



## RESEARCH ARTICLE

### THE EFFECTS OF ARTIFICIAL AGING HEAT TREATMENT ON MECHANICAL PROPERTIES AND CORROSION BEHAVIOUR OF AA6XXX ALUMINIUM ALLOYS

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#### ABSTRACT

Aluminium alloys are now widely preferred in industrial applications due to their low density and high strength values. The properties of these alloys can be further improved by precipitation hardening heat treatment. For this purpose, the temperature and time used in the precipitation hardening process plays a very effective role on the performance of the material. Therefore, in this study, the effects of artificial aging heat treatment on mechanical properties and corrosion behaviour of AA6XXX alloy were investigated. The effects of artificial aging time on microstructure and mechanical properties of alloy were analysed and at the same time corrosion behaviour was tried to be determined by corrosion tests. For experimental investigations, the alloy samples prepared in appropriate sizes were heated to 540°C ( $\pm 0.5^\circ\text{C}$ ) with heating rate 10°C/min. by electrical resistance ceramic furnace. For the solution heat treatment, samples were kept in the furnace at this temperature for 4 hours. The samples taken from the furnace were firstly poured into iced water at 10°C and then subjected to artificial aging at 190°C for 2, 4, 6, 10, 12 and 24 hours. Finally, the samples taken from the furnace were left to cool down in stagnant air. The results show that the mechanical properties and the corrosion resistance of the alloy increase with the increase of artificial aging time. The hardness of commercial alloy of 43 BHN is increased to 98 BHN and the corrosion resistance of the commercial alloy increased from 0.1753 mm/year to 0.0123 mm/year at a temperature of 190°C at 10-hour aging period.

#### INTRODUCTION

Aluminium is the second most used metal in the world. Due to its high specific strength (strength/weight ratio), easy formability, high thermal conductivity, compatibility with surface treatments and resistance to corrosion, aluminium and its alloys are used in a wide range of applications from automotive, building and packaging sectors to high voltage-electricity transmission lines to construction applications (Nandy *et al.*, 2015; Mandava *et al.*, 2014; Wu and Liao, 2013).

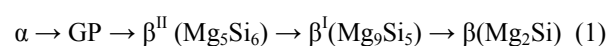
6XXX series aluminium alloys, containing Mg and Si as main alloy elements have generally good extrusion and rolling capabilities. These alloys also have good corrosion resistance, especially atmospheric environments. In addition to these favourable properties, the maintenance of the gloss of the anodized surface of the 6XXX series aluminium alloys also increases the amount of commercial use day-by-day (Panigrahi and Jayaganthan, 2010; Vargel, 2004; Yuksel, 2017). At the same time, the strength of this alloy group can be substantially increased by a two-stage heat treatment (Yuksel, 2017; Mohamed and Samuel, 2012). This two-stage heat treatment consists in solutioning and ageing process (Figure 1).

The solutioning process, holding the alloy in temperature below temperature of the eutectic reaction, is used to dissolve the precipitations of Mg<sub>2</sub>Si, homogenize the chemical elements concentration on the cross-section of dendrites of the  $\alpha$  phase and the change in the silicon precipitations morphology. The solutioning process is usually performed at 460-540°C for aluminium alloys of the 6XXX series.

The ageing process can be applied as natural aging at room temperature and the artificial aging at moderate temperatures. The ageing (soaking of the supersaturated alloy to separate strengthening phases from the supersaturated solid solution) the precipitation strengthening is obtained as a result of the phases precipitation of Mg<sub>2</sub>Si, Al<sub>2</sub>CuMg and Al<sub>2</sub>Cu (Pezda, 2014). Artificial ageing (holding the alloy at a constant moderate temperature during predetermined period) results in the improvement of the mechanical properties such as tensile strength and hardness and in simultaneous worsening of plasticity. Because of the growth of alloy's strength after heat treatment is very often accompanied with the reduction of the plasticity, their optimal relation should be selected depending on a given application of the alloy (Pezda, 2014). In the aging phase, which provides increased strength in the material, the precipitation process of the oversaturated melt takes place:

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The maximum hardness is possible by obtaining the phase  $\beta''$  during aging. On the other hand, the conversion of the  $\beta''$  phase to the  $\beta$  phase is defined as over-aging, and the Face-Centred Cubic (FCC) crystal structure of the equilibrium phase  $\beta$  results in a reduction of the hardness of the alloy (Maisonnette *et al.*, 2011; Li *et al.*, 2013).

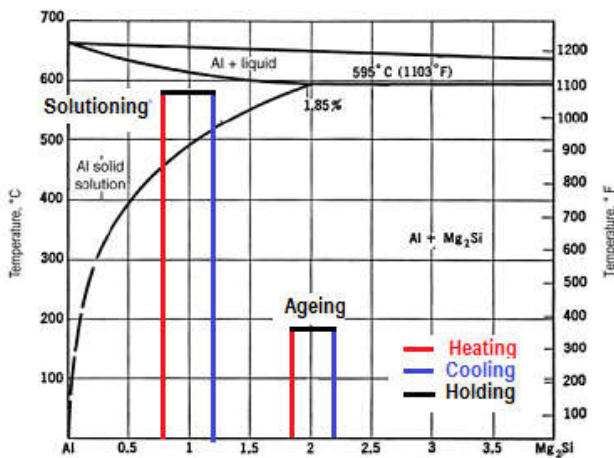


Figure 1. The schematics of precipitation heat treatment on Al-Mg<sub>2</sub>Si phase diagram

Depending on the temperature and duration of the applied artificial aging heat treatment, the type, size and quantity of the precipitated phase play an active role on the mechanical properties of the aluminium alloy and corrosion behaviour (Wu and Liao, 2013; Yuksel, 2017). For practical applications, the corrosion resistance is one of the most decisive factors beside the mechanical properties. For this reason, in this study, the effects of artificial aging on the hardness and corrosion resistance of AA6XXX alloy were investigated.

**EXPERIMENTAL STUDIES**

The chemical analysis of commercial AA6XXX aluminium alloy used in this study and in rod form is given in Table 1.

Table 1. The chemical analysis of AA6XXX aluminium alloy (wt.%)

Mg	Si	Mn	Fe	Cu	Cr	Zn	Ti	Al
0.66	0.96	0.49	0.204	0.02	0.004	0.014	0.016	97.63

The specimens, have dimensions of 12.09 mm in diameter and 15 mm, were heated to 540 ± 0.5°C with heating rate 10°C/min. in an electric resistance ceramic furnace. They were subjected to a solutioning treatment process for 4 hours at this temperature. The samples taken from the furnace were first put into iced water at 10°C and then subjected to artificial aging at 190°C for 2, 4, 6, 10, 12 and 24 hours. Finally, the samples taken from the furnace were allowed to cool down in stagnant air. Figure 2 shows the temperature versus time (thermal) profile for the T6 heat treatment carried out on all the AA6XXX alloy samples in this study. The heat-treated samples were prepared metallographically to examine their microstructural properties. For this purpose, the samples passed through the emery papers of 400, 600, 800, 1000 and 1200 mesh respectively were polished using 3 µm diamond paste solution and etched for about 30 seconds in Kellers Etch. Microstructure studies were performed using a JEOLJSM-6060LV brand SEM microscope. The hardness measurements of the samples were carried out on a Brinell hardness tester

using a 2.5 mm diameter hardened steel ball and a load of 62.5 kg. Hardness values were determined by taking the arithmetic average of at least 5 measurements for each sample.

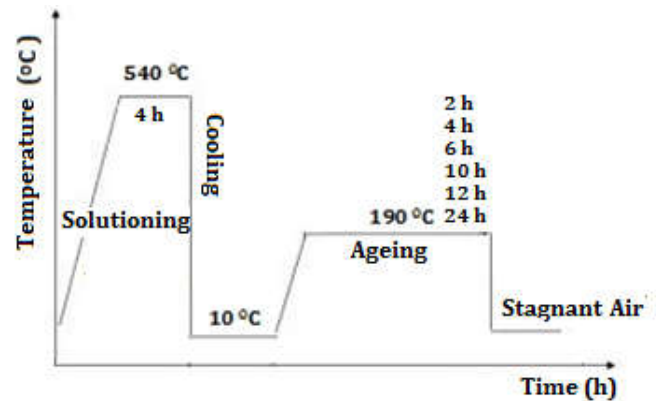


Figure 2. The schematics of heat treatment stages applied in experimental studies

Potentiodynamic polarization (Tafel) method was used to determine the corrosion behaviour of the samples. Three electrode techniques were used in the experiments performed in the Gamry potentiostat/galvanostat device, using saturated Ag/AgCl as the reference electrode and graphite electrodes as the auxiliary electrode. Experiments were performed at room temperature and 3.5% NaCl solution, and potentiodynamic polarization measurements were carried out at 5 mV/s for 3600 seconds with a voltage between -1 and +1V. Prior to the corrosion tests, the surfaces of the specimens, 12.09 mm in diameter and 5.16 mm in dimensions, were passed through standard metallographic sample preparation steps up to 1200 mesh emery paper.

**RESULTS AND DISCUSSION**

The change in hardness values according to the aging time of samples subjected to artificial aging at different durations at 190°C after solutioning treatment for 4 hours at 540°C is given in Figure 3.

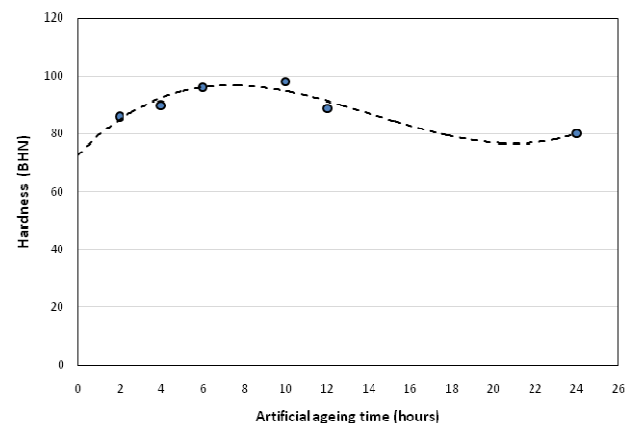


Figure 3. The effect of the artificial aging time on the hardness value of AA6XXX alloy

The commercial hardness value of AA6XXX aluminium alloy was 43 HB and after solutioning treatment the hardness reduced to 40 HB. As seen in Figure 3, the hardness value of the samples increases with increasing artificial ageing time. The highest hardness value (98 HB) was obtained at 10 h aging time at 190°C.

The hardness tended to decrease again with the prolongation of the aging period and the hardness value fell down to 89 HB in 12 hours and to 78 HB in 24 hours respectively. Based on the results of the performed investigations, a mathematical relationship between the hardness of the alloy and the heat treatment parameters can be established. Pezda, J. (8) informed the mathematical dependence of the effect of heat treatment parameters on the change of the alloy HB hardness as second order polynomial. The details of equation are given below:

$$HB = -1252,03 + 4,58X_1 + 101,1X_2 - 1,23X_2^2 + 0,94X_3 - 2,14X_4 - 0,4 X_4^2 - 0,18X_1X_2 + 0,02X_1X_4 - 0,01X_2X_3 - 0,03X_2X_4 - 0,02X_3X_4 \dots\dots\dots (2)$$

where:

- $X_1$ : solutioning temperature
- $X_2$ : solutioning time
- $X_3$ : ageing temperature
- $X_4$ : ageing time

At the end of the aging process, the increase in the hardness values of the alloys is due to the variations in the phases, precipitates and grain sizes formed in the microstructure. The increase in hardness is accepted as a sign of the success of the aging process after the dissolution in the literature (Mrowka and Sieniawski, 2005). The highest hardness value can be achieved by adjusting the optimum aging temperature and time. When the aging process starts, both magnesium and silicon are beginning to precipitate as  $Mg_xSi_y$ , as indicated by the formula of the solid solution. The precipitate phase formed at the start of aging is in perfect coherence with the aluminium matrix. But at high temperature, the  $Mg_xSi_y$  precipitates become incompatible with the matrix together with increasing duration. As a result of the artificial aging process, the maximum hardness value of the samples is obtained by precipitation of  $\beta^{II}$  ( $Mg_5Si_6$ ) phase in needle form. The  $\beta^{II}$  phase begins to turn into a rod like  $\beta^I$  ( $Mg_9Si_5$ ) phase during the increasing aging process, leading to a decrease in the hardness of the material. As the aging time increases, the precipitation of the  $\beta$  ( $Mg_2Si$ ) phase becomes more dominant, resulting in a marked decrease in the hardness of the aluminium alloy (Li *et al.*, 2013). In this case, full conformity is required to achieve higher strength and stiffness (Pratikno, 2015; Edwards *et al.*, 1998; El-Menshawey *et al.*, 2012). At the same time, coarse particles and constantly growing grain sizes are observed due to the combination of increasingly coexisting precipitates in the over aging period. In this period, the factors that prevent dislocation movements being decreasing and consequently the mechanical properties of the material are becoming increasingly smaller (Meyveci *et al.*, 2010). Corrosion current ( $I_{Cor}$ ) and corrosion potential ( $E_{Cor}$ ) values measured from the corrosion experiments of the samples by the potentiodynamic polarization (Tafel) method are given in Table 2.

**Table 2. The corrosion test results of AA6XXX**

Duration (Hour)	$I_{Cor}$ ( $\mu A$ )	$E_{Cor}$ (V)
2	-0,714	15,67
4	-0,838	9,13
6	-0,793	8,49
10	-1,140	1,30
12	-0,721	7,64
24	-0,744	5,73

The following formulas were used to calculate the corrosion rate ( $R_{cor}$ ) of the samples from these results (Baboian, 2016):

$$R_{cor} = \frac{I_{cor} \cdot K \cdot EA}{d \cdot A} \dots\dots\dots (3)$$

where:

- $R_{Cor}$ : Corrosion rate (mm/year)
- $I_{Cor}$ : Corrosion Current Intensity ( $\mu A$ )
- K: Constant (K:  $3.272 \times 10^{-3}$  mm/year)
- EA: Equivalent weight (atomic weight/valance)
- d: Density ( $g/cm^3$ )
- A: Surface area ( $cm^2$ )

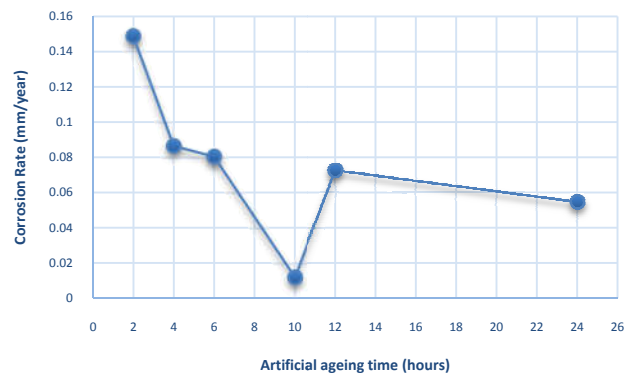
The T6 heat treated (solutioning and artificially ageing) AA6XXX alloy has a density of  $2,700 g/cm^3$ . The surface area of samples was  $1.1499 cm^2$  for corrosion test in this study and EA (equivalent weight) was calculated according to the chemical composition of alloy the by formula given below:

$$EA = \frac{1}{\sum \frac{n \cdot fi}{M_A}} \dots\dots\dots (4)$$

where:

- N: Atomic valance of element
- fi: Chemical composition of element
- $M_A$ : Atomic weight of element

When the data of alloying elements of AA6XXX alloy are substituted in the formula, the equivalent weight value is found to be 9.0027. The corrosion rate ( $R_{cor}$ ) was then calculated according to the formula given above. The change in corrosion rate with the heat treatment time is plotted in Figure 4.



**Figure 4. The change of corrosion rate with ageing time**

The corrosion rate of commercial alloy samples was detected as 0.1753 mm/year. As can be seen from Figure 2, the corrosion rate rapidly decreases with aging time and obtained the lowest value of 0.0123 mm/year in 10 hours, which is the optimum artificial aging time. In the case of over aging, the corrosion rate increases rapidly and reached to 0.0724 mm/year in 12 hours. Aluminium and its alloys are highly resistant to corrosion due to the thin, protective barrier oxide layer on their surface. However, in the presence of chloride ions in the environment this protective oxide layer of the region is likely to suffer from corrosion.

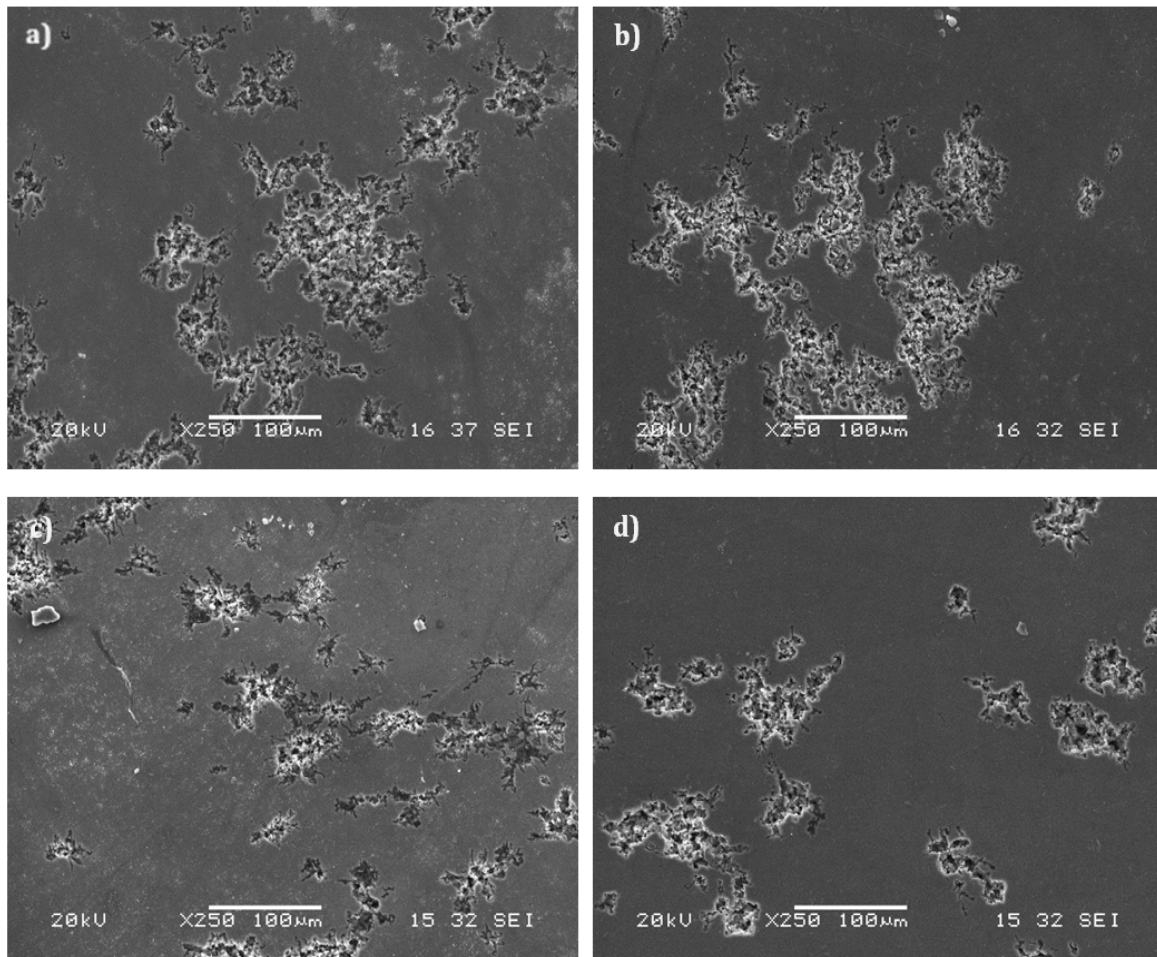
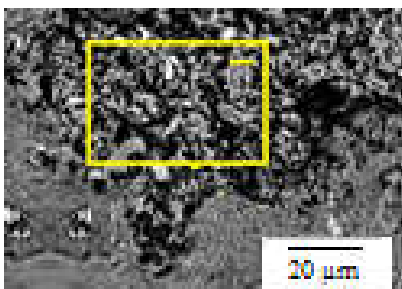


Figure 5. The SEM images of corrosion damages variations of AA6XXX alloy according to ageing durations a) 2 hours b) 6 hours c) 10 hours d) 24 hours



The EDS Analysis of marked area

Element	Rate (wt.%)
Si	0.62
Mg	0.94
Cl	0.65
Al	98.44

Figure 6. Surface morphology after artificial aging AA6XXX alloy after corrosion test and EDS analysis of corrosion products

The most important factors affecting the corrosion resistance of aluminium alloys are the alloying elements they possess. However, temperatures and durations used in heat treatments also play a dominant role in corrosion behaviour (Jones, 1996). After the corrosion test, surface morphologies of the samples were examined on a SEM microscope. A pronounced pitting corrosion was observed on the surfaces of each specimens (Fig. 5). Similar results have been obtained in the literature reviews and it has been observed that with increasing aging period, the pitting corrosion also increases (Svenningsen et al., 2006). A similar situation has been detected by Yuksel (Yuksel, 2017) by the corrosion tests performed to AA6063 alloys artificial aged at different temperatures and durations. It has been found that intergranular corrosion decreases with increasing aging time, and with the increase of pitting corrosion and over-aging, pitting corrosion becomes dominant.

The corroded areas of the material surface were examined by SEM microscopy and the obtained SEM image and EDS analysis are given in Figure 6. The EDS analysis of the marked corrosion zone on the microstructure resulted in a concentration of chlorine ions (0.65%) in the corrosive solution, which is similar to many studies in the literature (Yuksel, 2017; Svenningsen et al., 2006). When the aging process starts, both Mg and Si are beginning to precipitate in the form of solid solution  $Mg_2Si$  and there is an Al matrix around this phase. In this case, electrochemically the  $Mg_2Si$  phase is more active than the Al matrix so this situation causes the formation of a micro-galvanic corrosion cell and the intergranular corrosion between the phase bound to the grain boundaries and the matrix (Yuksel, 2017). As the selected temperature for artificial aging process increases, the aging time required to achieve optimum properties is shortened. In this case, however, the increasing aging time leads to the

transition of the precipitated phase from needle-shaped to rod-like form (Maisonnette *et al.*, 2011). The increase in pitting corrosion associated with increased aging time is due to the increase in the physical dimension of the more active precipitate phases compared to matrix. But this is not exactly proven (Svenningsen *et al.*, 2006; Svenningsen *et al.*, 2006).

## CONCLUSION

The obtained results from the artificial aging process applied to AA6XXX aluminium alloy at 190°C for 2, 4, 6, 10, 12 and 24 hours and their pertinent discussion allow drawing the following conclusions:

- The AA6XXX Al-Mg-Si alloy exhibits strong ageing response in spite of having very small amounts of Mg and Si.
- The experimental results have revealed that aging between 8 and 10 h at 190°C is the most suitable combination of time and temperature imparting maximum corrosion resistance and hardness to the alloy.
- Up to the completion of the precipitation of the  $\beta^{II}$  phase, a significant increase in the hardness of the material with increasing aging time was obtained and the highest hardness value (98 HB) was detected at the 10-hour aging time.
- With longer aging times, during the transformation of  $\beta^{II}$  phase to  $\beta^I$  and  $\beta$  phases (over-aging), the hardness of the alloy decreases rapidly as the sizes and distributions of the precipitates grow out of homogeneity and join with the adjacent precipitates and grow to extreme levels. The hardness value fell down to 89 HB in 12 hours and to 78 HB in 24 hours respectively.
- Likewise, the corrosion rate of alloys is reduced with increasing aging time, the minimum corrosion rate (0.856 mm/year) was obtained at 10-hour aging time.
- In the aging process of the AA6XXX alloy, two types of corrosion were observed, namely intergranular corrosion and pitting corrosion.
- During the artificial aging, the intergranular corrosion susceptibility of the alloy continues to decrease until the precipitation of the  $\beta^{II}$  phase is complete. However, as the size of the precipitated phase increased, the intergranular corrosion resistance increased, while the susceptibility of the alloy to pitting corrosion increased.

## ACKNOWLEDGEMENT

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