

Topic : Metallurgy and Materials Engineering

**Effect of Titanium Addition on Characteristic of Al6061/ Al₂O₃
Reinforced Composites Produced by Stir Casting Process**

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Abstract

Al6061/Al₂O₃ reinforced composites have been successfully produced by stir casting process. The master alloy Al6061 as a matrix composites was prepared by melting at 800 °C and mixed with various of Al-5TiB from 0.02 wt-% to 0.15 wt-%, 0.025 wt-% Al-5Sr as modifier and 10 wt-% Mg as wetting agent respectively. The addition of Al-5Ti-1B on Al6061 is to strengthen the matrix phase and Al-15Sr is to bring the effect of matrix modification. The role of Al-5Ti-1B is to be grain refinement agent which mainly provides more nucleating sites and restricts the growth of grains and the role of Al-15Sr is to be a modifier of the morphology of the second-phase particles. The molten aluminium then flushed with argon to remove all the gas bubble from liquid aluminium, then stirred for 3 minutes to produce a perfect vortex flow, afterwards Al₂O₃ was poured and stirred again for 3 minutes. The composites produced then characterised both mechanical properties and microstructural analysis. The optimum mechanical properties of composite is obtained at 0.02 wt-% Ti addition with tensile strength of 204 MPa and elongation of 11.75 % at 0.09 wt-% Ti, while hardness is 50 HRB. These preferable properties are also supported by perceiving its microstructure. The role of Al-5Ti-1B is to be grain refinement agent which mainly provides more nucleating sites and restricts the growth of grains. It is obvious that the smaller-fragmented area of aluminium and finer morphology of the second phases contributed to increase mechanical properties

Keywords : Al6061; Al₂O₃ particles; Al-5TiB; composites; stir casting

1. Introduction

Technology of materials is significantly being an important demand for the human being to provide their needs and ease their activities for nowadays. The development of technology nowadays needs material's properties that can not be found in metal, polymer, and ceramic themselves. This properties cover high specific strength, high temperature creep resistance, and better fatigue strength, therefore composite is

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made (Akio et al, 1999 and Mussert et al, 2002). This material will be used for application that need high performance such as automotive, military and aerospace industries. For instance, the demand for more lightweight materials, fuel efficient and enhanced performance automobile stimulates the research and development of many materials have to be fulfilled by the automotive industries (Bussines Market, MMC,2013).

One of the recent research is carried out to improve mechanical properties with high performance and lighter weight. The present study has been subjected to extensive research of using Al alloy to be developed as aluminum matrix composite which is combined with ceramic particles reinforced. The most common Al alloy used for automotive application, is Al alloy 6 series wrought product which consists of Mg and Si as its prime constituents [Dobrzanski and Woarezyk, 2005]. Al-Mg-Si is generally used in structural, aircraft, nuclear application and etc (Calister, 1994) because it has medium strength among other Al alloys, which is about 185 to 215 MPa (Anne, 2013). This number of strength is considered to be sufficient for the strength required before being continuously treated. For it has major constituents of Mg and Si, so it categorizes as the heat-treatable alloy and subsequently be increased in strength (Mitchell, 2012). The selection of Al-Mg-Si alloy is also due to the strengthening process that might be involved. For instances, it could be subjected to solid solution strengthening, grain refinement strengthening and aging process to enhance its mechanical properties according to its application (Anne and Maman, 2011). While aluminium composites is considered to improve such mechanical properties by adding ceramic particles. In general, ceramic material which is selected for being the reinforcement is Al_2O_3 particles. The particles-type of reinforce is chosen by its isotropic characteristics which tends to be more durable if being fabricated and economically in process rather than fibers and whiskers-type of reinforce (Zulfia and Hand, 2002). The addition of reinforce particles to the matrix is likely to give more strengthening effect in purpose. This will be obtained as if the reinforcement is fully wet by the matrix. Moreover, the particles-type of reinforcement is hard to be completely wet therefore need a dopant to promote wetting between Al and ceramic. Stirr casting process is selected to aid the randoly distribution of ceramic particles in molten aluminium

According to the previous research, the highest strength of composite was obtained at the composition of 10 wt% of Al_2O_{3p} with the value of 190 MPa without any additional constituents for modification (Anne, 2013). The present study is to obtain higher mechanical properties by modification of Al-5Ti-1B master alloy and addition of the modifier, Al-15Sr and the wetting agent of Mg. The amount of 0.1 wt% Ti addition in Al 6061 results in about 185 to 215 MPa (Samuel et al, 2014). At the same point, the optimal Ti addition to the composite 6061/ Al_2O_{3p} which is below 0.05 wt% gives 198.5 ± 10.2 MPa of the yield strength and 264.3 ± 5.1 MPa UTS, respectively (Zhiqiang et al, 2005). Therefore, this present study is in purpose to define the optimum amount of Ti addition from 0.02 to 0.15 wt% to give remarkable mechanical properties.

2. Experimental Procedure

2.1. Making of Al6061/ Al_2O_3 composites

The composites in this study were prepared from the rod of Al 6061 alloy, Al-5TiB and Al-15Sr as master alloy matrix, 10 wt% Mg ingot (99.99%) as a wetting agent, and 10 Vf% of Al_2O_3 particles with size of $65\mu m$ as reinforcement. The raw materials, Al 6061 alloy ingots were melted in electric furnace at $800^\circ C$, then Al5TiB and Al15Sr were added into molten aluminium then stirred at 415 rpm for 3 minutes and flushed with Argon for 1 minute. The Al5TiB used was various from 0.02 to 0.15

wt%, while Al-15Sr was constant at 0.03wt%. At the same time, the Al_2O_{3p} was preheated in the furnace at 1000 °C for approximately one hour to decrease all moisture on the surface of particles. The chemical composition of the Al 6061 ingots were analyzed by Optical Emission Spectroscopy is listed in Table 1.

Table 1. The composition of Al 6061

Alloy	Al	Mg	Si	Cu	Mn	Sr	Ti	B	Fe	Cr
wt%	96	2.69	0.819	0.217	0.0168	<0.0001	0.0073	<0.0005	0.161	0.0577

After the Al ingots being fully melt, the process of degassing was carried out for 2 to 3 minutes by argon. The molten alloy was held and stirred for 3 minutes to homogenize the composition. Next was the preheated Al_2O_{3p} mixed to the molten alloy and continuously being stirred to aid the randomly distribution of Al_2O_{3p} in the molten matrix. The stirring process was also followed by the degassing process to ensure all the gas remove from the aluminium molten. After all of the processes finished then the molten composite was being casted to the SKD61 -metal mold and it was cooled in the air.

2.2. Characterization of AlA356/ Al_2O_3 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 E8M-09. 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. The porosity of composite was measured using Archimedean method. The microstructure of composites was analysed following the metallography procedure, the samples representative was prepared by grinding using emery paper started from 80#, #150, #240, #400, #600, #800, #1000, #1200 to #1500, then polished using alumina powder to remove strach from grinding process, then etched with 0.5% HF for 1 minute. All samples preparataion were observed using OLYMPUS BX41M-LED optical microscope and further analysys using Scanning Electron Microscope (SEM).

3. Results and Discussion

3.1. Chemical composition of Al6061/ Al_2O_3 cast composites

Table 2 shows the chemical composition of Al6061/ Al_2O_3 composite analysed by Optical Emission Spectroscopy (OES). The composition design was different from OES result. The Mg content should be 10wt% or less but the OES measured was 12wt%, while Ti closed to material balance which was design before casting but for Sr the OES measured was strayed away from designed alloy. It is assumed that deviation can be caused by the over reading of the instrument to measure the Mg and Sr content inside of the material.

Table 2. Chemical composition of Al6061/Al₂O₃ cast composites (wt-%).

Composites	Mg	Si	Ti	B	Sr	Cr	Fe	Mn	Al
1	>12	0.57	0.02	<0.0005	0.03	0.25	1.04	0.05	Bal
2	>12	0.56	0.03	0.01	0.03	0.33	1.50	0.06	Bal
3	>12	2.49	0.09	>0.025	0.065	0.271	1.62	0.766	Bal
4	>12	2.14	0.122	>0.025	0.047	0.198	1.42	0.769	Bal
5	>12	2.00	0.15	>0.025	0.048	0.328	1.62	0.60	Bal

3.2. Effect of Ti on mechanical properties of Al6061/ Al₂O₃ composite

The mechanical properties of Al6061/Al₂O₃ such as tensile strength, elongation and hardness are shown in Figure 1. They are seen that composites with 0.09 wt-% Ti addition has the highest both tensile strength and elongation (Fig. 1a,b), even though the addition of 0.02 wt-% Ti has the highest strength with the value of 204 MPa. The hardness of composites is 50 HRB which is optimum obtained at 0.12 wt-% Ti addition. There are many factors can cause the highest of mechanical properties, some of them might be the distribution of the Al₂O_{3p}, the porosity formed in the composites, the wettability of the matrix, and the formation of intermetallic compounds. The addition of Ti in the as cast composite is to be known for the effect of grain refinement. It brings the effect of refinement by heterogeneously nucleating the TiAl₃ and TiB₂ and restricting the growth of the grains (Kashyap and Chandrasekar, 2001). Heterogeneous nucleation involves peritectic reaction. The result of peritectic reaction, TiAl₃ and TiB₂, will become nucleates and continuously become sites for nucleus of Al. This condition subsequently will give more nucleating process rather than growing process of the Al. Thus, the process of refinement consists of two mechanisms, first is serve nucleating sites for Al nuclei and restrict the growth of Al. Boron is also counted as the refinement agent because it reacts along with Ti to form TiB₂ which are insoluble in Al. Even so, the superior effect of refinement is still generated by the formation of TiAl₃ intermetallic compound because it is believed that the lattice disregistry of TiAl₃ is smaller than TiB₂ (Sigworth and Johnsson, 1993).

The refinement will result to the finer grain of matrix Al generates the remarkable mechanical properties. Besides, the addition of Al-15Sr will also result good modification to the formation of the second phases. In this study, the Ti addition up to 0.02wt% and constant number of 0.03wt% Sr gives the highest UTS among other samples. It is firstly concluded that the refinement process takes place in the as cast composites.

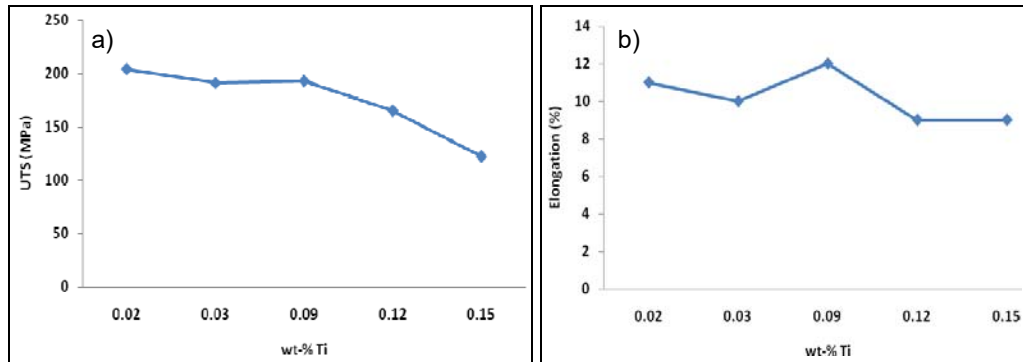


Figure 1. Mechanical properties as-cast Al6061/Al₂O_{3p} composites. a) Tensile Strength, and b) Elongation,

The hardness and porosity are shown in Figure 2. The highest number of hardness is obtained in composite with 0.12 wt-% Ti with the value of 50 HRB, and the lowest porosity is obtained at 0.09 wt-% Ti. The effect of Ti on grain refinement of Al has good impact to increase hardness. The other factor matters to the properties except the refinement process is the casting process. It will result casting defects, such as formation of shrinkage and porosity which deteriorates the mechanical properties. It seems that the addition of 0.09 wt-% Ti has high tensile strength, elongation and hardness due to the lowest of porosity. But here the highest UTS which is obtained at 0.02 wt-% Ti shows higher number of porosity than that of 0.09 wt-% Ti. This contradicts with the theory that the small number of porosity will give higher strength. Perhaps stress concentration that brings negative effect to the composite.

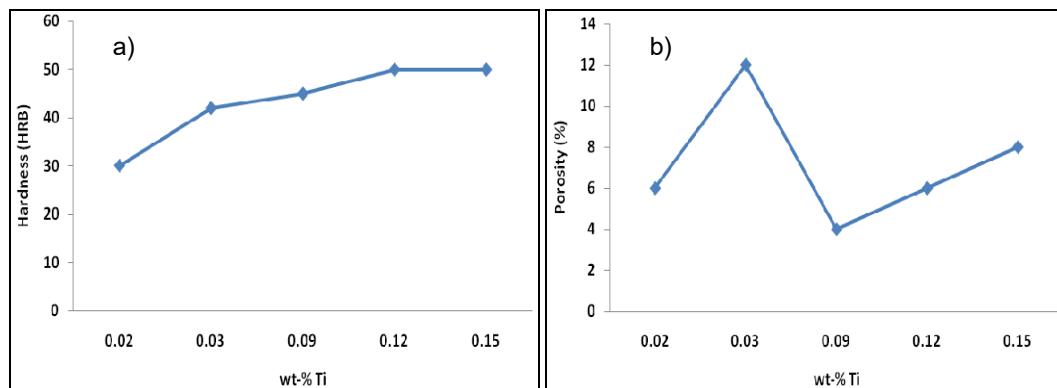


Figure 2. Mechanical properties as-cast Al6061/Al₂O_{3p} composites. a) Hardness, and b) Porosity

3.3. Effect of Ti on microstructure of Al6061/Al₂O₃ composite

The microstructure of the as-cast Al-Mg-Si/Al₂O_{3p} composites was examined using optical microscope in Figure 3. The most obvious difference between the as-cast unmodified and modified one is the structure of the matrix for each. The dominant structures of the modified ones are the likely-fragmented dendrites of Al and the fragmented-to-globular like of Al (Fig.3b). From these kind of morphology, it could be stated that the addition of Ti and Sr plays an important role which will definitely affect their properties. The inter-dendritic area has also been an important thing to be noticed that it is related to the formation of other constituents at the condition of as - cast

(Bartłomiej and Bogusława, 2015). For the composites 1 and 3, the difference are shown at the area of inter-dendritic which becomes smaller.

The fragmented area of dendritic is shielded by constituents as the second phases at its inter-dendritic area. These second phases formed at the inter-dendritic are predicted as the quaternary of Al, Al₃Fe, Al₃Mg₂, Mg₂Si (Liu et al, 1999). This quaternary eutectic blooms out as the network-like according to its solidification path, which is clearly shown by the arrow see Fig.4. The solidification path of this quaternary eutectics is commonly shown by the quaternary diagram of Al-Fe-Mg-Si because of the high presence of Fe in the rod (Belov and Eskin, 2005). The prediction to be the quaternary phases is due to the morphology shown according to the atlas of microstructure. The transition of smaller inter-dendritic area from Fig. 3a to 3b verifies that there is combination by the presence of Ti and Sr (George Van Der Voort, 2004).

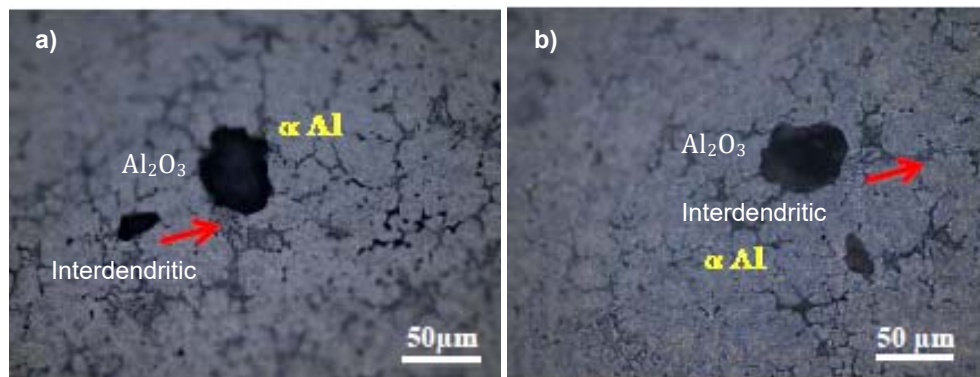


Figure 3. Microstructure of as-cast Al6061/Al₂O_{3p} composites with composition 1(a) and 3 (b)

When the microstructure is seen at lower magnification, both composites clearly exhibit other constituents which are represented in Fig. 4. The needle like indicates the phase of Al₃Fe. This morphology of Al₃Fe is commonly alike to the morphology of Al-Fe-Si with shorter appearance. Then, the second particles and snow flakes are predicted to be Al₃Mg₂ and Mg₂Si, respectively. All of these predicted constituents were according to its possible solidification sequence by the quaternary diagram and the morphology of phases is also confirmed by George Van Der Voort (see Table. 3). The presence of Ti and Sr also exhibit an important role that the morphology of each phases in composites 3 becomes smaller than that of in composite 1.

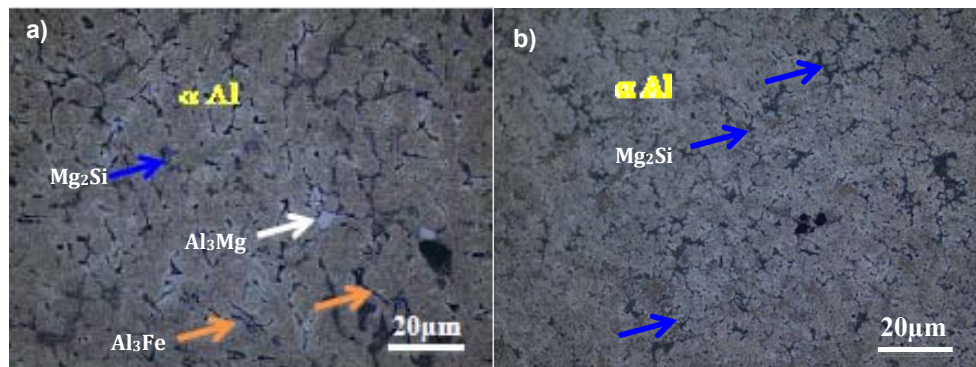


Figure 4. Microstructure of the as-cast Al6061/Al₂O₃ composite with composition 1 (a) and 3 (b)

Table 3 : Phases identification performed according to ASM (George Van Der Voort, 2004).

Phase	Morphology
α Al-Fe-Si	chinese script, blocky
β Al-Fe-Si	needle, platelet
Mg ₂ Si	chinese script, irregular, polyhedral
Al ₃ Fe	platelet
Al ₃ Mg ₂	polyhedral, irregular, rounded
Al-Si	platelet, irregular, polyhedral blocky

The transition of the Al matrix is obtained at the composition of 0.03 wt% Ti and 0.025 wt% Sr which is in the composite 2 (Fig.5). It is clearly shown that the almost fragmented-dendritic like of Al turns out into fragmented-to-globular like of Al (Fig. 5a). The case is similar to the study presented by Martorano and Capocchi et.al, 2005. The result of their study is the transformation of columnar dendritic of Cu-8Sn alloy to be globular by the addition of Cu-50Zr alloy. This is categorized to become an evidence of the role of Ti addition takes place. Al-5TiB which is added to the molten metal will act as inoculant, such as Ti₃Al or TiB₂. The Ti₃Al dissolved and TiB₂ undissolved which go into solution will become nuclean for nucleus of dendritic Al and will subsequently restrict the growth of dendritic Al (Bartlomiej and Boguslawa, 2015).

Being compared to the composite 1 and 3, the most globular-like grains of Al in Fig. 5 could be examined at low magnification. It more likely indicates that refinement takes place at the Ti addition up to 0.03 wt% and the network-like which tends to be inter-dendritic area considering as chinese script Mg₂Si (Fig. 5b). It is considered to the effect of modification by Sr is also ocured in this study as well as previous study by Tong Min et al, 2014.

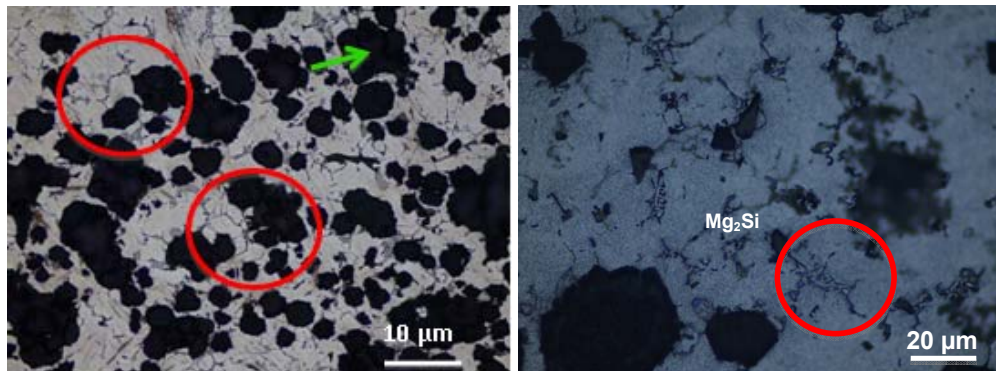


Figure 5. Microstructure of as-cast Al6061/Al₂O₃ composites with composition 2.

The microstructure of composite 4, with Ti addition up to 0.122 wt% shows bigger globular of Al than that of composite 2 (Fig. 6a). As compared to the composite 2, this composition did not undergo appropriate refining process but it did if compared to the composite 1 and 3. The more addition of Ti which is up to 0.15 wt% represented by the composite 5 (Fig. 6b) gives deteriorated appearance in the microstructure. It is clearly shown that the globular of Al shielded by the network-like of Mg₂Si becomes bigger and the microstructure also shows the formation of some deteriorated phase, like it is predicted as Al₃Fe which is caused the mechanical properties become low for this composite.

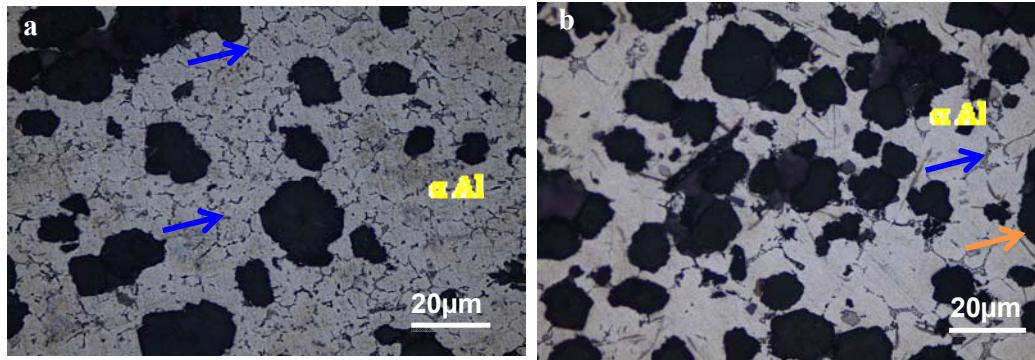


Figure 6. Microstructure of as-cast Al6061/Al₂O_{3p} composite with composition 4 (a) and 5 (b)

It could be concluded that the addition of Ti up to 0.1 wt% will not give any significant effect of grain refinement of Al matrix. It is proved by the result of its tensile strength of the samples which decreases as the number of Ti increase. It is clearly showed that the addition of refining agent has its optimum level and too much addition has it be insignificant. At some point, modification by Ti and Sr in this study is not the only thing signifying the higher strength of the composites achieve. From the level of porosity among all, composite 2 gives the higher one (Fig. 2). By gaining this result, composite 2 should had been the lowest strength rather than others, but in this study is seen odd results since tensile strength and hardness has high value eventhough still lower than composite 3. Yet it is a composite, the role of its reinforcement also plays important to raise its mechanical properties. From the micrographs of each composite, it could be seen that composite 2 has better distribution of Al₂O_{3p} (Fig. 5). All of these, such as the distribution of Al₂O_{3p}, the refinement effect, the porositycontent and phases formation will affect to the mechanical properties of composite.

3.4. XRD Analysis of Al6061/Al₂O₃ composite

The phases present in composites is represented in composition 2 are Al, AlSi, Mg₂Si, AlMg, AlFe, MgAl₂O₄ as shown in XRD analysis in Fig. 7. Refer to microstructure of composite above the phases present has been confirmed by George Van der Voort [15] and XRD analysis.

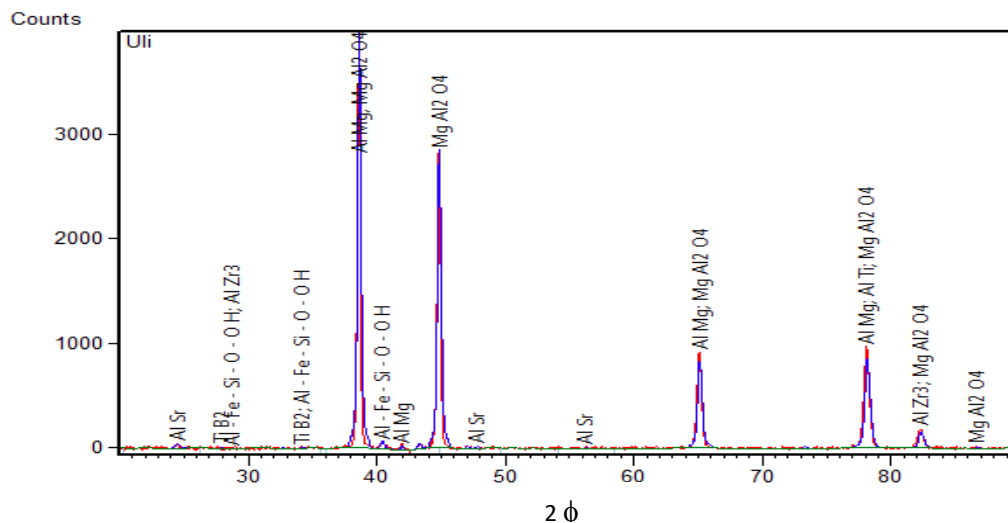


Figure 7. X-Ray Diffraction Pattern of Al6061/Al₂O₃ Composite for composition 2

4 .Conclusion

Based on the results obtained in this work, there are some major conclusions that could be highlighted as follows :

1. The optimum mechanical properties is obtained in compsites with Ti addition of 0.09 wt-% both tensile strength and elongation as well as the lowest porosity content but the highest hardness found at composition of 0.12 wt-% Ti, further addition of Ti up to 0.15 wt% the mechanical properties decrease.
2. Ti addition gives transformation of each matrix Al morphology and so does Sr addition gives modification to the Mg₂Si and Al₃Fe phases in the form of chinese script.
3. The phases present in composites is Al, MgAl₂O₄, Mg₂Si, Al₃Fe and AlSi analysed by XRD.

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References

- Akio, K., Atsushi, O., Toshiro, K., Hiroyuki, T. (1999). Fabrication, Process of Metal Matrix Composite with Nano-size SiC Particle Produced by Vortex Method. *J Japan Institute Light Metals*, 49, p149 – 154.
- Anne, Z. (2013). Characteristic of Al-Mg-Si/ Al₂O₃ Produced by Stirr Casting Procees, Research Project. Unpublished, Universitas Indonesia,
- Anne. Z and Maman, K.A. (2011), Effect of Artificial Aging (T6) on Microstructure of Al-AC8H/Al₂O₃ MMC Produced by Stir Casting Route, *Advanced Materials Research Vols. 328-330* (2011), *Mechatronics and Materials Processing I*, pp 1552-1555
- Bartlomiej. D and Boguslawa, A.C. (2015), "The Microstructure of AlSi7Mg Alloy in As-Cast Condition," *Solid State Phenomena*, pp. 3-10.
- Belov, N. A. and Eskin, D. G. (2005), *Multicomponent Phase Diagrams : Applications for Commercial Aluminum Alloys.*: Amsterdam, Boston, Elsevier
- Business Wire. MMC Market - Global Industry Analysis, Size, Share, Growth, Trends, and Forecast 2013-2019. [Online]. <http://www.businesswire.com/news/home/2-15-323--5591/en/metal-matrix-composites-mmc-market-rides-success#.VXMbyqqko>
- Callister, W. D. (1994), *Materials Science and Engineering: An Introduction*. New York: John Wiley and Sons, Inc., vol. 3rd.
- Dobrzanski, L. A. and Woarezyk, F. (2005) "Properties and Corrosion Resistance of PM Composite Materials Based on EN AW-Al Cu4Mg1(A) Aluminum Alloy Reinforced with The Ti(C,N) Particles," in *International Scientific Conference on The Contemporary Achivements in Mechanics Manufacturing and Materials Science*, pp. 289-295.
- George F. Vander Voort. (2004), *ASM Handbook, Commitee, ASM Metal Handbook*, , Ed: ASM International, Vol. 9.
- Kashyap. K. T. and Chandrasekar. T. (2001), "Effects and Mechanism of Grain Refinement in Al Alloys," *Bull. Mat. Sci.*, vol. 24, pp. 345-353
- Liu, Y. L, Kang, S. B. and Kim, H. W. (1999), "The Complex Microstructures in an As-Cast Al-

- Mg-Si Alloy," *Materials Letter*, vol. 41, pp. 267-272
- Martorano, M. A. and Capocchi. J. D. T. (2005), "Dendrite Structure Control in Directionally Solidified Bronze Castings," Department of Metallurgical and Materials Engineering, Polytechnic School, University of Sao Paulo, Sao Paulo, 05508-900.
- Mussert, K.M., Vellinga, W.P., Bakker, S. (2002). A Nano-indentation Study on the Mechanical Behaviour of the Matrix Material in an AA6061 – Al₂O₃ MMC. *Journal of Materials Science*, 37. p789 – 794.
- Mitchell C. A. (2012) "A Study of The Powder Processing, Tribological Performance and Metallurgy of Aluminium-Based, Discontinuously Reinforced Metal Matrix Composites,," Napier University, Edinburgh.
- Samuel. E, Golbahar. B and Samuel, A.M. (2014), "Effect of Grain Refiner on The Tensile and Impact Properties of Al-Si-Mg Cast Alloys," *Journal of Material and Design*, vol. 56, pp. 468-479
- Sigworth. G.K and Johnsson. M. (1993), "Study of The Mechanism of Grain Refinement of Aluminum After Addition of Ti- and B- Containing Master Alloys," *Metallurgical Transactions A*, vol. 24A, pp. 481-491
- Tongmin. W, Zhao. Y, and Huijun. K. (2014), "Effect of Sr on The Microstructure and Mechanical Properties of In-Situ TiB₂ Reinforced A356 Composite," *Materials and Design*, vol. 64, pp. 185-193.
- Zhiqiang. Y, Wu. G, Jiang. L and Sun. D. (2005), "Effect of Coating Al₂O₃ Reinforcing Particles on The Interface and Mechanical Properties of 6061 Alloy Aluminium Matrix Composites," *Materials Letters*, pp. 2281-2284,
- Zulfia. A , and Hand. R.J. (2002), The production of Al-Mg alloys into SiC Preform in Production of Metal Matrix Composites, *Journal of Materials Science*, Vol 37.No. 5, pp.955-961