The Effect of Quenching in Polymer and Addition of 0.1% Zr on Properties of Al-5.6% Zn-2.5%Mg-1.6%Cu Alloy

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Abstract. The Al-Zn-Mg-Cu aluminum alloys are primarily used in the aerospace industry as structural components. This study aim to improve properties of Al-5.6% Zn-2.5%Mg-1.6%Cu such as impact toughness, thermal stability and corrosion resistance by using quenching in 30% polyethylene glycol and addition 0.1%Zr to this alloy. Results showed that the addition 0.1% Zr to base alloy improve impact toughness by (40%) when quenching in water, and by (60 %) when quenching in 30%PAG corresponding to the base alloy when aging at 150 ºC. Also results showed that the thermal stability improved when we addition 0.1% Zr by (20%). An improvement of corrosion resistance when addition 0.1% Zr (B alloy) by (326 %), at aging time 150ºC in comparison to the base alloy.

Introduction

Aluminum alloys fall into two general categories: heat-treatable and non-heat treatable. Series 7xxx alloys, considered the high strength aircraft alloy family, are heat treatable by solution and aging. Various aging cycles produce desired attributes such as maximum attainable strength (T6 temper) or stress corrosion resistance (T73 temper). Either way, the alloy must go through solution treatment, the goal of which is to completely dissolve into solid solution all alloy elements responsible for subsequent precipitation hardening. The Al-Zn-Mg-Cu alloy, has one of the highest attainable strengths of all aluminum alloys [1].

Al-Zn-Mg-Cu series high –strength aluminum alloys with high specific strength and excellent mechanical properties have been the primary structural materials of aircrafts in the space and ground transportation vehicles. But the requirements for high yield stress and good fracture toughness are known to be contradictory. This has hampered the extensive development and utilization of high strength 7xxx series aluminum alloys. The relationships between microstructure and those two properties have been the focus of much research effort.

Adding trace elements to high-strength aluminum alloys can enhance their mechanical properties. It is found that trace elements and aluminum can form dispersoids to improve the recrystallization resistance of aluminum alloys, control the structure of grain boundary etc. These can stop cracks penetration along grain boundary [2].

Quenching of aluminum alloys includes heating to 465- 565 ºC and rapid cooling in order to obtain a supersaturated solid solution. Then the alloys are subjected to aging at different temperatures in order to obtain the requisite strength and plasticity. Minimum warping and minimum internal stresses are obtained by changing the cooling rate in quenching. It is known that well-mixed cold water is a good quenching medium that provides high strength characteristics for the alloys. Unfortunately, high cooling rates in cold water cause a considerable temperature gradient that leads to the appearance of residual stresses and warping and, quite often, the initiation of cracks.
By increasing the temperature of the quenching medium the temperature gradient in the quenched parts can be decreased while decreasing or eliminating the warping. It should be noted that parts produced from aluminum alloys often have a complex geometry [3].

One of the ways to decrease warping while preserving the requisite mechanical characteristics consists in using aqueous solutions of polymers as a quenching liquid. In this case the coefficient of convective (film) heat exchange between the part and the quenching medium is decreased, which decreases the rate of heat removal from the surface of the part and improves the uniformity of the temperature distribution in the volume of massive parts with a variable cross section. We estimated the possibility of using well-known UCON polymer for quenching aluminum alloys for the aircraft industry [3].

Aqueous solutions of poly alkylene glycol (PAG) are used to improve the cooling characteristics of the quenching medium and to reduce the machining requirements after the heat treatment. PAG concentrations vary from 4 to 30%, depending on the type of product being processed. For the heat treatment of aluminum alloys, such polymeric solutions have been widely applied during more than 30 years[4].

Warpage is a major problem, which leads to laborious, expensive, and time consuming straightening operations and in some cases to scrapping of large expensive parts. Warpage can be minimized by adding polymer to water quenchants to reduce the convective or film coefficient between the parts and the water. These synthetic quenchants retard the heat transfer from the components surface and reduce the temperature differential between different thicknesses of the material [5].

The reduction of warping in the process of quenching is important not only for rolled products but also for castings, finished parts, and semi finished products[6].

Heat treatable aluminum alloys are widely used in aircraft structural applications and are susceptible to localized corrosion in chloride environments, such as pitting, crevice corrosion, intergranular corrosion, exfoliation corrosion and stress corrosion cracking [7]. Metallic corrosion under wet conditions is generally electrochemical, occurring in corrosion cells at the metal surface. Being corrosion phenomena in aqueous environments generally of electrochemical nature they are dominated by the corrosion potential, Ecorr., of the metal. [8].

In this work we study the effect of trace alloying element (i.e. 0.1%Zr) and mediums of quenching (i.e. water and 30% polyethylene glycol) on impact toughness, thermal stability, corrosion resistance, of alloy that used in this study.

### Experimental Work

#### Used Alloys

The Al-based alloys used in this research are shown in Table (2-1):

<table>
<thead>
<tr>
<th>Alloy</th>
<th>% Zn</th>
<th>% Mg</th>
<th>% Cu</th>
<th>% Zr</th>
<th>% Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>5.6</td>
<td>2.5</td>
<td>1.6</td>
<td>-</td>
<td>Bal</td>
</tr>
<tr>
<td>B</td>
<td>5.6</td>
<td>2.5</td>
<td>1.6</td>
<td>0.1</td>
<td>Bal</td>
</tr>
</tbody>
</table>

#### Sample Preparation

All specimens were prepared firstly, by casting process and then machined to the required dimensions. These ingots were prepared as follows:

**Casting Process.** Casting process includes that, die designing and manufacturing. Ingots as shown in Fig.(2-1) were prepared by melting and then cast in especially design die with respect to the required ingot.

**Specimens Machining.** Samples machining involves the following steps:

1. This process for obtaining sheets of aluminum with 10.25 mm thickness and rod of aluminum with 15 mm diameter and 10 mm high.

2. Grinding Process: to obtain the final thickness of the sheet (10mm) according to the standard specimens thickness that used for impact testing.[10].
Heat Treatment of Specimens. Heat treatment usually includes three main stages namely:

1) Solution Heat Treatment
Heating alloys into solid solution at 482°C for two hours [9]. Later, specimens quenched by different ways of cooling.

2) Quenching
In this study water, and polymer solutions are common quenching mediums for aluminum alloys. The mediums differ in the rate at which they dissipate heat out of a quenched part. Medium of water have temperature [-1 ºC] and medium of polymer have [ 30%PAG +70% water].

3) Aging
The final stage to optimize properties in the heat-treatable aluminum alloys was aging. In this study we used only artificial aging at temperature 150°C and different times.

Mechanical Testing. Many mechanical testing have been done.

4) Hardness Test
Hardness is a measure of a material’s resistance to localized plastic deformation (e.g., a small dent or a scratch). Early hardness tests were based on natural minerals with a scale constructed solely on the ability of one material to scratch another that was softer [11]. Appropriate grinding and polishing was done before subject specimens to hardness tests.

5) Impact Test
Impact testing was performed on Charpy testing machine using 55mm length with 45° V notch, 2mm deep with 0.25 mm root radius.

The number of specimens of impact test shown in Fig. (2-2).

Corrosion Test. Calculated current of corrosion in this study depend on electrochemistry way.

6) Linear Polarization
The tester consist of electrochemical corrosion test cell and electrodes. The cell made from materials anti-corrosion as glass and the shape of the cell was seems- spherical with volume 1000 ml. The cell contain three electrodes are: Working electrode, Auxiliary electrode and reference electrode.

We used in this study [( 3.5% NaCl ) ( by weight)] and [( 97% distilled water ) (by volume)] and we used specimens have [dimensions ( diameter = 8 mm, thickness = 2 mm) and aged at temperature 150 ºC ( best aging temperature)].

Drawing the polarization curve anodic and cathodic has been automatically by computer that communicated with Potentionstat and we used the program (Bank-Elechtionies) to drawing the curves. The Tafel tester to test electrochemical corrosion.

Results and Discussion
The results are presented and discussed under various aspects difference between quenching in two mediums (polyethylene glycol and water) and note the difference by compared the results we obtained in each parts of work such as corrosion resistance, and mechanical properties such as (impact toughness and micro hardness properties).

Thermal Stability Tests. The little change in hardness at 150 ºC with aging time indicate to thermal stability at these temperatures.

The relationship between Vickers hardness and exposure time at temperature 150 ºC appears in Figs. [(3-1) and (3-2)].

From Fig.(3-1) we concluded A alloy (quenched in 30% PAG) have maximum value of hardness (at aging time 4 hr.) was 288.7 kg/mm2, and the same alloy (quenched in water) have maximum value of hardness (at aging time 4 hr.) was 295.7 kg/mm2 because the medium of water was faster quenching than PAG, and grain size in A alloy (quenched in water) become very small, these reasons cause high hardness.

Fig (3-2) appears that B alloy (quenched in 30% PAG) have stability in values of hardness in a most of aging time than same alloy (quenched in water) because the following reasons: first, the quenching in pure water originates the very high residual stresses, which lowered by addition of PAG (UCON) polymer quenchant to the bath, and second, the quenching with a concentration of 30% of this polymer gives a product free from residual stresses.
From Figs. (3-1) and (3-2) we concluded B alloy (quenched in water) caused an increase in thermal stability by (20%) than A alloy (quenching in water) because the element of Zr (in B alloy) have high thermal stability.

Figs (3-1) and (3-2) appears that B alloy have high thermal stability. The cause of high thermal stability two reasons, first this reasons containing Zr element and that element work fine grain size to aluminum matrix with dispersoids., second reason Zr element have limit solubility and limit diffusivity in aluminum therefore Zr produce stable dispersoids and these dispersoids have shape and volume remains with no change.

**Impact Toughness Tests.** The Charpy impact test, also known as the Charpy v-notch test, is a standardized high strain-rate test which determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of a given material's toughness and acts as a tool to study temperature-dependent brittle-ductile transition. It is widely applied in industry, since it is easy to prepare and conduct and results can be obtained quickly and cheaply.

The relationship between energy absorbed and exposure time at temperature (150 °C ) appears in Figs. [(3-3) and (3-4)].

We found from Fig. (3-3) that the impact toughness of A alloy (quenching in 30% polyethylene glycol) increased by (20%) in comparison to the A alloy (quenching in water) because when we quenching A alloy that cause to reducing residual stresses and producing uniform in precipitate along grain size.

We obtained from Figs. (3-3) and (3-4) that the impact toughness at 150 °C of B alloy (quenched in water) an increase by (40%) in comparison to the base alloy because B alloy contains Zr and the element of Zr work to prevent the processes of recrystallization.

**The Corrosion Test by Electrochemical Method.** We used linear polarization to calculate corrosion potential (E corr.) and corrosion current density (I corr.) through point of intersect the both Tafel lines (Ecorr. and Icorr.) that makes possible the estimation of the corrosion current density by the extrapolation of the Tafel slopes with corrosion potential.

We used this method to calculate corrosion rates in (3.5% NaCl) solution. The samples of test quenched in two medium (water and 30% polyethylene glycol) and aged at 150 °C (at this temperature we obtained best mechanical properties).

Fig. (3-5) shows that A alloy (quenched in water) have:

\[
\begin{align*}
I_{corr.} &= 338 \, \mu A/cm^2 \\
E_{corr.} &= -727.1 \, mV
\end{align*}
\]

The values of corrosion current density above indicates that A alloy have low resistance to corrosion.

Fig. (3-6) shows that A alloy (quenched in 30% PAG) have:

\[
\begin{align*}
I_{corr.} &= 79.25 \, \mu A/cm^2 \\
E_{corr.} &= -742.6 \, mV
\end{align*}
\]

From Figs. (3-5) and (3-6) we obtained that value of corrosion current density at 150 °C of A alloy (quenched in 30% polyethylene glycol) decreased by (326%) in comparison to the base alloy (quenching in water).

The value above indicates these alloy quenched in PAG have resistance corrosion best (A) alloy quenched in water because the alloy quenched in 30% polyethylene glycol the precipitates are uniformly distributed throughout the whole grain and these precipitates have higher efficiencies than those that accumulate along the grain boundaries perhaps because of greater breakdown of the passive oxide network.

Fig. (3-7) shows that B alloy (quenched in water) have:

\[
\begin{align*}
I_{corr.} &= 79.25 \, \mu A/cm^2 \\
E_{corr.} &= -742.6 \, mV
\end{align*}
\]

Above values of corrosion current density indicates that B alloy (containing Zr) have high corrosion resistance than A alloy because Zr element work to producing small grain size from dispersoids.

That is the directions of boundary movement and atomic motions are opposite to each other. Thus it means that diffuses uniformly it leads to shrinkage of grain boundary.
Fig. (3-8) shows that B alloy (quenched in 30% PAG) have:

\[ I_{\text{corr.}} = 58.15 \, \mu\text{A/cm}^2 \]
\[ E_{\text{corr.}} = -745.2 \, \text{mV} \]

From above values of corrosion current density we found B alloy (quenched in 30% PAG) have best corrosion resistance than B alloy (quenched in water).

From values of corrosion current density, the B alloy (quenched in 30% PAG) have higher value of corrosion resistance compare with base alloy (quenched in water and 30%PAG) because it contains element of Zr and that element work in best efficiency to prevent corrosion.

Conclusions

According to results of present work, the following can be concluded:

1- Quenching in medium of 30% polyethylene glycol improve most of properties of alloys that used in this study such as impact toughness, corrosion resistance and thermal stability for alloys at the most aging times especially at 150°C.

2- Addition 0.1% Zr to the base alloy improve impact toughness by (40%) when quenching in water, and by (60 %) when quenching in 30%PAG at 150 °C in comparison to the base alloy.

3- Thermal stability improved when we addition 0.1% Zr (B alloy) by (20%) at aging temperature 150°C in comparison to the base alloy.

4- Corrosion resistance improved addition 0.1% Zr (B alloy) by (326 %) at aging temperature 150°C in comparison to the base alloy.

References


Fig. (2-1) Shown that the a: rod ingots, b: plate ingots.

Fig. (2-2) Shown that the specimens of impact test, a: before fracture, b: after fracture.

Fig. (3-1) Variation of hardness of A alloy sample (quenching in a: water medium, b: 30% polyethylene glycol medium) with ageing temperature at 150°C.

Fig. (3-2) Variation of hardness of B alloy sample (quenching in a: water medium, b: 30% polyethylene glycol medium) with ageing temperature at 150°C.
Fig. (3-3) Variation of energy absorbed of A alloy sample (quenching in a: water medium, b: 30% polyethylene glycol medium) with ageing temperature at 150°C.

Fig. (3-4) Variation of energy absorbed of B alloy sample (quenching in a: water medium, b: 30% polyethylene glycol medium) with ageing temperature at 150°C.

Fig. (3-5) Tafel curve for A alloy (base alloy) that quenched in water.

Fig. (3-6) Tafel curve for A alloy (base alloy) that quenched in 30% PAG.
Fig. (3-7) Tafel curve for B alloy (containing Zr) that quenched in water.

Fig. (3-8) Tafel curve for B alloy (containing Zr) that quenched in 30% PAG.
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