PREPARATION AND TRIBOLOGICAL CHARACTERIZATION OF Ni-P COATED GLASS FIBER REINFORCED EPOXY COMPOSITES

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“It is never too late to be what you might have been.”

George Eliot

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*CERTIFICATE OF APPROVAL **

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This is to certify that the thesis entitled, ***"PREPARATION AND TRIBOLOGICAL CHARACTERIZATION OF Ni-P COATED GLASS FIBER REINFORCED EPOXY COMPOSITES"*** *which is being submitted by* ***Mr. Jayanta Sarkar*** *in the partial fulfillment of the requirements for the award of degree of* ***Master of Mechanical Engineering*** *of Jadavpur University, Kolkata-700032 during the academic session 2014-2016, is the record of student's own work carried by him under the supervision of* ***Dr. Nipu Modak*** *and* ***Prof. Prasanta Sahoo****.*

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CHAPTER-1

INTRODUCTION

This chapter provides the introduction to Glass fiber reinforced Epoxy composites prepared by Hand lay-up method and the introduction to Electroless deposition technique and development in the field of electroless Ni-P coating. It also gives the overview of present scope of work for the thesis.

1.1 POLYMER MATRIX COMPOSITES

Polymer materials have been finding great potentials in the industry as a class of important engineering materials. For centuries, the use of polymers in everyday life has become important part of human life. The growth of polymer starts in the early 19th century. The development of polymer technology was slow until 1930s, before the materials such as phenol, vinyl, polystyrene and polyester were developed. After the development of these materials, the polymer research has taken a huge growth that is still going on. Polymers are rapidly developing material with the attractive advantages of low density and cost compared to metals and ceramics. Generally, polymers are very large molecules consisting of many small molecules called monomers or repeating units that can be linked together to form long chains and are known as macro-molecules. A typical polymer may include tens of thousands of monomers. This specific long chain like structures is responsible for their fascinating properties with a wide range of stiffness, strength, heat resistance etc. Humans have taken advantage of the versatility of polymers for years in different fields of consumer durables, electrical and electronic equipments, aerospace, packaging, medical equipments, automobiles and other engineering applications.

However, polymer in its pure form alone could not satisfy the demands for various applications where a combination of good mechanical and wear properties is required. Hence reinforcements are needed to provide additional strength for the polymers. The addition of reinforcements into polymers is termed as polymer matrix reinforcement composite or shortly PMCs. PMCs are engineering materials consists of two or more significantly different physical or chemical properties of constituent materials separated by a distinct interface. Upon the combination of this material, a material with properties different from the individual characteristics is produced. The constituent materials contain matrix and reinforcement.

The primary phase of the composite materials is the matrix phase which is usually more ductile and less hard phase as well as it holds the reinforcing phase normally stronger than the matrix and transfers stresses between the reinforcements. The matrix phase of the PMCs is generally two types which could be chosen under the two main category, thermoset and thermoplastic materials.

Thermoset resins are the materials which undergo chemical reactions when heating that crosslink the entire polymer chains into a three dimensional network and tends to have high dimensional stability, high temperature resistance and good resistance to solvents. Thermoset resins are irreversible process which includes phenol formaldehyde, unsaturated polyester, vinyl ester, and epoxy. Epoxy has some good advantages such as high mechanical and thermal properties, high water resistance, long working times available, temperature resistance can be up to 140°C for wet and 220°C for dry and low cure shrinkage.

Thermoplastic materials include polyethylene, polypropylene, acrylonitrile-butadiene-styrene, polyamide, polyetherimide etc. Upon heating the thermoplastics to the process temperature, the material can be formed into any shape. Unlike thermoset resins, in thermoplastics the process is reversible and by simply reheating the material it can be formed into another shape which does not happen in thermoset resins. Thermoplastic materials offer a great advantage from a manufacturing point of view, because it is easier and faster in processing compared to thermoset resins which attract high volume industries such as automotive industries.

The reinforcement phase of PMCs is particulate or fiber types of reinforcements. The particulate reinforcements are composed of particles which include aluminum, alumina, titanium di-oxide, calcium-carbonate, kaolin, zinc-oxide, talc, potassium-titanate etc. The fiber reinforcements include various types of natural fibers which consist of animal and plant fibers and man-made fibers which are synthetic fibers.

1.2 CLASSIFICATION OF NATURAL AND SYNTHETIC FIBERS

Various types of fibers are used to make various types of composites. In general fibers are classified into two main groups i.e. natural and synthetic or man-made and further classified into many sub categories. Fibers classification in details is shown in Fig.1.1. In the present study, our main focus is on glass fibers.

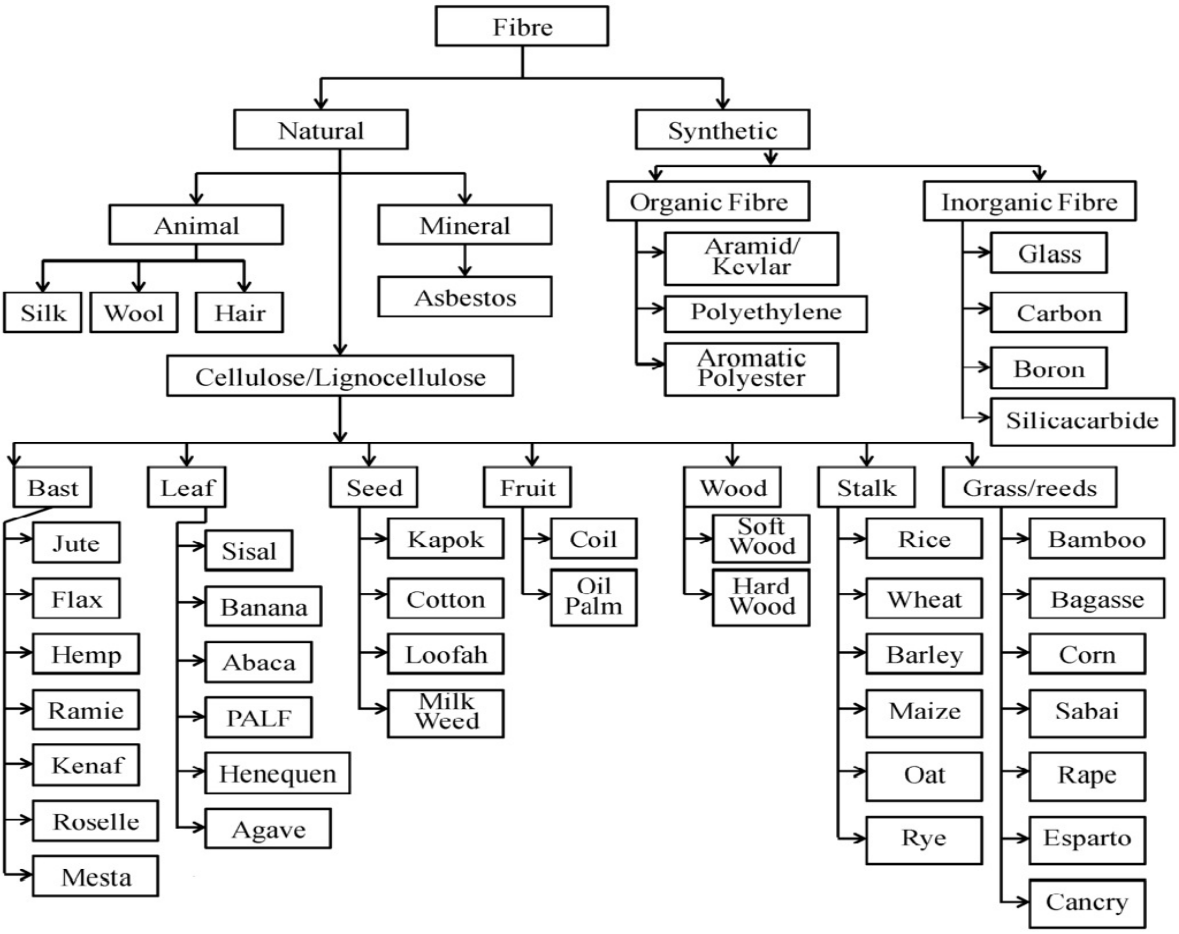
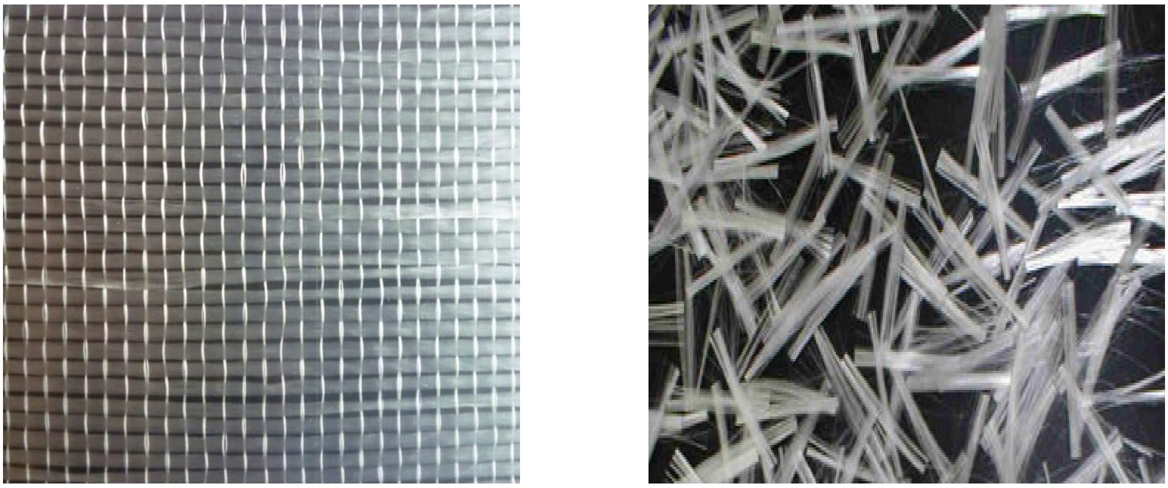


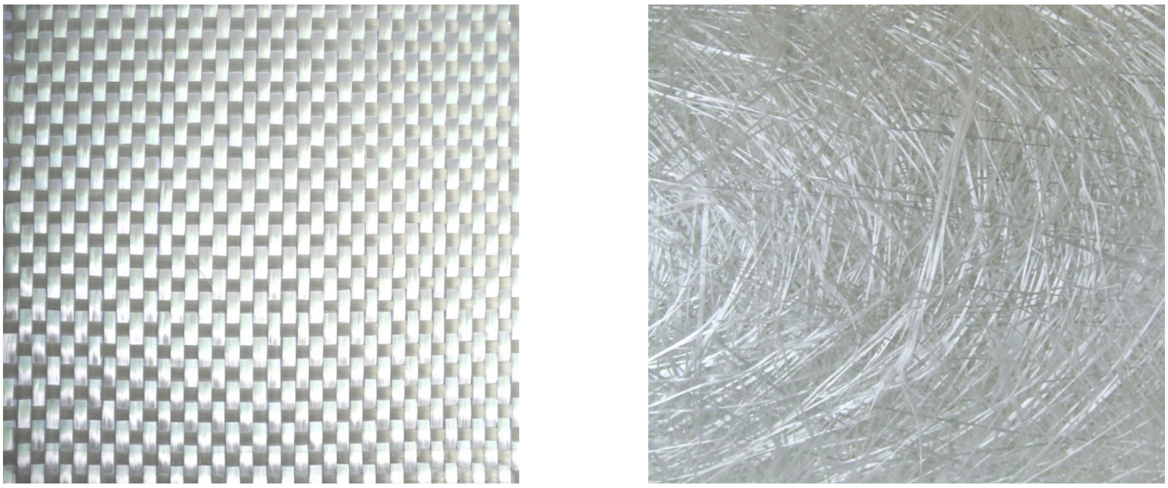
Fig. 1.1 Classification of natural and synthetic fibers.

1.3 GLASS FIBERS

The glass fibers in all their various forms are the most widely used materials for the reinforcement of polymer and other matrices. This is attributed to their availability, low cost, high strength, chemical inertness and well established production technology. Several types of glass fiber exist but all are based on Silica (SiO_2) which is melted and mixed with other elements to create speciality glasses. The fibers of glass are produced by extruding molten glass through holes in a spinneret with diameter of 1mm to 2mm and then drawing the filaments by one of two techniques to produce fibers having diameters usually between 5 and 15 µm. The spinnerets usually contain several hundred holes so that a strand of glass fiber is produced. For the purpose of fiber reinforcement the most commonly used glass is lime-alumina-borosilicate complex known as E glass. This glass exhibits high strength and possess good characteristics for fiber reinforcement. Other types of glass used are : A-glass has higher durability and strength, C-glass has higher corrosion resistance, D-glass has relatively low strength, low stiffness, low dielectric constant and also used in electronic applications, R-glass has higher strength and acid corrosion resistance, and S-glass has superior strength and stiffness characteristics. Glass fibers are used in a variety of forms as reinforcement in composite. The various types of glass fibers, i.e. unidirectional, chopped, woven and chopped strand mat are shown in Fig.1.2. The major classification of glass fibers and the physical properties are shown in Table 1.1. The physical and mechanical properties of glass fibers are shown in Table 1.2. Also the chemical compositions of glass fibers in weight percentage are shown in Table 1.3.



Unidirectional fiber Chopped fiber



Woven Fiber Chopped strand mat fiber

Fig.1.2 Unidirectional, chopped, woven and chopped strand mat glass fibers.

Table 1.1 Classification of glass fibers and the physical properties [1].

Classification	**Physical Properties**
A glass	Higher durability, strength and electric resistivity
C glass	Higher corrosion resistance
D glass	Low dielectric constant
E glass	Higher strength and electrical resistivity
R glass	Higher strength and acid corrosion resistance
S glass	Highest tensile strength

Table 1.2 The physical and mechanical properties of glass fibers [2].

Fiber	**Density (gm/cm^3)**	**Tensile Strength (GPa)**	**Young's modulus (GPa)**	**Elongation (%)**	**Coefficient of thermal expansion ($10^{-7}/^oC$)**	**Poisson's ratio**	**Refractive index**
E-glass	2.58	3.445	72.3	4.8	54	0.2	1.558
C-glass	2.52	3.310	68.9	4.8	63	-	1.533
S-glass	2.46	4.890	86.9	5.7	16	0.22	1.521
A-glass	2.44	3.310	68.9	4.8	73	-	1.538
D-glass	2.11-2.14	2.415	51.7	4.6	25	-	1.465
R-glass	2.54	4.135	85.5	4.8	33	-	1.546

Table 1.3 Chemical compositions of glass fibers in weight percentage [2].

Type	**SiO_2**	**Al_2O_3**	**TiO_2**	**B_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**
E-glass	55.0	14.0	0.2	7.0	22.0	1.0	0.5	0.3
C-Glass	64.6	4.1	-	5.0	13.4	3.3	9.6	0.5
S-glass	65.0	25.0	-	-	-	10.0	-	-
A-glass	67.5	3.5	-	1.5	6.5	4.5	13.5	3.0
D-glass	74.0	-	-	22.5	-	-	1.5	2.0
R-glass	60.0	24.0	-	-	9.0	6.0	0.5	0.1

1.4 ADVANTAGES OF POLYMER COMPOSITES

The objective of polymer composites is to take advantage of the superior properties of both matrix and reinforcement and form new tailor made properties. Due to availability of wide variety of matrix and reinforcements, the design potentials are greater in terms of low weight, corrosion resistance, dimensional stability, quick assembly etc. than the other composites such as metal matrix or ceramic matrix composites.

Polymer based composite materials are becoming an essential part of today's materials and have successfully substituted the traditional materials in most applications where high strength to weight ratio, high tensile strength at elevated temperatures, high creep resistance, high toughness, light weight, good surface finish, low cost etc. are required.

Due to the better properties of polymer composites, these materials are used extensively in engineering components and play a key role in the sectors such as aerospace, automobile, aircraft, electronics, packaging, healthcare, construction, defense and marine etc. For the past few decades, polymer composites have generated wide interest in tribo-engineering applications and used in mechanical components such as bearing material, rollers, seals, gears, cams, wheels, clutches, bushes etc. due to their special property of self lubrication, corrosion resistance, light weight etc. The polymer composite materials are well suited for non-lubricated condition and it is the key factor for this material selection in tribological applications.

1.5 ELECTROLESS NICKEL COATING

The performance and duration of engineering components can be improved by applying hard coating over the surface of the components. Electroplating and electroless deposition are the most cost-effective processes for applying metallic coatings of thickness between 10 to 500μm on many engineering components. Their rates of deposition can provide the required product quality at relatively low investment and operating expenses. Electroplating is a well recognized technology but the electroless coating has emerged recently in comparison. In the middle of the twentieth century, electroless plating deposition method was proposed by Brenner and Riddell [3].

Electroless nickel coating is an electrochemical method of coating deposition in which no electricity is required as compared to the conventional electrolytic method. Electroless coating uses more complex solution. It contains water, a metal salt and a reducing agent as basic components. Electroless coating is basically a chemically deposited coating in which the substrate is submerged in to a chemical solution called bath which consists of solution of metal ions, reducing agent, stabilizers, buffer solution etc.

Electroless Nickel (EN) coating is an autocatalytic chemical reduction process in which the reducing agent is oxidized and Ni^{2+} ions are deposited (reduced) on the substrate surface. Once the first layer of Ni is deposited, it acts as a catalyst for the process. The EN deposition process starts only on the catalytic surface and it involves the diffusion of chemicals to the deposited surface and by-products away from the surface. During the electroless plating process hydrogen is one of the by-products evolved from the deposit surface. Furthermore, in the past decade; advancements have been made in autocatalytic nickel plating solutions. Reducing agents other than sodium hypophosphite are used for special applications, composites of nickel with diamonds, and silicon carbide are available, and ternary alloys may be applied. Also, baths have been formulated to yield specific results, i.e., high corrosion resistance, brightness, high plating rate, improved ductility and low levels of magnetic response.

Applications for electroless nickel in engineering field can be found virtually in every industry. A variety of physical characteristics of electroless nickel coatings such as hardness, wear resistance, coating uniformity and corrosion resistance makes this a coating of choice for many engineering applications such as aerospace, aviation, automotives, oil and gas processing, food processing, microelectronics, radio electronics, computer engineering, chemical processing, textiles, machinery, mining and metallization of plastic etc[4]. In addition, the unique ability to plate non-conductors such as glass, ceramics, plastics and graphite has made electroless nickel a very useful coating. It can be regarded as a problem-solving, cost-effective alternative with equivalent performance to more expensive metal alloys.

Electroless nickel coating has also been used to improve the wear and wear corrosion properties of CFRP (carbon fiber reinforced polymer) materials which are frequently utilized in the aerospace, aviation and automobile industries due to their light weight [5]. The potential of Ni-P coatings for marine applications have been investigated by various researchers. UHMWPE(Ultra-high-molecular-weight polyethylene), an advanced engineering plastic used in making seawater hydraulic drive is found to produce the lowest friction coefficient when sliding against electroless Ni-P coating under sea water lubrication[6].

1.5.1 ADVANTAGES OF ELECTROLESS NICKEL COMPOSITE COATING

The main advantages of electroless Nickel based composite coating are

- Resistance against wear and corrosion.
- Increase in micro hardness.
- Increase thermal resistance.
- Smooth & uniform deposit regardless of the geometry of the substrates.
- Oxidation resistance.
- Good solder ability, bond ability and weld ability properties.
- Greater lubricity, Good chemical stability.
- Nonmagnetic properties.
- Good appearance and brightness.
- Low co-efficient of thermal expansion and high thermal conductivity.

1.5.2 DISADVANTAGES OF ELECTROLESS NICKEL COATING

- Lifespan of chemicals is limited.
- Waste treatment cost is high due to the speedy chemical renewal.

1.6 CLASSIFICATION AND APPLICATIONS OF ELECTROLESS NICKEL BASE COATINGS

Engineering applications for electroless nickel can be found virtually in every industry. Various physical characteristics of electroless nickel coatings, such as hardness, wear resistance, coating uniformity, and corrosion resistance, as well as the ability to plate non-conductive surfaces makes this a coating of choice for many engineering applications. Table- 1.4 represents the classification of electroless nickel alloy coatings [7] and Table-1.5 reflects the applications of electroless nickel deposits depending upon the coating thickness.

Table 1.4 Classification of electroless nickel alloy coatings.

Alloy	Coating
Binary alloy	Ni-P
	Ni-B
Ternary alloy	Ni-P-B
	Ni-P-W
	Ni-P-Cu
	Ni-P-Co
	Ni-P-Sn
	Ni-P-Fe
Quaternary alloy	Ni-P-W-Cu
	Ni-P-W-Cr
	Ni-P-Fe-Co

Table 1.5 Applications of electroless nickel deposits depending upon the coating thickness.

Application areas	Coating Thickness (µm)	Components
Automotive	2–38	Heat sinks, carburetor components, fuel injection, differential pinion ball shafts, disk brake pistons, transmission thrust washers, knuckle pins, exhaust manifolds and pipes, mufflers, shock absorbers, lock components, hose couplings, gear and gear assemblies. Fuel pump motors, aluminum wheels, water pump components, steering column wheel components, air conditioning compressor components.
Air craft/ Aerospace	10–50	Bearing journals, servo valves, compressor blades, pistons heads, engine main shafts and propellers, hydraulic actuator splines, seal snaps and spacers, landing gear components, pilot tables, oil nozzle components, flanges, engine oil feed tubes, flexible bearing supports, universal joints
Chemical & Petroleum	25-125	Pressure vessels, pumps and impellers, heat exchangers, filters components, turbine blades and rotor assemble compressor blades and impellers, spray nozzles.

Electrical	12–25	Motor shafts, rotor blades of stator rings
Electronics	2–25	Head sinks, computer drive mechanisms, chassis memory drums and discs, terminals of lead wires, connectors, diode and transistor cans, interlocks, junction fittings and PCB
Food	12–25	Pneumatic canning machinery, baking pans, moulds, freezers, mixing louts, and feed screw and extruders
Marine	25–50	Marine hardware, pumps and equipment
Medical & Pharmaceutical	12–25	Disposable surgical instruments and equipment, size-ring screens, pill sorters and feed screws and extruders
Military	8–75	Fuse assemblies, tank tarred bearings, radar wave guides, mirrors, motors, detonators and firearms
Mining	30–60	Hydraulic systems, jetting pump heads, mine engine components, piping connections, framing hardware
Wood & paper	~30	Knife holder corer plates, abrading plates and machine parts

1.7 PRESENT WORK

The first phase of the present study deals with the development of glass fiber reinforced epoxy composite (GFRE) using hand layup method. Once the composite is developed, electroless Ni-P coating is synthesized on GFRE composite substrate. The second phase involves analyzing the variation of wear depth and friction coefficient of glass fiber epoxy composite and Ni-P coated GFRE composite under dry sliding condition by varying the tribo-testing parameters. The specimens are tested on a pin on disc tribotester apparatus. Also a comparison has been drawn to evaluate the tribological performance of Ni-P coated GFRE composite and bare composite and find out if there has been any improvement or not. The microstructure and composition analysis of the GFRE composite and coated GFRE are done by means of scanning electron microscope, energy dispersive X-ray analyzer and X-ray diffraction analyzer respectively.

CHAPTER-2

LITERATURE REVIEW

This chapter provides an outline of glass fiber reinforced polymer composites and electroless Ni-P coating with its properties along with the relevant literature review required to set a scene for the present study. It also highlights the scope of the present work.

2.1 INTRODUCTION

Over the past decades fiber reinforced polymer matrix composite materials have gained much attention and are being widely used in sliding engineering components such as gears, bushes, bearings, cams, rollers, wheels, clutches, blades, many other application in aerospace and aviation industries etc. due to its intrinsic lubrication characteristics, excellent strength-to-weight ratio, low cost, chemical resistance, easy processability and excellent wear resistance [8]. Most of the researchers have studied the effect of testing parameters such as normal load, sliding speed, filler content and sliding distance on the tribological behavior of various polymer composites [9]. The friction and wear resistance of polymer matrix could be improved with the incorporation of reinforcement materials usually fibers or particulate fillers. The polymer matrix can be of thermoset or thermoplastics. In the present study thermoset material and inorganic fibers are considered, hence most of the reviews are based on thermoset and inorganic based polymer composites [10]. However, few thermoplastic materials as well as particulate fillers and fiber reinforced polymer composites are also discussed to get the knowledge of the tribological performances of polymer composites [11]. Tribological performance of polymer and its composites is generally sensitive to operating and test conditions where the fiber orientations play a dominating effect on wear and friction characteristics [12]. Another important parameter is surface temperature which is generated in polymer matrix composites. The wear intensity of polymer composites can occur due to thermal softening. It is found that a little attention has been paid towards it [13]. There is a need for better understanding of how and why different types of reinforcement and composites influence the tribological properties at different conditions.

2.2 MANUFACTURING TECHNIQUE OF GLASS FIBER REINFORCED POLYMER MATRIX COMPOSITES

The glass fiber reinforced polymer matrix composites were prepared by various manufacturing techniques as discussed below.

2.2.1 HAND LAY-UP METHOD FOLLOWED BY COMPRESSING MOULDING

Erden et al. [14] prepared the woven roving mat E-glass fibers or unmodified and modified polyester matrix composites using hand lay-up technique. Six glass fabric layers were used to fabricate laminates with 3.5 mm thickness. Then, composite laminates were compression molded at a pressure of 120 bars at room temperature for 120 min. All composites were fabricated with about 37 % fiber volume fraction.

2.2.2 HAND LAY-UP METHOD FOLLOWED BY HYDRAULIC PRESS

Suresha et al. [9] developed woven mat glass fibers reinforced epoxy composite using hand lay-up method. The epoxy resin is mixed with the hardener in the ratio 100:12 by weight. The stacking procedure consists of placing the fabric one above the other with the resin mix well spread between the fabrics. A porous teflon film is placed on the completed stack. To ensure uniform thickness of the sample a spacer of size 3 mm is used. The mold plates have a release agent soiled on it. The whole assembly is pressed in a hydraulic press (0.5 MPa) and allowed to cure for a day at room temperature. After de-molding, post curing was done at 120°C for 2 hours using an electrical oven. The laminate prepared has a size of 250 mm × 250 mm × 3 mm.

2.2.3 HAND LAY-UP METHOD

Suresha et al. [15] developed the woven fabric glass fibers reinforced vinyl ester composites using hand lay-up technique and the individual fiber diameter was 8–12 µm. The resin was mixed into Methyl ethyl ketone peroxide (MEKP) as catalyst, cobalt naphthanate as accelerator and, N-dimethyl aniline as promoter used, vinyl ester resin, cobalt naphthanate and MEKP were mixed the weight ratio of 1: 0.015: 0.015. Two different fillers are mixed into resin such as 50 µm of graphite and 25 µm size of silicon carbide (SiC). The woven mat piling up one above other and the mixture of resin spread over the fabrics by dry hand lay-up moulding and the whole die assembly compressed at constant pressure 0.5MPa of by using dead weight. The prepared slabs size was 250mm×250mm×3 mm.

2.2.4 SILICONE RUBBER METHOD

Aramide et al. [16] have made woven-mat glass fiber reinforced unsaturated polyester composite using silicone rubber mould. The mould was cleaned and dried. The mould surface was coated with releasing agent of hard wax for easy removal of composites. Initially, unsaturated polyester resin containing curing additives was applied in the mould surface by using brush. The glass fiber was placed on the resin and fully wetted. A steel roller was used to make full wetting of fiber in resin. A final sealing layer of resin was poured on the fiber. Over the period of time, the laminated composite was fully hardened and it was removed from the mould. The hand file was used to time the edges of the cured composites plate for obtaining the final size. The different fiber contents (5% to 30%) were used to prepare the composites plates.

2.2.5 HOT PRESS TECHNIQUE

Atas et al. [17] prepared the woven mat glass fiber reinforced epoxy composites using hot press technique. Non-orthogonal woven fabric prepared with dimension of 305mm×305 mm, an orthogonal fabric was transformed into non-orthogonal fabric by using shearing process with various weaving angles between fill and warp. The weight ratio epoxy resin to hardener was 10:3. The composite panels were cured at 60°C for 2 hours and 93°C for 4 hours during the curing process and 0.35MPa pressure was employed on the laminated composites.

2.2.6 COMPRESSING MOULDING

Hameed et al. [18] developed chopped stand mat E-glass fibers reinforced modified epoxy composite using compression moulding technique with various fiber volume fractions (10% to 60%). Fiber mats were cut in to size and heated in an air oven at 150°C to make it moisture free. The hardener was mixed in epoxy resin. Pre-weighted fiber mat and resin were used to get 3mm thickness of composite. The laminates was compressed in mould and cured at 180°C for 3 hours. The cured composite was post cured at 200°C for 2 hours and then cooled slowly to room temperature for obtained final composites.

2.2.7 MIXING AND MOULDING

Gupta et al. [19] prepared the discontinuous E- glass fibers reinforced epoxy composites with addition of filler-like fly ash. The diameter of glass fiber was 10 µm and cut into 2.54 cm length for composite preparation. The ratio of epoxy resin and hardener mixed is around 100:10. The two different concentrations of fly ash filler were selected as 2.5 and 5% vol%, along with calcium carbonate fillers added during the mixing of resin and hardener. Small size and spherical shape of the fly ash particles facilitate their good mixing and wetting of fiber and matrix. The mould was prepared with dimension of 154mm×78mm×12 mm. The fiber and matrix were transferred to the mould and that was allowed to cure at room temperature for 24 hours.

Kajorncheappunngam et al. [20] developed the 30cm square lamina of E-glass fabric in the epoxy resin and was cured in between the two Teflon coated layer of 0.32 cm thickness. The roller was used to remove the excess resin and leave for 3 days curing. The cured composite was post-cured 3 hours at atmospheric on 60°C in an oven. The resulting composite was of 1.5mm thickness with 47% weight fraction.

2.2.8 RESIN TRANSFER MOULDING

Wan et al. [21] developed polyacrylonitrile (PAN) based composite specimens by a vacuum assisted resin transfer molding (VARTM) process. Five braided fabrics with a nominal size of 160mm ×12mm×2mm were placed in a mould. A toughened epoxy resin and a curing agent were intimately mixed at ambient temperature and de-gassed at 70°C for 30 minutes in a vacuum oven. The mould containing the braided fabrics was also equilibrated at 70°C prior to resin pouring. The degassed mixed resin was then introduced into the mould under a pressure of 0.4–0.6MPa and assisted by the vacuum. The introduction of resin continued until no bubbles were observed within the resin flowing out from the exit of the mould. The evacuating process did not stop until the completion of resin introduction. The mould was then put into an oven for curing (at 90°C for 2 hours) and post curing (at 140°C for 3 hours).

2.3 INFLUENTIAL PARAMETERS FOR TRIBOLOGICAL PROPERTIES OF GLASS FIBER RREINFORCED POLYMER MATRIX COMPOSITES

2.3.1 NORMAL LOAD AND SLIDING SPEED

Normal load and sliding speed play a very important role on the friction and wear characteristics of glass fiber polymer matrix composite.

N.S.M. El-Tayeb and I.M. Mostafa [22] investigated the wear and friction characteristics of glass fiber reinforced polymer composites under different orientation with respect to sliding i.e.- parallel laminate(PL), normal laminate(NL) and cross laminate(CL). In all the cases both friction coefficient and wear resistance showed scattered trend of increasing with increase in normal load and beyond 20N an substantial fall in the values were observed. Apart from the above observation, the COF of PL is higher than NL and CL. At the same time the wear resistant offered by the PL is higher than NL. Mechanisms responsible for the wear resistance and COF in different orientation considered are enhanced by increasing the normal load and sliding velocity.

Sampathkumaran et al. [23] studied the wear behaviour of glass fiber reinforced epoxy composite under dry sliding condition. The varying test parameters were applied load (20–60 N), velocity (2–4 m/s) and sliding distance (0.5–6 km). Pin on disc experimental result showed that increasing the load and velocity increased the weight loss. The debris rate was lower for smaller distance and higher for larger distance.

Pihtili et al. [24] observed the wear behaviour of chop mat E glass fiber reinforced polymer composite with the different loading (0.5kg- 1kg) and sliding speed (500-710rpm). The weight losses were measured after sliding distances of 235.5 mm, 471 mm, 706.5 mm, 942 mm, 1177.5 mm, 1413 mm, 1648.5mm and 1884 mm. When the sliding distance exceeds 942 mm weight loss of the plain polyester was increased. The result showed that glass fiber reinforced polyester matrix composite was more wear resistant than the plain polyester.

El-Tayeb et al. [25] studied the friction characteristics of GFRE composite while sliding against hard steel, cast iron and aluminum, other than aluminum former two configuration showed a common trend of increase in COF with increase in normal load and the COF decreases with the increase in the sliding velocities in all the cases, but the value of COF was examined to be maintained at lower level when sliding against aluminum at higher velocities, in comparison of hard steel and cast iron.

El-Tayeb et al. [26] observed the tribological properties of polyester reinforced chopped strand mat (CSM) 450-R-glass fiber sliding against stainless steel counter face under different sliding configurations. Applied normal load has more pronounced effect on wear than sliding distance and wear rate increase significantly with increase in normal load. The combined effect of load and speed caused increased in the temperature at the interface playing a dominating effect on friction and wear rate.

Quintelier et al. [27] studied the friction and wear behavior of glass fiber reinforced polyester composites with loading range from 60 to 300N at a constant speed of 10 mm/s under dry conditions. The SEM observations showed that parallel orientation had lower friction than transverse orientation.

El-Tayeb et al. [28] investigated the multi pass two body abrasive wear behavior of chopped strand mat 450-R-glass fiber reinforced polyester composite with various sliding velocities such as 0.157 and 0.314 m/s and the applied normal loads of ranging from 5N to 25 N. The test was conducted with sliding against water proof SiC abrasive paper under dry contact condition. Wear rate decreased with increasing load and decreasing rotational speed.

2.3.2 FILLER MATERIALS

Polymer composites containing different filler materials are frequently used for the application like automotive parts, gear assemblies used inside home appliances in which the friction and wear are the critical issues. In particular, they are now been used in sliding elements, which were properly composed of metallic materials only. Filler material does not require improving the tribological properties as it was shown both positive and negative results on the tribological properties of the polymer.

Kishore et al. [29] observed the effects of velocity and load on the sliding wear behaviour of plain weave bi-directional E-glass fabric reinforced epoxy composites with different fillers such as oxide particle and rubber particle under the sliding velocity between 0.5 and 1.5 m/s at three different loads of 42, 140 and 190 N. Block on roller test result showed that the oxide particle filled composite had better wear resistance compared to rubber particles at low load conditions. But during higher load condition, rubber particles had better wear resistance than oxide particles.

Chand et al. [30] investigated the three-body abrasive wear behaviour of short E-glass fiber reinforced polymer composites with and without filler at various sliding speed, abrasive particle (angular silica sand) size ranging between 100–200 and 200–300 µm were used as dry and loose abrasives and applied load. Increasing the weight fraction of fiber in composite decreased the volume loss of composite. They have concluded that higher glass fiber content had less wear loss.

Hasim Pihtili and Nihat Tosun [31] carried an experiment to observe the effect of load and speed on the wear behaviour of a woven glass fabrics and aramid fiber- reinforced composites. It was observed that as the aramid fiber possess low friction coefficient and high wear strength characteristics than glass fabric reinforced composites, aramid fiber exhibited lower wear losses than glass fabric reinforced composites.

Suresha et al. [9] observed the role of fillers in wear and friction behaviour of woven mat glass fiber reinforced epoxy composites with varying load and sliding velocities under dry sliding conditions. Two different inorganic fillers were added such as SiC particles (5 wt %) and graphite (5 wt %). The SEM observation reported that graphite filled composites have lower coefficient of friction than unfilled SiC filled composites and SiC filled composite exhibited the maximum wear resistance.

Suresha et al. [32] investigated the friction and wear behaviour of woven mat E glass fiber reinforced epoxy composites with and without SiC particles. The result showed that (5 wt %) SiC particles filled composite had higher coefficient of friction and higher resistance to wear at sliding distance ranging from 2000m to 4000m compared to without SiC filled composites.

V.K.Srivastave and S.Wahne [33] studied tribological behaviour of short E-glass fiber reinforced epoxy composite filled with soft particles i.e. particles of mica and tri-calcium phospate (TCP). Results indicated that due to the filler contributed to enhance the bonding strength between fiber and epoxy resin, hence improved mechanical properties and the wear resistance were observed.

Larsen et al. [34] observed the friction and wear behavior of woven mat glass and weave carbon or aramid reinforced epoxy hybrid composites with various pressures (p), sliding velocity (v) also referred to as PV conditions. Glass fiber reinforced composites were higher wear rate than carbon or aramid composites. As a result friction rate was decreased 35% by adding weave glass fiber in carbon or aramid weave fiber reinforced epoxy composites.

Suresha et al. [35] found the three-body abrasive wear behaviour of woven E glass fiber reinforced vinyl ester composites with particulate filled like SiC and graphite fillers. The test was conducted at different loads such as 22N and 32N under the sliding distance ranging from 270m to 1080 m. The experimental result showed that the graphite and silicon filled composites had more abrasion resistance and lower specific wear rate ($1.95\times10^{-11} m^3/(Nm)$) than unfilled composites.

Basavarajappa et al. [11] investigated the comparative performance of glass–epoxy composite with influence of Graphite filler were experimentally investigated under varying applied load, sliding distance and sliding velocity by using a pin-on-disc apparatus. For increased applied load situation, higher weight loss was recorded. Addition of Graphite in glass–epoxy composite exhibits lower weight loss, whose value drops as the percentage of Graphite increases in the composite. SEM analysis has been made to identify the phenomenon of wear as a function of applied load, sliding distance and sliding velocity.

Patnaik et al. [36] studied the wear behaviour of randomly oriented E glass fiber reinforced epoxy composites with particulate filled like Al_2O_3, SiC and pine bark dust. The different compositions of specimens were prepared using glass fiber and epoxy (50 wt% : 50 wt%), glass fiber, epoxy and alumina (60wt%: 40wt% :10 wt%), glass fiber, epoxy and pine bark dust (50 wt% : 40wt% : 10wt%), glass fiber, epoxy and SiC (50 wt%:wt%:10 wt%). The experimental tests were conducted at different loads like 50N and 75N and the sliding distance ranging from 200m to 600 m. They have concluded that glass fiber, epoxy and pine bark dust (50 wt%: 40wt%: 10wt %) composite had better wear resistance (0.000881– 0.001365mm^3/(N-m)) for all sliding distance.

Mohan et al. [37] observed the abrasive wear behaviour of hard powder filled glass fabric-epoxy hybrid composite was carried out, where the results indicated that hard powder filled Glass-epoxy composite system exhibit lower wear volume loss and lower specific wear rate as compared to unfilled glass-epoxy system. Wear loss was observed at 200rpm sliding speed at 22-32N applied load.

2.3.3 ORIENTATION OF FIBER

Friction and wear mechanism are influenced by the orientation of fibers of glass fiber polymer matrix composite.

N.S.M. El Tayeb and I.M.Mostafa [22] studied the effect of the laminate orientation on the friction and wear mechanisms of chopped strand mat (CSM) glass fabric reinforced polymer composites in three different orientation parallel laminate (PL), normal laminate(NL) and cross laminate(CL) orientation, when sliding against silicon carbide abrasive paper. Experimental results showed that the PL orientation gives the highest value of friction coefficients followed by NL and CL. Microscopic investigation revealed that the weaving configuration in CL orientation inhibits an easy detachment of fibers during wear process. Thus intermediate values of wear resistance were observed for CL between PL and NL. In NL orientation, the individual fibers within the laminates do not get mutual support as in PL orientation. As a result fibers suffer bending at their ends, leading to an easy shear mechanism.

Bijwe et al. [38] investigated the friction and wear behavior of polyetherimide glass fiber composites with varying fiber percentage and under different wear modes. The author has revealed that rate of wear resistance of composites is different for different types of wear modes and fiber percentage.

El-Tayeb et al. [26] studied the tribological properties of polyester reinforced with CSM 450-R-glass fiber reinforced composites in three different orientation parallel(P), anti parallel(AP), normal(N) under dry sliding condition against smooth stainless steel counter face, the results indicated that the highest wear rate is observed when sliding took place in N orientation at all sliding speeds and magnitude was two times the P and AP orientation, where as lowest wear rate is exhibited when sliding took place in AP orientation at higher speed levels. At lower speed levels P orientation gave lower wear rate. Normal orientation showed 25-55% higher COF than that of other orientation.

El-Tayeb et al. [28] observed the multi pass two body abrasive wear behavior of chopped strand mat (CSM) 450-R-glass fiber reinforced polyester (CGRP) composite with various sliding velocities such as 0.157 and 0.314 m/s and the applied normal loads of ranging from 5N to 25 N. Anti parallel orientations enhanced the abrasive resistance of CGRP composite. They concluded that anti parallel orientation had lowest wear rate than other orientations and scanning electron microscope (SEM) result indicated anti parallel orientation had no fiber damage.

Mathew et al. [39] observed the tribological properties of E glass fiber reinforced polyester composites with various directly oriented warp knit fibers such as biaxial, biaxial non-woven, tri-axial and quad-axial fabric with various thermoset resins like polyester, vinyl ester and epoxy resin. Biaxial non woven reinforced vinyl ester composite had better performance than other combinations.

El-Tayeb et al. [40] found the worn surfaces of chopped glass fiber reinforced unsaturated polyester composites of parallel or anti-parallel chopped glass fiber orientations with various sliding velocities such as 2.8, 3.52 and 3.9 m/s and various load of 30N, 60N and 90N at ambient temperature. The experimental result showed that sliding in parallel orientation had lower friction coefficient at lower load and higher speed compared to anti parallel orientation. Sliding in anti parallel orientation had lower friction coefficient at higher load, speed and distance compared to parallel orientation. Anti parallel orientation exhibited less mass loss (16%) compared to the parallel orientation.

Yousif et al. [41] carried out the friction and interface temperature behaviour of chopped strand mat glass fiber reinforced unsaturated polyester composites with various sliding velocities such as 2.8, 3.52 and 3.9 m/s and various loads 30, 60 and 90N under dry contact sliding against smooth stainless steel. Parallel and anti-parallel chopped glass fiber orientations were measured at ambient temperature. The anti parallel orientation had more friction coefficients (0.5–0.6) and interface temperature (29°C to 50°C) compared to parallel orientation.

Yousif et al. [42] studied the wear and friction behaviour of chopped strand mat glass fiber polyester composites with various loads (30N to 100 N) under wet contact condition using two different test techniques such as pin-on-disc and block-on-ring. This was conducted with two different fiber orientations like parallel and anti-parallel. From the experiment, they concluded that presence of water content increased the roughness value in both orientations. Moreover, anti-parallel orientation had more wear and frictional resistance than parallel orientation.

2.3.4 TEMPARATURE EFFECT

Many researchers have conducted study of the temperature effect on the wear and the friction characteristics of the fiber reinforced polymer composites.

Hameed et al. [10] investigated the thermal behaviour of chopped strand E-glass fiber reinforced modified epoxy composites with different volume fraction (V_f) of fibers such as 10%, 20%, 30%, 40%, 50% and 60%. The test was conducted in nitrogen atmosphere at the temperature range from 30°C to 900°C. The thermo gravimetric analysis (TGA) showed that 60% V_f of composites had higher thermal stability and its degradation temperature was shifted from 357°C to 390°C.

Kim et al. [43] examined the tribological properties of short glass fiber reinforced polyamide 12 composite sliding against medium carbon steel, and result showed that the applied load has strongly affected the wear resistance of the composite due to increase in temperature at the sliding interface and rapid wear was observed when the interface temperature went beyond glass transition temperature

2.4 ELECTROLESS NICKEL-PHOSPHORUS (EN) COATINGS

2.4.1 INTRODUCTION

Electroless Nickel Phosphorus (EN) coatings have been used increasingly in various industries since the early 1980’s. Some of the outstanding characteristics of these coatings are superior corrosion and wear resistance, excellent uniformity, wide range of thickness as well as mechanical and physical properties; good solder ability and surface lubricity. They are widely used either as protective or decorative coatings in many industries, including petroleum, chemical, plastic, optics, printing, mining, aerospace, nuclear, automotive, electronics, computer, textile, paper, and food [44]. In addition, compared to conventional electroplating methods, EN coatings can be applied on different substrates (conductive and nonconductive) since no external current is applied to the component [45]. Although EN coatings are fairly new, the discovery of the fact that nickel could be deposited on a surface from an aqueous solution of its salt by reduction with hypophosphite was made by Waltz in 1844. Due to the poor quality of the reducing agents that leads to rough deposits with inferior properties, Waltz's idea was not developed for a whole century. In 1944, the first laboratory experiment reported on EN was completed by Brenner and Riddel who were later given credit for introducing the EN method to the world [46]. Since then, there have been many advances in the EN method. The choice of nickel as the base metal for deposit has remained popular throughout as electroless nickel coatings in particular are very much suitable from tribological point of view. This implies electroless nickel coatings can be used to reduce wear, friction and induce lubricity into the system. It can also improve the surface roughness and hardness properties.

2.5 PROPERTIES OF ELECTROLESS NICKEL COATING

2.5.1 HARDNESS EVALUATION OF THE COATINGS

Keong et al. [47, 48], observed that the electroless nickel coatings are characterized by high hardness and wear resistance. Their characteristics are further improved by heat treatment. The hardness of electroless Ni-P coatings depends on the phosphorous content. Hardness of electroless Ni-P coating increases with decrease of phosphorous content.

Yan et al. [49] investigated the hardness of the coating at first increases with the increase in phosphorous content up to 8% and then decreases shown in Fig.2.1. The maximum hardness attained by the as-deposited coating was found to reach a value of 910 $HV_{0.1}$. Hardness ($HV_{0.1}$) of electroless Ni-P coatings with different content of phosphorous is shown in Table2.1.

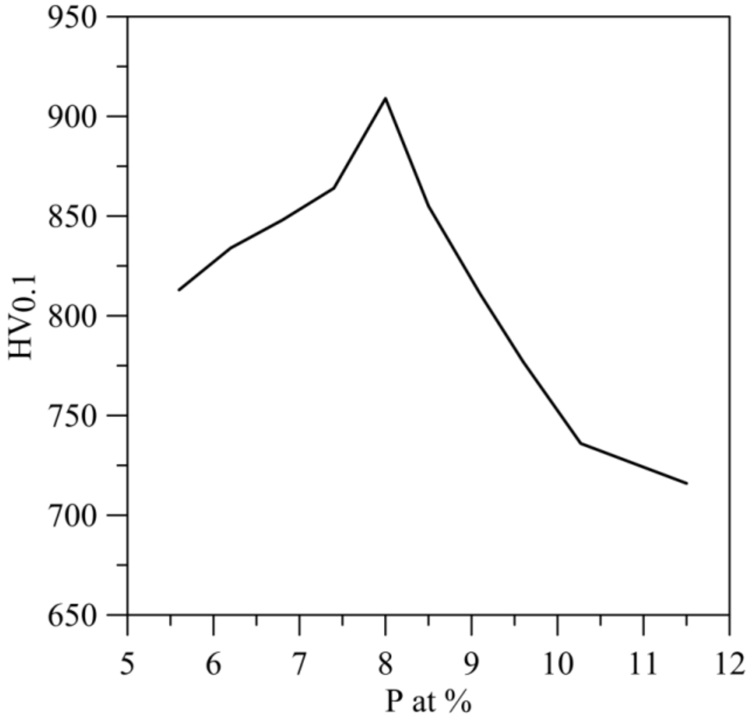


Fig. 2.1 Effect of phosphorous content on Micro hardness of Ni-P coatings

Table 2.1 Hardness ($HV_{0.1}$) of electroless Ni-P coatings

Phosphorous content in percentage	As-deposited	Heat-treated (400°C/1h)
2-3	650	1200
6-9	620	1100
10-12	570	1050

Ashassi Sorkhabi and Rafizadeh [50] found the thickness of coating increased with increasing coating time. The hardness of the coatings increases from approximately 502–650 HV with an increase in coating times and hardness of Ni–P rise with heat treatment from 790 to a maximum of 1045 HV at 400°C.

2.5.2 ROUGHNESS EVALUATION OF THE COATING

Mallory and Hadju [51] studied that the surface roughness has a large impact on the mechanical properties such as fatigue behaviour, corrosion resistance, creep life, etc. It also affects other functional attributes of machine components such as friction, wear, light reflection, heat transmission, lubrication, electrical conductivity, etc. Electroless nickel deposition has become commercially important for finishing steel, aluminum, copper, plastics and many other materials.

Vitry et al. [52] found the general roughness of the coating does not vary much from the roughness of the substrate. However, electroless coatings are suspected to have some smoothening effect above a critical substrate roughness. Electroless nickel coatings are found to present a very smooth surface. Heat treatments of the coatings caused a further decrease of average roughness.

Kanta et al. [53] again observed that after plating, the roughness of electroless coatings is significantly higher. But the dual Ni–P/Ni–B coating is smoother than the Ni–B coating. The use of a Ni–P pre-coating is believed to have a leveling effect on the etched surface and produces a smoother top deposit in the end.

Elansezhian et al. [54] found the presence of surfactants is to affect the surface roughness of electroless nickel coatings. Average roughness value of Ni-P coatings is found to reduce at higher concentration (>0.6g/l) of surfactants.

Sahoo [55] has studied the average surface roughness of electroless Ni-P coating on the basis of various coating process parameters. It was observed that concentration of nickel source has got the most significant influence for roughness characteristic of electroless Ni-P coating.

Sahoo [56] has employed five different roughness parameters, viz., centre line average roughness (Ra), root mean square roughness (R_q), skewness (R_{sk}), kurtosis (R_{ku}) and mean line peak spacing (R_{sm}) in order to study the surface texture generated in electroless Ni-P coating and minimize the same by optimizing the electroless bath parameters. It was found that concentration of the reducing agent and its interaction with concentration of the nickel source solution, have significant influence in controlling the roughness characteristics of electroless Ni-P coating.

Roy and Sahoo [57] investigated the chemical deposition of Ni-P-W coating on mild steel substrate and optimization of multiple surface roughness characteristics on the basis of different coating process parameters. Here, three coating parameters which are varied to have the optimum condition are concentration of nickel source, concentration of reducing agent, and concentration of tungsten source. Roughness of these coating is influenced not only by concentration of reducing agent and nickel source but also as tungsten source. It was found that tungsten is the most important parameter for roughness characteristic of Ni-P-W coating.

2.5.3 TRIBOLOGICAL STUDIES ON ELECTROLESS NI-P COATING

Electroless Ni-P coating offers good frictional and wear resistance and that is the most important property for which it is widely used. A number of works were done to study the friction and wear activities of such coatings. Along with these various researchers also studied the comparison of friction and wear behavior of electroless coating with other composite coatings which were developed by introducing other particles along with the Ni-P.

Sahoo and Das [58] investigated the tribological advances in different electroless nickel coatings based on the bath types, structure and also the tribo-testing parameters. A study of wear on electroless Ni-P coating revealed that the coating in the as deposited state exhibits high rates of wear. The wear resistance of electroless Ni-P coating which are heat treated increased considerably although small differences were obtained for the friction coefficient for the different heat treatment temperature.

Papachristos et al. [59] studied the sliding wear behavior of Ni-P-W composition modulated coatings established that the friction coefficient decreases as the load increases. Coatings with a smaller layer periodicity showed a better wear resistance than those with larger layer periodicities. Two major wear mechanisms were found: brittle fracture and delamination of the parts of the coating.

Balaraju and Seshadri [60] prepared electroless Ni–P coating by an acidic hypophosphite-based bath to produce 10–12% P in the electroless nickel coating. The wear resistance of the samples, in as-deposited and heat-treated (at 400ºC for 1 h) conditions are assessed using a disc-on-disc method under un-lubricated sliding conditions for 30min. A low specific wear rate of electroless Ni–P in heat-treated condition is observed when compared to that with as coated samples, which is considered to be due to very low mutual solubility of nickel phosphide and iron thus presenting a relatively incompatible surface.

Straffelini et al. [61] investigated the friction and wear behavior of several electroless Ni-P composite deposits. In addition to Ni-P deposit, deposits containing SiC, PTFE, MoS_2 and BN particles were produced. The sliding behaviour of the reference Ni–P deposit develops through two stages. During stage I, the deposit undergoes a mild abrasive wear whereas during stage II wear is severe and characterized by the brittle detachment of debris. The friction coefficient of Ni-P / PTFE co-deposit was found to be very low as 0.07. The Ni-P / SiC-PTFE co-deposit displays better sliding properties than the reference Ni-P deposit but worse than that of the Ni-P / MoS2 and Ni-P / PTFE co-deposits. The best rolling–sliding surface durability is displayed by the Ni–P/BN co-deposit

Bozzini et al. [62] studied the wear resistance in dry sliding condition of as plated and heat treated Ni-P and Ni-P / B_4C coatings in a slider on cylinder tribometer. Ni-P coatings showed a lower wear resistance than the corresponding composite coatings. The wear resistance increases for both the coatings after the heat treatment.

Panagopoulos et al. [63] observed the lubricated sliding wear behavior of Ni-P-W multi-layered alloy coating produced by pulse plating was done under loading conditions of 2-110N with a coating thickness of 25µm and with a sliding speed of 14-90 cm/s. Frictions coefficients were found to within the range of 0.035-1.0. Three lubrication modes were identified in this study such as elastohydrodynamic, mixed and boundary lubrication. No wear is basically observed in elastohydrodynamic mode; low wear occurred at mixed lubrication mode and severe wear found to occur during the boundary lubrication mode. In mixed lubrication mode the friction coefficient was mostly determined by the sliding speed while wear is mainly dependent on load, if other test parameters remain constant.

Quercia et al. [64] investigated the sliding friction test of electroless Ni-P coating. It showed smooth variation of friction coefficient (0.16-0.21) when tested under 2-10N loading conditions which revealed that Ni-P electroless coating better performs in sliding friction and wear.

Taheri et al. [65] investigated the tribological characteristics of electroless Ni-P coatings. It was concluded that EN coatings follow the substrate surface profile rather than just fill the spaces between surface asperities and the surface roughness value is independent of the coating thickness. The surface roughness parameter is practically identical of the substrate and the EN coating. EN coatings lower the kinetic coefficient of friction of the substrate, irrespective of their heat treated condition. It was also found that the degree of hardening of EN deposits at a given temperature depends on phosphorous content and aging time. The results further showed that, unlike conventional electroplated deposits, EN coatings do not subvert the substrate surface shape and profile even for large thickness coats. It was found that the wear resistance of EN coatings is significantly affected by heat treatment.

Narayanan et al. [66] developed duplex coatings with Ni-P and Ni-B in order to study the friction and wear behavior and to compare with Ni-P and Ni-B coating of similar thickness. It was found that the duplex coatings were uniform and the micro-hardness, wear resistance and corrosion resistance of the duplex coating is higher than Ni-P or Ni-B coating of similar thickness. Among the two types of duplex coatings studied, hardness and wear resistance is higher for coating having Ni-B coating as the outer layer whereas better corrosion resistance is offered by coatings having Ni-P coating as the outer layer.

Yang et al. [68] studied the tribological properties of the Ni-P / single-walled carbon nanotube composite coating compare with Ni-P coating it showed that the composite coating should not have only higher wear resistance but also lower friction co-efficient and provided higher corrosion resistance than Ni-P coating.

Xu et al. [69] has shown that electroless Ni-P-Nanometer Diamond composite coatings exhibit not only high wear resistance but also low frictional coefficient compared with the Ni-P coating.

Wang et al. [70] experimented on friction and wear behavior of electroless nickel coating under lubricated condition. In an experiment on sliding wear behavior used a range of normal loads and a range of sliding speeds at lubricated contact. Here they used Energol GRXP 100 as lubricant and friction coefficients in the range of 0.035 to 0.1 were recorded. All friction coefficients show a running-in period, during which they take higher values, before entering a more steady-state period.

Sahoo and Pal [71] are carried out by utilizing three test parameters, namely, normal load, operating speed and duration of machining to study the behaviour of electroless Ni–P coating with respect to friction and wear characteristics. Here the researchers observed that all the three test parameters have significant contribution in controlling the friction and wear behaviour of electroless Ni–P coating.

Alirezaei et al. [72] investigated the co-deposition of alumina particles within Ni-P coating matrix which changed the roughness and surface morphology and hardness and wear resistance also increased and it was seen that heat treatment of Ni-P-Al_2O_3 composite coatings only caused the phase transformation of the Ni-P matrix. Abrasive wear is the most dominant mechanism of wear for Ni-P-Al_2O_3 coatings.

Sahoo [73] observed the friction characteristics of electroless Ni–P (EN) coatings sliding against steel and optimization of coating process parameters based on the Taguchi method. It is observed that concentration of nickel source solution has the most significant influence in controlling friction characteristics of EN coating. The optimum combination of process parameters for minimum friction coefficient is obtained from the analysis. It is seen that concentration of nickel source solution has got the most significant influence on friction coefficient at the confidence level of 95% while bath temperature is significant at the 75% confidence level within the specific test range.

Sahoo [74] investigated an experimental study of roughness characteristic of electroless Ni-P coating. Experiments are carried out by utilizing the combination of three process parameters, bath design, concentration of nickel source solution and concentration of reducing agent. It is observed that concentration of nickel source solution has got the most significant influence in controlling roughness characteristic of electroless Ni-P coating. The optimum combination of process parameters for minimum roughness is obtained from the analysis.

Sahoo [75] investigated the roughness characteristics of electroless Ni-P coatings. Results show that concentration of the reducing agent and its interaction with concentration of the nickel source solution has significant influence in controlling the roughness characteristics of electroless Ni-P coating. Grey-based Taguchi method is found to optimize the coating parameters fairly well. No significant change in nickel and phosphorous content of coatings occurs with annealing. The Ni-P deposit is nanocrystalline in the as-plated condition, and upon heat treatment at 400^{o}C it produces Ni_5P_2, Ni_2P, and NiP_2 as major compound constituents.

Sahoo [76] investigated an experimental study of wear characteristics of electroless Ni–P coatings sliding against steel. Taguchi orthogonal array is employed to optimize the coating process parameters with respect to wear behaviour. It is seen that annealing temperature and bath temperature have the most significant influence in controlling wear characteristics of electroless Ni–P coating. The interaction of bath temperature and concentration of nickel source solution has some significant influence.

Sahoo and Das [77] reviewed that electroless nickel coatings have emerged as suitable coatings that can serve as feasible replacements to the conventional electroplating in suitable situations. Their properties such as hardness, low friction, wear resistance and corrosion resistance have led to their usage in tribological applications. Besides, the uniform deposition and the ability to coat any materials have served as an added advantage to their application in various areas. The advantages of modifying the properties of electroless nickel coatings by suitable surface treatments (heat treatment, laser treatment, etc.) and the incorporation of various elements (copper, tungsten, etc.) and particles (SiC, TiO_2, Si_3N_4, etc.) have been utilized by various researchers to evaluate the suitability of these coatings for various applications. The review reveals that the electroless coatings are mainly applied for wear resistance and corrosion resistance applications. The coatings have been found useful in marine environments as well as for coating of small arms weapons to be used in desert and dusty areas.

2.6 SCOPE OF PRESENT WORK

From the extensive literature review it is reflected that the characteristics of glass fiber reinforced composites and electroless Ni-P coating have currently become an active field of research since past decade. Many researchers have paid the attention to electroless Ni-P coating on convention materials due to superior qualities as a coating material. Although there have already been many contributions in the study of Ni-P as a coat material, some areas still need to be enlightened more clearly. One of the areas where there is still much scope of work to be done is the tribological properties of the Ni-P coatings on composite materials in terms of their friction and wear characteristics. It is seen through the literature review that very little work has been done in this regard. The present work is a humble attempt in that direction.

CHAPTER-3

EXPERIMENTAL METHODS

This chapter provides an overview of experiments, procedure of material production, design factors, selection of process parameter, coating process, tribological tests and instruments used for the experiment.

3.1 INTRODUCTION

The study of wear and frictional behavior of polymer based composites have gained interest in various engineering components due to their flexibility, improved strength-weight ratio, corrosion resistance, easy to manufacture, light weight and heat resistance etc. as mentioned in earlier chapter. The use of polymer based composites has found to be very effective in situation where sliding contact wear involved. The polymer based materials are preferred in recent years over metal-based counterparts in view of their low coefficient of friction and ability to sustain loads. Electroless Ni-P coating improved properties likes hardness, wears resistance, coating uniformity and corrosion resistance makes this a coating of choice for many engineering applications such as aerospace, automotives, oil and gas processing, food processing, microelectronics, machinery and metallization of plastic.

This present study gives the procedure to develop the glass fiber reinforced epoxy composite and the deposition of electroless Ni-P coating on glass fiber reinforced epoxy composite substrate. Then the tribological behaviour of the glass fiber reinforced epoxy composite and electroless nickel phosphorous (Ni-P) coatings on GFRE composite are experimentally evaluated in terms of friction and wear characteristics under dry condition. Before tribological test, surface roughnesses of the speciments are measured by the Talysurf Profilometer. The surface roughness played an important role in deciding the wear and friction minimization. Tribological tests are carried out for different combinations of the tribo tester parameters – load, speed and constant time. The surface morphology study is done by the SEM, composition analysis is studied by the EDX and XRD analysis is studied. In this chapter, the details of sample preparation, tribological tests and microscopic analysis have been discussed.

3.2 SELECTION OF PROCESS PARAMETER AND RESPONSE VARIABLES

The tribological performance of glass fiber reinforced epoxy composite and the electroless Ni-P coating on glass fiber reinforced epoxy composite are affected by the several test parameter such as load, speed, time, temperature and track diameter of rotating disc. From extensive study of the literatures, it is seen that applied normal load, sliding speed and time of sliding are the most widely used parameters by researchers to control the tribological behavior of glass fiber reinforced epoxy composite. Five levels of the test parameters are considered in the present study under fixed time frame. The output parameters are wear depth and coefficient of friction of both bare and coated composites. The wear depth is displayed in real time on the computer interfaced with the tribo-tester. The friction force is also displayed. Coefficient of friction is obtained by dividing the friction force with the applied normal load. The design variables and their levels have been shown in Table 3.1.

Table 3.1 Design variables and their levels.

Design factors	**Unit**	**Levels**				
		1	**2**	**3**	**4**	**5**
Load	N	20	30	40	50	60
Speed of rotating disc	RPM	60	70	80	90	100

3.3 MANUFACTURING PROCESS OF GLASS FIBER REINFORCED EPOXY COMPOSITE BY HAND LAYUP METHOD

The hand lay-up is one of the oldest and most commonly used methods for manufacturing of composite. Unidirectional or woven fibers and polymer matrix are combined to result in material exhibited desirable properties in one or more directions. Each layer is oriented to achieve the maximum utilization of its properties. Layers of different materials (different fibers in different directions) can be combined to further enhance the overall performance of the laminated composite material. In hand lay-up method, a very thin plastic sheet of few microns is cut and sticks both inner parts of the top and bottom cover of the mold which also give good surface finish of the product. Woven glass fibers reinforcement is cut as per the mold size and placed at the surface of mold. Then epoxy in liquid form is mixed with hardener (curing agent) 10:1 weight ratio by using wooden stick and poured onto the glass reinforcement already placed in the mold. The polymer is uniformly spread with the help of wooden stick. Second layer of glass fibers is then placed on the first layer and poured epoxy resin on the second layer and a wooden stick is moved with some pressure on the mat-polymer layer to remove any air trapped as well as the excess epoxy resin present. The process is repeated for each layer of glass fiber, till the required layers are stacked. After placing the plastic sheet on required finish layer, the top mold plate is then kept on the stacked layers and required pressure is applied. After curing at room temperature, mold is opened and the developed composite part is taken out for further processed. For epoxy based system, normal curing time at room temperature is 24 to 48 hours. Simple and low cost instruments are required for hand layup process. In this experiment, high speed steel is used to develop mold cavity and required thickness can be adjusted using variable thickness of the plates. An experimental set-up of hand layup method is shown in Fig.3.1 (a)-(b).

3.3.1 ADVANTAGES OF HAND LAYUP METHOD

- The process results in low cost tooling with the use of room-temperature cure resins.
- The process is simple to use.
- Any combination of fibers and matrix materials are used.

3.3.2 DISADVANTAGES OF HAND LAYUP METHOD

- Since the process is worked by hands, there are safety and hazard considerations.
- The resin needs to be less viscous so that it can be easily worked by hands.
- The quality of the final product is highly skill dependent of the labours.
- Uniform distribution of resin inside the fabric is not possible. It leads to voids in the laminates.

3.3.3 CHEMICALS USED

A. Epoxy Resin and Hardener

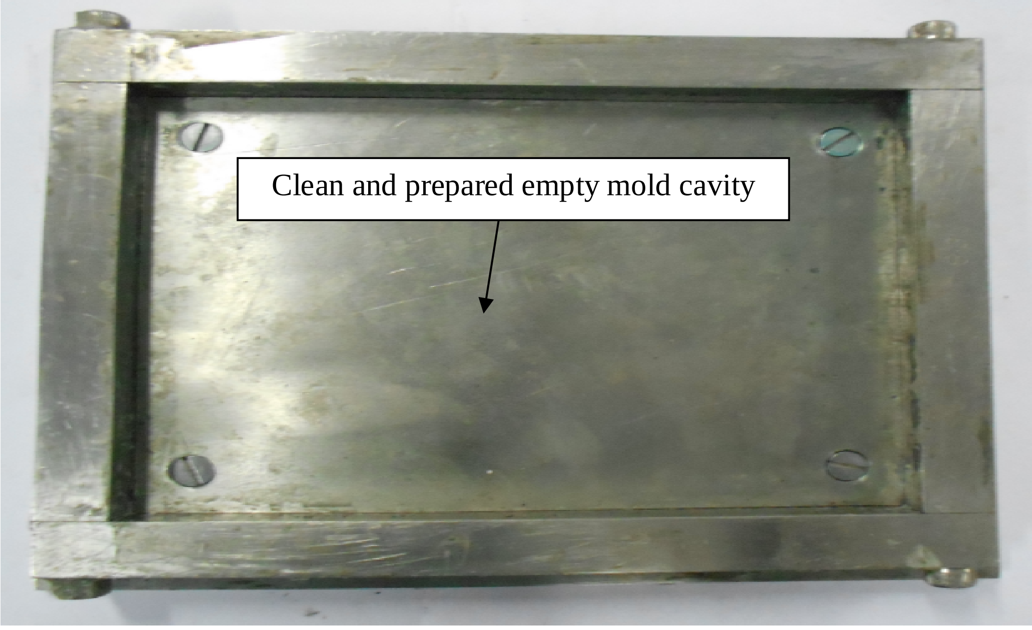
Epoxy Resin- ARALDITE CY-205 manufactured by Humtsman Advanced Material (India) Pvt. Ltd, density-1.27gm/cm^3, glass transition temperature (Tg) - 108°C.

Hardener HY-951, Density- 0.94 gm/cm^3 mixed in ratio of 10:1 (epoxy: hardener)

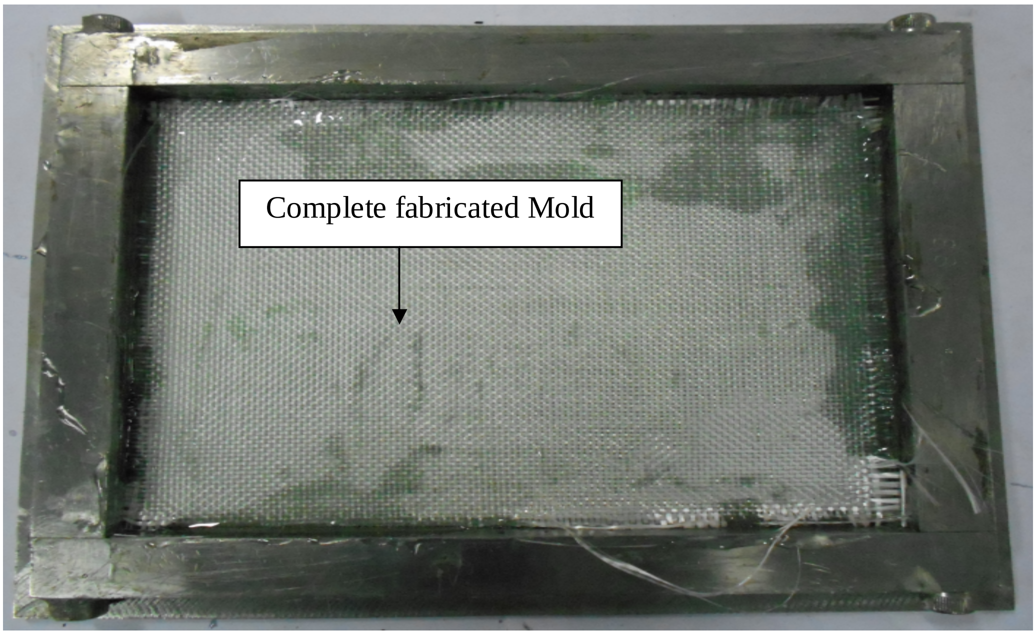
B. Glass Fiber

Woven fabric E-glass fiber, 300g/m^2

C. AcetoneCH_3COCH_3 , M= 38.08g/mol



(a)



(b)

Fig. 3.1 (a) Empty mold for hand layup method, (b) Mold with fabrication of composite.

Hand layup process consists of the following instruments:

a. **Mold** (Made by HSS to avoid bending): Mold used for making the desired shape and size. Mold dimension: Area= 200mm×150mm and Depth=15mm

b. **Consumable materials:**
   - very thin plastic sheet(60 microns, 1500 mm×100m length)
   - wooden stick
   - hand gloves
   - Brush
   - Clean jute
   - Cello tape (Made by Millennium Speciality Packaging Limited)

c. **Mixing Container** (Borosil made glass biker): Used for mixing of epoxy, hardener and acetone and to obtain the epoxy resin mixture.

d. **Electronic Balance** (Made by Satwik Scale Industries): To measure weights of glass fibers, epoxy, hardener chemicals and composite.
   Model No-VIK102, Serial No-NE4506, Class-II, high accuracy
   Max. load - 10kg, Min. load- 20gm.

e. **Chemicals** :Used for matrix
   - Araldite CY 205 IN, Batch No- AL40684, ID No- 24587325
   - Aradur HY 951, Batch No- 2001530

f. **Acetone GR** (C_3H_6O), MW-58.08 g/mol, Batch No- TT-538642

g. **Reinforcing fibers** (Woven fabric E-glass fiber): Used for reinforcement.

h. **Scissors**(Delux): Used for cutting

i. **Dead weight:** Used for compression

3.3.4 METHOD OF OBTAINING GLASS FIBER REINFORCED EPOXY COMPOSITE BY HAND LAYUP METHOD

Hand lay-up technique is the simplest method of composite processing. The infrastructural requirement for this method is very minimal. The fabrication processes are described in details.

3.3.4.1 WORK STATION PREPARATION

An initial preparation is needed for all the materials and tools that are going to be used is a fundamental standard procedure when working with composites. The main cause is that once the resin and the hardener are mixed, the working time is limited by the speed of the hardener chemically reacting with the epoxy producing an exothermic reaction.

3.3.4.2 CUTTING AND POSITIONING THE REINFORCEMENT

Reinforcement fibers are cut the appropriate size of the moulding and always tried to make the part using a single piece of fabric for each layer. The glass fibers kept in a roller rack is measured and cut on a wooden platform according to the mould dimension (200mmx150 mm) by steel rule and scissors to ensure not to change the actual orientation of the fiber. After cutting all fabric, dimension is checked carefully that it 200mm length and 150mm width. Layers of fabric are placed one by one into the mould in same orientation of the fibers. The total glass fiber quantity is defined as approximate 60% of the total weight of the fabricated composite. We have placed 16 layers of E-glass fiber woven fabric which have 176 gm weight for making 6mm thickness composite and maintain the 60:40 ratio of glass fibers and epoxy resin.

3.3.4.3 PREPARATION OF MOLD SURFACE

Mold is placed on the wooden platform or working station. Mold is cleaned with the help of clean cloth, acetone and jute. After cleaned the mold surface, plastic sheets are tapped with the help of cello tape to the both inner parts of the top and bottom cover of the mold. This plastic film is performed the similar function of releasing agent in a better way as there is every possibility of infusion of releasing agent into the glass fiber.

3.3.4.4 LAY-UP PROCESS

The first step is to mix the resin and the hardener. The proportions are usually given by the supplier and can be found on the containers of the hardener or resin. The proportion can be either measured by weight or by volume but it is important to follow these proportions exactly as this is a complete chemical reaction and all components must react completely for maximum strength of the matrix. In this case the epoxy and hardener are mixed with 100:10 weight ratios. The total epoxy resin quantity is defined as approximate 40% of the total weight of the fabricated composite. The mixing is performed manually in the mixing containers with the help of wooden stick very slowly for ten to fifteen minutes so that any excess air bubbles enter into the resin. Acetone is added for lowering down the viscosity of the epoxy in order to ensure the easy diffusion of the mixture into the Glass fiber stack.

After mixed up adequate quantity of resin and hardener (10:1) is deposited in the mold and a brush is used to spread it around all surface. It is important not to add too much resin which cause thick layer of resin, nor to add less than the necessary amount which cause holes in the surface of the part when it is cured. A definite amount of resin is deposited to wet the glass fibers.

The first layer of glass fiber reinforcement is then laid. This layer is completely wetted with epoxy resin mixture and then softly press using a brush makes the resin that was added in the previous step wick up through the fiberglass cloth. After that a second layer of glass fiber is added and special care is taken to eliminate all possible air bubbles. This step is repeated until the desired thickness is obtained. The sixteen number of glass fiber layers are added to build the final laminates and total thickness is 6mm. The top cover of the mold is placed on the final layer and compressed the mold by using 400N dead weight. The compression ensured that the entrapped air bubbles are completely removed and the excess resin flows out. The amount of excess resin is also calculated to account the actual quantity of resin used. The mould is left for thirty hours at room temperature and then material is taken out from the mould and allows at room temperature for twenty four hours completing the curing process. The details of the composites fabricated regarding the matrix and reinforcement material are given in the Table 3.2.

Table 3.2 Compositions of the Composite materials

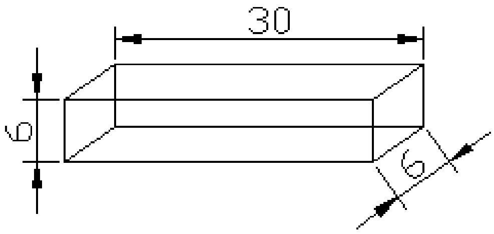
Materials	Glass fibers	Epoxy resin
Wt%	60	40

3.4 DEPOSITION OF ELECTROLESS Ni-P COATING

3.4.1 PREPARATION OF THE SUBSTRATE MATERIAL

The preparation of the substrate is an essential step for the proper development of any coating on its surface. Glass fiber epoxy composite specimens are used as the substrate material for the deposition of the electroless Ni-P coating. A schematic diagram of the specimen is shown in

Fig.3.2. Size of the specimen (6mm×6mm×30mm) is cut from a glass fiber reinforced epoxy composite plate and prepared very carefully for the deposition of the electroless Ni-P coating. This specific dimension of the substrate is selected in accordance with its counterpart in the tribo-tester apparatus where the sample has to be fixed for tribological testing. Specimens of specified dimension are cut by a diamond cutter in a specially designed and fabricated machine for this purpose. Finish cut of the samples are carried out with the help of fine hacksaw, flat file, sandpaper. Final finishing is done with the help of fine grade silicon carbide paper. The substrates are washed with soap and water to clean off any dirt particles. After that, acetone is employed to clean any remaining organic products. Finally before coating the substrates are subjected to pickling treatment in dilute hydrochloric acid for one minute to remove rust and other oxide layers.



- Surface area of this specimen= 2(0.6×0.0.6) + 4(0.6×3) =7.92cm^2

Fig. 3.2 Specimen dimension details

3.4.2 CHEMICALS USED

The chemicals used to prepare the solution for electroless Ni-P bath are

1) Nickel sulfate ($NiSO_4$, $6H_2O$): Merck Specialities Pvt. Ltd., Mumbai, India.
2) Nickel chloride ($NiCl_2$, $6H_2O$): Merck Specialities Pvt. Ltd., Mumbai, India.
3) Sodium hypophosphite (NaH_2PO_2, H_2O): LobaChemie Pvt. Ltd., Mumbai, India.
4) Sodium succinate ($C_4H_4Na_2O_4$, $6H_2O$): LobaChemie Pvt. Ltd., Mumbai, India.
5) Palladium Chloride ($PdCl_2$): Merck Specialities Pvt. Ltd., Mumbai, India

Nickel sulphate and nickel chloride salts are generally used as the source of nickel ion, whereas sodium hypophosphite acts as the reducing agent. In an electroless system, the reducing agent is a very important component as it supplies the necessary electrons for reducing the nickel into its free form. The sodium hypophosphite cost is lower and gives better corrosion resistance. The presence of foreign particles or unwanted precipitations resulting from the breakdown of bath reagents may lead to spontaneous decomposition of the electroless bath during plating. To prevent the breakdown of bath during coating, sodium succinate is used to keep the solution stable for the deposition time of 2 hour or may be more. Palladium chloride is used as an activator.

COMPOSITION OF BATH

Ranges of chemicals for stable solution for Ni-P coating on glass fiber reinforced epoxy composite samples is shown in Table 3.3.

Table 3.3 Ingredients of electroless bath and their ranges.

Sl. No.	Parameters	Range of parameters
1	Nickel sulphate	15 g/l
2	Nickel chloride	15 g/l
3	Sodium hypophosphite	10 g/l
4	Sodium succinate	12 g/l
5	Deposition temperature	80±2ºC

3.4.3 EXPERIMENTAL SETUP FOR ELECTROLESS NI-P DEPOSITION

Digital Balance: High precision electronic balance was used to measure weights of substrates and chemicals.
Afcoset, Model No. ER-182A, SL. No. 0108017, Max. range 180 g, Min. range 0.01mg (shown in Fig. 3.3).

Magnetic Stirrer: It is used for proper mixing of the bath solution.
1MLH Magnetic stirrer, Manufactured by Remi Instruments.

Electroless Bath Heater: Used for heating the electroless Ni-P bath (shown in Fig. 3.4).

Thermometer: To measure the temperature of bath temperature during the coating process.

Glass Beakers: Used to hold the bath solution (Borosil).

Fig. 3.3 Digital balance

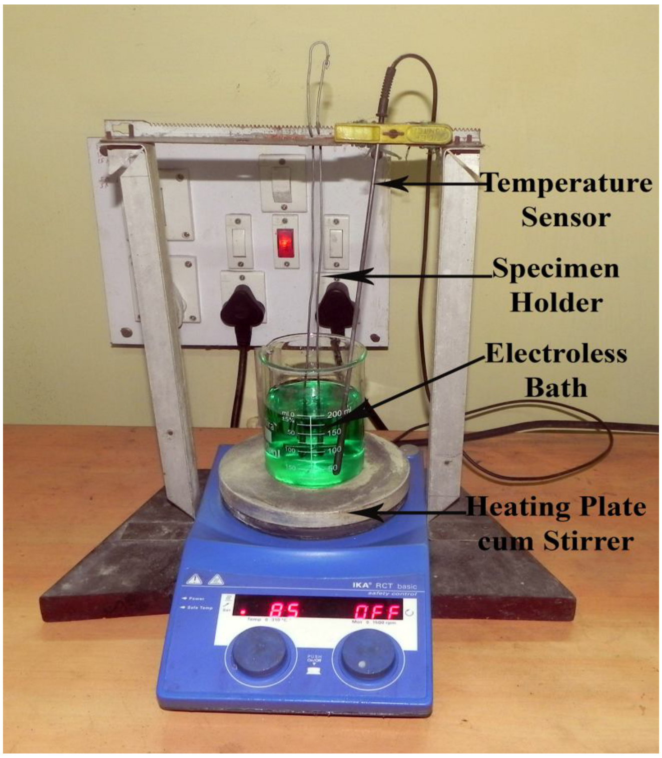


Fig. 3.4 Setup for electroless deposition

3.4.4 METHOD OF OBTAINING ELECTROLESS NI-P COATING ON GFRE COMPOSITE

The following procedure has been adopted for the deposition of electroless Ni-P on the glass fiber reinforced epoxy composite specimen:

- The glass fiber reinforced epoxy composite specimen, after thorough cleaning is given a pickling treatment with dilute hydrochloric acid for one minute. Subsequently, they are rinsed in distilled water prior to coating.
- One 250 ml bicker properly cleaned and 200ml distilled water was taken in it. An adequate amount (Table- 3.3) of Nickel Chloride, Nickel Sulphate, Sodium Hypophosphite and Sodium Succinate are added in the bicker which is placed on the magnetic stirrer for ten minutes.
- The prepared 200ml of the electroless bath solution was then placed on a temperature controllable heater. The solution is heated at required temperature 80ºC by using the digital heater which has also a temperature sensor.

- Activation solution (Palladium chloride) was taken in a separate beaker and heated to a temperature of about 50ºC - 55ºC.
- The cleaned glass fiber epoxy composite samples were first rinsed in distilled water and then dipped in the activation solutions for about a few seconds and again rinsed in distilled water.
- Activated samples are merged into the above 200ml solution which is heated at the steady temperature (80ºC) by digital heater.
- The temperature of the solution is checked at regular intervals by using a thermometer.
- After two hours of deposition the samples were taken out and rinsed with distilled water.

The above mentioned steps are repeated for all the specimens. The coating bath parameters are kept constant for all the specimens so that uniform thickness of the deposits is obtained. The obtained deposit thickness is found that the average thickness is within the range of 25 to 30µm which is necessary for carrying out tribological tests.

3.4.5 DEPOSITION MECHANISM

In case of electroless deposition processes where no external current supply is used. Although the mechanism of the electroless Ni-P deposition reactions taking place is still not well understood, the most widely accepted mechanisms are illustrated by the following equations [67].
In electrochemical mechanism the catalytic oxidation of the hypophosphite yields electrons at the catalytic surface which in turn reduces nickel and hydrogen ions as illustrated below

$$H_2PO_2^- + H_2O \longrightarrow H_2PO_3^- + 2H^+ + 2e^- \qquad (3.1)$$

$$Ni^{++} + 2e^- \longrightarrow Ni \qquad (3.2)$$

$$2H^+ + 2e^- \longrightarrow H_2 \qquad (3.3)$$

$$H_2PO_2^- + 2H^- + e^- \longrightarrow P + 2H_2O \qquad (3.4)$$

In atomic hydrogen mechanism the atomic hydrogen is released as a result of the catalytic dehydrogenation of hypophosphite molecule adsorbed at the surface as illustrated below.

$$H_2PO_2^- + H_2O \longrightarrow HPO_3^{--} + H^+ + 2H_{ads} \qquad (3.5)$$

$2H_{ads} + Ni^{++} \longrightarrow Ni + 2H^{+}$ (3.6)

$H_2PO_2 + H_{ads} \longrightarrow P + H_2O + OH^{-}$ (3.7)

The adsorbed active hydrogen, (3.6) reduces nickel at the surface of the catalyst.

$(H_2PO_2)^{2-} + H_2O \longrightarrow H^{+} + (HPO_3)^{2-} + H_2$ (3.8)

The reduction mechanism of metal ions by hypophosphite involves two reactions, i.e., hypophosphite ions are catalytically oxidized and nickel ions are reduced at the catalytic surface. A part of released hydrogen is absorbed onto the catalytic surface and this is an anodic reaction. The nickel ion at the surface of the catalyst is then reduced by the absorbed active hydrogen and this is a cathodic reaction. Some of the adsorbed hydrogen reduces a small amount of the hypophosphite ion at the catalytic surface into water, hydroxyl ion and phosphorus. Most of the hypophosphite present is catalytic, which in turn is oxidized to orthophosphate and gaseous hydrogen, independent of the deposition of the nickel and phosphorus [78].

3.4.6 MEASUREMENT OF COATING THICKNESS

W_b = Mass of sample in gm before deposition.

W_a = Mass of sample in gm after deposition.

$W_a - W_b = W$, Difference of masses in gm/Deposited mass.

Deposition rate = $\frac{W}{2A} = \frac{W_a - W_b}{2A}$ gm/cm^2

Deposition thickness= Y= $\frac{W \times 10^4}{2A\rho}$ in µm (3.9)

Where ρ is the density of Ni-P coating, ρ = 8gm/cm^3

'A' is the surface area of the sample = 7.92cm^2.

3.4.7 MEASUREMENT OF SURFACE ROUGHNESS

The surface roughness of a material literally means its surface texture. The deviation of an actual surface from its ideal one along the normal vector to the surface is known as surface roughness. A larger deviation means higher surface roughness. Since tribology is the science of interacting surfaces, the surface roughness is an important factor in deciding the fate of a material used for tribological purposes such as wear reduction and friction mimization. Roughness is often a good predictor of the performance of a mechanical component, since irregularities in the surface may form nucleation sites for cracks or corrosion. Sometimes higher surface roughness proves to be beneficial. A higher surface roughness means higher wear and coefficient of friction between the interacting surfaces. A higher surface roughness of the substrate material on the other hand, promotes higher adhesion of electroless nickel coatings with the substrate material. Hence it is necessary to control the surface roughness of coatings used for tribological applications.

Surface roughness is commonly characterized by centre line average (R_a). To estimate the surface roughness of the Ni-P coatings, a typical stylus surface-measuring instrument is used. In this present study, centre line average roughnesses of the samples are measured. Center line average, R_a is the arithmetic mean value of the departure of the profile from the centre line along the sampling length given by the formula as follows:

$$R_a = \frac{1}{L}\int_0^L |z(x)| dx \tag{3.10}$$

Where, z (x) is the height of the surface above mean line at a distance x from the origin and L is the measured length of the profile as shown in Fig. 3.5. It is ensured that the specimens have a uniform and low surface roughness values so that no ambiguity creeps into the measured data for wear depth and friction coefficient. The surface roughness parameter R_a on the coated surfaces is measured using Talysurf (Make – Taylor Hobson, UK) shown in Fig. 3.6. The Talysurf instrument (Surtronic 3+) is a portable, self-contained instrument for the measurement of surface texture.

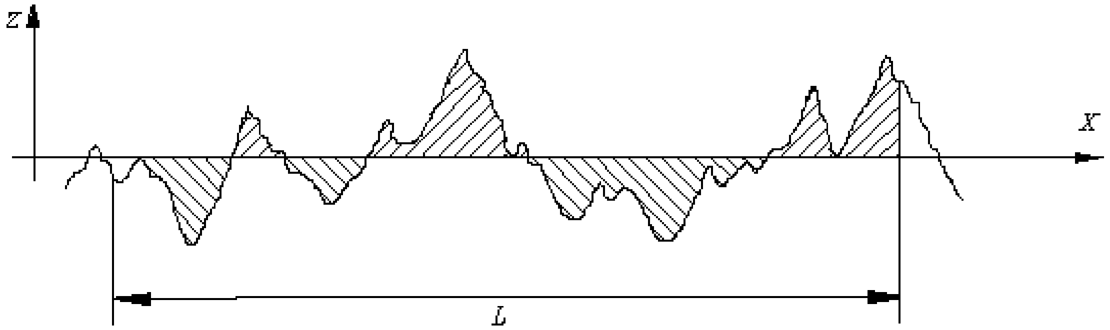


Fig. 3.5 Center line average of a surface over sampling length L.

In this study, the measurements are taken using 0.8 mm sampling length and 4 mm traversing length. Roughness measurements on the coated samples have been repeated at least four times and average of four surface roughness parameter values was recorded accordingly. The measured profile of Talysurf is digitized and processed through the advanced surface finish analysis software Talyprofile for evaluation of all the roughness parameters.

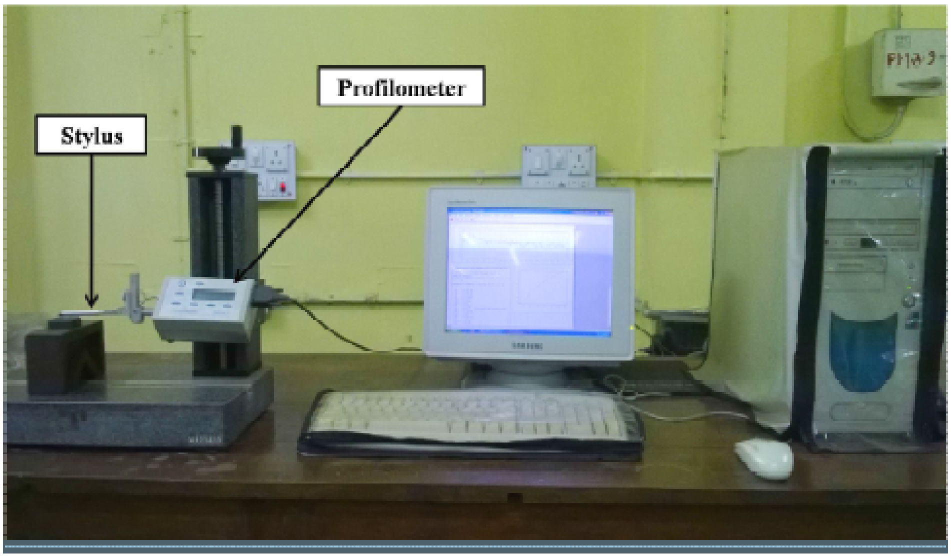


Fig. 3.6 Talysurf Profilometer.

3.5 MEASUREMENT OF FRICTION AND WEAR

Friction is the resistance to sliding between two surfaces in contact when pressed together. Friction is often described with a single coefficient which is the sliding resistance force divided by the normal pressure pushing the two surfaces together. This coefficient of friction (COF) is often considered a material property that is applicable across a broad range of pressures, velocities and temperatures. Wear on the other hand is generally defined as the loss of material or dimension or both of a surface due to the mechanical action of the interacting opposite surface. Wear, an important aspect of tribology is often the limiting mechanism of device service life. Hence, except in some limited cases, wear is an unwanted phenomenon and needs to be minimized if not completely eliminated.

The friction and wear test is carried out on a pin-on-disc type tribotester (WEAR AND FRICTION MONITOR, DUCOM, INDIA) with a computer interfaced data acquisition system (TR-20LE-CHM-400) at an ambient temperature of about 30^{o}C. The applied normal load and sliding speed are varied with a range of 20N to 60N and 60rpm to 100rpm respectively. The track diameter and test duration are fixed at 70mm and 10minute respectively. A photograph of the tribotester apparatus is shown in Fig.3.7. The specimen is held stationary with the help of an attachment provided. It presses against a rotating counter face disc of dimensions ϕ165mm × 8mm and material EN31 hardened to 60HRC, ground to 1.6Ra surface roughness. The speed and time for which the disc rotates can be controlled using the controller device provided with the tribotester. The normal load is applied by placing dead weights on the loading pan which is attached to a loading lever that transmits the normal load to the specimen at a ratio of 1:1 which means if one kg load is applied on loading pan, one kg load act on specimen. A load cell is used to measure the frictional force experienced by the specimen. An LVDT (Linear Variable Differential Transformer) is used to measure the displacement of the specimen which is a direct indication of the wear depth encountered by the specimen. Wear and frictional force are automatically recorded and their plots in real time are displayed on the computer screen. In the present case, wear is measured in terms of displacement (microns).

Similarly for the second set of experiment the friction and wear test is done at different loading and speed conditions. The factors and their levels are chosen based on studies from manuals of the tribotester, loading and speed conditions found from different research journals as used by different researchers.

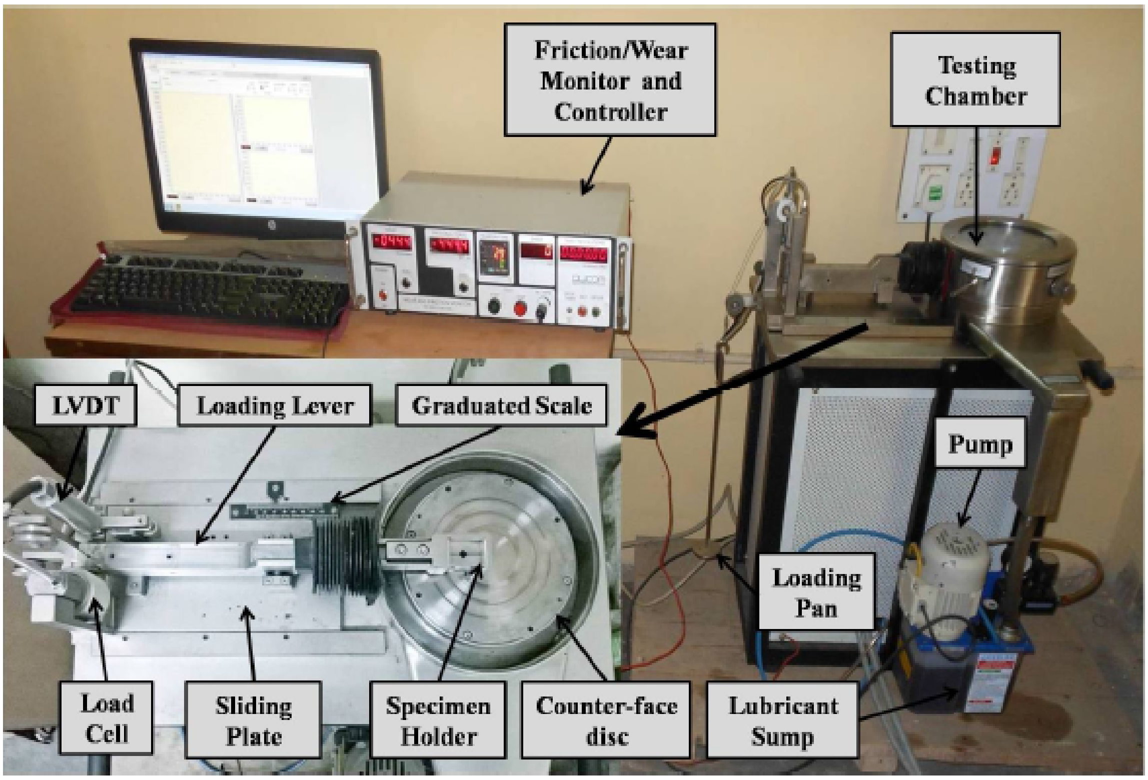


Fig. 3.7 Pin on disc tribotester.

3.6 STUDY OF MICROSTRUCTURAL ANALYSIS

The microstructure characterization of a material is mostly done by using scanning electron microscopy (SEM) images and it is very useful in order to study the microstructure in detail or the surface morphology of a material. In this method, the surface of a specimen to be examined is scanned with an electron beam, and the reflected beam of electrons is collected and then displayed at the same scanning rate on a CRT (cathode ray tube) monitor or a LCD (liquid crystal display). The image displayed on the screen represents the surface features of the specimen and are photographed for further examination. The magnification in SEM ranges from about 10 to 50000 times. Scanning electron microscopy examinations are carried out for the specimen surface to observe the morphology of wear tracks in comparison with that of specimen before test.

This particular study has been done to analyze the morphology of wear track of the glass fiber reinforced epoxy composite after and before tribology test and also analyzed the microstructure and wear morphology of Ni-P deposited GFRE composite before and after tribology test. A scanning electron microscope (JEOL, JSM – 6360) is used for this study which is shown in Fig.3.8.

Fig 3.8 Photograph of the Scanning Electron Microscope (SEM) Machine used

3.7 STUDY OF COMPOSITIONAL ANALYSIS

Energy dispersive X-ray analysis (EDX) is used in conjunction with a scanning electron microscope (SEM) as both shares a common electron source. In this process a flux of electrons instead of light is used to develop a magnified image of the observed object. When atoms on the surface of an object are bombarded by electrons, X-rays are emitted with photon energies that are unique to each chemical present. The X-rays are generated by jumps of electrons between electron shells in an atom that occur during external electron bombardment. When jumped electron returns to its equilibrium electron shell, a photon with energy dictated by quantum physics is emitted. The energy is sufficiently high to produce an X-ray photon. The penetration of electrons into a sample is typically in the range of 0.1 to 1μm and the elemental composition of coatings with this range of thickness can be accurately identified. EDX can be either used to analyze composition at a point or over a wide area. Specific elements can also be mapped over a test surface by scanning for X-ray emission at a specified energy.

This particular study is done for the compositional analysis of the electroless nickel phosphorous coating that is to study the percentages of nickel and phosphorous in the deposited coating as well as indicate whether the coatings are low or medium or high phosphorous coatings which will further help us for the evaluation of microstructure that depending upon the phosphorous contents what type of structure it is. The presence of other elements if any can also be detected.

3.8 STUDY OF XRD ANALYSIS

X-ray diffraction applies the ability of X-rays to be diffracted by a crystalline lattice since the wavelength of X-rays is comparable to inter-planar spacing. The diffraction pattern formed is characteristic to a specific crystalline structure and XRD is particularly useful for inorganic chemical analysis. Oxides, nitrides, carbides and sulphides of metals can usually be clearly distinguished by this technique. It is less effective for organic compounds. A diffract-meter consists of an X-ray source, a sample holder with ability for rotation and a series of counter to monitor the diffracted intensity. The X-rays used are monochromatic which is different from the X-rays used in imaging. Rotation of the sample facilitates detection of the diffracted pattern, which is a series of dark and light bands that denote destructive and constructive interference between scattered X-rays. The angular spacing between dark and light bands indicates the size of the inter-planar spacing, which is used to determine the compounds. XRD is best applied to bulk samples but can be used to analyze thicker coatings and power samples. Identification of compounds can be obtained by comparing the X-ray diffraction graph of unknown sample with JCPDS (joint committee on powder diffraction standards) file.

This particular study is done for the analysis of the electroless nickel phosphorous coating that is to study the compounds formed of nickel and phosphorous in the deposited coating. An X-ray diffraction analyzer is used for this study. The experimental set up for this analysis is shown in Fig.3.9

Fig.3.9 Photograph of the XRD machine used.

CHAPTER-4

RESULTS AND DISCUSSION

In this chapter, the variation of friction and wear of glass fiber epoxy composite and electroless Ni-P coating on GFRE composite with applied normal load and sliding speed have been analyzed. The results of SEM, EDX and XRD have been reported. Finally, the tribological results of GFRE composite are compared with the Ni-P coated GFRE composite under dry conditions.

4.1 INTRODUCTION

This chapter presents the results and discussion of friction and wears behavior of GFRE composites and coated GFRE composite under various loads and rotating speed. The corresponding friction and wear behaviors are compared to coated and bare GFRE composites. It also discusses the results of scanning electron microscope for analysis of microstructure, energy disperse x-ray for analysis of composition and x-ray diffraction.

Roughness is often made synonymous with terms such as uneven, irregular, coarse in texture and so on. Before introducing the specimens for tribological tests, surface roughness of the GFRE composite and coated composite specimens are measured randomly. Friction and wear are controlled by the roughness of the surface. The higher surface roughness indicates that the area of contact of the asperities is high which leads to higher values of wear. For ensuring the tribological results are not affected by the roughness of the contact surfaces, only the specimens which have a uniform and good surface roughness values are chosen. Centre line average roughness (R_a) values of GFRE composite is seen in the range of 1.01 to 1.33 µm and for the coated substrates in the range of 1.09 to 1.68 µm.

Experimental results of wear depth and friction coefficient of GFRE composite and coated GFRE composite in dry condition are illustrated in Table 4.1 and Table 4.2 respectively.

Table 4.1 Experimental results of wear depth and friction coefficient of GFRE composite.

Sl No	Load (N)	Speed (rpm)	Wear (µm)	COF
1	20	60	5.53	0.245
2		70	7.7	0.263
3		80	8.21	0.252
4		90	9.4	0.228
5		100	10.26	0.206
6	30	60	9.57	0.207
7		70	10.23	0.258
8		80	14.13	0.227
9		90	14.51	0.196
10		100	17.86	0.177
11	40	60	9.97	0.2035
12		70	22.12	0.286
13		80	25.46	0.21
14		90	29.47	0.18
15		100	33.27	0.167
16	50	60	10.77	0.1694
17		70	30.88	0.229
18		80	34.28	0.175
19		90	45.14	0.145
20		100	54.71	0.1456
21	60	60	15.21	0.1497
22		70	42.5	0.2056
23		80	48.56	0.102
24		90	72.34	0.1295
25		100	82.7	0.1694

Table 4.2 Experimental results of wear depth and friction coefficient of coated GFRE composite.

Sl No	Load (N)	Speed (rpm)	Wear(µm)	COF
1	20	60	3.47	0.314
2		70	4.31	0.3984
3		80	3.12	0.318
4		90	4.83	0.293
5		100	5.94	0.268
6	30	60	5.59	0.297
7		70	6.87	0.317
8		80	6.83	0.300
9		90	6.97	0.244
10		100	8.56	0.208
11	40	60	9.67	0.301
12		70	10.38	0.314
13		80	10.24	0.295
14		90	11.12	0.241
15		100	13.99	0.196
16	50	60	12.11	0.2912
17		70	12.31	0.286
18		80	15.16	0.266
19		90	16.16	0.223
20		100	19.57	0.155
21	60	60	15.07	0.268
22		70	16.21	0.279
23		80	18.23	0.254
24		90	19.2	0.219
25		100	22.27	0.155

4.2 MICROSTRUCTURE STUDY

Whenever a material is developed, it is necessary to characterize the same to ensure that the structure, chemical composition, etc. are exactly similar as desired. Moreover, characterizing a material also helps us to better understand its properties, physical behavior and performance on a certain perspective and explain the same. Microscopic techniques are in demand to study and characterize materials. These techniques are very coherent and fast and allow one to quickly determine the associations between the properties and structure of a material.

4.2.1 GFRE COMPOSITE

SEM observations were made to identify the surface morphology of the GFRE composite specimens before and after tests. Several specimens have chosen and observed under SEM. Fig.4.1 shows the SEM micrograph of the bare composite surface before test. There is no damage or breaking fibers and each and every glass fibers have its original shape and orientation and the distribution of the epoxy resin is uniform. The surface morphologies of different magnification for the condition of 40N load and 80rpm rotating speed are shown in Fig.4.2 (a)-(c). Fig.4.2 (a) shows the wear track, Fig.4.2 (b) shows the matrix is debonded from the composite due to adhesive and abrasive wear. Fig.4.2 (c) shows the fiber fracture, which seems to be a brittle failure that takes place with less proportion in the region of wear. The fibers were fractured along its own length. Material is plastically deformed under applied load.

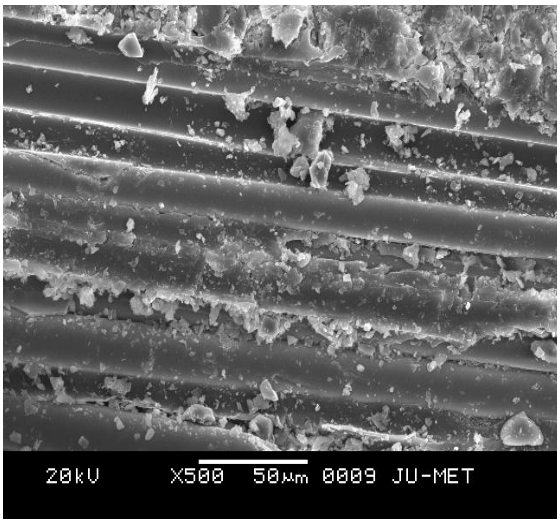
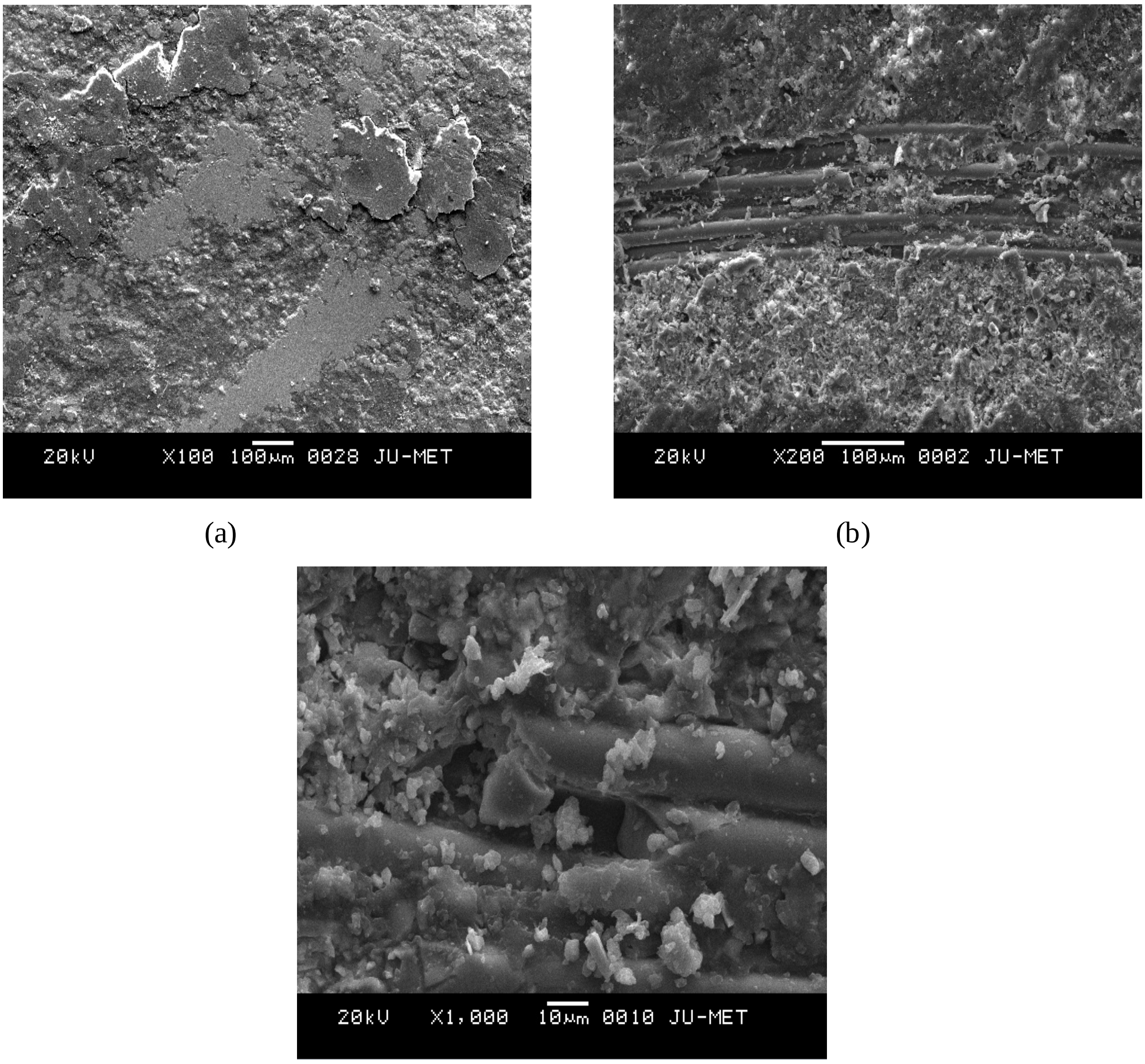


Fig. 4.1 SEM micrograph of GFRE composite before test at 500X magnification.



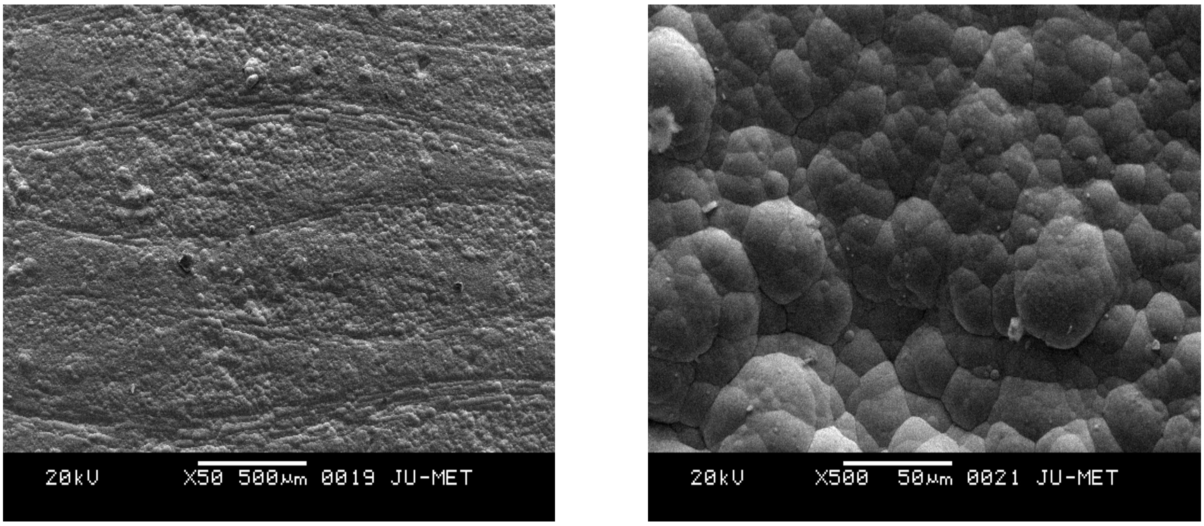
(a) (b)

(c)

Fig. 4.2 SEM micrographs of GFRE composite after test at a 40N load and 80rpm rotating speed at magnification of (a) 100X (b) 200X (c) 1000X.

4.2.2 COATED GFRE COMPOSITE

The microstructure of the coating is studied in order to ensure that the coating is developed without any defect and with the precise composition. The surface morphology of the developed electroless Ni-P coating is carried out in scanning electron microscope. Some of the samples are chosen randomly and observed under SEM. The SEM micrograph of coated GFRE composite surface is presented in Fig.4.3 (a)-(b). The SEM micrographs of the coated samples display that there are many globular particles on the coating surface of the specimens. The surface is optically smooth and of low porosity. No obvious surface damage is found. Hence, it can be concluded that the coating has been properly developed.



(a) (b)

Fig. 4.3 SEM micrographs of coated GFRE composite before test at magnification of (a) 50X (b) 500X

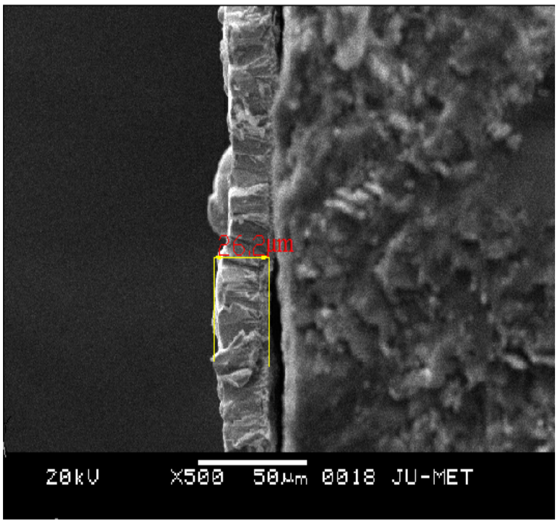
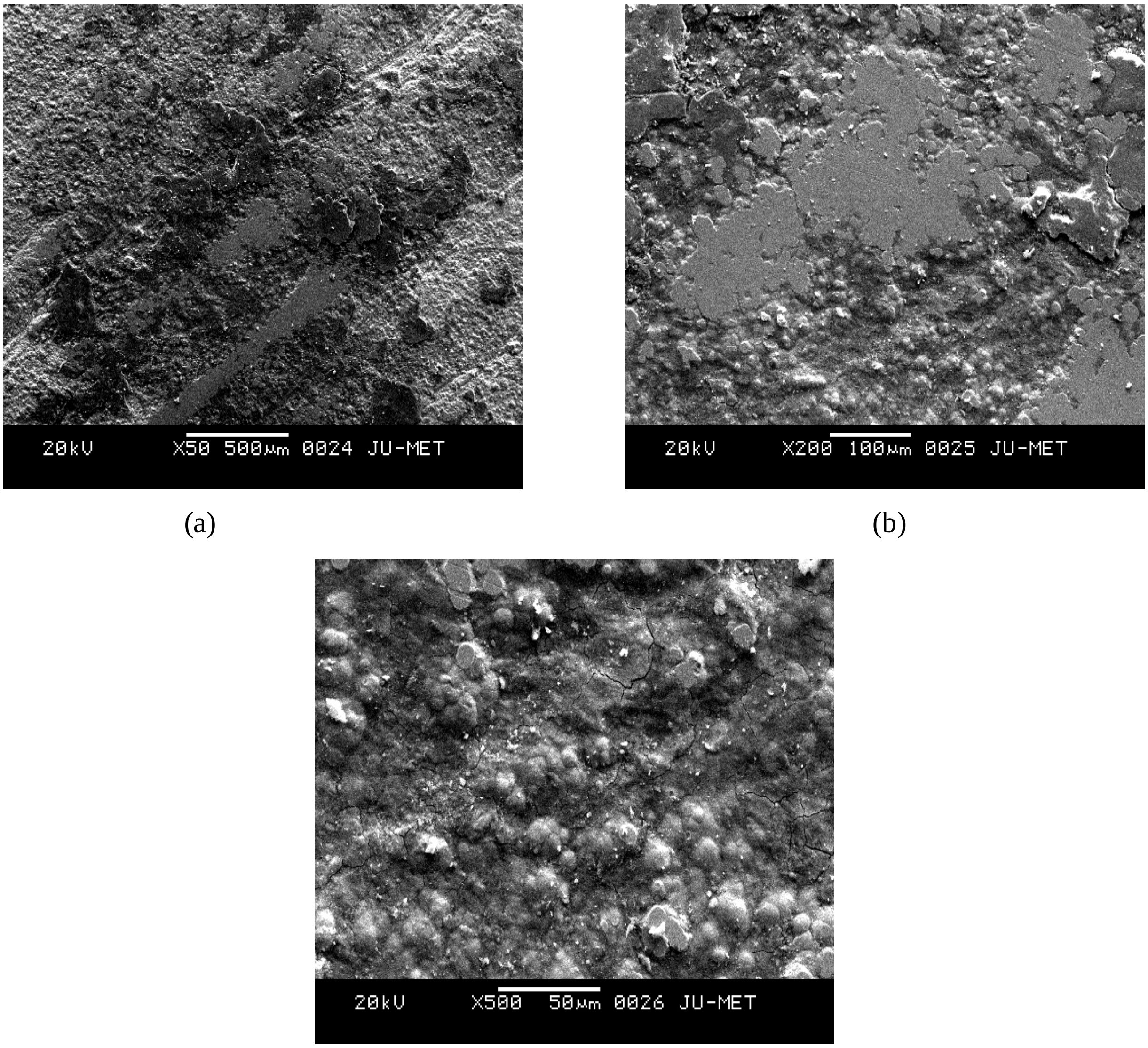


Fig. 4.4 SEM micrograph of cross cut Ni-P coating

The thickness of the deposits obtained is an important aspect since, if significant amount of thickness is not obtained, after testing the coating is worn out, the substrate of GFRE composite will undergo wear. The cross cut surface of the coated sample under SEM has been shown in

Fig.4.4. However, before observing under the microscope the cross cut surface is well ground to get a smooth section so that true representation of the coating thickness could be obtained. The thickness of the deposits is seen to vary between 25 to 30 µm which is higher than the maximum amount of wear depth for coated specimens as seen from Table 4.2. Thus there is no case where the whole thickness of coating wears out.

The surface morphology of the worn surface of Ni-P coated GRRE composite specimens after tribology test is carried out in scanning electron microscope.



(a) (b)

(c)

Fig. 4.5 SEM micrographs of coated GFRE composite after test at a 40N load and 80rpm rotating speed at magnification of (a) 50X (b) 200X (c) 500X

Several specimens are tested and Fig.4.5 shows the surface morphology at different magnifications for the condition of 40N load and 80rpm rotating speed. Fig.4.5 (a) shows the wear track. Fig.4.5 (b) shows the worn surface, from the SEM image it is clear that the load is taken by some of the peak globules where the other peaks remain undeformed. Fig.4.5 (c) shows the type of wear; from the SEM image it is clear that the wear mechanism is generally adhesive in nature and few abrasive particles are observed on the worn surface.

The composition of the coated GFRE composite is measured by energy dispersive X-ray analysis which is used in conjunction with the SEM instrument. The EDX spectrum of the coated specimen is shown in Fig.4.6. The peaks of the nickel and phosphorous is quite specific. The average weight percentages of nickel and phosphorous is found to be around 90.6% and 9.4% respectively.

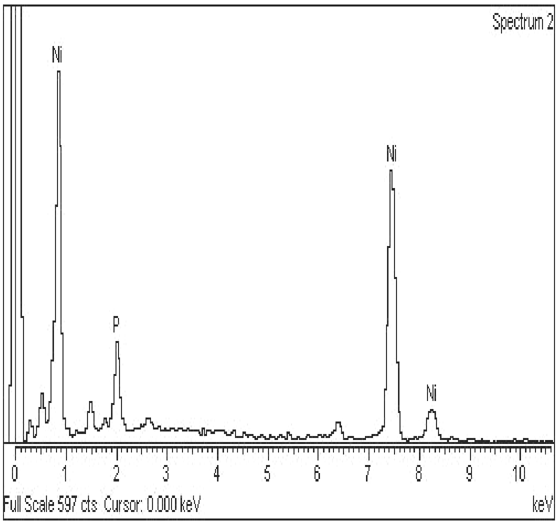


Fig.4.6 EDX pattern of Ni-P deposited sample

The phase structure analysis of the coating specimen is carried out in X-ray diffraction analysis. The presence of different crystalline phases induces certain physiological behaviour in a material. Hence, determination of the phase content is vital in understanding and explaining some specific performance of the material. The XRD plot for Ni-p coated GFRE composite has been shown in Fig.4.7. It is clear from the plot that there is no sharp peak at all and it has indicates an amorphous structure.

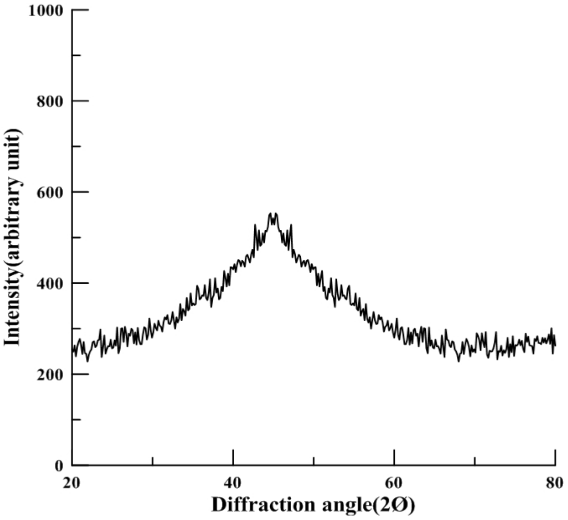


Fig.4.7 XRD plots of Ni-P coated GFRE composite

4.3 FRICTION BEHAVIOR

4.3.1 GFRE COMPOSITE

The friction behaviour of GFRE composite is studied under dry condition on pin on disc apparatus. The friction characteristics under different load for various sliding speeds of GFRE composite are presented in Fig.4.8. In general, friction coefficient decreases with increase in applied load, with few exceptions as it occurs at a load of 40N. In most of the cases, friction coefficient decreases with an increase in sliding speed for a particular load. With increase in the sliding velocity, temperature rise in the sliding interface occurs. The increase in temperature causes thermal penetration as a result of softening of matrix resin. Consequently coefficient of friction decreases with increase in sliding speed. The COF decreases with increase in load due to elastic deformation and adhesion of the surface asperities. It can also be observed that there is a rise in friction coefficient at an applied load of 40N for a sliding speed of 70 rpm. Multiple samples are tested at this condition and the same result is observed. From Fig.4.8, it is also seen that the coefficient of friction increases with increase speed up to 70 rpm after that decreases for a load of 20N. Same thing happens to other loads also. It occurs due to transfer film formation on the mating surface.

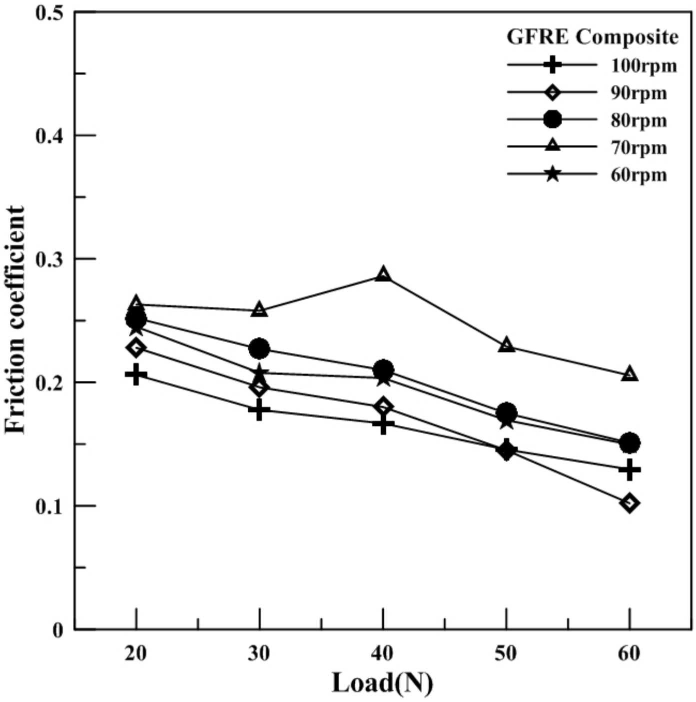


Fig.4.8 Variation of coefficient of friction with applied load for various sliding speed for GFRE composite.

4.3.2 COATED GFRE COMPOSITE

The coated GFRE composite is tested on pin on disc apparatus under the dry condition to evaluate the friction coefficient. Coefficient of friction of Ni-P coated GFRE composite as a function of applied load under different sliding velocities is shown in Fig.4.9. It is observed that coefficient of friction decreases with increase in applied load at all sliding velocities and then remained at nearly steady level at higher applied load. It has been seen that with increase in load, more wear takes place leading to surface smoothening. As a result, friction decreases with increase in load. Also the presence of large quantity of wear debris is believed to be responsible for decrease in friction with applied load. Moreover, friction coefficient decreases with the increase in sliding velocity for a particular load. Friction between two interacting surfaces depends on the instantaneous mechanical properties (changes in shear rate) of the mating materials at the interface [79]. The strength of the new layers of Ni-P coating coming in contact with the counter face material may be greater at higher strain rates which may be contributing to lower friction value. Also, it is found that the friction coefficient does not vary much with applied load for 100 rpm sliding speed.

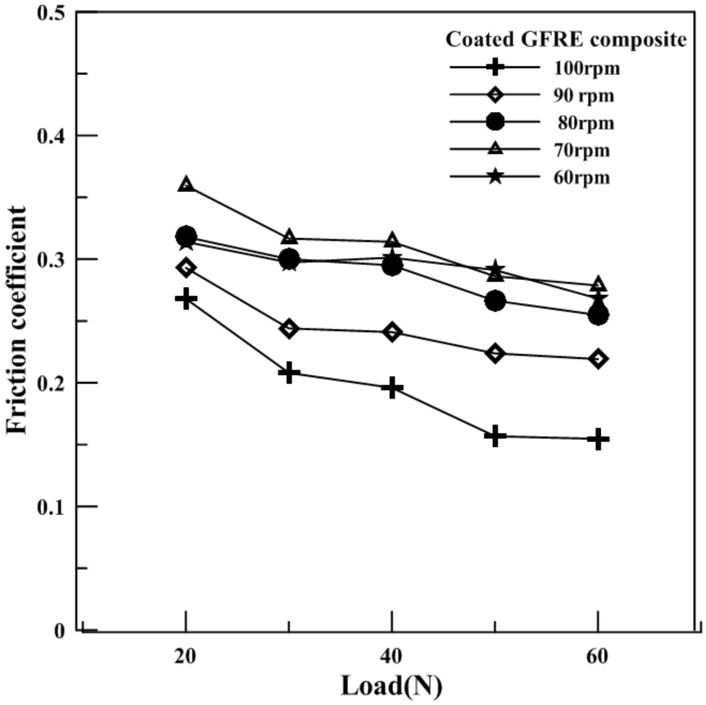


Fig.4.9 Variation of friction coefficient with applied load for various sliding speed for coated GFRE composite.

4.4 WEAR BEHAVIOR

4.4.1 GFRE COMPOSITE

The GFRE composite is tested on pin on disc apparatus under the dry condition to evaluate the wear. Wear of bare composite as a function of applied load under different sliding speeds is shown in Fig.4.10. It has been observed that wear increases with the increase in applied load. It is also observed that wear increases with increase of sliding speed for a particular applied load. Wear is found to increase marginally with increase in applied load for 60rpm disc speed. For most of the sliding speeds except 60rpm for all applied loads, an increasing wear trends are seen for increase in both load and speed. It is the combined effect of load and speed which leads to melting at the interface of composite resulting in increase wear [26].

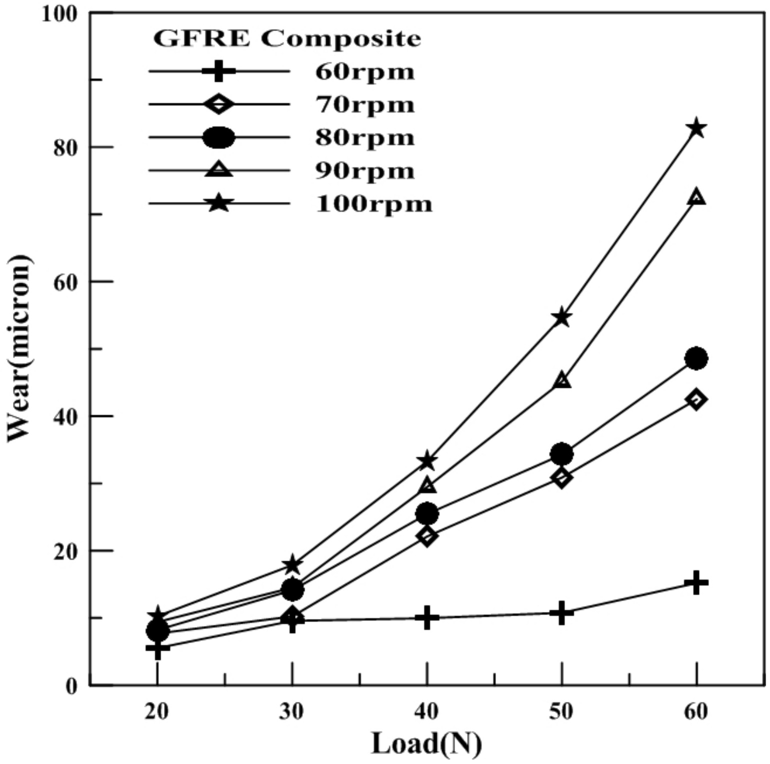


Fig.4.10 Variation of wear with applied load for various sliding speed for GFRE composite.

4.4.2 COATED GFRE COMPOSITE

The wear behaviour of coated GFRE composite is studied under dry condition on tribotester apparatus. Fig.4.11 shows the variation of wear of the coated specimens with applied load for various sliding velocities. It is observed that wear increases gradually with increase in applied load independent of sliding speeds. The wear loss is relatively low at smaller loads because of less penetration of the asperities and less number of peaks was in action with sliding surface. At higher load, surface asperities of the counter face material have higher penetration into the coating surface. This results in more plastic deformation of the coating surface. It is also observed that wear increases with increase in sliding velocity for a particular load. This may be due to the more sliding distance traveled for same test duration.

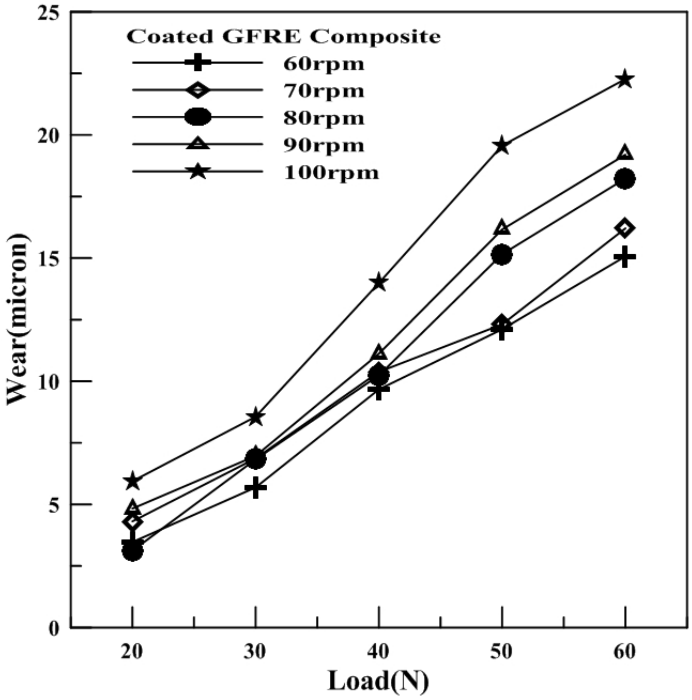


Fig.4.11 Variation of wear with applied load for various sliding speed for coated GFRE composite.

4.5 COMPARATIVE STUDY OF GFRE COMPOSITE AND COATED GFRE COMPOSITE

In this section, an attempt has been made to draw a comparison of the tribological behavior of GFRE composite and Ni-P coated GFRE composite. Fig. 4.12 compares the wear depth of the GFRE composites and coated GFRE composite while Fig. 4.13 compares the COF. It can be clearly seen that there has been a sharp reduction in wear but increase in COF in coated GFRE composite for all the 25 cases compared to the GFRE composite in the dry condition. But in some cases there has been an exception, where the wear under coated specimen is seen to be higher than the bare specimen. This comparison can be helpful in determining the suitability of using electroless Ni-P coating on GFRE composite for reduction of wear in dry condition.

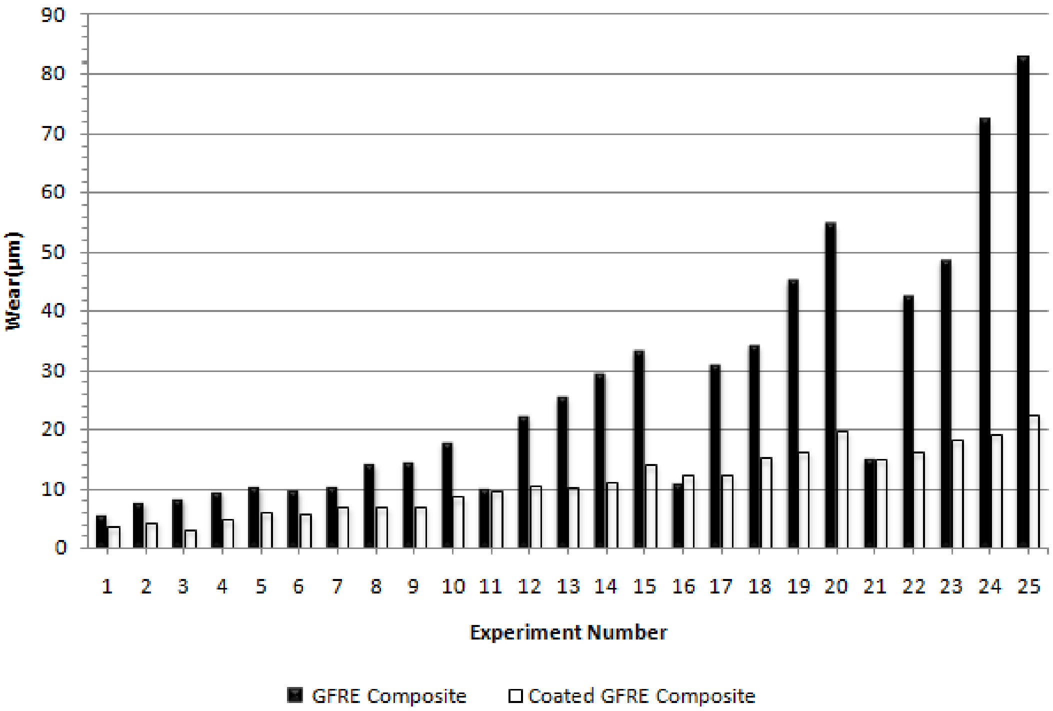


Fig. 4.12 Comparison of wear of coated and bare GFRE composite.

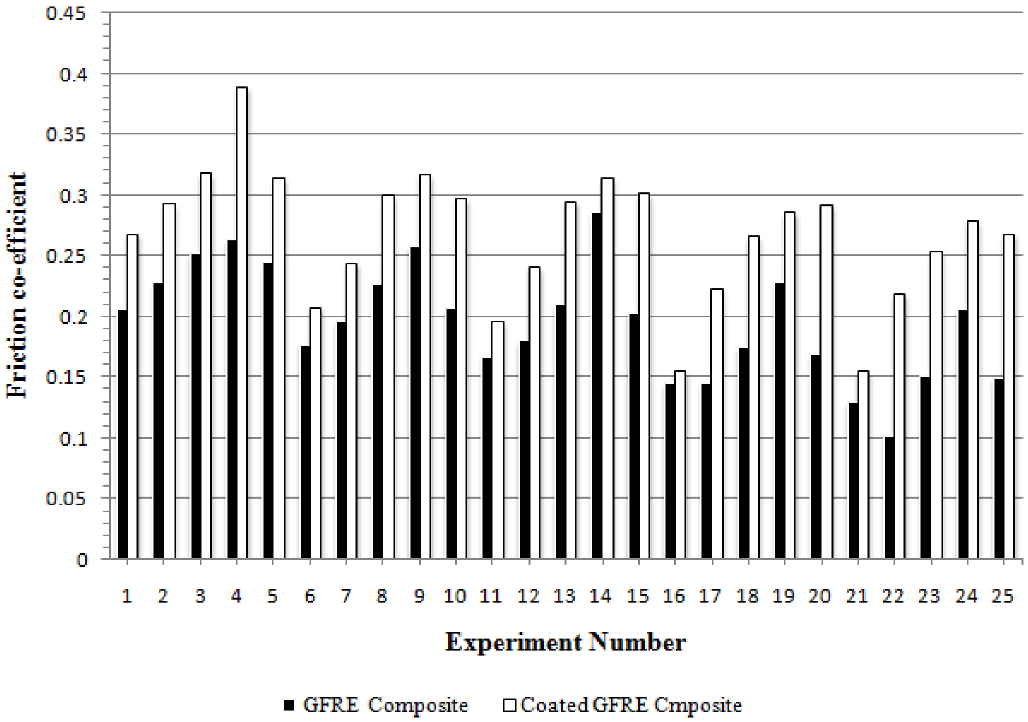


Fig. 4.13 Comparison of friction coefficient of coated and bare GFRE composite.

From Fig. 4.12, it can be seen that there has been a decreasing value of wear of coated GFRE composite. GFRE composite has higher wear loss due to abrasive particles, which are in action with the sliding surface and these particles create grooves, resulting more wear loss.

Fig. 4.13 shows the comparison of friction coefficient of bare and coated GFRE composite samples. In general, coated samples have higher coefficient of friction. Thus coated specimens offer good wear resistance but higher friction coefficients. Overall it can be concluded that coating on GFRE composite is advantageous for low wear requirement situations but at the cost of higher friction.

CHAPTER-5

CONCLUSIONS AND FUTURE SCOPE OF WORK

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This chapter summarizes the important conclusions that can be drawn from the present work and the future scope of the study is given.

5.1 CONCLUSIONS

In this study, the tribological behaviour of glass fiber reinforced epoxy composite and electroless Ni-P coated GFRE composite have been investigated on a pin on disc tribotester. The behaviour of the chosen process parameter on friction and wear were analyzed. Both the main effects as well as interaction effect of the parameters are examined. The following conclusions can be directly inferred from the study of GFRE composite and electroless Ni-P coated GFRE composite under dry condition.

- The wear depth of both coated and uncoated GFRP composite strongly depends on the experimental test parameters i.e. load, sliding velocity and time.
- Wear depth increases with the increase in load and speed. This can be attributed to the fact that on increasing the load, the number of asperities coming in contact and the area of contact of the individual asperities increases. The sliding speed increases the sliding distance and leading to higher values of wear.
- The variation of COF is somewhat different. COF decreases with the increase in load and speed, with few exceptions. In GFRE composite, this occurs because of thermal softening of resin and elastic deformation of surface asperities. And for coated GFRE composite, this occurs due to surface smoothening.
- The friction coefficient at a 70rpm sliding speed of GFRE composite is observed higher value due to formation of film transfer.

- In all the 25 experiments performed, the values of wear depth obtained under coated specimens are lesser than that obtained in bare specimens. This might have happened at bare specimen because of higher abrasive particles are in action with sliding surface. And the values of COF obtained under coated specimens are higher than that obtained in composite specimens. For composite specimens, it occurred due to elastic deformation.

- SEM micrographs have shown various wear mechanism of GFRE specimens which is a combination of adhesive and abrasive nature. These mechanisms are fiber breaking, fiber matrix debonding etc. Also in coated GFRE specimens, SEM exhibits many globular particles on the coated surface. The surface of the coating is optically smooth and of low porosity. No obvious surface damage is found. And also, it is concluded that wear mechanism is generally adhesive of coated GFRE specimens.

- EDX results confirm the presence of nickel and phosphorus in the deposits.

- XRD analysis of the Ni-P coating indicates that the coating is almost amorphous in structure.

5.2 FUTURE SCOPE OF WORK

The present study is an attempt to analyze the trends of wear depth and friction coefficient of woven E-glass fiber reinforced laminated composites and electroless Ni-P coating on GFRE composite with varying tribo-testing parameters under dry condition. There are still several other areas which can be explored for further research work.

- More number of test parameters can be included for the investigation of wear depth and coefficient of friction such as lubricating condition, the temperature at which the test is carried out. Temperature which seemed to playing an important role could be included in the process parameter to evaluate its effect more extensively.

- There are various optimization tools such as Taguchi, grey fuzzy logic etc. that can be used to optimize tribological parameters.

- The tribological behaviour of GFRE composite and electroless Ni-P coated GFRE composite in various corrosive environments and lubricated conditions can be studied.
- The tribological behavior of electroless Ni-P coated glass fiber reinforced polymer composites in dry condition; various lubricated conditions and corrosive environments can be investigated.

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