EFFECT OF TWO STAGES ARTIFICIAL AGEING ON MICROSTRUCTURE AND MECHANICAL BEHAVIOR OF LM 25 ALUMINIUM ALLOY

BHASKARA RAJU VVS1, SAMBI REDDY SEVANAM2, ANANDA BABU VARADALA3, SURESH CHITTURI4
1,3,4Mechanical Engineering Department, Vignan’s Institute of Engineering for Women, Visakhapatnam, India.
2Retired NSTL Scientist D, Visakhapatnam, India
Email: chitturisuresh007@gmail.com

Abstract. In the present investigation the influence of two stages artificial ageing, 1st stage at 160°C followed by 2nd stage at 140°C on microstructure and mechanical properties of LM 25 alloy was studied. It is observed that the microstructure changed under the influence of two stages ageing. It is also observed that the decrease in ageing temperature in the second stage, increases mechanical properties of the alloy. The tensile strength of the alloy in single stage ageing at 160°C is studied and compared with two stages ageing.

Keywords: Artificial ageing; Microstructure; Tensile strength; Hardness.

1. Introduction
Aluminium and its alloys exhibit multiple desirable properties like high resistance to corrosion, light weight and good strength, which made it possible for its extensive use in very broad range of applications from regular house hold to advanced applications in defence. Aluminum alloys are primarily classified as wrought and cast alloys depending upon the ability of fabrication. The wrought and cast alloys are labelled with four-digit numerals from 1xxx to 9xxx groups but in cast alloys a decimal is introduced before the fourth digit as 1xx.x to 9xx.x. Mechanical properties of aluminum alloys possessing high solid solubility, which are called heat-treatable alloys are enhanced by thermal treatment, quenching and age hardening, whereas other Aluminum alloys which are non-heat treatable are subjected to Strain hardening or Cold working. Aluminum alloys with silicon as a main alloying element form the most significant part of critical shape castings, especially in under water applications because of its high strength to weight ratio, good processability, weldability and corrosion resistance. Apart from casting process, microstructure of cast Al-Si is influenced by solidification rate, dendrite arm space. The micro structural characteristics such as morphology of silicon and intermetallic compounds depend upon the presence of alloying element. Factors like solutionizing time & temperature, quenching temperature & cooling rate, artificial ageing delay period are strategic heat treatment methods which are essential for control of mechanical and microstructure properties .Of late a variety of studies have been carried to study the affect of age hardening on Al-Si alloys with microstructure and mechanical properties as main objective. In age hardening of these alloys, the alloy was heated to solutionizing temperature and then water quench followed by a single stage artificial ageing, to enhance the mechanical properties. Elagin et al. [1] experimentally studied methods for producing high strength and high temperature structural aluminum alloys, observed improvement in strength with the incorporation of transition metals and earth race metals in to their composition.
Zakharov et al. [2] examined and developed high strength corrosion resistance weldable aluminum alloy by using Al-Zn-Mgs system with the additives of Cu, Ti, Zr and Sn. The incorporation of Cu element enriches corrosion resistance and the addition of Sn, Zr, Ti enriches the weldable characteristics of the aluminum alloy. Zhang et al. [3] studied precipitation of excess silicon in the cast alloy under different heat treatment process. They do not observe silicon precipitate at initial stage of ageing with high quench rate and at low quench rate the silicon precipitate forms during ageing. ESKIN et al. [4] studied precipitation kinetics through TEM and observed two types of interfaces in Al-Mg-Si alloys. Matsuda et al. [5] studied β” crystal structure in Al–1.0mass%Mg:Si–0.4mass%Si alloy and observed needle shape β” with their chemical composition as Mg:Al:Si =1:3:6. Cavazos et al. [6] studied precipitation in heat treatable aluminum cooled at different rates. They performed experiments at various age temperatures 130°C, 180°C, 230°C and observed the precipitation behaviour and concluded that reduction in hardness if the sample cooled below 10°C/s. Naga Raju et al. [7] studied AA 2219 aluminum alloy altered with Sc, Mg and Zr additives and observed these modified compositions have equiaxed grains and high temperature withstand precipitations. Lynch et al. [8] investigated micro analysis of precipitates in aluminum alloys, observed maximum hardness at early stage of precipitation. Wahi et al. [9] studied metastable Phase β” in Al-Si-Mg alloy and observed different ageing behaviour in the two alloys based on the present of Cu in their composition. Matsuda et al. [10] studied precipitates morphology in Al-1wt%Mg:Si alloy and classified orientation relationships of precipitates and matrix. They also observed that precipitates are elongated in prescribed direction in the matrix. Gupta et al. [11] analysed hardening of 6061/10% SiC composite and observed increased activation energies of β” precipitations with elevation of solution temperature from 510 to 600°C. Their micro structural analysis revealed increase in grain size at high solution temperature. Takeda et al. [12] examined Al–Mg–Si ternary alloys and concluded β” metastable precipitates plays a vital role in enhancing the components hardness.

The current research is an attempt to investigate the combined effect of 1st stage ageing at 160°C for one hour followed by 2nd stage ageing at 140°C for different holding periods, on tensile properties and microstructure of LM 25 (Ternary alloy of Al-Si-Mg). The binary alloys of Al-Si are not responding to heat treatment. But by the addition of Mg to this binary Al-Si alloy, respond to heat treatment by which the mechanical properties are enhanced. This alloy was chosen as it is used for many of the torpedo components, especially for shells.

2. Experimental Procedure
2.1 Casting test bars

The test bars used for experiment are prepared by melting commercially available LM 25 alloy ingots in an electrical muffle furnace. The constitution of the alloy is presented in Table 1. Degassing of the alloy has been carried by adding 0.05 wt. % degassing agent in which 2/3 sodium fluoride and 1/3 sodium chloride are present. The molten alloy was poured in a metallic mould having proper riser and runner mechanism. About 15 sets, each set with 3 samples are cast and used for tensile testing.

### Table 1. Chemical constitution of LM alloy

| Element | Wt.% |
|---------|------|
| Si      | 7.47 |
| Mg      | 0.74 |
| Fe      | 0.043|
| Mn      | 0.82 |
| Cu      | 0.028|
| Zn      | 0.028|
| Ti      | 0.004|
| Sn      | 0.009|
| Al      | 90.906|

2.2 Age hardening of cast bars and fabrication of test samples

The cast bars used for tensile testing and metallographic examination are solutionized at 520°C for 6 hrs and water quenched at 20°C. The bars cooled to room temperature are artificially aged in two stages. The 1st stage ageing is carried out at 160°C for 1hr and then cooled to 140°C in the furnace where the 2nd stage ageing is done. The 2nd stage ageing is performed for the different periods from 4
to 40 hrs. After age hardening, the tensile test bars have been fabricated as per ASTM E8M specification. The metallographic and hardness samples are made of diameter 10 mm and height of 20 mm. The heat treatment cycle for two stage artificial age hardening is as shown in figure 1.

![Two stage artificial ageing cycle](image)

**Figure 1. Two stage artificial ageing cycle**

### 2.3 Evaluation of microstructures
The samples with diameter of 10 mm and height 20 mm cut from the cast bars for micro structural studies are heat treated along with tensile and hardness samples. The samples are polished with a standard metallographic technique. Refined samples are etched by use of Keller’s reagent. Olympus make inverted optical microscope is used for micro structural examination.

### 2.4 Evaluation of mechanical properties
The samples for hardness testing are polished with 2/0 grade emery paper and hardness tests are carried out by using Reichert make computerized Universal hardness testing machine in Vickers scale. The hardness is evaluated at various places over the entire cross-section of the test specimen using a load 5 kgf and the average hardness is taken. The tensile tests are conducted on specimens prepared as per ASTM E8M specification at a constant strain rate of 2mm/min using FIE make UNITEK 95100 computerized tensile testing machine of 10 tons capacity. The ductility and UTS are calculated in terms of % of elongation.

### 3. Results and discussion
#### 3.1 Influence of two stages ageing on microstructure
The microstructure of LM25 as cast condition is shown in figure 2. It indicates that the structure is coarse eutectic with primary Si dendrites which may be due to slow cooling during solidification. The microstructure in solutionized condition is shown in figure 3. It was observed from the structure that a small fraction of un dissolved Si is present in the super saturated solid solution of alpha aluminum. The microlevel crystalline structure of alloy in the 1st stage ageing at 160°C for one hour is shown in figure 4. It can be seen from the structure that high density of second phase particles are nucleated at grains and grain boundary regions. It is obvious to say that the nucleation and growth rate of precipitates increase with increase in ageing temperatures. The microstructures of the alloy in 2nd stage ageing at 140°C with different holding time are shown in figures from 4 to 7. The microstructure of the alloy in 1st 4 hours ageing time shows a mixture of coarse and fine precipitates. This may be revealed that the precipitates, which are already nucleated in the 1st stage, are coarsen during furnace cooling to 140°C and the fine precipitates are the freshly nucleated particles at 140°C. From the microstructures of the alloy in 2nd stage ageing at different holding times, it was observed that the structure consists of different forms and sizes of precipitates. First the Guinier Preston (GP) zones form. These are the clusters of solute atoms which are only few atoms thick. Then intermediate precipitates β' and β'' formed which are much larger than GP zones and have definite composition and crystal structures slightly different to equilibrium precipitate Mg₃Si. The sequence of precipitates is GP zones β' - β'' - Mg₃Si.
3.2 Influence of two stages ageing on mechanical properties

The mechanical properties of the alloy as cast conditions are shown in Table 2. In this condition, the properties are low due to slow cooling during solidification and the presence of Si dendrites in the microstructure. The mechanical properties in solutionized condition are shown in Table 2. The results
indicated that improvement in the properties than in cast condition. This can be stated that in solutionized condition the solid solution strengthening was occur due to formation of supersaturated solid solution, by quenching the alloy from solutionizing temperature. Long holding time at terminal solid solution temperature results homogenization of alloying elements, might be another reason for improvement in tensile properties than in cast properties. The mechanical properties of the alloy in 1st stage ageing at 160°C for one hour are shown in table 2. Further improvement in strength and hardness were found in this condition. This can be reviewed that the high temperature ageing accelerates the nucleation and growth rate of precipitates. The mechanical properties of the alloy in 2nd stage ageing at 140°C for different holding times are shown in table 2. It is reported that strength and hardness increase with ageing time and reaches to peak values at 16 hrs of holding time. With further increase in ageing time, the strength and hardness decreased. The increase in hardness and strength to peak values with increase in ageing time can be obtained due to the growth of precipitates to a critical size and due to strong coherency of these precipitates with parent matrix. With further increase in ageing time tends to decrease in strength. This phenomenon is called over ageing. During coarsening, the size of the particles increased the number of particles decreased and the interparticle distance decreased. This leads to less resistance to dislocation motion in the matrix and makes the alloy soft. The variation of mechanical properties with ageing time in 2nd stage ageing under various holding times is shown figure 8 & 9.

| Condition of the material | Hardness VHN | UTS (MPa) | Elongation (%) |
|---------------------------|--------------|-----------|----------------|
| In cast condition         | 98           | 161       | 1.21           |
| In solutionized condition | 89           | 195       | 1.75           |
| 1st stage aged condition, aged at 160°C for 1 hour | 106 | 216.5 | 1.21 |
| 2nd stage aged condition, aged at 140°C for different holding times | 105 | 207 | 1.92 |
| 4hrs                      | 111          | 241       | 1.86           |
| 8hrs                      | 117          | 260       | 1.73           |
| 12hrs                     | 128          | 270       | 1.14           |
| 16hrs                     | 120          | 235       | 2.95           |
| 20hrs                     | 110          | 226       | 3.50           |
| 24hrs                     | 112          | 235       | 3.64           |
| 32hrs                     | 97           | 161       | 3.64           |
The tensile strength in single stage ageing at 160\(^\circ\)C for different holding times is shown in table 3. It was observed that enhancement in strength with ageing time and reaches to peak value at 4 hours ageing time and then decreased with further ageing time. The peak strength with short duration might be due to accelerated nucleation and growth rate of precipitates with increase in ageing temperature.
Table 3. UTS under 1st ageing at various holding times

| Holding time (hrs) | 1   | 2   | 4   | 6   | 8   | 10  |
|-------------------|-----|-----|-----|-----|-----|-----|
| UTS (MPa)         | 216 | 238 | 255 | 250 | 242 | 214 |

4. Conclusions
The microstructure of LM25 in as cast condition shows coarse eutectic with primary Si dendrites due to slow cooling during solidification. The microstructure of LM 25 in as solutionized condition shows small fraction of un dissolved Si in the super saturated solid solution of alpha aluminum. The microstructure of the alloy in 1st 4 hours ageing time shows a mixture of coarse and fine precipitates. This may be revealed that the precipitates, which are already nucleated in the 1st stage, are coarsen during furnace cooling to 140°C and the fine precipitates are the freshly nucleated particles at 140°C. The microstructures of the alloy in 2nd stage ageing at different holding times, it was observed that the structure consists of different forms and sizes of precipitates. First the Guinier Preston (GP) zones form. These are the clusters of solute atoms which are only few atoms thick. Then intermediate precipitates β´ and β' formed which are much larger than GP zones and have definite composition and crystal structures slightly different to equilibrium precipitate Mg₂Si. The sequence of precipitates is GP zones β´-β'-Mg₂Si. In two stage ageing, 5-8% improvement in tensile strength was observed than in single stage ageing. In two stage ageing, peak values of strength and hardness were achieved with long ageing periods.

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