Experimental Investigation of Enhanced Damage Resistant Fiber Metal Laminates

Cannelle Metang Tefoung

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EXPERIMENTAL INVESTIGATION OF ENHANCED DAMAGE RESISTANT FIBER METAL LAMINATES

A Thesis

Submitted to the Faculty

of

Embry-Riddle Aeronautical University

by

Cannelle Metang Tefoung

In Partial Fulfillment of the Requirements for the Degree

of

Master of Science in Aerospace Engineering

May 2018

Embry-Riddle Aeronautical University

Daytona Beach, Florida
EXPERIMENTAL INVESTIGATION OF ENHANCED DAMAGE RESISTANT FIBER METAL LAMINATES

by

Cannelle Metang Tefoung

A Thesis prepared under the direction of the candidate’s committee chairman, Dr. David J. Sypleck, Department of Aerospace Engineering, and has been approved by the members of the thesis committee. It was submitted to the School of Graduate Studies and Research and was accepted in partial fulfillment of the requirements for the degree of Master of Science in Aerospace Engineering.

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Finally, I am very grateful to God, for making all this possible.
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<th>Description</th>
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<tr>
<td>ARALL</td>
<td>Aramid Reinforced Aluminum Laminate</td>
</tr>
<tr>
<td>CAA</td>
<td>Chromic Acid Anodizing</td>
</tr>
<tr>
<td>ERAU</td>
<td>Embry-Riddle Aeronautical University</td>
</tr>
<tr>
<td>FMLC</td>
<td>Fiber Metal Laminates Centre of Competence</td>
</tr>
<tr>
<td>FMLs</td>
<td>Fiber Metal Laminates</td>
</tr>
<tr>
<td>GLARE</td>
<td>Glass Laminate Aluminum Reinforced Epoxy</td>
</tr>
<tr>
<td>PAA</td>
<td>Phosphoric Acid Anodizing</td>
</tr>
<tr>
<td>SFT</td>
<td>Self-forming Technique</td>
</tr>
<tr>
<td>SLC</td>
<td>Structural Laminate Company</td>
</tr>
<tr>
<td>SRPP</td>
<td>Self-Reinforced Polypropylene</td>
</tr>
<tr>
<td>TFMLs</td>
<td>Thermoplastic Fiber Metal Laminates</td>
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ABSTRACT

Cannelle Metang Tefoung, MSAE, Embry-Riddle Aeronautical University, May 2018.

Experimental Investigation of Enhanced Damage Resistant Fiber Metal Laminates.

Fiber metal laminates (FMLs) are hybrid composite structures made from fiber reinforced polymeric materials sandwiched between layers of thin metal alloy sheets. FMLs combine the advantages of metallic materials and fiber reinforced matrix systems. Some of these advantages include: lighter weight, enhanced impact tolerance, improved corrosion resistance, better fire resistance, longer fatigue life, greater tensile strength, easier repair ability and maintainability, etc. The most well-known commercially available FML is GLARE (Glass Laminate Aluminum Reinforced Epoxy), however there are ongoing issues, which include a very high fabrication cost compared to many competing material systems and substantial delamination upon impact and failure. The main purpose of this study is to fabricate and test similar types of FMLs, but ones made using simpler, less expensive approaches. Two different adhesive systems were used; high strain epoxy as the matrix for all S-2 glass fiber reinforced composite layers and either the same epoxy or a high strain methacrylate to bond the composite layers to the metal layers. For effective comparison, the new FMLs laminate arrangement(s) closely mimic a few common grades of GLARE. The experimental investigation comprised of shear lap testing, tensile testing, and microscopy (e.g., optical and SEM). These experimental methods were used to assess potential improvements. The fabricated FMLs showed similar behaviors to GLARE but with some substantially improved damage tolerance characteristics.
1. Introduction

1.1. Problem Statement

Fiber metal laminates (FMLs) are hybrid composite structures made from fiber reinforced polymeric materials sandwiched between layers of thin metal alloy sheets. FMLs combine the advantages of metallic materials and fiber reinforced matrix system. Some of these advantages include: lighter weight, enhanced impact tolerance, improved corrosion resistance, better fire resistance, longer fatigue life, greater tensile strength, easier repair ability and maintainability, etc. The most well-known commercially available FML is GLARE (Glass Laminate Aluminum Reinforced Epoxy) however, there are ongoing issues, which include a very high fabrication cost compared to many competing material systems and substantial delamination upon impact and failure.

1.2. Purpose of Study

The main purpose of this study is to fabricate and test similar types of FMLs while using simpler fabrication approaches in order to reduce cost. In addition to cost reduction, the fabricated FMLs are also desired to have improved mechanical properties mainly; an increased percentage elongation before failure and improved bonding characteristics between laminate layers.

1.3. Significance of Study

The sought outcomes of this study are:

- Develop a new light weight fiber metal laminate that can potentially outperform similar grades of GLARE in mainly two categories: delamination and percent elongation to failure.
• Reduce the cost of FMLs by using simpler fabrication approaches without compromising quality.

The newly fabricated FMLs could eventually be introduced into aerospace products. Lower cost of fabrication and their inherent light weight would be attractive for use in multiple components. By doing so, the overall weight of aircraft, rockets, spacecraft, or any other product would be reduced, in turn reducing fuel consumption, increasing range and payload, etc. These changes can result in overall improved fuel efficiency and reduced operating cost. Finally, this study seeks to improve the understanding of FMLs, to enhance laboratory expertise, develop creative thinking, and improve hands on problem solving abilities.
2. Literature Review

2.1. Overview of Fiber Metal Laminates

Weight reduction is one of the main objectives in the aerospace industry. Fiber metal laminates came about due to the search for lighter and more damage tolerant materials that could replace aluminum alloys that are so widely used in aerospace structures. Much attention is placed on damage tolerance designs especially in the aircraft fuselages. This is due to the ever-increasing fuselage diameter as well as cabin pressures (Vogelsang & Vlot, 2000). The use of aluminum alloys alone has certain inherent disadvantages such as susceptibility to fatigue and corrosion. FMLs (Fiber Metal Laminates) combine the advantages of metallic materials and fiber reinforced composite systems (Sinmazçelik, Avcu, Bora, & Çoban, 2011). The presence of the composite materials mostly yields weight specific advantages.

The idea of metal bonding came about due to a failed experiment by Delft University (Delft, Netherlands) researchers, during the early 1940s. At the time, de Havilland (Hatfield, Hertfordshire, United Kingdom) a British aircraft company was one of the last manufacturers to design aircrafts made mainly from wood. They had built the famous Mosquito, a wooden fighter-bomber aircraft whose wings and fuselage were designed by bonding layers of wood with an adhesive in a curved mold. To improve their wood bonding expertise, De Bruijne, a Dean of Cambridge University (Cambridge, England, United Kingdom) experimented with different types and mixtures of adhesives. He accidentally found that the adhesive in his experiment effectively bonded the wooden aircraft parts to the metal hot plates of their heated press. An engineer, Rob Schliekelmann then conducted some experiments using this idea and he found that bonded metal sheets
under compression were 60% stronger than riveted metal sheets and 5% lighter (Vlot, 2001). Schliekelmann later moved to Fokker (Amsterdam, The Netherlands), another aircraft manufacturer, where 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 of high enough quality to be used in the center wings of the Fokker F-27 Friendship.

After a visit to NASA Langley Research Center (Hampton, Virginia, USA) where researchers investigated reinforcements bonded to aluminum structures in Space Shuttle components, Schliekelmann’s group used the idea to create FMLs. This was done by reinforcing the adhesives used in their metal laminates with nylon and carbon fibers. Woven nylon fibers and unidirectional carbon fibers were embedded within 1 mm thick sheets of aluminum. The crack growth rates in these structures were found to be two to three times slower than in the aluminum alloys alone, with the carbon fiber reinforced structures showing a more significant reduction. This property was however not remarkable for future usage when considering the cost of materials (Vlot & Gunnink, 2001).

Have, Schijve and Vogelesang conducted flight simulation tests at the Delft University of Technology (Delft, The Netherlands) in 1978. These tests were performed on carbon and aramid fiber reinforced 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 all aligned in the loading direction but only half of the aramid fibers had substantial applied loads (this was due to their woven nature). Further research was carried out at Delft University to improve the performance of FMLs with
aramid fibers. An analytical model based on fracture mechanics was developed by Marissen to predict fatigue crack growth. This was done by calculating the stress intensity factor of the fatigue crack while taking into consideration fatigue cracks in the metal layers and the load sustained by crack bridging of intact fibers at the center of the crack. The predicted crack growth rates from his model were in line with the observed crack growths. These results led to two significant concepts in FML development; higher strength fibers were better at bridging fatigue cracks, while thinner metal layers could enhance the use of more fiber layers, which would consequently minimize the shear stresses in the adhesive between the metal and fiber layers hence reducing delamination (Vlot & Gunnink, 2001).

Following Marissen’s concepts, ARALL (Aramid Reinforced Aluminum Laminate) was designed and fabricated. It was the first commercial FML launched by ALCOA (New Kensington, PA). The aramid fibers were embedded in an epoxy adhesive matrix and then sandwiched between 2024 or 7075 aluminum alloy sheets (creating the first two ARALL grades). 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), and with 33% weight saving benefits. Later research, introduced other aluminum alloys into ARALL. The Boeing Company (Chicago, IL) C-17 military transport airplane cargo doors were manufactured using ARALL 3 (made with Al-7475 alloy and post-cure stretching) resulting in 26% weight savings. High manufacturing cost was associated with this process (about 8 to 10 times more than aluminum cargo doors), hence only about thirty airplanes were built with ARALL doors. Fuselage studies conducted on ARALL were unsatisfactory, as only 8% weight savings were achieved. (Vlot & Gunnink, 2001).
ARALL was later found to have strength reduction issues (mostly caused by drilled holes) while doublers attached to the underlying structure to improve strength experienced premature fatigue cracking. Hence it could not be applicable for fuselage structures. Furthermore, experimental research by Roebroeks claimed that aramid fibers didn’t effectively bond to the adhesive, causing fiber pull out and eventually fiber breakage when subjected to compressive loads (Vlot & Gunnink, 2001). To improve upon ARALL laminates, high strength glass fibers instead of aramid fibers were introduced (Sinmazcelik, Avcu, Bora, & Çoban, 2011). Hence creating a new FML called GLARE (Glass Laminate Aluminum Reinforced Epoxy).

2.1.1. Overview of GLARE

Roebroeks and Vogelesang were both students from Delft University who worked on various experimental investigations to improve upon ARALL. In October 1987, a patent for GLARE (United States Patent 5,039,571) was filed by AKZO (Amsterdam, Netherlands) with both as named inventors. However, GLARE’s production and commercialization only started in 1991 following a partnership between AKZO and ALCOA. Their partnership led to the foundation of the Structural Laminate Company (SLC), (Vlot & Gunnink, 2001).

The new large airplane group at Boeing at the time, solicited SLC’s assistance in design studies. Hence the use of GLARE for the Boeing 777 was investigated. Since the aircraft was in its final stages of production, GLARE could not be considered for vital parts of the aircraft, but its material properties advantages could be used elsewhere. Hence various research was carried out to identify its performance characteristics. A few commercial applications came about.
The impact resistance of GLARE was studied by Vlot. He investigated the effect of both high and low velocity impact. Results showed that during low velocity impact, GLARE could withstand impact loads as good as aluminum alloys and much better than carbon fiber composites. Meanwhile at high velocity impact, GLARE’s impact properties were significantly better than aluminum due to the much stronger glass fibers within the laminates. Another substantial practical advantage of GLARE was that impact damage could be easily detected with the naked eye as opposed to most other composite materials. This was because, once impacted, the outer aluminum layer permanently deformed or dented, due to yielding. For this reason, GLARE was the material used in the cargo floor of the Boeing 777, a structure highly susceptible to impact damage. (Vlot & Gunnink, 2001).

GLARE is currently being used on the Airbus (Toulouse, France) A380 for components such as upper fuselage skins, fuselage butt straps and leading edges of the horizontal and vertical stabilizers (Norris, 2005). This is shown in Figure 2.1. Various prior studies carried out on GLARE concluded outstanding fatigue resistant properties, enhanced damage tolerance, light weight and cost effectiveness (Vogelsang & Vlot, 2000).
Figure 2.1 Usage of GLARE on several components of the Airbus A380 (Norris, 2005).

GLARE Lay-up and Fabrication Process

GLARE is made by surrounding S-glass or S-2 glass fibers with an epoxy adhesive (these are called prepreg layers), arranging and sandwiching them between thin aluminum alloy sheets (Figure 2.2).

Figure 2.2 Arrangement of layers within GLARE (Sadighi, Alderliesten, & Benedictus, 2012).
The aluminum alloy sheets have a thickness ranging from 0.2 mm (0.008 in) to 0.5 mm (0.020 in). The aluminum alloy used varies; 7475-T761 is used in GLARE 1 while 2024-T3 is used in all other grades of GLARE. All of the aluminum layers are arranged in the same orientation, called their rolling direction, which is defined as the 0° orientation throughout the GLARE grades. The adhesive used within the prepreg layers is FM-94 epoxy, manufactured by Cytec Industries Inc. (Woodland Hills, NJ). AGY (Aiken, SC) uses it with their S-2 glass fibers to make the prepreg. The volume fraction of the fibers in the prepreg after consolidation is about 59% (Roebroeks, 2001). Microstructural analysis and tensile testing of various grades of GLARE revealed that some of these grades have different mechanical properties depending on the loading direction (‘strong’ and ‘weak’ direction) with the weak direction showing the least favorable properties (Benedict, 2012). Additional details of ‘directions’ are discussed later in section 2.2.

Depending on fiber orientation, the number of prepreg and aluminum layers in the FML, six standard grades of GLARE are available; GLARE 1, GLARE 2, GLARE 3, GLARE 4, GLARE 5 and GLARE 6. A laminate coding system is used to differentiate these materials. For example, GLARE 4B-4/3 0.3 is defined as a GLARE laminate with 4B fiber orientation, 4/3-layer arrangement; meaning 4 aluminum layers and 3 fiber layers and finally, each aluminum layer is 0.3 mm (0.012 in) thick.

The aluminum alloy sheets are pretreated before layup for better adhesion. They are either Chromic Acid Anodized (CAA) (old method) or Phosphoric Acid Anodized (PAA) (latest method) and then primed with a corrosion inhibiting primer, known as BR-127 epoxy (Cytec Industries Inc.). The pretreated surface is then bonded to the FM-94 based prepreg(s). The S-2 glass fibers in the prepreg layers are not woven, and various
laminate properties in different orientations are achieved by varying prepreg fiber orientations during layup. This lay-up is placed in a pre-shaped mold, vacuum bagged and then cured in an autoclave at high temperature and pressure (about 120°C and 600 - 1100 kPa ≈ 10 atmosphere) to consolidate and cure (Sinke, 2009).

**Splicing Concept**

According to Boeing, GLARE was too expensive to be considered for aluminum replacement (Vlot & Gunnink, 2001). To help reduce the cost, a production method for larger GLARE sheets had to be developed, hence the splicing concept came about. This concept enables the production of larger GLARE sheets while slightly reducing the overall cost. With splicing, the aluminum alloy layers have a small gap between each normally continuous sheet, while the prepreg layers are left continuous to bridge the gaps. The interruptions in these metal layers are called splices. A schematic representation is shown in Figure 2.3.

![Figure 2.3 Representation of splicing technique](Roebroeks, 2001)
However, with this technique, the outer aluminum layers were observed to experience sudden delamination when the transverse stress near the splice exceeded about 400 MPa. Hence resulting in the splice being the weakest component of the laminate. 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 spliced areas. The same adhesive used in the prepreg layers are also often added to fill up gaps. (Roebroeks, 2001).

Self-forming Technique (SFT) is a method using autoclave pressure to form the laminate with doublers and adhesive in one cure cycle. This more cost effective technique was developed to solve the difficulty of bonding spliced GLARE in two bonding cycles. 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 (Roebroeks, 2001).

**Delamination and Fiber Bridging**

Fatigue crack growth is a very common, yet complex problem, faced by aerospace and many other products. It was one of the failure mechanisms observed during experimental investigation of FMLs at Delft University.

During cracking of metal layers, fiber bridging of the cracked metal layers mostly occurs. This is a process where by some of the loads normally tolerated by the aluminum alloy layers are transferred to the fibers via shear stress with the adhesive. By doing so, the metal layers are less loaded, and crack growth in the metal layer is eventually slowed down or arrested. However, at this stage, the adhesive is more heavily loaded and starts separating from the metal layers. Due to this, even more additional shear stress occurs at the metal-prepreg interface, hence delamination occurs. For a given laminate thickness, thinner metal
layers allow for more fibers, which is thought to result in smaller shear stress at the interface. While these shear stresses create delamination, the loads sustained by the fibers are reduced, resulting in fewer breaks. Figure 2.4 is an illustration of crack bridging and delamination (Vlot, 2001).

Figure 2.4 Bridging of fatigue cracks in the metal layers (Vlot, 2001).
Advantages of GLARE Over Aluminum 2024-T3 Alloy

Using GLARE has several advantages over aluminum alloys. As mentioned above, a common aerospace aluminum alloy Al-2024-T3 is one of the components in most grades of GLARE. Table 2.1 summarizes the performance ratio for various properties. GLARE has significantly higher performance when considering damage tolerance, flame resistance, thermal insulation, impact blast resistance and corrosion resistance as compared to Al-2024-T3.

Table 2.1 Comparison of GLARE and Al-2024-T3 (AGY, 2013).

<table>
<thead>
<tr>
<th>Property</th>
<th>GLARE to Al-2024-T3 ratio</th>
</tr>
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<tr>
<td>Weight</td>
<td>0.7 - 0.9</td>
</tr>
<tr>
<td>Strength</td>
<td>1 - 2</td>
</tr>
<tr>
<td>Fatigue</td>
<td>3 - 100</td>
</tr>
<tr>
<td>Damage Tolerance</td>
<td>1 - 2</td>
</tr>
<tr>
<td>Impact Blast Resistance</td>
<td>2 - 10</td>
</tr>
<tr>
<td>Flame Resistance</td>
<td>5 - 50</td>
</tr>
<tr>
<td>Lightning Strike</td>
<td>1.5 - 2.5</td>
</tr>
<tr>
<td>Thermal Insulation</td>
<td>100 - 150</td>
</tr>
<tr>
<td>Corrosion Resistance</td>
<td>2 - 10</td>
</tr>
<tr>
<td>Reparability</td>
<td>1+</td>
</tr>
<tr>
<td>Maintenance</td>
<td>1+</td>
</tr>
</tbody>
</table>

2.1.2. Other Types of FMLs

Due to the outstanding properties of GLARE, the idea to extend the concept of FMLs to other components came about. For example, aluminum alloys could be replaced with a more temperature resistant materials (e.g., titanium alloys) to create an FML useful
in thermal protection systems for space vehicles. Hence by choosing specific components and laminate build-ups, a FML can conceivably be custom-made for a number of desired applications (Vlot & Gunnink, 2001). One such class of tailored FMLs are thermoplastic FMLs which are currently still being studied.

**Thermoplastic FMLs (TFMLs)**

This is a sandwich structure that usually consists of aluminum alloys bonded between thermoplastic reinforced fiber layers. Some of the fibers include glass, carbon, aramid, etc. The most common thermoplastic used in such FMLs so far is polypropylene, creating self-reinforced polypropylene (SRPP) composite layers (fibers + polypropylene). The manufacturing technique for these types of FMLs usually involves stacking the layers, using a thin (about 60 micron) film to bond the aluminum layers to SRPP composite layers, heat them under pressure (usually in a pneumatic press) and then allow the layup to cool (Carrillo & Cantwell, 2009).

**Tensile Properties of TFMLs**

A general study of the mechanical properties of TFMLs was compared to those of its constituents. The tensile test revealed that the TFML had higher strength when compared to the plain thermoplastic composite as well higher strain to failure when compared to that of the plain aluminum alloy. The study also showed that in TFML, the fiber orientation only slightly affects its properties (Carrillo & Cantwell, 2009).

Another study on TFML with glass fiber reinforced polypropylene stated that the ultimate strength and elastic modulus of TFML follows the law of mixtures relationship. Here, ultimate tensile strength increases with the addition of composites, meanwhile elastic
modulus reduces. Delamination was observed to be more prominent in thin laminates, meanwhile good bonding was observed in thicker laminates (Reyes & Cantwell, 2000).

Further studies of TFMLs based on aramid fiber reinforced polypropylene showed an increase in the strain to failure of the TFML when compared to the aluminum alloy alone. This increase was due to the excellent bonding between the constituents, hence enabling global plastic deformations in the aluminum sheet (Gonzalez-Canche, Flores-Johnson, & Carrillo, 2017).

**Impact Properties of TFMLs**

Round TFML plates based on glass fiber reinforced polypropylene were subjected to low velocity impact. It was observed that the laminate absorbed a large amount of energy. The energy absorption was explained through observation of the large plastic deformation in the aluminum and composite layers as well as micro-cracking in the glass fiber reinforced polypropylene. The study further stated that the thermoplastic behavior of the laminates meant that the impact damage could easily be repaired by reheating the laminate. This property makes the TFML more advantageous when compared to standard epoxy reinforced FMLs. The TFML are hence said to be completely recyclable (Reyes & Cantwell, 2000).

**Failure Characteristics of TFMLs**

As with most FMLs, failure usually started with fiber/matrix de-bonding with final failure being complete delamination and fiber breakage. The fiber/matrix de-bonding occurred at the laminate boundaries (Hinz, Omoori, Hojo, & Schulte, 2009). TFMLs were found to have a better resistance to localized impact loading when compared to epoxy.
reinforced FMLs, hence making them more appropriate for dynamically loaded structures. Unfortunately, TFMLs were also found to have low toughness at the metal/polymer interface hence making them unsuitable for impact situations leading to high deformation and fractures (Santiago, Cantwell, & Alves, 2017). More research and studies are still needed in order to see these TFMLs applied in future engineering applications.

2.1.3. FMLs Summary

In general, most types of FMLs have similar behavior. They combine the advantages of metallic materials and fiber reinforced matrix system. Some of the many benefits of using FMLs over aluminum alloys have been discussed. An ongoing issue with most FMLs is high cost and ongoing delamination issues. This has motivated some prior ERAU research.

2.2. Previous ERAU Research

Three previous ERAU graduate students performed research that were of paramount importance to this thesis.

Firstly, Olaniyan (2010) began the ERAU work by obtaining several GLARE sheet samples from the Fiber Metal Laminates Centre of Competence (Delft, The Netherlands). He then fabricated his own FMLs through vacuum bagging which were tensile tested. The tests revealed a paramount importance for using appropriate surface treatments and a good adhesive to resist delamination.

Next, Benedict (2012) carried out an experimental investigation (comprised of optical microscopy and tensile testing) on different grades of GLARE. The microscopy revealed interesting construction and defect details of GLARE, while tensile testing provided a
means of measuring and analyzing its stress-strain response. The investigation revealed that some grades of GLARE have two different loading directions; weak and strong.

Figure 2.5 shows 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 tested in the transverse direction is the ‘weak’ specimen. In the weak direction, only three unidirectional prepreg layers are pointing out of the page (Benedict, 2012).

![Figure 2.5 Microscopic image of GLARE 4A-4/3-0.3 in its strong direction (Benedict, 2012). Note that her original picture has been slightly adjusted here for better contrast.](image)

She later examined different metal surface pretreatments used during FML fabrication. These included grit blasting, Phosphoric Acid Anodizing (PAA), and AC-130 Sol-Gel (3M™ Company, Maplewood MN) treatment. Recall that PAA is the surface treatment used in GLARE manufacture. Two different metal surface pretreatment combinations were explored: grit blast + PAA and grit blast + AC130 Sol-Gel. AC130 Sol-Gel is a two-part surface pretreatment solution used to enhance adhesion between metals
and another substrate. The tensile test results showed that every grade of GLARE she tested had significant delamination issues upon failure and that new types of surface pretreatment with grit blast + AC130 Sol-Gel yielded preeminent results that were simpler and cheaper than the PAA plus BR-127 treatment. The need for BR-127 epoxy primer and its additional weight were questioned. The FMLs Benedict fabricated through vacuum bagging using S-2 glass fibers and common epoxies, and grit blast + AC130 Sol-Gel treated Al-2024 alloy sheets were nearly as good as GLARE, encouraging additional ERAU work.

Meanwhile, Rudradat (2013) investigated the tensile and impact behavior of FMLs. Her laminates were made by vacuum bagging woven 5052 aluminum mesh or 2024-T3 aluminum sheets, woven 2.47 N (8.9 oz) or 6.67 N (24 oz) ShieldStrand® S glass fibers (Owens Corning, Toledo, OH) that were donated by the company, and high failure strain Hysol EA 9313 epoxy (Henkel North America, Bay Point, CA). Inspired by Benedict, grit blast + AC130 Sol-Gel was the surface pretreatment. The tensile test results revealed that FMLs made with the combination of grit blast + AC130 Sol-Gel treated 2024-T3 aluminum alloy sheets and Hysol EA 9313 epoxy had beneficial elongation at failure properties likely owing to the high failure strain of the epoxy. The exceptional properties of ShieldStrand® S glass fibers also made it an important component for this study. Note that Hysol EA 9313 epoxy adhesive is more recently known as Loctite® EA 9313 epoxy by the manufacturer.
3. Methodology

The primary motivation for the current fabrication approach was simplicity and low cost with good performance. From previous ERAU studies, delamination was a recurring issue with most FMLs. According to Vlot (2001) there is a balance between delamination and crack growth that is assured by the strength of the adhesive and its resistance to delamination. Furthermore, varying the epoxy adhesive was found to influence the strain range in composite materials (Anees, Gbaguidi, Kim, & Namilae, 2018). Hence, the adhesive system selection was a main consideration for the fabrication. Other important factors considered were the choice of fiber and metal layers, surface pretreatments, lay-up method and consolidation pressure. All prior ERAU studies involved room temperature vacuum bagging with substantial post consolidation porosity being observed within the epoxy. Note that GLARE is consolidated in an autoclave at much higher pressure and temperature, like most aerospace grade composites.

3.1. Layer Bonding Adhesive System Selection

To bond aluminum alloy layers to composite layers, various adhesive companies were contacted during this process. The companies considered were: Axson Technologies US (Madison Heights, MI), Cytec Solvay Group (Bruxelles, Belgium), ITW Polymer Adhesives (Danvers, MA) and Henkel Corporation Aerospace (Bay Point, CA). Apart from performance characteristics of these adhesives, cost and practicality were also deciding factors. The following characteristics were considered when choosing the adhesive system:

- High peel/lap shear strength to ensure strong bonding between laminate layers
- High tensile strength
• High percent elongation at failure (at least 4.5%)
• Low viscosity for ease of use during wet fabrication
• Long pot life
• Suitability for bonding aluminum alloys and glass fibers
• Ability to produce flexible bonds
• Low cure temperature

Plexus® MA530 (ITW, Danvers, MA), a two-part methacrylate adhesive was one of the adhesives selected for this study due to its very high strain to failure (90-160%) and proven ability to bond metal and composite assemblies. It has a 1:1 mix ratio by weight and can be cured at room temperature. Some other properties of this adhesive can be seen in Table 3.1 (ITW PANA, 2017). It was purchased in a 400 ml dual cartridge for $55.89. A Cox™ M400XMR manual 400 ml applicator gun by Sulzer Mixpac USA (Haslett, MI) and mixing tips were needed for mixing. Per manufacturer’s recommendation, Plexus® PC120 primer (ITW, Danvers, MA) purchased for $35.79 per quart was used alongside the adhesive to prepare the aluminum alloy bonding surfaces. Both Plexus® products were purchased from R.S. Hughes Company, Inc. (Orlando, FL).

Another adhesive, Loctite® EA 9313 Epoxy adhesive from Henkel Corporation was one of two adhesives (the other was Loctite® EA 9396) recommended by Henkel’s technical support. Loctite® EA 9313 was selected for this study due to its high lap shear strength, high failure strain, ability to produce flexible bonds and because it showed favorable results when used by a previous ERAU graduate student (Rudradat, 2013). It is a low viscosity, two-part adhesive with a mix ratio of 4:1 by weight for part A (resin) and part B (hardener) respectively. Loctite® EA 9313 is recommended to be cured for 5 days
at 25°C (77°F) or for 1 hour at 82°C (180°F) (this is only applicable for small masses).

Some other properties can be seen in Table 3.1 (Henkel Corporation Aerospace, 2013). A free sample quart kit was donated by Henkel Corporation, another one was purchased for $134.95 from Sky Geek (LaGrangeville, NY).

Table 3.1 Reported properties of selected adhesives.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Plexus® MA530 Methacrylate</th>
<th>Loctite® EA 9313 Epoxy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lap Shear Strength, MPa (psi)</td>
<td>11.0 - 15.2 (1600 - 2200)</td>
<td>27.6 (4006)</td>
</tr>
<tr>
<td>Tensile Strength, MPa (psi)</td>
<td>17.2 - 24.1 (2500 - 3500)</td>
<td>45 (6300)</td>
</tr>
<tr>
<td>Elongation at Break, %</td>
<td>90 - 160</td>
<td>8</td>
</tr>
<tr>
<td>Young’s Modulus, MPa (ksi)</td>
<td>551.6 - 827.4 (80 - 120)</td>
<td>2274 (330)</td>
</tr>
<tr>
<td>Working Time (pot life), min</td>
<td>30 - 40</td>
<td>60</td>
</tr>
</tbody>
</table>

According to the adhesive properties shown in Table 3.1, it can be inferred that; during the adhesive selection process, a compromise had to be made between lap shear strength, elongation at break and tensile strength.

3.2. Composite Layer Material Selection

CES Edupack 2017 (Granta Design USA, Materials Park, OH) is a software tool used for engineering material selection. It provides a database of materials and process information (Granta Design, 2017). The idea was to use the software to rank the best available lightweight aerospace structural composite materials in regard to elongation at failure and fracture toughness. The material search was focused mostly on polymer matrix composites, technical ceramics and glasses. Figure 3.1 shows the summary of the material selection chart obtained. Percent elongation property was plotted on the y-axis while fracture toughness was plotted on the x-axis.
Figure 3.1 Material selection chart based on polymer matrix composites, technical ceramics and glasses.

Figure 3.1 shows that Epoxy/S-glass fiber prepreg lay-up has the highest percent elongation at break (about 3.7%) and highest fracture toughness of about 98 MPa\(\sqrt{\text{m}}\) (89 ksi\(\sqrt{\text{in}}\)) for the materials selected. It was followed by PEEK/carbon fiber prepreg layup, BMI/carbon fiber prepreg layup and Epoxy/carbon fiber prepreg layup which all had percent elongations ranging between 1.0 - 1.8% and fracture toughnesses of about 80 MPa\(\sqrt{\text{m}}\) (73 ksi\(\sqrt{\text{in}}\)). It can then be inferred that using a combination of epoxy and S-glass fibers produces the toughest and most damage tolerant composites. GLARE already takes advantage of this finding, so shall this work.
3.2.1. Fiber Selection

GLARE is the reference material used for this study. Hence the constituents of the fabricated FMLs where chosen to be as close as possible to those of GLARE while keeping cost reduction in mind. The fibers used in GLARE are unidirectional S-2 glass fibers from AGY. Table 3.2 (Benedict, 2012) summarizes some of their reported properties. The fibers are bonded together with FM-94 epoxy (Alderliesten, 2017) and purchased as frozen prepreg layers (Alderliesten, 2017).

Table 3.2 Reported properties of S-2 glass fibers found in GLARE (Benedict, 2012).

<table>
<thead>
<tr>
<th>Reported properties</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber Diameter</td>
<td>5 - 9 µm (197 - 354 µin)</td>
</tr>
<tr>
<td>Strength</td>
<td>4585 MPa (665 ksi)</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>86 - 90 GPa (12.5 - 13 Msi)</td>
</tr>
<tr>
<td>Strain at Failure</td>
<td>5.4 - 5.8 %</td>
</tr>
</tbody>
</table>

For this study, woven S-2 glass fibers instead of unidirectional fibers were used in order to eliminate the complexity of laying fibers in different orientations and also to reduce cost. Although, a consequence of the weaving process is fiber swimming which tends to give slightly less performance than aligned fibers. A prior Master’s thesis (Rudradat, 2013) researched various types of glass fibers and concluded that ShieldStrand® S woven glass fibers had preeminent properties.

It is a high strength performance lightweight fabric. According to the manufacturer, it has high corrosion resistance, excellent fire, smoke and toxicity performance, high ballistic (or impact) performance and durability in extreme environments. It offers up to 40% weight saving when replacing aluminum (Owens Corning, 2010). 2.47 N (8.9 oz)
(weight of material per yd²) ShieldStrand® S bi-directional woven roving material (EPS-S 11) obtained during previous research (Rudradat, 2013) was used in this study. These glass fibers are coated with epoxy compatible sizing to assist with bonding and resin infusion processes. They have similar strain energy properties when compared to S-2 glass fibers (Rudradat, 2013). The fibers were originally donated by Owens Corning Corporation.

3.2.2. Sheet Metal Selection

The aluminum alloy used in most grades of GLARE is Al-2024-T3. Its thickness ranges between 0.2 - 0.5 mm (0.008 - 0.020 in), depending on the grade of GLARE. The thickness of the metal layers plays an important role in reducing delamination and enhancing the fiber bridging effect in FMLs. When using thick metal layers, the loads in the adhesive become too high and much delamination occurs. Hence, lower thicknesses were found to be more advantageous (Vlot, 2001). For this study, bare Al-2024-T3 alloy sheets with a thickness of 0.3 mm (0.012 in) were used. This was chosen to allow effective comparison with GLARE 4A-4/3 0.3 which uses the same metal alloy and thickness.

These aluminum sheets were manufactured by Kaiser Aluminum (Spokane, WA). The material specification is AMS/QQA250/4 and it was purchased from JIT Metals LLC (Boynton Beach, FL). Table 3.3 summarizes some reported mechanical properties of this Al-2024-T3 sheet.

Table 3.3 Reported properties of the Al-2024-T3 sheet (Kaiser Aluminum, 2013).

<table>
<thead>
<tr>
<th>Reported properties</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultimate Strength</td>
<td>460 MPa (66.7 ksi)</td>
</tr>
<tr>
<td>Yield Strength</td>
<td>321 MPa (46.6 ksi)</td>
</tr>
<tr>
<td>Elongation</td>
<td>12 %</td>
</tr>
</tbody>
</table>
3.3. Metal Surface Pretreatment

The surface treatment of the metal layers in FMLs affect the inter-laminate bonding strength. Hence various surface treatment methods are used to enhance adhesion to metal layers during fabrication (Davis & Bond, 1999). These surface treatments mostly proceed in three steps. The first step is usually solvent degreasing to remove any contaminant on the metal surface; this can then be followed by surface abrasion (e.g., grit-blasting) and/or chemical treatments (e.g., phosphoric acid anodizing like the process used in GLARE) to produce a rough surface that favors mechanical interlocking as well as provides an effective surface area for bonding (Critchlow & Brewis, 1996).

Grit blasting was the mechanical abrasion process chosen for this study. The aluminum alloy sheets were uniformly blasted with Glass Bead 80 Grit Abrasive Media (Harbor Freight Tools, Camarillo, CA) in an abrasive blast cabinet using 500 kPa (80 psi) air pressure. The samples were then thoroughly rinsed with tap water to remove the abrasive particles. They were further rinsed with methanol (Duda Diesel LLC, Madison, AL) to remove any contaminants that could be present and finally dried with a lint free cloth, Kimtech Kimwipes 34133 ( Kimberly-Clark Professional, Roswell, GA).

The abrasion process was followed by a chemical treatment. The chosen process was based on previous ERAU research. Two different surface pretreatments of aluminum were studied; namely grit blast + phosphoric acid anodizing and grit blast + AC-130-2 Sol Gel (3M™ Company, Maplewood MN), the latter was found to have preeminent results (Benedict, 2012).
3M™ surface pre-treatment AC-130-2 Solution Gelation (Sol-Gel) enhances adhesion because of the chemical interaction at the interfaces between the metal, the AC-130-2 and the adhesive and/or primer. It is a two-part 500 ml kit and was purchased for $94.50 from Sky Geek. It was applied to the grit blasted aluminum alloy sheets following the spray application process as detailed in the manufacturer application guide. The two chemical parts were mixed, transferred into a nonreactive plastic spray bottle and allowed to rest for 30 min (induction time required to activate the solution). Once the solution was activated, it was then uniformly sprayed onto the sheets, allowed to drain for about 5 - 10 min and then air dried for 60 min in ambient. The process was carried out on both sides of the aluminum sheets. It was then necessary to apply the adhesive system to them within 24 hours of the surface pre-treatment (3M Company, 2015).

### 3.4. Fabrication

The fabricated FMLs closely followed the arrangement in GLARE 4A-4/3-0.3 which has a 4/3 laminate configuration. This comprised of 4 aluminum alloy 2024-T3 layers and 3 composite layers (S-2 glass fiber + epoxy) layers. The composite layers in GLARE 4A-4/3-0.3 are made up of 3 cross ply (0°/90°/0°) unidirectional prepreg layers with a thickness of 0.127 mm (0.005 in) each. Hence the total thickness of each composite layer is 0.381 mm (0.015 in) (Benedict, 2012).

The measured thickness of Shielstrand® S woven glass fibers was 0.23 mm (0.009 in), hence two layers of these glass fibers were used for each composite layer totaling 0.46 mm (0.018 in) thick for each composite layer within the fabricated FMLs. This thickness was chosen to obtain composite layer thicknesses close to those in GLARE 4A-4/3-0.3 to allow for effective comparison of mechanical properties.
The aluminum alloy 2024-T3 sheets were cut using a ClassicCut® Ingento™ paper trimmer (Swingline, Lake Zurich, IL) into pieces of about 114 by 266 mm (4.5 by 10.5 in). The paper trimmer (Figure 3.2) provided straight and smooth edges. Each sheet was then grit blasted and surface pretreated with AC-130-2 Sol Gel. The glass fibers were also cut into similar dimensions using a composite fiber rotary blade cutter and carefully handled to ensure a good alignment during layup.

![Figure 3.2 Paper trimmer used to cut Al-2024-T3 sheets.](image)

### 3.4.1. Preliminary Samples

For the first trial samples, 8H satin weave S-2 glass fibers (excess supply from previous ERAU research, A.V. Benedict, 2012) rather than ShieldStrand® S glass fibers were used to make the composite layers. The glass fibers were bonded together using Loctite® EA 9313 Epoxy adhesive by using a 50/50 by weight ratio (i.e., the weight of the
glass fibers was equivalent to the weight of the adhesive system) prior to vacuum bagging. Three composite layers with dimensions 114 by 266 mm (4.5 by 10.5 in) were fabricated.

**Wet Vacuum Bagging Lay-up Method**

A flat aluminum mold plate was tap water sanded with 320 grit SiC sandpaper, tap water rinsed, degreased with acetone (Duda Diesel LLC, Madison, AL) and waxed (three layers of wax where applied) with Meguiar’s® Mirror Glaze No.8 Maximum Mold Release Wax (Meguiar’s Inc., Irvine, CA). The two glass fiber layers were carefully aligned on the mold and wetted with the epoxy adhesive. A 4 mil (0.004 in) clear plastic sheet was then placed over the wet glass fibers, then with the aid of a Bondo® spreader (3M™ Company, Maplewood MN), excess epoxy was squeezed out (Figure 3.3).

![Figure 3.3 Fiber wet-out process.](image-url)
The clear plastic was discarded, and the lay-up was covered with a porous Teflon® release film, followed by a breather bleeder ply and then a vacuum bagging film. The vacuum bagging film was sealed to the mold using vacuum bagging sealant tape (Figure 3.4). The vacuum bagging supplies were obtained from Aircraft Spruce & Specialty Co. (Peachtree City, GA). A 6 CFM rated Robinair® CoolTech Model 15600 Vacuum Pump (Bosch Automotive Service Solutions, Warren, MI) with appropriate tubes and fittings was used to achieve a vacuum of about $10^{-3}$ Torr leading to about 1 atm (0.101 MPa or 14.7 psi) consolidation pressure on the composite layup. The composite was then allowed to cure under atmospheric pressure overnight, with the vacuum pump on.

![Figure 3.4 Vacuum bagging of one of the composite layers.](image)

After all three composite layer were fabricated, the FML lay-up was made. The grit blasted + AC-130-2 pretreated aluminum alloy 2024-T3 sheets where uniformly coated on their side(s) to be bonded with a thin layer of Plexus® PC120 primer using a paint roller. The first aluminum alloy 2024-T3 layer was placed on the waxed aluminum mold and
evenly coated with Plexus® MA530 adhesive using a paint brush. One cured composite layer was also coated on both sides to be bonded with Plexus® MA530 adhesive and then carefully laid on the metal layer. The process was repeated to create a FML with 3 composite layers sandwiched between four aluminum alloy 2024-T3 layers. The FML was then vacuum bagged following the same process that was used for the cured composite layers, overnight.

**Machining**

Three tensile samples were then cut from the cured FML panels to 25.4 mm (1 in) wide by 254 mm (10 in) dimensions using a Workforce THD550 Tile Cutter (Home Depot, Atlanta, GA) with a water lubricated diamond coated blade (Figure 3.5). The edges of the cut samples were then smoothened using a Buehler (Lake Bluff, IL) tap water lubricated rotating grinding/polishing machine (Figure 3.6). 320 grit PSA SiC sandpaper was used during this process. Wet sanding the edges of these samples avoids undercuts, rough or uneven surfaces which can lead to stress concentrations and premature delamination or fracture.
Figure 3.5 Water lubricated diamond blade tile cutter.

Figure 3.6 Buehler grinding/polishing machine.
The density of each sample was determined using the measured weight and dimensions. The samples seemed thick, hence revealing that the pressure applied during vacuum bagging was not enough to squeeze out all of the excess MA530 adhesive, which is rather viscous compared to the EA 9313 epoxy.

Table 3.4 Calculated densities of preliminary FML samples.

<table>
<thead>
<tr>
<th>Preliminary samples</th>
<th>Density, g/cm³ (lb/in³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trial FML 1</td>
<td>1.68 (0.0607)</td>
</tr>
<tr>
<td>Trial FML 2</td>
<td>1.47 (0.0531)</td>
</tr>
<tr>
<td>Trial FML 3</td>
<td>1.52 (0.0549)</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td><strong>1.56 (0.0564)</strong></td>
</tr>
</tbody>
</table>

**Tensile Testing**

ASTM D3039/D3039M-17 was used as a guide when fabricating the tensile samples. The samples were 25.4 mm (1 in) wide by 254 mm (10 in) long according to the standard (ASTM International, 2017). Gripping tabs 25.4 mm (1 in) wide and 76.2 mm (3 in) long were made from aluminum alloy 6061-T651. For simplicity, they were rectangular for this initial set of tests. These tabs were then attached on both ends and on both sides of the samples using SilverTip Meltweld epoxy adhesive (System Three Resins, Inc., Auburn, WA). Excess adhesive was then squeezed out of the tabs using a small vacuum base vise and wiped off with lint free Kimwipes that had been dabbed with methanol. The tabs were secured in place with rubber bands and allowed to cure undisturbed at room temperature for about 48 hr. After curing, the rubber bands were removed, and the samples were wet
sanded (320 grit) along their edges to eliminate any extra tab epoxy (which can lead to stress concentrations) and also produce a uniform sample width. This allows the test results to be accurate and reproducible (not affected by sample preparation defects).

The tensile tests were performed on an Instron (Norwood, MA) 8802 Servohydraulic Material Testing Instrument (Figure 3.7). The testing system had a capacity of ± 250KN, was equipped with flat serrated face hydraulic grips, and FastTrack™ 8800 digital acquisition and control.

Figure 3.7 Material testing machine.
An Instron relative ramp generator program controlled the actuator displacement rate at 1 mm/min. Position, load and strain data were digitally recorded by sampling at 0.01 kHz (i.e., 10 data sets are recorded every second). For sample testing, an Instron Mod 8802Q3338 strain gauge extensometer with ±12.7mm (0.5 in) gage length was extended to a gauge length of 50.8 mm (2 in) and attached to the center of each test sample prior to testing. It was adhered to the sample using small dabs of BSI-201 5M Quick-Cure™ epoxy (Bob Smith Industries Incorporated, Atascadero, CA), and allowed to cure for 20 min to ensure good strength.

**Preliminary Results**

The force and strain data obtained from the tensile tests were later converted to stress-strain data using each sample’s cross-sectional area (Figure 3.8).

It is observed that the three samples showed similar stress-strain patterns, however sample 3 had a relatively lower ultimate tensile strength (UTS) and percentage strain. The stress-strain curves clearly show a change between the elastic region and plastic deformation regions. The elastic region is seen to end at about 0.5% strain, this is likely to be the point at which the aluminum alloy 2024-T3 layers yielded. The average UTS was about 200 MPa (20 ksi) and the elongation at failure was less than 2%. Note that during constant displacement rate testing, damage relieves the load, and if the damage occurs outside of the extensometer region, the reduced load in that region causes it to elastically contract. This is evident by negative slope at the end of the stress-strain curves.
Figure 3.8 Stress-strain response of vacuum bagged Trial FML samples made with MA530.

Further analysis of the failed samples (Figure 3.9) revealed delamination and most fractures occurring within the gripping tabs. The results from this preliminary test were not satisfactory, however much knowledge and experience were gained motivating various methods of improvement.
3.5. Lap Shear Test

Due to the delamination experienced in the preliminary tensile tests, there was a need to quantify the various bond strengths of the chosen adhesive systems. Different surface pretreatments + adhesive combinations were studied (Figure 3.10).

Figure 3.9 Vacuum bagged trial FML samples made with MA530 after tensile failure.

Figure 3.10 Flow chart of the different surface pretreated + adhesive combinations. The number of samples is in parentheses.
As seen in Figure 3.10, twelve different lap shear specimens were made. All samples were grit blasted as previously described. Nine of these samples were surface pretreated with AC-130-2 Sol Gel; three of which were bonded with Loctite® EA 9313, three were bonded with Plexus® MA530 and three were further treated with Plexus® PC120 primer and finally bonded with Plexus® MA530. The final three samples were treated with Plexus® PC120 primer and then bonded with Plexus® MA530.

3.5.1. Lap Shear Sample Fabrication

Alclad aluminum alloy 2024-T3 2.54 mm (0.1 in) thick sheets were cut into 24.5 mm (1in) wide by 245 mm (10 in) long strips using a foot stomp shear cutting machine found at the ERAU machine workshop. A tap water lubricated MSX255 Series Benchtop Sectioning Machine (Leco Corporation, Saint Joseph, MI) was later used to cut these pieces into 101.6 mm (4 in) long samples (used to make the lap shear specimens) and 50.8 mm (2 in) long samples (used to make the gripping tabs). In total, twenty-four of each were obtained.

Lap Shear Specimens

All of the aluminum alloy 2024-T3 pieces were grit blasted and surface pretreated using the methods summarized in Figure 3.10. Grit blasted 50.8 mm (2 in) long gripping tabs were attached to lap shear specimens using SilverTip Meltweld epoxy adhesive and allowed to cure for 48 hr. In accordance to ASTM D5868-01 (ASTM International, 2014), the lap shear overlay was 25.4 by 25.4 mm (1 by 1 in). To squeeze out the excess adhesive, pressure needed to be applied during the curing process. It is noted that during autoclave curing of most aerospace grade composites, the highest pressure is usually about 0.07 atm.
(150 psi) (Sinke, 2009). Hence 0.07 atm (150 psi) pressure was applied to the shear overlap area. This was accomplished by applying an equivalent compressive force based on the overlay area. Hence about 670 N (150 lb) force was applied using a Tinius Olsen (Horsham, PA) Model 290 Lo-Cap Testing Machine (Figure 3.11).

Figure 3.11 Pressure applied during curing of lap shear specimens.

An aluminum alloy block of 25.4 by 25.4 mm (1 in\(^2\)) cross-sectional area was used to strictly concentrate the applied force to the overlay area. The squeezed excess adhesive was immediately cleaned from the samples using cotton swabs damped with methanol. The pressure was held on each specimen for about 4 hr, then released and the specimens were allowed to cure for another 5 days (for Loctite® EA 9313 bonded specimens) or 2 days (for Plexus® MA530 bonded specimens). This ensured full bond strength before testing.
3.5.2. Lap Shear Test Procedure

The edges of the 12 cured lap shear specimens were then sanded (as previously described) to ensure uniform width and smooth edges. The lap shear specimens were pulled in tension on the Instron material testing instrument (Figure 3.7). The test procedures and displacement rates were similar to those described in the tensile testing subsection above.

Before each test, the strain gauge extensometer was attached to each lap shear specimen as previously described. The extensometer was positioned in such a way that each of its knife edges contacted one of the two components of the lap shear specimens (Figure 3.12). This effectively recorded the displacement within the overlay area during
The aluminum alloy 2024-T3 pieces are comparatively thick, so most of the displacement measured in this way is due to adhesive shearing, not aluminum deformation.

After the lap shear tests, each lap shear overlay area was observed using stereozoom and metallurgical optical light microscopes, ML 7500 Series (Meiji Techno America, San Jose, CA) to characterize the mode of failure experienced by the adhesives. These microscopes (Figure 3.13) were equipped with a 3 megapixel Pixelink (Ottawa, Ontario, Canada) PL-B623CU microscope camera and PixeLink Capture OEM software.

Figure 3.13 Metallurgical (left) and stereozoom (right) optical microscopes.
3.5.3. Lap Shear Test Results

The data obtained during each test were converted to stress-displacement data. The displacement (mm) was obtained from extensometer strain, and was reported as displacement since it allows for comparison of the motion within the bonded area before adhesive failure/break.

**Specimens Bonded with Plexus® MA530 Methacrylate Adhesive**

9 lap shear specimens were bonded with Plexus® MA530. Lap shear test results revealed that all 9 specimens had a similar stress-displacement pattern. The results are summarized in Figure 3.14. Please note that the curves appear thinner here when compared to Figure 3.8 due to low noise effect. The average shear strength was 13 MPa (1.9 ksi) and it lies within the range of the manufacturer’s reported lap shear properties; 11.0 - 15.2 MPa (1600 - 2200 psi) as was previously reported in Table 3.1.
The lap shear specimens treated with Plexus® PC120 primer and bonded with Plexus® MA530 generally experienced the largest displacements with the maximum being about 0.23 mm (0.009 in) upon failure. Surface pretreatment with AC-130-2 Sol Gel had little beneficial effect and was deemed unnecessary when using Plexus® MA530 methacrylate adhesive. The manufacturer’s recommendation of using Plexus® PC120 primer along with Plexus® MA530 methacrylate adhesive was the simplest and best option.
Further microscopic observation of the lap shear overlay clearly showed adhesive still bonded on the two separated surfaces. The adhesive was evenly distributed on either side, hence indicating cohesive failure. This is usually the desired type of failure during lap shear tests (Anderson Materials Evaluation, Inc, 2018).

Figure 3.15 Cohesive failure of Plexus PC120+MA530 lap shear specimen, overlay area viewed under the metallurgical light microscope at 40X magnification.

**Comparison of Specimens Bonded with Plexus® MA530 and Loctite® EA 9313**

9 lap shear specimens were surface pretreated with AC-130-2 Sol Gel. Figure 3.14 also summarizes the stress-displacement behavior of these samples. For effective comparison of the two adhesives’ bond strength, the lap shear specimens were subjected to similar surface pretreatments.

Two of the three specimens bonded with Loctite® EA 9313 epoxy adhesive had the highest shear strength, about 18 MPa (2.6 ksi). Even though the other specimen had a
relatively lower shear strength, it still had the maximum displacement of about 0.21 mm (0.008 in) upon failure. Hence the best combination was that of AC-130-2 Sol Gel surface pretreatment and Loctite® EA 9313 Epoxy adhesive.

Analysis of lap shear overlay areas using the metallurgical microscope revealed that these failures were cohesive failures as well.

Figure 3.16 Cohesive failure of AC130 + EA 9313 lap shear specimen, overlay area viewed under the metallurgical light microscope at 40X magnification.

3.6. FML Fabrication

Following the lap shear test results, two different adhesive systems were chosen for the FML fabrication. One with Plexus® PC120 primer along with Plexus® MA530 methacrylate adhesive and the other with AC-130-2 Sol Gel surface pretreatment followed by Loctite® EA 9313 epoxy adhesive. The composite layers in both FMLs were made from 2.47 N (8.9 oz) ShieldStrand® S bi-directional woven roving material (EPS-S 11) bonded together using Loctite® EA 9313 Epoxy adhesive in 50/50 ratio by weight prior to
consolidation. Two layers of these glass fibers were used for each composite layer which was 0.46 mm (0.018 in thick) of the fabricated FMLs.

3.6.1. Plexus® MA530 Bonded Samples

Aluminum alloy 2024-T3 sheets were cut into pieces of about 114 mm by 266 mm (4.5 in by 10.5 in) and grit blasted as previously described. The glass fibers were also cut into similar dimensions using the rotary blade cutter. The composite layers for these samples were pre-cured before final FML layup. This was done to avoid the mixture of the different uncured adhesives.

Composite Layer Lay-up

To prepare for a high temperature and pressure curing approach, a flat aluminum plate mold was dry sanded with a 5 in single speed random orbit sander (DeWalt, Leola, PA) using 220 grit PSA sandpaper, cleaned and degreased with acetone (Duda Diesel LLC) and then coated with a thin layer of FibRelease® 1153 (Fibre Glast Corporation, Brookville, OH). A porous Nylon Release Peel Ply (Fibre Glast Corporation) was placed on the aluminum mold, two ShieldStrand® S woven glass fiber layers were carefully aligned on the ply. This process enabled easy removal of the composite layer from the plate mold after curing. The glass fibers were then wetted with the EA 9313 epoxy adhesive in a 50/50 ratio by weight. The wet-out process was similar to that previously described and shown in Figure 3.3. The composite lay-up was covered with the porous Nylon Release Peel Ply, followed by a breather bleeder ply (Fiber Glast Corporation) and finally thin aluminum foil. Vacuum bagging was not involved since pressure from the press does the job.
Press Curing Method

To mimic autoclave composite consolidation as well as to reduce porosity and squeeze out excess adhesive, a Wabash (Wabash, IN) Genesis Hydraulic Press G30H-ASTM (Figure 3.17) was used to consolidate and cure all samples.

![Genesis Hydraulic Press G30H-ASTM](image)

Figure 3.17 Genesis Hydraulic Press G30H-ASTM.

A pressure of about 0.07 atm (150 psi) was applied on each sample by using equivalent ton compressive force based on the compressed lay-up area. To protect the platens from spills or damage, they were fully covered with aluminum foil during each cure cycle. The composite layer lay-up was cured in the hydraulic press at 82°C (180°F) for 60 min per adhesive manufacturer’s recommendation (Henkel Corporation Aerospace, 2013).
FML MA530 Lay-up

The previously described platen preparation and lay-up procedure was performed with the previously described MA530 + PC120 FML lay-up process. The FML MA530 lay-up was allowed to cure in the hydraulic press (Figure 3.17) at room temperature for 120 min per manufacturer’s recommendation (ITW PANA, 2017). So pressure was applied but not heat.

3.6.2. Loctite® EA 9313 Epoxy Bonded Samples

Here the FML was made in one cure cycle since the same adhesive was used for both the composite layers and the composite/metal interfaces. The lay-up procedure was performed with the previously described EA 9313 + AC-130-2 lay-up process.

A flat aluminum plate mold was prepared for FML lay-up as previously described. The first pretreated aluminum alloy 2024-T3 layer was placed on the porous Nylon Release Peel Ply and evenly coated with Loctite® EA 9313 Epoxy adhesive using a foam paint brush (Figure 3.18). Two ShieldStrand® S woven glass fiber layers were carefully aligned on the wet metal sheet and wetted with the epoxy adhesive as described above (50/50 ratio by weight prior to consolidation). The process was repeated to create a FML with three composite layers (S-2 glass fibers + epoxy) sandwiched between four aluminum alloy 2024-T3 layers referred to as FML EA9313. The FML EA9313 was then covered with the porous Nylon Release Peel Ply, followed by the breather bleeder ply and finally aluminum foil. It was finally allowed to cure in the hydraulic press (Figure 3.17) at 82°C (180°F) for 60 min per manufacturer’s recommendation (Henkel Corporation Aerospace, 2013).
The analysis of the single step cured FML EA9313 revealed that the laminates did not stay fully aligned during the cure process. The slippery characteristics of the uncured epoxy, allowed the laminates to marginally slide amongst each other while the FML was consolidating within the press platens.

3.6.3. Improved Loctite® EA 9313 Epoxy FML Lay-up

To resolve the sliding laminates’ issue, another FML EA9313 was fabricated. Here the metal layers were cut with drilled alignment tabs at each end (Figure 3.18). The FML lay-up was like that described for FML EA9313 fabrication.

![Drilled tabs]

Figure 3.18 The application of Loctite® EA 9313 Epoxy on Al-2024-T3 using a foam paint brush.
The completed lay-up was secured in place by attaching an aluminum wire through the drilled holes of every metal layer as shown in Figure 3.19. This restricted the motion of the metal layers and eventually alleviated the laminate sliding issue.

Figure 3.19 FML EA9313 lay-up secured in place with alignment tabs and twisted aluminum wires.
Considering the limited ERAU supply of ShieldStrand® S woven glass fibers, this improvement method was first tested using inexpensive and readily available Bondo® Fiberglass Cloth (3M™ Company, Maplewood MN) purchased at Walmart for $4.27 per 8 ft². This trial experiment showed that the improvement method suggested above was enough to keep the laminates aligned. A composite only layer lay-up with Bondo® Fiberglass Cloth was also fabricated to later analyze its properties.

Hence a final lay-up was fabricated with ShieldStrand® S woven glass fibers following all of the steps mentioned above (Figure 3.20). The alignment tabs were then cut from the cured panel using the water lubricated tile saw. For comparison sake, this FML panel will be referred to as Improved FML EA9313.

Figure 3.20 Improved FML EA9313 after cure in the hydraulic press.
3.6.4. Final Tensile Samples

ASTM D3039/D3039M-17 was used as a guide when fabricating the tensile samples. The samples were 25.4 mm (1 in) wide by 254 mm (10 in) long according to the standard (ASTM International, 2017). Gripping tabs with width of 25.4 mm (1 in) and length of 76.2 mm (3 in) were made from aluminum alloy 6061-T651. This time, these tabs had a small bevel to more gradually apply load to the test section of interest. The bevel angle was 45º. This helped minimize stress concentration and avoid fracture at the grips that were previously experienced when there were no bevels. Three tensile samples were cut out of each fabricated FML panel following the process described in the previously described machining subsection.

It was also important to investigate the mechanical properties of the constituent elements of the FMLs. Hence three tensile samples were also cut from Al-2024-T3 sheets, as well as out of two press fabricated composite layers (ShieldStrand® S woven glass fibers and Bondo® Fiberglass). These properties will later be helpful to understand and predict the behavior/mechanical properties of the fabricated FMLs.

Before adhering gripping tabs, dimensions of all tensile test samples were recorded, and their densities calculated. A total of 15 tensile test samples were made.
3.7. Grip Pressure Test

The grip fracture experienced in the preliminary tensile test also motivated a minimum gripping pressure experiment. A tensile sample was made from an aluminum alloy 6061-T651 bar 25.4 mm (1 in) wide by 4.65 mm (0.18 in) thick and 254 mm (10 in) in length following the processes previously described. Rectangular gripping tabs 25.4 mm (1 in) wide and 76.2 mm (3 in) long, made from aluminum alloy 6061-T651 were adhered to the specimen using SilverTip Meltweld epoxy adhesive. The sample was pulled in tension using the Instron Material Testing Instrument (Figure 3.7), following the tensile test method stated previously. The gripping pressure was varied from 1.4 MPa (200 psi) to 6.9 MPa (1000 psi). After each grip pressure trial, the tab exteriors that had been burrowed into by the flat serrated grip faces were wet sanded (320 grit) to remove any marks. The test was repeated for five different gripping pressures. A plot of stress versus actuator displacement was made to summarize the results from the grip pressure tests.
3.7.1. Grip Pressure Test Results

From Figure 3.22, it can be observed that the test sample experience slipping at grip pressures of 1.4 MPa (200 psi) and 2.8 MPa (400 psi) with the latter being less noticeable. The stress/actuator displacement curves for a pressure range of 4.1 MPa (600 psi) to 6.9 MPa (1000 psi) were all aligned, meaning that there was no slipping experienced at this grip pressure range. Hence 4.1 MPa (600 psi) was found to be the minimum grip pressure required for tensile testing. To account for possible variations, the grip pressure used during the FML tensile tests was set slightly higher to 4.8 MPa (700 psi).

![Figure 3.22 Stress-actuator displacement response at various gripping pressures.](image-url)
3.8. Final Tensile Testing

The final tensile tests were performed on the Instron Material Testing Instrument (Figure 3.7), following the tensile test settings previously described. For FML test samples, the grip pressure was 4.8 MPa (700 psi). It was thought that the small thickness ~ 0.3 mm (0.012 in) of the composite layer and Al-2024-T3 tensile test samples might require extra gripping pressure, so the grip pressure was changed to 6.9 MPa (1000 psi) during their respective tensile tests.
4. Results

4.1. Properties of Aluminum Alloy Al-2024-T3 Sheets

All samples were tested in the metal’s rolling direction since they were laid as such during the FML fabrication process.

Figure 4.1 shows that all three Al-2024-T3 tensile samples had similar stress-strain behavior. However, Test 1 was the only sample that had an elongation to failure of 12.3% which was 0.3% greater than the previously reported spec sheet value (12%, Table 3.3). Edge damage may have led to slightly premature failure of the other two samples. The stress-strain curve clearly shows a change between the elastic and plastic deformation regions. The elastic region is seen to end at about 0.57% strain, this is the point at which the aluminum alloy 2024-T3 yielded. The 0.2% yield strength, \( \sigma_y \) was found to be approximately 340 MPa (49 ksi) while the ultimate tensile strength, \( \sigma_u \) was 476 MPa (69 ksi) which are very close to spec values. The Young’s modulus was 70.2 GPa (10.2 x 10^3 ksi), which is in line with reported values.
Figure 4.1 Stress-strain response of Al-2024-T3 tensile test samples.

4.2. Properties of the Composite Layer (ShieldStrand® S Woven Fibers)

Figure 4.2 shows all three ShieldStrand® S tensile samples had similar behavior, performing like a brittle material in most ways. Test 2 had a slight higher elongation at failure (3.4%). There was no evidence of plastic deformation, hence no yield strength, $\sigma_y$. The ultimate tensile strength, $\sigma_u$ was about 627 MPa (91 ksi).
Figure 4.2 Stress-strain response of ShieldStrand® S composite layer tensile test samples.

The load bearing capacity shakedown beyond ultimate is rather encouraging considering this jagged drop is likely caused by fibers fracturing within the material, yet the material holds together. In the absence of good damage tolerance, many composites would have immediately failed. Such shakedown was not observed in previous ERAU research.
4.3. Comparison of the Two Different Composite Layers

The composite layers made with Bondo® Fiberglass were also tested and observed to be substantially weaker and less stiff than those made with ShieldStrand® S woven glass fibers. The performance of these composite layers, obtained during the tensile tests are shown in Figure 4.3 with properties summarized in Table 4.1. From Figure 4.3, it can be observed that the ShieldStrand® S composite layer also shows much better damage resistance. This is evidenced by the gradual drop in tensile strength once the fibers started substantially breaking beyond ultimate. ShieldStrand® S reinforced composites perform much better than the Bondo® Fiberglass reinforced composites, albeit, at higher cost and they are not as readily available.

Figure 4.3 Stress-strain response of both types of composite layer tensile test samples.
Table 4.1 Properties of fabricated composite layers.

<table>
<thead>
<tr>
<th>Composite layers</th>
<th>Density g/cm³ (lb/in³)</th>
<th>Ultimate tensile strength, σu MPa (ksi)</th>
<th>Elongation at failure %</th>
<th>Young’s modulus GPa (Msi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bondo® Fiberglass</td>
<td>1.41 (0.0509)</td>
<td>367 (53)</td>
<td>2.2</td>
<td>16.7 (2.42)</td>
</tr>
<tr>
<td>ShieldStrand® S</td>
<td>1.46 (0.0527)</td>
<td>627 (91)</td>
<td>3.4</td>
<td>18.5 (2.68)</td>
</tr>
</tbody>
</table>

4.4. FML Tensile Test Results for Differing Adhesive (MA530 and EA 9313)

FML EA9313 were found to be relatively tougher than FML MA530. Both FMLs show similar stress-strain behavior; there was visible transition between the elastic zone and the plastic deformation zone. For FML MA530, that transition happened at approximately 0.6% elongation meanwhile for FML EA9313, it happened at around 0.5% elongation. These numbers are reasonable since they coincide with the yield strain of aluminum alloy 2024-T3 (the constituent metal in both FMLs). For the best FML MA530 sample, the 0.2% yield strength, σy, and ultimate tensile strength, σu, were 197 MPa (29 ksi) and 449 MPa (65 ksi) respectively. For the best FML EA9313 sample, these values were 230 MPa (33 ksi) and 532 MPa (77 ksi). FML EA9313 had the preeminent stiffness, strength and elongation at failure between these two FMLs.
Figure 4.4 Stress-strain response of both FML tensile test samples (MA530 and EA9313).

Observing Figure 4.4, FML EA9313 curves showed a gradual drop in tensile strength during fiber breakage while FML MA530 showed a nearly instantaneous fracture once the fibers broke. This implied that FML EA9313 had more damage resistant characteristics. Table 4.2 summarizes the FMLs’ measured and calculated parameters. Specific properties are obtained by dividing any property by density.
Table 4.2 Measured and calculated parameters of FML MA530 and FML EA9313.

<table>
<thead>
<tr>
<th>FML</th>
<th>Panel thickness (mm (in))</th>
<th>Density g/cm³ (lb/in³)</th>
<th>σy MPa (ksi)</th>
<th>σu MPa (ksi)</th>
<th>Elongation at failure %</th>
<th>E GPa (Msi)</th>
<th>Specific stiffness (10^6 \text{ m}^2\text{s}^{-2})</th>
</tr>
</thead>
<tbody>
<tr>
<td>FML MA530</td>
<td>3.86 (0.15)</td>
<td>1.78 (0.0643)</td>
<td>~197</td>
<td>449 (65)</td>
<td>3.54</td>
<td>32.8 (4.8)</td>
<td>18.4</td>
</tr>
<tr>
<td>FML EA9313</td>
<td>2.67 (0.11)</td>
<td>2.07 (0.0748)</td>
<td>~230</td>
<td>532 (77)</td>
<td>3.74</td>
<td>46.5 (6.7)</td>
<td>22.5</td>
</tr>
</tbody>
</table>

4.5. Comparison between S.A. Rudradat (MSAE 2013) FML and FML EA9313

In her thesis research, Rudradat (2013) fabricated a FML with the same constituents (3 mm thick Al-2020-T3 alloy, ShieldStrand® S woven fibers and Loctite® EA 9313) as those used in this study. The metal layers used in her research were also subjected to the same surface pre-treatment. However, there were some differences in the curing processes used. Vacuum bagging techniques were used in her study, meanwhile this study focuses on the use of a hydraulic press and high pressure during consolidation.

Observing the stress-strain behavior of each of those FML in Figure 4.5 there were some similarities in the curve patterns for both materials. The curves showed three distinct sections; yielding, initial failure and maintained stress after failure. Note that the maintained stress was only recorded for one sample of FML EA9313 due to de-bonding of the strain gauge during the initial failure for the other two samples, however this maintained stress was present and observed by the author during all tests. There was no clear explanation for the maintained stress observed after failure.
From the tensile test results, FML EA9313 showed an overall improvement in performance even though it had a slightly lower Young’s modulus. More noticeable, is the increased damage tolerance near ultimate which was further confirmed upon inspection of the failed samples. Properties are summarized in Table 4.3.

Table 4.3 Mechanical properties of S.A. Rudradat FML and FML EA9313.

<table>
<thead>
<tr>
<th>FML</th>
<th>Panel thickness mm (in)</th>
<th>Density g/cm$^3$ (lb/in$^3$)</th>
<th>UTS, $\sigma_u$ MPa (ksi)</th>
<th>Elongation at failure %</th>
<th>Young’s modulus GPa (Msi)</th>
<th>Specific stiffness $10^6$ m$^2$s$^{-2}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>S.A. Rudradat</td>
<td>3.04 (0.120)</td>
<td>2.32 (0.0838)</td>
<td>485 (70)</td>
<td>3.65</td>
<td>52.5 (7.6)</td>
<td>22.6</td>
</tr>
<tr>
<td>FML EA9313</td>
<td>2.67 (0.105)</td>
<td>2.07 (0.0748)</td>
<td>532 (77)</td>
<td>3.74</td>
<td>46.5 (6.7)</td>
<td>22.5</td>
</tr>
</tbody>
</table>
Considering the initial failure zone, both FMLs revealed some damage tolerance characteristics. This is evident by the gradual decrease in tensile strength at the start of fiber breakage as opposed to the more usual, instant catastrophic failure. However, FML EA9313 had an extended smoother curve before final failure. This implied that it could resist the applied load much longer after the fibers started breaking, hence there was an overall improvement in the damage tolerance property of these FMLs.

Further observation of the failed samples showed that S.A. Rudradat FMLs experienced significantly more delamination when compared to FML EA9313. Both FMLs experienced fiber breakage; it was the dominant mode of failure in FML EA9313 rather than delamination. Figure 4.6 and Figure 4.7 clearly shows these differences.

Figure 4.6 Delamination experience in previously fabricated FML (Rudradat, 2013).
Figure 4.7 Fiber break/detachment and substantially less delamination experienced by FML EA9313.

4.6. Comparison between FML EA9313 and Improved FML EA9313

To keep the metal layers and composite layers aligned during the consolidation process in the hydraulic press, another FML was fabricated with a process described previously named Improved FML EA9313. This slight change in the fabrication process had a noticeable impact on the overall performance of the material. The stress-strain curves are shown in Figure 4.8 along with those of the previously fabricated FML EA9313. There was an improvement in ultimate tensile strength, $\sigma_u$ from 532 MPa (77 ksi) to 592 MPa (86 ksi) which is an approximately 11.3% increase. The elongation at failure improved from 3.74% to 3.93% which is roughly a 5% increase. Yielding happened at approximately 0.55% elongation and the 0.2% yield strength, $\sigma_y$ improved from 230 MPa (33 ksi) to 247 MPa (36 ksi) which was a 7.4% increase. It is assumed that better alignment of the fibers, more parallel to the loading direction have led to these improvements.
Figure 4.8 Stress-strain response of EA9313 bonded FML tensile test samples.

Observing the initial failure zone (Figure 4.8) revealed that both FMLs showed damage tolerant behavior. All six stress-strain responses had an extended smoother curve before final failure hence indicating that they could resist the applied load much longer after the start of fiber breakage.
Figure 4.9 Fiber break and negligible delamination experienced by Improved FML EA9313.

Observing Figure 4.9, Improved FML EA9313 experienced less fiber detachment during failure when compared to FML EA9313 (Figure 4.7). Yet, both FMLs still experience reduced delamination. This was nearly negligible for Improved FML EA9313.

4.7. Comparison Between Improved FML EA9313 and GLARE 4A-4/3-0.3

The overall aim of this study was to compare simpler and less expensive FMLs with the commercially available FML, GLARE. GLARE 4A-4/3-0.3 was the focus here, and its tensile test data was obtained from previous research (Benedict, 2012). It was tested in both its weak and strong directions. The weak direction has 3 unidirectional fiber layers aligned along the loading direction while the strong direction has 6. Amongst all of the fabricated FMLs in this study, Improved FML EA9313 had the most outstanding performance characteristics, hence it will be the only material used for additional comparison.
The stress-strain response of both GLARE 4A-4/3-0.3 and Improved FML EA9313 show two distinct regions; the region before the yield point (elastic region) and the region after the yield point (plastic deformation). The first thing to notice in Figure 4.10 is that the stress-strain curves for Improved FML EA9313 lie between the weak and strong stress-strain curves for GLARE 4A-4/3-0.3 as anticipated owing to the differing number of fiber layers oriented along the loading direction. All five curves experienced yielding at ~ 0.55% elongation. This yield point coincides with the yield point in Al-2024-T3.

![Stress-strain response of GLARE 4A-4/3-0.3 and Improved FML EA9313.](image)

Figure 4.10 Stress-strain response of GLARE 4A-4/3-0.3 and Improved FML EA9313.
Looking at Figure 4.10, the tensile test results revealed that Improved FML EA9313 outperformed GLARE 4A-4/3-0.3 (weak direction) in all categories except elongation at failure (these parameters can be seen in Table 4.4). This is partially due to the fact that GLARE is made with unidirectional S-2 glass fibers which have a slightly higher strength and elongation at failure when compared to the ShieldStrand® S woven glass fibers used in this study. More importantly, the fibers used in GLARE are unidirectional, all reasonably straightly aligned within each composite layer, and not woven like those of the present study. Weaving tends to slightly pre-stress the fibers by pre-bending them. If one ends up tensioning an already bent tensile member, its stiffness would not be as linear and portions of it would be tensile/compression pre-stressed causing them to prematurely fail. This is likely what caused the slightly impaired performance of the present FMLs. Satin weaves, as many as 4 or more wefts over one warp can get more of the fibers straight over longer distances resulting in improved mechanical performance when compared to plain square weaves made from the same fibers. Weaving can also scratch the fibers, hence reducing their strength.
Table 4.4 Measured and calculated parameters of GLARE 4A-4/3-0.3 and Improved FML EA9313.

<table>
<thead>
<tr>
<th>FML</th>
<th>Panel thickness mm (in)</th>
<th>Density g/cm³ (lb/in³)</th>
<th>σy MPa (ksi)</th>
<th>σu MPa (ksi)</th>
<th>Elongation at failure %</th>
<th>E GPa (Msi)</th>
<th>Specific stiffness 10⁶ m²/s²</th>
</tr>
</thead>
<tbody>
<tr>
<td>GLARE 4A-4/3-0.3 (strong)</td>
<td>2.45 (0.096)</td>
<td>2.30 (0.0831)</td>
<td>~270 (39)</td>
<td>880 (128)</td>
<td>4.34</td>
<td>52 (7.5)</td>
<td>22.6</td>
</tr>
<tr>
<td>GLARE 4A-4/3-0.3 (weak)</td>
<td>2.45 (0.096)</td>
<td>2.30 (0.0831)</td>
<td>~220 (32)</td>
<td>510 (74)</td>
<td>4.10</td>
<td>44 (6.3)</td>
<td>19.1</td>
</tr>
<tr>
<td>Improved FML EA9313</td>
<td>2.69 (0.105)</td>
<td>2.12 (0.0766)</td>
<td>~247 (36)</td>
<td>592 (86)</td>
<td>3.93</td>
<td>49.4 (7.2)</td>
<td>23.3</td>
</tr>
</tbody>
</table>

Though GLARE 4A-4/3-0.3 (strong direction) had excellent mechanical properties, further analysis of the samples after failure revealed significant delamination (Figure 4.11). Meanwhile, improved FML EA9313 showed no delamination and mostly failed due to fiber breakage and fracture in some metal layers. The integrity of the surfaces away from the fracture zone in improved FML EA9313 essentially stayed unharmed (Figure 4.9). Pervasive delamination of various grades of GLARE has been an ongoing observation during testing at ERAU.
Figure 4.11 Delamination experienced by GLARE 4A-4/3-0.3 after a tensile test along its strong direction (left) (Benedict, 2012) and behavior of Improved FML EA9313 after the tensile test (right).

4.8. Comparison between Improved FML EA9313 and its constituents

The performance characteristics of most composite materials are entirely dependent on and/or limited by the properties of each of their constituents, along with geometry and constituent interaction. In FMLs, these constituents are the metal and composite layers. With the composite layer being the brittle constituent, it is a limiting factor when considering the elongation at failure for the FML. Figure 4.12 shows the stress-strain behavior of Improved FML EA9313 and its constituents. From these results, it was observed that the FML properties were very close to those of ShieldStrand® S composite layer, see Table 4.5. The elongation at failure of the FML was 3.93%, this was higher than its constituent composite layer hence the increase was due to the ductility in the metal layers as well as the strength and high strain toughness of the EA 9313 adhesive.
Figure 4.12 Stress-strain response of Improved FML EA9313 and its constituents.

Table 4.5 Measured and calculated parameters of Improved FML EA9313 and its constituents.

<table>
<thead>
<tr>
<th></th>
<th>Panel thickness</th>
<th>Density g/cm³ (lb/in³)</th>
<th>σ_y MPa (ksi)</th>
<th>σ_u MPa (ksi)</th>
<th>Elongation at failure %</th>
<th>E GPa (Msi)</th>
<th>Specific stiffness 10⁶ m²s²</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-2024-T3</td>
<td>0.3 (0.012)</td>
<td>2.75 (0.0994)</td>
<td>~337 (48.9)</td>
<td>473 (69)</td>
<td>12</td>
<td>70.2 (10)</td>
<td>25.5</td>
</tr>
<tr>
<td>ShieldStrand® S Composite</td>
<td>0.48 (0.019)</td>
<td>1.47 (0.0531)</td>
<td>-</td>
<td>627 (91)</td>
<td>3.4</td>
<td>18.4 (2.7)</td>
<td>12.5</td>
</tr>
<tr>
<td>Layer</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Improved FML EA9313</td>
<td>2.69 (0.106)</td>
<td>2.12 (0.0766)</td>
<td>~247 (36)</td>
<td>592 (86)</td>
<td>3.93</td>
<td>49.4 (7.2)</td>
<td>23.3</td>
</tr>
</tbody>
</table>
5. Mechanical Properties Calculations (Rule of Mixtures)

The rule of mixtures was used to compute various mechanical properties for the fabricated FMLs. The properties obtained were Young’s modulus, 0.2% yield strength and failure strength (ultimate tensile strength) for FML MA530, FML EA9313 and Improved FML EA9313.

5.1. Young’s Modulus Calculations

The Young’s modulus of the FML is a volume weighted mixture of the Young’s moduli of its constituent elements as shown in the following equations.

\[
E_{FML} = E_c V_c + E_{met} V_{met}
\]  

(1)

Where:

- \( E_{FML} \) - Young’s modulus of the fiber metal laminate
- \( E_c \) - Young’s modulus of the composite layer (S-2 glass fibers + epoxy)
- \( V_c \) - Volume fraction of the composite layer
- \( E_{met} \) - Young’s modulus of the metal layer
- \( V_{met} \) - Volume fraction of the metal layer

The volume fraction of the metal layer is calculated by finding the ratio between the thickness of all metal layers and the total thickness of the FML. These thicknesses were initially measured prior to tensile testing. Four aluminum layers of 0.3 mm (0.012 in) thickness were used in every fabricated FML.
\[ V_{met} = \frac{n_{Al}t_{Al}}{n_{Al}t_{Al} + n_{c}t_{c}} \]  

(2)

Where:

- \( n_{Al} \) - Number of aluminum layers
- \( t_{Al} \) - Thickness of the aluminum layer
- \( t_{c} \) - Thickness of the composite layer

The volume fraction of the composite layer is calculated as follows

\[ V_{c} = 1 - V_{met} \]  

(3)

Three composite layers (ShieldStrand\textsuperscript{®} S woven glass fibers + EA 9313 epoxy) were used in all fabricated FMLs. These layers were found to be ~0.48 mm (0.019 in) thick.

Hence the volume fractions were calculated as follows

\[ V_{met} = \frac{4 \times 0.3}{(4 \times 0.3) + (3 \times 0.48)} = 0.45 \]  

(4)

\[ V_{c} = 1 - V_{met} = 1 - 0.45 = 0.55 \]  

(5)

Obtained from previous tests, 70.2 GPa and 18.4 GPa were used as the Young’s moduli for the aluminum 2024-T3 alloy and the composite layer respectively. The 0.2% offset yield strength used for calculations was the stress at 0.75% strain from the aluminum alloy stress-strain curve which was 337 MPa (49 ksi). Recall, the proportional limit was at 0.55% strain for the Al-2024-T3 alloy along the rolling direction.

\[ E_{FML} = E_{c}V_{c} + E_{met}V_{met} = 18.4(0.55) + 70.2(0.45) = 41.7 \text{ GPa} \]  

(6)
5.2. 0.2% Yield Strength and Failure Stress Calculations

The 0.2% Yield Strength of the FML is a mixture of the Young’s moduli of its constituent elements as shown in eqn.7. Values obtained in eqn. 4 and eqn. 5 will be substituted in the following computations.

\[ \sigma_{yFML} = \sigma_c V_c + \sigma_{ymet} V_{met} \]  

(7)

Where:

\( \sigma_{yFML} \) - 0.2% yield strength of the fiber metal laminate

\( \sigma_c \) - Stress in the composite layer at the required instant

\( \sigma_{ymet} \) - 0.2% yield strength of the metal layer

The failure stress can also be roughly estimated using the following expression

\[ \sigma_{FML\,fail} = \sigma_c V_c + \sigma_{met\,fail} V_{met} \]  

(8)

Where:

\( \sigma_{FML\,fail} \) - Failure stress of the fiber metal laminate

The 0.2% offset yield strain for all FMLs is ~0.75% (obtained from test results, it was the average yield point for all FMLs) and it is substituted in the expression for FML yield strength calculation. At 0.75%, the stress in the composite layers was 144.6 MPa (21 ksi), obtained from their stress-strain curves.

\[ \sigma_{yFML} = \sigma_c V_c + \sigma_{ymet} V_{met} = 144.6 \times (0.55) + 337 \times (0.45) = 231.2 \, MPa \]  

(9)
The ultimate strain of the composite layers was 3.4%. For calculation purposes, the ultimate tensile stress in the aluminum was determined by observing its stress-strain behavior and obtaining the stress equivalent to the composite layer’s ultimate strain. It was found to be 445 MPa (64.5 ksi)

\[ \sigma_{FML\ fai} = \sigma_c V_c + \sigma_{met\ fail} V_{met} = 627 (0.55) + 445 (0.45) = 545 \text{ MPa} \quad (10) \]

**FML MA530**

The average overall thickness for FML MA530 was 3.86 mm (0.15 in). From the FML MA530 tensile test, \( E_{FML}, \sigma_{yFML} \) and \( \sigma_{FML\ fai} \) were 32.8 GPa, 197 MPa and 449 MPa respectively. These experimental values were compared to the estimated values (from rule of mixtures) and the percentage differences were computed. These differences were 21%, 14.8%, and 17.6% respectively.

**FML EA9313**

The average overall thickness for FML EA9313 was 2.67 mm (0.105 in). From the FML EA9313 tensile test, \( E_{FML}, \sigma_{yFML} \) and \( \sigma_{FML\ fai} \) were 46.5 GPa, 230 MPa and 532 MPa respectively. The respective percentage differences were 11.5%, 0.5%, and 2.4% respectively.

**Improved FML EA9313**

The average overall thickness for FML EA9313 was 2.69 mm (0.106 in). From the FML EA9313 tensile test, \( E_{FML}, \sigma_{yFML} \) and \( \sigma_{FML\ fai} \) were 49.4 GPa, 247 MPa and 592 MPa respectively. The respective percentage differences were 18.5%, 6.8%, and 8.6% respectively.
The laminates bonded together with Loctite® EA 9313 Epoxy had the smallest percent differences when comparing the estimated properties (rule of mixtures) and the experimentally obtained properties. These are summarized in Table 5.1.

Table 5.1 Comparison between estimated and experimental mechanical properties of the fabricated FMLs.

<table>
<thead>
<tr>
<th></th>
<th>Young’s modulus</th>
<th>0.2 % Yield strength</th>
<th>Failure stress</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Value GPa</td>
<td>% Difference</td>
<td>Value MPa</td>
</tr>
<tr>
<td><strong>Estimated</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FML MA530</td>
<td>41.7</td>
<td>-231</td>
<td>231</td>
</tr>
<tr>
<td>FML EA9313</td>
<td>46.5</td>
<td>+230</td>
<td>230</td>
</tr>
<tr>
<td>Improved FML EA9313</td>
<td>49.4</td>
<td>+247</td>
<td>247</td>
</tr>
</tbody>
</table>
6. Microscopy

To effectively compare the structural arrangement of GLARE and the fabricated FMLs, a close-up investigation of their cross-sections was required. Careful surface preparations (grinding, polishing and etching) were needed to reveal important details not seen with the naked eye.

6.1. Metallographic Samples Preparation

Metallographic samples were made from GLARE 4A-4/3-0.3 panels previously obtained from FMLC by a former ERAU student (Olaniyan, 2010) along with one fabricated FML panel (Improved FML EA9313). Planar samples of approximately 10 by 10 mm (0.4 by 0.4 in) were cut using the previously described benchtop sectioning machine. Two perpendicular planar samples were obtained from each panel (four samples in total). The samples were mounted in epoxy in 30.6 mm (1.25 in) inner diameter plastic mold SamplKups™ (Buehler) cups. An LC Epoxy (long cure) kit (Leco Corporation) was used. It is a two-part kit with a mix ratio of 14 g hardener to 100 g resin, it has a working time of 45 min and a cure time of 4 - 8 hr (Leco Corporation, 2013).

The SamplKups™ were first cleaned with methanol and then coated with GUNK silicone spray lubricant (Radiator Specialty Company, Indian Trail, NC) to ease demolding after epoxy cure. Metal specimen mounting clips were attached to the planar sections to keep the samples vertically aligned in the epoxy. The planar samples were then placed in the mold cups (with desirable cross-section facing down) and the mixed epoxy was poured into the cups until the planar samples were fully covered. A graphite pencil written paper label was inserted in the epoxy to aid identification of each sample after cure. The samples were left undisturbed to cure at room temperature for 8 hr.
6.2. Procedure

The bottom face of the metallographic samples was then sanded on a water lubricated HandiMet® 2 roll grinder (Buehler), Figure 6.2. 240, 320, 400 and 600 grit SiC paper were used; each sample was passed about 5 times on each grit going from the coarsest to finest. They were then semi-automatically polished for approximately 1 hr on a soft nap Buehler MicroCloth® PSA (Figure 3.6) lubricated with Buehler de-agglomerated alpha alumina 0.3 μ and tap water. During the polishing process, the surface was periodically checked for scratches under the stereozoom optical microscope (Figure 3.13). The end of the polishing process was defined by a mirror-like surface free of any substantial scratches. The polished samples where then placed in a beaker filled with a cleaning solution (Extreme Simple Green®, Sunshine Makers Inc., Huntington Beach, CA) and then cleaned ultrasonically for about 30 minutes. Figure 6.1 shows one of the metallographic samples made.

Figure 6.1 Metallographic sample mounted in epoxy and etched.
To observe the microstructure of the aluminum alloy layer in the FMLs, the metallographic samples were etched. The etchant solution used was Kroll’s reagent which is suited for Al-Cu alloys. The etchant was composed of 92 ml of distilled water, 6 ml of nitric acid and 2 ml of hydrofluoric acid. (Petzow, 1999). The samples were etched for about 15 sec and the quickly rinsed with tap water followed by a methanol rinse and finally air dried. They were first observed under the metallurgical optical microscope (Figure 3.13). The top surface of each sample was then gold coated using a Cressington Sputter Coater Model 108 (Cressington Scientific Instruments, Watford, England, UK) to make the surface conductive. A copper tape was then attached along the length of the samples (this is done to facilitate the flow of electrons) and they were finally viewed in the scanning electron microscope (Quanta 650, FEI, Hillsboro, OR) (Figure 6.3).
6.3. Microscopy Results/Analysis

Various images were obtained from the optical microscope and the scanning electron microscope (SEM). The accelerating voltage in the SEM ranged from 12.5 KV to 30 KV and the working distance ranged from 10 mm to 12 mm. Specific details for each SEM picture taken can be seen on the bottom of the images.

Figure 6.4 displays the lay-up arrangement in GLARE 4A-4/3-0.3. It is made up of 3 composite layers (S-2 glass fibers + epoxy) sandwiched in between aluminum layers. It clearly shows that each composite layer began with 3 unidirectional glass fiber prepreg layers. On the left picture, six composite layers are pointing out of page meanwhile on the
right picture only three composite layers are pointed out of page. The former is said to be the strong direction while the latter is said to be the weak direction. Note that these two cross-sections were both obtained from the same GLARE 4A-4/3-0.3 panel with one being cut in a direction perpendicular to the other (longitudinal vs. transverse cut).

![Composite Layers Diagram](image)

Figure 6.4 Microscopic view of GLARE 4A-4/3-0.3, showing unidirectional fiber arrangement in weak (left) and strong directions (right).

Improved FML EA9313 is also made up of three composite layers (S-2 glass fibers + epoxy) sandwiched in between aluminum layers. However, the composite layers here are made up from two layers of woven glass fibers. As can be seen in Figure 6.5, the fiber rows are more difficult to see than they are for GLARE. Approximately equal amounts of glass fibers are swimming within the epoxy in both longitudinal and transverse cuts, hence the cross-section is said to be somewhat symmetrical. This therefore results in little stiffness and strength discrepancy between the two directions. Some porosity is observed within the composite layers in both directions.
Figure 6.5 Microscopic view of Improved FML EA9313 (transverse cut on left and longitudinal cut on right), showing no clear distinction of glass fibers within the composite layers.

Figure 6.6 shows a closer view into the composite layer of both GLARE 4A-4/3-0.3 (left) and Improved FML EA9313 (right). The picture was taken at about 600X magnification. The glass fibers in GLARE were seen to be much aligned when compared to those in the fabricated FML of this study, however the glass fibers in the latter were more densely packed together. The densely packed glass fibers in the fabricated FML was an indication that the use of the hydraulic press during consolidation effectively squeezed out all excess adhesives from the glass fibers. There is evidence of porosity and voids within both FMLs.
Figure 6.6 Microscopic view of the composite layers in GLARE 4A-4/3 (left) and in Improved FML EA9313 (right).

The aluminum layer of both GLARE 4A-4/3-0.3 (left) and Improved FML EA9313 (right) was observed in an optical microscope at 200X magnification (Figure 6.7).

Figure 6.7 Microscopic view of the aluminum layer in GLARE 4A-4/3 (left) and in Improved FML EA9313 (right).

Figure 6.8 shows a closer view into the aluminum alloy 2024-T3 layer in Improved FML EA9313. The picture was taken at about 2200X magnification. The microstructure in every aluminum layer revealed elongated grains, this implies that both metals were rolled into sheets as expected. The rolling direction is the direction in which the grains seem more elongated.
Figure 6.8 Microscopic view of Al-2024 T3 layer in Improved FML EA9313.
7. Discussion

7.1. Tensile Tests

Reducing the grip pressure during the test as well as using tabs with a slight bevel angle influenced the position of final failure. Figure 7.1 shows Improved FML EA9313 tensile test samples after failure. It was observed that two of the three samples experienced failure near the center of the gauge length. Hence implying that the high grip pressure and non-beveled tabs used early on introduced concentrated stresses on the test samples. These stresses can eventually be reduced by using tabs with progressive bevel angle towards the gauge area.

![Figure 7.1 Improved FML EA9313 samples after tensile failure.](image)

7.2. Failure Analysis

All of the fabricated FMLs as well as GLARE 4A-4/3-0.3 experienced similar stress-strain behavior. The aluminum layers were the constituents responsible for the yielding region observed on those curves. During yielding, aluminum layers uphold the
fibers and allow for greater strains (elongation at failure) in the FML. The fibers provide high ultimate tensile strengths but lesser amount of strains (Auffret & Gennai, 2001). Delamination was mostly observed in GLARE 4A-4/3-0.3 (Figure 7.2) hence implying that the strength and high strain to failure toughness of the adhesives used in the fabricated FMLs resisted delamination (Vlot, 2001).

Figure 7.2 Delamination of GLARE 4A-4/3-0.3 (Benedict, 2012).

The composite (S-2 glass fibers + epoxy) test samples displayed quite different failure characteristics. Close up images of failed composite samples are shown in Figure 7.3 and Figure 7.4.
7.3. Microstructure Analysis

The microstructure revealed that the composite layers in GLARE 4A-4/3-0.3 were found to be better aligned when compared to those in the fabricated FMLs. This was the main explanation for GLARE’s higher strain at failure. This strain however was not higher than the reported average failure strain (shown in Table 3.2) of its constituent S-2 glass fibers. There was a 25% difference between these values, with this said, GLARE 4A-4/3-0.3 does not appear to attain its peak performance characteristics.
The fibers in the fabricated FMLs were observed to be more densely packed together when compared to those in GLARE 4A-4/3-0.3. Hence the former had less epoxy adhesives within its laminate leading to a further reduction in weight. When comparing their mass densities, there was a 7.8% difference, with GLARE 4A-4/3-0.3 having a slightly higher density.

Even though the fibers in Improved FML EA9313 were not aligned, it was found to outperform its constituent composite layer in elongation at failure and there was only a 5.6% difference between their ultimate tensile strength, FML being slightly stronger. This indicates that with a few further improvements in the fabrication process, these FMLs could have potential to be used in future engineering applications. Their simplicity, lower cost, damage tolerance and delamination resistance as compared to GLARE are very attractive from a commercial applications point of view.
8. Conclusion

From the test results and observations made throughout this study, the following can be concluded:

1. FMLs fabricated through vacuum bagging process do not yield the most favorable performance characteristics.
2. The mechanical properties of fabricated FMLs were compared to those of GLARE 4A-4/3-0.3 and test results indicated similar stress-strain behavior.
3. GLARE exhibited higher elongation at failure when compared to the fabricated FMLs. The unidirectional fibers in GLARE had an elongation at failure greater than 4.5% meanwhile the woven fibers used in the FML had an elongation of 3.4%. Hence the failure strain of fibers is a limiting factor in FMLs strain performance. Fibers in GLARE are unidirectional as compared to woven (in ERAU FMLs), further improving its performance.
4. The use of the hydraulic press during consolidation substantially improved FMLs properties compared to prior ERAU fabricated FMLs.
   a. Much less delamination was observed. This was a remarkable improvement since several grades of the commercially available material GLARE always experience delamination at failure.
   b. Higher ultimate tensile strength was observed, and it was found to outperform GLARE 4A-4/3-0.3 (in its weak direction).
   c. Greater elongation at failure were also observed and this property outperformed its constituent composite layer (ShieldStrand® S woven glass fibers + EA 9313 epoxy).
5. The absence of delamination after failure implied that the adhesives used in the fabricated FMLs had enough strength and strain to failure toughness to avoid such occurrence.

6. Metallographic samples for both fabricated FMLs and GLARE 4A-4/3-0.3 were made and observed using a scanning electron microscope. These observations revealed the following:
   a. Porosity and voids were present throughout the FMLs. The voids in GLARE were found to be slightly smaller.
   b. The composite layers in GLARE were found to be better aligned when compared to those in the fabricated FMLs. However, the fibers in the latter were found to be more densely packed.

7. The alignment (unidirectional or woven fibers) and arrangement of fiber laminates within the FMLs has a great impact on the material properties and their performance characteristics.

8. Due to the simpler fabrication processes, the cost associated with the FMLs in this study is lower than that of GLARE.

9. The densities of the fabricated FMLs were lower than the density of GLARE 4A-4/3-0.3.

10. Many design and process variables could be changed to improve FMLs properties; the beauty of composites. From this study, some of those variables were found to be: different adhesives and surface pre-treatment, alignment of fiber layers within the FML and improved fabrication processes.
9. Recommendations

- Improve the FMLs fabrication to ensure that the glass fibers + epoxy layers stay aligned with the metal layers and then perform tests to see how the material properties improve.
- Investigate the impact properties of the fabricated FMLs.
- Investigate the effect of strain rate on the FMLs material properties.
- Investigate the effect of reducing porosity and voids within FMLs.
- Fabricate FMLs with unidirectional fibers using the inexpensive ERAU approaches.
REFERENCES


