Root cause analysis of pitting corrosion of AA2024 and AA7075 after exposure in salt fog environment

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Abstract: Two grades of anodised aerospace aluminium alloys, AA2024 and AA7075, were analysed to determine root causes of pitting corrosion after the samples were exposed to a salt spray test according to ASTM B177. Methods to investigate pitting corrosion behaviour of the aluminium alloys were divided into two steps: 1) characterisation of pre-exposed (intact) anodised film and stimulated pitting corrosion by polarisation; 2) comparison of samples that passed and failed the salt spray test. The surface properties were characterised by roughness measurements, scanning electron microscope (SEM) and energy dispersion X-ray spectroscopy (EDS). Pitting corrosion was stimulated by potentiodynamic polarisation prior to characterisation of pitting by SEM and EDS to determine pitting initiation point. The results reveal that thickness of the film does not prevent localised corrosion. Cu-bearing intermetallic particles are detrimental to anodic film formation and cause pitting in the film. Potentiodynamic polarisation tests alone cannot determine whether the film will pass or fail the salt spray test for 96 hours.

Keywords: anodised film; pitting corrosion; aluminium alloys; salt spray.


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1 Introduction

Aluminium alloys (AA) possess high strength to weight ratio due to alloying elements. In aerospace applications, AA, especially 2xxx and 7xxx series, are used in many parts because of their strength. Pure aluminium has excellent uniform corrosion protection property because of its natural passive oxide film. Its passive film protects AA from environment. However, when ubiquitous chloride ions are present, they destroy the passive films and cause localised corrosion (Frankel, 1981). Alloying elements are added to improve mechanical properties of the materials. However, the alloying elements are the weakest point for corrosion attack (Szklarska-Smialowska, 1999). When passive films are exposed to chloride environment, damages start at defect sites. Corrosion of AA, especially 2xxx series, are mostly initiated at intermetallic particles. Originated by inhomogenous distribution of Cu and other elements in alloy microstructure, these intermetallic are thermodynamically stable because of its low solubility in aluminium (Boag et al., 2011; Buchheit et al., 1997).

One of the ways to protect AA from localised corrosion is anodisation. Anodisation is a process which passive films of AA are created and enhanced by electrochemical process. A naturally formed passive film is replaced with an artificially formed, thick, and dense passive film (Lee and Park, 2014). Porous anodised aluminium layer is self-organised of hexagonal arrays over a barrier layer at the bottom (Abd-Elnaiem and Gaber, 2013). This anodised film can protect AA when exposed to actual environment. However, localised corrosion, e.g., pitting corrosion can still occur under aggressive environment. Pitting can lead to fatigue failure which is detrimental to the material in service (Rokhlin et al., 1999; Sankaran et al., 2001).

Zhu et al. (2017) investigated the effect of Si particle on corrosion protection of anodised cast Al-Si alloy. The study found that an oxide layer on Si particles was thinner than the one on the Al matrix. Si constituents also affected cracks and defects in the anodised film. Ma et al. (2019) conducted immersion test to induce corrosion on anodised AA2055 and found that localised corrosion initiated beneath the film and propagated through cracks. The rate of propagation was controlled by diffusion process. Veys-Renaux et al. (2016) studied AA1050, 7175 and 2618 and found that impedance was lower in the presence of defects. Curioni et al. (2010) found that morphology of the anodised film on intermetallic particles of AA2024 was different from the film on the matrix. The different morphology of the film on intermetallic particles was confirmed by Moon et al. (2004) who used confocal microscope to characterise the oxide film formed on Al-Mg and Al-Mg-Fe intermetallic phases.
In an aerospace industry, the formation of surface defects is very critical and has to be minimised as low as possible. Anodised AA2024 (copper-rich Al alloy) and AA7075 (zinc-rich Al alloy) are used as aerospace parts, i.e., structural frame and fuselage. The differences of these two grades were the composition and intermetallic compounds. Intermetallic particles in AA2024 included Al$_2$Cu, Al$_2$CuMg, Al$_4$Cu$_2$Fe, and Al$_{20}$Cu$_2$Mn$_3$ (Li, 2014). The most abundant intermetallic particles in AA7075 were Al$_7$Cu$_2$Fe, (Al,Cu)$_6$(Fe,Cu), and MgZn$_2$ (Andreatta et al., 2003).

Based on the technical requirements in aerospace applications, the materials have to pass the accelerated corrosion test under salt fog environment according to ISO 9227 or ASTM B177 for 96 hours prior to further production. For this reason, the anodising of aerospace parts made of AA is widely employed in order to achieve excellent corrosion resistance performance. It is well-known that corrosion resistance of AA is improved by anodisation. However, there is still lack of information regarding root cause determination when such anodised film is destroyed by localised corrosion attack. Root causes of pitting corrosion of anodised AA are critical in order to minimise rejected parts in the future production. This study aimed to investigate the effect of the anodised film thickness of AA2024 and AA7075 alloys on protecting the localised corrosion and to understand corrosion root causes. In addition, potentiodynamic polarisation tests were used to identify whether the film will pass or fail the salt spray.

2 Experimental procedures

The investigated materials were two grades of AA sheets: AA2024 and AA7075. Chemical compositions of these two grades were provided in Table 1. It shows that AA2024 is a copper-rich AA and AA7075 is a zinc-rich alloy.

Table 1 Chemical compositions of the two grades of AA in this study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Cu</th>
<th>Zn</th>
<th>Fe</th>
<th>Si</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Ti</th>
<th>Zr</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>AA2024</td>
<td>4.94</td>
<td>0.08</td>
<td>0.22</td>
<td>0.08</td>
<td>0.63</td>
<td>1.54</td>
<td>0.02</td>
<td>0.02</td>
<td>0.003</td>
<td>Bal.</td>
</tr>
<tr>
<td>AA7075</td>
<td>1.50</td>
<td>5.82</td>
<td>0.17</td>
<td>0.08</td>
<td>0.04</td>
<td>2.81</td>
<td>0.19</td>
<td>0.02</td>
<td>0.01</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

The anodised film was introduced on the surface of AA samples by using an industrial-scaled anodising process. The steps of anodisation included, first, alkalining step with 45 g/L concentration of Turco 4215 NCLT solution at 55°C. Deoxidising step was done in a bath filled with 23%v Ardrox 295GD at 35°C. The constant voltage was applied during anodisation at 14 volt. Lastly, the sealing step was done in deionised water at 90°C. Each step was preceded with rinsing the sheet with DI water to minimise contamination occurred in the anodising process. The amount of chloride ions was controlled to be less than 80 ppm in all baths. Prior to surface analysis, every sample was rinsed thoroughly with DI water and kept in a dehumidified chamber. Three types of samples were compared to determine root causes of pitting corrosion:
pre-exposed films that had not been through the salt spray test
2. samples that passed the salt spray test (passed samples)
3. samples that failed the salt spray test (failed samples).

The pre-exposed intact films were used as a reference, which was assumed to have no damage of the anodised film. The duration of the salt spray test was 96 hours. Passed/failed criteria was determined by number of pits per surface area. A passed sample must have less than 10 pits/100 cm².

Surface morphology of the film was characterised by high-resolution Olympus STM7 optical microscope and JEOL 7800F Prime scanning electron microscope (SEM). Roughness measurements were conducted with an Olympus LEXT confocal microscope by measuring three areas of 0.6 × 0.6 μm field of view. The roughness parameters were measured for the entire image. The average values were shown in the results section. Energy dispersion X-ray spectroscopy (EDS) was used to determine elemental distribution in an area with the presence of pitting corrosion and its neighbourhood.

Pre-exposed samples were polarised to create pitting corrosion by anodic polarisation. Pitting from anodic polarisation was compared with pitting from passed/failed samples. To overcome the challenge of doing electrochemical experiments on nonconductive anodised films, it was required to soak the films in 1 M NaCl solution for ten days prior to electrochemical experiments, based on our preliminary study. A conventional 3-electrode corrosion cell connected to Autolab 302N potentiostat was employed for electrochemical corrosion study. The Ag/AgCl reference and graphite counter electrodes were used. In order to obtain pitting corrosion resistance property, potentiodynamic polarisation measurements were conducted in 1 M NaCl solution at 25°C. The samples were polarised from 300 mV below open circuit potential (OCP) until pitting potential at a scan rate of 0.5 mV/s. All of the electrochemical procedures with three replicates were done. After completion of electrochemical corrosion study, the tested samples were analysed by SEM and EDS to qualitatively determine elemental compositions of areas around and inside the pits.

Comparison and analysis of all samples elucidated the roles of morphology, intermetallic particles, defects, and electrochemical behaviour on pitting corrosion of anodised film.

### 3 Results

#### 3.1 Roughness measurements

In fact, surface profile of AA is one of the crucial factors affecting their corrosion resistance property. It is well known that increased surface roughness can facilitate the pit initiation. A chance of pit initiation on a round surface is higher than on a flat surface. Usually, arithmetic mean of the profile, \( R_a \), is the most popular parameter used to describe average roughness of the surface. \( R_p \) is the maximum peak height. \( R_v \) is the maximum valley depth. \( R_z \) is the distance between \( R_p \) and \( R_v \) as shown in Figure 1. The roughness measurements were made on three different areas on the same sample.
Figure 2 shows the comparison of roughness parameters of pre-exposed, passed and failed samples. In AA7075, $R_a$, $R_p$ and $R_z$ of the failed sample were the highest compared to those of the pre-exposed and passed samples. $R_v$ of the passed sample was slightly higher than that in the pre-exposed and failed sample. In AA2024, all of the roughness parameters ($R_a$, $R_p$, $R_v$, and $R_z$) of passed samples were the highest. The failed sample of AA2024 had lower roughness parameters than those of failed samples of AA7075.

**Figure 1** Diagram showing roughness parameters (see online version for colours)

3.2 **Morphology by optical microscope**

Figures 3 and 4 show the optical micrographs of (a) passed and (b) failed AA2024 and AA7075 samples, respectively. The surface morphologies of passed samples of both grades were similar and contained a lot more scratches than those of the failed samples. However, the failed samples contained a larger number of small pores and pits, that were formed during the salt spray test, than the passed samples.

3.3 **SEM images**

Figure 5 shows SEM images of surface morphology of pre-exposed AA2024 and AA7075 samples. Overall, the anodised film looks dense and smooth. However, in some regions, there are some cracks (dark lines) and holes as shown in Figure 5(a). The inside of the holes seems to be very rough and contains many dimples as shown in Figure 5(b). Figure 5(c) shows that pre-exposed film of AA7075 contains some cracks and defects like the ones in AA2024.

Figures 6 and 7 show SEM images of the surface of the film of AA2024 and AA7075 after the salt spray test, respectively. Passed sample of AA2024 seems to be denser than the failed samples. Both passed and failed samples contain pits. Diameter of a pit in the passed samples seems to be smaller than the one in the failed samples. Cracks of the film were observed around the pits in all samples. Surfaces of the passed/failed samples seemed to be rougher than the pre-exposed film, which appeared smoother (Figure 5).
Figure 2  Roughness parameters from pre-exposed, passed and failed samples of AA2024 and AA7075 (see online version for colours)
Figure 3  OM images showing surface morphology of, (a) passed (b) failed AA2024 samples after 96-hour salt spray testing (see online version for colours)

Figure 4  OM images showing surface morphology of, (a) passed (b) failed AA7075 samples after 96-hour salt spray testing (see online version for colours)

Figure 5  SEM images of pre-exposed surface of anodised AA2024 show, (a) some cracks on the surface (b) dimples on the surface (c) pre-exposed surface of anodised AA7075
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Figure 5  SEM images of pre-exposed surface of anodised AA2024 show, (a) some cracks on the surface (b) dimples on the surface (c) pre-exposed surface of anodised AA7075 (continued)

Figure 6  SEM images of AA2024, (a) passed (b) failed samples after 96-hour salt spray testing

Figure 7  SEM images of AA7075, (a) passed (b) failed samples after 96-hour salt spray testing
3.4 EDS mapping

Energy dispersive X-ray spectroscopy (EDS) mapping shows an elemental distribution on the surface. EDS mapping results are capable of revealing impurities on the anodised surface because EDS shows which area is enriched by certain element. Random three
areas on the sample surface were observed for each specimen. There is a possibility that aluminium oxides do not cover impurity in certain spots. Figure 8 shows the elemental distribution maps of pre-exposed intact AA2024 that contained some defects. Only important elements were shown. The defects contained high Mg, low oxygen and low aluminium. Cu does not seem to be concentrated in the defect. These spots are cavity in the anodised film. However, these spots are not necessary becoming pitting corrosion when exposed to the salt spray test.

3.5 Cross-sectioned SEM

Cross-section analysis of the passed and failed samples of AA2024 and 7075 alloys was conducted using SEM. As shown in Figures 9, the anodised film thickness of around 2–3 µm was obtained for both passed and failed AA2024 samples. In case of AA7075 alloy, the anodised film of failed samples is much thicker than that of the passed sample. The anodised film thickness of passed sample was approximately 3 µm while that of the failed samples was approximately 40 µm as shown in Figure 10.

Figure 9  Cross-sectioned images showing the anodised film thickness of, (a) passed (b) failed AA2024 samples

Figure 10  Cross-sectioned images showing the anodised film thickness of, (a) passed (b) failed AA7075 samples
Figure 11  Polarisation curves of pre-exposed (black dot), passed (red square), and failed (blue triangle) samples in, (a) AA2024 (b) AA7075 (see online version for colours)
3.6 Electrochemical behaviour of the films

In potentiodynamic polarisation curves, corrosion potential \( (E_{corr}) \) and corrosion current density \( (i_{corr}) \) had to be evaluated simultaneously to determine how anodic and cathodic branch were affected. Figure 11 shows the polarisation curves obtained from both AA2024 and AA7075. It can be seen that the anodic current density was obviously reduced after the samples were exposed to the salt spray environment while the cathodic current density remained relatively in the same order of magnitude. When Tafel extrapolation was fitted to the polarisation curves, corrosion potential \( (E_{corr}) \) and corrosion current density \( (i_{corr}) \) could be extracted out from the plots. In addition, the breakdown potential was also analysed from the plots in order to classify the ability of
Al alloys to resist pitting corrosion attack. Figure 12 indicates the $E_{\text{corr}}$, $i_{\text{corr}}$ and $E_b$ values extracted from the polarisation curves of AA2024 and 7075 alloys with pre-exposed, passed and failed conditions. In both AA7075 and AA2024, the failed samples exhibit the highest value of all three, despite marginal differences. These results are in contrast with the visual observation that the failed sample has more pits than the passed samples.

4 Discussion

From electrochemical results, the failed samples has the highest breakdown potential, the lowest corrosion current, and the lowest corrosion potential. High breakdown means that the films are strong. Low corrosion potential with low current density can imply that the rate of the cathodic reaction decreases as illustrated in Figure 13.

The results suggest that the anodised film on the failed sample slows down oxygen migration from environment, thus, the rate of the cathodic reaction decreases. The results yield different indication of corrosion tendency compared to the visual observation. Passivity range of the pre-exposed samples was longer than ones of the passed and failed samples. Longer passivity range indicates that the passive film is stronger than ones whose passivity is short. In other words, exposure in salt spray chamber weakened the anodised film of a sample. Shorten passivity of anodised film correlates well with low corrosion protection. Amin et al. (2008) published potentiodynamic polarisation curves of pure Al in 0.25 M NaClO$_4$ solution and found that adding SO$_3^{2-}$ as an inhibitor strengthened the passivity. Therefore, potentiodynamic polarisation behaviours alone cannot be used to identify the tendency for localised corrosion. It can only determine passivity range of the samples. The reason was that the area of testing was only 1 cm$^2$. The pitting corrosion that occurred on the whole sample, was not in the level that shows
any significant differences on the polarisation curves. The area outside of testing area may be the point of attack. The electrochemical techniques alone cannot determine if the film will pass or fail the salt spray test unless the whole area is tested.

Surface roughness of the anodised film cannot predict if the sample will pass or fail the salt spray test. From the results, average roughness ($R_a$), peaks ($R_p$), and distance from peak to valley ($R_z$) were the greatest in failed samples of AA7075. However, the passed samples of AA2024 exhibited the greatest values. The roughness parameters from AA2024 and AA7075 were contradicting. The protectiveness of pitting corrosion of anodised film cannot be determined by roughness measurement alone. Figure 3 and 4 shows the difference of the passed and failed sample. The film on the passed samples seems to be denser than that of the failed samples. The failed samples have small holes on the surface while the passed samples contain rather large scratches. Scratches and cracks could also affect the roughness results. These cracks and defects can sometimes, but not necessary, be an origin of pitting corrosion of AA. Cracks in the film may be caused by residual stress during film growth (Li et al., 1998).

Thickness of the anodised film cannot prevent localised corrosion after the salt spray test. The failed samples has film thickness of 40 µm while the passed samples have film thickness of 3 µm. This shows that thickness of the film does not prevent the pit initiation. All samples were anodised with the exact same procedures despite differences in thickness. Passed and failed samples did not show any difference in morphology from cross-sectioned images. Anodised aluminium film is typically developed into two layers: porous and barrier layer. The porous layer is columnous and does not contribute significantly to corrosion protection. On the other hand, the barrier layer is dense and provide protection to corrosion (Abd-Elnaiem and Gaber, 2013; Kim et al., 2005; Ghahremaninezhad and Dolati, 2009; Lou et al., 2020). In the current investigation, the thickness of the anodised film includes both porous and barrier layers. The film might contain bigger proportion of porous layer, so it has poor corrosion protection. The corrosion protectiveness should be determined by the thickness of the barrier layer, specifically. This claim was confirmed by Ma et al. (2020) who studied the effect of barrier layer on anodic film of AA2055. They found that barrier layer was a decisive factor in corrosion protection (Ma et al., 2020).

After pitting corrosion was initiated by anodic polarisation, the samples were analysed by SEM/EDS to determine the morphology and the composition of pits and surrounding areas. Inside a pit, there seems to have high Cu peaks while other areas do not show high Cu peaks. At the edge of the pit, the EDS peaks shown in Figure 14 are similar to the matrix and inside the small pit.

EDS spot analysis on pitting corrosion of AA2024 and AA7075 for both passed and failed samples also shows similar results. Overall, chloride seemed to be present after the salt spray test on both passed and failed samples. In AA2024 samples, besides Al and O peaks, Cu and Mn peaks were present. Inside a pit, the EDS spectrum shows higher Cu peaks than in the matrix whose spectrum contained Al, O, Cu, and Mn peaks. In AA7075 samples, Cu and Zn peaks were higher inside the pit than the matrix, which showed Al, O, and Zn peaks. Cu peak was not present on the matrix. This result was in agreement with the results from pit initiated from anodic polarisation. Cu-bearing intermetallic particle was the point of attack for localised corrosion.
Figure 14  SEM image and EDS spectra of a pit of pre-exposed AA2024 sample (see online version for colours)

Note: The pit was initiated by anodic polarisation.

AA2024 has Cu as a primary alloying element while AA7075 contains Zn as a primary alloying element. Typically, Cu-bearing intermetallic particles increase corrosion potential (Aballe et al., 2000). The results agree that corrosion potential of AA2024 is higher than that of AA7075. This implies that corrosion protection of AA2024 should be better than AA7075. However, the pitting corrosion attack was very localised. AA2024 is not any better than AA7075 after the salt spray test. Both AA2024 and AA7075 samples failed the salt spray test due to Cu-bearing intermetallic particles that were a weak spot for corrosion attack.

AA2024 contains intermetallic particles that are detrimental for corrosion resistance. The primary intermetallic particle is S phase (Al₂CuMg) (Buchheit et al., 1997). This phase is a pit origination in most cases. The passive film that covers this phase is not as strong as other regions in the matrix, so this is the point of attack. In other words, pitting corrosion initiates from intermetallic particles. From the EDS results, it can be concluded that Cu-bearing intermetallic particles seems to be the point of pit initiation in both grades. The intermetallic particles in AA systems include Mg₂Si, MgZn₂, Al₁₂₂₃₃₄, Al₃₄₃₃₄, Al₅₆₇₈₉₀, Al₅₆₇₈₉₀, and Mg₆₇₈₉₀ (Birbilis and Buchheit, 2005). It is important to note that not all of these intermetallic particles are present simultaneously depending on the composition and the heat treatment. Anodised film that forms on Cu-bearing intermetallic particles is not as strong as the one formed on the Al matrix. This phenomenon happened in both grades, AA2024 and AA7075, in this study.

Anodising AA can be challenging due to the presence of second phase particles or intermetallic phases. These phases can alter compositions and morphology of the film. During anodising, some of the intermetallic particles are partially anodised and become a point of corrosion attack. Moreover, after the intermetallic was completely corroded, remnant of these particles could promote cracks and void in the anodised film.
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(Usman et al., 2020). There are some evidences indicating that the film contains Cu-bearing intermetallic particles. Wu et al. (2018) studied low-copper-containing (LCCPs) and high-copper-containing Al-Fe-Mn-Cu particles (HCCPs) and found that HCCPs dissolved preferentially in tartaric-sulphuric acid and had much higher tendency to fail in immersion test in 3.5% NaCl solution for 24 hours than LCCPs. The corrosion resistance of the anodised film was directly related to locations of intermetallic particles at the film/alloy interface (Wu et al., 2018). Ma et al. (2019) studied anodised film on Al-Cu-Li alloys and claimed that cavity between film/alloy interface were responsible for localised corrosion resistance property of anodised AA. Intermetallic particles were difficult to be anodised. During anodisation process, some oxide film formed on intermetallic particles and dissolved completely while some dissolved partially. In both cases, those regions became more susceptible to corrosion attack (Ma et al., 2019). Veys-Renaux studied anodising of multiphase AA in sulphuric acid. They found that second phases of AA created defects and greatly reduced the corrosion resistance of the film (Veys-Renaux et al., 2016). During anodisation process, tartaric-sulphuric could dissolve HCCPs and caused cavity in the film while LCCPs were anodised at much slower rate than the matrix leaving remaining particles in the film (Ma et al., 2011). Anodised AA2099 were exposed to salt spray test and the results agreed with our experiment that HCCPs was a point of attack (Ma et al., 2016). The generation and rupture of oxygen gas bubbles during anodising of Cu at the film alloy interface was the reason why defects occurred at HCCPs (Curioni et al., 2010; Ma et al., 2011).

All literature supports our findings that Cu-bearing intermetallic particles are the problem in anodic film formation. To improve localised corrosion resistance, it is suggested to reduce high Cu-bearing intermetallic particles by heat treatment or improving process of anodisation on Cu-bearing intermetallic particles.

5 Conclusions

Anodised films of AA2024 and AA7075 were analysed using various techniques to determine the root cause of the failure, so possible solution can be suggested. From experimental results, we can conclude the following:

• Roughness alone could not tell how protective the film was. There was no significant difference in roughness between pre-exposed, passed, or failed samples.

• Pre-exposed anodised films were dense but contained some cracks and defects that showed a depletion of oxygen and aluminium.

• Characterisation of corrosion protectiveness of the anodised film by electrochemical test could not predict whether the film would pass or fail the salt spray test. The potentiodynamic polarisation test alone was not a suitable test method to determine the protectiveness to localised corrosion.

• Thickness of the film did not prevent the pit initiation. The failed samples with thick film had higher porosity than the passed samples. The thickness of the barrier layer was more important in protecting the film from aggressive environment.

• Cu-bearing intermetallic particles were the point of pit initiation in both AA2024 and AA7075.
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