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Faculty of Mechanical Engineering, Universiti Teknikal Malaysia Melaka (UTeM), Durian Tunggal, Melaka, Malaysia Email: sivakumard@utem.edu.my **Abstract:** This study aims to determine how the incorporation of carbon nanotube (CNT) fillers can enhance the mechanical and tribological properties of silicon rubber (SiR). The nanocomposite is created by combining the materials in a three-roll mill, compressing the mixture at 170°C, and then curing it for four hours at 200°C. The tensile strength improves by up to 3% for every 0% to 7% increase in the CNT weight percentage, after which it remains constant. When the load is 3% CNT, the coefficient of friction (COF) is at its lowest, and it declines as the load increases. There is a suggestion that the effect of heat and the aggregation of nanoparticles is responsible for the increase in COF that happens as the fraction of CNTs increases. Researchers found that a change in the sliding velocity affected both the coefficient of friction and the specific wear rate.

Keywords: silicon rubber; carbon nano tubes; CNT; wear; friction; mechanical properties; nanocomposites; morphology; rollmixing; compression moulding.

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1 Introduction

Silicon rubber (SiR) possesses superior thermal stability, chemical resistance, and flexibility when compared to other polymeric materials. Because of these benefits, SiR is being utilised in a wide variety of industries, including the automotive, aerospace, and medical sectors, amongst others. Various fillers, including nanoscale conducting particles, are used in experiments to investigate various characteristics of SiR. Carbon black (CB) (Jani et al., 2017), carbon black-carbon nanotube hybrid filler (Song et al., 2020), silver-plated glass microspheres (Yang et al., 2020, 2021), nickel-coated carbon fibre (Wang et al., 2014), carbon fibre fabric/Ni-Fe/CNT (Luan et al., 2021), MWCNT/Fe₃O₄ (Yang et al., 2019) and carbon back/nanographite (Jeddi et al., 2019). It has also been observed that the inclusion of nanofillers can improve the material's mechanical properties (Lin et al., 2020; Kumar et al., 2019, 2021b; Diani and Gall, 2006). In spite of tremendous interest, uniform dispersion of CNT in polymer is still a challenge. Polysilsesquioxane-modified CNTs (CNTs-PSQ) obtained from click chemistry reaction greatly improves the dispersion of CNT (Han et al., 2022). The mechanical, thermal and electrical conductivity performance of SiR with the addition of carbon-based nanofillers is reviewed in detail (Kumar et al., 2021a). In addition to its mechanical performance, the tribological analysis of SiR composite is also an essential aspect of its development. The longevity of the product is ultimately determined by the tribological performance of the SiR, and the restriction of the SiR is that it has a low wear resistance, which prevents it from being used for a variety of various applications. When compared to pure silicon matrix, the mechanical performance of nickel in micro and nano form that was employed in silicon matrix exhibited significant improvement (Song et al., 2013). It was discovered that SiR reinforced with fumed silica and precipitated silica shows an obvious increase in the mechanical performance. On the other hand, fumed silica demonstrated better wear performance than precipitated silica (He et al., 2018). The mechanical and tribological properties of nitrile rubber that has been reinforced with expanded graphite and made via one of two distinct processes, namely mechanical blending or latex compounding, are investigated in this study. It was discovered that the rate of wear might be reduced by increasing both the loading and the sliding velocity (Wang et al., 2012). The mechanical and wear performance of styrene butadiene rubber that has been reinforced with two different types of graphene nanosheets – one that has been functionalised and one that has been fluorinated – is being studied. The findings demonstrated a decline in the material's antifriction characteristics (Wu et al., 2017). Zhong et al. (2019) have tested the mechanical and tribological performance of graphene-silicon rubber. Tribological analysis of silicon rubber reinforced with liquid modified graphene oxide is studied experimentally as well as using artificial neural network. Using the multilayer perceptron, experimental values are used to train the ANN to predict the tribological properties (Sarath et al., 2022). The surface erosion property of silicon rubber under the standards voltage is quantified using the FTIR and SEM analysis. The surface erosion in silicon rubber is more when positive DC voltage is applied as compared with the AC voltage (Patel and Patel, 2022).

The abrasive wear resistance of silicone reinforced with manganese was significantly increased; nevertheless, the material's mechanical performance was significantly reduced (Mrówka et al., 2021). During mechanical and tribological testing, natural rubber that was reinforced with carbon black and Al₂O₃ performed significantly better than unreinforced natural rubber (Fu et al., 2013). The rubber material that is used for the fabrication of o-rings has been examined, and the tribological results have been promising (Szczypinski-Sala and Lubas, 2020). In addition, a degradation and tribological research of silicon rubber that has been subjected to a variety of biolubricants has been conducted by Farfán-Cabrera et al. (2018). The frictional performance of phenyl silicon rubber that has been strengthened with nanoscale CeO_2 and graphene is being investigated in service. It has been noticed that the coefficient of friction reduces as the load increases (Han et al., 2020). The effect of applied load, temperature and sliding speed on tribological performance is evaluated for silicon rubber reinforced with fumed silica (Sarath et al., 2022). A review on suitability of CNT for tribological application is also presented considering the effect of different parameters (Gu et al., 2022). The suitability and reliability of pin on disc testing machine is evaluated and it is found to good estimates of the coefficient of friction (Cristino et al., 2010).

After conducting a scan of the relevant literature, it was discovered that several kinds of rubbers, combined with a variety of fillers, are utilised in mechanical and tribological research. On the other hand, to the best of the authors' knowledge, there is no article that discusses the tribological performance of a SiR/CNT nanocomposite that was synthesised utilising the melt blending method. Also, the standard equipment that are utilised for the manufacturing of rubber products, such as roll mixing and compression moulding, are the sole machinery that are used for the manufacturing of the samples for this study. Because of this article's one-of-a-kind quality, which also contributes to its commercial viability and ease of adaptability.

2 Manufacturing and testing of material

2.1 Materials information

Krupa Chemicals in Pune, India is the company that provides the SiR of grade SH5060U and the peroxide-based accelerator known as Dicup-40 (DCP). Ad-Nano Technologies Private Limited, located in Shimoga, India, is the supplier of carbon nanotubes (CNT) that have a purity level of 99%. CNTs have an inner diameter of 5–10 nm, an outer diameter of 10–30 nm, and a length of greater than 10 nm, respectively. The specifics of the material are provided in Table 1.

Property	Silicon rubber (SH5060 U)	CNT
Specific gravity (g/cc)	1.15	1.6
Tensile strength, ultimate	5 MPa	63 GPa
Elongation (%)	500	5
Hardness, shore	50 (A)	70 HRC

Table 1 Properties of the matrix and filler in their provided forms from a mechanical standpoint

2.2 Composite manufacturing

Compounds of silicon rubber and dicumyl peroxide (DCP) are produced by combining various weight fractions of carbon nanotubes (CNT), including 0%, 1%, 3%, 5% and 7%. These compounds will be referred to as SRC1, SRC2, SRC3, SRC4, and SRC5, respectively. The manufacturing process is depicted in a schematic form in Figure 1, and Table 2 provides information regarding the weight percentage of filler and curing agent that is combined with SiR. On a 2-roll mixing machine, the silicon rubber and CNT are thoroughly roll mixed for around 20 minutes to create a homogenous mixture. The mixture is then subjected to curing in a hot press machine (make: SANTEC, capacity: 30 ton) for a period of five minutes at a temperature of 170°C and a moulding pressure of 50 bars. After that, it is placed in a hot air oven (brand name: Athena Technology; model name: ATAO-3 S/G) and baked to a temperature of 200°C for four hours.



Figure 1 Illustration of composite manufacturing route (see online version for colours)

Post Curing in hot air oven

Compression molding

Table 2	Weight percentages	of filler and curing agent followed	in nanocomposite making
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Type/grade	SRC1	SRC2	SRC3	SRC4	SRC5
CNT	0	1	3	5	7
DCP	2	2	2	2	2

2.3 Characterisations

The mechanical, tribological, thermal, and electromagnetic interference suppression evaluations are carried out on a variety of materials. Additionally, FESEM and XRD studies are carried out in order to verify the distribution of CNT and get insight into the development of the composite. The next sections will cover the specifics of each of the characterisations discussed previously. The technique of X-ray diffraction, often known as XRD, is used to investigate the development of a composite and to verify the blending of its constituent parts. The Rigaku Miniflex 600 XRD equipment is used for the XRD testing, and x-ray Cu K α radiations with a wavelength of 0.154 nanometres are used. The measurement is taken at scattering angles ranging from 20 degrees to 80 degrees. A VEGA3 TSCAN machine with ultra-high resolution is used to do the scanning electron microscopy investigation. For the tension test [shown in Figure 2(a)], samples are cut according to ASTM D 412, and the tear test [shown in Figure 2(b)] adheres to ASTM D 624. The ASTM D2240 standard is followed in order to assess the Shore D hardness of the material. A strain rate of 450 mm/min is maintained at all times. Atmospheric conditions are used for conducting tensile tests at a strain rate of 450 mm/min.





2.4 Friction and wear studies

As shown in Table 3, it was chosen between three distinct degrees of weight and sliding speed and considered two distinct levels of sliding distance. Experiments are carried out with the assistance of a computerised pin-on-disc wear testing machine, in which discs rotate at a predetermined speed while a pin rubs on the surface of the disc. The rectangular pin specimens with a cross section that measures 10×10 mm and a height that measures 30 mm is studied. Discs are made out of the 60 HRC EN-31 material. Every experiment is run with the ambient conditions of temperature and humidity.

The weight of the sample was determined before the experiment was carried out. The specimen's weight is measured once more after it has been subjected to examination for the predetermined levels of the various parameters. When taking the readings, an

analytical weighing balance that has an accuracy of up to four decimals is utilised. The friction coefficient (μ) is calculated using equation (1).

$$\mu = F_f / F_n \tag{1}$$

where F_f is the frictional force and F_n is normal force due to applied load. Also, specific wear rate (SWR) is given by equation (2).

 $SWR = Weight \ loss/(Density \times Normal \ load \times Sliding \ distance)$ (2)

Parameter Level Composition SRC1 SRC2 SRC3 SRC4 SRC5 Load (N) 10 20 30 Sliding speed (m/s) 2 4 6 Sliding distance (m) 1,800 3,200

 Table 3
 Details of experimental parameters an their levels followed in wear studies

3 Results and discussion

3.1 X-ray diffraction studies

The XRD graph for MWCNT, SRC1, and SRC5 samples may be found depicted in Figure 3. It can be seen that the MWCNT has a higher crystallinity, as it displays a sharp peak at 25.38°. When looking at the spectrum of the SRC5 composition, the same peak at 25.38° can be seen, albeit with less strength. This observation justifies the independent presence of MWCNT particles in the SRC5. The crystalline structure of MWCNT remains unaffected by the imposition of SiR polymer molecules, which instead leads to the creation of a polymer composite that is reinforced with MWCNT.

Figure 3 XRD patterns of pure SR, CNT powder and SRC-4 composition (see online version for colours)



3.2 Mechanical characterisation

3.2.1 Tensile strength

It has been discovered that an increase in the concentration of MWCNT results in an increase in the tensile strength of nanocomposites comprised of SiR and MWCNT (Figure 4). Tensile strength was measured at 5.39 MPa for SRC1, while this increased to 14.74 MPa for the SRC5 composition. When compared to the pure SiR, this rise in tensile strength is 173% greater. According to the findings of the SEM analysis, the dramatic increase in tensile strength can be attributed to the increased density of MWCNT filler in the SR3 composition. Because of the increased density, the interatomic distance between atoms is decreased, which ultimately results in strong interatomic bonds between the SiR and the MWCNT.





3.2.2 Tear strength

The tear strength demonstrates rising trend as it transitions from SRC1 to SRC2 composition (Figure 4). The tear strength improves to 3.05 MPa for SRC2 composition which is 16.41% greater than pure SiR. There is no appreciable improvement in tensile strength between SRC2 and SRC3 compositions. With each additional rise in the MWCNT percentage, the ripping strength keeps growing and it reaches its maximum value of 3.57 MPa for the SRC5 composition.

3.2.3 SEM analysis of fractured surface

After the specimens are tensile fractured, a SEM analysis is carried out to better understand the bonding between the SiR and the MWCNT. Figure 5 displays a SEM image of MWCNT powder on the left, while Figures 5(b) through 5(f) show SEM images of the broken surfaces of samples SRC1 through SRC5 on the right.

SRC1 is a pure form of rubber that has a smooth exterior. Figure 5 make it quite easy to see that MWCNT has significantly risen in density [Figures 5(c)-5(f)]. The enhanced concentration of MWCNT can be attributed for dramatic improvement in both the tensile strength and the tear strength of the SRC2 composition. This can be seen in the SEM image as well, and it occurs more frequently as the density of MWCNT increases. Further improvement in the characteristics has been noticed for the SRC4 composition, which can be seen to be corroborated by the SEM image. This improvement is attributed to the higher MWCNT content and its uniform distribution. For the compositions SRC4 and SRC5 there is very little but not appreciable improvement in the in the attributes, this could be the result of voids present, as seen in the SEM images. This also refers to the beginning phase of the agglomeration process for MWCNT, which occurs as the concentration of the material increases.

Figure 5 SEM images of (a) CNT powder and fracture surfaces of (b) SRC1 (c) SRC2 (d) SRC3 (e) SRC4 and (f) SRC5 composition (see online version for colours)



The major factor that demonstrates an improvement in the tensile properties of the nanocomposites is improved adhesion between the matrix and filler.

3.3 Tribological analysis

3.3.1 Effect of load and sliding velocity

The tests are conducted at constant sliding speed and sliding distance to understand how typically normal loading affects the COF and SWR, While the effect of sliding velocity can be comprehended by keeping the load and sliding distance constant. Figure 6 displays the trend of COF and SWR under the effect of normal loading and sliding velocity. The

figure is broken down into four sections: Figures 6(a)-6(d). The COF and SWR are found to be at their peak for a loading of 10 N, and they begin to decline as the load is raised [Figures 6(a)-6(b)]. The average reduction in the COF is 18% from 10 N to 20 N loading and 31% from 20 N to 30 N loading. However the SWR experiences a dramatic reduction of 66% from 20 N to 30 N loading. A similar trend of COF and SWR is observed at constant sliding speed, however the percentage reduction in COF and SWR is 16% and 30%, respectively. This is because the lighter loads are not adequate to disrupt the interfacial bonding and hence does not result in the formation of chips. This causes an increase in the amount of friction between the surfaces that are in contact with one another.

Figure 6 Trends of (a) coefficient of friction (COF), (b) SWR at constant load (sliding speed 2 m/s, sliding distance 1,800 m) and (c) coefficient of friction (COF) (d) SWR at constant sliding velocity (20 N and 1,800 m) (see online version for colours)



At a constant loading, the COF and SWR tend to decrease as the CNT concentration rises up to the point when the SRC3 composition is reached, but then they sharply rise again for the SRC4 and SRC5 compositions. A trend analogous to this one can be seen when sliding at a constant velocity. When the sliding speed is increased, the COF and SWR both sink. As it degrades, rubber has a predisposition toward the formation of a layer that acts as a lubricant. Therefore, when the sliding velocity is lower, there is a greater amount of contact time, and this results in the production of the lubricating layer (Bahadur and Sunkara, 2005; Schwartz and Bahadur, 2000), which inhibits the direct contact of rubbing surfaces. One possible explanation for this is that there has been an increase in the

concentration of CNT. It should come as no surprise that the SRC3 composition achieves the best tribological performance when compared to the other compositions.

3.3.2 SEM analysis of worn surface

SEM pictures of all specimens after pin on disc testing are shown in Figure 7. Hysteresis and adhesive friction are two mechanisms of rubber material wear that occur naturally (Tabor, 1960), (Greenwood and Tabor, 1958). To put it another way, the sliding friction action causes the rubber material to deflect and this results in the hysteresis loss. Due to the heat created, adhesion between the contacting surfaces increases, which causes wear. The poor mechanical properties of the SRC1 sample are evident in Figure 7, where it is shown to be more sensitive to adhesive friction [Figure 7(a)]. There is a lack of adhesion between the matrix and filler, which causes MWCNT to chip off of the surface. The rubber also deforms and adheres to the disc when it is subjected to loads and sliding motions. The surface has been scraped away at these stuck connections. These chipped off particles form a layer that keeps the touching surfaces from sticking together, for SRC2. MWCNT helps in lowering the contacting area, resulting in decreased friction for SRC3 formulation. Lubricating film formation is affected when MWCNT's weight percentage is greater than 3%. These sticky connections result in a material that experiences a dual friction mechanism (Sarath et al., 2020). Adhesive wear is the fundamental and most basic process of wear based on SEM images.

Figure 7 SEM images of wear surface for (a) SRC1, (b) SRC2, (c) SRC3, (d) SRC4 and (e) SRC5 composition (see online version for colours)



4 Conclusions

Using compression moulding, the SiR/MWCNT nanocomposite is produced and tested for mechanical and tribological performance. Filler loading has been proven to boost tensile and tear strength with 14.74 MPa and 3.57 MPa, respectively, with SRC5 composition having the best results. However, nanoparticle dispersion is mostly responsible for improving rubber molecule mobility under strain. As the progressive stress is increased, the nanoparticle addition greatly helps to postpone the onset of tensile and tear fracture failures. The CNT nanoparticles in the rubber also make it more resistant to dynamic loading, such as sliding wear. At constant loading the COF and SWR in total is decreased by almost 49 and 93% with loading increased from 10 to 30 N, respectively. The stronger mechanical bond between the CNT and rubber base is shown via electron imaging adhesion wear. SRC3 composition, on the other hand, exhibits the best tribological features. Rubber's frictional responsiveness and wear resistance improved significantly as a result of this investigation.

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