Effect of retrogression re-aging treatment on corrosion behavior of 7055 Al-Zn-Mg alloy

Fuqiang Guo∗, Shuwei Duan†, Dongting Wu‡, Kenji Matsuda‡, Tao Wang∗† and Yong Zou∗∗

1 School of Materials Science and Engineering, Shandong University, Jinan 250061, People’s Republic of China
2 Key Laboratory of Liquid-Solid Structural Evolution and Processing of Materials, Ministry of Education, Shandong University, Jinan, 250061, People’s Republic of China
3 Faculty of Sustainable Design, University of Toyama, Toyama 930-8555, Japan
4 Shandong Engineering & Technology Research Center for Modern Welding, Shandong University, Jinan, 250061, People’s Republic of China

∗ Authors to whom any correspondence should be addressed.
E-mail: wxm689wxm@sdu.edu.cn and yzou@sdu.edu.cn

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Abstract
The effect of retrogression re-aging treatment (RRA) on the corrosion behavior of 7055 Al-Zn-Mg alloy was studied in this paper. Results provided that corrosion resistance could be greatly improved by RRA. After the RRA treatment, the isolated precipitates occurred on grain boundaries (GBs) and the low angle grain boundaries (LAGBs) presented a larger fraction compared to that of single-stage peak aging treatment. The samples after RRA treatment also show better corrosion resistance than that of single-stage peak aging treatment. In addition, of the electrochemical impedance spectroscopy (EIS) analysis showed that the impedance spectrum was consisted of semi-infinite layer diffusion impedance and stagnant Weber impedance. The semi-infinite layer diffusion impedance corresponded to a limited retention layer on the electrode surface while the Weber impedance represented a stagnant layer of corrosion products generated by the anode branches. The aluminum alloy after RRA treatment achieved higher Rf and low Cf,Cp values and its corrosion current density was ten times smaller compared to the single-stage peak aging samples with the 10% hardness losing.

1. Introduction
In the automotive and aerospace industries, 10% reduction in weight will bring an 8% increase for the fuel economy. Therefore, the structural components of lightweight aluminum alloy have been the preferred materials for transportation industry [1]. Among them, the Al-Zn-Mg alloy is widely used in the field of aerospace due to its high-strength, low density, high hardness and good machining property.

Up to now, various heat treatment methods have been studied to enhance the performance of the aluminum alloy, for instance single-stage aging (T6), double-stage aging and retrogression re-aging treatment. The single-stage aging treatment is performed at a specific temperature for a certain period where the selection of the temperature and the aging time is crucial for the microstructure and properties of the aluminum alloy. The precipitation behavior of the strengthening phases for the Al-Mg-Zn aluminum alloy can be described in the following sequence: Supersaturated solid solution (SSSS) → Guinier → Preston (G.P) zones Metastable $\eta'$ → Stable $\eta$ (MgZn2) [2]. As for the 7 series aluminum alloy, T6 treatment is more efficient for improving the strength compared to the other heat treatment processes. This is due to that the density of the precipitates is promoted inside the matrix after T6 treatment. However, after peak aging, the coarser chain-like precipitates, continuously distributing along the grain boundary are very sensitive to stress corrosion cracking (SCC) and exfoliation corrosion. Retrogression re-aging treatment (RRA) can improve the SCC resistance of the alloy with slightly sacrificing the strength or plasticity. This is attributed to the discontinuous precipitates along the grain boundaries. The influence of heterogeneous precipitation is complicated for the 7xxx alloy, metastable G.P zones plays an important role in precipitation kinetics [3]; $\eta'$ and $\eta$ phase (MgZn2) precipitate inside the grain
interior and along the grain boundary simultaneously, which enables the alloys after RRA treatment obtain high corrosion resistance and mechanical strength [4]. It has been suggested that in order to achieve a good balance between the strength and corrosion resistance, the G.P zones should be sufficiently formed during the pre-aging stage while the $\eta'$ phase should dissolve during the retrogression stage and then precipitate during the re-aging stage. In addition, enough time is necessary for Cu to diffuse from the matrix to the precipitates [5].

A number of researchers focused on improving the performance of the 7xxx aluminum alloys, especially in terms of strength and corrosion resistance through the heat treatment of. Chen et al [6] found that high the 7055 alloy developed superior strength, toughness and ductility after the two-stage solution treatment. It is suggested that the peak aging treatment could improve the alloy strength, but the continuous $\eta$ phase along the grain boundaries became the susceptible anode channel, and induced inter-granular corrosion [7]. Lin et al [8] argued that the alloys treated by RRA process showed higher tensile strength compared to that treated by the peak aging and over-aging treatments. However, the alloys after peak aging treatment demonstrated the highest resistance to the stress corrosion t. Zhang et al [9] highlighted that the 3D morphology and cross-sectional morphology after exfoliation corrosion for the peak-aged AA 7075 were obviously different under various solid solution conditions and a large number of corrosion pits appeared on the surface. It can be seen that the corrosion resistance of 7 series alloys can be greatly improved through changing the single-stage peak aging (T6) treatment to the retrogression re-aging (RRA) treatment. Therefore, the RRA treatment is of great importance to improve the corrosion resistance of 7 series aluminum alloys without significant loss of the strength.

Moreover, some researchers found that the grain characteristics also affected the corrosion resistance of the aluminum alloy. Amjad Saleh El-Amoush et al [10] discovered that the grain coarsening at single-stage aging could reduce the breakdown potential in the degassed NaCl solution while Huang et al [11] found that the corrosion morphology was closely related to the grain size of the T6 treated aluminum alloy. Accordingly, it is also very necessary to understand the influence of heat treatment process on the properties of 7xxx aluminum alloy.

However, the present researches mainly focused on the balance between high strength and corrosion resistance of the 7055 alloy. It is still difficult to prepare aluminum alloys with both high strength and high corrosion resistance property [12]. In this research, the influence of retrogression re-aging treatment and single-stage aging treatment on the microstructure and corrosion behavior of 7055 alloy were systematically studied, and the corrosion mechanism was further discussed.

2. Materials and methods

a. Materials and heat treatment

The experimental material was commercially rolled 7055 aluminum alloy. The chemical composition is shown in table 1.

The samples was cut by electric spark from the rolled plate. For both the single-stage aging and retrogression re-aging process, the samples were first treated by the solution treatment at 485 °C for 8 h, followed by water quench in room temperature. Then the aging treatments were carried out. The aging parameters are listed in table 2 and table 3 respectively:

b. Hardness test

The Vickers hardness was conducted on a DHV-1000 digital hardness tester under a load of 100 g and a holding time of 15 s.

c. Intergranular corrosion test

Intergranular corrosion tests were carried out for the samples with peak aging parameters and some retrogression re-aging parameters. After heat treatment, the samples with the same surface area were grounded to 3000 mesh sandpaper and soaked into the corrosive aqueous solution of 57 g l$^{-1}$ NaCl $+ 10$ ml l$^{-1}$ H$_2$O$_2$ (ISO 11846-1995) for 6 h [13]. Afterwards, the samples were ultrasonically cleaned with concentrated nitric acid. The surface morphology and roughness were analyzed by the confocal scanning microscope (LSM-800, Carl Zeiss). The extent of the intergranular corrosion for the samples with different heat treatments were determined by the corrosion depth.

![](image1.png)

| Table 1. The chemical composition of the 7055Al. |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Element | Zn | Mg | Cu | Mn | Si | Ti | Fe | Zr | Al |
| wt.%  | 7.745 | 1.824 | 2.130 | 0.002 | 0.092 | 0.100 | 0.110 | 0.085 | Bal. |
d. Electrochemical test
The samples were sanded to 3000 meshes with sandpaper, wiped with alcohol, then electrochemical measurement, polarization scanning and electrochemical impedance spectroscopy were prepared in a conventional standard electrolytic cell. In the cell, the samples were the working electrode, a platinum foil as the auxiliary electrode, and a saturated calomel electrode (SCE) was the reference electrode. Electrolyte solution of 3.5% NaCl was used. The analysis was carried out with CHI660e electrochemical workstation of Shanghai Chenhua Instrument Co., Ltd, using a three-electrode system, the working face of the sample is 15 mm × 10 mm × 5 mm. Then, open circuit potential was determined by the open circuit potential-time curve test. The polarization curve was tested by potentiodynamic scanning method, and the scanning frequency of polarization curve was 0.001 V s⁻¹. Electrochemical impedance spectroscopy test used AC sine wave with amplitude of 10 mV as excitation signal and frequency range of 0.01 ~ 10⁵ Hz. ZView2 software was used to fit the impedance spectrum data.

| Table 2. Single-stage aging heat treatment parameters. |
|------------------------------------------------------|
| Sample number | Aging temperature | Aging time |
| D1 | 120 °C | 4 h |
| D2 | 120 °C | 8 h |
| D3 | 120 °C | 10 h |
| D4 | 120 °C | 12 h |
| D5 | 130 °C | 4 h |
| D6 | 130 °C | 8 h |
| D7 | 130 °C | 10 h |
| D8 | 130 °C | 12 h |
| D9 | 140 °C | 4 h |
| D10 | 140 °C | 8 h |
| D11 | 140 °C | 10 h |
| D12 | 140 °C | 12 h |

| Table 3. Retrogression re-aging heat treatment parameters. |
|-------------------------------------------------------------|
| Sample number | Pre-aging | Retrogression | Re-prescription |
| RRA1 | 120 °C/8 h | 170°C/30 min | 120 °C/24 h |
| RRA2 | 120°C/8 h | 170°C/1 h | 120°C/24 h |
| RRA3 | 120 °C/8 h | 170 °C/2 h | 120 °C/24 h |
| RRA4 | 120°C/8 h | 190°C/30 min | 120°C/24 h |
| RRA5 | 120 °C/8 h | 190°C/1 h | 120°C/24 h |
| RRA6 | 120°C/8 h | 190°C/2 h | 120°C/24 h |
| RRA7 | 120°C/24 h | 190°C/30 min | 120°C/24 h |
| RRA8 | 120°C/24 h | 190°C/1 h | 120°C/24 h |
| RRA9 | 120°C/24 h | 190°C/2 h | 120°C/24 h |

e. X-ray diffraction (XRD)
X-ray diffraction (XRD) patterns were obtained by using an DX—2700 X-ray diffractometer at a scanning rate of 0.02° s⁻¹.

f. Electron backscattered diffraction (EBSD)
EBSD samples were electropolished at 4 °C–8 °C for 1 min at a voltage of 16 V, in a mixture of perchloric acid and ethanol (1:9 v/v) with agitation, using a magnetic stirrer, and the substrates and the copper plate was taken as the working and counter electrodes, respectively. We used a JSM-7800F scanning electron microscope (SEM) (Tokyo, Japan) and Oxford NordlysMax3 system to acquire EBSD data, and then processed them with Channel 5 commercial software (v5.12.67.0).
g. Transmission electron microscope (TEM)
The samples were mechanically thinned to 120 μm. Thin foils with diameter of 3 mm were punched from the slices and then electro-polished to 30 μm at -4°C–8°C for 1 min at a voltage of 16 V, in a mixture of perchloric acid and ethanol (1:9 v/v). Finally, the TEM specimens were prepared by twin-jet electropolishing in a solution of 30% HNO₃ in methanol at -25°C and 15 V. TEM micrographs, including selected area diffraction (SAD) and bright-field (BF) images were taken with EM-002B operated at 120 kV.

3. Results

h. Hardness and the polarization curves
Figure 1 shows microhardness for the samples treated by different parameters. Figure 1(a) gives the relationship between the hardness and the single-stage aging time while figure 1(b) indicates the relationship between the hardness and the time of RRA aging retrogression. It can be seen that D6 sample (130°C/8h) had the highest hardness, while samples of RRA6 (120°C/8h + 190°C/2h + 120°C/24h) achieved the lowest hardness.

i. The polarization curves
The potentiodynamic polarization curves for the single-stage aging treatment of D2, D6, D11 samples and the retrogression re-aging treatment of RRA2, RRA3, RRA4, RRA6, RRA7, RRA9 samples immersed in 3.5% NaCl solution are presented in figure 2. It is found that a current platform was formed for all the samples and the current platform of RRA samples was more obvious than T6 samples. The cathode branch was attributed to the hydrogen evolution reaction and the anode branch is related to the dissolution of the aluminum matrix which represented the transformation of aluminum ions [9, 14]. The corrosion current density is displayed in table 4. The low corrosion current density represent the high corrosion resistance. It can be seen that the corrosion resistance order of the samples was RRA 6 > RRA 9 > RRA 7 > RRA 4 > RRA 2 > RRA 3 > D 11 > D 2 > D 6. All T6 samples demonstrated poorer corrosion resistance than that of RRA samples. D6 sample showed the lowest corrosion resistance while RRA6 sample showed the highest corrosion resistance. Based on the corrosion current density, the corrosion resistance order of the RRA samples was RRA 2 > RRA 3, RRA...
6 > RRA 4, RRA 9 > RRA 7. Through the electrochemical tests, RRA 2, 6, 9 samples with good corrosion resistance were selected for the subsequent study.

j. Intergranular corrosion
The influence of intergranular corrosion on surface roughness was analyzed by the laser confocal microscopy. Figure 3 displays the typical morphologies of the corroded-surface for the single-stage aged D2, D6, D11 and the retrogression re-aged RRA2, RRA6, RRA9. Figure 4 depicts the surface roughness observation. As presented in figure 3 and figure 4, intergranular and pitting corrosion occurred in all samples. However, the pitting corrosion for the samples treated by the retrogression re-aging process was less significant that the samples of T6 state were more susceptible to the pitting corrosion. As presented in figure 3, large pitting pits and deep intergranular corrosion appeared on the D6 sample, it showed that the D6 sample has the lower corrosion resistance than samples treated by other treatments. Retrogression re-aging samples showed better corrosion resistance and only slight pitting attacks were apperceived on their surface.

As presented in figure 5, the average arithmetic height of D6 samples is 1.70μm, and this value is 0.975μm of RRA6 samples, which indicates that the average roughness of corrosion samples after RRA6 aging is lower than the d6 samples. The average root mean square height of the D6 sample is 2.98μm, and the average root mean square height of the RRA6 samples is 1.86μm, which indicates that the standard deviation of the average height of the RRA6 corrosion samples is lower than that of the D6 corrosion samples, so the RRA6 samples have higher corrosion resistance than other samples. According to the study of grain size, grain coarsening of seven-series aluminum alloy results in the decreasing corrosion resistance [10]. However, in general, the corrosion depth of RRA samples is shallower than that of T6 samples. As indicated in the table 5, only D6 sample has an intergranular corrosion level of 3, which proves the D6 sample has the lower corrosion resistance than the samples treated by other treatments.

### Table 4. Corrosion potential and corrosion current density.

| Sample number | corrosion current density (A/cm²) |
|---------------|----------------------------------|
| D2            | 3.04 E-6                         |
| D6            | 5.48 E-6                         |
| D11           | 2.58 E-6                         |
| RRA 2         | 1.62 E-6                         |
| RRA 3         | 3.08 E-6                         |
| RRA 4         | 8.08 E-7                         |
| RRA 6         | 4.28 E-7                         |
| RRA 7         | 6.98 E-7                         |
| RRA 9         | 6.59E-7                          |

Figure 3. Corrosion metallographic photograph: (a) the D2 sample; (b) the D6 sample; (c) the D11 sample; (d) the RRA2 sample; (e) the RRA6 sample; (f) the RRA9 sample.
k. The Impedance spectrum

Figure 6 shows the electrochemical impedance spectroscopy plots of samples in 3.5 wt% NaCl aqueous solution. It can be seen that the impedance spectrum conforms to semi-infinite diffusion type. The low frequency band of the impedance spectrum develops the trend of a straight line, and the high frequency band arc can be understood.
as electrochemical polarization capacitance. The straight line is electrode reaction controlled by diffusion. During electrochemical corrosion process, the corrosion products were generated in anode branch, which affects the flowing of corrosion liquid, and formed a stagnant layer with a thin thickness. As a result, the low frequency band of the impedance spectrum has tendency of a straight line and a semi-infinite diffusion. An equivalent circuit diagram of the corresponding to impedance spectrum data is presented in figure 6. The resistance and capacitance are changed until the predicted alternate current behavior is as close as possible to the corrosion experimental data. The physical meaning of equivalent circuit elements is as follows: Rs is the ohmic resistance of electrolyte, Rf is the film resistance, Cf is the film capacitance, Rp is the hole resistance of film, Cp is the capacitance of aluminum alloy double layer, Rp is the polarization resistance of electrode, and W is Weber impedance. In this equivalent circuit, all capacitors are modeled by constant phase element (CPE) to obtain better simulation results between the model and experimental data. Then it defined by the following equation:

$$Z_{\text{CPE}} = \frac{Z_0}{(j\omega)^n}$$

Where n can be between 0 and 1 (for a perfect capacitor) and Z0 is a constant [15, 16]. The electrochemical impedance data of its equivalent circuit elements are displayed in table 6. It is found that all samples have similar Rs values. Due to the dissolution of aluminum and the formation of protective oxide, all samples have larger Rf values, indicating that the corrosion process is slowly, and the increasing of oxide layer thickness is attributed to the decreasing of interfacial capacitance [17]. The D2 sample has larger Rf resistance value and smaller Cp capacitance value than other samples, which indicates the oxide layer has the larger thickness and higher corrosion resistance than the D6 and D11 samples. The Rf resistance value of all RRA samples is larger than that of T6 samples, and the Cf capacitance is smaller than that of T6 samples, indicating that the RRA samples have larger oxide layer thickness and higher corrosion resistance than that of T6 samples. The system involves film formation, so the systems are not easy to polarization and have good corrosion resistance due to the high Rs, Rp and low Cf, Cp. Similarly, the D6 sample is more vulnerable to corrode and has the lower corrosion resistance than other samples. RRA6 sample has the high Rf and low Cf, Cp values, so it has better polarization and corrosion resistance than other samples. This conclusion is consistent with the above conclusions of corrosion current density and roughness test. From the above results, it can be seen that the T6 and RRA samples have the

Table 6. The electrochemical impedance data of its equivalent circuit elements.

| Elements | D2      | D6      | D11     | RRA2    | RRA6    | RRA9    |
|----------|---------|---------|---------|---------|---------|---------|
| Rs (Ωcm²)| 28.11   | 10      | 32.74   | 28.73   | 28.09   | 26.9    |
| Rf (Ωcm²)| 3616    | 49.85   | 121.2   | 8760    | 10410   | 7241    |
| Rp (Ωcm²)| 3.107E4 | 4.985E4 | 1.212E4 | 8.760E4 | 1.041E5 | 7.241E4 |
| Cf (μF cm⁻²)| 2.315E-5| 8.166E-5| 1.474E-5| 1.818E-5| 1.474E-5| 2.396E-5|
| n       | 0.803   | 0.4447  | 0.5424  | 0.8347  | 0.8433  | 0.7824  |
| Cp (μF cm⁻²)| 1.546E-4| 4.438E-4| 3.489E-4| 1.158E-4| 8.929E-4| 1.534E-4|
| n       | 0.714   | 0.9341  | 0.8704  | 1.0993  | 0.8745  |
| W(S·sec⁻⁵·cm⁻²)| 2.313E5 | 634.1   | 0.007848| 2.48E4 | 1.4685E4| 1.252E6|
same corrosion mechanism, but the samples show obvious difference in impedance spectrum due to the change of surface corrosion oxide thickness caused by the differences of precipitation quantity and distribution.

4. Discussion

4.1 Electron backscattered diffraction

The average grain size was measured from EBSD grain-boundary photograph (figure 7). It can be seen that the samples treated by T6 or RRA treatment have similar average grain size. The nature of the grain boundaries in the alloy is illustrated in figure 8 by means of the disorientation distribution histogram. The disorientation distribution histogram was made from EBSD grain-boundary photograph taking into account boundaries with misorientations more than 2 deg. Low angle grain boundaries (LAGB) in 7055 alloy refers to the grain boundary where the misorientations of two adjacent grains are less than 15°. High angle grain boundaries (HAGB) in 7055 alloy refers to the grain boundary where the misorientations of two adjacent grains are more than 15°. The LAGBs have lower energy than the HAGBs, so the solute atoms experience more difficulty in diffusing along LAGBs than that along HAGBs. It can be seen that the percentages of the LAGBs after T6 and RRA treatment are 32.461% and 42.893% respectively, thus the solute atoms experience less difficulty in diffusing along GBs of the T6 samples that of the RRA samples, and the T6 samples are easy to generate continuous distributed high density precipitates on GBs, which offers a path for inter-granular corrosion, the instability grain boundaries with high energy are very sensitive to the corrosion.

4.2 Microstructure

The precipitates of samples were studied using the TEM observation. The typical bright field TEM micrographs and the selected area diffractions of the 7055 alloy under different aging treatments were shown in figure 9. In T6 treatment samples, TEM revealed a high density of fine precipitates dispersed relatively uniformly throughout the matrix (figure 9(b)). The T6 samples have many $\eta'$ phases, $\eta$ phases and some G.P zones in the matrix (figure 9(b)), however the RRA samples have many $\eta'$ phases and $\eta$ phases in the matrix (figure 9(d)). The T6
samples have finer precipitates than the RRA samples, so the T6 samples show better mechanical properties than the RRA samples.

The XRD patterns of the samples treated by different heat treatment parameters are presented in figure 10. The precipitated phases were detected for the single-stage aging D11 and retrogression re-aging RRA 9 treatment. Phase precipitation could contributed the precipitation strengthening, but it also plays the anodic role compared to the matrix during corrosion. As a result, the degree of the corrosion or the hydrogen embrittlement of the alloy was increased [18]. Moreover, it has been confirmed that the grain size and orientation, grain boundary precipitation and dislocation also influences the corrosion behavior of the aluminum alloy [19]. The precipitated phases had lower Ecorr but higher Icorr than the matrix phases [20]. This decreased corrosion resistance.

The samples experiencing T6 and RRA treatment showed different precipitation distribution. A high density of the precipitated phases are continuous distributed along the GBs under T6 treatment (figure 9(a)). After RRA treatment (figure 9(c)), the precipitates distributed discontinuously and became isolated along the GBs and their sizes were larger than that of the T6 treatment. After T6 treatment, the high-density precipitated phases, continuously distributing on GBs served as the main corrosion paths. As for the RRA samples, the isolated precipitates on GBs could interrupt the path continuity (figure 9(c)) and therefore the corrosion susceptibility was eliminated or reduced.

5. Conclusions

(1) The corrosion current density of the RRA 6 (120°C/8h + 190°C/2h + 240°C/24h) treatment samples is ten times less than the single-stage peak aging samples with the hardness losing 10%.
The corrosion products are generated by the anode branches to form a stagnant layer in corrosion process, and the stagnant layer exhibits Weber impedance. The samples after retrogression re-aging treatment have high $R_f$, $R_p$ values and low $C_f$, $C_p$ values, so RRA samples show better corrosion resistance than T6 samples.

(3) The RRA samples showed the isolated precipitates on GBs and have more contents of the LAGBs than the T6 samples, so the susceptibility to corrosion of RRA samples is reduced.

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ORCID iDs

Fuqiang Guo https://orcid.org/0000-0003-1317-6948
Shuwei Duan https://orcid.org/0000-0002-5413-0076
Dongting Wu https://orcid.org/0000-0002-3463-2888

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Figure 10. XRD pattern of the samples with different heat treatments.
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