

# Effect of friction stir processing on microstructure and microhardness of Al–TiC composites

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## Abstract

Friction stir processing (FSP) is a metalworking technology based on the same basic principles as friction stir welding (FSW). Using a stiff tool, FSP can be applied for the bulk or superficial processing of metallic materials. The process could be also applied for local modification of the microstructure for specific property improvement. Moreover, processed surfaces show an enhancement of mechanical properties, such as tensile strength, hardness, wear and corrosion resistance. In this paper, it has been shown that friction stir processing can be used efficiently to the TiC particles distribution in Al based samples. The groove was made in the aluminium alloy 6082 in which reinforcement particles are dispersed using a tool. The sample was subjected to single pass FSP and its effect on the microstructure and microhardness was evaluated.

*Keywords:* Friction Stir Processing (FSP); aluminium alloy 6082; titanium carbide (TiC); microstructure; microhardness

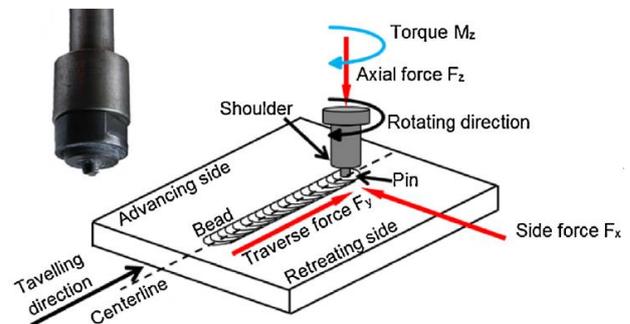
## 1. Introduction

In the past three decades the aluminium matrix composites, owing to their higher strength, stiffness, and wear resistance compared to the unreinforced Al alloys, gained demand for their use in in the automotive and aerospace industries. In general, various types of ceramic particles (like carbides, oxides, borides and nitrides) with high strength, elastic modulus and wear resistance are introduced as reinforcements to fabricate the particulate reinforced Al composites [1-4].

TiC is an attractive reinforcement for particulate composites because of its high elastic modulus and hardness. Titanium carbide is a chemical compound of titanium and carbide with the chemical formula TiC. It is the reinforcement powder mostly used in structural applications, automobile, aerospace industries and specific cutting tools etc. It has cubic crystal phase and black colour. It has very high melting point of 3200°C and the density of 4.93g/cm<sup>3</sup>. Particle reinforced metal matrix composites are likely to find many commercial applications due to their isotropy, improved properties and ease of fabrication [4-5].

For over a decade, friction stir processing (FSP) is emerging as a generic tool for material processing and microstructure modification. Friction stir processing is a novel technique developed by Mishra et al., [6]. The idea was derived to obtain microstructural modification based on the basic principles of friction stir welding (FSW). The process has been applied for: achieving ultra-fine grains and microstructural/compositional homogeneity, hardening and modification of surface, increasing plasticity, repairing defective components, the fabrication of surface and bulk level metal-matrix composites and many more [4, 7].

The FSP method applies a non-consumable rotating tool with a pin and shoulder, which is plunged at one end of the workpiece and traversed across the piece. As it travels forward, the workpiece material moves from the front to the back of the pin [8-11]. The schematic illustration of FSP is shown in Figure 1.



*Figure 1. Schematic illustration of FSP method [12].*

The friction between the rotating tool and the workpiece generates heat, and softens the material resulting in the plastic deformation and grain refinement. The temperature during FSP is significantly lower than the melting point of the materials which avoids porosity and interfacial reactions [9-10, 13].

Material flow involved in FSP is depended by the process parameters, tool geometry and material properties. In accordance with the principles of dynamic recrystallization, the increase in the degree of deformation during FSP results in a reduction of recrystallized grain size, extending the fine grain stir zone to the advancing side [11].

In this study, the effect of FSP process on the microstructure and microhardness of Al–TiC composites has been evaluated.

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## 2. Friction Stir Processing (FSP)

### 2.1. Processed zone

The microstructure that develops during FSP results from the influence of material flow, elevated temperature, plastic deformation and is characterized by a stir zone (SZ) surrounded by a thermo-mechanically affected zone (TMAZ) and heat affected zone (HAZ) [12] as presented in Figure 2.

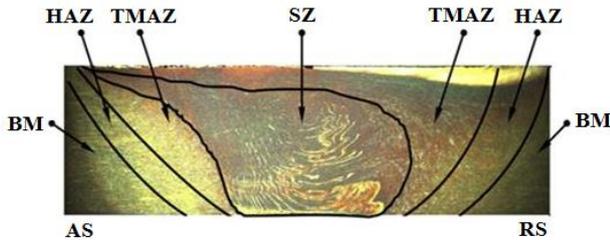


Figure 2. Zones arising in the FSP process [14].

The tool probe is lead to shear material from advancing side (AS) and transmits it to retreating side (RS). At the centre, the stir zone (SZ) is generated due to the combined effect of intensive plastic deformation caused by the tool, and the consequent heat generation. The combination of the mechanical and thermal phenomena ultimately leads to recrystallization and generation of fine equiaxed grains. A transition between refined grain and plate base material, is known as the thermo-mechanically affected zone (TMAZ). In the vicinity of the TMAZ, a heat affected zone (HAZ) can be observed where grain structure remains unchanged. The hardness of the TMAZ is universally higher than the minimum measured for the HAZ [11, 15].

### 2.2. Process parameters

FSP parameters specify the amount of temperature generation and plastic deformation, affecting the material flow around the non-consumable tool, thus determining the results obtained. It is important to know the effect of each parameter in order to have a better control over the process [11].

The principal technological parameters of the FSP process are:

- kind of the tool (shape, length and diameter of the pin, diameter and shape of the shoulder),
- penetration depth of the tool,
- travelling speed,
- rotational speed,
- alloying material,
- tilt angle,
- cooling system,
- clamping system [12].

The effect of the different process parameters has been widely documented by several authors [16-18], but it is unanimously believed that heat generation and plastic deformation are essential to establish material flow and to achieve good consolidation. Insufficient heating results in improper material consolidation and hence low ductility and strength. Increasing heat generation will

cause a greater grain refinement, improving material properties. However, a considerable increase in tool rotation rate or axial force or a very low transverse speed may result in a higher temperature than desired, slower cooling rate or excessive release of stirred material, causing degradation of properties [11].

## 3. Materials and methods

### 3.1. Materials

The 6082 aluminium alloy with the chemical composition presented in Table 1 was used as the base material (BM) in this study. Plate of 10 mm thickness was used for the tests. The micron sized TiC particles was used as reinforcement materials.

Table 1. Chemical composition (wt.%) of 6082 aluminium alloy [19].

Mg	Si	Fe	Mn	Cu	Cr	Zn	Ti	Al
0.78	1.06	0.21	0.55	0.09	0.03	0.06	0.01	Bal.

### 3.2. Composite fabrication by FSP

In order to add to the particles into the plate, firstly, a groove of 2 mm depth and 2 mm width was made along the centre line of the plate and then embed TiC particles into it. The FSP was carried out on FSW machine. In the present research, only the single pass FSP procedure was investigated.

The process parameters employed were: tool rotational speed of 560 rpm and traverse speed of 40 mm/min.

The FSP procedure to produce the composite in groove method is schematically shown in Figure 3.

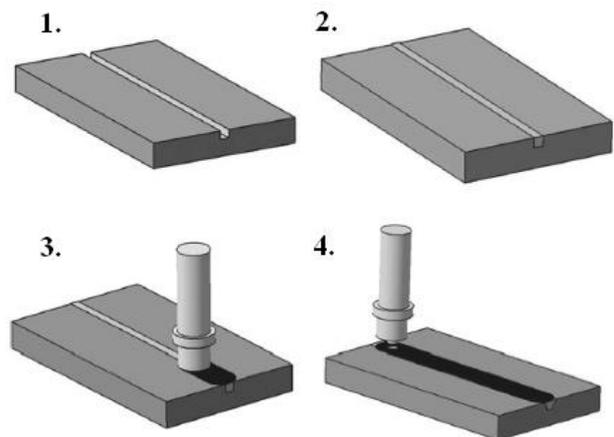


Figure 3. FSP procedure to fabricate composite in groove method [19].

In the first step, the groove is machined on the plate and the reinforcement particles are filled in the groove. The next step consists of using a pinless tool on the groove. The groove is completely packed in this step. In the last step, the tool with pin is applied on the packed groove [8].

### 3.3. Characterization

After FSP, specimen was cut at its centre perpendicular to processing direction to carry out microstructural and mechanical characterization. The polishing was prepared using an automatic grinding and polishing machine (Metkon DIGIPREP 251). The specimen was ground with emery paper about 320 grit followed by polishing with 9  $\mu\text{m}$  and 3  $\mu\text{m}$  diamond paste. The sample was finally polished finish with 50 nm colloidal silica on a velvet cloth. Then it was etched using 5% HF solution.

The image of a cross section of etched specimen was captured using a MOTIC SMZ-168 stereoscopic microscope. The microstructure was observed using a Nikon Eclipse ME600 optical microscope with digital image recording and scanning electron microscope (SEM) JEOL-JSM-820. Microanalysis of chemical composition was performed by SEM coupled with energy dispersive spectroscopy (EDS).

### 3.4. Microhardness testing

Microhardness test was carried out to assess the effect of FSP on mechanical properties of the Al-TiC composite. Vickers microhardness test across the cross section of FSPed sample was measured using a microhardness tester (Future-Tech FM-700e) at 200 g load applied for 10 sec. The mean value of the measurements was reported as the hardness of each area.

## 4. Results and discussion

### 4.1. Microstructure of Al-TiC composite

Figure 4 shows a macrostructure of friction stir zone of Al-TiC composite. The boundary of the FSP zone is marked using a continuous black line.

Based on microstructural characterization of grains and precipitates, the joint can be divided into three distinct zones, stir zone (SZ), thermo-mechanically affected zone (TMAZ), and heat affected zone (HAZ). The contribution of intense plastic deformation and high temperature exposure within the stir zone during FSP results in recrystallization and development of texture within the stirred zone. The boundary between the recrystallized stir zone and the base metal is relatively diffuse on the retreating side of the tool, but quite sharp on the advancing side of the tool. The TMAZ is characterized by a highly deformed structure. The HAZ does not undergo any plastic deformation. The microstructural changes in various zones have significant effect on post weld mechanical properties [20].

The geometry of the stir zone is symmetrical about the axis of tool rotation. Although it is widely known that single pass FSP/FSW tends to induce an asymmetric material flow, thus leading to the asymmetry of the macrostructure of the SZ [21]. The width of the resulting stir zone reduces from the top surface to the bottom surface. This change in width from top to bottom is attributed to the material flow characteristics during FSP [1, 19].

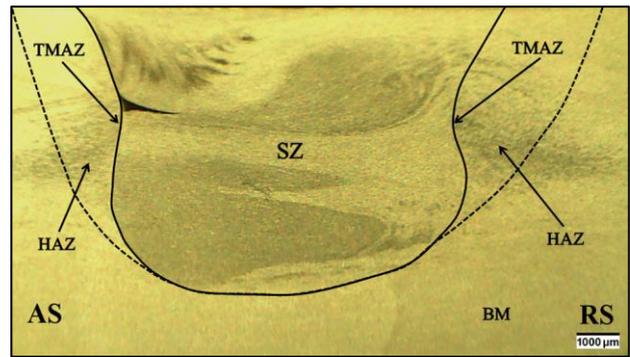


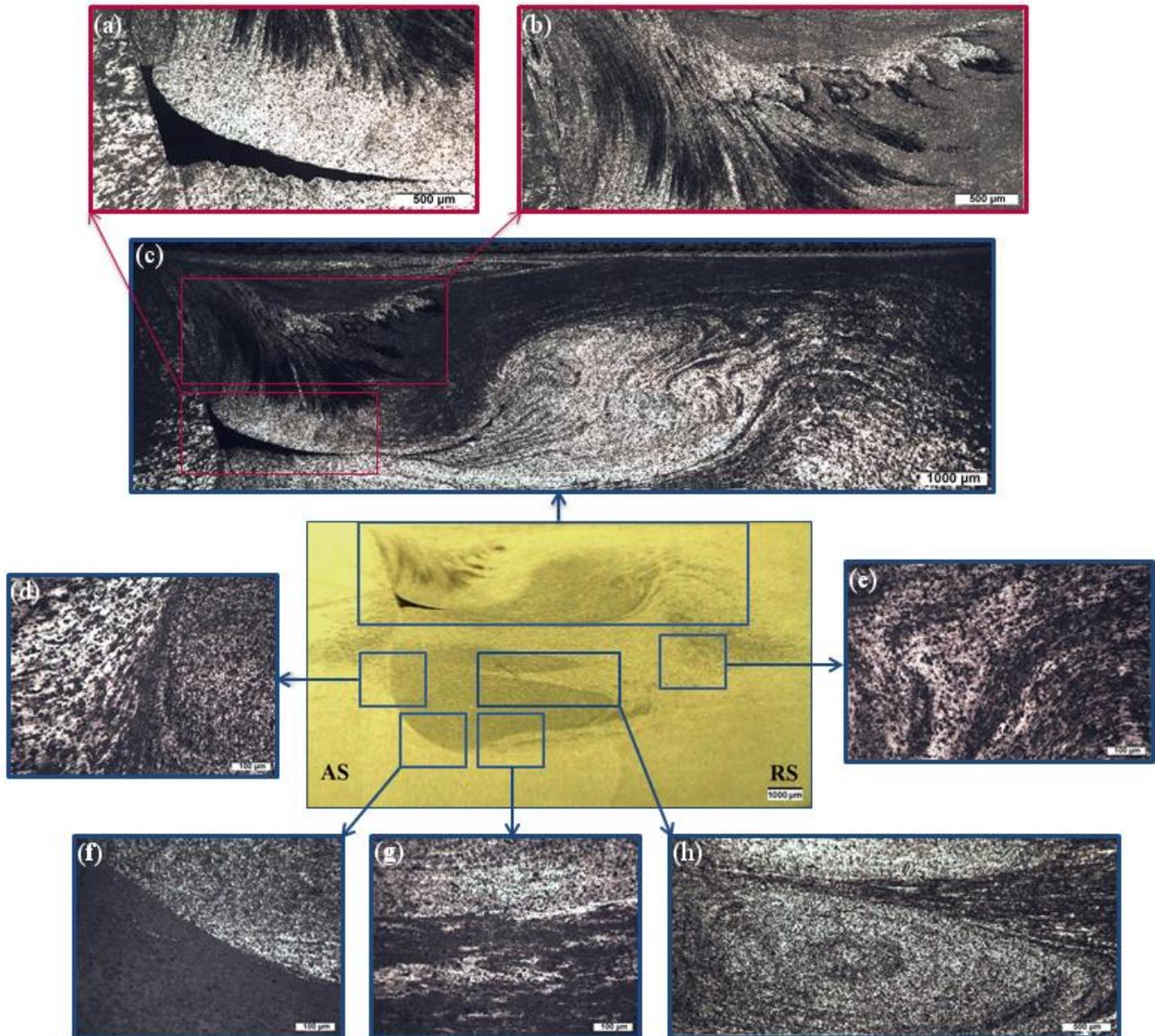
Figure 4. Macrostructure of the FSP zone of Al-TiC composite.

Figure 5 depicts several distinct microstructural regions observed along a friction stir processing bead cross section. The top surface shows very smooth quality and there are almost no prominences or depressions, due to the tool stirring.

The analysis of macrostructure showed a typical FSW defect, called a worm hole (Fig. 5a). A worm hole is a cavity which is manifested by lack of convergence of the materials flowing through various zones on account of insufficient material driving force (incorporation of second phase particles may hinder material flow during fabrication of composites and simultaneously increase chance of defects formation). A worm hole is probably continuous and stable internal channel throughout the processed line. Commonly, the defects tend to occur on the advancing side where a sudden microstructural transition appears from the SZ to the TMAZ, while the transition is gradual on the retreating side [22]. Similarly in this case, the worm hole was majorly noticed at the interface of the SZ and TMAZ on the advancing side. This defect can be caused by higher flow stress of the substrate material and low stirring momentum resulted from low tool rotation speed [8, 15, 23-25].

The distribution of TiC particles is not uniform. It is observed that, powder has been accumulated on the advancing side (AS) of the processed zone. The AS experiences higher level of material stirring, which leads to more intense mixing of reinforcement particles. If the plunge force or rotation momentum is not sufficient to counteract the flow stresses produced by the material, it may not be able to lift the material. And as an effect the material may not be transferred from AS to RS, which leads to higher agglomeration AS [24]. Segregation reduces mechanical and tribological properties of the composite [10]. Perhaps, would be necessary a second pass in order to achieve a suitable distribution. Therefore, darker zones (Fig. 5a-c), correspond to TiC reinforced AA6082 zones.

At the centre, the stir zone is generated due to the combined effect of intense plastic deformation impelled by the tool, and the consequent heat generation (Fig. 5h). A transition between refined grain and plate base material structure, known as the TMAZ is shown in Fig. 5d. Studies also show that, although some new grain nucleation is observed, microstructure remains elongated and deformed due to the generated flow around the stir zone and a precipitate dissolution caused by temperature exposure [11].



**Figure 5.** A cross section macrograph showing various microstructural zones in FSP Al-TiC composite.

Figure 6 shows the representative SEM micrographs of the composite containing of TiC particles in Al matrix. Fig. 6a presents image for 100x magnification. The etchant etches the matrix alone, which leads to the protrusion of TiC particles out of matrix surface. An agglomeration of TiC particles was observed at few places. The higher magnification SEM image in Fig. 6b shows the cluster of particles. Also, many ultrafine particles can also be observed around the relatively large TiC particles (Fig. 6e, g), which may originate largely from the fragmentation of the large TiC particles caused by the high plastic strain and the vigorous stirring action of the rotating tool [1, 4, 19]. An energy dispersive spectroscopy (EDS) analysis taken on one such particle confirms these to be TiC (Fig. 6h). The total composition was: 74.1 wt.% of Al and 25.9 wt.% of Ti.

EDS analysis was conducted to evaluate the morphological features of the specimen. Fig. 6c-d shows the EDS spectrum taken from a rectangular areas 1 and 2 of the micrograph in Fig. 6b,

respectively. The total composition was: 99.5 wt.% of Al and 0.5 wt.% of Ti for area 1 and 91.6 wt.% of Al and 8.4 wt.% of Ti for area 2. Fig. 6f shows the EDS spectrum taken from a rectangular areas of the micrograph in Fig. 6e. The total composition was: 85.5 wt.% of Al and 14.5 wt.% of Ti.

The thermo-mechanical aspect, i.e., combination of heat and plastic deformation during FSP leads to dynamic recrystallization resulting in a finer grain structure. The temperature of the process plays a key role to initiate any kind of reaction between the TiC particle and the matrix [1, 4, 19].

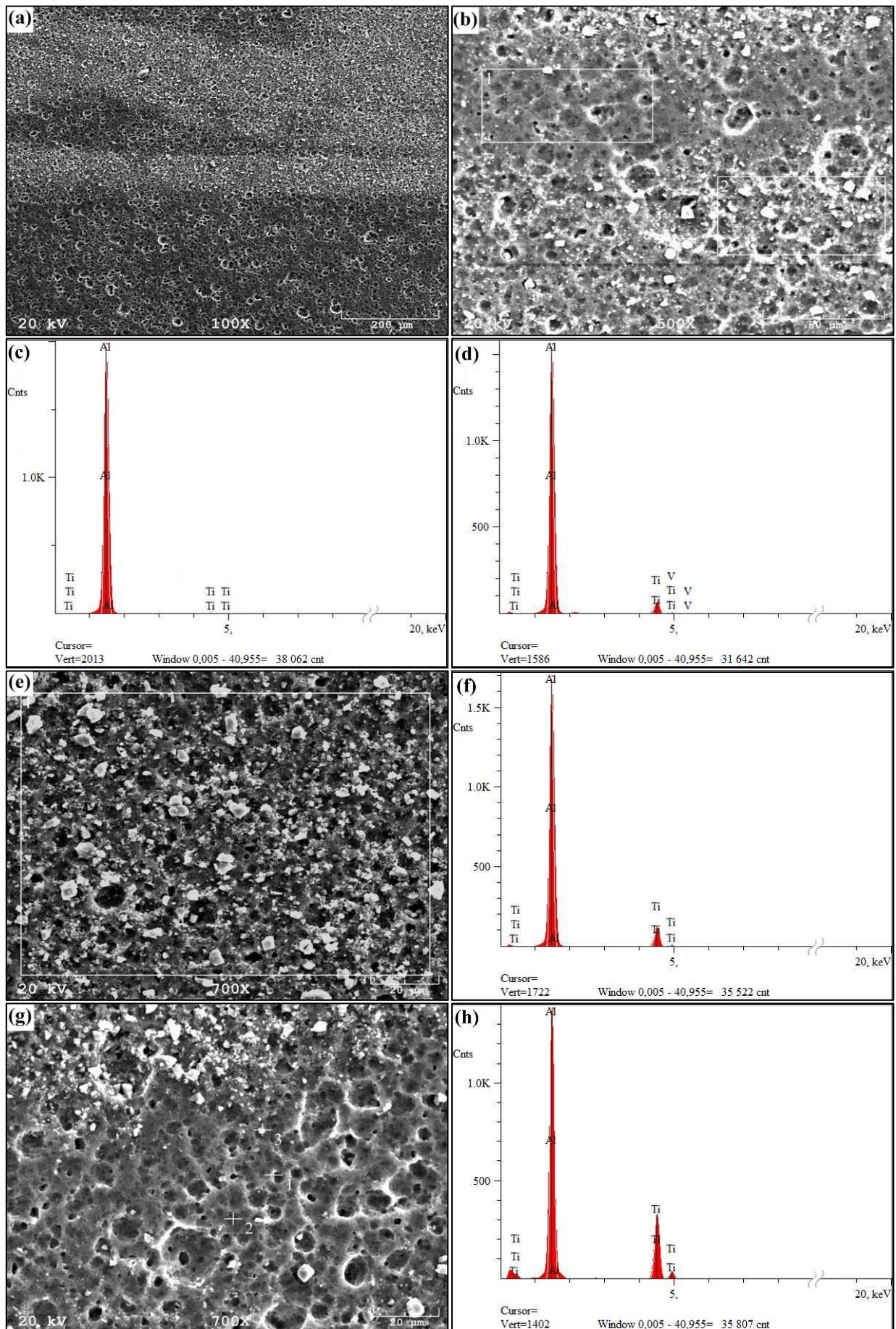


Figure 6. Representative Al-TiC composite microstructures and EDS spectra.

## 4.2. Microhardness

The results of microhardness measurements in different zones of FSP sample are shown in Figure 7. Each hardness data is average of few measurements and the standard deviations are marked on the chart. The microhardness is increased after FSP.

The highest hardness was obtained for zone A, in which the addition of TiC particles accumulated. In this area it was 73.5 HV. After FSP of Al with TiC particles, a considerable improvement in the hardness of the SZ occurred, which reached a value of 63.8 HV. The rise in the microhardness of FSP composite indicates that TiC particles contributed remarkably to the strengthening of the Al matrix.

Furthermore, the microhardness is found to be 62.0 HV in TMAZ and 57.2 HV for zone B. It can be seen that the average hardness of Al without TiC particles (BM) was about 50.9 HV, which was the lowest value in contrast to other areas.

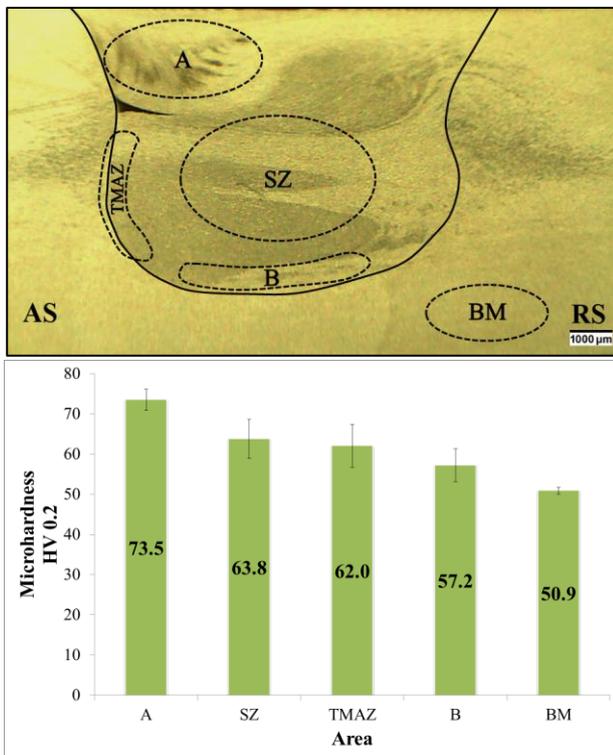


Figure 7. Microhardness distribution in different zones of Al-TiC composite.

## 5. Conclusions

In the present work, AA6082-TiC composite was fabricated using the novel method of FSP and the effect of TiC particles on microstructure and microhardness was analysed. The obtained results can be summarized as follows:

- (1) The top surface is very smooth and there is almost no depressions visible on it. It is caused by the friction stir process.
- (2) Defect like worm hole was observed at the interface of the SZ and TMAZ. The discontinuity appeared

on the advancing side. A worm hole is probably continuous and stable internal channel throughout the processed line. This defect can be caused by higher flow stress of the substrate material and low stirring momentum resulted from low tool rotation speed.

- (3) Macrostructure through a friction stir processing showed a inhomogeneous appearance. The distribution of TiC particles was inhomogeneous in the stir zone. It is observed that, powder has been accumulated on the advancing side (AS). But further experiment may be carried out for improvement in the stirring and there by powder distribution by altering more parameters like tool rotation speed, other powder filling pattern, more powder sizes, direction of each pass, tool tilt angle and number of passes.
- (4) TiC particles were fragmented and broken up due to the high plastic strain and the vigorous rotating action of the tool during FSP.
- (5) The mechanical properties improved after FSP due to modification in the microstructure. TiC particles significantly improved the microhardness and strengthened the composite. The highest hardness is equal to 73.5 HV and was measured for the zone in which the addition of TiC particles accumulated.

## Symbols

AS	advancing side
BM	base material
HAZ	heat affected zone
RS	retreating side
SZ	stir zone
TMAZ	thermo-mechanically affected zone

## References

- [1] Huang G., Hou W., Shen Y.: *Evaluation of the microstructure and mechanical properties of WC particle reinforced aluminium matrix composites fabricated by friction stir processing*, Materials Characterization, 138 (2018) 26-37.
- [2] Jeshurun Lijay K., Raja Selvam J.D., Dinaharan I., Vijay S.J.: *Microstructure and mechanical properties characterization of AA6061/TiC aluminium matrix composites synthesized by in situ reaction of silicon carbide and potassium fluotitanate*, Transactions of Nonferrous Metals Society of China, 26 (2016) 1791-1800.
- [3] Huang G., Shen Y., Guo R., Guan R.: *Fabrication of tungsten particles reinforced aluminium matrix composites using multi-pass friction stir processing: Evaluation of microstructural, mechanical and electrical behavior*, Materials Science & Engineering A, 674 (2016) 504-513.
- [4] Bauri R., Yadav D., Suhas G.: *Effect of friction stir processing (FSP) on microstructure and properties of Al-TiC in situ composite*, Materials Science & Engineering A, 528 (2011) 4732-4739.
- [5] Pandey U., Purohit R., Agarwal P., Dhakad S.K., Rana R.S.: *Effect of TiC particles on the mechanical properties of aluminium alloy metal matrix composites (MMCs)*, Materials Today: Proceedings, 4 (2017) 5452-5460
- [6] Ma Z.Y.: *Friction Stir Processing Technology: A Review*, Metallurgical and Materials Transactions A, 2008.

- [7] Padhy G.K., Wu C.S., Gao S.: *Friction stir based welding and processing technologies – processes, parameters, microstructures and applications: A review*, Journal of Materials Science & Technology, 34 (2018) 1-38.
- [8] Sharma V., Prakash U., Manoj Kumar B.V.: *Surface composites by friction stir processing: A review*, Journal of Materials Processing Technology, 224 (2015) 117-134.
- [9] Sudhakar M., Srinivasa Rao CH., Meera Saheb K.: *Production of Surface Composites by Friction Stir Processing – A Review*, Materials Today: Proceedings, 5 (2018) 929-935.
- [10] Thangarasu A., Murugan N., Dinaharan I.: *Production and wear characterization of AA6082-TiC surface composites by friction stir processing*, Procedia Engineering, 97 (2014) 590-597.
- [11] Miranda R.M., Gandra J.P., Vilaca P., Quintino L., Santos T.G.: *Surface Modification by Solid State Processing*, Woodhead Publishing, Cambridge 2014.
- [12] Węglowski M.S.: *Friction stir processing – State of the art*, Archives of Civil and Mechanical Engineering, 18 (2018) 114-129.
- [13] Nelaturu P., Jana S., Mishra R.S., Grant G., Carlson B.E.: *Influence of friction stir processing on the room temperature fatigue cracking mechanisms of A356 aluminium alloy*, Materials Science & Engineering A, 716 (2018) 165-178.
- [14] Salman A.A., Takak A.M., Resan K.K.: *Study the mechanical properties and Numerical evaluation of Friction Stir Processing (FSP) for 7075-T73 aluminum alloys*, Babylon university conference, 2015.
- [15] Rana H., Badheka V.: *Influence of friction stir processing conditions on the manufacturing of Al-Mg-Zn-Cu alloy/boron carbide surface composite*, Journal of Materials Processing Tech., 255 (2018) 795-807.
- [16] Karthikeyan L., Senthilkumar V.S., Padmanabhan K.A.: *On the role of process variables in the friction stir processing of cast aluminium A319 alloy*, Materials and Design, 31 (2010) 761–71.
- [17] Surekha K., Murty B.S., Rao, Prasad K.: *Effect of processing parameters on the corrosion behaviour of friction stir processed AA 2219 aluminum alloy*, Solid State Sciences, 11 (2009) 907–17.
- [18] Elangovan K., Balasubramanian V.: *Influences of tool pin profile and welding speed on the formation of friction stir processing zone in AA2219 aluminum alloy*, Journal of Materials Processing Technology, 200 (2008) 163–75.
- [19] Thangarasu A., Murugan N., Dinaharan I., Vijay S.J.: *Synthesis and characterization of titanium carbide particulate reinforced AA6082 aluminium alloy composites via friction stir processing*, Archives of Civil and Mechanical Engineering, 15 (2015) 324-334.
- [20] Mishra R.S., Ma Z.Y.: *Friction stir welding and processing*, Materials Science and Engineering R, 50 (2005) 1-78.
- [21] Rao D., Huber K., Heerens J., et al.: *Asymmetric mechanical properties and tensile behaviour prediction of aluminium alloy 5083 friction stir welding joints*, Materials Science & Engineering A, 565 (2013) 44-50.
- [22] Nandan R., Debroy T., Bhadeshia H.: *Recent advances in friction stir welding process, weldment structure and properties*, Progress in Materials Science, 53 (2008) 980-1023.
- [23] Chainarong S., Muangjunburee P., Suthummanon S.: *Friction Stir Processing of SSM356 Aluminium Alloy*, Procedia Engineering, 97 (2014) 732-740.
- [24] Rana H.G., Badheka V.J., Kumar A.: *Fabrication of Al7075/B4C surface composite by novel Friction Stir Processing (FSP) and investigation on wear properties*, Procedia Technology, 23 (2016) 519-528.
- [25] Mahmoud E., Ikeuchi K., Takahashi M.: *Fabrication of SiC particle reinforced composite on aluminium surface by friction stir processing*, Science and Technology of Welding & Joining, 13 (2008) 607-618.