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RESEARCH

CNTs, Al_2O_3 and SiO_2 Reinforced Epoxy: Tribological Properties of Polymer Nanocomposites

M.A. Ramadan[a], R. Reda[b]

[a] *Mechanical Engineering Department, Faculty of Engineering, Helwan University, Helwan, Egypt,*
[b] *Mechanical Engineering Department, Faculty of Engineering, Suez University, Suez, Egypt.*

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Corresponding author:

M.A. Ramadan
Mechanical Engineering Department,
Faculty of Engineering,
Helwan University, Helwan, Egypt.
E-mail: eng_mohamedramadan@yahoo.com

A B S T R A C T

The present work studied the effect of filling epoxy matrix by different types and concentrations of nanoparticles on the friction and wear behaviors. Various concentrations (0.2 %, 0.4 %, 0.6 %, 0.8 % and 1 wt.%) of multi walled carbon nano tubes (MWCNTs), aluminum oxide (Al_2O_3), and silica (SiO_2) nanoparticles were used to reinforce epoxy matrix. These epoxy nanocomposites are widely used as indoor flooring tiles in schools, boutiques, hospitals, offices, conference rooms, homes, trade fair stands and homes for the aged. Experiments involved sliding of the epoxy nanocomposite specimens against rotating steel disc at dry sliding condition. Experiments were carried out using a test rig of pin-on-disc, designed and manufactured for the test. The friction force was measured using load cell which connected with a digital screen to detect the friction force. All experiments were done at room temperature and carried out at constant normal load (7 N), constant speed (0.93 m/sec) and constant running time (300 seconds). The worn surfaces were investigated with back scattered scanning electron microscopy (SEM). Based on the observations in the present work, it was found that addition of the tested filling nanoparticles-have greatly affected the friction and highly improves wear resistance.

1. INTRODUCTION

Polymer nanocomposites have now a wide spread applications in a number of tribological applications including such components as bearings, gears, cams, seals, vacuum pumps [1-3]. The field of tribology of the nanocomposite materials have grown and developed over the last two decades. The most used type of polymer nanocomposites are based on matrix of epoxy resins or on thermoplastics with high thermal resistance [4-6].

The incorporation of nano size ceramic particles of oxides, carbides or nitrides like TiO_2, SiO_2, ZrO, SiC and Al_2O_3 to polymer matrices has led to better enhancement in wear resistance [1-13]. This is due to that the nanoparticles have high specific surface areas and high surface energy due to their small scales so they can react with macromolecular chains of polymers, chemically or physically, to enhance the interactions between the macromolecular chains after they are added to polymer [8].

The mechanisms which have been proposed to illustrate the improvement of tribological properties of polymers using nanoparticles of ceramic can be summarized as following, [1]: (i) increase of the mechanical resistance and thermal stability of the polymer-matrix nanocomposite with respect to the base polymer, (ii) formation of protective stable transfer films on the counter-face surface so the nanoparticles can fill the surface asperities and blend with wear debris, reducing surface roughness, and (iii)reduction of surface roughness and material removal from the nanocomposite due to the similar size of the fillers and the segments of the polymer chains. One of the potential advantages of nanoparticles with respect to micro-particles is that the nanoparticles would be less abrasive. In this way, it would be possible to reduce the surface damage caused by abrasion by reducing the size scale of the additives, from micro- to nano-size [2,13].

Carbon nanotubes (CNTs) are becoming one of the most relevant materials in nanotechnology and their exceptional properties are opening new fields in science and engineering [1,10,11]. CNTs nanocomposites have been developed aiming to produce new novel strong and light composite materials with excellent tribological properties. Previous studies on the tribological properties of CNTs were mostly done with CNT-containing composites [10-12,14]. CNTs can withstand repeated bending, buckling and twisting to build excellent nanocomposite matrices so they can show lower friction coefficient and wear rate compared with the pure substrate matrix [10-12]. Improvements in friction and wear properties were frequently observed due to the reinforcement supplied by high-aspect-ratio CNTs [14].

One of the key factors influencing the performance of the final nanocomposites material is the additive concentration [1,5,7,10,15]. In the current paper, the effect of addition of different types of nanoparticles, namely SiO_2, Al_2O_3 and CNTs with different concentrations to a matrix of epoxy resin on the friction and wear behaviors were studied.

2. EXPREMENTAL

Experiments were carried out using a pin-on-disc machine under ambient and dry conditions at room temperature**.** Pin-on-disc consists of an electric motor which provides rotational motion for the disc (140 r.p.m). The test rig was designed and manufactured to measure the friction coefficient between pin (test specimen) and disc surface. A steel disc of hardness (269 HB) was used as the counterpart. The disc size was 170 mm diameter ×10 mm thickness, the specimens were contacted the disc at track of 126.6 mm diameter, as shown in Fig. 1. The configuration of the tester was illustrated in Fig. 2. All experiments were carried out at constant normal load (7 N), constant sliding velocity (0.93 m/sec) and constant running time (300 seconds). The friction force was measured using load cell which connected with a digital screen to detect the friction force. The friction coefficient was calculated from the measured friction force divided by the normal load exerted on the pin by the testing machine's dead weight. The wear was measured by weight loss of specimen using an analytical balance of resolution 0.1 mg. Before the experiment, the surface of the specimens and the disc were rubbed with abrasive paper before final polish, and then cleaned with acetone. Each experiment was repeated three times to ensure the reliability and accuracy of the data.

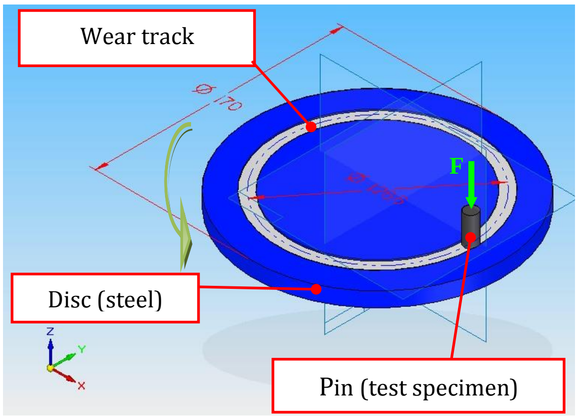


Fig. 1. Schematic illustration of pin-on-disc wear test.

Three types of nanocomposites were used in experiments :1. epoxy filled by nano particles of carbon nano tube, 2. epoxy filled by nanoparticles of silica and 3. epoxy filled by nano particles of alumina, in addition to epoxy free particles specimens for comparisons. Different concentrations of each nanoparticles

(0.2 %, 0.4 %, 0.6 %, 0.8 % and 1 wt.%) had been added to the epoxy resin. The CNTs used in this study were multi-walled MWCNTs, produced by Egyptian Petroleum Research Institute (EPRI). The specifications of these CNTs are average diameter 8.5 - 40 nm and average length 0.2 µm, as shown in Fig. 3. The purity of CNTs was higher than 95 wt.%.The average particle size of alumina was about 90 nm size and the average particle size of silica was 120 nm.

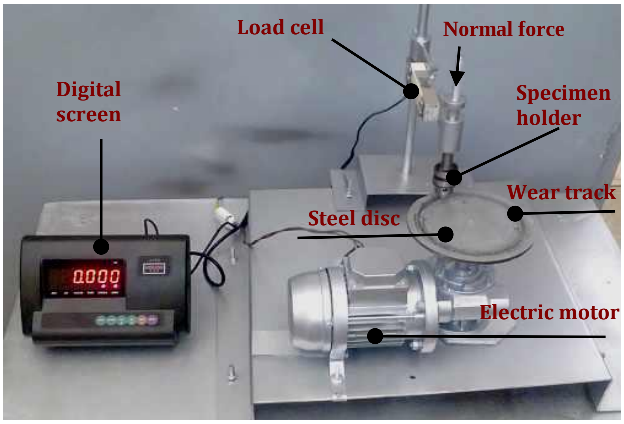
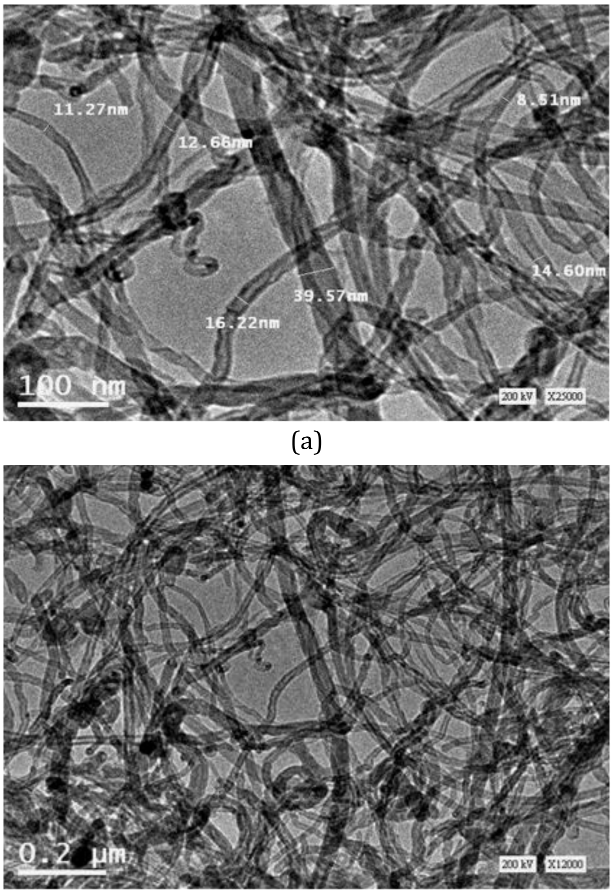


Fig. 2. Configuration of pin-on-disc machine.



(a)

(b)

Fig. 3. Specifications of multi walled CNTs: (a) average diameter and (b) average length.

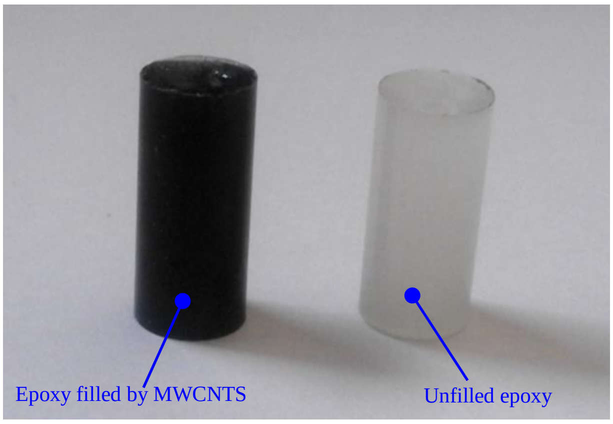


Fig. 4. Test specimens.

Regarding to specimen preparation, the nano particles were mixed with the epoxy resin in a plastic container and the mixture was then stirred manually for 5 min. Syringes without needle of dimension 9 mm diameter x 22 mm long were chosen as molds, to facilitate proper flow of materials into the mold and furthermore to easy eject the specimens by the piston of syringe which act as ejector. The mixture was then drawn by the syringe and left for 24 hours at the room temperature for curing in vertical position. After curing, the top nozzle of syringe was cut and specimen was easy removed from the molds by the ejector. The size of the specimen was 9 mm diameter x 20 mm length, as shown in Fig. 4.

3. RESULTS AND DISCUSSION

Friction and wear behaviors of epoxy filled by nanoparticles of CNTs, alumina and silica were investigated. The friction coefficient value recorded for each specimen was the average over the entire test. Figure 5 shows the effect of different concentrations (0.2 %, 0.4 %, 0.6 %, 0.8 % and 1 wt.%) of these nanoparticles on the friction coefficient values of the resultant nanocomposites. It is clearly shown that the highest values of friction coefficient were observed for epoxy filled by CNTs as compared with alumina and silica nanoparticles. This may be attributed to the high strength and hardness of CNTs. With increasing the CNTs concentrations, the value of friction coefficient decreases. This is attributed to formations of a smooth transferred film of carbon between the sliding surfaces which acts as a self-lubricant and reduces the friction coefficient value, as reported previously [14,16]. The results show that addition of CNTs up to 0.8 wt.% results in higher friction coefficient compared with pure epoxy resin and other epoxy nanocomposites.

With respect to epoxy filled by nano silica particles Fig. 5, it can be noticed that the friction coefficient decreases with increasing silica content. The friction coefficient deceases from about 0.6 to about 0.5. This may be due to the addition of appropriate size of silica nanoparticles which is favorable for lowering the friction coefficient, especially, when the size of silica in the composite is below 150 nm, as reported previously [15]. The friction coefficient of epoxy filled by alumina nanoparticles is also shown in Fig. 5. The behavior of this type of nanocomposite is different as compared with CNTs and silica nanocomposites. Low concentration of alumina (0.2 wt.%) resulted in the lowest value of friction coefficient of 0.53 as compared with other concentrations of Al_2O_3/epoxy nanocomposites. As the concentration of alumina exceeds 0.2 wt.% in the epoxy matrix, the value of friction coefficient increase up to 0.63 at concentration of 0.8 % of alumina. The increase in friction coefficient is attributed to the formation of coherent and adherent transfer film by the alumina nanoparticles on the contact surface [13]. At high concentration of alumina (1 %), friction coefficient decreases to 0.6 that is still lower than the unfilled epoxy.

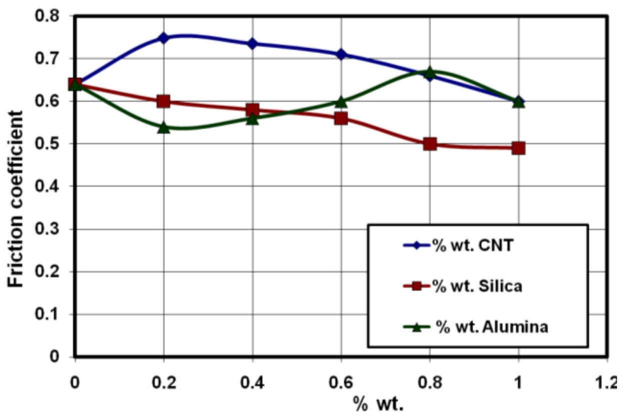


Fig. 5. The effect ofnanoparticles on the friction coefficient of epoxy nanocomposites.

The effect of different concentrations (0.2 %, 0.4 %, 0.6 %, 0.8 % and 1 wt.%) of carbon nano tubes (CNTs), aluminum oxide, and silica nanoparticles on the wear of the epoxy nanocomposites is shown in Fig. 6. For CNT/epoxy composite, it is clear that wear decrease with increasing the concentration of CNT. This may be attributed to the transferred film that deforms at the contact surfaces and decreases the wear. The value of wear obtained at the ratio of 0.2 wt.% CNTs, was about 0.25 of that of pure epoxy resin. According to these results, it can be concluded that CNTs provide different friction coefficient values (higher and lower than that of pure epoxy) with low weight loss as compared to pure epoxy that makes this type of nanocomposite (epoxy filled by CNTs) suitable for various applications. For example, the epoxy nanocomposites containing CNTs lower than 1 wt.%, can be used as friction materials which provide high friction coefficient and high wear resistance. The epoxy nanocomposites containing CNTs higher than 1 wt.%, can be used as bearing materials which provide low friction coefficient and high wear resistance.

For epoxy filled by nano silica particles, it can be noticed that the addition of 0.2 wt. % silica leads to a decrease in wear from 0.037 g to 0.0025g. Increasing the ratio of nano silica over 0.6 wt.%, the wear was in steady state as the silica content increases. Nano silica composites with 0.4 wt % have the lowest wear value. The improvement in wear properties of silica/epoxy composites is due to the contribution of the nano filler which enhances the strength and mechanical properties of the composites.

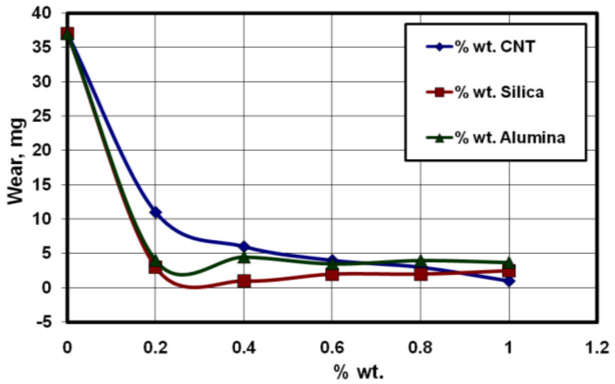
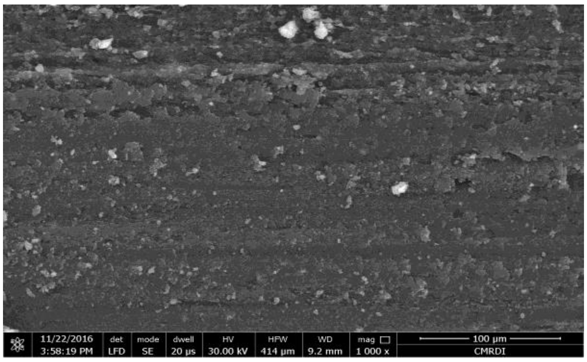


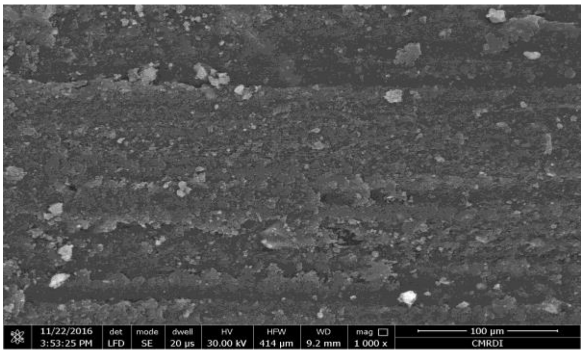
Fig. 6. The effect of nanoparticles on the wear of epoxy nanocomposites.

The similar trend was observed for epoxy filled by nano alumina particles as shown in Fig. 6. As the alumina content increases from 0 % to 0.2 wt.%, the wear decreases from 0.037 gm to 0.004 gm. When the ratio of nano alumina exceed 0.4 wt.%, the wear was in steady state as the alumina content increases. The improvement of wear resistance can be explained by the reinforcement of pure epoxy by high strength and hardness alumina nanoparticles. This improvement in the tribological behavior of Al_2O_3/epoxy composite is also may be related to the improved characteristics of the transfer film [13]. During wear process, alumina particles afford a lot of load and decrease the stress between epoxy matrix and counter part, therefore, the epoxy matrix is effectively protected and wear resistance of new composites is improved.

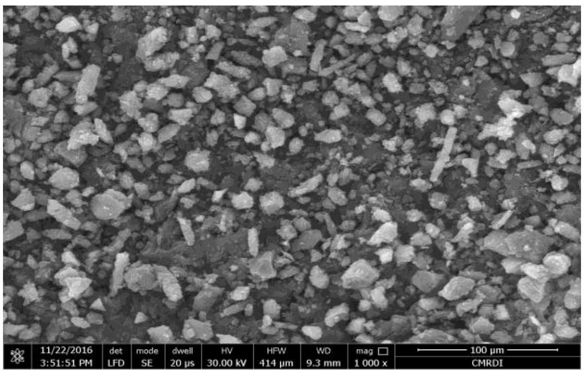
Back scattered scanning electron microscopy (SEM) was used to examine the worn surfaces of the tested specimens. SEM micrographs of selected specimens are illustrated in Fig. 7. All SEM micrographs of the surfaces of the nanocomposite samples after wear testing displays scratches, suggesting an abrasive sliding wear mechanism.



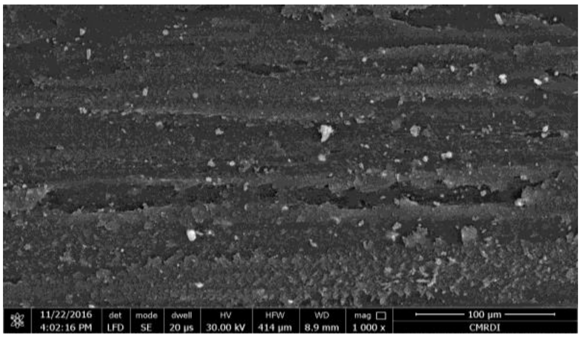
(a) Epoxy filled by 0.2 wt.% CNTs



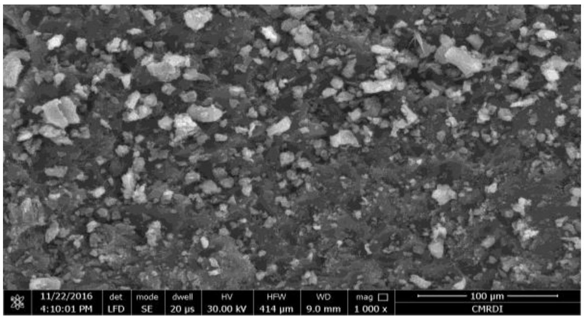
(b) Epoxy filled by 0.8 wt.% CNTs



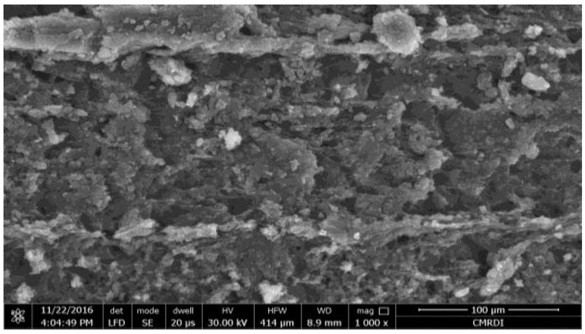
(c) Epoxy filled by 1 wt.% CNTs



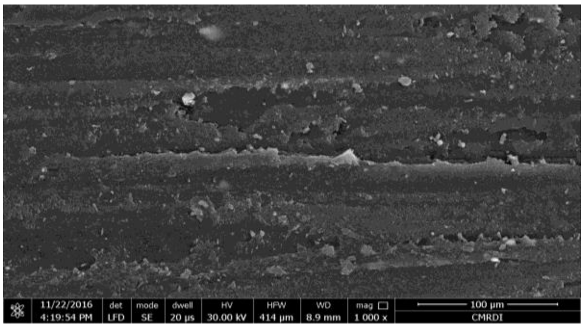
(d) Epoxy filled by 0.2 wt.% silica



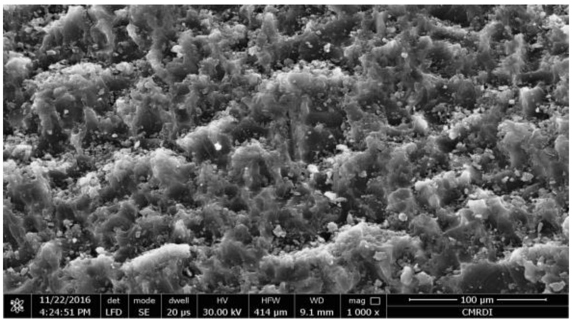
(e) Epoxy filled by 0.4 wt.% silica



(f) Epoxy filled by 0.6 wt.% silica



(g) Epoxy filled by 0.2 wt.% alumina



(h) Epoxy filled by 0.4 wt.% alumina

Fig. 7. Back scattered SEM images of the tested specimens after wear test.

For CNTs/epoxy composites, large amount of the epoxy fragments at the contact surface were observed for specimen containing 0.2 wt.% CNTs, as shown in Fig. 7a. This is associated with the drastic increase in weight loss (as shown in Fig. 6 at 0.2 wt.% CNTs). The SEM image of specimen containing 0.8 wt.% CNTs is shown in Fig. 7b, which reveals shallow abrasive grooves. As reported in [16], most of the friction surface of CNTs/epoxy composites seemed to be covered with the film-like layers, so the MWCNTs structure can be hardly observed. The MWCNTs that existed in the friction surface may be damaged and deformed during sliding, which resulted in film-like structure, namely transferred film. The resultant friction behaviors may be related to the smearing of transferred film over the contact area, which was expected to permit easy shear and then help to achieve a lubricating effect during sliding. The worn surface of specimen containing 1 wt.% CNTs is shown in Fig. 7c, which reveals rough worn surface. The SEM images of CNTs/epoxy composites are in agreement with the wear test results (Fig. 6).

The worn surface of epoxy filled by 0.2 wt.% silica is shown in Fig. 7d. SEM image shows deep wear tracks lead to high wear value as shown in Fig. 6 (at 0.2 wt.% silica). Shallow wear tracks were observed for epoxy filled by 0.4 wt.% Silica, as shown in Fig. 7e. It can be observed that increasing the amount of nano-silica particles content up to 0.4 wt.% had reduced the amount of fragments. When the silica content increases to 0.6 wt.% some fragments of epoxy were observed on the wear tracks, as shown in Fig. 7f. These tracks indicate the presence of hard particles during the sliding wear. During wear, when two bodies move relative to each other, plastic deformation occurs due to the plowing of nanoparticles on the wear surface. Once the friction induced strain exceeds the limit held by the strength of bonding between the nanoparticles and the matrix, the nanoparticles may break down to form scattered debris, the hardness and morphology of the debris then governs the wear mechanism [10].

Good wear resistance was observed for epoxy filled by 0.2 wt.% alumina. The SEM image reveals shallow wear tracks as shown in Fig. 7g. It is suggested to be a deformation-controlled wear mechanism, which is a characteristic of Al_2O_3 under mild wear conditions, as reported previously [10].The worn surface of specimen containing 0.4 wt.% alumina displayed severe matrix failure as shown in Fig. 7h. Furthermore, presence of micro-voids on the surface of specimen due to loosing of nanoparticles from the matrix during the sliding.

4. CONCLUSIONS

Based on the results of this study, the following conclusions can be drawn:

1. The addition of CNTs to pure epoxy strongly improves the wear resistance of epoxy nanocomposites and provides different friction coefficient values (higher and lower than that of pure epoxy). This makes epoxy filled by CNTs, suitable for various applications.
2. As the concentration of SiO_2 on SiO_2/epoxy composites increases, the friction coefficient decreases. On the other hand, increasing SiO_2 content up to 0.4 wt.% results in increasing the wear resistance of SiO_2/epoxy composites. Over 0.6 wt.% SiO_2, the weight loss reaches a constant value.
3. Addition of 0.2 wt.% Al_2O_3 has the lowest friction coefficient and weight loss as compared with other Al_2O_3/epoxy composites. The friction coefficient starts to increase with increasing alumina contents from 0.4 wt.% to 0.8 wt.% while the weight loss reaches a constant value.
4. The lowest values of wear were obtained by epoxy nanocomposites containing 1 wt.% CNTs, 0.4 wt.% silica and 0.2 wt.% alumina.

REFERENCES

[1] M.D. Bermúdez, F.J. Carrión, C. Espejo, J. Sanes and G. Ojados, *Tribology of Bulk Polymer Nanocomposites and Nanocomposite Coatings*. Tribology of Nanocomposites, Materials Forming, Machining and Tribology, Springer-Verlag Berlin Heidelberg, 2013.

[2] L. Chang, Z. Zhang, L. Ye and K. Friedrich, "Tribological Properties of Epoxy Nanocomposites III. Characteristics of Transfer Films", *Wear*, vol. 262, pp. 699-706, 2007.

[3] N.L. Mc-Cook, M.A. Hamilton, D.L. Burris and W.G. Sawyer, "Tribological Results of PEEK

Nanocomposites in Dry Sliding Against 440 C in Various Gas Environments", *Wear*, vol. 262, pp. 1511-1515, 2007.

[4] J.D. Souza and J.D. Reis, "A Thermomechanical and Adhesion Analysis of Epoxy/Al_2O_3 Nanocomposites", *Nanomaterials and Nanotechnology*, vol. 18, no. 5, pp. 1-7, 2015.

[5] T. Larsen, T.L. Andersen, B. Thorning, A. Horsewell and M.E. Vigild, "Changes in The Tribological Behavior of An Epoxy Resin by Incorporating CuO Nanoparticles and PTFE Microparticles", *Wear*, vol. 265, pp. 203-213, 2008.

[6] S.K. Ghosh, *On The Development of Novel Multifunctional Max Reinforced Polymers (MRPs) Matrix Composites*. Master of Science-dissertation, ProQuest LLC, 2017.

[7] G.W. Sawyer, K.D. Freudenberg, P. Bhimaraj and L.S. Chadler, "A Study on The Friction and Wear of PTFE Filled with Alumina Nanoparticles", *Wear*, vol. 254, pp. 573-580, 2003.

[8] L.H. Sun, Z.G. Yang and X.H. Li, "Study on The Friction and Wear Behaviour of POM/Al_2O_3Nanocomposites", *Wear*, vol. 264, pp. 693-700, 2008.

[9] T. Larsen, T.L. Andersen, B. Thorning and M.E. Vigild, "The Effect of Particle Addition and Fibrous Reinforcement on Epoxy-Matrix Composites for Severe Sliding Conditions", *Wear*, vol. 264, pp. 857-868, 2008.

[10] I. Ahmad, A. Kennedy and Y.Q. Zhu, "Wear Resistant Properties of Multi-Walled Carbon Nanotubes Reinforced Al_2O_3 Nanocomposites", *Wear*, vol. 269, pp. 71-78, 2010.

[11] Y. Liu and S. Kumar, "Polymer/Carbon Nanotube Nano Composite Fibers–A Review", *ACS Appl. Mater. Interfaces*, vol. 6, no. 9, pp. 6069-6087, 2014.

[12] C. Wang, T. Xue, B. Dong, Z. Wang and H.L. Li, "Polystyrene–Acrylonitrile–CNTs Nanocomposites Preparations and Tribological Behavior Research", *Wear*, vol. 265, pp. 1923-1926, 2008.

[13] N. Ai, S. Hussein, M. Jawad and I. AL-Ajaj, "Effect of Al_2O_3 and SiO_2 Nanopartical on Wear, Hardness and Impact Behavior of Epoxy Composites", *Chemistry and Materials Research*, vol. 7, no. 4, pp. 34-40, 2015.

[14] G. Yamamoto, T. Hashida, K. Adachi and T. Takagi, "Tribological Properties of Single-Walled Carbon Nanotube Solids", *Nanoscience and Nanotechnolgy*, vol. 8, pp. 2665-2670, 2008.

[15] S.Q. Lai, T.S. Li, F.D. Wang, X.J. Li and L. Yue, "The Effect of Silica Size on The Friction and Wear Behaviors of Polymid/ Silica Hybrids by Sol-Gel Processing", *Wear*, vol. 262, pp. 1048-1055, 2007.

[16] G. Yamamoto, M. Omori, K. Yokomizo, T. Hashid and K. Adachi, "Structural Characterization and Frictional Properties of Carbon Nanotube/Alumina Composites Prepared By Precursor Method", *Materials Science and Engineering B*, vol. 148, pp. 265-269, 2008.