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EXPERIMENTAL INVESTIGATIONS ON TRIBOLOGICAL AND MECHANICAL BEHAVIOR OF CNTs/Al_2O_3/GNP REINFORCED EPOXY AND POLYAMIDE HYBRID NANOCOMPOSITES

EKSPERIMENTALNE PREISKAVE TRIBOLOŠKEGA IN MEHANSKEGA OBNAŠANJA S CNTs, Al_2O_3 IN GNP OJAČANIMI EPOKSIDNIMI TER POLIAMIDNIMI HIBRIDNIMI NANOKOMPOZITI

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In the present investigation, dry sliding tribological behavior and mechanical properties of pure epoxy, Polyamide 66, and their hybrid nanocomposites were systematically analyzed. The experimental investigation primarily focused on two critical aspects: firstly, the influence of contact pressure on the wear behavior under varying loading conditions, and secondly, the role of mechanical interlocking and adhesion between the polymer matrix and hybrid nanocomposites, enhancing the tensile properties. Nanocomposite formulations included multiwall carbon nanotubes (MWCNTs), graphene nanoplatelets (GNPs), and alumina (Al_2O_3) as the reinforcement materials, each added at a weight fraction of 0.2 %. The study explored significant contributions of these nanofillers in terms of their functional properties and impact on tribological performance and load transfer mechanisms within the composite materials. It was observed that epoxy and Polyamide 66 reinforced with 0.2 % CNT/Al_2O_3 demonstrated superior wear resistance, particularly under conditions of high contact pressure. Additionally, both polymer matrices exhibited enhanced tensile strength and nanoindentation when reinforced with a combination of 0.2 % CNT/Al_2O_3. This research highlights the potential of carefully selected nanofillers in optimizing the mechanical and tribological performance of polymer-based composites, providing valuable insights required for future material design and application in various engineering fields.

Keywords: wear, tensile properties, nanoindentation, CNTs, Al_2O_3, graphene nanoplatelets

Povzetek: v pričujočem članku avtorji opisujejo raziskavo v kateri so sistematično analizirali tribološke in mehanske lastnosti čistega epoksija in poliamida tipa 66 ter jih primerjali z njunimi hibridnimi nanokompoziti. Z eksperimentalnimi preiskavami so se osredotočili predvsem na dva vidika: najprej kako vpliva kontaktni tlak na obrabo izbranih (izdelanih) materialov pri različnih obremenitvah in drugič, kakšna je vloga mehanske vezave (prepletanja) ter adhezije med polimerno matrico in ojačitveno fazo hibridnih nanokompozitov. Pri nanokompozitih so avtorji v polimerne matrice vgradili, kot ojačitvene faze, naslednje materiale: večstenske ogljikove nano- cevčice (MWCNTs; angl.: Multiwall Carbon Nanotubes), grafenske nano ploščice (GNPs) in aluminijev oksid (Al_2O_3). Vsakega od teh so vgradili v polimerno matrico po 0,2 %. Študija je pokazala pomembno vlogo teh nano polnil pri izboljšanju funkcionalnih lastnosti in mehanizmov prenosa obremenitev nanokompozitov. Avtorji so ugotovili, da epoksidna smola in poliamid 66 ojačana z 0,2 % CNT in 0,2 % Al_2O_3 imata najboljšo odpornost proti suhi (nemazani) drsni obrabi, še posebej pri uporabi višjih kontaktnih tlakov. Obe ojačani polimerni matrici (epoksidna in poliamidna) sta imeli izboljšano odpornost proti nateznim obremenitvam in višjo odpornost proti nanoindentaciji (vtiskavanju nano indenterja v matrico kompozita). S to raziskavo so avtorji potrdili, da ustrezna izbira vrste in deleža ojačitve lahko ugodno vpliva na mehanske in tribološke lastnosti polimernih materialov, ki kot taki lahko predstavljajo nove bodoče materiale za različne inženirske aplikacije.

Ključne besede: obraba, natezne obremenitve, nanoindentacija, ogljikove večstenske nano-cevčice, Al_2O_3, grafen, nano ploščice

1 INTRODUCTION

The combination of carbon nanotubes (CNTs) and alumina in epoxy composites has been shown to significantly enhance both thermal and tribological properties. This enhancement is primarily due to the improved load distribution and reduced wear rate under dry sliding conditions.[1] The hybridization of CNTs and graphene nanoplatelets (GNPs) in epoxy matrices has been observed to enhance the tensile modulus and tribological performance, attributed to the synergistic effects of both nanofillers in reducing friction and wear.[2] Incorporating GNPs and alumina in epoxy composites offers improved tribological properties, such as lower friction coefficients and better wear resistance, which are crucial for advanced engineering applications.[3] Polyamide 66 composites reinforced with CNTs and alumina reveal a significant reduction in the wear rate and friction coefficient, owing to the synergistic effect of CNTs and alumina, improving the load distribution and wear resistance under dry sliding conditions. An optimized composite demonstrates enhanced tribological properties, particularly at

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higher loads and sliding speeds, making it suitable for demanding engineering applications.[4] The incorporation of CNTs and GNPs into a Polyamide 66 matrix significantly enhances the material's tribological performance. The hybrid reinforcement reduces the coefficient of friction and wear rate, primarily due to the improved load-bearing capacity and self-lubricating properties exhibited by the CNT/GNP combination, providing a balanced distribution of applied stress during sliding.[5] Studies on Polyamide 66 reinforced with GNPs and alumina particles demonstrate substantial improvements in wear resistance and frictional behavior. A hybrid nanocomposite effectively reduces the material's degradation under various sliding conditions, attributed to the enhanced hardness and thermal stability provided by the alumina, combined with the lubricating effect of GNPs.[6] The inclusion of CNTs and alumina in Polyamide 66 enhances the wear resistance under varying load conditions. The composite exhibits lower friction and improved durability, which is beneficial for applications involving sliding contact.[7] Tribological studies show that CNT and GNP-reinforced Polyamide 66 composites show reduced wear rates and better load-carrying capacity. This is attributed to the improved distribution of fillers within a polymer matrix.[8]

The incorporation of CNTs into alumina-reinforced epoxy composites significantly enhances the tensile strength due to improved load transfer and interfacial bonding.[9] A combination of CNTs and GNPs in epoxy composites creates a synergistic effect that leads to significant improvements in both elastic and viscoelastic properties.[10] Hybridization of CNTs and GNPs in epoxy resin leads to improved tensile modulus and fracture toughness due to an efficient load transfer between the matrix and the nanofillers. The inclusion of GNPs in alumina-epoxy composites improves the thermal conductivity while maintaining the mechanical strength, making the material suitable for high-performance applications.[11] A study found that the hybrid reinforcement of GNPs and alumina in Polyamide 66 leads to improved impact resistance and mechanical stiffness. The composite shows significant potential for use in automotive and aerospace industries.[12]

The incorporation of CNTs and GNPs into polymer matrices, particularly epoxy and Polyamide 66, significantly enhances both thermal and tribological properties. The synergistic effects of these nanofillers lead to improved load distribution, reduced wear rates, and better mechanical strength, making these composites suitable for advanced engineering applications. Optimized combinations of CNTs, GNPs, and alumina offer promising potential for use in high-performance environments, including automotive and aerospace sectors. These findings underscore the importance of hybrid nanocomposites in advancing material science for industrial applications.

2 MATERIALS AND METHODS

2.1 Materials

The polymer composites investigated consist of bisphenol-A diglycidyl ether (DGEBA) epoxy resin (Dow DER 331) and Polyamide 66 (PA66, Ultramid A3WG7) matrices hereafter referred to as EP and PA66, chosen for their high mechanical and thermal properties. The DGEBA epoxy exhibits strong tensile strength and chemical resistance, while PA66 provides excellent wear resistance and toughness. To enhance these matrices, CNTs with an average diameter of 10 nm, a length of 1–5 µm, and a specific surface area of 200–400 m^2/g, along with GNPs with a thickness of 5–10 nm, lateral dimensions of 1–5 µm, and a specific surface area of 750 m^2/g were incorporated. CNTs improve load transfer and reduce wear, while GNPs enhance tensile modulus and thermal stability. Additionally, alumina (Al_2O_3) nanoparticles with an average size of 50 nm were used as a ceramic filler, known for their hardness and thermal conductivity, further boosting wear resistance and mechanical strength. The synergistic effects of these nanofillers result in composites with lower friction coefficients, enhanced thermal properties, and improved durability. The PA66 composites were fabricated using a twin-screw extruder at 292 °C, followed by injection

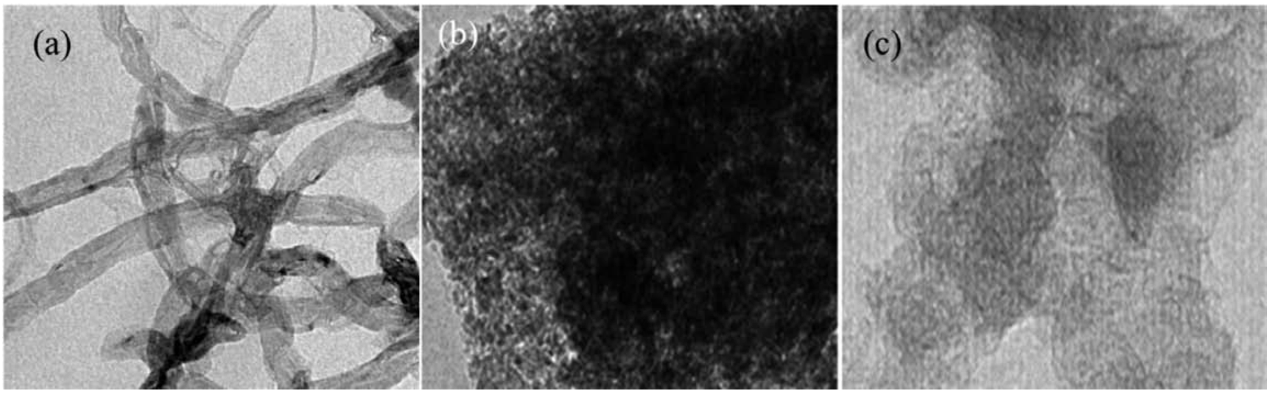

Figure 1: TEM images: a) CNT, b) alumina and c) GNP nanofillers

molding at 500 bar, ensuring uniform nanofiller dispersion. The epoxy composites were mechanically mixed and cured with a polyamine hardener (Dow HY 2954) to optimize interfacial bonding and load transfer. Morphologies of CNTs, alumina and GNPs obtained with TEM are shown in **Figure 1**.

2.2 Functionalization of the nanofillers

Surface functionalization is a crucial process for enhancing the interfacial properties of nanomaterials, thereby improving their compatibility with polymer matrices. This modification enables better dispersion, stronger adhesion, and optimized performance of composites in various advanced applications. Multiwall carbon nanotubes (MWCNTs) are commonly functionalized using a mixture of concentrated nitric acid (HNO_3) and sulfuric acid (H_2SO_4) in a typical ratio of 1:3. MWCNTs are dispersed in this acidic mixture and subjected to ultrasonication for 2 h or refluxed at around 140 °C for 30–60 min. This process introduces carboxyl (-COOH) and hydroxyl (-OH) groups onto the nanotube surfaces, improving their hydrophilicity and compatibility with various polymer matrices. After the reaction, the MWCNTs are thoroughly washed with deionized water until the pH is neutral and then dried under a vacuum at 60 °C.

GNPs are often treated with a mixture of sulfuric acid (H_2SO_4) and nitric acid (HNO_3) in a 3:1 ratio. GNPs are immersed into an acid mixture and subjected to ultrasonication or stirring at an elevated temperature (usually 60–80 °C) for 2–4 h. This process generates oxygen-containing functional groups such as carboxyl (-COOH) and hydroxyl (-OH) on the graphene surface, enhancing their dispersibility in polar solvents and polymer matrices. Alumina particles are often modified using silane coupling agents, such as 3-aminopropyltriethoxysilane (APTES) or γ-methacryloxypropyltrimethoxysilane (γ-MPS). The process involves dispersing alumina in an organic solvent like ethanol or toluene, followed by the addition of the silane agent. The mixture is stirred at room temperature or slightly elevated temperatures (60–80 °C) for several hours. This treatment forms a covalent bond between the silane molecules and the hydroxyl groups on the alumina surface, introducing functional groups that enhance adhesion with polymers, particularly in composite materials.

2.3 Preparation of hybrid nanocomposites samples

In order to prepare test samples, mechanical mixing and solution deposition were combined. Shear mixing was then performed in a co-rotating twin-screw injection molding machine. Using ultrasonication, an ethanol-based nanofluid with a predetermined number of fillers was created. After adding PA66 billets to the nanofluid, the mixture was heated to promote filler deposition onto the PA66 billets as the ethanol evaporated. After that, the pellets were dried in an oven for around ten hours at 75 °C to remove any traces of moisture. Samples were made using a 50-ton injection molding machine equipped with a twin-screw heating chamber, with both screws rotating synchronously. There were four phases of the processing temperature, which varied from around 40 °C at the hopper to about 165 °C at the injection nozzle. To guarantee perfect filling of the dies without any flaws, the cycle time was set to roughly one minute, the screw speed was set to sixty revolutions per minute, and all other parameters were optimized. In a similar manner, after sonication, EP composites were created by curing them with a polyamine hardener. Seven samples – one of which was pure Polyamide 66 – were made by combining pre-measured amounts of surface-modified fillers to make hybrid nanocomposites. By introducing specific fillers into the matrix to create a hybrid nanocomposite, the efficacy of EP and PA66 was evaluated. To create the nanocomposites, three fillers – multi-walled carbon nanotubes (MWCNTs), graphite nanoplatelets (GNPs) and alumina – were used, each at 0.2 *w*/%. Three epoxy and three polyamide hybrid nanocomposites, as well as two pure epoxy and two polyamide samples were created. **Table 1** shows the filler contents and labelling of epoxy and polyamide hybrid nanocomposites. For tribology tests, test samples of 10 mm in diameter and 30 mm in length were created. Additionally, the samples were machined to their final dimensions for the tensile testing.

Table 1: Polymer matrix and nanofiller composition used in this investigation

Matrix	Nanofiller composition (wt.%)			Label	Density
	CNTs	GNPs	Al_2O_3		
Epoxy	0.2	0.2	0	ECG	1.212
	0.2	0	0.2	ECA	1.284
	0	0.2	0.2	EGA	1.310
PA66	0.2	0.2	0	PCG	1.209
	0.2	0	0.2	PCA	1.313
	0	0.2	0.2	PGA	1.249

2.4 Sliding tribological tests

A sliding tribology wear experiment was carried out using a Ducom TR 20 pin-on-disc machine, a widely recognized apparatus for evaluating wear behavior under controlled conditions. The test samples, including epoxy or polyamide (PA66), were meticulously machined to precise dimensions, measuring 30 mm in length and 10 mm in diameter. These hybrid nanocomposites were chosen due to their potential to exhibit improved mechanical and tribological properties, which are crucial in various industrial applications. For the counterpart material in this experiment, a hardened steel disc was used. This disc, made of 100Cr6-grade steel, was a high-carbon chromium alloy known for its high wear resistance and hardness. The selection of this material ensured that the wear behavior observed was primarily due to the nanocomposites tested, rather than to the counterpart material. The rotating disc acted as the sliding surface, against which the nanocomposite samples were tested.

The primary objective of the experiment was to analyze the wear characteristics of the hybrid nanocomposites by varying the applied functional load, which directly influenced the contact pressure at the interface between the pin and the disc. The contact pressure, a critical factor in tribological studies, was calculated by dividing the applied load by the contact surface area of the test samples. This relationship between load and surface area is essential for understanding how different levels of stress impact the wear rate and frictional properties of a material. Three distinct levels of applied load were utilized in the experiment: low (15 N), medium (30 N), and high (45 N). These values were chosen to represent a broad range of operational conditions, allowing for a comprehensive analysis of how varying loads affect the wear performance of the hybrid nanocomposites. By testing them at multiple load levels, the experiment was able to simulate a range of real-world applications where contact pressures may fluctuate due to changes in load-bearing requirements. The wear performance of the samples was measured using a pin-on-disc machine equipped with a computer-integrated LVDT (linear variable differential transformer) sensor. This sensor is highly sensitive and capable of providing precise measurements of wear over time, making it ideal for capturing small changes in the material's surface. The data collected by the LVDT sensor enabled a detailed analysis of the wear behavior of the samples in relation to both contact pressure and time, providing insights into how the material degrades under different operating conditions.

The scope of this tribological study is broad and aims to provide a comprehensive understanding of the mechanisms behind the wear and friction characteristics of epoxy and PA66 hybrid nanocomposites. In particular, the study seeks to elucidate how the incorporation of hybrid fillers within the nanocomposites influences their wear resistance and frictional behavior. By examining the interaction between the hybrid fillers and the matrix material under different loads, the study contributes valuable knowledge to the development of advanced materials with enhanced performance in wear-intensive applications.

2.5 Tensile testing

Tensile tests of epoxy and polyamide (PA66) hybrid nanocomposites played a pivotal role in evaluating the mechanical behavior of the materials. Specimens for the tensile test were fabricated following the ASTM D3039 standard, ensuring consistency and comparability with other studies. The use of standardized specimen dimensions is necessary in maintaining uniformity of test results, which are crucial for mechanical characterization. The tensile tests were performed using a Shimadzu tensile test machine, applying a load rate of 0.1 N/mm/min, and conducted at room temperature with an overhead speed of 5 mm/min. The controlled conditions ensure that the test data accurately reflect the material's performance under typical environmental and operational settings. To ensure statistical accuracy and reliability, five specimens of identical composition were tested, and the average tensile strength values were calculated. This approach minimizes variability due to potential anomalies in individual specimens and provides a more reliable representation of the material's mechanical properties. During the tensile testing, key properties such as ultimate tensile strength, Young's modulus, and elongation at break were assessed. These parameters are essential for understanding the load-bearing capabilities and deformation behavior of the hybrid nanocomposites. The inclusion of hybrid fillers, such as nanomaterials, within the epoxy matrix likely enhances the tensile strength and stiffness, as these fillers improve the load transfer and resistance to crack propagation.

2.6 Nanoindentation testing

Quasistatic nanoindentation tests were used to examine the mechanical properties of epoxy, PA66, and its hybrid nanocomposites, including hardness, stiffness, and surface piercing. A cut specimen's final measurements were (20 × 20 × 0.5) mm, and it was adhered to the carbon steel plate using glue. A Hysitron TI 950 Triboindenter was employed as the nanoindenter. The following parameters were used for the tests: a maximum force of 6000 μN, loading and unloading times of 15 s, and a dwell time of 5 s. A 100-nm radius Berkovich diamond tip, shaped like a three-sided pyramid, was employed. The angle of inclusion was 135.3, the curvature radius was approximately 125 nm, the depth resolution was 0.02 nm, and the load resolution was less than 1 nN. Five indents were made on each specimen and the values were recorded.

3 RESULT AND DISCUSSION

3.1 Wear characteristics

The experimental investigation focused on examining the impact of contact pressure on the wear characteristics of each tested sample. **Figure 2** (a, b, c) depicts the contact pressure and specific wear rate for each sample at applied loads of (15, 30 N and 45) N, respectively. At these loads, the contact pressures for epoxy and PA66 were (24.7, 46.1, and 72.1) N/mm^3 for epoxy, and (26.12, 48.8, and 76.2) N/mm^3 for PA66. The results demonstrated a significant reduction in the contact pressure for the ECA nanocomposite when subjected to identical loads. Specifically, the contact pressure decreased by (68, 62, and 58) % in comparison to the epoxy matrix. This trend underscores the superior performance of ECA in lowering the contact pressure. Furthermore, when examining the hierarchy of contact pressure induced across

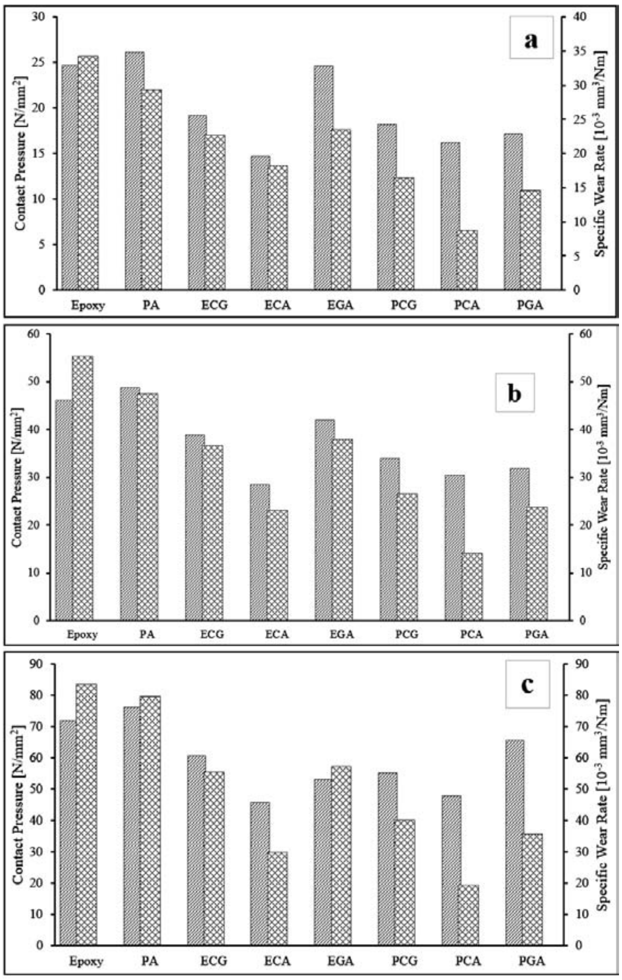

Figure 2: Contact pressure vs. specific wear rate of epoxy, polyamide and its hybrid nanocomposites

the various nanocomposites, the order observed was as follows: EPOXY > EGA > ECG > ECA. This ranking highlights the effectiveness of ECA in reducing the surface stress relative to other nanocomposites. Similarly, the polyamide (PA66) matrix and its nanocomposites exhibited the lowest contact pressure among the materials tested. Notably, the PCA hybrid nanocomposites consistently demonstrated substantial reductions in the contact pressure at (15, 30, and 45) N, showing decreases of (61, 59, and 60) %, respectively, when compared to pure PA66. These findings confirm the enhanced load-bearing capacity and improved performance of the PCA nanocomposite under varying load conditions.

The specific wear rate of epoxy and its nanocomposites was measured at (15, 30, and 45) N, with the corresponding contact pressures. Among the epoxy nanocomposites, ECA demonstrated specific wear rates of (18.19, 23.08, and 29.30 (× 10^{-3})) N/mm^2 for the respective loads. Compared to pure epoxy, the results show that the specific wear rate of ECA decreased significantly by (46, 58, and 65) %, highlighting its enhanced wear resistance. In parallel, the specific wear rate of PA66 and its nanocomposites, specifically PCA, exhibited substantial reductions of (70, 71, and 76) % relative to pure PA66. This marked improvement in the wear resistance further validates the superior performance of the nanocomposites under varying load conditions. The investigation clearly demonstrates that, compared to their pure counterparts, the nanocomposites ECA and PCA consistently exhibited the lowest wear across all evaluated load intensities.

3.2 Comparison of specific wear rates of hybrid nanocomposites

In a comprehensive comparison of the analyzed samples, ECA exhibits the lowest contact pressure, while PGA generates the highest, particularly when considering hybrid composites. The distinct behavior of these materials can be attributed to the distribution and interaction of GNPs and alumina within the (PA66) matrix. In the case of the 0.2 GNP/0.2 Al_2O_3 hybrid nanocomposite, the expected enhancement in the synergy between the fillers is not achieved although the volumetric presence of GNPs is significantly higher than that of alumina. This lack of synergy stems from the tendency of GNPs and Al_2O_3 to intercalate and form an interstitial solid solution, which hinders effective load transfer and stress distribution throughout the composite. An even distribution of the fillers within the polymer matrix is crucial, yet not sufficient to fully realize the potential of the hybrid nanocomposite properties. The degree of filler-matrix adhesion plays a pivotal role, and in this case, the interfacial bonding between the fillers and PA66 is only moderate, resulting in suboptimal filler-matrix interlocking. This moderate interlocking mechanism restricts the ability of the hybrid nanocomposite to resist applied stresses effectively, leading to localized stress concentrations and potential failure points within the material. Furthermore, the presence of GNPs on the alumina surface in the PA66 matrix was observed to induce a stepped morphology on the surface. This stepped pattern indicates an incomplete cohesion at the filler-filler interface, particularly due to the weak van der Waals forces associated with alumina. Alumina's relatively weak surface energy leads to insufficient adhesion, causing the material to slide against the counterpart surface during testing. This sliding results in surface deformation and wear, which are evident in the stepped morphology. Despite these challenges, alumina plays a crucial role in load-bearing and thermal stability. During sliding wear tests, alumina withstands the applied loads with induced thermal stresses, helping to prevent excessive deformation of the composite.

Alumina's contribution to the thermal resistance of the material is crucial, as it protects the PA66 matrix from softening under high-friction conditions. Its ability to support the structural integrity of the composite during prolonged testing is vital for maintaining performance, especially in applications where thermal stability and wear resistance are paramount. The samples' worn sur-

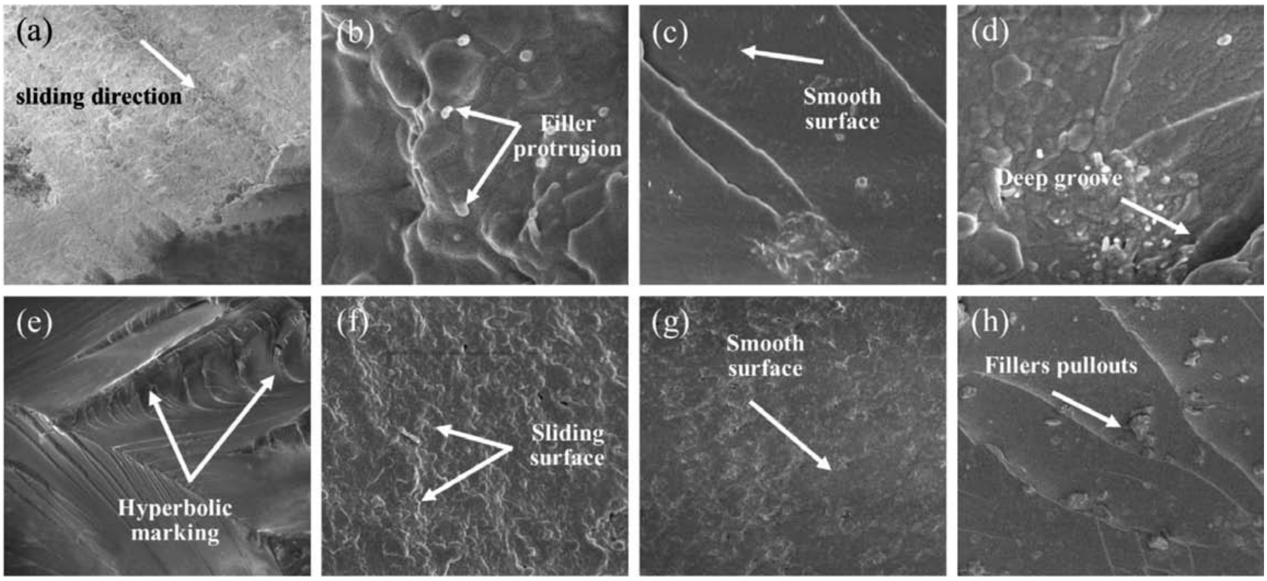

Figure 3: FESEM morphology analysis of worn surfaces: a) epoxy, b) ECG, c) ECA, d) EGA, e) PA66, f) PCG, g) PCA, h) PGA at the load of 45 N

faces observed with a field emission scanning electron microscope are shown in **Figure 3** (a–h).

3.3 *Tan delta vs. contact pressure*

The parameter Tan δ, or damping factor, is a crucial indicator for assessing the viscoelastic behavior of materials, particularly when distinguishing between their elastic and viscous responses. Mathematically, Tan δ represents the ratio of the loss modulus to the storage modulus (Tan $\delta = E''/E'$), where the loss modulus measures the energy dissipated as heat, and the storage modulus reflects the energy stored elastically within the material. A Tan δ value approaching zero signifies a highly elastic material, indicating minimal energy dissipation and excellent resilience. Conversely, a value closer to 1 indicates a predominantly viscous material, where energy is lost through internal friction, signifying poor elastic recovery. In polymer science, the viscoelastic transition is often observed with temperature variations. For instance, polymers exhibit increased viscous behavior upon extended heating, particularly during the leathery phase, when polymer chains exhibit sufficient mobility to slide past one another without fully melting. This phenomenon is crucial for understanding the thermal and mechanical stability of polymeric materials.

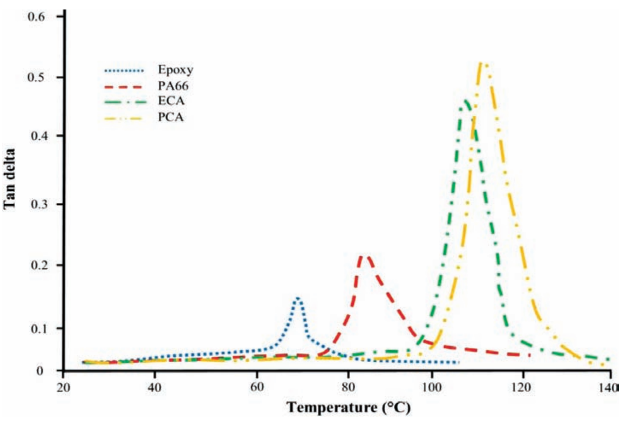

Figure 4: Tan delta vs. elevated temperature of the samples

The Tan δ curves of the analyzed samples, presented in **Figure 4**, illustrate these behaviors for both epoxy and PA66 matrices. Specifically, epoxy and PA66 have Tan δ values of 0.16 and 0.21, respectively, indicating that both materials exhibit a relatively elastic nature under the test conditions. These values suggest that while some energy is dissipated, a significant portion is retained elastically. In contrast, hybrid nanocomposites show distinct viscoelastic properties, particularly under elevated temperatures (25–130 °C). For example, the Tan δ peaks of the hybrid nanocomposites ECA and PCA are 0.49 and 0.53, respectively. These elevated values indicate a pronounced viscous response, suggesting that, at higher temperatures, the hybrid nanocomposites demonstrate more significant energy dissipation, characteristic of a material transitioning from elastic to viscous behavior. Higher Tan δ values of the hybrid nanocomposites reflect an increase in energy loss due to internal friction between the polymer matrix and the embedded fillers. This heightened viscous behavior corresponds to the improved elasticity of the hybrid materials, indicating their capacity to withstand greater mechanical stresses while dissipating energy.

A Tan δ value close to or exceeding 0.5 highlights a superior viscoelastic balance, when the composite can deform elastically but also absorb and dissipate energy effectively. However, a reduction in Tan δ values towards zero would suggest diminished elasticity, as the material would localize the applied stress without adequately distributing or dissipating it. This localized pressure buildup could potentially lead to premature mechanical failure in applications demanding high energy absorption. The synergistic interaction of multi-walled carbon nanotubes (MWCNTs) and alumina within epoxy and polyamide matrices provides valuable insight into the correlation between the lowest recorded contact pressure in PCA and the highest Tan δ value of 0.5 observed in both ECA and PCA. While alumina is known for its high hardness, this property alone is insufficient to reduce contact pressure effectively, as the material must also dissipate localized stress to mitigate the effects of stress concentration. This phenomenon is crucial for the performance of hybrid composites under mechanical loading. The ability of the composite to dissipate energy can be attributed to the weak van der Waals forces between the GNP layers. When subjected to stress beyond a certain threshold, these layers tend to slide, facilitating the dissipation of stored energy, which reduces the buildup of localized stress. This sliding mechanism is particularly beneficial in preventing catastrophic failure, especially under conditions of dynamic loading.

Moreover, the Tan δ values across all tested samples exhibit a clear temperature dependence, increasing from 30 °C to 110 °C. At lower temperatures, the polymer chains within the epoxy and polyamide matrices are in a relatively rigid state, limiting their molecular mobility. This rigidity hampers the dissipation of stored energy, as internal friction within the material is minimized. Consequently, the material behaves more elastically at lower temperatures, with less energy lost through viscous dissipation. As the temperature increases, the polymer chains gain greater mobility, leading to enhanced molecular movement. This increase in molecular mobility results in a corresponding rise in internal friction, which facilitates the release of stored energy as heat. The elevated Tan δ values observed at higher temperatures reflect this transition, indicating that the material exhibits more viscous behavior, and more energy dissipation due to internal friction. This temperature-induced shift from predominantly elastic to viscoelastic behavior is crucial for understanding the mechanical response of these hybrid nanocomposites under thermal stress.

3.4 Mechanical characteristics

3.4.1 Tensile characteristics

The tensile properties of epoxy and polyamide 66 (PA66) hybrid nanocomposites were thoroughly evaluated, with the results illustrated in **Figure 5**. The findings show that the hybrid nanocomposites ECA and PCA demonstrate a notable enhancement in the tensile strength and modulus compared to their respective host matrices. This significant improvement in mechanical performance can be attributed to superior interfacial adhesion between the nanofillers and the polymer matrix, which promotes more efficient stress transfer during mechanical loading. The effective distribution of stress across the nanocomposite structure plays a pivotal role in reinforcing the mechanical integrity of the material. In contrast, other hybrid nanocomposites, such as ECG, EGA, PCD, and PGA, exhibit comparatively lower tensile values than ECA and PCA. This decline in performance can be linked to suboptimal dispersion of nanofillers within the matrix. The curing process and the influence of temperature during the extrusion process are critical factors affecting the distribution uniformity of nanofillers like GNPs, alumina, and carbon nanotubes. When the extrusion temperature is not adequately controlled, it can lead to agglomeration of nanofillers, thereby weakening the overall mechanical properties of the composite.

In addition to temperature, the structure and morphology of the nanofillers themselves also play a crucial role in determining the tensile behavior of hybrid composites. Fillers with higher aspect ratios, such as MWCNTs, tend to create more extensive reinforcement networks within the polymer matrix, leading to an enhanced load-bearing capacity. However, fillers with irregular shapes or lower aspect ratios may not interact as effectively with the matrix, resulting in weaker interfacial bonding and poorer stress transfer efficiency. The inferior dispersion of nanofillers in ECG, EGA, PCD, and PGA can thus be attributed to a combination of inadequate temperature control during processing and intrinsic characteristics of the fillers. These factors contribute to the formation of filler aggregates, which act as stress concentrators rather than reinforcing agents. The presence of these aggregates disrupts the homogeneity of the composite, leading to localized weak points that reduce the overall tensile strength and elasticity of the material. Therefore, to achieve optimal tensile properties in epoxy and PA66 hybrid nanocomposites, precise control of the processing conditions such as curing temperature, extrusion speed, and filler dispersion method is essential. Furthermore, careful selection of nanofiller types and their surface treatments can enhance interfacial interactions, ensuring that the mechanical properties of the composite are maximized for specific applications.

The epoxy-based nanocomposite ECA demonstrated a notable improvement in the tensile strength, reaching 97.2 MPa, with corresponding enhancements in the elongation at break by 14.2 %, and 17.6 % for ECG and ECA, respectively, compared to pure epoxy. Similarly, the PA66 nanocomposite PCA exhibited a comparable increase in the tensile strength, reaching 102 MPa, along with significant improvements in elongation at break by the same percentages when compared to pure PA66. The simultaneous enhancement in both tensile strength and elongation at break can be primarily attributed to the incorporation of oxide nanoparticles into the polymer matrix. The addition of these nanoparticles contributes to a significant enhancement in the material's mechanical properties by fostering stronger interactions between the nanoparticles and the polymer matrix. A key factor in these improvements is the large specific surface area of

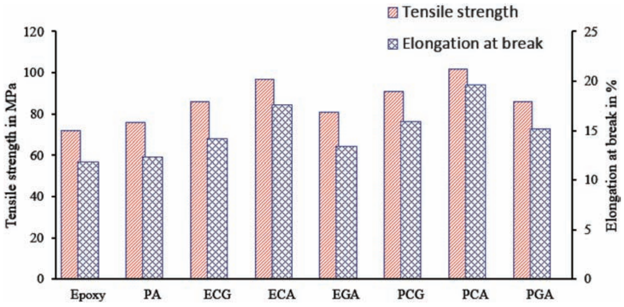

Figure 5: Tensile characteristics of epoxy, PA and their nanocomposites

the nanoparticles, which promotes extensive interaction with the surrounding polymer chains. This interaction leads to localized changes in the structural arrangement of the polymer chains, influencing the overall morphology of the matrix. The integration of nanoparticles can alter the morphological architecture of the polymer matrix, thereby affecting its mechanical performance. Factors such as the type, concentration, size, surface chemistry, distribution, and dispersion of the nanoparticles, as well as the properties of the immobilized polymer-nanoparticle interface, all play important roles in determining how the nanoparticles modify the matrix structure. The fractured surface morphologies of epoxy, polyamide and their hybrid nanocomposites are presented in **Figure 6**.

In nanocomposites like ECA and PCA, the observed enhancement in the tensile strength is indicative of a strong nanoparticle-matrix interface, which is essential for efficient stress transfer from the polymer matrix to the nanoparticles. The absence of a significant defect formation further supports the conclusion that the interfacial bonding between the matrix and the nanoparticles is robust, allowing a stable mechanical performance under load. This strong interface ensures that an applied mechanical stress is effectively transmitted from the matrix to the filler, resulting in an increase in the yield strength of the polymer nanocomposite. The improved stress transmission can be attributed to the effective dispersion of nanoparticles throughout the matrix, which reduces the likelihood of agglomeration and defects. The homogeneous distribution of nanoparticles creates a more uniform stress field within the matrix, thereby preventing the localization of stress that could lead to premature failure. Moreover, the surface chemistry of the nanoparticles plays a vital role in enhancing interfacial adhesion, further contributing to the improved mechanical properties of the nanocomposites.

3.4.2 Nanoindentation

Indent load versus indent depth curves for the test specimens are presented in **Figure 7**, illustrating the mechanical properties of the materials under investigation. The loading and unloading segments of the curve provide crucial insights into the hardness characteristics of the samples. A steeper slope during the loading phase indicates that a greater force is required to achieve the same penetration depth, thereby signifying higher hardness of the material. In this study, the hybrid nanocomposites PCA and ECA display the highest and lowest slopes, respectively. This stark contrast in the curvature illustrates a significant variance in hardness levels between the samples, with PCA demonstrating superior hardness compared to the more compliant ECA. During the indentation process, the retracting indenter experiences thermal drift, a phenomenon represented by the pop-out in the unloading curve. To mitigate the effects of thermal drift and ensure accurate measurements, a controlled approach is employed whereby the indenter is held at 90 % of the peak load until thermal stabilization

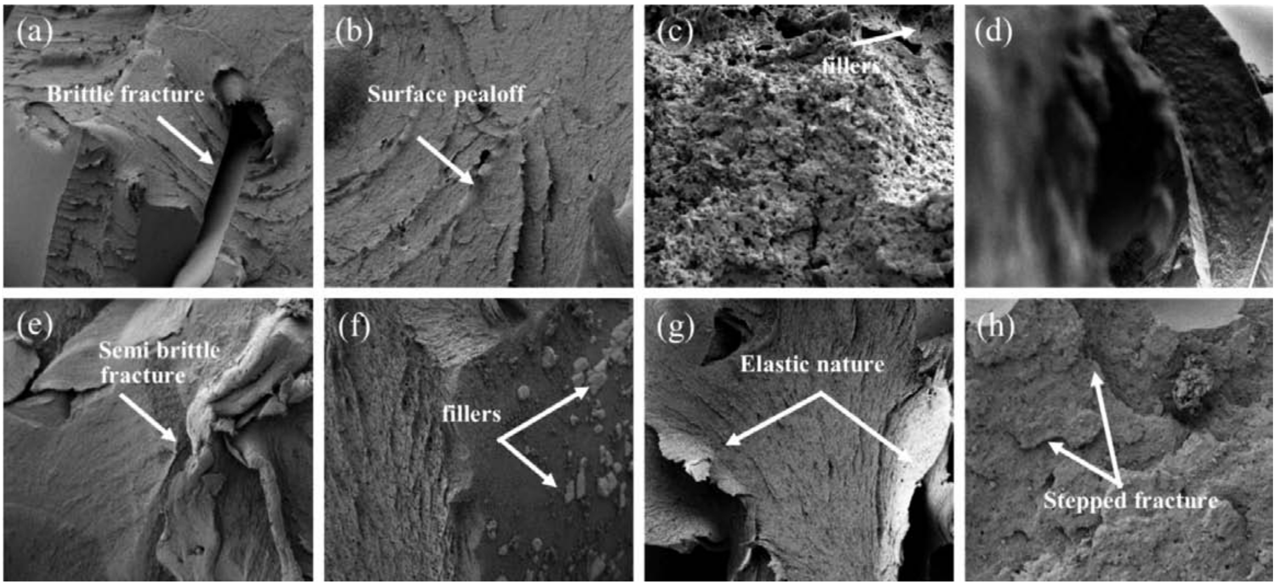

Figure 6: FESEM fractured surface morphology analysis of a) epoxy, b) ECG, c) ECA, d) EGA, e) PA66, f) PCG, g) PCA, h) PGA

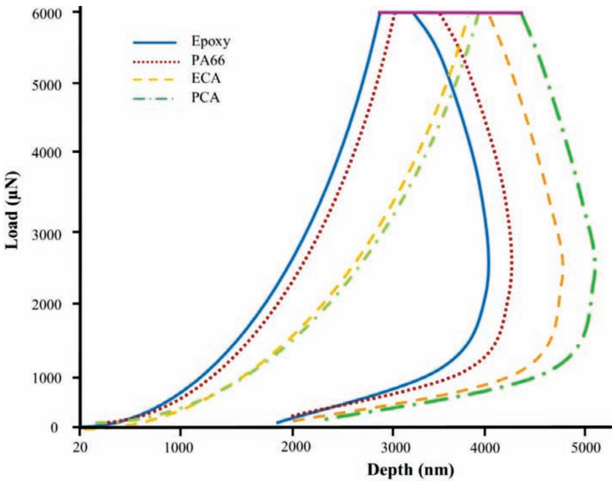

Figure 7: Load vs. depth of epoxy, PA66, ECA, and PCA hybrid nanocomposites

is achieved. This method is essential for minimizing heat generation due to friction between the indenter and the base material, which can otherwise skew the results. As the indenter penetrates the material, the interaction at the molecular level generates heat, potentially affecting the mechanical response of the polymer nanocomposites. In this investigation, the peak load was 6000 mN, with recorded indent depths for various materials: epoxy (3284 nm), PA66 (3491 nm), ECG (4314 nm), ECA (4434 nm), EGA (4271 nm), PCG (4361 nm), PCA (4492 nm), and PGA (4308 nm). These depth measurements are indicative of the material's resistance to deformation under load, providing quantitative evidence of the mechanical performance of each specimen. The observed depths reveal that epoxy and PA66 exhibit the greatest resistance to indentation, suggesting robust mechanical integrity, while the hybrid composites show varying degrees of indentation resistance, influenced by their respective filler compositions and interactions.

Distinct differences in indentation depths across the tested samples further elucidate the role of nanofiller dispersion and interfacial adhesion in enhancing the mechanical properties of the polymer matrices. This data underscores the importance of optimized processing techniques to achieve the desired hardness and mechanical performance in polymer nanocomposites. In conclusion, the findings from the load-displacement curves not only affirm the mechanical advantages of hybrid nanocomposites but also highlight the necessity of precise measurement techniques to accurately assess the hardness and mechanical characteristics crucial for applications in advanced materials engineering.

4 CONCLUSIONS

The ECA and PCA nanocomposites exhibited significantly reduced contact pressure compared to pure epoxy and PA66, with reductions of up to 68 % and 61 %, respectively, across different load levels, highlighting their superior performance in minimizing surface stress.

Both ECA and PCA demonstrated enhanced wear resistance, with wear rate reductions of up to 65 % for ECA and 76 % for PCA. They consistently showed the lowest wear volumes and contact pressures across all tested loads, confirming their improved load-bearing capacity.

The moderate interfacial bonding in the 0.2 GNP/0.2 alumina hybrid nanocomposite limited its load-bearing capacity, leading to suboptimal stress distribution. However, alumina played a crucial role in enhancing thermal stability and reducing surface deformation under high-friction conditions.

The epoxy-based nanocomposite ECA and PA66 nanocomposite PCA exhibited significant improvements in the tensile strength (97.2 MPa for ECA and 102 MPa for PCA) and elongation at break (14.2 % for ECA and 17.6 % for PCA) compared to their pure counterparts. These enhancements are primarily due to superior interfacial adhesion between nanofillers and the polymer matrix, which facilitates efficient stress transfer during mechanical loading.

The Tan δ analysis indicated that epoxy and PA66 exhibited more elastic behavior with lower Tan δ values, while hybrid nanocomposites (ECA and PCA) had higher Tan δ values, reflecting increased viscous behavior and better energy dissipation at elevated temperatures.

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