

Wear Testing of Hybrid Epoxy Nanocomposites

Dissertation submitted in partial fulfillment for the requirement of degree of

**Master of Engineering
in
CAD/CAM & Robotics**

By

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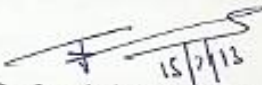
This is to certify that the thesis entitled "Wear Testing of Hybrid Epoxy Nanocomposites", is an authentic record of my own work carried out as requirements for the award of degree of Master of Engineering in CAD/CAM & Robotics from Thapar University, Patiala, under the guidance of Dr. Haripada Bhunia (Associate Professor, ChED) and Dr. Jaswinder Singh Saini (Assistant Professor, MED) during July 2012 to July 2013.

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ABSTRACT

Polymer composites containing different fillers and/or reinforcements are frequently used for applications like automotive parts, gear assemblies etc., in which friction and wear are critical issues. In particular, they are now being used as sliding elements, which were formerly composed of metallic materials only. The extremely large surface area is one of the most attractive characteristics of nano-particles because it facilitates creating a strong interaction between fillers. These also have been found to improve the wear properties. Various nano-particles have been incorporated in polymer matrices for this purpose and there have been positive results.

Epoxy and hybrid epoxy nano-composites are developed by in situ polymerisation. Specific wear rate of the nano-composites developed for wear applications have been presented in this work. The study of variation of different parameters like loading and velocity and their consequent effects on the specific wear rate is done. A number of tests are conducted on a pin-on-disc type machine. Different sliding velocities for conducting the wear tests are 105, 209 and 314 cm/s. Different normal loading conditions for consideration in the experiment are 20 N, 30 N and 40 N. It is evident from the tests that for every sample, the specific wear rate increases with the increase in the normal loading. However, in case of variation in velocity, it is initially higher at 105 cm/sec, significantly drops at 209 cm/sec and rises to the maximum value at 314 cm/sec. Firstly, variation of a single nanoparticle Cloisite 30B is done in order to find its optimum concentration. It is varied from 1% to 4% and the tests present that 2% Cloisite 30B exhibits the minimum specific wear rate. Then, addition of TiO_2 is done. It is varied from 2% to 6% and the tests show that of all the samples the hybrid sample of 2% Cloisite 30-B and 2% TiO_2 shows the least specific wear rate. This reduction in specific wear rate is quite significant when compared with specific wear rate of the neat sample. This indicates that hybridisation of organo-modified nano-clay (Cloisite 30B) and TiO_2 is a justified combination for enhancing the wear properties of epoxy hybrid nano-composites.

CONTENTS

	Title	Page No.
	Certificate	ii
	Acknowledgements	iii
	Abstract	iv
	Contents	v
	List of Figures	vi
	List of Tables	vii
Chapter 1	Introduction	1
	1.1 Composites	1
	1.2 Composition of composites	2
	1.3 Matrix	2
	1.3.1 Polymer matrix composites	3
	1.4 Reinforcements	6
	1.5 Nanocomposites	7
	1.5.1 Polymer nanocomposites	9
Chapter 2	Literature review	10
	2.1 Literature review	10
	2.2 Summary of literature review	18
	2.3 Problem formulation	20
Chapter 3	Experimentation	21
	3.1 Materials	21
	3.2 Preparation of composites	21
	3.2.1 Processes	22
	3.2.2 Overview of the process for sample preparation	24
	3.3 Testing	25
Chapter 4	Results and discussion	28
	4.1 Results and discussion	28
Chapter 5	Conclusion and future scope	35
	5.1 Conclusion	35
	5.2 Future scope	35
	References	36
	Publications	38

LIST OF FIGURES

Fig. No.	Name of the figure	Page no.
Fig. 1.1	Type of thermosets	4
Fig. 1.2	Different types of reinforcements	6
Fig. 3.1	a) Magnetic stirrer with hot plate	23
	b) Mechanical stirrer of remi motors	23
Fig. 3.2	Water bath sonicator machine	23
Fig. 3.3	Teflon parting mould	23
Fig. 3.4	a) Pin on disk machine used for the experiment	26
	b) Control unit of the machine	26
	c) Specimen holder	26
	d) Hanging weights used for applying normal load	26
Fig. 3.5	a) Specimens of Coisite 30B (0%-4%)	27
	b) Specimens of hybrid Cloisite 30B(2%) and TiO ₂ (2%,4% and 6%)	27
Fig. 3.6	CAS weighing machine	27
Fig. 4.1	Specific wear rate of Cloisite 30B (0%-4%) at 3 kg loading	32
Fig. 4.2	Specific wear rate of Cloisite 30B (0%-4%) at 4 kg loading	32
Fig. 4.3	Specific wear rate of Cloisite 30B (0%-4%) at 5 kg loading	33
Fig. 4.4	Specific Wear Rate at 3 kg loading of NC and TiO ₂	33
Fig. 4.5	Specific Wear Rate at 4 kg loading of NC and TiO ₂	34
Fig. 4.6	Specific Wear Rate at 5 kg loading of NC and TiO ₂	34

LIST OF TABLES

Table no.	Name of the table	Page no.
Table 1.1	Thermosets and thermoplastics	5
Table 2.1	Summary of the literature review	18
Table 3.1	Specifications of the pin-on-disk machine	26
Table 4.1	Wear results for neat epoxy	28
Table 4.2	Wear results for 1% Cloisite 30B	28
Table 4.3	Wear results for 2% Cloisite 30B	29
Table 4.4	Wear results for 3% Cloisite 30B	29
Table 4.5	Wear results for 4% Cloisite 30B	30
Table 4.6	Wear results for 2% Cloisite 30B + 2% TiO ₂	30
Table 4.7	Wear results for 2% Cloisite 30B + 4% TiO ₂	31
Table 4.8	Wear results for 2% Cloisite 30B + 6% TiO ₂	31

CHAPTER 1

INTRODUCTION

1.1 COMPOSITES

From a long time mankind has been using composites even before its modern day definition came into existence. One of the earliest known man made composites material that was used 6000 years ago was wattle and daub which is a combination of the woven thin branches within the mixture consisting a combination of wet soil, clay, sand, animal dung and straw. From the ancient mud houses that used straw and mud composite for making bricks that were used for constructional purposes to the concrete that is found in almost all the buildings in this modern world mankind has come a long way in formulation of composites as well as better understanding of the various properties that can be harnessed and produced that otherwise were not possible to be obtained from the conventional metals and other materials. Concrete is one of the most famous and widely used composite materials. According to a survey in the year 2006 approximately 7.5 billion cubic meters of concrete was made each year by that time which is more than one cubic meter for every person on Earth. The daily lives get encountered with so many composites material goods without even realizing that without them it would have been quite difficult to do certain works with such an ease. Let's take for an instance the disk brake pads. They have a soft metal matrix in which there are hard ceramics particles embedded. There are shower stalls and even the bath tubs are made of composite materials (made of fibre glass). Not only in or general lives but taking the account of extensive usage of composites in the aerospace applications anyone would be astonished with the jaw dropping performance of these and the weight saving advantages along with increase in endurance, flame retardancy, better overall mechanical properties and much more. These qualities are just a beginning of a long list.

Arriving at a particular definition of the composite materials is very difficult. But in general, composite materials as those materials that are engineered or naturally occurring that are made from two or more constituent materials that have significantly different physical and/or chemical properties that remain separate and distinct within the finished product. Although there may be a significant influence of each and every constituent material in the properties of the final composite material synthesized and for this purpose and proper determination of the final product properties there has been and is an enormous amount of research going on.

1.2 COMPOSITION OF COMPOSITES

Composites consist of different individual materials that can be referred to as constituent materials. They can broadly be classified into two main categories of constituent:

- a) The Matrix.
- b) The Reinforcement.

Any one of these can be more than one in number but at least one portion of each type is required in every composite material (for it to be a composite material). The matrix occupies most of the volume in the composite materials which is much more than the reinforcement. The main purpose of the matrix material is that it surrounds and supports the reinforcement materials by maintaining their relative positions in the matrix. Whereas the foremost purpose of the reinforcements is to impart their physical and special mechanical properties to enhance the matrix properties and this further strengthens the resulting composite materials. A synergism of these two produces material properties that otherwise are unavailable from the individual constituent materials. The wide variety of matrix and strengthening materials available and feasible in combination allows the designer of the product or structure to choose an optimum combination among them.

Manmade or engineered composite materials must be formed to shape. For this purpose the matrix material can be introduced to the reinforcement before or after the reinforcement material is placed into the mould cavity or onto the mould surface. The part is set after an event known as the melding event experienced by the matrix. This event can happen in many ways including polymerization or simple solidification from a melted state depending upon the type of matrix material selected and its nature and properties.

Depending upon the design requirements of the finished product different moulding methods can be used. The key factors influencing the methodology of the method chosen are dependent on the natures of the matrix chosen and reinforcement materials selected. The following section describes in detail about each of the constituents.

1.3 MATRIX

The matrix is the primary component of the composites. It is the constituent that serves the two most important functions of

- a) Binding the reinforcement phases in place.
- b) Deforming to distribute the stresses among the constituent reinforcement materials under an applied force.

Though there are many other demands that are to be met by the matrix apart from these like protecting the environmental degradation of the reinforcing fibres, carrying the inter-laminar shears, enhancement of transverse properties of the laminates, improvement of fracture resistance and impact resistance and proper distribution of the loads on all the fibres. They may need to withstand temperature variations i.e. have less Coefficient of Thermal Expansion (CTE), be conductors or resistors of electricity, have moisture sensitivity etc. They are also desired to offer weight advantages, ease of handling and other merits. These functions among various others are kept in mind while selecting the appropriate matrix material for numerous different purposes.

Apart from the above mentioned functions there are certain properties expected from the various matrices chosen. The properties like being chemically inert, dimensionally stable at ambient and higher temperatures, having less absorption affinity for moisture, lower coefficient of thermal expansion, having low shrinkage on dropping in the operating temperatures, having good elastic properties for the efficient transferring of load to the fibres, having good flow characteristics for easy penetration of fibre bundles completely and elimination of the any voids formed during the curing process, easily process able into the final shape and having a reasonable strength, modulus and elongation. The elongation should be more than that of the fibres reinforced.

Different types of materials that can be used as matrices are given below.

1.3.1 POLYMER MATRIX COMPOSITES

A polymer is a long chain molecule containing one or more repeating units of atoms joined together by strong covalent bonds. A polymeric material is a collection of a large number of polymer molecules of similar chemical structure (but not of equal length). In the solid state, these molecules are frozen in space, either in a random fashion (amorphous polymers) or in a mixture of random and orderly (folded) fashion (for semi crystalline polymers). However, on a sub microscopic scale various segments in a polymer molecule may be in a state of random excitation. The frequency, intensity, and number of these segmental motions increase with increasing temperature, giving rise to the temperature dependent properties of a polymeric solid. Polymers are desirable matrix material as they can be easily processed; are lightweight, have good adhesion with reinforcement and good mechanical properties. The polymer matrices are very versatile that has led to their widespread use in various applications. They can be further divided into Thermosets and Thermoplastics.

THERMOSETS

Thermoset materials are generally liquid or malleable prior to curing, and designed to be moulded into their final form. They have the property of undergoing a chemical reaction by the action of heat,

catalyst, UV light, etc., to become a relatively insoluble and infusible substance. They develop a well bonded 3D structure upon curing. Once hardened or cross linked, they decompose rather than melt. They are generally stronger than thermoplastics due to their 3D network of bonds and are also better suited to high temperature applications up to the decomposition temperature of the material. They can be retained in a partially cured condition over a prolonged period of time, making thermosets very flexible. They are most suited as matrix bases for advanced conditions fibre reinforced composites. They are used with fibre reinforcements in aerospace components, automobile parts, defence systems etc. The further classification of thermosets is given in Fig. 1.1.

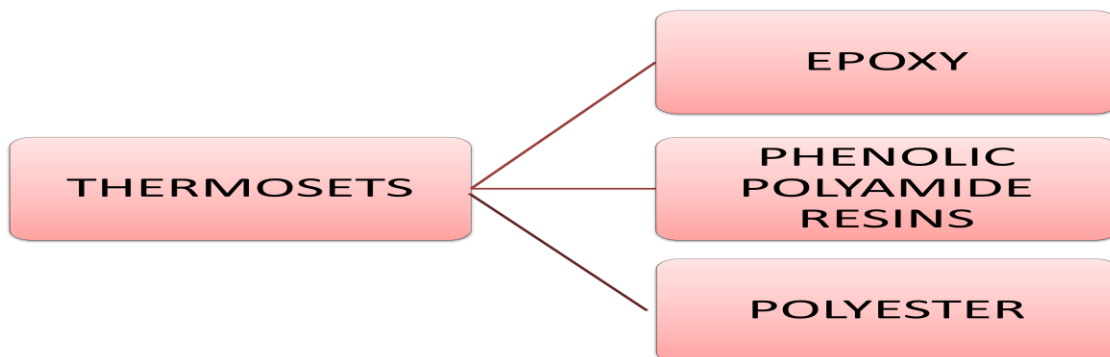


Fig. 1.1 Types of Thermosets

EPOXY AND POLYESTER

Epoxy and polyester react to form a network structure without formation of a volatile by-product. Polyester matrices have been in use for the longest period in the widest range of structures. Polyesters cure with the addition of a catalyst (usually a peroxide) resulting in an exothermic reaction, which can be initiated at room temperature. The most widely used matrices for advanced composites have been the epoxy resins. These resins cost more than polyesters and do not have the high temperature capability of the Bismaleimides or Polyimides. Epoxies are widely used due to their number of advantages like good adhesion with fibres and resin, resistance to creep and fatigue, wide range of curative options, good handling characteristics, low shrinkage, excellent adhesive properties, flame resistant, good chemical resistance, good mechanical properties including toughness, no by-products formation during cure, Offering considerable variety for formulating prepreg resins and curing temperature of 120-175°C (250-350°F) which is very less. Despite these numerous advantages they do have certain disadvantages like their resins and curatives are somewhat toxic in uncured form, usage

limited to about 200°C (392°F), have a high thermal coefficient of expansion, high degree of smoke liberation in a fire, may be sensitive to ultraviolet light degradation and curing is slow.

PHENOLS

Phenols react to form a volatile by product i.e., water. Some properties of phenols are low cost, capability to be B-Staged, excellent high temperature resistance up to 205-260°C (400-500°F), good mechanical strength, dimensional and thermal stability, good laminate properties, hot molding (cold molding very rarely), good fire resistance and very low smoke emission, curing temperature is 175°C (350°F), high chemical resistance, good dielectric properties.

Some of the disadvantages are: by-products are produced during curing, there is high shrinkage on cure, and phenolic laminates are porous.

THERMOPLASTICS

A thermoplastic, also known as a thermo softeners plastic, is a polymer that becomes pliable or mouldable above a specific temperature, and returns to a solid state upon cooling. Most thermoplastics have a high molecular weight, whose chains associate through intermolecular forces; this property allows thermoplastics to be remoulded because the intermolecular interactions spontaneously reform upon cooling. In this way, thermoplastics differ from thermosetting polymers, which form irreversible chemical bonds during the curing process; thermoset bonds break down upon melting and do not reform upon cooling is also the main reason for their widespread use. Following is a table illustrating the difference between thermosets and thermoplastics.

Table 1.1 Thermosets and Thermoplastics

Thermosets	Thermoplastics
Resin cost is low.	Resin cost is slightly higher.
Thermosets exhibit moderate shrinkage.	Shrinkage of thermoplastics is low.
Interlaminar fracture toughness is low.	Interlaminar fracture toughness is high.
Thermosets exhibit good resistance to fluids and solvents.	Thermoplastics exhibit poor resistance to fluids and solvents.
Composite mechanical properties are good.	Composite mechanical properties are good.
Prepregability characteristics are excellent.	Prepregability characteristics are poor.
Prepreg shelf life and out time are poor.	Prepreg shelf life and out time are excellent.

1.4 REINFORCEMENTS

Reinforcement usually adds rigidity and greatly impedes crack propagation. Thin fibers can have very high strength, provided they are mechanically well attached to the matrix. Reinforcements for the composites can be fibers, fabrics particles or whiskers. Fibers are essentially characterized by one very long axis with other two axes either often circular or near circular. Particles have no preferred orientation and so does their shape. Whiskers have a preferred shape but are small both in diameter and length as compared to fibers. Different types of reinforcements that can be used are illustrated in figure 1.2.

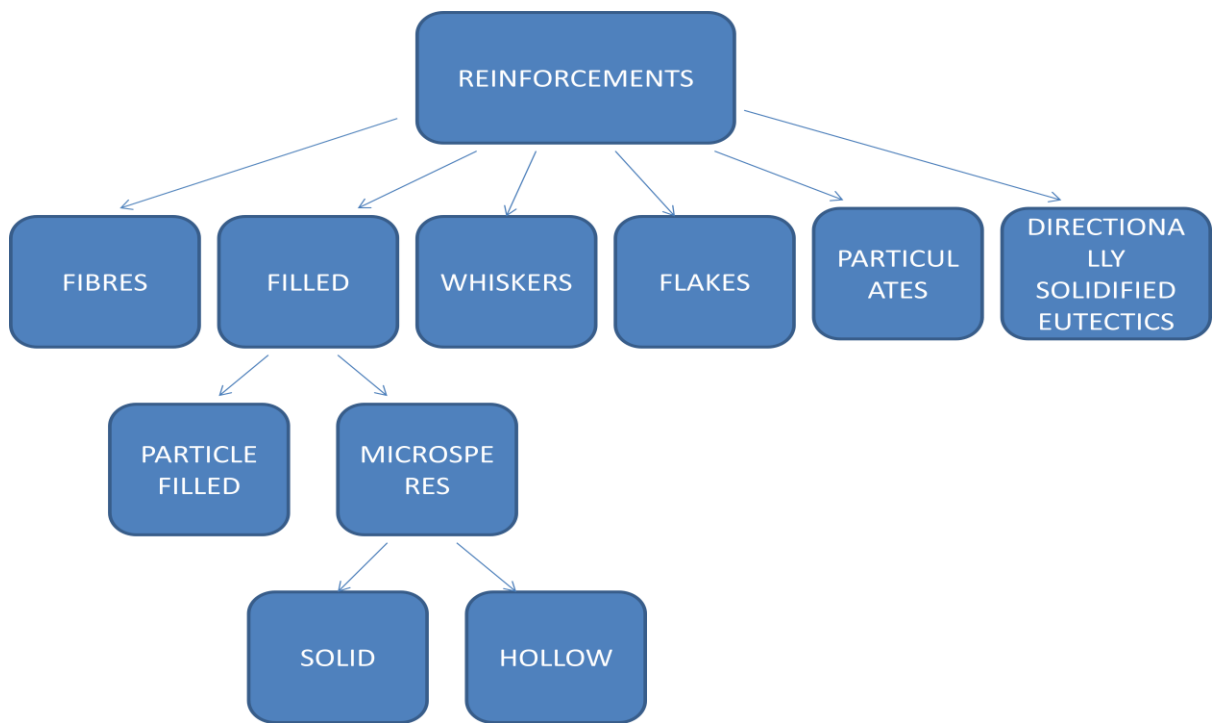


Figure 1.2 Different types of reinforcements

FIBER REINFORCEMENT

Fibers are the principal constituent in a fiber-reinforced composite material. They occupy the largest volume fraction in a composite and share the major portion of the load acting on a composite structure. Proper selection of the type, amount, and orientation of fibers is very important, since it influences the different characteristics of a composite laminates i.e. specific gravity, tensile strength and modulus, compressive strength and modulus, fatigue strength as well as fatigue failure mechanisms, electrical and thermal conductivities, and cost.

Glass fibers are the earliest known fibers used to reinforce materials. Ceramic and metal fibers were subsequently found out and put to extensive use, to render composites stiffer more resistant to heat.

CARBON FIBRES

Carbon fibre, not surprisingly, is made of carbon crystals aligned in the long axis. These honeycomb shaped crystals organize themselves in long flattened ribbons. This crystal alignment makes the fiber strong. These ribbons are formed within long fibers. The fiber shape is the original shape of the material used to produce the Carbon Fiber. These fibers in turn are bundled in large numbers and are woven, made into felt, twisted or as bundled without twisting. This is referred to as Roving. Carbon fiber is also offered as chopped strands and powder. While use of the term carbon for graphite is permissible, there is one basic difference between the two. Element analysis of poly-acrylo-nitrile (PAN) base carbon fibers show that they consist of 91 to 94% carbon. But graphite fibers are over 99% carbon. The difference arises from the fact that the fibers are made at different temperatures. PAN-based carbon cloth or fiber is produced at about 1320 °C, while graphite fibers and cloth are graphitised at 1950 to 3000 °C. The properties of graphite remain unchanged even at high temperatures, but its willingness to react readily with most metals at the fabrication stage or during use at very high temperatures is often a stumbling block, as seen in aluminium matrices when carbides are produced at the interface. These carbides react with moisture with disastrous effects on the composite material. Graphite fibers are some of the stiffer fibers known. The stiffness of the fiber is as high as the graphite content. But a major drawback is that stiffness and strength are inversely proportional to each other. Forbidding costs make the use of graphite fibers prohibitive. The best glass fibers are far less expensive than the cheapest, lowest quality of graphite. Some of the properties of Carbon Fiber include high strength to weight ratio, good rigidity, corrosion resistance, electrical conductivity, fatigue resistance, good tensile strength but Brittleness, fire resistance, high thermal conductivity in some forms, low coefficient of thermal expansion, non-poisonous, biologically inert, X-ray permeability, self-lubrication, excellent EMI (Electromagnetic Interference) shielding property, relatively expensive, requirement of specialized experience and equipment to use.

1.5 NANOCOMPOSITES

One of the hottest topics of research in today's era is 'The Nanocomposites'. As such there is no fixed definition, Nanocomposites can be classified on the basis of different matrix materials. Other variations between nanocomposites can be that of the reinforcing fibres. Then there are different nano particles that are used as the filler particles. These particles have very high surface area of contact with the matrix and thereby impart their properties to the composite material. They also have high aspect ratios. A nanocomposite is a multiphase solid material where one of the phases has one, two or three

dimensions of less than 100 nanometers (nm), or structures having nano-scale repeat distances between the different phases that make up the material (Ajayan *et al.*,2003). In the broadest sense this definition can include porous media, colloids, gels and copolymers, but is more usually taken to mean the solid combination of a bulk matrix and nano-dimensional phase(s) differing in properties due to dissimilarities in structure and chemistry. The mechanical, electrical, thermal, optical, electrochemical, catalytic properties of the nanocomposite will differ markedly from that of the component materials. Size limits for these effects have been proposed by some, <20 nm for making a hard magnetic material soft, <50 nm for refractive index changes, and <100 nm for achieving super paramagnetism, mechanical strengthening or restricting matrix dislocation movement (Kamigaito, 1994). Nanocomposites are also found in nature, for example in the structure of the abalone shell and bone. The use of nanoparticle rich materials long predates the understanding of the physical and chemical nature of these materials. Jose Yacaman *et al.* (1996) investigated the origin of the depth of colour and the resistance to acids and bio-corrosion of Maya blue paint, attributing it to a nanoparticle mechanism. From the mid-1950s nanoscale organo-clays have been used to control flow of polymer solutions (e.g. as paint viscosifiers) or the constitution of gels (e.g. as a thickening substance in cosmetics, keeping the preparations in homogeneous form). In mechanical terms, nanocomposites differ from conventional composite materials due to the exceptionally high surface to volume ratio of the reinforcing phase and/or its exceptionally high aspect ratio. The reinforcing material can be made up of particles (e.g. minerals), sheets (e.g. exfoliated clay stacks) or fibres (e.g. carbon nanotubes or electrospun fibres). The area of the interface between the matrix and reinforcement phase(s) is typically an order of magnitude greater than for conventional composite materials. The matrix material properties are significantly affected in the vicinity of the reinforcement. Ajayan *et al.*(2003) noted that with polymer nanocomposites, properties related to local chemistry, degree of thermoset cure, polymer chain mobility, polymer chain conformation, degree of polymer chain ordering or crystallinity can all vary significantly and continuously from the interface with the reinforcement into the bulk of the matrix. This large amount of reinforcement surface area means that a relatively small amount of nano scale reinforcement can have an observable effect on the macro scale properties of the composite. For example, adding carbon nanotubes improves the electrical and thermal conductivity. Other kinds of nanoparticulates may result in enhanced optical properties, dielectric properties, heat resistance or mechanical properties such as stiffness, strength and resistance to wear and damage. In general, the nano reinforcement is dispersed into the matrix during processing. The percentage by weight (called *mass fraction*) of the nanoparticulates introduced can remain very low (on the order of 0.5% to 5%) due to the low filler percolation threshold, especially for the most commonly used non-spherical,

high aspect ratio fillers (e.g. nanometre-thin platelets, such as clays, or nanometer-diameter cylinders, such as carbon nanotubes). They can be classified into three broad categories based on the type of matrix material:-

1. CERAMIC NANOCOMPOSITES
2. METAL NANOCOMPOSITES
3. POLYMER NANOCOMPOSITES

1.5.1 POLYMER NANOCOMPOSITES

In the simplest case, appropriately adding nanoparticulates to a polymer matrix can enhance its performance, often in very dramatic degree, by simply capitalizing on the nature and properties of the nanoscale filler (these materials are better described by the term nanofilled polymer composites) (Manias *et al.*, 2007). This strategy is particularly effective in yielding high performance composites, when good dispersion of the filler is achieved and the properties of the nanoscale filler are substantially different or better than those of the matrix, for example, reinforcing a polymer matrix by much stiffer nanoparticles (Usuki *et al.*, 1993) of ceramics, clays, or carbon nanotubes. It should be noted that the improvement in mechanical properties may not be limited to stiffness or strength. Alternatively, the enhanced properties of high performance nanocomposites may be mainly due to the high aspect ratio and/or the high surface area of the fillers (Usuki *et al.*, 1993), since nanoparticulates have extremely high surface area to volume ratios when good dispersion is achieved. Nanoscale dispersion of filler or controlled nanostructures in the composite can introduce new physical properties and novel behaviours that are absent in the unfilled matrices, effectively changing the nature of the original matrix (Manias *et al.*, 1997) (such composite materials can be better described by the term genuine nanocomposites or hybrids (Manias *et al.*, 1997)). Some examples of such new properties are fire resistance or flame retardancy (A. B. Morgan, 2007) and accelerated biodegradability.

The present work focusses on the polymer matrix nanocomposites with a combination of different filler materials (TiO₂ and nanoclay) and their sliding properties. Different nano particles can enhance properties up to a different extent. The nano particles when embedded in the matrix can change the properties, so a proper research and selection should be done before choosing them. The properties enhancement due to nano particles can be attributed due to the large surface area interaction of theirs with the matrix material. This leads to better transferring of properties and thereby causing a significant rise (although not always).

CHAPTER 2

LITERATURE REVIEW

2.1 LITERATURE REVIEW

Polymer composites having various nano sized fillers have been frequently used in numerous applications where friction and wear are dire issues like in automotive parts, gear assemblies etc. Polymer composites are mainly used because of the weight saving advantage. In particular, they are now being used as sliding elements, which were formerly composed of metallic materials only. From a notable time period there has been a considerable amount of research work done and still a lot is going on in the determination of wear and tribological properties of polymer nanocomposites. This includes the determination of wear rates, the effect on the wear properties, the change in coefficient of friction on addition of different nanoparticles or even change in their amount by % volume or % weight of the total, the inspection of worn surface, the analysis of transfer films formed on the counterface, just to name a few among them. The importance of the topic also increases because wear losses are one of the major concerns in various operations or jobs in which nanocomposites are used and could be used in future. There has also been a significant rise of interest in the research fraternity in the hybrid nanocomposites that use a combination of different types of nano particles and/ reinforcement(s). Following is a brief outlook on some of the research work done related to the wear testing of composites having polymer (thermoset) as matrix with filler materials (mostly nano-sized) :-

L. Chang *et al.* (2005) investigated the tribological performances of epoxy-based composites, filled with short carbon fibre, graphite, PTFE and nano-TiO₂ in different proportions and combinations. The patterns of frictional coefficient, wear resistance and contact temperature were examined by a pin-on-disc apparatus in a dry sliding condition under different contact pressures and sliding velocities. The experimental results indicated that the addition of nano-TiO₂ apparently reduced the frictional coefficient, and consequently reduced the contact temperature of fibre-reinforced epoxy composites. This led to the wear resistance of the composites being significantly enhanced, especially at extreme wear conditions, i.e. high contact pressures and sliding velocities. Compared to traditional fillers, epoxy nanocomposites attained improved both wear resistance and load-carrying capacity. Wear results of pin-on-disc were well comparable to that of block-on-ring under a standard sliding condition, i.e. 1MPa and 1 m/s. The lower wear rates resulted from the Block-On-Ring indicated to the good mechanical load-carrying capacity of nanocomposites. Owing to a nano-scale rolling effect of TiO₂ particles on short carbon fibres, the frictional coefficient of epoxy nanocomposites was reduced and the contact

temperature was consequently decreased under all test conditions. A good coincidence between the variations of frictional energy and contact temperature during sliding was observed. Wear resistance was improved by incorporating nano-TiO₂ (300 nm) into epoxy composites, especially at high contact pressure and high sliding speed. A reduction of the basic wear factor and an enhancement of the load-carrying capacity were achieved, which promote this materials for the applications under more severe wear conditions.

L. Chang *et al.* (2006) studied the wear performances of a series of epoxy based nanocomposites by a pin-on-disk apparatus under different sliding conditions. The addition of spherical TiO₂ nanoparticles (300 nm in diameter) was found to be able to apparently reduce the frictional coefficient and consequently to decrease the contact temperature and wear rate of fibre reinforced epoxy composites. The research work intended to understand the wear mechanisms involved in micro- and nanoscales. Based on a Scanning Electron Microscopy (SEM) and an atomic force (AFM) microscopy observations of the worn surfaces a positive rolling effect of the nanoparticles between the material pairs was proposed which led to the remarkable reduction of the frictional coefficient. This rolling effect protected the short carbon fibres from more severe wear mechanisms, especially at high sliding pressure and speed situations. In order to validate the assumption proposed, the influence of the counterpart roughness on the wear performance were carried out as well. The wear performances of carbon fibre reinforced composites were strongly related to the procedure of the fibre peeling off. The enhancement on the wear resistance of epoxy nanocomposites was generally associated with less fibre removal. Nanoscale grooves were observed on the SCF surfaces, which were probably scratched by the nanoparticles embedded in the counterpart surfaces. The behaviour of these grooves was found to be strongly influenced by the sliding conditions. Based on SEM and AFM observations, a positive rolling effect of the nanoparticles between the material pairs was proposed. This rolling effect helps to reduce the frictional coefficient during sliding, accordingly to reduce the shear stress and contact temperature. It was found that the addition of nanoparticles could not reduce the frictional coefficient when the nanocomposites slide against a very smooth disk.

Guijun Xian *et al.* (2006) evaluated the tribological properties of an epoxy-based nanocomposite containing graphite powder (7 vol %) and nano-scale TiO₂ (4 vol %). A series of composites containing additional fillers such as short carbon fibers (SCF), Aramid and polytetrafluoroethylene (PTFE) particles was developed and evaluated in adhesive and low amplitude oscillating wear modes. The incorporation of SCF and Aramid particles resulted in a remarkable improvement in the sliding wear resistance. SCF impaired the low amplitude oscillating wear resistance. The further addition of PTFE to the SCF filled nanocomposites reduced the friction and wear under both wear conditions. An adverse

effect of PTFE was found for the Aramid particles filled nanocomposites. Under sliding conditions, the lowest wear rate and coefficient of friction showed the 2–4 vol% PTFE filled SCF nanocomposite. Aramid particles containing nanocomposites (without PTFE) exhibited the best wear and friction behaviour under low amplitude oscillating wear conditions among the selected composites. The wear mechanisms were studied by scanning electron microscopy. Inclusion of Aramid in the nanocomposite containing TiO₂ and graphite deteriorated both impact and flexural strength, while inclusion of SCF was slightly beneficial to the flexural strength though damaging the impact strength. Addition of PTFE deteriorated both these properties. Under adhesive wear conditions, addition of Aramid particles to the epoxy nanocomposite significantly improved the wear resistance. The further addition of PTFE adversely was significantly detrimental to both wear and coefficient of friction. Addition of SCF also proved detrimental to both wear and coefficient of friction. However, SCF in combination with PTFE proved very good for both wear and coefficient of friction. Composition of SCF and PTFE in the range of 2–4% proved optimal for tribo-performance of the composites under sliding. Under low amplitude oscillating wear conditions, Aramid particles did not show potential and SCF showed detrimental influence on the wear behavior. The combination of SCF and PTFE (4%) proved very effective in reducing the wear rate.

Hui Zhang *et al.* (2007) investigated the influence of fibre length on tribological properties of short carbon fibre (SCF) reinforced epoxy composites. Both a block-on-ring and a pin-on-disk apparatus were applied for the study of sliding performance of composite specimens against polished steel counterparts under dry conditions. It was found that composites with longer SCF (nominal length = 400 µm) exhibited better wear resistance than those with shorter SCF (nominal length = 90 µm), in both cases either with or without graphite flakes and TiO₂ nanoparticles. This effect seemed to be more pronounced at higher contact pressures applied. The steady frictional coefficient and contact temperature were reduced slightly by longer fibres. The relationships among the frictional coefficient, the contact temperature and the wear rate were discussed under the support of scanning electron microscope observations of the worn surfaces. In terms of SEM observations of worn surfaces, the longer SCFs were more difficult to be peeled-off from the matrix, even at higher contact pressure. The worn surface became relatively smooth. This effect further caused a slight decrease in the frictional coefficient and contact temperature. All these factors in turn contributed positively to the wear resistance of the composites. In addition, the enhanced load-carrying capacity of the longer SCF reinforced composites could be favourable to the improved wear resistance, at higher contact pressures.

Jin Chein Lin (2007) carried out extrusion compression and dry sliding on the various nanoparticle filled composites by using cylindrical specimens. For studying the effect of exfoliated nanoparticles on

the epoxy matrix to friction and wear, Na-montmorillonite and titanium dioxide nanoparticles were prepared with the filler content varied from 0 to 10 vol. %. Compression tests were conducted by using cylindrical blocks to obtain the mechanical properties of the nanocomposites. For the determination of the tribological property, the sliding wear tests with high pressure were performed at room temperatures by using a block-on-disc apparatus. The morphologies of the wear trace of the as-spun material were obtained by using X-ray diffractometer (XRD) and scanning electron microscopy (SEM). Experimental results showed that the compression strength, fracture strength and Young's modulus for both reinforced nanocomposites were much higher than that of pure epoxy matrix. The friction coefficient and wear coefficient of Cloisite_30B nanocomposites were effectively reduced with rise in filler content which was attributed to the improved dispersion of the nanoparticles. The SEM observation on the wear tracks surface for the pure epoxy matrix and its composites filled with various kinds of nanoparticle were discussed.

Sirong Yu *et al.* (2008) investigated the effect of nanorubber particles on the tribological properties of epoxy under dry sliding friction and different applied loads. The microhardness, morphologies and chemical compositions of the surfaces of epoxy resin and its nanocomposites before and after wear were analyzed. The results revealed the influence of the nanorubber particle content on the wear resistance and friction coefficients of the nanocomposites. The 5 wt% content of the nanorubber particles was the most effective in reducing the wear mass loss and friction coefficient of the nanocomposites. With further increase in the content of the nanorubber particles, the nanorubber particles agglomerated, and the hardness of the nanocomposites decreased, which resulted in the increase of the specific wear rate. The wear mass losses of epoxy and its nanocomposites increased with increasing sliding time and applied load. The content of the nanorubber particles influenced the wear mass loss and friction coefficient of nanocomposites. The friction coefficients of epoxy and its nanocomposites increased initially with increasing applied load, and then decreased when the applied load was higher than 50 N.

Thomas O. Larsen *et al.* (2008) took different amounts of CuO nanoparticles and incorporated them into both a neat epoxy resin and an epoxy resin containing PTFE micro particles. The content of CuO was varied in the range of 0–10 vol. % while the PTFE content was fixed at 7.5 vol.%. The dispersion state of added particles was examined by SEM and Transmission Electron Microscopy (TEM), which showed a relatively good dispersion of both kinds of particles. Differential Scanning Calorimetry (DSC) and Vickers hardness measurements showed no clear changes in glass transition temperature or hardness as a function of the nano-CuO content. Both parameters were reduced when PTFE was added. Friction and wear data was collected using a custom-made tribotester of the pin-on-disk type.

Measurements were performed under dry-sliding conditions against smooth steel counterfaces. When a pressure–velocity (*PV*) condition of 0.25MPa, 6.0 m/s was applied the following was found: without PTFE, the coefficient of friction (μ) was roughly independent of the nano-CuO content. When PTFE was added, an average reduction in μ of 35% was found in the CuO range of 0–0.4 vol. %. At higher CuO concentrations the friction lowering effect of PTFE deteriorated. Addition of CuO increased wear relative to the neat epoxy at all concentrations. When nano-CuO was added to epoxy with PTFE incorporated, the wear rate decreased slightly up to a CuO content of 0.4 vol.% after which it increased. The measurements were repeated for some of the composites using a smoother counterface. This gave rise to significantly less wear, which for composites without PTFE was attributed to formation of a protective transfer film. At a *pv* condition of 1.16MPa, 1.0 m/s the following was found: composites without PTFE generally showed an unsteady behaviour with high average wear rates whereas composites with PTFE generally showed a good friction and wear performance. The best results were seen at CuO content in the range of 0.1–0.4 vol. %. The latter showed a positive synergistic effect of the addition of a small amount of nano-CuO together with PTFE into the epoxy resin.

Qing Bing Guo *et al.* (2009) prepared epoxy based composites filled with hybrid nano-SiO₂ particles and short pitch based carbon fiber. Copolymer of styrene and maleic anhydride was grafted onto the nanoparticles prior to the compounding so that the nanoparticles could be covalently connected to the composites' matrix through the reaction between anhydride and epoxide groups during curing. The nano- SiO₂/matrix interfacial interaction was enhanced. By evaluating sliding wear properties of the composites as a function of the components concentrations, positive synergetic effect was found. Both wear rate and friction coefficient of the hybrid composites were significantly lower than those of the composites containing individual nano- SiO₂ or short carbon fiber. The composite with 4 wt.% nano SiO₂ and 6 wt.% carbon fiber offered the greatest improvement of the tribological performance. Compared to the results of hybrid composites reported so far, the mentioned composite was characterized by relatively lower filler content, which facilitated processing in practice. Increased surface hardness, lubricating effect of the sheet-like wear debris reinforced by nano- SiO₂ and rapidly formed transfer film were the key issues that accounted for the remarkable wear resisting and friction reducing behaviours.

X. H. Zhou *et al.* (2009) analysed several composites of epoxy reinforced with carbon fabric fabricated with different processes. Tribological properties of the composites which paired with 45 carbon steel under unlubricated ring-on-ring face contact sliding condition were carried out on a friction and wear tester which was typed by MPX-2000. A SEM equipped with an Energy-Dispersive X-ray Analyzer (EDS) attachment was used to analyze the worn surfaces of carbon fabric/epoxy (CF/EP) composites

and counterparts after experiments. The results showed that composite fabricated with semi dry process tended to exhibit a uniform $\mu - t$ behaviour among specimens in the same group as well as on different layers of each specimen as compared with those fabricated with wet process. The $\mu - t$ behaviour of the composite was characterized by two steady stages when the cohesive content was less than 40 vol.%. The first steady-state μ decreased with the lubricant filler contents increasing and the second steady-state μ tended to be close (0.35–0.4). The $\mu - t$ behaviour remained smoothed when the cohesive content was more than 40 vol. %. Lower steady-state μ could be achieved by the composite fabricated by epoxy resin with lower softening point and modified by MoS₂ and graphite. But the wear rate increased correspondingly. SEM and EDS results indicated that lubricant component in the wear debris evenly distributed on the worn surface which underwent fatigue wear played an important part in reducing the CF/EP composite friction coefficient.

Qing Bing Guo *et al.* (2010) investigated the epoxy based quaternary composites that were produced by filling lubricant oil-loaded microcapsules, surface grafted nano-SiO₂ and discontinuous carbon fibers. Through orthographic tests, the optimal contents of the fillers were determined for achieving significantly improved tribological properties. The lowest specific wear rate and friction coefficient of the composite was found to be much lower than those of unfilled epoxy. The mechanical properties, especially the maximal loading ability, of the quaternary composite were evidently higher than those of the binary composite with oil-loaded microcapsules. The maximal loading ability of epoxy was increased by over two times. The oil-loaded microcapsules were very effective in reducing wear rate and friction coefficient by releasing trace quantity of lubricant to the rubbing surface, while the grafted nano-SiO₂ and short carbon fibers played dual roles of both solid lubricant and reinforcement. The weakness brought by the addition of the oil-loaded microcapsules (i.e. mechanical properties deterioration of the composites) was overcome by introducing the grafted nano-SiO₂ and short carbon fibers. During sliding wear, molecular structural degradation of frictional surface was greatly inhibited, and wear rate and friction coefficient of the composite was further decreased to a very low level accordingly.

Siddhartha *et al.* (2011) investigated Titania (TiO₂) reinforced homogeneous and functionally graded epoxy composites developed by simple mechanical stirring and vertical centrifugal casting technique respectively. Investigations on mechanical and wear characteristics of TiO₂ reinforced homogeneous epoxy composites and its functionally graded composite materials developed for tribological applications were presented. The effect of various operational variables, material parameters and their interactive influences on specific wear behaviour of these composites were studied systematically. A series of test were conducted on a pin-on-disc machine with three sliding velocities of 105, 209 and 314

cm/s under three different normal loading of 20 N, 30 N and 40 N. Out of all samples 20 wt.% epoxy–TiO₂ epoxy graded composites exhibited lowest specific wear rate TiO₂ particle additions on epoxy graded composites have a dramatic effect on the flexural strength, tensile modulus and impact strength in comparison to homogeneous composites. SEM observations also indicated that in homogeneous composites TiO₂ particles peeled off from the matrix to form holes while in graded composite materials under same experimental conditions the TiO₂ particles remained quite intact to the matrix.

D Lingaraju *et al.* (2011) analyzed the effects of nanoparticles as fillers in glass–epoxy composite systems on the mechanical and tribological properties. The mechanical properties such as tensile strength, impact strength, flexural strength, and hardness were studied. The composites employed in the study were fabricated using hand lay-up technique. By varying notch radius impact strength was studied. The clay and silica used in the present system were treated with 3-aminopropyltriethoxysilane. The effect of variants in sliding speed, time and applied load on the wear behaviour of polymer nanocomposites was studied by measuring the weight changes and observing the surface features using SEM. In the experiments with wear test pin having flat face in contact with hardening rotating steel disc, sliding speed, time and loads in the range of 640–1000 RPM, 300–900 s and 5–25 N respectively was used. Applied load and sliding velocity influenced the wear process. The low load and speed combination showed tendency for the matrix adhering to the nanoparticles and lesser degree of debris formation. It was found that the mechanical properties of the fibre reinforced composite showed improvement with the inclusion of nanoparticles. The dry slide wear test results of hybrid nanocomposites showed lower slide wear rates irrespective of the load/sliding speed when compared to pure composite. Addition of 2 wt% of HNT found 28% improvement of impact strength, 12% improvement in tensile strength, 4.2% improvement in hardness and 490% improvement in wear rate. Addition of 1 wt% of silica showed improvement of 6.6% in impact strength, 2.09% in tensile strength, 2% improvement of Barcoll hardness and 750% reduction in wear rate.

C. Kanchanomai *et al.* (2011) evaluated the dry-sliding wear of epoxy resin filled with 68.5 wt. % of crushed-silica particles (20–40 μm) using pin-on-disk tests. At low sliding speeds, the bulk temperatures at contacts were below glass-transition temperature ($<T_g$); the wear process was time dependence. The volume loss increased with increase in sliding speed. Wear mechanisms were either abrasive wear or abrasive wear and particle detachment with epoxy matrix deformation at low temperature. The wear process became temperature dependent at high sliding speeds ($>T_g$), i.e. the volume loss decreased with increase in sliding speed. Wear mechanisms were abrasive wear and particle detachment with epoxy matrix deformation at high temperature.

Yingke Kang *et al.* (2012) investigated the hydrophilic silica nano particles (abridged as nano-SiO₂) surface-capped with epoxide dispersed in the solution of epoxy resin (abridged as EP) in tetrahydrofuran under magnetic stirring. Resultant suspension of nano-SiO₂ in EP was then coated onto the surface of glass slides and dried at 80 °C in a vacuum oven for 2 hours, generating epoxy resin-nanosilica composite coatings (coded as EP/nano-SiO₂). EP coating without nano-SiO₂ was also prepared as a reference in the same manner. A water contact angle meter and a surface profiler were separately performed to measure the water contact angles and surface roughness of as-prepared EP/nano-SiO₂ composite coatings. The friction and wear behavior of as prepared EP/nano-SiO₂ composite coatings sliding against steel in a ball-on-plate contact configuration under unlubricated conditions were evaluated. The effect of coating composition on the friction and wear behavior of the composite coatings in particular was highlighted in relation to their microstructure and worn surface morphology examined by means of SEM. Results indicated that EP/nano-SiO₂ composite coatings had a higher surface roughness and water contact angle than EP coating. The EP-SiO₂ coatings doped with a proper amount of hydrophilic SiO₂ nanoparticles show lower friction coefficient than EP coating. But the introduction of surface capped nanosilica as the filler resulted in inconsistent change in the friction coefficient and wear rate of the filled EP-matrix composite..

G. Zhang *et al.* (2012) examined the role of mono dispersed SiO₂-nanoparticles, with an average diameter of 20 nm (as introduced via a sol-gel process), on the tribological behaviour of conventional epoxy (EP) composites, i.e. EP filled with short carbon fibers and graphite flakes. Moreover, the contribution of these nanoparticles was directly compared to that of sub-micron (TiO₂/ZnS) particles (300nm in diameter). The tribological performance of the composites when sliding either against a rough stainless steel ($R_a = 0.30$ mm) surface or an initially mirror polished counterface ($R_a = 0.01$ mm) was compared. The mono dispersed nanoparticles lead to a significant reduction in the friction and wear rate, and they contributed significantly better to the tribological performance than the sub-micron particles. When sliding took place against the rough surface, the nanoparticles resulted in an extremely thin transfer film, which was created from the initial roughness while its grooves were filled up completely. A rough counterface seemed to be an essential precondition for the starting of material transfer. With the polished counterface, significant material transfer started only after the counterface was scratched. This was true especially for the conventional composite and the hybrid composite reinforced with sub-micron particles. The addition of nanoparticles promoted the formation of a homogeneous transfer film before the counter body was severely scratched.

2.2 SUMMARY OF LITERATURE REVIEW

Following is a table of summary of the literature review.

Table 2.1 Summary of the literature review

Sr. No.	Name of the Author (Year of publication)	Title of the paper	Materials used	Type of testing apparatus used
1.	L. Chang <i>et al.</i> (2005)	Tribological properties of epoxy nanocomposites, I. Enhancement of the wear resistance by nano-TiO ₂ particles.	Epoxy filled with short carbon fibre, graphite, PTFE and nano TiO ₂ .	Pin on disk apparatus.
2.	L. Chang <i>et al.</i> (2006)	Tribological properties of epoxy nanocomposites Part II. A combinative effect of short carbon fibre with nano-TiO ₂ .	Epoxy filled with short carbon fibre, graphite, PTFE and nano TiO ₂ .	Pin on disk apparatus.
3.	Guijun Xian <i>et al.</i> (2006)	Friction and wear of epoxy/TiO ₂ nanocomposites: Influence of additional short carbon fibers, Aramid and PTFE particles.	Epoxy-based nanocomposite containing graphite powder (7 vol %) and nano-scale TiO ₂ (4 vol %)	Block on ring apparatus.
4.	Hui Zhang <i>et al.</i> (2007)	Effect of fiber length on the wear resistance of short carbon fiber reinforced epoxy composites.	Short carbon fiber (SCF) reinforced epoxy composites	Block on ring and a pin on disk apparatus
5.	Jin Chein Lin (2007)	Compression and wear behaviour of composites filled with various nanoparticles.	Epoxy matrix with Na-montmorillonite and titanium dioxide nanoparticles.	Block on disc apparatus.
6.	Sirong Yu <i>et al.</i> (2008)	Tribological properties of epoxy/rubber nanocomposites.	Epoxy with nanorubber particles.	Block on wheel apparatus.
7.	Thomas O. Larsen <i>et al.</i> (2008)	Changes in the tribological behaviour of an epoxy resin by incorporating CuO nanoparticles and PTFE microparticles	Epoxy and Epoxy containing PTFE microparticles with CuO nanoparticles.	Pin on disk apparatus.

8.	Qing Bing Guo <i>et al.</i> (2009)	Sliding wear performance of nano-SiO ₂ /short carbon fiber/epoxy hybrid composites	Epoxy based composites filled with hybrid nano-SiO ₂ particles and short pitch based Carbon fibre.	Block on ring apparatus.
9.	X.H. Zhou <i>et al.</i> (2009)	Influences of carbon fabric/epoxy composites fabrication process on its friction and wear properties, Journal of Materials Processing Technology	Epoxy reinforced with carbon fabric.	Ring on ring face apparatus.
10.	Qing Bing Guo <i>et al.</i> (2010)	Optimization of tribological and mechanical properties of epoxy through hybrid filling	Epoxy based quaternary composites with filled lubricant oil-loaded Microcapsules, surface grafted nano-SiO ₂ and discontinuous carbon fibers.	Block on ring apparatus.
11.	Siddhartha <i>et al.</i> (2011)	Mechanical and dry sliding wear characterization of epoxy-TiO ₂ particulate filled functionally graded composites materials using Taguchi design of experiment.	Titania (TiO ₂) reinforced homogeneous and functionally graded epoxy composites.	Pin on disk apparatus.
12.	D Lingaraju <i>et al.</i> (2011)	Mechanical and tribological studies of polymer hybrid nanocomposites with nano reinforcements	Epoxy resin with the reinforcing clay nano particle Halloysite nanotubler (HNT) clay.	Pin on disk apparatus.
13.	C. Kanchanomai <i>et al.</i> (2011)	Wear characteristic of epoxy resin filled with crushed-silica particles	Epoxy matrix with crushed silica.	Pin on disk apparatus.
14.	Yingke Kang <i>et al.</i> (2012)	Friction and wear behaviour of nanosilica filled epoxy resin composite coatings	Nanosilica-filled epoxy resin.	Ball on plate apparatus.

15.	G. Zhang <i>et al.</i> (2012)	Role of mono dispersed nanoparticles on the tribological behaviour of conventional epoxy composites filled with carbon fibers and graphite lubricants.	Epoxy with monodispersed SiO ₂ -nano particles.	Pin on disk apparatus.
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2.3 PROBLEM FORMULATION

From the literature review it is evident that there is very less amount of work done and reported so far in the field of polymer hybrid nano-composites therefore a lot of scope of research is there. Although there has been a significant research done in the area of testing the wear properties of polymer nano-composites with nano particles of a single type, still there is a lot of scope in testing the synergistic effects of the different feasible combinations possible among the different nano particles available. So, the present work deals with finding the wear properties of polymer hybrid nano-composites. The matrix material to be taken is epoxy with TiO₂ and nano-clay as nano particles.

CHAPTER 3

EXPERIMENTATION

The following sections give the details of different materials used, methodology followed and different apparatus used for the experimentation of the present work. The present work is based on epoxy matrix nano-composites. The detail of the materials is given below.

3.1 MATERIALS

Resin, Hardener, Accelerator

DGEBA (Di Glycidyl Ether of Bisphenol-A) base resin was used as epoxy matrix. The resin was labelled as 'L-12' and was supplied by Atul Ltd., Gujarat, India. The epoxy resin used is a di-functional base resin. The epoxy resin used was of light green color. K-12 hardener was used for the resin. K-13 accelerator was used for the resin. Both the hardener and the accelerator were amine based and were also supplied by Atul Ltd., Gujarat.

Cloisite 30B

CLOISITE 30B (Organically modified nanoclay) is an alkyl quaternary ammonium salt bentonite. It had a moisture content of less than 3%, typical Dry Particle Size of $<10\mu\text{m}$ (d50), Off White colour, X Ray Results: $d_{001} = 1.85\text{nm}$, packed bulk density of 365g/l and density of 1.98g/cc . it was supplied by 'Southern Clay Products', Mumbai, Maharashtra, India.

TiO₂

TiO₂ was of white color. TiO₂ (Degussa P-25), which was 99% pure and size less than 20 nm, was purchased from 'Degussa company', Frankfurt, Germany. It has BET surface area of $50\pm 15\text{ m}^2/\text{g}$ and is 80% in anatase crystal form and 20% rutile with average particle size of 30 nm.

3.2 PREPARATION OF COMPOSITES

The nano-composites were prepared following a standard procedure which included mechanical stirring followed by sonication, then mixing of hardener along with stirring and sonication followed by curing at elevated temperature for a certain time period. This procedure was followed after an intense study of the literature. The final nano-composite is used to prepare the samples. The detail of the process is given in the following section.

3.2.1 PROCESSES

1. Mixing of nanoparticles in Epoxy resin

Nano-particles were mixed with the resin using a 'Hot Plate Magnetic Stirrer' shown in Figure 3.1. The temperature of the plate was kept at 60°C. At elevated temperatures the viscosity of the resin reduced significantly therefore helping the nano-particles to get mixed easily. A magnetic bead was used which carried out the stirring action. The stirring was done for 2 hours.

2. Sonication.

Sonication is the process of using sound waves for the purpose of obtaining a better mixing action. Two types of sonicators exist. Water bath sonicator and probe sonicator. Water bath sonicator was used in the present work is shown in Figure 3.2. Sonication helps in better stirring action as it disrupts the intermolecular connections.

3. Mixing of hardener and accelerator in the Epoxy and nano-particles mixture.

The hardener was mixed in the ratio 1:1 of epoxy resin. Accelerator was added after the hardener to accelerate the action of curing. Both the hardener and accelerator were amine based. Manual mixing of the hardener and accelerator were done.

4. Stirring using mechanical stirrer

After the addition of hardener and accelerator, the resultant mixture was stirred again for 15 minutes at 2000 rpm on a stirrer for effective dissolution of nanoparticles. The mechanical stirrer is shown in Figure 3.3. Thereafter, ultrasonication was done for a brief interval of 15 minutes.

5. Degassing in a vacuum oven

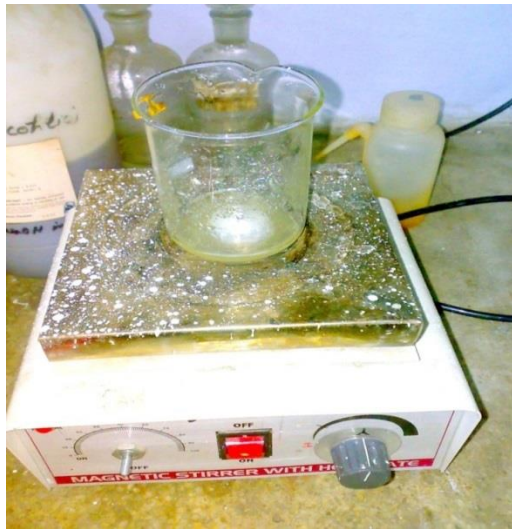
Degassing of the mixture was done in order to remove any bubbles that were formed during the stirring and ultrasonication. Degassing is important as the retention of bubbles may vary the results. It was done in a vacuum oven. This was done at room temperature as the elevation in temperature would have led to the curing of the mixture.

6. Teflon mould

Finally prepared nano-composite was used to prepare the samples. After a number of trials on different materials, the samples were prepared using Teflon material. The prepared mould was of parting type. Vacuum grease (LR) was applied in the mould so that the samples could be removed easily and prevented any kind of leakage from the mould.

7. Curing in the oven

An electric oven was used for curing the mixture by adjusting to the desired temperature settings.



a)



b)

Fig. 3.1 a) Magnetic stirrer with hot plate b) Mechanical stirrer of Remi motors



Fig. 3.2 Water bath sonicator machine



Fig. 3.3 Teflon parting mold

3.2.2 Overview of the process for sample preparation

a) Preparation of neat epoxy sample

- i. Epoxy (L-12) of about 14.70 g weight was taken.
- ii. Hardener (K-12) in the ratio 1:1 and accelerator 2 % by weight was taken.
- iii. Mechanical stirring was done on a 'Hot plate magnetic stirrer' at 60°C for about 2 hours.
- iv. Degassing was done in a vacuum oven for 1 hr.
- v. The resultant mixture was then poured into a Teflon mould.
- vi. The mixture was allowed to cure in the oven.
- vii. The temperature of the oven was maintained at 60°C for 1 hour to achieve the gel time.
- viii. Then the temperature was elevated to 180°C for 30 minutes.
- ix. It was then left at room temperature for about 12 hours for post curing.

b) Preparation of 1% Cloisite 30B sample.

- i. Epoxy resin (L-12) was taken.
- ii. It was mixed with 1% (Weight) of Cloisite 30B.
- iii. The mixture was mechanically stirred with a glass rod for 10 minutes and then stirring was carried on a 'Hot plate magnetic stirrer' at 60°C for about 2 hours.
- iv. The mixture was then sonicated for 2 hours in bath sonicator.
- v. Hardener (K-12) in 1:1 ratio with respect to the resin and accelerator (K-13) 2% (wt.) was mixed and stirred for about 15 minutes at 2000 rpm on the stirrer.
- vi. Sonication was done for 15 minutes in water bath sonicator.
- vii. Degassing was done in a vacuum oven for 2 hours.
- viii. The resultant mixture was then poured in a Teflon mould.
- ix. The mixture was allowed to cure in the oven.
- x. The temperature of the oven was maintained at 60°C for 1 hour to achieve the gel time.
- xi. Then the temperature was elevated to 180°C for 30 minutes.
- xii. It was then left at room temperature for about 12 hours for post curing.

Similarly, the samples of 2%, 3% and 4% of Cloisite 30B were prepared following the same procedure.

c) Preparation of 2% Cloisite 30B and 2% TiO₂ hybrid nano-composite sample.

- i. Epoxy resin (L-12) was taken.
- ii. TiO₂ of 2% (wt.) was taken.
- iii. The mixture was mechanically stirred with a glass rod for 10 minutes and then stirring was carried on a 'Hot plate magnetic stirrer' at 60°C for about 2 hours.
- iv. 2% (wt.) of Cloisite 30B was added and again stirring was done on hot plate magnetic stirrer at 60 °C for 2 hours.
- v. Sonication was done in the water bath sonicator for 2 hours.
- vi. Hardener (K-12) weighing 14.1 g was mixed.
- vii. Addition of 0.6 g of accelerator (K-13) was done.
- viii. Stirring at 2000 rpm was done for 15 minutes on a stirrer.
- ix. Sonication in a water bath sonicator was done for 15 minutes.
- x. Degassing was done in a vacuum oven for 2 hours.
- xi. The resultant mixture was poured in the Teflon mould.
- xii. The mixture was allowed to cure in the oven.
- xiii. The temperature of the oven was maintained at 60°C for 1 hour to achieve the gel time.
- xiv. Then the temperature was elevated to 180°C for 30 minutes.
- xv. It was then left at room temperature for about 12 hours for post curing.

Similarly, samples of 2% Cloisite 30B and 4% TiO₂, and 2% Cloisite 30B and 6% TiO₂ concentration were made following the above mentioned steps.

3.3 TESTING

Pin on disk type machine (shown in Fig. 3.4) was used for performing the wear tests. The machine (TR-20 LE) used, was wear monitoring test rig manufactured by Ducom Ltd, to estimate the performance of the prepared nano-composites under dry sliding conditions. The machine housed a rotating disk which acted as the counter body made of hardened ground steel with a surface roughness of 5µm (EN-32, hardness 72 HRC). The specimen was held stationary and the steel disk rotates. A pin was pressed upon it by a lever that was attached to the load carrier on which the weights were put on. The pin was held by a specimen holder (shown in Fig. 3.4 c)) that could hold a cylindrical object with diameter of 12 mm. The velocity of the wear testing machine and the duration of the test were adjusted by the controller of the machine with manually adjusting the rpm lever and the timer dial respectively. All the tests were conducted in the ambient conditions. The

tests on the wear testing machine were in accordance with ASTM g-99 standard. So, all the specimens (shown in Fig. 3.5) made for testing were made in accordance with ASTM g-99 standard as compatible with the wear testing machine. The specifications of the wear testing machine are enlisted in the table 3.1

Table 3.1 Specifications of the Pin-on-Disk machine.

Parameter	Minimum	Maximum
Pin size(mm)	3	12
Disk size(mm)	165	165
Wear track diameter (mm)	20	145
Disk rotation (rpm)	0	2000

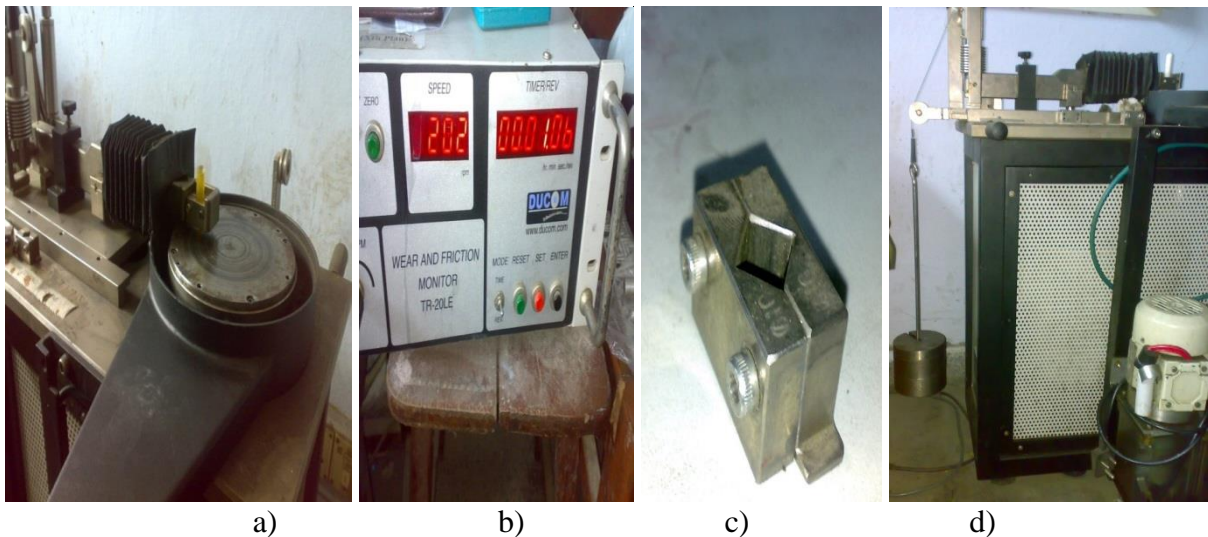


Fig. 3.4 a) Pin-on-disk machine used for the experiment b) Control unit of the machine c) Specimen holder d) Hanging weights used for applying normal load

The specimens were tested at different loading and velocity conditions. Loads ranging from 3kg-5kg were used and velocities of 200, 400 and 600 rpm were chosen. All the tests were conducted for 1 hour duration. Each test was performed three times. For the calculation of wear, the load was taken in Newton, the velocity in m/sec and the time duration in seconds. The wear track was cleaned with acetone prior to every test to remove any debris left over from the previous test. Specimens were desired to have a flat face for the wear testing. So, all the specimens were pre-run on the disk for 2 hours prior to the actual test to get a smooth flat surface and maximum surface in

contact for uniform material removal. A precision electronic balance with accuracy of ± 0.0001 g (Shown in Fig. 3.5) was used to measure the material loss from the composite surface during the sliding test. The specific wear rate was expressed on volume loss basis. Equation 3.1 was used to compute the specific wear rate (W_s).

$$W_s = \frac{\Delta m}{\rho \times t \times V_s \times F_N} \quad \text{--(3.1)}$$

Where,

W_s = the specific wear rate in $\text{mm}^3/\text{N m}$.

Δm = the material removed in g.

ρ = density of the composite in g/mm^3 .

t = time in seconds.

V_s = sliding velocity in m/sec.

F_N = average normal load in Newton.

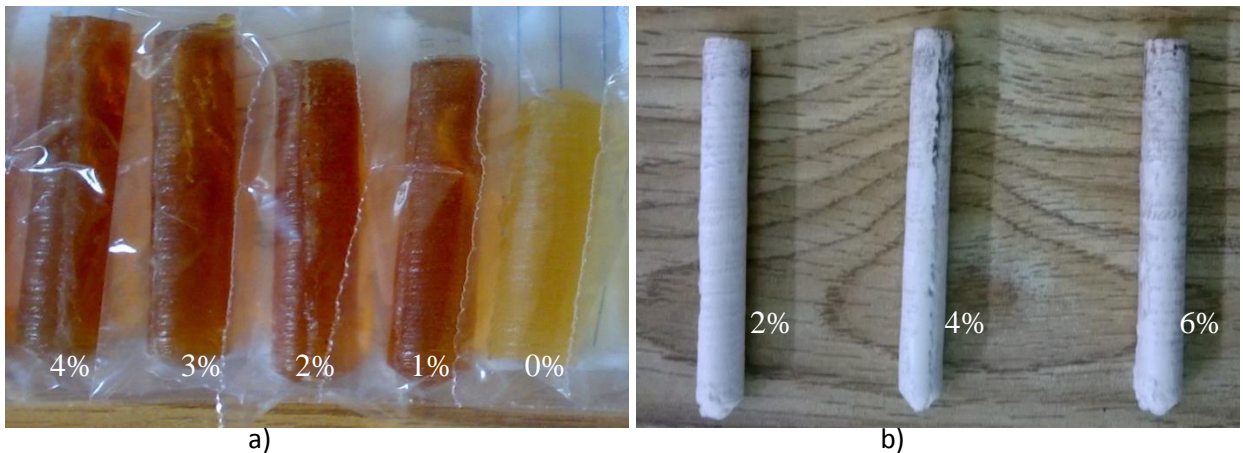


Fig 3.5 a) Specimens of Cloisite 30B(0%-4%) b) Specimens of hybrid Cloisite 30B (2%) and TiO_2 (2%,4% and 6%)



Fig. 3.6 CAS weighing machine

4.1 RESULTS AND DISCUSSION

The material loss in the wear tests was measured and used for the calculation of the specific wear rate. At different loading conditions, different amount of removal of material was found. The data depicting the material loss under different loading conditions and different velocities is reported in the following tables:-

Table 4.1 Wear results for neat epoxy

Sr. No.	Load (kg)	Time (sec)	Velocity V_s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss ' Δw ' (g)	Wear Rate W_s (mm^3/Nm)
1.	3 kg	3600	200	4.7696	4.7646	5.0×10^{-3}	2.64×10^{-4}
		3600	400	4.7646	4.7588	5.8×10^{-3}	1.54×10^{-4}
		3600	600	4.7588	4.7440	1.48×10^{-2}	2.79×10^{-4}
2.	4 kg	3600	200	4.7440	4.7354	8.6×10^{-3}	3.41×10^{-4}
		3600	400	4.7354	4.7234	12.0×10^{-3}	2.39×10^{-4}
		3600	600	4.7234	4.6956	2.78×10^{-2}	3.69×10^{-4}
3.	5 kg	3600	200	4.6956	4.6788	1.68×10^{-2}	5.33×10^{-4}
		3600	400	4.6788	4.6608	1.80×10^{-2}	2.87×10^{-4}
		3600	600	4.6608	4.6072	5.36×10^{-2}	5.69×10^{-4}

Table 4.2 Wear results for 1% Cloisite 30B

Sr. No.	Load (kg)	Time (sec)	Velocity V_s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss ' Δw ' (g)	Wear Rate W_s (mm^3/Nm)
1.	3 kg	3600	200	5.9239	5.9227	1.2×10^{-3}	7.13×10^{-5}
		3600	400	5.9227	5.9209	1.8×10^{-3}	5.37×10^{-5}
		3600	600	5.9209	5.9186	2.3×10^{-3}	7.95×10^{-5}

2.	4 kg	3600	200	5.9186	5.9165	2.1×10^{-3}	9.36×10^{-5}
		3600	400	5.9165	5.9140	2.5×10^{-3}	5.60×10^{-5}
		3600	600	5.9140	5.9066	7.4×10^{-3}	1.10×10^{-4}
3.	5 kg	3600	200	5.9066	5.9031	3.5×10^{-3}	1.24×10^{-4}
		3600	400	5.9031	5.8989	4.2×10^{-3}	7.52×10^{-5}
		3600	600	5.8989	5.8872	1.17×10^{-3}	1.39×10^{-4}

Table 4.3 Wear results for 2% Cloisite 30B

Sr. No.	Load (kg)	Time (sec)	Velocity V_s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss ' Δw ' (g)	Wear Rate W_s (mm^3/Nm)
1.	3 kg	3600	200	5.0905	5.0892	1.3×10^{-3}	6.75×10^{-5}
		3600	400	5.0892	5.0872	2.0×10^{-3}	5.22×10^{-5}
		3600	600	5.0872	5.0830	4.2×10^{-3}	7.29×10^{-5}
2.	4 kg	3600	200	5.0830	5.0810	2.0×10^{-3}	7.79×10^{-5}
		3600	400	5.0810	5.0784	2.6×10^{-3}	5.10×10^{-5}
		3600	600	5.0784	5.0721	6.3×10^{-3}	8.21×10^{-5}
3.	5 kg	3600	200	5.0721	5.0688	3.3×10^{-3}	1.02×10^{-4}
		3600	400	5.0688	5.0646	4.2×10^{-3}	6.57×10^{-5}
		3600	600	5.0646	5.0541	1.05×10^{-2}	1.09×10^{-4}

Table 4.4 Wear results for 3% Cloisite 30B

Sr. No.	Load (kg)	Time (sec)	Velocity V_s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss ' Δw ' (g)	Wear Rate W_s (mm^3/Nm)
1.	3 kg	3600	200	5.9612	5.9594	1.8×10^{-3}	9.35×10^{-5}
		3600	400	5.9594	5.9572	2.2×10^{-3}	5.47×10^{-5}
		3600	600	5.9572	5.9510	6.2×10^{-3}	9.97×10^{-5}

2.	4 kg	3600	200	5.9510	5.9486	2.4×10^{-3}	8.65×10^{-5}
		3600	400	5.9486	5.9448	3.8×10^{-3}	6.88×10^{-5}
		3600	600	5.9448	5.9372	7.6×10^{-3}	9.16×10^{-5}
3.	5 kg	3600	200	5.9372	5.9312	6.0×10^{-3}	1.73×10^{-4}
		3600	400	5.9312	5.9247	6.5×10^{-3}	9.42×10^{-5}
		3600	600	5.9247	5.9061	1.86×10^{-2}	1.79×10^{-4}

Table 4.5 Wear results for 4% Cloisite 30B

Sr. No.	Load (kg)	Time (sec)	Velocity V_s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss ' Δw ' (g)	Wear Rate W_s (mm^3/Nm)
1.	3 kg	3600	200	5.6538	5.6518	2.0×10^{-3}	9.54×10^{-5}
		3600	400	5.6518	5.6493	2.5×10^{-3}	5.99×10^{-5}
		3600	600	5.6493	5.6430	6.3×10^{-3}	1.00×10^{-4}
2.	4 kg	3600	200	5.6430	5.6404	2.6×10^{-3}	9.30×10^{-5}
		3600	400	5.6404	5.6363	4.1×10^{-3}	7.37×10^{-5}
		3600	600	5.6363	5.6278	8.5×10^{-3}	1.01×10^{-4}
3.	5 kg	3600	200	5.6278	5.6212	6.6×10^{-3}	1.89×10^{-4}
		3600	400	5.6212	5.6135	7.7×10^{-3}	1.10×10^{-4}
		3600	600	5.6135	5.5911	2.24×10^{-2}	2.14×10^{-4}

Table 4.6 Wear results for 2% Cloisite 30B + 2% TiO₂

Sr. No.	Load (kg)	Time (sec)	Velocity V_s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss ' Δw ' (g)	Wear Rate W_s (mm^3/Nm)
1.	3 kg	3600	200	7.0084	7.0078	6.0×10^{-4}	3.17×10^{-5}
		3600	400	7.0078	7.0071	7.0×10^{-4}	1.86×10^{-5}
		3600	600	7.0071	7.0051	2.0×10^{-3}	3.53×10^{-5}
2.	4 kg	3600	200	7.0051	7.0041	1.0×10^{-3}	3.96×10^{-5}

		3600	400	7.0041	7.0028	1.3×10^{-3}	2.59×10^{-5}
		3600	600	7.0028	6.9994	3.4×10^{-3}	4.51×10^{-5}
3.	5 kg	3600	200	6.9994	6.9977	1.7×10^{-3}	5.39×10^{-5}
		3600	400	6.9977	6.9954	2.3×10^{-3}	3.66×10^{-5}
		3600	600	6.9954	6.9890	6.4×10^{-3}	6.79×10^{-5}

Table 4.7 Wear results for 2% Cloisite 30B + 4% TiO₂

Sr. No.	Load (kg)	Time (sec)	Velocity V _s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss 'Δw' (g)	Wear Rate W _s (mm ³ /Nm)
1.	3 kg	3600	200	6.9688	6.9680	8.0×10^{-4}	4.23×10^{-5}
		3600	400	6.9680	6.9669	1.1×10^{-3}	2.92×10^{-5}
		3600	600	6.9669	6.9642	2.7×10^{-3}	4.77×10^{-5}
2.	4 kg	3600	200	6.9642	6.9629	1.3×10^{-3}	5.16×10^{-5}
		3600	400	6.9629	6.9610	1.9×10^{-3}	3.78×10^{-5}
		3600	600	6.9610	6.9567	4.3×10^{-3}	5.30×10^{-5}
3.	5 kg	3600	200	6.9567	6.9547	2.0×10^{-3}	6.35×10^{-5}
		3600	400	6.9547	6.9516	3.1×10^{-3}	4.94×10^{-5}
		3600	600	6.9516	6.9441	7.5×10^{-3}	7.96×10^{-5}

Table 4.8 Wear results for 2% Cloisite 30B + 6% TiO₂

Sr. No.	Load (kg)	Time (sec)	Velocity V _s (rpm)	Initial weight 'w1' (g)	Final weight 'w2' (g)	Weight loss 'Δw' (g)	Wear Rate W _s (mm ³ /Nm)
1.	3 kg	3600	200	7.2556	7.2545	1.1×10^{-3}	5.86×10^{-5}
		3600	400	7.2545	7.2530	1.5×10^{-3}	4.01×10^{-5}
		3600	600	7.2530	7.2496	3.4×10^{-3}	6.01×10^{-5}
2.	4 kg	3600	200	7.2496	7.2480	1.6×10^{-3}	6.35×10^{-5}
		3600	400	7.2480	7.2454	2.6×10^{-3}	5.18×10^{-5}

		3600	600	7.2454	7.2402	5.2×10^{-3}	6.90×10^{-5}
3.	5 kg	3600	200	7.2402	7.2378	2.4×10^{-3}	7.62×10^{-5}
		3600	400	7.2378	7.2340	3.8×10^{-3}	6.06×10^{-5}
		3600	600	7.2340	7.2258	8.2×10^{-3}	8.70×10^{-5}

The results were plotted graphically for different load conditions. These are shown in Fig. 4.1, 4.2, 4.3, 4.4, 4.5 and 4.6.

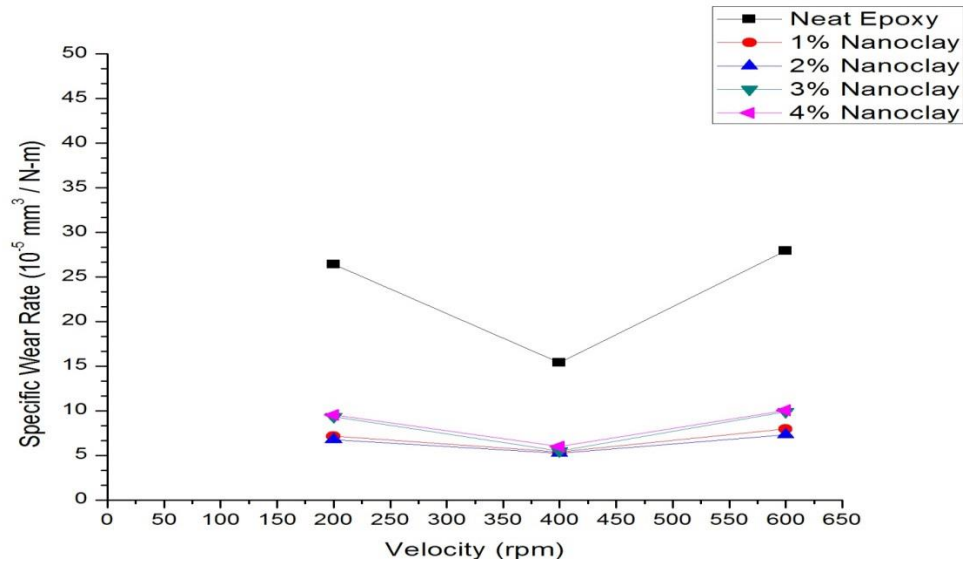


Fig. 4.1 Specific Wear Rate of Cloisite 30B (0%-4%) at 3 kg loading

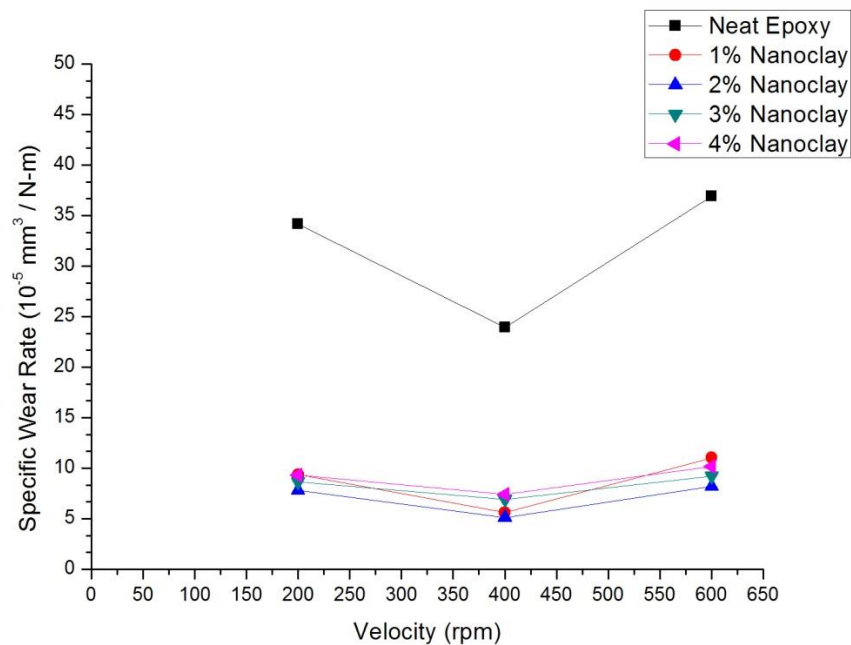


Fig. 4.2 Specific Wear Rate of Cloisite 30B (0%-4%) at 4 kg loading

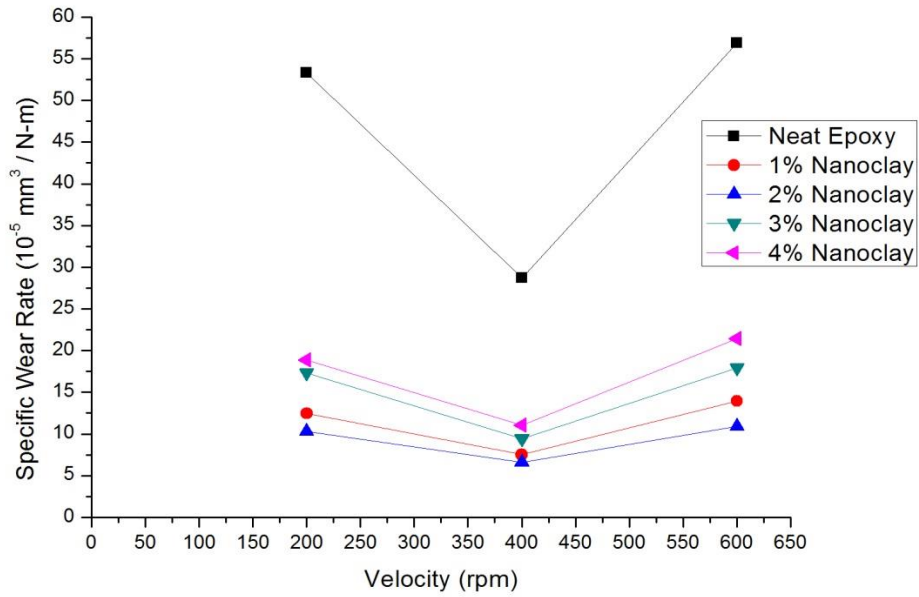


Fig. 4.3 Specific Wear Rate of Cloisite 30B (0%-4%) at 5 kg loading

It is evident from the graphs plotted that at all loading conditions and sliding velocities the nano-composite with 2% Cloisite 30B exhibits the best result followed by 1%, 3% and 4% respectively. The difference between specific wear rates of all the concentrations at lighter loading is less. As the loading increases the difference between the specific wear rates becomes significantly visible. Since, 2% Cloisite 30B displays the least specific wear rate at all the normal loading conditions and sliding velocities so this concentration becomes as an optimized concentration for hybridization.

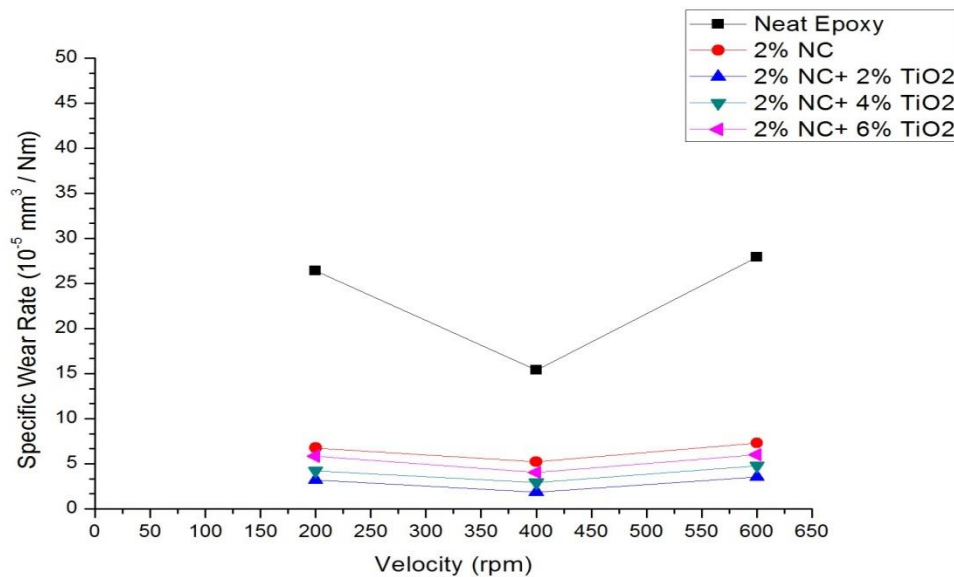


Fig. 4.4 Specific Wear Rate at 3 kg loading of NC and TiO₂

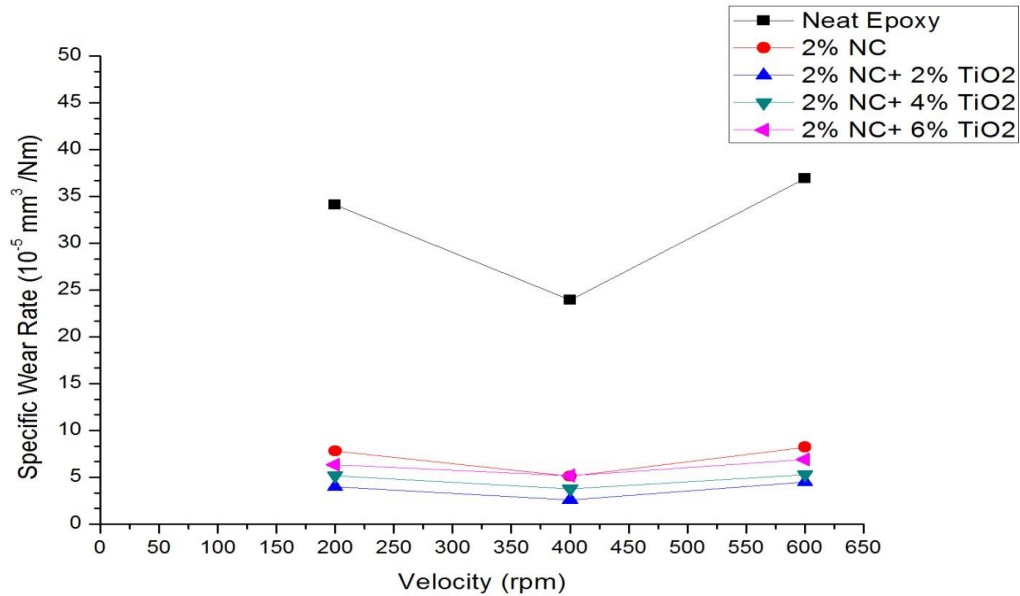


Fig. 4.5 Specific Wear Rate at 4 kg loading of NC and TiO₂

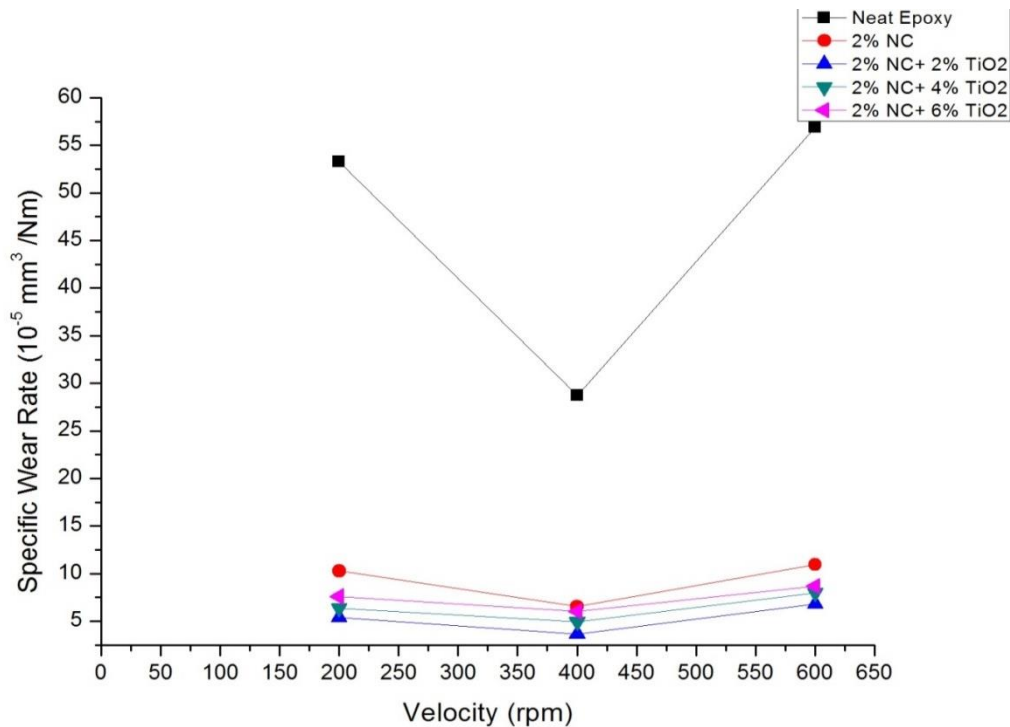


Fig. 4.6 Specific Wear Rate at 5 kg loading of NC and TiO₂

The best wear performance was found to be of 2% Coisite 30B and 2% TiO₂ sample. At this concentration the samples exhibit the least specific wear rate. This holds true for all the normal loading conditions and sliding velocities. This is followed by the sample of 2% Cloisite 30B and 4% TiO₂, and the sample of 2% Cloisite 30B and 6% TiO₂ respectively on all normal loading conditions and sliding velocities. This indicates that only a small addition of nano TiO₂ proves fruitful in improving the specific wear rate and increasing its concentration beyond that deteriorates the wear performance.

5.1 Conclusion

The wear results conducted on neat epoxy composite indicate that the specific wear rates of neat composite were not up to the standard acceptable level. But, with the addition of Cloisite 30B (organic modified nanoclay); there was a significant reduction in specific wear rate at all loading conditions and sliding velocities. The amount of Cloisite 30B was varied from 1 wt. % to 4 wt. % to obtain its optimum concentration. The least specific wear rate was noticed for 2 wt. % of Cloisite 30B. The nanocomposites were then hybridized with the addition of TiO₂ nano particles with 2 wt. %, 4 wt. % and 6 wt. % concentration keeping 2 wt. % Cloisite 30B fixed. The specific wear rates were again tested on pin on disk type machine. The results show that the least specific wear was at 2% Cloisite 30B + 2% TiO₂ concentration followed by 2% Cloisite 30B + 4% TiO₂, and 2% Cloisite 30B + 6% TiO₂ respectively. These observations indicate that amongst the hybridized samples, a small addition of TiO₂ (i.e. 2%) is the optimum concentration in reducing the specific wear rates.

5.2 Future Scope

The present work can be extended in the following ways

- i. The duration of the wear testing experiment can be increased in order to improve the accuracy and reproducibility of the results.
- ii. The test can be conducted at elevated temperatures, for showing the thermal stability of the nanocomposites.
- iii. Carbon fibre reinforcement may be incorporated.

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