Investigation on the Mechanical Properties of TiO_2/SiO_2 Epoxy Hybrid Nanocomposites

Dissertation submitted in partial fulfillment of the requirement for the award of degree

MASTER OF ENGINEERING
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Submitted by

PARVESH KUMAR
Roll No. 801182018

Under Guidance of

Dr. Haripada Bhunia
Associate Professor
Chemical Engg. Department

Dr. Jaswinder Singh Saini
Assistant Professor
Mechannical Engg. Department

MECHANICAL ENGINEERING DEPARTMENT
THAPAR UNIVERSITY
PATIALA-147004, PUNJAB, INDIA

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CERTIFICATE

This is to certify that the dissertation entitled "Investigation on the Mechanical Properties of TiO_2/SiO_2 Epoxy Hybrid Nanocomposites", is an authentic record of my own work carried out as requirements for the award of degree of Master of Engineering in Production and Industrial engineering from Thapar University, Patiala, under the guidance of Dr. Haripada Bhunia (Associate Professor, Chemical Engineering Department) and Dr. J.S. Saini (Assistant Professor, Mechanical Engineering Department) during July 2012 to July 2013.

Date 15/07/13

Parvesh Kumar
(PARVESH KUMAR)

It is certified that the above statement made by the student is correct to the best of our knowledge and belief.

15/7/2013
(Dr. Haripada Bhunia)
Associate Professor
Department of Chemical Engineering
Thapar University, Patiala

15/7/13
(Dr. J. S. Saini)
Assistant Professor
Department of Mechanical Engineering
Thapar University, Patiala

Countersigned by

(Dr. Ajay Batish)
Professor & Head
Department of Mechanical Engineering
Thapar University, Patiala

(Dr. S. K. Mohapatra)
Dean of Academic Affairs
Thapar University, Patiala

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ABSTRACT

Composite is a material that consists of two or more combined constituents that are combined at a macroscopic level. One constituent is called the reinforcing phase and the other in which reinforcement is embedded is called the matrix phase. Composites are one of the most widely used materials because of their adaptability to different situations and the relative ease of combination with other materials to serve specific purposes and exhibit desirable properties. Introduction of small amount of nanoparticles to polymer significantly enhance their properties. Hence the nanocomposites are developed in the present work.

Hand layup technique was used to prepare the nanocomposite with addition of TiO_2 in 2 wt%, 4 wt%, 6 wt% and 8 wt% in epoxy. The best tensile strength, flexural strength and hardness was obtained at 8 wt% of TiO_2. 86 % increase in hardness was obtained at 8 wt% of TiO_2 as compared to epoxy nanocomposites. By fixing 8 wt% of TiO_2 and adding SiO_2 in compositions (2 wt%, 4 wt% and 6 wt%) the hybrid Nanocomposites were prepared, for which mechanical properties were obtained.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 PREVIEW

Composites are one of the most widely used materials because of their adaptability to different situations and the relative ease of combination with other materials to serve specific purposes and exhibit desirable properties. They provide ample scope and receptiveness to design changes, materials and processes. The strength-weight ratios of composites higher than other materials. Their stiffness and cost effectiveness offered, apart from easy availability of raw materials, make them the obvious choice for many applications. In composites, materials are combined in such a way as to enable us to make better use of their virtues while minimizing to some extent the effects of their deficiencies. This process of optimization can release a designer from the constraints associated with the selection and manufacture of conventional materials. The designer can make use of tougher and lighter materials, with properties that can be tailored to suit particular design requirements and because of the ease with which complex shapes can be manufactured, the complete rethinking of an established design in terms of composites can often lead to both cheaper and better solutions. The 'composites' concept is not a human invention. Wood is a natural composite material consisting of one species of polymer cellulose fibers with good strength and stiffness in a resinous matrix of another polymer, the polysaccharide lignin. Nature makes a much better job of design and manufacture than we do, although Man was able to recognize that the way of overcoming two major disadvantages of natural wood that of size (a tree has a limited transverse dimension), and that of anisotropy (properties are markedly different in the axial and radial directions) was to make the composite material that we call plywood. Bone, teeth and mollusk shells are other natural composites, combining hard ceramic reinforcing phases in natural organic polymer matrices. Man was aware, even from the earliest times, of the concept that combining materials could be advantageous, and the down-to-earth procedures of wattle-and-daub (mud and straw) and 'pide' (heather incorporated in hard-rammed earth) building construction, still in use today, pre-date the use of reinforced concrete by the Romans which foreshadowed the pre-tensioned and post-tensioned reinforced concretes of our own era. But it is only in the last half century that the science and technology of composite materials have developed to provide the engineer with a novel class of materials and the necessary tools to enable him to use them advantageously. In many cases, using composites is more efficient. For example, in the highly competitive airline market, one is

continuously looking for ways to lower the overall mass of the aircraft without decreasing the stiffness and strength of its components. This is possible by replacing conventional metal alloys with composite materials. Even if the composite material costs may be higher, the reduction in the number of parts in an assembly and the savings in fuel costs make them more profitable. Composites offer several other advantages over conventional materials. These may include improved strength, stiffness, fatigue and impact resistance, thermal conductivity, corrosion resistance etc. **[1]**

1.2 COMPOSITE MATERIALS

A composite is a structural material that consists of two or more combined constituents that are combined at a macroscopic level and are not soluble in each other. One constituent is called the reinforcing phase and the one in which it is embedded is called the matrix. The reinforcing phase material may be in the form of fibers, particles, or flakes. The matrix phase materials are generally continuous. Composite materials, often shortened to composites or called composition materials, are engineered or naturally occurring materials Examples of composite systems include concrete reinforced with steel and epoxy reinforced with graphite fibers, etc. Examples include wood, where the lignin matrix is reinforced with cellulose fibers and bones in which the bone-salt plates made of calcium and phosphate ions reinforce soft collagen. The reinforcing phase provides the strength and stiffness. In most cases, the reinforcement is harder, stronger, and stiffer than the matrix. The reinforcement is usually a fiber or a particulate. Particulate composites have dimensions that are approximately equal in all directions. They may be spherical, platelets, or any other regular or irregular geometry. Particulate composites tend to be much weaker and less stiff than continuous fiber composites, but they are usually much less expensive. Particulate reinforced composites usually contain less reinforcement due to processing difficulties and brittleness. A fiber has a length that is much greater than its diameter. The length-to-diameter ratio is known as the aspect ratio and can vary greatly. Continuous fibers have long aspect ratios, while discontinuous fibers have short aspect ratios. Continuous-fiber composites normally have a preferred orientation, while discontinuous fibers generally have a random orientation. Examples of continuous reinforcements include unidirectional, woven cloth, and helical winding, while examples of discontinuous reinforcements are chopped fibers and random mat. The continuous phase is the matrix, which is a polymer, metal, or ceramic. The matrix (continuous phase) performs several critical functions, including maintaining the fibers in the proper orientation and spacing and protecting them from abrasion and the environment. The type and quantity of the reinforcement determine the final properties.

1.3 COMPONENTS OF COMPOSITE MATERIALS

1.3.1 Matrix

The matrix is the binder material that supports, separates, and protects the fibers. The matrix binds the fibers together, holding them aligned in the important stressed directions. Loads applied to the composite are then transferred into the fibers, the principal load-bearing component, through the matrix, enabling the composite to withstand compression, flexural and shear forces as well as tensile loads. The ability of composites reinforced with short fibers to support loads of any kind is dependent on the presence of the matrix as the load-transfer medium, and the efficiency of this load transfer is directly related to the quality of the fiber/matrix bond. Although matrices by themselves generally have low mechanical properties compared to those of fibers, the matrix influences many mechanical properties of the composite. These properties include transverse modulus and strength, shear modulus and strength, compressive strength, inter laminar shear strength, thermal expansion coefficient, thermal resistance, and fatigue strength. A ductile matrix will provide a means of slowing down or stopping cracks that might have originated at broken fibers: conversely, a brittle matrix may depend upon the fibers to act as matrix crack stoppers. Through the quality of its 'grip' on the fibers (the interfacial bond strength), the matrix can also be an important means of increasing the toughness of the composite. By comparison with the common reinforcing filaments most matrix materials are weak and flexible and their strengths and moduli are often neglected in calculating composite properties. But metals are structural materials in their own right and in MMCs their inherent shear stiffness and compressional rigidity are important in determining the behavior of the composite in shear and compression. The potential for reinforcing any given material will depend to some extent on its ability to carry out some or all of these matrix functions, but there are often other considerations.

1.3.2 REINFORCEMENT

The strength, stiffness, and density of the composite material are very dependent on the reinforcing material. The ultimate tensile strength of a composite is a result of the synergy between the reinforcement and the matrix. The matrix forces load sharing among all the fibers, strengthening the material. The main types of reinforcements are continuous fibers, discontinuous (short fibers), whiskers, and particulates.

Continuous fibers are long strands of fibers having a small cross sectional area. The fibers are placed in bundles containing in between 1,000 to 12,000 fibers each depending on the parameters of the load the

composite must carry. The bundles are then placed in the matrix in geometrical patterns. If aligned in a single direction, the resulting composite will have anisotropic properties. Anisotropic materials have different properties in different directions. When a load is applied in the direction of the fibers, the fibers become the principle load carrying constituent of the composite. Anisotropic composites are extremely strong in the fiber direction but are generally weak in a direction perpendicular to the fiber. This is analogous to bone, a natural composite. Sometimes one directional properties are not what is called for in the design. A composite may need to bend when it is twisted, or be able to withstand loads in two directions. Laminates, allow for such flexibility in design. Laminates are made by combining one directional composites which have fibers oriented at different angles. The properties of the laminate as a whole depend on the orientation and thickness of each layer.

A second type of reinforcement is discontinuous (short) fibers, which are placed randomly into a matrix. The advantage of discontinuous fibers lies in the fact that the resulting composite tends to be more isotropic than continuous fibers and they are generally easier for industry to fabricate particularly for complex irregularly-shaped component geometries.

1.4 CLASSIFICATION OF COMPOSITES

Composite materials are commonly classified at following two distinct levels:

(i) The first level of classification is usually made with respect to the matrix constituent. The major composite classes include Organic Matrix Composites (OMCs), Metal Matrix Composites (MMCs) and Ceramic Matrix Composites (CMCs). The term organic matrix composite is generally assumed to include two classes of composites, namely Polymer Matrix Composites (PMCs) and Carbon Matrix Composites commonly referred to as carbon-carbon composites.

(ii) The second level of classification refers to the reinforcement form –fiber reinforced composites, laminar composites and particulate composites. Fiber Reinforced composites (FRP) can be further divided into those containing discontinuous or continuous fibers.

In the present chapter only first level of classification is introduced.

- **Organic Matrix Composites**

Polymers make ideal materials as they can be processed easily, possess lightweight, and desirable mechanical properties. It follows, therefore, that high temperature resins are extensively used in aeronautical applications. Two main kinds of polymers are thermosets and thermoplastics. Thermosets

have qualities such as a well-bonded three-dimensional molecular structure after curing. They decompose instead of melting on hardening. Merely changing the basic composition of the resin is enough to alter the conditions suitably for curing and determine its other characteristics. They can be retained in a partially cured condition too over prolonged periods of time, rendering Thermosets very flexible. Thus, they are most suited as matrix bases for advanced conditions fiber reinforced composites. Thermosets find wide ranging applications in the chopped fiber composites form particularly when a premixed or moulding compound with fibers of specific quality and aspect ratio happens to be starting material as in epoxy, polymer and phenolic polyamide resins.

- **Metal Matrix Composites (MMC)**

Metal matrix composites, at present though generating a wide interest in research fraternity, are not as widely in use as their plastic counterparts. High strength, fracture toughness and stiffness are offered by metal matrices than those offered by their polymer counterparts. They can withstand elevated temperature in corrosive environment than polymer composites. Most metals and alloys could be used as matrices and they require reinforcement materials which need to be stable over a range of temperature and non-reactive too. However the guiding aspect for the choice depends essentially on the matrix material. Light metals form the matrix for temperature application and the reinforcements in addition to the aforementioned reasons are characterized by high moduli. Metal Matrix Composites are composed of a metallic matrix (aluminum, magnesium, iron, cobalt, copper) and a dispersed ceramic (oxides, carbides) or metallic (lead, tungsten, molybdenum) phase.

- **Ceramic Matrix Composites (CMC)**

Ceramics can be described as solid materials which exhibit very strong ionic bonding in general and in few cases covalent bonding. High melting points, good corrosion resistance, stability at elevated temperatures and high compressive strength, render ceramic-based matrix materials a favorite for applications requiring a structural material that doesn't give way at temperatures above 1500ºC. Naturally, ceramic matrices are the obvious choice for high temperature applications. Ceramic Matrix Composites are composed of a ceramic matrix and imbedded fibers of other ceramic material (dispersed phase).

- **Polymer Matrix Composites (PMC)**

Recently, many advances have been made in the design, manufacture and application of composite materials which can be very strong and stiff, yet very light in weight, that is strength to weight ratio

and stiffness to weight ratio are several times greater than steel and aluminum. These composites also exhibit fatigue and toughness properties better than common engineering materials. The main drawbacks of PMCs include low operating temperatures, high coefficient of thermal, moisture expansion, and low elastic properties in certain directions. Applications of PMCs range from tennis racquets to the space shuttle. Polymer matrix composites are being increasingly used in industry because of their unique combination of mechanical, electrical, and thermal properties. Typically they have high specific strength and modulus, excellent fracture toughness and fatigue properties, and good corrosion, thermal and electrical resistance properties. This combination of properties, particularly their high strength stiffness to weight ratio, make them very attractive materials for transport applications where there is commercial advantage in minimizing vehicle weight. One such application is in the transport and handling of bulk solids. Two main kinds of polymers are thermosets and thermoplastics. These are discussed below.

Thermoplastic Polymers: Thermoplastics have one or two-dimensional molecular structure and they tend to at an elevated temperature and show exaggerated melting point. Another advantage is that the process of softening at elevated temperatures can reversed to regain its properties during cooling, facilitating applications of conventional compress techniques to mould the compounds. Resins reinforced with thermoplastics now comprised an emerging group of composites. The theme of most experiments in this area is to improve the base properties of the resins and extract the greatest functional advantages from them in new avenues, including attempts to replace metals in die-casting processes. In crystalline thermoplastics, the reinforcement affects the morphology to a considerable extent, prompting the reinforcement to empower nucleation. Whenever crystalline or amorphous, these resins possess the facility to alter their creep over an extensive range of temperature. But this range includes the point at which the usage of resins is constrained, and the reinforcement in such systems can increase the failure load as well as creep resistance. Fig.1.1 shows the types of thermoplastics.

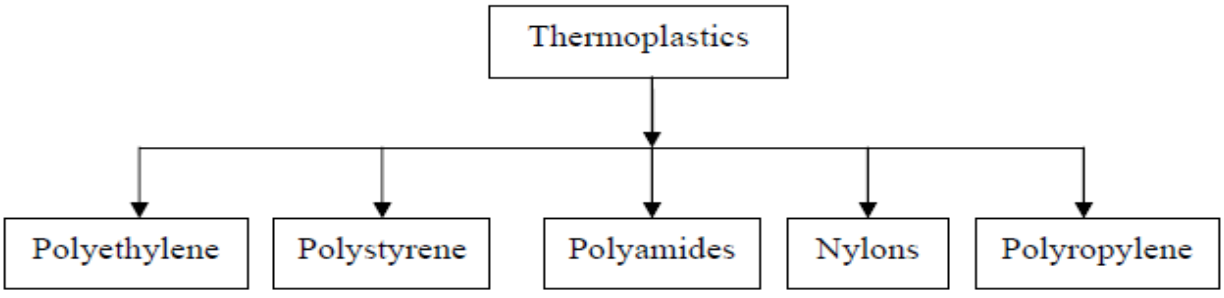

Fig 1.1 Types of Thermoplastics

Thermosetting Polymers: Thermosets have qualities such as a well-bonded three-dimensional molecular structure after curing. They decompose instead of melting on hardening. Merely changing the basic composition of the resin is enough to alter the conditions suitably for curing and determine its other characteristics. They can be retained in a partially cured condition too over prolonged periods of time, rendering thermosets very flexible. Thus, they are most suited as matrix bases for advanced conditions fiber reinforced composites. Fig. 1.2 shows some kinds of thermosets.

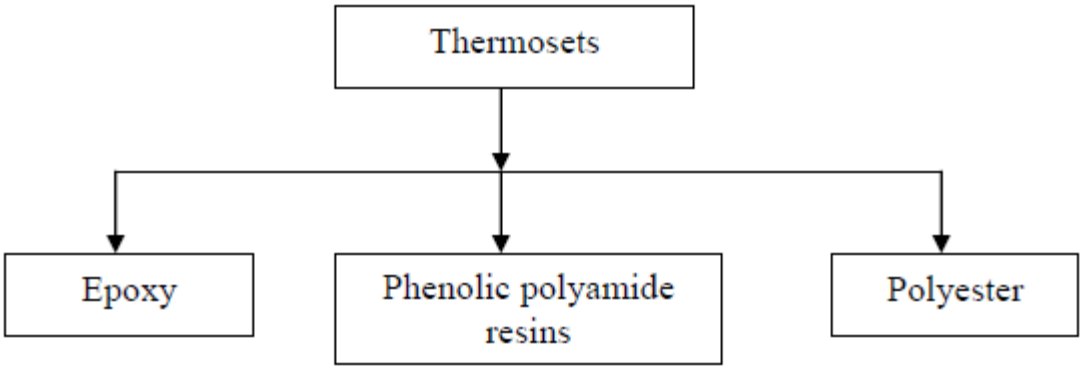

Fig 1.2 Various types of Thermosets

Direct condensation polymerization followed by rearrangement reactions to form heterocyclic entities is the method generally used to produce thermosets resins. Water, a product of the reaction, in both methods, hinders production of void-free composites. These voids have a negative effect on properties of the composites in terms of strength and dielectric properties. Polyesters phenolic and Epoxies are the two important classes of thermosets resins. Epoxy resins are widely used in filament-wound composites and are suitable for moulding prepress. They are reasonably stable to chemical attacks and are excellent adherents having slow shrinkage during curing and no emission of volatile gases. These advantages, however, make the use of epoxies rather expensive. Also, they cannot be expected beyond a temperature of 140ºC. Their use in high technology areas where service temperatures are higher, as a result, is ruled out. Polyester resins on the other hand are quite easily accessible, cheap and find use in a wide range of fields. Liquid polyesters are stored at room temperature for months, sometimes for years and the mere addition of a catalyst can cure the matrix material within a short time. They are used in automobile and structural applications. The cured polyester is usually rigid or flexible as the case may be and transparent. Polyesters withstand the variations of environment and stable against chemicals. Depending on the formulation of the resin or service requirement of application, they can be used up to about 75ºC or higher. Other advantages of polyesters include easy compatibility with few

glass fibers and can be used with verify of reinforced plastic accountrey. Aromatic Polyamides are the most sought after candidates as the matrices of advanced fiber composites for structural applications demanding long duration exposure for continuous service at around 200-250ºC.

1.5 Nano-Composites [2]

A nanocomposite is defined such that the size of the matrix or reinforcement falls within the nanoscale. The physical properties and performance of the nanocomposite will greatly differ from those of the component materials according to the type of matrix, nanocomposites can be classified into ceramic matrix nanocomposites, metal matrix nanocomposites, and polymer matrix nanocomposites.

These days polymer composites are undergoing extensive research because for many fields of the modern industry appreciable improvement of physiomechanical properties of polymeric materials are desirable. Nowadays, the most perspective decision of this problem is a modification polymer with nanostructure modifiers – creation of polymeric nanocomposites. Polymer nanocomposites (PNC) are polymers (thermoplastics, thermo sets, elastomers) that have been reinforced with small quantities (less than 5% by weight) of nanosized particles. Molecular interaction between polymer and nanofillers do not posses. Uniform dispersion of nanoparticles in polymer matrix produces ultra- large interfacial area per volume between the nanoparticle and the host polymer. This immense internal interfacial area and the nanoscopic dimension between nanoparticle fundamentally differentiate PNCs from traditional composites and filled plastics. Introduction to nanoparticles to polymer matrix ensure significant property improvements with very low loading levels. Traditional micro particles additives require much higher filler concentration to achieve similar results. The most commonly used nano fillers are:

(i) Manomorillonite Organoclays (MMT)
(ii) Carbon Nanofiber (CNFs)
(iii) Carbon nanotubes (CNTs)
(iv) Metallic nanoparticles

The value of PNC technology comes from providing value added properties not present in the neat polymer, without sacrificing the inherent processibility and mechanical properties of the polymer. Some of the major advantages of the polymer nanocomposites are related to improvement in mechanical, optical and magnetic properties.

Mechanical properties

- High adhesion of nanoparticles to polymer matrix result in the enhanced strength of nanocomposites relative to conventional composite.
- Small size of nanoparticles ensures small size of pores in the case of exfoliation of a matrix from filler particles. It also results in the increase in strength.
- Introduction of small amount of nanoparticles to polymer significantly enhance the adhesion of polymer to different substance.

Optical properties

- Nanocomposites are optically more transparent in comparison to conventional composites.
- Special optical effects.
- Optical clarity in comparison to conventionally filled polymers.

Magnetic properties

- In some case composites with magnetic nanoparticles have the magneto resistance and magnetic permeability much higher than in the case of conventional composites.
- It is possible to make composites with magnetic threshold concentration smaller than electrical percolation threshold.

CHAPTER 2

LITERATURE REVIEW

A lot of research has been done on polymer composites in the recent years. Polymer Composites have been extensively used in structural components as well as in aeronautical, automotive, marine and sporting industries for a wide range of applications because of their attractive properties, fatigue and corrosion resistance combined with lightweight, high specific stiffness and strength. Extensive literature review has been carried out for defining the research problem. Most of the work carried out is on the characterization of composites made by adding nanofillers to matrix system. A review of the literature is presented in the following section.

2.1 LITERATURE REVIEW

Lu *et al.* [3] studied the mechanical and wear properties of composites. Addition of nano filler TiO_2 and SiO_2 significantly improve the impact and tensile strength which was almost 2-3 times as much as that of pure epoxy resins. The wear and friction performance of composites had been improved with addition of TiO_2 and SiO_2 nano particles.

Yang *et al.* [4] studied that the nano SiO_2 particles were used to modify epoxy emulsion sizing of carbon fibers to improve the reinforced epoxy composites. For investigation of mechanical interfacial strength between fiber and matrix, fragmentation test and three point short beam sheet tests were conducted on unmodified sizing and nano-SiO_2 modified sizing. Dynamic contact analysis X-Ray photoelectron spectrometry and atomic force microscopy also performed on them. The results indicated that modified sizing with nano-SiO_2 1.0 wt% has maximum Interfacial shear strength and Interlaminar shear strength values. SEM images show the fracture section of composites with modified sizing is more compact and fiber debonding is more difficult than that with unmodified sizing. From AFM analysis it was seen that modified sizing changed the surface roughness values on a microscopy scale. As a result nano-SiO_2 modified epoxy emulsion sizing for carbon fibers further improved the chemical and physical properties of the fiber surface, resulting in the increase of the mechanical interfacial properties of the composites.

Chowdhary *et al.*[5] used a nanomar to modify the matrix of carbon fiber/SC-15 epoxy composite to determine the effect of particle reinforcement on the response of these material to flexural and thermo mechanical loading. Nano clay with different percentage was dispersed in SC-15 epoxy with

sonication route and after that hand layup process was used to manufacture plain weave carbon/epoxy nano composite from nanophased epoxy material. Control samples for comparison purpose of woven carbon fiber/epoxy were fabricated. Using dynamic mechanical analysis and three-point bend flexure test effect of post curing was investigated on 8- and 3-layered samples. From the results it was shown that flexural strength and modulus for nano clay reinforced composites as compared to the control samples was significantly improved and also enhancement in mechanical properties especially in storage modulus but in glass transition temperature no significant change was seen.

Zhou *et al.* [6] Conducted thermal and mechanical tests on carbon nano fiber (CNF) filled epoxy and carbon fabric/epoxy composite. The optimal CNF content was 2.0 wt. %, which produced the highest improvement in tensile strength as compared to the neat epoxy resin. The composite fabricated with 2.0 wt. % CNF filled epoxy produced 22.3% improvement in flexural strength and 11% improvement in tensile strength. The addition of CNF in the epoxy matrix also improved the fatigue performance of the composite.

Xu and Hoa [7] studied the carbon fiber reinforced epoxy/clay nanocomposites were manufactured through hot melt layup plus autoclave process. The CFRENCs have uniform fiber volume fraction very few dry spots and very few resin rich areas. The high pressure mixing method was used to disperse nanoclay into TGDDM .From the results of this method it proves to be good for disperse of nanoclay into TGDDM epoxy system. Mode I interlaminar fracture toughness of unidirectional carbon fiber composite was also increased by 53% and 85% with 2 and 4 phrnanoclay. A small amount of nanoclay also contributes to increase of flexural strength. But adding more clay does not improve the flexural properties more.

Chen *et al.* [8] studied the thermal and mechanical properties of nano composites with SiO_2 as a nano filler. With increasing SiO_2 loading, decrease in cure temperature and glass transition temperature occurred. At 10% wt. of SiO_2 increase of 25% in tensile modulus and 30% in fracture toughness was obtained. Further addition of SiO_2 increase in modulus occurred but it decreased the strength and fracture toughness.

Kishore *et al.* [9] studied the failure mode and failure loads for multi-pin joints in unidirectional glass fiber epoxy composite laminates by taking different sizes of sample. With variation in pitch-to-

diameter, side width-to-diameter and edge-to-diameter ratios. With using finite element analysis the theoretical results were validated with the experimental results.

Ogasawara *et al.* [10] studied the effect of fullerene dispersion on the mechanical properties such as tensile, flexural, compression, open hole compression, compression after impact, bending, short beam shear and intermolecular fracture toughness of laminates. Increase in tensile and compression strength had seen up to 2-12% improvement in intermolecular fracture toughness also seen. Fullerene dispersion was improving the CFRP strength.

Khan *et al.* [11] studied the effect of nanoclay inclusion on cyclic fatigue behavior and residual properties of carbon fiber reinforced composites after fatigue. To establish S-N curve tension-tension cycle fatigue test were conducted at various load levels. The residual strength and modulus were measured at different stage of fatigue cycles. From the results of these tests, it was shown that fatigue life significantly extended with the incorporation of nanoclay to CFRP composite and the maximum improvement is about 74% with 3 wt% clay content. The clay-CFRP hybrid composites showed better performance in terms of residual tensile strength and modulus then the neat composite after a given fatigue cycle.

Osman Asi [12] studied the bearing strength behavior of pinned joints of glass fiber reinforced composite which was filled with different percentage of Al_2O_3 (7.7, 10, 15). Single-hole pin loaded specimens of each percentage composite was tested in tension. From the result it was shown that with increase in percentage of Al_2O_3 bearing strength of composite increased and further increase in percentage of Al_2O_3 bearing strength decreased. The highest bearing strengths were obtained for composite specimens with 10 wt.% Al_2O_3 particle content. It compared with the bearing strength of the unfilled glass fiber reinforced epoxy composite, with the addition of 10 wt. % Al_2O_3 particle in the matrix, bearing strength increased by 20%.

A. Jumahat *et al.* [13] implemented fiber kinking and fiber micro-buckling models to predict the compressive strength of the UD HTS40/977-2 toughened composite laminate. SEM micrograph revealed that the failure of the UD HTS40/977-2 composite laminate was initiated by fiber micro-buckling and subsequent plastic kinking of the materials. Therefore, a combined modes model was developed to predict the compressive strength of the system. The combined modes model yielded a successful compressive strength prediction in which the predicted compressive strength was in a good

agreement to the measured value. Considering all the parameters that had been investigated, the in-plane shear properties and the initial fiber misalignment angle between the fibers and the loading axis were identified as the most critical parameters that affect the compressive strength of the UD toughened composite laminates.

Khan *et al.* [14] studied the mechanical properties of clay-epoxy nanocomposites and clay-CFRP hybrid composites. For investigating the impact fracture toughness and quasi-static fracture toughness charpy impact test and compact tension test was conducted on both the materials. The result showed that the properties were increased in both the material with addition of clay up to 3 wt% but decrease after further addition of clay. The impact and quasi-static fracture toughness values of clay-epoxy nano composite was less than the clay-CFRP hybrid composite because of carbon fiber which showed additional toughening.

Zeng *et al.* [15] studied the epoxy matrix with rubber and silica nano particles either single or jointly and applied Double-cantilever-beam test to investigate the interlaminar fracture toughness. From the result it was shown that the toughness was improved by the presence of these nano-particles. Nano rubber was more effective than nano silica. Jointly addition of rubber and silica particles improve toughness but not more than single rubber if added or single silica.

Shadlou *et al.* [16] studied the carbon nano reinforcements of three different shapes for the mixed mode fracture behavior of epoxy-based nanocomposites. On the basis of experimental results, while the cylindrical (CNF) and platelets (GO) particles were more successful in ameliorating the fracture resistance under mode I and mixed mode loading, the spherical (ND) particles developed better fracture resistance in pure mode II.

Zhou *et al.* [17] studied the effect of clay weight fraction on thermal and mechanical properties of the epoxy matrix. The dynamic mechanical analysis, thermo-gravimetric analysis and flexural test were perform in unfilled 1wt%, 2 wt%, 3wt% and 4 wt% clay filled SC-15 epoxy. From the result of analysis it was shown that 2 wt. % loading of MMT clays in SC-15 epoxy resin had the highest improvement in the strength as compared to neat and other nano phased systems. The composite fabricated with 2.0 wt. % clay filled epoxy produced 22.3 % improvement in flexural strength and 11 % improvement in tensile strength. The addition of clay in the epoxy matrix also improved the fatigue performance of the composite.

The summary of discussed research work is given in the Table 2.1.

Table 2.1 Summary of Literature Review

S.No.	AUTHOR	MATERIAL USED	PROPERTIES TESTED
1	Lu *et al.* [3]	Epoxy/SiO2-TiO2	Impact strength, Flexural strength, Tensile strength and Ring-on-block wear
2	Yang *et al.* [4]	Carbon fibers/epoxy composites with nano SiO2	Mechanical Interfacial strength
3	Chowdhary *et al.* [5]	Carbon epoxy nanoclay	Thermal and flexural properties
4	Zhou *et al.* [6]	Carbon /epoxy composite with carbon nano fibers	Tensile, fatigue, fracture toughness, thermal properties
5	Xu and Hoa [7]	Epoxy carbon fiber clay nano composite	Interlaminar fracture toughness, flexural strength
6	Chen *et al.* [8]	Epoxy/SiO_2	Glass transition temperature, Tensile modulus, Fracture toughness
7	Kishore *et al.* [9]	Glass fiber/epoxy composite	Progressive damage
8	Ogasawara *et al.* [10]	carbon fiber reinforced epoxy composite	Tension, Compression, Comparession after impact, Short beam Shear, and Interlaminar Fracture Toughness
9	Khan *et al.* [11]	Epoxy carbon fiber reinforced composite with nano clay	Fatigue, Interfacial strength

10	Osman Asi [12]	Epoxy/glass fiber reinforced with Al2 O3	Bearing strength
11	Jumahat *et al.* [13]	HTS40/977-2	Compressive strength
12	Khan *et al.* [14]	Epoxy Carbon fiber reinforced composite with nanoclay	Flexural strength, fracture toughness
13	Zeng *et al.* [15]	Carbon fiber/epoxy With silica and rubber nano-particle	Interminar Fracture toughness
14	Shadlou *et al.* [16]	Epoxy nano composites reinforced with different carbon nano-reinforcements	Fracture toughness
15	Zhou *et al.* [17]	carbon fiber reinforced clay/epoxy composite	Fatigue strength, flexural strength, thermal stability

2.3 PROBLEM DEFINITION

Use of nano composites is increasing day by day. Engineers are searching for materials which are light in weight and have a good strength. From the literature review it is found that broad researches have done in the area of nanocomposites with single nano filler but limited for hybrid nanocomposites. By adding small amount of single nano filler or integrating two different nano filler with epoxy system mechanical properties can be enhanced. Objective of the present work is to investigate the mechanical properties for hybrid epoxy nano composites. The hybrid epoxy nano composites were prepared using TiO_2 and SiO_2 as nano fillers.

CHAPTER 3
EXPERIMENTATION

3.1 MATERIALS

Epoxy

DGBA based epoxy resin system (Epoxy Resin L-2, Hardener K-12, Accelerator K-13) was obtained from Atul Ltd, Gujrat, India.

Nano Filler

P25 TiO_2 was obtained from Degussa Company, Frankfurt, Germany. It has a BET surface area of 50 ± 15 m^2/g and is 80% in antase crystal form and 20% rutile with average particle size of 30 nm.

SiO_2 nano powder was obtained from Nanoshel, USA. It was of 98% pure and 62 to 70 nm particle size.

3.2 MATERIAL PREPARATION

Material was prepared using DGBA based epoxy with different proportion of nano-fillers. The following steps were used for the preparation:

(i) Mechanical stirring

To get the proper dispersion of epoxy and nano filler mechanical stirring was done. Mechanical stirring, shown in Fig. 3.1 is done at 2000 rpm for 2 hours.

Fig. 3.1 Setup of Mechanical Stirrer

Different percentage of TiO_2 – 2 wt%, 4 wt%, 6 wt% and 8 w% of epoxy were added to the epoxy resin and stirred for 2 hours. To reduce the viscosity of epoxy solution preheating at 60^{o} C for 30 min. was also done.

Fig. 3.2 Setup of Ultrasonication

(ii) Ultrasonication

In sonication sound energy is used to agitate particle in a mixture. Sonication breaks the intermolecular interaction and is used for speed dissolution. Sonication is usually carried out using an ultrasonication bath or an ultrasonication probe. In the present work ultrasonication bath used for 2 hours, as shown in Fig. 3.2.

(iii) Mixing of Epoxy Base Solution with Hardener and accelerator

After ultrasonication, the solution was mixed with the hardener in the ratio of 1:1 by weight, Thereafter 1 wt% accelerator was added as per the instructions of the epoxy system resin. After mixing, mechanical stirring up to 15 minutes was done, followed by ultrasonication for 15 minutes which results in proper dispersion of nanoparticles.

(iv) Degassing

After mixing, stirring and sonication large amount of bubbles appear in the solution. If these are not removed than they can produce defects in the specimen and may effect the results. For removing these bubbles degassing is done. For degassing beaker is placed in vacuum oven for 2 hours.

(v) Post Curing

After degassing a bubble free mixture was then poured in the Teflon sheet mould and left in air for 24 hours. Epoxy is a very sticky material which sticks even on metal sheets. So a Teflon sheet mould was used to prepare the sample sheets. After placing in air for 24 hours it is kept in electric oven (capacity of 300^{o}C) at 120^{o}C up to 2 hours for post curing.

(vi) Specimen Preparation

Specimen was prepared as per ASTM standard D638 and D790 for tensile and flexural tests. The specification are given in Table 3.1

Table 3.1 Specimen specification for testing

Parameters for specimen	Specimen for tensile testing	Specimen for flexural testing
Length (mm)	150	56
Width (mm)	25	25
Thickness (mm)	3	3

The specimens were cut manually, as shown in Fig. 3.3, from the prepared sheet.

Fig. 3.3 Cutting of Specimen from sheet

3.3 MECHANICAL PROPERTIES

The prepared specimens were tested for mechanical properties.

3.3.1 Tensile Testing

A Universal Tensile Machine (UTM), shown in Fig. 3.4 was used for the testing of the epoxy nanocomposite specimen for its tensile strength. The tensile test was performed with a test speed of 5 mm/min and a preload of 0.1 MPa.

Fig. 3.4 UTM machine with specimen

3.3.2 Flexural Testing

The flexural test was done on UTM machine. Settings of specimen in the jaws of machine are shown in Fig.3.5. The flexural test was performed with the test speed of 1%/min and a preload of 0.1 MPa.

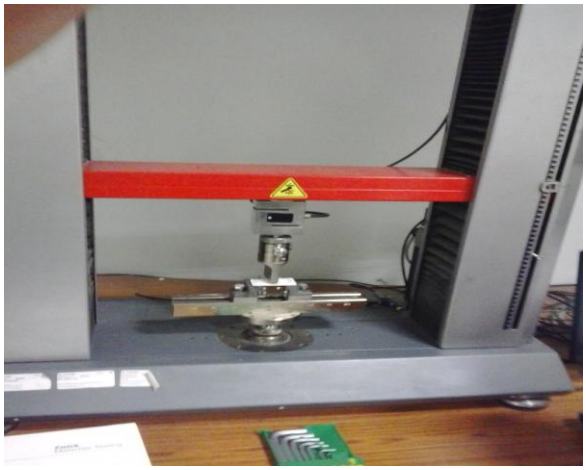

Fig. 3.5 Three point bend test

3.3.3 Micro Hardness Testing

Micro hardness test was conducted on prepared specimen using Micro Hardness Tester, shown in Fig. 3.6. VHN values were determined by applying 50 gm load by using a calibration distance of 50 units in Quantinet software which was used for image analyzing. 20 seconds dwell time was used during load application. After applying the load, an indent was formed on the specimen. These indents were formed on three random different places on the specimen. These indents were then used to calculate the average value of VHN. A view of one such indent is shown in Fig. 3.7.

Fig. 3.6 Micro hardness equipment

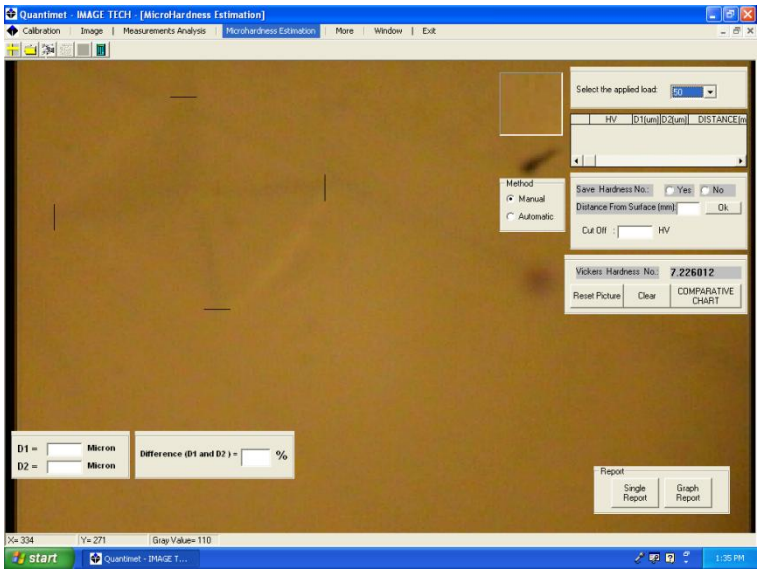

Fig. 3.7 Indent of Specimen

CHAPTER 4

RESULTS AND DISCUSSIONS

The different mechanical properties were tested on the prepared samples of epoxy with variation of TiO_2 content. The samples were prepared using the procedure discussed in chapter 3. Table 4.1 shows the number of samples prepared.

Table 4.1 Initial Testing Specimens

Specimen with wt% of TiO_2	**No. of Specimens**		**Total Specimens**
	Tensile Test	**Bending Test**	
0 wt% *i.e* Neat Epoxy	3	3	6
2 wt%	3	3	6
4 wt%	3	3	6
6 wt%	3	3	6
8 wt%	3	3	6
Total Specimens			30

4.1 TENSILE TEST

The test result, shown in Table 4.2 indicates that the tensile strength of specimen at 2 wt% of TiO_2 decreases as compare to neat epoxy.

Table 4.2 Results of Specimens from Tensile Test

Specimen	**Specimen No.**	**Tensile Strength (MPa)**
0 wt%	**Specimen No. 1**	9.24
	Specimen No. 2	8.97
	Specimen No. 3	8.70
2 wt%	**Specimen No. 1**	7.03
	Specimen No. 2	6.90
	Specimen No. 3	6.40

4 wt%	**Specimen No. 1**	24.0
	Specimen No. 2	8.90
	Specimen No. 3	29.8
6 wt%	**Specimen No. 1**	37.3
	Specimen No. 2	33.3
	Specimen No. 3	26.0
8 wt%	**Specimen no. 1**	35.7
	Specimen no. 2	31.5
	Specimen No. 3	33.2

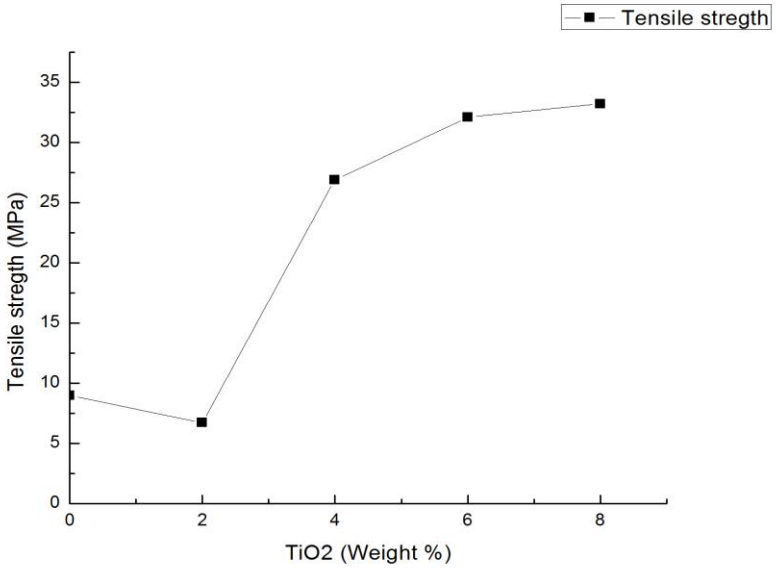

Fig 4.1 Tensile Strength of Specimens as a function of weight % of TiO_2 in epoxy.

An abrupt increase in tensile strength was seen from 2 wt% of TiO_2 to 4 wt% of TiO_2 and a gradual increase in tensile strength occurred from 4 wt% to extent of 8 wt% TiO_2. 270 % increase in tensile strength was obtained with addition of TiO_2. The increase in the strength of the Epoxy samples is due to the addition of TiO_2. Fig. 4.1 shows the variation in tensile strength plotted against weight percentage of TiO_2.

Fig. 4.2 shows the percent elongation at break point. It can be seen from the Fig. 4.2 that neat epoxy has the highest elongation and at 2 wt% of TiO_2 abrupt decrease in elongation occurs. Maximum elongation was seen only in 4 wt% of TiO_2.

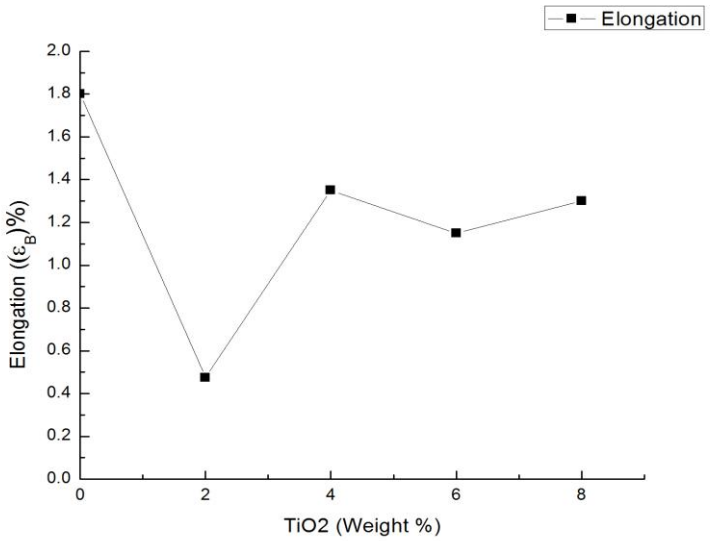

Fig 4.2 % age elongation as a function of weight % TiO_2 in epoxy

4.2 THREE POINT FLEXURAL TEST

Table 4.3 shows the flexural test observations which were done on UTM machine.

Table 4.3 Results of Specimens from Three-Point Bending Test

Specimen	Specimen No.	Flexural Strength (MPa)
0 wt%	**Specimen No. 1**	73.6
	Specimen No. 2	77.4
	Specimen No. 3	81.2
2 wt%	**Specimen No. 1**	11.2
	Specimen No. 2	12.2
	Specimen No. 3	10.2
4 wt%	**Specimen No. 1**	7.84
	Specimen No. 2	14.3
	Specimen No. 3	17.3
6 wt%	**Specimen No. 1**	29.0
	Specimen No. 2	9.83

	Specimen No. 3	23.6
8 wt%	**Specimen No. 1**	24.4
	Specimen No. 2	25.4
	Specimen No. 3	34.4

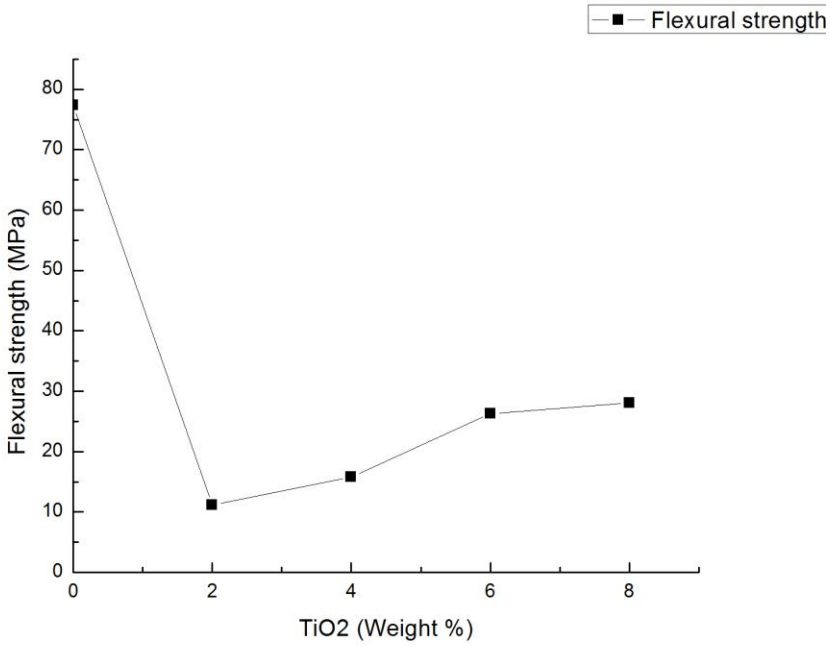

Fig 4.3 Flexural strength of Specimens as a function of weight % of TiO_2 in epoxy

As can be seen from Fig. 4.3, an enormous decrease in flexural strength is at 2 wt% of TiO_2. Further addition of TiO_2 to an extent of 8 wt% of TiO_2 shows a gradual increase in flexural strength but is 57% less than the neat epoxy.

4.3 MICRO HARDNESS TEST

The micro-hardness of specimen prepared at different TiO_2 loading was tested. Table 4.4 shows the experimental observations of the nanocomposites with different TiO_2 contents. An average hardness was calculated using three random indents in each specimen.

Table 4.4 Micro Hardness Values for Different TiO_2 Loading Specimens

TiO_2 Loading → / Loading Points ↓	Micro hardness values				
	0 wt%	**2wt%**	**4 wt%**	**6 wt%**	**8 wt%**
Point 1	6.675	6.019	7.759	11.835	10.972
Point 2	6.616	6.419	7.308	9.149	11.183
Point 3	6.884	6.019	6.884	13.311	13.416
Average	**6.718**	**6.201**	**7.430**	**10.717**	**12.522**

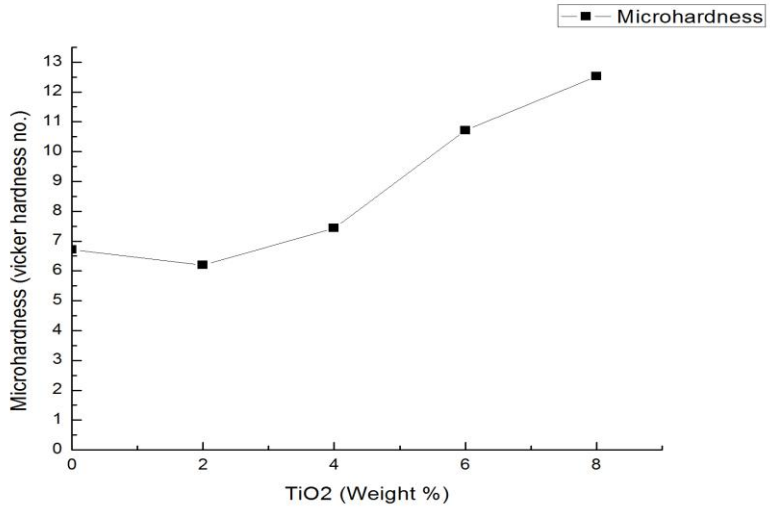

Fig. 4.4 Vickers' hardness value of specimen as a function of weight percentage TiO_2 in epoxy

As can be seen from Fig. 4.4, maximum value of hardness was measured at 8 wt% of TiO_2. There was a gradual increase in hardness from 2 wt% to 8 wt% of TiO_2. The hardness is around 86% higher than the neat epoxy.

4.4 OPTIMIZATION

As per the results of the Tensile Test, Three-Point Bending Test and Vicker's Hardness Test, the specimen with 8 wt% of TiO_2 gave the best results. Thereafter, the hybrid Nanocomposites were prepared with fixing 8 wt% of TiO_2 and adding SiO_2 in different compositions. The procedure

discussed in chapter 3 was followed for the preparation and testing of the samples. The compositions of SiO_2 were 2 wt%, 4 wt% and 6 wt%. Table 4.5 shows the number of samples prepared after optimization.

Table 4.5 Testing specimens after the Optimization

Specimen		**No. of specimens**		**Total specimens**
TiO_2	SiO_2	**Tensile Test**	**Bending Test**	
8 wt%	2 wt%	3	3	6
8 wt%	4 wt%	3	3	6
8 wt%	6 wt%	3	3	6
Total specimens				18

4.5 TENSILE TEST

The results obtained by re-conducting tensile tests on prepared Hybrid Nanocomposites on UTM are shown in Table 4.6.

Table 4.6 Results of Specimens from Tensile Test

Specimen		**Specimen No.**	**Tensile Strength(MPa)**
TiO_2	**SiO_2**		
8 wt%	**0 wt%**	**Specimen No. 1**	35.7
		Specimen No. 2	31.5
		Specimen No. 3	33.2
8 wt%	**2 wt%**	**Specimen No. 1**	3.58
		Specimen No. 2	2.43
		Specimen No. 3	2.70
8 wt%	**4 wt%**	**Specimen No. 1**	2.49
		Specimen No. 2	3.44
		Specimen No. 3	3.72
8 wt%	**6 wt%**	**Specimen no. 1**	1.66
		Specimen no. 2	1.83
		Specimen No. 3	1.58

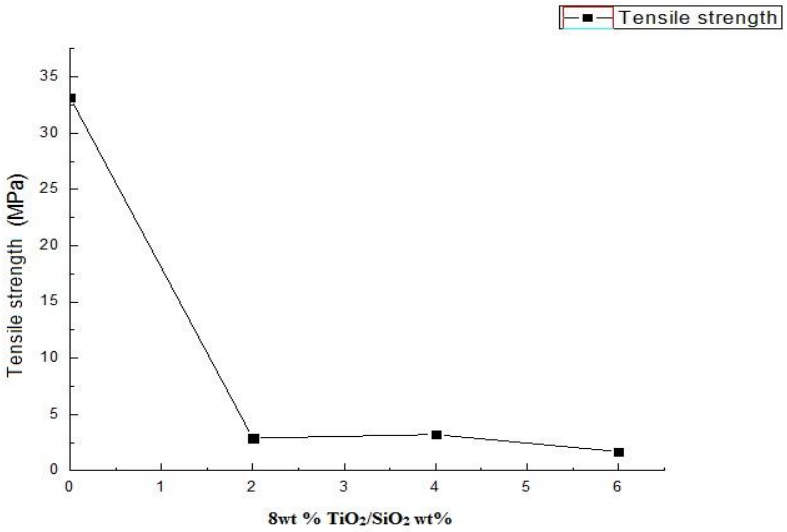

Fig. 4.5 Tensile strength of Specimen as a function of weight percentage of SiO_2 in 8 wt% TiO_2 Epoxy

After optimization of TiO_2 at 8 wt%, SiO_2 was added in 2 wt%, 4 wt% and 6 wt%. Fig. 4.5 shows that addition of SiO_2 decreases the tensile strength abruptly as compare to 8 wt% of TiO_2. Thereafter a very small increase in tensile strength occurred from 2 wt% to 4 wt% of SiO_2 and a decrease at 6 wt% of SiO_2. The results obtained with further addition of SiO_2 were not significant.

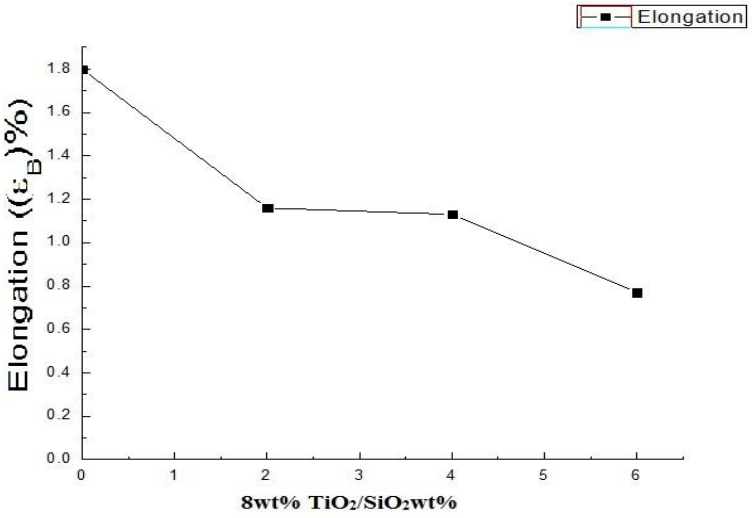

Fig 4.6 % age elongation as a function of weight % of SiO_2 in 8 wt% TiO_2 epoxy

Fig. 4.6 shows that the maximum elongation was at 2 wt% of SiO_2 which was less than the neat epoxy composite.

4.6 THREE POINT FLEXURAL TEST

The results obtained by re-conducting Three Point Flexural tests on prepared Hybrid Nanocomposites are shown in Table 4.7.

Table 4.7 Results of Specimens from Three-Point Bending Test

Specimen		Specimen No.	Flexural Strength (MPa)
TiO_2	SiO_2		
8 wt%	**0 wt%**	**Specimen No. 1**	24.4
		Specimen No. 2	25.4
		Specimen No. 3	34.4
8 wt%	**2 wt%**	**Specimen No. 1**	27.5
		Specimen No. 2	23.1
		Specimen No. 3	28.9
8 wt%	**4 wt%**	**Specimen No. 1**	8.44
		Specimen No. 2	13.4
		Specimen No. 3	14.6
8 wt%	**6 wt%**	**Specimen No. 1**	6.59
		Specimen No. 2	8.02
		Specimen No. 3	7.72

After optimization of TiO_2 at 8 wt%, SiO_2 was added 2 wt%, 4 wt% and 6 wt%. Table 4.7 shows the flexural test observations. As can be seen from Fig. 4.7, maximum flexural strength was found at 2 wt% of SiO_2 and thereafter it had a decreasing trend from 2 wt% to 6 wt% of SiO_2. Decrease in flexural strength shows decrease in interfacial properties. It can be due to increase in size of nanoparticles.

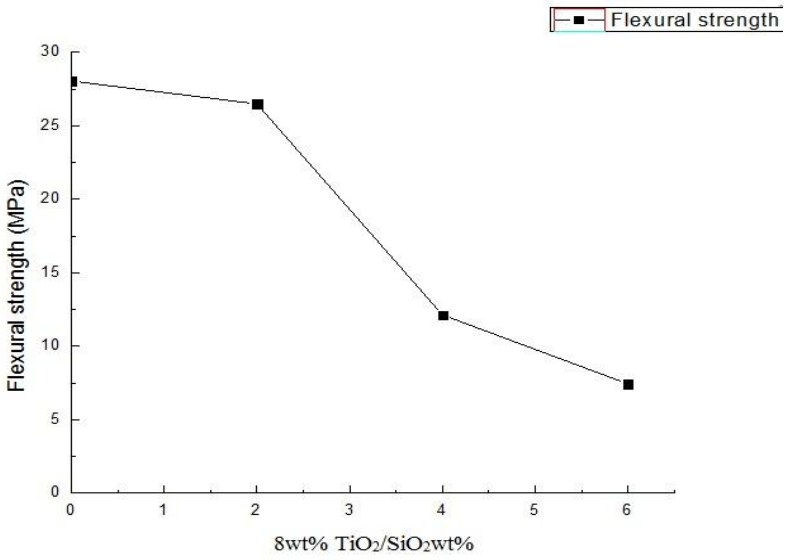

Fig. 4.7 Flexural Strength of samples as a function of weight percentage of SiO_2 in 8wt% TiO_2 Epoxy

4.7 MICRO HARDNESS TEST

The micro hardness was again tested at different points on the prepared hybrid nanocomposites. Table 4.8 shows the hardness value for different specimen.

Table 4.8 Micro Hardness Values for Different SiO_2 Loading Specimens

Clay/TiO_2 Contents ⇒ / Loading Points ⇓		**Micro Hardness Values**			
	TiO_2	**8 wt%**	**8 wt%**	**8 wt%**	**8 wt%**
	SiO_2	**0 wt%**	**2 wt%**	**4 wt%**	**6 wt%**
Point 1		10.972	9.083	7.428	6.034
Point 2		11.183	9.238	7.778	6.134

Point 3	13.416	9.126	7.628	6.004
Average	**12.522**	**9.149**	**7.611**	**6.057**

The variation of Micro Hardness as a function of weight % of SO_2 in 8 wt% of TiO_2 was shown in Fig. 4.8. It can be seen from the figure that the micro hardness value decreased from 2 wt% to 6 wt% of SiO_2.

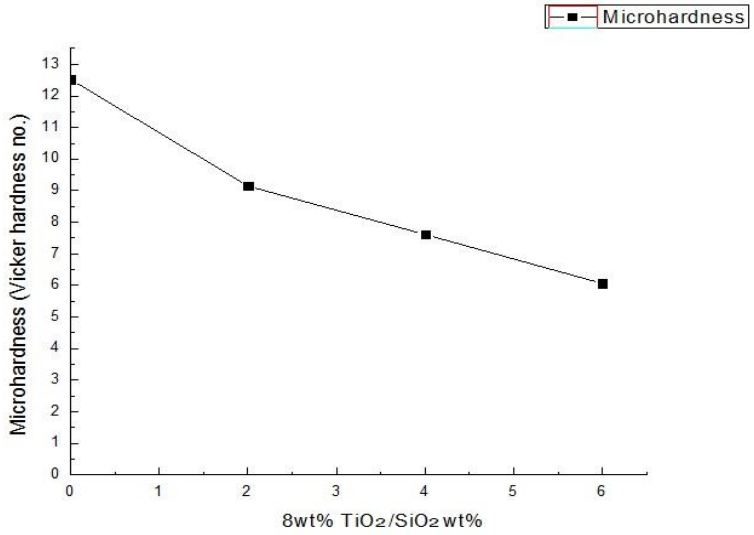

Fig. 4.8 Vickers' Hardness value of specimen as a function of weight percentage of SiO_2 in 8wt% TiO_2 Epoxy

CHAPTER 5
CONCLUSIONS

5.1 CONCLUSIONS

Hybrid nanocomposite had been manufactured with in situ polymerization. TiO_2, SiO_2 were used as nano filler in nanocomposite. For proper dispersion of nano filler in epoxy system Mechanical stirring and ultrasonication was done. Hand lay-up technique was used to manufacture hybrid nanocomposites. Best tensile, flexural and hardness properties were seen at 8 wt% of TiO_2. Thereafter a hybrid nanocomposite was prepared with fixing TiO_2 at 8 wt% and adding SiO_2 in 2 wt%, 4 wt% and 6 wt%. Following conclusions are drawn from the present work:

(i) Increase in tensile strength of nanocomposite was seen with increasing the wt% of TiO_2, with maximum at 8 wt% of TiO_2. Gradual increase in strength was found from 2 wt% to 8 wt% of TiO_2. This strength is around 270% higher than the neat epoxy nano composite. After optimization, SiO_2 in different concentration is added into 8 wt% of TiO_2. With addition of SiO_2 decrease in strength was found.

(ii) Enormous decrease in flexural strength of nano composite was found with addition of TiO_2 in epoxy. 57% decrease in flexural strength had seen from neat epoxy to 8 wt% of TiO_2. After optimization, SiO_2 again degraded the flexural strength of prepared nano composite.

(iii) Vicker hardness value increases with increase in the TiO_2 concentration in epoxy. The specimens with 8 wt% of TiO_2 had the maximum hardness. Gradual increase in hardness was found from neat epoxy to 8 wt% of TiO_2. Addition of SiO_2 in the TiO_2 modified epoxy composite, it was seen that the hardness increase a bit with addition of 2 wt% of SiO_2 but thereafter the harness decreases with increase in the composition of SiO_2.

5.2 FUTURE SCOPE

The present work can be extended in following ways:

(i) Different nano filler can be used.
(ii) Other preparation technique can be used for the nanocomposite
(iii) Hygrothermal study can be carried on these nanocomposites.
(iv) Fiber can be used for reinforcing the nanocomposite.

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PUBLICATIONS

1. **Parvesh Kumar, Toyesh Upreti, Jaswinder Singh Saini, Haripada Bhunia,** Investigation on Mechanical Properties of Nano-TiO_2/ Nano-SiO_2/ epoxy hybrid nanocomposites**,**International Conference on Nanotechnology (ICNT 2013), to be held on 25-26^{th} October, 2013 at Haldia Institute of Technology, Haldia, Purba Medinipur, West Bengal, INDIA (accepted).