An Experimental Investigation of GLARE and Restructured Fiber Metal Laminates

Adelina Vanessa Benedict

Embry-Riddle Aeronautical University - Daytona Beach

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AN EXPERIMENTAL INVESTIGATION OF GLARE AND RESTRUCTURED FIBER METAL LAMINATES

by

Adelina Vanessa Benedict

A Thesis Submitted to the College of Engineering, Department of Aerospace Engineering in Partial Fulfillment of the Requirements for the Degree of Master of Science in Aerospace Engineering

Embry-Riddle Aeronautical University
Daytona Beach, Florida
June 2012
AN EXPERIMENTAL INVESTIGATION OF GLARE AND RESTRUCTURED FIBER METAL LAMINATES

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Adelina Vanessa Benedict

This thesis was prepared under the direction of the candidate’s Thesis Committee Chair, Dr. David J. Sypeck, Associate Professor, Daytona Beach Campus, and Thesis Committee Members Dr. Daewon Kim, Assistant Professor, Daytona Beach Campus, and Professor John M. Weavil, P. E., Daytona Beach Campus, and has been approved by the Thesis Committee. It was submitted to the Department of Aerospace Engineering in partial fulfillment of the requirements for the degree of Master of Science in Aerospace Engineering.

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Acknowledgements

I dedicate this thesis to my advisor, Dr. David J. Sypeck. I cannot thank him enough for the tremendous amount of time and effort he has spent in helping me, for making me a more hands on student, for teaching me the value of patience and humbleness, and for giving me the opportunity to become a graduate teaching assistant, as the job was very meaningful to me.

I sincerely thank Dr. Daewon Kim and Professor Weavil for taking time out of their schedules to provide me with feedback in the completion of this thesis. I would also like to thank Yi Zhang and Mathew Carlton for their help at the composites lab in some parts of this project.

Last but not least, I take this opportunity to thank my parents, Augustes and Mary, my siblings, family, and good friends for their continuous encouragement and support throughout my school years. Not forgetting my best friend, Deep Lad for always being there for me throughout my years in college.
Fiber Metal Laminates (FMLs) are a group of materials fabricated by bonding glass/epoxy layers within metal layers. This class of materials can provide good mechanical properties, as well as weight savings. An FML known as Glass Laminate Aluminum Reinforced Epoxy (GLARE) was studied. An experimental investigation comprising of microscopy and tensile testing was carried out using different grades of GLARE. Microscopy revealed the construction details of GLARE, while tensile testing provided means of measuring and analyzing its stress-strain responses. Next, different metal surface pretreatment methods were explored. These included sandblasting, Phosphoric Acid Anodizing (PAA), and AC-130 Sol-Gel treatment. Woven S-2 glass, an epoxy adhesive, and aluminum alloy sheet metal were used to fabricate restructured FMLs using time and cost effective procedures. Additional microscopy and tensile testing allowed for comparisons with GLARE and aircraft grade aluminum alloys. The restructured FMLs showed similar behaviors to GLARE with potential significant improvements in fabrication efficiency.
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I INTRODUCTION

Purpose and Significance of Study

The purpose of this fabrication and testing procedure was to fabricate fiber metal laminates that provided weight specific advantages with cost and time effective procedures.

Fiber metal laminates (FMLs) are materials that have composite layers sandwiched between metal layers. These composite layers typically consist of fibers embedded in an adhesive system.

Glass Laminate Aluminum Reinforced Epoxy (GLARE) is a very popular FML, especially in Europe. For this thesis, some background research was first conducted to study GLARE, which included its history, construction, features and properties. Next, an experimental investigation was carried out to determine how this material behaves mechanically and to compare its properties to conventional aircraft grade aluminum alloys. Microscopy examination and tensile testing provided important information about the material.

The significance of the study of GLARE performed in Embry-Riddle Aeronautical University (ERAU) helped to better understand FMLs and also provided an opportunity to redesign and fabricate new FMLs.

Before laminate layup procedures were performed, a study of different metal surface pretreatments was conducted to determine bonding characteristics. Those surface pretreatment methods were sandblasting, Phosphoric Acid Anodizing (PAA) and AC-130 Sol-Gel. Furthermore, these FMLs were tensile tested for comparison to GLARE.
### List of Acronyms

<table>
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<th>Acronym</th>
<th>Description</th>
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<tr>
<td>FML</td>
<td>Fiber Metal Laminate</td>
</tr>
<tr>
<td>GLARE</td>
<td>Glass Laminate Aluminum Reinforced Epoxy</td>
</tr>
<tr>
<td>ERAU</td>
<td>Embry-Riddle Aeronautical University</td>
</tr>
<tr>
<td>ARALL</td>
<td>Aramid Reinforced ALuminum Laminate</td>
</tr>
<tr>
<td>PAA</td>
<td>Phosphoric Acid Anodizing</td>
</tr>
<tr>
<td>SFT</td>
<td>Self-forming Technique</td>
</tr>
<tr>
<td>FMLC</td>
<td>Fiber Metal Laminates Centre of Competence</td>
</tr>
<tr>
<td>HSS</td>
<td>High Static Strength</td>
</tr>
<tr>
<td>GTMS</td>
<td>Glycidoxypropyltrimethoxysilane</td>
</tr>
<tr>
<td>TPOZ</td>
<td>Zirconium n-propoxide</td>
</tr>
<tr>
<td>GAA</td>
<td>Glacial Acetic Acid</td>
</tr>
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II LITERATURE REVIEW

History of Fiber Metal Laminates

Overview of the Predecessors of GLARE

Throughout the history of aerospace and aviation, intense research and development on different materials have been conducted. Research and development continues all around the world. It is an ongoing process. No material is perfect or flawless and as new technology is developed, materials can be improved.

Wood was widely used in the early days of aviation. However, wood is susceptible to a variety of issues including decay, attack by insects, weakening of glued joints, and dimensional instability caused by differences in moisture [1].

The need for larger aircraft and the development of more powerful propulsion systems required a stronger, yet lightweight material. That material was aluminum. It too had deficiencies. For example, aluminum aircraft skins burn away in less than a minute in kerosene fires [2]. Extremely high temperature applications, such as those experienced in space exploration will have a similar effect. Aluminum alloys like duralumin (2024 alloy) suffer from inter-crystalline corrosion, which is difficult to detect on the surface of the material. This can result in a dramatic reduction in the stress resistant properties of the structure.

FMLs provide the opportunity to utilize the combination of aluminum with glass/epoxy composite.
The British aircraft company, De Havilland (Hertfordshire, UK) started bonding metal parts together with adhesives in the 1940’s. This technology was later used at Fokker (Amsterdam, The Netherlands), another aircraft manufacturer, when an engineer named Rob Schliekelmann moved from De Havilland to Fokker [3]. He enhanced the production of these bonded structures with the use of autoclaves and improved pretreatment of the aluminum layers in laminated structures, which were then used in the center wings of the Fokker F-27 Friendship shown in Fig. 1.

![The F-27 Friendship by Fokker was one of the first aircraft to use laminated structures [4].](image)

**Figure 1.** The F-27 Friendship by Fokker was one of the first aircraft to use laminated structures [4].

Fatigue tests that were conducted on the laminated metal structure used for the center wings revealed crack growth initiation in a single layer, while the other layers bridged the crack, consequently slowing down its rate of growth [3]. This is a huge advantage of laminated metal structures and also FMLs.

Schliekelmann’s group reinforced their metal laminate adhesives with nylon and carbon fibers. Woven nylon fibers and unidirectional carbon fibers were embedded within 1 mm thick sheets of aluminum. These structures were considered FMLs and achieved 2 to 3 times slower crack growth rates when compared to just aluminum [3].
In 1978, at the Delft University of Technology (Delft, The Netherlands) Have, Schijve, and Vogelesang compared the strengths of unidirectional carbon and woven aramid fiber based laminates. They found that the carbon fibers remained fully intact, while the aramid fibers at a certain distance from the crack tip were broken. The carbon fibers were in the loading direction and only half of the aramid fibers had applied loads (because they are woven) [3].

Further research on FMLs that used aramid fibers were conducted at Delft University. An analytical model based on fracture mechanics was developed by Marissen to predict fatigue crack growth by calculating the stress intensity factor of the fatigue crack. His calculations took into consideration fatigue cracks in the metal layers and the load sustained by crack bridging of intact fibers at the affinity of the crack [3]. The comparable crack growth rates between his observations and predictions established two important concepts in the development process of FMLs. Higher strength fibers will better bridge fatigue cracks and thinner metal layers will compensate for more fiber layers, which would consequently minimize the shear stresses in the adhesive between the metal and fiber layers (reduces delamination) [3].

With this concept, ARALL (Aramid Reinforced ALuminum Laminate) was born. The aramid fibers were embedded in an epoxy adhesive and then sandwiched between sheets of aluminum alloy 2024 or 7475. ARALL was commercially launched in 1982/83. It was a success in wing panel applications with only minor cracks when subjected to 3 times the design life of the Fokker F-27 (270,000 flights), with 33% weight saving benefits [3].
Nevertheless, ARALL had its limitations. It had low blunt notch strengths (strength reductions caused by drilled holes) and required the use of doublers to prevent premature fatigue cracking, making it unsuitable for fuselages. Research by Roebroeks claims that aramid fibers lay loose without sufficient bonding to the adhesive, causing fiber pull out. The fibers then break easily when compressive loads are applied to them [3]. This led to the development of a modified ARALL with glass fibers by Roebroeks and Vogelesang at Delft. Vermeeren worked with carbon fibers and titanium to replace the aramid fibers and aluminum respectively for higher temperature applications, an area of continued interest.

The Boeing (Chicago, IL) C-17 military transport airplane cargo doors were manufactured using ARALL 3 (made with layers of aluminum alloy 7475 that were stretched after curing) resulting in 26% weight savings. The cost of this manufacturing process was 8 to 10 times more than aluminum cargo doors, resulting in only about 30 airplanes built with ARALL doors. Also, fuselage studies conducted on ARALL were unsatisfactory, as only 8% weight savings were achieved [3]. This led to the development of GLARE.

**Overview of GLARE**

The first patent for GLARE (United States Patent 5,039,571) was awarded to AkzoNobel (Amsterdam, The Netherlands) on October 14, 1987 [5]. Roebroeks and Vogelesang were listed as the inventors of the material. However, the commercialization of GLARE began in 1991 [3]. Around this time, the Boeing 777 was at its final stages of development. Although, it was too late for GLARE to be used as material for the
airliner’s main parts, various studies were carried out to better understand its capabilities and competencies.

Vlot studied the impact properties of GLARE at high and low impact velocities [3]. He discovered that at low impact velocities, GLARE could withstand impact loads as good as aluminum and better than carbon fiber composites. Additionally, the glass fibers in GLARE demonstrated greater impact properties and were much stronger than aluminum at higher velocities [3]. A major advantage of FMLs or more specifically GLARE, is that the outer aluminum layer will dent due to plastic deformation when subjected to impact. This dent can be detected due to its visibility, unlike other composite materials. For this reason, GLARE was used in the manufacturing of the Boeing 777 cargo floor, a structure highly susceptible to impact damage [3].

Boeing also conducted tests for fire resistance of GLARE. GLARE resisted temperatures as high as 1200°C for up to 15 minutes at which GLARE layers separate after that creating enhanced insulation, keeping the inside temperature at about 100°C, while the outside aluminum layers melt away. In 1995, Galaxy Scientific Corporation, now SRA International Inc. (Fairfax, VA) used this material to create a blast resistance container that was tested by the FAA [3].

Larger sheets of GLARE were manufactured as a way of reducing costs. A fuselage study conducted on GLARE in 1990, resulted in approximately 26% weight savings. For each kilogram of weight saved, GLARE cost $280 extra (compared to aluminum) [3]. The bonding of aluminum sheets in the autoclave resulted in 2.5 m wider panels.
Garesché along with Delft engineers came up with a splicing concept, which reduced costs and led to large scale applications of this material [3]. The splicing concept will be discussed in more detail later in this chapter.

Oostrom first produced double curved panels in 1996 by laminating the metal and composite layers in double curved molds, curing it in a single autoclave cycle. This prevents the wrinkling of sheets and eliminates the need for expensive stretch forming operations to attain the desired contours [3]. An illustration of this process is shown in Fig. 2.

Figure 2. Curved GLARE panel formed by layup using a mold and cured in an autoclave [6].

GLARE proved its effectiveness when barrel tests (fatigue tests) were conducted for the Airbus (Toulouse, France) A330 and A340 fuselage sections (100,000 flight cycles). Fatigue tests were performed and then damage was inflicted by using saws to produce cracks and dents (stress concentrations).
In some cases, the aluminum layers on the outside were removed to study corrosion. The claim is that GLARE showed success [3]. Fig. 3 depicts a GLARE fuselage section undergoing cyclic loading.

![Image of a fuselage section being tested for fatigue](image)

**Figure 3.** Fuselage section being tested for fatigue [3].

The applications of GLARE became more and more prevalent in the aerospace industry. Sections of the Airbus A340 airliner and Learjet (Wichita, KS) 45 business jet fuselage bulkheads used GLARE extensively. For repair works over cracks, Fredell concluded that GLARE patches worked better because of a smaller thermal mismatch with aluminum structures [3]. Fig. 4 on the following page shows a bonded GLARE repair patch that was used on a Lockheed (Bethesda, MD) C-5A Galaxy. If an aircraft
interior is exposed to saltwater due to the transportation of seafood, GLARE stiffeners provide enhanced protection against corrosion. A study conducted by Soerjanto showed corrosion only on the outer layer, while composite layers served as corrosion stoppers [3].

![GLARE repair patch used on the C-5A Galaxy aircraft](image1)

**Figure 4.** GLARE repair patch used on the C-5A Galaxy aircraft [3].

A more recent application of GLARE is its use as the forward and aft top fuselage skins in the Airbus A380. Fig. 5 indicates areas on the A380 in which this material is used.

![A380-800 Materials Overview](image2)

**Figure 5.** The wide use of GLARE in the fuselage sections of Airbus A380 [7].
GLARE Features, Characteristics, and Properties

Constituents and Buildup

The focal purpose of creating a GLARE type FML is to combine the advantages of metal and glass/epoxy material to provide properties that are superior to conventional materials. GLARE consists of thin aluminum sheets with glass fibers embedded in epoxy adhesive laid up within. Their glass fibers embedded in epoxy are commonly referred to as prepreg.

The aluminum sheets have different thicknesses ranging from 0.2 to 0.5 mm. The prepreg consists of unidirectional S-glass and more recently S-2 glass fibers embedded in an adhesive, known as FM-94 by Cytec Industries Inc. (Woodland Hills, NJ). The volume fraction of the fibers in the prepreg is 59% [2].

There are six standard grades of GLARE; GLARE 1, GLARE 2, GLARE 3, GLARE 4, GLARE 5 and GLARE 6 [3]. These different grades are differentiated by fiber orientations and number of prepreg layers. Tests were performed on a few grades of GLARE and results are presented as part of this thesis and elaborated on in later chapters.

The aluminum sheets used in the manufacturing of GLARE are pretreated before layup. They are either Chromic or Phosphoric Acid Anodized (PAA) and primed with a corrosion inhibiting primer, known as BR-127. This primer is also manufactured by Cytec Industries Inc. The pretreated surface is bonded to the FM-94 prepreg. The quality of the pretreatment and adhesive primarily determines the strength of the bond between the aluminum layers and fiber layers [3].
A simple schematic representation of GLARE is shown in Fig. 6 to familiarize the reader with the constituents of the material. This figure only illustrates the aluminum and glass/epoxy layers without taking into account the orientation of the glass fibers, which will be discussed in later chapters. Fig. 6 represents GLARE 4A-4/3-0.3. The designation 4/3 indicates 4 layers of aluminum and 3 layers of glass/epoxy where the aluminum sheet thickness is 0.3 mm. Within each glass/epoxy layer, there are two or more prepreg layers whose orientation and layup vary depending on the grade of GLARE.

![Figure 6. GLARE 4A-4/3-0.3 construction.](image)

The various grades of GLARE are defined by the number of prepreg layers relative to the number of aluminum layers and the orientation of the fibers in the prepreg. The glass fibers used in GLARE contribute significantly to the strength of the laminate. Table 1 on the following page shows some of the mechanical properties of the S-glass fibers originally used to make GLARE. Properties for unidirectional S-glass fibers were established from tensile tests in the longitudinal orientation of the fibers. The more recent S-2 glass fibers have superior material properties and will be discussed in more detail later.
Table 1. Mechanical properties of unidirectional S-glass fibers used to manufacture GLARE [2].

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber diameter</td>
<td>~10 μm</td>
</tr>
<tr>
<td>Strength</td>
<td>4000 MPa</td>
</tr>
<tr>
<td>Young’s modulus</td>
<td>88 GPa</td>
</tr>
<tr>
<td>Strain at failure</td>
<td>4.45%</td>
</tr>
</tbody>
</table>

According to Roebroeks, although the adhesive system determines the bond strength and performance of the laminate, its mechanical properties are often low [2]. It can be observed in Table 2, that these values are much lower when compared to the mechanical properties of the fibers.

Table 2. Mechanical properties of the adhesive system used to manufacture GLARE [2].

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strength</td>
<td>±50 MPa</td>
</tr>
<tr>
<td>Young’s modulus</td>
<td>±1.7 GPa</td>
</tr>
<tr>
<td>Strain at failure</td>
<td>5-10% (depends on strain rate)</td>
</tr>
</tbody>
</table>

In this thesis, a material similar to GLARE was fabricated at ERAU. The purpose was to create a material with properties similar to GLARE, while reducing fabricating costs and simplifying. This laminate consists of surface pretreated aluminum layers and glass/epoxy layers that are made from woven S-2 glass embedded in epoxy adhesive. Details on raw materials and fabrication will be described in coming chapters.

Splicing Concept

One major problem with GLARE was producing wider panels within acceptable economic constraints. Fuselage skins require a minimum panel width of 2 m [3]. This restraint led to the development of the splicing concept. With splicing, the aluminum
layers have a small gap between each normally continuous sheet, while the prepreg layers are left continuous. The interruptions in these metal layers are called splices. This configuration is illustrated in Fig.7.

![Figure 7](image)

**Figure 7.** Splicing to manufacture larger panels by creating interruptions in the aluminum layers [6].

However, this technique resulted in the splice becoming the weakest component of the laminate. Typically, the outer aluminum layer delaminates when the transverse (out-of-plane) stress in the vicinity of the splice exceeds 400 MPa. This difficulty was overcome by using doublers and additional adhesive. Doublers are extra aluminum sheets or extra GLARE sheets added to reduce stress in the splice area. The adhesive used in the prepreg layers are often added to fill up gaps.

The method using autoclave pressure to form the laminate with doublers and adhesive in one cure cycle is known as the self-forming technique (SFT) [2]. Aluminum sheets added to the outside of the laminate to form a bridge are called external doublers, while internal bridging sheets are known as internal doublers.
Fiber Bridging and Delamination

Fatigue crack growth is a very common, yet complex problem faced by aerospace and many other products. Although prediction techniques have been developed, such as the Palmgren-Miner method, one can never be too certain of the occurrence of failure due to fatigue cracks. This is detrimental to the safety of the airplane or spacecraft. Materials with better resistance to rapid crack growth have always been sought. Schijve found that FMLs have smaller crack growth rates when compared to monolithic materials (single unreinforced materials) because of crack bridging and delamination effects [8]. This is said to be one of the advantages of GLARE.

With crack bridging, some of the loads normally borne by the aluminum layers are transferred to the fibers. Due to this, additional shear stress occurs at the metal-composite interface. For a given laminate thickness, thinner metal layers allows for more fibers, which is thought to result in small shear stresses at the interface. While these shear stresses create delaminations, the loads sustained by the fibers are reduced, resulting in fewer breaks [9]. A situation similar to fiber bridging is known as fiber nesting which removes some of the strain energy away from the crack tip [9]. Fig. 8 on the following page is an illustration of the crack bridging phenomena.
Properties

GLARE has properties that exceed aircraft grade aluminum alloys in many categories. This includes better corrosion resistance, impact tolerance, fire resistance, fatigue life, tensile strength, repair ability and maintainability [10]. Some of these properties as reported by the Fiber Metal Laminates Centre of Competence (FMLC) in Delft, The Netherlands are shown in Table 3.

Table 3. GLARE properties that exceed conventional aircraft grade aluminum alloy [10].

<table>
<thead>
<tr>
<th>Property</th>
<th>Ratio (GLARE / Al 2024)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>0.85 - 0.90</td>
</tr>
<tr>
<td>Structural weight</td>
<td>0.70 - 0.85</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>1.0 - 2.0</td>
</tr>
<tr>
<td>Compressive strength</td>
<td>0.9 - 0.95</td>
</tr>
<tr>
<td>Stiffness</td>
<td>0.70 - 0.85</td>
</tr>
<tr>
<td>Fatigue</td>
<td>3.0 - 100.0</td>
</tr>
<tr>
<td>Damage tolerance</td>
<td>1.0 - 2.0</td>
</tr>
</tbody>
</table>
III EXPERIMENTAL INVESTIGATION OF GLARE

Microscopy

*Introduction*

The simple schematic of the GLARE cross section in Fig. 6 (pg. 12) showed the basic construction of GLARE, but does not give a clear indication of fiber orientation and layup for different grades of GLARE. In this chapter, metallography and light microscopy are utilized to understand a few grades of GLARE.

A former ERAU student, T. A. Olaniyan [11] obtained selected GLARE panels (approximately 26 cm by 36 cm, which is slightly larger than A4 paper size) from the FMLC. Table 4 lists the grades of GLARE that were used for this study. Here, High Static Static Strength (HSS) is a special grade to be discussed in more detail later in this chapter. Two specimens perpendicular to each other from each panel were tensile tested because of potential anisotropy.

*Table 4.* The different grades of GLARE that were used in this study.

| GLARE grade | GLARE 3-4/3 0.4 HSS | GLARE 4A-4/3 0.3 | GLARE 4B-3/2 0.4 |

*Equipment and Procedure*

Small planar samples (approximately 10 mm by 10 mm) were cut from the GLARE panels using a tap water lubricated 7 in diameter diamond blade tile saw, Fig. 9.
Samples were press fit into aluminum sample holders (see Fig. 10 on pg. 19), then wet sanded on edge using a HandiMet® 2 hand sander (Buehler, Lake Bluff, IL) and subjected to two to three passes for each grit on 240, 320, 400 and 600 grit SiC paper using tap water. Then, they were polished (semi-automatically) for approximately 45 minutes on a soft nap Buehler MicroCloth® PSA lubricated with Buehler alpha alumina 0.3μ and water, Fig. 11 (pg. 20). This grit of alpha alumina is recommended for polishing ceramic materials [12]. A mirror like surface on the polished side is a good indication of process completion.

Samples were then rinsed with tap water followed by methanol. Methanol helps to remove impurities and promotes rapid drying. Polished surfaces were not touched or wiped with any sort of cloth to prevent damaging the surface or adhering contaminants to it. Once dry, surfaces were viewed under a metallurgical microscope and digital images were captured using a 3.0 megapixel microscope camera, Fig. 12 (pg. 21).

A similar polishing procedure was carried out using a planar aluminum 2024-T3 only sample (approximately 10 mm by 10 mm). However, this sample was polished on its largest planar surface to observe grain structure. Then, the surface was lightly etched using Kroll’s reagent which consists of 92 ml water, 6 ml HNO₃, and 2 ml HF [13], followed by microscopic viewing.
Figure 9. Water lubricated diamond blade tile saw used to cut GLARE samples.

Figure 10. 240, 320, 400, and 600 grit SiC paper on the hand sander with sample holder to hold 10 mm by 10 mm sample.
Figure 11. Buchler water lubricated polishing machine with SiC sand paper (left) and soft nap polishing cloth (right).
Results and Analysis

Images obtained from the microscope camera provided important information about the material. The fiber orientation is rather interesting in GLARE. There are two or more different prepreg layers in each glass/epoxy layer. Their layup differs depending on the grade of GLARE. Fig. 13 on the following page shows the construction of GLARE 4A-4/3 0.3.
Figure 13. Microscopic image of GLARE 4A-4/3 0.3. Composite layers each consist of 3 unidirectional cross ply glass/epoxy prepreg layers (each 0.127 mm thick). All aluminum layers are 0.3 mm thick.

Fig. 13 depicts six unidirectional prepreg layers whose fibers are pointing out of the page. A test specimen subjected to tensile loading with fibers oriented in this direction is denoted as the “strong” specimen, whereas a specimen pulled in the transverse direction is the “weak” specimen. Only three prepreg layers are in the weak direction. The strong and weak directions of GLARE 4B were determined in the same manner, Fig. 14 (next page). However, GLARE 3-4/3 0.4 HSS has a symmetric cross section, Fig. 15 on the following page.
Figure 14. GLARE 4B cross section (40X magnification) with 4 prepreg layers pointing out of the page indicating its strong direction.

Figure 15. GLARE 3 cross section (40X magnification) with 3 prepreg layers pointing out of the page and 3 prepreg layers pointing in the direction perpendicular to it.

In the GLARE 3 configuration, equal amounts of prepreg layers pointing in the two directions should result in little preferential stiffness and strength. However, like all grades of GLARE, the rolling direction of the aluminum sheets may have an effect and should be investigated. Note that the direction of fibers in the prepreg layer closest to the outer aluminum layer is defined as the $0^\circ$ orientation, and by convention this is the aluminum rolling direction [3].
Microscopic images that follow were able to reveal the rolling direction of the aluminum 2024-T3 only sample. The direction in which the grains appear to be more elongated is the rolling direction. By rotating the sample, images clearly show the orientation of the elongated grains. It is apparent that the grains were elongated in the rolling direction.

**Figure 16.** Grains elongated in the horizontal direction indicate a horizontal rolling direction. Taken at 40X (left) and 100X (right).

**Figure 17.** Grains elongated in the vertical direction indicate a vertical rolling direction. Taken at 40X (left) and 100X (right).
Tensile Testing

Introduction

Tensile tests were performed on two (3.18 mm thick) aluminum 2024-T3 specimens (cut perpendicular to each other to differentiate them based upon rolling direction) and results were compared to GLARE mechanical properties.

GLARE test specimens were fabricated from the different grades of GLARE; GLARE 3-4/3-0.4 HSS, GLARE 4A-4/3-0.3 and GLARE 4B-3/2-0.4. Two tensile specimens were cut from each panel in directions perpendicular to each other. A total of 8 tensile specimens were pulled using a test system complete with data acquisition which captured position, load, and extensometer displacement. The force vs. displacement data was later converted to stress-strain data. Apparently strong and weak specimens for the aluminum alloy 2024-T3 and each GLARE panel were noted before testing, using prior information obtained from the microscopic investigation.

Equipment and Procedure

The previously mentioned diamond blade tile saw was used to cut two 25.4 mm wide by 254 mm long specimens out of each panel. Tabs made from aluminum alloy 6061-T651 (25.4 mm wide by 76.2 mm long) were attached to test specimens to provide effective gripping. These tabs had no bevel angle and were adhered onto the specimens using SilverTip MetlWeld epoxy adhesive by System Three Resins, Inc. (Auburn, WA). After excess adhesive was squeezed out of the material-tab interface, tabbed specimens were left to cure at room temperature for approximately 48 hr. To produce uniform
specimen widths and eliminate stress concentrations, they were wet sanded along their edges and then, lightly scrubbed lengthwise using Scotch-Brite (3M, St. Paul, MN) pads. This was done to make sure the test results were little affected by sample preparation. See ASTM D 3039/D 3039M - 08 for some suggestions on sample dimensions [14]. Fig. 18 depicts a typical GLARE sample and aluminum 2024-T3 sample prior to testing.

![Figure 18](image)

**Figure 18.** Fabricated tensile specimens made to approximately the same size for stress-strain comparisons. GLARE 4A (top) and aluminum alloy 2024-T3 (bottom).

An Instron (Norwood, MA) 8802 hydraulic test system was used to apply tensile force on the samples. It was equipped with a ±250 kN load cell, flat serrated face hydraulic grips, FastTrack™ 8800 digital control/acquisition and a ±12.7 mm strain gauge extensometer which was extended to a gauge length of 50.8 mm for testing. Ten load and strain readings were acquired per second. A relative ramp generator controlled the crosshead speed at 2 mm/min in accordance with ASTM D 3039/D 3039M – 08 [14]. The test system is shown in Fig. 19 on the following page.
Figure 19. Testing machine (left) and close up of specimen/extensometer (right). The specimen width is approximately 25.4 mm.

Results and Analysis

Eight tests were carried out to compare the stress-strain responses. Figs. 20-23 (pgs. 28 and 29) show the stress-strain curves for each grade of GLARE, as well as aluminum alloy 2024-T3. In these plots, the strong direction has more fibers aligned in the direction of loading, while the weaker direction has less fibers aligned in the direction of loading. The microscopic investigation conducted earlier determined the number of prepreg layers oriented most favorably to resist tensile loading. This is analogous to rope and cable loaded axially.
Figure 20. Stress-strain response of symmetric GLARE 3-4/3-0.4 tested along the aluminum rolling direction and transverse to it.

Figure 21. Stress-strain response of GLARE 4A-4/3-0.3 tested along its strong and weak directions.
Figure 22. Stress-strain response of GLARE 4B-3/2-0.4 tested along its strong and weak directions.

Figure 23. Stress-strain response of aluminum alloy 2024-T3 tested along its rolling (L) and transverse to rolling directions (LT).
Looking at the plots, the three different grades of GLARE exhibit very similar stress-strain characteristics. GLARE 4A and 4B clearly shows difference in ultimate strength, $\sigma_u$, between its strong and weak directions. This is because GLARE 4A has 6 out of 9 prepreg layers oriented in its strong direction, leaving only 3 prepreg layers oriented in its weak direction. GLARE 4B follows a similar pattern as it has 4 out of 6 prepreg layers oriented in its strong direction. However, GLARE 3’s symmetric configuration (equal amount of prepreg in each direction) results in nearly identical stress-strain responses in the two orientations, showing little preferential strength. Nevertheless, GLARE 3 seems to be slightly stronger in its aluminum rolling direction. Recall, the aluminum rolling direction in GLARE 3 is defined by the orientation of the outer prepreg layer. The direction of fibers closest to the outer aluminum layer is defined as the $0^\circ$ orientation, which is also the aluminum rolling direction [2].

The 0.2% offset yield strength, $\sigma_y$, and yield strain, $\varepsilon_y$, of GLARE 3 is higher than GLARE 4A and GLARE 4B. This is because GLARE 3 uses 7000 series aluminum alloys instead of aluminum alloy 2024-T3 like the other two grades. It is labeled HSS GLARE for this reason [15]. The 7000 series aluminum alloys typically have higher yield strengths and strains than the 2000 series alloys.

A comparison is now made between the yield strains of GLARE 4A, GLARE 4B and aluminum alloy 2024-T3, Fig. 24 on the following page. GLARE 4A and 4B show 0.2% offset yield strains of up to $\sim0.70\%$ in both strong and weak directions, which is similar to aluminum alloy 2024-T3 in the absence of residual stresses. The aluminum alloy 2023-T3 only specimen yielded at 0.6% strain in the L-direction (longitudinal) and LT-direction (transverse). These values were confirmed using MIL-HDBK-5J yield
strains for aluminum alloy 2024-T3 (~0.50% strain in the L-direction and ~0.40% strain in the LT-direction) [16]. Any slight differences between yielding of GLARE 4A and 4B in strong and weak directions may be a consequence of rolling direction. The rolling direction for GLARE 4A and 4B must be determined through metallography. Observe that GLARE 3 has a 0.2% offset yield strain of ~0.85%, but is not included in the plot because it uses 7000 series aluminum alloys and a comparison of GLARE 4A and 4B to 2000 series aluminum alloys was desired. Note that offset yield strengths for all tested materials are higher than proportional limit yield strains.

![Figure 24](image_url)

**Figure 24.** Yield strain comparison between GLARE 4A, GLARE 4B, and aluminum alloy 2024-T3.

Two stages can be observed in the laminate stress-strain curves. Stage I (first part) is primarily elastic deformation of the metal and prepreg, while Stage II (second
part) indicates primarily plastic deformation of the metal and elastic deformation of the prepreg, with some possible matrix damage and fiber fractures. If unloaded, these stress-strain curves would exhibit a line nearly parallel to the initial slope before yield. This results in similar Young’s moduli values for Stage I and II.

Tensile tests provided valuable information on the mechanical properties of each material. This is summarized for comparison in Table 5. Density, \( \rho \) for the different grades were obtained through mass and volume measurement of the panels.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Panel Thickness (mm)</th>
<th>Al Layers</th>
<th>Glass/ Epoxy Layers</th>
<th>Prepreg Orientations In Glass/ Epoxy Layers</th>
<th>( E ) (GPa)</th>
<th>( \sigma_y ) (MPa)</th>
<th>( \sigma_u ) (MPa)</th>
<th>( \rho ) (g/cm(^3))</th>
<th>Density Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>GLARE 3-4/3-0.4</td>
<td>2.43</td>
<td>4</td>
<td>3</td>
<td>0/90 90/0 90/0</td>
<td>~55</td>
<td>~365</td>
<td>690</td>
<td>2.48</td>
<td>0.89</td>
</tr>
<tr>
<td>HSS</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GLARE 4A-4/3-0.3</td>
<td>2.45</td>
<td>4</td>
<td>3</td>
<td>0/90/0 0/90/0</td>
<td>~52</td>
<td>~270</td>
<td>880</td>
<td>2.30</td>
<td>0.83</td>
</tr>
<tr>
<td>GLARE 4B-3/2-0.4</td>
<td>2.06</td>
<td>3</td>
<td>2</td>
<td>90/0/90/90/90/90</td>
<td>~55</td>
<td>~270</td>
<td>801</td>
<td>2.45</td>
<td>0.88</td>
</tr>
<tr>
<td>Al 2024-T3 (L)</td>
<td>3.23</td>
<td>-</td>
<td>-</td>
<td></td>
<td>~72</td>
<td>~365</td>
<td>484</td>
<td>2.78</td>
<td>1.00</td>
</tr>
<tr>
<td>Al 2024-T3 (LT)</td>
<td>3.23</td>
<td>-</td>
<td>-</td>
<td></td>
<td>~71</td>
<td>~310</td>
<td>468</td>
<td>2.78</td>
<td>1.00</td>
</tr>
</tbody>
</table>

Note: The 0° direction is the aluminum rolling direction while 90° is transverse to it. The Young’s modulus, yield strength (0.2% offset) and ultimate strength values for each GLARE type are the highest measured values of the two different test directions for each grade of GLARE (e.g., strong). Density ratio is that of GLARE to aluminum alloy 2024 (~2.78 g/cm\(^3\)).

From the manufacturer data (Table 3 on pg. 16) and experimental results, it can be seen that GLARE has greater ultimate strengths compared to aluminum alloy 2024-T3,
while exhibiting a lower density. However, it is not as stiff as conventional aircraft grade aluminum as evidenced by the smaller Young’s modulus values.

Another interesting observation is the failure characteristics in GLARE, where ultimate strength defines failure. Specimens pulled in the strong direction experienced more delamination when compared to specimens pulled in the weak direction. This was true for all grades of GLARE that were tested. The load once supported by a failed constituent is transferred through crack bridging to intact constituent(s). This transfer creates additional shear stress between adjacent constituents which can lead to delaminations [9]. Fig. 25 on the following page shows the delamination experienced by GLARE 4A-4/3-0.3 when pulled in its strong direction. On the other hand, specimens tested in the direction with fewer fibers (weak direction) showed more fiber breaks, Fig. 26 on the following page. Differences in the way strong and weak specimens failed are shown in Fig. 27 (pg. 35). Similar failure characteristics were observed in GLARE 4B-3/2-0.4. It appears that when loaded in the weak direction, fewer fibers are present to support the tensile load, resulting in more fiber breaks and complete separation of the test specimen.
Figure 25. Delaminations experienced by GLARE 4A-4/3-0.3 tested along its strong direction.

Figure 26. Specimens tested in the direction with less fibers (weak) show more fiber breaks than delamination.
Rule of Mixtures

The rule of mixtures used here is to estimate Young’s modulus, 0.2% offset yield strength and strength at failure. The calculations presented in this section are for GLARE 4B-3/2-0.4 in its strong direction because the new FML fabricated uses a 3/2 construction. Note that for this grade of GLARE, the strong direction is transverse to the aluminum rolling direction. The Young’s modulus of the laminate is a combination of the Young’s modulus of the glass/epoxy layers and the Young’s modulus of metal layers. The Young’s modulus of the glass/epoxy layer is a contribution of the glass fibers and epoxy matrix. These relationships are illustrated by the equations below.

\[ E_{lam} = V_c E_c + V_{met} E_{met} \]  \hspace{1cm} (1)

\[ E_c = V_f E_f + V_m E_m \]  \hspace{1cm} (2)
Where:

\[ E_{\text{lam}} = V_c (E_f + V_m E_m) + V_{\text{met}} E_{\text{met}} \]  \hspace{1cm} (3)


- \( E_{\text{lam}} \) - Young’s modulus of laminate
- \( V_c \) - Volume fraction of glass/epoxy composite
- \( E_c \) - Young’s modulus of glass/epoxy composite
- \( V_{\text{met}} \) - Volume fraction of metal
- \( E_{\text{met}} \) - Young’s modulus of metal
- \( V_f \) - Volume fraction of fibers
- \( E_f \) - Young’s modulus of fibers
- \( V_m \) - Volume fraction of matrix
- \( E_m \) - Young’s modulus of matrix

\( E_f \) is 88 GPa (S-glass) and \( E_m \) is 1.7 GPa taken from Tables 1 and 2 (pg. 13) respectively. \( E_{\text{met}} \) is determined to be 71 GPa (from aluminum alloy 2024-T3 LT direction stress-strain curve) and \( V_{\text{met}} \) is calculated by measuring the thickness of the metal layers relative to the total laminate thickness, Fig. 14 (pg. 23). This approximate ratio is 0.59 and is calculated from Equation 4.

\[ V_{\text{met}} = \frac{n_{al} t_{al}}{n_{al} t_{al} + n_c t_c} \]  \hspace{1cm} (4)

Where:

- \( n_{al} \) - Number of aluminum layers
- \( t_{al} \) - Thickness of each aluminum layer
\( n_c \) - Number of glass/epoxy layers

\( t_c \) - Thickness of each glass/epoxy layer

Glass/epoxy volume fraction is

\[
V_c = 1 - V_{net} = 1 - 0.59 = 0.41
\]  \hspace{1cm} (5)

The fiber volume fraction in any one GLARE prepreg or glass/epoxy layer is 0.59 [3]. Referring to Fig. 14 (pg. 23), there are 4 out of 6 prepreg layers, or 2/3 oriented in the strong direction, in this case the load bearing direction. The true fiber volume fraction, \( V_{true} \), of fibers that bear the load in any prepreg or glass/epoxy layer is calculated by multiplying the load bearing fraction by the composite volume fraction.

\[
V_{true} = \frac{2}{3} V_f = \frac{2}{3} (0.59) = 0.39
\]  \hspace{1cm} (6)

Similarly, \( V_m \) for any prepreg or glass/epoxy layer can be estimated using

\[
V_m = 1 - V_f = 1 - 0.59 = 0.41
\]  \hspace{1cm} (7)

Substituting known values into Equation 3,
The estimated $E_{\text{lamine}}$ is compared to the experimental Young’s modulus from stress-strain data (55 GPa). This yields a 1.8% difference. Similar rule of mixtures can be used to estimate stress at various points on the stress-strain curve. Equation 9 is used to calculate laminate stress at yield, while Equation 10 is used to estimate stress at failure.

Isostrain conditions (all constituents strain the same amount) and only elastic behavior of both fiber and matrix are assumed.

\[
E_{\text{lamine}} = V_e (V_{\text{true}} E_f + V_m E_m) + V_{\text{met}} E_{\text{met}}
\]

\[
= 0.41[(0.39)(88 \text{ GPa}) + (0.41)(1.7 \text{ GPa})] + 0.59(71 \text{ GPa}) \quad (8)
\]

\[
= 56 \text{ GPa}
\]

\[
\sigma_{\text{lam}} = V_e (V_{\text{true}} \epsilon_f E_f + V_m \epsilon_m E_m) + V_{\text{met}} \sigma_{\text{ymet}} \quad (9)
\]

Where:

- $\sigma_{\text{lam}}$ - Yield stress of laminate
- $\epsilon_f$ - Fiber strain
- $\epsilon_m$ - Matrix strain
- $\sigma_{\text{ymet}}$ - Yield stress of metal

\[
\sigma_{\text{lam fail}} = V_e (V_{\text{true}} \epsilon_f E_f + V_m \epsilon_m E_m) + V_{\text{met}} \sigma_{\text{met fail}} \quad (10)
\]

Where:

- $\sigma_{\text{lam fail}}$ - Failure stress of laminate
- $\sigma_{\text{met fail}}$ - Failure stress of metal
Estimated 0.2% offset yield stress and stress at failure are calculated for GLARE 4B-3/2-0.4. The 0.2% yield strain of GLARE 4B-3/2-0.4 is 0.70% (from test results) and this value is incorporated in the estimation of laminate yield stress. The 0.2% yield stress and stress at failure used for aluminum 2024-T3 LT direction are 324 MPa and 416 MPa respectively. The aluminum alloy yield stress is the stress at 0.70% strain, while the ultimate stress is the stress at 4.46% strain (when GLARE 4B fails).

\[
\sigma_{\text{lam}} = V_c (V_{frue} \varepsilon_f E_f + V_m \varepsilon_m E_m) + V_m \sigma_{\text{yfail}}
\]

\[
= 0.41[(0.39)(0.0070)(88 \text{ GPa}) + (0.41)(0.0070)(1.7 \text{ GPa})] + 0.59(324 \text{ MPa})
\]

\[
= 291 \text{ MPa}
\]

\[
\sigma_{\text{lam fail}} = V_c (V_{frue} \varepsilon_f E_f + V_m \varepsilon_m E_m) + V_m \sigma_{\text{yfail}}
\]

\[
= 0.41[(0.39)(0.0446)(88 \text{ GPa}) + (0.41)(0.0446)(1.7 \text{ GPa})] + 0.59(416 \text{ MPa})
\]

\[
= 886 \text{ MPa}
\]

The experimental stress-strain curve for this laminate gives a 0.2% offset yield stress of 270 MPa and stress at failure of 801 MPa. This gives a 7.5% and 10.1% difference respectively. This estimation for stress at failure is higher than the actual experimental value. This is commonly observed when attempting to predict failure strengths for composites along with significant observed experimental scatter. For complex composite structures, it is difficult to predict mechanical properties. The small difference experienced in this case is encouraging.
Introduction

While GLARE has pronounced mechanical properties, its manufacturing process is regarded as costly and quite complex [17]. Industrially, the metal sheets are first cut to appropriate dimensions, degreased, PAA surface treated and lastly primed with BR-127 primer [17]. The rolled prepreg is cut and removed from its backing before layup. Next, the metal and prepreg layers are laid up on a pre-shaped mold, vacuum bagged and cured in an autoclave at high temperature and pressure (120°C, 600-1100 kPa) [17]. To create larger panels, splicing and reinforcements are necessary.

A pie chart depicts the manufacturing costs of GLARE, Fig. 28 (pg. 41). It is an estimation of cost distribution to manufacture a GLARE 3-3/2-0.4 panel with 1 m² area. The aluminum sheets make up 6% of total costs, 19% comes from the FM-94 prepreg, while the other 75% is incurred by manufacturing processes [18]. For many successful high volume production processes, 99% or more of the cost comes from base material cost.
A small scale fabrication process of FMLs was performed at ERAU to better understand GLARE manufacturing procedures and possibly simplify them. Different surface pretreatments on aluminum alloy 2024-T3 sheets were investigated to study their differences, advantages, and disadvantages. The use of a primer was eliminated in the process. The glass/epoxy layers in these restructured FMLs consisted of S-2 glass and an epoxy adhesive, instead of the normal FM-94 prepreg. The main purpose was to reduce material and fabrication cost as well as simplify. Cost estimates obtained (not including shipping or dry ice packaging) from Cytec Industries Inc. for small quantities of the GLARE primer and prepreg are shown in Table 6. Note that the mat prepreg is different from the knit because the mat prepreg backing is made up of randomly oriented fibers [19]. Both are loosely held together by a binder.

<table>
<thead>
<tr>
<th>Item</th>
<th>Quantity</th>
<th>Price</th>
</tr>
</thead>
<tbody>
<tr>
<td>BR-127 primer</td>
<td>1 quart</td>
<td>$172.21</td>
</tr>
<tr>
<td>FM-94 K (knit)</td>
<td>50 ft²</td>
<td>$704.70</td>
</tr>
<tr>
<td>FM-94-1M (mat)</td>
<td>50 ft²</td>
<td>$545.40</td>
</tr>
</tbody>
</table>
The elimination of an autoclave would contribute to great cost reductions. Also, the pressure during cure can be reduced by using epoxy adhesives with lower viscosity [17]. For the FMLs fabricated at ERAU, nearly 760 mm Hg pressure (atmosphere) was used while the laminate cured during vacuum bagging at room temperature. A rotary vane pump capable of $10^{-3}$ Torr or better created the vacuum. These are just two advantages of the particular epoxy that was used. However, methods and final results are likely to vary with different epoxies, an aspect that should be explored in the future.

**Aluminum Surface Pretreatment**

The surface quality of the aluminum layers in FMLs affect the occurrence of debonding when load is applied [20], making the surface pretreatment process a very crucial part of FML fabrication. The effects of three different methods are presented in this thesis; sandblasting, phosphoric acid anodizing (PAA) and AC-130 Sol-Gel pretreatment by 3M (St. Paul, MN). A number of shear tests were carried out to help determine the most feasible surface pretreatment for the fabrication of this set of FMLs.

**Sandblasting**

Sandblasting, or also known as grit blasting, is a dry mechanical abrasion process that is carried out on a surface. A suitable abrasive is blasted with compressed air through a nozzle at high pressure. Common abrasives include aluminum oxide, silicon carbide, ceramic grit, steel grit, glass beads and others. They vary in shape, density, hardness and particle size depending on the specific application [21]. When using
adhesives, a sandblasted surface creates a mechanical interlocking with the adhesive, providing a better bond than a regular smoother surface. Mechanical abrasion roughness also provides a larger effective surface area [22].

To study the effects of sandblasting, small (25.4 mm by 50.8 mm by 0.51 mm thick) aluminum alloy 2024-T3 sheets were made into shear test samples, Fig. 29. Sheets were blasted with sharp edge GA #75 Medium Fine glass abrasive with 5.5-6 Mohs hardness (Tacoma Company, Mead, WA) using 60 psi pressure and a commercial blast cabinet. Sheets were first degreased with Naturalizer Multi-Purpose Remover (Chemsearch, Irving, TX), sandblasted, rinsed with acetone, air dried and then a unidirectional Cytec 381 prepreg was laid up between the sheets. The samples were cured in a laboratory oven (Grieve Corporation, Round Lake, IL) for 2 hours at 80°C and pulled in tension using a Tinius Olsen Lo-Torq Universal Testing Machine (Horsham, PA).

![Figure 29. Small 25.4 mm by 50.8 mm aluminum alloy 2024-T3 sheets and unidirectional prepreg used to make the shear samples shown (after it was pulled in tension).](image)

When tested, the smoother aluminum sheets failed before even enough load was applied to fully grip the sample. The sandblasted samples on the other hand sustained loads up to 2280 N. Although ASTM standards were not employed, the tests showed that
sandblasting helps with bonding. The microscopic image in Fig. 30 (next page) shows the surface of a sandblasted aluminum alloy 2024-T3 sheet compared to the same type of sheet without any mechanical abrasion.

![Image](https://example.com/image1.png) ![Image](https://example.com/image2.png)

**Figure 30.** Sandblasted aluminum 2024-T3 surface (left) compared to similar as received surface (right) taken at 40X magnification.

*Phosphoric Acid Anodizing*

PAA anodizing has been used by the aerospace industry since the 1970’s, replacing the not so environmentally friendly chromic acid anodizing and etching process. Anodizing is carried out to prepare aluminum alloys for stronger bonds at the adhesive/aluminum interface. With anodizing, special treatments are used to grow relatively thick aluminum oxide layers on aluminum surfaces. The aluminum oxide layer provides keying or mechanical interlock with the adhesive. Tests carried out and reported in United States Patent 4,085,012 claim that surfaces prepared with PAA exhibit primarily cohesive failure (failure within the adhesive layer) as opposed to adhesive failure (failure at the metal-adhesive interface) [23]. The flow chart in Fig. 31 on the following page is a summary of the PAA process outlined in the patent.
Figure 31. PAA process eliminates the need for etching and use of more harmful acids.

The PAA process was carried out at ERAU to grow aluminum oxide layers which were then viewed under the metallurgical microscope. Aluminum alloy 2024-T3 sheets (25.4 mm by 50.8 mm by 0.51 mm to 1 mm thick) were degreased with the Chemsearch degreaser mentioned earlier, rinsed with hot tap water and PAA anodized. For PAA, the electrolyte was phosphoric acid ($\text{H}_3\text{PO}_4$), the cathode used was 304 stainless steel, while the anode was the aluminum sheet. Aluminum wires were used for electrical connections with voltage monitored using a digital multimeter. Black electrical tape prevented undesired electrical contact (see Figs. 32 and 33 on the following page). A Hewlett-Packard (Palo Alto, CA) 6284A DC Power Supply was used.

The experiment setup in Fig. 32 includes a Cole-Parmer (Vernon Hills, IL) 8853 ultrasonic cleaner, which accommodates heating of acids and vibration capabilities. However, since the experiments in ERAU only required room temperature PAA process,
it was used as a holder for the Pyrex beaker in which the anode and cathode were placed. Temperature was constantly monitored using an Omega Engineering Inc. (Stamford, Connecticut) type J thermocouple connected to a thermocouple readout meter.

Figure 32. PAA setup with voltage and temperature measurement devices for constant monitoring.

Figure 33. 304 stainless steel cathode, aluminum sheet as anode, H₃PO₄ as electrolyte connected by aluminum wires in a 1000 ml Pyrex beaker.
Initially, different anodizing durations were tried (30 min, 1 hr and 7 hr) to compare the amount of oxide grown. This first setup used a DC voltage of 5V, ran at room temperature using 17% H₃PO₄ [23]. The H₃PO₄ from DudaDiesel (Madison, AL) had an initial concentration of 85%. Equation 13 was used to determine how much distilled water was needed to dilute the acid to 17% concentration. An 85% acid concentration indicates 85 ml of pure acid in a 100 ml solution. Solving for $X_{H_2O}$ below gives the volume of distilled water required per 100 ml acid for dilution to produce 17% H₃PO₄.

$$\frac{85 \text{ ml}}{100 \text{ ml} + X_{H_2O}} = 0.17 \tag{13}$$

From microscopic observations, a longer PAA process did grow a denser and thicker oxide layer. However, thicker oxide layers may not be favorable for bonding with adhesive [23]. Referring to test data from the patent mentioned earlier, the final PAA process used the same setup but ran for 30 minutes. The metallurgical microscope was used to provide the image presented on the next page.
Figure 34. Aluminum oxide layer as a result of PAA taken at 40X magnification.

AC-130 Sol-Gel

AC-130 Sol-Gel is a product licensed by Boeing (Chicago, IL) to Advanced Chemistry & Technology, Inc., now part of 3M. Like PAA, AC-130 Sol-Gel is also used to provide better bonding characteristics. Sol-Gel stands for solution gelation, which denotes what happens when soluble metals form metal hydroxide with the application of this aqueous solution [24]. One side of the AC-130 system is bonded to the oxide layer of the metal, while the other is bonded to the epoxy adhesive system, Fig. 35 on the following page [25].
This surface pretreatment is used not only for aluminum alloys, but for improved adhesion of steel, titanium, nickel and composite material [26]. This provides a more versatile use of this surface treatment, as compared to PAA for example. It is usually followed by the application of a primer or epoxy adhesive onto the pretreated metal surface. Sometimes both primer and epoxy are used. This product comes in 2 and 4 part kits with 4 chemical components in each kit; water, $\gamma$-glycidoxypropyltrimethoxysilane (GTMS), zirconium n-propoxide (TPOZ) and glacial acetic acid (GAA). These chemicals are each packed separately in the 4 component kit. In the 2 component kit, Part A consists of water, TPOZ, GAA and a surfactant (added to reduce surface tension in fluids), while Part B consist of GTMS [24].

Here, the focus remains on use of this product. The following application procedure was used with reference to the 3M application guide [26] although slightly...
different (3M uses Al₂O₃ grit). The purpose was to coat the aluminum alloy 2024-T3 sheets (varying thicknesses depending on the tests) with AC-130 Sol-Gel before application of epoxy adhesive. Note that induction time is the period before the solution becomes active after mixing all the components. Pot life is the duration this preparation can be used after mixing.

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Aluminum sheets machined and sandblasted (60 psi, glass grit)</td>
</tr>
<tr>
<td>2</td>
<td>Washed with tap water and air dried</td>
</tr>
<tr>
<td>3</td>
<td>Rinsed with acetone</td>
</tr>
<tr>
<td>4</td>
<td>Mixed the AC-130 parts according to instructions (induction time 30 min, pot life 10 hr)</td>
</tr>
<tr>
<td>5</td>
<td>Keep spraying to keep the surface wet for at least 1 min</td>
</tr>
<tr>
<td>6</td>
<td>Let the surface drain for 5-10 min</td>
</tr>
<tr>
<td>7</td>
<td>Used spray bottle to spray aluminum within 8 hr from sandblasting</td>
</tr>
<tr>
<td>8</td>
<td>Air dried for a minimum of 60 min</td>
</tr>
<tr>
<td>9</td>
<td>Applied epoxy adhesive within 24 hours</td>
</tr>
</tbody>
</table>

**Figure 36.** AC-130 Sol-Gel pretreatment process carried out at ERAU on aluminum 2024-T3 sheets.

### Lap shear adhesion

**Preliminary Testing and Results**

Lap shear tests are commonly used for determining the bond strength of adhesives and effectiveness of surface treatments. In this case, the type of adhesive used was treated as a constant while surface treatment was the variable. The most favorable
A method for preparing the aluminum surface was sought to ensure good bonding characteristics for the restructured FMLs. Another series of lap shear tests were carried out, but this time to compare all three different surface pretreatments: sandblasting, sandblasting + PAA, sandblasting + AC-130 Sol-Gel.

Aluminum alloy 2024-T3 sheet specimens (24.5 mm wide by 101.6 mm long by 0.51 mm thick) were cut and sanded along their edges. Care was taken to ensure sample flatness after cutting. All sheets were sandblasted as before to provide enhanced bonding characteristics. These sheets were surface pretreated using the different methods discussed earlier (Table 7) and bonded to each other with Polypoxy epoxy by System Three Resins, Inc. (Auburn, WA), called Poly Epox purchased from Aircraft Spruce & Specialty Co. (Peachtree City, GA). The epoxy will be discussed further in the epoxy adhesive section. The lap shear overlay was 25.4 mm by 25.4 mm in accordance with ASTM D 5868 - 01 [27]. A 3:1 resin to hardener ratio by weight was used. After mixing, a paint brush was used to apply a thin layer to the surfaces, shear samples were laid up and then vacuum bagged for 6 hours to ensure a good bond. Details of the vacuum bagging methods are described later. Next, samples were removed from vacuum and oven-cured for 2 hours at 60°C [28] (see Fig. 37 on the next page).

<table>
<thead>
<tr>
<th>Method</th>
<th>Shear samples</th>
<th>Process details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sandblast</td>
<td>3</td>
<td>Uniform sandblasting over surface using 60 psi.</td>
</tr>
<tr>
<td>Sandblast + PAA</td>
<td>3</td>
<td>Anodizing occurred at 5 V, room temperature, for 30 min, using 17% H₃PO₄.</td>
</tr>
<tr>
<td>Sandblast + AC-130</td>
<td>3</td>
<td>Sprayed and kept wet for 5 min and dried overnight.</td>
</tr>
</tbody>
</table>
A total of 9 shear samples were pulled in tension using the previously described Instron 8802 test equipment. As before, 10 values of load and displacement were recorded each second. The crosshead speed was 2 mm/min. Although the recommended ASTM rate for shear testing was 13 mm/min, testing here was performed more slowly for consistency with tensile testing. Load-crosshead position data was converted to stress-strain data, Fig. 38 on the following page.

Although the tests were performed to determine the shear strength of the adhesive bond, the aluminum was the weaker part of the system and yielded prior to the adhesive bond failing. While not providing an accurate shear strength value, it still provided a surface pretreatment method comparison for this particular epoxy adhesive. Sample strain is based upon the distance between grips which was 82.5 mm.
For the most part, sandblasting + AC-130 Sol-Gel sustained higher shear strengths and strains. Due to unforeseen variables such as observed moisture trapped in the shear overlay, values obtained from the stress-strain plot can indicate an anomaly (a very low value for one of the AC-130 samples). The reason for this one is unclear. Note that the metal yielding might have contributed to lower lap shear adhesion strengths (compared to typical lap shear adhesion data). For example, Table 8 (next page) shows shear strengths of typical epoxy adhesives from lap shear tests [29]. The next series of tests used thicker aluminum sheets to eliminate metal yielding prior to bond failure. This should provide more accurate shear strength values.
Table 8. Approximate shear strengths of common epoxy adhesives from lap shear tests.

<table>
<thead>
<tr>
<th>Category</th>
<th>Approximate shear strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>High</td>
<td>17.2 - 27.6 MPa</td>
</tr>
<tr>
<td>Average</td>
<td>8.3 - 13.1 MPa</td>
</tr>
<tr>
<td>Low</td>
<td>&lt;5.5 MPa</td>
</tr>
</tbody>
</table>

Final Testing and Results

Since the width of samples (25.4 mm) needed to be maintained according to ASTM D 5868 – 01 [27], thicker and also Alclad aluminum alloy 2024-T3 sheets (these typically have 5-10% thickness of pure Al cladding per side) that were 2.54 mm (0.10 in) thick were used for this series of tests. The thicker sheets of aluminum were selected to prevent yielding. Examining Fig. 38 (pg. 53), sandblasting + AC-130 Sol-Gel provided better results. As a consequence, the aluminum sheets were sandblasted and sprayed with AC-130 Sol-Gel. Polypoxy was again used as the epoxy adhesive for shear overlap and this time, also to adhere tabs of the same sheet (25.4 mm wide by 50.8 mm long by 2.54 mm thick) onto the specimens, Fig. 39, which helped ensure uniform shear sample thickness. A total of 4 shear test samples were fabricated. As before, shear lap adhesion tests were carried out, Fig. 40 (next page).

Figure 39. Test specimen fabricated with sandblasted + AC-130 pretreated Alclad aluminum alloy 2024-T3.
Shear lap adhesion tests for the sandblasted + AC-130 pretreated Alclad aluminum alloy 2024-T3 specimens. Here, the ±12.7 mm strain gauge extensometer was adhered to the shear overlay portion in Test 3 and Test 4 for strain measurement.

Observing Fig. 40, shear strength values are consistent with the exception of test 4. No explanation for this anomaly can be provided. Also, these shear strengths are very close to 12 MPa, putting them in the upper average range of Table 8 (pg. 54). This indicates that the epoxy adhesive is biased towards the higher end of typical adhesive shear strengths. This makes it a potentially suitable epoxy adhesive for FML fabrication.

Additionally, based on microscopic observation, the specimens experienced predominantly cohesive failure. Cohesive failure is when failure occurs within the adhesive leaving it bonded to both metal surfaces. On the other hand, adhesive failure is when failure occurs at the adhesive-metal interface. This is not the more desirable
occurrence. Examination of the test specimens lap shear overlay (Fig. 41), after testing revealed adhesive evenly distributed on the two surfaces and adhering to them. This is an indication of cohesive failure, rather than adhesive failure [29].

![Figure 41. Cohesive failure of lap shear specimen viewed under stereozoom microscope (shear lap overlay area) at 7X magnification.](image)

### Epoxy Adhesive

Within a composite material, the purpose of a matrix is to bind together fibers, protect them from the environment, transfer load to and in between them, provide interlaminar shear strength, help with load distribution among fibers, maintain fiber orientation and contribute to damage resistance [30]. Matrices with high toughness and high ultimate strain can extend the fatigue life of a composite material [31].

Epoxy adhesives are most commonly used for aerospace applications due to good mechanical properties, handling capabilities and affordable costs. Polyester and vinyl
ester resins are not widely used in the aerospace sector because of their relatively lower mechanical properties and tendency to shrink during cure [30].

Polyoxy was used to fabricate restructured the FMLs. It was purchased for $41.50 per quart kit. It consists of two cure phases; the first is a thin film setting cure that occurs at room temperature after 6 hr, while the second is a post cure attained by subsequent heating to 60°C (recommended temperature) for 2 hr. Aircraft Spruce & Specialty Co. data indicates that epoxy exposed to post curing exhibit better mechanical properties than those without post cure, Table 9. It is said that softening the epoxy during post cure enhances crosslinking of molecules, which improves properties [28].

<table>
<thead>
<tr>
<th>Mechanical properties</th>
<th>With post cure</th>
<th>Without post cure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>66.2 MPa</td>
<td>60.7 MPa</td>
</tr>
<tr>
<td>Strain at failure</td>
<td>7.5%</td>
<td>3.6%</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>3240 MPa</td>
<td>3172 MPa</td>
</tr>
<tr>
<td>Compressive strength</td>
<td>220.6 MPa</td>
<td>227.5 MPa</td>
</tr>
</tbody>
</table>

After the hardener and resin are mixed at room temperature, the pot life of this epoxy is 105 minutes. To determine the density, a mold cup was used to cure some of this epoxy (room temperature for one day and later in the oven at 60°C for 2 hr). The calculated density of this epoxy is 1.15 g/cm³ using mass and volume measurements.

**S-2 glass**

Initially, GLARE was manufactured using S-glass for its glass/epoxy fiber component. However, due to recent glass fiber advancements, an improved version
known as S-2 glass is used in the FM-94 prepreg from which current versions of GLARE are made. This glass is mainly composed of SiO₂, Al₂O₃ and MgO [33]. S-2 glass is known as high strength fiberglass because it has better mechanical properties compared to other types; E-glass, A-glass and C-glass. Table 10 lists some properties of S-2 glass. The E, A, and C versions are also fiberglass but with low to medium strength range. Even so, these types of fiberglass cost less than S-2 glass, making them suitable for less stress resisting applications.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber diameter</td>
<td>5-9 μm</td>
</tr>
<tr>
<td>Strength</td>
<td>4585 MPa</td>
</tr>
<tr>
<td>Young’s modulus</td>
<td>86-90 GPa</td>
</tr>
<tr>
<td>Strain at failure</td>
<td>5.4-5.8%</td>
</tr>
</tbody>
</table>

Table 10. Mechanical properties of unidirectional S-2 glass fibers [33].

To fabricate restructured FMLs at ERAU, plain weave and satin weave S-2 glass were used instead of the conventional FM-94 prepreg (unidirectional). The purpose of this was to create simpler symmetric laminates, without having to lay up fibers and/or unidirectional prepregs in different orientations. This also provided potentially lower costs. One FML panel used plain weave S-2 glass, while the other was fabricated with satin weave S-2 glass.

Important terminologies when discussing woven fabrics are yarn, warp and fill. A yarn refers to a twisted strand of fiberglass filaments. A warp yarn is a lengthwise yarn in a weave, while a fill (or weft) yarn is crosswise to the warp yarn [19]. The plain weave fiberglass used at ERAU had twisted filaments (yarns). Both are shown in Figs. 42 and 43 (next page). The plain weave has a fiber weave resulting in an over one under
one configuration, while the satin weave has a fiber weave resulting in an over one under more than one (minimum of four) configuration [19]. In this particular case, the satin weave S-2 glass was an 8H (eight harness) indicating fibers going over one under seven.

Figure 42. Plain weave S-2 glass with twisted filaments (18 by 18 per in) used to fabricate FML at ERAU.

Figure 43. Satin weave S-2 glass with straight filaments (57 by 57 per in) used to fabricate FML at ERAU.

Plain weave S-2 glass is more common in surfboard and recreational applications, while satin weave S-2 glass is normally used for higher strength applications. This
“surfboard S-2 glass” is most often designated as 4533 S-2 glass (some suppliers list as 6533) while 8H satin weave S-2 glass is designated as 6781 S-2 glass. These two types of cloth were obtained from Illstreet Composites (Charleston, SC). The 6 oz plain weave S-2 glass cost $5.99 per yard for the 27 in width, while 8.9 oz satin weave S-2 glass was $10.59 per yard for the 38 in width. For style 4533, the warp and fill count are listed as 18 by 18 (e.g., yarn per in) while for style 6781 these are listed as either 57 by 54 or 57 by 57 depending on the source. The measured thickness for the plain weave S-2 glass was 0.23 mm, while the satin weave fiberglass was 0.21 mm thick. The densities were 0.72 g/cm$^3$ and 1.12 g/cm$^3$ respectively (using mass and volume measurements).

Glass fabrics usually have finishes applied to them during manufacture that promote bonding to the adhesive. These finishes are also known as bonding/coupling agents [34]. Most likely, the S-2 glass used here had a Volan® finish (DuPont Specialty Chemicals, Wilmington, DE) because of a slight green tint observed after cure. Information about the exact finish could not be obtained from the supplier.

**Panel Fabrication**

The restructured FMLs fabricated at ERAU used a 3/2 laminate configuration. This consisted of 3 aluminum alloy 2024-T3 layers with 2 composite layers sandwiched in between. Bare 2024-T3 aluminum alloy sheets (0.020 in or 0.51 mm thick) were used. In the future, Alclad aluminum sheets could be ideal to help inhibit corrosion. Here, the fabrication process did not use a corrosion inhibiting primer for potential cost savings and simplicity.
Each composite layer in GLARE 4B-3/2-0.4 is made up from 3 0.127 mm thick prepregs for a total thickness of 0.381 mm. Since, the measured thickness for plain and satin weave S-2 glass are 0.23 mm and 0.21 mm respectively, two layers of S-2 glass were used for each composite layer of the restructured FMLs. This configuration was selected to try and achieve composite layer thicknesses that are close to GLARE for mechanical property comparisons.

First, approximately 150 mm by 260 mm aluminum 2024-T3 sheets were cut using a band saw and their edges were sanded to smooth them. Sheets were then sandblast + AC-130 Sol-Gel surface pretreated as before. S-2 glass cloths slightly larger than the aluminum sheets were cut for layup within the aluminum alloy sheets. Yarns were gently manipulated to ensure straight and aligned edges for layup.

The first aluminum layer was placed onto a flat, thick, waxed aluminum mold covered by a porous Teflon release film. A foam paint brush was then used to apply a thin layer of mixed Polypoxy onto the sheet, Fig. 44 (next page). While still wet, the S-2 glass cloth was placed on top of the metal sheet, with its edges properly aligned. The paint brush was then used to dab (dabbing helps keep the fiberglass cloth in place as opposed to brush stroking) the fiberglass layer with just enough epoxy to wet the entire cloth. Excess epoxy was then carefully removed using a squeegee as shown in Fig. 45 (pg. 63). Another layer of fiberglass was then laid up in a similar fashion to complete one composite layer. Then, another aluminum layer was added, followed by another complete composite layer and lastly a final aluminum layer. These procedures were carried out for both plain and satin weave reinforced FMLs.
Glass/epoxy panels which consisted of only two layers of S-2 glass and epoxy adhesive were also made in a similar fashion, Fig. 46 (pg. 63). FML and glass/epoxy layups were covered with the porous Teflon release film, a breather bleeder ply and then vacuum bagging film, which was sealed to the mold with resistant sealant tape (supplies were obtained from Aircraft Spruce & Specialty Co.). A vacuum of approximately 1 atm (760 mm Hg) was used to create pressure on the panels overnight (6 hr minimum is recommended for this particular epoxy) to complete the first epoxy cure phase. Panels were then removed from the bag/mold and then placed in the oven for post curing at 60°C for 2 hours, then cooled prior to final cutting and machining.

Figure 44. Applying a thin layer of epoxy adhesive onto the first aluminum layer of the laminate.
Figure 45. Removing excess epoxy with the help of a squeegee.

Figure 46. Making the glass/epoxy sample once the panel layup was complete.
Fig. 47. Vacuum bagging one of the panel and glass/epoxy samples.

Fig. 48 shows the end product of the plain and satin weave FML fabrication process. The calculated densities (using measured mass and volume) for fabricated panels are listed in Table 11 on the next page. Microscopic and tensile specimens with dimensions identical to GLARE specimens were then made and prepared as previously described.

Fig. 48. Fabricated plain weave and satin weave FML panels.
Table 11. Calculated densities of fabricated panels.

<table>
<thead>
<tr>
<th>Fabricated sample</th>
<th>Density (g/cm$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plain weave sample (S-2 glass + epoxy)</td>
<td>1.10</td>
</tr>
<tr>
<td>Satin weave sample (S-2 glass + epoxy)</td>
<td>1.61</td>
</tr>
<tr>
<td>Plain weave FML</td>
<td>2.19</td>
</tr>
<tr>
<td>Satin weave FML</td>
<td>2.23</td>
</tr>
</tbody>
</table>

Microscopy

Results and Analysis

Cross sectional microscopic images of the plain and satin weave laminates are shown on the next page in Figs. 49 and 50. One concern with these new laminates is the amount of matrix porosity in the glass/epoxy layers, Fig. 51 (pg. 67). This is more evident in the plain weave laminate which could be due to its relatively large amount of weave looseness (much space between yarns) leading to more trapped air as opposed to the more tightly woven satin weave. Taking measures to reduce porosity during fabrication should be an area of further investigation, although it does make them lighter.
Figure 49. Plain weave laminate cross section taken at 40X magnification.

Figure 50. Satin weave laminate cross section taken at 40X magnification.
**Figure 51.** Observed porosity in a plain weave laminate glass/epoxy layer taken at 100X magnification.

**Tensile Testing**

**Results and Analysis**

A total of 8 specimens were tensile tested; 4 glass/epoxy specimens (2 plain weave and 2 satin weave) and 4 FMLs (2 plain weave and 2 satin weave), Fig. 52 (next page). Tensile testing occurred in the fashion previously described for GLARE.
Figure 52. The 8 fabricated specimens that were tensile tested.

Figs. 53 and 54 on the following page show stress-strain results from two sets of tests (specimens 1-4 and specimens 5-8). Each set consisted of a plain weave glass/epoxy specimen, a satin weave glass/epoxy specimen, a plain weave FML specimen and a satin weave FML specimen. The second complete set was conducted to verify the consistency of results which can vary widely, particularly with the properties of new, un-scrutinized composites.
Figure 53. Stress-strain responses obtained from first set of tensile tests.

Figure 54. Stress-strain responses obtained from second set of tensile tests.
Stress-strain responses from the two sets of test show similar behavior to demonstrate consistency. Ultimate strength differences for glass/epoxy specimens are relatively high; the satin weave failed at 533 MPa, while the plain weave failed at 264 MPa. Similarly, Young’s modulus for the satin weave glass/epoxy was much higher than the plain weave. However, Young’s modulus of the FML for both plain and satin weave specimens are almost identical; ~49 GPa for both. The laminate specimen stress-strain behavior is further compared to GLARE and aluminum alloy 2024-T3 stress-strain behavior, Fig. 55 below and Fig. 56 on the following page.

Figure 55. Stress-strain responses of plain and satin weave FMLs compared to GLARE.
Figure 56. Stress-strain responses of plain and satin weave FMLs compared to aluminum alloy 2024-T3 tested in its rolling and transverse to rolling direction.

From Fig. 55, plain weave and satin weave laminates exhibit similar trends to GLARE. Stage I (first part) is primarily elastic deformation of the metal and glass/epoxy, while Stage II (second part) indicates plastic deformation of metal and primarily elastic deformation of glass/epoxy, with some possible matrix damage and fiber fractures. However, the new FML did not experience as much strain to failure as GLARE (the highest is satin weave FML at 3.11%). Further research on epoxy adhesives, fibers that have higher strains to failure, surface and weaving effects would be an area of future interest. Adhesive and fibers with this property might stretch the laminate stress strain curve further.

Looking at the plots, the satin weave FML seems to have slightly better ultimate strength and ultimate strain when compared to the plain weave FML. A most likely
cause for this is the more densely packed fibers in the satin weave configuration, enabling it to sustain higher loads as well as straighter fibers with the 8H style. Also, satin weave S-2 glass seemed to hold more epoxy in the vicinity of its fibers with reduced porosity as compared to the plain weave laminate. Plain weave yarns have large amounts of open space and should hold more epoxy. The 0.2% offset yield strains of both new FMLs are approximately ~0.75%, which is close to aluminum 2024-T3 yield strains. This indicates yielding of the metal in the FML (beginning of stage II). A mechanical property comparison between all tested specimens in test 1 (slightly better properties) is presented in Table 12. These values are used for the rule of mixtures relations in the next section.

<table>
<thead>
<tr>
<th>Property</th>
<th>Plain weave glass/epoxy</th>
<th>Satin weave glass/epoxy</th>
<th>Plain weave FML</th>
<th>Satin weave FML</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E$ (GPa)</td>
<td>~11</td>
<td>~19</td>
<td>~49</td>
<td>~49</td>
</tr>
<tr>
<td>$\sigma_y$ (MPa)</td>
<td>-</td>
<td>-</td>
<td>~255</td>
<td>~255</td>
</tr>
<tr>
<td>$\sigma$ (MPa)</td>
<td>265</td>
<td>533</td>
<td>376</td>
<td>451</td>
</tr>
</tbody>
</table>

Additional tests were performed to see how much the neat (fibers with no epoxy) S-2 glass weaves would withstand load and strain on their own before failing. Two specimens of each woven S-2 glass were prepared and then tensile tested as all others. Specimens were made by cutting fiberglass material and adhering 0.51 mm (0.020 in) thick aluminum alloy 2024-T3 tabs using SilverTip MetlWeld epoxy adhesive as outlined earlier. Specimen size and fiber orientation were the same as prior tensile specimens. Results are presented in Figs. 57 and 58 on the next page.
Figure 57. Plain weave S-2 glass tested to observe its strain to failure.

Figure 58. Satin weave S-2 glass tested to observe its strain to failure.
The observed strains to failure are all less than 1.5% whereas published failure strains for unidirectional S-2 glass fibers are greater than 5% [33]. The lower values are probably a consequence of weaving where bent portions of the fibers experience added stress caused by the weaving angle (and sometimes twisting). Thus, weaving should result in lower sustained load and stretch capabilities as compared to unidirectional S-2 glass.

Also, a lack of matrix allows the fibers to abrade one another, leading to stress raisers and premature failure. The matrix assists in transferring of load from one fiber to an adjacent fiber. In the absence of a matrix, there is nothing to transfer the load from one part of a broken fiber to another part. Note that when epoxy was added to these weaves, failure strains were about doubled.

There are some advantages of using woven fiberglass, such as more symmetry within the laminate resulting in similar strengths and mechanical properties. It also provides ease of fabrication and improved handling. Recall, GLARE 4A and GLARE 4B have strong and weak directions, with different preferential strengths because of the differing number of fibers in two perpendicular directions. However, the effect of weaving on FMLs made from woven composite layers should tend to reduce ultimate loads and failure strains as compared to unidirectional reinforced FMLs. A ductile metal like aluminum alloy 2024-T3 seems to extend things further.
Rule of Mixtures

In this section, the rule of mixtures is used to predict Young’s modulus, 0.2% offset yield stress and stress at failure for the plain and satin weave FMLs. The equations used for these calculations are presented below.

\[
E_{\text{lam}} = V_c E_c + V_{\text{met}} E_{\text{met}} \quad (14)
\]

\[
\sigma_{\text{ylam}} = V_c \sigma_c + V_{\text{met}} \sigma_{\text{ymet}} \quad (15)
\]

\[
\sigma_{\text{lam fail}} = V_c \varepsilon_c E_c + V_{\text{met}} \sigma_{\text{met fail}} \quad (16)
\]

Where:

- \( \sigma_c \) - Stress in composite at time of interest
- \( \varepsilon_c \) - Ultimate strain of FML

The 0.2% offset yield strain for both FMLs are \(~0.75\%\) (from test results) and this value is incorporated in the estimation of laminate yield stress. At 0.75%, the stress in the glass/epoxy layers can be found from the stress-strain curves for glass/epoxy only specimens. These were 95 MPa for the plain weave and 167 MPa for the satin weave.

The Young’s modulus for aluminum alloy 2024-T3 L direction used is 72 GPa (FMLs were fabricated in the aluminum rolling direction). The 0.2% offset yield stress used for both FMLs is the stress at 0.75% strain from the aluminum alloy stress-strain curve. The ultimate stress in the aluminum was determined by examining the stress-strain diagram for the aluminum used and obtaining the stress associated with a strain of 2.90% for the plain weave and a strain of 3.11% for the satin weave (ultimate strains).
$V_{\text{met}}$ is calculated by measuring the thickness of metal layers relative to total laminate thickness, Figs. 49 and 50 (pg. 66). This approximate ratio is 0.64 for the plain weave and 0.61 for the satin weave FML (calculated using Eqn. 4). For plain weave FML, composite volume fraction is

$$V_c = 1 - V_{\text{met}} = 1 - 0.64 = 0.36$$  \hspace{1cm} (17)$$

For satin weave FML, composite volume fraction is

$$V_c = 1 - V_{\text{met}} = 1 - 0.61 = 0.39$$  \hspace{1cm} (18)$$

Substituting measured values and solving for Young’s modulus, 0.2% offset yield stress and stress at failure for the plain weave FML one finds

$$E_{\text{lam}} = V_c E_c + V_{\text{met}} E_{\text{met}} = 0.36(11 \text{ GPa}) + 0.64(72 \text{ GPa}) = 50 \text{ GPa}$$  \hspace{1cm} (19)$$

$$\sigma_{\text{lam}} = V_c \sigma_c + V_{\text{met}} \sigma_{\text{met}} = 0.36(95 \text{ MPa}) + 0.64(365 \text{ MPa}) = 268 \text{ MPa}$$  \hspace{1cm} (20)$$

$$\sigma_{\text{lam fail}} = V_c \epsilon_c E_c + V_{\text{met}} \sigma_{\text{met fail}} = 0.36(0.0290)(11 \text{ GPa}) + 0.64(409 \text{ MPa}) = 377 \text{ MPa}$$  \hspace{1cm} (21)$$

From the plain weave FML tensile test, $E_{\text{lam}}$ is 49 GPa, $\sigma_{\text{lam}}$ is 255 GPa and $\sigma_{\text{lam fail}}$ is 376 MPa. This gives a difference of 2.0%, 5.0%, and 0.3% respectively. Substituting measured values and solving for Young’s modulus, 0.2% offset yield stress, and stress at failure for the satin weave FML one finds
\[ E_{\text{lam}} = V_c E_c + V_{\text{met}} E_{\text{met}} = 0.39(19 \text{ GPa}) + 0.61(72 \text{ GPa}) = 51 \text{ GPa} \quad (22) \]

\[ \sigma_{y\text{lam}} = V_c \sigma_c + V_{\text{met}} \sigma_{\text{met}} = 0.39(167 \text{ MPa}) + 0.61(365 \text{ MPa}) = 288 \text{ MPa} \quad (23) \]

\[ \sigma_{\text{lam fail}} = V_c \varepsilon_c E_c + V_{\text{met}} \sigma_{\text{met fail}} = 0.39(0.0311)(19 \text{ GPa}) + 0.61(413 \text{ MPa}) = 482 \text{ MPa} \quad (24) \]

From the satin weave FML tensile test, \( E_{\text{lam}} \) is 49 GPa, \( \sigma_{y\text{lam}} \) is 255 GPa and \( \sigma_{\text{lam fail}} \) is 451 MPa. This gives a difference of 4.0\%, 12.2\%, and 6.6\% respectively. Estimated values using the rule of mixtures prove to be slightly more accurate for the plain weave FML compared to the satin weave FML.
V DISCUSSION

Mechanical Properties Comparison

Presented in Table 13 are mechanical properties of the grades of GLARE tested compared to the restructured FMLs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Panel Thickness (mm)</th>
<th>Al Layers</th>
<th>Glass/Epoxy Layers</th>
<th>Prepreg Orientations In Glass/Epoxy Layers</th>
<th>E (GPa)</th>
<th>(\sigma_y) (MPa)</th>
<th>(\sigma_u) (MPa)</th>
<th>(\rho) (g/cm(^3))</th>
<th>Density Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>GLARE 3-4/3-0.4</td>
<td>2.43</td>
<td>4</td>
<td>3</td>
<td>0/90/90/90/0/90/0/90</td>
<td>-55</td>
<td>365</td>
<td>690</td>
<td>2.48</td>
<td>0.89</td>
</tr>
<tr>
<td>GLARE 4A-4/3-0.3</td>
<td>2.45</td>
<td>4</td>
<td>3</td>
<td>0/90/0/0/0/90/0/90</td>
<td>-52</td>
<td>270</td>
<td>880</td>
<td>2.30</td>
<td>0.83</td>
</tr>
<tr>
<td>GLARE 4B-3/2-0.4</td>
<td>2.06</td>
<td>3</td>
<td>2</td>
<td>90/0/90/90/0/90/0/90</td>
<td>-55</td>
<td>-270</td>
<td>801</td>
<td>2.45</td>
<td>0.88</td>
</tr>
<tr>
<td>Al 2024-T3 (L)</td>
<td>3.23</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-72</td>
<td>365</td>
<td>484</td>
<td>2.78</td>
<td>1.00</td>
</tr>
<tr>
<td>Al 2024-T3 (LT)</td>
<td>3.23</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-71</td>
<td>310</td>
<td>468</td>
<td>2.78</td>
<td>1.00</td>
</tr>
<tr>
<td>Plain weave FML</td>
<td>2.53</td>
<td>3</td>
<td>2</td>
<td>Symmetric woven</td>
<td>-49</td>
<td>-255</td>
<td>376</td>
<td>2.19</td>
<td>0.79</td>
</tr>
<tr>
<td>Satin weave FML</td>
<td>2.73</td>
<td>3</td>
<td>2</td>
<td>Symmetric woven</td>
<td>-49</td>
<td>-255</td>
<td>451</td>
<td>2.23</td>
<td>0.80</td>
</tr>
<tr>
<td>Plain weave composite</td>
<td>0.51</td>
<td>-</td>
<td>-</td>
<td>Symmetric woven</td>
<td>-11</td>
<td>-</td>
<td>265</td>
<td>1.10</td>
<td>0.40</td>
</tr>
<tr>
<td>Satin weave composite</td>
<td>0.54</td>
<td>-</td>
<td>-</td>
<td>Symmetric woven</td>
<td>-19</td>
<td>-</td>
<td>533</td>
<td>1.61</td>
<td>0.58</td>
</tr>
</tbody>
</table>

Note: The 0° direction is the aluminum rolling direction while 90° is transverse to it. The Young’s modulus, yield strength (0.2% offset) and ultimate strength values for each GLARE type are the highest measured values of the two different test directions for each grade of GLARE (e.g., strong). Density ratio is that of GLARE to aluminum alloy 2024 (~2.78 g/cm\(^3\)).
Failure Characteristics

Each component in an FML contributes to holding the laminate together. When the aluminum yields, it will provide support for the fibers and allow for greater strains in the FML. The fibers provide good ultimate strengths but at a small amount of strain [20].

While the FML specimens experienced delamination, glass/epoxy only specimens exhibited quite different failure characteristics. Close up images of failed glass/epoxy specimens are shown in Fig. 59 and Fig. 60 on the following page.

The occurrence of delamination is said to be mainly because of the epoxy matrix at the aluminum-fiber interface. Under bending, it is said that the location of delamination is mostly at these matrix rich layers close to the laminate neutral axis (this is the location of highest shear) [20]. Here, although specimens were tensile tested, similar types of delamination failures were observed, Figs. 61, 62 and 63 that follow.

![Image](image.jpg)

**Figure 59.** Broken fibers in plain weave S-2 glass + epoxy specimen after it failed. Thickness of specimen is 0.51 mm.
Figure 60. Satin weave S-2 glass + epoxy specimen after it failed. Thickness of specimen is 0.51 mm.

Figure 61. Delamination of GLARE 4A-4/3-0.3. Each metal layer is 0.3 mm thick.
Figure 62. Delamination experienced by plain weave laminate. Each metal layer is 0.51 mm thick.

Figure 63. Delamination experienced by satin weave laminate. Each metal layer is 0.51 mm thick.
VI CONCLUSIONS

From tests performed at ERAU, it can be concluded that;

1) GLARE 4A, GLARE 4B and GLARE 3 can produce ultimate strengths that are significantly higher than aircraft grade aluminum alloy.

2) GLARE 4A and 4B oriented in the strong direction exhibited higher ultimate strengths. When loaded in the strong orientation, specimens experienced delamination. In contrast, when loaded in the weak orientation, they fractured with minimal delamination.

3) GLARE 3 is a symmetric grade that had similar stress-strain behavior in both orientations. However, the specimen tested in the aluminum rolling direction showed a slightly higher strength.

4) GLARE 4A, GLARE 4B and GLARE 3 exhibited low strains at failure (<5%) while aluminum alloy 2024-T3 experienced high strains (>14%).

5) Values obtained from shear testing specimens that were sandblasted compared to specimens that were not sandblasted indicated that sandblasting appears to be a necessary mechanical abrasion process before each surface treatment.

6) AC-130 Sol-Gel is a more innovative, easy to apply process, providing good bonding characteristics between the aluminum and the adhesive.

7) Restructured FMLs were fabricated and mechanical properties compared to those of GLARE. Test results indicated similar stress-strain behavior, but FML ultimate strengths were lower than GLARE ultimate strengths.

8) The densities of these FMLs were lower than the density of GLARE which could result in weight savings.
9) The cost associated with fabrication and manufacture of the FMLs are lower than those associated with GLARE.

10) To avoid abrupt failure, it is advantageous for the matrix material to exhibit high strains. GLARE exhibited higher strains at failure than the FMLs. The woven fibers used in the FML limited the strain at failure of the laminates to a maximum of 3.11% compared to the unidirectional fibers in GLARE (>4%).

11) There are many variables that can be altered to improve FML properties. Those variables include but are not limited to different adhesives, different types of fiberglass, other metals and improved fabrication processes. This opens up countless possibilities for improvement.

Some future considerations would be eliminating porosity in glass/epoxy layers of the laminate and stretching the strain to failure further towards conventional metal, while maintaining high strength to weight ratios.
References


Appendix A

Bibliography


