Marine atmospheric corrosion of Al-Mg joints by friction stir blind riveting

Shengxi Li, Haris Khan, Lloyd H. Hihara*, Jingjing Li*

Department of Mechanical Engineering, University of Hawaii at Manoa, Honolulu, Hawaii
96822, USA

*Corresponding author: Tel.: +1 808 956 3679; Fax: +1 808 956 2373.

E-mail: hihara@hawaii.edu (L.H.Hihara), lj8@hawaii.edu (J.J.Li),

Abstract

This paper investigated the atmospheric corrosion behavior of Al-Mg friction stir blind riveting joints exposed at a severe marine test environment. Besides the severe corrosion that occurred on Mg alloy AZ31B-H23, Al alloy 5754-O also corroded in the coupled region because of the alkaline condition generated by the cathodic reaction inside of the crevice. The highly alkaline environment, however, passivated Mg deep inside the crevice. The corrosion products on Mg were identified as magnesium carbonates and that on Al was mainly gibbsite, which was an unusual corrosion product of Al exposed to atmospheric conditions.

Keywords: A. Aluminum alloy; A. Magnesium alloy; B. IR spectroscopy; B. XRD; C. Alkaline corrosion; C. Crevice corrosion

1. Introduction

The joining of dissimilar light metals is of increasing demand in the automotive industry for weight reduction and tailored properties. However, the joining of dissimilar metals was limited by traditional fusion welding method which cannot be applied in welding of different metals

having vast discrepancy in melting temperatures and may generate brittle intermetallics [1, 2]. Friction stir blind riveting (FSBR) is a new mechanical dissimilar material joining method developed by General Motors in 2011 [3], where work materials are softened by the friction stir process to facilitate rivet penetration. FSBR has been successfully applied to the joining of various similar and dissimilar metallic materials, such as Al-Al [4-8], Mg-Al [5], Steel-Mg [9, 10], Al-CFRP (carbon fiber reinforced plastics) [11], and CFRP-CFRP [12]. These emerging research activities have suggested that FSBR is suitable for joining various metallic and nonmetallic materials that result in joints having superior mechanical properties.

In addition to FSBR, other mechanical riveting methods, such as self-piercing riveting [13], friction self-piercing riveting [14, 15], and friction stir riveting [16, 17], have also been investigated for joining dissimilar materials. Most of the research, however, focused on process optimization and mechanical behavior of the joints. Very limited research has been reported on the corrosion behavior of these mechanical joints, which is a potential application issue for dissimilar-material joints because of the possibility of galvanic corrosion and crevice corrosion at the joint interface. The galvanic corrosion between the dissimilar material couples have been widely studied in saline environments [18-24]. In these couples, the less noble material usually experiences severe corrosion and the more noble material is protected. However, it was found in the present work that the galvanic couple between Al and Mg behaves differently from conventional understandings when a crevice forms between the two materials.

The Mg-Al dissimilar joints were selected in the present work because of great interests in the automobile industry. While Al alloys have found a wide range of applications as lightweight and high-strength structural materials, Mg alloys are also gaining interests as they are considered as the lightest structural metallic materials (approximately 34% lighter than Al) and provide an

attractive high strength to weight ratio for automotive applications. However, Mg alloys can be used in a few automotive parts [25] but not in others because of the limits in their mechanical properties, which therefore, requires the joining of Mg with other structural materials, such as Al. Besides the specific choice of the materials, the selection of a severe marine environment for exposure tests was due to the large presence of airborne salt particles in such environment [26, 27] and their great influence on the corrosion behavior of metallic materials [28-32].

The present study aims to 1) identify the various types of corrosion products that formed in the Al-Mg FSBR joint exposed in a marine environment, and 2) elucidate the corrosion mechanism through the characterization of corrosion products. The results will enhance the understanding of corrosion behavior not only in FSBR joints but also in other mechanical joints constituted with dissimilar metals.

2. Experimental

2.1 Materials and FSBR joining

The raw materials used were Mg AZ31B-H23 and Al 5754-O with chemical compositions shown in Table 1. Fig. 1 illustrates the FSBR process for joining the Mg and Al sheets. The Al-Mg FSBR joints were formed on a computer numerical control (CNC) machining center (model: Bridgeport Discovery 308) using 1 mm thick Mg sheets and 3 mm Al sheets. Each work piece was 38 mm wide and 127 mm long. The overlap region had a size of 38 mm × 38 mm. The blind rivet with a diameter of 6.4 mm was made of mild steel with zinc coating. The spindle speed was 5000 rpm and the feed rate was 120 mm/min.

2.2 Outdoor exposure

The Al-Mg FSBR joints (**in triplicate**) including the rivets were exposed to a severe marine test site located in the Marine Corps Base Hawaii (MCBH) in Kaneohe, Hawaii. This test site is

maintained by the Hawaii Corrosion Laboratory and is considered a severe marine site with regards to corrosion rates. The test site is located within 40 meters of a shoreline with waves breaking consistently throughout the year. The average temperature and relative humidity are 23.6 °C and 77%, respectively. The average chloride (Cl⁻) deposition rate was 2900 mg/m²/day and the sulfate (SO₄²⁻) deposition rate was 390 mg/m²/day, which were determined using the conventional dry chloride candle method. The corroded joints were retrieved to the laboratory after 6-month exposure for characterization. All three joints showed similar corrosion characteristics, however, only one was characterized.

2.3 Corroded sample characterization

The corrosion products inside the crevice were revealed by pulling the joint to fracture using a tensile machine. The surfaces of the corroded Mg and Al sheets were first characterized using a JEOL JXA-8500F field emission scanning electron microscope (FE-SEM) with energy dispersive spectroscopy (EDS) capability. Then, corrosion products were scraped from the Al and Mg sheets and ground into fine powders using an agate pestle and mortar. The powder samples were characterized with Fourier transform infrared spectroscopy (FTIR, Jasco FT/IR 4100) and powder X-ray diffraction (XRD, Rigaku MiniFlex™ II) to identify the compositions of the corrosion products.

The corroded Al sheet was also chemically cleaned using the procedures and solutions described in ISO 8407: 2009 [33]. Three pristine Al sheets were also cleaned together with the corroded sample. Multiple cleaning cycles were employed until the mass loss of the corroded sample between cleaning cycles was equal to, or less than, the average mass loss of the pristine samples. The cleaned sample was then characterized using the same FE-SEM.

2.4 Microstructural characterization

The Mg and Al sheets that were in contact with the rivet were sectioned from the Al-Mg FSBR joint using a low-speed diamond saw for microstructural characterization. The sectioned samples were mounted in epoxy resin (BUEHLER Epoxicure); ground with 600 and 1200 grit SiC grinding paper; and polished with 9.0, 3.0, and 1.0 μm polycrystalline diamond suspensions (METADI SUPREME, BUEHLER). The Mg sample was polished with isopropanol as a lubricant. After polishing, the samples were ultrasonically cleaned in isopropanol for 5 min and dried under cold air.

The polished samples, especially the regions that were in close contact with the rivet, were first characterized using FE-SEM. Then, the samples were etched to reveal grain structures. The Mg sample was etched using an Acetic Picral reagent [34, 35] to reveal grain boundaries. The grain boundaries of the Al sample were revealed using the Baker's etching method [36, 37]. The Al sample was oxidized in a solution consisting of 5 ml HBF_4 (48%, Sigma-Aldrich) in 200 ml water at a current of 0.2 A/cm^2 for 40 – 80 seconds. Then, the grain structure of the etched/oxidized samples were observed under an optical microscope (AxioPlan, Zeiss). Notice that the grain structure of the oxidized Al sample was observed with polarized light plus a sensitive tint filter.

3. Results and discussion

3.1 Microstructure of the jointed materials

Fig. 2 shows the microstructures of the Al and Mg sheets in regions that are in close contact with the galvanized steel rivet. Many strips of white particles were found on both Al (Fig. 2b and c) and Mg (Fig. 2d and e). EDS elemental analysis of these white particles confirms that Zn was infused into both Al and Mg after the FSBR process (Table 2). The widths of the Zn-contaminated regions are in a range of approximately 50 – 150 μm on both Al and Mg.

Fig. 3 shows the grain structures of Mg and Al in the FSBR-affected regions (b and d) as compared to those of the base materials (a and c). It can be seen that the FSBR process refined the grains in both Mg and Al near the rivet owing to the coupled thermomechanical effect generated from the frictional stir penetration process [38]. The widths of grain-refined regions in Mg and Al are approximately 100 – 150 μm and 50 – 80 μm , respectively. Notice that the etching of Al in the refined region (Fig. 3d) did not result in well-defined grains due to the corrosion of Zn particles (that were infused into Al during FSBR) in the acidic etching solution.

3.2 Corrosion characteristics and morphology

Fig. 4a shows the images of the coupled sides of Mg and Al sheets after being separated by tensile test. Severe corrosion occurred in most regions on Mg (e.g., location 1 in Fig. 4a), but not in regions surrounding the rivet (e.g., location 2 in Fig. 4a) within the crevice of the joint. SEM images (Fig. 4b1 and b2) from the corroded regions illustrate typical porous morphology of Mg corrosion products [39], and those from the uncorroded regions (Fig. 4c1 and c2) even showed some original scratches from material fabrication or handling. On Al, significant corrosion did not occur on the regions outside of the joints (Fig. 4e). However, the central region of the coupled area (within the dotted line on Al in Fig. 4a) was covered by a thick layer of yellowish-brown corrosion products, which looked distinct from the white corrosion products on Mg. Notice that the yellowish-brown corrosion products on Mg (marked by thick solid arrows in Fig. 4a) was actually from Al (marked by thick open arrows in Fig. 4a). An SEM image (Fig. 4d) of the yellowish-brown corrosion product further indicated that it was visually different from the porous corrosion products on Mg (Fig. 4b2), possibly indicating that it came from the corrosion of Al.

Table 3 summaries the EDS results from different locations (1-4) in Fig. 4a. Location 1 (Table 3) has a very high content of O, suggesting that Mg was significantly corroded in uncoupled

regions. As compared to location 1 in Fig. 4a, location 2 has a much lower O content and a much higher Mg content (Table 3), implying the possible absence of corrosion on the coupled side of Mg in regions near the rivet. The O in uncorroded Mg is likely from the surface oxide films. The high contents of Al and O at location 3 (Table 3) indicate the occurrence of corrosion on the coupled side of Al in regions close to the rivet. However, the uncoupled Al (e.g., location 4 in Fig. 4a) shows much less O but more Al (Table 3) as compared to Al in the center of the coupled area. Therefore, the uncoupled Al experienced only slight corrosion.

After collecting the yellowish-brown corrosion products on Al, the rivet was removed by cutting it apart together with the Al sheet using a low speed diamond saw (black dashed line in Fig. 4a). The sectioned aluminum sheet was chemically cleaned according to ISO 8407: 2009 [33]. The cleaned sample (Fig. 5a) clearly reveals that severe corrosion occurred on Al in the center of the coupled region (inside the dotted line) but not on the edge (outside of the dotted line). Both visual observation (Fig. 5a) and low-magnification SEM observation (Fig. 5b1) suggest that corrosion was generally uniform on a macro scale with no large corrosion pits (i.e., pits larger than 1 mm). A high-magnification SEM image (Fig. 5b2) from the corroded region shows only micron-sized corrosion pits. The micron-sized corrosion pits are also seen in the SEM images (Fig. 5c1 and c2) from the intermediate region (along the dotted line in Fig. 2a) between the corroded and uncorroded regions. Some scattered regions with insignificant corrosion (remnants of the original Al surface, marked by arrows in Fig. 5c2) are also present in the intermediate region. The SEM images (Fig. 5d1 and d2) from the edge of the coupled region on Al show that most of the Al surface is pristine and only mild corrosion occurred with the formation of micron-sized pits.

3.3 Corrosion product characterization

Fig. 6a presents a typical FTIR spectrum obtained from the corrosion products formed on Mg, either in the coupled region or the uncoupled area because they have the same spectrum. The peaks at 716, 745, 796, 854, 882, 1113, 1426, and 1479 cm^{-1} can be assigned to hydromagnesite ($\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$), which has been identified as the corrosion products on Mg after atmospheric corrosion [40, 41]. However, it was reported that dypingite ($\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 5\text{H}_2\text{O}$) also has similar peaks as those shown in Fig. 6a [42]. Therefore, hydromagnesite and dypingite could coexist in the corrosion products on Mg, which is corroborated by XRD analysis in the following section. The peak at 876 cm^{-1} and a possible hidden peak at approximately 1447 cm^{-1} indicated the presence of magnesite (MgCO_3) [40]. Notice that a prominent peak for brucite ($\text{Mg}(\text{OH})_2$) at 3699 cm^{-1} [40] is absent from Fig. 6a, which suggests that the corrosion products on Mg exposed to a marine environment are mainly carbonate compounds. The yellowish-brown corrosion products formed on Al in the central coupled region (close to the rivet) showed a spectrum with major peaks at 663, 747, 800, 914, 970, 1022, and 3444 cm^{-1} (Fig. 6b), which match well with those of gibbsite ($\gamma\text{-Al}(\text{OH})_3$) [43, 44]. Therefore, FTIR analysis demonstrated that the corrosion products on Al are mainly due to the corrosion of Al, corroborating the EDS results in the above section.

Fig. 7a and b presents the XRD patterns of the corrosion products formed on Mg and Al in the Al-Mg FSB joint, respectively. Most of the XRD peaks in Fig. 7a can be assigned to either hydromagnesite (PDF Card No.: 00-025-0513) or dypingite (PDF Card No.: 00-023-1218) [39, 45-48]. The peak at 8.16° corroborates the existence of dypingite since hydromagnesite does not have such a peak. The majority of the XRD peaks in Fig. 7b, especially those with strong intensities, are caused by gibbsite (PDF Card No.: 00-012-0460) [49-51], indicating gibbsite as the major component of the corrosion products on Al. The peak at approximately 18.66° might have

originated from the other two polymorphs of $\text{Al}(\text{OH})_3$, bayerite (PDF Card No.: 01-077-0250) and nordstrandite (PDF Card No.: 00-018-0050) [52], the concentration of which were lower than that of gibbsite. The two weak peaks at 11.64° and 23.4° are not identified. Notice that the XRD peaks in Fig. 7b have much stronger intensities than those in Fig. 7a, which implies that the corrosion product on Al has a higher degree of crystallinity than that on Mg. Generally, the identification of gibbsite on Al and magnesium carbonates on Mg using XRD agrees well with previous FTIR results.

3.4 Corrosion mechanism

When Mg is coupled to Al, galvanic corrosion occurs on Mg because Mg is less noble as compared to Al. In this case, Al serves as the cathode at which cathodic reactions occur. Due to the highly confined environment inside the crevice, O_2 should not be readily accessible to the crevice. Therefore, it is reasonable to assume that the cathodic reactions are H_2 evolution (Eq. 1) inside of the crevice and oxygen reduction (Eq. 2) just outside of the crevice (Fig. 8a).

The accumulation of OH^- ions increases the local pH because of the cathodic reaction inside the crevice. Since Al corrodes in the environment with a pH higher than approximately 8.5 [53], the increase of pH in the crevice resulted in the corrosion of Al (Fig. 8b). It has been reported that the corrosion product of Al in a pH higher than 9 is gibbsite (Fig. 8c) [54], which is corroborated in the present study by FTIR and XRD analyses. Notice that gibbsite is not usually found as the corrosion product of Al exposed to various atmospheric conditions [55-58]. The reason for the formation of gibbsite in the present study was the high pH environment in the confines of the crevice. After the dissolution of Al initiated inside the crevice, the major cathodic reaction was oxygen reduction occurring on Al in the uncoupled regions.

The increase of pH inside the crevice, if up to approximately 11.4 will passivate Mg [53]. Since it was observed that corrosion did not occur on Mg deep inside the crevice or in the thinnest crevice (Fig. 4c1 and c2), it is believed that the local pH rose to 11.4. The Pourbaix diagram of Mg [53] also shows that Mg would spontaneously passivate at potentials greater than the Mg equilibrium potential, indicating that Mg could therefore passivate in the crevice where the corrosion potential would be more positive than the Mg equilibrium potential due to polarization of hydrogen evolution. The extremely high pH in the crevice was likely caused by the generation of a large amount of OH⁻ ions in a highly confined space. In addition, the excess OH⁻ ions electrostatically repelled other anions (i.e., Cl⁻) from the crevice to maintain charge neutrality, which also enhances passivation of Mg. The EDS results (Table 3) showed that Cl was undetectable on Mg in the crevice (location 2) but present on Mg outside of the crevice (location 1). Similar behavior has been observed for Mg-SiC couples in chloride-containing environments where SEM and EDS analysis showed minimal corrosion and low Cl⁻-concentration within the crevice, and significant corrosion and high Cl⁻-concentration outside of the crevice [59].

The initial corrosion product formed on Mg is believed to be brucite (Mg(OH)₂) (Fig. 8b) in an environment where NaCl and high humidity are present [60, 61]. Brucite will then react with CO₂ from the atmosphere, forming magnesium carbonates hydrates (Fig. 8c), which are the primary corrosion product of Mg exposed to atmospheric corrosion conditions [47, 60, 61].

It is worth to mention that the effects of microstructural changes in Mg and Al (caused by the FSBF process) on the crevice corrosion behavior can be excluded, because it is believed that the regions with microstructural alterations were not exposed to electrolyte. These regions have a maximum width of approximately 150 μm, as shown in Figs. 2 and 3. However, the deformation zone (marked by a dashed rectangle in Fig. 9a), which prevents the electrolyte inside the crevice

from reaching the rivet/Mg and rivet/Al interfaces, has a width of approximately 0.5 – 1 mm. In addition, the close contact between the rivet and the Mg and Al sheets (marked by dashed arrows in Fig. 9b) hinders the electrolyte access to the rivet hole. As the corrosion of Al initiates inside the crevice, the Al/electrolyte interface (region 3 in Fig. 9b) will move towards the rivet hole. However, the formation of corrosion product (i.e., $\text{Al}(\text{OH})_3$) inside the crevice (Fig. 9c) would have quickly clogged the tip of the crevice and thus stopped the movement of electrolyte towards the rivet hole. Notice that bulk electrolyte was not usually present on the samples exposed in atmospheric conditions (except during rain). However, we believe that only a small amount of water is needed to fill the crevice and that amount of water is readily available in the severe marine test site where the Al-Mg couples were exposed. In addition, it is believed that the crevice areas can retain moisture much longer than open surfaces since it takes longer for moisture to evaporate from the crevice [62]. In an environment with relative humidity values in the range of approximately 67% to 94%, any region that is exposed to sea salt will be wet (or with the presence of electrolyte) since the critical relative humidities for NaCl and MgCl_2 are ~75% and ~33%, respectively, and both species are present in seawater.

Finally, it is believed that the corrosion caused by the Zn-coated carbon steel rivet also does not affect the crevice corrosion behavior discussed above. The first type of rivet-related corrosion is the self-corrosion of the rivet (regions 1 and 4 in Fig. 9b) due to low corrosion resistivity of galvanized steel in severe marine environment. The second type of rivet-related corrosion is galvanic corrosion, which accelerates the corrosion of Mg near the rivet/Mg interface (region 2 in Fig. 9b) as well as the corrosion of the rivet close to the rivet/Al interface (region 4 in Fig. 9b). However, all these types of corrosion do not alter the crevice corrosion behavior because they occur separately from the corrosion inside the crevice.

The present study implies that when Al is coupled to Mg using FSBR, not only has Mg to be protected, but also Al especially when a crevice forms in between the coupled materials. This implication also applies to other cases when Al is mechanically coupled to less noble metallic materials and crevices form in between them.

4. Conclusions

1. Outdoor exposure tests showed that when Al was coupled to Mg using FSBR, both crevice and external corrosion (outside of the crevice) occurred. Within the crevice, Mg passivated and significant corrosion was observed on Al. A high pH environment was created within the crevice due to the hydrogen-evolution cathodic reaction that generates OH^- . The excess OH^- also repelled Cl^- from the crevice and enhanced the passivation of Mg. Since aluminum oxide is amphoteric, Al lost passivation and corroded in the high pH environment of the crevice. On the outside of the crevice, corrosion on Al was minimal and corrosion on Mg was substantial.
2. A combination of FTIR and XRD facilitated the identification of the corrosion products on Mg as hydromagnesite, dypingite, and magnesite, which are common corrosion products formed on Mg exposed to atmospheric conditions. The abnormal identification of gibbsite as the corrosion product on Al exposed to marine atmospheric conditions is caused by the high pH environment inside the crevice formed during FSBR process.
3. The findings add new knowledge to the crevice corrosion between mechanically jointed Al and Mg sheets.

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426

427 **Tables:**

428 Table 1

429 Chemical compositions (wt.%) of the raw materials.

Material	Si	Fe	Mn	Zn	Cu	Ti	Cr	Ni	Al	Mg
5754-O	0.4	0.4	0.5	0.2	0.1	0.15	0.03	-	balance	2.6
AZ31B-H23	0.009	0.004	0.28	0.9	-	-	-	0.0008	2.95	balance

430

431 Table 2.

432 Elemental compositions (at.%) of locations 1 and 2 in Fig. 2.

Locations	Zn	Mg	Al
1 (on Mg)	34.62	60.13	5.25
2 (on Al)	28.15	1.65	70.2

433

434 Table 3

435 Elemental compositions (at.%) of locations 1 – 4 in Fig. 4a.

Locations	C	O	Na	Mg	Al	S	Cl	Zn
1: Mg, uncoupled	17.20	61.44	0.85	15.69	2.44	0.19	1.81	0.39
2: Mg, coupled	3.73	8.54	-	85.64	1.69	-	-	0.31
3: Al, coupled	4.57	73.07	1.13	3.23	17.99	-	-	-
4: Al, uncoupled	6.54	13.49	-	2.90	74.04	-	-	-

436

Figure captions:

Fig. 1. Illustration of the FSBR process [7]: (a) rotating blind rivet approaching the workpieces; (b) frictional penetration of the rivet; (c) pulling out the mandrel; and (d) completion.

Fig. 2. Microstructures of the Mg and Al sheets adjacent to the rivet. Intermetallic particles are marked by arrows. Unmarked black scale bars are 50 μm and white scale bars are 2 μm .

Fig. 3. Microstructure comparisons between the base and after FSBR metals: (a and c) Grain structures of Mg and Al base materials; (b and d) grain structures of Mg and Al in the FSBR-affected regions. Black scale bars are 10 μm and white scale bars are 50 μm . The center embedded figure indicates the observation locations of (a-d).

Fig. 4. (a) Images of the Al and Mg sheets separated from the Al-Mg FSBR joints by tensile test, showing the growth of corrosion products on both sheets after 6-month exposure in a marine environment; (b1, b2, c1, c2, d, and e) SEM images from different locations (1-4) in (a). The dashed line indicates where the sample together with the rivet was sectioned. Black scale bars are 100 μm and white scale bars are 10 μm .

Fig. 5. (a) An image of the Al sheet after chemical clearing; (b1, b2, c1, c2, d1, and d2) SEM images from different locations (1-3) in (a). Black scale bars are 100 μm and white scale bars are 10 μm .

Fig. 6. FTIR spectra obtained from the corrosion products (a) on Mg and (b) on Al in the Al-Mg BSBR joint.

461 Fig. 7. XRD patterns of powder samples made from (a) corrosion product on Mg and (b) corrosion
462 product on Al in the coupled region.

463

464 Fig. 8. Schematic diagrams of the corrosion process inside the crevice between Al and Mg coupled
465 by FSBR.

466

467 Fig. 9. Schematic diagrams showing the various types of corrosion in the Al-Mg FSBR joints.

Figure1

Figure2

Figure3

Figure4

Figure5

Figure6

Figure7

Figure8

Figure9