HYDROGEN FUEL CELL & ENERGY STORAGE

The Effect of Pre-Stretching Treatment on the Microstructure, Mechanical Properties, and Corrosion Resistance of 7075 Aluminum Alloy used in PEMFC

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Abstract

Among various fuel cells, Proton Exchange Membrane Fuel Cells (PEMFC) have many advantages, such as low-temperature operation, fast starting, and high energy density. Therefore, the PEMFC can be widely applied in power generation, portable electric devices, and hybrid vehicles. Because of their high strength, low density, and corrosion resistance, heat-treatable aluminum alloys, such as 7075, are widely used in industry and for application to PEMFC. In addition to mechanical properties, corrosion resistance is also very important for working in an industrial environment. T6 heat treatment is recommended for this purpose, but this tempering treatment is sensitive to corrosion and Stress Corrosion Cracking (SCC). In this study, a 4% pre-stretch after solution was applied to samples to investigate its effect on SCC resistance. The results showed that with this treatment, the precipitates immigrate from the grain boundary to the interior of the grains and improve the SCC properties, which is very important for use in PEMFC. The results also show that a 4% pre-stretching after solution improves both tensile strength and elongation, and a tensile strength of 473 MPa with 12% elongation can be achieved by this heat treatment.

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1. Introduction

The Aluminum 7XXX alloy series (Al-Zn-Mg-Cu) introduced in 1943 are used in the majority of aircraft bodies, aerospace, lightweight equipment, and most recently, PEM fuel cells due to their good mechanical properties [1]. The AA7075 alloy is the most famous 7XXX alloy series, and the formation of precipitation during heat treatment gives remarkable strength to these alloys [2-5]. T6 heat treatment not only results in the greatest strength in this alloy but also decreases Stress Crack Corrosion (SCC) resistance. Using metallic plates for the PEMFC offers many advantages over conventional graphitic materials. These include relatively low cost, high strength, and ease of manufacture. A metallic plate can be easily shaped from a thin sheet into a complex shape, so the fuel cell power/volume ratio can be significantly improved. Some researchers have tried to increase Al alloy corrosion resistance by using a coating [6-8], but this treatment is expensive and sometimes a time waster. Other researchers [9] have tried to investigate the effect of pre-stretching on creep properties, but investigation of the SCC properties of this alloy has been minimal. The T7351 heat treatment, in which 1 to 5% stretching is performed after quenching, changes the microstructure so that the mechanical and SCC properties of AA 7075 improve compared to T73 and T6 treatment. Deschamps et al. [10] investigated the effect of pre-strain on 7000 aluminum series alloy and found that the precipitation rate increases rapidly at high temperatures. Rubaie and Barroso [11] reported increased strength after applying pre-stretching on samples. Huang et al. [12] reported that pre-heat treatment before final aging increases the amount of Cu and precipitation of η ($MgZn_2$) phase on grain boundaries, which increases resistance to SCC. Moreover, they showed that re-aging of 7075 aluminum alloy resulted in an increase in resistance to SCC in this alloy because of the dispersion of precipitation alongside grain boundaries.

2. Experimental Procedure:

2.1 Material Selection

The selected material was 7075 traditional Aluminum with 4mm thickness, commercially produced by the Alcoa Company. Quantometery analysis was used to determine the alloy's chemical composition, and the results are shown in Table 1.

Specimens were machined to a 200 mm length and 25 mm width in a rolling direction. The samples were then heat treated by T73 and T7351 cycles, as shown in Table 2. Before heat treatment, the samples were fully annealed (heated at 415˚C for 2 hours and then cooled in the furnace) to compare with heat-treated samples.

Table 1: The Composition of AA7075 Alloy

Element	Al	Si	Fe	Mn	Mg	Zn	Ti	Cr	Rem.
%	90.3	0.106	0.357	0.031	2.31	5.41	0.028	0.019	0.15

Table 2: Conditions of Different Heat Treatment Cycles for Al7075 Alloy

Heat Treatment	Condition of different heat treatment cycle
T6	$470°c / 8h$+quench in water + $120°c / 24h$
T73	$470°c / 8h$ + quench in water + $120°c / 24h$ Cooling in air + $175°c / 12h$
T7351	$470°c / 8h$ +quench in water + 5% stretching + $120°c / 24h$ Cooling in air + $175°c / 12h$

2.2 SCC Test

The standard number AStMG39-90 was used to investigate the sample's SCC resistance. A four-point method is recommended in this standard (Fig. 1). Samples based on this instruction are immersed in 3% NaCl solution, removed after a specific time, and maximum tensile strength is obtained after the test by formula below.

$$\sigma = 12ETY \ / (3H^2 - 4A^2) \qquad \text{Eq. (1)}$$

σ: Maximum tensile strength
E: Elasticity Modules
T: Thickness of Sample
Y: Maximum Deflection
H: Distance between points
A: Distance between outer and inner points

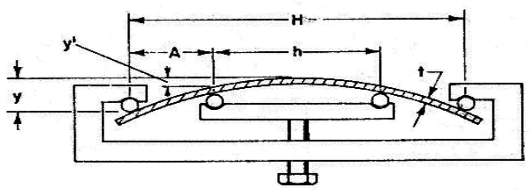
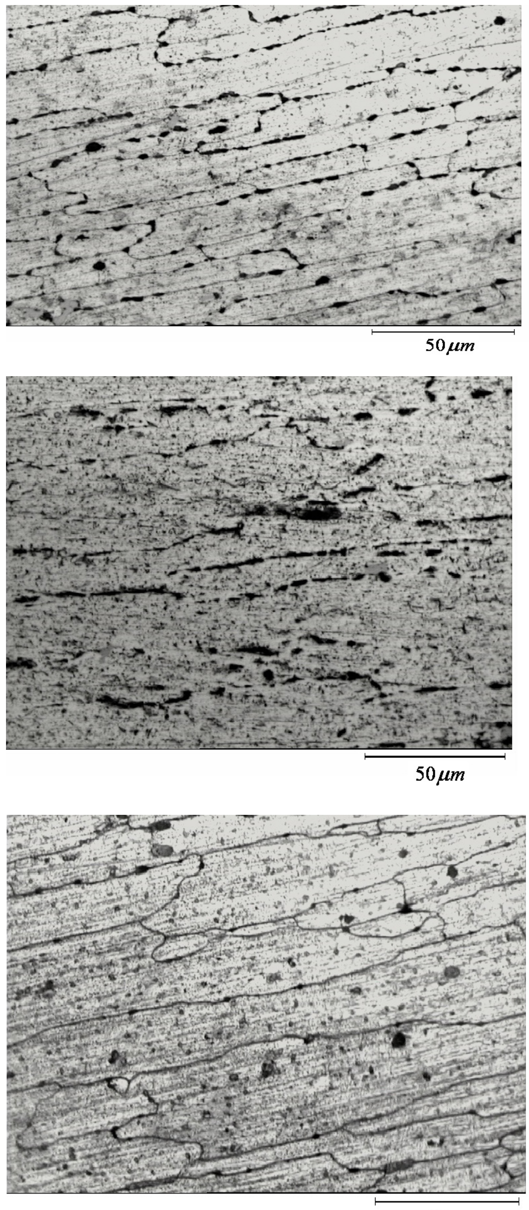


Fig .1. Schematic position of samples in a 4-point SCC test.

3. Results and Discussion

3.1. Optical Microstructure

To evaluate sample microstructures, the specimens before and after T73 and T7351 heat treatments were cut and assessed by Optical and Electron microscopy. The microstructure of full annealed samples (Fig. 2a) showed that dark precipitations are continuous and distributed along grain boundaries. After T73 heat treatment, some of these precipitates separate from each other and are distributed inside the grains (Fig. 2b). After the T7351 heat treatment, all the dark precipitates immigrated from the grain boundary into the inside of the grains (Fig. 2c). It seems that stretching after quenching in T7351 samples can produces enough energy and reduces barrier energy for nucleation, resulting in nucleation of precipitates inside the grains. Moreover, stretching samples in the T7351 treatment caused the distribution of more dislocation inside the grains, resulting in the nucleation of precipitates on dislocation in samples and the production of more homogenous nucleation inside the grains compared with T73 samples [13].



(a)
(b)
(c)

Fig. 2. Illustration of microstructure evolution in samples: a) Full Annealed, b) T73 heat treatment, and c) T7351 heat treatment. Etchant: HNO_3 25%

3.2. EDX Analysis

EDX analysis of the fully annealed T73 and T7351 samples showed that there are two kinds of precipitation, dark and grey; the chemical composition of these precipitations is presented in Table 3.

Table 3: EDX Analysis of Different Precipitates in the Sample under Full Annealing Cycle

	Dark precipitate		Bright precipitate	
Element	Weight percent	Atomic percent	Weight percent	Atomic percent
Mg	2/706	3/335	3/431	4/153
Al	77/879	87/605	81/720	89/122
Ti	1/243	0/777	0/000	0/000
Cr	1/873	1/079	0/049	0/028
Fe	1/807	0/970	0/000	0/000
Cu	3/512	1/649	2/669	1/236
Zn	3/902	4/539	12/131	5/546

In a fully annealed sample, dark precipitate is commonly deposited on grain boundaries, such as $Al_{23}CuFe$ or Al_2CuMg. With T73 heat treatment, dark precipitates separated together in the grain boundaries. As a result of the T7351 heat treatment, dark precipitates immigrated from the boundaries and are scattered uniformly inside the grains. The precipitates inside the grains are a mix of dark and grey precipitates. The dark ones may be Al_7CuFe or Al_2CuMg, and the lighter ones could be $MgZn_2$ or Al_2Cu [14].

3.3. AFM topography images for the T7351 sample

Fig. 3 shows AFM Photographs for the T7351 sample. The images show that there are two common precipitates, one small grey (400 to 500 nm) and the other dark (500 to 1000 nm), which are compatible with optical microscopy images. The small one seems to be η phase, which can be $MgZn_2$ and is normally semi-coherent. The bigger one seems to be η' phase and normally has incoherency with the matrix.

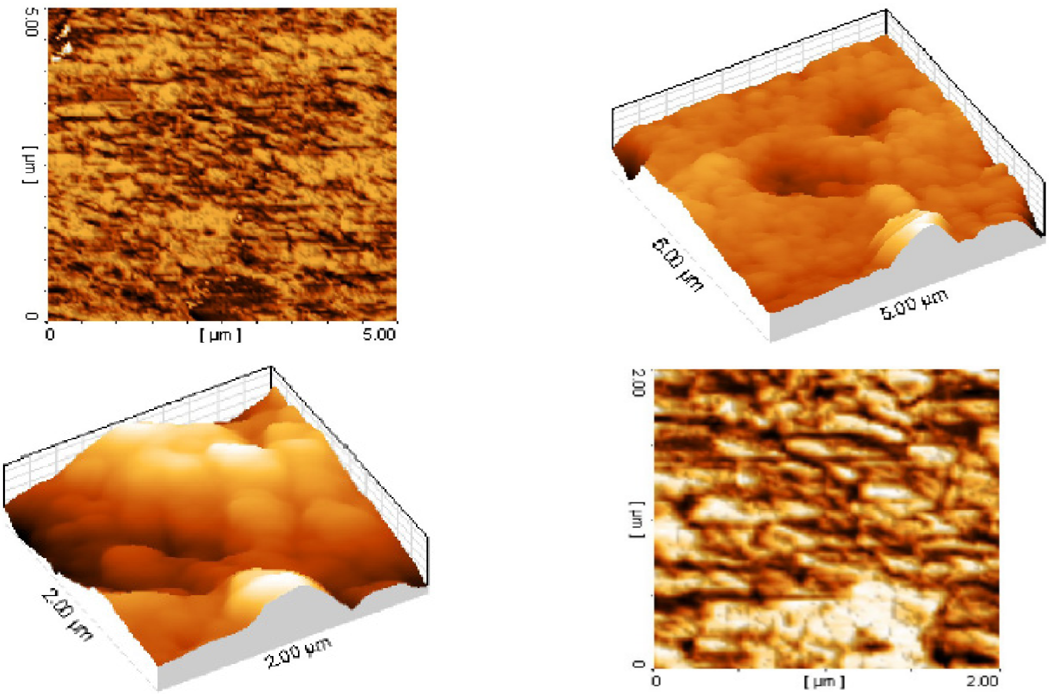


Fig. 3. 3D AFM topography image for T7351 sample in A) a $5\mu m * 5\mu m$ section and B) a $2\mu m * 2\mu m$ section.

3.4. Mechanical properties

Table 4 shows the mechanical properties of fully annealed T73 and T7351 heat-treated samples. The results show that maximum tensile strength, yield strength, and elongation were obtained in the T7351 heat treatment.

Table 4: Mechanical Properties of Different Samples

Sample	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation %	Hardness (BHN)
Full Annealed	100	215	16.5	51.5
T73	410	452	8	162
T7351	420	473	12	164

After applying T7351 heat treatment, a lot of dislocation can be produced because of 1-5% stretching after quenching. These dislocations are preferable sites for nucleation and precipitation growth inside the grains, which prevent the movement of dislocation and then increase mechanical properties [15]. It can be noted that T7351 heat treatment not only increases yield and tensile strength but also increases elongation. This must be because of the immigration of large dark precipitates from grain boundaries into the inside of the grains with homogenous and small size distribution.

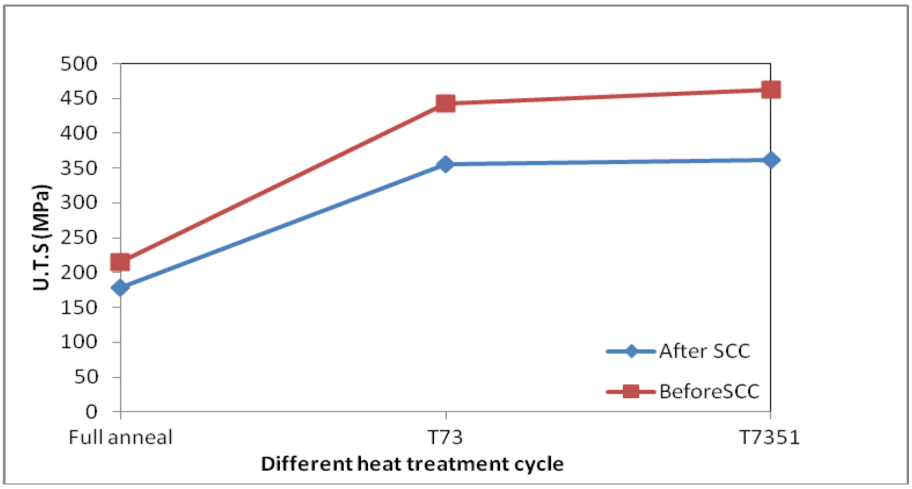


Fig. 4. Illustration of sample strength in different heat treatment cycles before and after the SCC test.

Fig. 4 shows the relationship between the strength of samples and different heat treatments before and after the SCC test. All the results show that the strength of samples decreases after the SCC test, which is consistent with Song et al. [16]. Moreover, a comparison between the T73 and T7351 cycle shows that resistance to SCC in samples with T7351 treatment is higher than the T73 cycle. It can be deduced that the immigration of some elements, such as Mg and Zn, from grain boundaries into the grains (Table 3) improves resistance to SCC. In fact, a crack in the SCC process initiated from the grain boundary due to the T7351 cycle caused the immigration of brittle precipitation from the grain boundaries inside the grains, resulting in improved SCC resistance. It seems that the immigration of Cu-enrich precipitation into the grains increases the amount of Cu inside the grains, resulting in increased resistance to SCC in samples, consistent with Marlaud et al. [17].

Conclusion

By applying the T7351 treatment of 4% stretching after quenching, not only do the tensile and yield strength properties increase compared to the T73 cycle (410 MPa yield strength compared to 420 MPa and 452 MPa tensile strength compared to 473 MPa), but the elongation also increases from 8% to 12% in this alloy. This treatment appears to improve mechanical properties compared to the T73 treatment, which can be better exploited in PEMFC.

- In T7351 heat treatment, the immigration of some Cu-enriched precipitates from the grain boundaries into the grains improves the resistance to SCC in samples, which is very important in PEMC.

- It can be concluded that T7351 is a better treatment for parts used in PEMC compared to T6 and T73 treatments.

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