



Mechanism of low exfoliation corrosion resistance due to slow quenching in high strength aluminium alloy



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ABSTRACT

Electrochemical impedance spectroscopy and accelerated immersion testing results indicate that a lower quench rate leads to lower exfoliation corrosion resistance in 7055 aluminium alloy. The mechanism was discussed based on the change in grain boundaries microstructure and microchemistry due to slow quenching. Because of the increase of Zn and Mg content, grain boundary particles in the slowly-quenched specimens are more electrochemically active and tend to accelerate corrosion despite their large size and spacing; moreover the wide precipitates free zones depleted of solutes near grain boundaries tend to be more active as well and accelerate corrosion along grain boundaries.

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1. Introduction

Al–Zn–Mg–Cu aluminium alloys have been widely used as aeronautical structural materials due to their high specific strength. However, these alloys are often susceptible to localized corrosion generally along grain boundaries (GBs), such as intergranular corrosion, exfoliation corrosion and stress corrosion cracking [1–6]. Exfoliation corrosion (EXCO) predominantly appears in the rolled and extruded semi-products with elongated grains [7,8], and often results in premature failure of these products [9]. Therefore, a good compromise between high strength and high corrosion resistance is always desirable for these alloys. The strength of Al–Zn–Mg–Cu aluminium alloys is primarily dependent on the size and volume fraction of GP zones and η' precipitates in the matrix after aging [10–12]; while the localized corrosion resistance is primarily associated with GBs microstructure and microchemistry [13–16].

A number of investigations have been carried out on the effect of GBs microstructure and microchemistry on localized corrosion of the alloys subjected to different aging treatments. It is known that η phase at grain boundaries is anodic with respect to the alloy matrix, and thus tends to be preferentially attacked in corrosive solution [3,17]. As a result, a continuous layer of η phase at grain boundaries often gives rise to low corrosion resistance of T6 peak-aged alloys; after T7X over-aging, η phase particles become

larger and more-spaced, therefore can improve corrosion resistance [6,15,18–21]. Simultaneously, the chemical compositions in grain boundary particles (GBPs) can also be changed, and the increase of Cu content tends to enhance corrosion resistance effectively [13,22–26]. Moreover, it was found precipitates free zones (PFZs) adjacent to grain boundaries may play an important role. Wide PFZs may increase the susceptibility to intergranular corrosion [27]; depletion of Cu within PFZs was believed to be the primary reason for the increased resistance to stress corrosion cracking of 7075 alloys after T73 and retrogression and re-aging treatments [28].

Apart from aging, quenching is another important factor that has great effect on the GBs microstructure and microchemistry, and thus on corrosion resistance. For alloys with a high Cu concentration, the decrease of quench rate often leads to lower resistance to intergranular corrosion, exfoliation corrosion and stress corrosion cracking [3,27,29–31]; but for alloys with a low Cu concentration, the decrease of quench rate seems to increase resistance to stress corrosion cracking [25,32]. For a 7085 aluminium alloy forging, it was shown that the content of Zn and Cu in GBPs decreased with the decrease of quench rate, and the low content of Cu in GBPs was supposed to be the reason for the worst resistance to stress corrosion cracking of the slowly quenched specimens [31]. For an Al–Zn–Mg–Cu alloy thick plate, the content of Cu in GBPs was also found to decrease with the decrease of quench rate, which possibly contributes to lower corrosion resistance [30]. For a 7050 aluminium alloy sheet, however, the Cu content in GBPs was found to increase with the increase of quench delayed time, which was supposed to be the reason for the improvement of corrosion resistance [33]. For a 76 mm plate of 7079 aluminium alloy, the content of Cu

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in GBPs was higher in the mid-thickness position than the T/6 position because of a lower quench rate, and this was believed to be the main reason for higher resistance to stress corrosion cracking [24]. From these investigations, it is still controversial for the effect of quench rate on the chemical compositions of GBPs, and little attention has been paid to the effect of PFZs despite a significant increase in the width after slow quenching [31,34]. So the mechanism of low corrosion resistance of the alloys with a high Cu concentration due to slow quenching still needs to be explored. This problem is very important for very thick plates or heavy forgings, because quench rate in the center layer in these products is often low due to the large section or requirement of controlling residual stress [35,36].

7055 aluminium alloy is a typical Al–Zn–Mg–Cu alloy with a high Cu concentration from 2.0 to 2.6 (wt.%). The resistance to intergranular corrosion and exfoliation corrosion was found to decrease with the decrease of quench rate [30,37]. In this work, an attempt has been made to further understand the mechanism of low resistance to exfoliation corrosion due to slow quenching based on GBs microstructure characterization by transmission electron microscopy (TEM) and GBs microchemistry examination by scanning transmission electron microscopy (STEM).

2. Experimental

2.1. Materials and heat treatment

Specimens with a size of 125 mm in length \times 25 mm in width \times 25 mm in thickness were cut from a hot-rolled thick plate of 7055 aluminium alloy (3.58 Zn, 2.49 Mg, 1.01 Cu, 0.035 Zr and balance Al, in at.%). Small holes were drilled at different positions in the specimen and inserted thermocouples to record time–temperature curves during end quenching test. After solution heat treating at 470 °C for 1 h in an air furnace, the specimens were taken out rapidly, placed in a fixture and cooled to ambient temperature by exposing at one end to a vertical stream of cold water. Based on the recorded time–temperature curves, the quench rate was estimated from about 138 °C/min to 1250 °C/min over the length of the end-quenched specimen [34]. After end quenching the specimens were aged at 120 °C for 24 h in an oil bath immediately. Fig. 1 shows typical optical micrographs at different positions in the end-quenched and aged specimen. The partial-recrystallized microstructure shown in Fig. 1(a) is quite typical in high strength Al–Zn–Mg–Cu alloys plates. The decrease of quench rate did not change grain structure but led to the precipitation of a number of large quench-induced η phase particles, which were corroded during specimen etching [34,38], therefore it was difficult to reveal the grain structure clearly (Fig. 1(b)). The unrecrystallized grains and most recrystallized grains were elongated along rolling direction, and this provided a condition for the occurrence of exfoliation corrosion [7,8,39,40]. In the unrecrystallized regions there were a number of subgrains. The large black particles in Fig. 1(a) were identified to be $\text{Al}_7\text{Cu}_2\text{Fe}$ and S phase [34], which often appear in Al–Zn–Mg–Cu alloys. In general, these particles receive little effect from quench rate and have minor effect on exfoliation corrosion [41], and thus will not be discussed hereafter.

2.2. Corrosion tests

Electrochemical impedance spectroscopy (EIS) test and accelerated immersion test are useful for evaluating the resistance to exfoliation corrosion of aluminium alloys [4,15,42,43], and they were also used in this work.

Some slices with thickness of 2 mm were cut at different positions with the corresponding quench rates of 1250 °C/min, 630 °C/min, 164 °C/min and 138 °C/min, respectively, in the end-quenched and aged specimens for EIS test using instrument IM6ex.

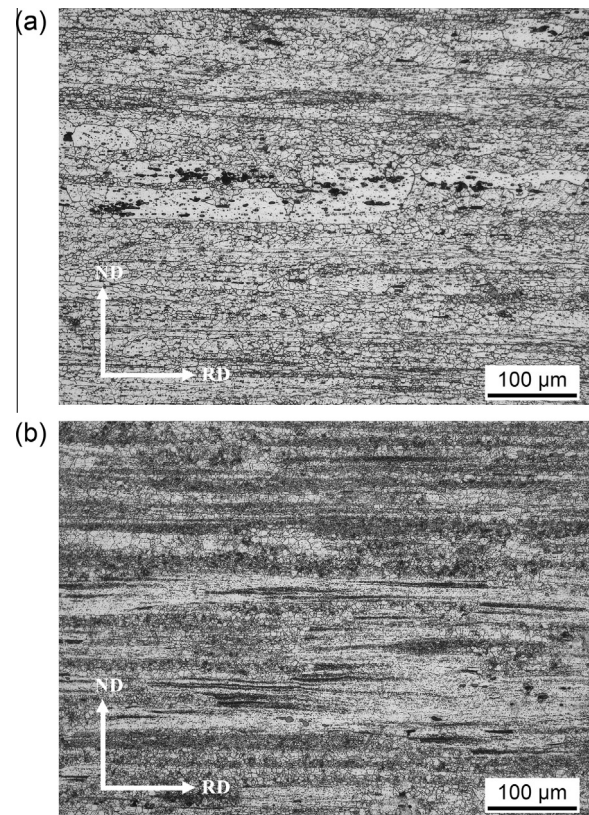


Fig. 1. Optical micrographs of the specimens cooled at (a) 1250 °C/min (b) 138 °C/min, RD: rolling direction, ND: normal direction.

The slices were degreased using 10% NaOH solution, pickled with 30% HNO_3 solution, rinsed using acetone and distilled water, and then dried in air. A three-electrode cell with a platinum counter electrode and a saturated calomel reference electrode (SCE) were used for EIS test. All tests were performed at room temperature in a solution containing 4.0 M NaCl, 0.5 M KNO_3 and 0.1 M HNO_3 , which was the same as that for the accelerated immersion test. The tests were performed at the open circuit potential with the frequency ranging from 0.01 Hz to 100 kHz using a 10 mV AC signal. The exposed surface area was 1 cm^2 .

Some samples with a size of 10 cm \times 2 cm were cut from the center layer of the end-quenched and aged specimens for accelerated exfoliation test according to GB/T 22639-2008 specification [44]. They were immersed in the solution containing 4.0 M NaCl, 0.5 M KNO_3 and 0.1 M HNO_3 for 48 h, and the solution temperature was maintained at 25 ± 2 °C in a thermostat. The exposed area was 20 cm^2 , and the ratio of solution volume to metal surface was 25 mL/ cm^2 . The EXCO rating was determined by visual examination to be pitting (P), superficial exfoliation corrosion (EA), moderate exfoliation corrosion (EB), severe exfoliation corrosion (EC), and very severe exfoliation corrosion (ED) according to GB/T 22639-2008 specification [44]. The surface morphology after immersion was examined by a Quanta-200 scanning electron microscopy (SEM). Moreover, cross-section samples were also cut and prepared by standard method for metallographic examination to observe corrosion morphology and to measure corrosion depth on a Leica DM IL LED optical microscopy.

2.3. Grain boundary microstructure and microchemistry examination

Thin foils at different locations in the end-quenched and aged specimen were cut, thinned carefully to be 0.08 mm in thickness, punched into 3 mm in diameter and then electropolished in a solu-

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