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# The Effect of Artificial Age Time on Crystal Size, Dislocation Density, Hardness and Micro Structure on Al 6061 Materials Alloy

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Abstract. Research on the effect of T6 heat treatment and artificial life time on crystal size, density dislocation, hardness, and microstructure of Al 6061 alloy material made from powder metallurgy. The T6 heat treatment starts with a solid solution which is heated at 530 ° C, held for 60 minutes, then quenching into the water media, and the artificial aging process at 200 ° C and variations in the holding time of 1h, 24h, and 30h. Crystal size, dislocation density and lattice microstructure testing using X-ray diffractometer, hardness testing with Vickers scale and surface microstructure with SEM-EDX. Test results of crystal size, dislocation density, and micro lattice strain on 4 phase  $\alpha$ -Al at the miller index plane (111), (200), (220), and (311) show that the crystal size increases with the duration of heating time of artificial aging. While dislocation density and micro lattice strain increase over a heating period of 1h to 30h, dislocation density and lattice strain decrease at the Miller index plane (111) to (311). The hardness testing of Al 6061 as-cast material was 54 HV after quenching the water hardness value of 75 HV, but after artificial aging the hardness decreased with a longer holding time from 45.50 HV to 39.95 HV. Microstructure observations with SEM-EDX, showed that the Al 6061 test sample without heat treatment showed a dominant  $\alpha$ -Al matrix, whereas in the Al 6061 sample after the T6 process it was seen that the Mg2Si phase functioned to harden the Al 6061 alloy.

#### **INTRODUCTION**

Aluminum is the 4th most abundant element on earth which is a lightweight metal which has mild properties, good corrosion resistance and good conductivity of electricity and heat, easily formed either through the process of forming or machining, and other good properties as metal properties. In nature, aluminum is a stable oxide which cannot be reduced in the same way as other metals. Aluminum reduction can only be done by electrolysis. In addition to its mechanical strength which is greatly increased with the addition of Cu, Mg, Si. Mn, Zn, Ni, etc., individually or together, also provide other good qualities such as corrosion resistance, wear resistance, low expansion coefficient and so on [1,2].

Aluminum is a material that is widely used for construction, ranging from bicycles, automotive, ships, rockets, to aircraft. The advantages of aluminum material are its light weight and strength which can be increased according to needs. The strength of aluminum is usually increased by means of alloying and heat treatment. Most aluminum materials are strengthened by a metal strengthening mechanism called precipitation hardening. In precipitation hardening must have two phases, namely a phase which is more numerous is called a matrix and a phase whose number is smaller is called precipitate. This strengthening mechanism includes three stages, namely a). solid solution treatment: heating up above the solvus line to get a homogeneous solid solution phase, b). quenching: better quickly to maintain a homogeneous solid phase microstructure to prevent diffusion, and c). aging: heated to a temperature not too high so that the alpha phase diffusion occurs at a short distance to form precipitate [3,4].

The International Conference on Chemical Science and Technology (ICCST – 2020) AIP Conf. Proc. 2342, 060001-1–060001-8; https://doi.org/10.1063/5.0045373 Published by AIP Publishing. 978-0-7354-4085-2/\$30.00 In the industrial world, especially automotive, the use of aluminum materials and alloys is often used, so innovations for aluminum materials continue to be developed to get maximum results. In the aluminum-based casting process for sub-assy transmission components one of the problems faced is the lack of perfection in the sub-assy transmission component products, such as uncomplate filling, low mechanical properties, shrinkage, and porosity. Through the heat treatment process aluminum alloy serves to improve the mechanical properties of the material, and modification of the microstructure of the material itself. Research on the addition of Sr to the mechanical properties analysis of Aluminum Casting with the modifier process based on variations of 0wt strontium variations. % Sr, 0.05wt. % Sr, 0.075wt. % Sr, and 0.1 wt. % Sr will increase tensile strength by 30.1% and compressive strength by 14.8% and the formation of Al-Fe-Mn-Si phase, silicon phase and  $\alpha$ -Al phase has been carried out [5].

Hanges in properties that occur at the end of the heat treatment process are caused by changes in the microstructure of the metal during the heating and cooling process. The magnitude of this microstructure is greatly influenced by the chemical composition of metals or their alloys and the type of heat treatment they undergo, therefore the type of heat treatment process is divided into, heat treatment which causes changes in the micro structure throughout the metal parts. Precipitation of the second phase particles involves a diffusion process that is highly dependent on temperature and time, therefore both temperature and aging time are two important parameters in the process of hardening the precipitate. During the aging process, the hardness and tensile strength of the material will increase and reach a maximum value when coherent deposition particles reach optimal distortion and size. If the size of the particles is too coarse, the distance between the particles will be too far away so that it is no longer effective in resisting the movement of dislocations [6,7].

This study aims to determine the effect of artificial aging resistance time on crystal size, dislocation density, lattice microstructure by X-ray diffraction method, hardness and microstructure in Al 6061 alloys made with powder metallurgy method after T6 process.

#### METHODOLOGY

The material used in the form of Al 6061 cylindrical shape is made by powder metallurgy method by weighing, mixing Al, Mg, and Si powder then printed and pressed 20 Ton and sintered temperature 475 <sup>o</sup>C. Size 20 mm in diameter, 2 mm thick.

TABLE	1. Data or	n the com	position of	of the chen	nical eleme	ents of A	l 6061 allo	ys with an	OE Specti	rometer
Ulement	Al	Si	Mg	Cu	Mn	Zn	Fe	Pb	Cr	Ti
Wt%	97,11	0,86	1,52	0,018	0,002	0,51	0,005	0,001	0,003	0,002

Before testing, the material was given a T6 heat treatment at a temperature of 530 °C solid solution treatment for 1 hour, then quenched in cold water media. After that artificial aging is carried out with a temperature of 200 °C held for 1 hour, 24 hours, and 30 hours. The metallographic process starts grinding, polishing to etching. The Al 6061 alloy specimens were then subjected to three tests, namely the Vickers micro scale hardness test, Crystal size test, dislocation density, and micro lattice strain with the XRD tool at UPT FMIPA, UI, and surface microstructure test with SEM-EDXS and hardness testing using a standard Vickers micro scale ASTM E384 with a compressive time of 12 seconds and a 0.5 KgF major load at the Forentik Laboratory, Puslabfor, Police Headquarters.

Testing of crystal structure such as crystal size, dislocation density, and microstructure of Al 6061 alloy lattice using XRD (Smartlab-Rigaku model) with Cu K $\alpha$  radiation ( $\lambda = 1.5406$  Å). XRD data were obtained at room temperature in the range of 25 ° to 100 ° using a scan speed of 2 °/min and a step width of 0.02 °. Crystal parameters such as, average crystal size, lattice strain, dislocation density, and lattice parameters (a and c) are determined from the results of XRD analysis. The average crystal size (D) of Al 6061 alloys is estimated using the Derby Scherrer equation [4],

 $D = 0.9 \lambda / \beta \cos \theta$  ......(1) where,  $\lambda$  is the wavelength of x-rays (1.5405 Å),  $\beta$  is FWHM (full width at half maximum) of the peak (hkl) and  $\theta$  is the diffraction angle.

Lattice micro strain ( $\varepsilon$ ) is calculated using the following equation [4],

	·	-				
$\varepsilon = \beta / 4 \tan \theta$						2)
The density of disloca	tion (o) due to lat	tice strain can	be expresse	ed by the relations	ship [4].	

e density of dislocation ( $\rho$ ) due to lattice strain can be expressed by the relationship [4],  $\rho = 1 / D^2$ .....(3) 

# **RESULTS AND DISCUSSION**

#### Analysis of Crystal Size, Dislocation Density, Micro Lattice Strain with XRD



FIGURE 1. Difractogram from Al 6061 alloy on temperature 200 °C artificially age with holding time 1 h, 24 h, and 30h.



FIGURE 2. Graph of relationship between Miller's index field and crystal size at the time variation artificial age of Al 6061 alloys



FIGURE 3. Graph of relationship between Miller's index field and density of dislocation at the time variation artificial age of Al 6061 alloys



FIGURE 4. Graph of relationship between Miller's index field and micro lattice strain at the time variation artificial age of Al 6061 alloys

The diffraction peaks of the XRD results show that Al 6061 alloys have 4  $\alpha$ -Al phases in the Miller index plane namely (111), (200), (220), and (311) of the crystal size seen the longer the holding time the smaller the crystalline size (6.3nm to 5.8nm) in the Miller index field (111), as well as in the Miller index field (311) the smaller the crystal size (4.3nm to 4.0 nm) see in Figure 2.

The size of the crystallites in the Miller index field (111) which is 6.3nm decreases with the holding time of artificial aging 30 hours is 4 nm in the Miller index plane (311). These data indicate that the increase in crystallite size is likely due to the recrystallization process and grain growth during aging. This can be explained by the micro lattice strain, that the majority of Al 6061 alloy plastic deformations occur through a dislocation process slip and twin. Thus the artificial aging imposed on the Al 6061 alloy material does not turn into grain strain but into rotation on the crystal lattice. This crystal lattice shift produces crystallites. Because the heat treatment (artificial age), the second causes the diffusion of atoms at the grain boundary, which is characterized by an increase in crystal size. In Figure 2 shows the value of the dislocation density seen the longer the holding time the greater the density of the dislocation (0.028 lines / mm2 in the Miller index plane (111) to 0.058 lines / mm2 in the Miller index grain (311).

In Figure 3, there are 4 diffraction peaks from the XRD results showing that Al 6061 alloys consisting of Miller index fields namely (111), (200), (220), and (311) of the lattice microstructure show that the longer the holding time the greater micro lattice strain (17.2%) in the Miller index field (111), as well as in the Miller index field (311) the longer the holding time the micro lattice strain is getting smaller at 13%.

Figures 1.2,3, and 4, show that increasing the holding time of aging heat treatment results in narrowing of the diffraction peak, which is marked by a decrease in the FWHM value. The results revealed that grain growth occurred from Al 6061 alloys. In addition, the diffractogram pattern also showed that the intensity of the diffraction peaks increased with increasing time to hold artificial aging during heat treatment. This indicates that there is an increase in the crystal quality of Al 6061 alloys. The crystal quality of Al 6061 alloys is influenced by micro lattice strain and dislocation density. Low strain value and dislocation density indicate good crystal quality. These results are closely related to the reduction in line shape crystal defects in Al 6061 alloys.

Due to the increase in the crystal defect line shape is shown by increasing the lattice microstructure value resulting in an increase in the density of the dislocation. Because lattice microstructure affects the length of the dislocation line per unit volume of crystal [7].

In addition the micro lattice strain of Al 6061 alloy samples resulting from rapid dipping and artificial aging within 24 hours in the Miller index field (311) is 13% smaller when compared to the Miller index field (111) artificial aging within 24 hours ie 19%. This data shows that the low lattice microstructure is likely to occur the process of recrystallization and grain growth during artificial aging. Based on data that the dislocation density resulting from artificial aging in the Miller index (311) is greater than that of the Miller index field (111), it means that there is a maximum strain limit due to deformation. The maximum strain is caused by the deformation mechanism and must meet the constant c / a ratio, so the dislocation density increases. Increasing the dislocation density results in a further increase in residual stress to the yield strength [7,8].

#### **Analysis of Micro Vickers Hardness**

From the results of the average hardness testing of the Al 6061 test sample which experienced T6 heat treatment, namely heating of a solid solution at a temperature of 530 0C and artificial aging at an temperature of 200 $\Box$ C with time variations for 1 hour, 24 hours and 30 hours.

No.	Н	ardness Vickers (HV)	HV)		
1	As cast		54		
2	Quenching		75		
3	Aging	Time 1 hour	45,5		
4		Time 24 hours	42		
5		Time 30 hours	39.95		

 TABLE 2. Results of Vickers micro-scale Hardness testing from Al 6061 alloys

 No.
 Hardness Vickers (HV)





Hardness test results for Al 6061 alloys using the Micro Vickers scale see Table 2. Samples of Al 6061 as cast (original) alloys were 54 HV and after quenching 75 HV. The artificial aging process with a temperature of 200°C and a holding time variation of 1 hour, 24 hours, and 30 hours decreased the value of hardness compared to materials without heat treatment by 15.74% or originally 54 HV to 45.5 HV. The highest hardness value after quenching is 75 HV. This is caused by the presence of the second phase, Mg2Si as precipitate. The presence of this precipitate plays a role in increasing the hardness of Al 6061 alloys, by blocking dislocation movements and dislocation buildup which causes lattice distortion in Al 6061 alloys when subjected to deformation. [7] After artificial aging the value of violence decreases with length of detention time from 1 hour (45.5 HV) to 30 hours (39.95 HV). This is consistent for all variations of artificial aging time on Al 6061 alloys. The phenomenon of decreasing the value of violence during the artificial aging process is called over aging. [10/8] decrease the value of violence. This is due to the joining of precipitates into larger sized particles which causes the dislocation movement barrier to become weaker, thus decreasing mechanical properties. Also caused by precipitates not spread evenly and large sizes of precipitates in Al 6061 alloys [7,9].

### Analysis of Micro Structure with SEM-EDXS



FIGURE 6. Micrographs of Al 6061 alloy micrographs at age temperatures of 200 °C, 1 hour time, 10,000X enlargement



FIGURE 7. Micrographs of Al 6061 alloy micrographs at age temperatures of 200 °C, 24 hours time, 10,000X enlargement



FIGURE 8. Micrographs of Al 6061 alloy micrographs at age temperatures of 200 °C, 30 hours time, 10,000X enlargement

From Figures 6, 7, and 8 show that micrograms of Al 6061 alloy after aging help with time variations of 1 hour, 24 hours, and 30 hours. Everything looks equaxial in the condition of 1 to 30 hours. Artificial aging grains indicate elongated grain. The changing shape of the grain after artificial aging is due to deformation so that it changes the shape of the Al 6061 alloy material. This will also have an impact on the mechanical properties ie the hardness produced.

## CONCLUSIONS

From the results of calculations and analysis, it was concluded that after the T6 process began solid solution treatment, quenching and holding time variations artificially aging on Al 6061 alloys resulted in recrystallization and grain growth as evidenced by the increase in lattice microstructure and crystallite size of the  $\alpha$ -Al phase at the Miller index fields (111), (200), (220) and (311) within 24 hours, and the dislocation density decreases. Hardness value decreases with increasing aging time. This is due to the joining of precipitates as phase two Mg2Si into larger sized particles which causes dislocation movement barriers to become weaker, so that the value of hardness (mechanical properties) decreases. The changing shape of the grain after artificial aging is due to deformation so that it changes the shape of the Al 6061 alloy material. This will also have an impact on the mechanical properties ie the hardness produced.

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