

DRY SLIDING WEAR BEHAVIOUR OF CARBON NANOTUBE/ALUMINA/EPOXY HYBRID NANOCOMPOSITES

DRSNA OBRABA HIBRIDNIH NANOKOMPOZITOV, SESTAVLJENIH IZ OGLJIKOVIH NANOCEVK, ALUMINIJEVEGA OKSIDA IN EPOKSIDNE SMOLE

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In the field of materials science, polymer composites have been extensively used in various industries such as marine, automotive, aerospace, sports and other industries due to their good dimensional stability and excellent structural properties. In this present research investigation, epoxy served as the polymer material, while multi-walled carbon nanotubes (MWCNTs) and alumina nanofillers were employed for reinforcing the matrix through hybridization. The wear characteristics of the composite material were examined under dry sliding conditions, employing a pin-on-disc machine with a track diameter of 50 mm. The load on the specimen was varied between low (20 N), medium (40 N) and high (60 N), while the weight fraction of the hybrid nanofillers underwent variations in a range of 0.1–0.5 w/% with an increment of 0.1 w/%. The results showed that the reinforcement of hybrid nanofillers significantly reduces the wear phenomena of the composite material. Hybrid nanocomposites with (0.1, 0.2 and 0.3) w/% of MWCNTs-Al₂O₃ exhibit noteworthy advancements in the wear resistance. Particularly the 0.3 w/% MWCNTs-Al₂O₃ hybrid nanocomposite demonstrates exceptional wear resistance compared to pure epoxy. The incorporation of 0.3 w/% of MWCNTs-Al₂O₃ results in a significantly improved wear resistance, with enhancements of (83, 81 and 80) % observed during low (20 N), moderate (40 N) and high (60 N) loading conditions, respectively, compared to pure epoxy. Similarly, deformation, delamination and filler plugging were observed with medium and high load. The surface morphology of the worn specimens was assessed through the application of field emission scanning electron microscopy.

Keywords: wear, epoxy, MWCNTs, alumina, morphology analysis

Nova spoznanja o kompozitnih materialih na osnovi polimerne matrice so prispevala k njihovi intenzivni uporabi na različnih industrijskih področjih kot so ladjedelnštvo, proizvodnja avtomobilov, letalska industrija, industrija izdelave športne opreme in še na mnogo drugih. Te vrste kompozitov imajo dobro dimenzionalno stabilnost in odlične strukturne lastnosti. V tem članku avtorji opisujejo raziskavo obrabe hibridnega kompozita s polimerno matrico in ojačitveno fazo iz večstenskih ogljikovih nanocevk in delcev aluminijevega oksida. Drсно obrabo kompozitnega materiala so določili z napravo, ki ima vrtljivi disk in trn, ki pod izbranim tlakom drsi po preizkušancu (angl.: pin-on-disc machine) s premerom sledi 50 mm. Izbrali so naslednje obremenitve na trnu: nizko (20 N), srednjo (40 N) in visoko (60 N), medtem ko se je delež hibridnega polnila v kompozitu gibal med 0,1 w/% in 0,5 w/% v korakih po 0,1 w/%. Rezultati preizkusov so pokazali, da dodatek polnila oziroma ojačitvenih faz znatno zmanjša dršno obrabo hibridnega kompozita. Hibridni nanokompozit z 0,1 w/%, 0,2 w/% in 0,3 w/% polnila so imeli občutno boljšo odpornost proti drsni obrabi. Hibridni kompozit z 0,3 w/% polnila pa je imel še posebej dobro odpornost proti obrabi in sicer: kompozit z 0,3 w/% polnila je imel za 83 % manjšo obrabo pri obremenitvi 20 N, za 81 % manjšo obrabo pri obremenitvi 40 N in za 80 % manjšo obrabo pri obremenitvi 60 N v primerjavi s čistim epoksijem. Opazovali so tudi deformacijo, cepljenje (delaminacija) in utrditev nanodelcev v hibridnih kompozitih v odvisnosti od masne vsebnosti polnila. Površinsko morfologijo obrabljenih preizkušancev so opazovali s pomočjo vrstičnega elektronskega mikroskopa na emisiji polja elektronov.

Ključne besede: obraba, epoksi, večstenske ogljikove nanoceve, aluminijev oksid, morfološka analiza

1 INTRODUCTION

Epoxy resins are extensively utilized in scientific and technological applications, covering the marine, automotive, aerospace and sports industries, due to their remarkable mechanical properties.^{1,2} However, the wear phenomena and coefficient of friction exhibited by pure epoxy are substantial.³ Thus, hybrid nanocomposites have emerged as a novel category of materials, augment-

ing the performance of composite materials by unlocking novel characteristics and facilitating distinctive interactions among materials. Numerous studies indicate that hybrid nanocomposites exhibit elevated mechanical and thermal properties in comparison to traditional composites.⁴⁻⁷ Due to their increased aspect ratio and noteworthy mechanical characteristics, fitting physical, thermal and electrical properties, carbon nanotubes (CNTs) are presently acknowledged as a pivotal category of nanomaterial for the synthesis of polymer-infused nanocomposites. Carbon nanotubes (CNTs) have the capability to

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reinforce a polymeric matrix, enhance its toughness, and retard the propagation of cracks by establishing a bridging mechanism that binds crack surfaces.^{8,9} Hence, researchers in polymer science are actively seeking alternative strategies for the dispersion of CNTs within polymer matrices. In recent times, the integration of CNTs with inorganic additives has been observed to substantially enhance mechanical properties when compared with individual filler systems. In recent advancements, alumina (Al_2O_3) has emerged as an integral hybrid element within the composition of CNT hybrid compounds, playing a role as the filler in polymer composite systems.¹⁰ The widespread adoption of Al_2O_3 in hybridizations alongside CNTs can be attributed to their elevated hardness, refractoriness, exceptional dielectric and favourable thermal properties.¹¹

In this study, the hybrid compound of CNT- Al_2O_3 was synthesized through the CNTs on alumina particles, employing a catalyst precursor. High-density polyethylene exhibits admirable tensile and flexural properties, however, its proneness to tribological behaviour limits its use in minimum-load industrial application components. Furthermore, the incorporation of 0.1 w/% graphite nanoplatelets and graphene oxide demonstrated a lower contact pressure coupled with an increase in the wear resistance.¹³ Experiments conducted on multi-filler reinforced epoxy composites revealed enhancements in the tensile strength, tensile modulus, elongation at break, and wear resistance. These improvements were attributed to the reinforcement of multiple fillers and their compatibility with the epoxy matrix.¹⁴ The study aimed to evaluate the influence of MXene@Ag hybrids on the thermal, mechanical, and tribological properties of epoxy resin. The findings indicated a substantial reduction in the tribological properties, namely wear phenomena and friction characteristics of the composites when compared to the values observed for epoxy resin.¹⁵ Carbon nanotubes (CNTs) were synthesized and dispersed within the matrix of magnesium (Mg) with the aid of alumina as a 'vehicle.' This approach resulted in notable enhancements in the mechanical properties. Optimizing both mechanical and electrical characteristics necessitates a strategic improvement in the distribution of the hybrid filler within the epoxy matrix. An increased weight percentage of the filler will increase the mechanical properties and heat conductivity of ZrB₂ and decrease the smoothening of the hybrid composites due to the heat accumulation due to friction with a significant heat transfer. This phenomenon contributes to the reduction in the wear and adhesive wear of epoxy hybrid composites.¹⁶

Numerous research endeavours are dedicated to advancing polymer materials through the incorporation of nanofillers, with a specific emphasis on hybrid composites. This pursuit aims to not only enhance tribological properties but also to achieve multifunctional materials. Recent publications extensively delved into elucidating advancements in the mechanical and thermal properties

achieved by incorporating MWCNTs- Al_2O_3 hybrids into a polymer matrix. However, there have been limited investigations on the prediction of tribological performance and determining the optimum weight of hybrid composites. Consequently, the potential advantages of MWCNTs- Al_2O_3 hybrid particles have been harnessed to enhance the tribological properties of pure epoxy, and discussions have ensued regarding the optimal weight of MWCNTs- Al_2O_3 hybrid composites.

2 EXPERIMENTAL PART

2.1 Pure epoxy

In this study, the epoxy matrix used was Bisphenol A. The initial constituent is denoted as the epoxy resin, while the subsequent element is a curing agent, also known as a hardener. The solidification process, facilitated by the curing agent, enables the formation of a multidimensional cross-linked network within the epoxy resin. The supplier of the epoxy was BottomUp Technologies, Bangalore, India. The density of the epoxy was 1300 kg/cm^3 . The tensile (70 MPa), flexural (110 MPa) and compressive (380 MPa) strength of the epoxy were used in this study. The glass transition temperature of the epoxy was found to be $110 \text{ }^\circ\text{C}$. Moreover, the water absorption of the epoxy was recorded to be 0.5 w/%.

2.2 Synthesis of CNT-alumina hybrid nanofillers

The synthesis of CNT-alumina hybrid nanofillers was achieved through the chemical vapour deposition (CVD) method. Utilizing $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ as the catalyst precursor, carbon nanotubes (CNTs) were directly grown on alumina nanoparticles. First, alumina nanoparticles with an average size of 100–300 nm according to the manufacturer's data, shown in **Figure 1a**, was cleaned and prepared by treating it with a solution of HCl and rinsing it with deionized water. A solution of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was then deposited onto the alumina substrate. This is typically done by dipping the substrate into the solution and then drying it in air. The catalyst solution was formulated by blending $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and alumina in ethanol, subsequently subjecting it to sonication for a duration of 30 min. The resultant mixture was kept in an oven at $100 \text{ }^\circ\text{C}$ for 12 h for drying. Then, the CVD process was performed in a reaction chamber with the gas flow consisting of a mixture of acetylene (C_2H_2), hydrogen (H_2) and argon (Ar), with a ratio of 2:5:8 and a flow rate of about 1000 sccm. To facilitate the CNT growth, a distilled water bubbler was used. The reaction was performed at $900 \text{ }^\circ\text{C}$ for a duration of 30 min. Composites containing Co/alumina were synthesized, and the Co content was varied from 1.0 to 5.0 w/% with increments of 1.0 %.

The hybrid nanofillers synthesized from 1.0 w/% of Co/alumina displayed a limited quantity of grown CNTs, and these were uniformly distributed within the matrix.

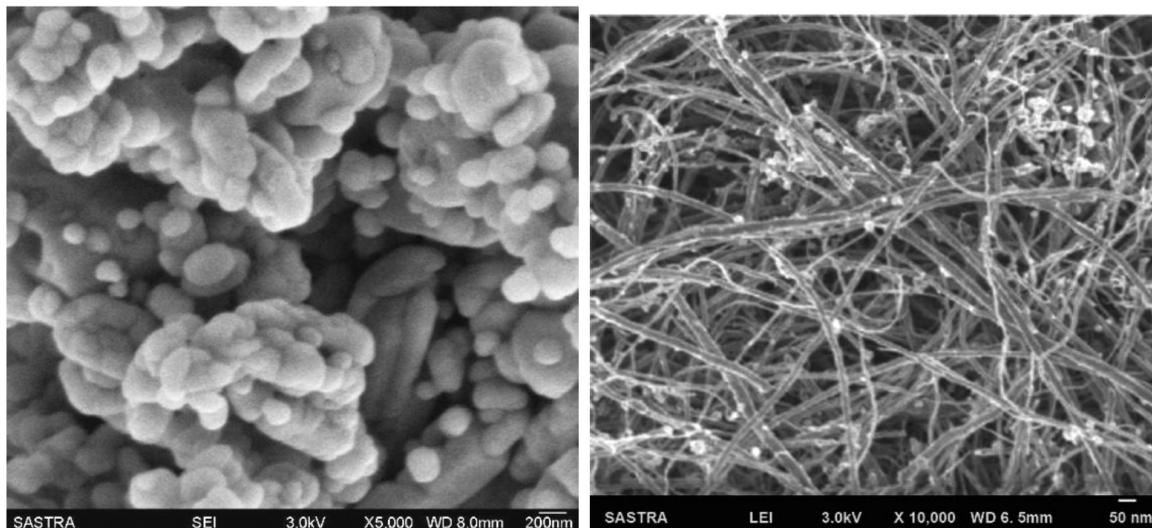


Figure 1: FESEM images of: a) alumina powder and b) CNT-alumina used in this experiment

An increase in the Co/alumina ratio from 2.0 w/% to 3.0 w/% resulted in a significant growth of CNTs in the alumina, which were also uniformly distributed in the matrix. However, a ratio of 4.0 w/% to 5.0 w/% demonstrated excessive growth of CNTs, forming bundles in the alumina. The CNTs were wrapped around the alumina particles due to the large quantity of CNTs grown as a result of the increase in the Co/alumina ratio. The grown CNTs had a diameter of about 50–100 nm and a length of about 10–20 μm . The results suggest that a higher concentration of the cobalt catalyst led to an increased yield of carbon nanotubes as shown in **Figure 1b**.

2.3 Fabrication of epoxy-CNT-alumina hybrid nanocomposites

The CNT-alumina hybrid nanofillers were dispersed into a solution of ethanol and water with a ratio of 5:3, along with a dispersant agent of 1.5 % sodium hexametaphosphate. The solution was then stirred using an ultrasonicator at a frequency of 75 kHz for 30 min, after which the sonication process was continued. Pure epoxy was introduced into the solution, and a vigorous sonication process was continued for another 20 min to enhance the blending between the pure epoxy and CNT-alumina hybrid nanofillers. Utilizing an ultrasonicator at a specific frequency during the dispersion process can potentially enhance the homogeneity and uniform distribution of CNT-alumina hybrid nanofillers within the matrix. The curing agent was then added into the solution with a ratio of 10:2, and the sonication process was continued with uniform mixing of the curing agent. The resultant solution was poured into an acrylic mould measuring 10 mm in diameter and 60 mm in length, as illustrated in **Figure 2**. Subsequently, the mould was kept in a vacuum oven for 2 h to evacuate air bubbles from the mixture, followed by maintaining it at

80 °C for 12 h. The mould was then cooled to room temperature, and hybrid nanocomposite samples were obtained. The weight ratio of the filler in the cured samples varied from 0.1 w/% to 0.5 w/% with an increment of 0.1 %. Five samples with each weight ratio were prepared.

2.4 Wear test

A pin-on-disc machine was employed to carry out a dry-sliding wear test, using a Ducom TR20, following the guidelines outlined in the ASTM standard G99. Low (20 N), medium (40 N) and high (60 N) sliding loads were applied, and the counterpart disc speed was set to 200 min^{-1} . The experiment ran for a total of 30 min with a sliding distance of 1000 m.¹⁷ A wear test sample with a pin sample size of 10 mm in diameter and 30 mm in height was used. A 100Cr6 steel disc was used as the counter-part material and, to ensure a uniform contact, a fine emery sheet was employed to meticulously polish the surface of the pin. The diameter of the wear track

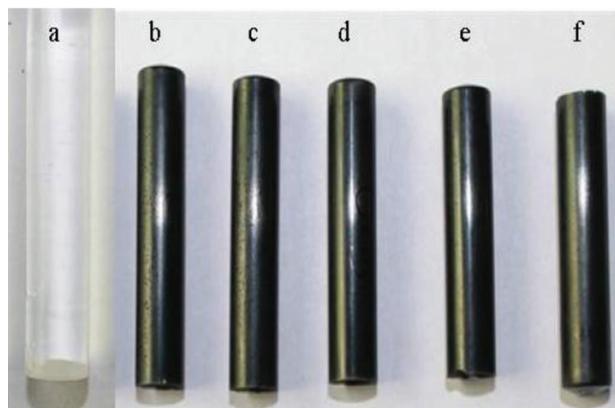


Figure 2: Wear specimens of: a) pure epoxy, b) 0.1, c) 0.2, d) 0.3, e) 0.4, f) 0.5 weight fraction of MWCNTs- Al_2O_3 hybrid nanocomposites

was 50 mm. The software and hardware components of the machine were utilized to configure the parameters required for the initialization, such as the disc speed, specimen load and duration of the experiment. The software module supported the recording of the wear and friction values. Prior to loading them into the machine, the samples were weighed using an electronic balance with a precision of 0.1 mg. The wear loss was calculated based on the initial and final weight of the samples; before and after the test, acetone soaked soft paper was used to clean the counterpart. The surface morphology of both pure epoxy and hybrid nanocomposite worn surface were scrutinized using a field emission microscope, providing a deeper understanding of the wear phenomena.

3 RESULTS AND DISCUSSION

3.1 Tribological properties

The investigation into the tribological properties of epoxy and hybrid nanocomposites, specifically multi-walled carbon nanotubes (MWCNTs)/alumina, reinforcing the epoxy, was conducted employing a pin-on-disc

machine. The pure epoxy exhibited average wear rates of (0.06, 0.10 and 0.15) mm³/Nm under the low, medium and high load, respectively. However, the additions of 0.1 w/% and 0.2 w/% of hybrid nanofillers into the epoxy matrix considerably reduced the wear rate to (0.05, 0.07, 0.12 and 0.03, 0.05, 0.08) mm³/Nm, respectively, in low, medium and high load conditions. Furthermore, the significant reduction in the wear rate was observed to be linked with the hybrid nanofiller content of 0.3 w/% in the epoxy matrix and the wear values were (0.01, 0.02, 0.03) mm³/Nm under different loading conditions. This result showed that the wear resistance was improved by (83, 81 and 80) % in comparison with pure epoxy under different loading conditions. Similarly, the addition of 0.4 w/% caused the wear rate to be lower than that of pure epoxy and higher than that of 0.3 w/% hybrid nanofiller composite. Furthermore, the increase in the hybrid nanofiller content to 0.5 w/% caused the wear rate to be the same as that of pure epoxy. The spatial network structure within the epoxy resin, formed by MWCNT-alumina, effectively dispersed the frictional heat generated at the interface, promptly hindering resin degradation and softening. Additionally, the optimized proportion of hybrid nanofillers guaranteed a uniform and efficient distribution within the pure epoxy. The optimal weight percentage of the hybrid nanofillers played a significant role in the formation of a high-quality friction film, facilitating the transfer of friction. Furthermore, the worn surface of the 0.3 w/% hybrid nanocomposite exhibited punctate protrusions, suggesting an enhancement in the shear strength of the pure epoxy and an effective protection against material peeling during the friction process, resulting in a reduction of the wear rate. An excessive incorporation of hybrid nanofillers into the epoxy resulted in the formation of voids, compromising the material's structural integrity, mechanical properties and overall performance within the host matrix, attributable to the non-uniform distribution of the filler materials. In **Figures 3a to 3c**, the specific wear rates of pure epoxy and different weight fractions of the hybrid nanocomposites under the low, medium and high loads are graphically represented.

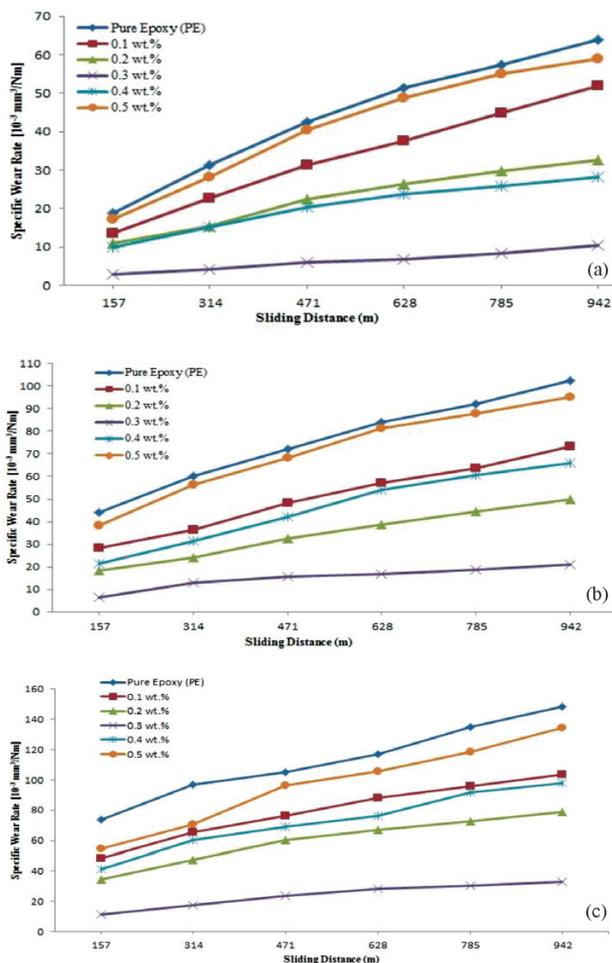


Figure 3: Specific wear rate of pure epoxy and hybrid nanocomposites at: a) low-load (20 N) conditions, b) medium-load (40 N) conditions, c) high-load (60 N) conditions

3.2 Morphology analysis of epoxy and hybrid nanocomposites

The surface morphology and internal wear mechanism of pure epoxy and epoxy/MWCNTs-alumina hybrid nanocomposites were investigated (**Figures 4 to 6**). The worn surface of pure epoxy exhibited hyperbolic marking, pull-outs, deep grooves and fractures, indicating poor wear resistance and brittleness in low (20 N), medium (40 N) and high (60 N) load conditions. The introduction of 0.1 w/% to 0.5 w/% of MWCNTs-alumina hybrid nanofillers into the epoxy considerably improved the wear resistance of the host matrix. In low, medium and high load conditions, a specimen's worn surface exhibited a petal surface, grooves, plastic deformation,

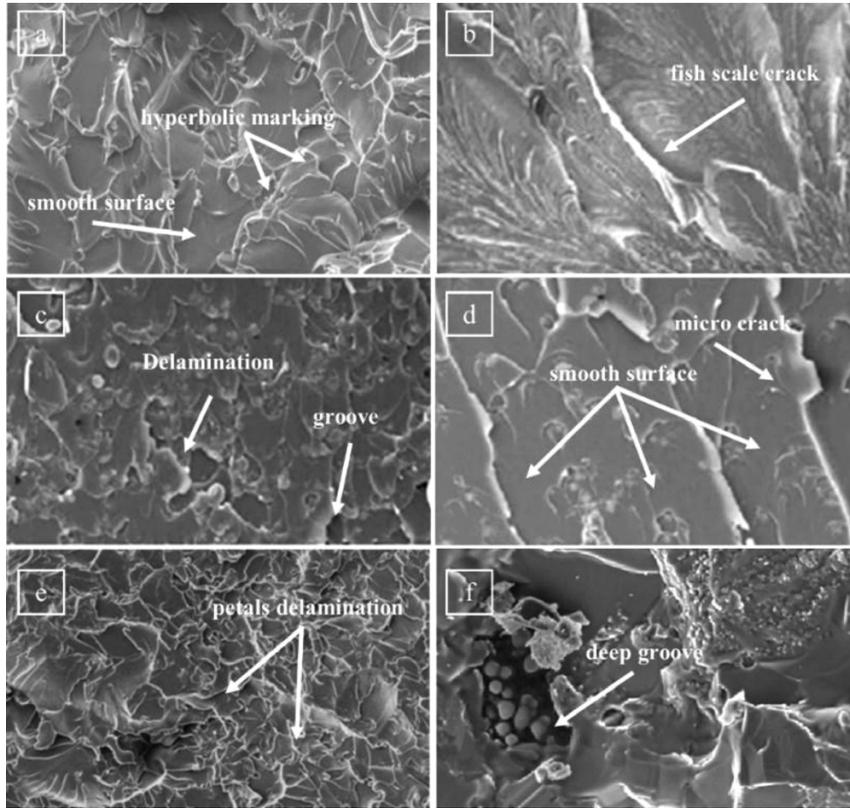


Figure 4: Morphology analysis of a) pure epoxy, b) 0.1, c) 0.2, d) 0.3, e) 0.4, and f) 0.5 weight fraction of MWCNTs- alumina hybrid nanocomposites during low (20 N) loading

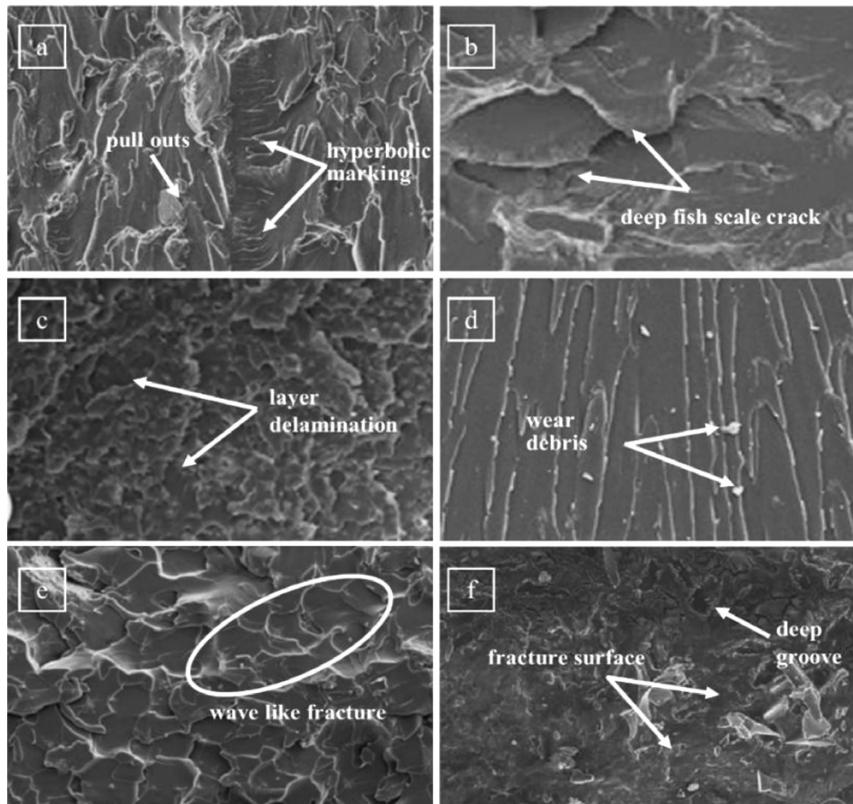


Figure 5: Morphology analysis of a) pure epoxy, b) 0.1, c) 0.2, d) 0.3, e) 0.4, and f) 0.5 weight fraction of MWCNTs-alumina hybrid nanocomposites during low (40 N) loading

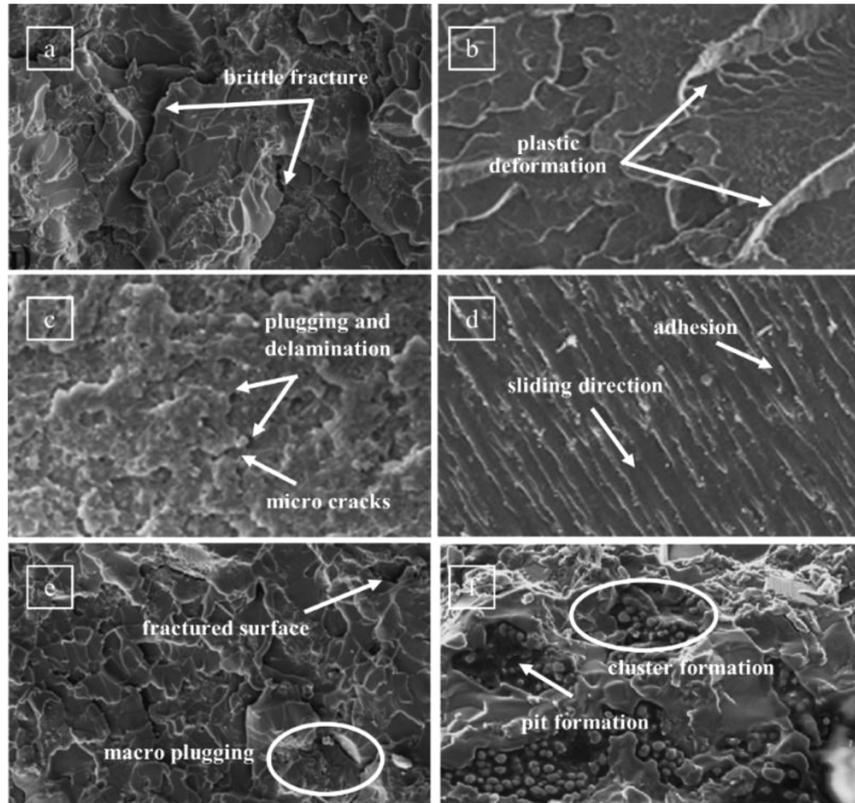


Figure 6: Morphology analysis of a) pure epoxy, b) 0.1, c) 0.2, d) 0.3, e) 0.4, and f) 0.5 weight fraction of MWCNTs-alumina hybrid nanocomposites during low (60 N) loading

delamination, matrix plugging and microcracks, respectively. Plastic distortion on a worn surface indicates that the sample is brittle in nature, thus easily subjected to plastic deformation. Further addition of 0.3 w% of hybrid nanofillers to pure epoxy significantly improved the wear resistance, resulting in a worn surface with microcracks, smoothness, tinny wear debris, and adhesion delamination. A smooth surface was identified on the sample, which was attributed to the large frictional force developed on the worn surface due to the tangential displacement due to continuous initiation and propagation of cracks that finally evolved into fatigue phenomena. These were due to the accumulation of continuous stress and destress of the worn surface, leading to the brittleness of the material. Furthermore, it was observed that the debris rolled between the worn surface and the counterpart were pulverised into fine particles. The fine particle occupied the microcracks on a worn surface, providing long-run stability. Similarly, further additions of 0.4 w% and 0.5 w% of hybrid nanofillers to pure epoxy caused deep grooves, petal-surface layer deformation, wave-like fracture, the pit and cluster formation on the surface.

4 CONCLUSIONS

In this research study, MWCNTs-alumina nanocomposites were fabricated using chemical vapour deposition where carbon nanotubes were grown directly on alumina particles. The hybrid nanofiller amount ranged from 0.1 w% to 0.5 w% with an increment of 0.1 w%. The primary accomplishments of this investigation can be listed as follows:

- MWCNTs- Al_2O_3 hybrid nanocomposites with (0.1, 0.2 and 0.3) w% exhibit noteworthy advancements in wear resistance. Particularly, the 0.3 w% MWCNTs- Al_2O_3 hybrid nanocomposite demonstrates exceptional wear resistance compared to pure epoxy.
- The introduction of a low amount of the MWCNTs- Al_2O_3 hybrid nanocomposite results in a substantial enhancement of the tribological performance of the pure epoxy matrix.
- The incorporation of 0.3 w% of MWCNTs- Al_2O_3 hybrid nanocomposite results in significantly improved wear resistance, with enhancements by (83, 81 and 80) % observed during low (20 N), moderate (40 N) and high (60 N) loading, respectively, compared to pure epoxy.
- The addition of 0.3 w% of hybrid nanofillers into pure epoxy significantly improves the wear resistance, as evidenced by the presence of microcracks,

smooth surface, tiny wear debris and adhesion delamination on the worn surface, indicating enhanced material integrity and performance.

- The presence of deep grooves, petal-surface layer deformation, wave-like fractures, pits, and cluster formations on the worn surfaces of epoxy composites with 0.4 w/% and 0.5 w/% hybrid nanofillers indicates increased wear and potential degradation, highlighting the importance of optimizing the nanofiller amount for maintaining the material performance and integrity under high load conditions.
- The presence of plastic deformation, delamination, matrix plugging and microcracks on a worn surface under various load conditions indicates a transition from a brittle to a more plastic nature of the sample, suggesting enhanced ductility and resistance to wear.

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