

POLITECNICO DI TORINO

Corso di Laurea Magistrale in Ingegneria dell'Autoveicolo

Master Degree Thesis

Effect of Cyclic Corrosion on Static Strength of Composite/Aluminum Double Lap Joints



Supervisor:

Prof. Giovanni Belingardi

Prof. Sayed Nassar

Candidate:

Chao Yang

August 2019

Effect of Cyclic Corrosion on Static Strength of Composite/Aluminum Double Lap Joints

©Copyright Chao Yang 2019

All rights reserved

Acknowledgements

First, Thanks to my supervisor in PoliTo, Prof. Belingardi. You picked me into this program and your trust was my best motivation. It was a pleasure to work with you in the last 6 months. Your suggestions and instructions were crucial to my work. This program has changed my life totally and Thank you for giving me this opportunity.

Next, I would like to go with all the people I met in Oakland University.

Thank you, Prof. Nassar. Thank you for your trust and help during my experience in Oakland University. You taught me so much about life and study. There is a Chinese saying “give a man a fish and you feed him a day, teach a man how to fish and you feed him for a lifetime”. You were the man who taught me how to fish. I will be always grateful for what I have learnt from you.

Thanks to Hugh Foran and Continental Structural Plastic. You guys helped me out when I was struggling with my experimental materials. Mr. Foran, you are so nice and knowledgeable. I would always remember the first time when I went to your company. You opened a door to composite materials for me. Thank you for your help and donation. Hope we can have more cooperation in the future.

Thanks to Marco Gerini Romagnoli. My poor language cannot explain how important your help and support mean to me. It was you that helped me settle down when I just got there. You showed me US first. Because of you and your “Big Buick”, I could have my apartment, car and furniture easily. Thanks also for your help on my project. As I told you more than once, you lighted a fire when I was walking in dark at the beginning of this research. I really enjoyed working with you.

What I learned from you was probably more than what I learned in university these years. You showed me how to be an engineer and how good an engineer could be. Thanks for the fun we had and sorry if I bothered you sometimes with my stupid questions.

Thanks to Morgan Szczepaniak. You are my first American friend. Because of you and Marco, I could have opportunities to live as a local. Thanks to your family as well for the cottage and the best cheese burgers I have ever had. It has been a pleasure to spend six months with you and please tell Alba, I will always miss her.

Thanks to Matteo Spano, my coolest roommate. It was surprising that we liked the same kind of stuff. Those good restaurants we had tried together saw our friendship. Thanks for driving me every day. You were always nice even sometimes you needed to go back to OU to pick me up. The Italian food you made was much better than that I had in Italy. It was amazing to live with you. Best wishes for you and your family

Thanks to Tianwu Li, you were always there when I needed your help. Matt, you helped out when I did my test. Hope you will have a good time when you go to Italy next year. Shraddha, you always gave me good suggestions when I had any problem. Thanks to you guys in FAJRI. We are like a big family. Go, FAJRI!

Thanks to PoliTo, Edisu, FAJRI, OU and FCA. It was you that gave me this chance and supported me financially. With these help, I could focus on my study without having nay expense problem. It is clear that you have changed my life. Thank you for your fair help and support.

Last but not least, Thanks to my girlfriend and my family.

Lin Chang, I love you. You are always with me. When I am happy, you are the one who I can share with. When I am sad, you are the one who can cheer me up. You will never realize how important and how nice you are. Thanks god for letting us meet together and our relationship is the best gift I have ever received.

Dad and mom, it is your love that encourages me all the time. We Chinese are shy and usually don't say love aloud, but I want you to know I will love you forever. Before I came abroad, I didn't realize how important home meant to me. Now I notice that home is all my life. Only the place where you are can be called home. Your pride of me is my best reward.

Yours sincerely

Chao Yang

Aug. 2019

Abstract

This study investigates effect of cyclic corrosion on the static strength of double lap joints (DLJs) that are either bonded-only, bolted-only, or hybrid bonded-and- bolted. Carbon-Fiber-Reinforced thermoplastic composite material is bonded to Aluminum Alloy 6060 using either polyurethane-based or epoxy-based structural adhesives. Bolting is achieved using an M4-0.7 Class 8.8 threaded fasteners that are properly preloaded. Treatment of bonded surfaces include abrasion blasting, followed by ultrasonic solvent cleaning and roughness measurement using Wyko optical profilometer. Assembled DLJs are placed in a cyclic corrosion (salt-fog) test chamber following standard GMW 14872 3-stage cyclic corrosion test. Joints are pulled out for tensile-shear testing and visual inspection after 1, 7, 14, 22, and 30 days of exposure to cyclic corrosion. Results, discussion, observations, and conclusions are provided.

Table of Contents

Acknowledgements	III
Abstract	VI
List of Figures	IX
List of Tables	XIII
Nomenclature	XIV
Glossary	XV
1 Introduction and Literature Survey	1
1.1 Introduction.....	1
1.2 Literature Survey	4
1.2.1 Composite materials classification	4
1.2.2 Adhesive bonding in the automotive field	5
1.2.3 Double lap joints.....	6
1.2.4 Cyclic corrosion testing	7
1.2.5 Relevant research.....	8
1.2.6 Research objective and methodology	11
2 Experimental Setup and Test Procedure.....	12
2.1 Joint design and manufacturing.....	12
2.1.1 Substrate materials.....	12
2.1.2 Double lap joints geometry	14
2.1.3 Joining methods	16
2.2 Cyclic corrosion test.....	23
2.2 Static tensile test	26
2.3 Failure mode inspection	27
3 Results and Discussion	28
3.1 Mechanical behavior of baseline joints	28
3.1.1 Mechanical behavior of baseline bonded-only joints.....	28
3.1.2 Mechanical behavior of baseline bolted-only joints	30
3.1.3 Mechanical behavior of baseline hybrid bonded/bolted joints.....	31

3.2	Effect of corrosion cycles on Joints Static LTC	33
3.2.1	Effect of cyclic corrosion on Uralane bonded-only DLJs.....	33
3.2.2	Effect of cyclic corrosion on Epibond bonded-only DLJs	37
3.2.3	Effect of cyclic corrosion on bolted-only DLJs	39
3.2.4	Effect of cyclic corrosion on hybrid bonded/bolted joints	41
3.3	Effect of different joining methods.....	44
3.3.1	Adhesive bonding-only joints	44
3.3.2	Uralane vs Uralane hybrid bonding and bolting joints	45
3.3.3	Bonding-only vs bolting-only	47
3.4	Effect of bolts on corrosion	49
4	<i>Conclusion</i>	51
5	<i>Continuation work</i>	53
	<i>References</i>	55
	<i>Appendix.....</i>	60

List of Figures

Figure 1-1: Double Lap Joints configuration (bonded-only)	7
Figure 2-1: Double Lap Joints geometry	15
Figure 2-2: Model of DLJs (Hybrid bonded/bolted).....	15
Figure 2-3: Surface roughness data.....	17
Figure 2-4: Fastener testing system	21
Figure 2-5: Torque tension test result	21
Figure 2-6: Corrosion cycle profile.....	25
Figure 2-8: Arrangement of DLJs.....	25
Figure 2-9: Hybrid bonded/bolted DLJs under tensile test.....	26
Figure 2-10: Corresponding FMs of bonded-only joints [8]	27
Figure 2-11: Corresponding FMs of bolted-only joints [8]	27
Figure 3-1: Representative trend of load/displacement (Bonded-only joints).....	30
Figure 3-2: Representative trend of load/displacement (Bolted-only joints).....	31
Figure 3-3: Representative trend of load/displacement (Hybrid bonded/bolted joints)	32
Figure 3-4 LTC evolution of Uralane bonded-only DLJs with corrosion cycle(s).....	35

Figure 3-5: Representative load/displacement curves (Uralane)	35
Figure 3-6: A schematic reorientation of the chemical process with chlorine [35]	37
Figure 3-7: LTC evolution of Epibond bonded-only DLJs with corrosion cycle(s).....	38
Figure 3-8: Representative load/displacement curves (Epibond)	39
Figure 3-9: LTC evolution of bolted-only DLJs with corrosion cycle(s)	40
Figure 3-10: Representative load/displacement curves (bolted-only)	40
Figure 3-11: LTC evolution of hybrid bonded/bolted DLJs with corrosion cycle(s)	42
Figure 3-12: Representative load/displacement curves (hybrid bolted/bonded)	43
Figure 3-13: LTC revolution of bonded-only DLJs (Uralane vs Epibond)	45
Figure 3-14: LTC revolution (Uralane vs Uralane hybrid bonding and bolting)	46
Figure 3-15: LTC revolution (Bolting-only vs Bonding-only)	48
Figure 3-16: LTC revolution of hybrid DLJs (Bonding vs Bolting)	48
Figure 3-17: Diameter evolution of uncorroded area	50
Figure A-1: FM of Uralane bonded-only DLJs - 0 cycle.....	60
Figure A-2: FM of Uralane bonded-only DLJs -1 cycle.....	60
Figure A-3: FM of Uralane bonded-only DLJs -7 cycles	61
Figure A-4: FM of Uralane bonded-only DLJs – 14 cycles	61

Figure A-5: FM of Uralane bonded-only DLJs - 22 cycles	61
Figure A-6: FM of Uralane bonded-only DLJs - 30 cycles	61
Figure A-7: FM of Epibond bonded-only DLJs - 0 cycle.....	62
Figure A-8: FM of Epibond bonded-only DLJs - 1 cycle.....	62
Figure A-9: FM of Epibond bonded-only DLJs - 7 cycles	62
Figure A-10: FM of Epibond bonded-only DLJs - 14 cycles	62
Figure A-11: FM of Epibond bonded-only DLJs - 22 cycles	63
Figure A-12: FM of Epibond bonded-only DLJs - 30 cycles	63
Figure A-13: FM of bolted-only DLJs - 0 cycle	64
Figure A-14: FM of bolted-only DLJs - 1 cycle	64
Figure A-15: FM of bolted-only DLJs - 7 cycles	64
Figure A-16: FM of bolted-only DLJs - 14 cycles	64
Figure A-17: FM of bolted-only DLJs - 22 cycles	65
Figure A-18: FM of bolted-only DLJs - 30 cycles	65
Figure A-19: FM of hybrid bonded/bolted DLJs - 0 cycle	66
Figure A-20: FM of hybrid bonded/bolted DLJs - 1 cycle	66
Figure A-21: FM of hybrid bonded/bolted DLJs - 7 cycles.....	66

Figure A-22: FM of hybrid bonded/bolted DLJs - 14 cycles.....	66
Figure A-23: FM of hybrid bonded/bolted DLJs - 22 cycles.....	67
Figure A-24: FM of hybrid bonded/bolted DLJs - 30 cycles.....	67
Figure A-25: Al surface degradation with corrosion cycle(s) (Adhesive bonding-only joints) ...	68
Figure A-26: Remaining adhesive on CFRTP surface (Adhesive bonding-only joints)	69
Figure A-27: Al surface degradation (Uralane vs Uralane hybrid bonding/bolting).....	70
Figure A-28: Remaining adhesive on CFRTP (Uralane vs Uralane hybrid bonding/bolting)	71
Figure A-29: Al surface degradation with corrosion cycle(s) (Bonding-only vs Bolting-only)...	72
Figure A-30 Overlap surface comparison.....	73
Figure A-31: Decreasing uncorroded area.....	74
Figure A-32: A scheme of bolted-only DLJs.....	75

List of Tables

Table 2-1: Mechanical properties of the Sereebo I series	13
Table 2-2: Chemical composition of AA 6060 according to EN573-3	13
Table 2-3: Mechanical properties of AA 6060	14
Table 2-4: DLJs manufactured for baseline and CCT	16
Table 2-5: Typical properties of Uralane 5774 A/C	18
Table 2-6: Typical cured properties of Uralane 5774 A/C	18
Table 2-7: Typical properties of Epibond 121 0 A/hardener 9861	19
Table 2-8: Typical cured properties of Epibond 1210 A/ hardener 9861	19
Table 2-9: Bolts, nuts and washers	20
Table 2-10: Desired preload.....	22
Table 3-1: Average Static LTC (kN) with corrosion cycles (s).....	33
Table 3-2: Diameter of uncorroded area, washer and stress circle	50

Nomenclature

CO₂ Carbon dioxide

g gram

Kg kilogram

Km kilometer

kN kilo newton

Nm Newton meter

Glossary

FRP	Fiber-reinforced plastic
DLJs	Double Lap Joints
SLJ	Single lap Joints
LTC	Load Transfer Capacity
CFRTP	Carbon Fiber Reinforced Thermoplastic Composite
ASTM	American Society for Testing Materials
CCT	Cyclic Corrosion Test

1 Introduction and Literature Survey

This section consists of two main parts: industrial background introduction and literature survey. Research objective and methodology are introduced at the end. .

1.1 Introduction

Nowadays car manufacturers are designing their car increasingly lighter to reduce both fuel consumption and environment pollution. In the European Union, the target of emission reduction for 2030 will be to cut at least 40% of greenhouse gas emissions from 1990 levels and to reach an improvement in energy efficiency of at least 32.5% [1]. Therefore, the automotive field is paying more and more attention on composite materials to meet the demands of weight reduction.

A composite is usually made of a high-performance fiber (carbon- or glass-based) in a matrix material (such as an epoxy polymer). When combined, matrix and reinforcement provide enhanced properties compared to the individual materials. Carbon fiber and glass fiber composite materials have been used for many years in the automotive industry to create components that have unique characteristics, such as being strong and light.

Carbon fiber and glass fiber composites have different mechanical properties [2]. Carbon-fiber composites weigh about one-fifth as much as steel but are as good or better in terms of stiffness and strength [3, 4, 5]. Glass fiber is made by melting glass and extruding it under high pressure, then combining the resulting strands of material with an epoxy resin to create what is known as a fiber-reinforced plastic (FRP). Compared to carbon fiber, glass fiber has lower stiffness and durability. However, fiberglass materials are used in a much broader range of applications, the result being that more fiberglass is manufactured, and prices are lower.

Coupling traditional materials such as steel or aluminum with composite materials has become more widespread since the cost of composites is still relatively high in 2019. Due to their high specific strength, carbon fiber composites and aluminum alloy are the most attractive combination in the automotive industry. Glass-based composites are also a valid choice because of their lower cost compared to carbon fiber composites.

However, composite materials carry some problems, one of which is the resistance to joining. Joining methods can be divided into three categories: mechanical joining, adhesive bonding, and welding. Mechanical joining and welding are the most applied methods in the present automotive industry. Unfortunately, these traditional joining approaches cannot translate directly to composite materials. The presence of a hole would cause stress concentration, and the lack of plasticity in composites limit stress redistribution. Another reason is that the additional weight of bolts and nuts would penalize the strength-to-weight advantage typical of lightweight materials. Therefore, the most promising technology for composites in the automotive sector is adhesive bonding [6]. In the 1960s, the aerospace industry started using adhesive bonding technology, showing that it was an optimal solution for the manufacture of lightweight, resistant structures [7]. Nowadays, adhesive bonding is increasingly applied in the automotive industry for composite materials assembly.

Another problem with composite materials is their susceptibility to corrosion. Composites themselves are considered to have good corrosion resistance. However, hybrid combination with metal alloys can present some challenges. The static and dynamic performance of composites-based Single Lap Joints (SLJ) is affected by temperature and humidity [8]. Salt corrosion is also a major cause of degradation of fiber composites [9]. Additionally, exposure to HCL and NaOH solutions can affect the mechanical behavior of composite joints [10]. Several papers in literature can be found about multi-material joining. The behavior and mechanical characteristics of hybrid composite-metal joints are assessed in the studies by Kabche et al., Lambiase, Fiorea, and Matsuzakia [11, 12, 13, 14].

Hence, what deserves to be further discussed is the stability and durability of hybrid composite/metal joints in real, critical environments, for their long-term application. The mechanical degradation of hybrid composite/metal joints can be caused by both the corrosion on the metal substrates and the degradation of composite substrates. Therefore, temperature, humidity or salt spray can have a huge influence on the strength of hybrid joints.

1.2 Literature Survey

A brief literature review is addressed, and the following points have been considered:

- Composite materials
- Adhesive bonding
- Double lap joints
- Cyclic corrosion test

1.2.1 Composite materials classification

A composite material can be classified by its matrix (e.g. Polymer Matrix, Ceramic Matrix and Metal Matrix) and by the nature of the reinforcement (e.g. Particle reinforced, Fiber reinforced, and Structural).

Polymer matrix composites can be divided into two different categories: thermoset, for structural application, and thermoplastic, for general use [15]. Ceramic Matrix and Metal Matrix are preferred in high wear and temperature applications [15].

Particle reinforced composite are the cheapest and most widely used. They are applied where high levels of wear resistance are required, such as in a road surface. However, they are inferior to average fiber reinforced composites in terms of strength and toughness.

Optimum strength and stiffness can be achieved by fiber reinforced materials. The reinforcement is classified as either continuous (long) or discontinuous (short) fibers. When the fibers are aligned,

they provide maximum strength, but only along the direction of alignment. This anisotropy can be solved by randomly aligning fibers in any direction [16, 17]. Components that require strength in one direction will use aligned fibers, while components that are loaded in more than one direction will use randomly oriented fibers. In continuous fiber composites, fibers have the same length of component itself, and they usually have a specific orientation. On the other hand, discontinuous fiber composites have shorter fibers and they are usually randomly oriented.

1.2.2 Adhesive bonding in the automotive field

Some composite materials are not compatible with traditional joining methods. Adhesive bonding is now sometimes utilized in place of bolting, riveting and welding. Moreover, traditional joining methods, compared with adhesive bonding often add extra weight to the structure.

Adhesive bonding is considered an optimal solution for manufacturing lightweight structures, it is increasingly prevalent in the automotive field, and it shows a number of advantages over traditional joining:

- Adhesive bonding produces continuous material joining instead of localized, discrete joining, which results in a more uniform stress distribution over a larger surface area and reduces stress concentration at the joints edges, thereby providing good fatigue resistance [18].
- Adhesive bonding can also improve joint stiffness because of the continuous bonded contact [19].
- Adhesive bonding has high energy absorption capacity, providing good noise and vibration damping [19].

- Adhesive joints can also prevent water ingress into the joint area, similarly to a seal.

However, there are many limitations that influence the performance of adhesive bonding:

- One of them is heat curing time, which makes adhesive bonding in assembly more complicated than traditional joining methods [20].
- Adhesive bonding may require surface preparation that includes surface cleaning and surface pre-treatment.
- Another notable limitation is the difficulties in extensive utilization of adhesive bonding in volume production.

1.2.3 Double lap joints

Mechanical tests have been carried out on Double Lap Joints (DLJs) in this research. Single Lap Joint (SLJ) and DLJs are the most studied joints in research and industrial environments. This is because they are easy to be assembled and their shear strength is an important parameter to evaluate the joints' mechanical properties. DLJs have 3 substrates: 2 outer and 1 inner substrates. The configuration of bonded-only DLJs is shown in Figure 1-1, as an example.

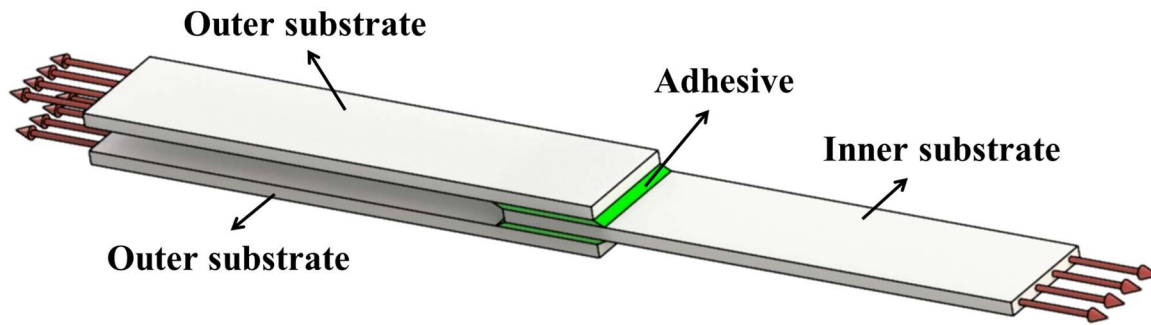


Figure 1-1: Double Lap Joints configuration (bonded-only)

1.2.4 Cyclic corrosion testing

Cyclic Corrosion Test (CCT) is a standardized method to assess the corrosion resistance of materials, such as metal/alloy or composites. Nowadays, CCT has been a common practice in the automotive industry since modern cars are required to work well in various challenging situations.

CCT can test how automotive materials react to critical environments, helping automotive designers to select materials according to their longevity and durability requirements [21]. A wide variety of automotive components need to be tested via CCT. Generally, any component that has contact with corrosive matter or is at risk of galvanic or crevice corrosion is required to be tested in a CCT chamber.

CCT usually gives better corrosion simulation than conventional steady salt fog spray. The relative corrosion rate and structure of CCT are more similar to what happens outdoors [22, 23]. . The actual working atmosphere usually includes both wet and dry conditions and the environment is

dynamically changing. During CCT, specimens are exposed to a series different environment conditions in a repeated cycle.

Multi-step cycles that may incorporate immersion, humidity and condensation, along with salt fog and dry off are typically used. Cycle's duration, temperature, and electrolytic composition can be unique to each specification. Therefore, CCT helps manufactures ensure that their products and materials can bear repeated environment wear and exposure.

There are various CCT standards adapted by different car manufacturers: GMW 14872 [24], GM 9540 P [25], SAE J2334 [26], Honda CCT, Toyota TSH 1555G, and so on.

1.2.5 Relevant research

A lot of relevant literature works can be found and most of them focus on the effect of critical environmental factors on the mechanical performance of traditional joints, with static and fatigue tests

Sakai et al. [8] evaluated the effect of environmental loading on static and dynamic behavior of single lap joints (SLJ). Lightweight multi-material substrates including glass fiber reinforced polymer (GFRP), steel (St), aluminum (Al) and magnesium (Mg) are used. The results show that cyclic heat at low relative humidity can increase the static load transfer capacity (LTC) of bonded samples while high humidity will reduce the LTC of bonded specimens by 70% and 25% respectively for steel-to-magnesium and composite-to-magnesium joints. As for the dynamic performance of SLJ, the fatigue life was reduced significantly after heat cycling at high relative humidity.

V. Fiorea et al. [9] reported the effect of salt fog spray on hybrid composite/metal rivet joints. Carbon fiber, epoxy composite, and E-glass fiber composite substrates were riveted to AA 6060, and were exposed to salt-fog environmental cycling. The mechanical performance of the hybrid joints decreased significantly with exposure time and glass fiber/epoxy composite joints showed a more sensitive degradation compared to carbon fiber composite joints.

Hao et al. [27] assessed the fatigue degradation of hybrid composite/metal joints after salt spray cycling. The mechanical performance of carbon fiber reinforced plastics (CFRP) /Al electromagnetically riveted lap joints after exposure in neutral salt spray environment at various ageing time intervals was studied. The shear strength and fatigue life of CFRP/Al electromagnetically riveted lap joints decreased gradually with exposure time. 70.3 % of the baseline maximum load and 35.1 % of the original fatigue life were retained after 7 weeks of salt fog exposure.

Chen et al. [28] examined the static and fatigue strength of bolted composite-based joints under hydrothermal cycles. In their study, the room humidity history is the key for bolt relaxation. Each humid day resulted in a peak in the relaxation curve. The sensitivity of bolt clamp torque to moisture contamination is also analyzed.

Aylor et al. [29] reported the effect of seawater on graphite/epoxy composites electrically coupled to metal. The corrosion behavior between graphite/epoxy composite and metals – steel and Nickel Aluminum Bronze alloy (NAB) – was evaluated after 180 days seawater exposure. Galvanic couple and laboratory electrochemical tests followed. The results indicated that corrosion of steel and NAB would occur when these metals were coupled to Gr/epoxy composites individually and galvanic current could be detected. Corrosion reduction rate and galvanic current rate were measured for different composite/metal couples. With the increasing exposed area of graphite to

the seawater, the corrosion would gradually progress. Corrosion could also happen between Gr/epoxy composite to metal in seawater environment without any graphite fibers being initially exposed, which is believed to be caused by the adsorption of moisture.

Nguyen et al. [30] discusses the mechanical performance of steel/CFRP double strap joints exposed to harsh environments. One-year sea water exposure at 20°C and 50°C, 1000 hours' cyclic temperature and humidity were simulated in a laboratory environment. Buck adhesive and carbon fiber reinforced epoxy coupons were also manufactured and exposed to the same conditions for comparison purposes. The strength and stiffness of Steel/CFRP joints degraded rapidly in the first 2–4 months of exposure, for both 20°C and 50 °C groups. After 4 months, there was little change in strength. The strength reduction rate at 50 °C was higher than at 20°C. With cyclic heat and humidity, the steel/CFRP joints suffered a more severe degradation than the buck adhesive by itself, and more degradation was found with steel/CFRP joints at early exposure stage.

On the one hand, traditional salt spray tests cannot simulate realistic environmental conditions precisely and cyclic corrosion tests are common in the automotive field. On the other hand, few studies are found that investigate how corrosion affects different joining methods. Adhesive bonding is optimal for dissimilar materials, and hybrid bolting-bonding can combine the strengths of bolted connections to the advantages of bonding.

It is of interest to combine these two topics together and to investigate the behavior of composite/metal joints, with different joining methods, and in critical environment conditions. Few works are present in the literature about this important issue, especially in the automotive field, leading to a lack of data for automotive designers.

1.2.6 Research objective and methodology

This research aims evaluating the effect of cyclic corrosion on static strength of composite/aluminum DLJs. DLJs are placed in a cyclic corrosion (salt-fog) test chamber following standard GMW 14872. Joints are pulled out for tensile-shear testing and failure mode inspection during corrosion test.

Total 72 DLJs are made of carbon fiber reinforced thermoplastic composite (CFRTP) material and aluminum alloy AA 6060 with 4 joining methods:

- Bonded-only with polyurethane-based adhesive
- Bonded-only with epoxy adhesive
- Bolted-only with M4-0.7 bolts
- Hybrid bonded/bolted (polyurethane-based adhesive/M4-0.7)

60 DLJs are placed in corrosion chamber, with 12 (3 per joining method) as baseline. 12 joints (3 per joining method) are pulled out each time after 1, 7, 14, 22, and 30 days of exposure to cyclic corrosion. Then static tensile test and failure mode inspection are carried out to evaluate effect of cyclic corrosion on joints' static strength.

2 Experimental Setup and Test Procedure

In this section, materials, geometry and joining methods of DLJs are introduced firstly. Then assembled DLJs are prepared for cyclic corrosion test following standard GMW 14872. Joints are pulled out for static tensile test and failure mode inspection after certain cycles.

2.1 Joint design and manufacturing

The materials and geometry of DLJs need to be defined firstly. Once they are known, substrates are assembled with different joining methods.

2.1.1 Substrate materials

The outer substrates are made of carbon fiber reinforced thermoplastic composite material (CFRTP), and the inner substrate is made of Aluminum Alloy AA 6060.

Carbon fiber reinforced thermoplastic composite (CFRTP)

Carbon-Fiber-Reinforced thermoplastic composite material (CFRTP) is the Serebo I-series from Continental Structural Plastics (Auburn Hills, US). The Serebo series is the world's first technology for high-volume production of CFRTP composite materials with a Takt time less than 1 minute [31]. Its characteristic is shown in Table 2-1:

Table 2-1: Mechanical properties of the Sereebo I series

Materials	Carbon fiber composite
Thickness [mm]	3
Matrix	Thermoplastic resin
Fiber Vol [%]	35
Density [kg/m^3]	1380
Tensile modulus [GPa]	26
Tensile strength [MPa]	350
Yield strength [MPa]	350
Bending modulus [GPa]	26
Bending strength [MPa]	480

Aluminum alloy 6060

Aluminum alloy AA 6060 is an alloy in the wrought aluminum-magnesium-silicon family (6xxx series). Its chemical composition and mechanical properties are shown in Table 2-2 and Table 2-3 respectively.

Table 2-2: Chemical composition of AA 6060 according to EN573-3

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
6060	0.30-0.60	0.10-0.30	0.1	0.1	0.35-0.60	0.05	0.15	0.1	rest

Table 2-3: Mechanical properties of AA 6060

Alloys EN-AW	6060
Thickness [mm]	3
Density [g/m^3]	2.7
Temper	T4 (solution heat treated & natural aged)
Metallic range [$^{\circ}\text{C}$]	585-650
Electrical conductivity [MS/m]	34-38
Thermal conductivity [W/(m K)]	200-220
Specific Heat [J/(K g K)]	898
Young modulus [Mpa]	69500
Shear Modulus [Mpa]	26100

2.1.2 Double lap joints geometry

The geometry of DLJs is shown in Figure 2-1. The thickness of each substrate is 3 mm and the width of the substrate is 25.4 mm (1 inch). The length of each substrate is 127 mm (5 inches), and the bond line is 25.4 mm (1 inch) long. Because M4 bolts are used for bolted-only and hybrid bonded/bolted joints, a 4.5 mm diameter hole is positioned in the center of the overlap area for every DLJs.

A spacer is used to compensate for the inner substrate thickness and has no effect on the strength of DLJs. It is made of the same material as the outer substrate, Aluminum Alloy AA 6060. The spacer is about 20 mm long, and its width is the same as the DLJs (Figure 2-2).

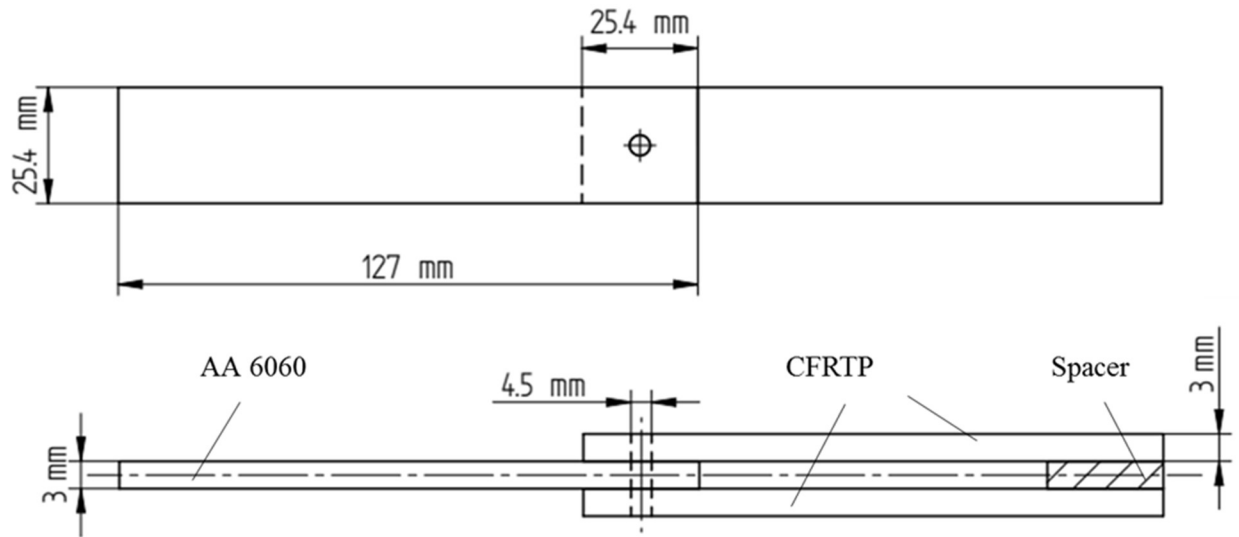


Figure 2-1: Double Lap Joints geometry

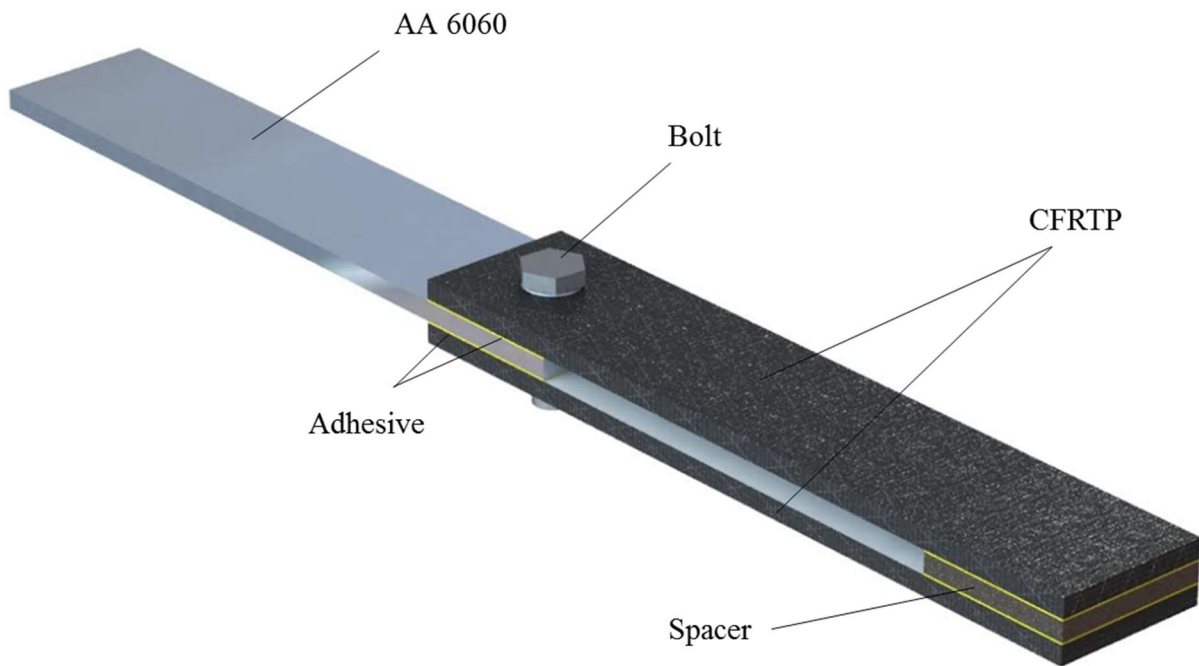


Figure 2-2: Model of DLJs (Hybrid bonded/bolted)

2.1.3 Joining methods

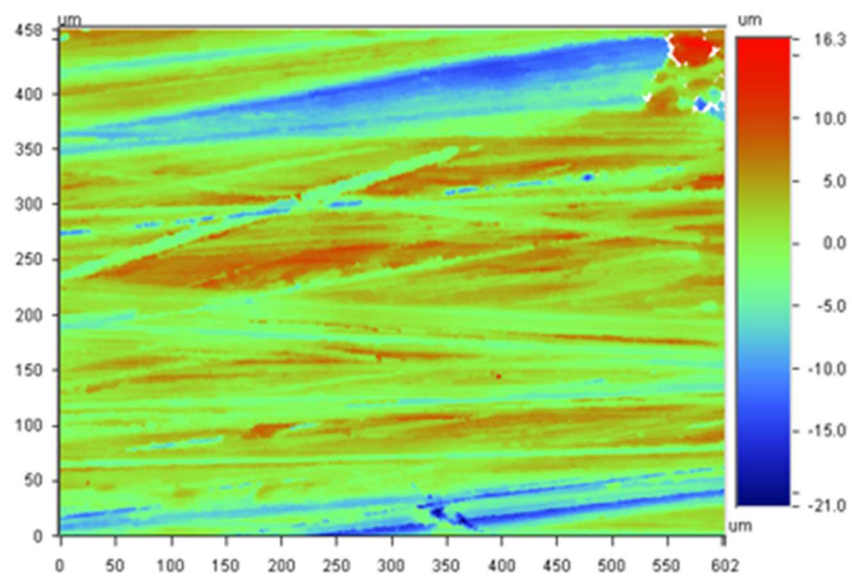
Total 72 DLJs are manufactured with 4 different joining methods, as shown in Table 2-4:

- bonded with polyurethane-based adhesive
- bonded with epoxy adhesive
- bolted with M4-0.7 bolts
- hybrid bonded/bolted (polyurethane-based adhesive/M4-0.7)

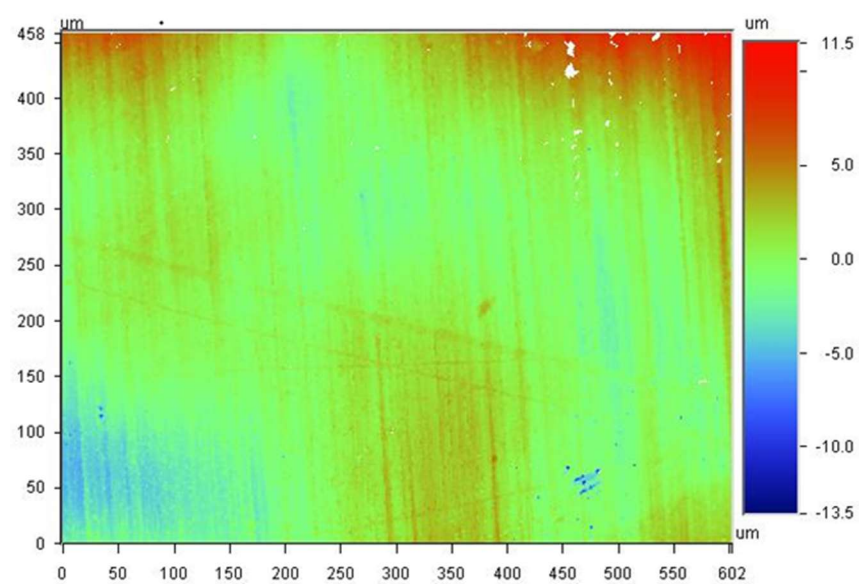
Table 2-4: DLJs manufactured for baseline and CCT

Joining methods	Bonded (Polyurethane)	Bonded (Epoxy)	Bolted (M4)	Hybrid (Polyurethane + Bolt)
Materials	Carbon-Fiber-Reinforced Thermoplastic Composite material & AA6060			
# of Samples	18	18	18	18

Before assembling DLJs, the surfaces of CFRTP and AA 6060 need to be cleaned. The way to clean CFRTP sheets is via ultrasonic cleaning with acetone and aluminum surface is sanded before ultrasonic cleaning because of oxide film. The surface roughness R_a of AA 6060 is $3.3\ \mu\text{m}$ and the surface roughness for CFRTP is $1.5\ \mu\text{m}$ and it is measured with a Wyko optical profilometer. The AA 6060 coupons that do not fall in the pre-defined target roughness range are re-sanded and cleaned. Roughness data is shown in Figure 2-3.



AA 6060 T4
$Ra=3.3\pm0.2\ \mu\text{m}$



CFRTP composite
$Ra=1.5\pm0.2\ \mu\text{m}$

Figure 2-3: Surface roughness data

Bonded-only joints with polyurethane-based adhesive (Uralane 5774 A/C)

The polyurethane-based adhesive is Huntsman Uralane 5774 A/C, which has two component and can be self-extinguishing. Normal and cured properties are shown in Table 2-5 and Table 2-6 respectively.

The ratio between 5774-A resin and 5774-C hardener is controlled by the dispensing gun and is 2:1 part by volume. The joints are cured in the oven for 2 hours at 95°C, 30 minutes after the assembly.

Table 2-5: Typical properties of Uralane 5774 A/C

Property	5774-A	5774-C	5774 A/C	Test Methods
Color	Off-white	Beige	Beige	Visual
Specific Gravity	1.1±5	1.2±05	1.15	ASTM-D-792
Viscosity cp at 25 °C	20-35,000	Semi-paste	Semi-paste	ASTM-D-2196
Gel time. 100 gms, at 25 °C	-	-	12-25 min	ASTM-D-2471
Shelf time, at 25 °C, unopened, months	6	6	-	-

Table 2-6: Typical cured properties of Uralane 5774 A/C

Test	Results		
Tensile lap Shear, ASTM D1002, Psi (MPa)	(-55°C)	(25°C)	(82°C)
AL/AL	4500	2200	1300
Polycarbonate/Polycarbonate	1500	1000	900
Declar T	1550	1350	600
Kydex 6565	1500	1200	450
T-peel, ASTM D-1876 Psi (N/mm)			
AL/AL	50	35	-
Polycarbonate/Polycarbonate	-	35	-
Hardness, ASTM D-2240, Shore D	60	50	60

Bonded-only joints with epoxy adhesive (Epibond 1210 A/hardener 9861)

Epoxy adhesive is Huntsman Epibond 1210 A/hardener 9861. Normal and cured properties are shown in Table 2-7 and Table 2-8 respectively.

The ratio of Epibond 1210 A-resin/ hardener 9861 is 5:1 by weight. Because of the very high viscosity of the two components, manual mixing is required. Once the adhesive is applied, the joint is cured for 2 hours at 65°C in the oven.

Table 2-7: Typical properties of Epibond 1210 A/hardener 9861

Property	1210-A Resin	1210-B Hardener	1210-A/B System	Test Method
Color	Tan	Amber	Tan	Visual
Specific gravity	1.5	1	1.3	ASTM-D-792
Viscosity, Cp @ 25 °C	250,000	30,000	Semi-paste	ASTM-D-2393
Flash Point, COC, °C	254	135	--	ASTM-D-92
Work life, 100 gms at 25 °C, minutes	--	--	50-75	ASTM-D-1338
Shelf life at 25 °C, unopened, months	12	12	--	FTM-204
Solids, %	--	--	100	ASTM-D-1644

Table 2-8: Typical cured properties of Epibond 1210 A/ hardener 9861

Tests	Results	Standard
Maximum service temperature, °C	93	ASTM-D-648
Tensile Shear test, MPa	-	
AL/AL, 25 °C	17.2	ASTM-D-1002
Steel/Steel, , 25 °C	17.2	-
Steel/Steel, 66 °C	3.4	
Polyester/polyester 25 °C	12.4	-
Hardness Shore D	80	ASTM-D-2204

Bolted-only joints

Hex bolts, nuts and washers are from Fastenal. Their mechanical properties are shown in Table 2-9.

Optimal preload is required for bolting assembly. Hence, torque tension test is carried out via fastener testing machine in Figure 2-4. Four bolts and nuts are tested and the result is shown in Figure 2-5. It shows that clamping force has a linear relationship with input torque at the beginning. After clamping force reaches the maximum, it doesn't increase with input torque any more. The maximum clamp force is achieved with input torque between 4 Nm and 5 Nm. Therefore, 4.5 kN is chosen as preload and the corresponding torque is 4 Nm, as shown in Table 2-10. The target preload is achieved with the corresponding tightening torque, which is obtained with a digital torque wrench.

Table 2-9: Bolts, nuts and washers

Property	Bolts	Nuts	Washers
Thread size	M4-0.7	M4-0.7	M4
Finish	Zinc	Plain	Plain
Grade	Class 8.8	A2	A2
Length	20 mm	-	-
Materials	Steel	Stainless steel	Stainless steel
Thickness	-	3.2 mm	0.8 mm
Inner diameter	-	-	4.3 mm
Outer diameter	-	-	9 mm
Thread	Coarse	Coarse	-
Type	Hex Cap Screw	Hex Nut	General Purpose Flat Washer
Wrench size	7 mm	7 mm	-

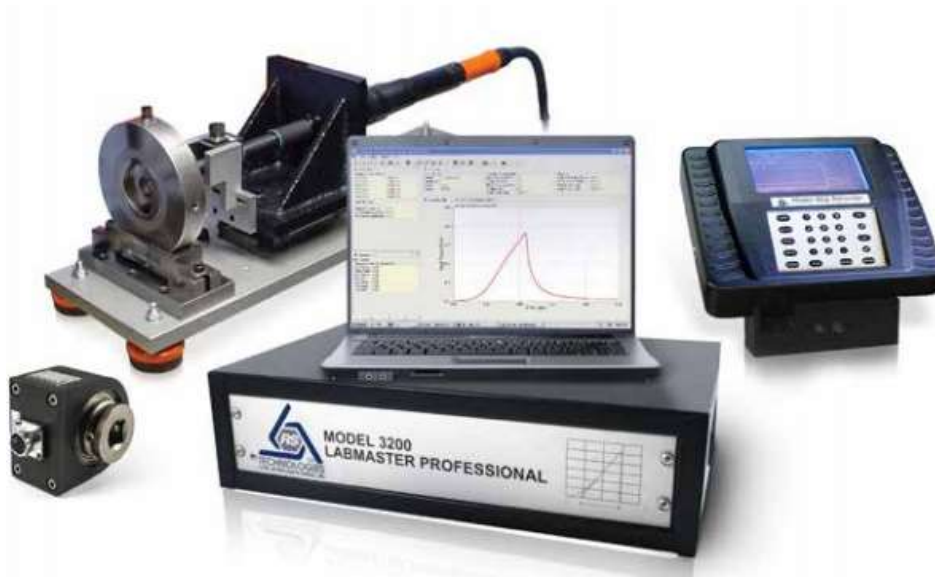


Figure 2-4: Fastener testing system

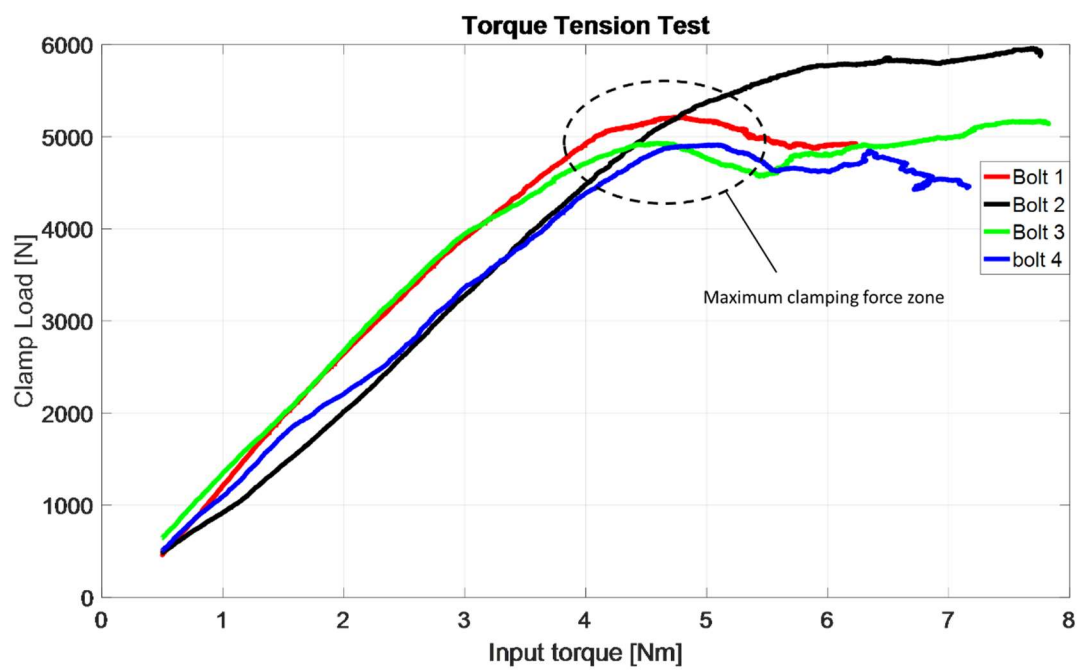


Figure 2-5: Torque tension test result

Table 2-10: Desired preload

Preload	4.5 kN
Corresponding torque	4 Nm

Hybrid Bonded/bolted joints

Hybrid bonded/bolted DLJs are using Uralane 5774 A/C adhesive and M4-0.7 bolts. They are bonded first and then bolted with same preload.

2.2 Cyclic corrosion test

GMW 14872 (2010) [24] is picked as cyclic corrosion standard in this research. This standard offers a combination of cyclic condition including 1% (approximate) salt spray, and three different stages [24]. Corrosion cycle profile is shown in Figure 2-6. 3 joints for each configuration are removed periodically after 1, 7, 14, 22, 30 cycles for mechanical test. The layout of joints inside the chamber is shown in Figure 2-7.

Ambient stage

The apparatus must be able to maintain the following environmental condition:

- Temperature: $25 \pm 2^{\circ}\text{C}$.
- Humidity: $45 \pm 10\%$ Relative Humidity (RH)
- Period: 8 h per cycle.

Humidity stage

The apparatus must be able to achieve the following environmental condition in 1 hour and maintain it for next 7 hours.

- Temperature: $49 \pm 2^{\circ}\text{C}$.
- Humidity: 100% RH.
- Period: 8h per cycle.

Dry off stage

The apparatus must be able to achieve the following environmental condition in 3 hours and maintain it for 5 hours.

- Temperature: 60 ± 2 °C.
- Humidity: $\leq 30\%$
- Period: 8 h per cycle.

Salt spray application

1% salt spray application is required to mist the samples until all the areas are totally wet/dripping. The amount of salt spray has to be enough to remove all the previous sprays. The first salt mist starts at the beginning of ambient stage and each following application should occur approximately 1.5 h (at least 1 h) after the previous one. The salt solution should be defined in percentage by mass as blow:

- Solution Chloride ($NaCl$): 0.9%
- Calcium Chloride ($CaCl_2$): 0.1%
- Sodium Bicarbonate ($NaHCO_2$): 0.075%

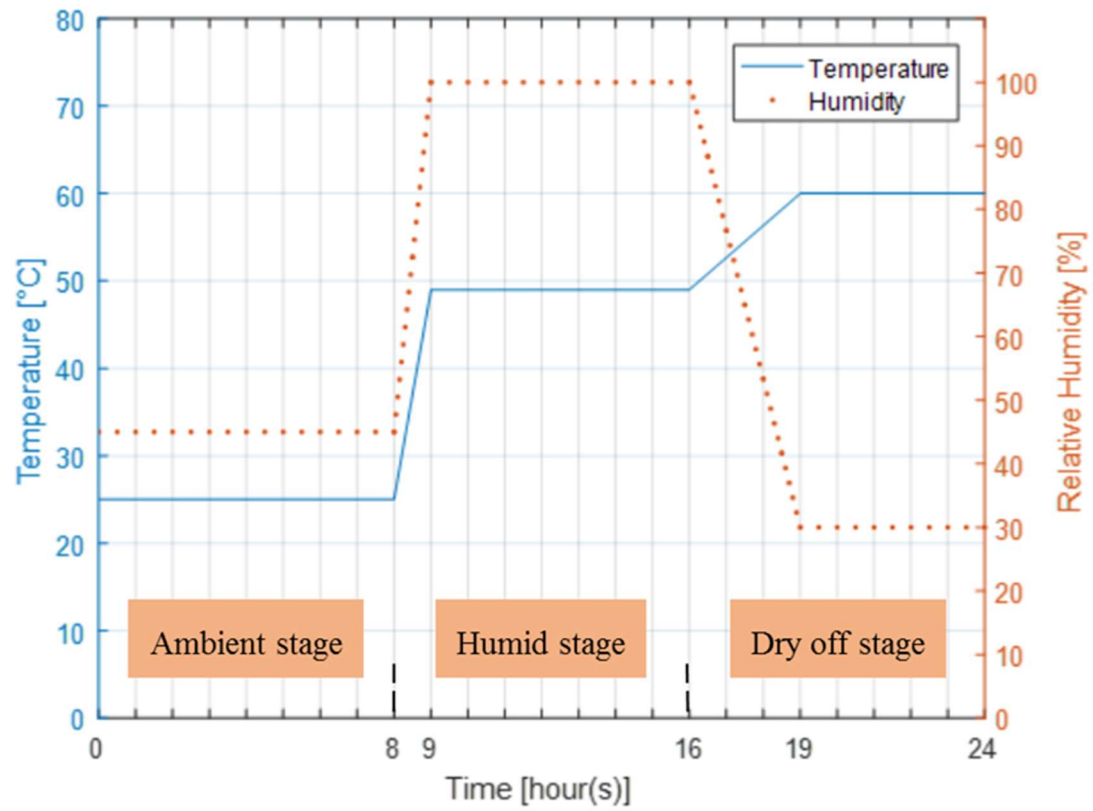


Figure 2-6: Corrosion cycle profile



Figure 2-7: Arrangement of DLJs

2.2 Static tensile test

Static tensile test is carried out at room temperature with a hydraulic tensile test machine produced by MTS, shown in Figure 2-8. In particular, 3 test repetitions per corrosion condition are carried out.

An axial load is applied at a constant speed of 1.27 mm/min (0.5 in/min). Two rectangular spacers with the same thick thickness of CFRTP substrate are placed between the end of Al substrate and grippers. The bending effect during test can be reduced in this way. Data is recorded automatically, and load vs displacement curve can be generated to obtain the information of load transfer capacity (LTC) of each DLJs, which refers to the maximum force that each DLJs can sustain.



Figure 2-8: Hybrid bonded/bolted DLJs under tensile test

2.3 Failure mode inspection

Corresponding failure modes are discussed after static tensile test. Bonded-only joints mainly have 6 different kinds of failure modes (Figure 2-9) and bolted-only joints mainly have 4 kinds of failure modes (Figure 2-10)

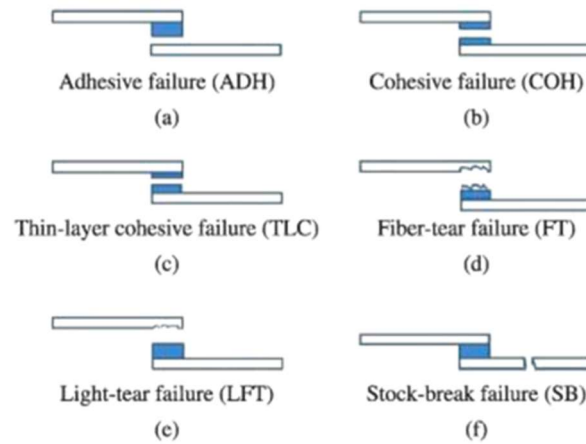


Figure 2-9: Corresponding FMs of bonded-only joints [8]

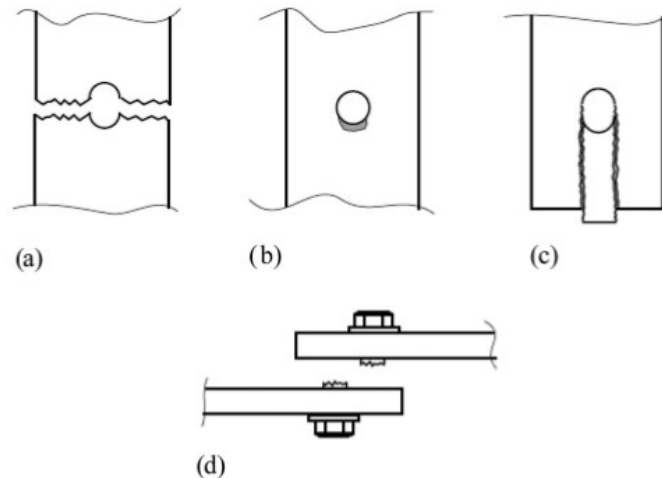


Figure 2-10: Corresponding FMs of bolted-only joints [8]

(a): net-section failure; (b): bearing failure; (c): TO failure; (d): bolt shear failure

3 Results and Discussion

Test data are presented and discussed in this section. Mechanical behavior of baseline DLJs is discussed at first. Then average LTC data with different corrosion cycles is shown to evaluate the effect cyclic corrosion on DLJs' static strength. Performance of different joining methods under corrosion are compared to compare each joining method. Failure mode inspection is reported as well with statistical results.

3.1 Mechanical behavior of baseline joints

Mechanical behavior of baseline DLJs with various joining methods are presented, in order to study the joints' performance. Bonded-only DLJs, bolted-only DLJs and hybrid bonded/bolted joints are discussed respectively.

3.1.1 Mechanical behavior of baseline bonded-only joints

Bonded-only DLJs with 2 different adhesives are discussed together since they have similar mechanical behavior under static tensile test. Uralane 5774 A/C is a ductile adhesive while Epibond 1210 A/hardener 9861 is a brittle adhesive. 3 significant regions for Uralane bonded-only joints and 2 regions for Epibond bonded-only joints can be identified in Figure 3-1:

Uralane (Ductile) bonded-only joints

The first zone is linear stage. Force is proportional to displacement. In this region, adhesive has an elastic deformation and Hooke's law is applicable.

The second zone is the strain hardening stage, starting at the initiation point of plastic deformation. When force is beyond the yield point, adhesive starts to elongate until force reaches the ultimate point. Hence, force is not linear with displacement.

The third zone is necking region. After force reaches the ultimate point, adhesive bonding fails and DLJs are separated.

Epibond (Brittle) bonded-only joints

The first zone is linear stage. Force increases linearly with displacement. Hooke's law can be applied since materials are undergoing an elastic deformation.

The second zone is fracture stage. For brittle materials, yield limit is the same with ultimate limit. When force reaches the ultimate limit, adhesive bonding fails and DLJs are separated.

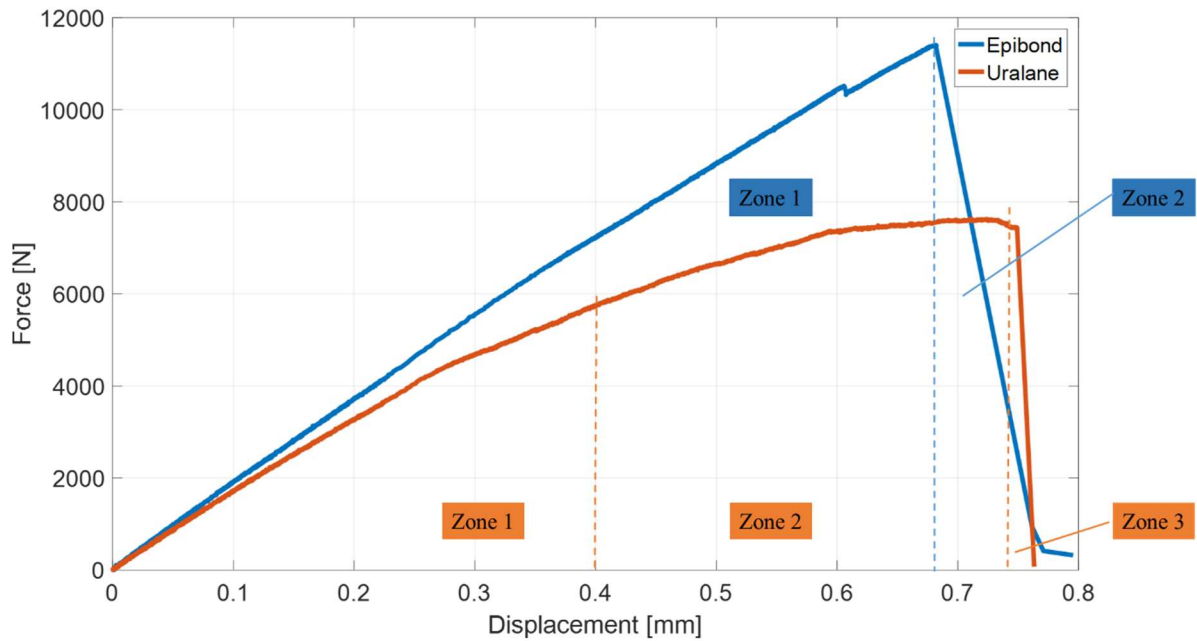


Figure 3-1: Representative trend of load/displacement (Bonded-only joints)

3.1.2 Mechanical behavior of baseline bolted-only joints

4 regions can be defined in Figure 3-2 for bolted-only joints. The first zone is the maximum static friction zone caused by preload clamping force. There is no relative sliding between CFRTP substrate and AA 6060 substrate.

The second zone is dynamic friction stage. It starts from the initial of relative sliding until substrates touches bolts, since there is a 0.5 mm clearance between hole and bolt,

The third zone is after bolts touches substrates. The shear force increases with displacement linearly until materials yield limit.

The fourth zone is bolts start eating materials. The shear force increases with displacement until bolts' ultimate limit. Bolts shear off after ultimate limit and bolted DLJs fail.

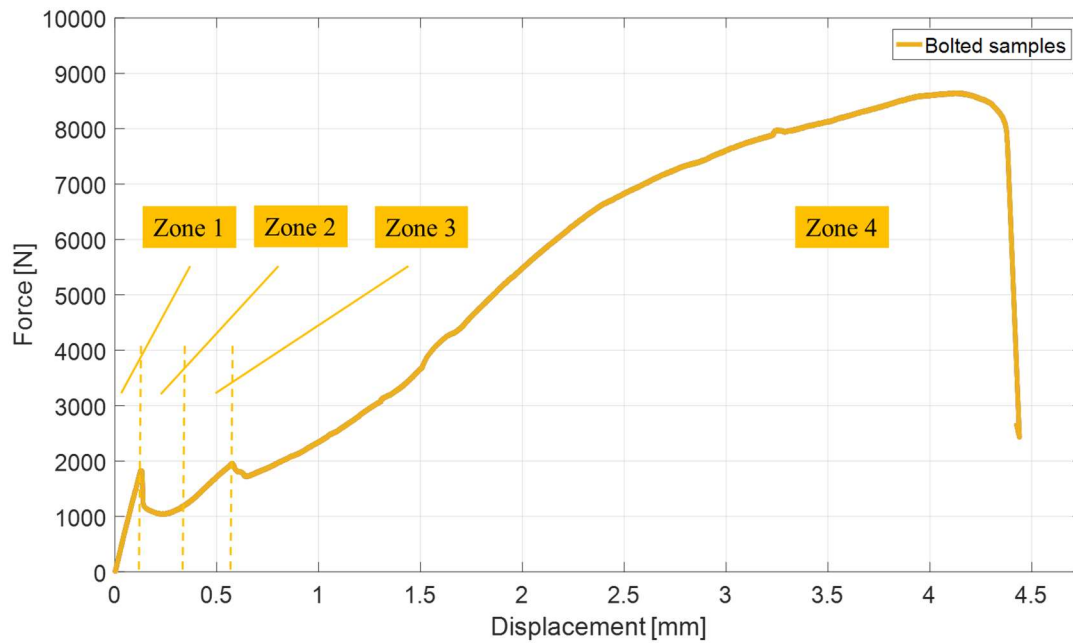


Figure 3-2: Representative trend of load/displacement (Bolted-only joints)

3.1.3 Mechanical behavior of baseline hybrid bonded/bolted joints

2 significant regions can be found in Figure 3-3 for hybrid bonded/bolted joints.

The first zone is bonding region. At the beginning, adhesive bonding works against the pull force and force increases linearly with displacement. After ultimate limit of adhesive, force decreases suddenly and adhesive bonding fails.

The second region is bolting region. Substrates start move relatively and clearance reduces. Then bolts start eating the substrates and force increases until bolts shear off.

2 force peaks can be obtained in load/displacement curve for hybrid bonded/bolted joints: bonding peak and bolting peak. The performance of these 2 peaks under corrosion test are discussed later.

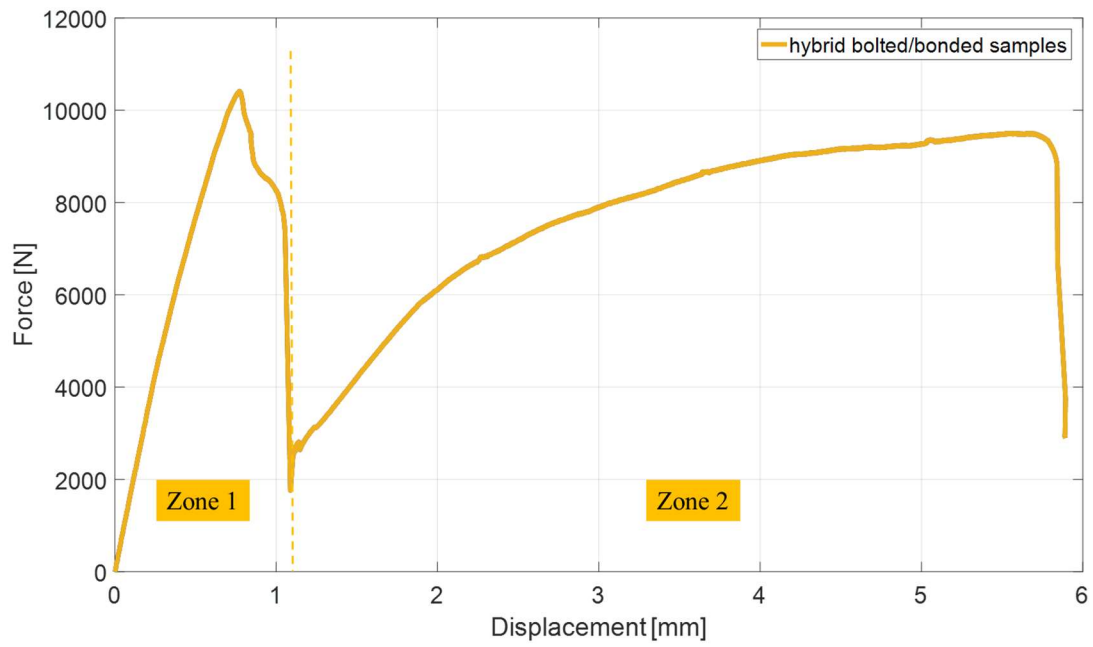


Figure 3-3: Representative trend of load/displacement (Hybrid bonded/bolted joints)

3.2 Effect of corrosion cycles on Joints Static LTC

The average LTC data is shown in Table 3-1. For hybrid bonded/bolted joints, bonding LTC and bolting LTC are discussed separately.

Table 3-1: Average Static LTC (kN) with corrosion cycles (s)

Joining methods	Bonding (Uralane)	Bonding (Epibond)	Bolting-only	Hybrid bonding(Uralane)	Hybrid bolting
Corrosion cycle (s)	LTC [kN]				
Baseline	8.77	10.48	9.23	10.03	9.61
1 cycle	8.81	9.68	10.88	10.88	9.43
7 cycles	8.06	9.57	8.61	6.35	9.22
14 cycles	7.37	8.85	8.55	4.98	9.44
22 cycles	3.84	5.46	8.84	2.41	9.49
30 cycles	1.37	6.11	8.96	2.44	9.35

From Table 3-1, it shows that cyclic corrosion has a huge effect on static strength of Uralane bonded-only DLJs and bonding peak of hybrid bonded/bolted DLJs. the strength of Epibond bonded-only DLJs don't show a significant reduce at the first two weeks and have about 60% left at the end. Bolted-only DLJs and bolting peak of hybrid bonded/bolted joints are not affected significantly by cyclic corrosion test. Their LTC remains the same level after corrosion test.

3.2.1 Effect of cyclic corrosion on Uralane bonded-only DLJs

Uralane bonded-only DLJs are significantly affected by corrosion. Their LTC decreases from 8.77 kN at baseline to 1.37 kN after 30 cycles, with only 16% left at the end (Table 3-1). The strength decreases slowly in the first 2 weeks with a 16% drop, as shown in Figure 3-4. After 2 weeks, the LTC reduce comes to be noticeable. Between 14 cycles and 22 cycles, there is a 48% reduce and 64% of LTC reduces from 22 cycles to 30 cycles. Figure 3-5 shows representative load-

displacement curves of Uralane bonded-only DLJs with increasing corrosion cycles. It is clear that the maximum force decreases with corrosion cycles.

Figure A-1 to Figure A-6 are the FMs of Uralane bonded only DLJs with different corrosion cycles. They are always interfacial adhesive failure (ADH). However, the failure position changes from CFRTP surface to Al surface with corrosion cycles. The remaining adhesive moves from Al surface to CFRTP surface.

There is obvious increasing corrosion on Al substrate surface. Figure A-3 shows that, a few corrosions occurs on Al surface after 1 week and remaining adhesive after tensile test starts showing on CFRTP surface. It has the same trend with their LTC that starts to decrease after 1 week in Figure 3-4. After 22 cycle and 30 cycles, there is much more corrosion on AL surface and almost all the adhesive remains on CFRTP surface after tensile test. LTC after 22 and 30 cycles, as mentioned above, are suffering big drops, which is also the same with corrosion trend.

It is clear that the bond between adhesive and Al surface is stronger than that between adhesive and CFRTP surface at the beginning. However, corrosion makes AL surface worse, which weakens bond between adhesive and Al surface. Therefore, failure happens on Al surface after certain cycles. Using the profilometer, the surface is scanned for the evidence of advanced stage pitting corrosion, the measurement shows no pits or superficial cavities In conclusion, there is increasing corrosion on Al surface with corrosion cycles and as a result, strength of Uralane bonded-only DLJs reduces gradually.

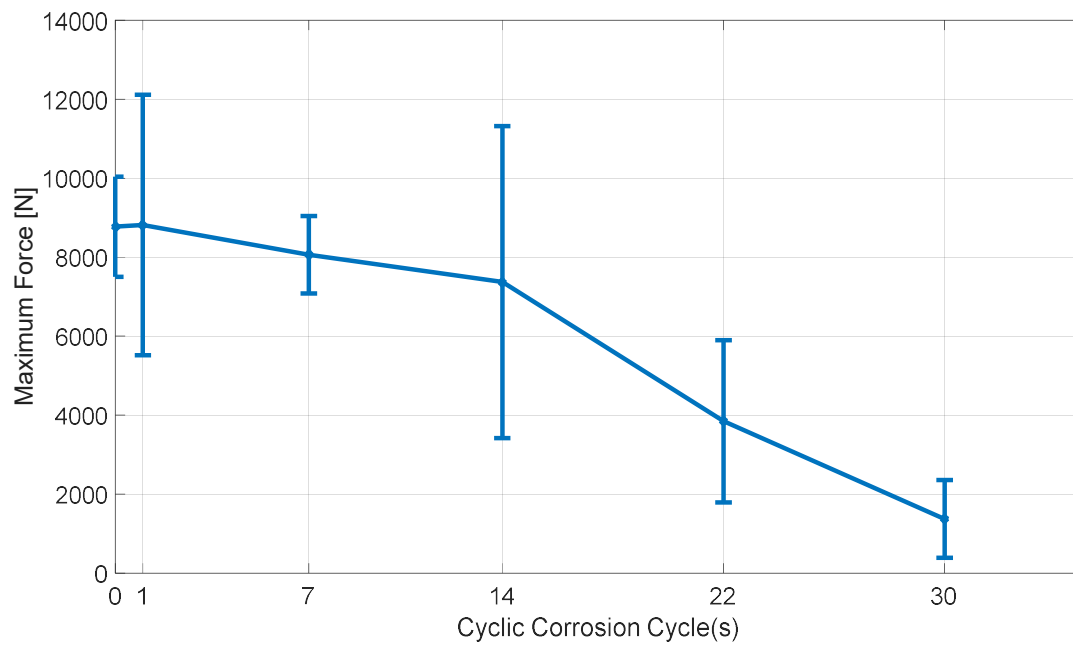


Figure 3-4 LTC evolution of Uralane bonded-only DLJs with corrosion cycle(s)

Error bar = $\pm 1 \sigma$ and the below

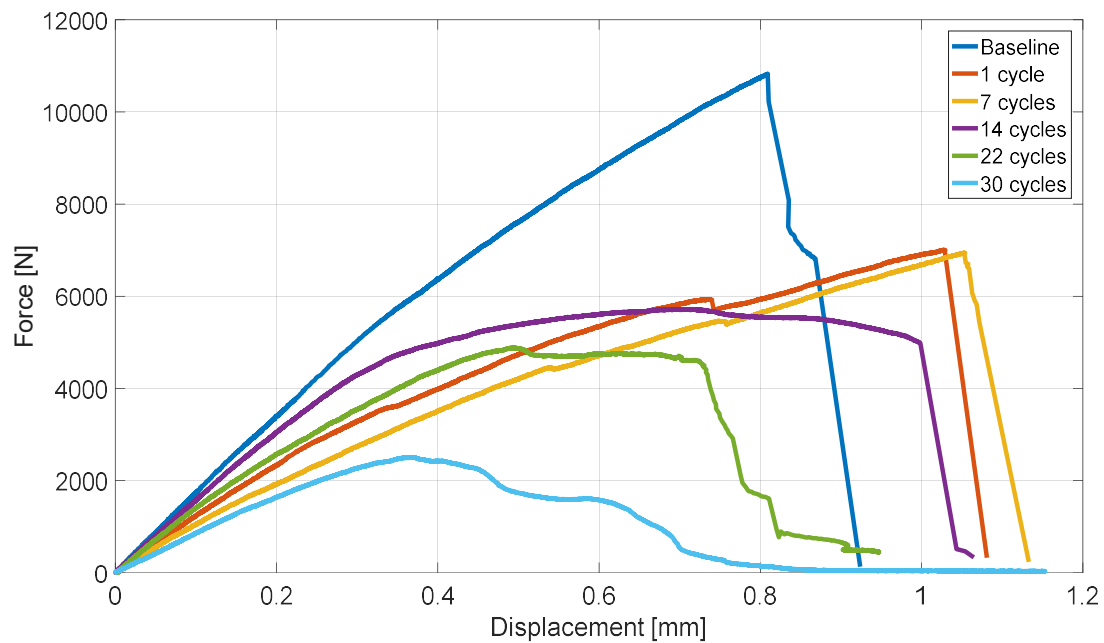


Figure 3-5: Representative load/displacement curves (Uralane)

Adhesive bonding, compared with mechanical riveting or bolting, is able to reduce crevice corrosion since adhesive can prevent salt spray solution into joints gap. Uralane 5774 A/C is polyurethane-based, and it can absorb moisture from environment. Therefore, crevice corrosion can still happen.

W. A. Landford et al [32] investigated the conversion of oxide film for aluminum in presence of water or water vapor. Oxide film can slow down corrosion rate of aluminum in normal environment and it would be converted into a hydrous form in the presence of water or water vapor. As a result, the film would be highly porous, which helps water penetration on Al surface. Oxide film is imperfect and susceptible to the complex chemistry of aqueous surface layer and anions in this aqueous solution are rapidly incorporated by aluminum [33]. Therefore, corrosion of aluminum appears. Pitting corrosion is considered as particular characteristics of chloride ion attack and carboxylic acid corrosion [34]. Because of salt spray application ($NaCl$, $CaCl_2$ and $NaHCO_2$), it is clear that it should be pitting corrosion that happens on Al substrates. .

T.E. Graedel [35] discusses the corrosion mechanisms for aluminum when it is exposed to chlorides. Figure 3-6 shows the mechanical progress of corrosion in the presence of water. Oxide film is dissolved, and chlorides would attack Al as deep pits occurring principally at the grain boundaries.

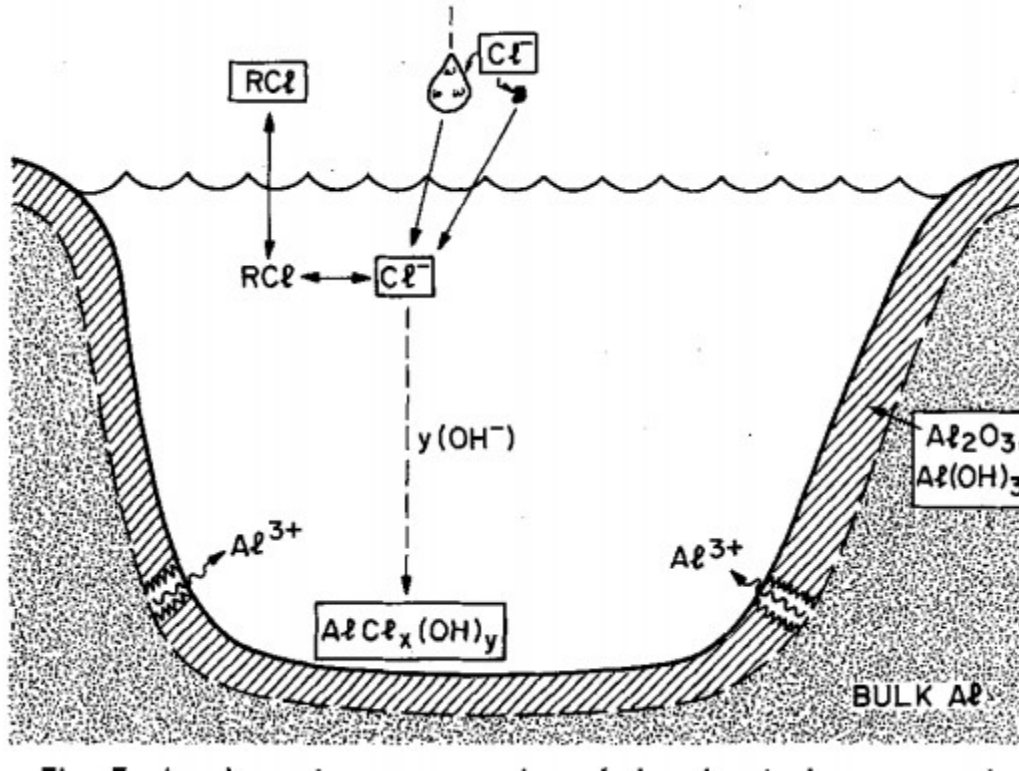


Figure 3-6: A schematic reorientation of the chemical process with chlorine [35]

3.2.2 Effect of cyclic corrosion on Epibond bonded-only DLJs

Table 3-1 shows that Epibond bonded-only DLJs have better corrosion resistance and almost 60% strength is left after 30 cycles. Figure 3-7 is the LTC revolution with corrosion cycles. There is no significant change after 2 weeks, considering the deviation. Figure 3-8 also supports this find. 6 curves in Figure 3-8 can be divided into 2 groups: upper 4 curves and lower 2 curves. There is no significant difference between load/displacement curves of DLJs with 1, 7, 14 cycles compared to baseline curve. After 22 cycles, it is clear that LTC of Epibond bonded-only DLJs reduces faster than before, from 8.85kN to 5.46kN. LTC is almost the same from 22 cycles to 30 cycles and curves are also similar. In short, corrosion doesn't significantly influence Epibond bonded-only DLJs in first 2 weeks and salt spray requires more time to diffuse to Al surface.

Corresponding failure modes also support this find. Failure modes are always interfacial adhesive failure and most adhesive remains on Al surface all the time. From Figure A-7 to Figure A-12, it can be noticed that there is no visible corrosion on Al surface after 14 cycles and almost all adhesive remains on Al surface. After 22 cycles, remaining adhesive starts moving to CF RTP surface and visible degradation shows up. However, these is much less corrosion comparing to Uralane bonded-only DLJs. In conclusion, DLJs bonded with Epibond adhesive have better corrosion resistance. Surface measurement shows that no visible pit is on Al surface.

Epibond adhesive is epoxy adhesive, which is considered with good water resistance. Since it is hard for salt spray to diffuse into gap between substrates, crevice corrosion needs more time. Pitting corrosion on Al surface has the same problem. Without influence of corrosion, DLJs bonded with Epibond can show a better performance. However, Epibond adhesive is not 100% water resistant. When there is enough solution in gap, corrosion still occurs.

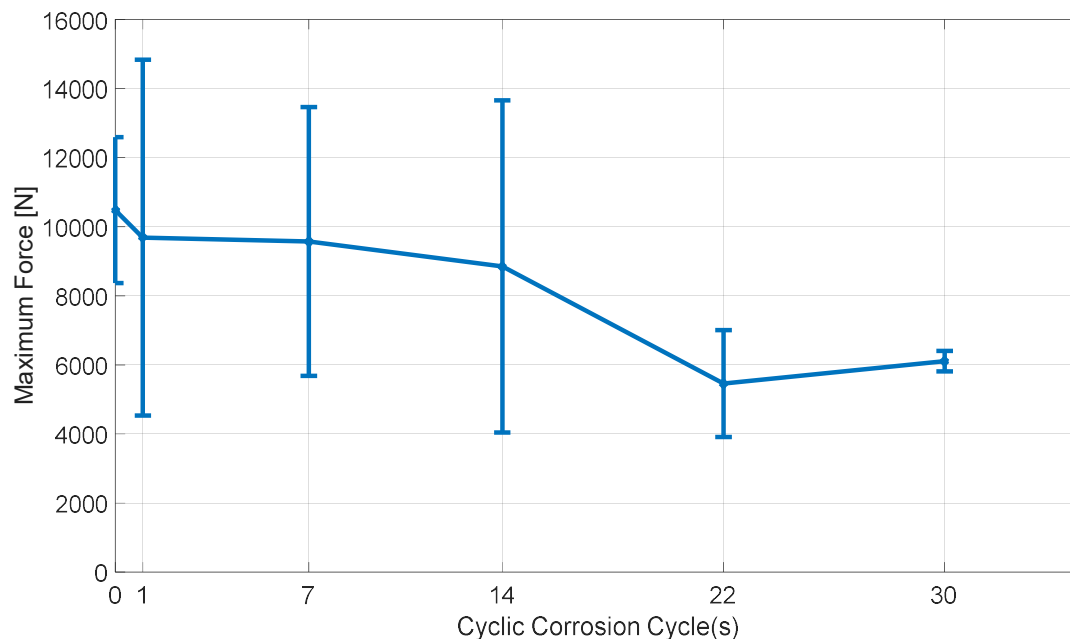


Figure 3-7: LTC evolution of Epibond bonded-only DLJs with corrosion cycle(s)

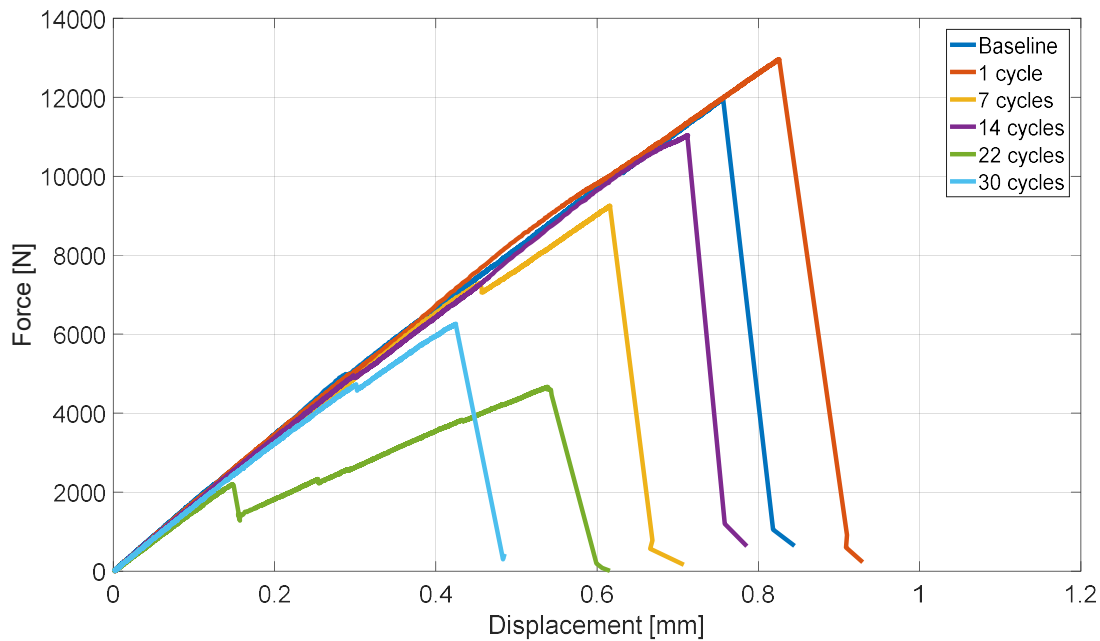


Figure 3-8: Representative load/displacement curves (Epibond)

3.2.3 Effect of cyclic corrosion on bolted-only DLJs

Bolted-only DLJs show an opposite trend with bonded-only DLJs. Although there is increasing corrosion on Al surface (Figure A-13 to Figure A-18), LTC of bolted-only DLJs is not significantly affected (Table 3-1 and Figure 3-9). The FMs of bolted-only DLJs are always bolt shear off. Although crevice corrosion is one of the biggest drawbacks of bolted joining methods and Al surface is corroded badly, bolting strength is still stable. It is considered that bolts are only corroded facially, and they are still able to work.

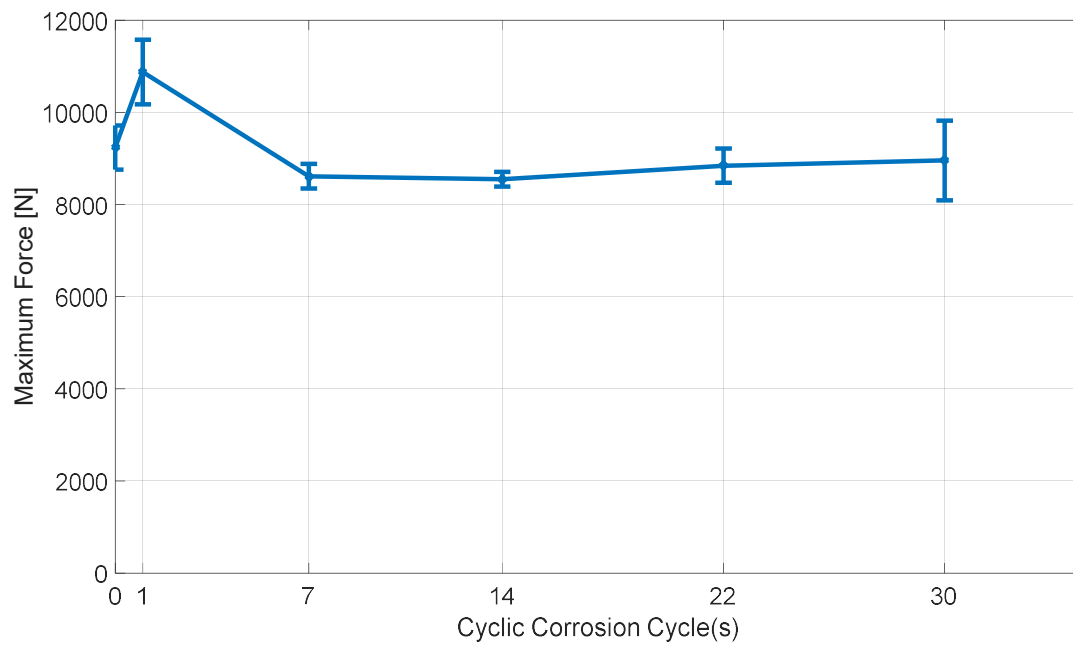


Figure 3-9: LTC evolution of bolted-only DLJs with corrosion cycle(s)

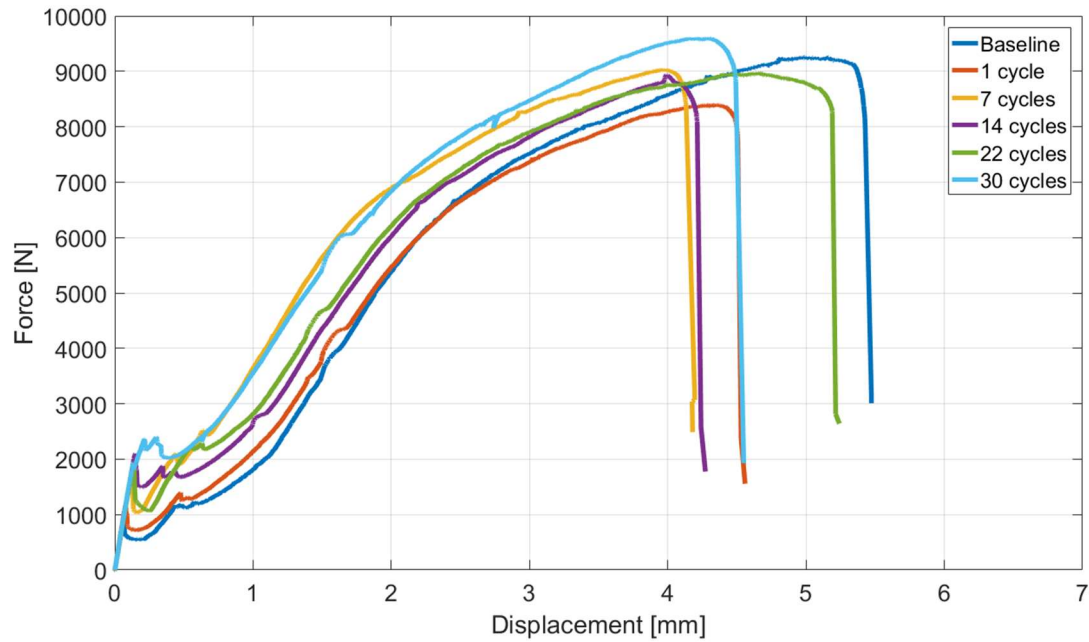


Figure 3-10: Representative load/displacement curves (bolted-only)

3.2.4 Effect of cyclic corrosion on hybrid bonded/bolted joints

Hybrid bonded/bolted DLJs have a similar bonding strength revolution with Uralane bonded-only DLJs, decreasing from 10.03kN to 2.44 kN after 30 cycles. Only about 24% of strength are retained at the end. From Figure 3-11, it can be noticed that bonding strength of hybrid bonded/bolted DLJs has a significant reduce (42%) from 10.88 kN to 6.35 kN after one week while bonded-only samples with the same adhesive has this similar change after 2 weeks. The bonding strength reduction in next 2 weeks (14 cycles and 22 cycles) is 22%, 52% respectively. Figure 3-12 shows that the bonding peak in hybrid bonded/bolted DLJs decreases obviously with corrosion cycles. At the end (after 22 cycles and 30 cycles), load/displacement of hybrid bonded/bolted DLJs is almost same with bolted-only DLJs, which means adhesive bonding has lost its function and only bolting is able to work.

Figure 3-11 and Table 3-1 shows that bolting peak in hybrid bonded/bolted DLJs is not affected significantly by corrosion. It remains the same level after 30 cycles. Figure 3-12 also evidences this find. The load/displacement curves of bolting are always the same. The break elongation scatter is given by test variance, which is not significant.

From Figure A-19 to Figure A-24 are FMs of hybrid bonded/bolted DLJs. FMs of adhesive bonding are always interfacial adhesive failure and FMs of bolting are always bolt shear off, which are same with Uralane bonded-only DLJs and bolted-only DLJs. It is clear that there is increasing corrosion on Al surface and remaining adhesive after tensile test moves from Al surface to CF RTP surface. Corrosion weakens the bond between adhesive and Al surface and as a result, the interfacial failure happens on Al surface after certain cycles. Figure A-20 shows that crevice corrosion starts at the overlap edge after 1 cycle. Figure A-21 tells that corrosion starts to be clear

after 1 week, which has the same trend with bonding peak (Figure 3-11). Figure A-23 and Figure A-24 report that, almost all the overlap areas on Al surface are corroded and almost no adhesive remains on Al surface. In conclusion, corrosion on Al surface shows a similar trend with bonding peak variation and however has no influence on bolting strength.

Surface measurement doesn't show any visible pit on Al surface. Hybrid bonded/bolted DLJs are using polyurethane-based adhesive, which can absorb moisture. Therefore, most oxide film are corroded after 30 cyclic and aluminum corrosion requires more time to show up.

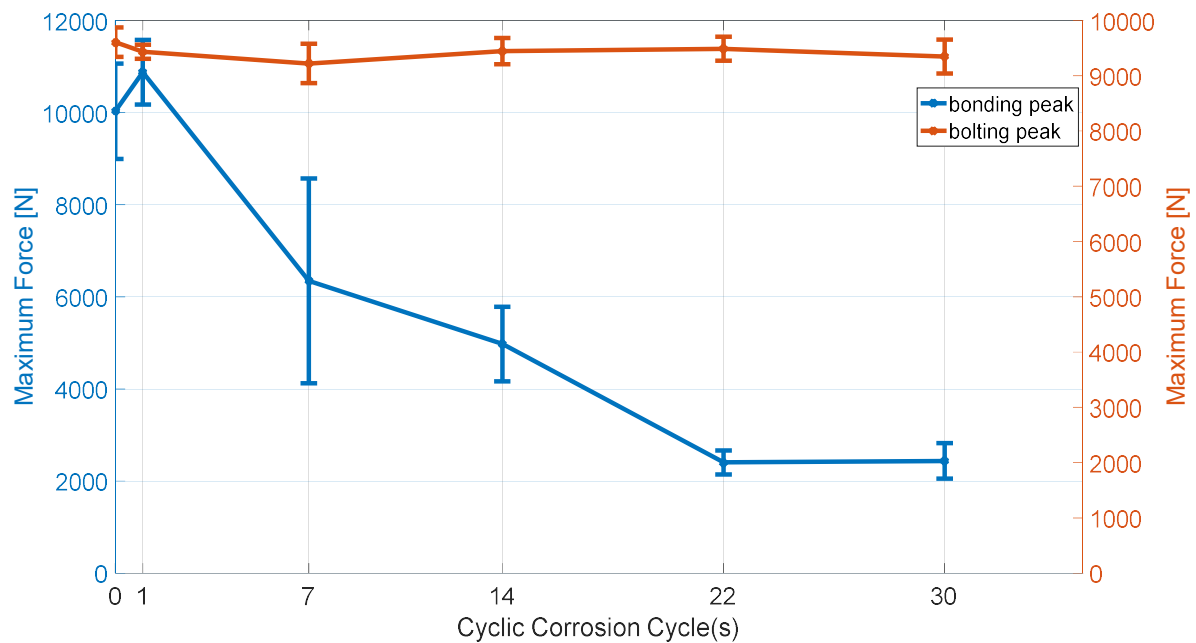


Figure 3-11: LTC evolution of hybrid bonded/bolted DLJs with corrosion cycle(s)

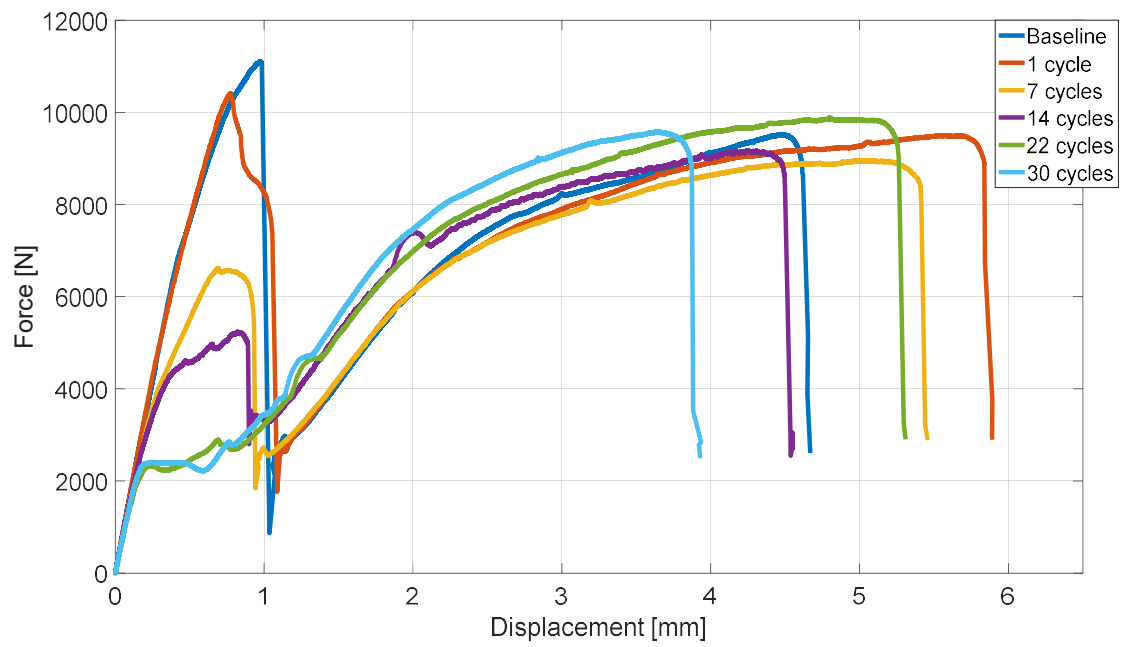


Figure 3-12: Representative load/displacement curves (hybrid bolted/bonded)

3.3 Effect of different joining methods

In this chapter, the performance of different joining methods are compared. At first, bonded-only DLJs with 2 different adhesives are discussed. Based on various adhesive characteristics, these two kinds of DLJs show different corrosion resistance. Then Uralane adhesive is discussed by comparing its performance in bonded-only DLJs with it in hybrid bonded/bolted DLJs. At last, the performance of bolting-only and bonding-only is compared.

3.3.1 Adhesive bonding-only joints

Figure 3-13 shows the difference of LTC revolution between bonded-only DLJs with 2 adhesives. It is clear that Epibond bonded-only DLJs has better corrosion resistance. Although both are not affected obviously in 2 weeks, Uralane bonded-only DLJs shows more decrease after 2 weeks. After 2 weeks, about 85% of strength remains for both bonded-only DLJs and in the next 2 weeks, 43% and 15.6% are left respectively for Uralane bonded-only DLJs while 52% and 58% are left for Epibond bonded-only DLJs. Epibond bonded-only DLJs can be thought stable in the first 2 weeks considering error and deviation and visible degradation is not found until 22 cycles. On the contrary, Uralane bonded-only DLJs are suffering a continual degradation with corrosion cycles.

This find is supported not only by the LTC revolution curves (Figure 3-13) but also by the Failure mode analysis (Figure A-25). It is clear that, increasing surface degradation occurs on surface of Uralane bonded-only DLJs, which starts being visible after 1 week. After 30 days, uniform degradation is shown on Al surface. Visible surface degradation starts on Epibond bonded-only DLJs after almost 22 cycles. There is still little corrosion after 30 cycles. The remaining Uralane adhesive after tensile test moves from Al surface to CFRTP surface while most Epibond adhesive

still remains on Al surface (Figure A-26). The trend that more visible corrosion on Al surface means more adhesive remains on CFRTP surface is followed by both of them.

In conclusion, bonded-only DLJs are affected by corrosion since adhesive is sensitive with substrate surface. The influence of corrosion depends on corrosion rate and further adhesive itself. Using different adhesive characteristics can have different performance.

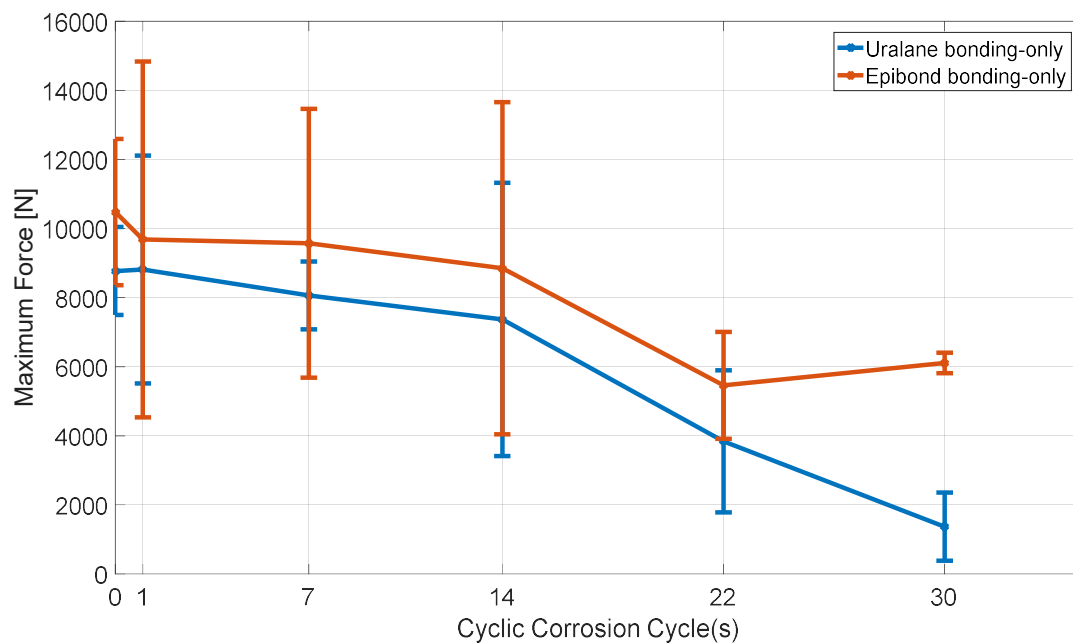


Figure 3-13: LTC revolution of bonded-only DLJs (Uralane vs Epibond)

3.3.2 Uralane vs Uralane hybrid bonding and bolting joints

Hybrid bonded/bolted DLJs perform a faster degradation than bonded-only DLJs with the same adhesive (Table 3-1). Figure 3-14 shows that, 40% of bonding LTC of hybrid joints reduces after 1 week while no significant change occurs on Uralane bonded-only DLJs. Uralane bonded-only DLJs starts continuous decrease one week later (after 14 cycles). After 30 cycles, both of these DLJs lose their function and only about 20% of strength remains.

Figure A-27 and Figure A-28 support this find. Worse corrosion occurs on hybrid joints than Uralane bonded-only DLJs at the same corrosion cycle. Visible corrosion shows up after 1 week for hybrid joints while it happens to Uralane bonded-only after 2 weeks. Remaining adhesives has the same change. More adhesive remains on CFRTP surface for hybrid bonded/bolted joints. After 22 cycles, all the adhesives stay on CFRTP surface of hybrid joints and it happens to Uralane bonded-only DLJs after 30 cycles.

Hence, it is clear that bolting system has a negative influence on adhesive bonding of hybrid bonded/bolted joints. It speeds up the corrosion rate on Al surface and as a result, the bonding strength decrease faster.

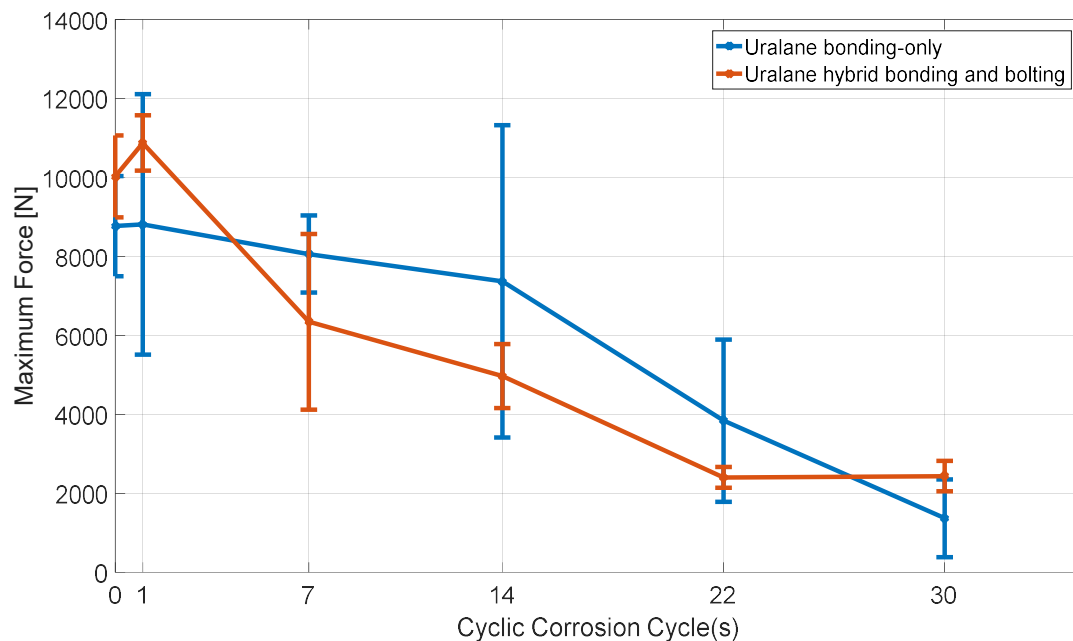


Figure 3-14: LTC revolution (Uralane vs Uralane hybrid bonding and bolting)

3.3.3 Bonding-only vs bolting-only

Bolted-only DLJs shows an opposite performance with bonded-only DLJs. Although more corrosion occurs on Al surface of bolted-only joints (Figure A-29), the strength is not affected significantly (Table 3-1). Figure 3-15 compares the LTC revolution curves between bond-only DLJs and bolted-only DLJs. No matter which adhesive is used, strength of bonded-only DLJs decreases with corrosion cycles while bolting strength remains stable. In hybrid bonded/bolted DLJs, the same trend is followed. Figure 3-16 shows that bonding strength of hybrid bonded/bolted joints reduces gradually, and bolting strength is stable. Figure A-29 shows that, corrosion develops faster on bolted-only joints, which is one of the main drawbacks of bolted joints. Bolts and nuts are also corroded with corrosion test.

Bolting relies on bolts and nuts, not on the substrates surface. Corrosion on substrate surface has no negative influence on bolts strength. There is corrosion on the surface of bolts and nuts, which however requires more time to affect their strength. On the contract, adhesive bonding is sensitive with substrate surface although adhesive can reduce corrosion by preventing salt spray. Even little corrosion will result strength lose. Therefore, although adhesive bonding has better corrosion resistance, it is easier to lose function once corrosion happens.

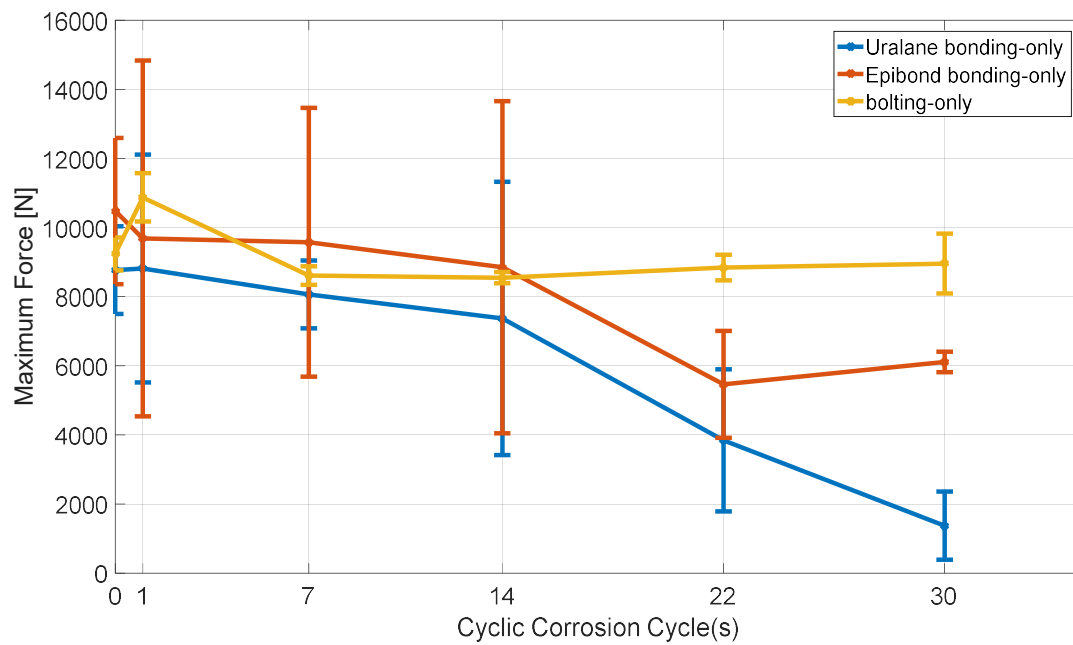


Figure 3-15: LTC revolution (Bolting-only vs Bonding-only)

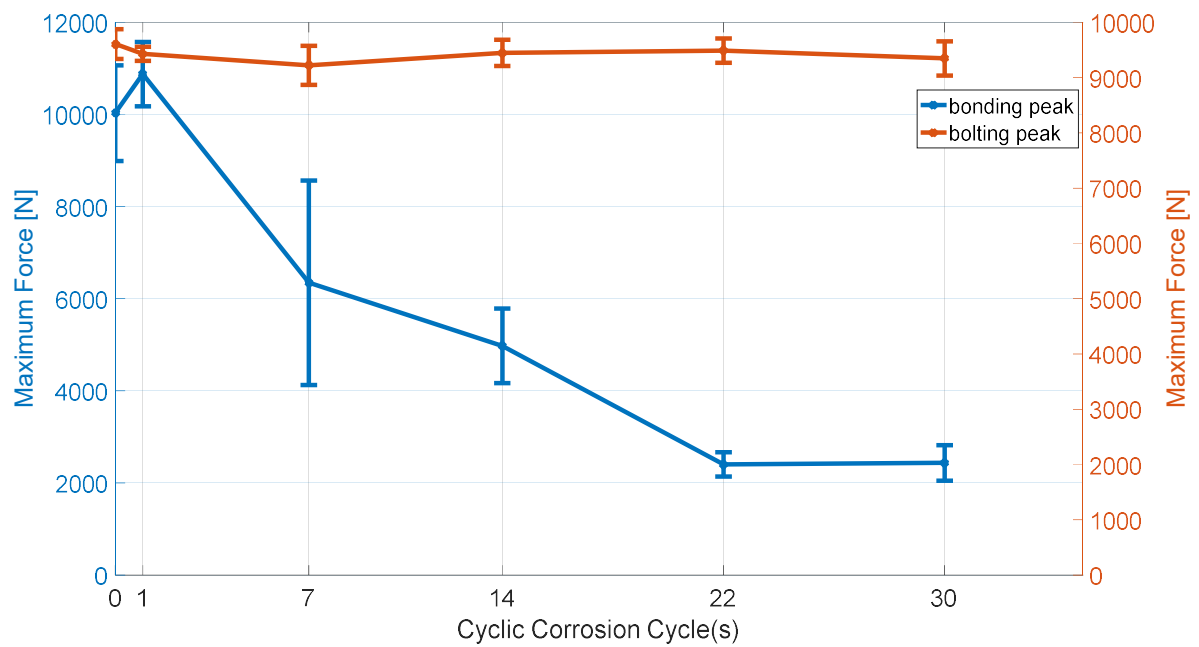


Figure 3-16: LTC revolution of hybrid DLJs (Bonding vs Bolting)

3.4 Effect of bolts on corrosion

In theory, Al surface around the hole should be corroded preferentially because of crevice corrosion. However, the result is opposite when bolts and preload are applied. There is a circular uncorroded area around the hole, shown in Figure A-30. Al overlap area is only corroded uniformly for bonded-only DLJs.

Figure A-31 shows the variation of uncorroded area. After 1 week, bolted-only DLJs has a clear uncorroded “ring” around the hole and hybrid joints has the same visible “ring” after 22 cycles. The diameter of uncorroded “ring” decreases with corrosion cycles, as shown in Figure 3-17. The average diameter of all uncorroded area is 8.97 mm, shown in Table 3-2

One possible reason is the effect of bolting and preload. According to Figure A-32, theoretical diameter of stress circle that is caused by bolting preload can be calculated as 15.97 mm. The diameter of washer is known as 8.5 mm. The diameter of uncorroded “ring” is between them. Figure 3-17 shows a clear decrease of uncorroded “ring”, so it can be thought that the outer area of stress circles is affected by corrosion and salt spray can diffuse gradually.

So, one interesting question will be whether the uncorroded “ring” would appear if zero preload is applied.

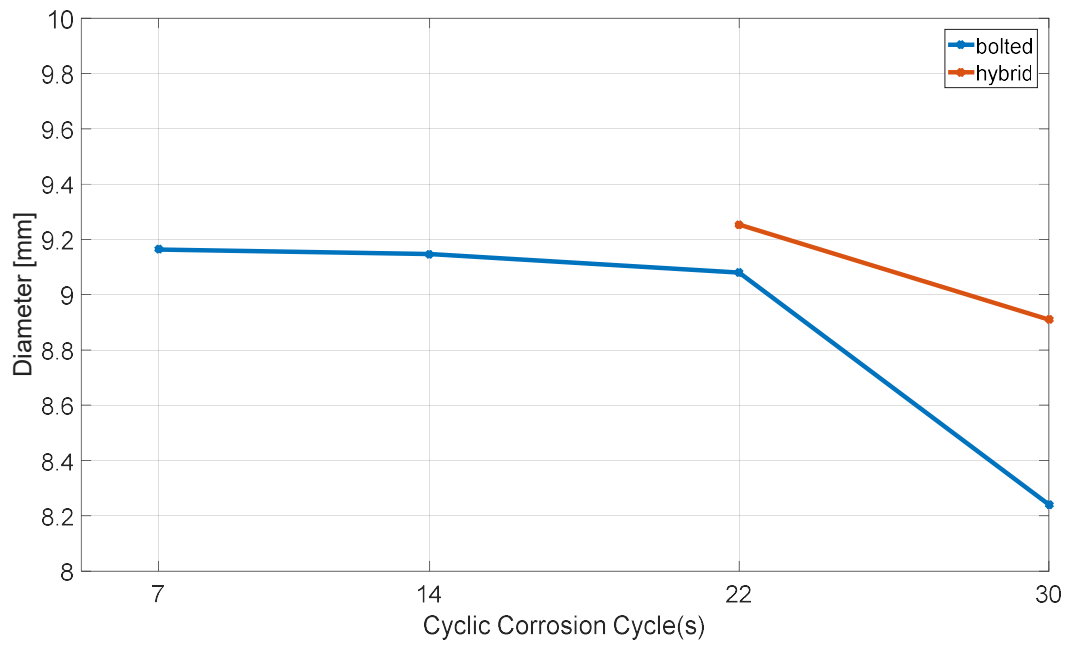


Figure 3-17: Diameter evolution of uncorroded area

Table 3-2: Diameter of uncorroded area, washer and stress circle

Average diameter of uncorroded "ring", [mm]	8.97
Diameter of washers, [mm]	8.5
Theoretical diameter of stress circle, [mm]	15.97

4 Conclusion

In this research, the effect of cyclic corrosion on static strength of composite/AA 6060 DLJs is investigated. DLJs are made of CFRTP and AA 6060 with three different joining methods: bonded-only, bolted-only or hybrid bolted/bonded. To this scope, cyclic corrosion test is carried out and static tensile test follows during corrosion test. Corresponding failure mode inspection is provided at the end. From the experimental results, it is possible to state that:

- The strength of bonded-only DLJs decreases with corrosion cycles. After 14 cycles, Uralane bonded-only joints start to significantly lose their strength.
- Adhesive characteristics can affect corrosion resistance. Bonded-only DLJs with polyurethane-based adhesive shows a higher sensitivity to corrosion than bonded-only DLJs with epoxy adhesive.
- Bolted DLJs suffer worse corrosion than bonded-only DLJs, which however doesn't significantly affect their strength. Bolted DLJs requires more time to degrade with corrosion.
- Bolting in hybrid bonded/bolted DLJs helps corrosion, resulting a faster degradation of bonding strength. On the contrary, adhesive doesn't affect the work of bolting.

These findings can provide an effective support during the design of joining methods for automotive applications that are required to work in severe environmental condition. Although adhesive bonding can slow down corrosion rate, bonded-only joints are more sensitive with corrosion than bolted-only joints. Hybrid bonded/bolted joints have the advantages from both joining methods in normal condition. However, bolting can have a negative influence on bonding strength by accelerating corrosion.

5 Continuation work

This research can be considered as one part of several tests made for studying the complete corrosion resistance of joints. Due to local material availability and time limit, only CFRTTP composite material and aluminum alloy are used and only effect of cyclic corrosion on joints' static strength is evaluated. Therefore, continuation work can extend materials and tests.

Some continuation work can be also based on results of this research. Polyurethane-based adhesive bonded-only joints show different behavior with corrosion from epoxy adhesive bonded-only joints, which can be analyzed deeper. The uncorroded “ring” appear when bolts and preload are applied. So another interesting question is whether the uncorroded “ring” would appear if the zero preload is applied.

Here are some recommendations are therefore given:

- Glass fiber reinforced composite material can be used for joints since it is also an attractive material in automotive field.
- Dynamic strength of DLJs in extreme condition is also worthy to evaluate. Static and dynamic strength of materials with cyclic corrosion can provide a complete image of their corrosion resistance.

- More types of adhesive can be used for bonded-only joints in order to find out the effect of adhesive characteristics on their performance with cyclic corrosion.
- Bolted-only joints without preload or hybrid bonded/bolted without preload can be tested as well, which can evaluate the effect of preload on corrosion.

References

- [1] 2030 framework for climate&energy, European Council, 2014.
- [2] K. S. C. H. Jin Zhang, "Hybrid composite laminates reinforced with glass/carbon woven fabrics for lightweight load bearing structures," *Materials&Design*, vol. 36, pp. 75-80, 2012.
- [3] D. D. E. John D. Buckley, Carbon-Carbon Materials and Composites, Noyes publications, 1993.
- [4] T. Stephen W, "Strength Characteristics of Composite Materials."
- [5] Isaac M. Daniel, Ori Ishai, Engineering mechanics of composite materials, OXFORD UNIVERSITY PRESS, 2006.
- [6] Enrico Mangino, Giuseppe Pitarresi, Joe Carruthers, "The future use of structural composite materials in the automotive industry," *International Journal of Vehicle Design*, 2007.

- [7] AHiggins, "Adhesive bonding of aircraft structures," *International Journal of Adhesion and Adhesive*, vol. 20, no. 5, pp. 367-376, 2000.
- [8] S. A. N. a. K. Sakai, "Effect of Cyclic Heat, Humidity, and Joining Method on the Static and Dynamic Performance of Lightweight Multimaterial Single-Lap Joints," *Journal of Manufacturing Science and Engineering*, vol. 137, no. 5, p. 11, 2014.
- [9] L. E. E. E. V.Fiorea, "Salt spray fog ageing of hybrid composite/metal rivet joints for automotive applications," *Composites*, vol. 108, pp. 65-74, 2017.
- [10] H. S. BinWeib, "Tensile behavior contrast of basalt and glass fibers after chemical treatment," *Materials&Design*, vol. 31, no. 9, pp. 4244-4250, 2010.
- [11] Jean-Paul Kabche, Vincent Caccese, Keith A. Berube, Randy Bragg, "Experimental characterization of hybrid composite-to-metal bolted joints under flexural loading," *Composites Part B: Engineering*, vol. 38, no. 1, pp. 66-78, 2007.
- [12] F. Lambiase, "Mechanical behaviour of polymer–metal hybrid joints produced by clinching using different tools," *Materials & Design*, vol. 87, pp. 608-618, 2015.
- [13] F. G. B. A. V.Fiorea, "On the mechanical behavior of BFRP to aluminum AA6086 mixed joints," *Composites*, vol. 48, pp. 79-87, 2013.
- [14] M. A. RyosukeMatsuzakia, "Improving performance of GFRP/aluminum single lap joints using bolted/co-cured hybrid method," *Composites*, vol. 39, no. 2, pp. 154-163, 2008.

- [15] M.D. Downey, Toughening of carbon fiber-reinforced epoxy polymer composites via copolymer and graphene nanoplatelets, PHD dissertation. Michigan State University, 2016.
- [16] Byung Chul Kim, Sang Wook Park, Dai Gil Lee, "Fracture toughness of the nano-particle reinforced epoxy composite," *Composite Structure*, vol. 86, no. 1-3, pp. 69-77, 2008.
- [17] P. Mallick, Fiber-reinforced composites: materials, manufacturing, and design, 2007.
- [18] P. Mallick, Materials, Design and manufacturing for lightweight vehicle, 2010.
- [19] I. P. T.A Barnes, "Joining techniques for aluminum spaceframes used in automobiles: Part II - adhesive bonding and mechanical fasteners," *Journal of Materials Processing Technology*, vol. 99, no. 1-3, pp. 72-79, 2000.
- [20] Swift EJ Jr, Perdigao J, Combe EC, Simpson CH Jr, Nunes MF, "effect of restorative and adhesive curing methods on dentin bond strengths," *American journal of Dentistry*, vol. 14, no. 3, pp. 137-140, 2001.
- [21] S. Z. J. D. W. K. Long Hao, "Evolution of corrosion of MnCuP weathering steel submitted to wet/dry cyclic tests in a simulated coastal atmosphere," *Corrosion Science*, vol. 58, pp. 175-180, 2012.
- [22] Simpson. C H, Ray, C J, Skerry B S, "Accelerated corrosion testing of industrial maintenance paints using a cyclic corrosion weathering method," *Journal of protective coating & linings*, vol. 8, no. 5, pp. 28-36, 1991.

- [23] Z. Z. E. A. K. T. B. Z. Songjie Li, "Evaluation of susceptibility of high strength steels to delay fracture by using cyclic corrosion test and slow strain rate test," *Corrosion Science*, vol. 52, no. 5, pp. 1660-1667, 2010.
- [24] GMW14872 Cyclic corrosion laboratory test, General Motors, 2010.
- [25] GM 9540P accelerated corrosion test, General Motors, 1997.
- [26] SAE J2334 Laboratory cyclic corrosion test, SAE international surface vehicle standard, 2003.
- [27] Y. C. X. Z. G. L. J. C. Hao Jianga, "Fatigue degradation after salt spray ageing of electromagnetically riveted joints for CFRP/Al hybrid structure," *Materials&Design*, vol. 142, pp. 297-307, 2018.
- [28] H.-S. Chen, "The static and fatigue strength of bolted joints in composites with hygrothermal cycling," *Composites Structures*, vol. 52, no. 3-4, pp. 295-306, 2001.
- [29] D. M. Aylor and J. N. Murray, "The Effect of a Seawater Environment on the Galvanic Corrosion Behavior of Graphite/Epoxy Composites Coupled to Metals," Defence Technical Information Center, 1992.
- [30] Tien-Cuong Nguyen, YuBai, Xiao-Ling Zhao, Riadh Al-Mahaidi, "Durability of steel/CFRP double strap joints exposed to sea water, cyclic temperature and humidity," *Composite Structures*, vol. 94, no. 5, pp. 1834-1845, 2012.

- [31] "Changing the Future of Carbon Fiber Reinforced Thermoplastic Composites," Continental Structural Plastics, TEIJIN.
- [32] William A. Lanford, Robert S. Alwitt, Christopher K. Dyer, "Hydrogen Profiles of Anodic Aluminum Oxide Films," *Journal of the Electrochemical Society*, 1980.
- [33] J. J. augustynski, R.P. frankenthal, J.P. kruger, "in "passivity of metals," *The electrochemical society*, 1978.
- [34] R. Foley, "corrosion," p. 42, 1986.
- [35] T. E. Graedel, "Corrosion Mechanisms for Aluminum Exposed to the Atmosphere," *Journal of the Electrochemical Society*, 1989.

Appendix

Uralane bonded-only DLJS



Figure A-1: FM of Uralane bonded-only DLJs - 0 cycle

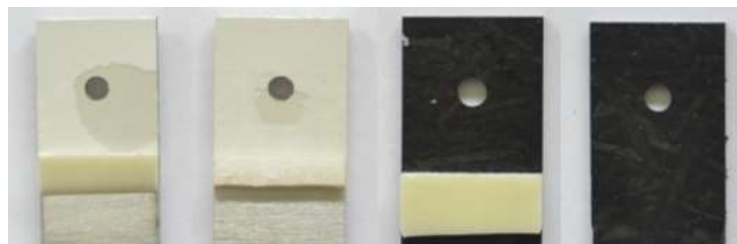


Figure A-2: FM of Uralane bonded-only DLJs -1 cycle



Figure A-3: FM of Uralane bonded-only DLJs -7 cycles



Figure A-4: FM of Uralane bonded-only DLJs – 14 cycles



Figure A-5: FM of Uralane bonded-only DLJs - 22 cycles



Figure A-6: FM of Uralane bonded-only DLJs - 30 cycles

Epibond bonded-only DLJS



Figure A-7: FM of Epibond bonded-only DLJs - 0 cycle

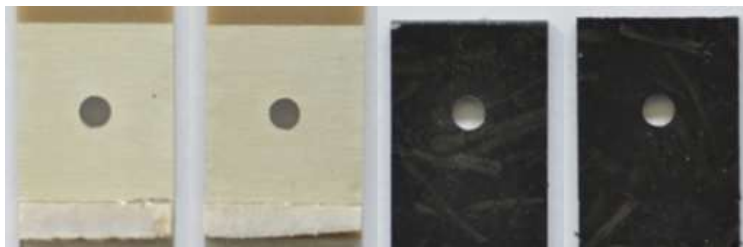


Figure A-8: FM of Epibond bonded-only DLJs - 1 cycle

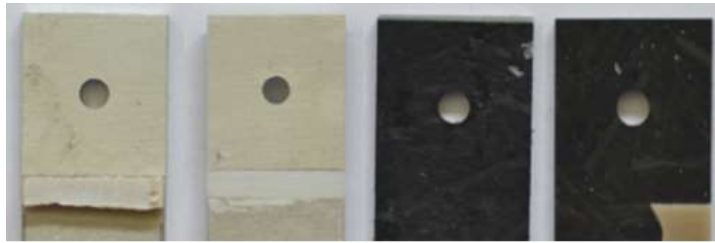


Figure A-9: FM of Epibond bonded-only DLJs - 7 cycles



Figure A-10: FM of Epibond bonded-only DLJs - 14 cycles



Figure A-11: FM of Epibond bonded-only DLJs - 22 cycles



Figure A-12: FM of Epibond bonded-only DLJs - 30 cycles

Bolted-only DLJs (M4)

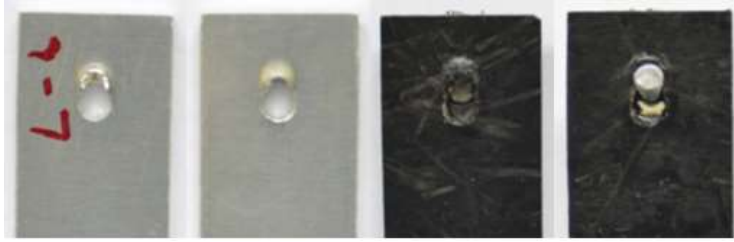


Figure A-13: FM of bolted-only DLJs - 0 cycle



Figure A-14: FM of bolted-only DLJs - 1 cycle



Figure A-15: FM of bolted-only DLJs - 7 cycles



Figure A-16: FM of bolted-only DLJs - 14 cycles

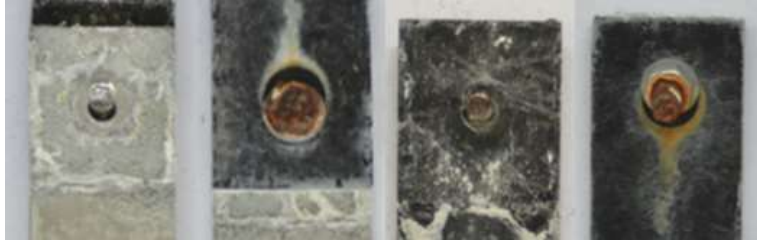


Figure A-17: FM of bolted-only DLJs - 22 cycles



Figure A-18: FM of bolted-only DLJs - 30 cycles

Hybrid bonded/bolted DLJs (M4/Uralane)



Figure A-19: FM of hybrid bonded/bolted DLJs - 0 cycle



Figure A-20: FM of hybrid bonded/bolted DLJs - 1 cycle

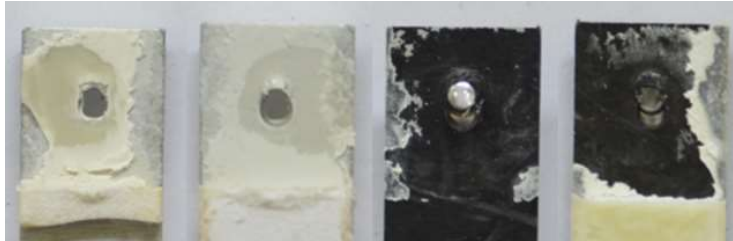


Figure A-21: FM of hybrid bonded/bolted DLJs - 7 cycles



Figure A-22: FM of hybrid bonded/bolted DLJs - 14 cycles



Figure A-23: FM of hybrid bonded/bolted DLJs - 22 cycles



Figure A-24: FM of hybrid bonded/bolted DLJs - 30 cycles

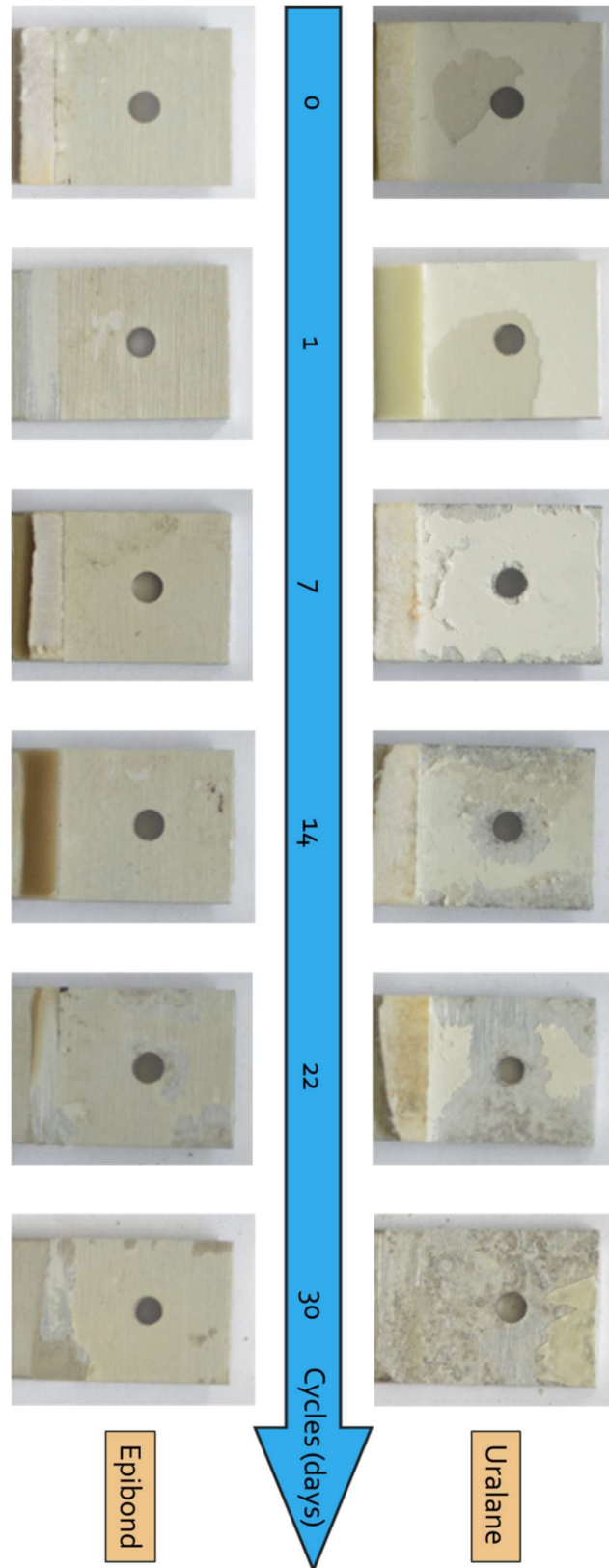


Figure A-25: Al surface degradation with corrosion cycle(s) (Adhesive bonding-only joints)

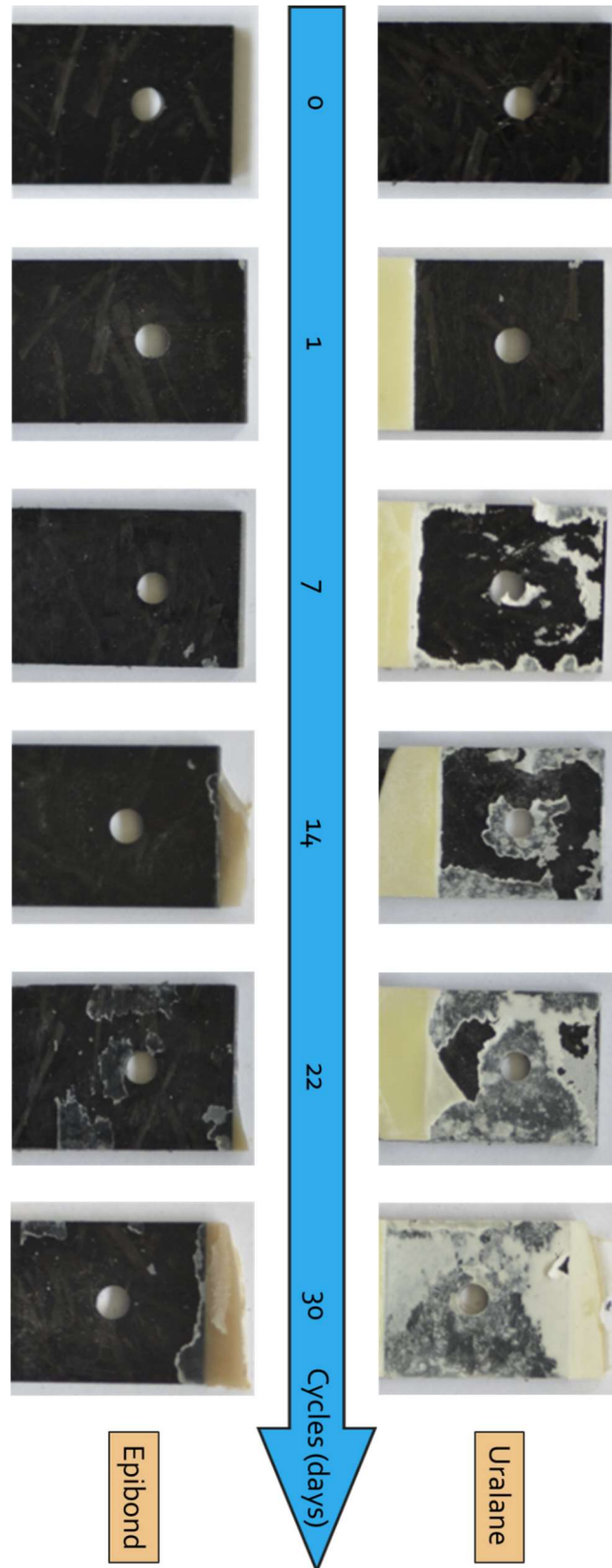


Figure A-26: Remaining adhesive on CFRTP surface (Adhesive bonding-only joints)

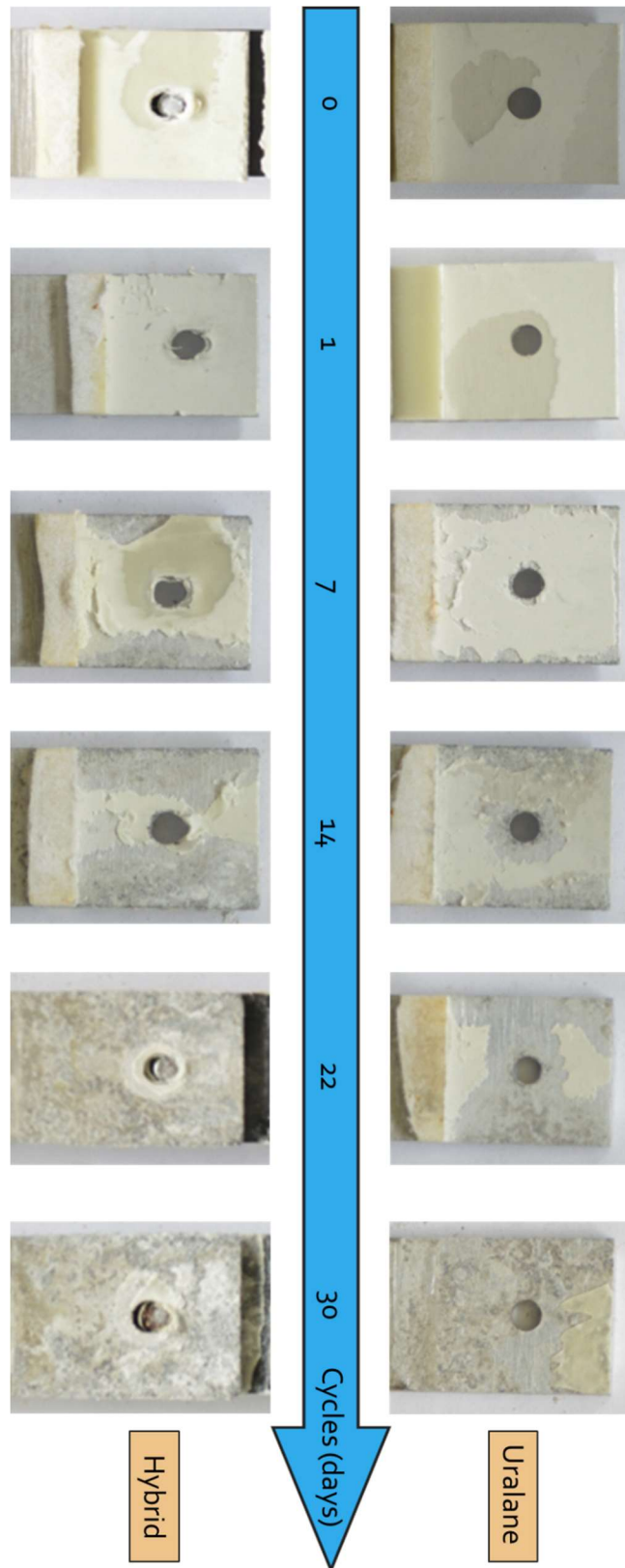


Figure A-27: Al surface degradation (Uralane vs Uralane hybrid bonding/bolting)

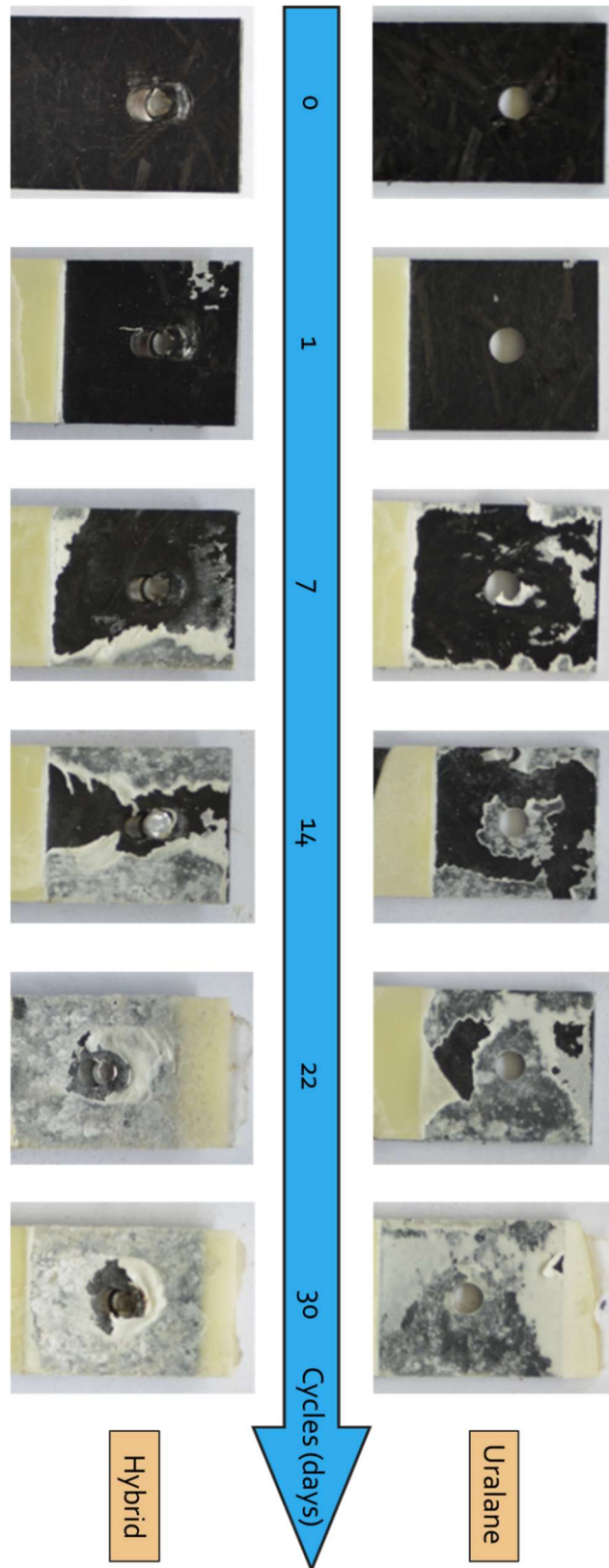


Figure A-28: Remaining adhesive on CFRTP (Uralane vs Uralane hybrid bonding/bolting)

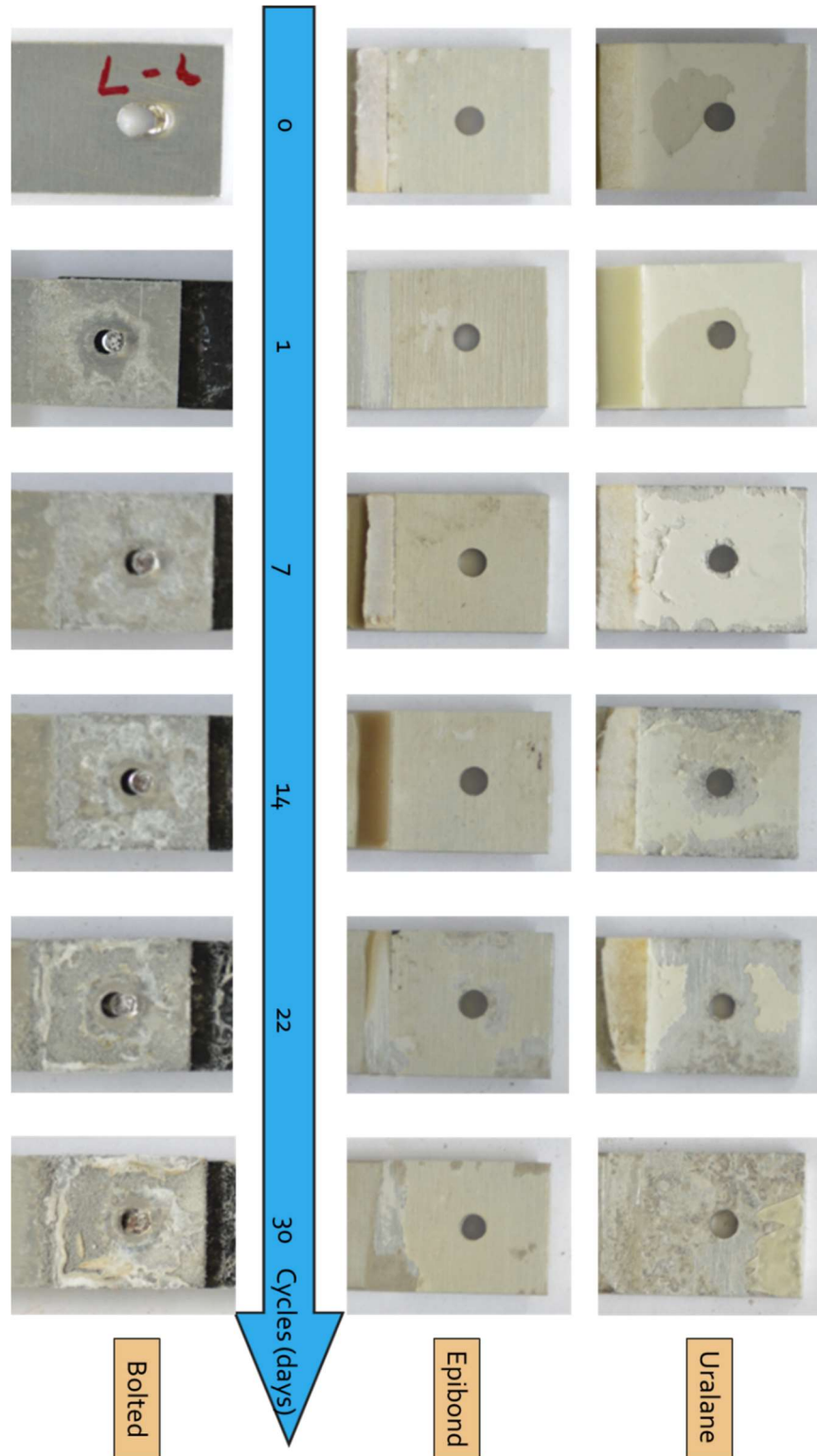


Figure A-29: Al surface degradation with corrosion cycle(s) (Bonding-only vs Bolting-only)



A



B



C



D

Figure A-30 Overlap surface comparison

A: Uralane; B: Epibond; C: Hybrid; D: Bolted

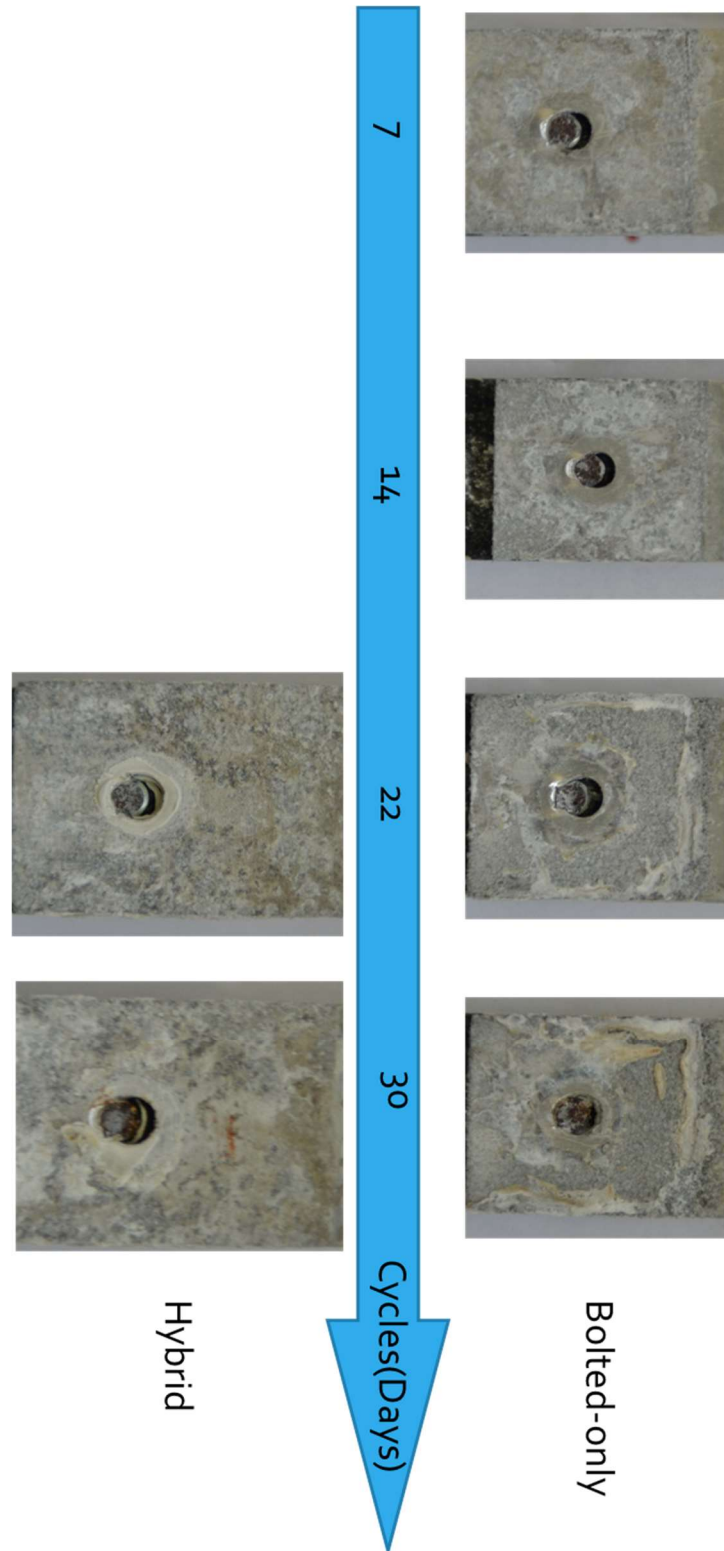


Figure A-31: Decreasing uncorroded area

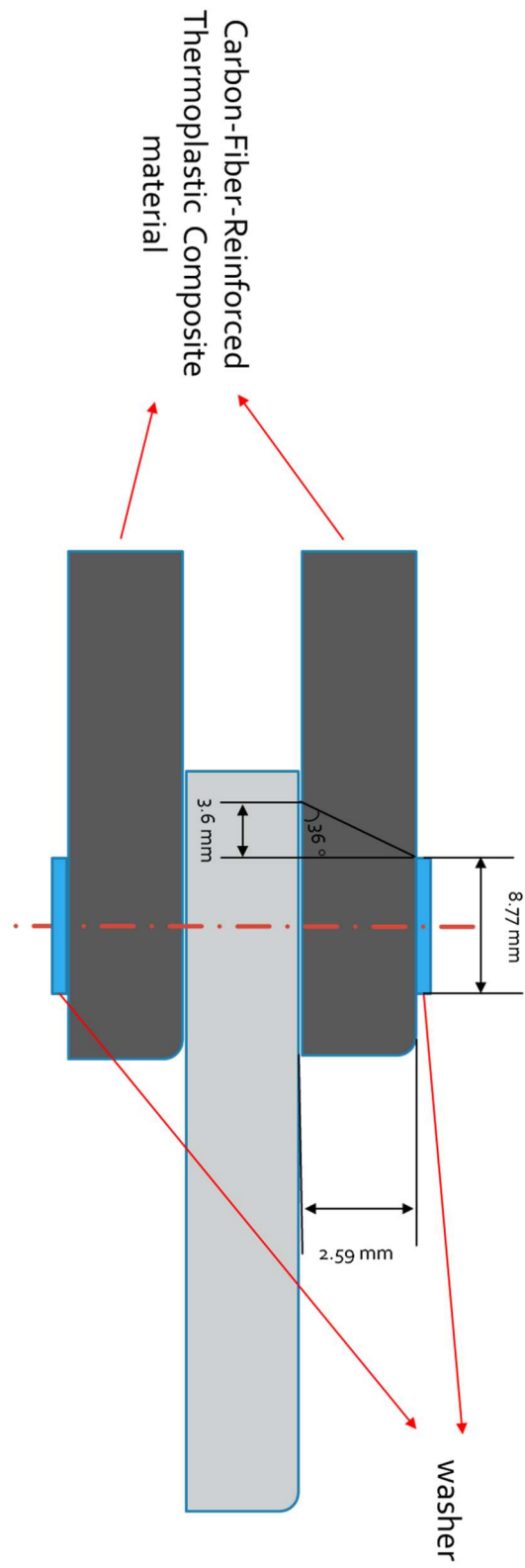


Figure A-32: A scheme of bolted-only DLJs

