



Investigating the Microhardness Behavior of Al6061/TiC Surface Composites Produced by Friction Stir Processing

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ABSTRACT

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The continual pursuit of fuel efficiency, cost-effectiveness, and desirable physical and mechanical properties of materials has steered researchers towards the latest generation of aluminum matrix composites for automotive and aerospace applications. In this context, the present study investigates the microhardness behavior of Al6061/TiC composites produced by friction stir processing. The morphological characteristics of the produced surface composites were analyzed using optical microscopy and Scanning Electron Microscopy (SEM). SEM micrographs confirmed the presence of TiC particles and their uniform distribution within the aluminum matrix. The mechanical properties of the composites were explored using a microhardness tester, revealing a distinctive feature of the Al6061/TiC composites - a 35% increase in microhardness value compared to the base Al6061 alloy. This improvement in microhardness can be attributed to enhanced interfacial bonding, obstructions in dislocation movement, and grain refinement, all contributing to Hall-Petch strengthening.

1. INTRODUCTION

In contrast to traditional monolithic engineering materials, composite materials provide the ideal combination of qualities needed to design and manufacture various cost-effective engineering products and structures. Due to their enticing features, including low density, high specific stiffness, and better dimensional stability, aluminum (Al) matrix composites have brought much interest as lightweight materials possible in vehicle and aviation structural applications [1-3]. Ceramic particles, particularly those of TiC, TiB₂, B₄C, and SiC, have excellent stiffness, strength, and hardness and are frequently employed as reinforcing particles in Al matrix composites [4]. Refinement in the microstructure could improve the mechanical characteristics of composite materials, such as their hardness and tensile strength, following the Hall-patch relationship [5].

As potential approaches for fabricating metal matrix composites (MMC) reinforced with particles, friction stir additive processing [6] and friction stir processing (FSP [7] have appeared as promising methods in recent years. In the FSP approach, a specific tool's rotation and linear movement causes plastic deformation at a high temperature, enabling the incorporation of ceramic particles in the matrix [8]. The consistent distribution of particles is the most important aspect of the manufacturing process, which can be accomplished by

carefully choosing process parameters, like transverse speed and tool rotation [9, 10], and/or the number of passes [11]. The FSP approach has a lot of benefits viz. a homogeneous dispersion of reinforced particles and grain refinement, which is brought about by dynamic recrystallization [12]. Thus, MMCs that have undergone FSP processing have improved mechanical characteristics.

The advantages of aluminum alloy 6061, such as its high strength and light weight, make it widely used in the aircraft and automobile industries. For any exposed surface applications, hardness of components or machinery parts is an important factor to be considered. Addition of TiC particles in Al6061 revealed high resistance to indentation or scratch on the produced surface composites. Many researchers explored the effect of different ceramic particulates as fillers in aluminum alloys on wearing capacity [12, 13]. But there is little literature available regarding addition of TiC in Al6061 using FSP route of fabrication. In the present work, microstructural and microhardness behavior of Al6061/TiC composites produced by FSP has been investigated.

2. MATERIALS AND METHODOLOGY

A detailed experimental investigation was conducted for fabricating the defect free surface composites with enhanced

microhardness behavior of Al6061/TiC composites.

2.1 Fabrication of composites

The Al6061 alloy plates of size 150 mm×150 mm with 5 mm standard size (purchased from Samnai Energy and Engineering SDN. BHD., Malaysia) were used as base alloy matrix. The chemical composition of the received Al6061 obtained from the company is represented in Table 1. The TiC nano particles between size 90 nm and 800 nm were utilized as a reinforcement with 99.9% purity. The chemical composition of alloying elements are within permissible range as of ASTM B209.

Table 1. Chemical compositions of Al6061 alloy

Chemical Compositions (Wt.%)							
Mn	Mg	Fe	Si	Cu	Zn	Cr	Al
0.12	0.82	0.39	0.56	0.23	0.02	0.18	Bal

Friction stir processing is a recently established superior green production method along with various environmental and metallurgical benefits compared with orthodox composite fabrication techniques. As depicted in Figure 1, in the course of FSP, a non-consumable rotational tool with a shoulder and pin is first injected, and later it is pushed into the matrix along a predetermined track. Owing to the friction between the spinning tool's shoulder and the specimen, heat is produced, and the revolving tool's pin provokes the heated material as it flows across the rotating pin.

A non-consumable tool with specific straight cylindrical profile of H13 steel having diameter 20 mm, the length 25 mm, diameter of pin 6 mm, pin length 4.5 mm was produced. Consequently, the friction stir processing was carried out with suitable process parameters, for instance rotational speed of tool 1000 rpm, feed rate of tool 40 mm/min, tool tilt angle 2.5°, with force of 10kN and no intersecting for the following pass.

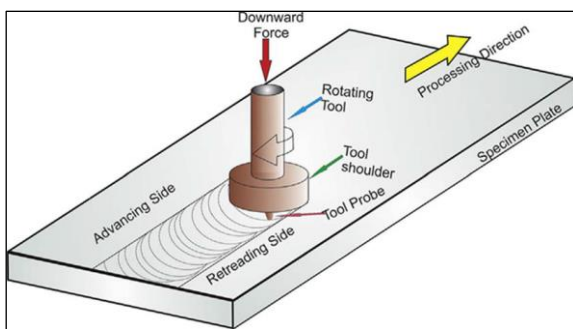


Figure 1. Illustration of composite development by friction stir processing technique [14]

Figure 2 illustrates the steps of experimental work to carried out the investigation. The Al6061 plate is undergone the Friction Stir Processing to fabricate the samples, as shown in first section of the figure. The produced composite samplesl has undergone the processes of mounting by using automatic mounting machine, grinding and polishing machine to observe the microstructure and microhardness testing as represented the second section of Figure 2. For each sample, the surface being evaluated is coated with a release agent, which is then adhered to with phenolic powder for approximately 20 minutes each. The sample is then polished using 400, 600, 800,

and 1200 grit of Silicon Carbide (SiC) paper, each using water as a lubricant for approximately 15 minutes. The sample is held perpendicular to the grinder's movement and parallel to the next SiC paper with different grit sizes as shown in Figure 2, and the procedure is repeated for the next grit sandpaper.

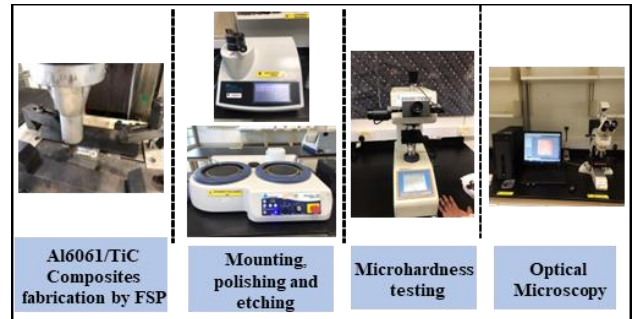


Figure 2. Illustrations of different experimental steps adopted for the present investigations

The sample then went through a polishing process with a velvet cloth without any water flow. Then apply 3 μm diamond polishing compound spray to the surface of the velvet cloth to enhance the surface of the sample. Techniques perpendicular and parallel to the movement of the grinding machine are applied until the sample is mirror polished. The next step is to etch the composite with Keller's etchant. This is a consisting of several types of liquids that visualize grain boundaries. Measure distilled water, nitric acid, hydrochloric acid, and hydrofluoric acid up to 190 mL, 5 mL, 3 mL, and 2 mL, respectively. Then soak the sample in fresh caustic alkali for 30 seconds and rinse with tap water around 10 seconds and straight away get it into the oven for dry purpose.

2.2 Characterizations

Scanning electron microscopy was used to assess the microstructural characteristics of the produced samples. At a sufficient accelerating voltage, the highest resolution microstructures were captured. Micro-Vickers hardness testing machine (Leco LM 247 AT, Saint Joseph, MO, USA), as illustrated in Figure 2. The standard test method was utilized to evaluate the microhardness (HV) of composite material. The diamond shaped indenter was subjected to a force of 0.5 kgf for 10 s. Hardness was assessed on the polished and etched samples at 6 separate positions and the median value of HV was noted. The well-polished and Keller etched portions of the surface composites were exposed to observe the optical microscopic images, homogenization of the grain size was noted after adding the TiC particles.

3. RESULTS AND DISCUSSIONS

3.1 Microstructural analysis

Microstructural analysis of prepared composites were observed using optical micrography and SEM imaging. Figure 3 depicts the SEM images of as received matrix Al6061 and reinforcement TiC particles. Figure 3(a) represents the SEM micrograph of Al6061 plate, and Figure 3(b) depicts the TiC particles that has been utilized as a reinforcing element. It is observed from Figure 3(b), that TiC particles are irregular in shape and varies in size.

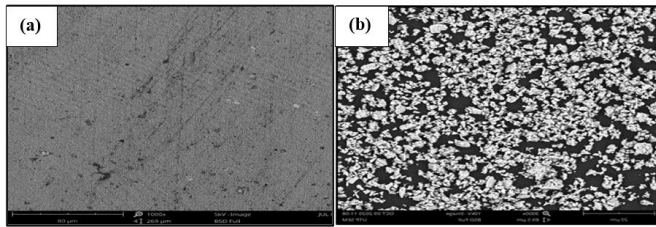


Figure 3. Materials utilized for developing the composite. (a) SEM micrograph of Al6061 base alloy, (b) SEM image of reinforcement TiC powder

Figure 4 depicts the SEM images of TiC reinforced Al6061 based surface composites. The micrographic image exposes the interface of the TiC particles and the matrix aluminum. For all composite samples, good interfacial bonding of reinforcement particles with the aluminum 6061 matrix was observed. Figure 4(a) illustrates the SEM image of Al6061-1wt.%TiC composites, few surface cracks and micropores were observed. The uniform distribution of TiC particles was achieved for 3 wt.% of TiC as shown in Figure 4(b). Additionally, for the chosen sequence of the process parameters, the absence of flaws like pores indicates good material movement throughout the processed zone. To improve strong interfacial bonding with the base aluminum, sufficient plasticization during FSP led to homogenous spreading and fragmentation of the filler particles. When temperatures are at their optimum level, good interfacial bonding increases strength; however, when processing temperatures are comparatively higher, poor interfacial bonding among the aluminum and ceramic particles reduces interfacial strength.

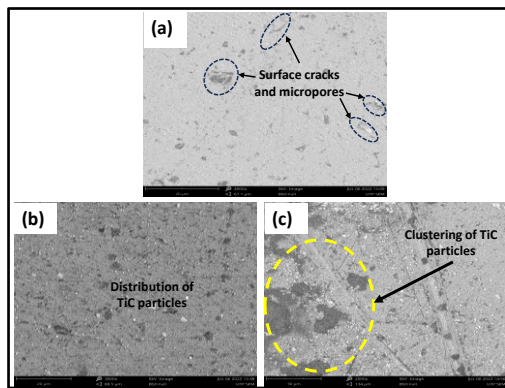


Figure 4. SEM micrograph of composites (a) Al6061/1wt.% TiC, (b) Al6061/3wt.% TiC, (c) Al6061/5wt.% TiC

The aforementioned observation leads to the conclusion that FSP is suggested as a possible method for producing surface composites. The characteristics of the technique, which produces composites in a solid state without melting, have an impact on the homogenous distribution of ceramic particles in the processed zone. In addition, the fluctuation in grain size in the processed zone (i) FSP helps refine the grains and raises the dislocation density of the grains, which prevents further grain growth, (ii) The fragmented reinforcing particles fill the space between the nucleated grains and prevent grain expansion brought on by dynamic recrystallization, (iii) When contrasted to soft aluminum, hard ceramic particles always deform plastically differently. The evidence above demonstrates that aluminum grains have undergone more

refining than ceramic particles [15, 16]. As a result, the treated zone's grain size for surface composites is constantly variable.

Figure 5 depicts a typical optical micrograph of FSP Al6061 base alloy and Al/TiC composites with varying proportions of reinforcement content. Figure 5(a) depicts the surface of the base alloy, without any reinforcements, Figure 5 (b), (c), and (d) represents the surface of the composites with 1, 3, and 5 wt. % of TiC content. A fine microstructure was obtained for all composites. Since grain refinement is accomplished in a single stage, the FSP process helps to refine the microstructure of composites to the ultrafine grain size. Distinct alignment of grains was reflected.

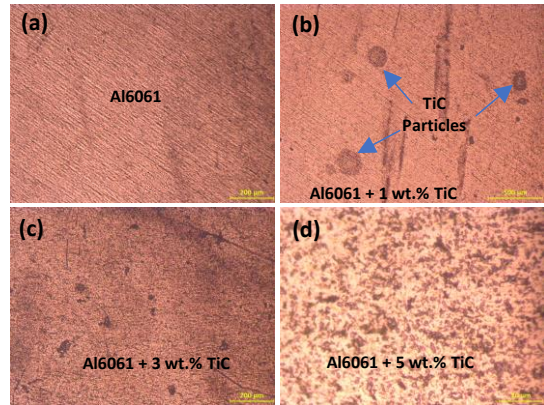


Figure 5. Optical micrograph of (a) base Al6061 alloy, (b) Composites with 1 wt.% TiC, (c) Composites 3 wt.% TiC, and (d) Composites 5 wt.% TiC

3.2 Microhardness evaluation

The microhardness of the distinct surface composites is depicted in Figure 6. In comparison to the base Al6061 microhardness, the addition of reinforcing particles has dramatically increased the microhardness in the treated zone. The matrix aluminum has a microhardness of 76.5 HV; after considerable recrystallisation, the microhardness in the processed zone was improved to 84.6 HV, 102 HV and 98.7 HV in case of C1 (Al6061+1 wt.% TiC), C2 (Al6061+3 wt. % TiC) and C3 (Al6061+5 wt.% TiC) surface composites respectively. Particles and grain refinement at the treated zone influence the higher microhardness. Amongst the three produced composites, surface composites with 3wt.% revealed the highest microhardness as compared to 1 wt.%, and 5wt.% surface composites.

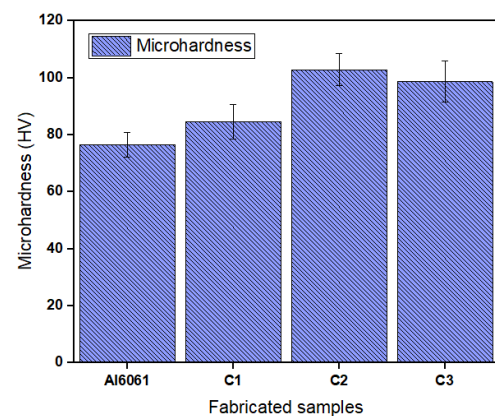


Figure 6. Microhardness of the surface composites

It has been observed that the existence of TiC particles has improved the grain structure of the aluminum matrix. As a result, a substantial improvement in the microhardness characteristics of the aluminum composites was observed. The results are in line with the previous work [17-19].

Some of the mechanisms that control the microhardness of the Al6061 include the solid solution of the matrix, strengthening of the grain boundary, and the second phase strengthening offered by the β -phase. Greater Hall-Petch effect as a result of grain refinement; grain size decrease enhanced the strengthening of the grain boundary [4, 20, 21]. Grain refining may greatly increase the alloy's hardness since Al6061 alloys have a high grain boundary pinning factor and a significant Hall-Petch coefficient.

4. CONCLUSIONS

In the present study, Al6061 matrix composites reinforced with (1, 3 and 5 wt.%) TiC particles were successfully produced by FSP without any macro-level defect. The microstructural evolution and microhardness characteristics of FSPed samples were studied, and the strengthening mechanisms were found and quantified. The microhardness behavior was improved significantly (up to 35%) as compared with base Al6061 alloy. The fine equiaxed grain structures and activated grain size strengthening were produced as a result of the reinforcement particles' dispersion, which restrained the grain boundaries' expansions during recrystallization. The main conclusions are summarized below. The SEM and OM micrographs of surface morphology suggests that the consistent particles distribution takes place in the surface composites. Among the three surface composite compositions 3 wt.% TiC exhibits highest microhardness as compared to other samples.

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REFERENCES

[1] Shozib, I.A., Ahmad, A., Rahaman, M.S.A., majdi Abdul-Rani, A., Alam, M.A., Beheshti, M., Taufiqurrahman, I. (2021). Modelling and optimization of microhardness of electroless Ni-P-TiO₂ composite coating based on machine learning approaches and RSM. *Journal of Materials Research and Technology*, 12: 1010-1025. <https://doi.org/10.1016/j.jmrt.2021.03.063>

[2] Alam, M.A., Hamdan, H.Y., Azeem, M., Hussain, P.B., bin Salit, M.S., Khan, R., Arif, S., Ansari, A.H. (2020). Modelling and optimisation of hardness behaviour of sintered Al/SiC composites using RSM and ANN: A comparative study. *Journal of Materials Research and*

Technology, 9(6): 14036-14050. <https://doi.org/10.1016/j.jmrt.2020.09.087>

[3] Alam, M.A., Sapuan, S.M., Ya, H.H., Hussain, P.B., Azeem, M., Ilyas, R.A. (2021). Application of biocomposites in automotive components: A review. *Biocomposite and Synthetic Composites for Automotive Applications*, 1-17. <https://doi.org/10.1016/b978-0-12-820559-4.00001-8>

[4] Alam, M.A., Ya, H.H., Azeem, M., Yusuf, M., Sapuan, S.M., Masood, F. (2021). Investigating the effect of mixing time on the crystallite size and lattice strain of the AA7075/TiC composites. *Materialwissenschaft und Werkstofftechnik*, 52(10): 1112-1120. <https://doi.org/10.1002/mawe.202000324>

[5] Roy, P., Singh, S., Pal, K. (2019). Enhancement of mechanical and tribological properties of SiC-and CB-reinforced aluminium 7075 hybrid composites through friction stir processing. *Advanced Composite Materials*, 28(sup1): 1-18. <https://doi.org/10.1080/09243046.2017.1405596>

[6] Raja, S., Muhamad, M.R., Jamaludin, M.F., Yusof, F. (2020). A review on nanomaterials reinforcement in friction stir welding. *Journal of Materials Research and Technology*, 9(6): 16459-16487. <https://doi.org/10.1016/j.jmrt.2020.11.072>

[7] DebRoy, T., Wei, H.L., Zuback, J.S., Mukherjee, T., Elmer, J.W., Milewski, J.O., Beese, A.M., Wilson-Heid, A., De, A., Zhang, W. (2018). Additive manufacturing of metallic components—process, structure and properties. *Progress in Materials Science*, 92: 112-224. <https://doi.org/10.1016/j.pmatsci.2017.10.001>

[8] Mishra, R.S., Ma, Z.Y., Charit, I. (2003). Friction stir processing: A novel technique for fabrication of surface composite. *Materials Science and Engineering: A*, 341(1-2): 307-310. [https://doi.org/10.1016/S0921-5093\(02\)00199-5](https://doi.org/10.1016/S0921-5093(02)00199-5)

[9] Rana, H.G., Badheka, V.J., Kumar, A. (2016). Fabrication of Al7075/B4C surface composite by novel friction stir processing (FSP) and investigation on wear properties. *Procedia Technology*, 23: 519-528. <https://doi.org/10.1016/J.PROTCY.2016.03.058>

[10] Dolatkah, A., Golbabaie, P., Givi, M.B., Molaiekiya, F. (2012). Investigating effects of process parameters on microstructural and mechanical properties of Al5052/SiC metal matrix composite fabricated via friction stir processing. *Materials & Design*, 37: 458-464. <https://doi.org/10.1016/J.MATDES.2011.09.035>

[11] Rajeshkumar, R., Udhayabanu, V., Srinivasan, A., Ravi, K.R. (2017). Microstructural evolution in ultrafine grained Al-Graphite composite synthesized via combined use of ultrasonic treatment and friction stir processing. *Journal of Alloys and Compounds*, 726: 358-366. <https://doi.org/10.1016/j.jallcom.2017.07.280>

[12] Orłowska, M., Pixner, F., Hütter, A., Enzinger, N., Olejnik, L., Lewandowska, M. (2022). Manufacturing of coarse and ultrafine-grained aluminum matrix composites reinforced with Al₂O₃ nanoparticles via friction stir processing. *Journal of Manufacturing Processes*, 80: 359-373. <https://doi.org/10.1016/J.JMAPRO.2022.06.011>

[13] Natrayan, L., Kumar, M.S. (2020). An integrated artificial neural network and Taguchi approach to optimize the squeeze cast process parameters of AA6061/Al₂O₃/SiC/Gr hybrid composites prepared by

- novel encapsulation feeding technique. *Materials Today Communications*, 25: 101586. <https://doi.org/10.1016/j.mtcomm.2020.101586>
- [14] Seifiyan, H., Sohi, M.H., Ansari, M., Ahmadkhaniha, D., Saremi, M. (2019). Influence of friction stir processing conditions on corrosion behavior of AZ31B magnesium alloy. *Journal of Magnesium and Alloys*, 7(4): 605-616. <https://doi.org/https://doi.org/10.1016/j.jma.2019.11.004>
- [15] Alam, M.A., Ya, H.H., Ahmad, A., Yusuf, M., Azeem, M., Masood, F. (2022). Influence of aluminum addition on the mechanical properties of brass/Al composites fabricated by stir casting. *Materials Today: Proceedings*, 48: 811-814. <https://doi.org/10.1016/j.matpr.2021.02.321>
- [16] Wu, B., Ibrahim, M.Z., Raja, S., Yusof, F., Abdul Razak B. b., Bin Muhamad M.R., Huang, R., Zhang, Y., Badruddin, I.A., Hussien, M., Kamangar, S. (2022). The influence of reinforcement particles friction stir processing on microstructure, mechanical properties, tribological and corrosion behaviors: A review. *Journal of Materials Research and Technology*, 20: 1940-1975. <https://doi.org/https://doi.org/10.1016/j.jmrt.2022.07.172>
- [17] AnandhaKumar, C.J., Gopi, S., Kumar, S.S., Mohan, D.G. (2021). Mechanical, metallurgical and tribological properties of friction stir processed aluminium alloy 6061 hybrid surface composites. *Surface Topography: Metrology and Properties*, 9(4): 045019. <https://doi.org/10.1088/2051-672X/ac3120>
- [18] Bhasha, A.C., Balamurugan, K. (2021). Studies on mechanical properties of Al6061/RHC/TiC hybrid composite. *International Journal of Lightweight Materials and Manufacture*, 4(4): 405-415. <https://doi.org/10.1016/j.ijlmm.2021.06.003>
- [19] Reddy, P.V., Prasad, P.R., Krishnudu, D.M., Goud, E.V. (2019). An investigation on mechanical and wear characteristics of Al 6063/TiC metal matrix composites using RSM. *Journal of Bio-and Tribo-Corrosion*, 5(4): 90. <https://doi.org/10.1007/s40735-019-0282-0>
- [20] Alam, M.A., Ya, H.H., Hussain, P.B., Azeem, M., Sapuan, S.M., Ahamad, T. Experimental investigations on the surface hardness of synthesized polystyrene/ZnO nanocomposites. *Advances in Manufacturing Engineering. Lecture Notes in Mechanical Engineering*. https://doi.org/10.1007/978-981-15-5753-8_32
- [21] Alam, M.A., Ya, H.H., Azeem, M., Yusuf, M., Soomro, I.A., Masood, F., Shozib, I.A., Sapuan, S.M., Akhter, J. (2022). Artificial neural network modeling to predict the effect of milling time and TiC content on the crystallite size and lattice strain of Al7075-TiC composites fabricated by powder metallurgy. *Crystals*, 12(3): 372. <https://doi.org/10.3390/cryst12030372>