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Research Article

The Effects of Multi-Walled Carbon Nanotubes on the Mechanical Properties of Silicone Rubber-Based Nanocomposites

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ABSTRACT

In this study, multi-walled carbon nanotubes (MWCNTs) were used as reinforcement for silicone rubber at different weight percentages (i.e. 0, 0.25, 0.5, and 1 wt.%). The tensile, compressive, and compression set behaviors of the prepared composites were examined through standard tests. The fracture surface of the tensile samples was also analyzed using a scanning electron microscope (SEM). The results indicate that incorporating 0.25 wt.% MWCNTs into silicone rubber leads to an 11% increase in tensile strength and a 27% increase in elastic modulus compared to pure silicone rubber. Furthermore, the presence of MWCNTs nanoparticles enhances the compressive strength from 3.2 MPa in pure silicone to 6.2 MPa at 0.25 wt.%, while increasing the MWCNT content up to 1 wt.% gradually decreases the compressive strength. Additionally, the presence of MWCNTs reduces the compression set of silicone rubber.

1. Introduction

Carbon-based nanoparticles play a crucial role in the development of polymer nanocomposites. These nanoparticles exhibit unique properties, including mechanical strength, electrical conductivity, and thermal stability, which vary depending on their type [1-4]. Among them, multi-walled carbon nanotubes (MWCNTs) stand out due to their exceptional mechanical and electrical properties. Since their discovery, nanotubes have attracted significant interest in chemistry, physics, biology, and environmental science due to their remarkable properties. In engineering and materials science, even small amounts of nanotubes have been shown to significantly enhance the mechanical, thermal, and electrical properties of

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nanocomposites. However, their poor dispersion in solvents and polymer matrices remains a major challenge for practical applications. This inadequate dispersion is primarily attributed to the strong van der Waals interactions between nanotubes and their high aspect ratio. Consequently, nanotubes tend to accumulate and agglomerate in polymers and solvents, which hampers the enhancement of composite properties [5-8]. Various physical or chemical surface treatment methods have been explored to enhance the dispersion of nanomaterials. Similarly, different techniques have been employed to optimize nanotube dispersion in polymer matrices. Although these methods may slightly reduce the inherent properties of nanotubes, they significantly improve dispersion. These methods include solubilization, ultrasonic waves, melt composition, in situ polymerization, oxidation, chemical functionalization of the nanotube surface, absorption or mixing with polymer threads and biomolecules, and the use of surfactants. Functionalization via certain methods leads to the covalent bond formation at defect sites on the nanotubes, causing a transition from sp^2 to sp^3 hybridization [8-11].

Silicone rubber is a versatile material composed of silicon, oxygen, hydrogen, and carbon. It is known for its non-reactivity, stability, and resistance to extreme temperatures [12]. Due to these properties, along with its ease of manufacturing and shaping, silicone rubber is widely used in various products, including voltage line insulators, automotive components, cooking and baking tools, sportswear, electronics, medical devices and implants, as well as home repair and hardware products like silicone sealants [13]. The incorporation of nanofillers is essential for enhancing the performance of next-generation flexible rubber composites [14, 15]. Among the available options, room temperature vulcanized (RTV) silicone rubber is a particularly effective choice due to its ease of processing, high flexibility, low viscosity, and impressive mechanical and electrical properties [16]. Furthermore, the selection of nanofiller plays a crucial role in determining the overall performance of the composite [17]. Commonly used nanofillers include carbon nanotubes (CNT) [15, 16, 18], graphene [19], carbon black [20], and titanium dioxide [21]. Composites made from RTV silicone rubber and these nanofillers have been applied in various fields such as coatings [22], medical devices [23], actuation [14, 24], energy harvesting [25], and strain sensing [26].

The addition of single-walled and multi-walled carbon nanotubes significantly improves the properties of silicone rubber composites. With just 1 wt.% of CNT reinforcement, the modulus of the composite and tensile strength increase by approximately 50% and 28%, respectively, compared to pristine silicone rubber [27, 28]. Najib Alam et al. [29] investigated a stretchable, synergistically toughened silicone nanocomposite-based piezoresistive strain sensor using multi-walled carbon nanotubes (MWCNTs). Their findings revealed that one-dimensional MWCNTs mechanically reinforce the silicone rubber matrix, improving tensile strength by 95% but decreasing fracture strain by 27% at 5 phr, while also forming an electrical percolation network. Furthermore, the incorporation of graphite nanoplates enables silicone rubber nanocomposites to exhibit significant piezoresistive behavior under very low pressures [30]. Combining carbon nanotubes and carbon black enhances electrical conductivity, making them suitable for pressure-sensitive sensors [31]. However, previous studies on the mechanical and thermal properties of silicone rubber indicates varying effects depending on the type of used filler. While some research reports positive impacts, others remain inconclusive. In particular, the influence of multi-walled carbon nanotubes (MWCNTs) on the mechanical behavior of silicone rubber is still debated. These conflicting findings highlight the need for further investigation to determine the optimal conditions for incorporating MWCNTs into silicone rubber composites. To address this gap in the literature, the present study aims to examine the effect of MWCNTs on the mechanical properties of RTV silicone rubber.

2. Experimental Procedure

2.1. Materials

In this study, we used Bluesil RTV 3325, a white liquid silicone rubber purchased from Mouldlife Company

(China). The curing agent, also designated as RTV 3325, is a colorless peroxide obtained from the same supplier. The rubber is cured at room temperature in a 10:1 ratio of silicone rubber to peroxide. Upon the addition of the curing agent, a cross-linked Si–O–Si network structure forms. Once cross-linking is complete, each Si-O-Si molecular chain is covalently bonded to adjacent molecular chain, creating a stable, interconnected structure. The carbon nanotube (purchased from Nonolin company) used in this research has an outer diameter of 20-30 nm and an approximate length of 30 µm. It is functionalized with a carboxyl agent (-COOH) to prevent agglomeration due to its huge surface area and to enhance compatibility and adhesion. Fig. 1 shows transmission electron microscope (TEM) micrographs of the used multi-walled carbon nanotube at different magnifications.

2.2. Sample preparation

To investigate the impact of MWCNTs on the mechanical properties of silicone rubber, different weight percentages of MWCNTs (i.e. 0, 0.25, 0.5, and 1 wt.%) were incorporated into the silicone rubber. In this study, the MWCNTs content was limited to a maximum of 1 wt.% due to several challenges. One significant issue was the agglomeration of MWCNTs, which hindered dispersion and negatively affected the mechanical and electrical properties of the composites. Additionally, the presence of MWCNTs increased the viscosity of the silicone mixture, complicating processing methods such as mixing and molding. To ensure consistent sample production, polymethyl methacrylate (PMMA) molds were utilized. PMMA was chosen due to its ease of removal, preventing damage or adhesion to the sample surfaces. The PMMA sheet was shaped into a dumbbell mold for tensile test samples, following the ASTM-D412 standard. The test samples had a width of 6 ± 0.05, a length of 33 ± 2 mm, and a thickness of 3 ± 0.2 mm. Fig. 2 shows the mold used for producing the tensile samples.

Similar to the tensile specimen, the PMMA sheet for the compression samples was shaped into a cylinder with a hollow-centered cylinder following the ASTM-D575 standard. The cylinder has an inner diameter of 28.5 mm and a height of 12.5 mm.

Different weight percentages of MWCNTs (i.e. 0, 0.25, 0.5, and 1 wt.%) were added to a mixture of RTV silicone rubber and its hardener. The mixture was sonicated using a probe at a constant frequency of approximately 20 kHz for 25 minutes to ensure uniform dispersion of the particles. After sonication, the curing agent was added to the rubber at a 1:10 ratio. To remove any air bubbles that formed during mixing, the final mixture was placed under a vacuum pump. The resulting nanocomposite was then injected into the mold using a large syringe. All samples were produced at room temperature. Table 1 provides a summary of the produced samples.

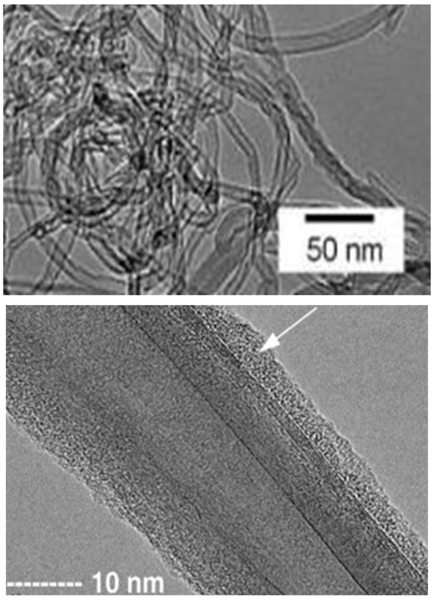

Fig. 1. TEM micrographs taken from the used MWCNTs. The white arrow showing the MWCNTs.

Fig. 2. PMMA mold for producing tensile standard samples.

Table 1. Chemical composition of the spring steel 54SiCr6 (wt.%)

Sample code	Silicone rubber (wt.%)	MWCNTs (wt.%)
SR	100	0
SR/0.25 wt.% MWCNTs	99.75	0.25
SR/0.5 wt.% MWCNTs	99.5	0.5
SR/1 wt.% MWCNTs	99.0	1.0

2.3. Tensile test

To evaluate the tensile properties, dumbbell-shaped specimens were prepared following the ASTM-D412 standard. The tests were conducted at room temperature using a Santam Universal Tensile Testing Machine (Model STM-150) with a crosshead speed of 400 mm/min.

2.4. Compression test

To perform the test, samples were prepared using the pressure mold described in the previous section. The prepared samples were then tested using the Santam Universal Tensile Testing Machine (Model STM-150).

2.5. Compression set test

The samples were prepared according to the ASTM-D395 standard, with a diameter of 29 mm and a thickness of 12.5 mm. Each sample was placed between two plates and compressed to approximately 25% of its original height. After relieving the stress, the samples were left undisturbed for 30 minutes. The compression set percentage (C_s) was then calculated using Eq. (1):

$$C_s\,(\%) = [(t_0 - t_i) / (t_0 - t_s)] \times 100 \qquad (1)$$

Where C_s represents the compression set, t_0 and t_i correspond to the initial and final thicknesses of the sample, and t_s denotes the spacer thickness (approximately 9.5 mm).

2.6. Microscopic evaluation

A scanning electron microscope (SEM, Cambridge) was used to evaluate the fracture surface of the tensile samples. Before imaging, a thin layer of gold was applied using a coating device to avoid surface charging. This coating improved conductivity and enhanced the quality of the SEM images.

3. Results and Discussion

3.1. Tensile properties

Fig. 3 presents the stress-strain curves for silicone rubber composites containing varying amounts of MWCNT (i.e. 0, 0.25, 0.5, and 1 wt.%). A summary of the tensile properties is provided in Table 2. The tensile strength of silicone rubber increases with the addition of MWCNTs up to 0.25 wt.%, but decreases at 1 wt.%. This decline is attributed to agglomeration at higher concentrations, which causes the applied stress to be used for debonding rather than promoting matrix shear yielding. Consequently, the effective surface area is reduced, impairing the ability of samples to withstand additional stress. At lower MWCNT concentrations, the applied stress facilitates matrix yielding without diminishing yield strength. The presence of nanotubes within the polymer chains raises the tension required for alignment, leading to lower strain under constant tension. As tension escalates, debonding may occur, creating occasional voids. The rough surface of MWCNTs fosters a strong mechanical bond with the substrate, thereby enhancing material properties. Furthermore, the incorporation of MWCNTs increases energy dissipation in the nanocomposites compared to neat rubber, as reported in previous studies [32, 33]. Carbon nanotubes (CNTs) have gained considerable interest in materials science for their ability to enhance the mechanical properties of silicone rubber. As reinforcing agents, CNTs improve tensile strength and stiffness due to their high aspect ratio and exceptional mechanical properties.

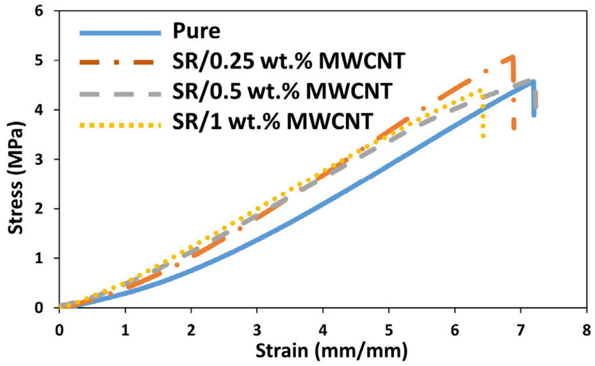

Fig. 3. Stress-strain curves for silicone rubber nanocomposites containing different amounts of MWCNT.

Table 2. The tensile properties of the prepared materials

MWCNT (wt.%)	**0**	**0.25**	**0.5**	**1**
Tensile strength (MPa)	4.57	5.07	4.62	4.41
Elongation (%)	733.13	701.37	735.76	645.89
Young's modulus (MPa)	0.29	0.37	0.47	0.55
Toughness (MJ/m^3)	14.13	15.89	16.8	13.88

They facilitate efficient load transfer within the rubber matrix, increasing the elastic modulus and toughness, which allows the material to absorb more energy before fracturing. Additionally, CNTs improve crack resistance by impeding crack propagation, enhancing durability, and contributing to better thermal and electrical conductivity. Their presence also influences the viscoelastic behavior of silicone rubber, which is critical for specific applications. However, achieving uniform CNTs dispersion remains a challenge, as poor dispersion can adversely affect the material's properties. The incorporation of agglomerated CNTs rubber can weaken tensile properties due to several factors, including the formation of weak points, inadequate interfacial bonding that hampers effective load transfer, increased viscosity that complicates processing, and stress concentrations that initiate cracks. Agglomerates can also disrupt the elastic network of the silicone, reducing its stretchability and tensile strength. To mitigate these issues and enhance tensile properties, achieving proper dispersion of CNTs through techniques like surfactants, high-shear mixing, or using functionalized CNTs are essential [34-37].

Figs. 4(a) and 4(b) present scanning electron microscopy (SEM) micrographs of the fracture surfaces of tensile samples of silicone rubber reinforced with 0.5 and 1 wt.% MWCNTs, respectively. The images clearly show the presence of MWCNT particles, demonstrating their dispersion within the matrix. However, some agglomeration is also evident, indicating a lack of compatibility between the matrix and the nanoparticles. Voids are also visible, likely formed due to stress concentration resulting from the modulus difference between RTV silicone rubber and MWCNTs. As with other nanoparticles, MWCNTs tend to agglomerate at higher concentrations due to mutual attraction through van der Waals forces, which hinders property enhancement and limits the potential applications of nanostructured materials [38]. This clustering reduces the effectiveness of MWCNT as reinforcements, making it easier for applied forces to cause debonding rather than improving load resistance. Regarding modulus, cavitation and debonding were not observed as the measurements were taken at very small strains. However, since MWCNTs have a significantly higher modulus than silicone rubber, an overall increase in the modulus of the composite is expected. The introduction of nanoparticles restricts polymer chain mobility, leading to a more brittle composite with an increased modulus. This trend aligns with previous research. For example, Rafiee et al. [39] reported a 31% increase in the modulus of elasticity with the addition of 0.1 wt.% MWCNTs to epoxy. Similarly, Liang et al. [40] observed a 76% increase in Young's modulus with the incorporation of just 0.7 wt.% graphene into a polyvinyl alcohol nanocomposite.

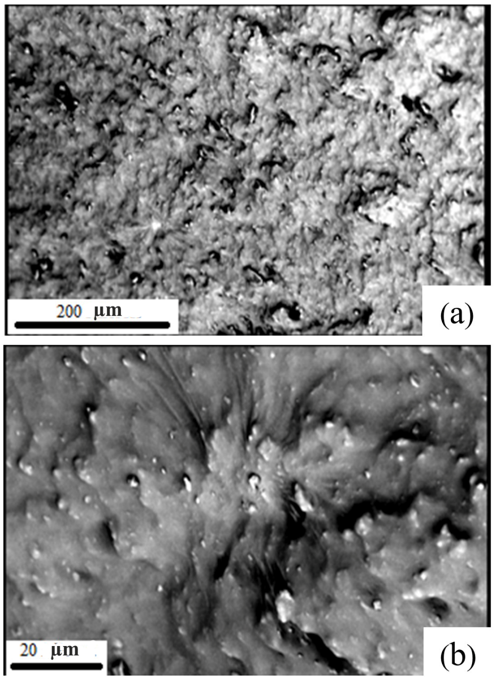

Fig. 4. SEM micrographs taken from the fracture surface of a tensile specimen of silicone rubber reinforced with (a) 0.5 wt.% MWCNTs, (b) 1 wt.% MWCNTs.

3.2. Compressive properties

The compressive behavior of silicone rubber matrix composite reinforced with varying amounts of multi-walled carbon nanotubes (MWCNTs) was investigated. Compression tests were performed on all samples until the strain reached approximately 0.6, as none of the samples fractured during testing. Fig. 5 shows the engineering stress-strain curves for silicone rubber reinforced with 0, 0.25, 0.5, and 1 wt.% MWCNTs. The increase in compressive stress can be attributed to the efficient stress transfer from the matrix to the filler, which has a large surface area. Additionally, some of the applied stress contributes to the buckling and barreling of the nanotubes, and the multi-walled structure of the nanotubes enhances this effect. In other words, an effect enhanced by their multi-walled structure. In other words, the compressive strength improvement in silicone rubber composites is closely linked to the ability of the applied load to transfer effectively from the matrix to the MWCNTs. Furthermore, MWCNT absorbs more energy during testing due to direction change, bending, and wrinkling. The applied pressure during testing can also enhance the bonding between the MWCNTs and the matrix, delaying their debonding. For instance, with the addition of 0.25 wt.% MWCNTs, the stress at 0.6 increased by over 85% compared to the pure sample. In related research on nanocomposite compressive strength, Yue et al. [41] reported that using two different methods to produce cement reinforced with multi-walled carbon nanotubes led to an increase in compressive strength from 12.53 MPa to 13.5 MPa and 15 MPa with the addition of only 0.2 wt.% MWCNTs over a seven-day curing period.

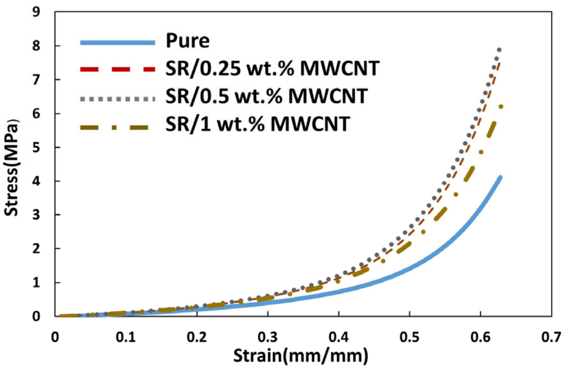

Fig. 5. The compression test of silicone rubber reinforced with 0, 0.25, 0.5 and 1 wt.% MWCNTs.

Similarly, Naik et al. [42] investigated the impact of adding MWCNTs at 0, 1, and 3 wt.% to thermoset epoxy resin and observed an increase in compressive strength and energy absorption capacity. They attributed this enhancement to improved toughness and hardening of the resin, as well as delayed failure initiation and propagation due to nanotube reinforcement, ultimately leading to a stronger material.

3.3. Compression set behavior

The compression set test results, calculated using Eq. (1) after subjecting the silicone rubber and its nanocomposites to pressure for 24 hours, are presented in Table 3. The results indicate that the MWCNT agglomeration of particles has reduced the compression set. In summary, the MWCNTs agglomeration negatively affects the compression set behavior of silicone rubber composites by impairing dispersion, reducing interfacial bonding, and altering stress distribution. To enhance the performance characteristics of silicone rubber composites, achieving uniform dispersion of MWCNTs is essential.

Table 3. Compression set results of silicone rubber and its nanocomposites

Sample	**Pure SR**	**SR/0.25 wt.% MWCNTs**	**SR/0.5 wt.% MWCNTs**	**SR/1 wt.% MWCNTs**
Compression set (%)	2.76	2.0	1.3	1.1

Compression set quantifies the ability of rubber to change shape and return to its original thickness after enduring a compression load at a constant temperature. This value is primarily influenced by the elastic recovery of the material, which is directly influenced by the viscoelastic behavior of polymers.

Soft materials like silicone rubber exhibit higher viscosity, leading to significant energy dissipation in the radial direction as the sample flows and expands under applied pressure. The incorporation of multi-walled carbon nanotubes (MWCNTs) into the matrix further enhances this energy dissipation by creating additional transverse connections.

A closer look at the compression set results reveals that the numerous transverse joints (cross-linkers) act as barriers against pressure, increasing internal stress within the material. This resistance can cause some of these bonds to fracture. Consequently, when the applied load is released, fewer joints remain intact to recover strain, preventing the specimen from regaining its original thickness.

In other words, MWCNTs hinder polymer chain movement under pressure due to their high specific surface area and their ability to form transverse connections with the matrix. As a result, the elastic properties of the RTV nanocomposite improve with increasing MWCNT content, reducing their ability to return to their initial height. However, the random distribution of nanotubes within the matrix enables certain nanotubes to resist buckling and revert to their original state after pressure release, enhancing the mechanical resilience of the composite. This unique behavior not only increases the material's durability under stress but also helps it maintain structural integrity through repeated cycles of loading and unloading. Consequently, the composite performs better in applications where flexibility and strength are essential, such as aerospace, automotive, and wearable technologies. Additionally, the nanotubes' ability to dissipate energy efficiently reduces potential damage, making these materials ideal for innovative engineering solutions. This phenomenon explains the observed decrease in compression as the content of MWCNTs increases, aligning with findings from previous studies. For instance, Lu et al. [43] examined the impact of varying amounts of carbon black and silica, both separately and in combination, on polysulfide sealant. They found that as the amount of carbon black increased to 60 phr, compression decreased proportionally. Carbon black serves as a cross-linking agent, enhancing the density of cross-links and ultimately reducing compression. Similarly, Thomas et al. [44] investigated the effects of different fillers on a blend of nitrile butadiene rubber and natural rubber in an 80:20 ratio. They reported that adding 1 phr of MWCNTs reduced compression from 3.95% to 3.85% compared to the pure state. Additionally, Jalham et al. [45] studied energy dispersion during pressure and the improvement of material properties in composite rubber reinforced with silica sand, noting that compression increased with both particle size and filler amount.

4. Conclusion

In this study, we investigated the effect of different concentrations (i.e., 0, 0.25, 0.5, and 1 wt.%) of MWCNTs on the mechanical properties of silicone rubber. The composites were evaluated through tensile, compression, and compression set tests, and their fracture surfaces were analyzed using scanning electron microscopy (SEM). The results demonstrated that MWCNTs positively influenced the tensile and compressive properties of the RTV composite, with the optimal content being approximately 0.25 wt.% for tensile properties and 0.5 wt.% for compressive strength. Here are the key findings:

- The tensile results indicated that the addition of 0.25 wt.% MWCNTs led to an 11% increase in tensile strength, a 27% increase in elastic modulus.
- The presence of MWCNTs also enhanced the compressive strength at a strain of 0.6, increasing it from 3.2 MPa in pure silicone to 6.0 MPa at a weight percentage of 0.25%. However, increasing the MWCNTs content up to 1 wt.% gradually decreased the compressive strength of the silicone rubber.
- The results demonstrated that the incorporation of MWCNTs led to a decrease in the compression set of the silicone rubber.

Conflict of interest

The authors declare that there is no conflict of interest.

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