HARDNESS AND TRIBOLOGICAL CHARACTERISTICS OF EPOXY COMPOSITE FILLED WITH REDUCED GRAPHENE NANOPARTICLES

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Abstract - *Epoxy resin is widely used in engineering applications due to its high elasticity and light weight. However, their low thermal stability and wear resistance limits their usage at high sliding velocities and loads. The mechanical properties and thermal stability of a machine element subjected to friction and wear is very important. Filler materials are introduced to composite matrixes to improve their mechanical, thermal or tribological properties. The properties of the epoxy composites are highly influenced by the type of reinforcement like graphene, single walled carbon nanotubes (SWCNTs), double walled carbon nanotubes (DWCNTs), multi-walled carbon nanotubes, nano clays, nano silica, carbon nano fibers, etc., to increase the strength and modulus of composites. As variety of combinations of composites are possible by choosing the right mixture of materials for matrix and reinforcement to obtain optimized composite properties. In this Paper, reduced Graphene nanoparticles composites with different weight proportions of reduced GnP is fabricated by solution casting technique. Mechanical properties are evaluated as per ASTM test standards and sliding wear test is performed following a design of experiment approach.*

1. INTRODUCTION

Composite materials are the combination of two or more materials which are different in form and chemical composition. These constituent materials have particularly different chemical or physical properties, and they are dissolved to create a material with properties different from the individual elements. Within the completed structure, the individual elements remain distinct and distinct, separating the composites from mixtures and solid solutions. Composites are composed of two phases, the matrix phase which provides the bulk form to the composite like metals, ceramics, polymer and the dispersed phase which reinforces the matrix phase and can be in the form of fibres, particles or flakes. Filler materials are materials that are added to the composite to enhance desired properties that might not otherwise be achieved by the reinforcement and matrix ingredients alone. Composite materials are being developed to replace conventional materials for many reasons such as higher strength, better fracture toughness, good thermal resistance, low price etc Polymer composites are polymer materials with a reinforcement in which the polymer works as a matrix resin that penetrates the reinforcement knots and bonds to reinforcement. These composites are advanced materials that have high strength at low weight, which make them usable at various applications like automobile, aerospace and household appliances. The decision of the material for specific applications depend on variables such as material cost, thickness, quality, and working conditions. Polymer composites used in sliding conditions are commonly used in low energy transfer. Lightweight polymer matrix composites are the most suitable materials for weight sensitive application in the aerospace and automobile industries. Polymer composites replace traditional metal and ceramic materials to make high strength and low conductivity applications like pump wear ring, bushings, line shaft bearings, interstage bushings and pressure reducing bushings. Epoxy resins are the most commonly used thermoset plastic in polymer matrix composites which do not give off reaction products when they cure and thus have low cure shrinkage. Epoxy composites are a type of polymer material that uses an epoxy resin to create a polymer matrix reinforced with fibers or other fillers. This allows for the construction of long-lasting parts with very high power-to-weight ratios.

2. MATERIALS AND METHODS

Composite Preparation

Reduced Graphene nano platelets was synthesized from graphene oxide. The 99.8% pure graphite flakes was received from Alfa Asear, USA. Concentrated sulfuric acid (H_2SO_4), phosphoric acid (H_3PO_4), hydrochloric acid (HCl), hydrogen peroxide(H_2O_2), potassium permanganate ($KMnO_4$), ethanol, N,N dimethylformamide (DMF) were procured as analytical grade from S. D. Fine chemicals, India. The analytical grade diglycidyl ether of bisphenol-A (DGEBA) (Lapox L-12, Atul Ltd, India) was used as the matrix material, and N,N-bis (2-aminoethyl) ethane-1,2-diamine (Lapox K-6, Atul Ltd., India) was used as a hardener, and resin was mixed with hardener in the ratio of 10:1.

Reduced GnP is used as the filler material in this investigation. It is produced through the servothermal reduction method where Dimethylformamide (DMF) is used as the solvent. This particulate filler is thoroughly mixed with the epoxy resin in different weight proportions (0, 0.25, o0.5, 0.75, 1 wt. %). The rGnP is dispersed in DMF and heated under constant pressure and added in different concentration in epoxy. The dough was then slowly poured into the mould of different shapes, coated beforehand with silicone-releasing agent. These were left to cure for 24 h after which the moulds were broken and specimens were released. Then samples are cut into 3cm long cylinders with 8mm diameter to conduct hardness and sliding wear tests.

Fig 2.1 Neat Epoxy

Fig 2.2 Epoxy/rGnP composite

Sl No.	Composition
1	Epoxy + rGnP(0% volume fraction)
2	Epoxy + rGnP (0.25% volume fraction)
3	Epoxy + rGnP (0.5% volume fraction)
4	Epoxy + rGnP (0.75% volume fraction)
5	Epoxy + rGnP (1% volume fraction)

Table 2.1

3. COMPOSITE TESTING

3.1 Hardness Test

Hardness is the resistance of a material subjected to a localized plastic deformation caused by mechanical indentation or abrasion. It has important diagnostic advantages in mineral identification or abrasion. Stiffness is based on plasticity, ductility, elastic stiffness, strain, strength, hardness, viscoelasticity, and viscoelasticity. In general, different materials differ in their hardness; For example, metals such as titanium, beryllium are harder than metals such as sodium and metallic tin, or materials like wood and common plastic.

The Rockwell hardness test is one of the most commonly used hardness test method. It is used in all kinds of metals except in situations where the surface conditions and metal structures create high variations. The Rockwell hardness test is performed primarily by a test force, commonly known as a small load or preload. It is applied to a specific sample with the help of an indender. This reflects the reference or zero, the finish. Once the minor load step is completed, the additional load application is applied to achieve the required test load. After the use of force, it is withheld for a predetermined period to allow for elastic recovery. Then, the main load is subjected to release and the resulting position is measured against the preload position. The depth indentation variation between the values of the main load and preload is also measured. The distance obtained is then transformed into a hardness number. In simpler terms, under this method, the hardness of the specimen is measured by assessing the depth of the indentation made by the indenter under the test load on the specimen surface.

Test Procedure

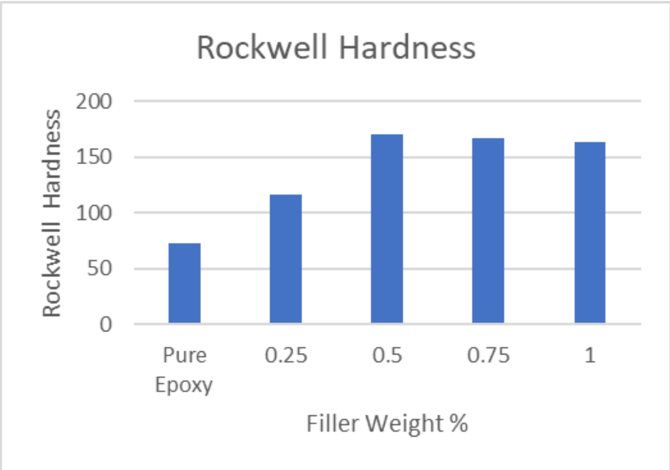
ASTM E 384 standard was adopted for hardness test and five indentations taken for each sample. Using mathematical formula with indentation dimensions, hardness for each sample were taken with the help of optical microscope with a magnification of 2000 ×.

Fig 3.1 Rockwell hardness testing of the samples

The experimental results of composite testing by varying the filler content are presented in table 3.1

Sample Number	Concentration (wt%)	Rockwell Hardness	% Increase In Hardness
1	Pure Epoxy	73	0
2	0.25	116	59
3	0.5	171	134
4	0.75	167	128
5	1	164	124

Table 3.1 Rockwell Hardness for various concentration of rGnP



Graph 4.1 Rockwell Hardness for various concentration of rGnP

3.2 Sliding wear Test

Wear process took place by sliding wear and wear by hard particles. The degree of contact angle between both surfaces decides the wear process. when adjoining surfaces slide against each other wear happens. Various parameters such as loading type, testing condition, temperature, type and quantity of lubricants decides the wear test results.

The sliding wear can be classified as the relative motion between two smooth solid surfaces in contact below the load, where surface damage during translational sliding does not occur through deep surface grooving because of asperities or intrusion of foreigner particles. Surfaces can be metallic or nonmetallic in nature, lubricated or unlubricated. Many different parameters of a tribosystem are involved to a certain extent in the friction and wear of the sliding pairs. In sliding contact, wear and tear can occur due to adhesive, surface fatigue, tribochemical reaction and/or abrasion. Many factors influence the existing wear and tear system. The type of contact either elastic or plastic is a function of tangential traction on the surface, the contact area and physical properties such as yield strength. Examples of sliding wear are the reciprocating and rotating machines. More specifically, sliding wear is common in automotive and heavy-duty piston ring applications, synchronizer rings, transmission systems, automotive, and large cylinder bores for gas transmission applications, hydraulic rods for earth-moving devices, and landing gears for mainframe aerospace applications instead of hard chrome plating.

The characterization of friction and wear (commonly worn rates and wear resistance) of materials is usually performed using various types of tribometers, while pin on disk test being probably one of the most common type.

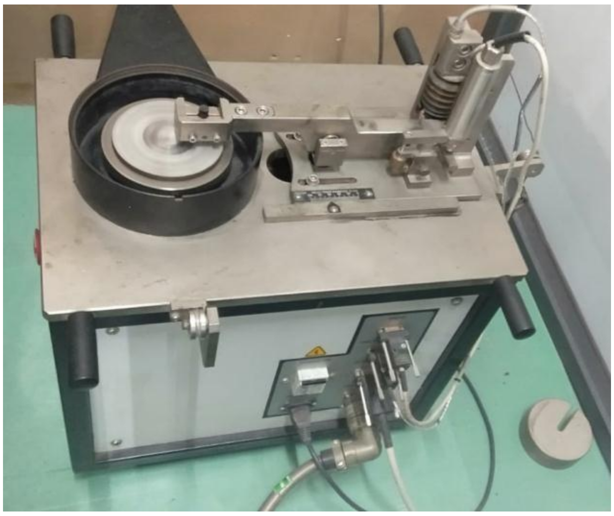


Fig 3.2 pin on disc wear testing of the samples

Test Procedure

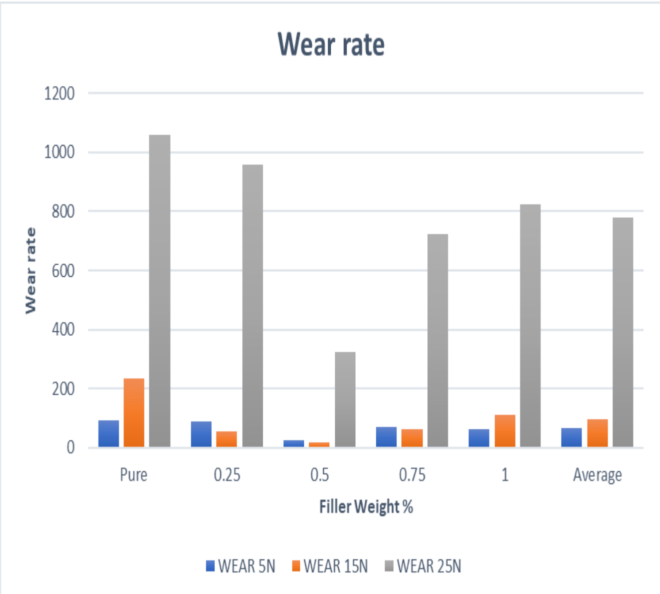
A pin on disc tester (Make: Ducom, Bangalore, India; Model: TR20- PHM 600) under dry sliding (ambient temperature) contact condition were used for the test set up. Fig. 3.2 shows test setup for wear test.

For load range from 0 to 60 N with 3 loads 5,15,25 N and disc speed from 0 to 1000 rpm were employed. Test pin is pressed against the surface with corresponding loads. The friction disk got a diameter of 165 mm and a depth of 8 mm, EN-31 hardened steel with a hardness of 50-62 HRC and surface roughness value (Ra) 0.55-0.65 micrometer. The composite pin got a dimensions of 8 mm diameter and 30 mm length (ASTM G99 standards).

The experimental results of composite testing by varying the filler content and load are presented in table 3.2 and table 3.3

Concentration	WEAR 5N	WEAR 15N	WEAR 25N
Pure	92.38403	234.245	1059.928
0.25	88.31621	53.95362	957.4183
0.5	25.64642	18.90858	321.7141
0.75	69.80835	63.97254	724.9341
1	60.79464	110.4529	825.3104
Average	**67.3899**	**96.3065**	**777.861**

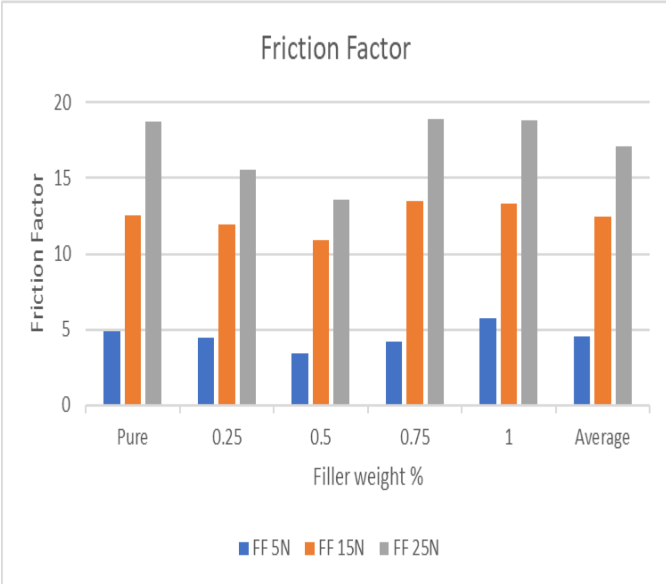
Table 3.2 Wear ate for various concentrations of rGnP



Graph 3.2 Wear rate for various concentration of rGnP

Concentration	FF 5N	FF 15N	FF 25N
Pure	4.93032	12.54328	18.75183
0.25	4.454828	11.91577	15.53899
0.5	3.45966	10.87353	13.55351
0.75	4.16129	13.50613	18.93977
1	5.74193	13.28799	18.80157
Average	**4.54961**	**12.4253**	**17.1171**

Table 3.3 Friction factor for various concentration of rGnP



Graph 3.2 Friction factor for various concentration of rGnP

4. RESULTS

4.1 Hardness Test

The surface hardness of the neat epoxy and epoxy based Reduced nanocomposite specimens were measured through the Rockwell micro hardness (Hv) test setup to reduced GnP fillers on the epoxy matrix. Graph 3.1 shows the effect of various filler contents on the hardness value of epoxy-based nanocomposites. It is evidenced, the hardness value of specimens was enhanced with the increasing of filler content.

Neat epoxy showed a Rockwell hardness value of 73 HRC, and this value increased to 116 HRC (59 % rise) with the addition of 0.25 wt. % rGnP and 171 HRC (134 % rise) with the addition of 0.5 wt. % GnPs. For the other concentrations (0.75 and 1wt.%), rGnP embedded composites showed significant increase of the hardness i.e., 128% and 124%, respectively. Major reasons for the increase in the hardness of particles-filled composites is the fine dispersion of the particles in the matrix giving more surface area for the GnPs and thereby strengthening the bond between epoxy and rGnP matrix.

From this result, it is also evident that the highest micro hardness value was achieved by the 0.5wt% specimen with an increment up to171 i.e 134% higher than that of the neat specimen (73 HRC). The increase in the percentage of filler loading reduced graphene nanoplatelets (varying from 0.1, 0.25, 0.5 and 1 wt.%) has decreased for other filler concentration. Here, infiltration between epoxy and rGnP particle is higher for 0.5 % rGnP corresponds to the higher hardness thereby reducing nano- void formation which is the main cause for the improved hardness properties. The decrease in hardness at higher weight loading of 0.75 wt% and 1 wt.% GnP is due to the GnP agglomeration.

4.2 SLIDING WEAR TEST

The average CoF as a function of sliding distance (2000 m) at the 3 different applied load values (5,15,25 N) is shown in Figure. The average coefficient of friction value of the epoxy resin matrix was decreased with the addition of filler particles graphene nanoplatelets. Thus, by increasing the filler in matrix, the average coefficient of friction decreased.

Neat epoxy showed an average wear rate of 461.1856 mg/m, and this value decreased to 366.5627mg/m (26 % less) with the addition of 0.25 wt. % rGnP and 122.089mg/m (74 % less) with the addition of 0.5 wt. % GnPs. For the other concentrations (0.75 and 1wt.%), rGnP embedded composites showed significant decrease of the wear rate i.e., 38%and 28%, respectively.

Due to the lubricating property of the GnP, the sliding contact surface creates a lubricant film thereby an improvement in the CoF and wear rate. But for higher concentration CoF and wear rate values due to the higher filler concentration and improper interfacial bonding between the filler and epoxy led to early wear debris which generates higher CoF in wear test. This foreign body wear debris sticks to the contact surface and creates a higher coefficient of friction and wear rate for the higher filler composites.

CONCLUSION

In this work, the effect of rGnP for wear and hardness behavior of epoxy matrix nano-composites was investigated. The concentration of rGnP varied between 0 to 1 wt. %, with 0.25 wt.% interval and compared with results of pure epoxy composite. The following conclusions were drawn: In comparison to the base epoxy sample, the maximum gain of 134% in the Rockwell hardness was achieved for 0.5 wt. % rGnP loading into the epoxy matrix due to the superior bonding between rGnP, and epoxy. The least wear rate of 73 % was observed for 0.5 wt. % rGnP loading into epoxy matrix with moderate surface texture property.

Hence, with this superior mechanical property at 0.5 wt. % rGnP embedded epoxy-based composites, the currently rGnP composite material finds applications where the resistance to wear is dominant and weight to strength ratio is a major constraint like aerospace applications.

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