# PRODUCTION AND HEAT TREATMENT OF 6111 ALUMINUM ALLOY SHEET FOR CAR BODY PANEL APPLICATION

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# ABSTRACT


## PRODUCTION AND HEAT TREATMENT OF 6111 ALUMINUM ALLOY SHEET FOR CAR BODY PANEL APPLICATION



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Aluminum is one of the major candidates to supply sufficient weight reduction without missing mechanical properties and performance. Therefore, studies have been focused on improving properties of aluminum in automotive application since 1920s. Heat treatable aluminum alloy are used in car body panels of automotive application such as 2xxx, 7xxx and 6xxx series aluminum alloys base on the mechanical properties. On the other hand, based on physical and economical properties 6xxx series aluminum alloys are widely preferred by automotive manufactures. The car body panel needs to be stiff to carry bulk, to sustain stress and to be tough enough to satisfy risk of collision and impact. Besides, it is designed as light as possible for fuel consumption economy and driving performance. For these properties, 6xxx series are important options. In the North America, AA6111 aluminum alloy has been used especially for outer panel of car up to 1 mm thickness. For this purpose, firstly composition as 6111 aluminum alloy was adjusted and prepared AA6111 aluminum alloy was cast into ingots as plate shape. Later, the plate were firstly hot rolled then cold rolled to the desired thickness at convenient condition. Then, to get the desired strength and formability, precipitation strengthening mechanism was chosen to control easily. Hereby, T4 heat treatment was applied. By varying process parameters, the formation of a sheet has been studied and outcome of process have been inspected. Different temperatures and ageing times were studied to find the maximum strength values while considering cost and processing time. Hence, the convenient process parameters were determined to design aluminum sheet for car body panel applications considering sector requirement of performance and safety, lower cost and waste of energy and finally environmental affect.

**Keywords**: Aluminum alloy sheet, 6000 series, heat treatment, car body panel application, and microstructure.

**ÖZ**

**6111 ALÜMİNYUM PLAKALARININ ARABA GÖVDELERİNDE KULLANILMASI İÇİN ÜRETİMİ VE ISIL İŞLEMİ**

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Mekanik ve performans kaybına maruz kalmadan ağırlık azaltabilmeyi sağlamak için alüminyum başlıca adaylardan biridir. Bu nedenle, 1920 yıllarından beri çalışmalar otomotiv uygulamalarında alüminyumumun özelliklerini geliştirme üzere yoğunlaşmıştır. Mekanik özellikleri baz alındığında, araba üreticileri panel yapımında ısıl işlem uygulanabilen 2xxx,7xxx ve 6xxxx seri alüminyum alaşımları kullanmaktadırlar. Diğer bir taraftan, ekonomik ve fiziksel özellikleri baz alındığında 6xxx seri alüminyum alaşımlar otomotiv üreticileri tarafından genellikle tercih edilmektedir. Arabanın panel kısımları kütleyi ve maruz kaldığı stresi kaldırabilmek için sert ayrıca; çarpışma ve etiklere maruz kalma riskini karşılamak için de yeterince tok bir yapıya sahip olmalıdır. Bunun yansıra; mümkün olduğu kadar yakıt tüketim ekonomisi yapabilmek ve sürüş performansı düşürmemek için hafif dizayn edilir. Bu amaçla ilk önce 6111 alüminyum alaşımlı levhalar uygun kompozisyonlarda dökülür. Sonra, ilk önce uygun koşullarda sıcak daha sonra soğuk haddeleme ile istenilen kalınlıkta sac elde edilir. Daha sonra; istenilen mukavemeti sağlayabilmek için çökeltme ile sertleştirme mekanizması kolay kontrol edilebilmesi için tercih edilir. Bu nedenle T4 ısıl işlemi uygulanır. İşlem parametreleri değiştirilerek sacın şekillendirilmesi çalışılmış ve işlem sonucu sacın mekanik özellikleri ve içyapısı incelenmiştir. Maliyet ve işlem süresi de düşünülerek farklı sıcaklık ve ısıl işlem süreleri maksimum mukavemete sahip sacı bulabilmek için uygulanmıştır. Böylece; araba panellerinin şekillendirilme ihtimallerini görebilmek için sektörün performans ve güvenlik gereklilikleri, maliyet ve enerji tüketimi ve çevresel etkileri düşünülerek uygun işlem parametreleri araba gövde panel uygulamaları için tanımlanmıştır.

**Anahtar Kelimeler**: Alüminyum alaşımlı sac, 6000 seri, ısıl işlem, araba gövde panel uygulamaları ve mikro yapı.

To my beloved Sevimli’s family

Alaettin, Ayten,Nurgül

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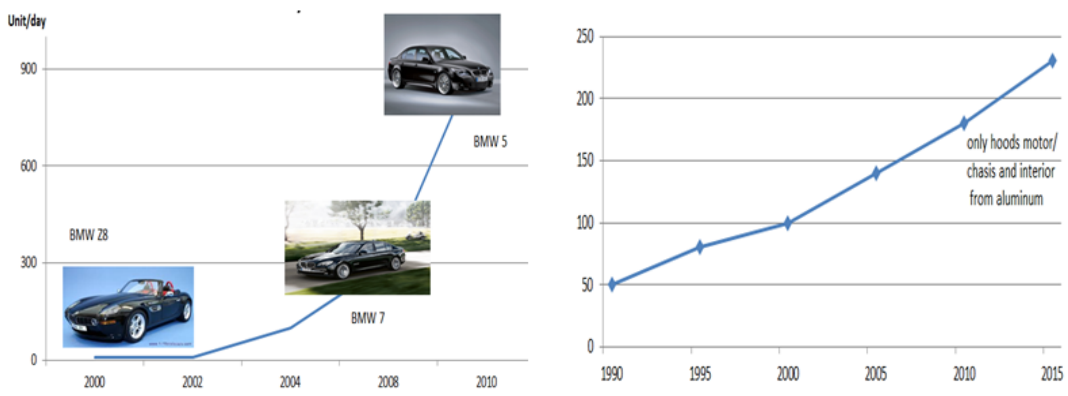
# CHAPTER 1

# INTRODUCTION

## 1. INTRODUCTION

While technology is progressing and time is passing, the requirements, desires and expectations of people are unbelievably increasing. To satisfy the customer expectation, demand of collusion safety, passenger protection, assembly of technological devices, drivability, ride quality, additional accessories and comfort the cars are the factors which leave the vehicle no choice but become heavier. On the other hand, there is a direct connection with weight of vehicle and environmental issue. As the weight increases, the CO and $NO_x$ emission is increasing too [1], [2]. Nowadays, there is a great competition. Environmental requirements, cost, quality and power consumption ratio are the primal issues for modern industry. Especially, environmental necessities are paid attention since Kyoto Protocol in 1997 [3]. These environmental and safety issues have forced the application and research in lighter and safer materials for automotive production. Even if weight of car is decreased 100 kg, it is obtained chance of 1 km/l [4]. Firstly, as innovative car concept thinner steel-sheet body panels by increasing the strength of the steels are used to decrease the weight of automotive. On the other hand, the thickness cannot be reduced more. This was limited because while diminishing the thickness, it causes decrease in stiffness, too. In that case, decreasing stiffness creates a problem. Hence, alternative materials are tested in many parts of automobile instead of conventional materials for weight reduction such as; high strength steels, aluminum alloys, magnesium alloys and polymers. Among of automotive manufactures, especially aluminum alloys are under spot light since, it is major candidate.

Using aluminum alloys have started in engine part as little casting pieces in high class car and racing car. Then, the first time for preferring aluminum alloys in manufacturing car body was 1920s [5], [6]. However, preferring aluminum has been challenged by improvement in technology about automobile industry in last several years [7]. According to research, during manufacturing of more than 2.5 million vehicles, aluminum alloy panels are consumed every year. Today, this number increases with technological improvements. Graphics in figure 1-1 [8] show that use of aluminum in door and closure in product of German company, BMW and Western Europe is increasing year by year.




(a) (b)

**Figure 1-1**: Aluminum car body panel usage of BMW & a. Western Europe b. Company by years [9], [10].

Aluminum alloys are widely preferred for various parts of the automotive through allowing weight saving without compromising safety and performance. Moreover, it has advantages as especially low specific gravity and other better mechanical properties such as high strength/ weight ratio, formability, having protective natural oxide film, weldability, corrosion resistance etc. [11]. Now, today aluminum is used any part of the automobile such as engine block, hoods, panels, trunk, chassis, hang on parts, frames and doors etc. in industry. Contemporarily, cars made from aluminum are able to be manufactured. For instance, the heat treatable 6XXX series aluminum alloy as outer body panels of cars and light trucks, 5XXX series as inner part of the panels, engine blocks are preferred. In figure 1-2, car body panel which are made from 6xxx series aluminum are shown.

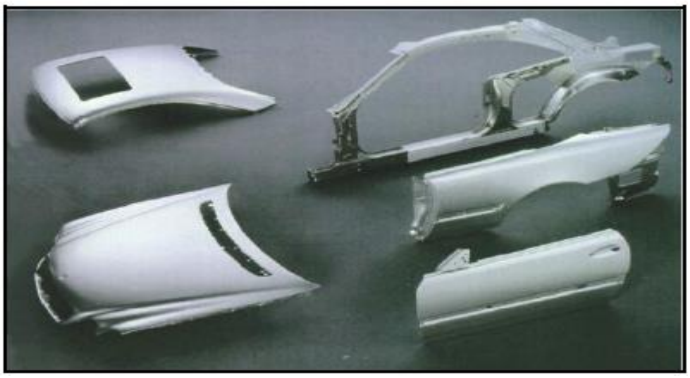


**Figure 1-2**: Car body panel parts made from 6xxx series aluminum alloy [8]

6016 aluminum alloy is used in manufacturing body panel of Audi A8 [12]. In the North America, AA6111 aluminum alloy has been used especially for outer panel of car up to 1.mm thickness [13], [14]. Jaguar Company, the largest single aluminum body part manufacturer in the world, provides 2,8kg/ vehicle and about 40 pound/ vehicle piece cost saving by designing aluminum body part [15]. In Europe, 6016 having better formability and corrosion resistance but less bake hardened strength is preferred up to 1.2mm thickness [14]. The companies preferred aluminum with application parts are listed below in table 1-1.

Most of aluminum alloy sheets with 0.8 mm to 1.3 mm thickness used in car body panel manufacturing. Forming of sheets is related to chemical composition, mechanical properties, grain size, grain morphology, distribution of precipitation, precipitation mechanism and ageing conditions [16]. 5xxx and 6xxx series aluminum alloys are widely preferred in automotive industry. 5xxx series have 125 to 350 MPa tensile strength, good formability and corrosion resistance. For car body panel usually 6000 series are preferred. Because by heat treatment process it is easy to obtain convenient strength. 2xxx, 7xxx are the other series which can be strengthened by heat treatment. But, corrosion resistance of 6xxx aluminum alloy is better than 7xxx and 2xxx series. [Al-Si] alloys have relatively better castability [17].

**Table 1-1:** Company preferred aluminum with application parts [4], [10] [18].

| COMPANY | MODEL | ALUMINUM PARTS | COMPANY | MODEL | ALUMINUM PARTS |
|---|---|---|---|---|---|
| TOYOTA | Crown/MJ | Hood | MERCEDES BENZ | 300SL Gullwing | All part |
| | Prius | Hood back-door | | CL coupe | All part |
| TOYOTA [Lexus] | LS | Hood | JAGUAR | D type | All part |
| | GS | Hood | FORD | GT40 | All part |
| | SC | Hood, Roof | AUDI | A8 | All part |
| | IS | Hood | BMW | Z8 | All part |
| DAIHATSU | Copen | Hood, Roof | FERRARI | 360 Modena | All part |
| | Fuga | Hood, Door, Trunk Lid | SUBARU | Legacy | Hood, Back door |
| | Cima | Hood, Trunk Lid | | Inpressa | Hood |
| | Skyline | Hood | | Forester | Hood |
| | Stagea | Hood | MAZDA | RX-8 | Hood ,Rear door |
| | Fairlayd Z | Hood | | Roadster | Hood, Trunk Lid |
| HONDA | Legend | Hood, Trunk lid, fr fender | MITSUBISHI | Lancher Evo | Hood, Roof, Trunk lid |
| | S2000 | Hood | | Pajero | Hood |

They have medium strength and their strength can be enhanced with addition of Mg, Cu and Zn elements providing heat treatment. Plus, they have formability and weldabilty properties. Besides, 6xxx series are more preferable in case of economical and safety issues. They have lower cost, higher dent resistance and higher strength with respect to 2xxx and 7xxx series [19]. Their tensile strength is between 125 to 400 MPa. 6xxx series aluminum alloy has large amount of intermetallic formed during cooling processing and ageing. Strength is derived from homogeneously distributed precipitations during heat treatment which significantly concern about their useful property. Plus, 6111 aluminum alloy grade is most suitable in case of being formed easily without flaw. Besides, 6xxx series has ultimate strength; it has better weldability compared to 5xxx series. Moreover, 6111 aluminum grade has better surface appearance which is more important point for automotive application due to aesthetic requirements. In contrast, in 5xxx series aluminum alloy is sensitive to occurring Lüders band defects during rolling. Therefore, 5xxx series are usually preferred in non-visible and especially produced by extruded parts such as inner door, inner panel.

After completing forming parts of car, some parts which need painting are applied to painting and after that to baking manufacturing process. While heat treatable strength of 6xxx series alloy and dent resistance increase and as a result of the process, 5xxx series alloy has negative effect and they become softer because 5xxx series are non-heat treatable. In brief, 6xxx series are favorable due to combination of better surface quality after forming, suitable strength values after paint baking and availability of hemming and bending processes. All these show that controlling the production is the way to produce desired materials.

Considering all these parameters, the aim of this study is to find suitable material for providing decrease in vehicle weight by examining the production and forming of 6111 aluminum alloy sheets for car body panel. It is taken advantages of heat treatment to obtain desired mechanical properties for panel parts. The deformation effect during rolling process and effects of grain structure after heat treatment is analyzed by microscope and heat treatment effects were determined.

Basically, production of heat treated 6111 aluminum alloy sheet can be completed with 4 steps:

I. After defined the suitable composition as 6111 aluminum alloy, the plate were cast.
II. Later, firstly hot rolled then cold rolled to the intended thickness at convenient condition.

III. Then, to get the desired strength and formability, T4 and T6 heat treatments were applied.

IV. Finally, evaluation of effects of heat treatment on properties and microstructure were determined.

In this study, firstly AA6111 aluminum sheets with nearly 1mm thickness were produced. Then, sheets were solutionized and water quenched for heat treatment. After all, performance of microstructure and mechanical characterization of sheets were examined. In the 3$^{rd}$ step, precipitation hardening mechanism works. Therefore, microstructure and size, density and condition of precipitates have important effects on mechanical property and forming of car body panels. The purpose of this study is by varying process parameters the formation of car body panel and then to investigate the outcome of process. Hence, the convenient process parameters for designed aluminum sheet will be determined.

This thesis consists of five main chapters that give information about the topics covered in the study. Chapter 2 includes theory and literature review of general knowledge about aluminum alloys and 6000 series aluminum alloys. In chapter 3, experimental procedure expressing all of the steps which are followed for producing sheets and applying heat treatment were explained in detail. Moreover, in Chapter 2 & 3, concept of heat treatment and process parameters' effects on material will be mentioned. Chapter 4 presents the experimental results which include all mechanical results and observation of microstructural characterization were presented to state the method and outcomes. Finally, conclusion is given in Chapter 5.

# CHAPTER 2

# THEORETICAL BACKGROUND

## 2.1 Aluminum and Aluminum Alloys

Aluminum purified in 1888 is soft and ductile. Extraction of aluminum from ore is difficult. Therefore, aluminum has not been classified as economical and industrial materials until Charles Martin and Paul T Heroult have studied about electrolytic reduction method of aluminum oxide ore [20]. On the other hand, commercial uses need more strength because pure aluminum has just about 90 MPa tensile strength. Therefore, cold working makes tensile strength doubled at the same time, aluminum is combined with other elements to be strengthened and hardened so it can be used in many applications. Aluminum alloys are preferred in many application areas such as structural aircraft, storage tanks hydraulics tubing and automotive components owing to their properties [21]. Examples of application area according to types are listed in table 1-1. In addition, aluminum includes important and effective properties and characteristic for automobile applications. That is why, Pierce Arrow produced car by using aluminum in the mid-1920s [22].

There are many reasons of widespread usage of aluminum [5], [20]

- Aluminum has low density. This makes aluminum have one third of steel weight and makes it 30% lighter than copper. After magnesium, it is the lightest metal among common metals. As well as, it has high strength to weight ratio. Therefore, it is a key material for especially aircraft and automobile applications. For instance; if car is made from aluminum, nearly 300 kg weight can be decreased compared to steel. This rate is equal to 30% of total car weight. Moreover; especially in the structural application 1.2 mm aluminum sheet can be preferred instead of 0.8 mm steel sheet. This means that almost 50% of weight reduction can be obtained [23].

- Not only high ductile and low strength but also tough and high strength alloy types can be produced by applying heat treatment according to application areas.
- Aluminum alloys are widely preferred in marina applications owing to its corrosion resistance. Because, it has thin oxide films being formed on the surface in natural environment. Even aluminum materials are not painted or coated; they are durable to corrosion problem due to water or salt on the road.
- Having high elasticity provides advantages under suck load. Besides, toughness of aluminum is 3 times greater than steel.
- Rolling aluminum sheet to very thin thickness is easy. Owing to having lower yielding strength, aluminum can be formed easily.
- It can be used for food containers and packages owing to nontoxic properties.
- It can stay tough even at low temperatures.
- Furthermore, aluminum alloys have a great market thanks to good electrical and heat conductivity, recyclability and ability of mass production. Owing to recyclability, used and beaten wasted aluminum parts are recycled at about 80% rates. Recycling is applied at relatively low energy cost. Strength and tendency to light weight vehicles enhance this position and preferring aluminum become widespread for designers and manufactures [20].
- On the other hand, aluminum has some weaknesses such as low melting point, strength, toughness and fatigue resistance, high tendency for wrinkling and cracking and higher cost compared to steel. It has worse properties with compared to steel which is summarized in table 2-1.

**Table 2-1:** Comparison of aluminum and steel [20]

| Properties | Al alloys | Low alloy steel |
|---|---|---|
| Density, ρ | 2.7 g/cm3 | 7.8 g/cm3 |
| Young Modulus, E | 70Gpa | 210Gpa |
| Yield Strength, $\sigma_y$ | 100-600 Mpa | 500-1900 Mpa |
| Tensile Strength, $\sigma_{UTS}$ | 300-700 Mpa | 680-2400 Mpa |
| Ductility $\varepsilon_f$ | 0.05-0.3 | 0.02-0.3 |
| Fracture Toughness | 23-45 Mpa√m | 50-150 Mpa√m |
| Cost | 1 £/kg | 0.5 £/kg |

### 2.1.1 Alloying Elements and Their Effects

Pure aluminum is upgraded by adding alloying elements. Alloying elements join the crystal structure as solid solution or second phases. This thermodynamics occur under the sway of surrounding condition such as temperature, diffusion, composition kinetics of nucleation and growth mechanism. Working parameters are the factors which are guides to determine forming and distribution of second phases, microstructure, properties and behavior of alloys.

Generally, alloying elements have low solid solubility in solid Al. Zinc is the maximum soluble element in the solid solution of aluminum alloy up to 70%. Besides, 1.65% silver, 17.4% magnesium, and 1.82% copper are the other important soluble elements in aluminum [24]. There are major and minor alloying elements. Major element have basic role alloy to gain such as strengthening, strain hardening, precipitation or age hardening. Minor elements such as Ti, B, Cr etc. usually have low solubility and forms coarse intermetallic [25], [8]. Some of these elements form substitution solid solution in aluminum matrix and have coherent boundary as seen in 6000 series aluminum alloys and/or partially coherent boundary with aluminum lattice.

Each of alloying elements is added to meet specific properties of materials. While aluminum alloys having strength and other properties like grain refining, machinability corrosion resistance, stress corrosion cracking, conductivity, density etc. are achieved by adding elements widely used such as copper, magnesium, zinc, silicon, manganese etc., other alloying elements such as bismuth, brome ,chromium, nickel, titanium, sodium etc. are important for end of users.

Alloy composition has operative performance effect under working and environmental conditions. Materials gain properties such as strength, sensitivity to corrosion or weldability properties vary according to addition of alloying elements. The desired tensile strength should be optimized with ductility, corrosion behavior under working condition, weldability, forming ability, thermal working condition for application areas. Therefore, the first basic parameter which is needed for characterization is composition of alloys.

Elements have high solubility at high temperature while low solubility at lower temperature. These elements are annealed at sufficient temperature to be solved completely then, cooled rapidly enough to be dissolved and formed solute atoms homogeneously distributed.

- **Effect of Silicon and Magnesium Content**

Mg and Si are the key elements for 6000 series aluminum alloys. If Si is not incorporated with Al matrix or Al-Fe-Si intermetallic, it has chance to combine with Mg with Mg/Si proportion as 1.73 to form $Mg_2Si$ precipitates during ageing in several forms. Alloy had excess amount of Si dispersion provides work hardening rate and elongation increment, alloy having excess amount Mg effective in corrosion resistance and raises the recrystallization temperature as well as makes the flow stress increase. On the other hand, as $Mg_2Si$ and Si precipitate at grain boundaries, these cause dislocation pile ups. Precipitates retard grain growth and also effective in grain refinement [26]. During rolling process, Si precipitates through grain boundaries cause intergranular fracture [5], [14].

According to Grupta et al research, excess Si have effect on strength of T4 and T6 tempers by providing uniformly distributed β’’ particles [5].

- **Effect of Copper**

Cu improves the precipitation hardening kinetics and grain refinements with incorporation of Mg and Si combination [4]. Thus, it affects the hardness and refines microstructure [5]. Increase in Cu makes alloy heavier while weldability and corrosion resistance affected negatively.

- **Effect of Iron and Intermetallic**

  Fe combines with Si and Al to form intermetallic. Intermetallic has no effect on strength however, they are important during forming steps. Besides, iron has negative effect on conductivity.

- **Effect of Chromium and Manganese:**

Addition of Mn and Cr inhibit the precipitation of $Mg_2Si$ and Si on the grain boundaries and coarse grain growth. Therefore, brittleness and fracture toughness may be controlled with addition of Cr and Mn owing grain refinement effects and by preventing precipitates of Si on grain boundary as adding Mn. Besides, Mn makes deformation homogenize and reduces the stress concentration at grain boundaries [5]. Mn has positive effect on corrosion resistance [14].

Ti and B elements are added as grain refinement during solidification.

Global, an international system is generated for designation of identifying wrought aluminum alloys to give information about alloying elements content and application area with regard to U.S. Aluminum Association (AA) Standard. By helping the four digits, alloys are taken the identification. First digit is used for determining the alloy group. Second digit determines the impurity limit. The last two digits determine the aluminum impurity. Prefix and suffix letters shows the incorporation with additional activity. If a letter used as prefix, it shows experimental condition. If it is used as suffix, it shows national variations. Using letter prefix gives information about specific treatment applied. Basic ones are listed below as table 2-2 taken from European Standards EN 515 (1993).

**Table 2-2**: Temper designation [14].

| Prefix | Explanation | Prefix | Explanation |
|---|---|---|---|
| **F** | As fabricated | **W** | Solution heat treated |
| **0** | Annealed | **T** | Stable temper |
| **H** | Strain hardened | **T4** | Solution heat treated and naturally aged |
| **H1** | Only work hardened | **T5** | Cooled from an elevated temperature and shaping process and then artificially aged. |
| **H2** | Work hardened and partially annealed | **T6** | Solution heat treated and then artificially aged. |
| **H3** | Work hardened and stabilized | | |

**Table 2-3:** Aluminum alloy types are accepted by worldwide designation: [27], [5]

| Al Alloys | Containing Elements | Application Areas |
|---|---|---|
| **1xxx** | <1% addition | Sheet tube foil, food packing trays, electrical bus bar. |
| **2xxx** | Cu,Mg,Li | Aircraft structures, structural beams, fuel tanks and booster rockets of Space shuttle internal railroad car structural members |
| **3xxx** | Mn,Mg | Cooking utensils packaging, chemical equipment, automotive radiator heat exchanger |
| **4xxx** | Si | Cooking utensils pump casing, automated welding of an auto body structure |
| **5xxx** | Mg | Electrical wire drink can tops Marina structures boats, pressure vessel |
| **6xxx** | Mg,Si | Architectural structures, portable bridges, car body panels and space frames |
| **7xxx** | Zn, Mg, Cu | High strength and tough aircraft structures, car bumper high speed ships welded structure |
| **8xxx** | Li, Mg,Cu | Helicopter parts, packaging, microelectronics, architecture, lithography industries [28]. |

## 2.2 Heat Treatment and Hardening Mechanisms

Chemical composition and production technology are arranged according to mission where it will be used as indicated above in examples in table 2-3. Additional elements have important role in strengthening. For instance, although casting alloys have lower strength, toughness strength can be improved by heat treatments with applying precipitation mechanism of Cu and/or Mg or by work hardening mechanism. Additional elements and their strengthening mechanisms are summarized in figure 2-1.

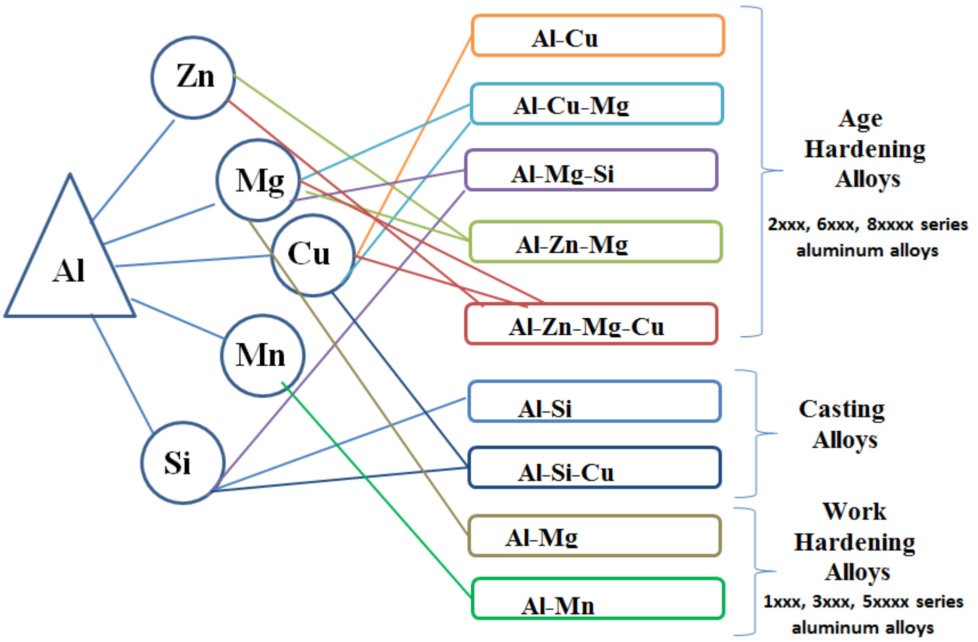



**Figure 2-1**: Additional elements and strengthening mechanisms

For wrought aluminum alloys, one mechanism for improving strength is work hardening. It is a non-heat treatable application. During forming, deformation of material causes dislocation movement and also new dislocation formed. During dislocation movements, dislocations meet and make tangle due to increment of density. This reduces the dislocation spacing in the plane and as well as mobility of dislocations. Thus, more energy is needed for providing dislocation motion and by this way, yield stress is improved. 1xxx, 3xxx and 5xxx series aluminum alloys are hardened by work hardening.

Moreover, while dislocation is moving in the matrix, it meets solute atoms such as particle. Solute atoms in matrix block the motion and make dislocation motion harder. Namely, higher density of these particles, harder motion of dislocation is. Dislocation shear and cut the particle and keep on moving or cannot break so it loops the particle. Thence, there is more energy requirement for make dislocations move. Therefore, yield stress increases. These mechanisms are simulated in figure 2-2. When the precipitates are small and soft, dislocations shear particles when they are hard and larger, dislocations bow or make a ring around particles. In the Orowan mechanism, alloy strength decreases as the particle size increases. The other non-heat treatable process for strengthening is solid solution strengthening. Therefore, over ageing causes decrease in strength in alloy. This is seen especially in 3xxx and 5xxx aluminum alloy series.

Size, shape, density and distribution of second phases are related to mechanical properties of aluminum alloys. As result of rolling and heat treatment, precipitates processes are formed, grew and distributed. Therefore, according to physical condition of precipitates, the energy needed for making dislocation movement changes. It is a heat treatable process for strengthening and is seen in 2xxx, 6xxx and 8xxx aluminum alloy series.

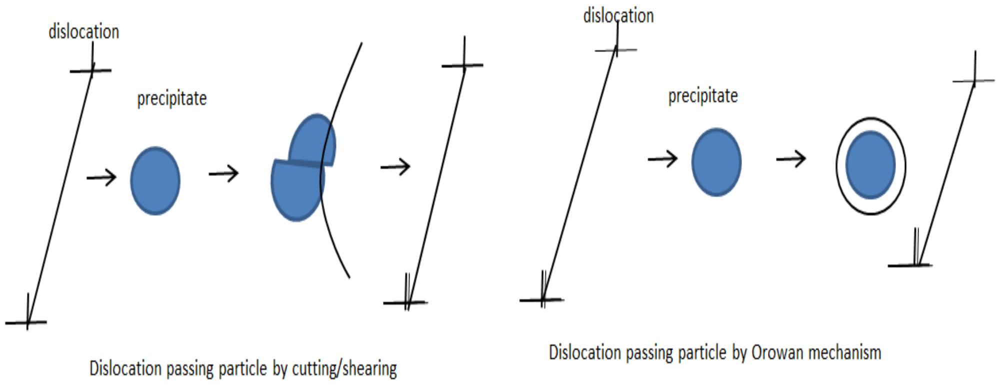



**Figure 2-2:** Dislocation passing mechanism

Both 6xxx and 5xxx series have high formability. But, 5xxx series are hardened by strain hardening mechanism. They are non-heat treatable alloys and so after annealing the strength of 5xxx series decreases. Therewith, car body panels are needed to be applied to paint baking process after shaping.  During paint baking, the strength value decreases. On the other hand; 6xxx series are heat treatable alloys and strengthened by precipitation hardening mechanism. Owing to mechanism, higher strength and stain values are obtained. Strengthening by heat treatment advances their application areas on automobile parts [6]. They are especially preferred for outer body part owing to mechanical and physical sufficiency.

Heat treatment thermodynamic works as changing the solubility and stability of the alloying elements which form intermetallic according to condition of material. Aluminum alloys are heat treated when material is subjected to heating to elevated temperature and cooling rapidly as quenching or naturally or artificially ageing. Solution heat treatment, quenching and age hardening process are the steps for materials to be strengthened and/or hardened.

### 2.2.1 Homogenization

Homogenization is performed after melting and casting process at temperature in between 460 to 550 $^0$C. Purpose of homogenization is to decrease segregation, dissolution of unstable phases and precipitates, precipitation of supersaturated elements occurred during casting, grain growth and so that obtaining homogenized composition in the material after melting and casting processes [14].

Alloying elements have different solid solubility and diffusion rate in Al matrix. For instance, Cr, Mn, Zr have low diffusion rate, Fe has low solid solubility, Cu, Mg and S, have high solubility and diffusion rate.

The higher solutionizing temperature, the higher diffusion rate is [5]. However, the temperature should be in the optimized range for improvements in mechanical properties. Time and temperature are important parameters for solutionizing. If solutionizing temperature is too high, overheating may cause cracking or local melting process. In addition, the mechanical properties such as ductility, tensile strength and facture toughness might be affected negatively [5]. On the other hand, desired mechanical properties cannot be reached by too low solutionzing temperature. The alloying elements may not be dissolved at lower temperature and short time. Moreover, increasing time of solutionizing causes coarsening of grain size and precipitates so the mechanical properties affected in negative way.

The other basic parameter for solutionizing is time. If the time is longer, hardness values decrease due to coarsening of Si, dissolving of intermetallic and increasing space of intermetallic [17].

Alloy, chemical composition, desired mechanical properties and thickness are important factors on the time required for solution heat treatment [5].

At the end of solution treatment, metastable phases decrease, having low melting phases dissolved and so hindered grain growth by fine dispersoids is eliminated. Thus, grains have chance to growth and ductility is enhanced. Finally, uniform microstructure is observed.

### 2.2.2 Quenching

Cooling from elevated temperature to room temperature causes super saturated solid solution to form. Aluminum alloy is plunged into water for achieving rapid cooling to derive more fraction of hardening elements in solid solution as supersaturated form at room temperature and provide vacancies [29]. Optimized combination of rolling and rapid solidification make mechanical properties enhance by extension of solid solubility limit in Al lattice, providing grain refinement and formation of metastable intermetallic phases [28]. Rate of cooling is major parameters for mechanical properties. Slow cooling rate provides residual stress however; it provides distortion of particles, growth of grain, and possibility of corrosion tendency and decrease the benefit of ageing due to over ageing effect. Desired microstructure is obtained by rapid quenching for the optimized mechanical properties consisting of ductility and strength.

### 2.2.3 Age Hardening

After homogenization and quenching process, matrix involves supersaturated solute atoms and vacancies. Supersaturated solid solution is formed at room temperature. During waiting at room temperature or ageing at elevated temperature where is below line of GP zones, non-equilibrium metastable phases as GP zone and hexagonal β double prime and β prime phases, semi coherent or intermediate precipitates are generated with aluminum lattice which are the source of hardening process. Ardel is the first man who mentioned and found about precipitation hardening in 1906 [30]. As Ardel emphasizes, clusters of atoms in supersaturate matrix come together and forms coherent GP zones first. The atoms are coalescence by diffusion so precipitates grow by diffusion of atoms. This ageing process includes nucleation, growth and coarsening steps.

Ageing processes influence material behavior and ability of forming while fraction, size and distribution precipitates identify age hardening effect [29]. Improvement in tensile stress, decreasing in residual stress, and stabilize microstructure can be regulated by ageing process. The working condition for process is room temperature for natural ageing and coherent clusters (GP zones) form in the aluminum matrix. The process condition for artificial ageing is between 90-260 $^0$C temperatures. Artificial ageing can occur even below meta-stable miscibility gap where it can be seen as GP zones (Guinier-Preston zone) below solvus line. GP zone is shown as cluster part in ageing time vs temperature graphics of figure 2-4.

Precipitates in the matrix are obstacle for moving dislocations. While small and not too hard precipitates are able to be sheared for moving dislocations, large precipitates are more difficult for the moving of dislocations by passing precipitates as bowing, or leaving a dislocation ring around the precipitate [31]. This mechanism is shown as schematically in figure 2-4. As the obstacle become larger, the strength increases [30].

Heat treatable alloys are hardened by waiting at normal, room temperature. Time for hardening can be decreased by increasing ambient temperature in the range 100-200$^0$C. Time and temperature vary depends on the alloy composition. Results of ageing configured by size, distribution and coherency of precipitates .As Mohammed and Samuel indicates, Al-Si-Cu-Mg or Al-Si-Mg alloys is affected on hardening than Al-Si-Cu alloys [30].

## 2.3 6xxx Al-Si-Mg Alloys

Magnesium and silicon are the major elements of 6xxx series aluminum alloys. Thus, it can be strengthening with combination of Mg and Si as $Mg_2Si$ with Mg/Si ratio as 1.73 by precipitation hardening. However, copper, manganese, chromium and iron are the other alloying elements in the composition as transition metals. [5]. They are not able to form new phases. These transition elements are affected in selection and volume fraction of intermetallic phases.

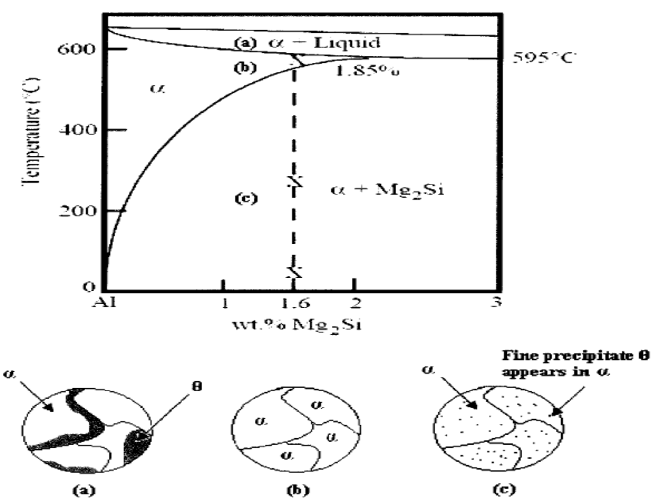



**Figure 2-3**: Al-$Mg_2Si$ phase diagram [5]

During homogenization, the temperature is increased above liquidus line. At close to homogenization temperature (550-600 $^0$C), all precipitates and intermetallic are dissolved in Al matrix and solid solution phase are obtained. There is single phase called as α phase is formed. Solid solution phase has FCC structure. After that, quenching is done and supersaturated solid solution is obtained. It contains Al matrix and precipitated intermetallic. Then, the solution comes to equilibrium situation with ageing as second phase's precipitates out over time [21], [32]. The chemical composition, casting and rolling parameters and heat treatment conditions are the factors which change the morphology of the precipitates.

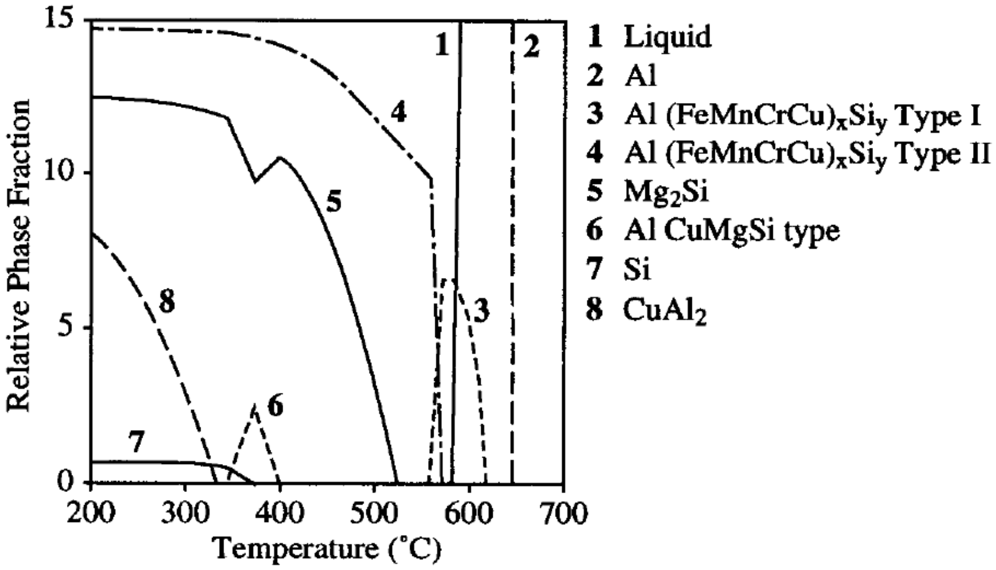



**Figure 2-4**: Equilibrium phases and amount of 6111 Aluminum alloy at different temperatures

Homogenization makes $Al_2Cu$, AlCuMgSi and $Mg_2Si$ phases melted. Then, AlFeSi precipitates combines with Mn and formed ALFeMnSi phases. According to homogenization temperature transformation and dissolving occur as seen in figure 2-4 [33]. During cooling especially in the direct chill casting there is cooling rate variation from edge to center. So that, intermetallic phases with different types and morphology are formed at different positions depending on the cooling rate and composition. These differences identify the materials characterizations. $(Fe,Mn)_3SiAl_{12}$, $Mg_2Si$, $(Fe,MnCu)_3SiAl_{12}$, intermetallic phases of wrought AA6xxx series alloy [5].

The precipitation sequences start with α [SSS] after quenching. GP zones called as clusters keep on progressing as spherical. These spherical GP zones come together and make β'' precipitates occur.

Precipitates occur in several forms as during stages of ageing. β'' have rod shaped. It is the smallest type and provides strength as dispersing densely. β combines and forms β' precipitates. It has same rod shape but larger character as β''. β' has cube shape.

α supersaturated solid solution → [SSS] → GP zones → needle like β'' ppts rod like β' → β [ppts] [34].

α supersaturated solid solution → [SSS] → GP zones → needle like θ'' ppts rod like θ' → θ [ppts] [33].

GP: Guinier-Preston zones

β=equilibrium $Mg_2Si$

θ=equilibrium $Al_2Cu$

β', θ' & β'', θ'' =metastable precursors of β and θ

At the first stage of aging, Mg, Si and Cu atoms resolve from solid solution and then form clusters by diffusion from supersaturated solution. By time and/or at high temperature stage clusters come together and form GP zones and keep on growing. Temperature, remain of supersaturated solute atoms are the parameters which effect the clustering reactions [35]. These GP zones convert into metastable phase. In figure 2-4, stages of phases with temperature and time are shown in the cooling diagrams and solubility diagrams of alloys. In case of end of super saturation situation, precipitates keep on getting larger by joining as well in Oswald Ripening mechanism.

When β', θ' are formed, coherency of precipitates transform to incoherency form. β and θ are incoherent with matrix. Therefore, long range coherency strain field make strength decrease while ageing continues.

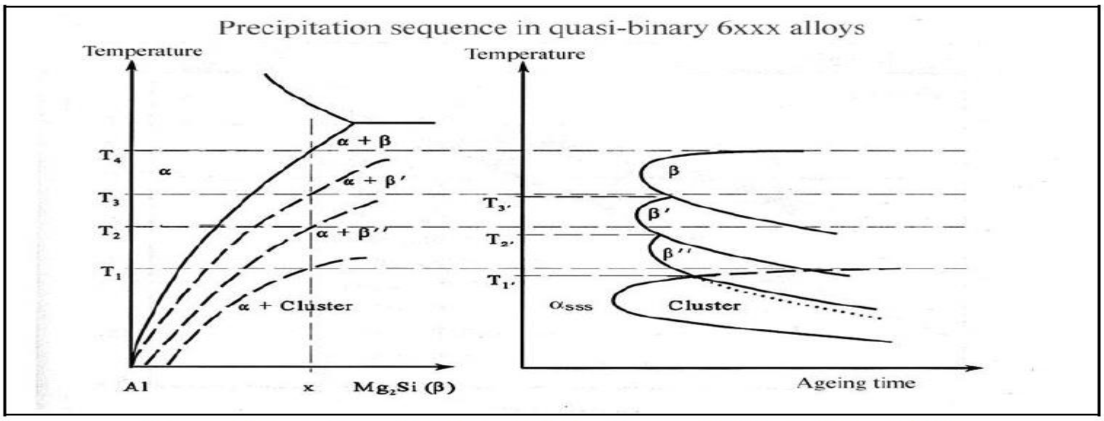
- **Strengthening in 6xxx**

Strengthening of 6xxx series aluminum alloys have 3 steps [36]:

-solution treatment: dissolving of second phases and intermetallic and so forming solid solution.

-quenching: forming supersaturated solid solution

-ageing: precipitating of unstable phases at room temperature or elevated temperature called as precipitation hardening.




**Beta solvus curves (left) and TTP curves (right) in 6xxx alloys**
**Source: G. Huppert-Schemme, 1997**

**Figure 2-5**: Precipitation sequence of 6xxx aluminum alloys [8].

Experimental procedures of hardening aluminum alloys have hard and complex content. Stable and metastable phases precipitate during aging and growth according to temperature and holding time. They cause lattice distortion and impede dislocation motions. Hence, extra force is required for motion of dislocation. Besides, stress and hardness increase with aging [5]. According to Pythagorean addition law (equation 2.2), strength is calculated as sum of initial stress, solid solution stress and square root of stress occurred from obstacle formed by these clusters and precipitation.

$$\sigma_{ys} = \sigma_i + \sigma_{ss} + \left(\sigma_{cluster}^2 + \sigma_{ppt}^2\right)^{1/2}$$ [2.1]

Besides chemical composition, hot and cold working processes, annealing and ageing steps are the craters for determining the AA6xxx aluminum alloys properties. For example, hot rolling is applied at close to melting temperature. Thus, less forming load is needed and forming is easier.

Moreover, size and density of precipitation formed during heat treatment are important factors for strength. The distribution and size are affected by heat treatment condition and composition of alloy as alloying elements. However, the formability of alloy is related to size and distribution of precipitations. For instance, growing of intermetallic as $Al_3Fe$ eutectic fibers' size causes cracks and notches and this have a bad effect on formability and fatigue resistance.

# CHAPTER 3

# EXPERIMENTAL PROCEDURE

Working parameters have been being developed for producing AA6111 aluminum sheets for car body panel applications. After producing sheets, heat treatment parameters involving solutionizing and ageing temperatures and duration time with quenching conditions were developed to obtain enough strength for car body panel. The mechanical responses according to working condition were evaluated and working parameters were set through the test results. Forming behavior was determined by mechanical properties and influence of storage condition.

There were two basic scenarios to see more convenient way for forming the sheets with desired mechanical properties. T4 temper as the first scenario and T6 temper with different time and temperature as second scenario were applied.

One of the aims of this study was to determine the optimum processing conditions of high strength, high formable aged aluminum sheets.  Microstructure examinations of 6111 aluminum alloy after rolling and heat treatment were studied by optical microscope and scanning electron microscope. Grain distribution, grain boundary and grain size were examined, precipitations and intermetallic were analyzed by microscopic studies and mechanical testing. The analyses were utilized to determine behavior of material after T4 and T6 heat treatments on mechanical properties and microstructure.

# 3. EXPERIMENTAL PROCEDURE

## 3.1 Casting 6111 Aluminum Alloy Plate

Casting is primer basic step for obtaining and controlling desired properties of final product. Semi continuous direct-chill (DC) casting method was currently preferred in producing aluminum alloys.

Firstly, the elements and amounts were determined as indicated in appendix A. Nearly, 2.7 kg mixture were composed. Pure aluminum alloy and additive elements were melted in furnace at around $650^0$C so that each of elements indicated in table 3-1 was able to melt at this temperature and mixed homogenously.

**Table 3-1**: Chemical Composition of 6111 aluminum alloy.

| 6111 Alloy | Chemical Compositon (wt%) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Si | Fe | Cu | Mn | Mg | Cr | Zn | Ti |
| | 0,6-1,1 | 0,4 max | 0,5-0,9 | 0,1-0,45 | 0,5-1 | 0,1 max | 0,15 max | 0,1 max |
| | 0,82 | 0,11 | 0,84 | 0,45 | 0,60 | 0,01 | 0,05 | 0,08 |

AlTiB has important effect to obtain fine equiaxed solid α crystal grain structure, more uniform distribution and to decrease the possibility of meeting failure such as porosity or cracks during casting. When grain refiner is not added, the structure has columnar grain at exterior and equiaxed grain just only in the center [37].

Then, mixture was poured through a rectangular form usually copper mould with 6.7 mm thickness with typically 1-3 mm/s velocity. Advantage of copper mould is higher thermal conductivity. It is needed for high cooling rate to form fine intermetallic. During pouring the mould stayed with angle ($\alpha$=40-55 $^0$) as shown in figure 3-2 to prevent the casting hole through providing gas output while pouring and so to provide cast successfully.

Microstructure is affected by cooling rate so mechanical properties vary due to different rate in different location of ingots. Besides, according to rate of cooling, morphology of grain, dendritic arm spacing, grain size and intermetallic were formed. Hence, each of these variables identified the mechanical properties of as-cast form.

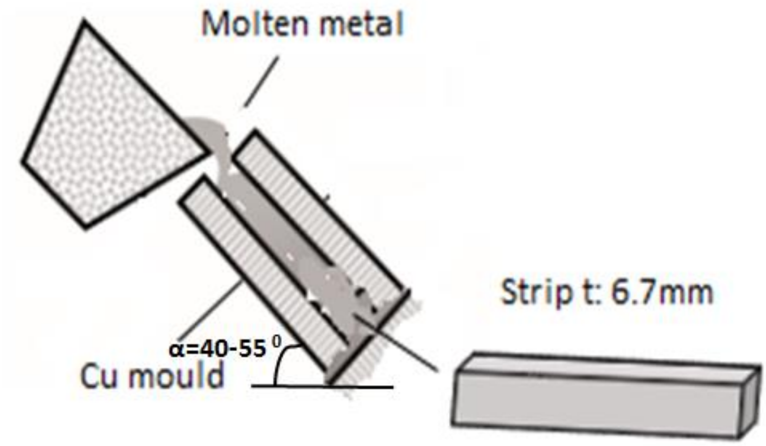



**Figure 3-1:** Casting process

## 3.2 Rolling of AA6111 Plates

Rolling process was applied in order to impart specific properties to sheets. Typically, rectangular ingot or slab plates are used.

To implement rolling experiment, two cylindrical roller strips made from mild steel and with a size of 30 cm in external diameter and 80 cm length were used in the foundry and metal processing laboratory. It works with 22 PS KW power. The maximum roller speed was 940 cycle/minutes. Preferred thickness in general for car body panel is approximately and experimentally 1-2 mm. Hot rolled as first and then cold rolled were applied to obtain the desired thickness.

Firstly, aluminum ingot plate was put into the furnace and heated for solutionizing. Before hot rolling, the rollers were heated to about 70 $^{0}$C as seen in the figure 3-3.

The homogenization temperature was more than 550 $^0$C for 4-12 hours [19]. It is a general procedure to heat plate up to higher than $0.5T_{melting}$ during hot rolling. Then, homogenized ingot plate was applied to hot rolling process at temperature around 350-450 $^0$C until the thickness was decreased to 2-3 mm. The temperature of rolling process depends on initial temperature of material and rollers, heat transformation between material and rollers, heat created by friction and surrounding [14].

**Figure 3-2**: Rollers heated to about 70$^0$C to prevent edge cracking

Before feeding the sheets, the gap of roller was arranged as 4 mm. The thickness was reduced by passing through roller. After reaching to about 4 mm thickness, the gap was rearranged as about 2mm and rolling was kept on. Heating roller prevents forming of surface crack by preventing thermal stress. Because, when the sheets touch the roller, crack may occur at edges due to temperature differences [38]. That was the reason of heating rollers before rolling process.

After hot rolling, sheets were annealed at 400 $^0$C for 2 hours. Hence, the marks on the surface would be eliminated. Besides, these lines and marks behave as stress concentration site during forming and increase the possibility of occurring crack. By decreasing marking on surface, cracking was also prevented. Although it is not certain, it is considered as that during hot rolling a band of grains consists of [100]<001> plane form marks, rolling lines on the surface by acting single large grains during deformation. Therefore, grains have chance to recrystallization and homogenization of the randomly oriented grains [39].

Finally, the sheet was cold rolled to 1 mm thickness especially due to necessity of surface appearance. In the end, 6 mm thick plate was rolled to about 1 mm thickness and 69 mm width. Schematic lay out and temperature time graph were shown in figure 3-4. During reduction of thickness, almost %40 CW was applied.

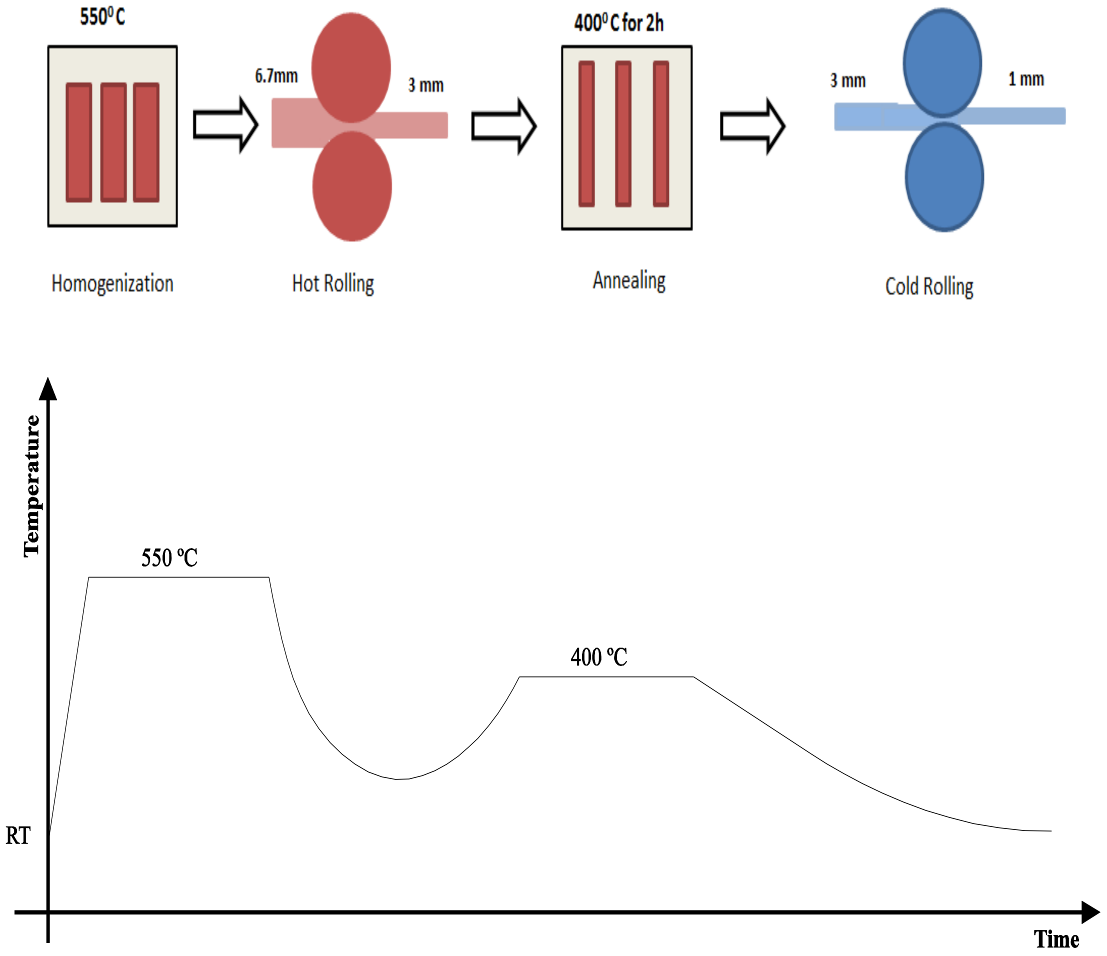
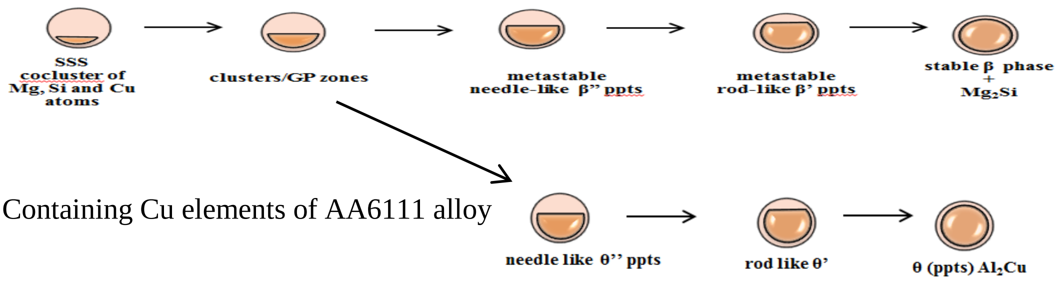



**Figure 3-3**: Hot and Cold Rolling Process of AA6111

## 3.3 Heat Treatment

After rolling process, aluminum alloy was in supersaturated form. It was not a stable form because of that: the excess amount would precipitate and reach the stable form over time. This is called as ageing. The precipitates prevent the movement of dislocation and this causes increment of strength over time. As Lumley et al. mentioned in their studies, mechanical performance of aluminum alloys can be upgraded by arranging secondary precipitates properties such as size, distribution, morphology. [40]. By controlling the size and distribution of precipitates, desired strength was studied [41].

The precipitation of intermetallic for alloy was followed through below sequence as stated in the literature:




Containing Fe elemenets of AA6111 alloy FeAl$_3$ $\longrightarrow$ $\beta$-AlFeSi $\longrightarrow$ $\alpha$-Al[Fe, Mn]Si

**Figure 3-4:** Intermetallic precipitation sequence [34].

During annealing, the all of the precipitates dissolved completely at around 550 $^0$C. Supersaturated phase needs rapid cooling. Cooling rate is important for material behavior. If cooling rate is not sufficient, ductility is reduced due to intermediate phases as seen in the figure 2-4 cooling diagrams. On the other hand, if the cooling rate is too high, residual stress problem may occur.

The sheets were heat treated as condition of T6 and T4 via purpose of material hardening. Firstly, the sheet was heated in the furnace at 530 $^0$C for 30 minutes. Then water quenched was applied. It stayed more than 5 days at room temperature for natural ageing. This heat treatment is called as T4 application. After that, the sheet was put into the furnace again. But this time, the temperature was 160, 200 and 240 $^0$C in addition; it stayed at furnace for 90, 180, 240 and 360 minutes for completing artificial ageing. This application is T6 temper as heat treatment [42]. The lay out for temper and temperature time graph is shown in figures 3-6 and 3-7.

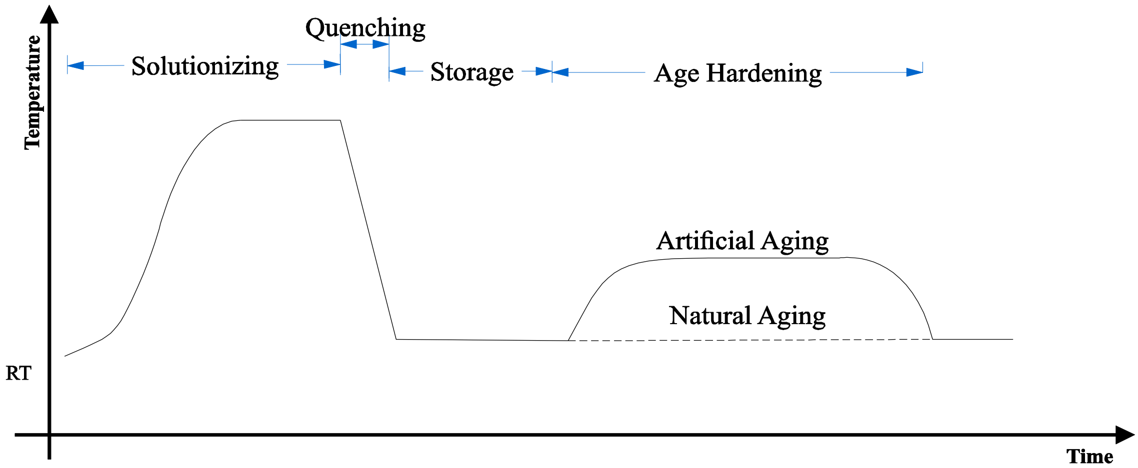



**Figure 3-5**: Temperature vs. time graphics for heat treatment-ageing of 6111

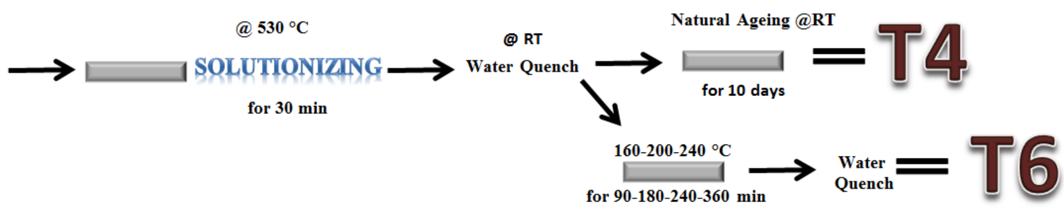



**Figure 3-6**: Lay out for heat treatment-ageing of 6111

## 3.4 Characterization and Mechanical Tests

### 3.4.1 Microstructural Examination:

In this study, microstructure characterization was examined by using optical microscopy and also scanning electron microscopy (SEM) with EDS analysis to identify the intermetallic phases and texture evaluation of as-cast, rolled and heat treated alloy. The grain size was also measured by image analysis software materials plus. Microstructure changes by process and precipitation mechanism were followed.

The samples were prepared as grinding at up to 1200 mesh and applying $Al_2O_3$ polishing paste. Then, Keller solution (0.5% HF in 50ml $H_20$ or 2.5 ml HNO3, 1.5 ml HCl, 1 ml HF, 95 ml pure water) was used to etch until about 1.5 minutes for the polished samples to see the precipitates and grains clearly. After that, the surfaces of the samples were washed with pure water and alcohol. After drying the samples, microstructure was ready to see under the optic microscopes and also SEM.

XRD (X-ray diffraction) method was needed to determine the intermetallic. As Nowotnik and workmate state, not only microscopic examination but also XRD result are needed to identify the intermetallic confidently.

### 3.4.2 Mechanical Tests:

Tensile strength and strain values were measured by tensile tests graphics. Dimensions of tensile test specimen were indicated in figure3-8. Tests were applied according to standard ISO6892-1:2009 LT (long transverse). Results of tensile test gave information about warm forming behavior due to acting as similarly during stamping process.

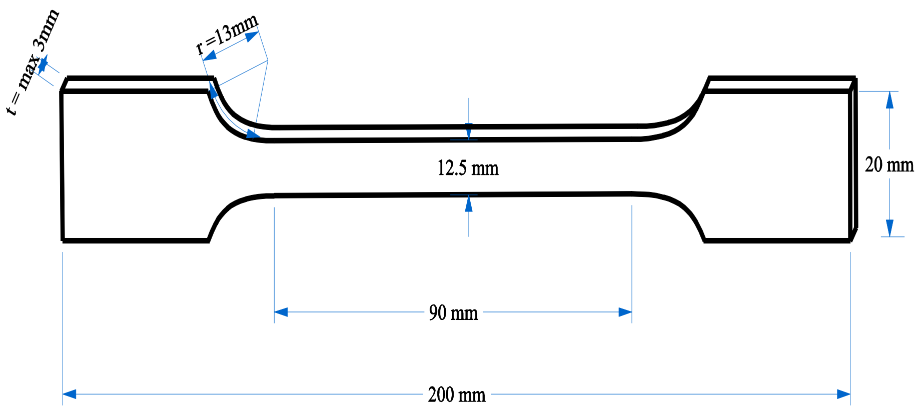



**Figure 3-7**: Dimensions of tensile test specimen [43].

### 3.4.3 Bending Test

Bending test was applied to see the formability limits of sheets. Sheets were cut to pieces and each of the surfaces and edges were ground well because, notches and cracks would behave as crack initiation point. After grinding, the samples were bended $180^0$ as shown schematically in figure 3-9. Then, the surfaces where the dense forming occurs were checked by eyes whether crack appeared or not.

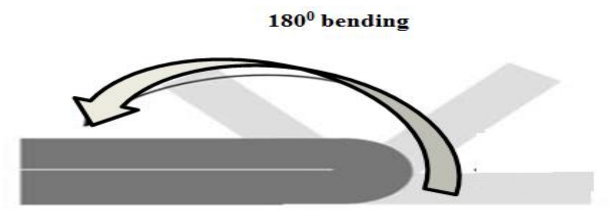



**Figure 3-8:** Schematic view of $180\ ^0$ bending test

### 3.4.4 Erichsen Test:

Erichsen test was applied to see the sheet strecthability of forming after T4 and T6 heat treatments according to ISO 16630 standards. Lubricant as stamping oil was used. 400 kN force was applied. The test was completed when a crack was seen on surface. The height of elongation and maximum force were recorded for investigation and comparison with literature.

### 3.4.5 Hardness Test:

Hardness values were examined after each of heat treatment process and after changing each of parameters of heat treatment to analyze the effects on hardness values. Micro hardness was performed as Vickers by an HXP-1000M micro hardness tester under the load of 0.49 N at room temperature. The micro hardness values for each of samples were taken the average of ten measured results.

### 3.4.6 Electrical Conductivity Test:

Electrical conductivity values were measured after each of heat treatment process and after changing each of parameters of heat treatment to make a comment about precipitation size, amount and situation. Fisher Sigmascope instrument was used with 60 KHz at room temperature. Tests were repeated three times and averages of them were taken into consideration.

# CHAPTER 4

# RESULTS

## 4.1 Microstructural Features

During pouring, mould stayed at 40-$55^0$ angle and casted with suitable casting speed so density of defects such as casting holes, segregations was eased off. Cooling rate and thermal conductivity were related to microstructure, final mechanical properties, intermetallic and precipitation distribution.

**Figure 4-1:** Experimental as-cast 6.7 mm AA6111 plate

After casting, a piece of samples from plate seen in figure 4-1 was taken for analysis. XRD was applied to investigate the phases present in the as cast state of splat. According to result of optic emission spectrometry, 6111 aluminum alloy grade was achieved as seen in the table 4-1. Chemical composition virtually met standards of UNS 96111 limit.

In this study, polished samples were examined by optical light microscopy and scanning electron microscopy with EDS analysis to identify the intermetallic phases and texture evaluation of cast, rolled and heat treated alloy. According to the rate of cooling; morphology of grain, dendritic arm spacing, grain size and intermetallic phases were formed.

**Table 4-1**: Chemical composition of 6111 aluminum alloy plate

| | Chemical Compositon (wt%) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 6111 Alloy | Si | Fe | Cu | Mn | Mg | Cr | Zn | Ti |
| | 0,6-1,1 | 0,4 max | 0,5-0,9 | 0,1-0,45 | 0,5-1 | 0,1 max | 0,15 max | 0,1 max |
| | 0,82 | 0,11 | 0,84 | 0,45 | 0,60 | 0,01 | 0,05 | 0,08 |

Ascast microstructure had almost 96 µm grain size. Different types of intermetallic phases were precipitated along the grain boundaries and inside the grains of α-Al matrix as seen in figure 4-2. Grain refiner provided forming columnar grains at exterior and equiaxed grains in the center as seen in figures 4-2 and 4-6 (a) [37].

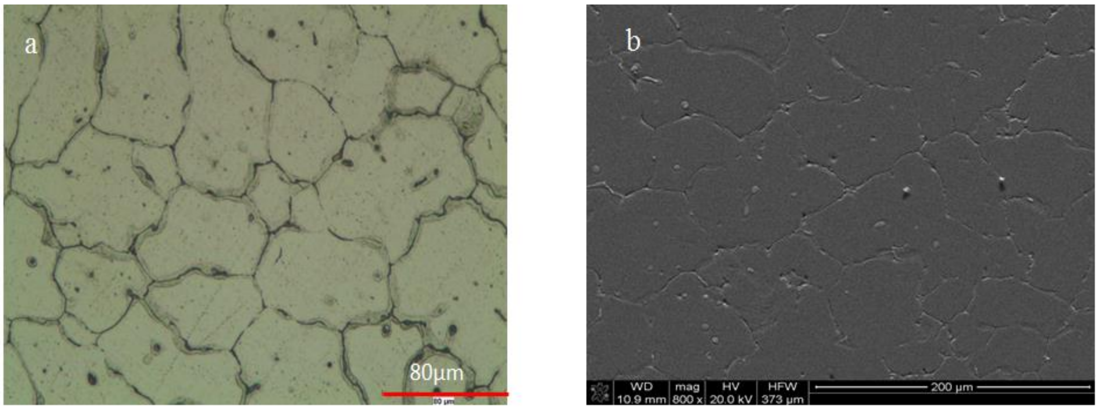



**Figure 4-2:** As cast microstructure of AA6111 alloy a. Optic microscope image b. SEM image

Hot and cold rolling caused deformation and changed the grain structure morphology of α-Al matrix and grains. Elongated grains were observed along the rolling direction as it was seen in figure 4-3 (a) and (b). Grain size was measured as nearly 180 µm. Blistering problem was not occurred.

Intermetallic phases precipitates were seen in sample homogenized 530 $^0$C; water quenched and then, stayed at room temperature for more than 5 days for natural ageing in figure 4-4 (a) and (c). Smaller than 5 µm size intermetallic phases precipitated over Al matrix as seen clearly in SEM images of figure 4-4 (c). Due to rapid cooling, there was no time for separation of θ-phase so that the alloy was unstable and supersaturated even at low temperature.

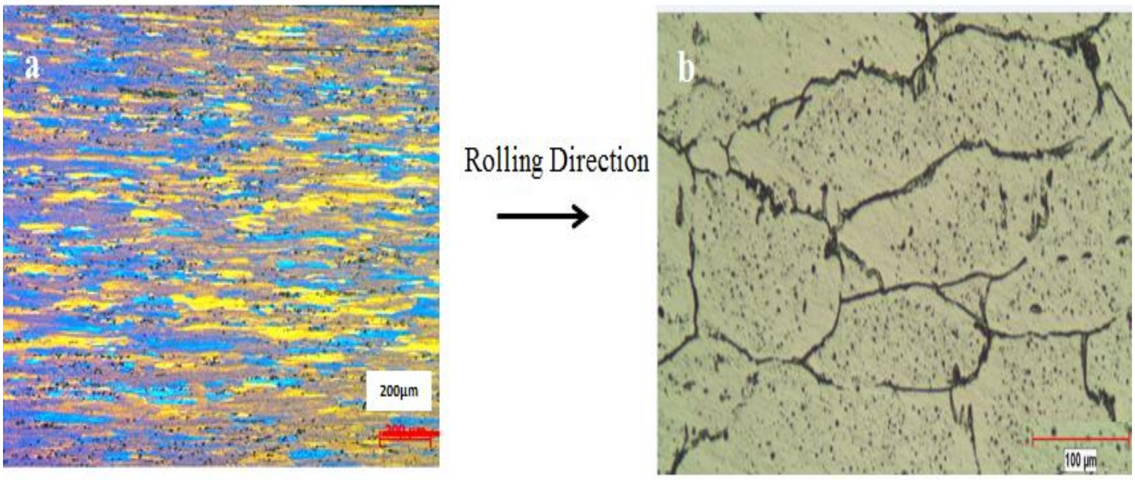



**Figure 4-3:** Optic images of rolled microstructure of AA6111 alloy with a. low magnification b. high magnification

On the other hand, as in Smallman and Bishop's studies, when the alloy stays for enough time which is called as ageing, the second phase precipitation is expected. [44]. When the samples reheated in the furnace to subject to ageing process then water quenched, it was seen that fine precipitation distributed in the figure 4-5 (a) and (c). Moreover; the size of intermetallic phases grew with respect to precipitates of natural aged microstructure as it can be openly distinguished the differences between figures 4-4 (a) and 4-5 (a). The intermetallic occurred during cooling due to low solubility of Fe, Cu, Cr, Sn, Mg and Si in aluminum matrix. According to G. Mrówka-Nowotnik, characteristically there is $Al_2Cu$, $\beta Al_5FeSi$ and $\alpha$-$Al_{15}(FeMn)_3Si_2$ intermetallic and rarely $Mg_2Si$ in typical as-cast microstructure of AA6111 aluminum alloys. They were seen along grain boundaries [45] [46]. AlCuMgSi has non-equilibrium morphology in the grain interior [46]. Determining the $Al_2Cu$ intermetallic is not possible under the optic or scanning electron microscopy so that their sizes are nearly 5-10 and 30-50 nm [47].

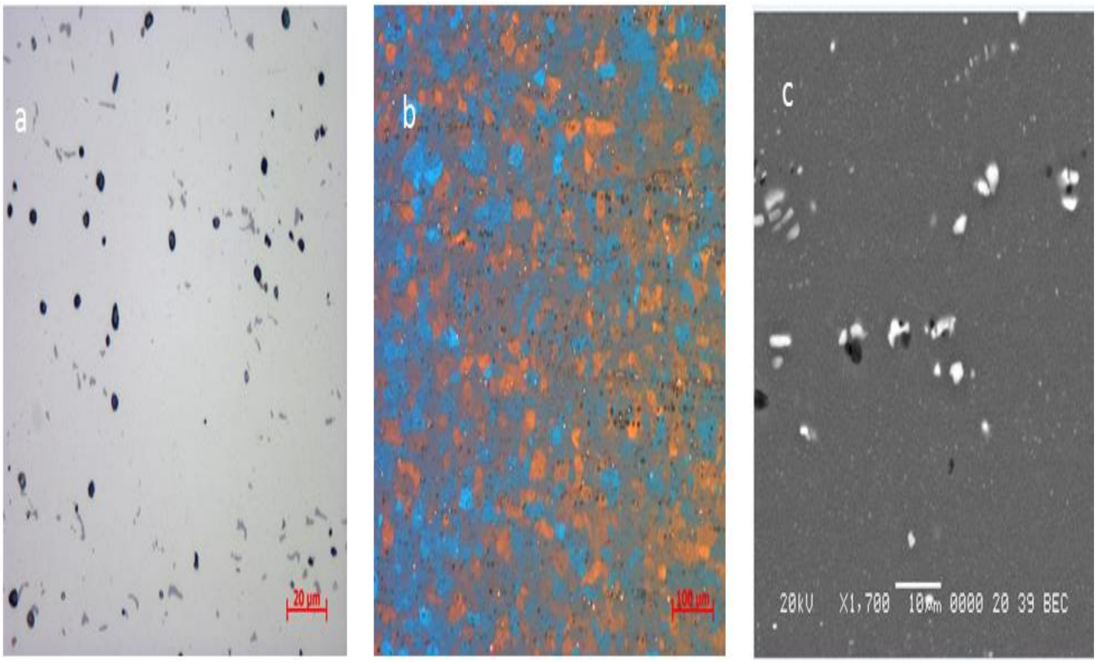



**Figure 4-4**: Microstructure of natural aged samples a. as polished surface b. etched surface in optic microscope c. SEM images with precipitates

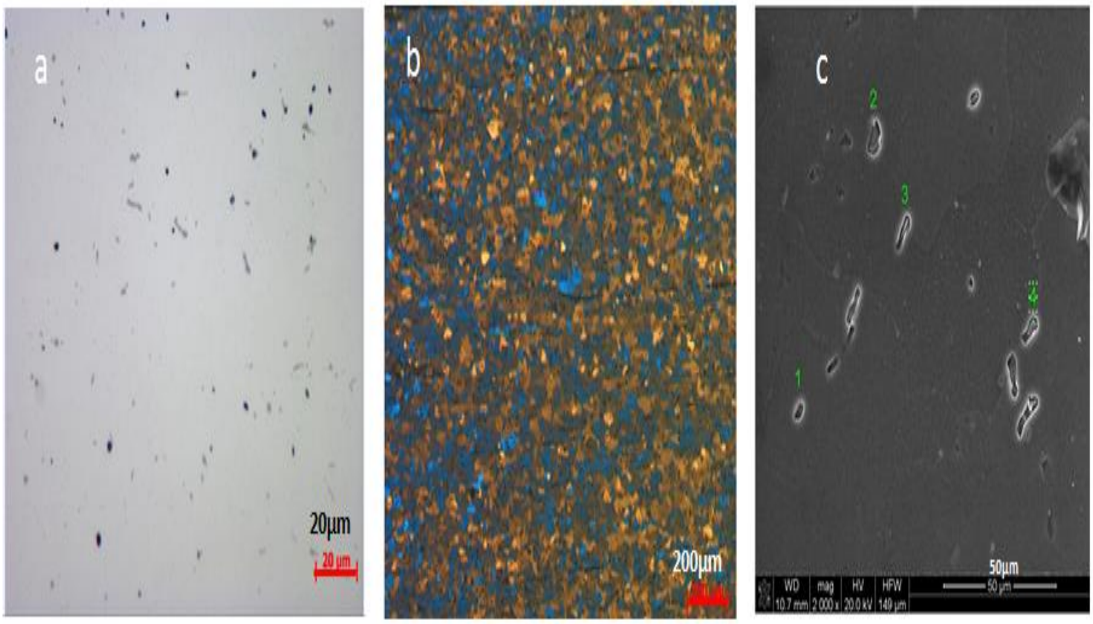



**Figure 4-5:** Microstructure of artificial aged samples a. as polished surface b. etched surface in optic microscope c. SEM images with precipitates

As cast structure had Al matrix and dissolved Mg, Si and AlFeSi precipitates along grain boundaries. Shape and distribution of Si particles and spherical and evenly distributed intermetallic precipitates are the most important parameters to obtain successive mechanical behavior such as strength, fatigue and impact properties. Fine dispersoids dissolved during solutionizing and so grains had chance to growth. Moreover, during homogenization of the alloy, β-AlFeSi phase might transform to spheroidal α-Al(FeMn)Si. There were fine precipitates which are displayed in figures 4-4 and 4-5 microstructures. These might be β-$Mg_2Si$ phase as G. Mrówka-Nowotnik assumed [48].

When examined with SEM, the precipitations and distribution of precipitations were seen in figures 4-8, 4-9 and 4-10 and with EDS analysis, morphology and the results of XRD patterns in figure 4-7, the phases can be predicted as $(Mn,Cu)_3SiAl_{12,}$ β $Al_5FeSi$, $Al_2Cu$, α-Al(FeMn)Si [48], [49] [48], [50], [51], [52], [53]. At the same time, there were the dark parts which were voids over the as polished surfaces. These voids formed during grinding or polishing steps. Because, the particles were hard while the aluminum matrix was soft and any mechanical movement as friction or brushing made hard particles separated from matrix [5]. Therefore, it was better to apply back scattering mode of SEM analysis. Eventually, this might eventually enable to discover the $Mg_2Si$ and other intermetallic more easily.

As it is mentioned in literature research, intermetallic particles can be determined according to their morphologies. They have platelet, rod, polyhedron or Chinese script type's morphology in figure 4-10 [54].

SEM elemental mapping analysis shows that intermetallic had Fe, Si, Mn and Mg. Most of intermetallic had Fe in composition due to max 0.05 wt. % Fe solubility in Al matrix [55]. $Mg_2Si$ and AlFeMn, AlFeMnSi intermetallic precipitates determined in figures 4-11, 4-12 and 4-13 according to results of elemental analysis images.

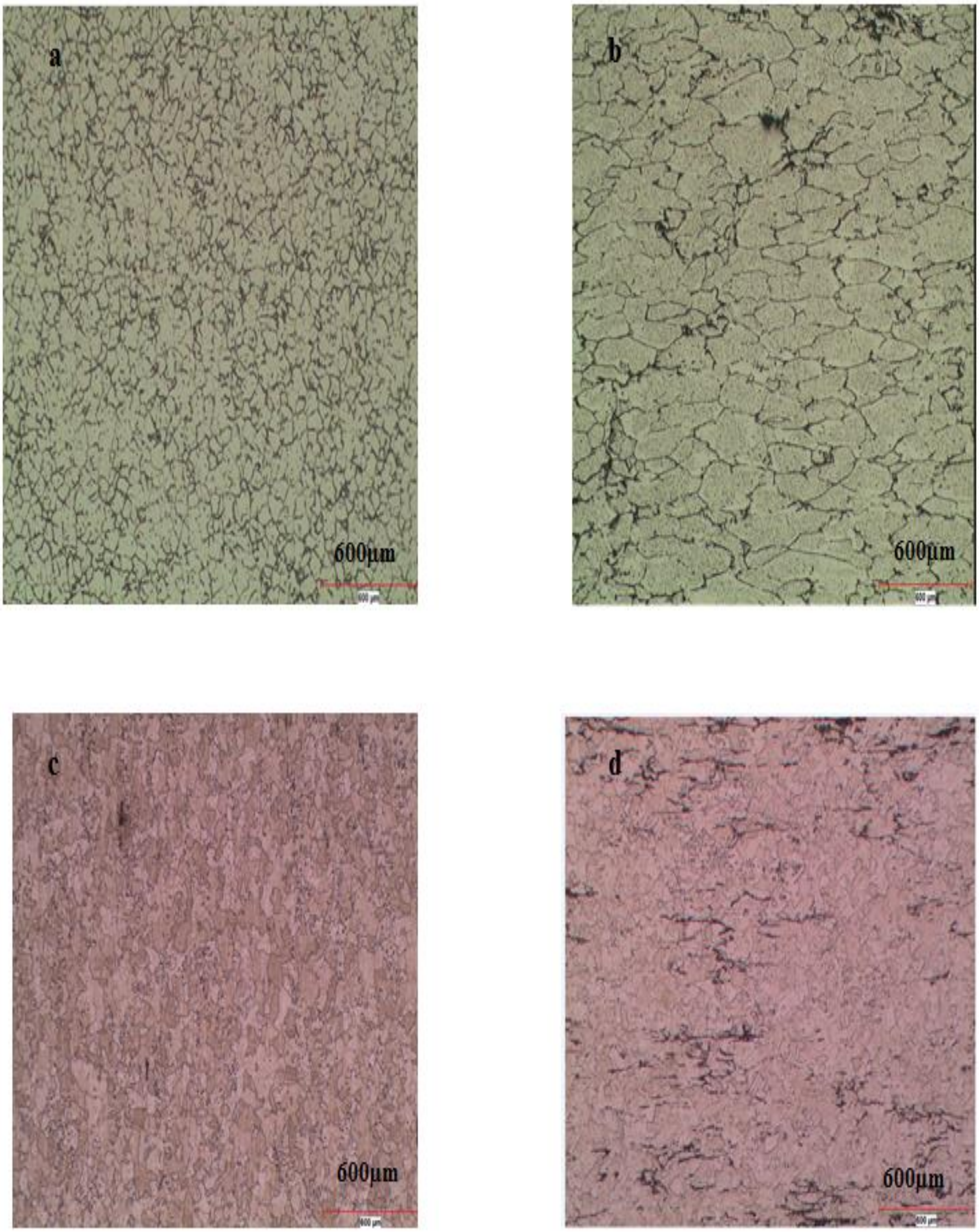



**Figure 4-6:** Optical images of AA6111 a. as-cast b. rolled c. T4 (natural aged) temper d. T6 (artificial aged) temper microstructures

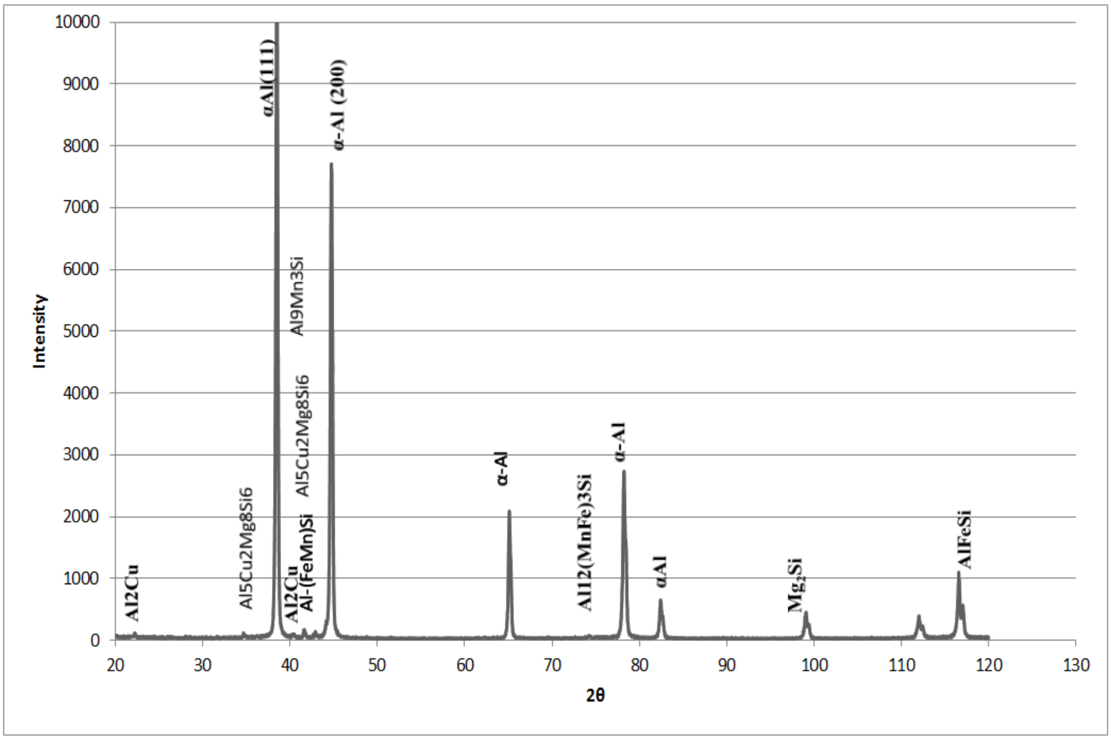



**Figure 4-7**: XRD pattern of AA6111 alloy processed in this work

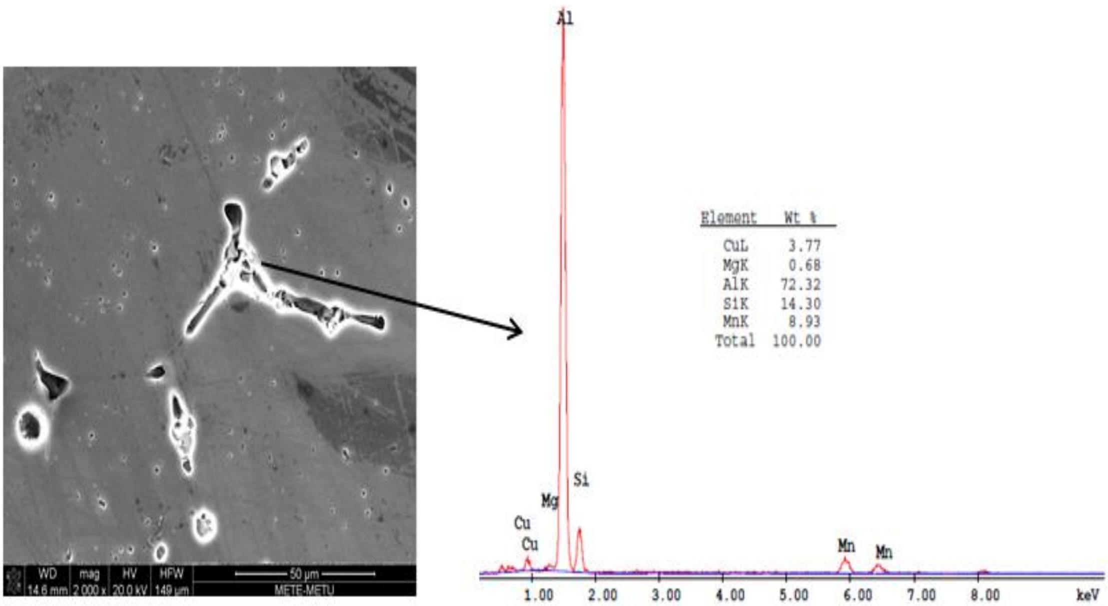



**Figure 4-8:** SEM image and EDS analysis, morphology of plate like β AlCuMgSi phase analysis result

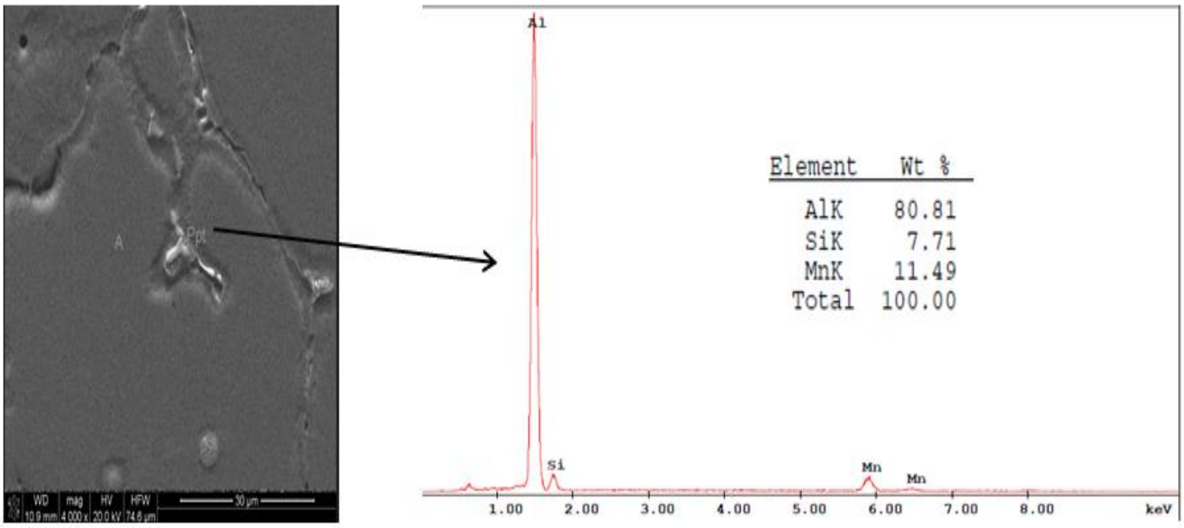



**Figure 4-9**: SEM image and EDS analysis of AA 6111 sample and AlMnSi phase analysis result

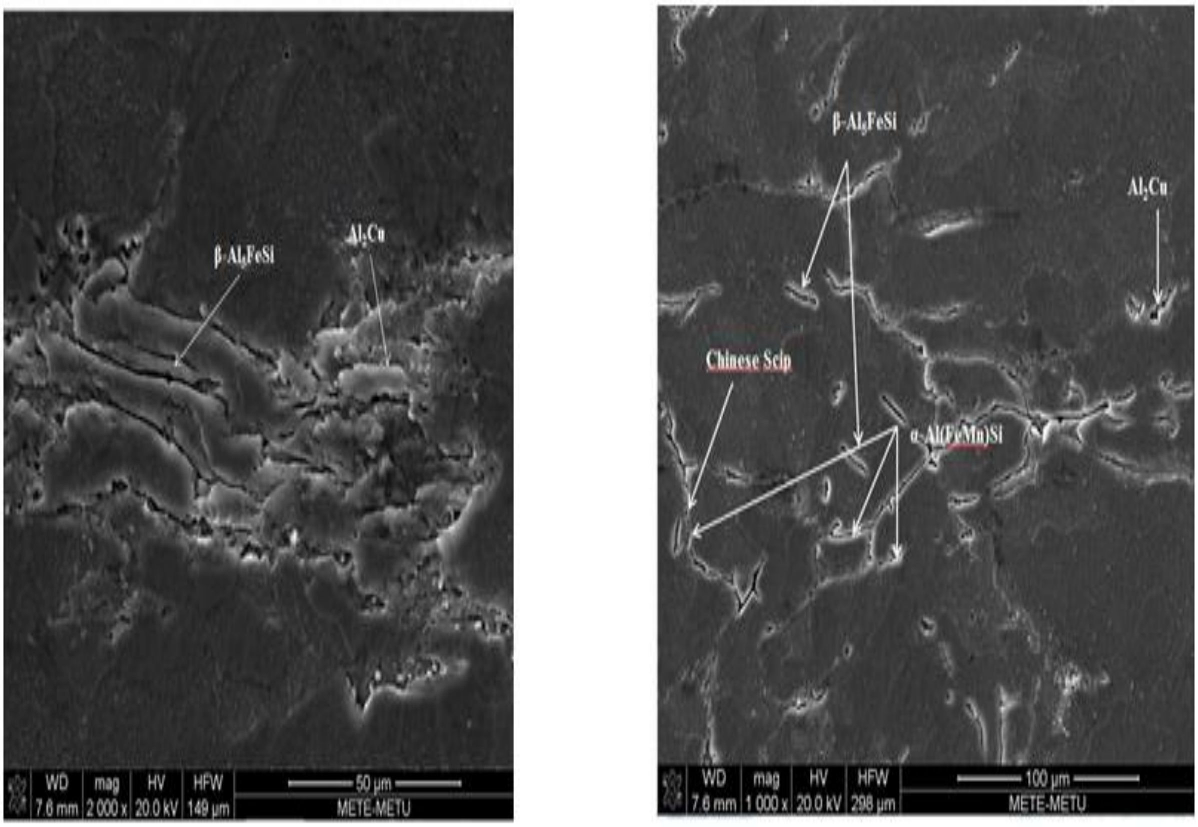



**Figure 4-10:** SEM image and Chinese scrip and columnar morphology intermetallic phases

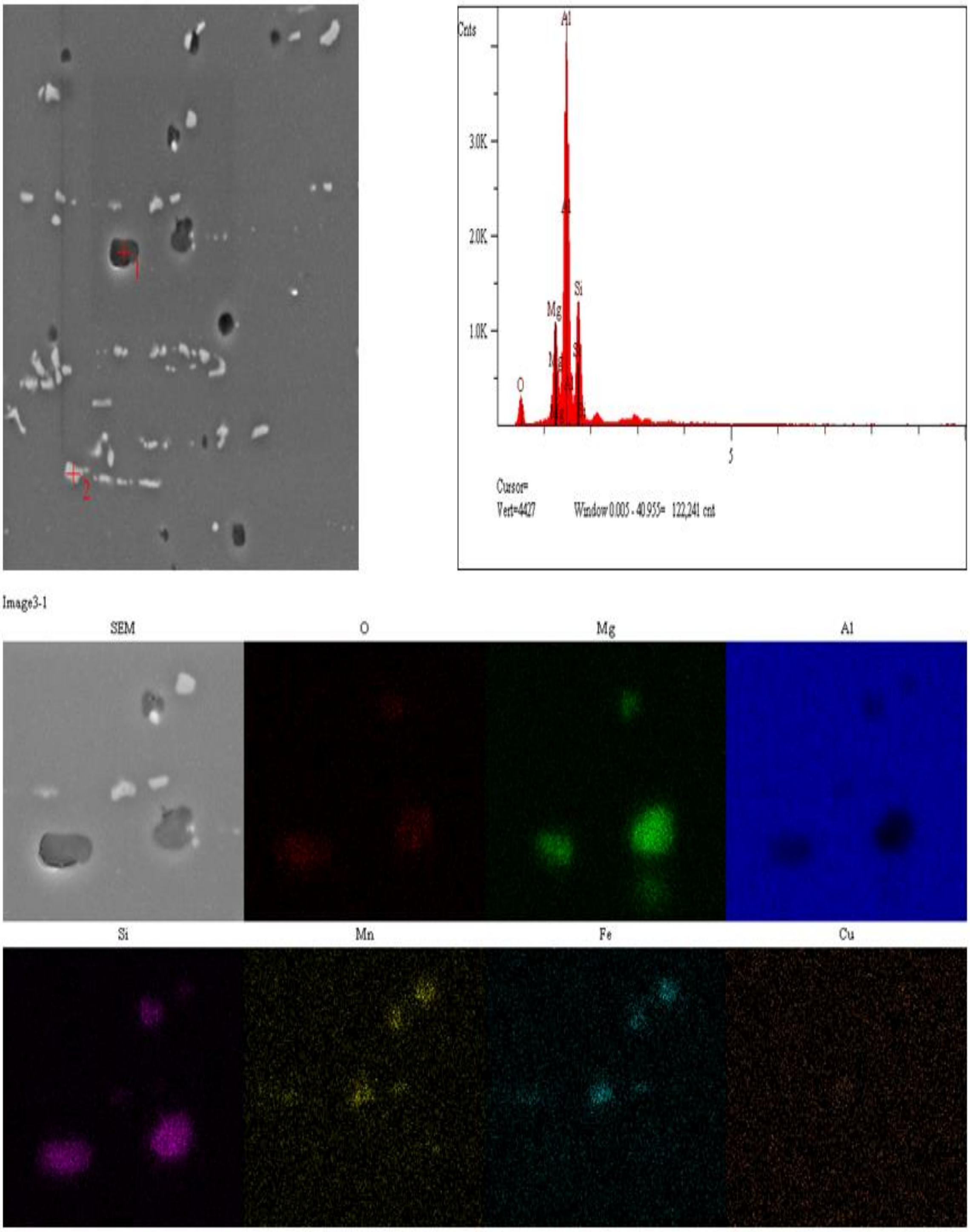



**Figure 4-11**: SEM images of $Mg_2Si$ phases & corresponding elemental mapping analysis

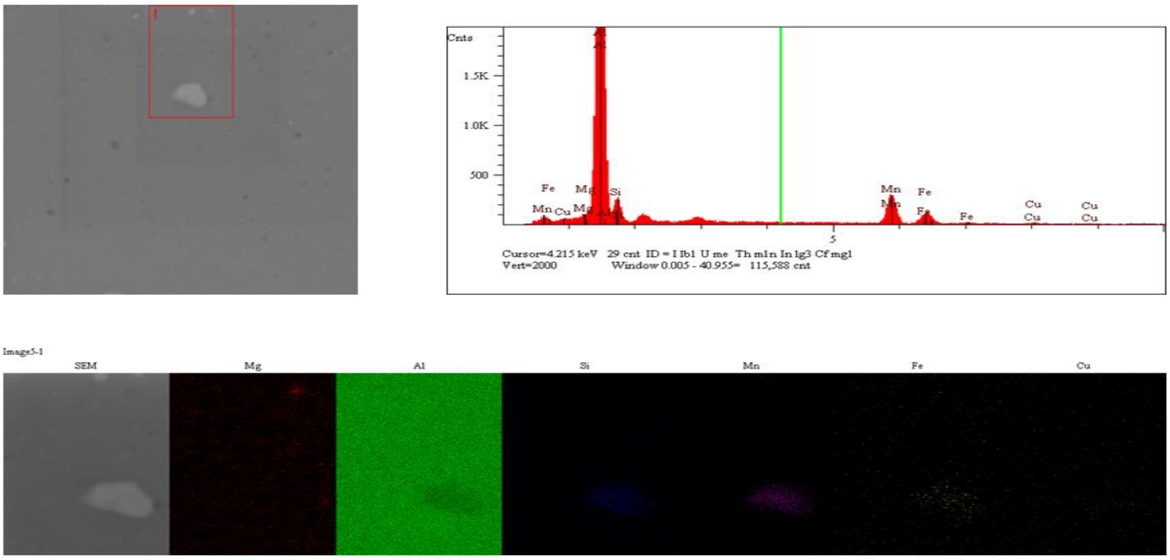



**Figure 4-12:** SEM images of AlFeMnSi phases & corresponding elemental mapping analysis

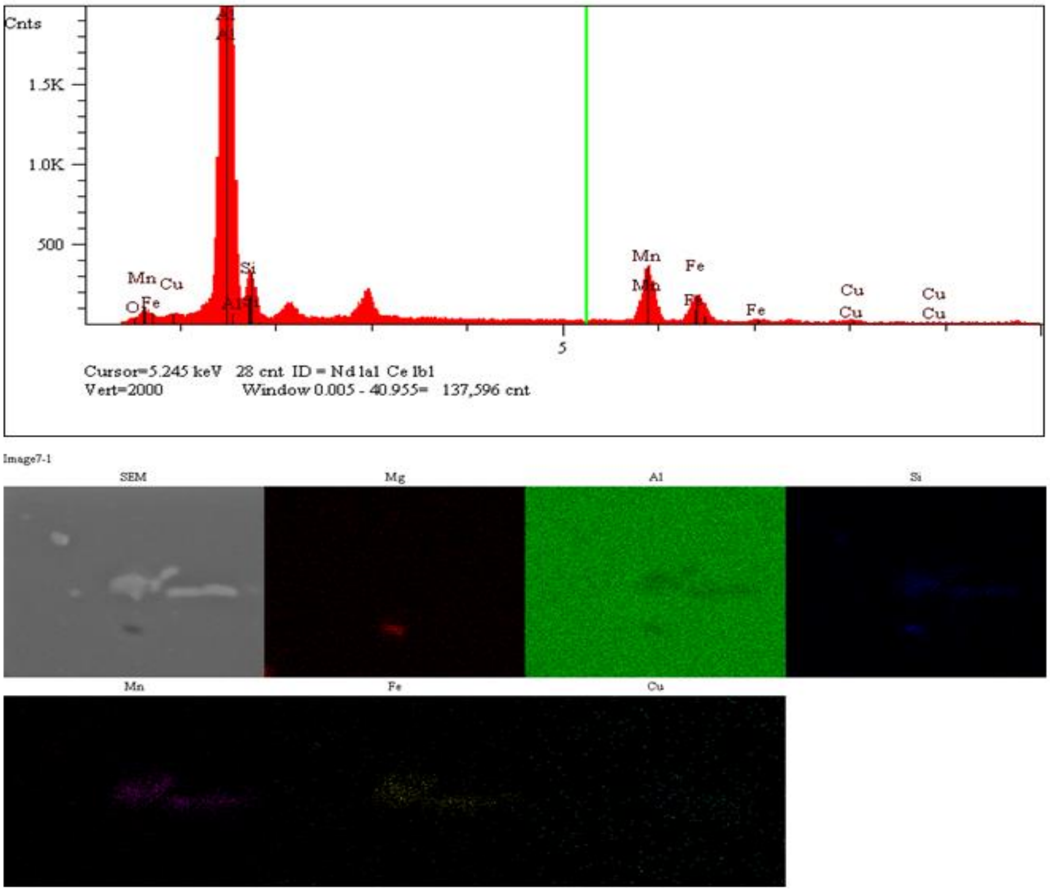



**Figure 4-13**: SEM images of (Mn,Cu)SiAl phases & corresponding elemental mapping analysis

Intermetallic particles of quenched samples after solutionized at 530 $^0$C and then artificially aged had coarse structure as seen in SEM images figure 4-16. According to effects of length of ageing period and temperature, precipitates of intermetallic became finer and distributed homogeneously. GP zones, side nearby intermetallic like dust cloud were obtained. As determined in Erdoğan and his workmates studies, these sides were similar to their outcomes [56].

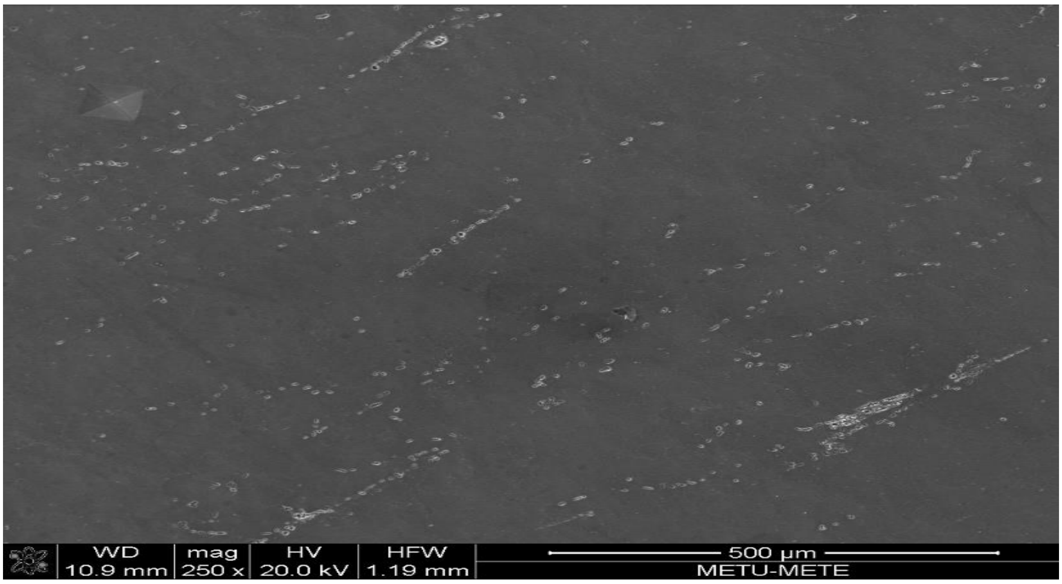



**Figure 4-14**: Distribution of precipitation on SEM image AA6111-T4 sample

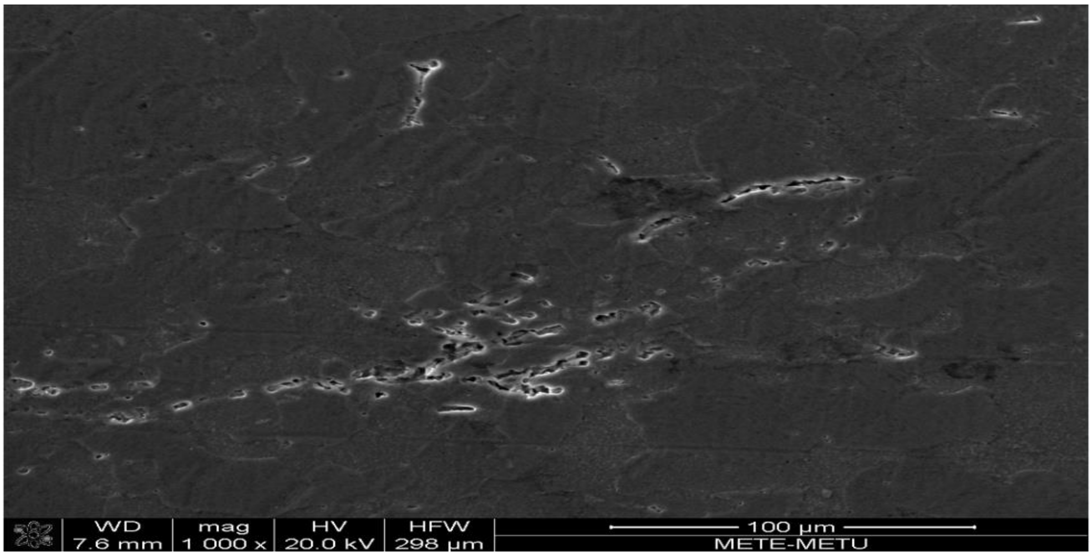



**Figure 4-15**: SEM images of ageing samples at 160 $^0$C for 6h.

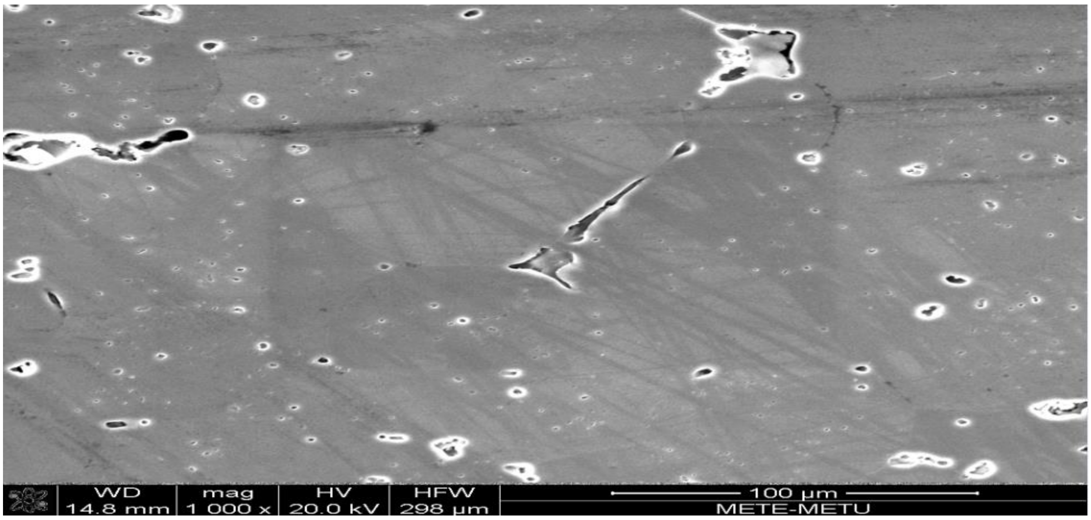



**Figure 4-16**: SEM images of ageing samples at 240 $^{0}$C for 6h.

## 4.2 Mechanical Properties

The mechanical properties of 6111 alloy sheets proceed in this work was given in table 4-2 for each treatment.

**Table 4-2**: Mechanical Properties of AA6111

| Samples | Yield Strength MPa | Tensile Strength MPa | % | Avg. Hardness $HV_{0.5}$ |
|---|---|---|---|---|
| **As rolled** | 253 | 265 | 2.5 | 80 ± 3 |
| **As solutionized** | 113 | 247 | 21.1 | 79 ± 3 |
| **T6 160°C-90min** | 299 | 360 | 11.9 | 108 ± 9 |
| **T6 160°C-3h** | 288 | 384 | 10.5 | 120 ± 4 |
| **T6 160°C-4h** | 280 | 359 | 14.7 | 108 ± 4 |
| **T6 160°C-6h** | 217 | 295 | 8.08 | 95 ± 2 |
| **T6 200°C-90min** | 337 | 368 | 7.4 | 123 ± 4 |
| **T6 200°C-3h** | 337 | 356 | 5.8 | 120 ± 4 |
| **T6 200°C-4h** | 345 | 356 | 6.7 | 122 ± 5 |
| **T6 200°C-6h** | 313 | 344 | 7.4 | 117 ± 1 |
| **T6 240°C-90min** | 253 | 298 | 6.4 | 87 ± 4 |
| **T6 240°C-3h** | 214 | 283 | 7.4 | 85 ± 4 |
| **T6 240°C-4h** | 220 | 277 | 6.6 | 85 ± 2 |
| **T6 240°C-6h** | 218 | 273 | 8.6 | 81 ± 6 |

Solution treatment was set according to sample thickness, microstructure and furnace condition [17]. Solutionizing provided homogenization of microstructure and dissolving of almost all round, spherodized Si particles, $Mg_2Si$, $Al_2Cu$ and Cu rich intermetallic. Thus, ductility was enhanced. Solutionizing was important for catching the desired ageing effects and mechanical properties. Thus, it enabled almost all heterogeneously distributed precipitates to be dissolved first after that, new fine intermetallic to be precipitated and dispersed homogeneously during ageing.

By precipitation age hardening, mechanical stress can be easily controlled. It is the fact that precipitates prevent the movement of dislocation. Therefore; by controlling the size and distribution of precipitates mechanical stress can be enhanced.

During forming and stamping processes, low yield strength is required. It is assumed to be lower than 140 MPa with respect to scientists' opinion. Therefore, springback problem can be eliminated and high elongation can be obtained for shaping processes. After solutionizing step, average yield strength value was measured below 140 MPa as 113 MPa. This might enable shaping smooth without defects such as crack, notch etc. and without springback.

After T4 ageing, precipitation occurred and prevented the dislocation movements. Therefore, strength was enhanced. On the other hand; after T6 ageing, new precipitation occurred and old precipitation came together and size became larger. According to Orowan's mechanism, when the sample is applied aged at optimum condition, the precipitates size will be fine precipitate. These fine precipitates make dislocation movement difficult, yielding Orowan's Looping [57]. In service condition, panel should be tough enough to any impact and dent so minimum 200 MPa is needed according to view of scientist [58]. In this experiment, yield strength requirement were met for each ageing condition.

After ageing, the particles made unit and so that the amounts of particles decreased while size increased by time as seen in the microstures of figure 4-17. Therefore, dislocation movement became harder than T4 condition. As a result, the mechanical strength was higher than T4. On contrast, elongation decreased due to movement

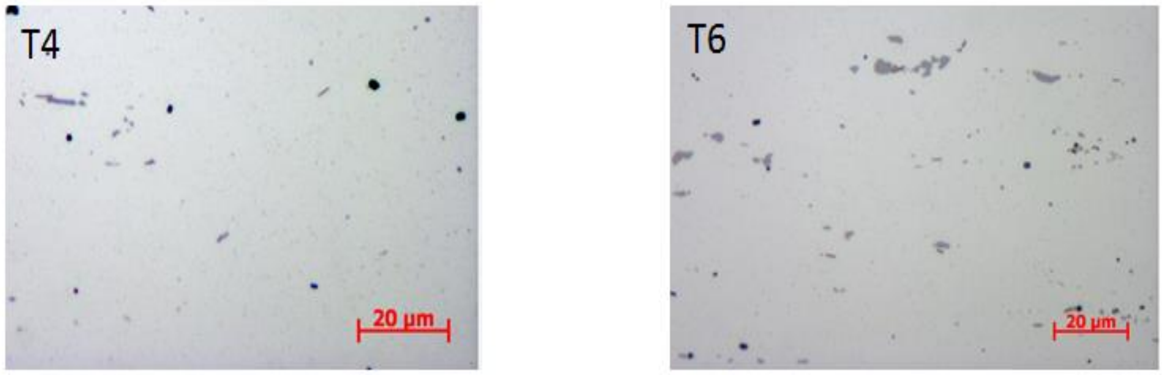



**Figure 4-17:** Polished surface of AA6111 T4 and T6 samples

difficulties of dislocation motion. Elongation is more important for preventing crack during forming. Increase in strength caused increase in hardness due to loss of ductility [57]. It was deduced from the mechanical result that maximum elongation was obtained when samples was solutionized and precipitates dissolved in the matrix as seen in table 4-2 and in figure 4.18 as it was assumed.

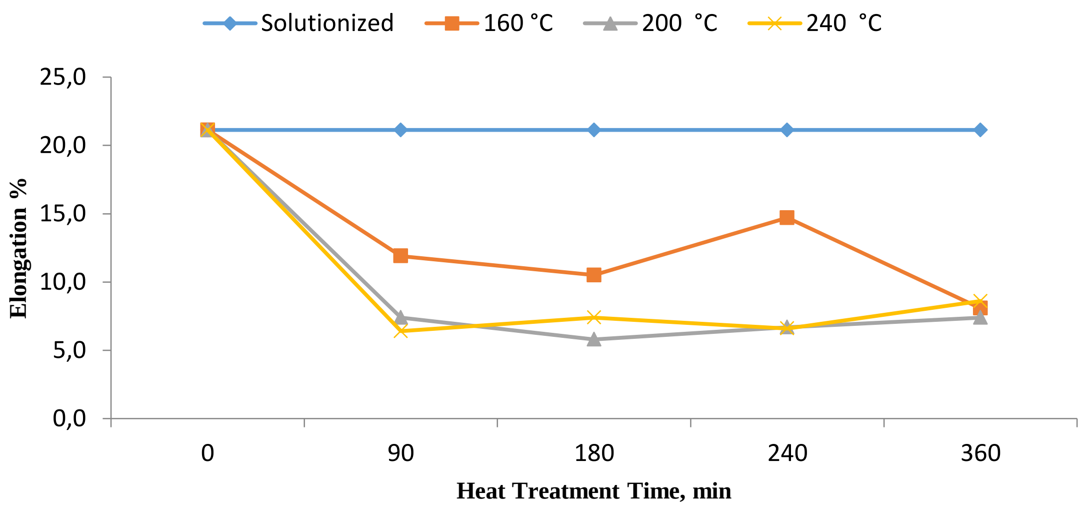



**Figure 4-18:** Elongation values variation with heat treatment time and temperature

Hardness values were higher in T6 treated samples. Mn, Si and Cu alloying elements played important role in this results. Precipitation of excess Si enhanced the hardness and alloying elements increased the strength values [14]. The results of heat treatments in table 4-2 and stress-strain graphic in figure 4-20 were parallel to the literature as it was assumed.

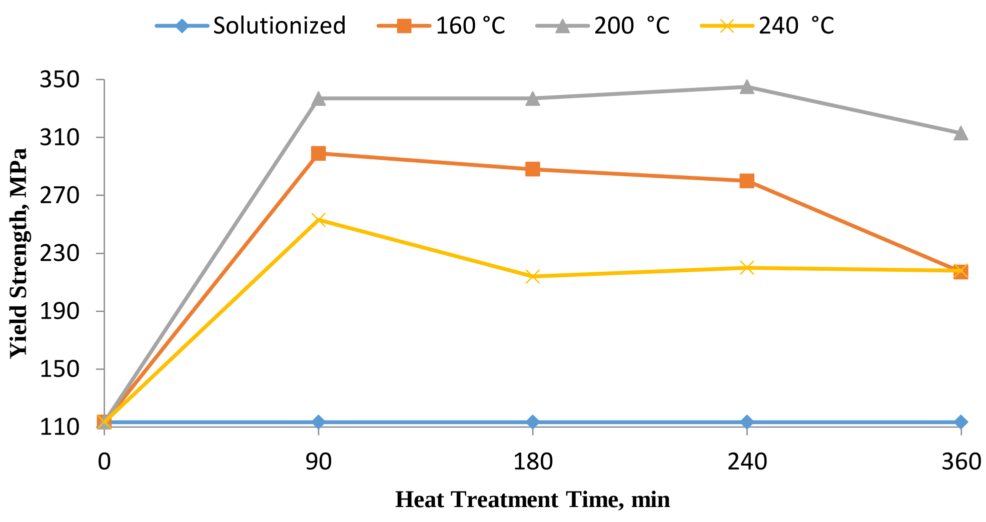



**Figure 4-19:** Yield Strength variation with heat treatment time and temperature

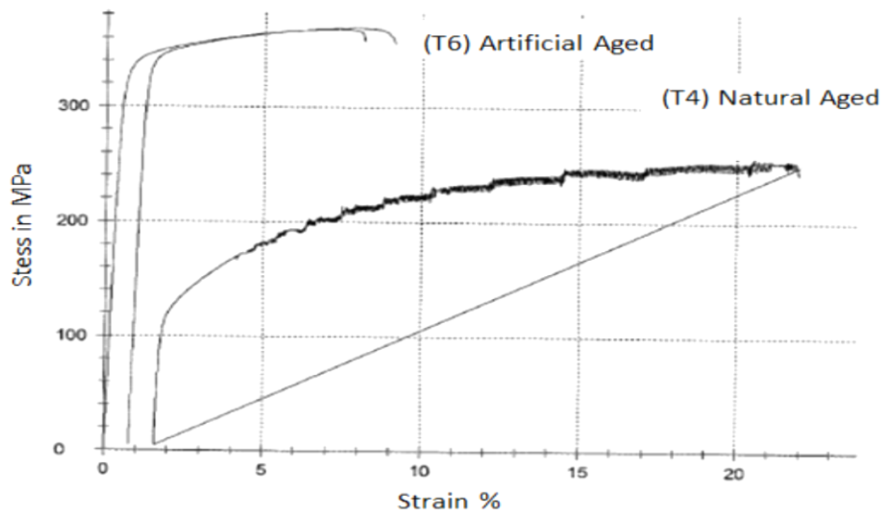



**Figure 4-20:** Stress-Strain graphic of A6111 aluminum alloys

The values of strength and elongation were not catched due to inclusions and holes in the structure. As seen in SEM analysis of tensile test samples crack surfaces (figure 4-21), there were holes. Besides, during casting steps, elements and composed accumulated due to nonclear casting. These were the reasons for low strength and elongation measurements. As tensile stress were applied, these segragation behaved as stress concantration sides and voids moved and/or combined. These accelerated the fracture and elongation also strength decreased.

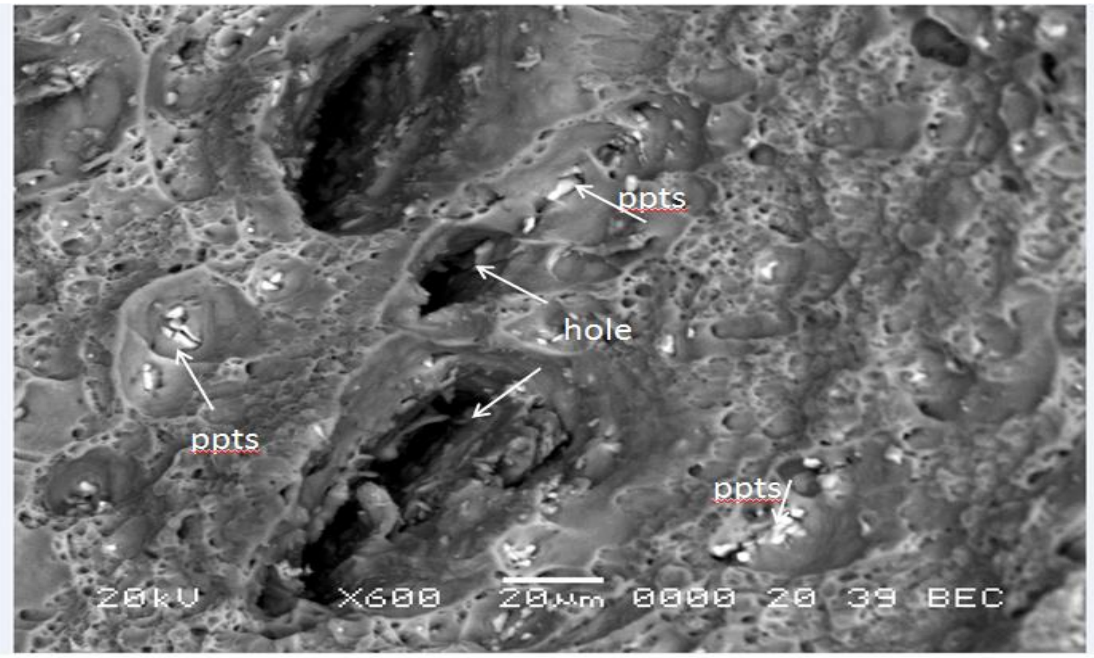



**Figure 4-21:** Crack surface of tensile test specimen

Inclusions located on the microstructures. Segregated particles elongated through the rolling direction as it was seen on optic images of figure 4-22. EDS analysis showed that these particles could be $Mg_xO_y$. Affinity of Mg with O compared to other elements is high. Therefore, during casting Mg elements easily oxidized and hard particles rolled and elongated during rolling steps.

The figure 4-23 and table 4-3 displayed the results of microhardness for each treatments. The higher microhardness of alloy is related to the fine grain and the supersaturated solid solution of α-Al and presence of dislocation density which are formed during cold rolling.

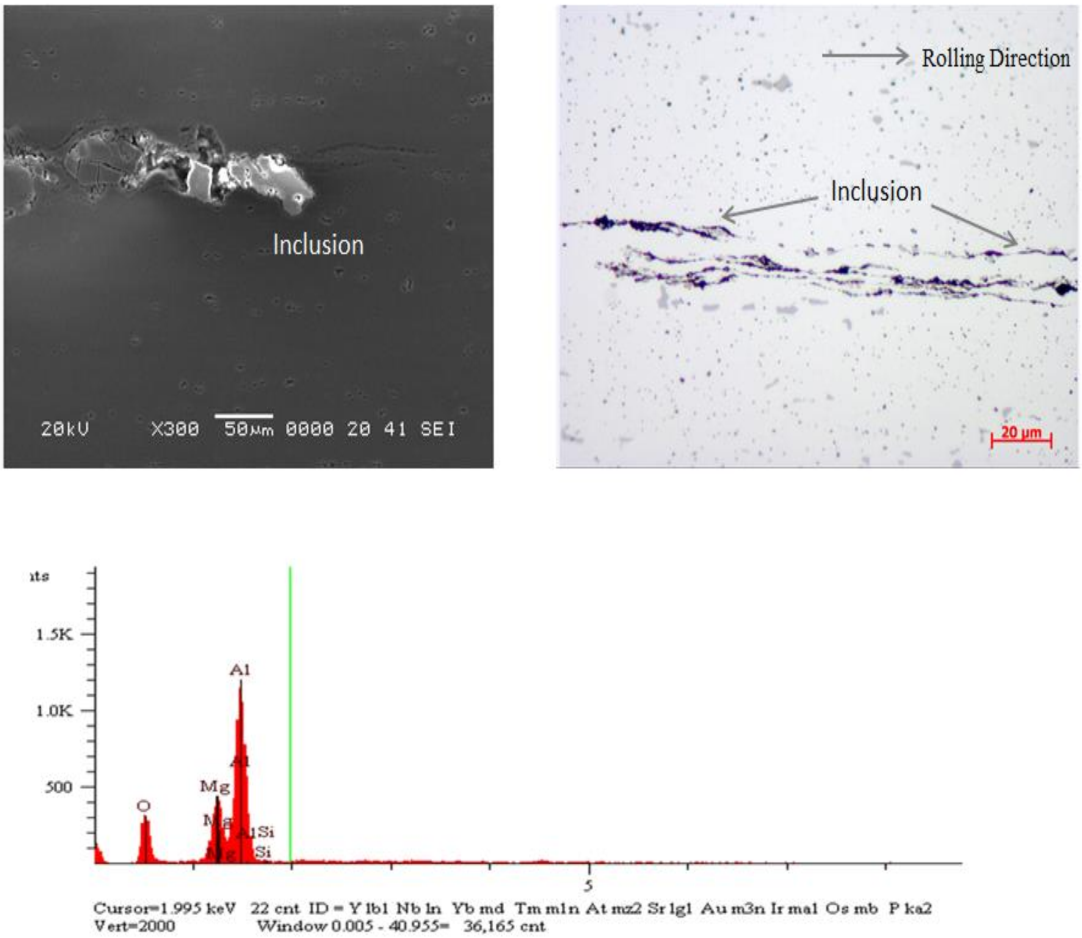



**Figure 4-22:** Segregation through rolling direction

The figure 4-23 and table 4-3 displayed the results of microhardness and tensile strength values for each treatments. The higher microhardness of alloy is related to the fine grain and the supersaturated solid solution of α-Al and presence of dislocation density which are formed during cold rolling.

During ageing process, hardness increased related to ageing time and temperature. As temperature increased, the size of intermetallic grew and intermetallic kept on unit with each other due to heat treatment. However, increase in hardness stopped when it reached optimum values. After that, it began to decarease as seen figure 4-23. Accoring to Mrowka, either forming different precipitates or overageing may make hardness decrease [59]. As a result of measurement, the maximum hardness was obtained at 200 $^{0}$C temperature when it stayed at furnace until nearly 90 minutes. The hardness values reached nearly up to 123 HV. Solutionized at 530 $^{0}$C samples as T4 samples had smaller hardness values. Few amount and small size of intermetallic compared to aged samples was the reasons of minimum hardness values.

**Table 4-3:** Hardness values according to ageing time and temperature

| Ageing Time (min) | As rolled | Ageing Temperature | | |
|---|---|---|---|---|
| | | 160 °C | 200 °C | 240 °C |
| 0 | 80 ± 3 HV | 80 ± 3 HV | 80 ± 3 HV | 80 ± 3 HV |
| 90 | 80 ± 3 HV | 108 ± 9 HV | 123 ±4 HV | 87 ± 4 HV |
| 180 | 80 ± 3 HV | 120 ± 4 HV | 120 ± 4 HV | 85 ± 4 HV |
| 240 | 80 ± 3 HV | 108 ± 4 HV | 122 ± 5 HV | 85 ± 2 HV |
| 360 | 80 ± 3 HV | 95 ± 2 HV | 117 ± 1 HV | 81 ± 6 HV |

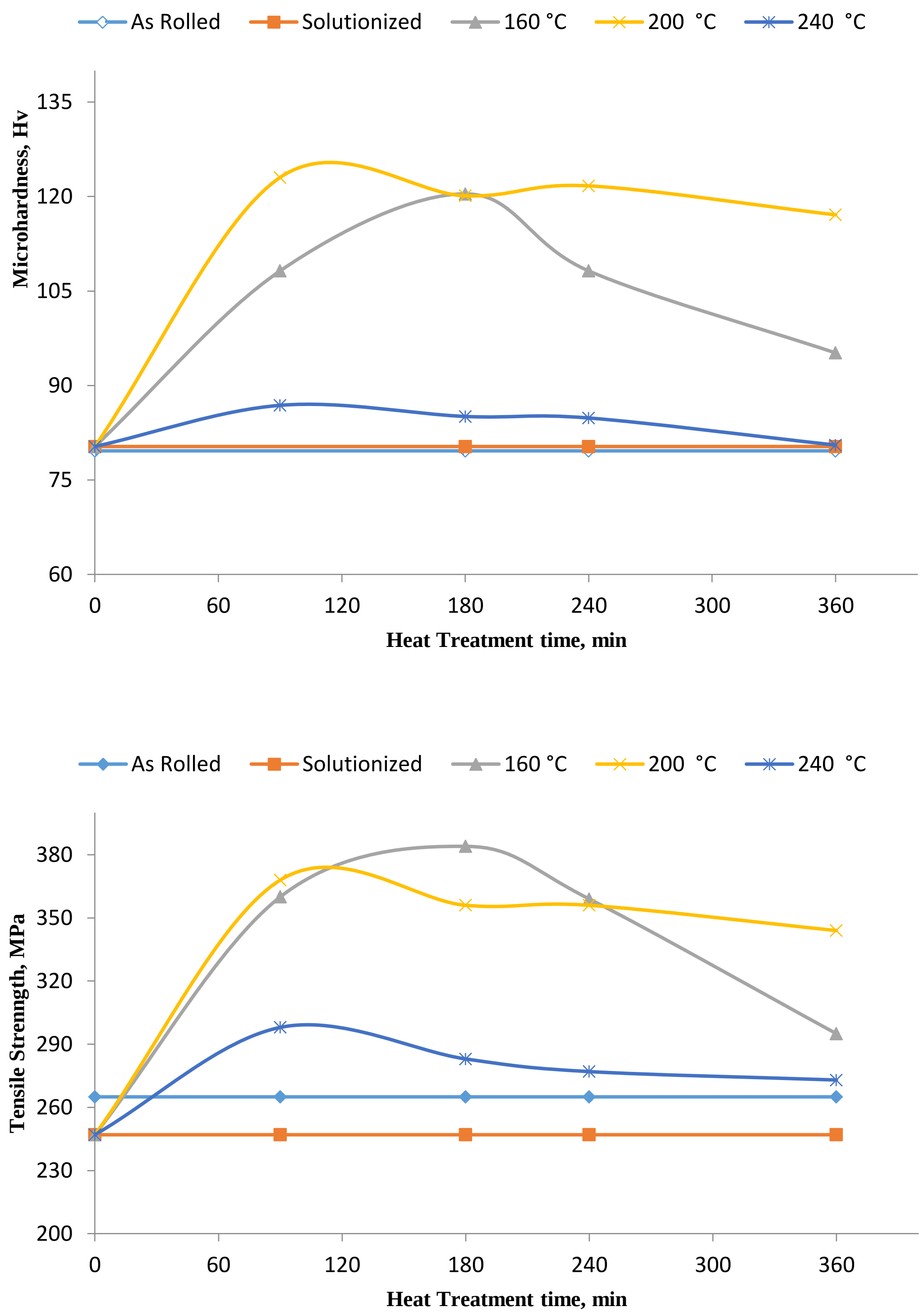



**Figure 4-23:** Result of heat treatment time and temperature on micro hardness and tensile strength of samples

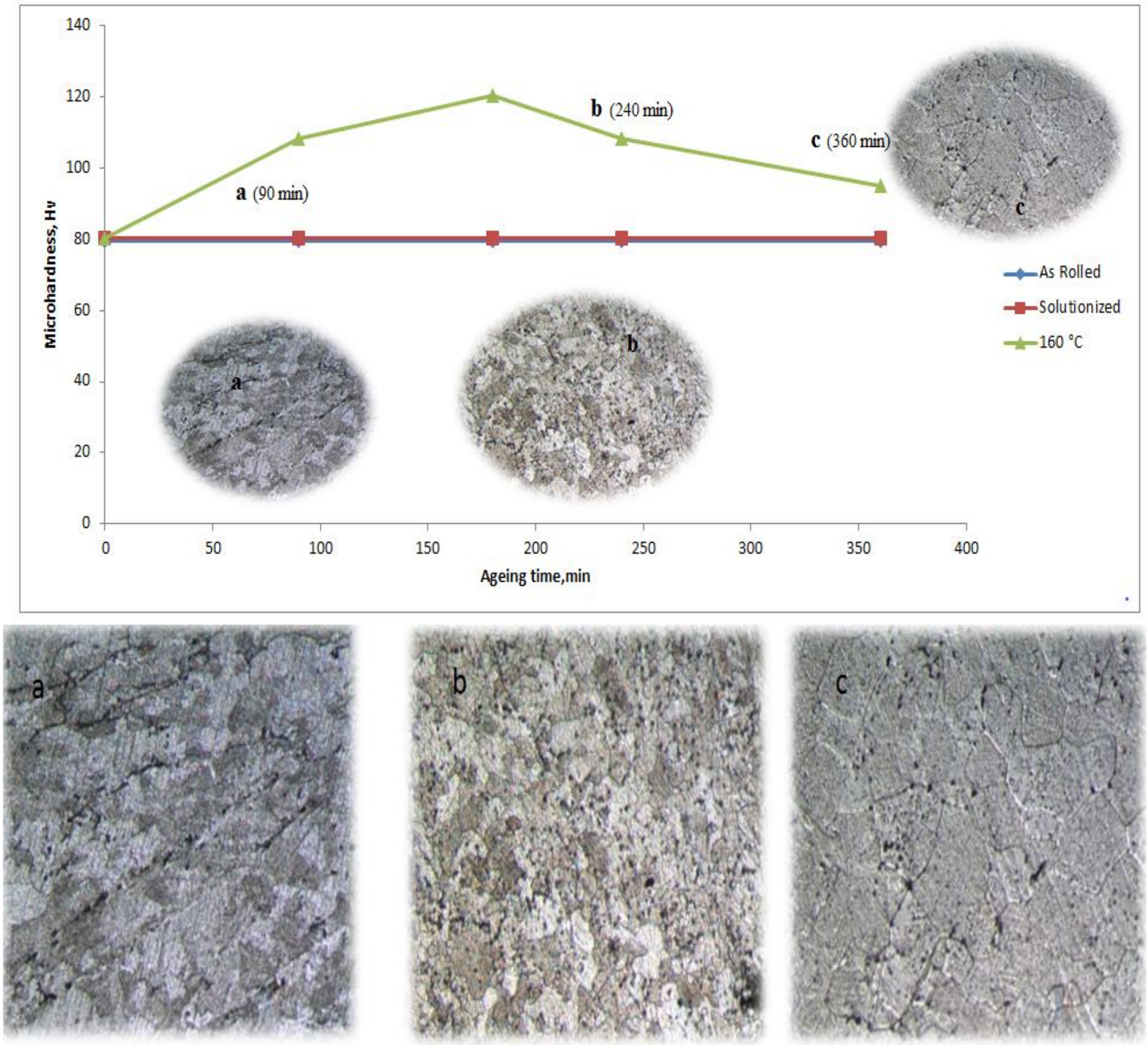
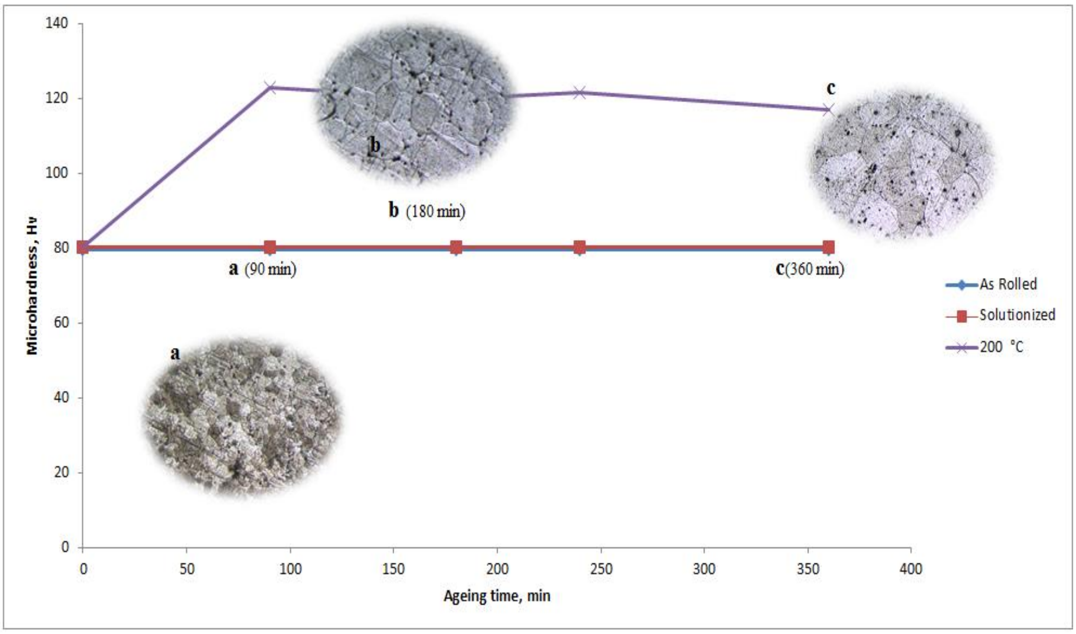



**Figure 4-24**: Micro hardness and microstructure of aging times at 160 °C, a. after 90 min, b. 240 min and c. 360 min aging. X100

Figure 4-24 shows the relationship between ageing time and hardness values at 160 $^0$C. The hardness slightly increased and after 3 hours, it reached the optimum values as 120 HV then, kept on decreasing. β" which provides hardening for AA61111 may not find enough time for precipitatation during 90 min at this temperature. After 90 minutes, the β' precipitates found enough time to transform to β'' precipitates.

Figure 4-25 represents the relationship between ageing time and hardness values at 200 $^0$C. The hardness slightly increased by time. Until 90 min, fistly GP zones were occurred. This is why slight increasement was observed in hardness values. GP zones as sphere dust clouds transformed to β" coherent type by time until 180 minutes.

After 90 minutes, the hardness reached the optimum vales as 123 HV then, kept on decreasing. The reason was related to β” precipitates according to Barbosa and his workmates demostration [60]. Then, slope of hardness began to



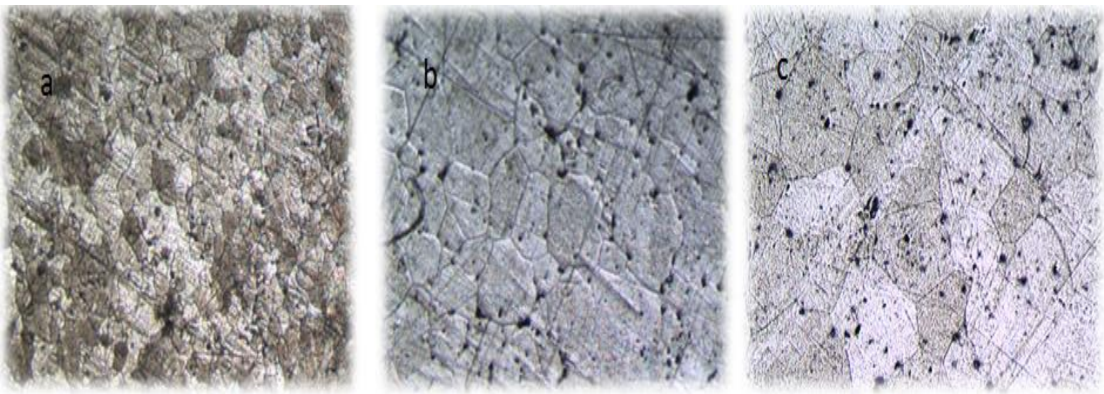




**Figure 4-25:** : Micro hardness and microstructure of aging times at 200°C, a. after 90 min, b. 180 min and c. 360 min aging. X100

decrease. After 180 minutes, the coherent β” transformeed to β’ precipitates and provided less hardness compared to cohenert phases. As Barboraos and his friends demonstrated, β’phases has nearly 100 HV [60]. Following aging until 360 min, some unstable phase β' transformed to stable phase β which made hardness decarease more. Neverthless, following ageing until 360 min, some coherent unstable phase β', transformed to stable phase β. β is not coherent with the matrix. Therefore; it caused a decrease in hardness. That is to say; overageing was occurred which causes easify the movement of dislocation.

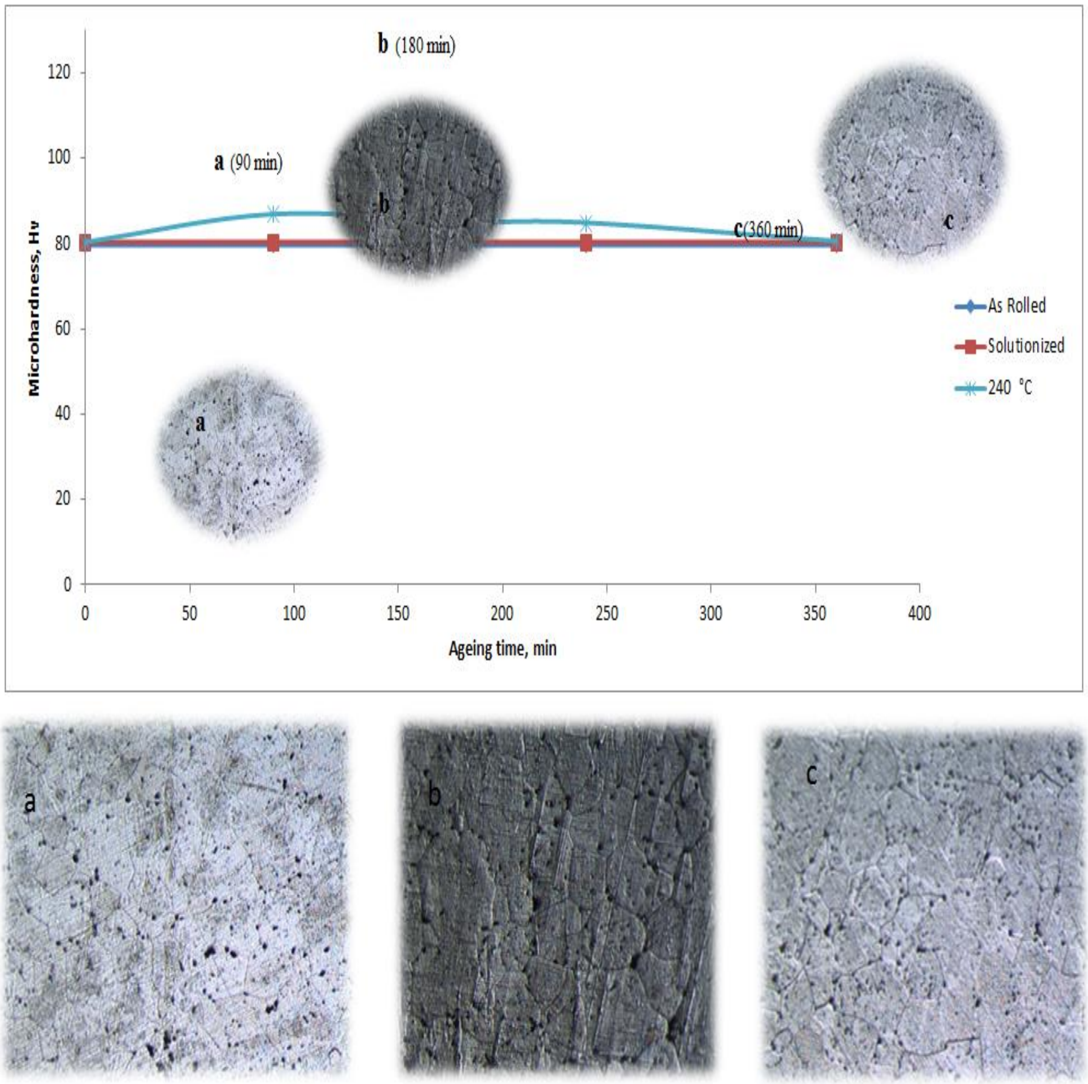



**Figure 4-26**: Micro hardness and microstructure of aging times at 240°C, a. after 90 min, b. 180 min and c. 360 min aging. X100

In figure 4-26 graph represents the relationship between ageing time and hardness values at 240 $^0$C. There was little increase in hardness values. 240 $^0$C temperature value was high for this experiment. Therefore, the samples could not be aged by time.

To determine the variation of precipitates amount and size, property of electrical conductivity was studied as result showed in figure 4-27. It is the truth that alloying elements diminsh electric conductivity of aluminum matrix.

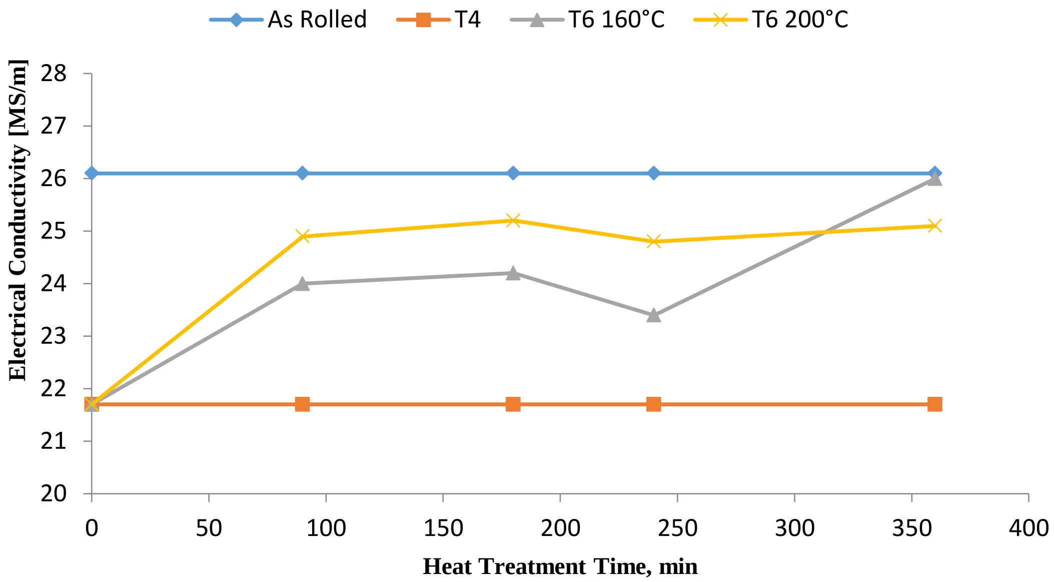



**Figure 4-27:** Result of heat treatment time and temperature on electrical conductivity of samples

According to scientists' assumption; during T4 natural aged and solutionized samples have got GP phases so that electrical conductivity diminishes so far as amount, size and coherency of the phases [61]. That is the reason of that electrons are scattered due to GP phases. In this manner, T4 samples had minimum conductiviy as supporting study of Birol and Esmaeili et al [58] [62]. During ageing, while density of vacancies decreased to approach to equlibrium condition, concentration of GP zones decreased, preciptates coelesenced and size grew, conductivity increased. Because, electrons had chance to move along matrix easily. These results verify the studies of Esmaeili et al. , B. Raeisina et al. and Birol [58] [63] [64].

Generally at first. hardness and strength values were investigated to see the intermetallic effect. Then, microsturecture were analyzed for detecting the reasons. In consequences of not detecting changes of GP zones to β phases, conductivity properties were applied. Hardness, strength and electrical conductivity have almost linear line correlation as seen in figure 4-28. As it was expected, results of testes verfied each other.

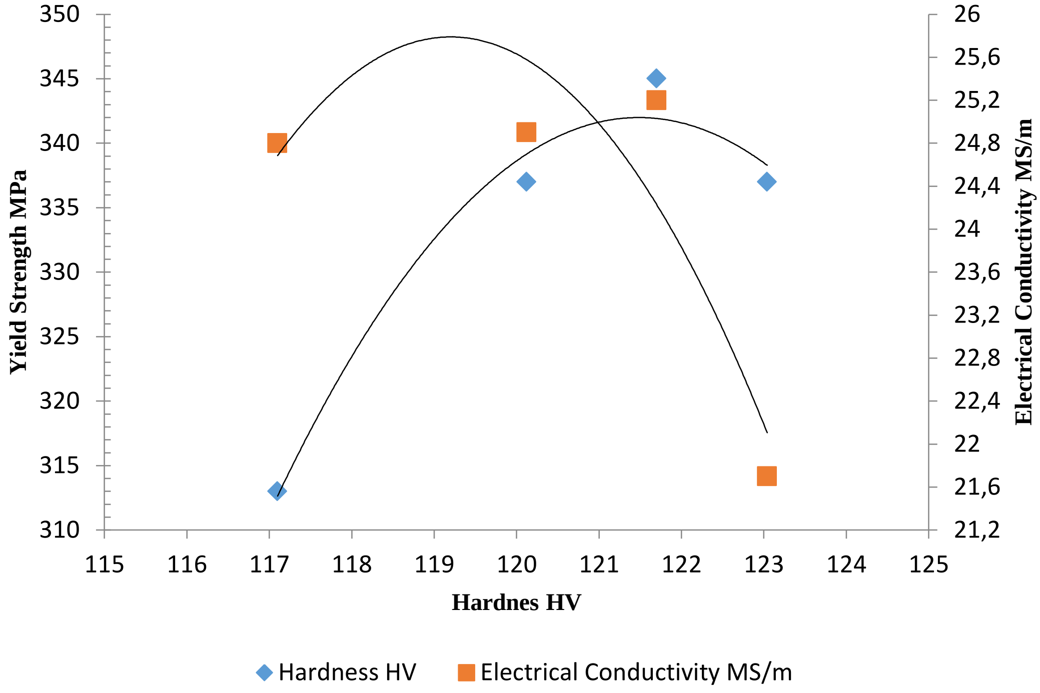



**Figure 4-28:** Relationship between strength, hardness and electrical conductivity values of aged at 200°C sample

Mechanical properties variation gave signal about forming or dissolving intermetallic during heat treatments. The XRD results supported these signs, too. Changing amount of intermetallic phases was seen clearly in figure 4-29. As the strength and hardness values increased, peak intensities of AlFe, $Mg_2Si$ and $Al_2Cu$ intermetallic increased too. After 4 hours treatment, reduction of the peak intensity was pursued in XRD results due to over aging.

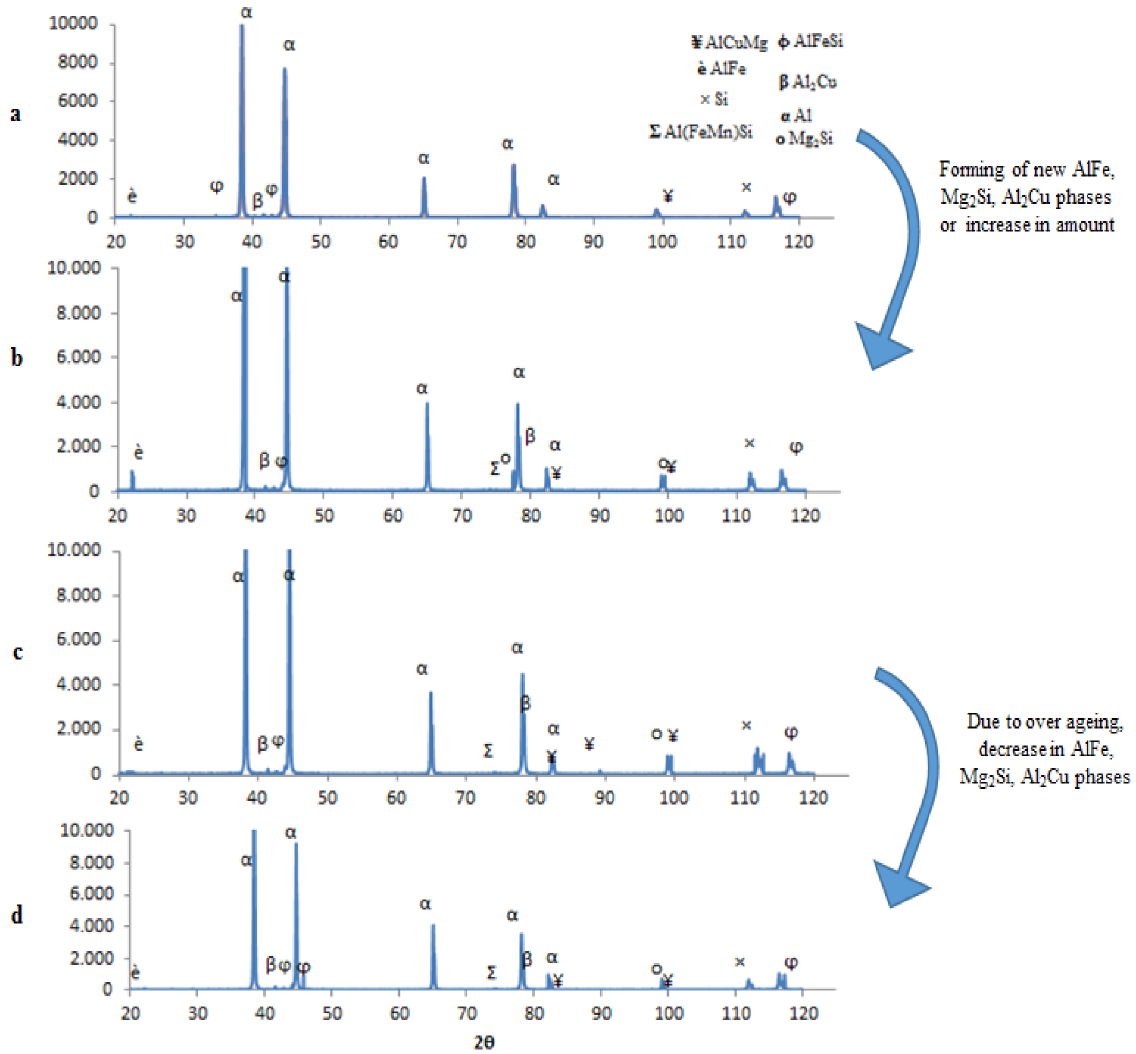



**Figure 4-29**: XRD results of heat treated at 200 $^{0}$C AA6111 for a. natural aged b. 90 min . 180 min d.360 min

Figure 4-30 shows the results of 180 $^{0}$ bending test of samples obtained after T4 and T6 heat treatment test samples. Firstly, annealing process between hot and cold rolling was not applied. Therefore, roller marks occurred on the surface of strip since aluminum is a very soft material. Even grinding surface, these marks could not be eliminated. After hot rolling, applying annealing process decreased and weakened these marks. As a results, no crack can be seen at the surface [42]. At the presence of Fe in the composition of AA6111 alloy, metastable phase $Fe_3Al$ may be formed.

When it transforms to stable phase it combines with Mn which causes sides for nucleation of new small grain during recrystallization. This recrystallization during hot rolling may ropping effect during forming as figure 4-30 a [14]. Therefore, annealing was done also to prevent ropping on the surface by getting rid of the Mn effect [14]. On the other hand, this intermetallic have harmful effects by reducing stress and fatigue resistance acting stress raiser and reducing ductility [5].

Results of bending test means that T4 and T6 has enough ductility for forming applications. However, T4 was chosen to be on the safe side by controlling the elongation values. In brief, T4 was better choice to obtain optimum combination of strength and ductility. On the other hand, T6 treatment has some more steps. This means that more energy and time are required. Production time and cost are major parameters in manufacturing [65].

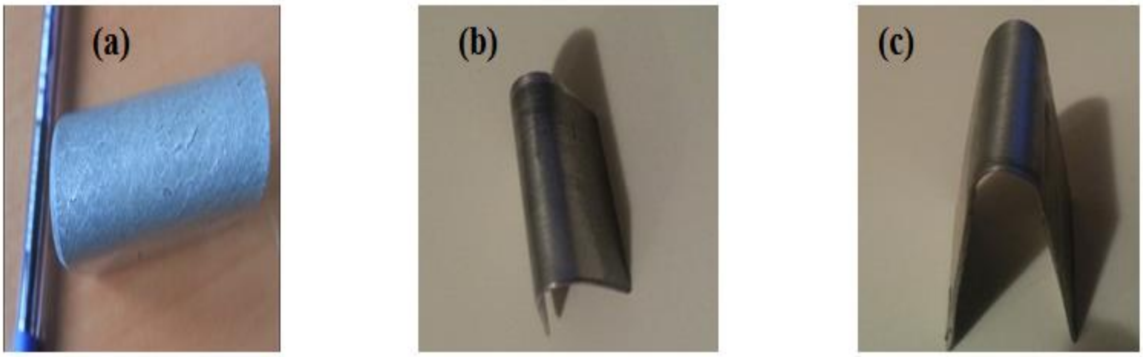



**Figure 4-30:** 180 $^0$ bending test a.T4 sample without annealing b.T4 sample with annealing c.T6 sample with annealing

Hem forming is an important forming application used in the panels of automobiles. During manufacturing of car, pieces are sustained $90^0$ bending, $180^0$ hemming operations. The probability of occurring crack under this operation is high. Having high deformation was one of the reasons to see crack. Relatively large precipitates and inclusions which stay at grain or/and grain boundaries may cause crack. Sizes of precipitates are effective in forming [4]. Precipitation size of T6 is larger than T4. Occurring crack problem after T6 heat treatment had higher potential. Erichsen test was applied to see the sheet strecthability of forming. The test was completed when a crack is seen on the surface of samples. This test gave opinion about deep drawability of samples. The elongation height was less compared to literature. Because, casting was not successful and there were large and dense inclusions.

**Figure 4-31:** Erichsen test specimens

**Table 4-4**: Erichsen test results of AA6111 alloy T4 and other alloys stayed in literature [66]

| Samples | Elongation Height (mm) | | | | |
|---|---|---|---|---|---|
| | AA6111 | AA6016 | AA6005 | AA6063 | AA6013 |
| #1 | 9.9 | 18.2 | 18.4 | 17.9 | 15.3 |
| #2 | 8.7 | 18.2 | 18.0 | 17.2 | 15.0 |
| #3 | 10.7 | 18.2 | 17.9 | 16.6 | 14.7 |

# CHAPTER 5

## CONCLUSIONS

To show the relation between mechanical properties such as strength and hardness variation, microstructure and intermetallic phases, samples from each heat treatment steps were analyzed. Then, in order to find the optimum heat treatment flow chart for energy, time and cost consumption, two variables which are optimum ageing temperature and time were investigated. By the virtue of this study, results and observations may be clarified as bellows;

- Microstructure of as-cast AA6111 alloy includes intermetallic phases which are lath-shaped β-Al5FeSi, Al15(MnFe)3Si2, $Mg_2Si$ stayed on grain boundaries and spherical Al5Cu2Mg8Si6 intermetallic stayed in the grain interiors with respect to result of XRD and EDS..

- By examining images of optic microscope and SEM microstructures besides, applying XRD and EDS analysis and finally comparing literature evidence intermetallic phases of AA6111 heat treated samples were identified as α-Al, βAl5FeSi, Al(FeMn)Si, Al9Mn3Si, AlFe, $Mg_2Si$ and $Al_2Cu$.

- Before rolling, the roller was needed to be heat up to 80 $^0$C. Otherwise, there would be crack at the edges of sheets due to thermal differences.

- Heat treatable alloy 6111 sheets were hardened by solution heat treatment and quenching. When AA6111 alloys sheets are cold rolled before ageing, formed dislocations provides nucleation side for precipitation during ageing. Besides esthetic care, it is useful for mechanical properties, too. [67]

- After hot rolling, sheets were needed to be annealed to eliminate the roller marks before cold rolling. Therefore, surface quality was improved and sheets most probably not fail during bending operations.

- Qualitative and quantitative results show that produced materials can be used in production of car body panel parts. Quantitative mechanical results are able to meet production requirements either after T4 or T6 treatments. During T4 heat treatment, sheets were gained sufficient ductility then, during T6 treatment sheets were aged so that sheets were obtained sufficient a high strength to improve properties as dentability and/or rigidity. Plus, qualiltaltievely surface quality was met esthetic expectations.

- As seen in the microstructures, different precipitates occur at different aging times and temperatures. As the temperature increases, diffusion increases and precipitation forms completed quickly so optimum hardening value was obtained in a shorter time. This is important for production speed which is one of the most important issues for automotive manufacturers. At temperature 200 $^{0}$C and after ageing 90 min, the max hardness value as 123 HV hardness values was obtained.

- As the time and temperature of ageing was increased, the intermetallic might have been got larger, the divergence of intermetallic might be grown or intermetallic might dissolve in matrix again by time. Terminally, these lead to decrease in hardness values which are described as peak aged. Peak aged condition was noted as 160 °C for 6 hours

- The maximum strength alloy was produced after aging at 200 $^{0}$C for 90 minutes which provides reduction in cost and gaining time.

- The deformation percent as 40% CW was not enough for observing mechanical properties. Minimum grains size was measured as almost 100 μm. Optimum grain size is between 1.5-50 μm for successful sheet drawing. Thicker sheet should be used to obtain enough reduction.

- Results of conductivity test showed that, amount of GP zones was high for solutionized and T4 temper samples. Therefore, they had got minimum conductivity. As ageing temperature increased, conductivity result increased, too. Dissolution of GP zones and growing size of precipitation, forming of stable β'' phase had positive effect on mobility of electrons. As a result, conductivity of samples increased.

- According to Erichsen test results, the specimen elongated about 9.8 mm. Moreover, the crack was observed after 6.8 KN force was applied. The height difference was not good compared to literature. This was most probably due to inclusions which probably came from casting condition. For improving the test results, cleaner microstructure was needed.

# CHAPTER 6

# SUGGESTED FUTURE WORK

Some aspects are still need to be clarified and improved in this experiment. During experiment, mechanical properties were measured less than standard. However; when the test samples were examined, dense of inclusions were observed which probably came from casting conditions. Therefore, it is need to provide inert atmosphere condition during casting so surface oxidation can be eliminated. Moreover, in filter system can be used to prevent oxidation which is formed inside. Besides; improving of mechanical properties can be provided by forming finer grains size. Therefore, it is needed more %CW and more thick slab should be casted and then, be rolled.

## APPENDIX A

| Elements | Weight (g) |
|---|---|
| **Al** | 2565 |
| **Si** | 20 |
| **Cu** | 20 |
| **Mn** | 19 |
| **Zn** | 2 |
| **Mg** | 20 |
| **FeCr** | 2.8 |
| **Total Weigth** | 2648.8 |
| **Grain Ref. (AlTiB)** | 42 |
| **Σ** | **2690.8** |

## APPENDIX B

| Samples | Ageing Time (min) | Hardeness ($HV_{0.5}$) | | | | | | | | | | Av. Hardness ($HV_{0.5}$) | Standard Deviation | Hardness with Error ($HV_{0.5}$) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| As rolled | 0 | 80 | 79 | 79 | 76 | 78 | 78 | 85 | 79 | 85 | 77 | **80** | 3 | 80 ± 3 |
| Solutionized | 0 | 79 | 81 | 78 | 80 | 80 | 82 | 78 | 81 | 87 | 78 | **79** | 3 | 79 ± 3 |
| Aged at 160 °C | 90 | 123 | 127 | 104 | 100 | 105 | 108 | 107 | 102 | 106 | 101 | **108** | 9 | **108 ± 9** |
| | 180 | 114 | 120 | 122 | 121 | 116 | 118 | 129 | 122 | 124 | 117 | **120** | 4 | **120 ± 4** |
| | 240 | 106 | 107 | 112 | 108 | 115 | 115 | 107 | 101 | 105 | 106 | **108** | 4 | **108±4** |
| | 360 | 95 | 95 | 95 | 92 | 98 | 94 | 97 | 98 | 93 | 95 | **95** | 2 | **95 ± 2** |
| Aged at 200° C | 90 | 129 | 118 | 125 | 127 | 118 | 124 | 118 | 124 | 128 | 120 | **123** | 4 | **123 ± 4** |
| | 180 | 126 | 112 | 118 | 117 | 124 | 121 | 118 | 125 | 120 | 121 | **120** | 4 | **120 ± 4** |
| | 240 | 130 | 120 | 120 | 113 | 117 | 120 | 121 | 119 | 130 | 127 | **122** | 5 | **122 ± 5** |
| | 360 | 119 | 117 | 116 | 118 | 117 | 118 | 116 | 115 | 118 | 117 | **117** | 1 | **117 ± 1** |
| Aged at 240° C | 90 | 74 | 86 | 89 | 87 | 89 | 90 | 87 | 89 | 89 | 89 | **87** | 4 | **87 ± 4** |
| | 180 | 79 | 85 | 84 | 81 | 85 | 89 | 87 | 91 | 84 | 85 | **85** | 4 | **85 ± 4** |
| | 240 | 88 | 87 | 88 | 82 | 84 | 84 | 86 | 83 | 86 | 81 | **85** | 2 | **85 ± 2** |
| | 360 | 96 | 82 | 79 | 79 | 76 | 75 | 80 | 82 | 81 | 75 | **81** | 6 | **81 ± 6** |

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