

## Effect of Overageing Conditions on Microstructure and Mechanical Properties in Al–Si–Mg Alloy

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**ABSTRACT:** Al-Si alloys have occupied significant position in the field of automobile applications. They are mainly used in engine parts where the alloys have to withstand high temperature for considerable length of time i.e ageing effect. This research work has been carried out to investigate the overageing effect on a series of heat treatable Al-Si-Mg alloy (A355 alloy). The alloys were heat treated at 175 °C for different length of time and microstructure and mechanical properties were studied. Considerable changes in microstructure were observed by SEM. Microstructure of moderately aged (1, 2 & 5 hours) alloys showed small precipitated particles, where overaged (1000, 10000 & 100000 hours) alloys showed coarse precipitated particles in grain boundary. Composition of the matrix and precipitated phase were ensured by EDS. These changes in microstructure significantly changed mechanical properties of the alloys over different ageing time. Initially the yield strength and hardness of the alloys increased up to a certain length of heat treatment and then it started to decrease with increasing heating time. Total elongation before fracture reduced initially and then increased with increasing heating time. Initially the dispersed second phase particles increased the mechanical strength. But eventually these properties decreased due to the coarsening of the particles. The study leads to the conclusion that the optimum aged was achieved between 3 to 5 hours of ageing time.

**Keywords:** Ageing effect, precipitated phase, coarsening of the particles, Al alloys, tensile properties, precipitation hardening.

### I. INTRODUCTION

Precipitation-hardenable Al alloys have the potential for being used in the manufacture of several parts and components in the automotive and aerospace industries, including strategic power train components [1-5], mostly due to their inherent lightness and good weight-to-strength ratio [6]. Among them, Al-Si alloys have shown excellent formability and corrosion resistance in practical application [7]. However, effectiveness of these applications requires development in high operating temperature and internal pressure for a remarkable length of time. The Al-Si alloys have limited thermal stability and lose their strength above approximately 150°C [8-10]. Thus, search for new Al-Si based grades with sufficient thermal stability at continuously increasing temperatures are going on [10-13]. According to recent findings, alloying elements, such as Cu, Zn, Mg and transitional metals are being used in the base Al-Si chemistry to modify mechanical properties and corrosion resistance at elevated temperature [10, 12, 14-19]. As documented in the literature, the effects of Mg content on the mechanical properties and quality index of Al–Si casting alloys were examined in a number of studies. Drouzy et al. [20] reported that increasing the Mg content from 0.25 to 0.44 % in a sand cast Al–Si–Mg alloy results in increasing the strength of the alloy without any appreciable change being observed in its quality index. A further study [21, 22] investigated the influence of Mg content on the tensile properties and quality index of Al–Si–Mg casting alloys containing Mg in the range of 0.06–0.44 %. It was observed that increasing Mg levels in this range resulted in increasing the strength and raising the quality index of the alloys. The improved quality of the casting, in this case, is related to the fact that the amount by which alloy ductility is decreased as a result of adding Mg is less than the amount by which the strength is increased; thus, the overall effect of increasing Mg from 0.06 to 0.44 % is an increase in the quality index. [23]. Ca´ceres and Barresi [24] studied the influence of Mg content (0.3, 0.5, and 0.7 %) on the quality index of Al–Si–Mg casting alloys investigated under conditions of various iron levels, solidification rates, and heat treatments. It was concluded that the quality index value of 355 alloys may be optimized if the Mg content does not exceed 0.5 % in order to avoid the precipitation of the p-Al<sub>18</sub>Mg<sub>3</sub>FeSi<sub>6</sub> phase. The general precipitation sequence of Al–Mg–Si alloys is as follows. Super-saturated solid solution (SSSS) → clusters/GP zones → β'' → β' → β [25–28].

The needle-like monoclinic  $\beta''$  phase is considered to be the most effective strengthening precipitate among all types of precipitates in Al–Mg–Si alloys. Its composition is normally accepted as Mg<sub>5</sub>Si<sub>6</sub> [25, 29]. Natural aging (NA) can significantly depress the hardening kinetics and the maximum strength obtained in subsequent artificial aging of Al–Mg–Si alloys. This phenomenon, also well-known as “negative NA effect”, has attracted a lot of interest because of its link with practical industrial process [30–34]. However, overageing has adverse effect on this alloy [23]. So, it is very beneficial to have clear concept regarding the actual state of mechanical properties at that severe ageing condition. In this research work change of microstructure and mechanical properties with different ageing time have been investigated. Thus this project has a potential of determining the actual state of strength and hardness when any Al-Si alloy parts experience any elevated temperature for any length of time.

## II. EXPERIMENTAL

### a) Preparation of master alloy

Two distinct alloys have been taken to make the Al-Si master alloy of composition around 4-7% Si and 0.3-0.6% Mg. The nominal composition selected for this experiment are shown in Table-01.

**Table-01:** Nominal composition of modified alloys and master alloy

Alloy	Si (%)	Mg (%)	Taken amount
Alloy 1	3.60	0.04	65 %
Alloy 2	6.33	1.07	35 %
Master Alloy	4.50	0.40	100 %

Selected alloys were melted in pit furnace at temperature 750°C and permanent metal mold was used to prepare master alloy. The composition of the alloys obtained from OES analysis shown in Table-02 resembles with desired composition.

**Table-02:** Average composition from OES analysis of master alloy.

Constituent	Al	Si	Mg	Cu	Fe	Zn	Ti	Cr
wt (%)	91.15	4.59	0.45	1.64	0.85	0.26	0.15	0.22

### b) Preparation of Test samples

Master alloy was melted in pit furnace at 750°C and rectangular test bar were prepared from sand mold. Sand mold were preheated for half an hour to resist thermal shock and also to remove moisture content. Total 45 rectangular samples were used to study nine different conditions of alloys. From rectangular plate, cubic shaped samples were sectioned for SEM analysis. The alloys were fine polished on a polishing wheel using  $\beta$ -alumina particles powder as polishing medium. Non-ferrous metallographic fine polishing standard technique was carried out with 0.5 $\mu$ m  $\beta$ -alumina particles in order to obtain the microstructure. Keller’s reagent was used as etching reagent. The ingots were mechanically machined into tensile specimens with a gauge length of 25.0 mm for each sample as per sub-standard size of the ASTM E8/E8M-11 and the width and thickness of the samples were 6.00 mm and 5.00 mm respectively. For hardness test cubic shaped samples were prepared.

### c) Solution treatment

The samples were packed together with steel wire and placed in automatic digital induction furnace. Temperature was raised to 540 °C at the rate of 4 °C per minute to avoid cracking and distortion. The samples were kept at 540°C for 10 hours. After that, they were brought out of the furnace and quickly quenched in water. Next they were refrigerated at 15°C for 24 hours.

### d) Ageing treatment

After solution treatment and ageing delay the test samples were divided into 8 groups. Seven ageing periods (1, 2, 5, 100, 1000, 10000, 100000 hours) were aged at 175°C. After ageing, the samples were cooled in air. Overall heat treatment cycle is shown in Fig-1. The modified ageing temperature and ageing time are shown in Table-03.

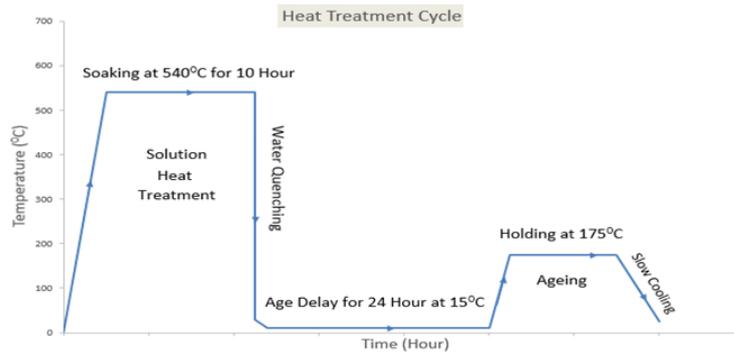


Fig-1: Heat Treatment cycle.

Table-03: Modification of ageing time and temperature.

Group	Ageing time at 175°C (hour)	Modified Ageing temperature(°C)	Modified ageing time (hour)
1	1	-	-
2	2	-	-
3	5	-	-
4	100	225	3.125
5	1000	255	3.90
6	10000	285	4.88
7	100000	315	6.10

e) Mechanical tests

Tensile and hardness tests were performed with the samples obtained from different heat treatment. Tensile tests were carried out with a universal testing machine (Instron-3369 Universal Testing Machine) at a strain rate of 3.00 mm/min at 25°C to obtain data on the stress–strain curves containing information of elongation at fracture and UTS. Hardness tests were performed in Brinell Hardness Testing Machine. 250 kg load was applied for 10 seconds on the samples to create indentation. The size of the indenter was determined by measuring two diagonals of the round indenter using a portable microscope. The average of the two diagonals is used in the following formula to calculate the Brinell hardness.

$$\text{Brinell hardness number} = \frac{L}{\left(\frac{D}{2}\right) \left(D - \sqrt{D^2 - d^2}\right)}$$

Where L = Test load in kg, D = Diameter of the ball in mm and d = Diameter of impression in mm.

f) Microstructure observation

After heat treatment of as-cast samples, SEM analysis was performed to observe the microstructure containing precipitated particles of different shapes. EDS analysis was also performed to be confirmed about the composition of precipitated particles and matrix phase.

III. RESULTS AND DISCUSSION

a) Microstructural Change

As-cast Al alloy contains proeutectic Al solution, eutectic mixture of Al and Si, Al oxide due to oxidation at higher temperature and precipitated particles. As the alloy is solution treated, almost all particles get dissolved in the Al matrix. High temperature during solution treatment increased the solubility and lead to quiet uniform microstructure as revealed by FESEM image (Fig-2,a).

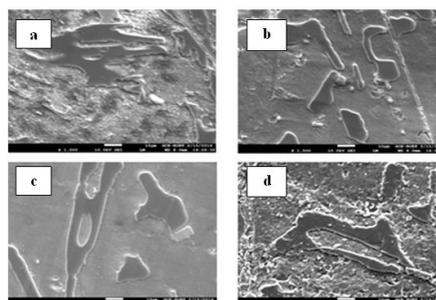


Fig-2: FESEM images of (a) as cast, (b) as treated, (c) aged for 5 hours, (d) over-aged for 100000 hours. All 300X magnification

When the alloy was quenched in water, the solubility decreased, but Si and Mg particles did not get sufficient time to be precipitated. Therefore, the structure became supersaturated solid solution. At ageing operation, with increasing time the supersaturated particles get enough time to precipitate. Thus the amount of precipitation gradually increased with increasing precipitation time. FESEM image (Fig 2,c) aged for 5 hours shows medium sized particles precipitated in Al matrix. Finally excess ageing time facilitates the precipitated particles to grow and become coarser. This phenomenon is recognized as overageing. Coarse precipitated particles due to overageing for 10000 hour are seen Fig-2, d. The EDS analysis of four different conditions are shown in Fig 3. EDS shows the chemical composition of precipitated (Fig-3, left) and matrix phase (Fig-3, right). Precipitated phase is Si rich, where Al enrichment of matrix phase also ensured by EDS.

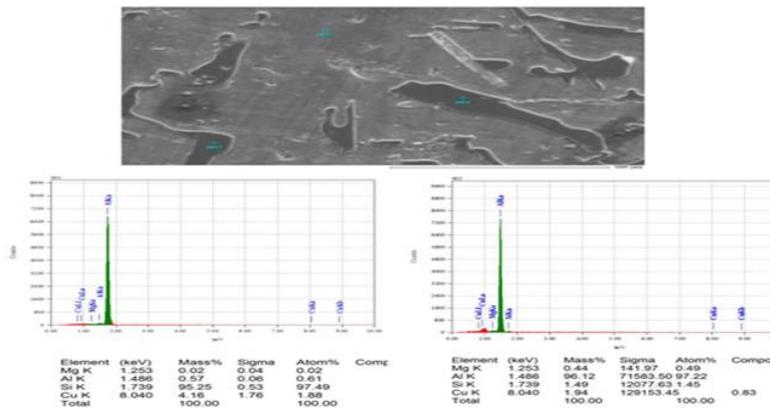


Fig.-3: EDS analysis. Composition of precipitation (Left) and Matrix (Right).

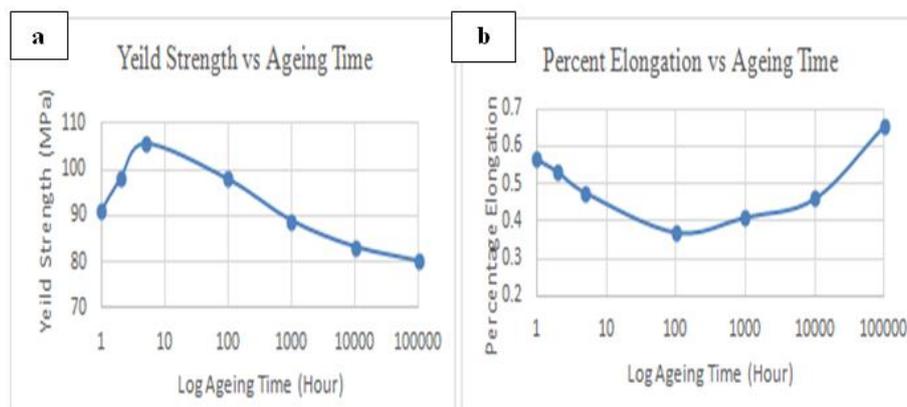
**b) Ageing effect on mechanical properties**

To observe ageing effect on mechanical properties tensile and hardness test has been performed. Obtained results of all tests in different conditions are given below in tabular form:

**Table-4: Effect of ageing time on Mechanical properties (Tensile and hardness test result).**

Average Result of 3 samples/sample condition	As cast	As Treated	Ageing time						
			1 hour	2 hours	5 hours	100 hours	1000 hours	10000 hours	100000 hours
% of Elongation	0.50	0.49	0.57	0.53	0.48	0.37	0.41	0.46	0.65
Yield Strength (MPa)	84.27	88.28	91.05	97.95	105.75	97.94	88.87	83.22	80.10
Brinnel hardness (BHN)	57.25	63.03	72.56	86.61	103.1	70.81	59.80	55.6367	50.82

From above table it is seen that Yield strength for as cast alloy is lower than solution treated alloy. Then it increased with the ageing time and reached maximum 105.75MPa for ageing at 5 hours. Then, gradually decreased to 80.10MPa for ageing at 100000 hours. Initially, with increasing yield strength total percentage of elongation decreased significantly and then as yield strength started to decrease total percentage elongation increased with increasing ageing time. These changing trends of yield strength (Fig-4, a) and total percentage of elongation (Fig. 4, b) with ageing time have been shown graphically in Fig-4. Brinnel hardness of the alloy also gradually increased until ageing for 5 hours and then decreased gradually with increasing ageing time.



**Fig-4: Changes of (a) yield strength and (b) percentage of elongation with ageing time**

These phenomena of mechanical properties can be described by relating ageing effect on microstructure. After solution treatment microstructure of the alloy became almost homogeneous without significant precipitated particle in grain boundary. As the alloy was heated to observe ageing effect, intermetallic particles started to precipitate in grain boundary. With the increasing ageing time, amount of precipitated particles increased gradually until ageing for 5 hours. These precipitated particles hindered dislocation to move and consequently increased yield strength. Total elongation also reduced due to pinning effect by precipitated particles. When the alloys were heated further more time, precipitated particles began to become coarser. These coarse particles reduced effective pinning effect of particles allowing dislocations to move more easily than before. As a result yield strength of the alloy gradually became lower and as dislocation could move easily total percentage of elongation also increased gradually.

#### IV. CONCLUSION

Effect of ageing time on microstructure and mechanical properties has been studied in the work. With increasing ageing time amount of precipitated particles increased gradually. These precipitated particles in grain boundary hindered the movement of dislocation. As a result yield strength of the alloy increased and total percentage of elongation decreased gradually until a particular ageing time. With increasing ageing time precipitated particles become coarser and reduced the pinning effect of the dislocation. Consequently yield strength decreased and total percentage of elongation increased.

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