though appears to flatten beyond a certain loading. For example, as found by Xie et al. [147] where for the prepared polypropylene (PP)/CNT composite the viscosity flattens beyond 5 wt.%. In such a case, when higher viscosity is considered necessary for a good dispersion, adding a proper compatibilizer to polymer/CNT composites is useful and efficient to enhance the viscosity of the melt. Xie et al. [147] prepared polypropylene (PP)/CNTcomposites compatibilized with maleic anhydride grafted styrene–(ethylene-co-butylene)–styrene copolymer (MA-SEBS) by using a combination of ball milling and melt mixing. The observed improved electrical conductivity PP/CNT/MA-SEBS composites, by addition of MA-SEBS compatibilizer implied a good dispersion to enhance formation of a desired conductive percolation network of CNTs in the matrix. An approach developed by Jin et al [148] introduces polymer-coated MWCNT (rather than pristine MWCNT) into the polymer melt to promote compatibilization.

Shear force is another factor that needs to be optimized in the melt blending method. The high shear force that may be required to achieve CNT dispersion can also lead to CNT fragmentation and the concomitant deterioration of the composite properties. Generally, an optimization of process of CNT-polymer composite is found to be necessary [149].

Using melt blending/shear mixing method, some of the prominent studies of CNT-polymer composite systems where reasonably high electrical conductivity (>1S/m) have been obtained, are included in Tables 2 and 3.

Conductive Polymer matrix	**CNT type**	**Weight fraction (wt%)**	**Composite electrical conductivity (S/m)**	**Percolation Threshold (wt%)**	**Fabrication Process**	**Reference**
PANI	SWCNT	15	$2x10^3$	-	Solution mixing	Blanchet et.al[91]
P3HT	MWCNT, purified	36	7	10.62	Solution mixing	Yoshino et.al[96]
PmPV	CNT	<10	3	8.4		Coleman et.al[97]
PA	SWCNT	13	100	-	Compression/hot molding	Tchmutin et.al[73]
PTH	SWCNT	50	41	-	In situ chemical oxidative polymerization	Karim et.al[95]
PPy	CNT	25	$2.31x10^3$	-	In situ chemical oxidative polymerization	Zhang et.al[93]
PPy	MWCNT	3	$3.89x10^2$	-	In situ chemical oxidative polymerization	Wu et.al[94]
PANI	MWCNT	24.8	127	<24.8	In situ chemical oxidative polymerization	Long et.al[92]

Table 2: Electrical conductivity of CNT-Insulating Polymer composites:
The values of composite conductivity ≥ 1 S/m only as reported in literature have been noted. The list is indicative rather than exhaustive covering various composite preparation methods; solution-mixing, melt blending and in-situ polymerization

Insulating Polymer matrix	CNT type	weight fraction (wt%)	Composite electrical conductivity (S/m)	Percolation Threshold (wt%)	Fabrication Process	Reference
PS	SWCNT-PPE functionalized	7	6.89	0.045	Solution mixing	Ramasubramaniam et.al[66]
Epoxy	SWCNT	15	10	0.062	“	Li et.al[68]
PC	SWCNT-PPE functionalized	7	480	0.11	“	Ramasubramaniam et.al[66]
PC	SWCNT - P3HT-g-PCL compatibilizer	5	64	-	“	Kim et.al[70]
PMMA	**MWCNT**	**0.4**	**3000**	**0.003**	“	**Kim et.al[75]**
PU	**MWCNT**	**27**	**2000**	**0.009**	“	**Koerner et.al[25]**
PDMS	SWCNT	1.2	100	-	“	Worsley et.al[76]
HDPE	SWCNT	8	70	0.13	Solution mixing, Fast crystallization	Jeon et.al[79]
PP	SWCNT	10.4 (vol%)	8	5.85	Melt blending	Tchmutin et.al [73]
PP	MWCNT	10.7	4.6	1.10	“	Miˇcuˇsik et.al [74]
PC	MWCNT	15	20	1-2	“	Potschke[71]
PC	**MWCNT**	**15**	**1000**	**1-1.5**	“	**Potschke [72]**
PET	SWCNT	5	1	0.024	“	Hernandez et.al[78]
PU	MWCNT	3	30	-	In-situ polymerization	Yoo et.al[77]
PI	MWCNT	6.68	10	0.27	“	Jiang et.al[80]

Table 3: Electrical conductivity of CNT-(Conductive) Polymer composites:
The values of composite conductivity ≥ 1 S/m only as reported in literature have been noted. The list is indicative rather than exhaustive covering various composite preparation methods; solution-mixing, melt blending and in-situ polymerization

4.2.3. In-Situ Polymerization

This technique is particularly important for the preparation of insoluble and thermally unstable polymers which cannot be processed by solution or melt processing, discussed in preceding sections. The in- situ polymerization of monomers in the presence of CNT material produces polymer grafted CNTs, mixed with free polymer chains. Due to the small size of monomeric molecules, the homogeneity of the resulting composite is much higher than mixing CNTs and polymer chains in solution or melt mixing methods. In this sense, the method allows the preparation of composites with high CNT weight fraction. It has been extensively studied for the preparation of functional CNT-polymer composites including the conductive composites with very good miscibility with almost all polymer matrix.

The process enables to improve the dispersion and processability by grafting of polymer macromolecules onto the walls of CNTs. It is important to note that as polymerization progresses and the viscosity of the reaction medium increases, the extent of in-situ polymerization reactions might be limited.

The CNTs can be non-covalently or covalently bound to polymer matrix. Non-covalent binding between polymer and nanotube involves physical adsorption and wrapping of polymer molecules through van der Waals and л–л interactions. For example, some conjugated or conducting polymers are attached to their surfaces by in-situ polymerization; in-situ radical polymerization of pyrrole [150] and phenylacetylene [151] in the presence of CNT material. Spectroscopic characterization of the composites indicated no significant chemical attachment of polymer chains onto the graphitic sidewalls, but rather a helical wrapping mode [102]. The resulting composites exhibited enhanced dispersability in various organic solvents, whereas the electrical, magnetic, and thermal properties of the CNTs were modified by the conducting polymer chains [150].Tang and Xu [151] synthesized poly(phenylacetylene)-wrapped carbon nanotubes (PPA-CNTs), which were soluble in organic solvents, such as tetrahydrofuran, toluene, chloroform and 1,4-dioxane. Star et al. [152] synthesized poly(metaphenylenevinylene)-wrapped single-walled carbon nanotubes (PmPV-SWCNTs),and the UV–vis absorption spectra confirmed p–p interactions between SWCNT and fully conjugated PmPV backbone.

Cochet et al. [153] synthesized polyaniline (PANI)/MWCNT composites by in situ polymerization in the presence of MWCNTs. Their results reveal the site-selective interaction between the quinoid ring of PANI and MWCNTs, thus opening the way for charge transfer processes, and improving the electric properties of PANI/MWCNT composites. Xiao and Zhou [100]] deposited polypyrrole (PPY) or poly(3-methylthiophene) (PMeT) on the surfaces of the MWCNTs by in situ polymerization.

Using the in-situ polymerization process, some of the prominent studies of CNT-polymer composite systems where reasonably high electrical conductivity (>1S/m) have been obtained, are included in Tables 2 and3.

5. Conductive CNT-Polymer Composites and their Properties

Much effort has gone into the preparation of composites of various insulating as well as conductive polymers with functionalized or pure MWCNT/SWCNT as fillers, mostly by the methods described in the previous section 4, namely the solution mixing, melt mixing and in-situ–plymerization fabrication methods. The insulating matrices, such as epoxy, polypropylene, polystyrene, PMMA, poly(styrene-co-butylacrylate), PVA, PC, PC-PE blends, PDMS,PU etc. and conductive polymer matrices, such as PA,PANI,PPY,P3HT, PTH,PmPV etc., as referred in the introductory section have been used.

A wide range of values for electrical conductivity and percolation thresholds of CNT composites have been reported in literature during the last decade; indeed, the electrical conductivities of an order 10^{-4} to of 10^{3} S/m [Table1] have been reported . Similarly, the percolation threshold values of CNT in the composite as low as 0.003 wt% to high values of 10wt% or even higher [Table 2] have been reported. One investigation in particular, has yielded a SWCNT-epoxy composite exhibiting a percolation threshold of 0.0025 wt %, [13]. The electrical conductivity tailored to certain range of values by varying CNT filler content have also been reported e.g.in a range of 0.01–3480 S/m by varying the nanotube content from 0.11 to 15 wt% [66,154]. Here, some of selective results from literature, where reasonably high conductivities, >1S/m have been obtained, are presented in Table 2 for the case of insulating polymer matrices and in Table 3 for conductive polymer matrices. The tables serve a comparison between different types of material systems using various polymeric matrices, CNT type, method of composite fabrication with additional treatment of purification/functionalization, if any. The results indicate that there may not be any specific preference for a given CNT type for choice of a polymer matrix nor the method of fabrication. There may be many other factors that may control the conductivity threshold value for CNTs as well the conductivity of CNT-polymer.

The electrical conductivity of carbon nanotube-based composites depends on many factors. The intrinsic conductivity of individual carbon nanotubes plays an important role and is the upper bound for electrical conductivity 10^{4}–10^{7} S/m (Table 1). It has been found by Gojny et al. [155] that multi-walled carbon nanotubes offer the highest potential for enhancement of electrical conductivity, reason is said to be their better dispersability than single-walled nanotubes. Besides intrinsic conductivity, the discussions in section 3 already have shown significant role of CNT properties such as the aspect ratio, waviness/curl, that ultimately contribute to CNT-polymer composite conductivity. The conduction mechanisms discussed in this section show that the efficiency of conductive network formation in composites is the key in obtaining higher conductivities.

Besides the CNT properties various other factors have been cited in literature related to their processing methods and parameters that can benefit for the appropriate conductive networks. Studies show that the percolation threshold concentration and nanocomposite conductivity also depends on polymer type, fabrication/synthesis method, disentanglement of CNT agglomerates, uniform dispersion or spatial distribution of individual CNTs and degree of alignment [40,142,143]. Agglomeration is significant in chemical vapor deposition (CVD) grown multi-walled nanotubes because of nanoscale entanglement. The composite process steps such as, high speed mixing/ultrasonication and high shear–mixing in melt processing discussed in section4 help overcome this problem and achieve improved dispersion. The functionalization of nanotubes with the polymer matrix as in in-situ polymerization method of composite process also improves dispersion. The *in-situ* prepared composites possess enhanced polymer – nanotube interactions. In one study, both *in-situ* and *ex-situ* polymerization methods were employed, and it wasfound that the *in-situ* polymerization conditions caused fragmentation of the nanotubes [153] It has been established that improvements on the dispersion and alignment of CNTs in a polymer matrix could decrease the percolation threshold value [13,138, ,156]. Whereas, randomly oriented nanotubes embedded in polymer matrices have exhibited substantially lower electrical and thermal conductivities than expected [157,158].

However, for influence of nanotube alignment, there are some controversial results regarding the dependence of electrical conductivity in nanotubes–polymer composites. Haggenmueller et al. [146] and Choi et al. [156] reported enhanced electrical properties by aligning nanotubes in the matrix. The effect was explained by the more efficient percolation path for the parallel direction and/or the decease of disorder by alignment of nanotubes. But the results of Du et al. [159] indicated that the alignment of nanotubes in the polymer matrix significantly lowers the electrical conductivity compared with that of the unaligned composite with the same nanotube concentration. Du et al. [142] further concluded that the highest conductivity occurs when the nanotubes in the composite are slightly aligned rather than randomly isotropic. Studies also show that the type of CNT/functionalization and methods used for aligning the CNTs also play a role in influencing the conductivity of composites. Haggenmueller et al. [160] have attempted a combination of solvent casting and melt mixing methods to disperse single-walled CNTs in PMMA films to subsequently spin them to fibres; only the melt mixing method was found to be successful in forming continuous fibers and enhancement in conductivity. Similarly, Ma et al. [161] studied alignment and dispersion of functionalized nanotube composites of PMMA induced by electric field and obtained significant enhancement in dispersion quality and alignment stability for oxidized MWCNTs as compared to pristine MWCNTs. The enhancement achieved in this study however does not specify the role of various parameters that may have contributed to enhancements since the chemical functionalization of CNT by itself reduces intrinsic electrical conductivity of isolated CNTs because it disrupts the extended π-conjugation of nanotubes . Sulong et al. [162] showed that incorporation of acid and octadecylated functionalized MWCNT in epoxy matrix decreased the electrical conductivity of nanocomposites. Similar results have also been reported by Park et.al [163]. On the other hand, Polythiophene- functionalized MWNTs nanocomposites have been reported, in which poly(2-hydroxyethyl)thiophene is tethered to the nanotubes by ester linkages [164]. So prepared nanocomposite in this study, exhibited higher conductivity than that prepared by post-polymerization mixing; the authors however did not report any *in-situ* polymerization results. In above various studies, it is indicated that functionalization or modification of CNT along with the methods for fabrication are to be optimized to achieve minimum deterioration of electronic properties of CNTs.

6. CNT-Polymers Composite Based Sensors

CNTs represent a rare class of materials, which demonstrate a number of outstanding properties in a single material system, such as high aspect ratio, small diameter, light weight, high mechanical strength, high electrical and thermal conductivities, and tunable near-IR optical and optoelectronic properties. CNT–polymer composites, if engineered appropriately, display both passive and active material functions. The synergy between multifunctional CNTs and the polymer matrix has been judiciously exploited to create highly desirable smart polymer composite devices such as shape-memory and shape-changing actuators, strain sensors, gas sensors, fluid flow sensors and IR sensors and opto- electronic devices.

The CNT based sensors can find vast applications. To mention a few, in bio-medical applications, the strain /pressure sensors can be used in eye surgery, hospital beds, respiratory devices, patient monitors inhalers, and kidney dialysis machines. Intelligent pressure sensing systems play an important role in portable respiratory devices that consist of both diagnostic (spirometers, ergometers, and plethysmographs) and therapeutic equipments (ventilators, humidifiers, nebulizers, and oxygen therapy equipment). Strain sensors have a significant role in monitoring of structural health. e.g. of bridges, dams and aircraft bodies. In automobile applications, they can help acquire information about vehicle parameters such as pressure, vehicle altitudes, temperature, heat, humidity, speed and acceleration, exhaust gas, and engine knock and torque for improved and safer designs. An early and quick crash sensing to initiate safety air-bag is a most attractive application. Gas–sensors have applications in variety of vapour detection in chemically hazardous and polluting environments. In food industry, gas/chemical sensors can be employed in detection of pathogens, food freshness, monitoring during transportation, storage and on-shelf life. In agriculture and fisheries, the water vapour and temperature sensors can be employed for environment monitoring and control such as in green houses, and fishery ponds for biological oxygen demand and pH control. Gas sensors are also important for control and monitoring of hydrogen. In manufacturing, in petroleum conversion process , rocket engines and welding etc. CNT-based sensors can be potentially applied in defense and homeland security. They can be deployed in unmanned defense systems such as unmanned aerial vehicles. Besides, as electronic-nose for detection of dangerous and contraband chemicals a task presently is performed by sniffer dogs. The application list for CNT based sensors can go on and on, the above applications are just sundry examples. There however remain challenges, primarily to manufacture CNTs of high purity, and desired structural characteristics such as chirality, dimensions and type (SWCNT/MWCNT) at low cost. Secondly, for using as practical and cheap sensors by incorporation in polymeric matrices to provide repeatable and reliable performance, a more concerted research may be needed, particularly in dispersion and functionalization of CNTs.

In this section, as one of the objectives of this article, strain-sensors, gas/vapour sensors and infra-red (IR) sensors are briefly overviewed with primary focus on the review of research on their sensing mechanism. Illustrations exampled from available literature of some of related sensor-devices, also have been presented.

6.1. Srain-Sensors

Electronically, single-walled carbon nanotubes can be metallic, semiconducting, or small-gap semiconducting.. They also have very interesting electromechanical properties [[29]/2] and could be useful in applications for piezoresistive strain sensors such as strain gauges. Electromechanical coupling refers to Intrinsic coupling of electrical properties and mechanical deformation with a general behavior of increased bending/load leading to decreased conductance [165].

Tombler *et al.* [165] conducted an experimental study on the effects of local-probe manipulations on the electrical properties of the nanotubes. In this experiment of cantilever nanotube deflection under a load corresponding to 3% strain, the conductance of nanotube decreased by more than two order of magnitude. It was also observed that both the electrical conductance and the mechanical deformations are highly reversible. This property implies that electromechanical nanotubes-based sensors can be made reliable over many cycles use. The phenomenon was attributed to structural change of nanotubes translated to increase decrease of their electronic band gap [166]. While the metallic carbon nanotubes with decreased conductance were small-gap semiconducting carbon nanotubes, the semiconducting carbon nanotubes showed both an increase and a decrease in band gap. The metallic carbon nanotubes for chirality, $n = m$ have no change in conductance. Applications of above discovered piezoresistivity (electromechanical coupling) resulted to so called CNT buckypaper sensors. Buckypaper sensors, despite being highly sensitive to strain as said above compared to conventional strain sensor e.g., strain gauges ,however have very small fracture strain [167] and poor stability that limit their wide applications. Moreover, the theory of chirality based piezo-resistivity is not well understood and with discrepancies in theoretical and experimental results. However, the buckypaper strain sensors employed isotropic, randomly oriented CNT networks, and showed that the resistance increase linearly under tension and decreased linearly under compression. The isotropy allows multi-directional, multilocation strain measurements and is considered a great advantage over the conventional strain gages that are mostly unidirectional strain sensing devices.

Fiedler et al. [168] proposed first the concept of conductive modification with nanotubes as having potential for both strain and damage sensing. The conductive CNT-polymer composite have been effectively developed as practical 'pizo-resistive' type strain sensors. Here the piezoelectric effect is achieved by the conductive network of carbon nanotubes in the polymer matrices that can be altered from mechanical deformation; there is an increase in the resistivity. In section3 of this article, conductivity of CNT-polymer composite has been discussed where the conductivity networks of CNT play the primary function to control conductance. The intrinsic conductivity of CNT, inter-nanotube conductivity by tunneling mechanism controlled by a nano thin polymer matrix (resistivity in case of insulating polymer) are the contributors to conductivity of CNT conductive network. When under mechanical strain, contact arrangements and the tunneling distance between carbon nanotubes is altered, so also in the conductivity(or resistivity) and hence the so-called piezoresitivity in CNT-polymer composites. Using the above concept, a large number of studies for development of strain sensors of CNT-polymer composites have been conducted.

Li et al. [169] investigated the capability of using thin films of multiwalled carbon nanotubes as strain sensors. They used a uniaxial load/unload tensile test to estimate the sensing characteristics of the multiwalled carbon nanotube thin film. The results showed that a change in resistance was related linearly to the applied strain. Similar linear relationship between change in voltage with strain was also found by Vemuru et al.[170] using multiwalled carbon nanotubes.

Ramaratnam and Jalili [171] demonstrated the use of multiwalled carbon nanotube thin films as carbon nanotube/polycarbonate composite and obtained the fractional change ($\Delta R/Ro$; R_0 resistance without loading) of the nanocomposite when subjected to tensile strain with a gauge factor of about 3.5times that of a conventional strain gauge.

Yin and others studied [172] an epoxy-based nanocomposites strain sensor using two types of multiwalled carbon nanotubes of differing diameters and shape to determine the piezoelectric sensing mechanism. They detected that the piezoresitivity of the strain sensor was linear and antisymmetric in case of smaller diameter and curved shapes of MWCNT, probably due deformation induced piezoresistivity of multiwalled carbon nanotubes. On the other hand for the case of MWCNT of relatively straight and larger diameter, the main working mechanism of the strain sensoris is said to be the tunneling effect due to changes in distance among the multiwalled carbon nanotubes caused by applied strain.

Zhang et al. [173] presented a study on MWCNT-polycarbonate (PC) composites as multifunctional strain sensors, where a 5 wt% composite showed instantaneous electrical resistance response to linear and sinusoidal dynamic strain inputsand a sensitivity of ~3.5 times that of a typical strain gage.

Thostenson *et al.* [174] synthesized vinyl ester monomer from the epoxy resin to overcome processing challenges associated with volatility of the styrene monomer in vinyl ester resin. Calendering was employed for MWCNT dispersion in vinyl ester monomer and the subsequent processing of nanotube/vinyl ester composites. The high aspect ratios of the carbon nanotubes were preserved during processing, and an electrical percolation threshold below 0.1 wt.% carbon nanotubes in vinyl ester was observed.

A systematic study of the effect of SWCNTs on the enhanced piezoresistive sensitivity of polyimide nanocomposites from below to above percolation was reported by Kang *et al.* [175]. In this study, the maximum piezoresistive stress coefficient obtained just above the percolation threshold concentration (0.05 wt%) exceeded those of metallic piezoresistive materials by two orders of magnitude. Billoti *et al.* [176] presented a study on thermoplastic polyurethane (TPU) fibers containing MWCNTs, fabricated via an extrusion process, which demonstrated a tuneable level of electrical conductivity.

Bautista-Quijanoa *et al.* [177] reported the electrical and piezoresistive responses of thin polymer films made of polysulfone (PSF) modified with 0.05–1% w/w MWCNTs. Gage factors were measured for films with 0.2–1% CNT weight loadings. The films were then bonded to macroscopic aluminum specimens and evaluated as strain sensing elements during quasi-static and cycling tensile loading. Excellent piezoresistive capabilities were found for films with MWCNT loadings as low as 0.5% w/w.

Abraham et al. [178] reported the development and characterization of a CNT-PMMA anocomposite flexible strain sensor for wearable health monitoring applications. These strain sensors can be used to measure the respiration rhythm which is a vital signal required in health monitoring. A number of strain sensor prototypes with different CNT compositions have been fabricated and their characteristics for both static as well as dynamic strain have been measured.

Several other flexible strain sensors have been investigated by a number of researchers. Bliznyuk et al. [179], Loh et al. [180] and Dang et al. [181] applied CNT to various rubber & plastic composites for sensor applications.

6.2. Gas/Vapour Sensors

It is found that the electronic properties of CNTs are sensitive to the adsorptions of oxygen, nitrogen and carbon monoxide gases on the surface. P. G. Collins et al. [182] reported that the electrical resistance of CNTs was found to decrease on being exposed to oxidizing gases such as O_2 whereas, it increased when exposed to reducing gaseous species as N_2 as reported by Zhao et al. [183]. Collins et al., found that oxygen gas has dramatic effects on conductivity, thermoelectric power, and the local density of states of nanotube. N.D. Lang et al [184] reported that the conductance of a single oxygen doped (6, 6) nano-tube decreases by about 30% with respect to that of the perfect nano-tube. The gas sensing ability of nanotube arises from the changes in conductivity by the charge transfer mechanism due conjugated π-π^* bonds on CNT surface. This mechanism is similar to as in conductive conjugated polymers and has been described earlier in the present article in section 3.

Kong *et al.* [185] demonstrated chemical sensors based on individual SWNTs. They found that the electrical resistance of a semi-conducting SWNT changed dramatically upon exposure to gas molecules such as NO_2 or NH_3. There have been other gas sensing studies [186-188].

The use of the pristine *nonaligned* carbon nanotubes for gas sensing as reported in studies as above often involves tedious processes for integrating single carbon nanotubes into sensor devices, and the number of analytes to be determined is also hampered by the limited specific interactions with the unmodified nanotubes. The aligned/non-aligned, functionalized CNT embedded in a polymer matrix mitigating some of these difficulties have been researched and practical gas/vapour sensors have been invented. The sensing mechanism though in the composites is based now on the adsorption characteristics of gas/ vapour on CNT surface as well of the polymer matrix. The later can modify the conductive network of CNT and thus contributes to overall sensing mechanism. Some of significant studies of CNT-polymer based gas/vapour sensors will be highlighted here.

B. Philip etal [189] prepared CNT/polymethylmethacrylate (PMMA) nanocomposite thin films for the detection of organic vapors. Using pristine CNTs and surface-functionalized CNTs (f-CNTs), composite thin films were fabricated on a printed circuit board (PCB). CNT Surfaces were functionalized by oxidation using potassium permanganate with the help of a commercial phase transfer catalyst. Both pristine and oxidized CNTs were dispersed in PMMA polymer to fabricate a composite film. The composite was coated on a PCB with two parallel conducting lines (0.5 mm apart) on it for electrical connection. The composite film was deposited between the two conductors on the PCB by solution casting. The change in electrical resistance due to the presence of various volatile organic vapors namely dichloromethane, chloroform, acetone, methanol, ethyl acetate, toluene and hexane was evaluated. While, the change in resistance for the case of pristine CNT was less than an order for all type of tested vapours except the hexane. The resistance change for the functionalized CNTs was much higher, two to three orders of magnitude, particularly for the cases dichloromethane, chloroform and acetone, and lesser of 3-4 times for methanol, ethyl acetate while toluene showed no change. Resistance change Response of the CNT/PMMA composite to dichloromethane, chloroform and acetone vapors(a) using pristine CNT (b) using functionalized (oxidized)CNT as obtained in [189] is shown in Fig10.

The gas- sensing mechanism in the above study [189] is said to be due to the two mechanisms discussed earlier above; due to adsorption of vapors on CNT surfaces leading to intrinsic change.

in their conductivity, secondly due modification of conductive CNT network of the composite. Due to absorption of organic vapors, polymer matrix swells to consequently modify (increase) the volume and thus increases the distance between nanotubes, thereby increasing the contact resistance between the nanotubes. The extent of swelling, and hence the electrical response, depends on the solubility of the polymer in the solvent. The selectivity may be due to extent of polar nature of vapours that get adsorbed selectively on the CNTs. The sensing characteristics were found to be reversible though response times for various experimented vapors varied owing to varying solubility in polymer PMMA matrix. Similar polymer swelling effect was also noted by Yoon et al. [190] that the different gas concentrations diffuse into the polymer affecting the distance between carbon nanotubes to modify the conductive nanotube networks.

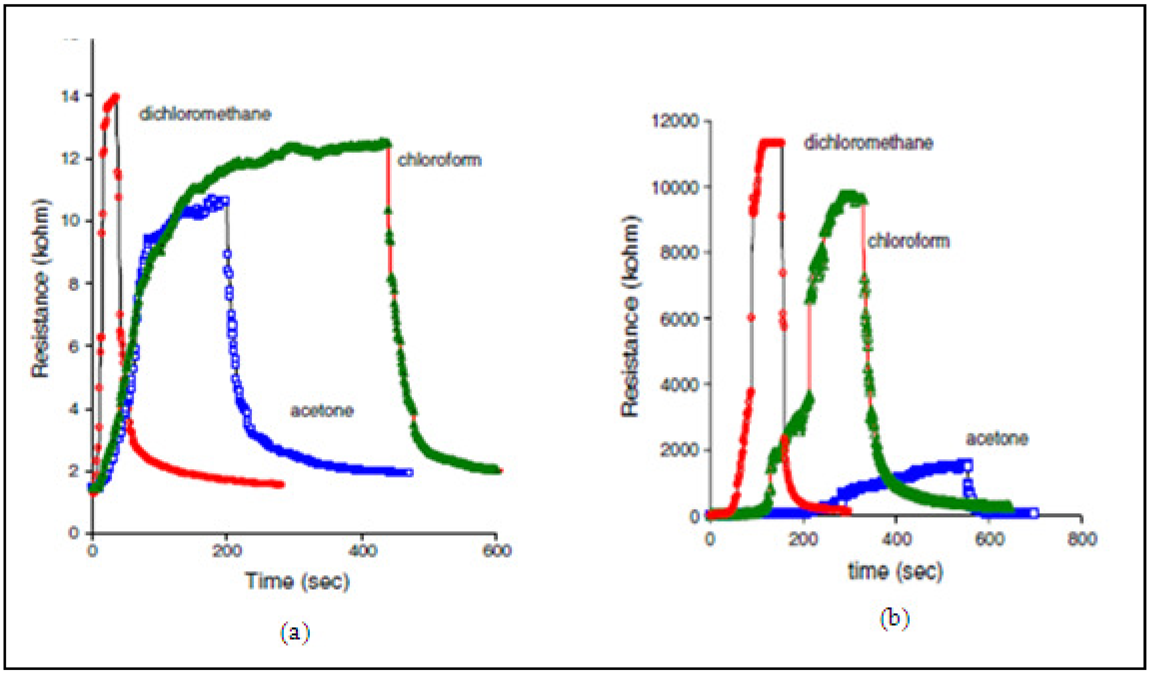

Figure 10: Resistance change Response of the CNT/PMMA composite to dichloromethane, chloroform and acetone vapors(a) using pristine CNT (b) using functionalized (oxidized)CNT (Acknowledment: Philip etal [189]

Kumar etal [191] prepared Conductive CNT/ bio-polymer, poly(lactic acid) (PLA) nanocomposites via solution mixing for dispersing multi-wall carbon nanotubes to develop volatile organic compounds sensors. The composite transducers were fabricated by spray layer by layer (sLbL) technique and the derived sensors chemo-resistive properties have been investigated by exposition to a set of organic vapours (chloroform, methanol, toluene and water). The used polymer matrix PLA is a widely known biomaterial that is biodegradable. The PLA/CNT suspension was sprayed onto electrodes consisting of a series of interdigitated metal lines separated by 30 µm (25 wt% Ag/75 wt%Pd).

The Chemo-resistive characteristics of the fabricated CNT/PLA transducers show high affinity towards chloroform; the resistance change on exposure reaches an equilibrium almost instantaneously whereas it is more progressive for methanol, toluene and water. Also, the resistance change for chloroform is highest with fractional change ($\Delta R/R_0$; R_0 being base resistance without vapour exposure) of 0.5 and progressively reducing for other experimented methanol, toluene and water, in that order. The reason of the selectivity is said to be due to the nature of interactions between analyte molecules and macromolecules from the matrix hosting the CNT conducting network. A larger carbon network disconnection resulting in a greater electrical response occurs when the penetrant molecules have higher affinity for the polymer coating carbon/carbon junctions. When the CNT filer percolation threshold is small for a given CNT/polymer composite a smaller solubility parameter results to sharper response since the disrupted conductive network is yet able to provide conduction by tunneling mechanism. The chloroform vapor was found to be most sensitive for CNT filler content (2%) closest to percolation threshold.

There have been other reports that have proposed the use of nanotube based composites as gas/vapor sensors [192, 193]. In these reports also, the changes in electrical resistivity of the composite are attributed to swelling of the polymer matrix and/or conductive modification due to the gas/vapour absorption. One of the reports [83] used the aligned MWCNT for preparing CNT/polymer composite demonstrated the sensing mechanism by change in conductivity both by the charge transfer mechanism (discussed in Section 3 of this article) and modification of conductive nanotube network. For preparing a composite free standing film, the aligned CNT array were partially covered with a polymer coating top-downalong their tube length by depositing a droplet of polymer solution of poly(vinyl acetate) PVA and, polyisoprene onto the nanotube film. A 130% increase in the resistance change, ΔR, for a composite film of PVAc and aligned carbon nanotubes after being exposed to tetrahydrofuran (THF) vapor for several minutes was observed.

6.3. Infra-red Sensors

Optoelectronic materials that are responsive at the wavelengths in the near-infrared (NIR) region (e.g., wavelength 800–2000 nm) are highly desirable for various demanding applications such as telecommunication, thermal imaging, remote sensing, thermal photovoltaics, and solar cells. Broadband infrared (IR) detectors enable night vision, imaging, and non-destructive testing of objects and thus can be used in medical diagnosis, industry, space, and defense applications. The organic opto-electronic materials for IR detection however are restricted to ≈800nm. On the other hand, semiconducting SWCNTs have strong and specific absorptions in the near-IR region owing to the first optical transition (S11), a known characteristic associated with optical absorption [194, 195]. Moreover, the inverse diameter dependence of such nanotubes with the S11 optical transition energy enables the wavelength-tuning of the near-IR absorptions of SWNTs.

Levitsky and Euler [196] demonstrated for the first time that the arc-produced SWCNT (SWCNTarc) film is capable of generating a very weak photocurrent upon continuous-wave IR illumination (12 mW mm22) in the air at room temperature, though the current increase upon IR illumination was noted to be only approximately 0.2%.

Later, Chen and co-workers [34-36] have discovered that the IR photoresponse in the electrical conductivity of SWCNTs is dramatically enhanced by embedding SWNTs in an insulating polymer matrix such as polycarbonate (PC) in the air at room temperature. This led to development of IR sensors based on CNT-polymer composites. The IR sensors are thus a class of smart sensors where the synergy between CNTs and the polymer matrix has been judiciously exploited.

Schematic of experimental IR sensor due to, Chen and co-workers referred above is shown in Fig11 [reproduced from J Chen ;as acknowledged in figure]. It may be seen in the figure (b), The SWCNT/polycarbonate composite shows strong absorption due to S_{11} optical transition.

The reported results of the study for the 5 wt% SWCNT (produced by CoMoCAT process) and PC polymer nanocomposite demonstrate a very strong conductivity change of 23.45% upon the IR irradiation (7 mW mm^{-2}) in the air at room temperature [Fig.11(c)] which is 5.5 times compared to another experimented composite film using CNT filler with much lesser semiconducting nanotubes, the SWCNT (produced by HiPco process) and PC composite film and 27 times of that observed in the pure SWCNTCoMoCAT film in the air at room temperature (12 mW mm^2). In addition, the 5 wt% SWCNTCoMoCAT–PC composite film shows a detectable IR photoresponse at a light intensity as low as 23.4 mW mm^{-2}[35, 36].

Despite demonstration of CNT based IR sensors as above [35, 36] and other studies [197, 198] where sensing mechanism is said to be based on photo signal generation by free charge carriers, the origin of the large photoresponse has been debated. Haddon and co-workers [199] reported that the IR photoresponse in the electrical conductivity of a SWNTarc film is dramatically enhanced when the nanotube film is suspended in vacuum at low temperature. For example, at 50 K, the SWCNT film suspended in vacuum shows a resistance drop of 0.7% under an extremely low incident power of 0.12 μW of IR radiation. Their experimental data suggested that the IR photoresponse of the SWNTarc film arises mainly from the thermal effect.

The bolometric effect has been clearly pointed out on suspended (free- standing) CNT mat by these authors [199]. Apparently, this observation is akin to well known operating principle of IR absorbers used for thermal detectors that is, the absorption of incident radiation as heat causes a change in the resistivity or frequency. Based on the above heat induced change of resistivity of CNTs, the thermal sensing mechanism, the so called bolometric effect has also been proposed to explain the IR sensing in CNT films [200-202].

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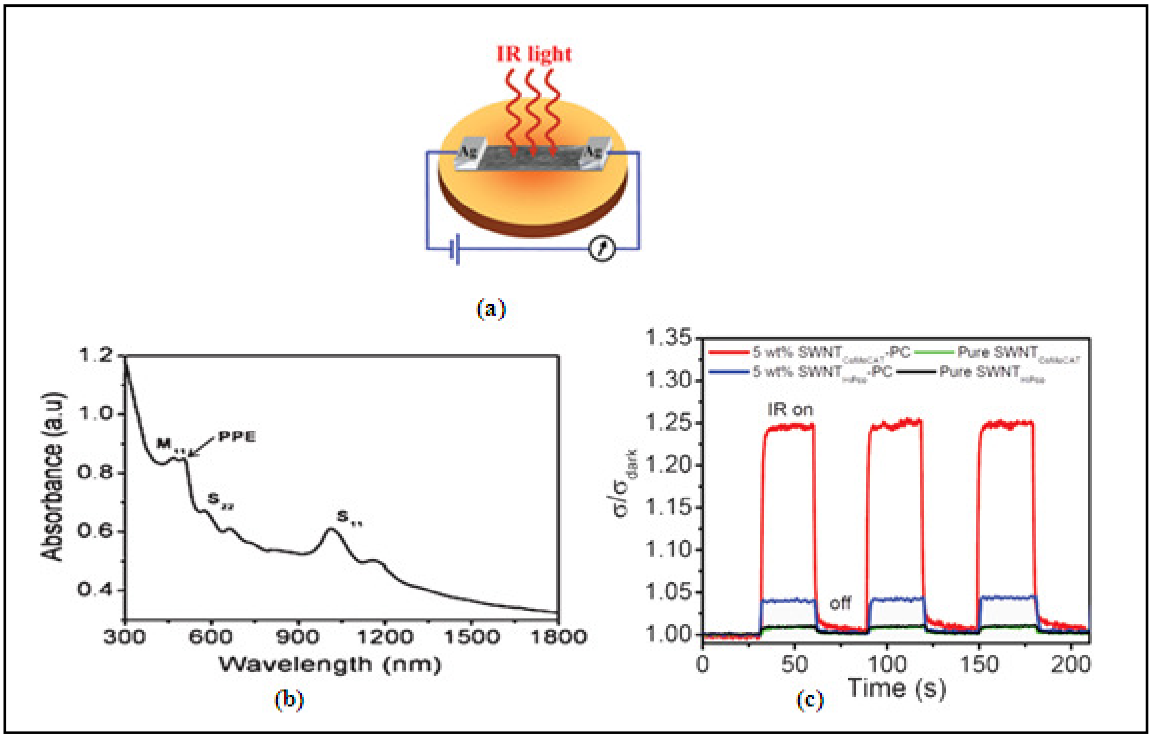

Figure11(a) Schematic representation of CNT–polymer composite IR sensor (b) UV-Vis-near-IR spectra of 5 wt% SWNTCoMoCAT–PC composite thin films. M11, S11, and S22 represent optical transitions in metallic and semiconducting SWNTs, respectively (c) Relative conductivity σ/σ_{dark} responses of SWNT–PC nanocomposites; σ is conductivity when IR illuminated of IR power intensity: 7 mW mm^{-2}

[From; Jian Chen, Actuators and Infrared Sensors Based on Carbon Nanotube–Polymer Composites, In Book RSC Nanoscience & Nanotechnology No. 27Carbon Nanotube–Polymer Composites, Chapter2,p243 Fig2.17 Ed. Dimitrios Tasis}

The above controversy apparently appears to have been resolved in earlier referred studies [35, 36] by Chen etal on SWCNT/polycarbonate composites where, both of the above said sensing mechanisms that is due to thermal effect and photo induced charge generation may occur individually and jointly due to IR absorption. It is stated in the study [35], while the thermal effect predominates in the IR photoresponse of the pure SWCNT (HiPco) film, the photo effect predominates in the IR photoresponse of the 5 wt % SWCNT(HiPco)-poly carbonate (PC) nanocomposite. The thermally insulating PC matrix may promote the photo induced exciton dissociation to free charge carriers via two possible mechanisms. First, the local temperature of SWCNTs embedded in the thermally insulating PC matrix can be significantly increased upon IR illumination, providing sufficient thermal energy required for the exciton dissociation. Secondly, in SWCNT-PC composites, the SWNTs are coated with an electrically insulating PC thin layer, which acts as a potential barrier to inter-nanotube hopping. The enhanced local electric field at the SWCNT-PC interface could help the exciton dissociation. Further, it has been clarified in [36], the content of semiconducting SWCNT in the as produced CNTs has a role for defining sensing mechanisms. The SWCNT produced from the HiPco method that are said to contain lesser semiconducting type of SWCNT compared to produced by the CoMoCAT method. Composite IR sensors based on CoMoCAT-produced SWNTs (SWCNTs CoMoCAT), which have more semiconducting tubes, significantly outperform those based on SWCNTsHiPco.

It is implied from above that in a SWCNT/*thermally* insulating polymer, the above two sensing mechanisms may both occur individually and jointly. The magnitude and predominance of photo generation or thermal effect will depend on the specific CNT composite composition i.e, on the extent of semiconducting nature of SWCNT and the thermal conductivity of insulating polymer and also on other factors such as ability to convect or dissipate heat when deposited on hard substrate or free standing CNT film. It is also an implied inference from this observation that the MWCNT, that do not have IR absorption, may be expected to operate by the thermal sensing mechanism when employed for IR sensing.

Gohier etal [89] presented realization of an all printed MWCNTs based IR sensor on polyimide substrate as flexible free standing film. On flexible polyimide film of thickness 120μm with silver ink printed electrodes, MWCNTs dispersed in a solvent were deposited to fabricate the IR sensor. The device exhibited a negative temperature coefficient of resistance (TCR) of ~0.19% K^{-1}, fractional change in resistance upto more than 30% varying with the thermal power density from 0-0.5mW mm^{-2} and a response time of around ~1 s. It was also observed in the study that the resistance significantly drops with decreasing humidity amount, underling the water molecule desorption.

7. Conclusions

The review of literature reveals that conductive CNT/polymer composites for various sensing functions have been developed successfully. From the reported achieved values of conductivities as discussed in the present article, there exists a large potential for enhanced conductivity for CNT/polymer composites by controlling the properties of CNTs as well the optimization of fabrication method for a given polymer matrix. The dispersion of CNTs in polymer matrix for forming a conductive percolating network is crucial to sensor performance. The conductance of CNT/polymers is also highly sensitive to changes in moisture and temperature. In general, significant challenges exist in the development of sensors based upon nanocomposites for example, the efficient growth of macroscopic-length carbon nanotubes, controlled growth of nanotubes on desired substances, durability of nanotube-based sensors, effective dispersion of nanotubes in polymer matrices and the control of their alignment, and the scale-up in fabrication.

For stain sensors, the interfacial bonding characteristics between CNT and polymer may be significant. Although fundamental studies on processing-structure-property relationship in CNT nanocomposites need to be continued, allied efforts will need to be devoted to large-area strain mapping, cumulative stress/strain tracking, damage detection and life prediction algorithms, and data acquisition to utilize strain sensing for structural health monitoring.

Gas sensors based on electrical conductance changes have certain limitations, such as poor diffusion kinetics, inability to identify gases with low adsorption energies, and low capability to distinguish between gases or gas mixtures and gas-flow velocity.

The IR sensors require mostly semiconducting SWCNTs as fillers that means well developed and inexpensive separation methods for CNT type separation.

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