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The influence of grain structure on the corrosion behaviour of 2A97-T3 Al-Cu-Li alloy

X. Zhang¹, X. Zhou¹,*, T. Hashimoto¹, J. Lindsay¹, O. Ciuca¹,
C. Luo², Z. Sun², X. Zhang², Z. Tang²

¹ Corrosion and Protection Centre, School of Materials, the University of Manchester, Manchester, M13 9PL, UK
² Aviation Key Laboratory of Science and Technology on Advanced Corrosion and Protection for Aviation Materials, Institute of Aeronautical Materials, Beijing 100095, China

* Corresponding Author: telephone: 01613064832, e-mail address: xiaorong.zhou@manchester.ac.uk

Highlights:

- Corrosion occurs preferentially along the subgrain boundaries in 2A97-T3 alloy.
- Kikuchi diffraction enables the correlation of corrosion to local grain structure.
- The regions with high stored energy are more susceptible to corrosion.
- Grain stored energy plays a decisive role in corrosion propagation.

Abstract

In the present study, the corrosion behaviour of 2A97-T3 Al-Cu-Li alloy is investigated. The corrosion propagation path is characterized with electron backscatter diffraction (EBSD) and transmission Kikuchi diffraction (TKD), and correlated to local grain structure and the distribution of T₁ (Al₂CuLi) and T₈ (Al₇Cu₄Li) phase precipitates in the alloy. Localized corrosion occurs preferentially along the subgrain boundaries within unrecrystallized grains of this partially recrystallized alloy. The grain stored energy plays a more decisive role in corrosion propagation compared to T₁ phase precipitate. The grain boundaries decorated by T₈ phase precipitates exhibit relatively low susceptibility to corrosion.

Keywords:

A. Aluminium
B. SEM, TEM
C. Intergranular corrosion
1 Introduction

In order to reduce the weight of aircraft, new generation Al-Cu-Li alloys are increasingly used for structure parts such as floor beams, fuselage and wing stringer in modern aircrafts. However, microstructural heterogeneity in the alloys may compromise corrosion resistance and limits wide application of Al-Cu-Li alloys in aircraft industry. Thus, numerous works are devoted to the understanding of corrosion behaviour of Al-Cu-Li alloys, with particular focus on intergranular corrosion (IGC), which is considered as one of the most harmful type of corrosion in structural parts.

Previous work suggests that intergranular corrosion susceptibility of Al-Cu-Li alloys is mainly associated with compositional heterogeneity along the grain boundary area [1-12], i.e. the presence of precipitates and precipitate free zone along grain boundary. It is found that [1] the selective dissolution of grain boundary $T_1$ phase precipitates results in intergranular corrosion [1]. It is also found that during the immersion of Al-Cu-Li alloys in NaCl solution, the grain boundaries decorated with high population density of $T_1$ phase precipitates display high intergranular corrosion susceptibility. In contrast, relatively low susceptibility to corrosion was observed at the grain boundaries with a comparatively low population density of $T_1$ phase [2]. In Kumai’s work, it is suggested that pitting in the precipitate free zone along grain boundaries initiates intergranular attack in Al-Cu-Li alloys [3]. Further, it is also pointed out that intergranular corrosion susceptibility is closely associated with the width of precipitate free zone and the size of grain boundary $T_1$ phase precipitates in Al-Cu-Li alloys [4, 5].

Recent investigation on the relationship between the dislocation distribution in the periphery of grain boundary and the susceptibility to IGC [8, 9, 13-15] indicates that, in Al-Cu-Li alloys, increased number of dislocations leads to higher localized corrosion susceptibility, resulting in preferential attack of grain boundaries surrounding grains of relatively high stored energy, i.e. high density of dislocations. In order to further advance the understanding of the relationship between the distribution of precipitates, the grain structure, especially local plastic deformation and the associated thermomechanical history, and the corrosion behaviour of Al-Cu-Li alloys, in the present study, electron microscopy equipped with electron backscatter diffraction (EBSD) and transmission Kikuchi diffraction (TKD) facilities are employed to investigate the influence of both compositional and structural heterogeneity on the corrosion behaviour of 2A97 Al-Cu-Li alloy in T3 condition.

2 Experimental procedure

2A97-T3 Al-Cu-Li alloy (Li 1.5 wt.%; Cu 3.7 wt.%; Zn 0.5 wt.%; Mg 0.3 wt.%; Mn 0.3 wt.%; Zr 0.1 wt.%; Al rem.) was employed in this study. The alloy was solution treated, cold worked and naturally aged to achieve the T3 condition. The specimens were prepared from a cold-rolled thin sheet of 1 mm thickness. The specimens were successively ground with silicon carbide papers to 4000 grit and polished sequentially using 3 μm and 1 μm diamond pastes.
After the mechanical polishing, the specimens were cleaned ultrasonically in an acetone bath and dried in a cool air stream.

The mechanically-polished specimen was immersed in a 3.5 wt.% NaCl solution at ambient temperature for 5 h. After the immersion testing, the specimen was thoroughly rinsed with deionized water and dried in a cool air stream. In order to characterise the corrosion propagation path beneath corrosion product, the specimen was gently polished with 0.25 µm diamond paste to remove the corrosion product and then cleaned by low intensity argon plasma etching using glow discharge optical emission spectroscopy (GDOES) equipment (Horiba Jobin Yvon GDOES) for 10 s at an argon pressure of 750 Pa and applied voltage of 25 V with a power of 15 W.

Electron transparent thin foils for transmission electron microscopy (TEM) were prepared from the as-received alloy using twin-jet electropolishing in a mixture of 700 ml methanol and 300 ml nitric acid at the temperature of -30 °C. The mixture of methanol and nitric acid was also employed to electropolish the alloy surface to obtain a contamination free surface for low accelerating voltage scanning electron microscopy (SEM) and EBSD analysis. Ultramicrotomy was used to generate cross sectional blocks and electron transparent foils from the corrosion sites of the tested specimens. The latter were examined by TEM operating at 300 kV along with energy dispersive X-ray (EDX) and electron energy loss spectroscopy (EELS). In addition, focussed ion beam (FIB) was also employed to obtain cross sectional thin foils from corrosion sites for transmission Kikuchi diffraction (TKD) analysis.

3 Results

3.1 Microstructural characterization

3.1.1 Precipitates

Figure 1 (a) displays a SEM micrograph of the as-received 2A97-T3 Al-Cu-Li alloy, revealing a grain boundary and two needle-shaped precipitates with the length up to approximate 1 µm, as indicated by the arrows. Such precipitate is also shown in the bright field TEM micrograph of Figure 1 (b), as indicated by the white arrow. EDX analysis of the precipitate reveals its copper rich nature. Since lithium is undetectable by EDX, EELS was employed to determine the distribution of lithium. The Li EELS map obtained from the framed area in Figure 1 (b) is shown in Figure 1 (c), revealing that the needle-shaped precipitate is also rich in lithium. In order to determine its crystal structure, lattice fringe image of the needle-shaped precipitate was obtained, as shown in Figure 1 (d). The d-spacing and the angle between the planes are consistent with the parameters of the cubic T_B phase (Al_7Cu_4Li) [2, 16, 17]. It is worth noting that T_B phase precipitates are only occasionally present at the grain boundaries in the alloy.

In addition to the needle-shaped T_B phase precipitates of micrometre length at the grain boundaries, significantly shorter needle-shaped precipitates are also occasionally found at grain boundaries in the alloy. The backscatter electron SEM micrograph of Figure 2 (a)
shows a short needle-shaped precipitate with the length of approximate 100 nm at the grain boundary, as indicated by white arrows. The bright field TEM image of Figure 2 (b) shows three short needle-shaped precipitates at the grain boundary, as indicated by the white arrows. The corresponding copper and lithium EELS maps of the region shown in Figure 2 (b) are displayed in Figures 2 (c)-(d), indicating that the short needle-shaped precipitates are rich in both lithium and copper. Due to the relatively strong background copper intensity associated with the solute in the alloy matrix, the precipitates outlined by copper EELS map seems to appear thicker than that in the lithium EELS map. A lattice image of a typical short needle-shaped precipitate is displayed in Figure 2 (e), revealing that the short needle-shaped precipitates have the hexagonal structure of T1 phase [18]. Further, examination of a large number of randomly selected grain and subgrain boundaries indicates that T1 phase precipitates are present at a very small portion of the grain and subgrain boundaries in the alloy.

3.1.2 Grain structure

EBSD was performed on the electropolished alloy surface to determine its grain structure. A step size of 0.5 µm was used and over 90% of the examined area could be indexed. Due to the presence of intermetallics or other defects, the diffraction intensity of aluminium is low in the other 10% of the examined area, therefore, was not indexed. Figure 3 (a) shows the crystallographic orientation distribution of the examined region in Euler’s colours, with yellow lines and black lines representing high angle grain boundaries (HAGBs, θ>15°) and low angle grain boundaries (LAGBs, 1°<θ<15°) respectively. It is evident that two types of coarse grains of similar sizes, with and without subgrains, are present in the alloy, indicating that the alloy is partially recrystallized. The unrecrystallized, but recovered grains consist of subgrains with dimensions of 5-10 µm.

The distribution of stored energy [13, 14] in the region is displayed in Figures 3 (b)-(c). For a neighbouring pixel pair with misorientation above threshold value of 1°, the stored energy is calculated using Read-Shockley equation [13, 14]. Then, the stored energy of an individual grain / subgrain is determined by averaging the sum of energy of all pixel pairs within the grain / subgrain. Subsequently, the spatial distribution of grain-stored energy is determined and displayed as stored energy map in grey-scale, with higher brightness representing higher level of stored energy. As expected, the recrystallized grains exhibit relatively low stored energy and the unrecrystallized grains with subgrains possess relatively high stored energy. Interestingly, the stored energy varies significantly from subgrain to subgrain within the unrecrystallized grains, as shown in Figure 3 (c).

3.2 Characterization of corrosion sites

3.2.1 Corrosion morphology
Examination of the alloy surface after immersion in a 3.5 wt. % NaCl solution for 5 hours reveals a number of localized corrosion sites. Figure 4 (a) displays the plan-view of a typical localized corrosion site on the alloy surface. The bright feature on the alloy surface is corrosion product. Narrow and dark bands are evident within the corrosion site, suggesting that localized corrosion developed in the form of intergranular corrosion. Interestingly, the sizes of the grains outlined by the attacked grain boundaries in Figure 4 (a) are in the same range as that of the subgrains revealed in Figure 3. Thus, the development of the localized corrosion indicates heterogeneous corrosion susceptibility of the alloy with the subgrain boundaries within unrecrystallized grains being preferentially attacked.

Further, Figure 4 (b) displays the cross section of another localized corrosion site in the alloy after immersion in a 3.5 wt. % NaCl solution for 5 hours, exhibiting attacked grain boundaries at various depths. Again, the sizes of the grains outlined by the attacked grain boundaries are in the same range as that of the subgrains, indicating that corrosion propagated along the subgrain boundaries beneath the alloy surface. The framed areas in Figure 4 (b) are displayed in Figures 4 (c)-(d) at increased magnifications, revealing needle-shaped precipitates at grain boundaries, as indicated by the white arrows. The dimensions of the needle-shaped precipitates are in good agreement with those of T_B phase precipitates. It is evident the grain boundary decorated with T_B phase precipitates remains intact (Figure 4 (c)). Further, the T_B phase precipitate intersecting the alloy surface also remains intact (Figure 4 (d)), suggesting its relatively low susceptibility to corrosion.

3.2.2 The influence of grain structure

In order to determine the influence of grain structure on the corrosion behaviour, EBSD analysis was carried out on the tested specimens. Figure 5 shows a SEM micrograph of typical localized corrosion sites and corresponding EBSD maps, exhibiting attacked grain boundaries in the region. Again, the sizes of the grains outlined by the attacked boundaries are in the same range as that of the subgrains, indicating preferential dissolution of subgrain boundaries. As described in the experimental section, the tested specimen was cleaned using low intensity argon plasma etching prior to the EBSD analysis in order to obtain good quality EBSD. However, such plasma etching might introduce artefact to the specimen. To assess the effect of plasma etching, the tested specimens were examined in SEM before and after plasma etching, as shown in Figures 5 (a) and (b). It is evident that the corrosion morphology is identical on both the specimens with and without plasma cleaning although the widths of the attacked grain boundaries are increased slightly due to enhanced etching rate at the edges and shallow craters are introduced to the specimen surface due to non-uniform etching rate at locations of intermetallic particles on the specimen with plasma cleaning. Thus, the EBSD obtained from the plasma etched specimen provides reliable orientation map of the intact grain matrix. The grain orientation distribution of the localized corrosion site is displayed in Euler’s colour in Figure 5 (d). Due to the low diffraction intensity, the corroded regions could not be indexed. The corresponding grain-stored energy distribution is displayed in Figures 5 (e), with higher brightness representing higher level of stored energy. Comparing the SEM micrograph (Figure 5 (c)) with the corresponding grain-stored energy map (Figure 5 (e)), it is evident that corrosion preferentially occurred in the region with high level of stored energy.
Further, the distribution of subgrain-stored energy is also shown in Figure 5 (f), exhibiting subgrains of various stored energy. It is evident that the subgrain boundaries within an unrecrystallized grain were preferentially attacked.

However, due to the limitation to the spatial resolution of EBSD analysis in SEM, it is difficult to correlate corrosion susceptibility of an individual subgrain boundary with the associated stored energy. Therefore, TKD analysis with step size of 15 nm along with TEM observation was performed. Focussed ion beam was employed to obtain electron transparent cross sections from localized corrosion sites in the alloy after the immersion testing for TKD analysis. Figure 6 (a) displays a HAADF micrograph of a localized corrosion site characterized by attacked subgrain boundaries. The corresponding grain orientation distribution in Euler’s colour, with green being applied to regions that could not be indexed due to low diffraction intensity, is shown in Figure 6 (b). The non-indexed attacked subgrain boundaries and the subgrains are clearly revealed.

The distribution of stored energy of the subgrains is shown in Figure 6 (c) in grey scale. Comparing the corrosion morphology (Figure 6 (a)) with stored energy distribution (Figure 6 (c)), it is evident that the attacked boundaries are those of subgrains with high stored energy. For example, boundary A-F (the boundary between subgrains A and F) remains intact in contrast to the partially attacked boundary A-B (Figure 6 (d)) locating adjacent to grain B which has relatively high level of stored energy compared to grains A and F. Further, compared to boundary A-B, boundaries A-D and A-E, locating adjacent to subgrains D and E that have higher stored energy than subgrain B, were more severely attacked. In summary, for the boundaries surrounding subgrain A, namely boundaries A-B, A-D, A-E and A-F, those between subgrain A and the subgrains with higher stored energy were preferentially attacked. Therefore, it is suggested that boundaries surrounding the subgrains with high stored energy display higher susceptibility to corrosion.

Further, the bright field TEM micrograph of framed area 1 in Figure 6 (a) is shown in Figure 6 (d). It is evident that corrosion was not only confined to the subgrain boundary, but further developed unevenly into the two neighbouring subgrains. Scrutiny of Figure 6 (d) reveals that corrosion preferentially developed into subgrain B (higher stored energy) but not into subgrain A (lower stored energy), suggesting that corrosion tends to propagate into the subgrains with higher stored energy. The high angle annular dark field (HAADF) micrograph of the framed area 2 in Figure 6 (a) is displayed in Figure 6 (e). As indicated by the white solid line arrow, corrosion preferentially developed into grain C which has the highest stored energy amongst the subgrains within the region. It is worth mentioning that a number of the corrosion sites on specimens from repeating testing have been characterized with EBSD and TKD, showing the correlation between the corrosion propagation and the local plastic deformation.

In addition, the copper-rich T1 phase precipitates are clearly revealed as bright fine needles in Figures 6 (a) and (e) since HAADF imaging mode is sensitive to the atomic number with the higher brightness representing heavy element. Interestingly, the boundary A-F is decorated by relatively more T1 phase precipitates compared with other boundaries, but remains intact.
This perhaps indicates that in this alloy the distribution of stored energy plays a more decisive role in corrosion propagation along the subgrain boundaries compared to the presence of T$_1$ phase precipitates.

4 Discussion

In this 2A97-T3 Al-Cu-Li alloy, two types of precipitates, namely short needle-shaped T$_1$ phase precipitates and long needle-shaped T$_B$ phase precipitates, are detected at grain boundaries occasionally. As described in the introduction, previously the susceptibility to corrosion in Al-Cu-Li alloys is attributed to either the selective dissolution of T$_1$ phase along the grain boundary [2, 19] or the preferential attack of the precipitate free zone [3]. Due to the relatively high content of Li in T$_1$ phase, T$_1$ phase has a more negative potential (-1.096 V vs SCE) relative to the alloy matrix (-0.7529 V vs SCE) [1]. Thus, during the immersion in NaCl solution, selective dissolution of Li occurs first, resulting in the copper-rich T$_1$ phase remnant and, consequently, the potential shift to positive direction. Subsequently, the micro-coupling between the copper-rich remnant and the alloy matrix leads to the anodic dissolution of the adjacent alloy matrix [20, 21]. Therefore, T$_1$ phase precipitates facilitate the anodic dissolution of aluminium in the periphery, resulting in localized corrosion of the regions containing high population density of T$_1$ phase precipitates. Compared to T$_1$ phase precipitates, Li content in T$_B$ phase is much lower. This explains the relatively inert behaviour of T$_B$ phase during the immersion testing (Figure 4).

However, although only a small fraction of the grain/subgrain boundaries in this alloy in T3 condition is decorated by T$_1$ phase precipitates, localized corrosion occurred preferentially along the subgrain boundaries within unrecrystallized grains of the partially recrystallized alloy, suggesting that in addition to the influence of compositional variation in the grain boundary regions, there must be other factors that also influence the development of corrosion in the alloy.

In the present work, both EBSD and TKD were employed to determine the distribution of stored energy within individual grains. Since the stored energy is closely associated with the degree of misorientation, stored energy map is used to quantitatively visualise the distribution of dislocations. The stored energy is determined by the misorientation between the neighbouring pixels selected in EBSD or TKD analysis. When the misorientation is above a selected threshold value (in the present work, 1 °), its stored energy could be calculated by Read-Shockley equation [14, 15, 22]. The stored energy of an individual grain / subgrain is determined by averaging the sum of the stored energy of pixels within the individual grain / subgrain. Then, grain / subgrain-stored energy map is generated to represent the spatial distribution of grain /subgrain stored energy with different levels of brightness. The area with high population density of misorientation, i.e. high density of dislocations, appears relatively bright while the area with low population density of misorientation appears relatively dark.
As revealed in Figure 3, two types of coarse grains, with and without subgrains, are present in the partially recrystallized 2A97-T3 alloy. As expected, the recrystallized grains exhibit relatively low stored energy and the unrecrystallized grains with subgrains possess relatively high stored energy. Interestingly, as shown in Figures 5 and 6, corrosion occurs preferentially in regions of relatively high stored energy, i.e. the unrecrystallized grains; the boundaries surrounding the subgrains with high stored energy are more susceptible to corrosion. Further, as described in Section 3, Figure 6 (a) show that the grain stored energy plays a more decisive role in corrosion propagation along the subgrain boundaries compared to the presence of T1 phase precipitates at the boundaries. This is also evidenced by the feature shown in Figure 6 (d), i.e. corrosion developed unevenly into the two neighbouring subgrains and preferentially developed into the subgrain of higher stored energy. The regions with higher population density of dislocations are thermodynamically more unstable, namely more susceptible to corrosion. It has been revealed that dislocation density difference could result in potential difference around 20 mV in AA2024 aluminium alloy [23].
5 Conclusions

1. Localized corrosion occurs in 2A97-T3 Al-Cu-Li alloy during immersion in 3.5 wt. % NaCl solution, with the subgrain boundaries within unrecrystallized grains of the partially recrystallized alloy being preferentially attacked. The boundaries surrounding the subgrains with higher stored energy, i.e. high density of dislocation, are more susceptible to corrosion.

2. The local plastic deformation in the periphery of the subgrain boundaries plays a more decisive role in corrosion propagation along the boundaries compared to the presence of T$_1$ phase precipitates at the boundaries.

3. Compared with the grain boundaries decorated by T$_1$ phase precipitates, the grain boundaries decorated by T$_B$ phase precipitates displays relatively low susceptibility to corrosion due to its relatively low content of lithium.

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Figure 1: (a) SEM micrograph of the as-received 2A97-T3 Al-Cu-Li alloy, showing needle-shaped precipitates along a grain boundary; (b) bright field TEM micrograph of a grain boundary with the needle-shaped precipitates; (c) lithium EELS map of the framed area in (b); (d) lattice image of the precipitate along [001] zone axis.
Figure 2: (a) SEM micrograph of the as-received 2A97-T3 Al-Cu-Li alloy, revealing a short needle-shaped precipitate at a grain boundary; (b) bright field TEM micrograph of a grain boundary with short needle-shaped precipitates; (c) copper EELS map of the area shown in (b); (d) lithium EELS map of the area shown in (b); (e) lattice image of the short needle-shaped precipitate along [01\overline{1}] zone axis.
Figure 3: EBSD maps of the 2A97-T3 Al-Cu-Li alloy: (a) grain orientation distribution in Euler’s colour; (b) stored energy distribution of the grains; (c) stored energy distribution of the subgrains.
Figure 4: Scanning electron micrographs of localized corrosion sites in the 2A97-T3 Al-Cu-Li alloy after immersion in a 3.5 wt.% NaCl solution for 5 hours: (a) surface plan-view; (b) cross sectional view; (c) the framed area A in (b) at increased magnification; (d) the framed area B in (b) at increased magnification.
Figure 5: Scanning electron micrograph and EBSD maps of localized corrosion site on the surface of the 2A97-T3 Al-Cu-Li alloy after immersion in a 3.5 wt.% NaCl solution for 5 hours: (a) surface plan-view of a tested specimen without plasma etching; (b) surface plan-
view of a tested specimen with plasma etching; (c) surface plan-view of a localized corrosion site on the tested specimen with plasma etching; (d) grain orientation distribution in Euler’s colour (the area shown in (c) is indicated by the dashed line frame in (d)); (e) stored energy distribution of the grains shown in (d); (f) stored energy distribution of the subgrains shown in (d).
Figure 6: Transmission electron micrographs and TKD maps of a localized corrosion site in the 2A97-T3 Al-Cu-Li alloy after immersion in a 3.5 wt.% NaCl solution for 5 hours: (a) HAADF micrograph of the cross section; (b) the corresponding grain orientation distribution in Euler’s colour; (c) stored energy distribution of subgrains; (d) bright field TEM micrograph of framed area 1 in (a) at increased magnification; (e) HAADF micrograph of framed area 2 in (a) at increased magnification.