DEGRADATION OF PROPERTIES OF E-GLASS/EPOXY COMPOSITE UNDER HOSTILE ENVIRONMENT

Submitted in partial fulfilment of the requirements of the degree of

DOCTOR OF PHILOSOPHY

by

SURENDRA MOHAN SHRIVASTAVA

(Roll No.: 716020)

Supervisor:

Dr. N. SELVARAJ

(Professor, Dept. of Mechanical Engineering,

NIT, Warangal)

Co-Supervisor:

Dr. MANOJ KUMAR BURAGOHAIN

(Scientist 'G', Associate Director

ASL, DRDO, Hyderabad)

DEPARTMENT OF MECHANICAL ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY

WARANGAL – 506 004, TELANGANA STATE, INDIA

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DEPARTMENT OF MECHANICAL ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY WARANGAL
WARANGAL - 506004, INDIA

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This is to certify that the work presented in the thesis entitled **"DEGRADATION OF PROPERTIES OF E-GLASS/EPOXY COMPOSITE UNDER HOSTILE ENVIRONMENT"** which is being submitted by **Mr. Surendra Mohan Shrivastava (Roll No. 716020),** is a bonafide work submitted to National Institute of Technology, Warangal in partial fulfillment of the requirement for the award of the degree of **Doctor of Philosophy** in **Mechanical Engineering.**

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Prof. N. Selvaraj
Supervisor
Professor
Department of Mechanical Engineering
National Institute of Technology
Warangal, India-506004

Dr. Manoj Kumar Buragohain
Co-Supervisor
Scientist "G", Associate Director
Advanced Systems Laboratory
Defence R&D Organization
Hyderabad, India-500058

Prof. V. Suresh Babu
Chairman, DSC
Professor and Head
Department of Mechanical Engineering
National Institute of Technology
Warangal, India-506004

Approval Sheet

This Thesis entitled **"DEGRADATION OF PROPERTIES OF E-GLASS/EPOXY COMPOSITE UNDER HOSTILE ENVIRONMENT"** by **Surendra Mohan Shrivastava, roll no. 716020** is approved for the degree of Doctor of Philosophy

Examiners

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…….

Supervisor

…….

…….

Co-Supervisor

…….

…….

Chairman

…….

…….

Date:

Place:

Declaration

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(Signature)

Surendra Mohan Shrivastava

(Name of the Student)

(Roll No. 716020)

Date:…….

I would like to dedicate the thesis to my beloved father

Late. Shri Shyam Bihari Shrivastava

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(Surendra Mohan Shrivastava)

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List of Abbreviations

ASTM	:	American Society for Testing and Materials
ATF	:	Aviation Turbine Fuel
AVGAS	:	Aviation Gasoline
BD	:	Bidirectional
DSC	:	Differential Scanning Calorimetry
FTIR	:	Fourier Transform Infrared
GFRP	:	Glass Fiber Reinforced Polymer
CFRP	:	Carbon Fiber Reinforced Polymer
GPa	:	Giga Pascal
hrs	:	Hours
ILSS	:	Interlaminar Shear Strength
IPSS	:	In-plane Shear Strength
kN	:	Kilo Newton
mm	:	Millimetre
MPa	:	Mega Pascal
NDT	:	Non-Destructive Testing
SEM	:	Scanning Electron Microscope
UT	:	Ultrasonic Testing
VARTM	:	Vacuum Assisted Resin Transfer Molding
RTM	:	Resin Transfer Molding
WRC	:	Without Resin Coating

List of Symbols

A	:	Area of cross section (mm^2)
b_c	:	Width of the compressive test specimen (mm)
b_f	:	Width of the flexural test specimen (mm)
b_s	:	Width of the ILSS test specimen (mm)
b_t	:	Width of the tensile test specimen (mm)
d_f	:	Depth of flexural test specimen (mm)
d_s	:	Depth of ILSS test specimen (mm)
E	:	Modulus of elasticity in bending (GPa)
L	:	Length of flexural specimen (mm)
m	:	Slope of the tangent to the initial straight portion of the load deflection curve (N/mm)
M_t	:	ATF gain (%)
M_s	:	Seawater gain (%)
P_c	:	Compressive load (N)
P_f	:	Load at a given point on the load-deflection curve (N)
P_i	:	Applied load in IPSS test (N)
P_s	:	Maximum load observed during shear test (N)
P_t	:	Tensile load taken by test specimen (N)
t_c	:	Thickness of the compressive test specimen (mm)
t_t	:	Thickness of the tensile test specimen (mm)
w_0	:	Dry specimen weight before ATF immersion (grams)
w_{0s}	:	Dry specimen weight before seawater immersion (grams)
Δw	:	Weight gain after ATF immersion at time ‘t’ (grams)
w_t	:	Wet specimen weight after ATF immersion at time ‘t’ (grams)
w_{ts}	:	Wet specimen weight after seawater immersion at time ‘t’ (grams)
Δw_s	:	Weight gain after seawater immersion at time ‘t’ (grams)
σ_c	:	Compressive stress (MPa)
σ_f	:	Flexural stress (MPa)

σ_t	:	Tensile stress (MPa)
ε_x	:	Normal strain in the longitudinal direction ($\mu\varepsilon$)
ε_y	:	Normal strain in the lateral direction ($\mu\varepsilon$)
τ_s	:	Interlaminar shear stress (MPa)
τ_{12}	:	Maximum in-plane shear stress (MPa)
γ_{12}	:	Maximum shear strain ($\mu\varepsilon$)
$\Delta T/T$	:	Normalized tensile strength parameter
$\Delta L/L$	:	Normalized ILSS parameter
$\Delta F/F$	:	Normalized flexural strength parameter

Abstract

The relentless passion of the aerospace industry to augment its performance is constantly driving the way to explore high-performance material i.e., composite. Recent usage of composite has got increased as an alternate material of metals, primarily because of its low weight that comes out of its high specific strength and modulus and its improved properties. The other advantages like tailorbility to specific requirements, drapability to complex shapes, low coefficient of thermal expansion to get dimensional stability, and corrosion resistance, are a few other properties that have outweighed the metals.

E-glass/epoxy composite which is being used for various structural applications was identified as a candidate material for this study. Two types of exposure i.e., Aviation Turbine Fuel (ATF) and seawater exposure were identified as hostile environments to said composite which will degrade the same. This research work has identified the testing parameters with three different categories of test specimens i.e., bare, with 0.1 mm and 0.2 mm resin coated condition to quantify the percentage of degradation, depicts degradation mechanism and means to keep this degradation to the minimum. To quantify the degradation after the exposures like ATF and seawater, the properties like tensile, compressive, flexural and shear were evaluated and compared with as received properties.

To assess and compare the degradation mechanism, the test specimens have undergone examination of test specimens without and with the respective exposure, using a Stereo microscope for surface investigation. A Scanning Electron Microscope (SEM) was too used for sub-surface investigation. The fractured surface of the tested specimens without exposure (as received), and bare and resin-coated specimens after respective ATF/Seawater exposure, were investigated using SEM. SEM micrographs were analyzed and compared, to establish the degradation mechanism.

Test results show that ATF fuel exposure has reduced the interlaminar shear strength (ILSS) by 10.04%, 7.83%, and 6.01% for bare, with 0.1 mm and 0.2 mm resin coating, respectively. Similarly, in-plane shear strength (IPSS) was reduced by 14.75%, 11.22%, and 7.52% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and in-plane shear modulus was reduced by 10.87%, 8.94%, and 6.52% for bare, with 0.1 mm and 0.2 mm resin coating conditions as compared to as-received (without ATF exposure) specimens.

SEM micrographs and results too showed that properties were reduced and indicated that the E-glass/epoxy composite was resistive to fuel ingression. It was observed that bare specimens exhibited a reduction in shear properties due to ATF ingression to the polymeric network and induced internal stresses, which not only degraded the matrix and fiber-matrix adherence but created micro-cracks too in the resin at interfaces. Resin-coated specimens limit fuel ingression, which has led to a reduction in properties.

Test results show that seawater exposure to test specimens has notably reduced their tensile and flexural properties as compared to as-received specimens (without seawater exposure). The seawater exposure has reduced the tensile strength by 24.09%, 17.65%, and 10.23% in warp and 22.96%, 15.47%, and 8.37% in weft direction for bare, with 0.1 mm and 0.2 mm resin coating, respectively and 14.30%, 8.57% and 5.87% in warp and 12.96%, 8.85% and 6.33% degradation of tensile modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions. Similarly, flexural strength was reduced by 20.38%, 12.75%, and 9.93% in warp and by 17.41%, 10.85%, and 9.80% in weft direction for bare, with 0.1 mm and 0.2 mm resin coating, respectively and flexural modulus were reduced 12.52%, 8.58% and 5.79% in warp and 10.86%, 7.71% and 5.28% in weft direction for bare, with 0.1 mm and 0.2 mm resin coating conditions as compared to as-received specimens.

It was observed from SEM micrographs too, that bare specimens exhibited a reduction in properties due to the diffusion of seawater, which will be followed by a reaction of hydrolysis in the composite. It is not only degrading the reinforcement and fiber matrix adherence but will create micro-cracks too in the resin at interfaces. It was illustrated that the test specimens with barrier resin coated limit the seawater ingression, which has led to relatively less reduction in properties. The optimum thickness of the barrier resin coating was derived from plotting the normalized parameter w.r.t thickness of the coating.

Moreover, specimens with 0.1 mm and 0.2 mm resin-coated exhibited comparable slight decrement of mechanical properties after ATF/Seawater ingression. In addition, 0.2 mm resin-coated were comparable and more impermeable to ATF/Seawater ingression than 0.1 mm resin-coated specimens, and ATF/Seawater ingression was restricted to the top surface and prevented its ingression to fiber-matrix interfaces, which has further led to only a slight reduction in the properties to the specimens which were coated.

It was observed that Resin coat application is found, to help in minimizing the degradation of properties after ATF/Seawater exposure. The plot of normalized mechanical strength parameters for bare and resin-coated specimens was plotted with respect to coating thickness, in the form of a curve to establish the optimum coating thickness, which will exhibit a minimum reduction of properties.

Hence in nutshell, this investigation identified and explores the effect of some of the hostile environments like aviation turbine fuel and seawater exposure to this material to establish its design margin for its usage. In addition to this, the degradation mechanism of conditioned specimens was also studied using a stereo microscope, and SEM. It was noteworthy that resin coating of varying thickness over the test specimens were restricting the ATF/Seawater ingression and evolved the minimum coating thickness to keep the degradation to the minimum.

Chapter 1

Introduction

1.1 Introduction

Composite materials are distinct from the other materials as the constituent materials are discrete at the molecular level and are mechanically distinguishable too. In combined form, the constituent materials work together and their original forms will remain intact. The concluding composite materials properties are superior to constituent material properties. These days, composite materials have become engineering materials, which are common and developed and manufactured for several applications including aerospace parts, consumer goods, sporting goods, automotive components, and the oil and marine industries. The composite usage growth was also seen as a result of enhanced awareness regarding the performance of the product and the global market's growing competition for reduced-weight components. Composite materials have the potential to replace widely used aluminum and steel, and many times with superior performance relative to other materials.

Today, apparently composites are the preferable choice of materials for many applications of engineering [1,2].

Increased usage of composite in the aerospace industry was possible because of its advantages like significant weight reduction, drapability, high thermal stability and impact resistance, resistance to fatigue and corrosion, and ease of assembly. Composite materials enable the integration of parts, which will be led to the replacement of various metallic components by a single monolithic composite component [3]. Rapidly, these key factors of the composite are outweighing the usage of metal in the aerospace industry [4-5].

E-glass fibers were chosen as it is electrical grade of glass, which was being used as insulator, and having the excellent fiber forming capabilities, leading to be used exclusively as a reinforcement. This fiber is widely used among all fibrous reinforcement because of low in cost and early development compared to others.

Epoxy resin is the prime choice for the composites, which is being used as a structural material, because of its higher tensile strength, low shrinkage and low volatility.

1.2 Classification of Composites

Recently, fiber-reinforced polymer composites (FRPs), developed by several types of matrices (metallic, ceramics, polymeric, etc.), were reinforced with fiber materials, which were utilized to solve numerous technological issues. The classification based on reinforcement geometry is as follows and depicted as shown in Fig. 1.1.

1.2.1 Particulate Composites

Particles are immersed in matrices like ceramics and alloys. They are mostly isotropic in nature because particles reinforcement was added randomly. They possess advantages of increased operating temperature resistance, improved strength, oxidation resistance, etc. Typical examples include use of silicon carbide particles in aluminum and aluminum particles suspended in rubber; and cement, gravel and sand to make concrete.

1.2.2 Flake Composites

Reinforcement in the form of flakes will be added into the matrices to make such kind of composites. Orienting the flakes in definite direction is uphill task, so limited numbers of materials are available to use. added advantage like low-cost and high out of plane bending strength can be achieved using this composite.

1.2.3 Fibrous Composites

Reinforcement in the form of continuous (long) and discontinuous (short) fiber will be added into the matrices to make such kind of composites. Continuous fiber is being used

for making unidirectional and woven fabrics. Short fibers are being used for making randomly dispersed fiber composites.

In addition to this, composites were sub-categorised based on type of matrices used as polymeric, metal, ceramic ad carbon matrix composites. Woven fabrics impregnated with resin (matrices) will be stacked one over the other to make laminate of the composite. Each layer of the laminate can be oriented in different directions to fulfil the structural requirements and can be made of different material systems.

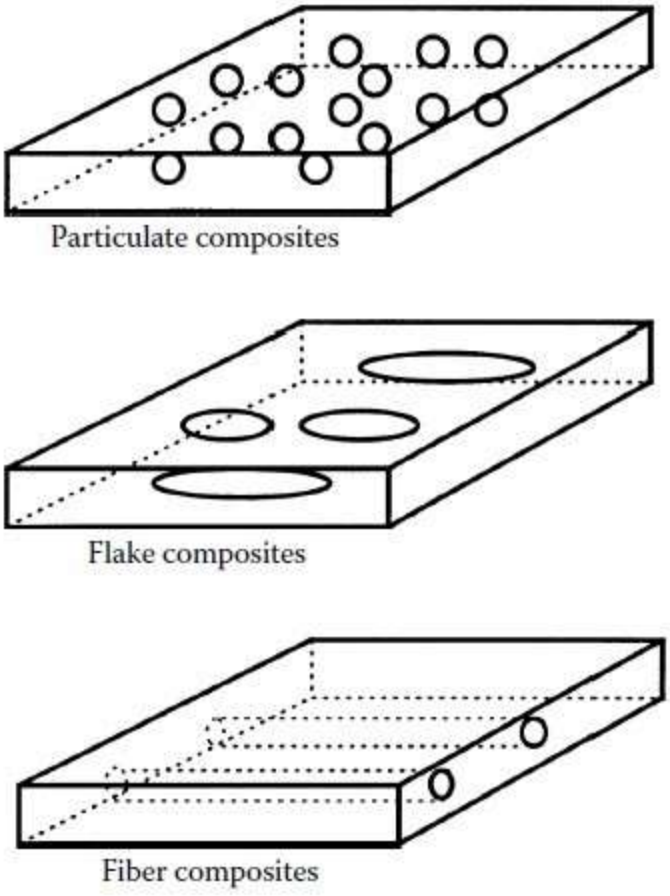


Fig. 1.1 Different types of composites based on reinforcement [Source: Kaw, A.K.,2006]

1.3 Directional Significance of Composite Laminate

The warp and weft are two basic elements that are being used in weaving to turn yarn into fabric. The longitudinal or lengthwise direction of the fabric is known as warp, similarly, the width-wise or transverse direction is designated as weft. While weaving the fabric, warp-direction yarns will be kept stationary and weft-direction yarns will weave through over and under the warp as shown in Fig. 1.2.

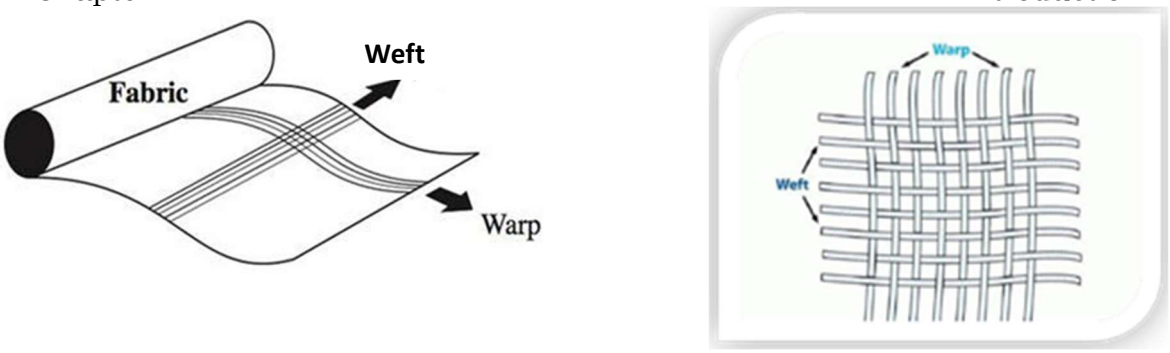


Fig. 1.2 Warp and weft direction of the fabric

1.4 Application of Composites

1.4.1 Aircraft Industry

Military aircraft is primarily using the structures made with composite, as the structure made with this, will have reduced weight ad high specific strength and stiffness. Contrary to that usage of composite is limited to commercial aircraft because of safety concerns, hence the secondary structure like panel, flooring, rudder, and elevator will be made.

1.4.2 Space Industry

The composite is primarily being used in space because of its low weight and exhibition of dimensional stability in large temperature variations in space. The outer envelope of space shuttle made with carbon-based composite not only provide requisite structural strength but also enhances its capability to bear the extreme temperature during re-entry. In other words, usage of composite provides the excellent thermal protection systems because of its ablative characteristics.

1.4.3 Sporting Goods

Increasing usage of composite in sporting goods industry has become the reality nowadays because of its low weight, vibration damping characteristics and torsional rigidity. It is being used for golf club shafts, bicycles, tennis racquets, ice hockey sticks etc.

1.4.4 Medical Devices

Mostly usage of composite in this field is catching attention because of its low weight and transparency to radiation characteristics. The face mask, portable artificial lungs and x-ray tables are being fabricated using composites.

1.4.5 Marine Application

The uses of composite in marine application were driven by its low weight and corrosion resistance. Boats are being made by using glass-based composites. The resin like vinyle ester is well suited

for this application because of its resistance towards high corrosive environments. Bridges are made using composite as they are durable and supports longer life span.

1.4.6 Automobile Industry

Composite is widely being used in automotive industry because of its low weight and excellent corrosion resistance. It is mostly used in manufacturing of leaf spring, doors, bumpers and body panels of automobiles.

1.5 Different Types of Hostile Environments

The structure made out of composite will undergo the exposure of various types of hostile environments during its lifespan i.e., seawater, ultraviolet, hygrothermal, salt water, ATF exposure etc. As this research work was focused to study the degradation of E-glass/epoxy composite, which is being used as structural material for aircraft application, its exposure to the environments like Aviation Turbine Fuel (ATF) and seawater were primarily chosen to know the affect over said composite.

1.5.1 ATF Environments

ATF exposure is one of the hostile environments, which can affect its mechanical properties. Composition of ATF fuel mainly comprises paraffins and aromatics. In addition to this, olefins, sulphur, nitrogen, and oxygen containing compounds were also present in its composition. The exposure to this environment were studied and effects of this to the said composite were analyzed.

1.5.2 Seawater Environments

Seawater environments is another type of hostile environments, which will affect the mechanical properties of E-glass/epoxy composite. The seawater contains primarily dissolved ions and mainly comprises the elements like oxygen, hydrogen, chlorine, sodium, magnesium, sulphur, calcium, potassium, bromine, and carbon. The exposure of this environments was studied and in detail analyzed for said composite.

1.6 Organization of the Thesis

This research work was focused to study the effect of ATF and seawater ingression over E-glass/epoxy composite in bare and with different thicknesses of resin-coated condition to quantify its degradation. In addition to this, its results were properly supported with stereoscope, SEM, and energy dispersive spectroscopy observations to give impetus to the degradation mechanism. The thesis is organized as follows:

Chapter 1 provides a brief introduction about the composites and explains the different types of environmental exposures and how they can degrade their performance.

Chapter 2 deals with the knowledge available to address this problem and quotes the various study in this regard. After elaborate deliberation over this topic literature gaps were identified and discussed. In addition to this, the objective of the problem was outlined.

Chapter 3 describes the material used for this study and the methodology to prepare the test specimens out of laminate as per their respective ASTM standard. It identifies the properties which have to be evaluated. It explains the testing methodology and evaluates the respective properties. In addition to this, this chapter focussed to establish the as-received property as this will be used as a reference value to compare the effect of exposure and will quantify the degradation.

Chapter 4 illuminated the periodic measurements of gain in weight data for test samples of various tests after ATF immersion and were demonstrated in the form of curve with respect to time for all three categories of test specimens for different mechanical properties evaluation after ATF exposure.

Chapter 5 elucidated gain in weight data after periodic measurements of the test samples of various tests after seawater immersion and were demonstrated in the form of curve with respect to time for all three categories of test specimens for different mechanical properties evaluation after seawater exposure.

Chapter 6 evaluated and exhibited the test results the various properties of E-glass/epoxy composite because of ATF and seawater exposure and quantifies the percentage of degradation of the said composite in ATF and seawater environments. This chapter has also examined and analyzed the fractured fiber and surface of ATF and seawater exposed tested samples using SEM and stereo microscope. In detail, this chapter discusses the results based on associated degradation mechanisms i.e., surficial and subsurface ingression. Surface ingression was based on a stereo microscope study and sub-surface degradation mechanisms were elaborated based on scanning electron microscope. In addition to this, evaluated the minimum coating thickness of resin coating for minimizing the degradation to ATF and seawater exposure.

Chapter 7 concludes, summarizes, and compares the results of the research work under ATF and seawater exposure and the scope of the future work were presented.

Chapter 2

Literature Review

2.1 Introduction

E-glass/epoxy prepreg was chosen as a candidate material for this study, as this material is being used as a structural material in various sectors like aircraft, aerospace automotive, sports, etc., to cater to their requirements. Among its various application, it was seen that worldwide, on one side this material is being used as a structural material of fuel carrier to carry additional fuel (ATF) supply to fighter aircraft, on the other side the structure made of this composite like helicopters and ultra-light aircraft operates over the water basin (like rivers, oceans, etc.) and fly over the low altitude. From these cases, it was evident that said composite absorbs the ATF/Seawater followed by its gradual degradation of the structure. Its wide-ranging application under different environments draws attention to establish its longer durability under extreme conditions. So, to quantify the degradation, assessment of the degradation mechanism and keeping the degradation to the minimum was the prime motive of this research work. As this degradation cannot be completely avoided, so to limit and quantify the degradation, specimens as

per ASTM standards were made and a portion of the specimens was coated with the resin of varying thickness i.e., 0.1 mm and 0.2 mm resin coated. Both the resin-coated and bare specimens were exposed to ATF and seawater separately by immersing them in respective environments till saturation of all conditions of the specimens, for around two months to ATF and two and half months for seawater exposure. Afterward, test specimens from the respective exposure were taken out and dried up with tissue paper, followed by mechanical testing. Before going further in this study, the specified properties (as-received) of this material are to be evolved separately to compare or to establish the correlation with any changes brought by these hostile environments exposures. Test results of both exposures were correlated and compared with separately evolved specified property (as-received property i.e., without any exposure) to quantify the degradation.

During the storage for a longer span, the long-term performance of the composite is to be ensured. As the structures, which are being made using these composites are expected to be in service for a longer time and it has to be exposed to various types of loading and environmental conditions. In aerospace, the composite drop tanks for fighter aircraft are made of E-glass/epoxy composites and are being used to carry aviation turbine fuel. Hence the E-glass/epoxy composite's tendency towards the absorption of the fuel and seawater will decide its degradation of mechanical properties. This degradation will be detrimental to the structural performance. So, studying the effect of ATF and seawater exposure on E-glass/epoxy composite has become very significant to establish the design margin for the composite structures, which store the fuel.

2.2 Effect of the Hostile Environments over Different Types of Composites

To analyze the effect of ATF and seawater effect over glass/epoxy composite, an extensive literature survey was carried out, as it was helpful in better understanding of the problem and focused the study in the right direction. It was observed that different researchers have taken different candidate materials and different environmental exposure to study this problem in bare specimens like Kumarasamy et al. [6] described the effect of different types of fuels over tensile and flexural properties of glass/epoxy composite. Three types of fuels namely bio-diesel, aviation fuel (Jet-A), and a blended mixture of bio-diesel and aviation fuel were used in their study for the environmental aging of said composite. The glass/epoxy specimens, which were made using the Vacuum Assisted Resin Transfer Molding (VARTM) method were immersed separately into these fuels till the saturation point was attained and it was observed that % of the mass gain of the fuel was highest in aviation fuel and lowest in the blended mixture of fuel. Once the specimen got saturated it has undergone tensile and compression tests. The data were correlated with as-received

(non-immersed) specimens and it was found that tensile and compressive properties were reduced slightly. The reduction to tensile strength was maximum to bio-diesel exposure and minimum to aviation fuel, likewise for tensile modulus biodiesel immersion has shown greater reduction. Similarly, the outcome of the compressive test exhibited that aviation fuel immersion has reduced the compressive properties the most. This study concluded that micro cracks and void formation during immersion were responsible for reduction as the fuel molecules penetrate the polymeric chain and deformed because of generated internal stresses.

The effect of Glass Fiber Reinforced Polymer (GFRP) laminate's flexural properties, after immersing the same in aviation fuel i.e., Aviation Gasoline (AVGAS) 100 LL and ATF, and the laminate was fabricated using a vacuum infusion technique, were elucidated [7-8]. It was reported that exposure of both the solutions of aviation fuel to GFRP laminate affected the flexural properties and showed a reduction. The effect was relatively more for ATF immersion on the reduction of flexural properties.

Researches [9-10] described the seawater ageing on composites used for the marine structure. The moisture's effect on the mechanical behavior of composites has been expanded to quantify interactions between mechanical stresses and diffusion of water. This combined approach was examined using experimental equipment and technique. Cavasin, et al. [11] explained the advanced testing methodology to determine the exposure effect of seawater over glass fiber-reinforced polymer composites and on their physical performance, were also studied.

Amaro, et al. [12] illustrated the influence of various types of acid solutions on E-glass/epoxy composites. In their investigation, glass/epoxy composite underwent exposure to acids like hydrochloric and sulphuric acid, and its flexural and impact strength response was recorded. It was observed that these solutions notably affect the said properties and hydrochloric acid affects the most. Research were reported [13-14] an in-service assessment of fiber-reinforced polymeric composite in distinct environmental conditions.

Baig, et al. [15] studied the recent advancement in the development of epoxy resin coating for the tribological application. It was observed that several metallic, polymeric, ceramic, and carbon-based filler suspensions to epoxy resin matrices will improve the tribological performance of the coating.

Gujjar, et al. [16] described the effect of different types of resin coating on a mild steel surface to get the ideal one. These resins (epoxy, phenolic, polyurethane, and polyester) were applied to the

metallic surface using the pneumatic spray method and after this immersion as well a salt spray test using a solution of NaCl was carried out, which was followed by the estimation of the rate of corrosion and evaluation of its mechanical properties and found that epoxy resin coating was extremely effective and exhibited better properties relative to other coatings.

The influence of the absorption of moisture on the mechanical properties of E-glass/epoxy composite under hygrothermal conditioning and hydrothermal immersion, were demonstrated [17-19]. It was found that hygrothermal exposure is more detrimental compared to hydrothermal, but temperature and time of exposure also play an important role irrespective of the type of exposure. The ILSS and glass transition temperature properties were reduced after hygrothermal conditioning and hydrothermal immersion, as the moisture not only induces the matrix plasticization and swelling but will lead to the breakdown of a chemical bond at the interfaces of the fiber and matrix.

The efficacy of seawater treatment on the reduction of bending and tensile strength of basalt and E-glass/epoxy composites, were illustrated by Wei et al. [20]. It was found that the deterioration of these composites was observed because of the combination of absorption of water and extraction of soluble material, where absorption of water led to matrix swelling and cracking and extraction of soluble material will degrade the interfacial adhesion of fiber and matrix.

Studies were carried out to know the effects of various environmental conditions on the epoxy's mechanical characteristics too [21-23]. One of the studies [24] reported the protective efficiency of epoxy-based coating on the metallic plate in NaOH, HCl, and distilled water media. Test result outcome shows that epoxy resin coating was effective for the metallic plate of mild steel in HCl and distilled water media but ineffective for NaOH media. The effect of montmorillonite addition to resin system of glass/epoxy composite were also analyzed [25] for its water absorption and mechanical properties.

The influence of moisture on the various mechanical properties of glass fiber-reinforced polyamide resin composites, were exhibited by Chaichanawong et al. [26] and Akay, et al. [27] focussed on the impact of moisture on the mechanical as well as the thermal properties of oven cured and autoclaved kevlar-49/epoxy laminates. One of the research discussed to evaluate the relaxation of fiber-matrix interfacial behavior caused by water absorption in composites by Tsenoglou et al. [28]. A study was conducted by Sateesh et al. [29] to know the degradation of GFRP composites in different environmental conditions to assess its influence on flexural modulus. The results outcome showed that modulus degradation caused by the absorption of moisture and temperature effects impacts the lifecycle of GFRP composites exposed to water for a long period.

The some of the researchers [30-42] studied and analyzed the effect of seawater on composites. Even, the degradation of fatigue characteristics of carbon vinylester composites under seawater exposure [43-46] and kinetic aspects of water absorption for glass polyester composite were also analysed by Camino et al. [47].

Seawater and water's effect on the mechanical properties of fiber-reinforced polymer composites were elaborately discussed [48-51]. since water can diffuse into the composites, this environment can affect the mechanical properties of composites, and the longer duration of the soaking process and the increase in temperature augments the diffusion of water into the composite. The mechanical properties reduction is primarily due to the degradation of the fiber, and matrix, debonding between the interfaces, and plasticization of the matrix resulting from diffusion of water. Different candidate materials of carbon and glass-based composites, were experimentally investigated by Kootsookos et al. [52], which are primarily being used for marine application, and underwent seawater immersion to explore their durability of the same. Towards this, said composite was exposed to a seawater environment for over two years and afterward it was taken out and mechanically characterized. Flexural strength and modulus were degraded notably but it has only generated a slight effect on the interlaminar fracture toughness property. This study derived one more fact, polyester-based composite caused by hydrolysis is relatively less chemically stable than vinyl ester-based composites. It was observed that sizing also plays important role in water absorption, as it will lead to more water absorption in fiberglass-based composites compared to carbon-based composites.

Effect of seawater absorption effect and desorption on the durable creep performance of glass fiber/epoxy composites embedded with graphene oxide, were also analyzed [53-54]. It was found that the modified composite with graphene oxide absorbs lesser seawater than the neat glass/epoxy composite. As it is happening due to the reduction of free volume inside the graphene oxide-modified matrix. It was noticed that epoxy resin is more hygroscopic and it generates hygroscopic stresses at graphene and epoxy interfaces, which has led to interfacial slippage in between, resulting in the degradation of its creep properties faster than neat glass/epoxy.

The seawater effect and warm environments on glass polyurethane and E-glass/epoxy composites, were described [55-56]. This study evaluated the seawater effect and temperature over the structural properties of said composites.

Some of the studies like Chakraverty et al. [57] focussed their study on seawater aging of GFRP composites. In their study, it was observed that the bulky nature of dissolved salts in seawater affects the ingression rate with time and the deposition of these elements on the composite surface

interrupts the diffusion process. Seawater comprises various salt components “K” component maximizes the penetration depth in immersion and “Na” gives the least. Mechanical properties like ILSS, stress and rupture strain, and elastic modulus deteriorated with the increase in immersion period.

The effect of seawater and aging which occurs naturally on the residual strength of epoxy-based laminates reinforced with carbon and glass-woven fabrics, were also investigated [58]. The comparison was also drawn [59] for seawater’s effects on viscoelastic and mechanical properties of epoxy composites with glass, carbon, and their hybrid fabric reinforcements.

The long-time performance of glass fiber with three different resin systems i.e., vinylester, unsaturated polyester and epoxy under seawater exposure, were explored [60-62] and found that glass with vinyl ester resin system is performed well for long time exposure compared to other two resin systems. One of the researchers Pal et al. [63] investigated the effect of artificial seawater ageing over different laminate thickness of glass and carbon fiber with different resin system combination like iso-polyester and vinylester. It was observed that seawater ingression in the case of higher laminate thickness were more compared to reduced thickness one, but time of saturation were same. The laminate with higher thickness were having more residual ILLS and flexural strength compared to lower one. The damage characterization of hybrid composites under seawater exposure were also explored by Arun et al. [64].

The hygrothermal ageing over glass/epoxy composite and were analyzed using DSC and FTIR imaging technique, were also studied [65].

Groysman [66] This work aimed to study the solutions and corrosion problems in the oil, refining, gas, and petrochemical industry. The phenomena and factors affecting the corrosion were discussed. In addition to this, Corrosion control and monitoring methods were illustrated. Even the effect of oil [67] over high-performance concrete’s strength, were analyzed.

Researcher Navneet et al. [68] analyzed the drilling performance of CFRP composites and carried out experimental trials to establish various cutting parameters under cryogenic and dry cooling conditions. And even developed lubrication and cooling technologies to cater to the requirement of machining metal matrix composites and Magnesium [69]. The improvement of cryogenic machining setups for composites and alloys, were also critically reviewed by Chetan et al. [70].

Taking all these researches into account, degradation of E-Glass/ Epoxy composite was analyzed under hostile environments like ATF and seawater.

2.3 Gaps identified from the Literature

It was seen that, most of the studies in this field, were related to the effect of water, seawater, etc. on the mechanical properties of bare E-glass/epoxy composite. Similarly, the available research data to study these effects on E-glass/epoxy composite were limited, and that too which has undergone ATF exposure. Even research studies to cater the effects of ATF and seawater over barrier resin-coated E-glass/epoxy composite were not seen.

Hence this research was carried out for a detailed understanding and enhanced learning of the ATF fuel and seawater exposure effect on the mechanical properties of E-glass/epoxy composite laminates, which is having barrier resin coating. Hence, the following literature gaps were identified from the overall literature survey:

a) It was found that, no investigation reports a study about the ATF exposure on composite laminates having barrier resin coating of different thicknesses. So, there was a need for research on laminates with a barrier coating.
b) It was observed that, research works were also not seen to cater the effect of seawater on composite laminates having barrier resin coating of different thicknesses. So, to address these requirements, research needs to be carried out.
c) There was scant information to study the effect of some of the hostile environments to few of the mechanical properties of E-glass/epoxy laminates, where warp and weft direction were considered during preparation of samples. Hence to exhibit directional significance (warp and weft directions) over significant mechanical properties, research is to be done.
d) The effect of ATF and seawater environments over the E-glass/epoxy laminate made with prepreg were also not seen, as mostly in research papers it was observed that the composite laminates were fabricated using vacuum infusion technique, resin transfer molding (RTM) process or by wetting the fabric with resin using tabletop process.
e) There is a little work on the effect of ATF and seawater on E-glass/epoxy laminate.
f) The effect of epoxy resin as a barrier coat over the E-glass/epoxy laminate and that too with different thicknesses, were not seen.
g) There is limited literature to account the effect of ATF and seawater exposure on interlaminar and in-plane shear properties of E-glass/epoxy laminates.

2.4 Objective of the Research

The objectives to research work were framed based on extensive literature survey, which is as follows.

i) Evaluation of the virgin material (as-received) properties and correlate the same with their respective properties after ATF and seawater exposure, to quantify the degradation.

ii) To study the "Effect of Aviation Turbine Fuel (ATF) exposure on mechanical properties of E-glass/epoxy composite."
   - Tensile Properties (Warp and Weft)
   - Compressive Properties (Warp and Weft)
   - Flexural Properties (Warp and Weft)
   - Interlaminar Shear Properties
   - In-plane shear properties

iii) To study the "Effect of Seawater exposure on mechanical properties of E-glass/epoxy composite."
   - Tensile Properties (Warp and Weft)
   - Compressive Properties (Warp and Weft)
   - Flexural Properties (Warp and Weft)
   - Interlaminar Shear Properties
   - In-plane shear properties

iv) To elucidate the degradation mechanism of E-glass/epoxy composite after ATF and seawater exposure using:
   - Stereo Microscope
   - Microstructural Examination using SEM

v) Comparative study and % of degradation of E-glass/epoxy composite under ATF and Seawater exposure.

vi) To establish the coating thickness of resin coating for minimizing the ATF and seawater exposure effect by using least squares quadratic polynomial approximation.

2.5 Research plan

The roadmap of the research work was framed in the form of a flow chart as shown in Fig. 2.1, to get a better idea of the research work.

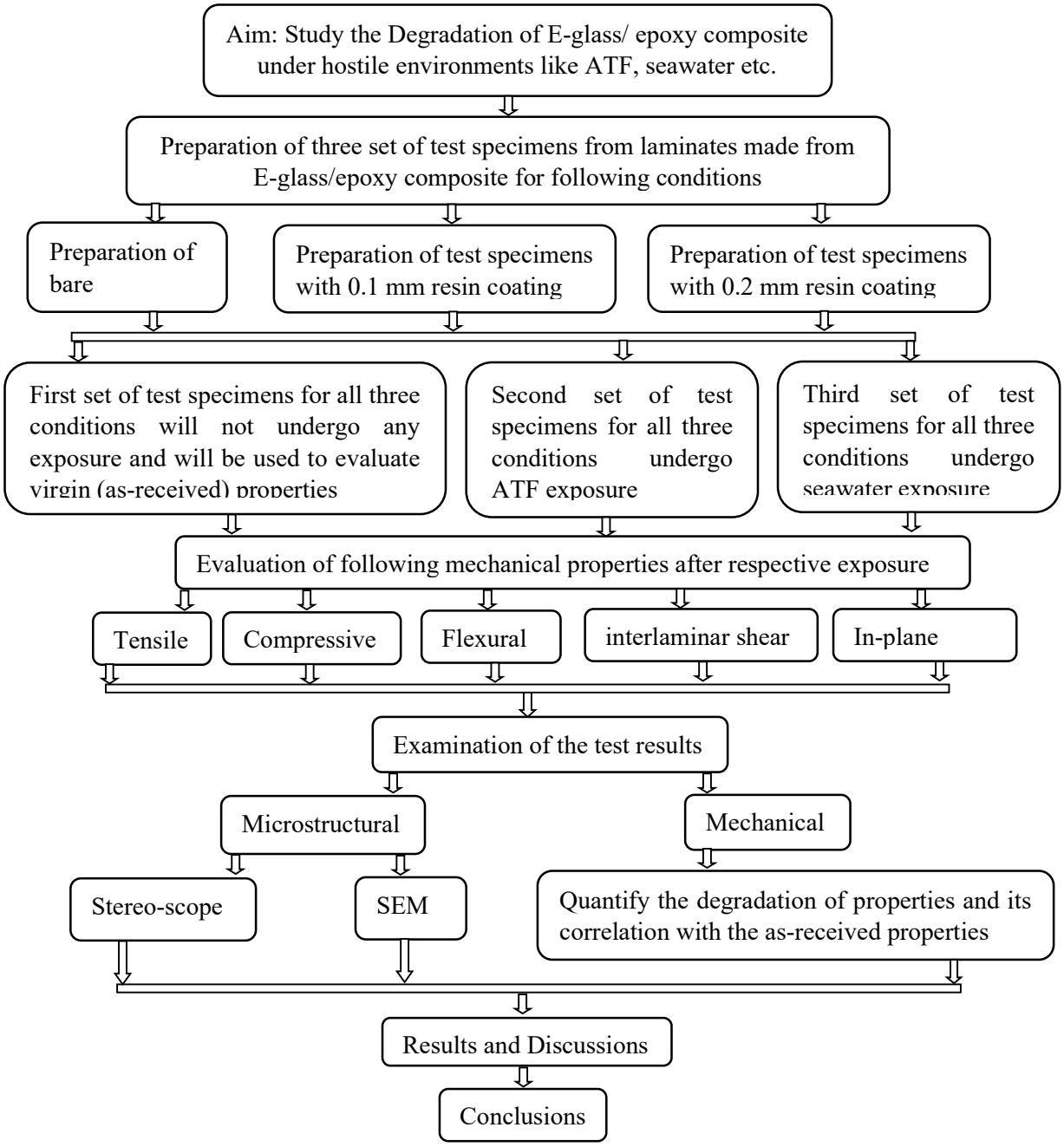


Fig. 2.1 Schematic diagram of research work

2.6 Summary

This chapter dealt with the outcome of extensive literature survey of different types of hostile environments over different types of composites and identified the gaps from the literature, to study the effect of ATF and seawater exposure on E-glass/epoxy composite. The objective of the research work was formulated and exhibited the schematic diagram of overall flow chart of the research plan.

Chapter 3

Material and Methods

3.1 Introduction

In recent times, increasing and widespread usage of composite has become a necessity for aerospace, automobiles, and marine application due to its high specific strength and stiffness and it has attracted not only the necessity of retaining its mechanical properties under exposure to hostile environments but encouraged to explore the possibilities of barrier resin coating to reduce this degradation. This study examined the ATF/Seawater exposure effect on the mechanical properties of E-glass/epoxy composite. The two varieties of test specimens namely bare and barrier resin-coated specimens were made as per the ASTM standard out of E-glass/epoxy composite laminate to evaluate its mechanical parameters and barrier resin-coated specimens were coated with the resin of varying thicknesses. These all types of specimens were immersed inside the aviation turbine fuel for two months and then afterward their effect on the reduction of mechanical properties was experimentally investigated. These all type of specimens was

immersed inside the ATF/Seawater and their effect on the degradation of mechanical properties was experimentally investigated.

This chapter will establish the as-received property for E-glass/epoxy composite, which will be used as a reference property for comparison to analyze the effect of ATF/Seawater exposure. Towards this investigation, as-received properties for mechanical properties like tensile, compressive, Flexural, interlaminar, and in-plane shear properties were identified and the test specimens for evaluation of these properties were made as per respective ASTM standards.

3.2 Materials and Methodology

3.2.1 Materials

The effect of ATF/Seawater effect over E-glass/epoxy composite was analyzed under this investigation. The candidate material chosen for this study was E-glass/epoxy prepreg which was used to make laminate, with the following properties as shown in Table 3.1.

Table 3.1 Pre-preg properties

Sl. no.	Testing parameters	Specified values
1.	Areal density of pre-preg, GSM	450
2.	Volatile content (% weight)	<1%
3.	Resin content (% weight)	37
4.	Fabric content (% weight)	63
5.	Areal density of the fabric, GSM	300
6.	Prepreg Thickness (mm)	0.23
7.	Tackiness	Medium tack condition
8.	Glass Type	E-Glass
9.	Resin	Epoxy

3.2.2 Laminate Preparation and Curing

As E-glass/epoxy prepreg is being used as a candidate material for laminate fabrication, which is having less than 1% volatile content and a zero-bleeding system, hence porosity levels in the test

specimens made by this prepreg will be extremely low. As the as-received property is to be established for tensile, compressive, flexural, interlaminar, and in-plane shear properties, to make the laminate for these properties evaluation, the flat molding plate was taken. The metallic flat plate was used to stack the required number of layers in the desired direction and the requisite laminates were made as per respective ASTM standards as mentioned in the Table 3.2.

Table 3.2 Applicable ASTM standards to test the specimens

Name of the Test	ASTM Standard
Tensile Strength and Modulus (Warp and Weft)	ASTM-D-3039
Compressive Strength and Modulus (Warp and Weft)	ASTM-D-3410
Flexural Strength and Modulus (Warp and Weft)	ASTM-D-790
Interlaminar Shear Strength	ASTM-D-2344
In-plane Shear Strength	ASTM-D-3518

Before fabricating the laminates, prepreg rolls were taken out from the cold storage chamber and were kept in the clean room to get desired tackiness. After this, requisite sizes of prepreg were cut from the prepreg roll in warp and weft direction as per respective ASTM standards to make requisite laminates. These laminates were fabricated using the contact hand lay-up method. The required number of layers in the desired direction is stacked up on the metallic mold and the laminates were made as per respective ASTM standards as shown in Fig 3.1.

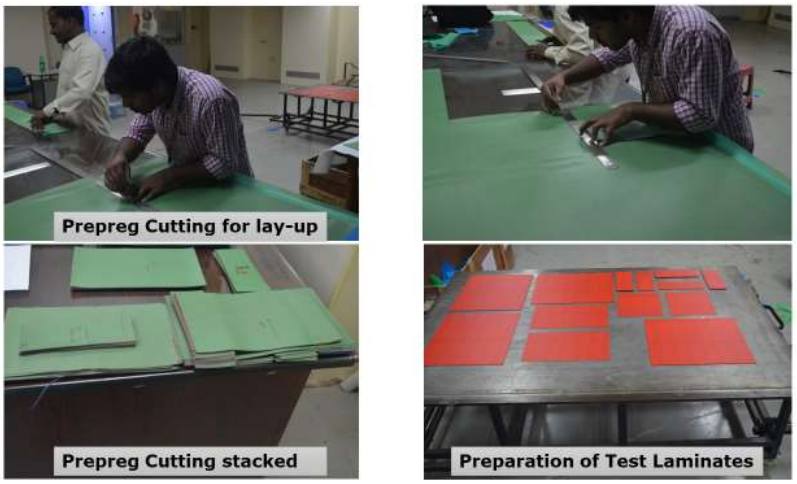


Fig. 3.1 Preparation of test laminates for evaluating the mechanical properties

After making the laminate, it was vacuum bagged, and afterward, it was cured in an autoclave under specified temperature, vacuum, and pressure as shown in Fig. 3.2. A vacuum level of 600 torr was maintained for initial 30 minutes, which was followed by maintaining 150 Torr for the remaining period of curing. It was cured at 135 ^{0}C and a pressure of 3750.31 Torr was maintained during curing.

Fig. 3.2 Curing of test laminates in the autoclave

Once the laminate was cured, the vacuum bagging was removed and respective laminates were extracted from the metallic mold as depicted in Fig. 3.3.

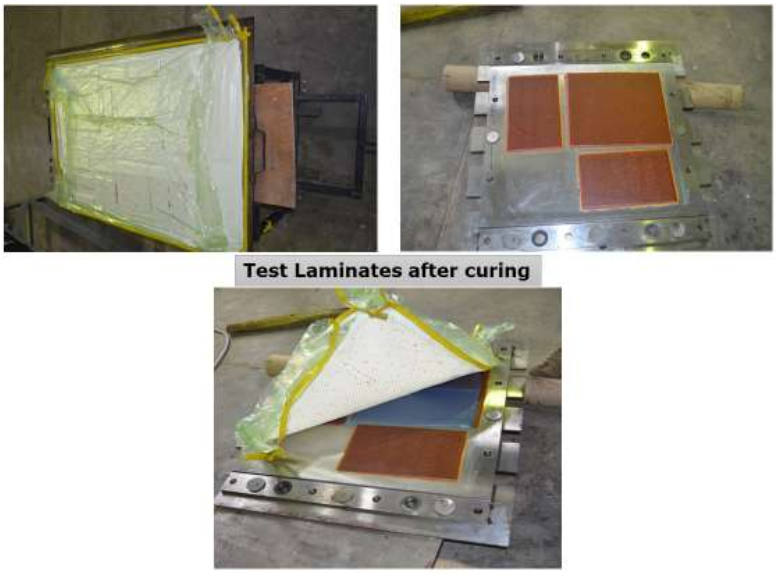


Fig. 3.3 Test laminates after curing

For easy identification of test laminates, all were labelled as per their respective mechanical properties evaluation and it was marked for warp and weft direction laminates as shown in Fig.3.4.

Fig. 3.4 Labelling of Test Laminates

3.2.3 Ultrasonic Testing of Laminate

Once the identification numbers for respective mechanical testing were marked over the test laminates, all the labelled laminates have undergone ultrasonic testing for defect investigation to assess their health or soundness. This test was required to know whether the laminate is free from delamination and other defects or not as shown in Fig. 3.5.

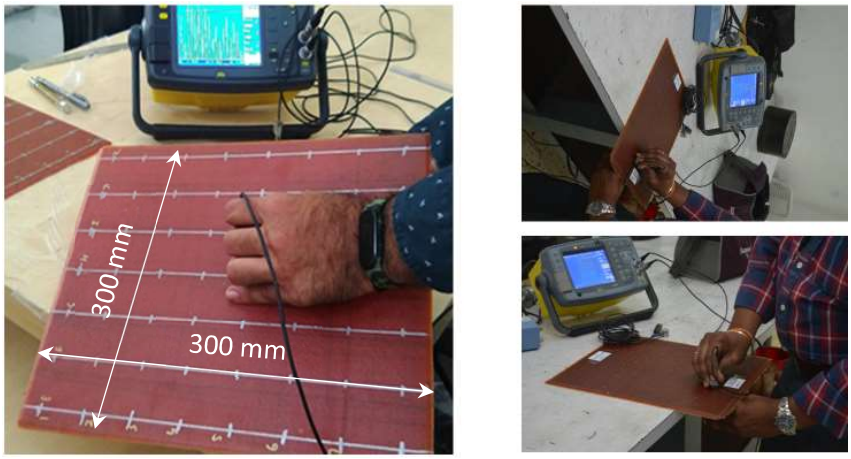


Fig. 3.5 Test laminate undergoing UT test

3.2.4 Cutting of the Test Specimens

The test specimens to evaluate respective mechanical properties were made of the desired size as per ASTM standard requirements. The specimens from laminate were initially cut using the cutting machine as depicted in Fig. 3.6 followed by its finishing cut by a diamond cutter to give edges without fuzzing and delamination. As specimens are to be checked for % of fuel as well as seawater ingression in definite intervals till it got saturated, different traveler coupons for respective tests were also cut and prepared separately for both kinds of exposure.

The bare specimens were cut directly out of the laminates and for making the resin-coated specimens, laminates were coated with a brush with a known amount of epoxy resin i.e., AY103 epoxy resin and HY951 hardener (100:10), as per their laminate size to get the requisite thickness. The uniformity of coating thickness was obtained by using the wiper/doctor blade. These blades were used to remove excess resin. After drying the coated resin at room temperature, the resin-coated specimens were cut from laminates; and after cutting, the specimen in the thickness direction was also coated with resin.

Fig. 3.6 Test specimens preparations using the cutting machine

The test specimens altogether were made to evaluate the mechanical properties for three different types namely bare, 0.1 mm, and 0.2 mm resin-coated, to evaluate the as-received property.

3.2.5 Sizes of Test Specimens

Three different sets of test specimens to evaluate the test parameters as mentioned in Table 3.3 were made as per respective ASTM standard, to cater to the requirements of the following needs.

a) To evaluate the virgin material (as-received) properties

b) To know the degradation of E-glass/epoxy composite under ATF exposure

c) To know the degradation of E-glass/epoxy composite under seawater exposure

Bi-directional (BD) fabric of E-glass/epoxy were stacked to make the laminates for different tests, as per its stacking sequence as mentioned below to get the requisite thickness for their respective test specimens.

Table 3.3 Sizes of the test specimens

Name of the Test	Size of the test specimens as per respective ASTM standard (length x width x thickness) in mm	Stacking Sequence
Tensile Strength and Modulus (Warp and Weft)	250 x 25.4 x 2.3	Bi-direction fabric $[0^0]_{10}$
Compressive Strength and Modulus (Warp and Weft)	140 x 25 x 2.53	Bi-direction fabric $[0^0]_{11}$
Flexural Strength and Modulus (Warp and Weft)	52 x 12.7 x 2.3	Bi-direction fabric $[0^0]_{10}$
In-plane Shear Strength	250 x 25 x 1.84	Bi-direction fabric $[\pm 45^0]_{2s}$
Interlaminar Shear Strength	40 x 10 x 4.14	Bi-direction fabric $[0^0]_{18}$

Sizes of the test specimens are shown in Fig. 3.7.

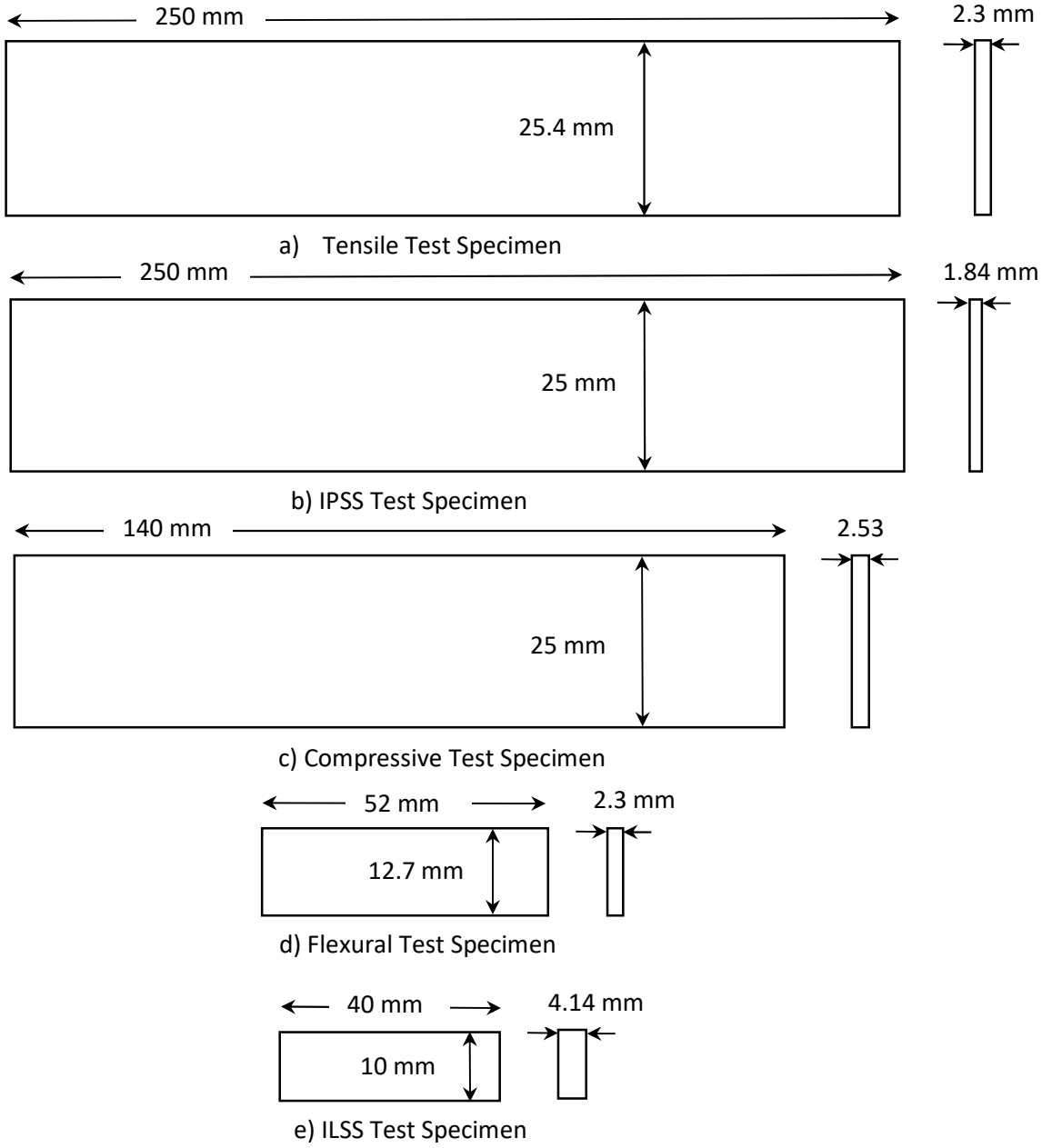


Fig. 3.7 Sizes of the Test specimens

3.3. Evaluation of Virgin Material (As-received) Properties

The following properties are to be evaluated for virgin material properties (as received). Towards this, the preparation of laminates to evaluate the following properties was carried out. As glass fabric is one kind of woven fabric which is having bi-directional fibers, hence fiber in the

lengthwise direction will be warp and transverse to this or in width direction will be named as weft direction fiber. So, for making the warp laminates all 0^0 plies of the prepreg are to be aligned lengthwise and for weft, all the transverse plies are to be aligned lengthwise

- Tensile Properties (Warp and Weft)
- Compressive Properties (Warp and Weft)
- Flexural Properties (Warp and Weft)
- Interlaminar Shear Properties
- In-plane Shear Properties

3.3.1 Tensile Properties

To evaluate the as-received property (without any ATF/seawater exposure) of tensile test in warp and weft direction, test specimens were made as shown in Fig. 3.8 with strain gauge bonding and testing was conducted using a servo mechanical universal testing machine as per ASTM D 3039 standard [71] as shown in Fig. 3.9. The crosshead speed during the test was 2 mm/min at room temperature. Strain gauges were used for strain measurement. Tensile strength was evaluated from the ultimate load and tensile modulus was determined from the stress-strain curve in the linear portion.

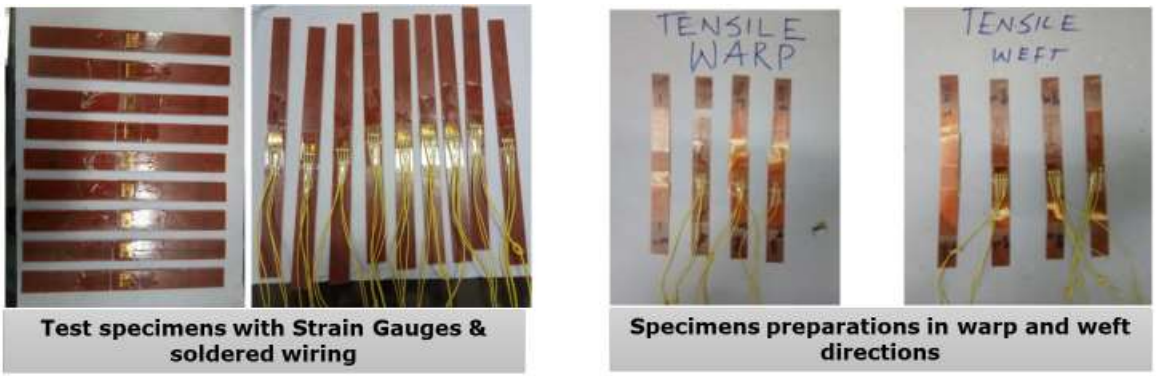


Fig. 3.8 Material characterization of test specimens for evaluation of as-received (virgin-without any exposure) properties

Make: ADMET UTM, USA

Model: 2556

Capacity: 100 kN

Speed Range: 0-550 mm/min

Load cell Accuracy: 0.5 % of full scale

Fig. 3.9 Test specimen undergoing tensile test

The test specimens were 250 mm long and tensile stress can be calculated as follows.

$$\sigma_t = P_t / b_t t_t \quad (3.1)$$

Where,

σ_t- Tensile stress (MPa)

P_t- load taken by test specimen (N)

b_t- width of the test specimen (mm)

t_t-thickness of the test specimen (mm)

Tensile strength and modulus of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens were determined in the warp direction and obtained a mean value of 380.32 MPa and 23.85 GPa, respectively. The test results of test specimens in warp direction are tabulated in Table 3.4 as follows.

Table 3.4 Tensile test results (warp direction) of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. Displacement (mm)	Max. load (k N)	Tensile Strength (MPa)	Tensile Modulus (GPa)
1	TS-WE-Warp-01	25.27 x 1.98	5.853	17.913	358	23.73
2	TS-WE-Warp-02	25.63 x 1.97	6.896	17.759	352	-
3	TS-WE-Warp-03	25.12 x 1.98	6.965	19.436	390.8	23.7

4	TS-WE-Warp-04	24.90 x 1.98	7.215	20.253	410.8	24.51
5	TS-WE-Warp-05	25.35 x 2.00	6.256	18.838	371.6	23.19
6	TS-WE-Warp-06	25.22 x 1.97	7.406	19.81	398.7	24.12
Average value					380.32	23.85
Standard Deviation (SD)					23.46	0.5

Tensile strength and modulus of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens were determined in the weft direction and obtained a mean value of 364 MPa and 22.16 GPa, respectively. The test results of test specimens in the weft direction are tabulated in Table 3.5 as follows.

Table 3.5 Tensile test results (weft direction) of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. Displacement (mm)	Max. load (k N)	Tensile Strength (MPa)	Tensile Modulus (GPa)
1	TS-WE-Weft-01	25.02 x 2.16	5.776	18.53	343	21.11
2	TS-WE-Weft-02	25.02 x 2.01	6.209	19.06	379	23.08
3	TS-WE-Weft-03	25.43 x 1.98	6.881	17.67	351	21.18
4	TS-WE-Weft-04	25.32 x 2.00	6.237	18.642	368	22.29
5	TS-WE-Weft-05	25.14 x 1.98	5.822	17.721	356	21.74
6	TS-WE-Weft-06	24.73 x 2.01	6.345	19.241	387	23.55
Average value					364	22.16
Standard Deviation (SD)					16.99	1

The tested samples of the tensile test (warp and weft), without giving any exposure is as depicted in Fig 3.10.

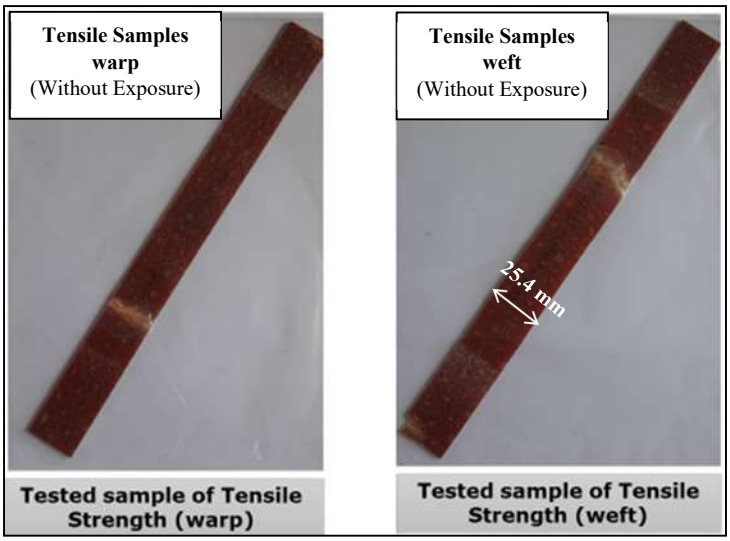


Fig. 3.10 Fractured specimen after tensile test (warp and weft direction)

3.3.2 Compressive Properties

To evaluate the as-received property (without any ATF/seawater exposure) of compressive test samples in warp and weft direction testing was conducted using a servo mechanical universal testing machine as per ASTM D 3410 standard [72] as shown in Fig. 3.11. The crosshead speed during the test was 1.27 mm/min at room temperature. The compressive properties of composites are generally determined by compressing flat or bar specimens. In the case of unidirectional bar specimens, loaded in fiber direction premature failure may occur by "localized brooming" at the ends. In the case of flat specimens, "specimen buckling" may take place. A successful method to eliminate these problems is to bond taps at the end of the specimens and by compressing the specimens using serrated wedge grips. Such a test method is given in the test standard ASTM D 3410 which has been adopted here.

Make: ADMET UTM, USA

Model: 2556

Capacity: 100 kN

Speed Range: 0-550 mm/min

Load cell Accuracy: 0.5 % of full scale

Fig. 3.11 Test specimen undergoing compression test

The test specimens were 140 mm long and compressive stress can be calculated as follows.

$$\sigma_c = P_c / b_c t_c \quad (3.2)$$

Where,

σ_c- Compressive stress (MPa)

P_c- load taken by test specimen (N)

b_c- width of the test specimen (mm)

t_c-thickness of the test specimen (mm)

The compressive strength was determined from the ultimate load and compression modulus was calculated from the stress-strain curve in the linear portion.

Compressive strength and modulus of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens were determined in the warp direction and obtained a mean value of 368.46 MPa and 24.38 GPa respectively as shown in Table 3.6.

Table 3.6 Compressive test results (warp direction) of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. displacement (mm)	Max. load (k N)	Compressive Strength (MPa)	Compressive Modulus (GPa)
1	CS-WE-Warp-01	25.27 x 2.43	4.541	23.143	376.89	25.62
2	CS-WE-Warp-02	25.17 x 2.46	4.517	23.038	372.08	24.91
3	CS-WE-Warp-03	25.14 x 2.52	4.419	22.654	357.58	23.02
4	CS-WE-Warp-04	24.74 x 2.45	4.148	22.262	367.28	23.97
Average value					368.46	24.38
Standard Deviation (SD)					8.24	1.13

Compressive strength and modulus of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens were determined in the weft direction and obtained a mean value of 347.83 MPa and 24.06 GPa respectively as tabulated in Table 3.7.

Table 3.7 Compressive test results (weft direction) of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. displacement (mm)	Max. load (k N)	Compressive Strength (MPa)	Compressive Modulus (GPa)
1	CS-WE-Weft-01	25.14 x 2.52	4.450	22.51	355.37	24.02
2	CS-WE-Weft-02	25.22 x 2.49	4.114	20.81	331.42	23.04
3	CS-WE-Weft-03	25.19 x 2.41	4.063	20.77	342.21	23.75
4	CS-WE-Weft-04	25.21 x 2.38	4.367	21.74	362.32	25.43
Average value					347.83	24.06
Standard Deviation (SD)					13.76	1.00

The tested samples of compressive test (warp and weft), without giving any exposure is as shown in Fig.3.12.

Fig. 3.12 Fractured specimen after compression test

3.3.3 Flexural Properties

To evaluate the as-received property (without any ATF/seawater exposure) of flexural test samples in warp and weft direction testing was conducted using a servo mechanical universal testing machine as per ASTM D 790 standard [73]. The crosshead speed during the test was 1 mm/min at room temperature. The direction of the material under investigation is to be oriented along the lengthwise test specimen's dimension. The test specimens were 52 mm long. The test specimens need a span length/depth (l/d) ratio sufficient to decimate the effect of deformation caused by inter-laminar shear and to exhibit bending failure rather than failure in shear.

When a test specimen is tested in flexure as a simple beam supported at two points as shown in Figure 3.13 and loaded at the midpoint, the maximum stress in the outer surface of the test specimen occurs at the midpoint.

Make: ADMET UTM, USA

Model: 2556

Capacity: 10 kN

Speed Range: 0-550 mm/min

Load cell Accuracy: 0.5 % of full scale

Fig. 3.13 Test specimen undergoing flexural test

This stress may be calculated for any point on the load-deflection curve using the following equation [73]:

$$\sigma_f = 3P_fL/2b_fd_f^2 \quad (3.3)$$

The modulus of elasticity will be calculated as follows

$$E = L^3m/4b_fd_f^3 \quad (3.4)$$

Where:

σ_f: Stress in the outer fibers at the midpoint (MPa)

E: Modulus of elasticity in bending (GPa)

P_f: load at a given point on the load-deflection curve (N)

L: length of specimen (mm)

b_f: width of the test specimen (mm)

d_f: depth of test specimen (mm)

m: the slope of the tangent to the initial straight portion of the load-deflection curve(N/mm)

Flexural strength and modulus of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens were determined in the warp direction and obtained a mean value of 670.75 MPa and 23.48 GPa respectively as shown in Table 3.8.

Table 3.8 Flexural test results (warp direction) of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. Displacement (mm)	Max. load (k N)	Flexural Strength (MPa)	Flexural Modulus (GPa)
1	FS-WE-Warp-01	12.68 x 2.00	2.393	0.633	636	23.86
2	FS-WE-Warp-02	-	-	Slipped	-	-
3	FS-WE-Warp-03	12.62 x 2.01	2.535	0.664	664	23.28
4	FS-WE-Warp-04	12.65 x 2.01	2.569	0.696	694	24.46
5	FS-WE-Warp-05	12.68 x 2.03	2.55	0.706	689	22.34
Average value					670.75	23.48
Standard Deviation (SD)					26.62	0.9

Flexural strength and modulus of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens were determined in the weft direction and obtained a mean value of 619.5 MPa and 22.61 GPa respectively as shown in Table 3.9.

Table 3.9 Flexural test results (weft direction) of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. Displacement (mm)	Max. load (k N)	Flexural Strength (MPa)	Flexural Modulus (GPa)
1	FS-WE-Weft-01	-	-	Slipped	-	-
2	FS-WE-Weft-02	12.64 x 2.10	2.398	0.696	637	22.81
3	FS-WE-Weft-03	12.69 x 2.00	2.33	0.616	619	22.67
4	FS-WE-Weft-04	12.72 x 1.99	2.641	0.592	599	22.28
5	FS-WE-Weft-05	12.67 x 2.00	2.345	0.619	623	22.68
Average value					619.5	22.61
Standard Deviation (SD)					15.7	0.23

The tested samples of the flexural test (warp and weft), without giving any exposure is as depicted in Fig. 3.14.

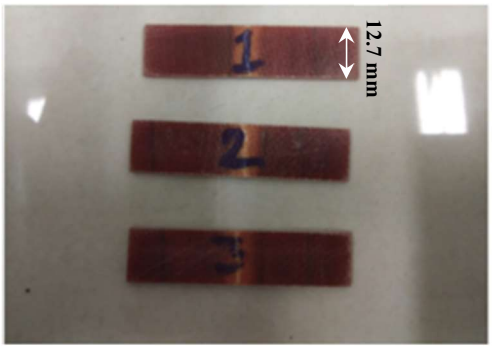


Fig. 3.14 Fractured specimen after flexural test

3.3.4 Interlaminar Properties

To establish the as-received property (without any ATF/seawater exposure) of interlaminar shear strength (ILSS) test samples testing was conducted using a servo mechanical universal testing machine as per ASTM D 2344 standard [74]. The dimensions of the specimens were maintained as per ASTM requirements i.e., 40 mm long x 12 mm wide x 4.14 mm thick. The interlaminar shear strength (ILSS) is determined using a fixture of the three-point test as shown in Fig. 3.15. The material direction which will undergo investigation shall be oriented along the lengthwise dimension of the test specimen. The span/depth ratio of the test pieces shall be kept low enough for minimizing the effect of bending deformation, resulting in shear failure rather than bending.

Make: ADMET UTM, USA

Model: 2556

Capacity: 10 kN

Speed Range: 0-550 mm/min

Load cell Accuracy: 0.5 % of full scale

Fig. 3.15 Test specimen undergoing ILSS test

The test specimens were 40 mm long and the interlaminar shear strength (ILSS) is determined as follows [74].

$$\tau_s = {}^{3P_s}/_{4b_s d_s} \quad (3.5)$$

Where:

τ_s: Interlaminar shear strength (MPa)

P_s: Maximum load observed during the test (N)

b_s: width of the specimen (mm)

d_s: the thickness of the specimen (mm)

Interlaminar shear strength of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens was determined and obtained a mean value of 68.21 MPa as shown in Table 3.10.

Table 3.10 ILSS test result of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. Displacement (mm)	Max. load (k N)	ILSS Strength (MPa)
1	ILSS-WE-01	11.89 x 4.03	0.812	4.547	71.12
2	ILSS-WE-02	11.93 x 4.13	0.801	4.289	65.29
3	ILSS-WE-03	12.01 x 4.18	0.742	4.297	64.19
4	ILSS-WE-04	12.07 x 4.08	0.854	4.787	72.91
5	ILSS-WE-05	12.12 x 4.04	0.787	4.408	67.52
Average value					68.21
Standard Deviation (SD)					3.73

The tested samples of the interlaminar shear test, without giving any exposure is as depicted in Fig. 3.16.

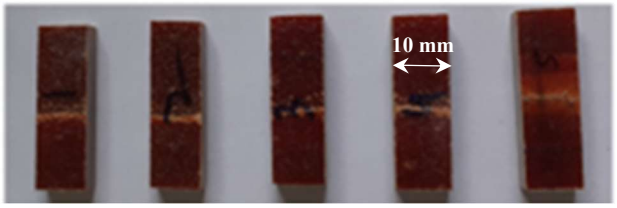


Fig. 3.16 Fractured specimen after ILSS test

3.3.5 In-plane Shear Properties

To establish the as-received property (without any ATF/seawater exposure) of in-plane shear (IPSS) test samples testing was conducted using a servo mechanical testing machine according to ASTM D 3518 standard [75]. The dimensions of the specimens were maintained as per ASTM requirements i.e., 250 mm long x 25 mm wide x 2.76 mm thick.

The laminate was prepared as shown in Fig. 3.17, using the stacking sequence of $+45^0/-45^0$. The significance of choosing this stacking sequence is such that the laminate is specially orthotropic and the coupling effect generated due to bending stretching and anisotropic effects of in-plane bending are avoided.

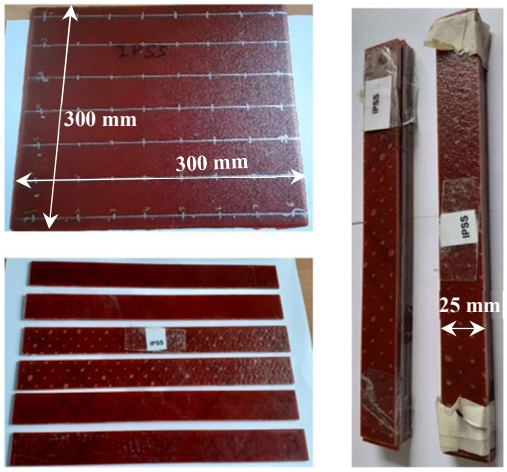


Fig. 3.17 IPSS laminate and test specimens (before testing)

The in-plane shear strength (IPSS) and modulus are calculated by this test. A uniaxial tension test on $+45^0/-45^0$ test specimens was conducted as shown in Fig. 3.18, to evaluate the in-plane shear properties. The test specimen will undergo that kind of load which will only generate a pure shear stress state and the corresponding strain is recorded.

Make: ADMET UTM, USA

Model: 2556

Capacity: 100 kN

Speed Range: 0-550 mm/min

Load cell Accuracy: 0.5 % of full scale

Fig. 3.18 Test specimen undergoing IPSS test

Rosette strain gauges are used on testing specimens to record strains along the direction of loading and perpendicular to the loading direction. The in-plane shear modulus is determined from the curve of stress-strain.

The test specimens were 250 mm long and in-plane shear stress is computed by using the formula as follows. [75]

$$\tau_{12} = {}^{P_i}/_{2A} \tag{3.6}$$

Shear strain is computed with the help of the following reaction.

$$\gamma_{12} = \varepsilon_x - \varepsilon_y \tag{3.7}$$

Where:

τ_{12}: Maximum in-plane shear stress (MPa)

γ_{12}: Maximum Shear Strain (με)

P_i: Applied load (N)

A: Area of cross section (mm^2)

ε_x: normal strain in the longitudinal direction (με)

ε_y: normal strain in the lateral direction (με)

In-plane shear strength and modulus of as-received (without any ATF/seawater exposure) E-glass/epoxy test specimens were determined and obtained a mean value of 99.12 MPa and 4.14 GPa respectively as shown in Table 3.11.

Table 3.11 IPSS test results of as-received specimens (without any exposure)

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. Displacement (mm)	Max. load (k N)	IPSS Strength (MPa)	In-plane Modulus (GPa)
1	IPSS-WE-01	25.54 x 1.81	23.978	9.708	105	4.48
2	IPSS-WE-02	25.48 x 1.74	23.398	9.133	103	4.35
3	IPSS-WE-03	25.47 x 1.82	22.063	9.364	101	4.37
4	IPSS-WE-04	25.48 x 1.82	22.511	9.646	104	4.33
5	IPSS-WE-05	25.60 x 1.77	21.245	9.062	100	4.31
6	IPSS-WE-06	24.92 x 1.80	30.477	7.805	87	slipped

7	IPSS-WE-07	25.35 x 1.78	27.656	8.664	96	3.54
8	IPSS-WE-08	25.02 x 1.79	29.360	8.688	97	3.63
Average value					99.12	4.14
Standard Deviation					5.84	0.39

The tested samples of the in-plane shear test, without giving any exposure is as depicted in Fig. 3.19.

Fig. 3.19 Fractured specimen after IPSS test

3.4 Summary

This chapter discusses the preparation of test laminates, curing and its extraction from the molding tool. In addition to this, cutting methodology of the test specimens from respective laminates as per respective ASTM standards for various tests, using cutting machines were also explained. Evaluation of the virgin material (as-received) properties of E-glass/epoxy composite i.e., tensile, compressive, flexural, interlaminar and in-plane shear properties, were also elaborated.

Chapter 4

Aviation Turbine Fuel (ATF) Ingression into Glass Fiber Reinforced Epoxy Composite

4.1 Introduction

Modern composites have been in existence for more than half a century; they have found widespread application in almost all industrial sectors and their usage is increasing at a rapid rate. Glass fiber-reinforced epoxy resin composites, a major class of composites, are used and in the manufacture of many aerospace products that demand long service life under structural and environmental loads. In this connection, exposure to ATF is a critical one, as these materials tend to absorb ATF and deteriorate in respect of their mechanical properties. However, most of the reported studies from the researchers used seawater or distilled water to immerse the test specimens, to explore the residual strength of epoxy laminates. The currently available data is limited in respect of E-glass/epoxy composite, which has undergone ATF exposure. Furthermore, there is an inadequate investigation that reports a comparative study about the ATF exposure on composite laminates having barrier resin coating of different thicknesses.

So, there is a need for research on laminates with a barrier coating. Moreover, due to different fill counts of fibers, ATF ingression may not be the same, so this chapter describes the ATF's ingression into E-glass/epoxy composite to explore the directional significance (i.e., warp and weft directions) of mechanical properties like tensile, compressive and flexural towards fuel absorption. In addition to this, ATF ingression to interlaminar and in-plane shear properties were also discussed.

4.2 Exposure to Aviation Turbine Fuel (ATF)

At the outset, the prepared specimens can have some degree of moisture ingression from the environment, which has to be dried up. To quantify the ingression test specimens weight, is to be recorded using weighing balance as shown in Fig. 4.1, which will be followed by de-moisturization by heating in an oven at 110^0 C for 1 hr as depicted in Fig. 4.2.

Fig. 4.1 ILSS and flexural test samples (before de-moisturization)

Fig. 4.2 De-moisturization cycle using hot air oven

4.3 Experimental Description

4.3.1 Effect of Aviation Turbine Fuel Exposure on Tensile Properties of E-glass/epoxy Composite

Tensile test samples of different types were immersed in ATF as shown in Fig. 4.3, for 1464 hours. This test was conducted using a servo mechanical universal testing machine as per ASTM D 3039 standard. Extensometer was used for strain measurement. Tensile strength was evaluated from the ultimate load and tensile modulus was determined from the stress-strain curve in the linear portion.

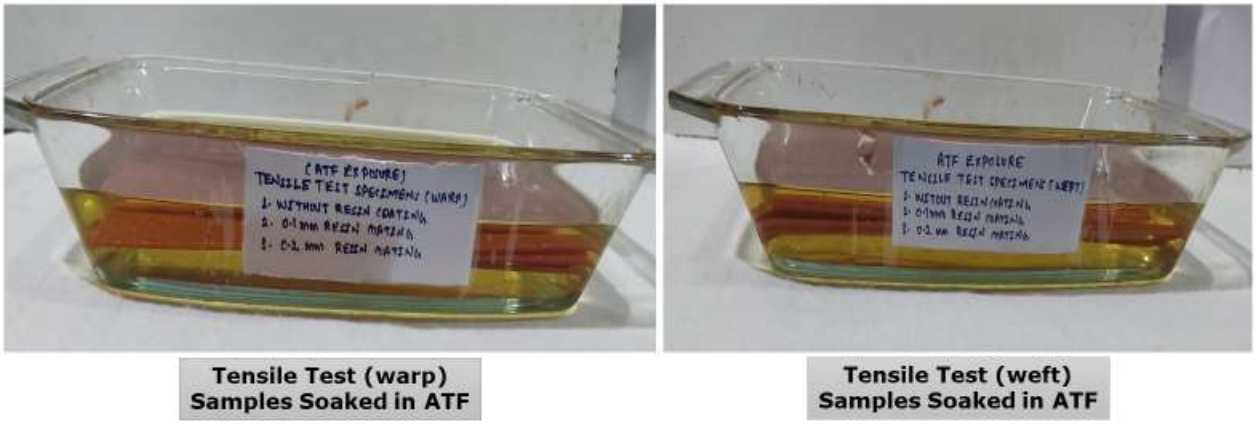


Fig. 4.3 Tensile test samples immersion in Aviation Turbine Fuel (ATF)

Once the test specimens are prepared, they will undergo for de-moisturization cycle to remove initially absorbed moisture from the atmosphere. After the completion of this cycle, all the specimens and traveler coupons were initially weighted before ATF exposure. After this, they were immersed inside the container, which contains the ATF. The traveler coupons were periodically removed, surface dried with a lint-free cloth, and weighed again using the electronic weighing balance of 0.1 mg accuracy, to know the rate and amount of ATF absorption for two months. Initially, the weight changes of the traveler coupons were measured at 24 hrs intervals and afterward interval was increased as the conditioning process continued. The ATF ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Percentage of weight gain of tension test (warp) specimens after ATF exposure is tabulated in Table 4.1. Initial weight of the traveller coupon of tensile test (warp):

a) Without resin coated: 23.511 grams
b) 0.1 mm resin coated: 25.293 grams

c) 0.2 mm resin coated: 27.138 grams

Table 4.1 Percentage of weight gain of tension test (warp) specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.05	0.3403	0.451	0.0807
3	0.25	1.4036	0.79	0.1615
4	1.5	2.2118	0.87	0.2423
5	24	2.6371	0.923	0.2826
6	96	2.8072	1.327	0.7671
7	264	3.1050	3.75	1.4939
8	432	3.7855	4.67	4.5625
9	600	4.1683	4.91	5.047
10	720	4.1683	5.23	5.4104
11	1080	4.1683	5.32	6.3390
12	1104	4.1683	5.32	6.6217
13	1440	4.1683	5.32	8.8020
14	1464	4.1683	5.32	8.8020

For each category of tensile specimens, a minimum of three samples were used for bare, 0.1 mm, and 0.2 mm resin coated and the average weight gain was determined.

The percentage (%) of ATF gain M_t is computed and is represented as a dry weight percentage

$$M_t = \frac{W_t - W_0}{W_0} \text{ X } 100 \qquad (4.1)$$

Where w_0 is the dry specimen weight in grams (before immersion) and w_t is the wet specimen weight at time 't' in grams (after ATF immersion).

The % of weight gain for the tensile test (warp) with respect to time of exposure was plotted as follows (Fig.4.4).

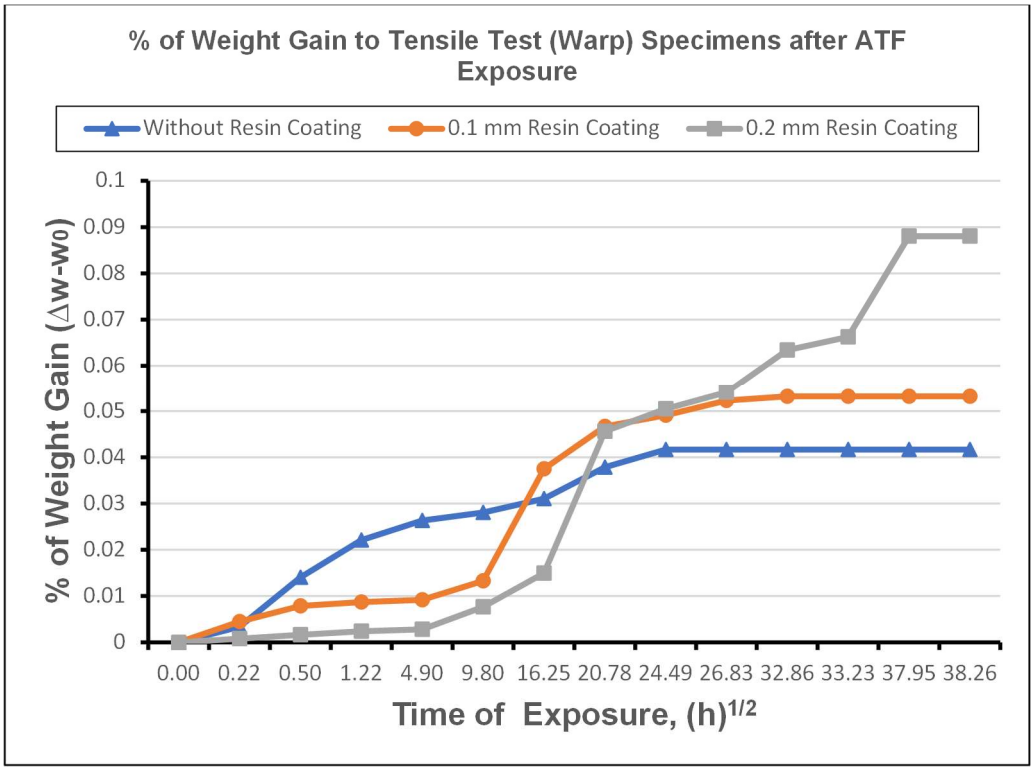


Fig. 4.4 Percentage (%) of weight gain with respect to time for tensile test (warp) specimens after ATF exposure

Similarly, the weight gain of the tension test (weft) is to be recorded as it was done for tensile warp test specimens, after immersing the same in ATF exposure. The traveler coupons will be taken out periodically to measure the percentage (%) of weight gain as follows (Table 4.2).
Initial weight of the traveller coupon of tensile test (weft):

a) Without resin coated: 23.011 grams
b) 0.1 mm resin coated: 24.786 grams
c) 0.2 mm resin coated: 26.624 grams

Table 4.2 Percentage of weight gain of tension test (weft) specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.05	0.4998	0.4056	0.6994
3	0.25	1.166	0.5795	1.6869
4	1.5	1.832	2.98	2.5097
5	24	2.0823	6.01	3.0857
6	96	2.2489	8.353	3.744
7	264	2.7903	12.33	6.5418
8	432	3.3734	12.616	11.1909
9	600	3.7066	12.641	12.6309
10	720	3.7066	12.687	14.1532
11	1080	3.7066	12.71	16.4161
12	1104	3.7066	12.71	17.0744
13	1440	3.7066	12.71	18.7201
14	1464	3.7066	12.71	18.7201

The percentage (%) of weight gain for the tensile test (weft) with respect to time of exposure was plotted as follows (Fig. 4.5).

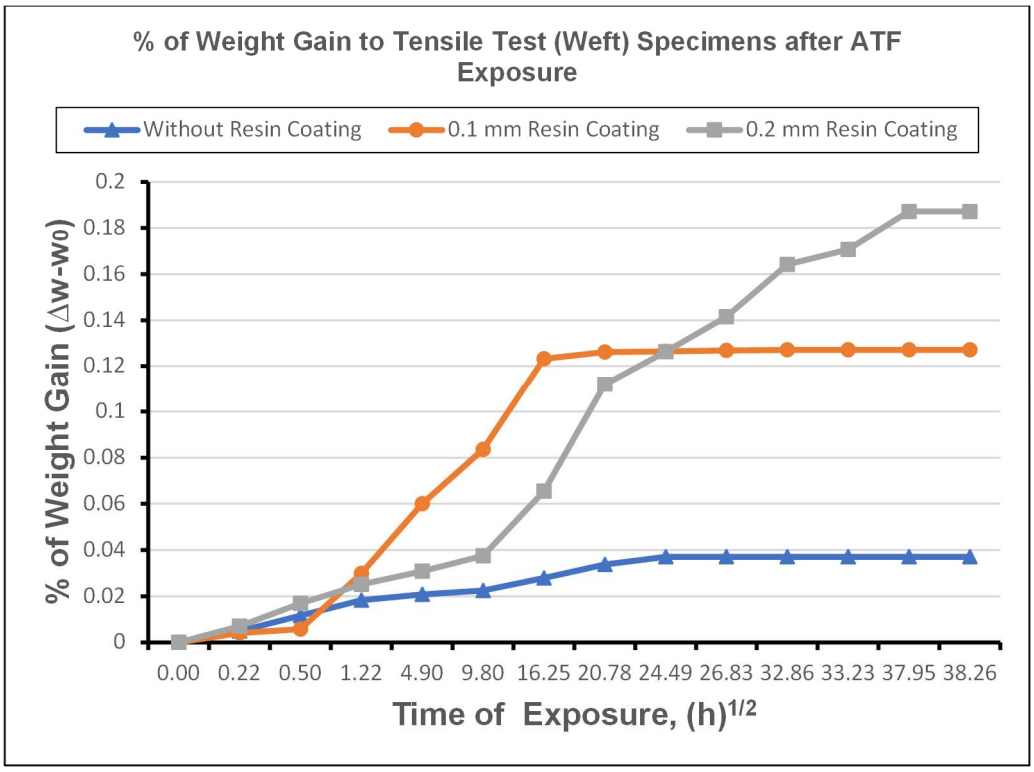


Fig. 4.5 Percentage (%) of weight gain with respect to time for tensile test (weft) specimens after ATF exposure

4.3.2 Effect of Aviation Turbine Fuel Exposure on Compressive Properties of E-glass/epoxy Composite

Compressive test samples of different types as shown in Fig. 4.6, were immersed in ATF for 1464 hours, this test was conducted using a servo mechanical universal testing machine as per ASTM D 3410 standard. Strain gauges were used for strain measurement. Compressive strength was evaluated from the ultimate load and compressive modulus was determined from the stress-strain curve in the linear portion.

Fig. 4.6 Bare samples without tab

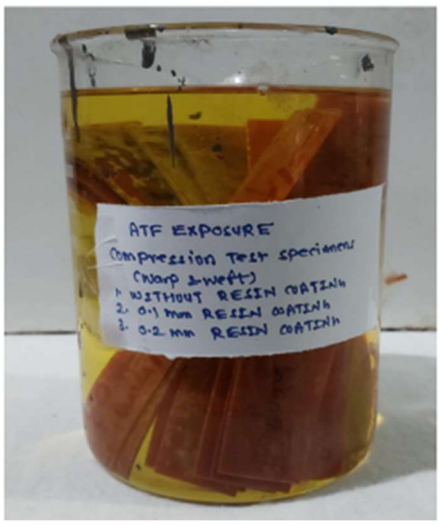


Fig. 4.7 Tested samples of compressive strength (warp and weft) soaked to ATF exposure

Once the test specimens for the compression test are prepared, they will undergo for de-moisturization cycle to remove initially absorbed moisture from the atmosphere. After this, all the specimens and traveler coupons were initially weighted before ATF exposure. After this, they were immersed inside the container, which contains the ATF (Fig. 4.7). The traveler coupons were periodically removed, surface dried with a lint-free cloth, and weighed again using the electronic weighing balance of 0.1 mg accuracy, to know the rate and amount of ATF absorption for two months. The weight changes of the traveler coupons were measured as mentioned in Table 4.3. The ATF ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Percentage of weight gain of test specimens after ATF exposure is tabulated in Table 4.3. Initial weight of the traveller coupon of compressive test (warp) is:

a) Without resin coated: 16.559 grams
b) 0.1 mm resin coated: 17.560 grams
c) 0.2 mm resin coated: 18.596 grams

Table 4.3 Percentage of weight gain of compression test (warp) specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.4872	0.4233	0.7206
3	0.05	0.9135	0.8467	1.32110
4	0.0833	1.4007	1.3305	2.0417
5	0.167	2.3142	1.8144	2.7623
6	0.25	3.3495	2.5401	3.9633
7	0.5	3.5931	3.0239	4.9842
8	1	3.4713	3.5078	5.6447
9	1.5	4.4457	4.3545	6.7256
10	24	4.9938	5.0802	7.4462
11	72	5.7246	6.2898	8.2869
12	240	6.5164	7.3179	8.9475
13	432	8.2215	7.9227	14.892
14	600	9.0742	8.7089	15.8533
15	720	9.0742	9.7976	18.2553
16	1080	9.0742	11.0071	20.2970
17	1104	9.0742	11.0071	21.3179
18	1440	9.0742	11.0071	22.5789
19	1464	9.0742	11.0071	22.5789

The percentage (%) of weight gain for the compressive test (warp) with respect to time of exposure was calculated from the equation 4.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after ATF exposure, which is as follows (Fig. 4.8).

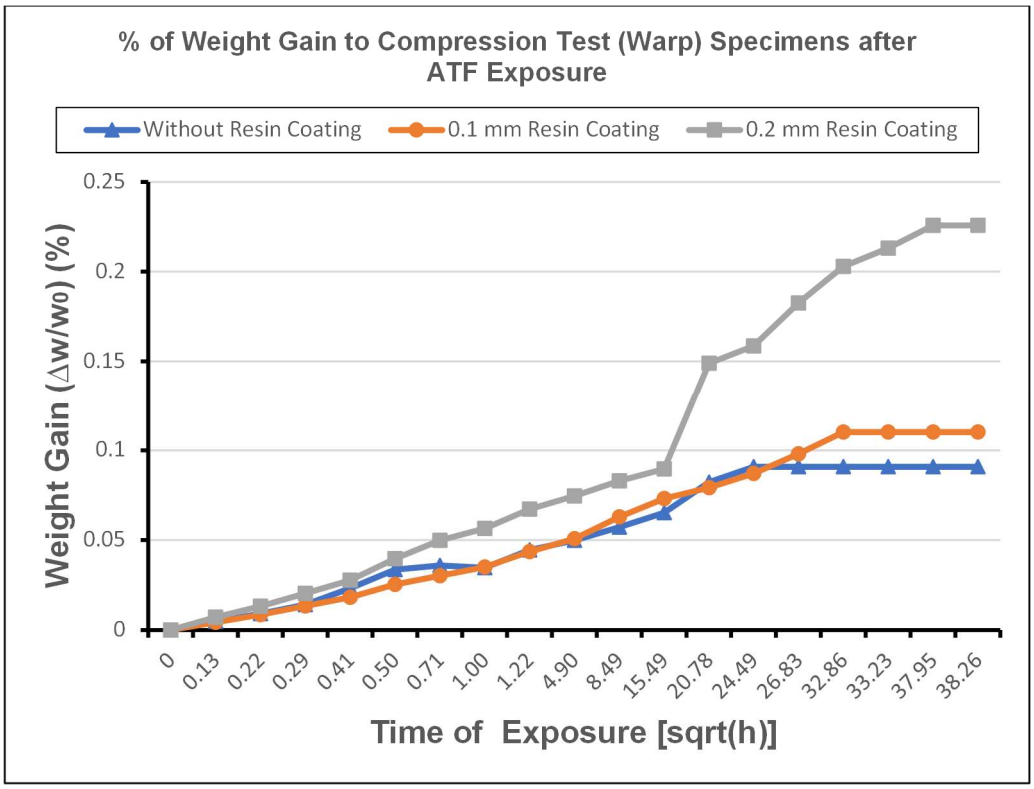


Fig. 4.8 Percentage of weight gain to compressive test (warp) specimens after ATF exposure

Similarly, the weight gain of the compression test (weft) is to be recorded as it was done for compressive warp test specimens, after immersing the same in ATF exposure. The traveler coupons will be taken out periodically to measure the percentage (%) of weight gain as depicted in Table 4.4.

Initial weight of the traveller coupon of compressive test (weft):

a) Without resin coated: 16.401 grams
b) 0.1 mm resin coated: 17.398 grams
c) 0.2 mm resin coated: 18.430 grams

Table 4.4 Percentage of weight gain of compression test (weft) specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.6102	1.0296	1.2632
3	0.05	0.7933	2.4225	1.9249
4	0.0833	1.3425	3.0887	2.8272
5	0.167	1.9527	4.0577	3.6092
6	0.25	2.6849	4.845	4.752
7	0.5	4.0884	5.5718	5.9552
8	1	5.3699	6.2986	6.6770
9	1.5	6.1631	7.9943	7.3387
10	24	6.8344	8.6605	7.6997
11	72	10.1295	10.538	8.0004
12	240	11.7161	12.3549	10.4066
13	432	13.3026	14.1209	13.0533
14	600	14.1570	15.545	14.6775
15	720	14.1569	15.9427	16.9032
16	1080	14.1569	16.1247	18.2265
17	1104	14.1569	16.1247	19.3093
18	1440	14.1569	16.1247	20.8131
19	1464	14.1569	16.1247	20.8131

The percentage (%) of weight gain for the compressive test (weft) with respect to time of exposure was plotted as follows (Fig. 4.9).

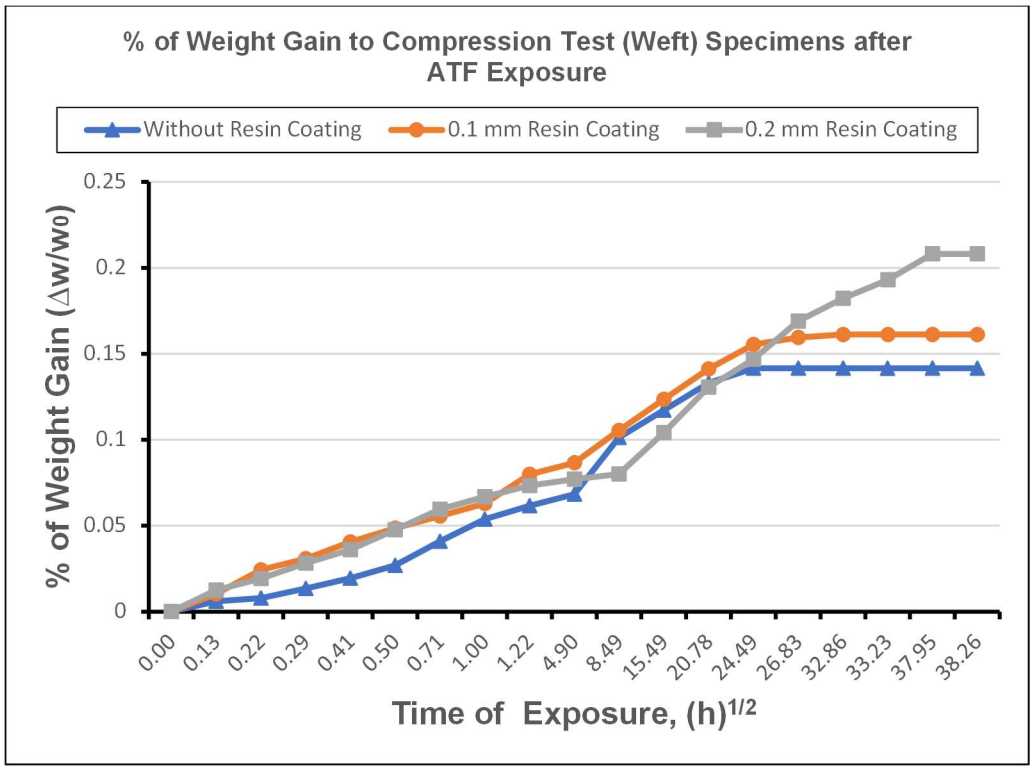


Fig. 4.9 Percentage of weight gain to compressive test (weft) specimens after ATF exposure

4.3.3 Effect of Aviation Turbine Fuel Exposure on Flexural Properties of E-glass/epoxy Composite

Flexural test samples of different types were immersed as shown in Fig. 4.10, in ATF for 1488 hours, this test was conducted using a servo mechanical universal testing machine as per ASTM D 790 standard. Flexural strength was evaluated from the ultimate load and flexural modulus was determined from the stress-strain curve in the linear portion.

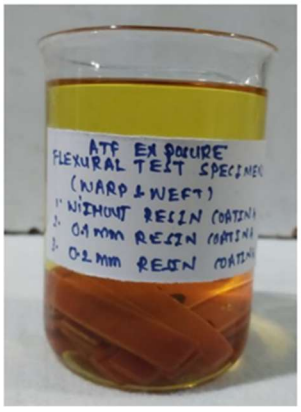


Fig.4.10 Flexural test (warp and weft) samples immersed in ATF

All the de-moisturized flexural test specimens and traveler coupons were initially weighted then followed by ATF exposure. The weight changes of the traveler coupons were measured as mentioned in Table 4.5. The ATF ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Initial weight of the traveller coupon of flexural test (warp):

a) Without resin coated: 2.379 grams
b) 0.1 mm resin coated: 2.587 grams
c) 0.2 mm resin coated: 2.806 grams

Table 4.5 Percentage of weight gain of flexural test (warp) specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.2569	0.1967	0.1264
3	0.05	0.4125	0.2910	0.2548
4	0.1	0.5265	0.4688	0.4347
5	0.2	0.7157	0.5654	0.3476
6	0.4	1.268	0.7642	0.7205
7	0.75	1.5687	1.0237	0.9030
8	1	1.9687	1.2456	1.0385
9	1.5	2.1768	1.6423	1.563
10	24	3.4652	2.2927	2.4856
11	72	5.1567	3.4390	3.8567
12	240	9.3476	5.3496	5.9476
13	576	11.578	10.31716	11.4756
14	1080	12.436	14.2847	16.3474
15	1440	12.436	15.2847	16.7534

16	1464	12.436	15.2847	17.0236
17	1488	12.436	15.2847	17.0236

The percentage (%) of weight gain for the flexural test (warp) with respect to time of exposure was calculated from the equation 4.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after ATF exposure, which is as follows (Fig. 4.11).

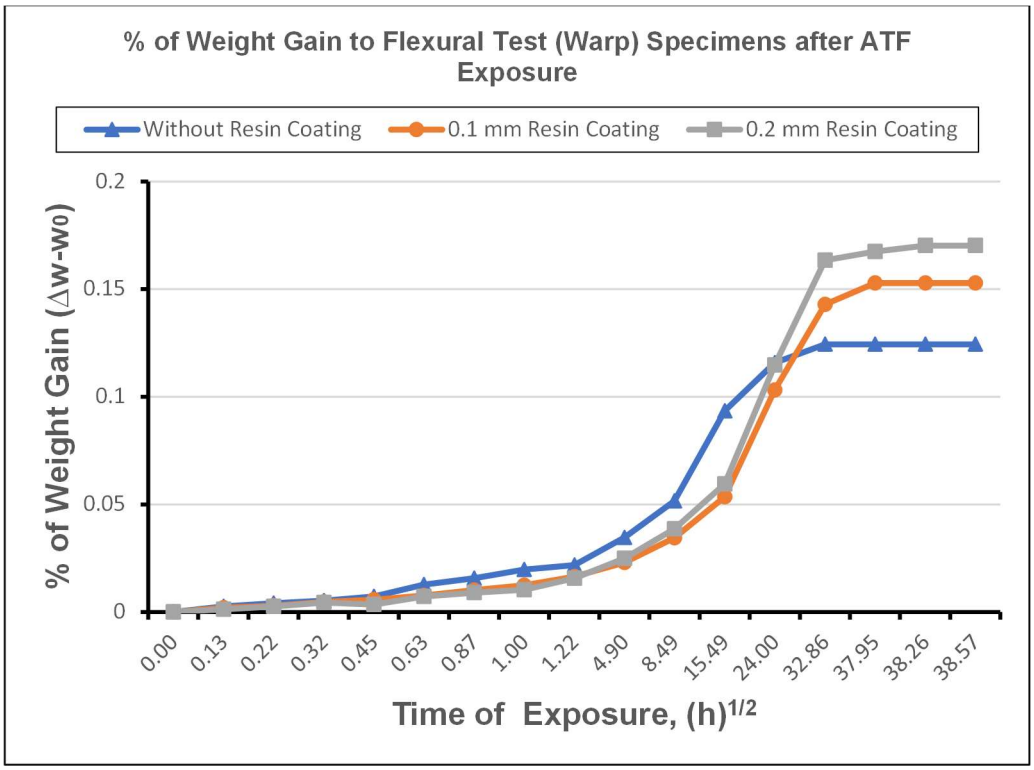


Fig.4.11 Percentage of weight gain to flexural test (warp) specimens after ATF exposure

Similarly, the weight gain of the flexural test (weft) is to be recorded as it was done for flexural warp test specimens, after immersing the same in ATF exposure. The traveler coupons will be taken out periodically to measure the percentage (%) of weight gain as depicted in Table 4.6.

Initial weight of the traveller coupon of flexural test (weft):

a) Without resin coated: 2.367 grams
b) 0.1 mm resin coated: 2.574 grams
c) 0.2 mm resin coated: 2.793 grams

Table 4.6 Percentage of weight gain of flexural test (weft) specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.2785	0.2153	0.1364
3	0.05	0.4265	0.3158	0.2897
4	0.1	0.5459	0.5140	0.4973
5	0.2	0.7587	0.6051	0.5232
6	0.4	1.4796	0.7945	0.8036
7	0.75	1.896	1.4469	0.9328
8	1	2.1867	1.6347	1.284
9	1.5	2.5684	1.9587	1.6036
10	24	3.9674	2.5861	2.785
11	72	5.5877	3.7317	4.893
12	240	12.867	5.9874	6.784
13	576	13.1498	11.6544	9.874
14	1080	13.2475	15.1742	15.198
15	1440	13.2475	15.9437	17.468
16	1464	13.2475	15.9437	18.143
17	1488	13.2475	15.9437	18.157

The percentage (%) of weight gain for the flexural test (weft) with respect to time of exposure was plotted as follows (Fig. 4.12).

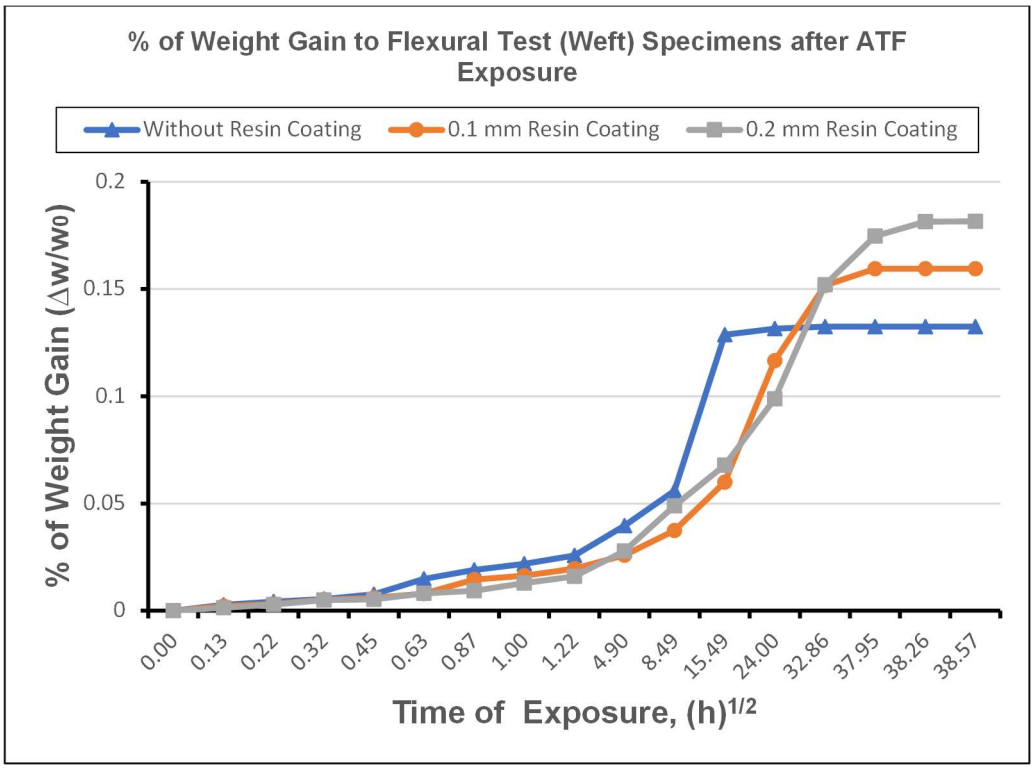


Fig.4.12 Percentage of weight gain to flexural test (weft) specimens after ATF exposure

4.3.4 Effect of Aviation Turbine Fuel Exposure on Interlaminar and In-plane Shear Properties of E-glass/epoxy Composite

Interlaminar shear strength (ILSS) and in-plane shear strength (IPSS) test samples of different types were immersed in ATF as shown in Fig. 4.13 and the samples were given 1488 hours of exposure to this ingression.

ILSS Test Samples

The ILSS test was conducted using a servo mechanical universal testing machine as per ASTM D 2344 standard. The dimensions of the specimens were maintained as per ASTM requirements. The interlaminar shear strength (ILSS) is determined using a fixture of the three-point test. The material direction which will undergo investigation shall be oriented along the lengthwise dimension of the test specimen. The span/depth ratio of the test pieces shall be kept low enough for minimizing the effect of bending deformation, resulting in shear failure rather than bending.

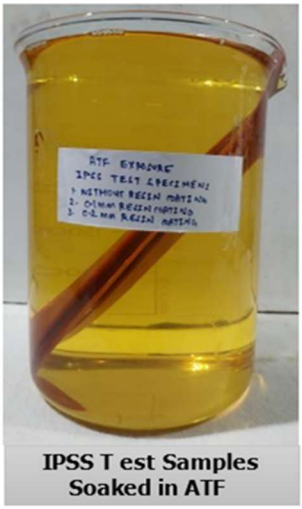


Fig.4.13 ILSS and IPSS samples immersed in ATF

All the de-moisturized ILSS test specimens and traveler coupons were initially weighted then followed by ATF exposure. The weight changes of the traveler coupons were measured as mentioned in Table 4.7. The ATF ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Initial weight of the traveller coupon of ILSS test:

a) Without resin coated: 3.567 grams
b) 0.1 mm resin coated: 3.723 grams
c) 0.2 mm resin coated: 3.888 grams

Table 4.7 Percentage of weight gain of ILSS test specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	4.2052	1.0995	1.9662
3	0.05	4.4891	0.274	2.1572
4	0.1	4.7682	0.374	2.3024
5	0.2	4.8758	0.549	2.6965
6	0.4	4.9489	0.649	2.7976
7	0.75	5.2145	0.824	2.9662

8	1	5.4462	1.099	3.0178
9	1.5	5.7894	1.649	3.6179
10	24	6.4480	3.783	5.4268
11	72	7.642	5.497	6.4268
12	240	8.167	7.421	7.5055
13	576	8.9341	9.345	9.9771
14	1080	9.5672	10.444	10.347
15	1440	9.5672	10.444	10.545
16	1464	9.5672	10.444	10.786
17	1488	9.5672	10.444	10.786

The percentage (%) of weight gain for the ILSS test with respect to time of exposure was calculated from the equation 4.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after ATF exposure, which is as follows (Fig. 4.14).

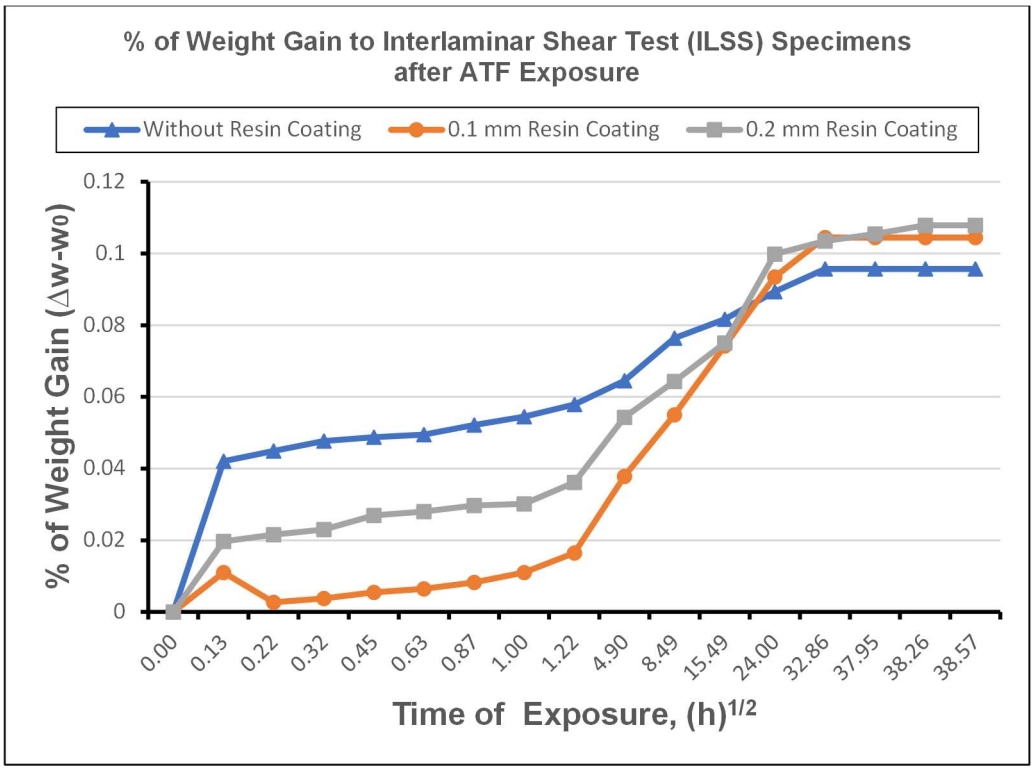


Fig. 4.14 Percentage of weight gain to interlaminar test specimens after ATF exposure

IPSS Test Samples

In-plane shear test samples of different types were immersed in ATF and the samples got exposed for 1464 hours with this ingression, afterward this test was conducted using a servo mechanical testing machine according to ASTM D 3518 standard. The dimensions of the IPSS specimens were maintained as per ASTM requirements.

The laminate was prepared using the stacking sequence of $+45^0/-45^0$. The significance of choosing this stacking sequence is such that the laminate is specially orthotropic and the coupling effect generated due to bending stretching and anisotropic effects of in-plane bending are avoided

All the de-moisturized IPSS test specimens and traveler coupons were initially weighted then followed by ATF exposure. The weight changes of the traveler coupons were measured as mentioned in Table 4.8. The ATF ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Initial weight of the traveller coupon of IPSS test:

a) Without resin coated: 18.921 grams
b) 0.1 mm resin coated: 20.646 grams
c) 0.2 mm resin coated: 22.431 grams

Table 4.8 Percentage of weight gain of IPSS test specimens after ATF exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.8456	0.8534	0.8348
3	0.05	1.5221	1.6876	1.5026
4	0.0833	2.1141	2.5943	2.3373
5	0.167	3.2980	3.8682	2.9217
6	0.25	3.7631	4.9849	3.506
7	0.5	4.4819	6.3871	3.9234
8	1	5.1584	7.1854	4.5912

9	1.5	6.0463	8.5673	5.8851
10	24	6.8073	10.3954	6.8451
11	96	7.7375	13.4876	7.9303
12	264	8.4563	14.6324	8.4729
13	432	11.3315	14.9876	8.765
14	600	13.0650	15.1328	10.6015
15	720	13.0650	15.2845	12.2293
16	1080	13.0650	15.2986	17.1127
17	1440	13.0650	15.2986	18.824
18	1464	13.0650	15.2986	18.824

The percentage (%) of weight gain for the IPSS test with respect to time of exposure was calculated from the equation 4.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after ATF exposure, which is as follows (Fig. 4.15).

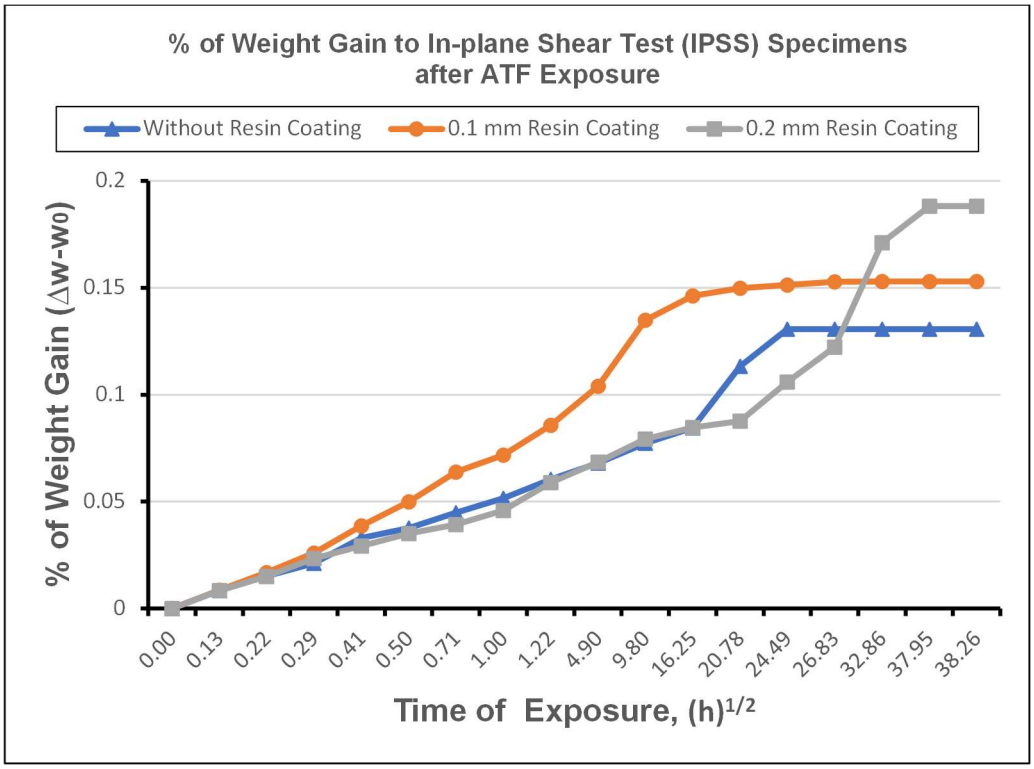


Fig. 4.15 Percentage of weight gain to in-plane shear test specimens after ATF exposure

4.4 Summary

ATF ingression to E-glass/epoxy composite were depicted in this chapter. Percentage of weight gain to respective traveller coupons of the tests were recorded and listed down during ATF exposure and the same was plotted with respect to time of exposure, for better understanding and correlation in between bare and resin coated specimens. The test specimens coated with 0.2 mm resin coated exhibited higher ATF ingression relative to bare and 0.1mm resin coated one.

Chapter 5

Seawater Ingression into Glass Fiber Reinforced Epoxy Composite

5.1 Introduction

Nowadays usage of composites, like glass fiber reinforced composites as structural load-bearing materials in aerospace applications, has become a reality. As the structures made of E-glass/epoxy composites are expected to be in service for a longer period and it has to undergo various types of loading and environmental conditions such as seawater, hygrothermal, UV radiation, chemical environments, biological conditions, etc., hence its long-term performance is to be ensured. The exposure of seawater of such kind, as E-glass/epoxy composite's tendency towards absorbing the same, will decide its degradation of mechanical properties. This degradation can affect structural performance. So, the study of this effect has become very important for said composite.

The tensile, compressive, flexural, and shear properties play an important role in designing structural composites, degradation of any kind will not only lead to failure but will restrict its

usage. Hence in this study seawater ingression effect on these properties of composites was examined.

Nevertheless, most of the above-mentioned studies used various environments, for immersion to explore the residual strength of epoxy laminates. The currently available data is limited to the E-glass/epoxy composite, which has undergone seawater exposure. Furthermore, there are not enough considerations that report a study about seawater exposure on mechanical properties of E-glass/epoxy composite laminates having barrier resin coating of different thicknesses. Hence seawater ingression to said composite were properly studied for three conditions of test specimens i.e., bare, 0.1 mm and 0.2 mm resin coated and that too for tensile, compressive, flexural, interlaminar and in-plane shear properties.

5.2 Exposure to Seawater

At the outset, the prepared specimens can have some degree of moisture ingression from the environment that has to be dried up inside a hot oven by executing a de-moisturization cycle at 110^0 C for 1 hr. After the completion of this cycle, all the specimens and traveler coupons were initially weighted before seawater exposure. After this, it was immersed inside the container, which contains seawater. The traveler coupon was periodically removed; the surface was dried with tissue paper and weighed again using the electronic weighing balance of 0.1 mg accuracy, to know the rate and amount of seawater absorption. Initially, the weight changes of the traveler coupons were measured and recorded at 24 hrs. intervals to determine the amount of seawater ingression. The weighing of coupons was frequent enough for the initial one week and then reduced as this process continues and was taken only once a week. Conditioning was continued for the period it got saturated. The seawater ingression of respective specimens was calculated using its initial weight and the weight of the specimens after this exposure at a given time.

As the specimens, which were immersed in seawater exposure were categorized under bare, 0.1 mm, and 0.2 mm resin coating, for each category of specimens, a minimum of three samples each was used for bare, 0.1 mm, and 0.2 mm resin coated, and the average weight gain was determined. It was observed that bare specimens were undergoing a high rate of seawater absorption relative to resin-coated specimens, which shows a lower rate of absorption.

The percentage (%) of seawater gain or loss is computed and is demonstrated as a dry weight percentage

$$M_s = \frac{W_{ts} - W_{0s}}{W_0} X\ 100 \qquad (5.1)$$

Where w_{0s} is the dry specimen weight in grams (before immersion) and w_{ts} is the wet specimen weight at the time 't' in grams (after seawater immersion)

5.3 Experimental Description

5.3.1 Effect of Seawater Exposure on Tensile Properties of E-glass/epoxy Composite

Tensile test samples of different types i.e., warp and weft specimens were immersed in seawater as shown in Fig. 5.1 and once the samples got saturated with seawater ingression, the tensile test was conducted using a servo mechanical testing machine according to ASTM D 3039 standard. The dimensions of the test specimens were maintained as per ASTM requirements. This test was conducted at room temperature with a crosshead speed of 2 mm/min as shown in Fig. 5.1. Extensometer was used for strain measurement.

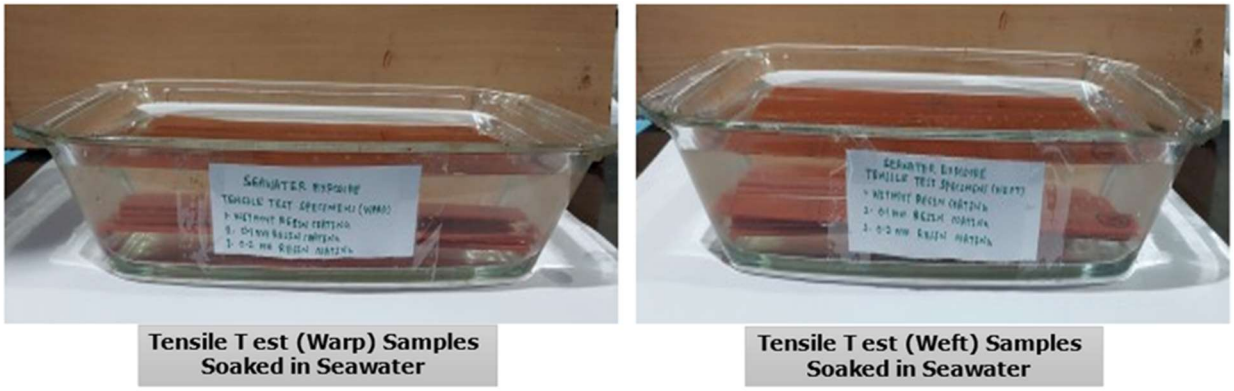


Fig. 5.1 Test samples of the tensile strength (warp and weft) immersed in seawater exposure

All the de-moisturized tensile test specimens and traveler coupons were initially weighted then followed by seawater exposure as per the procedure mentioned in section 5.2. The weight changes of the traveler coupons were measured as mentioned in Table 5.1.

Initial weight of the traveller coupon of tensile test (warp):

a) Without resin coated: 23.502 grams
b) 0.1 mm resin coated: 25.293 grams
c) 0.2 mm resin coated: 27.146 grams

Table 5.1 Percentage of weight gain of tensile test (warp) specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.05	0.9871	0.5542	4.4703
3	0.25	3.7339	16.9236	4.8076
4	1.5	4.3777	19.0125	11.7239
5	24	5.7940	29.4139	25.2191
6	120	14.4636	46.3375	45.8835
7	288	24.6783	55.7159	57.4810
8	456	36.1376	65.0516	70.5966
9	624	45.3223	83.5951	79.1576
10	792	55.1936	9.58296	89.7428
11	1272	55.1936	102.5650	98.8099
12	1680	55.1930	102.5650	107.0757
13	1704	55.1936	102.5650	107.0757

The percentage (%) of weight gain for the tensile test (warp) with respect to time of exposure was calculated from the equation 5.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after seawater exposure, which is as follows (Fig. 5.2).

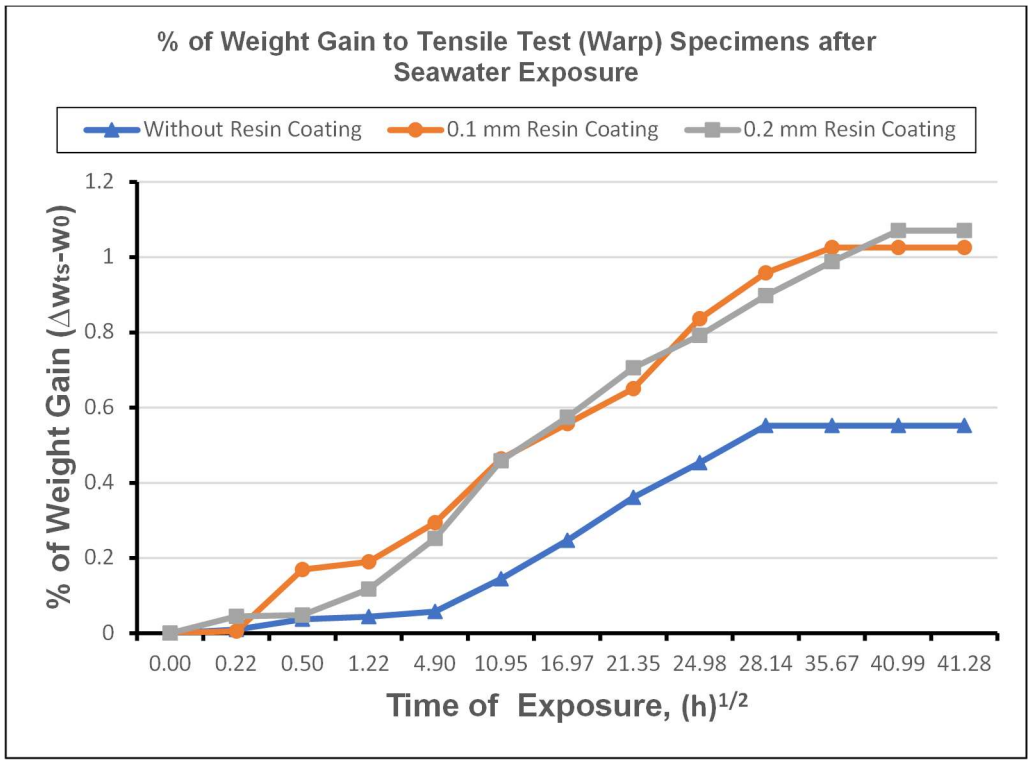


Fig. 5.2 Percentage of weight gain to tensile test (warp) specimens after seawater exposure

Similarly, the weight gain of the tensile test (weft) is to be recorded as it was done for tensile warp test specimens, after immersing the same in seawater exposure. The traveler coupons will be taken out periodically to measure the percentage (%) of weight gain as depicted in Table 5.2.

Initial weight of the traveller coupon of tensile test (weft):

a) Without resin coated: 23.487 grams
b) 0.1 mm resin coated: 25.265 grams
c) 0.2 mm resin coated: 27.105 grams

Table 5.2 Percentage of weight gain of tensile test (weft) specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.05	1.4023	0.1639	0.5290
3	0.25	2.4746	0.3689	1.7093

4	1.5	3.3407	0.7787	8.5463
5	24	6.1040	9.6319	24.0112
6	120	17.6521	24.1412	45.9875
7	288	24.4572	24.7560	48.6328
8	456	37.3251	41.7655	57.4233
9	624	53.8637	54.5124	89.6959
10	792	60.9987	51.2745	102.9631
11	1272	60.9987	63.6115	111.5502
12	1680	60.9987	63.6115	119.2012
13	1704	60.9987	63.6115	119.2012

The percentage (%) of weight gain for the tensile test (weft) with respect to time of exposure was plotted as follows (Fig. 5.3).

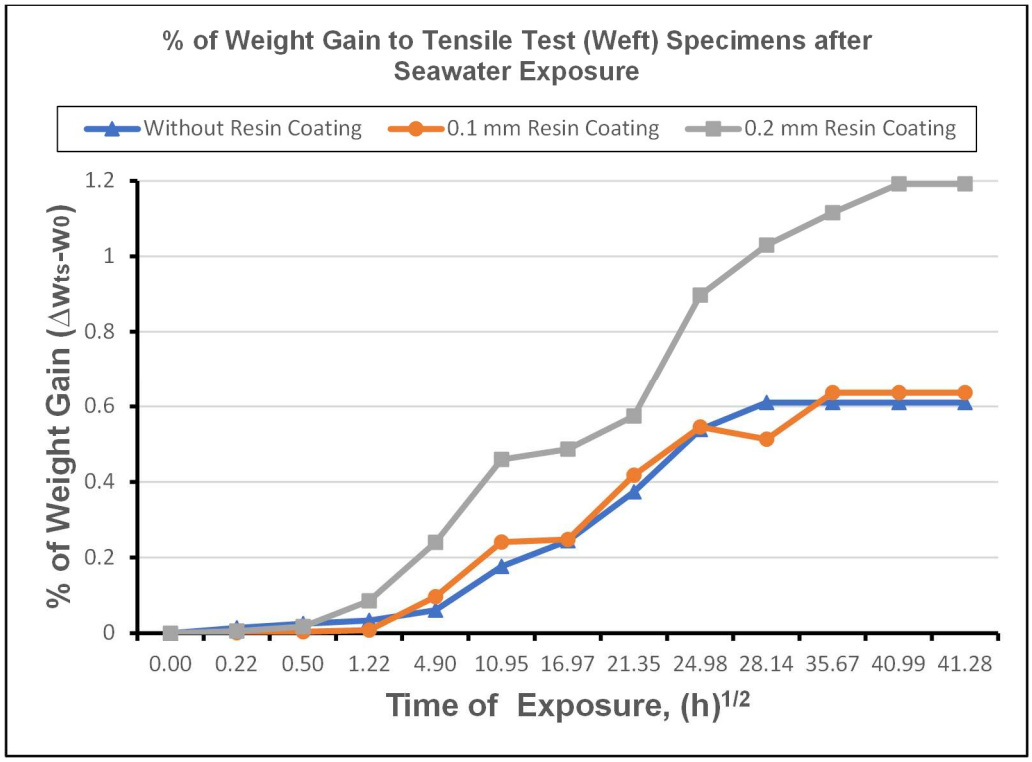


Fig. 5.3 Percentage (%) of weight gain to tensile test (weft) specimens after seawater exposure

5.3.2 Effect of Seawater Exposure on Compressive Properties of E-glass/epoxy Composite

Compressive test samples of different types were immersed in Seawater as shown in Fig.5.4, for the period of 1704 hrs, this test was conducted using a servo mechanical universal testing machine as per ASTM D 3410 standard. Strain gauges were used for strain measurement. Compressive strength was evaluated from the ultimate load and compressive modulus was determined from the stress-strain curve in the linear portion. Bare samples without the tab of compression test (Fig.5.5) for all three conditions, were immersed in seawater.

Once the test specimens for the compression test are prepared, they will undergo for de-moisturization cycle to remove initially absorbed moisture from the atmosphere. After this, all the specimens and traveler coupons were initially weighted before seawater exposure. After this, they were immersed inside the container, which contains the seawater.

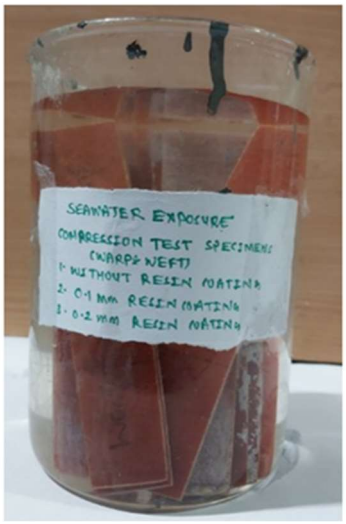


Fig. 5.4 Test samples of compressive strength (warp and weft) immersed to seawater exposure

Fig. 5.5 Bare test samples without tabs

The traveler coupons were periodically removed, surface dried with a lint-free cloth, and weighed again using the electronic weighing balance of 0.1 mg accuracy, to know the rate and amount of seawater absorption for a period of 1704 hours. The weight changes of the traveler coupons were measured as mentioned in Table 5.3. The seawater ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time. Initial weight of the traveller coupon of compressive test (warp):

a) Without resin coated: 16.384 grams
b) 0.1 mm resin coated: 17.389 grams
c) 0.2 mm resin coated: 18.431 grams

Table 5.3 Percentage of weight gain of compression test (warp) specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.4901	1.4596	0.2415
3	0.05	0.7965	2.4327	0.7848
4	0.0833	1.2866	3.6490	1.3885
5	0.167	1.9606	3.7707	2.0525
6	0.25	2.2057	3.8923	2.4147
7	0.5	2.9409	4.0139	4.4069
8	1	3.7986	4.2572	6.2784
9	1.5	4.4113	4.4397	7.9687
10	24	5.5754	9.9132	26.2605
11	120	13.1114	25.9081	54.6943
12	288	28.4286	34.4834	66.7681
13	456	30.8180	40.9908	75.8234
14	624	32.4110	53.0326	93.5115

15	792	33.7589	64.4055	104.9213
16	1292	33.7589	76.0216	117.5384
17	1680	33.7589	76.0216	18.6250
18	1704	33.7589	76.0216	118.6250

The percentage (%) of weight gain for the compressive test (warp) with respect to time of exposure was calculated from the equation 5.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after ATF exposure, which is as follows (Fig. 5.6).

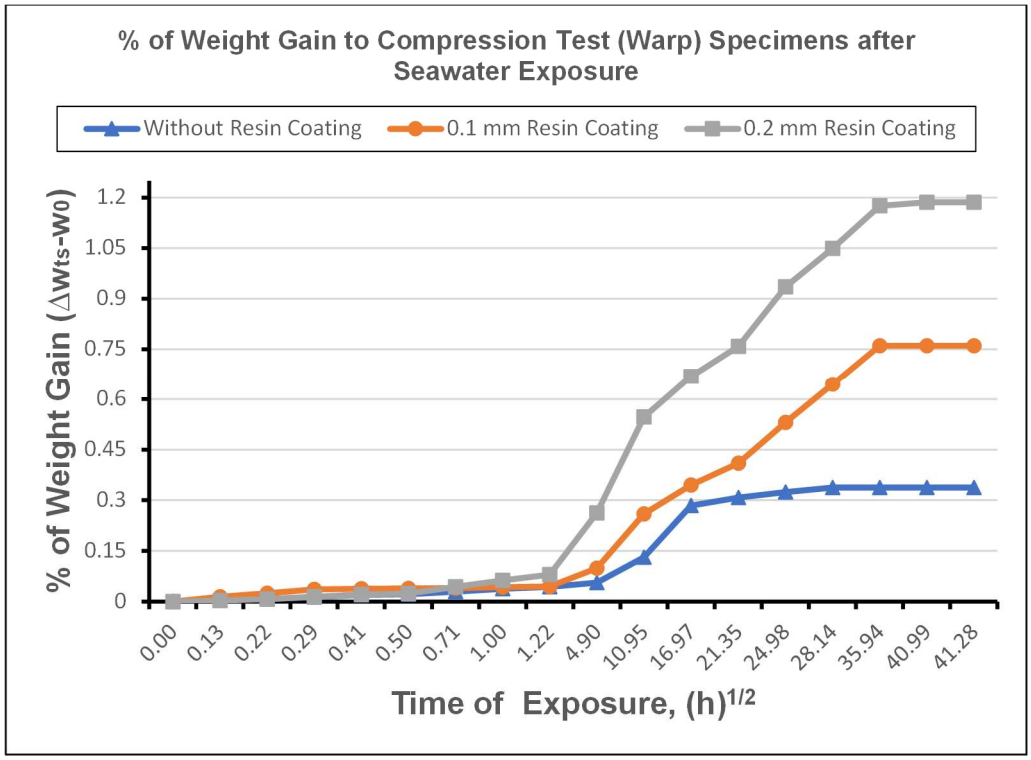


Fig. 5.6 Percentage of weight gain to compressive test (warp) specimens after seawater exposure

Similarly, the weight gain of the compression test (weft) is to be recorded as it was done for compressive warp test specimens, after immersing the same in seawater exposure. The traveler coupons will be taken out periodically to measure the percentage (%) of weight gain as depicted in Table 5.4.

Initial weight of the traveller coupon of compressive test (weft):

a) Without resin coated: 16.304 grams

b) 0.1 mm resin coated: 17.302 grams

c) 0.2 mm resin coated: 18.336 grams

Table 5.4 Percentage of weight gain Weight gain of compression test (weft) specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.00.4946	0.003069	0.003049
3	0.05	0.00.9273	0.007979	0.004268
4	0.0833	0.01.2983	0.015345	0.006707
5	0.167	0.01.9784	0.029463	0.007926
6	0.25	0.02.3493	0.034987	0.009756
7	0.5	0.02.9675	0.03.6215	0.02.4999
8	1	0.04.3895	0.03.8056	0.09.4507
9	1.5	0.05.0695	0.03.9284	0.03.7193
10	24	0.05.9351	0.13.1356	0.15.4261
11	120	0.14.8377	0.34.5577	0.36.5836
12	288	0.25.4714	0.46.6498	0.45.7295
13	456	0.32.7048	0.54.0156	0.55.9729
14	624	0.40.7419	0.60.5834	0.63.4115
15	792	0.47.1716	0.65.3097	0.68.8991
16	1292	0.47.1716	0.78.2612	0.80.1180
17	1680	0.47.1716	0.78.2612	0.81.3375
18	1704	0.47.1716	0.78.2612	0.81.3375

The percentage (%) of weight gain for the compressive test (weft) with respect to time of exposure was plotted as follows (Fig. 5.7).

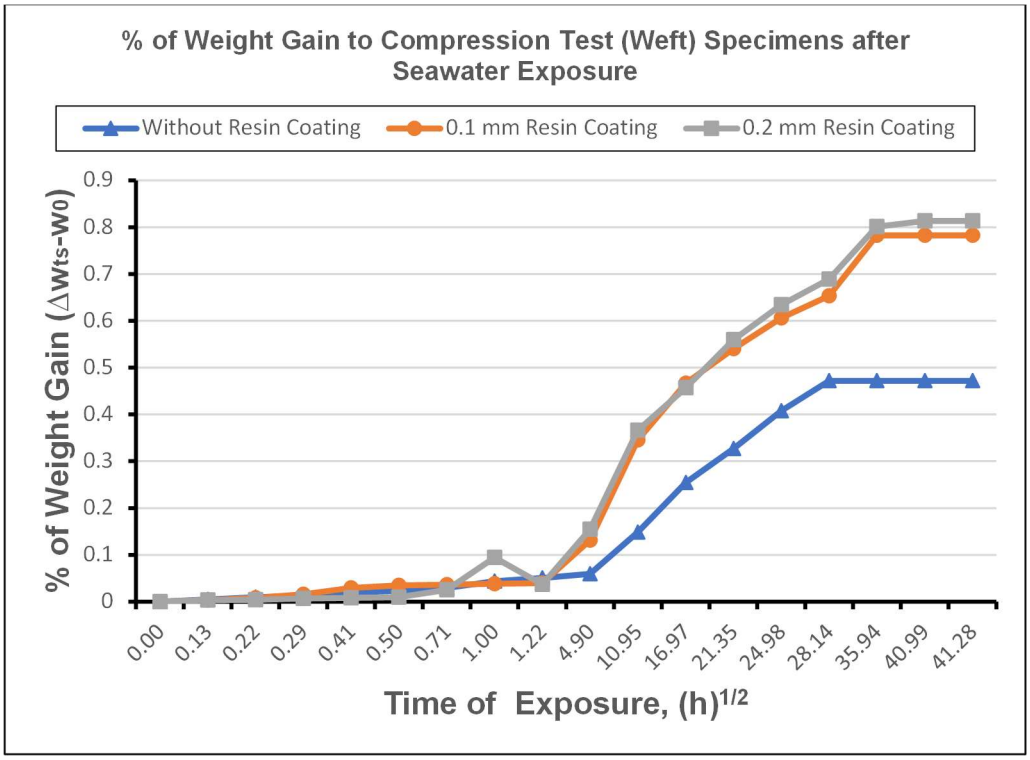


Fig. 5.7 Percentage of weight gain to compressive test (weft) specimens after seawater exposure

5.3.3 Effect of Seawater Exposure on Flexural Properties of E-glass/epoxy Composite

Flexural test samples of different types were immersed in seawater as shown in Fig. 5.8 for 1704 hrs, this test was conducted using a servo mechanical universal testing machine as per ASTM D 790 standard.

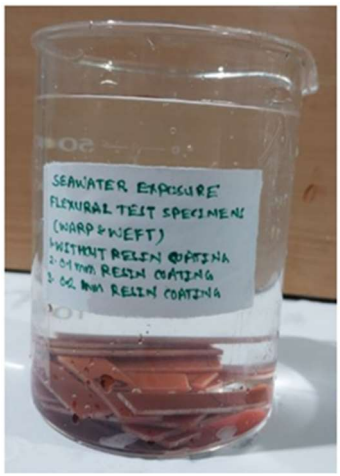


Fig.5.8 Flexural test (warp and weft) samples immersed in seawater

All the de-moisturized flexural test specimens and traveler coupons were initially weighted then followed by seawater exposure. The weight changes of the traveler coupons were measured as mentioned in Table 5.5. The seawater ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Initial weight of the traveller coupon of flexural test (warp):

a) Without resin coated: 2.584 grams
b) 0.1 mm resin coated: 2.793 grams
c) 0.2 mm resin coated: 3.013 grams

Table 5.5 Percentage of weight gain of flexural test (warp) specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.7789	2.2867	0.3750
3	0.05	1.1684	3.8111	0.7500
4	0.0833	1.1684	6.4789	1.4999
5	0.167	1.9473	8.0033	2.6249
6	0.25	2.3368	10.2900	2.9998
7	0.5	2.7263	10.6711	3.3748
8	1	3.5052	11.4334	4.1248
9	1.5	4.2841	11.8145	4.4998
10	24	5.4526	29.3456	12.3744
11	120	8.1788	51.4501	32.2484
12	288	18.6945	62.502	43.8728
13	456	24.5365	65.9324	49.1225
14	624	29.5996	73.1735	69.7465
15	792	38.5574	79.6524	86.6207
16	1292	38.5574	84.9880	91.4954

17	1680	38.5574	88.4180	91.4954
18	1704	38.5574	88.4180	91.4954

The percentage (%) of weight gain for the flexural test (warp) with respect to time of exposure was calculated from the equation 5.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after seawater exposure, which is as follows (Fig. 5.9).

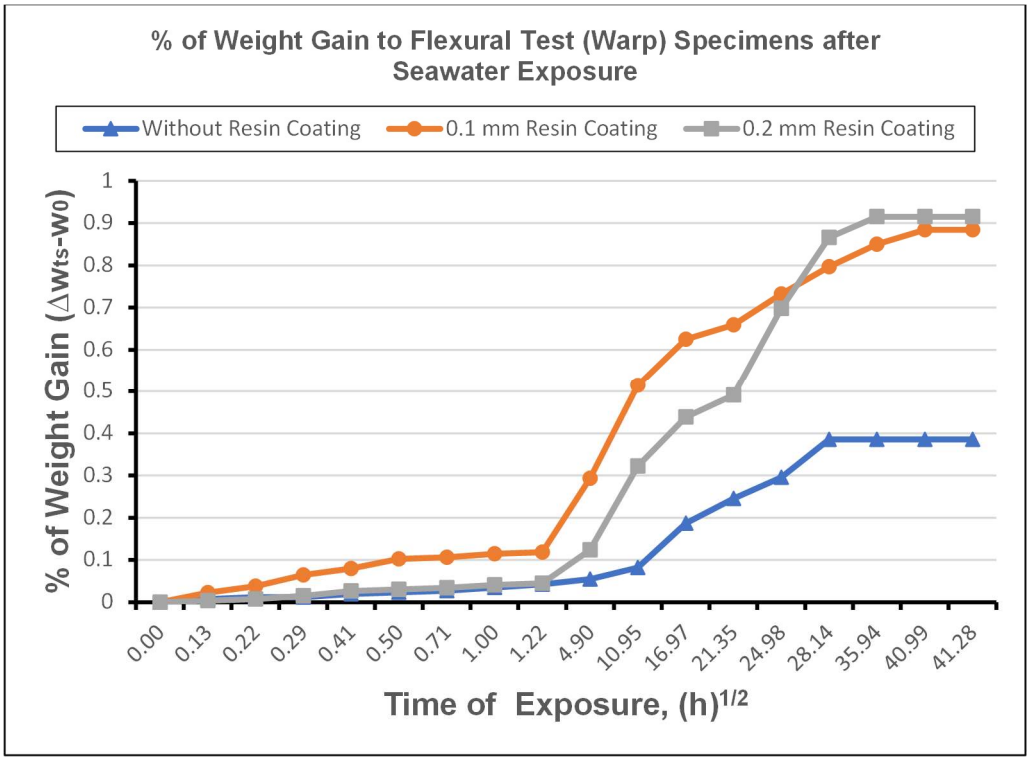


Fig. 5.9 Percentage (%) of weight gain to flexural test (warp) specimens after seawater exposure

Similarly, the weight gain of the flexural test (weft) is to be recorded as it was done for flexural warp test specimens, after immersing the same in seawater exposure. The traveler coupons will be taken out periodically to measure the percentage (%) of weight gain as depicted in Table 5.6.

Initial weight of the traveller coupon of flexural test (weft):

a) Without resin coated: 2.439 grams
b) 0.1 mm resin coated: 2.647 grams
c) 0.2 mm resin coated: 2.865 grams

Table 5.6 Percentage of weight gain of flexural test (weft) specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.7953	0.7826	0.3869
3	0.05	1.9883	1.5652	0.7738
4	0.0833	2.3860	2.7391	1.1607
5	0.167	3.1812	3.5217	1.5476
6	0.25	3.9766	4.6956	1.5476
7	0.5	4.3743	5.4782	3.8691
8	1	5.1696	6.6520	5.4167
9	1.5	5.5673	7.4346	6.5774
10	24	6.7602	16.4345	20.1192
11	120	19.0878	52.4339	58.0360
12	288	19.0878	57.5207	63.4527
13	456	29.4270	68.4771	71.5778
14	624	34.9942	75.9117	80.0898
15	792	40.9592	85.3029	89.3755
16	1292	40.9592	91.9549	94.7922
17	1680	40.9592	91.9549	98.6613
18	1704	40.9592	91.9549	98.6613

The percentage (%) of weight gain for the flexural test (weft) with respect to time of exposure was plotted as follows (Fig. 5.10).

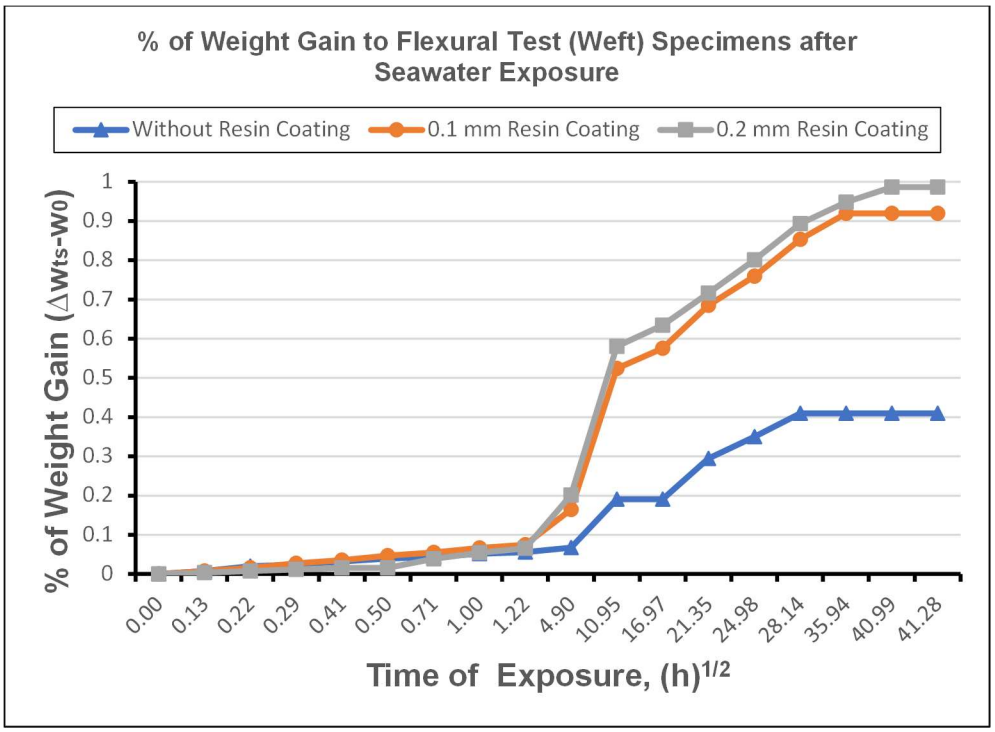


Fig. 5.10 Percentage (%) of weight gain to flexural test (weft) specimens after seawater exposure

5.3.4 Effect of Seawater Exposure on Interlaminar and In-plane Shear Properties of E-glass/epoxy Composite

Interlaminar Shear Properties

Interlaminar shear strength (ILSS) test samples of different types were immersed in seawater as shown in Fig. 5.11 and the samples were given 1704 hours of exposure to seawater ingression; this test was carried out by using a servo mechanical testing machine as per ASTM D 2344 standard. The ILSS test specimens dimensions were maintained as per ASTM requirements. The interlaminar shear strength is determined using a test fixture of three-point.

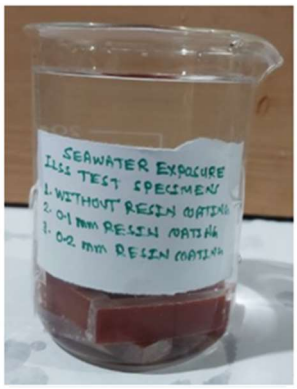


Fig. 5.11 ILSS test samples immersed in seawater

All the de-moisturized ILSS test specimens and traveler coupons were initially weighted then followed by seawater exposure as per the procedure mentioned in section 5.2. The weight changes of the traveler coupons were measured as mentioned in Table 5.7. The seawater ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Initial weight of the traveller coupon of ILSS test:

a) Without resin coated: 3.721 grams
b) 0.1 mm resin coated: 3.878 grams
c) 0.2 mm resin coated: 4.044 grams

Table 5.7 Percentage of weight gain of ILSS test specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	3.2492	0.5317	0.7826
3	0.05	3.5200	1.3292	2.0871
4	0.1	1.8954	1.5951	2.3480
5	0.2	0.5415	2.1268	2.6088
6	0.4	0.5415	2.3927	2.8697
7	0.75	1.3538	4.2536	3.9133
8	1	2.4369	8.7731	-73.3088
9	1.5	2.9784	11.4316	5.2177
10	24	4.0615	18.8754	14.3487
11	120	14.3507	31.6363	28.1756
12	288	17.3291	43.5996	42.7852
13	456	29.2429	46.2581	46.9594
14	576	30.5968	49.4483	52.1770
15	792	31.9506	52.9044	57.6557

16	1292	31.9506	62.7409	68.8737
17	1680	31.9506	62.7409	73.0479
18	1704	31.9506	62.7409	73.0479

The percentage (%) of weight gain for the ILSS test with respect to time of exposure was calculated from the equation 5.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after seawater exposure, which is as follows (Fig. 5.12).

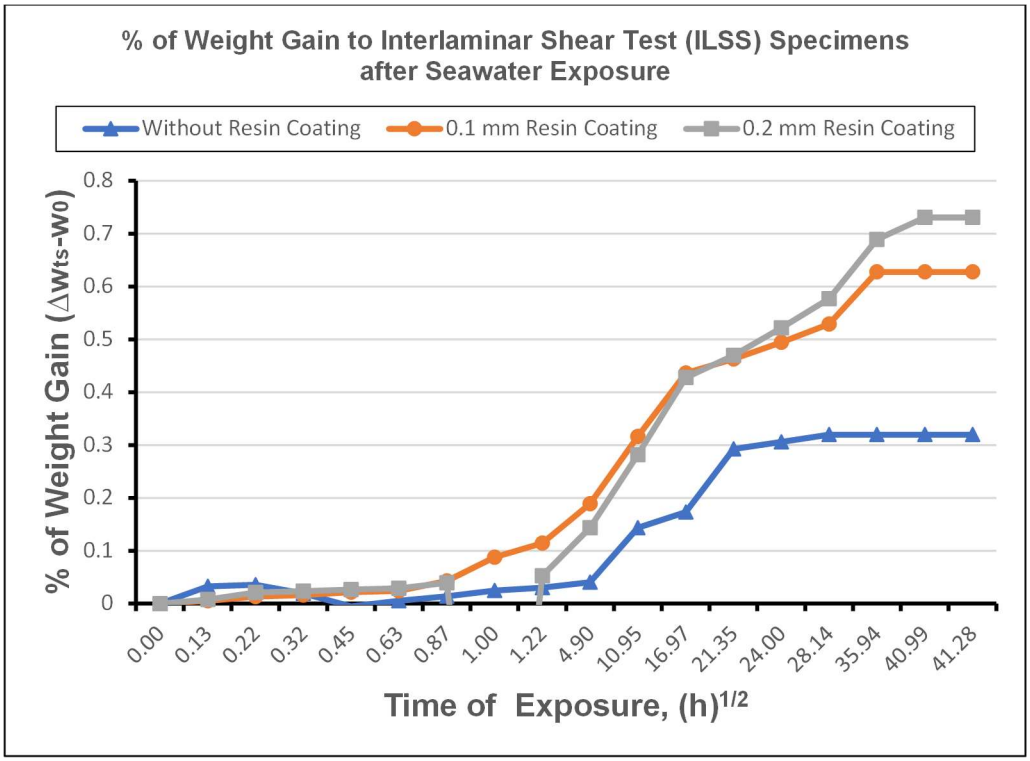


Fig. 5.12 Percentage of weight gain to interlaminar test specimens after seawater exposure

In-plane Shear Properties

In-plane shear strength (IPSS) test samples of different types were immersed in seawater as shown in Fig. 5.13 and the samples were given 1704 hours of exposure with seawater ingression, a servo mechanical testing machine was used while conducting this test according to ASTM D 3518 standard. The IPSS test specimens dimensions were maintained as per ASTM requirements. The laminate for this test was prepared by stacking the layers $+45^0$, and -45^0 as per standard.

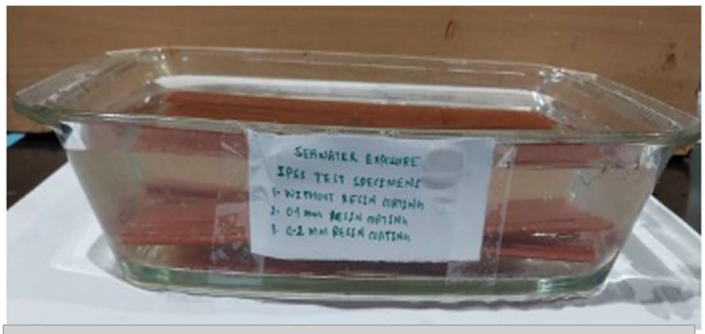


Fig. 5.13 IPSS test samples immersed in seawater

All the de-moisturized IPSS test specimens and traveler coupons were initially weighted then followed by seawater exposure as per the procedure mentioned in section 5.2. The weight changes of the traveler coupons were measured as mentioned in Table 5.8. The seawater ingression of respective specimens was calculated using the initial weight and weight of the specimens after this exposure at a given time.

Initial weight of the traveller coupon of IPSS test:

a) Without resin coated: 18.860 grams
b) 0.1 mm resin coated: 20.593 grams
c) 0.2 mm resin coated: 22.387 grams

Table 5.8 Percentage of weight gain of IPSS test specimens after seawater exposure

Sl No.	Time of Exposure (hours)	Percentage of weight gain to the test specimens (%)		
		Without Resin Coating (x 10^{-2})	0.1 mm Resin coating (x 10^{-2})	0.2 mm Resin coating (x 10^{-2})
1	0	0	0	0
2	0.0167	0.4586	0.1657	0.1235
3	0.05	1.0841	0.3729	0.3294
4	0.0833	1.3342	0.4971	0.4941
5	0.167	2.1264	0.5800	0.7000
6	0.25	2.6684	0.6629	0.8236
7	0.5	3.5440	0.8286	2.4707
8	1	4.4613	0.9529	5.3532

9	1.5	5.0450	1.0357	7.3710
10	24	8.5891	8.4930	23.5955
11	120	20.2634	28.5862	48.3028
12	288	31.3127	41.7607	58.0622
13	456	43.8627	66.9912	77.7869
14	624	48.2407	80.7457	94.2996
15	792	51.3261	95.2874	102.5766
16	1292	51.3261	100.8389	113.4478
17	1680	51.3261	100.8389	118.5951
18	1704	51.3261	100.8389	118.5951

The percentage (%) of weight gain for the IPSS test with respect to time of exposure was calculated from the equation 5.1 and plotted for bare specimen, with 0.1 mm and 0.2 mm resin coated after seawater exposure, which is as follows (Fig. 5.14).

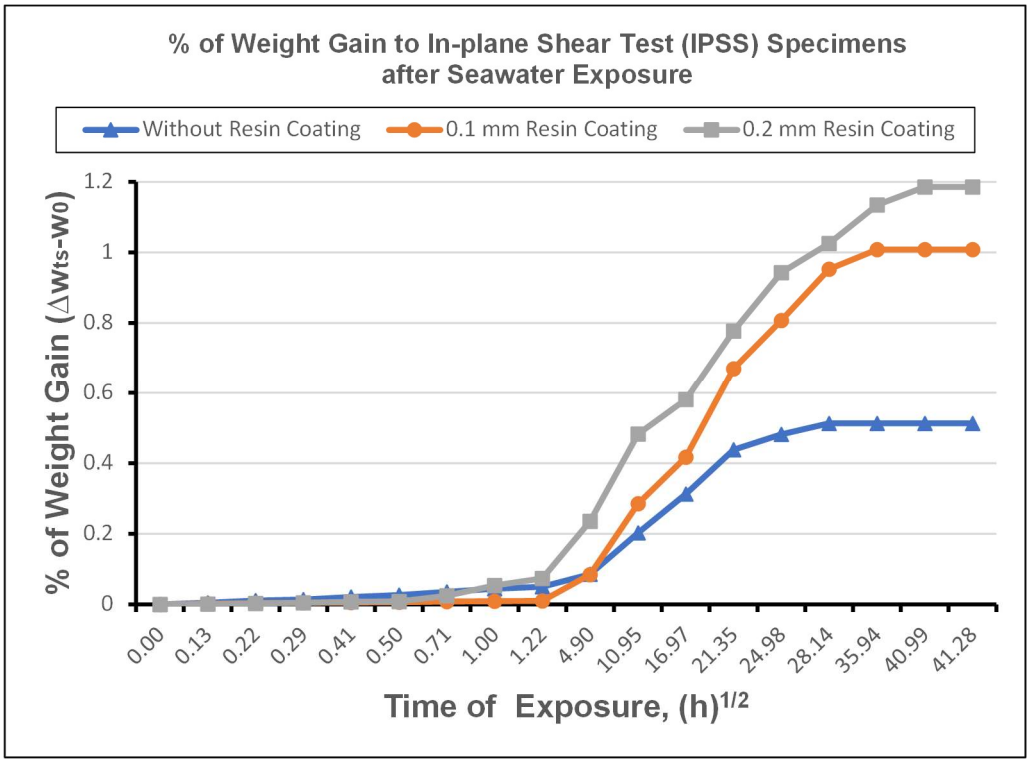


Fig. 5.14 Percentage (%) of weight gain to in-plane shear test specimens after seawater exposure

5.4 Summary

Seawater ingression to E-glass/epoxy composite were reported in this chapter. Percentage of weight gain to respective traveller coupons of the tests were recorded and listed down during seawater exposure and the same was plotted with respect to time of exposure, for better understanding and correlation in between bare and resin coated specimens. The test specimens coated with 0.2 mm resin coated exhibited higher seawater ingression relative to bare and 0.1mm resin coated one.

Chapter 6

Results and Discussions

6.1 Effect of ATF Exposure on Mechanical Properties of E-glass/epoxy composite

6.1.1 Introduction

Mechanical properties like tensile, compressive, flexural and shear properties play a significant contribution to the design and long-term performance of products made of glass fiber-reinforced composites. In this research work, to quantify the property degradation for use in design, the effect of ATF ingression on mechanical properties of E-glass/epoxy composites is studied. The purpose of this chapter is to characterize the effect of a longer duration of ATF exposure on the mechanical properties of laminates of fiber-reinforced composite. An orderly investigation is performed to determine and compare the influence of ATF ageing on the mechanical properties of the said composite in bare and with barrier resin-coated conditions for better understanding

and to improve the design of aerospace components which has ATF exposure. The durability of E-glass/epoxy composite under ATF exposure in bare and resin-coated conditions was investigated in this chapter. In addition to this, microstructural analysis was also carried out for better understanding of the effect of ATF exposure.

6.1.2 Mechanical Test Results of E-glass/epoxy Composite after ATF Exposure

6.1.2.1 Test results of Tensile Test Specimens

The ATF exposure has saturated tensile test specimens (warp) for all three conditions of tensile test specimens, where bare specimens were saturated first, followed by 0.1 mm resin coated, and last 0.2 mm resin coated specimens were saturated. Once it got saturated with ATF, will be taken out, and its surface dried up with tissue paper, followed by its mechanical testing using UTM. The results of the tensile test (warp) were as follows (Table 6.1).

Table 6.1 Tensile test specimens (warp) results after ATF exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Tensile Strength (MPa)
(Without Resin Coating)					
1	TS-ATF-Warp-WRC-01	25.01 x 2.04	5.983	18.277	358.23
2	TS-ATF-Warp-WRC-02	25.23 x 2.21	6.203	18.574	333.12
3	TS-ATF-Warp-WRC-03	25.21 x 1.96	5.194	17.36	351.34
4	TS-ATF-Warp-WRC-04	25.42 x 1.98	6.731	17.358	344.87
(With 0.1 mm Resin Coating)					
1	TS-ATF-Warp-0.1 mm RC-01	25.31 x 1.95	6.005	17.073	345.92
2	TS-ATF-Warp-0.1 mm RC-02	25.46 x 1.99	6.212	18.35	362.18
3	TS-ATF-Warp-0.1 mm RC-03	24.87 x 2.04	5.634	17.974	354.28
4	TS-ATF-Warp-0.1 mm RC-04	25.25 x 2.00	6.154	18.688	370.06
(With 0.2 mm Resin Coating)					
1	TS-ATF-Warp-0.2 mm RC-01	24.61 x 2.08	5.610	17.973	351.12

2	TS-ATF-Warp-0.2 mm RC-02	25.37 x 2.00	6.250	18.826	371.02
3	TS-ATF-Warp-0.2 mm RC-03	25.26 x 2.01	6.010	18.242	359.14
4	TS-ATF-Warp-0.2 mm RC-04	25.22 x 2.01	6.227	18.866	372.17

The brief of test results of tensile strength and modulus (warp) for all three conditions of test specimens are tabulated in Table 6.2.

Table 6.2 Tensile strength and modulus (warp) results after ATF exposure

Test Specimens	Tensile Strength-without resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.1 mm resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.2 mm resin coating (MPa)	Tensile Modulus (GPa)
1	358.23	21.47	345.92	22.62	351.12	22.33
2	333.12	20.07	362.18	21.53	371.02	23.07
3	351.34	21.52	354.28	22.13	359.14	22.18
4	344.87	20.75	370.06	21.81	372.17	22.52
Mean	346.89	20.95	358.11	22.02	363.36	22.53
SD	10.68	0.69	10.37	0.47	10.07	0.48
% Degradation	8.79%	12.18%	5.84%	7.67%	4.46%	5.55%

Once the tensile strength and modulus (warp) values were obtained for all three conditions of specimens, the average value is to be calculated and tabulated as follows in Table 6.3 and that has to be compared with the average value of as-received specimens.

Table 6.3 Effect of ATF exposure on tensile properties of E-glass/epoxy composite in the warp direction

Sample condition	Average tensile strength (warp) (MPa)		Average tensile modulus (warp) (GPa)	
	As-received	After ATF exposure	As-received	After ATF exposure
Bare samples	380.32	346.89	23.85	20.95
0.1 mm resin coated		358.11		22.02
0.2 mm resin coated		363.36		22.53

The tensile test specimens in the warp direction were tested experimentally and it was observed that tensile strength was reduced by 8.79%, 5.84%, and 4.46 % for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 12.18%, 7.67%, and 5.55% degradation in terms of tensile modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively. The comparison of average tensile properties (warp) of as-received specimens with respect to bare and resin-coated specimens, was represented in the form of a bar chart as depicted in Fig. 6.1.

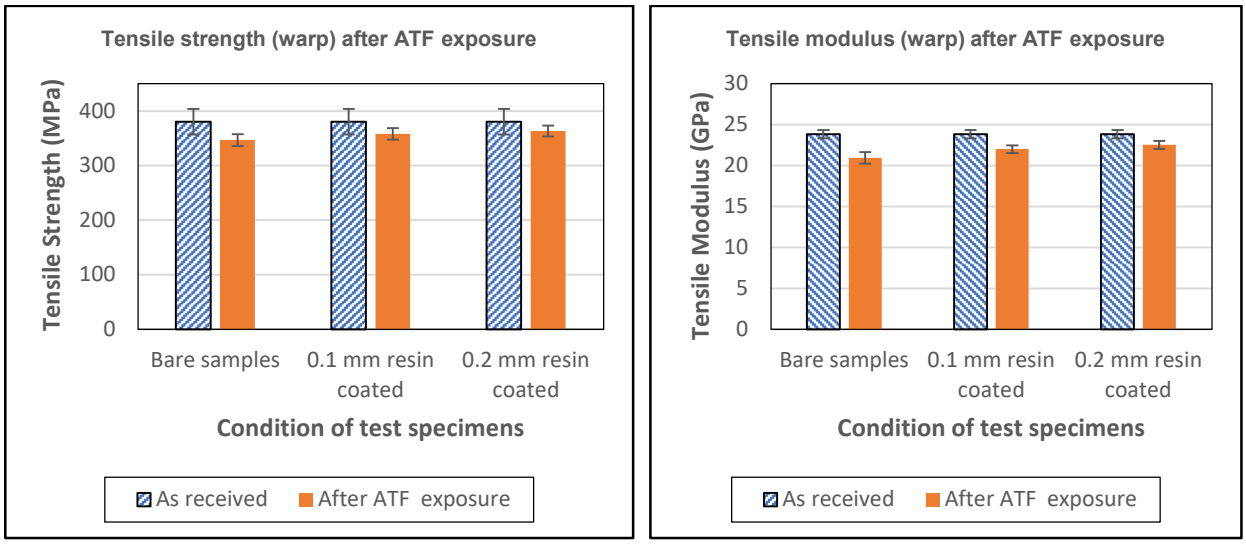


Fig. 6.1 Comparison chart of tensile properties (warp) for as-received specimens with bare and resin-coated specimens after ATF exposure

ATF exposed tensile test samples (warp) after testing are depicted in Fig. 6.2 as follows.

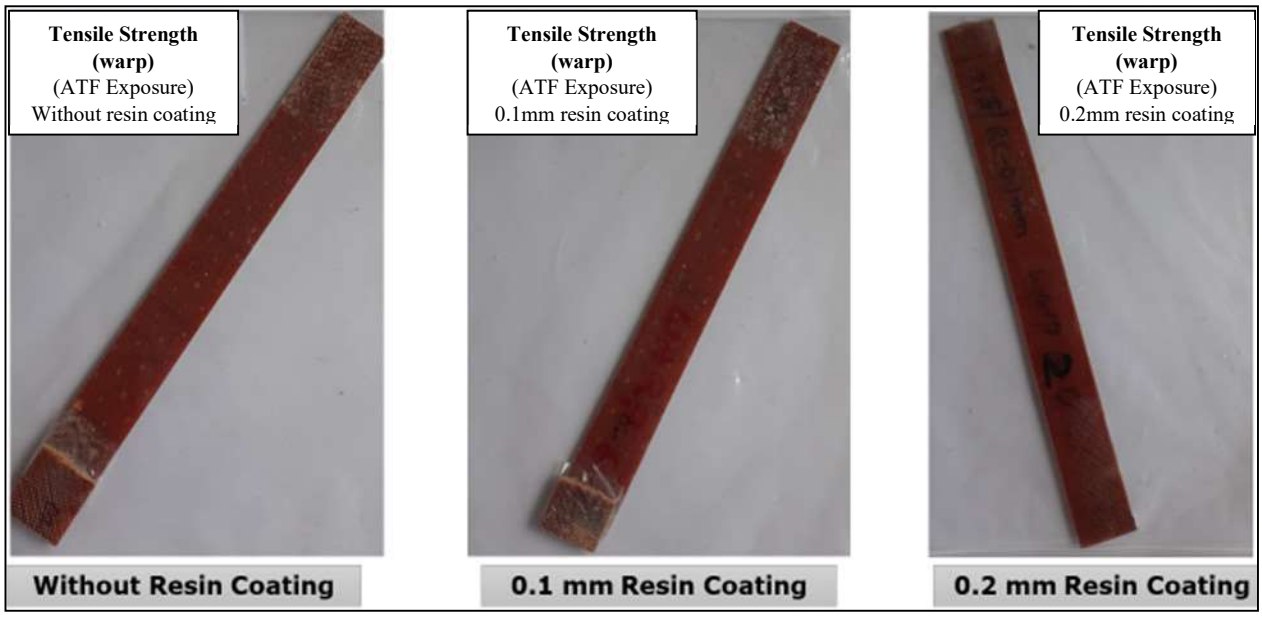


Fig. 6.2 Tensile strength (warp) test samples after ATF exposure (after testing)

The tensile test specimens (weft) for all three conditions were exposed and saturated in ATF environment where it was observed that bare specimens were saturated first, followed by with 0.1 mm resin coated, and last with 0.2 mm resin-coated specimens. Once it got saturated with ATF, these specimens were taken out, and its surface was dried up with tissue paper, followed by its mechanical testing using UTM. The results of the tensile test (weft) were as follows (Table 6.4).

Table 6.4 Tensile test specimens (weft) results after ATF exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Tensile Strength (MPa)
(Without Resin Coating)					
1	TS-ATF-Weft-WRC-01	25.32 x 2.10	5.61	17.766	333.87
2	TS-ATF-Weft-WRC-02	24.92 x 2.05	6.127	18.035	353.18
3	TS-ATF-Weft-WRC-03	24.82 x 2.11	4.65	16.514	316.44
4	TS-ATF-Weft-WRC-04	24.90 x 1.98	5.717	16.85	342.14
(With 0.1 mm Resin Coating)					
1	TS-ATF-Weft-0.1 mm RC -01	24.82 x 2.11	5.217	18.529	350.43

2	TS-ATF-Weft-0.1 mm RC -02	24.61 x 2.08	5.677	18.187	355.11
3	TS-ATF-Weft-0.1 mm RC -03	25.26 x 2.22	6.162	18.575	331.49
4	TS-ATF-Weft-0.1 mm RC -04	24.90 x 1.98	-	-	344.12
(With 0.2 mm Resin Coating)					
1	TS-ATF-Weft-0.2 mm RC -01	25.02 x 2.16	5.813	18.648	345.19
2	TS-ATF-Weft-0.2 mm RC -02	25.26 x 2.22	6.291	18.964	337.39
3	TS-ATF-Weft-0.2 mm RC -03	24.95 x 2.18	6.588	19.442	361.08
4	TS-ATF-Weft-0.2 mm RC -04	25.25 x 1.94	5.237	17.495	354.71

The brief of test results of tensile strength and modulus (weft) for all three conditions of test specimens are tabulated in Table 6.5.

Table 6.5 Tensile strength and modulus (weft) results after ATF exposure

Test Specimens	Tensile Strength-without resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.1 mm resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.2 mm resin coating (MPa)	Tensile Modulus (GPa)
1	333.87	19.29	350.43	20.18	345.19	20.85
2	353.18	20.42	355.11	21.37	337.39	20.41
3	316.44	18.76	331.49	20.29	361.08	21.61
4	342.14	19.68	344.12	20.68	354.71	21.84
Mean	336.41	19.54	345.29	20.63	349.59	21.18
SD	15.48	0.7	10.24	0.54	10.43	0.66
% Degradation	7.58%	11.82%	5.14%	6.92%	3.96%	4.44%

The average values of tensile strength and modulus (weft) were calculated, for all three conditions of specimens and tabulated as follows in Table 6.6 and that has to be compared with the average value of as-received specimens.

Table 6.6 Effect of ATF exposure on tensile properties of E-glass/epoxy composite in the weft direction

Sample condition	Average tensile strength (weft) (MPa)		Average tensile modulus (weft) (GPa)	
	As-received	After ATF exposure	As-received	After ATF exposure
Bare samples	364	336.41	22.16	19.54
0.1 mm resin coated		345.29		20.63
0.2 mm resin coated		349.59		21.18

However, the tensile test specimens in the weft direction were tested experimentally for bare and with 0.1 mm and 0.2 mm resin coated condition, and correlation of data was made with as-received specimen property. It was observed that tensile strength was reduced by 7.58%, 5.14%, and 3.96% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 11.82%, 6.92% and 4.44% degradation in terms of tensile modulus were observed for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively. The comparison of average tensile properties (weft) of as-received specimens with respect to bare and resin-coated specimens, was represented in the form of a bar chart as depicted in Fig. 6.3.

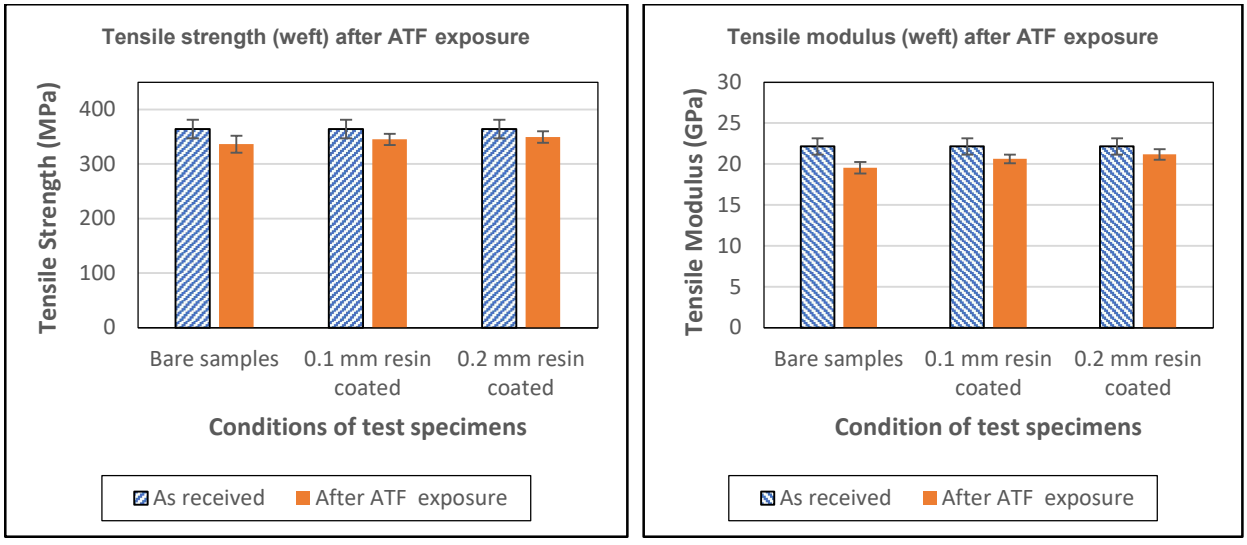


Fig. 6.3 Comparison chart of tensile properties (weft) for as-received specimens with bare and resin-coated specimens after ATF exposure

ATF exposed tensile test samples (weft) after testing are depicted in Fig. 6.4, as follows.

Fig. 6.4 Test samples of the tensile strength (weft) after ATF exposure (after testing)

The degradation of tensile properties in warp and weft direction after ATF exposure, are tabulated as follows in Table 6.7.

Table 6.7 Effect of ATF exposure on the degradation of Tensile properties of E-glass/epoxy composite in warp and weft direction

Sample condition	Degradation of Tensile Strength (%)		Degradation of Tensile Modulus (%)	
	Warp	Weft	Warp	Weft
Bare samples	8.79	7.58	12.18	11.82
0.1 mm resin coated	5.84	5.14	7.67	6.92
0.2 mm resin coated	4.46	3.96	5.55	4.44

6.1.2.2 Test results of Compressive Test Specimens

The compressive test specimens (warp) for all three conditions were saturated under ATF enviornment where it was found that bare specimens were saturated first, followed by with 0.1 mm resin coated, and last with 0.2 mm resin coated specimens. Once it got saturated with ATF, test specimens were taken out, and its surface was dried up with tissue paper. After this room temperature curable E-glass/epoxy tabs were bonded by maintaining 10 mm gage length. Strain

gauges were bonded at the gage length area as shown in Fig. 6.5, to record the corresponding strains relative to the load applied.

Fig.6.5 Test samples of compressive strength (warp) after ATF exposure (before testing)

The compressive specimens will undergo mechanical testing using UTM with specially designed wedge-type fixtures. The results of the compression test (warp) were as follows (Table 6.8).

Table 6.8 Compressive test specimens (warp) results after ATF exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Compressive Strength (MPa)
(Without Resin Coating)					
1	CS-ATF-Warp-WRC-01	25.07 x 2.38	5.099	20.10	336.87
2	CS-ATF-Warp-WRC-02	25.13 x 2.43	5.659	20.03	328.09
3	CS-ATF-Warp-WRC-03	25.03 x 2.48	4.831	19.50	314.06
(With 0.1 mm Resin Coating)					
1	CS-ATF-Warp-0.1 mm RC-01	25.18 x 2.61	4.918	22.36	340.23
2	CS-ATF-Warp-0.1 mm RC-02	25.22 x 2.58	4.682	21.43	329.43
3	CS-ATF-Warp-0.1 mm RC-03	25.19 x 2.49	4.697	22.43	357.57

(With 0.2 mm Resin Coating)					
1	CS-ATF-Warp-0.2 mm RC-01	25.26 x 2.65	4.638	22.60	337.65
2	CS-ATF-Warp-0.2 mm RC-02	25.31 x 2.68	4.832	24.47	360.79
3	CS-ATF-Warp-0.2 mm RC-03	25.221 x 2.73	4.163	23.97	348.23

The brief of test results of compressive strength and modulus (warp) for all three conditions of test specimens are tabulated in Table 6.9.

Table 6.9 Compressive strength and modulus (warp) results after ATF exposure

Test Specimens	Compr. Strength-without resin coating (MPa)	Compr. Modulus (GPa)	Compr. Strength-with 0.1 mm resin coating (MPa)	Compr. Modulus (GPa)	Compr. Strength-with 0.2 mm resin coating (MPa)	Compr. Modulus (GPa)
1	336.87	22.45	340.23	22.68	337.65	22.12
2	328.09	21.85	329.43	22.21	360.79	24.67
3	314.06	21.67	357.57	23.63	348.23	23.26
Mean	326.34	21.99	342.41	22.84	348.89	23.35
SD	11.51	0.41	14.20	0.72	11.58	1.28
% Degradation	11.43%	9.81%	7.07%	6.32%	5.31%	4.23%

After obtaining the compressive strength and modulus (warp) values for all three conditions of the test specimens, the average value is to be calculated and tabulated as follows in Table 6.10 and that has to be compared with the average value of as-received specimens.

Table 6.10 Effect of ATF exposure on compressive properties of E-glass/epoxy composite in the warp direction

Sample condition	Average compressive strength (warp) (MPa)		Average compressive modulus (warp) (GPa)	
	As-received	After ATF exposure	As-received	After ATF exposure
Bare samples	368.46	326.34	24.38	21.99
0.1 mm resin coated		342.41		22.84
0.2 mm resin coated		348.89		23.35

The compressive test specimens in the warp direction were tested experimentally and it was observed that compressive strength was reduced by 11.43%, 7.07%, and 5.31% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 9.81%, 6.32%, and 4.23% degradation in terms of compressive modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively.

The comparison of average compressive properties (warp) of as-received specimens with respect to bare and resin-coated specimens, was represented in the form of a bar chart as depicted in Fig. 6.6.

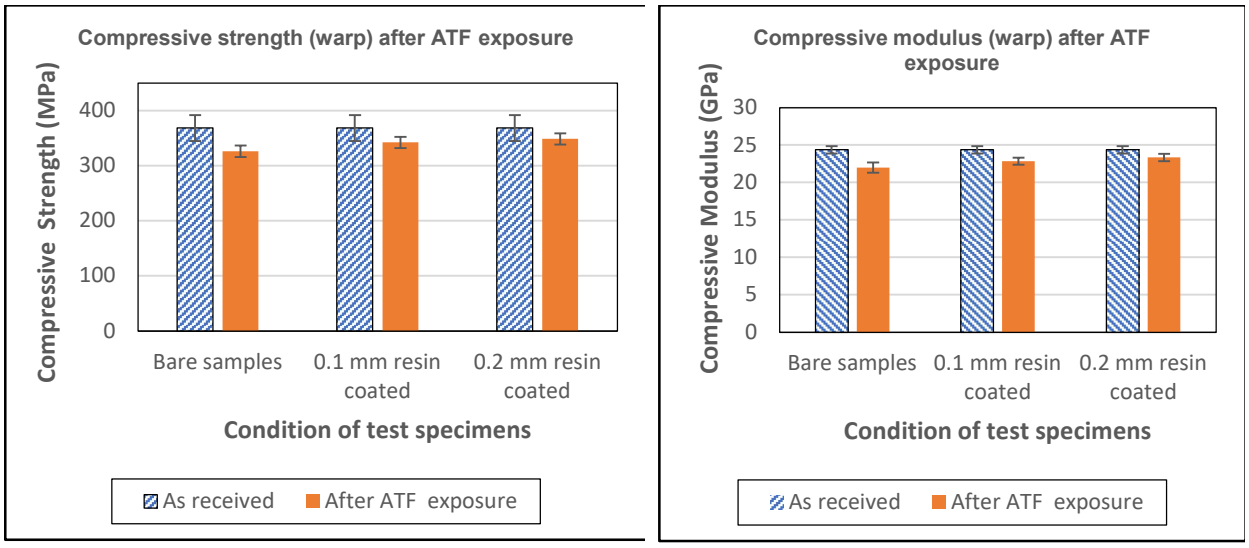


Fig. 6.6 Comparison chart of compressive properties (warp) for as-received specimens with bare and resin-coated specimens after ATF exposure.

ATF exposed compressive test samples (warp) after testing are depicted in Fig. 6.7, as follows.

Fig. 6.7 Compressive strength (warp) test samples after ATF exposure (after testing)

Similarly, the compressive test specimens (weft) for all three conditions were made and saturated under ATF exposure, where it was seen that bare specimens were saturated first, followed by with 0.1 mm resin coated, and last with 0.2 mm resin-coated specimens. Test samples got saturated with ATF, were taken out, and its surface dried up with tissue paper. After this room temperature curable E-glass/epoxy tabs and strain gauges were bonded as shown in Fig.6.8, in similar fashion as it was done for warp specimens.

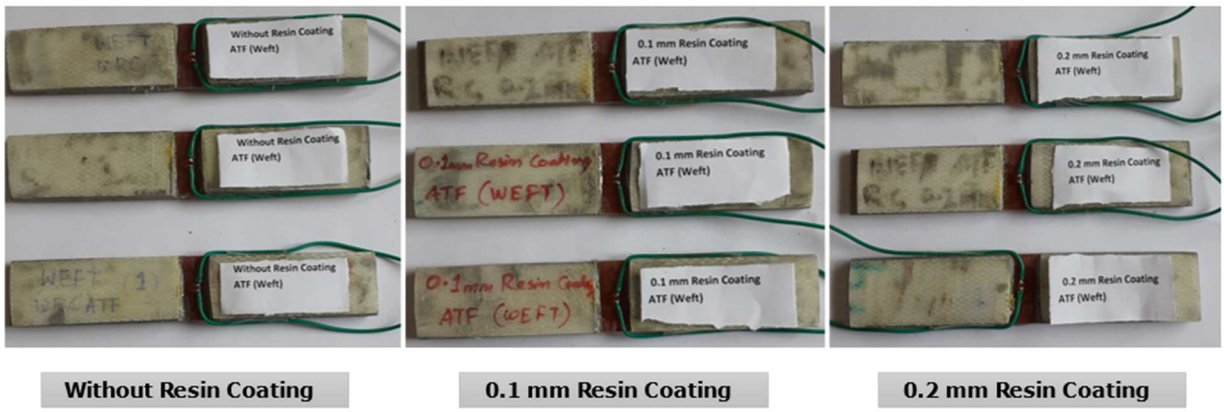


Fig. 6.8 Test samples of compressive strength (weft) after ATF exposure (before testing)

The compressive specimens will undergo mechanical testing using UTM with specially designed wedge-type fixtures. The results of the compression test (weft) were as follows (Table 6.11).

Table 6.11 Compressive test specimens (weft) results after ATF exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Compressive Strength (MPa)
(Without Resin Coating)					
1	CS-ATF-Weft-WRC-01	25.04 x 2.42	4.405	18.53	305.83
2	CS-ATF-Weft-WRC-02	25.11 x 2.47	4.207	19.73	318.09
3	CS-ATF-Weft-WRC-03	25.16 x 2.38	4.643	19.47	325.10
(With 0.1 mm Resin Coating)					
1	CS-ATF-Weft-0.1 mm RC-01	25.18 x 2.56	4.164	20.27	314.41
2	CS-ATF-Weft-0.1 mm RC-02	25.23 x 2.61	4.487	21.94	333.12
3	CS-ATF-Weft-0.1 mm RC-03	25.19 x 2.63	4.326	22.33	337.01
(With 0.2 mm Resin Coating)					
1	CS-ATF-Weft-0.2 mm RC-01	25.38 x 2.64	3.956	21.54	321.53
2	CS-ATF-Weft-0.2 mm RC-02	25.31 x 2.67	3.940	23.29	344.67
3	CS-ATF-Weft-0.2 mm RC-03	25.41 x 2.71	3.811	22.85	331.78

The brief of test results of compressive strength and modulus (weft) for all three conditions of test specimens are tabulated in Table 6.12.

Table 6.12 Compressive strength and modulus (weft) results after ATF exposure

Test Specimens	Compr. Strength-without resin coating (MPa)	Compr. Modulus (GPa)	Compr. Strength-with 0.1 mm resin coating (MPa)	Compr. Modulus (GPa)	Compr. Strength-with 0.2 mm resin coating (MPa)	Compr. Modulus (GPa)
1	305.83	21.12	314.41	21.24	321.53	22.13
2	318.09	21.79	333.12	22.87	344.67	24.56
3	325.10	23.54	337.01	24.56	331.78	23.12

Mean	316.34	22.15	328.18	22.89	332.66	23.27
SD	9.75	1.25	12.08	1.66	11.60	1.22
% Degradation	9.08%	7.93%	5.65%	4.87%	4.36%	3.28%

Once the compressive strength and modulus (weft) values were obtained for all three conditions of specimens the average value is to be calculated and tabulated as follows in Table 6.13 and that has to be compared with the average value of as-received specimens.

Table 6.13 Effect of ATF exposure on compressive properties of E-glass/epoxy composite in the weft direction

Sample condition	Average compressive strength (weft) (MPa)		Average compressive modulus (weft) (GPa)	
	As-received	After ATF exposure	As-received	After ATF exposure
Bare samples	347.83	316.24	24.06	22.15
0.1 mm resin coated		328.18		22.89
0.2 mm resin coated		332.66		23.27

However, the compressive test specimens in the weft direction were tested experimentally for bare and with 0.1 mm and 0.2 mm resin coated condition, and correlation of data was made with as-received specimen property. It was observed that compressive strength was reduced by 9.08%, 5.65%, and 4.36% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 7.93%, 4.87% and 3.28% degradation in terms of compressive modulus were observed for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively. The comparison of average compressive properties (weft) of as-received specimens with respect to bare and resin-coated specimens, was represented in the form of a bar chart as depicted in Fig.6.9.

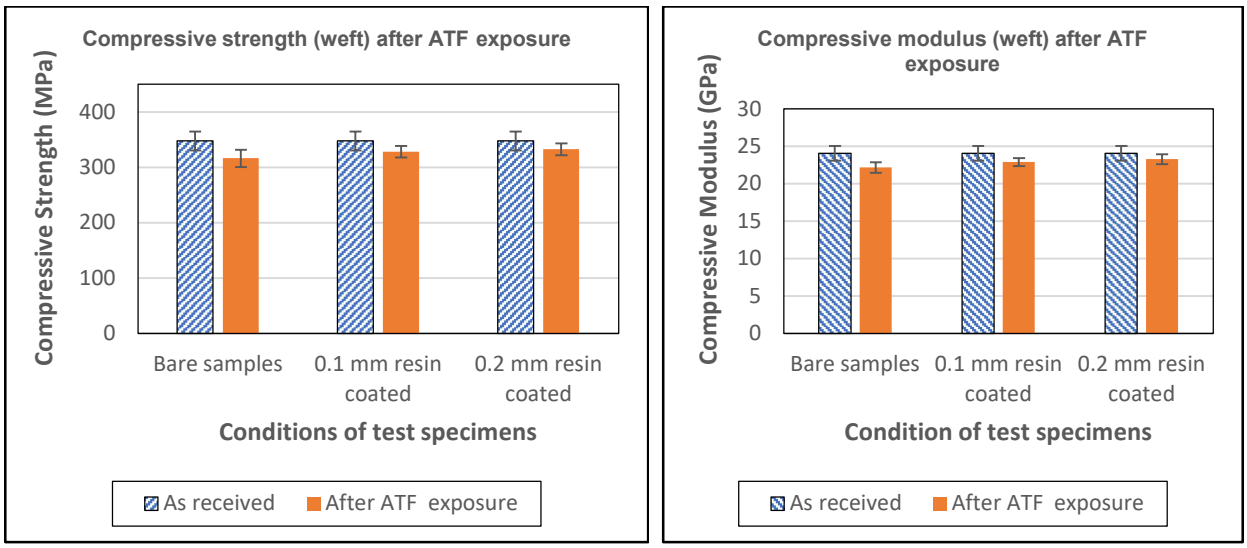


Fig. 6.9 Comparison chart of compressive properties (weft) for as-received specimens with bare and resin-coated specimens after ATF exposure

ATF exposed compressive test samples (weft) after testing are depicted in Fig. 6.10, as follows.

Fig. 6.10 Compressive strength (weft) test samples after ATF exposure (after testing)

The degradation of compressive properties in warp and weft direction after ATF exposure, are tabulated as follows in Table 6.14.

Table 6.14 Effect of ATF exposure on the degradation of compressive properties of E-glass/epoxy composite in warp and weft direction

Sample condition	Degradation of Compressive Strength (%)		Degradation of Compressive Modulus (%)	
	Warp	Weft	Warp	Weft
Bare samples	11.43	9.08	9.81	7.93
0.1 mm resin coated	7.07	5.65	6.32	4.87
0.2 mm resin coated	5.31	4.36	4.23	3.28

6.1.2.3 Test results of Flexural Test Specimens

Flexural test specimens (warp) were saturated for all three specimens conditions where bare specimens were saturated first, followed by with 0.1 mm resin coated, and last with 0.2 mm resin coated specimens. After this, the flexural test specimens will undergo mechanical testing using UTM with three-point bending fixture. The results of the flexural test (warp) were as follows (Table 6.15).

Table 6.15 Flexural test specimens (warp) results after ATF exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Flexural Strength (MPa)
(Without Resin Coating)					
1	FS-ATF-Warp-WRC-01	12.74 x 2.09	2.721	0.631	578.67
2	FS-ATF-Warp-WRC-02	12.76 x 1.99	2.610	0.587	592.34
3	FS-ATF-Warp-WRC-03	12.69 x 2.11	2.562	0.644	581.47
4	FS-ATF-Warp-WRC-04	-	-	-	slipped
(With 0.1 mm Resin Coating)					
1	FS-ATF-Warp-0.1 mm RC-01	12.79 x 2.18	2.65	0.717	601.56

2	FS-ATF-Warp-0.1 mm RC-02	12.76 x 2.14	2.311	0.704	614.24
3	FS-ATF-Warp-0.1 mm RC-03	12.81 x 2.19	2.637	0.721	598.21
4	FS-ATF-Warp-0.1 mm RC-04	12.71 x 2.07	2.345	0.665	623.23
(With 0.2 mm Resin Coating)					
1	FS-ATF-Warp-0.2 mm RC-01	12.78 x 2.21	2.681	0.745	608.23
2	FS-ATF-Warp-0.2 mm RC-02	12.83 x 2.25	2.36	0.794	623.34
3	FS-ATF-Warp-0.2 mm RC-03	12.73 x 2.16	2.34	0.747	641.12
4	FS-ATF-Warp-0.2 mm RC-04	12.87 x 2.27	2.493	0.849	653.36

The brief of test results of flexural strength and modulus (warp) for all three conditions of test specimens are tabulated in Table 6.16.

Table 6.16 Flexural strength and modulus (warp) after ATF exposure

Test Specimens	Flexural Strength-without resin coating (MPa)	Flexural Modulus (GPa)	Flexural Strength-with 0.1 mm resin coating (MPa)	Flexural Modulus (GPa)	Flexural Strength-with 0.2 mm resin coating (MPa)	Flexural Modulus (GPa)
1	578.67	21.7	601.56	21.45	608.23	21.95
2	592.34	22.85	614.24	22.66	623.34	22.92
3	581.47	21.93	598.21	22.41	641.12	23.54
4	slipped	Slipped	623.23	23.22	653.36	23.34
Mean	584.16	22.16	609.31	22.44	631.51	22.94
SD	7.22	0.61	11.57	0.74	19.82	0.71
% Degradation	12.91%	5.62%	9.16%	4.42%	5.85%	2.30%

Once the flexural strength and modulus (warp) values were obtained for all three conditions of specimens the average value is to be calculated and tabulated as follows in Table 6.17 and that has to be compared with the average value of as-received specimens.

Table 6.17 Effect of ATF exposure on flexural properties of E-glass/epoxy composite in the warp direction

Sample condition	Average flexural strength (warp) (MPa)		Average flexural modulus (warp) (GPa)	
	As-received	After ATF exposure	As-received	After ATF exposure
Bare samples	670.75	584.16	23.48	22.16
0.1 mm resin coated		609.31		22.44
0.2 mm resin coated		631.51		22.94

The flexural test specimens in the warp direction were tested experimentally and it was noticed that flexural strength was reduced by 12.91%, 9.16%, and 5.85% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 5.62%, 4.42%, and 2.30% degradation in terms of flexural modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively as depicted in Fig. 6.11.

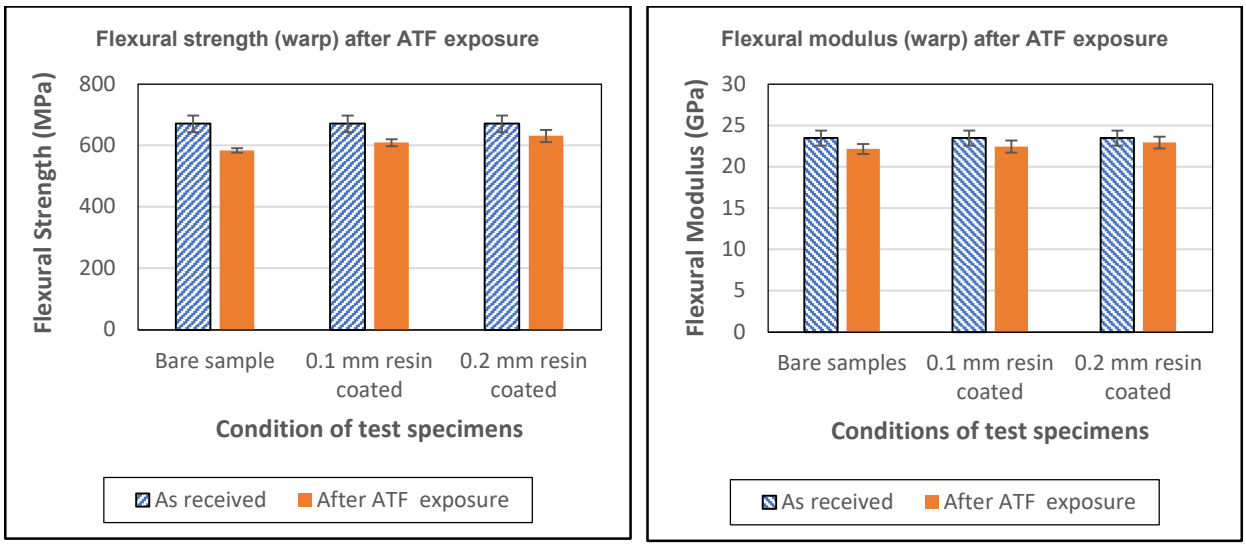


Fig. 6.11 Comparison chart of flexural properties (warp) for as-received specimens with bare and resin-coated specimens after ATF exposure

Similarly flexural test specimens (weft) were also undergone saturation in ATF environments and got tested and results are mentioned in Table 6.18.

Table 6.18 Flexural test specimens (weft) results after ATF exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Flexural Strength (MPa)
(Without Resin Coating)					
1	FS-ATF-Weft-WRC-01	12.68 x 2.08	2.754	0.607	564.11
2	FS-ATF-Weft-WRC-02	12.64 x 1.96	2.236	0.531	557.34
3	FS-ATF-Weft-WRC-03	12.58 x 2.11	2.538	0.582	530.07
4	FS-ATF-Weft-WRC-04	12.61 x 2.11	2.538	0.572	519.43
(With 0.1 mm Resin Coating)					
1	FS-ATF-Weft-0.1 mm RC -01	12.79 x 2.08	2.744	0.61	562.57
2	FS-ATF-Weft-0.1 mm RC -02	12.82 x 2.15	2.592	0.684	588.89
3	FS-ATF-Weft-0.1 mm RC -03	12.86 x 2.18	2.823	0.693	578.38
4	FS-ATF-Weft-0.1 mm RC -04	12.79 x 2.11	2.55	0.603	543.97
(With 0.2 mm Resin Coating)					
1	FS-ATF-Weft-0.2 mm RC -01	12.91 x 2.27	2.637	0.781	598.39
2	FS-ATF-Weft-0.2 mm RC -02	12.94 x 2.30	2.349	0.838	624.67
3	FS-ATF-Weft-0.2 mm RC -03	12.84 x 2.28	2.793	0.749	572.43
4	FS-ATF-Weft-0.2 mm RC -04	12.82 x 2.21	2.564	0.67	545.71

The brief of test results of flexural strength and modulus (weft) for all three conditions of test specimens are tabulated in Table 6.19.

Table 6.19 Flexural strength and modulus (weft) after ATF exposure

Test Specimens	Flexural Strength-without resin coating (MPa)	Flexural Modulus (GPa)	Flexural Strength-with 0.1 mm resin coating (MPa)	Flexural Modulus (Gap)	Flexural Strength-with 0.2 mm resin coating (MPa)	Flexural Modulus (GPa)
1	564.11	22.1	562.57	21.62	598.39	22.26
2	557.34	21.61	588.89	22.46	624.67	23.22
3	530.07	21.45	578.38	21.97	572.43	21.73
4	519.43	20.71	543.97	21.39	545.71	21.51
Mean	542.74	21.47	568.45	21.47	585.3	22.18
SD	20.4	0.58	19.58	0.58	33.93	0.76
% Degradation	12.39%	5.03%	8.24%	5.03%	5.52%	1.92%

The average flexural properties in the weft direction before (as-received) and after ATF exposure were calculated and are mentioned in Table 6.20.

Table 6.20 Effect of ATF exposure on flexural properties of E-glass/epoxy composite in the weft direction

Sample condition	Average flexural strength (weft) (MPa)		Average flexural modulus (weft) (GPa)	
	As-received	After ATF exposure	As-received	After ATF exposure
Bare samples	619.50	542.74	22.61	21.47
0.1 mm resin coated		568.45		21.86
0.2 mm resin coated		585.30		22.18

The flexural test specimens in the weft direction were tested experimentally for bare and with 0.1 mm and 0.2 mm, resin coated conditions, and correlation of data was made with as-received specimen property. It was noticed that flexural strength was reduced by 12.39%, 8.24%, and 5.52% for bare, with 0.1 mm, and 0.2 mm resin coating, respectively, and 5.03%, 3.32%, and 1.92%

degradation in terms of flexural modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively as depicted in Fig. 6.12.

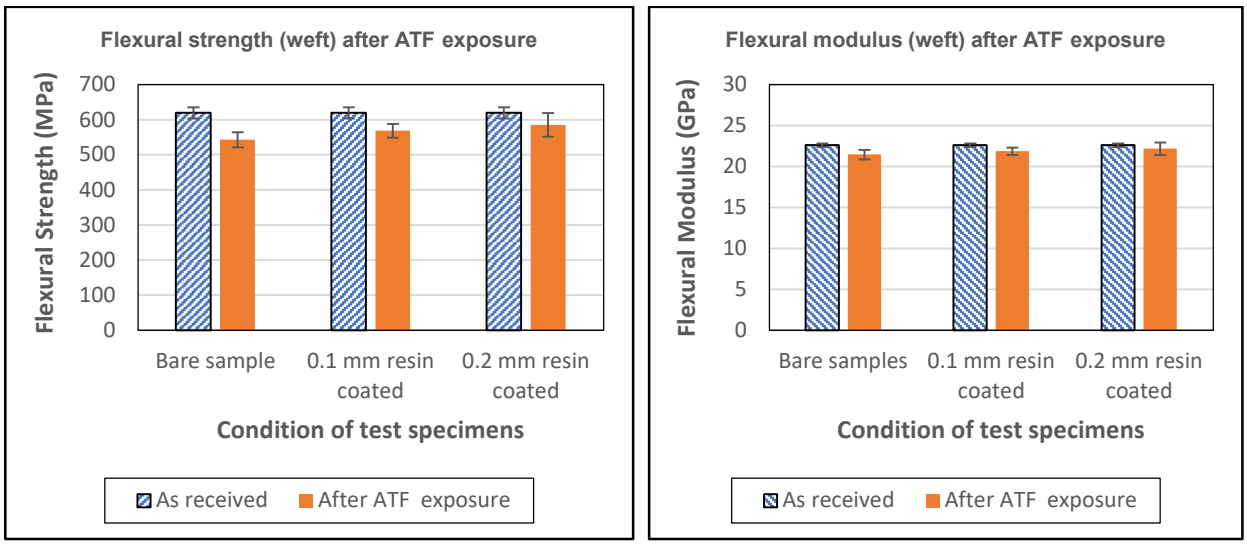
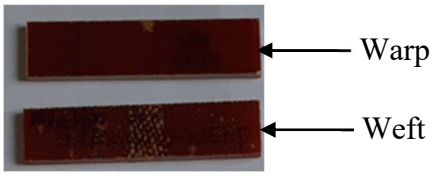
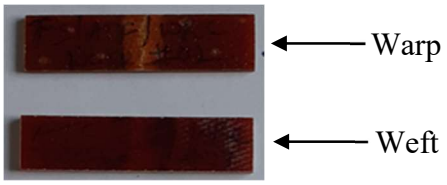


Fig. 6.12 Comparison chart of flexural properties (weft) for as-received specimens with bare and resin-coated specimens after ATF exposure

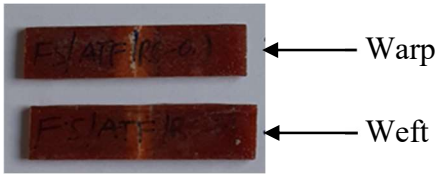
ATF exposed flexure test samples (warp and weft) after testing are depicted in Fig. 6.13, as follows.



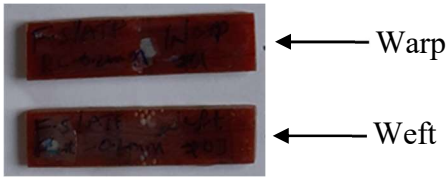
1. A bare specimen without exposure



2. A bare specimen without resin coating



3. Specimen with 0.1 mm resin coating



4. Specimen with 0.2 mm resin coating

Fig.6.13 Test specimens of flexural warp and weft (after testing)

The degradation of compressive properties in warp and weft direction after ATF exposure, are tabulated as follows in Table 6.21.

Table 6.21 Percentage (%) of degradation of flexural properties of E-glass/epoxy composite in warp and weft direction

Sample condition	Degradation of Flexural Strength (%)		Degradation of Flexural Modulus (%)	
	Warp	Weft	Warp	Weft
Bare samples	12.91	12.39	5.62	5.03
0.1 mm resin coated	`9.16	8.24	4.42	3.32
0.2 mm resin coated	5.85	5.52	2.30	1.92

6.1.2.4 Test results of ILSS Test Specimens

ILSS test specimens were saturated for all three specimen conditions and afterwards specimens will undergo mechanical testing using UTM with three-point bending fixture. The results of the ILSS test were as follows (Table 6.22).

Table 6.22 ILSS test specimens result after ATF exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	ILSS Strength (MPa)
(Without Resin Coating)					
1	ILSS-ATF-WRC-01	12.05 x 4.11	0.77	4.107	62.19
2	ILSS-ATF-WRC-02	12.19 x 4.1	0.72	3.987	59.83
3	ILSS-ATF-WRC-03	12.3 x 4.05	0.67	4.122	62.05
(With 0.1 mm Resin Coating)					
1	ILSS-ATF-0.1 mm RC-01	12.16 x 4.18	1.122	4.169	61.51
2	ILSS-ATF-0.1 mm RC-02	12.19 x 4.19	0.706	4.344	63.79
3	ILSS-ATF-0.1 mm RC-03	12.10 x 4.21	0.70	4.300	63.31
(With 0.2 mm Resin Coating)					
1	ILSS-ATF-0.2 mm RC-01	12.37 x 4.27	0.755	4.364	61.96

2	ILSS-ATF-0.2 mm RC-02	12.34 x 4.31	0.853	4.78	67.41
3	ILSS-ATF-0.2 mm RC-03	12.31 x 4.23	0.714	4.371	62.96

The brief of test results of interlaminar shear strength for all three conditions of test specimens are tabulated in Table 6.23.

Table 6.23 ILSS strength after ATF exposure

Test Specimens	ILSS Strength- without resin coating (MPa)	ILSS Strength- with 0.1 mm resin coating (MPa)	ILSS Strength- with 0.2 mm resin coating (MPa)
1	62.19	61.51	61.96
2	59.83	63.79	67.41
3	62.05	63.31	62.96
Mean	61.36	62.87	64.11
SD	1.32	1.2	2.9
% Degradation	10.04%	7.83%	6.01%

The average ILSS properties before (as-received) and after ATF exposure are as mentioned in Table 6.24.

Table 6.24 Effect of ATF exposure on inter-laminar shear properties of E-glass/epoxy composite

Sample condition	Average Interlaminar shear strength (MPa)	
	As-received	After ATF exposure
Bare samples	68.21	61.36
0.1 mm resin coated		62.87
0.2 mm resin coated		64.11

The interlaminar shear test specimens were tested experimentally for bare and with 0.1 mm and 0.2 mm resin-coated conditions and a correlation of data was made with the as-received specimen property. It was observed that interlaminar shear strength was reduced by 10.04%, 7.83%, and 6.01 % for bare, with 0.1 mm and 0.2 mm resin coating, respectively as depicted in Fig.6.14.

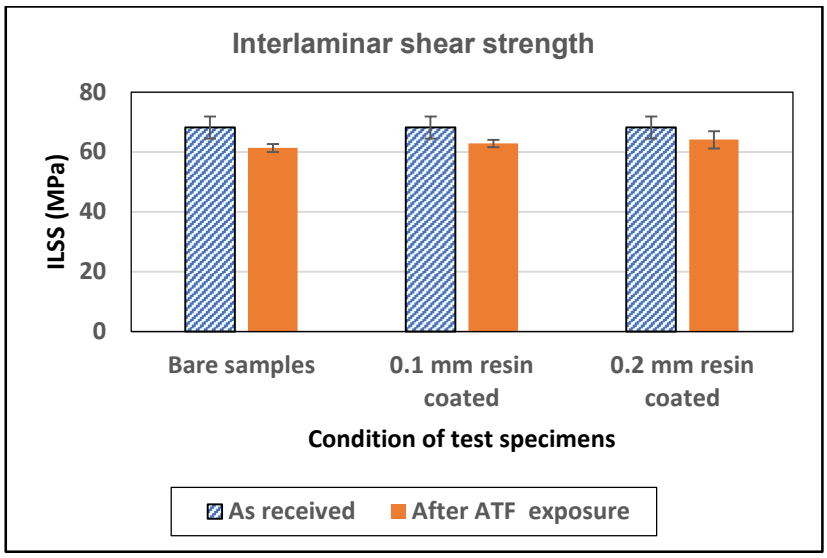


Fig. 6.14 Comparison chart of ILSS properties for as-received specimens with bare and resin-coated specimens after ATF exposure

6.1.2.5 Test results of IPSS Test Specimens

The IPSS test specimens for all three specimen conditions of were saturated and got tested. The results of the IPSS test were as follows (Table 6.25).

Table 6.25 IPSS test specimens result after ATF exposure

Sl No.	Test specimen identification	Test specimens dimension (mm)	Max. Displacement (mm))	Max. load (kN)	IPSS Strength (MPa)
(Without Resin Coating)					
1	IPSS-ATF-WRC-01	25.02 x 1.79	31.95	8.240	92
2	IPSS-ATF-WRC-02	24.93 x 1.81	24.927	7.129	79
3	IPSS-ATF-WRC-03	24.97 x 1.78	21.728	6.934	78
4	IPSS-ATF-WRC-04	25.05 x 1.77	30.793	7.892	89
(With 0.1 mm Resin Coating)					
1	IPSS-ATF-0.1 mm RC-01	25.14 x 1.97	29.733	9.707	98
2	IPSS-ATF-0.1 mm RC-02	25.18 x 1.95	24.008	8.249	84
3	IPSS-ATF-0.1 mm RC-03	25.11 x 2.01	30.812	8.782	87

4	IPSS-ATF-0.1 mm RC-04	25.16 x 1.98	23.70	8.270	83
(With 0.2 mm Resin Coating)					
1	IPSS-ATF-0.2 mm RC-01	25.34 x 2.16	28.892	10.509	96
2	IPSS-ATF-0.2 mm RC-02	25.29 x 2.15	29.84	10.222	94
3	IPSS-ATF-0.2 mm RC-03	25.36 x 2.19	28.141	9.441	85
4	IPSS-ATF-0.2 mm RC-04	25.31 x 2.17	-	-	slipped

The brief of test results of in-plane shear strength for all three conditions of test specimens are tabulated in Table 6.26.

Table 6.26 IPSS strength after ATF exposure

Test Specimens	IPSS Strength-without resin coating (MPa)	In-plane Shear Modulus (GPa)	IPSS Strength-with 0.1 mm resin coating (MPa)	In-plane Shear Modulus (GPa)	IPSS Strength-with 0.2 mm resin coating (MPa)	In-plane Shear Modulus (GPa)
1	92	3.93	98	4.14	96	4.03
2	79	3.61	84	3.63	94	3.89
3	78	3.41	87	3.83	85	3.69
4	89	3.81	83	3.48	slipped	slipped
Mean	84.5	3.69	88	3.77	91.67	3.87
SD	7.05	0.228	6.88	0.29	5.86	0.17
% Degradation	14.75%	10.87%	11.22%	8.94%	7.52%	6.52%

The average IPSS properties before (as-received) and after ATF exposure are mentioned in Table 6.27.

Table 6.27 Effect of ATF exposure on in-plane shear properties of E-glass/epoxy composite

Sample condition	Average In-plane shear strength (MPa)		Average In-plane shear modulus (GPa)	
	As-received	After ATF exposure	As-received	After ATF exposure
Bare samples	99.12	84.5	4.14	3.69
0.1 mm resin coated		88.0		3.77
0.2 mm resin coated		91.67		3.87

The in-plane shear specimens were tested experimentally for bare and with 0.1 mm and 0.2 mm resin-coated conditions and a correlation of data was made with the as-received specimen property. It was noticed that in-plane shear strength was reduced by 14.75%, 11.22%, and 7.52 % for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 10.87%, 8.94%, and 6.52% degradation in terms of in-plane shear modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively as depicted in Fig.6.15.

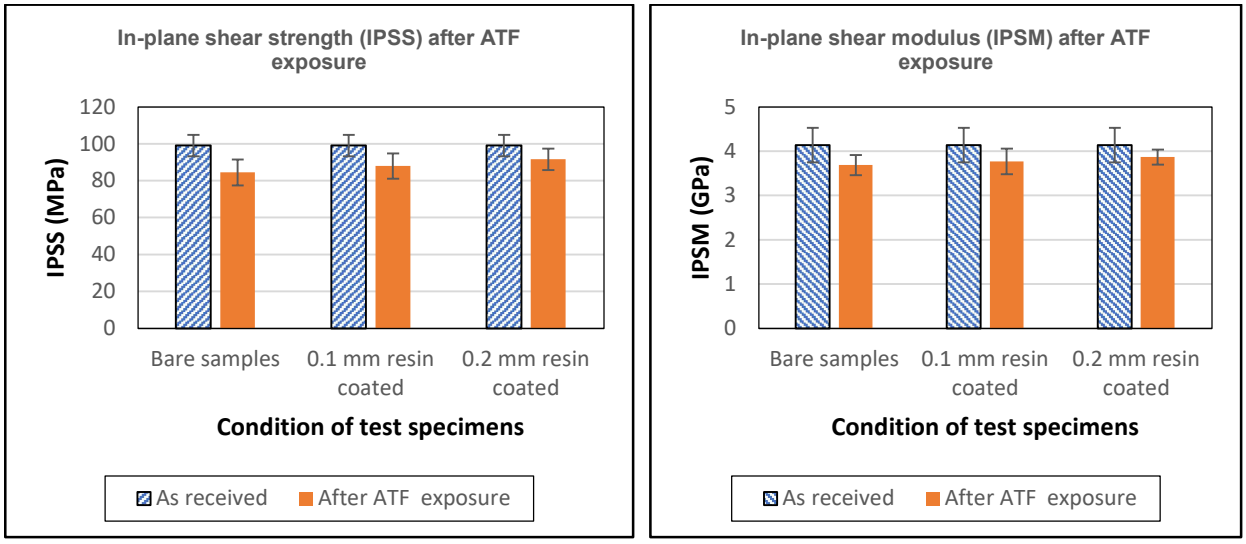


Fig. 6.15 Comparison chart of IPSS properties for as-received specimens with bare and resin-coated specimens after ATF exposure

ATF exposed IPSS test samples after testing are depicted in Fig. 6.16, as follows.

Fig. 6.16 IPSS test specimens after ATF exposure (after testing)

6.1.3 Microstructural Examination of ATF Exposed Samples

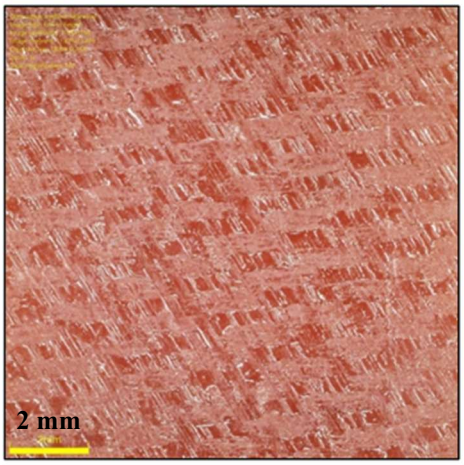
To know the degradation mechanism, the tested samples of E-glass/epoxy composite after ATF exposure, were surface examined using stereo microscope and sub-surface examination (fractured surface) was carried out using SEM as follows. This data was helpful to draw the conclusions for failure characteristics and associated degradation mechanism of tested samples.

6.1.3.1 Stereo Microscope Examination to Test Samples Exposed to ATF

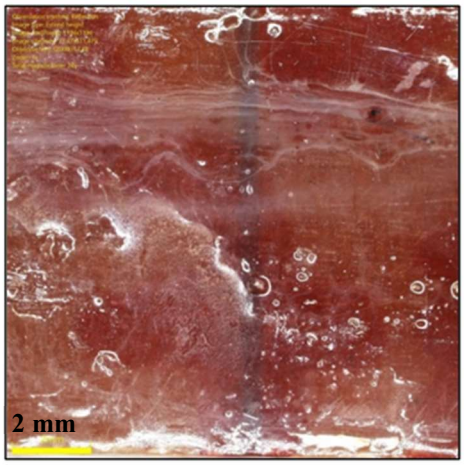
The tested samples of E-glass/epoxy composite after ATF exposure, was surface examined under a stereo microscope (Olympus DSX110) as depicted in Fig. 6.17 and 6.18. The test specimens that underwent investigation were ATF-exposed ones. It was observed that the test specimen coated with 0.2 mm resin coating has undergone the lowest fuel ingression relative to the bare specimen and epoxy coating is providing, one kind of impermeable and protective layer which was fuel ingression resistant as depicted below in the figures. Resin coating with 0.1 mm were showing small fuel ingression patches, because of the lesser thickness of the coating. Table 6.28. as below is provided, for giving an explicit idea about the conditions of the test specimens.

Table 6.28 Bare and resin coated test specimens of different type of tests, exposed to ATF

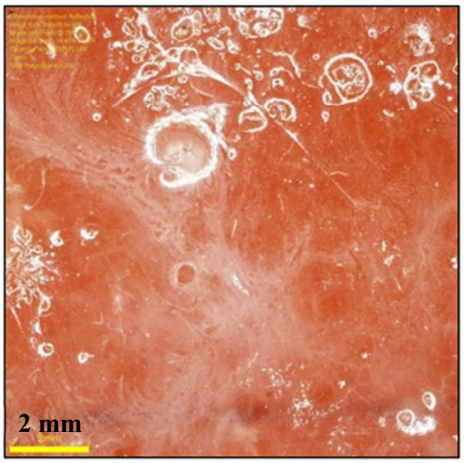
Sl No.	Name of test specimens	Condition of specimens	Remarks if any
a	Tensile (warp)	WRC (Without resin coating)	Bare specimen
b	Tensile (warp)	0.1mm RC (0.1 mm resin coating)	Resin coated
c	Tensile (warp)	0.2mm RC (0.2 mm resin coating)	Resin coated
d	ILSS (Top)	0.1mm RC (0.1 mm resin coating)	Resin-coated top surface
e	ILSS (Edge)	0.1mm RC (0.1 mm resin coating)	Resin coated Edge surface
f	ILSS (Tested Samples)	0.1mm RC (0.1 mm resin coating)	Failure zone after ILSS testing
g	ILSS (Top)	WRC (Without resin coating)	Bare specimen top surface
h	ILSS (Edge)	WRC (Without resin coating)	Bare specimen Edge surface
i	ILSS (Tested samples, bottom surface)	WRC (Without resin coating)	Bare specimen bottom surface
j	Compression (warp)	WRC (Without resin coating)	Bare specimen
k	Compression (warp)	0.1mm RC (0.1 mm resin coating)	Resin coated
l	Compression (warp)	0.2 mm RC (0.2 mm resin coating)	Resin coated



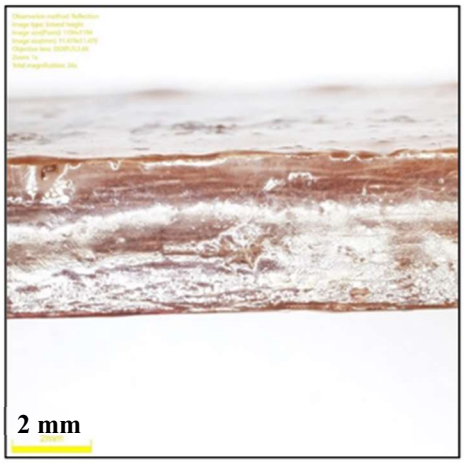
a) Tensile- WARP- WRC



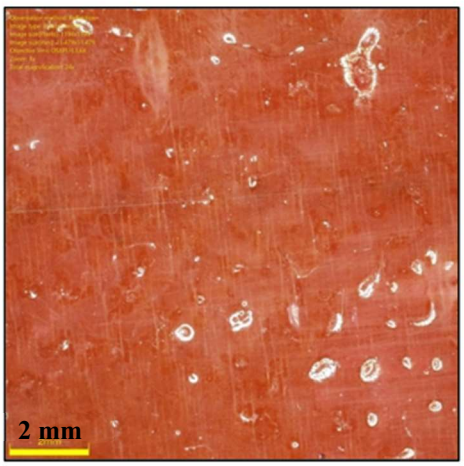
d) ILSS- TOP- 0.1 mm RC



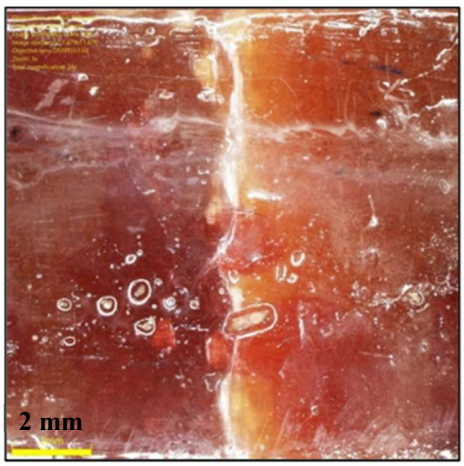
b) Tensile- WARP- 0.1 mm RC



e) ILSS- EDGE- 0.1 mm RC

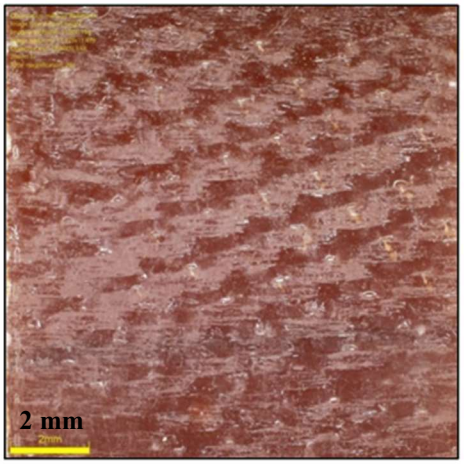


c) Tensile- WARP- 0.2 mm RC

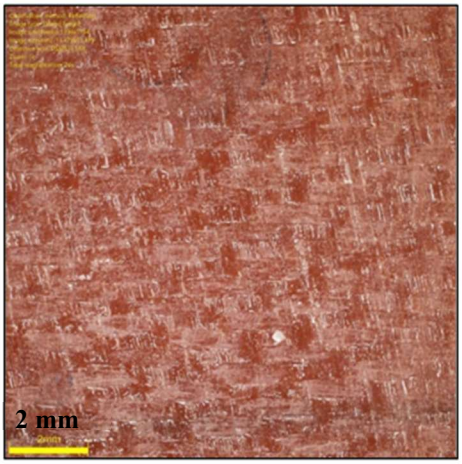


f) ILSS- Tested Sample- 0.1 mm RC

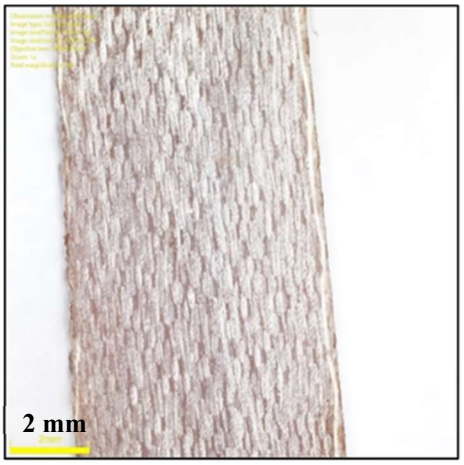
Fig. 6.17 Stereo microscope examinations of Tensile and ILSS test samples exposed to ATF



g) ILSS- TOP- WRC

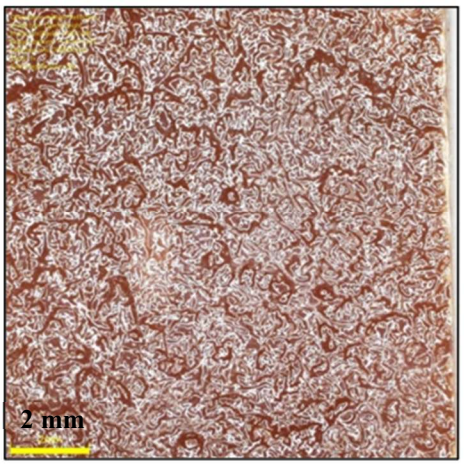


j) Compression- WARP- WRC

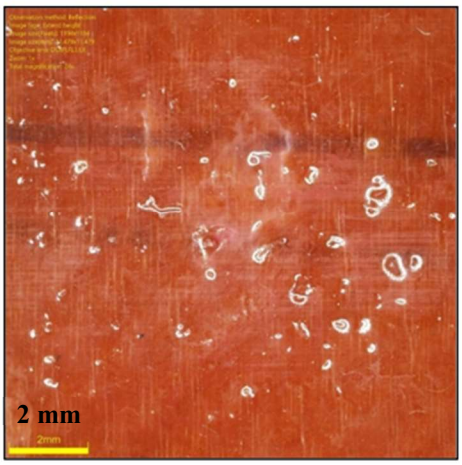


h) ILSS- EDGE- WRC

k) Compression- WARP- 0.1 mm RC



i) ILSS-BOT-WRC



j) Compression- WARP- 0.2 mm RC

Fig. 6.18 Stereo microscope examinations of ILSS and Compression test samples exposed to ATF

6.1.3.2 Microstructural Examination of Tested ATF Exposed Fractured Surface using SEM

A) Microstructural Examination of Tested ATF Exposed Fractured Surface using Scanning Electron Microscope (SEM)

The microstructural examination was carried out for bare specimens without ATF exposure, ATF-exposed bare specimens without resin coating, and resin-coated specimens. The test specimens like tensile, compressive, and IPSS were chosen for this investigation using SEM, as they all were having generating clean fractured surfaces to examine the fiber and its interfacial characteristics. The test specimens like ILSS and flexure were tested using a three-point bending fixture, exhibiting failure out of the plane of the loading and will not expose the fractured surface outside as it will be the sub-surface phenomenon. It was a bit difficult to capture the same using SEM so surficial characteristics of ILSS specimens were examined with SEM and the findings of the same are displayed here.

B) Microstructural Comparative Examination of the Fractured Surface with and without ATF-Exposed Tensile Test Specimens using SEM

The effect of ATF ingression in terms of degradation of tensile properties was notable. To compare and assess the damage mechanisms that originated in the fiber-matrix interface for different types of test specimens, before and after the ATF exposure, fractured fibers of the tested tensile (warp) specimens were investigated by using SEM. Fractures fibers of tensile test specimens were examined before and after ATF exposure and it was noticed that bare tested specimens exhibited maximum degradation compared to resin-coated ones. SEM micrographs revealed that bare specimens underwent matrix cracking, formation of microvoids, crumbling, and debonding on the matrix fiber interface as shown below in the figures, whereas the resin-coated tested specimens were limiting the ATF ingression, which gave relatively lower reduction as depicted in Fig.6.19 (a), (b), (c) and (d).

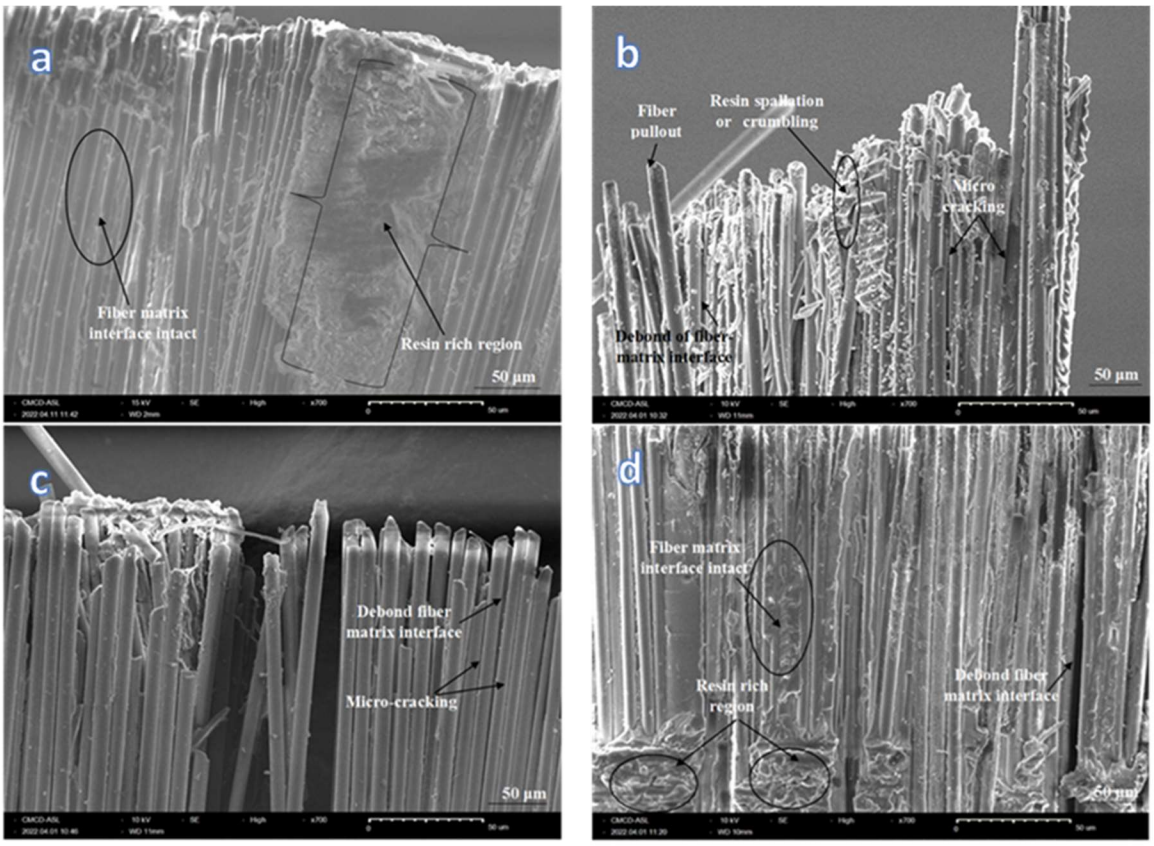


Fig. 6.19 SEM micrographs of tensile (weft) fractured composite (a) bare specimen without ATF exposure, (b) bare specimen exposed to ATF, (c) 0.1 mm resin-coated specimen exposed to ATF, and (d) 0.2 mm resin coated specimen exposed to ATF

C) Microstructural Comparative Examination of the Fractured Surface with and without ATF-Exposed Compressive Test Specimens using SEM

The effect of ATF ingression in terms of degradation of compressive properties was significant. To compare and assess the damage mechanisms that originated in the fiber-matrix interface for different types of test specimens, before and after the ATF exposure, fractured fibers of the tested compressive (warp) specimens were investigated by using SEM. Fractures fibers of compressive test specimens were examined before and after ATF exposure and it was noticed here also that bare tested specimens exhibited maximum degradation compared to resin-coated ones. SEM micrographs revealed that bare specimens underwent matrix cracking, formation of micro voids, crumbling, and debonding on the matrix fiber interface as shown below in the figures, whereas the resin-coated tested specimens were limiting the ATF ingression, which gave relatively lower reduction as depicted in Fig.6.20 (a), (b), (c) and (d).

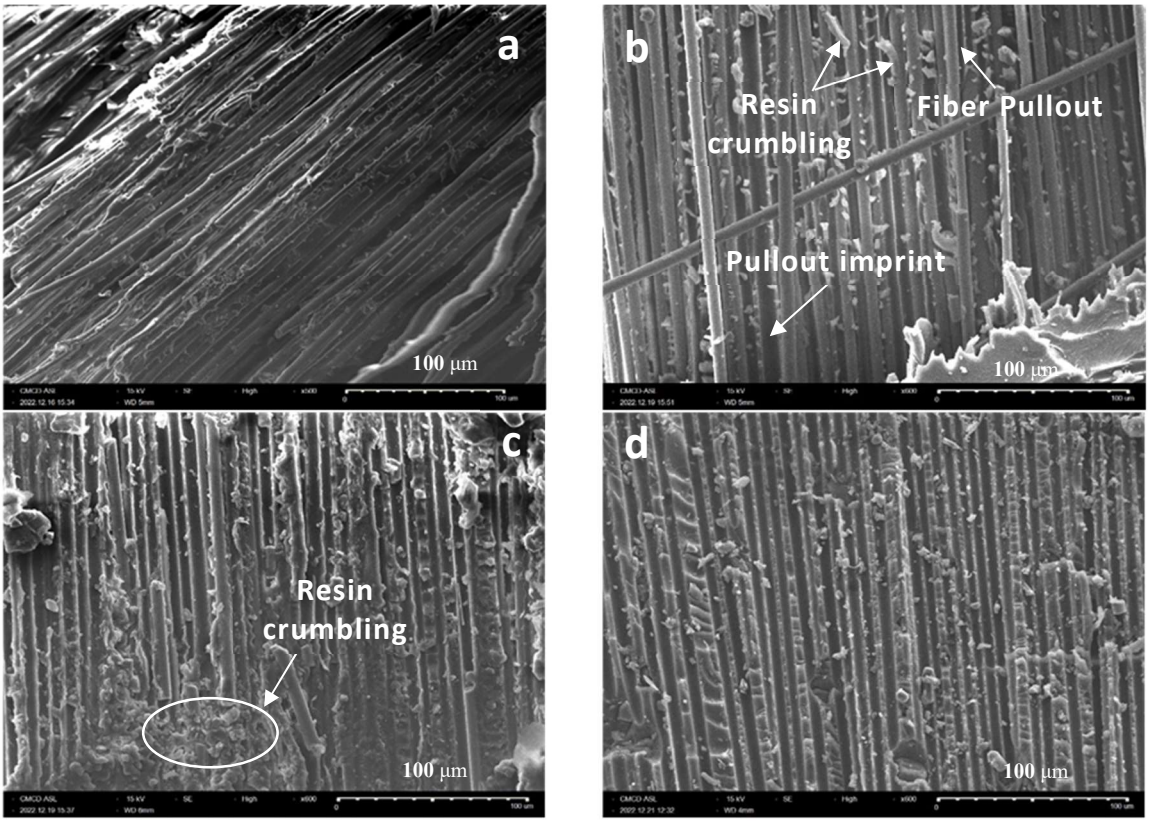


Fig.6.20 SEM micrographs of Compressive fractured composite (a) bare specimen without ATF exposure, (b) bare specimen exposed to ATF, (c) 0.1 mm resin coated specimen exposed to ATF, and (d) 0.2 mm resin coated specimen exposed to ATF

D) Microstructural Comparative Examination of the Fractured Surface with and without ATF-Exposed IPSS Test Specimens using SEM

Similarly fractures fibers of in-plane shear test specimens were examined before (Fig. 6.21 (a)) and after ATF exposure and it was noticed that bare-tested specimens exhibited maximum degradation compared to resin-coated ones. SEM micrographs revealed that bare specimens underwent matrix cracking, crumbling, and debonding on the matrix fiber interface as shown in Fig. 6.21 (b), whereas the resin-coated tested specimens were limiting the ATF ingression, which gave relatively lower reduction as depicted in Fig. 6.21 (c) and (d).

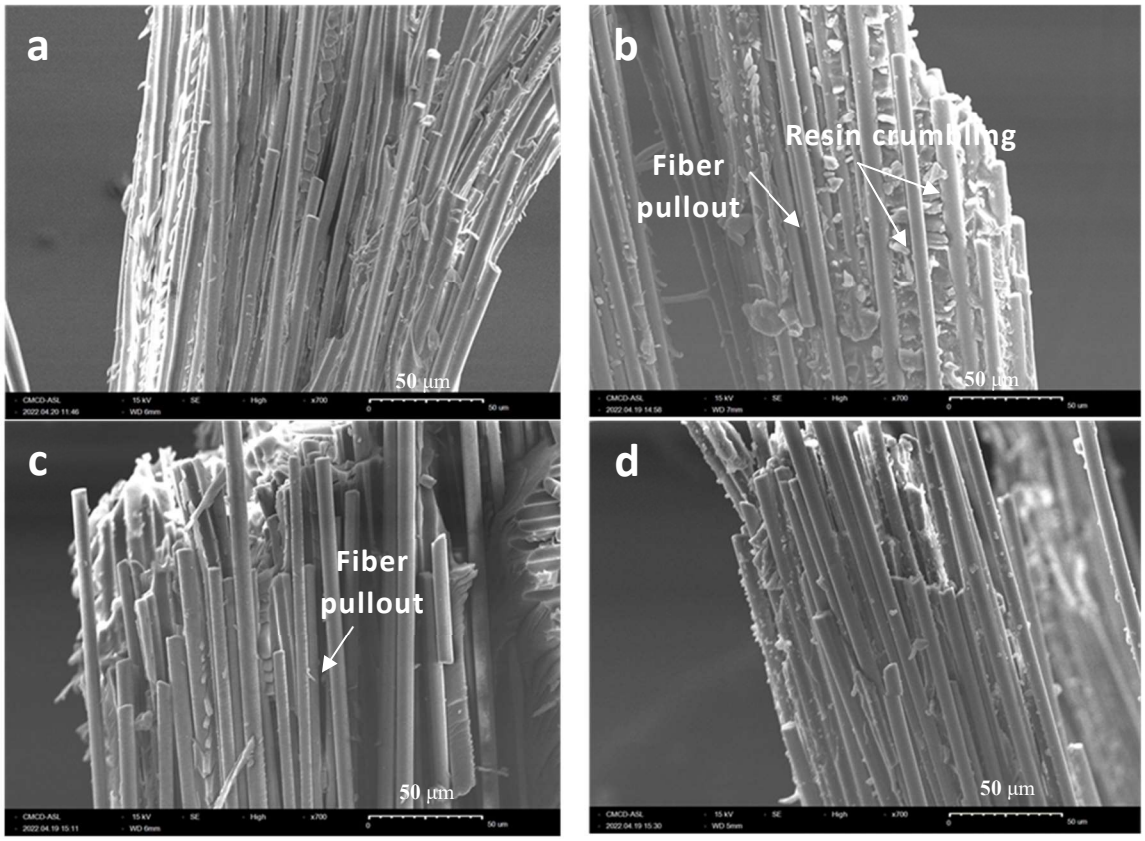


Fig. 6.21 SEM micrographs of IPSS fractured composite (a) bare specimen without ATF exposure, (b) bare specimen exposed to ATF, (c) 0.1 mm resin coated specimen exposed to ATF, and (d) 0.2 mm resin coated specimen exposed to ATF

E) Microstructural Comparative Surficial Examination with and without ATF-Exposed ILSS Test Specimens using SEM

The effect of ATF ingression in terms of degradation of interlaminar shear properties was notable. To compare and assess the damage mechanisms that originated in the fiber-matrix interface for different types of test specimens, before (Fig.6.22(a)) and after the ATF exposure, surficial examination of the tested interlaminar shear specimens was investigated by using SEM. The fractured surfaces of interlaminar shear strength (ILSS) test specimens were investigated using a scanning electron microscope (SEM). It was observed that fractured surfaces of bare ILSS test specimens underwent ATF exposure, and exhibits matrix cracking under different magnifications as shown in Fig.6.22 (b).

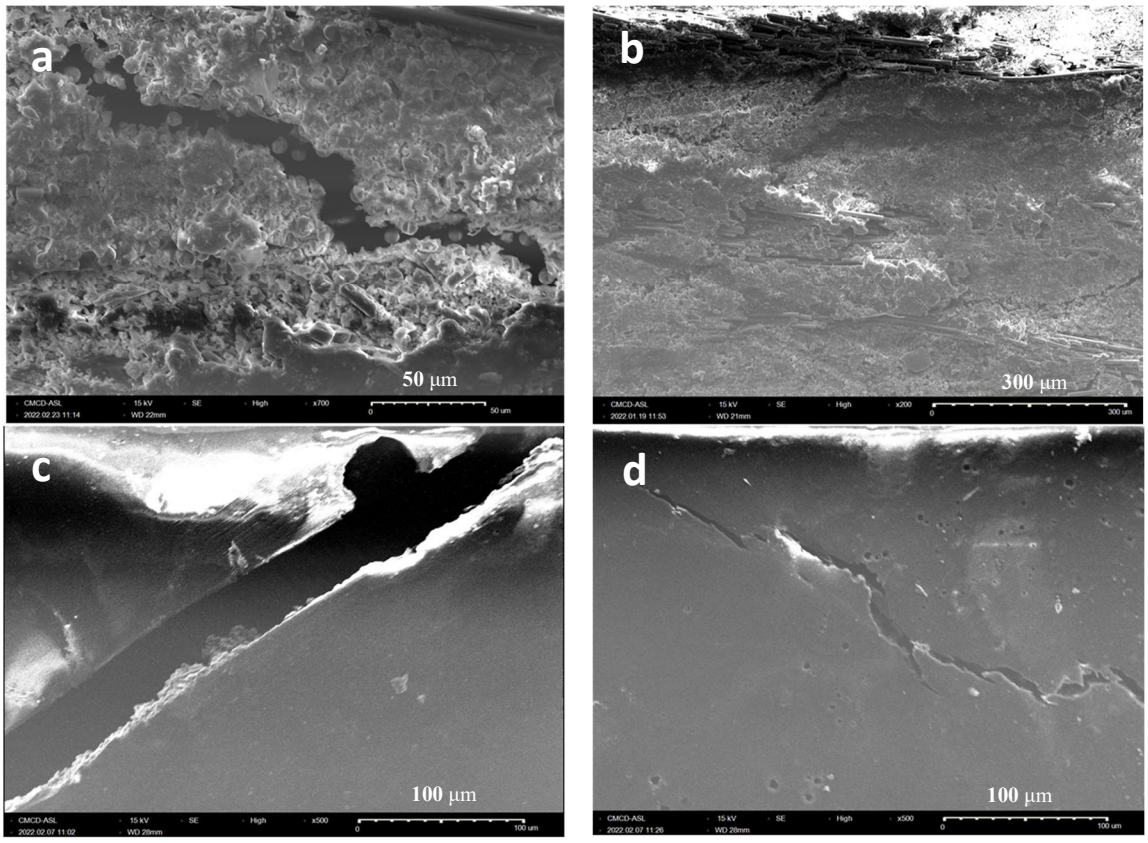


Fig. 6.22 SEM micrographs of ILSS tested samples of composite (a) bare specimen without ATF exposure, (b) bare specimen exposed to ATF (c) 0.1 mm resin coated specimen exposed to ATF, and (d) 0.2 mm resin coated specimen exposed to ATF

The test specimens which were coated with 0.1 and 0.2 mm resin coating also underwent SEM examinations and found that ATF exposure only degraded the resin coating on the surface and generated the crack, but it restricted the ATF ingression to test specimens. The size of the surface crack of the 0.1 mm resin-coated substrate was more than the 0.2 mm resin-coated one as shown in Fig. 6.22 (c) and (d).

6.1.4 Evaluation of Minimum coating thickness using Least Squares Quadratic Polynomial Approximation to Minimize the Effect of ATF Exposure

The normalization of the strength parameters approximated the tensile strength behavior in warp and weft direction and ILSS relatively well and may produce the nonlinear behavior of normalized strength parameters with respect to coating thickness, i.e., bare, 0.1 mm, and 0.2 mm resin coating over the test specimens. The curve was obtained by plotting the average

values of all the respective samples and determining the best-fit line through the data using least squares quadratic polynomial approximation. Normalized tensile strength parameters in warp and weft direction and ILSS were correlated with coating thickness and its behavioral pattern in the form of a curve were depicted in Fig. 6.23, 6.24 and 6.25 to establish the minimum coating thickness. This minimum coating thickness is the effective coating thickness required over the test specimens to reduce the fuel ingression and to keep the degradation due to ATF exposure to the minimum.

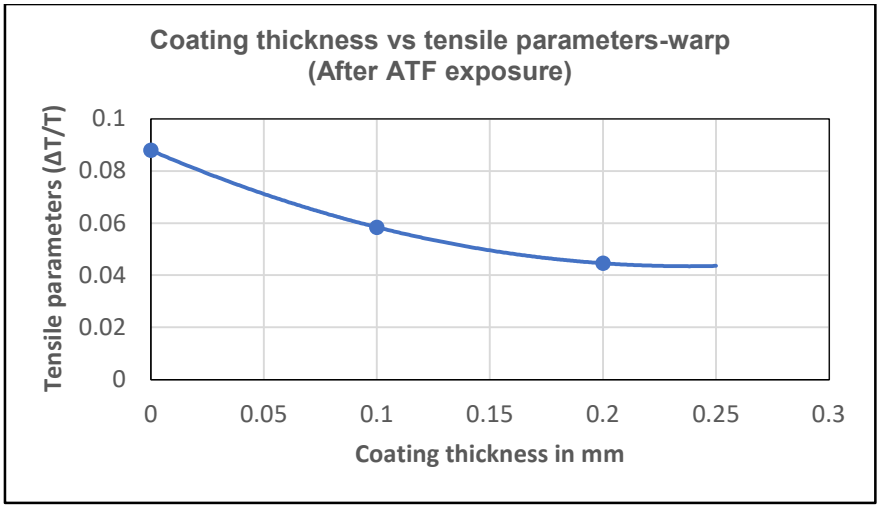


Fig. 6.23 Coating thickness vs tensile parameters-warp

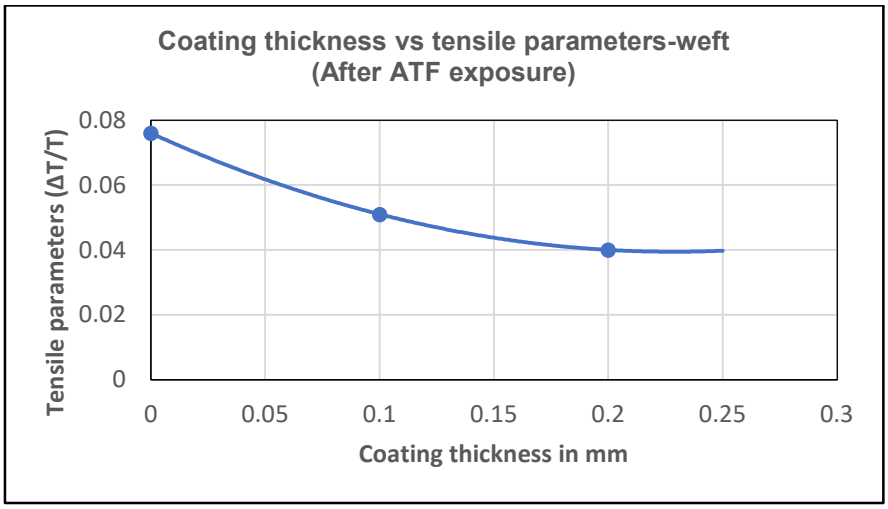


Fig. 6.24 Coating thickness vs tensile parameters-weft

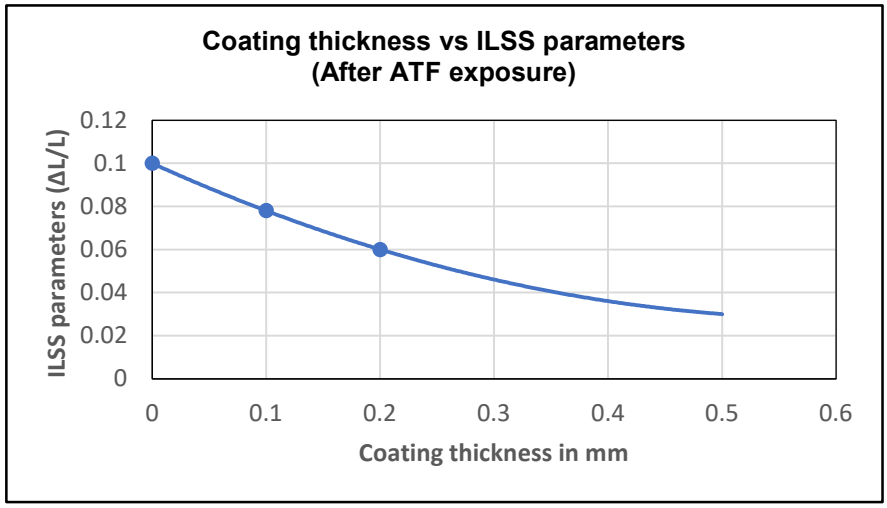


Fig. 6.25 Coating thickness vs ILSS parameters

Resin coat application is found, to help in minimizing the degradation of properties after ATF exposure. The plot of normalized tensile strength parameter ΔT/T (where ΔT is the difference between the values of average tensile strength before and after ATF exposure and T is the value of tensile strength before exposure) for bare and with resin coated specimens in warp and weft direction are shown. Similarly, normalized ILSS parameter ΔL/L (where ΔL is the difference between the values of average ILSS before and after ATF exposure and L is the value of ILSS before exposure) for bare and resin-coated specimens were plotted using least squares quadratic polynomial approximation. It is visible from the plot that the test specimen coated with 0.25 mm coating thickness will exhibit minimum degradation where tensile properties are design criteria unlike 0.5 mm coating thickness is required for shear properties.

6.1.5 Correlation of results with the Mechanism of Surface and Sub-surface Degradation of Different Types of Samples after ATF Exposure

- The tested specimens (bare condition), underwent ATF exposure exhibiting some reduction of tensile and flexural strength compared to as-received. It was observed from SEM's images, that for tested specimens (bare condition), the ATF ingression fuel penetrated to polymeric network and induces internal stresses and not only degrading interlaminar bonds but will create micro-voids too. The fractured surfaces were not clean, and it happened due

to fiber matrix interface degradation. This phenomenon is responsible for the degradation of tensile and flexural strength.

- The tested specimens (bare condition), underwent ATF exposure exhibiting some reduction of compression strength compared to as-received. It was observed from SEM's images too as these properties are matrix dominated, the fuel diffusion to the polymeric network will induce internal stresses that will not only generate micro-cracks but micro-voids formation too and this phenomenon will be responsible for its degradation.
- A slight reduction was observed in terms of tensile, compression, and flexural modulus too because once the test samples got immersed in ATF will drive the way for fuel molecules to enter into the polymeric network as the composite is porous, and because the capillary effect fuel ingresses. This kind of fuel diffusion will restrict the mobility of the polymeric network, resulting in brittle behavior [6]. This phenomenon will reduce the modulus.
- As epoxy are having long molecular chains which cross-linked with each other when it undergoes polymeric network formation, this ATF ingression increases the cross-linkage forces among the molecular polymeric chain between the resin and reinforcement. Fuel molecules that got penetrated a polymeric network caused the movement restriction of the polymeric chain as reported by Kumarasamy [6] and Genanu [78]. Thus, the material slightly lost its elasticity.
- The tested specimens (bare condition), underwent ATF exposure, exhibiting some reduction of interlaminar and in-plane shear strength compared to as-received. It was observed from SEM's images, that for tested specimens (bare condition), the ATF ingresses the polymer chain and generates internal stresses and it will not only degrade the fiber matrix adherence but will create micro-cracks too in the resin at interfaces. SEM Micrographs of fractured surfaces of the test specimens show that it was not clean, and fiber-matrix degradation was one of the reasons. Primarily, this is responsible for the reduction of interlaminar and in-plane shear strength.
- A slight reduction was observed in terms of in-plane shear modulus too, and this reduction is due to the increased cross-linkage force in the polymeric chain between the matrix and the fiber. The brittleness was exhibited for tested specimens, and it is due to the ATF molecule which ingresses into the polymeric network and restricted its mobility. Hence,

the composite will slightly lose its elasticity. A behavior similar to this was reported by Kumarasamy [6] and Genanu [78].

- Moreover, specimens with 0.1 mm and 0.2 mm resin coated exhibited a relatively slight decrement of the tensile, compression, flexural, and shear properties and not allowed significant penetration of fuel molecules inside the polymeric network. In addition, 0.2 mm resin coated were relatively more impermeable to fuel ingression than 0.1 mm resin-coated specimens, and ATF ingression was restricted to the top surface as shown in Fig.6.17 and Fig. 6.18 and prevented its penetration to fiber matrix interfaces as depicted in SEM micrographs of Fig.6.19, 6.20, 6.21 (c) and (d), which has further led to the only slight decrease in the properties.
- The E-glass/epoxy laminate in the weft direction exhibited less reduction compared to warp one as differing fill (weft) count and weave style 8H satin (1X7) is mostly responsible for this.
- Stereo microscope examination revealed that epoxy resin coating is resistive towards fuel ingression. 0.2 mm resin coating was provided near to impermeable layer relative to 0.1 mm and without resin coating scenario, to protect the parent material.
- Resin coat application is found to help in minimizing the degradation of properties after ATF exposure.

6.2 Effect of Seawater Exposure on Mechanical Properties of E-glass/epoxy composite

6.2.1 Introduction

The essence of this thesis is to characterize the long-duration seawater effect on the mechanical parameters of fiber-reinforced composite. Structured research is conducted to determine and compare the aging effect of seawater on the mechanical properties of the E-glass/epoxy composite in bare and resin-coated conditions. It was required for better understanding and to improve the design of aerospace components, which has seawater exposure. The durability of E-glass/epoxy composite under seawater exposure was investigated in this thesis.

6.2.2 Mechanical Test results of E-glass/epoxy Composite after Seawater Exposure

6.2.2.1 Test results of Tensile Test Specimens

The tensile test specimens (warp) for all three specimen conditions were saturated under seawater enviornment where bare specimens were saturated first, followed by 0.1 mm resin coated and last 0.2 mm resin coated specimens. Once it got saturated with seawater, will be taken out, and its surface dried up with tissue paper, followed by its mechanical testing using UTM as shown in Fig. 6.26.

Fig. 6.26 Test specimen undergoing tensile test

Tensile strength was determined from the ultimate load and tensile modulus was calculated from the stress-strain curve in the linear portion. For avoiding slippage at the grips emery paper was used to increase the friction for proper gripping at the jaws.

After this, tensile properties were evaluated experimentally for different types of E-glass/epoxy specimens namely bare and specimens with 0.1 and 0.2 mm epoxy resin coating, which has undergone seawater exposure.

The results of the tensile test (warp) were as follows (Table 6.29).

Table 6.29 Tensile test specimens (warp) test results after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Tensile Strength (MPa)
(Without Resin Coating)					
1	TS-SW-Warp-WRC-01	25.54 x 1.92	5.371	15.059	307.56
2	TS-SW-Warp-WRC-02	25.81 x 2.47	5.119	17.078	274.67
3	TS-SW-Warp-WRC-03	24.83 x 2.13	4.744	15.100	283.89
1	TS-SW-Warp-WRC-04	-	-	-	Slipped
(With 0.1 mm Resin Coating)					
1	TS-SW-Warp-0.1 mm RC-01	25.30 x 1.96	6.131	14.248	289.03
2	TS-SW-Warp-0.1 mm RC-02	24.72 x 2.02	5.021	15.847	317.34
3	TS-SW-Warp-0.1 mm RC-03	25.54 x 1.92	5.347	14.991	306.18
4	TS-SW-Warp-0.1 mm RC-04	24.95 x 2.18	6.259	18.473	340.21
(With 0.2 mm Resin Coating)					
1	TS-SW-Warp-0.2 mm RC-01	24.61 x 2.08	5.594	17.922	351.93
2	TS-SW-Warp-0.2 mm RC-02	25.27 x 1.98	5.674	17.365	347.05
3	TS-SW-Warp-0.2 mm RC-03	24.70 x 1.95	6.00	16.563	337.76
4	TS-SW-Warp-0.2 mm RC-04	24.90 x 1.98	5.512	16.245	328.89

The brief of test results of tensile strength and modulus (warp) for all three conditions of test specimens are tabulated in Table 6.30.

Table 6.30 Tensile strength and modulus (warp) test results after seawater exposure

Test Specimens	Tensile Strength-without resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.1 mm resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.2 mm resin coating (MPa)	Tensile Modulus (GPa)
1	307.56	21.44	289.03	19.96	351.93	22.85
2	274.67	19.31	317.34	22.87	347.05	23.17

3	283.89	20.58	306.18	21.22	337.76	22.07
4	Slipped	Slipped	340.21	23.24	328.89	21.71
Mean	288.7	20.44	313.19	21.8	341.41	22.45
SD	16.97	1.07	21.45	1.52	10.21	0.68
% Degradation	24.09%	14.30%	17.65%	8.57%	10.23%	5.87%

The average value is to be calculated and tabulated as follows and that has to be compared with the average value of as-received specimens. The tensile properties in the warp direction before (as-received) and after seawater exposure are mentioned in Table 6.31.

Table 6.31 Effect of seawater exposure on tensile properties of E-glass/epoxy composite in the warp direction

Sample condition	Average tensile strength (warp) (MPa)		Average tensile modulus (warp) (GPa)	
	As-received	After seawater exposure	As-received	After seawater exposure
Bare samples	380.32	288.70	23.85	20.44
0.1 mm resin coated		313.19		21.80
0.2 mm resin coated		341.41		22.45

After seawater exposure, the tensile test specimens in the warp direction were tested experimentally for bare and with 0.1 mm and 0.2 mm, resin coated conditions, and correlation of data were made with as-received specimen property. It was observed that tensile strength was reduced by 24.09%, 17.65%, and 10.23% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 14.30%, 8.57%, and 5.87% degradation in terms of tensile modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively as depicted in Fig. 6.27.

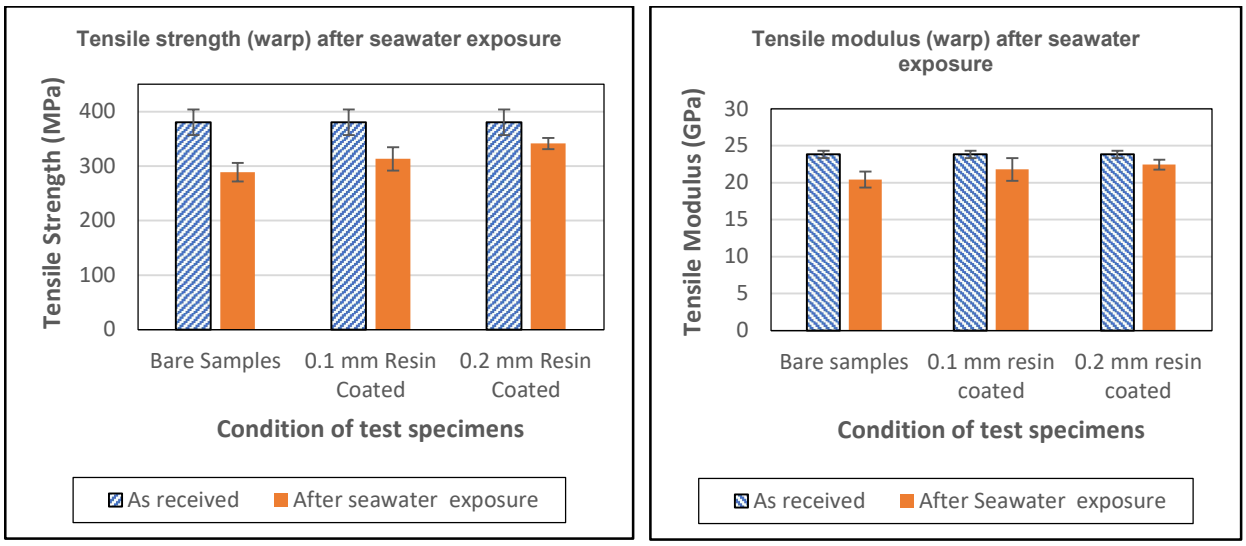


Fig. 6.27 Comparison chart of tensile properties (warp) for as-received specimens with bare and resin-coated specimens after seawater exposure

Seawater exposed tensile test samples (warp) after testing are depicted in Fig. 6.28, as follows.

Fig. 6.28 Test samples of the tensile strength (warp) after seawater exposure (after testing)

Table 6.32 Tensile test specimens (weft) results after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Tensile Strength (MPa)
(Without Resin Coating)					
1	TS-SW-Weft-WRC-01	24.91 x 2.23	5.191	16.781	301.12
2	TS-SW-Weft-WRC-02	25.12 x 2.11	6.342	14.148	267.02
3	TS-SW-Weft-WRC-03	24.99 x 2.24	5.215	15.282	273.16
4	TS-SW-Weft-WRC-04	-	-	-	slipped
(With 0.1 mm Resin Coating)					
1	TS-SW-Weft-0.1 mm RC-01	25.14 x 2.22	5.627	15.898	285.07
2	TS-SW-Weft-0.1 mm RC-02	24.75 x 1.98	7.244	15.395	314.23
3	TS-SW-Weft-0.1 mm RC-03	25.08 x 2.20	5.244	16.862	305.44
4	TS-SW-Weft-0.1 mm RC-04	25.16 x 2.09	7.159	17.158	326.03
(With 0.2 mm Resin Coating)					
1	TS-SW-Weft-0.2 mm RC-01	25.21 x 2.05	7.314	17.394	336.41
2	TS-SW-Weft-0.2 mm RC-02	25.07 x 2.12	5.145	17.305	325.78
3	TS-SW-Weft-0.2 mm RC-03	24.75 x 2.08	5.090	16.368	353.86
4	TS-SW-Weft-0.2 mm RC-04	25.10 x 2.12	5.185	17.743	318.07

The brief of test results of tensile strength and modulus (weft) for all three conditions of test specimens are tabulated in Table 6.33.

Table 6.33 Tensile strength and modulus (weft) results after seawater exposure

Test Specimens	Tensile Strength-without resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.1 mm resin coating (MPa)	Tensile Modulus (GPa)	Tensile Strength-with 0.2 mm resin coating (MPa)	Tensile Modulus (GPa)
1	301.12	19.14	285.07	20.73	336.41	20.98
2	267.02	19.94	314.23	20.02	325.78	20.83

3	273.16	18.79	305.44	18.82	353.86	22.04
4	slipped	slipped	326.03	21.24	318.07	19.19
Mean	280.43	19.29	307.69	20.2	333.53	20.76
SD	18.18	0.59	17.28	1.05	15.5	1.18
% Degradation	22.96%	12.96%	15.47%	8.85%	8.37%	6.33%

The tensile properties in the weft direction before (as-received) and after seawater exposure are mentioned in Table 6.34.

Table 6.34 Effect of seawater exposure on tensile properties of E-glass/epoxy composite in the weft direction

Sample condition	Average tensile strength (weft) (MPa)		Average tensile modulus (weft) (GPa)	
	As-received	After seawater exposure	As-received	After seawater exposure
Bare samples	364	280.43	22.16	19.29
0.1 mm resin coated		307.69		20.20
0.2 mm resin coated		333.53		20.76

However, the tensile test specimens in the weft direction were tested experimentally for bare and with 0.1 mm and 0.2 mm resin coated condition, and correlation of data was made with as-received specimen property. It was observed that tensile strength was reduced by 22.96%, 15.47%, and 8.37% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 12.96%, 8.85% and 6.33% degradation in terms of tensile modulus were observed for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively as depicted in Fig. 6.29.

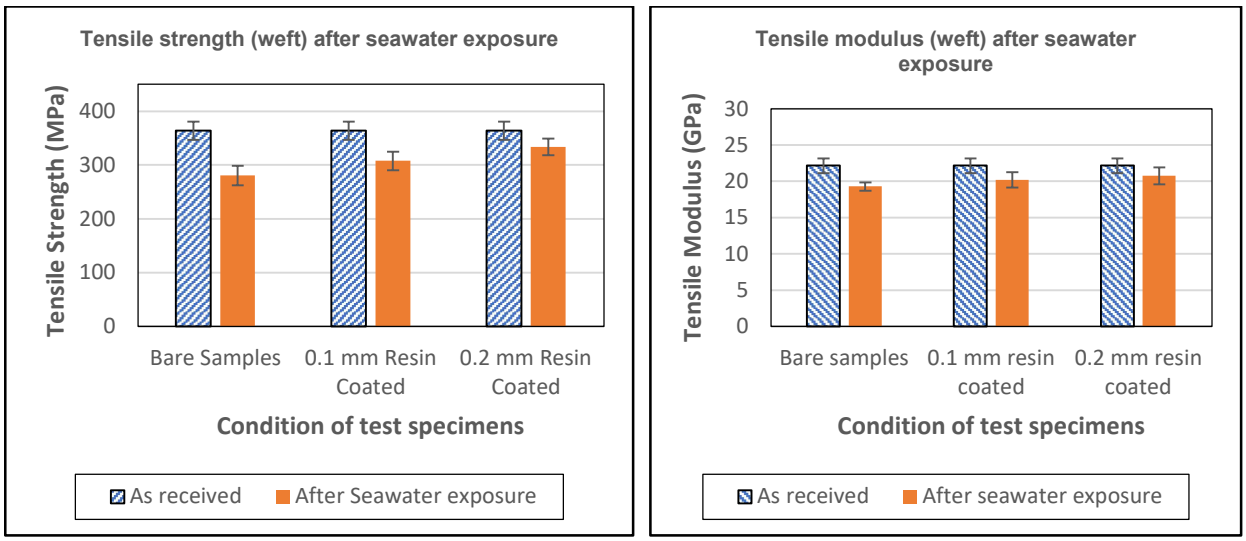


Fig. 6.29 Comparison chart of tensile properties (weft) for as-received specimens with bare and resin-coated specimens after seawater exposure

Seawater exposed tensile test samples (weft) after testing are depicted in Fig. 6.30, as follows.

Fig. 6.30 Test samples of the tensile strength (weft) after seawater exposure (after testing)

The percentage (%) of degradation of tensile properties of E-glass/epoxy composite in warp and weft direction under seawater exposure are as follows.

Table 6.35 Effect of seawater exposure on the degradation of Tensile properties of E-glass/epoxy composite in warp and weft direction

Sample condition	Degradation of tensile strength (%)		Degradation of tensile modulus (%)	
	Warp	Weft	Warp	Weft
Bare samples	24.09	22.96	14.30	12.96
0.1 mm resin coated	17.65	15.47	8.57	8.85
0.2 mm resin coated	10.23	8.37	5.87	6.33

6.2.2.2 Test results of Compressive Test Specimens

Nearly two and half months of seawater exposure has saturated compressive test specimens (warp) for all three conditions of compressive test specimens where bare specimens were saturated first, followed by 0.1 mm resin coated and last 0.2 mm resin coated specimens were saturated. Once it got saturated with seawater, will be taken out, and its surface dried up with tissue paper. After this room temperature curable E-glass/epoxy tabs will be bonded and a 10 mm gage length will be maintained. Strain gages will be bonded at the gage length area to record the corresponding strains relative to the load applied.

Fig. 6.31 Compressive test samples (warp and weft) with tabs (before testing)

The compressive specimens will undergo mechanical testing using UTM with specially designed wedge-type fixtures. The results of the compression test (warp) were as follows.

Table 6.36 Compressive test specimens (warp) results after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Compressive Strength (MPa)
(Without Resin Coating)					
1	CS-SW-Warp-WRC-01	25.02 x 2.41	2.974	16.782	278.32
2	CS-SW-Warp-WRC-02	25.11x 2.39	3.214	15.493	258.17
3	CS-SW-Warp-WRC-03	25.18 x 2.47	2.893	15.264	245.43
4	CS-SW-Warp-WRC-04	25.14 x 2.49	3.423	16.641	265.84
(With 0.1 mm Resin Coating)					
1	CS-SW-Warp-0.1 mm RC-01	25.22 x 2.51	4.123	19.786	312.57
2	CS-SW-Warp-0.1 mm RC-02	25.29 x 2.64	4.341	20.579	308.23
3	CS-SW-Warp-0.1 mm RC-03	25.31 x 2.59	3.843	18.67	284.80
4	CS-SW-Warp-0.1 mm RC-04	25.28 x 2.58	3.958	19.480	298.68
(With 0.2 mm Resin Coating)					
1	CS-SW-Warp-0.2 mm RC-01	25.43 x 2.63	3.934	22.448	335.65
2	CS-SW-Warp-0.2 mm RC-02	25.38 x 2.67	4.167	22.20	327.59
3	CS-SW-Warp-0.2 mm RC-03	25.47 x 2.71	3.798	21.155	306.49
4	CS-SW-Warp-0.2 mm RC-04	25.43 x 2.59	3.765	21.173	321.47

The brief of test results of compressive strength and modulus (warp) for all three conditions of test specimens are tabulated in Table 6.37.

Table 6.37 Compressive strength and modulus (warp) results after seawater exposure

Test Specimens	Compressive Strength- without resin coating (MPa)	Compressive Modulus (GPa)	Compressive Strength- with 0.1 mm resin coating (MPa)	Compressive Modulus (GPa)	Compressive strength- with 0.2 mm resin coating (MPa)	Compressive Modulus (GPa)
1	278.32	24.14	312.57	24.56	335.65	24.59
2	258.17	22.02	308.23	23.18	327.59	24.12

3	245.43	21.16	284.80	22.18	306.49	22.01
4	265.84	23.16	298.68	22.76	321.47	23.08
Mean	261.94	22.62	301.07	23.17	322.88	23.45
SD	13.79	1.30	12.30	1.01	12.33	1.15
% Degradatio	28.91%	7.23%	18.29%	4.95%	12.37%	3.82%

Once the compressive strength and modulus (warp) values were obtained for all three conditions of specimens the average value is to be calculated and tabulated as follows and that has to be compared with the average value of as-received specimens. The compressive properties in the warp direction before (as-received) and after seawater exposure are mentioned in Table 6.38.

Table 6.38 Effect of seawater exposure on compressive properties of E-glass/epoxy composite in the warp direction

Sample condition	Average compressive strength (warp) (MPa)		Average compressive modulus (warp) (GPa)	
	As-received	After seawater exposure	As-received	After seawater exposure
Bare samples	368.46	261.94	24.38	22.62
0.1 mm resin coated		301.07		23.17
0.2 mm resin coated		322.88		23.45

The effects of seawater exposure in terms of degradation of compressive properties were significant. The compression test specimens in the warp direction were tested experimentally for bare and with 0.1 mm and 0.2 mm, resin-coated conditions, and a correlation of data was made with the as-received specimen property. It was seen that compressive strength was reduced by 28.91%, 18.29%, and 12.37% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 7.23%, 4.95%, and 3.82% degradation in terms of compressive modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively.

The compressive properties in the warp direction before (as-received) and after seawater exposure are depicted in Fig. 6.32.

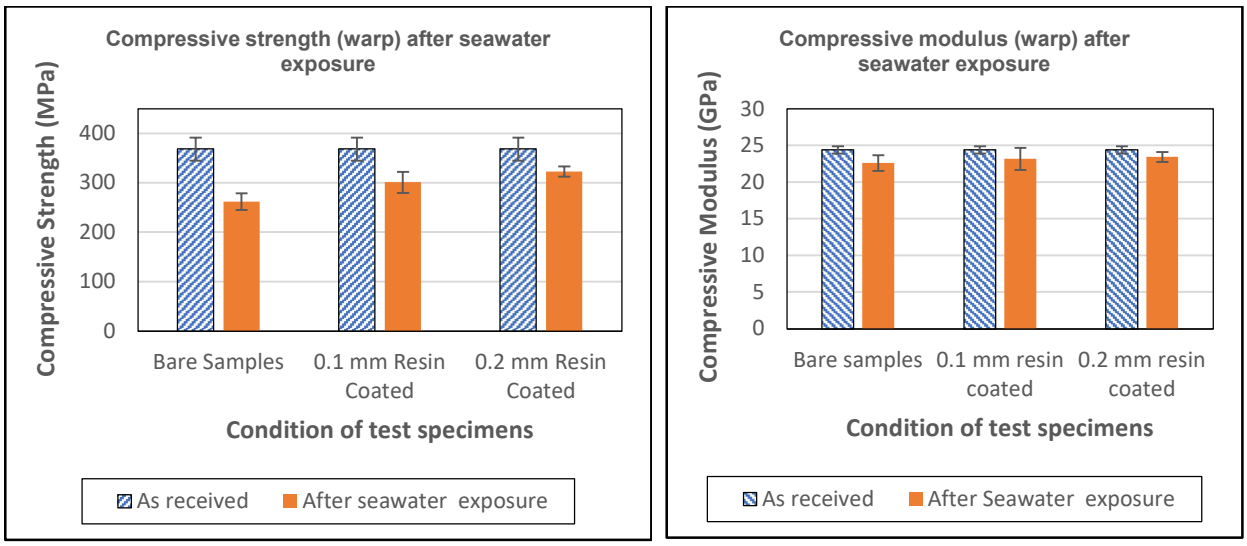


Fig. 6.32 Comparison chart of compressive properties (warp) for as-received specimens with bare and resin-coated specimens after seawater exposure

Nearly two months of seawater exposure has saturated compressive test specimens (weft) for all three conditions of compressive test specimens where bare specimens were saturated first, followed by 0.1 mm resin coated, and last 0.2 mm resin-coated specimens were saturated. Once it got saturated with seawater, will be taken out, and its surface dried up with tissue paper. After this room temperature curable E-glass/epoxy tabs will be bonded and a 10 mm gage length will be maintained. Strain gages will be bonded at the gage length area to record the corresponding strains relative to the load applied.

The compressive specimens will undergo mechanical testing using UTM with specially designed wedge-type fixtures. The results of the compression test (weft) were as follows (Table 6.39).

Table 6.39 Compressive test specimens (weft) results after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Compressive Strength (MPa)
Without Resin Coating					
1	CS-SW-Weft-WRC-01	25.08 x 2.46	3.247	16.857	273.23
2	CS-SW-Weft-WRC-02	25.01 x 2.41	3.634	14.934	247.78
3	CS-SW-Weft-WRC-03	25.12 x 2.39	3.432	15.798	263.14

4	CS-SW-Weft-WRC-04	25.07 x 2.37	3.143	15.194	255.73
With 0.1 mm Resin Coating					
1	CS-SW-Weft-0.1 mm RC-01	25.23 x 2.61	3.476	18.957	287.88
2	CS-SW-Weft-0.1 mm RC-02	25.18 x 2.49	3.789	19.147	305.39
3	CS-SW-Weft-0.1 mm RC-03	25.32 x 2.44	3.294	17.375	281.23
4	CS-SW-Weft-0.1 mm RC-04	25.34 x 2.58	3.341	17.752	271.54
With 0.2 mm Resin Coating					
1	CS-SW-Weft-0.2 mm RC-01	25.50 x 2.68	4.219	22.372	327.37
2	CS-SW-Weft-0.2 mm RC-02	25.41 x 2.57	3.964	20.565	314.92
3	CS-SW-Weft-0.2 mm RC-03	25.49 x 2.63	3.956	20.232	301.79
4	CS-SW-Weft-0.2 mm RC-04	25.52 x 2.72	4.186	21.205	305.48

The brief of test results of compressive strength and modulus (weft) for all three conditions of test specimens are tabulated in Table 6.40.

Table 6.40 Compressive strength and modulus (weft) results after seawater exposure

Test Specimens	Compr. Strength- without resin coating (MPa)	Compr. Modulus (GPa)	Compr. Strength- with 0.1 mm resin coating (MPa)	Compr. Modulus (GPa)	Compr. strength- with 0.2 mm resin coating (MPa)	Compr. Modulus (GPa)
1	273.23	23.92	287.88	23.85	327.37	24.78
2	247.78	21.95	305.39	24.30	314.92	23.86
3	263.14	22.77	281.23	23.12	301.79	21.73
4	255.73	22.02	271.54	21.65	305.48	22.87
Mean	259.97	22.67	286.51	23.23	312.39	23.31
SD	10.84	0.92	14.26	1.16	11.42	1.31
% Degradation	25.26%	5.87%	17.63%	3.54%	11.42%	3.19%

Once the compressive strength and modulus (weft) values were obtained for all three conditions of specimens the average value is to be calculated and tabulated as follows and that has to be compared with the average value of as-received specimens.

Table 6.41 Effect of ATF exposure on compressive properties of E-glass/epoxy composite in the weft direction

Sample condition	Average compressive strength (weft) (MPa)		Average compressive modulus (weft) (GPa)	
	As-received	After seawater exposure	As-received	After seawater exposure
Bare samples	347.83	259.97	24.06	22.67
0.1 mm resin coated		286.51		23.23
0.2 mm resin coated		312.39		23.31

The compressive test specimens in the weft direction were tested experimentally for bare and with 0.1 mm and 0.2 mm, resin coated conditions, and correlation of data was made with as-received specimen property. It was seen that compressive strength was reduced by 25.26%, 17.63%, and 10.19% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 5.87%, 3.54% and 3.19% degradation in terms of compressive modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively.

Similarly, Bazli et al. [76] also focused their research to know the effect of harsh environments like seawater at elevated temperature, alkaline solutions over pultruded GFRP with vinylester resin. It was observed that, in their study too exposure to these environments has also reduced the compressive properties.

The compressive properties in the weft direction before (as-received) and after seawater exposure are depicted in Fig. 6.33.

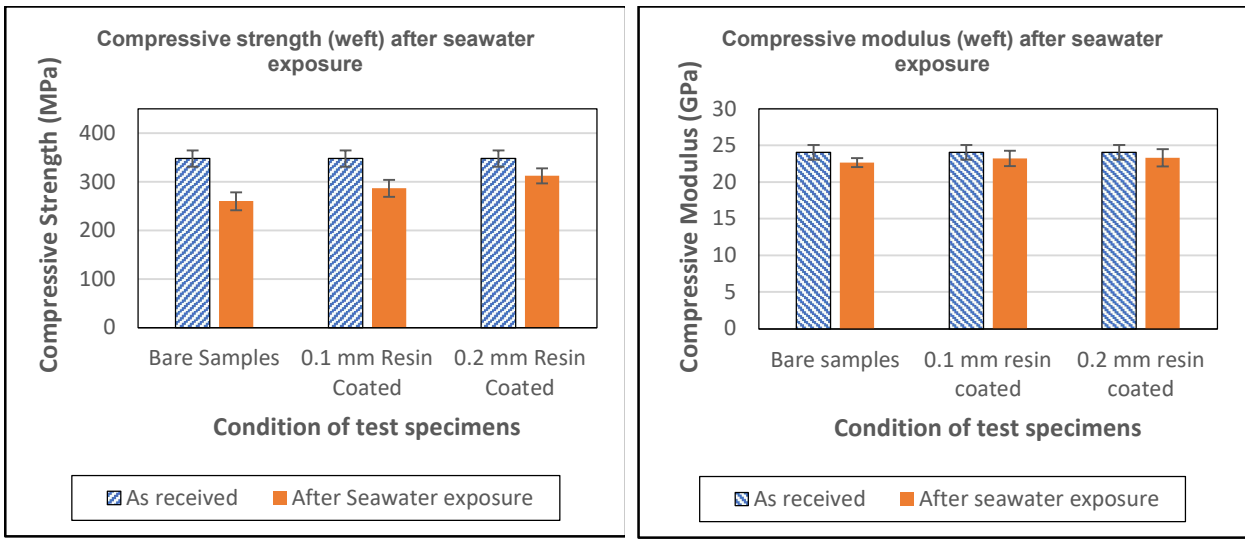


Fig. 6.33 Comparison chart of compressive properties (weft) for as-received specimens with bare and resin-coated specimens after seawater exposure

6.2.2.3 Test results of Flexural Test Specimens

Nearly two months of seawater exposure has saturated flexural test specimens (warp) for all three conditions of flexural test specimens where bare specimens were saturated first, followed by 0.1 mm resin coated, and last 0.2 mm resin-coated specimens were saturated.

Fig. 6.34 Test specimen undergoing flexural test

The flexural specimens will undergo mechanical testing using UTM with three-point bending configuration. The results of the flexural test (warp) were as follows.

Table 6.42 Flexural test specimens (warp) results after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Flexural Strength (MPa)
(Without Resin Coating)					
1	FS-SW-Warp-WRC-01	12.59 x 1.96	2.22	0.525	553.35
2	FS-SW-Warp-WRC-02	12.62 x 2.01	2.517	0.537	537.23
3	FS-SW-Warp-WRC-03	12.82 x 2.11	2.582	0.592	528.65
4	FS-SW-Warp-WRC-04	12.72 x 2.11	2.523	0.574	516.98
(With 0.1 mm Resin Coating)					
1	FS-SW-Warp-0.1 mm RC-01	12.82 x 2.12	2.659	0.681	603.11
2	FS-SW-Warp-0.1 mm RC-02	12.84 x 2.18	2.793	0.685	572.3
3	FS-SW-Warp-0.1 mm RC-03	12.76 x 2.19	2.557	0.696	580.28
4	FS-SW-Warp-0.1 mm RC-04				slipped
(With 0.2 mm Resin Coating)					
1	FS-SW-Warp-0.2 mm RC-01	12.87 x 2.29	2.655	0.797	602.12
2	FS-SW-Warp-0.2 mm RC-02	12.96 x 2.18	2.624	0.719	595.07
3	FS-SW-Warp-0.2 mm RC-03	12.88 x 2.31	2.334	0.829	615.23
4	FS-SW-Warp-0.2 mm RC-04	-	-	-	slipped

The brief of test results of flexural strength and modulus (warp) for all three conditions of test specimens are tabulated in Table 6.43.

Table 6.43 Flexural strength and modulus (warp) after seawater exposure

Test Specimens	Flexural Strength-without resin coating (MPa)	Flexural Modulus (GPa)	Flexural Strength-with 0.1 mm resin coating (MPa)	Flexural Modulus (GPa)	Flexural Strength-with 0.2 mm resin coating (MPa)	Flexural Modulus (GPa)
1	553.35	21.19	603.11	24.36	602.12	23.56
2	537.23	21.28	572.3	23.21	595.07	24.23
3	528.65	19.94	580.28	23.43	615.23	24.93
4	516.98	19.75	slipped	-	slipped	-
Mean	534.05	20.54	585.23	21.47	604.14	22.12
SD	15.31	0.81	15.99	0.61	10.23	0.71
% Degradation	20.38%	12.52%	12.75%	8.58%	9.93%	5.79%

Once the flexural strength and modulus (warp) values were obtained for all three conditions of specimens the average value is to be calculated and tabulated as follows and that has to be compared with the average value of as-received specimens. The flexural properties in the warp direction before (as-received) and after seawater exposure are mentioned in Table 6.44.

Table 6.44 Effect of seawater exposure on flexural properties of E-glass/epoxy composite in the warp direction

Sample condition	Average flexural strength (warp) (MPa)		Average flexural modulus (warp) (GPa)	
	As-received	After seawater exposure	As-received	After seawater exposure
Bare samples	670.75	534.05	23.48	20.54
0.1 mm resin coated		585.23		21.47
0.2 mm resin coated		604.14		22.12

The effects of seawater exposure in terms of degradation of flexural properties were significant. The flexural test specimens in the warp direction were tested experimentally for bare and with 0.1 mm and 0.2 mm, resin-coated conditions, and a correlation of data was made with the as-received specimen property. It was seen that flexural strength was reduced by 20.38%, 12.75%, and 9.93% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 12.52%, 8.58%, and 5.79% degradation in terms of flexural modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively as depicted in Fig. 6.35.

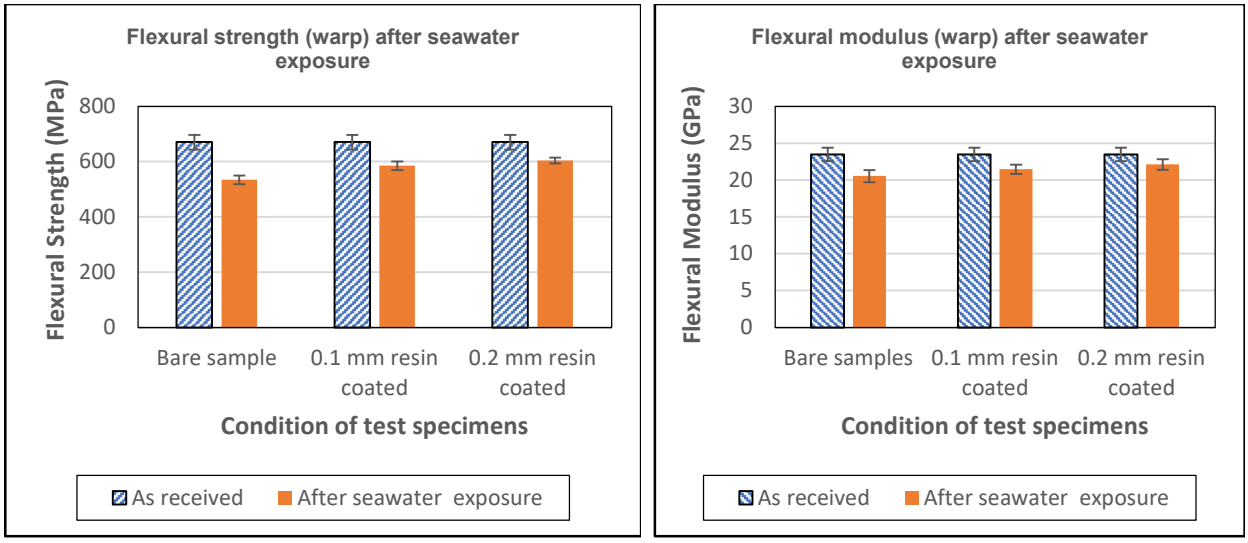


Fig. 6.35 Comparison chart of flexural properties (warp) for as-received specimens with bare and resin-coated specimens after seawater exposure

Table 6.45 Flexural test specimens (weft) results after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	Flexural Strength (MPa)
(Without Resin Coating)					
1	FS-SW-Weft-WRC-01	12.58 x 2.11	2.558	0.574	523.07
2	FS-SW-Weft-WRC-02	12.62 x 2.01	2.503	0.534	534.41
3	FS-SW-Weft-WRC-03	12.69 x 2.11	1.936	0.533	481.59
4	FS-SW-Weft-WRC-04	12.67 x 2.15	2.514	0.583	507.48

(With 0.1 mm Resin Coating)					
1	FS-SW-Weft-0.1 mm RC -01	12.92 x 2.11	2.572	0.593	526.12
2	FS-SW-Weft-0.1 mm RC -02	12.89 x 2.16	2.232	0.657	556.83
3	FS-SW-Weft-0.1 mm RC -03	12.76 x 2.19	2.588	0.705	587.16
4	FS-SW-Weft-0.1 mm RC -04	12.82 x 2.11	2.526	0.603	539.02
(With 0.2 mm Resin Coating)					
1	FS-SW-Weft-0.2 mm RC -01	12.96 x 2.27	2.557	0.760	580.32
2	FS-SW-Weft-0.2 mm RC -02	12.89 x 2.23	2.22	0.696	553.76
3	FS-SW-Weft-0.2 mm RC -03	12.94 x 2.28	2.773	0.750	568.79
4	FS-SW-Weft-0.2 mm RC -04	12.92 x 2.21	2.494	0.659	532.27

The brief of test results of flexural strength and modulus (weft) for all three conditions of test specimens are tabulated in Table 6.46.

Table 6.46 Flexural strength and modulus (weft) after seawater exposure

Test Specimens	Flexural Strength-without resin coating (MPa)	Flexural Modulus (GPa)	Flexural Strength-with 0.1 mm resin coating (MPa)	Flexural Modulus (GPa)	Flexural Strength-with 0.2 mm resin coating (MPa)	Flexural Modulus (GPa)
1	523.07	20.35	526.12	20.38	580.32	21.89
2	534.41	21.18	556.83	21.34	553.76	21.36
3	481.59	19.6	587.16	21.81	568.79	21.31
4	507.48	19.47	539.02	19.95	532.27	21.12
Mean	511.64	20.15	552.28	20.87	558.79	21.42
SD	22.87	0.79	26.44	0.87	20.75	0.33
% Degradation	17.41%	10.86%	10.85%	7.71%	9.80%	5.28%

The average flexural properties in the weft direction before (as-received) and after seawater exposure are mentioned in Table 6.47.

Table 6.47 Effect of seawater exposure on flexural properties of E-glass/epoxy composite in the weft direction

Sample condition	Average flexural strength (weft) (MPa)		Average flexural modulus (weft) (GPa)	
	As-received	After seawater exposure	As-received	After seawater exposure
Bare samples	619.50	511.64	22.61	20.15
0.1 mm resin coated		552.28		20.87
0.2 mm resin coated		558.79		21.42

The flexural test specimens in the weft direction were tested experimentally for bare and with 0.1 mm and 0.2 mm, resin coated conditions, and correlation of data was made with as-received specimen property. It was seen that flexural strength was reduced by 17.41%, 10.85%, and 9.80% for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and 10.86%, 7.71%, and 5.28% degradation in terms of flexural modulus for bare, with 0.1 mm and 0.2 mm resin coating conditions, respectively as depicted in Fig. 6.36.

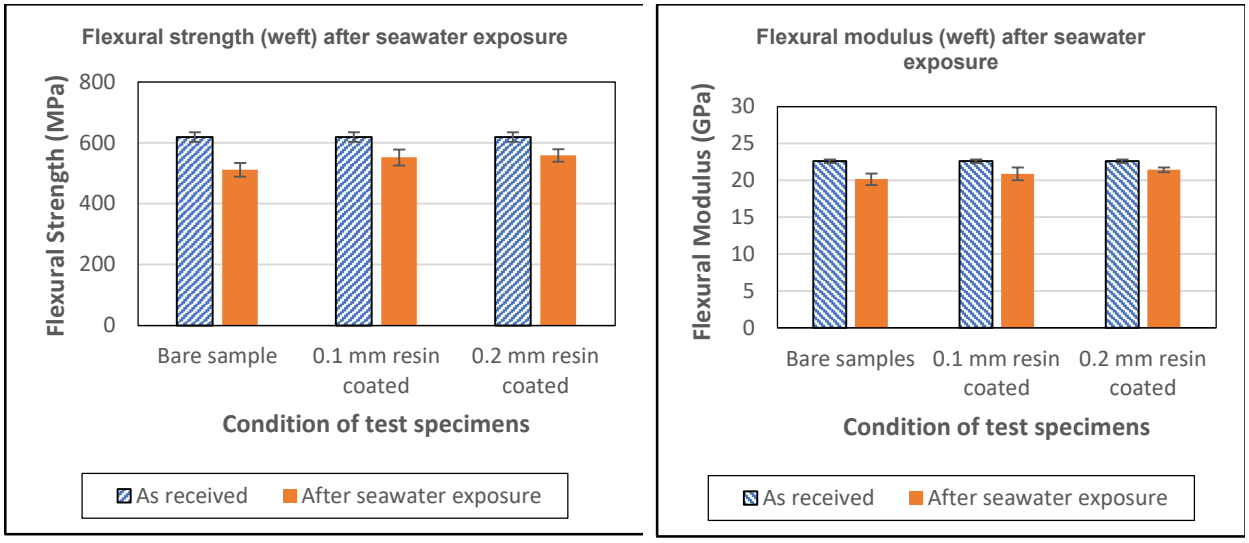


Fig. 6.36 Comparison chart of flexural properties (weft) for as-received specimens with bare and resin-coated specimens after seawater exposure

Similarly, Afshar et al. [77] too studied the effect of long-time exposure of marine environments to flexural properties of carbon/vinylester composites and reported notable degradation of flexural

strength as well as modulus, like to this research work of E-glass/epoxy composite. Seawater exposed flexural test samples (warp and weft) after testing are depicted in Fig. 6.37, as follows.

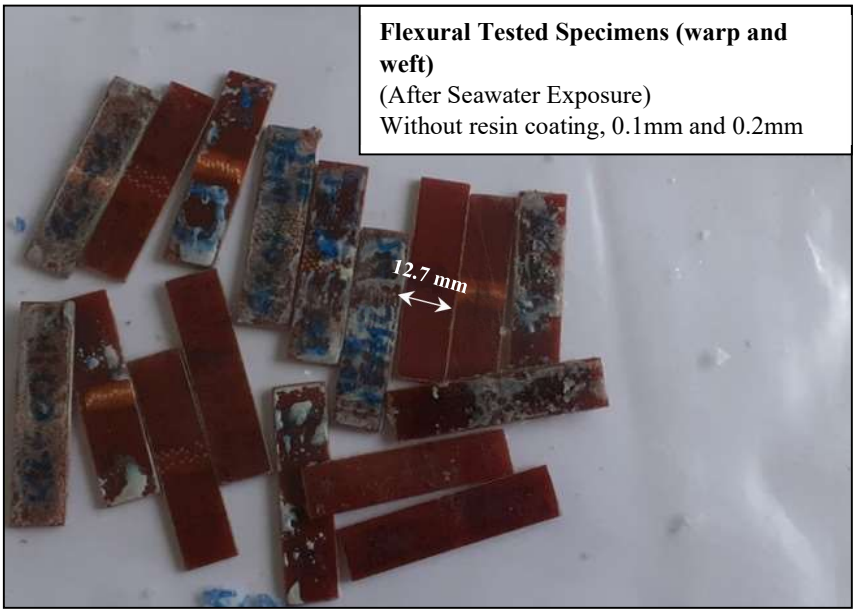


Fig. 6.37 Test samples of flexural warp and weft after seawater exposure (after testing)

6.2.2.4 Test results of ILSS Test Specimens

Nearly two and half months of seawater exposure has saturated ILSS test specimens for all three conditions of ILSS test specimens where bare specimens were saturated first, followed by 0.1 mm resin coated, and last 0.2 mm resin-coated specimens were saturated.

The ILSS specimens will undergo mechanical testing using UTM with three-point bending. The results of the ILSS test were as follows.

Fig. 6.38 Test specimen undergoing ILSS test

Table 6.48 ILSS test specimens result after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	ILSS Strength (MPa)
(Without Resin Coating)					
1	ILSS-SW-WRC-01	12.05 x 4.08	0.996	3.839	58.56
2	ILSS-SW-WRC-02	11.91 x 4.11	0.59	3.778	57.89
3	ILSS-SW-WRC-03	11.88 x 4.05	0.59	3.83	59.71
4	ILSS-SW-WRC-04	12.54 x 4.14	1.01	3.670	53.01
(With 0.1 mm Resin Coating)					
1	ILSS-SW-0.1 mm RC-01	12.14 x 4.19	0.705	4.211	62.09
2	ILSS-SW-0.1 mm RC-02	12.23 x 4.17	0.728	4.210	61.92
3	ILSS-SW-0.1 mm RC-03	12.18 x 4.13	0.661	4.093	61.03
4	ILSS-SW-0.1 mm RC-04	12.26 x 4.05	0.60	3.911	59.08
(With 0.2 mm Resin Coating)					
1	ILSS-SW-0.2 mm RC-01	12.41 x 4.38	0.743	4.572	63.08
2	ILSS-SW-0.2 mm RC-02	12.32 x 4.31	0.754	4.363	61.62
3	ILSS-SW-0.2 mm RC-03	12.38 x 4.29	0.736	4.397	62.09
4	ILSS-SW-0.2 mm RC-04	12.33 x 4.36	0.883	4.729	65.97

The brief of test results of ILSS strength for all three conditions of test specimens are tabulated in Table 6.49.

Table 6.49 ILSS strength after seawater exposure

Test Specimens	ILSS Strength- without resin coating (MPa)	ILSS Strength- with 0.1 mm resin coating (MPa)	ILSS Strength-with 0.2 mm resin coating (MPa)
1	58.56	62.09	63.08
2	57.89	61.92	61.62
3	59.71	61.03	62.09

4	53.01	59.08	65.97
Mean	57.29	61.03	63.19
SD	1.38	1.95	1.95
% Degradation	16.01 %	10.53%	7.36%

The average ILSS properties before (as-received) and after seawater exposure are mentioned in Table 6.50.

Table 6.50 Effect of seawater exposure on inter-laminar shear properties of E-glass/epoxy composite

Sample condition	Average Interlaminar shear strength (MPa)	
	As-received	After seawater exposure
Bare samples	68.21	57.29
0.1 mm resin coated		61.03
0.2 mm resin coated		63.19

interlaminar shear strength was evaluated experimentally for different types of E-glass/epoxy specimens namely bare and specimens with 0.1 and 0.2 mm thick epoxy resin coating, which have undergone seawater exposure. It was observed that the mean interlaminar strength of bare test specimens were 57.29 MPa, while for specimens with 0.1mm thick resin coating, the mean interlaminar shear strength was 61.03 MPa. Similarly, in specimens with 0.2 mm resin coating, the mean interlaminar shear strength was 63.19 MPa. Correlation of these data was made with as-received property and it shows that 16.01%, 10.53%, and 7.36% degradation in terms of interlaminar shear strength (ILSS) were observed for bare, with 0.1 mm and 0.2 mm thick resin coating conditions as shown (Fig. 6.39)

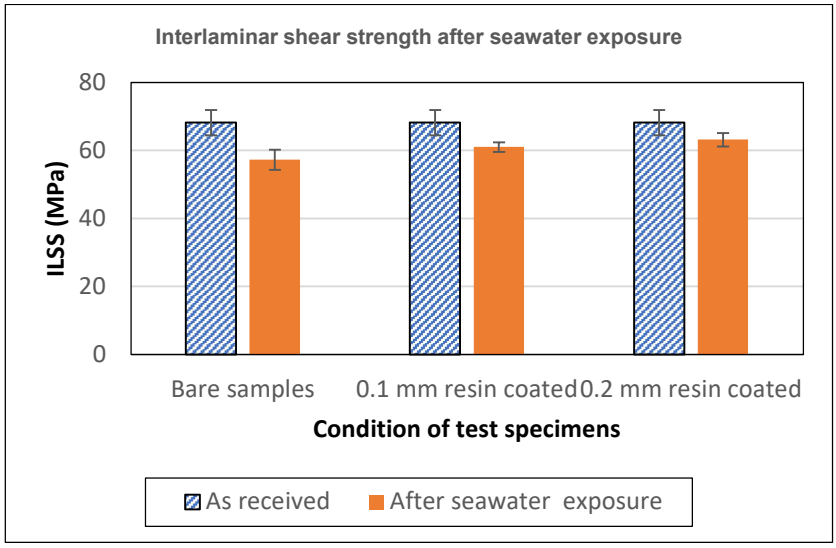


Fig. 6.39 Comparison chart of interlaminar shear properties for as-received specimens with bare and resin coated specimens after seawater exposure

Seawater exposed ILSS test samples after testing are depicted in Fig. 6.40, as follows.

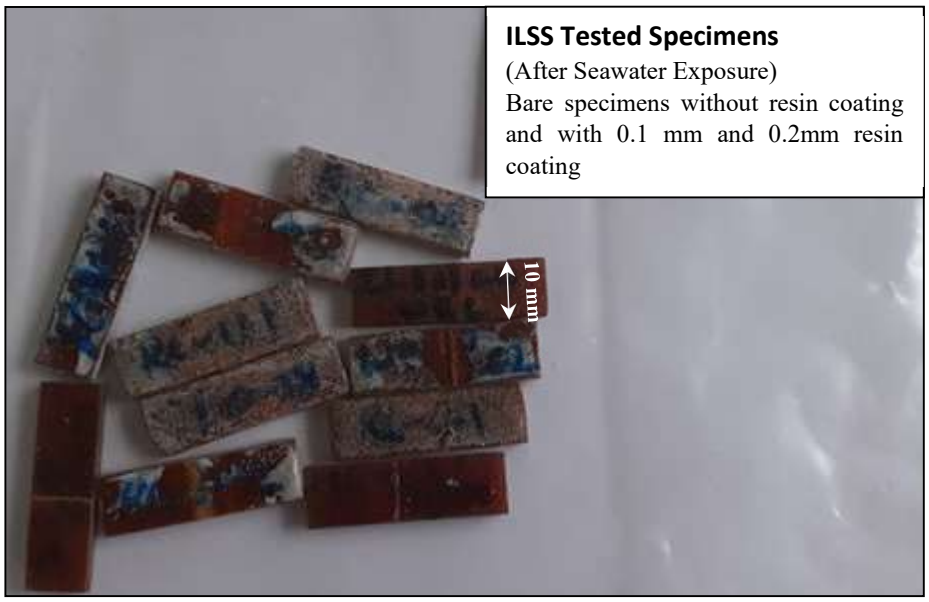


Fig. 6.40 ILSS test specimens after seawater exposure (after testing)

6.2.2.5 Test results of IPSS Test Specimens

Nearly two and half months of seawater exposure has saturated IPSS test specimens for all three conditions of IPSS test specimens where bare specimens were saturated first, followed by 0.1 mm resin coated, and last 0.2 mm resin-coated specimens were saturated. The results of the ILSS test were as follows.

Fig. 6.41 Test specimen undergoing IPSS test

Extensometer is used on test specimens to record the strains in loading as well as perpendicular to the loading direction. The in-plane shear modulus is determined from the curve of stress-strain.

Table 6.51 IPSS test specimens result after seawater exposure

Sl No.	Test specimen identification	Test specimens dimensions (mm)	Max. Displacement (mm)	Max. load (kN)	IPSS Strength (MPa)
(Without Resin Coating)					
1	IPSS-SW-WRC-01	24.91 x 1.82	21.728	6.710	74
2	IPSS-SW-WRC-02	24.99 x 1.78	13.935	5.694	64
3	IPSS-SW-WRC-03	25.05 x 1.81	24.987	7.526	83
4	IPSS-SW-WRC-04	25.01 x 1.80	18.745	6.483	72
(With 0.1 mm Resin Coating)					
1	IPSS-SW-0.1 mm RC-01	25.21 x1.99	30.275	8.528	85
2	IPSS-SW-0.1 mm RC-02	25.14 x 2.01	16.057	7.681	76
3	IPSS-SW-0.1 mm RC-03	25.17 x 1.94	26.441	8,203	84
4	IPSS-SW-0.1 mm RC-04	25.16 x 1.97	-	-	Slipped

(With 0.2 mm Resin Coating)					
1	IPSS-SW-0.2 mm RC-01	25.32 x 2.16	-	-	Slipped
2	IPSS-SW-0.2 mm RC-02	25.36 x 2.19	26.70	9.997	90
3	IPSS-SW-0.2 mm RC-03	25.28 x 2.14	28.54	10.062	93
4	IPSS-SW-0.2 mm RC-04	25.35 x 2.20	26.101	9.592	86

The brief of test results of IPSS strength and modulus for all three conditions of test specimens are tabulated in Table 6.52.

Table 6.52 In-plane shear strength and modulus after seawater exposure

Test Specimens	IPSS Strength-without resin coating (MPa)	In-plane Shear Modulus (GPa)	IPSS Strength-with 0.1 mm resin coating (MPa)	In-plane Shear Modulus (GPa)	IPSS Strength-with 0.2 mm resin coating (MPa)	In-plane Shear Modulus (GPa)
1	74	3.37	85	3.87	88	3.72
2	64	3.17	76	3.49	90	3.84
3	83	3.53	84	3.67	93	4.07
4	72	3.38	slipped	slipped	86	3.54
Mean	73.25	3.36	81.67	3.68	89.25	3.79
SD	7.8	0.15	4.93	0.19	2.99	0.22
% Degradation	26.10%	18.84%	17.60%	11.11%	9.96%	8.45%

The average IPSS properties before (as-received) and after seawater exposure are mentioned in Table 6.53.

Table 6.53 Effect of seawater exposure on in-plane shear properties of E-glass/epoxy composite

Sample condition	Average In-plane shear strength (MPa)		Average In-plane shear modulus GPa)	
	As-received	After seawater exposure	As-received	After seawater exposure
Bare samples	99.12	73.25	4.14	3.36
0.1 mm resin coated		81,67		3.68
0.2 mm resin coated		89.25		3.79

Correlation w.r.t specified property shows that 26.10%, 17.60%, and 9.96 % degradation in terms of in-plane shear strength (IPSS) were observed for bare, 0.1 mm and 0.2 mm resin coating and 18.84%, 11.11% and 8.45% in terms of in-plane shear modulus were observed for bare, 0.1 mm and 0.2 mm resin coating conditions as depicted (Fig. 6.42).

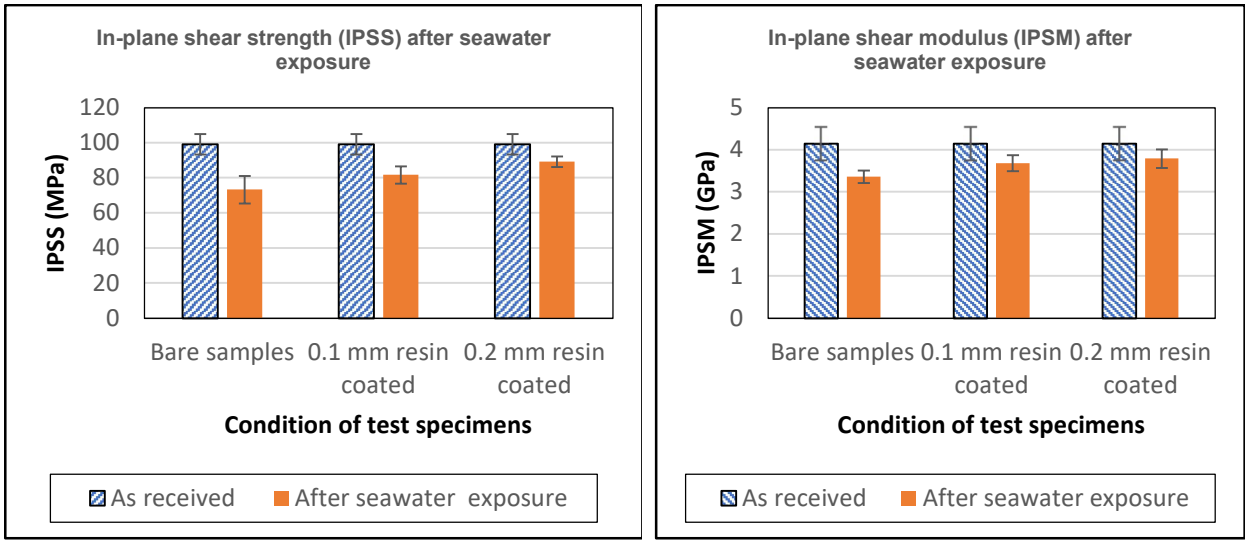


Fig. 6.42 Comparison chart of in-plane shear properties for as-received specimens with bare and resin-coated specimens after seawater exposure

Seawater exposed IPSS test samples after testing are depicted in Fig. 6.43, as follows.

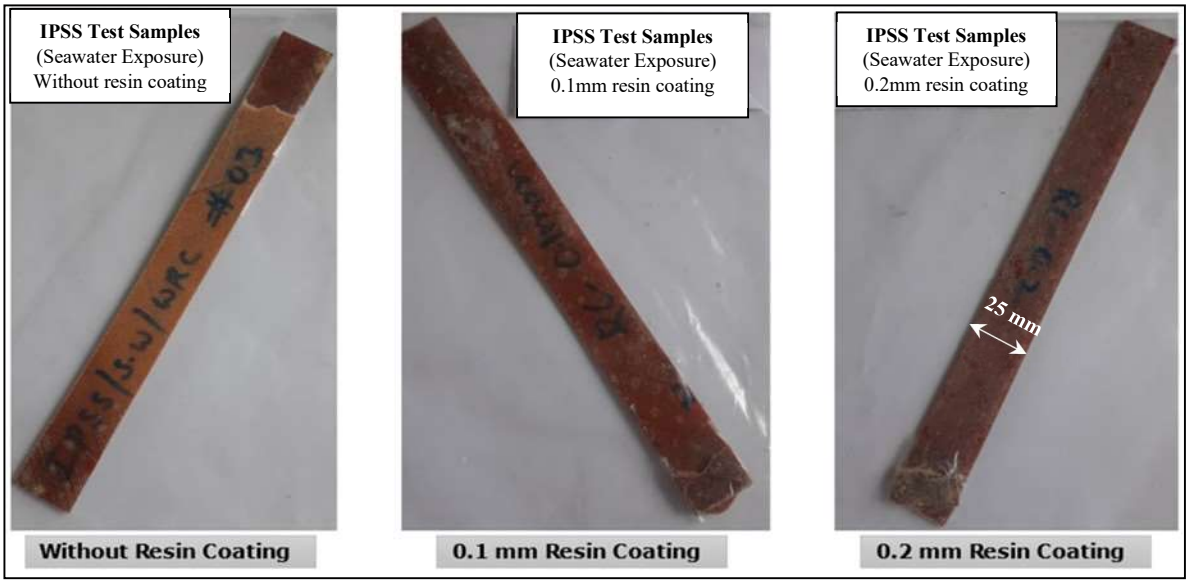


Fig. 6.43 IPSS test specimens after seawater exposure (after testing)

6.2.3 Microstructural Examination of Seawater Exposed Samples

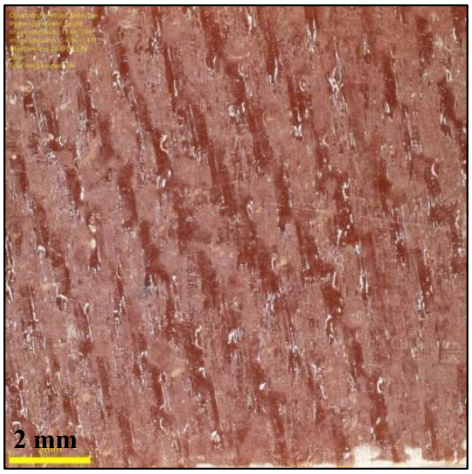
To know the degradation mechanism, the tested samples of E-glass/epoxy composite after seawater exposure, were surface examined using stereo microscope and sub-surface examination (fractured surface) was carried out using SEM as follows. This data was helpful to draw the conclusions for failure characteristics and associated degradation mechanism of tested samples.

6.2.3.1 Stereo microscope examination of test samples exposed to Seawater

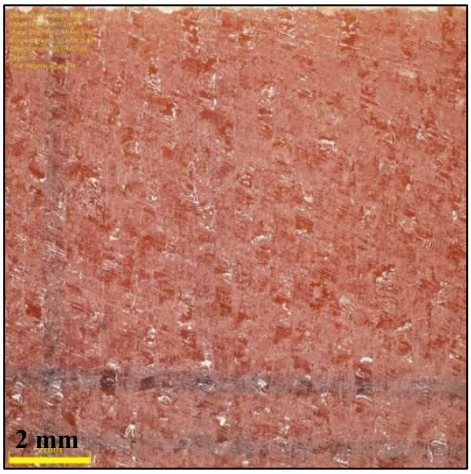
The test samples of E-glass/epoxy composite were examined under a stereo microscope (Olympus DSX110) as depicted in Fig. 6.41 and Fig. 6.42. The test specimens that underwent investigation were seawater-exposed ones. It was observed that the test specimen coated with 0.2 mm resin coating restricted the seawater ingression to the resin coating surface and limited the seawater ingression sub-surface. Hence epoxy coating is providing, one kind of sacrificial and protective layer which was degrading with seawater ingression as depicted below in the figures. Resin coating with 0.1 mm were showing small seawater ingression patches on the surface, because of the lesser thickness of the coating. Table 6.54 as below was provided to give an explicit idea about the conditions of the test specimens under examination.

Table 6.54 Bare and resin coated test specimens of different type of tests, exposed to seawater

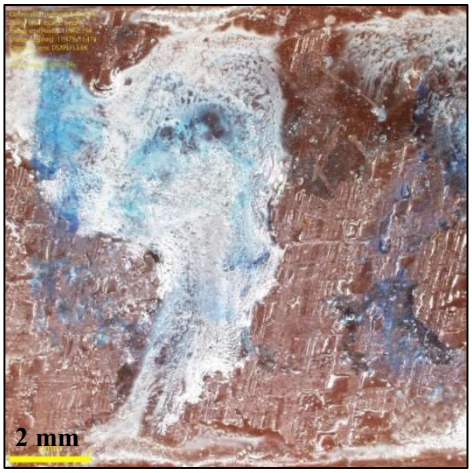
Sl No.	Name of test specimens	Condition of specimens	Remarks if any
a	ILSS	WRC (Without resin coating)	Bare specimen
b	ILSS	0.1mm RC (0.1 mm resin coating)	Resin coated
c	ILSS	0.2mm RC (0.2 mm resin coating)	Resin coated
d	Flexural (warp)	WRC (Without resin coating)	Bare specimen
e	Flexural (warp)	0.1mm RC (0.1 mm resin coating)	Resin coated
f	Flexural (warp)	0.2mm RC (0.2 mm resin coating)	Resin coated
g	ILSS (Top)	0.1mm RC (0.1 mm resin coating)	Resin-coated top surface
h	ILSS (Edge)	0.1mm RC (0.1 mm resin coating)	Resin coated Edge surface
i	ILSS (Tested)	0.1mm RC (0.1 mm resin coating)	Failure zone after ILSS testing
j	Compression (warp)	WRC (Without resin coating)	Bare specimen
k	Compression (warp)	0.1mm RC (0.1 mm resin coating)	Resin coated
l	Compression (warp)	0.2mm RC (0.2 mm resin coating)	Resin coated



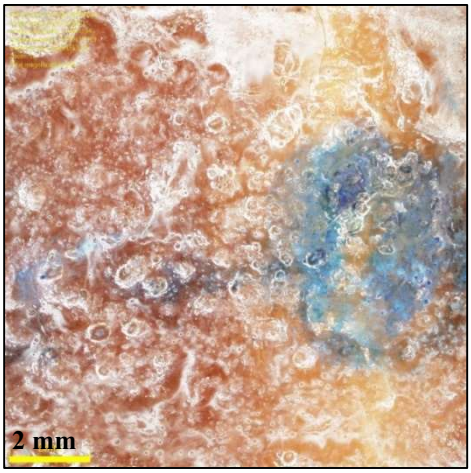
a) ILSS- WRC



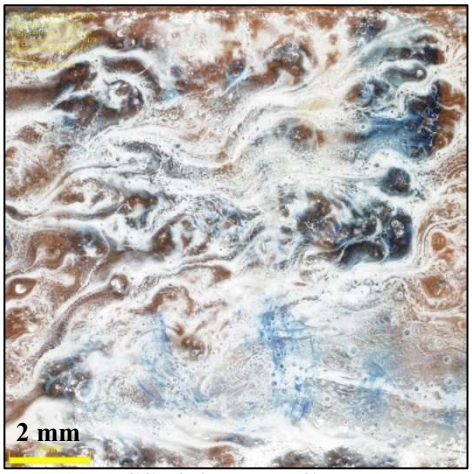
d) Flexural- WARP- WRC



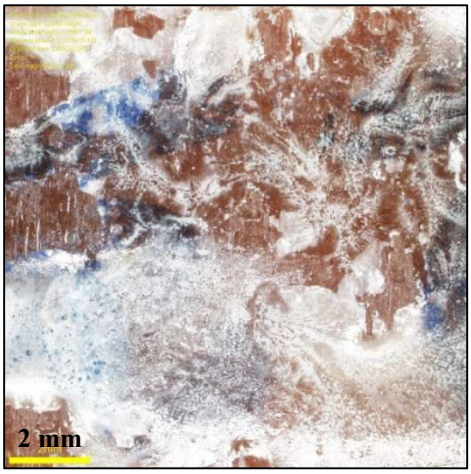
b) ILSS- 0.1 mm RC



e) Flexural-WARP- 0.1 mm RC

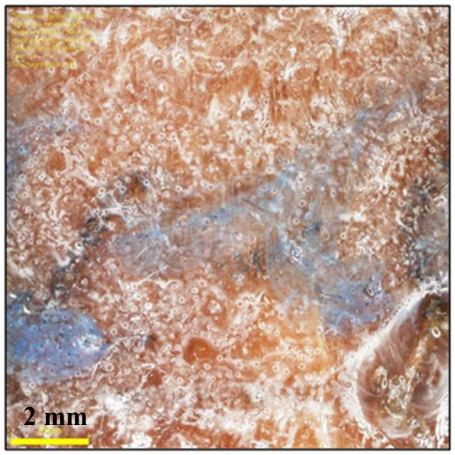


c) ILSS- 0.2 mm RC

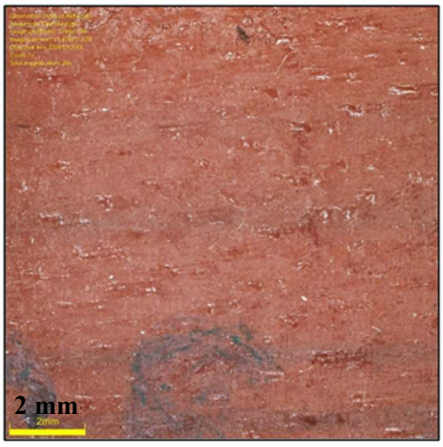


f) Flexural-WARP- 0.2 mm RC

Fig. 6.44 Stereo microscope examinations of ILSS and Flexural test samples exposed to seawater

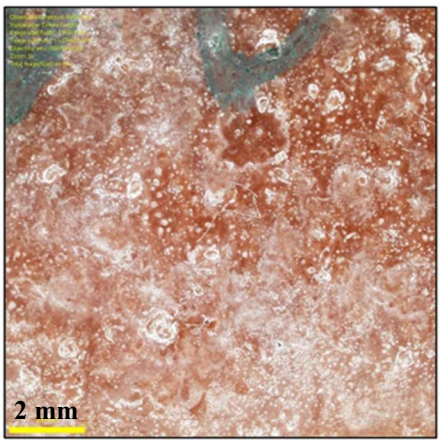


g) ILSS – 0.1 mm RC

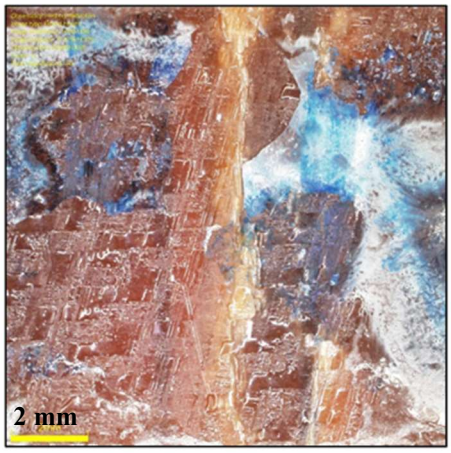


j) Compression-WARP-WRC

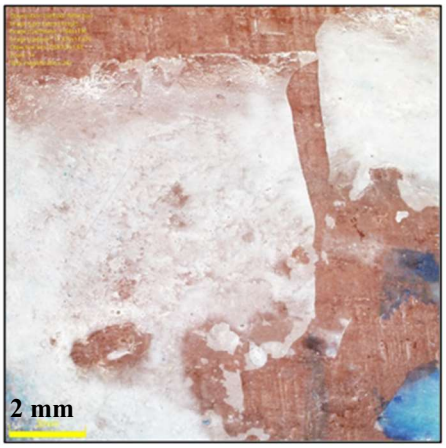
h) ILSS-Edge- 0.1 mm RC



k) Compression-WARP-0.1 mm RC



i) ILSS-Tested Samples-0.1 mm RC



l) Compression-WARP-0.2 mm RC

Fig. 6.45 Stereo microscope examinations of ILSS and Compression test samples exposed to seawater

6.2.3.2 Microstructural Examination of Tested Seawater Exposed Fractured Surface using Scanning Electron Microscope (SEM)

The microstructural examination was carried out for bare specimens without seawater exposure, seawater-exposed bare specimens without resin coating, and resin-coated specimens. The test specimens like tensile, compressive, and IPSS were chosen for this investigation using SEM, as they all were having generating clean fractured surfaces to examine the fiber and its interfacial characteristics. Grant et al. [79] described the degradation mechanism of graphite epoxy composite using SEM under seawater environment. The similar behavior of E-glass/epoxy composite were analyzed using SEM under seawater exposure.

A) Microstructural Comparative Examination of the Fractured Surface of with and without Seawater Exposed Tensile Test Specimens using SEM

The fractured surface of the tensile test specimen was analyzed using a scanning electron microscope (SEM). It was found that the specimen without seawater exposure (as received) was intact and exhibited a good bond between the matrix fiber interface as depicted in Fig. 6.46 (a). Although fractured surfaces of bare specimens (without resin coating), which undergo seawater exposure revealed that the diffusion of seawater into composites led to its degradation because of matrix micro-cracking, resin crumbling and debonding on the matrix fiber interface as shown in Fig. 6.46 (b). In addition to this, the diffusion of seawater drives the way for hydrolysis reaction in the composite, including the hydrolysis of the matrix and fiber-matrix interface. These phenomena are responsible for the reduction in tensile and flexural strength and its modulus too. The test specimens which were coated with 0.1 and 0.2 mm resin coating also underwent SEM examinations and found that seawater exposure only degraded the resin coating on the surface and generated the micro cracks, but it restricted the seawater ingression inside the test specimens, which notably reduced the degradation in properties cause of seawater diffusion as depicted in Fig. 6.46 (c) and Fig. 6.46 (d). As the epoxy resin is having hydrophilic characteristics, exhibiting a strong affinity towards water, hence it will undergo seawater absorption and this causes changes in the chemical and mechanical characteristics of the matrix by the processes of both hydrolysis and plasticization. This will be responsible for the degradation of its mechanical properties for bare specimens and resin-coated ones, degradation will be limited to the surface because coating and sub-surface

characteristics will not be affected much as shown in SEM micrographs, resulting in lower degradation of the properties.

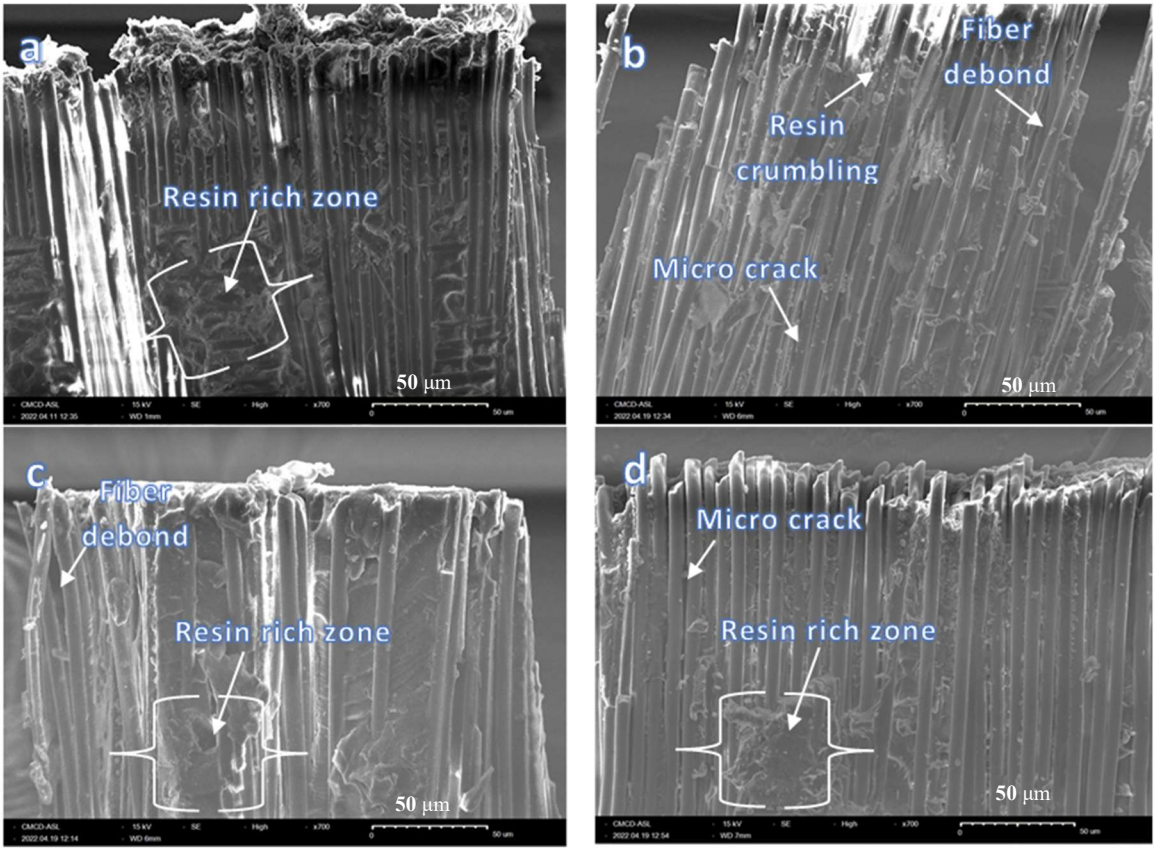


Fig.6.46 SEM micrographs of tensile fractured composite (a) bare specimen without seawater exposure, (b) bare specimen exposed to seawater, (c) 0.1 mm resin coated specimen exposed to seawater, and (d) 0.2 mm resin coated specimen exposed to seawater

B) Microstructural Comparative Examination of the Fractured Surface of with and without Seawater Exposed Tensile Test Specimens using SEM

The effect of seawater ingression in terms of degradation of compressive properties was significant. To compare and assess the damage mechanisms that originated in the fiber-matrix interface for different types of test specimens, before and after the seawater exposure, fractured fibers of the tested compressive (warp) specimens were investigated by using SEM. Fractures fibers of compressive test specimens were examined before and after seawater exposure and it was noticed here also that bare tested specimens exhibited maximum degradation compared to resin-coated ones. SEM micrographs revealed that for tested specimens (bare condition), the diffusion of seawater into composites led to degradation because of plasticization of the matrix, matrix

cracking, delamination, fiber debonding, and resin crumbling as shown below in the figures, whereas the resin-coated tested specimens were limiting the ATF ingression, which gave relatively lower reduction as depicted in Fig.6.47 (a), (b), (c) and (d).

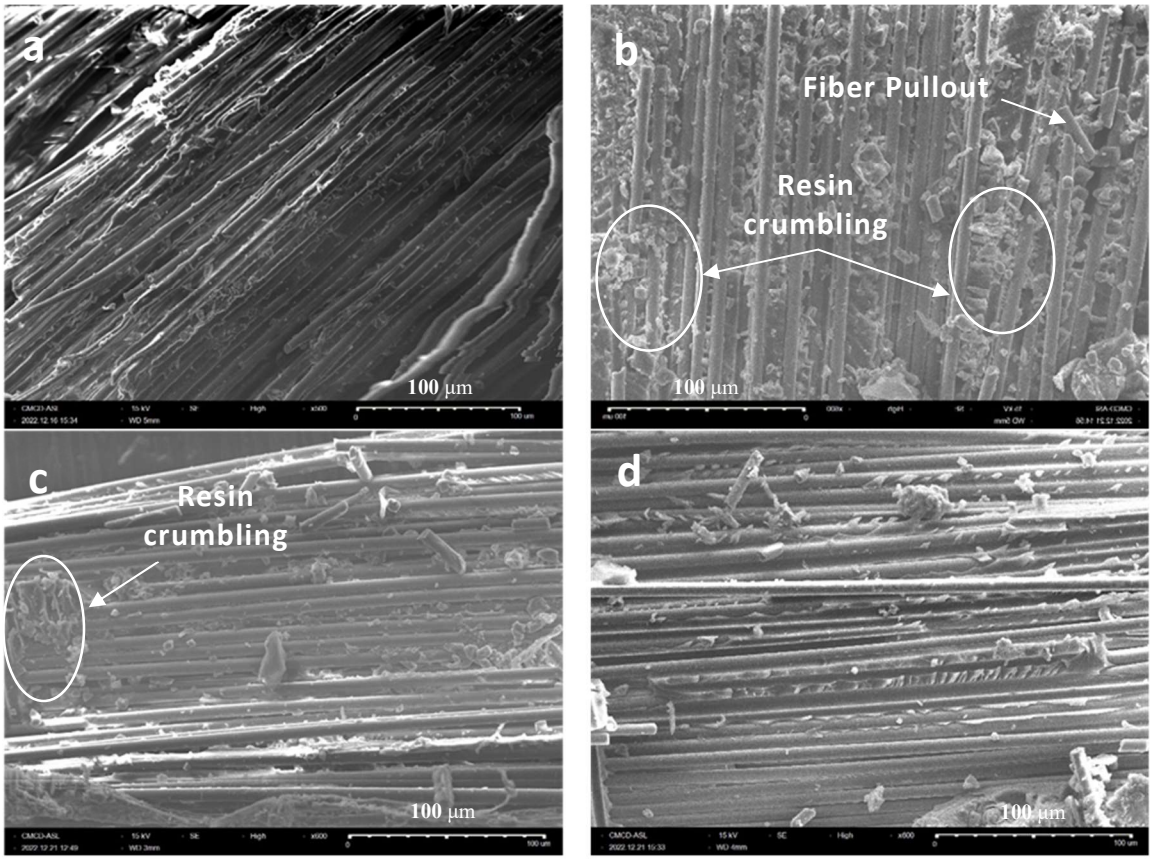


Fig. 6.47 SEM micrographs of Compressive fractured composite (a) bare specimen without seawater exposure, (b) bare specimen exposed to seawater, (c) 0.1 mm resin coated specimen exposed to seawater, and (d) 0.2 mm resin coated specimen exposed to seawater

C) Microstructural Comparative Examination of the Fractured Surface with and without Seawater Exposed In-plane Shear Test Specimens using SEM

A significant reduction of in-plane shear strength (IPSS) was observed in bare specimens compared to as-received specimens. This reduction of properties is happening because of the formation of micro voids and micro-cracks, generating out of induced internal stress because of seawater penetration to the matrix network. In addition to this penetration of seawater is weakening the interfacial properties of matrix and reinforcement, which is further leading to properties degradation.

The reduction of in-plane shear strength (IPSS) was observed for resin-coated specimens too, as initially, resin-coating barriers were not allowing seawater ingression. But after a significant time of seawater exposure, cracks are getting generated in the matrix and it will allow the seawater to penetrate through matrix cracking, which will degrade the interfacial characteristics. The degradation of reinforcement and matrix interface will further lead to a slight reduction of respective strength.

Fractured fibers of tested in-plane shear specimens were examined using SEM micrographs as shown in Fig. 6.48 (a), (b), (c), and (d), which were taken before and after the seawater exposure.

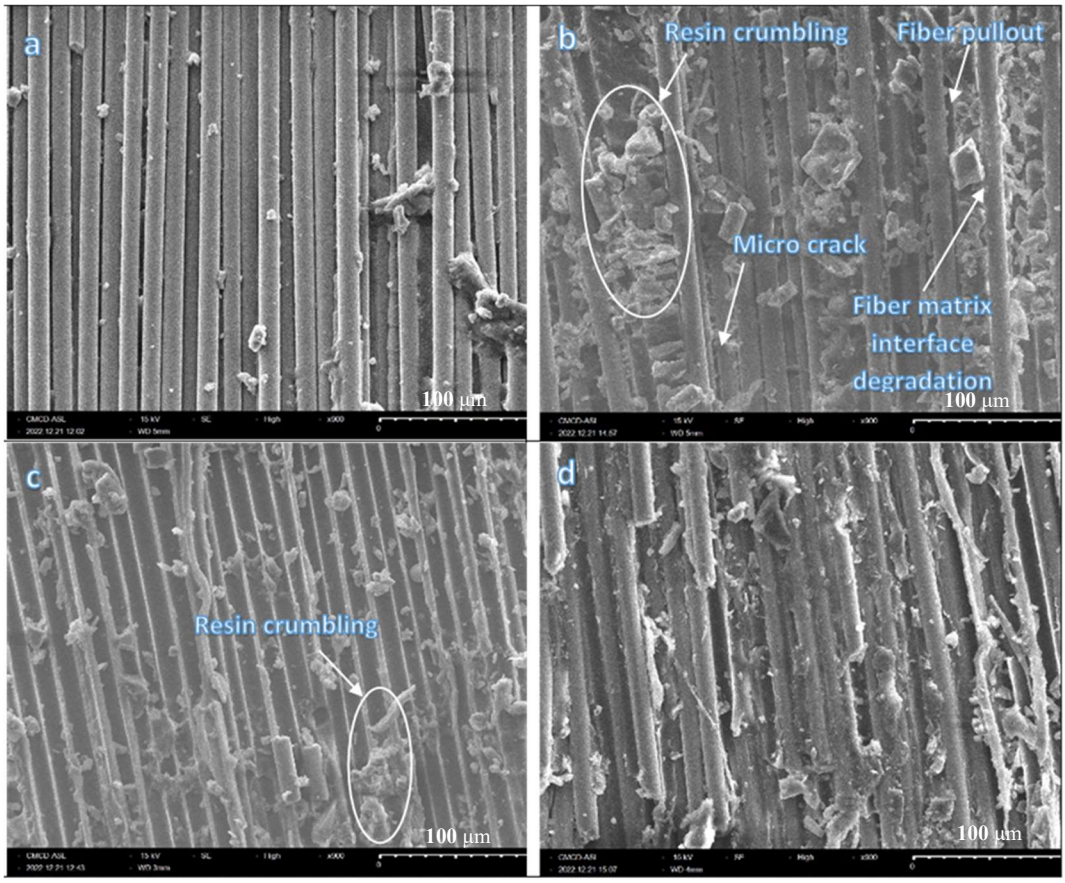


Fig. 6.48 SEM micrographs of IPSS fractured composite (a) bare specimen without seawater exposure, (b) bare specimen exposed to seawater, (c) 0.1 mm resin coated specimen exposed to seawater, and (d) 0.2 mm resin coated specimen exposed to seawater

6.2.4 Evaluate the Minimum Coating Thickness using Least Squares Quadratic Polynomial Approximation to Minimize the Effect of Seawater Exposure

- Resin coat application is found to help in minimizing the degradation of properties after seawater exposure. The chart of normalized flexural strength parameter ΔF/F (where ΔF is the difference of average flexural strength values before and after seawater exposure and F is the flexural strength value before exposure) for bare and resin-coated specimens is shown below. Similarly, the chart of normalized ILSS parameter ΔL/L (where ΔL is the difference of average ILSS values before and after seawater exposure and L is the ILSS value before exposure) for bare and resin-coated specimens, was also plotted using least squares quadratic polynomial approximation. The following were observations by plotting the curve:
- The plot in between coating thickness vs normalized parameters of flexural strength as depicted in Fig. 6.49 and 6.50, reveals that a minimum 0.25 mm coating will be enough for E-glass/epoxy laminate to restrict its degradation minimum after seawater exposure.
- The plot in between coating thickness vs normalized parameters of ILSS strength as depicted in Fig. 6.51, reveals that a minimum 0.3 mm coating will be enough for E-glass/epoxy laminate to restrict its degradation minimum after seawater exposure.

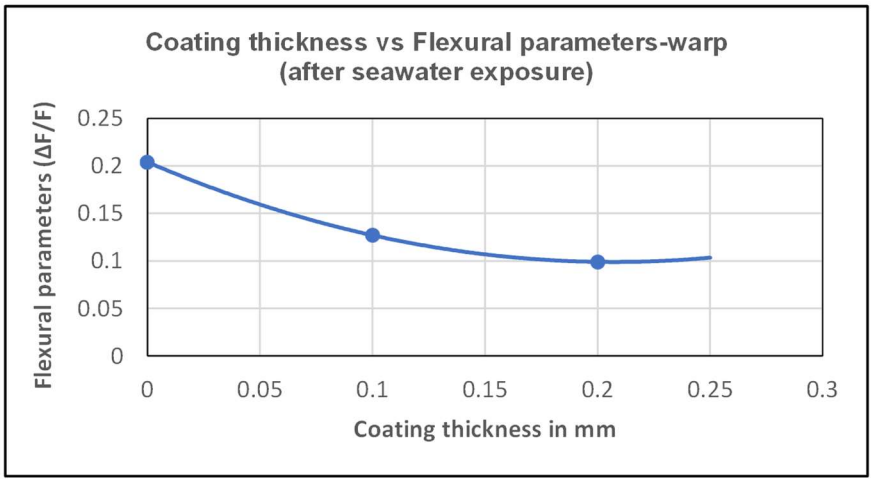


Fig. 6.49 Coating thickness vs Flexural parameters-warp

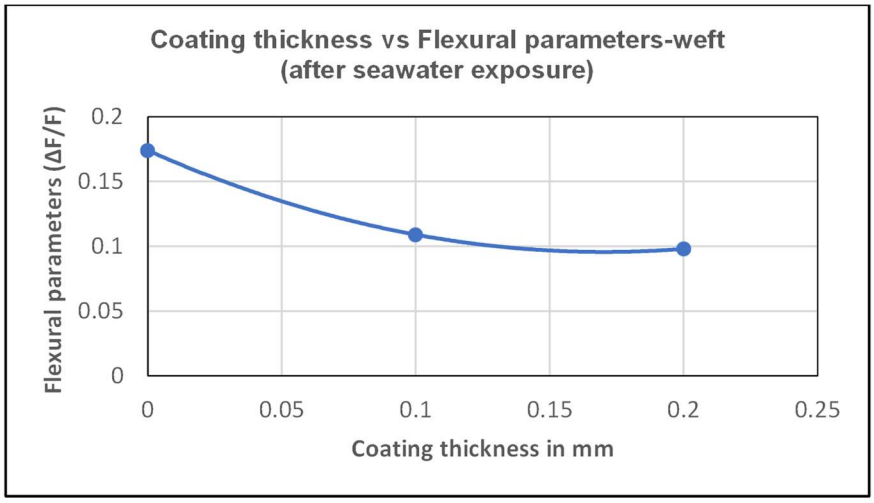


Fig. 6.50 Coating thickness vs Flexural parameters-weft

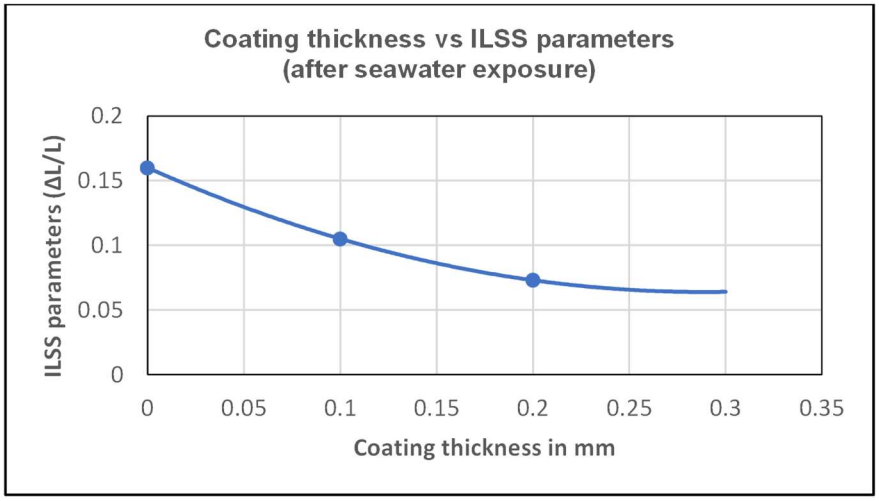


Fig. 6.51 Coating thickness vs ILSS parameters

6.2.5 Correlation of results with the Mechanism of Surface and Sub-surface Degradation of Different Types of Samples after Seawater Exposure

- The process of seawater diffusion in the composite is accelerated by its imperfection and thermal residual stresses. The seawater will be absorbed by capillary action from any surface crack or along the fiber matrix interface and this ingression-induced hydrolysis causes swelling of the matrix, which will change and relax the residual stress state near to fiber-matrix interface and provide a good mechanical adhesion at the interface. But hydrolysis will degrade the chemical bond at the interface, resulting in matrix and fiber separation. This will lead to low adherence between fiber and matrix interface for bare samples, resulting in interfacial failure.

- Fracture surface examination by scanning electron microscope (SEM) as depicted in Figures 6.46, 6.47, and 6.48 revealed that for tested specimens (bare condition), the diffusion of seawater into composites led to degradation because of plasticization of the matrix, matrix cracking, delamination, fiber debonding and resin crumbling.
- When the water molecules are penetrating through a polymer, degradation by the moisture occurs in the material. [Gu, H., and Hongxia, S. 2006] Also, plasticization and swelling can occur as shown in Fig. 6.52, due to debonding of Van Der Waals bonds between polymer chains by moisture. [Wong, K. J., 2013]. This phenomenon will be applicable once it will be exposed to seawater too.

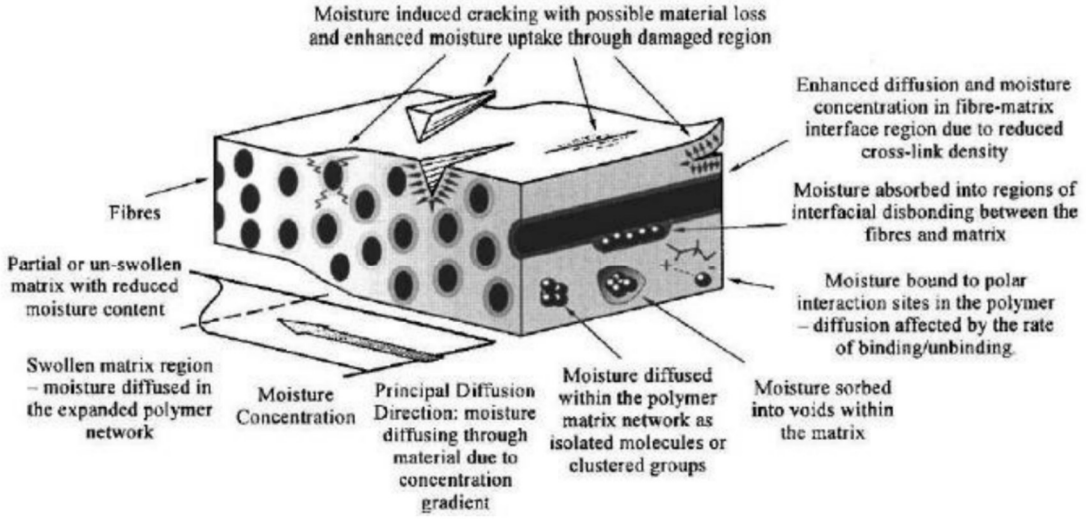


Fig. 6.52 Moisture absorption locations and mechanisms in polymeric composites [Source: Wong, K.J.,2013]

- Voids (spaces between molecules) inside the composites are getting expanded by water molecules and this will lead to a reduction of the interfacial strength of the polymer chains. Also, the water molecules will move more freely inside of the polymer and this will decrease the strength of the polymer. [Lv XJ, Zhang Q, Li XF, Xie GJ.,2008]
- Diffusion of seawater drives the way for hydrolysis reaction in the composite, including the hydrolysis of the matrix and their fiber-matrix interface. These phenomena are responsible for the reduction in tensile, compression, flexural, and shear strength. The seawater ingression was limited for resin-coated specimens, so its effect on these specimens was notably reduced. Similarly, Colin et al. [80] examined the various aspects of phenomenon of ageing generated by

water in the composite. The ageing induced physical process like matrix swelling and matrix plasticization, chemical process like hydrolysis were elaborated in their study.

- Tensile, compression, flexural, and shear modulus decreases rapidly over hydro aging, as moisture generally affects any property, which is dominated by the matrix and or interface. This hydrolysis may break the polymeric network.
- Moreover, specimens with 0.1 mm resin coating exhibited relatively more reduction than 0.2 mm resin coating. The specimens with 0.2 mm coating were limiting the reduction of tensile and flexural properties and not allowing significant penetration of seawater molecules inside the polymeric network. In addition to this, it was observed that 0.2 mm resin-coated test specimens were relatively more impermeable to seawater infiltration than 0.1 mm resin-coated specimens, and seawater diffusion was restricted to the top surface of the coating as shown in Fig.6.44 and 6.45 and prevented its penetration to resin inside the specimens, and fiber-matrix interfaces as depicted in SEM micrographs of Fig. 6.46, 6.47, 6.48 (c) and (d).
- The reported rate of absorption of seawater is also caused by the increased rate of moisture pick-up characteristics by the epoxy; since this resin, also called poly-epoxides, basically contains a group of epoxides. An epoxide is a cyclic ether equipped with a three-atom ring, which makes it strained and hence highly reactive. Epoxides can react with a broad range of nucleophiles like alcohols, amines, and water. When the specimen is immersed in seawater, the polar nature of the seawater molecules causes them to be attracted to the epoxide group. The polar attraction between the epoxide groups in polymeric chains gradually becomes weaker, resulting in a depletion of crystalline regions, therefore the interlaminar and in-plane shear properties will degrade.
- A stereo microscope examination revealed that epoxy resin coating is showing hydrophilic characteristics towards seawater ingression. 0.2 mm resin coating were providing a sacrificial layer relative to 0.1 mm and without resin coating scenario, to protect the parent material.
- Resin coat application is found to help in minimizing the degradation of properties after seawater exposure.

6.3 Comparative Study between ATF and Seawater Exposure

The following observations were recorded when a comparative study between ATF and seawater-exposed test specimens was investigated and as shown in Table 6.55.

- After ATF and Seawater exposure, mechanical properties in warp direction were degraded more than weft direction properties.
- After ATF and Seawater exposure, mechanical properties for 0.2 mm resin-coated specimens were degraded less compared to others.
- The % of degradation of mechanical properties was more for Seawater exposure relative to ATF exposure.
- In-plane shear properties were degraded the most because of ATF exposure, unlike compressive properties for seawater exposure.
- Resin coating over the test specimens will protect against ATF exposure, as epoxy coating is resistive against fuel and protect the said composite unlike seawater because of its hydrophilic characteristics it will provide a sacrificial layer to protect the parent material.

Table 6.55 Percentage (%) of degradation of mechanical properties when it is exposed to ATF and Seawater separately

Sl No.	Tensile Property		Compressive Property		Flexural Property		Inter laminar Shear Property	In-plane Shear Property
	Warp	Weft	Warp	Weft	Warp	Weft		
	(Strength after ATF Exposure)							
Bare	8.79	7.58	11.43	9.08	12.91	12.39	10.04	14.75
0.1 mm RC	5.84	5.14	7.07	5.65	9.16	8.24	7.83	11.22
0.2 mm RC	4.46	3.96	5.31	4.36	5.85	5.52	6.01	7.52
	(Strength after Seawater Exposure)							
Bare	24.09	22.96	28.91	25.26	20.38	17.41	16.01	26.10
0.1 mm RC	17.65	15.47	18.29	17.63	12.75	10.85	10.53	17.60
0.2 mm RC	10.23	8.37	12.37	10.19	9.93	9.80	7.36	9.96
	(Modulus after ATF Exposure)							
Bare	12.18	11.82	9.81	7.93	5.62	5.03	-	10.87
0.1 mm RC	7.67	6.92	6.32	4.87	4.42	3.32	-	8.94
0.2 mm RC	5.55	4.44	4.23	3.28	2.30	1.92	-	6.52
	(Modulus after Seawater Exposure)							
Bare	14.30	12.96	7.23	5.87	12.52	10.86	-	18.84
0.1 mm RC	8.57	8.85	4.95	3.54	8.58	7.71	-	11.11
0.2 mm RC	5.87	6.33	3.82	3.19	5.79	5.28	-	8.45

Note: Numerical values mentioned in the table were in %.

6.4 Summary

In this chapter, E-Glass epoxy composite were separately characterized under ATF & seawater exposure after saturating to respective environment. Test results of mechanical properties were recorded and its degradation were analyzed under ATF & seawater exposure and it was observed that E-glass/epoxy composite has degraded more in seawater exposure compared to ATF exposure. To understand the mechanism of degradation, stereo microscopy was carried out which has analyzed the surficial characteristics of degradation. Similarly for sub-surface characteristics of degradation were studied using Scanning Electron Microscopy (SEM). In last, to minimise the degradation effects of ATF & seawater exposure, minimum resin coating thickness were evaluated using least square quadratic polynomial approximation.

Chapter 7

Summary and Conclusions

7.1 Introduction

The conclusions were drawn from each chapter i.e., 3, 4, 5, and 6, and the implications of these conclusions were discussed in this chapter. Effect of ATF and seawater exposure on E-glass/epoxy composite were elaborately outlined and its degradation in respective environment were quantified for bare, with 0.1 mm and 0.2 mm resin coated conditions. Testing data outcome shows that mechanical properties of E-glass/ epoxy composite were degraded more in seawater exposure compared to ATF exposure.

7.2 Effect of ATF Exposure on E-glass/epoxy Composite

- The test specimens of tensile in warp and weft direction have not shown a significant reduction of properties w.r.t ATF exposure. Under ATF exposure, tensile strength in warp direction were reduced by 8.79%, 5.84% and 4.46% and in weft direction 7.58%, 5.14% and 3.96% for bare, with 0.1 mm and 0.2 mm resin coating conditions of the specimens.

- Comparatively test specimens for flexural properties exhibited more reduction than tensile one. As flexural strength in warp direction was reduced by 12.91%, 9.16% and 5.85% and in weft direction 12.39%, 8.24% and 5.52% for bare, with 0.1 mm and 0.2 mm resin coating conditions of the specimens.
- The tensile, compressive, and flexural properties in the weft direction exhibited relatively less reduction than the warp one.
- As the exposure was given to test specimens for three conditions namely bare, with 0.1 mm and 0.2 mm resin coating, it was observed that the test specimens which were resin coated with 0.2 mm were exhibiting less degradation compared to the other two conditions.
- It can be seen bare and with 0.1 mm resin-coated specimens got saturated relatively faster than 0.2 mm resin-coated ones. And the rate of absorption of these ingressions was less for 0.2 mm resin-coated specimens. It was as expected, as after applying the 0.2 mm resin coating not only porosity will be reduced but it will provide a nearly impermeable barrier too, to prevent the ATF ingression and will not allow the further degradation of tensile and flexural properties after exposure.
- The plot of coating thickness with normalized tensile strength parameters in warp and weft direction exhibits that 0.25 mm resin coating will be as good as virgin samples. Thus minimum 0.25 mm resin coat is recommended.
- The test specimens of Interlaminar and in-plane shear properties have shown a significant reduction of properties w.r.t ATF exposure. But test specimens for in-plane exhibited a 14.75% reduction in comparison to 10.04% of interlaminar.
- ATF exposure has reduced the in-plane shear modulus of the bare and resin-coated test specimens. For bare specimens, it was reduced to 10.87% and for 0.2 mm coating thickness, restricted to 6.52%.
- These property degradations were majorly due to resin plasticization and fiber-matrix interface degradation. As the fuel penetrates, it creates internal stresses in the cross-linked network, which will further lead to reduced mechanical properties.
- The plot of coating thickness with normalized ILSS parameters exhibits that 0.5 mm resin coating will be as good as virgin samples. Thus, a minimum 0.5 mm resin coat is recommended.
- As the exposure was given for three conditions, namely bare, 0.1 mm, and 0.2 mm resin coating, it was observed that the test specimens which were resin coated with 0.2 mm coating exhibited

lower degradation compared to the other two conditions. It was explicitly visible that bare and 0.1 mm resin-coated testing specimens got saturated comparably faster than 0.2 mm resin-coated ones.

- The rate of absorption of the ATF penetrations was less for 0.2 mm resin-coated specimens. It was expected as the resin coating was not allowing the ingression as, after the application of the same, porosity will be reduced. Application of resin coating to the test specimen of E-glass/epoxy composite is limiting the ATF ingression to the surface and restricting its detrimental effect on matrix interface degradation.

7.3 Effect of Seawater Exposure on E-glass/epoxy Composite

- The test specimens of tensile, compression, and flexural in warp and weft direction have shown a significant reduction of properties w.r.t seawater exposure for bare specimens, but for resin-coated specimens, the reduction will be less.
- The tensile strength in warp direction under seawater exposure were reduced by 24.09%, 17.65% and 10.23% and in weft direction 22.96%, 15.47% and 8.37% for bare, with 0.1 mm and 0.2 mm resin coating conditions of the specimens. Similarly flexural strength in warp direction were reduced by 20.38%, 12.75% and 9.93%, and in weft direction 17.41%, 10.85% and 9.80% for bare, with 0.1 mm and 0.2 mm resin coating conditions of the specimens. The tensile and flexural properties in the weft direction exhibited relatively less reduction than warp one under seawater exposure. This has happened due to differing fill counts of the fiber for this type of E-glass/epoxy woven fabric.
- Seawater exposure has degraded most to the compressive properties relatively to other mechanical parameters. As in warp direction compressive strength were reduced by 28.91%, 18.29% and 12.37%, and in weft direction reduction were 25.26%, 17.63% and 10.19%, for bare, with 0.1 mm and 0.2 mm resin coating conditions of the specimens.
- As the exposure was given for three conditions namely bare, 0.1 mm, and 0.2 mm resin coating, it was observed that the test specimens which were resin coated with 0.2 mm coating exhibited less decrement of properties compared to the other two conditions.
- It can be seen, that bare and 0.1 mm resin-coated specimens got saturated relatively faster w.r.t 0.2 mm resin-coated one. And the rate of absorption of seawater infiltration was less for 0.2 mm resin-coated specimens. It was expected, as resin coating was restricting the ingression, as, after

application of the same, porosity will be reduced. Application of resin coating to the test specimen of E-glass/epoxy composite is limiting the seawater ingression to the surface and restricting its detrimental effect on matrix interface degradation.

- The test specimens of Interlaminar and in-plane shear properties have shown a significant reduction of properties after seawater exposure. But test specimens exhibited that the specimens of interlaminar shear strength show less reduction in comparison to in-plane shear strength.
- Resin coat application is found to help in minimizing the degradation of properties after seawater exposure.

7.4 Scope for Future Research Work

This research work was focused on the degradation of E-glass/epoxy composites under hostile environments like ATF and seawater exposure, so there is scope to study the followings.

- Study of Degradation of mechanical properties of E-glass/epoxy composite under hygrothermal environment.
- Study of Degradation of mechanical properties of E-glass/epoxy composite under hydrothermal environment.
- Establish the comparison Study of barrier resin coated with different types of composites which involves different types of reinforcements and matrix systems under ATF and seawater.

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List of Publications

Journal Publications

Articles in Peer-Reviewed Journals

1. S. M. Shrivastava, G. Ramarao, Manoj K. Buragohain, N. Selvaraj, Effect of Aviation Turbine Fuel Exposure on Tensile and Flexural Properties of Glass Fiber Reinforced Epoxy Composite, Transactions of the Indian Institute of Metals, https://doi.org/ 10.1007/s12666-022-02805-0, Impact Factor: 1.391 **[SCIE]**
2. S. M. Shrivastava, G. Ramarao, Manoj K. Buragohain, N. Selvaraj, Effect of Aviation Turbine Fuel Exposure on Interlaminar and In-plane Shear Properties of Glass Fiber Reinforced Epoxy Composite, Defence Science Journal, https://doi.org/ 10.14429/dsj.72.18238, Impact Factor: 0.707 **[SCIE]**
3. S. M. Shrivastava, G. Ramarao, Manoj K. Buragohain, N. Selvaraj, Effect of Seawater Exposure on Tensile and Flexural Properties of E-glass/epoxy Composite, Material Today Proceedings, https://doi.org/10.1016/j.matpr.2023.02.143, Impact Factor: 1.46 **[Conference and Proceedings]**

Conferences

1. S. M. Shrivastava, G. Ramarao, Manoj K. Buragohain, N. Selvaraj, Effect of Seawater Exposure on Tensile and Flexural Properties of Glass/Epoxy Composite, ICFTMME-2022, 19-20 Aug 2022, SRM Institute of Science and Technology, Ghaziabad.

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ORIGINAL ARTICLE

Effect of Aviation Turbine Fuel Exposure on Tensile and Flexural Properties of Glass Fiber-Reinforced Epoxy Composite

S. M. Shrivastava[1] · G. Ramarao[1] · M. K. Buragohain[1] · N. Selvaraj[2]

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Abstract This study examined the effect of Aviation Turbine Fuel (ATF) exposure on tensile and flexural properties of E-Glass/epoxy composites. Specimens as per ASTM standards were made, and a portion of the specimens was coated with resin of varying thickness. Both the resin-coated and bare specimens were exposed to ATF by immersing them in the fuel till saturation. Tensile and flexural tests were carried out, and the results have been analyzed in the paper. Experimental results show that bare specimens exhibited a reduction in properties due to ATF ingression to the polymeric network and induced internal stresses, which not only degraded the reinforcement and fiber matrix adherence but created micro-cracks too in the resin at the interfaces. Resin-coated specimens limit fuel ingression, which has led to a slight reduction in properties. Fibers from the fractured surface were examined by scanning electron microscope, exhibiting slight degradation in fiber, interfacial characteristics and matrix.

Keywords Aviation turbine fuel · Fuel exposure · Tensile strength/stiffness · Flexural strength/stiffness · E-glass/epoxy

✉ S. M. Shrivastava
sure_shri@yahoo.com

[1] Advanced Systems Laboratory, DRDO, Hyderabad, Telangana 500069, India

[2] Mechanical Engineering Department, NIT, Warangal, Telangana 506004, India

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1 Introduction

Modern composites have been in existence for more than half a century; they have found widespread application in almost all industrial sectors and their usage is increasing at a rapid rate. Glass fiber-reinforced epoxy resin composites, a major class of composites, are used in the manufacture of many aerospace products that demand long service life under structural and environmental loads. In this connection, exposure to ATF is a critical one, as these materials tend to absorb ATF and deteriorate in respect of their mechanical properties. Tensile and flexural properties play a significant contribution in the design and long-term performance of the products made of glass fiber-reinforced composites. In this ware, to quantify the property degradation for use in design, the effect of ATF ingression on tensile and flexural properties of E-glass/epoxy composites is studied.

Kumarasamy et al. [1] investigated the impact of absorption of fuel on the mechanical properties of glass/epoxy laminates. Three different types of fuels viz. biofuel, aviation fuel and blended fuel mixture were chosen to see the effects over composite. Chakraverty et al. [2] reported a higher detrimental effect of hygrothermal exposure as compared to hydrothermal. Duration of given exposure also plays a significant role in the composite's mechanical stability irrespective of the type of exposure. Groysman [3] described and analyzed the problems of corrosion and their solutions in the gas, oil and refining industry. Yadav Khagendra Kumar [4] studied the influence of aviation fuel like AVGAS 100LL and ATF on mechanical properties like flexural strength and modulus of GFRP, made by vacuum infusion and reported

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Effect of Aviation Turbine Fuel Exposure on Interlaminar and In-plane Shear Properties of Glass Fiber Reinforced Epoxy Composite

S.M. Shrivastava,[#,*] G. Ramarao,[#] M.K. Buragohain[#] and N. Selvaraj[$]

[#]*DRDO - Advanced Systems Laboratory (ASL), Hyderabad – 500 069, India*
[$]*Department of ME, National Institute of Technology, Warangal – 506 004, India*
**Email: srivastavasm.asl@gov.in*

ABSTRACT

This study investigated the effect of aviation turbine fuel exposure on interlaminar and in-plane shear properties of E-glass/epoxy composite. The two types of test specimens, namely bare and resin-coated specimens with varying thicknesses as per the ASTM standard, were made out of E-glass/epoxy composite to evaluate their interlaminar and in-plane shear properties. These all types of specimens were immersed inside the aviation turbine fuel for two months and then afterward their effect on the reduction of mechanical properties like interlaminar and in-plane shear tests properties were experimentally investigated. Test results show that ATF fuel exposure has reduced the interlaminar shear strength by 10.04 %, 7.83 %, and 6.01 % for bare, with 0.1 mm and 0.2 mm resin coating, respectively. Similarly, in-plane shear strength was reduced by 14.75 %, 11.22 %, and 7.52 % for bare, with 0.1 mm and 0.2 mm resin coating, respectively, and in-plane shear modulus was reduced by 10.87 %, 8.94 %, and 6.52 % for bare, with 0.1 mm and 0.2 mm resin coating conditions as compared to as-received (without ATF exposure) specimens.

SEM micrographs and results too showed that properties were reduced and indicated that the glass/epoxy composite was resistive to fuel ingression. It was observed that bare specimens exhibited a reduction in shear properties due to ATF ingression to the polymeric network and induced internal stresses, which not only degraded the matrix and fiber-matrix adherence but created micro-cracks too in the resin at interfaces. Resin-coated specimens limit fuel ingression, which has led to a reduction in properties.

Keywords: Aviation turbine fuel; Mechanical property; Fuel exposure; E-glass/Epoxy; Inter laminar; In-plane shear

1. INTRODUCTION

The relentless passion of the aerospace industry to augment its performance is constantly driving the way to explore high-performance structural materials, i.e., composite. Composites are becoming the prime material in various industries because of their low weight and improved properties and are being used not only for structural application but ablative purposes too. These are widely used for spacecraft components, hot air balloons, gliders to fighter planes, space shuttles, and commercial aircraft. Increased usage of composite in the aerospace industry was possible because of its advantages like significant weight reduction, drapability, high thermal stability and impact resistance, resistance to fatigue and corrosion, and ease of assembly. Composite materials enable the integration of parts and lead to the replacement of various metallic components by a single monolithic composite component.[1] Rapidly, these key factors of the composite are outweighing the usage of metal in the aerospace industry.[2]

During the storage for a longer span, the long-term performance of the composite is to be ensured. As the structures, which are being made using these composites, need to be in service for a longer time, and it has to be exposed to various types of loading and environmental conditions. In aerospace, the composite drop tanks for fighter aircraft are made out of glass/epoxy composites and are being used to carry aviation turbine fuel. Hence, the glass/epoxy composite's tendency towards the absorption of the fuel will decide its degradation of mechanical properties. This degradation will be detrimental to the structural performance. So, studying the effect of ATF exposure on glass/epoxy composite has become very significant to establish the design margin for the composite structures, which store the fuel.

The interlaminar and in-plane shear properties play an important role in designing structural composites, so degradation of any kind will not only lead to failure but will restrict its usage.

The composite is porous and has the tendency of ATF ingression, to limit the same and to cater to the structure which is being used for storage of the liquids, providing the resin coating has become the must. Keeping that in the mind, bare and resin-coated specimens were tested after giving the ATF exposure separately to a distinct set of specimens, to know the actual effects on Interlaminar Shear Strength (ILSS) and In-Plane Shear Strength (IPSS).

Sandeep V. Gujjar, *et al.*[3] described the effect of different types of resin coating on a mild steel surface to get the ideal one. These resins (epoxy, phenolic, polyurethane, and polyester)

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Effect of seawater exposure on tensile and flexural properties of glass/epoxy composite

S.M. Shrivastava [a,*], G. Ramarao [a], M.K. Buragohain [a], N Selvaraj [b]

[a] Advanced Systems Laboratory, DRDO, Hyderabad-500069, Telangana, India
[b] Mechanical Engineering Department, N.I.T, Warangal-506004, Telangana, India

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ABSTRACT

In recent times, increasing and widespread usage of composite has become a necessity for aerospace, automobiles, and marine application due to its high specific strength and stiffness and it has attracted not only the necessity of retaining its mechanical properties under exposure to hostile environments but encouraged to explore the possibilities of barrier resin coating to reduce this degradation. This study examined the seawater exposure effect on the mechanical properties of E-glass/epoxy composite. The two varieties of test specimens namely bare and barrier resin-coated specimens were made as per the ASTM standard out of E-glass/epoxy composite laminate to evaluate its mechanical parameters and barrier resin-coated specimens were coated with the resin of varying thicknesses. These all type of specimens was immersed inside the seawater. Test results show that seawater exposure to test specimens has notably reduced their tensile and flexural properties as compared to as-received specimens. The seawater exposure has reduced the tensile strength by 24.09 %, 17.65 %, and 10.23 % in warp and 22.96 %, 15.47 %, and 8.37 % in weft direction for bare, with 0.1 mm and 0.2 mm resin coating, respectively. Similarly, flexural strength was reduced by 20.38 %, 12.75 %, and 9.93 % in warp and by 17.41 %, 10.85 %, and 9.80 % in weft direction for bare, with 0.1 mm and 0.2 mm resin coating, respectively as compared to as-received specimens.

It was observed from SEM micrographs too, that bare specimens exhibited a reduction in properties due to the diffusion of seawater, which will be followed by a reaction of hydrolysis in the composite. It was illustrated that the test specimens with barrier resin coated, limit the seawater ingression. The optimum thickness of the barrier resin coating was derived from plotting the normalized parameter w.r.t thickness of the coating.

Selection and peer-review under responsibility of the International Conference on Future Trends in Materials and Mechanical Engineering.

1. Introduction

Nowadays usage of composites, like glass fiber reinforced composites as structural load-bearing materials in aerospace applications, has become a reality [1]. E-glass fibers were chosen as it is mostly used for structural application in addition to their electrical applications and has excellent fiber forming capabilities, making this a prime candidate material as a reinforcement. This fiber is widely used among all fibrous reinforcement because of its low cost and early development compared to others. As the structures made of glass/epoxy composites are expected to be in service for a longer period and it has to undergo various types of loading and environmental conditions such as seawater, hygrothermal, UV radiation, chemical environments, biological conditions, etc., hence its long-term performance is to be ensured. The structure made with E-Glass epoxy composite is widely being used in aircraft, aerospace, and civil engineering projects because of its reduced weight and resistance towards corrosion, which will undergo a different type of exposure in its lifetime. For example, helicopters and ultralight aircraft which uses these composites, operate over water basins and fly at relatively low altitude. Their flight can be exposed to seawater and the influence of seawater and its absorption over these structures can be studied in terms of mechanical property degradation [2]. This degradation can affect structural performance. So, the study of this effect has become very important for said composite.

The tensile and flexural properties play an important role in designing structural composites, degradation of any kind will not

* Corresponding author.
E-mail address: sure_shri@yahoo.com (S.M. Shrivastava).

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on

Future Trends in Materials and Mechanical Engineering (ICFTMME- 2022)

Certificate of Participation

This is to certify that Mr./Ms./Dr./Prof. S. M. Shrivastava from Advanced Systems Laboratory D.R.D.O Hyderabad presented his/her research paper entitled

Effect of Seawater Exposure on Tensile and Flexural Properties of Glass/Epoxy Composite

in ICFTMME-2022, organized by the Department of Mechanical and Automobile Engineering, SRM IST, Delhi-NCR Campus, Ghaziabad, from 19th & 20th August, 2022.

Dr. Ankit Sonthalia	Dr. D. K. Sharma	Dr. S. Vishwanathan
Convenor	Dean	Director

Curriculum-Vitae

Name: Surendra Mohan Shrivastava

Designation: Scientist-F, in DRDO

Contact: +91 4024309235

Email: sure_shri@yahoo.com

Address: Advanced Systems Laboratory, DRDO,
Kanchanbagh, Hyderabad-500058,
Telangana, India

Area of Research: Primary research interests are in the fields of polymeric composite material and processes which include geodesic and non-geodesic filament winding and contact lay-up. In addition to this, in the area of composite overwrapped pressure vessel technology too.

Educational Qualification:

Degree	Institute	Specialization	Graduated
H. S. C.	Model School, Rewa	Mathematics, Science, and Social Studies	1991
B. E.	Government Engineering College, Rewa	Mechanical Engineering	1998
M. Tech	Indian Institute of Technology, Roorkee	Welding Engineering	2001
Ph. D.	National Institute of Technology, Warangal	Mechanical Engineering	Present

Work Experience:

1. 2001-Present – Scientist, Defence R & D Organisation (DRDO)

Publications: 3

1. Peer-Reviewed Journals: 3

Memberships

1. Life Member – Indian Society for Advancement of materials and Processing Engineering (ISAMPE)