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Study and Analysis on Mechanical and Wear Behavior of SiC Filled Epoxy Composite

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Abstract: **Silicon carbide possesses ample reinforcing potential to be used as a filler material in polymer matrix composites. Successful fabrication of epoxy matrix composites reinforced with silicon carbide particles is possible by simple hand-lay-up technique. These composites possess very low amount of porosity and improved micro-hardness, also it provide slightly superior tensile, flexural and inter-laminar shear strengths than those of the neat epoxy. This study reveals that silicon carbide possesses good filler characteristics as it improves the sliding wear resistance of the polymeric resin. Dry sliding wear characteristics of these composites have been gainfully analysed using a design-of-experiment approach based on Taguchi method. The analysis of experimental results shows that factors like filler content, sliding velocity and normal load, in this sequence, are identified as the significant factors affecting the specific wear rate of the composites under investigation. The silicon carbide-epoxy composites fabricated and experimented upon in this investigation are found to have adequate potential for a wide variety of applications particularly in wear prone environment. When wear is not the predominant degrading factor, epoxy without silicon carbide can be recommended. However, the weight fraction of filler in the composite is to be decided from the view point of required strength. If the place of use is hostile with sliding wear situations, then silicon carbide epoxy composites are to be preferred due to their fairly good wear resistance. Use of these composites may be suggested in applications like engineering structures in dusty environment and low cost building materials in desert.**

Keywords: **SiC- Silicon Carbide, Dry Sliding wear, Epoxy resins and composites, pin on disc apparatus, SEM Micrograph.**

I. INTRODUCTION

Composite materials were developed because no single, homogeneous structural material could be found and had all of the desired attributes for a given application. Mud and straw combined best use in the form of bricks it's an archaic composite materials for building construction. Brick-making process can still be seen on Egyptian tomb paintings in the Metropolitan Museum of Art. The most advanced examples perform routinely on spacecraft in demanding environment. Composites are made up of individual materials referred to as constituent materials. There are two categories of constituent materials in composite: matrix and reinforcement. At least one portion of each type is required to make composite. The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcements impart their special mechanical and physical properties to enhance the matrix properties.

Epoxy is a category of polymers which contain the ether as functional group in their main chain. The term unsaturated epoxy resin is generally referred to the unsaturated (means containing chemical double bonds) resins formed by the reaction of dibasic organic acids and polyhydric alcohols. Its common name is Bisphenol-A-Diglycidyl-Ether. Epoxy resin is also known as a thermosetting plastic, which implies the plastic sets at high temperatures as opposed to thermoplastics which can be formed at high temperatures. Epoxy resin can attach things together to itself, creating a strong bond. Epoxy resins can be formulated to obtain a wide range of properties ranging from soft and ductile to hard and brittle. Their advantages include low viscosity, low cost, and fast cure time. In addition, epoxy resins have long been considered least toxic. In addition to that they have low shrinkage upon curing and good chemical resistance. Due to several advantages over other thermoset polymers, epoxy (LY 556) is chosen as the matrix material for the present research work. Silicon carbide (SiC) is silicon based ceramic filler. Silicon carbide is composed of tetrahedral of carbon and silicon atoms with strong bonds in the crystal lattice. This produces a very hard and strong material. It is not attacked by any acids or alkalis or molten salts up to 800°C. The high thermal conductivity coupled with low thermal expansion and high strength gives this material exceptional thermal shock resistant qualities. Silicon carbide has low density of about 3.1 gm/cc, low thermal expansion, high elastic modulus, high strength, high thermal conductivity, high hardness, excellent thermal shock resistance and superior chemical inertness. Experience on the use of silicon carbide as a filler in polymer matrices is very limited. In the best case, investigations have been performed with micro sized aggregates of nano-particles, because the filler has been added to the resin in the form of a powder. The reinforcement of the epoxy matrix by silicon carbide particles is especially attractive, because these fillers increase the strength and thermal stability of the material and impart resistance to corrosive media, except for strongly alkaline media. Silicon carbide are mainly used as a conductive filler in microelectronics application because of it high thermal conductivity and low electrical conductivity. Various thermal properties like thermal conductivity, coefficient of thermal expansion, glass transition temperature were studied by Zhou et al. [21] and found improvement in all such property because of incorporation of SiC. Apart from thermal studies, some mechanical and electrical work are also been reported. Wenying et al. studied the mechanical and dielectric property of SiC reinforced polymer composite when polymer is low-density polyethylene composites. They also stated the improvement in thermal conductivity of LLDPE to 1.48 W/m-K when 55 wt % of SiC is added and at the same time not much increase in the value of dielectric constant is reported. Though substantial amount of work has been reported on many other properties but wear characterization of such SiC filled epoxy composites is not yet reported. In view of this, wear characterization of such silicon carbide reinforced epoxy composites is investigated in present work together with its physical, morphological and mechanical behaviour.

II. EXPERIMENTAL DETAILS

2.1. Composite Fabrication:

Micro-sized silicon carbide is reinforced in unsaturated epoxy resin in six different weight proportions (0 wt %, 10 wt %, 20 wt %, 30 wt %, 40 wt % and 50 wt %) to prepare the composite specimens. The composite slabs are made by conventional hand-layup technique. Then as per recommendation, 10% by weight, Triethylene tetramine (TETA, HY 951) as hardener is mixed in the resin. Care has been taken to avoid formation of air bubbles. Pressure was then applied from the top and the mold was allowed to cure at room temperature for 24 hrs. During application of pressure some amount of epoxy and hardener squeezes out. Proper care has been taken to consider this loss during manufacturing so that a constant thickness of sample can be maintained. The other composite samples with particulate fillers of fixed weight percentage are fabricated by the same technique. Specimens of suitable dimension are cut using a diamond cutter for physical characterization and mechanical testing. A plastic mold of dimension (150×60×3) mm was used for casting the composite sheet. The various test samples according to the ASTM standards are than taken out from the fabricated sheet. Further the test has been performed for all the composition.

Fig. 1 Epoxy matrix and its corresponding hardener

2.2. Sliding wear test:

A pin-on-disc sliding wear test setup (supplied by DUCOM) is employed for evaluating the performance of the samples. The test is carried out as per the ASTM G99 standard for dry sliding wear of polymer composites. The counter body is a disc made of hardened alloy steel with hardness value of 72 HRC and surface roughness (Ra) of 0.6 µm. The specimen is held stationary against the rotating steel disc and the normal force is applied through a lever mechanism. The specimen is held stationary and the disc is rotated while a normal force is applied through a lever mechanism. Four sliding velocities of 100, 200, 300 and 400 cm/s under four different normal loading of 5N, 10N, 15N and 20 N are used for conducting wear tests. The dimension of the square shaped samples for the test is 10 mm × 10 mm and this sample piece is glued to one end of a mild steel cylindrical pin of 9 mm diameter and 120mm length. The surfaces of both the sample and the disc are cleaned with a soft paper soaked in acetone and thoroughly dried before the test. The pin with sample assembly is weighed in a precision electronic balance to an accuracy of ±0.1mg. The difference between the initial and final weights of the assembly is the *sliding wear loss* or the mass loss of the specimen under sliding. The specific wear rate can be defined as the volume of material removed per unit load per unit sliding distance and it can be expressed in terms of 'volume loss' basis as:

$$W_s = \frac{\Delta m}{\rho t} V_s F_n$$

Where,

Δm is the mass loss (gm), ρ is the density of the sample (gm/mm^3), t is the test duration (second), Vs is the sliding velocity (m/s) and F_n is the normal load (N).

2.3. Mechanical Characterization:

Measurement of density is done using- simple water immersion technique (ASTM D 792-91). Morphology of the particulates together with its dispersion characteristics in the matrix body have been studied using a scanning electron microscope JEOL JSM-6480LV. Micro-hardness measurement is done using a Leitz micro-hardness tester (ASTM E384). A diamond indenter, in the form of a right pyramid with a square base and an angle 136^0 between opposite faces, is forced into the material under a load *F*. The tensile test (ASTM D3039-76) and compressive test (ASTM D695) are performed using a Universal testing machine Instron 1195. Flexural strength is evaluated using three point bend test.

Table 1: Control factors and their selected levels used in sliding wear test

Command factors	**Level**				
	I	**II**	**III**	**IV**	**Units**
Factor A : Sliding velocity	100	200	300	400	cm/sec
Factor B : Normal load	5	10	15	20	N
Factor C : Silicon carbide content	0	10	30	50	wt. %
Factor D : Distance of sliding	1200	1600	2000	2400	M

2.4 Experimental Design:

Statistical methods are commonly used to improve the quality of a product or a process. Such methods enable the user to define and study the effect of every single condition possible in an experiment where numerous factors are involved. Wear processes such as sliding and/or erosion are such processes in which a number of control factors collectively determine the performance output i.e. the wear rate. Hence, in the present work a statistical technique called Taguchi method is used to optimize the process parameters leading to minimum wear of the composites under study. The experimental observations are further transformed into signal-to-noise (S/N) ratios. There are several S/N ratios available depending on the type of characteristics as given by following equations:

'Smaller- the- better' characteristic: $\frac{S}{N} = -10\log\frac{1}{n}\left(\sum y^2\right)$ (2.4.1)

Table 2: Taguchi orthogonal array design (L_{16}) for dry sliding wear test

Test run	Factor A Sliding velocity	Factor B Normal load	Factor C Silicon carbide content	Factor D Sliding distant
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	1	4	4	4
5	2	1	2	3
6	2	2	1	4
7	2	3	4	1
8	2	4	3	2
9	3	1	3	4
10	3	2	4	3
11	3	3	1	2
12	3	4	2	1
13	4	1	4	2
14	4	2	3	1
15	4	3	2	4
16	4	4	1	3

‘Nominal- the- better’ characteristics:

$$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum\frac{\overline{Y}}{S_y^2}\right) \qquad (2.4.2)$$

‘Larger- the- better’ characteristics:

$$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum\frac{1}{y^2}\right) \qquad (2.4.3)$$

Where, ‘n’ is the number of observations and ‘y’ is the observed data. The S/N ratio for minimum wear rate comes under ‘smaller is better’ characteristic, which can be calculated as logarithmic transformation of the loss function by using Equation (2.4.1).

III. RESULTS AND DISCUSSION

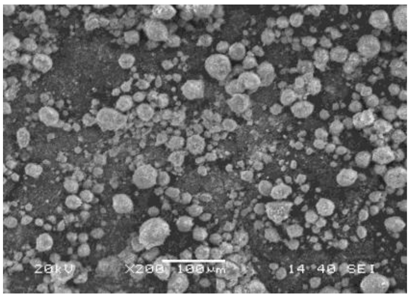
3.1 Mechanical Properties:

The theoretical and measured densities along with the corresponding volume fractions of voids are presented. It is clear from table that in composite contains 10 wt % filler, the volume fraction of voids is negligible and this is due to the small quantity of particulate fillers. With the addition of filler material more voids are found in the composites. As the filler content increases from 10 wt% to 20 wt% the volume fraction of voids is also found to be increasing. This trend is observed in the complete range of the filler. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering.

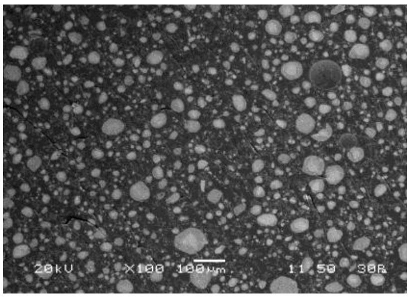
Table 3: Density and void fraction

S. No	Filler Content (wt %)	Measured density (gm/cm^3)	Theoretical density (gm/cm^3)	Voids (%)
1	0	1.1	-----	-----
2	10	1.15	1.176	2.21
3	20	1.22	1.263	3.40
4	30	1.30	1.364	4.70
5	40	1.39	1.483	6.27
6	50	1.49	1.624	8.25

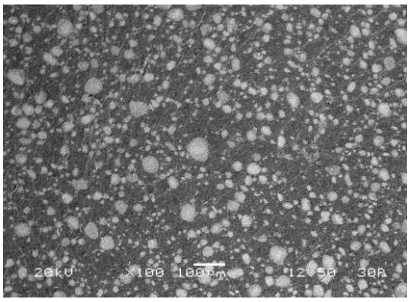
Various physical, mechanical and tribological properties of particulate filled polymer composites are strongly affected by the compatibility between the organic and inorganic phase. The scanning electron microscope images of the SiC micro size particles, the surface morphologies of epoxy/SiC composites for different filler content are shown in figure 2-a. It can also observed from SEM images that further increase of filler content into matrix material beyond 50 %weight fraction is quite difficult task, as increase in filler content reduces the inter particle distance upto the limit that particle start to interfere with each other, which may degrade the properties of filler as well as composite.



(a- Silicon carbide particles)



(b- 20 wt%)



(c- 40 wt%)

Fig. 2: SEM micrograph (a) silicon carbide particles ; Composite with (b) 20 wt% (c) 40 wt % filler.

Hardness of any material is considered as one of the important factor which governs the wear resistance of any material. Micro-hardness values of the epoxy composites for different silicon carbide content have been measured and the variation of micro-hardness with the filler loading in the composites is shown in Figure 3.a. A gradual increase in both tensile strength as well as ultimate tensile strain with the filler weight fraction is noticed. It clearly indicates that inclusion of silicon carbide particles improves the load bearing capacity and the ability to withstand fracture of the composites. Similar observations have been reported by Harsha et al. for fibre reinforced thermoplastics such as polyaryletherketone composites. The variation of tensile strength of the particulate filler polymer composite is presented in Figure 3.b. Epoxy resin as a polymer matrix in present work possesses tensile strength of 32 MPa whereas ultimate tensile strain of epoxy is 1.21 %. The effect of incorporation of SiC in ultimate tensile strain of epoxy is reflected in Figure 3.d. It is observed that a gradual increase in tensile strength from minimum 32 MPa of neat epoxy to a maximum of 46.5 MPa of epoxy composites with 50 wt. % silicon carbide. It clearly indicates that the addition of silicon carbide enhances the load carrying capacity of the composite. Also from Figure 4.5 it is clearly visible that the ultimate tensile strain drastically increases from 1.21 to 1.69 % for maximum 50 wt % of filler loading. Though earlier it has been reported that incorporation of particulate filler decreases the tensile strength of the composite system because of irregular shape of filler and stress concentration originate due to such filler but in present work it has been seen that incorporation of filler enhances the strength remarkably. This is because of the uniform spherical shape of the filler which avoid and stress concentration inside the composite body and can be seen in SEM image of filler. Also interfacial adhesion between the filler particles and the matrix are strong which may be another reason of strengthening of composites body with filler content.

Composite materials used in structures are prone to fail in bending and therefore the development of new

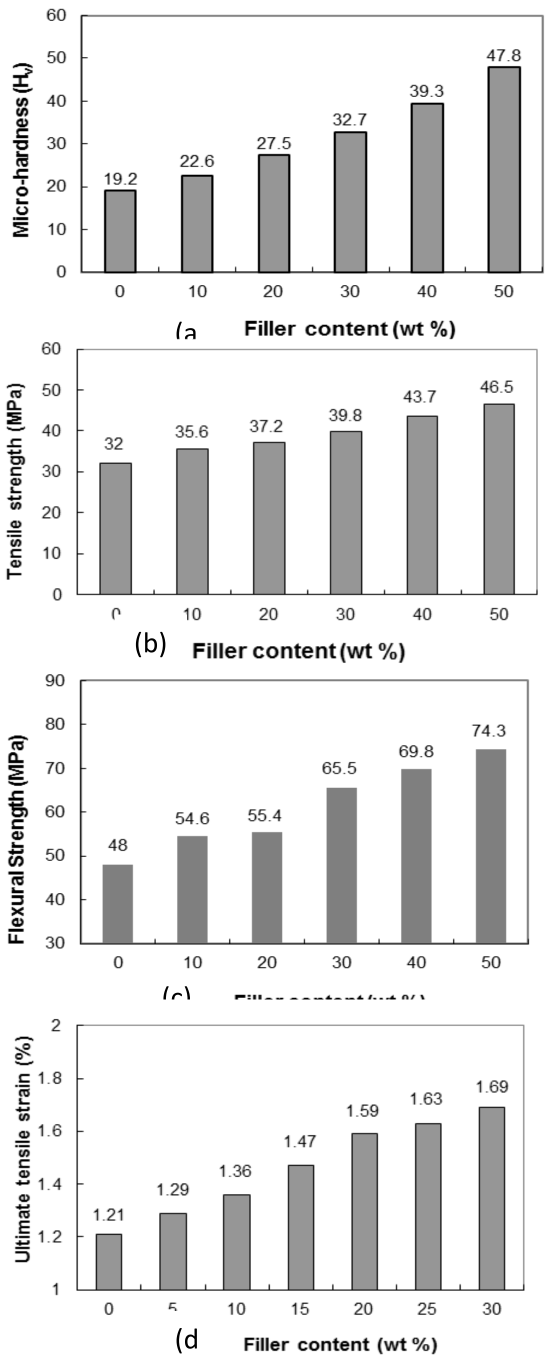


Fig. 3: Mechanical properties

Composites with improved flexural characteristics are essential. In the present work, the variation of flexural strength of silicon carbide-epoxy composites with different particulate filler content in the range of 10 wt% to 50 wt% is shown in Figure 3-c. A gradual increase in flexural strength from minimum of 48 MPa in neat epoxy to a maximum of 74.3MPa in epoxy composites with 50 wt % silicon carbide is recorded. The enhancement in the flexural strengths of the composites with filler content is probably caused because of excellent compatibility of the particulates and the epoxy matrix, leading to strong interfacial bonding. The higher values of flexural properties may also be attributed to good filler to filler interaction, less voids and uniform dispersion. However, it also depends on other factors such as the size, shape and type of the filler material. In present work all this factors are gainfully enhancing the various property because of almost constant size and uniform shape. It is evident from this study that as far as the flexural strength is concerned, SiC-epoxy composites are found to be an excellent material.

3.2 Dry Sliding wear characteristics:

3.2.1 Surface morphology of worn composite samples:

The SEM (scanning electron microscope) image of the unworn surface of Silicon carbide-epoxy composite filled with 30 wt.% SiC is illustrated in Figure 4. The silicon carbide particles are seen to be evenly distributed in the matrix body. The morphology is indicative of a good quality composite with good interfacial adhesion among filler-matrix with some minor pores. Figure 5 shows the SEM micrograph of the worn surface of the same composite which is subjected to dry sliding wear test. This micrograph is taken after 30 minutes of test duration with a sliding velocity of 300 cm/s under a normal load of 10 N. Indication of plastic deformation of the matrix body is evident in this image. Local removal of the matrix material is also possible as the duration of sliding increases. These features are observed in the SEM images given in Figures 6.

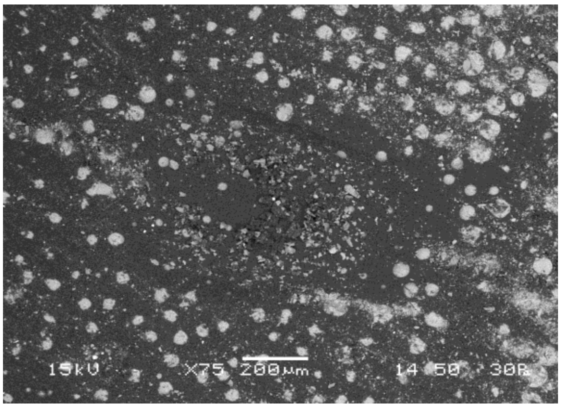


Fig. 4 SEM image of the unworn surface of the silicon carbide filled epoxy composite

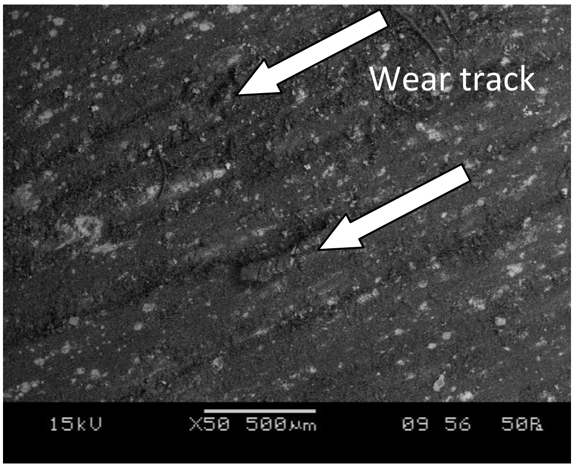


Fig. 5 SEM image of the worn surface of the composite after wear test

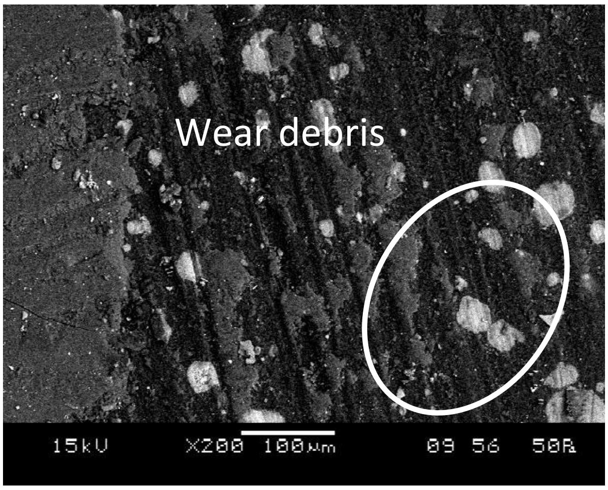


Fig. 6 SEM micrographs showing plastic deformation on the worn surfaces

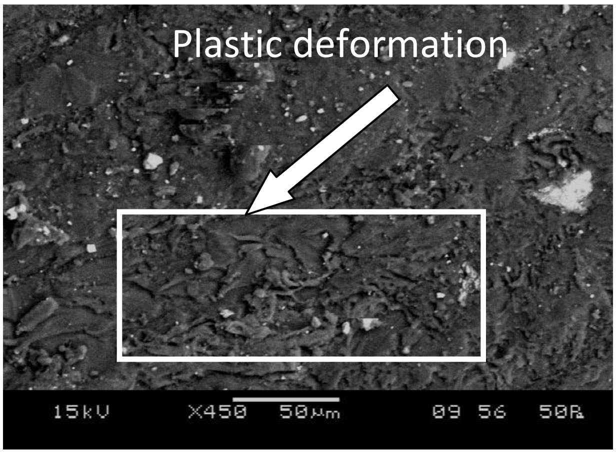


Fig. 7 SEM micrographs showing of wear debris on the worn surfaces

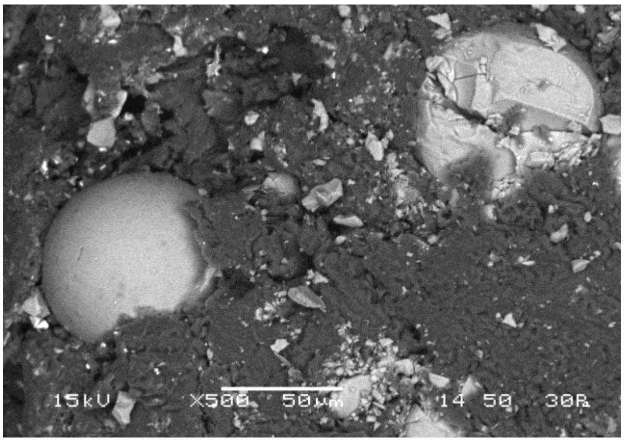


Fig. 8 SEM micrographs showing of broken SiC particle after wear test

3.3 Sliding wear test results and Taguchi analysis:

The dry sliding wear rates of silicon carbide filled epoxy composites under various test conditions are given in Table 5.1. The difference between the initial and final weights of the pin and specimen assembly is the sliding wear loss or the mass loss of the composite under sliding. The loss is then converted to volume loss per unit load per unit sliding distance and this quantity is taken as the specific wear rate. Three samples are run for each combination of the

Table 4: Erosion wear test result with corresponding S/N ratios
The overall mean for the S/N ratio of the wear rate is found to be -18.70 dB.

Test run	Factor A Sliding velocity (m/sec)	Factor B Normal load (N)	Factor C Silicon carbide content (wt. %)	Factor D Sliding distant (m)	W_s Specific wear rate (mm^3/N-m)	S/N ratio (db)
1	100	5	0	1200	9.36	-19.4255
2	100	10	10	1600	8.27	-18.3501
3	100	15	30	2000	6.48	-16.2315
4	100	20	50	2400	5.16	-14.2530
5	200	5	10	2000	9.96	-19.9652
6	200	10	0	2400	9.02	-19.1041
7	200	15	50	1200	6.22	-15.8758
8	200	20	30	1600	7.14	-17.0740
9	300	5	30	2400	8.61	-18.7001
10	300	10	50	2000	7.70	-17.7298
11	300	15	0	1600	10.95	-20.7883
12	300	20	10	1200	10.02	-20.0174
13	400	5	50	1600	9.37	-19.4348
14	400	10	30	1200	9.81	-19.8334
15	400	15	10	2400	11.07	-20.8830
16	400	20	0	1600	11.98	-21.5691

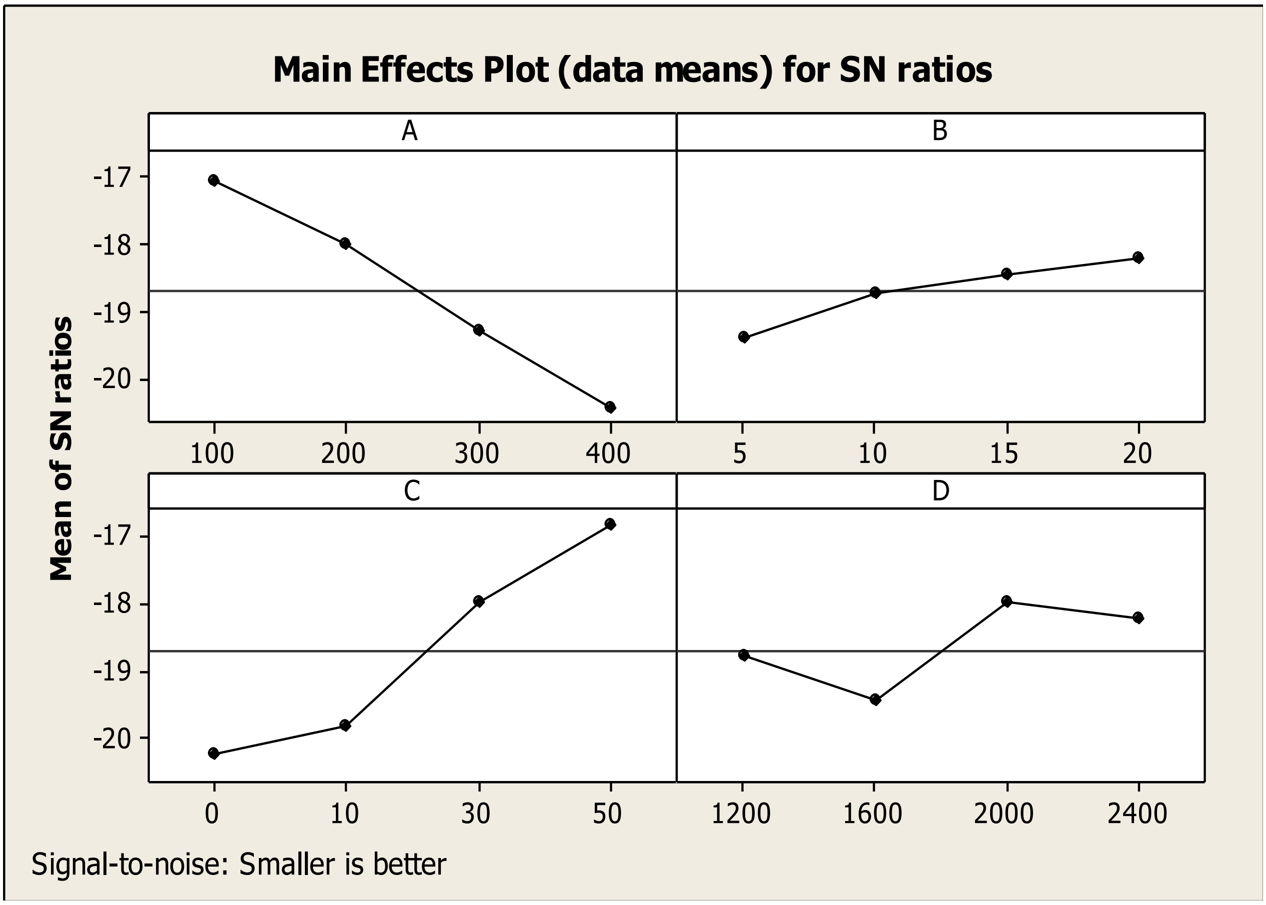


Fig. 9 Effect of control factors on sliding wear rate of composites

test parameters employed. The results reported are thus the average of the three readings. These experimental observations are then transformed into signal-to-noise (S/N) ratios. In Table 5.1, the last column represents S/N ratio of the wear rate which is in fact the average of three replications. There are several S/N ratios available depending on the type of characteristics. The S/N ratio for minimum specific wear rate coming under smaller-is-better characteristic can be calculated as logarithmic transformation of the loss function as shown below:

$$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum y^2\right)$$

where, ‘n’ the number of observations, and ‘y’ the observed data. The analysis is made using the popular software specifically used for design of experiment applications known as MINITAB 14. Figure 5.6 shows graphically the effect of the four control factors on the specific wear rate.

Table 5: Signal to noise ratio response table for erosion wear rate of composites

Stage	**A**	**B**	**C**	**D**
1	-17.07	-19.38	-20.22	-18.79
2	-18.00	-18.75	-19.80	-19.44
3	-19.31	-18.44	-17.96	-17.98
4	-20.43	-18.23	-16.82	-18.24
Delta	3.37	1.15	3.40	1.47
Rank	2	4	1	3

The S/N ratio response is given in the Table 5, from which it can be concluded that among all the factors, silicon carbide content in the composites is the most significant factor followed by sliding velocity and normal load while the sliding distance has the least or negligible significance on wear rate of silicon carbide-epoxy composites. It also leads to the conclusion that factor combination of A1, B4, C4 and D4 gives minimum wear in dry sliding situations.

IV. SUMMARY OF RESEARCH FINDINGS

Some of the worth noting findings of these tests are summarized below.

- By incorporating silicon carbide in epoxy, synergistic effects, as expected are achieved in the form of modified mechanical properties and wear resistance. Inclusion of silicon carbide in neat epoxy has not resulted in any improvement in the load bearing capacity (tensile strength) but slight increase in the ability to withstand bending (flexural strength) of the composites is noticed.

- Hardness values have been found to have improved invariably for all the composites on addition of silicon carbide. The improvement in hardness with the incorporation of filler particles can be explained as follows: a compression or pressing stress is in action. So the polymeric matrix phase and the solid filler phase would be pressed together and touch each other more tightly. Thus, the interface can transfer pressure more effectively although the interfacial bond may be poor. This might have resulted in an enhancement of hardness.

- In the present investigation, it is noticed that the composites filled with silicon carbide have higher void fraction compared to the unfilled composites. The presence of pores and voids in the composite structure significantly affects some of the mechanical properties and even the performance of the composites. Higher void contents usually mean lower fatigue resistance, greater susceptibility to water penetration and weathering. However, presence of void is unavoidable in composite making particularly through hand-lay-up route.

V. CONCLUSIONS

This present investigation has led to the following conclusions:

1) Successful fabrication of epoxy matrix composites reinforced with silicon carbide particles is possible by simple hand-lay-up technique.
2) The density of the fabricated composites increases with increase in weight fraction and it also possesses low void content; even it is prepared by hand lay-up technique.
3) These composites possess very low amount of porosity and improved micro-hardness, flexural and inter-laminar shear strengths than those of the neat epoxy. However, they did not exhibit similar behavior in case of tensile strength

This study reveals that silicon carbide possesses good filler characteristics as it improves the sliding wear resistance of the polymeric resin. Dry sliding wear characteristics of these composites can be gainfully analyzed using a design-of-experiment approach based on Taguchi method. The analysis of experimental results shows that factors like filler content, sliding velocity and normal load, in this sequence, are identified as the significant factors affecting the specific wear rate of the composites under investigation.

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