Microstructure Change of Aluminum 6061 through Natural and Artificial Aging

by Agus Suprapto

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6.1 INTRODUCTION: BACKGROUND

Aluminum alloy is a kind of material that is widely used in the world. Recently, the use of aluminum has high demand and it will increase rapidly over the years (Puspitasari et al., 2016). This can be up to 9.9% per year in tons (Tsamroh, 2021). In Indonesia, the Ministry of Industry targeted that Indonesia should be able to produce up to 1.5–2 million tons of aluminum by 2025 (Indonesia, 2018). This high demand is due to the ongoing development of the manufacturing industry. The high use of aluminum is attributed to its several beneficial properties, including being lightweight, ductile, and resistant to corrosion; besides, it can also be recycled (Woodford, 2021). Figure 6.1 shows the various uses of aluminum.

From Figure 6.1, it can be known that aluminum is widely used as a transportation component. For example, the aerospace industry uses aluminum for up to 90% of its components (Rambabu et al., 2017). Many kinds of automotive components are made of aluminum, such as valves, engine blocks, etc. (Ogunsemi et al., 2021). Due to its widespread use in various fields, aluminum needs to be improved in its properties continuously. The increasing use of aluminum alloys certainly affects the production and consumption of aluminum in the world. Recently, research on aluminum alloy has been conducted in many countries, for example, China, the USA, UAE, India, etc. (Woodford, 2021).

Aluminum has been classified into seven series. One of them is aluminum 6061, which is a part of series 6xxx. Aluminum 6061 consists of aluminum-magnesium-silicon, one of the treatable heat alloys, and has medium strength (Rajasekaran et al., 2012). However, compared to other metals or other aluminum series such as the 2xxx

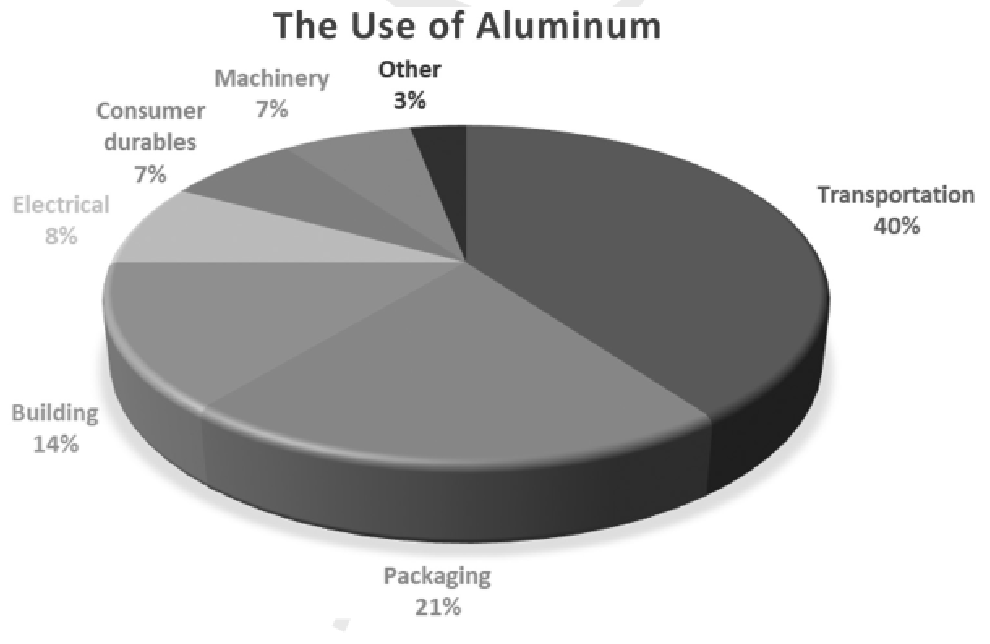

FIGURE 6.1 The use of aluminum in several sectors (Based on Woodford, 2021).

series, aluminum 6061 has lower strength (Nulhaqem & Abdul, 2013). Therefore, effort is required to make better its properties, especially its mechanical properties. Aluminum 6061 is a material widely used in the industrial world such as automotive and aerospace because it has high strength, is lightweight, and is corrosion-resistant. This alloy is commonly applied in the transportation industry, such as truck frames, rail coaches, shipbuilding operations, and military and commercial bridges (Abo Zeid, 2019).

One of the uses of aluminum alloy AA 6061 in the aerospace world is the wings and body of small-scale aircraft. Meanwhile, in the automotive industry, AA6061 is used to manufacture several types of important vehicle parts, such as wheels, panels, and even in-vehicle structures (Wardani et al., 2022). This material is widely used because it has good extrusion, formability, and weldability. In addition, this material also has medium hardness and strength, good corrosion resistance, and a good surface finish (Andoko et al., 2020).

Various processes can be applied to make better the properties of aluminum, such as mechanical and physical properties (Tsamroh et al., 2018). One of them is heat treatment. One kind of heat treatment is aging (Chacko & Nayak, 2014); this treatment can produce homogeneous and evenly distributed precipitates so that an optimal increase in the mechanical properties of the material could be obtained (Rymer et al., 2021). While the aging process is of several kinds, aging treatments that are often used to improve mechanical properties are natural aging and artificial aging (Cochard et al., 2017).

Artificial aging is the aging for aluminum alloys which are treated with age hardening in a hot state. In this study, the strength of AA6061 will be increased through by combining the treatments of natural aging and artificial aging, but this study focuses on the microstructural changes that occur during the aging process. Several studies using artificial aging methods that aim to improve the material's mechanical properties have been carried out on aluminum alloy Al 2024 at a temperature of 0°C–177 °C for 20 hours and have shown an increase in hardness (Prudhomme et al., 2018). The application of the artificial aging treatment on aluminum alloy Al 7075 with a temperature of 120°C with time variations for 60, 120, 180, and 240 minutes achieved a maximum increase in hardness at 120 minutes of holding (Lee et al., 2018). Furthermore, the Al-Si-Mg-Cu alloy with artificial aging treatment achieved maximum tensile strength at an aging temperature of 180°C with a holding time of 240 minutes (Jin et al., 2018). Aluminum alloy AA1350-H19 gained the highest tensile strength in the artificial aging treatment at a temperature of 200 °C for 4 hours of holding (Flores et al., 2018).

Based on the explanation above, it is necessary to study how changes in the microstructure of Al6061 due to natural aging and artificial aging heat treatments can affect the mechanical properties of the material. Two important parameters strongly influence changes in the microstructure of Al6061, namely, heating temperature and holding time (Tsamroh et al., 2018). The heating temperature and holding time used in the heat treatment should be determined precisely; the use of a heating temperature that is too high and holding for a long time will actually damage the material microstructure. This will also affect the mechanical properties of the material.

6.2 ALUMINUM

Materials have been classified into four major materials: metal, composite, ceramic, and fiber. Metal is divided into ferrous metals and non-hero metals. In general, aluminum can be classified into three big groups: wrought non-heat-treatable alloys, wrought heat-treatable alloys, and casting alloys (Davis, 1993). Meanwhile, aluminum casting alloys are classified into two types: alloys that have the ability to be treated by using heat treatment and alloys that do not have the ability to be treated by using heat treatment. Table 6.1 presents the aluminum classification and the aluminum alloy naming code.

Aluminum is obtained from certain types of clay (bauxite). Bauxite is first separated from pure alum (aluminum oxide). Bauxite is one of the most important materials for aluminum production, which is hydrated aluminum oxide containing 1 to 20% Fe_2O_3; 1 to 10% silicate to a lesser extent of zirconium, titanium, vanadium, and several transition metal oxides; 20 to 30% is water, and 50 to 60% Al_2O_3. Bauxite can be purified by using the process of Bayer. By filtration, sodium hydroxide can dissolve the crude bauxite and be separated from hydrated iron oxide and other insoluble foreign substances (Davis, 2001).

Then the molten aluminum oxide is calcined by an electrical procedure. Because the melting temperature of aluminum oxide is very high, namely, 2050ºC, the processing of aluminum is very difficult. Aluminum metal has the symbol Al, which has a specific gravity of 2.6–2.7 with a melting point of 659ºC. Aluminum is a soft metal and is harder than lead but softer than zinc. The color of aluminum is bluish-white. Aluminum can be produced through electrolysis process. The electrolysis process developed for industrial production is the Hall–Heroult electrolysis process. The process is electrolysis of alumina (Al_2O_3) solution in molten cryolite (Na_3AlF_6) at a temperature of 960ºC to produce molten aluminum.

TABLE 6.1
Aluminum Classification and Naming Code

Aluminum Type	Classification	Naming Code
Aluminum alloy for machining	Wrought non-heat-treatable alloy	Pure Al (1000 series) Al-Mn Alloy (3000 series) Al-Si Alloy (4000 series) Al-Mg Alloy (5000 series)
	Wrought heat-treatable alloy	Al-Cu Alloy (2000 series) Al-Mg-Si Alloy (6000 series) Al-Zn Alloy (7000 series)
Aluminum alloy for casting	Non-heat-treatable casting alloy	Al-Si Alloy (Silumin) Al-Mg Alloy (Hydronarium)
	Heat-treatable casting alloy	Al-Cu Alloy (Lautal) Al-Si-Mg Alloy (Silumin, Lo-ex)

Source: Irawan, 2015.

6.2.1 PURE ALUMINUM

Aluminum is obtained in the molten state by electrolysis, which generally attains purity of 99.85% by weight. However, if further electrolysis is carried out, aluminum will be obtained with a purity of 99.99%. Corrosion resistance varies with purity; generally, 99.0% or above purity can be used in resistant air for many years. The electrical conductivity of Al is about 65% of the electrical conductivity of copper, but its density is about one-third of that of copper, so it is possible to expand its cross-section. Therefore, it can be used for cables and in various forms, such as a thin sheet (foil). Al with a purity of 99.0% can be used in this case. Al with that level of purity is used for reflectors that require high reflectivity and electrolytic coders.

6.2.2 ALUMINUM ALLOY

Aluminum alloys are grouped in various standards by various countries in the world. However, the most well-known and perfect classification is the Aluminum Association (AA) standard in America which is based on the previous standard from Aluminum Company of America (Alcoa).

Table 6.2 presents the physical characteristics of aluminum.

6.2.2.1 Al-Cu Alloy

Al-Cu and Al-Cu-Mg alloys are one of the main aluminum alloys. Copper is the main alloying element in aluminum in the 2000 series aluminum, which is often added with Mg as an additional alloying element (Davis, 2001). Al-Cu-Mg alloys contain 4% Cu and 0.5% Mg, which can harden greatly within a few days by aging at ordinary temperatures after solution heat treatment. Aluminum alloyed with Cu has poor corrosion resistance, so it is necessary to coat the surface with pure aluminum or a corrosion-resistant aluminum alloy (alclad plate). However, the alloy is used as an aircraft material (Surdia & Saito, 1999). Aluminum and copper alloys (Al-Cu alloys) are aluminum alloys known as duraluminium or super duraluminum. Duraluminum is also often referred to as duralumin or duralium (Junkers, 2014).

TABLE 6.2
Physical Characteristics of Aluminum

Character	High Pure Aluminum
Crystal structure	FCC
Density at 20ºC (sat. 10^3 kg/m^3)	2.698
Melting point (ºC)	660.1
Wire heat creep coefficient 20ºC~100ºC (10^6/K)	23.9
Heat conductivity 20ºC~400ºC (W/(m-K))	238
Electrical resistance 20ºC (10^{-8} KΩ-m)	2.69
Modulus of elasticity (GPa)	70.5
Stiffness modulus (GPa)	26.0

6.2.2.2 Al-Mn Alloy

Manganese (Mn) is the main element in 3000 series aluminum alloys. Generally, this alloy is an alloy that cannot be heat treated but has 20% more strength than 1000 series aluminum (Davis, 2001). The addition of the element Mn to aluminum can strengthen aluminum without reducing[9]s corrosion resistance and is used to make corrosion-resistant alloys. The alloys Al-1.2% Mn and Al-1.2%Mn-1.0%Mg are alloys 3003 and 3004 that are used as corrosion-resistant alloys without heat stiffening (Surdia & Saito, 1999). This alloy is often used for kitchen utensils and panels (Irawan, 2015).

6.2.2.3 Al-Si Alloy

The main alloying element in 4000 series aluminum alloys is silicon. Silicon can be added in sufficient quantities (nearly 12%). The addition of silicon elements to the alloy lowers its melting range without causing the alloy to become brittle. This alloy is widely used in welding wire as brazing (Davis, 2001). Al-Si alloys have very good fluidity, have a good surface, have no heat flexibility, and are very good as alloy castings (silumin). This alloy also has good corrosion resistance, is very light, has a small expansion coefficient, and is a good conductor of electricity and heat. Al-12%Si alloys are widely used for cast alloys (silumin). The alloy that is treated with dissolution and aging is called silumin. The properties of silumin can be increased by applying heat treatment and slightly improved by alloying elements. Generally, alloys with 0.15–0.4% Mn and 0.5% Mg are used. Alloys that require heat treatment are added with Mg as well as Cu and Ni to provide hardness when hot; these materials are commonly used for motor pistons (Surdia & Saito, 1999).

6.2.2.4 Al-Mg Alloy

The 5000 series aluminum alloy is mainly alloyed with Mg. When combined with manganese, it produces a medium to high strength working alloy. As a hardener, magnesium is better than manganese, with 0.8%Mg equivalent to 1.25%Mn (Davis, 2001). Al-Mg alloy has good corrosion resistance; this alloy has long been called hydronalium and is known as an alloy that is resistant to corrosion (Surdia & Saito, 1999). To increase the strength of the alloy against stress corrosion, Mn and Cr elements are added (Irawan, 2015).

Alloys with a content of 2–3% Mg have the character of being easy to forge, roll, and extract. Aluminum alloy 5005 is an alloy that has a low Mg content and is often used as an accessory. Aluminum alloy 5052 is an alloy that is often used as a forging or construction material. Aluminum alloy 5056 is the strongest alloy where it is used after being hardened by strain hardening if high hardness is required. The annealed alloy 5083 is an alloy of 4.5%Mg, which is strong and easy to weld; therefore, it is used in liquefied natural gas (LNG) tanks (Irawan, 2015; Surdia & Saito, 1999).

6.2.2.5 Al-Mg-Si Alloy

Aluminum is a non-ferrous metal that is widely used in the industry. It is a light metal with a density of 2.7g/cm^3 and a melting point of 600°C. Aluminum has good corrosion resistance and is also a good conductor of heat and electricity. There is an oxide layer (Al_2O_3) on the aluminum surface, which serves to protect it from corrosion. This layer is hard and has a high melting point of about 2050ºC. Because the melting point is much higher than that of the parent metal, the coating becomes a serious problem in the aluminum welding process, making it difficult to mix the base metal and filler metal and thus causing incomplete melting and resulting in defects in the form of fine holes in the weld (Ogunsemi et al., 2021). To remove the oxide layer (Al_2O_3), the surface to be welded must first be brushed with a steel brush. The use of noble gases during welding will prevent the oxide layer from forming again and prevent unwanted deposits from forming during the welding process.

Aluminum has light properties, good electrical, good resistance to corrosion, and heat conductivity, and is easy to form both through forming and machining processes. In nature, aluminum is a kind of oxide that is stable; thus, reduction method cannot be applied to aluminum that is usually applied in other metals. The reduction of aluminum only can be made by applying electrolysis method. In order to improve its mechanical strength, several elements can be added, such as Mg, Cu, Si, Zn, Mn, Ni, and so on, together or individually, and also to improve other good properties of aluminum, such as wear resistance, corrosion resistance, low coefficient of expansion, etc. Aluminum alloys can be divided into two groups, namely, sheet aluminum and cast aluminum. Aluminum (99.99%) has a specific gravity of 2.7 g/cm^3; above the magnesium (1.7 g/cm^3) and beryllium (1.85 g/cm^3) or about 1/3 of the specific gravity of iron or copper, a density of 2.685 kg/cm^3, and its melting point is 660ºC. Aluminum has the higher strength to weight ratio compared to steel. Its electrical conductivity is 60% more than copper, so it is used for electrical equipment. In addition, aluminum is a good conductor of heat and has good reflecting properties. Therefore, it is also used in engine components, heat exchangers, reflecting mirrors, chemical industry components, etc. (Irawan, 2015). The corrosion-resistant properties of aluminum are obtained from forming an aluminum oxide layer on the aluminum surface. This oxide layer is firmly and tightly attached to the surface and is stable (does not react with the surrounding environment) to protect the inside. Aluminum and its alloys have unique properties that make aluminum one of the simplest, most economical, and often applied metallic materials for various applications and it ranks second only to steel in its use as a structural metal (Davis, 2001).

The 6xxx series aluminum alloys contain Mg and Si in the right ratios to form Mg_2Si when heat treated. Even though it is not as strong as the 2xxx and 7xxx series aluminum alloys, the 6xxx series aluminum alloys have good properties, such as weldability, formability, corrosion resistance and machinability (Andersen et al., 2018).

In addition to the above properties, 6xxx series aluminum alloy is also very good for formability for forging and extrusion and good for high formability at ordinary temperatures. After processing, these alloys can be strengthened by heat treatment (Surdia & Saito, 1999). Tables 6.3–6.5 present the chemical composition of Al6061.

6.3 PRECIPITATION HARDENING

Precipitation hardening, also known as particle hardening, is a technique in heat treatment. It is a metal alloy hardening process by spreading fine particles evenly (Ataiwi et al., 2021). The strength and hardness of the metal can be increased by the formation of very small uniformly distributed particles that occur in the second

TABLE 6.3
Chemical Composition of Al6061

Element	Value %
Silicon (Si)	0.40–0.80
Iron (Fe)	0.70
Copper (Cu)	0.15–0.40
Manganese (Mn)	0.15
Magnesium (Mg)	0.80–1.20
Chromium (Cr)	0.04–0.35
Zinc (Zn)	0.25
Titanium (Ti)	0.15
Other (Each)	0.05
Other (Total)	0.15
Aluminum (Al)	Balance

Source: ASTM B221.

TABLE 6.4
Properties of Al6061 by Heat Treatment

Alloy	Composition	Temper	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation in 50 mm (%)
6061	1.0 Mg, 0.6 Si, 0.2 Cr	O	125	55	25
		T4	245	245	25
		T6	315	315	12
		T91	410	400	6

Source: Surdia & Saito, 1999.

TABLE 6.5
Mechanical Properties of Al-Mg-Si Alloy

Alloy	Condition	Tensile Strength (kgf/mm^2)	Creep Strength (kgf/mm^2)	Elongation (%)	Shear Strength (kgf/mm^2)	Hardness (Brinell)
6061	O	12.6	5.6	30	8.4	30
	T4	24.6	14.8	28	16.9	65
	T6	31.6	28.0	15	21.0	95

Source: Surdia & Saito, 1999.

stage of the original metric phase. Examples of alloys that are increased in hardness by precipitation hardening are aluminum-copper, copper-beryllium, copper-tin, and magnesium-aluminum. Some irons can also increase hardness through precipitation hardening (precipitation), but precipitation on iron is a different phenomenon, although the healing process is almost the same (Callister & Rethwisch, 2015).

For precipitation of supersaturated solid solutions, the basic requirement of a precipitation-hardening alloy system is that the solubility limit of the solid should decrease with decreasing temperature. The hardening heat treatment procedure is first subjected to a dissolution heat treatment at a high temperature and then rapidly cooled in water or other cooling media (Bishop & Smallman, 1999).

Rapid cooling can inhibit the phase separation so that at low temperatures, the alloy is in an unstable supersaturated state, but after a rapid cooling process, if the alloy undergoes "aging" treatment for a long time, a second phase precipitates. This precipitation occurs through the process of nucleation and growth, fluctuations in the concentration of dissolved material form clusters of small atoms in the lattice, which become nucleates. As the size of the sediment becomes finer as the temperature at which precipitation is lowered, and the alloy undergoes a significant increase in hardness which is associated with a critical dispersion of the precipitate. If aging is allowed to continue at a certain temperature, there will be coarsening of the particles (small particles tend to dissolve again, and large particles get bigger). Coarser particles gradually replace numerous particles that are finely dispersed with large dispersion distances. In this state, the alloy becomes softer, and the metal is said to be in the form of late aging (ASM International Handbook, 2001).

Heat treatment improves various mechanical and alloying properties because both dissolved atoms and point defects above equilibrium concentrations are maintained during this process. The rapid cooling process often eliminates lattice strain. The property that undergoes the most change is the electrical resistance which is usually a very large increase. On the other hand, the mechanical properties are not significantly affected (Bishop & Smallman, 1999).

Changes in the properties of the quenched material after aging are more pronounced. In particular, the mechanical properties undergo major modifications. For example, the tensile strength of duralumin (an aluminum alloy – 4% copper containing magnesium silicon and manganese) can be increased from 0.21 to 0.41 GN/m^2. Structural sensitive properties such as hardness, yield stress, and so on, of course, depend on the distribution of the phase structure; thus, such alloys experience softening when the finely dispersed precipitate hardens (Bishop & Smallman, 1999). The process of precipitation hardening or hardening can be divided into several stages (Callister & Rethwisch, 2015) as follows:

1. Solution heat treatment, which is heating the alloy above the solvus line.
2. Rapid cooling (quenching).
3. Precipitation heat treatment (aging), where the workpiece is heated to a temperature of T2, where the supersaturated solid solution begins to form the phase. This phase appears in the form of a fine precipitate which is dispersed and increases the strength of the metal. The heating duration depends on the formation stage of the optimum solidifying sediment.

There are several types of precipitation heat treatment (Callister & Rethwisch, 2015), which are as follows:

a. Natural Aging

In the natural aging process, the alloy does not experience heating. Only left at room temperature, in this process, it takes a long time, and the strengthening effect given is not so great. The precipitates in the matrix are still random (Triantafyllidis et al., 2015).

b. Artificial Aging

Artificial aging is a process where the alloy is heated to a certain temperature. At this stage, the precipitates are evenly distributed and form groups; at this stage the optimum strengthening effect can be produced (Abo Zeid, 2019).

c. Over-Aging

Over-aging is the aging process which is carried out for too long, or where the temperature is too high; at this stage, the precipitate and matrix are in balance. Over-aging can reduce the strength of the material that has been achieved previously (Liao et al., 2020).

6.3.1 NATURAL AGING

Natural aging is the aging for aluminum alloys which are treated with age hardening in a cold state. Natural aging occurs at room temperature between 15°C and 25°C and with a holding time of 5 to 8 days.

6.3.2 ARTIFICIAL AGING

Artificial aging is the aging for aluminum alloys that are treated with age hardening in a hot state. Artificial aging occurs at temperatures between 100°C and 200°C and with a holding time of 1 to 24 hours (Smith et al., 2015). After solution heat treatment and quenching, alloy hardening can be achieved in two ways: at room temperature (natural aging) or by precipitation heat treatment (artificial aging). Aging at room temperature will take a longer time, usually around 96 hours, to achieve a more stable strength, whereas if aging is done artificially, the aging time depends on the heating temperature. The higher the aging temperature, the shorter the time required to reach a certain strength (Cochard et al., 2017).

The use of the alloy hardening method depends on the type of alloy that one wants to increase the hardness. For alloys with a slow precipitation reaction, precipitation is always carried out at temperatures above the room temperature (artificial aging). In contrast, natural aging is sufficient for those with a fast precipitation reaction to obtain desired mechanical properties. In the aging process, the formation and growth of nuclei occur, leading to the formation of stable precipitates. The formation of this

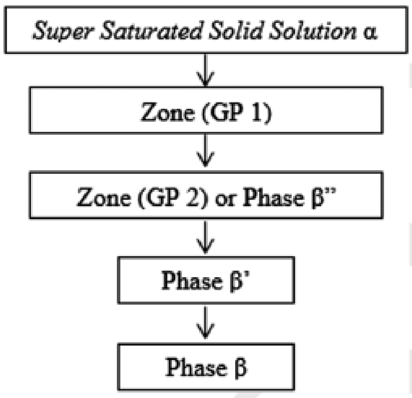

FIGURE 6.2 Phase change sequence in the artificial aging process (Based on Smith et al., 2015).

phase occurs through several phase transitions which also affect the mechanical properties of the alloy (Liao et al., 2020).

At the artificial aging stage in the age-hardening process, several variations of treatment can be carried out, which can affect the results of the age-hardening process. One of these variations is the artificial aging temperature variation. The artificial aging temperature can be set at the temperature at which the aluminum alloy crystallizes (150°C), below the crystallization temperature, or above the crystallization temperature of the aluminum alloy metal (Smith et al., 2015).

Taking the artificial aging temperature into consideration, a temperature between 100°C and 200°C will affect the level of hardness because there will be changes in the phase or structure of the artificial aging process. This phase change will contribute to the hardening of the aluminum alloy (Smith et al., 2015). The phase change in the artificial aging process is explained in Figure 6.2.

The explanation of the diagram in Figure 6.2 is as follows.

a. Supersaturated Solid Solution α

After the aluminum alloy passes through the solution heat treatment and quenching stages, a supersaturated solid solution will be obtained at room temperature. Under these conditions simultaneously, the atomic vacancies in thermal equilibrium at high temperatures remain in place. After cooling or quenching, the aluminum alloy becomes soft compared to its initial condition (Singh et al., 2018).

b. Zone (GP 1)

Zone (GP 1) is a precipitation zone formed by low aging or aging temperatures and is formed by the segregation of Mg-Si atoms in a supersaturated solid solution. Zone (GP 1) will appear in the early stage(s) of the artificial aging process. This zone is formed when the artificial aging temperature is below 100°C, and the Zone (GP 1) will not be formed at too high an artificial aging temperature. The formation of the Zone (GP 1) will begin to increase the hardness of the aluminum alloy (Smith et al., 2015).

If artificial aging is set at a temperature of 100°C, then the phase change stage is only until the formation of Zone (GP 1). The process of hardening from a supersaturated solid solution to forming a Zone (GP 1) is commonly referred to as first stage hardening (Yang et al., 2016).

c. Zone (GP 2) or β" Phase

After the artificial aging temperature passes 100°C and above, the β" or Zone (GP 2) phase will begin to appear. At a temperature of 130°C, a zone (GP 2) will be formed, and if the artificial aging holding time is fulfilled, the optimal hardness level will be obtained (Andersen et al., 2018).

Usually, the artificial aging process stops when a zone (GP 2) is formed. A fine intermediate phase is formed (β" precipitation) because after passing through the Zone (GP 2), the alloy will become soft again. If the artificial aging process continues until the β" or Zone (GP 2) phase is formed, it is called the second stage of hardening.

d. β' Phase

Suppose the aging temperature of the aluminum alloy is increased or the aging time is extended but the temperature remains constant. In that case, it will form precipitation with a stable crystal structure different from the phase. This phase is called the intermediate phase or β' phase. The formation of this β' phase can still contribute to an increase in the hardness of aluminum alloys. The increase in hardness that occurs in the β' phase is very slow (Chen et al., 2021).

e. β Phase

The holding time in artificial aging is one component that can affect the results of the overall age-hardening process. As with temperature, the holding time in the artificial aging stage will affect changes in the structure or phase of the aluminum alloys. If the temperature increases or the aging time is extended, then the β' phase changes to the β phase. If the phase is formed, it will cause the aluminum alloy to become soft again. Meanwhile, artificial aging holding time must be selected carefully (Rymer et al., 2021).

The relationship between aging time and aluminum alloy hardness begins with a phase change process that is formed in the precipitation hardening process, where the phase starts from a supersaturated solid solution after the quenching process. Then the alloy will experience aging or the appearance of new precipitates with time.

Several previous studies have developed the artificial aging method to improve the mechanical properties of aluminum alloy. Aluminum alloy AA7049 has experienced an increase in tensile strength due to multistage heat treatment (there are two times of heating). Multistage heat treatment, called the retrogression and re-aging process in this study, has been shown to increase the tensile strength of aluminum alloy AA7049 as evidenced by changes in the microstructure, where the sediment grows and fills grain boundaries (Ranganatha et al., 2013). Furthermore, previous study about the multistage-aging process on Al-Zn-Mg-Cu alloys with two heating showed a change in the microstructure, which means that the treatment influences the mechanical properties of the aluminum alloy (Mandal et al., 2020).

In 2017, the artificial aging method was developed into a multistage artificial aging method, with variations in the number of stages of aging (there are single stage, double stage, and triple stage aging) which are carried out to improve the mechanical properties of Al-Cu alloy, which through this treatment produces tensile strength and hardness. In other words, this treatment makes the Al-Cu alloy more resilient and increases its hardness (Tsamroh et al., 2017).

6.4 MICROSTRUCTURE CHANGE

Metals are generally constructed from a large number of crystals (the grains are referred to as grains of sand on a beach) consisting of one or more phases. Generally small, ranging from 10 μm to 1 μm, but there are also grains with sizes ranging from nm to cm; this microscopic metal arrangement is called a microstructure and can only be observed using a microscope. The microstructure of the grain size affects the strength of the material based on the grain size. Grain size cannot be used to control the strength of aluminum or its alloys, but it reduces the risk of hot cracking (Chen et al., 2021; García-rentería et al., 2020).

This study combines natural aging and artificial aging treatments intending to know how the microstructure changes Al6061 in natural aging treatment only with specimens receiving artificial aging treatment. Figure 6.3 shows the heat treatment diagram.

The research method used in this study is a laboratory experimental method which is intended to obtain descriptive data about changes in the microstructure of aluminum 6061 with natural aging, and natural aging treatments followed by artificial aging. Natural aging was conducted at room temperature for 7 days after the solution

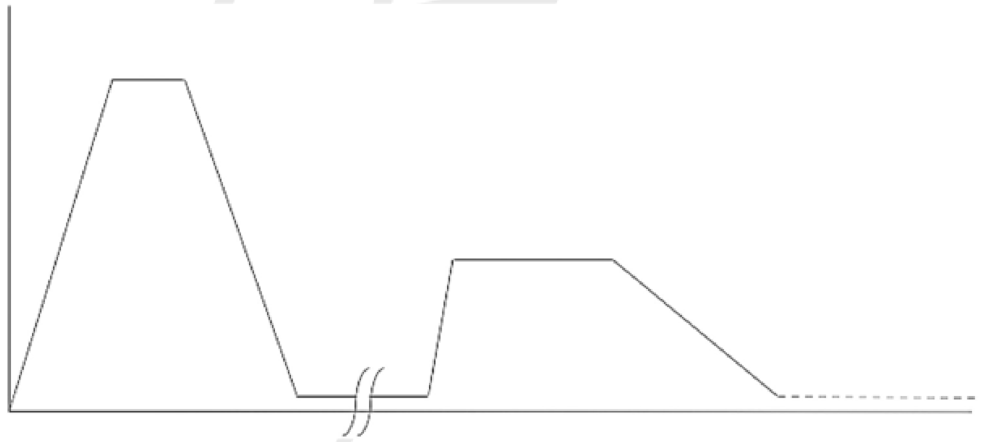

FIGURE 6.3 Heat treatment diagram.

heat treatment process at a temperature of 540°C and cooled rapidly using the mixture of water and dromus oil with a ratio of 1:1. The dependent variable of this study was microstructure change, while the independent variable of this study was the duration of holding time during the artificial aging process (2, 4, and 6 hours) with a temperature of 200°C. Figures 6.4–6.8 are the results of microstructure testing on Al6061 after getting natural–artificial aging treatment using an optical microscope. The microstructure of Al6061 was taken with a magnification of 200×. Changes in the microstructure of the material due to heat treatment can simply be seen in the grain size formed.

Precipitation that is spread evenly can increase the hardness and tensile strength of the material but causes the material to have brittle properties (Polmear, 2004). Based on the observation in Figure 6.4 it can be said that raw material of Al6061 has heterogeneous grain size and few residues on the surface of the specimen. Figure 6.5 is an image of the microstructure of Al6061 with natural aging treatment; it can be seen that the grain size is smaller than that of the raw material specimen (Figure 6.4). Figure 6.5 also shows the presence of residues formed on the surface of the specimen but not evenly distributed.

Figure 6.6 is a specimen of Al6061 with natural aging treatment followed by artificial aging treatment for 2 hours. From the figure, it can be seen that the grain size is more homogeneous, and the precipitates marked with black spots are seen more

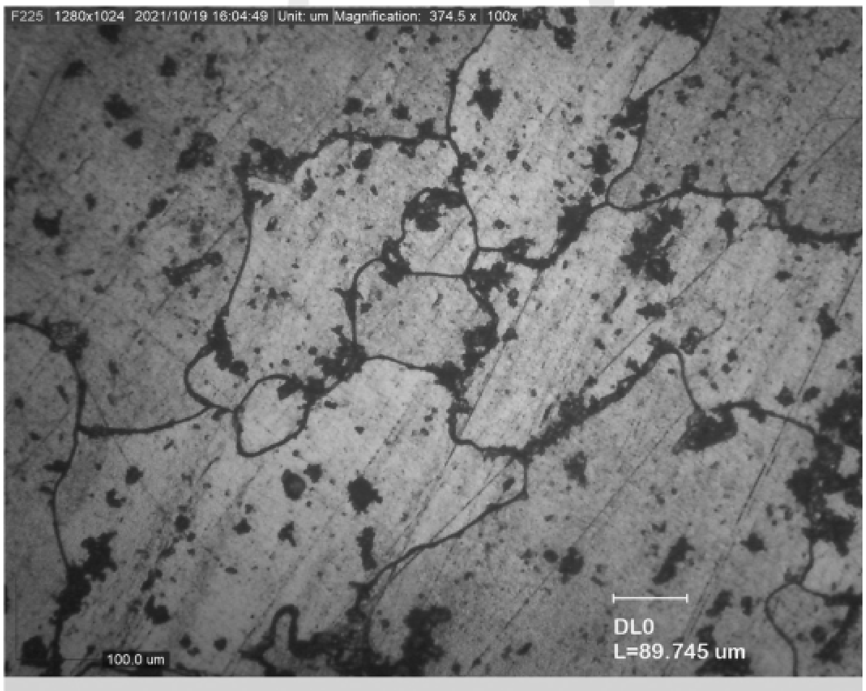

FIGURE 6.4 Microstructure of Al6061 without treatment (raw material).

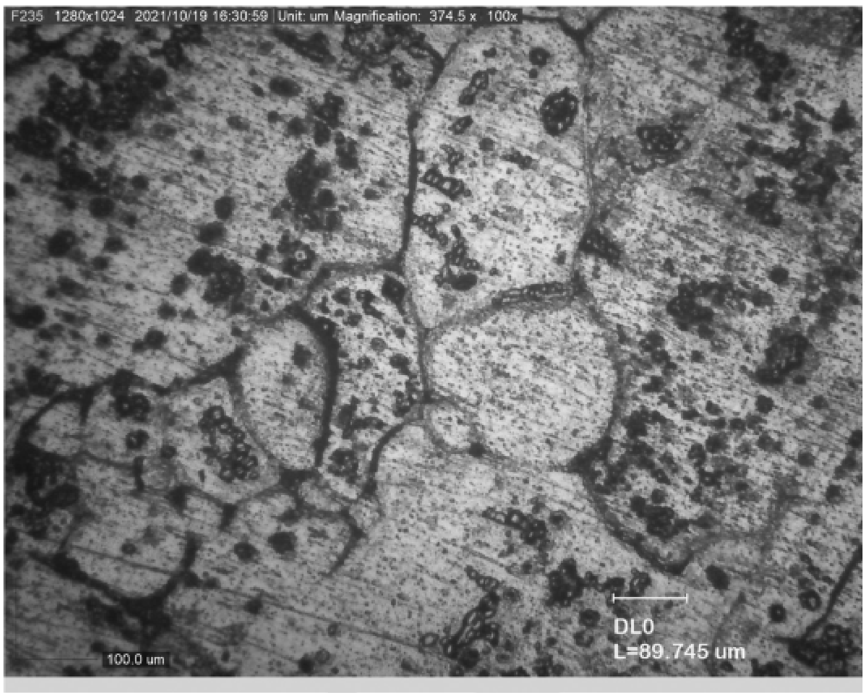

FIGURE 6.5 Microstructure of Al6061 with natural aging treatment.

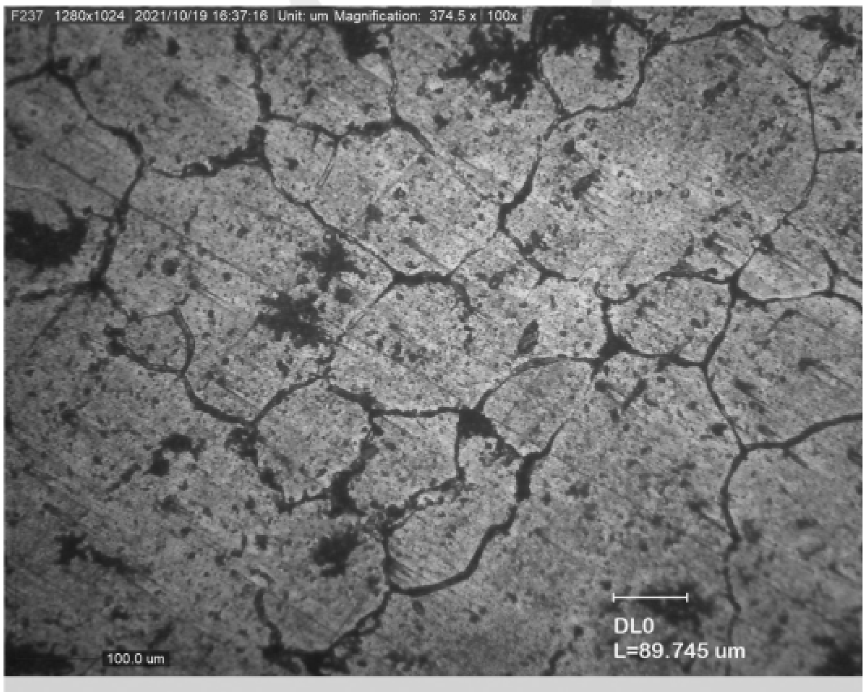

FIGURE 6.6 Microstructure of Al6061 with natural aging followed by artificial aging for 2 hours.

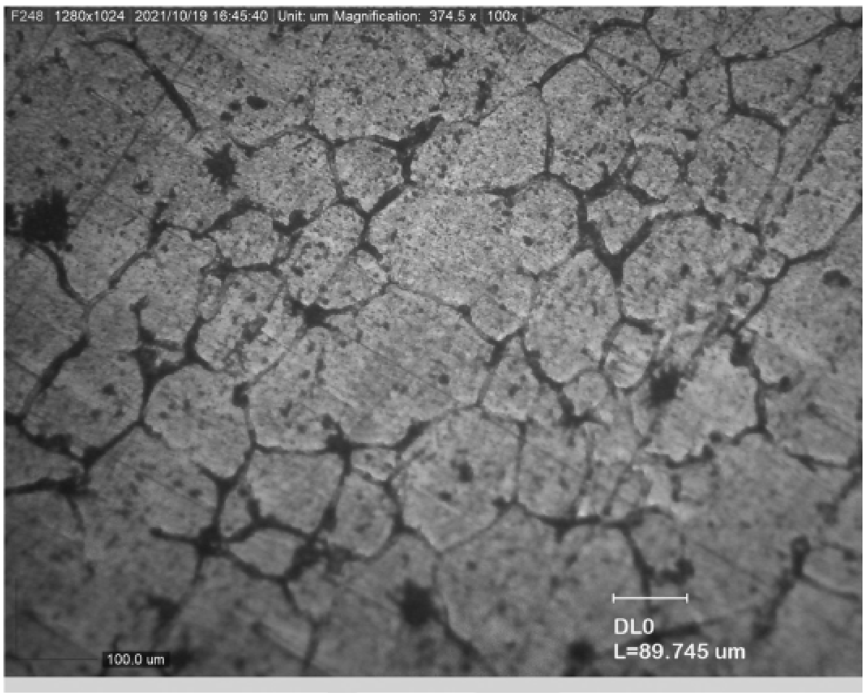

FIGURE 6.7 Microstructure of Al6061 with natural aging followed by artificial aging for 4 hours.

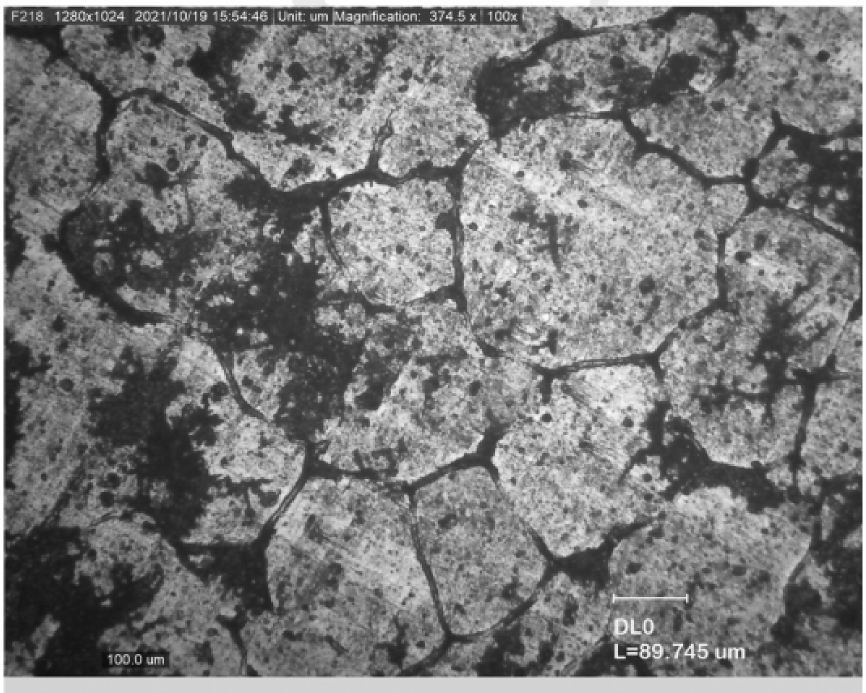

FIGURE 6.8 Microstructure of Al6061 with natural aging followed by artificial aging for 6 hours.

evenly on the surface of the specimen, both on the grainline and within the grain. Figure 6.7 is a specimen of Al6061 with natural aging treatment followed by an artificial aging process for 4 hours. The grain size of the specimen looks more homogeneous than the previous specimen, and the distribution of precipitates on the surface of the specimen also looks more even.

The last specimen shown in Figure 6.8 is a specimen with natural aging followed by artificial aging for 6 hours. In this specimen, it can be seen that the grain size has increased again; this is probably due to the material experiencing over-aging due to a longer holding time. The precipitate formed on Al6061 which is an Al-Mg-Si alloy is Mg_2Si; usually the precipitate formed is in the form of rods, needles, and laths (Andersen et al., 2018). The precipitation hardening process in Al-Mg-Si alloys can be identified in five stages:

$$\text{Supersaturated Solid Solution } \alpha \rightarrow \text{GP Zone 1} \rightarrow \beta'' \rightarrow \beta' \rightarrow \beta = Mg_2Si$$

The hardening of the precipitate should be coherent or semi-coherent, usually the aluminum matrix has coherent or semi-coherent precipitates (S. Coriell, 2000). Coherent precipitates have a small mismatch lattice with the metal matrix, and there is a tight interfacial tension lattice. A small coherence value will affect the hardness of the metal; the smaller the coherence value, the higher the hardness of the metal will be, and the phase formed is called the β' phase, whereas if the coherence value is not there at all, the hardness of the metal will decrease (this is called the β phase). The change from phase β" to phase β' occurs when the highest hardness number increases (Marioara et al., 2002).

This research proves that by applying artificial aging after natural aging, the β' phase can be identified which gives the material strengthening properties. It is proven by the amount of precipitate formed, which increases with increasing time with a constant temperature. In the artificial aging process, the β' phase is formed from the transition of GP zone 2 (β") to the β' phase, which leads to coherent precipitates against the Al-Mg-Si alloy matrix. The first aging peak, the transition from GP zone 2 (β") to the β' phase, increases significantly so that the hardness of the material in the β' phase increases. If the aging temperature or the aging time is extended but with a constant temperature, precipitation with a different crystal structure from the phase will be formed (Jin et al., 2018).

Changes in the microstructure of Al6061 in this study with natural aging and natural aging treatments followed by artificial aging were quite significant, and this can be seen from changes in grain size and the formation and distribution of precipitates (Mg_2Si). Based on Figure 6.5–Figure 6.8, the precipitate (Mg_2Si) which is the β' phase formed during the heat treatment process is shown in black spots that spread on the surface of the specimen. The precipitates formed are rods.

Based on the theory, the hardness number of a material will increase if the grain size gets smaller (Hajihashemi et al., 2016). To prove this, hardness testing was carried out on all Al6061 specimens (raw material, natural aging, and natural aging followed by artificial aging) 8 Hardness testing was carried out using a Rockwell Hardness Tester machine, with a major load of 100 kg, and a 1/8" steel ball indenter. The test was carried out on the E scale. Based on observations of the results of the microstructure

TABLE 6.6
Hardness Number of Al6061

Specimen	Holding Time (Hour)	Hardness (HR_E)
Raw material	-	98
Natural aging	-	84,6
	2	93,4
Artificial aging	4	106,8
	6	105,8

test, it can be seen that the specimens with relatively small and homogeneous grain sizes are those with natural aging treatment followed by artificial aging for 4 hours. Table 6.6 presents the result of hardness testing on Al6061.

From Table 6.6, it can be observed that specimens achieved the highest hardness number with natural aging treatment, followed by artificial aging treatment with a holding time of 4 hours with a figure of 106.8 HRE. Specimens that only received natural aging treatment had the lowest hardness number, which was 84.6 HRE. While the specimens underwent natural aging treatment followed by artificial aging with a holding time of 6 hours, the hardness number slightly decreased compared to specimens with a holding time of 4 hours, which was 105.8 HRE.

When compared with the hardness number of the raw material, it can be seen that the treatment with natural aging followed by artificial aging for 4 and 6 hours showed an increase in the hardness number. Thus, it can be estimated that the most optimum treatment to increase the hardness of Al6061 is natural aging followed by artificial aging with a holding time of 4–6 hours. However, specimens that only underwent natural aging treatment had the lowest hardness numbers. Thus, it can be concluded that the natural aging treatment has no significant effect on increasing the hardness of Al6061. The results of this study are almost the same as that of studies carried out by previous researchers, where natural aging did not affect the trend of increasing specimen hardness (Wardani et al., 2022). The holding time during artificial aging certainly affects the transformation/change of the microstructure of a material, which is also related to the hardness of the material (Rymer et al., 2021).

6.5 CONCLUSION

According to the discussion presented in this chapter, the following conclusions could be drawn:

1. The microstructure change on Al6061 was obtained due to the heat treatment process. The microstructure change is mainly affected by the holding time during artificial aging.
2. The best result of this study was a specimen that had been aged artificially for 4 hours after the natural aging process, which had a smaller grain size that was likely homogeneous.

3. The change of microstructure affected the hardness of Al6061, with the highest hardness number of 106.8 HRE (specimen that aged artificially for 4 hours). The increasing hardness was thought to be caused by the formation of Mg_2Si precipitates.

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REFERENCES

Abo Zeid, E. F. (2019). Mechanical and electrochemical characteristics of solutionized AA 6061, AA6013 and AA 5086 aluminum alloys. *Journal of Materials Research and Technology*, *8*(2), 1870–1877. https://doi.org/10.1016/j.jmrt.2018.12.014

Andersen, S. J., Marioara, C. D., Friis, J., Wenner, S., & Holmestad, R. (2018). Precipitates in aluminium alloys. *Advances in Physics: X*, *3*(1), 790–814. https://doi.org/10.1080/23746149.2018.1479984

Andoko, A., Prasetya, Y. A., Puspitasari, P., & Ariestoni, T. B. (2020). The effects of artificial-aging temperature on tensile strength, hardness, micro- structure, and fault morphology in AlSiMg. *Journal of Achievement in Materials and Manufacturing Engineering*. *98*(2), 49–55. https://doi.org/10.5604/01.3001.0014.1480

ASM International Handbook, V. 4. (2001). ASM handbook: heat treating. *Technology*, *4*, 3470. https://doi.org/10.1016/S0026-0576(03)90166-8

Ataiwi, A. H., Dawood, J. J., & Madhloom, M. A. (2021). Effect of precipitation hardening treatments on tensile properties, impact toughness, and microstructural changes of aluminum alloy AA6061. *Materials Today: Proceedings*. https://doi.org/10.1016/J.MATPR.2021.06.011

Bishop, R. J., & Smallman, R. E. (1999). *Modern Physic Metallurgy & Materials Engineering*. Elsevier. 256.

Callister, W., & Rethwisch, D. (2015). Materials science and engineering: an introduction. *Materials Science and Engineering*. https://doi.org/10.1016/0025-5416(87)90343-0

Chacko, M., & Nayak, J. (2014). Aging behaviour of 6061 Al-15 vol% SiC composite in T4 and T6 treatments. *International Journal of Chemical, Molecular, Nuclear, Materials and Metallurgical Engineering*, *8*(3), 195–198.

Chen, C., Yin, X., Liao, W., Xiang, Y., Gao, M., & Zhang, Y. (2021). Microstructure and properties of 6061/2A12 dissimilar aluminum alloy weld by laser oscillation scanning. *Journal of Materials Research and Technology*, *14*, 2789–2798. https://doi.org/10.1016/j.jmrt.2021.08.105

Cochard, A., Zhu, K., Joulié, S., Douin, J., Huez, J., Robbiola, L., Sciau, P., & Brunet, M. (2017). Natural aging on Al-Cu-Mg structural hardening alloys – investigation of two historical duralumins for aeronautics. *Materials Science and Engineering A*, *690*, 259–269. https://doi.org/10.1016/j.msea.2017.03.003

Coriell, S. (2000). Precipitation hardening of metal alloys. *A Century of Excellence in Measurements, Standards, and Technology*. CRC Press 14–15.

Davis, J. R. (1993). *Aluminum and Aluminum Alloys*. ASM International.

Davis, J. R. (2001). Aluminum and Aluminum Alloys. *Light Metals and Alloys*, 66. https://doi.org/10.1361/autb2001p351

Flores, F. U., Seidman, D. N., Dunand, D. C., & Vo, N. Q. (2018). Development of high-strength and high-electrical-conductivity aluminum alloys for power transmission conductors. In: Martin, O. (eds) *Light Metals 2018*. TMS 2018. The Minerals, Metals & Materials Series. Springer, Cham. https://doi.org/10.1007/978-3-319-72284-9_34

García-Rentería, M. A., Torres-Gonzalez, R., & Cruz-Hern, V. L. (2020). First assessment on the microstructure and mechanical properties of gtaw-gmaw hybrid welding of 6061-t6 AA. *Journal of Manufacturing Processes*, *59*(October), 658–667. https://doi.org/10.1016/j.jmapro.2020.09.069

Hajihashemi, M., Shamanian, M., & Niroumand, B. (2016). Microstructure and mechanical properties of Al-6061-T6 alloy welded by a new hybrid FSW/SSW joining process. *Science and Technology of Welding and Joining*, *21*(6), 493–503. https://doi.org/10.1080/13621718.2015.1138019

Indonesia, K. P. R. (2018). *Kemenperin Kejar Produksi Aluminium Nasional 2 Juta Ton Tahun 2025*. Kementerian Perindustrian Republik Indonesia.

Irawan, Y. S. (2015). Aluminium dan Paduannya – Material Teknik (Aluminium dan Tembaga Paduan). Chapter 12. *Material teknik. seri*, 1–8.

Jin, S., Ngai, T., Zhang, G., Zhai, T., Jia, S., & Li, L. (2018). Precipitation strengthening mechanisms during natural ageing and subsequent artificial aging in an Al-Mg-Si-Cu alloy. *Materials Science and Engineering A*, *724*, 53–59. https://doi.org/10.1016/j.msea.2018.03.006

Junkers, H. (2014). *Duralumin and the Origins of Rivets*. Duralumin and the Origins of Rivets - Airstream

Lee, Y. S., Koh, D. H., Kim, H. W., & Ahn, Y. S. (2018). Improved bake-hardening response of Al-Zn-Mg-Cu alloy through pre-aging treatment. *Scripta Materialia*, *147*, 45–49. https://doi.org/10.1016/j.scriptamat.2017.12.030

Liao, X., Kong, X., Dong, P., & Chen, K. (2020). Effect of pre-aging, over-aging and re-aging on exfoliation corrosion and electrochemical corrosion behavior of Al-Zn-Mg-Cu alloys. *Journal of Materials Science and Chemical Engineering*, *8*(2), 81–88. https://doi.org/10.4236/msce.2020.82008

Mandal, P. K., John Felix Kumar, R., & Merrin Varkey, J. (2020). Effect of artificial ageing treatment and precipitation on mechanical properties and fracture mechanism of friction stir processed $MgZn_2$ and Al_3Sc phases in aluminium alloy. *Materials Today: Proceedings*. https://doi.org/10.1016/J.MATPR.2020.10.389

Marioara, C. D., Andersen, S. J., Jansen, J., & Zandbergen, H. W. (2002). The GP-Zone to β Transformation in the Al-Mg-Si System. *Microsc. Microanal.*, *8*(2), 1444–1445.

Nulhaqem, L., & Abdul, B. I. N. (2013). *Influence of Heat Treatment on the Microstructure and Mechanical Properties of 6061 Aluminum Alloy* . Bachelor Thesis. Universiti Malaysia Pahang. June. CD7752.pdf (ump.edu.my)

Ogunsemi, B. T., Abioye, T. E., Ogedengbe, T. I., & Zuhailawati, H. (2021). A review of various improvement strategies for joint quality of AA 6061-T6 friction stir weldments. *Journal of Materials Research and Technology*, *11*, 1061–1089. https://doi.org/10.1016/j.jmrt.2021.01.070

Polmear, I. (2004). Aluminium alloys – a century of age hardening. *Materials Forum*, *28*, 1–14. https://doi.org/7F6104775CD4BCE9E9D087602166B700

Prudhomme, M., Billy, F., Alexis, J., Benoit, G., Hamon, F., Larignon, C., Odemer, G., Blanc, C., & Hénaff, G. (2018). Effect of actual and accelerated ageing on microstructure evolution and mechanical properties of a 2024-T351 aluminium alloy. *International Journal of Fatigue*, *107*(October 2017), 60–71. https://doi.org/10.1016/j.ijfatigue.2017.10.015

Puspitasari, P., Puspitasari, D., Sasongko, M. I. N., Andoko, & Suryanto, H. (2016). Tensile strength differences and type of fracture in artificial aging process of duralium against cooling media variation. *AIP Conference Proceedings*, *1778*, 0–4. https://doi.org/10.1063/1.4965746

Rajasekaran, S., Udayashankar, N. K., & Nayak, J. (2012). *T4 and T6 Treatment of 6061 Al-15 Vol. % SiC P Composite*. International Scholarly Research Network. 2012. Article ID 374719. 1–5 https://doi.org/10.5402/2012/374719

Rambabu, P., Prasad, N. E., & Kutumbarao, V. V. (2017). *Aerospace Materials and Material Technologies*. Springer. https://doi.org/10.1007/978-981-10-2143-5

Ranganatha, R., Anil Kumar, V., Nandi, V. S., Bhat, R. R., & Muralidhara, B. K. (2013). Multi-stage heat treatment of aluminum alloy AA7049. *Transactions of Nonferrous Metals Society of China (English Edition)*, *23*(6), 1570–1575. https://doi.org/10.1016/S1003-6326(13)62632-1

Rymer, L. M., Winter, L., Hockauf, K., & Lampke, T. (2021). Artificial aging time influencing the crack propagation behavior of the aluminum alloy 6060 processed by equal channel angular pressing. *Materials Science and Engineering: A*, *811*, 141039. https://doi.org/10.1016/J.MSEA.2021.141039

Singh, R., Sachan, D., Verma, R., Goel, S., Jayaganthan, R., & Kumar, A. (2018). Mechanical behavior of 304 Austenitic stainless steel processed by cryogenic rolling. *Materials Today: Proceedings*, *5*(9), 16880–16886. https://doi.org/10.1016/j.matpr.2018.04.090

Smith, W. F., Javad, H., & Prakash, R. (2015). *Introduction to Materials Science and Engineering*. McGraw-Hill Education

Surdia, T., & Saito, S. (1999). *Pengetahuan Bahan Teknik*. Pradnya Paramita. 372.

Triantafyllidis, G. K., Pukhalska, N. V., & Zagkliveris, D. I. (2015). Natural aging effects on the solutionizing heat treatment process of the A6060 Al alloy as-cast billets for profile production. *Materials Sciences and Applications*, *6*(2). *February*, 111–116.

Tsamroh, D. I. (2021). Comparison finite element analysis on duralium strength against multi-stage artificial aging process. *Archives of Materials Science and Engineering*, *109*(1), 29–34. https://doi.org/10.5604/01.3001.0015.0512

Tsamroh, D. I., Puspitasari, P., Andoko, A., Permanasari, A. A., & Setyawan, P. E. (2018). Optimization of multistage artificial aging parameters on Al-Cu alloy mechanical properties. *Journal of Achievements in Materials and Manufacturing Engineering*, *87*(2), 62–67. https://doi.org/10.5604/01.3001.0012.2828

Tsamroh, D. I., Puspitasari, P., Andoko, Sasongko, M. I. N., & Yazirin, C. (2017). Comparison study on mechanical properties single step and three step artificial aging on duralium. *AIP Conference Proceedings*, *1887*. https://doi.org/10.1063/1.5003553

Wardani, I. P., Setyowati, V. A., Suheni, I., Saputro, B., Teknik, J., Institut, M., Adhi, T., & Surabaya, T. (2022). Pengaruh natural aging Sebelum Proses artificial aging Terhadap Sifat Mekanik Aluminium 6061. *Seminar Nasional Sains Dan Teknologi Terapan VIII*, *2020*, 109–114.

Woodford, C. (2021). *Aluminum*. www.explainthatstuff.com/aluminum.html.

Yang, R. Xian, Liu, Z. Yi, Ying, P. You, Li, J. Lin, Lin, L. Hua, & Zeng, S. Min. (2016). Multistage-aging process effect on formation of GP zones and mechanical properties in Al-Zn-Mg-Cu alloy. *Transactions of Nonferrous Metals Society of China (English Edition)*, *26*(5), 1183–1190. https://doi.org/10.1016/S1003-6326(16)64221-8

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