

# **AN ASSESSMENT OF MECHANICAL BEHAVIOR OF ENVIRONMENTALLY CONDITIONED FIBRE REINFORCED POLYMER COMPOSITES**

*A thesis submitted in partial fulfillment of the requirement for the degree of*

**Master of technology  
in  
Metallurgical and Materials Engineering**

By

**KISHORE KUMAR MAHATO  
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Department of Metallurgical and Materials Engineering  
National Institute of Technology  
Rourkela  
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Under the guidance of

**Prof. B. C. Ray**



Department of Metallurgical and Materials Engineering  
National Institute of Technology

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2014



**National Institute of Technology  
Rourkela**

**CERTIFICATE**

This is to certify that the thesis entitled, “**An Assessment of Mechanical Behavior of Environmentally Conditioned Fibre Reinforced Polymer Composites**” submitted by Mr. Kishore Kumar Mahato in partial fulfillment of the requirements for the award of Master of Technology Degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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## List of tables:

Table 2.1: Compositions of different types of glass used for fiber manufacture

Table 2.2: Typical properties of different types of Carbon Fiber

Table 2.3: Typical properties of cast Epoxy Resins (at 23°C)

Table 4.1: % change in ILSS values for GFRP composites exposed to thermal conditioning at +50°C temperature for different times and tested at different loading rates.

Table 4.2: % change in ILSS values for GFRP composites exposed to thermal spike conditioning at +100°C temperature for different times and tested at different loading rates.

Table 4.3: % change in ILSS values for GFRP composites exposed to thermal spike conditioning at +150°C temperature for different times and tested at different loading rates.

Table 4.4: % change in ILSS values for GFRP composites exposed to thermal spike conditioning at +200°C temperature for different times and tested at different loading rates.

Table 4.5: % change in ILSS values for CFRP composites exposed to thermal conditioning at +50°C temperature for different times and tested at different loading rates.

Table 4.6: % change in ILSS values for CFRP composites exposed to thermal spike conditioning at +100°C temperature for different times and tested at different loading rates 200mm/min

Table 4.7: % change in ILSS values for CFRP composites exposed to thermal spike conditioning at +150°C temperature for different times and tested at different loading rates.

Table 4.8: % change in ILSS values for CFRP composites exposed to thermal spike conditioning at +200°C temperature for different times and tested at different loading rates

## List of figures:

Figure 1.1: Record useful connected with blend products (a) straw tough will get bricks regarding constructing residences in Egypt in 4000 BC(b) 12th century Mongolian blend bows(c) 1st manned hot air go up in 1783 (d) Havilland Mosquito Bomber of the British isles Regal Air flow Push, Planet Conflict II.

Figure 2.1: Classification of composite according to Matrix

Figure 2.2: Classification of composite according to reinforcement

Figure 2.3: Different forms of reinforcement inside composites.

Figure 2.4: Polyhedra network structure of glass.

Figure 2.5: Chemical process during surface treatment silaceous material by a silane coupling agent.

Figure 2.6: Schematic of Carbon Fibres

Figure 2.7: Schematic representation of repeat unit and chain structures for Kevlar fiber

Figure 2.8: woven roving glass (a), carbon (b) and Kevlar (c) fabrics

Figure 2.9: Structure of DGEBA

Figure 2.10: The curing of epoxy resin with primary amines

Figure 2.11: Diagram of aircraft with different material composition.

Figure 2.12: The structure of bridge deck and FRP sheets.

Figure 2.13: The consumer & sporting items made of FRP

Figure 2.14: FRP usage in medical applications.

Figure 2.15: Fiber-matrix interface and interphase

Figure 2.16: Schematic of Crack opening modes

Figure 2.17: Diagram showing a crack front.

Figure 2.18: Crack tip shows direction of crack propagation.

Figure 3.1: Schematic of loading configuration of short beam shear test

Figure 3.2: Effect of stress concentrations on short beam shear samples:(a) thin sample;(b) thick sample.

Figure 3.3: Shear stress distributions across the thickness of a three-point bending sample in a short beam shear test.

Figure 3.4: Scanning Electron Microscope (JEOL-JSM 6480 LV SEM)

Figure 3.5: (a) Mettler -Toledo 821 instrument along with intra cooler with regard to DSC measurements and (b) reference sample chamber

Figure 3.6: (a) FTIR spectrophotometer, (b) AIM-800 Automatic Infra-red Microscope

Figure 4.1: Comparison of ILSS Vs Loading Rate at 25°C, -20°C, -40°C and -60°C temperatures.

Figure 4.2: Stress Vs Strain plot at 25°C with different loading speed

Figure 4.3: Stress Vs Strain plot at -20°C with different loading speed

Figure 4.4: Stress Vs Strain plot at -40°C with different loading speed

Figure 4.5: Stress Vs Strain plot at -60°C with different loading speed

Figure 4.6: Stress Vs Strain plot at 1mm/min loading speed and at different temperature.

Figure 4.7: ILSS Vs Temperature plot at 100 mm/min loading rate

Figure 4.8: Scanning Electron Micrographs (SEM) at 25°C at (a) 1mm/min loading rate (b) 100 mm/min loading rate (c) 300 mm/min loading rate (d) 1000 mm/min loading rate.

Figure 4.9: Scanning Electron Micrographs (SEM) at -20°C at (a) 1mm/min loading rate (b) 100 mm/min loading rate (c) 1000 mm/min loading rate (d) 1000 mm/min loading rate

Figure 4.10: Scanning Electron Micrographs (SEM) at -40°C at (a) 1mm/min loading rate (b) 100 mm/min loading rate (c) 1000 mm/min loading rate (d) 1000 mm/min loading rate.

Figure 4.11: Scanning Electron Micrographs (SEM) at -60°C at (a) 1mm/min loading rate (b) 100 mm/min loading rate (c) 100 mm/min loading rate (d) 1000 mm/min loading rate.

Figure 4.12: DSC plot shows  $T_g$  at ambient, -20°C and -40°C temperatures of GFRP samples.

Figure 4.13: FTIR plot of GFRP samples at ambient, -20 °C, -40°C and -60°C temperature.

Figure 4.14: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 50°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.15: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 100°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.16: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 150°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.17: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 200°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.18: Flexure stress Vs Flexure strain plot at 50°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.19: Flexure stress Vs Flexure strain plot at 100°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.20: Flexure stress Vs Flexure strain plot at 150°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.21: Flexure stress Vs Flexure strain plot at 200°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.22: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 50°C for a holding time of 5, 10, 15 and 20 minutes.

Figure 4.23: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 100°C for a holding time of 5, 10, 15 and 20 minutes.

Figure 4.24: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 150°C for a holding time of 5, 10, 15 and 20 minutes.

Figure 4.25: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 200°C for a holding time of 5, 10, 15 and 20 minutes.

Figure 4.26: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 50°C for a holding time of 5 and 10 minutes.

Figure 4.27: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 50°C for a holding time of 15 and 20 minutes

Figure 4.28: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 100°C for a holding time of 5 and 10 minutes.

Figure 4.29: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 150°C for a holding time of 15 and 20 minutes.



Figure 4.30: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 200°C for a holding time of 5 and 10 minutes.

Figure 4.31: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 200°C for a holding time of 15 and 20 minutes.

Figure 4.32: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 50°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.33: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 100°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.34: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 150°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.35: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 200°C for a holding time of 5, 10, 15, 20 minutes respectively.

Figure 4.36: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 50°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.37: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 100°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.38: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 150°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.39: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 200°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.

Figure 4.40: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 50°C for a holding time of 5, 10, 15 and 20 minutes.

Figure 4.41: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 100°C for a holding time of 5, 10, 15 and 20 minutes.

Figure 4.42: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 5, 10 and 15 minutes.

Figure 4.43: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 5, 10 and 15 minutes.

Figure 4.44: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 50°C for a holding time of 5 and 10 minutes.

Figure 4.45: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 50°C for a holding time of 15 and 20 minutes.

Figure 4.46: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 100°C for a holding time of 15 and 20 minutes.

Figure 4.47: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 5 and 10 minutes.

Figure 4.48: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 15 and 20 minutes.

Figure 4.49: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 200°C for a holding time of 15 and 20 minutes.

# Table of Contents

Certificate.....	iii
Acknowledgement.....	iv
List of tables.....	v
List of Figures.....	vi
Abstract.....	xv
1. Introduction.....	1
1.1. Introduction.....	2
1.2. Background.....	4
1.3. Objective of the present work.....	8
2. Literature survey.....	10
2.1. Composite.....	11
2.1.1. Classification of composite.....	11
2.2. FRP composite.....	13
2.2.1. Why use FRP composites?.....	15
2.2.2. Advantages of FRP composites.....	16
2.3. Drawback of composites.....	17
2.4. Reinforcement materials.....	17
2.5. Types of fibers used in FRP composites.....	18
2.5.1. Glass fiber.....	18
2.5.1.1. Structure of glass fiber.....	20
2.5.2.2. Silane treatments of glass fibers.....	20
2.5.2. Carbon fibers.....	21
2.5.3. Kevlar fibers.....	23
2.6. Types of matrix materials used in FRP composites.....	25

2.6.1. Epoxy resins.....	25
2.6.2. Unsaturated polyester resins.....	28
2.7. Applications of composites.....	30
2.8. Interface and Interface in composites.....	33
2.9. Effect of loading rate on FRP composites.....	34
2.10. Environmental Effects.....	35
2.10.1. Effect of temperature on FRP composites.....	36
2.10.1.1. Effect of low temperature.....	37
2.10.1.2 Effect of thermal spike.....	38
2.11. Effect of temperature on glass transition temperature of polymer.....	38
2.12. Effect of glass transition temperature on mechanical properties.....	39
2.13. Failure study of FRP composite.....	40
2.13.1. Delamination.....	41
2.13.2. Debonding and fiber pullout.....	42
2.13.3. Matrix microcracking.....	43
2.13.4. Fiber fracture.....	44
3. Experimental Details.....	45
3.1. Materials.....	46
3.2. Experimental Methods.....	46
3.2.1. Short Beam Shear (SBS) Test.....	46
3.2.2. Scanning Electron Microscope (SEM).....	48
3.2.3. Differential Scanning Calorimetry (DSC).....	49
3.2.4. FTIR-ATR Spectroscopy Analysis.....	49
3.3. Experimental Procedure.....	51
3.3.1. Effect of low temperature on glass fiber/epoxy composite.....	52
3.3.2. Effect of thermal spiking on glass/epoxy and carbon/epoxy composite.....	53



5. Conclusion.....99

6. Scope of future work.....100

References

Appendix 1

List of paper presented in conferences based on this project work

## Abstract

Fibre reinforced polymer (FRP) composites are one of the most commonly used materials due to their adaptability to diverse environmental conditions and the comparative ease of combination with different materials to perform definite purposes and reveal advantageous properties. These materials exhibit exceptionally good characteristics such as low density, high specific strength, good anticorrosion properties, fatigue resistance and low manufacturing costs. The components made up of fibre reinforced polymeric composites are exposed to temperature variations (thermal shock, thermal spike, low temperature environment, high temperature environment, freeze thaw), humidity variations, UV radiation and often the combined exposure of these environments leads to more detrimental effect on the performance of the composites during fabrication, in-service time and storage. Further, the rate of loading has significant effects on the mechanical performance of FRP's. Rate of loading can significantly change the mode of failure. The combined effect of harsh environmental conditionings and loading rates leads to very complex situations and more than one damage micro-mechanisms act simultaneously and result in composite failure. Therefore the performance of FRP composites should be well assessed under these complex situations to improve the reliability of FRP composite systems under various critical applications. The present experimental investigation deals with the mechanical behavior of FRP composites exposed at low temperature and thermal spiking conditioning with different loading rate and holding time. The glass/epoxy samples are exposed to ambient temperature, -20 °C, -40 °C and -60 °C temperatures and tested in 3-point bending test at 1, 10, 100, 300, 600, 1000 mm/min loading rates. Also, the thermal spike conditioning of glass/epoxy and carbon/epoxy samples were carried out at 50 °C, 100 °C, 150 °C, and 200 °C temperatures for a holding time of 5, 10, 15 and 20 minutes respectively and then tested for the interlaminar shear strength (ILSS) assessment in short beam shear (SBS) test at 1, 100, 200, 700 and 1000 mm/min loading rates. Scanning electron microscopy (SEM) analysis was performed to identify various degradation mechanisms in fractured samples. Also, DSC measurement have been done to evaluate the glass transition temperature ( $T_g$ ) is very important because it calculates the critical service temperature of the polymer composites. From FTIR analysis it is further confirmed that these environmental conditionings affects the bonding characteristics of the polymeric composites.

**Keywords:** FRP composites, Thermal spike, loading rate, Interlaminar shear strength (ILSS)

# Chapter 1

## Introduction



## 1.1. INTRODUCTION

Worldwide engineering and high performance structural applications have made FRP composite materials ubiquitous in the present century. Fiber A suitable Polymer (FRP) would be the common conditions for just a distinctly extremely versatile group of composites utilized in broad spectrum of qualities discover huge use inside aircrafts, city, automotive, sea, satellites, sporting activities goods, robots, along with cold weather efficiency constructions and other companies. These materials possess attractive mechanical properties for designers and manufacturers. Especially thanks to light weight, high specific strength and specific modulus, corrosion resistant, good fatigue properties, low manufacturing costs and the ability to tailor the properties in required direction as per the application. The application of composite resources in professional transport plane is attractive as a consequence of lessened airframe bodyweight that enables greater energy economic climate and for that reason decreases managing charges. Nowadays polymer composite supplies are usually in massive require with regard to apps in the field of lower temperatures such as cryostats with regard to lower temperatures technological innovation, hydrogen technological innovation tanks and also in superconductivity and in addition in biomedicine with regard to body works implants. [1,2]. These materials have received increased attention for applications in cryogenic environment [3]. Just lately one location identified as prospective source for considerable weight-loss is the alternative involving classic metallic cryogenic gasoline tanks using innovative polymeric matrix blend (PMC) tanks. Epoxy resins as the matrix for nutritional fibre tough plastic composites happen to be used in cryogenic tankage within RLV (Reusable Release Vehicle), winter padding, power padding, structural support in addition to adhesives for vacuum tight important joints. [4]. So now Polymer composites usually are contenders with regard to easy use in reusable start car or truck parts. Largely these kinds of parts usually are cryogenic gas tanks, cryogenic gas shipping and delivery traces, as well as aspects of the actual cryogenic area connected with turbo-pumps [5]. Glass reinforced polymer composites are utilized primarily within thermal efficiency, electrical efficiency, and structural support, along with within permeability limitations that present nominal structural support within superconducting magnets from small temperature ranges [6, 7]. Although regrettably polymeric composites are susceptible to high temperature, low temperature, moisture and humidity when operating in changing environmental conditions. An creative inflatable composite-concrete arch bridge known as the Bridge-in-a-Backpack got its start to reduce development occasion and also expenses, increase

life-span, minimize upkeep price tag and also slow up the impact regarding connect development for 2014 Winter season Olympics inside Russian federation [8].

The aircraft structure may perhaps typically encounter kinetic warming throughout its journey. [9, 10]. Such conditions the item gets essential to realize the actual hardware habits of the composite houses under hygrothermal conditions. The health outcomes in these instances are classified as the presence involving winter history inside way of any winter surge. With scorching and moist conditions, FRP composite process water. Wetness absorption will cause irritation of the plastic, supplying increase to hygroscopic worries. Presence involving winter spikes brings about progress involving winter worries. The presence of the worries can lead to matrix cracking. Within an FRP composite, the actual dietary fiber plus the matrix get distinct coefficients involving winter expansions (CTE). Consequently, expeditions through the exact same heat range results in differential growth. That induces extra worries on the fiber-matrix interface, in so doing deterioration the actual interfacial region. That can lead to debonding and ultimately delamination of the composite by means of interfacial inability. The shrinking has been through the composites throughout alleviating producing alleviating worries. The degree involving cross-linking may improve throughout winter health. The substantial cross-linked networks get lower molecular mobility. Consequently, the mechanical behavior of these composites is different from the composites without any form of thermal conditioning [11]. The state in the composites gets all the more complex a result of the running problems throughout their utilization. The deviation with stress charges throughout effectiveness induces unique degrees of brittleness from the matrix. The packaged relationships of the components lead to damage of physical qualities as well as following stress fracture. The stress fracture weight of FRP composites is usually defined judging by elongation of fibers. These people elongate in a stepwise method seeing that folded domain names or maybe loops are drawn wide open. The molecular elongation seems to become the primary reason regarding toughness [12]. The thermal background is usually launched by providing thermal spike conditioning to various batches of FRP composites at 50 °C, 100 °C, 150 °C and 200 °C.

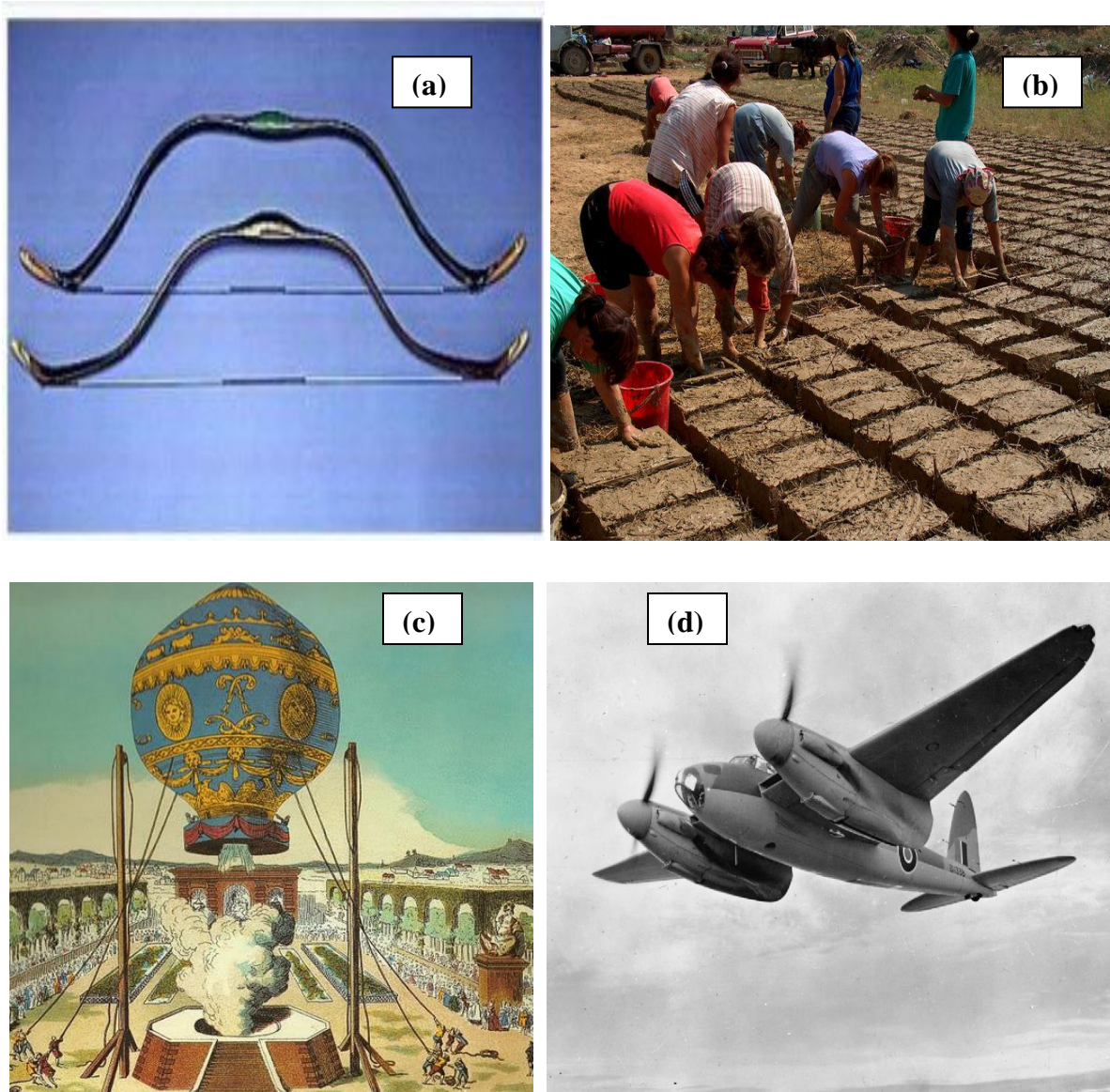
## 1.2. BACKGROUND

The word “composites” includes a contemporary wedding ring. Yet with all the higher durability of fibres in order to restrict and bolster a cheap matrix material may well be over the actual controls.

The particular composite materials is seen via Egypt all-around 4000 BC in which fibrous composite resources ended up employed for arranging the actual publishing materials. Just read was the actual laminated publishing resources created from your papyrus plant. Additional, Egyptians produced storage containers via coarse fibres that had been sketched via temperature softened cup. One more critical request of composites is seen all-around 1200 BC via Mongols. Mongols developed the actual and so called “modern” composite bend. Bicycles show that their early current of composite bows goes in order to 3000 BC as forecasted through Angara Dating. The particular bend utilized a variety of resources including solid wood, horn, sinew (tendon), household leather, bamboo bed sheets and antler. The particular horn and antler ended up accustomed to produce the leading entire body of the bend as it is very versatile and tough. Sinews ended up accustomed to become a member of and include the actual horn and antler together. Glue ended up being well prepared from your bladder of bass and that is accustomed to glue everything constantly in place. The particular string of the bend ended up being created from sinew, mount hair and silk. The particular composite bend and so well prepared accustomed to take virtually a year intended for manufacture. The particular bows ended up and so effective any particular one can certainly throw the actual arrows virtually 1.5 kilometre aside. Before finding of gun-powder the actual composite bend was once an extremely fatal system the way it ended up being a short and handy system.

The particular expert approach throughout early Babylon, one of many lesser miracles with the early world, had been made of bitumen tough with plaited hay. Straw in addition to indy hair are already accustomed to boost off-road stones (improving their break toughness) for at the least 5000 decades. Papers are really a composite; so is tangible: equally were known to these Romans. And just about all organic supplies which often need to have insert – wooden, cuboid, muscle mass usually are composites.. The particular existence regarding composite isn't brand-new. The word “composite” has become very popular throughout latest four-five generations due to usage of modern day composite supplies in several applications. They have harvested quickly in past

times 40 decades while using the improvement regarding fibrous composites: firstly, glass-fibre tough polymers (GFRP) and even more not long ago, carbon-fibre tough polymers (CFRP). Their own easy use in fishing vessels in addition to their increasing replacement regarding precious metals throughout plane in addition to soil transportation devices is really a trend throughout stuff use which can be even now speeding up.



**Figure 1.1: Record useful connected with blend products (a) straw tough will get bricks regarding constructing residences in Egypt in 4000 BC(b) 12th century Mongolian blend bows(c) 1st manned hot air go up in 1783 (d) Havilland Mosquito Bomber of the British isles Regal Air flow Push, Planet Conflict II.**

Because claimed, “Need may be the mother of most inventions”, the current composites that is certainly polymer composites has been around since over the Second Entire world Struggle. Most of the best developments throughout composites were incubated by simply struggle. In the same way this Mongols produced this upvc composite ribbon, Entire world Struggle II introduced this FRP industry through the lab directly into true output. Substitute supplies were required for light applications throughout army plane. Fitters rapidly recognized various other great things about composites beyond becoming light in addition to sturdy. It absolutely was learned that fibreglass composites were clear to help airwaves frequencies, and also the stuff had been rapidly modified for easy use in sheltering electronic radar gear (Radomes). Because of the conclusion with the Entire world Struggle II, a little area of interest composites industry what food was in complete swing. Having decrease requirement for army products, this several composites innovators were now ambitiously seeking to bring in composites directly into various other marketplaces. Boats were an understandable fit for composites, and also the initial business vessel hull had been presented throughout 1946.

At the moment Brandt Goldsworthy, often referred to as the particular “grandfather involving composites,” developed brand new creation operations as well as goods. They are awarded having a lot of breakthroughs including currently being the initial in order to fibreglass the surfboard, which revolutionized the sport. Goldsworthy also devised the creation course of action referred to as pultrusion. Nowadays, goods constructed from this procedure contain ladder train track, software handles, water lines, arrow shafts, armour, educate surfaces for example. Initially involving 1950s, the particular planes as well as auto sector 1st introduced FRP composites in planes building parts as well as in auto body’s parts. Throughout the Minute World War as a result of restriction impositions in numerous nations around the world with regard to traversing border and also adding as well as exporting the particular products, there was clearly shortage involving products, specifically from the military services purposes. During this time the particular martial artist air carriers ended up by far the most superior preventing suggests. This light weight nevertheless powerful products ended up in sought after. Additionally, with regard to app including housing involving electronic digital radar gadgets demand non-metallic products. Therefore, the particular Glass Nutritional fibre Reinforced Plastics (GFRP) was initially utilised in these kind of purposes. Phenolic resins ended up employed since the matrix substance. The 1st

use of composite laminates is visible from the Havilland Mosquito Bomber with the British isles Noble Fresh air Drive.

A result of the particularly useful light weight, rust resistant, substantial unique power as well as unique modulus, excellent fatigue qualities, lower creation fees from the FRP composites, investigation work happen to be spot lighted into the enhancement involving structural purposes with the composite products as well as numerous creation operations. Through this the particular evolution involving creation techniques commenced as well as well known as filament winding as well as pultrusion, that boosted the particular increase involving composite technology from the earth. It had been located which the aerospace, auto market sectors did start to make use of the FRP composites in pots, force vessels as well as non-structural planes ingredients. In 1960's, the particular British isles as well as YOU Navies ended up in unison developed minesweeper delivers as FRP composites which are more advanced than conventional products in severe marine setting as well as non-magnetic in character. Top rated of these products have been proven in superior technology planes including the F-117 stealth martial artist as well as B-2 Bomber. The 1st municipal app in composites has been the dome composition built-in Benghazi in 1968, along with other buildings used slowly but surely. Inside 1970s the particular composites sector did start to develop fully. Better plastic-type material resins as well as increased reinforcing materials ended up developed. DuPont developed a good aramide soluble fiber referred to as Kevlar; this soluble fiber is among the most normal in armour because of its substantial tenacity. Carbon dioxide soluble fiber has been also developed with this in mind occasion; it's given that recently been changing metallic since the brand new substance of choice.

Throughout the later 1970's as well as beginning 1980's many purposes involving FRP composite products ended up illustrated in Europe as well as Parts of Asia. While using improving demand with regard to composites, brand new as well as increased creation operations like pultrusion, resin move molding, as well as filament winding ended up developed as well as put in place from the beginning 1990s. With one of these improvements available, the existing target is to repair north america transport national infrastructure making use of FRP composites with regard to maintenance as well as rehabilitation involving recent connections and also brand new building. This composites sector is increasing, having most of the particular increase is concentrated all-around renewable strength. Wind turbine knives are continually forcing the particular boundaries in dimension and are also demanding superior products, styles, as well as creation.

In the foreseeable future, composites will utilize more desirable materials as well as resins quite a few that will include nano-materials. Devoted university or college packages as well as investigation corporations will always build increased products as well as approaches to production these in to goods. Moreover, composites are for the course in the direction of currently being far more eco-friendly. Resins will include recycled materials as well as bio-based polymers. Composites will always help make the entire world light, more robust, more durable, and a superior spot for a dwell.

### **1.3. OBJECTIVE OF THE PRESENT PROJECT WORK**

#### **A. Effect of low temperature & loading rate on interlaminar shear strength (ILSS) of glass fibre/epoxy Composite**

- Effect of varying loading rate at ambient and at low temperature by flexural (short beam shear test) method.
- Effect of varying loading rate on stress – strain behavior of glass fibre/epoxy composite by flexural (short beam shear test) method.
- Effect of temperature on stress – strain behavior of glass fibre/epoxy composite by flexural (short beam shear test) method.
- Effect of temperature on interlaminar shear strength behavior of glass fibre/epoxy composite by flexural (short beam shear test) method.
- Study of various failure mechanisms of the ambient, low temperature treatment samples after the short beam shear test by SEM micrographs.
- Effect of low temperature on the glass transition temperature of glass fibre/epoxy composite.
- Characterization of small interaction between the fibers and matrix by FTIR imaging techniques.

#### **B. Effects of thermal spike conditioning on mechanical behavior of FRP composites.**

- Effect of loading rate on the interlaminar shear stress (ILSS) values of glass fibre/epoxy and carbon fibre/epoxy composite by flexural (short beam shear test) method.
- Stress – strain behavior of glass fibre/epoxy and carbon fibre/epoxy composite.

→ Effect of thermal spiking conditioning on the glass transition temperature of glass fibre/epoxy composite.

→ Characterization of small interaction between the fibers and matrix by FTIR imaging techniques.



# Chapter 2

## LITERATURE SURVEY

## **2. Literature survey**

### **2.1. Composite**

A composite material is done by combining several dissimilar components. They are combined in such a way that your resulting composite material or composite owns superior properties, which will not be obtainable which has a single component material. Therefore, in technical terms, we can easily define some sort of composite as being a multiphase material from a combination of materials, different in composition or form, which continue being bonded jointly, but retain their identities and properties, without doing any chemical reactions.

Although composites were known to mankind since prehistoric times, the concept and technology have undergone a huge change with better understanding the basics like bonding mechanism between the matrix and fibre [13]. A composite is combination of couple of materials in which first is the reinforcing phase, which is in the form of fibers, sheets, or particles, embedded in another material called the matrix phase [14]. Normally, the reinforcing materials have low weight while the matrix is generally a tough or ductile substance. Polymer, metal and ceramic are usually the reinforcing material and the matrix material.

The biggest benefit of modern-day composite resources is usually that they are light as well as strong. By picking out an appropriate mixture of matrix along with reinforcement stuff, a fresh stuff might be manufactured that will specifically fulfills certain requirements of your distinct application. Composites offer design overall flexibility simply because most of them might be molded in complicated forms. The drawback is normally the price. Although the caused product or service is usually better, the particular recycleables tend to be expensive.

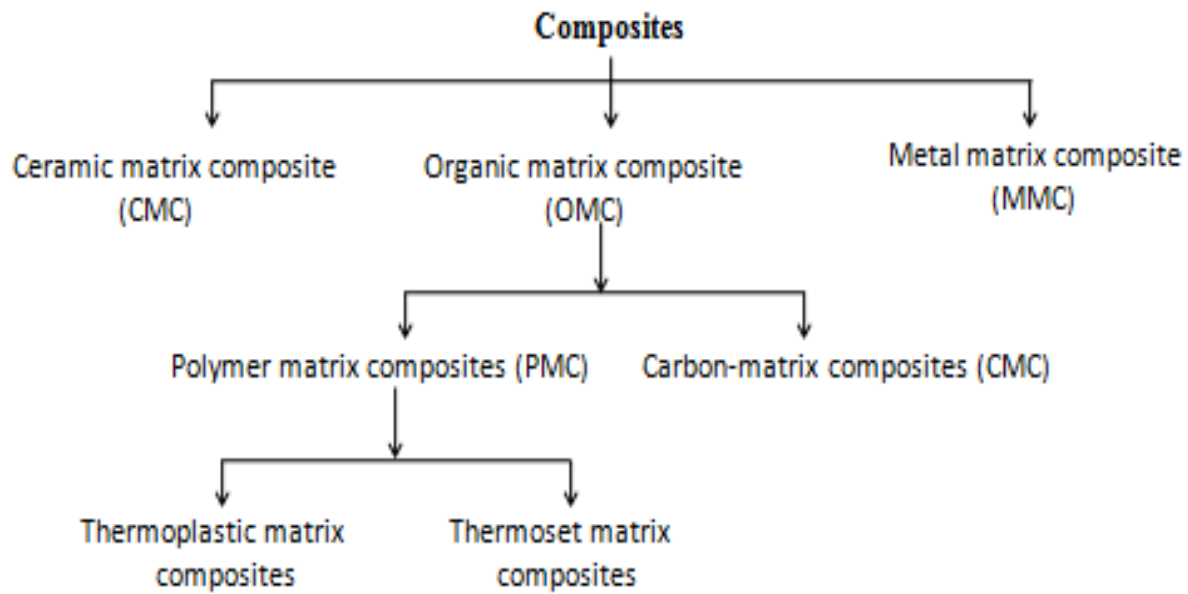
#### **2.1.1. Classification of composites**

Typically composites are generally grouped on two unique organizations. The initial group of group is mostly made depending on matrix component. The actual major composite group consist of

1. Organic-matrix composite (OMCs),
2. Metal-matrix composite (MMCs),
3. Ceramic-matrix composite (CMCs).

The term of “organic-matrix composite” is normally presumed to incorporate two forms of composites:

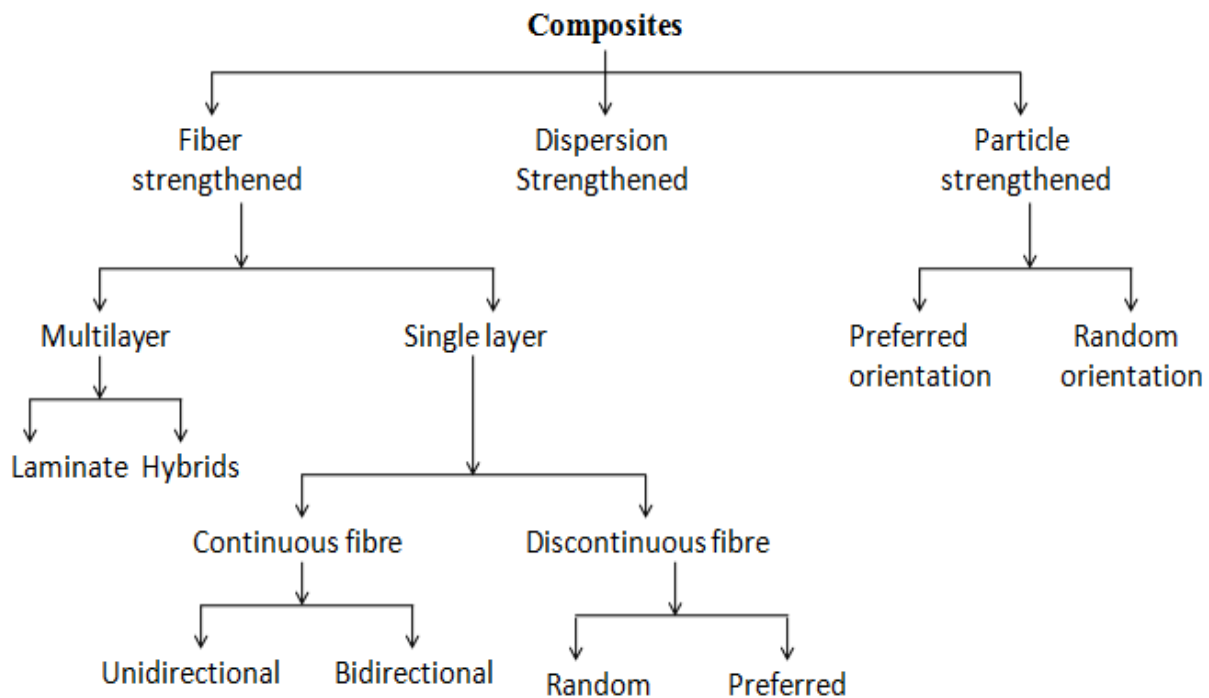
1. Polymer-matrix composite (PMCs)
2. Carbon-matrix composite (commonly mention to as carbon-carbon composites) [15].



**Figure 2.1: Classification of composite according to Matrix**

The one more group of classification is the term for the reinforcement form

1. Particle reinforced,
2. Fiber reinforced,
3. Structural composites.



**Figure 2.2: Classification of composite according to reinforcement**

## **2.2. Fibre reinforced polymer (FRP) composite**

Fibre-reinforced polymer (FRP) is a composite material created from the polymer matrix reinforced together with fibers. The particular fibers usually are glass, carbon, or maybe aramid, though additional fibers like paper or maybe solid wood or maybe asbestos are typically applied. The commonly used polymer is vinylester, polyester thermosetting plastic phenol formaldehyde resins and epoxy. FRPs are normally find applications in aircraft, automobile sector, marine, sporting goods, chemical industry, medical, low temperature applications and also structural sectors etc.

Composite components are usually made or by natural means occurrence components constructed from two or more ingredient components having appreciably various physical or chemical properties which usually stay on different along with distinct within the complete design. Most composites include robust, hard fibers in a matrix which is less strong along with less stiff. The aim is to discover a material, which have high strength and low density. Typically, glass, carbon or aramid fibers are embedded in matrices such as polyester resins or epoxies. From time to time, thermoplastic polymers may be recommended, being that they are moldable following primary generation. For the most part, these are even now in the developmental stage, together with issues of high manufacturing costs but being defeat [16]. Besides, in these composites the purposes behind including the strands (or, in a few cases, particles) are frequently rather mind boggling; for instance, enhancements may be looked for in wear, creep, fracture toughness, thermal stability, and so forth [17].

FRP are generally composites employed in nearly all kind of superior structure, using application starting from planes, helicopters and also spacecraft to motorboats, cruises and also ocean going systems and vehicles, athletics merchandise, chemical handing out apparatus and also civil communications including bridges and also properties. The effective use of FRP composites is maintaining growth in an extraordinary charge because these components are used additional in their current promotes and turn into recognized inside somewhat brand-new promotes including biomedical gadgets and also civil buildings. An important factor travelling the actual elevated software connected with composites within the modern times could be the growth connected with brand-new superior varieties of FRP components. This consists of improvements throughout good resin structure and brand recent types of reinforcement, for example carbon nanotubes and nanoparticles. This gives an advanced description of the fabrication, impact resistance, delamination resistance, mechanical properties and applications of 3D FRP composites [18].

FRP composites are lightweight, non-destructive, show high particular quality and particular solidness, are effortlessly built, and could be custom-made to fulfill execution prerequisites. Because of these favorable qualities, FRP composites have been incorporated in new development and restoration of structures through its utilization as support in cement, extension decks, measured structures, formwork, and outer fortification for fortifying and seismic redesign [19].

FRP composites offer quite a few benefits such as corrosion resistance, non-magnetic properties, excessive tensile strength, light in weight in addition to easier handling. These people get rid of important strength after bending, plus they are hypersensitive to stress-rupture consequences. Moreover, his or her expense, no matter whether considered for each device bodyweight or maybe based on push transporting potential, will be excessive in comparison to standard metal reinforcing bars. Answers in addition to disadvantages of usage are actually made available in addition to constant advancements are anticipated down the road. The machine expense connected with FRP reinforcements will be expected to minimize appreciably with increased industry write about in addition to demand. Even so, there are purposes where by FRP reinforcements tend to be affordable in addition to justifiable. Like cases contain the application of FRP sheets throughout restore in addition to fortifying connected with concrete buildings, in addition to the application of FRP works or maybe textiles or maybe material throughout lean bare concrete goods. The price tag on restore in addition to rehabilitation of any structure is always, throughout family member terminology, substantially beyond the price tag on the original structure. Fix typically uses a comparatively little variety of restore materials yet a excessive determination throughout labor. Moreover the price tag on labor throughout created international locations is excessive of which the price tag on product turns into secondary. Thus the greatest performance in addition to toughness with the repair material could be the additional cost-effective could be the renovate[20].

Ultimately, to ensure composites to really often be a workable alternative, the doctor has to possibly be structurally as well as financially possible[21]. On the other hand, constrained investigations are accessible on financial and ecological plausibility of these components from the viewpoint of a life cycle approach, as transient information is accessible. Furthermore, the extensive influences of utilizing these materials need to be dead set. The subject will be tended to in a straightforward and fundamental for improved understanding.

### 2.2.1 Why use FRP composites?

When contemplating simply strength along with materials it seems, at first glance, the particular debate regarding FRP composites in very lasting constructed surroundings is in question. On the other hand, probable rewards specific to usage of FRP composites linked to criteria like:

- Higher strength and stiffness
- Low density
- Higher performance
- Longer lasting i.e. resistance to corrosion
- Defence organisation
- Space organisation
- Marine environments

When it comes to FRP composites, environmentally friendly problems look like the hurdle to help their feasibility being an environmentally friendly material especially when contemplating fossil energy destruction, polluting of the environment, smog, along with acidification linked to their generation. Moreover, the ability to recycling FRP composites is fixed along with, as opposed to metal along with timber, structural components are not used again to execute much the same function with a different design. Nonetheless, considering the environmental result associated with FRP composites with structure software, especially by way of existence circuit examination, may show direct along with oblique rewards that are additional competing than conservative materials.

Composite material have urbanized tremendously given that they were being first presented. Nonetheless, ahead of composite materials works extremely well as an option to conventional materials during the environmentally friendly setting numerous desires continue being.

- Availability associated with standardised strength characterization files for FRP composite materials.
- Integration associated with strength files along with means of assistance existence prediction associated with structural members utilizing FRP composites.
- Development associated with approaches along with systems for materials assortment based on existence circuit tests associated with structural components along with techniques.

### **2.2.2. Advantages of FRP composites**

- 1. Light weight:** Reduction in lifeless load leads to a greater dwell load volume along with possible removing of weight limits.
- 2. Fast instalment:** FRPs could be fast implemented on account of modular, pre-fabricated, as well a slight weight models of which eliminate creating as well as healing efforts required for regular components for instance real decks or even sophisticated welding as well as fascinating required throughout major metallic building.
- 3. Lowered interruption:** small down-time of in-service structure using fast instalment techniques can bring about reduced end user charges, reduced servicing, increased safe practices, as well as superior properties.
- 4 Good strength:** outstanding amount of resistance to be able to de-icing salts along with substances leads to removing corrosion, cracking, as well as spalling regarding steel reinforced concrete..
- 5. Long services lifestyle:** substantial, non-civil FRP buildings have got carried out very well throughout hard surroundings for decades. To give an example, FRP connect decks need to deliver services lifestyle around 75-100 a long time along with minor servicing.
- 6. Fatigue and impact resistance:** FRPs have got higher weakness staying power as well as result amount of resistance.
- 7. Good quality control:** look fabrication of FRP leads to outstanding top quality control along with reduced transportation expense.
- 8. Ease of instalment:** FRP structural devices or even subsystems for instance bridge decks are

as used by general contractors or even servicing folks using common specifics along with instalment moment decline as high as 80%, as a result removing targeted visitors blockage as well as building site similar automobile accident[22].

### **2.3. Drawbacks of Composites**

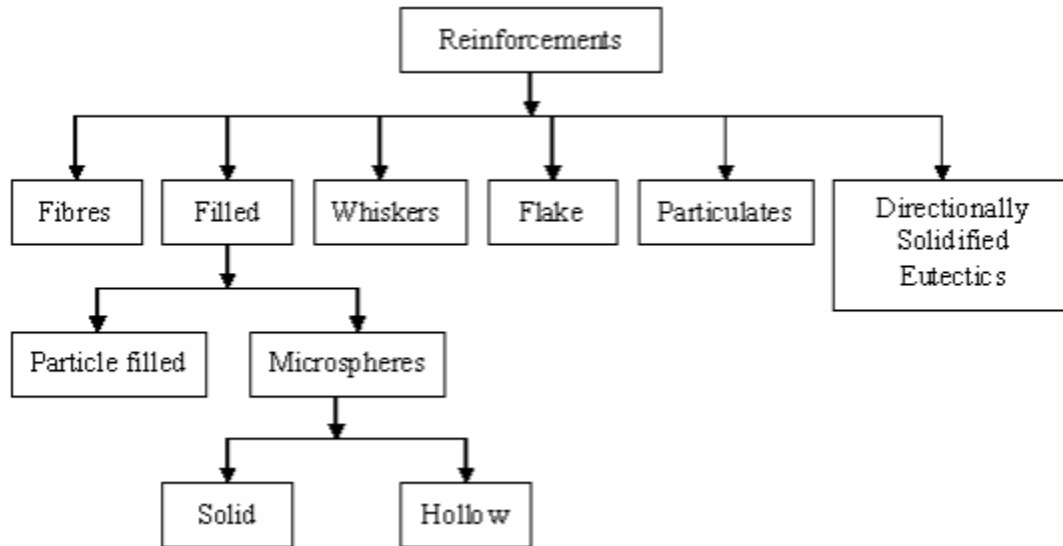
Composite owns a bit increased original prices, confined encounter using these components simply by design and style professionals along with installers, insufficient files on long-term subject functionality, along with lack of whole variety associated with rules along with technical specifications just like conventional components.

1. Composites are more brittle than wrought metals and thus more easily damaged.
2. Repair originates new problems such as curing either hot or cold takes time, materials require refrigerated transport and storage.
3. Composites must be thoroughly cleaned of all contamination before repair.
4. Composites must be dried before repair because all resin matrices and some fibres absorb moisture [23].
5. Due to cooling at ultra-low temperatures, glass fibres show compressive stress and due to this thermal residual stress is developed in the matrix [24].
6. Susceptibility to de-lamination is one of the inherent weaknesses of laminated composite structures [25].

### **2.4. Reinforcement materials**

Reinforcements to the composites may be fibers, fabric particles as well as whiskers. Fibres are usually essentially seen as an a single for an extended time axis having other a pair of axes sometimes usually sale paper as well as next to sale paper. Particles have no desired orientation therefore may their particular shape. Whiskers employ desired shapes however are usually smaller each inside diameter along with length as compared with fibers. The figure demonstrates forms of reinforcements inside composites.





**Figure 2.3: Different forms of reinforcement inside composites.**

Reinforcing constituents in composites, since the expression suggests, provide the strength that creates the particular composite what exactly it truly is. Nonetheless they additionally work certain further requirements involving heat resistance or maybe conduction, resistance to corrosion to be able to rust and offer stiffness. Reinforcement could be meant to accomplish just about all or maybe one of these simple operates according to what's needed.

A reinforcement that will embellishes the particular matrix strength have to be more robust as well as more stiffer compared to the matrix as well as effective at changing failing system to be able to the benefit of the particular composite. Consequently the particular ductility needs to be little or perhaps nil the particular composite ought to behave as brittle as you possibly can.

## **2.5. Types of fibers used in fibre reinforced polymer composites**

1. Glass fibre
2. Carbon fibre
3. Kevlar fibre

### **2.5.1. Glass Fiber**

Glass fibers are silica based glass compounds that include some metal oxides which can be used to modify to produce different varieties of glass. The leading oxide will be silica available as silica sand, one other oxides for instance Ca, Na along with Al are generally involved to melting

temperature along with hamper crystallization. The most important qualities regarding glass fibre are generally:

**E-glass:** Electronic glass has low alkali information of the purchase of 2%. It really is useful for common purpose structural applications and it's used in building sector, they have excellent temperature and electric powered level of resistance.

**S-glass:** This can be a stronger and stiffer fiber having a greater corrosion resistance level of resistance compared to E-glass fiber. It has excellent temperature level of resistance capability.

**C-glass:** Chemical glass has excellent corrosion resistance level of resistance for you to acid solution and angles and it has compound stableness in chemically corrosion conditions.

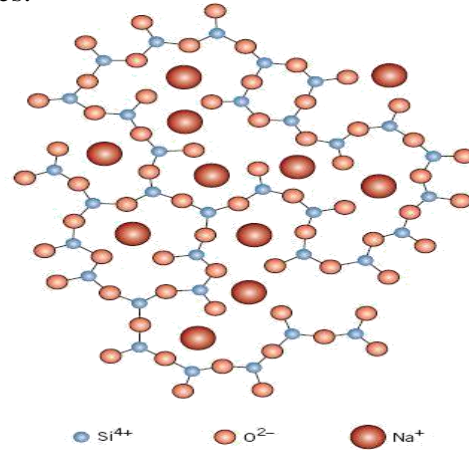
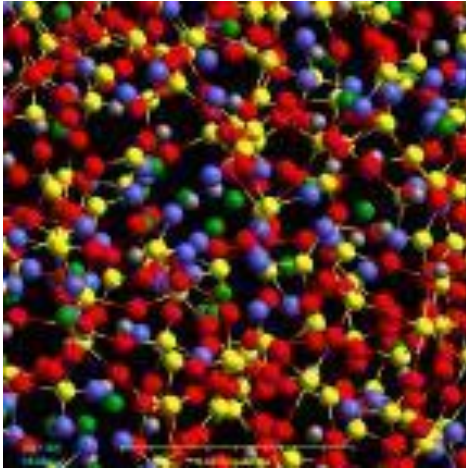
**R-glass:** R glass features a greater tensile modulus and tensile strength and higher level of aging, resistance to fatigue, and temperature corrosion compared to that of Electronic glass.

Components	Glass Type			
	E	C	A	S
SiO <sub>2</sub>	55.2	65	72	65
Al <sub>2</sub> O <sub>3</sub>	14.8	4	2.5	25
CaO	18.7	14	9.0	-
MgO	3.3	3	0.9	10.0
B <sub>2</sub> O <sub>3</sub>	7.3	5	0.5	-
Na <sub>2</sub> O	0.5	8.5	12.5	-
K <sub>2</sub> O	0.2	-	1.5	-

**Table 2.1: Compositions of different types of glass used for fiber manufacture**

### 2.5.1.2. Structure of glass fiber

Glass fibers have substantial tensile strength, effect skills and also substantial element weight. Although these kind of have comparatively lower fatigue resistance, self-abrasiveness, lower modulus, and also very weak adhesion to matrix composites.



**Figure 2.4: Polyhedra network structure of glass [26]**

The particular 3-D network regarding composition regarding glass ends in isotropic components regarding glass fibres, as opposed to people regarding carbon and also Kevlar aramid fibres which can be anisotropic. The particular elastic modulus regarding glass fibres assessed along the fibre axis is the same as in which assessed within the transverse direction, some sort of trait special to be able to glass fibres.

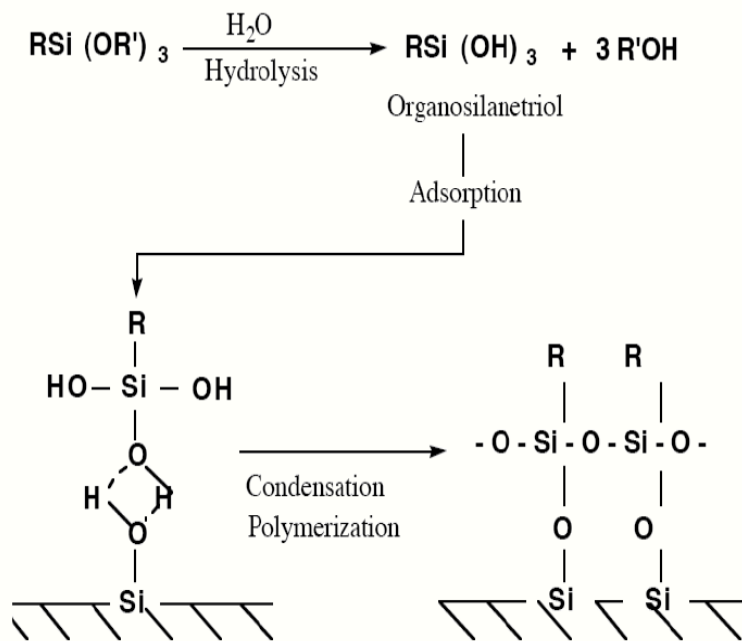
### Surface Treatment of Reinforcing Materials

Surface remedy is done to further improve this adhesion connected with filler injections as well as fibres to be able to matrix resin through changing the top of solid. Generally, chemical substance design as well as sometimes topology in the surface area change upon process.

### 2.5.1.3 Silane treatments of glass fibers

Sizing materials are coated on the surface of the glass fibers as protection against mechanical damage. For glass reinforcement the sizing usually contains a coupling agent to bridge the fiber surface with the resin matrix used in composite. These coupling agents are usually organo silanes with all the structure  $X_3SiR$ . The particular R group may capable to reply that has a group inside

polymer matrix, the X groups could hydrolyze inside existence connected with water to silanol groups which can condense with all the silanol groups on the outside towards the glass fibers to siloxanes. Beyond the adhesion promotion, coupling agents help with protecting fiber surfaces and forestall inhibition connected with polymerization because of the solid surfaces. A tiny bit of some sort of coupling agent can often drastically enhance the mechanical along with actual physical attributes connected with composites. The chemical reaction of a coupling agent occurring during the treatment and drying of the filler is shown below using a silane coupling agent [26].



**Figure 2.5: Chemical process during surface treatment silaceous material by a silane couplingagent [26].**

### 2.5.2. Carbon fibers

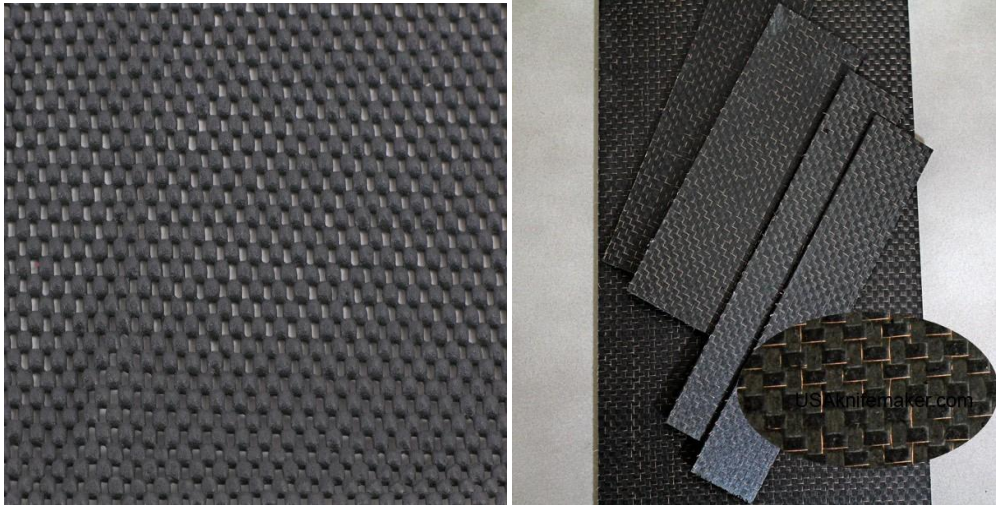
Carbon fiber is the all costly of an extra usual reinforcements, playing throughout space programs and combination connected with incredible performance attributes along with easy fold allow it to become crucial encouragement together with price being associated with extra magnitude. Carbon fibers contain small crystallite associated with turbostratic graphite. These appear to be graphite single crystals except that the covering air carriers are not packed inside a typical vogue over the c-axis path. In the graphite single crystal the carbon atoms are inside a basal jet usually are fixed in hexagonal arrays along with held with each other by strong covalent

bonds. Between the basal planes just weak Van-der-waal forces can be found. Meaning that the single crystals usually are hugely anisotropic with the plane moduli of the order associated with 100 GPa whilst your elements perpendicular for the basal plane are just concerning 70 GPa. It is hence evident which to generate higher modulus along with higher durability fibers, your basal air carriers of the graphite need to be parallel for the fibre axis. They have already reduce thermal expansion coefficients compared to the glass in addition to aramid fibres. The carbon fibre can be an anisotropic material, and it is transverse modulus can be obtain involving specifications a smaller amount than the longitudinal modulus.

The tension from crack will likely is usually dramatically reduced. With the material brittleness on higher modulus, it becomes important with joint and connection details, that will have high stress concentrations. Via this phenomenon, carbon composite laminates tend to be added effective throughout adhesive bonding and the idea eliminates mechanical fasteners [27].

Typical Properties	Density (g/cm <sup>3</sup> )	Young's Modulus (GPa)	Tensile Strength (GPa)	Tensile Elongation(%)
High Strength	1.8	230	2.48	1.1
High Modulus	1.9	370	1.79	0.5
Ultra High Modulus	2.0-2.1	520-620	1.03-1.31	0.2

**Table 2.2: Typical properties of different types of Carbon Fiber**



**Figure 2.6: Schematic of Carbon Fibres**

### **2.5.3. Kevlar fibres**

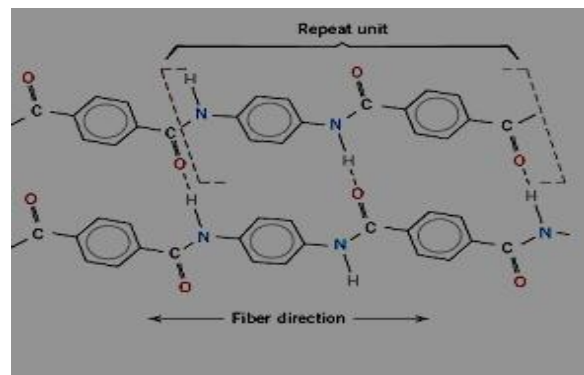
Kevlar (poly-paraphenyleneterephthalamide) may be the DuPont Company's manufacturer for a synthetic stuff made out of para-aramid fibres that the business boasts is actually a few situations more powerful as opposed to identical bodyweight of metallic, even though becoming light, accommodating and also comfortable. It is also incredibly temperature resistant and also decomposes above 400 °C without melting. It was invented by Stephanie Kwolek of DuPont from research into high performance polymers, and patented by her in 1966 and first marketed in 1971. Kevlar is a registered trademark of E.I. du Pont de Nemours and Company [28].

Formerly that will substitute your metal belts in tires, this is just about the most well known name in soft suits (bulletproof vests). It is also found in serious sporting activities apparatus, high-tension drumhead applications, animal controlling protection, composite planes development, fire accommodates, yacht sails, so that as a asbestos substitute. When this polymer is actually spun in the same manner which as high temperature along with slice resistant. Para-aramid fibres do not corrosion or perhaps corrode, along with the strength are actually unaffected by means of immersion in mineral water. When weaved jointly, these type form a good materials with regard to mooring traces and also other marine objects. Nevertheless, unless especially waterproofed, para-aramid fibre's ability to halt bullets and also other projectiles is actually degraded as soon as moist. The particular reputation involving salts along with a number of other pollutants, in

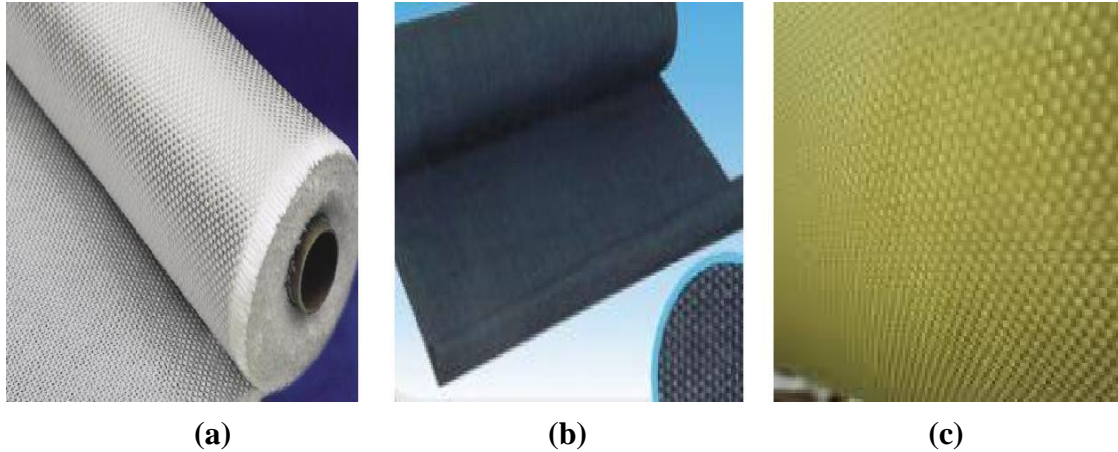
particular calcium, would obstruct your follicle communications along with should be eliminated from the production procedure. Kevlar involves comparatively firm substances, which often type a new planar sheet-like framework much like a silk filled duvet protein.

These types of attributes cause its large mechanical strength and its particular remarkable high temperature resistance. Because doing so is actually remarkably unsaturated, i.e. the relation involving carbon to hydrogen atoms is quite large, it's got a low flammability. Kevlar substances have polar offered with regard to hydrogen connecting. Water that enters the lining of the fiber can certainly replace connecting between substances along with slow up the material's strength, as the accessible groups in the surface cause great wetting attributes. It is of importance to connecting fiber to other types involving polymer, building a new fiber strengthened plastic material.

This kind of bonding in addition makes fiber normal along with "sticky" when compared with non-polar polymers including polyethylene. Throughout structural application, Kevlar fiber is usually bonded to each other so they can other supplies to form a new composite. Kevlar's primary weak spots are usually which it decomposes within alkaline problems or perhaps as soon as subjected to chlorine. Whilst it have a fantastic tensile strength, occasionally over 4. 0 GPa, including all muscle this does buckle in compression.



**Figure 2.7: Schematic representation of repeat unit and chain structures for Kevlar fiber[28].**



**Figure 2.8: woven roving glass (a), carbon (b) and Kevlar (c) fabrics**

## **2.6. Types of Matrix Material used in fibre reinforced polymer composites**

Fibres, simply because they cannot transfer loads in one to a different, get minimal utilization in engineering applications. If they are generally stuck in a matrix product, to create some sort of composite, the matrix acts to binds the fibres with each other, transfer load for the fibres, and also deterioration as a result of controlling.

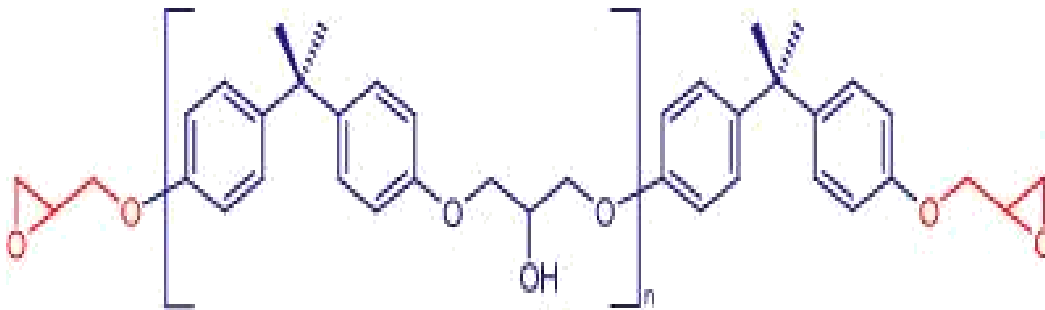
The actual matrix carries a strong have an effect on upon various mechanical properties from the composite for example transverse modulus and also strength, shear properties, and also components with compression. Physical and chemical characteristics from the matrix for example melting as well as curing heat range, viscosity, and also reactivity together with fibres have an effect on the option connected with manufacturing course of action. The matrix material for a composite system is selected, keeping in view all these factors. Commonly used matrix materials are described below:

### **2.6.1. Epoxy resin**

Epoxy resins are generally somewhat small molecular weight pre-polymers competent at currently being prepared beneath various circumstances. A couple of important advantages of these resins are generally above unsaturated polyester resins are generally: first, they can be partly cured and saved for the reason that express, and second they demonstrate small shrinking in the course of



cure. On the other hand, your viscosity connected with regular epoxy resins is increased and they are costlier when compared to polyester resins. The particular cured resins have higher chemical, corrosion level of resistance, good mechanical and thermal components, outstanding adhesion to be able to various substrates, and good and electro-mechanical components. Approximately 45% of an entire number involving epoxy resins produced is usually considered throughout protective coatings though ones remaining is taken within structural applications just like laminates as well as composites, tooling, moulding, casting, construction, adhesives, etc [29]. Epoxy resins are usually characterized by your existence of a three-membered ring containing a couple carbons and also the oxygen (epoxy group or maybe epoxide or maybe oxirane ring). Epoxy would be the initial liquefied effect product or service regarding bisphenol-A using far more than epichlorohidrin and this also resin is termed diglycidylether regarding bisphenol Some sort of (DGEBA). DGEBA is employed broadly inside business because excessive fluidity, running ease, and also very good real qualities with the cured regarding resin.

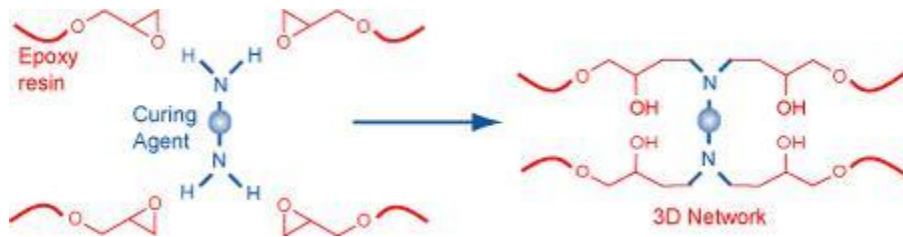


**Figure 2.9: Structure of DGEBA**

A range of epoxy resins is accessible, different through relatively tough small temperature epoxies regarding use within structure market place to help brittle epoxies regarding use within structure market place to help brittle epoxies valuable within aerospace industry. This prevalent request regarding epoxy resin can be largely due to accessibility to resins having distinct anchor buildings and molecular weight load to offer items having small viscosity (liquids) to help small melting place solids.

Ethylene diamines are hottest aliphatic amines regarding cured epoxy resins. These include

remarkably reactive, small molecular weight recovering agents of which lead to firmly cross-linked circle. A single major amino party behaves having two epoxy groupings. The key and legitimate amines are reactive recovering agents. The key amino party can be additional reactive towards epoxy when compared with legitimate amino groupings are consumed (95%), in contrast to simply 28% regarding legitimate amino groupings are consumed.



**Figure 2.10: The curing of epoxy resin with primary amines**

The primary amino-epoxy reaction ends in linear polymerization even though legitimate amino-epoxy reaction results in branching along with cross-linking. The actual treated epoxy resins come across a range of applications because adhesives, laminates, sealants, films, for example.

The actual optimum recovering temperatures as well as the thermal balance of epoxy resin count on the type of curing agent. The actual anhydride treated epoxy resins have good electrical, chemical, and mechanical properties and used for power along with electronic applications. Stuffing of cracks in cement constructions is usually attained through epoxies. Inside construction sector, pertaining to bonding along with layer requirements, minimal temperatures curing of epoxies is usually attained through the use of thiols which demonstrate larger curing rates. Epoxy dependent prepregs are already employed in several airplane factors including rudders, stabilizers, elevators, wing tips, launching items doors, radomes, ailerons, for example. The actual composite materials be construed as 3-9% of complete structural bodyweight from the business oriented aircrafts including Boeing 767 or perhaps Boeing 777. The composite along with laminate sector works by using 28% of epoxy resins created. Other than most of this software, the particular software, the particular main individual of epoxy is usually layer sector

Property	
Density, g/cm <sup>3</sup>	1.2-1.3
Tensile modulus, MPa	55-130
Tensile modulus, GPa	2.75-4.10
Thermal expansion, 10 <sup>-6</sup> /°C	45-65
Water absorption, %in 24h	0.08-0.15

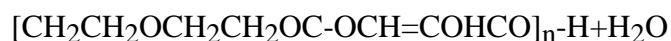
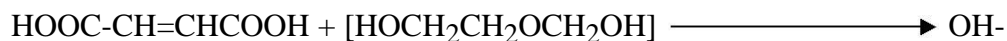
**Table 2.3: Typical properties of cast Epoxy Resins (at 23°C)**

### 2.6.2. Unsaturated Polyester Resins

Some sort of polyester resin is an unsaturated (reactive) polyester stable wiped out in a polymerizable monomer. Unsaturated polyesters are generally long-chain linear polymers containing numerous carbon double bonds. They are made by any moisture build-up or condensation response between any glycol (ethylene, propylene, diethylene glycol) along with a good unsaturated dibasic acid solution (maleic or even fumaric).

This polymerizable (reactive) monomer for example styrene, which usually in addition includes carbon double bonds, operates like a cross-linking real estate agent by simply connecting adjoining polyester substances on their unsaturated factors. This monomer in addition operates like a diluent, lessens viscosity, along with makes it much better to method. This treating or even cross-linking method is actually begun with the help of a compact variety of any catalyst for example natural and organic peroxide or even a good aliphatic azo ingredient.

Since there is simply no by-product in the response, your treating is done on area temp connected with enhanced temperature having or even without software connected with strain. A normal polyester resin produced from reaction of maleic acid solution along with diethylene glycol is actually demonstrated underneath:



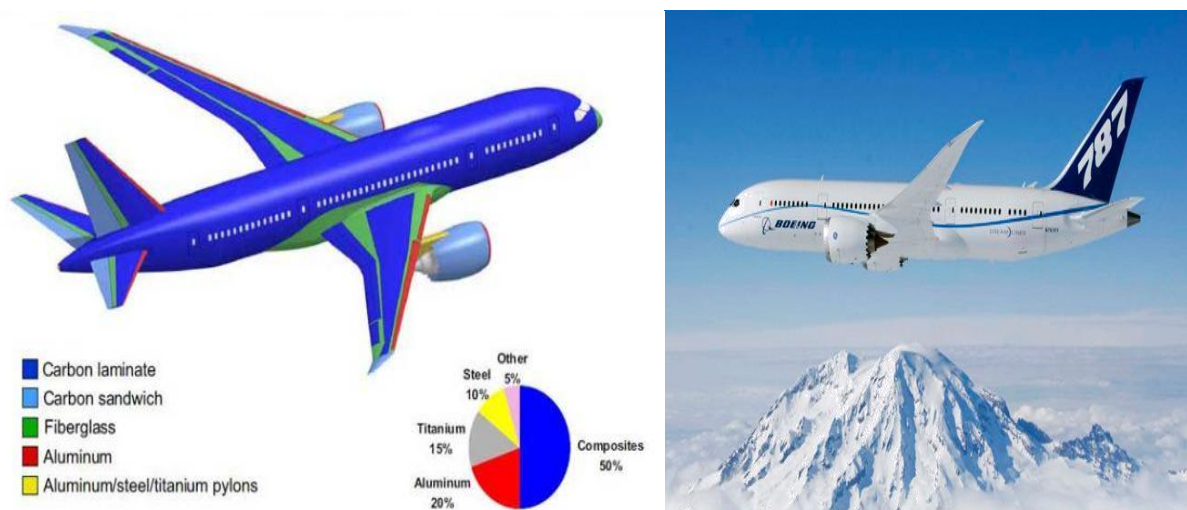
The sizes of the particular molecule or perhaps quantity polymerization are different. The resin will probably typically certainly be a reliable but is actually contained inside a monomer like styrene. The perfect solution is viscosity can be manipulated with the per cent styrene and is typically quite smooth (less compared to viscosity involving honey). The change coming from liquid to solid in order to reliable happens by using any free-radical initiator (e. grams., benzoyl peroxide) or perhaps curing agent. Te styrene monomer cross-links or perhaps does respond while using the two times bond from the polyester anchor above to make any circle polymer bonded. Your reaction does not develop almost any by-product and is exothermic reaction. Hence, the particular treating method is actually associated with shrinking along with heat increase. Capabilities of modifying or tailoring the chemical structure of polyesters by processing techniques and raw materials selection make them versatile [29].

## 2.7. Applications of Composites

Composites are generally one of the most widespread materials because of the customization to be able to unique conditions along with the comparative easy blend with different materials to be able to work specific functions and also demonstrate suitable components. This commercial programs of composites offer to provide larger online business offerings compared to aerospace industry as a result of pure dimensions of transportation business. Thus your move of composite programs through plane to be able to different commercial works by using is becoming dominant in recent times.

### Aircraft business

The employment of fiber sturdy composites is becoming increasingly attractive replacement for then, the materials precious metals for many people plane factors largely because of the greater energy, durability, deterioration level of resistance, level of resistance to be able to low energy and also harm patience characteristics. Composites provide higher freedom for the reason that material might be adapted in order to meet the structure requirements and they also furthermore deliver substantial pounds advantages. Thoroughly created particular person composite areas, at this time, are generally concerning 20-30% light in comparison with their standard metal counterparts. Though all-composite aircraft are available on the planet marketplace, yet innovations within the realistic using composite materials should enable additionally decrease in your structural weight of air. This matrix is normally a great epoxy based method needing curing conditions between 120° and also 180°C (250° and also 350°F).



**Figure 2.11: Diagram of aircraft with different material composition.**

## Structural Applications

Composites get long been utilized in this design business. Applications cover anything from non-structural gratings and also claddings to be able to whole structural programs for professional sustains, properties, prolonged cover roof constructions, tanks, connection ingredients and also full connection programs. Their particular features about deterioration resistance and also small weight get tested interesting in many small tension purposes.

A great expansion to be able to the application of top rated FRP within primary structural purposes, even so, may be slow to get acceptance even though there is significantly progress action. Composites existing enormous chances to be able to perform raising position as an alternative stuff to replace wooden, metallic, alloy and also concrete within properties.



**Figure 2.12: The structure of bridge deck and FRP sheets.**

## Electrical and Electronics

Composites capable of good electric insulation, antimagnetic and spark-free, good adhesion to help glue and fresh paint, self-extinguishing attributes are employed with the development regarding distribution pillars, link boxes, information with the break up regarding current-transport levels to avoid incomplete circuits and many others. Additional potential purposes regarding composites with this industry are usually 3rd railroad insures pertaining to undercover train, Structures pertaining to cost to do business sign wrinkles pertaining to train, power brand insulations, lightning rods, power pole corner forearms, optical fibers, switch gear casings, aerial man-lift device etc.

## Offshore Oil and Gas market place

Materials as well as concrete floor are classified as the resources of preference for just offshore gas and oil output websites, with metallic prominent inside the topside applications. Composites have found their particular method in confined apps, specially wherever rust as well as the requirement to minimize large servicing fees have been a worry. As the industry movements to help higher mineral water heights, the involving fat conserving has become gradually more crucial in combination with the program involving buoyant pressure for your knee set ups. Composites may find excellent consumption within manufacture from the using: Users for gas polluting of the environment boundaries, gratings, ladders as well as railings about oil-drilling websites as well as lines, path methods, Sucker fishing rods.

## Consumer and sports goods

The ideal pattern associated with sporting activities gear requires the usage of many martial arts styles, not simply regarding performance but to create the tools seeing that user-friendly is possible in the understanding associated with harm elimination. With building sporting activities gear, the various traits associated with materials must be thought to be. Between these kinds of traits are usually power, ductility, solidity, tiredness resistance, strength, modulus (damping), along with cost. To meet the prerequisites associated with sporting activities gear, the particular equipment of selection frequently consist of a mixture of materials forms. Pursuing are classified as the customer along with sporting activities items wherever a lots of prospective for composites in the future are Pole vault, golf stick, tennis rackets, sporting bicycles etc.



**Figure 2.13: The consumer & sporting items made of FRP**

## Chemical Industry

Chemical sector Supplemented through the benefits of composites involving light-weight, mould ability, flame resistance houses, resistance in order to chemical compounds possesses manufactured the actual materials favorite in the compound industry. Composites tend to be extensively utilised in professional gratings, ducting, storage devices tanks, scrubbers, structural supports, piping, wear out stacks, pumps as well as blowers, reactors, posts for example concerning acidic as well as alkaline conditions. Some of the probable applications tend to be composite wrecks regarding liquid gas regarding choice gasoline automobile, racked baby bottles regarding flame services, rock climbing, double-wall FRP wrecks having an first warning method regarding leakage discovery, storage devices tanks, travel shafts, regarding electrostatic precipitator, enthusiast cutting blades (for equally axial as well as centrifugal fans), ducts.

## Medicals

Nowadays composite are used for medical purposes in several applications.



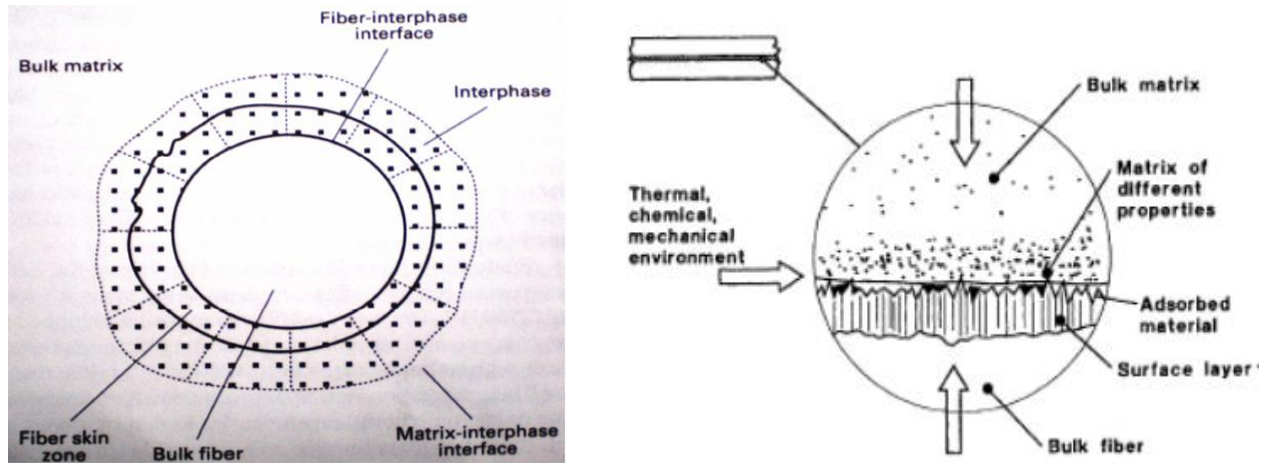
**Figure 2.14: FRP usage in medical applications.**

### 2.8. Interface and Interphase in Composites

The mechanical behavior of composites not only depends on the constituent fiber and matrix properties but also on the fiber/matrix interface. The interface acts as the heart of the composite. The fiber/matrix interface within the composite plays an important role in determining the gross mechanical performance. Under loading, the interface is responsible for transmitting the load from the matrix to the fibres, which contributes the greater portion of the composite strength. In contrast interphase is a two dimensional mathematical plane having some finite unknown thickness [30], wherein the physical, chemical and mechanical



properties differ either constantly or in a stepwise manner between those of the matrix materials and the bulk fiber [31].



**Fig 2.15: Fiber-matrix interface and interphase [30].**

The bonding between the fibre and matrix depends on the arrangement of atoms, molecular and chemical composition of the fibre and matrix, further the morphological properties of the fibre and the diffusivity of elements in each constituent also have pronounced effects [31], therefore the interface may be specific to each fibre/matrix system. Fibre /matrix adhesion in general can be attributed to various mechanisms, which include adsorption and wetting, electrostatic attraction, chemical bonding, reaction bonding and exchange reaction bonding [31]. Better the interfacial bond better will be the ILSS, de-lamination resistance, fatigue and corrosion resistance [32].

## **2.9. Effect of loading rate on FRP composites**

The present study emphasizes on the loading rate sensitivity of fiber reinforced polymeric composites, as the past researches in this area is bit far from any considerable conclusion. With micro-composite tests inside it your locus in addition to modes regarding failure be required to become consistent inside what are generally originally produced because of its composite inside order regarding a great catered work is to end up being right [33]. Fibrous polymeric composites equipped with a wide range of advantages, it play major role in various sectors such as aerospace, transportation, chemical industry, electrical and electronic parts, construction, consumer and sports good. Composites materials

experience different running conditions throughout their assistance existence, electronic. g. having tools with high running price to help force shipwrecks with lower loading speed [34]. The effects of diverse running charges upon mechanical attributes of FRP composites are researched along with observed a variety of contrary observations along with results [35]. The fiber/matrix interfacial bond greatly affects the particular mechanised behavior of composite products given it transmitting the strain on the matrix towards the fibres [36]. The fiber/matrix software mechanical attributes can also be vulnerable towards the running price. The de-bonding allows, frictional dropping allows along with evident shear power beliefs boost together with increasing displacement price [37]. Through the earlier experiments the info describing of which influence of stress price around the mechanised attributes are sparse the particular studied composite products are glass/epoxy along with carbon/epoxy [38]. The failing mode inside the GFRP laminates improvements coming from fiber brittle failing together with fiber pullout with quasi-static crosshead loads, to help brittle failing together with sizeable matrix injury because cross punch scalp pace raises [39]. E-Glass fabric are already located to get load sensitive, details easily obtainable in literature wasn't considerable to be able to bring virtually any cement summary. Woven along with unidirectional GFRP usually are load centered, both the modulus along with strength boost as the check load usually are greater, strain to be able to failing lower having escalating strain rate [39-41]. Thermal degradation connected with epoxy resin involves chemical substance effect along with actual physical improvements. Chemical substance effect can be showed by means of oxidation, additionally cross-linking and additional result of un-reacted monomers, whilst actual physical change will be the visco-elastic behavior [42]. This visco-elastic deliver behavior connected with plastic is usually temperature along with running fee centered [43]. This arctic health results in post-curing conditioning impact [44]. The present inspections are already aimed at the actual loading rate along with temperature dependency connected with glass/epoxy along with carbon/epoxy composites.

## **2.10. Environmental Effects**

Fibrous composites are generally gradually additional used in many purposes on account of different attractive qualities as well as high specific toughness, high specific stiffness and manipulated anisotropy. Yet sad to say, polymeric composites are generally prone to heat and humidity when operating within changing ecological conditions. Pertaining to much better using this kind of programs total knowledge of the particular things of ageing and environmental subjection connected wear and tear is usually of massive magnitude.

### **2.10.1 Effect of Temperature on FRP composites**

Effects of temperature on fibrous polymeric materials are one of the most difficult challenges for neophytes and composite structure designers because it can significantly change the response of the composite materials. A material that exhibits ductile behavior at room temperature may become brittle from low temperatures or maybe may soften and also creep with elevated temperatures [45]. With these temperature fluctuations changes with strength as well as or perhaps stiffness are generally observed. In the same way well. Thermal conditioning imparts better adhesion and thus, an improved ILSS value especially at the less conditioning time [46]. When considering the temperature dependence for the physical properties of many polymeric materials, various relaxation effects are extremely important. In polymer molecules a dynamic mechanical relaxation occurs due to heat transfer between the intermolecular mode (strain-sensitive mode) and the intra-molecular mode (strain-insensitive mode) [47]. Thermal degradation of resin involves chemical reaction and physical changes. Chemical reaction is represented by oxidation, further cross-linking and further reaction of un-reacted monomers, while physical change is the visco-elastic behavior [42]. The visco-elastic yield behavior generally depends upon the temperature and loading rate. At the macromolecular scale chain scission and cross-linking affect the polymer network and thus, alter the mechanical properties of the oxidized layer. At the macroscopic level, the hindered shrinkage of the oxidized layer induces a stress gradient susceptible to initiate and propagate cracks [48]. Thermal conditioning behavior of glass/epoxy and carbon/epoxy composites is of special interest, because of their expanding use for structural applications, where increased temperatures are common environmental conditions. Fracture processes of polymers are strongly influenced by deformation or yielding processes which depend on temperature and time. At very low temperature no yielding is possible and fracture is brittle. At high temperatures two characteristic phenomena occur after the yield point: strain softening and strain hardening. The samples tested at lower temperature are considered by a superior level of micro-cracking and de-lamination. These effects are believed due to higher thermal residual stresses [42]. Due to heterogeneous nature of the fiber and matrix there is large thermal expansion and contraction mismatch which generates thermal stresses. This weakens the fiber/matrix interface as a result their inter-laminar shear strength (ILSS) values decreases progressively.

### **2. 10.1.1. Effect of low temperature**

The cryogenic liquid fuels, which are used as propellants of launch vehicles and rockets in aerospace applications, have low calorific energy to volume ratio. Thus they have to be stored in large and heavy pressurized tanks made of metallic materials. Polymer matrix composites are used as alternative materials for the tanks because of their high mechanical performance as well as low thermal expansion [49, 50]. Fiber composites are proving themselves to be attractive alternatives to even metals because of their high specific strength stiffness their low electrical and thermal conductivities.

However, their disadvantage arises from the weak polymeric matrix and results in low interlaminar shear strength. In the course of cooling to low temperatures, difference in thermal contractions of fiber and matrix gives rise to thermal residual stresses and strains which influence most of the mechanical properties. At low temperatures the majority of currently used matrices are brittle and do not allow relaxation of residual stresses or stress concentrations to take place. Carbon composites exhibit a high stiffness or a high tensile strength, a very low thermal conductivity at low temperatures, and a low or even negative coefficient of thermal expansion which can be achieved at special fiber orientations [51]. In low temperature applications, the brittleness of the cross-linked epoxy resin matrix reduces the mechanical strength, especially under multiaxial load conditions.

Under conditions such as cycling or cryogenic aging from room temperature to cryogenic temperature with loads, micro-cracks develop in the composite matrix due to dissimilar coefficients of thermal expansion (CTEs) between the fiber and matrix. As a result, this structural damage gives rise to the degradation of mechanical properties in the structures, such as fiber/matrix potholing, interfacial debonding or delamination [52]. A better fiber/matrix interfacial adhesion/bond will impart better properties such as interlaminar shear strength, delamination resistance and fatigue resistance to a polymeric composite [53]. The local response of fiber matrix interface within the composite plays an important role in determining the gross mechanical performance [54]. It provides a means of stress transfer from fiber to fiber through the matrix. In addition, the Kevlar fibers have a microstructure of fibrillar bundles with longitudinal voids between them [55]. Accordingly, the transverse and shear strengths and stiffness are very low. Carbon and Kevlar fibers exhibit a very small or even negative coefficient of thermal expansion only in the fiber directions [56].

Thus in low temperature applications, each kind of fiber composite has its definite qualities. A suitable combination of various types of fibre in hybrid composites releases a huge field for optimizing composite properties.

#### **2.10.1.2. Effect of thermal spiking**

Common prior conditioning consequences in these instances are classified as the reputation associated with thermal spike. With very hot and moist conditions, FRP composite process wetness. Water intake causes irritation on the polymer bonded, presenting go up to hygroscopic challenges. Existence associated with thermal shocks and thermal spikes guide to expansion associated with thermal stresses. This reputation of these stresses could lead to matrix damage. In the FRP composite, this soluble fiber and the matrix have various coefficients of thermal expansions (CTE). Therefore, trips over the identical heat range variety results in differential development. That induces further challenges at the fiber-matrix program, and thus worsening this interfacial place. That could lead to debonding and finally delamination on the composite by simply interfacial failure. This shrinking gone through by the composites while in recovering lead to curing stresses. The high cross-linked networks have lower molecular mobility. The mechanical behavior of these composites is different from the composites without any form of thermal conditioning [57]. The state on the composites will become more intricate a result of the filling circumstances throughout their application. The bundled interactions of those elements cause destruction of mechanical attributes as well as succeeding fracture. The fracture amount of resistance of FRP composites may be described based on elongation of material. They will elongate in a stepwise manner because folded away domain names or loops are taken open. The molecular elongation looks to be the key reason for toughness [58]. This phenomenon is actually associated with several concern with aerospace applications, through which rapid temperature variations may occur during take-off.

#### **2.11. Effect of temperature on glass transition temperature of polymer**

Within the examine connected with polymers and the programs, it is comprehend to know the technique of the glass transition temperature,  $T_g$ . As the temperature of the polymer falls down below  $T_g$ , the idea behaves within an significantly brittle manner. As the temperature goes up over the  $T_g$ , the polymer gets additional rubber-like. Therefore, familiarity with  $T_g$  is

crucial inside picking a supplies with regard to several programs. In general, valuations connected with  $T_g$  well down below room temperature specify the website connected with elastomers and valuations over room temperature specify rigid, structural polymers.

This particular behavior may be understood with regards to the design connected with glassy supplies which are produced usually by means of materials comprising prolonged chains, network connected with joined atoms or perhaps those that have a very intricate molecular design. Normally like supplies have a substantial viscosity inside the water point out. When speedy air conditioning happens with a temperature when the crystalline point out is actually anticipated to function as the additional steady, molecular mobility is actually far too gradual or perhaps the geometry far too awkward to take upward a crystalline conformation. Which means haphazard design characteristic of the water continues into conditions when the viscosity is so substantial that the product is actually thought to be stable. The term glassy possesses grown into synonymous which has a chronic non-equilibrium state.

To get additional quantitative about the depiction of the liquid-glass transition phenomenon and  $T_g$ , most of us note that inside air conditioning a amorphous product from your water point out, there isn't any abrupt modify inside level such as happens in the matter of air conditioning of the crystalline product via the freezing point,  $T_f$ .

## **2.12. Effect of Glass transition temperature ( $T_g$ ) on mechanical properties**

$T_g$  is used for evaluating the flexibility of a polymer molecule and the type of response the polymeric material would exhibit to mechanical stress. Polymers above their  $T_g$  will exhibit a delayed elastic response (visco-elasticity), while those above  $T_g$  will exhibit dimensions stability. General commonsense prevails that higher the  $T_g$  better the mechanical properties.

Another significant property connected with polymers, furthermore powerfully dependent on their temperature ranges, is actually their reply to the effective use of some sort of force, since indicated by simply a couple principal forms of behavior: elastic and plastic. Elastic materials will probably come back to their unique design once the force is actually taken out. Plastic materials will never get back their design. Within Plastic materials, circulation is occurring, much like a highly viscous fruit juice. Nearly all materials illustrate a mixture of elastic and plastic behavior, displaying plastic behavior following elastic materials have been realized. Glass is just about the number of fully elastic material even as it is actually under it

is  $T_g$ . It will eventually continue to be elastic until that grows to its breaking point. The  $T_g$  connected with glass takes place concerning 510 and 560 degrees C, which means that that will be some sort of brittle solid on room temperature.

### 2.13 The failure study of FRP Composite

Fractographic investigation may be used to research micro-mechanisms connected with fracture connected with operating components. The fundamental approach should be to separate your fracture morphologies of the samples failed under various inability settings and then comparison these types of morphologies to help unidentified inability.

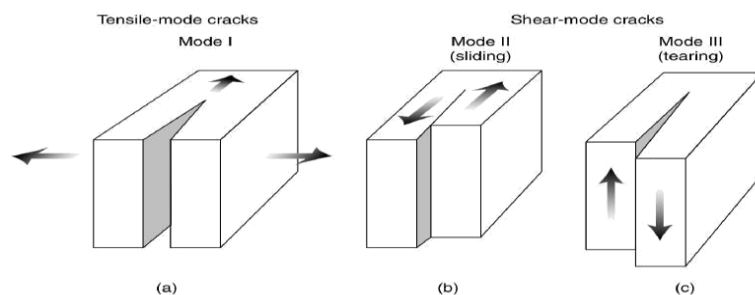
Inside composites the main factors that cause failure to be

1. Delamination
2. Debonding (separation of fibres and matrix)
3. Matrix Microcracking
4. Fibres Fracture
5. Cusps and fiber imprints
6. Matrix rolling

The different failure modes in composites are of three types

1. Interlaminar
2. Intralaminar
3. Translaminar.

At the point when measured on microscale, intralaminar and interlaminar crack sorts could be comparably described. Crack occurs in the direction plane parallel to the fiber reinforcement. In an equivalent way to that depicted for conventional metals, break of any sort can happen under mode I tension, mode II in-plane shear, mode III anti-plane shear, or any blend of these load conditions. Translaminar cracks are those situated transverse to the laminated plane in which states of fiber fractures are produced.



**Figure 2.16: Schematic of Crack opening modes.**

### 2.13.1. DELAMINATION:

Delamination is the basic damage form in composite materials, not so much in light of the fact that it will result in the structure to break into two or more parts, since it can debase the laminate to the point that it gets futile in service. The interfacial division caused by delamination may guide to untimely buckling of laminate, loss of fatigue life, intrusion of moisture, excessive vibration and stiffness degradation [30].

Delamination can unveiled throughout control or in overhaul surroundings. It could outcome from low pace result, from eccentricities inside the structural fill journey and from discontinuities inside the buildings which stimulate an excess of plane stress.

Also inside the nonexistence of such lack of continuity of delamination might also outcome in plane compressive load leading to local buckling. Along with mechanical load, the actual temperature and humidity might also stimulate interlaminar stresses within a laminate. The delamination may result in redistribution concerning stresses that will finally encourage failure.

Irwin's methods is one's free rate across delamination is the front usually are worked out regarding linear elastic fracture movement and successive improvements by simply Rybicki and Kanninen. The energy unveiled with the self-similar propagation of the fracture regarding duration „a“ to that particular of the + Δa because of sequential thermo-mechanical running is just the job forced to near the actual fracture from the + Δa duration for you to „a“. The fracture development setting as shown in figure 3, the worries vitality unveiled from the delamination extendable is equivalent to job forced to near the actual fracture.

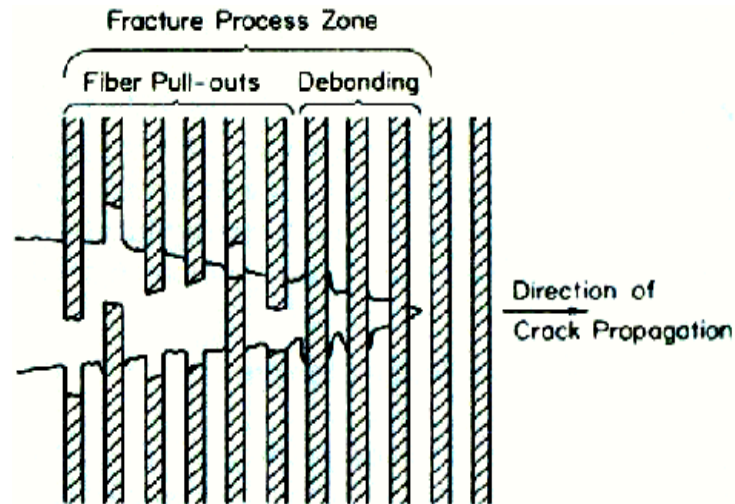
$$W = \frac{1}{2} \int_0^{\Delta a} [\sigma_M(n) + \sigma_T(n)][\delta_M(n - \Delta a) + \delta_T(n - \Delta a)] dn$$

Where the subscripts “T” and “M” represent respectively the thermal and mechanical effects of denoted specifications.  $\sigma(x)$  is the stress at the crack front required to close the delaminated area and  $\delta(x - \Delta a)$  is the crack opening displacement between the top and bottom delaminated surface.

For a straight-edged crack front, the curvature plane and normal is constant everywhere. So mode definition is intuitive and constant for the entire front. Then the energy rate is calculated as [59]:







**Figure 2.18: Crack tip shows direction of crack propagation.**

Any time brittle materials tend to be nicely bonded to some ductile matrix, these materials have a tendency to snap prior to the crack tip, leaving behind links connected with matrix substance that fret board down in addition to fracture inside a entirely ductile fashion. As well as most of these local failing mechanisms, with achieving this program of the two laminate inside a laminated.

With fibre reinforced materials using both equally brittle matrices in addition to brittle materials, robustness is evolved from couple of ways. Firstly, should the crack could be complete to function in every single fibre throughout it is path this you will see a large amount of brand recent surface area designed to really tiny enhance throughout break location verticle with respect towards greatest primary pressure -- Interfacial Strength -- in addition to to get this materials to help crack they need to possibly be filled for their fracture strength and also this frequently demands added community variable perform, in addition to subsequently should the materials tend not to crack and thus bridge this gap subsequently perform has to be carried out to help draw this materials. With geometric models we can calculate the input of every process in terms of general toughness of the composite [60].

### **2.13.3 Matrix Microcracking**

The very initial type of crack throughout laminates is normally matrix micro cracking. These are ply fractures that traverse actual depth from the work in addition to function parallel towards the fibers from the ply [61]. Commonest visible micro damage is damage inside nine hundred plies while in axial loading inside the path. These kinds of micro fractures tend to be

transverse towards the loading path and are also usually termed as the actual transverse fractures. Thermal cycling, environment, fatigue loading, and tensile loading can every one direct to microcrack development [62]. Microcracks will application form with any ply which has a significant component of your applied populate transverse for the fibers in That ply. Microcracks lead in order to degradation in properties of the laminate like changes in effective moduli, Poisson ratios, along with thermal expansion coefficients [63]. while these changes usually are sometimes small, microcracks will nucleate various other forms involving damage.

#### **2.13.4. Fiber Fracture**

If how much stress currently being employed struggles to end up being permanent from the material any time allocated on fibre from the matrix because of incapability from the development of sturdy interfacial bonds, breaking of fibers may well take place. This tends to happen in the event the glass fiber can be aptly held from the matrix and you will be and then this shatter from the material which is often due to strain field and localized stress from the polymeric composite.

# Chapter 3

## Experimental Details

### 3. Experimental details

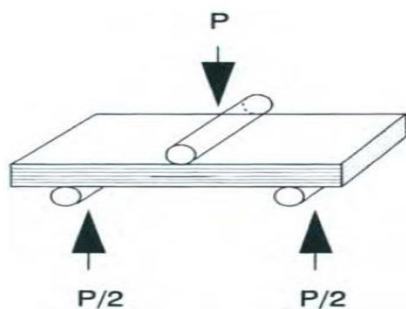
#### 3.1. Materials

1. Woven fabric E-Glass Fibers (FGP, RP-10)
2. Woven fabric Carbon Fibers (TC-33)
3. Epoxy Resin (Lapox,L-12) based on Bisphenol A
4. Hardener (K 6, primary amine)

#### 3.2. Experimental Methods

##### 3.2.1. Short Beam Shear (SBS) Test or Flexural Test

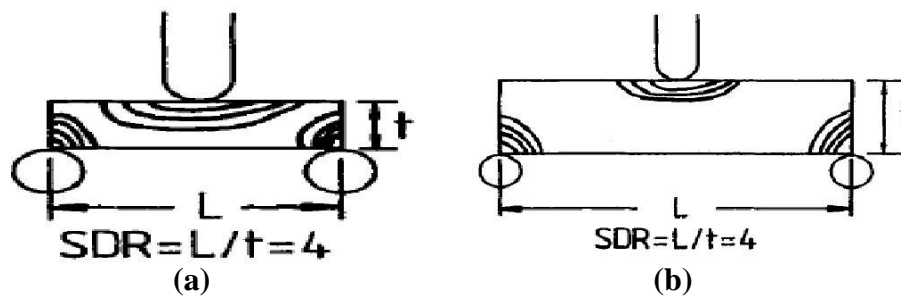
The flexural technique is relevant to fibre reinforced polymer (FRP) composite materials. It is a testing mechanism with convenient loading rate, which is used in combination with a loading fixture. The three point flexural test done on a sample with a minute span, which stimulate failure by interlaminar shear [65]. While shear stress is usually involved in the beam encountered with the bending load, which is directly proportional to the magnitude in the employed load along with independent of the span length. So the support span in the short beam shear (SBS) sample is usually kept short to produce a great interlaminar shear failure ahead of bending failure.



**Fig. 3.1 Schematic of loading configuration of short beam shear test**

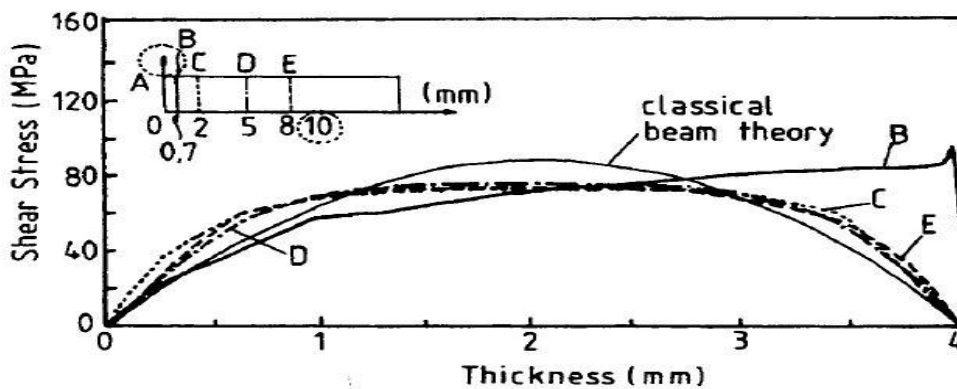
The Flexural technique test is well defined by ASTM D 2344, which describes a span length to sample thickness ratio of five for lower stiffer composite materials and four for higher stiffer composite materials. The test has a natural problem related with thenon-linear plastic deformation and the stress concentration incorporated by the loading roller nose of small diameter. This is schematically shown in fig 3.2, where the effects of stress concentration in a thin sample are compared with those in a thick sample. In both cases the samples have the similar span-to-depth ratio (SDR). Therefore the stress condition is much further complex

than the pure shear stress condition given by the simple beam theory.



**Figure: 3.2 Effect of stress concentrations on short beam shear samples: (a) thin sample;(b) thick sample [9].**

In relation to the classical beam theory, the distribution of shear stress along the thickness of the sample is a parabolic function, which is equivalent about the neutral axis where it is highest and then decreases to zero at the tensile and compressive profile. In actual cases, the stress field is influenced by the stress concentration close to the loading roller nose, which totally demolishes the parabolic shear distribution used to evaluate the evident ILSS. Detailed studies about the flexural technique have been done by several investigators to identify the imperfection.



**Figure: 3.3 Shear stress distributions across the thickness of a three-point bending sample in a short beam shear test [9]**

The experimental show that by considerably increasing the diameters of the support and loading cylinders and increasing the span to length thickness ratio to about eight. With these variations the results shows that the parabolic division of shear stress throughout the thickness of the sample anticipated by simple beam theory could be extremely good in the areas between the loading roller and support cylinder so that the sample fail in a shear mode [66]. With these improvements ASTM D 2344 could so far turn into a technically appropriate with accepted shear test method.

### 3.2.2. Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) has been a well established instrument for several years in the inspection of fracture surfaces. The well-known imaging advantages are the vast depth of field and elevated spatial resolution and the image is reasonably simple to understand with naked eye.

The various fractographic study of the tested samples were carried out by the help of JEOL-JSM 6480 LV SEM. The samples were stacked onto the sample holder and located inside the SEM. Altering the working distance, so that the spot size the chamber was closed and vacuum was applied.

**Principle:** when an electron beam is focused finely through the scanned surface of the sample generates secondary electrons, backscattered electrons, and characteristic X-rays. These signals were composed by detectors to construct images of the sample displayed on a cathode ray tube screen. The description seen in the SEM figure may then be directly examined for elemental composition using EDS or WDS. The electrons that are emitted from the sample surface have a spectrum of energies. Conventionally there is a separation of energies in between the secondary and backscattered electrons. When the emission of electron energy is less than 50eV, it is called as secondary electron and backscattered electrons are considered to be the electrons that exit the sample with energy greater than 50eV. A significant point in understanding the formation of SEM images of fractured surfaces, and their explanation, is an appreciation of the factors that affect this energized volume of electrons in the sample [67]. To identify the different failure mechanisms in FRP composites, micrographs were taken using a SEM. There is a remarkable change in the structure and properties of the composite when exposed to low and high temperatures.



**Figure: 3.4 Scanning Electron Microscope (JEOL-JSM 6480 LV SEM)**

### 3.2.3. Differential Scanning Calorimetry (DSC)

The Differential Scanning Calorimetry (DSC) measurements were being performed simply by the help of some sort of Mettler-Toledo 821 instrument having intra cooler, with the STAR software. The calculations of times continual, temperature calibration from the instrument were being carried out simply by higher level of In and Zn, along with the heat flow calibration having Within. The heating rate for the particular small sample is about 10°C/min. To be able to adjust the heat flow signal, some sort of write off manage through an empty pan around the research facet and a clear pan as well as a cover for the small sample facet ended up being carried out ahead of the small sample sizes. Standardised aluminium lightweight pans were being used to spot the particular samples. The examination ended up being appropriate within the temp selection of 25°C to 120°C.



(a)



(b)

**Figure: 3.5 (a) Mettler -Toledo 821 instrument along with intra cooler with regard to DSC measurements and (b) reference sample chamber**

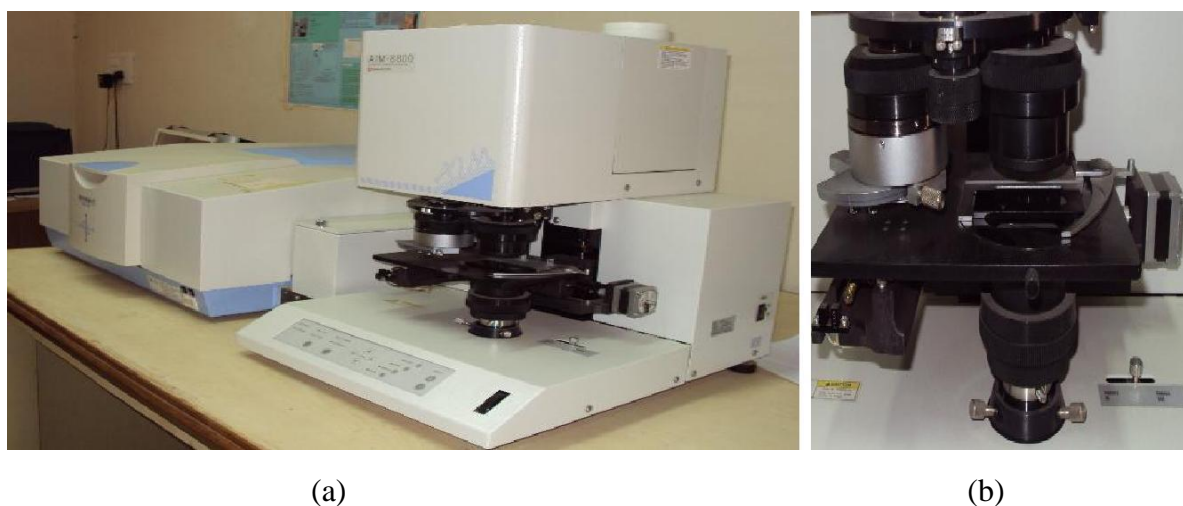
**Principle:** Differential scanning calorimetry (DSC) is a thermoanalytical method essential to set up the just about no temperatures distinction in between a great inert reference material plus the sample, while two samples are usually subjected to identical temperatures set up in an natural environment mind or even cooled for a licensed price.

### 3.2.4. FTIR-ATR Spectroscopy analysis

**Principle:** With infrared spectroscopy, infrared the radiation can be handed down through a test. Some of the infrared the radiation can be consumed because of the test and a few of the usb ports can be handed down as a result of (transmitted). The particular ensuing array shows this molecular absorption and transmission, setting up a molecular fingerprint from the test.



As being a fingerprint simply no a pair of exclusive molecular structures generates the identical infrared array. That tends to make infrared spectroscopy useful for various kinds of analysis. Infrared spectroscopy pays to in the area to acquire structural data regarding organic compounds. This region is divided into two parts i.e. the functional group region  $4000-1300\text{ cm}^{-1}$  and  $1300-650\text{ cm}^{-1}$  finger print region. Most of the functional groups give absorption bands in the high frequency part of the spectrum, which give small number of bands [68]. The FTIR image analysis suggests that there is a variation in the chemical structure of the matrix from the fiber to the bulk polymer [69].



**Figure: 3.6 (a) FTIR spectrophotometer, (b) AIM-800 Automatic Infra red Microscope**

This is the technique for keeping track of the transportation process of low molecular weight species had been set up relying on these types of concepts. This particular empowered the keeping track of regarding individual species in-situ, whilst providing added substance home elevators any improvements that could be developing over the transportation practice. The in-situ assessment regarding diffusivity relies on the opportunity to calibrate the FTIR-ATR spectra attained. The IR absorbance values of a sample usually are in connection with its concentration, analogous for the method attention relates to absorbance within sign spectroscopy from the Beer-Lambert regulation.

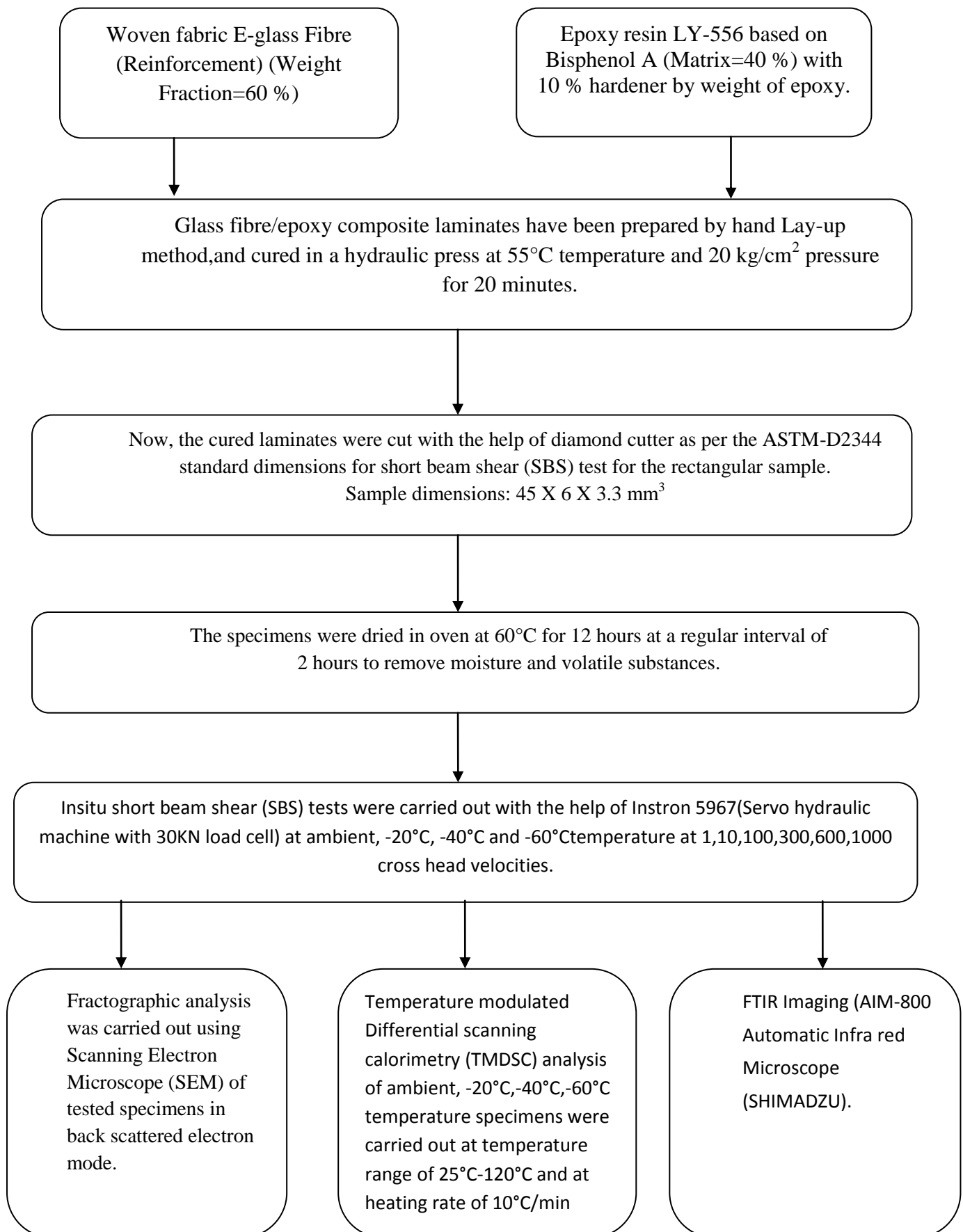
The region of the infrared spectrum from  $4000\text{cm}^{-1}$ - $1400\text{cm}^{-1}$  exhibit absorption bands falls under the functional group regions. The FTIR aids in identifying chemical bonds, and thus chemical composition of materials. FTIR imaging suggests that there is a chemical gradient near the interface of the composite materials [68].

### 3.3. Experimental Procedure

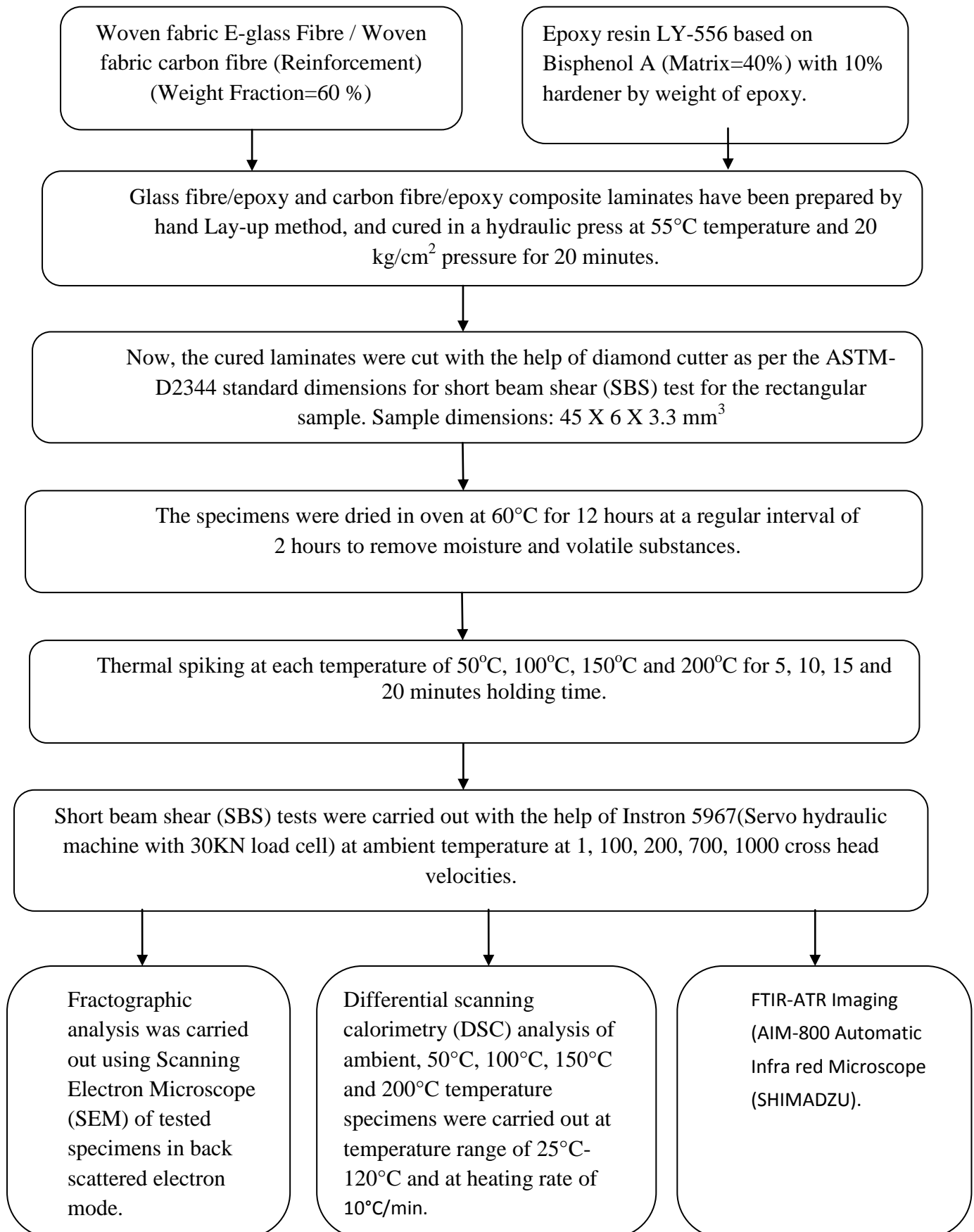
Woven fabric E- glass fibre were cut into the size of 25 cm X 20 cm to form 16 layer sheets (laminates) and weighed. LY-556 epoxy resins based on Bisphenol A is weighed to be 40% of the total weight of the fibre and epoxy resin. For fabrication of glass/epoxy (weight fraction of glass fibre is 60%) and of carbon/epoxy (weight fraction of glass fibre is 60%). Then, Hardener HY 951 (aliphatic primary amine) at the ratio of 10% by weight of epoxy resin was used. Glass fibre/epoxy and carbon fibre/epoxy composite laminate have been prepared by hand lay-up method and cured in a hydraulic press by compression moulding method at 55° C temperature and 20 kg/cm<sup>2</sup> pressure for 20 minutes. Now, the cured laminates were cut with the help of diamond cutter as per the ASTM-2344 standard dimensions for short beam shear (SBS) test specimens. The specimens were dried in oven at 60° C for 12 hours at a regular interval of 2 hours to remove moisture and volatile substances. In-situ short beam shear (SBS) tests of glass/epoxy specimens were carried out with the help of INSTRON 5967 ( Servo hydraulic machine with 30 KN load cell) at ambient, -20°C, -40°C and -60°C temperature at 1, 10, 100, 300, 600,1000 mm/min loading rate. Fractographic analysis was carried out using scanning electron microscope (JEOL-JSM 6480 LV SEM) of ambient and low temperature treated samples in back scattered electron mode. The TMDSC( Temperature modulated differential scanning calorimetry) were done by Mettler-Toledo 821 and analysis of ambient and low temperature treated specimens were carried out at a temperature range of 25°C-120°C at a heating rate of 10°C/min. Then, the FTIR (Fourier transform infrared spectroscopy) imaging was performed in AIM-800 Automatic Infra red Microscope (SHIMADZU).

Similarly, thermal spiking of glass/epoxy and carbon /epoxy specimens was done at 50°C, 100°C,150°C and 200°C for 5,10,15 and 20 minutes holding time at each temperature. The 3-point bend test (ASTM standard D2344) using INSTRON 5967 at a loading rate of 1, 100, 200, 700, and 1000 mm/min was carried out. Fractographic analysis was carried out using scanning electron microscope (JEOL-JSM 6480 LV SEM) of ambient and low temperature treated samples in back scattered electron mode. The TMDSC( Temperature modulated differential scanning calorimetry) were done by Mettler-Toledo 821 and analysis of ambient and low temperature treated specimens were carried out at a temperature range of 25°C-120°C at a heating rate of 10°C/min. Then, the FTIR (Fourier transform infrared spectroscopy) imaging was performed in AIM-800 Automatic Infra red Microscope (SHIMADZU).

### 3.3.1. Effect of low temperature on Glass fibre/epoxy composite



### 3.3.2 Effect of Thermal Spiking on Glass fibre/epoxy and Carbon fibre/epoxy composite



# **Chapter 4**

## **Results and Discussions**

## 4. Results and Discussions

### 4.1. Effect of low temperature and loading rate on interlaminar shear strength (ILSS) of glass/epoxy composites

#### 4.1.1. Short beam shear (SBS) test

The interlaminar shear strength (ILSS) of low temperature conditioning samples during short beam shear test can be calculated as  $ILSS = 0.75P/bd$

Where, P is maximum load, b is the width of specimen and d is the thickness of specimen.

#### 4.1.1.1. Effect of loading rate on the interlaminar shear strength (ILSS) of glass/epoxy composite

The variation of ILSS Vs loading rate for the ambient and low temperature conditioned glass/epoxy composite is shown in the figure 4.1.

The tests which were carried out at ambient temperature and at 1, 10, 100, 300, 600, 100 mm/min loading rates reveal that initially the ILSS increases with the loading rate upto 300mm/min and then it decreases. The lower ILSS is at 1mm/min loading rate and the higher ILSS is obtained at 300 mm/min loading rate.

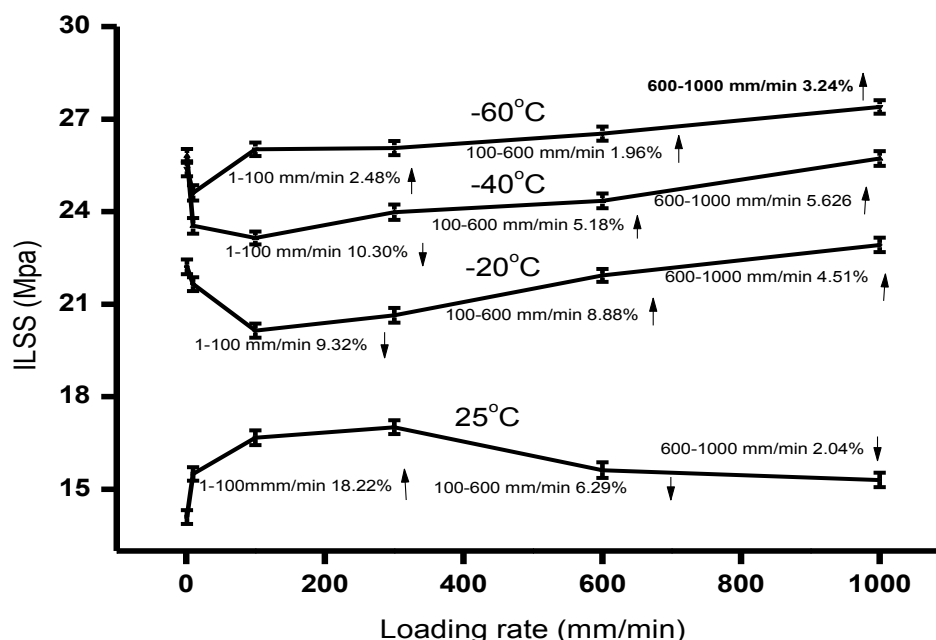


Figure 4.1: Comparison of ILSS Vs Loading Rate at 25°C, -20°C, -40°C and -60°C temperatures.

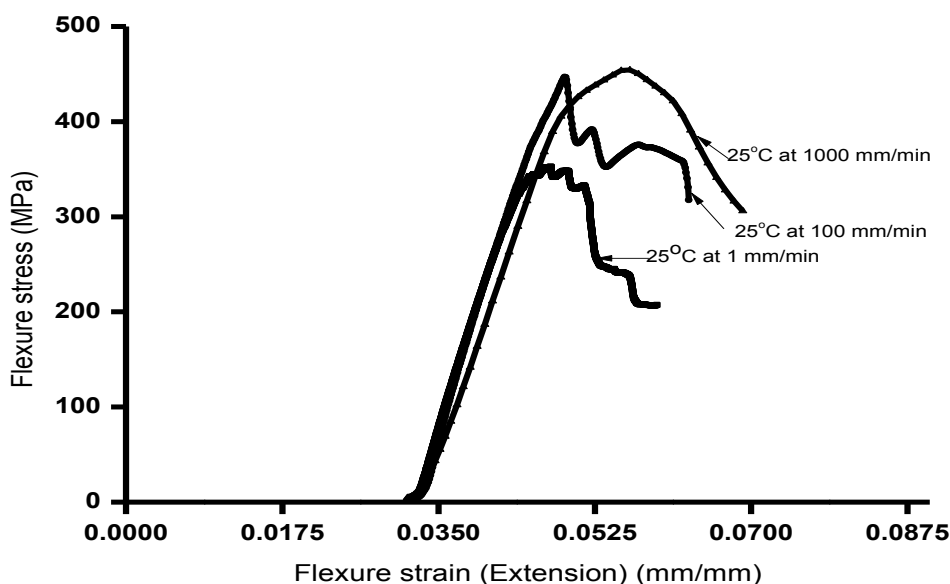
There is an increase of 18.22 % in ILSS value from 1 to 100 mm/min loading rate. In the context, there is a decrease of 6.29 % in ILSS value from 100 mm/min to 600 mm/min loading rate. From 600 mm/min to 1000 mm/min loading rate there is slight decrease of 2.04 % in the ILSS value.

The tests which were carried out at -20°C temperature & at 1, 10, 100, 300, 600 and 1000 mm/min loading rates shows that the ILSS decreases with increase in loading rate upto 100mm/min and then it increases. The lower ILSS is at 100 mm/min loading rate and the higher ILSS is obtained at 1000 mm/min. In the context, there is a decrease of 9.32 % in ILSS value from 1 mm/min to 100 mm/min loading rate. There is an increase of 8.88 % in ILSS value from 100 to 600 mm/min loading rate. From 600 mm/min to 1000 mm/min loading rate there is slight increase of 2.04 % in the ILSS value.

The test which was carried out at -40°C temperature & at 1, 10,100,300,600,100 mm/min loading rates shows that the ILSS decreases with the loading rates upto100mm/min and then its increases. The lower ILSS is at 100 mm/min loading rate and the higher ILSS is obtained at 1000 mm/min loading rate. There is a decrease of 10.30 % in ILSS value from 1 mm/min to 100 mm/min loading rate. From 100 mm/min to 600 mm/min loading rate there is slight increase of 5.18 % in the ILSS value. Similarly, from 600 mm/min to 1000 mm/min loading rate there is increase of 5.62 % in the ILSS value.

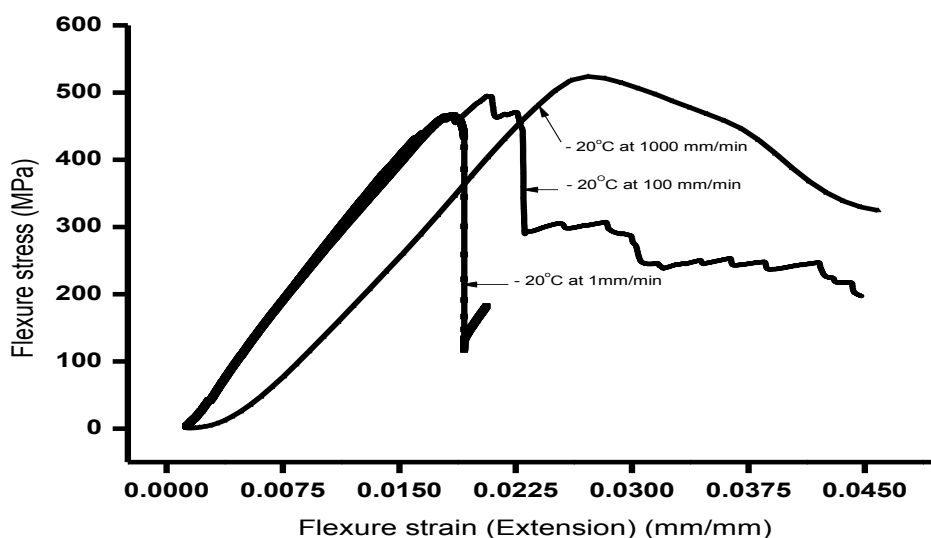
The test which was carried out at -60°C temperature & at 1, 10,100,300,600,100 mm/min loading rates shows that the ILSS decreases with the loading rates upto10 mm/min and then its increases. The lower ILSS is at 10 mm/min loading rate and the higher ILSS is obtained at 1000 mm/min loading rate. In terms of percentage, there is an increase of 2.48 % in ILSS value from 1 mm/min to 100 mm/min loading rate. There is an increase of 1.96 % in ILSS value from 100 to 600 mm/min loading rate. From 600 mm/min to 1000 mm/min loading rate there is increase of 3.24 % in the ILSS value.

#### 4.1.1.2. Effect of loading rate on stress vs strain behavior of glass/epoxy composite



**Figure 4.2: Stress Vs Strain plot at 25°C with different loading speed**

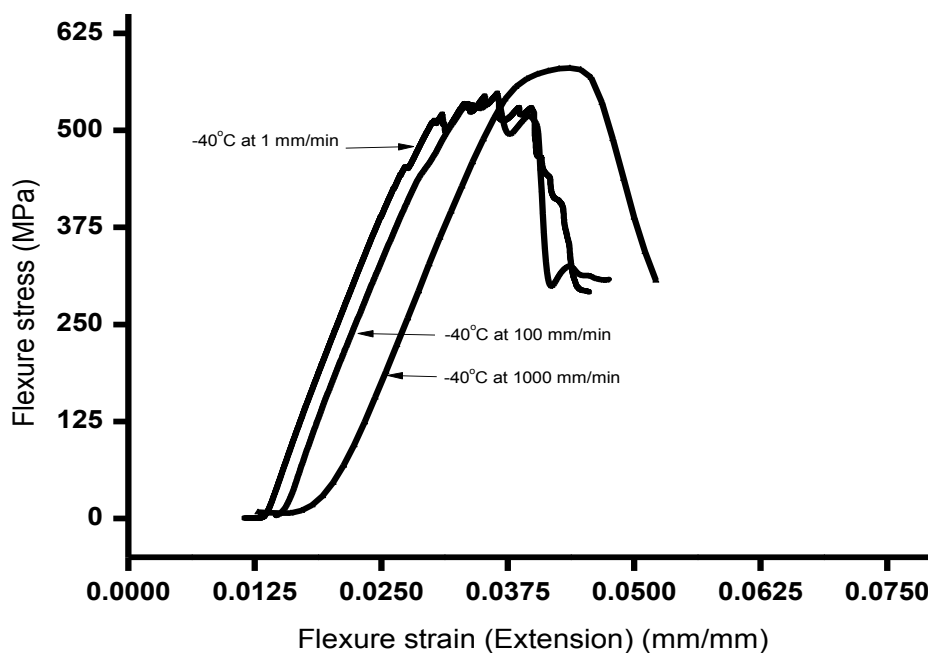
The variation of Stress Vs Strain at 25°C with 1, 100 and 1000 mm/min loading rate is shown figure 4.2 and it shows that maximum stress of glass/epoxy composite was obtained at 1000 mm/min loading rate. The curve at 25°C temperature with 1 mm/min loading rate reveals successive stepped behavior due to fibre breakage. The curve at 25°C temperature with 100 mm/min loading rate shows sharp fall due to delamination of fibres. The curve at 25°C temperature with 1000 mm/min loading rate reveals compression side damage mechanism.



**Figure 4.3: Stress Vs Strain plot at -20°C with different loading speed**

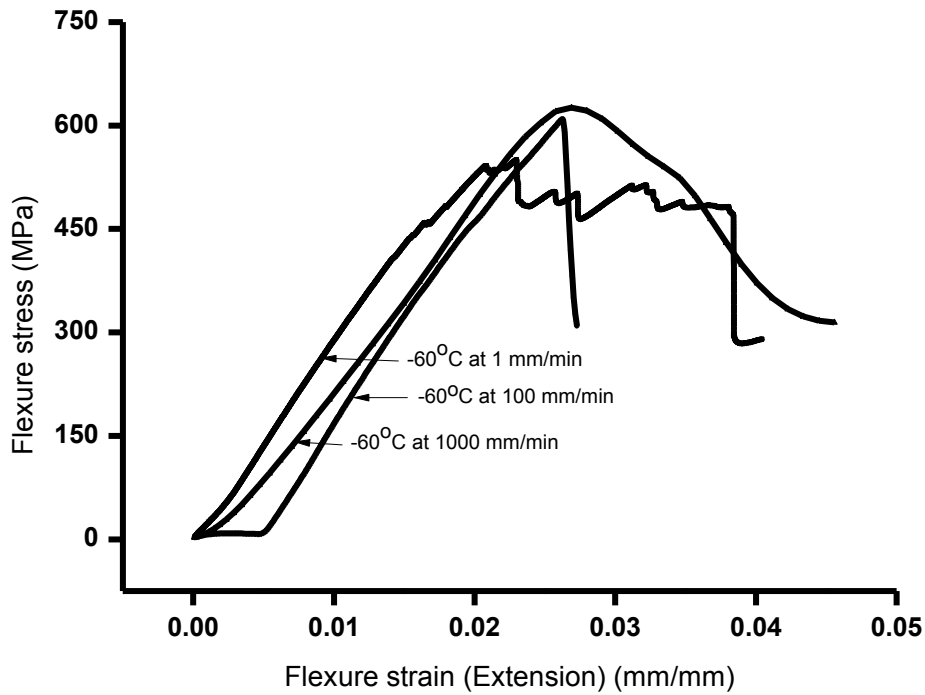


The variation of Stress Vs Strain at  $-20^{\circ}\text{C}$  with 1, 100 and 1000 mm/min loading rate is shown figure 4.3 and it shows that maximum stress of glass/epoxy composite was obtained at 1000 mm/min loading rate. The curve at  $-20^{\circ}\text{C}$  temperature with 1 mm/min loading rate reveals sharp fall due to delamination of fibres. The curve at  $-20^{\circ}\text{C}$  temperature with 100 mm/min loading rate reveals successive stepped behavior due to fibre breakage. At higher loading rate i.e. at 1000 mm/min the composite behavior is governed by reinforcement.



**Figure 4.4: Stress Vs Strain plot at  $-40^{\circ}\text{C}$  with different loading speed**

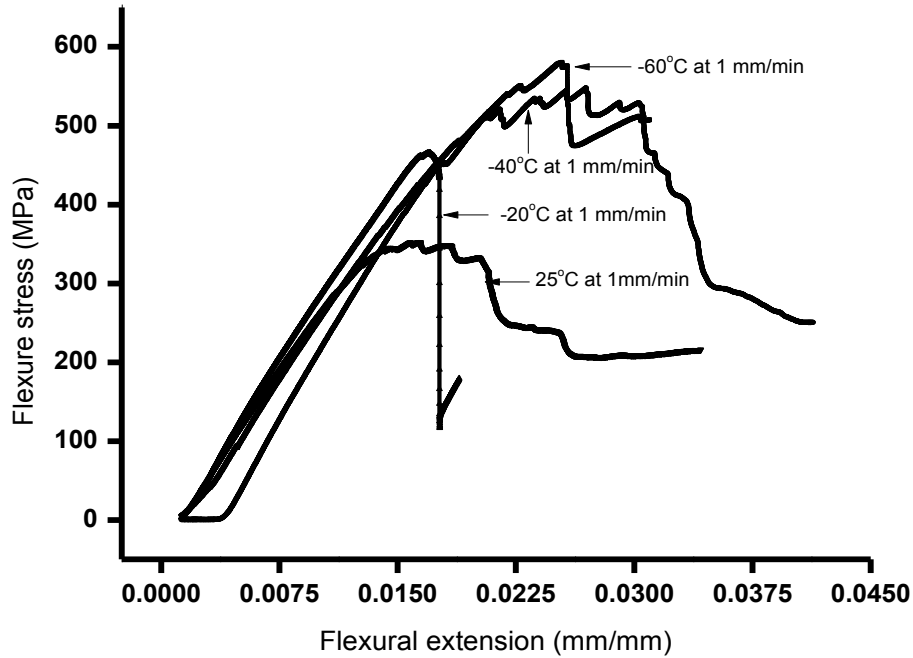
The variation of Stress Vs Strain at  $-40^{\circ}\text{C}$  with 1, 100 and 1000 mm/min loading rate is shown figure 4.4 and it shows that maximum stress of glass/epoxy composite was obtained at 1000 mm/min loading rate. The curve at  $-40^{\circ}\text{C}$  temperature with 1 mm/min loading rate reveals initial damage of fibre/ epoxy specimen below loading roller and then delamination of fibre occurs. The curve at  $-40^{\circ}\text{C}$  temperature with 100 mm/min loading rate shows successive stepped behavior due to fibre breakage. At higher loading rate i.e. at 1000 mm/min the composite behavior is governed by reinforcement.



**Figure 4.5: Stress Vs Strain plot at -60°C with different loading speed**

The variation of Stress Vs Strain at -60°C with 1, 100 and 1000 mm/min loading rate is shown figure 4.5 and it shows that maximum stress of glass/epoxy composite was obtained at 1000 mm/min loading rate. The curve at -60°C temperature with 1 mm/min loading rate reveals initial damage of fibre/ epoxy specimen below loading roller and then delamination of fibre occurs. The curve at -60°C temperature with 100 mm/min loading rate shows sharp fall due to delamination of fibres. The curve at -60°C temperature with 1000 mm/min loading rate reveals compression side damage mechanism.

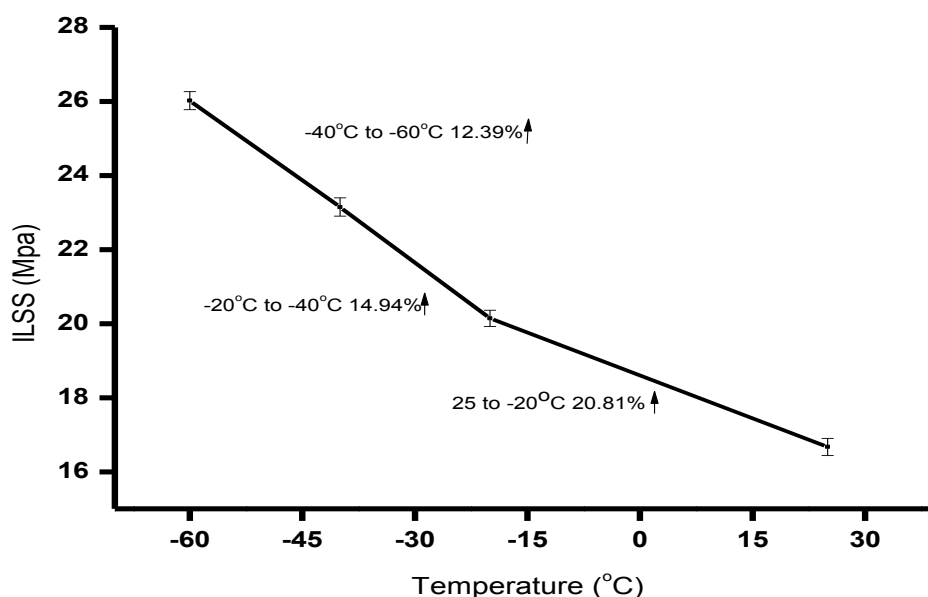
### 4.1.1.3. Effect of temperature on stress vs strain behavior of glass/epoxy polymer composite



**Figure 4.6: Stress Vs Strain plot at 1mm/min loading speed and at different temperature**

The variation of Stress Vs Strain at 1 mm/min loading rate with 25°C, -20°C, -40°C and -60°C temperatures are shown in figure 4.6 and it shows that maximum stress of glass/epoxy composite was obtained at -60°C temperature and at 1 mm/min loading rate. The curve at 25°C temperature reveals successive layer breakage of fibre with lower stress value. The curve at -20°C temperature depicts sharp fall which results in delamination of fibres. The curve at -40°C temperature shows tooth saw profile and successive layer breakage of fibre due higher strength value. Similarly, the curve at -40°C shows initial damage of fibres below loading roller and then delamination of fibres occurs.

#### 4.1.1.4. Effect of temperature on interlaminar shear strength (ILSS) behavior



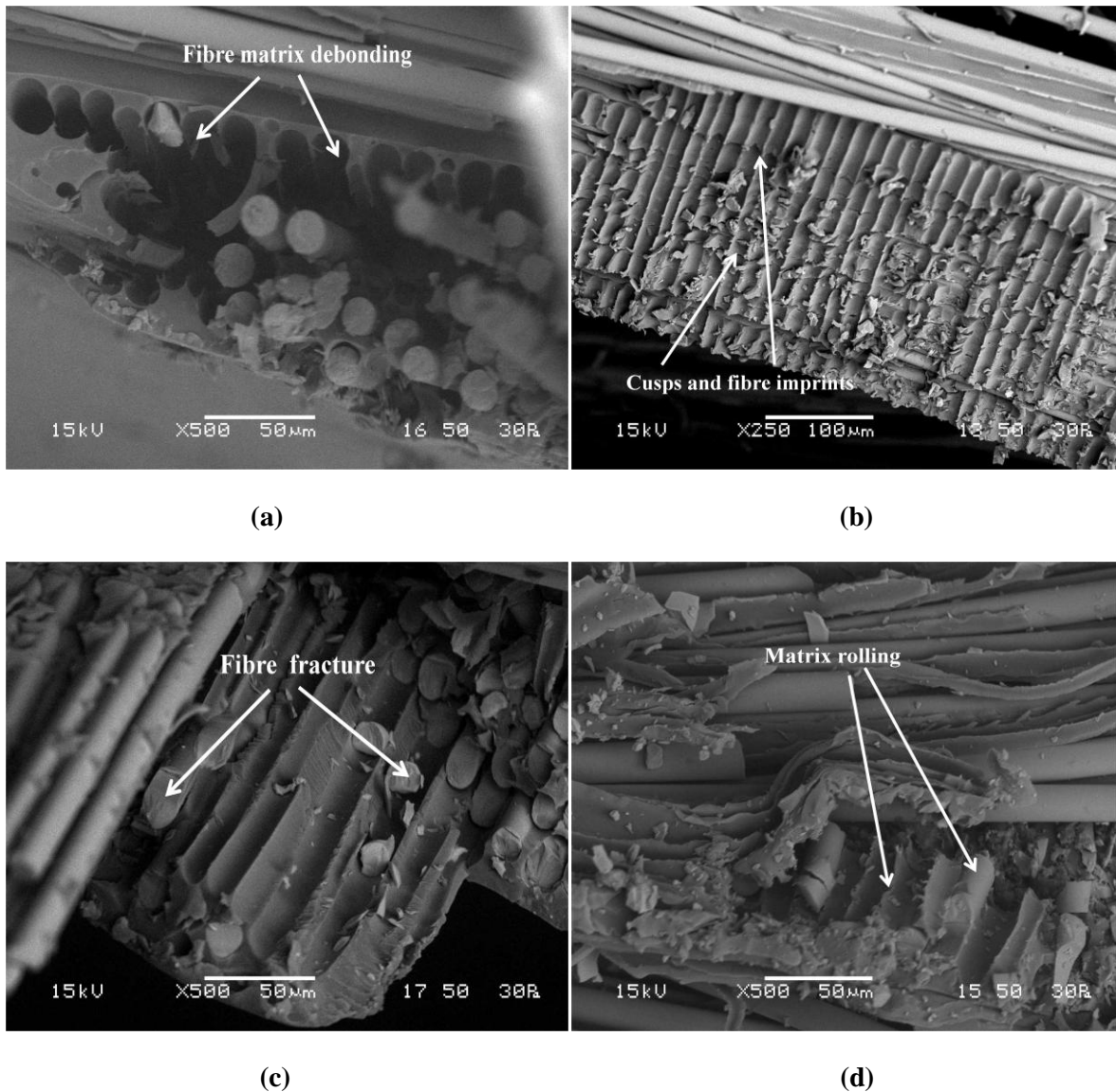
**Figure 4.7: ILSS Vs Temperature plot at 100 mm/min loading rate**

The variation of interlaminar shear strength (ILSS) Vs temperature of glass/epoxy composite at 100 mm/min loading rate is shown in the figure 4.7. It is evident from the figure that as the temperature decreases from ambient to  $-60^{\circ}\text{C}$  temperature, the interlaminar shear strength (ILSS) of the glass/epoxy composite increases. From ambient temperature to  $-20^{\circ}\text{C}$  temperature, the increase in ILSS value is 20.81 %. There is an increase of 14.94 % in the ILSS value from  $-20^{\circ}\text{C}$  to  $-40^{\circ}\text{C}$  temperature. Similarly, from  $-40^{\circ}\text{C}$  to  $-60^{\circ}\text{C}$  temperature, the increase in ILSS value is 12.39 %.

#### 4.1.2. Failure analysis by SEM

The Scanning electron micrographs (SEM) shows different modes of failure at ambient and low temperatures at different loading rates.

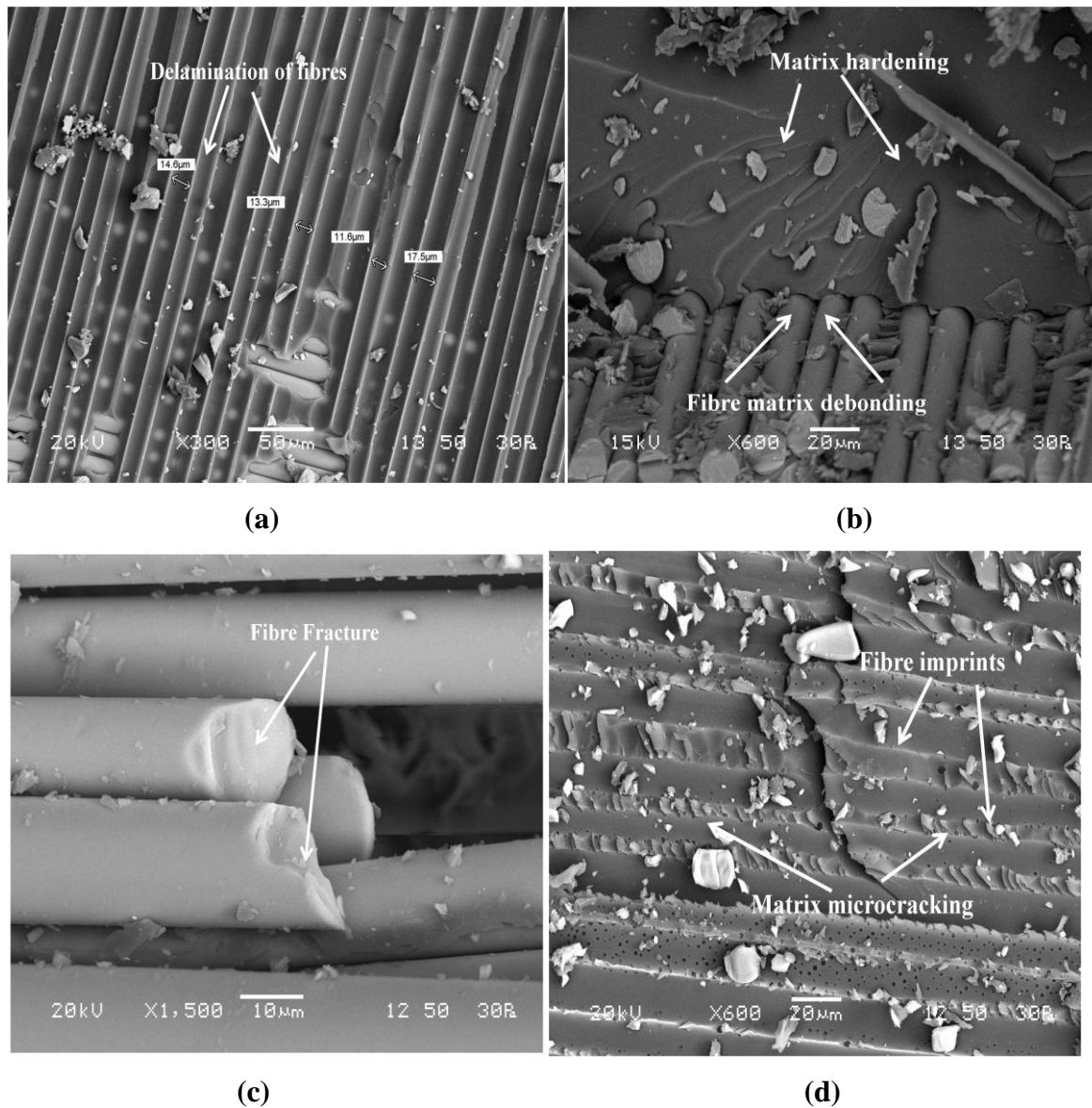
The Figure 4.8 shows scanning electron micrographs (SEM) at ambient temperature at 1, 100, 300 and 1000 mm/min loading rate. The figure 4.8(a) shows fiber-matrix debonding and it is due to weaker fiber/matrix interface. The figure 4.8(b) shows cusps and fiber imprints due to development of cusps when the local stress state in the resin rich layer between plies is such that microcrack form at an angle to the interface. The figure 4.8(c) shows fiber fracture due to when the fibre undergoes maximum strain limit it fails. The figure 4.8(d) shows matrix rolling due to high load transfer along the matrix surface and the matrix makes a rolls like structure.



**Figure 4.8: Scanning Electron Micrographs(SEM) at 25°C at (a) 1mm/min loading rate (b) 100 mm/min loading rate (c) 300 mm/min loading rate (d) 1000 mm/min loading rate.**

The Figure 4.9 shows scanning electron micrographs (SEM) at -20°C temperature at 1, 100 and 1000 mm/min loading rate. The figure 4.9(a) shows delamination of fibres may provide stress relief and lead to premature buckling of the laminate, excessive vibration, intrusion of moisture, stiffness degradation and loss of fatigue life. The figure 4.9(b) shows fibre matrix debonding and it is due to weaker fiber/matrix interface. The figure 4.9(c) shows fiber fracture due to when the fibre undergoes maximum strain limit it fails. The figure 4.9(d) shows matrix microcracking is prominent at higher loading rate, which absorb energy and

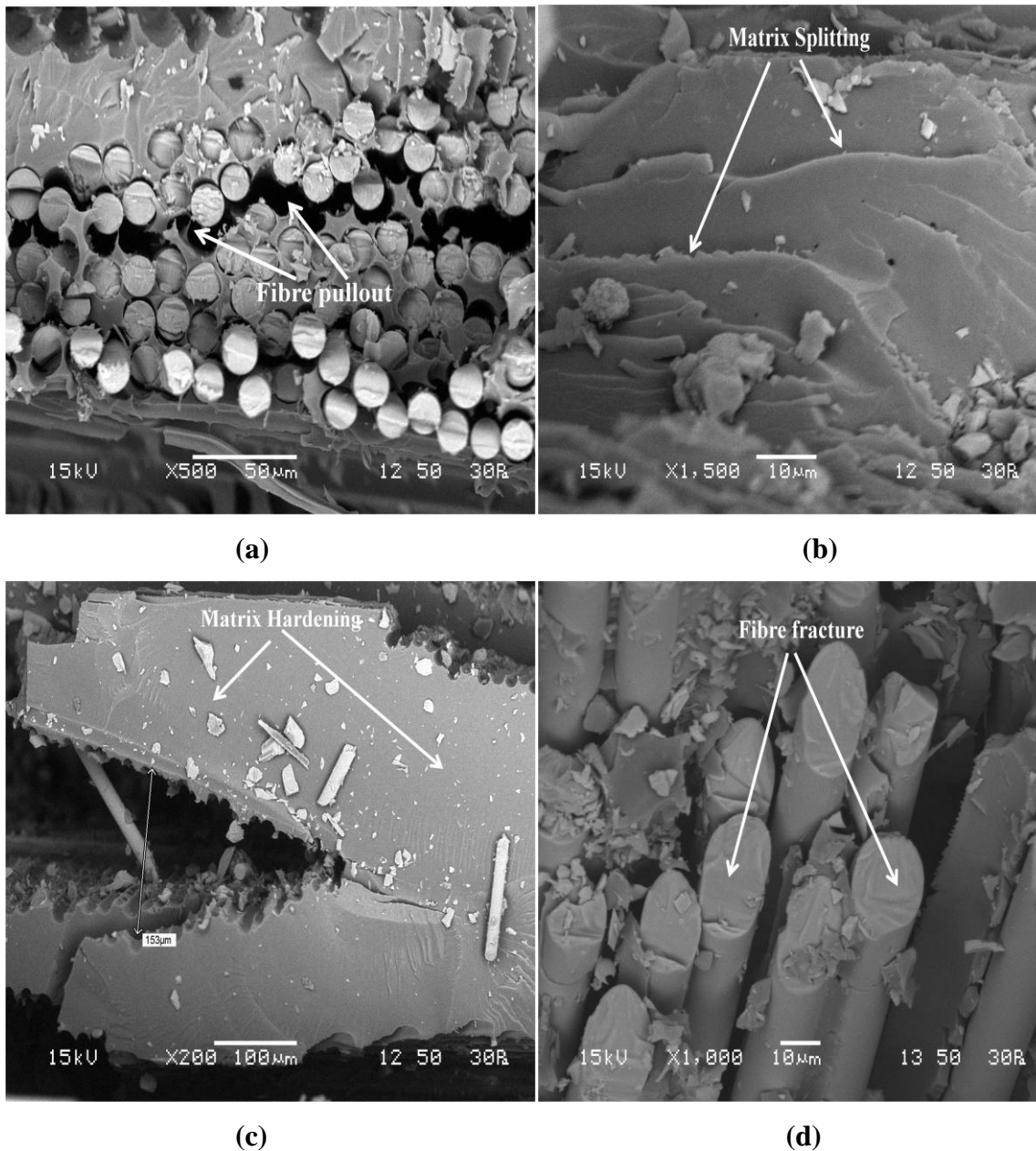
thereby increase the fracture toughness of the fibrous composite, and does not allow it to fail catastrophically.



**Figure 4.9: Scanning Electron Micrographs(SEM) at -20°C at (a) 1mm/min loading rate(b) 100 mm/min loading rate (c) 1000 mm/min loading rate (d) 1000 mm/min loading rate.**

The Figure 4.10 shows scanning electron micrographs (SEM) at -40°C temperature at 1, 100 and 1000 mm/min loading rate. The figure 4.10(a) shows fibre pullout, which is due to the weak adhesion between the fibre and matrix phase. The possible reasons may be the improper curing process as well as weak interfacial bonds between the constituents phases. The figure 4.10(b) shows matrix splitting due to the brittleness of the epoxy at low temperature. The figure 4.10(c) shows matrix hardening. The figure 4.10(d) shows fibre fracture at high loading rate due to the excessive stress being applied was unable to be withstand by the fibres

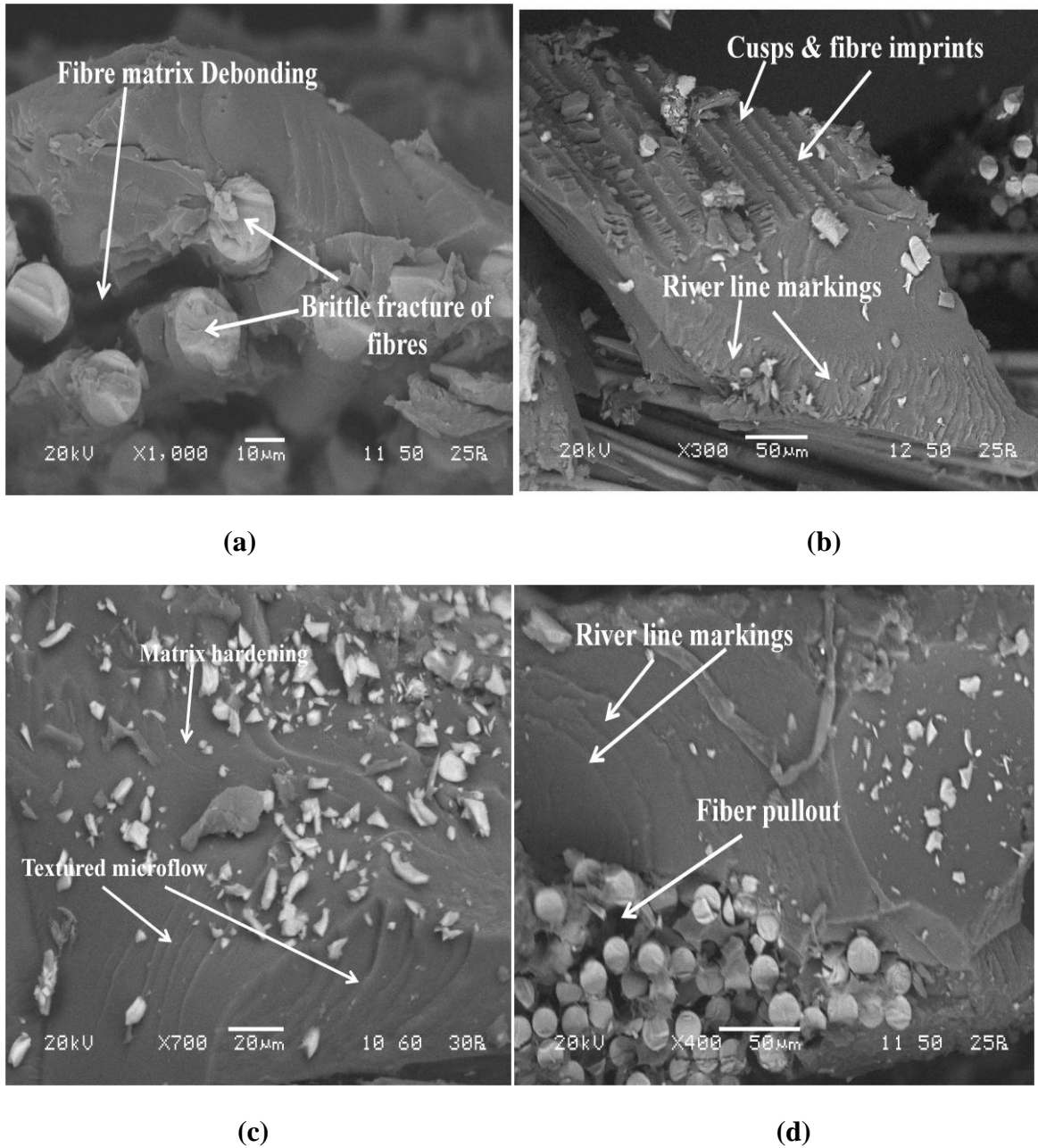
when distributed on them by the matrix due to the inability of the formation of strong interfacial bonds.



**Figure 4.10: Scanning Electron Micrographs(SEM) at -40°C at (a) 1mm/min loading rate(b) 100 mm/min loading rate (c) 1000 mm/min loading rate (d) 1000 mm/min loading rate.**

The Figure 4.11 shows scanning electron micrographs (SEM) at -60°C temperature at 1, 100 and 1000 mm/min loading rate. The figure 4.11(a) shows fibre matrix debonding and brittle fracture of fibres due to the excessive stress being applied was unable to be withstand by the fibres when distributed on them by the matrix due to the inability of the formation of strong interfacial bonds. The figure 4.11(b) cusps & fibre imprints due to development of cusps

when the local stress state in the resin rich layer between plies is such that microcrack form at an angle to the interface. Riverlines marking also forms due to the convergence of pairs of planes from the tributaries of the rivers, which converging into one crack and the direction of crack growth is the direction in which the riverlines converge. .The figure 4.11(c) shows textured microflow, which is most widespread in brittle matrix systems and in tougher materials such as epoxy, textured microflow is rare. .The figure 4.11(d) shows river lines markings and fiber pull out.



**Figure 4.11: Scanning Electron Micrographs(SEM) at -60°C at (a) 1mm/min loading rate(b) 100 mm/min loading rate (c) 100 mm/min loading rate (d) 1000 mm/min loading rate.**



### 4.1.3. Differential Scanning Calorimetry (DSC) Measurements

The measurement of glass transition temperature ( $T_g$ ) is very important because it calculates the critical service temperature of the polymer composites and finally their engineering application. Polymer composites are used at a temperature below their  $T_g$  [70]. But when they are exposed to environmental conditioning the glass transition temperature ( $T_g$ ) may be altered. The glass transition temperature ( $T_g$ ) at ambient,  $-20^\circ\text{C}$ ,  $-40^\circ\text{C}$  and  $-60^\circ\text{C}$  temperature was found to be  $71.08^\circ\text{C}$ ,  $68.98^\circ\text{C}$ ,  $68.91^\circ\text{C}$  and  $74.57^\circ\text{C}$  respectively. As compared to the ambient temperature,  $T_g$  decreases in case of  $-20^\circ\text{C}$ ,  $-40^\circ\text{C}$  temperature, but it is also evident from the DSC plots that there is no significant difference between glass transition temperature ( $T_g$ ) at  $20^\circ\text{C}$  and  $-40^\circ\text{C}$  temperature. Further at  $-60^\circ\text{C}$  temperature the rise in glass transition temperature was found about  $4.66^\circ\text{C}$ . The increase in  $T_g$  at  $-60^\circ\text{C}$  temperature can be attributed to the matrix hardening in the polymeric composite.

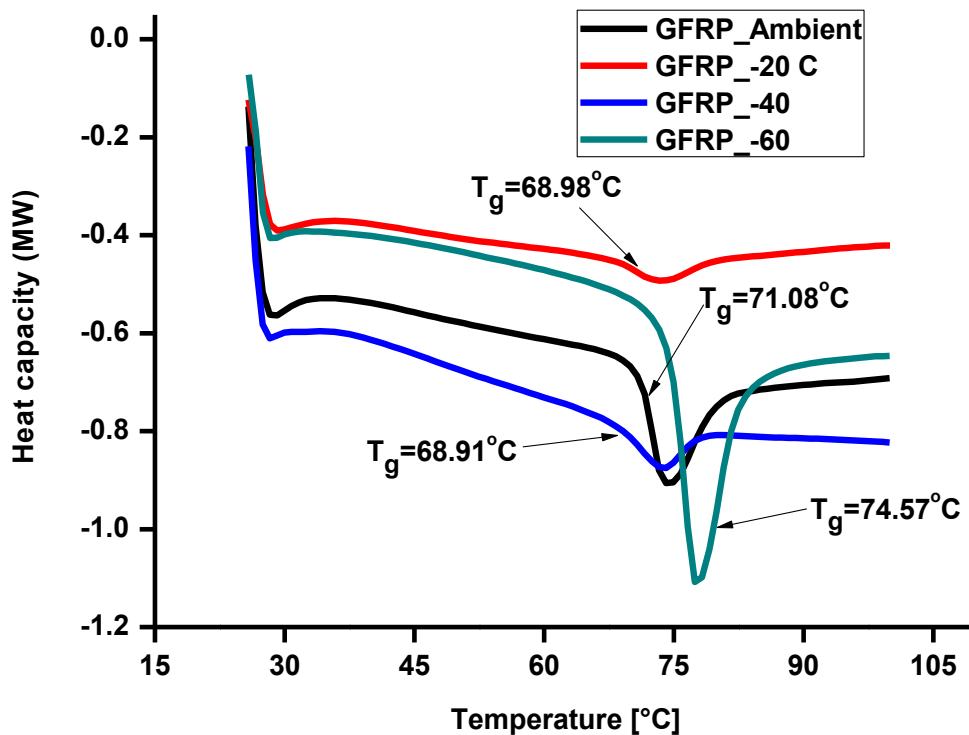
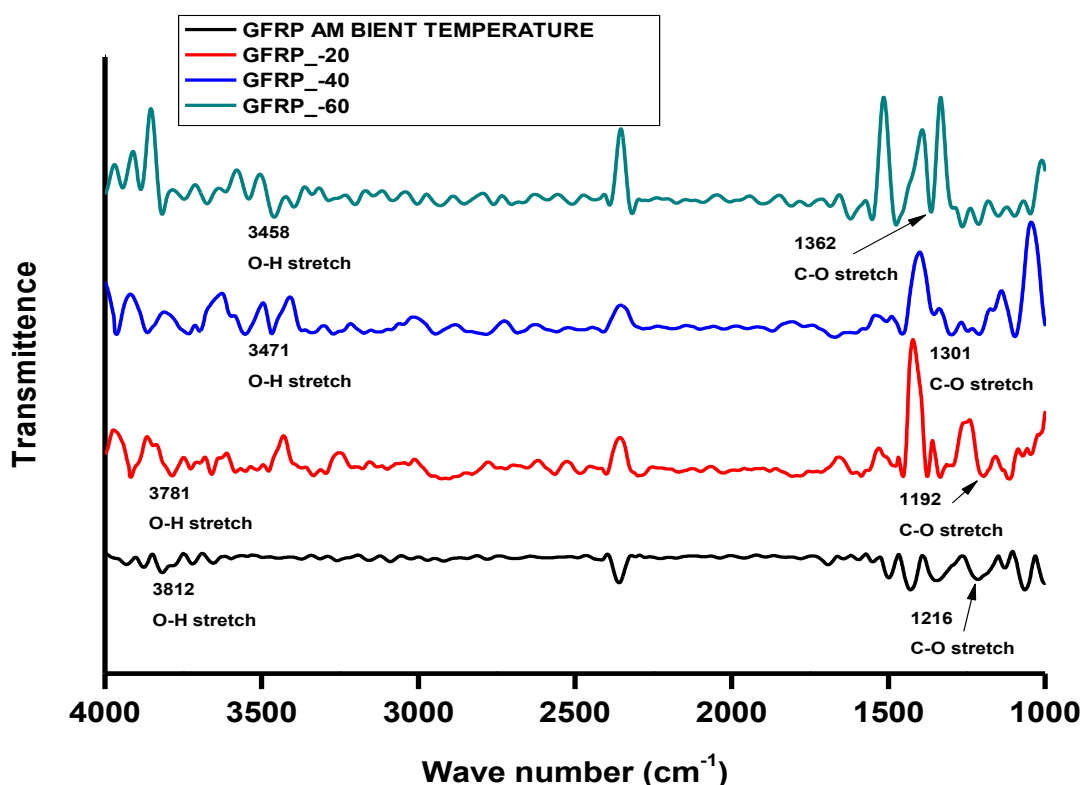


Figure 4.12: DSC plot shows  $T_g$  at ambient,  $-20^\circ\text{C}$ ,  $-40^\circ\text{C}$  and  $-60^\circ\text{C}$  temperatures of GFRP samples.

#### 4.1.4. Fourier Transform Infrared Spectroscopy (FTIR-ATR) Analysis

FTIR-Imaging experiments were performed in this study to analysis the low temperature treated GFRP composite as IR measurements are very sensitive to hydrogen bonds.

Fourier transform infrared spectroscopy (FTIR) is an extensively used method that for many years and measures wavelength ranging from  $4000\text{cm}^{-1}$  to  $1000\text{cm}^{-1}$  and results from vibrational plus rotational transitions. This region is particularly meant for organic chemists since the vibrations induced in organic molecules are absorbed in this region.



**Figure 4.13: FTIR plot of GFRP samples at ambient, -20 °C, -40°C and -60°C temperature.**

The figure 4.13 represents FTIR spectra of ambient, -20 °C, -40°C and -60°C of GFRP samples. At ambient temperature O-H bond and C-O bond were found at  $3812\text{cm}^{-1}$  and  $1216\text{cm}^{-1}$  wavelength respectively. At -20°C temperature O-H bond and C-O bond were found at  $3781\text{cm}^{-1}$  and  $1192\text{cm}^{-1}$  wavelength respectively. At -40°C temperature O-H bond and C-O bond were found at  $3471\text{cm}^{-1}$  and  $1301\text{cm}^{-1}$  wavelength respectively. Similarly, at -60°C temperature O-H bond and C-O bond were found at  $3458\text{cm}^{-1}$  and  $1362\text{cm}^{-1}$  wavelength respectively.

## 4.2. Effect of thermal spike conditioning on mechanical behavior of fibre reinforced polymer (FRP) composites.

### 4.2.1. Glass fibre reinforced polymer (GFRP) Composites

#### 4.2.1.1. Short beam shear (SBS) test

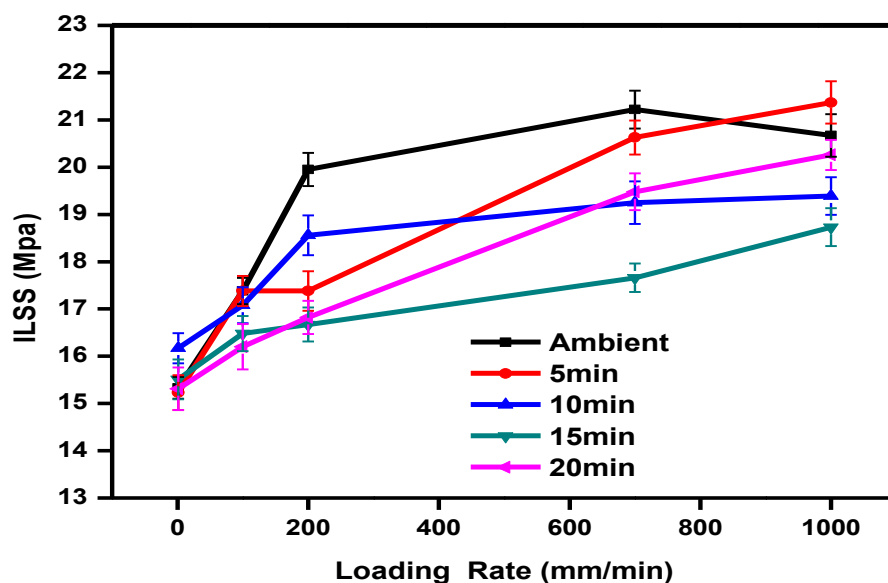
The interlaminar shear strength (ILSS) of low temperature conditioning samples during short beam shear test can be calculated as  $ILSS = 0.75P/bd$

Where, P is maximum load, b is the width of specimen and d is the thickness of specimen.

#### 4.2.1.1.1. Effect of loading rate on the interlaminar shear strength (ILSS) of glass/epoxy composite

The tests were carried out at ambient temperature, 50°C, 100°C, 150°C and 200°C temperature with holding time 5, 10, 15 and 20 minutes at 1, 100, 200, 700, 1000 mm/min loading rates.

The figure 4.14 reveals that higher ILSS is obtained at ambient temperature at 200 mm/min loading rate. The lower ILSS is obtained at 50°C with 20 minutes holding time at 1mm/min loading rate.



**Figure 4.14: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 50°C for a holding time of 5, 10, 15, 20 minutes respectively.**

Due to the exposure of thermal spike conditionings there is variation in ILSS of the glass fibre/epoxy composites which is evident from the figure 4.14. The variation in ILSS can be attributed to the strain softening of the matrix in the composite. Further the mechanical

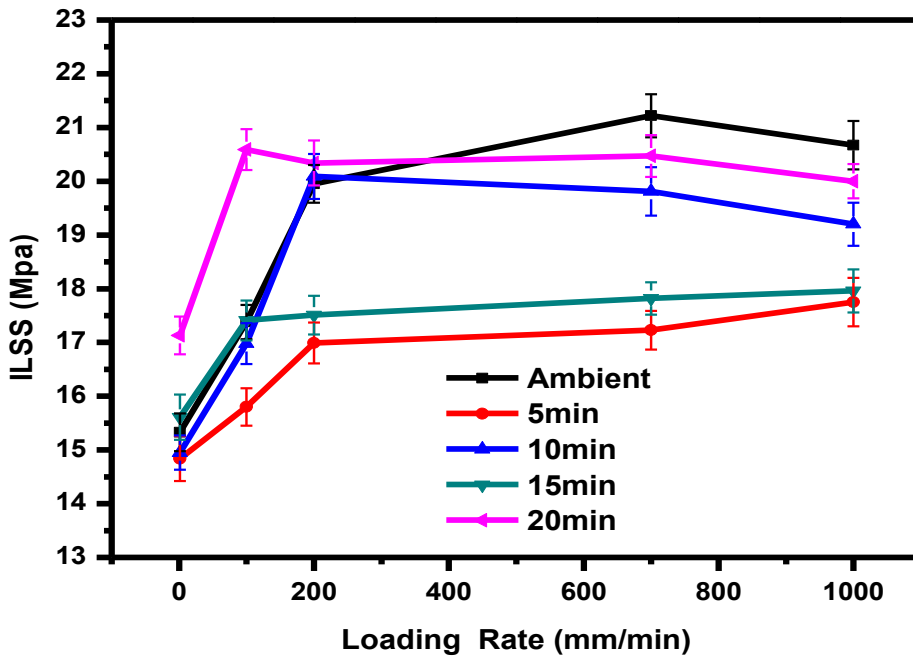
behaviour of composite at low loading rate is primarily governed by polymer matrix and/or fibre/polymer interface, whereas at higher loading rate reinforcing fibres play dominant role in determining composite strength.

Environmental exposure	% change in ILSS between 1mm/min-200mm/min	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+30.13 %	+3.6 %
Thermal spike at +50°C for 5 min	+14.11 %	+22.95 %
Thermal spike at +50°C for 10 min	+14.78 %	+4.47 %
Thermal spike at +50°C for 15 min	+7.4 %	+12.35 %
Thermal spike at +50°C for 20 min	+9.8 %	+20.45 %

**Table 4.1: % change in ILSS values for GFRP composites exposed to thermal conditioning at +50°C temperature for different times and tested at different loading rates.**

The figure 4.15 reveals that higher ILSS is obtained at ambient temperature at 200 mm/min loading rate. The lower ILSS is obtained at 100°C with 5 minutes holding time at 1mm/min loading rate.

From figure 4.15 it is clearly evident that the short exposure (5, 10, 15minutes) to thermal spike conditioning results in degradation in ILSS as compared to ambient environment in the given range of loading rate. But it is very interesting to note that initially for 20min exposure the ILSS is higher than the ILSS at ambient temperature and this increase in ILSS can be attributed to the further cross-linking in the polymer matrix which also overcomes the weakening effects of differential thermal expansion of fibre and polymer matrix.

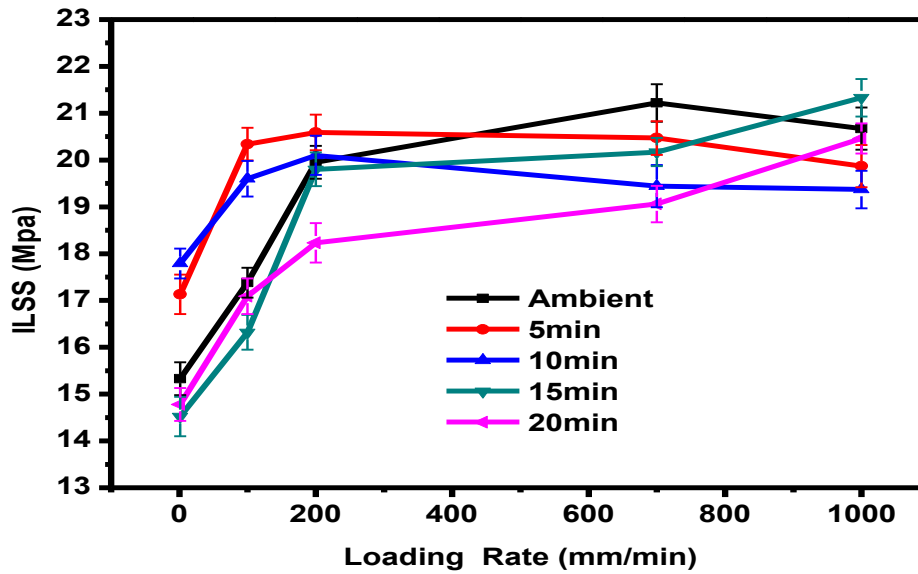


**Figure 4.15: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 100°C for a holding time of 5, 10, 15, 20 minutes respectively.**

Environmental exposure	% change in ILSS between 1mm/min-200mm/min	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+30.13 %	+3.6 %
Thermal spike at +100°C for 5 min	+14.48 %	+4.47 %
Thermal spike at +100°C for 10 min	+34.38 %	-4.43 %
Thermal spike at +100°C for 15 min	+12.17 %	+2.56 %
Thermal spike at +100°C for 20 min	+18.73 %	-1.6 %

**Table 4.2: % change in ILSS values for GFRP composites exposed to thermal spike conditioning at +100°C temperature for different times and tested at different loading rates.**

The figure 4.16 reveals that higher ILSS is obtained at 150°C temperature with 5 minutes holding time at 1000 mm/min loading rate. The lower ILSS is obtained at 150°C temperature with 15 minutes holding time at 1mm/min loading rate



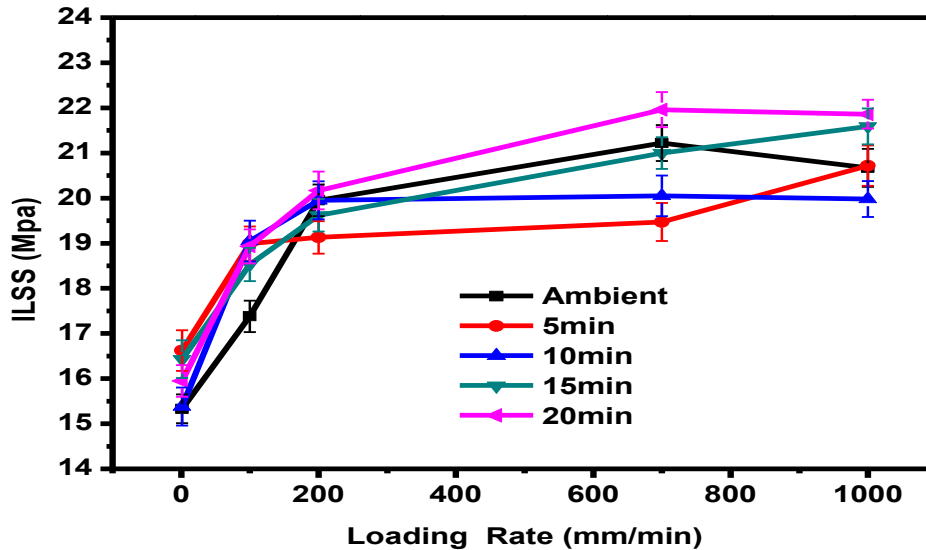
**Figure 4.16: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 150°C for a holding time of 5, 10, 15, 20 minutes respectively.**

Environmental exposure	% change in ILSS between 1mm/min-200mm/min	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+30.13 %	+3.6 %
Thermal spike at +150°C for 5 min	+20.19 %	-3.5 %
Thermal spike at +150°C for 10 min	+12.98 %	-3.63 %
Thermal spike at +150°C for 15 min	+36.36 %	+ 7.72%
Thermal spike at +150°C for 20 min	+ 23.34%	+12.23 %

**Table 4.3: % change in ILSS values for GFRP composites exposed to thermal spike conditioning at +150°C temperature for different times and tested at different loading rates.**

Exposure to 150°C and 200°C thermal spike conditioning on glass fibre/epoxy composites leads to no significant degradation in ILSS of the composite (shown in figure 4.16 and 4.17) which can be attributed to the recovery of bonds at fibre/polymer interface during cooling

from glass transition temperature to ambient temperature. Table: % change in ILSS values for GFRP composites exposed to thermal spike conditioning at +200°C temperature for different times and tested at different loading rates.



**Figure 4.17: Comparison of ILSS Vs loading rate of thermal spiking conditioning of GFRP samples at 200°C for a holding time of 5, 10, 15, 20 minutes respectively.**

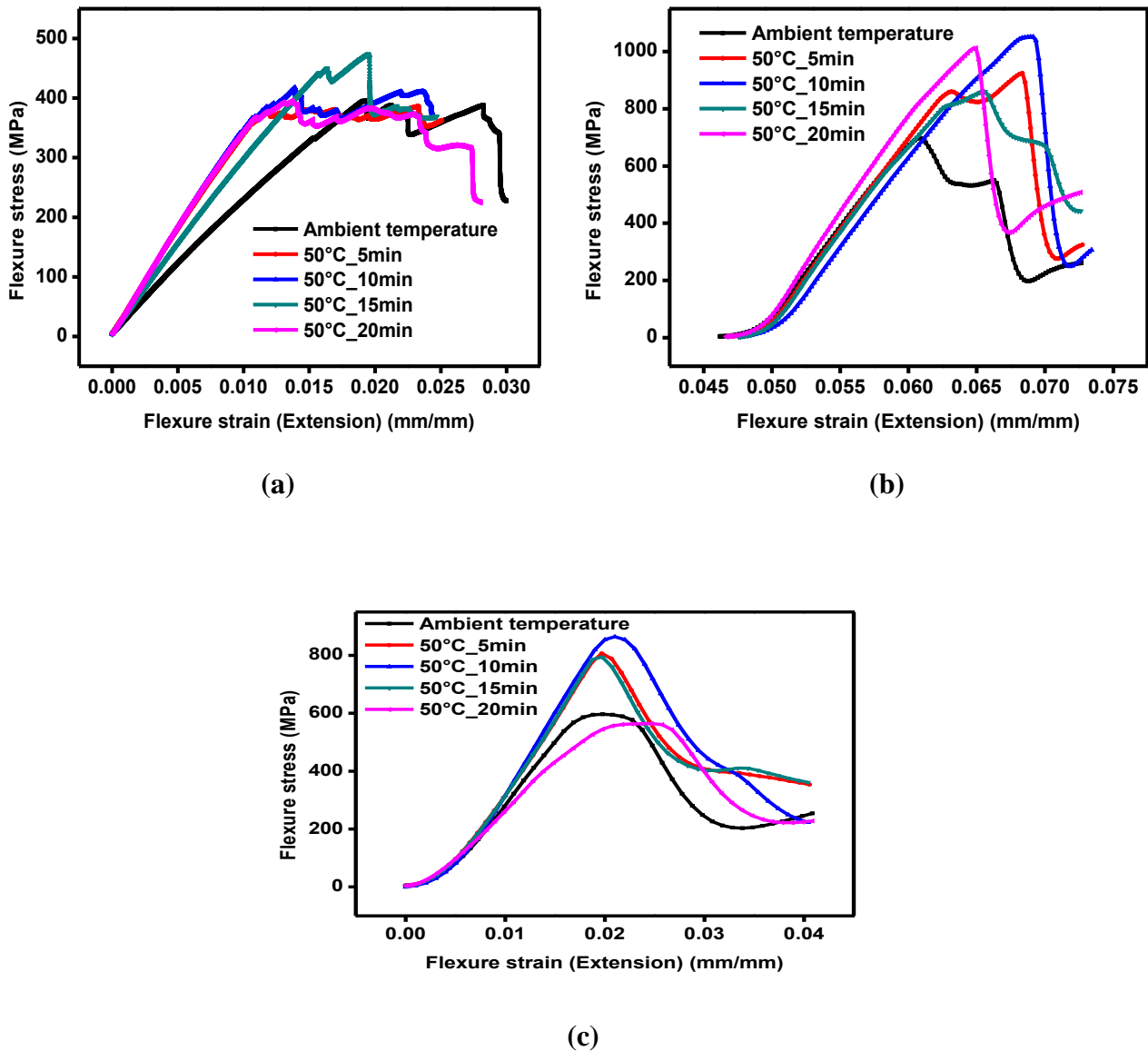
The figure 4.17 reveals that higher ILSS is obtained at 200°C temperature with 10 minutes holding time at 200 mm/min loading rate. The lower ILSS is obtained at ambient temperature at 1mm/min loading rate.

Environmental exposure	% change in ILSS between 1mm/min-200mm/min	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+30.13 %	+3.6 %
Thermal spike at +200°C for 5 min	+15.10 %	+8.31 %
Thermal spike at +200°C for 10 min	+29.71 %	+0.15%
Thermal spike at +200°C for 15 min	+19.41 %	+10.04 %
Thermal spike at +200°C for 20 min	+ 26.45 %	+8.37 %

**Table 4.4: % change in ILSS values for GFRP composites exposed to thermal spike conditioning at +200°C temperature for different times and tested at different loading rates.**

#### 4.2.1.1.2. Stress- strain behavior of GFRP composites

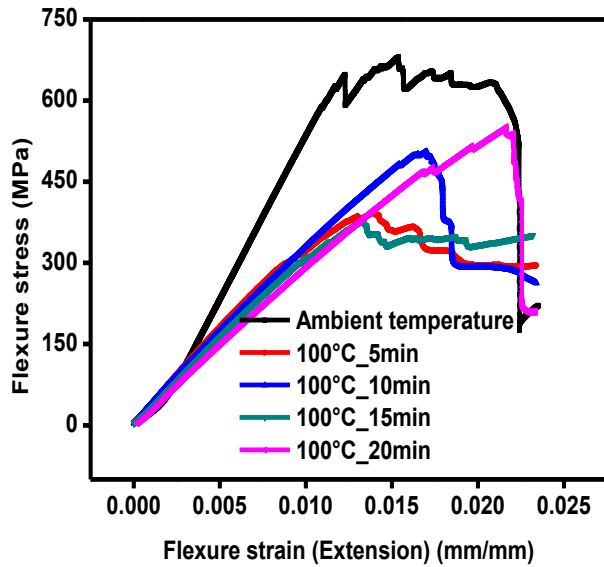
The figure 4.18(a) shows that maximum stress at 1mm/min is obtained at 50°C temperature with 15 minutes holding time. The figure 4.18(b) and 4.18(c) shows that maximum stress at 200 mm/min and 1000 mm/min is obtained at 50°C temperature with 10 minutes holding time respectively.



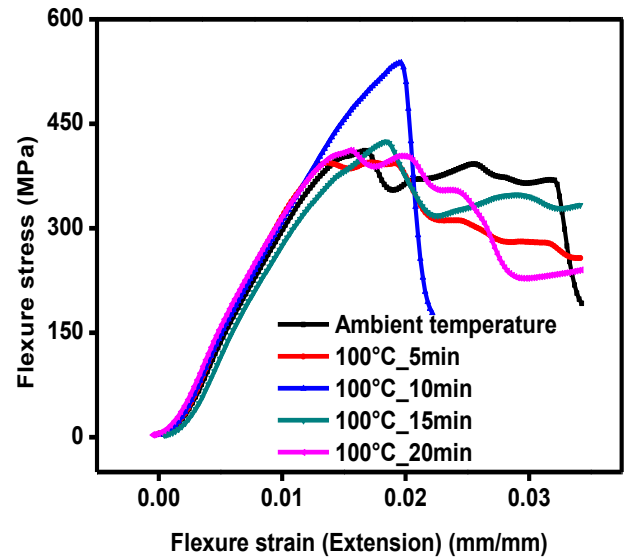
**Figure 4.18: Flexure stress Vs Flexure strain plot at 50°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**



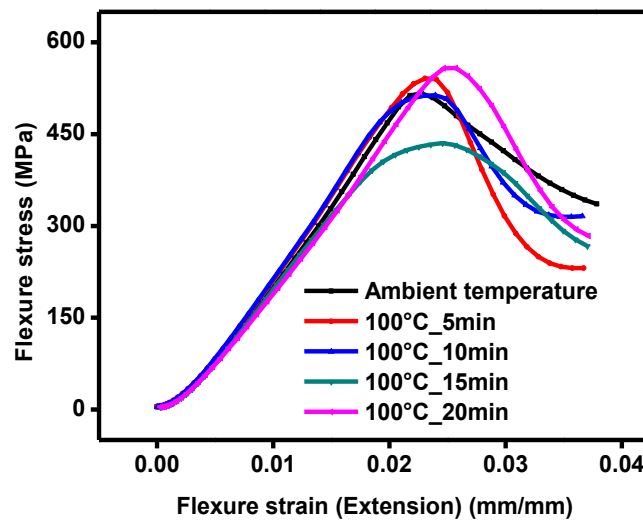
The figure 4.19(a) shows that maximum stress at 1mm/min is obtained at ambient temperature. The figure 4.19(b) reveal maximum stress at 200 mm/min is obtained at 100°C with 15 minutes holding time. The figure 4.19(c) shows maximum stress at 1000 mm/min is obtained at 100°C with 20 minutes holding time.



(a)



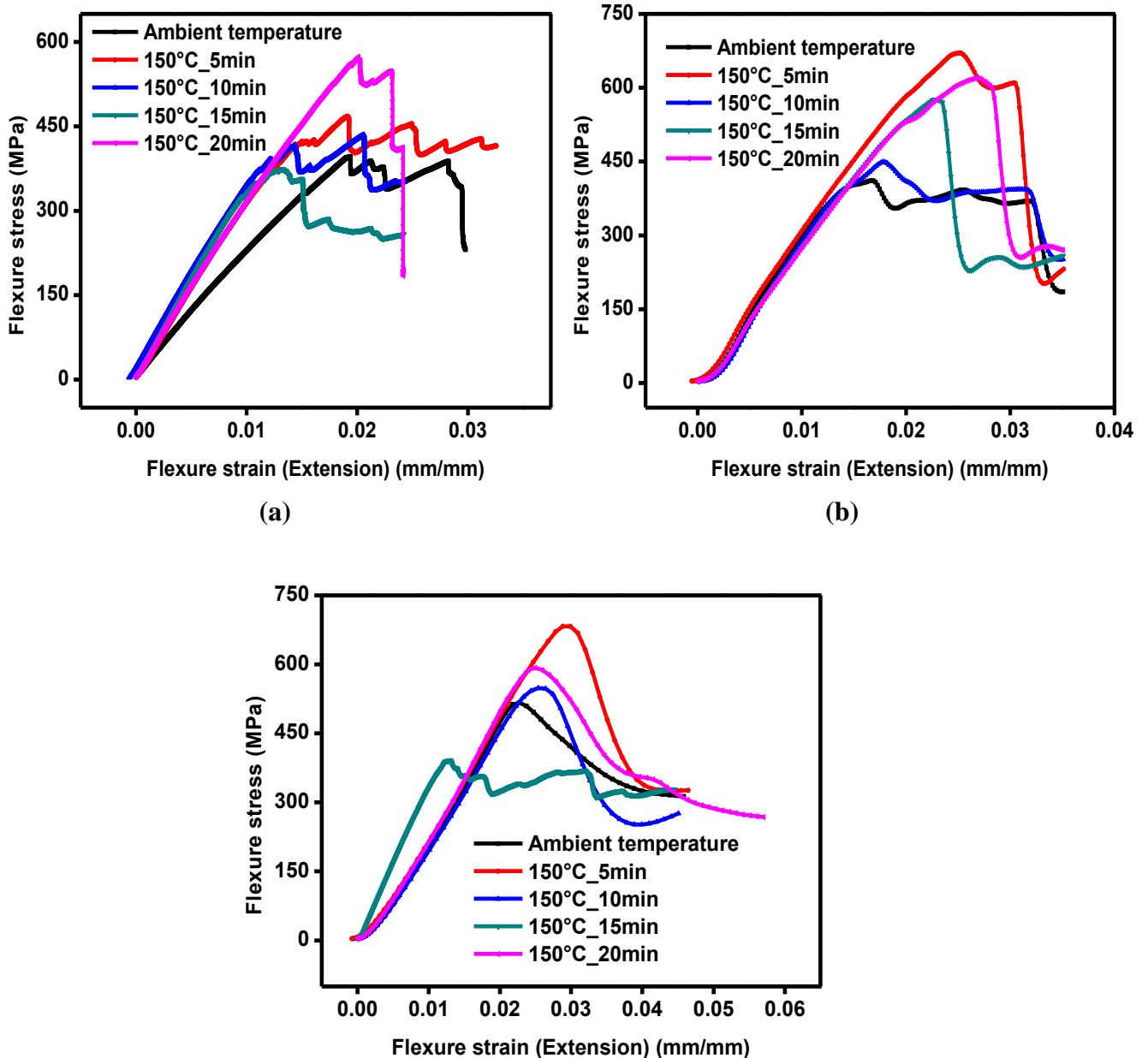
(b)



(c)

**Figure 4.19: Flexure stress Vs Flexure strain plot at 100°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**

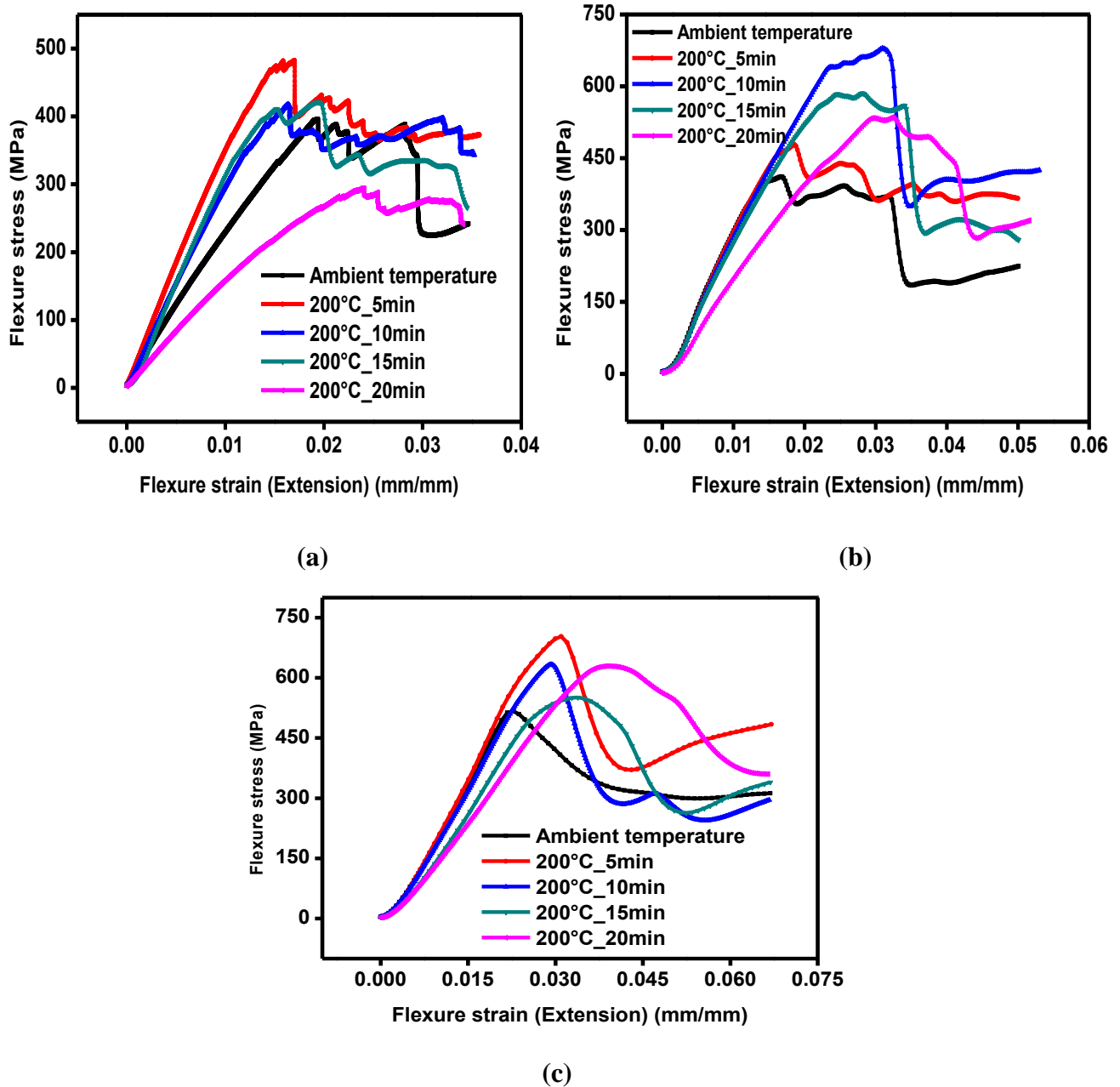
The figure 4.20(a) shows that maximum stress at 1mm/min is obtained at 150°C temperature with 20 minutes holding time. The figure 4.20(b) reveal maximum stress at 200 mm/min is obtained at 150°C with 5 minutes holding time. The figure 4.20(c) shows maximum stress at 1000 mm/min is obtained at 150°C with 5 minutes holding time.



(c)

**Figure 4.20: Flexure stress Vs Flexure strain plot at 150°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**

The figure 4.21(a) shows that maximum stress at 1mm/min is obtained at 200°C temperature with 5 minutes holding time. The figure 4.21(b) reveal maximum stress at 200 mm/min is obtained at 200°C with 10 minutes holding time. The figure 4.21(c) shows maximum stress at 1000 mm/min is obtained at 200°C with 5 minutes holding time.



**Figure 4.21: Flexure stress Vs Flexure strain plot at 200°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**

#### 4.2.1.2. Differential Scanning Calorimetry (DSC) Measurements

The glass fibre reinforced polymer (GFRP) composite when exposed to thermal conditioning the glass transition temperature ( $T_g$ ) may be altered. The glass transition temperature ( $T_g$ ) decreases as the holding time of the thermal conditioning samples increases. This can be attributed to plasticization of the polymer matrix. Glass transition temperature ( $T_g$ ) decreases for low temperature spiking i.e. at 50°C with holding time 5, 10, 15 and 20 minutes which may be due to debonding. But it increases in case of high temperature thermal spiking i.e. at 100°C, 150°C and 200°C with holding time 5, 10, 15 and 20 minutes due to matrix softening.

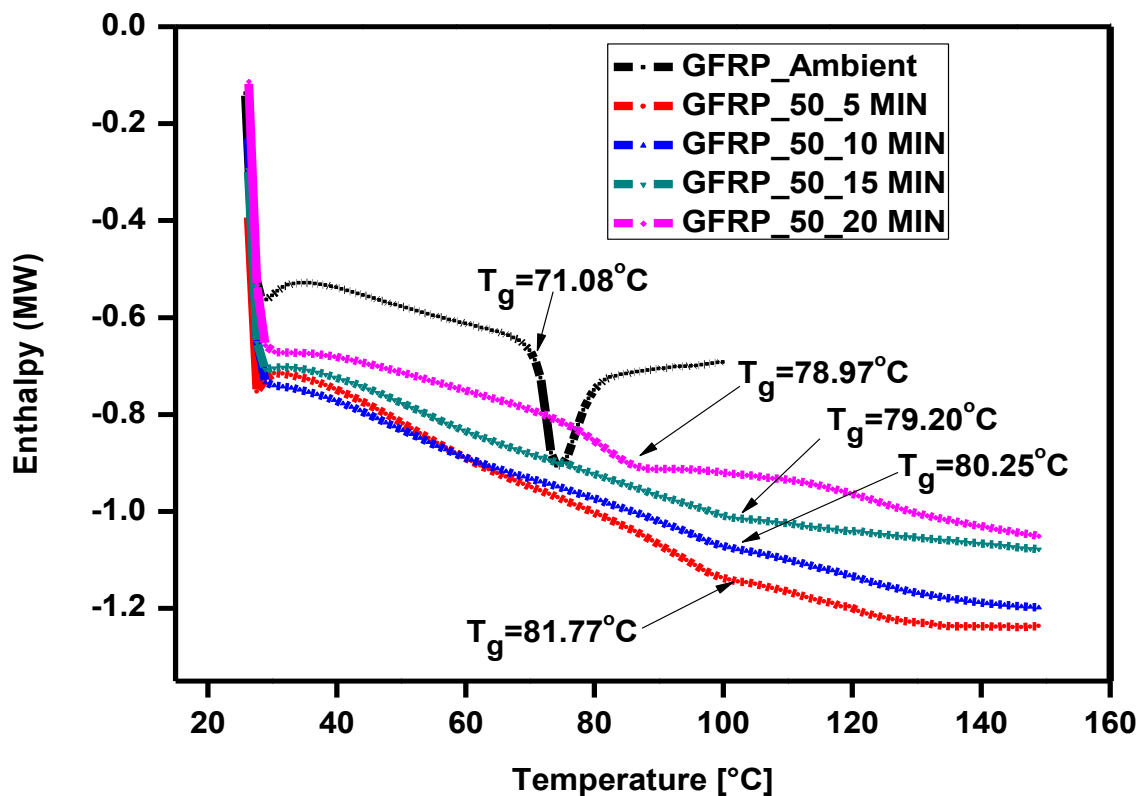


Figure 4.22: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 50°C for a holding time of 5, 10, 15 and 20 minutes.

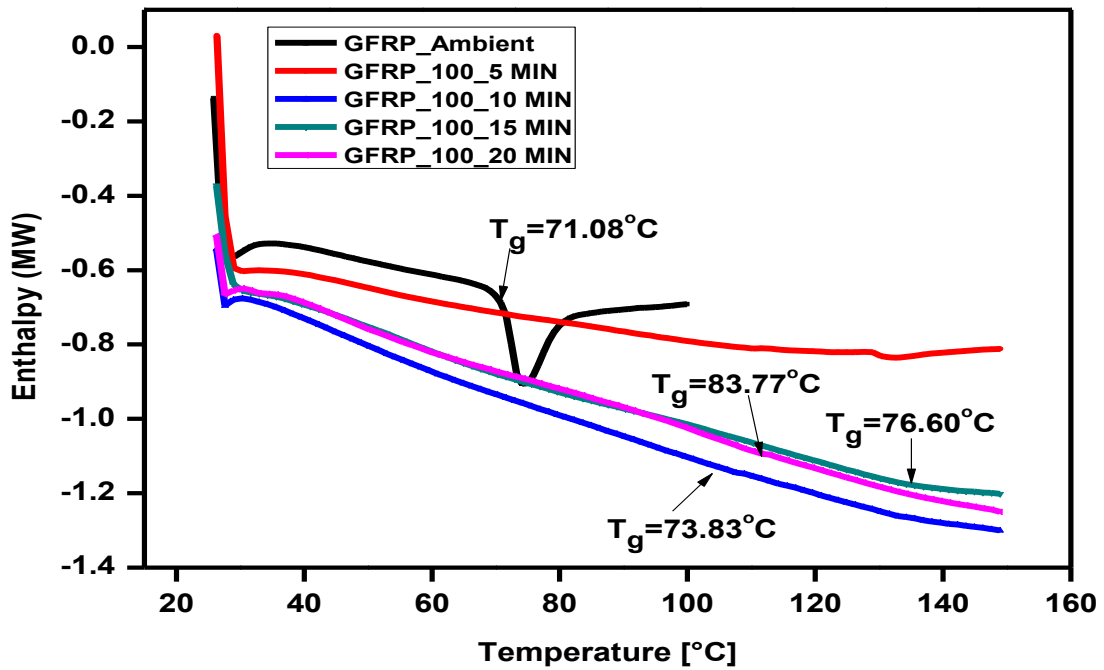


Figure 4.23: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 100°C for a holding time of 5, 10, 15 and 20 minutes.

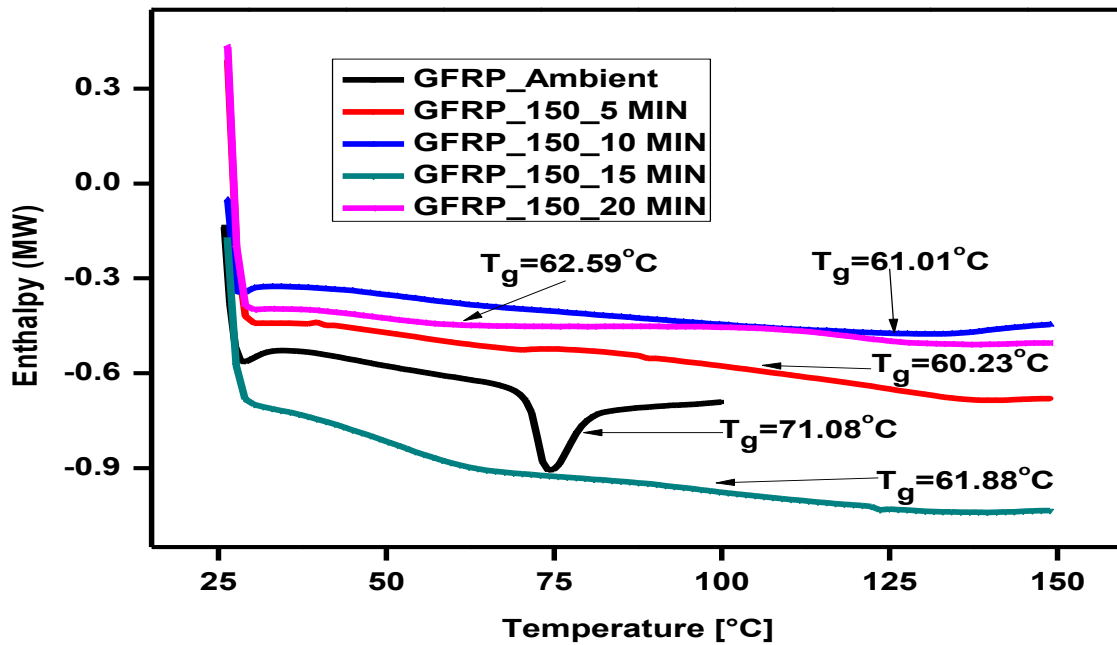


Figure 4.24: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 150°C for a holding time of 5, 10, 15 and 20 minutes.

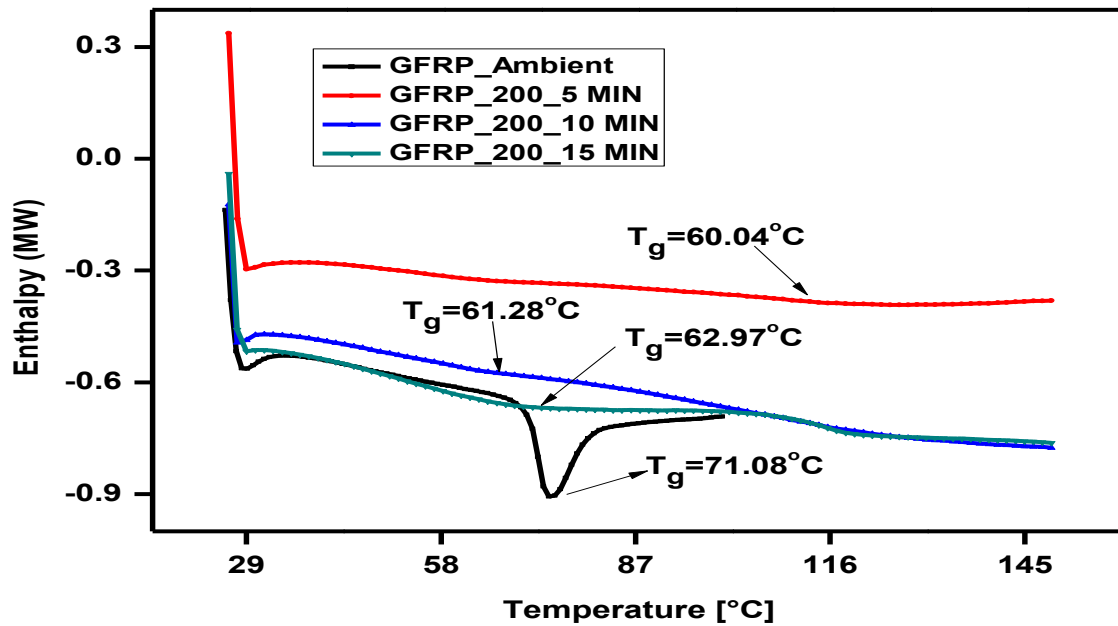
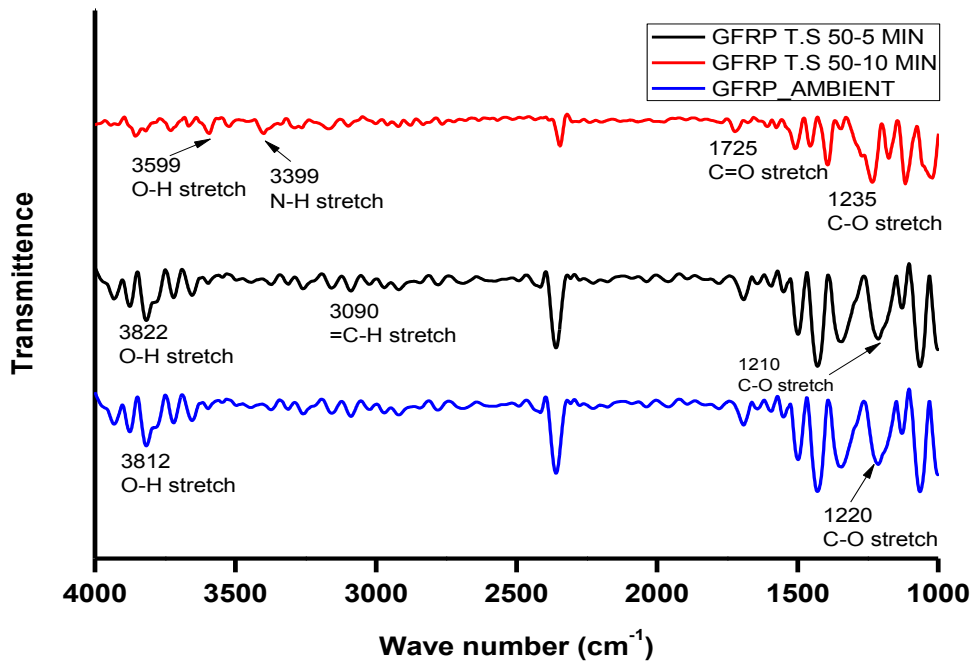


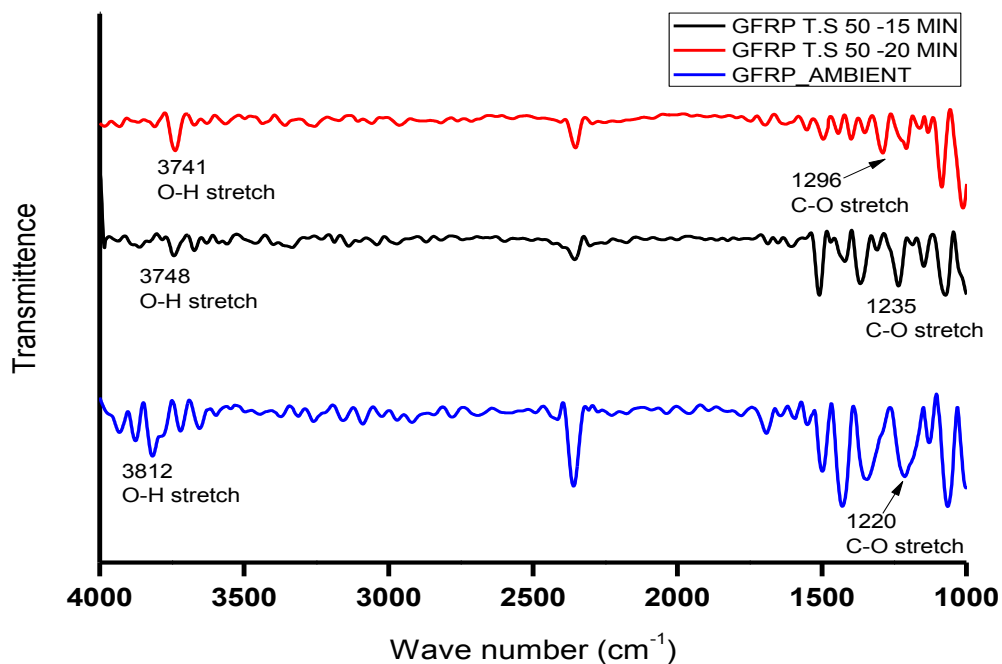
Figure 4.25: DSC plot of thermal spike conditioned GFRP samples at ambient temperature and 200°C for a holding time of 5, 10, 15 and 20 minutes.

#### 4.2.1.3. Fourier Transformation Infrared Spectroscopy (FTIR-ATR) Analysis

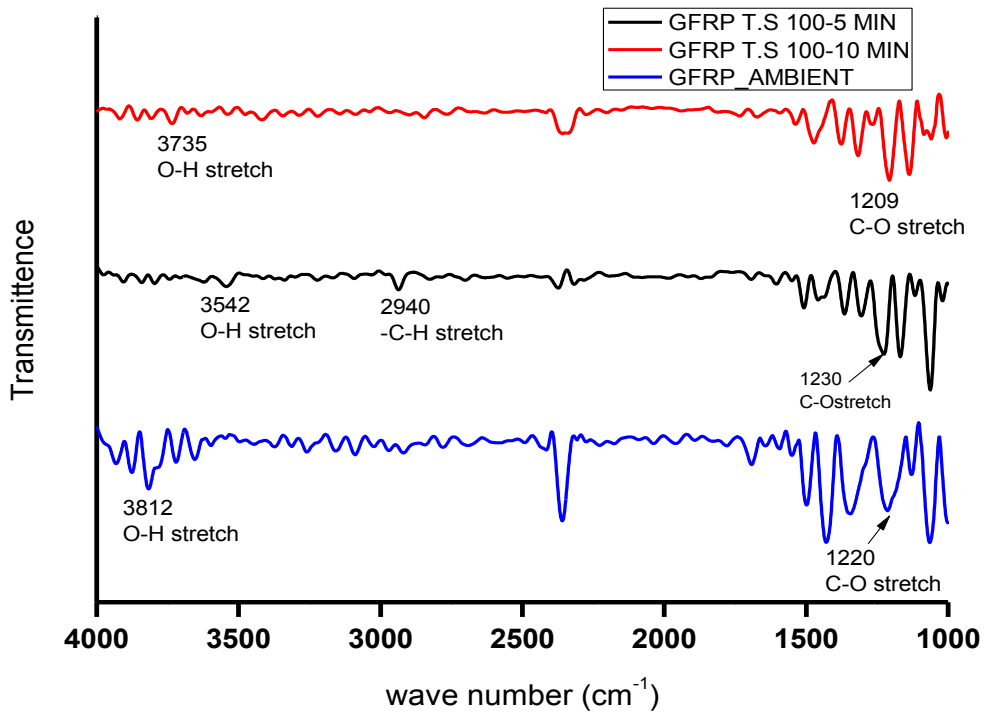
FTIR-Imaging experiments were performed in this study to analysis the thermal spiking conditioning of CFRP composite as IR measurements are very sensitive to hydrogen bonds. The Figure 4.45 to 4.50 shows FTIR spectra of CFRP samples at ambient and 50°C, 100°C, 150°C and 200°C temperature for a holding time of 5, 10, 15 and 20 minutes respectively. Several bond formations occur in between  $4000\text{cm}^{-1}$  to  $1000\text{cm}^{-1}$  wavelength range such as O-H bond, N-H bond, =C-H bond, C=O bond and C-O bond. The O-H bond leads to very complicated spectrum by both intermolecular and intramolecular hydrogen bonding. Any spectral changes are due to the molecular changes in the composites [18]. The fundamental stretching vibration and the in-plane bending vibration occur within the  $3900\text{-}2800\text{cm}^{-1}$  region.



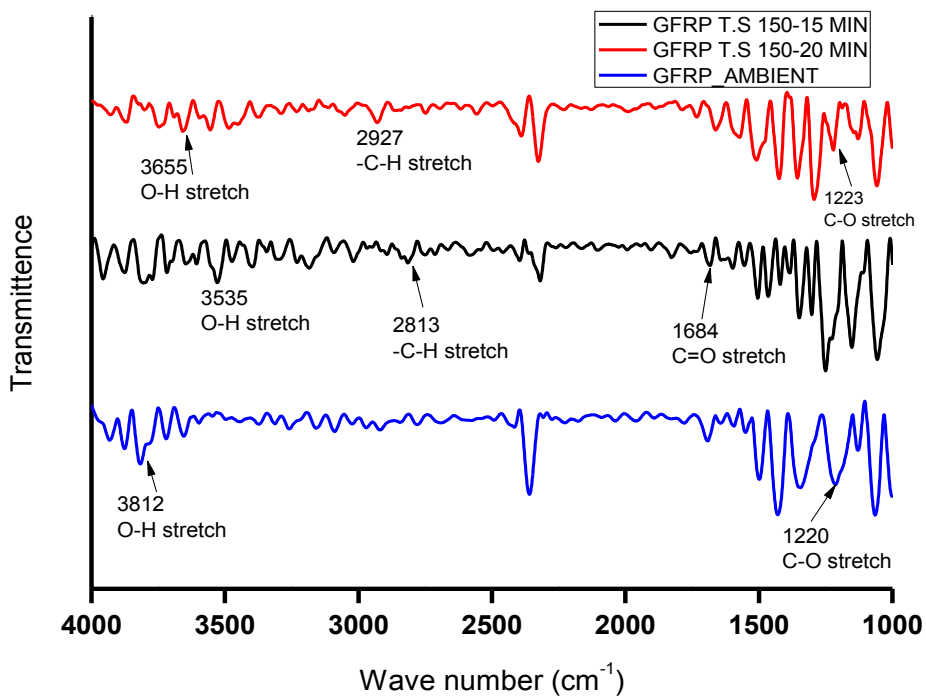
**Figure 4.26: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 50°C for a holding time of 5 and 10 minutes.**



**Figure 4.27: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 50°C for a holding time of 15 and 20 minutes**

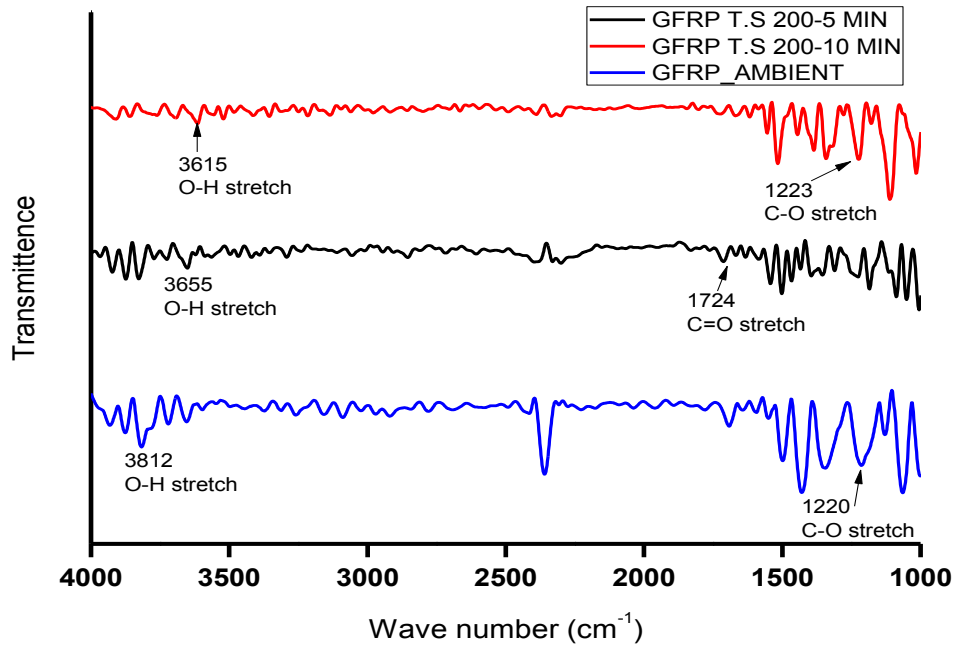


**Figure 4.28: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 100°C for a holding time of 5 and 10 minutes.**

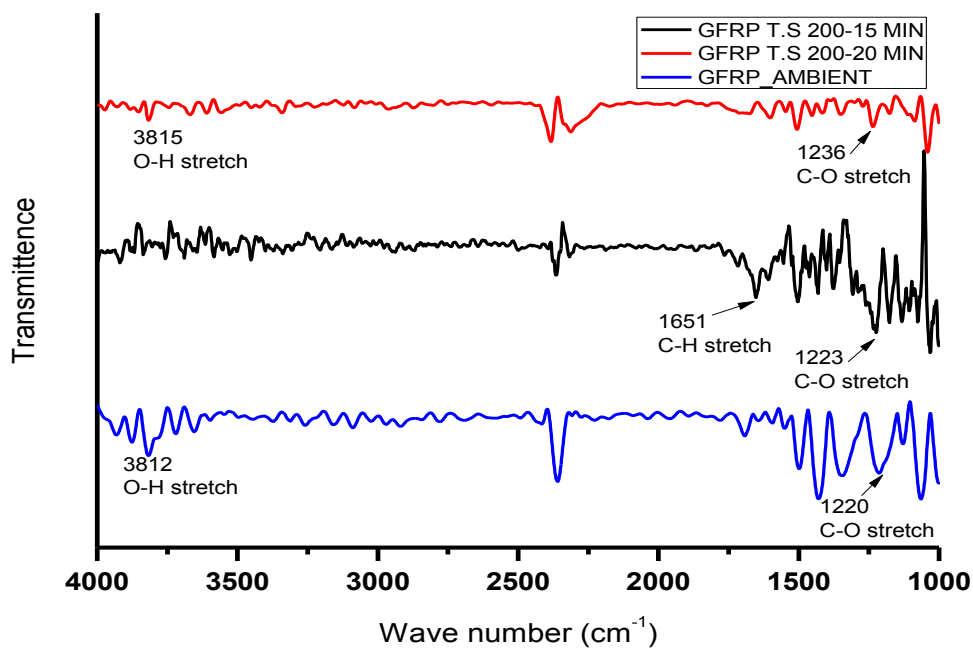


**Figure 4.29: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 150°C for a holding time of 15 and 20 minutes.**





**Figure 4.30: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 200°C for a holding time of 5 and 10 minutes.**



**Figure 4.31: FTIR-ATR plot of thermal spike conditioned GFRP samples at ambient temperature and 200°C for a holding time of 15 and 20 minutes.**

## 4.2.2. Carbon fibre reinforced polymer (CFRP) Composites

### 4.2.2.1. Short beam shear (SBS) test

The interlaminar shear strength (ILSS) of low temperature conditioning samples during short beam shear test can be calculated as  $ILSS = 0.75P/bd$

Where, P is maximum load, b is the width of specimen and d is the thickness of specimen.

#### 4.2.2.1.1. Effect of loading rate on the interlaminar shear strength (ILSS) of carbon/epoxy composite

The tests were carried out at ambient temperature, 50°C, 100°C, 150°C and 200°C temperature with holding time 5, 10, 15 and 20 minutes at 1, 100, 200, 700, 1000 mm/min loading rates.

The figure 4.32 reveals that higher ILSS is obtained at 50°C with 10 min holding time at 200 mm/min loading rate. The lower ILSS is obtained at ambient temperature at 1mm/min loading rate.

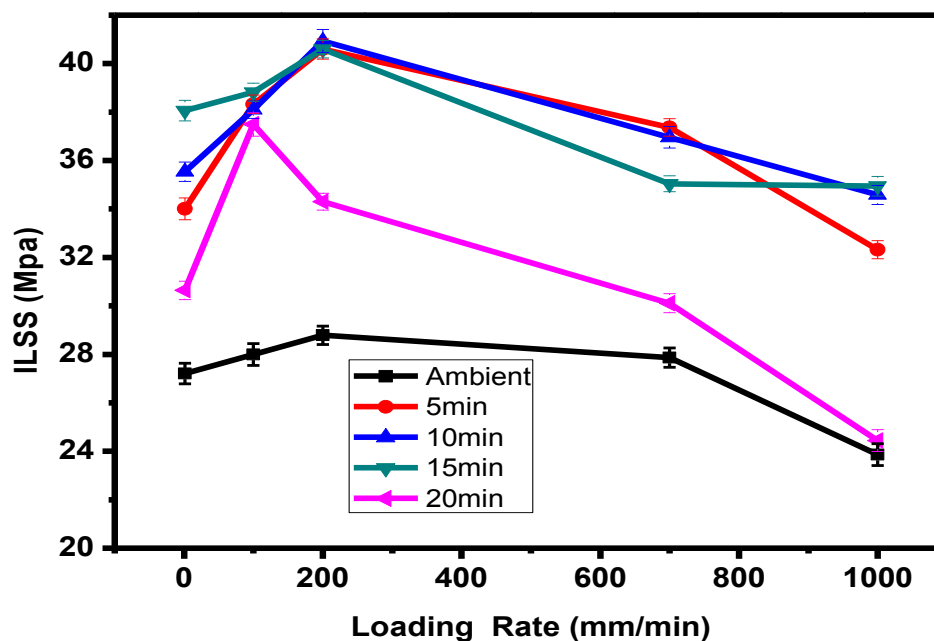
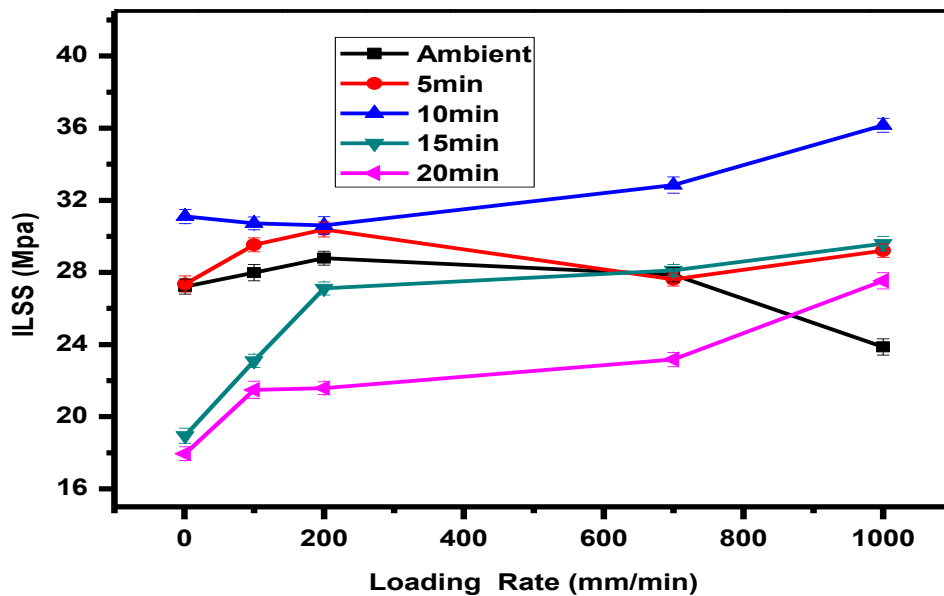


Figure 4.32: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 50°C for a holding time of 5, 10, 15, 20 minutes respectively.

Environmental exposure	% change in ILSS between 1mm/min-200mm/min	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+ 5.8 %	-17.12 %
Thermal spike at +50°C for 5 min	+ 19.37 %	-25.61 %
Thermal spike at +50°C for 10 min	+ 14.99 %	15.51 %
Thermal spike at +50°C for 15 min	+6.67 %	-13.94 %
Thermal spike at +50°C for 20 min	+11.94 %	-28.74 %

**Table 4.5: % change in ILSS values for CFRP composites exposed to thermal conditioning at +50°C temperature for different times and tested at different loading rates.**

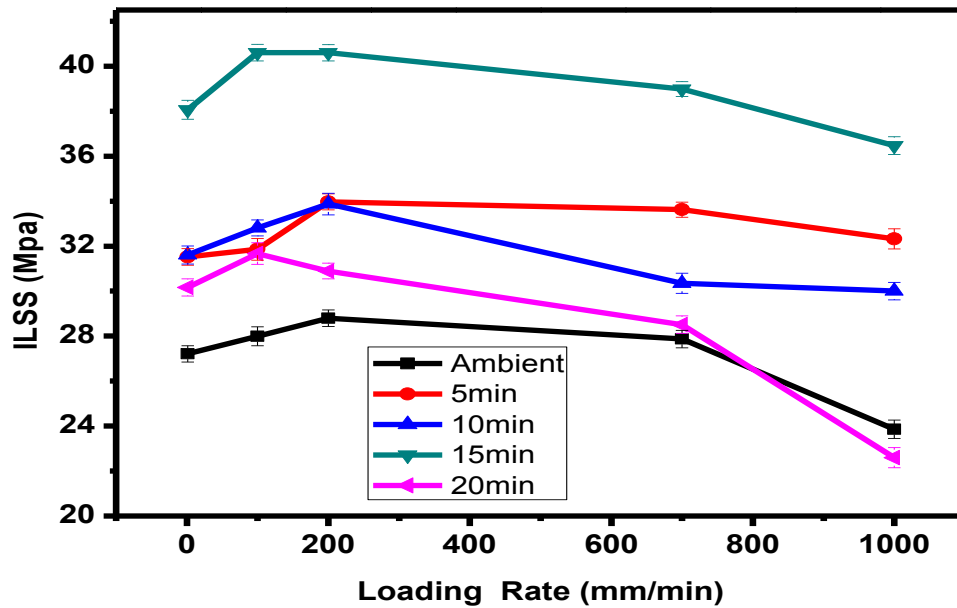
The figure 4.33 reveals that higher ILSS is obtained at 100°C with 10 minutes holding time at 1000 mm/min loading rate. The lower ILSS is obtained at 100°C with 20 minutes holding time at 1mm/min loading rate.



**Figure 4.33: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 100°C for a holding time of 5, 10, 15, 20 minutes respectively.**

Environmental exposure	% change in ILSS between 1mm/min-	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+ 5.8 %	-17.12 %
Thermal spike at +100°C for 5 min	+ 12.20%	-9.84 %
Thermal spike at +100°C for 10 min	-1.57 %	+18.13%
Thermal spike at +100°C for 15 min	+ 43.13%	+9.22 %
Thermal spike at +100°C for 20 min	+ 20.22%	+27.57 %

**Table 4.6: % change in ILSS values for CFRP composites exposed to thermal spike conditioning at +100°C temperature for different times and tested at different loading rates 200mm/min**

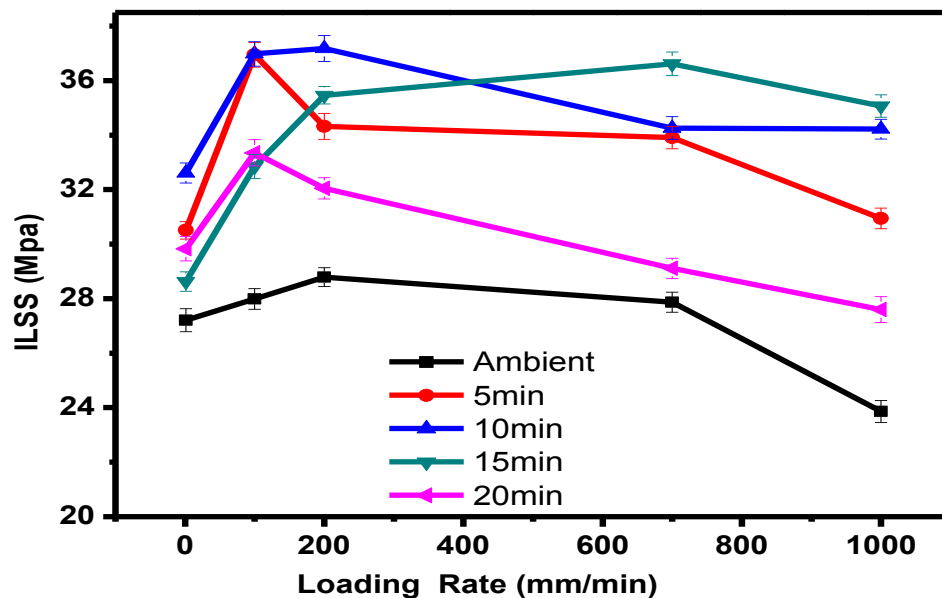


**Figure 4.34: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 150°C for a holding time of 5, 10, 15, 20 minutes respectively.**

The figure 4.34 reveals that higher ILSS is obtained at 150°C with 15 minutes holding time at 1 mm/min loading rate. The lower ILSS is obtained at ambient temperature at 1000 mm/min loading rate.

Environmental exposure	% change in ILSS between 1mm/min-200mm/min	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+ 5.8 %	-17.12 %
Thermal spike at +150°C for 5 min	+7.77 %	- 4.85%
Thermal spike at +150°C for 10 min	+ 7.14%	- 11.42%
Thermal spike at +150°C for 15 min	+6.67 %	-10.17 %
Thermal spike at +150°C for 20 min	+2.42 %	-26.86 %

**Table 4.7: % change in ILSS values for CFRP composites exposed to thermal spike conditioning at +150°C temperature for different times and tested at different loading rates.**



**Figure 4.35: Comparison of ILSS Vs loading rate of thermal spiking conditioning of CFRP samples at 200°C for a holding time of 5, 10, 15, 20 minutes respectively.**

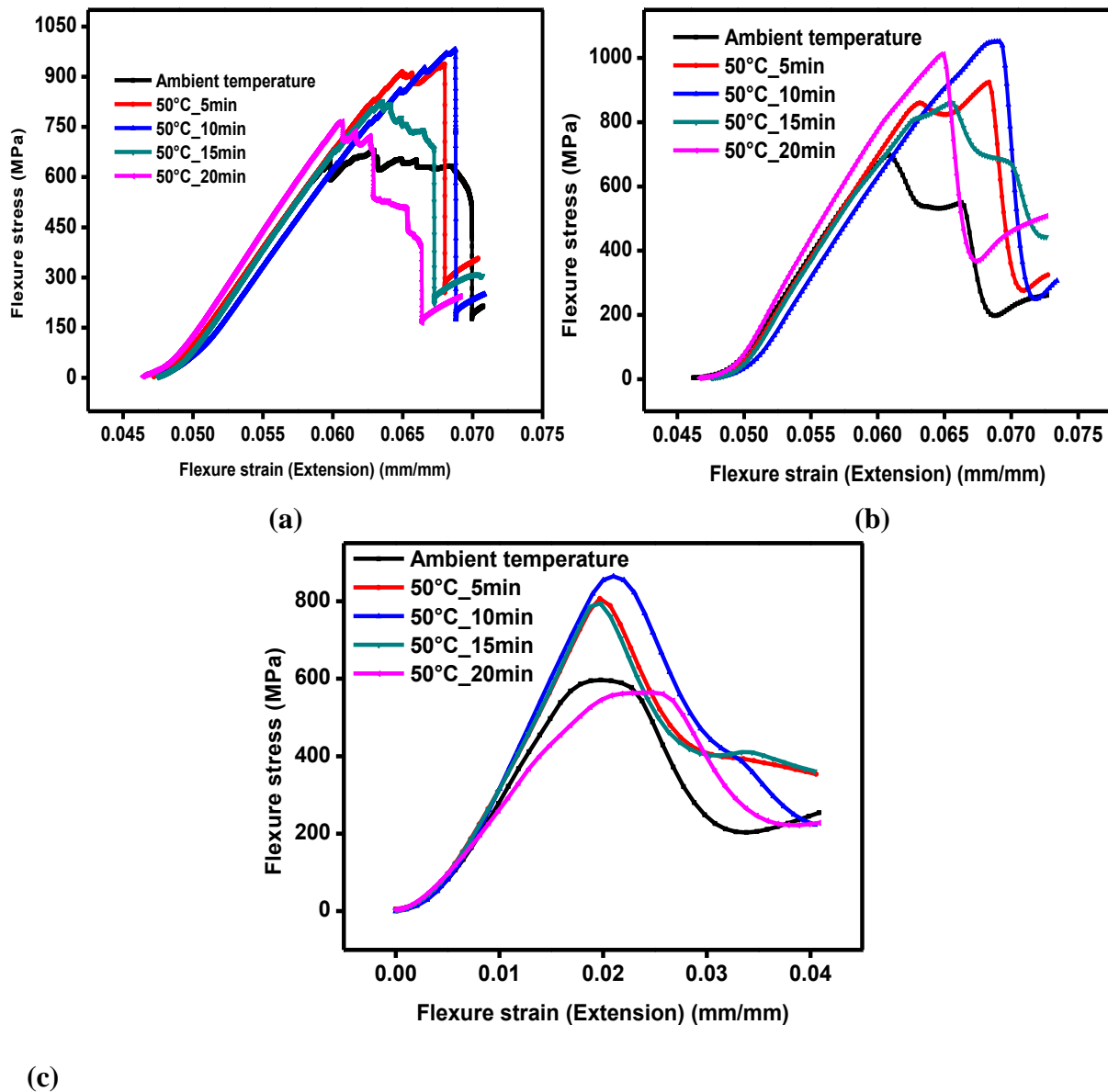
The figure 4.35 reveals that higher ILSS is obtained at 200°C with 10 minutes holding time at 200 mm/min loading rate. The lower ILSS is obtained at ambient temperature at 1 mm/min loading rate.

Environmental exposure	% change in ILSS between 1mm/min-200mm/min	% change in ILSS between 200mm/min-1000mm/min
Ambient temperature	+ 5.8 %	-17.12 %
Thermal spike at +200°C for 5 min	+ 15.10 %	+8.31 %
Thermal spike at +200°C for 10 min	+ 29.71 %	+0.15 %
Thermal spike at +200°C for 15 min	+ 19.41 %	+ 10.04 %
Thermal spike at +200°C for 20 min	+ 26.45 %	+ 8.37 %

**Table 4.8: % change in ILSS values for CFRP composites exposed to thermal spike conditioning at +200°C temperature for different times and tested at different loading rates**

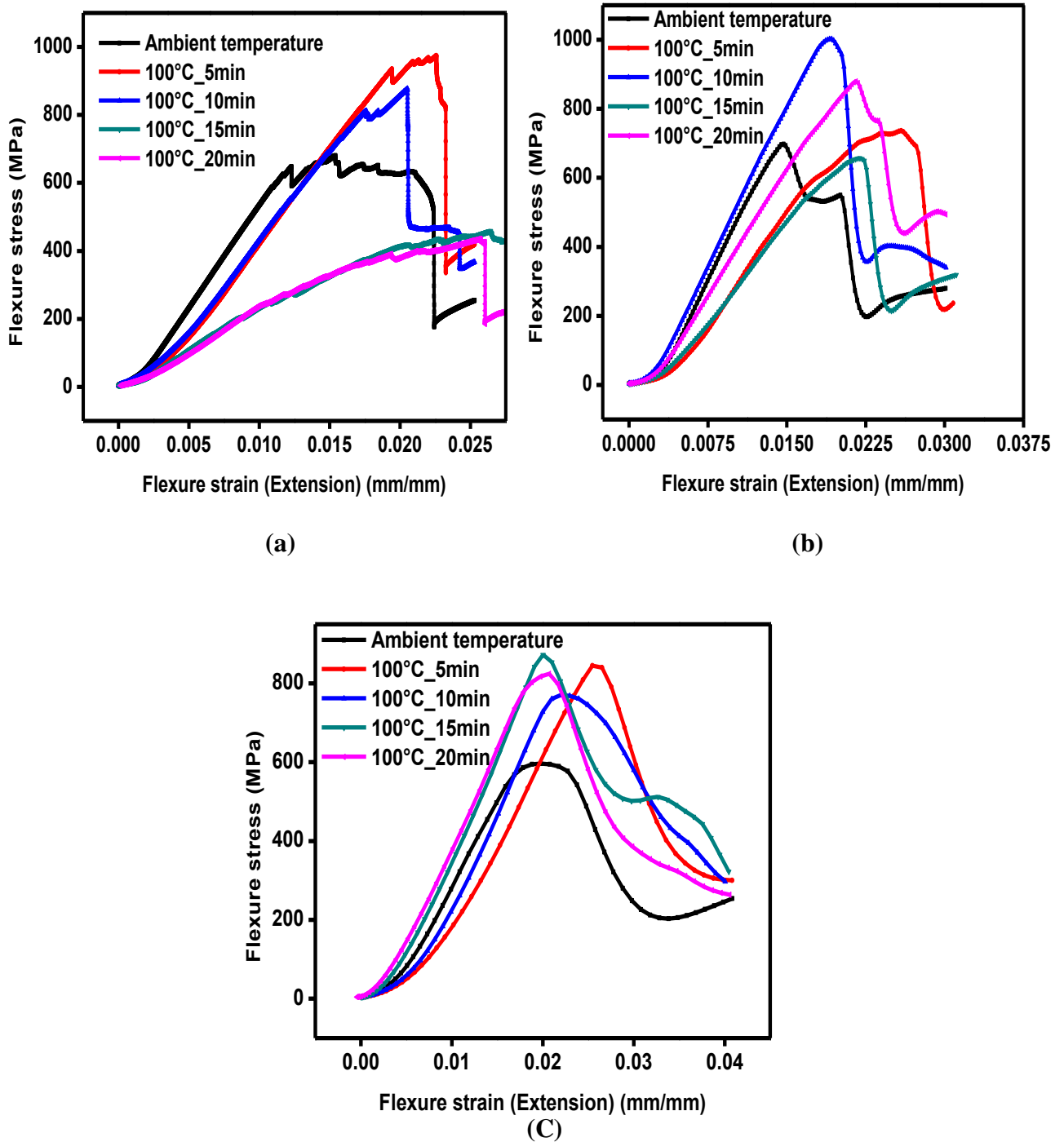
#### 4.2.2.1.2. Stress- strain behavior of CFRP composite

The figure 4.36(a) shows that maximum stress at 1mm/min is obtained 50°C with 10 minutes holding time. The figure 4.36(b) reveal maximum stress at 200 mm/min is obtained at 50°C with 10 minutes holding time. The figure 4.36(c) shows maximum stress at 1000 mm/min is obtained at 50°C with 10 minutes holding time.



**Figure 4.36: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 50°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**

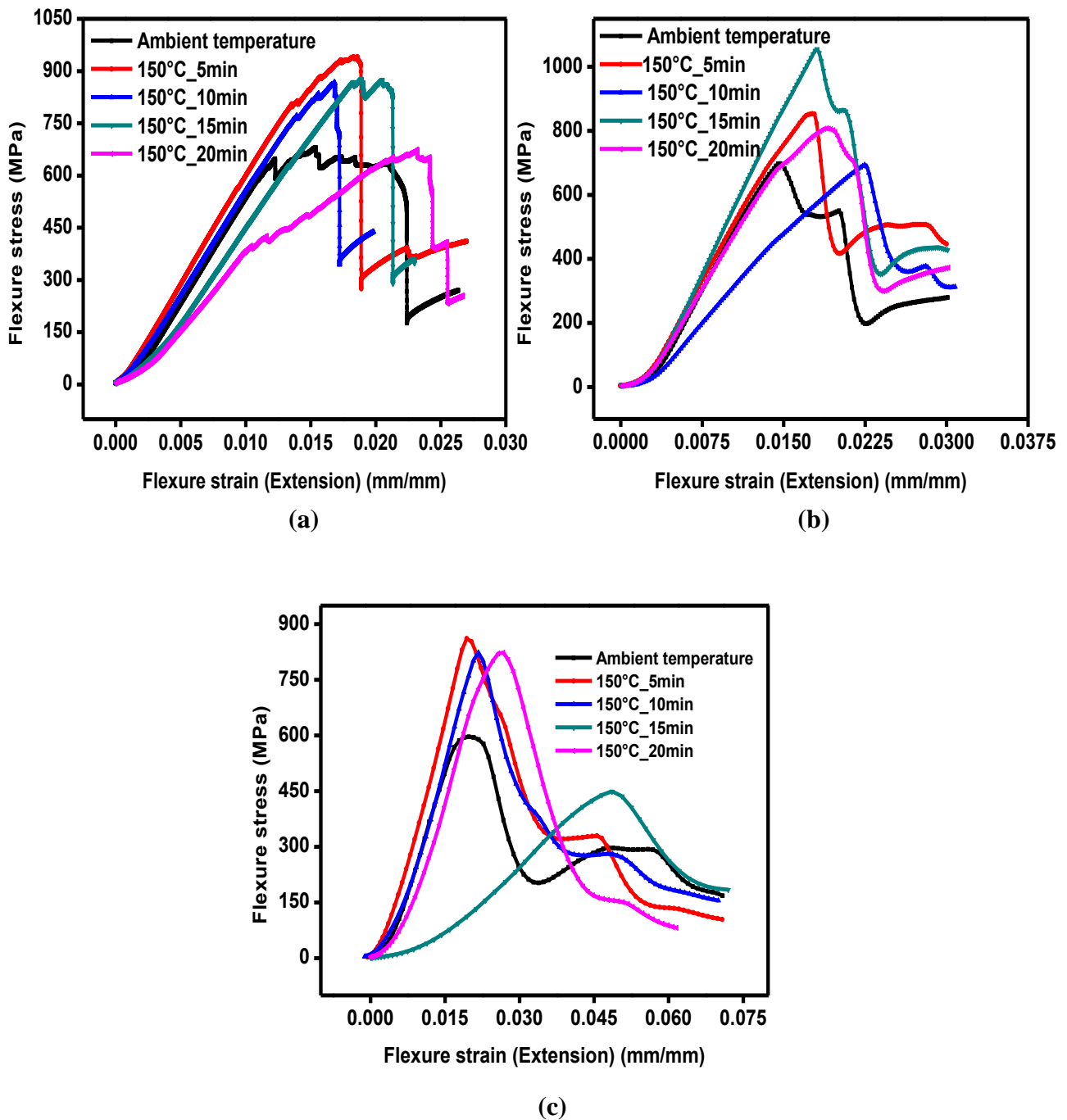
The figure 4.37(a) shows that maximum stress at 1mm/min is obtained 100°C with 5 minutes holding time. The figure 4.37(b) reveal maximum stress at 200 mm/min is obtained at 100°C with 10 minutes holding time. The figure 4.37(c) shows maximum stress at 1000 mm/min is obtained at 100°C with 15 minutes holding time.



**Figure 4.37: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 100°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**

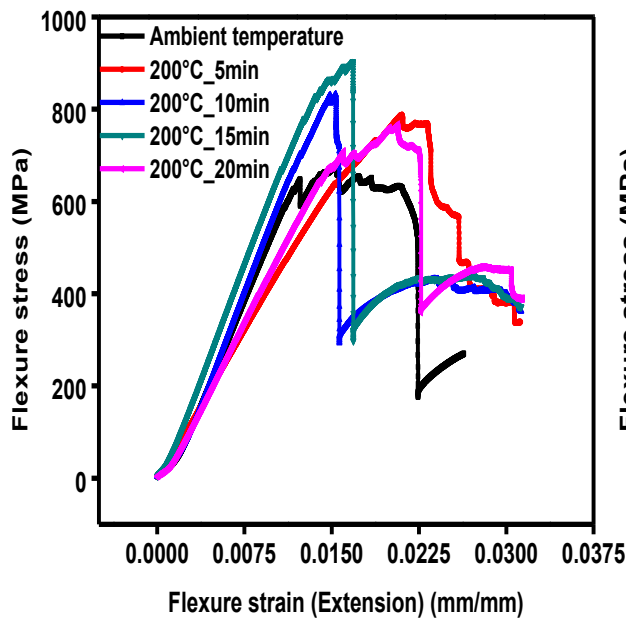
The figure 4.38(a) shows that maximum stress at 1mm/min is obtained 150°C with 5 minutes holding time. The figure 4.38(b) reveal maximum stress at 200 mm/min is obtained at 150°C with 15 minutes holding time. The figure 4.38(c) shows maximum stress at 1000 mm/min is obtained at 150°C with 5 minutes holding time.



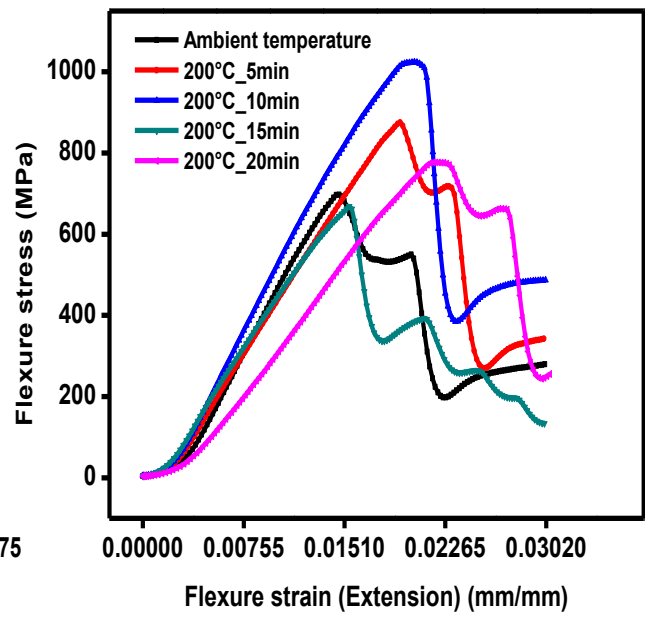


**Figure 4.38: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 150°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**

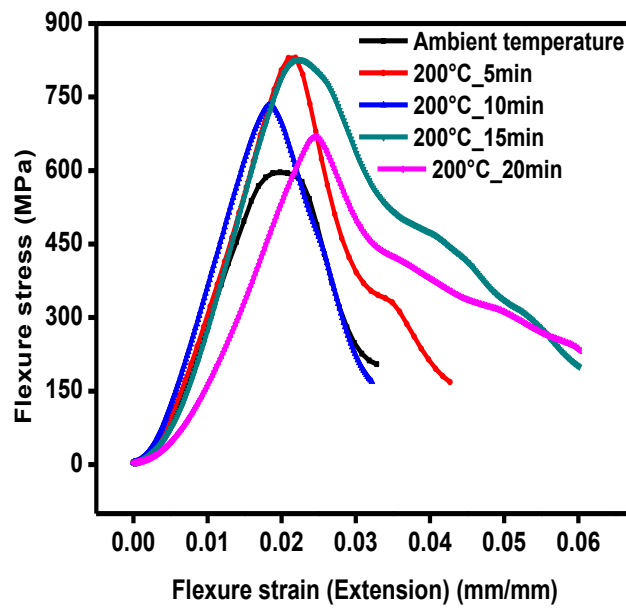
The figure 4.39(a) shows that maximum stress at 1mm/min is obtained 200°C with 15 minutes holding time. The figure 4.39(b) reveal maximum stress at 200 mm/min is obtained at 200°C with 10 minutes holding time. The figure 4.39(c) shows maximum stress at 1000 mm/min is obtained at 200°C with 5 minutes holding time.



(a)



(b)



(c)

**Figure 4.39: Flexure stress Vs Flexure strain plot of thermal spike CFRP samples at 200°C temperature for a holding time of 5, 10, 15 and 20 minutes at (a) 1mm/min loading rate (b) 200 mm/min loading rate and (c) 1000 mm/min loading rate.**

#### 4.2.2.2 Differential Scanning Calorimetry (DSC) Measurements

The glass fibre reinforced polymer (CFRP) composite when exposed to thermal conditioning the glass transition temperature ( $T_g$ ) may be altered.

The glass transition temperature ( $T_g$ ) decreases as the holding time of the thermal conditioning samples increases. This can be attributed to plasticization of the polymer matrix. Glass transition temperature ( $T_g$ ) decreases for low temperature spiking i.e. at 50°C with holding time 5, 10, 15 and 20 minutes, which may be due to debonding. But it increases in case of high temperature thermal spiking i.e. at 100°C, 150°C and 200°C with holding time 5, 10, 15 and 20 minutes due to matrix softening.

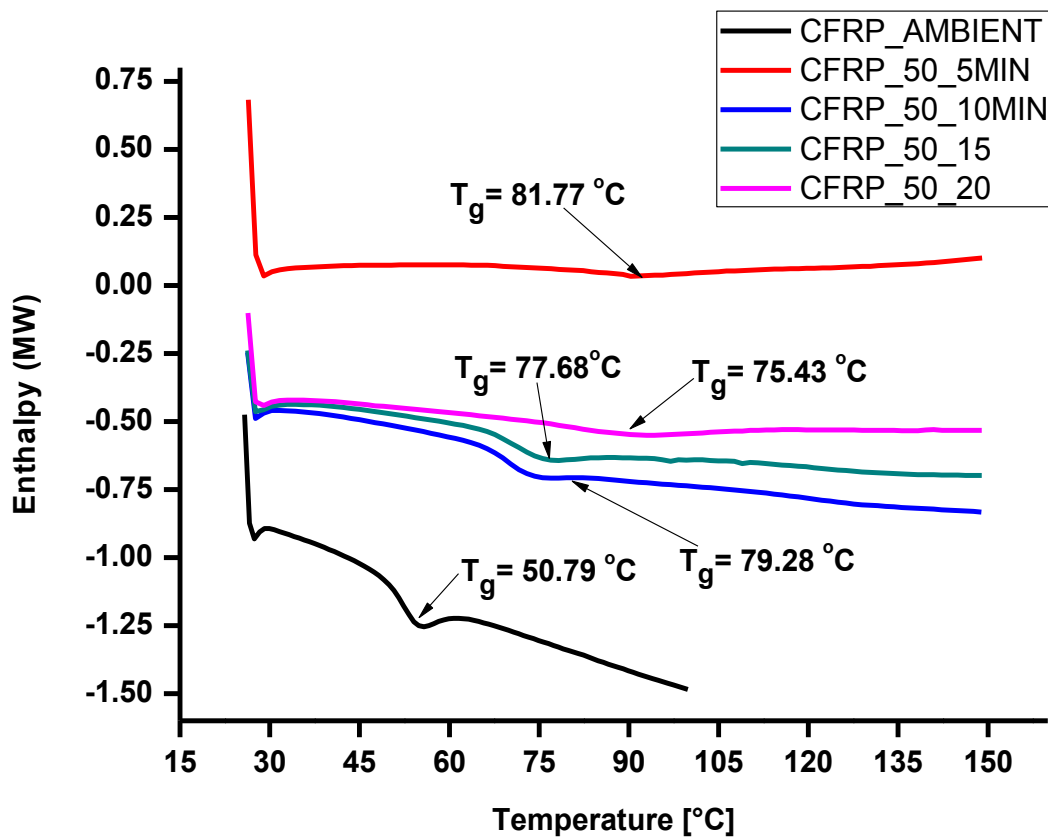


Figure 4.40: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 50°C for a holding time of 5, 10, 15, and 20 minutes.

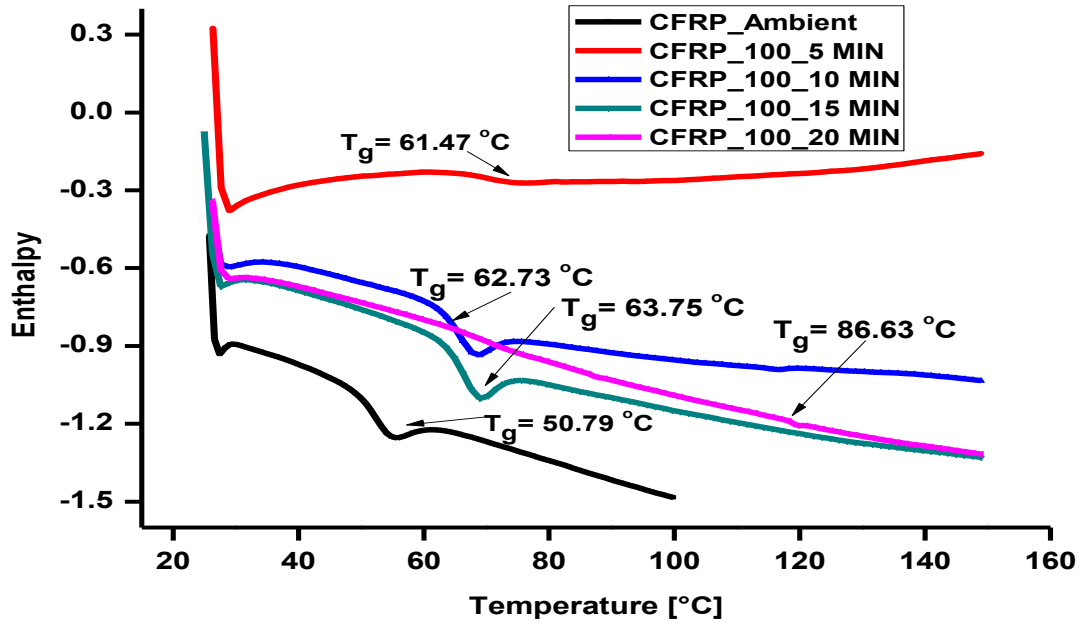


Figure 4.41: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 100°C for a holding time of 5, 10, 15 and 20 minutes.

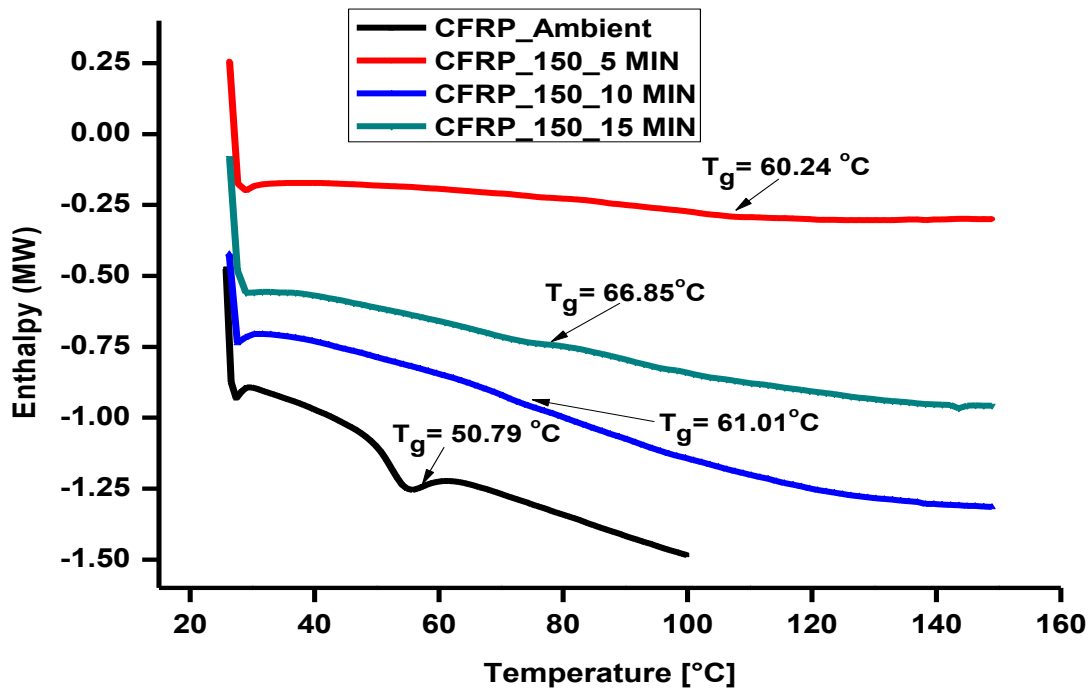
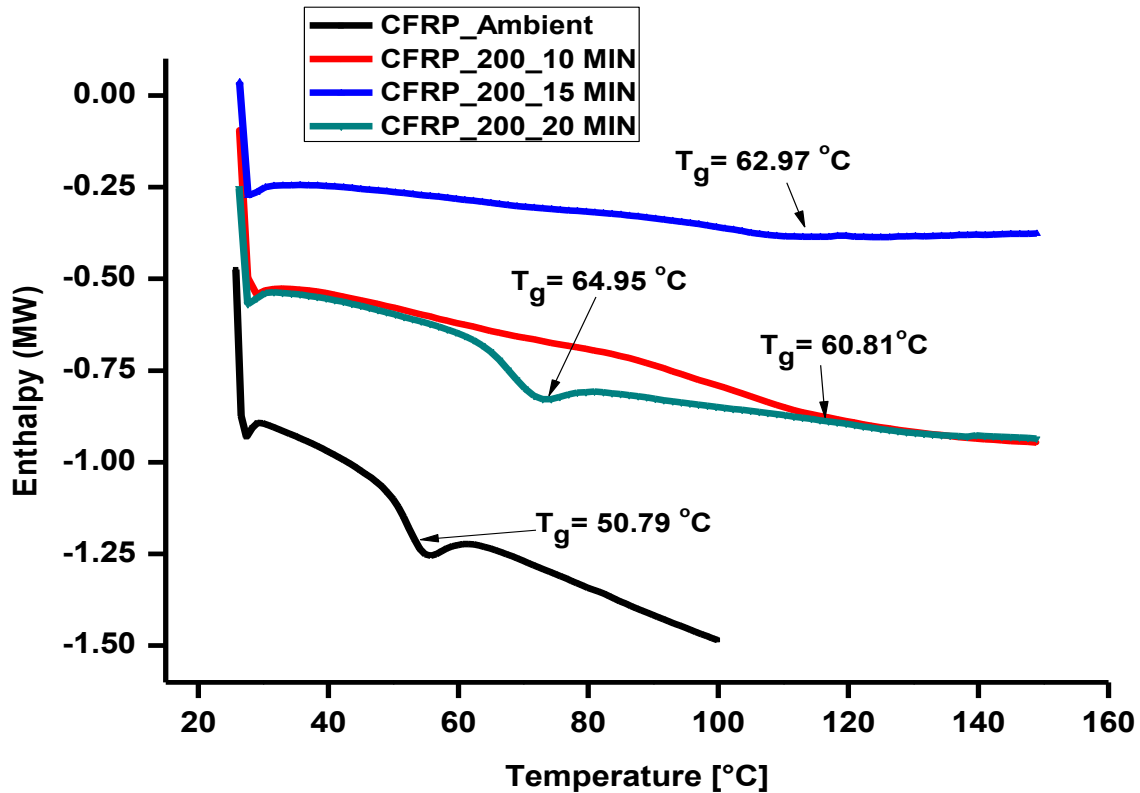


Figure 4.42: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 5, 10 and 15 minutes.



**Figure 4.43: DSC plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 5, 10 and 15 minutes.**

#### 4.2.2.3 Fourier Transformation Infrared Spectroscopy (FTIR-ATR) Analysis

FTIR-Imaging experiments were performed in this study to analysis the thermal spiking conditioning of CFRP composite as IR measurements are very sensitive to hydrogen bonds. The Figure 4.45 to 4.50 shows FTIR spectra of CFRP samples at ambient and 50°C, 100°C,150°C and 200°Ctemperature for a holding time of 5, 10, 15 and 20 minutes respectively. Several bond formations occur in between 4000 $\text{cm}^{-1}$  to 1000  $\text{cm}^{-1}$  wavelength range such as O-H bond, N-H bond, =C-H bond, C=O bond and C-O bond. The O-H bond leads to very complicated spectrum by both intermolecular and intramolecular hydrogen bonding. Any spectral changes are due to the molecular changes in the composites [70]. The fundamental stretching vibration and the in-plane bending vibration occur within the 3900-2800  $\text{cm}^{-1}$  region.

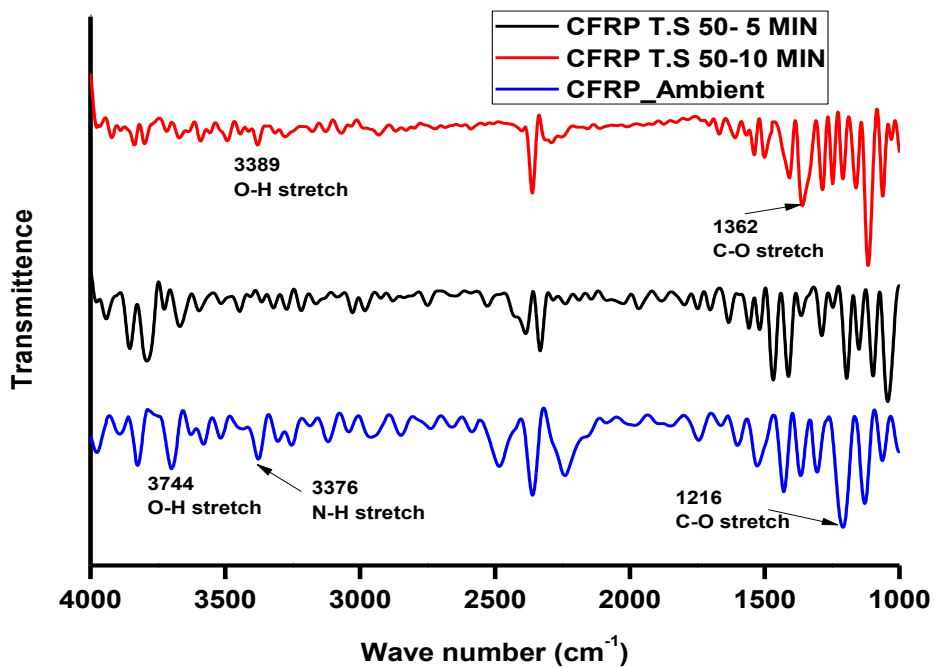


Figure 4.44: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 50°C for a holding time of 5 and 10 minutes.

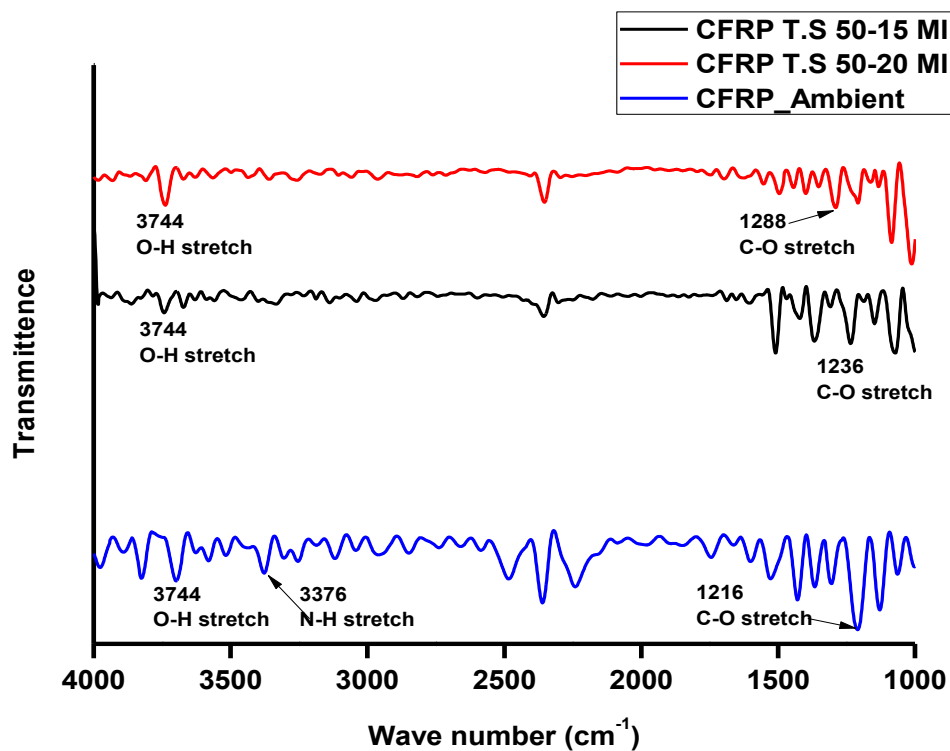


Figure 4.45: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 50°C for a holding time of 15 and 20 minutes.

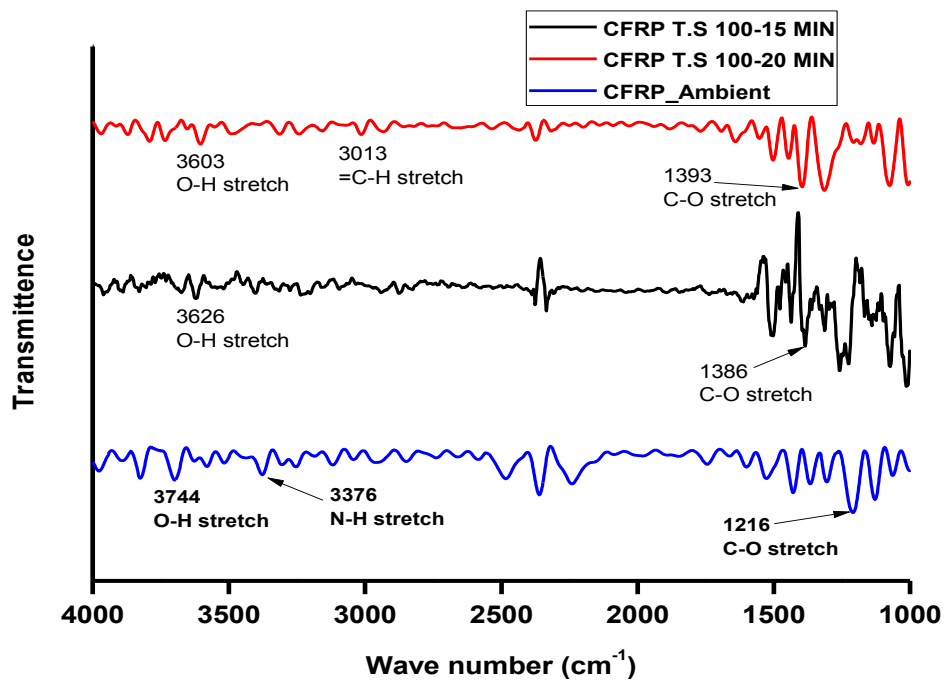


Figure 4.46: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 100°C for a holding time of 15 and 20 minutes.

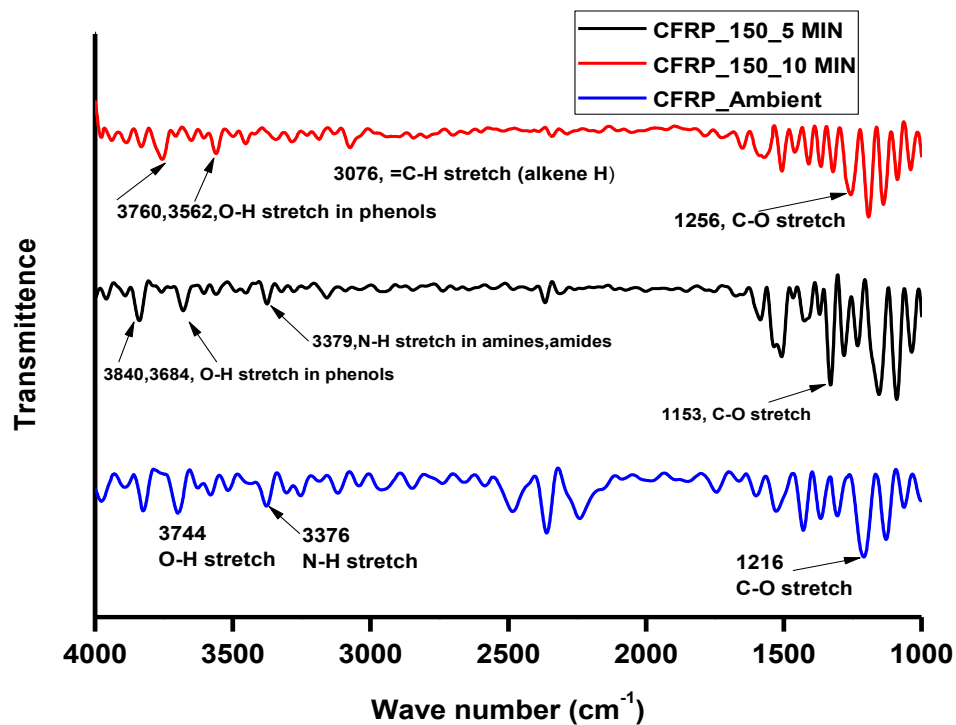


Figure 4.47: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 5 and 10 minutes.

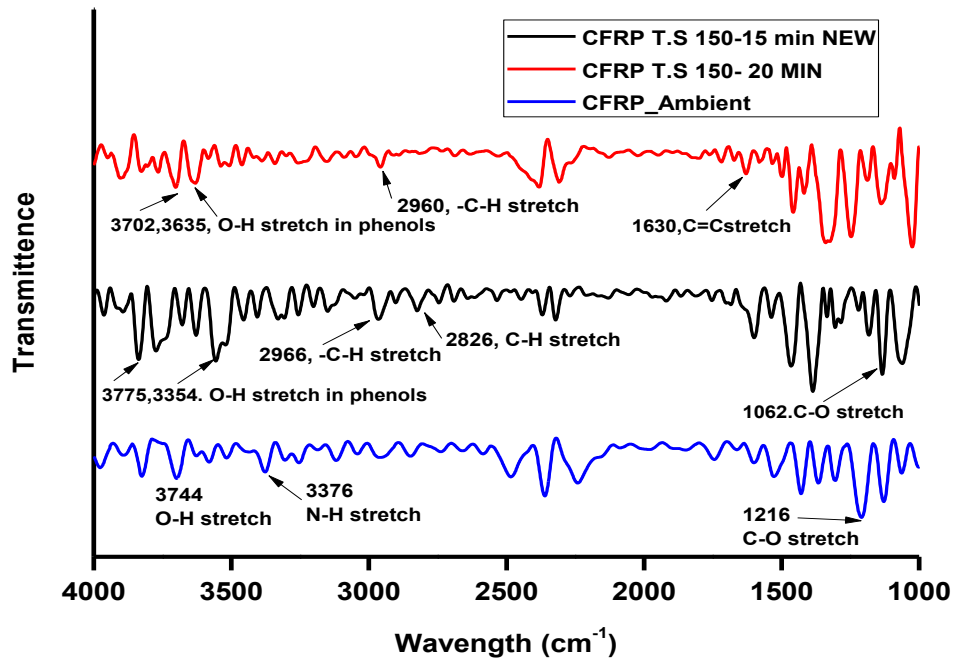


Figure 4.48: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 150°C for a holding time of 15 and 20 minutes.

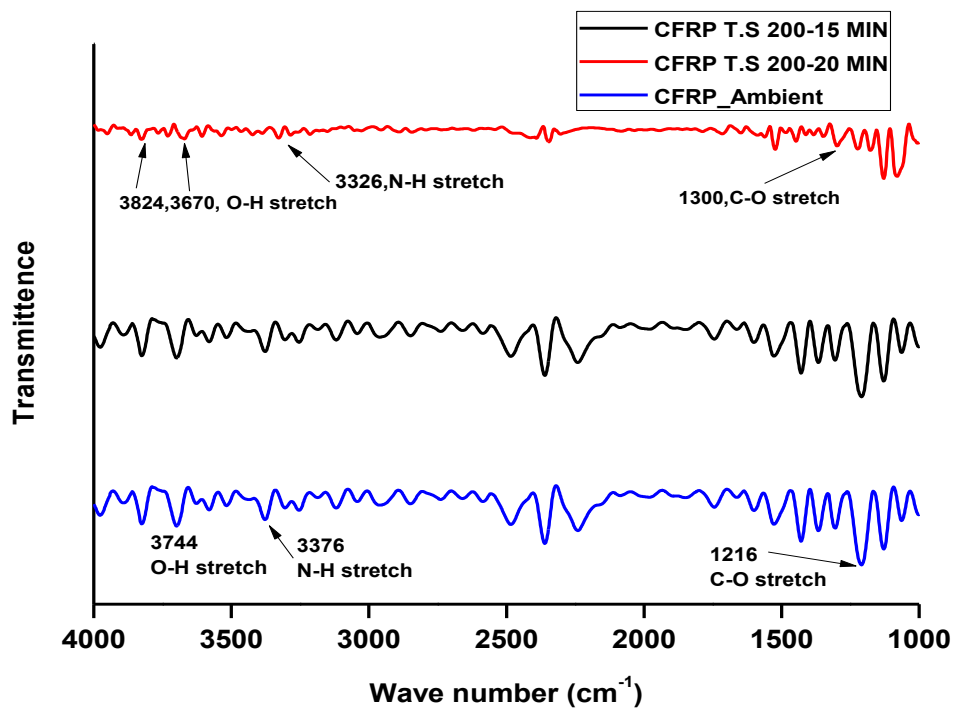


Figure 4.49: FTIR-ATR plot of thermal spike conditioned CFRP samples at ambient temperature and 200°C for a holding time of 15 and 20 minutes.



# Chapter 5

# Conclusion

## 5. Conclusions

The interlaminar shear strength(ILSS) increases with increasing loading rates. At low temperature and low loading rate the interlaminar shear strength(ILSS) is lower, while at low temperature and high loading rate matrix ductility becomes the limiting factor for composite failure. As the temperature increases the interlaminar shear strength(ILSS) goes on decreasing. High interlaminar shear strength have been found at  $-60^{\circ}\text{C}$  which can be attributed for the hardening effect of the polymer matrix and increased mechanical interlocking at fibre/matrix interface. The failure at ambient temperature includes fiber-matrix debonding, ductile fracture of matrix, cusps & fiber imprints, fiber breakage and fiber pullout. Further the effects of thermal spike conditionings on the interlaminar shear behaviour of glass fibre/epoxy and carbon fibre/epoxy composites were also studied. The net result of thermal spike conditionings on the mechanical behaviour of polymer composites is primarily governed by the two mechanism, namely: post curing, and formation of microcracks inside the polymer matrix and/or fibre/polymer interface due to the differential expansion of the fibre and polymer matrix at elevated temperature. It is also found that the ILSS for the specimen undergone thermal spike conditioning are loading rate sensitive. When the specimens were exposed above their glass transition temperature (i.e.)  $150^{\circ}\text{C}$  and  $200^{\circ}\text{C}$  (temperature) Char formation on the surface of the composites were observed. The glass transition temperature due to exposure to  $50^{\circ}\text{C}$  and  $100^{\circ}\text{C}$  thermal spike conditionings for various time do not significantly changes but at  $150^{\circ}\text{C}$  and  $200^{\circ}\text{C}$  there was significant change in  $T_g$ . From FTIR analysis it is further confirmed that these environmental conditionings affects the bonding characteristics of the polymeric composites.

## **6. Scope for future work**

In summary the present piece of work leaves a wide scope for future investigators to explore many other aspects of loading rate sensitivity of thermal conditioned FRP composites at different temperatures. The complex failure mechanisms of glass/epoxy and carbon/epoxy composites require more experimentation for a better characterization of these materials. Implications of thermal conditioning most often lead an improved adhesion of the interface (at above- ambient) and increased crack density (at below- ambient) temperatures. These changes might lead further complications in accessing the loading rate sensitivity which itself as contradictory as on today.

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## **Appendix 1**

### **List of papers presented in conferences based on this project work**

#### **In Conferences**

Presented paper entitled “An assessment of loading rate sensitivity of fibre reinforced polymer composites at low temperatures” **K.K.Mahato\***, **S.Sethi**, and **B.C.Ray** ;  
**International Union of material research society (IUMRS) 2013** at IISC Bangalore, India.