The Role of Al_2O_3 Nanoparticles Addition on Characteristic of Al6061 Composite Produced by Stir Casting Process

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Abstract: Aluminium Matrix Composites (AMCs) reinforced with Al_2O_3 nano particles are widely used for high performance application such as aerospace because aluminium is light weight and alumina has good performance at high temperature. Alumina nano particles is added into molten Al with different volume fraction from 0.2 vf-% to 1.2 vf-% while Mg is used as an external dopant with 10 wt-% to promote wetting between aluminium and Al_2O_3. The Al alloy was then melted and Mg along with the reinforcement was blended inside the molten metal by stirrier with rotational speed of 500 rpm at 800°C for 2 minutes and degassing with Ar for 4 minutes to remove all of gas in molten Al. The molten composites then was casted into plate and tensile test sample molds. The effect of Al_2O_3 nano particles on mechanical properties and microstructure of composites was investigated. The optimum tensile strength, hardness and elongation of composite was achieved at additon of 0.2vf-% Al_2O_{3np} with the value of 220 MPa, 61 HRB and 5.48% respectivelly. Increasing hardness was caused by impedation of dislocation movements by nano-Al_2O_3 particles. It is found that the addition of more Al_2O_{3np}, the mechanical propeties decreased. The microstructure observations showed that the composites yield finer grains than the unreinforced alloy Addition of nano-Al_2O_3 particles also tend to form microporosity and agglomeration which would decrease the tensile strength of composites.

Keywords: Aluminium composites, Al_2O_3 nano particles, mechanical properties, stir casting process.

1. Introduction

Aluminium is the third abundant element in earth's crust, up to 8% of total earth's crust mass [1]. Aluminium is light weight metal which has one-third of steel's weight. This metal also has good corrosion resistance because formation of protective passive film. Aluminium alloy 6061 content of magnesium and silicon as main alloying elements and can be heat treated to improve its mechanical properties. This alloy widely used for several applications such as trucks, towers, canoes, railroads cars, furniture, pipelines, and other structural applications where strength, weldability, and corrosion resistance are needed [2]. With recent technology development, the aluminium alloy is more widely used for automoitive, and aircraft components.

Aluminium Matrix Composites (AMCs). Aluminium is used as matrix because it has low density, high ductility, and low melting temperature. Nano-sized Al_2O_3 can be used as reinforcement in AMCs to give significant strengthening effects without reducing its ductility. It is caused by nanoparticles have high surface to volume ratio of the reinforcing phase [3]. However, the ductility of the MMCs deteriorates significantly with high ceramic particle concentration.

In order to improve mechanical properties of composite therefore good interface bonding is needed. Good interface will be formed when all reinforcement particles can be wetted perfectly by the matrix phase. Wettability of materials can be tested by sessile drop method. This method is conducted by measuring the contact angle between reinforcement particle and molten matrix which is dropped on it. The wetting can occur when contact angle between reinforcement and matrix are smaller than 90^o . Wettability is also can be measured by Young Equation as follow :

$$\gamma_{SL} = \gamma_{SV} - \gamma_{LV} \cos\theta \quad (1)$$

where good wettability is showed by surface energy of reinforcement which is bigger than that of surface energy of matrix. If the contact angle between aluminium and Al_2O_3 is bigger than 90^o under 1000^oC so the wettability is poor [4]. Wettability of reinforcement particles can be improved with several ways, one of them is wetting agent addition. The addition of magnesium will improve wettability between aluminium and Al_2O_3 by enhancing surface energy of reinforcement, decreasing surface tension of matrix, and or decreasing interface energy between reinforcement and matrix on particle/alloy interface [5]. Magnesium addition will react with oxygen to reduce gas film between matrix and reinforcement by chemical reaction:

$$Mg_{(l)} + O_{2(g)} \leftrightarrow MgO_{(s)} \quad (2)$$

Beside react with oxygen, magnesium also can react with Al_2O_3 to form spinel ($MgAl_2O_4$). This phase can reduce the surface tension between matrix and reinforcement with this reaction :

$$Mg_{(s)} + 2Al_{(l)} + 2O_{2(g)} \rightarrow MgAl_2O_{4(s)} \quad (3)$$

Metal matrix nano composite can be made through variety of methods such as ultrasonic dispersion, mechanical milling, ball milling, and spray deposition. But ultrasonic dispersion only efficient in lab scale [4]. Stir casting has some important advantages such as better matrix-particle bonding, easier control of matrix structure, simplicity, low cost of processing, and nearer net shape. However, it is extremely difficult for the stir casting method to distribute and disperse nano particles uniformly in metal melts due to large surface-to-volume ratio and their low wettability in metal melts, which induce agglomeration and clustering [6]. Schultz *et al.* [4] used Mg in stir casting method to reduce particle clustering. In this study nano alumina is expected to improve mechanical propertiees such tensile strength but still remain ductility of nano composites produced by strir casting.

In this study nano alumina is expected to improve mechanical propertiees such tensile strength but still remain ductility of nano composites produced by strir casting. The aim of this study is to obtained the optimum volume fraction of Al_2O_3 addition to Al6061 in order to achieve composite with maximum mechanical properties.

2. Procedures

2.1. Making of Al6061/Al_2O_3 Nano Composite

Materials used for making nano composite were aluminium alloy 6061 as master alloy matrix with composition in Table 1. α-Al_2O_3 with average size of 80 nm as particles reinforcement in Fig. 1 shows agglomerate particles (supplied by US Nanomaterials Research, Inc). Commercially pure magnesium (supplied by PT Baralogam Multijaya, Indonesia) as wetting agent with conten of 10 wt-%. The reinforcement content was varied from 0.2 Vf-%, 0.5 Vf-%, 0.7 Vf-%, 1.0 Vf-%, and 1.2 Vf-% Al_2O_3 nanoparticles. The composition of Al_2O_3 is in Table 2. The reinforcement powder first was vibrated by giving 42 kHz vibration using ultrasonic vibrator to protec agglomeration of nano particles then pre-heated in 1000^{o}C for 1 hour to remove all of the moisture and to improve the wettability of particle in the melt. Al6061 alloy and magnesium was melted at 800^o C, then alumina particle was poured into alloy melt after the dross was removed. The melt then was stirred at 500 rpm for 2 minutes and degassing by flushing with Argon for 4 minutes.

Table 1. Composition of Aluminium Alloy 6061(wt-%)

Al	Mg	Si	Cu	Fe	Mn	Sn	Cr
≥99	2.6800	0.8190	0.2170	0.1610	0.01680	0.0100	0.05770

Table 2. Composition of Al_2O_3 Nanoparticles

Al_2O_3	Ca	V	Cl	Na	Mn	Co
≥99	≤20ppm	≤5ppm	≤280ppm	≤30ppm	≤5ppm	≤5 ppm

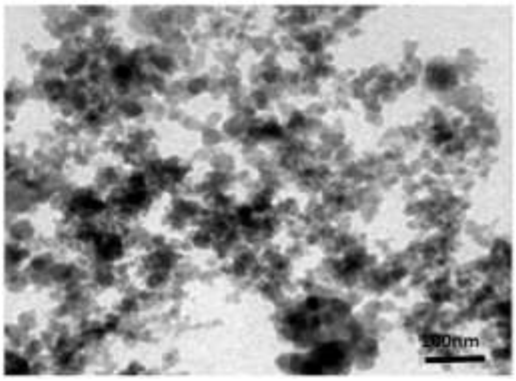


Fig. 1. Morphology of Al_2O_3 nano particles.

2.2. Characterization of Al6061/ Al_2O_3 Nano Composite

The mechanical properties of composites were measured including tensile strength, hardness, density, porosity, and wear resistance. Tensile testing was carried out using GOTECH AI-7000 LA 10 in accordance with ASTM B557M-02A. The tensile testing was carried out using three specimen each variable. Hardness testing was carried out using Rocky machine with Rockwell B method in accordance with ASTM E18-11. For each sample, five hardness readings on randomly selected regions were taken in order to eliminate the segregation effects and get a representative value of the matrix material hardness. Metallography preparation was conducted by grinding using emery paper started from 80#, #150, #240, #400, #600, #800, #1000, #1200 to #1500, then polished using TiO_2 powder to remove strach from grinding process. Keller's reagent (2 ml HF (conc.), 5 ml HNO_3 (conc.), 3 ml HCL and 190 ml aquadest was used as eching agent. All preparataion samples were observed using OLYMPUS BX41M-LED optical microscope and further analysys using Scanning Electron Microscope (SEM) link to Energy Dispersive Spectrum (EDS) and Field Emission Scanning Electron Microscope (FESEM), X Ray Diffraction (XRD) to analyse the phases present in composites.

3. Results and Discussion

3.1. Mechanical Properties of Al6061/Al_2O_3 Nano Composite

The effect of Vf% nano alumina on mechanivcal properties is shown in Fig 2. The addition of Al_2O_{3np} can increase tensile strength of Al6061 compared to Al6061 unreinforced (Fig. 2a). The optimum tensile strength is achieved at composites with 0.2 Vf-% Al_2O_{3np}, then decrease with addition in the range between 0.5 Vf-% and 1.2 Vf-% Al_2O_{3np}. Increasing tensile strength is caused by grain refinement mechanism and the presence of nano alumina particle in Al6061 matrix that hindered dislocation movement [7]. Al_2O_3 nanoparticles were also strengthened composite because they hold bigger load than that of base alloy. It will happen if only the load can be transferred from matrix to reinforce effectively. Load transfer will be effective when the composite possessed good wettability between matrix and reinforcement.

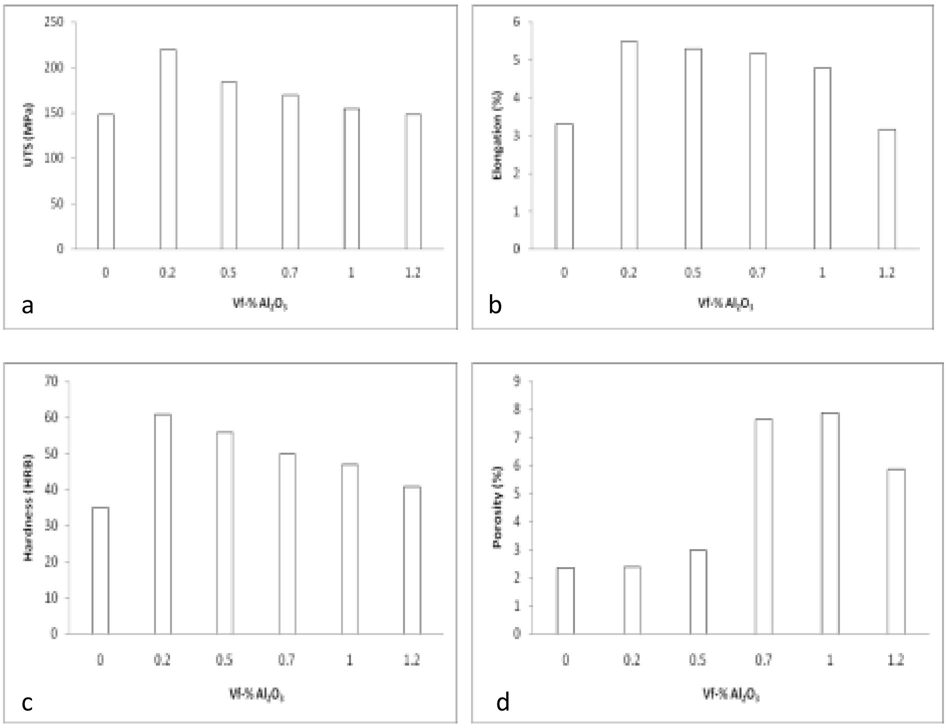


Fig. 2 . Effect of Al_2O_3 nano particles on mechanical properties of Al6061/Al_2O_3 composites a) tensile strength, b) elongation, c) hardness and d) porosity.

Tensile strength decreased because of nano alumina can not be wetted completelly by aluminium as well as the presence of micro porosities with higher volume fraction of nano alumina. The agglomeration of Al_2O_3 used has a little effect in impede dislocation movement and micro porosities in this composite and act as stress concentration in matrix. Moreover, agglomeration and micro porosities make the stress can not be flowed effectively from matrix to reinforcement. Porosity can be occured during melting process of aluminium since hydrogen was very aggressive to solve in the molten Al. Dinesh *et al.* [8] said that the addition of particulate content on molten Al until 900^{o}C would increase porosity level. Beside that, composites with higher content of reinforcement particles will increase the possibility of agglomeration and micro-porosity formation in the interface between matrix and reinforcement).

In the other hand the addition of nano alumina into Al6061 can increases the elongation of composite in almost of all volume fraction compared to Al6061 unreinforced. The highest elongation also is achieved at composites with 0.2 Vf-% Al_2O_3 its mean that the addition of nano alumina up to 0.2Vf% can increased both tensile strength and elongation. The enhancement of elongation is caused by grain refinement mechanism see Fig. 3a. The previous work [9] was also found that by this mechanism, tensile strength can be enchanced while maintaining good ductility. This can be happened because strain distribution is more homogen and stress concentration reduction [10]. The addition of nano alumina between 1 and 2 % Vf resulted decrased in elongation, because grain refinement mechanism is gained its maximum effectiveness at certain volume fraction. When the volume fraction is exceeded its critical, it will not have any significant effect on grain size [11].

Fig 2c shows the hardness of Al6061/Al_2O_3 composite with different volume fraction of nano alumina. It is clear that all composites provide higher hardness than that of Al6061 base alloy in the as-cast state. The highest hardness is obtained at composites with 0.2 Vf-% Al_2O_3. Increasing hardness due to impede dislocation movement, and grain refinement mechanism by reinforcement particles and Mg_2Si formation which occured in this composites (see Fig. 4). Mg_2Si is also contributed to the hardness. The hardnes then decrease with higher of nano alumina content. They are caused by agglomeration of nano alumina and dewetting so increasing porosity level. Porosity occured due to poor wetting between alumina particles and matrix as well as gas entrapment when stirring process and hindered liquid metal flow thus more particle clustering distributed which will decrease mechanical properties of this composite.

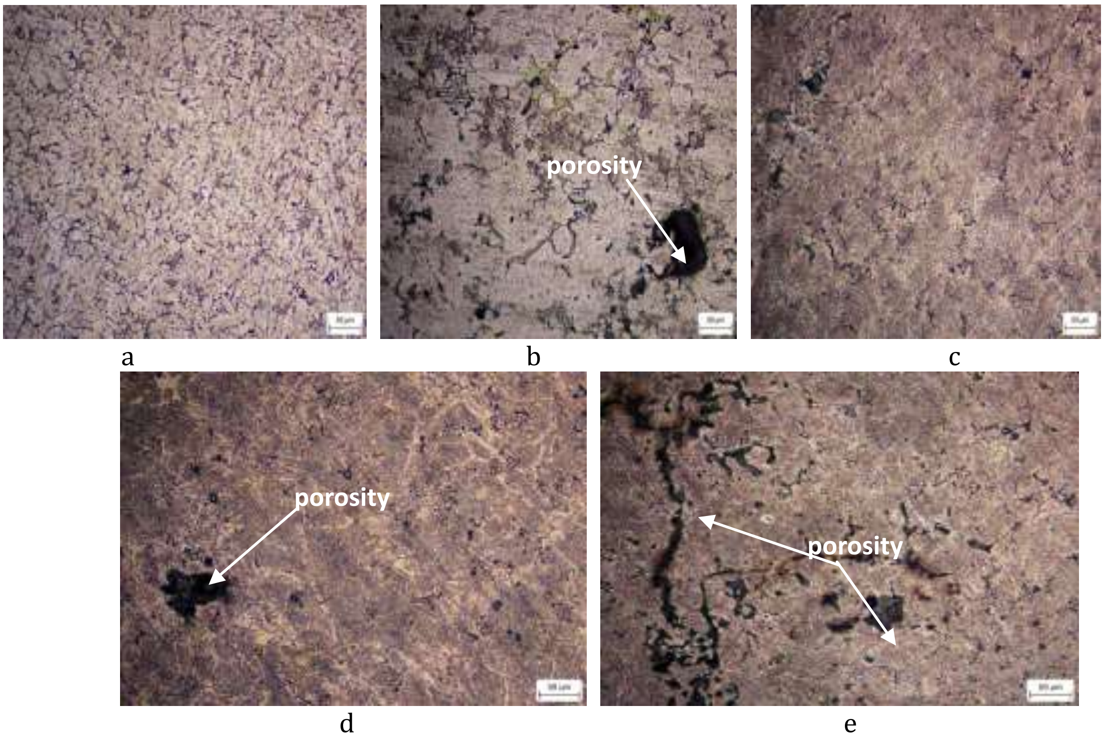
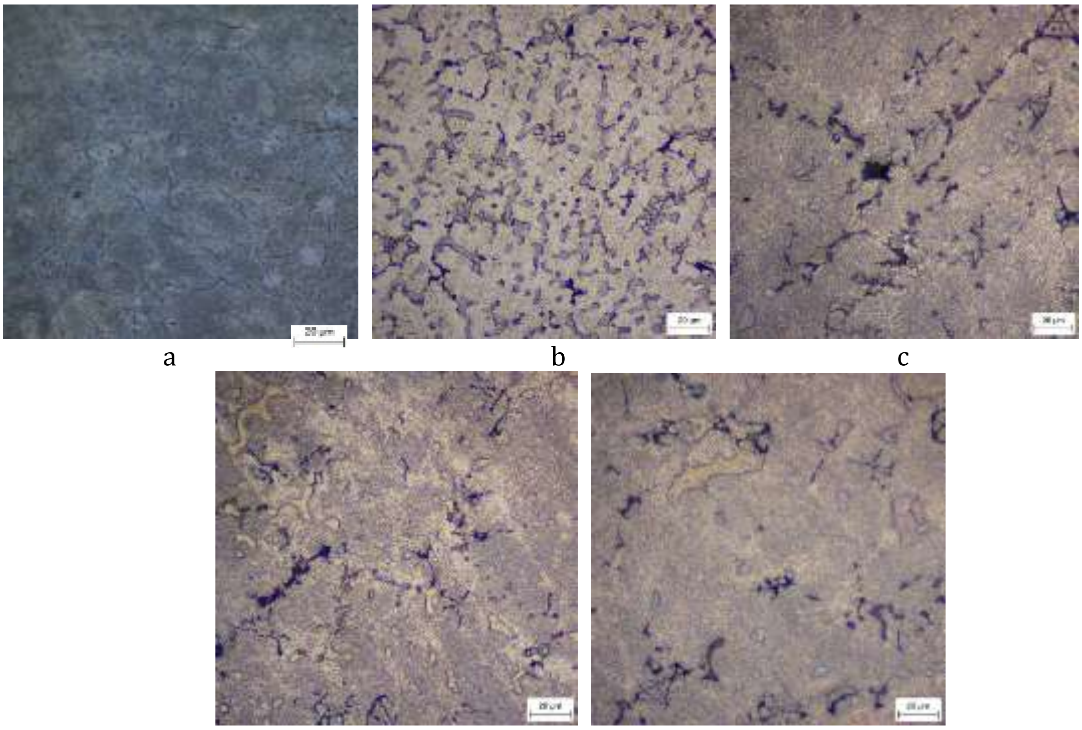


Fig. 3. Microstructure of Al6061/Al_2O_3 composites with different volume fraction of Al_2O_3 nanoparticles; a) 0.2 Vf-%, b) 0.5 Vf-%, c) 0.7 Vf-%,d) 1.0 Vf-%, and e) 1.2 Vf-%.

3.2. Microstructural Observation of Al6061/Al_2O_3 Nano Composite

Microstructures of nano composite with different volume fraction of nano alumina is shown in Fig. 3 for low magnification (200x) to determine the porosity level in composites and to confirmed the resultf of measurement by densitometry method. It seems that the addition of nano alumina into Al6061 generated more porosity than that of less nano alumina as seen in Fig. 2d. The finest grain is obtained at 0.2 Vf-% Al_2O_3. The addition of Al_2O_3 between 1 and 5 % Vf generated the grain size larger than that of composite with 0.2 Vf-% Al_2O_3. The grain refinement mechanism at 0.2 Vf-% Al_2O_3. is predicted by well distributed of nano alumina particles in Al6061 matrix that act as nucleating of grain formation [6]. Furthermore, the smaller grain size is caused by pinning effect of particle alumina that delay grain growth process [12]. The addition of nano alumina between 0.5 Vf-% and 1.2 Vf-% caused larger grain size because it is predicted of poor distributed of alumina in aluminium matrix, so grain refinement only occur in some point. Moreover, grain refinement mechanism only effective at critical volume fraction of nano alumina is achieved. When alumina content is larger than critical point, saturation happened and the effect is no longer significant [13]. Level porosity increase with increasing reinforcement content which also proved by decreasing mechanical properties of composite. The porosity content in composites is in the form of gas porosity which is occured by gas solve in the molten aluminium during casting procees and shrinkage porosity occured by early solidification which happened when unstable pouring technique.

Fig. 4 shows microstructure of Al6061/Al_2O_3 composites with different volume fraction of Al_2O_3 nanoparticles observed at higher magnification (500x). At higher magnification showed more clear the phases present in the matrix. Fig. 4a is microstructure of Al6061 unreinforced shows dendrite structure. Fig. 4b-e are microstructure of Al6061/Al_2O_3 nanoparticle composites. Composite with 0.2Vf-% shows well-distributed of Al_8Mg_5 dendrite and fine Mg_2Si chinese script is found so their existence is proved that mechanical properties of this composite improved. Fig. 4 (c-e) are also show the present of coarse Mg_2Si and agglomerate in some points therefore mechanical properties of composites at this composition are lower than that of 0.2Vf-% Al_2O_3. Beside Mg_2Si formation is also showed the sludge formation on microstructure of composite with 1.0 Vf-% and 1.2Vf-% Al_2O_3 addition. By comparing morphology and color of the microstructure with previous workso that compound is assumed as sludge which contain Al-Fe-Cr [14]. The formation of this sludge is caused by stirring process using SS 304 stirrer which has high chromium content. Seifeddine *et al.* [14] showed that high content of iron, manganese, and chromium will increase sludge excess and deteriorate the mechanical properties of materials.



a b c

d e

Fig. 4 .Microstructure of aluminium composites with (a) 0.2%, (b) 0.5%, (c) 0.7%, (d) 1.0%, (e) 1.2% volume fraction of Al_2O_3 nanoparticles.

3.3. Microstructural Observation at Composites with 0.2Vf-% Al_2O_3

This observation is carried out to see further the microstructure and phases present at composites with 0.2Vf-% Al_2O_3 under scanning electron microscope in Fig. 5. SEM image clearly shows Al_8Mg_5 dendrite structure and Mg_2Si Chinese script. EDS examination is conducted for this composites showed that at point 1 is MgO while at point 2 is $MgAl_2O_4$ which are confirmed by XRD (Fig. 6). MgO and $MgAl_2O_4$ is assumed as reaction product at interface and has good impact in the properties of composite due to better wetting than that of composites with higher Vf-% Al_2O_3.

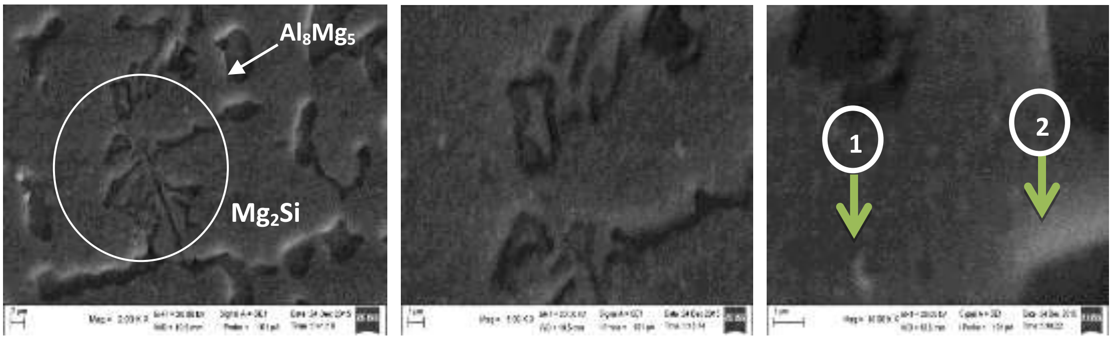


Fig. 5. Microstructure of aluminium composites with 0.2Vf-% Al_2O_3

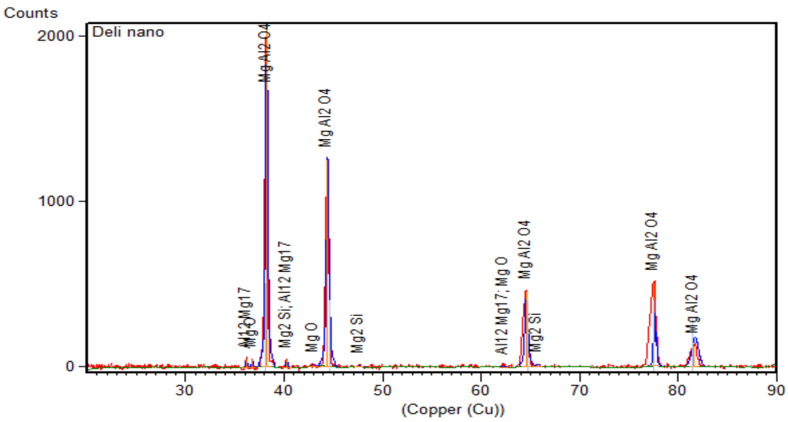


Fig. 6. XRD pattern of aluminium composites with 0.2Vf-% Al_2O_3.

The XRD also identified others phase present in composites such as $Al_{12}Mg_{17}$ and Mg_2Si. The existence of Mg_2Si inter metallic phases give hardening effect in aluminium matrix composite and they will impede dislocation movement as well as Al_2O_3 particles reinforced.

3.4. Fractography of Aluminium Composites with 0.2Vf-% Al_2O_3

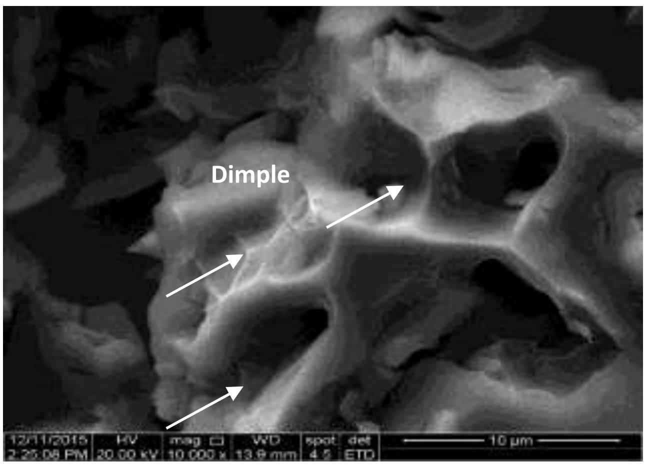


Fig. 7. Fractography of aluminium composites reinforced by 0.2 Vf Al_2O_3.

Fig. 7 shows the fracture surface of aluminium composite with 0.2Vf-% Al_2O_3 which was analysed by FESEM. It is clear that composites with . 0.2Vf-% Al_2O_3 is ductile due to the formation of dimples on the fracture surface and it is confirmed by higher elongation of this composites than that of composites with higher Al_2O_3 content. The effect of Al_2O_3 nano particles addition into Al6061 not only improved in strength and hardness but remain ductility specially for composites with 0.2Vf-% Al_2O_3.

4. Conclusion

In general, aluminium composite reinforced by Al_2O_3 nanoparticles can be fabricated by stir casting process. The addition of Al_2O_3 nanoparticles improved mechanical properties of base alloy such as strength and hardness and still remain the ductility. The optimum mechanical properties is achieved for composites with 0.2 Vf-% of Al_2O_3 nanoparticles which has tensile strength, elongation and hardness are up to 220.16 MPa, 5.8% and 61 HRB respectively. The existence of Chinese script Mg_2Si also contributed to strengthening mechanism in this composites.

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Deliana Ramdaniawati has just finished her undergraduate, she join with this project since 2016 to take her final year project under my supervision. Now she is applying to work in the manufacturing company in Indonesia.

Donanta Dhaneswara Ph.D is my colleague; he is an Associate Professor in the same department with me. He has experienced in casting process specially aluminum casting and mesoporous material