

Failure analysis of pin joints in carbon-epoxy nanoclay composite laminates

Dissertation

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

Master of Engineering

in

CAD/CAM Engineering

Submitted

by

Rohit Badhwar
(Roll No. 801281018)

Under Supervision of

Dr. Jaswinder Singh Saini
Assistant Professor
Mechanical Engineering Department

Dr. Haripada Bhunia
Associate Professor
Chemical Engineering Department



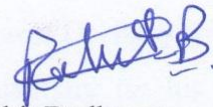
**Mechanical Engineering Department
Thapar University
Patiala-147004, Punjab**

July 2014

Certificate

This is to certify that the dissertation entitled “**Failure analysis of pin joints in carbon-epoxy nanoclay composite laminates**”, is an authentic record of my own work carried out as requirements for the award of degree of Master of Engineering in CAD/CAM Engineering from Thapar University, Patiala, under the guidance of Dr. Haripada Bhunia (Associate Professor, Chemical Engineering Department) and Dr. Jaswinder Singh Saini (Assistant Professor, Mechanical Engineering Department).

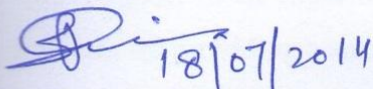
Date: 18/07/2014



Rohit Badhwar

(Roll No: 801281018)

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

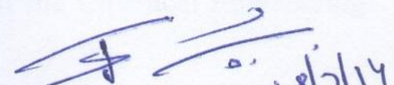


(Dr. Haripada Bhunia)

Associate Professor

Chemical Engineering Department

Thapar University, Patiala



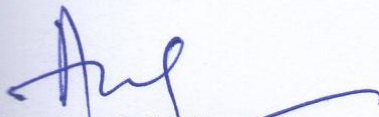
(Dr. Jaswinder Singh Saini)

Assistant Professor

Mechanical Engineering Department

Thapar University, Patiala

Countersigned by:



(Dr. Ajay Batish)

Head, Mechanical Engineering Department

Thapar University, Patiala



(Dr. S. K. Mohapatra)

Dean of Academic Affairs

Thapar University, Patiala

Acknowledgement

My sincere gratefulness goes to both of my supervisors Dr. Haripada Bhunia and Dr. J.S. Saini for their help, support and understanding, financially and emotionally, that made it possible for me to be successful in the initiation and finalization of this project. I am thankful for their encouragement to keep going and to constantly learn new things.

Specially, I would take this opportunity to thank Dr. Haripada Bhunia, for guiding me and correcting my mistakes along the way. I would also like to thank, Dr. J.S. Saini for his guidance regarding the basic principles. I would like to extend my gratitude towards Dr. Ajay Batish, Head and Professor, Department of Mechanical Engineering, Thapar University for his support. I would also like to thank the members of the labs I visited and worked in for helping me with my experiments and for providing intelligent insights that made my work easier, thank you for being so friendly. I would like to thank Mr. Manjeet Singh Sekhon (Research Scholar in Mechanical Engineering Department) and Mr. Gaurav Madhu (Research Scholar in Chemical Engineering Department) for their invaluable inputs regarding the finer parts of my work. I thank my friends Mr. Mandeep Singh and Mr. Gaurav Singla for helping me out with the smallest of things and for their support in all experimental work and the staff members of the Chemical Engineering Department for providing a suitable environment to work in.

I would also like to thank and acknowledge **Atul Pvt. Ltd, Gujarat** for supplying us with Epoxy, Hardener and Accelerator. I would also like to thank and acknowledge **AURO Carbon & Chemicals, Vadodara** for supplying us with Unidirectional carbon fabric used as reinforcing agent. Also I would like to thank and acknowledge **Connell Bros. Company Pvt. Ltd., Mumbai**, for supplying us with nanoclay Cloisite 30B used as reinforcing agent.

Finally, I would like to express my utmost gratitude to my parents, for their unconditional affection and support.

Rohit Badhwar

Abstract

Fiber reinforced composite materials have been gaining wide application in aircraft submarine and spacecraft constructions. These applications require joining composites either to composites or to metals. Most commonly, joints are formed using mechanical fasteners. Therefore, suitable revealing methods for the failure strength would help in selecting the appropriate joint size in a given application. The objective of the present study is to investigate the effect of joint geometry on strength of pin-loaded carbon-epoxy nanoclay nanocomposite laminates experimentally and numerically. Composite laminates were prepared using press moulding technique at 150⁰C. Specimens were made according to ASTM standards. Geometric parameters that were investigated during analyses; the distance from the free edge of plate to the diameter of the first hole (E/D) ratio, width of the specimen to the diameter of the holes (W/D) ratio. Both the experimental and analytical results were compared. The results showed that the pin hole farthest from the free edge is subjected to the highest stress. It can also be seen that geometric parameters such as E/D and W/D ratios are crucial for pinned laminated composite joints.

Contents

Certificate	i
Acknowledgement	ii
Abstract	iii
Contents	iv
List of Figures	vi
List of Tables	vii
Chapter 1 Introduction	1
1.1 Introduction to composites	1
1.1.1 Matrix Phase	2
1.1.2 Reinforcement Phase	2
1.2 Classification	3
1.2.1 Classification with respect to Matrix constituent	3
1.2.2 Classification with respect to Reinforcement constituent	9
1.3 Advantages and Disadvantages of Composites	13
1.4 Joints in composite structures	14
Chapter 2 Literature Review	17
2.1 Literature Review	17
2.2 Problem Formulation	21
2.3 Objectives	21
Chapter 3 Experimentation	22
3.1 Materials	22
3.1.1 Resin	22
3.1.2 Carbon Fabric	23
3.1.3 Nanoclay	24

3.2 Methods	24
3.2.1 Material processing and sample preparation	24
3.3 Testing	26
3.3.1 Tensile Test	26
3.3.2 Shear Test	27
3.3.3 Flexural Test	27
3.4 Pin Joint Geometry	29
Chapter 4 Results and Discussions	32
4.1 Mechanical properties of composite laminates	32
4.2 Flexural Test	33
4.3 Test results of carbon/epoxy pin joints configuration for unfilled and filled samples	34
4.3.1 Results of neat carbon/epoxy composite laminates	34
4.3.2 Results of single pin joint carbon/epoxy nanocomposites at 3% by wt. of nanoclay	36
4.4 Numerical analysis	39
4.5 Comparison of Finite Element Analysis and Experimental results	42
Chapter 5 Conclusion and Future Scope	44
5.1 Conclusions	44
5.2 Future Scope	44
References	45

List of Figures

Figure No.	Title	Page No.
Figure 1.1	Classification with respect to the Matrix constituent	4
Figure 1.2	Various types of Thermosets	5
Figure 1.3	Types of Thermoplastics	6
Figure 1.4	Classification with respect to reinforcement constituent	9
Figure 1.5	Common failure modes in composite plates	16
Figure 3.1	Carbon Fabric	23
Figure 3.2	Geometry of the Iosipescu shear test specimen	27
Figure 3.3	Iosipescu test fixture	27
Figure 3.4	Geometry of composite plate with hole	29
Figure 4.1	Young's modulus vs. weight percentage of nanoclay	32
Figure 4.2	Flexural strength versus nanoclay wt.% plots	33
Figure 4.3	Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=5	34
Figure 4.4	Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=4	35
Figure 4.5	Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=3	35
Figure 4.6	Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=2	36
Figure 4.7	Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=5	37
Figure 4.8	Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=4	37
Figure 4.9	Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=3	38
Figure 4.10	Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=2	38
Figure 4.11	Geometric modelling and meshing	39
Figure 4.12	Loading and boundary conditions	40
Figure 4.13	Stress distribution around hole at nanoclay 0% for W/D=4 and E/D=4	41
Figure 4.14	Stress distribution around hole at nanoclay 3% for W/D=4 and E/D=4	41
Figure 4.15	Experimental vs. FEA results of pin joints at nanoclay 0% and 3%	42
Figure 4.16	Experimental vs. FEA results of pin joints at nanoclay 0% and 3%	42
Figure 4.17	Experimental vs. FEA results of pin joints at nanoclay 0% and 3%	43
Figure 4.18	Experimental vs. FEA results of pin joints at nanoclay 0% and 3%	43

List of Tables

Table No.	Title	Page No.
Table 3.1	Physical properties of resin	22
Table 3.2	Processing properties of resin	22
Table 3.3	Mechanical properties of resin	23
Table 3.4	Mechanical properties of unidirectional Carbon fabric	23
Table 3.5	Physical properties of unidirectional carbon fabric	24
Table 3.6	Physical properties of Cloisite 30B	24
Table 3.7	Formulation and geometrical parameters of carbon/epoxy nanoclay laminates	26
Table 3.8	Properties and ASTM standards used	28
Table 3.9	Geometric parameters of pin joint samples tested	29
Table 4.1	Mechanical properties of carbon fiber/epoxy composite laminates	32
Table 4.2	Mechanical properties of carbon fiber/epoxy nanoclay composite laminates	33

1.1 Introduction to composites

Composites are specially made materials constituting two or more constituent materials with significantly different physical or chemical properties and the two constituting phases remain separate and distinct on a macroscopic level within the finished composite structure.

Composite Materials exhibit excellent physical and chemical properties thereby getting high attention for their use as engineering materials. These materials may replace the traditional engineering materials in the near future due to their remarkable properties. Modern composite materials are generally optimized to achieve desired properties for a given range of applications. Because a very vast range of materials may be considered as composites and there is a broad range of applications for which composite materials may be designed, so it is quite difficult to agree upon a single, simple, and useful definition. However, in general, composite materials may be defined as those materials that contain a continuous matrix constituent that binds together a stronger, stiffer reinforcement constituent. Hence the resulting composite material has a balance of structural properties of constituent materials. In composites the improved structural properties generally result from a load-sharing mechanism between matrix and reinforcement phases.

Generally, in composites desired strength and other properties come from reinforcement phase with matrix phase being soft and ductile. But it is not a thumb rule, there are exceptions that may still be considered composites, such as rubber-modified polymers, where the reinforcement phase is more compliant and more ductile than the polymer, resulting in improved toughness. Matrix phase is also very significant because ultimately it is the matrix phase which provides continuity and uniform hold to the stiffer and stronger reinforcement phase. Similarly, steel wires have been used to reinforce gray cast iron in truck and trailer brake drums.

Matrix phase is a continuous phase that surrounds and supports the reinforcement materials by maintaining their relative positions in the combination. The reinforcements impart their special mechanical and structural properties to enhance the matrix properties. These two categories of

constituent materials are also called two phases of composites. These two phases are given below:-

1.1.1 Matrix Phase

Matrix phase is the primary phase, a continuous one usually more flexible and less harder than the reinforcement. It holds the dispersed phase and shares a load with it. The various functions associated with the matrix phase are given below:-

- (i) To hold the reinforcement material together.
- (ii) To protect the reinforcement material from external corrosive environment.
- (iii) To distribute the loads evenly between the fibers so that all fibers are subjected to same amount of strain in all directions.
- (iv) It enhances transverse properties of laminates.
- (v) To improve impact and fracture resistance of the component.
- (vi) To carry inter-laminar shears.

Desired properties of a matrix material:-

- (i) Less moisture absorption.
- (ii) Low shrinkage.
- (iii) Small coefficient of thermal expansion.
- (iv) Elasticity to transfer load to fibers.
- (v) Strength at elevated temperatures (depending upon application).
- (vi) Resistance to chemical corrosion.
- (vii) Dimensional stability.

1.1.2 Reinforcement Phase

This is the secondary phase (or dispersed phase) which is imbedded in the matrix in a discontinuous form. This phase is usually stronger than the matrix, therefore it is sometimes called reinforcing phase. Various functions of reinforcement are given below:-

- (i) To provide strength to the composite.
- (ii) It provides stiffness.

- (iii) To give shape to the composite material.
- (iv) To enhance the properties that of the matrix material.

Desired properties of the reinforcements are given below:-

High stiffness and stronger than the matrix phase.

- (i) Good thermal and electrical conductivity.
- (ii) Excellent wear resistance.
- (iii) Low coefficient of thermal expansion than matrix.

1.2 Classification

Composites are commonly classified with respect to Matrix and Reinforcement constituents.

1.2.1 Classification with respect to Matrix constituent

Composites can be classified based on the matrix phase as follows:-

- (i) Organic-Matrix Composites (OMCs).
- (ii) Metal-Matrix Composites (MMCs).
- (iii) Ceramic-Matrix Composites (CMCs).

(i) Organic-Matrix Composites (OMCs)

Organic-matrix composites are the ones having organic material based matrix phase. These include two classes of composites:

Polymer-Matrix Composites (PMCs) and Carbon-Matrix Composites (commonly referred to as Carbon-Carbon Composites). There are two extra steps of carbonizing and densifying to make Carbon-matrix composites from PMCs.

In the research and development community, Inter-metallic Matrix Composites (IMCs) are sometimes listed as a classification that is distinct from MMCs. However, significant commercial applications of IMCs do not yet exist, and in a practical sense these materials do not provide a radically different set of properties relative to MMCs. In each of these systems, the matrix is

typically a continuous phase throughout the component. Fig.1.1 shows the classification of the composite with respect to the matrix.

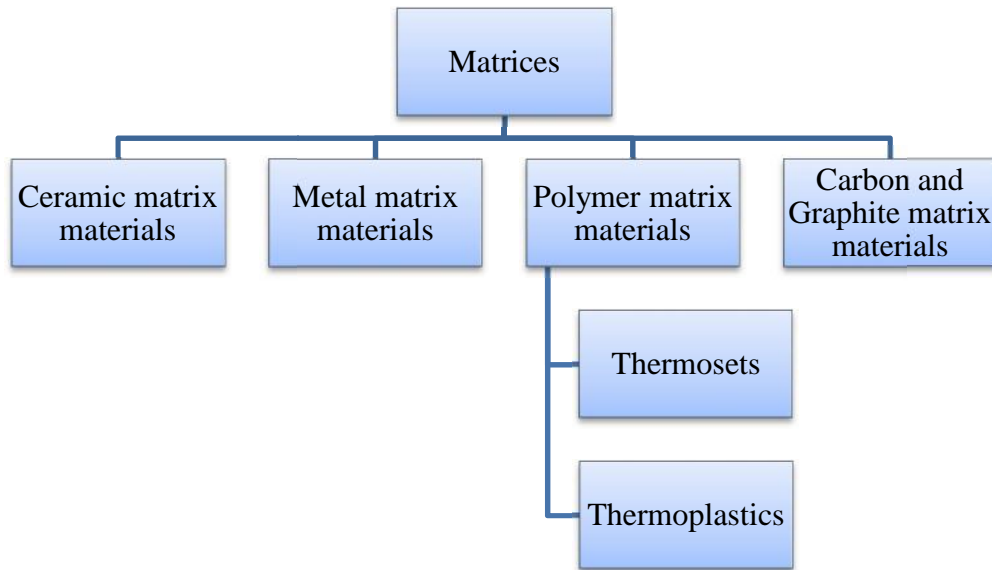


Fig. 1.1 Classification with respect to the matrix constituent

- **Polymer Matrix Materials**

Polymers possess excellent properties such as high strength, lightweight and can be processed easily so they are ideal materials for certain desirable mechanical applications. There are two main kinds of polymers : thermosets and thermoplastics.

(a) Thermosets

Thermosets are materials that decompose instead of melting on hardening and have qualities such as a well-bonded three-dimensional molecular structure after curing. Only changing the basic composition of the resin is sufficient to alter the conditions suitably for curing. They can be retained in a semi-cured condition too over prolonged periods of time, rendering thermosets very flexible. Hence, they are most suited as matrix bases for advanced conditions fiber reinforced composites. They 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 is the starting material as in epoxy, polymer and phenolic polyamide resins.

Thermosets are the most popular of the fiber composite matrices without which, research and development in structural engineering field could get truncated. Aerospace industry components,

automobile parts, defense systems etc., use a great deal of this type of fiber composites. Epoxy matrix materials are used in printed circuit boards and similar areas. Fig.1.2 shows some kinds of thermosets.

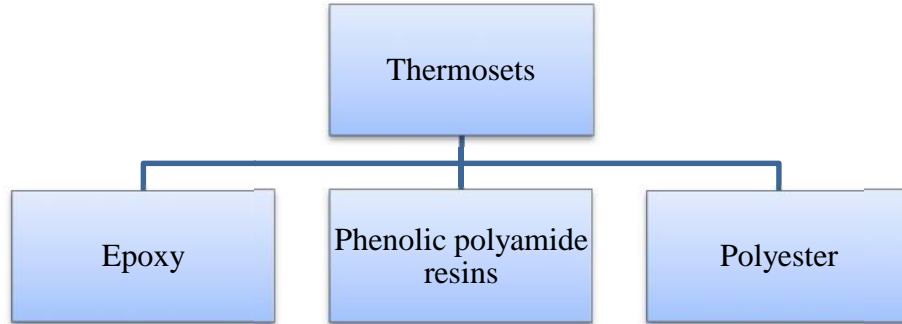


Fig. 1.1 Various types of Thermosets

Rearrangement reactions to form heterocyclic entities after direct condensation polymerization is the method generally used to produce thermoset resins. Water, a product of the reaction, in both methods, hinders production of void-free composites. Voids have a negative effect on properties of the composites in terms of strength, dielectric properties etc. Polyesters phenolic and epoxies are the two important classes of thermoset resins.

Epoxy resins find use in filament-wound composites. These are reasonably stable to chemical attacks and are good adherents having slow shrinkage during curing and no emission of volatile gases. But for these advantages, make the use of epoxies rather expensive. Also, they cannot be raised beyond a temperature of 140°C. They can't be used in high technology areas where service temperatures are higher.

Polyester resins on the other hand are quite easily accessible, cheap and are used in a wide range of fields. Liquid polyesters are stored at room temperature for very long time 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 are stable against chemicals. Depending on the formulation of the resin or service requirement of application, they can be used up to about 75°C or higher. Other advantage of polyesters is easy compatibility with few glass fibers. 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 200-250°C.

(b) Thermoplastics

Thermoplastics are materials that tend to melt at an elevated temperature and show exaggerated melting point and have one or two-dimensional molecular structure. Advantage is that the process of softening at elevated temperatures can be reversed to regain its properties during cooling, facilitating applications such as conventional compression techniques to mould the compounds. Fig.1.3 shows the types of thermoplastics.

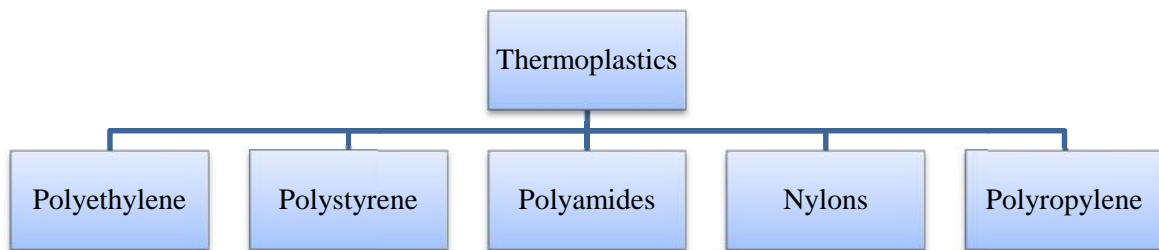


Fig. 1.2 Types of Thermoplastics

A small shrinkage and the tendency of the shape to retain its original form are also the factors to be accounted for. But reinforcements can alter this condition also. The advantage of thermoplastics systems over thermosets are that there are no chemical reactions involved, which often result in the release of gases or heat. Manufacturing is controlled by the time required for heating, shaping and cooling the structures. Thermoplastics resins are sold as moulding compounds. Fiber reinforcement is quite appropriate for these resins. Since the fibers are randomly dispersed, so the reinforcement will be almost isotropic. However, when subjected to moulding processes, they can be aligned directionally.

There are some options to increase heat resistance in thermoplastics. Addition of fillers also raises the heat resistance. But all thermoplastic composites tend to lose their strength at elevated temperatures. But their redeeming qualities like toughness, rigidity and ability to resist creep, place thermoplastics in the important composite materials bracket. They are used in automotive control panels, electronic products encasement etc.

Newer developments are broadening the scope of applications of thermoplastics. Huge sheets of reinforced thermoplastics are now available and they just require sampling and heating to be moulded into the required shapes. This has facilitated easy fabrication of bulky components, doing away with the more cumbersome moulding compounds.

- **Carbon Matrices**

Carbon and graphite find a special place in composite materials options, both being highly superior, strong and rigid at high temperature up to 2300°C. This Carbon-Carbon composite is fabricated by compaction of carbon or multiple impregnations of porous frames with liquid carbonizer precursors and subsequent pyrolysis. They can also be fabricated by chemical vapour deposition of pyrolytic carbon. Carbon-Carbon composites are not to be applied at elevated temperatures, as many composites have proved to be far superior at these temperatures. Moreover, their capacity to retain their properties at room temperature as well as at temperature in the range of 2400°C and their dimensional stability make them the obvious choice in a number of applications related to aeronautics, industry, military and space. Components, that are exposed to higher temperature and from which the demands for high standard performance are many, are most likely to have Carbon-Carbon composites used in them.

- **Glass Matrices**

Glass matrices are found to be more reinforcement-friendly. Glasses are meant to improve upon performance of several applications. Glass matrix composite with high strength and modulus can be obtained and they can be maintained upto temperature of the order of 650°C.

Composites with glass matrices are considered superior in dimensions to polymer or metal system, due to their low thermal expansion behaviour. This property allows fabrication of many components in intricate shapes along with their special tribological characters.

- (i) **Metal Matrix Materials**

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 are 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 mentioned reasons are characterized by high moduli.

Most metals and alloys make good matrices. But practically, the choices for low temperature applications are not many. Only light metals are responsive, with their low density proving an advantage. Titanium, Aluminium and magnesium are the popular matrix metals currently in vogue, which are particularly useful for aircraft applications. If metallic matrix materials have to offer high strength, they require high modulus reinforcements. The strength-to-weight ratios of resulting composites can be higher than most alloys.

The melting point, physical and mechanical properties of the composite at various temperatures determine the service temperature of composites. Most metals, ceramics and compounds can be used with matrices of low melting point alloys. The choice of reinforcements becomes more stunted with increase in the melting temperature of matrix materials.

(ii) Ceramic Matrix Materials

Ceramics are solid materials which exhibit very strong ionic bonding in general and in few cases covalent bonding. High melting points, good corrosion resistance, thermal stability and high compressive strength, render ceramic-based matrix materials a favourite for applications requiring a structural material that doesn't give way at temperatures above 1500°C. Naturally, ceramic matrices are a preferred choice for high temperature applications.

High modulus of elasticity and smaller tensile strain, which most ceramics possess have caused the failure of attempts to add reinforcements to obtain strength improvement. It is because at the stress levels at which ceramics rupture, there is insufficient elongation of the matrix which doesn't allow matrix to transfer an effective quantum of load to the reinforcement and the composite may fail unless and until the percentage of fiber volume is high enough. It is seen that addition of high-strength fiber to a weaker ceramic has not always been successful and often the resultant composite has proved to be weaker. The use of reinforcement with high modulus of

elasticity may solve the problem to some extent, also pre-stressing of the fiber in the ceramic matrix is being increasingly resorted to as an option.

It is observed that when ceramics have a higher thermal expansion coefficient than reinforcement materials, the resultant composite is likely to have a inferior level of strength. In such a case, the composite will develop strength within ceramic at the time of cooling which results in micro-cracks extending from fiber to fiber within the matrix. Micro-cracking may result in a composite with tensile strength lower than that of the matrix.

1.2.2 Classification with respect to Reinforcement constituent

Reinforcements for the composites can be fibers, fabrics, particles or whiskers as shown in Fig.1.4. Fibers are essentially characterized by one very long axis with other two axes either often circular or near circular. Particles have no preferred orientation and so does their shape. Whiskers have a preferred shape but are small both in diameter and length as compared to fibers.

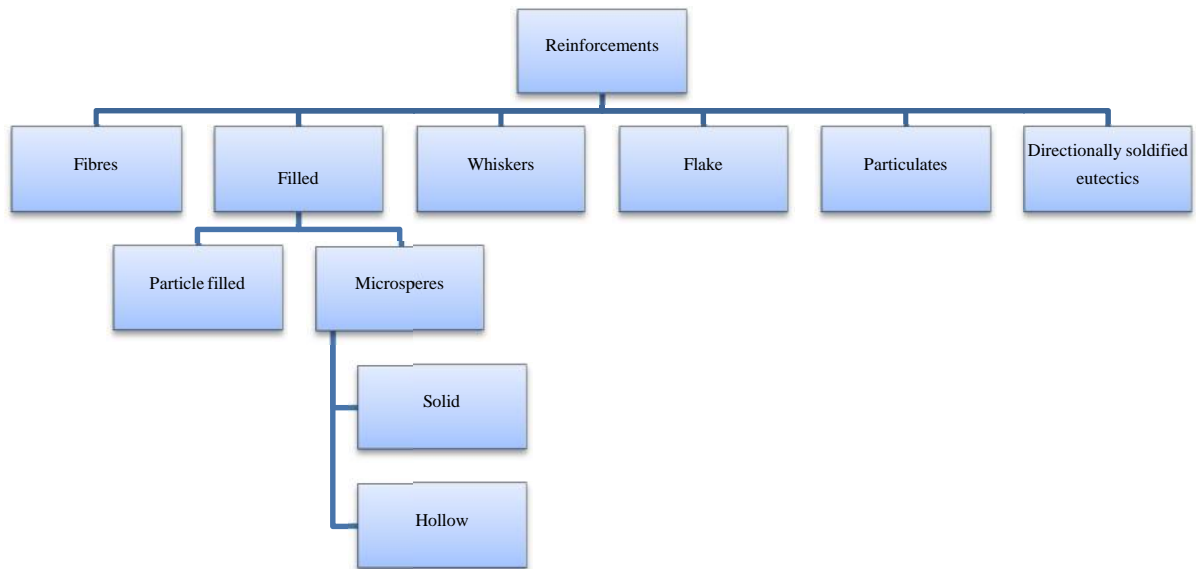


Fig. 1.3 Classification with respect to Reinforcement constituent

(i) Fiber Reinforcement

Fibers are an important class of reinforcements, as they satisfy the desired conditions and transfer strength to the matrix constituent influencing and enhancing their properties as desired.

Fibers fall short of ideal performance due to several factors. The performance of a fiber composite is judged by its size, shape, orientation and composition of the fibers and the mechanical properties of the matrix material. The orientation of the fiber in the matrix is an indication of the strength of the composite and the strength is greatest along the longitudinal directional of fiber. This does not mean that the longitudinal fibers can take the same quantum of load irrespective of the direction in which it is applied. Maximum performance from longitudinal fibers can be obtained if the load is applied along its direction. A little shift in the angle of loading may drastically reduce the strength of the composite.

Unidirectional loading is found in few structures and hence it is prudent to give a mix of orientations to fibers in composites particularly where the load is expected to be the heaviest. The strength of the fiber in any one of the three axes would, therefore be one-third in the unidirectional fiber composite, assuming that the volume percentage is equal in all three axes. However, orientation of short fibers by different methods is also possible like random orientations by sprinkling them on to given plane or addition of matrix in liquid or solid state before or after the fiber deposition. Even three-dimensional orientations can be achieved in this way.

There are several methods of random fiber orientations, which in a two-dimensional one, yield composites with one-third of the strength of a unidirectional fiber-stressed composite, in the direction of fibers. In 3D, it would result in a composite with a comparable ratio, about less than one-fifth. Most of the fibers in use currently are solids which are easy to produce and handle, having a circular cross-section, although a few non-conventional shaped and hollow fibers show signs of capabilities that can improve the mechanical properties of the composites.

Given the fact that a vast difference in length and an effective diameter of the fiber are assets to a fiber composite, it follows that greater strength in the fiber can be achieved by smaller diameters due to minimization or total elimination of surface defects.

Various types of fibers that are used to reinforce composite materials may be organic and inorganic. Organic fibers have low density, flexibility and elasticity where as inorganic fibers are of high modulus of elasticity, high thermal stability and posses greater rigidity than organic fibers.

(ii) Laminar Composites

Laminar composites can be described as materials comprising of layers of materials bonded together. These may be of several layers of two or more metal materials occurring alternately or in a determined order more than once and as many times as required for a specific purpose. They are found in as many combinations as the number of materials.

Powder metallurgical processes like roll bonding, hot pressing, diffusion bonding, brazing etc. can be employed for the fabrication of different alloys of sheet, foil, powder or sprayed materials. But sheets and foils can be made isotropic in two dimensions more easily than fibers. Sheets and foils are also made to exhibit high percentages of material of which they are made. For example, a strong sheet may use over 92% in laminar structure, while it is difficult to make fibers of such compositions. The main functional types of metal-metal laminates that do not possess high strength or stiffness are single layered ones that enrich the composites with special properties, apart from being cost-effective.

There are various combinations of sheet and foil which function as adhesives at low temperatures. Such materials, plastics or metals, may be clubbed with a third constituent. Pre-painted or pre-finished metals whose primary advantage is elimination of final finishing by the user are the best known metal-organic laminates. Several combinations of metal-plastic, vinyl-metal laminates, metal and organic films, account for upto 95% of metal-plastic laminates known. They are made by adhesive bonding processes.

(iii) Flake Composites

Flakes can be used in place of fibers as they can be densely packed. Metal flakes if are in close contact with each other in polymer matrices can conduct electricity or heat, while mica flakes and glass can resist both. Flakes are inexpensive to produce and usually cost less than fibers.

But they fall short of expectations in aspects like size control, shape defects in the end product. Glass flakes tend to have notches or cracks around the edges, which weaken the final product. They are also resistant to be lined up parallel to each other in a matrix, causing uneven strength. They are usually set in matrices, or more simply, held together by a matrix with a glue-type binder. Depending on the end-use of the product, flakes are present in small quantities or occupy the whole composite.

Flakes have various advantages over fibers in structural applications. Parallel flakes filled composites provide uniform mechanical properties in the same plane as the flakes. While angle-plying is difficult in continuous fibers which need to approach isotropic properties, it is not so in flakes. Flake composites have a higher theoretical modulus of elasticity than fiber reinforced composites. They are relatively cheaper to produce and can be handled in small quantities.

(iv) Filled Composites

Filled composites result from addition of filler materials to plastic matrices to replace a portion of the matrix, enhance or change the properties of the composites. The fillers also enhance strength and reduce weight. Another type of filled composite is the product of structure infiltrated with a second-phase filler material. The skeleton could be a group of cells, honeycomb structures, like a network of open pores. The infiltrant could also be independent of the matrix and yet bind the components like powders or fibers, or they could just be used to fill voids. Fillers produced from powders are also considered as particulate composite. In the open matrices of a porous or spongy composite, the formation is the natural result of processing and such matrices can be strengthened with different materials. Metal impregnates are used to improve strength or tolerance of the matrix. Metal casting, graphite, powder metallurgy parts and ceramics belong to this class of filled composites. In the honeycomb structure, the matrix is not naturally formed, but specifically designed to a predetermined shape. Sheet materials in the hexagonal shapes are impregnated with resin or foam and are used as a core material in sandwich composites.

Fillers may be the main ingredient or an additional one in a composite. The filler particles may be irregular structures, or have precise geometrical shapes like polyhedrons, short fibers or spheres. While their purpose is far from adding visual embellishment to the composites, they occasionally impart colour or opacity to the composite which they fill. The final composite properties can be affected by the shape, surface treatment, blend of particle types, size of the particle in the filler material and the size distribution.

Filled plastics tend to behave like two different constituents. They do not alloy and accept the bonding. They are meant to develop mutually; they don't react chemically with each other. It is vital that the constituents remain in co-ordination and do not destroy each other's desired

properties. The benefits offered by fillers include increase stiffness, thermal resistance, stability, strength and abrasion resistance, porosity and a favorable coefficient of thermal expansion. However, the methods of fabrication are very limited and the curing of some resins is greatly inhibited. They also shorten the life span of some resins and are known to weaken a few composites.

(v) Particulate Reinforced Composites

Microstructures of metal and ceramics composites, which show particles of one phase scattered in the other, are known as particle reinforced composites. Square, triangular and round shapes of reinforcement are known, but the dimensions of all their sides are observed to be more or less equal. The size and volume concentration of the dispersoid distinguishes it from dispersion hardened materials.

The dispersed size in particulate composites is of the order of a few microns and volume concentration is greater than 28%. The difference between particulate composite and dispersion strengthened ones is, thus, oblivious. The mechanism used to strengthen each of them is also different. The dispersed in the dispersion-strengthen materials reinforces the matrix alloy by arresting motion of dislocations and needs large forces to fracture the restriction created by dispersion.

Three-dimensional reinforcement in composites offers isotropic properties, because of the three systematical orthogonal planes. Since it is not homogeneous, the material properties acquire sensitivity to the constituent properties, as well as the interfacial properties and geometric shapes of the array. The composite's strength usually depends on the diameter of the particles, the inter-particle spacing, and the volume fraction of the reinforcement. The matrix properties influence the behaviour of particulate composite too.

1.3 Advantages and Disadvantages of Composites

Various advantages of the composites are as follows:-

- High resistance to fatigue and corrosion degradation.
- High strength to weight ratio.
- Due to greater reliability, there are fewer inspections and structural repairs.

- Fiber to fiber redundant load path.
- Improved dent resistance is normally achieved. Composite panels do not sustain damage as easily as thin gage sheet metals.
- High resistance to impact damage.
- Thermoplastics have rapid process cycles, making them attractive for high volume commercial applications that traditionally have been the domain of sheet metals. Moreover, thermoplastics can also be reformed.
- Like metals, thermoplastics have indefinite shelf life.
- Composites are dimensionally stable *i.e.* they have low thermal conductivity and low coefficient of thermal expansion.
- Manufacture and assembly are simplified because of part integration (joint/fastener reduction) thereby reducing cost.
- Close tolerances can be achieved without machining.
- Improved friction and wear properties.

Along with advantages, composites have various disadvantages also, which are as follows:-

- High cost of raw material and fabrication.
- Composites are more brittle than that of metals and thus are more easily damaged.
- The matrix material used is weak, which leads to low toughness.
- The disposal and reuse of the matrix material is very difficult.
- Composites are difficult to attach.
- The repair work of the composites is not favorable, because it can arise some more problems.
- The analysis of the composite materials is difficult as compare to that of metals.
- The matrix material of the composite is subjected to environmental degradation.

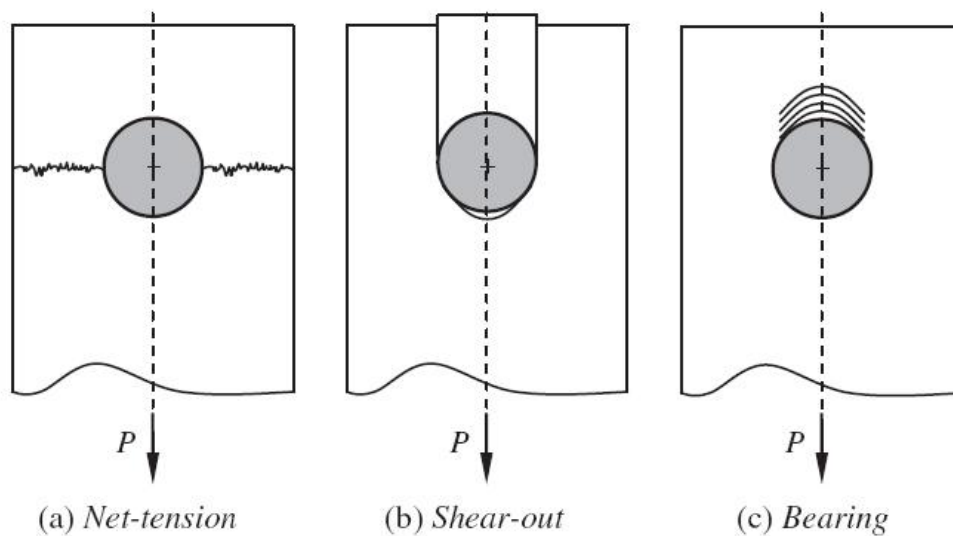
1.4 Joints in composite structures

In the last decade, composite materials are being commonly used in structures that demand a high level of mechanical performance. Their high strength to weight and stiffness to weight ratios has facilitated the development of lighter structures, which often replace conventional

metal structures. Fiber-reinforced composites offer the most reliable engineering materials in automotive, marine and aircraft industrial engineering applications due to their outstanding mechanical properties, like impact resistance, high durability, low coefficient of friction and thermal expansion, ability to provide higher load bearing capability in the directions of high loading, flexibility in design capabilities and relatively low density.

The applications usually require the joining of composites either to other composites or to metals. Unfortunately, it is rarely possible to produce a construction without joints due to limitations on material size, convenience in manufacture or transportation and the need for access. But these joints are usually weak points in structures due to complicated stress field in the vicinity of the hole. They are susceptible to high stress concentrations which occur around and in the vicinity of the hole and are often the cause of unexpected failure in composite structures containing joints [Aluko (2011)]. The high stress concentrations reduce the efficiency of the joint along with the redistribution of stresses near the discontinuities. Because of the complicated stress field near the hole area, they often causes a reduction of load capacity of composite structure. Further, in pursuit of increasing efficiency of the structure, the operational load continues to grow, the load carried by each fastener increases accordingly. This increases probability of failure.

The literature shows that there are five basic failure modes for composite mechanical joints: net tension, shear out, bearing, tear out and cleavage [Pisano *et al.* (2012)], shown in Fig. 1.5.



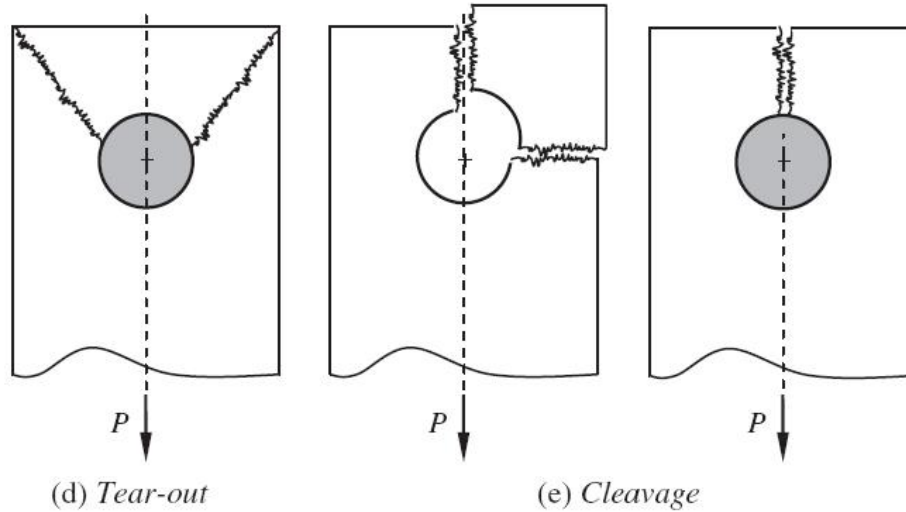


Fig. 1.5 Common failure modes in composite plates [**Pisano** *et al.* (2012)]

Studies show that the failure behaviours of mechanical joints can be affected by many parameters, such as the geometric dimensions, material properties, laminate lay-up, ply orientation, and fit, whether clearance or interference, between the pin and the hole and can be grouped together into following different types:

- i. Material parameters:** fiber types and form, resin type, fiber orientation, laminate stacking sequence, etc.
- ii. Geometry parameters:** specimen width (W) or ratio of width to hole diameter (W/D), edge distance (E) or ratio of the edge distance to hole diameter (E/D), specimen thickness (t), hole size (D), and pitch for multiple joints.
- iii. Fastener parameters:** fastener type, fastener size, clamping area and pressure, washer size and hole size and tolerance.
- iv. Design parameters:** loading type (tension, compression, fatigue, etc.), loading direction, joint type (single lap, double lap), geometry (pitch, edge distance, hole pattern etc.), environment and failure criteria.

The present work deals with the failure analysis of pin joint based on the geometry parameters *i.e.* specimen width to hole diameter (W/D) and edge distance to hole diameter (E/D), keeping the material parameters constant.

2.1 Literature Review

The following papers give the literature review of the work done by different researchers on pin joints using carbon fibers:

Ramazan and Murat (2002) investigated the failure strength and failure mode of a pinned-joint carbon–epoxy composite plate of arbitrary orientations. The main objective was to investigate the possibility of predicting the properties of the joint from the properties of the material measured with standard tests. The failure load and the failure mode were analysed numerically and experimentally. A computer code developed for such an analysis was used to calculate the failure load, the failure mode and the propagation of failure of plate with different fiber orientation, different material properties and different geometries. Hoffman and Hashin failure criteria were used in failure analysis. Experimental results concerning damage progression and ultimate strength of the joint were obtained and compared with predictions. A good agreement between experimental results and numerical predictions was obtained.

McCarthy *et al.* (2004) developed three-dimensional Finite Element models to study the effects of bolt–hole clearance on the mechanical behaviour of bolted composite (graphite/epoxy) joints. The joint type studied was single-bolt, single-lap, which is a standard test configuration in both a civilian and a military standard for composite joints. In this Part I of a two part paper the model was constructed in the non-linear Finite Element code, MSC Marc and attempts were made to validate it by comparing results with experiments on composite bolted joints. Issues in modelling the contact between the joint parts, which affect the accuracy and efficiency of the model were presented. Experimental measurements of surface strains and joint stiffness were compared with results from a Finite Element Parameter study involving variations in mesh density, element order, boundary conditions, analysis type and material modelling. The ability of the models to capture three-dimensional effects such as secondary bending and through-thickness variations in stress and strain was evaluated, and the presence of mathematical singularities in such models was highlighted. The validated model was used in Part II to investigate the effects of clearance on joint stiffness, stress state and failure initiation.

McCarthy et al. (2005) developed a three-dimensional finite element models to study the effects of bolt-hole clearance on the mechanical behaviour of single-bolt, single-lap bolted composite (graphite/epoxy) joints.

Yuan and Hoa (2007) fabricated Epoxy/clay nanocomposites (ENCs) from TGDDM epoxy and montmorillonite nanoclay. Carbon fiber reinforced epoxy/clay nano-composites (CFRENCs) were manufactured through hot melt lay-up plus autoclave process. The CFRENCs had uniform fiber volume fraction, very few dry spots, and very few resin rich areas. The interlaminar fracture toughness of CFRENCs was increased by 85% with the introduction of 4 phr nanoclay in epoxy. Small amount of nanoclay (2 phr) added into the epoxy of carbon/epoxy composites enhanced the flexural strength by 38%.

Spanos and Kontsos (2008) introduced a multi-scale Monte Carlo Finite Element Method (MCFEM) for determining mechanical properties of Polymer Nanocomposites (PNC) that consist of polymers reinforced with Single-Walled Carbon Nanotubes (SWCNT). Several approaches discussed in the literature suggest values for the mechanical properties of PNC that differ significantly from the corresponding ones derived by experimental procedures. The discrepancy was addressed by the proposed MCFEM which accounts for the effect of the non-uniform dispersion and distribution of SWCNT in polymers in the macroscopic mechanical behaviour of PNC. Specifically, the method had used a multi-scale homogenization approach to link the structural variability at the nano/micro scales with the local constitutive behaviour. Subsequently, the method incorporated a FE scheme to determine the Young's Modulus and Poisson Ratio of PNC. The use of the computed properties in macro-scale modelling was validated by comparison with experimental tensile test data.

Murat Pakdil (2009) studied the failure analysis of composite single bolted joints with effect to joint geometry and stacking sequence. Experimental studies were carried out by varying E/D and W/D ratio, in addition to bolt pretensions.

Akula and Garnich (2009) gave a brief summary of the major classes of failure criteria pertaining to the degradation models and was followed by a review of degradation models that had been developed for unidirectional polymer matrix composite laminates. The review was organized around the relationships of the various models to associated failure criteria as well as the various constitutive frameworks for finite element implementation. Models that invoke residual properties as a one-time sudden degradation of the original properties were described

followed by models where the mathematical representation of at least one property invoked gradual property degradation as a function of some other evolving field variable.

Wang et al. (2010) established a Finite Element Model of Carbon Fiber Reinforced Polymer (CFRP) pressure vessel with aluminum liner by ANSYS software package. The outer filament wound Fibers were overwrapped by both hoop winding and helical winding methods. The safety was vital because of high working pressure which was more than 35MPa. In the modelling process, the number of filament winding layers was treated as composite laminates with thickness and wrap angle variations of every unidirectional layer in the vessel section. The static analysis of the vessel was conducted. Based on the maximum stress criteria, burst pressure of the vessel was predicted. There was good agreement between model prediction and experimental data.

Liu et al. (2010) gave a comprehensive review on recent development of numerical simulation and optimization for the designed composite vessel. First, methods on damage modeling for predicting the failure properties and degradation mechanisms of the composite vessel were reviewed. Second, research on predicting the burst pressure and lifetime of the composite vessel was reviewed. The academic work on the damage modeling, progressive failure analysis and Finite Element implementation which explained the failure properties and stiffness degradation mechanisms of the composite vessel was summarized. Computational methods on the burst pressure, the strength reliability and lifetime of the composite vessel were also evaluated. Finally, ideal design which aims to lessen the weight of a composite vessel to the maximum extent under strength and stiffness constraints was commented. The optimization efficiency using different algorithms was also comparatively studied. The numerical simulation and optimization as important fundamental research had constituted a design platform for the composite vessel. It deserved pointing out the lightweight design conception as a remarkable tendency that combines advanced numerical methods and manufacturing technique developed rapidly, commits to improving the reliability and practicability of the composite vessel. It was expected that lightweight design technique played an increasingly important role in developing the composite vessel.

Limam et al. (2011) presented the limit analysis theory for determining the ultimate strength of a pin-loaded composite laminate. The analysis was compared with available experimental data for

composite pinned joints with varying E/D and W/D ratios and with available experimental data for riveted joints with varying the number of interfaces

Aluko (2011) developed an analytical method for predicting the bearing strength of pinned loaded composite joints using the Yamada-Sun failure criterion and characteristic curve model. The characteristic dimensions that define the characteristic curve were obtained by stress analysis associated with no-bearing and also without tensile tests of the plate unlike the conventional method that requires bearing test and tensile test of plates with and without hole.

Irisarri et al. (2012) presented a refined Finite Element modelling for strength prediction, and especially bearing strength prediction, of mechanically fastened joints in CFRP laminates. The model took into account the delamination onset and propagation by means of cohesive elements. The ply behaviour was described through a viscoelastic model combined with a progressive damage approach.

Chai et al. (2012) provided a thorough review of research work on fatigue and life prediction of fiber-reinforced composites. In order to summarize and present a comprehensive overview of the current state-of-the-art published works, the reviews in this contribution were broadly summarized into four groups of review:

(1) fatigue of fiber -reinforced composites, (2) composite damage mechanism, (3) composite failure criteria and (4) composite fatigue modelling and life prediction. The review began with a discussion of inherent and environmental factors affecting the fatigue of composites. This was followed by a rather extensive description of the composite damage mechanism and a summary of commonly used failure criteria for life prediction. And towards the end, models and methods for fatigue and life prediction of composites were summarized and discussed.

Ilic et al. (2012) developed a computational procedure to analyze initial failure load for pin-loaded holes at layered composite structures using Finite Element Methods. The influence of stacking sequences of layered composites containing pin-loaded holes was also investigated.

Zhang and Rowland (2012) developed a novel Finite Element based framework to study the effect of extreme temperatures on damage progression in carbon-fiber reinforced composite pin-joints. A progressive damage method that incorporated Hashin-type damage criteria was implemented in the model. The model was verified with a double-shear pin joint tensile test conducted at room temperature.

Zheng (2013) performed the progressive failure analysis using explicit Finite Element Method based on continuum damage mechanics to predict the failure properties and burst strengths of aluminium–carbon fiber/epoxy cylindrical laminate structures in terms of three composite pressure vessels with different geometry sizes. The failure analysis employed the Hashin damage initiation criterion and the fracture energy-based damage evolution law for composite layers. The numerical convergence problem was solved by introducing viscous damping effect into finite element equations for strain softening phenomenon. Effects of the calculation time and mesh sizes on the failure properties of composite laminates were explored. In addition, the predicted failure strengths of composite laminates using explicit Finite Element Analysis were also compared with experimental data and implicit Finite Element Analysis.

Turan et al. (2014) determined the failure modes and failure loads at pinned joint unidirectional laminated carbon/epoxy composite plates. Parametric studies were performed experimentally and numerically to evaluate the effects of joint geometry and fiber orientation on the failure loads and failure modes.

2.2 Problem Formulation

From the literature review it is seen that various parameters have to be study to find the strength of pin joints. Moreover very less amount of work is reported on the effect of addition of filler particles on the strength of pin joints in carbon composite laminates. Hence the present work deals with the development of nanocomposites using nanoclay (Cloisite 30B) as filler material. Thereafter the developed material is used to prepare the samples for the pin joints. These pin joints are then analyzed both experimentally and numerically.

2.3 Objectives

The specific objectives of the present work are:

1. Preparation of epoxy based unidirectional carbon fabric nanocomposite laminates.
2. Preparation of pin joint samples based upon optimized nanomaterial.
3. Failure analysis of single pin joints for unfilled and filled carbon epoxy nanocomposite laminates.

This section gives the details of different materials used, methodology followed and different apparatus used for the experimentation of the present work. The details of the materials are given below.

3.1 Materials

3.1.1 Resin

Materials used for the preparation of the laminates were epoxy, hardener, accelerator and carbon fabric. The epoxy used 'L-12' also known as Lapox is DGEBA (Di Glycidyl Ether of Bisphenol-A). K-12 hardener and K-13 accelerator were used. Resin *i.e.* (epoxy, hardener and accelerator) was supplied by Atul Ltd., Gujarat, India (www.atul.co.in). The properties of epoxy, hardener and accelerator are shown in Table 3.1, 3.2 and 3.3 respectively.

Table 3.1 Physical properties of resin

Description	Density(g/cm ³)	Viscosity(mPa.s)
Epoxy(L-12)	1.1-1.2	9000-12000
Hardener(K-12)	1.15-1.25	150-230
Accelerator(K-13)	0.88-0.92	<10

Table 3.2 Processing properties of resin

Property	Resin : Hardener : Accelerator	Initial Mix Viscosity	Minimum Curing Schedule
Unit	w/w	cPs	Hours
Value	100:100:0.1-2	450	120 ⁰ C/2h 160 ⁰ C/(1/2)h

Table 3.3 Mechanical properties of resin

Description	Specific Gravity	Tensile Strength (N/mm ²)	Elastic Modulus in Tension (N/mm ²)	Flexural Strength (N/mm ²)	Compressive Strength (N/mm ²)	Impact Strength (KJ/mm ²)	Co-efficient of Liner Thermal (°K ⁻¹)
L-12(100)+K-12(100)+K-13	1.80-1.85	70-90	15000-16000	100-120	190-210	4-7	28-32 x 10 ⁻⁶

3.1.2 Carbon Fabric

Unidirectional carbon fabric as shown in Fig.3.1 used as reinforcing agent was supplied by the AURO Carbon & Chemicals, Vadodara. It was 12K UD 214.7 gsm fabric. Mechanical and physical properties of unidirectional carbon fabric are shown in Table 3.4 and 3.5 respectively.



Fig. 3.1 Carbon Fabric

Table 3.4 Mechanical properties of unidirectional Carbon fabric

Description	Tensile strength (Kgf/Cm ²)	Tensile modulus in tension (Kgf/Cm ²)	Elongation at breaking load (%)
Carbon fabric	41863	2530000	2.28

Table 3.5 Physical properties of unidirectional carbon fabric

Description	Fiber type	Fiber Thickness(mm)	Area density (gsm)
Carbon fabric	12K UD	0.11	214.7

3.1.3 Nanoclay

Nanoclay Cloisite 30B was used and it was supplied by the Connell Bros. Company (India) Pvt. Ltd., Mumbai. Cloisite 30B is a natural montmorillonite modified with a quaternary ammonium salt. Cloisite 30B is an additive for plastics to improve various physical properties such as reinforcement are shown in Table 3.6.

Table 3.6 Physical properties of Cloisite 30B

Sr. no	Composition	Moisture	Typical dry particle size (μm)	Color	Packed bulk density (g/l)	Density (g/cm^3)	X ray results (nm)
1	Alkyl quaternary ammonium salt bentonite	<3%	<10	Off white	365	1.98	$d_{001} = 1.85$

3.1.4 Acetone

Acetone is an important solvent with molecular formula $(\text{C}_3\text{H})_2\text{CO}$ and has been used to concentrate the mixture of epoxy and nanoclay. It was supplied by the Loba Chemie Pvt. Ltd, Mumbai (<http://www.lobachemie.com/>).

3.2 Methods

3.2.1 Material processing and sample preparation

Laminates were prepared using DGEBA based epoxy resin as matrix and carbon fiber as reinforcement. There are several methods for the preparation of laminates but the one used in the present work was using compression moulding technique. After in depth study of the literature, the procedure for the laminates preparation is given below:

Addition of nanoclay in epoxy resin

Epoxy resin and nanoclay were mixed in the ratio as per the standard procedure. After the hardener and epoxy resin were properly mixed, accelerator was added to the solution to accelerate the process.

Stirring of epoxy-nanoclay mixture in homogeniser

After the nanoclay was added to the epoxy in required proportion, stirring with homogeniser was done for 15 minutes as per the procedure for proper mixing of the solution.

Stirring of epoxy-nanoclay mixture in sonicator after homogenisation

After the stirring with homogenizer, sonication of the mixture was done for 15 minutes for proper dispersion of nanoclay content in the epoxy resin.

Addition of hardener and accelerator in epoxy-nanoclay mixture

After sonication process keeping hardener to resin ratio 1:1, hardener and accelerator were added to the epoxy-nanoclay mixture. The mixture should be again kept stirred with homogeniser for 15 minutes for insuring proper mixing. In this way resin was prepared for laminated composites.

Formation of Laminate Layers

Carbon fabric were cut into the required sizes with a cutter. Placing the first layer of the carbon fiber on the Teflon sheet, resin was applied on it with a brush and then second layer was placed on it, following this procedure the laminate was formed with six layers of carbon fiber. The hand roller was used to remove any entrapped air particles between the layers so that the laminates can properly stick to each other. The formulations and geometric parameters of the laminates are given in Table 3.7.

Curing

After the laminate layers were prepared with the resin. Laminate are cured at room temperature for 24 hours so that they are properly bonded to each other with the help of compression moulding technique on hydraulic press.

Fabrication of Composite Plate

After the curing at room temperature, laminates were finally made with the hydraulic press. Teflon sheets were used to avoid sticking of the carbon fiber with the mould. Mould of square shape having cavity of required shape was used. Laminated plate was held at constant pressure of 120 MPa and temperature of 150 °C for two hours for the curing process and then the composite plate was cooled to room temperature at the same pressure. The thickness of the plate obtained was nearly 2mm.

Table 3.7 Formulation and geometrical parameters of carbon/epoxy nanoclay laminates

Sr. No.	Sample code	Fabric ply orientation	Number of plies	Weight fraction %		
				Resin	Fiber content	Nanoclay
1	RC0	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]	6	67.04	32.96	0
2	RC1	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]	6	59.64	39.76	0.60
3	RC2	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]	6	59.27	39.52	1.21
4	RC3	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]	6	58.91	39.27	1.82
5	RC5	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]	6	58.16	38.78	3.06

3.3 Testing

3.3.1 Tensile Test

The tensile tests were performed at 25 ± 2 °C on a Zwick-Roell Universal Testing Machine – Model Z010, Zwick-Roell, Germany, according to ASTM D3039 standard. The grip to grip separation of the samples having 138 mm gauge length was 238 mm at the start position. A crosshead speed of 2 mm/min was maintained. At least 3 specimens of each blend were tested and the average values were reported.

3.3.2 Shear Test

The shear tests were performed at 25 ± 2 °C on a Zwick-Roell Universal Testing Machine – Model Z010, Zwick-Roell, Germany, according to ASTM D5379 standard. A crosshead speed of 2 mm/min was maintained. At least 3 specimens of each blend were tested and the average values were reported.

The Iosipescu test specimen, shown in Fig.3.2, was tested using Iosipescu test fixture, shown in Fig. 3.3. The specimens were placed in Iosipescu test fixture in which the specimen was centered using the alignment pin and lightly clamped with the adjustable wedges. Then the load was applied to the specimen.

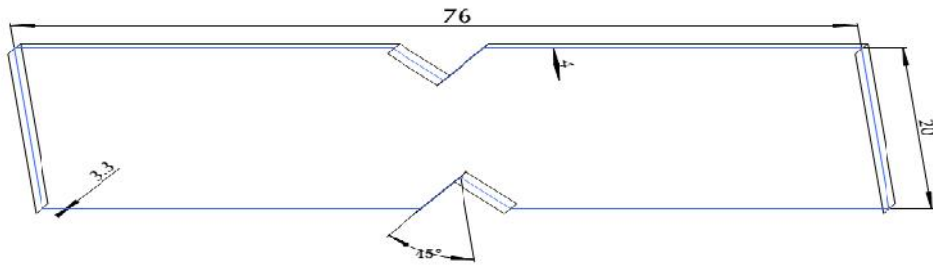


Fig. 3.1 Geometry of the Iosipescu shear test specimen

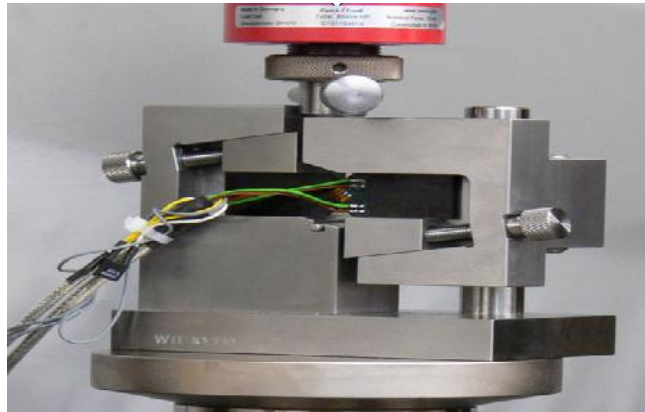


Fig. 3.2 Iosipescu Test fixture

3.3.3 Flexural Test

The flexural tests were performed at 25 ± 2 °C on a Zwick-Roell Universal Testing Machine – Model Z010, Zwick-Roell, Germany, according to ASTM D7264 standard. The standard span-to-thickness ratio is 40:1, the standard specimen thickness at notch was 2mm and the standard

specimen width was 7mm. The flexural tests were run at a crosshead movement at a rate of 1 mm/ min. At least three samples were tested for each sample from which the mean values were reported.

For a test sample, the Flexural strength (σ_F), Modulus (E_F) and Strain to Failure (ϵ_F) are given by Equation (3.1), (3.2) and (3.3) resp.

$$\sigma_F = \frac{3 \times P_{\max} \times L}{2 \times b \times h^2} \quad \text{when } L/h \geq 16 \quad (3.1)$$


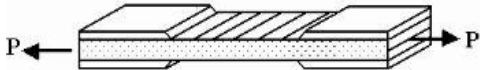

$$E_F = \frac{m \times L^3}{4 \times b \times h^3} \quad (3.2)$$

$$\epsilon_F = \frac{6 \times D \times h}{L^2} \quad (3.3)$$

Where L, b and h are the span (length), width and depth of the sample, m is the slope of the tangent to the initial straight-line portion of the load–deflection curve, D is the maximum deflection before failure, and P_{\max} is the maximum load encountered before failure.

Table 3.8 shows summary of geometry and ASTM standards which were used. In Table 3.8, E_1 , E_2 are the Longitudinal Young's Modulus and G_{12} are Shear Modulus.

Table 3.8 Properties and ASTM standards used

Determinable Property	Symbol	Geometry	ASTM standard
Longitudinal or Tensile Modulus	E_1 (MPa)		ASTM D3039
Transverse Modulus	E_2 (MPa)		ASTM D3039
Shear Modulus	G_{12} (MPa)		ASTM D 5379

3.4 Pin Joint Geometry

In the study, plate (length L , width W , thickness t) made of carbon fiber-reinforced unidirectional plies with a single circular hole to have a rigid pin is used. The ply orientation of the laminate is $[0^0/45^0/90^0/-45^0/90^0/0^0]$. The geometry of composite plate is shown in Fig. 3.4. A hole of diameter D (4 mm) is located along the centerline of the plate at a distance E from one end of the plate. A uniformed tensile load P is applied on the plate and the rigid pin, supported outside the laminate, resists this load. Load is parallel to the plate and is symmetric with respect to the centerline. Hence the load cannot create bending moments about the x , y , and z -axes.

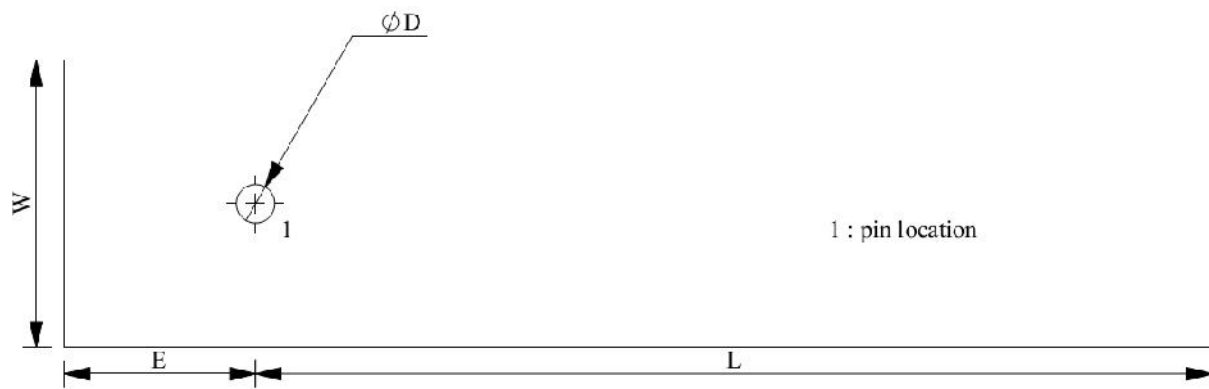


Fig. 3.3 Geometry of composite plate with hole.

The samples with different E/D and W/D ratios having six plies of carbon fiber are given in Table 3.9.

Table 3.9 Geometric parameters of pin joint samples tested

		Diameter of Hole (mm)	E (mm)	W	Thickness (mm)	Length from hole to edge (mm)	Layup
E/D =2	W/D=2	4	8	8	2	100	$[0^0/45^0/90^0/-45^0/90^0/0^0]$
	W/D=3	4	8	12	2	100	$[0^0/45^0/90^0/-45^0/90^0/0^0]$
	W/D=4	4	8	16	2	100	$[0^0/45^0/90^0/-45^0/90^0/0^0]$
	W/D=5	4	8	20	2	100	$[0^0/45^0/90^0/-45^0/90^0/0^0]$

		Diameter of Hole (mm)	E (mm)	W	Thickness (mm)	Length from hole to edge (mm)	Layup
E/D =3	W/D=2	4	12	8	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=3	4	12	12	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=4	4	12	16	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=5	4	12	20	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]

		Diameter of Hole (mm)	E (mm)	W	Thickness (mm)	Length from hole to edge (mm)	Layup
E/D =4	W/D=2	4	16	8	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=3	4	16	12	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=4	4	16	16	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=5	4	16	20	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]

		Diameter of Hole (mm)	E (mm)	W	Thickness (mm)	Length from hole to edge (mm)	Layup
E/D =5	W/D=2	4	20	8	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=3	4	20	12	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=4	4	20	16	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]
	W/D=5	4	20	20	2	100	[0 ⁰ /45 ⁰ /90 ⁰ /-45 ⁰ /90 ⁰ /0 ⁰]

The samples as per the ASTM D3039 standards were prepared with different wt %age of nanoclay *i.e.* 1, 2, 3, 5, to find their mechanical properties. The optimum wt %age of nanoclay was decided on the basis of maximum strength achieved. Thereafter the pin joint samples were prepared as per the Table 3.9 for the achieved optimum value of wt %age of nanoclay in epoxy carbon fiber laminates. The strength of these samples was compared with the neat (without nanoclay) epoxy carbon fiber laminates.

Tests have been carried out on single pinned joints in carbon-fiber reinforced epoxy for a range of laminate configurations and specimen geometry. For single-hole joints the strengths have been obtained experimentally, the effects of variables such as edge distance and width are discussed.

4.1 Mechanical properties of composite laminates

The results obtained by conducting tensile and shear tests on prepared nanoclay laminates using Universal Testing Machine are shown in Table 4.1.

Table 4.1 Mechanical properties of neat carbon fiber/epoxy composite laminates

Tensile Modulus (N/mm ²)		Shear Modulus (G ₁₁) N/mm ²	Poisson Ratio
MD (±SD)	TD (±SD)		
12499(±430)	4203(±200)	2800(±50)	0.32

*MD is Machine or Longitudinal Direction and TD is Transverse Direction.

Thereafter nanoclay in the wt % of 1, 2, 3 and 5 were added to the laminates to find out the change in properties. The results obtained for various compositions of nanoclay are shown in Fig. 4.1.

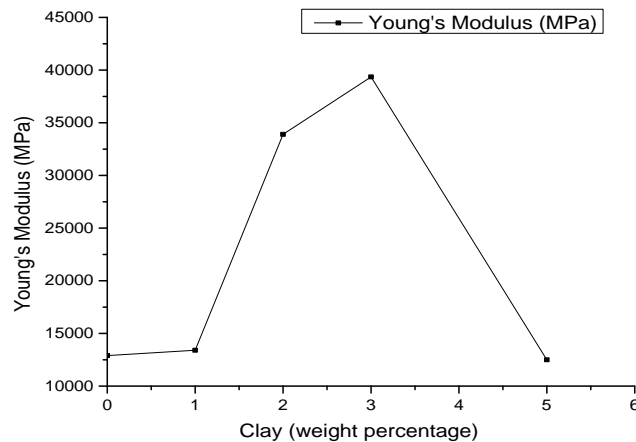


Fig.4.1. Young's modulus vs. weight percentage of nanoclay

It can be observed from Fig. 4.1 that at 3% wt. of nanoclay, the laminates show maximum value of Young's Modulus. Table 4.2 shows the mechanical properties of carbon/epoxy nanoclay nanocomposite laminates.

Table 4.2 Mechanical properties of carbon fiber/epoxy nanoclay composite laminates

Tensile Modulus (N/mm ²)		Shear Modulus (G ₁₁) N/mm ²	Poisson Ratio
MD (±SD)	TD (±SD)		
39300(±400)	13204(±300)	3600(±50)	0.336

4.2 Flexural Test

The results obtained by conducting three point flexural tests on prepared carbon/epoxy nanoclay composites at different nanoclay wt% are shown in Fig.4.2.

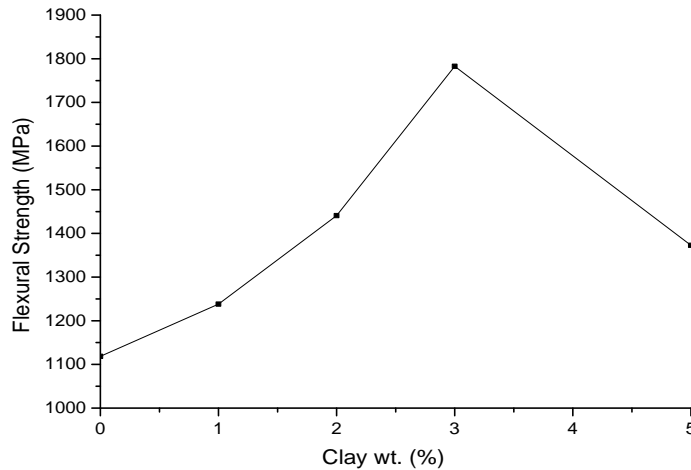


Fig. 4.2. Flexural strength versus nanoclay wt. % plots

As can be seen from Fig. 4.2, the addition of nanoclay increases the Flexural Strength. A significant rise in the Flexural Strength was seen at 3 wt% of nanoclay. A 24% improvement in strength of specimen with 3 wt% Nanoclay loading was observed when compared with neat Epoxy Nanocomposite. The enhancement in the flexural properties is attributed to the improved

interfacial properties responsible for transfer of stresses and elastic deformation in the presence of Nanoclay particles.

4.3 Test results of carbon/epoxy pin joints configuration for unfilled and filled samples

Tests were conducted on pin joint configuration of carbon/epoxy nanocomposites with unfilled (no nanoclay) and at achieved optimum nanoclay percentage (*i.e.* 3%wt.) obtained from the results of the tensile tests. Experimental results for pin joints configuration are discussed in the following sections.

4.3.1 Results of neat carbon/epoxy composite laminates

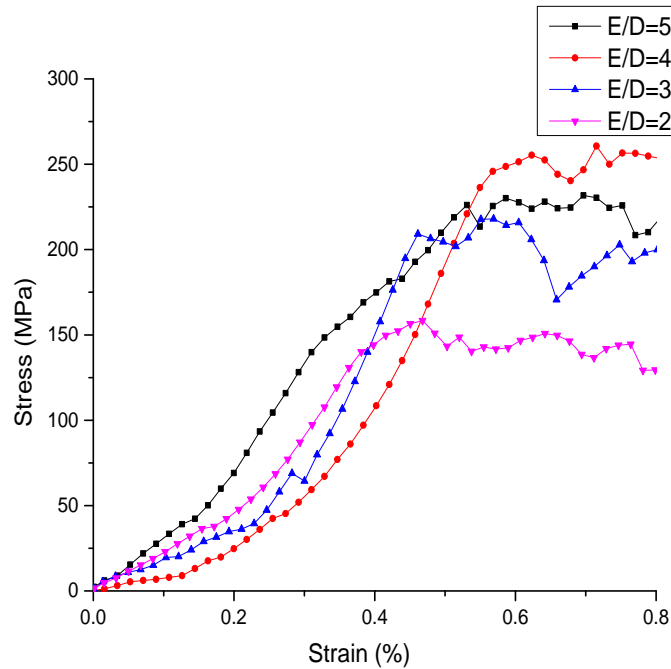


Fig.4.3. Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=5

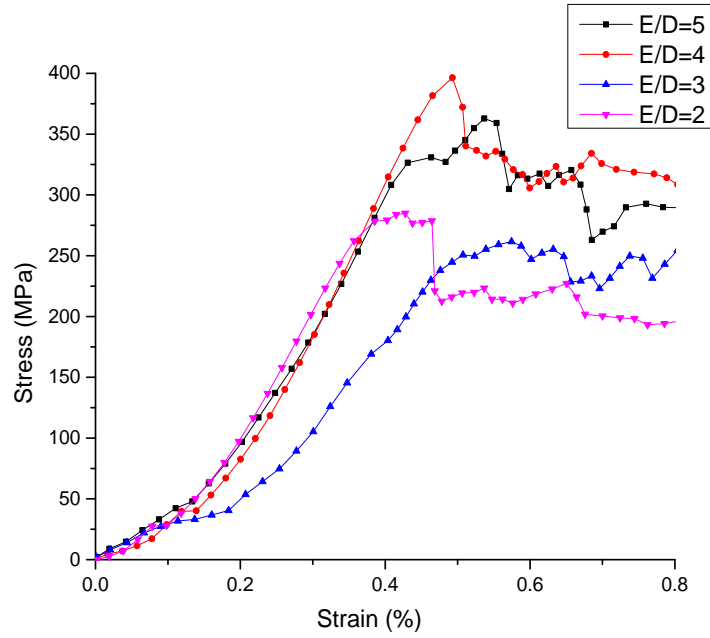


Fig.4.4. Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=4

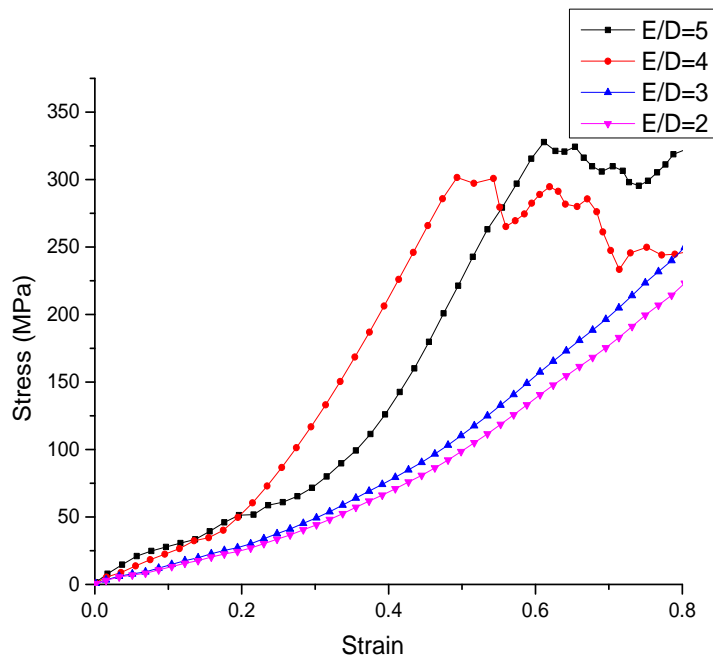


Fig.4.5. Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=3

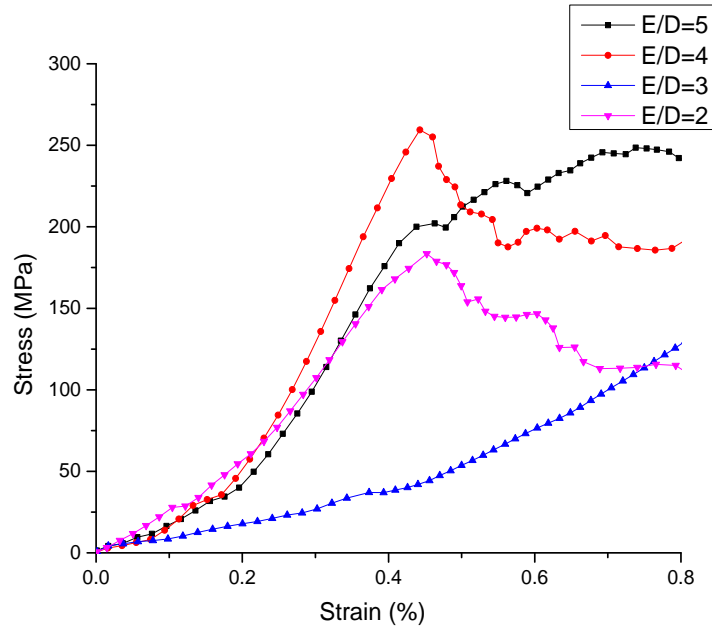


Fig.4.6. Stress vs. Strain curves of pin joints for nanoclay 0% at W/D=2

From the above results it is seen that the results of E/D=4 and E/D=5 and at any W/D ratio does not have much difference so E/D=4 can be taken as the optimum ratio for pin joint configuration. Also, the bearing strength of the joints increases with increase in W/D or E/D ratios. When E/D and W/D ratios have larger values the joint has maximum strength. Net-tension and shear-out failure modes occur with small W/D and E/D values, respectively. Joint failure with the net-tension and shearing modes is catastrophic, immediate, without warning and results from excessive tensile and shear stresses. However, bearing damage is gradual and is related to compressive failure.

4.3.2 Results of single pin joint carbon/epoxy nanocomposites at 3% by wt. of nanoclay

It is seen that nanoclay enhances the mechanical properties of the carbon/epoxy composites. Hence the pin joint testing of carbon/epoxy laminates at 3% wt. of nanoclay was carried out. The graphical plots of these results are shown in Fig. 4.7 to 4.10.

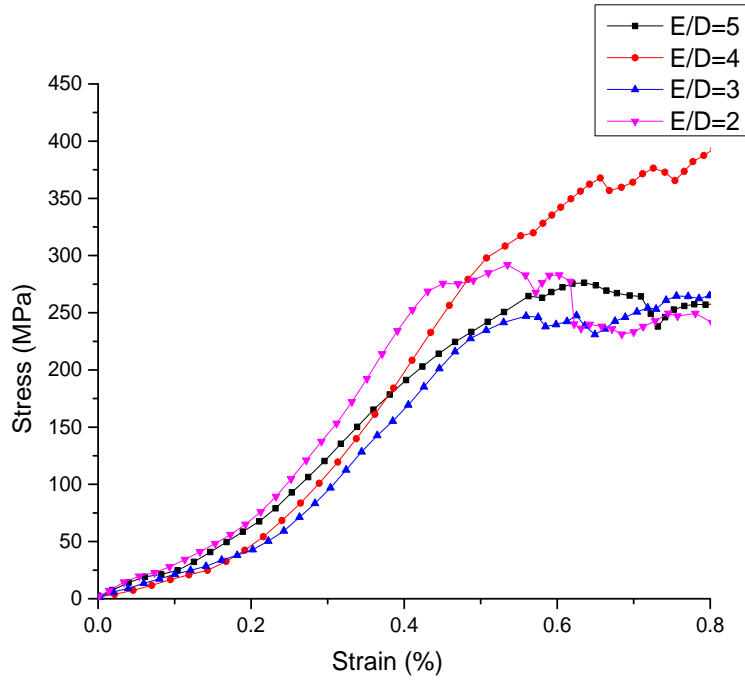


Fig.4.7. Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=5

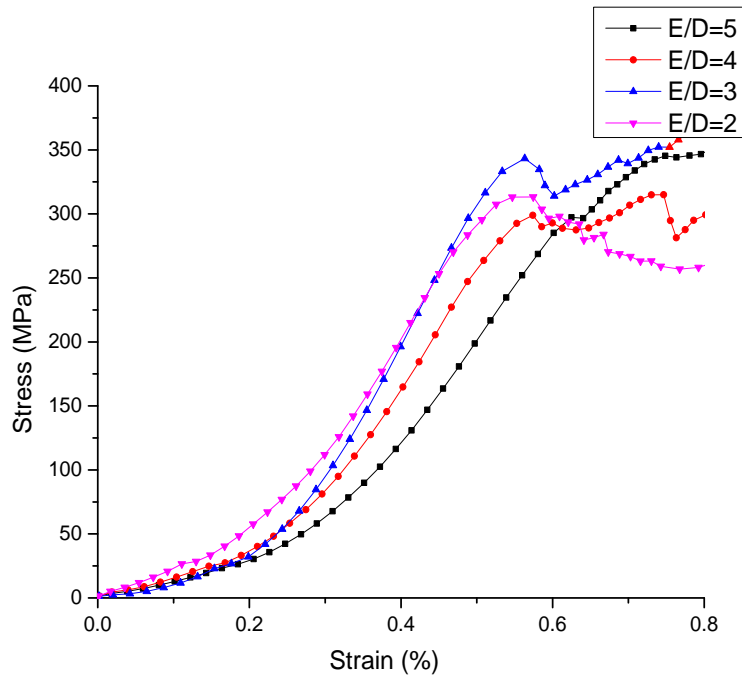


Fig.4.8. Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=4

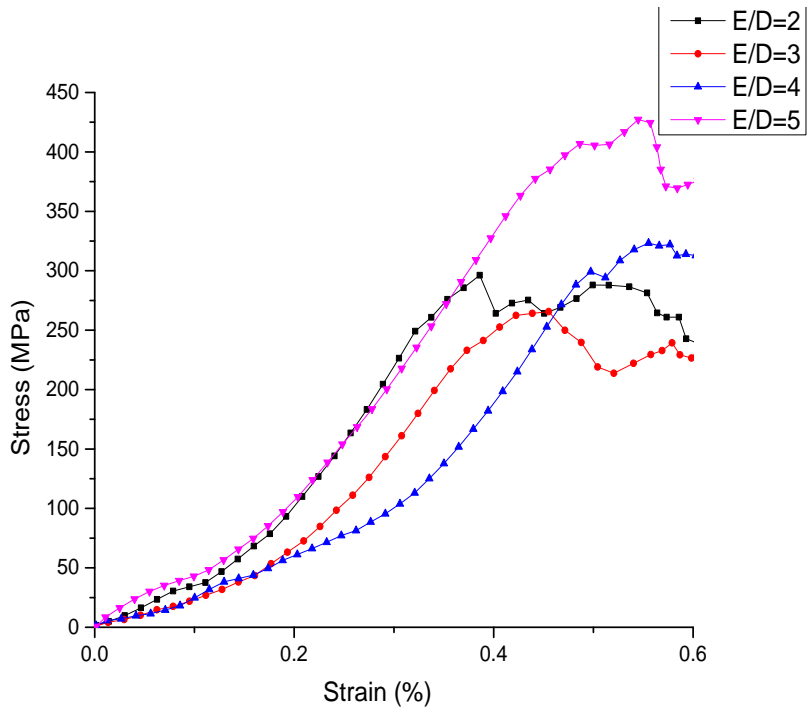


Fig.4.9. Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=3

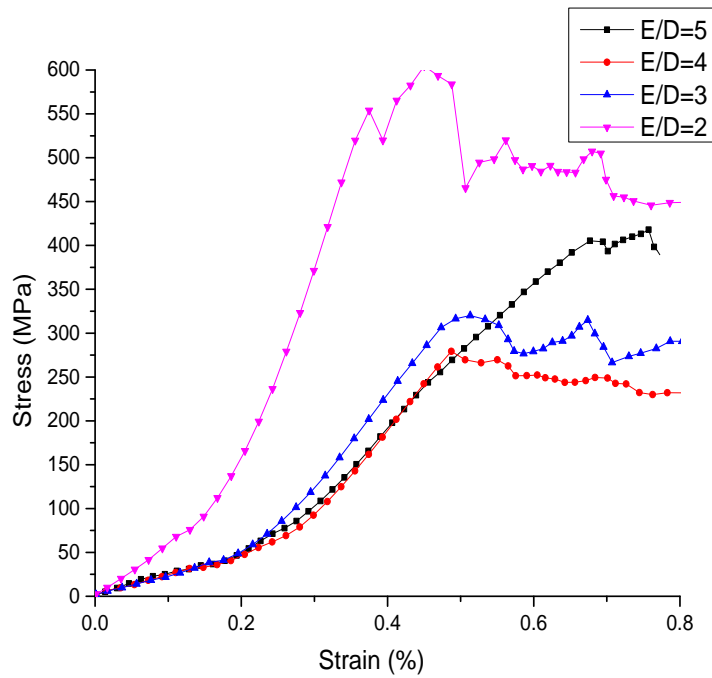


Fig.4.10. Stress vs. Strain curves of pin joints for nanoclay 3% at W/D=2

From the above results it is seen that the nanoclay increases the strength of the pin joints. Also, the bearing strength of the joints increases as W/D or E/D ratios increase. When E/D and W/D ratios have larger values the joint has maximum strength. Net-tension and shear-out failure modes occur with small W/D and E/D values respectively. Joint failure with the net-tension and shearing modes is catastrophic, immediate and without warning which results from excessive tensile and shear stresses. However, bearing damage is progressive and is related to compressive failure. From the results, it can be concluded that E/D ratio 4 and 5 does not have much difference in their strengths.

4.4 Numerical analysis

Finite Element Method is a very versatile tool which can be used for analysis of composites. Number of researchers has used this tool for the analysis of different composites.

4.4.1 Finite Element Modeling

The geometry of the plate investigated in this study was shown in Fig. 3.5. The ply orientation is symmetric with respect to the $z = 0$ plane. A rigid pin is inserted into the hole. Perfect bonding between each ply and frictionless contact are assumed. The composite plate is loaded with an in-plane load, P for the pinned joint.

4.4.2 Geometric modeling and meshing

Geometric model was made in the ANSYS 14.5 with the standard dimensions. Model was meshed using quadrilateral elements. High density mesh was used at the high stress concentration areas. The meshed model is shown in Fig. 4.11.

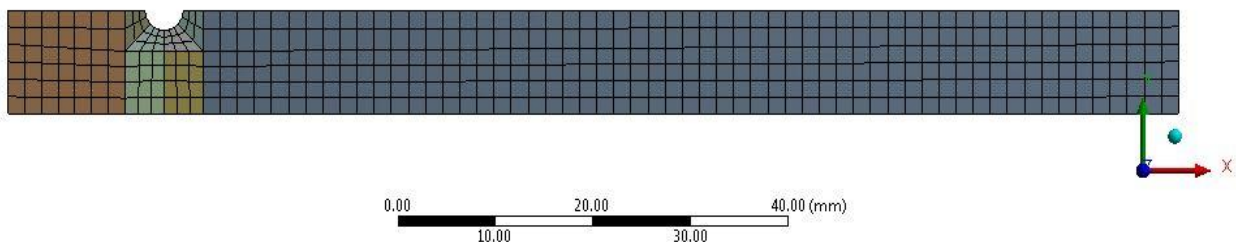


Fig.4.11. Geometric modeling and meshing

4.4.3 Boundary conditions and loading

Radial displacement boundary conditions were applied semi circularly, to nodes around the hole where the pin will contact the composite. These nodes are free tangentially. Composite is loaded gradually in the form of displacements from one end. Radial and symmetric boundary conditions are shown below in Fig.4.12.

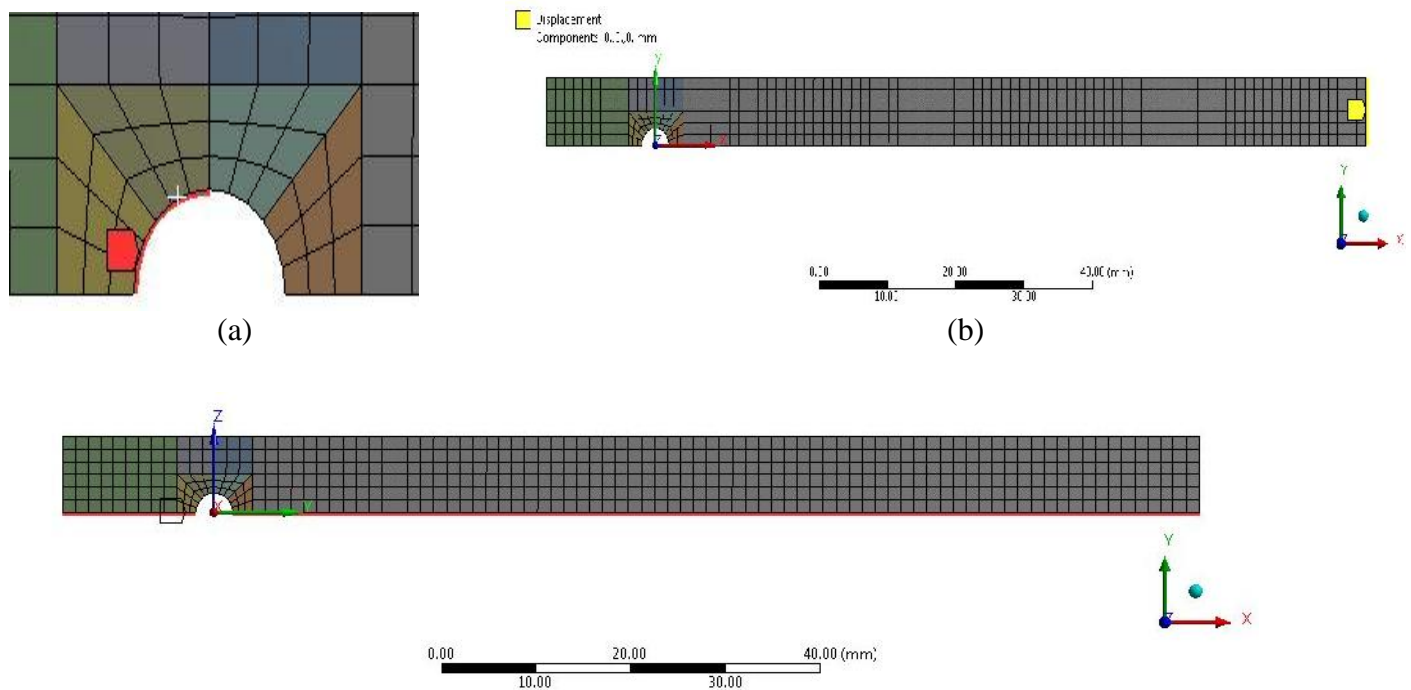


Fig.4.12 (a) Radial boundary conditions (b) Displacement (c) Symmetric boundary conditions

Finite Element Analysis was carried out on the carbon/epoxy pin joint samples and it was found that the unfilled (with no nanoclay) specimens fail early and were not able to sustain large values of stresses but the specimens having 3% nanoclay content were able to sustain larger stress values and show fairly good correlation with experimental results. Fig.4.13 and 4.14 shows the stress distribution around the hole for optimum W/D and E/D ratios *i.e.* 4 for unfilled and filled (3 % wt.) respectively.

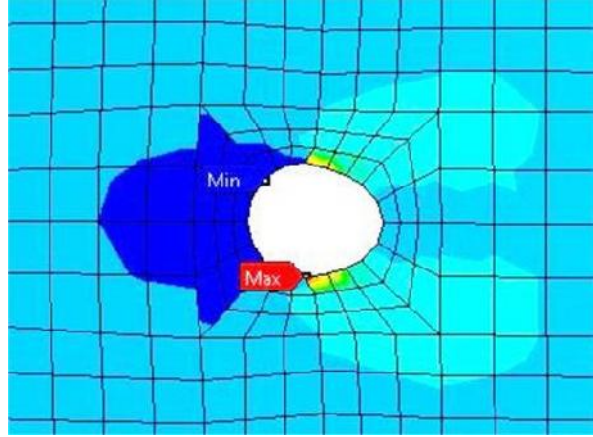


Fig.4.13 Stress distribution around hole at nanoclay 0% for $W/D=4$ and $E/D=4$

From the Fig.4.13 it can be seen that specimen starts failing with the bearing failure. The stresses increases with increase in load and the failure mode starts changing from bearing to shearing. Dark blue colour represents the change in failure mode from bearing to shearing. Bearing failure mode is the desired mode for the failure of the specimen. So at $E/D=4$ and $W/D=4$ specimen undergoes bearing failure mode.

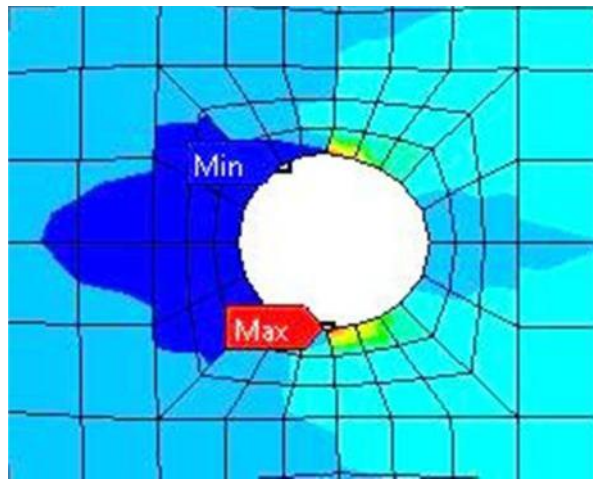


Fig.4.14 Stress distribution around hole at nanoclay 3% for $W/D=4$ and $E/D=4$

From the Fig.4.14 it can be seen that specimen starts failing with the bearing failure. The stresses increases with increase in load and the failure mode starts changing from bearing to shearing. Dark blue colour represents the change in failure mode from bearing to shearing. It can be seen from the above two figures that the deformation around the hole is very less in the samples made with nanoclay contents as compared to the unfilled ones.

4.5 Comparison of Finite Element Analysis and Experimental results

From the above results it can be seen that the optimum strength for carbon/epoxy nanoclay nanocomposites comes at $E/D=4$ for any W/D ratios *i.e.* 2, 3, 4, 5. FEM tool is used to investigate the failure analysis at $E/D=4$ and various W/D ratios. Comparisons between FEA and experimental results have been shown in the Fig. 4.15 to 4.18.

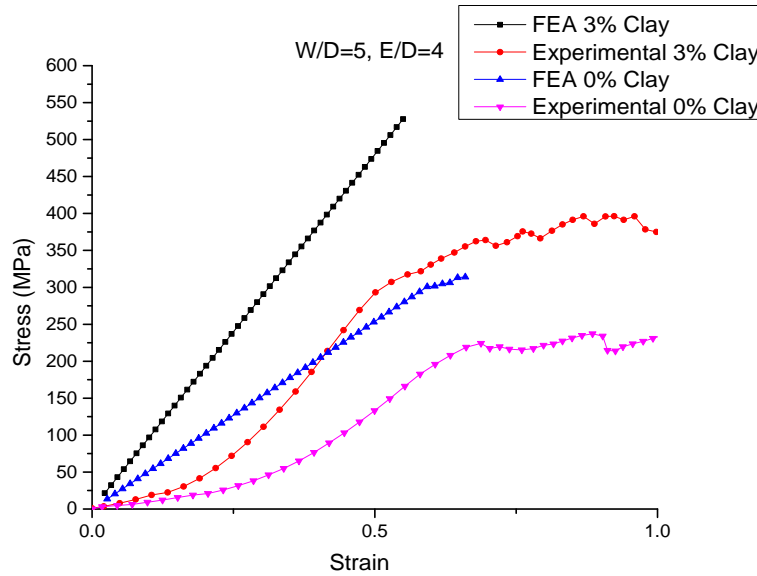


Fig.4.15 Experimental vs. FEA results of pin joints at nanoclay 0% and 3%

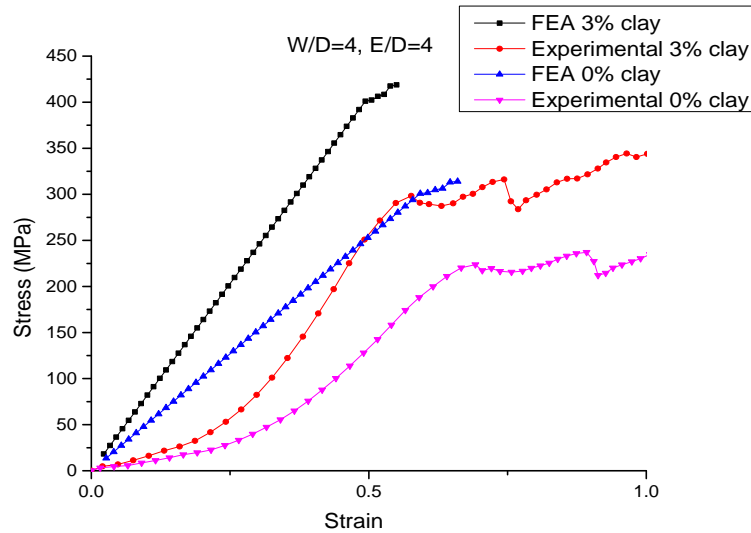


Fig.4.16 Experimental vs. FEA results of pin joints at nanoclay 0% and 3%

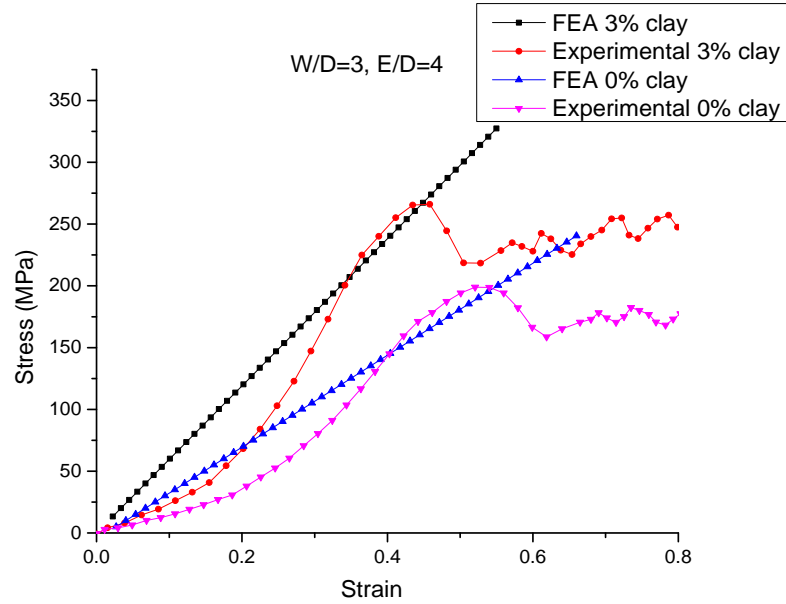


Fig.4.17 Experimental vs. FEA results of pin joints at nanoclay 0% and 3%

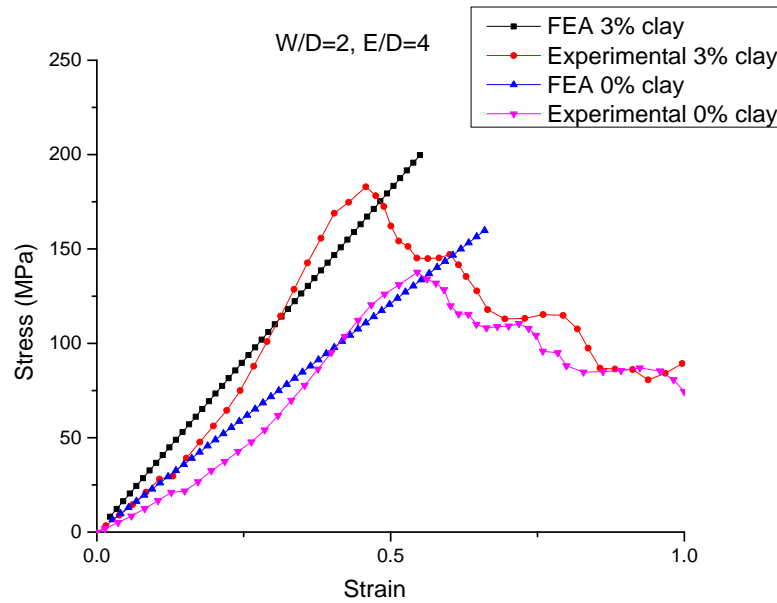


Fig.4.18 Experimental vs. FEA results of pin joints at nanoclay 0% and 3%

Above results show that there is fairly good correlation between experimental and numerical results. The difference in experimental and FEA results can be due to the material processing *i.e.* proper layer orientation, proper curing and the material non linearity.

5.1 Conclusions

The present work deals with the experimental and numerical analysis of the failure behavior of pin-loaded Carbon epoxy nanoclay composite laminates which show fairly close correlation. The effects of geometric parameters and varying compositions on the strength of pinned joints are analyzed by keeping fiber orientation constant. Following inferences can be drawn from the results obtained:

- (i) It is evident that there is a strong correlation between strength of laminated pinned joints and the geometric parameters *i.e.* for higher values of E/D and W/D pinned joints exhibit higher strength but with increase in E/D above four increase in strength is insignificant.
- (ii) For higher values of E/D (>3) for a given width of specimen bearing failure occurs but failure mode changes to shear failure as E/D approaches two. Bearing failure is not catastrophic.
- (iii) Strength of laminates increases with increase in nanoclay content upto wt. 3% , further increase in nanoclay content decreases strength as more nanoclay imparts discontinuity in the matrix.

5.2 Future Scope

The present work can be extended for the following:

- (i) Different compositions of resin-fiber weight percentages can be studied to obtain higher strength and refined properties.
- (ii) Other techniques of curing at high temperature can be employed for obtaining more uniformity in properties.
- (iii) Different combinations of ply-orientation can be tried for improving strength and desired properties.
- (iv) Other filler materials apart from nanoclay can be used to get higher strength.

References

- [1] Icten B. M. and Karakuzu Ramazan, "Progressive failure analysis of pin-loaded carbon-epoxy woven composite plates," *Composites Science and Technology*, vol. 62, pp. 1259-1271, 2002.
- [2] S. Goutianos, R.J. Young P.W.J. van den Heuvel and Peijs, "Failure phenomena in fiber -reinforced composites. Part 6: a finite element study of stress concentrations in unidirectional carbon fiber -reinforced epoxy composites," *Composites Science and Technology*, vol. 64, pp. 645-656, 2004.
- [3] C.T. McCarthy, V.P. Lawlor M.A. McCarthy and W.F. Stanley, "Three-dimensional finite element analysis of single-bolt,single-lap composite bolted joints : part I-model development and validation," *Composite Structures*, vol. 71, pp. 140-158, 2004.
- [4] Shao-Rong Lu, Jing-Hongyu, Hai-Liang Zhang, Xia-Yu Wang, "Wear and mechanical properties of epoxy/SiO₂-TiO₂ composites," *Journal of Materials Science*, vol. 40, pp. 2815 – 2821, 2005.
- [5] M.A. McCarthy, C.T. McCarthy, V.P. Lawlor, W.F. Stanley, "Three-dimensional finite element analysis of single-bolt, single-lap composite bolted joints: part I—model development and validation," *Composite Structures*, vol. 71, pp. 140–158, 2005.
- [6] P.D. Spanos and A. Kontsos, "A multiscale Monte Carlo finite element method for determining mechanical properties of polymer nanocomposites," *Probabilistic Engineering Mechanics*, vol. 23, pp. 456-470, 2008.
- [7] Yuan Xu, Suong Van Hoa, "Mechanical properties of carbon fiber reinforced epoxy/clay nanocomposites," *Composites Science and Technology*, vol. 68, pp. 854-861, 2008.
- [8] Murat Pakdil, "Failure analysis of composite single bolted-joints subjected to bolt pretension," *Indian Journal of Engineering & Materials Sciences*, vol. 16, pp. 79-85, 2009.
- [9] Venkata M. K. Akula and Mark R. Garnich, "Review of Degradation Models for Progressive Failure Analysis of Fiber Reinforced Polymer Composites," *Applied Mechanics Reviews*, vol. 62, pp. 10801-10833, 2009.

- [10] Sun Minqing, Zheng Zixiong, Wang Yingjun and Zhu Sirong, "Finite Element Modeling of Carbon Fiber Reinforced Polymer Pressure Vessel," *International Conference on Educational and Network Technology*, 2010.
- [11] P.F. Liu and J.Y. Zheng, "Recent developments on damage modeling and finite element analysis for composite laminates: A review," *Materials and Design*, vol. 31, pp. 3825-3834, 2010.
- [12] Oualid Limam, Gilles Foret, Hatem Zenzri, "Ultimate strength of pin-loaded composite laminates: A limit analysis approach," *Composite Structures*, vol. 93, pp. 1217–1224, 2011.
- [13] Olanrewaju Aluko, 'An Analytical Method for Failure Prediction of Composite Pinned Joints," *Proceedings of the World Congress on Engineering*, London, U.K, 2011.
- [14] F.X. Irisarri, F. Laurin, N. Carrere, J.F. Maire, "Progressive damage and failure of mechanically fastened joints in CFRP laminates – Part I: Refined Finite Element modelling of single-fastener joints," *Composite Structures*, vol. 94, pp. 2269–2277, 2012.
- [15] Satrio Wicaksono and Gin Boay Chai, "A review of advances in fatigue and life prediction of fiber -reinforced composites," *Materials Design and Applications*, vol. 227(3), pp. 179-195, 2012.
- [16] A.A. Pisano, P. Fuschi, D. De Domenico, "A layered limit analysis of pinned-joints composite laminates: Numerical versus experimental findings," *Composites: Part B*, vol. 43, pp. 940–952, 2012.
- [17] Ivana Ilic, Zlatko Petrovic, Mirko Maksimovic, Slobodan Stupar, Dragi Stamenkovic, "Computation method in failure analysis of mechanically fastened joints at layered composites," *Journal of Mechanical Engineering*, vol. 58, pp. 553-559, 2012.
- [18] Jing Zhang and Jason Rowland, "Damage modeling of carbon-fiber reinforced polymer composite pin-joints at extreme temperatures," *Composite Structures*, vol. 94, pp. 2314–2325, 2012.
- [19] J.K. Chu, S.J. Hou, P. Xu P.F. Liu and J.Y. Zheng, "Numerical simulation and optimal design for composite high-pressure hydrogen storage vessel: A review," *Renewable and Sustainable Energy Reviews*, vol. 16, pp. 1817-1827, 2012.

- [20] P.F. Liu J.Y. Zheng and L.J. Xing, "Failure analysis of carbon fiber /epoxy composite cylindrical laminates using explicit finite element method," *Composites: Part B*, vol. 56, pp. 54-61, 2014.
- [21] Kadir Turan , Mustafa Gur and Mete Onur Kaman, Progressive Failure Analysis of Pin-Loaded Unidirectional Carbon-Epoxy Laminated Composites," *Mechanics of Advanced Materials and Structures*, vol. 21, pp. 98–106, 2014.