Effect of silane functionalization on mechanical properties of carbon fiber reinforced composites

A Dissertation

Submitted in partial fulfilment of the requirement for the award of degree of

Master of Engineering
in
CAD/CAM Engineering

Submitted

by

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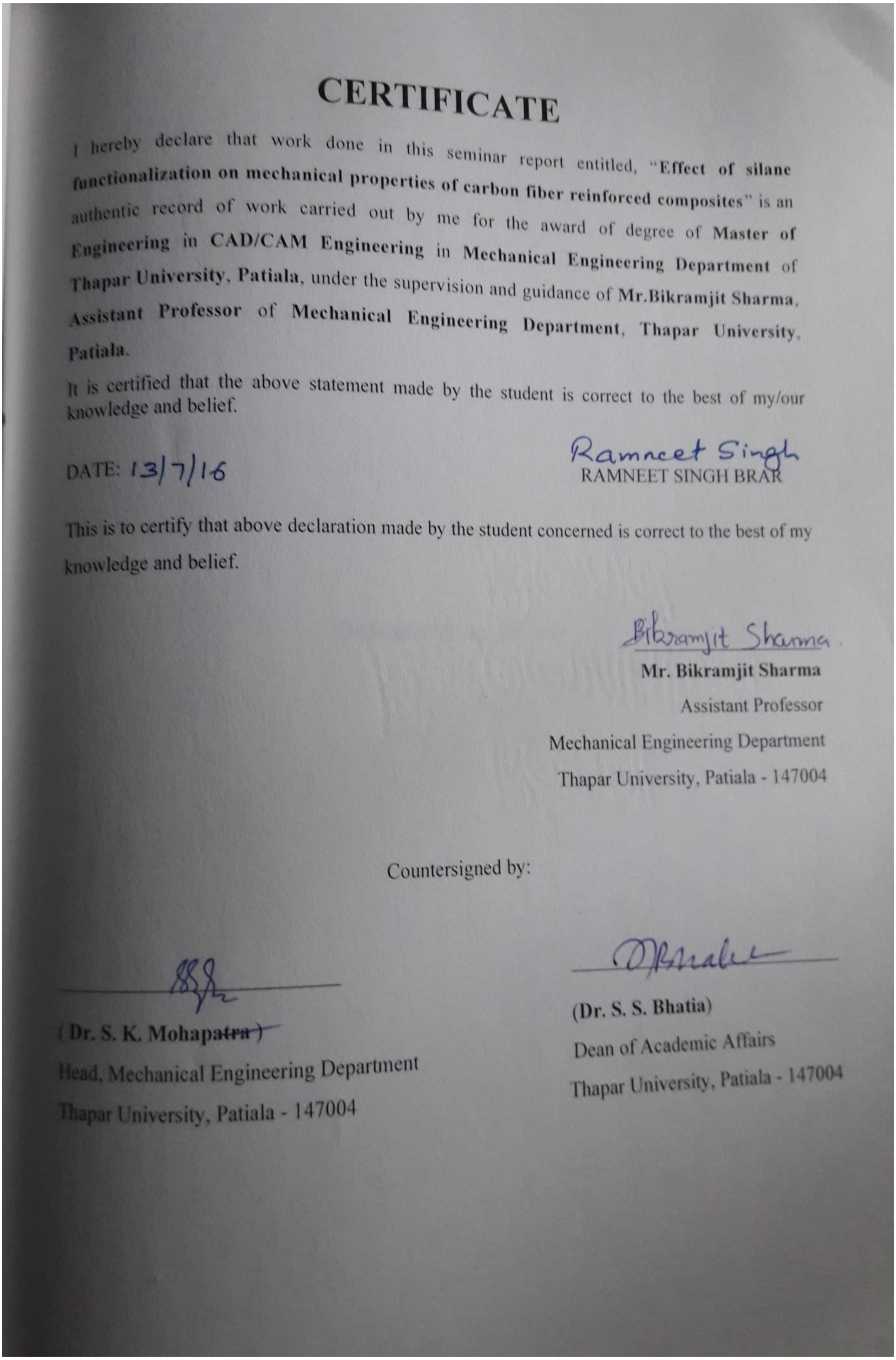

CERTIFICATE

I hereby declare that work done in this seminar report entitled, "**Effect of silane functionalization on mechanical properties of carbon fiber reinforced composites**" is an authentic record of work carried out by me for the award of degree of **Master of Engineering** in **CAD/CAM Engineering** in **Mechanical Engineering Department** of **Thapar University, Patiala**, under the supervision and guidance of **Mr.Bikramjit Sharma, Assistant Professor** of **Mechanical Engineering Department, Thapar University, Patiala.**

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

DATE: 13/7/16

Ramneet Singh
RAMNEET SINGH BRAR

This is to certify that above declaration made by the student concerned is correct to the best of my knowledge and belief.

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Dedicated to my parents

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Above all, I express my sincere thanks to " ALMIGHTY" for the blessings.

RAMNEET SINGH BRAR

ABSTRACT

In this work, unidirectional carbon fibers are treated with silane coupling agents and the effect of surface treatment on mechanical properties of fiber reinforced epoxy composites was studied. The fiber surface modification was carried out using wet method. The silane functional groups and concentration of the silane influence the grafting of silane as confirmed by FTIR studies. The effect of reaction time and change in silane concentration influenced the mechanical properties of the composites. The composite containing the carbon fiber treated with 3-Amino propyl triethoxy silane at 1.5% concentration (v/v) with treatment time 30 minutes possess the best mechanical properties with an increase of 33.6%, 32.38%, 33.72% in flexural modulus, tensile modulus and inter laminar shear strength respectively from the neat CFRC. The flexural and ILSS properties with treatment time 30 minutes increase in following order: Neat CFRC< GPS(1.5)< GPS(2.5)< APS(2.5)< APS(1)< GPS(1)< APS(1.5). The tensile properties with treatment time 30 minutes increase in the following order: Neat CFRC< GPS(1.5)< APS(2.5)< APS(1)< GPS(2.5)< GPS(1)< APS(1.5). Finite element analysis is done using ANSYS 15.0 software comparing the experimental deformation of the tensile tested samples and the simulated deformation. Results indicated that the experimental values match reasonably validating the experimental results.

TABLE OF CONTENTS

List of Figures

List of Equation

List of Tables

LIST OF ABBREVATIONS

CF	Carbon Fiber
CFRC	Carbon Fiber reinforced composite
GFRC	Glass Fiber reinforced composite
FTIR	Fourier transform infrared spectroscopy
NCF	Neat carbon fiber
SEM	Scanning electron microscope
APS	3-Amino propyl triethoxy silane
GPS	3-Glycidyloxypropyl trimethoxy silane
ILSS	Inter laminar shear strength
UTM	Universal testing machine
DGEBA	Di glycidyl ether of bisphenol A
FEA	Finite element analysis
APS(1)	1% v/v concentration treated fiber
APS(1.5)	1.5% v/v concentration APS treated fiber
APS(2.5)	2.5% v/v concentration APS treated fiber
GPS(1)	1% v/v concentration GPS treated fiber
GPS(1.5)	1.5% v/v concentration GPS treated fiber
GPS(2.5)	2.5% v/v concentration GPS treated fiber
APS30(1)	1% v/v concentration APS treated fiber for 30 minutes
APS30(1.5)	1.5% v/v concentration APS treated fiber for 30 minutes
APS40(1.5)	1.5% v/v concentration APS treated fiber for 40 minutes
GPS30(1)	1% v/v concentration GPS treated fiber for 30 minutes
GPS30(1.5)	1.5% v/v concentration GPS treated fiber for 30 minutes

Chapter 1

Introduction

1.1 Fiber reinforced composites

Fiber reinforced composites materials are developed with combination of high strength and modulus embedded in or bonded to a matrix with an interface different in chemical composition (P.K. Mallick 2007). Composite materials consist of three constituents:

- Matrix (primary phase)
- Reinforcement (secondary phase)
- Interface

In general matrix plays a supporting role in a composite which keeps them in desired location and orientation and acts as a load transfer medium between them. Fiber reinforced composites are most commonly prepared by stacking layer by layer fibers and the matrix producing a laminate. The size of a lamina varies from 0.1-1mm.

1.2 General characteristics

Fiber reinforced polymers are so good in strength and modulus that they are comparable and many a times better than the traditional metallic materials. Properties such as low density, strength to weight ratio and modulus weight ratio makes them better than the metallic materials used. Fatigue strength and fatigue damage tolerance are also exceptionally good of certain composites. Traditional structured material were isotropic they produce equal properties in any direction of measurement whereas FRC are not isotropic as for unidirectional composites the properties are good when measured along the longitudinal direction of the fiber (Kaushik & Singh 2010).

1.3 Fibers most commonly used

Fibers are chief constituent in composite materials as they carry a massive majority of the load on a composite structure. The choice of fiber depend on the application of composite as it affects various characteristics of composite such as specific gravity, tensile modulus, fatigue strength and others.

1.3.1 Glass fibers

Glass fiber is most commonly used fiber because of its low cost, high tensile strength, high chemical resistance and excellent insulating properties. Modulus tensile modulus and high density (comparing with other commercial fibers) relatively low fatigue resistance, and high hardness (which makes molding dies and cutting tools wear out) are the disadvantages with the glass fiber. The tensile strength of neat glass fiber is nearly 2 GPa and may go to 3 GPa if there are no manufacturing defects. Commonly used glass fiber in polymer industry is continuous strand roving, Woven roving Chopped strands, Chopped strand mat, Woven roving mat (P.K. Mallick 2007).

1.3.2 Boron fibers

Boron fibers have a very high tensile strength varying from 379–414 GPa. Boron carbons possess a very good resistance to buckling which gives a very high compressive strength to boron reinforced polymers. Following such high properties boron fibers are not very commonly used because of their very high cost. The raw material used in manufacturing of boron fibers is tungsten wire.

1.3.3 Ceramic Fibers

Most commonly used ceramic fibers are Silicon carbide (SiC) and aluminium oxide (Al_2O_3) for they can bear high temperatures having melting points of 2830^oC and 2045^oC respectively. Silicon carbide fibers availability is in three different forms monofilament SiC, multifilament SiC and whiskers. Another ceramic fiber available is the combination of silicon carbide and aluminium oxide.

1.3.4 Natural Fibers

Natural fibers commonly used are flax, hemp, jute, remi, sisal, coconut fiber and banana fiber. Natural fibers are grown all over the world in agricultural fields and find their application in making ropes, bags, carpets and many more. Natural fibers constituents are cellulose, lignin and hemicellulose. Natural fibers are giving a very tough competition in automotive industry to the other fibers as they are environmentally friendly (Biodegradable), the density of natural fibers is less as compared to glass and carbon fibers, the modulus to weight ratio of some natural fibers is better than the e-glass fibers, and more importantly they are less expensive than all the other fibers.

1.4 Matrix

In general matrix plays a supporting role in a composite which keeps them in desired location and orientation and acts as a load transfer medium between them (distributes stresses between the fibers) and also prevents the environment damages like temperature. The matrix also helps in preventing buckling under compressive loading (Kaynak et al. 2005).

1.4.1 Polymeric

A. Thermoplastic polymers

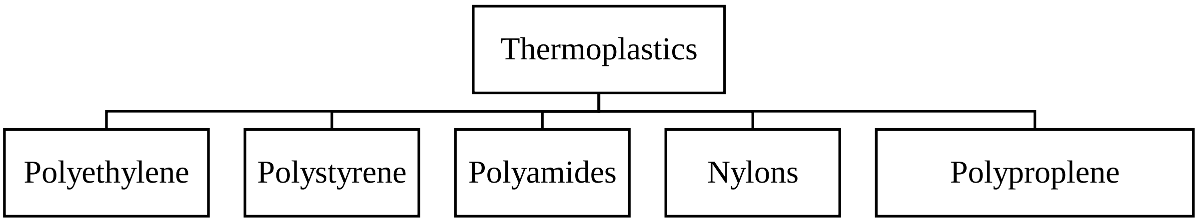


Figure 1.1 : Thermoplastic polymers

B. Thermoset polymers

- Epoxies: used in aerospace industry.
- Polyesters, vinyl esters: used in electrical, chemical and automobile industries

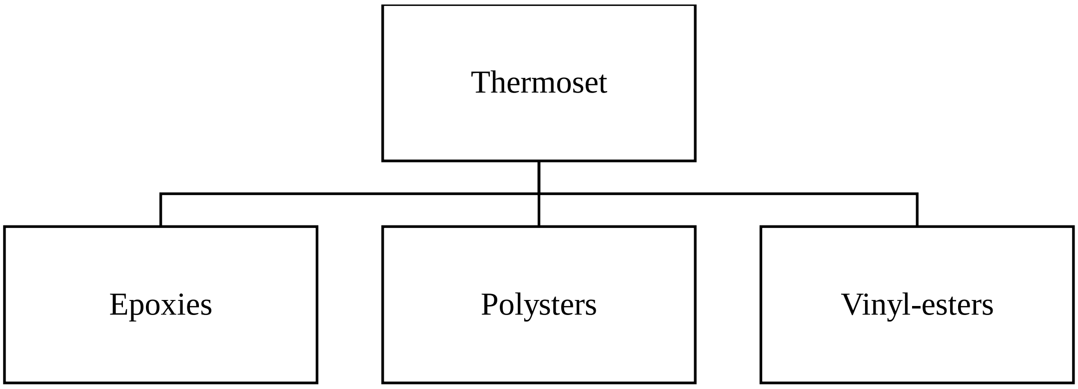


Figure 1.2 : Thermoset polymers

- Epoxy

When an epoxy resin and a curing agent react epoxies are produced. Di-glycidyl ether of bisphenol A (DGEBA) is the material on which majority of epoxy resins are based.

This is represents the smallest type of epoxy resin. The curing agents usually hardeners are used to produce epoxies. Epoxy resins can have variable chain lengths and properties depending on their molecular weight (Wang et al. 2015).

1.4.2 Metallic

Aluminum and its alloys, titanium alloys, magnesium alloys and stainless steel.

1.4.3 Ceramic

Aluminum oxide, silicon carbide, silicon nitride.

1.5 Defects in composites

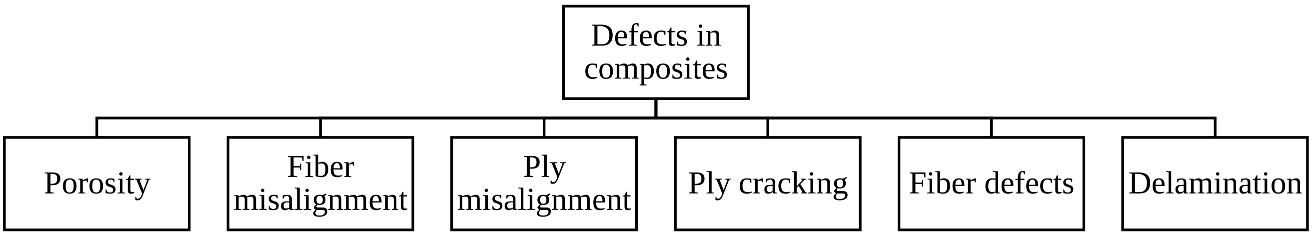


Figure 1.3 : Deflection in composites (Dai et al. 2011)

- **Porosity** It depicts the presence of small voids in the matrix. This defect occurs due to the presence of volatile component or when air gets trapped during the curing process. Porosity levels directly effects the mechanical properties of composites.

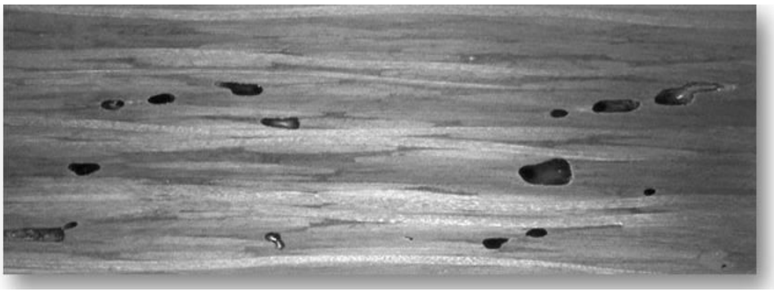


Figure 1.4 : Porosity defect

- **Fiber misalignment** This defect produces uneven stress distribution which will have a significant change in test results.
- **Ply misalignment** Ply misalignment is also a very crucial defect that is produced in composites and affects the test results to a large extent.
- **Ply cracking** Due to differential contraction thermal cracks are originated in the composites and defect is named as ply cracking.
- **Fiber defects** When the defect is present in fiber, it can adversely affect the strength and other properties of the composite.

- **Delamination** When a composite is subjected to repeated stress it can separate the layers which results in overall low mechanical properties

1.6 Advantages of composite material

a) Excellent specific strength and specific stiffness characteristics
b) Easy machinability
c) High resistance to corrosion, chemical degradation and other environmental conditions
d) Low coefficient of thermal expansion
e) Design Flexibility.

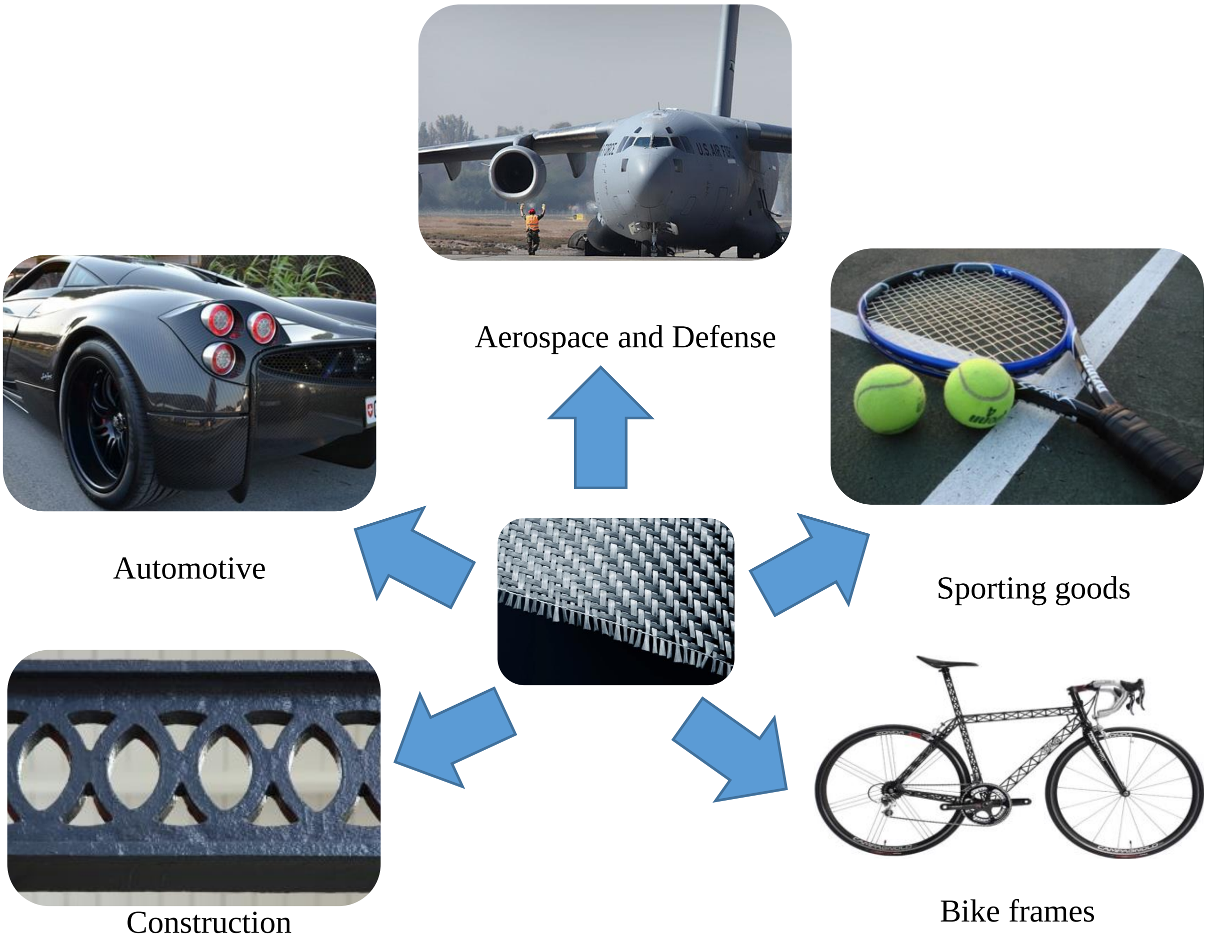


Figure 1.5 : Application of composites

1.7 Isotropic, Anisotropic, and Orthotropic Materials

If the properties of a material are same in all the directions than it is an isotropic material. If the properties are different in all the directions and there are no planes of material property symmetry than the material is an anisotropic material. Materials with varying properties in all directions and made up of several planes of material property symmetry which ultimately have large impact on its properties are the orthotropic materials. Fiber reinforced polymers are a type of orthotropic material. The dependent variables are E_{11}, E_{22}, E_{33}, G_{12}, G_{13}, G_{23}, ν_{12}, ν_{13}, and ν_{23}.But in case of unidirectional oriented fiber composites are a special case of isotropic materials because it can be depicted the elastic properties are equal in the second and third direction which makes $E_{22}= E_{33}$,$\nu_{12}=\nu_{13}$, and $G_{12}=G_{13}$.

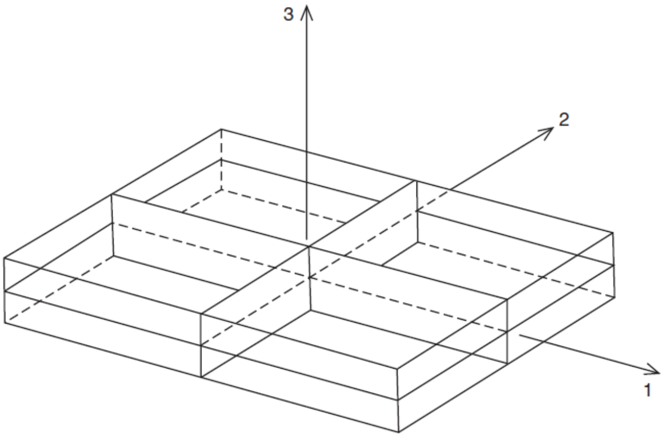


Figure 1.6 : Three planes of symmetry in an orthotropic material (P.K. Mallick 2007).

1.8 Carbon Fibers

Carbon atoms bonded together in crystals constitutes a carbon fiber. Several fibers bundled together form a tow, which are woven to fabric and made available to the industries. Amorphous and graphitic carbon constitutes a carbon fiber. Carbon fibers are arranged in in crystallographic form of parallel plane which gives the fibers a very high tensile modulus. In the same plane the atoms are bonded with strong covalent bonds whereas the bond between the planes is much weaker and is due to the Vander wall forces. Carbon Fibers are available in three forms:

1. Long and continuous tow

2. Chopped (6-50 mm long)
3. Milled (30-3000μm long)

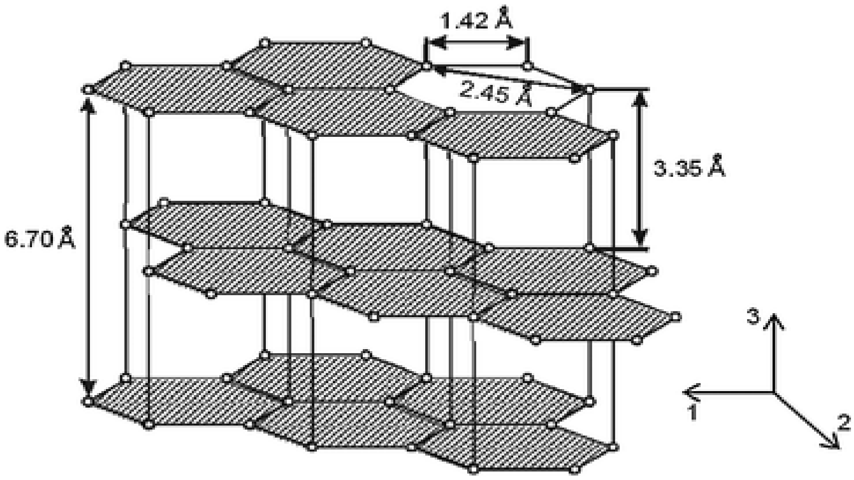


Figure 1.7 : Carbon fiber structure (Yuan et al. 2012)

Advantages of carbon fiber

- High stiffness
- High tensile strength
- Low weight
- High chemical resistance
- High thermal expansion

1.9 Chemical treatment of carbon fibers

The basic purpose of surface treatment is to improve the fiber matrix interface bonding. The surface treatment of carbon fiber can be classified in two categories.

1.9.1 Oxidative treatments

a) Dry oxidation
b) Plasma etching
c) Wet oxidation
d) Anodic oxidation

- **Dry Oxidation**

Dry oxidation treatment process takes place with air and oxygen or using any other gases like O_3 and CO_2 along with oxygen. Fibers should be treated at low temperatures in dry oxidation treatment method as high temperatures reduce fiber tensile strengths.

- **Plasma Etching**

Plasma etching treatment is very commonly used to increase the fiber matrix adhesion as in this method formation of electrons, ions, ionized atoms is initialized which leads to abstraction of material from surface. Oxygen and fluorine are most commonly used gases in this process. The plasma is initiated by directing high energy beam to gases or air. Plasmas commonly used are thermal plasma, glow plasma and corona discharge.

- **Wet oxidation**

Wet oxidation makes use oxidizing agents like nitric acid, acidic potassium permanganate, acidic potassium dichromate, dichromate permanganate, hydrogen peroxide, ammonium bicarbonate which are in liquid form. This treatment is milder in comparison to dry oxidation so produces better tensile properties.

- **Electrolytic or Anodic oxidation**

Anodic oxidation is fast and uniform type of fiber treatment process. In Anodic oxidation material is dissolved in the electrolyte of solution which is alkaline in nature during which carbon dioxide is formed and degradation of product occur simultaneously. The main advantage of this method is that fibers produced are with nominal weight loss (Choi et al. 2016).

1.9.2 Non oxidative treatment

Commonly used non oxidative treatment processes are whiskerization and Plasma polymerization. Whiskerization is a nucleation process in which high strength single crystals of chemical compound get developed on the fiber surface in perpendicular direction. Chemical compounds usually used are silicon carbide and titanium dioxide. In Plasma polymerization process the surface polar free energy of carbon fiber is surged. This process includes polymerizable organic vapors like, organosilanes, polyamide and propylene

1.10 Silane coupling agents

Silane coupling agents are compounds whose molecules contain functional groups that can bond with organic as well as inorganic materials. A silane coupling agent acts as a sort of intermediary which bonds organic materials to inorganic materials. This is the reason that the silane coupling agents improve the mechanical strength of composite materials, adhesion, resin modification and surface modification (Fu et al. 2012).

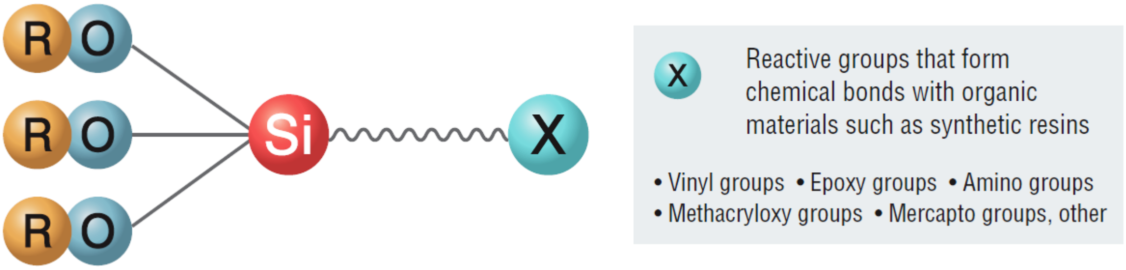


Figure 1.8 : General formula for the silane coupling agent (Plueddemann et al. 2014)

Mechanism Process of action on silane coupling agent:

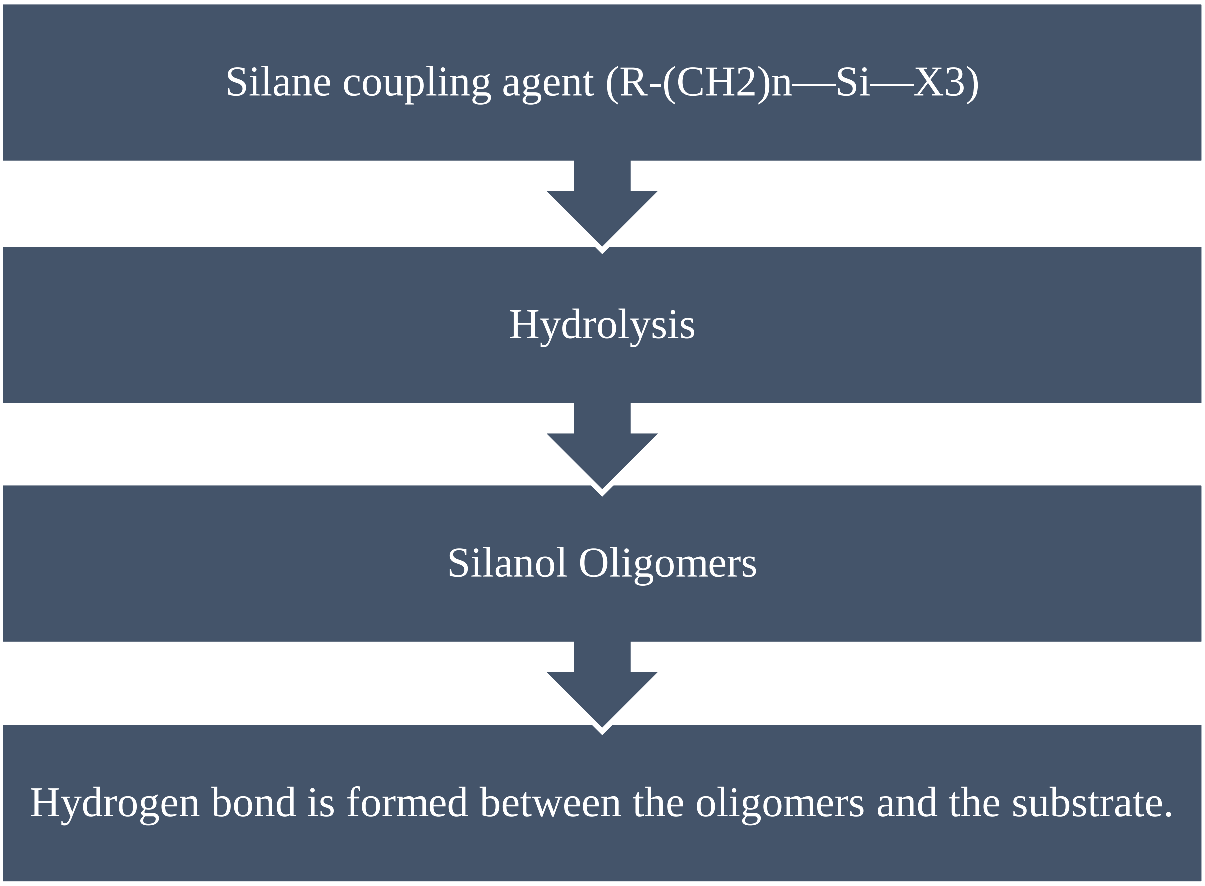


Figure 1.9 : Mechanism process of action on silane coupling agent

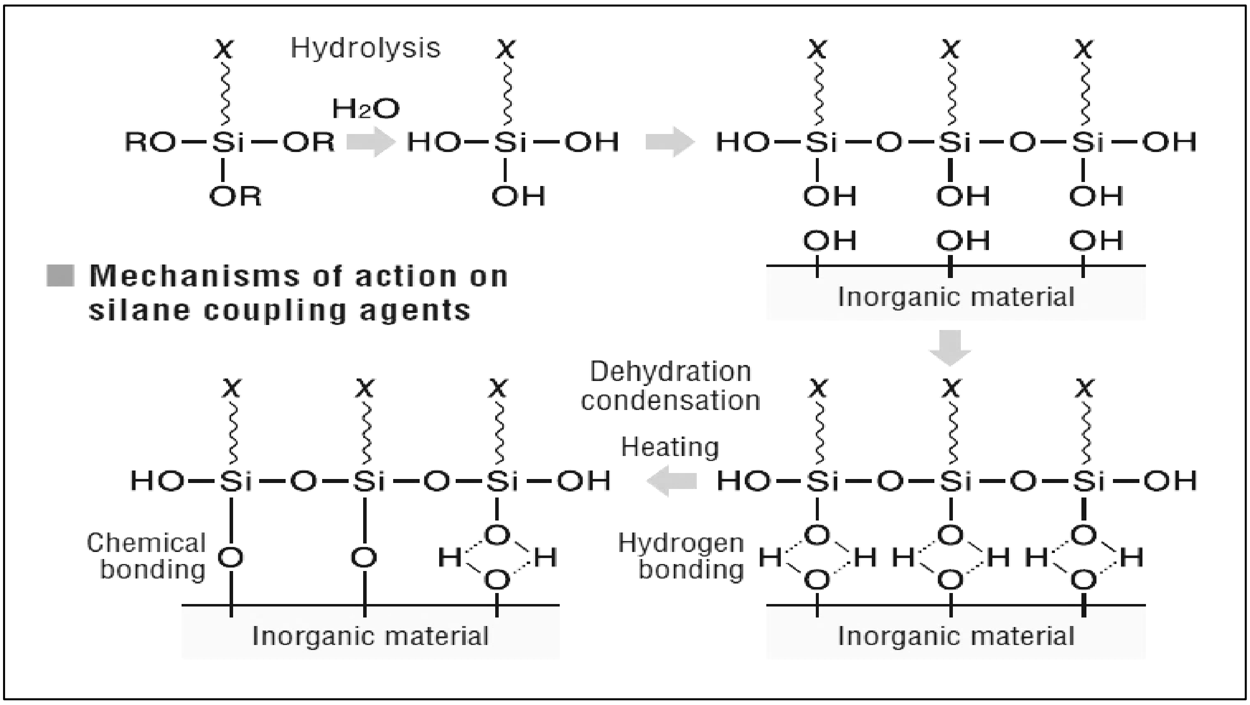


Figure 1.10 : Mechanisms of action on silane coupling agent

1.11 Mechanical Properties

1.11.1 Tensile testing

Tensile testing is done on the samples by applying tensile force on them until their failure. Results of tensile testing are very important as material selection is done for its correlated application. Various important properties ranging from stresses, strains, Young’s modulus, elongation of sample at brake are obtained. Tensile testing is usually done on universal testing machine. For tensile testing UTM consists of two crossheads out of which one is adjusted according to length of the specimen and then fixed and the other crosshead is driven by machine itself which applies the tension on the specimen.

1.11.2 Flexural testing

Flexural testing is defined as the ability of brittle material to resist bending, when a force is applied (Patel et al. 2015). Flexural testing is also called transverse bending test and samples tested have a rectangular shape which are applied force till fracture or circular shape which are twisted until rupture. Flexural testing provides us flexural modulus, bending strength, interlaminar shear strength. Three point flexural test is commonly used for flexural testing of composite samples. Universal testing machine is used for the three point bending test. For flexural testing the specimen is supported on two precision machined (free to rotate or stationary) anvils of a defined radius. The force is applied centrally in the 3 point flexural test.

Figure 1.11 : Universal Testing Machine

(Photo courtesy: Chemical Engineering Department, Thapar University, Patiala)

1.12 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR is a technique which is used to get an infrared spectrum of absorption or emission of a solid, liquid or gas. It gives the complete information about the molecular structure of the sample. A spectrum is obtained on analysis representing the molecular absorption and transmittance. An infrared spectrum gives us the information about the absorption peaks which are directly related to the frequencies of the vibration of the atomic bonds (Lee et al. 2015).

Figure 1.12 : FTIR Spectrometer

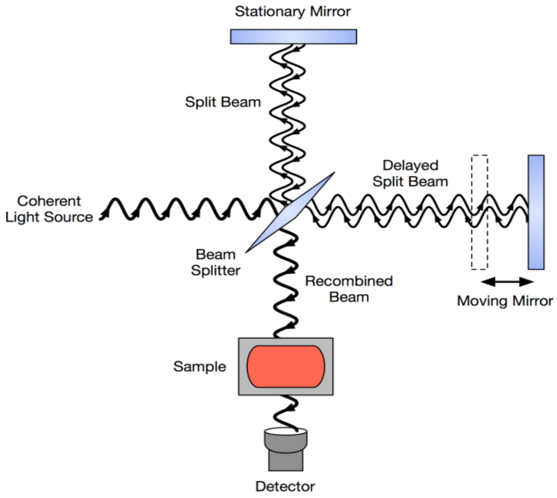


Figure 1.13 : FTIR Spectrometer working process(Gillie et al. 2000)

1.13 Vacuum assisted resin infusion molding (VARIM)

Vacuum assisted resin infusion molding is a composite manufacturing technique in which the resin flows with the help of vacuum and the mould is covered by a plastic vacuum bag (Yang et al. 2013). Release fabrics, peel ply, the perforated film and the infusion mesh are directly laid on the fiber preform and then sealed using vacuum bagging. The air is evacuated in this process as sealant tapes are used on all sides of the bagging to maintain complete vacuum. The liquid resin flows into the fibers from its source by the vacuum drawn through the mould with the help of a breather cloth. The laminates are kept under the vacuum and a desired temperature is maintained for the complete curing process. For the purpose of heating the table electrical heating resistances are used and thermocouples are attached under the table for maintaining the desired temperature (Yang et al. 2013).

Figure 1.12 : Vacuum assisted resin infusion molding machine with on mould heating system

(Photo courtesy: Chemical Engineering Department, Thapar University, Patiala)

1.14 Finite element analysis (FEA)

Finite element method is a practical application of finite element method which the engineers use to solve problems which are very complex related to structural, fluidic, and multi physics problems where finite element method (FEM) is a numerical method in which a solid is modeled in computer and analyzed for obtaining various results. A finite element model is made up of small points known as nodes which collectively constitute a model that is used for designing (A & Mohanty 2015). These nodes form a finite element mesh of the finite elements. The size and fineness of the mesh totally depends upon the number of the elements being used for defining the design model. FEA is done in three stages:

1. Preprocessing
2. Processing
3. Post processing

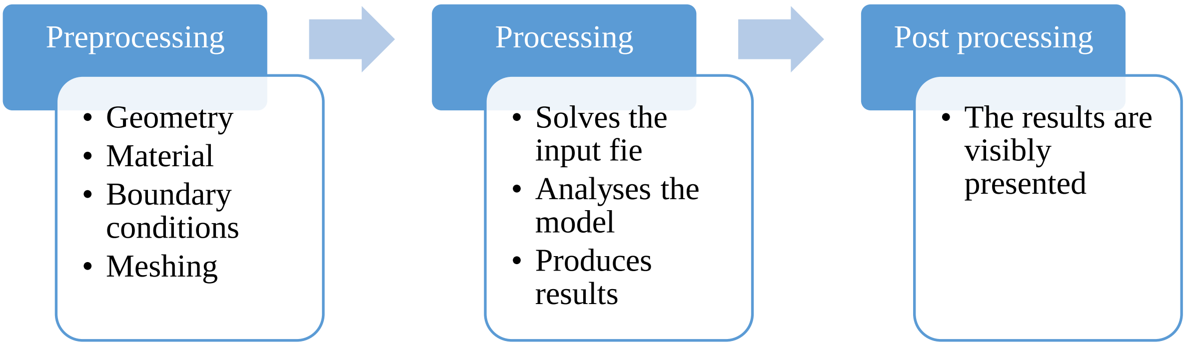


Figure 1.13 : Finite element analysis process

Chapter 2

Literature review

Donnet *et al.* (1996) studied the effect of plasma treatment on the T300 carbon fibers for (2-30 min.) and compared the mechanical properties of the treated carbon fibers to the neat carbon fibers. Results indicated a decrease of 8% in the tensile breaking load of CFs. After the plasma treatment process oxygen content on the carbon fiber surface increased, presence of polar compounds was found on the surface. Along with these changes on the surface considerable etching with deep grooves was observed.

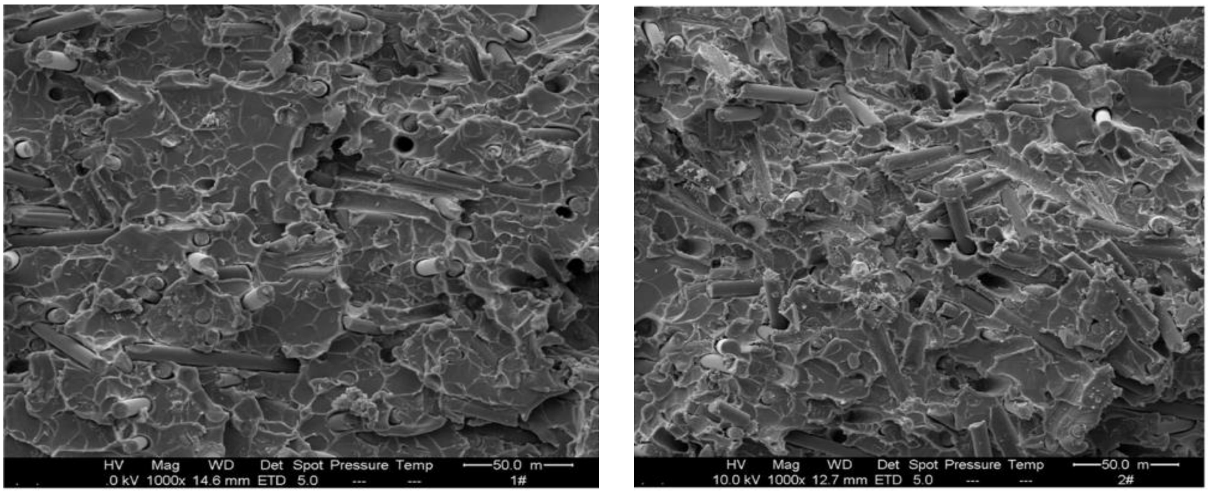
Zielke *et al.* (1996) studied surface oxidized carbon fibers, their surface structure and chemistry.The study was carried out by using various techniques like scanning electron microscope, scanning tunneling microscope, cantact angle measurement. Results suggested that carbons fibers received from the producers are adulterated by absorbed oxidation products. For proper surface study of carbon fibers removal of every oxidation product is very important. The removal process is carried out by extraction with water and post drying process without any change in surface chemistry.

Xu. *et al.* (1999) surface treated carbon fibers with O_3, an aqueous solution of ($K_2Cr_2O_7$, 30 wt.%) and (H_2SO_4, 40 wt.%) and silane coupling agents mixture of Z-6020 and Z-6040. Cement reinforced carbon composite prepared with silane treated carbon fiber produced 56% higher tensile strength and 39% higher tensile modulus in comparison with the untreated carbon fibers. Also a 26% higher flexural strength and 14% higher flexural modulus were obtained after the treatment process.

Choi *et al.* (2004) studied the effect of silane coupling agent on electrical and mechanical properties on CFRC. The coupling agent used is glutaric dialdehyde. The coupling agent increased electrical as well as mechanical properties. Dielectric behaviour was also affected of short carbon fibers which were silane treated. The SEM images clearly show the there were no holes on the fractured surfaces of silane treated composite which suggests that the coupling agent treatment increases adhesion between the fiber and resin The SEM results and improved mechanical properties after silane treatment suggested that the interfacial adhesion was improved between the fiber and the matrix due of the formation of chemical bonds between the fiber and the resin..

Xu *et al.* (2007) studied the effect of using acrylic acid for finding out the wetting and adsorption ability of carbon fiber with epoxy. Various reduction-oxidation and pre irradiation methods were used to find out the results. Also the interfacial properties of the CFRC were studied. Various parameters of carbon fibers like fiber surface structure, surface energy, chemical composition, ability to absorb were studied. Tensile test were also done on the treated CFRC. Results indicated increase of roughness on the treated fiber surface as compared to untreated carbon fiber. Tensile strength improved by pre-irradiation process whereas there was a little decrease in the oxidized and reduced carbon fibers but the combined treatment of pre-irradiation and oxidation-reduction processes resulted in a 15% increase in the inter laminar shear strength of the carbon epoxy composites. So the both methods pre-irradiation and oxidation-reduction processes were considered good surface treatment methods.

Li *et al.* (2010) studied the effect of silane coupling agent on tensile properties of carbon fiber reinforced composites as well as reported the improvement in interfacial adhesion of matrix and the fiber by comparing SEM images. Carbon fiber was treated with different concentrations varying from 0.1% to 0.5% wt. and found out increase of tensile strength as the silane concentrations increased. The SEM images comparison of fractured surfaces between the untreated fiber and silane treated fiber showed that the surface at fracture site was flat and the fibers were broken along with matrix without leaving holes, which showed that improvement in adhesion between the matrix and the fiber.



Untreated carbon fibers (Li 2010)

Silane treated carbon fibers (Li 2010)

Figure 2.1 : Scanning electron micrographs

Dai *et al.* (2011) studied the sizing effect on the surface properties of CF and the fiber/matrix interfacial adhesion. The study was done by comparing sized and desized T300B and T700SC carbon fibers. The surface morphological investigation using X-ray photoelectron spectroscopy (XPS) showed that the activated carbon concentration was very less in desized carbon fiber as compared to the sized carbon fiber. The interfacial shear strength (IFSS) value obtained of desized CF's was higher than of T300B and T700SC. As far as interfacial bonding was concerned the occurrence of sizing agent on T300B and T700SC fiber surface proved negative. The results also proved that the interfacial shear strength for CFRC depends upon physical and adhesive interfaces and not only on chemical bonding

Yuan *et al.* (2012) studied the silane functionalization on carbon fibre surface using APS silane coupling agent (3-Aminopropyl) trimethoxy silane). SEM, XPS and FTIR techniques were used for investigating surface characteristic and morphological changes of carbon fiber. APS surface treated carbon fiber shows increase of C-O-R bonds. Silane coupling agent on hydrolysis forms hydroxyl groups and in case of 3-Aminopropyl trimethoxy silane amino group is attached to Si which further increases bonding of fiber and matrix. Si-O bonds are formed on the surface of fiber. This clearly proves the surface functionalization on the fiber surface. Tensile strength of silane treated carbon fiber increased by 15.5% as compared to untreated fiber. This shows there is an increase in interfacial adhesion which ultimately improved the mechanical properties.

Shi *et al.* (2012) compared the tensile and flexural strength of the CFRC with CFRC having carbon content (2-15%) increased in CFRC. The results indicated increase in tensile as well as flexural strength. Further a comparison of the mechanical properties of the neat CFRC to the nitric acid and silane coupling agent treated CFRC was done. The mechanical properties were enhanced by oxidation process (increase in tensile, flexural, impact strength), which further elevated with treatment of silane coupling agent (KH550). FTIR analysis showed the addition of functional groups on the carbon fiber surface.

Han *et al.* (2013) treated the carbon fiber with (3:1 v/v mixture of concentrated H_2SO_4 and HNO_3 at 60°C) and neopentyl (diallyl) oxy, tri (dodecyl) pyro-phosphate titanate coupling agent. The effect of these surface treatments was checked on the interfacial properties of carbon fiber/epoxy composites. Surface roughness was increased by acid treatment process on carbon fiber. The tensile strength increased by 45.6% with 1 wt. % silane treatment for 3 hrs. The tensile strength was increased by only 14.6%, after the acid treatment process. The decrease may be due to the pore formation by oxidation effect. The combined treatment fiber with acid and titanate coupling agent moderated the reduced tensile strength because of the pore filling effect of the coupling agent.

Yang *et al.* (2013) studied the matrix modification of CFRC with two silane coupling agents. Effect of Modifications was studied by comparing mechanical properties, SEM images and FTIR results. Results of flexural testing, tensile testing, ILSS showed improvements in silane treated fibers. The silane coupling agents N-(2-aminoethyl)-3-aminopropylmethyldimethoxysilane)(YDH-602) and N-(2-aminoethyl)-3-aminopropylmethyltrimethoxysilane)(YDH-792) were compared at 0.5% wt. and mechanical properties of the YDH792-modified epoxy composites was better than that of the YDH602-modified epoxy composites at the 0.5% wt. concentration. An improvement of 4% and 44%, in tensile and flexural properties respectively in YDH792-modified epoxy composites was obtained in comparison to the unmodified epoxy composite. Comparison of ILSS at 0.5%wt of the YDH602-modified epoxy composites and YDH792-modified epoxy composites showed increase of 17% and 42% in ILSS respectively as compared to the untreated fiber respectively. FTIR results of both the silane treated fibers showed the presence of NH_2 amino group between 3200cm-1 and 3400cm-1 range. SEM images clearly showed the effect of silane functionalization as the images of fractured surface of untreated epoxy composite showed a very little amount of epoxy resin adhesion and the matrix is found to be broken to pieces between the fibers. This clears the fact that the silane treated polymer had better adhesion between them

Nurul *et al.* (2014) compared the results by treating the natural CF with alkalis, silane and using both (silane on alkalized CF) and varying their concentrations. The comparisons were done by tensile strength, impact strength and the SEM images of different samples. The results indicated increase of mechanical properties by surface treatments and increase of interfacial adhesion between carbon fiber and polymer matrix. The tensile strength increased by 28% of the silane treated fibers and increase of 41% of silane treated on alkalized CF.

Table 2.1 : Concentrations (v/v) %

Sample	Alkali	Silane	Silane Alkalised CF
A	2.5	.05	2.5+0.25

B	5	0.5	5+0.5
C	7.5	0.75	7.5+0.75
D	10	1	10+1

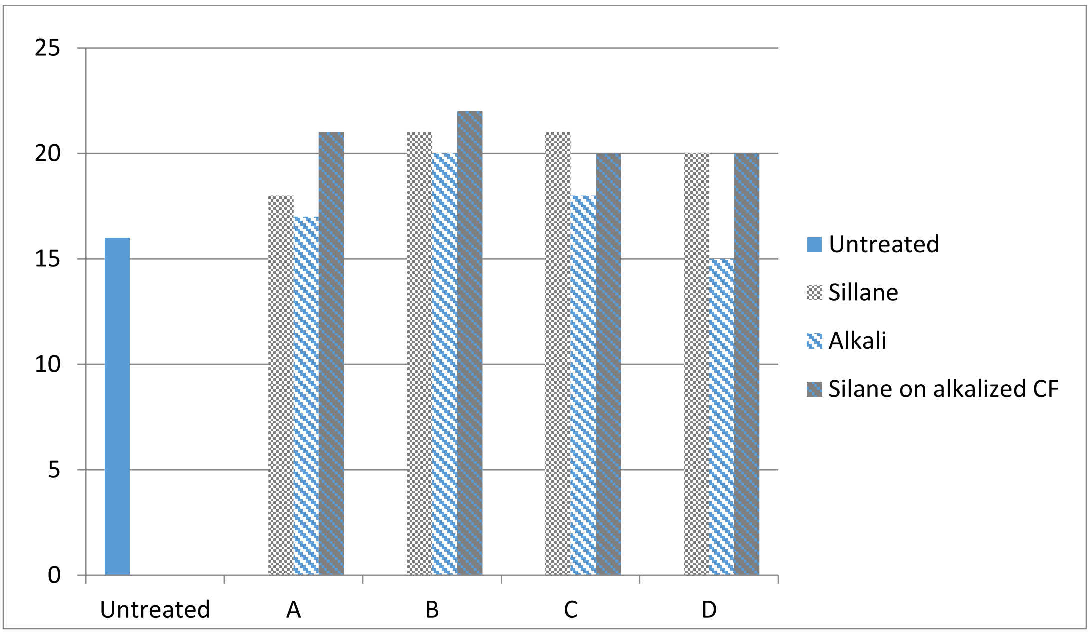


Figure 2.2 : Detailed tensile results(Munirah Abdullah & Ahmad 2012)

Sharma *et al.* (2014) reviewed the work done in the area of surface changes involved by treatments of carbon fibers and the carbon fiber/matrix interfacial adhesion. Surface modifications techniques such as wet, dry and multi-scale are defined in a proper way. The probable theoretical strength of carbon fiber is about 100GPa, whereas commercially obtained value was about 7GPa.There are numerous factors which effects the strength of a composite, fiber matrix interaction, fiber volume fraction, residual stress, stress concentration factor. To improve and compensate any factor decreasing the strength selective treatment and coatings methods were derived. The study suggests that multi scale treatment methods by ascribing Nano particles are more promising than the dry and wet treatments. Although wet treatment methods like acid treatments, silane coupling treatments on carbon fibers have proved very successful in increasing the strength of the CFRC.

Shi *et al.* (2014) treated the carbon fiber surface in a three way process(acetone desizing, nitric acid oxidation, $LiALH_4$ reduction and salinization reaction) and checked the effect of these surface treatments on the interfacial adhesion of CF composite. The most effective method came out to be silane surface treatment which increased the interfacial adhesion by 37% percentage higher than the other two methods. Surface treatments resulted in improved wettability, polarity and toughness of carbon fiber without any decrement in tensile strength of CF.

Turla *et.al* (2014) compared the flexural strength of glass fiber reinforced composite, carbon fiber reinforced composite and combined hybrid composite. The flexural results indicated high flexural strength values of GFRC compared to CFRC and the hybrid composite. The brittleness of carbon fibers may be the reason for comparatively less flexural strength of CFRC may be the although carbon fiber reinforced composites possesses a high value of tensile strength. The hybrid composite possessed a middle value of flexural strength that of a Carbon fiber reinforced composite and Glass fiber reinforced composite.

Jiang *et al.* (2015) studied the change of interfacial adhesion on surface treated carbon fiber composites. A three step surface treatment process (acetone desizing, nitric acid oxidation, LiAlH4 reduction and salinization reaction) helped in the improvement of interfacial adhesion of CF composite. Improvement of almost 47% occurred in IFSS by treating the fibers with silane coupling agent. The silane coupling agent used was [3-(2-Aminoethyl) amino propyl] trimethoxy silane. Composition changes with various surface treatments can be seen from the results as Si becomes 7.2% of the all surface elements abundance and nitrogen forms 3.6% in the Silanized carbon fibers as compared to the untreated carbon fibers which have nitrogen 1.5% and no traces of Si. This change occurred due to the formation Si-O-Si by the silane coupling agent treatment. The carbon content decreases in the treated fibers due to the addition of other elements.

Patel *et al.* (2015) reported the contribution of epoxy and fibers on the flexural strength, ILSS and energy absorption by glass fiber reinforced composites. Two types of different glass fibers and two resin systems were used in manufacturing process. VARIM was used in the composite manufacturing. Two fabrics with Fabric counts (197×157 threads/m) and (629×551 threads/m) namely GF1 and GF2 respectively are used with resin systems SC15 and SC780 preparing four different combinations. An increase of 24% higher ultimate strength was obtained when GF2 was used with SC15 as compared to that when GF1 was used. Increase of ultimate strength obtained with resin system SC780 was 82% with GF2 as compared to GF1.With lower fiber diameter GF2 had a higher glass density which made the GF2 composites stronger. The difference of the strength can also be attributed from the fact that SC780 resin had greater diffusion in to the glass fibers. Same trend was followed by inter laminar shear strength of the composites. In case of thinner glass fibers (GF2) the higher ILSS value may be obtained due to more surface area being in contact with the resin.

Choi *et al.* (2015) evaluated the effect of sizing treatments by the silane coupling agents on the interfacial adhesion properties between the fibers and a thermoplastic matrix. Three types of coupling agents were used vinyltriethoxy silane (VTES), 3-amino propyl triethoxy silane (APS), and 3-methacryloxy propyl trimethoxy silane (MPTS).FTIR analysis showed functionalization on the fiber surface (-COOH, C=O) at 1600-1800 cm$^{–1}$ (-OH) at 3450 cm^{-1}, (-CH) at 2980 cm^{-1} (Si-O-C) at 1380 cm^{-1} and Si-O-Si at 1073 cm^{-1}).Further the ILSS values of treated carbon fibers were compared which showed MPTS was more effective followed by APS and VTES treated composites respectively.

Joun *et al.* (2015) reported tensile testing results analysing by FEA emphasising on sample necking. FEA approach for analysing tensile tests has been presented. A mechanically perfect, rectangular bar is used as a finite element model. When the true strain becomes equal to the strain hardening exponent of the material at the point the necking takes place. The strain hardening behaviour of a material decides the necking process so it is a material characteristic property. The boundary conditions given in the model were, one end of the specimen is provided with the velocity in same plane in which the tensile tests are done giving zero velocity in the other two directions. The velocity component in the direction in which velocity is given is made zero in the opposite face. When the effect of area reduction becomes weaker than the strain hardening then necking takes place due to strain hardening effect, but the results showed that necking can’t be predicted because the deformation in the tensile test is non-uniform. So when the effect of area reduction becomes stronger than the strain hardening the region of major deformation becomes weak and necking takes places due to another additional deformation.

Mathapati *et al.* (2015) prepared three samples by varying the glass fiber content 1,2,3 per cent using bisphenol A as binding material and conducted mechanical tests on the samples. The experimental results were compared with the FEA results using ANSYS. It is clearly seen that the deformation increases as the load increases. The deformation obtained by FEA analysis is less than the experimental value. This difference of deformation results is due to the manufacturing defects like blow holes, porosity, improper mixture of fibers and matrix and reinforcement agglomeration.

Krishnamoorthy *et al.* (2016) compared the deformation results obtained by tensile testing of GFRC to results obtained by FEA approach using ANSYS. From the experiments Poisson ratio and Young's modulus value were obtained and used in FEA process. Meshing is done in 2D as well as in 3D and the deformation obtained in both cases by FEA approach is compared to the experimental values. The deformation results obtained by FEA approach are better than the experimental results and reason for this deviation might be due to manufacturing defects like blow holes, porosity.

Chapter 3

Methodology

3.1 Introduction

This chapter describes the methodology used in the present work. It consists of all the details including the objectives, material selection, equipment used in the testing, method of surface treatments and procedure of fabrication of composites.

3.2 Scope of the study

The scope and requirement of this study could be explained by keeping in mind the applications of CFRC and material and time saving experimenting methods.

1. Improvement of mechanical properties: Keeping in mind the application part of CFRC like in automotive and aerospace industry, in sports industry, in constructions and many more the mechanical properties play a very important role.
2. Selection of optimized methods of surface treatments on fibers so that the selection process of surface treating methods (%concentrations, treatment time) is made easy for future work.

3.3 Establishing the Objective Function

From the literature review it is very clear that effect of silane treatments on carbon fibers has been studied but no literature is available on effect of varying the reaction times with different silane concentrations. The main points that are covered in this study include:

1. Effect of silane concentration and reaction time on properties of carbon fibers and their composites.
2. Finite element modeling and deformations analysis of carbon fiber reinforced composites.

3.4 Work Plan

3.4.1 Material Selection

- **Epoxy Resin**

The epoxy system used is Araldite GY 257 and a Hardener HY 837 (supplier Huntsman Advanced materials, India). Araldite GY 257 is an epoxy resin with low viscosity and is aromatic glycidyl ether of Biphenol A. Araldite GY 257 has very good mechanical properties, good surface penetration, good chemical resistance. Hardener HY 837 has good chemical resistance, room temperature fast curing and excellent adhesion. The properties of both constituents are given in Appendix -I.

- **Silane coupling agents**

Silane coupling agents used are 3-Amino propyl triethoxy silane and 3-Glycidyloxypropyl trimethoxy silane (supplier TCI chemicals LTD Chennai).

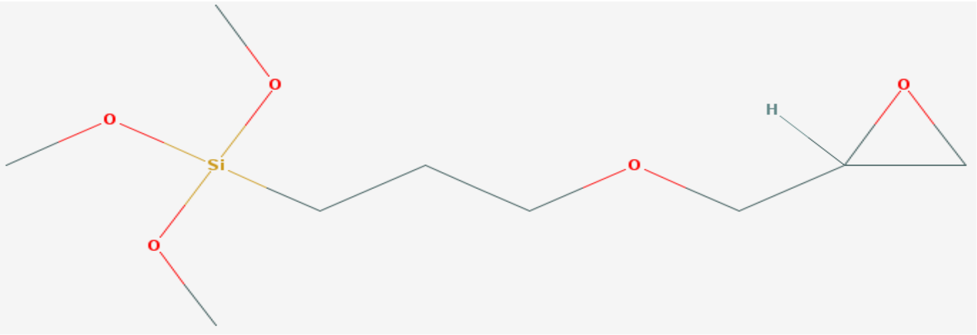


Figure 3.1 : 3-Glycidyloxypropyl trimethoxy silane chemical structure(Erden et al. 2010)

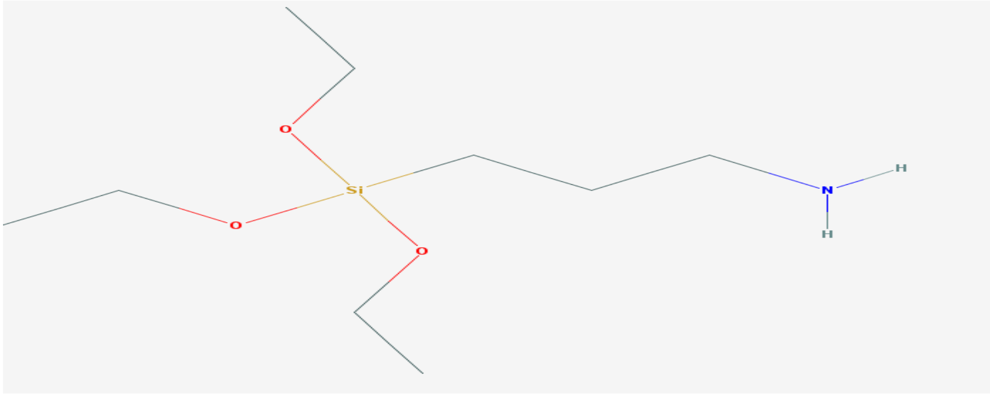


Figure 3.2 : 3-Amino propyl triethoxy silane chemical structure(Erden et al. 2010)

- **Carbon Fibers**

  Unidirectional carbon fibers were purchased from BASF India Limited, Mumbai. The properties are given appendix-II.

3.5 Methodology for Phase-I

This phase comprises of the surface treatment of carbon fibers. The reaction times for three time intervals were 20, 30 and 40 minutes. The purpose of doing the work in two phases was to first arrive at optimized reaction time of silane treatment and then in second phase determine the effect of silane functionalization on mechanical properties of composites.

Treatment with 3-Aminopropyltriethoxy silane coupling agent-1% (v/v) concentration was used of the amino silane for the surface treatment process. Amino silane was added to distilled water and the solution was stirred for 15 minutes using mechanical stirrer at 550 rpm. pH was maintained between 4-4.5 of the solution . Fibers were immersed for two time periods: 20 or 40 minutes and then were washed with distilled water. The washed wet fibers were kept in room temperature for 24 hours for drying. Before using the fibers for preparing composites, they were kept at 120^{o}C in a vacuum oven for 5 minutes for the purpose activating the silane on the fiber surface (Sharma et al. 2014).

Figure 3.3 : pH meter

This phase comprises of preparation of carbon fiber reinforced composite.

3.5.1 Manufacturing of Composites

The epoxy is prepared by: EPOXY = A + B

A = Amount of resin
B = Amount of hardener

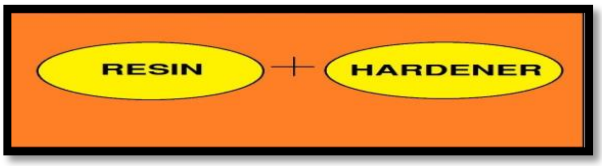


Figure 3.4 : Weighing machine

Resin being used **Araldite GY 257 IN**

Hardener being used **Araldite 837**

Mixing weight ratio of epoxy to hardener **100:35**

- A beaker was taken and simultaneously keeping in mind the weight percentage, is mixed with each other. The hardener was poured into the beaker containing the epoxy resin slowly and mixed at 1200 rpm for 2 minutes using a mechanical stirrer (Kaushik & Singh 2010).

Figure 3.5 : Mechanical Stirrer

- The dried fibers were kept in an oven for 5 minutes at 120°C for the purpose of activating the silane (Zielke et al. 1996). The vacuum bagging equipment was made ready. All the consumables needed for the process are made ready. After taking out the fibers from the oven three plies are laid on the equipment’s surface. The epoxy prepared is quickly applied on the fibers one by one and stacking the plies on each other. A scrapper is used to apply the epoxy system. Considering very less curing time of the epoxy system this process is done very fast. After this the consumables are used in order the peel ply, the perforated film and the infusion mesh. Vacuum connections were made using a breather cloth and in the final step vacuum bagging are done and the mould is sealed. The laminates are kept under the vacuum at a temperature maintained at 45°C for 4 hours and then 12 hours at room temperature (Wang et al. 2015).

3.6 Methodology for Phase-II

The appropriate time after comparing the mechanical properties the samples (Tensile, flexural, ILSS) was taken as 30 minutes for the silane treatment. In second phase two silane coupling agents were used namely 3-Amino propyl triethoxy silane and 3-Glycidyloxypropyl trimethoxy silane. Fibers are treatment using these two silane agents also varying their treatment concentrations. 1%, 2%, 2.5% (v/v) is the concentrations used for the treatment process.

Treatment with 3-Glycidyloxypropyl trimethoxy silane coupling agent- Three concentrations were used of the GPS silane for the surface treatment process.1%, 1.5%, 2.5% (v/v) is the concentrations used. The solvent used was prepared using water and ethanol in 90:10 ratios respectively. GPS silane was added to the solvent and the solution was stirred for 15 minutes using mechanical stirrer at 550 rpm. pH was maintained between 4-4.5 of the solution (Turla et al. 2014). Fibers were immersed for two time period of 30 minutes and then were washed with distilled water. The washed wet fibers were kept in room temperature for 24 hours for drying (Han et al. 2013). Before using the fibers for preparing composites, they were kept at 120°C in a vacuum oven for 5 minutes for the purpose of activating the silane.

Treatment with 3-Aminopropyltriethoxy silane coupling agent-1%, 1.5%, 2.5% (v/v) concentrations were used of the amino silane for the surface treatment process. Amino silane was added to distilled water and the solution was stirred for 15 minutes using mechanical stirrer at 550 rpm. pH was maintained between 4-4.5 of the solution . Fibers were immersed for a time period of 30 minutes and then were washed with distilled water. The washed wet fibers were kept in room temperature for 24 hours for drying. Before using the fibers for preparing composites, they were kept at $120^{o}C$ in a vacuum oven for 5 minutes.

Preparation of epoxy resin is done by the using same methodology discussed in the phase-I using the same resin and hardener ratio.

Figure 3.6 : Consumables used in the vacuum bagging process

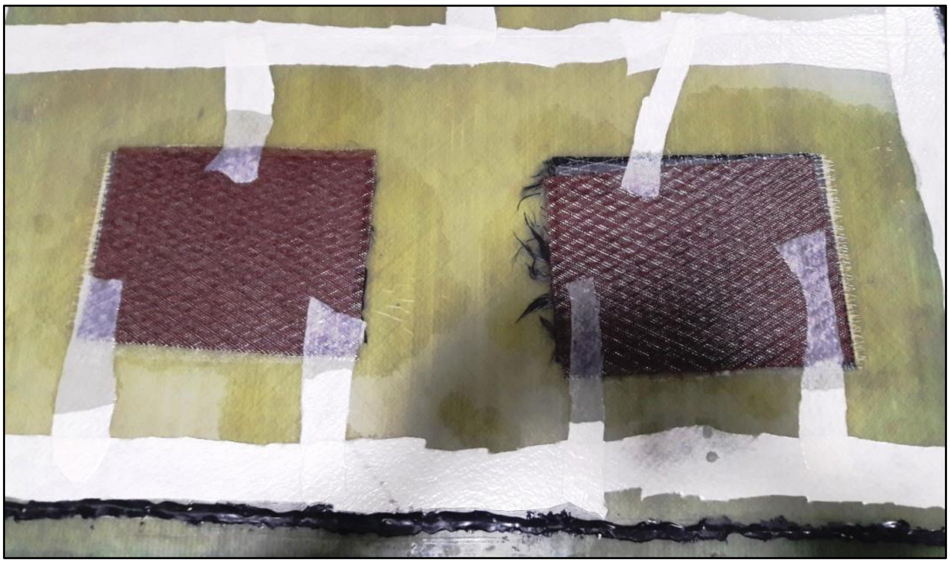


Figure 3.7 : Samples being prepared using the vacuum bagging

3.7 Mechanical testing

3.7.1 Three point bending test

CFRC specimens were primed as per the ASTM standard D790 for flexural testing on the UTM. The specimens were cut in the form of rectangular bars with dimensions 25.4 mm×13mm×1.02mm using hand grinder available in the central workshop, Thapar University, Patiala .The rate of crosshead motion was kept as 1.02 mm/min which was calculated using the equation 1. Three samples (minimum) were tested for each of the composite prepared.

The specimens were notched according to the ASTM standard D790. Rate of crosshead motion for machine was premeditated by:

$$R=ZL^2/6d \qquad \text{Equation 1 Rate of crosshead motion}$$

Where

R= rate of crosshead motion (mm/min)

L= support span (mm)

d= depth of beam (mm)

Z= rate of straining of the outer Fiber, mm/mm/min. Z should be equal to 0.01

Specimens were test using Zwick/Roell universal testing machine

3.7.2 Tensile testing

CFRC specimens were primed as per the ASTM standard D3039 for tensile testing of the material on the UTM. The specimens were cut in the form of rectangular bars with dimensions 250 mm×25mm×1.02mm using hand grinder available in the central workshop, Thapar University, Patiala .The rate of crosshead motion is kept as 1 mm/min. Three samples (minimum) were tested for each of the composite prepared.

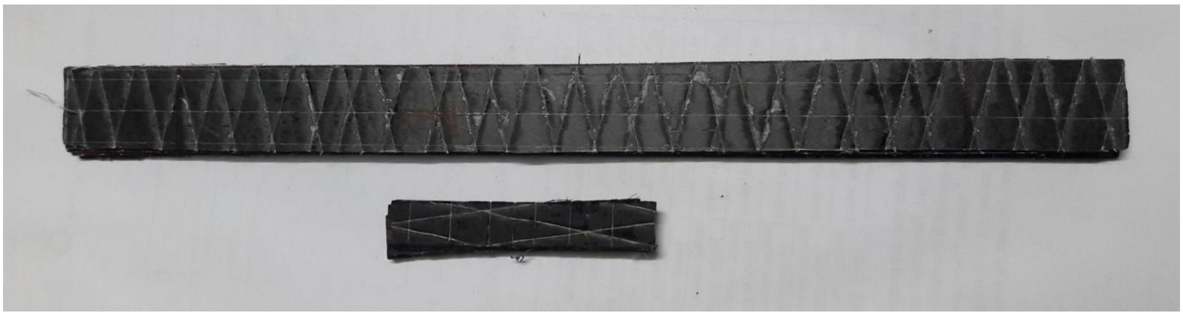


Figure 3.8 : Tensile and flexural rectangular specimens

Chapter 4

Results and Discussion

The results obtained from flexural testing and tensile testing of CFRC as a function of silanization reaction time

Table 4.1 : Flexural testing results of composite as a function of silanization reaction time

Specimens	Samples	Flexural Modulus (MPa)	Mean Flexural modulus (MPa) (Mean)	Mean Flexural strength (MPa)
Neat CFRC	1	34750	34416.67	309.33
	2	34000		
	3	34500		
1%v/v APS(20min)	1	39500	39233.33	353.59
	2	39000		
	3	39200		
1%v/v APS(30min)	1	42300	42100	373.22
	2	42000		
	3	42000		
1%v/v APS(40min)	1	42400	42166.67	369.97
	2	42100		
	3	42000		

Table 4.2 : Tensile testing results of composite as a function of silane reaction time

Specimens	Samples	Tensile strength (MPa)	Mean Tensile strength (MPa)
Neat CFRC	1	628.938	627.394
	2	627.122	
	3	626.122	
1%v/v APS(20min)	1	682.123	683.4157
	2	684.112	
	3	684.012	
1%v/v APS(30min)	1	721.612	720.7713
	2	720.148	
	3	720.554	
1%v/v APS(40min)	1	724.221	725.111
	2	725.120	
	3	725.960	

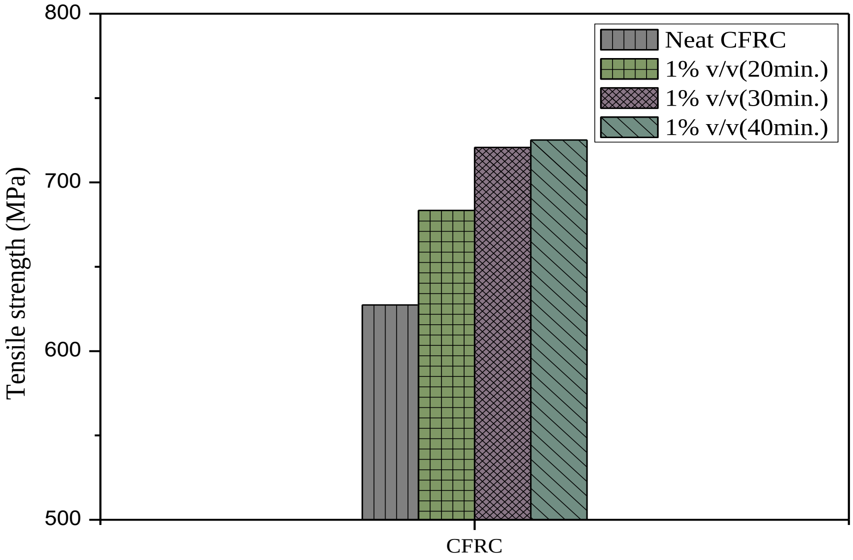


Figure 4.1 : Tensile strength (MPa)

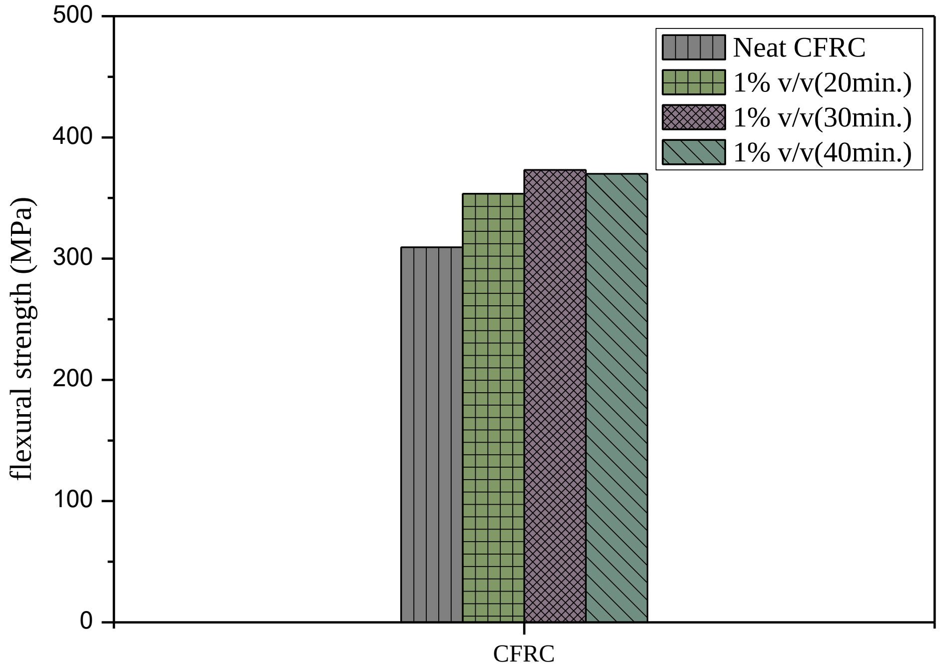


Figure 4.2 : Flexural strength (MPa)

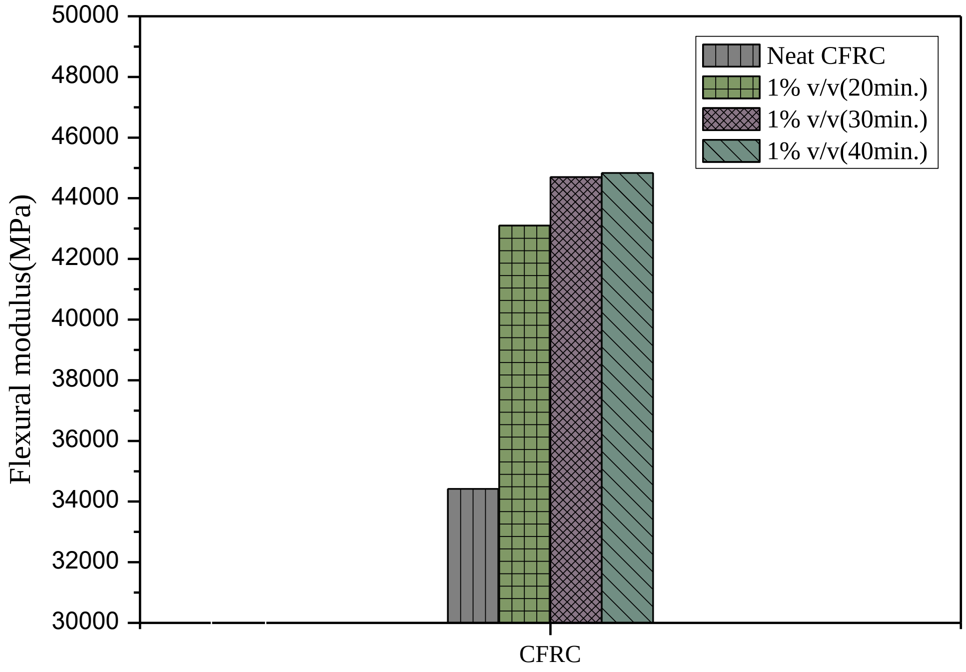


Figure 4.3 : Flexural modulus (MPa)

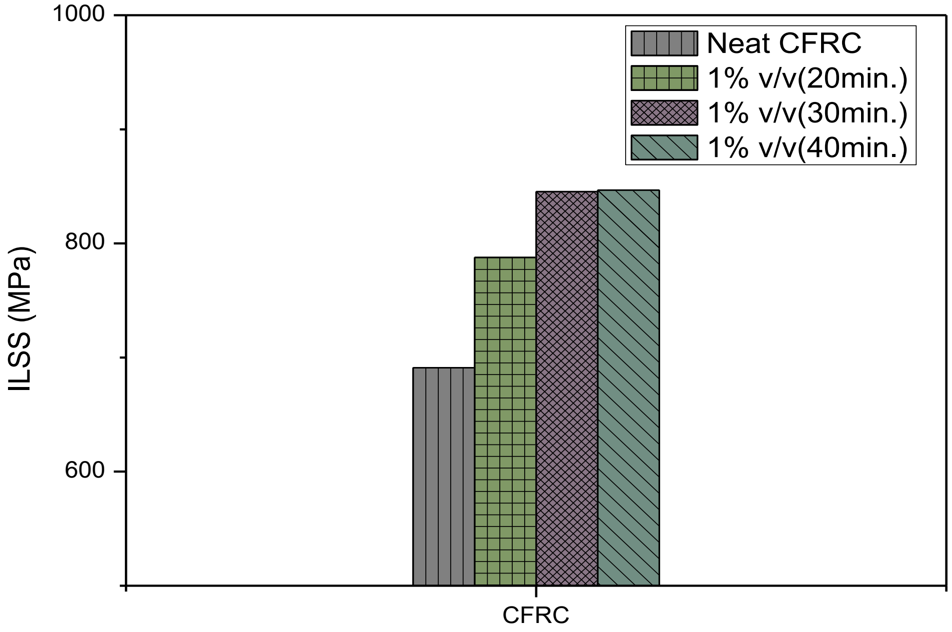


Figure 4.4 : ILSS (MPa)

It can be observed from table 4.1 that flexural modulus value of neat CFRC increased by 13.9% when treated with APS silane coupling agent for 20 minutes. When the time was increased from 20 to 30 minutes flexural modulus value increased by 7.3%.From table 4.2 it can be observed that the tensile strength increases by 5.42% when treatment time was increased from 20 to 30 minutes. The change in ILSS value from 20 to 30 minutes was 7.4% increase. But when treatment time was increased from 30 to 40 minutes there was a negligible change in all the discussed properties.

The same trend can be observed from the figures 4.1-4.4 that there was a large increase in all above mechanical properties of silane treated CFRC for 20 minutes as compared to neat CFRC. Also with the change of time from 20 to 30 minutes there was considerable change of mechanical properties. So it can be concluded that the optimized treatment time is 30 minutes. Thereafter for all the other comparisons in the work the treatment time was kept 30 minutes.

4.1 FTIR Results

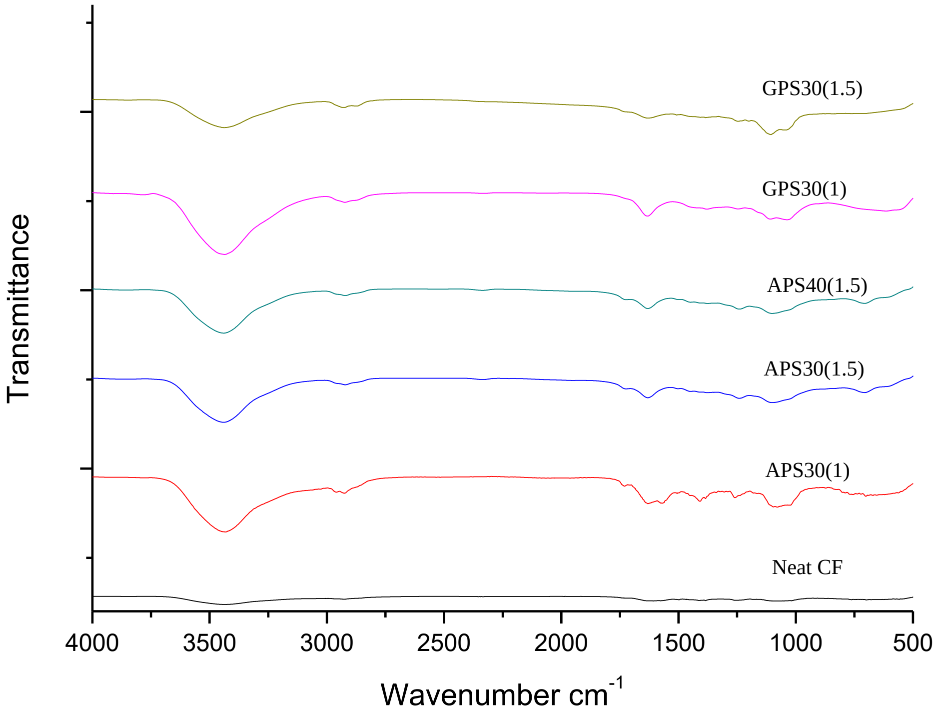


Figure 4.5 : FTIR spectra of neat CF and silane treated CF

Figure 4.5 shows the FTIR spectra of neat carbon fiber, APS and GPS coupling agent treated carbon fibers. FTIR results indicate chemical modification in fiber after the silane treatment process. In case of modified carbon fibers band 3450cm^{-1} is is assigned to hydroxyl group and H-bonded OH stretch(Sharma et al. 2016). The band between 2920cm^{-1}and 2960 cm^{-1} are due to –CH_2 symmetric and –CH asymmetric stretching(Yuan et al. 2012). The band between 1250cm^{-1} and 1270cm^{-1} is assigned to Si-O-C bonding and 1020cm^{-1}-1150cm^{-1} to C-O stretch.

Band at 3450cm^{-1} broadens when the concentration increases in both the silane explaining increase chain lengths. Band between 2920cm^{-1} and 2960 cm^{-1} broadens with concentration increase from 1% to 1.5% in case of APS silane and opposite trend in case of GPS depicting better –CH_2 symmetric and –CH asymmetric bonding at concentration 1.5% in case of APS and 1% in case of GPS. A noticeable change in between band 1250cm^{-1} and 1270cm^{-1} is observed in case of APS which can explain a better Si-O-C bonding at higher concentration. Band between 1020cm^{-1}and 1110 cm^{-1} corresponds to C-O stretch which broadens with concentration increase of 1% to 1.5% in case of APS silane and is broader at 1% in case GPS silane. This may be due to better C-O bonding at concentration 1.5% in case of aps and 1% in case of GPS. Band between 1590cm^{-1} and 1690cm^{-1} there is a considerable change in case of gps and this change is attributed to C=C double bond formation. This change is not seen in case of aps treated fibers. Similarly at 1380 cm^{-1} wavenumber a peak is formed in case aps treated fibers which can be due NH_2 bending.

Table 4.3 : Flexural testing results of composite as a function of silanization concentration

Specimens	Samples	Flexural Modulus	Average flexural modulus (MPa)	Average flexural strength(MPa)
Neat CFRC	1	34750	34416.67	309.33
	2	34000		
	3	34500		
1%v/v GPS(30min)	1	43000	43233.33	397.44
	2	43100		
	3	43600		
1.5%v/v GPS(30min)	1	39300	39000	329.25
	2	38700		
	3	39000		
2.5%v/v GPS(30min)	1	40000	40050	331.54
	2	39800		
	3	40350		
1%v/v APS(30min)	1	42300	42100	373.22
	2	42000		
	3	42000		
1.5%v/v APS(30min)	1	46100	46033.33	384.59
	2	46000		
	3	46000		
2.5% v/v APS(30min)	1	43600	41933.33	363.5
	2	43700		
	3	43600		

Table 4.4 : Tensile testing results of composite as a function of silane concentration

Specimens	Samples No	Tensile strength(MPa)	Average Tensile strength(MPa) strength(MPa)	Average Tensile modulus (GPa)
Neat CFRC	1	628.938	627.394	18.93
	2	627.122		
	3	626.122		
1%v/v GPS(30min)	1	730.121	733.4333	23.29
	2	735.061		
	3	735.121		
1.5%v/v GPS(30min)	1	659.197	659.928	19.97
	2	660.675		
	3	659.912		
2.5%v/v GPS(30min)	1	730.055	728.8497	21.39
	2	726.374		
	3	730.121		
1%v/v APS(30min)	1	721.612	720.7713	21.24
	2	720.148		
	3	720.554		
1.5%v/v APS(30min)	1	743.142	741.8393	25.06
	2	742.151		
	3	740.225		
2.5v/v APS(30min)	1	717.949	714.1083	20.11
	2	710.989		
	3	713.387		

Table 4.5 : Inter laminar shear strength (ILSS)

Specimens	Average ILSS (MPa)
Neat CFRC	691.0434
1%v/v GPS(30min)	868.0708
1.5%v/v GPS(30min)	783.0709
2.5%v/v GPS(30min)	804.1535
1%v/v APS(30min)	845.3149
1.5%v/v APS(30min)	924.2912
2.5%v/v APS(30min)	841.9684

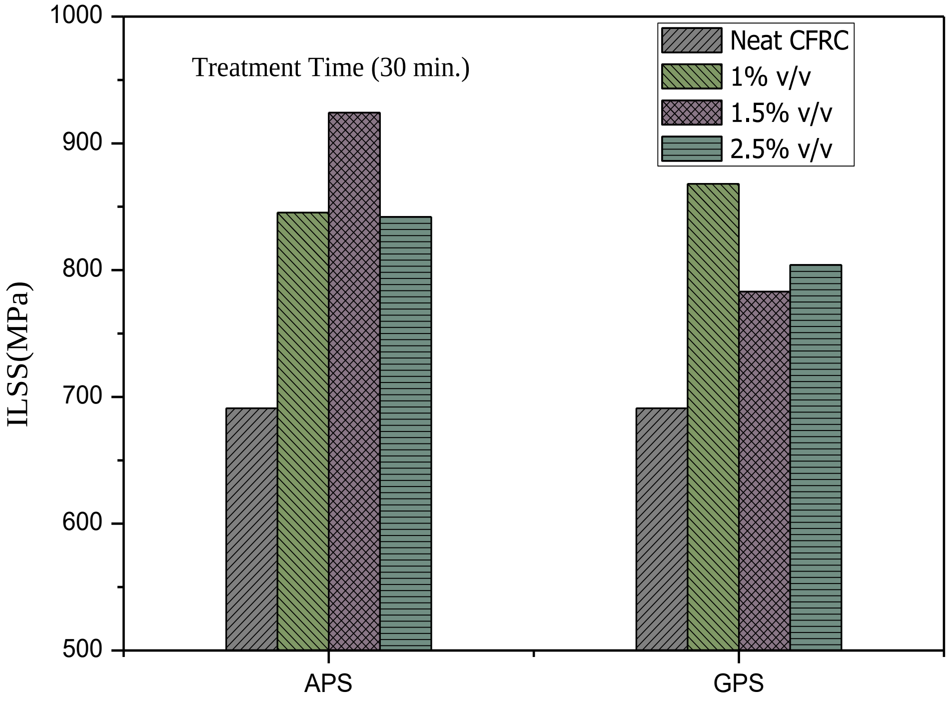


Figure 4.6 : ILSS (MPa)

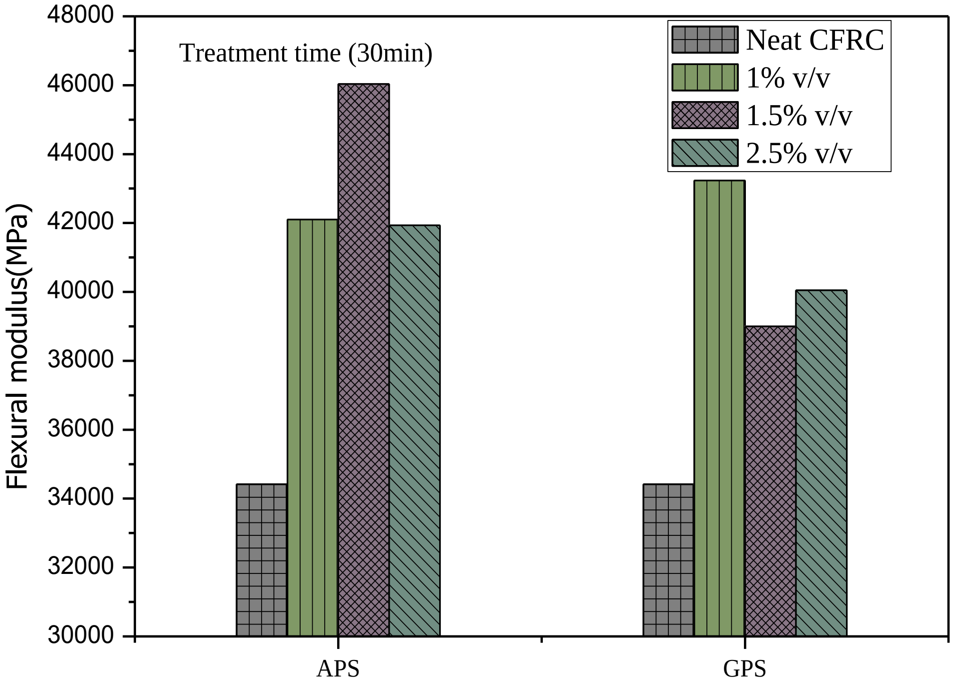


Figure 4.7 : Flexural modulus (MPa)

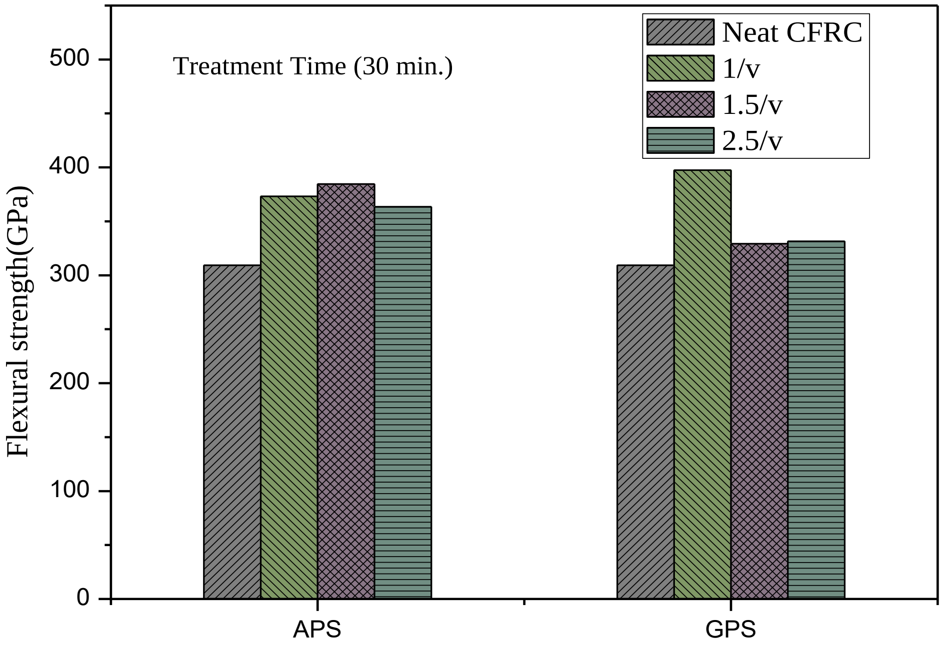


Figure 4.8 : Flexural strength (GPa)

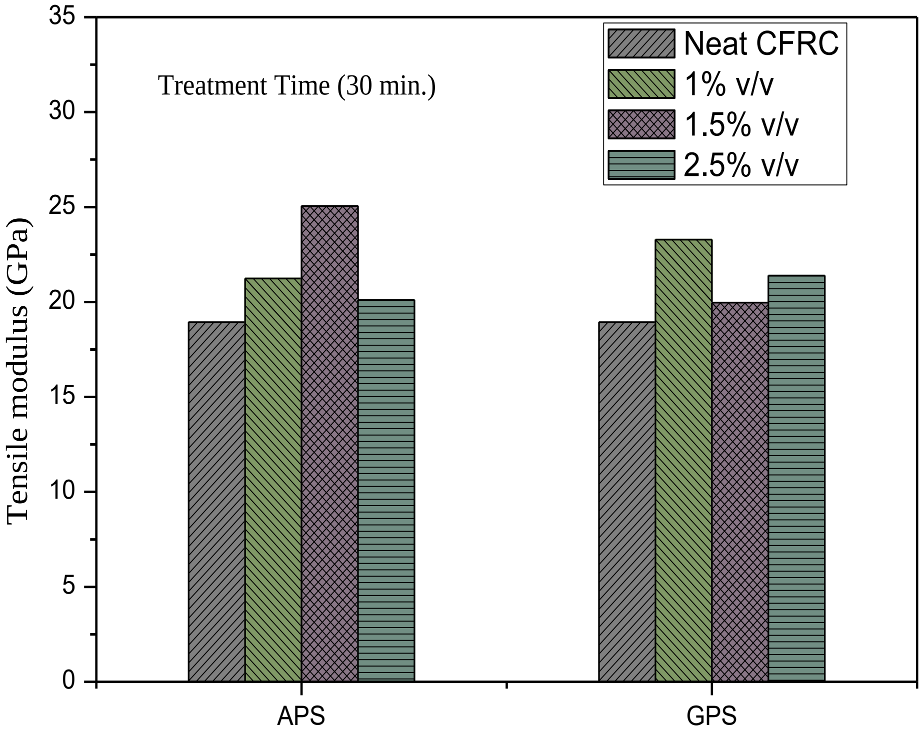


Figure 4.9 : Tensile modulus

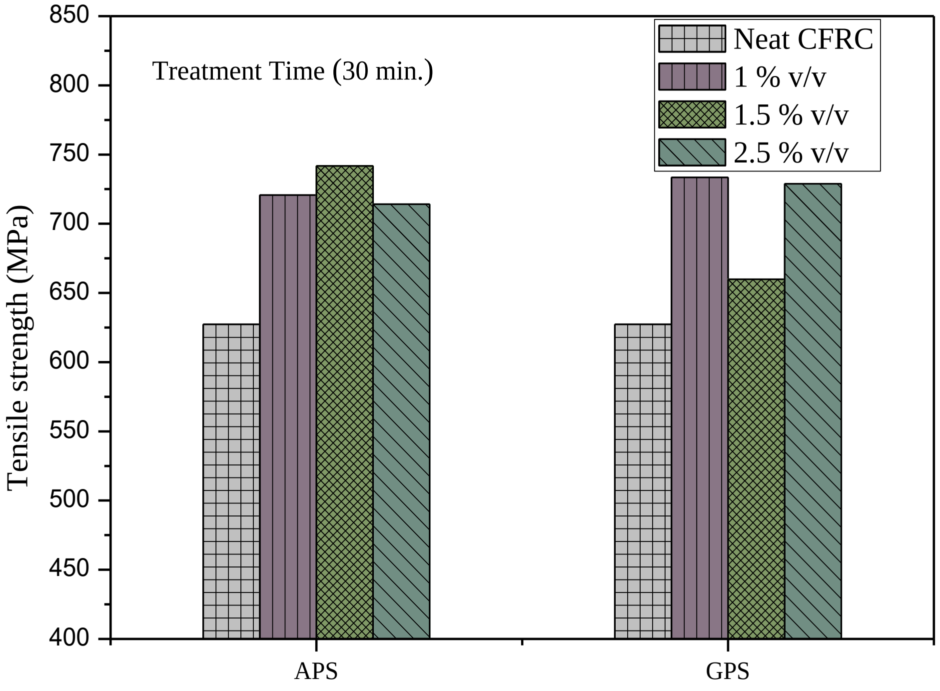


Figure 4.10 : Tensile strength (MPa)

Table 4.3 contains the flexural modulus and flexural strength results of composites treated with two silane coupling agents APS and GPS using three concentrations 1%, 2%, 3% (v/v). For both the silane coupling agent results follow a different trend. In case of GPS the maximum value of flexural modulus and flexural strength obtained is at 1% which is 25.6% and 28.4% respectively higher than the neat CFRC values, whereas in case of APS the maximum value of composites is at 1.5% which is 33.7%and 24.2% respectively higher than the neat CFRC values. In case of GPS there is a decrease of 10.85% and 20.66% of flexural modulus and flexural strength of composites respectively with the concentration change of 1% to 1.5% and in case of APS there is an increase of 9.7% and 2.9% in the composite values for the same concentration change. With the change of concentration from 1.5% to 2.5% there is no considerable change in flexural values in case of GPS but there is a decrease of 2.69% and 5.7% in flexural modulus and flexural strength of composites respectively as compared to the values obtained at 1.5%. Figures 4.8 and 4.7 clearly show the trend of flexural strength and flexural modulus followed by both the silane coupling agents.

Table 4.4 contains the Tensile modulus and Tensile strength results of composites treated with two silane coupling agents APS and GPS using three concentrations 1%, 2%, 3% (v/v). For both the silane coupling agent the results follow a different trend. In case of GPS the maximum value of Tensile modulus and Tensile strength obtained is at 1% which is 23.03% and 16.9% respectively higher than the neat CFRC values, whereas in case of APS the maximum value of composites is at 1.5% which is 32.38%and 18.18% respectively higher than the neat CFRC values. In case of GPS there is a decrease of 23.03% and 11.22% of tensile modulus and tensile strength of composites respectively with the concentration change of 1% to 1.5% and in case of APS there is an increase of 17.98% and 2.91% in the composite values for the same concentration change. With the change of concentration from 1.5% to 2.5% there is an increase of 16.62% and 10.47% in tensile modulus and Tensile strength of composites respectively in case of GPS and 24.61% and 3.78% decrease in case of APS as compared to the values obtained at 1.5%. Figures 4.9 and 4.10 clearly show the trend of Tensile strength and Tensile modulus followed by both the silane coupling agents.

Table 4.5 contains the ILSS results. In case of GPS the maximum ILSS value obtained is at 1% which is 25.61% higher than the neat CFRC values, whereas in case of APS the maximum value of composites is at 1.5% which is 33.72% higher than the neat CFRC values. In case of GPS there is a decrease of 10.85% in ILSS values of composites with the concentration change of 1% to 1.5% and in case of APS there is an increase 9.3% in the composite values for the same concentration change. With the change of concentration from 1.5% to 2.5% there is no considerable change in ILSS values in case of GPS but there is a decrease of 9.86% as compared to the values obtained at 1.5%. Figure 4.6 clearly shows the trend of ILSS followed by both the silane coupling agents

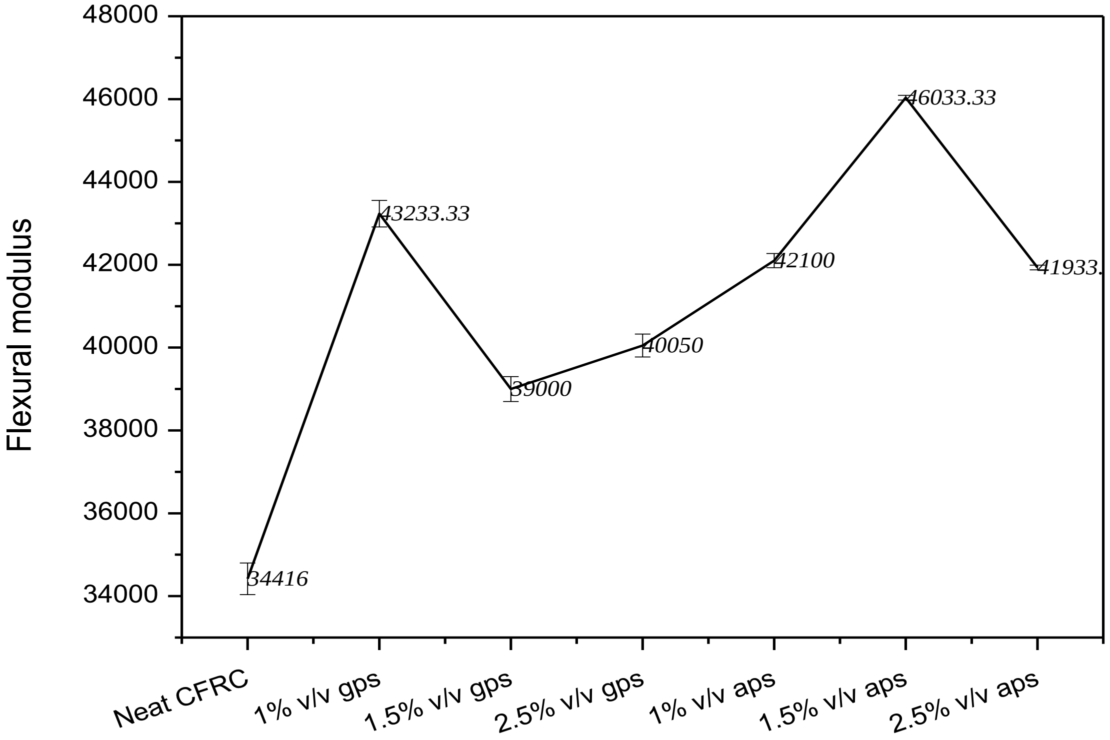


Figure 4.11 : Error bar graph for flexural modulus values

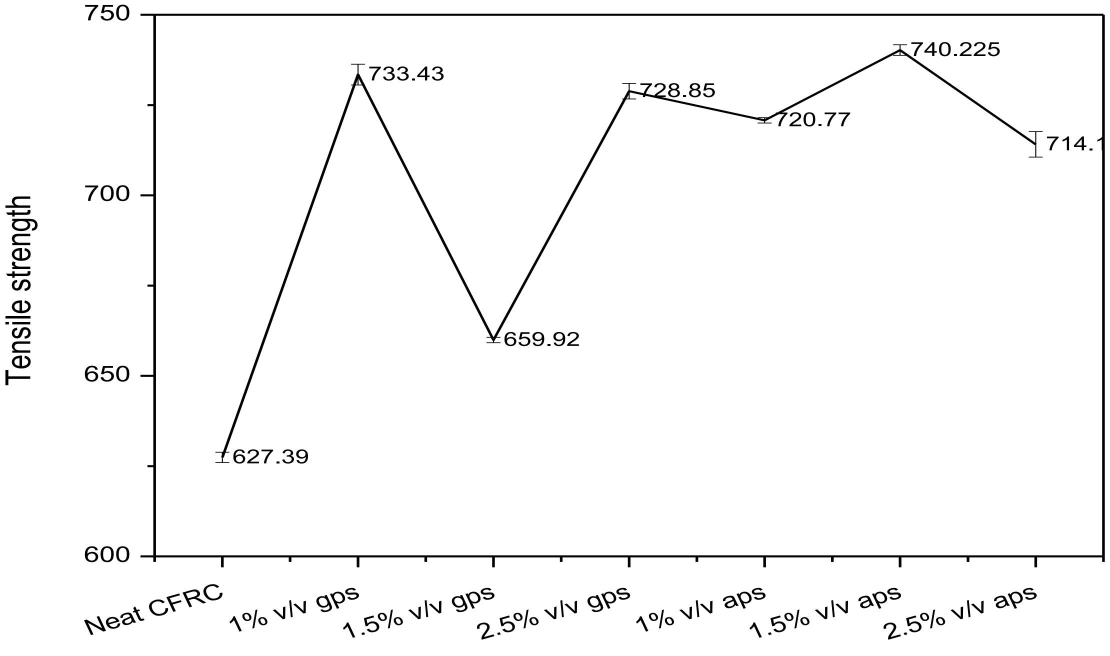


Figure 4.12 : Error bar graph for Tensile strength values

Figure 4.10 and Figure 4.11 are the error plots graphs for flexural modulus and tensile strength and these plots depict that there are noticeable changes in values when the silane concentrations are changed.

4.2 Finite element analysis

Table 4.5 : Actual and simulated deformation values

Specimens	Samples No	Load at break(KN)	Deformation(mm)	Simulated deformation(mm)
1%v/v GPS(30min)	1	19.93228	6.84	6.71
	2	20.06714	7.04	6.96
1.5%v/v GPS(30min)	1	17.65	6.16	5.79
	2	17.68957	6.2	5.86
2.5%v/v GPS(30min)	1	19.47057	7.29	6.97
	2	19.37239	7.1	6.93
1%v/v APS(30min)	1	19.70001	6.48	6.194
	2	19.66001	7.09	6.97
1.5%v/v APS(30min)	1	20.28999	7.12	6.868
	2	20.20801	6.99	6.93
2.5%v/v APS(30min)	1	19.37099	6.96	5.99
	2	19.47547	6.48	6.078

4.2.1 Methodology used

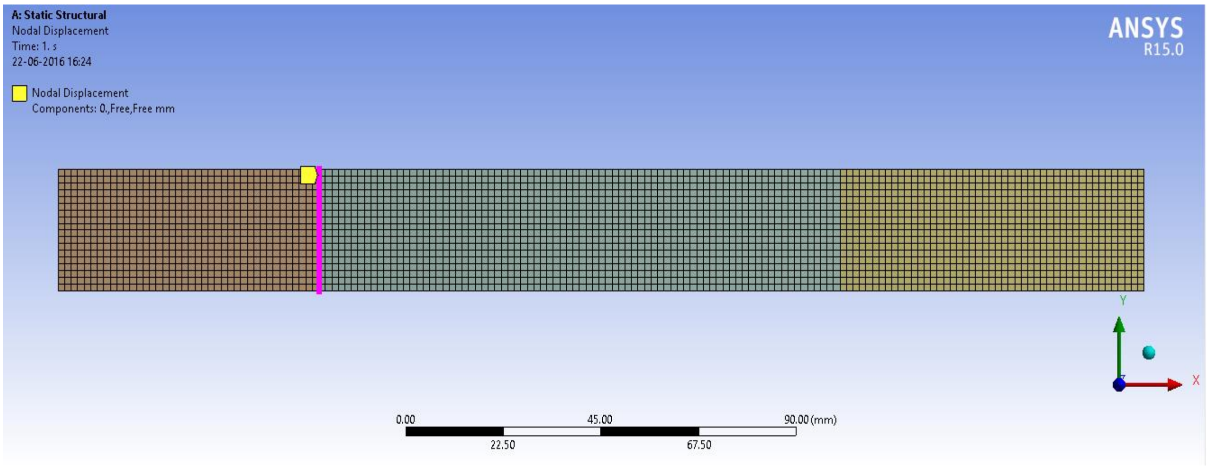


Figure 4.13 : Sample meshing with displacement boundary condition

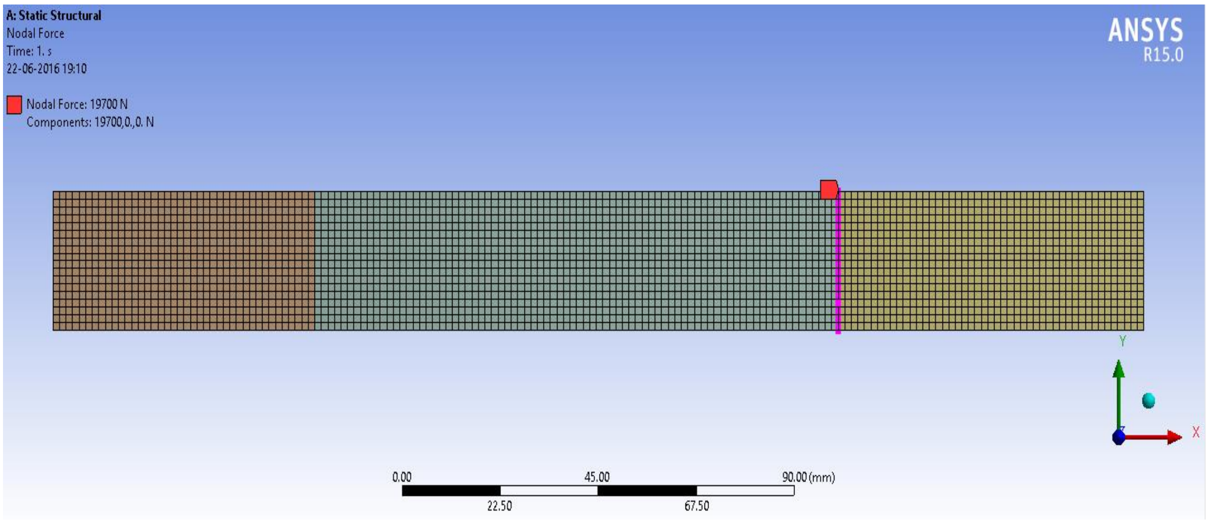


Figure 4.14 : Sample meshing with force boundary condition ((Mathapati & Mathapati 2015)

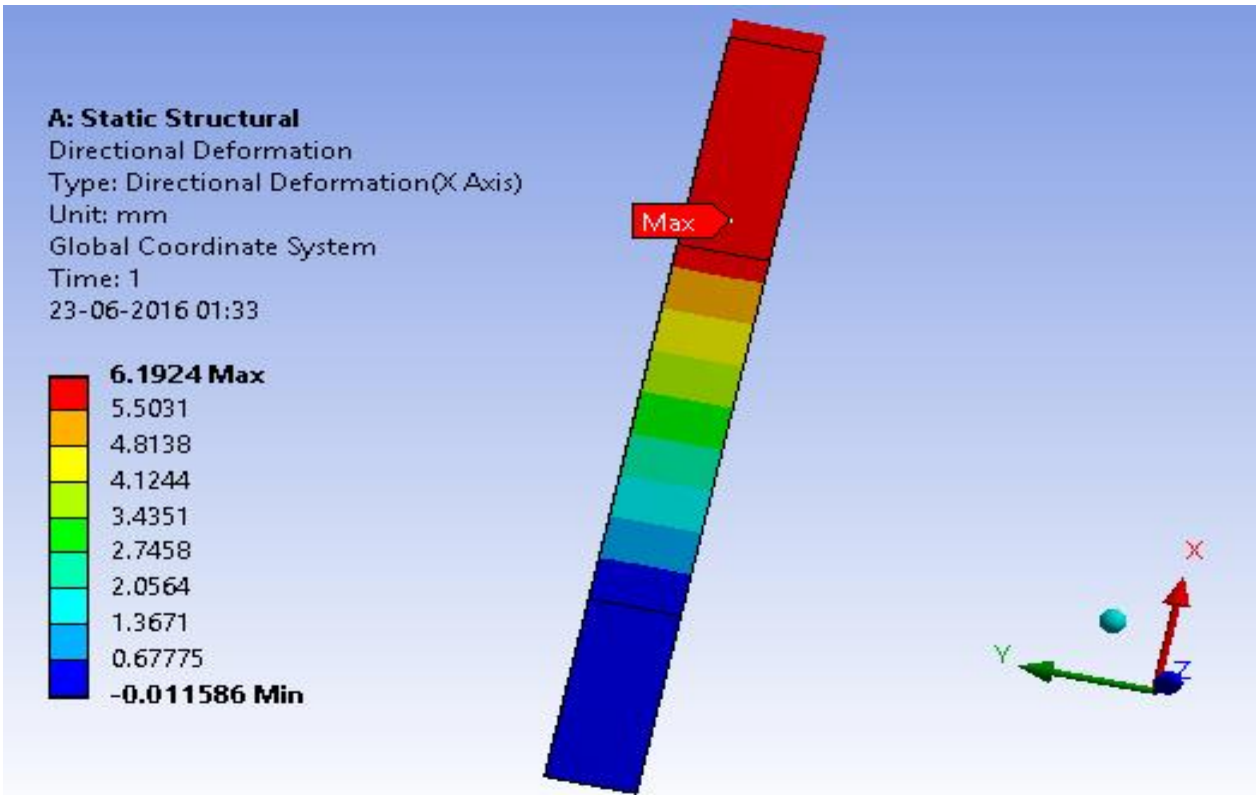


Figure 4.15 : Simulated Deformation of finite element model (1%v/v APS (30min))

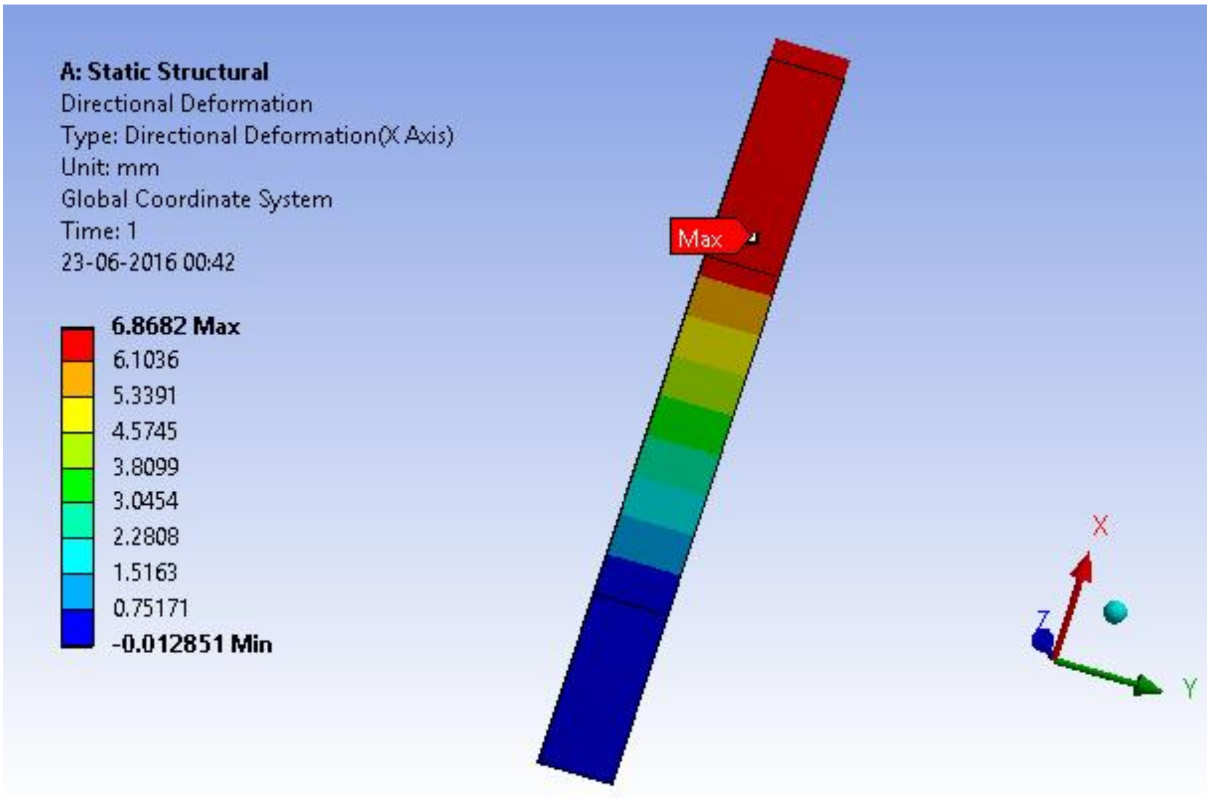


Figure 4.16 : Simulated Deformation of finite element model (1.5%v/v APS (30min))

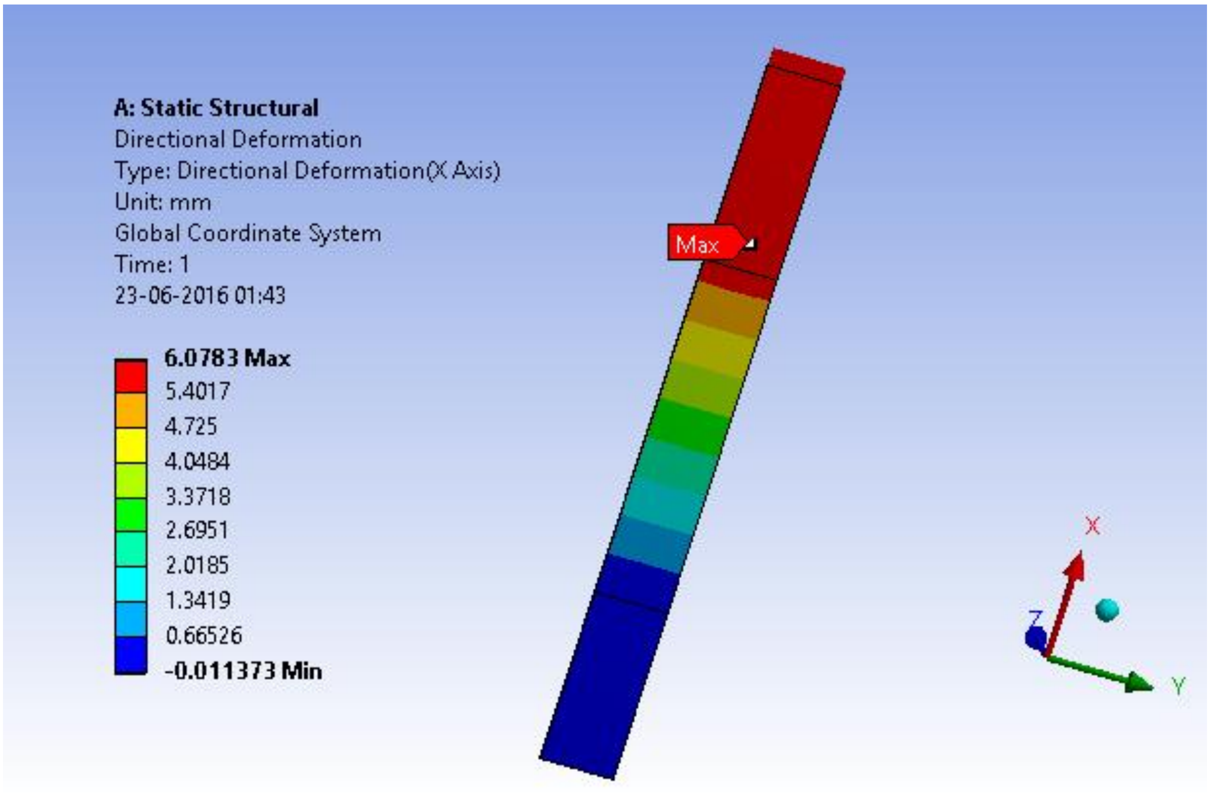


Figure 4.17 : Simulated Deformation of finite element model (2.5%v/v APS (30min))

The stage of pre-processing includes defining boundary conditions, external loads and the generation of mesh. The displacement conditions include restraining nodal displacements at one end and applying tensile load on the other end of the model. The slicing of FE model was done to generate different bodies for application of nodal forces and nodal displacement. In the next stage of FEM differential equations are solved within solver. The solution was obtained by ANSYS 15.0 solver and results were obtained in post-processing stage. The results shown in Fig. 4.15-4.17 indicate the Directional deformation of specimens. The values for all specimens are given in Table 4.6.

The simulated deformation of every sample is compared with the experimental deformed value as shown in Table 4.5. Error percentage of these results were calculated and the acceptable error percentage for model validation is within 20%(Anon 2016). This is an important step to verify the effectiveness of the model that is created by the software ANSYS. The range of error percentage for this study is between 2–9% which is considered to be within the acceptable range. The trend of the tensile modulus results between experimental and finite element analysis is similar. As observed in the fig 4.5, the trend of the deformation results predicted by FEA is in agreement with those obtained by experimental. From the comparison it is clear that deformation obtained by FEA results is less than that of the experimental values. This deviation happened due to the manufacturing defects of the composites (porosity, bubbles, void), discontinuities in the test (Joun et al. 2007).

Chapter 5

Conclusion and Future Scope

5.1 Conclusion

The silane functional groups and concentration of the silane influence the grafting of silane. The Si-O-C characteristic peak in FTIR spectra of carbon fibers confirmed successful grafting of silanes on fiber surface. The effect of reaction time and change in silane concentration influenced the mechanical properties of the composites containing silylated carbon fibers. For finding out appropriate treatment time fibers were treated with APS silane coupling agent for 20, 30, 40 minutes. Based on the tensile and flexural tests the treatment time of 30 minutes was found sufficient. The composite containing the carbon fiber treated with 3-Amino propyl triethoxy silane at 1.5% concentration (v/v) with treatment time 30 minutes possess the best mechanical properties with an increase of 33.6%, 32.38%, 33.72% in flexural modulus, tensile modulus and inter laminar shear strength respectively from the neat CFRC. The flexural and ILSS properties with treatment time 30 minutes increase in order Neat CFRC< GPS(1.5)< GPS(2.5)< APS(2.5)< APS(1)< GPS(1)< APS(1.5). The tensile properties with treatment time 30 minutes increase in the following order Neat CFRC< GPS(1.5)< GPS(2.5)< APS(2.5)< APS(1)< GPS(1)< APS(1.5). These improvements in mechanical properties indicate that incorporation of silane coupling agent results in better adhesion between the fiber and the resin for carbon/epoxy composite. Composite containing carbon fiber treated with APS possesses better mechanical properties than GPS. This is attributed to the fact that APS contains amino function and amino functions interact better with epoxy thus producing better interfacial adhesion between the matrix and epoxy (Plueddemann 1990). Finite element analysis is done using ANSYS 15.0 software comparing the experimental deformation of the tensile tested samples and the simulated deformation. Results indicated that the experimental values match reasonably validating the experimental results.

5.2 Future Scope

- Various other methods of silane coupling agent treatments like spray-on method, integral blend method(addition of alkoxysilanes to dry-blends of polymer and filler prior to compounding) could be used.
- Comparisons could be done of the impact testing results of the composites.
- Hygrothermal effect on composite materials could be checked.
- Zirconate coupling agents can be a better substitute to silane coupling agents as they possess the capability of crosslinking the polymer matrix with the establishment of donor-acceptor interactions and hydrogen bonding between it and the epoxy particles, leading to very good interfacial adhesion (Vaz et al. 2002).

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Appendix-I

Araldite GY 257 resin data sheet

General- Araldite GY 257 is a low viscose epoxy resin based on Bisphenol A modified with an aromatic glycidyl ether.

Applications- Araldite GY 257 can be cured with polyamines, polyamidoamines or their adducts for solvent-free coatings, self-leveling and mortor floors, concrete injections, structure adhesives, trowelling compounds and impregnation systems. In combination with unmodified epoxy resins, like Araldite GY 250 and Araldite GY 6010 the viscosity of the end formulation can be reduced.

Properties- Due to the low viscosity and the full crystallization resistance Araldite GY 257 is a most suitable epoxy resin in formulating solvent free systems yielding,

- Very good processing properties
- Good mechanical performances
- Good surface penetration
- Medium chemical resistance / good acid and solvent resistance
- Appearance Clear, no contamination
- Viscosity at 25 °C –(500 – 700) mPa.s
- Density at 25°C- 1.15 g/cm^3
- Flash point ≥ 138 °C

Aradur HY 837 hardener

General- H+ equivalent weight is 68. Mix Ratio with GY 6010 is 35. It is an medium viscosity liquid amine adduct.

Applications- HY 837 is suitable for the formulation of two-component wood, concrete and metal coatings, linings and flooring. An accelerator for polyamides.

Properties-

- Fast cure
- Excellent adhesion

- Good chemical resistance
- Viscosity at 25 °C –(2,700 – 3,700) mPa.s
- Density at 25°C- 0.98 g/cm^3
- Flash point ≥ 138 °C

Appendix –II

Unidirectional carbon fiber properties

Technical data of fiber	430 gsm
Modulus of elasticity	230 kN/mm^2
Tensile strength	4900 N/mm^2
Weight of carbon fiber (main direction)	400 g/m^2
Density	1.8 g/cm^3
Thickness for static design weight / density	0.25 mm