Carbon nanotube-reinforced elastomeric nanocomposites: a review

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This review is focused on carbon nanotube (CNT)-elastomeric polymer nanocomposites, which have attracted industrial and academic interest over the years due to their enhanced properties. Major factors notably CNT type, surface modification, dispersion of CNT, and processing techniques that affect the physical properties of CNT-elastomeric polymer nanocomposites are reviewed, and several key physical properties, including tensile, electrical, and thermal properties, were also included in this review. Some of the key challenges that undermine the effectiveness of CNTs and their composites with elastomeric polymers, and the potential applications of CNT-elastomeric composites are also captured.

Keywords: carbon nanotubes; elastomeric polymers; nanocomposites; surface modification; physical properties

1. Introduction

The carbon nanotubes (CNTs) can be formed by rolling of graphite sheets into a tubular structure [1,2]. They can be classified into different forms depending on the number of layers. They include single-walled carbon nanotubes (SWCNTs), double-walled carbon nanotubes (DWCNTs), and multiwalled carbon nanotubes (MWCNTs) (Figure 1). As can be seen in Figure 2, the basic characteristics of CNTs can be determined by the way of rolling of graphite sheet leading to a specific chirality defined by the chiral (roll-up) vector, \( \mathbf{C}_k \), and chiral angle, \( \theta \), as can be described by the Equation [3–7]:

\[
\mathbf{C}_k = n\mathbf{a}_1 + m\mathbf{a}_2
\]

where the integers \( n \) and \( m \) are the number of steps along the zigzag carbon bonds of the hexagonal lattice and \( \mathbf{a}_1 \) and \( \mathbf{a}_2 \) are unit vectors. Two limiting cases of the geometrical structure are possible. The zigzag (\( \theta = 0^\circ \)) and armchair (\( \theta = 30^\circ \)) structure is shown in Figure 3. Generally, the property of CNTs depends on their atomic arrangement, diameter and length, and surface morphology. The chirality of CNTs strongly affects the mechanical and electric conductivity [1,4,5,7,8].

Since the first detailed characterization of CNTs in 1991 by Iijima [9], they have received much attention because of their unique properties. The CNTs possess high

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Figure 1. Structure of (a) graphite sheet, (b) single-wall (SWCNT), (c) double-wall (DWCNT), and (d) multiwall carbon nanotube (MWCNT) (available from online: http://jnm.snmjournals.org/content/48/7/1039/F1.expansion.html).

Figure 2. Schematic diagram showing how a hexagonal sheet of graphite is ‘rolled’ to form a carbon nanotube. Reprinted from reference [4].

Figure 3. Illustrations of the atomic structure of (a) an armchair and (b) a ziz-zag nanotube. Reprinted from reference [4].
flexibility, low mass density, and large aspect ratio (typically: 100 ~ 1000) in addition to very high electrical and thermal conductivities. Some nanotubes are stronger than steel (about 10 ~ 100 times higher at a fraction of the weight), lighter than aluminum, and more conductive than copper [10,11]. For example, theoretical and experimental results on individual SWCNTs show extremely high tensile modulus (640 GPa to 1 TPa) and tensile strength 6 (150 ~ 180 GPa) [12]. The SWCNTs exhibit large phonon mean free path lengths that result in high thermal conductivity (theoretically > 6000 Wm$^{-1}$K$^{-1}$).

The superior mechanical and electrical properties of CNTs offer an exciting potential for new class of polymer composite materials to replace the traditional fillers. A number of polymers such as epoxy [13,14], polyvinylacetate (PVA) [15], polyvinylchloride (PVC) [16,17], polyethylene (PE) [18], polyamide (PA) [19], and many other polymers have been employed as matrices to prepare CNT-polymer nanocomposites. Figure 4 shows a trend of the CNT-based research activity in terms of scientific publication and patents over the years since 1992. An exponential growth of research activity indicates that the topic has become one of the hot issues recently. Figure 5 shows a similar trend of CNT-based research activity (research publication only) focusing on the elastomeric polymer matrices including rubbers. The data in Figure 5 were collected in a recent literature pool on CNT-elastomer-based compounds [20–118]. The gradual increase in the number of research publication also suggests that a great attention is now extended to the reinforcing of rubber by CNTs, even though the total number of publications is relatively lower.

Elastomeric polymers and rubbers have long been widely used for various applications including tires, seals, shock-absorbing mounts and gaskets for energy, structural, electronic, and electrical equipment [33–41]. No other polymers without elasticity can replace the unique function of the rubbery polymers. The employment of CNTs into rubbery matrices can provide further improvement of durability, strength, light weight, as well as design and processing flexibility compared with the conventional fillers like carbon blacks (CBs). Since the research works on CNT-reinforced rubber nanocomposites have been published by many researchers [41–47], there is a clear need to collect and analyze the recent technologies developed so far. The current review paper presents the streamlined technologies of various CNT-elastomer nanocomposites and their hybrid system by

![Figure 4](https://sites.google.com/site/cntcomposites/Home).
focusing on modification of CNTs, mixing, morphology and state of dispersion of CNTs, mechanical, electrical, and thermal properties.

2. Modification of CNT

A crucial issue in fabricating CNT-polymer nanocomposites is the dispersion of individual CNT into the host polymer matrix without losing the effective interactions at the CNT–matrix interface. Individual dispersion of CNT is strongly affected by van der Waals forces of attraction between adjacent tubes and high aspect ratio, which gives rise to the formation of aggregates [3, 10, 12, 48]. Several methods have been reported for the fabrication of CNT-elastomer composites, focusing on the successful dispersion of CNT filler in the host matrix and their effective interfacial interactions with the matrix. One of the most promising means of enhancing the dispersion of CNTs and their interfacial bonding with polymer matrix is the modification/function alization of the CNTs [49, 50, 51]. The modification can be categorized into covalent and non-covalent. We focus here on a brief explanation and their relevance to processing of CNT-elastomeric nanocomposites.

Covalent functionalization is one popular method to attain modified CNTs; it deals with chemical bonding (grafting) of polymer chains to CNTs, where strong chemical bonds between nanotubes and polymers are established. There are two ways of grafting of CNTs. The ‘grafting to’ approach involves the synthesis of a polymer with a specific molecular weight terminated with reactive groups or radical precursors. In a subsequent reaction, the polymer chain is attached to the surface of nanotubes by addition reactions. In comparison, the ‘grafting from’ approach involves growing polymers from CNT surfaces via in-situ polymerization of monomers initiated by chemical species immobilized on the CNT sidewalls and CNT edges [3, 48]. A notable drawback of covalent modification is the disruption of the extended π conjugation in CNTs, which can strongly affect negatively the electrical property [3]. Figure 6 presents the various approaches used to chemically modify CNTs.
The next categorized method is the non-covalent modification, which can modify the interfacial properties of CNTs [3,53]. It is primarily concerned with the physical adsorption and/or wrapping of polymers to the surface of the CNTs. The graphitic sidewalls of CNTs provide the possibility of π-π stacking interactions with conjugated polymers, as well as organic polymers containing hetero-atoms with free electron pairs [3,54]. The use of surfactants falls in the non-covalent method and it is widely accepted. Surfactants consist of hydrophilic polar head and hydrophobic nonpolar tail. They can interact with the hydrophobic surface of CNTs to provide them a hydrophilic character, which makes the CNTs soluble in several solvents including water [55–58]. Unlike the chemical modification, the non-covalent method does not destroy the conjugated system of CNTs, leading to a less effect on the electrical performance. However, the load transferring capability might be lower due to weaker interactions at the interfaces [18,19,53].

Islam and co-workers [59] explored the dispersing power of a range of surfactants: sodium dodecylbenzenesulfonate (NaDDBS: C\textsubscript{12}H\textsubscript{25}C\textsubscript{6}H\textsubscript{4}SO\textsubscript{3}Na), sodium octylbenzene sulfonate (NaOBS: C\textsubscript{8}H\textsubscript{17}C\textsubscript{6}H\textsubscript{4}SO\textsubscript{3}Na), sodium butylbenzene sulfonate (NaBBS: C\textsubscript{4}H\textsubscript{9}C\textsubscript{6}H\textsubscript{4}SO\textsubscript{3}Na), sodium benzoate (C\textsubscript{6}H\textsubscript{5}CO\textsubscript{2}Na), sodium dodecyl sulfate (NaDS: CH\textsubscript{3}(CH\textsubscript{2})\textsubscript{11}-OSO\textsubscript{3}Na), Triton X-100 (TX100: C\textsubscript{8}H\textsubscript{17}C\textsubscript{6}H\textsubscript{4}(OCH\textsubscript{2}CH\textsubscript{2})\textsubscript{n}-OH: \textit{n} \equiv 10), dodecyltrimethylammonium bromide (DTAB: CH\textsubscript{3}(CH\textsubscript{2})\textsubscript{11} N(CH\textsubscript{3})\textsubscript{3}Br), dextrin, and poly(styrene)-poly(ethylene oxide) (PS-PEO) diblock copolymer. It was shown that the stabilization of CNTs depends on the surfactant molecules that lie on
CNTs surface parallel to the cylindrical axis. They reported that the alkyl chain groups of a surfactant molecule adsorb flat along the length of the tube instead of the diameter. The NaDDBS and Triton X-100 (TX100) were dispersed CNTs better than SDS due to the role of benzene rings. NaDDBS was reported to be dispersed better than TX100 because of its head group and slightly longer alkyl chain. They argued that the spacing between the benzene rings on the surfactants and the CNTs surface was large enough to accommodate the $\text{SO}_3^-$ charged groups [59]. Figure 7 is a schematic representation of how surfactants may be adsorbed onto the nanotube surface.

Table 1 summarizes the modification class of CNTs and their composites with rubber matrices. The majority research works on CNT-rubber nanocomposites have been focused on the covalent modification of CNT, typically by acid treatment of CNT for processing CNT-elastomeric composites. Other covalent modification includes the amino/silane treatment, and hydroxyl and grafting modification method. Among the non-covalent modification, the treatment by ionic surfactants was widely used by researchers. However, the choice of modification method may strongly depend on the application purpose of the resulting nanocomposites.

![Figure 7. A schematic representation of adsorption behavior of surfactants onto the surface of CNTs. Reprinted from reference [59].](image)

| Type of modification | Modifier | Rubber–CNT composite |
|----------------------|----------|----------------------|
| Covalent Acid treatment | NR/SBR-MWCNT [21], NR-MWCNT [60,61], NR-SWCNT [36,62], TPE-MWCNT [27], IIR-MWCNT [38], NBR/SBR-MWCNT [63], PDMS-MWCNT [47,64], CIIR-MWCNT [44], Soft epoxy [65], EPDM-MWCNT [66], SBR-CNT [67], TPU-MWCNT [68] |
| Hydroxyl (-OH) | SBR/BR-MWCNT [69] |
| Grafting | TPU-MWCNT [70] |
| Amino/silane | NR-MWCNT [33], PDMS-MWCNT [71], BADGE/ATBN-MWCNT [72], TPU-MWCNT [73] |
| Non-covalent Ionic surfactant | CR-MWCNT [34,41,74,75,76], PDMS-SWCNT [77,78], NR-MWCNT [79,80], PDMS-MWCNT [81], SBR/BR [82], NR |
| Non-ionic | NR/SBR-MWCNT [83], SBR/BR-MWCNT [69] |
| Catalyst | SEBS-SWCNT [39] |
3. Mixing of CNT with elastomeric polymer

One of the issues in the CNT-based elastomeric polymer nanocomposites is how to achieve a homogenous good dispersion of CNTs in the polymer matrix to maximize the enhancement in various performances of the resulting composites. The major considering factors would be the CNT type and concentration, modification of CNTs, type of polymer matrix, and mixing techniques used [3, 50, 51]. Although, there are several mixing techniques available, most of the CNT-filled elastomeric composites mixing is widely done by using the conventional techniques such as solution mixing, melt mixing, and in-situ polymerization [15, 16].

Solution mixing is one of the approaches for processing polymer nanocomposites. It involves the use of a dissolvable polymer in a solvent such as tetrahydrofuran (THF), dimethyl formamide (DMF), toluene or acetone. Then the nanofillers are also dispersed in one of these dispersible solvents by the sonication or any kind of mechanical agitation approach. The two solutions are then homogenously mixed together by mechanical mixing, magnetic agitation, or high-energy sonication. Subsequently, the CNT-polymer composites can be obtained by either vaporizing the solvent or coagulation by adding a poor solvent [25, 77, 78]. This method is generally considered when a better dispersion and distribution of film-type composites are needed [10]. One limitation of this method is that the polymer should be dissolved in a solvent for further processing. Badaire group [84] established that a high-power ultrasonication for a long period of time shortens the nanotube length (shorter aspect ratio), which is detrimental to the composite properties. The general guideline for the optimum sonication conditions (time, power) are not systematically established and will depend on nanotube concentration and original shape of CNTs [10]. Many CNT-rubber composites with different kinds of elastomeric matrices and CNTs have been reported via this method [25, 83, 85, 86]. Recently, Bokobza [87] enhanced the electrical and mechanical properties of rubber based on styrene–butadiene rubber (SBR), natural rubber (NR), and ethylene–propylene–diene monomer (EPDM) with MWCNTs. In terms of mechanical properties (ultimate tensile strength), SBR obtained 1.08 MPa, NR ~ 5.88 MPa, and EPDM ~ 10.7 MPa, respectively, at 0.5 phr of filler loading. This is a clear indication that the solution mixing is sensitive to the type of matrix involved. Furthermore, the processing conditions had a strong effect on the electrical properties, which are also very sensitive to nanotube dispersion within the elastomeric matrix. A new mixing technique which can be categorized under solvent mixing approach was recently adopted by Shin et al [88] to prepare a soft, flexible, highly stretchable, and conductive composites of MWCNT and PU (poly[4,40- methylene-bis (phenyl isocyanate)-alt-1,4 butanediol/poly(butylene adipate)]). In this study, aligned MWCNT (MWCNT forest, diameter: 10 nm) were grown on iron-catalyst-coated Si wafers using a conventional chemical vapor deposition (CVD) method. Initially, the MWCNT forests were infiltrated with a PU solution in N,N-dimethyl formamide (DMF) using a simple drop-casting procedure. Later, the solvent was evaporated and a composite of MWCNT forest/PU which could be peeled off the underlying Si wafer was obtained. The prepared film was composed of a black and conductive side facing the substrate (forest side) and the other whitish and insulating side (PU side). The MWCNT/PU material could be stretched up to about 1400% relative to the pure PU polymer (760% strain). The elongation of the material was also higher than the highly stretchable sheath-core conducting fibers (strain up to 1320%) created by wrapping CNT sheets oriented in the fiber direction on stretched rubber fiber cores prepared by Liu et al [89] and the stretchable CNT material (280% strain) for human motion detection [90]. Moreover, the
MWCNT forest/PU film obtained an overall electrical conductivity of 50–100 Sm$^{-1}$, Young’s Modulus and ultimate strength of about 148 and 38% higher than the pure PU, respectively.

Melt blending is another method broadly accepted in fabricating the CNT-elastomeric nanocomposites [10]. CNTs are dispersed within the elastomeric polymer matrix by shearing stresses. The generated shear forces help to break CNT aggregates or prevent their reaggregations during mixing. This approach is generally noted to be simple and common for industrial-based mass production. Verge et al [91] reported on the dispersion by simple melt blending of tiny amounts of CNT in acrylonitrile–butadiene rubber (NBR). The CNT bundles were more effectively exfoliated and dispersed in the NBR with higher acrylonitrile content due to stronger interaction between the acrylonitrile and CNTs. Wu et al [92] fabricated styrene–ethylene–butylene–styrene (SEBS)/CNT composites in the presence of bis(diphenyl phosphate) (BDP) by melt mixing method and explored their flame-retardant and mechanical properties. They reported that the pretreatment of CNTs by BDP greatly improved the final dispersion in SEBS matrix. The improved CNT dispersion was reported to enhance the flame retardency and thermal stability of the composite. A double filler system such as CNT and CBs mixtures have also been reported to affect the dispersion of CNT during melt mixing process by Dang et al [28].

In-situ polymerization technique is the third traditional method for processing polymer nanocomposites. It is regarded as an alternative method, whenever both the solution and melt mixing are not suitable. With this approach, the CNTs are dispersed into a given monomer followed by polymerizing the monomers under certain reaction conditions [93]. The in-situ polymerization methods enable covalent bonding between functionalized nanotubes and the polymer matrix using various condensation reactions [48]. Higher percentage of CNTs may be easily dispersed by this method resulting in a stronger interaction with the polymer matrix. However, this method is only limited to the preparation of nanocomposites that are insoluble in solvent and thermally unstable. Majority of the scientific reports of in-situ polymerization for fabrication of thermosetting composites used epoxy as the base matrix material [94–97]. In this case, CNTs are first dispersed in a resin followed by curing the resin with a hardener under certain conditions. Nonetheless, a number of CNT-rubbery composites have been fabricated using this method and yielded good results. For example, nanocomposites based on poly(trimethylene terephthalate)-block–poly(tetra-methylene oxide) (PTT–PTMO)-segmented copolymer and COOH-functionalized SWCNTs were prepared via in-situ polymerization method by Szymczyk [98]. It was observed that the nanocomposites with low SWCNTs loading (<0.5 wt%) showed uniform dispersion of CNT in the polymer matrix. Again, the tensile strength of the nanocomposites with 0.05–0.3 wt% loading of SWCNTs obtained an improved tensile strength better than the neat PTT–PTMO copolymer without reduction in elongation at break. Roslaniec et al [99] used in-situ method to synthesize a nanocomposite based on multiblock polyester elastomers (PEE) and CNTs, and reported their enhanced mechanical and thermal properties. A key concern here is that the effectiveness of this approach may rely on several parameters such as reaction conditions, type of monomer, nature of filler and its properties, as well as the filler concentration.

The efficiency of composite processing techniques is summarized in Table 2 and Table 3, showing the influence of the techniques on the physical properties (tensile and percolation threshold) of various elastomeric polymer matrices.

In general, the melt mixing technique has been widely used to reinforce elastomers with CNTs. Recently, the combinational approach involving two or more of the individual
processing techniques has also been adopted by many researchers \cite{60,83,112} to achieve better dispersion of CNTs in the elastomeric polymers. Although this approach seems to give complementary advantage offered by each technique, a further study would be needed for compromising laborious procedure, high cost, and longer processing time involved.

| Composite                  | Processing       | TS increase (%) | EM increase (%) | EB increase (%) | Ref  |
|----------------------------|------------------|-----------------|-----------------|-----------------|------|
| NR + 3 phr MWCNT           | Melt             | ~1378           | ~260            | (226)           | [100]* |
| NR + 1 phr MWCNT + 29 phr silica | Melt         | ~38             | ~26             | (33)            | [101]* |
| NR + 25 phr SWCNT          | Melt             | ~173            | ~317            | (27)            | [62]* |
| NR + 3 phr MWCNT + 40 phr silica | Melt         | ~2422           | ~380            | (264)           | [100]* |
| NR + NBR + 2 phr MWCNT     | Melt             | ~18             | ~100            | (5)             | [83]* |
| EPDM + 3 phr MWCNT         | Melt             | ~161            | ~90             | (71)            | [100]* |
| EPDM + 3 phr MWCNT + 40 phr CB | Melt         | ~1587           | ~260            | (203)           | [100]* |
| SBR + 5 phr MWCNT          | Melt             | ~248            | ~85             | (38)            | [67]* |
| SBR + 5 phr MWCNT + 20 phr EG | Melt         | ~146            | ~157            | (10)            | [102]* |
| SBR/BR + 5 phr MWCNT       | Melt             | ~296            | ~530            | (3)             | [102]* |
| SBR + BR + 3 phr CNT + 40 phr silica | Melt | ~839            | ~320            | (71)            | [100]* |
| TPU + 3 wt% MWCNT-g-PTMEG  | Melt             | ~4              | ~6              | (6)             | [70]* |
| SBR + 15 phr MWCNT         | Solid (two-roll) | ~293            | ~262            | N/A             | [63]* |
| PDMS + 5 wt% MWCNT         | Solid (two-roll) | ~155            | N/A             | (21)            | [64]* |
| MVQ + 0.75 wt% MWCNT       | Solvent          | ~38             | ~82             | (46)            | [103]* |
| MVQ + MWCNT + G (0.75 wt%) | Solvent          | ~109            | ~96             | (1)             | [103]* |
| PDMS + 1.6 wt% SWCNT       | Solvent          | ~14             | ~32             | (33)            | [77]* |
| PDMS + 4 wt% SWCNT         | Solvent          | ~183            | ~629            | (7)             | [78]* |
| TPU + 1 wt% (MWCNT+HNT)    | Solvent          | ~143            | ~35             | (68)            | [68]* |
| NR + 25 phr CNT            | Solvent          | ~244            | ~594            | (27)            | [36]* |
| SEBS + 1.5 wt% SWCNT       | Solvent          | ~24             | ~154            | (8)             | [39]* |
| NR + 1 phr MWCNT           | Solvent and melt | ~26             | ~26             | (31)            | [60]* |
| NR + 1 wt% MWCNT           | Latex            | ~58             | ~33             | (3)             | [61]* |
| SBR + 25 phr MWCNT         | Latex and melt   | ~80             | ~163            | (18)            | [104]* |

*untreated CNT, *covalently treated CNT, *non-covalently treated CNT.

Table 2. Mechanical properties of elastomeric/CNT nanocomposites.
In summary, although the melt mixing is widely accepted in the mass production of CNT-filled elastomeric polymer nanocomposites, there are still many arguments for the effectiveness of the melt blending of CNTs into elastomeric polymers, due to incomplete dispersion of CNTs agglomerates at milder mixing conditions and some damages of CNTs at severer shearing conditions. The solution mixing approach usually provides more uniform dispersion of CNTs, but it is however preferred on a laboratory scale. Therefore, CNT-elastomeric composites research should be further focused on finding an improved or efficient way of dispersing CNTs into polymer matrix.

4. Morphology and state of dispersion of CNT

A precise characterization skill of observing the dispersed CNTs in polymer is inevitably crucial for the better understanding of structure–property relations of polymer composites. Most of the properties of composites are strongly related to the morphology and dispersion status of CNTs [1,82]. Thus, the analytical technique which can monitor any type of properties of the polymer composites would be a tool for characterizing the morphology. Several typical techniques marked for these studies include transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), wide-angle x-ray diffractometry (WAXD), thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC) [27,31,32,60,83,85]. The combination of the techniques is often employed to fully understand the dispersion and morphology of CNTs.
The direct observation of CNTs in the polymer matrix is one of the most powerful but difficult techniques. Several images of CNT dispersion and morphology in elastomeric polymers using TEM, SEM, and AFM analyses are shown in Figure 8 for a case study. Tsuchiya et al [109] compared the CNT dispersion in SBR between a new mixer, called ‘rotation-revolution mixer’, and a conventional Banbury mixer based on TEM observation. There is a clear difference in both the shape and dispersion of CNT, as can be seen in Figure 8(a) and (b). A more uniform and homogeneous dispersion was observed in their newly proposed mixer system. A similar difference in morphology and dispersion of CNTs in NR matrix is also noticeable by the presence of acid treatment as shown in Figure 8(c) and (d) [60]. The acid treatment leads to the decoration of the surface of CNTs.

Figure 8. TEM images of SBR-MWCNT composite (0.75 phr) prepared by (a) a rotation–revolution mixer, (b) Banbury mixer [109]; TEM images of NR composite filled with 1% of (c) unmodified CNT, (d) acid-treated CNT [60]; SEM micrographs of NR composites filled with (e) acid-treated MWCNTs, (f) unmodified MWCNTs [61]; SEM images of CNT-filled rubber composite containing 7 phr CNT: (g) without stretching; (h) at 15% stretching [113].
with functional groups such as the -COOH. This in turn improves the interaction of CNTs with elastomeric polymer matrix.

The effect of acid treatment of CNTs is also confirmed using the SEM observation for the cryogenically fractured surface of NR-MWCNT composites by Peng et al [61]. In this experiment, they employed the covalently treated MWCNTs by a latex blending method. The unmodified MWCNTs still remain as agglomerates (Figure 8(f)), while the acid-modified MWCNTs show better dispersion (Figure 8(e)). It should be noted that the SEM observation generally does not give a clearer image of CNTs on the fractured surfaces of elastomeric polymers probably due to the nanosized nature of the CNTs or the mismatch in elongation between polymers and CNTs during rupture. The broken ends of CNTs tend to be embedded deeply into the bulk of polymers. In our previous study, we proposed a new approach for a clearer observation technique [113,114]. In this case, a slight compression was imposed on the polymer sheet for making the CNTs push out to the fractured surfaces during SEM observation, as shown in Figure 8(g) and (h). When the CNTs on the fractured surface of the rubber matrix were observed later, it was noticed that they have reverted into the matrix. Hence, a relatively weak interaction between the untreated CNTs and elastomeric polymers was confirmed. A similar approach was also used to observe clearly the presence of graphene oxide sheet in NBR matrix by SEM after compression of composites [115].

Another observation method is the AFM technique. But the resolution of the AFM technique has been known to be very sensitive to the hardness and smoothness of the surface of specimens. Most of the elastomeric polymers form a smoother surface. Generally, it is very difficult to obtain a successful AFM morphology (especially in topology mode) of elastomeric polymer composites, as shown in Figure 9(a) and (b). Thus, the phase mode of the AFM technique is more useful for such analysis of soft polymer-CNT composites, because the phase mode can provide the variation of surface stiffness [82]. In effect, there is great difference in stiffness between soft polymers and CNTs. Another new approach (called electronic force microscopy, EFM) was proposed in our earlier work [116]. A low current of few volts was applied onto the surface of the specimen by the AFM tip. When the tip is placed on the CNTs, higher currents are tunneling, while on the polymers, lower current is passing. The image based on the current map was quite successful to represent the dispersion of CNTs in an elastomeric polymer system, as shown in Figure 9(c) and (d).

A unique way to quantify the structures and dispersion of CNTs in an elastomer matrix is the technique based on the Raman spectroscopy. It is a widely used technique for studying structure and electronic properties of CNTs and graphitic materials [1], and the interfacial interactions between rubber matrix and MWCNTs [76,82]. Figure 10 illustrates the Raman spectra in the frequency range from 1200 to 1700 cm$^{-1}$ for MWCNT, MWCNT in a rubber matrix, and MWCNT in a rubber matrix in the presence of ionic liquid [82]. MWCNTs show two characteristic peaks, one at 1326 cm$^{-1}$ assigned to the D band and the other to G band at 1593 cm$^{-1}$. The D band is originated from disordered graphite structure and the G band is associated with tangential C–C bond stretching of the graphite-like structures. No change of the D and G band positions was observed upon incorporation of 3 phr of MWCNTs into the rubber. On other hand, a noticeable shift of G band toward the higher frequencies is been observed with ionic liquid. It is consequently established that the disentanglement of the MWCNTs and their subsequent dispersion and chemical interaction with the rubber by the ionic coupling agent might be the explanation behind the shift [82,117].
Figure 10. Raman spectrum of MWCNT, MWCNT-rubber, and MWCNT-IL-rubber composites. IL represents an ionic liquid of 1-allyl-3-methylimidazolium chloride, and the MWCNT loading was 3 phr [82].

Figure 9. AFM and EFM images of CNT dispersion in elastomeric matrix: (a) AFM images of composite with 2.8 wt% MWCNT before coagulation, (b) after coagulation of the latex beads at 60°C [24], (c) EFM phase image at 0 V, and (d) EFM phase image at 7 V of NBR-MWCNT composite [116].
Based on these observations, it is evident that the efficacy of CNT dispersion in an elastomer depends strongly on several factors as already mentioned. It should be emphasized here that no single technique provides sufficient analytical information related to the structural characteristics of rubber nanocomposites. Thus, it is recommended to use more than one technique to understand the structures and properties as well as the performance of the polymer nanocomposites [118].

5. Mechanical property

The physical properties of the elastomeric material strongly depend on the various parameters of the employed filler system, such as volume fraction, size and shape of particles, interactions among fillers, and filler–matrix interactions [24,119]. The interaction between the filler particles and the rubber matrix (often called as bound rubber) is generally considered to be the most important aspect [119]. The reinforcing behavior by fillers is explained by various theoretical models, including Einstein (Equation 2), Guth–Gold (Equation 3), Thomas (Equation 4), and Halpin–Tsai (Equations 5, 6) [64,119,120–122]. More extended models are available in reference [85].

\[
\frac{E_f}{E_u} = 1 + 2.5\phi 
\]

\[
\frac{E_f}{E_u} = 1 + 2.5\phi + 14.1\phi^2 
\]

\[
\frac{E_f}{E_u} = 1 + 2.4\phi + 10.2\phi^2 + Ae^{B\phi}
\]

\[
\frac{E_f}{E_u} = \frac{(1 + 2f\phi\eta)}{(1 - \phi\eta)}
\]

\[
\eta = \frac{(E_f/E_u - 1)}{(E_f/E_u + 2f)}
\]

where \(E_f\) and \(E_u\) are the moduli at a given elongation of filled and unfilled polymer, \(\phi\) is the volume fraction of fillers, \(f\) is the aspect ratio of the fillers, and \(A, B\) are constant parameters obtained experimentally.

It has been suggested that the fiber shape of nanofillers such as CNTs are the most efficient in enhancing the stiffness [123]. Previously, we reported a significant improvement in both the modulus and the strength, when the unmodified MWCNTs were added to the NR matrix (Figure 11(a)) [42].

The 100% modulus of the MWCNT-NR nanocomposites was found to be about 150% higher than that of CB-NR composites at the same loading level, even though the bound rubber content denoting filler–rubber interactions was even smaller for the MWCNT nanocomposites. Based on the SEM observation of the poor interaction between MWCNT and NR matrix, the main cause of the reinforcing mechanism of the
MWCNT-NR nanocomposites was considered to be the effective load transfer due to the high aspect ratio of MWCNTs. The poor interactions between CNTs and rubber matrix are also contained in several literature [25,93,113,121,124]. In addition, a higher reduction in the storage modulus with increasing dynamic strain amplitude was observed for the MWCNT-NR nanocomposites than the CB-NR compounds at the same loading level (Figure 11(b)). This strongly indicates that the Payne effect is more noticeable in the nanocomposites, possibly due to the higher breakdown of the filler–filler networks among MWCNTs. A similar result was also reported later by Bokobza [25]. Another point which should be mentioned here for the mechanical reinforcement is the state of dispersion of CNTs in the rubber matrix. The surface modification of CNTs is widely accepted for the

Figure 11. (a) Stress–strain curves of NR, NR/CNT, and NR/CB compounds and (b) storage modulus of NR/CNT and NR/CB compounds at different filler loadings as a function of dynamic strain amplitude [114].
better dispersion \[60,63,65,83\]. For instance, a 10-times improvement in the modulus without severe deterioration of elongation at break was reported by employing a carboxylated MWCNT \[24\], and 58% improvement in tensile strength was reported by employing a small amount (1 wt%) of acid-modified MWCNTs in NR \[61\].

Recently, the hybridization of components of composites is receiving a greater attention for any synergistic effect among them. They include a dual filler system \[46,101,103,108\], dual matrix system \[63,69,125\], and ternary matrix system \[126,127\]. A synergistic effect of the fillers between MWCNT and graphene (G) was found in polydimethylsiloxane (PDMS) \[103\]. They reported a considerable improvement in tensile strength of 110% and tensile modulus of 137% by using an extremely small amount of filler loading of 0.75 wt%. Kueseng et al \[83\] reported another way of enhancing mechanical property based on dual rubber system. They initially prepared the master batches by mixing CNTs in NR matrix, and then the master batches were blended with NR–NBR blends of various ratios. A significant enhancement in mechanical properties was found for the master batch system than the conventional mixing approach. This was ascribed to the improved CNT dispersion, which mostly occurred at lower filler concentration between 0 and 4 phr CNT. This study clearly shows the importance of CNT dispersion in rubber matrix.

In brief, the incorporation of CNTs in elastomeric matrices characteristically increases their mechanical properties due to the enhanced load transfer capability of the fibril shape of CNTs with high aspect ratio. The proper surface modification of CNTs by either covalently or non-covalently can further improve the mechanical performance through strong chemical bonding of CNTs with the rubber molecules. A synergistic effect can also be possible by employing dual fillers and rubber system.

6. Electrical property

Since the electrical conductivity of CNTs is known to be extremely high (~18,000 S cm\(^{-1}\)) \[85\], a significant increase of the conductivity of elastomers can be expected when they are properly dispersed. A better dispersion of CNTs generally results in larger number of conducting paths in the rubber matrix. The critical volume fraction leading to a sharp drop in the electrical resistance is known as the percolation threshold (Figure 12).

![Figure 12. An illustration of percolation threshold by network formation of CNTs in a rubber matrix.](image-url)
The percolation threshold depends on the CNT type (SWCNT, MWCNT), basic features of CNTs such as amorphous carbon content, ratio of metallic to semiconductive tubes, aspect ratio, morphology, type of rubber matrix, and dispersion state and particle orientation [20,24]. Although, there are considerable disagreements in the percolation threshold values among researchers [13,15,128,129,130], it is generally agreed that the percolation threshold of CNTs can be achieved at much smaller loading (≥ 0.1 phr), when compared with conventional CB-filled elastomeric compounds [28,131].

The estimation of the electrical conductivity is generally made by measuring the electrical resistance, and it is represented in the volume resistivity (σ) given by:

\[
\sigma = \frac{L}{AR}
\]

where \( L \) and \( A \) are the length and cross-sectional area of the specimen, respectively, and \( R \) is the electrical resistance. The prediction of percolation behavior is given by the scaling law [132,133] as follows:

\[
\sigma = \sigma_0 (\phi - \phi_c)^t
\]

where \( \sigma \) is the electrical conductivity, \( \sigma_0 \) is a fitting parameter, \( \phi \) is the volume fraction of CNTs and \( \phi_c \) is the critical percolation concentration, and \( t \) is an exponent that governs the scaling law in the vicinity of percolation. The parameters \( t \) and \( \phi_c \) are usually determined by plotting log \( \sigma \) vs. log (\( \phi - \phi_c \)) [129]. However, the use of Equation (8) is only limited to the solid matrix composite system like polymer-CNTs composites. A dynamic colloid theory is needed for the fluid-CNTs system like CNTs colloids [134,135].

Quite a different percolation threshold was reported based on the experimental observation depending on the type of CNTs and rubber matrix. Katihabwa et al [64] reported the percolation threshold between 5 and 10 wt% of an unmodified CNT-filled PDMS compounds. Koerner et al [85] found the percolation threshold to be 0.5 ~ 10 vol% for MWCNT-filled thermoplastic polyurethane (TPU) compounds, and above the percolation threshold the conductivity reached was 1 ~ 10 Scm\(^{-1}\). The lower percolation threshold seems to be due to the high aspect ratio of fibrous shape of CNTs.

Modification of CNTs can provide further enhancement in dispersion, and the better dispersion affects the electrical conductivity of the composite systems. Yadav et al [38] found the percolation threshold of 2 wt% by employing the non-covalent modification SWCNTs in SEBS via solvent casting method. Subramaniam et al [76] observed a good dispersion of ionic liquid-treated MWCNTs in chloroprene rubber (CR) matrix via a two-roll mill mixing. They achieved an electrical conductivity of 0.1 Scm\(^{-1}\) at the percolation threshold at 5 phr. However, a controversial result of electrical conductivity was reported by Abdullateef et al [60], when nitric acid-treated CNTs were dispersed in NR matrix with excellent dispersion. In this case, the electrical conductivity of CNTs themselves was reduced by breaking the unsaturated double bonds on the CNT’s surface.

Another approach to improve the electrical conductivity is the hybridization of polymers and fillers. Yesil [127] reported the reinforcing behavior of CNTs on the recycled poly(ethylene terephthalate) (PET)–poly(ethylene naphthalate) (PEN) blends using functional elastomers, notably ethylene-methacrylate copolymer (EMA)–glycidyl methacrylate (GMA). The miscibility between PET and PEN was greatly improved due to the compatibilizer role of the EMA–GMA, resulting in a better dispersion of CNTs in polymer matrix. This finally led to the improvement in the conductivity. It has been also reported
that the use of dual fillers system can enhance the conductivity [108]. When the CNTs were used together with G, the dispersion and the conductivity was improved compared with that of CNT-reinforced compounds. The synergistic effect of the hybrid materials is also available for various matrix systems [63,100,103].

It should be mentioned here that the processing techniques affect the electrical conductivity as well. Bokobza [87] studied the percolation threshold of CNTs in various types of elastomers, including NR, SBR, and EPDM (Figure 13). The percolation threshold was generally found to be around 0.5 phr (0.002 in volume fraction) regardless of rubber matrix. The rotation–revolution mixer was reported to give better dispersion leading to a better conductivity than that of conventional Banbury type mixer [109]. They reported a percolation threshold less than ~1 phr for SBR-CNT composites. Table 3 summarized several factors affecting the electrical percolation threshold of representative CNT-elastomeric-based nanocomposites.

7. Thermal property

CNTs are also known to be thermally stable and have higher thermal conductivity of ~6600 Wm$^{-1}$K$^{-1}$ for SWCNTs [136] and 3000 Wm$^{-1}$K$^{-1}$ for MWCNTs [137]. When the thermally stable CNTs are dispersed in less stable rubber matrices, the thermal stability of the polymer composites can be improved depending on the loading amount of CNTs. Another reason for the thermal stability improvement of the composites is that the dispersed CNTs in rubber matrices help to dissipate the heat more quickly in the composites because of their higher thermal conductivity [138]. The heat conduction mechanism of CNTs in a rubber matrix was proposed by He et al [32] based on the molecular dynamics simulation. Meanwhile, it should be emphasized that the stability of a composite may also depend more on the chemical nature (backbone or side groups of the polymer) of the matrix material employed.
Pham et al [139] studied the thermal decomposition property of FKM reinforced with unmodified MWCNTs. The decomposition temperature gradually increased with increased MWCNTs. They pointed out two reasons for the improvement. Firstly, the presence of MWCNTs made the vulnerable sites of fluorocarbon backbone inactive by physical and chemical interactions between MWCNTs and FKM. Secondly, the MWCNTs played an antioxidant role. Perez et al [63] also investigated the effect of content and modification of MWCNTs on the thermal degradation stability of two rubber matrices, including NBR and SBR using TGA under nitrogen condition.

As summarized in Table 4 and Figure 14, the SBR-MWCNT composites showed a single drop in the weight loss curve, while the NBR-MWCNT composites showed two at the lower loading level of 0 ~ 5 phr, probably due to the characteristic feature of NBR degradation. The first loss peak of NBR-MWCNTs disappeared when the MWCNTs loading was further increased above 10 phr, where the percolation threshold was reached. They explained that the disappearance of the first loss from gaseous subproducts peak was attributed to the barrier role of percolation networks of MWCNTs.

It was reported that the modification of CNT was also effective for improving the thermal stability of NR matrix [60]. They argued that the thermal degradation improvement of ~10°C in the thermal degradation temperature by adding the acid-treated CNTs was attained, while no noticeable improvement for unmodified CNTs occurred. Shang et al [140] also studied the significance of CNT treatment on the thermal behavior of PDMS, as depicted in the TGA and their derivative curves in Figure 14.

The chitosan hydrochloric acid salt (CSCI)-treated MWCNTs improved thermal degradation stability of the SR composite with a one-step weight loss process. When non-pretreated MWCNTs were added into SR, the composite showed a three-step weight loss process and the thermal stability did not improve as well. They claimed this phenomenon indicated that the untreated MWCNTs damaged the internal structure of PDMS to some extent. More research results on the modification effect of CNTs on the thermal stability are reported [41,70,72].

Although several approaches were reported by researchers regarding the thermal stability boost of CNT-polymer-based composites, yet it seems that good dispersion is a common prerequisite of the formation of CNT networks, leading to a slowdown of thermal degradation. The proper selection of the rubber matrix would also enhance the thermal stability of the composite. For instance, FKM is known to show a superior thermal stability compared with many other rubbers due to the presence of fluorine in

| CNT (phr) | SBR First weight loss | NBR First weight loss | Second weight loss |
|----------|----------------------|----------------------|-------------------|
|          | Maximum (°C) Residue (wt%) | Maximum (°C) Residue (wt%) | Maximum (°C) Residue (wt%) |
| 0        | 447.4 5.0 | 423.9 64.9 | 445.7 9.2 |
| 2        | 450.0 6.4 | 424.1 64.1 | 446.5 12.0 |
| 5        | 451.3 8.8 | 425.8 61.7 | 425.8 11.8 |
| 10       | 454.8 12.9 | 443.5 17.7 | |
| 15       | 455.1 16.3 | 452.7 20.6 | |
their backbone. Thus, the highly polar C–F bonds and helical structure of the main chain impart high temperature stability. It was also reported earlier that NBR containing a higher amount of acrylonitrile content tends to show higher thermal stability in compounds with CNTs [91,141].

8. Applications

The unique properties of CNTs makes them superior compared to conventional fillers such as carbon blacks and siliconas which require higher filler loading to boost the physico-mechanical properties of polymers [3,9,142]. Thus, the multipurpose nature of rubber-CNT-based nanocomposites makes them potential candidates for widespread applications in various fields. The characteristic features of flexibility, stretchability, conductivity and robustness of rubber-CNT nanocomposites brands them highly applicable in pressure, and strain sensors [47,71,90], super-capacitors, and charge-storage capacitors[38,41,77], thermally stable and electrical cable materials [60,92], oil/fuel hoses and gaskets [105], and

Figure 14. TGA thermograms (a) and corresponding DTG curve (b) of pure silicone rubber (SR) and SR/MWCNTs nanocomposites [140].
microwave, structural applications in the aerospace industry [143], etc. It is therefore obvious that rubber-CNT could enjoy unlimited applications if researchers across the globe further engage deeply into the studies of functionalization, processing techniques, structure–property–dynamics relation of elastomers, and the physics of interactions of CNT–elastomer, as well as the interactions that occur among their hybrid systems.

9. Concluding remarks
The CNT-reinforced elastomeric nanocomposites have received a gradually increased attention. Various functional properties, including mechanical, electrical, and thermal property of rubbery materials, have been altered even at a small amount of CNTs, when the proper dispersion of CNTs is achieved with the help of surface treatment. This review article has addressed the recent research issues focusing on the surface treatment of CNTs, processing, state of dispersion, and several properties of the CNT-reinforced elastomeric polymer nanocomposites.

The accepted method of CNT modification can be classified into covalent and non-covalent type. The covalent modification method, notably by acid treatment, was widely adopted for CNT-rubber nanocomposites. In the non-covalent modification, the ionic surfactants were widely selected. The proper choice of modification method seems to be crucial for the application purpose of the resulting nanocomposites.

The mixing of CNTs into rubber matrices was done by solution mixing, melt mixing, and in-situ polymerization. Even though the melt mixing was widely used in a large-scale production, the solution mixing was selected for more complete dispersion with lower damages of CNTs. There should be further challenging ways for finding more efficient mixing techniques in view of either quality of dispersion or surface damages. The characterization skills of CNTs and the state of dispersion in the rubber matrices have been successfully developed recently. They include TEM, SEM, AFM, WAXD, TGA, DSC, and others. Raman spectroscopy is newly proposed as the characterization technique for CNTs.

The incorporation of CNTs in elastomeric matrices characteristically enhances the mechanical properties due to their higher load transfer capability of the fibril shape with high aspect ratio, although there is a poor interaction between rubber and CNTs. A further improvement is expected with a proper surface modification of CNTs. The percolation threshold of the electrical conductivity of CNTs is comparatively lower than those of CBs due to their easier susceptibility for making CNT network owing to their fibril structure. Care should be given to the selection of the modification type of CNTs, since it greatly affects the electrical conductivity of CNTs themselves. There are two types of mechanisms for the improvement of the stability of CNT-reinforced rubber composites. One is from their originally higher thermal stability (when properly dispersed). The thermally stable CNTs can replace the less stable rubber matrix throughout the volume. Another is from the improved thermal conductivity which results in fast dissipation of accumulated heat inside the bulk polymer matrix.

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