Tribological Characterisation and Morphological Study of Epoxy Composites filled with WS_2 under Dry Sliding

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Abstract: ***The tribological behaviour of epoxy composites filled with Ws_2 particulates were studied using a pin-on-disc wear apparatus under dry sliding conditions. The influence of friction and wear parameters like, percentage of filler content, normal load, sliding velocity and sliding distance on the friction and sliding wear rate were investigated. A plan of experiments, based on the Taguchi technique, was performed to acquire data in a controlled way. The Taguchi design of experiment approach eliminates the need for repeated experiments and thus saves time, material and cost. The results showed that filler content is the most predominant factor as far as the specific wear rate is concerned. The SEM images of the tested samples are also interpreted which shows the anisotropic wear and the wear mechanisms like three body abrasive wear.***

Keywords: Composites; Filler, SEM; Taguchi Technique; Tribology

1. Introduction

Polymer based materials are emerging as a good replacement for metals especially in tribological applications due to its unique advantages such as light weight, low coefficient of friction, wear resistance and self lubrication capacity [1]. Polymers are reinforced with fibres and fillers to improve the mechanical and tribological properties and to achieve better strength to weight ratio. Numerous types of non-metallic bearing materials are now available commercially. Polymeric bearing materials are used specifically where fluid lubricants are ineffective because of hostile environmental conditions, difficulty to provide maintenance, and because of product and environmental contaminations [2].

Lot of research has been done on the polymer composites for tribological applications. Various polymers can be reinforced with solid lubricants like graphite, MoS_2, PTFE etc. PPS (Poly Phenylene sulphide) filled with graphite particles, and Epoxy filled with bronze can be used up to 200ºC [3]. Cement kiln dust is a waste by-product of the cement making process can be used as filler in Epoxy for tribological applications. It seems to be an effective reinforcement to improve the wear resistance of the composite [4]. Glass epoxy polymer composites with SiC and Graphite particles as secondary fillers was studied, the results showed that the inclusion of SiC in the polymer matrix increases the wear resistance of the material[5]. The wear performance evaluation of Pine wood dust filled Epoxy composites showed that the pine wood dust improves the wear resistance of the polymeric resin [6]. PTFE filled with different oxides of aluminium; zirconium revealed that only 2% Al_2O_3 in PTFE gives minimum wear rate 0.4-0.2×10^{-6} kg/h [7]. The selection of polymers as materials for sliding as well as rolling components of machines and devices is very important task for tribologist. Non-polymer-on-polymer as well as polymer-on-polymer contacts are important nowadays in design of machines and devices in modern technology. The non-polymer-on-polymer tribosystems are often applied in various machines and devices. This is probably due to good mechanical and thermal properties of the counter-face rubbing element. The most popular and also practically confirmed as the best tribological combination is steel on polymer frictional tribosystem [8].

2. Experimental Details

2.1 Materials

Bisphenol-A (BPA) ARL-136 is an organic compound with the chemical formula $(CH_3)_2C(C_6H_4OH)_2$. It is a colourless resin that is soluble in organic solvents, but poorly soluble in water. Having two phenol functional group, it is used to make polycarbonate polymers and epoxy resins along with other materials used to make plastics [9].

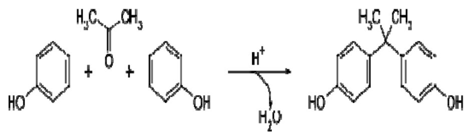

Figure 1: Chain reaction of Phenol

The hardener Lapox AH-126 is a liquid modified anhydride. The resin and hardener is supplied by M/S ATUL LIMITED polymers Division, Valsad (G.S.) and the particulate fillers, tungsten disulphide (WS_2) powder of 4 micron is supplied by the Sajan Oversease, Vatva, GIDC (G.S.). Tungsten disulfide (WS.sub.2) is a known dry-film lubricant that was developed for NASA by Stanford University in the 1960's. Following its initial debut, tungsten disulfide found its way into industrial applications, primarily in aerospace and defence applications. Tungsten disulfide is known to improve wear properties and to enhance lubricity. It also has an affinity for lubricants, resulting in oil-retention properties in "Wet" application.

Tungsten disulfide is commercially available as a powder that comprises finely divided tungsten disulfide particles with a mean particle size ranging between about 1 micron and about 3 micron, depending upon the commercial supplier. Tungsten disulfide adheres to a substrate surface through a molecular/mechanical interlock and takes on the characteristic of the substrate regardless of whether the substrate is ferrous, non-ferrous, a composite, carbide or plastic. When applied to a substrate material, tungsten disulfide also forms a very thin layer due to the fact that it does not bond to itself. As a result, the dimensions and tolerances of treated parts are not compromised or appreciably affected when a substrate is treated with tungsten disulfide. Further, these aspects of tungsten disulfide prevent chipping, flaking or contamination problems.

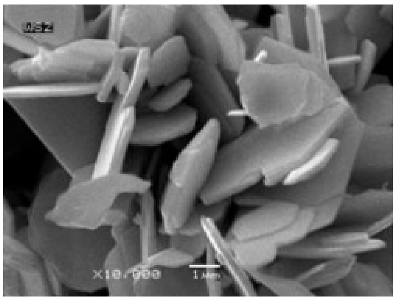

Figure 2: Electron microscope picture of WS2

Table 1: Properties of Epoxy resin ARL-136

S.No.	*Description*	*Standard*	*Unit*	*Values*
1	Appearance	-	visual	Clear to Hazy liquid
2	Density@ 25^0C	ISO 1183	g/cm^3	1.14-1.17
3	Viscosity@ 25^0C	ASTM D5478	mPa.s	2500-4500
4	Epoxy value	ASTM D1652	Eq/Kg	5.3-5.7
5	EEW	DIN 16945	g/eq	176-186

Table 2: Properties of Hardener AH-126

S. No	*Description*	*Standard*	*Unit*	*Values*
1	Appearance	-	visual	Yellow liquid
2	Density@ 25^0C	ISO 1183	g/cm^3	100-300
3	Viscosity@ 25^0C	ASTM D5478	mPa.s	1.10-1.20

Table 3: Properties of Filler WS2

Sr.no	*Description*	*Standard*	*Unit*	*Values*
1	Appearance	-	visual	Blue gray powder
2	Density solid	ISO 1183	g/cm^3	7.5
3	Melting point		^{o}C	1250
4	Coefficient of friction			0.03 dynamic 0.07 -static

2.2 Specimen Preparation

The epoxy resin is mixed with the hardener in the ratio 100:90 by volume in a glass vessel. The required quantities of the filler WS2 were stirred gently into the liquid epoxy resin, taking care to avoid the introduction of air bubbles. The AH-126 hardener was added to the above mixture in the required ratio. The mixed composite is stirred gently to ensure complete homogeneous mixing. The mixture is then poured into a metallic mould cavity coated with a release agent to yield specimens of 30mm height and 11mm diameter. The mould is placed in an electric oven and heated at constant temperature of 180^{o}C for 1 Hour for curing and upon curing mould is opened to release specimen from the mould after 30minutes. These test specimens were then post cured at constant temperature of 100^{o}C in an electric oven for 60 minutes Composition of test specimens of composites was varied 1-4% by volume of filler.

Figure 3: Metallic die cavity

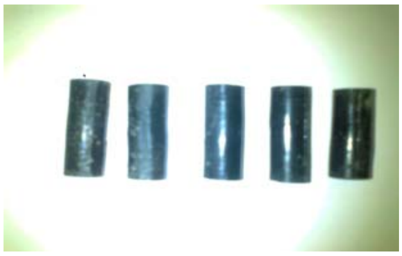

Figure 4: Composite specimen for testing.

2.3 Dry Sliding Wear Test

A pin-on-disk setup (ASTM G99 standard, Make: DUCOM Instruments, Bangalore) was used for dry sliding wear tests. Eight numbers of samples were made by different compositions and both the ends of each sample were rubbed against EN-31 steel disc at different parameters. The surface of both the sample and the disc were cleaned and thoroughly dried before the test. The control factors its levels for the present work are listed in table 4. Each sample pin was weighed before and after the test in an electronic balance with an accuracy of 0.0001g. The difference between the initial and final weight is the measure of sliding wear loss.

Table 4: levels of variables used in experiment

Control Factor	*Level*				*Units*
	1	2	3	4	
A:Sliding Velocity	1.25	2.5	3.75	5.0	m/sec
B:Normal Load	29.43 (3Kg)	39.24 (4kg)	49.05 (5Kg)	58.86 (6Kg)	N(kg)
C:Filler Content	1	2	3	4	Vol %
D:Sliding Distance	1500	2000	2500	3000	M

A series of tests are conducted by selecting L16 orthogonal array, and specific wear rate is calculated.

Sample 1:
Initial weight of the specimen: 2.980 gms
Final weight of the specimen: 2.970 gms

Weight loss during experimentation:2.980-20970=0.01gms

For Filler content =1%, Density of specimen=1.23gms/cm^3
Volume of material wear out = mass/Density=0.01/1.23X1000=8.1300mm^3

For above wear volume calculations of specific wear rate are as under Specific Wear rate in mm^3/N-m = 8.1300/29.43 × 1500=0.00018

3. Results and Discussions

3.1 Tribological Properties

Table 5 lists the experimental measurements of specific wear rates and s/n ratios of epoxy composites filled with different volume percent of WS_2. From the table, it can be observed that the incorporation of WS_2 filler into epoxy initially showed slightly increase in specific wear rate with increase in filler content, but it decreases as filler content further increased. Further, it showed that increase in load also increases the specific wear rate slightly. Observing the s/n ratios of the different experiments in the table one can conclude that the highest s/n ratio is observed in sample 15 which indicates minimum wear in the sample. And the lowest s/n ratio is observed in sample 16 which indicates the maximum wear in the sample. The S/N ratio response analysis, presented in Table 5 shows that among all the factors, filler content is the most significant factor followed by sliding velocity and normal load while the sliding distance has the least or almost no significance on wear rate of the particulate filled composites under this investigation.

For all experiments wear values in mm^3 / N. m are calculated from the wear readings observed in gms/cm ^{3}. These values are tabulated in Table 5 from above calculations.

calculations of SN ratio

For the function Smaller is better the formula is
$SN1 = - 10 \log (\sum y^2)$
where, SN1=is SN ratio for experiment no 1
y = specific wear rate in mm^3/N-m
$= -10 \log(0.000184)^2$
= 74.7036

Table 5: Specific wear rate and SN ratios

Expt. No.	sliding Velocity (m/sec)	Normal Load (N)	Filler content (Vol%)	Sliding distance (m)	Specific wear rate (mm3 /N-m)	S/N Ratio
1	1.25	29.43	1	1500	0.000184	74.7036
2	1.25	39.24	2	2000	0.000196	74.1548
3	1.25	49.05	3	2500	0.0000595	84.5097
4	1.25	58.86	4	3000	0.0000375	88.5193
5	2.5	29.43	2	2500	0.0000836	81.5550
6	2.5	39.24	1	3000	0.00110	59.5762
7	2.5	49.05	4	1500	0.00009	80.9055
8	2.5	58.86	3	2000	0.000062	84.1521
9	3.75	29.43	3	3000	0.000124	78.1315
10	3.75	39.24	4	2500	0.0000337	89.4474
11	3.75	49.05	1	2000	0.000124	78.1315
12	3.75	58.86	2	1500	0.000130	77.7211
13	5.00	29.43	4	2000	0.000562	85.0052
14	5.00	39.24	3	1500	0.000062	84.1521
15	5.00	49.05	2	3000	0.0000261	91.6671
16	5.00	58.86	1	2500	0.000331	69.6034

Table 6: Signal to Noise ratio response table for wear rate

Level	Sliding velocity (A)	Normal Load (B)	%Filler content (C)	Sliding distance (D)
1	80.47	79.84	70.50	79.37
2	76.54	76.83	81.27	80.36
3	80.85	**83.80**	82.73	**81.27**
4	**82.60**	79.99	**85.96**	79.47
Delta	6.06	6.97	15.46	1.9
Rank	**3**	**2**	**1**	**4**

The delta is the difference between minimum & maximum values of SN ratio for Particular parameter. Highest value is ranked 1 & has the maximum influence on the process.

3.2 Morphology of Worn Surfaces

Scanning electron microscope (SEM) was used to analyze the worn surfaces of the composites. Worn surface samples were mounted on aluminium stub and were sputter coated with gold prior to SEM examination. The surfaces of the samples were examined directly by scanning electron microscope Leica-Stereoscan-440. Figure 5 shows the worn surface of 1 % of WS_2 filled epoxy composites tested under 58.86 N. The micrograph of the sample clearly shows that at higher load and minimum filler content 1%, the wear and damage to the surface was maximum. Further in this sample abrasive and adhesive wear is predominant. The composite of same filler content at minimum load of 29.43 N shows low wear and micro ploughing marks are clearly visible.

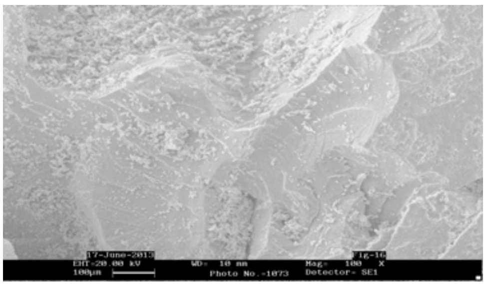

Figure 5: SEM picture of 1% filler at 58.86 N

Figure 6 shows the worn surface of 2% WS_2 filled epoxy composites tested at 58.86 N. The material is chipped of from the surface and severe localised damage is seen due to higher load. At minimum load of 29.43N the same composite don't show any crack or measurable wear. Further looking at figure it is clear that the filler is well bonded with the matrix.

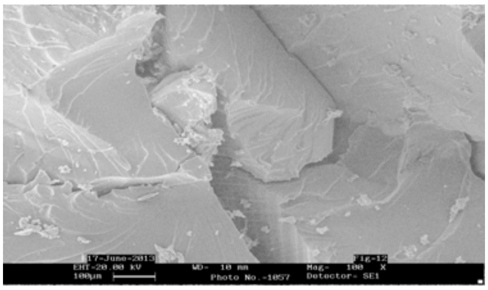

Figure 6: SEM Picture of 2% filler at 58.86 N

From figure 7 of 3% filler content and load applied 58.86 N we can conclude that the wear of the sample is greater than the same sample at when the load is 49.05 N, the patches of compacted material are clearly visible. Further the sample with higher load and velocity shows the worn particles held together by thermo-mechanical process due to frictional heat.

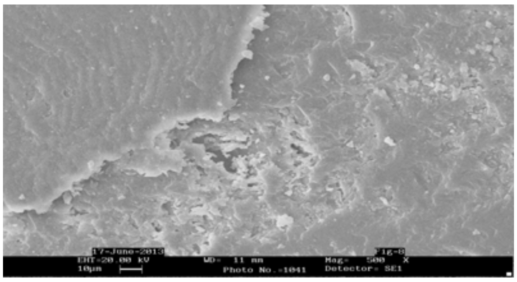

Figure 7: SEM picture of 3% filler at 58.86 N

As seen in figure 8 of 3% filler at 39.24 N number of micro cracks can be seen along with debris particles deposited in the cracks region. Few micro pits are formed which indicates that the fatigue wear is predominant as the filler content increases. It also shows three body abrasive wear.

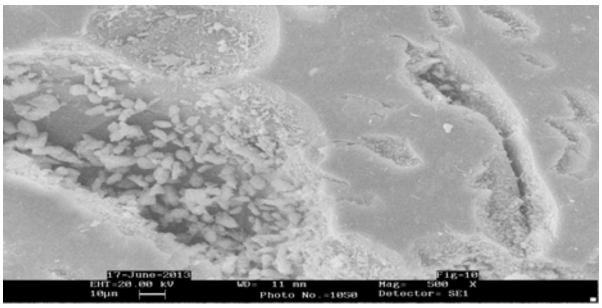

Figure 8: SEM picture of 4% of filler at39.24 N

4. Conclusions

- According to the micrograph it can be seen that the WS_2 filler particles are well bonded with the epoxy matrix, so fabrication of epoxy composites with WS_2 is quite possible.
- Filler content is the most predominant factor in wear of the composites observed in the tribotest followed by normal load, sliding velocity and sliding distance.
- As the filler content increases the wear resistance of the composites also increases.
- Micrograph images reveals that in low filler content composites the abrasive as well as adhesive wear is predominant wear mechanisms.
- As the filler content increases the material becomes brittle and fatigue wear is predominant in that case.

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