The Effect of Quench Delay on Structure and Corrosion Properties of Aluminum Lithium Alloy 2091

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Abstract. The aerospace industry is always trying to improve aircraft performance and reduce aircraft weight. Therefore aluminum lithium alloys are targeted as advanced materials for aerospace technology because of their low density, good corrosion resistance, high toughness and lightweight. This 2091 alloy is an aluminum-lithium alloy which has a composition 2.1 wt% Cu, 2.0 wt% Li dan 0.1 wt% Zr. This alloy has been subjected to quench delay heat treatment to enhance corrosion resistance. The experiments were carried out to determine the relationship between delay quenching and its corrosion properties. Aluminum-lithium 2091 had solutionized at 525°C for 6 hours and then has been quenched in water with a variation of delay quenching (30 second, 60 second, 90 second). Characterization using x-ray diffraction were used to investigate the phase of each sample. The results show different diffraction patterns from one sample to another due to delay quenching. While electrochemical testing using a 3.5% NaCl solution show different corrosion resistance due different quench delay.

1. Introduction
Aluminum-lithium alloy is targeted as advanced materials for aerospace technology because it has a low density, high toughness, good corrosion resistance and lightweight. The aerospace industry is always trying to reduce aircraft weight to save on fuel costs and improve aircraft performance. Al-Li 2091 alloy is one way to save aircraft weight, because every 1% by weight of lithium added to aluminum can reduce density by 3% and increase elastic modulus [1]. The 2091 lithium aluminum alloy has a 7% elastic modulus and the microstructure is determined by the thickness of the material. If the material thickness is > 3.5mm has non recrystallized microstructure, while if < 3.5mm or thin sheet material has an elongated recrystallized grain structure [2].

The mechanical properties of 2091 alloy are affected by the microstructure and the rate of material cooling. If the greater the degree of non recrystallization, the more uniform the exfoliation corrosion. The more fibrous the structure, the higher the stress corrosion cracking threshold value. The melting range for Al-Li alloys is between 560 - 670°C [1]. Aluminum-lithium 2091 had solutionized at 525°C for 6 hours. The purpose of the solutionize procedure is to simply put into solution the maximum amount of alloying elements that are present in that particular alloy. This solutionized process aims to help the process of precipitation hardening where after the heat treatment is continued with quenching.

Quench delay is a heat treatment process where the process is carried out by cooling relatively quickly from austenitization temperature but given a cooling pause. Quenching media used is water. The purpose of the quenching process is for the hardening process, which is expected to obtain a large
price of violence, strength, and toughness but with minimal residual stress, distortion, and cracking. This experiment uses quench delay to study the effect of cooling rate. If the cooling rate is too fast it will cause distortion, but if the cooling rate is too slow, undesirable grain boundary precipitation will result[3,4]. Therefore, this study wants to investigate the most optimal cooling conditions in order to obtain minimal distortion and no corrosion.

2. Experimental Procedure

2.1. Sample Preparation
The material used in this study is aluminum lithium alloy 2091. The composition of this alloy is shown in table 1. There were 5 different samples in this experiment which is one sample of Al-Li 2091 alloy without quenching and heat treatment, and 4 samples of Al-Li 2091 alloy with heat treatment with different delay quench intervals (0 seconds, 30 seconds, 60 seconds, 90 seconds). Aluminum-lithium 2091 had solutionized at 525°C for 6 hours in a furnace and then have been quenched in water at room temperature with a variation delay quench. Before the heat treatment begins, the surface samples are cleaned using silicon carbide (SiC) paper grit-200. Heat treatment aims to make form a homogeneous solid solution that will eventually strengthen the aluminum-lithium alloy 2091.

All samples were analyzed at the UPP IPD Research Laboratory, Faculty of Mathematics and Natural Sciences, University of Indonesia. This x-ray diffraction aims to see the characterization of each Al-Li 2091 alloy treatment. The result of the XRD show different diffraction pattern from one sample to another due to delay quenching. For XRD analysis will be explained further in the part result and discussion.

| Element | Li | Cu | Zr | Mg | Fe | Si | Ti | Al |
|---------|----|----|----|----|----|----|----|----|
| Weight (%) | 1.9 | 1.85 | 0.07 | 1.2 | 0.07 | 0.02 | 0.02 | 94.72 |

2.2. Corrosion Test
In the corrosion test data were collected out using potentiodynamic polarization tests to estimate local corrosion of aluminum alloys. Five types of samples were used to evaluate corrosion resistance and the dimensions of the samples are 1.5 cm X 0.7 cm. Electrolytes used in corrosion test were 3.5% NaCl solution and 7% NaCl solution. The electrodes used to quantify the current and voltage are called the Working Electrode and the Reference Electrode. Three-electrode system was used to analyze the electrochemical response to the specimens. The three-electrode system was consists of an Ag/AgCl electrode as a reference electrode, the platinum sheet as an anauxiliary electrode and the Al-Li alloy as a working electrode[5].

Electrochemical test aims to investigate the corrosion rate of each sample and determine the effect of quench delay on the corrosion properties of aluminum lithium alloy. After data from Potentiostat is collected, the corrosion rate of each sample can be calculated using Faraday’s Law in Determination Corrosion Rates of Metals and Alloys[6].

\[
\text{Corrosion rate of alloy, } r = C \frac{M_i}{n_\rho} = \sum C \frac{M_i}{n_\rho A}
\]

Where \( C \) is corrosion rate constant (3.27 x 10^{-3} \text{ mm}^3/\text{year}), \( M \) is atomic weight of each element in the alloy (gr/mol), \( i \) is current density in (A/cm²) where the cm² defines the cross-sectional area exposed to NaCl, \( n \) is number of electrons involved in corrosion process, \( \rho \) is mass density of each element in the alloy (gr/cm³)[7].
3. Results and Discussion

3.1. XRD Analysis
In Figure 1, it can be seen the XRD plot results from five samples in this experiment and the data collected from the sample characterization results are presented in Table 2. In Figure 1, the diffraction pattern shows five peaks where the first peak has the highest intensity. For untreated Al-Li sample, five peaks were identified with angle positions of 38.024°, 44.137°, 64.880°, 77.763°, and 82.130°. For delay 0 second of quenching, five peaks were identified with angle positions of 37.805°, 44.574°, 64.662°, 77.763°, and 82.130°. For delay 30 second of quenching, five peaks were identified with angle positions of 38.024°, 44.137°, 64.880°, and 82.348°. For delay 60 second of quenching, five peaks were identified with angle positions of 38.024°, 44.356°, 64.662°, 77.981°, and 82.566°. For delay 90 second of quenching, five peaks were identified with angle positions of 37.805°, 44.137°, 64.880°, 78.199°, and 82.130°.

Figure 1 show the XRD plot results from five samples in this experiment. The graph shows that aluminum-lithium alloy 2091 with delay 90 second has the highest peak with the highest intensity. This means that at this peak aluminum content is in maximum condition. Although the diffraction pattern shows the difference of each peak is very small, but there is still a shift in the peak, where the shift in the peak affects the different microstructure of the crystal system that is seen in the difference in microstrain. From the result of XRD, shows that only one phase was detected from all samples, i.e aluminum. This is because the XRD is unable to detect phases below 5 wt%. Table 2, shows all samples have a cubic crystal structure, so the value of lattice parameters are the same for a, b, and c. Where each sample has a different cubic crystal structure (values a, b and c differ between samples). The sample with delay 0 seconds of delay quenching has the largest lattice parameters. The value of lattice parameters decreases if the delay quenching is getting longer, this is because the cooling time affects grain boundary precipitation[4,8]. Different lattice parameter values between samples indicate different crystal structure sizes so that the volume also changes. The volume of unit cell decreases if the delay time of quenching is getting longer.
Table 2. Crystallographic parameters of the aluminum lithium alloy 2091

| Sample                          | Al-Li without heat treatment | Al-Li Delay 0 | Al-Li Delay 30 | Al-Li Delay 60 | Al-Li Delay 90 |
|--------------------------------|------------------------------|---------------|----------------|----------------|----------------|
| First peak’s height (cps)      | 486.63                       | 1057.72       | 1129.24        | 1163.24        | 1420.84        |
| First peak’s d (Å)             | 2.3691                       | 2.3722        | 2.3690         | 2.3688         | 2.3668         |
| Lattice Parameter (Å)          |                              |               |                |                |                |
| a = 4.0454                     | a = 4.0479                   | a = 4.0431    | a = 4.0430     | a = 4.0429     |
| b = 4.0454                     | b = 4.0479                   | b = 4.0431    | b = 4.0430     | b = 4.0429     |
| c = 4.0454                     | c = 4.0479                   | c = 4.0431    | c = 4.0430     | c = 4.0429     |
| Volume (Å³)                    | 66,208                       | 66,327        | 66,09509       | 66,088         | 66,08422       |
| Density (g/cm³)                | 2.71                         | 2.7           | 2.71           | 2.71           | 2.71           |
| Concentration (%)              | 100                          | 100           | 100            | 100            | 100            |

3.2. Potentiodynamic Polarization Curves Analysis

Potentiodynamic polarization plots of untreated Al-Li and quenched Al-Li at 3.5% NaCl and 7% NaCl are shown in Figure 2 and Figure 3.

Figure 2. Potentiodynamic polarization plots of the aluminum lithium 2091 in 3.5 wt% NaCl

The results of the electrochemical test are shown in Table 3. Table 3 shows that the corrosion resistance of five Al-Li samples decreases when the chloride concentration results in anodic alloy surface dissolution. On the surface of the sample anodic dissolution occurs where aluminum-lithium is ionized and produces cations that come out into solution and then hydrolyze. Subsequently, the reaction of aluminum alloy with H2O as follows
The anodic reaction is:

\[ Al \rightarrow Al^{3+} + 3e^- \]  (2)

The cathodic reaction occurs on the alloys surface according to the reaction below:

\[ O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \]  (3)

Main reaction:

\[ 2Al^{3+} + 6Cl^- \rightarrow Al_2Cl_6 \]  (4)

\[ Al_2Cl_6 + 6H_2O \rightarrow Al_2(OH)_6 + 6HCl \]  (5)

**Table 3.** Potentiodynamic polarization result for untreated and quenched Al-Li 2091 in the 3.5 wt% and 7 wt% NaCl solution

| Wt% NaCl | Sample       | R (ohm)    | \(E_{\text{corrosion}}\) (V) | \(I_{\text{corrosion}}\) (A) | Corrosion Rate (mm/year) |
|----------|--------------|------------|-----------------------------|-----------------------------|-------------------------|
| 3.5      | Without heat treatment | 2.226 × 10^2 | -0.924                     | 1.154 × 10^4             | 1.279                  |
|          | Delay 0     | 3.144 × 10^2 | -0.920                     | 8.172 × 10^5             | 0.9055                 |
|          | Delay 30    | 4.019 × 10^2 | -0.876                     | 6.393 × 10^5             | 0.7084                 |
|          | Delay 60    | 5.136 × 10^2 | -0.840                     | 5.003 × 10^5             | 0.5543                 |
|          | Delay 90    | 4.131 × 10^2 | -0.825                     | 6.220 × 10^5             | 0.6892                 |
| 7.0      | Without heat treatment | 5.082 × 10^1 | -1.586                     | 5.055 × 10^4             | 5.601                  |
|          | Delay 0     | 1.866 × 10^2 | -1.495                     | 1.377 × 10^4             | 1.526                  |
|          | Delay 30    | 4.025 × 10^1 | -1.577                     | 6.383 × 10^4             | 7.072                  |
|          | Delay 60    | 6.808 × 10^1 | -1.582                     | 3.725 × 10^4             | 4.127                  |
|          | Delay 90    | 4.468 × 10^2 | -1.404                     | 5.750 × 10^5             | 0.6371                 |

Corrosion rate is influenced by potential corrosion \(E\) (V) and corrosion current \(I\) (A). Table 3 shows the greatest corrosion rate in aluminum lithium samples without heat treatment, where the sample has the most negative corrosion potential value than the other samples. This means that the more negative corrosion potential will cause low corrosion resistance. This is caused by the release of electrons to metals in anodic reactions which causes excess electrons in the metal to shift the potential of the metal in a more negative direction which results in slowing the anodic reaction and the acceleration of the cathodic reaction.

Table 3 also states the concentration of the solution can affect the rate of corrosion. It is seen that the highest corrosion rate is aluminum-lithium samples with 30 seconds of delay quench in the 7 wt% NaCl. Electrochemical test in 3.5% NaCl solution, samples with 60 seconds of quench delay have the best corrosion resistance, with a corrosion rate of 0.5543 mm/year. While in the electrochemical test in a solution of 7% NaCl, the sample that had the best corrosion resistance was a sample with a 90 second quench delay, with a corrosion rate of 0.6371 mm/year. This means that the right time to make aluminum-lithium has good corrosion resistance is around 60-90 seconds.
Figure 3. Potentiodynamic polarization plots of the aluminum lithium 2091 in 7 wt% NaCl

4. Conclusion
The corrosion rate is influenced by the time of quench delay, corrosion potential, and concentration of the electrolyte solution. Samples can get better corrosion resistance when subjected to cooling in air for 60-90 seconds and then water quenching process. Different lattice parameter values between samples indicate different crystal structure sizes so that the volume also changes. The volume of unit cell decreases if the delay time of quenching is getting longer.

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