Pacific Northwest
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PNNL-35364

# Mitigating Corrosion in Mg Sheet in Conjunction with a Sheet-Joining Method that Satisfies Structural Requirements within Sub-assemblies

CRADA 392

April 2023

Saumyadeep Jana (PNNL)
Aashish Rohatgi (PNNL)
Bill Kokosza (Magna-Stronach Centre for Innovation)

U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Pacific Northwest National Laboratory
Richland, Washington 99354

# Cooperative Research and Development Agreement (CRADA) Final Report

**April 2023**

In accordance with Requirements set forth in the terms of the CRADA, this document is the CRADA Final Report, including a list of Subject Inventions, to be provided to PNNL Information Release who will forward to the DOE Office of Scientific and Technical Information as part of the commitment to the public to demonstrate results of federally funded research.

**Parties to the Agreement: Pacific Northwest National Laboratory (PNNL) and Magna-Stronach Centre for Innovation (Magna-SCFI)**

**CRADA number: 392**

**CRADA Title: Mitigating Corrosion in Mg Sheet in Conjunction with a Sheet-Joining Method that Satisfies Structural Requirements within Sub-assemblies**

**Responsible Technical Contact at DOE Lab: Aashish Rohatgi**

**Name and Email Address of POC at Company: Bill Kokosza, Bill.Kokosza@magna.com**

**DOE Program Office: EERE-Vehicle Technologies Office**

**Joint Work Statement Funding Table showing DOE funding commitment:**

| **Funding** | **Project Year 1** | **Project Year 2** | **TOTAL** |
|---|---|---|---|
| **DOE** | $150,000 | $150,000 | $300,000 |
| **Other** | 0 | 0 | 0 |
| **Total Govt.** | $150,000 | $150,000 | $300,000 |
| | | | |
| **Participant** | | | |
| In-Kind | $210,000 | $215,000 | $425,000 |
| Funds-In | 0 | 0 | 0 |
| FAC | 0 | 0 | 0 |
| **Total Participant** | $210,000 | $215,000 | $425,000 |
| | | | |
| **TOTAL CRADA VALUE** | $360,000 | $365,000 | $725,000 |

# Executive Summary

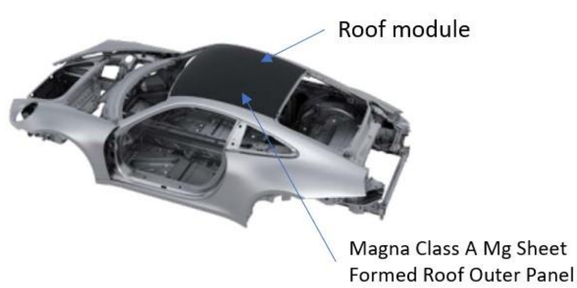
This work was undertaken as a LightMAT project funded by the DOE-Vehicles Technology Office. The goal of this work was to develop corrosion protection strategies that simultaneously mitigate corrosion and achieve Class-A surface finish for Mg components in automotive applications. While automotive metals such as steel and aluminum are protected against corrosion through a variety of coating schemes/packages, the efficacy of these existing coating schemes for Mg and Mg- joints is not clear and needs to be determined. Therefore, five commercially available coating schemes and two joining techniques (riveting and Arplas resistance spot welding) were evaluated. The corresponding individual Mg sheet coupons or Mg/Mg joint test coupons were provided by Magna that were then corrosion tested at PNNL using ASTM B117 procedure. The microstructures and mechanical properties of the coupons were analyzed to determine the effectiveness of the joining and corrosion mitigation strategies. Of the coating schemes evaluated, Henkel Bonderite MgC 2.0 pre-treatment + E-coat showed the best corrosion protection and surface finish for individual Mg coupons and Arplas resistance spot welded coupons. However, the strength of Mg/Mg welded joint was reduced after corrosion testing due to some corrosion at the weld nugget. Mg/Mg rivet joints in conjunction with Chemetall oxisilan pre-treatment + polyurethane coating showed good corrosion resistance and some discoloration on the surface finish. Coating schemes comprising pre-treatment with Alodine 5200 + E-coat or Bonderite 1455 + polyurethane coating, in conjunction with Al rivet joints, showed significant corrosion and extensive discoloration of the surface. We anticipate that the results from this work will provide useful guidance to the automotive industry in selecting the appropriate combinations of corrosion protection coatings and joining techniques to fabricate light-weight Mg-based automotive components.

# Summary of Research Results

## 1 Introduction

As the US automotive and heavy truck industries continue to push for lightweighting solutions, the use of magnesium (Mg) alloy sheet materials will naturally propagate in the future. A recent Multi-Material Lightweight Vehicle report [1] shows a 155 kg (47.5%) body-in-white mass reduction in a Multi-Material Lightweight Vehicle Mach-II design relative to a 2013 Fusion baseline vehicle, where Class A exterior body panels are warm-formed Mg sheets. The report also identified two major challenges related to the use of multi-material solutions: (1) corrosion; and (2) joining.

The process for forming magnesium sheet into automotive or heavy truck panels is one challenge, which is currently being effectively addressed for series production. Recently, Magna has been able to successfully form and manufacture the world's first production Class A exterior Mg sheet panel which has been used in commercial vehicles (see images below). Over 8000 production roofs have been successfully produced as part of this work. The next challenge in greater use of Mg sheet for lightweighting will be the effective joining and assembly (similar or dissimilar metals) of magnesium sheet panels to mitigate corrosion while simultaneously producing a Class-A surface finish. In the roof panel example shown below (Figure 1), corrosion mitigation was achievable but only due to the fact that the roof design was for modular bolt-on assembly. The roof was mounted to a support frame using only adhesive with no further physical or mechanical fastening method. The frame which was a molded composite structure was then bolted to the BIW (body-in-white). This assembly method is rare in the closure world. If the use of light-weight Mg panels needs to be extended to additional components, such as doors (inner and outer panels), hood, decklid, side panels, etc. some type of a joint needs to be introduced to provide more robust support that is not feasible with an adhesives-only design. However, a mechanical joint (similar/dissimilar metals) will introduce the problem of corrosion and may also adversely affect surface finish. **Therefore, the goal of this project was to develop strategies to join Mg with Mg/dissimilar metals while simultaneously mitigating corrosion and achieving Class-A surface finish.**




| Comparison | Weight Savings |
|---|---|
| Mg vs. Steel Roof | ≈ 69 % |
| Mg vs. Current Serial Al Roof | ≈ 30 % |

Figure 1: Class A magnesium sheet roof panel produced by Magna and weight savings as compared to a steel or an Al roof.

## 2 Objectives

This project used Mg panels welded using a proprietary resistance welding method developed by Magna's partner, Arplas Systems. A second partner, Henkel Technologies, provided their corrosion mitigation treatments/coatings specially formulated for Mg. Therefore, the project objective was to perform corrosion testing, microstructural characterization and mechanical testing to analyze the effectiveness of the joining and corrosion mitigation strategies and provide feedback to the industry to develop a commercially viable solution.

## 3 Experimental Approach

3.1 Test coupon fabrication: AZ31 was selected as the representative Mg sheet alloy for this project and the final test matrix comprised individual AZ31 coupons and Mg/Mg joint coupons. All the test coupons (with their respective coatings) were provided by Magna and delivered to PNNL for corrosion testing and post-corrosion characterization.

3.2 Corrosion protection coating packages: Several different corrosion protection coating packages were evaluated and are listed below:

*Coating package #A: Mg coupons were coated with a Henkel pre-treatment (HP) layer which is available under the name Bonderite MgC 2.0. This approach is termed as HP in the text.*

*Coating package #B: Mg coupons were coated with Henkel pre-treatment (HP) + E-coat where the E-coat is a polymer-based organic coating. This approach is termed as HPEC in the text.*

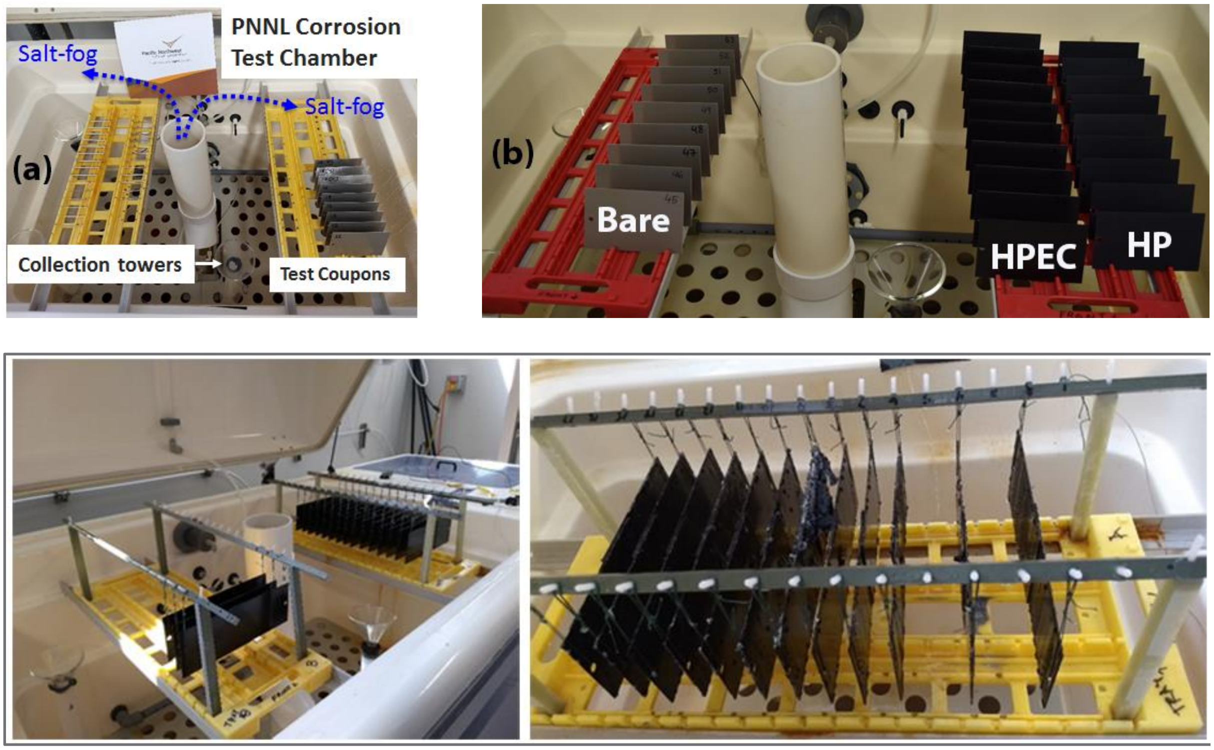
*Coating package #C: Mg coupons were pre-treated with Henkel Alodine 5200 and topped by E-coating. Henkel Alodine 5200 is a common pre-treatment method applied on Al, steel, etc. in the automotive industry.*

*Coating package #D: Mg coupons were coated with Chemetall oxisilan pre-treatment (10 or 20 $\mu$m thickness) followed by a "non-E-coat" E-coat (a polyurethane spray).*

*Coating package #E: Mg coupons were coated with Henkel Bonderite 1455 pre-treatment layer followed by a "non-E-coat" E-coat (a polyurethane spray). Bonderite 1455 is a common conversion coating for Al, Mg and steel.*

3.3 Individual Mg AZ31 coupons: The individual AZ31 coupons were 3 in. x 5 in. A majority of the testing and characterization was performed on coupons with coating packages #A (HP) and #B (HPEC).

3.4 Mg/Mg joint coupons: The three initial joining techniques identified by Magna were (i) Arplas resistance spot welding (RSW), (ii) breakaway stem rivet and (iii) clinch lock. Of these, only the first two were selected for testing. Unless noted otherwise, lap-joints were fabricated between 1.1 mm and 1.5 mm thick AZ31 sheets and the joint coupons measured 3 in. x 8.5 in. Additional details of joining techniques are described below. Majority of the testing and characterization was performed on Arplas RSW joints and rivet joints with coating package #C.




(c)

Figure 2 (a) Salt-fog test set-up at PNNL; (b) Bare and coated (HP and HPEC) individual AZ31 coupons placed in a rack for testing; (c) Two views of joint coupons hung in the corrosion chamber at PNNL.

i. Arplas resistance spot welding (RSW): Both Mg sheets were coated with coating package #B (HPEC). Experimental trials at Magna revealed that for a successful lap joint, such as the one tested in the current work, the pre-treatment layer (HP) had to be removed from the top sheet on both sides, and from the mating side of the bottom sheet. Removal of HP layer was found to affect the corrosion behavior of the joint.

ii. Breakaway stem rivet: For the rivet joints, closed-end 3/16 in. all-Al rivets were used. Additionally, two types of rivet hole configuration were evaluated: (1) rivet holes in deburred condition; and (2) rivet holes in non-deburred condition. A majority of the testing and characterization was performed on joints with coating package #C (Henkel Alodine 5200 pre-treatment + E-coating).

3.5 Corrosion Testing: Corrosion behavior of the base material and Mg/Mg joint coupons was determined according to the American Society for Testing and Materials (ASTM) B117 standard salt (sodium chloride) spray (fog) test method. As per ASTM B117, corrosion coupons were subjected to a continuous exposure of 5% salt fog at 35°C for a total duration of up to 1500 hours (~ 60 days) at a pH of 6.5–7.2. During ASTM B117 testing, corrosion coupons were retrieved at regular intervals, gently washed under tap water, and immediately dried. Subsequently, the coupons were weighed to record the change in weight due to corrosion/corrosion product build-up. The salt-fog set-up at PNNL is shown in Figure 2a, where a row of bare AZ31 coupons can be seen. Figure 2b shows a picture of test coupons in uncoated and in two types of coating conditions (package #A and #B). Figure 2c shows pictures of joint coupons hung vertically in the corrosion chamber.

3.6 Mechanical Testing: The effect of corrosion on joint strength was determined by testing samples before and after corrosion chamber exposure. Mechanical strength of joint coupons was determined through a room-temperature standard lap-shear test. Joint coupons are pulled to failure in a uniaxial testing machine at a constant crosshead velocity of 1 mm/min. In selected cases, mechanical robustness of the coatings, especially at the coating/base Mg AZ31 interface, was evaluated using nano-indentation technique. Metallographically polished samples of transverse cross-sections of corrosion tested coupons (e.g., package #A (HP) and package #B (HPEC)) were indented along the interface using a Berkovich indenter for a series of loads ranging from 10–200 mN. The positions of the indents were controlled to be at the coating/base Mg AZ31 interface, or within the coating. Subsequently, the indented locations were imaged in the SEM to examine for propensity and location of cracks (if any) around the indent or along the interface.

3.7 Microstructural Characterization: Post-mortem analysis of joint coupons was performed using various microscopy techniques (e.g., stereo microscope and scanning electron microscope (SEM)) before and after corrosion testing. For microstructural characterization, transverse cross-sections of corrosion tested coupons were prepared using standard metallographic techniques and imaged in a SEM. The samples were imaged using the back-scattered electron imaging mode to help distinguish corrosion products, protective coatings, and the base material.

# 4 Results and Discussion

The effectiveness of corrosion protection schemes (packages #A - #E) and the effect of corrosion test on the mechanical strength of different joining techniques (Arplas RSW and breakaway stem rivet) were evaluated in this study. The key results are presented and discussed below.

4.1 Base Material Corrosion Behavior: Figure 3 shows pictures of base AZ31 coupons in bare (uncoated), and with corrosion protection packages (#A, #B, #D and #E) in the as-received untested state and following corrosion testing per the ASTM B117 method for the durations listed. Figure 4 shows the plots of change in weight of the three types of coupons (i.e., bare untested AZ31, HP (package #A), and HPEC (package #B)) as a function of corrosion test duration.

No coatings (Bare coupon): Figure 3b shows that the bare AZ31 coupons experienced significant uniform and pitting corrosion after 1200 hours of testing, as evidenced by the mottled black and white surface and material loss at the edges, in contrast to the smooth, uniform, and shiny surface of the untested coupon, shown in Figure 3a. Bare AZ31 coupons, were tested in two separate batches and substantial weight gain, as high as 7% and plotted in Fig. 4, was observed in these coupons. This weight gain is attributed to the formation of $Mg(OH)_2$ due to the reaction between Mg and water vapor and appears as a white substance sticking on to the coupon surface.

Coating package #A (HP coupons): Discoloration and loss of shine is noted after 1200 hours of salt fog exposure, shown in Figure 3d compared to the untested coupon. As compared to bare Mg, HP coupons show a slight (<1%) weight loss which may be due to the pre-treatment coating being gradually removed over the duration of the test.

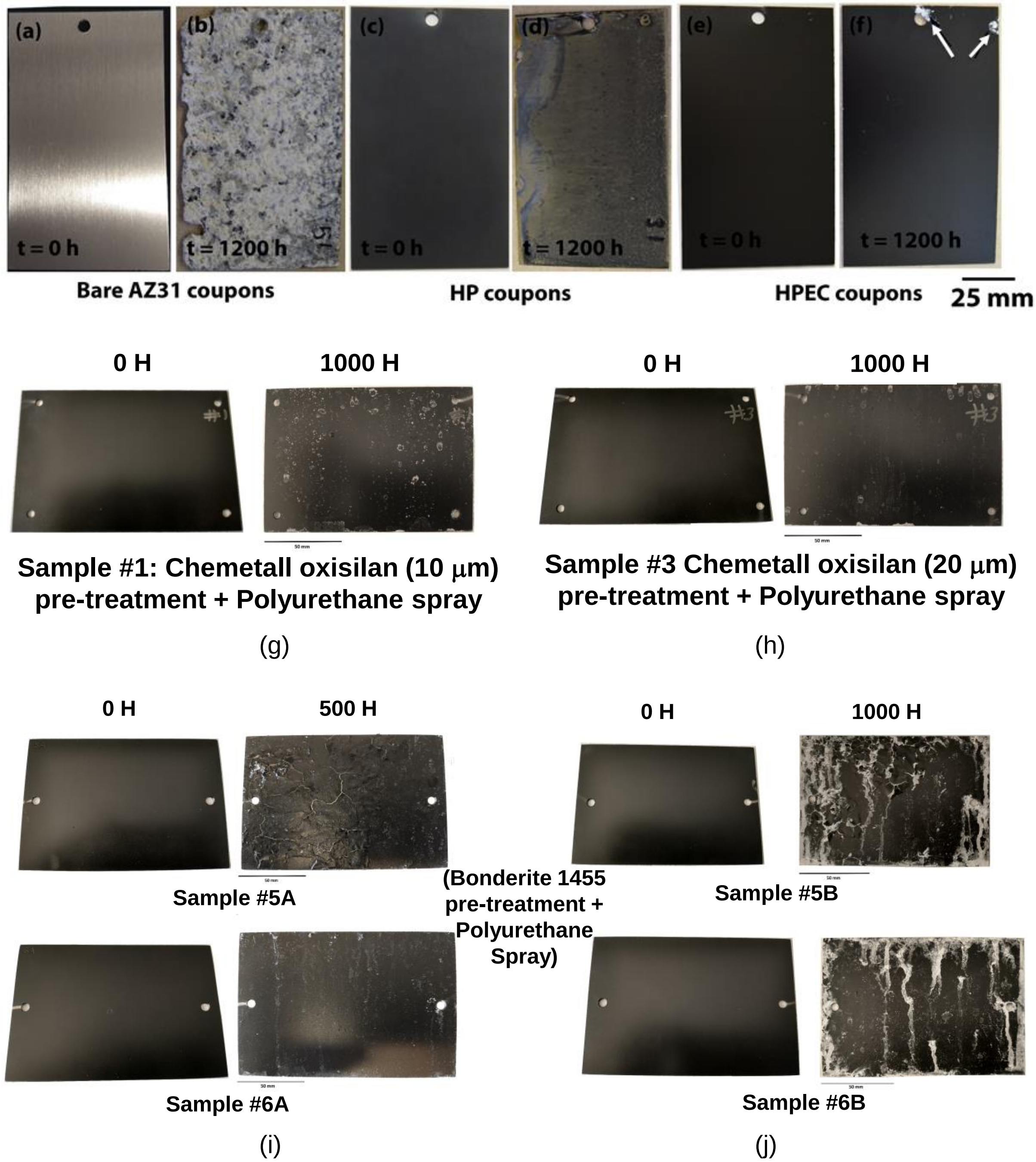



Figure 3: External appearance of the Mg AZ31 corrosion coupons before and after ASTM B117 test. Bare coupon – (a) t = 0 hours, (b) t = 1200 hours; package #A (HP) – (c) t = 0 hours, (d) t = 1200 hours; package #B (HPEC) – (e) t = 0 hours, and (f) t = 1200 hours, arrows indicate localized corrosion product build-up; package #D – (g) t = 0 and 1000 hours; package #E – (i) 2 samples at t = 0 and 500 hours, and (j) 2 samples at t = 0 and 1000 hours.

Coating package #B (HPEC coupons): Among the coupons tested and shown in Fig. 3, the best surface appearance after 1200 hours of corrosion testing is observed in the HPEC (package #B) coupons shown in Figure 3f – HPEC coupons show no apparent signs of general corrosion and resemble untested coupon shown in Figure 3e. Small accumulation of corrosion products was also observed around the punch holes and coupon edges, shown in Figure 3f as indicated by the arrows, and are likely due to the protective coating being damaged during coupon preparation and leading to localized corrosion of the underlying Mg substrate. Finally, no change in weight was observed in the HPEC coupons (Figure 4) suggesting the best corrosion protection among the coupons tested.

Coating package #C (Alodine 5200 + E-coat): This package was not tested on individual coupons.

Coating package #D (Chemetall oxisilan + polyurethane): Figures 3g and 3h show coupons with Chemetall oxisilan pre-treatment coating (10 or 20 $\mu$m, respectively) topped with a polyurethane spray layer instead of a usual E-coating. After 1000 hours of testing, both coupons show a small amount of whitish-color deposit of corrosion products and a corresponding weight gain of 0.05% and 0.3%, respectively.

Coating package #E (Bonderite 1455 + polyurethane): Figures 3i and 3j show coupons with Bonderite 1455 pre-treatment coating topped with a polyurethane spray layer (instead of a usual E-coating). Some corrosion is seen after 500 hours itself and significant corrosion product deposit is seen after 1000 hours of corrosion testing. Sample #5 showed a weight gain of 2.5% and 2.7% after 500 and 1000 hours, respectively. Sample #6 showed a weight gain of 0.3% and 1.3% after 500 and 1000 hours, respectively.

In summary, based on the corrosion tests on individual Mg AZ31 coupons, **the coating package #B (HPEC) shows the best performance** i.e. least corrosion and weight change. **Next lower performance is by coating package #A (HP) and #D (Chemetall**

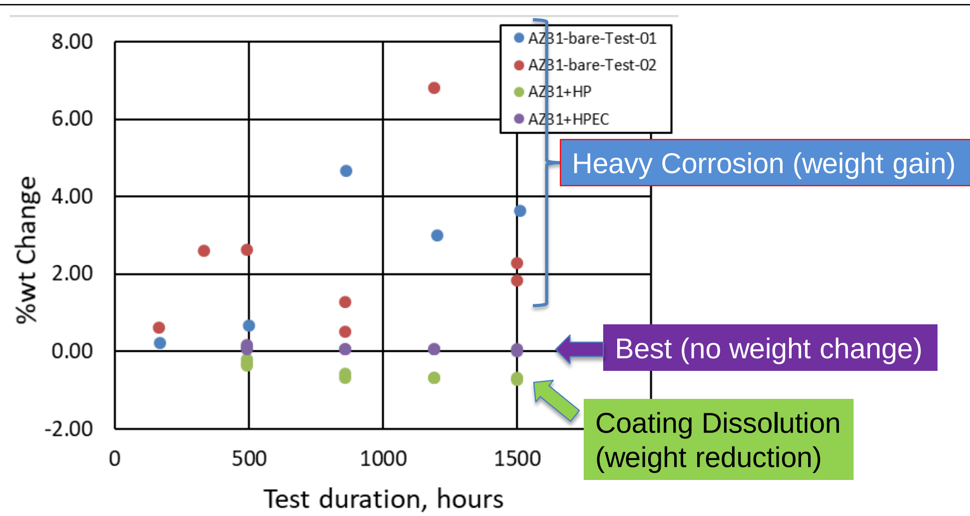



Figure 4: Change in individual AZ31 coupon weight vs. test duration following ASTM B117 testing: (a) bare AZ31; (b) HP (package #A); and (c) HPEC (package #B).

**oxisilan + polyurethane)** as they show small amount of corrosion. **Next lower performance is by the coating package #E (Bonderite 1455 + polyurethane)**.

4.2 Base Material Microstructural Characterization: Figure 5 shows a SEM image of the transverse cross-section of a bare untreated AZ31 coupon after corrosion testing for 500 hours. Build-up of $Mg(OH)_2$ on the coupon surface and inward growth into the AZ31 matrix through pitting is noted in the lower-magnification of Figure 5b. Further, the corrosion film contains several cracks which is evident in the adjoining higher magnification image (see image of section in Figure 5a). At a macro-level, **presence of numerous such cracks provides easy paths for the corrosive media to contact the underlying base metal, and thus, continue the corrosion process and corresponding weight gain** (Fig. 4).

Figure 6 shows the transverse cross-section images of HP (package #A) coupons as a function of test duration for 500 hours in Figure 6a and 850 hours in Figure 6b. The thickness of the HP layer is measured to be ~10 µm. In addition, the pre-treatment layer is noted to contain pores, which might render it prone to corrosion. Formation of $Mg(OH)_2$, the corrosion product, on the underlying AZ31 matrix could be seen at a location where the pre-treatment layer is believed to have washed off during testing, as shown in Figure 6c. Although further degradation of the HP layer is observed after 1500 hours of testing, shown in Figure 6d, **the HP layer seems to be able to protect the underlying metal surface from corrosion**.

Figure 7 shows the transverse cross-section images of HPEC (package #B) coupons as a function of test duration, showing the HP layer in contact with the underlying AZ31 substrate and the E-coat (EC) layer on top of the HP layer. The HP layer is porous and ~10 µm thick, as also seen in the HP coupons in Figure 6, while the overlying EC layer appears to be pore-free and ~15–20 µm thick. Additionally, the presence of numerous bright particles could be seen within the e-coat (EC) layer. The EC protective layer appears to be stable, and no apparent damage was recorded over the course of corrosion testing. However, after 1500 hours of testing, some cracks, as shown by the arrows in Figure 7c, could be observed at the HP/AZ31 interface and may act as a potential failure location of the overall corrosion protection scheme. **In summary, it appears that coating package #B (HPEC) offer better corrosion protection compared to bare or package #A (HP) samples; the top EC layer seems unaffected when exposed to salt-fog environment of ASTM B117 test for 1500 hours**.

4.3 Base Material Mechanical Property Characterization: In some coupons, the mechanical robustness of the coatings was evaluated through the nano-indentation technique. If the coatings have poor interfacial adhesion, the use of indentation point load at the coating/base Mg AZ31 interface is expected to lead to interfacial cracking and/or delamination of the corrosion protection layer. Images of the indents on the HP (package #A) coupons that had been corrosion tested for 500 hours of ASTM B117 tests prior to indentation, are shown in **Error! Reference source not found.**. The indents have been m arked by a dashed line for the purpose of illustration. No interfacial delamination between the HP layer and the base Mg AZ31 sheet could be noted after the indentation. However, a few cracks could be observed when the indent was located completely within the HP layer, as shown in Figure 8b and 8c. In the case of indents made on the HPEC (package #B) coupons, as shown in Figure 9a and 9b after 500 hours of ASTM B117 tests, no cracks could be observed inside the EC layer or along the HP/EC interface. The interface between the HP/EC appears to be mechanically strong and no indentation-induced cracks could be observed in the EC layer.

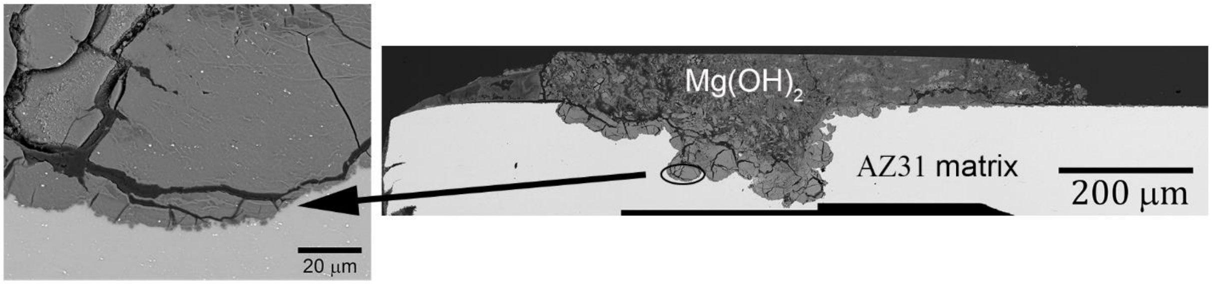



Figure 5: Back-scattered SEM image of (a) the transverse cross-section of a bare individual AZ31 coupon after corrosion testing for t = 500 hours. (b) $Mg(OH)_2$, the corrosion product, is noted to form on the surface, and contains multiple cracks that might be responsible for continuation of the corrosion process.

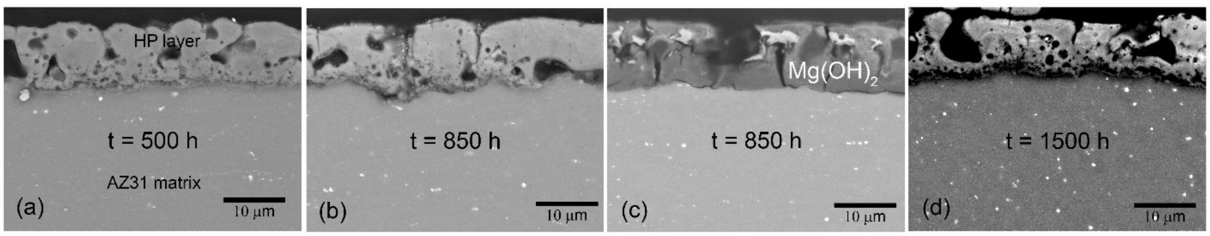



Figure 6: SEM images of the transverse cross-section of AZ31 HP (package #A) coupons: (a) after 500 hours; (b) after 850 hours; (c) after 850 hours, some areas showed build-up of $Mg(OH)_2$ corrosion product; and (d) after 1500 hours.

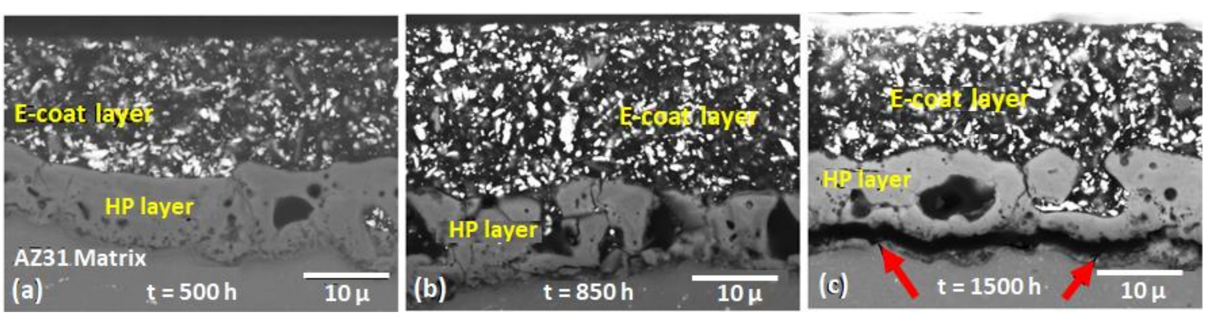



Figure 7: SEM images of the transverse cross-section of individual AZ31 HPEC (package #B) coupons: (a) after 500 hours; (b) after 850 hours; and (c) after 1500 hours. Arrows show cracks at the HP/AZ31 interface.

4.4 Mg/Mg Joint Corrosion Behavior: The joint coupons were placed vertically in the corrosion chamber (Figure 2c) which ensured that the front and back face of each joint was exposed to a similar environment inside the corrosion test chamber and any artifacts associated with joint orientation were avoided.

4.4.1 Arplas RSW (HPEC-package #B) vs. Al rivet (Alodine 5200 + E-coat - package #C)

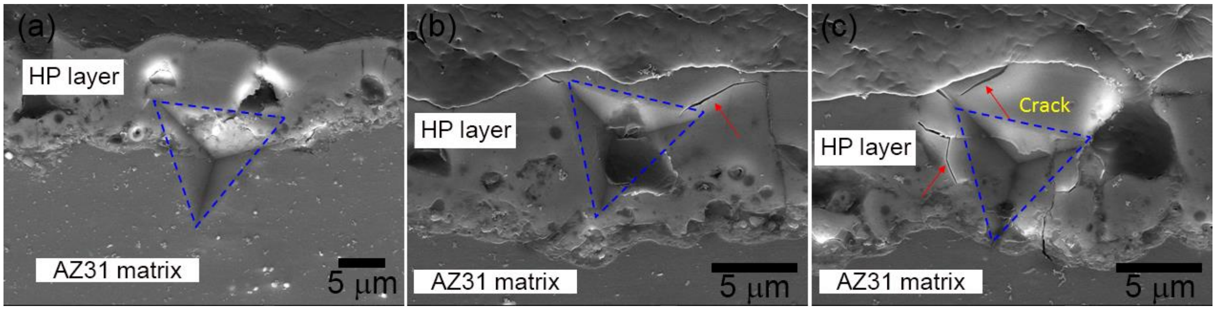



Figure 8: SEM image of the indents in individual AZ31 HP coupons following 500 hours of corrosion testing; (a) indent along HP/AZ31 interface, (b) indent inside the HP-layer, (c) some cracking noted around the indent.

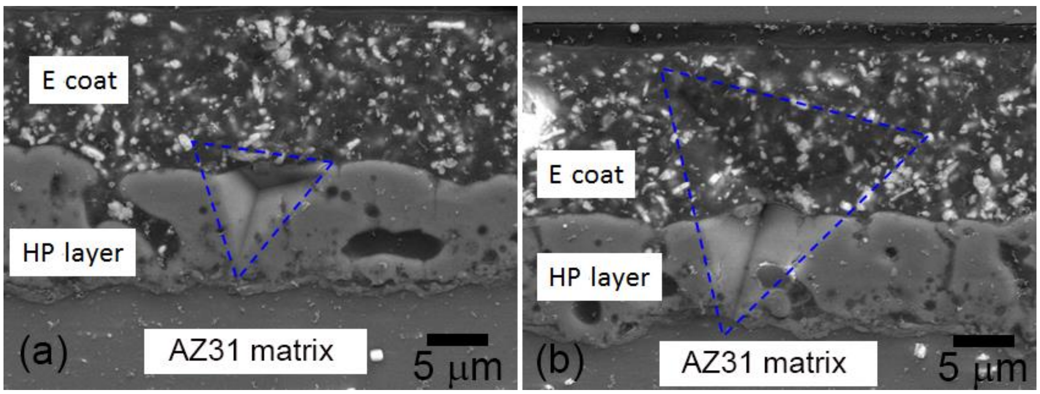



Figure 9: SEM image of the indents in individual AZ31 HPEC coupon following 500 hours of corrosion testing: (a) indent along HP/EC interface; and (b) indent inside EC layer.

Figure 10 shows the condition of joint coupons after 1500 h of continuous salt fog testing. The condition of Mg/Mg Arplas RSW joints is shown in Figures 10a and 10b, capturing the appearance of the joint front and joint back sides, respectively. The appearance of the Mg/Mg rivet joints (coating package #C) after the corrosion tests is shown in Figures 10c and 10d. The images of the respective as-received joint coupons, i.e. without any corrosion testing, are also included in each figure for a relative comparison of the extent of corrosion attack.

The formation of corrosion debris around the resistance spot weld mark could be noticed on the front face of the Arplas RSW joints, which is marked by a yellow circle as shown in Figure 10a. In contrast, the back side of the same Arplas RSW joint does not show any such corrosion product build-up. Unlike Arplas RSW joints, the rivet joints (Figures 10c and 10d show an extensive corrosion attack on the front and back faces of the joints. It was also observed that in most of the rivet joint coupons, a portion of the top E-coat film peeled off during ASTM B117 testing, thus exposing the underlying metal surface to further corrosion attack. The relative change of corrosion coupons as a function of test duration is plotted in Figure 11 and confirms the visual observations above. Figure 11 shows that the rivet joints showed a large weight change (~10%) indicative of significant corrosion whereas the Arplas RSW joints showed only a small weight change (~2%)

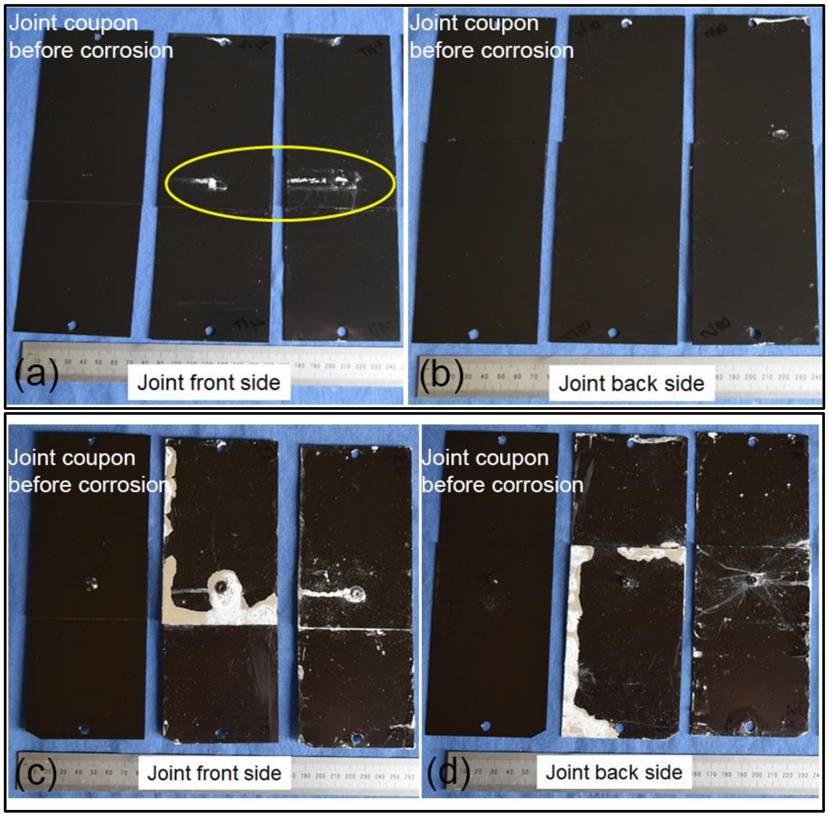



Figure 10: The condition of joint coupons before (one coupon) and after (two coupons) 1500 h of ASTM B117 test. Arplas coupons with coating package #B (HPEC) and riveted coupons with coating package #C. (a) Arplas RSW joint front side; (b) Arplas RSW joint back side; (c) Al rivet joint front side; and (d) Al rivet joint back side.

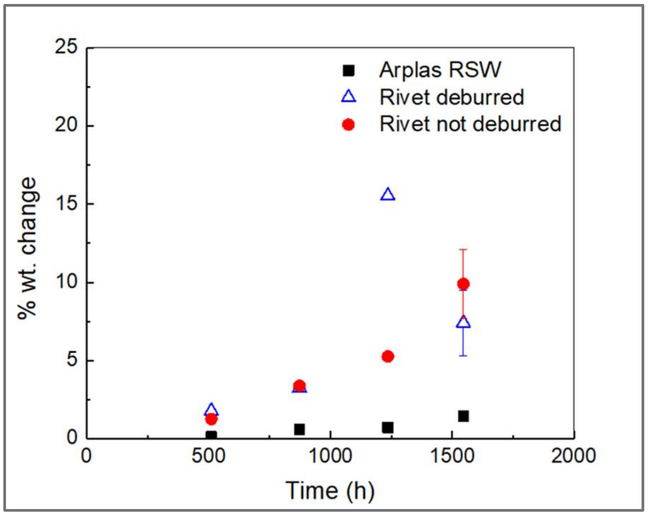



Figure 11: Relative change in weight of joint coupons as a function of time during ASTM B117 corrosion test method. Arplas RSW (HPEC) experiences minimal corrosion attack as compared to Al riveted joints (coating package #C).

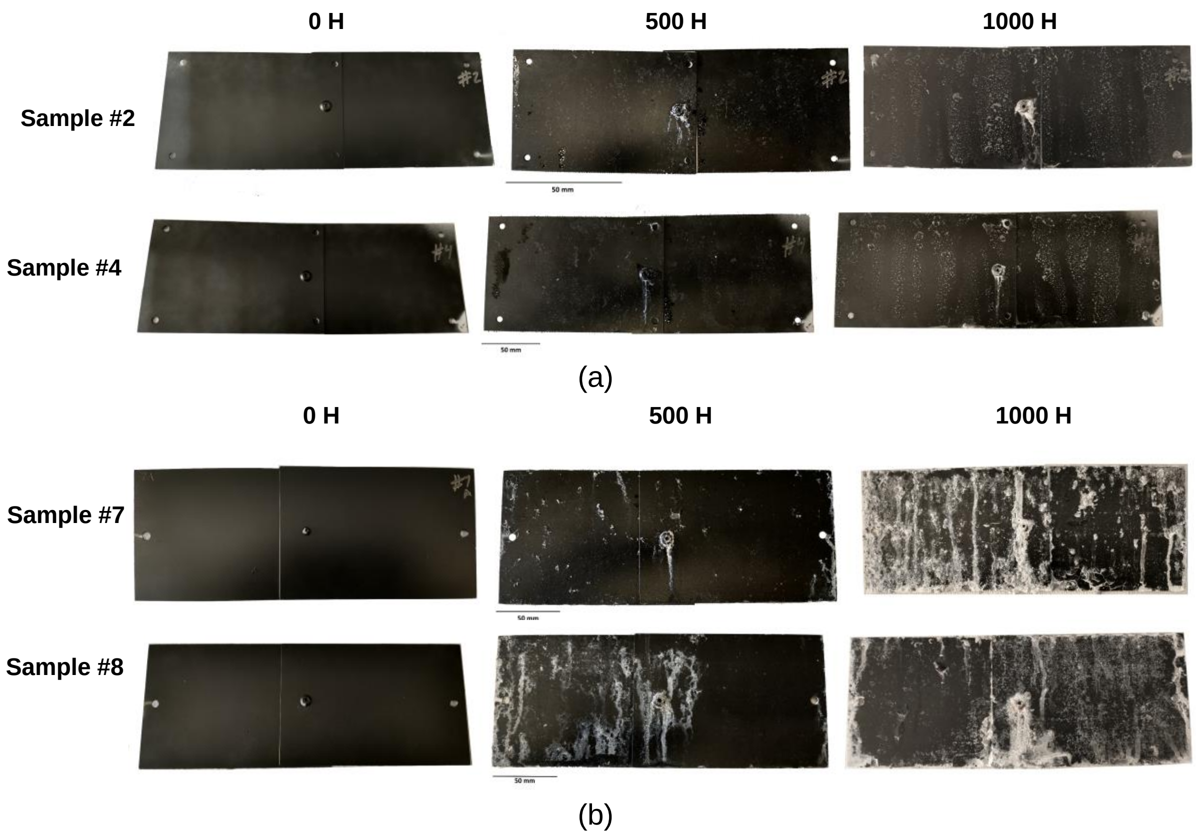



Figure 12: The condition of Mg/Mg Al rivet joint coupons after ASTM B117 test. (a) Coating package #D - Chemetall oxisilan pre-treatment + polyurethane spray, and (b) coating package #E – Henkel Bonderite 1455 pre-treatment + polyurethane spray.

confirming greater resistance to corrosion attack. In summary**, the Mg/Mg similar joints fabricated by the Arplas RSW method with HPEC (coating package #B) showed the least amount of surface corrosion under current test conditions relative to joints fabricated by the all-Al rivet method and coating package #C.**

Visual examination of the tested joint coupons indicates that that the top E-coat does not have the required adherence with the underlying metal whenever the pre-treatment is Alodine 5200-based (coating package #C). It could be the major reason behind higher degree of corrosion attack observed in Mg/Mg rivet joints, since 5200 Alodine pre-treatment was used there. Additionally, signs of corrosion attack could also be seen on the front face of Arplas RSW joints. As mentioned in the experimental procedure section, successful Arplas RSW joint fabrication required removal of the HP layer from the front & back faces of the top sheet of RSW joints. Thus, **the absence of HP layer on the top sheet at the joint location could be the reason behind the small amount of corrosion attack observed in Arplas RSW joints** (Figure 10a).

4.4.2 All-Al rivet joints (coating package #D vs. #E): Figure 12 shows images of corrosion tested (upto 1000 hours) Mg/Mg Al rivet joint coupons prepared from Mg AZ31 sheets with coating packages #D or #E. The images show that after 1000 hours of corrosion testing, joint coupons with coating package #D (Chemetall Oxisilan pre-treatment + polyurethane spray) showed a small amount of corrosion, mainly concentrated around

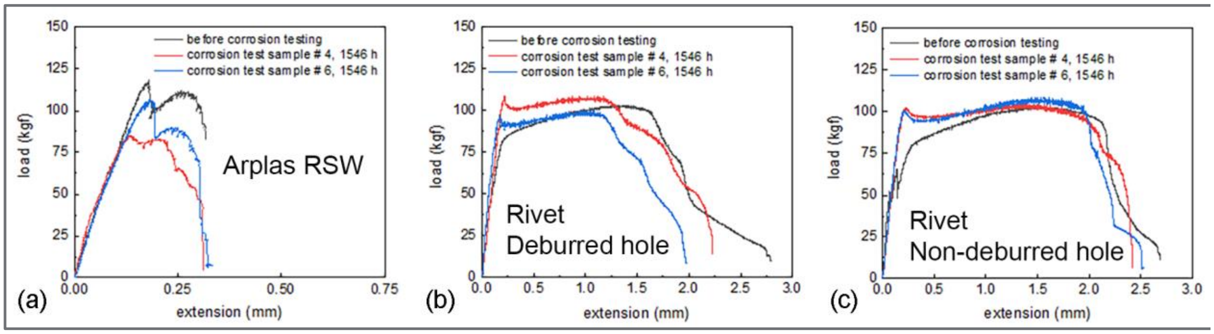



Figure 13 Room temperature lap-shear strength of Mg/Mg similar joints before and after exposure to corrosion test chamber (a) Arplas RSW (HPEC), (b) rivet joint (coating package #C), deburred hole, (c) rivet joint (coating package #C), non-deburred hole.

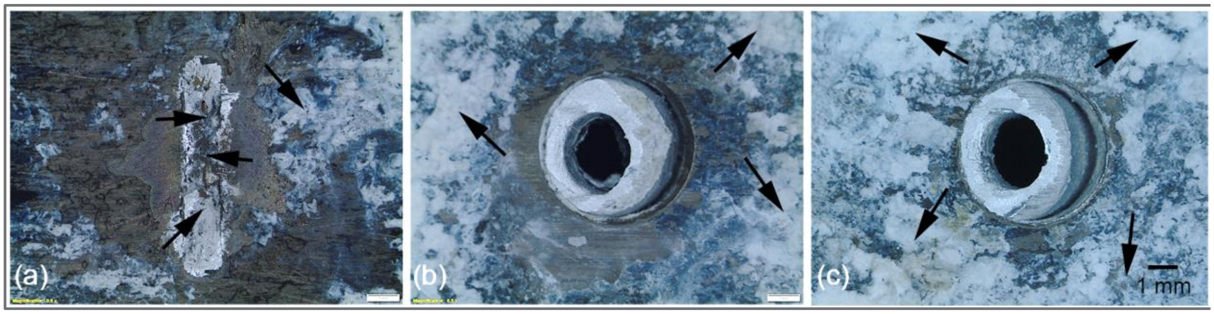



Figure 14: Stereo micrograph of joint interface of corrosion coupons following a 1500 h exposure and subsequent lap-shear test. The occurrence of corrosion is noted by the black arrows for: (a) the Arplas RSW (HPEC); (b) the non-deburred hole rivet (coating package #C); and (c) the deburred hole rivet (coating package #C).

the rivet joint. These coupons showed a weight gain of <0.5% on average. On the other hand, joint coupons with coating package #E (Henkel Bonderite 1455 pre-treatment + polyurethane spray) showed significant corrosion at 500 hours itself. The corrosion worsened after 1000 hours duration and resulted in an average weight gain of 5-10%.

In summary, among the Mg/Mg joints produced by Arplas RSW (coating package #B) and rivet joints (coating packages #C, #D and #E), **the Arplas RSW and rivet (package #D) show the least corrosion and weight change while rivet joint (package #C and #E) show extensive corrosion and weight gain**.

4.5 Mg/Mg Joint Mechanical Property Characterization: Figure 13 shows the lap-shear strength of the Mg/Mg joints after ~1500 h of corrosion test. One joint sample each for the three different joint configurations currently investigated (e.g., Arplas RSW, rivet with deburred hole, rivet with non-deburred hole) were also tested in their as-received condition (i.e., without any exposure to the corrosion test chamber). Based on the limited lap-shear test data in Figure 13, it appears that the Arplas RSW joint has a higher maximum load-bearing capability (~125 kgf) than the rivet joints (~100 kgf), but with a lower ductility in its as-received condition. However, exposure of the Mg/Mg joints to a corrosion chamber environment has a detrimental effect on the Arplas RSW joint strength in comparison to the rivet joints. Maximum load-bearing capability of the two Arplas RSW joints after corrosion chamber exposure are 84 kgf and 106 kgf, respectively, which is lower than in their as-received condition, ~125 kgf, as seen in Figure 13a. In comparison, lap-shear strength of rivet joints (coating package #C) after corrosion testing is ~100 kgf for all of the four samples tested (e.g., two samples with rivet holes being deburred, and two samples with rivet holes being non-deburred), as observed in Figures 13b and 13c, which is the same as the rivet joint strength in the as-received condition.

Since Arplas RSW involved spot welding, the joint interface will feature a solidified microstructure of parent AZ31 alloy. The chances of corrosion attack on a solidified Mg alloy are much higher, especially when the protective Henkel pretreatment coating (HP) had to be removed for a successful welded joint fabrication. **The formation of an inhomogeneous solidification microstructure at the joint interface together with the removal of a protective pretreatment layer could be the major reason behind localized corrosion and associated monotonic strength drop observed in Arplas RSW joints after the corrosion test**. In comparison, rivet joints were fabricated with all-Al rivets. Since Al is more noble than the parent AZ31 matrix, the galvanic corrosion would affect the surrounding AZ31 matrix and leave the Al rivets undamaged. However, the galvanic corrosion is highly influenced by the relative area of cathode (e.g., Al rivet) and anode (AZ31 matrix). The least amount of galvanic corrosion happens with a large anode to cathode area ratio, which is exactly the situation in the case of the rivet joints. As a result, we don't observe any strength reduction in rivet joints after corrosion testing. In summary, **the rivet joints after corrosion testing maintain maximum load-bearing capability at par with its un-corroded counterpart**. However, from the point of view of corrosion extent/weight change (Figure 11) and surface appearance (of the joint) (Figure 10), **the rivet joints (coating package #C) undergo most corrosion and have the worst appearance while the Arplas RSW joints (coating package #B) show least corrosion and better surface appearance**.

4.6 Mg/Mg Joint Microstructural Characterization: The joint interfaces of the corrosion coupons (after ~1500 h test) followed by lap-shear testing were studied using an optical stereo microscope. Lap-shear testing results in the separation of the top and bottom sheets of the lap-joints, and thus enables investigation of the joint interface. Figure 14 is a compilation of low-magnification stereo-micrographs obtained from the mating surface of the top sheet for the three different lap-joints presently studied. The Arplas RSW joint (package #B) interface, after ~1500 h of corrosion testing, is shown in Figure 14a. The resistance spot weld nugget, which shows some signs of corrosion and associated corrosion product build-up, is noticed at the center of the image. The adjoining AZ31 matrix away from the weld nugget shows some signs of corrosion as well (e.g., whitish product build-up, indicated by the arrows). In comparison, much heavier corrosion of the AZ31 matrix material is noted in the case of the rivet joints (package #C), as observed in Figure 14b for the rivet hole non-deburred and Figure 14c for the rivet hole deburred. Failed Al rivets could be seen at the center of both images and do not show any apparent signs of corrosion. **Heavy corrosion of AZ31 matrix surrounding the Al rivets is due to galvanic coupling between Mg sheet and Al rivet**. Since AZ31 is electrochemically more active than Al, it suffers galvanic corrosion when Al rivets are used. Moreover, the lap-joint configuration itself leads to a crevice corrosion-type of attack. In summary, the microstructural observation indicates the corrosion attack mostly happening at the joint interface.

## 5 Conclusions

Base AZ31 Mg sheet coupons were fabricated in uncoated and with coating packages #A, #B, #D and #E. Two different joining techniques were evaluated for Mg/Mg joint coupons: Arplas RSW (coating package #B) and Al rivet (coating packages #C, #D and #E). Based on the corrosion test data of the individual and joint coupons (ASTM B117 salt-fog test) for up to 1500 hours, the following conclusions were reached:

1. The coating package #B (HPEC) shows the best performance i.e. least corrosion and weight change - the topmost layer (E-coat) is pore-free, shows almost no degradation after corrosion testing, and protects the underlying porous pre-treat layer. Next lower corrosion performance is by coating package #A (HP) and #D (Chemetall oxisilan + polyurethane) as they show small amount of corrosion. Next lower performance is by the coating package #E (Bonderite 1455 + polyurethane). The worst situation is bare untreated Mg AZ31 that undergoes severe corrosion.

2. Among the Mg/Mg joints produced by Arplas RSW (coating package #B) and rivet joints (coating packages #C, #D and #E), the Arplas RSW and rivet (package #D) show the least corrosion and weight change while rivet joint (package #C and #E) show extensive corrosion and weight gain.

3. Although Mg/Mg sheets joined by Arplas RSW method (coating package #B) show greater corrosion resistance and better surface appearance as compared to the Al rivet joints (coating package #C), the Arplas RSW joints are weaker after corrosion testing due to corrosion of the weld nugget. By contrast, Mg/Mg rivet joint strength is unaffected after corrosion testing since the Al rivets are more noble than the adjoining AZ31 matrix, and therefore, do not experience any galvanic corrosion.

4. Arplas RSW of Mg sheets (coating package #B) requires removal of the underlying Henkel pre-treatment layer from both sides of the front sheet and the mating surface of the bottom sheet. Removal of this pre-treatment layer can result in some corrosion and weakening of the joint.

5. There is no apparent effect of rivet holes being deburred vs. non-deburred on the corrosion performance of Mg/Mg rivet joints.

## 6 Publications and Presentations

1. U.S. Department of Energy – Office of Energy Efficiency and Renewable Energy, Vehicle Technologies Office, Materials FY 2018 Annual Progress Report.
2. U.S. Department of Energy – Office of Energy Efficiency and Renewable Energy, Vehicle Technologies Office, Materials FY 2019 Annual Progress Report.
3. Presentation at the U.S. Department of Energy Vehicle Technologies Office 2018 Annual Merit Review and Peer Evaluation Meeting, Project ID# mat143, June 18-21, 2018, Washington, D.C.
4. Presentation at the U.S. Department of Energy Vehicle Technologies Office 2019 Annual Merit Review and Peer Evaluation Meeting, Project ID# mat143, June 10-13, 2019, Washington, D.C.

## 7 References

1. U.S. Department of Energy, National Energy Technology Laboratory, 2015, "Demonstration Project for a Multi-Material Lightweight Prototype Vehicle as Part of the Clean Energy Dialogue with Canada," DOE Award # DE-EE0005574. https://www.osti.gov/servlets/purl/1332277.

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