Poly dimethylsiloxane/carbon nanofiber nanocomposites: fabrication and characterization of electrical and thermal properties

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ABSTRACT
This article presents the fabrication and characterization of poly dimethylsiloxane/carbon nanofiber (CNF)-based nanocomposites. Although silica and carbon nanoparticles have been traditionally used to reinforce mechanical properties in PDMS matrix nanocomposites, this article focuses on understanding their impacts on electrical and thermal properties. By adjusting both the silica and CNF concentrations, 12 different nanocomposite formulations were studied, and the thermal and electrical properties of these materials were experimentally characterized. The developed nanocomposites were prepared using a solvent-assisted method providing uniform dispersion of the CNFs in the polymer matrix. Scanning electron microscopy was employed to determine the dispersion of the CNFs at different length scales. The thermal properties, such as thermal stability and thermal diffusivity, of the developed nanocomposites were studied using thermogravimetric and laser flash techniques. Furthermore, the electrical volume conductivity of each type of nanocomposite was tested using the four-probe method to eliminate the effects of contact electrical resistance during measurement. Experimental results showed that both CNFs and silica were able to impact on the overall properties of the synthesized PDMS/CNF nanocomposites. The developed nanocomposites have the potential to be applied to the development of new load sensors in the future.

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1. Introduction
Elastomers with high and reversible deformability have significant industrial applications. However, most elastomers have low initial modules and durability. Additional reinforcing phase is required to enhance their properties for practical use. Poly dimethylsiloxane (PMDS) is a non-conductive elastomer that has been under special focus for more than a decade for its flexibility and ease of fabrication. In general, silica particles are most commonly used materials for property reinforcement in elastomers, such as PDMS [1]. Recent development of polymer-based nanocomposites provides another approach to...
enhance critical material properties and even beneficial functionalities, such as load sensing. By dispersing strong nanoparticles and ‘smart’ materials in a polymer matrix, high-performance lightweight nanocomposites can be developed and tailored to individual applications. Various types of carbon-based nanoparticles, such as carbon nanofiber (CNF) [2], carbon nanotube [3], and graphene [4], have been incorporated within different polymer materials. In particular, CNFs have excellent electrical, thermal, and mechanical characteristics, as well as their simple incorporation and dispersion within polymers at low fabrication cost. Therefore, the development of CNF-reinforced nanocomposites have become an interesting research field for both material science and mechanical engineering.

Enhanced thermal properties due to inclusion of CNFs in polymers have been reported [5–7]. The dispersion quality of CNFs in polymer matrix and the purity of CNFs are able to significantly impact on the thermal properties of fabricated nanocomposites [5,6]. Hossain et al. recently reported the effects of dispersion conditions on both thermal and mechanical properties of CNF-reinforced polyester nanocomposites. At low CNF loading of 0.2 wt%, the decomposition temperature increased about 10°C and the glass transition temperature increased about 5°C. Roy et al. compared pristine and amine-modified CNFs on thermal properties of PDMS matrix nanocomposites [8]. Due to the depolymerization of PDMS in presence of amine functionalities, pristine CNF-reinforced PDMS nanocomposite showed higher decomposition temperature compared to amine-modified CNF-reinforced nanocomposites. More complicated theories, such as the role of tube-end transport and Kapitza contact resistances, have been reported in literature [9].

Incorporating limited amount of CNFs to polymers can significantly tailor the electrical conductivity of synthesized nanocomposites. Since virtually all of the electrical conductivity in CNF/polymer nanocomposites is through the network of CNFs, both good dispersion and high length to diameter ratio contribute to the high conductivity in nanocomposites. Reducing percolation threshold at low CNF loading and achieving sufficient electrical conductivity at high CNF loading are the two main goals for this type of research. In 2000, Goravev et al. [10] reported their fundamental discovery of the electrical conductivity of CNF-based nanocomposites. Complex time- and voltage-dependent changes in the electrical resistivity were reported because the CNF network adjusted under the influence of electrical current flow. Finegan and Tibbetts [11] reported extensive conductivity measurements for CNF/polypropylene and nylon composites. They found a percolation threshold of 3 vol% and were able to reach the electrical resistivity value as low as 0.15 Ω cm with about 20 vol% CNF loading. Xu et al. [12] were able to fabricate conducting CNF/vinyl ester nanocomposites, investigating a variety of compounding techniques including high-speed mechanical and shear mixing. Although percolation thresholds of 2–3 wt% were obtained, but resistivity values below about 10 Ω cm were not achieved even at high weight fraction, probably due to imperfect fiber dispersion. In 2012, Roy and Bhowmick reported the preparation and electrical property characterization of CNF-reinforced PDMS matrix nanocomposites [13]. Low percolation threshold was attained using amine-functionalized CNF-based nanocomposites. Using the same nanofiber loading, nanocomposites using functionalized CNFs were nearly 10 times more conductive than those using intrinsic CNFs.
In this article, CNFs were employed to enhance the thermal and electrical properties of PDMS due to their outstanding properties and low fabrication cost. The objective of this article is to characterize the electrical conductivity and thermal properties of the developed CNF-based nanocomposites for potential sensing applications. We first fabricated the PDMS/CNF nanocomposites by adjusting both the silica concentration in PDMS formula and the CNF loading. The nanocomposites were fabricated using a solvent-assisted ultrasonication method to ensure the uniform dispersion of CNF within polymer matrix. The morphology of fabricated nanocomposite was investigated using scanning electron microscope (SEM). The thermal stability was characterized using thermogravimetric (TGA), and thermal diffusivity was measured using a Hyperflash laser system. Finally, the electrical conductivity was tested using the four-probe method.

2. Methodology

2.1 Materials

Unless otherwise stated, all the following listed materials were used as received. Vinyl-terminated poly dimethylsiloxane (DMS-V31), methylhydrosiloxane-dimethylsiloxane copolymer (HMS-301), hexamethyldisilazane-treated silica filler (SIS6962.0), and high temperature platinum catalyst (SIP6832.2) constitute the PDMS formulation and were purchased from Gelest Inc. Tetrahydrofuran (THF ACS stabilized) was obtained from Macron Fine Chemicals. PR-24XT-LHT CNF was used as conductive nanomaterials and was received from Applied Science Inc. This specific type CNF was chosen due to their high electrical conductivity provided by the supplier [14].

2.2. Preparation of PDMS formulations

Different formulations of PDMS were prepared as polymer before mixing the CNF by adjusting the polymer (DMS-V31) to filler silica ratio. As shown in Table 1, three different DMS-V31 to silica weight ratios were chosen in this study. Once PDMS formulation was decided, the amount of CNF nanofillers in nanocomposites was calculated accordingly. Four CNF loadings (0 wt%, 3 wt%, 5 wt%, 8 wt%) were used for each type of PDMS formulation which resulted in a total of 12 unique nanocomposites. The resultant silica content varied from 0 wt% to 16.1 wt% depending on polymer to silica ratio and CNF loading. The aim was to find out the optimized formulation in terms of achieving higher conductivity, obtaining a flexible range of strain, and having reasonable viscosity for material processing and fabrication.

Table 1. PDMS formulations employed with different silica concentration.

| Base Polymer (DMS-V31) | Co-polymer (HMS-301) | Silica (SIS6962.0) | Catalyst (SIP6832.2) | Polymer to silica weight ratio |
|------------------------|----------------------|--------------------|----------------------|-----------------------------|
| 1                      | 100 parts            | 3 parts            | 0 parts              | 150–200 ppm                | 10:0                        |
| 2                      | 100 parts            | 3 parts            | 10 parts             | 150–200 ppm                | 10:1                        |
| 3                      | 100 parts            | 3 parts            | 20 parts             | 150–200 ppm                | 10:2                        |
2.3. Fabrication of nanocomposites

Pre-calculated amount of CNFs were placed in a glass beaker containing THF as solvent. For every gram of CNFs, 25 ml of THF was used. The prepared solution was subsequently dispersed using a low-viscosity-solution-based ultra-sonication technique. A high-intensity ultrasonic probe (Sonic Vibracell with Ti horn) with low amplitude (20%-22% of total power) was used to avoid breakage of CNFs and excessive heat generation during mixing. The CNFs were sonicated for 4 h in an ice bath and the temperature of the bath was kept under 4°C. In a separate container, DMS-V31 and silica of required weight ratios were mixed using a mechanical mixture at 1500 rpm for 10 min and then was added into the CNF–THF mixture for further sonication and dispersion. All other parameters of the sonication were kept the same except duration was for 2 h. Upon the uniform mixing of CNFs and PDMS polymer, excess THF was evaporated by keeping the solution in a silica oil bath for 8–10 h (depending on the CNF content) at 60°C. This specific temperature was chosen due to the fact that THF has boiling temperature of 66°C. To expedite the solvent removal process, the solution was continuously stirred using a magnetic stirrer at 350 RPM until the residual THF in the mixture was found to be less than 5 wt%. Further, the resultant viscous solution was placed in a vacuum oven at low vacuum regime (0.1 MPa) for 12 h to remove the trace of THF left in prepared materials. After this stage, the THF content was found to be less than 1 wt%. Following that the copolymer HMS-301 and the catalyst were manually mixed for 10 min. The mixture was again degassed in vacuum oven at room temperature for additional 30 min. The mixture was then casted into mold and cured inside a hot press at 80°C and under 1100 psi pressure for 3 h. Finally, the nanocomposites were post-cured at 150°C for 2 h. The entire manufacturing process is shown schematically in Figure 1.

2.4. Nanocomposites morphology characterization

Uniform dispersion of the CNFs inside polymer matrix is a key parameter which dictates the enhancement of both electrical and thermal conductivities of the nanocomposites. SEM images were taken using VEGA TESCAN instrument with 20 KV accelerating voltage at different locations of prepared materials to characterize the dispersion state. For better SEM observation, samples were fractured and coated with gold using a sputter coater.

Figure 1. Schematic of CNF/PDMS-based nanocomposites fabrication process.
2.5 Thermal property characterization

Thermogravimetric analysis of 12 types of samples was carried out in order to analyze the thermal stability using TA instruments Q-50 machine with a ceramic pan under nitrogen purge gas condition. All the samples were heated from 35°C to 950°C at the rate of 15°C/min. All the obtained data was processed using TA Universal Analysis software.

All the thermal diffusivity properties were measured using a LFA HyperFlash laser system. The schematic of the laser system is shown in Figure 2. The light beam heats the lower sample surface and an infrared (IR) detector measures the temperature change on the upper surface. Circular samples of the diameter of 12.5 mm and the thickness of 2 mm were first prepared and then coated with graphite spray. Each sample was measured 12 times to improve the accuracy. All the thermal diffusivity measurements were taken from 22°C to 25°C.

2.6 Electrical property characterization

To measure the volume conductivity and resistivity of the prepared nanocomposites, four-probe method was used to minimize the contact resistance at room temperature. Although two-probe method was also tested, it was noted that the contact resistance became dominant if two-probe method was employed. However, the contact resistance can be eliminated if four-probe method is used.

During all the experiments, resistance values were continuously recorded using Agilent multimeter and RS-232 data logger with a sampling frequency of 3 Hz. Each measurement lasted for at least 1 min and the electrical conductivity was averaged from three measurements.

3. Results and discussion

3.1 Morphology of prepared nanocomposites

SEM was used to observe the dispersion and distribution of CNFs within polymer matrix. The effects of silica filler to CNF distribution were studied by comparing SEM images.
taken from samples with different silica concentration and CNF loadings. As shown in Figure 3(a), CNF aggregation was clearly observed in the nanocomposites samples of 10:0 polymer to silica ratio and 3 wt% CNF loading. However, the distribution of CNFs in PDMS polymer was significantly improved when silica fillers were also used in the synthesized nanocomposites. As shown in Figure 3(b), in the sample of 10:1 polymer to silica ratio and 3 wt% of CNF loading, the CNFs were much more uniformly distributed in PDMS polymer. Since the CNF loadings in Figures 3(a) and 3(b) were the same, the improvement of CNF distribution in PDMS polymer was mainly caused by the introduction of silica fillers. Similar results were also observed in other fabricated nanocomposite samples. For example, in the nanocomposite of 10:2 polymer to silica ratio and 8 wt% of CNF loading, both good CNF and silica distributions were observed. The improvement of CNF distribution also led to the improvement of electrical resistance (see Section 3.3 for more details).

3.2 Thermal properties of the PDMS/CNF nanocomposites

3.2.1 Thermal stability

TGA tests were conducted to characterize the thermal stability of the cured nanocomposites. Figure 4 shows the effects of silica concentration and CNF loading on the thermal stability. The degradation of samples without silica took place in only one stage, as shown in Figure 4(a). However, the degradation of all the samples with both silica and CNF loading took place in two stages, as shown in Figures 4(b) and (c). The first decomposition temperature ($T_{d1}$) was around 500°C and the second decomposition temperature ($T_{d2}$) was around 690°C. $T_{d1}$ and $T_{d2}$, as well as the 5 wt% loss temperature ($T_{5\%}$), are summarized in Table 2. There is no report on which parts (bonds) decompose at the two different stages yet. However, there exists a relationship between bonding intensity and the decomposition, i.e. a strong bond has a corresponding higher decomposition temperature, while a weak bond has a lower decomposition temperature. Typical bond energies of C–C, C–O, C–H, and Si–Si are 349, 370, 337, and 327 kJ/mol, respectively [15,16]. The single bonds of C–C, C–O, C–H, and Si–Si are susceptible to chain scission during thermal degradation and act as weak links, which correspond to the decomposition of PDMS polymer at $T_{d1}$. The further decomposition at $T_{d2}$ can be

Figure 3. SEM micrographs of synthesized nanocomposites: (a) 10:0 polymer to silica weight ratio 3 wt% CNF; (b) 10:1 polymer to silica weight ratio 3 wt% CNF; (c) 10:2 polymer to silica weight ratio 8 wt% CNF.
attributed to the degradation of Si–O bond, where the Si–O bond energy is 798 kJ/mol \[15,16\].

In the nanocomposites, the CNF loading also affect the \(T_d1\) and \(T_d2\) of the nanocomposites reinforced with CNFs. When no silica concentration was included in PDMS formulation, the influence of CNF loading was significant. The variation of \(T_d1\) was about than 110°C for nanocomposites with no silica concentration. As silica concentration increased, the variation of \(T_d1\) was about 60°C and 30°C, respectively, for nanocomposites with 10:1 silica concentration and those with 10:2 silica concentration. The influence of CNF on \(T_d2\) was not as large as those to \(T_d1\), which is less than 30°C for all the tested samples. It is worth noticing that \(T_d1\) decreases slightly with the increase of

**Figure 4.** TGA curves of samples with (a) 10:0 silica (b) 10:1 silica (c) 10:2 silica ratios with respect to base polymer DMS-v31.

**Table 2.** Thermal stability of PDMS/CNF nanocomposites.

| Polymer to silica weight ratio | CNF wt% | Temperature of 5 wt% loss (°C) | \(T_d1\) (°C) | \(T_d2\) (°C) |
|-------------------------------|---------|-------------------------------|--------------|--------------|
| 10:0                          | 0       | 483                           | 573          | N /A         |
|                               | 3       | 495                           | 691          | N /A         |
|                               | 5       | 497                           | 689          | N /A         |
|                               | 8       | 516                           | 688          | N /A         |
| 10:1                          | 0       | 456                           | 435          | 696          |
|                               | 3       | 502                           | 509          | 697          |
|                               | 5       | 494                           | 491          | 693          |
|                               | 8       | 506                           | 494          | 690          |
| 10:2                          | 0       | 454                           | 477          | 710          |
|                               | 3       | 490                           | 504          | 682          |
|                               | 5       | 501                           | 511          | 692          |
|                               | 8       | 501                           | 522          | 688          |
CNF loading. This slight decrease of decomposition temperature is attributed to the increased free volume of nanocomposites upon the increased CNF loading, which is consistent with the results of fumed silica particles in glassy amorphous poly(4-methyl-2-pentyne) reported in the literature [17].

3.2.2 Thermal diffusivity

The thermal diffusivity in the through-thickness direction of the developed PDMS/CNF nanocomposites was measured using a laser flash method. The principle of the laser flash method is to irradiate the front side of a specimen using a short energy pulse which is provided by a laser, and record the subsequent temperature rise on the rear side of the specimen using an IR detector [18]. From the shape of the temperature–time curve of the rear side and the specimen thickness, the thermal diffusivity of the specimen can be determined. Due to the transparent nature of the PDMS specimens, they had to be coated prior to testing to ensure absorption and emission at the front and rear faces, respectively: graphite spray was used in these experiments.

The directly measured properties of thermal diffusivity are presented in Figure 5. Scatter bars show the level of repeatability of results, at the 95% confidence level, the values being obtained from 12 repeated tests. Less than 3% of variation was observed from all the measurement which indicated the reliability and repeatability of the experiments. Both CNF and silica loading in PDMS/CNF nanocomposites have impact on the thermal diffusivities. The silica effect on thermal diffusivity can be clearly seen by comparing the samples without any CNF. The thermal diffusivity of such samples increased from 0.12 mm$^2$/sec to 0.14 mm$^2$/sec (increased by 17%) when silica loading increased from 10:0 to 10:2 by weight ratio. In addition, the CNF effect on thermal diffusivity can be observed by comparing the samples without any silica loading. The thermal diffusivity of such samples increased from 0.12 mm$^2$/sec to 22 mm$^2$/sec (increased by 84%) when CNF content increased from 0 wt% to 8 wt%. Since intrinsic CNF is highly thermal and electrical conductive nanoparticle, it is reasonable to see more significant change of thermal diffusivity when more CNFs were dispersed in nanocomposites. In the future, the content of CNF and silica can be adjusted if the thermal

![Figure 5. Thermal diffusivity of the cured nanocomposites measured by laser flash method.](image)
diffusivity properties of PDMS/CNF nanocomposites needed to be tailored and controlled.

3.3 Electrical properties of the PDMS/CNF nanocomposites

The electrical resistance of each nanocomposite sample was first measured using four-probe method and the electrical resistivity and conductivity were calculated using Equations (1) and (2).

\[ \rho = \frac{RA}{L} \]  
\[ \sigma = \frac{1}{\rho} \]

where \( R \) is the electrical resistance measured from the rectangular nanocomposite sample using four-probe method; \( L \) is the length between the two probes in the middle; \( A \) is the cross-sectional area of the rectangular sample, and \( \sigma \) is defined as the electrical conductivity.

All the nanocomposite samples with different silica and CNF concentrations were cut into rectangular shape and four aluminum probes were inserted into the sample before measuring electrical resistance. Table 3 showed the electrical resistivity and volume conductivity of the nanocomposite samples. The electrical resistivity of nanocomposite with 10:1 silica concentration and 3 wt% CNF was significantly higher than the other samples. Therefore, it is reasonable to expect the percolation of nanocomposite with 10:1 silica should be slightly lower than 3 wt% CNF. The percolation for other two silica concentration was not observed. As listed in Table 3, both silica and CNF concentration can improve the electrical conductivity of the cured nanocomposites. For example, the nanocomposites with 8 wt% CNF and no silica concentration showed about five times higher conductivity compared to the samples with 3 wt% CNFs. The similar trend was also observed in nanocomposite samples with high silica concentrations. Compared to silica, CNF has more significant effects on the electrical conductivity of the developed nanocomposites because of the superior electrical conductivity of intrinsic CNF. The observed trend of electrical properties can be useful while designing the formation of new PDMS/CNF nanocomposites.

| Table 3. Electrical resistivity and volume conductivity of manufactured nanocomposites. |
|-------------------------------------------------
| | Polymer to silica weight ratio 10:0 | Polymer to silica weight ratio 10:1 | Polymer to silica weight ratio 10:2 |
| | 3 % CNF | 5% CNF | 8% CNF | 3% CNF | 5% CNF | 8% CNF | 3% CNF | 5% CNF | 8% CNF |
| Resistivity (ohm – m) | 0.159 | 0.059 | 0.032 | 24.1 | 0.031 | 0.012 | 0.148 | 0.032 | 0.012 |
| Volume Conductivity (siemens/m) | 6.312 | 17.178 | 31.733 | 0.042 | 32.631 | 80.919 | 6.760 | 31.311 | 83.138 |
4. Conclusion

This article presented the preparation, fabrication, and characterization of a unique set of PDMS/CNF nanocomposites by adjusting the silica concentration in PDMS formulation and CNF loadings in nanocomposites. A solvent-assisted dispersion method was used during the fabrication, providing good dispersion of CNF within PDMS polymer matrix. SEM was employed to determine the dispersion of CNF in nanocomposites. The thermal and electrical properties of the developed nanocomposites were experimentally characterized. A laser flash method was used to characterize the thermal diffusivity. The results showed that CNF showed stronger influence than silica to the thermal diffusivity properties. A TGA system was employed to characterize the thermal decomposition of each nanocomposite. Two-stage decomposition was recorded if the nanocomposites consisted of silica content. The electrical resistivity and conductivity were characterized using four-probe method to eliminate the effects of contact resistance during measurement. The percolation of nanocomposite with 10:1 silica concentration was observed at 3 wt% CNF concentration. The characterized PDMS/CNF nanocomposites can be useful for future design of CNF-enhanced nanocomposites.

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