

## Investigating the Effect of Tool Dimension and Rotational Speed on Microstructure of Al-B<sub>4</sub>C Surface Composite Layer Produced by Friction Stir Processing (FSP)

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### ABSTRACT

Friction stir processing (FSP) was used for the fabrication of Al-B<sub>4</sub>C surface composite. Al-Mg-Si alloy was considered as the substrate and B<sub>4</sub>C particles were incorporated into the substrate by thermo-mechanical effect of FSP. The effect of tool dimensions and different rotational speeds on the microstructure and microhardness of the composite layers was evaluated and the optimum process parameters were determined. Microstructural evaluation of the samples after FSP was conducted by optical microscopy (OM) and scanning electron microscopy (SEM) of the cross-sections of surface composite layers fabricated by FSP. Hardness profiles were obtained from microhardness measurements across the cross-sections of the FSPed samples. The results showed that by increasing the tool size and rotational speed, the size of nugget zone increases and the volume fraction of reinforcing particles decreases in the FSPed samples. Moreover, composite layers containing higher volume fractions of B<sub>4</sub>C particles obtained from smaller tool size exhibited higher values of hardness.

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### 1. Introduction

Metal matrix composites (MMCs) have attained a wide range of applications in ground transportation, aerospace, and industrial structures due to their interesting properties such as higher strength, stiffness, and wear resistance in comparison to the preliminary matrix. Many processing methods have been developed for the production of metal matrix composites. These methods may simply be classified as solid state, liquid state, gas state and in situ methods. Among these categories

stir casting, liquid metal infiltration and powder metallurgy have been developed to fabricate metal matrix composites. However, these processes are expensive and agglomeration of the ceramic particles results in the reduction of ductility and thermal conductivity of the material. Insufficient stirring of the material during these processes and the difference between the density of substrate and ceramic particles in liquid state processes cause the particles to gather in specific places and create agglomerates. Stirring of the material during

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Solidification or at solid state may reduce the effect of density difference and inhibit the agglomeration of particles [1-3]. Recently, friction stir processing (FSP) has been developed as a solid state process based on friction stir welding (FSW) to produce surface metal matrix composites [4-6]. In this process, a non-consumable spinning tool is inserted into the matrix and the friction between tool and matrix generates heat, increases the temperature, and softens the material. Most of the heat generation occurs at the interface between the tool shoulder and the work-piece. The rotating tool travels along the long axis of the work piece and this combination of rotation and travelling leads to the movement of material from the front to the back of the pin. Achieving a defect free composite layer with appropriate mechanical properties depends on process parameters [7, 8].

It has been reported that the addition of  $\text{Al}_2\text{O}_3$  and SiC particles to Al matrix by FSP can increase the hardness of the material up to 30% [3]. Although, when FSP is applied without the addition of reinforcing powder, the mean value of hardness decreases and this behavior is attributed to the annealing effect of the process [3]. It has been reported that increasing the number of passes may result in a better distribution of particles and enhance the microhardness of the composite layer [9]. Moreover, incorporating ceramic particles has a brilliant effect on grain size reduction compared to the unreinforced matrix [10]. According to previous studies, decreasing the particle size may result in considerable reduction of the matrix grain size. Also, the average hardness of composite has been reported to increase by increasing the volume fraction of particles [11, 12].

Among conventional ceramic particles boron carbide can be considered as a suitable candidate to produce surface metal matrix composites.  $\text{B}_4\text{C}$  possesses attractive physical and mechanical properties that are comparable to or better than SiC and  $\text{Al}_2\text{O}_3$ . It has a high hardness ( $2800 \text{ kg/mm}^2$ ) with a low theoretical density ( $2.54 \text{ gr/cm}^3$ ), high melting point ( $2450^\circ\text{C}$ ), and good resistance to chemical agents [13, 14]. Recently, applying  $\text{B}_4\text{C}$  particles on AA5083 by FSP has been reported

to improve the tensile strength and wear behavior. Also, decreasing the size of  $\text{B}_4\text{C}$  particles resulted in the enhancement of mechanical and wear properties [15].

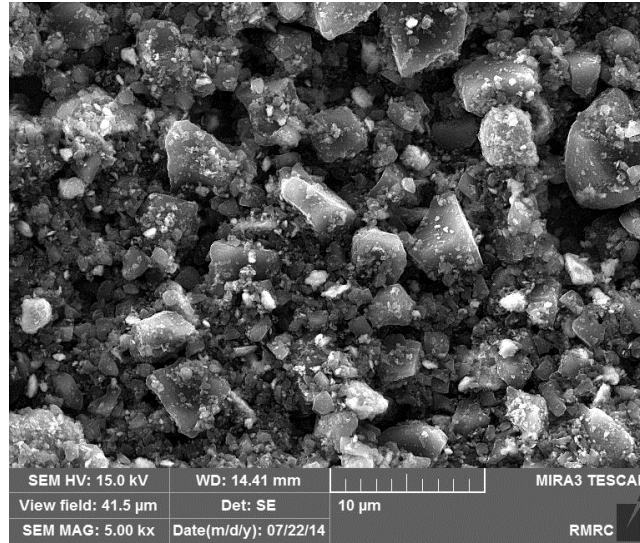
The aim of this study was to produce surface composite layer of Al- $\text{B}_4\text{C}$  by FSP and evaluate the effect of tool size and rotational speed on microstructure and wear resistant of the obtained composite.

## 2. Materials and methods

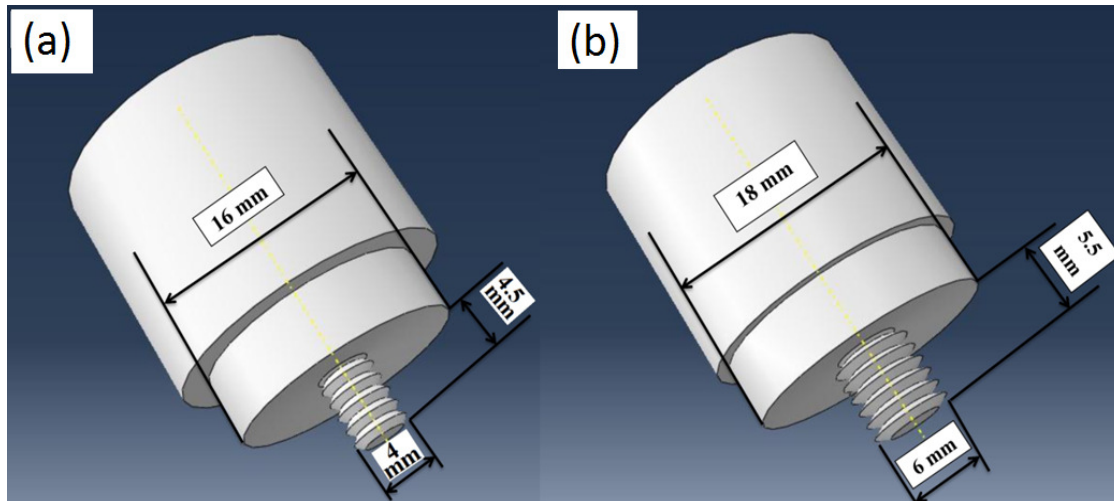
Aluminum alloy with the chemical composition of 6XXX series as presented in Table1 and thickness of 10 mm was used as the substrate. Milled  $\text{B}_4\text{C}$  powder with a size range of submicron to 7 microns (Fig. 1) was compacted into a groove with the width and depth of 1mm and 4.5 mm, respectively. Conventional milling machine with the nominal maximum rotational speed of 1600 rpm was used to perform FSP. A thin layer of the substrate was created on the groove surface due to the heat generated between the surface of rotating pinless tool and the substrate. Two cylindrical threaded pins of different dimensions were used to create the surface composite layer (Fig. 2). The FSP tools were made of H13 hot worked tool steel that have been oil hardened after the tools were made. The tilt angle of the tool was  $2^\circ$  with respect to the vertical axis. Traverse speed of 40 mm/min with rotational speeds of 630, 800, and 1000 rpm were used for each tool. The process was performed in three passes in opposite directions in order to exchange the retreat side and advance side in each pass. Table 2 presents the details of the process parameters used to produce surface composite layers by FSP. Microstructural evaluation was conducted by Meiji Techno IM7200 optical microscopy and LEO 11455 VP scanning electron microscopy (SEM) on the cross sections of composite layers. MIP4STUDENT software was used for analyzing the macro and microstructure of composite layers. Hardness profiles were obtained across the cross sections of the composite layers using a Nexus Innova 4300 microhardness testing machine. Vickers microhardness values were determined along a 2 mm line beyond the surface under 200 gr load at 10 sec dwell time.

**Table 1.** Chemical composition of the as-received aluminum alloy (wt. %)

Si	Fe	Cu	Mn	Mg	Cr	Zn	B	Al
0.57	0.43	0.1	0.3	0.84	0.12	0.05	0.06	Bal.



**Fig. 1.** Morphology of boron carbide powder



**Fig. 2.** Schematic illustration of tools with different dimensions used for friction stir processing: (a) small tool and (b) big tool

### 3. Results and discussion

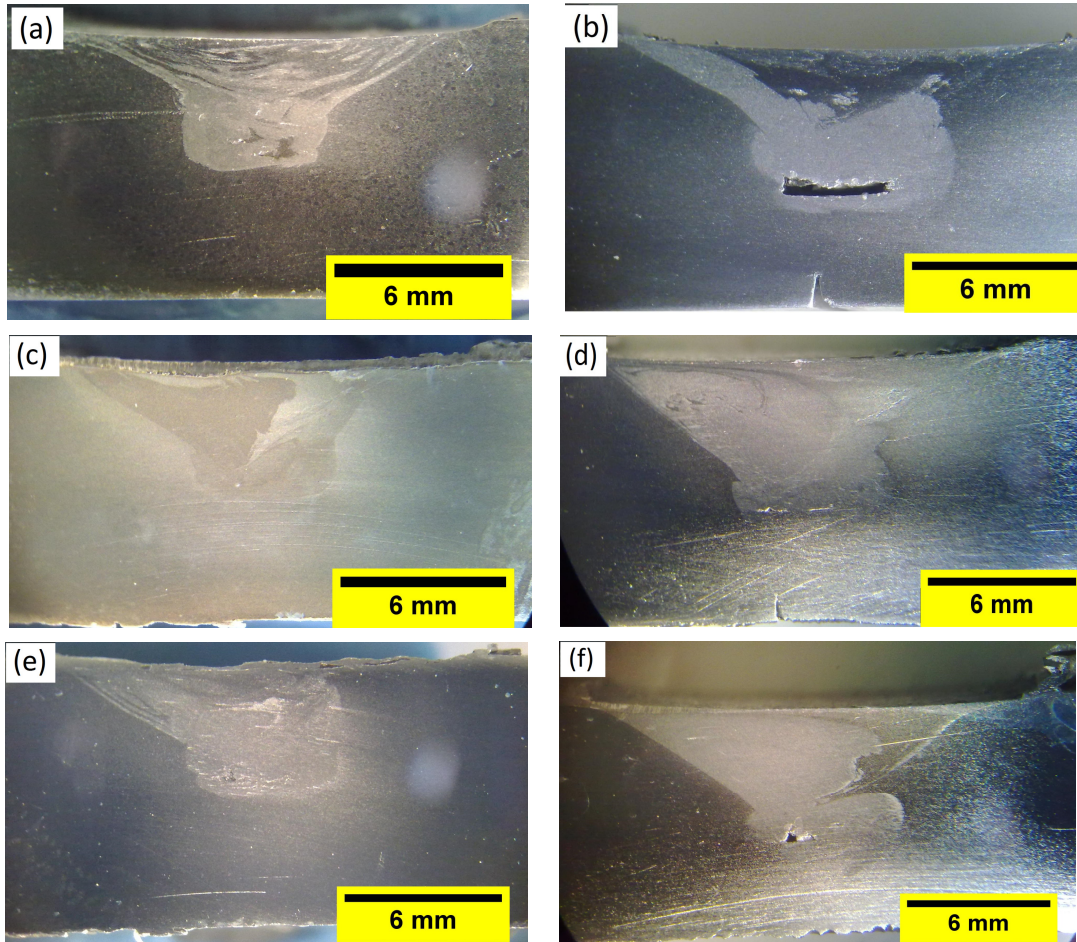
#### 3. 1. Microstructure

Fig. 3 shows the cross sectional macrostructures of surface composite layers produced by friction stir processing with two different tools at 630, 800 and 1000 rpm rotation speeds. Macrostructures obtained from

both pins at 630 rpm indicated ununiform distribution of the particles in the nugget and wormhole defect was observed in the stir zone of both samples (Fig. 3(a) and (b)). This defect can be attributed to the poor deformation of the material due to low heat input. Frigaard et al. [16] proved that the heat input during friction

**Table 2.** Process parameters used for the fabrication of surface composite layers by FSP

Code	Rotation Speed	Tool Name
S <sub>630a</sub>	630	a
S <sub>630b</sub>	630	b
S <sub>800a</sub>	800	a
S <sub>800b</sub>	800	b
S <sub>1000a</sub>	1000	a
S <sub>1000b</sub>	1000	b

**Fig. 3.** Cross sectional macrostructures of: (a) S<sub>630a</sub>; (b) S<sub>630b</sub>; (c) S<sub>800a</sub>; (d) S<sub>800b</sub>; (e) S<sub>1000a</sub> and (f) S<sub>1000b</sub>

Stir welding depends on process parameters. Generated heat input during the process has been shown to impress the consequent structure and the creation of defects [17, 18]. Macrostructure examination of the nugget obtained from 800 rpm rotational speed indicated that the reinforcing particles were

distributed in a wider region (Fig. 3(c) and (d)). No obvious defect was observed in the nugget, which can be attributed to the sufficient stirring of material during the process. FSP at 1000 rpm rotational speed resulted in the formation of a hole in the nugget for both pins (Fig. 3(e) and (f)). Different temperatures of the upper part

near the surface and the lower part due to the high ratio of rotational speed to travel speed led to ununiformed stirring of the material [18].

Many studies reported that tool geometry is an influential parameter on material flow in the nugget and on the shape of the nugget [19, 20]. It seems that tool dimension does not affect the nugget shape, and the nuggets created by both pins were found to be similar in shape especially at higher rotational speeds.

The size of nugget is affected by the tool size and it is even comparable with the pin size. This phenomenon has been reported by several researchers [21-23].

Fig.4 shows the SEM micrographs of the Al-B<sub>4</sub>C composite produced by two different tools at 630, 800 and 1000 rpm rotational speeds. According to the EDS analysis, the black areas indicate the B<sub>4</sub>C particles and the white contrast areas are the precipitates in the matrix (Fig. 4(a)). At the rotational speed of 630 rpm, some regions in the matrix remained without the presence of particles. On the other hand, in the regions containing reinforcing particles, agglomeration could be observed. Increasing the rotational speed to 800 and 1000 rpm resulted in better distribution of the particles in the nugget and decreased the mean distance between the particles (Fig. 4(c), (d), (e) and (f)). Appropriate material flow as a result of sufficient heat input during the process can be considered as the main reason of this feature.

Comparison of Fig. 3 and Fig. 4 showed that the volume fraction of particles in the nugget is affected by the size of nugget. Fig. 5 shows the computational results obtained from particle volume fraction determination with MIP4STUDENT software. Large sizes of nugget caused the volume fraction of B<sub>4</sub>C particles to decrease. Since the variation of the process parameters can change the stirred area, in a constant groove size, the particles volume fraction in the nugget is affected by various variables [24]. Tool size and rotational speed have considerable effects on the size of nugget. Increasing the tool size and rotational speed causes the size of the nugget to increase and the volume fraction of particles in the nugget to decrease. Very low rotational speeds can result in limited stirred area with high volume

fraction of the particles.

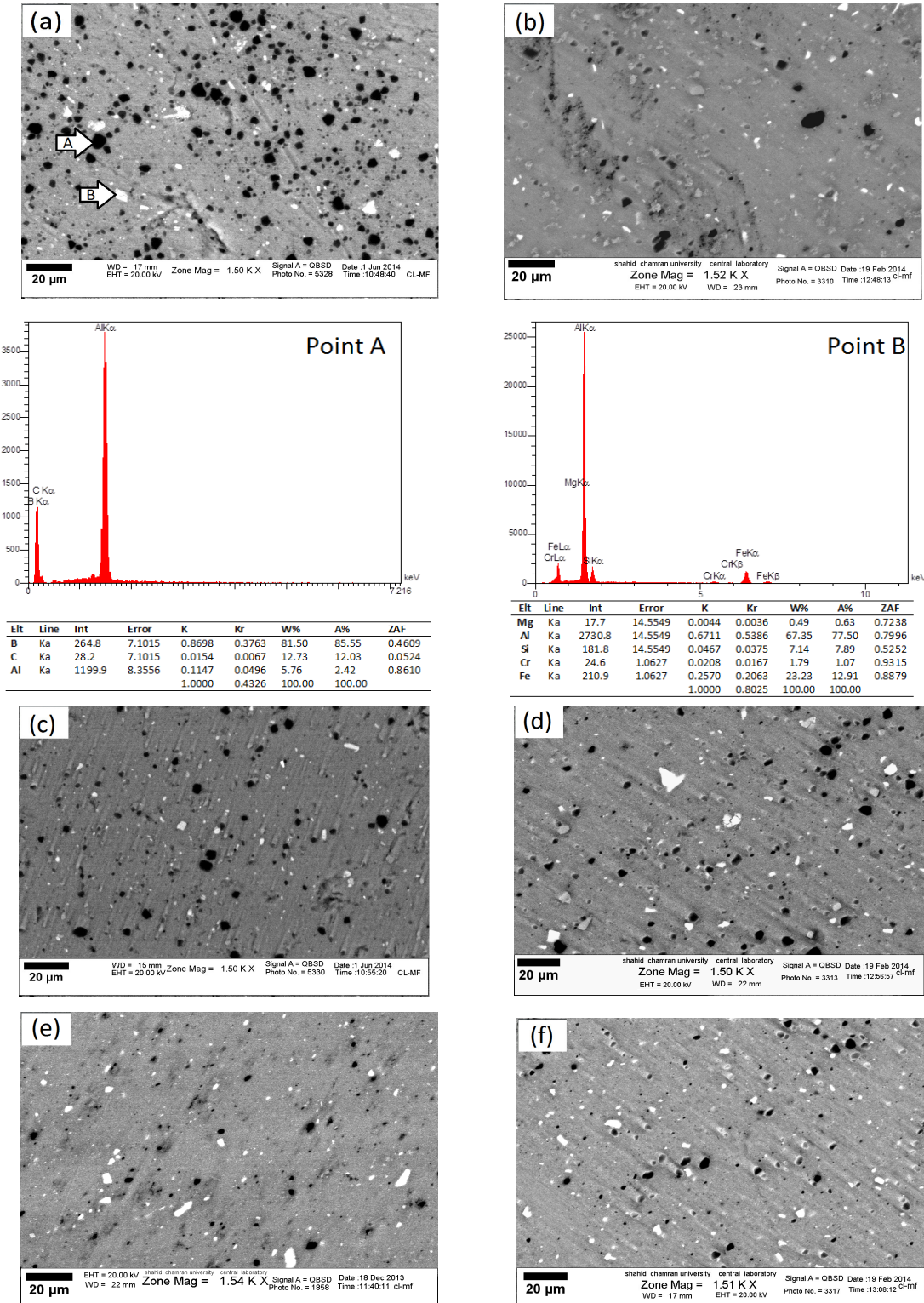
Thermomechanical stresses created by severe plastic deformation during the process may cause the precipitates to break in the matrix to finer sizes and better distribution. Meanwhile, movement of the hard B<sub>4</sub>C reinforcing particles with sharp edges could have assisted the fracture of precipitates during the process. It is obvious from Fig. 4 that at lower particle volume fractions the size of precipitates is larger.

### 3. 2. Microhardness

Microhardness profiles of the specimens produced at different rotation speeds with both tools are presented in Fig. 6. Microhardness of composite layers noticeably increased due to the presence of hard B<sub>4</sub>C ceramic particles and thermomechanical effect of FSP that distributes the reinforcing particles in the matrix. Moreover, the composite layers, which have been produced by smaller tool, possess higher hardness values due to the higher volume fractions of B<sub>4</sub>C. The microhardness of the obtained composites by FSP is affected by the annealing effect of heat input, dynamic recrystallization, natural hardness and volume fraction of reinforcing particles. As the volume fraction of the particles increases, the density of dislocations in the matrix increases. This results in higher interactions between particles and dislocations which further increases the microhardness [25].

As a result, known strengthening mechanisms would contribute to the enhancement of hardness in the composite layer [26]. Strengthening mechanisms can be mainly considered as Orowan strengthening due to the dispersion of fine ceramic particles, grain and substructure strengthening and, quench hardening resulted from the generation of dislocations to accommodate the differential thermal contraction between the reinforcing particles and the matrix.

However, the measured hardness values in each composite showed disparity. This is due to the different volume fractions of reinforcing particles in some regions of the matrix. By increasing the rotational speed, the disparity of hardness measurements decreased. Also, the hardness of the composite layer decreased as a



**Fig. 4.** SEM micrographs of the cross section of Al-B<sub>4</sub>C composite layers produced by FSP: (a) S<sub>630a</sub>; (b) S<sub>630b</sub>; (c) S<sub>800a</sub>; (d) S<sub>800b</sub>; (e) S<sub>1000a</sub> and (f) S<sub>1000b</sub>

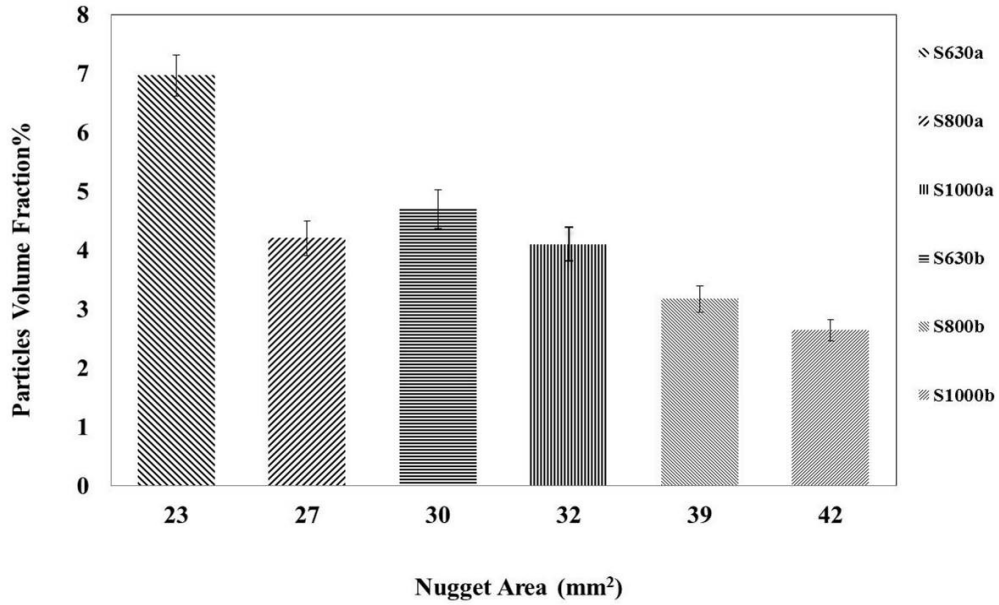


Fig. 5. Variations of particle volume fractions with respect to the nugget area in the Al-B<sub>4</sub>C composite layers with different tool dimensions and rotational speeds

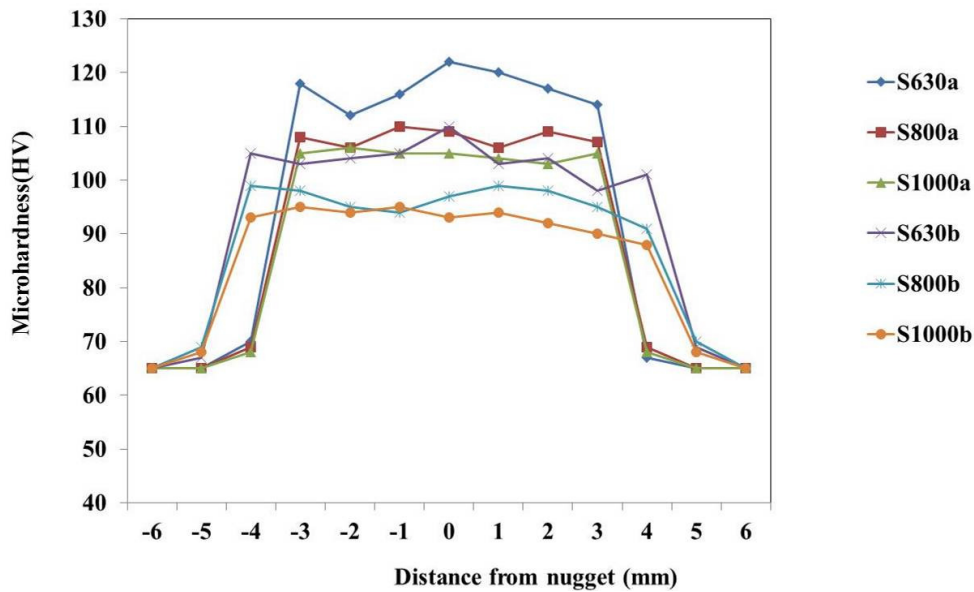


Fig. 6. Microhardness profiles of the composite layers produced by FSP with different tool dimensions and rotational speeds

Result of increasing the rotational speed, which can be attributed to higher heat input. By increasing the rotational speed, the heat input increased and the cooling rate decreased that could result in larger final grain size after FSP.

### 3. Conclusions

Al-B<sub>4</sub>C surface composite layer was produced successfully by FSP. Rotational speed and tool size showed significant effect on the nugget size, so that by increasing each of these parameters the nugget size increased.

Increasing the nugget size showed a reverse effect on the volume fraction of the B<sub>4</sub>C reinforcing particles within the nugget. Composite layers produced by 800 rpm rotational speed showed better characteristics for two different pin sizes. Sufficient stirring of the material during the process with no defect resulted in a relatively uniform distribution of the B<sub>4</sub>C particles in aluminum matrix. Volume fraction of the B<sub>4</sub>C particles affected the precipitate distribution in the matrix during FSP. It was proposed that increasing the volume fraction of the reinforcing particles resulted in the breaking of precipitates which were distributed in the matrix by the natural behavior of the process. Decreasing the rotational speed to 630 rpm, shoulder diameter to 16 mm and pin diameter to 4 mm resulted in the improvement of the hardness of the composite layer. The main reasons were considered to be the increase in the particles volume fraction and decrease in the heat input.

## References

1. D. B. Miracle, "Metal matrix composites – From science to technological significance", *Compos Sci Technol*, Vol. 65, 2005, pp. 2526–2540.
2. Cantor, B., Dunne, F. & Stone I, *Metal and Ceramic Matrix Composites*, Institute of Physics Publishing, IOP Publishing Ltd. (2004) First edition.
3. J. Qua, H. Xua, Z. Fenga, D. Alan Frederick, L. An, H. Heinrich, "Improving the tribological characteristics of aluminum 6061 alloy by surface compositing with sub-micro-size ceramic particles via friction stir processing", *Wear*, Vol. 271, 2011, pp. 1940–1945.
4. D. K. Lim, T. Shibayanagi, A. P. Gerlich, "Synthesis of multi-walled CNT reinforced aluminium alloy composite via friction stir processing", *Materials Science and Engineering A*, Vol. 507, 2009, pp. 194–199.
5. M. Zohoor, M. K. Besharati Givi, P. Salami, "Effect of processing parameters on fabrication of Al–Mg/Cu composites via friction stir processing", *Materials and Design*, Vol. 39, 2012, pp. 358–365.
6. Yong X. Gan, Structural assessment of nanocomposites, *Micron*, Vol. 43, 2012, pp. 782–817.
7. R. S. Mishra, & M. W. Mahoney, *Friction Stir Welding and Processing*, ASM International, (2007) First edition.
8. R. S. Mishra, Z. Y. Ma, I. Charit, "Friction stir processing: a novel technique for fabrication of surface composite", *Mater Sci Eng A*, Vol. 341, 2003, pp. 307–310.
9. A. Shafiei-Zarghani, S. F. Kashani-Bozorg, A. Zarei- Hanzaki, "Wear assessment of Al–Al<sub>2</sub>O<sub>3</sub> nano-composite surface layer produced using friction stir processing", *Wear*, Vol. 270, 2011, pp.403–412.
10. A. Dolatkah, P. Golbabaie, M. K. Besharati Givi, F. Molaiekiya. "Investigating effects of process parameters on microstructural and mechanical properties of Al5052/SiC metal matrix composite fabricated via friction stir processing.", *Materials and Design*, Vol. 37, 2012, pp.458–464.
11. M. Barmouz, P. Asadi, M. K. Besharati Givi, M. Taherishargh, "Investigation of mechanical properties of Cu/SiC composite fabricated by FSP: Effect of SiC particles' size and volume fraction", *Materials Science and Engineering A*, Vol.528, 2011, pp. 1740–1749.
12. M. Barmouz, M. K. Besharati Givi, J. Seyfi, "On the role of processing parameters in producing Cu/SiC metal matrix composites via friction stir processing: Investigating microstructure, microhardness, wear and tensile behavior", *Mater Charact*, Vol. 62, 2011, pp. 108–117.
13. M. Khakbiz, F. Akhlaghi, "Synthesis and structural characterization of Al–B<sub>4</sub>C nano-composite powders by mechanical alloying", *J Alloy Compd*, Vol. 479, 2009, pp. 334–341.
14. T. Ya. Kosolapova, *Carbides: properties, production and applications*, (1971) plenum press First edition.
15. N. Yuvaraj, S. Aravindan, Vipin, "Fabrication of Al5083/B<sub>4</sub>C surface composite by friction stir processing and its tribological characterization" *J Mater Res Tech*, In press, doi:10.1016/j.jmrt. 2015.02.006
16. F. Frigaard, F. Grong, O. T. Midling, "A Process Model for Friction Stir Welding of Age Hardening Aluminum Alloys", *Metall Mater Trans A*, Vol. 32(5), 2001, pp. 1189–1200.
17. Y. G. Kim, H. Fujii, T. Tsumura, T. Komazaki, K. Nakata, "Three defect types in friction stir welding of aluminum die casting alloy", *Mater Sci Eng A*, Vol. 415, 2006, pp. 250–254.
18. R. Nandan, T. DebRoy, H.K. Bhadeshia, "Recent advances in friction-stir welding-Process, weldment structure and properties",



- Prog Mater Sci, Vol. 53, 2008, pp. 980-1023.
19. K. Elangovan, V. Balasubramanian, "Influences of tool pin profile and tool shoulder diameter on the formation of friction stir processing zone in AA6061 aluminium alloy", *Mater Design*, Vol. 29, 2008, pp. 362-373.
  20. E. Salari, M. Jahazi, A. Khodabandeh, H. Ghasemi-Nanesa, "Influence of tool geometry and rotational speed on mechanical properties and defect formation in friction stir lap welded 5456 aluminum alloy sheets", *Mater Design*, Vol. 58, 2014, pp. 381-389.
  21. M. Sharifitabar, A. Sarani, S. Khorshahian, M. Shafiee Afarani, Fabrication of 5052Al/Al<sub>2</sub>O<sub>3</sub> nanoceramic particle reinforced composite via friction stir processing route, *Mater Design*, Vol. 32, 2011, pp. 4164-4172.
  22. A. Devaraju, A. Kumar, B. Kotiveerachari, "Influence of addition of Gr/Al<sub>2</sub>O<sub>3</sub> with SiC on wear properties of aluminum alloy 6061-T6 hybrid composites via friction stir processing", *T Nonferr Metal Soc*, Vol.23, 2013, pp.1275-1280.
  23. A. Devaraju, A. Kumar, A. Kumaraswamy, B. Kotiveerachari, "Wear and mechanical properties of 6061-T6 aluminum alloy surface hybrid composites [(SiC + Gr) and (SiC + Al<sub>2</sub>O<sub>3</sub>)]fabricated by friction stir processing", *J Mater Res Technol*, Vol. 2(4), 2013, pp. 362-369.
  24. R. Sathiskumar, N. Murugan, I. Dinaharan, S. J. Vijay, "Effect of Traverse Speed on Microstructure and Microhardness of Cu/B<sub>4</sub>C Surface Composite Produced by Friction Stir Processing", *T Indian Inst Metall*, Vol. 66(4), 2013, pp. 333-337.
  25. S. Shahraki, S. Khorasani, R. A. Behnagh, Y. Fotouhi, H. Bisadi, "Producing of AA5083/ZrO<sub>2</sub> Nanocomposite by Friction Stir Processing (FSP)", *Metall Mater Trans B*, Vol. 44(6), 2013, pp. 1546-1553.
  26. D. J. Lloyd, "Particle reinforced aluminum and magnesium matrix composites", *Int Mater Rev*, Vol. 39, 1994, pp. 1-24.

