

ANALYSIS OF MAGNESIUM ADDITION, HYDROGEN POROSITY AND T6
HEAT TREATMENT EFFECTS ON MECHANICAL AND
MICROSTRUCTURAL PROPERTIES OF PRESSURE DIE CAST 7075
ALUMINUM ALLOY

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MICROSTRUCTURAL PROPERTIES OF PRESSURE DIE CAST 7075
ALUMINUM ALLOY**

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I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these declare and conduct, I have fully cited and referenced all material and results that are not original to this work.

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ABSTRACT

ANALYSIS OF MAGNESIUM ADDITION, HYDROGEN POROSITY AND T6 HEAT TREATMENT EFFECTS ON MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF PRESSURE DIE CAST 7075 ALUMINUM ALLOY

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Aluminum alloys are having more attention due to their high specific stiffness and processing advantages. 7075 aluminum alloy is a wrought composition aluminum alloy in the Al-Zn-Mg-Cu series. Due to the significant addition of these alloying elements, 7075 has higher strength compared to all other aluminum alloys and effective precipitation hardenability characteristic.

On the other hand, aluminum alloys have some drawbacks, which hinder the widespread application of them. One of the most commonly encountered defects in aluminum alloys is the hydrogen porosity. Additionally, in case of 7075, another

problem is the lack of fluidity. Magnesium addition is thought to be effective in compensating this deficiency. Accordingly, in this study, die cast 7075 aluminum alloy samples with hydrogen porosity and additional magnesium content were investigated. The aim was to determine the relationship between hydrogen content and hydrogen porosity, and the effects of hydrogen porosity, additional magnesium and T6 heat treatment on ultimate tensile and flexural strength properties of pressure die cast 7075 aluminum alloy.

7075 aluminum alloy returns were supplied from a local pressure die casting company. After spectral analysis, pressure die casting was conducted at two stages. In the first stage, 7075 aluminum alloy with an increase in magnesium concentration was melted and secondly 7075 aluminum alloy was cast directly without any alloying addition. While making those castings, hydrogen content was measured continuously before each casting operation. As a final operation T6 heat treatment is carried out for certain samples. Finally, in order to accomplish our aim, mechanical and microstructural examination tests were conducted.

Keywords : 7075, tensile strength, flexural strength, hydrogen porosity, T6 heat treatment, pressure die casting.

ÖZ

BASINÇLI DÖKÜM YÖNTEMİYLE ÜRETİLMİŞ 7075 ALÜMİNYUM ALAŞIMININ MEKANİK VE MİKROYAPISAL ÖZELLİKLERİNE MAGNEZYUM ARTIŞININ, HİDROJEN POROZİTESİNİN VE T6 ISIL İŞLEMİNİN ETKİLERİNİN ANALİZİ

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Alüminyum alaşımları, düşük yoğunluğun yanı sıra sahip olduğu işlenebilirlik gibi özellikleri nedeniyle büyük önem arz eder. 7075 alüminyum alaşımı Al-Zn-Mg-Cu grubunda yer alan bir dövme alaşımıdır. Bu elementlerin alaşıma katılmış olmaları, 7075 alüminyum alaşımına diğer tüm alüminyum alaşımlarından daha yüksek mukavemet ve etkin bir çökelti sertleştirilebilme özelliği kazandırmıştır.

Öte yandan, alüminyum alaşımlarının uygulamalarda yaygın olarak kullanılmasına engel teşkil eden bazı hususlar mevcuttur. Alüminyum alaşımlarında en sık karşılaşılan problemlerden bir tanesi, hidrojen poroziteleridir. Buna ek olarak, 7075

alaşımındaki bir durum da yeterince akışkan olmamaktır. Magnezyum artışının, bu eksikliği telafi edeceği düşünülmektedir. Buna göre, bu çalışmada, hidrojen porozitesi ve fazladan magnezyum içeren basınçlı kalıp döküm yöntemi ile üretilmiş, 7075 alüminyum alaşım numuneleri incelenmiştir. Amacımız, hidrojen miktarı ile hidrojen porozitesi arasındaki ilişkiyi ve hidrojen porozitesi, magnezyum artışı ve T6 ısıtma işlem uygulamasının basınçlı kalıp döküm yöntemi ile üretilmiş 7075 alüminyum alaşımının çekme ve bükülme dayancı özellikleri üzerine etkilerini belirlemektir.

7075 alüminyum alaşım hurdaları bir basınçlı döküm firmasından temin edilmiştir. Gerekli spektral incelemelerin ardından, basınçlı döküm prosesi iki aşamada gerçekleştirilmiştir. Birinci aşamada, magnezyum konsantrasyonu artırılmış 7075 alüminyum alaşımı dökülmüştür, ikinci aşamada ise herhangi bir alaşım değişikliği yapılmamış 7075 alaşımının dökümü gerçekleştirilmiştir. Bu dökümler yapılırken, her bir döküm öncesi hidrojen miktarı ölçümü gerçekleştirilmiştir. Son bir operasyon olarak da belli numunelere T6 ısıtma işlemi uygulanmıştır.

Anahtar kelimeler : 7075, çekme dayancı, bükülme dayancı, hidrojen porozite, T6 ısıtma işlemi, basınçlı kalıp döküm.

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CHAPTER 1

INTRODUCTION

Among the metallic elements on Earth's crust, Aluminum is the second most abundant one. Aluminum has miscellaneous properties, such as light weight (Density of Al is 2.7 g/cm^3 , approximately one-third of steel (7.83 g/cm^3), copper (8.93 g/cm^3), or brass (8.53 g/cm^3); Density of 7075 is 2.80 g/cm^3 at 20°C and 7075-T6 is 2.79568 g/cm^3), high corrosion resistance, progressive oxidation resistance, good weldability, high fracture tolerance toughness, high age hardening potential and fabricability [1, 2, 3].

Because of the fact that movement of anything requires energy, materials used in the construction of vehicles need to combine good structural strength with high resistance to corrosion, and both they and any containers must be as light as possible in order to promote handling and save fuel. In reference to the properties that were emphasized above, aluminum is a good choice for these kinds of applications especially in aerospace, automotive and energy industry [1, 4, 5].

Although aluminum has advanced properties compared to other non-ferrous alloys, it has low strength as all pure metals; for that reason, it is not possible to use aluminum in pure form in applications where deformation and fracture resistance are necessary. In the competitive environment that we live, deficient properties of aluminum are tried to be improved continuously. Although they tend to diminish corrosion resistance, two main methods to improve the mechanical strength of aluminum are cold working and alloying. As a property improvement endeavor,

alloying is done by adding elements, principally copper, magnesium, silicon, manganese, and zinc into pure aluminum. While designing aluminum alloys, the primary objective is to improve strength, hardness, and resistance to wear, creep, stress relaxation, or fatigue. Improvement in those properties depends on the type of the alloying elements, amount of the alloying, alloy phase diagrams, solidification microstructure, heat treatment and manufacturing processes. [5, 7]

Since alloying elements have different characteristics, they provide different properties for the established alloy. Accordingly, properties obtained by the factors listed above changes with respect to the alloy chemical composition. For instance, alloying determines whether an alloy is heat-treatable or not and the effects of the factors emphasized above highly depends on whether the alloy is a heat-treatable or a non-heat-treatable alloy [1].

Aluminum alloys are mainly classified in two groups according to the alloying: Wrought and casting compositions. In this study, wrought composition type 7075 aluminum alloy, which was developed during Second World War to be used in air forces and stands in Al-Zn-Mg-Cu group, was decided to be examined due to its advanced properties such as high strength and heat-treatability compared to other aluminum alloys. [1, 8]

Strengthening mechanism in aluminum alloys is generally satisfied by either strain hardening as a result of cold working or with heat treatment, or both. Since 7075 is a heat-treatable alloy and cold working is a more troublesome process compared to heat treatment owing to the great forces and more powerful equipments that are required and more control on undesirable residual stresses that occur during cold working; heat treatment is a more preferential process, which is applied to acquire increase in strength of 7075 alloys.

As it is mentioned previously, 7075 has advanced properties, especially when T6 heat treatment is applied. However, because of low castability characteristic that it has, due to its low silicon content, 7075 alloy products are used in wrought form instead of as-cast. In these days, in addition to studies that are carried out to increase strength, some other studies are also conducted to make possible the application of 7075 in as-cast form, which may increase productivity rate. In 7075 aluminum alloy, silicon content is low; therefore, magnesium may increase fluidity and decrease surface energy. So, magnesium is added to 7075, yet the effects of this addition should be examined. Moreover, another important fact in aluminum and its alloys is the hydrogen problem, which should also be considered.

Based on these, the scope of this study is the investigation of the relationship between hydrogen content and hydrogen porosity; relationship between hydrogen porosity and ultimate tensile strength; relationship between hydrogen porosity and flexural strength; increased Mg concentration effect on tensile and flexural strength and T6 heat treatment effect on tensile and flexural strength properties of pressure die cast 7075 components.

In 7xxx alloys, zinc is the principle alloying element. Other elements that are high in concentration in this alloy group are magnesium and copper. Al-Zn-Mg-Cu combination in these alloys, provide the highest strength values of all aluminum alloys. Alloys in this series are strong heat-treatable alloys. They can be further strengthened by heat treatment through the combination of magnesium and zinc. Due to its high strength, this group of alloys is used in high-strength and good corrosion resistance applications such as structural components in aircrafts, buildings, military and nuclear applications [1, 9].

One of the most important alloys due to its advanced properties in this 7xxx series alloys is 7075. The chemical composition of 7075 aluminum alloy as weight percentage values is presented in Table 1 [1].

Table 1. The Chemical Composition of 7075 as Weight Percentage [1].

Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Unspecified other elements		Al minimum
								each	total	
0.40 max	0.50 max	1.2- 2.0	0.30 max	2.1- 2.9	0.18- 0.28	5.1- 6.1	0.20 max	0.05 max	0.15 max	Bal.

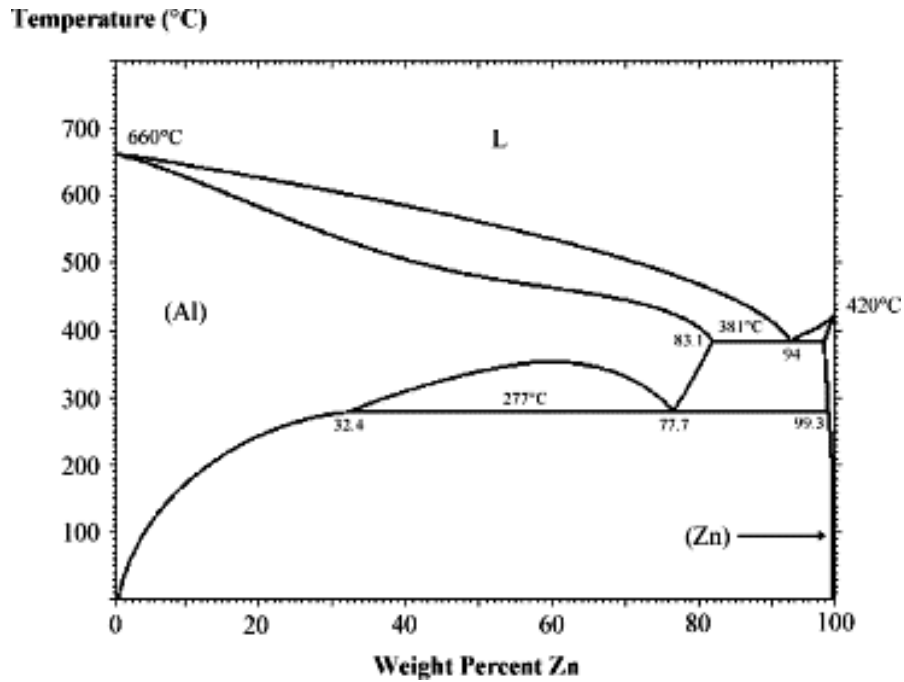


Figure 2. The Binary Phase Diagram of Al-Zn Alloy [1].

Al-Zn binary phase diagram can provide sufficient insight to understand phase equilibria for 7075, which is presented in Figure 2; but, due to the reason that there is magnesium in its chemical composition at a considerable amount, it may be more suitable to use the Al-Mg-Zn ternary phase diagram. The liquidus projection and solidus projection for Al-Mg-Zn ternary system are presented in Figure 3 and Figure 4, respectively [1, 10].

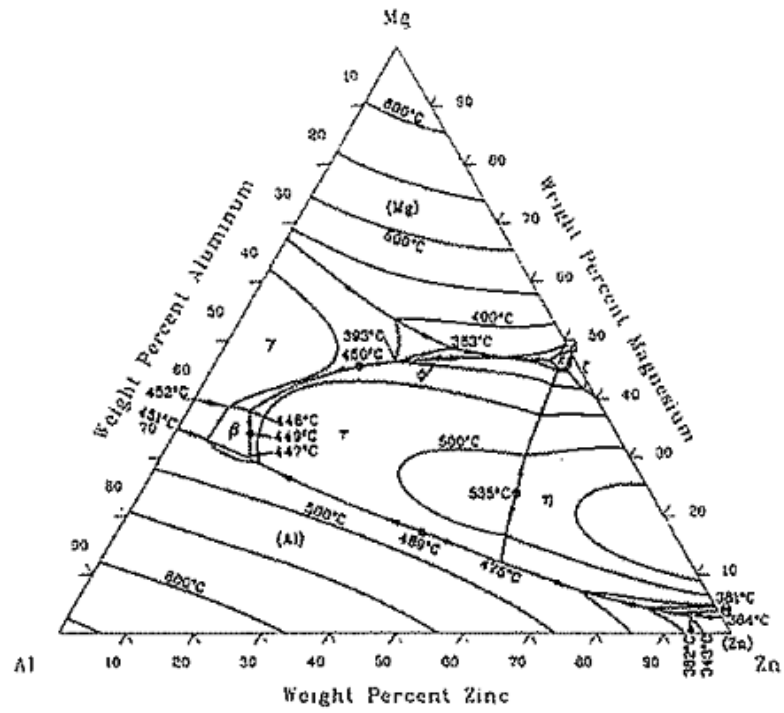


Figure 3. Al- Mg-Zn Ternary Phase Diagram, Liquidus Projection [1].

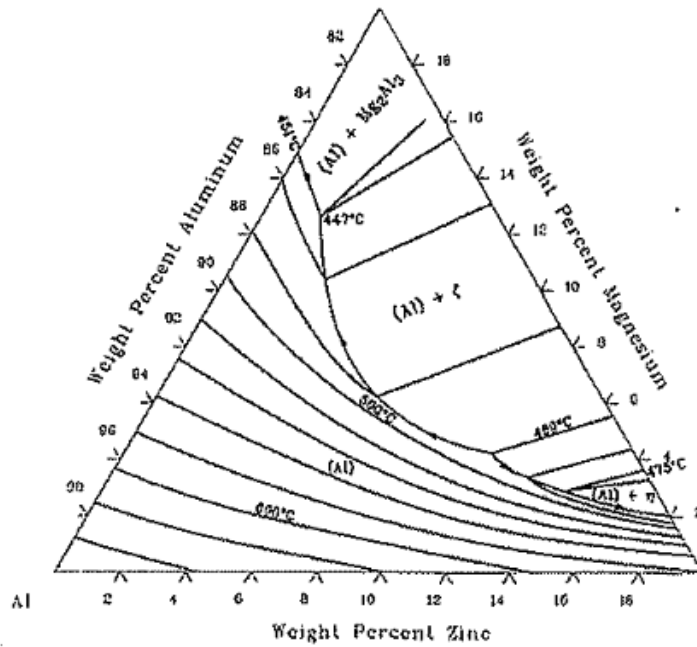


Figure 4. Al- Mg-Zn Ternary Phase Diagram, Solidus Projection [1].

According to those ternary phase diagram figures, liquidus temperature for 7075 is 635°C and solidus temperature, which is the eutectic temperature, is 477°C [1].

2.2. Alloying and Impurity Effects

The main reason of alloying in aluminum is to increase strength, but it should not exceed the limit of 15 wt%, to avoid occurrence of brittle characteristic, which destroys engineering value [11]. Additionally, other effects of alloying should also be considered, in order to obtain optimum properties that we seek.

When alloying elements in 7075 aluminum alloy are thought, we see that zinc is the one that is found in highest concentration. Following zinc, we have magnesium, chromium, manganese and silicon as the alloying elements. The alloying effects of these elements are described below.

Zinc is the primary alloying element in 7075 alloy. Zinc is more fluid than aluminum. Although aluminum-zinc alloy was known for years, it was unable to be used widespread due to its stress corrosion cracking tendency and hot cracking characteristic of the castings. In order to lessen this deficiency, certain additional alloying modifications are applied and highest tensile properties of all wrought aluminum alloys are achieved [1, 11].

Another alloying element in 7075 aluminum alloy is magnesium. Mg in 0.5-6 wt%, provide increase in strength in the solid solution for alloys as the ones in aluminum-

magnesium (5xxx) series. This strengthening is due to the restriction of the dislocation motion. Aluminum has face-centered cubic (FCC) structure. Dislocations are not stable in the FCC structures; eventually, they split into halves to decrease the elastic strain energy of the crystal, which forms the stacking fault region between those halves. There also formed hexagonal close-packed (HCP) structure at both sides of this region. Solute atoms, such as magnesium, which has HCP structure, migrate to these stacking faults to decrease their elastic strain energy (strain-age). This accumulation decreases the energy of faults, so in this situation, more energy is required to move dislocations. This situation is called "Suzuki Locking" and the concept is the solid solution strengthening. This immobilisation of dislocations by solute atoms increases work hardening at low strains, while decreasing work hardening at large strains. Magnesium has 17.4 wt% maximum solid solubility in aluminum, yet level of it is kept below 5.5 wt% for wrought alloys, because magnesium forms precipitates as Mg_5Al_3 or Mg_5Al_8 at grain boundaries, which causes intergranular cracking and stress corrosion cracking susceptibility. Another deficiency that occurs when Mg concentration exceeds 6 wt% is the decrease in fluidity and castability problems. In properly fabricated wrought alloys with up to 5 wt% Mg, magnesium addition increases strength without decreasing ductility [1, 6, 11].

In the wrought aluminum-zinc alloys, magnesium together with zinc (3-7.5 wt%) forms $MgZn_2$, which produce a response to heat treatment and increase in tensile strength. The addition of magnesium more than that is required to form $MgZn_2$ augments tensile strength more [1].

Heat treatable Al-Zn-Mg alloys have good formability and corrosion resistance. Copper addition to them raises their strength significantly. Those Al-Zn-Mg alloys also contain chromium and manganese each up to 0.30 per cent. These additions also improve properties; for instance, aluminum-zinc-magnesium-copper system alloys have highest tensile and yield strength values (600-700 MPa). Zinc and

magnesium control the aging process in Al-Zn-Mg alloys. Copper increases the degree of supersaturation and increases aging rate, which may be as a result of nucleation of the CuMgAl_2 phase. Another effect of copper is to augment quench sensitivity upon heat treatment. Additionally, in this alloy system, chromium and zirconium, in minor amounts, have significant effect on mechanical properties and corrosion resistance [1, 12].

Chromium is a common addition to aluminum-magnesium-zinc alloys, but in this addition, the value of 0.35 wt% is not exceeded. Otherwise, chromium forms coarse constituents with other impurities or additions such as manganese, and iron. The limiting value for the chromium is reduced, in accordance with the increase in the transition metal content. In casting alloys, peritectic precipitation causes the formation of sludge due to excess chromium and this sludge formation can be indicated as the value exceeds the value of 1.8 max calculated according to the formula: $\% \text{Fe} + 2x \% \text{Mn} + 3x \% \text{Cr} = 1.80$. In wrought products, due to slow diffusion rate, chromium forms finely dispersed phases, which obstruct nucleation and grain growth. So, chromium is used to control grain structure in Al-Mg alloys and recrystallization in Al-Mg-Si or Al-Zn alloys during hot working or heat treatment. Chromium, which is a finely dispersed phase in solid solution, also has a positive effect on increasing strength. However, an opposite effect of chromium is that, it increases quench sensitivity in heat-treatable alloys, when the hardening phase precipitates on the pre-existing chromium-phase particles [1, 6, 11].

As an alloying element, silicon is contained with magnesium up to 1.5 wt% each in wrought alloys of the 6xxx group to satisfy the formation of Mg_2Si , which has a maximum solubility limit of 1.85 wt%. During age hardening, precipitation of silicon occurs by forming G-P zones and a very fine precipitate, which provide an increase in strength. But excess magnesium, more than the necessary amount for the formation of Mg_2Si , reduces solid solubility of this compound [1].

In wrought alloys, in work hardened condition, magnesium-manganese system has high strength. If the amount of either magnesium or manganese increases, fabricability decreases [1].

Chromium, zirconium and manganese, singly or in combination, in controlled small proportions, are added into alloys and they are precipitated as finely distributed particles (dispersoids) by high temperature solid-state heating during ingot preheating to control grain size and recrystallization behavior in heat treatment and fabrication. Grain refinement of aluminium alloys occurs by heterogeneous nucleation and growth of grains. When the solute effect is considered, it can be understood that even though nucleating effects in grain refinement are important, solute effects are vital in refinement. Grain size refinement improves formability significantly [13, 14].

Furthermore, there are also impurities that exist in the structure, in addition to alloying elements. The most common impurity in aluminum is iron. Because, although it has high solubility in molten aluminum, which provides it an opportunity to be dissolved at all molten stages of production, its solid solubility is approximately 0.05 wt%, which is very low. Accordingly, iron that is present in aluminum exceeding that value, combines with aluminum or other elements to form an intermetallic second phase. Another important point about iron is that although it is considered as an impurity, it increases strength, hardness and reduces hot cracking tendency when its concentration is below 1.7 wt% [1, 11].

Another common impurity in aluminum is manganese with a concentration of 5 to 50 ppm. Manganese has augmentative effect either as a finely precipitated intermetallic phase or in solid solution. Although, when normal impurities are present

in aluminum, manganese shows limited solid solubility, it remains in solution while chill casting. As a result, majority of added manganese is preserved in the solution. Furthermore, instead of being an impurity, manganese can also be added as an alloying element, which will provide increase in strength and control on the grain structure. In addition, in heat-treatable alloys, precipitation of manganese augments quench sensitivity. Moreover, the combined effect of manganese, iron, chromium, and other transition metals have to be limited, in order to avoid the precipitation of large primary intermetallic crystals [1].

2.3. Hydrogen in Aluminum

The most soluble gas in aluminum and its alloys is the hydrogen gas.. Hydrogen is produced by the reduction of water vapor in the atmosphere by aluminum and by the decomposition of hydrocarbons. When a metal is in contact with hydrogen or involved in hydrogen-producing environment such as electroplating, pickling, casting, corrosion reactions, cathodic protection and fuel cell reactions, hydrogen can be absorbed easily into metal. This absorption of hydrogen into metal occurs in a more detrimental way in aluminum alloys [1, 15, 16].

Hydrogen pickup in both solid and liquid aluminum is enhanced by the presence of certain impurities, such as sulfur compounds, on the surface and in the atmosphere. Hydride-forming elements in the metal increase the pickup of hydrogen in the liquid. Other elements, such as beryllium, copper, tin, and silicon, decrease hydrogen pickup [16, 17].

In aluminum alloys, since hydrogen has higher solubility in the liquid aluminum alloy than in the solid, gas porosity can occur during solidification. The difference between hydrogen solubilities can be explained more clearly by emphasizing that hydrogen solubility in aluminum near the melting point is at 10^{-6} and decreases with temperature rapidly. From the high temperature side, hydrogen content extrapolated is $3.9 \times 10^{-4} \text{ cm}^3$ at room temperature. This is equivalent to the volume of hydrogen gas at 0°C and 1 atm per 100 g aluminum, but hydrogen content measurements for wrought alloys, range from 0.11 to 0.60 cm^3 per 100 g Al, which are 3-4 orders of magnitude larger than the mentioned hydrogen solubility [18].

As it can be observed from the Figure 5, solubility of hydrogen is much higher in liquid than that in the solid state. The exact values for liquid and solid solubilities of hydrogen, above and below the solidus line for pure aluminum, are 0.65 and 0.034 mL/100g, respectively. As alloying takes place, these values deviate slightly. When molten aluminum solidifies, excess hydrogen that is above the solid solubility limit, which was dissolved in liquid prior to solidification, may precipitate in molecular form, leading to the formation of primary and/or secondary voids [1].

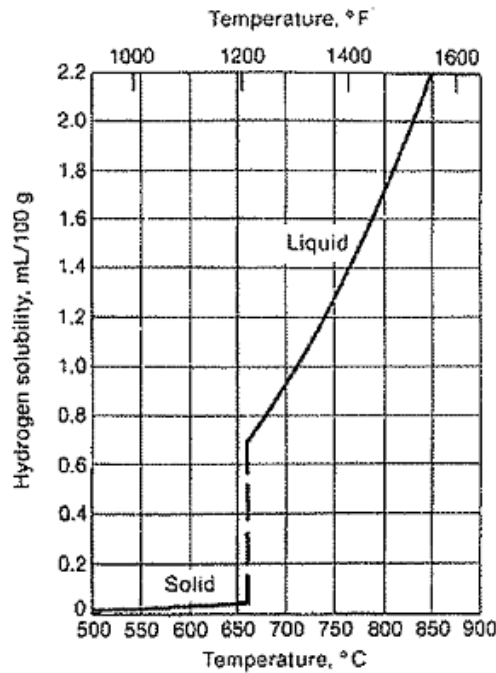


Figure 5. Solubility of Hydrogen in Aluminum at 1 atm Hydrogen Pressure [1].

The solubility of gas in liquid metal can be expressed by Sievert's relation, which states that the concentration of dissolved gas is proportional to the square root of the partial pressure of the gas in the contacting atmosphere. Thus, for hydrogen:

$$[H_{\text{solution}}] = K \{p(H_2)\}^{1/2}$$

The constant K is temperature-dependent. The solubility of gases decreases during the course of freezing, usually quite abruptly, and they are rejected in the form of gas bubbles which may become entrapped within and between the crystals, forming weakening blowholes. It follows from Sievert's relation that reducing the pressure of the contacting atmosphere will reduce the gas content of the melt; this principle is the basis of vacuum melting and vacuum degassing. Similarly, the passage of numerous bubbles of an inert, low-solubility gas through the melt will also favour gas removal. Conversely, freezing under high applied pressure, as in the die casting

process for light alloys, suppresses the precipitation of dissolved gas and produces cast shape of high density [19].

In order to achieve the formation of hydrogen porosity, there should be oxygen in the alloy according to the study of Zuithoff, who showed that pores could nucleate, if the oxygen was high; otherwise, pores might not form in the absence of oxides which facilitate nucleation. In addition to entrained oxide necessity as nucleation site for hydrogen precipitation, surface tension forces and liquid cooling/solidification rates also play an important role in withstanding the formation of hydrogen porosity. Accordingly, when there were no oxide, and the hydrogen concentration was below 0.15mL/100g, hydrogen porosity did not form. For that reason, it is indicated that, when when there is lack of oxide to facilitate nucleation, to achieve hydrogen precipitation, high levels of hydrogen concentration, such as more than 0.30 mL/100g, is required [1, 15].

In addition to causing primary porosity in casting, hydrogen causes secondary porosity, blistering, and high-temperature deterioration (advanced internal gas precipitation) during heat treating. Hydrogen gas in the molten alloy is controlled by either hydrogen-free gase fluxing or vacuum degassing [1, 20].

2.3.1. Hydrogen Sources

The potential sources for hydrogen in aluminum can be listed as following:

- Charge materials, such as foundry returns, ingot and scrap.
- Fluxes.

- External components, such as furnace tools, furnace refractories, sampling ladles etc.
- Melt/mold reactions, which occurs when metal flow is extremely turbulent during the casting process [1].

2.3.2. Hydrogen Porosity

The two types of hydrogen porosity that may occur during casting of aluminum are interdendritic and secondary porosity. Interdendritic porosity is more important compared to the secondary one. The interdendritic porosity occurs when rejected hydrogen at the solidification front in course of solidification causes a solution pressure which is higher than atmospheric, due to sufficiently high hydrogen content. On the other hand, secondary (micron-size) porosity takes place if dissolved hydrogen content is low [1].

Besides, hydrogen porosity may also be beneficial if it is finely distributed structure. In that case, it can change the distribution and shape of shrinkage porosity, which is usually more detrimental in casting [1].

Nonetheless, hydrogen porosity generally has a negative effect on mechanical properties of aluminum alloys. Figure 6 presents hydrogen content and porosity relationship for sand-cast aluminum and various aluminum alloys. As can be distinguished from this figure, the degree of hydrogen content effect on hydrogen porosity differs with respect to the alloy. Following, the effect of this porosity on the ultimate tensile strength and yield strength of miscellaneous alloys are presented in Figure 7 and Figure 8 [1].

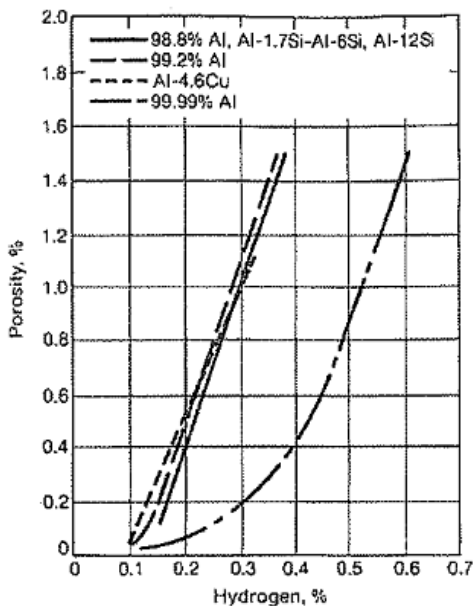


Figure 6. Porosity as a Function of Hydrogen Content in Sand-Cast Aluminum and Aluminum Alloy Bars [1].

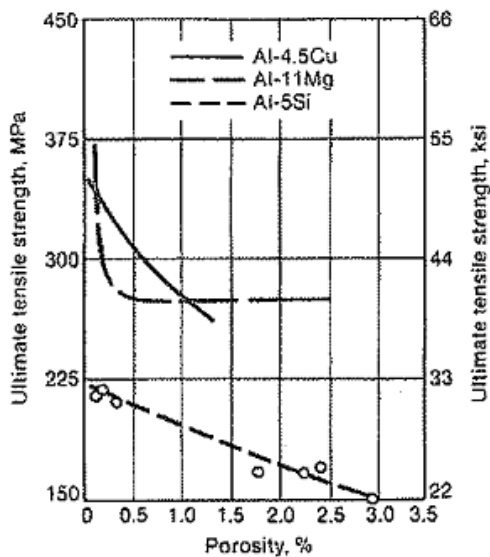


Figure 7. Ultimate Tensile Strength versus Hydrogen Porosity for Sand-Cast Bars of Three Aluminum Alloys [1].

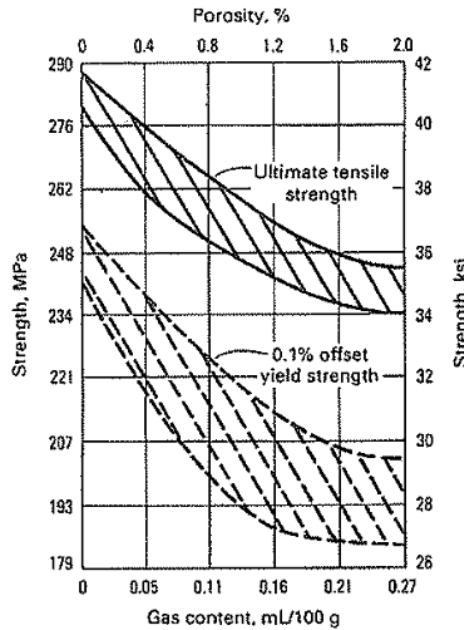


Figure 8. Influence of Gas Content on the Tensile and Yield Strengths of Aluminum Alloy 356 [1].

2.3.3. Hydrogen in Solid Solution

The level of hydrogen that is dissolved and solidification conditions determine the tendency of hydrogen in a solidified structure. Hydrogen porosity is formed by diffusion-controlled nucleation and growth. Consequently, if there occurs a diminishment in the hydrogen concentration and an augmentation in the rate of solidification, the formation and growth of porosity can be ceased. Accordingly, it can be interpreted that lower values of hydrogen porosity can be attained in pressure die casting process which involves high solidification rate [1].

2.3.4. Hydrogen Removal (Degassing)

Since hydrogen porosity is a defect that should be avoided in order to obtain better products, certain methods were developed to decrease the level of hydrogen, which is dissolved in the molten metal. These methods can be listed as following:

- Holding the metal at a lower temperature at a lower hydrogen solubility limit and satisfy natural outgassing.
- Gas purging or gas lushing.
- Tableted flux degassing.
- Mechanical mixer degassing [1].

2.4. Die Casting

In casting processes, a solid material is first melted, heated to proper temperature, and sometimes treated to modify its chemical composition. The molten material, generally metal, is then poured into a cavity or mold that contains it in the desired shape during solidification. Thus, in a single step, simple or complex shapes can be made from any material that can be melted. The resulting product can have virtually any configuration the designer desires. In addition, the resistance to working stresses can be optimized, directional properties can be controlled, and a pleasing appearance can be produced. Six basic requirements that are associated with most casting processes are: Mold cavity, melting process, pouring technique, solidification process, mold removal and finishing operations [21, 22].

Die casting is used in manufacturing at almost every field, such as automobiles, aircraft, agricultural equipment, building hardware, home appliances, computer hardware etc. Die casting is the fastest manufacturing process to produce precise nonferrous castings in which liquid metal is injected into reusable steel molds by high velocity and at high pressure in order to produce metal parts with accurate dimension and smooth or textured surface. High velocity provides a high turbulent flow [11].

Die casting involves steel dies with two halves that has cavities to produce the casting. They consist of mechanical features as a metal flow system and a thermal system [11].

While dealing with die casting, some assumptions are made. Those are: [11]

- Casting alloy behaves like a hydraulic fluid during cavity fill due to superheated temperature that it has.
- During cavity fill, casting metal follows the path with least resistance.
- Die casting is a turbulent process.

Three basic factors that affect final products in die casting are: [11]

- The thermal behavior of the casting alloy,
- The shot end of the casting machine and the shot sleeve,
- The shape of the part that defines the flow path of the liquid metal.

Aluminum alloy castings are routinely produced by pressure die, permanent mold, green and dry sand, investment and plaster casting. Castings are produced by filling molds with molten aluminum and are used for products with intricate contours and hollow or cored areas. The choice of castings over other product forms is often based on net shape considerations. Reinforcing ribs, internal passageways, and complex design features, which would be costly to machine in a part made from a wrought product, can often be cast by appropriate pattern and mold or die design. Premium engineered castings display extreme integrity, close dimensional tolerances, and consistently controlled mechanical properties in the upper range of existing high strength capabilities for selected alloys and tempers [1, 23].

Due to the moderate strength-to-weight ratio and easier casting characteristics, aluminum alloys are the most popular alloys for die casting operations, but because of the abrasiveness of aluminum to the die materials, die life is limited. As a caution, gate speeds have to be decreased to minimize turbulence within the cavity during filling, accordingly, cavity fill time is increased and sometimes quality is accommodated with some sacrifice. Aluminum is always cast in cold chamber to avoid rapid dissolution of iron [11, 24].

The property that distinguishes high pressure die casting (HPDC) from all other foundry processes is rapid solidification rate. The quick transformation from liquid to solid and thus crystallization provides fine grain size, dense structure and superior mechanical properties by diminishing the atomic movement into and out of the crystal structure, which could even occur at the solid state, due to solid solubility [11].

2.4.1. The Die Casting Machine

Aluminum was first tried to be die cast around the early 1900s. Aluminum has higher melting point compared to lead, tin and zinc, which were the only materials that were cast by die casting up to that date. With efforts to die cast aluminum, it was determined that molten aluminum, since it acts as a solvent for iron, corroded the iron and thus steel parts of the casting machines. Additionally, the castings were also contaminated by the iron inclusions. As a result, direct contact of machine plunger with the aluminum alloy during die casting was tried to be restricted and gooseneck machine was invented in 1907. Today, two types of die casting machines are used: cold and hot chamber, which are classified according to the way the molten metal is delivered to the metal feed system of the die. The one that has made possible the die casting of aluminum and brass since 1932 is the cold chamber type. In the cold chamber machines, metal injection system is not immersed into the liquid metal bath [11].

Key components of modern cold chamber die casting machine are presented in Figure 9 and Figure 10 and their names are listed in Table 2 and Table 3, from operator side and top view respectively.

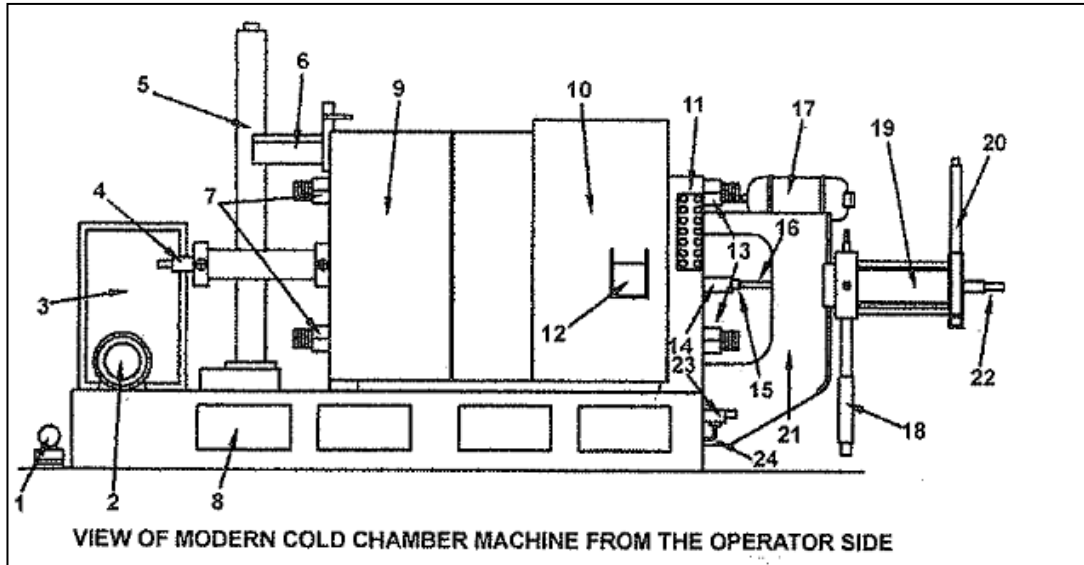


Figure 9. Key Components of Modern Cold Chamber Die Casting Machine from Operator Side [11].

Table 2 Key Components of Modern Cold Chamber Die Casting Machine from Operator Side [11].

Item No	Description of component
1	Heat Exchanger (sometimes referred to as after cooler)
2	Main motor
3	Electrical cabinet
4	Die lock cylinder
5	Die lock accumulator (piston type illustrated)
6	Safety latch mechanism
7	Tie bar nuts
8	Access cover to reservoir (typical)
9	Linkage guard

- 10 Safety gate
- 11 Operator's control station
- 12 Observation window
- 13 Tie bar nuts
- 14 Cold chamber (sometimes referred to as shot sleeve)
- 15 Plunger tip
- 16 Shot arm
- 17 Shot nitrogen accumulator
- 18 Jacking mechanism to change shot end positions (center or below)
- 19 Shot cylinder
- 20 Shot accumulator (piston type)
- 21 "C" frame that supports shot end
- 22 Shot stroke adjustment
- 23 Shot speed control
- 24 Hydraulic return line

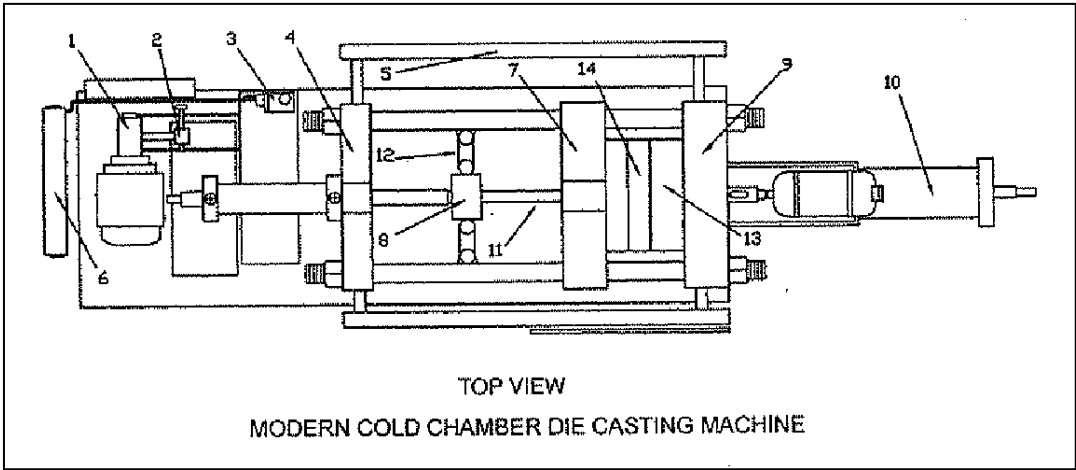


Figure 10. Key Components of Modern Cold Chamber Die Casting Machine from Top View [11].

Table 3. Key Components of Modern Cold Chamber Die Casting Machine from Top View [11].

Item No	Description of component
1	Pumps
2	Suction valve
3	Circulating pump and filter
4	Adjustable platen
5	Helper side safety guard
6	Heat exchanger
7	Moving (ejector) platen
8	Cross head
9	Stationary (cover) platen
10	Shot cylinder

- 11 Cross head guide rods
- 12 Mechanical locking linkage
- 13 Cover die
- 14 Ejector die

The shot sleeve is generally located in horizontal position as shown in Figure 11 to minimize splashing of metal. During casting, metal is ladled into sleeve, plunger advances, seals off the way, and forces the metal into die. After solidification of the casting, die opens, plunger forces biscuit from shot sleeve and casting ejection occurs [11].

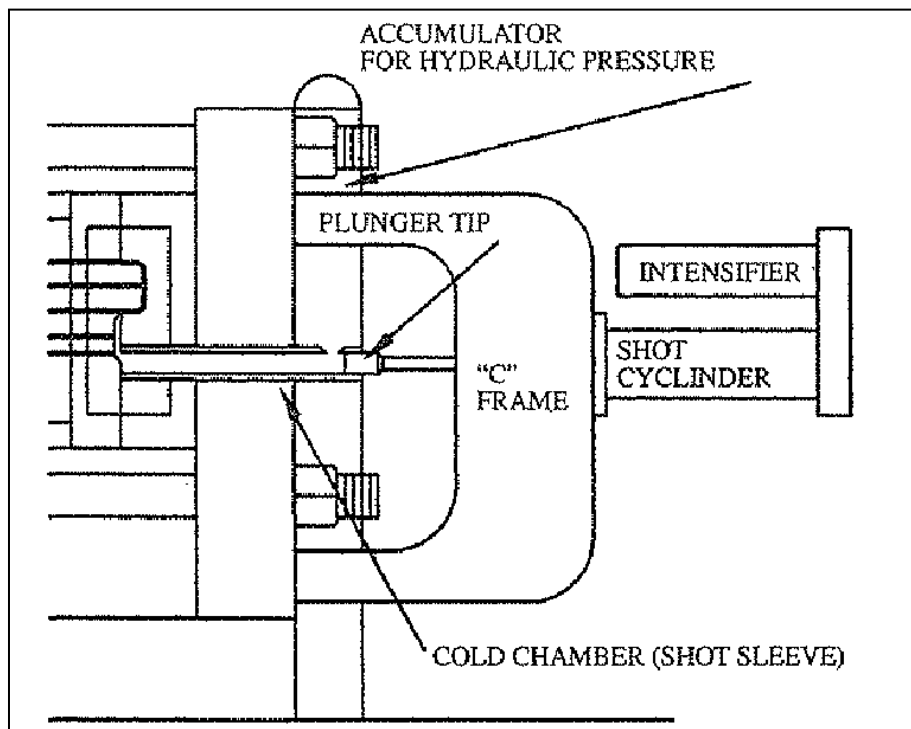


Figure 11. Cold Chamber [11].

In die casting process, die design has an important place. The most important point in this die design is the gating design concept. In gating system design certain specifications about the following issues should be set:

- Elements of the gating system in the die
- Proper set-up of the die casting machine
- Operating parameter for the die casting machine [25].

2.4.2. Casting Properties of Aluminum Alloys

2.4.2.1. Molten Aluminum Processing and Casting

Aluminum and aluminum alloys are melted for conversion into castings. The latent heat of fusion value for melting of aluminum is 95 cal/g. Since this is a large amount, forced convectional heat transfer within the melt, sometimes plays a significant role in facilitating this transition by providing the better mixing of the solid metal with already molten metal; hence, time and energy input are also decreased [1].

Both the nature of the furnace charge and the furnace type are important in metal casting operations. Molten aluminum is prone to three types of degradation even under optimum melting and melt-holding conditions:

- Hydrogen adsorption occurs as time passes at high temperature.
- Oxidation occurs with time at high temperature.

- Transient elements are diminished as time passes at high temperature [1].

These degradation concepts are influenced by turbulence or agitation of the melt and increased holding temperature [1].

Casting soundness determines the mechanical properties of aluminum alloys. During casting operation, one should be careful, because there can occur certain defects such as: cold shut, which is poor fill due to low temperature; soldering, or die sticking, that occurs when molten aluminum sticks to die surface and dimensional distortion. Additionally, there are other defects such as oxides, gas and inclusions that can also be harmful to the products, and which can be prevented by careful melting, fluxing, and handling during transfer. Among those defects, hydrogen porosity and entrained nonmetallic inclusions are the most effective ones and they can be eliminated by treatment before pouring, as explained before in this section. Specifically, common cast defects in 7xxx series aluminum alloys are gas or shrinkage porosity which are mainly due to the wide mushy zone in this series alloys. Although stated methods are applied to produce high-quality metal, defects can still occur inevitably. This situation cannot be prevented, since there is no certain way of following and observing metal quality consistently [1, 11, 26, 27].

2.4.2.2. Solidification Structures of Aluminum Alloy Ingots

2.4.2.2.1. Solidification Time

The amount of heat that must be removed from a casting to cause it to solidify is directly proportional to the amount of superheating and the amount of metal in the casting, or the casting volume. Conversely, the ability to remove heat from a casting is directly related to the amount of exposed surface area through which the heat can be extracted and the environment surrounding the molten material. These observations are reflected in Chvorinov's rule, which states that t_s , the total solidification time, can be computed by: $t_s = B (V/A)^n$ where $n = 1.5$ to 2.0 [22].

The total solidification time, t_s , is the time from pouring to the completion of solidification; V is the volume of the casting; A is the surface area; and B is the mold constant, which incorporates the characteristics of the metal being cast (its density, heat capacity, and heat of fusion), the mold material (its density, thermal conductivity, and heat capacity), the mold thickness, and the amount of superheat [22].

2.4.2.2.2. Dendrites

During solidification, crystals form, crystal lattice is repeated, individual crystals contact, grains are formed, crystal growth progresses, dendrite arms are formed. Dendritic solidification macrostructure is characteristic of all aluminum alloy castings [1, 23, 24].

2.4.2.2.3. Dendrite Arm Spacing

In all commercial processes, solidification takes place through the formation of dendrites in the liquid solution. In casting applications, solidification rate should be investigated carefully in order to obtain optimum mechanical properties. Solidification rate controls the cells, which are contained within the dendrite structure, match with the dimensions that divide primary dendrite arms. In die casting, because of the rapid solidification, this is not critical and due to fine dense grain structure obtained by the restriction of dendritic growth with rapid solidification, advanced properties are achieved, which makes heat treatment operation less required in die castings [1, 11, 23, 24].

2.4.2.2.4. Grain Structure

In aluminum castings, a fine, equiaxed grain structure is wanted to be achieved. Solidification rate, alloy composition, and grain refiner containing intermetallic phase particle addition determines the size and type of the grain [1].

2.5. Effect of Metallurgical Factors on Processing of Aluminum Alloys

2.5.1. Forming

Aluminum materials generally fail either by localized necking or by ductile fracture. Bulk material properties, such as work hardening and strain hardening, control necking. On the other hand, in ductile fracture, microscopic voids nucleate and link at particles. Then, strain concentration takes place in narrow shear bands [1].

Magnesium or copper, in minor quantities, diminish the strain-hardening rate. As a result, the amount of useful diffuse necking, which takes place after the uniform elongation, will be decreased. Furthermore, magnesium addition causes an additional reduction in fracture, and it also encourages the localization of strain into shear bands in solid solution. On the contrary, the significance of zinc on work hardening or necking is low for dilute alloys. Additionally zinc also does not stimulate strain aging.

2.6. Physical Metallurgy in Aluminum Alloys

The physical metallurgy of aluminum alloys is mainly interested in the influences of chemical composition, heat treatment and/or mechanical working on physical and mechanical properties of the alloy. As it was emphasized previously, since aluminum has very low strength in its pure form, which is 10 MPa of tensile yield strength in the annealed condition, the primary objective in aluminum alloy preparation is to increase strength. The two major methods that are used to augment the strength of aluminum alloys are:

- Disseminating elements or second-phase constituents in the solid solution, and following that, applying cold working. This is the general method for non heat-treatable alloys.
- Adding alloying elements, dissolving them in solid solution and precipitating them as coherent submicroscopic particles. This is the common method for heat-treatable or precipitation-hardening alloys.

Zinc, magnesium, copper, manganese and silicon are the major alloying elements that are added into alloys to ensure increase in strength when cold working or heat treatment is applied [1, 6, 21].

2.6.1. Second-Phase Constituents.

Intermetallic phases of the respective binary and ternary systems are rarely isomorphous in quaternary systems, and they form series of solid solutions in equilibrium with aluminum solid solution. For the aluminum-copper-magnesium-zinc quaternary system, there are three such pairs: $\text{MgZn}_2 + \text{CuMgAl}$, $\text{Mg}_2\text{Zn}_{11} + \text{Cu}_6\text{Mg}_2\text{Al}_5$, and $\text{CuMg}_4\text{Al}_6 + \text{Mg}_3\text{Zn}_3\text{Al}_2$ [1].

The three types of second-phase particles that are said to be having an effect on the fatigue and fracture behavior of high-strength aluminum alloys are listed in Table 4 [1, 6].

Table 4. Three Types of Second-Phase Particles.

Type	Size	Typical examples
	μm	
Constituent particles	2-50	Cu_2FeAl_7 , CuAl_2 , FeAl_6
Dispersoid particles	0.01-0.5	ZrAl_3 , $\text{CrMg}_2\text{Al}_{12}$
Strengthening precipitates	0.001-0.5	Guinier-Preston zones

2.6.2. Prediction of Intermetallic Phases in Aluminum Alloys

Predicting the intermetallic phase requires detailed information on both the ratios and total amounts of alloying elements present in the structure, and phase diagrams. However, metastable conditions, which are not shown on the equilibrium diagrams, should also be considered [1].

In cast structure, there are some phases, which are not stable. During subsequent heat treatment processes, those unstable phases dissolve totally or are replaced by another phase. The phases that can form in either a cast or a wrought structure are listed in Table 5. Since the rate of solidification determines the appearance of phases in a cast structure, all of the phases mentioned in Table 5 may not be observed [1, 6].

Table 5. Phases Present in the Al-Cu-Mg-Zn-Fe-Si-Cr Alloy System [1].

Alloy system	Examples of alloy	Alloy form	Phases
Al-Cu-Mg-Zn-Fe-Si-Cr	7075	Ingot	(Fe,Cr)Al ₃ , (Fe,Cr) ₃ SiAl ₁₂ , Mg ₂ Si, Mg(Zn ₂ AlCu), CrAl ₂ (only when chromium content is near high side of range)
		Wrought	(Fe,Cr) ₃ SiAl ₁₂ , Cu ₂ FeAl ₂ , Mg ₂ Si, CuMgAl ₂ , Mg(Zn ₂ AlCu), Cr ₂ Mg ₃ Al ₁₈ (may be identity of fine precipitate which comes out at elevated temperatures; not positively identified)

2.7. Heat Treatment

The term "heat treating" refers to the heating and cooling operations that are performed in order to change the mechanical properties, metallurgical structure or residual stress state of a metal product. In aluminum alloys, the heat treatment term refers to the specific operations, such as solution heat treatment, quenching, and precipitation (age) hardening performed to increase strength and hardness of the precipitation-hardenable wrought and cast alloys. Annealing, a process that reduces strength and hardness while increasing ductility, can also be used for both the non-heat treatable and heat treatable grades of wrought and cast alloys. Precipitation-hardenable wrought and cast alloys usually are referred to as the "heat-treatable" alloys due to the fact that they respond to thermal treatment based on phase solubilities [1, 9].

Heat treatment process has several variables, such as time, temperature and quenching rate. The type of the alloy plays the major role in deciding on the values of these variables, in order to achieve a certain temper. Designations for the common tempers are as following:

- F, As Fabricated.
- O, Annealed.
- H, Strain-Hardened (Wrought Products Only).
- W, Solution Heat Treated.
- T, Solution Heat-Treated [1].

When we concentrate on the solution heat-treatment, which was indicated by "T", we see that specific sequence of T treatments are separated from each other by a number that is written beside the T. These treatment sequences can be summarised as following:

- T1, Cooled from an Elevated-Temperature Shaping Process and Naturally Aged to a 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 Ages 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 and Artificially Aged.

- T7, Solution Heat-Treated and Over-aged or Stabilized.
- T8, Solution Heat-Treated, Cold-Worked, and Artificially Aged.
- T9, Solution Heat-Treated, Artificially Aged, and Cold-Worked.
- T10, Cooled from an Elevated-Temperature Shaping Process, Cold-Worked, and Artificially Aged [1, 28].

Alloys in 7xxx series are solution heat treatable. Alloys in this series can be shaped more easily in the fully annealed O-temper form (7075-O Ultimate Tensile Strength= 228 MPa). On the other hand, they can have ultimate tensile strength values as much as 572 MPa, when higher tempers are applied. Furthermore, cold working can be used to further strengthen them. The significant role of precipitation hardening on the strengthening of aluminum alloys can be realized from Figure 12, in which data for the artificially aged (T6 heat treatment) 7075 is presented. Here it can be observed that although some reduction in elongation occurs, there is an impressive increase in strength because of precipitation hardening [9].

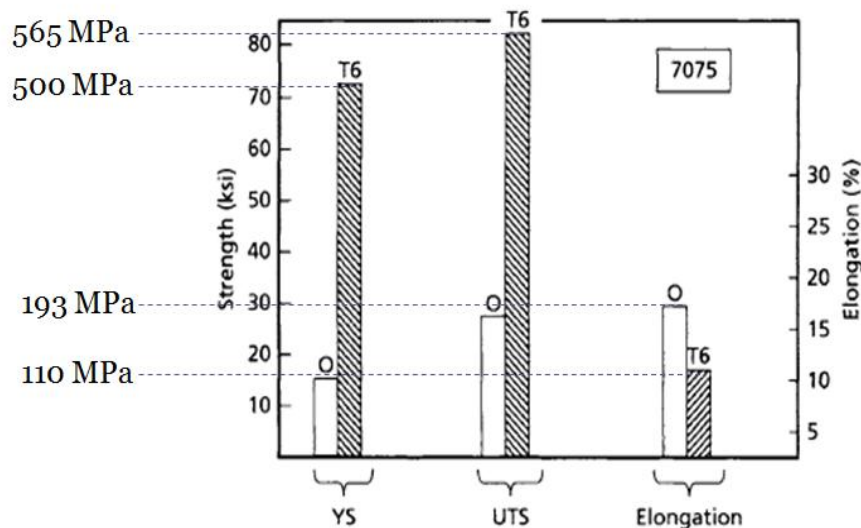


Figure 12. Effect of Heat Treatment on 7075 [9].

For an aluminum alloy to be precipitation hardened, certain conditions must be satisfied. First, heat-treatable (precipitation-hardening) aluminum alloys for wrought and cast products must contain at least one element or compound in a sufficient amount that has a decreasing solid solubility in aluminum with decreasing temperature. In other words, the elements or compounds must have an appreciable solubility at high temperatures and only minimal solubility at lower temperatures. Elements that have this characteristic are copper, zinc, silicon, and magnesium, with compounds such as CuAl_2 , Mg_2Si , and MgZn_2 . However, this feature is not enough. The second requirement is that the element or compound that is put into solution during the solution heat treating operation must be capable of forming a fine precipitate that will produce lattice strains in the aluminum matrix. The precipitation of these elements or compounds progressively hardens the alloy until a maximum hardness is obtained [1, 9].

The precipitation hardening process is conducted in three steps:

1. Heating to the solution heat treating temperature and soaking for long enough to put the elements or compounds into solution.
2. Quenching to room or some intermediate temperature (e.g., boiling water) to keep the alloying elements or compounds in solution; essentially this creates a supersaturated solid solution.
3. Aging at either room temperature (natural aging) or a moderately elevated temperature (artificial aging) to cause the supersaturated solution to form a very fine precipitate in the aluminum matrix [1, 9].

As it is explained above, firstly, solution heat treatment is performed. During this process, elements- such as zinc, magnesium and copper- are dissolved in the solid solution. Zinc has a solid solubility of 66.4 at% in aluminum. The solid solubilities of magnesium and silver are also higher than 10 at%. Copper and silicon have maximum solubility values between 1 and 10 at%. As temperature decreases, the solubility limits decrease. This characteristic is a prerequisite for the alloying elements, in order to achieve increase in hardness and strength by solution heat treatment and subsequent precipitation aging operations. Then, quenching is applied [1].

Following the quenching process, ageing is performed. At the age hardening step, solute atoms form coherent clusters. Due to the difference in size between these solute and solvent atoms, a strain occurs. At that point, dislocations try to diminish the strain, which immobilize dislocations. As a result, it can be concluded that strain fields, which surround the coherent particles, in the matrix restrain dislocation movement and thus, assure higher strength and hardness [1]. More detailed information is provided in the following sub-section.

2.7.1. Solution Heat Treating and Ageing

Solution heat treatment is the first step of precipitation strengthening. In this process, aluminum alloys are brought to a high temperature, which is dependent on the chemical composition and production method, and soaked for sufficient time, which is determined according to the size of the component, to maximize the solubility of elements and accordingly, achieve the maximum amount of solute in the solid solution. The minimum temperature for solution heat treatment should be above the solvus. On the other hand, maximum solubility and high diffusion rate are

achieved near the solidus or eutectic temperature, so the alloys are heated to a temperature slightly below the eutectic temperature for solution heat treatment, but while determining the temperature, one should also consider the production method. For example, solution heat treatment temperature for 7075 aluminum alloy ranges from 465°C for extruded components to 490°C for rolled or cold finished components. Additionally, other factors that can have an effect on the determination of the maximum temperature can also be stated as surface effects, grain growth, and economy of operation [1, 9, 29].

After the elements are dissolved into solution, the alloy is quenched to a relatively low temperature to obtain a solid solution supersaturated with both solute elements and vacancies. This rapid cooling or quenching provides the driving force for precipitation on ageing. The problem is to quench the part fast enough to keep the hardening elements in solution, while at the same time minimizing residual quenching stresses that cause warpage and distortion. In general, the highest strength levels, and the best combinations of strength and toughness, are obtained by using the fastest quench rate possible. While fast quenching rates can be achieved by cold water, slower quenching rates (e.g., hot or boiling water) are often used to sacrifice some strength and corrosion resistance for reduced warpage and distortion. In order to prevent premature precipitation during quenching, time interval for the transfer from furnace to the quench tank must be short and quench tank must be large enough. For example, 7XXX alloys should be cooled at a rate more than 430 °C/sec through the temperature range of 400-300 °C [1, 9, 29, 30].

Finally, ageing is conducted. During ageing treatment, as time passes at the constant ageing temperature, elements -which were dissolved at high solution heat treatment temperature- precipitate because of low solid solubility, due to lower temperature. These finely dispersed precipitates play the major role in increasing ultimate strength, yield strength and hardness. For that reason, it is also called precipitation hardening [1, 9].

Ageing can be applied either at room temperature, which is called natural ageing, or at an elevated temperature, which is called artificial ageing.

In natural ageing, depending on the alloy, solute atoms either accumulate or separate into certain atomic lattice planes, forming the Guinier-Preston (G-P) zones spontaneously, which resist to dislocation movement [1, 9].

On the other hand, in artificial ageing, alloy is heated to a temperature between 95 and 230°C, and soaked at the ageing temperature for 5-48 hours. For instance, in order to achieve T6 temper in 7XXX series alloys an ageing temperature of 120°C for up to 24 hours or longer is generally applied. Details for the precipitation are provided in the following sub-section [1, 9, 29, 31].

When we heat the quenched material in the range of 95-205°C, precipitation is accelerated. Time and temperature determines the scope of structural changes. As the ageing temperature gets higher, the time that is required to obtain maximum properties, gets shorter. So, high ageing temperature could cause over-ageing problems at the fastest heated parts of items, if the heating of sample is not homogenous. On the other hand, if alloys are not artificially aged at a sufficient ageing temperature for sufficient time, there could occur underageing, which will result in lower hardness values than estimated. As an example, the ageing behavior of artificially aged 2024-T4 is presented in Figure 13 [9, 29].

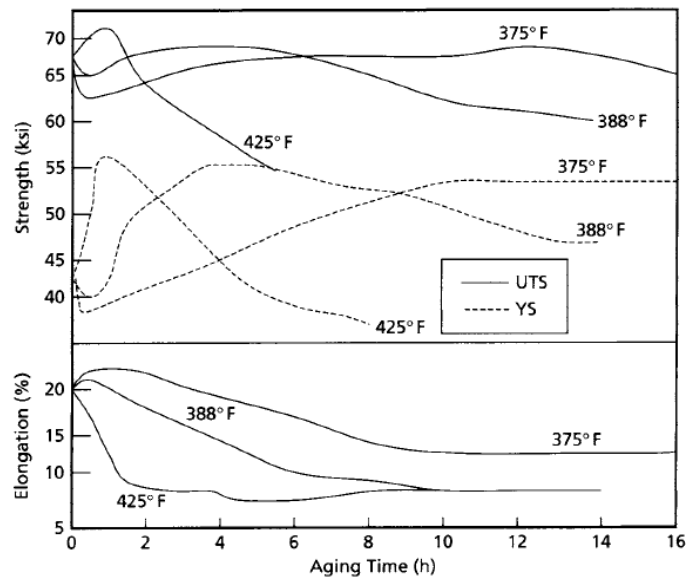


Figure 13. Artificial Ageing Curves for 2024-T4 Sheet [29].

In order to verify the quality of heat treatment, hardness and electrical conductivity tests are conducted [9].

2.7.2. Precipitation Sequence in Al-Zn-Mg-Cu Alloys

The general precipitation sequence can be summarized as the clustering of vacancies -which makes diffusion and hence zone formation occur much faster-, the formation of the metastable miscibility gap that is called G-P zone, the nucleation of a coherent precipitate, the precipitation of an incoherent precipitate and the coarsening of the precipitates. The last step takes place below the G-P zone solvus line. An in depth explanation is provided below [1, 9, 29, 31].

Firstly, the supersaturated solid solution starts to disintegrate, by forming solute clusters. Afterwards, solute atoms diffuse into these initial clusters; then, when the diffused amount of solute becomes sufficient, coherent precipitates nucleate. Because of the fact that there is a lattice parameter mismatch between the solute clusters and aluminum matrix, a strain field develops around the solute clusters. Meanwhile, solute atoms continue to diffuse; in the end, the matrix mismatch becomes unbearable and a semi-coherent precipitate forms. Then, those semi-coherent precipitates grow. When the matrix cannot accommodate the crystallographic mismatch, equilibrium phase forms as incoherent precipitates [1, 9, 29, 31].

The four precipitation sequences, which are determined for 7XXX (Al-Zn-Mg-Cu) alloys, are presented schematically in Figure 14.

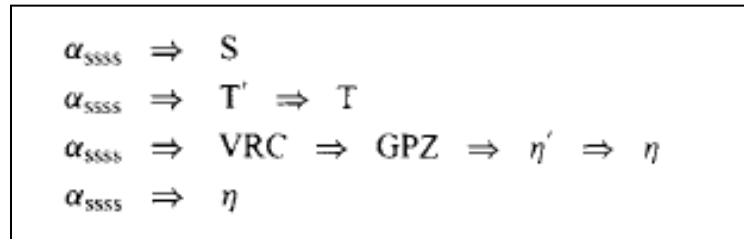


Figure 14. Four Precipitation Sequence of Al-Zn-Mg-Cu Alloys [29].

The first precipitation sequence shows the direct precipitation of the S phase, which is Al_2CuMg , from the supersaturated α solid solution (α_{SSSS}). This is indicated to be a coarse intermetallic and it is not soluble in Al-Zn-Mg-Cu alloys at 465°C [6, 29].

In the second sequence, an intermediate phase T' forms before the equilibrium T phase. It is identified that the chemical formula of the T phase is $\text{Mg}_{32}(\text{Al},\text{Zn})_{49}$ and

it is incoherent with the aluminium matrix. The precipitation of the T phase usually occurs above 200°C [6, 13, 29].

In the third precipitation sequence, firstly vacancy-rich clusters (VRC) form with the decomposition of the supersaturated solid solution. Then, G-P zones (GPZ), η' , and η (eta) phases form, one after another. The final, η , phase is the equilibrium phase and it is identified as MgZn_2 [6, 13, 29].

The fourth precipitation sequence presents the direct precipitation of η from the supersaturated α solid solution (α_{SSS}) [6, 13, 29].

2.7.3. Defects Associated with Heat Treatment

Defects can form, while a component is being manufactured. Some of these defects that occur according to the process can be stated as following:

- Casting defects: inclusions, and midline porosity.
- Homogenisation defects: hard intermetallics formation, other second phase particle formation, and segregation.
- Solution heat treatment defects: incipient melting, underheating, and oxidation (blistering).
- Quenching defects: distortion of the part, precipitation during quenching, and inadequate supersaturation. 7075 is indicated to be quench sensitive.
- Ageing defects: growth or shrinkage of the part [9, 11, 29, 30].

The two main solution heat treatment defects are explained in detail in the following sub-sections, due to relevancy to the study.

2.7.3.1. Blistering

When a component is heated to a high temperature and soaked for a long time long, there could occur "blistering (or high temperature oxidation)". This kind of a situation mainly takes place in solution heat treatment. Moisture in the air is a source of hydrogen and this hydrogen diffuses into the aluminum alloy component during solution heat treatment. Inclusions or other discontinuities act as sites for void formation. Afterwards, hydrogen gas aggregates, similar to the Ostwald Ripening fact. In the end, a blister on the surface of the component is formed. The most susceptible alloys to this kind of defect are the 7XXX alloys [9, 11, 29].

Since moisture is the source of hydrogen, and hydrogen is the cause of surface blistering, this surface blistering problem can be minimised by annihilating the moisture. There are several precautions that can be taken in order to fulfill this requirement, which can be stated as following:

- by ensuring that the solution heat treatment furnace, furnace loads and load racks are free of moisture before the solution heat treatment
- by placing doors over quench tanks in a sequence
- If the ambient relative humidity is too high, then alternative precautions can be such as:
 - The usage of ammonium fluoroborate

- The anodisation of components before the solution heat treatment [9, 11, 29].

2.7.3.2. Eutectic Melting

As it was emphasized previously in the determination of solution heat treatment temperature in order to obtain maximum solid solubility of the elements, the alloy should be heated to a temperature that is close to the eutectic. Accordingly, when we deal with alloys, which have low eutectic temperature, more attention should be paid to avoid heating to a temperature above the eutectic temperature. In that case, eutectic (incipient) melting occurs, which results in the formation of quench cracks, ductility losses, and reduction in toughness and tensile properties; eventually, the component is devastated.

On the other hand, eutectic melting becomes more critical when it occurs in solution heat treatments that are conducted at temperatures below the eutectic point. The reason for this is the non-equilibrium conditions that are caused by the increase in localised solute concentrations. This localisation could result in the reduction of the eutectic temperature and thus, localised melting [1, 29].

In 7XXX series aluminium alloys, eutectic melting can occur under certain conditions. The two soluble phases in this series alloys are $MgZn_2$ and Al_2CuMg , but among these two, Al_2CuMg is the one that has very slow dissolution. Consequently, when we heat the components to temperatures between 485-490°C during solution heat treatment, since Al_2CuMg is very slow to dissolve, it can cause local concentrations. This situation leads to the destruction of equilibrium, and as a

result, eutectic melting can occur. For that reason, 7XXX alloys should be homogenised in order to prevent this risk [29].

2.8. Metallographic Practices in 7xxx Series Aluminum Alloys

As it was previously indicated, zinc is the main alloying element in 7xxx series aluminum alloys group. Because of the fact that zinc is highly soluble in aluminum; it exerts no appreciable influence on the microstructure. However, this class of alloy also contains magnesium and copper, in addition to additives such as manganese, chromium, iron and silicon [1, 6].

7075 alloy in the as-cast structure forms one or more variants of Mg_2Si , $(Fe,Cr)_3SiAl_{12}$, and $Mg(Zn,Cu,Al)$. As we heat the component, there occurs several changes in those phases, such as the transformation of the iron-rich phases to Al_7Cu_2Fe , spheroidization of Mg_2Si , dissolution of $Mg(Zn,Cu,Al)_2$, and precipitation of Al_2CuMg precipitates [1].

The precipitation of $Cr_2Mg_3Al_{18}$ dispersoids from the supersaturated solution mainly occurs at the primary dendrite regions. If a wrought alloy is well-solutionized, Mg_2Si , Al_7Cu_2Fe , and $(Fe,Cr)_3SiAl_{12}$ also appear in addition to the dispersoid. Dispersoid bending causes recrystallized grains to become elongated. The unrecrystallized regions are common. The unrecrystallized regions whose boundaries are indicated by hardening precipitate can be observed in Figure 15. In this figure the fine particles of $MgZn_2$ (dark) and the insoluble particles of $FeAl_3$ (light gray, outlined) could be observed [1].



Figure 15. Alloy 7075-O Sheet, Annealed, 500X [1].

For the characterization of an alloy, a single microstructural aspect is not enough. Heat removal rate determines the initial size of second-phase particles throughout solidification. The individual alloy composition and thermal treatment are effective on the excess soluble phase amount. Mechanical deformation severity and heating rate determines the degree of recovery and recrystallization, in addition to the size and shape of the recrystallized grains on [1].

CHAPTER 3

EXPERIMENTAL PROCEDURE

The experiments in this study were carried out according to the experimental procedure that is summarized in Figure 16 on the next page.

First of all, 16 kgs of 7075 aluminum alloy was purchased from a local pressure die casting company, in order to be used in this study. The chemical composition of this purchased 7075 sample is presented in Table 6. 6 kg of this sample was separated to be used in evaluation of Mg addition effect on 7075 and the rest is separated to be used in evaluation of 7075 alloy.

Table 6. The Chemical Composition of 7075 Alloy that was used in this Study.

Si	Fe	Cu	Mn	Mg	Cr	Zn	Al
0.114	0.185	1.450	0.001	2.540	0.190	5.790	89.600

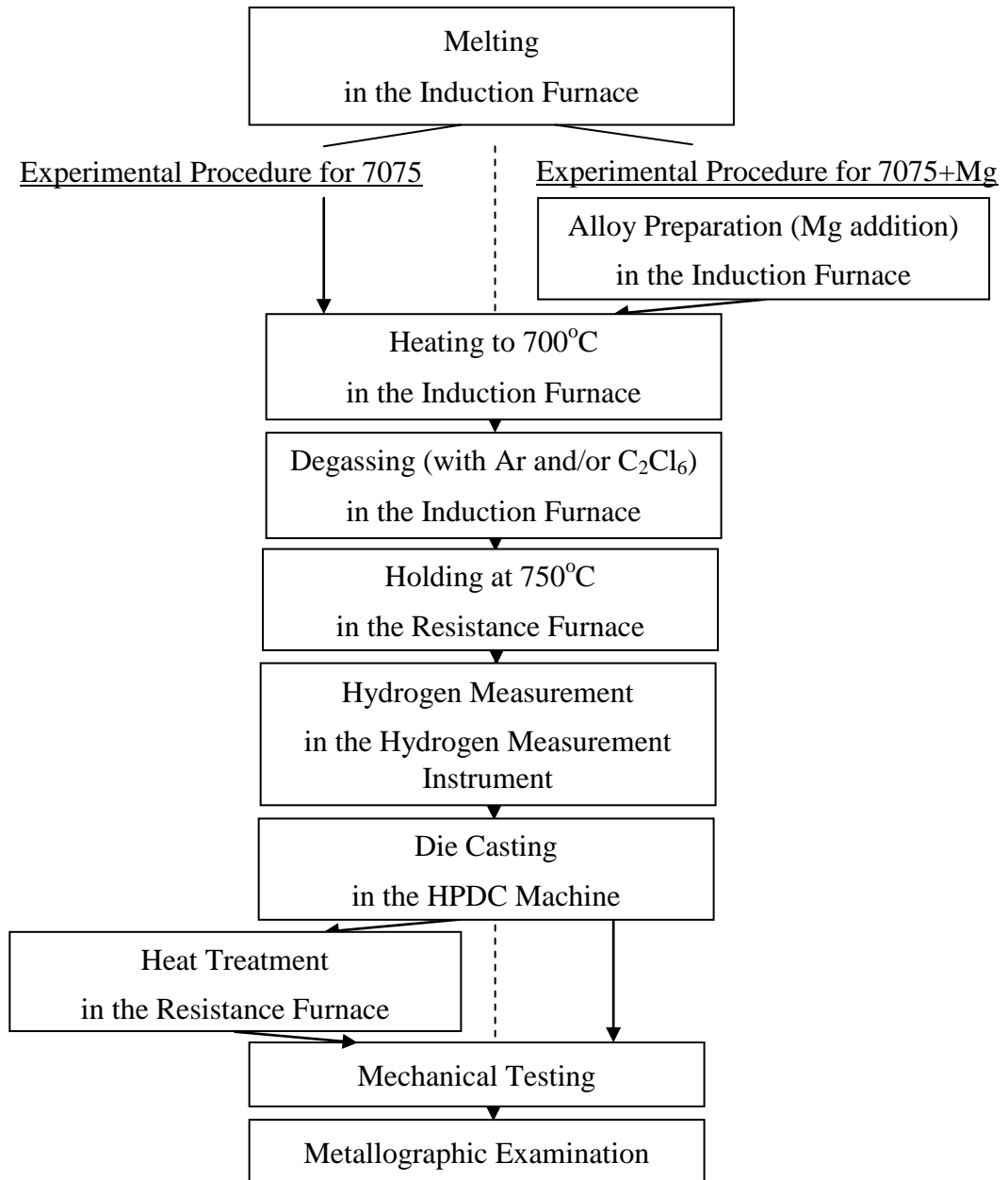


Figure 16. Experimental Procedure Flowchart.

3.1. 7075 + Mg Experiments

In the first part of this study, with the aim of examining magnesium effect, magnesium was added during the alloy preparation step in the induction furnace. The added amounts of magnesium for 16 different casting operations were listed in Table 7.

Table 7. The Weight of Samples After Mg Addition.

#	Initial Total	Initial Mg	Added Mg	Final Total
	g	g	g	g
	6000	152	0	0
1	6000	152.4	24	6024
2	6000	152.4	32	6032
3	6000	152.4	36	6036
4	6000	152.4	40	6040
5	6000	152.4	44	6044
6	6000	152.4	54	6054
7	6000	152.4	55	6055
8	6000	152.4	65	6065
9	6000	152.4	68	6068
10	6000	152.4	71	6071
11	6000	152.4	90	6090
12	6000	152.4	93	6093
13	6000	152.4	98	6098
14	6000	152.4	124	6124
15	6000	152.4	159	6159
16	6000	152.4	183	6183

3.2. 7075 Experiments

In the second part of this study, 7075 aluminum alloy with the chemical composition which was presented in Table 6 was used. A total of 27 casting operations were performed for 7075 alloy.

The detailed information for the experimental procedure applications are provided in Section 3.3.

3.3. Experimental Procedure Applications in Equipments

3.3.1. Induction Furnace

During experiments of both 7075 and 7075+Mg alloys, firstly, aluminum alloy samples were melted in the induction furnace (Front view in Figure 17 and top view in Figure 18). The induction equipment manufactured by the Ajax Magnethermic Corp. has the properties that are given in Table 8.

Table 8. Induction Equipment Properties.

Power	VOLTS	KVA	AMPS	PHASE	FREQUENCY
Input Rating	380	59	90	3	50
Output Rating	600	-	2000	1	2500

Secondly, the charge alloy was heated to 700°C in the induction furnace. This temperature is approximately 50°C higher than the liquidus temperature of 7075 aluminum alloy. In the electric induction furnace, a magnetic field is created directly within the liquid aluminum alloy bath. Eddy currents induced due to the magnetic field produce heat and directional forces. These directional forces cause the desirable circulation of the alloy. As a result, a homogenous mixture is obtained [1, 11].

Thirdly, degassing operation is performed to control hydrogen content in reasonable limits. These degassing operations were applied in two different ways. While in the first one, Argon gas was introduced into the melt, in the other one hexachloroethane (C₂Cl₆) was introduced into the induction furnace. During the second type of degassing treatment, hexachloroethane was immersed into the melt inside a crucible. Hexachloroethane that is dissolved inside the molten metal forms chloride, which forces hydrogen towards melt surface as AlCl₃ gas bubbles [17].

3.3.2. Resistance Furnace

After degassing process, the alloy sample in molten form was passed into the resistance furnace in order to limit the interaction of alloy with the air. The temperature of the resistance furnace was kept at 750°C, 50°C higher than the temperature at the induction furnace and approximately 100°C higher than the liquidus temperature of 7075. This high value in temperature was adjusted as a caution for heat losses that occur in cold chamber die casting operations while passing from induction furnace to the pressure die casting machine.

3.3.3. Hydrogen Measurement

Hydrogen content measurement operation was done for each alloy sample before pressure die casting process. Because of the fact that there is a large difference between the hydrogen solubility in liquid and solid, which can be shown by the ratio of liquid to solid solubility as 20:1, hydrogen bubbles are formed during solidification. Eventually, these bubbles cause porosity in castings and blisters on components [1, 11, 32].

In this study, HYSCAN II (Figure 19) instrument is used to measure hydrogen content in samples. This instrument measures hydrogen in molten aluminum using a reduced pressure technique developed by the BNF Metals Technology Centre for the UK Light Metal Founders' Association.

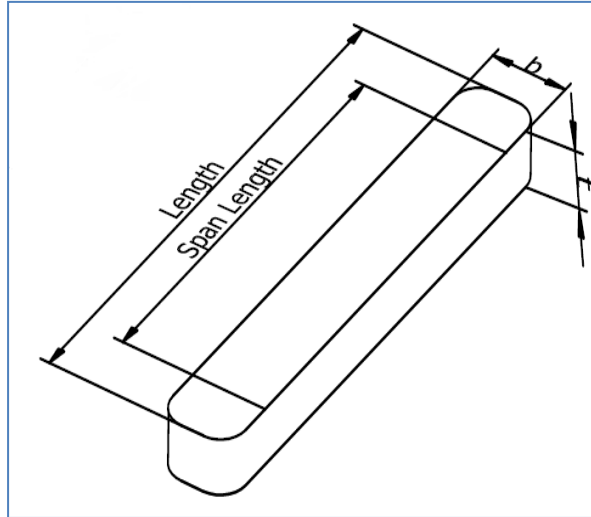


Figure 31. Three Point Bending Test Specimen Dimensions.

The dimensions of the three point bending test specimen that is presented in the Figure 31 are as following:

Length = 65 ± 0.1 mm

Span length = 50 mm

b (width) = 10 ± 0.1 mm

t (height) = 10.3 ± 0.3 mm

3.4.3. Hardness Test

Hardness test was done with the help of EMCO Universal Digital Hardness Testing Machine. The test was conducted according to the ASTM E 10-01 Standard Test Method for Brinell Hardness of Metallic Materials [37]. With reference to that

standard, tungsten carbide ball with 2.5 mm diameter was used as indenter and 1.839 kN (187.5 kf) was applied. HBW value for 7075-T6 sample was expected to be 150 according to the value provided in literature [1].

3.5. Characterization

3.5.1. Optical Microscopy

Microstrure examination is generally done for determining the effects of mechanical and thermal treatments and analyzing cause of failures [1].

In order to examine a sample under microscope, the samples should be cut to the convenient dimension that would make it possible to handle it. In this study, samples to be examined by optical microscopy were cut from the tensile test specimens by Metacut-M 250 Cut-Off Machine (Figure 32). Pictures of cut samples are presented in Figure 33.

CHAPTER 4

RESULTS AND DISCUSSION

In this study, die casting trials were performed by high pressure die casting machine. Following this, we conducted mechanical and metallographic examinations with the aim of determining the effect of hydrogen content on hydrogen porosity and the effects of hydrogen porosity, Mg concentration and T6 heat treatment on mechanical properties of pressure die cast 7075 alloy.

4.1. Magnesium Effect

In this study, magnesium was added to compensate the lack of fluidity in 7075 alloy. Because of the fact that this addition caused a change in chemical composition of the alloy, mechanical tests and metallographic examinations were carried out to evaluate the properties of the developed alloy. In previous studies, it was concluded that while increasing magnesium content, attention should be paid to the maximum level of magnesium concentration; because, although increase in magnesium up to 6 wt% increases strength and provide castability, when its concentration exceeds that value, there occurs decrease in fluidity and castability problems. In reference to this, alloys were prepared with the chemical compositions as listed in Table 10 for 16 die casting operations [1, 11, 38].

coldshut in the cast products, which could be an evidence for the achievement of fluidity. However, it can be interpreted from the figures that as magnesium concentration increased, UTS and Flexural Strength properties diminished. The reason for this could be either existence of intermetallic phases and/or porosity.

In case of porosity, since hydrogen content was not considered during preparation of these figures, the evaluation of the results may not be reasonable. For that reason, hydrogen content effect was tried to be incorporated by selecting the results, which had the same amount of hydrogen, from the 16 casting operations, grouping them according to their hydrogen amounts, and comparing the results for different Mg concentrations in each group. The UTS and Flexural Strength values, which are listed accordingly, are presented in Table 11, and Table 12, respectively. It can be observed that there is no certain relationship for the values and if there is, it is not possible to qualify this relationship with only those parameters. This variation in results could be due to shrinkage porosities that occur because of certain factors, such as the die temperature, casting velocity, gating design etc. In this study, although all the parameters, such as phase adjustments and die temperature, of the pressure die casting machine were kept constant, and even though necessary precautions were taken during the study, the formation of shrinkage porosity showed variation. In other words, although there was no shrinkage porosity formation in one casting, there could be in the one that was cast right after, even with the same chemical composition. For that reason, it was not possible to control and hinder the formation of them.

Table 11. Magnesium Concentration and UTS Values.

Hydrogen content cc / 100 g	Mg wt %	Ultimate Tensile Strength MPa
0.23	3.59	235
0.23	3.06	206
0.38	3.68	166
0.38	3.43	201
0.50	4.02	218
0.50	3.63	196
0.54	3.25	193
0.54	3.19	222
0.58	5.06	198
0.58	4.10	191

Table 12. Magnesium Composition and Flexural Strength.

Hydrogen content	Mg	Flexural Strength
cc / 100 g	wt %	MPa
0.23	3.59	343
0.23	3.06	361
0.38	3.68	320
0.38	3.43	3014
0.50	4.51	367
0.50	4.02	338
0.50	3.63	348
0.54	3.25	363
0.54	3.19	397
0.58	5.06	327
0.58	4.10	356

4.2. Hydrogen Content Effect

In the liquid state, hydrogen is more soluble compared to that in the solid state. When the metal solidifies, hydrogen, which exceeds the solid solubility limit, cannot remain in the solid solution, eventually it transforms into gas, forming bubbles in the liquid. Meanwhile, solid surface that is formed during solidification proceeds. As solidification proceeds, this solid interface progresses. In the end, it reaches and captures those bubbles; consequently, porosity will be formed.

Extent of porosity in a particular casting can be determined by certain methods such as:

- Archimedes' method for density measurement.
- Radiography.
- Ultrasonic attenuation.
- Metallographic examination [11].

In our study, we used metallographic examination method to detect porosities.

Microscopic investigation was carried out with the help of optical microscopy and image analyser. But, before evaluating the results of hydrogen porosity, one must consider another porosity that is faced with in castings during microscopic examination, which is shrinkage porosity.

Shrinkage occurs, when metal solidifies. Since rapid solidification rate promotes shrinkage, it is a more critical situation in die casting operations. The shrinkage amount is basically dependent on the composition of the alloy, the temperature of

the die surface, the injection temperature, cast part configuration and the injection pressure. Alloys, which contain iron less than 0.8 wt%, magnesium less than 0.3 wt%, and zinc more than 4 wt%, are determined to be more prone to shrinkage defects. The concentrations of our alloys were in this range, except magnesium content, so this could be one of the reasons for the shrinkage porosities in our samples [11, 25].

Shrinkage porosity is distinguished from gas porosity by its rough and irregular inside surface formed due to dendritic structure associated with it. It affects the internal integrity of the casting. Since, die casting is a rapid solidification process, in which solidification starts from the surface and goes through the center of the casting, shrinkage occurs at the center of the casting part [11].

In this study, in the samples that were investigated by image analyser, the distinguishment between gas porosities and shrinkage porosities were done according to that difference in their shapes. For instance, as it can be seen from the Figure 38, there are certain irregularly shaped porosities at the right side and small spherical like porosities distributed all around. Because of the fact that those irregularly shaped porosities, either large or small, are shrinkage porosities, during image analysis of hydrogen porosities, they were ignored.

Table 13. Hydrogen Content and Porosity Percentage Values for 7075.

#	Hydrogen content (cc / 100 g)	Hydrogen porosity (%)
1	0.56	2.117
2	0.40	1.709
3	0.40	1.613
4	0.40	1.633
5	0.65	2.302
6	0.65	2.508
7	0.65	2.422
8	0.65	2.020
9	0.65	2.417
10	0.48	1.765
11	0.48	1.756
12	0.48	1.742
13	0.48	1.835
14	0.48	2.017
15	0.69	1.889
16	0.69	3.185

4.4. The Evaluation of the Effect of T6 Heat Treatment on Pressure Die Cast 7075 Sample

4.4.1. Tensile Test

In this study, T6 heat treatment was performed through the application of solution heat treatment, quenching and artificial ageing. The steps of this heat treatment were previously presented in Figure 25, in the Experimental Procedure section. After heat treatment, tensile test was conducted for 7075 sample. The Ultimate Tensile Strength value for the sample with a hydrogen content of 0.69 cc/100 g was determined to be 344.93 MPa. This value was approximately 150 MPa higher than the non heat-treated counterpart of that sample. This increase was expected, because, in the wrought aluminum-zinc alloys, during T6 heat treatment, mainly the precipitation of $MgZn_2$ takes place, which hinders dislocation motion, and thus, increases the tensile and yield strengths [1, 6, 8, 27]. Normally a higher value, close to 500 MPa was anticipated in reference to ASM Aluminum Handbook, but a lower value was obtained. The reason for this lower value could be hydrogen porosity, shrinkage porosity and/or eutectic melting.

4.4.2. Hardness Test

Hardness Test was conducted according to the ASTM E 10-01 Standard Test Method for Brinell Hardness of Metallic Materials [37]. HBW is calculated by the digital hardness testing machine, according to the following formula:

$$HBW = 0.102 \times \frac{2 F}{\pi D \left(D - \sqrt{D^2 - d^2} \right)}$$

where:

D = diameter of the ball, mm

F = test force, N

d = mean diameter of the indentation, mm.

HBW value for 7075-T6 sample was expected to be 150 HBW, according to the hardness test that was carried out with 10 mm ball at 4.90 kN (500 kgf) [1]. In this study, the test was conducted with tungsten carbide ball of 2.5 mm indenter diameter and with 1.839 kN (187.5 kgf). The data conversion was conducted according to the formula: $0.102 F/D^2$. The value that was calculated for 2.5 mm and 187.5 kgf data was found to be 30, which corresponds to 29.42 kN (3000 kgf) force and 10 mm ball, in reference to the relevant standard. The data conversion was conducted by the digital hardness measurement equipment. Accordingly, test results came out to be as listed in Table 14.

Table 14. HBW Test Results.

	HBW 2.5/187.5
Indentation 1	179
Indentation 2	173
Indentation 3	184

CHAPTER 5

CONCLUSIONS

The importance of aluminum alloys is increasing day after day, due to their advanced properties such as high strength, in addition to their low density property. Among all aluminum alloys, 7075 is the one that has highest tensile strength and effective precipitation hardenability characteristic. However, it can only be used properly in wrought form, instead of as-cast structure, since it is hard to obtain good castability characteristics, due to its low silicon content. Recently, some of the studies that are conducted on 7075 are concentrated on rendering the application of as-cast form of this alloy possible. In this study, magnesium is added to 7075, in order to achieve practicability of this alloy by augmenting its fluidity. Hydrogen content effect was also in the scope of this study. After the conducted tests, the following statements were concluded for high pressure die cast 7075 aluminum alloy:

1. *As the magnesium content increased, tensile and flexural strength values slightly diminished.* At the beginning of this study, it was expected that magnesium addition would increase strength due to the solid solution strengthening mechanism and increased fluidity, but after the tests, it is observed that magnesium addition did not have a favorable effect on tensile and flexural strength of this alloy. Due to the fact that, as the magnesium composition increased from 3 wt% to 5.5 wt%, the UTS value decreased from 230 MPa to 180 MPa, and flexural strength decreased from 350 MPa to 330 MPa.

2. *As hydrogen content increased, hydrogen porosity increased linearly.* This relationship was found to be as:

$$\text{Hydrogen porosity \%} = 3.0325 \times \text{hydrogen content} + 0.4006$$

Hydrogen is more soluble in liquid state than that in the solid state. During solidification, hydrogen that exceeds the solid solubility limit, transforms into gas by forming bubbles in the liquid. As solidification proceeds, formed solid interface progresses, and when it captures those bubbles, porosity is formed. So, it is reasonable to have hydrogen porosity in proportion with the hydrogen, which exceeds solid solubility limit.

3. *As hydrogen content increased, hydrogen porosity increased, and eventually, tensile and flexural strength values diminished.* When the hydrogen content increased from 0.4 to 0.65, hydrogen porosity increased from 1.6 to 2.3 %. Consequently the UTS and flexural strength values decreased from 200 to 170 MPa, and from 360 to 350 MPa respectively. This situation can be explained by the fact that, hydrogen porosity destroys the fine dense grain structure. As a result, a weak structure is formed, which shows lower tensile and flexural strength properties.
4. *In high pressure die casting operations, gating design effect should also be considered.* During casting operation, if the gating design is not adjusted conveniently, coarse particle flow situation could occur, causing additional porosity. Accordingly, hydrogen porosity at the runner should also be evaluated, in addition to the hydrogen porosity analysis from casting products.

5. *Shrinkage porosity has damaging effect on tensile and flexural strength properties.* Although all the phase adjustments were determined regarding the related properties -such as alloy composition, gate velocity-, and kept fixed during the study, the encountered shrinkage porosity values were not constant. For that reason, they were unable to be prevented. Eventually, they disturbed the dense structure and caused degradation of some of the samples.

6. *Increasing magnesium concentration increases hydrogen solubility.* It is observed that increasing magnesium concentration from 3 to 5 wt% in the alloy, resulted in an increase of hydrogen concentration from 0.23 cc/100g to 0.58 cc/100g in the 7075 alloy.

7. *Heating to high temperature causes blistering on the surface of the samples, due to the hydrogen gas entrapped inside the component.* Especially during the solution heat treatment stage of the T6 heat treatment, hydrogen diffuses into the aluminum alloy component. While this diffusion takes place, sites such as inclusions help to the formation of voids. Then, hydrogen gas gathers analogous to the Ostwald Ripening phenomenon and forms blister on the surface of the component.

8. *Blistering decreases the heat treatment effect on mechanical properties.* The UTS value for the sample with a hydrogen content of 0.69 cc/100g was determined to be 344.93 MPa, which is a lower value compared to 560 MPa UTS reported for wrought 7075 alloy after T6 heat treatment. The reason of this is the destruction of the dense structure due to blister formation, which diminishes the amount of increase in tensile strength.

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3	324.88648	0.01331	466.00632
4	309.65578	0.01211	444.15988
5	302.09529	0.01223	433.31536

	Flexure extension at Yield (Offset 0.2 %) (mm)
1	0.70912
2	0.54737
3	0.54511
4	0.49595
5	0.50124

Figure B. 2. Three Point Bending Test Report for the 2-x Identified 7075+Mg samples (continued)

6	0.77834	0.79000	4781.09375
7	0.64686	0.65326	4806.90039
8	0.84166	0.87161	4797.18359
9	0.55335	0.58163	4390.29541
10	0.74659	0.79323	3980.08350
11	0.71341	0.73679	4927.23291
12	0.77015	0.81176	4191.62207
13	0.84339	0.86515	4889.55469
14	0.74342	0.75658	5154.47852
15	0.68842	0.71813	4434.05371
16	0.57350	0.57828	4606.32617
17	0.66163	0.68124	3463.18115
18	0.73332	0.75173	5078.94482
19	0.78169	0.79837	5036.20801
20	0.81517	0.83418	4538.87061
21	0.59335	0.61673	4461.16602
22	0.67174	0.68023	4534.34082
23	0.80997	0.81499	5106.57617
24	0.65655	0.66002	4271.99316
25	0.79502	0.81613	4381.20996

	Flexure strain at Break (Standard) (mm/mm)	Flexure stress at Break (Standard) (MPa)	Modulus (Automatic Young's) (MPa)
1	0.02630	338.82443	36018.48736
2	0.01895	346.84210	36725.03116
3	0.02054	90.80239	35731.86366
4	0.01798	334.17682	36477.24723
5	0.02003	345.56128	35387.80604
6	0.01915	344.62408	35567.42123
7	0.01584	343.12030	36470.64445
8	0.02113	345.78384	34719.48655
9	0.01410	316.45511	36751.07440
10	0.01923	284.10147	35448.46882
11	0.01786	355.15787	36009.74082
12	0.01968	300.66077	34362.98294
13	0.02108	345.59006	35351.51601
14	0.01834	367.93069	36476.76666
15	0.01741	319.60922	36621.72877
16	0.01402	328.80316	36440.94275
17	0.01651	249.62814	35593.32278
18	0.01822	366.09335	37149.79296
19	0.01935	359.48846	36595.53733
20	0.02022	327.16449	35923.72232
21	0.01495	318.44150	35996.93333
22	0.01657	322.04715	34585.52585
23	0.01976	366.28949	35556.47334
24	0.01600	306.42569	33811.92646
25	0.01988	309.66064	33934.52820

Figure B. 3. Three Point Bending Test Report for 7075 Samples
(Continued)

	Flexure extension at Yield (Offset 0.2 %) (mm)	Flexure load at Yield (Offset 0.2 %) (N)	Flexure strain at Yield (Offset 0.2 %) (mm/mm)
1	0.62813	4154.31729	0.01523
2	0.52786	4265.97837	0.01280
3	0.49084	4015.82965	0.01184
4	0.52149	4274.65565	0.01264
5	0.55311	4299.83380	0.01341
6	0.53470	4179.34310	0.01296
7	0.51416	4406.06315	0.01246
8	0.52237	4122.10879	0.01266
9	0.50183	4418.12579	0.01216
10	0.54102	4248.11538	0.01311
11	0.52595	4506.97360	0.01275
12	0.50410	4078.03682	0.01222
13	0.53584	4496.76565	0.01305
14	0.45969	4458.76282	0.01114
15	0.54314	4340.25356	0.01317
16	0.50992	4410.42886	0.01236
17	0.50753	4207.62979	0.01230
18	0.53432	4602.58222	0.01295
19	0.53130	4404.75140	0.01288
20	0.53115	4375.56560	0.01288
21	0.47545	4231.14387	0.01152
22	0.51635	4150.96624	0.01258
23	0.49438	4295.95643	0.01198
24	0.52852	3911.70835	0.01281
25	0.54277	4236.88543	0.01322

Figure B. 3. Three Point Bending Test Report for 7075 Samples (Continued)