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Research article

Exploring the Tribological Response of Graphene Reinforced Glass – Epoxy Nano Composites: Effect of Process Parameters

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A B S T R A C T

The effect of experimental parameters on the sliding wears behavior of Glass fabric -epoxy filled with nano Graphene has been studied. The effect of higher loading of nano graphene (2wt%) on the tribological response of Glass - epoxy composites under the influence of velocity and sliding pressure is investigated. These composites were developed using vacuum bag moulding method. The dry sliding wear test has been carried out according to ASTM G99 standards. The result shows the effect of higher loading of nano graphene has appreciably enhanced the wear resistance of the nano composites, but it reflects the brittle nature. Further, the structural stability of nano composites was strongly supported by high modulus glass fabrics. The reinforcement of nano graphene has effectively reduced the stress concentration thereby avoiding the wearing of glass fabrics. The impact of sliding velocity has severe effect than load. The SEM photographs reflect the fatigue loading, slight agglomeration and uniform scattering of nano particles across the matrix zone.

1. INTRODUCTION

In Today's scenario industry needs materials which posses excellent properties which can be easily tailored to meet needs of industry. Polymers are one such class of materials which exhibit excellent characteristics and properties when they are reinforced with some other materials like the glass fiber it gives strength and stability to these composites to make them excellent materials for various applications. among various polymers thermo sets plays an important class of materials which meets all the requirements such as light weight, chemical stability, better thermal properties, but exhibits poor wear resistant properties. The addition of fillers in micro and nano scale enhances the sliding wear properties Wear behavior of these polymeric materials M.

Sudheer et.al [1] in their findings showed that addition of PTW and graphite(Gr) improved wear behavior of epoxy composites. They proved that tensile properties of epoxy/glass composites mproved with addition of graphite further there was slight improvement in tensile behavior with ceramic filler addition. the flexural strength was improved with the addition of graphite, impact and hardness followed the same trend, the wear behavior of these composites was evaluated by determining coefficient of friction and specific wear rate, it is observed from results that, there is a slight decrease in COF with addition of ceramic filler and Gr, and there is drastic decrease in COF, with addition of both the fillers, the specific wear rate decreased under different load and velocity. SEM images of worn surfaces are used to analyze sliding wear behavior of the test specimens Anuja.H et.al [2] studied mechanical and tribological properties of epoxy composites with Ws_2 and Zro_2 as fillers using taguchi technique they revealed that, with 12.5wt% of Zro_2 and 4wt% WS_2, optimum mechanical properties were achieved, L16 orthogonal array was used for the analysis of tribological behavior of composites, with 4parameters and 4levels .the analysis show that the most influencing parameter on wear rate is time, followed by velocity, filler content and load. Patil and Prasad [3] designed a polymer composite material for sleeve bearing applications and analyzed wear and frictional properties of epoxy reinforced with short fibers and graphite, it is noticed that wear and frictional coefficient reduced with increase in load, sliding distance and velocity, Shen et.al [4] in their publication studied modification in tribological performance with low GO content of 0.5%, wear resistance of these composites is improved, compared to reinforcement of other fillers, Go reinforcement shows better results. To determine particle size and dispersion of graphene and GO XRD analysis is carried out. The peaks appears at around $2\theta=10.2^0$ and $2\theta=25^0$-27.5^0 showing the presence of GO and Graphite respectively, Further SEM and TEM images were captured for analysis of worn surfaces. Rashmi et.al [5] in their research findings revealed that with addition of organo-modified montmorrillonite into epoxy matrix enhance wear behavior under dry sliding wear they used taguchi approach with L9 array to investigate tribological behavior the experimental data shows that with addition of 5 wt% of OMMT nano filler improved wear resistance SEM images were used to analyze wear mechanism and correlate test results. They has selected 4 parameters with each parameter having 3 levels with characteristic lower the better (sliding wear) the response of these experiments with taguchi approach concludes that among the parameters considered sliding distance is most effective parameter, further, normal load and filler content follows, finally sliding velocity has least or no significant effect on wear behavior of epoxy composites. Yadav et.al [5] have presented the behavior of epoxy glass composites with or without addition of silica on the mechanical and sliding wear behavior of glass reinforced epoxy composites silica is varied from 5-15wt% in steps of 5, it is observed that with addition of silica sliding wear, hardness, flexural strength is increased and tensile strength, impact strength has reduced. From the study of sliding wear behavior under different loads, it is found that addition of silica improved the wear resistant properties and Sevier wear happens due to catastrophic failure of silica particles, it is also observed from SEM analysis, wear was predominant at lower and higher loads. Yadaw et.al [6]in their research article investigated the mechanical and sliding wear behavior with addition of silica, the epoxy glass fiber composites were prepared by hand layup process mechanical and wear behavior of these composite were studied with/without addition of silica it is observed that both mechanical and wear properties were enhanced with addition of silica, Kurahatti et.al [7] in their investigation they used high shear mixing process to disperse Zro_2 in nano size in 0.5-10wt% into epoxy resin, the experimental results revealed that the dispersion of nano particles improved flexural strength and flexural modulus, however, there is decrease in impact strength with increase in nano filler content, which in turn increase the wear resistance properties.

2. MATERIALS AND MATERIAL FORMULATION

2.1 Materials

Materials used for the study are Lapox L12 epoxy with computability hardner K-6, purchased from Yuje enterprises Bangalore and Glass fabric 360GSM purchased from scinetech technologies the nano graphene functional grade purchased from graphite India, Bangalore, the composites are fabricated by hand layup followed by vacuum bagging technique, the specimens were prepared as per ASTM standards using abrasive water jet machining. the specimens for wear test are prepared according to G99 standards with dimensions 6mm X 6mm X 3mm the wear test is conducted using Ducon pin on disc machine. The materials used for the formulation of composites with their properties are listed in Table 1.

Table 1. The materials with their properties.

Material	Density gr/cc	Melting point °C	Form
Epoxy	1.202	75-82	Liquid
Glass Fabric	2.57	2450	Fiber
Graphene	0.7	3560	Amorphous

We have considered four different material systems to know the effect of different reinforcements, the formulation are as follows I Neat epoxy 2 Epoxy with glass fabric 3 glass epoxy with 1wt% of nano graphene filler and 4 glass epoxy with 2wt% nano graphene filler.

Table 2. Material formulation with their proportion.

Material ID	Epoxy	Glass Fiber	Graphene
Pure epoxy/Neat epoxy(EP)	100		
Glass epoxy (GE)	67	33	
Glass epoxy with 1wt% Graphene (GE1)	66	33	1
Glass epoxy with 2wt% of graphene (GE2)	65	33	2

2.2 Wear test

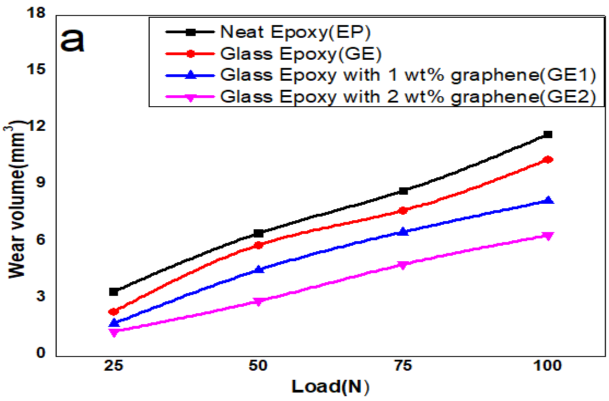
The wear test specimens of size 6mmX6mmX 3mm thick are cut as per ASTM G99 standards using abrasive water jet machining process, the sliding wear test is conducted on Ducom pin on system disc machine. the specimens are fastened to specimens holders of 8mm diameter with gel, . the test is conducted using weight loss method weight of the specimen along with specimen holder is weighed using four digit accuracy weighing balance, before and after the conduction of the test to record weight loss. The wear test is with the normal load, sliding velocity, and sliding distance as parameters. The test is carried with varying the load from 25 to100N in steps of 25N sliding velocities from 0.5 m/s to 2 m/s in steps of 0.5m/s and sliding distance varying from 1000 to 4000m in steps of 1000m respectively. The friction force for each trial is also recorded, the steel disc is cleaned with acetone before and after each trail to clean the disc surface, results are average of two specimens were recorded and tabulated.

3. RESULTS AND DISCUSSION

The test results obtained with variation of load at a sliding velocity of 0.5m/s and sliding distance of 1000m are tabulated in Table 3 and responses are plotted in Fig. 1.

Table 3. Wear test data of composites at different loads.

Material	Load(N)	Wear Volume (mm^3)	Specific wear rate X 10^{-4} (mm^3/N-m)
Neat epoxy(EP)	25	3.41431	74.194
	50	6.49252	63.052
	75	8.7371	52.210
	100	11.71714	43.508
Glass epoxy (GE)	25	2.35135	65.352
	50	5.86777	53.507
	75	7.698	45.562
	100	10.39953	35.256
Glass epoxy+ graphene with 1wt% (GE1)	25	1.47137	57.352
	50	4.5425	44.563
	75	6.56062	35.659
	100	8.21091	24.356
Glass epoxy + graphene with 2wt% (GE2)	25	0.90456	55.652
	50	3.90667	41.262
	75	4.84347	32.952
	100	6.379	21.367



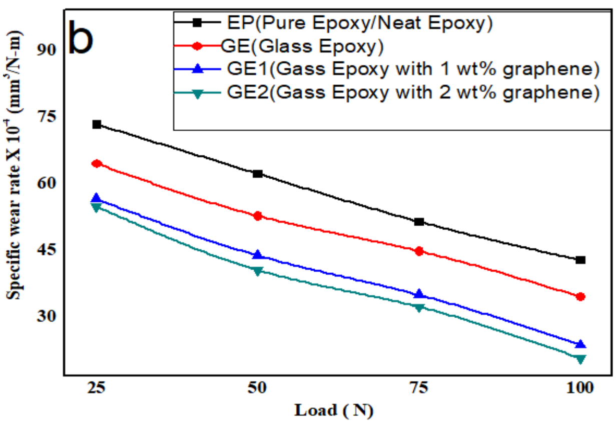


Fig. 1. Plots for variation of (a) wear volume V/s Load, (b) Specific wear rate V/s Load.

The tribological response is analyzed by the evaluation of the wear volume loss and specific wear rate. It is observed from the test data, that wear volume loss increases, with increase in load and specific wear rate decreases with increase in load, neat epoxy shows a wear volume loss of 3.41431mm^3 at a lower pressure of 25N and wear volume loss increases to 11.7171mm^3 with an increase of 243.17%, the same trend follows for the remaining material systems but with incorporation of glass fabric into epoxy matrix improves, the wear volume loss, under a load of 100N, the epoxy shows wear volume loss of 11.7171mm^3 but with addition of glass fabric wear volume loss is reduced to 10.3995mm^3, a reduction of 11.25% is noticed. Further, addition of 1wt% of graphene in to glass epoxy system reduced wear volume loss by 21%, the higher loading of graphene(2wt%) into glass epoxy system, further reduces wear volume loss by 38.66%, this wear loss with increase in normal load is attributed to, increase in shear force and frictional resistance, [1-5], with increase in applied normal load, the lower wear rates is exhibited by graphene reinforced composites compared to glass filled and neat epoxy system, with variation in load, speed and sliding distance is due to the fact that, the presence of graphene acts effective barrier to prevent fragmentation of epoxy, most of researchers reveal that [8], graphene acts as effective filler material in enhancing the tribological behavior of epoxy matrix composites, carbon as graphene provide lubricating effect thus enhancing wear resistance. Further, wear behavior also depends on surface temperature [9], the increase in surface temperature leads to thermal softening. The specimen temperature increases with increase in load, speed and distance, as the interface temperature is increased, the pulverized carbon particles becomes harder and steel counter face becomes softer, the hardened carbon particles plough disc surface, leaving rough surface [10,11], carbon retains its strength at high temperature but steel can't retain its temperature at high temperatures.

The enhancement of wear resistance due to reinforcement of graphene, can be analyzed by studying the atomic structure of graphene atoms, they are arranged in hexagonal in planar condensed ring with covalent bond [12,13] but layers are bonded by weak vander wall's forces, this anisotropic nature, results in two types of bonds in different directions, these contrasting chemical bonds results in the formation of lubricant layer, thus reducing wear loss of samples [14-17], further the weak Vander wall's forces causes individual layers to slide one over other making ideal lubricating layer, thus reducing the wear loss. Fig. 1(b) Fig. 2(b) and Fig. 3(b) shows the variation of specific wear rate Fig. 1(b) & Fig. 3(b)shows variation of specific wear rate with varying load and distance it is observed from the graph that specific wear rate shows a decreasing trend with increase in load and distance but increasing trend with increase in velocity, the similar trend is reported by many researchers, the common mode of wear in glass epoxy composites is, wear of matrix, which includes cracks in matrix and plastic deformation.

Table 4. Wear data for varying velocity.

Material ID	Velocity (m/s)	Wear volume loss (mm^3)	Specific wear rate X 10^{-5} (mm^3/N-m)
Neat Epoxy (EP)	0.5	20.05	20.35
	1	25.36	30.28
	1.5	50.26	50.72
	2	73.46	79.15
Glass Epoxy(GE)	0.5	12.26	16.72
	1	19.35	22.35
	1.5	37.28	38.1
	2	62.75	68.39
Glass Epoxy with 1wt% graphene (GE1)	0.5	8.26	11.21
	1	12.35	17.33
	1.5	26.35	26.75
	2	48.49	42.42
Glass Epoxy with 2wt% graphene (GE2)	0.5	4.46	7.65
	1	8.73	13.37
	1.5	17.27	20.38
	2	27.36	27.36

Other mode refers to wear in fibers, such as fiber cracking, rupture and pulverizing [18], the reduction in specific wear rate is due to formation of transfer film on the counter face. The Table 5 shows the wear data for varying sliding distance the sliding distance is varied from 1km to 4km, wear volume increases with increase in sliding distance, where as specific wear rate decreases with increase in sliding distance, this trend is same for all material systems however, with addition of glass fabric and nano graphene the material system shows improvement in both wear volume and wear rate.

Table 5. Wear data for varying sliding distance.

Material ID	Sliding distance(m)	Wear volume (mm^3)	Specific wear rateX10^{-5} (mm^3/N-m)
Neat Epoxy(EP)	1000	20.23	20.15
	2000	34.27	15.27
	3000	55.37	11.69
	4000	79.37	9.95
Glass Epoxy(GE)	1000	15.13	14.89
	2000	21.43	11.97
	3000	34.48	9.98
	4000	58.78	8.95
Glass Epoxy with 1 Wt% Graphene (GE1)	1000	9.83	12.75
	2000	14.35	9.25
	3000	19.45	7.52
	4000	29.85	5.75
Glass Epoxy with 2Wt% Graphene (GE2)	1000	6.75	10.15
	2000	9.78	7.1
	3000	13.33	5.43
	4000	16.73	4.75

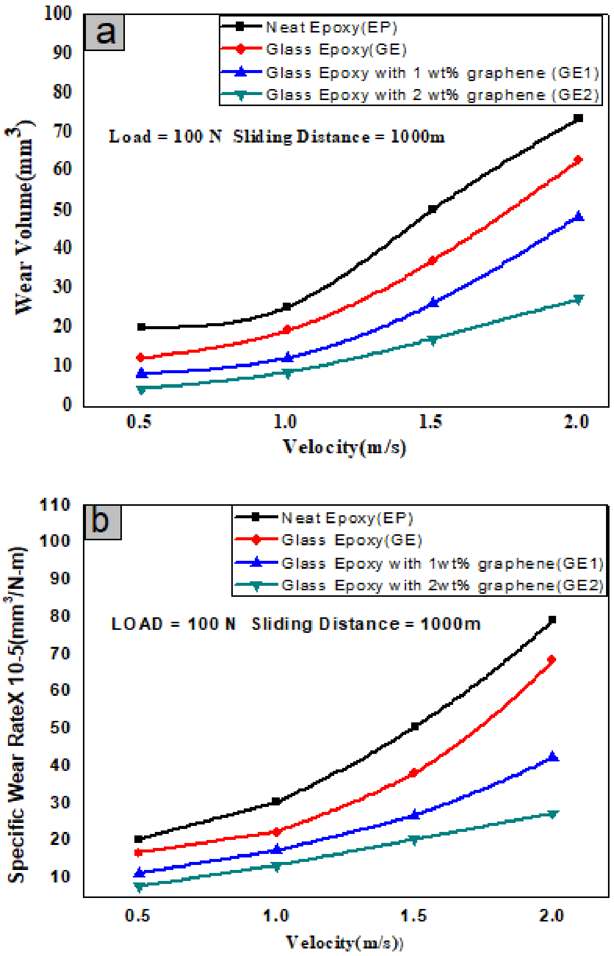


Fig. 2. Plot of variation of (a) wear volume v/s velocity and (b) specific wear rate v/s velocity.

The plot for variation of wear volume loss and wear rate with varying sliding distance at a sliding velocity of 0.5m/s and normal load of 100N is shown in Fig. 3(a) & Fig. 3(b).At lower sliding distances, lesser wear volume loss is encountered but wear volume increases with increase in sliding distance the wear loss is severe at higher sliding distances this improvement in tribological behavior is due to synergism [10,16] between the graphene and matrix, at sliding distance of 1000mm an decrease of 47.89% wear rate is observed, similarly at a sliding distance of 4000mm a decrease in specific wear rate of about 52.26% is noticed, with the addition of 2 wt % of nano graphene.

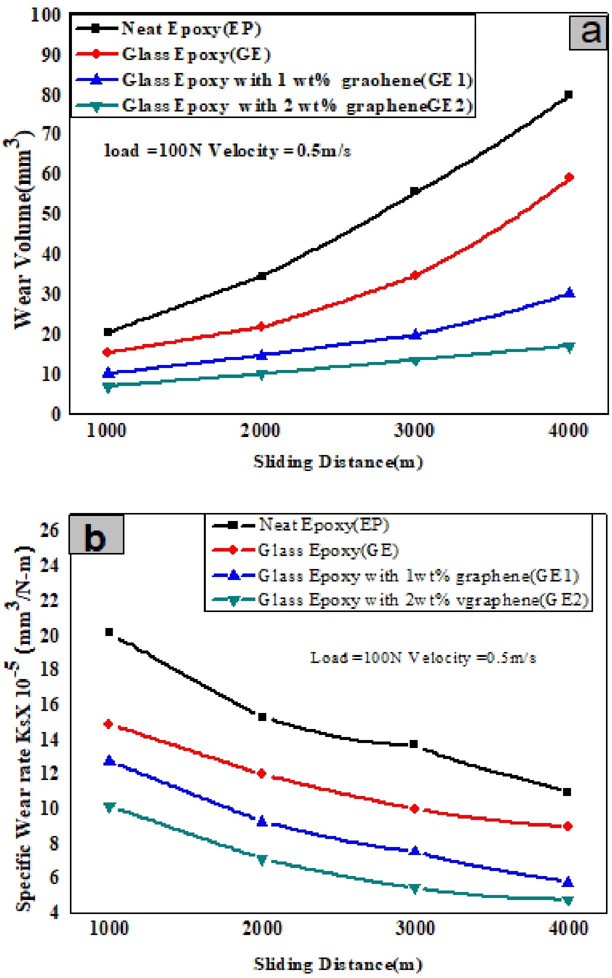


Fig. 3. Plots for variation of (a)wear volume V/s distance (b)Specific wear rate V/s distance.

This improvement in wear resistance is due to the presence of nano graphene particles, which inhibit fragmentation of polymer matrix, mean while graphene addition changes topography of composite surface. The interlocking between pin and disc is reduced due to formation of smooth surface, the friction coefficient is also reduced due incorporation of graphene, as a result of reduced contact area between counter parts. Further, polymers forms polymer chains entanglement and nano fillers, graphene filled composites shows better wear resistance compared to neat epoxy, because of the triggering of wear mechanism, as a result of formation and detachment of new layers and particles [14,19] on the surface of the test specimen, the flakes and tiny particles are unstable and get accommodated with sliding pairs.

In dry sliding conditions wear resistance and COF[14,20] of pure epoxy at lower loads, velocities and sliding distances shows variable results due to existence of adherent layer over the contact surface, at lower loads these adherent particles sometimes gather near the wear track due to ploughing action, the small applied load is enough to initiate crack in the subsequent cycles, this crack grows increasing the load further, to some extent the wear rate decreases as a result of surface smoothening process, the graphene filled composite shows lesser wear rate due to the lubricating action of graphene flakes and decrease of intensity of furrows, some pits and spoliation, occurs [21,22] on wear track, however, the effect of it is minimized by the formation of lubricant layer by the graphene, it is observed that small percentage variation in particles will enhance the wear characteristics of composite system. Graphene acts as solid lubricant to reduce COF and wear rate, the wear rate reduces with excess loading of graphene. Micrographs of worn surfaces and wear debris of epoxy composites are shown in Fig. 4, Fig. 5 and Fig. 6 we can clearly observe that neat epoxy shows severe platelets and micro sized debris due to peeling off of the soft surface while for epoxy filled with different wt% of nano graphene [15] exhibits uneven wear debris are small uniform sized with milder wear surface this is due the presence of GO in oxy matrix having poor dispensability and interfacial adhesion as loading of graphene increases, the mechanical and tribological properties increases along the sliding direction, the debris becomes small forming a moderate scratch along the sliding direction. The friction, wear mechanism changes to combined adhesive and plow wear, Graphene as a good filler reinforcement & lubricant enhances the tribological behavior of composites under sliding wear behavior.

Fig. 2 to Fig. 5 shows worn surfaces of the samples at room temperature wear track for Epoxy is wider than the wear track of other material systems this is because [16] the wear will be severe in absence of micro and nano size particles coarse scuff is visible in epoxy surface since epoxy is brittle in nature the wear mechanism between epoxy and steel disc is of abrasive nature cracks perpendicular to sliding direction are also seen in micrographs

Fig. 4 to 7 shows the worn surface features of the sliding wear test specimens with varying load, velocity and Sliding Distance SEM pictures are recorded at Load of 25N.100N velocity of 0.5m/s and 2 m/s and with sliding distance of 1km and 4km It is observed from the micrographs that at lower load, velocity and distance smooth surface with micro cracks is formed at higher load and velocities there is increase an in temperature and wear rate the rise in temperature causes accelerated damage of matrix at interfacial region from the SEM images it is evident that the fiber detachment has reduced with addition of nano graphene as secondary filler at higher load, velocity and distance wear surface is smooth and followed by micro surface damage due to fatigue wear occurring at high temperatures removing surface by micro cracks the debris from graphene reinforced composites is much thinner compared to glass fabric filled composites further with addition of graphene (fibers) fabric is maintained in epoxy matrix with gradual wear conditions at higher load and velocities the higher loading of graphene into glass fabric filled epoxy composites shows better wear properties. Additional lubricants contribute to better wear resistance the transfer of graphene from composite to disc surface is observed. Smaller size of wear debris formed in graphene filled composites this demonstrates the role of micro/nano fillers in impending the large scale defragmentation process the enhanced sliding wear behavior of graphene filled composites depends on several factors such as increased bonding strength between fiber and matrix, lesser voids creation due to reinforcement of nano fillers.

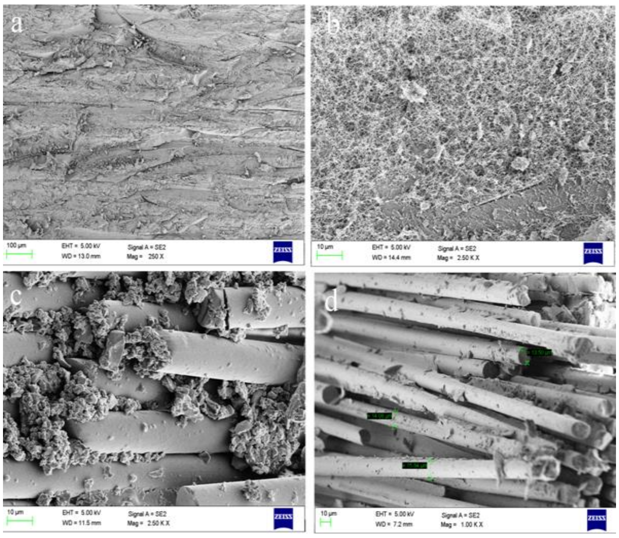


Fig. 4. SEM images at different loads(a)EP 25N, (b) EP 100N, (c) GE25N, (d) GE100N.

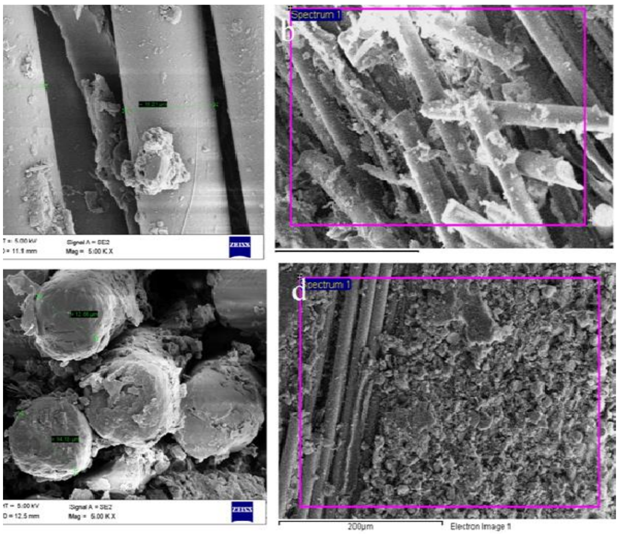


Fig. 5. SEM images at different loads (a) GE1 25N, (b) GE1 100N, (c) GE2 25N, (d) GE2 100N.

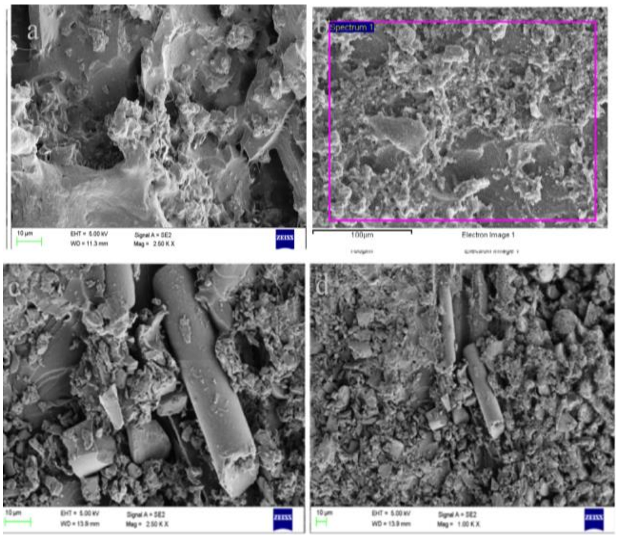


Fig. 6. SEM images at different velocities (a) EP 0.5m/s, (b) EP 2m/s, (c) GE 0.5m, (d) GE 2 m/s.

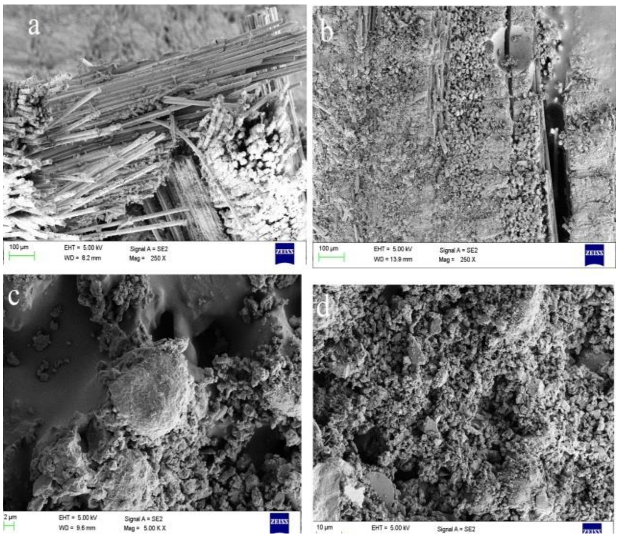


Fig. 7. SEM images at different Velocities (a) GE1 0.5m/s, (b) GE1 2m/s, (c) GE2 0.5m/s, (d) GE2 2m/s.

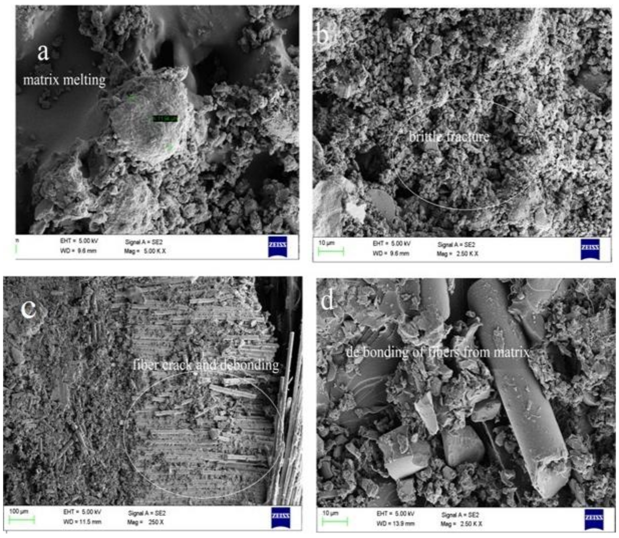


Fig. 8. SEM images at different sliding distances (a) EP 1000m, (b) EP 4000m, (c) GE 1000m, (d) GE4000m.

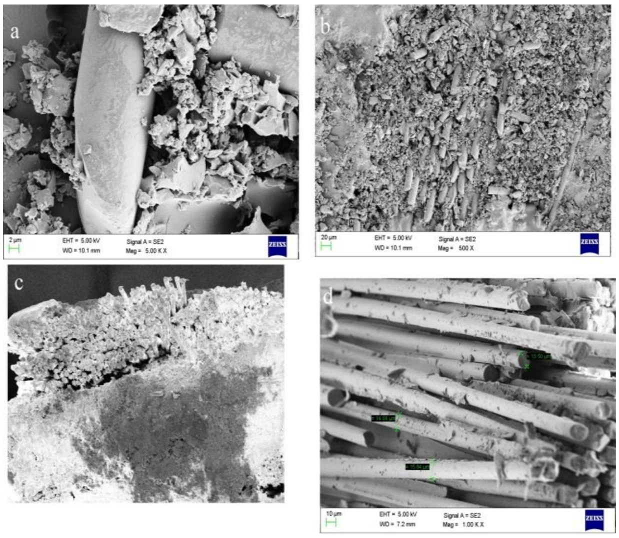


Fig. 9. SEM images at different velocities (a) GE1 1000m, (b) GE1 4000m, (c) GE2 1000m, (d) GE2 4000m.

4. CONCLUSIONS

1. The glass fabric gives structural integrity to the polymer, thus enhancing the tribological properties.
2. Loading of nano graphene into epoxy resin further improves the tribological responses of the formulated composites.
3. Higher loading of nano graphene has a positive response on wear behavior of glass epoxy composites.
4. Wear volume loss increases with increase in experimental parameters such as sliding pressure velocity and distance, where as specific wear rate decreases with increase in load & velocity but increases with increase in sliding distance.
5. Study of SEM micrographs shows the different modes of failure such as micro cracking.
6. The presence of graphene acts effective barrier to prevent fragmentation of epoxy.
7. Tearing of glass fabric, wear track, furrows are some of the failures noticed from the experimental results.

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