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Effect of ageing time 200 °C on microstructure behaviour of Al-Zn-Cu-Mg cast alloys

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Abstract. Al-Zn-Cu-Mg is heat treatable alloy that can be used in many hightech applications, such as aerospace and military. The main objective of this study is to investigate the influence of ageing process in microstrucure behaviour of Al-9Zn-5Cu-4Mg cast alloy by performing SEM analysis and its correlation with hardness tests of as-cast Al-9Zn-5Cu-4Mg alloy and heat treated Al-9Zn-5Cu-4Mg cast alloy. The results show the deployment of precipitation spread over the dendrite and also the presence of second phases $Mg_3Zn_3Al_2$, Cu_2FeA_{l7} , $CuAl_2$, and $CuMgAl_2$ in as-cast Al-9Zn-5Cu-4Mg alloy. The presence of all these second phases are affecting to the toughness of aluminium alloy and the presence of $MgZn_2$ leads the impairment of hardness value of heat-treated Al-9Zn-5Cu-5Mg cast alloy.

1 Introduction

Over the past fifty years, aluminum has become the metal used broadly after the use of steel. Aluminium selected as an alternative material to increase efficiency because its mechanical behaviour : light weight, easy to design, easy to recycle, good in machinability and manufacturability, good heat conductor and corrosion resistant [1]. However, Pure aluminum can not stand alone because it has low strength and unstable mechanical properties. Therefore, to improve the mechanical properties of aluminum and modify the microstructure can be done through alloying and heat treatment [2].

Aluminium 7xxx series has a higher strength when compared to any other classes of aluminum alloys. The main properties of 7xxx series alloy indispensable in some high-tech applications such as on an airplane wing is of the nature of strength, toughness and resistance properties to stress corrosion cracking and fatigue [3].

The addition of zinc will improve the homogenity of the martix aluminium. Zinc provides a solid solution strengthening effect or work hardening in aluminium. This element improve the potential of the solution in aluminium and it makes Zn usually used in in the protective layer and the sacrificial anode [4]. The addition of 9 % Zn is increasing the strength that followed by the decrease in ductility. It is happen through the mechanism of solid solution strengthening, where Zn dissolved as a substitute in the lattice of aluminum atoms. The addition of magnesium provides the improvement and strengthening in work-hardening characteristics of aluminium so it produces good corrosion resistance and weldability.

The impact energy and hardness of the matrix are enhanced when Cu addition is within 2-4 vol % [5]. The effect of addition copper in aluminium is improving the machinability with increasing matrix hardness. In other words, Copper has a greatest impact on the strength and hardness of aluminum casting alloys, in both heat treated and not heat treated condition. The addition of Cu to the ternary Al-Zn-Mg system will produce the heat-treatable compositions which contributed to the increased hardness and strength [6].

Study on evolution of eutectic structures intheir Al-Zn-Mg-Cu alloys reported that several coarse intermetallic phases can be formed below the solidus line during solidification of Aluminium 7xxx alloys as a result of solute redistribution of metals [7]. These properties are determined by the major phases formed in the alloy, the GP zone η ', η, T, and S. Thus the nature of the 7xxx alloys can be optimized by modifying the microstructure through changes in alloy composition and heat treatment variations. System Al-Zn-Mg-Cu system is quite complex, chemical composition into primary particles present is η-$MgZn_2$, T-$Al_2Mg_3Zn_3$, S-Al_2CuMg, and θ-Al_2Cu, Al_7Cu_2Fe, $Al_{13}Fe_4$ and Mg_2Si [8,9].

During the casting process, there is the hardening of atoms that remain in solution. When the temperature increase, the rate of nucleation and growth of precipitates can be accelerated. The increase in strength occurs in line with the decrease in ductility on dendritic material. T5 is an artificial aging that are commonly used in increasing reinforcement through hardening precipitates. Benefits of T5 heat treatment can be lost when done quenching after casting.

The main mechanical properties of metal in addition affected by the chemical composition but also strongly influenced by the microstructure of the metal. By heat treatment, Aluminium will show the change in

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its structure. Artificial ageing in aluminum alloys above room temperature that is taking place between 100-200 °C. In this work, we were used T5 artificial ageing with heating temperature at 200 °C for 200 hours without added any solution treatment processes.

The main objective of this study is to investigate the influence of ageing process on microstrucure behaviour of Al-9Zn-5Cu-4Mg cast alloy by performing SEM analysis and its correlation with hardness tests.

2 Experimental Details

2.1 Preparation of Al-9Zn-5Cu-4Mg Alloy

Casting process of Al-9Zn-5Cu–4Mg alloy was conducted using a graphite crucible in muffle furnace with melting temperature measured stable in 750°C. Aluminium and alloy composition weight of the charge is 4.5 kg with calculation of mass balance of each alloy and the possible evaporation by 14% Zn and Mg 10% are as shown in Table 1.

Table 1. Mass Balance of Aluminum Alloys

Al	Zn (0,09)	Mg (0,04)	Cu (0,05)	Zn Assumed evaporated 14%	Mg Assumed evaporated 10 %
3615	405	180	225	461,7	198

The melting process begins with inserting aluminium into crucible for approximately 1 hour until it is completely dissolved. Then Cu, Zn, Mg inserted sequentially at Muffle furnace for 2 hours until all elements also completely dissolved.

To minimize the presence of hydrogen in liquid aluminum, can done by lowering the melting temperature so that the solubility of hydrogen in joined aluminum decreases with decreasing temperature. The degassing process must be done properly on all aluminium liquid parts. The degassing process that is useful to remove impurities dissolved performed using argon gas for approximately 2 minutes. Mold that used are permanent metal molds made from mild steel which has been coated with clay.

Coating with clay intended to casting product can be removed easily. The molten metal is cooled in a metal mold at furnace temperature until solidification occurs. After the molten metal have solidifying as a whole, the casting product then released from the mold and followed by a visual inspection of the results of the casting product's surface.

Cast products then tested for chemical compositions analysist by using emission spectrometer with type PMI– Master Pro Oxford instrument analytical GmBH the results shown in Table 2.

Table 2. Chemical Compositions of Al-9Zn-5Cu-4Mg cast alloy

Zn	Cu	Mg	Fe	Al
[%]	[%]	[%]	[%]	[%]
9.63	5.52	4.43	0.14	Rest

2.2 Cutting Samples

Casting product cutted to some parts based on samples requirement of testing to be performed. to ease process of cutting samples, small samples were cut by using hand grinder. Cutting process of samples is using a metal cutting saws 9" Type Krisbow KW1500053. Refinering process of surface was conducted by sandpapers to ensure the surface of samples completely smooth.

2.3 Heat Treatment

The experiment were prepared in two conditions, as-cast and heat-treated alloy. Artificial ageing was conducted by using Hofman K1 furnace at temperature 200 ° C for 200 hour, while as-cast aloy was not conducted any heat treatment process.

Furnace was preheated to 200°C before performing the heat treatment process. Cooling process was carried out in the heater furnace, and it takes 3 hr until the temperature in the furnace back to normal. Time required to heating the samples was 20 days with range of warm-up time approximately 10 hours each day.

2.4 Hardness Test

Artificial ageing carried out to observe the change in characteristic and the strength of Al-9Zn-5Cu-4Mg cast alloy to a long period and hight temperature heat treatment. Hardness test was performed in the two conditions of the samples, as-cast alloy and heat treated Al - 9Zn - 5Cu – 4Mg cast alloy in which each condition was conducted in 3(three) test samples to analyze the uniformity of hardness value in the alloy. Sample preparation was done by casting, sample cutting, heat treatment, surface smoothing and polishing. The load that used in this test is 10 kgf. Hardness (HV) obtained by using Equation 1 below

$$HV = \frac{2p\sin\left(\frac{\theta}{2}\right)}{L^2} = 1{,}854\ \frac{P}{L^2} \qquad (1)$$

2.5 Microstructure Observations

Microstructure observations were conducted using optical microscopy (OM), and Scanning Electron Microscope (SEM) with Energy-dispersive spectroscopy observation (EDS). All tests were carried out in two material conditions, as-cast and heat treated alloy.

Sample preparation process for these two observations are relatively similar; sample cutting, mounting, grinding, polishing, etching and the addition of the coating process using Au element for SEM samples.

Polishing process was conducted using TiO_2 and etching process was conducted using a mixture of 12.5 ml 8 ml HNO3 + HF + 85 ml of water for optical microscope and 0.5% HF for Scanning Electron Microscope observation.

3 Results and Discussions

3.1 Mechanical Characteristics

The hardness test results for as-cast and heat-treated alloy show in Table 3 and Fig 1. It was found that as-cast alloy has the highest hardness value. In the other hand, there were the impairment value of hardness in heat-treated alloy.

Table 3. Hardness Test Results

Al-Zn-Cu-Mg Cast Alloy	Hardness (VH)
As -cast	138
As –cast	139
As –cast	142
Heat treated T5	136
Heat treated T5	136
Heat treated T5	137

As-cast alloy has higher hardness because the presence of microsegregations in its structure that embrittle the Al-Zn-Cu-Mg alloy. The previous studies reported that smaller grain boundaries will make the metal harder and stronger [10]. In the smaller grain, there is one square unit of grain boundary for each dislocation. There is a much greater chance for a dislocation to be stopped at a grain boundary in the smaller grain. Therefore, the smaller grain is stronger. In the larger grain, a dislocation can travel up to 4 units without being stopped by a grain boundary, indicating the potential for extensive plastic flow. In the small grain, no dislocation can travel more than 1 unit of distance [11].

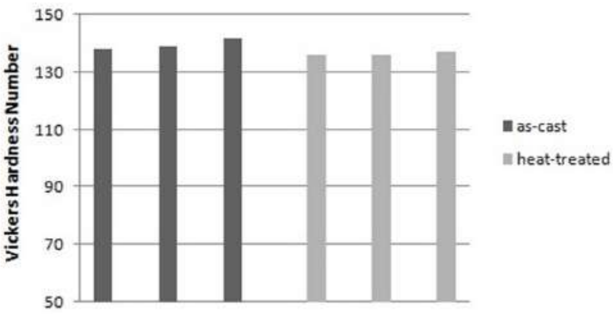


Fig. 1. Heat Treatment effects on hardness of Al-9Zn-5Cu-4Mg

The deployment of precipitation that affecting to the brittleness and the toughness of heat treated alloy, it makes the and hardness value were not exceed the value of as-cast alloy.

3.2 Microstructure Characteristic

Fig 2 and Fig 3 shows the difference of microstructure between as-cast and heat-treated Al - 9Zn - 5Cu – 4Mg cast alloy. In the as-cast alloys, the deployment of precipitation only occurs at the edge of grain boudary. Heat treatment process makes the precipitation spread to all over dendrite.

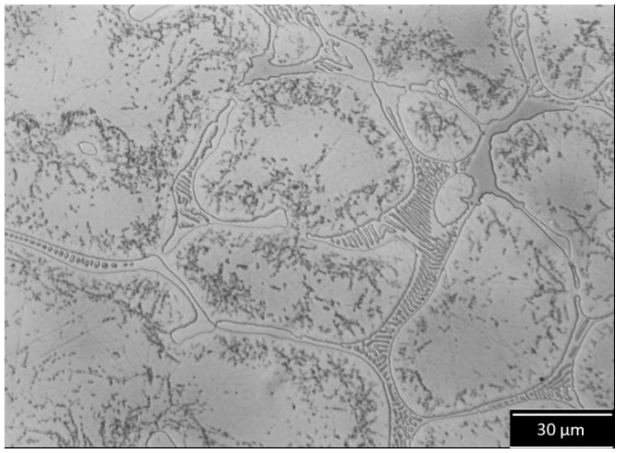


Fig. 2. Microstructure of as-cast Al-9Zn-5Cu-4Mg alloy

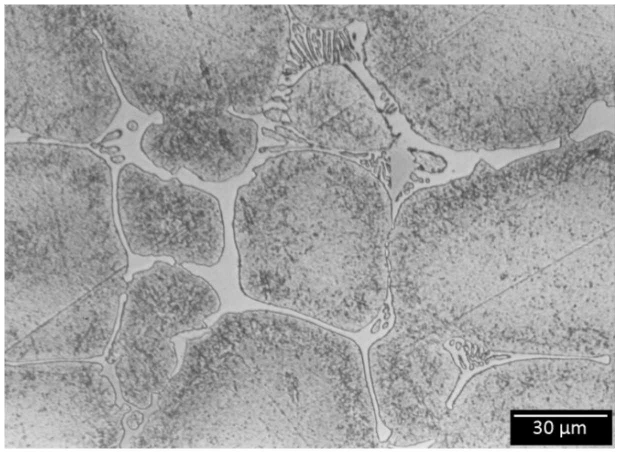
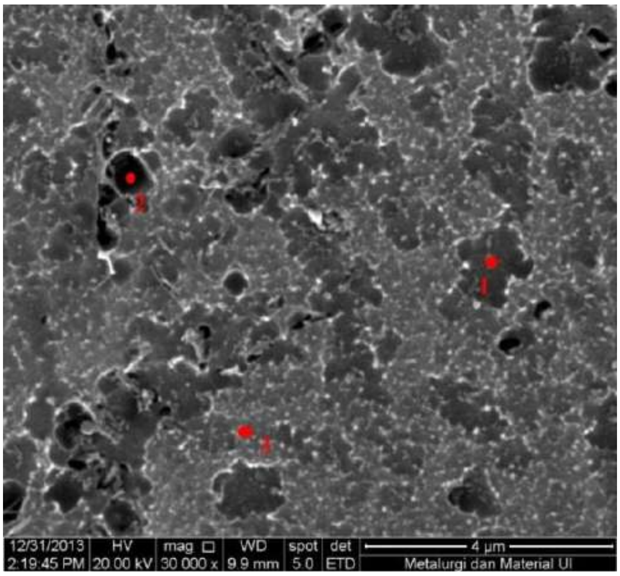


Fig. 3. Microstructure of heat-treated Al-9Zn-5Cu-4Mg cast alloy

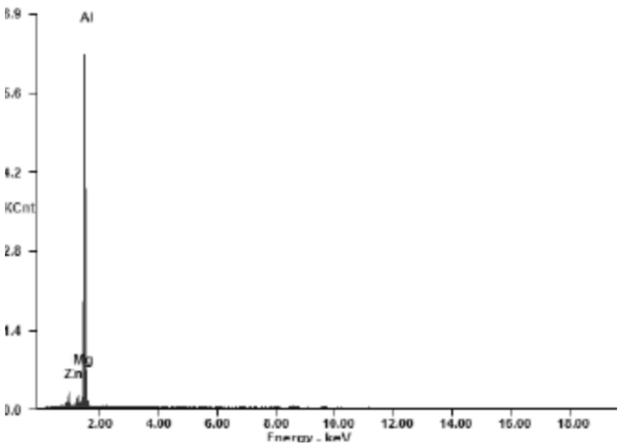
Observation points of Scanning Electron Microscope (SEM) on dendrite of as- cast alloy and its microanalysis report by Energy - dispersive spectroscopy (EDS) observation are shown in Figure 4 (a) and 4(b). While the analysis of grain boundary of as- cast alloy and its microanalysis report from Energy - dispersive spectroscopy (EDS) observation were shown in Figure 5(a) and 5(b). The phase that may be formed in the as-cast alloy are shown in Table 4 and Table 5.

Fig. 6(a) shows the results of Scanning Electron Microscope (SEM) on dendrite of heat-treated alloy while Fig.6(b) shows and its microanalysis report by

Energy - dispersive spectroscopy (EDS) observation. The analysis of grain boundary of heat-treated alloy is shown in Fig. 7(a), while its microanalysis report from Energy - dispersive spectroscopy (EDS) observation is shown in Fig. 7(b). The phase that may be formed in the heat-treated alloy are shown in Table 6 and Table 7.



(a)

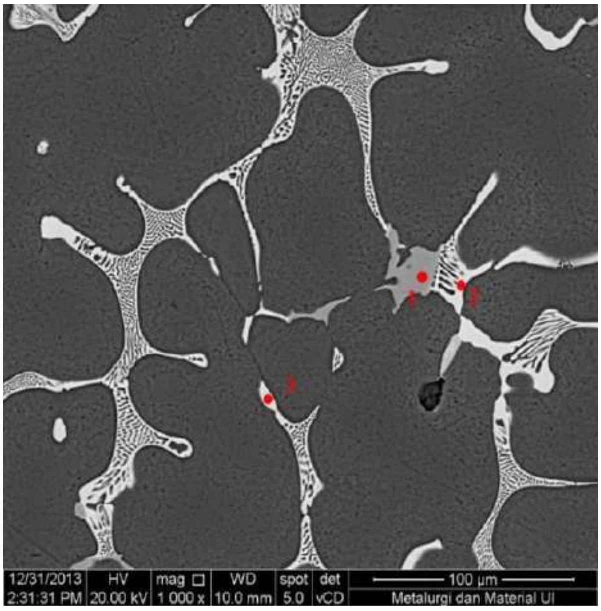


(b)

Fig 4. (a) Microstructure observation in dendrite of as-cast Al-9Zn-5Cu-4Mg Alloy (b) microanalysis report by Energy - dispersive spectroscopy (EDS)

Table 4. Summary of EDS observation on dendrite as-cast Al - 9.7Zn - 5.5Cu - 4.5Mg cast alloy based on SEM pictures

No	Average element wt.%			Phase may be formed*
	Al	Zn	Mg	
1	90.34	6.81	2.85	$Mg_3Zn_3Al_2$
2	89.40	7.30	3.30	$Mg_3Zn_3Al_2$
3	89.65	7.32	3.03	$Mg_3Zn_3Al_2$



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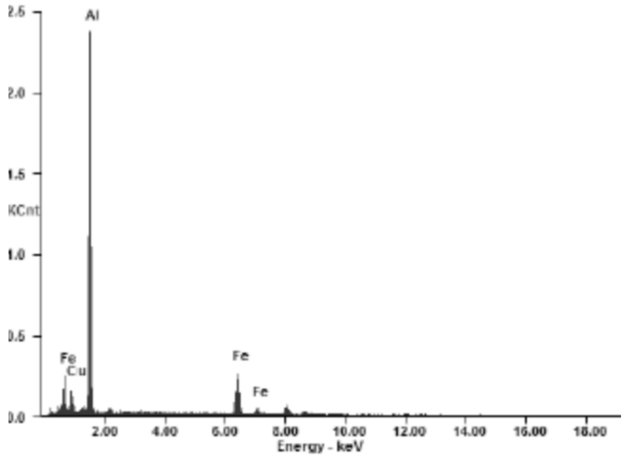
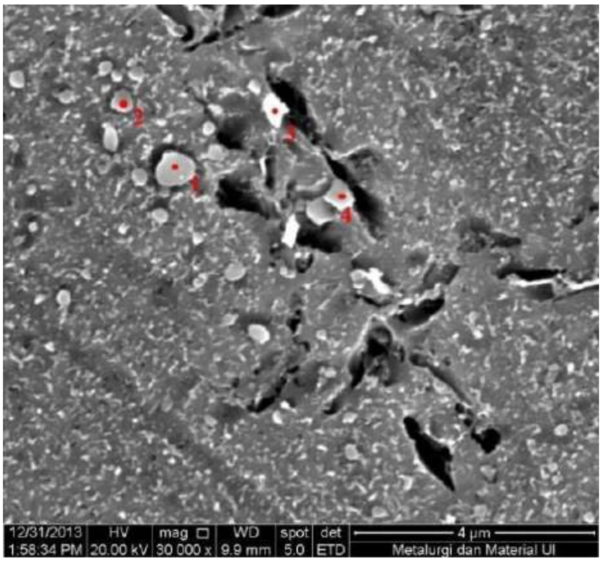


(b)

Fig 5. (a) Microstructure observation in grain boundary of as-cast Al-9Zn-5Cu-4Mg Alloy (b) microanalysis report by Energy - dispersive spectroscopy (EDS)

Table 5. Summary of EDS observation on grain boundary as-cast Al - 9.7Zn - 5.5Cu - 4.5Mg cast alloy based on SEM pictures.

No	Average element wt.%					Phase may be formed*
	Al	Zn	Mg	Cu	Fe	
1	68.59	-	-	11.31	22.09	Cu_2FeAl_7
2	24.72	27.29	23.85	24.14	-	$Mg_3Zn_3Al_2$, $CuAl_2$, $CuMgAl_2$
3	57.83	10.68	13.67	17.82	-	$Mg_3Zn_3Al_2$, $CuAl_2$, $CuMgAl_2$



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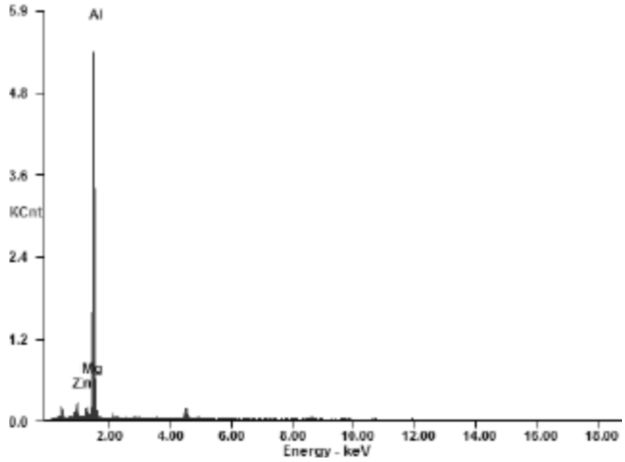


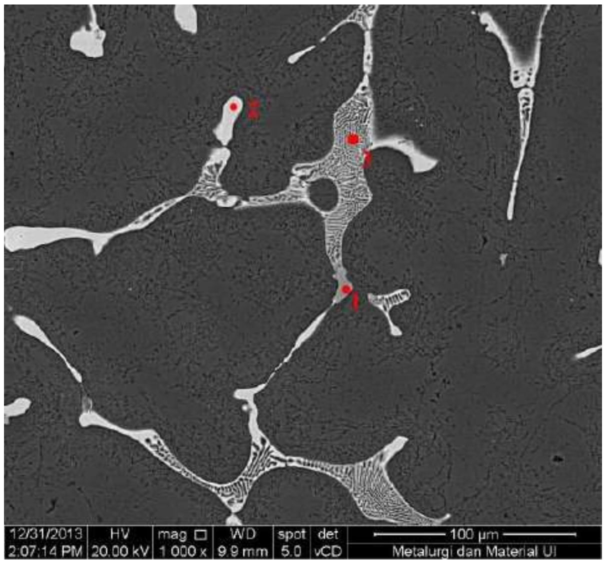
(b)

Fig 6. (a) Microstructure observation in dendrite of heat-treated Al-9Zn-5Cu-4Mg Alloy (b) microanalysis report by Energy - dispersive spectroscopy (EDS)

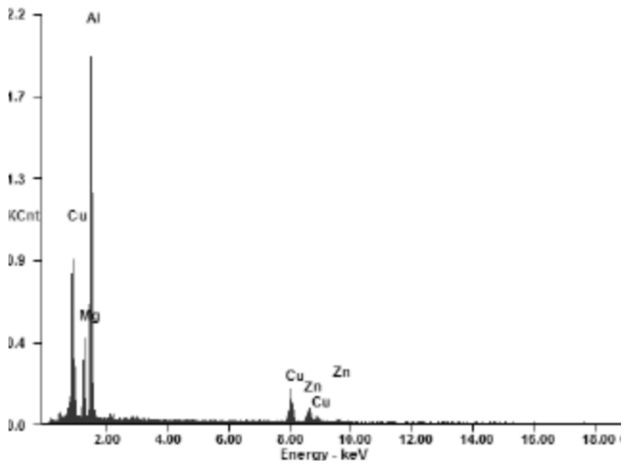
Table 6. Summary of EDS observation on dendrite heat-treated Al - 9.7Zn - 5.5Cu - 4.5Mg cast alloy based on SEM pictures

No	Average element wt.%			Phase may be formed*
	Al	Zn	Mg	
1	90.05	7.08	2.87	$Mg_3Zn_3Al_2$
2	89.38	3.09	7.53	$MgZn_2$
3	89.91	2.75	7.34	$MgZn_2$
4	90.52	2.75	6.73	$MgZn_2$

Based on SEM images,the precipitation in as-cast alloy is not visible clearly, $Mg_3Zn_3Al_2$ is the most dominant phase due to present at all three points of testing (see table 5), it may be possible because the comparison of Zn and Mg greater than 2.2 on as-cast alloy. While in heat-treated alloy, the precipitation is visible clearly. The holes in the surface indicate the presence of second phase precipitations which were detached from the surface due to etching and polishing processes.These precipitation will form the second phase that contained in dendrites and the grain boundaries. According to Table 7, the second phase that may be formed are $MgZn_2$ and $Mg_3Zn_3Al_2$.



(a)



(b)

Fig 7. (a) Microstructure observation in grain boundary of heat-treated Al-9Zn-5Cu-4Mg Alloy (b) microanalysis report by Energy - dispersive spectroscopy (EDS)

Table 7. Summary of EDS observation on dendrite heat-treated Al - 9.7Zn - 5.5Cu - 4.5Mg cast alloy based on SEM pictures

No	Average element wt.%					Phase may be formed*
	Al	Zn	Mg	Cu	Fe	
1	69.51	-	-	7.70	22.79	Cu_2FeAl_7
2	25.97	25.02	25.20	23.82	-	$Mg_3Zn_3Al_2$, $CuAl_2$, $CuMgAl_2$
3	57.83	10.68	13.67	17.82	-	$Mg_3Zn_3Al_2$, $CuAl_2$, $CuMgAl_2$

*) Prediction phase based on the phase data that may appear in Al-Zn – Mg [12]

The presence of Fe element as impurities made aluminum become coarse grains, reducing the ductility of alloy, and leads the presence of Cu_2FeAl_7. SEM observations at the grain boundaries of heat-treated alloy are not really different much with as-cast alloy, the conclusions of EDS can be seen in Table 5. The presence of these second phases are very influential to the strength and brittleness.

4 Conclusions

Phases that may be formed on as-cast alloy are $Mg_3Zn_3Al_2$, $CuAl_2$, $CuMgAl_2$ and Cu_2FeAl_7. While in heat-treated alloy, the phases that may be formed are the same with the as-cast. But the long period heating process and high temperatures heat treatment generate the presence of embrittled phase $MgZn_2$ within the dendrites. It phase is very influential for toughness and the strength of Al-9Zn-5Cu-5Mg cast alloy. The Presence of $MgZn_2$ leads the impairment of hardness value of heat-treated Al-9Zn-5Cu-5Mg cast alloy.

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