# THE EFFECT OF HOLD-MELT TIME OF MICRO-REGIME PRECIPITATION SIZE AND HARDNESS IN AI-Cu ALLOY

*Wahyono Suprapto*⊠ Department of Mechanical Engineering<sup>1</sup> wahyos@ub.ac.id

**Yudy Surya Irawan** Department of Mechanical Engineering<sup>1</sup>

*Sudjito Suparman* Department of Mechanical Engineering<sup>1</sup>

**Muhammad Rafi Amrullah** Department of Mechanical Engineering<sup>1</sup>

**Pratama Auliasyah** Department of Mechanical Engineering<sup>1</sup>

*Aditya Rafi Ramdhani* Department of Mechanical Engineering<sup>1</sup>

<sup>1</sup>Brawijaya University 167 MT Haryono str., Malang, Indonesia, 65145

Corresponding author

#### Abstract

This study aims to control the characterization and mechanical properties of smelting Al-Cu Alloy through Hold-Melt Time. This research uses aluminum scrap and copper wire scrap to produce quality as-cast ingots, clean the environment, and increase waste utilization. Copper melting point of 1083 °C is immersed in molten aluminum at a temperature of 900 °C for 10–30 minutes causing copper to dissolve in aluminum due to smelting events based on diffusion phenomena. Parameters of temperature and immersion time of copper in molten aluminum in this study are expressed by hold-melt time. In the copper aluminum alloy trade, commonly called Duralumin, it is commonly used for impact loads and is heat-treatable. Resistance to cryogenic temperatures, in the future Duralumin has the potential to replace stainless steel. This study used an electric resistance furnace with the specifications for smelting aluminum 3 kg, electric power 2.5–3.0 kW, electric voltage 220 Volts, maximum temperature 1000 °C. It had been conducted an experiment where copper had been melted under its melting point in duralumin ingot casting. In this study, copper pieces were soaked in liquid aluminum with temperature of 900 °C. After 10–30 minutes of holding melt, the soaked copper became Al-Cu alloys and was called molten Duralumin. After the molten duralumin had been cleaned from dross, it was poured into ingot casting. From specific weight test, more soaking time of the copper in liquid aluminum caused specific weight of ingot duralumin increase from 47.08 % to 57.56 % and its hardness increase from 93 to 113 BHN. This study contributes on melting energy saving and improves the characteristic and hardness of ingot aluminum type 2xxx.

Keywords: ingot as-cast, smelting Al-Cu alloy, holding time, atomic diffusion, submerged aluminium melt.

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#### 1. Introduction

Aluminum alloys have more benefits compared to pure aluminum. They have lower melting point, higher mechanical characteristics, and specific characteristics such as corrosion resistant and tear resistant. Pure aluminum is very soft where its tensile strength is between 90 and 140 N/mm<sup>2</sup>. Aluminum alloys are very strong where it is similar to structural steel. Generally, aluminum alloys are used for mechanical component because of their high specific strength, [1]. However, high melting point of copper (1200 °C) as the main material for the process still becomes a problem in producing aluminum especially duralumin. This is different from non-ferrous metal elements like

zinc, magnesium, and aluminum which have low melting temperature and will evaporate when burned with high temperature. [2] states that melting aluminum at high temperature will increase the amount of dross and decrease melting efficiency. Scientifically, the base of metal melting experiment cannot be separated from diffusion process, which is controlled by time and temperature.

Besides time and temperature, quality and quantity of raw material also control the quality of aluminum alloys (Al-Cu alloys) through compound formation and grain geometry. In this study, chemical and kinetic equilibrium and reaction heat and heat loss were used to analyze Al-Cu alloys characteristics. Based on metallurgy science, one of the ways to identify the metal characteristics is to identify its shape, size, amount, and texture distribution. Microstructure in casting metal is classified based on three types which are:

1) regular;

2) complex regular;

3) irregular.

Regular microstructure contains lamellar or fibrous with branches inside the matrix. Thus, it is appropriate to use as a composite. In regular microstructure, there is random repetition of regular shape. Microstructure irregular is similar with complex regular microstructure with 2 phases of random orientation. [3] states that structure and crystallization characteristic of casting metal are influenced by condition of chemical elements change and parameter stimulation of pouring structure.

Al-Cu alloys has some advantages. They are heaTable and have good corrosion resistance, electric conductivity, high fracture toughness, fatigue resistance, high comparation of strength towards specific weight (in condition where O: 288 MPa and T4: 713 MPa). In terms of quantity and quality, ingot Al-Cu alloys are highly needed in manufacture industry both as raw material for casting, forging, and cutting. [4] conclude that copper and magnesium are main alloys of type 2000 and 5000 and each is usually added into aluminum casting with concentration of 4 % to 8 % wt. However, high casting temperature causes hydrogen gas dissolved in aluminum increase and cause porosity. Besides that, high melting and pouring temperature also increase specific volume gradient of liquid and solid metals, which can cause shrinkage. [5] state that porosity and pores morphology are influenced by complexity of casting process and alloy elements.

Generally, precipitant is generally found in grain boundary of alloys including Al-Cu alloys, which are formed because of metal elements, and cooling history. Higher temperature and faster grain growth rate are potential to cause cracks. Important result of controlled low temperature melting may prevent grain growth, which guarantees the quality of a product. This is in line with the increase of demand of aluminum alloys with higher strength, ductility, and toughness. Nowa-days, many studies control grain size in order to improve ductility and toughness by reducing stress concentration on grain boundary, which produce cracks. Precipitation can promote extremely fine sediment particle dispersion by preventing grain growth through second phase particle. Higher temperature, faster grain growth rate, and the lack of fine sediment may prompt rough grain and susceptibility towards surface crack especially under oscillation boundary [6]. Thus, it can be concluded that producing alloys in low temperature can control casting quality.

The melting process does not only influence the energy and the casting cost but also can be used to control quality, composition, and physical and chemical characteristics of a casting product, [7]. According to [8] induction furnace provides quick melting cycle around 30–35 minutes with automatic stirring effect and very high melting power which is 0.7–1 kWh/kg Al. The crisis of energy and raw material happens in manufacture industry and this triggers the industrialists to be selective and efficient in using resources. The estimation of melting energy consumption is very important for casting manufactures in order to reduce production cost by improving the melting process such as replacing coal with electricity. Aluminum melting at low temperature gives two benefits. First, in terms of raw material, it reduces hydrogen dissolution, low melting temperature element evaporation, aluminum sticking on the crucible, and dross formation. The second benefit is that it increases the permanent mold's durability.

Using the use of hold-melt time, this work seeks to regulate the characterization and mechanical properties of smelting Al-Cu Alloy. Chemical composition, microstructure and hardness analysis were conducted to determine the microstructure and mechanical properties of the alloy.

# 2. Materials and Methods

This study used aluminum and copper as raw material based on optical emission spectrometry (OES) test of their chemical elements as displayed in **Table 1**. Aluminum and copper were cut, cleaned from impurity elements (oil and dust) and scaled. The weight of each is 125 g and 5 g.

# Table 1

The Result Test of Spark Spectrometry of the Research Materials

Names of the Material	Elements [%]							
Ingot Aluminum (main material)	Al	Cu	Mn	Si	Mg	Zn	Fe	
	99.500	0.006	0.108	0.280	0.008	< 0.005	< 0.001	
	Ti	Cr	Ni	Pb	Sn	_	_	
	0.001	0.029	0.023	< 0.002	< 0.010	_	_	
Copper pipe (alloy material)	Al	Cu	Mn	Si	Mg	Zn	Fe	
	98.500	0.100	< 0.005	< 0.005	0.011	0.003	0.358	
	Zn	Со	Si	Mg	Cr	Al	Bi	
	0.280	0.042	0.245	0.001	0.002	< 0.002	0.363	

The aluminum and copper cuts melting in this research used Electrical Induction Furnace (EIF) as seen in **Fig. 1**. The melting process was started by furnace heating (ladle) with temperature up to 450 °C and then the aluminum cuts, which had been preheated with temperature 125 °C, were put into the ladle. When EIF temperature reached 650 °C, the aluminum in the ladle melted. Next, when the molten aluminum's temperature in the ladle reached 900 °C, the copper cuts, which had been preheated with temperature 125 °C, were put into the molten aluminum. After 10 minutes of *hold-melt* time, liquid Al-Cu alloys was cleaned from dross and then the molten Al-Cu alloys from the ladle was poured gravitationally into permanent mold (ingot) which had been preheated with temperature 250 °C. In this study, *hold-melt* time is amount of time where the element is being held under its melting point in certain amount of time. Next, after Al-Cu alloys had solidification, the ingot (specimen) was taken out from the mold. Next melting process as free variables in the research was performed with the same procedure with *hold-melt* time of 15 minutes, 20 minutes, 25 minutes, and 30 minutes. **Fig. 2**, *a* shows ingot permanent mold and ingot specimen used in the study.

EIF specification: Capacity of molten aluminum is 3 kg; maximum temperature for the operation is 1000 °C, and electric current and voltage are 12–14 Ampere and  $\pm$ 220 volt.

After the process of Al-Cu alloys ingot melting (specimen) was completed, the next stage was to conduct a measurement on dependent variable in the study. The preparation and the examination covered:

- a) chemical element;
- b) mass density;
- c) micro structure;
- d) hardness of duralumin ingot.



**Fig. 1.** Electrical Induction Furnace in the Research: *a* – Overview of Overview of Electrical Induction Furnace; *b* – Molten Duralumin in Electrical Induction Furnace ladle





**Fig. 2.** Permanent ingot mold in the research: a – The Design of permanent ingot mold; b – Al-Cu alloy ingot

Each was tested by OES, pycnometry, optical and scanning electron microscopy, and Brinell hardness test. Specification of the test instruments in the research is as follows:

a. Optical emission spectrometry (OES).

The content of the main materials (aluminum and copper) and Al-Cu alloys ingot can be found out by using OES tool. The mechanism of OES tool is to burn the specimen with electrode until the flame appears. Next, the flame is elaborated into light spectrum ( $\lambda$ ), intensity of each type, and percentage of specific weight.

b. Pycnometer.

Pycnometry test was performed to know the apparent density of Al-Cu alloys ingot (specimen). The test was conducted by measuring the specimen's weight in the air and in water. Analytically, the specific weight of specimen is stated in equation:

$$\gamma_D = \frac{W_a}{W_a - (W_{wb} - W_b)} \times \gamma_w,\tag{1}$$

where  $W_a$  – weight specimen in air [g],  $W_{wb}$  – weight specimen and bucket in water [g],  $W_b$  – weight bucket in air [g],  $\gamma_D$  – mass density of specimen [g/cm<sup>3</sup>],  $\gamma_W$  – mass density of water [g/cm<sup>3</sup>].

Fig. 3 shows the installation of pycnometer instrument which had been used in the study with specification: Accuracy: 0.001, Maximum weight of the specimen: 300 gr.



Fig. 3. Pycnometer instrument

c. Optical and Scanning Electron Microscopy.
OM:
Magnification; 50x, 100x, 200x, 500x, and 1000x
Model BX41RF-LED
100–120/220–240 V~, 0.12/0.08 A 50/60 Hz
Brand: Olympus, Made in Japan
Etza which was used to obtain the grain geometry (size and shape) and porosity morphology used:

1 % NaOH+4 % KMnO<sub>4</sub>+95 % H<sub>2</sub>O [9] and 5 % HF+95 % H<sub>2</sub>O.

d. Multi-meter pliers.

UNI-T UT210E Pocket Size Digital Multimeter Pliers Shape AC/DC 2A/20A/100A Current Voltage Tester Meter Pen Multi Meter. Measuring Voltage Range: 2V/20V/200V/600V. It was used to measure electric current and voltage on the operation of the melting furnace.

e. Brinell Hardness Testing.

The Brinell hardness test for the cast samples was performed on diameter of  $15 \times 10$  mm test pieces. Both grinding and polishing were carried out starting with coarse filing and finishing using a motor-driven emery belt. A load of 125 kg was applied on the test piece for 15 seconds and the diameter of the impression measured.

# 3. Result and Discussion

The test result in this study is categorized into:

a) chemical elements and specific weight;

b) microstructure;

c) hardness which can be explained as follows:

d) chemical Elements and Specific weight.

Holding time variation in this study had produced different percentage of Al-Cu alloy elements as shown in **Table 2**.

#### Table 2

The Result of the Experiment on Copper Dissolution in Molten Aluminum. Reference: Optical Emission Spectrometry Result (2022)

Hold	old Elements [weigh %]									Donsity [g/om <sup>3</sup> ]	
Melt [minutes]	Mg	Cu	Zn	Si	Fe	Mn	Ti	Ni	Cr	Al	Density [g/cm]
60	0.270	2.495	0.268	0.696	3.325	0.072	0.005	0.002	0.003	Rem	2.73
90	0.261	3.490	0.260	0.508	3.229	0.076	0.010	0.003	0.003	Rem	2.77
120	0.239	3.893	0.245	0.346	3.120	0.098	0.013	0.004	0.003	Rem	2.80
150	0.214	4.490	0.045	0.290	3.101	0.106	0.017	0.004	0.003	Rem	2.89

The difference was caused by the lost elements which was cause by the evaporation (low melting point such as Mg and Zn) was bound by ladle wall (reactive like Fe). It may be caused by the difference of elements which unites with aluminum such as Cu and Mn, and elements with high melting point (Ti, Ni, and Cr). Those elements are influential to liquid and solid Al-Cu alloys characteristic which in the end influences the physical characteristic (specific weight) and metallographic characteristic (phase and microstructure).

In molten condition, Fe in Al-Cu alloy accelerates copper phase dissolution [10] declares that: 1) in liquid, aluminum and copper including metal are easy to absorb hydrogen gas from their surroundings such as air moisture, grease and oil, furnace water content, and others;

2) solid Al-Cu alloys has a combination of high strength and high temperature. Besides that, elements with low melting temperature (Mg, Zn, and other substances) evaporate which causes the percentage of Mg and Zn decreases. Referring to the mass balance system in the system of mixture and alloys, percentage decrease of an element causes the percentage increase of the other element.

Generally, **Table 2** shows an increase tendency of specific weight along with the increase of hold-melt time. The result is in line with Second Fick Law and mass balance, which state that the contact time between elements at high temperature can control diffuse depth and the alloys' specific weight and it is the result of fraction times with the elements contained in it. Analytically, both laws are formulated as follows:

a) hold-melt time:

$$C_{(x)} = C_o + \left(C_s - C_o\right) \left[1 - \left(\frac{X}{2\sqrt{Dt}}\right)\right],\tag{2}$$

where C = concentration of the element, erf = error function, D = diffusion coefficient, t = contact time.

The ideal boundary conditions from equation (2) can be simplified to equation 3:

$$\frac{C_s - C_x}{C_s - C_o} = erf\left(\frac{X}{2\sqrt{Dt}}\right).$$
(3)

Where is  $C_{(x)}$  – concentration of the Copper in distance X [% weight];  $C_o$  – concentration of the Copper in an atmosphere [% weight];  $C_s$  – concentration of the Copper in surface contact [% weight]; X – distance [cm];  $(X/2\sqrt{Dt}) = erf$  – error function; D – diffusion coefficient [cm<sup>2</sup>/s]; t – contact time [s];

b) the balance of each element is stated in:

$$\sum_{i=1}^{j=m} m_i (\%X) = \sum_{j=1}^{j=m} m_j (\%X) + \Delta m_x.$$
(4)

#### Input = output + accumulation.

Where is  $m_i$  – each type of material which goes into the furnace [g],  $m_j$  – each type of product made [g], (% X) the percentage of element's weight of raw material or product,  $\Delta m_x$  – mass of X which is accumulated in the process [g]. It is noted that if the furnace is eroded, negative  $\Delta m_x$  in steady zero  $\Delta m_x$ .

The influence of Al-Cu alloys homogenization with copper content which is beyond the solution boundary increases the strength because the strain induced into aluminum grates of FCC crystal is as a result of copper atom insertion in order to reduce atom emptiness. This happens because copper solution in  $\alpha$ -Al decreases drastically at room temperature under thermodynamical balance condition. The addition of fine material and casting parameter optimization may prevent the growth of columnar crystal on its end and form structure uniformity of ingot cross section. The extra fine micro structure depends on the availability of the heterogeneous core potential which is soluted on solidification condition (colling rate). Generally, the process of casting with micro structure uniformity and chemical composition increases mechanical and physical characteristics before deformation process is conducted. According to Dolic and Zovko [8], solidification and homogenization process on EN AW-5083 alloy correlate with mechanical characteristics and the amount of grain per width.

**Table 2** also displays the increase of Fe in Al-Cu alloys and this happens because metal elements with low temperature (like Zn and Mg) evaporate in molten Al-Cu alloy. Solid strengthening solution (SSS) and secondary intermetallic phase shaping are two kinds of mechanism, which

have created strength increase in Al-Cu alloys on pre-ageing stage. SSS happens when the atom of any alloy element has different atomic radius. This creates dimension change or grates suspension. Thus, more forces are needed to overcome bonds between atoms and dislocation slip is prevented. The strengthening level depends on two factors which are atom size difference and the amount of strange atoms.

Copper dissolution for 15 to 30 minutes in liquid aluminum at 900 °C produces liquid of binary Al-Cu alloys. In solution cooling mechanism, each solluted copper and aluminum forms segregated grain boundary and grain body. Generally, the metal alloy solution which is segregated at grain boundary is called secondary phase (precipitant) and the grain body is called primary phase. The more the copper dissolved is, the bigger the grain boundary is and the grain body becomes smaller. Formerly, the characteristic of aluminum and copper was very soft. However, after it has become Al-Cu alloys, it is much more strong and tough. Some types' characteristic can be altered through cooling and heating treatment but there are only few non-ferrous alloys (copper, magnesium, manganese, silicon, and zinc) that can harden with the same controlled heating treatment conducted on carbon steel. The former element in solid solution gives strengthening effect when the element in it tends to increase along with the increase of atomic nucleus difference (Al) and dissolved substance (alloy elements), [11].

The copper content contributes important information about the microstructure formation, precipitation, and mushy zone in solidification process of Al-Cu alloys. The mechanism of microstructure formation and precipitation of duralumin casting are generally influenced by three main factors which are: compound component, turbulence effect, and surrounding air humidity [12] explains that alloy element influences porosity formation through three mechanisms. First, alloy element can alter the range of alloy freezing if the freezing range decreases, mushy zone in material solidification decreases, and the next is that porosity decreases. Second, alloy element can form dendritic intermetallic during the solidification and porosity can be formed along the intermetallic dendrite. Third, alloy element can form low melting point phase and cannot fill if the solidification is between dendrites.

After the melting, the molten Al-Cu alloys is poured and the molten metal which has contact with the mold wall produces thin skin on the mold interface/metal (alloy) which increases around the mold interface/metal during the solidification. Mold surface roughness will cause the decrease of heat transfer coefficient on metal/mold interface. Generally, the solidification level depends on the heat transfer rate from melting into mold, metal thermal characteristics, solidification level that is controlled by the mold geometry and mold physical characteristics. The darker round zone is  $\alpha$ -Al which is surrounded (network) by soft phase called precipitant which is full of copper. This microstructure is noted as aluminum alloy phase type 2000. The longer the dissolution time of precipitant network copper, which contains 4 % of copper in super-saturated solid solution, is, the harder the round precipitant network is. The desired  $\alpha$ -Al grain shape is round and disconnected even though the precipitant appears on the grain boundary (also vertical).

The desired precipitant with comparation is round and disconnected even though the two features appear on the grain boundary (also vertical). The shrinkage defect seriously limits the material strength especially if shear forces are applied on the same zone as defects, which are more or less parallel. The defect is probably caused by the low ratio of alloy volume on the mold surface area, which gives high sub-cooling level. Pre-heating the mold and reducing cooling rate will help preventing shrinkage defects. Besides, this type of alloys is usually used in forging condition. A process like roll forging and post production will help breaking the parallel structure of seeds and defects and also gives more strength which is prompted by work enforcement. Al-Cu alloys contain 5.8–6.8 % of copper and small amount of other alloys elements like manganese, titanium, and vanadium. The process of precipitation enforcement heating treatment is used in Al-Cu alloys to increase its strength and hardness. In AA2219, copper atom is an alloy element, which helps the precipitant formation. The forming element of solid solution gives strengthening effect when the element in the solution tends to increase along with the increase of atomic nucleus difference (Al) and dissolved substance (alloy element);

c) microstructure.

Fig. 4 shows the transformation of copper solid solution in aluminum, which is around 5.67 % at temperature of 548 °C, and the dissolution keeps decreasing until 0.4 % Cu at room

temperature. Copper rejection of  $\alpha$ -Al will form secondary phase (Al<sub>2</sub>Cu) which precipitates on the grain boundary (precipitant). In the formation, Al<sub>2</sub>Cu contributes balance of ternary system with maximum amount of dissolved copper atom in aluminum grate structure. This causes strain of atom grates, which increase strength. This fact considers that copper is good for aluminum alloys system because of super-saturated solid solution with precipitant spread in the ageing process. After the homogenization, the alloy's strength increases with copper content exceeding the solution limit because of strain of aluminum grate of crystal FCC as the result of much bigger copper atom.



Fig. 4. Mechanism of copper precipitate in Al-Cu equilibrium diagram

The superheated liquid of Al-Cu binary alloy is poured into the permanent mold with preheating of 250 °C and forms matrix of main phase of  $\alpha$ -aluminum surrounding double phase (precipitant). The mechanism of precipitant growth in the Al-Cu alloy casting causes the shape of crystal orientation, which is different from pure metal. Schematically, **Fig. 5** shows the soaking level of 15 minutes (5a) until soaking 30 minutes (5d) with solidification in mold produces different precipitant. In this case, there are two main causes. First, the concentration of Cu solution in liquids increases along with the increase of grain growth of  $\alpha$ -aluminum main phase. Second, the temperature of ingot center, which tends to imitate interface happens simultaneously. If the center zone of equiaxed appears in ingot, it means that supercooling change happens in the area and it can spread to the nucleation point in the casting center liquid. In this area, crystallization appears and continued by the precipitant growth. The growth is not continued by the lengthwise grain in the columnar area.

When the material dissolution is exceeded by adding too much alloy elements, the second phase shape is produced. The boundary between two phases is the surface where the atomic order is imperfect. In metal, the boundary does not disturb dislocation slip and it strengthens the material. The general term of strengthening by the second phase introduction is dispersion strengthening. The detail of more than one phase has to appear in each dispersion-strengthening alloy. Continuous phase usually appears in bigger amount, which is called matrix. The second phase usually appears in smaller amount, which is known as precipitant. In some cases, two phases are formed simultaneously. The structure is defined differently calling the intimate mixture of micro constituent phase. There are some general considerations to determine how matrix characteristics and precipitant influence the overall characteristics of metal alloys. **Fig. 5** shows the ingot microstructure of aluminum alloys which has been treated with melt holding time and casted with permanent mold. The increase of holding time in the melting furnace produces rounder microstructure boundary because the Cu dissolution in the aluminum increases. The copper dissolution helps composition dissolution formation in the alloy as shown in the bigger gray zone.

Optical microscopic after solidification shows copper sediment at Al-Cu alloy grain boundary. The sediment, which is rich of Cu, has nucleation on the grain boundary as a sign of highest energy distribution to the ongoing diffusion. It can be seen that precipitant has grown until it forms interconnected long change of precipitant. The center of primary precipitant grows before the freezing ends. Primary precipitant is usually bigger compared to secondary and tertiary precipitant. Because of its big size, primary precipitant appears on the micro, which has big distance and does not contribute to the precipitant strengthening. Primary precipitant is nucleation which is potential to fatigue cracks in dynamic loading condition. However, primary precipitant generally is not involved in precipitant reaction evolution after solidification because of its big size and it may influence the matrix chemical composition (precipitant domain) by reducing the available element amount. Precipitant of second phase is formed as particle, which is dispersed finely.



Fig. 5. The Result of microstructure photo of aluminum alloy type 2xxx, 500X magnification, etched by (1 % NaOH+4 % KMnO<sub>4</sub>+95 % H<sub>2</sub>O) and 5 % HF+95 % H<sub>2</sub>O: a - 60 Minutes Hold-Melt; b - 90 Minutes Hold-Melt; c - 120 Hold-Melt; d - 150 Hold-Melt

Fig. 6 shows the observation of specimens under a 500X magnification SEM that can clearly be seen from the Al<sub>2</sub>Cu precipitates formed during solidification. It appears in the picture that the shape of the precipitate resembles a long flakes. Increasing the hold-melt time, the flak gets bigger and new flak colonies appear with irregular shapes. And the process is so dynamic as shown from the beginning (Fig. 5, a) to the end (Fig. 5, d).

Thus, there is a large driving force for precipitation, and nucleation begins quickly, homogeneously, and in many places.



**Fig. 6.** Microstructure of aluminum alloy type 2xx.x by scanning electron microscopy: a - 60 Minutes Hold-Melt; b - 90 Minutes Hold-Melt; c - 120 Minutes Hold-Melt; d - 150 Minutes Hold-Melt

d) hardness.

Generally, precipitation hardening functions to increase the strength commercial alloys such as quenched-tempered steel and aluminum alloys (Al-Cu alloys). The precipitation hardening becomes one of the effective ways in developing ultrahigh strength. This is produced from dispersion of obstacle particle of dislocation movement, which uses second phase precipitation process. The quantification of precipitation hardening is joint mechanism of precipitation hardening and kinetic of rough growth of the surrounding matrix. The bigger precipitant formation is released by smaller particle dissolution. Sediment particle may hamper the dislocation movement through various interaction mechanisms of chemical strengthening, Caesar piling (stacking-fault) and monotonic relation hardening between shear stresses. The amount of precipitation hardening is determined by the type of precipitant and fraction size which is controlled by how fast the precipitation reaches balance fraction at peak hardness, [13].

The hardness test of Al-Cu alloys ingot generally uses Brinell penetration type, which is based on typical applications of identification hardness test. **Table 3** displays the hardness test result of Al-Cu alloys ingot during the copper dissolution in liquid aluminum increases and it causes the ingot hardness increase. Generally, the addition of alloy element including copper into aluminum is meant for substantial increase of strength and to facilitate precipitation hardening. However, aluminum-copper usually contain 2 % until 4 % of copper with smaller addition compared to other elements. Copper allows to achieve maximum strength of Al-Si alloys in as-cast condition. The maximum concentration is around 7 %÷8 wt. %. According [14], the copper addition as the main alloy element produces the strongest aluminum cast alloy. The grate gets more distortion with strange atom and it makes the hardness increase.

Holding time [minute]	Hardness of points [BHN]						
Holding time [minute]	1	2	3	4	5	Average	
15	93	96	96	89	94	93.6	
20	94	98	101	96	98	97.4	
25	99	101	105	98	102	101	
30	107	107	109	112	106	108.2	

## Table 3

The Hardness of Every Specimen

In solidification mechanism, molten Al-Cu alloys is poured into mold and it forms main dendrite, grain, and nucleation. Each happens on contact surface, subsurface and pouring duralumin center. The increase of solidification time of main dendrite transforms to second dendrite, grain boundary becomes planar structure, and the center becomes equiaxed dendritic. [15, 16] state that the hardness of Al-Cu alloys as-cast is generally between 90 BHN to 104 BHN. In this study, the ingot hardness can achieve 93.6 BHN to 113.6 BHN. This indicates the strengthening mechanism of the alloy, which is caused by the dissolved atom of Cu added into the aluminum. The dissolved atom causes grate distortion, which prevents dislocation movement, increase Al-Cu alloys strength, and hardness. Dissolved atom has stress zone around the matrix primary phases, which can interact with dislocation. The appearance of dissolved atom gives press and pull stresses to the atom side, which depends on the size of dissolved atom acts as potential dislocation barrier.

The solid solution strengthening mostly depends on the concentration of dissolved atom, shear modulus of the dissolved atom, the size of the dissolved atom, and the valence of dissolved atom. In the system of binary alloys, alloy, which is above the concentration given by the phase diagram, will cause second phase formation. The second phase also can be made with mechanical or thermal treatment. The particle, which forms second phase precipitant, functions as insertion point with similar way as in dissolved substance even though the particle is not always single atom. Ivailo et al, determines that the strength increase and hardness comes from the strengthening

of solid solution and non-equilibrium phase formation in micro material structure in balance, aluminum in solid solution with two other constituents (symbolized as a), copper and aluminum phase, CuAl ( $\theta$ ), silver and aluminum phase (AgAl) [17].

The increase of hardness values was observed and it is well known that the grain refinement is an important hardening mechanism used to increase the material's mechanical strength and its toughness by conditions presented the lowest grain sizes because the precipitant amount has stronger effect than casting speed on  $\alpha$  grain size. The increase of hardness value shows that grain refinement is important part of the hardening mechanism to increase the material's mechanical strength and toughness because the precipitant amount has stronger effect compared to the speed on  $\alpha$  grain size.

The correlation of hold-melt time and hardness in **Table 3** can also be seen in **Fig. 7** where **Fig. 7** indicates a trend of the increase of hardness along with the increase of the hold-melt time. From the statistical test, the hardness increases linearly (equation 4) with coefficient of determination  $R^2 = 0.9688$  and the other factor effect is very small (0.0312 %). This indicates that hold-melt time (independent variable) affects the hardness of Al-Cu alloy ingot (dependent variable). The value of hardness (Y) in this study is analytically expressed with equation:

$$Y = 4.74X + 88.2,$$
 (5)

where X is the *hold-melt* time and 88.2 is the constants.



Fig. 7. The effect of hold-melt time on hardness of the Al-Cu alloy

This research contributes to smelting energy saving and improving the characteristics and hardness of 2xxx type aluminum ingots. However, further analysis of the residual stresses that may be generated during the addition of other elements is needed. Residual stress is a concern as it reduces the energy savings of the fabrication process.

## 4. Conclusion

The diffusion phenomenon in the production of aluminum alloys (Al-Cu alloys) as conducted in the research has proven that in short time ( $\pm$ 30 minutes) copper can dissolve at temperature of 900 °C (in molten aluminum). Solidification after copper dissolution in molten aluminum in ingot permanent mold shows that percentage of metal weight of each low and high melting point experiences decrease and increase. Copper percentage in Al-Cu alloys increases along with the increase of copper soaking time in molten aluminum, which causes the specific weight of Al-Cu alloys increase from 2.73 to 2.89 g/cm<sup>3</sup>. In this study, the soaking time variations are 15, 20, 25, and 30 minutes and each variation produces copper dissolution in Al-Cu alloys 2.495, 3.490, 3.893, and 4.490 % copper weight. The increase of copper in Al-Cu alloys causes main phase ( $\alpha$ -Al) decrease with round and small grain while the network precipitant amount increases and becomes thicker. The copper content in Al-Cu alloys increases from 2.495 to 4.490 and it makes the hardness increase from 94 BHN to 114 BHN. Copper immersion in liquid aluminum can decrease the

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melting point. In the future, this technology can be used to reduce the melting energy consumption of manufacturing metal alloy process.

#### **Conflict of interest**

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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## Data availability

Manuscript has no associated data.

#### References

- [1] Khraisat, W., Abu Jadayil, W. (2010). Strengthening Aluminum Scrap by Alloying with Iron. Jordan Journal of Mechanical and Industrial Engineering, 4 (3), 372–377. Available at: http://jjmie.hu.edu.jo/files/v4n3/147-09%20done.pdf
- [2] Suprapto, W. (2017). Diffusion process in reverberatory furnace to control the quality of recycled aluminum. International Journal of Materials Engineering and Technology, 15 (4), 205–223. doi: https://doi.org/10.17654/mt015040205
- [3] Matisková, D., Gašpar, Š., Mura, L. (2013). Thermal Factors of Die Casting and Their Impact on the Service Life of Moulds and the Quality of Castings. Acta Polytechnica Hungarica, 10 (3). doi: https://doi.org/10.12700/aph.10.03.2013.3.6.
- [4] Di Sabatino, M., Arnberg, L. (2009). Castability of aluminium alloys. Transactions of the Indian Institute of Metals, 62 (4-5), 321–325. doi: https://doi.org/10.1007/s12666-009-0049-2
- [5] Suzuki, S., Kim, T.-B., Nakajima, H. (2009). Fabrication of Al-Cu alloy with elongated pores by continuous casting technique. Journal of Physics: Conference Series, 165, 012068. doi: https://doi.org/10.1088/1742-6596/165/1/012068
- [6] Xu, K., Thomas, B. G. (2009). Prediction of Grain Size, Precipitation and Crack Susceptibility in Continuous Casting. AISTech 2009 Steelmaking Conference Proc. Available at: http://ccc.illinois.edu/PDF%20Files/Publications/09\_XU\_K%20Prediction%20of%20Grain%20Size%20Precipitation%20and%20Crack%20Susceptability%20in%20CC\_AISTech.pdf
- [7] Suprapto, W., Suharno, B., Sudarsono, J. W., Priadi, D. (2012). Morphology and Quantity of Gas Porosity as the result of Duralumin Cast on Reveberatory Furnace. International Journal of Materials Engineering and Technology, 8 (2), 123–135.
- [8] Dolic, N., Brodarac, Z. (2017). Evaluation of EN AW-5083 aluminum alloy homogeneity using statistical analysis of mechanical properties. Journal of Mining and Metallurgy, Section B: Metallurgy, 53 (3), 429–439. doi: https://doi.org/10.2298/ jmmb170812046d
- [9] Hamasha, M. M., Mayyas, A. T., Hayajneh, M. T., Hassan, A. M. (2011). The Effect of Time, Percent of Copper and Nickel on the Natural Precipitation Hardness of Al–Cu–Ni Powder Metallurgy Alloys Using Design of Experiments. Journal of Minerals and Materials Characterization and Engineering, 10 (06), 479–492. doi: https://doi.org/10.4236/jmmce.2011.106036
- [10] Suprapto, W. (2019). Effect recycled aluminium structures of metallurgycal and melt efficiency. IOP Conference Series: Materials Science and Engineering, 494, 012085. doi: https://doi.org/10.1088/1757-899x/494/1/012085
- [11] Ritter, N. C., Sowa, R., Schauer, J. C., Gruber, D., Goehler, T., Rettig, R. et al. (2018). Effects of Solid Solution Strengthening Elements Mo, Re, Ru, and W on Transition Temperatures in Nickel-Based Superalloys with High γ'-Volume Fraction: Comparison of Experiment and CALPHAD Calculations. Metallurgical and Materials Transactions A, 49 (8), 3206–3216. doi: https:// doi.org/10.1007/s11661-018-4730-0
- [12] Wang, M.-X., Zhu, H., Yang, G.-J., Liu, K., Li, J.-F., Kong, L.-T. (2021). Solid-solution strengthening effects in binary Nibased alloys evaluated by high-throughput calculations. Materials & Design, 198, 109359. doi: https://doi.org/10.1016/j.matdes. 2020.109359
- [13] Ayoola, A., Adeosun, S. O., Sanni, O. S., Oyetunji, A. (2012). Effect Of Casting Mould On Mechanical Properties Of 6063 Aluminum Alloy. Journal of Engineering Science and Technology, 7 (1), 89–96. Available at: https://jestec.taylors.edu.my/ Vol%207%20Issue%201%20February%2012/Vol\_7\_1\_089\_096\_WASIU%20AJIBOLA%20AYOOLA.pdf
- [14] Nwaeju, C. C., Odo, J. U., Jisieike, S. C., Edoziuno, F. O. (2016). Effect Of Copper Addition On The Structure And Mechanical Properties Of Al-4 % Zn Alloy. International Journal of Scientific Research Engineering & Technology (IJSRET), 5 (1). Available at: https://www.researchgate.net/publication/330673301\_EFFECT\_OF\_COPPER\_ADDITION\_ON\_THE\_STRUC-TURE\_AND\_MECHANICAL\_PROPERTIES\_OF\_Al-4Zn\_ALLOY

- [15] Couch, J. (2017). Cast and investigate the characteristics of a high strength aluminium, copper and silver alloy, The Plymouth Student Scientist, 10 (1), 143–165. Available at: https://pearl.plymouth.ac.uk/bitstream/handle/10026.1/14142/TPSS-2017-Vol-10n1 143-165Couch.pdf?sequence=1&isAllowed=y
- [16] Faria, G. L. de, Leite, A. S. (2018). The Effect of Casting Speed and the Fraction of Al5%Ti1%B Inoculant on the Microstructure and Mechanical Properties of the AA5052 Aluminum Alloy Produced by the Direct Chill Process. Materials Research, 21 (2). doi: https://doi.org/10.1590/1980-5373-mr-2017-0997
- [17] Atanasov, I., Ferrando, R., Johnston, R. L. (2014). Structure and solid solution properties of Cu-Ag nanoalloys. Journal of Physics: Condensed Matter, 26 (27), 275301. doi: https://doi.org/10.1088/0953-8984/26/27/275301

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