DOKUZ EYLÜL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

T6 HEAT TREATMENT OPTIMIZATION OF A356 ALLOY

by
Alper GÜNEREN

July, 2019
İZMİR

T6 HEAT TREATMENT OPTIMIZATION OF A356 ALLOY

A Thesis Submitted to the
Graduate School of Natural and Applied Sciences of Dokuz Eylül University
In Partial Fulfillment of the Requirements for the Degree of Master of Science in
Metallurgical and Materials Engineering

by
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M.Sc THESIS EXAMINATION RESULT FORM

We have read the thesis entitled **"T6 HEAT TREATMENT OPTIMIZATION OF A356 ALLOY"** completed by **ALPER GÜNEREN** under supervision of **ASST. PROF. DR. ESRA DOKUMACI** and we certify that in our opinion its is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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ACKNOWLEDGMENT

First and foremost, I would like to thank my advisor Dr. Esra DOKUMACI for her support, patience and scientific guidance throughout the course of this thesis.

I would like to owe my special thanks to H. Emre ÇUBUKLUSU for his support and providing useful suggestions about this thesis. I would like to express my gratitude to Caner KALENDER and Bora YAY for their friendship and helps. I would also like thank each person who work in CMS Jant ve Makina Sanayi A.Ş. R&D and Foundry Departments.

Finally and deeply, I would like to thank my mother Filiz GÜNEREN, my father Sabri GÜNEREN and my brother Berkay GÜNEREN for believing in me and supporting me throughout my graduate career.

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T6 HEAT TREATMENT OPTIMIZATION OF A356 ALLOY

ABSTRACT

In this study, the effect of heat treatment stages on the yield-ultimate tensile strength, the hardness and the elongation of the wheels produced by low-pressure die-casting technology using Sr-modified A356 alloy, is examined. The study consists of fundamentally four steps as follow; investigation of the effect of current heat treatment stages and dye process, the effect of the solution treatment on the eutectic silicon morphology and the mechanical properties, determination of the main factors affecting the mechanical properties through the design of experiment and optimizing the conditions, and the effect of the quenching phase on the cooling rate and the mechanical properties. The experiments were carried out with temperature controlled laboratory furnaces. Tensile and hardness testing was applied to the specimen taken on the wheel and microstructure analyzes were performed by using an optical microscope. As a result of the study, it was determined that 480°C is not sufficient to provide a supersaturated solution for catalyzing the precipitation hardening and the elongation values can be enhanced through the proper spherodization of the eutectic silicons. Solution treatment has no effect on yield-ultimate tensile strength and hardness by oneself. Whereas it was determined that values vary considerably when the same aging or painting process applied after different solution treatments. According to design of experiment and regression analysis, the formulas were obtained based on solution temperature, solution time, artificial aging temperature, and artificial aging time. Optimal heat treatment parameters were determined for desired mechanical properties. Cooling rates and Leidenfrost points were obtained at different quenching mediums.

Keywords: A356 alloy, wheel, T6 heat treatment, optimization, quenching

A356 ALAŞIMI T6 ISIL İŞLEMİ OPTİMİZASYONU

ÖZ

Bu çalışmada, Sr-modifiyeli A356 alüminyum alaşımı kullanılarak alçak basınçlı döküm teknolojisi ile üretilen jantlarda, ısıl işlem aşamalarının akma-çekme mukavemeti, sertlik ve uzama değerleri üzerindeki etkisi incelenmiştir. Çalışma temel olarak; mevcut ısıl işlem aşamalarının ve boya prosesinin etkisinin incelenmesi, çözeltiye alma işleminin ötektik silisyum morfolojisi ve mekanik özellikler üzerindeki etkisinin incelenmesi, deney tasarımı yöntemi ile mekanik özellikleri etkileyen ana faktörlerin belirlenmesi ve şartların optimize edilmesi, su verme aşamasının mekanik özellikler ve soğuma hızı üzerindeki etkisinin incelenmesi olarak dört ana bölümden oluşmaktadır. Deneyler sıcaklık kontrollü laboratuvar tipi fırınlarda gerçekleştirilmiştir. Jant üzerinden alınan numunelere çekme ve sertlik testi uygulanmış, optik mikroskop ile mikroyapı analizleri yapılmıştır. Çalışma sonucunda aşırı doymuş katı özelti oluşturmak için 480°C'de çözeltiye alma işleminin yetersiz olduğu, uygun çözeltiye alma işleminin ötektik silisyumların küreselleşmesiyle uzama değerlerini önemli ölçüde arttırdığı, ancak akma ve sertlik değerlerini tek başına etkilemediği anlaşılmıştır. Bunun yanı sıra, farklı çözeltiye alma ve suni yaşlandırma kombinasyonlarında akma ve sertlik değerlerinde önemli değişkenlikler gözlemlenmiştir. Deney tasarımı ve regresyon analizine göre, çözeltiye alma sıcaklığı, çözeltiye alma süresi, suni yaşlandırma sıcaklığı ve suni yaşlandırma süresine bağlı formulasyonlar oluşturulmuştur ve istenilen mekanik özelliklere göre optimum şartlar belirlenmiştir. Farklı su verme ortamlarında yapılan deneyler sonucunda soğuma hızları ve Leidenfrost noktaları elde edilmiştir.

Anahtar Kelimeler. A356 alaşımı, jant, T6 ısıl işlemi, optimizasyon, su verme

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CHAPTER ONE
INTRODUCTION

Aluminum is an important element which exists on earth in different mineralogical forms such as Al_2O_3, corundum, bauxites, or bonded with silica and alumina-silicates (Lumley, 2011; Totten & MacKenzie, 2003). The metallurgical process is quite difficult due to its affinity to oxygen and the tendency of forming the oxide layer. Metallic aluminium is produced with using Bayer and Hall-Heroult processes consecutively**.**

For the last decades, its usage ratio in the industrial area only behind ferrous alloys.The extensive use of Aluminium lies in its strength, density ratio, toughness, low melting degree, good formability, weldability, high heat and electrical conductivity and its corrosion resistance (Ahmad, 2012; Kvačkaj & Bidulský, 2011). In these days, to decrease energy comsumption, otomotive industry need to alleviate car’s weight. At the same time low weight critical safety components must provide the technical requirements. Light aluminium alloy wheels are one of the major cosmetic parts of the vehicles and these parts have to meet high impact and fatigue requirements (Manente & Timelli, 2011).

Aluminium-Magnesium-Silicon alloys are the most demanded and widely used casting alloys in wheel industry (Manente & Timelli, 2011; Ma, 2006) because of their excellent fludity, high mechanical properties and lightness (Abdulwahab, Madugu, Yaro, Hassan & Popoola, 2011). However, without any heat treatment, these alloys are used in the as-cast condition because they exhibit deficient elongation (toughness) due to the coarse acicular plates eutectic silicons. This phases increases internal stress under an applied load (Paz, 2003; Ji-hua, Xiao-long, Jian-ting & De-ying, 2011). Modifying the microstructure and enhancing the properties of alloy, is only possible by applying such specific heat treatment after the casting process.

T6 heat treatment is widely applied in the wheel industry and stages of the process are; solution treatment, quenching, and artificial aging (Brooks, 1991). Altough T6 is an important process to enhance mechanical properties; high temperature (540°C) that using in solution treatment in order to attain homogeneous microstructure, and (180°C) in aging for enhancing mechanical properties with precipitation, increases natural gas and electricity consumption. Optimization of the amount of heat and time of the process is important for energy saving because solution treatment and artificial aging stages cause approximately 25% of natural gas consumption of the company. In quenching stage, wheels immersed in a cooling medium from high temperature to room temperature. Cooling should be fast enough to obtain supersaturated solid solution. Contrarily, cooling should be slow enough to refrain distortions and extensive residual stresses. Determination of the critical cooling rate is essential because of this conflict. Although many investigations examined the quenching phenomenon, only few references in the literature systematically describe the effect of cooling mediums for A356 aluminium alloy.

Tos um up, excess energy consumption and the distortion problems are the main disadvantages of wheel heat treatment process. Purpose of this study is optimizing the T6 heat treatment parameters. The findings of this thesis will use to determine the heat treatment conditions of the company in future.

In this study, the effect of heat treatment stages on the yield-ultimate tensile strength, the hardness and the elongation of the wheels produced by low-pressure die-casting technology using Sr-modified A356 alloy, is examined. The study consists of fundamentally four steps as follow; investigation of the effect of current heat treatment stages and dye process, the effect of the solution treatment on the eutectic silicon morphology and the mechanical properties, determination of the main factors affecting the mechanical properties through the design of experiment and optimizing the conditions, and the effect of the quenching phase on the cooling rate and the mechanical properties.

CHAPTER TWO
ALUMINIUM ALLOYS

In non-alloyed state, pure aluminium is a comparatively soft material with a yield strength of 34.5 MPa, a tensile strength of 90 MPa and density of approximately 2.7 gr/cm^3 (Sheasby & Pinner, 2001).

To generate different type of aluminium alloys, Silicon (Si), Magnesium (Mg), Copper (Cu), Zinc (Zn), Manganese (Mn), and Nickel (Ni) are used. All of these elements are added into the pure aluminium to enhance the mechanical properties. Adding Cu and Mg makes the alloy heat treatable by means of precipitation hardening mechanism.

2.1 Clasification of Aluminium Alloys

Aluminium alloys are classified by means of their production process, the heat treatment ability and the chemical composition (Mazzolani, 1995). There are two type of aluminium alloys as casting and wrought alloys. Invesment casting, semi-solid casting, sand casting, gravity die casting, low pressure die casting (LPDC), high pressure die casting (HPDC), squeeze casting, thixocasting are the main casting methods. Wrought alloys are cast in ingots or billets then worked (cold or hot) mechanically and transformed into various forms as extrusions, plate, sheet, forgings, foil, tube and wire (Sheasby & Pinner, 2001).

2.1.1 Designation of Aluminium Alloys

In order to describe wrought alloys 4 digit numerical system is used as given in Table 2.1. The first digit shows the group of alloy. The second digit indicates modifications (from 1 to 9) of the original alloy or impurity limits. The last two digits indicate the aluminium alloy or the aluminium purity.

Table 2.1 Wrought aluminum alloy designation system (Cayless, 1991)

Series	Alloying elements
1XXX	Controlled unalloyed (pure)compositions
2XXX	Alloys in which copper is the principal alloying element, though other elements, notably magnesium, may be specified
3XXX	Manganese is the principal alloying element
4XXX	Silicon is the principal alloying element
5XXX	Magnesium is the principal alloying element
6XXX	Magnesium and silicon are principal alloying elements
7XXX	Zinc is the principal alloying element, but other elements such as copper, magnesium, chromium, and zirconium may be specified
8XXX	Tin and some lithium compositions characterizing miscellaneous compositions

Casting alloys are shown with 4 digit numerical designation as given in Table 2.2. A modification of the original alloy or impurity limits is indicated by a serial letter before the numerical designation (exp. A356.2, B356.0). The first digit shows the group of alloy. The second two digits determine the alloy or indicate the aluminium purity. The last digit that separated by a point identify the production type such as. castings (0) or ingot (1, 2).

Table 2.2 Cast aluminium alloy designation system (Cayless, 1991)

Series	Alloying elements
1XX.X	Controlled unalloyed (pure) compositions, especially for rotor manufacture
2XX.X	Copper is the principal alloying element, but other alloying elements may be specified
3XX.X	Silicon is the principal alloying element, but other alloying elements such as copper and magnesium are specified
4XX.X	Silicon is the principal alloying element
5XX.X	Magnesium is the principal alloying element
7XX.X	Zinc is the principal alloying element, but other alloying elements such as copper and magnesium may be specified
8XX.X	Tin is the principal alloying element

Individual capital letters identifies the basic temper designations. Sub-branches of the main process are named with extra digits. Different type of temper conditions can be applied during heat treatment to obtain differ properties of the alloys. Basic temper designation and systems are given below Table 2.3 and 2.4.

Table 2.3 Basic temper designations (Cayless, 1991)

Designation	Treatment
F	As-Fabricated
O	Annealed
H	Strain-Hardened
T	Solution Heat-Treated

Table 2.4 Temper system for aluminium alloys (Cayless, 1991)

Treatment	Stages
T1	Cooled From an Elevated-Temperature Shaping Process and Naturally Aged to Substantially Stable Condition
T2	Cooled From an Elevated-Temperature Shaping Process, Cold Worked, and Naturally Aged to a Substantially Stable Condition
T3	Solution Heat Treated, Cold Worked, and Naturally Aged to a Substantially Stable Condition
T4	Solution Heat Treated and Naturally Aged to a Substantially Stable Condition.
T5	Cooled From an Elevated-Temperature Shaping Process and Artificially Aged.
T6	Solution Heat Treated, Quenched and Artificially Aged
T7	Solution Heat Treated and Overaged or Stabilized
T8	Solution Heat Treated, Cold Worked, and Artificially Aged

2.1.2 Al-Si-Mg Foundry Alloys

Al-Si alloys are composed of grains (1mm-10mm), dendrites (typically 10µ -150µ DAS), eutectic Silicons (2mm acicular shapes or 1 µm round particles), intermetallics and inclusions. Hard Si particles improve the wear resistance of aluminium alloys (Ernst, 2004).

In terms of the amount of atomic percentage of Si, the Al-Si casting alloys are seperated into 3 groups as shown in Fig. 2.1 and 2.2. Alloys that include 5%-10% Si are named Hypoeutectic alloys. Commonly, eutectic alloys contain 10%-14% Si, and hypereutectic alloys contain 14% and 20% Si (Paz, 2003).

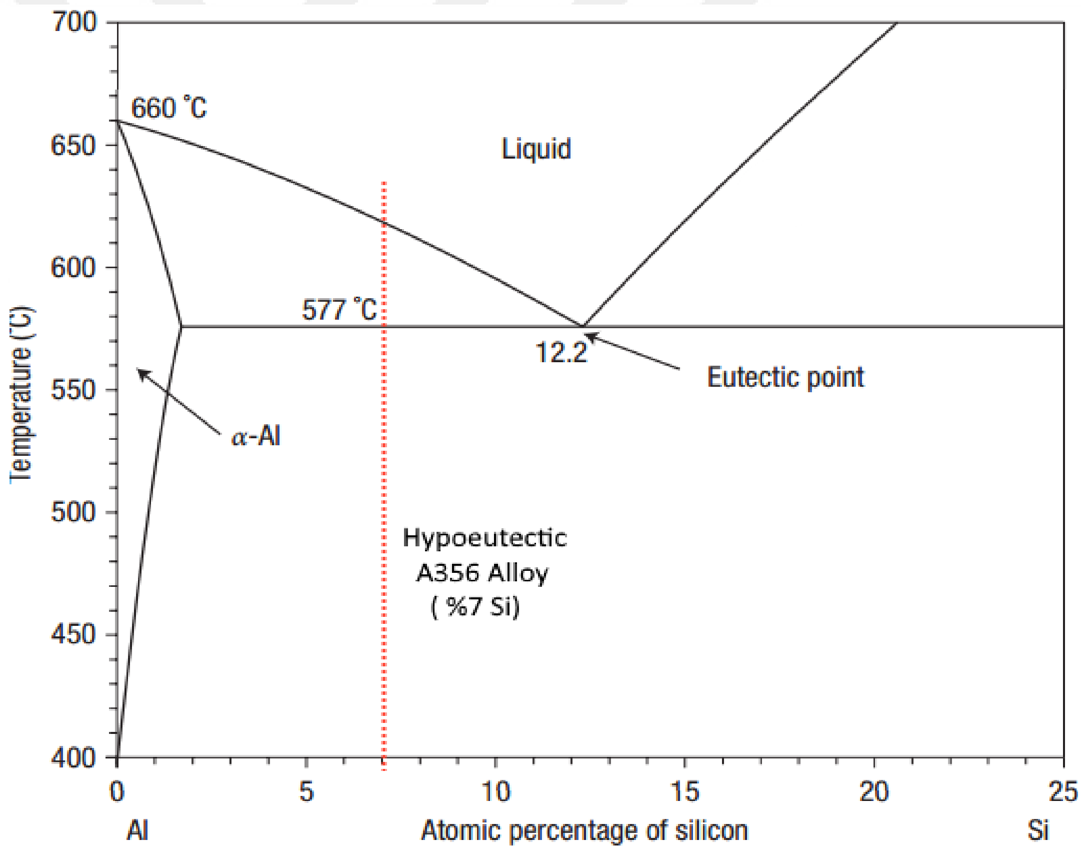


Figure 2.1 Al–Si binary phase diagram (Wang, Schmidt, Senz & Gösele, 2006)

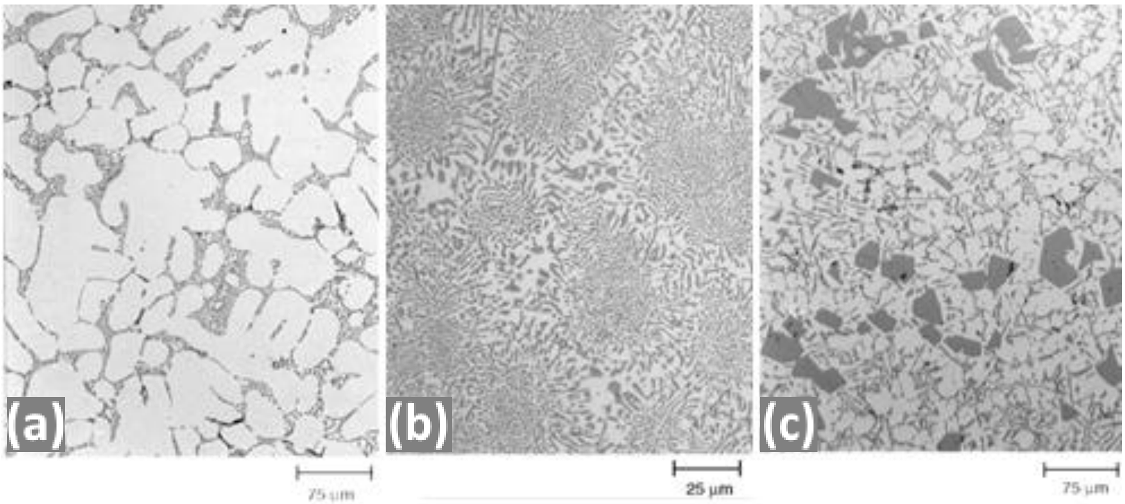


Figure 2.2 (a) Microstructure of hypoeutectic alloy. (b) eutectic alloy. (c) hypereutectic alloy (Warmuzek, 2004)

"Magnesium is the main solid solution strengthener to Al–Si alloy (to form Al–Si–Mg alloys) and its addition leads to increase in response to precipitation hardening which in turn results in higher yield strength in these alloys (Abdulwahab et al., 2011)." Mechanism of precipitation is shown in section 3.2.3 in detail.

Hypoeutectic cast A356 alloys (Al–7Si–0.3Mg) is common industry products. Chemical composition of A356.0 is given in Table 2.5.

Table 2.5 Composition of A356.0 aluminium alloy

	Si	Fe	Cu	Mn	Mg	Zn	Ti	Unspecified Other elements	Al min.
A356.0	6.5-7.5	0.20	0.20	0.10	0.25-0.45	0.10	0.20	0.15	Rem.

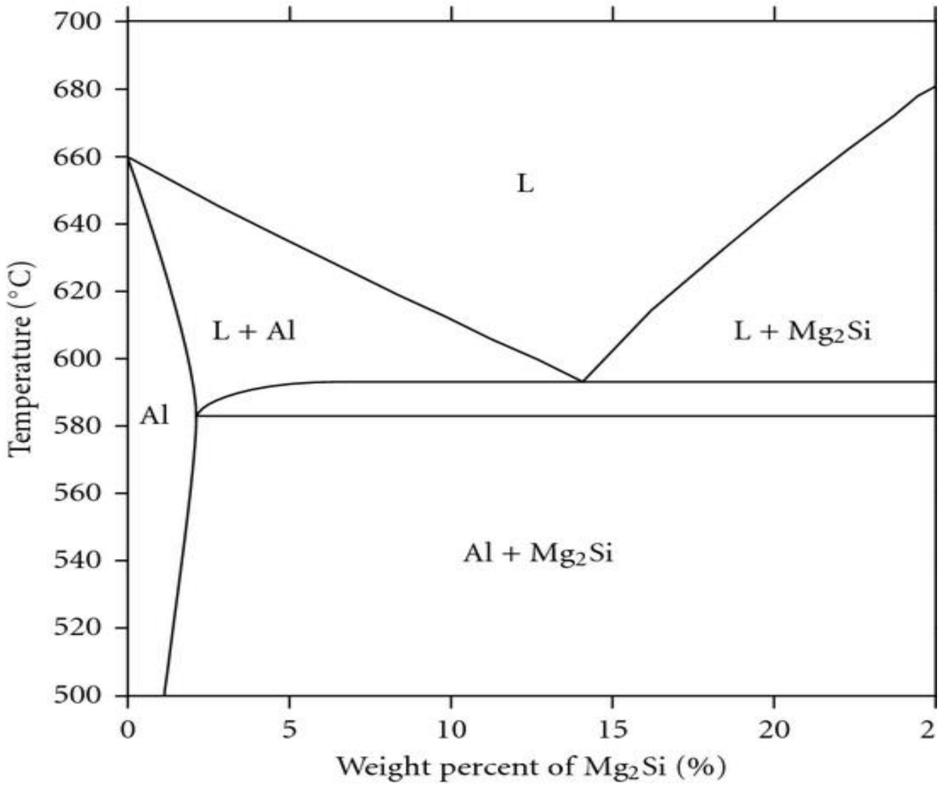


Figure 2.3 Phase diagram of Al–Mg_2Si pseudobinary section (Malekan, Emamy, Rassizadehghani & Malekan, 2012)

A356 alloys are consisted of soft primary α-Al dendrites, long acicular eutectic silicons and hard Mg_2Si which decreases the toughness and limits the usage ratio in industry (Zhu, Jian, Yang & Zhou, 2012). Al–Mg_2Si pseudo-binary diagram is shown above as Fig. 2.3. The mechanical properties such as elongation, hardness and yield strength depends on the microstructures of the alloys and morpholgy of silicion particles (size, shape and distiribution) (Shabestari & Shahri, 2004). Despite the high melting temperature (1085°C), low coefficient of thermal expansion (7.5×10-6 K^{-1}), low density (1.99 g/cm^3), high hardness (4.5 GPa), and high elastic modulus, plate type Mg_2Si phase is not desirable phase for toughness. Besides that its presence in the form of large blocky or Fe-based α-Chinese script shape and Fe-based β particles significantly decreases the alloy’s mechanical properties (Mandal & Makhlouf, 2016). Heat treatment changes the form of the Mg_2Si phase, fine Mg_2Si particles precipitate into the Al matrix homogeneously, therefore, mechanical properties are enhanced.

2.1.2.1 Modification of Eutectic Silicon

"Morphology of eutectic silicons are changed by the rapid solidification, the chemical modification and the thermal modification in the solid state (Shabestari & Shahri, 2004, p. 2023)."

First of all, the casting technique and solidification rate affect the microstructure of casting parts. Low-pressure die casting provides rapid and directional solidification and dendritic structure. To obtain better mechanical properties, size of DAS (Dentrite Arm Spacing) and SDAS (Secondary Dentrite Arm Spacing) must be reduced and eutectic silicons must be distributed homogeneously.

In order to transform the acicular shape eutectic silicon to the fibrous type, Stronsium (Sr), Sodium (Na), Potasium(K), Calcium (Ca) and Cerium (Ce) is used as chemical modificator. Sr and Na are commonly used for hypoeutectic alloys in industrial application due to their modification ability. Generally, 100-300 ppm Sr is used for modification of hypoeutectic aluminium alloys. Modified alloys show higher yield and elongation properties comparing to unmodified alloys (Kaufman & Roy, 2004). Finer eutectic distribution and fibrous type silicons can be obtain with high cooling rate or chemically modifying (Ogris, 2002).

The more eutectic silicon modification, in the casting process, provides the shorter solution treatment time and modification level than be increased easily. Modification levels are given in Fig. 2.4 and 2.5 with range of 50 and 12.5 nm. Transformation of the eutectic silicon, from acicular shape to spheroidal and finer shape, can be seen in detail.

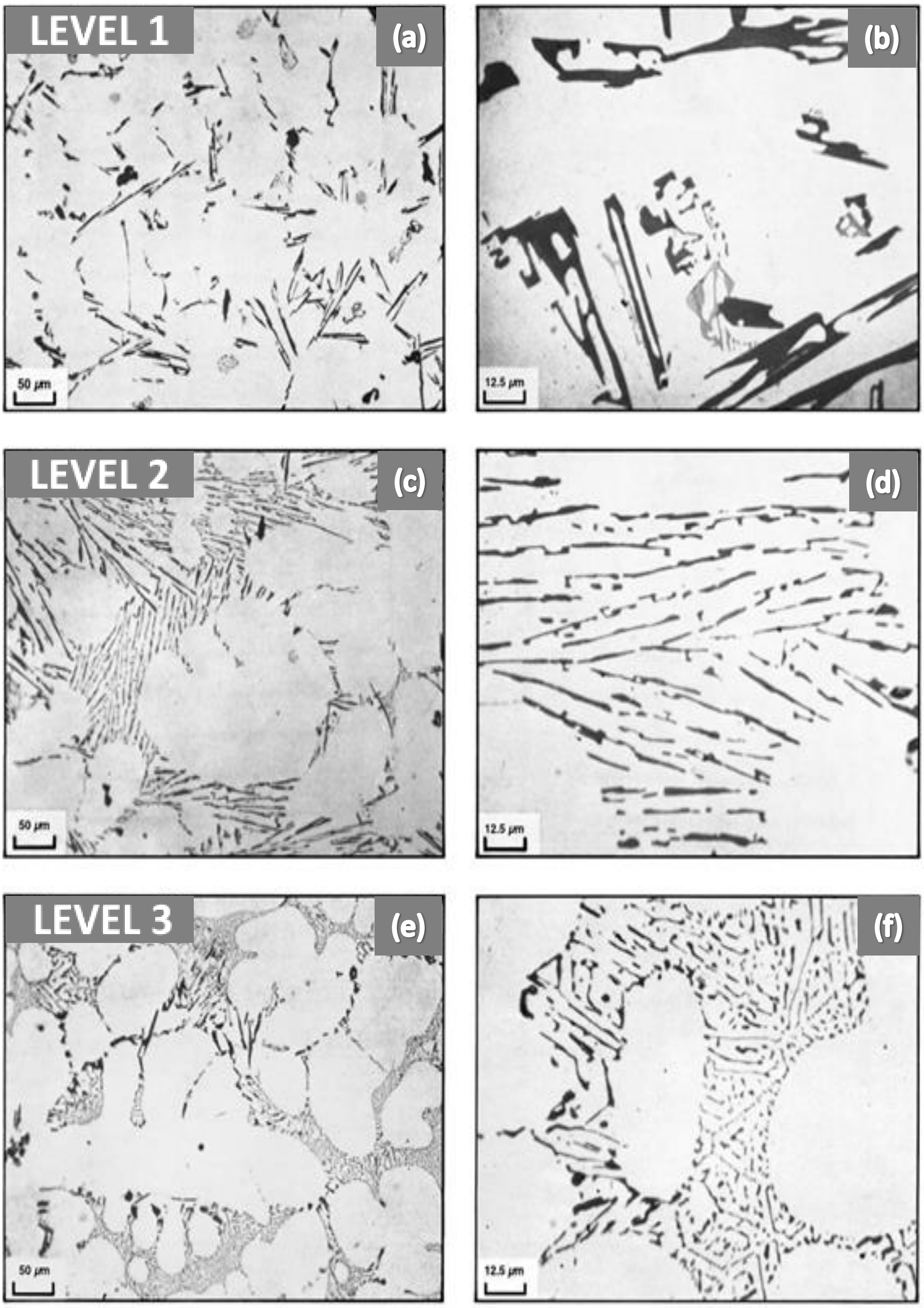


Figure 2.4 Degree of eutectic silicon modification. (a) Level 1, unmodified structure 200X. (b) 800X. (c) Level 2, lamellar structure 200X. (d) 800X. (e) Level 3, partial modification 200X. (f) 800X. (Kaufman & Roy, 2004)

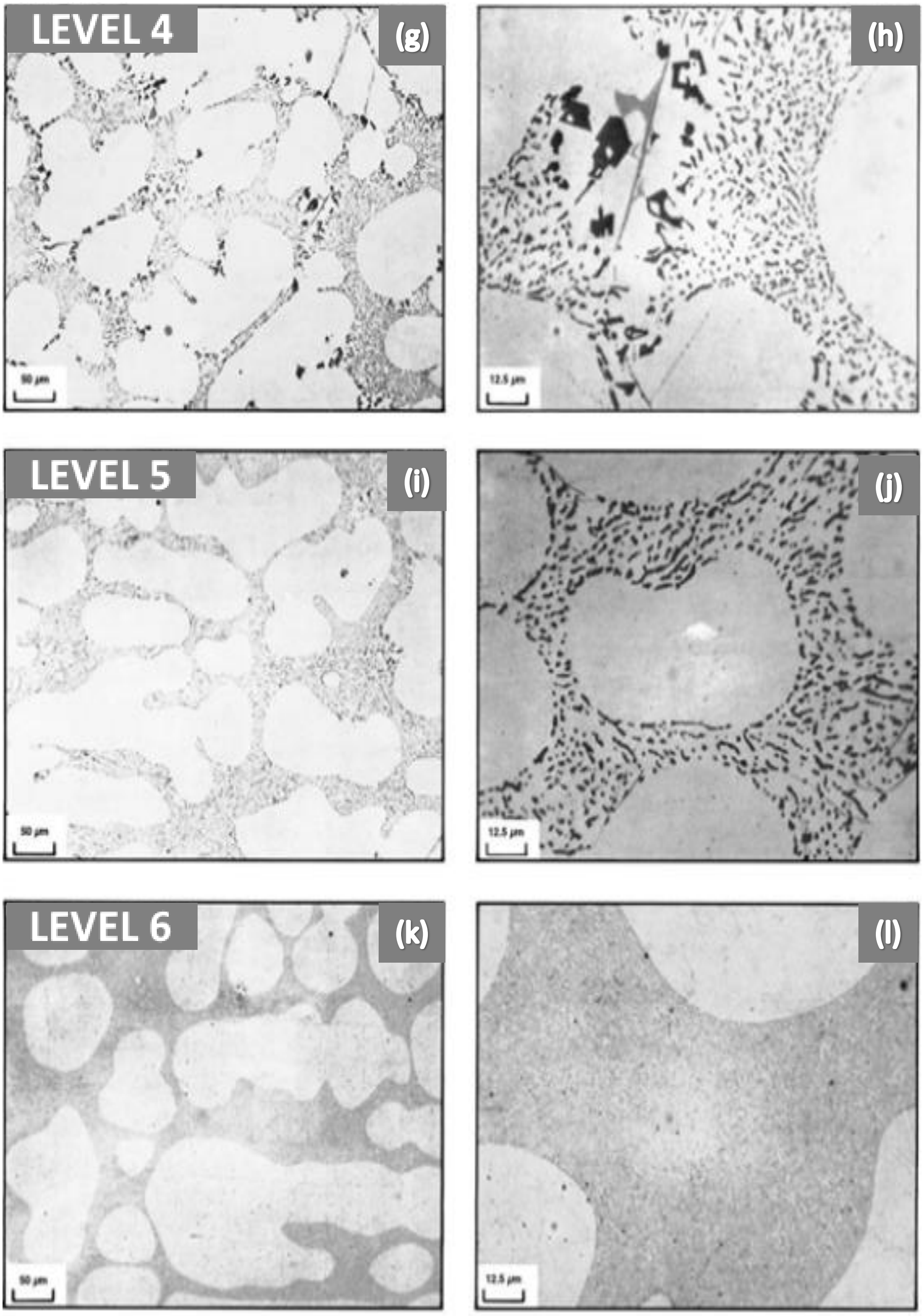


Figure 2.5 (g) Level 4, absence of lamellar structure 200X. (h) 800X. (i) Level 5, fibrous silicon eutectic 200X. (j) 800X. (k) Level 6, very fine structure 200X. (l) 800X. (Kaufman & Roy, 2004)

Finally, the thermal modification is occurred in the heat treatment process in solution treatment stage, already modified eutectic silicons are converted to fine fully spherical silicon particles in soft Al matrix as shown in Fig. 2.6 (Azadi & Shirazabad, 2012).

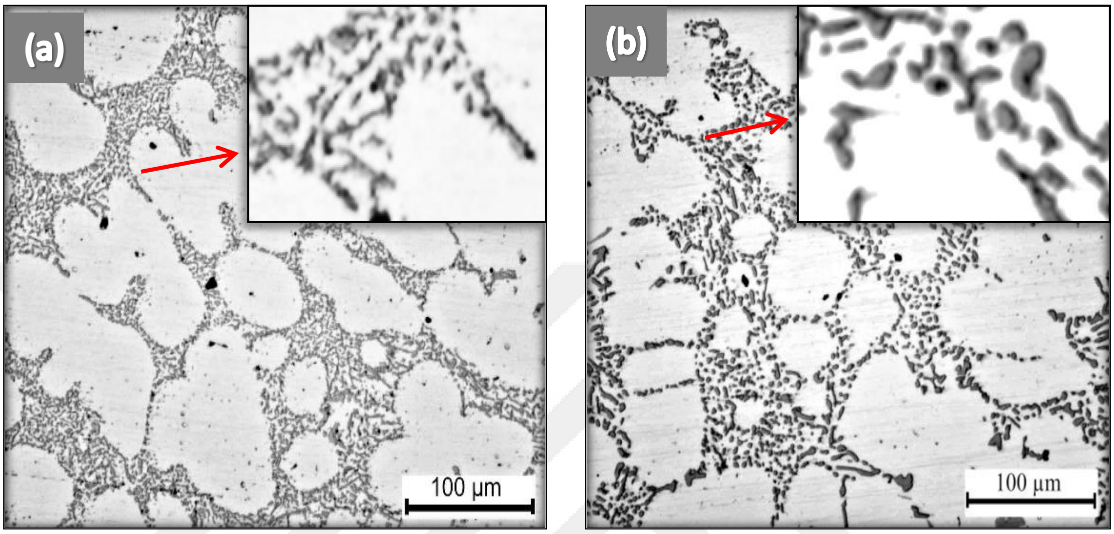


Figure 2.6 (a) Non-modified eutectic silicon (b) Spherical eutectic silicon after heat treatment

2.1.2.2 Grain Refinement

The grain refinement is the prior and best way in order to enhance mechanical properties without decreasing ductility (Muszka, Majta & Bienias, 2006). A plenty of article has been published about the grain refirenment and there have been several studies in the literature reporting that grain size is directly related to toughness, strength, fatigue properties alongside the ligthness of aluminium casting part (Birol, 2012a, 2012b). Reducing the grain size to the minimum, increase the wheel strength, thus the thickness of the wheel can be decreased and extra ligthness can be obtained.

During solidification grain growth must be decelerated because of the strength-grain size relationship (Hall-petch). In order to describe ability of restriction of grain growth, 'GFR' term is used. As given in Table 2.6. Titanium is the most coherent element with aluminium structure to use as grain refiner.

Table 2.6 GFR values of elements

	Ti	**Si**	**Cr**	**Ni**	**Mg**	**Fe**	**Cu**
GFR	246	5.9	3.5	3.3	3.0	2.9	2.8

Master alloying elements are added into liquid metal before the casting in order to attain fine grains (Fig. 2.7). Foundry companies generally use Ti-B master alloy with different ratio according to the spektrometer results. TiB_2 and Al_3Ti phases that comprise particles must distribute into melt homogeneously.

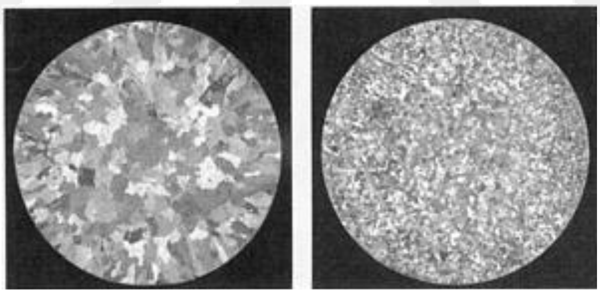


Figure 2.7 Grain refinement effect (a) No grain refiner, coarse grains. (b) fine grains (Rooy, 1992)

Together with material properties, heat treatment conditions are also effected by grain size. Hence, refirenment process must be carried out adequately. Otherwise, solution treatment time might extend and spending excess time causes stepdown of production rate and quantity. Not only treatment time but also process temperature might be increased.

2.2 Production of Light Alloy Wheel

2.2.1 Moulding

Metal moulds, in other words, permanent moulds are used for producing wheel at low pressure die casting technique. Moulds are designed by using such software as; Catia, Unigraphics, Solidworks, Sutcast etc. and the computer aided manufacturing system is used to produce. Mould consists of three part; upper, lower and sider core. Cores cover with refractory based sodium silicate material in order to provide better casting and non-wetting property. Cores have a certain lifetime, therefore, maintenance is made periodically. Sandblasting, welding, machining and coating are applied for preparing the mould to cast when cores damaged. Before setup to low pressure die casting machine, the mould should be heated up to 400-500°C to avoid casting defect.

2.2.2 Melting and Degassing

Striko furnace consists of melting and holding units. Primary aluminium ingots, wastage wheels and aluminium scraps are loaded to the melting unit of Striko. Liquid metal which is melted by using natural gas, are passed to holding unit. Brulor is burned for keeping the metal temperature stable in holding unit. Secondary aluminium scraps melted via reverberatory furnace and poured to Striko holding unit. Metal is taken to transfer ladle and alloying elements (Stronsium, Titanium Diboride and Magnesium) are added according to chemical analysis.

Hydrogen (H) is the solely element that soluble in aluminium metal and it has detrimental effect on mechanical properties. Rotary degassing is carried out with an inert gasses e.g. nitrogen or argon in order to remove the hydrogen gas.

Additionally, while the rotor is turning, flux is added simultaneously. Impurities, oxides and undesirable particles are removed from liquid metal and gather the top of the transfer ladle. These undesirable particles must be cleaned carefully otherwise they may affect material properties of the final product.

2.2.3 Low Pressure Die Casting

Low pressure die casting (LPDC) is currently applied for producing light alloy wheels due to the dimensional solidification, the high production rate and reduced the finishing requirements. As illustrated in Fig. 2.8 metal is filled into a mould cavity by passing through the riser tube by using air pressure and pressure is applied during the solidification. Then the air pressure is reduced to zero and rest of the molten metal turn back into the chamber. At the end of the casting process, upper and bottom cores separate to each other and plungers push the part out. Simulation programmes are used to determine solidification behaviour or casting defects before production in the design period.

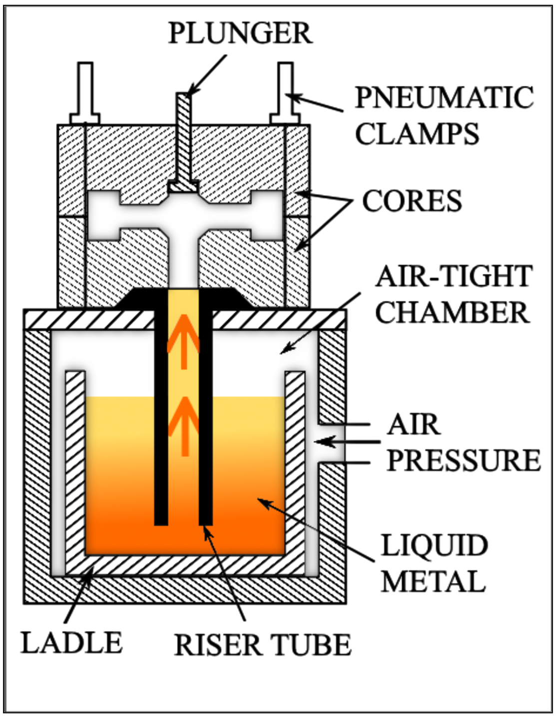


Figure 2.8 Schematically low pressure die casting working prencipe

2.2.4 Heat Treatment

Casting wheels are transferred with conveyors from casting shop to heat treatment plant which has a solution and aging treatment furnaces. These furnaces are heated up by using natural gas and electricity. Plant expends 24% and 8% of natural gas and electricity of total consumption respectively. In order to attain a homogeneous medium, hot air is circulated with fans.

Solution treatment is applied at 540°C for 240 minutes then wheels are cooled down from 540°C to room temperature. As quench medium, water at 80°C is used. After quenching stage, artificial aging is applied at 150°C for 180 minute. Quench tank temperature is held stable with circulation and all parameters are controlled and monitored by computer system.

2.2.5 Machining

Heat treated wheels are transported to machining shop. According to technical drawing, CNC programmes are prepared depending on the chosen machining tools. Machining of the wheels is done in three steps as illustrated in Fig. 2.9; in first operation, hub mating surface, spoke back surface, inner flange, center hole and inner tire contact areas are machined. Then the wheel is clamped to the chuck from the inner flange, outer rim part and tier mating surfaces are machined. As a final operation, lug holes are machined and chamfered as a single operation followed by valve hole drilling. For helium leak testing, wheels are taken to bath and test is applied. After these steps, proper wheels are sent to painting shop.

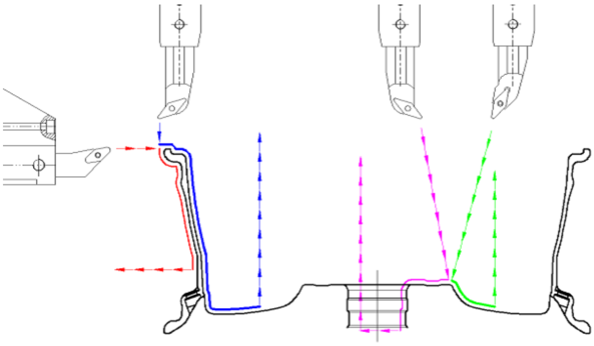


Figure 2.9 Machining process steps

2.2.6 Painting

Wheels surface that comes from the machining process should be cleaned because there might be dirt, dust or oil residues on it. Cleaning is done with using chemical baths not only for decreasing amount of wastage but also preparing the wheel surface to the painting process. Excess water is dried by using compressed air and painting is carried out respectively with powder dye, powder varnish, liquid dye and liquid varnish. Wheels are baked at painting furnace then cooled at room temperature. It is important that, this process may cause over-aging and mechanical properties may decrease. Temperature and time should be adjusted properly, over-aging should be taken into account.

2.2.7 Quality Control

Products are controlled in mechanical and material laboratory as cast, heat treated condition and painted condition. Before the first confirmation for new designs by consumer and after a certain production time in order to ensure that sustainable quality, quality control tests that shown below are carried out.

- Radial fatigue test
- Dual-axis fatigue test
- 13° and 90° Impact test
- ZWARP test
- Chemical composition analysis (Spectrometer)
- Inner flange deformation test
- Micro and macrostructure analysis
- Tensile testing
- Brinell hardness test
- Charpy-impact test
- Salt-bath corrosion test

CHAPTER THREE
HEAT TREATMENT

3.1 Heat Treatment of Aluminium Alloys

Heat treatment can be simply identfied as the operation of heating and cooling in order to change the metallurgical structure, enhance the mechanical properties and decrease the residual stresses of a metal product (Brooks, 2012). The strength of metallic materials can often be adjusted by special heat treatments (Milkereita, Wanderkab, Schickc & Olaf, 2012). Heat treatment of aluminium alloys is considerably different from steel treatment. Mg and Cu is main alloying element which makes aluminium alloy heat treatable and age hardenable.

3.2 T6 Heat Treatment

T6 heat treatment is the most common process to strength age-hardenable aluminium alloys which comprises solution treatment, quenching, and artificial aging (Fig. 3.1).

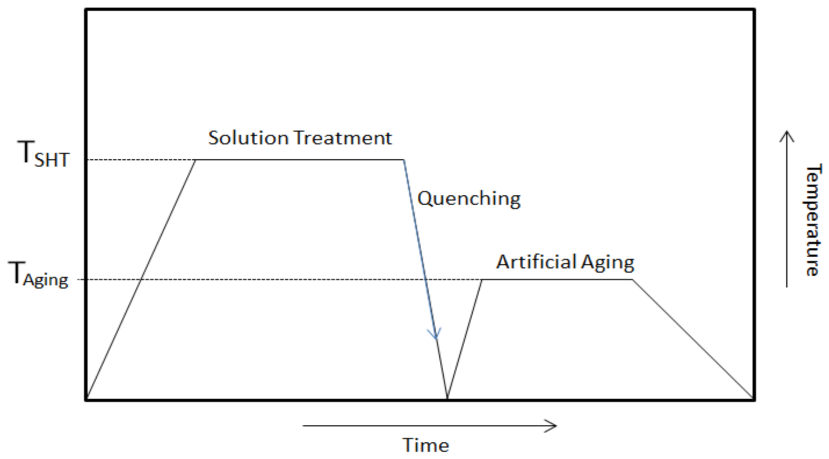


Figure 3.1 T6 heat treatment stages

The properties that achieved with this heat treatment, depend on the combination of the different parameters involved in the process. Fundamentally, aluminium casting part is heated up in solid state to single phase area then quenched in any medium to room temperature and artificially aged at relatively low temperature so as to precipitate Mg_2Si phase in aluminium matrix.

Process temperature and time depend on chemical composition, master alloying, casting part shape, and thickness. Additionally, grain size and secondary dendritic arm space (SDAS) are parameters which assist to designate treatment temperature and time. As illustrated in Fig. 3.2 phase diagrams, continuously cooling precipitation diagrams(CCP) are used in order to determine parameters. Standard treatment conditions have determined and published by ASTM as given below Table 3.1.

ASTM has determined the temperature and time of T6 heat tratment for parmenant mould casting and published in B917/B917M – 12.

Table 3.1 ASTM standard for A356 aluminum alloy T6 heat treatment (ASTM International, 2012)

Solution Treatment			**Artificial Aging**		
Temperature		Time	Temperature		Time
°C	°F		°C	°F	
540	1000	**4- 12 h**	**155**	310	**2-5 h**

Traditionality, these parameters are used by aluminium casting part producers despite the fact that each casting part has different thickness and shape. Both short-insufficient and long-redundant treatment effects production quality and quantity. As indicated above in Table 3.1 ASTM standard does not give information about cooling medium moreover quenching temperature. On account of these reasons, optimal heat treatment circumstances should be evaluated and empirical studies should be done to reach maximum or target properties with minimum energy consumption for A356 aluminium alloy.

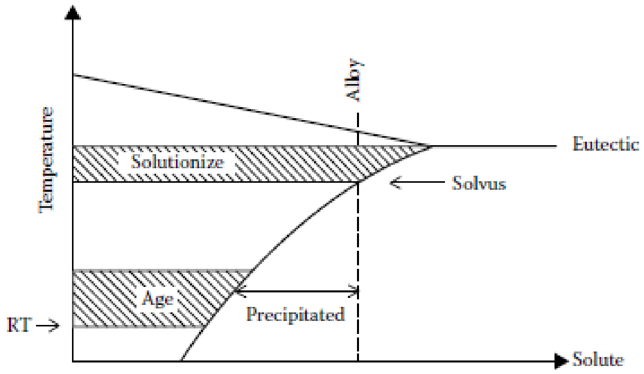


Figure 3.2 Chematic phase diagram for solution treatment and artificially aging zones (Liscic, Tensi, Canale & Totten, 2010)

T6 heat treatment provide two advantegeous effects in A356 alloy. Elongation and the fracture toughness values are increased by the spheroidization and globarization of the rod shape lamellar eutectic silicons (Fig. 3.3). Likewise, yield and ultimate tensile strength is enhanced through the formation of a large quantity of fine β’’ and β’ precipitates in α-Al matrix. The first advantage is provided through the application of only solution treatment and the second advantage is provided through the application of T6 treatment completely (Azadi & Shirazabad, 2012).

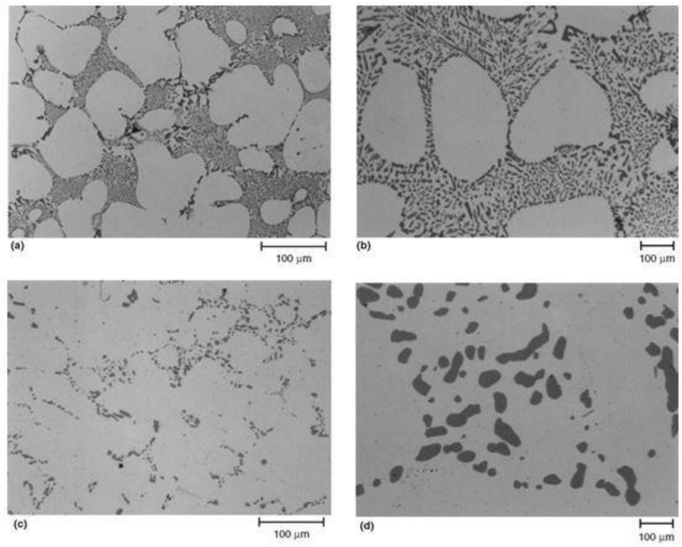


Figure 3.3 Microstructures of AlSi7Mg (Alloy 356.0) Light microscope micrographs; etched with 1% HF (a) As-cast modified, 150X (b) As-cast modified, 750X (c) After heat treatment (T6), 150X (d) After heat treatment (T6), 750X (Warmuzek, 2004)

Alongside the beneficial effects, there are two main problems in the process. One of these problems is high amount of natural gas which is consuming in order to obtain adequately modificated microstructure and the mechanical properties through the solution treatment and aging stages. Excess heat may use because of insufficient calculation or deficient experimental studies. The Second problem which is given in section 3.2.2.2 elaborately, occurs in quenching stage after solution treatment. Casting parts immersed quenching media and cooled down to room temperature. Adjusting the quench rate is important to avoid dimensional changes (distortions), micro cracks and residual stresses.

Prevention of these two main problems are quite momentous in order to save energy, decrease amount of wastage, increase the wheel quality, enhance the mechanical properties and the lifetime.

3.2.1 Solution Treatment

Solution treatment makes the large plate shape Mg_2Si particles in the aluminum alloy dissolved and diffuse throughout the matrix as solid solution. Supersaturated solid solution (SSS) is obtained after this stage. Besides that, during the treatment; homogenization of the casting microstructure occurs and the spheroidization and coarsening of the morphology of the eutectic silicon provides (Kuntongnum, Wisutmethangoon, Plookphol & Wannasin, 2008; Paz, 2003).

The solution treatment temperature is determined according to Pseudo-binary phase diagrams as illustrated in Fig. 3.4 and raised nearly to a eutectic point. Possible treatment temperature interval is marked with red dashed lines.

Besides that, chemical modification level, solidification rate and obtained microstructure (DAS, SDAS, grain size) after the casting process affects the solution treatment temperature and time. Effect of the percentage of the eutectic Si particles and the size of SDAS on solution treatment time for A356 is shown in Fig. 3.5.

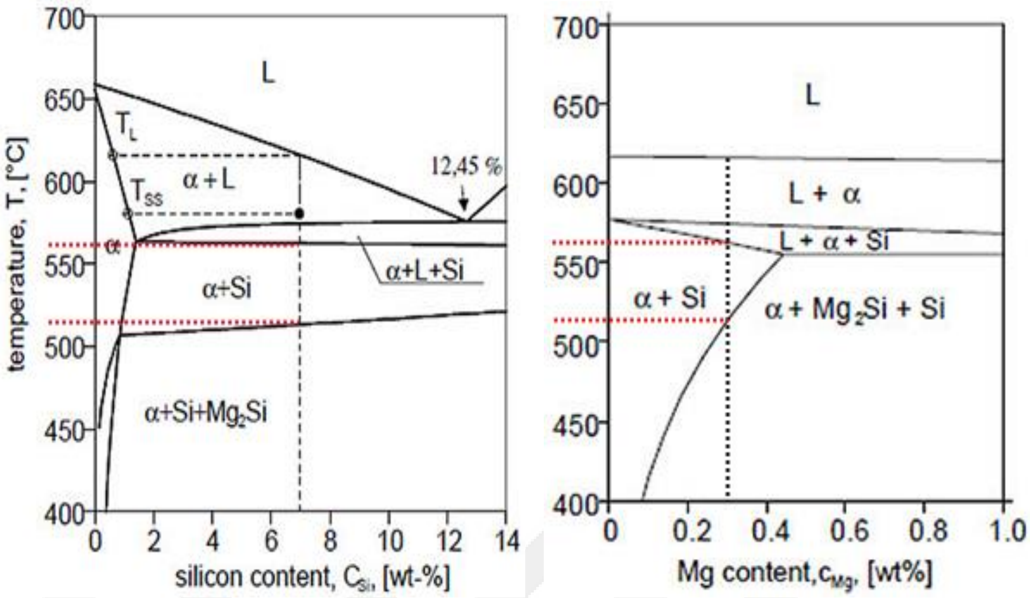


Figure 3.4 Pseudo-binary phase system of AlSiMg0.3 and Pseudo-binary phase system of AlSi7Mg

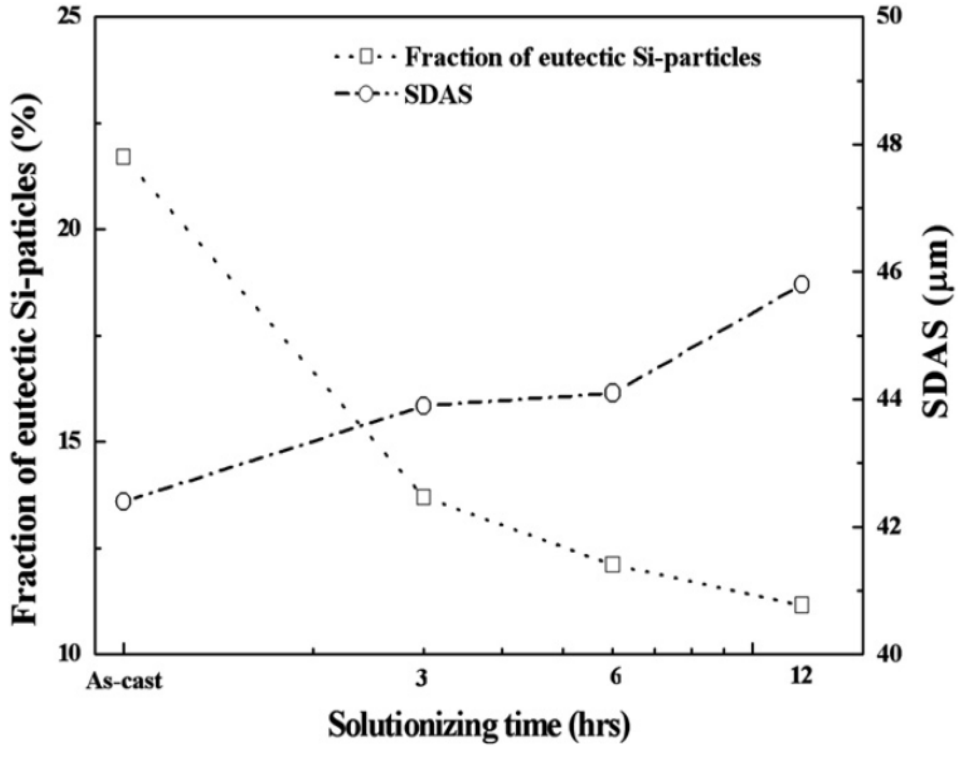


Figure 3.5 Effect of the percentage of the eutectic Si particles and the size of SDAS on solution treatment time (Lee, 2013)

The infuluence of solution treatment time on hardness after artificial aging is shown in some researches. Paz (2003) claim that the parts solution treated for 2 hours at 540°C, exhibit larger hardness than those treated for 4 and 6 hours as shown in Fig. 3.6. The improved of hardness value could only be explained by forming small vacancy clusters. Long, Chen, Liu, Li, & Li (2013) explained in their study that Mg atoms may diffuse to the boundary of porosities during solution treatment and amount of available solutes atoms to form Mg_2Si precipitates in aging phase decreases. Furthermore, as mentioned before diffused atoms leaves vacancies and creates distortion at the beginning of the solution treatment. At longer treatment, distortions (defects in matrix) are reduced and then this defects become less and less active as nuclei to precipitate for aging treatment (Zedan, Samuel, Samuel & Doty, 2010).

On the other hand, Fracasso (2010) mentioned that high temperature of solution treatment improves the hardness properties after ageing treatment.

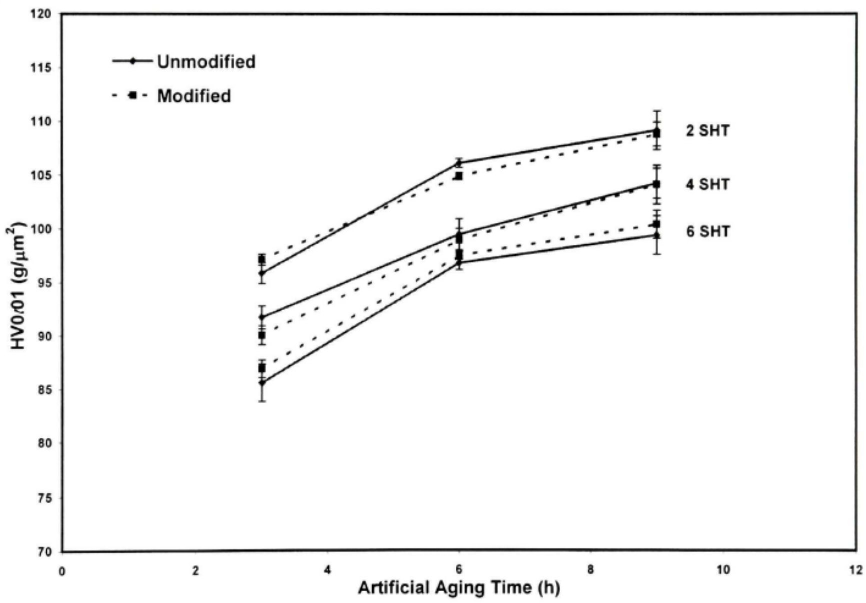


Figure 3.6 Solution treatment effects on hardness after aging process (Paz, 2003)

3.2.2 Quenching

Quenching is a controlled cooling application after solution treatment in variable mediums. Above the critical cooling rate, magnesium stays in the matrix as the supersaturated solid solution(SSS) and ready to precipitate out during the aging process (Kuntongnum et al., 2008). Quenching has basically three stages; Vapor blanket stage (film boiling), nucleate boiling stage, and convective cooling stage (Maniruzzaman, Fontecchio & Sisson, 2009) as shown in Fig. 3.7.

At vapor blanket stage, vapor/film blanket is composed around the surface in few seconds. If different quenching mediums is used such as polymer, polymer film formation is also seen on the surface. Then heat is transferred to the medium in transition stage through this film. According to Möller, Govender & Stumpf (2010), time of this stage determine the mechanical properties and fragmentation of film must be homogeneous.

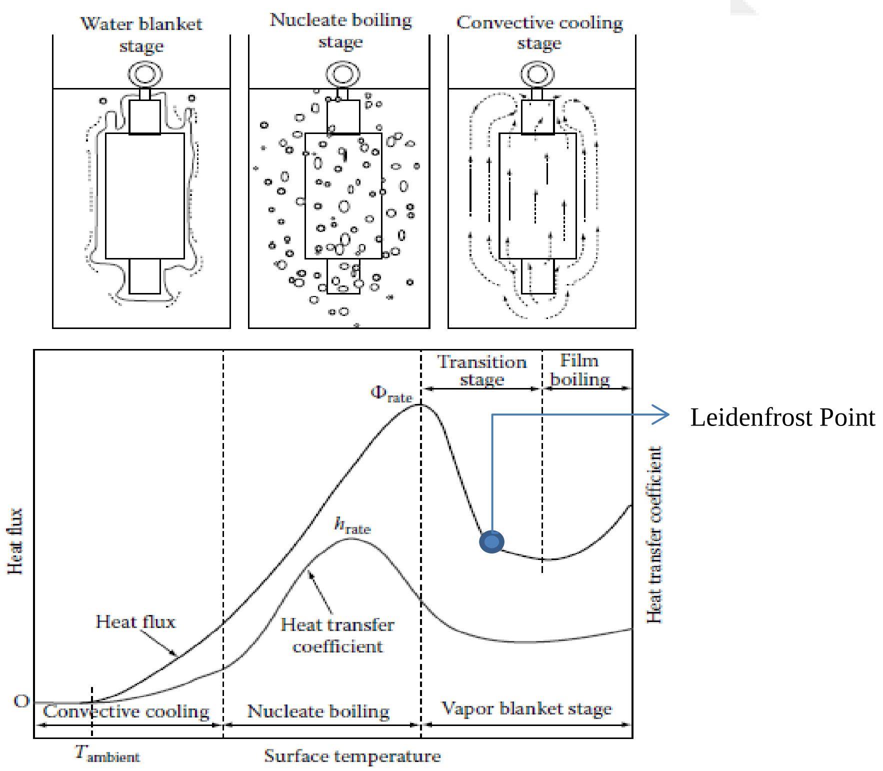


Figure 3.7 Quenching stages (Liscic et al., 2010)

At the second stage (boiling or nucleate boiling), fluid starts to contact directly with the hot surface and boiling regime developes. As illustrated in Fig. 3.8 the heat transfer mechanism includes complex pyhsical phenomenas such as nucleation of bubbles and growth, decomposition from the hot metal surface (Xiaoa, Wang, Jadhavc & Li, 2010).

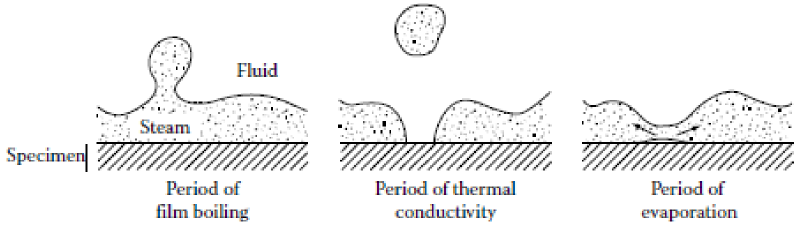


Figure 3.8 Heat transition phases and relation between surface, steam and fluid (Liscic, Tensi & Luty, 1992).

The transition temperature from the first phase to second phase is called the Leidenfrost Point or Temperature (LFP). When the temperature goes down below LFP, part begins contact directly and heat transfer increases. Non-uniform fragmentation causes heterogeneous cooling and distortion risk increases (Alam, 2011). For example, in aluminium quenching, keeping the cooling process in the Leidenfrost film boiling regime longer provides less distortionon the part (Huang & Carey, 2007).

Convection stage starts if the temperature of the metal surface is goes down below the liquid’s boiling point. The transition temperature from the boiling stage to the convective cooling stage is depends on the boiling point of the quenchant. The metal surface is completely wetted by the fluid therefore boiling stops at convection stage and heat transfer occurs by direct contact between surface and liquid as shown in Fig. 3.9. The cooling rates in this stage are very low and heat removal is much slower than in the boiling stage. Quench rate is determined by the rate of convection and the viscosity of the quenching fluid (Sahay, Mohapatra & Totten, 2009).

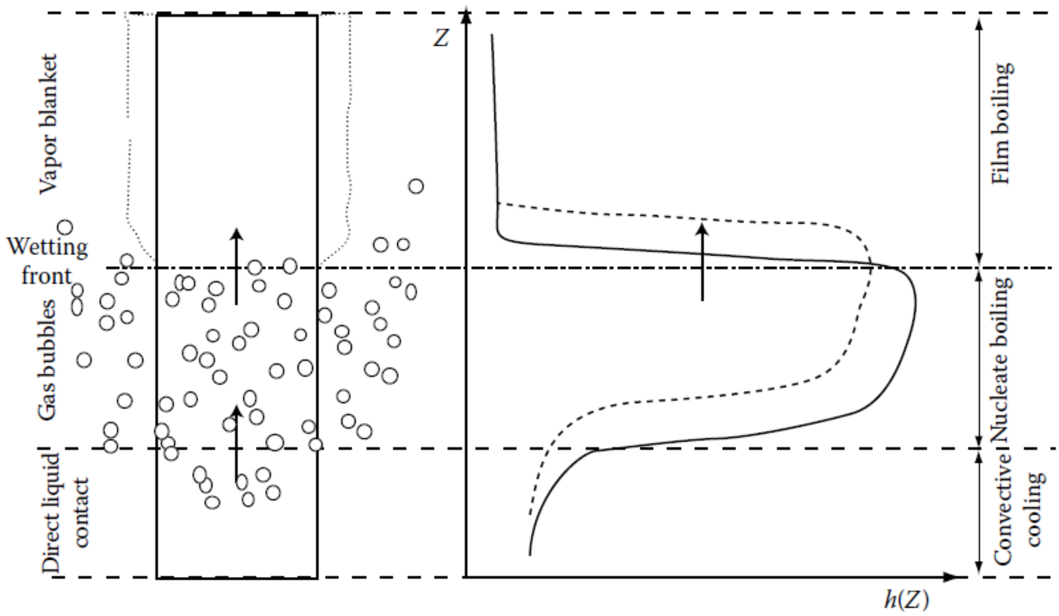


Figure 3.9 Wetting process during the quenching (Liscic et al., 2010)

Quench rate depends on; temperature, viscosity, surface tension, agitation rate, additives (quenchant), vaporation heat of cooling medium, leidenfrost temperature etc. (Huang & Carey, 2007).

Various coofficents are using to identify the quenching power such as;

- Grossmann H-value
- Heat-transfer coefficient W/(K.m^2)
- Quench time
- The cooling curves to determine cooling behaviour.

Firstly, the cooling rate of water quenchant drops sharply with the increasing of the temperature. Changing of cooling rate can be seen in Fig. 3.10 at different (19°C to 65°C) temperatures. However, the cooling intensity of oil increases slightly with the increase of temperature due to the decreasing of the viscosity of the oil.

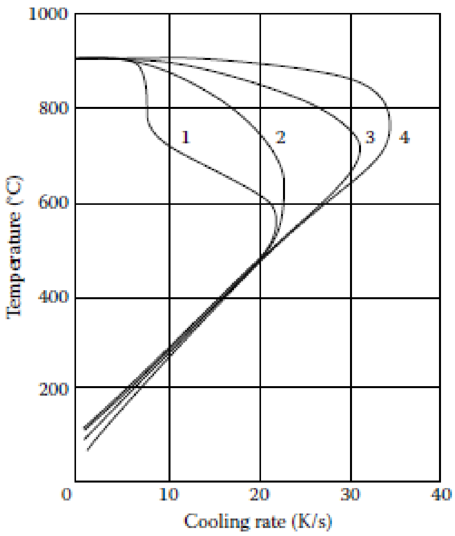


Figure 3.10 Cooling rate of (1) 65°C (2) 53°C (3) 41°C and (4) 19°C water medium (Liscic et al., 2010)

The higher flow rate provides better cooling. The uniformity of fluid flow around component has great influence on distortion.

Thirdly, surface properties are important for quench rate. The smaller the surface tension is, the less the stability of vapor film is, and the stronger the cooling intensity. The cooling rate of rough surface is higher than smooth surface.

Finally, the larger the heat vaporization is, the stronger the cooling intensity. The heat of vaporization of water at 100°C (212 F) is 2260 kJ/kg while the heat of vaporization of mineral oil is about 210 kJ/kg. Therefore, the cooling intensity of water is much more powerful than that of oil. The lower the vapor pressure is, the more difficult the gasification, and therefore the lower the cooling intensity is (Pan, 2002).

The cooling (quenching) rate is important for Al-Si casting alloys. especially in interval between 200 °C and 450 °C. At these temperatures, due to the high level of supersaturation and the diffusion rate, precipitation occurs quite rapidly. Above 450°C supersaturation is low and below 200°C diffusion rate is low for precipitation. (Mohamed & Samuel, 2012). The critical cooling rate for AlSi7Mg0.3 alloy is determined about 60 K/s in literature**.** Quenching with 3 K/s already significantly suppresses precipitation during quenching. Hardness of alloy increases after aging treatment with increasing quenching rate, due to the increasing content of supersaturated solid solution (Milkereit, Fröck, Schick & Kessler, 2014).

3.2.2.1 Cooling Mediums and Quenchants

Water, water solutions with inorganic salt and alkali (brine), oil, aqueous polymer solutions and different types of cooling mediums are used for immerse quenching. Besides that, there are different type of quenching technique such as air forced, spray, sand, fog etc. In this study immerse technique was used.

Water is widely used as traditionally quenching medium. Most important advantages of water are cheaphness and easy usage. When bath temperature increases, vapor phase extents, even tought the desire mechanical properties can not obtain, distortion problems decreases. Genereally, 25°C, 65°C and 80°C waters are commonly used for commercial quenching (Kaufman & Roy, 2004).

Salt addition in water (brine) is preferred for rapid cooling. The salt or borax sticking to component surface can promote the burst of vapor film. Generally is prepared with 10% NaCl or 3% NaOH. Salt crystalls accumulate on surface and then disperse with time so vapor phase is scattered thus quench rate increases. Disadvantage of brine is corrosion risk.

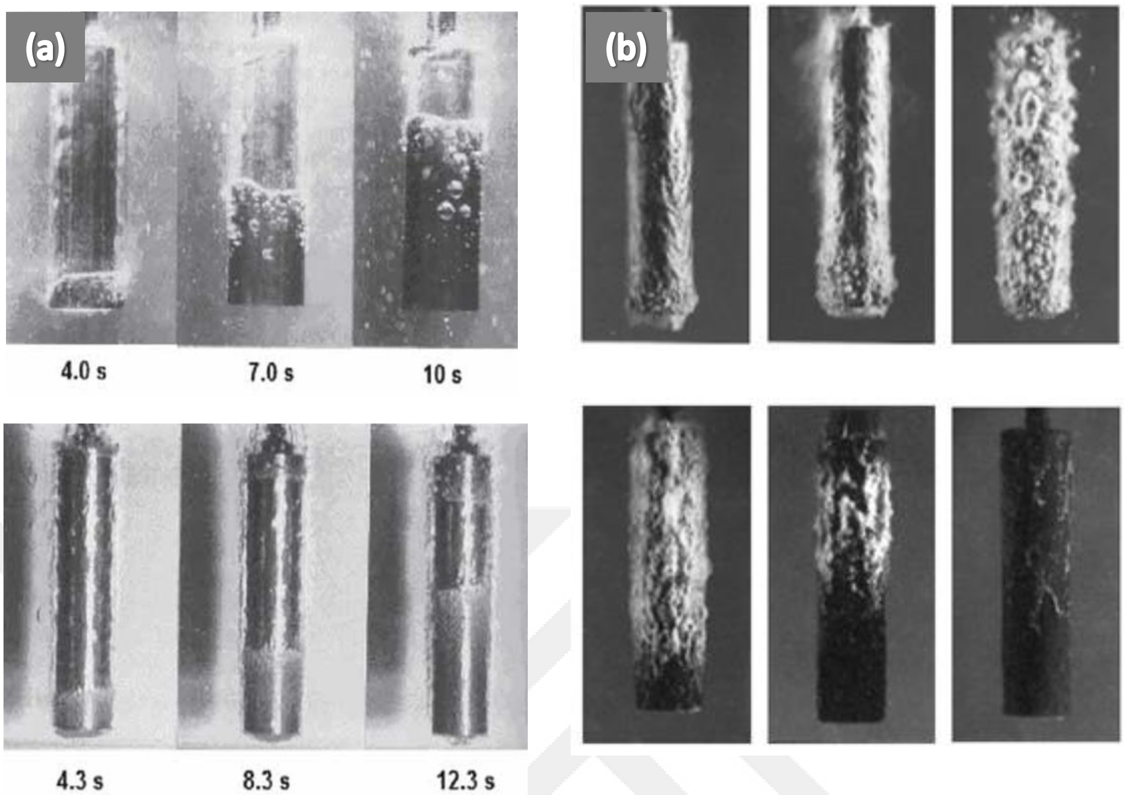


Figure 3.11 Illustration of the surface cooling mechanism of (a) water-quenched (Sahay, Mohapatra & Totten, 2009) and (b) polymer quenchant (Liscic et al., 2010)

Conventional oils are espacially used for preventing distortion and crack formation due to their low quenching rate. Burning and smoke makes the process dangerous while decreasing the mechanical properties.

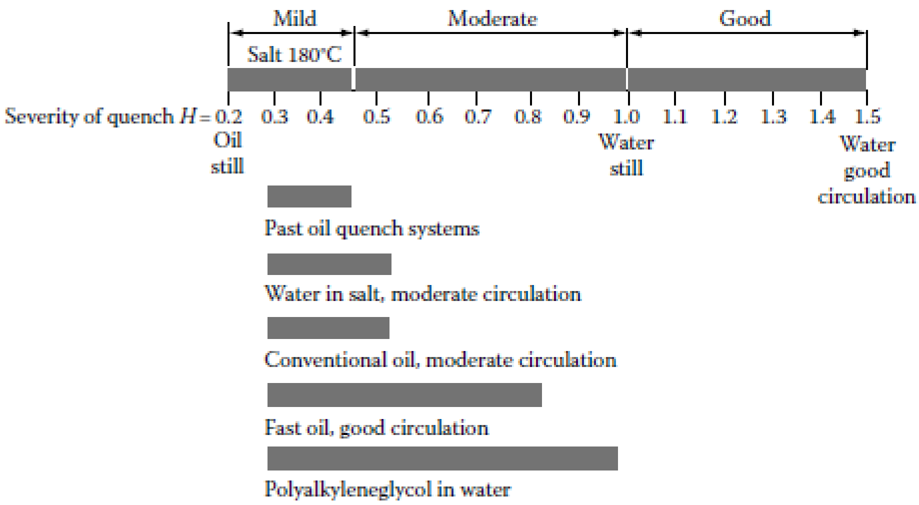


Figure 3.12 Quenching severity coeffi cients *H* of typical quenchants

Polymer based quenchants are consist of organic polymers and water. Polymer is reduced vapor blanket phase and provided homogeneous polymer layer on surface that decreases heat transfer (Croucher, 2008). Constant heat regime is obtained with this layer therefore distortion is controlled. Concentration, temperature and degree of agitation of quenchant affect the vapor blanket stage (Liscic et al., 2010). There is no burning and smoke problem in polymer quenchant (Pan, 2002).

Quenchant types affect the quenching severity considerably, therefore, the selection of cooling medium becomes more of an issue. As seen in Fig. 3.12 there are different types of quenchant with different cooling level from mild (0,2H) to good (1,5H). While past oil quench systems have low quench rate, circulation makes the system more effective as mentioned in section 3.2.2. Oil is accepted as the lowest cooling medium on the other hand brine is accepted as the highest quenchant. Property of polymer-based quenchant depends on polymer type and usage ratio. Generally quenching severity of this type cooling medium between the oil and water, The most commonly used quenchants are PAG, PVA, PSA, PVP, PAC, and PEOX. Although there were many researches about time and temperature heat treatment, few of them focused on quenching stage. So it is necessary to do deep research on cooling medium and cooling behavior.

Fig. 3.13 shows that when polymer concentration increases medium behave like oil so cooling rate decreases. Likewise, shows the difference between hot oil (200°C) and cold quench oil (40°C). Using brine as quenchant shortens the cooling time and also the fastest cooling is provide at 10% brine at 24°C.

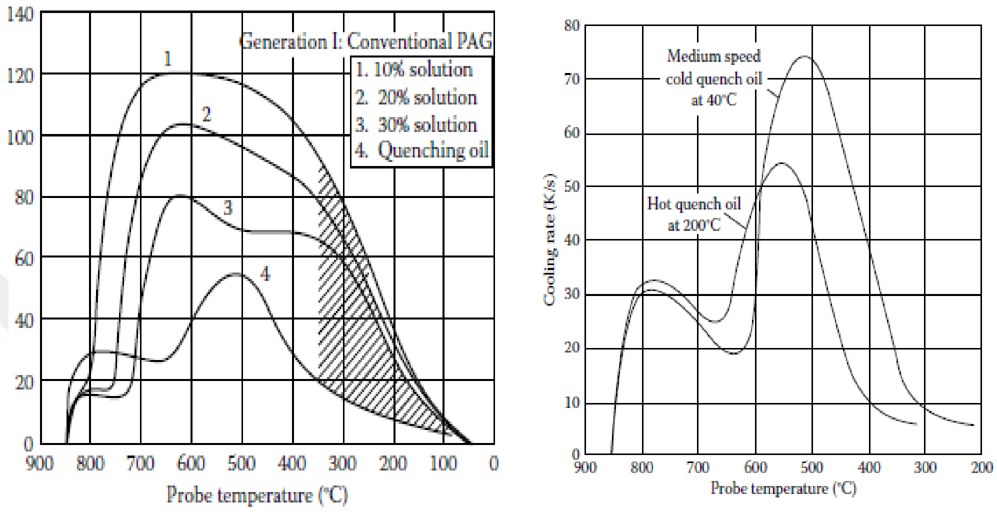


Figure 3.13 Cooling rate comparasion between PAG and oil (Liscic et al., 2010)

As shown in Fig. 3.14, while PVP and PAG have almost the same properties, cooling rate of polyacrylate based quenchant is lower due to its polymer structure. Similarly, variance of cooling rate with different content of oil can be seen in Fig. 3.14. Types of quenchant does not only affect the cooling rate, depends on that, it also affect the residual stresses and behavior of distortion as given in Table 3.2.

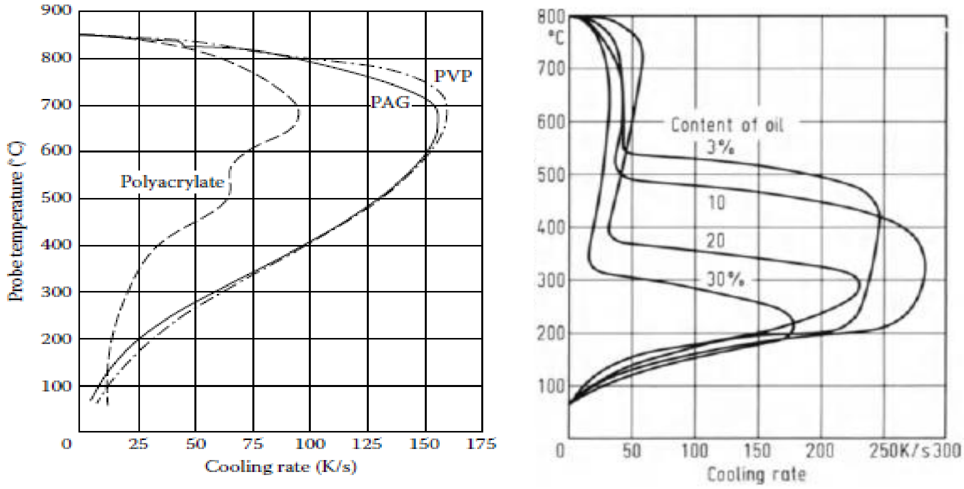


Figure 3.14 Cooling rate comparasion between different polymer quenchants and oil Liscic et al., 2010)

3.2.2.2 Distortion and Residual Stress Problems

Distortion is a expression of the "lack of dimensional stability". Distortion means basically "twist awry or out of shape; to make crooked or deformed." The term distortion is commonly used in heat treating plants interchangeably with the term "warpage" which is defined as "to bend or twist out of shape, especially from a straight or flat form." In this discussion, the generic term "distortion" will be used to mean "part movement that can occur any time" while the term "warpage" will be limited to mean "that distortion which occurs during the heat treating process**."** (Croucher, 2008).

"Cooling must therefore be fast enough to suppress precipitation. On the other hand, cooling should be slow enough to avoid extensive residual stresses and distortions (Rose, Kessler, Hoffmann & Zoch, 2006, p. 116)."

.

Table 3.2 Comparison of several commonly used quenchants in cooling rate and distortion (Pan, 2002)

Quenchant	Cooling Rate	Distortion
10% NaOH water solution		High
10% NaCl water solution		High
10% Na_2CO_3 water solution		High
50% NaOH water solution		High
Water		High
Polymer quenchants		Moderate
25% $NaNO_3$ + 20% KNO_3 + 20% $NaNO_2$ + 35% H_2O	Increase (↑)	Moderate
Accelerated quenching oil		Low
Alkaline salt bath		Very Low
Nitrate bath		Very Low
Ordinary mechanical oil		Very Low
Air		Very Low

"Faster rates of quenching retain a higher vacancy concentration enabling higher mobility of the elements in the primary Al phase during aging. An optimum rate of quenching is necessary to maximize retained vacancy concentration and minimize part distortion after quenching. A slow rate of quenching would reduce residual stresses and distortion in the components, however, it causes detrimental effects such as precipitation during quenching, localized over-ageing, reduction in grain boundaries, increase tendencies for corrosion and result in a reduced response to ageing treatment (Mohamed & Samuel, 2012, p. 62)."

3.2.3 Artificial Aging

Age-hardening is one of the most important strengthening methods aluminum alloys. Through the aging coherent secondary phases precipitate into soft matrix and causes the formation of dislocations. Aging time and temperature may changes the mechanical properties significantly. Tensile properties can be enchanced and residual stresses that occur in quenching can be reduced. In Fig. 3.15 mechanism of the T6 heat treatment and microstructural changes are shown schematically. The precipitation can also occurs at room temperature it is called natural aging. However, in industry, artificial aging at temperatures interval from 10°C to 300°C is applied to accelarete the process (Mohamed & Samuel, 2012).

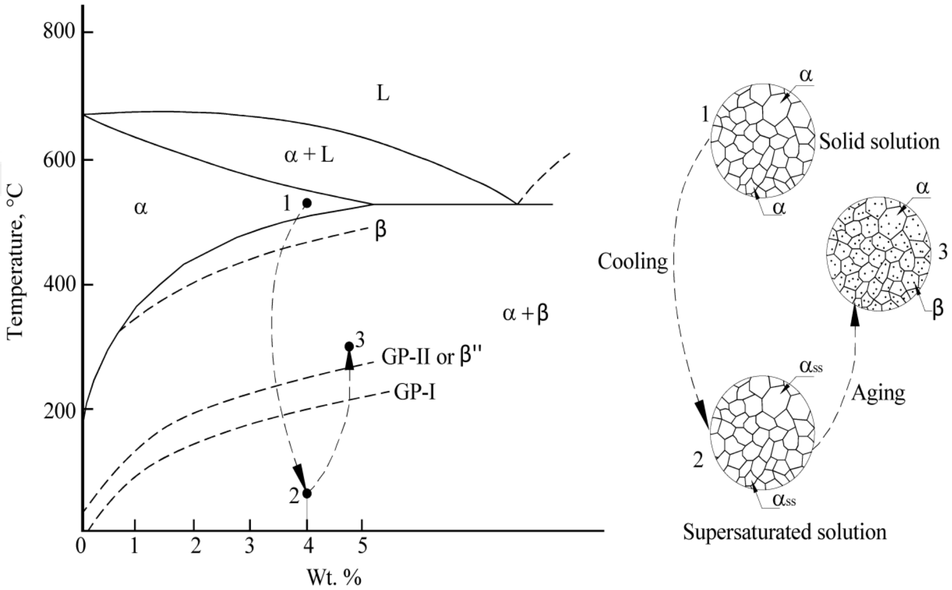


Figure 3.15 Illustration of the mechanism of T6 heat treatment and precipitation hardening

The precipitation series for Al-Si-Mg alloys can be shortened as follows;

$$\alpha\,(SSS) \longrightarrow GP_{zones} \longrightarrow \beta'' \longrightarrow \beta' \longrightarrow \beta$$

However, Zander & Sandström, (2008) define the series as;

SSS ⟶ clusters of Mg and Si atoms ⟶ spherical GP_{zones}

needle shaped coherent β''(Mg_2Si) ⟶ rod like semi-coherent β'($Mg_{1.7}Si$)

semi-coherent β plates ⟶ non-coherent β-plates (Mg_2Si)

Ogris (2002) was found that the following series;

α (SSS) ⟶ $GPI_{spherical}$ ⟶ GPII $β''_{needle}$ ⟶ $β'_{rod}$ ⟶ β(Mg_2Si) $_{plate}$

"where α (SSS) is a supersaturated solid solution, and GP zones are coherent Guiner-Preston zones (GP zones). The β'' corresponds to precipitate associated with the peak-aged condition; they are needle shaped and aligned along [100]Al directions of the matrix. The seni-coherent β' precipitate forms after coherent β'' precipitates in the aging sequence that are rod shaped and are aligned along [100]Al. The equilibrium phase β forms as incoherent platelets lying in [100]Al planes. (Ceschini, Morri & Morri, 2013)."

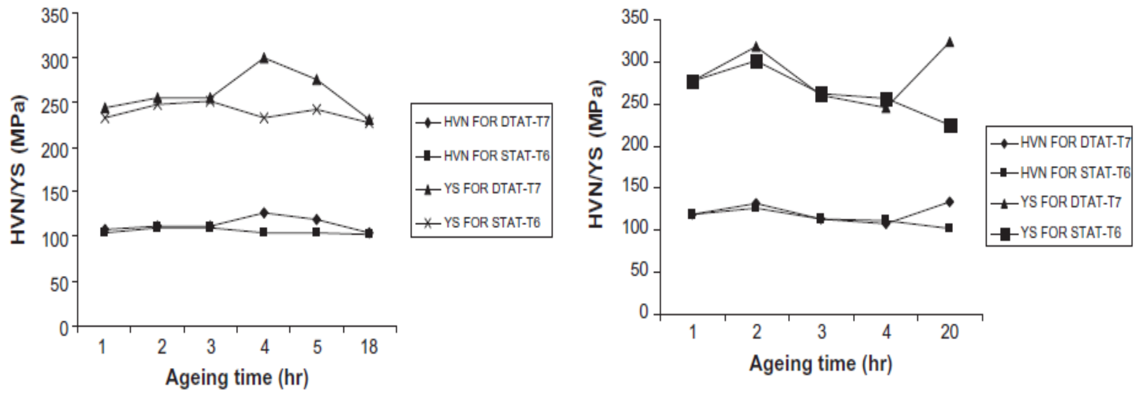


Figure 3.16 Hardness and Yield Strength curve for A356.0 aluinium alloy with DTAT and STAT with 150°C and 180 °C (Abdulwahab et al., 2011)

According to aging treatment temperature and time, obtained hardness, yield and tensile strength alternates. As shown in Fig. 3.16 at high temperature(180°C), YS value is higher at 2 hours comparing to low temperaure(150°C) and maximum strength is obtained in shorter time however elongation is sacrificed. Long-aging process at high temperature causes the grain growth and β’’ particles transforms to β plate shape therefore mechanical properties (strength, hardness) decrease as seen in Fig. 3.17 and 3.18. It is called over-aging.

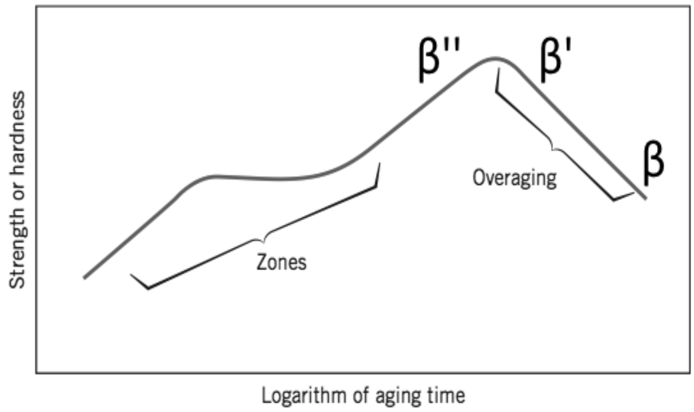


Figure 3.17 GP Zones formation and overaging

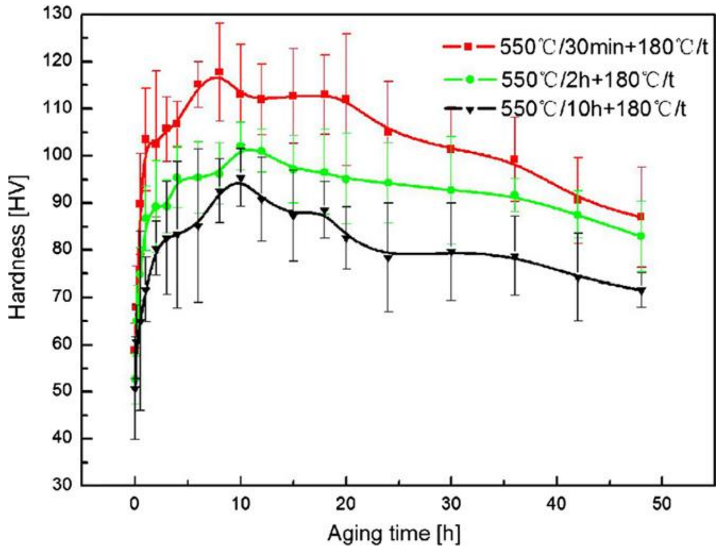


Figure 3.18 Hardness curves at different aging temperature and overaging (Long et al., 2013)

If strength is needed, higher artificial aging temperatures and shorter times should be used to obtain the desired strength and hardness. However, at high temperature, overaging can be seen and hardness values decreases sharply because of the increasing precipitation size as shown in Fig. 3.19 (Paz, 2003).

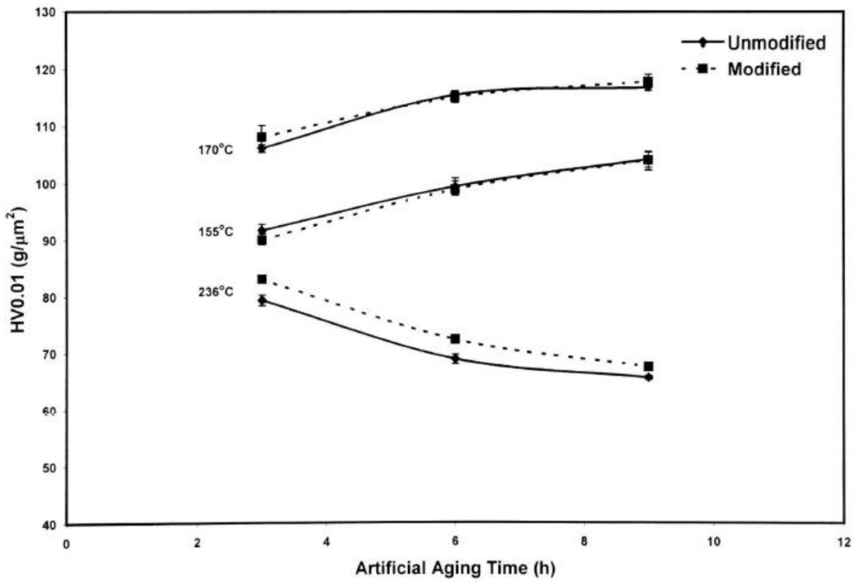


Figure 3.19 Modified and unmodified A356 alloy hardness comparison at different aging parameters (Paz, 2003)

In addition, quench rate also affect the aging results. TEM characterizations on A356 alloy were indicated that after quench in water at 25°C, the α-Al matrix includes in large quantaties needle-shaped and fine β″(Mg_2Si) precipitates. Characterization analysis were showed that the size of the β″(Mg_2Si) phases are roughly 3 to 4 nm in diameter and 10 to 20 in length. Contraraly, at 60°C water medium, researchers observed that the volume of the precipitates decreases and the size of the β″(Mg_2Si) precipitate phases grows. In addition, a momentous quantity of fine Si precipitates resulting from precipitation of excess Si can be seen in the microstructure (Mohamed & Samuel, 2012).

3.3 Recent Researches on T6 Heat Treatment

Zhang, Zheng & StJohn (2002) researched that the short solution treatment effects on tensile and impact properties of stronsium modified A356 alloy. Experiments carried out in this study at 540°C and 550°C for 10, 30 and 60 min. They found that a solution treatment at 540°C or 550°C for only 10 minutes is sufficient for the α-Al phase to homogenise and attain the maximum level of Mg and Si solubility. They also claimed that a solution treatment for 30 minitus provides spheroidization and coarsening of the eutectic silicons thus ductility and impact resistance improves.

Rometsch & Schaffer (2002) researched that the relationship between hardness(HB) and yield strength (YS) for different aluminium alloys. In this study, aging treatments carried out at 150°C to 250°C for variety times following by solution tratment at 540°C for 75 min. After the detailed study, they found the formulas that can be expressed reletionship between HB and YS;

$$YS=2.95HB[0.065]n \tag{3.1}$$

Where *n* is the strain hardening exponent calculated from the total true strain.

Paz (2003) found that well-modified A356 alloy shows very similar hardness values solution treated for 2, 4 and 6 hours after artificial aging at 155°C. He showed that 2 hours are enough to obtain similar properties. The artificial aging temperature and time should be chosen depending on the desired properties. If strength is needed, higher artificial aging temperatures and shorter times should be used to obtain the desired strength (236°C-30min). At low temperatures elongation values increases (155°C-6h). And also he indicated that higher temperatures provide more hardness only if stay in non-over aging temperatures.

Shabestari & Shahri (2004) researched the effects of cooling rate, Sr and Sb addition in alloy, the heat treatment and their relationship with microstructure and mechanical properties of A356 alloy in their study. They said that heat treatment is more effective on tensile properties than solidification rate and modification.

Kuntongnum et al. (2008) carried out T6 as follows: solution treatment at 520°C and 540°C for 4 hour before quenching and artificially aging at 135°C, 165°C and 195°C for 4, 8,12 and 16 hr. They found that as the aging temperature increase the optimum aging time is shorter and the maximum value of hardness decrease. Likewise Paz (2003) and Fracasso (2010) found the same results in his study.

Long time treatment (535°C-4 h + aging at 150°C- 15h) and short time treatment (550°C-2h + aging at 170°C-2h) were compared in Ji-hua et al. (2011) study and they found that **"**the solution at 535 °C for 4 h and the solution at 550°C for 2 h can reach full spheroidization of Si particle, over saturation of Si and Mg in α(Al) matrix. The heat treatments of T6 and ST produce almost the same microstructure of A356 alloy."

Abdulwahab et al. (2011) used modified A356 alloy with 0.01 Sodium(Na) in their study, single thermal aging treatment (STAT) and double thermal aging treatment (DTAT) was applied. Solution treatment carried out at 540°C for 1 hour and quenched in water followed by an artificial ageing temperatures at 150°C, 180°C, 210°C and ageing time of 1–5, 18, 20 h. Peak hardness value was obtained at 180°C for 20 hours aging (134.9 HVN) and they claimed that the most economic aging treatment is DTAT at 180°C for 2 h.

In their study Zhu et al. (2012) performed SHT at 535°C for 8 hours followed by quenching rapidly in water at 25°C, and then artificial aging applied at 160°C for 6 h followed by air cooling. They mentioned that "the YS-UTS and elongation of the T6-treated A356 alloys with and without modification are improved due to the speroidization of eutectic silicon particles and Mg_2Si precipitation hardening."

Long et al. (2013) researched the effect of solution treatment on the age hardening of an A356 alloy. They showed that prolonged solution treatment has a detrimental influence on age hardening due to the porosity in the cast A356 alloy. “The diffusion of Mg atoms toward porosities during solution treatment may lead to a significant reduction of the Mg solutes available to form Mg–Si-containing strengthening precipitates in the Al-matrix upon aging treatment. The Mg enrichment around porosity becomes more severe with the increase of solution treatment time.”

Lee (2013) investigated the T6 heat treatment conditions for low pressure die casting in order to optimize fatigue properties therefore the solution treatment was carried out at 540°C for 3,6 or 12 hours and the artificial aging was applied at 160°C for 4, 8 hours. He claim that “a prolonged solution treatment has a negative influence on the age hardening due to the existence of porosity in the cast A356 alloy similar with Long, et al. (2013).” Also one was mentioned that “apart from the spheroidization of the Si particles during solution treatment, thermally activated Mg atoms can diffuse out from the Al-matrix and enrich near the porosity pre-existed in the alloy. The Mg enrichment around porosity becomes more severe with the increase of solution treatment time." Researcher suggested 540°C for 6 hours and 160°C for 16 hours heat treatment for increasing the number of cycles to failure for A356 alloy at alternating stress amplitudes of less than 200 MPa.

In order to determine the effect of heat treatment on impact strength Akhil, SanjiviArul & Sellamuthu (2014) used different size cast components as 20-40-60-80 mm. Parts were heat treated at 537°C for 12 hours followed by a temperature of 155º for 5 hours. They indicated that “the as-cast condition impact strength increases with reduction in section size due to grain refinement in smaller section size caused by fast cooling rate”.

In an other study, A356 and A357 alloys were compared. Solution treatment experiments were carried out at 520°C, 530°C and 540°C for 5 minutes to 5 hours and quenched in water then artificially aged at 160°C, 170°C and 180°C. They concluded that solution heat treatment of A356 and A357 alloys for 15 minutes and at 540°C, quenched at 25°C and aged for 3h at 180°C, has shown similar hardness results (Menargues, Martín, Baile, & Picas, 2015).

Tarkun (2015) has reported the mechanical properties and microstructures of A356 alloy wheels which is produced in CMS Jant ve Makine Sanayi A.Ş. In thesis, as cast and T6 treated mechanical values are given as following YS 126-201 MPa, UTS 209-267 and elongation is given 5.7 - 4.3.

There are a quite few research studies on solution treatment and artificial aging. However, studies on quencing are rare to find in literature espacially aluminium alloys. Researcher focused on polymer solutions for steel heat treatment and literature about polymer quenchants is given below.

Kakhki et al. (2009) used PAG type polymer, oil and water for comparing hardness after quenching and claim that, with using polymer solution desired properties can be obtained together reducing distortion risk. On the other hand, according to Liscic et al. (2010) above 74°C PAG loses its effectiveness into water, PVP shows similar properties with oil because of its density and with adding 10% PVP can be reach to desired mechanical properties without cracking (Pan, 2002). 10% ACR usage as quenchant equals to oil medium so usage ratio above the 10% decreases the mechanical properties. Besides that, best mechanical properties obtain with 16% PAG concentration, medium properties obtain with 22-28% PAG and to achive optimum distortion control 32-40% PAG should add into water solution (Croucher, 2008).

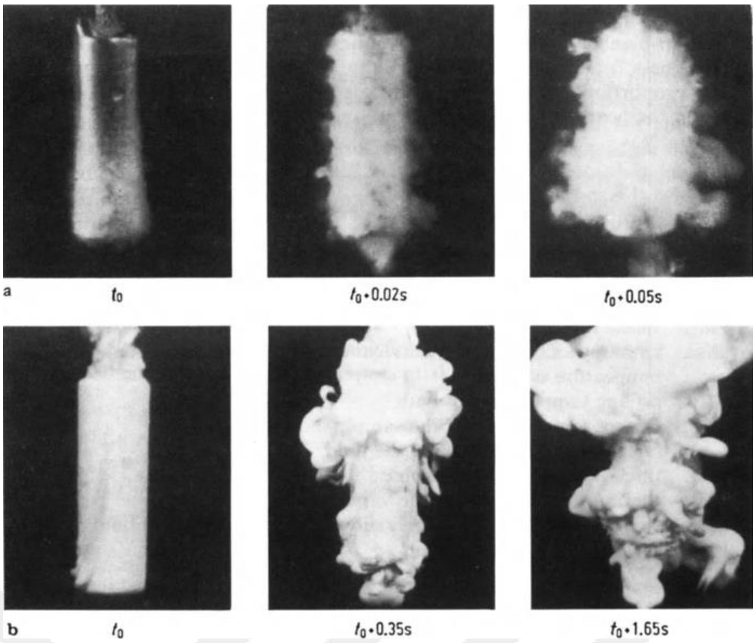


Figure 3.20 Transition stage with (a) Polymer quenchant (b) Polymer quenchant with surfactant additive (Liscic et al., 2010)

Liscic et al. (2010) mentioned that some chemical additives into polymer-water solution may increase the fragmentation time and accelerate the transition to second stage of cooling. Explotion effect or ligth of foaming can be seen in Fig. 3.20. Cooling graphs of different polymers are also shown in section 3.2.2.1.

It appears from the aforementioned investigations that most attention has been paid to quenching of steel parts. Although polymers have been widely used on the new quenching technologies, there are microbiological problems of their uses in industry. Quench tanks must be cleaned frequently and circulation must be obtained. Otherwise, it creates a bad smell in the quench tank and causes environmental risks and human healthy.

Due to this problem, the surfactant which similar to polymer chemical structure can be used as quenching medium instead of polymer quenchants.

3.4 Surfactants

"Surfactants are the similar chemicals with polymers which are added to the water for decreasing its surface tension and enhancing the wettability of fluid so that it can have a better contact with the solid surface (Alam, 2011)." The term surfactant is a blend of 'surface active agent'. There are three main type of surfeactant as following; ionic, non-ionic, anionic.

In earlier investigations on surfactants are generally gives information about the surface tension that effect the boiling phase but recent studies show that additives also effect the pool boiling similarly without do not change the surface tension. Besides, some researchers used the definition of dynamic surface tension instead of surface tension. Furthermore, Shah & Darby (1973) studied the heat transfer coefficients for various surfactant mediums. They found that improvement of heat transfer with increasing surfactant concentration may not caused by surface tension but by foaming. These results were explained with the phenomenon of light in their study. Light of foaming can be seen at Fig. 3.20.

Qiau & Chandra (1998) researched the cooling behavior of hot copper with SDS (Sodium Dodecyle Sulphate) quenchant with a concentration of 100 ppm. They analyzed the influence of addition of surfactant on boiling curve under the surface temperature of 240 °C and they found that addition of surfactant enhances the nucleate boiling heat flux by up to 300 %. Nevertheless, SDS addition reduced the transition boiling. Alam (2011) used a different surfactants known as Ethoxylated ester with concentration of 50-100-200-500 ppm.

According to previous studies in literature, it seems that addition of a small amount of surfactant into cooling medium enhance the heat transfer coefficients and change the quenching behavior extremely.

CHAPTER FOUR
EXPERIMENTAL STUDIES

4.1 Study of Current Situation

Understanding the current situation is the basis of this study. For this reason, firstly, the heat treatment of A356 aluminum wheel parts produced by low pressure casting was studied. Process steps were carried out under standard heat treatment conditions (temperature and time) applied at CMS Jant ve Makina Sanayi A.Ş. To study the current situation, solution treatment was performed at 540°C for 240 min. before quenching at 80°C. After that, artificial aging was performed at 150°C for 180 min. Finally, the painting was applied to the wheels at 180°C for 30 min. and parts were cooled at room temperature. In this study, different heat treatment procedures were also applied to understand the effect of each step of heat treatment. These procedures were given below;

- Only solution treatment
- Solution treatment + painting
- Solution treatment + artificially aging (T6)
- Solution treatment + artificially aging + painting (T6 + painting)
- Only artificially aging (T5)
- Only artificially aging (T5) + painting

4.2 Design of Experiment (DOE) and Heat Treatment Experiments

In order to evaluate the results of the experimental studies systematically, an experimental design (DOE) was made based on the temperature and time parameters applied in the literature studies. The experimental studies consist of three steps; solution treatment tests, solution treatment and aging optimization tests and quenching tests. For solution treatments, general full factorial 2 factor 5 level design was used and 25 experiments were planned. For solution and aging optimization tests, 4 factor 3 level 1/2 factorial design was used and 19 experiments were planned. For the last step, 4 experiments were performed in four different quenching media. Heat treatments were performed in laboratory type Protherm furnace. Heat treatment parameters were given in Table 4.1 and 4.2.

According to Minitab results; main effect plots and interaction plots were drawn. In order to obtain prediction and optimization plots, multiple regression model was used.

- A main effect is present when different levels of a categorical variable affect the response differently. For a variable with two levels, one level can increase the mean compared to the other level. This difference is a main effect.
- Interaction plots are used in order to assess two-way interactions. Lines is evaluated to understand how interactions affect the response. If the lines are parallel, there is no interaction.
- Regression investigates the relationship between a response (Y) and predictor(s) (X). Multiple regression analyze is an examination of the linear relationships between one continuous response and two or more predictors.

Solution treatment experiments were carried out respectively at 510°C, 520°C, 530°C, 540°C, 550°C for 80 min., 160 min., 200 min., 240 min., 280 min. followed by quenching immediately in water at 80°C for 120 second. The effect of these parameters on mechanical properties was evaluated in Minitab program. Microstructures of the samples were also investigated.

Optimization experiments were carried out respectively at 480°C, 510°C, 540°C for 80 min., 160 min., 240 min. followed by quenching immediately in water at 80°C and artificially aged at 120°C, 150°C, 180°C for 60 min, 120 min, 180 min..

In the last step of experimental studies, the effect of quenching medium (Table 4.3) on mechanical properties was studied. Different type of surfactants (Triton X-100, Tween 20, SDS) were used at same amount and other parameters were also kept constant.

Table 4.1 Design of solution treatment experiments

Experiment No	Solution Treatment Temperature (°C)	Solution Treatment Time (min.)
1	510	80
2		160
3		200
4		240
5		280
6	520	80
7		160
8		200
9		240
10		280
11	530	80
12		160
13		200
14		240
15		280
16	540	80
17		160
18		200
19		240
20		280
21	550	80
22		160
23		200
24		240
25		280

Table 4.2 Design of optimization experiments

Experimen t No	Std Order	Run Order	Center Pt	Blocks	Solution Treatment Temp. (°C)	Solution Treatment Time (min.)	Quenhing Temp. (°C)	Artificially Aging Temp. (°C)	Artificiall y Aging Time (min.)
26	15	1	1	1	480	240	80	180	180
27	6	2	1	1	540	80	80	180	60
28	5	3	1	1	480	80	80	180	60
29	13	4	1	1	480	80	80	180	180
30	12	5	1	1	540	240	80	120	180
31	4	6	1	1	540	240	80	120	60
32	17	7	0	1	510	160	80	150	120
33	8	8	1	1	540	240	80	180	60
34	1	9	1	1	480	80	80	120	60
35	16	10	1	1	540	240	80	180	180
36	3	11	1	1	480	240	80	120	60
37	14	12	1	1	540	80	80	180	180
38	18	13	0	1	510	160	80	150	120
39	19	14	0	1	510	160	80	150	120
40	10	15	1	1	540	80	80	120	180
41	2	16	1	1	540	80	80	120	60
42	9	17	1	1	480	80	80	120	180
43	7	18	1	1	480	240	80	180	60
44	11	19	1	1	480	240	80	120	180

Table 4.3 Design of quenching experiments

Experiment No	Quenchant	Amount of Addition
45	Water	-
46	SDS	1000 ppm
47	Triton X-100	1000 ppm
48	Tween 20	1000 ppm

In order to determine cooling behavior of the parts at different quenching mediums, termocuples were attached into the wheels as shown in Fig. 4.1. Datas collected by using datalogger and computer. Maximum cooling rate, Leidenfrost points and mechanical properties were examined.

Figure 4.1 Termocupl placed into the wheel (Personal archive, 2019)

4.3 Mechanical Testing

In order to determine the mechanical properties, tensile testing was applied and hardness measurement was performed to the samples. Samples taken for microstructure examination and mechanical tests were taken from the regions indicated in the schematic cross-sectional view of the wheel given in Fig. 4.2.

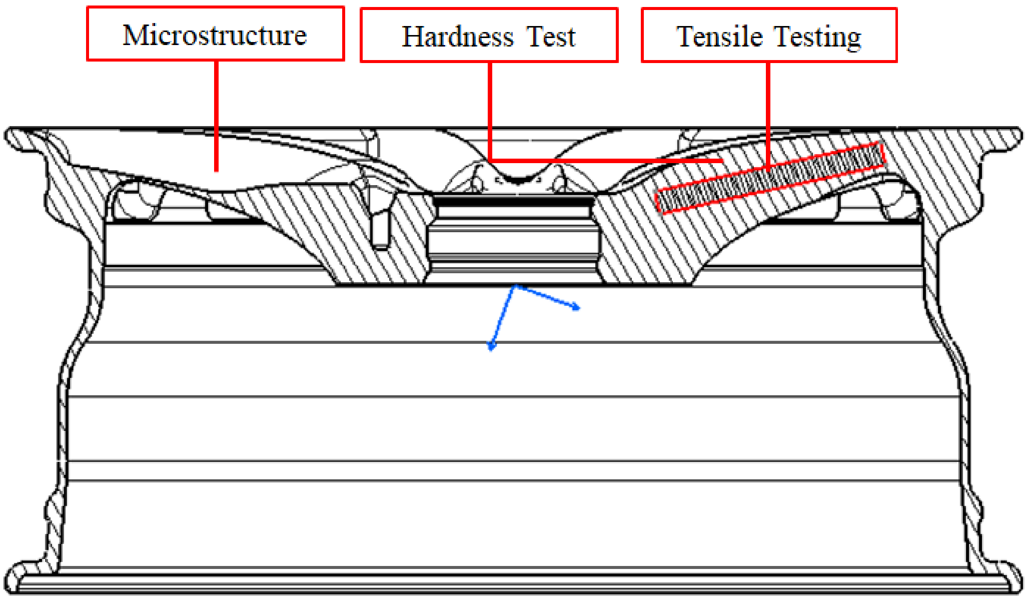


Figure 4.2 Area number of obtained testing specimen

4.3.1 Tensile Testing

Tensile test specimens were prepared as shown in Fig. 4.3 with dimension; 5.64 mm d_o, 28 mm L_o, 24.98 mm^2 So and test was performed according to TS EN ISO 6892-1 standard with Zwick Roell Z100 tensile testing device with 5 N/mm^2 pre-load, 5 N/mm^2 test speed and 100 kN Load-cell.

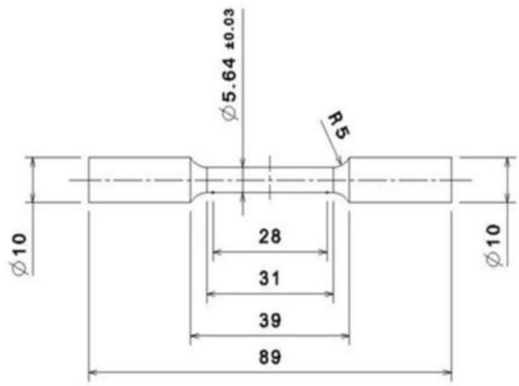


Figure 4.3 Tensile test specimen dimensions

Tensile testing results are shown as elastic module(E), yield strength (Rp0,2), ultimate tensile strength (Rm) and elongation(A28). Four tests was applied to each experiments and average was calculated.

4.3.2 Hardness Test

Heat treatment is directly related with hardness due to the homogenation of matrix, Mg and Si dissolution in alloy and their precipitation behavior. In this study, Brinell measurement was carried out according to TS EN ISO 6506-1 standard with Innovatest Nemesis 9000 machine. Specimens were prepared by using milling machine and grinding with 180 grit. Three tests was applied to each experiments and average was calculated.

4.4 Microstructure Analysis

After samples were cut from the wheel, samples were grinded with 180, 800, 1000, 1200 grit sand papers. After grinding, samples were polished with diamond paste. In order to observe the Microstructure, the samples were etched using 3% HF and the surface cleaned with alcohol. The microstructures were investigated using Nikon Epiphot 200 in different magnifications.

CHAPTER FIVE
EXPERIMENTAL RESULTS AND DISCUSSION

5.1 Tensile Testing Results

5.1.1 Tensile Testing Results of Current Situation

In order to understand the current situation and determine the effect of different heat treatment stages on mechanical properties, tensile testing was applied to low pressure die cast A356 aluminium wheels. Yield strength (YS), ultimate tensile strength (UTS), and elongation (ε%) results after different heat treatment procedures are shown in Fig. 5.1 and 5.2 in detail.

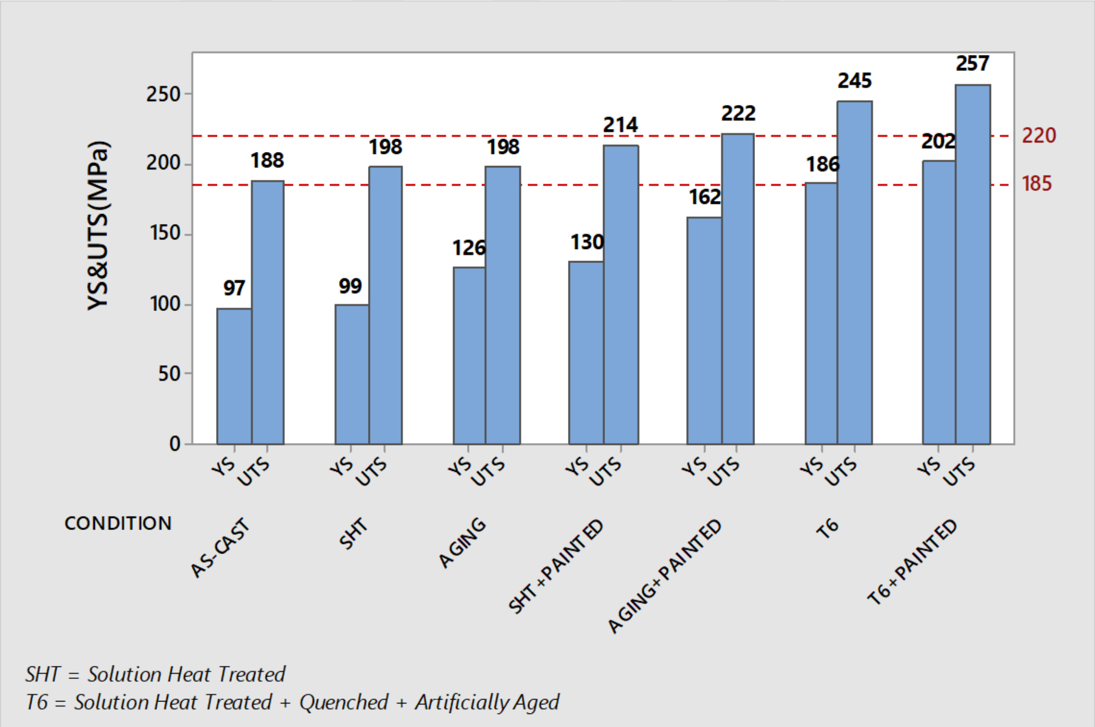


Figure 5.1 YS (Rp) and UTS (Rm) results after different heat treatment procedures

Original Equipment Manufacturers (OEM) require the specific yield-ultimate tensile strength (YS-UTS), elongation (ε%) and hardness (HB) values. The desired mechanical properties must be achieved in the final product. These desired values are indicated by red dashed lines on the graphs.

There is no significant change in YS and UTS values after solution treatment. However, as a result of the change in eutectic silicon morphology from acicular to fibrous shape, elongation value increased from 6 % to 10 %. This results are similar with Azadi & Shirazabad (2012) study. After artificial aging applied on the as-cast sample, YS value increased from 97 MPa to 126 MPa with a significant change 26 %.

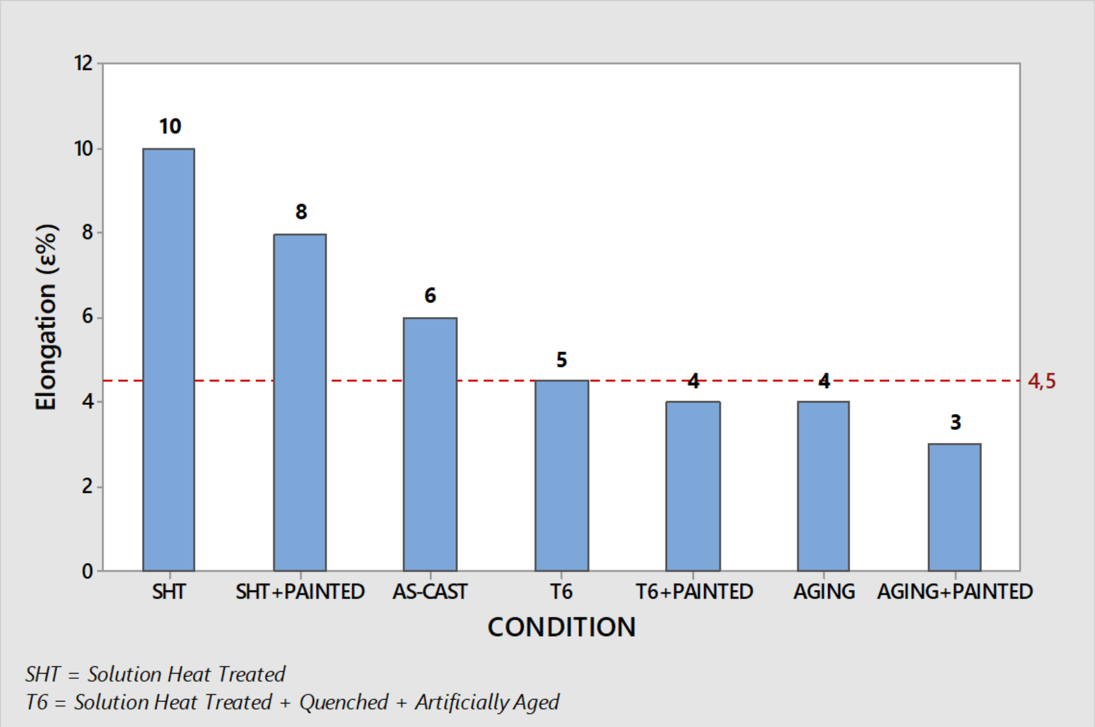


Figure 5.2 ε% (A28) results results after different heat treatment procedures

Whereas T5 (only aging) enhanced the YS-UTS, elongation values decreased from 6.0 % to 4.2 %. The results show that artificial aging temperature is not enough high to change the eutectic silicon morphology and homogenization of the microstructure.

According to the results, painting process applied after solution treatment and artificial aging is quite effective on YS and UTS and increased the YS and UTS values approximately 30 %. The lowest elongation value was obtained after aging and painting. Because of the process condition (temperature and time) are similar, aging and painting process together is like double aging and has negative effect on elongation.

Compared to as-cast samples, after T6 heat treatment YS and UTS values of the samples increased by 93 % and 30 %, respectively. However, elongation decreased from 6.0 % to 4.4 %. The highest YS and UTS values were obtained after T6 and painting process. A sharp decrease in mechanical properties was not observed. Results shows that it was not achieved to over-aging point.

At painting stage, YS and UTS values enhanced 23 % and 10 % respectively on average. On the other hand, elongation value was decreased approximately 2 %.

Applying the solution treatment is essential to meet the elongation values that are requested by customers. In addition, artificially aging should be applied to meet YS and UTS values. For these reasons, the optimization study of heat treatment becomes important.

5.1.2 Tensile Testing Results of Solution Treatment Experiments

The examination of current situation showed that the solution treatment stage of T6 is the most important stage for elongation, non-arguably. Therefore, in order to understand the effect of this stage on mechanical properties, the solution treatments performed at different temperatures and times as described in Table 4.1.

Yield strenght (YS) values are given in Table 5.1 as Rp_{max}, Rp_{min} and $Rp_{average}$. Each experiment was performed four times and the average was calculated.

Table 5.1 YS (Rp) values after solution treatment experiments

Experiment No	**Temp.(°C)**	**Time(min.)**	**Rp_{max} MPa**	**Rp_{min} MPa**	**$R_{paverage}$ MPa**
1		80	77	76	**76**
2		160	85	84	**85**
3	510	200	88	81	**85**
4		240	91	89	**90**
5		280	92	90	**91**
6		80	88	83	**86**
7		160	96	93	**94**
8	520	200	95	93	**94**
9		240	101	99	**100**
10		280	103	100	**101**
11		80	91	87	**88**
12		160	99	93	**94**
13	530	200	99	92	**95**
14		240	104	101	**98**
15		280	100	98	**96**
16		80	94	90	**92**
17		160	97	97	**97**
18	540	200	103	95	**99**
19		240	105	104	**104**
20		280	110	103	**106**
21		80	93	89	**91**
22		160	101	97	**99**
23	550	200	106	102	**103**
24		240	106	105	**105**
25		280	105	103	**104**

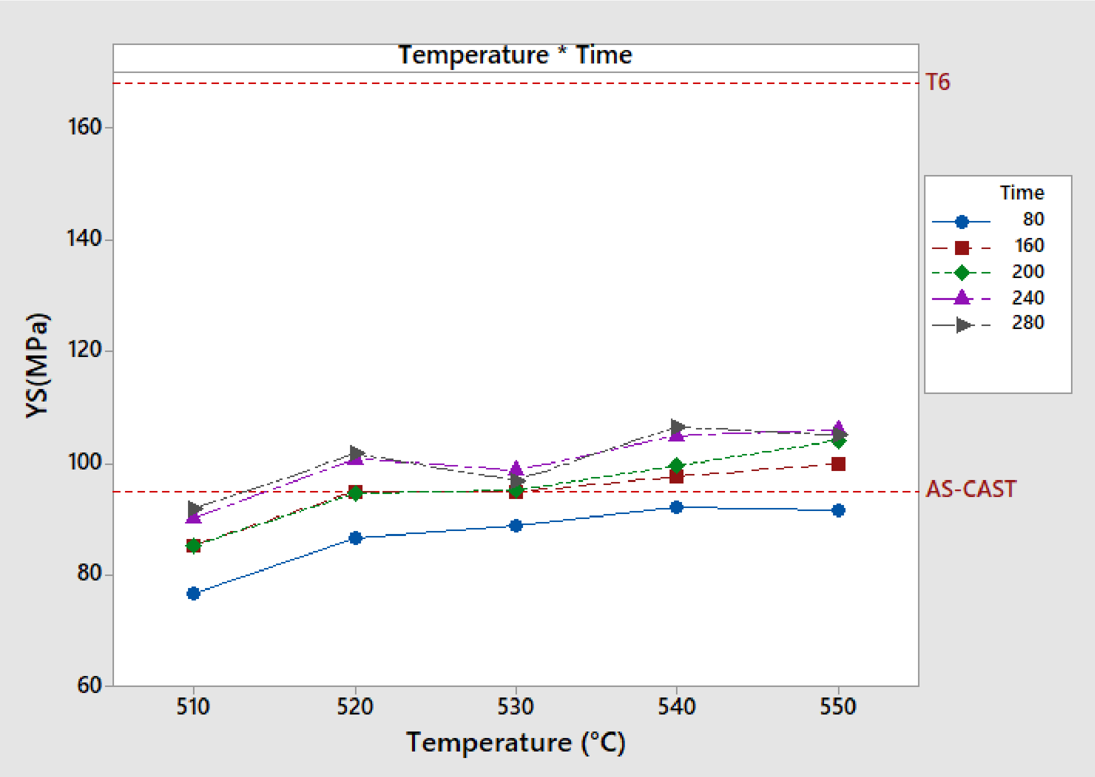


Figure 5.3 YS (Rp) values after solution treatment experiments

In the second part of the study, according to the tensile testing measurement results after solution treatment experiments, all yield strength (YS) values were obtained between the as-cast condition and T6 treatment condition.

As it is known from the literature, homogenization of the microstructure and dissolution of Mg and Si create vacancies during the solution treatment, which leads to distortionof Al matrix crystals and increase in YS (Lados, Diana, Apelian, Diran, 2011). As can be seen in Fig. 5.3, the increase in time and temperature was observed with the increase in YS values and the maximum YS value was determined at 550°C for 280 minutes. It can be said that there is no significant difference between the YS values obtained at different temperatures and times.

Ultimate tensile strenght (UTS) values are given in Table 5.2 as Rm_{max}, Rm_{min} and $Rm_{average}$. Each experiment was performed four times and average was calculated.

Table 5.2 UTS (Rm) values after solution treatment experiments

Experiment No	Temp.(°C)	Time(min.)	Rm_{max} MPa	Rm_{min} MPa	$R_{maverage}$ MPa
1		80	178	158	**168**
2		160	192	177	**185**
3	510	200	191	183	**187**
4		240	197	176	**189**
5		280	203	168	**190**
6		80	192	168	**181**
7		160	206	181	**195**
8	520	200	206	169	**186**
9		240	218	211	**214**
10		280	218	201	**210**
11		80	192	171	**180**
12		160	200	177	**186**
13	530	200	200	163	**186**
14		240	214	174	**193**
15		280	205	171	**191**
16		80	205	176	**190**
17		160	198	172	**189**
18	540	200	210	169	**190**
19		240	221	206	**215**
20		280	226	216	**221**
21		80	192	153	**174**
22		160	210	173	**190**
23	550	200	21	186	**204**
24		240	223	199	**212**
25		280	219	198	**209**

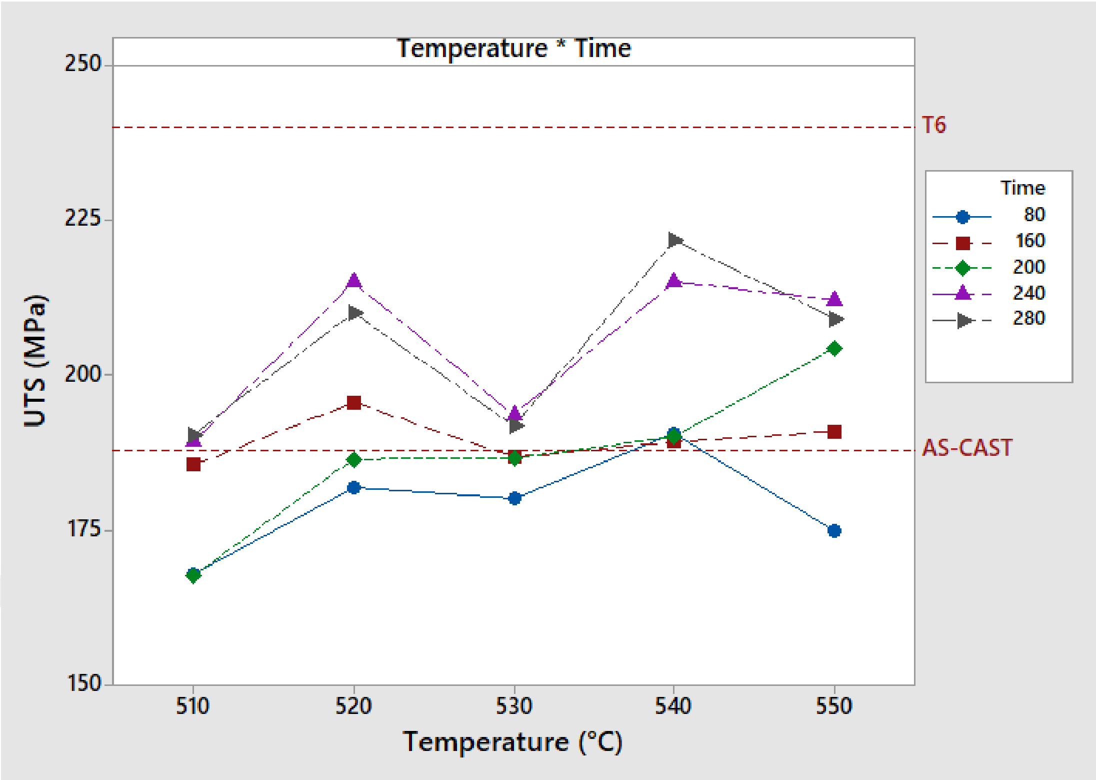


Figure 5.4 UTS (Rm) values after solution treatment experiments

Ultimate tensile strength (UTS) shows non-lineer behavior but similar with YS, UTS values between as-cast condition and T6 treatment condition because of the homogenization and Mg dissolution into Al matrix. Artificial aging which is the main strengthening stage, increases the YS and UTS however, in the solution treatment process, enhancing the mechanical properties must not be expected. To obtain above 200 MPa UTS, least 200 min. solution treatment must be applied to aluminium casting part as shown in Fig. 5.4.

ε% values are given in Table 5.3 as $A28_{max}$, $A28_{min}$ and $A28_{average}$. Each experiment was performed four times and average was calculated.

As it is known, in solution treatment stage, the morphology of eutectic silicon phase changes through the thermal modification. Spheroidization of the eutectic silicon phase provides ductility and better fracture toughness.

Table 5.3 ε% (A28) values after solution treatment experiments

Experiment No	Temp.(°C)	Time(min.)	$A28_{max}$ MPa	$A28_{min}$ MPa	$A28_{average}$ MPa
1		80	13.58	6.50	**9.79**
2		160	16.33	8.40	**12.62**
3	510	200	11.44	10.54	**10.99**
4		240	14.69	7.66	**11.27**
5		280	14.58	5.43	**11.15**
6		80	11.95	5.08	**9.16**
7		160	15.25	7.66	**11.50**
8	520	200	13.85	5.51	**9.18**
9		240	21.07	17.15	**18.96**
10		280	21.85	10.78	**15.93**
11		80	13.24	4.35	**8.45**
12		160	14.33	4.77	**8.85**
13	530	200	14.14	4.12	**8.83**
14		240	13.23	6.93	**9.61**
15		280	11.32	4.49	**9.99**
16		80	14.03	6.39	**10.32**
17		160	10.25	5.03	**8.34**
18	540	200	13.85	4.59	**8.25**
19		240	18.48	10.85	**15.40**
20		280	19.85	16.50	**18.58**
21		80	9.77	3.12	**6.60**
22		160	13.67	5.48	**8.88**
23	550	200	15.43	6.58	**11.00**
24		240	17.16	8.97	**12.84**
25		280	15.58	8.59	**11.79**

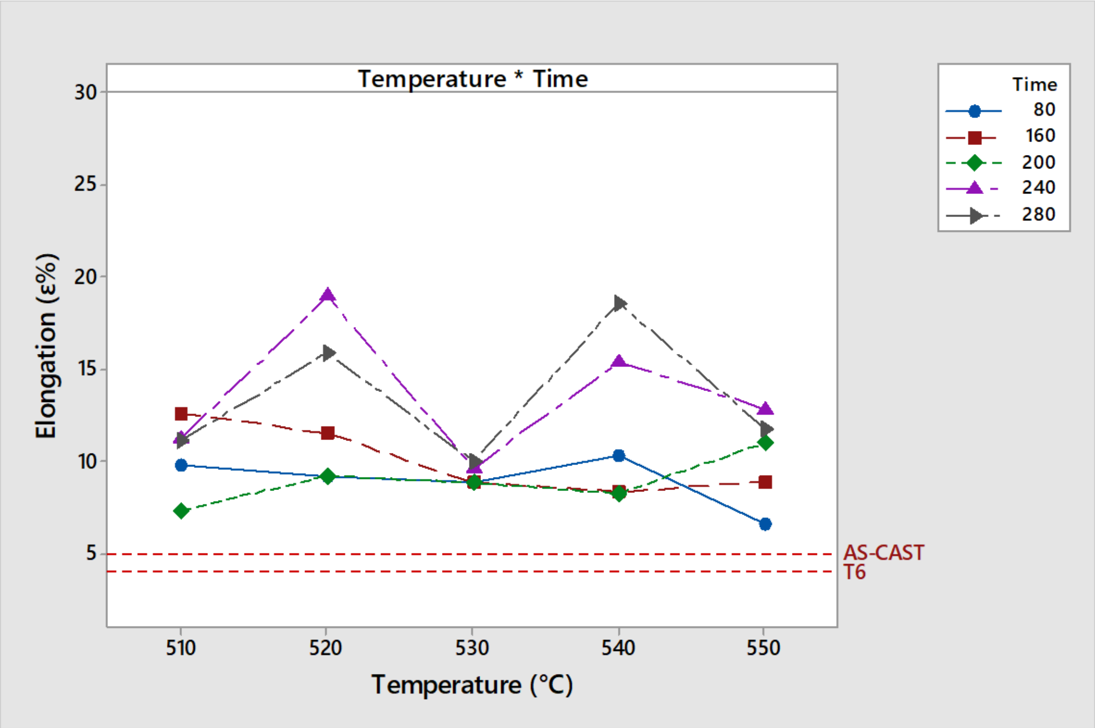


Figure 5.5 ε% (A28) values after solution treatment experiments

In addition, as mentioned in literature, solution treatment homogenizes the microstructure by dissolving the as-cast plate shape Mg_2Si precipitates, inclusions and by transforming the β-Al5FeSi intermetallic phases into the rounded phase (Zedan et al., 2010; Zander & Sandström, 2008). It reduces the segregation and elongation improves. Hence, test results as given in Fig. 5.5 show that elongation increases up to 19 % when parts treated at 520°C and 540°C for 240 min (Zhu et al., 2012).

Low elongation values were obtained at 530°C for all test time. Microstructure of the sample is shown in Fig. 5.26. It was observed that Fe-rich intermetallic phases exist in α-Al matrix. These phases are formed because of the low solidification rate at casting process. Fe intermetallic exists in α-Al matrix in two forms: α-Fe and β-Fe forms. α-Fe is also known as Chinese script. Comparing to the α-Fe phase, β-Fe effects the elongation and toughness more in negative way (Mohamed, Samuel, Samuel & Doty, 2009). In this case, obtained low values can be explained by these brittle Fe-rich intermetallic phases.

5.1.3 Tensile Testing Results Of Optimization Experiments

Results of optimization experiments (Rp, Rm, A28) were given in Table 5.4.

Table 5.4 YS (Rp), UTS (Rm) and Ɛ% (A28) values of optimization experiments

Exp. No	Std Order	Run Order	Center Pt	Blocks	SHT Temp. (C°)	SHT Time (min.)	Quenhing Temp. (C°)	Aging Temp. (C°)	Aging Time (min.)	Rp average MPa	Rm average MPa	A28 average MPa
26	15	1	1	1	480	240	80	180	180	**81**	**153**	**5.49**
27	6	2	1	1	540	80	80	180	60	**132**	**202**	**3.40**
28	5	3	1	1	480	80	80	180	60	**73**	**148**	**6.15**
29	13	4	1	1	480	80	80	180	180	**77**	**146**	**4.69**
30	12	5	1	1	540	240	80	120	180	**170**	**227**	**3.03**
31	4	6	1	1	540	240	80	120	60	**133**	**186**	**2.43**
32	17	7	0	1	510	160	80	150	120	**114**	**186**	**3.94**
33	8	8	1	1	540	240	80	180	60	**184**	**242**	**3.21**
34	1	9	1	1	480	80	80	120	60	**70**	**149**	**5.96**
35	16	10	1	1	540	240	80	180	180	**198**	**244**	**2.19**
36	3	11	1	1	480	240	80	120	60	**79**	**159**	**6.32**
37	14	12	1	1	540	80	80	180	180	**171**	**222**	**1.87**
38	18	13	0	1	510	160	80	150	120	**120**	**192**	**3.99**
39	19	14	0	1	510	160	80	150	120	**120**	**192**	**3.99**
40	10	15	1	1	540	80	80	120	180	**135**	**198**	**2.90**
41	2	16	1	1	540	80	80	120	60	**110**	**184**	**3.97**
42	9	17	1	1	480	80	80	120	180	**71**	**146**	**5.99**
43	7	18	1	1	480	240	80	180	60	**81**	**159**	**6.27**
44	11	19	1	1	480	240	80	120	180	**76**	**152**	**5.82**

5.1.3.1 Statistical Analysis of Yield Strength Results

According to the main effect analysis, it was found that solution treatment temperature is the most effective parameter on the YS values, but the values obtained at 480°C were the same as the casting state (80 MPa) and this temperature was not sufficient to provide the solubility of the Mg_2Si phase in the structure and to form a supersaturated solid solution which gave the precipitation hardening ability to material. Besides that YS is affected positively by solution treatment time, aging time and temperature in linear proportional as given in Fig. 5.6.

In order to obtain desired YS, solution treatment and aging must be carried out at least 516°C for 240 min. and 180°C for 180 min., respectively. Similar results might be obtained at 540°C for 80 min. and 180°C for 180 min. Interactions between parameters were given in Fig. 5.7. A gray background represents a term that was removed from the model because it is not statistically significant. Detailed optimization parameters and five different options were settled in Fig. 5.8.

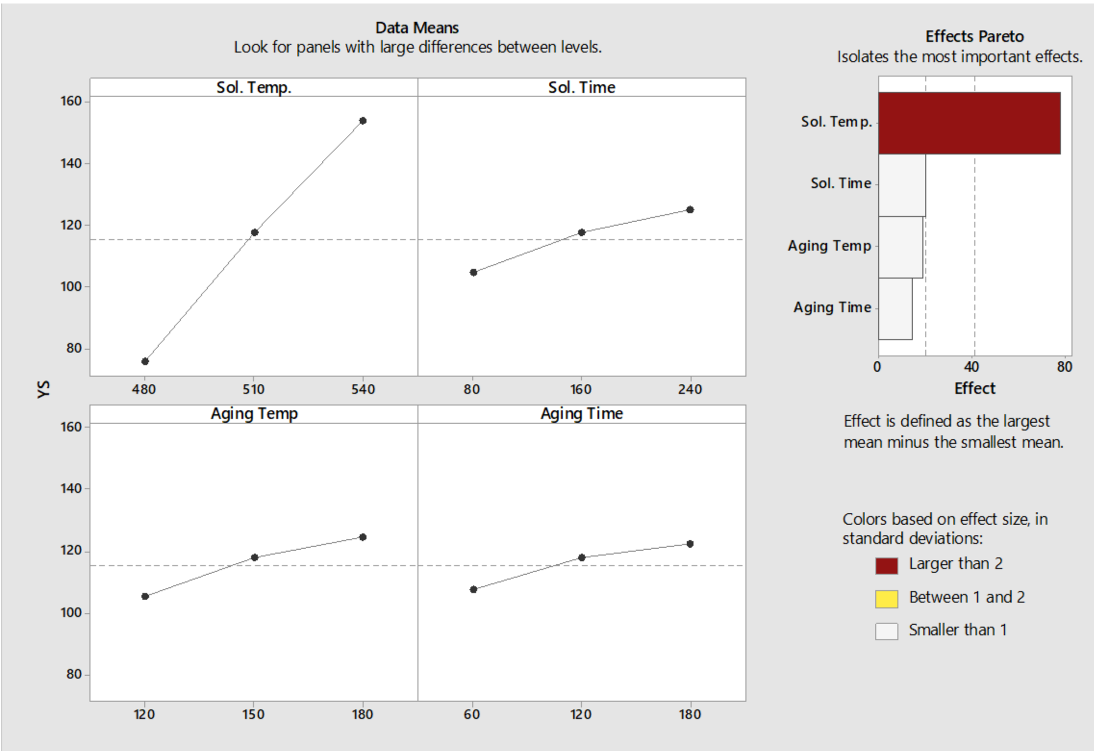


Figure 5.6 Main effects screener for yield strength

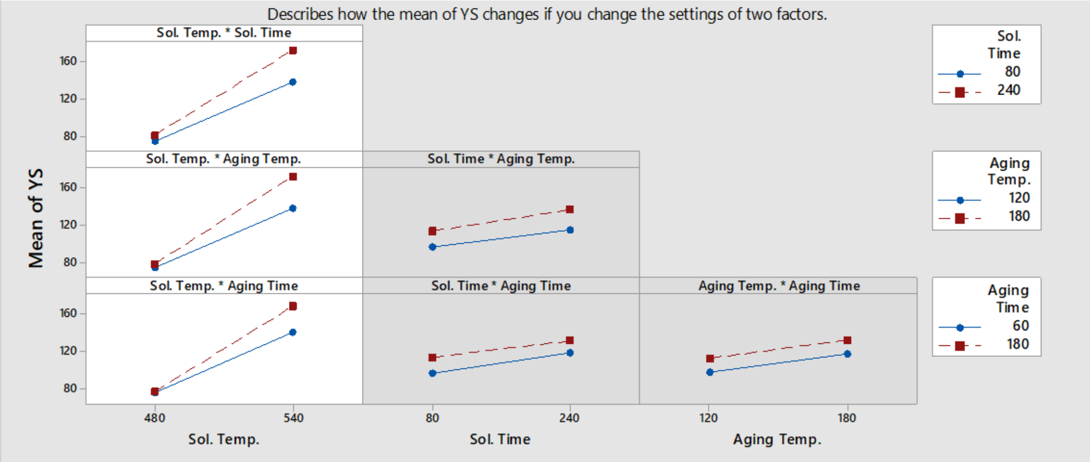


Figure 5.7 Interaction plots for yield strength

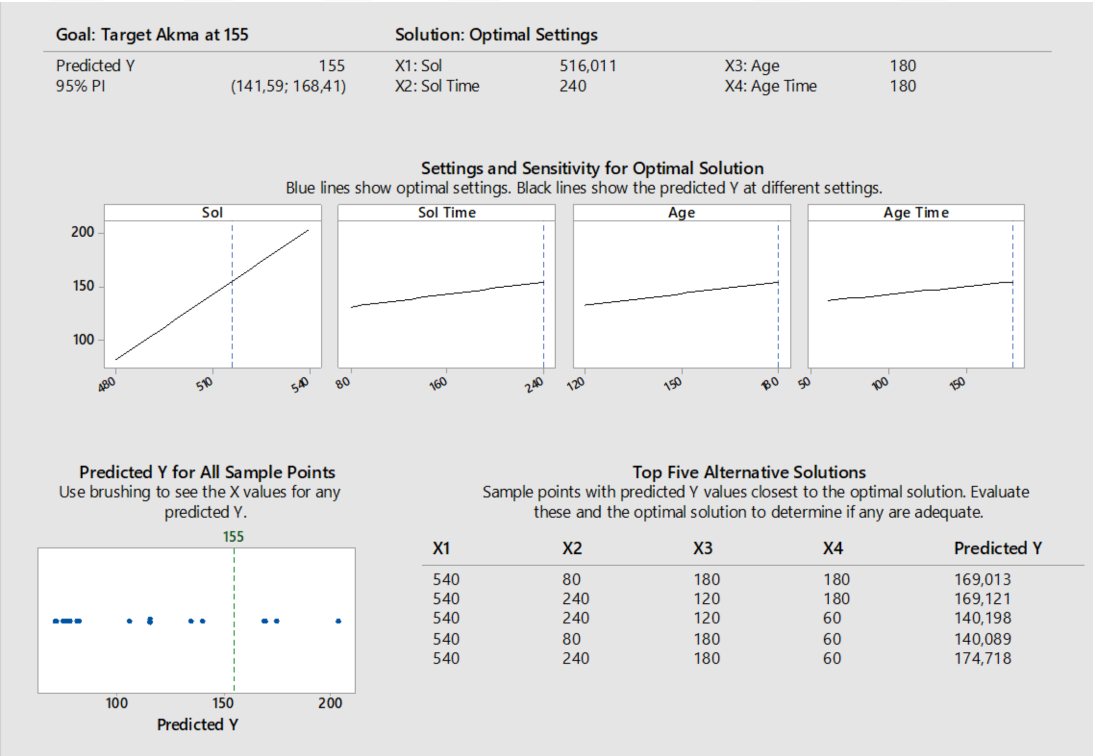
According to multiple regression analysis, the formulation of the YS value depending on the variables was given.

Multiple Regression for YS
Model Building Report

X1: Sol. Temp.   X2: Sol. Time   X3: Aging Temp.   X4: Aging Time

Final Model Equation

YS = 498 - 0,914 X1 - 1,366 X2 - 4,028 X3 - 1,871 X4 + 0,002931 X1*X2 + 0,00852 X1*X3 + 0,003910 X1*X4



X1	X2	X3	X4	Predicted Y
540	80	180	180	169,013
540	240	120	180	169,121
540	240	120	60	140,198
540	80	180	60	140,089
540	240	180	60	174,718

Figure 5.8 Prediction report and five alternative parameters for yield strength (Target 155MPa)

5.1.3.2 Statistical Analysis of Elongation Results

According to main effect analysis, it was found that solution treatment temperature is the most effective parameter on ε% value but the values obtained at 480°C were the same as the casting state (6 %) and this temperature is not sufficient to spherozation of acicular shape eutectic silicons in the microstructure. However, as the solution treatment temperature increases, obtained ε% values decreases after T6 treatment. Besides that ε% is affected negatively by aging temperature and time in linear proportional as given in Fig. 5.9.

In order to obtain the maximum value for ε%, solution treatment temperature, aging time and temperature must be minimize as much as possible however parameters must be adjusted to reach desired YS and HB at the same time. Hence, solution temperature must be 516°C at least. Detailed optimization parameters were settled in Fig. 5.11.

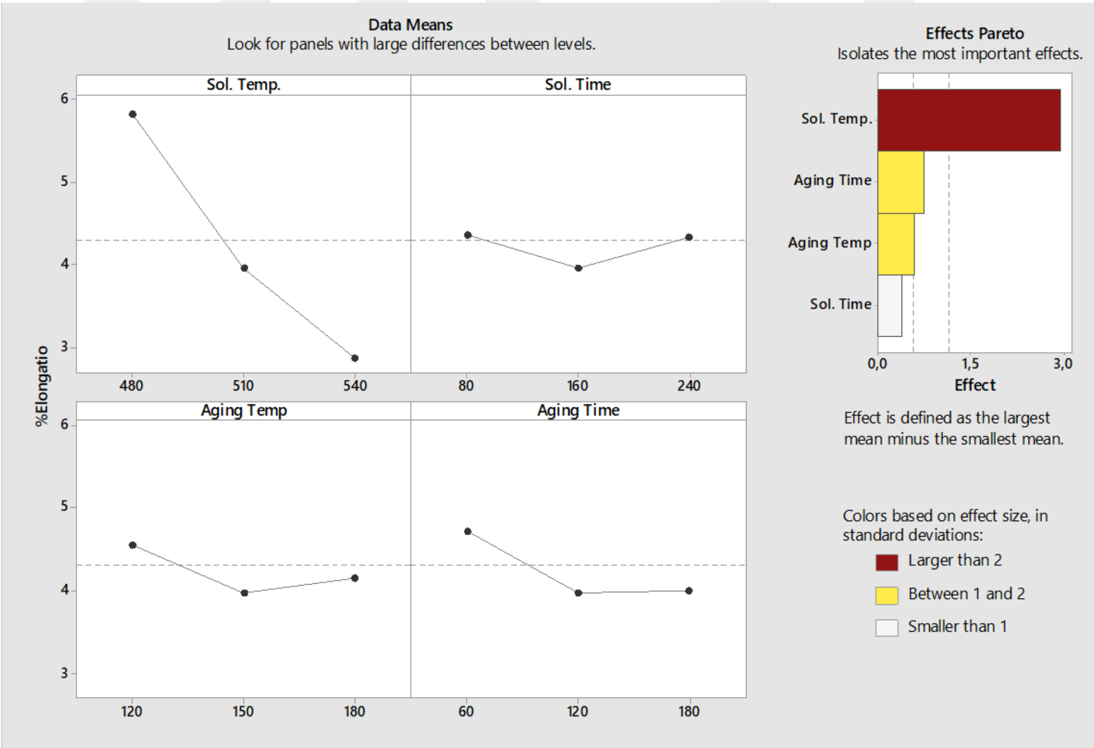


Figure 5.9 Main effect screener for elongation

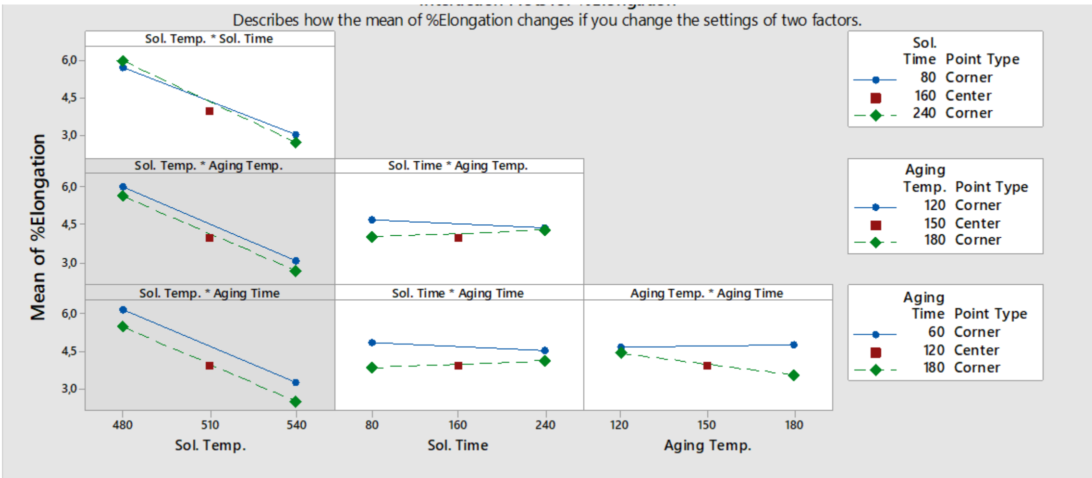


Figure 5.10 Interaction plots for elongation

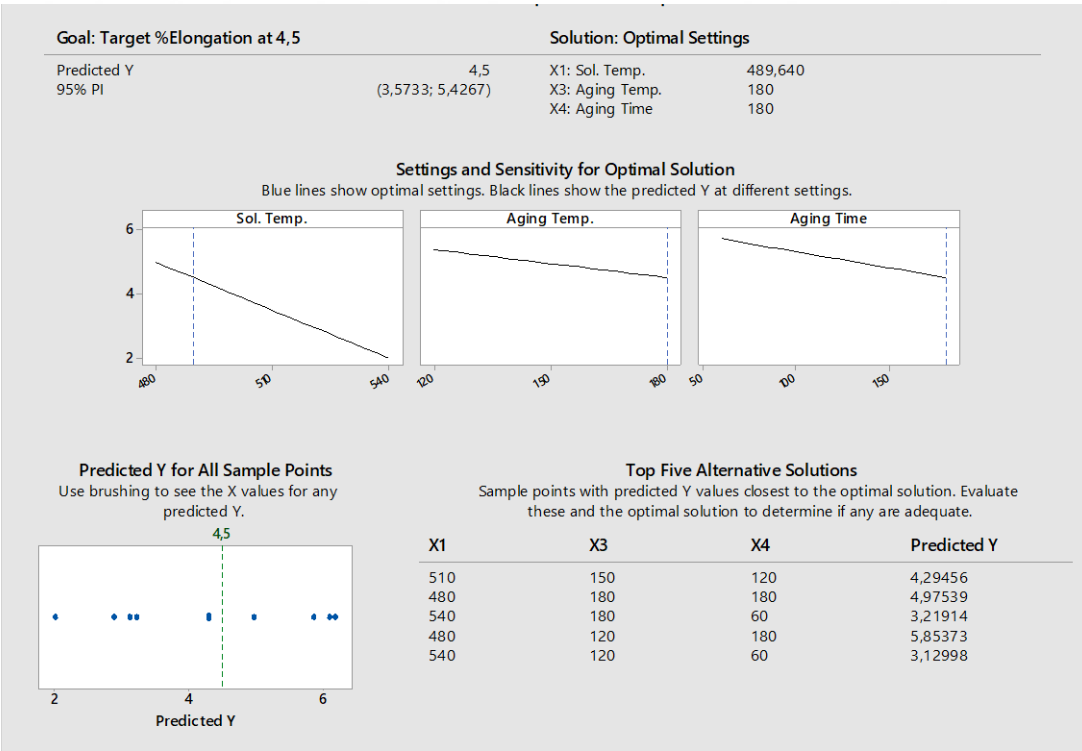
According to multiple regression analysis, the formulation of the ε% value depending on the variables was given.

Multiple Regression for %Elongation
Model Building Report

X1: Sol. Temp. X2: Sol. Time X3: Aging Temp. X4: Aging Time

Final Model Equation

%Elongation = 28,73 - 0,04931 X1 + 0,00955 X3 + 0,01417 X4 - 0,000134 X3*X4



X1	X3	X4	Predicted Y
510	150	120	4,29456
480	180	180	4,97539
540	180	60	3,21914
480	120	180	5,85373
540	120	60	3,12998

Figure 5.11 Prediction report and five alternative parameters for elongation (Target 4.5 ε%)

5.1.4 Tensile Testing Results of Quenching Experiments

Results of quenching experiments (Rp, Rm, A28), maximum cooling rates and Leidenfrost points were given in Table 5.5.

Table 5.5 YS (Rp), UTS (Rm), Ɛ% (A28) values of quenching experiments

Experiment No	Quenchant	Maximum Cooling Rate (°C/s)	Leidenfrost Point (°C)	Rp average MPa	Rm average MPa	A28 average MPa
45	Water	85	442	140	230	7.5
46	SDS	61	385	135	229	8.2
47	Triton X-100	68	448	135	229	8.2
48	Tween 20	70	453	129	225	8.2

According to results, quenching with water displays higher YS than the surfactant quenchants. Nevertheless, YS values were obtained similar between 129-140 MPa as seen in Table 5.5. It found that critical cooling rate was provided to suppress precipitation in all quenching mediums and 61°C/s (Tween 20) was enough to obtain supersaturated solid solution. In both water and surfactant quenchant cooling mediums, obtained the same elongation values and standard deviations were equal.

Cooling behavior of A356 wheel parts at four different mediums were given in Fig. 5.12. Experiment results show that the shortest (10 second) and the fastest (85°C/s) cooling was obtained with water. Triton X-100 and Tween quenchants slowed down the cooling rate to approximately 70°C/s. The longest (20 second) and the slowest cooling (61°C/s) was obtained with SDS quenchant.

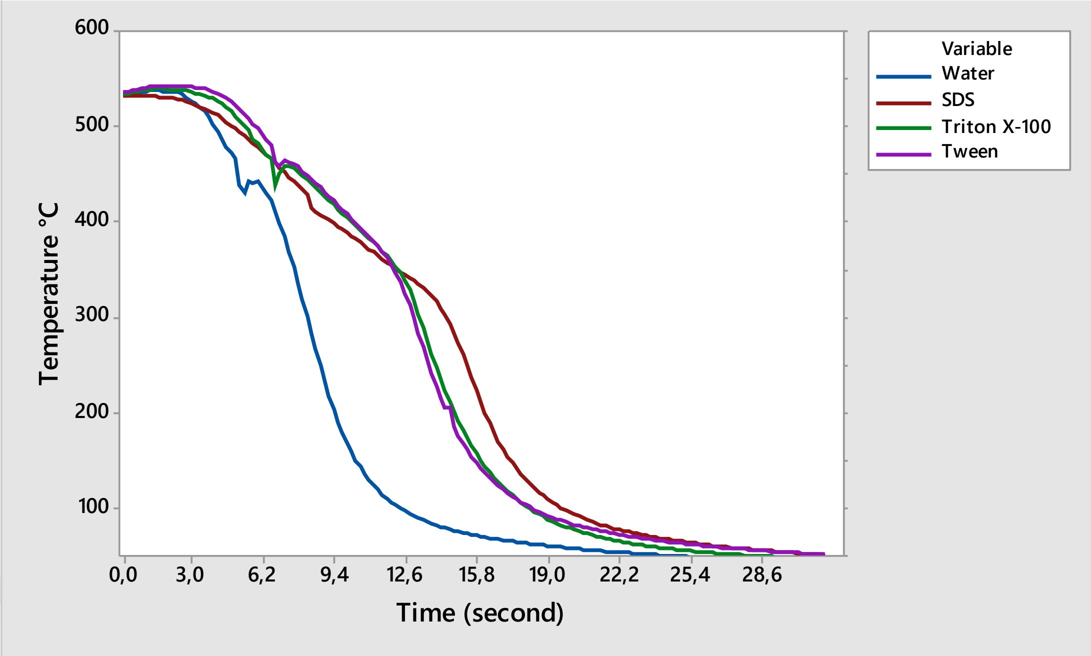


Figure 5.12 Cooling behavior at different quench mediums

Low cooling rates with SDS, Triton and Tween, can be explained by surfactant layer that composed on the surface at the beginning of cooling when part immersed into surfactant quenchants. This layer slows down the cooling rate like polymer or oil quenchants as mentioned in literature before. Fragmentation of layer and direct contact with aluminium surface begins when Leidenfrost point surpass. It was determined that surfactant layer fragmentation occurs in 4 seconds as illustrated in Fig. 5.13. In water medium, nucleate boiling phase was began directly after reaching Leidenfrost point.

Vapor blanket (film boiling), nucleate boiling and convection cooling phases can be seen on Fig. 5.12. and 5.13. Maximum cooling rate and Leidenfrost points of each quenchant were determined for the first time for A356 alloy by using surfactant addition.

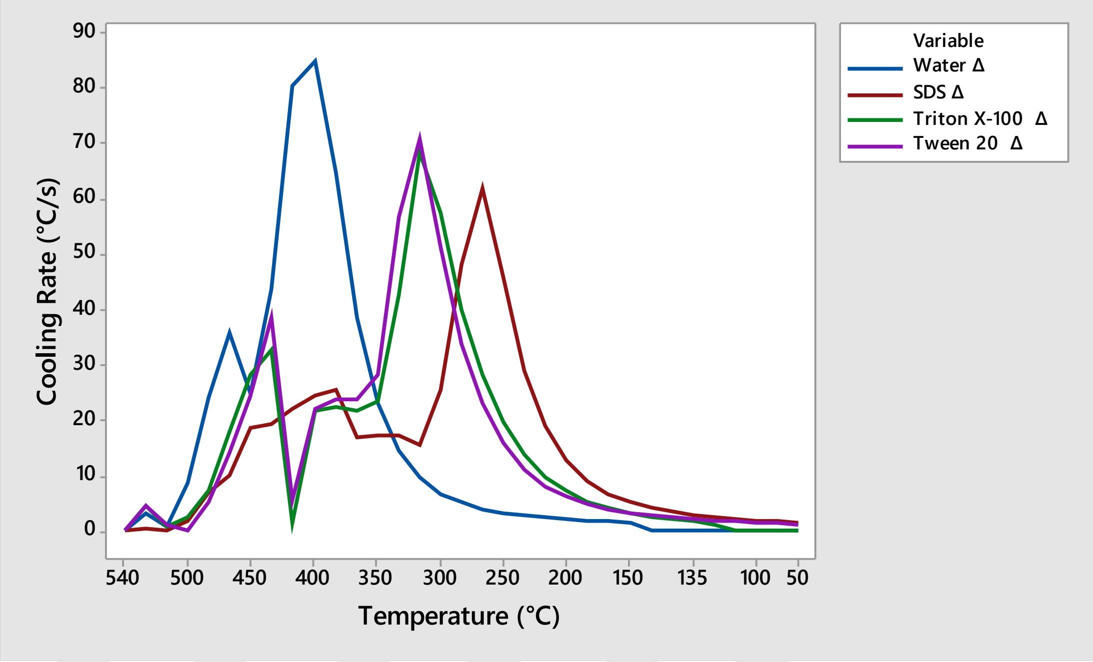


Figure 5.13 Cooling rate at different quench mediums

5.2 Hardness Test Results

5.2.1 Hardness Test Results of Current Situation

In order to analyze current situation and determine the effect of different heat treatment stages on mechanical properties, hardness testing was applied to low pressure die cast A356 aluminium wheels as indicated section 4.4.2.

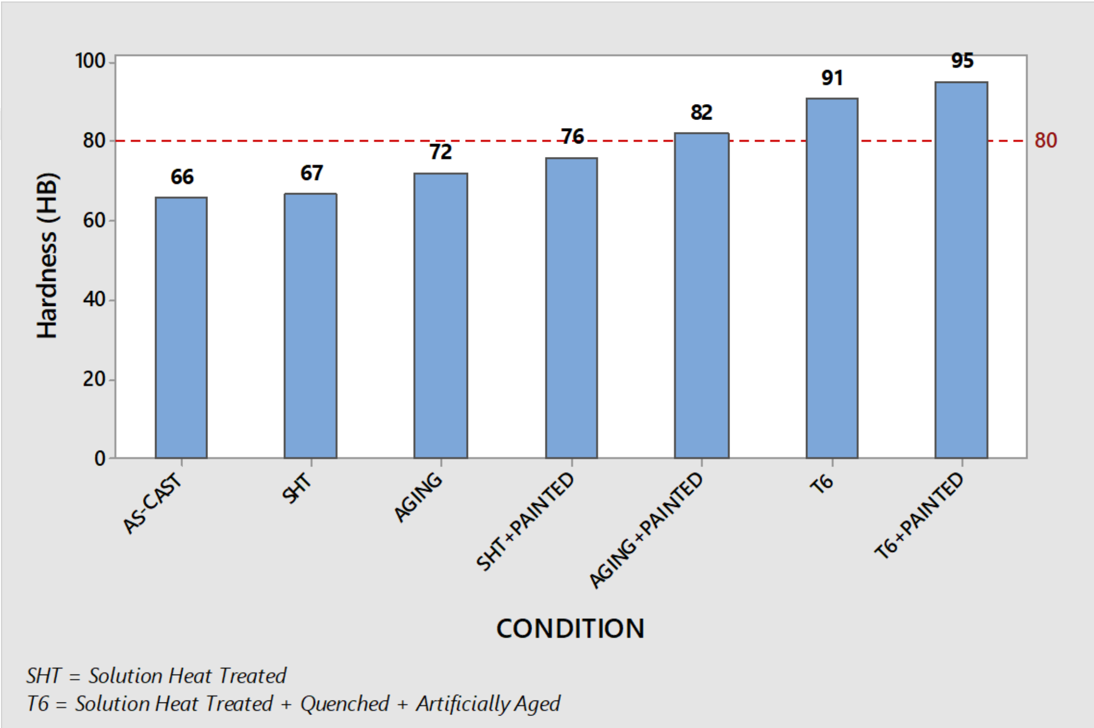


Figure 5.14 HB results at different heat treatment stages

There is no significant change in hardness values after solution treatment as shown in Fig. 5.14. Otherwise, hardness value increased after T5 treatment in which only only artificial aging. It can be explained by precipitation theory of Mg_2Si, formation of Mg-Si clusters and GP zones. Compared to as-cast samples, after the entire T6 heat treatment hardness values of casting parts increased by 40% from 66 HB to 91,42 HB. The highest hardness values were obtained after T6+painting process as 95HB. It was also determined that painting process, after only solution treatment or only artificially aging, increased the hardness value approximetely 12%

however after T6 heat treatment increased only 4% because of the majority of secondary hardening phases was already precipitated into Al matrix.

5.2.2 Hardness Test Results of Solution Treatment Experiments

Brinell hardness values are given in Table 5.6 as Brinell. Mean datas was obtained from three hardness measurement of each specimen.

Table 5.6 Hardness(HB) values of solution treatment experiments

Experiment No	Temp.(°C)	Time(min.)	Hardness (HB)
AS-CAST			**66**
T6			**91**
1	510	80	**57**
2		160	**62**
3		200	**56**
4		240	**62**
5		280	**53**
6	520	80	**63**
7		160	**65**
8		200	**65**
9		240	**65**
10		280	**60**
11	530	80	**57**
12		160	**63**
13		200	**60**
14		240	**64**
15		280	**65**
16	540	80	**63**
17		160	**63**
18		200	**66**
19		240	**63**
20		280	**64**
21	550	80	**63**
22		160	**60**
23		200	**68**
24		240	**65**
25		280	**64**

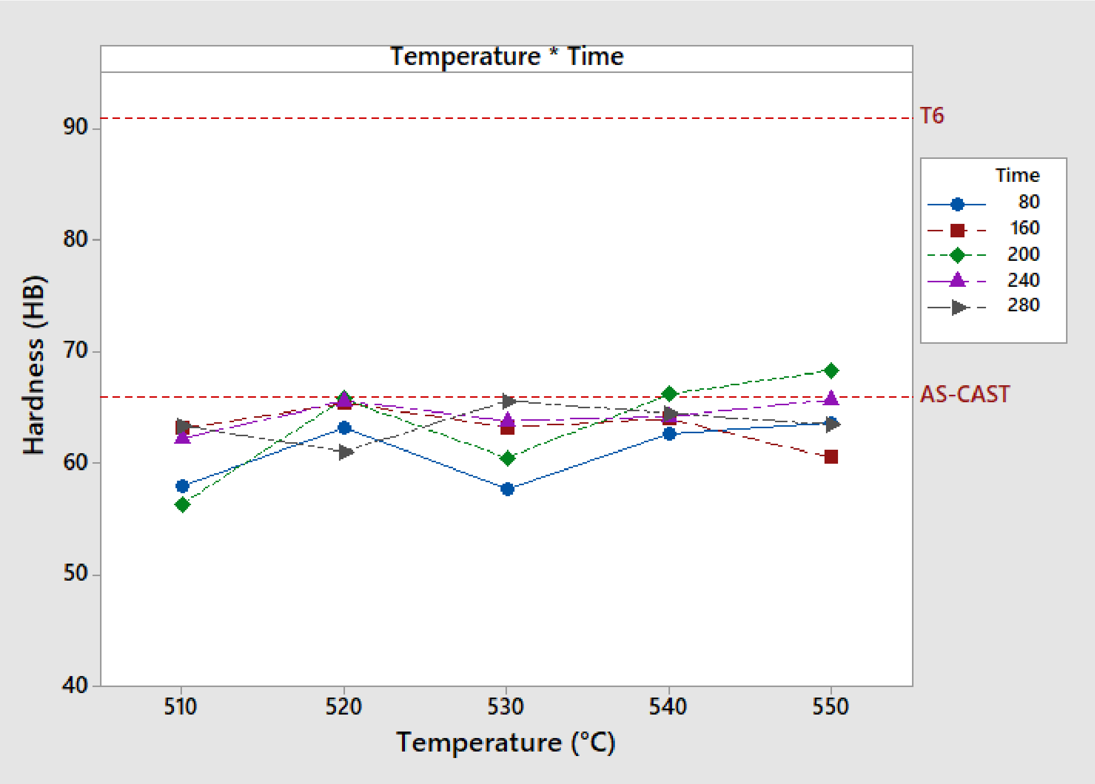


Figure 5.15 HB values of solution treatment experiments

Distribution of hardness values shows that soft aluminium matrix was hardened through the Mg-Si dissolution but there is no meaningful differences between low and high temperatures. As mentioned in section 3.2.3. hard Mg_2Si phase disperse into matrix homogeneously after aging proecess as fine β” and thus hardness of material increase. However, in solution treatment stage, hardness improvement was seen up to only 5 HB (Fig. 5.15). Hardness results are compatible with hardness of low pressure die cast wheels and it measured approximately 60-65 HB. With this study, it is understood that the solution treatment has no significant effect on hardness without any aging or painting process.

5.2.3 Hardness Test Results of Optimization Experiments

Hardness(HB) results of optimization experiments are given in Table 5.7.

Table 5.7 Hardness(HB) values of optimization experiments

Exp. No	**Std Order**	**Run Order**	**Center Pt**	**Blocks**	**SHT Temp.(C°)**	**SHT Time(min.)**	**Quenh Temp. (C°)**	**Aging Temp.(C°)**	**Aging Time(min.)**	**Hardness (HB)**
26	15	1	1	1	480	240	80	180	180	**54**
27	6	2	1	1	540	80	80	180	60	**71**
28	5	3	1	1	480	80	80	180	60	**49**
29	13	4	1	1	480	80	80	180	180	**50**
30	12	5	1	1	540	240	80	120	180	**78**
31	4	6	1	1	540	240	80	120	60	**79**
32	17	7	0	1	510	160	80	150	120	**66**
33	8	8	1	1	540	240	80	180	60	**82**
34	1	9	1	1	480	80	80	120	60	**48**
35	16	10	1	1	540	240	80	180	180	**86**
36	3	11	1	1	480	240	80	120	60	**51**
37	14	12	1	1	540	80	80	180	180	**84**
38	18	13	0	1	510	160	80	150	120	**68**
39	19	14	0	1	510	160	80	150	120	**68**
40	10	15	1	1	540	80	80	120	180	**68**
41	2	16	1	1	540	80	80	120	60	**61**
42	9	17	1	1	480	80	80	120	180	**47**
43	7	18	1	1	480	240	80	180	60	**49**
44	11	19	1	1	480	240	80	120	180	**49**

According to the main effect analysis, it was found that solution treatment temperature is the most effective parameter on HB values, but the values obtained at 480°C are the same as the hardness of the sample after casting (50 HB) and similarly with YS value assessment in section 5.1.3.1 this temperature was not sufficient to provide supersaturated solution for catalyzing the precipitation hardening. Therefore, hardness of the material was seen steady. On the other hand, HB increases to a certain limit value with increasing aging temperature and time (Fig. 5.16). This results reflects the findings of Fracasso(2010) and Menargues et al. (2015).

In order to obtain desired hardness, solution treatment and aging must be applied following at least 522°C for 240 min. and 180°C for 180 min. Similar results might be obtained, independent from aging time, at 540°C for 80 min. solution and 180°C aging or 540°C for 240 min. solution and 120°C aging. Detailed optimization parameters and five different options were settled in Fig.5.18.

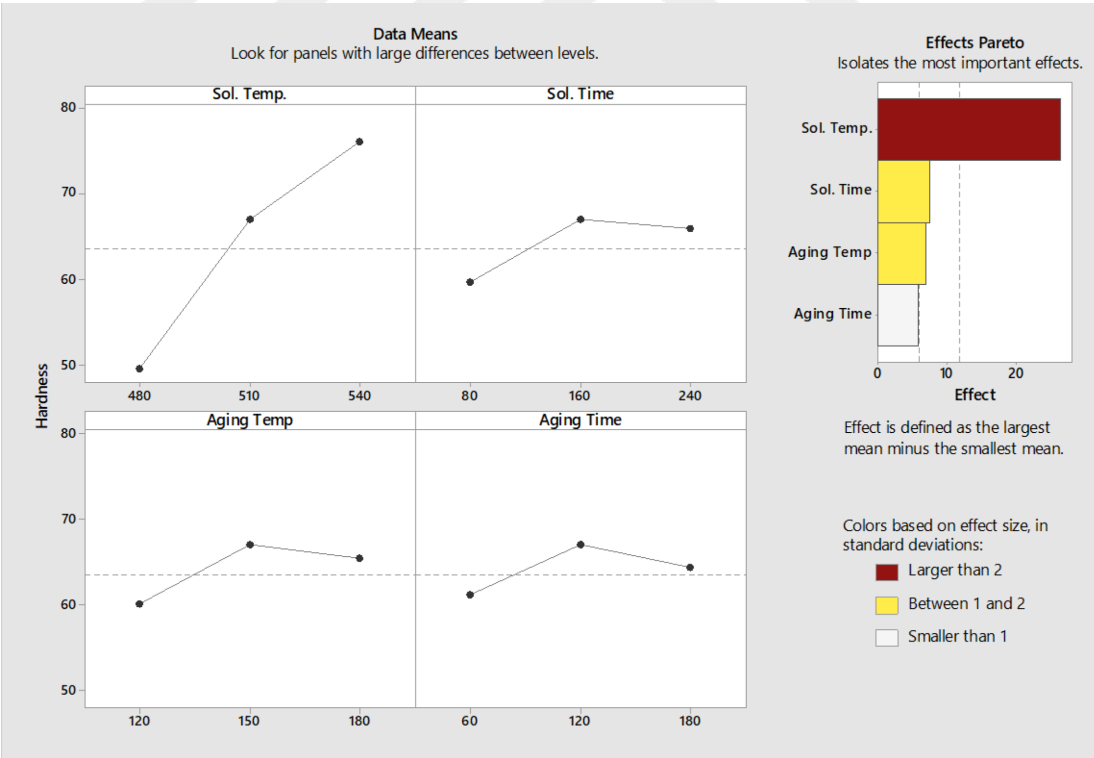


Figure 5.16 Main effect screener for hardness

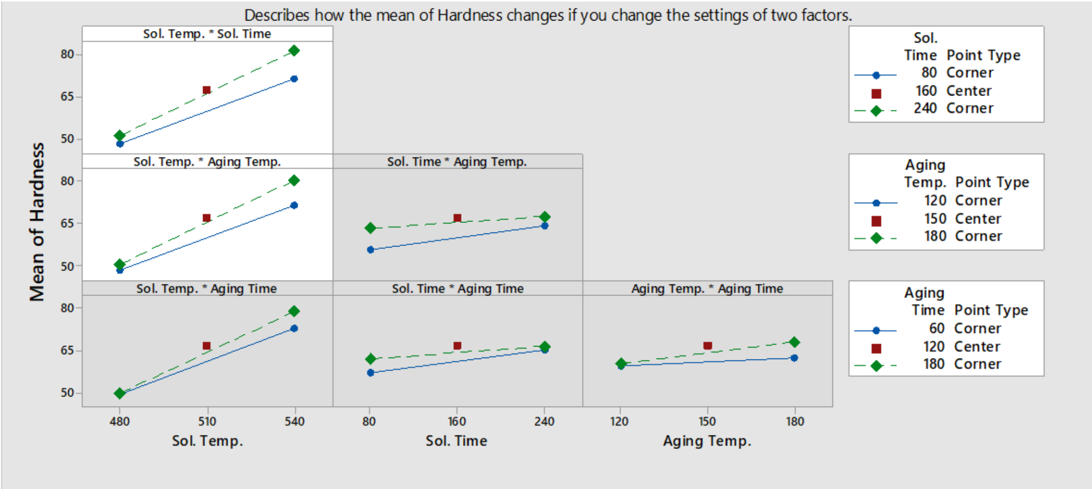


Figure 5.17 Interaction plots for hardness

According to multiple regression analysis, the formulation of the HB value depending on the variables was given.

Multiple Regression for Hardness
Model Building Report

X1: Sol. Temp. X2: Sol. Time X3: Aging Temp. X4: Aging Time

Final Model Equation

Hardness = -181,9 + 0,4424 X1 + 0,0398 X2 + 0,0896 X3

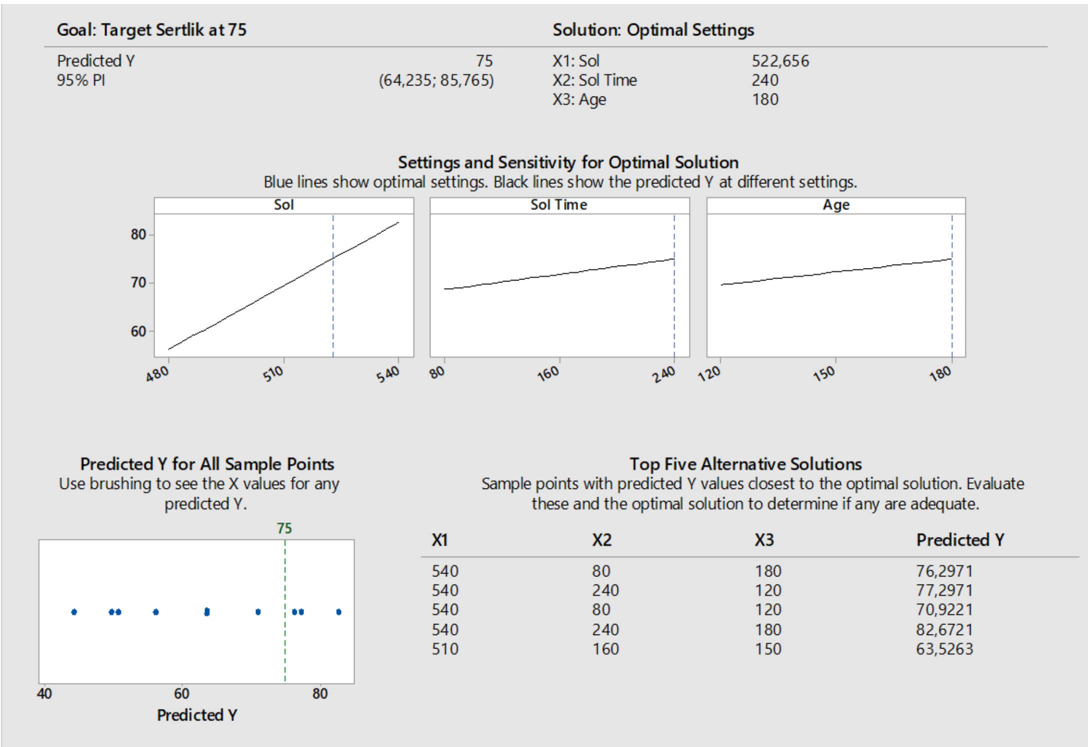


Figure 5.18 Prediction report and five alternative parameters for hardness (Target 75HB)

5.2.4 Hardness Test Results of Quenching Experiments

Hardness measurement of optimization experiments (HB) were given in Table 5.8. According to the hardness results, it was determined that 61°C/s cooling rate is enough to supress solute atoms into supersaturated solid solution (SSS) and critical rate for A356 alloy was similar with Milkereit et al. (2014) study. Hardness values were obtained at range of 61-85°C/s cooling rate as seen in Fig. 5.19.

Table 5.8 Hardness(HB) values of quenching experiments

Experiment No	Quenchant	Maximum Cooling Rate (°C/s)	Leidenfrost Point (°C)	Hardness (HB)
45	Water	85	430	77
46	SDS	61	415	78
47	Triton X-100	68	438	76
48	Tween 20	70	459	75

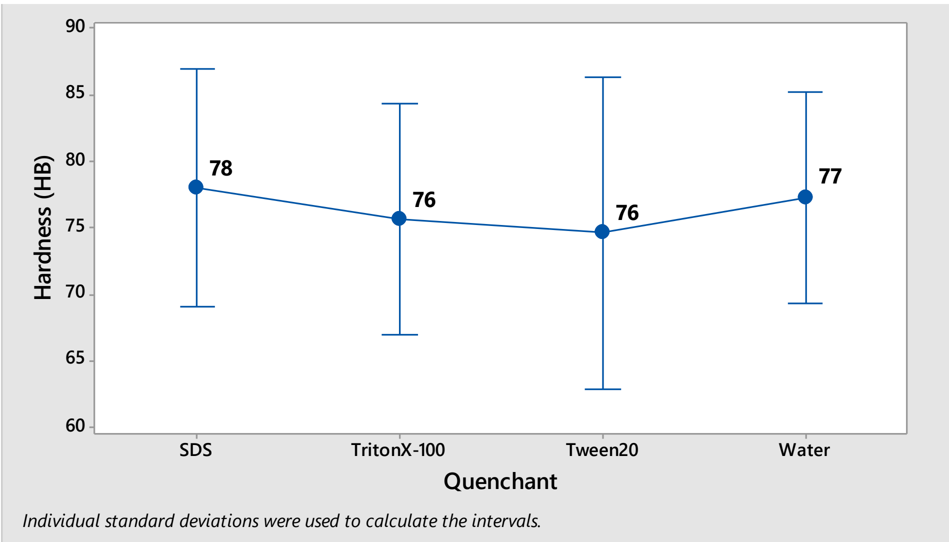


Figure 5.19 HB values of quenching experiments

5.3 Microstructure Analysis Results

Microstructures of the wheel parts after heat treatment are given in Fig. 5.20. Lamellar eutectic silicon phases and plate-shape intermetallic phases are seen in as-cast sample. After T6 treatment, spherodatization is occured and plate-shape Mg_2Si and other intermetallic phases disappared. Changes on phases directly effect the mechanical properties so microstructural analyze must be done adequately. After solution treatment experiments, all parts was examined with optical microscopy and micrographs were taken at 200x and 500x magnifications.

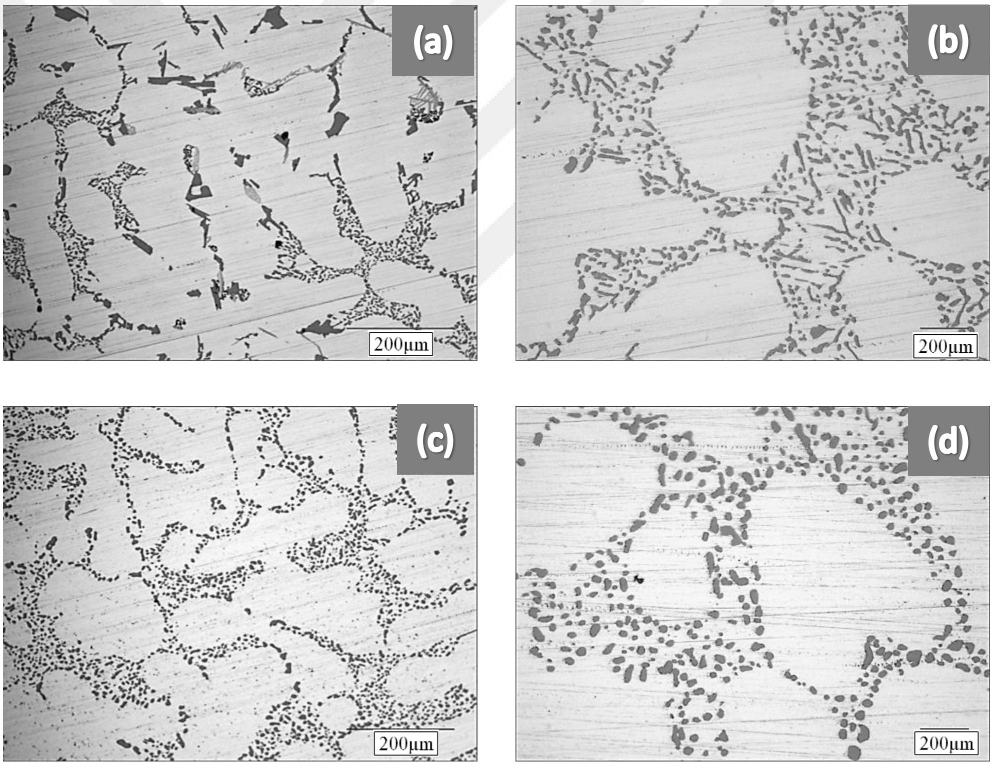


Figure 5.20 Microstructures of as-cast (a)200X, (b)500X and T6 heat treated (c)200X, (d)500X samples

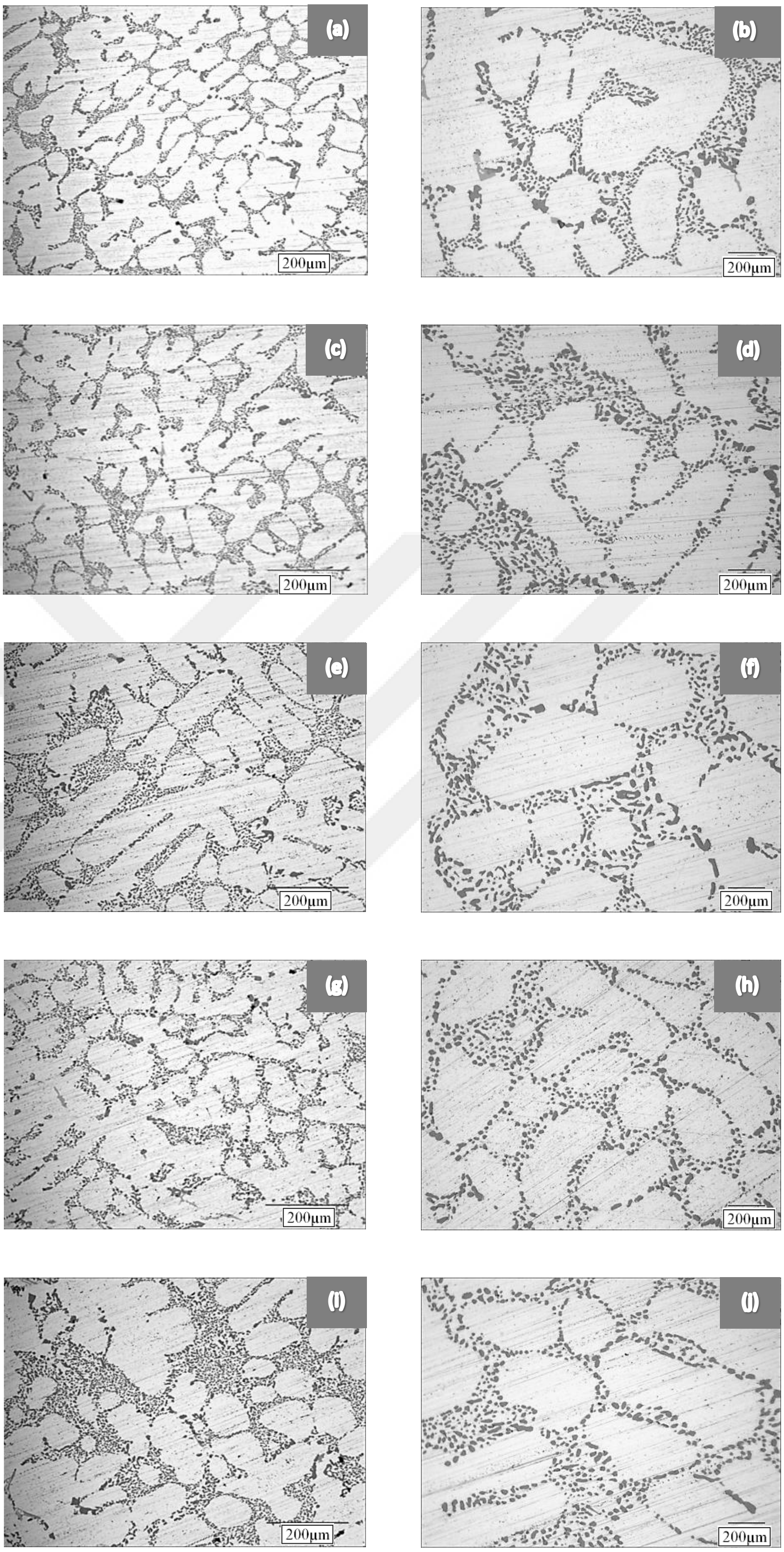


Figure 5.21 Microstructures of solution treated parts at 510°C at 200X and 500X for (a)(b)80 min, (c)(d)160min, (e)(f)200min, (g)(h)240min, (i)(j)280min

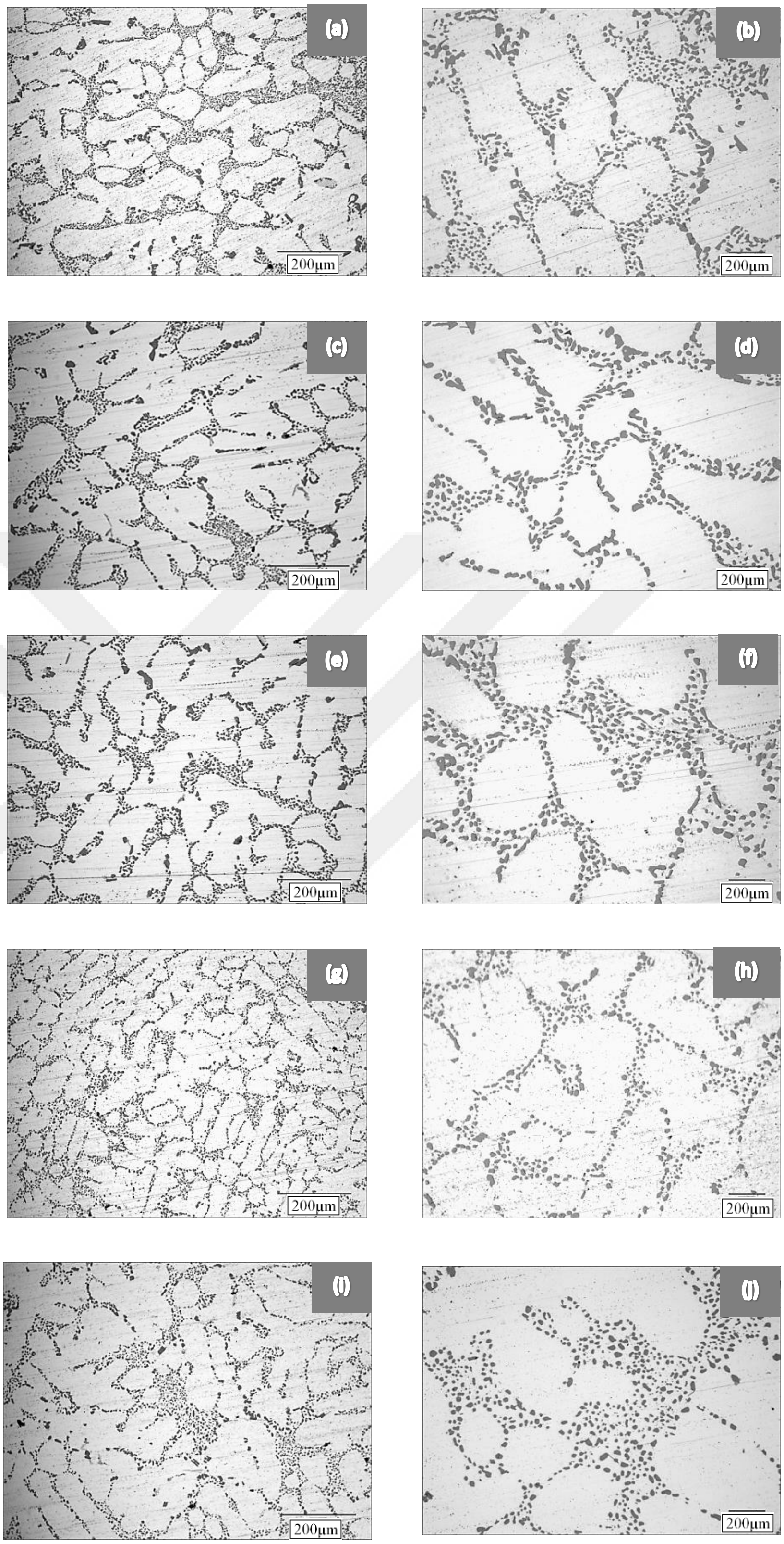


Figure 5.22 Microstructures of solution treated parts at 520°C at 200X and 500X for (a)(b)80 min, (c)(d)160min, (e)(f)200min, (g)(h)240min, (i)(j)280min

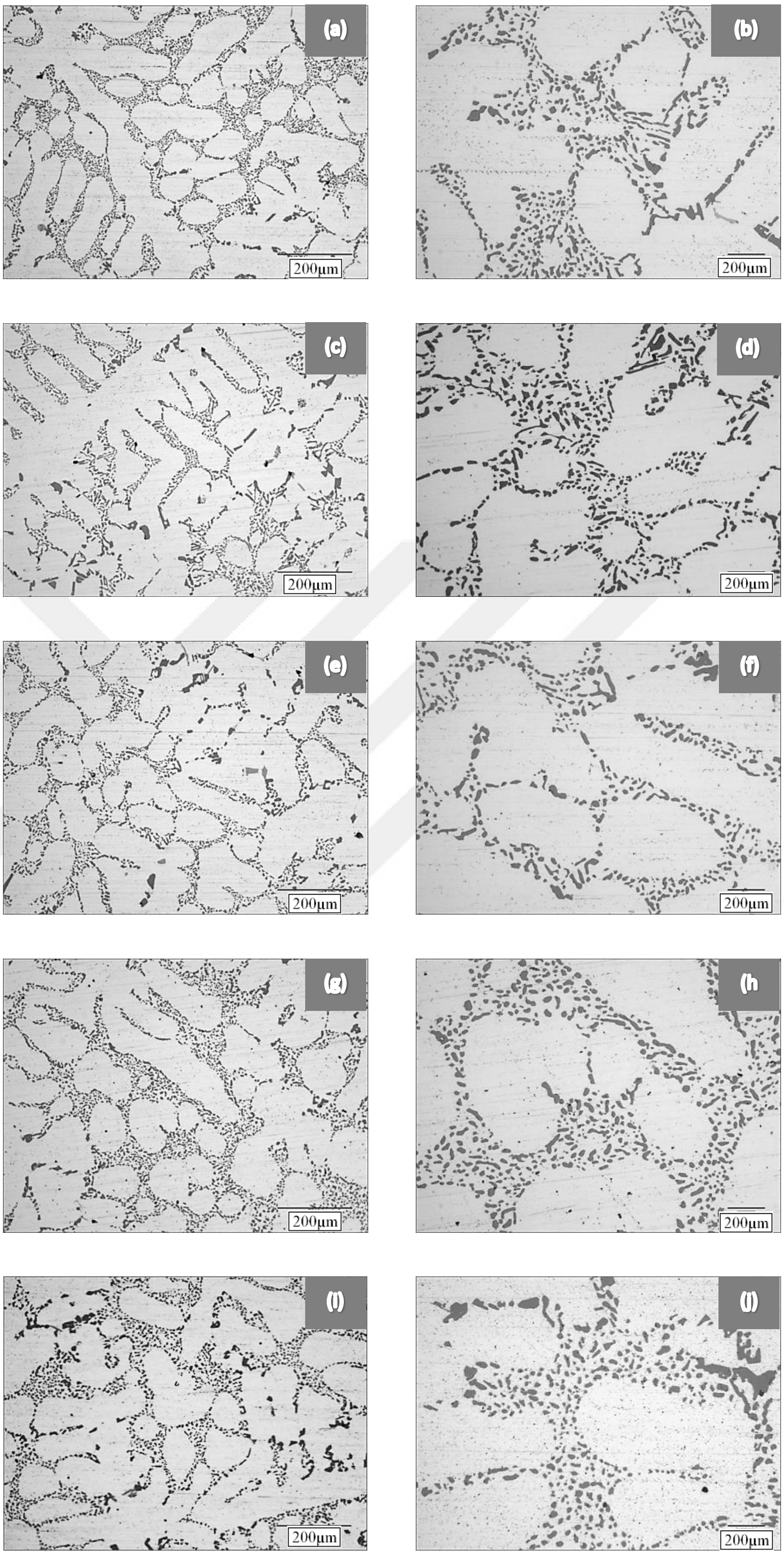


Figure 5.23 Microstructures of solution treated parts at 530°C at 200X and 500X for (a)(b)80 min, (c)(d)160min, (e)(f)200min, (g)(h)240min, (i)(j)280min

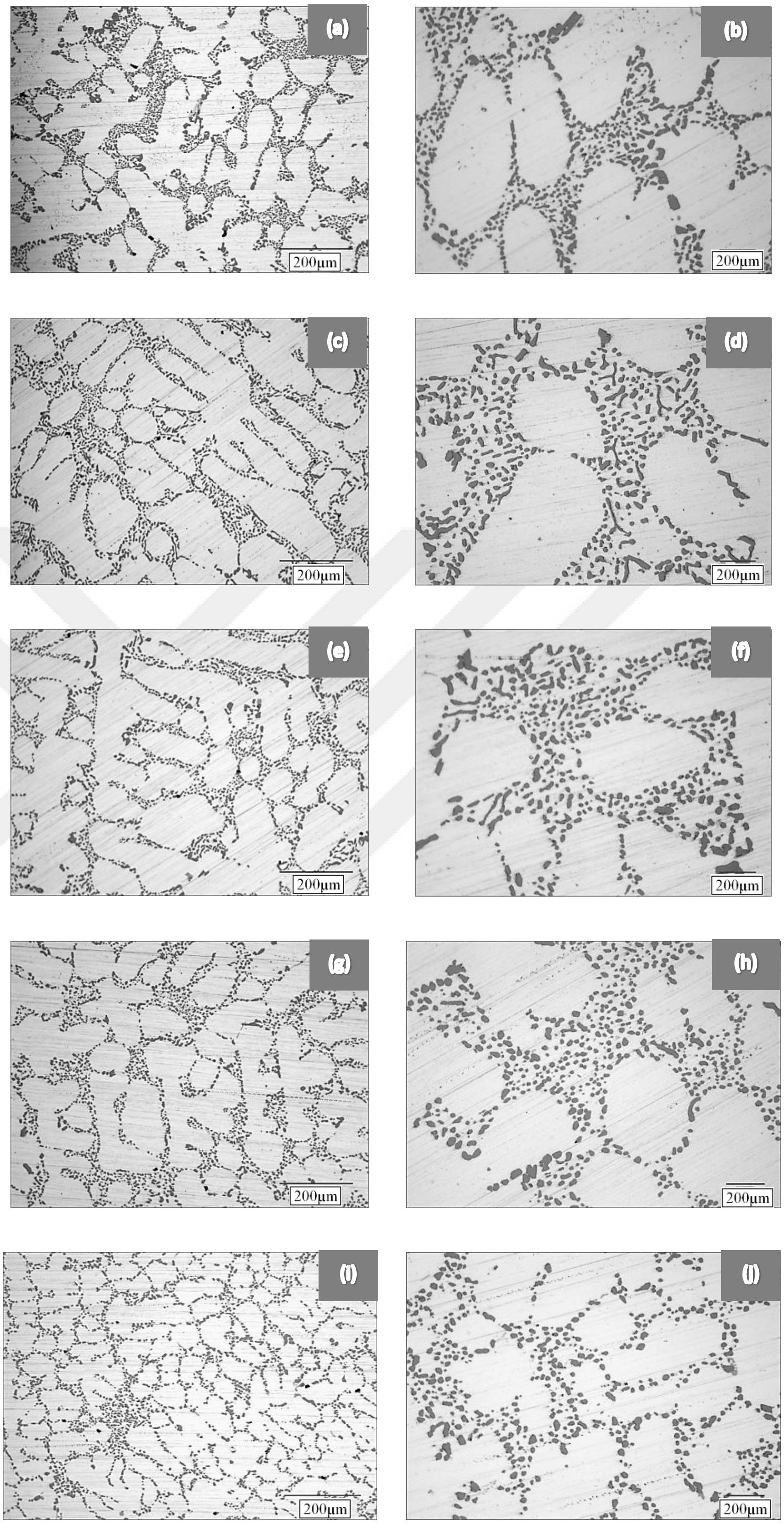


Figure 5.24 Microstructures of solution treated parts at 540°C at 200X and 500X for (a)(b)80 min, (c)(d)160min, (e)(f)200min, (g)(h)240min, (i)(j)280min

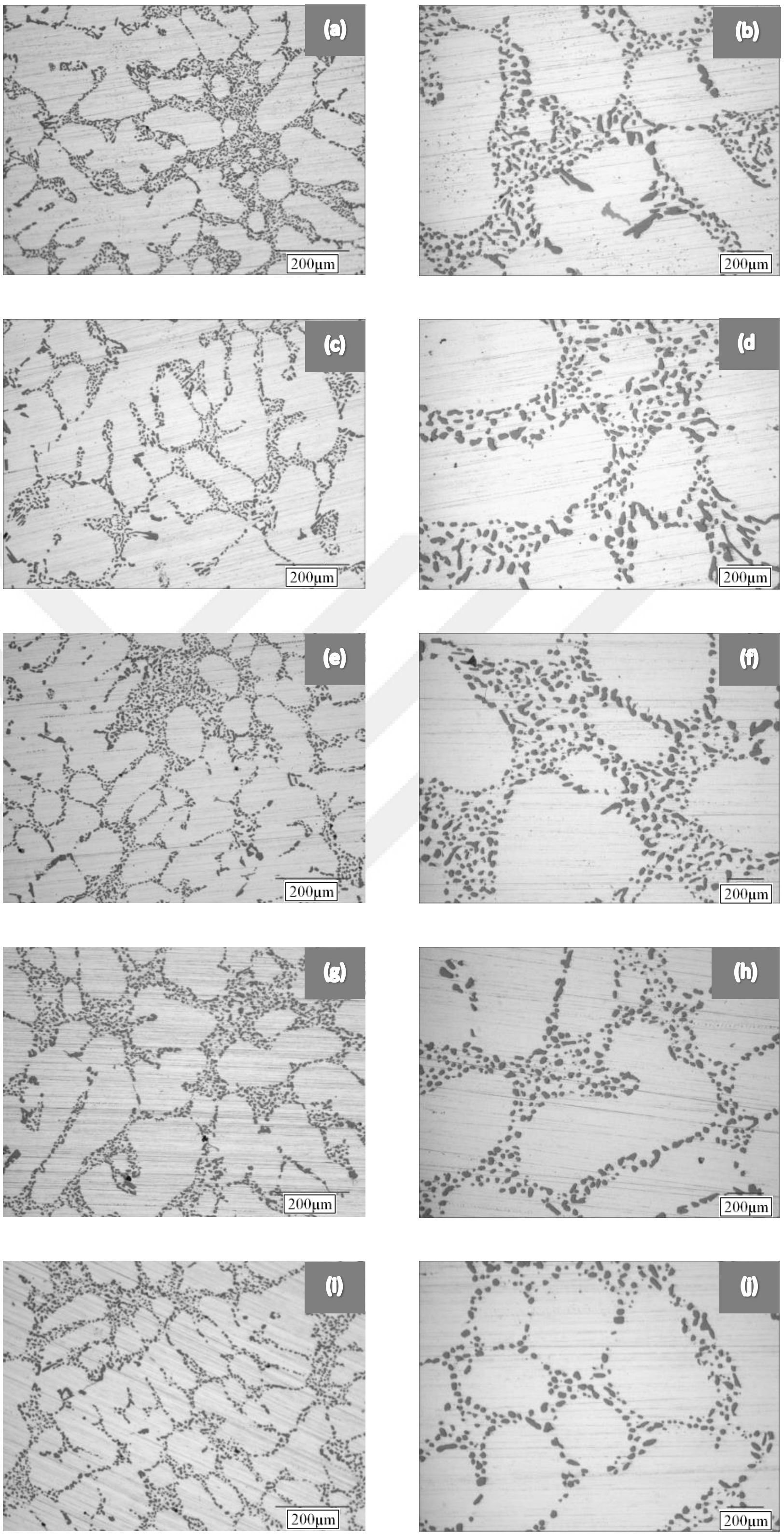


Figure 5.25 Microstructures of solution treated parts at 550°C at 200X and 500X for (a)(b)80 min, (c)(d)160min, (e)(f)200min, (g)(h)240min, (i)(j)280min

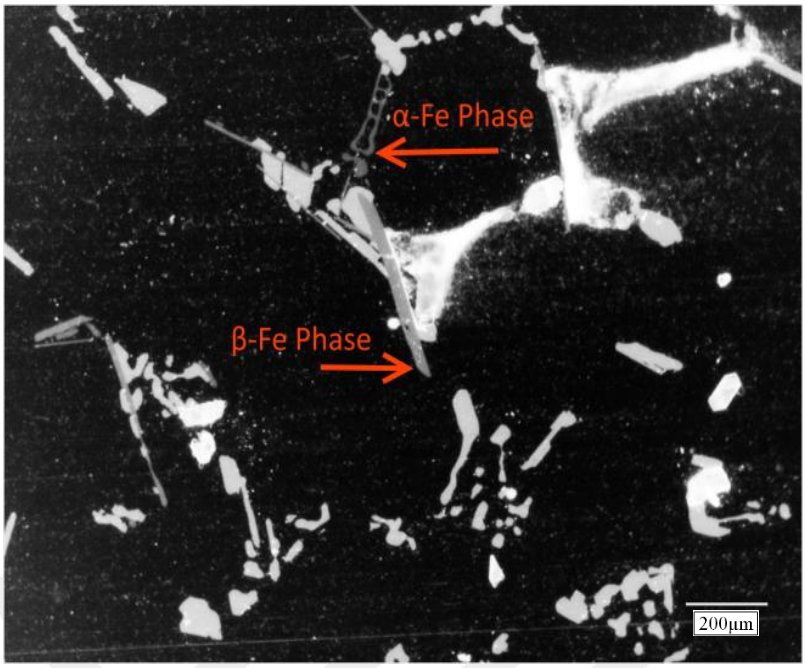


Figure 5.26 Fe-intermetallics in the microstructure of solution treated part at 530°C

Effect of solution treatment temperature and time on eutectic silicon phase formation and homogenization of the microstructure were given in Fig. 5.21-26. Spheroidization can be seen even at 510°C however degree of roundness increased gradually with the increasing temperature. At 540°C and 550°C silicon phases become fully rounded and larger.

The microstructure is more affected by time change than the solution treatment temperature variation. Intermetallics, plate shape Mg_2Si phases and half-rounded/rounded eutectic silicon phase were detected in the microstructure of solution treated sample at 510°C for 80 min. However, longer time as 240-280 min was appropriate to change the silicon morphology and diffuse intermetallic phases totally (Fig. 5.27).

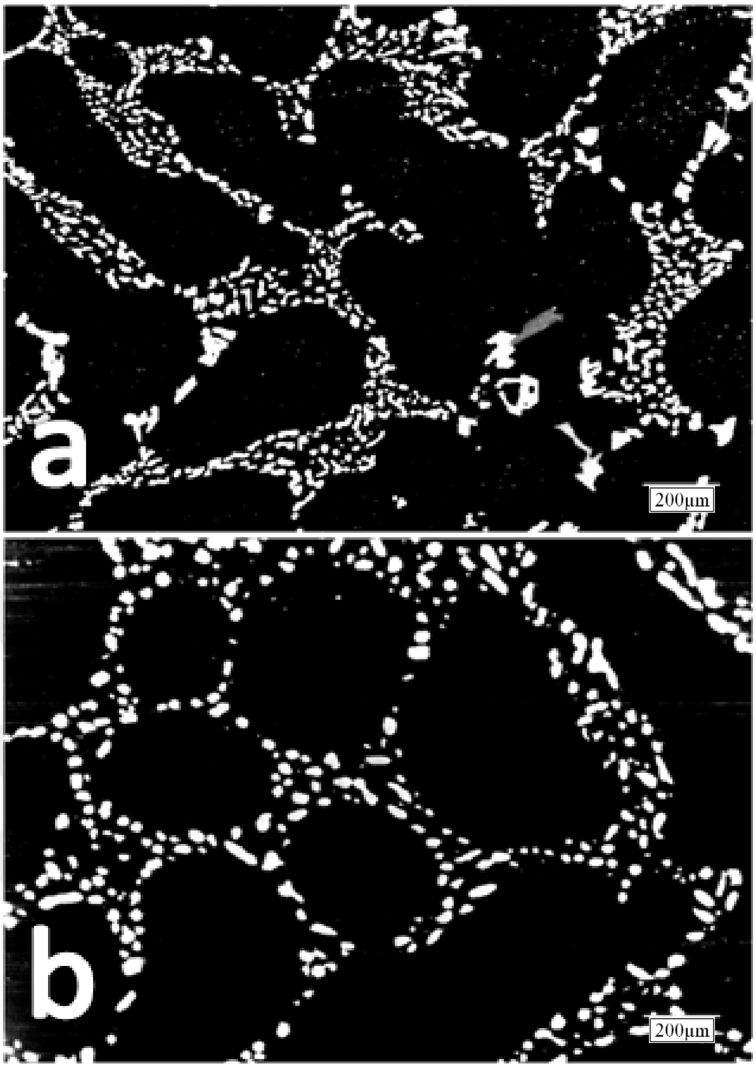


Figure 5.27 Microstructure of the Sr-modified A356 alloy samples after (a) 510°C for 80 min and (b) 510°C for 280 min solution treatment

CHAPTER SIX
CONCLUSION

In this thesis, effect of;

- Each of current heat treatment and painting stages on mechanical properties,
- Time and temperature of solution treatment on mechanical properties and microstructure,
- Different solution treatment and artificial aging conditions on mechanical properties,
- Quenching medium on cooling rate mechanical properties

of Sr modified-A356 low pressure die cast wheels was investigated and the main finding can be summarized as follow.

According to current situation analysis;

- Depending on the desired mechanical properties, different stages of the heat treatment can be applied to as-cast parts separately or in combination. The yield-ultimate tensile strength and hardness were enhanced by applying artificially aging and painting without solution treatment however desired elongation values were not obtained.

- It was shown that elongation predominantly depends on solution treatment and increases the elongation up to 40% due to the spheroidization of eutectic silicon and homogenization of microstructure. Besides that intermetallic phases that exist in microstructure because of low solidification rate, decreases elongation sharply.

- Solution treatment has no effect on yield-ultimate tensile strength and hardness by oneself. Whereas it was determined that values vary considerably when the same aging or painting process applied after different solution treatments.

- Artificially aging increases the yield strength respectively 46% and 23%, likewise, it hardened the material respectively 27% and 8%, with and without solution treatment.

- Painting increases the yield strength respectively 22% and 8%, likewise, it hardened the material respectively 12% and 4%, before and after T6 treatment.

According to solution treatment experiments and optimization plots;

- 510°C is quite enough to change the microstructure and after only solution treatment, same mechanical properties were obtained at 510°C for 80 min (Fig. 6.1). On the other hand, in microstructure, it was detected half rounded and lamellar eutectic silicons.

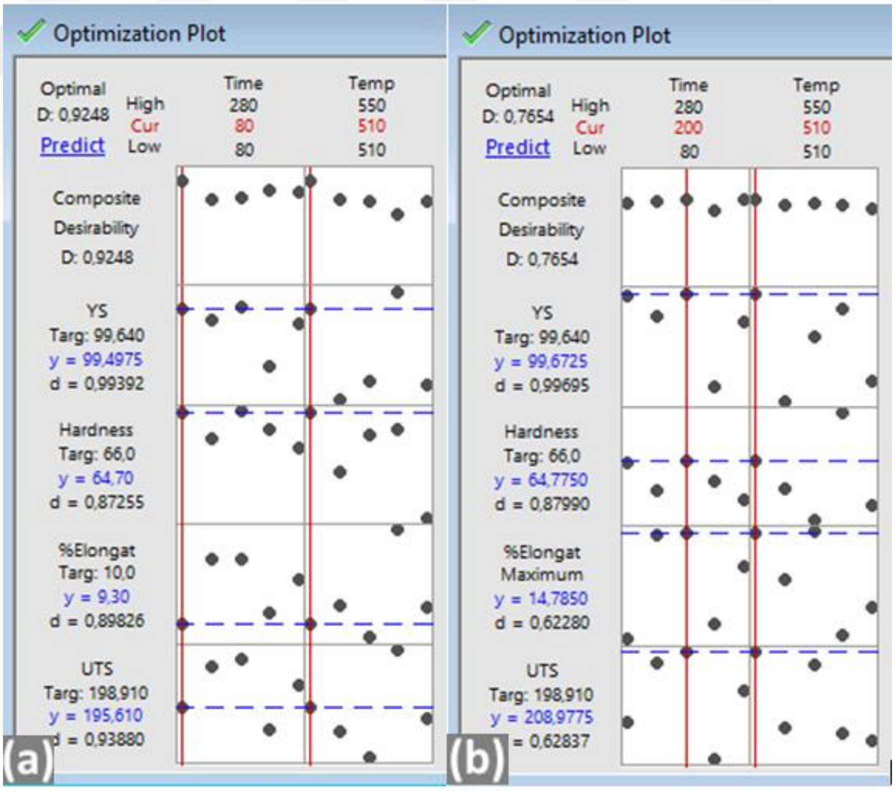


Figure 6.1 Optimization plot for (a) target elongation, (b) maximum elongation

According to optimization experiments;

- As a result of multiple regression analysis, formulas of mechanical properties related to heat treatment variables were formed.

- It was understood that 480°C is not sufficient to provide supersaturated solution for catalyzing the precipitation hardening and changing the morphology of acicular eutectic silicons.

- Although 510°C and 80 or 200 minutes solution treatment seems to be enough to obtain same mechanical properties (comparing only solution treated parts) and low solution treatment temperature causes more elongation, it was found that after the artificial aging, desired yield strength and hardness cannot be obtained at 510°C and temperature must be increased. (Mechanical properties of final product, excluding paint process, was accepted as 155MPa YS, 4.5 ε%, 75HB)

- Also it was found that solution treatment temperature must be least 516°C in order to obtain desired YS value (155MPa) and must be least 522°C in order to obtain desired HB value (75HB) after T6 treatment.

- Alternative heat treatment parameters were calculated and given in prediction reports.

According to quenching experiments;

- Cooling rate and leidenfrost points were determined for the first time for A356 alloy at water-surfactant quenchant medium.

- It was found that maximum cooling rate values of surfactant quenchants are between the water and polymer quenchants and calculated as 61-70°C/s. Maximum cooling rate was observed in water as 85°C/s.

- Critical cooling rate was determined as 61°C/s.

- Leidenfrost points of water and surfactant quenchants were determined and it was found that after reaching this point cooling rate increases dramatically in water. However, in surfactant quenhing medium, cooling rate increased after decomposition of surfactant layer.

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