ACCELERATED DEGRADATION OF COMPOSITE ADHESIVE BONDS

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DEDICATION

This thesis is dedicated my loving parents whose support, well wishes and love helped me to fulfill my dream and theirs too.

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Abstract
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Recent interest in reducing weight to improve fuel efficiency and incorporating composites in commercial aircraft had renewed interest in the behavior of adhesive bonding and their long term durability. This work considered an adhesive designed for moisture resistance (AF555) and an aerospace composite (Toray T899/39 00-2B). The aims of this study were to investigate the effect of moisture, accelerated degradation and surface preparation on adhesive bonds. The effects of moisture were considered by bonding dry and saturated adherends which were then immersed in water. Degradation was accelerated using constant temperature (-65,140,160 °F) and constant and fluctuating stress (45% to 90% of maximum load). Composite plaques were processed using three types of peel ply to consider surface preparation effects. Some surfaces were given subsequent abrasive treatment to consider benefits of secondary operations.

The diffusion coefficients of the adherend and adhesive were found to be 0.00106 mm^2 /hr and 0.0044 mm^2 /hr respectively for the saturation levels of 1.25 and 3.0 for the adherend and adhesive respectively. Results of shear and fracture tests showed a decrease in bond strength with increasing moisture content. Examination of the fracture surfaces showed increased adherend failure with moisture content. Adherend shear tests (no

adhesive) showed similar moisture sensitivity. The reduced bond strength was, therefore, attributed to the composite rather than the adhesive.

The crack growth rate of fractured coupons under fluctuating load was much higher than similar coupons with a constant load of the same magnitude. Additionally, the bond strength of shear coupons under constant load was observed to decrease by 18% over 1000 hours. Those observations suggest that stress can be used to accelerate degradation. Peel ply was observed to have a large effect on the bond strength. Polyester peel ply provided the highest strength followed by nylon and finally by SRB (siloxane coated polyester) peel plies. The peel ply effect was apparently related to debris left on the bond surface. Creep rupture tests showed that degradation was proportional to the bond quality, so that the weaker bonds also degraded faster.

Secondary abrasive techniques also had a measurable effect on bond quality. Sanding improved the fracture toughness by 8% while, grit blasted techniques decreased the fracture toughness by 22-50 %. The increase from sanding may be attributed to a cleaner and more uniform bond surface, while the decrease from grit blasting was shown to be due to surface erosion.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1) INTRODUCTION AND MOTIVATION

Some major manufacturing industries such as aircraft, marine and automotive are replacing the traditional fastening techniques namely bolting, screwing, welding and soldering with a modern technique called adhesive bonding. It is because adhesive bonds promise high structural integrity and reliability compared to the conventional way of fastening. However, the formation of a reliable adhesive bond between the composite aircraft components is complicated and needs greater insight. The critical parameters which dictate the durability of the adhesive bond include environmental effects, such as high elevated temperature and humidity, bond surface quality and service loads. Failure to understand the influence of these parameters may result in service disasters. The failure of the lap shear bonds of the Aloha flight 243 fuselage is one such service disaster which motivates the need for understanding the long term durability of adhesive bonds. Therefore, it is necessary to study the effects of these critical parameters on adhesive bond durability and quality which helps in building better bonded structures.

In an attempt to explicate the importance and motivation of the present work, a critical discourse on de-merits of the traditional methods of bonding and the remedies of critical problems by modern adhesive bonding are presented. Predominantly, riveting and welding were the two major ways of integrating structural components in typical industries like aircraft, marine and automotive. The major drawback was the presence of stress concentration hot spots which were primary sources of crack nucleation leading to

failure of the structure. Welding, on the other hand, resulted in residual stresses due to the thermal mismatch between the weld and the structure. These internal residual stresses were key reasons for crack nucleation under service loads. However, adhesive bonding has many advantages, over the traditional methods, prominent among them is uniform load distribution (which remedies the problem of stress concentration), see Fig.1.1. Moreover, it has good bond continuity, efficiency in bonding dissimilar materials of different thicknesses, greater fatigue resistance, and prevention of catalytic corrosion. It also has major economic advantages such as greater fuel efficiency (the Boeing 787 is 20% more fuel efficient when compared to current comparable aircraft; one third of the efficiency accounts to its light weight composite built structure [1]), improved design, easier assembly which requires less effort, and longer service life. Having known the importance of adhesive bonding in structural integrity, the present work draws motivation in gaining an understanding of the primary and secondary surface preparations of the composite. Also, the effects of moisture, temperature and stress on the adhesive bond integrity are studied to build better adhesive bonds.

The aims of this study were to investigate the effect of moisture, accelerated degradation and surface preparation on adhesive bonds. The Accelerated degradation study was done by conducting mechanical tests on BMS 8 276 (Toray) composite plaques bonded with AF555 (3M) adhesive. The effects of adherend moisture content, peel ply (fabric used for adherend surface preparation before bonding) and secondary techniques which are abrasive surface preparations on the bond strength, quality and durability were studied. The surface texture of a composite which is bonded with an adhesive is an important parameter which influences the strength of the bond. This texture is influenced by peel

2

ply which is used by the commercial aircraft industry. To further improve the quality of the composite bond surface secondary preparations such as abrasive techniques can also be used.
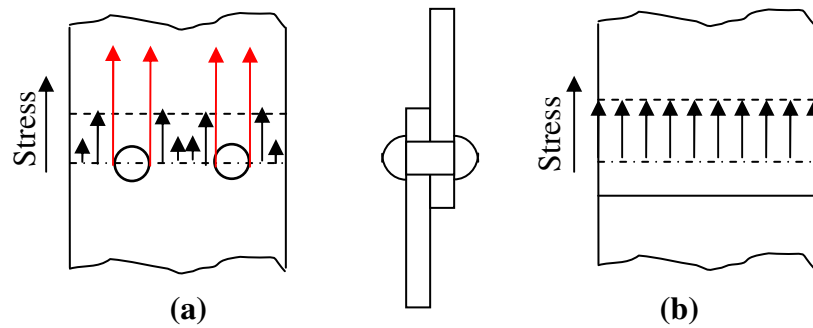
Fig. 1.1) Stress distribution in (a) riveted joints (b) adhesive bonding [2]

In order to achieve these goals, it was necessary to expose adhesively bonded joints to various environmental and loading conditions which simulate actual service conditions. The natural process of degradation by moisture absorption in adhesive structures is normally very slow and this makes it very difficult to reach an adequate degree of degradation in a structural test element in practical time-scales. It was found necessary, therefore, to speed up the moisture diffusion process by employing an accelerated conditioning technique that can ensure a representative level of degradation in a significantly reduced time. The usual approach to increase the diffusion rate of moisture into a material is by elevating the temperature and relative humidity of the conditioning environment [3, 4]. An alternative approach to try to reach an equilibrium conditioning is by accepting a chosen percentile of complete saturation. Conditioning to 95% of complete saturation will take less time than reaching 100% saturation [5].

At higher temperatures the polymer chains expand and create more volume for the water molecules to come in. This accelerates degradation as more amount of water molecules attack the polymer bonds. Supplementary polymer degradation occurs when these polymers are further exposed to stress. Therefore, the two main approaches of the accelerated degradation studies were to employ elevated temperatures and humidity levels (which increase the diffusion rate of moisture) and loading the specimen while exposed to these environments. Previously, tests have been done with the specimen conditioned unstressed in the desired environment and then evaluated. This may result in an overestimation of strength and fatigue life as it failed to account for the combined effects of stress and environment. Therefore, the test specimens were stressed while exposed to the desired conditioning environments which made then more capable of representing actual service conditions and accelerated degradation.

The thesis was organized as follows: Chapter 2 introduced the classification of composites and their primary manufacturing method along with the types of adhesives. In chapter 3, a literature review of the work done by various researchers was presented. In chapter 4, the test methods employed and the test setup were explained in detail which also explains the design of load frames. Chapter 5 presented details about material specifications and specimen preparation. Also, results of the work were presented where the effects of moisture content, peel ply surface preparations and secondary techniques were studied on the bonds involving composite adherends. Accelerated test methods were employed where temperature and stress were used to accelerate degradation.

CHAPTER 2

REVIEW OF COMPOSITES AND ADHESIVES

2.1) COMPOSITES CLASSIFICATION AND MANUFACTURING

A composite material, by the broadest definition is the one in which two or more materials that are different are combined to form a single structure. A generalized classification of composites is shown in Fig.2.1. Polymeric composites are of utmost interest to this study. They are highly engineered materials and are targeted for numerous applications.
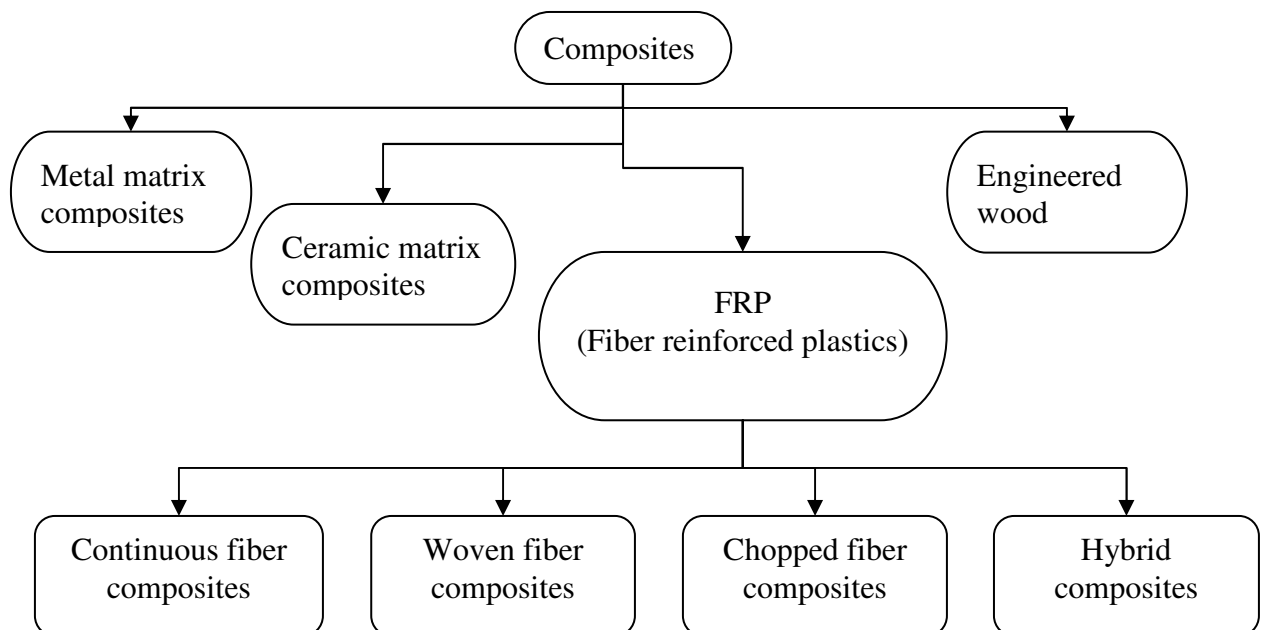
Fig. 2.1) Classification of composites

In polymeric composites, fiber reinforced plastics (FRP) are further classified by the type of fiber as carbon-fiber reinforced plastic (CRP) or glass-fiber reinforced plastic (GRP) and by the type of matrix as thermoplastic composites and thermoset composites. In FRP, one component is often a strong fiber such as fiberglass, Kelvar or carbon fiber that gives the material its tensile strength, while another component called the matrix is often a resin such as polyester, or epoxy that binds the fibers together and transfers load from broken fibers to unbroken ones and between fibers that are not oriented along the lines of tension. They also have fillers and additives which are mixed with the matrix material during fabrication. Fillers are additives which do not contribute to the mechanical properties of the composite but modify and enhance the final product for example, reduce weight, cost or protect against ultraviolet radiations. Resins usually are thermosetting plastics. Though thermoplastics are used, they are mostly used in the interior furnishings. Of all the resins used by the aerospace industry, epoxy resins have gained the widest acceptance. They are commonly used in adhesives and FRP. They are briefly described in sec 2.4.

According to the need for fiber placement and a particular application different types of fiber-reinforced composites are made. They are continuous fiber composites, woven fiber composites, chopped fiber composites and hybrid composites. The two main categories are short fiber reinforced composites and continuous fiber reinforced composites. Continuous reinforced materials often constitute a layered or laminated structure where individual fiber/matrix lamina are oriented in the required directions and bonded together to form a laminate. Although it is used extensively, the potential for delamination is still a major problem because the interlaminar strength is matrix-dominated. Chopped fiber

6

composites have short fibers randomly dispersed in the matrix. They are used extensively in high-volume applications due to low manufacturing cost, but their mechanical properties are considerably poorer than those of continuous fiber composites. Woven fiber composites are not susceptible to delamination as they do not have distinct lamina. The strength and stiffness are less as the fibers are not oriented straight. Finally, hybrid composite consist a mixture of chopped and continuous fibers or different fiber types such as glass or graphite.

In recent years, there is an increased use of composites for structural applications ranging from racecar bodies and aircraft components to wind turbine blades and building walls. Composites are replacing traditional all-metal constructions to reduce weight, increase corrosion resistance and support greater design flexibility. Moreover, with the right adhesive, composite assemblies can withstand exposure to temperature and environmental extremes as well as vibration even when the substrates have dissimilar coefficients of thermal expansion

COMPOSITE MANUFACTURING

The selection of the fabrication process depends on the constituent materials in the composite especially, the matrix material. The typical fabrication processes in manufacturing composite structural parts are open mold hand lay-up, autoclave, compression molding, filament winding, pultrusion, reinforced reaction injection molding (RRIM), thermoplastic molding, resin transfer molding (RTM) and structural reaction injection molding (SRIM). The fabrication processes used for polymer composites with various types or fiber reinforcement is summarized in the table 2.1. The fabrication

process used for building the composite samples in the present work was done using an autoclave. This is the only process described in this section. Before describing the fabrication process, a brief introduction of prepreg, peel ply, vacuum bagging and adherend surface preparations is necessary.

Table 2.1 Fabrication processes for polymer matrix composites [6]

Process	Type of fiber reinforcement			
	Continuous	Chopped	Woven	Hybrid
Open Mold - Hand lay-up		x	x	
- Spray-up		x		
Autoclave	X		x	
Compression Molding	X	x	x	x
Filament Winding	X			
Pultrusion	X		x	
Reinforced reaction injection molding (RRIM)		x		
Thermoplastic molding	X	x	x	x
Resin transfer molding (RTM)	X	x	x	x
Structural reaction injection molding (SRIM)	X	x	x	x

Prepreg

Prepreg is a tape consisting of fibers pre-coated with the polymer resin. If a thermosetting resin is used, the resin coating is partially cured, and the tape must be kept refrigerated to

8

prevent full curing until the final use. If a thermoplastic resin is used the tape can be stored at room temperature.

Peel Ply

A removable outside fabric ply molded onto the surface of a laminate to provide a bondable finish. They produce a range of finishes like fine, medium and coarse depending on the peel ply type. Peel plies used in this work were 60001, nylon, and SRB with 60001 being the finest and SRB being coarse manufactured by Precision Fabrics. Fig 2.2 shows the peel plies.

Fig.2.2) Peel plies and Adhesive

In the vacuum bagging process the prepreg laminate to be cured was covered with a peel ply and wrapped with a thick layer of absorbent material, called "breather." The whole assembly was then inserted into the vacuum bag and the air inside was removed by a vacuum pump. The vacuum created inside pushed the vacuum bag tightly from all sides, pressing the bag against the breather. While curing process the excess epoxy was squeezed out of the cloth and passed through the peel ply. It was soaked up by the breather. The breather allowed the gases to pass out of the bag and exit through the tube. The Fig. 2.3 shows a typical lay-up in a vacuum bag and Fig 2.4 shows vacuum bagging.
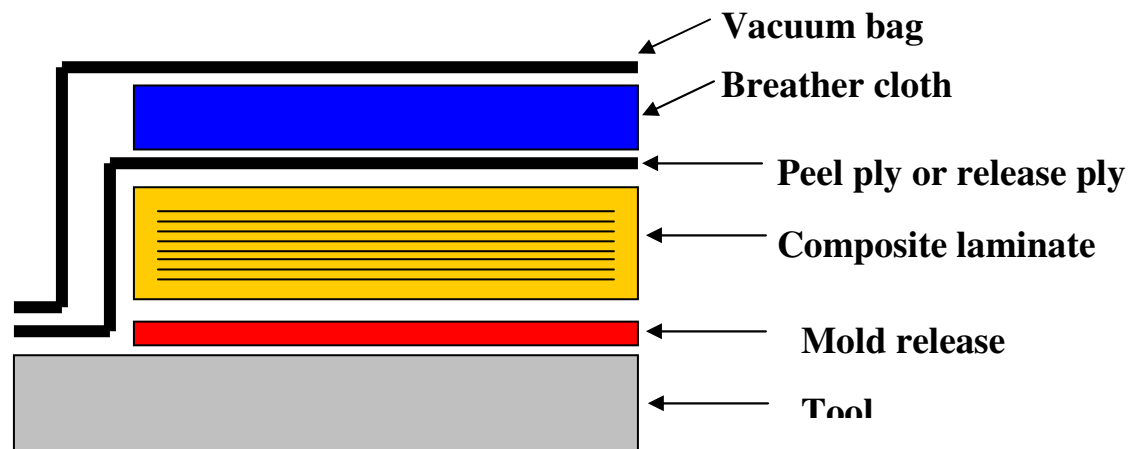
Fig. 2.3) Schematic lay-up of Composite adherends

Fig. 2.4) Vacuum bagging

Adherend surface preparation

Adherend surface preparation plays an important role in the development and evaluation of bonded joints. Peel ply contamination and insufficient surface roughening play a critical role and these factors can prevent adhesives from bonding properly to composites. They result in interfacial failures at lower loads relative to the loads that fail cohesively. The basic principles of surface preparation are that the surface must be free of contamination and be able to adhere to the adhesive. Secondary surface preparations remove or abrade the substrate to increase the surface area for mechanical interlocking

with the adhesive. Surface preparation varies depending on material type. Composites use sanding and grinding, surface texturing, grit blasting or solvent cleaning. Solvents like acetone, MEK and trichloroethylene must be used for degreasing as they evaporate quickly and leave no film on the substrate. Grit blasting is a finishing process in which abrasive particles are blasted onto the adherend surfaces in order to produce a roughened surface. The grit usually consists of iron, aluminum oxide, or any crushed or irregular abrasive.

Autoclave

Autoclave is a heated pressure vessel which is typically used to cure polymeric composites. It is mainly used by the aerospace industry to fabricate its aircraft parts. A typical fabrication process of a composite structure is usually done by curing a desired lay-up of prepreg tapes in the autoclave. In this study, curing of composite was done with a stack of unidirectional prepreg tapes which were laid on a mold covered with peel ply followed by a breather cloth on the top. This set up was vacuum bagged and was subjected to the required cure temperature and pressure in the autoclave. A typical autoclave setup is drawn below in Fig. 2.5.

2.2) <u>TYPES OF ADHESIVES</u>

To determine which structural adhesive is appropriate for a specific project, its characteristics and capabilities must be evaluated along with type of substrates, processing requirements anticipated operating conditions and desired end-part
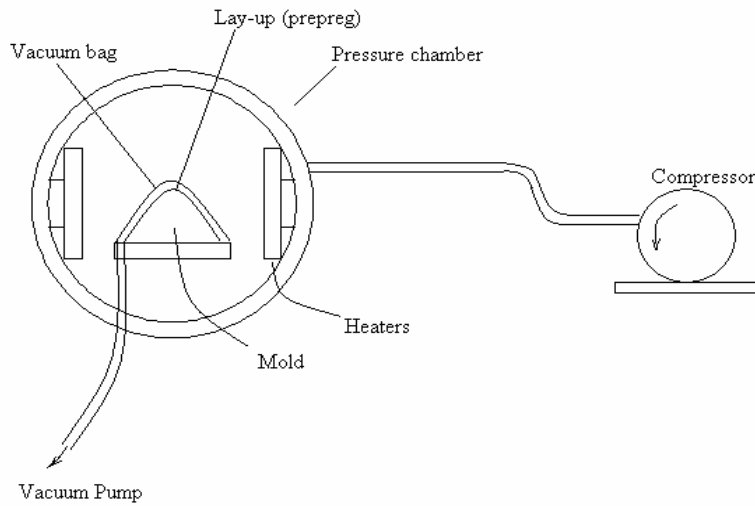
Fig. 2.5) Autoclave setup

performance. The three primary types of structural adhesives used in bonding composite parts are epoxies, polyurethanes and methacrylates. Each has its own distinct chemical composition and physical properties. They bond substrates with sufficient strength to transfer high loads without failure. In applications for a specific project the appropriate adhesive is selected by identifying its characteristics and capabilities with substrates, processing requirements, anticipated operating conditions and desired end-part performance. Comparison of typical properties of the epoxies, polyurethanes and methacrylates is shown in the tables 2.2 and 2.3 which are followed by a briefing of epoxies in the next subsection.

Epoxy adhesives

Many epoxies contain additives such as organic solvents, fillers such as fiberglass or sand, and pigments. Single molecules (monomers) of epoxy resin and the curing agent

13

Table 2.2 Typical Mixed Properties of adhesives [7]

Product	Viscosity	Work Life		Minimum Cure Time	
	cps	Min. @ 77°F	Hrs. @ 77°F	Min. @ 140°F	Min. @ 212°F
Epoxies	3500 to paste	1.5 to 2	1 to 48	10 to 60	2 to 20
Polyurethanes	9000 to paste	1 to 20	6 to 48	2 to 120	10 to 30
Methacrylates	45,000 to 180,000	3 to 10	15 to 30 min.	12	2

Table 2.3 Typical Cured Properties of adhesives [7]

Product	Shore hardness	Lap shear Strength		Elongation	Substrates
		psi @ 77°F	psi @ 180°F	%	
Epoxies	66 to 90D	1990 to 4100	140 to 3400	1 to 55	Metals and rigid plastics
Polyurethanes	10A to 70D	1700 to 2800	450 to 3500	30 to 250	Thermoplastics, GRP
Methacrylates	75 to 78D	2650 to 3625	1150 to 2465	35 to 75	Metals, Composites, thermoplastics

combine to form long chains of molecules (polymers). During curing epoxy adhesive becomes a hard polymer. The curing time of some epoxies is as little as five minutes where as others need additional time and heat to harden. The characteristics of hardened epoxies such as firmness or flexibility, or resistance to heat or chemicals depend on the epoxy monomers, curing agents, solvents, and fillers that are added. They have very low viscosities similar to water and very high viscosities similar to peanut butter accommodating numerous application methods. Their maximum service temperatures can be as high as 350°F (depending on formulation and cure temperatures) and elongations are typically from 1 to 55%. Hardness is in the range of 66 to 90D and lap shear strengths are from 1990 to 4100 psi.

Advantages and limitations of epoxies

Epoxies form strong rigid bonds to a wide variety of materials including thermoset plastics used in composite structures, metals, and wood. They feature good electrical insulating characteristics as well as excellent solvent and chemical resistance. They can cure at room temperature and are easy to handle as they attain handling strength in as little as five minutes. When exposed to mechanical stresses and vibration epoxies produce tough, durable joints and retain their strength even at elevated temperatures. These materials have good strength, produce limited volatiles during curing, and have low shrinkage. The only limitation of these adhesives is that they can have low peel strength and flexibility and can be brittle.

Epoxies are widely used in structural bonding in the marine, automotive, aerospace, appliance, general assembly and construction industries. Examples are bonding frames of the racecar vehicles, bonding metal hubs to GRP rotor blades in wind generators.

Motivated by the mechanical advantages of adhesive bonding in composite structures various researchers have studied adhesive bonding and its study of degradation under its service environments. Before reviewing the methodology of the testing methods, a brief review of the work done by various researchers is presented in the following section.

2.3) **TYPES OF FAILURE MODES**

An adhesively bonded composite joint typically fails in three types of failure modes. Fig 2.6 shows the three types of failure modes, viz. adherend failure, cohesive failure and adhesion failure. When the bonded composite fails with in the adherend (composite) it is considered as adherend failure. And, when it fails with in the adhesive it is called cohesive failure while, it fails between the adherend and the adhesive it is called adhesion failure. Bonds which are bonded well fail either in cohesive or adherend failure modes. Bonds which fail at the adhesive-adherend interface indicate that they are weak due to the presence of impurities.
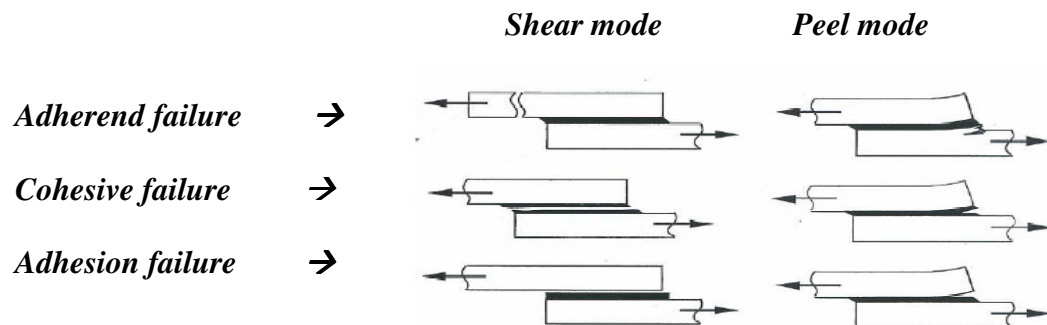
Fig. 2.6) Failure modes of an adhesively bonded composite joint [8]

CHAPTER 3

LITERATURE REVIEW

Among the many environmental conditions that may influence composite mechanical behavior hygro and thermal effects are discussed here, as they are the most important effects on the polymer matrix composites. The sorption of solvents and their effects on polymeric composites' performance is interdisciplinary and deals with polymer science and applied mechanics. The following subsection discusses the hygro-thermal effects on the polymeric composites followed by work done on the combined effects of load, temperature and moisture which is followed by examples of failure of adhesive joints and the work done on the strength and durability of adhesively bonded joints.

3.1) HYGRO-THERMAL EFFECTS ON THE MATERIAL PROPERTIES OF POLYMERS AND POLYMERIC COMPOSITES

The kinetics of fluid sorption in polymers has been studied for about one and half centuries beginning with Fick (1855). All polymers and polymeric composites absorb moisture in a humid atmosphere and when immersed in water. Several researchers conducted experiments on moisture uptake and their assumptions were based on one-dimensional diffusion called Fick's law. It defines the simplest model of diffusion of fluids in a solid represented by the curve LF in Fig. 3.1, recording the weight gain data *versus* time. But in many circumstances weight-gain data for the sorption and desorption of fluids in polymers do not follow the classical diffusion theory (linear Fick's law) and results in non-fickian behavior. Moisture diffusion in polymeric composites has been

shown to be Fickian and non-Fickian [9] in character. Weitsman in 1998 obtained some typical deviations from classical predictions shown in the Fig. 3.1.
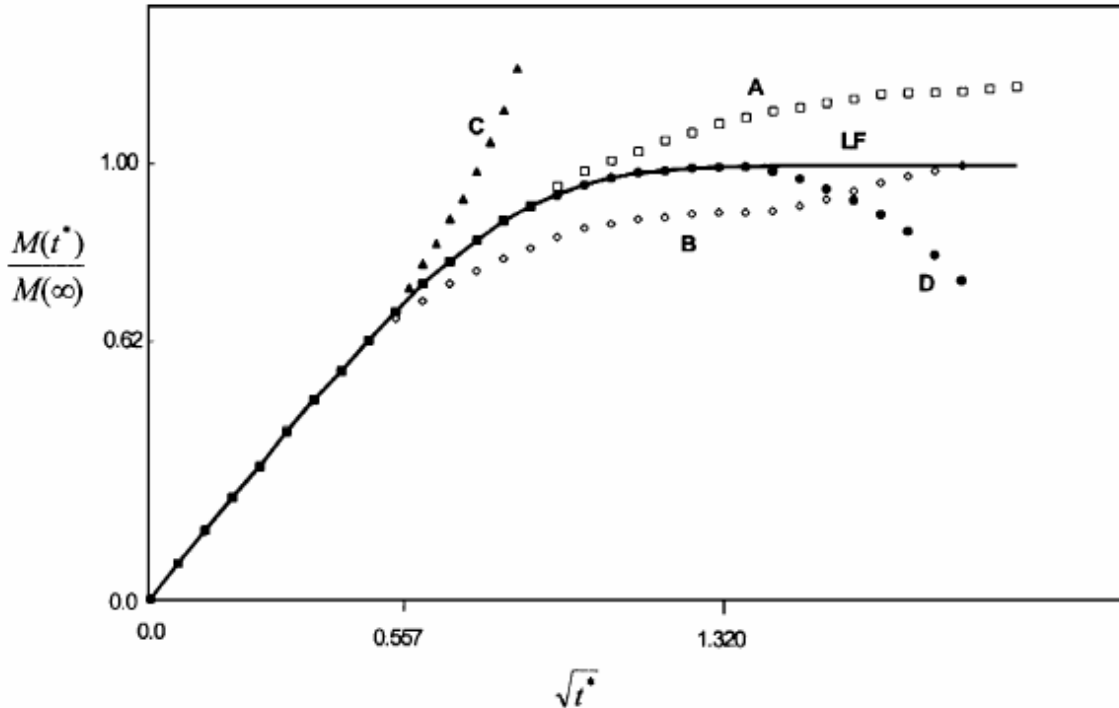
Fig. 3.1) Schematic curves representing four categories of recorded non-fickian weight-gain sorption data in polymers and polymeric composites [9]

Three types of irregular behavior of polymers are shown by curves "A", "B" and "S" in the Fig. 3.1 (Weitsman, 1998). In the figure curve "A" denotes "Pseudo fickian" and corresponds to continuous gradual increase in weight gain- never attaining equilibrium and curve "B represents a "two stage diffusion" behavior while LF corresponds to a linear Fickian behavior. However, curve "C" and "D" corresponds to rapid increase in fluid and weight loss respectively. Weight-gain behavior along C is likely associated with rapid increase in moisture content within the composite which is usually accompanied by large deformation, damage growth, material break down, and/or mechanical failure.

While D indicates leaching of material (polymer or glass fiber) into the ambient, it refers to irreversible chemical or physical breakdown of a material. The weight loss is due to hydrolysis, which is chain breakdown or separation of side groups from polymeric chains. It can also be dissociation of matter located at the fiber-matrix interfaces.

One should be cautious in choosing a specific composite material to operate under more severe circumstances by using the weight-gain data collected in a limited range of exposures. Weistman's [9] weight gain observations of graphite /epoxy (Gr/Ep) and glass/epoxy (Gl/Ep) composites followed a benign curve B for three years and beyond that the data shifted towards curves C and D indicating permanent losses. This material degradation was due to the slowly processing chemical reaction.

Researchers have documented the effects of moisture on aerospace composites. This moisture reduced the glass transition temperature of the polymeric matrix and reduced the strength of the fiber /matrix interface. Composites having some continuous fibers and high fiber contents absorbed little moisture and show negligible change in modulus with soaking time [6]. In hot, wet conditions the most significant loss was observed in the compressive strength of a composite material. The moisture absorption was usually limited to the matrix while graphite or glass fibers absorption was comparatively low. The effect of moisture at the fiber/matrix interface may be two-fold. First, the moisture can directly reduce the chemical bonding strength at the interface. Second, the matrix swelling that accompanies moisture absorption can change the residual stresses that develop due to curing-related shrinkage of the matrix, during cooling down from T_g and due to the differences in the thermal expansions between the polymer matrix and the

fibers [10]. Because the residual stresses that develop in the matrix during cool-down are tensile parallel to the fibers and compressive at the fiber/matrix interface, moisture absorption and the associated swelling will reduce the tensile stress parallel to the fibers and will also reduce the compressive stresses present at the fiber matrix interface. The interfacial shear strength is due to mechanical as well as chemical interaction at the interface, this relaxation of residual compressive stresses at the interface will reduce the interfacial shear strength [11]. Generally the tensile strength of unidirectional composites is unaffected by moisture absorption. However, reductions in compressive strength of unidirectional composites loaded parallel to the fiber direction or transverse tensile strength have indicated strength reductions that range from 10%-50% [11].

In this work tensile loading was applied on the bonded composite specimens to expose the adhesive under shear load. A thick wide area lap shear specimen was used which was designed for tensile tests [5]. Residual lap shear strengths were found subjecting the specimens to tension after the specimens were exposed to harsh environments. Also, diffusivity is observed to increase under tensile loading and decrease under compressive loading which helps diffusion to occur faster [6]. In addition, compressions tests of the composite alone were also done to study the moisture degradation effects on the unidirectional composites.

3.2) <u>COMBINED EFFECTS OF LOAD, TEMPERATURE AND MOISTURE</u>

The study of the combined effects of mechanical loading, temperature and moisture is scarce in literature. Studies performed on either of the mechanical stress and environmental attacks are numerous. Research effort by Buck et al. [12] recently reported

that the combination of moisture and sustained load at elevated temperatures on the E-glass/vinyl-ester composites caused a significant decrease in the ultimate tensile strength.

Beckry Abdel-Madgid performed a study on the combined effects of load, moisture and temperature on the properties of E-glass/epoxy composites and found that for short durations (500 and 1000 h) of applied tensile stress and submergence in distilled water at room temperature, the material exhibited an increase in strain to failure. These changes in properties were caused by fiber straightening and matrix plasticization.

After long duration (3000h) of applied stress and moisture conditioning at room temperature, a significant decrease in strength and strain-to-failure was exhibited indicating crack propagation in the matrix and at the interface causing the material to be less ductile and more brittle. When the conditioning temperature was raised to 65 ºC for 1000 hr, the strength decreased by 18%, and the modulus decreased by 28% while the strain-to-failure increased by 18%. The reduction is caused by matrix plasticization due to load, moisture and temperature. During a tensile test to failure the data showed that at higher temperatures, moisture penetrates the fiber/matrix interface resulting in massive fiber debonding. These results indicated that constant stress may have a positive effect in the short term, and that extended exposure to moisture at room temperature leads to brittle/ catastrophic failure while exposure at high temperatures results in ductile failure of the continuously laminated E-glass/epoxy composites

Substantial reductions in both strength and stiffness under stress-strain curve of a typical graphite epoxy matrix were observed previously under various combinations of temperature and absorbed moisture [13]. Also, strength and stiffness decreased when the

same material was observed under traverse loading. It is the most severe hygrothermal degradation. Similar degradation was observed in the case of in-plane shear loading of the composite since the behavior was matrix dominated in both the cases. On the other hand, the corresponding stress-strain curves for the composite under longitudinal loading showed little effect as the longitudinal strength and stiffness was fiber dominated.

Work done by L.V. Smith and K.L. DeVries [14] observed that synergistic effects of stress and NO_x environment on polymeric fibers. It was found that combined effects of stress and environments degraded both bare nylon-6 fibers and samples where fibers were embedded in epoxy matrix. It was observed that embedded samples in similar environments had 52-72% more residual strengths when unstressed than samples stressed to 20-27% of the ultimate fiber strength. It was observed that stress had pronounced effect on the rate of degradation down to stresses of approximately 20% of the ultimate fiber strength. The rate of degradation was very large with increase in concentrations, stress levels and exposure times. The rapid degradation was due to the weaker bonds that epoxies usually form to nylon fibers. The interface region between the epoxy and nylon was expected to have provided a path for the ingress of the environment. Cracking was evidently found parallel to the fiber direction. It was observed that stress induced slippage between the fibers of different lengths.

3.3) <u>FAILURE OF THE ADHESIVE JOINTS</u>

The failure of the Aloha flight 243 fuselage illustrates some important reasons as why the failure of adhesive joints occurs. From the report by the National Transportation Safety Board (NTSB), failure of Aloha airlines in 1988 was one evident example which

happened due to failure of adhesive joints accompanied by cracking. The report determined that the probable cause for the aircraft failure was due to significant de-bonding and fatigue damage that ultimately led to failure of the lap joint at S-10L stringer and separation of the fuselage upper lobe [15] (Stringers are long, thin horizontal members which give support and definition to the skin of a structure. They are used to connect upright members). The original design of the lap joint consisted of adjacent skin panels that were longitudinally overlapped about three inches. The overlapped area was fastened with three rows of rivets and a cold bonding adhesive as shown in Fig. 3.2(a). These fastened joints were designed to withstand cyclic compression/decompression loads associated with take offs and landings. It was learned that the cold bond adhesive was highly susceptible to corrosion and disbonding. The hoop stress was transferred through the bonded joint and not the rivets but once the bond de-bonded the load was transferred directly through the rivets. It was reported that the countersunk outer sheet formed a sharp, knife edge configuration, as shown in Fig. 3.2 (a). They created significant stress concentrations and thus made the rivet holes susceptible to cracking and eventually to multiple site damage (see Fig. 3.2 (b)). Lap joints on later productions were modified to avoid such structural problems.
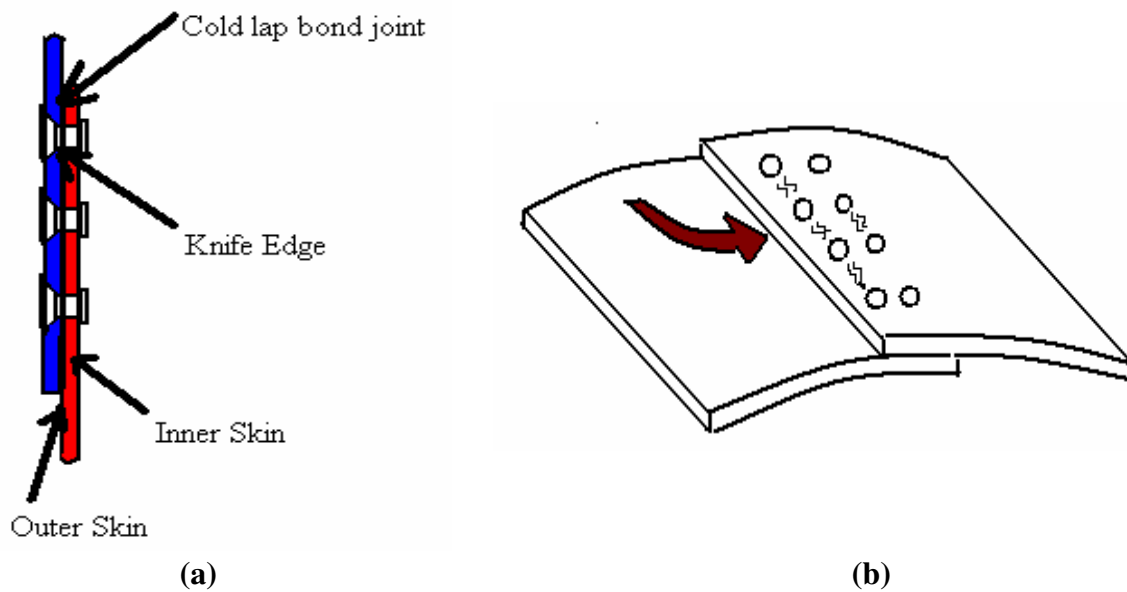
Figure 3.2) (a) Lap Joint Design (b) Multiple site damage [15]

While studying the environmental moisture effects on the performance of an adhesive joint it was known that adhesives especially epoxies were hydrophilic in nature i.e. absorb moisture. The cured joint's pre-bond absorbed moisture at elevated temperature may affect a bonded-joint by interfering with surface wetting, the cure reaction of the adhesive and causes excessive voiding in the adhesive [16]. These problems can generally be overcome through drying of adherends and dry storage of adhesives prior to bonding. The post-bond environmental effect on the adhesive joint was absorption of moisture into the joint. This resulted in an increase in compliance. Consequently, the interfacial bond strength between the adhesive and the adherend was reduced with increased chances of low strength interfacial adhesion failures.

To understand the durability of bonded structures, however, it is necessary to examine the effect of environmental exposure on the performance of the adhesive bondline. The

various mechanisms that cause degradation of the joints can occur before, during and after the installation/cure of the joint.

3.4) STRENGTH AND DURABILITY OF ADHESIVELY BONDED JOINTS

There are many durability factors that need to be characterized for an adhesively bonded joint. Bonded parts have resulted in debonding and corrosion of bondline after relatively short periods. Moreover, as the traditional evaluation tests and methods have not been capable of predicting the de-bonding in service, researchers have developed test methods to evaluate adhesive bond strength as will be described in chapter four in detail.

Previous work of adhesive bonded surface durability of an aluminum structure resulted in de-bonding of aluminum adherends with 250 ºF curing epoxy systems [17]. Delamination was interfacial at the primer/metal oxide interface, and progressive with time, involving relatively low stresses showing no plastic deformations of the metal. Delamination initiated at an exposed bondline such as edge or from the fastener hole.

For adhesive bond evaluation, generally, three traditional test methods were previously studied: (1) lap shear testing as a function of temperature, (2) peel testing as a function of temperature, and (3) exposure of unstressed lap shear specimens to various environments before testing. An examination done for these fractured specimens showed that these tests do not duplicate the characteristics of the service de-bonding, since these tests produced cohesive fracture with in the adhesive. The fracture surfaces showed de-bonding when the lap shear specimens were exposed to moisture environments for prolonged periods under load. A comparison of the residual lap shear strength after environmental exposure

under load and without load showed that the unstressed specimen retains a relatively high percentage of its lap shear strength even after very long exposure (> 50 days @ 140 F, 100% RH) while the specimen under load of 1200 psi failed in short period of 20 days and 900 psi failed at 40 days [18].

Recently much work has been done on the surface preparation of the composite adherends which affect the strength and durability of the composite adhesive bonds. The effect of surface preparations of the adherends was the major factor evaluating the effects of chemical contamination from peel ply release agents. The chemical and mechanical effects of grit blasting on the fracture toughness and failure mode of adhesively bonded composite joints are also under study. Over time in service other failures can occur as they were exposed to harsh environments [19, 20].

The qualitative analysis of modes of failure is as important as the quantitative values like loads and displacements. Joints which are bonded well should fail with in the adhesive (cohesive failure) or within the adherends (interlaminar failure) when broken apart. Failure at the adherend- adhesive interface (interfacial failure) generally indicates that the bond was not formed properly. Adherends cured against nylon coated release fabrics become chemically contaminated resulting in interfacial bond failure with low critical strain energy release rates (G_{IC}). The chemical contamination is due to the silicone and siloxane agents deposited onto the adherend surface from the peel ply during cure. While, adherends cured against vacuum bags had cohesive and interlaminar failures with higher G_{IC} values [20]. Grit blasting increased all the joints' G_{IC} values but did not change their modes of failure. This implied partial removal of the silicone and siloxane peel ply agents

ought to improve the bond strength, but not enough to change the mode of failure. The effects of peel plies, release fabrics, grit blasting and adhesive type (paste or film type) were evaluated.

The path by the load displacement curve of a double cantilever beam specimen defines a continuous crack growth if the crack propagation is smooth or an intermittent crack growth if the crack propagation is jagged. It is found that surfaces cured against the vacuum bag results in continuous crack growth and surfaces cured against nylon release fabric resulted in intermittent crack growth behavior as shown in the Fig. 3.3.

Grit blasting resulted in an increase in the initial failure load. The failure modes found for different cases were determined. Bonds made to nylon peel ply surfaces failed interfacially where as bonds made to vacuum bag surfaces failed in cohesive and interlaminar failure modes. Grit blasting did not change the mode of failure [20]. The critical strain energy release rates (G_{IC}) of tested coupons showed that bonds made to surfaces cured against vacuum bag material produces higher G_{IC} values than bonds to surfaces cured against nylon release fabric. Bonds made to grit-blasted surfaces produced higher G_{IC} values than non-blasted ones regardless of other surface preparation. The girt blasting varies according to its variables like pressure, grit size number of passes and speed of passes. Grit blasting had benefits to nylon peel ply surface preparations. Grit blasting has partially removed silicone and siloxane peel ply release agents thus improving the bond strength. However, this work considered grit blasting made on polyester peel ply.
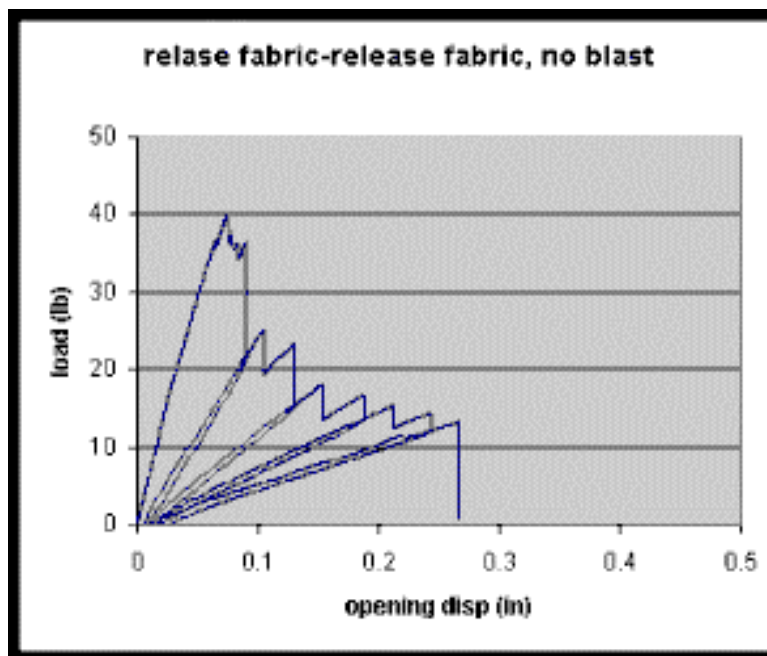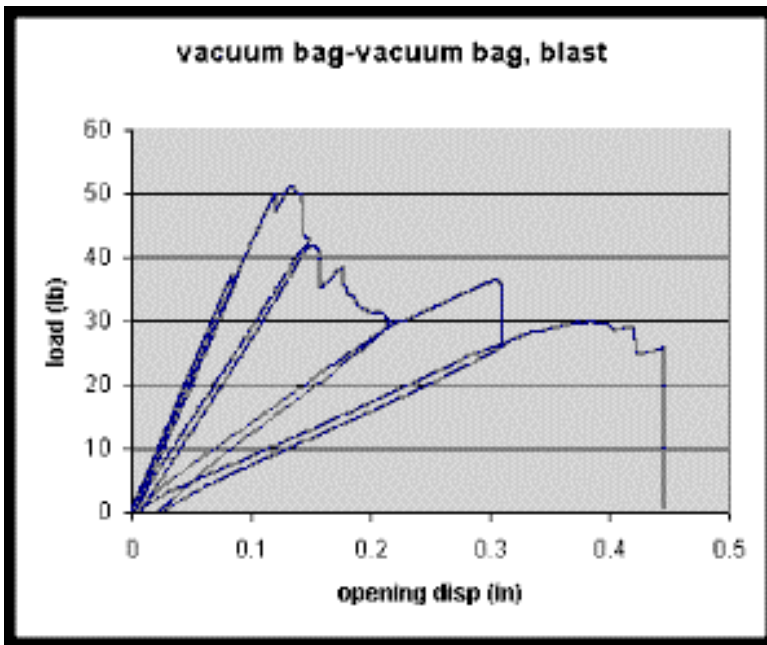
Fig. 3.3) Sample DCB test load displacement curves [20]

From the literature review it can be said that much work was done previously studying the hygro-thermal effects of the polymeric composites. The combined effects of moisture, load and temperature on the polymers and fibers was also done. However, there was not much literature found on the adhesive bonds involving composite adherends. Also, there is scarcity on the effects of surface treatments and degradation studies of composite adhesive bonds. In order to assure greater confidence in using composite structures there is a necessity to study and understand the long term durability of the adhesive bonds and also, to study the environmental effects on these bonds. Therefore, the present work considered the effect of moisture content, peel ply and secondary abrasive techniques on the adhesive bonds made from composite adherends. Also, accelerated degradation methods were employed where temperature and stress were used to accelerate degradation. Tensile, peel, compressive and bi-axial stresses were applied on the specimens. Creep and fatigue modes of stresses were employed under peel loading. These studies were done on a special aircraft composite, BMS 8 276 and toughened 3M adhesive, AF555.

CHAPTER 4

TEST METHODS, LOAD FRAMES AND TEST SETUP

4.1) INTRODUCTION & PURPOSE

The estimation of the adhesive bond strength was made by conducting various tests under exposure to elevated environmental conditions. Such accelerated methods were employed to study the degradation of the adhesively bonded joints. The test methods employed in the present work were lap shear test, double cantilever beam (DCB) test and wedge crack (WC) test. Lap shear tests were done to measure the ultimate shear strength of the composite-adhesive bonds. DCB tests were done to determine the critical strain energy release rate, which is an estimate of material resistance to fracture. The double cantilever beam (DCB) tests can be important for bonded joints as they were indicators of the quality of the bond. Wedge crack tests were done to measure the crack growth under constant load conditions. It gives an estimate of long-term durability of the composite adhesive bond. In the following section, each test method is explained in detail elaborating on specimen geometry, test method and data reduction.

4.2) TEST METHODS

4.2.1) DOUBLE CANTILEVER BEAM (DCB) TEST

Double cantilever beam tests were quantitative tests done to determine critical strain energy release rates (G_{IC}'s). This test was done under two parts which were the peel ply study and abrasive techniques study where, the effects of peel ply and the effect of various abrasive techniques on the adhesive bonds were studied respectively.

4.2.1a) Specimen Geometry

The typical DCB specimen geometry is shown in the Fig. 4.1. The specimen was 13
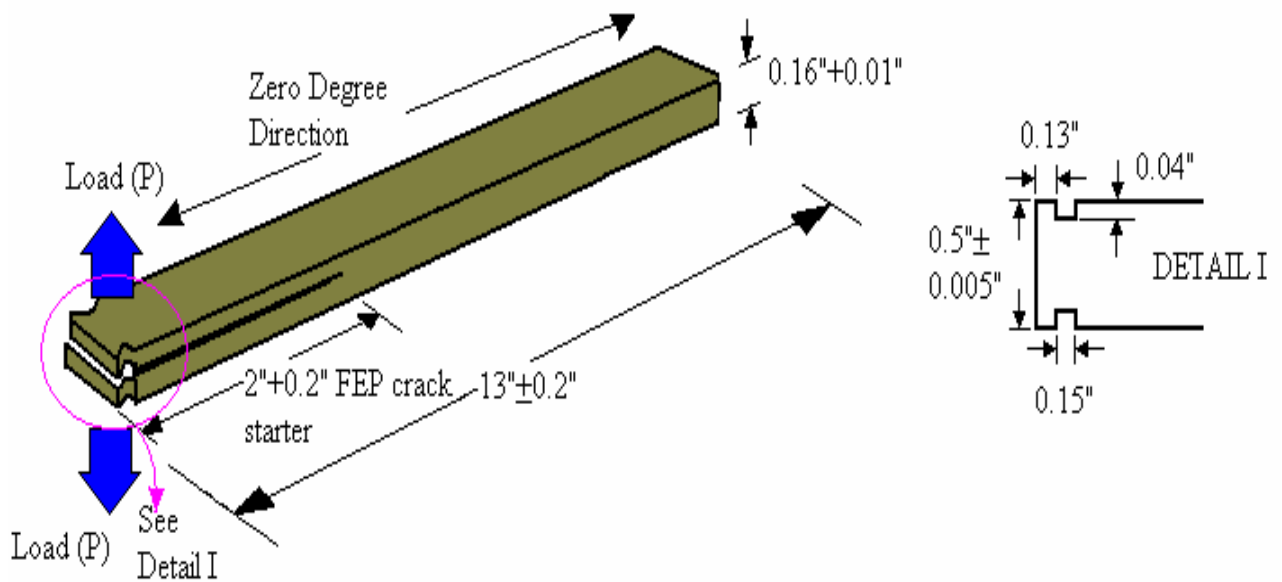
Fig. 4.1) Double Cantilever Beam (DCB) specimen geometry

inches in length, 0.5 inches in width and 0.16 inches thick. Each DCB adherend was composed of 10 [0°] composite plies. Each specimen had a crack starter of 2 inches in length which made it a double cantilever beam as can be seen from the Fig. 4.1. A triangular grip was used to pull each of the cantilever beams of the specimen. The dimensional details of the triangular grips are shown in the Fig. 4.2.

4.2.1b) <u>Testing Method</u>

In a Double Cantilever Beam (DCB) test, a DCB specimen was first subjected to a pre-crack. The pre-crack was manually made using a wrench in between the top and bottom end of DCB specimen. The wrench was slid down until the specimen cracked. The crack usually ranged from 2-3 inches measured from the starting point of the specimen. The crack tip of the specimen was marked using an optical microscope. This DCB sample

NOTE: All dimensions in inches
All Tolerances ± 0.005
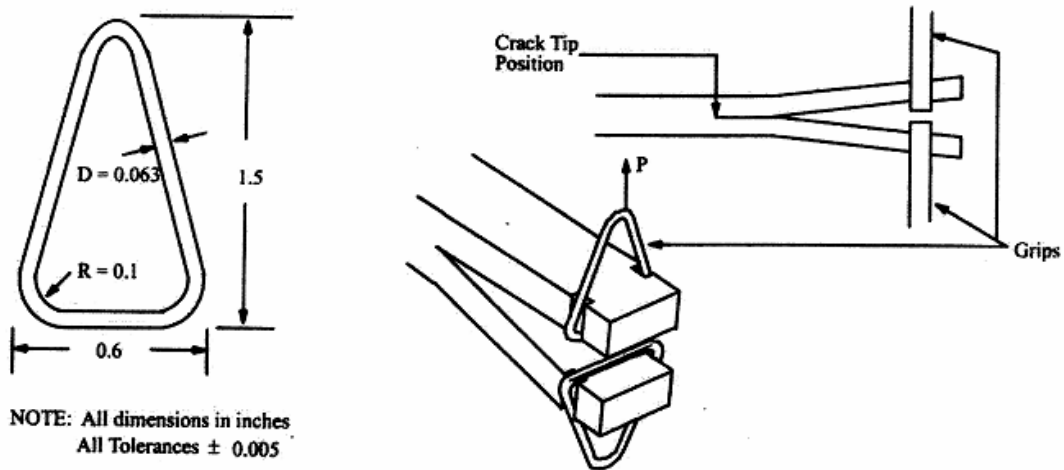
Fig. 4.2) Double Cantilever Beam (DCB) Test Fixtures (also shown triangular grips)

[21]

with a pre-crack was placed in the MTS machine using triangular grips as shown in Fig. 4.2. The specimen was pulled apart at a constant displacement rate of 1 inch/minute to produce a load deflection curve. The specimen was loaded until the crack was 5 inches past the crack tip and then unloaded thereafter to obtain the actual area under the load deflection curve. Fig 4.3 shows a typical load-displacement curve of a DCB specimen.
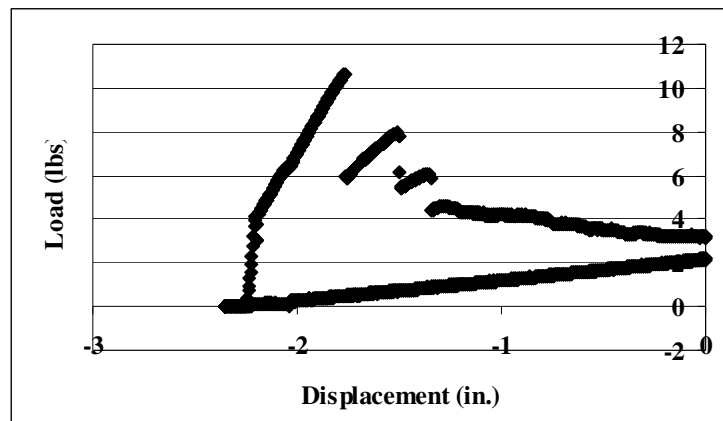
Fig 4.3) A typical load-displacement curve for a DCB specimen

The final crack tip position was marked using a 10X magnifying glass or under an optical microscope. This test was conducted at room temperature.

4.2.1c) Data Reduction

There are several methods to calculate critical strain energy release rates from the load-displacement and crack length data. The method that was followed in this work was the area method. The test machine recorded the load and crack opening displacement while the initial and final crack tip positions were marked. The area method is based upon a change in the DCB sample's compliance resulting from a change in the crack length found as

$$C = \frac{d}{P} \tag{1}$$

Where, C is the compliance, d is the deflection at load point, and P is the applied load. Therefore, the strain energy lost due to crack extension for a linear elastic body is the area between the loading and unloading curves on a load-displacement graph. Thus, the critical strain energy release rate, G_{1C} was obtained as

$$G_I = \left(\frac{dU}{b} \right) da \tag{2}$$

$$G_{IC} = \frac{E}{(\Delta a)b} \tag{3}$$

where, U is the total strain energy stored in the test specimen, E is the area between the loading and unloading curves on a load-displacement graph, b, is the specimen width and Δa is the crack length change from initial crack tip to the final crack tip.

4.2.2) <u>LAP SHEAR TEST</u>

Lap shear tests were done to determine lap shear strengths of the adhesive bonds. These tests were done in two sections namely adherend moisture sensitivity study and peel ply study.

4.2.2a) <u>Specimen Geometry</u>

The specimen geometry specification of a thick wide area lap shear specimen (TWLS) was provided by test standards of Boeing. Fig. 4.4 shows a detailed geometry of a TWLS
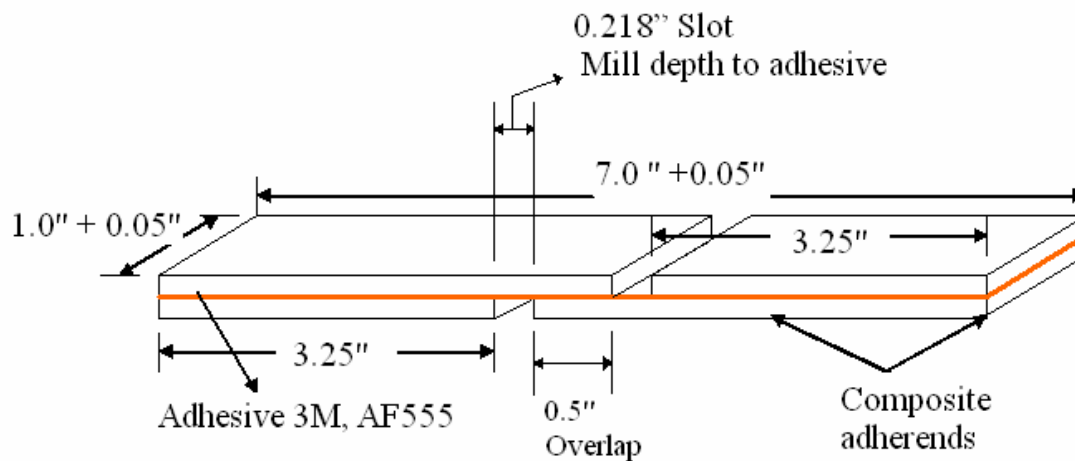
Fig.4.4) Thick wide area lap Shear (TWLS) Specimen geometry

the specimen with a depth until the center of the adhesive. It had an overlap of 0.5 inch in specimen with dimensions. The specimen was 7 inches long, 1 inch wide and 0.3 inches thick. The specimen had two notches, on either side, of 0.218 inches along the length of length and 1 inch in width. For shear strength calculations, nominal bond area was 0.5 square inches.

4.2.2b) <u>Testing Method</u>

The lap shear test specimen was measured at three places to record adhesive bondline thickness, width of the specimen and adhesive overlap length before conducting the test.

The specimen was then placed in the grips of an MTS test machine. A thermocouple was attached to the bond overlap area by clipping the thermocouple onto the specimen. Once the specimen reached $160° ± 5°$ F, it was held at that temperature for 2 minutes. A load rate of 0.05 inch/min was applied until failure.

4.2.2c) Data Reduction

The ultimate lap shear strength was calculated for each specimen where the specimens are exposed to water maintained at 140 °F for desired amounts of time. The average of all the specimens was recorded. The maximum load data for each specimen was obtained from the test machine. From the noted width and length of the overlap, lap strengths were obtained as,

$$L_{ST} = \frac{P}{WL}$$

(4)

where, L_{ST} is the ultimate lap shear strength, P is the maximum load, W is the width of the specimen and L is the length of the bond overlap.

4.2.3) <u>WEDGE CRACK TEST</u>

In wedge crack tests, crack growth of the bonds was compared to determine the long term durability of the adhesive bonds. These tests were done under peel ply study. Wedge crack specimens were made from three different peel plies which were polyester, nylon and SRB (siloxane coated polyester). The specimens were exposed in an environmental water bath maintained at 140 °F to measure the crack growth.

4.2.3a) Specimen Geometry

The wedge crack specimen was 6 inches in length and 1 inch in width. The specimen had a separation film of 2 inches in length. The separation film was used for creating a 2 inch

crack initiation length for the wedge crack specimen. The wedge was of 0.125 inches thick and 1 inch long along the width of the specimen. A detailed view of the wedge crack specimen and the dimensions of the wedge are shown in the Fig 4.5.

4.2.3b) <u>Testing Method</u>

This test method is a composite version of the ASTM D3762 which is the standard test method for adhesive-bonded surface durability of aluminum (wedge test). In this test instead of aluminum adherends, composite adherends were used. In the wedge crack test, a wedge was driven into the specimen thereby creating a tensile stress in the region of the resulting crack tip. The wedge stimulated the crack growth. The stressed specimen was then exposed to water maintained at a temperature of 140°F. The resulting crack growth with time was recorded.
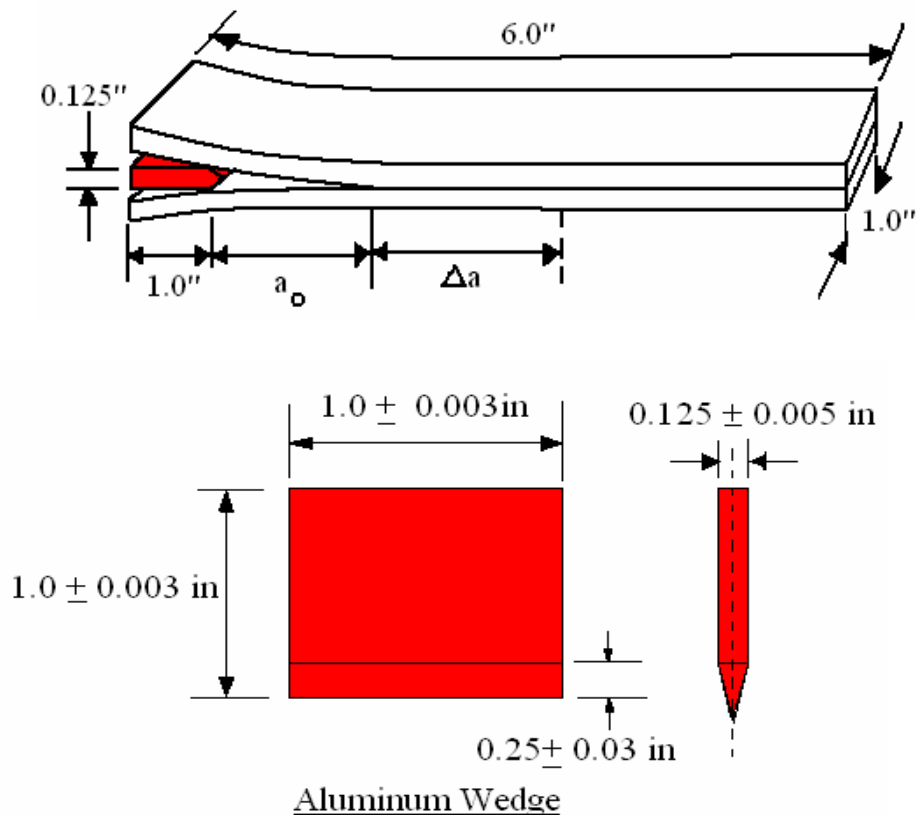
Fig. 4.5) Wedge crack (WC) Specimen geometry

4.2.3c) <u>Data Reduction</u>

After the wedge was inserted the original crack length, a_o was noted. The crack growth, Δa, was noted at the end of various time intervals such as 1, 4, 8, 24, hours; 7, 30 days. Crack length was measured from the edges of the wedge crack specimens which were painted white for visible crack growth as shown in Fig 4.6. Also, the adhesive-joint failure mode was reported at the conclusion of the test.

Fig 4.6) Crack measurement of an edge painted WC specimen

4.2.4) <u>COMPRESSION INTERLAMINAR SHEAR (CILS) TEST</u>

This test is done to study the effects of moisture on the interlaminar strength of the adherend. In hot/wet conditions the most significant loss is usually observed in the compressive interlaminar shear strength of a composite material. The interlaminar shear strength is due to mechanical as well as chemical interaction between the lamina. Residual compressive stresses are developed at hot/wet environments due to differential expansions or contractions of the constituent laminae. This will reduce the interlaminar shear strength. Therefore, CILS specimens were soaked at 160 ºF in water to determine the effects of moisture on the compression interlaminar shear strengths at various moisture levels.

4.2.4a) <u>Specimen Geometry</u>

All the specimens were processed and machined at Boeing, Seattle (WA). Each specimen was 3.18 inches in length and 0.5 inches in width as shown in the Fig. 4.7. A cut 'C' of 0.0925 ± 0.01 inches deep was penetrated on both sides of the adherend, as shown in the figure, so that the depth was half the thickness of the specimen. The overlap of the lap was around 0.2675 ± 0.01 inches. The inner edges of the cut were machined to have a minimum radius of 0.005 inches. Each specimen was processed with 24 [0°] plies of composite BMS 8 276 where the fiber direction is parallel to the length of the specimen.

4.2.4b) <u>Testing Method</u>

The CILS test specimens were taken out of the environmental exposure bath and tested in the load frame within a time span of one hour. Each specimen was measured at three places to record specimen width and specimen overlap length before conducting the test. The specimen was then placed in the grips of an MTS test machine. A thermocouple was attached to the bond overlap area of the specimen. Once the specimen reached $180° \pm 5°$ F, it was held at that temperature for 2 minutes and then a compressive load at a rate of 0.05 inch/min was applied until failure. The ultimate load at failure was noted for each specimen.

4.2.4c) <u>Data Reduction</u>

The compression interlaminar shear strengths for each of the specimens were calculated and the average value was obtained. The ultimate load at which a CILS specimen failed was obtained from the test machine. Then, from the noted specimen width and specimen

overlap of length, the compression interlaminar shear strength for each specimen was calculated as,

$$\tau_{COMP} = \frac{P}{bl} \qquad (5)$$

where, P is the ultimate load (lbs), b is the specimen width (inches) and l is the specimen overlap length (inches).
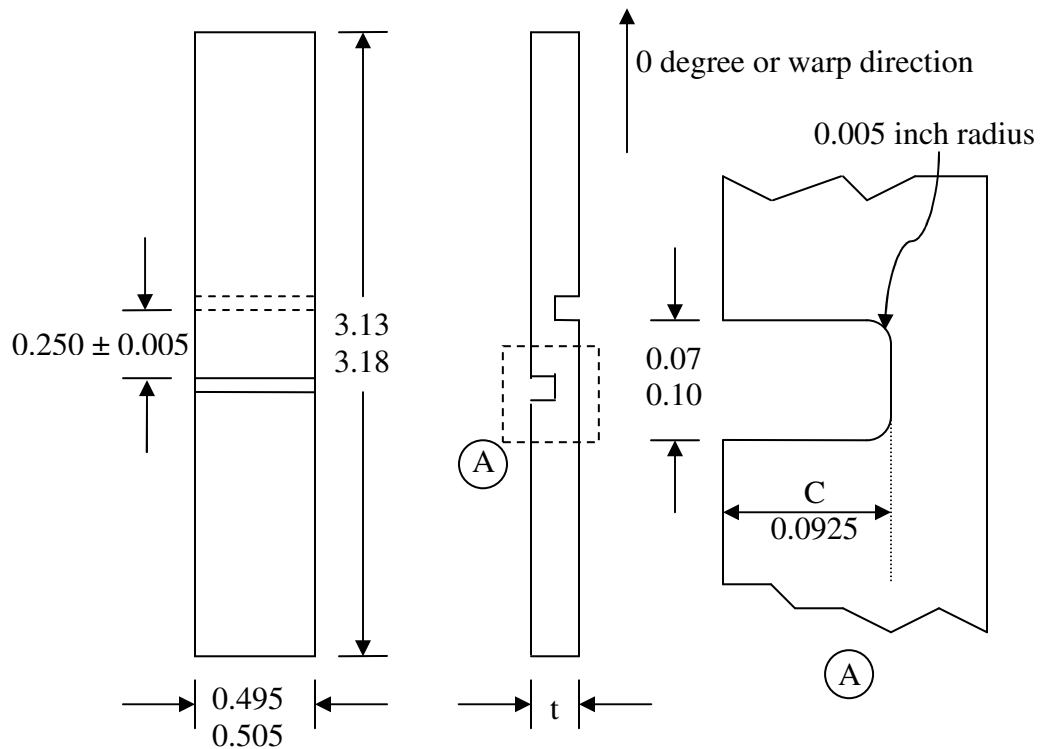
Note: All dimensions are in inches.

Fig 4.7) Compression interlaminar shear (CILS) specimen geometry

4.2.5) IN-PLANE SHEAR (IPS) TEST

This test was done to study the effects of moisture on the in-plane shear (IPS) strength of the composite. The immediate effects of moisture on the composite are usually observed in the matrix-dominated properties. One such property is the in-plane shear strength of

the composite. Therefore, in-plane shear specimens were soaked in water at 160 ºF to study the effects of moisture on their strengths at various moisture levels. The [±45°] $_S$ laminate tensile test method is described in ASTM D 3518/D3518M-91.

4.2.5a) <u>Specimen Geometry</u>

All the specimens were processed and machined at Boeing, Seattle (WA). Each IPS specimen was 0.5 inches in width and 9.0 inches in length. The geometry of an IPS specimen is shown in the Fig. 4.8. Each specimen was made of 4 composite plies where the orientation was [±45°] $_S$. The thickness of the specimen was approximately 0.03 ± 0.001 inches. The ends of each specimen were tabbed to a length of 2 inches so that the specimen can be firmly held in the load fixture while performing the test.

4.2.5b) <u>Testing Method</u>

This test was done at room temperature. Before conducting the test, each IPS test specimen was measured at three places to record specimen thickness and width. The specimen was then placed in the grips of an MTS test machine. An extensometer was gripped on to the specimen to measure the longitudinal and transverse strains. A tensile load at a rate of 0.5 ± 0.01 inch/min was applied until failure.

4.2.5c) <u>Data Reduction</u>

The ultimate in-plane shear strength and modulus were determined in accordance with Boeing BSS 7320 standards. The stress tensor components with respect to principal material axes can be expressed in terms of loading reference frame according to the transformation rule given by

$$\begin{Bmatrix} \sigma_1 \\ \sigma_2 \\ \tau_{12} \end{Bmatrix} = \begin{bmatrix} c^2 & s^2 & 2cs \\ s^2 & c^2 & -2cs \\ -cs & cs & c^2 - s^2 \end{bmatrix} \begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix} \tag{6}$$

where $\left(\sigma_1, \sigma_2, \tau_{12}\right)$ and $\left(\sigma_x, \sigma_y, \tau_{xy}\right)$ represent the stress components with respect to the material principal axes and loading reference frame respectively. For the case of a uni-axial tension loading and laminates oriented along ± 45°, the shear stress along the principal material axes is given by

$$\begin{Bmatrix} \sigma_1 \\ \sigma_2 \\ \tau_{12} \end{Bmatrix} = \begin{bmatrix} \frac{1}{2} & \frac{1}{2} & \pm 1 \\ \frac{1}{2} & \frac{1}{2} & \pm 1 \\ \pm \frac{1}{2} & \pm \frac{1}{2} & 0 \end{bmatrix} \begin{Bmatrix} \sigma_x \\ 0 \\ 0 \end{Bmatrix} \tag{7}$$

$$\tau_{12} = \pm \frac{\sigma_x}{2} \tag{8}$$

In the similar manner, the shear strain with respect to principal material axes is related to engineering strains with respect to loading reference frame as

$$\gamma_{12} = \pm \left(\varepsilon_x - \varepsilon_y \right) \tag{9}$$

Using the above relations the in-plane shear strength and modulus can be derived as

$$\tau_{12} = \frac{P}{2btN} \tag{10}$$

$$G_{12} = \frac{E_x}{2(1 + \upsilon)} \tag{11}$$

41

0.2 Min.

0.03 ± 0.001

9.0 ± 0.3

5 ± 0.01

Bonded tabs

4 plies, [±45] $_S$ Orientation

Taper achieved by (a) staggering each ply by 0.10 inch or (b) machining to a 6 ± 1 degree angle
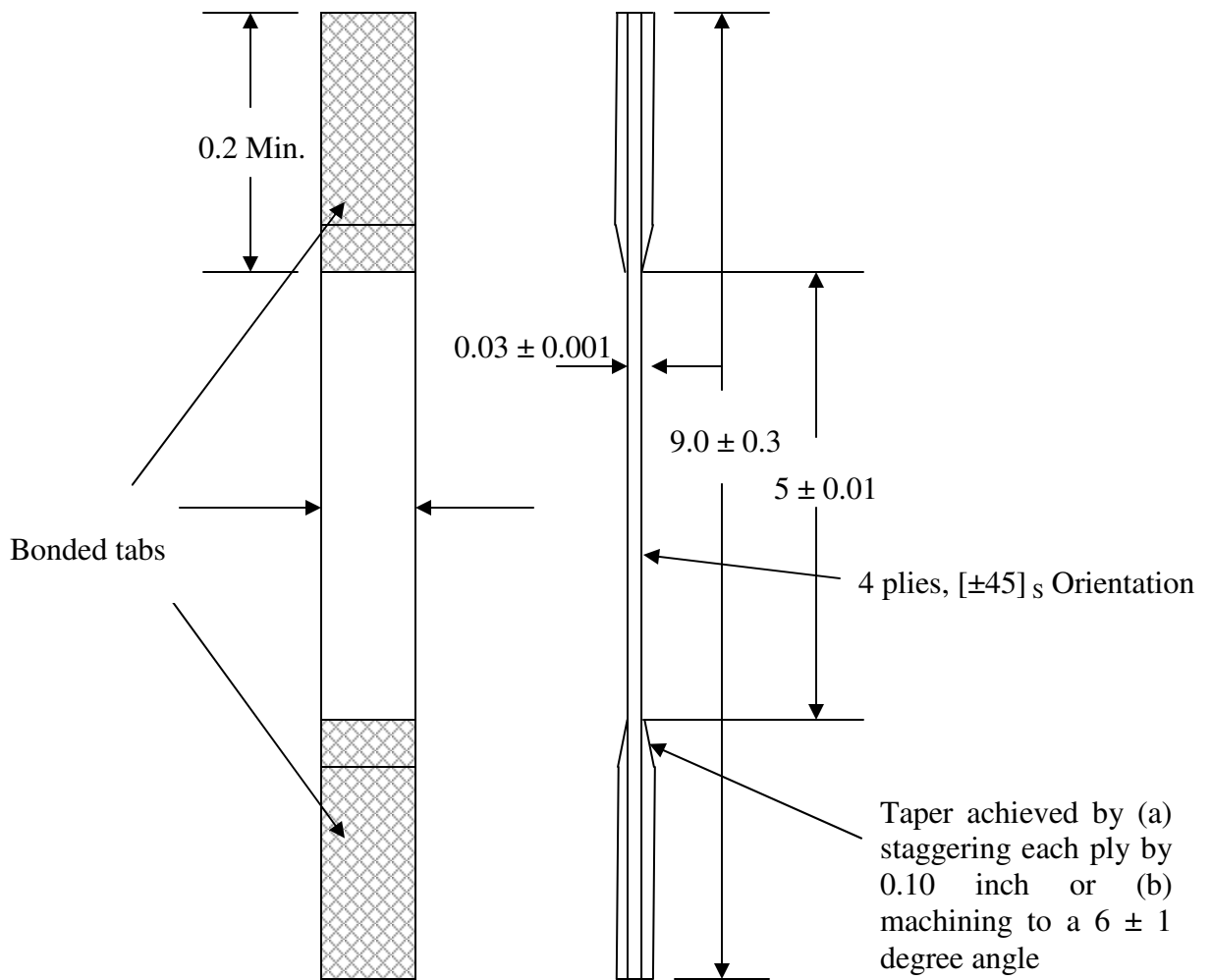
Note: All dimensions are in inches.

Fig 4.8) In-plane shear specimen (IPS) geometry

where, τ_{12} is the ultimate shear strength (psi), G_{12} is the in-plane shear modulus of elasticity (psi), P is the ultimate load (lbs), b is the specimen width (inches), t is the standard thickness per ply (inches), N is the number of plies, E_x is the axial secant modulus from a line drawn through the origin and to a point corresponding to 0.004 in/in strain for the axial loading deflection curve, υ is the Poisson ratio. The poisson's ratio is the absolute value of transverse strain/axial strain with the transverse strain measured at a load corresponding to 0.004 in/in axial strain.

4.3) LOAD FRAMES AND TEST SETUP
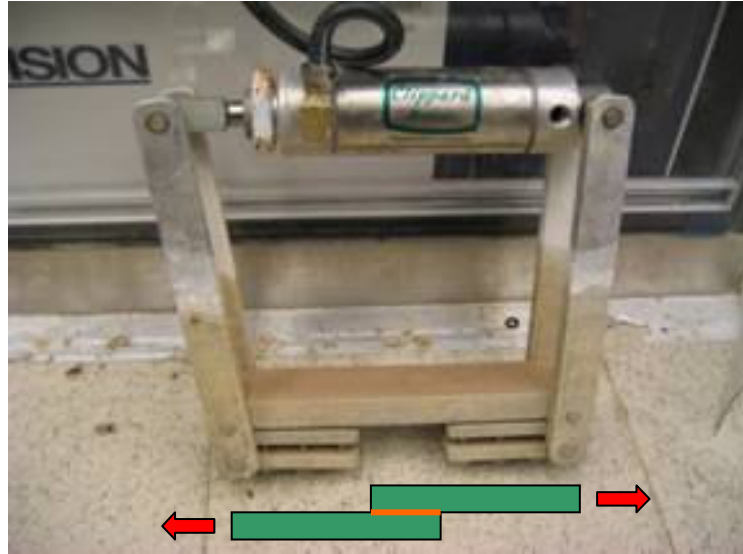
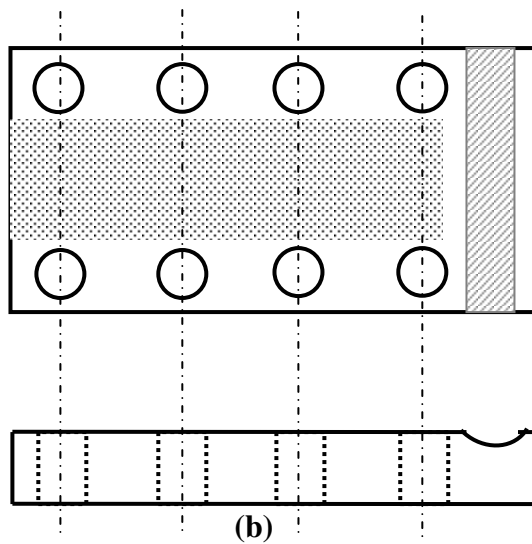### 4.3.1) CREEP LOAD FRAMES FOR LAP SHEAR SPECIMENS

The creep load frame for the lap shear test specimens had one cylinder-piston arrangement. A double acting cylinder of 2 inches bore size and 3 inches stroke length was used in these tests. The cylinder-piston assembly was supported by two movable rods at each end of the cylinder which act as moment arms. These moment arms were each 7.5 to 1 inches in length. Each end of these arms was connected to two metal grips designed to hold the specimen. The Fig. 4.9 (b) shows a schematic picture of these grips. Each grip has eight screws to hold the specimen tight. The screws were placed so that there was enough clearance with the fixture. The grips were designed to have a groove with enough clearance to fit the pin connecting to the moment arm. A torque of 90 lb-inches was applied on the screws to hold the specimen in between the metal plates. A picture of the creep load frame for the lap shear test specimens is shown in the Fig. 4.9(a). A load range of 700-1700 lbs was applied from the cylinders.

4.3.2) FATIGUE LOAD FRAMES FOR DCB SPECIMENS

The fatigue load frame for DCB specimens consisted of five cylinder-piston assemblies. A cylinder of 7/16 inches bore size, maximum stroke of 6 inches and a 250 psi maximum pressure was used in these tests. The head of the piston-cylinder assembly was held in position with the help of a mechanical lever which in turn was fixed to the load frame. A picture of a fatigue load frame for the DCB specimen is shown in the Fig. 4.10

(a)

(b)
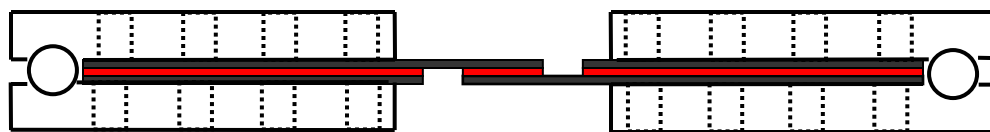
(c)

Fig 4.9) (a) Creep load frame for lap shear specimens (b) Schematic layout of the grip plates (c) Specimen gripped between the metal plates
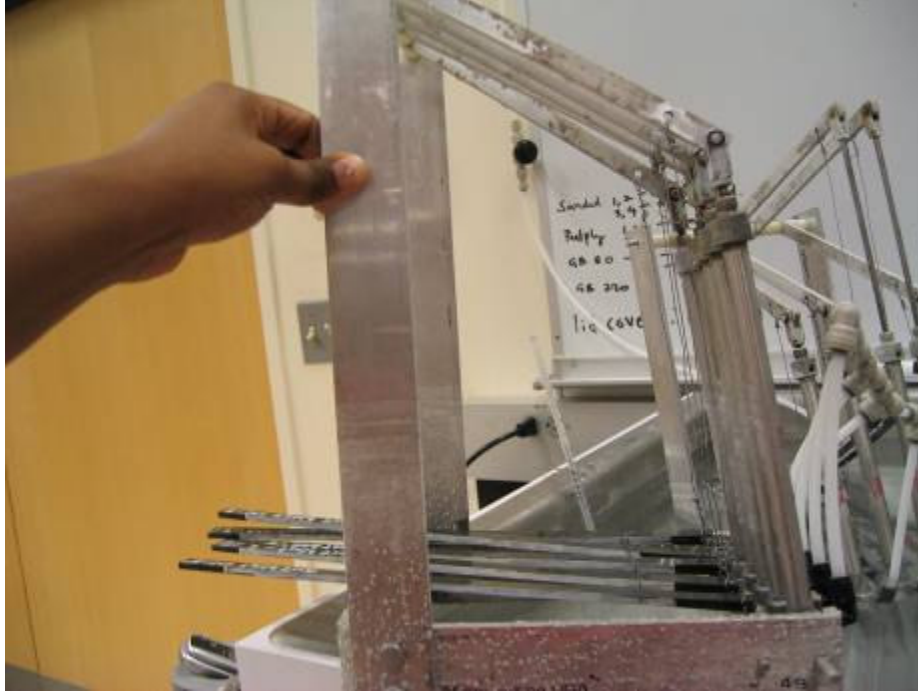
Fig 4.10) Fatigue load frame for a DCB specimen

4.3.3) CREEP AND FATIGUE TEST SETUP DETAILS

Creep tests were done on the lap shear specimens under the adherend moisture sensitivity study and peel ply study while fatigue tests were done on the DCB specimens under abrasive techniques study. The creep and fatigue load setup had a similar arrangement as shown in the schematic layout in Fig 4.11. Load frames were placed in an exposure bath which was a container that can be set at desired temperatures. In creep and fatigue tests, the specimens were loaded while they were immersed in a water bath maintained at 140ºF. Each load frame was connected to a pneumatic air line. The applied pressure on the cylinder was controlled by a pressure regulator. A load frame with a load cell was connected in parallel to the regulator which monitored the load applied on the cylinders. The load applied was dependent on the specimen type. In a lap shear creep test, a lap

shear specimen was mounted in the grips of the load fixture as shown in the Fig. 4.9(c). The air pressure applied a constant tensile load on a lap shear specimen. However, in a DCB creep and fatigue test the upper end of the specimen was wired to the mechanical lever while the lower beam of the specimen was fixed to the frame. The frequency of the load applied in the fatigue test was 0.5 hertz.

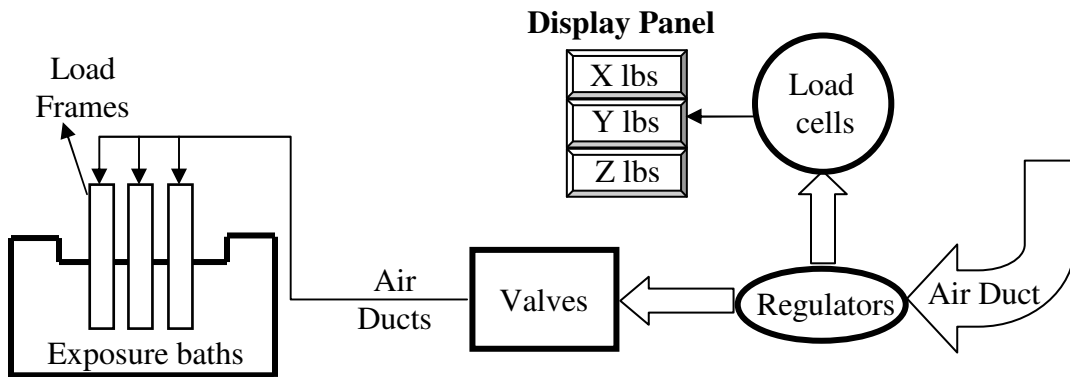**Fig. 4.11) Schematic diagram of test setups**

CHAPTER 5

STUDY DETAILS AND RESULTS

The effects of three different parameters (adherend moisture content, peel ply and abrasive treatments) on the strength of the adhesive bond were studied. Test methods were employed to accelerate test and loading conditions. Double cantilever beam, lap shear and wedge crack tests were performed while the dried specimens were immersed in water at elevated temperatures. The next subsection gives details about the material specifications followed by specimen preparation and then results and discussion.

5.1) MATERIAL SPECIFICATIONS

The composite used to make the specimens was Form 3 BMS 8-276-lamina (Toray T800/3900-2B). The matrix of the composite is an epoxy resin while the fiber is carbon fiber. Two classes of this composite namely, classic (C) and low cost (L) materials were used. C type composite was a higher composite version with almost the same fiber and resin properties as the L type composite. Polyester (Precision Fabrics 60001), nylon (Precision Fabrics 52006) and siloxane coated polyester (Super Release Blue or SRB) were the three different peel plies used for surface preparation which were fine, medium and coarse in texture respectively. A FEP (Fluorinated Ethylene Propylene) separator film was used as a crack initiator in the DCB and wedge crack specimens.

Adhesive, 3M AF555

The epoxy adhesive used for bonding was AF555 (3M). AF555 has been formulated to have high moisture resistance during and after bonding. It is also under consideration for large scale commercial use, for which an understanding of its durability is needed. It is a
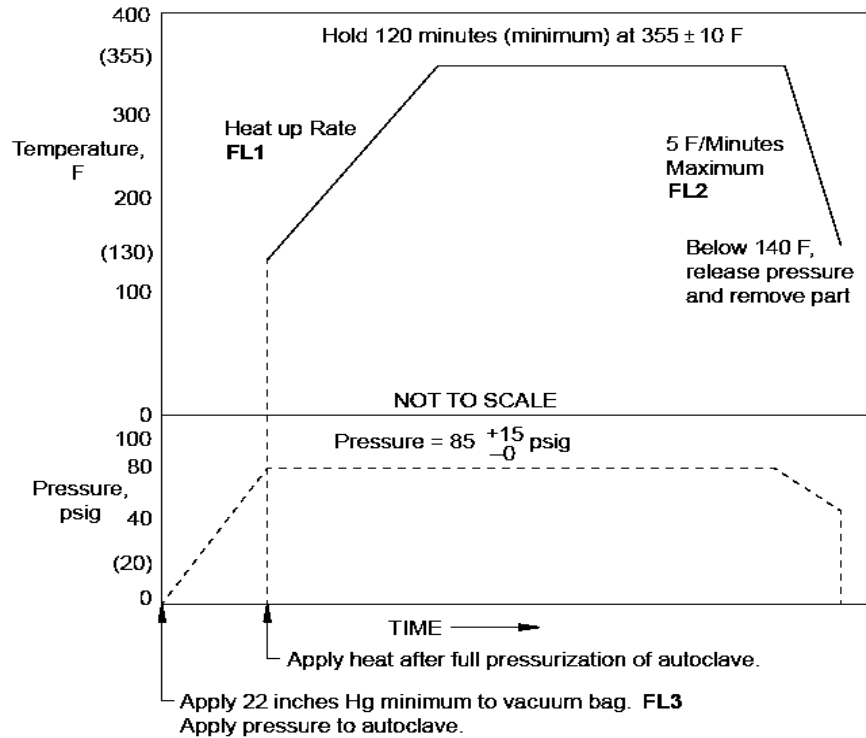
film adhesive with 0.01 ± 0.005 inch thickness and has a cure temperature of 350 ºF (similar to that of the curing temperature of composite)

5.2) SPECIMEN PREPARAT ION

The DCB and lap shear specimens were cured and bonded at Boeing, Seattle, where as wedge crack specimens were made at Washington State University, Pullman. A typical specimen was made up of two adherends bonded with an adhesive. The DCB and wedge crack adherends consisted of 10 [0°] plies of Form 3 BMS 8-276 pre-pregs of appropriate dimensions where as the thick wide area lap shear (TWLS) specimens were made up of 20 [0°] plies. Adherends were vacuum bagged with mold release on the tool side and a peel ply on top surface. This setup was cured to create panels with different surface properties on each peel ply side (bonded side) depending on the peel ply used. The cure cycle used herein was provided by Boeing [22] and is shown by the graph in the Fig 5.1.

Vacuum was applied (a minimum of 22 Hg) and autoclave pressure was raised to 85 psi per minute. The temperature of the autoclave was raised at a rate of 3 ± 2 ºF (1.7 ± 1.1 ºC) per minute up to 355+ ºF (180º ± 5 ºC) for 120 to 180 minutes. Cooling was done up to 140 ºF (60 ºC) under pressure at a maximum cool down rate of 5 ºF (3 ºC) per minute. After the panels were cured, the peel ply was removed. The time between removal of peel ply and bonding was 10 to 20 minutes. The peel ply surfaces were joined together with the adhesive, 3M AF555. For DCB or wedge crack specimens, a 2 inch FEP separator film was used as a crack initiator. This whole setup was cured using the above cure cycle and machined to obtain final specimens of desired dimensions. The lap shear test specimens underwent additional machining till the center of the adhesive layer on both

sides to create notches. For DCB and wedge crack specimens, the sides of the samples were painted white before testing to have clear vision of the crack tip as shown in Fig 5.2

Temperature, F

Hold 120 minutes (minimum) at 355 ± 10 F

Heat up Rate
FL1

5 F/Minutes
Maximum
FL2

Below 140 F, release pressure and remove part

NOT TO SCALE

Pressure = 85 $^{+15}_{-0}$ psig

Pressure, psig

TIME ———▶

Apply heat after full pressurization of autoclave.

Apply 22 inches Hg minimum to vacuum bag. FL3
Apply pressure to autoclave.

FL 1   Heat–up at 5.0 ± 0.5 F/minute based on autoclave air temperature except as noted.

FL 2   Cool under pressure until the part temperature reaches 140 F or below. Natural pressure drop down to an autoclave pressure of 57 psi due to cool down is allowed in the autoclave.

FL 3   Maintain full vacuum throughout the cure cycle.

Fig 5.1) Cure Cycle for fabrication of Composite specimens [21]

49

Fig 5.2) A Sample edge painted DCB specimen

5.3) <u>RESULTS AND DISCUSSION</u>

The results are shown in three parts namely, adherend moisture sensitivity study, peel ply study and abrasive techniques study. The results are presented in detail in the following sections along with discussions.

5.3.1) <u>ADHEREND MOISTURE SENSITIVITY STUDY</u>

The adherend moisture sensitivity study was done to study the moisture resistance of AF555 adhesive and to get a qualitative as well as quantitative estimate of the dependence of adhesive bond strength on the adherend moisture content. The lap shear specimens used under this study can be broadly classified into two categories, dry co-bonded specimens (D) and wet co-bonded specimens (W). D specimens were formed by bonding dry adherends whereas W were formed by bonding adherends soaked to 1% moisture gain before bonding, with an adhesive. All the adherends in the formation of specimens were cured against polyester peel ply. In this study, the specimens were pre-conditioned in water at 140 ºF for 1000 hours before testing.

Two kinds of tests were done on these pre-conditioned specimens which were base-line and creep load tests. An average ultimate shear strength of 4.4 ksi for D and 4.6 ksi for W specimens was obtained from the base-line tests. In the creep test, the pre-conditioned specimens were subjected to creep loads of 2 ksi, 3 ksi and 4 ksi for 1000 hrs while immersed in water maintained at 140 ℉. Table 5.1 shows the test matrix for this test which shows the number of specimens tested under each load conditions. After the creep test was done, these specimens were pulled to failure in tension.

The Fig. 5.3 below shows lap shear strengths of the D as well as W specimens subjected to different creep loads. As it can be observed from the graph, there was little difference

Table 5.1 Test matrix of the adherend moisture sensitivity study

Specimen type	Creep Stress			
	0 ksi	2 ksi	3 ksi	4 ksi
D	3	3	3	3
W	3	-	3	3

in strengths between D and W specimens. It meant that AF555 was insensitive to moisture during bonding. Residual shear strength shown in Fig. 5.3 decreased with increasing creep load. Previous studies have shown that there was a significant decrease (22.2-41.1%) in the lap shear strengths of adhesively bonded composite lap shear specimens while exposed to ninety days of accelerated conditioning in sea water [22]. Also, others have found similar observations where the strengths of composite-adhesive

51

bonds decreased due to the effects of moisture, temperatures and creep load [23, 24, 25 and 26]. The benevolent moisture resistance of AF555 apparently does not extend to its creep response.

The failed specimens were examined to obtain a qualitative estimate of the failure mode. Fig. 5.4 shows the SEM pictures of the failed surfaces of the D specimen which was under 4 ksi creep load. It was observed that fibers were exposed with no visible trace of peel ply texture, which implied a complete adherend failure. Fig. 5.5 is a typical SEM picture of failed surfaces of W specimen which was under a creep load of 4 ksi. A similar adherend failure mode was also observed in these specimens. From these observations, we may conclude that AF555 had low sensitivity to moisture both prior to and after
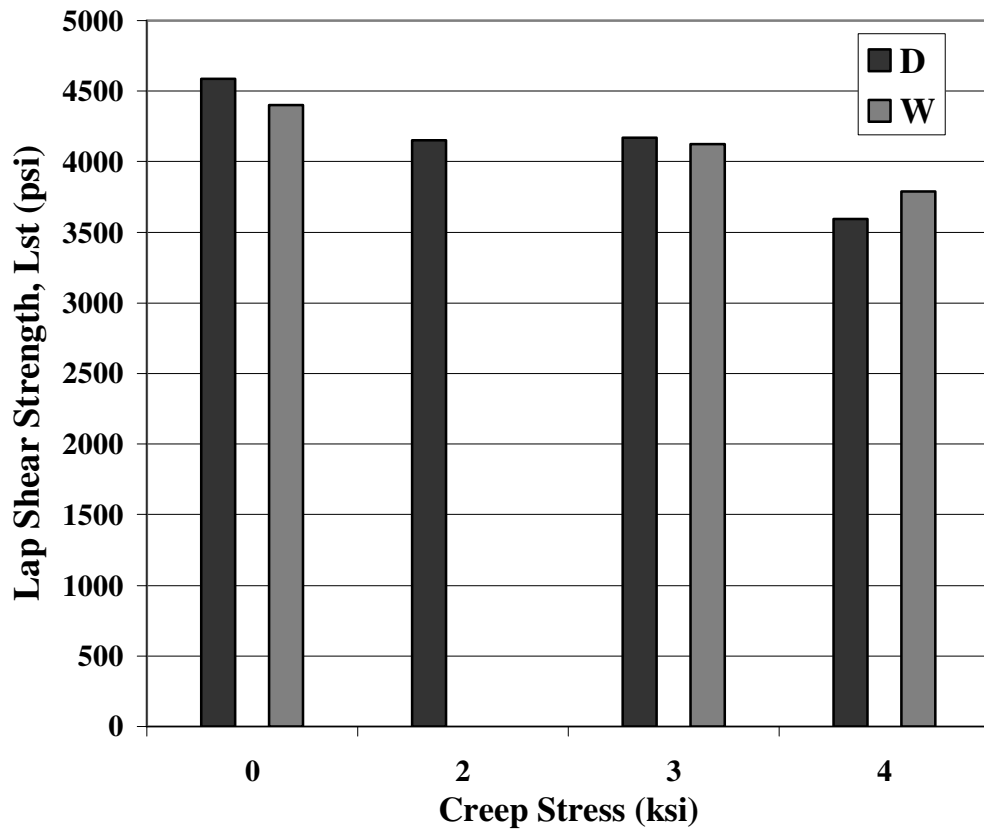

Fig. 5.3) Dependence of residual lap shear strength under increasing creep loads on D and W specimens

bonding. Usually, some epoxy adhesives are sensitive to moisture. Moisture can penetrate into the resin and weaken the bond by hydrolysis or plasticization. Plasticization will result in reduction of the glass transition temperatures of the epoxies. Exposure to moisture also decreases the mechanical properties of epoxy resins and can result in detrimental effects on the intermolecular structures [27]. It has also been observed previously that pre-cure exposure to high humidity of a structural film form epoxy adhesive had deleterious effects on their properties of the cured adhesive [28]. And also, experimental results have shown previously that absorbed moisture in the epoxy adhesives caused reduction in their shear strengths [29].
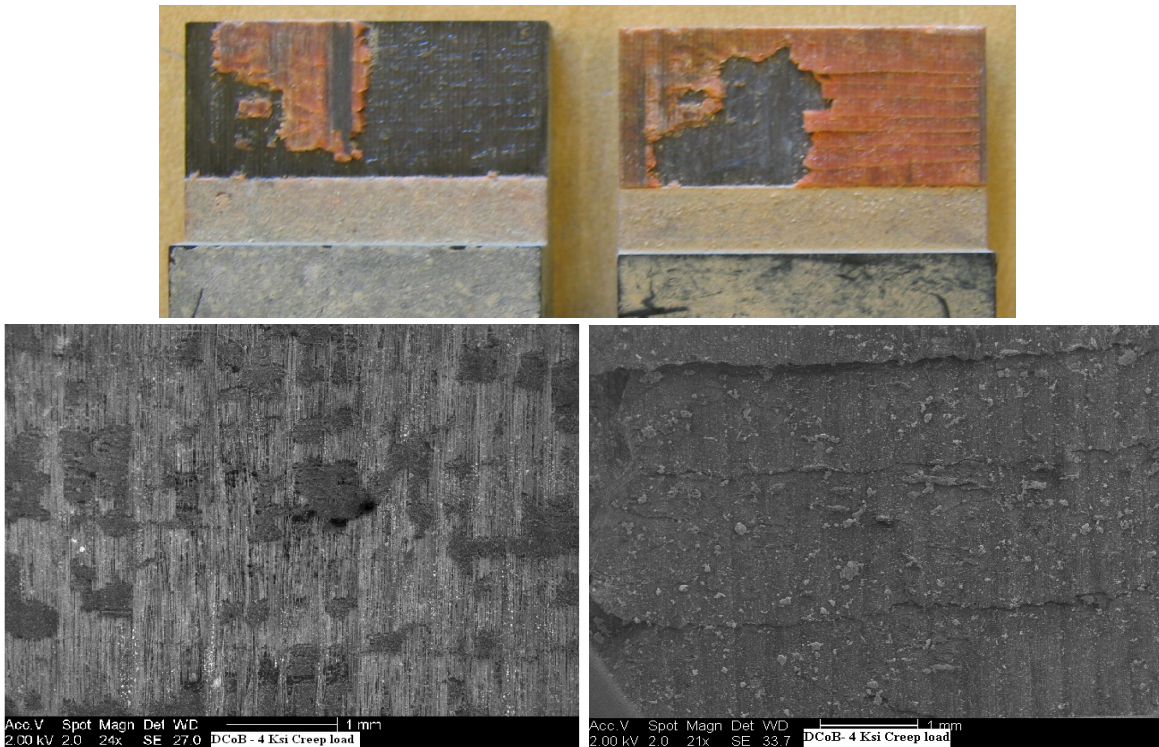

Fig 5.4) Fractured pictures of a D specimen under 4 ksi creep load (The figures belong to the same specimen. Magnification is 21X - 24X)
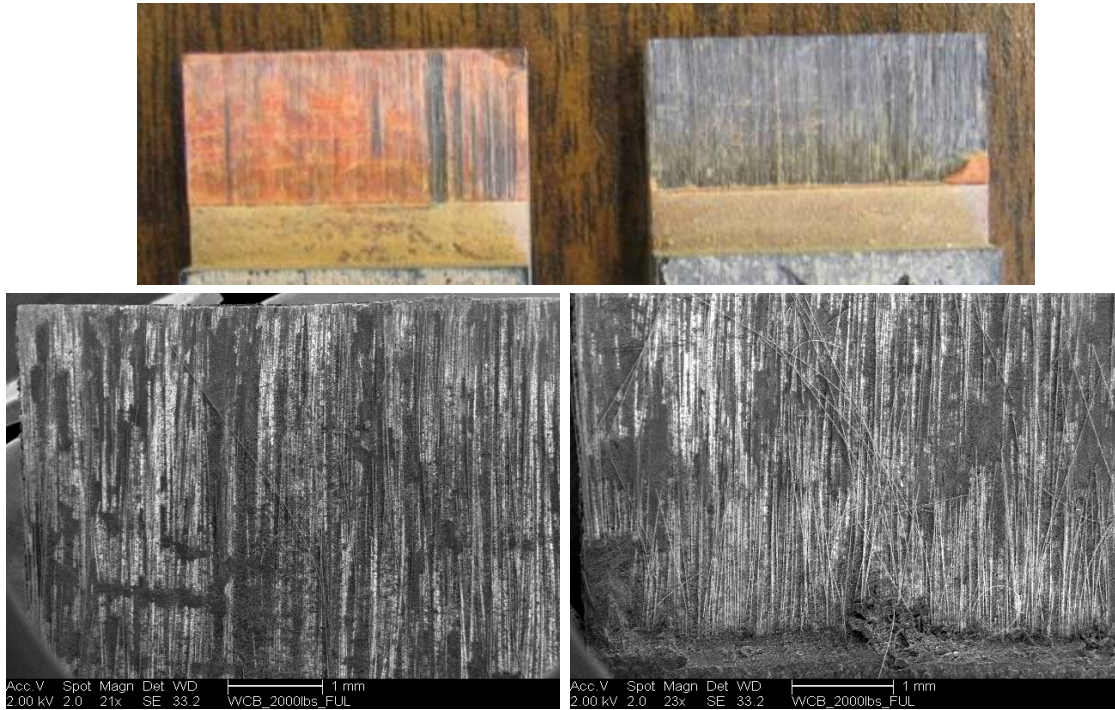
Fig 5.5) Fractured pictures of a W specimen under 4 ksi creep load (The figures belong to the same specimen. Magnification is 21X-23X)

Compression interlaminar (CILS) and In-plane shear tests (IPS)

The failure of composite substrate (adherend) was observed during the moisture soak studies. The composite version used in all through the work was a low cost (L) class of the system. The observed sensitivity of the composite to moisture motivated a study to investigate the effects of moisture on a higher class of the composite. Therefore, tests were done on both low cost and classic, which is a higher version of the composite. Compression interlaminar shear (CILS) and in-plane shear (IPS) tests were done for various moisture levels in the composite. The test specimens were immersed in a water bath maintained at 160 ºF and the subsequent mechanical tests were conducted at different moisture levels. For the CILS study, tests were conducted at dry state, six days immersion, 0.8 %, 1%, 1.2 % weight gain while for the IPS study tests were conducted at

dry state, 1% and 1.2% weight gain. The tests temperatures were 180 ºF and room temperatures for the CILS and IPS respectively.

 Fig 5.6 shows that the compression shear strengths of C and L class BMS 8 -276 composite specimens at various moisture levels. In case of L class material, there was 20 % decrease while, in C class there was 15% decrease in interlaminar shear strengths when exposed to water maintained at 160 ºF for a period of six days. It shows that with further increase in moisture concentration, there was no noticeable decrease in the shear strengths. It can be seen in the figure 5.6 that the compression shear strengths for the C and L class specimens have very little difference. And, Fig 5.7 shows the effects of moisture on the in-plane shear modulus of the C and L class composite specimens. It is evident that there is a decrease of 5% and 6% in the IPS modulus in the C and L class material respectively, when exposed to160 ºF for a period of six days. Further, for 1% moisture weight gain there was 8-10% decrease in the in-plane shear strength in the L class material. Subsequently, there was no significant decrease in the in-plane shear strengths with further increase in moisture content. But, there is no significance difference between the in-plane shear strengths of C and L class material as can be seen in the figure 5.7.

Summarizing the results, the combined effects of moisture and temperature resulted in a decrease of 15-20 % in the interlaminar shear strength and 8-10% in the in-plane shear strength. The interlaminar shear strengths and the in-plane shear strengths between C and L class material were found to be almost the same. Advisably, it is economically advantageous to choose L over C class material for manufacturing purposes as the strengths are not very different.
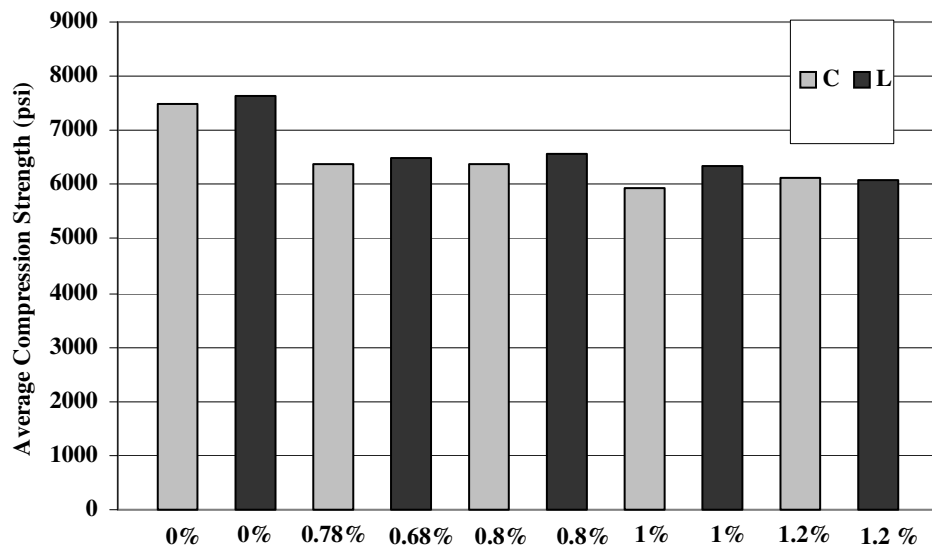
Fig. 5.6) Moisture content affects on the compression shear (CILS) strengths of C and L class composite for an exposure in water at 160 ºF. Test conducted at 180 ºF
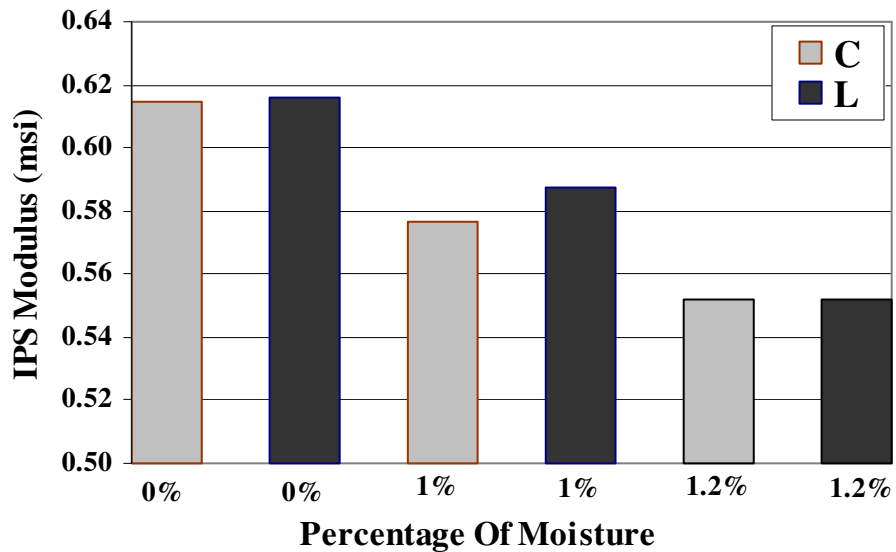

Fig. 5.7) Moisture content affects on the In-plane shear (IPS) of C and L class composite for an exposure in water at 160 ºF. Test conducted at RT.

5.3.2) PEEL PLY STUDY

A peel ply study was done to consider the effect of adherend surface texture on the adhesive bond strength of the specimen. The peel plies used on the adherend which dictate the surface texture were polyester, nylon, and SRB. Towards the objective of this study, lap shear, double cantilever beam (DCB) and wedge crack (WC) tests were done.

Lap Shear Test

In lap shear test, the specimens were pre-conditioned in water at 140 ºF for 6000 hrs, which was close to saturation, before testing. A thick wide area lap shear (TWLS) specimen was used. A comparison of percentage weight gain for thin (20 plies) and thick (40 plies) specimens was made along with corresponding fickian diffusive behavior in the Fig. 5.8. It can be inferred from the graph that the longer saturation time was due to the thicker specimens. The diffusion coefficients for the adherend and adhesive were found to be 0.00106 mm^2 /hr and 0.0044 mm^2 /hr respectively for the saturation levels of 1.25 and 3.0 for the adherend and adhesive respectively. The diffusion coefficient of the sandwich (bonded specimen) was calculated to be 0.00138 mm^2 /hr for a saturation level of 1.41. The lap specimens were subjected to base-line and creep load rupture tests. Table 5.2 shows the test matrix relevant to this study. In the base-line test, the pre-conditioned specimens were tested to failure to determine the ultimate lap shear strength. Fig. 5.9 shows the lap shear strengths obtained from base-line tests of specimens cured against polyester, nylon and SRB peel plies. It can be inferred from the graph that the lap shear strength was highest for polyester and lowest for SRB peel ply specimens. A qualitative analysis was done on the fractured surfaces to determine the failure modes. Fig. 5.10 depicts the failed surfaces of the specimens. The SRB and nylon peel ply specimens

Table 5.2) Test matrix for the lap shear tests, pre-exposure of 6k hours in 140 °F water to reach saturation.

Specimen type	No load	80% UTS
TWLS/Polyester	5	10
TWLS/Nylon	5	10
TWLS/SRB	5	10

resulted in complete adhesion failure whereas polyester specimens failed partly in adherend and partly in cohesive failure modes. In the creep load test, a creep load of 80% ultimate lap shear strength obtained from the base-line test of each surface was applied while immersed in water maintained at 140 ºF until rupture. The time taken to rupture by taken by various peel ply specimens. It can be observed from the graph that the polyester peel ply specimens took longer time to rupture followed by nylon and SRB.
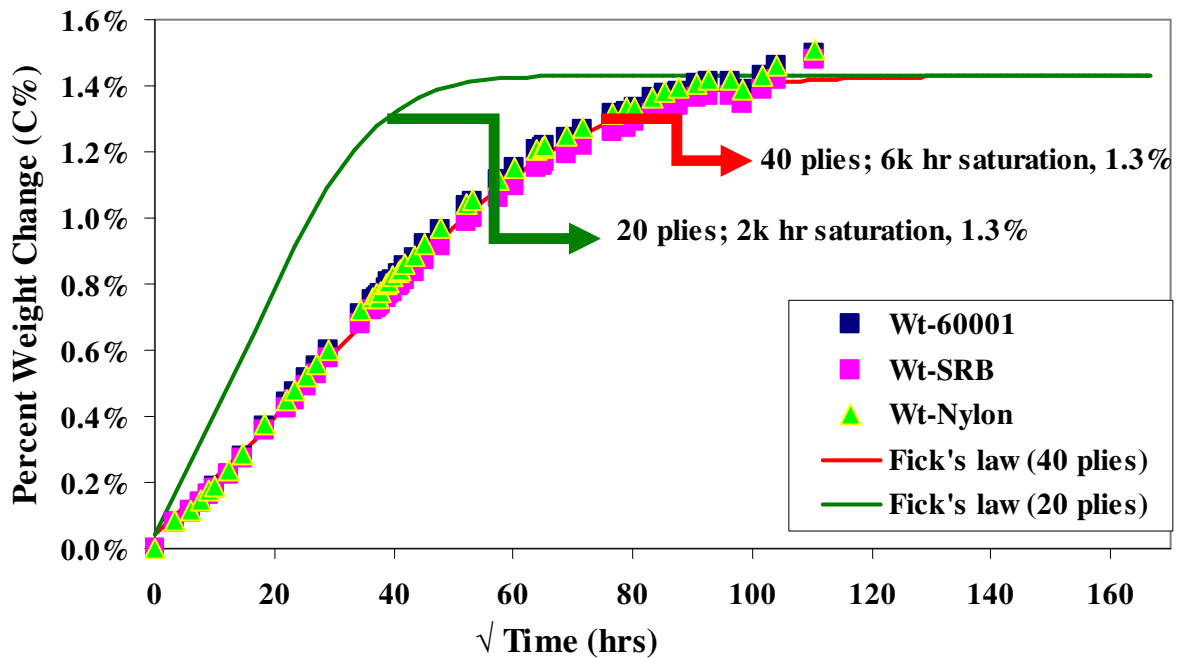

Fig 5.8) Weight gain data for BMS 8 276 Toray laminate adherend specimens of the three different peel plies
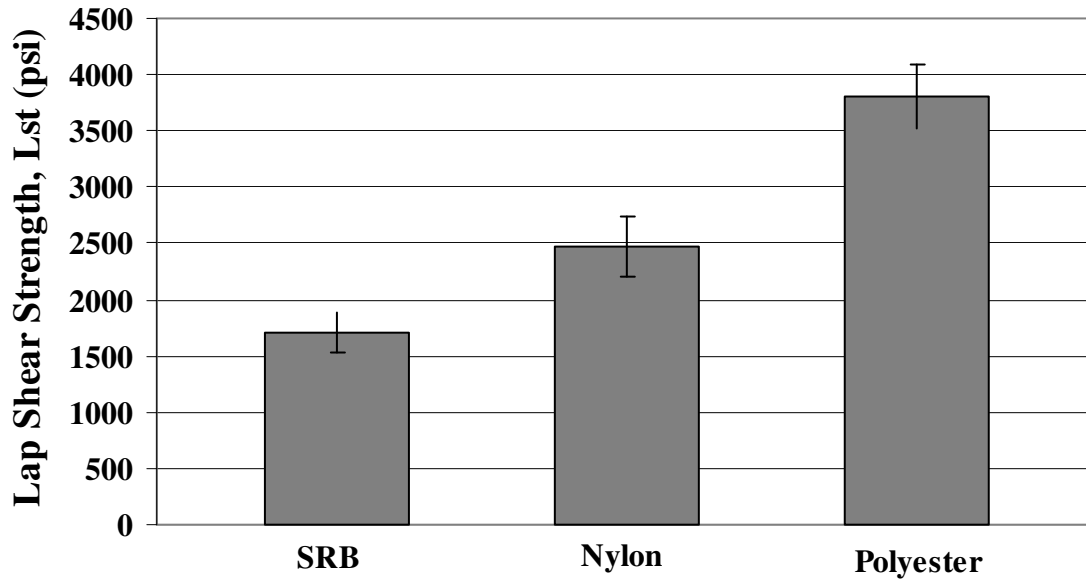
Fig 5.9) Baseline results for the lap shear tests after soak duration of 6k hours in water at 140 ºF

An investigation of the ruptured surfaces was done to get a qualitative estimate of the failure different peel ply specimens was observed. Fig. 5.11 shows the mean creep rupture time modes of the specimens. Fig. 5.12 shows the failure modes of ruptured specimens under creep load. It can be observed that the SRB and nylon peel ply specimens underwent complete adhesion failure whereas polyester peel ply specimen failed at about 70% by adherend and 30% by cohesive failure mode.
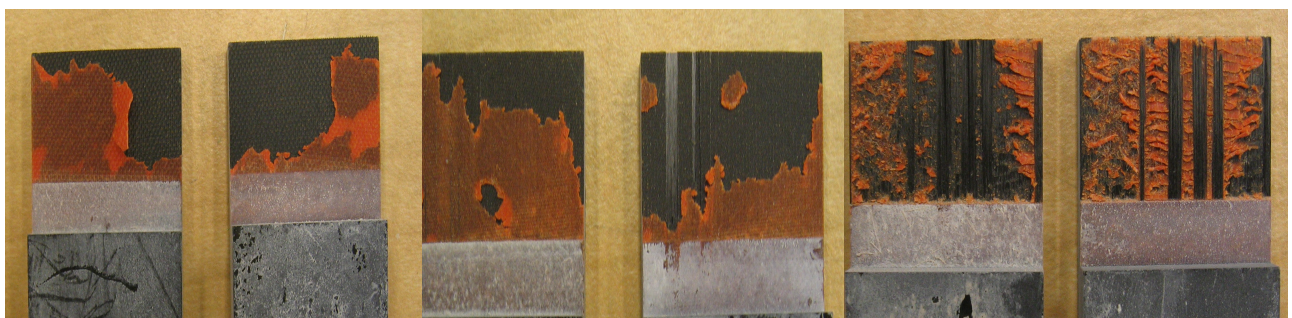
SRB	Nylon	Polyester
100% adhesion	100% adhesion	50% adherend, 50% cohesive

Fig 5.10) Failure modes of the lap shear specimens tested after soak duration of 6k hours in water at 140 ºF
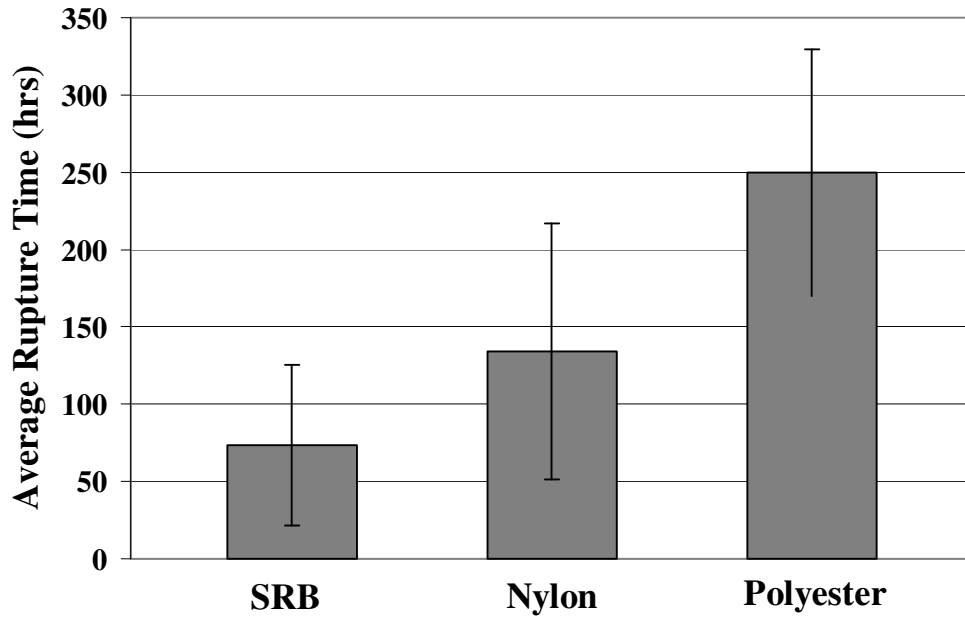
Fig 5.11) Mean creep rupture durations of the lap shear specimens

SRB	**Nylon**	**Polyester**
100% adhesion	100% adhesion	70% adherend, 30% cohesive
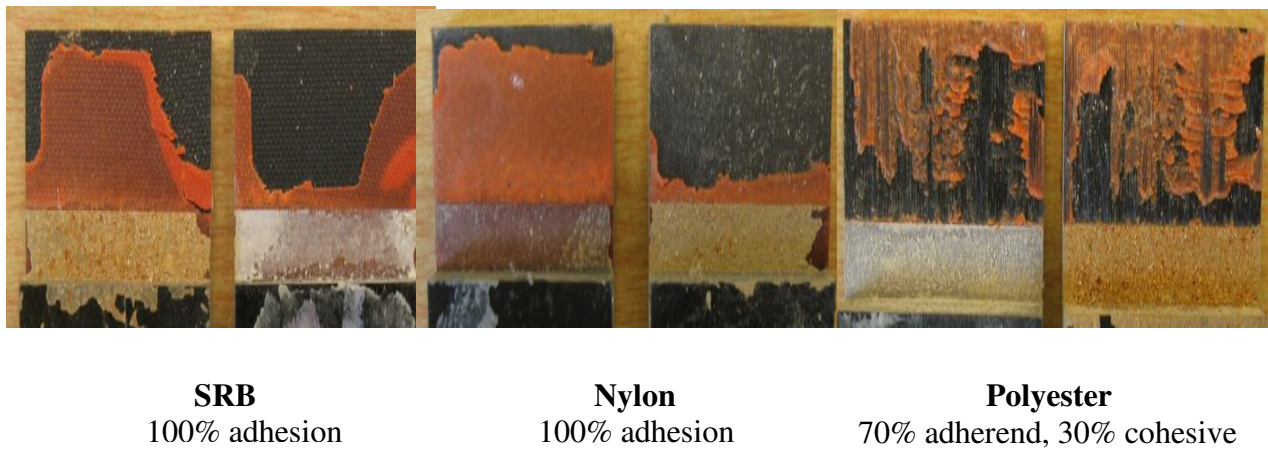
Fig 5.12) Failure modes of lap shear specimens resulting from the creep rupture tests

Double cantilever beam (DCB) test

Double cantilever beam tests were done to quantify the fracture resistance of the adhesive bonds. The specimens used in this test were pre-conditioned in water at 140 ºF for 6000 hours. DCB specimens were subjected to a constant displacement load and the critical strain energy release rates were recorded. Table 5.3 shows the test matrix concerned with these tests. Fig 5.13 shows the average G_{IC} of the DCB specimens cured against various peel plies. It can be noticed that the average critical strain energy was highest for polyester peel ply specimens followed by nylon and SRB peel ply specimens. A qualitative examination of the fractured surfaces revealed adherend failure modes in case of polyester peel ply bonds while adhesion failure modes in nylon and SRB peel ply surfaces as shown in Fig 5.14. The load-displacement curves generated by a typical specimen of each kind are shown in the Fig. 5.15. It can be inferred from the graphs that the peak loads were highest for the polyester bonds. Compared to polyester the peak loads for the nylon and SRB bonds were lower by 60% and 88% respectively. The crack propagation behavior showed jagged propagation for polyester and smooth propagation for nylon and SRB bonds. More energy was required to create fractured surfaces for polyester bonds and less energy was required to create fractured surfaces for nylon and SRB bonds

Table 5.3 Test matrix for the DCB tests, 6k hour pre-exposure to reach saturation

Specimen type	6k hrs	10 k hrs
DCB/Polyester	5	5
DCB/Nylon	5	5
DCB/SRB	5	5
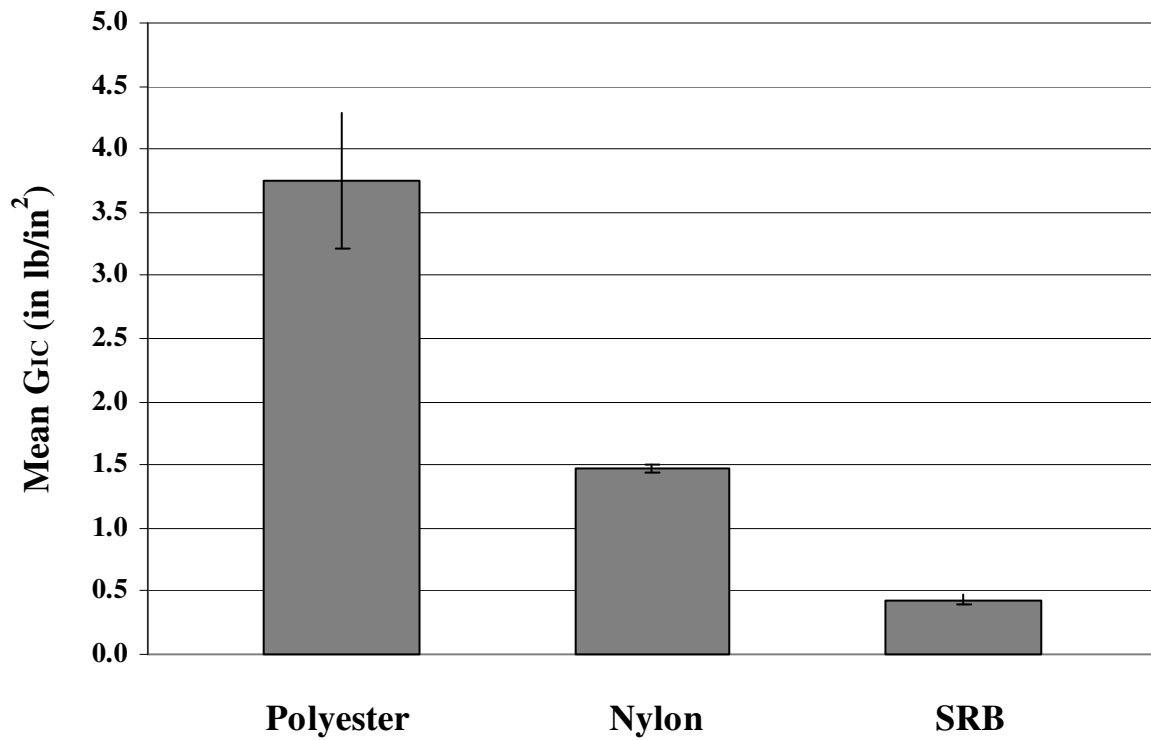
Fig 5.13) Mean critical strain energy release rates (G_{IC}) of DCB specimens

SRB	Nylon	Polyester
100% adhesion	90% adhesion, 10% adherend	100% adherend

Fig. 5.14) Failure modes of DCB specimens tested after soak duration of 6k hours in water at 140 ºF

**Fig 5.15) Load-displacement curves of DCB tests**

Wedge crack (WC) test

The wedge crack (WC) test method was a durability test to study the long term durability of adhesive bonds. Previous studies have found that this test predicted long term durability of adhesive bonds made from aluminum adherends. The Boeing we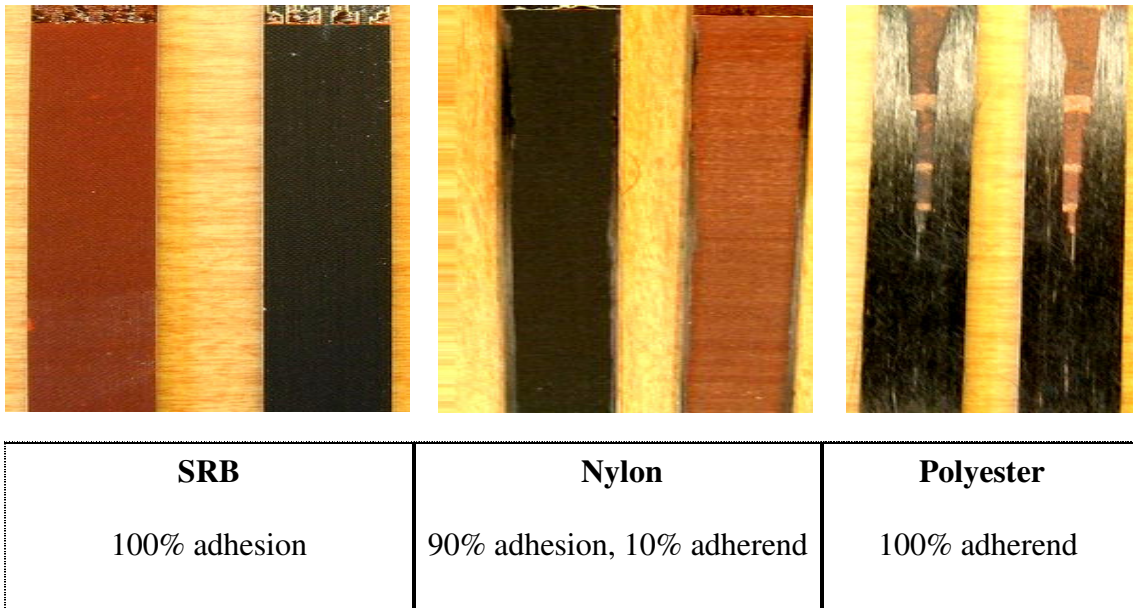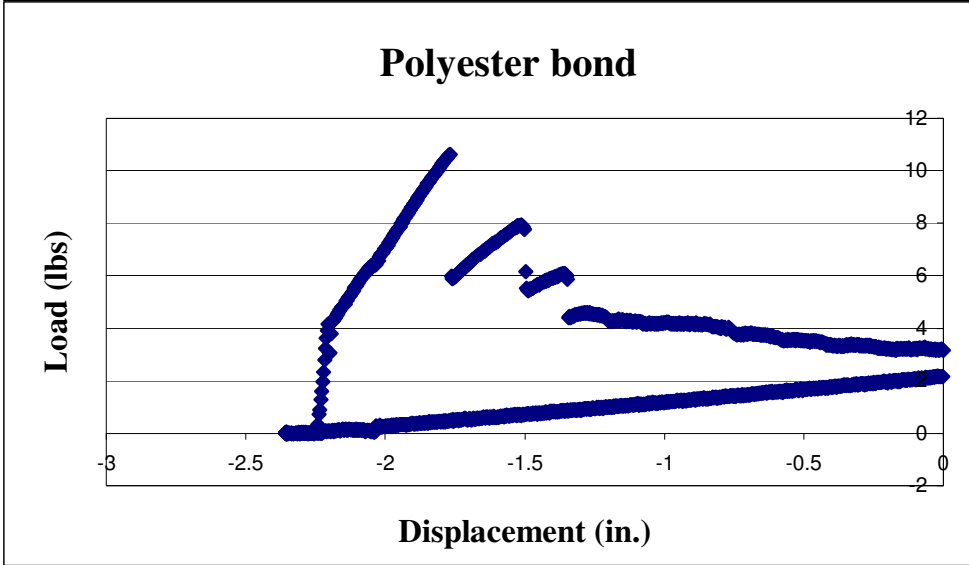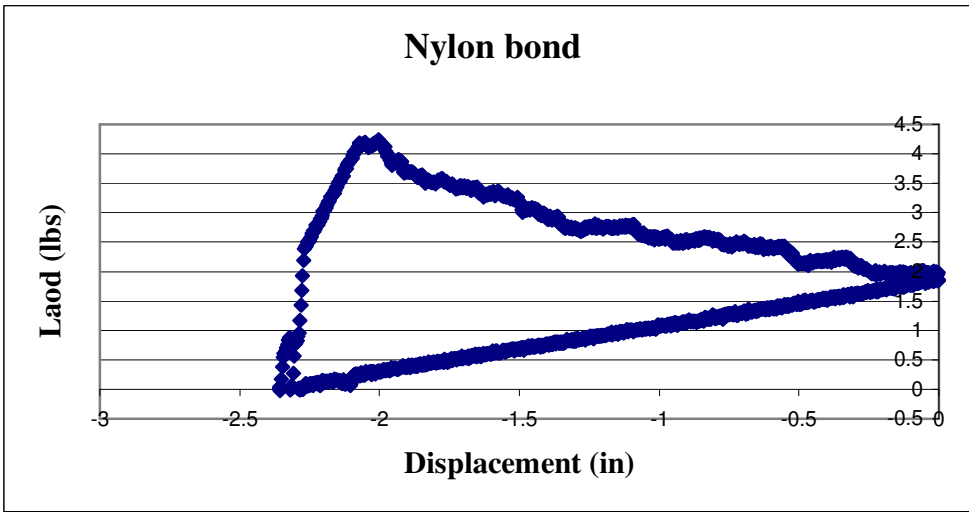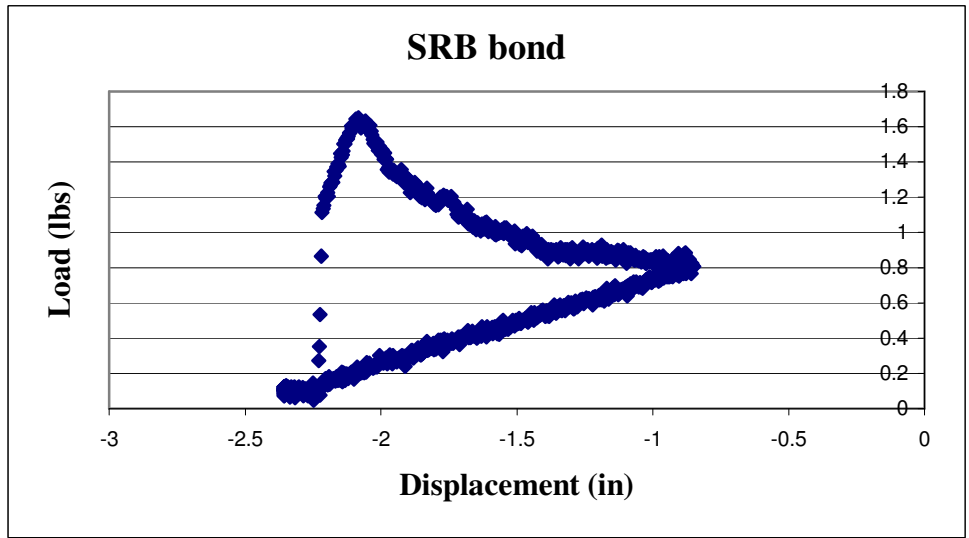dge test demonstrated that durable surface preparation (such as, phosphoric acid anodize) resulted in no/low crack growth for all adhesives evaluated. With non-durable surface preparations of the aluminum adherends, the crack growth was high regardless of the adhesive. The driving mechanism which helped in accelerated crack propagation was the oxidizing of the primer surfaces in humid environments which allowed crevice corrosion to be propagated by the wedge (mode I) stress. This crack propagation was not found with phosphoric acid anodizing surface treatment of the aluminum adherends which consistently produced superior performance to that produced by other conventional industry standard methods [17]. It was due to hydration resistance of oxides produced by anodization in phosphoric acid. The maximum crack propagation of the wedge specimens prepared by phosphoric acid anodizing was approximately 0.2 inch, while the FPL (forest products laboratory sulfuric acid-sodium dichromate) etch had some values exceeding the 0.75 inch process control limit [18]. It was predicted the wedge crack test would not work with composite adherends as they are resistant to corrosion. But an attempt was made to study the wedge test by choosing a range of stiffnesses by varying the adherend thicknesses. WC tests for composite adherend were done for the three peel ply surface preparations in water maintained at 140 ºF. Adherend thicknesses nearly equal to 0.059 ± 0.001 inches (8 plies), 0.074 ± 0.001 inches (10 plies) and 0.0888 ± 0.001 inches (12 plies) were bonded to adhesive and the crack growth was observed. The bending

stiffnesses (EI) of the 8, 10 and 12 plies WC composite specimens were 365.2, 695.6 and 1202 lb in^2 respectively while, the stiffness of the WC aluminum specimen was 1571 lb in^2. The test matrix is shown in table 5.4.

The mean crack initiation length upon the insertion of the wedge was noted for each kind of specimen as shown in Fig 5.16 (a). All the specimens with inserted wedges were immersed in water at 140 ºF to observe further increase in crack growth under constant load conditions. Fig 5.16(b) shows average increase in crack growth while immersed in water for 24 hours. It can be seen from figure 5.16a) that in case 1 and 2, where the adherend were made of 8 plies and 10 plies respectively, the mean initial crack length after insertion of wedge and mean increase in crack length after exposure to water at 140 ºF for a period of 24 hours was noticeably higher for SRB WC specimens than the nylon and polyester WC specimens. While in case 3, where the adherend was made of 12 plies,

Table 5.4 Test matrix for theWC tests; Test conducted on dried WC specimens in water immersion at 140 °F

Specimen type	8 plies	10 plies	12 plies
WC/Polyester	5	5	5
WC/Nylon	5	5	5
WC/SRB	5	5	5

the mean initial crack length of the SRB WC specimens when compared to nylon and polyester WC specimens after insertion of wedge was little higher but there was no further increase in the crack length after exposure in water at 140 ºF for a period of 24 hours. SRB bonds showed lower durability with much faster crack growth while nylon

and polyester bonds did not clearly differentiate the bond durability. The crack trend showed by 8, 10, and 12 ply composite WC was not clearly understood.

Summarizing the effects of peel ply surface preparations it can be concluded that specimens cured against polyester peel ply resulted in better bonds with the highest ultimate lap shear strengths, longer mean creep rupture time and greater toughness. The durability of the bonds could not differentiate clearly between polyester and nylon bonds. The bonds made with SRB and nylon peel plies were weak due to the contaminants left by them. It was found that SRB peel ply transfers silicon coating on to the adherend surface which results in weaker bonds made to SRB peel ply surfaces [30]. Lower toughness of the nylon bonds was found due to significant amounts of nitrogen and amide groups on the surfaces processed by nylon peel ply [30].
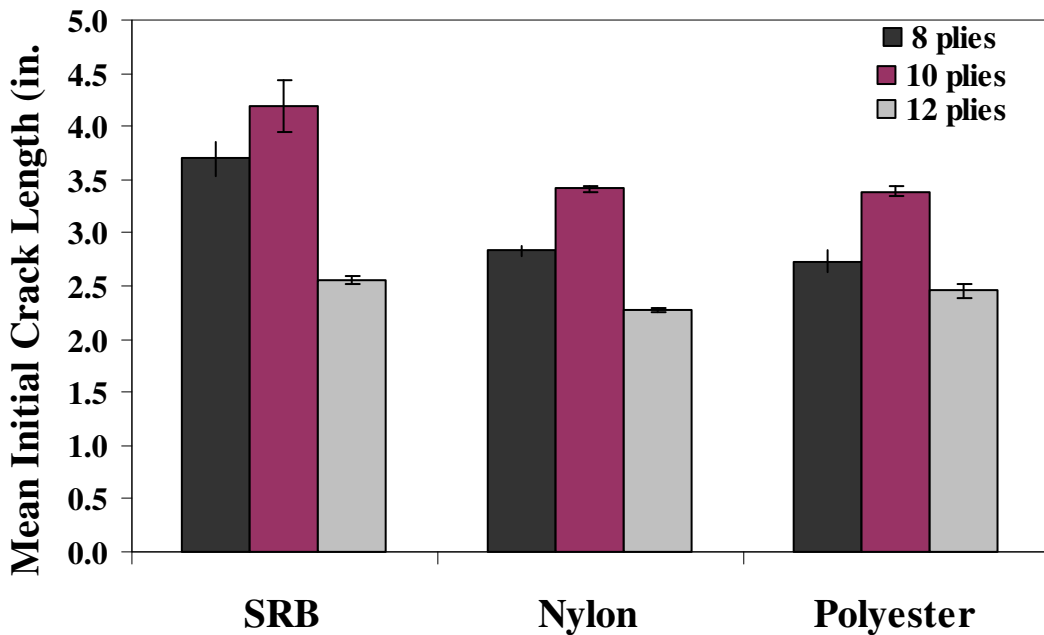
5.16) (a) Mean initial crack length after insertion of a wedge for various adherend ply thicknesses
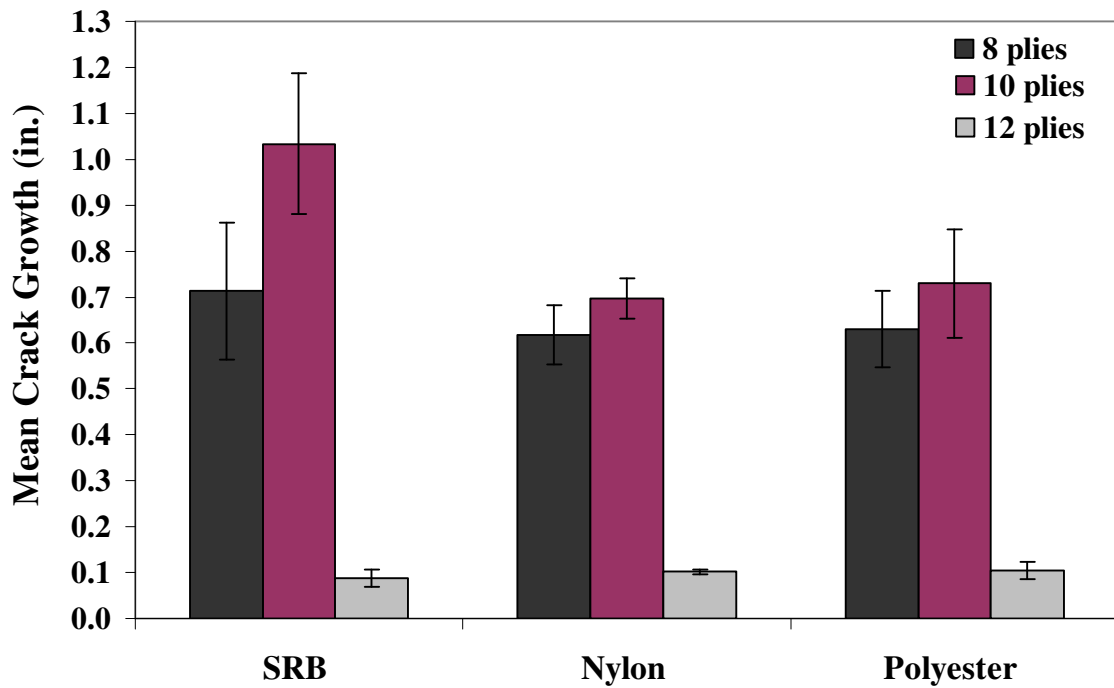
5.16) (b) Mean increase in crack length after exposure to water at 140 ºF for a period of 24 hours for various adherend ply thicknesses

5.3.3) <u>ABRASIVE TECHNIQUES STUDY</u>

The objective of this study was to find the effect of different abrasives techniques, treated on the adherend surface, on the fracture resistance of the adhesive bond. The two different kinds of abrasive techniques used in this study were grit blasting (GB), with grits of sizes 80 and 220, and sanding of grit 220. The specimens used were dried and there was no pre-conditioning before loading. The test matrix for this study is shown below in table 5.5.

The DCB specimens were tested to obtain their critical strain energy release rates as a measure of fracture resistance of the adhesive bond. In this study, DCB specimens were tested at no load exposure (baseline) and also subjected to constant and fluctuating load tests. Specimens were dried in an oven at 160 ºF before testing. In the base-line test, an

average mode-I strain energy release rates of the specimens was determined. Fig. 5.17 shows the dependence of mean critical strain energy release rate on various abrasive techniques used in the specimen preparation.

Table 5.5) Test matrix for the abrasive study

Type of specimen	140 ºF Water immersion			-65 ºF in air	No exposure, No load (Baseline)	Total coupons
	Constant load	Fluctuating load	Fluctuating load (9.5 lbs)	Fluctuating load		
Polyester	2	10	8	10	5	35
Sanded	2	10	9	10	5	36
GB 80	2	10	8	10	5	35
GB 220	2	10	9	10	5	36

Specimens prepared using sand paper of grit 220 had the highest resistance to mode-I fracture followed by polyester, GB 220 and GB 80. Fig 5.18 shows the SEM images of the adherend surfaces of polyester, sanded and GB treatments. The lower fracture resistance of specimen prepared using GB 220 and 80 was expected due to harsher effects of the grit blasting on the adherend surface as can be seen in the SEM images. Fig 5.19 shows the images of the failed surfaces of the DCB specimens from base-line tests. The failure modes of specimens indicated that bonds made to sanded surfaces have the highest percentage, nearly 99%, of cohesive failure, polyester resulted in 90 % cohesive and 10 % adherend failures, GB 220 had 70% cohesive and 30% adherend failures, while GB 80 resulted in 100% adherend failure.
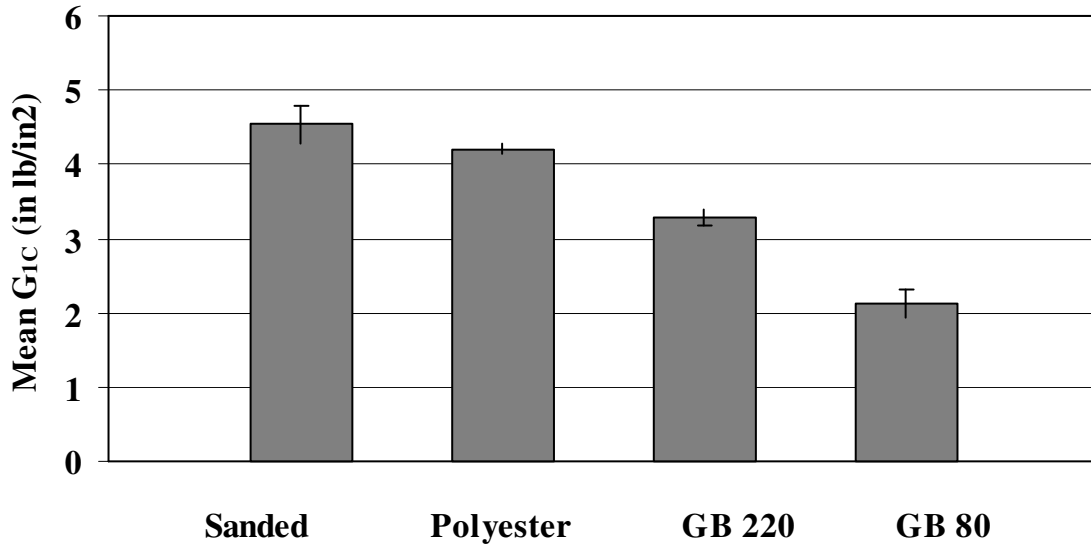
Fig. 5.17) Dependence of critical energy release rate on various abrasive techniques

POLYESTER

GB- 80

Surface erosion
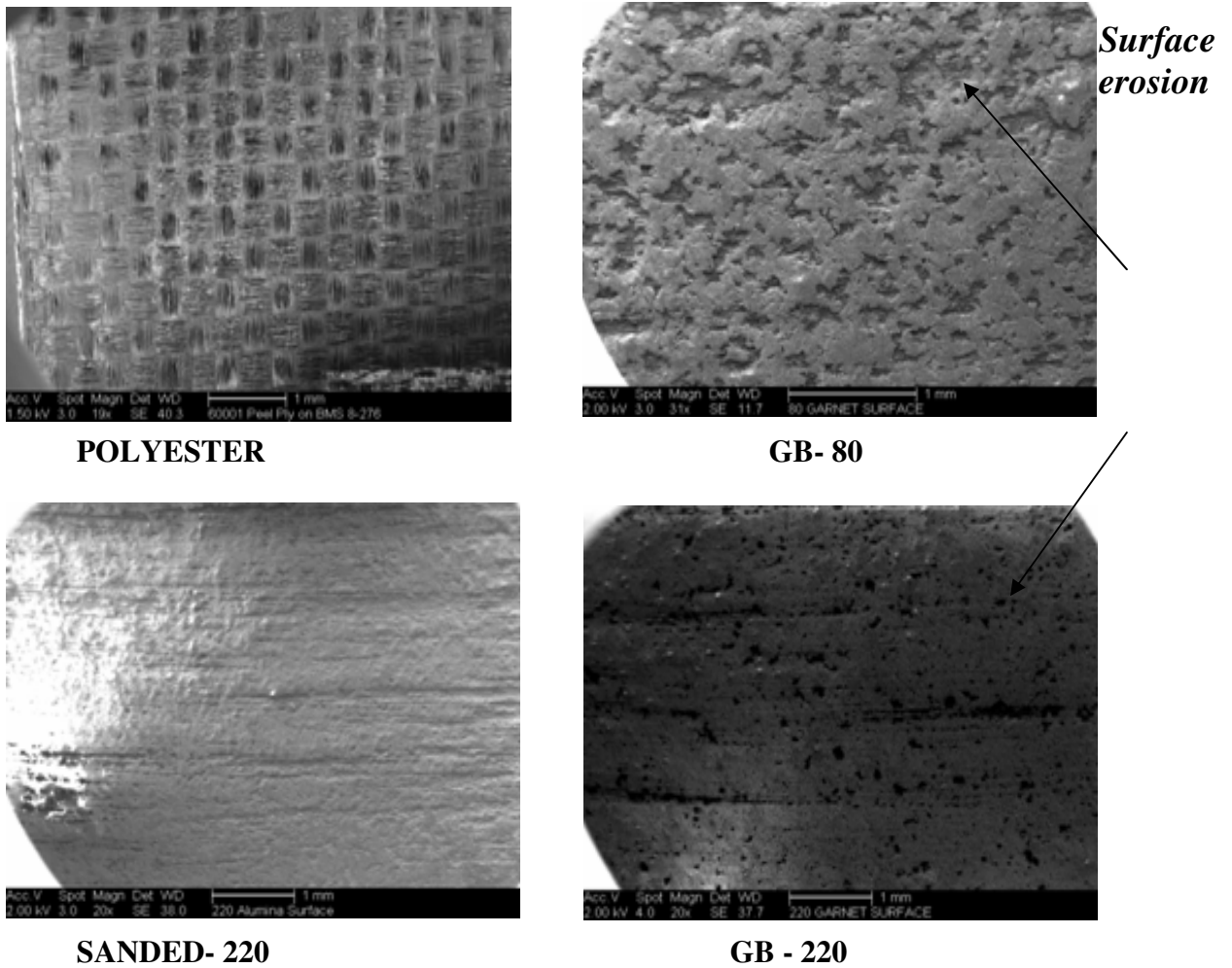
SANDED- 220

GB - 220

Fig 5.18) SEM images of the various abrasive surface preparations

In the constant and fluctuating load tests, the DCB specimens prepared using various abrasive techniques were subjected to 90% of their respective failure loads determined from the base-line test. Constant and fluctuating loads of 6.2 lbs, 8.4 lbs, 9.8 lbs and 10.3 lbs were applied on the GB 80, GB 220, polyester and sanded DCB specimens respectively. The specimens were immersed in water at 140 °F while under constant and fluctuating loading. Fig 5.20 and Fig 5.21 show the mean crack growths in the DCB

	Sanded
	99% cohesive, 1 % adherend
	Polyester
	90% cohesive, 10% adherend
	GB 220
	70% cohesive, 30% adherend
	GB 80
	100% adherend

Fig 5.19) Failure modes of DCB specimens resulting from base-line tests

specimens under constant and fluctuating loading, respectively. Results showed that specimens stressed at 90% of their respective fracture loads under constant load exposure showed no changed in crack growth except for GB 80 which had little crack growth. Fluctuating loading showed noticeable crack growth as can be seen in Fig. 5.21 and Fig. 5.22. . Figure 5.21 shows the mean crack growth with respect to time of the specimens under fluctuating loading of 90% fracture load. It was found that when applied a fluctuating load of 90% of fracture load for their respective DCB specimens results showed that 90% of polyester, 50% of sanded, 20% of GB 220 and none of GB 80 specimens failed after an exposure period of 5 days. The slopes were highest for the polyester and sanded specimens and lower for GB 220 followed by GB 80. It implied that

Fig 5.20) Mean crack growth for the DCB specimens prepared from abrasive techniques under constant loading of 90% of fracture load while exposed to water at 140 ºF

71

GB 80 had highest durability followed by GB 220, polyester and sanded. These results would be useful for a manufacturer under design criteria. The failure modes of the specimens represented 100% adherend failure for GB 80; 70-90% adherend failure for GB 220; 60-70% adherend and 30-40% cohesive failures for polyester while 90% cohesive failures for sanded specimens. When a mean fluctuating load of the fracture loads was applied there was a different trend showed by the DCB specimens. Fig.5.22 shows the results of mean crack growth rate of the DCB specimens under fluctuating loading of 9.5 lbs, while they were exposed to water at 140 ºF. It was observed that though the mean initial crack lengths of GB, sanded and polyester specimens were almost the same as before; the mean crack growth with respect to time
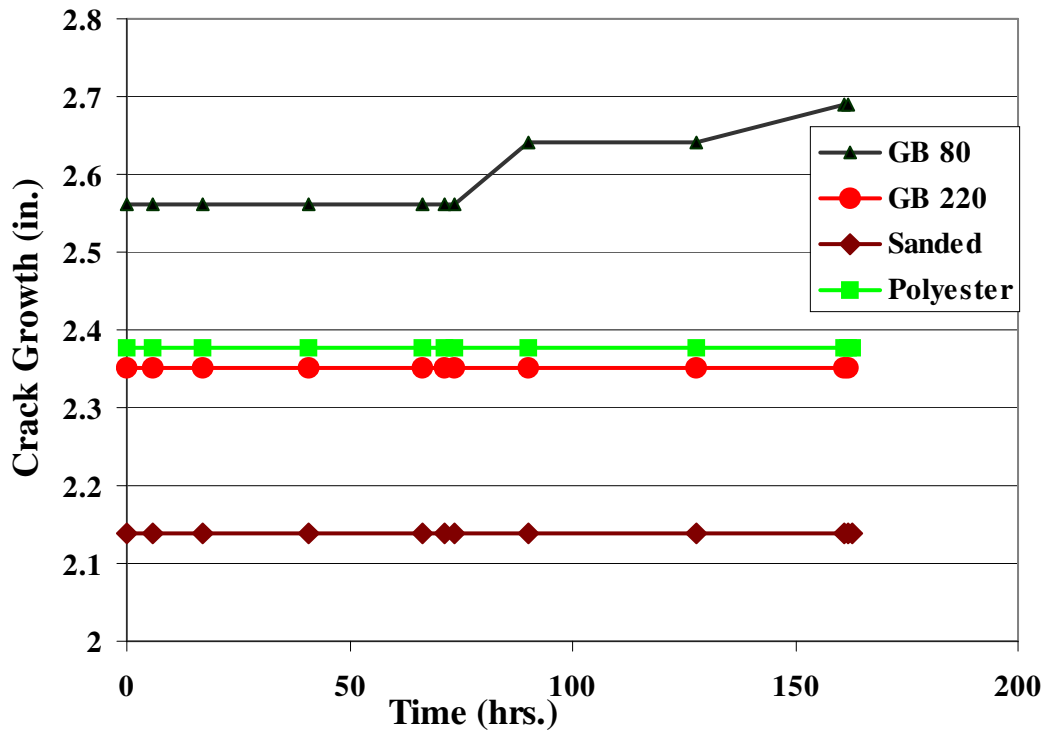

Fig 5.21) Mean crack growth for the DCB specimens prepared from abrasive techniques under fluctuating loading of 90% of fracture load while exposed to water at 140 ºF

was very different. The slopes of the mean crack growth for GB 220 and 80 specimens were highest followed by polyester and sanded specimens respectively. This meant that the mean crack growth was faster for the grit blasted specimens followed by polyester and sanded specimens respectively under the same load. After 550 hours of load exposure, all of the GB 220 specimens, 60% of GB 80, 40% of polyester and 30 % of sanded specimens failed. Sanded specimens showed longer durability, better bond quality with lowest slopes of mean crack growths under the application of same load of 9.5 lbs. Sanded resulted in 80% cohesive failure modes while GB 80 had complete adherend failure and GB 220 resulted in 60% cohesive and 40% adherend failure. Polyester peel

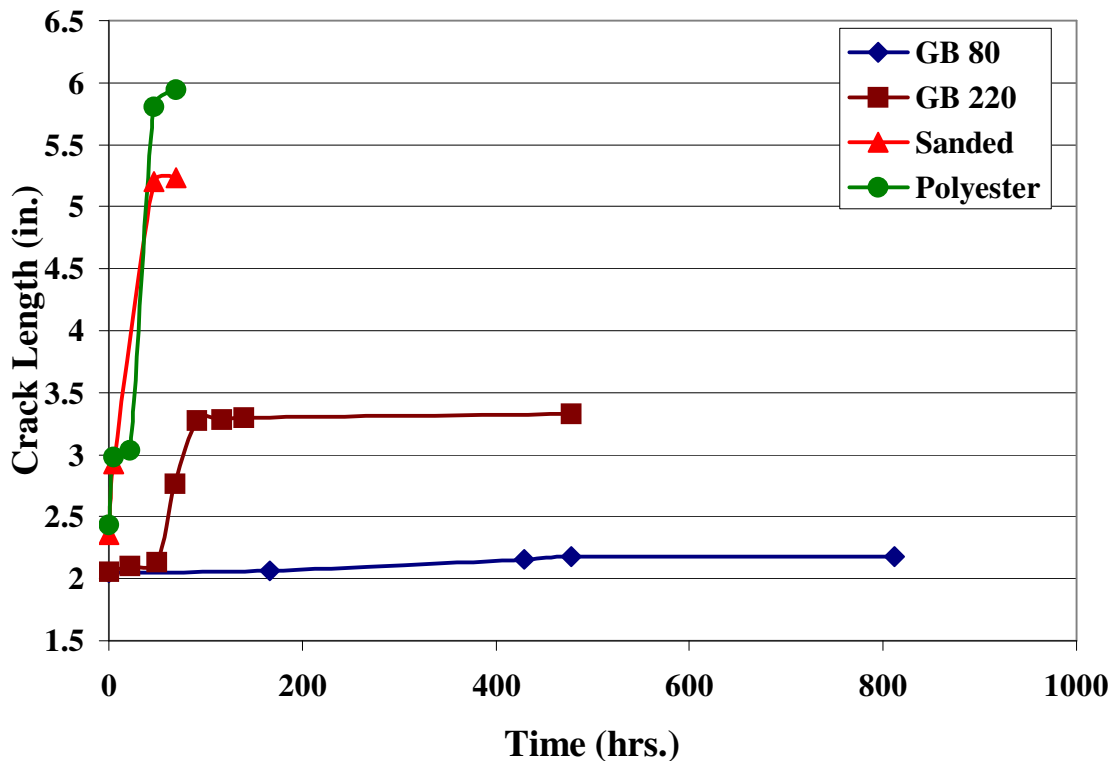**Fig 5.22) Mean crack growth for the DCB specimens prepared from abrasive techniques under fluctuating loading of 9.5 lbs while exposed to water at 140 ºF**

ply specimens had nearly 50-50% adherend and cohesive failure. The failure modes are shown in Fig 5.23. It was found that critical strain energy release rates of the bonds increase with increase in exposure temperatures [31]. Therefore, at an exposure to -65 ºF, only 45% of the fracture loads were applied. A fluctuating load of 3.1 lbs, 4.2 lbs, 4.9 lbs and 5.15 lbs were applied on the GB 80, GB 220, polyester and sanded DCB specimens respectively. Figure 5.24 shows the mean crack growth rate of the DCB specimens under fluctuating loading of their respective loads, while they were exposed to -65ºF

GB 80	
100% adherend	
GB 220	
60% cohesive, 40% adherend	
Polyester	
50% cohesive, 50% adherend	
Sanded	
90% cohesive, 10 % adherend	

Fig 5.23) Failure modes of DCB specimens resulting from fluctuating load test. Load applied is 9.5 lbs on all specimens.

temperatures. The slopes were lowest for the GB 220 followed by GB 80, polyester and sanded specimens. It implied that under -65ºF exposure temperature and fluctuating load of 45% fracture load durability was highest for GB 220 followed by GB 80, polyester and sanded specimens respectively. This test under went significant operational issues. At -65ºF temperatures some of the DCB fixtures were frozen and the blocked actuators did not apply the desired load. Therefore, the differences in the performances of GB 220 and GB 80 specimens can be attributed to the uncertainty of the crack growth values under such disabled operating conditions.
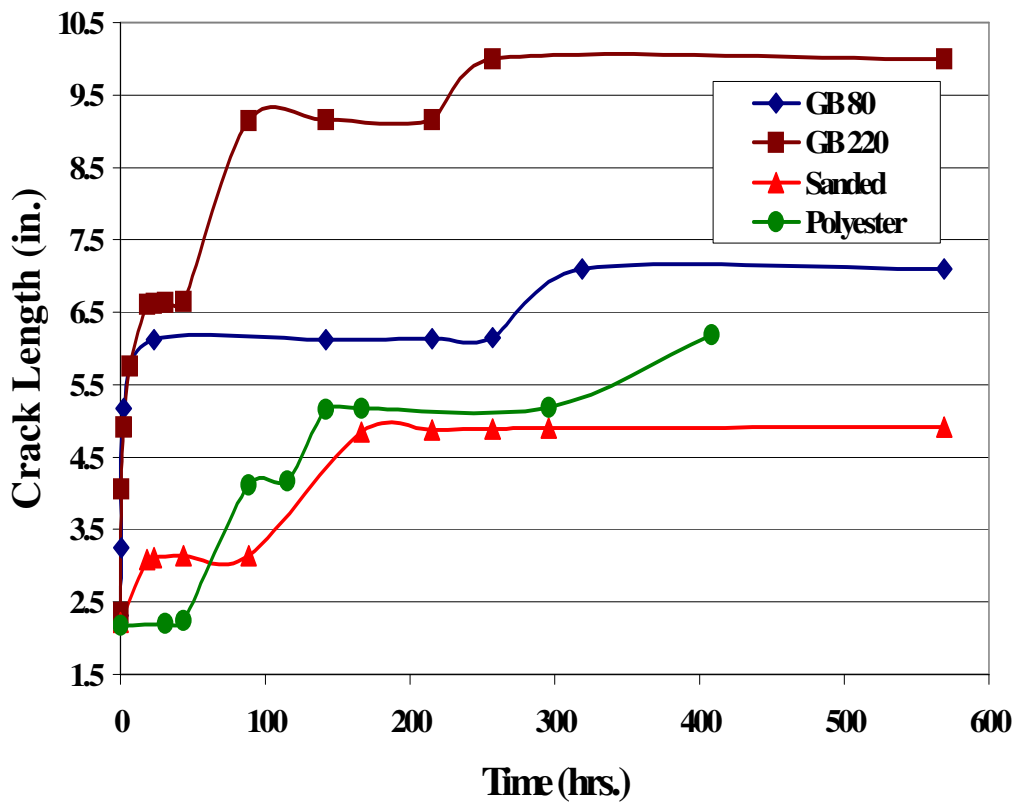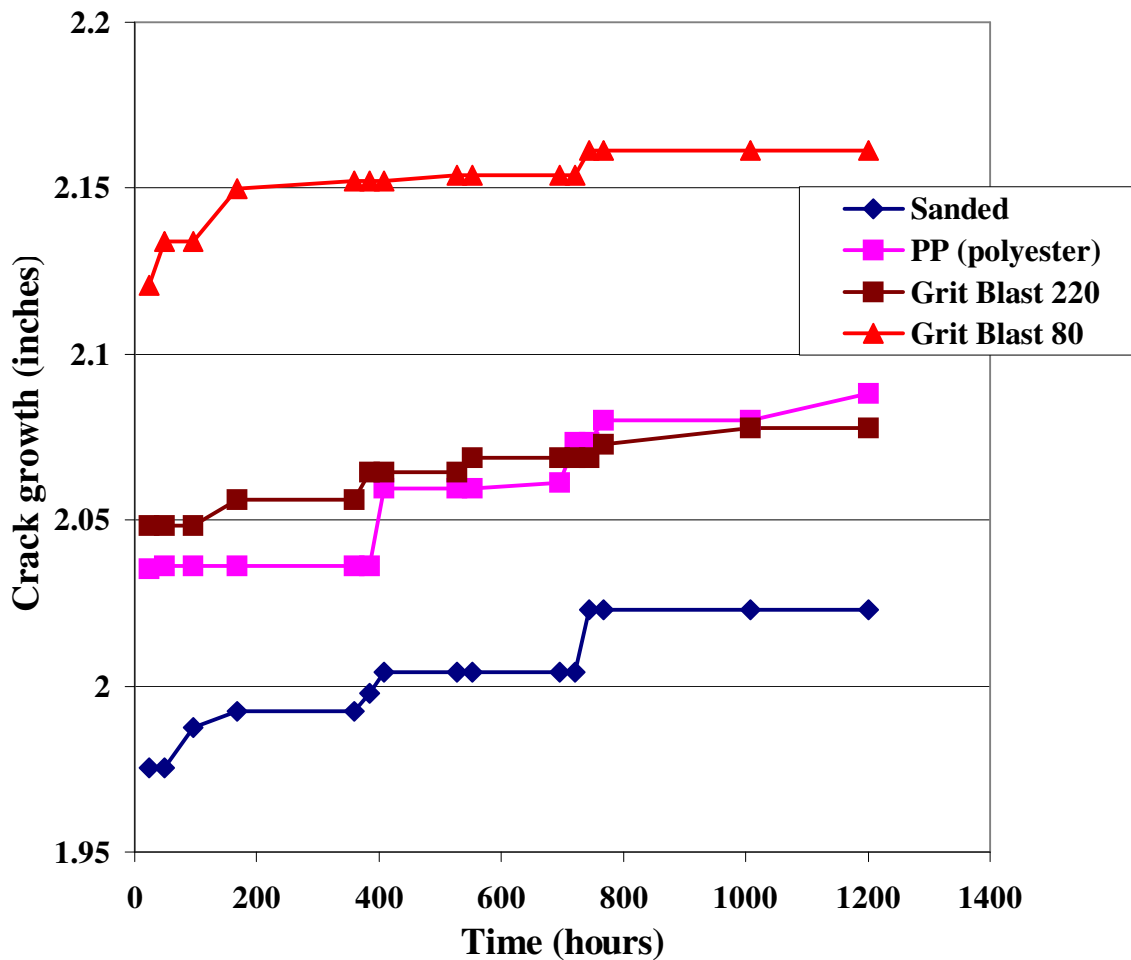

Fig 5.24) Mean crack growth for the DCB specimens prepared from abrasive techniques under fluctuating loading of 45% of fracture load while exposed to -65 °F temperature

Summarizing the constant and fluctuating load results, it was found that under the criteria of applying 90% of fracture loads GB 80 followed by GB 200 had better durability while, under the application of same mean load of 9.5 lbs, sanded followed by polyester specimens had better durability among the DCB specimens. GB specimens had lower fracture loads and critical strain energy release rates. This was due to the harsher grit blasting which resulted in surface erosion. The performance of sanded specimens can be ascribed to the cleaner surface areas produced due to sanding. As there was only little difference in the durability of polyester to sanded specimens, it is economically advantageous to use polyester peel ply which is a primary method of surface preparation. Secondary techniques need extra labor and are expensive.

SUMMARY AND CONCLUSIONS

The long term durability of adhesive bonds involving composite adherends was studied. The aims of the study were to investigate the effects of moisture, surface preparation and to investigate accelerated degradation methods on the adhesive bonds. Dry and saturated (to 1% by weight moisture content) adherends were bonded which were then immersed in water to study the effects of moisture. Degradation was accelerated using constant temperature (-65,140,160 °F) and constant and fluctuating stress (45% to 90% of maximum load). To consider surface preparation effects composite plaques were processed using three types of peel ply. Peel ply played a significant role as the surface preparation of the pre-bonded composite surfaces. Subsequent abrasive treatments were given to some surfaces to consider benefits of secondary operations.

In studying the effects of adherend moisture content the diffusion coefficients at 140 °F were found to be 0.00106 mm^2 /hr and 0.0044 mm^2 /hr respectively, while the saturation levels were found to be nearly 1.25 and 3.0 for the adherend and adhesive respectively. The results showed no difference in the lap shear strengths in dry as well as wet bonded specimens. This implied that the AF555 adhesive was insensitive to moisture. Examination of the fracture surfaces showed increased adherend failure with moisture content. With the increase in moisture content the shear and fracture toughness were found to decrease. There was a decrease of 12% in the shear strength and 10%-18% in the fracture toughness of the bonds for a soak period of 5k and 6k-10k hours respectively. Adherend shear tests (no adhesive) showed similar moisture sensitivity with a decrease of 15-20 % and 8-10% in the interlaminar shear and in-plane shear strengths respectively.

This implied that the reduced bond strength was due to the effects of moisture on the composite rather than the adhesive.

When the degradation was studied by applying stress, fluctuating load showed much higher crack growth in the fracture coupons than a constant load of the same magnitude. This implied that application of fluctuating load was a better accelerating technique in evaluating degradation of the bonds. On application of a constant load of 4 psi the bond strength of shear coupons was observed to decrease by 18% over 1000 hours. Those observations suggested that stress can be used to accelerate degradation. Surface preparation and surface contamination were important factors not to be neglected. Hence, upon investigation peel ply was found to play a significant role in the strength of a bond. Polyester peel ply resulted in highest strength followed by nylon and finally by SRB (35% and 55% greater than nylon and SRB respectively). The strengths of the nylon and SRB bonds were low due the surface contamination [30]. The debris left by the peel ply affected the bond. Nylon and SRB specimens had adhesion failure but polyester specimens had predominant adherend failure. During the creep rupture tests the weaker bonds also degraded faster which showed that degradation was proportional to the bond quality.

The effects of secondary surface preparations were studied by using grit blasting and sanding abrasive techniques. They also had a measurable effect on bond quality. Sanding improved the fracture toughness by 8% while; GB 80 and 220 techniques decreased the fracture toughness by 22% and 50% respectively. The improved bond by sanding may be attributed to a cleaner and more uniform bond surface, while the decrease bond quality from grit blasting was shown to be due to surface erosion. The cost of labor and

production is higher in using secondary techniques which is a disadvantage and so polyester peel ply can be favored.

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