



# Influence of volume percentage of NanoTiB<sub>2</sub> particles on tribological & mechanical behaviour of 6061-T6 Al alloy nano-surface composite layer prepared via friction stir process



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## ARTICLE INFO

### Article history:

Received 8 August 2016

Received in revised form

14 October 2016

Accepted 11 November 2016

Available online 23 December 2016

### Keywords:

Friction stir process

Al alloy

Surface nano composites

Tensile

Tribology

## ABSTRACT

The aim of present study is to analyze the influence of volume percentage (vol.%) of nano-sized particles (TiB<sub>2</sub>: average size is 35 nm) on microstructure, mechanical and tribological behavior of 6061-T6 Al alloy surface nano composite prepared via Friction stir process (FSP). The microstructure of the fabricated surface nanocomposites is examined using optical microscopy (OM) and scanning electron microscope (SEM) for distribution of TiB<sub>2</sub> nano reinforcement particles, thickness of nano composite layer formed on the Aluminum alloy substrate and fracture features. The depth of surface nano composite layer is measured as 3683.82 μm along the cross section of stir zone of nano composite perpendicular to FSP. It was observed that increase in volume percentage of TiB<sub>2</sub> particles, the microhardness is increased up to 132 Hv and it is greater than as-received Al alloy's microhardness (104 Hv). It is also observed that at 4 volume percentage higher tensile properties exhibited as compared with the 2 and 8 vol. %. It is found that high wear resistance exhibited at 4 volume percentage as-compared with the 2 and 8 vol. %. The observed wear and mechanical properties are interrelated with microstructure, fractography and worn morphology.

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## 1. Introduction

Aluminum alloy 6061-T6 is widely utilized in aircraft, defence, automobiles and marine areas due to their good strength, light weight and better corrosion properties. But, they exhibits inferior tribological properties in extensive usage [1]. Aluminum matrix composites (AMCs) which are produced by reinforcing Aluminum alloys with particles like SiC, Al<sub>2</sub>O<sub>3</sub> and TiB<sub>2</sub> etc., are the new generation materials. These AMCs exhibit higher properties than parent alloy such as stiffness, improved tribological characteristics and high strength. Further these properties can be enhanced by using nano scale ceramic materials [2–4]. Distribution of nano reinforcement particles on Al alloy surface and its control is complex to achieve in conventional surface modification methods [4,5].

Earlier researches [6,7] reported that thermal spraying and laser beam techniques were utilized to prepare surface composites, in which it degrades the properties due to creation of unfavorable phases. These techniques operate at higher temperatures and impossible to avoid the reaction between the reinforcements and the matrix, which forms detrimental phase. A process can be employed which is operated at a below melting temperature of the matrix for the fabrication of surface composites which can be avoided the above mentioned problems. Considering these problems, Friction stir processing (FSP) is the best technique suited for preparation of surface composites and surface modification. In FSP, a rotating tool with the shoulder and pin is plunge onto the surface of a material, which creates frictional heat and dynamic mixing of material area underneath of the tool and it results to incorporate and/or disperse the reinforcement particles in the matrix material such as Aluminum alloys, Magnesium alloys and Copper alloys [8–12]. This investigation is aimed to fabricate the nano Titanium Boride (TiB<sub>2</sub>) particle reinforced 6061-T6 Al alloy surface nano-composites by using FSP. Extended to study, influence of volume percentage of nano sized TiB<sub>2</sub> (average size is 35 nm) reinforcement

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Peer review under responsibility of China Ordnance Society.

particles on microstructural characterization, mechanical and tribological behaviour of 6061-T6 Al alloy based nano surface-composites prepared via FSP.

## 2. Experimental

The base material employed in this study is 6 mm thick Aluminum alloy 6061-T6. The chemical composition of the base material is given in Table 1. The nano sized reinforcement particles such as  $TiB_2$  is used at different volume percentages (vol. %) such as 2, 4 and 8. The average size of the reinforcement particles is 35 nm and scanning electron microscope (SEM) microstructure of as-received  $TiB_2$  nano-particles are shown in Fig. 1. The square groove was made tangent to the pin in the advancing side and which is 1 mm far away from the center line of the tool rotation on the Aluminum alloy 6061-T6 plate. The H13 tool steel having screwed taper pin profile with shoulder diameter of 24 mm, pin diameter of 8 mm and 3.5 mm height was used. The groove opening initially closed by means of the tool which is having shoulder without pin to avoid the escapement of reinforcement particles from groove while processing. Working range of process parameters along the center line used in FSP are presented in Table 2. The FSP is carried out on a Vertical milling machine (Make HMT FM-2, 10 hp, 3000 rpm) (See Figs. 2 and 3).

After FSP, microstructural observations were carried out at the cross section of NZ of surface nano composites normal to the FSP direction, mechanically polished and etched with Keller's reagent (2 ml HF, 3 ml HCl, 20 ml  $HNO_3$  and 175 ml  $H_2O$ ) by employing optical microscope (OM). Microhardness tests were carried out at the cross section of NZ of surface hybrid composites normal to the FSP direction, samples with a load of 15 g and duration of 15 s using a Vickers digital microhardness tester. The tensile specimens were taken from the surface hybrid composites normal to the FSP direction and made as per ASTM: E8/E8M-011 standard by wire cut Electrical discharge machining to the required dimensions. Wear test is carried out on a pin-on-disk tribometer as per ASTM: G99-05 standard. The prismatic pins of 8 mm dia are cut from the stir zone, where the axis of the pin perpendicular to the FSP process direction. The EN31 steel having a hardness of 62 HRC is used as a disc. The dia of the sliding track on the disc surface is 100 mm. The wear test was carrying out under dry-sliding condition with a constant load of 40 N, disc rotational of speed 650 rpm and sliding speed of 3.4 m/s. Wear rate is determined by, Wear rate ( $mm^3/m$ ) is equal to (volume loss/sliding distance).

## 3. Results and discussion

### 3.1. Microstructure

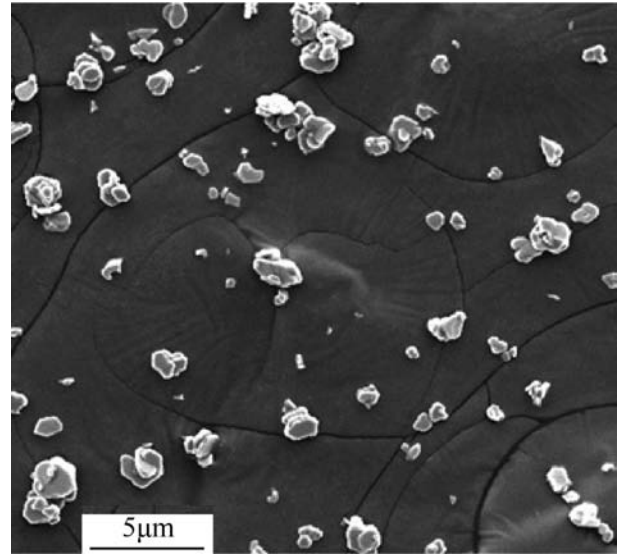
The friction stirred zone was typically about the size of the rotating pin width and depth of 8 mm and 4 mm respectively. The cross section of stir zone of surface nano composite normal to the FSP direction is shown in Fig. 5 and depth of surface nano composite layer is measured as 3683.82  $\mu m$ . The SEM micrographs of Al- $TiB_2$  surface nano composites and as-received Al alloy are shown in Fig. 6.

It is observed that nano-size  $TiB_2$  particles are distributed within this zone because of that event about dynamic stirring during the

**Table 1**

Chemical composition of Aluminum 6061-T6 alloy (Wt. %).

Element	Mg	Si	Cu	Zn	Ti	Mn	Cr	Al
Amount/(Wt %)	0.85	0.68	0.22	0.07	0.05	0.32	0.06	Balance



**Fig. 1.** SEM micrographs of as-received  $TiB_2$  particles.

FSP. It is revealed that  $TiB_2$  nano particles are dispersed uniformly in nugget zone (NZ) at 4 vol. % as compared to other 2 and 8 vol. % of Al/ $TiB_2$  surface nano-composites prepared by FSP because of rotating tool provides adequate heat and circumferential force to dispense the reinforcement particles to occupied in wider zone [12,13].

### 3.2. Microhardness

Microhardness survey of Al- $TiB_2$ -2 vol. %, Al- $TiB_2$ -4 vol. % and Al- $TiB_2$ -8 vol. % surface nano-composites and base metal (i.e. Al alloy) as seen in Fig. 4. Mostly, the microhardness value is depends on the presence of  $TiB_2$  particles and their homogeneous dispersal. It is seen that expansion over volume rate of  $TiB_2$ , microhardness increments up to 132 Hv and also which may be higher over as-received Aluminum alloy (104 Hv). This will be because of at 1400 rpm, tool shoulder generates sufficient heat and shear force utilized to create the reinforcement particles more effortlessly wrapped by the softened material and revolves around the FSP tool which results in proper severance and allocation in the nugget zone. The volume percentages of  $TiB_2$  particles are increased causes refinement of grain size and reducing of ductile matrix content which results in reduce the ductility of the AMCs [13,14]. It is also observed that the increasing the volume percentage of  $TiB_2$  particles immensely increases the microhardness due to the presence and pinning effect of the  $TiB_2$  particles. The presence of  $TiB_2$  particles is considered for more effective formation of fine grain

**Table 2**

Working range of process parameters.

Parameters	Value
Tool rotational speed/rpm	1120
Tool traverse speed/( $mm \cdot min^{-1}$ )	40
Tool vertical force/kN	5
Tool tilt angle/( $^\circ$ )	2
Tool pin profile	Taper with threaded
Tool shoulder diameter/mm	24
Tool pin diameter/mm	8
$D_s/D_p$ ratio	3
Number of passes	1
Volume percentage of $TiB_2$ nano particles/(vol.%)	2, 4 and 8

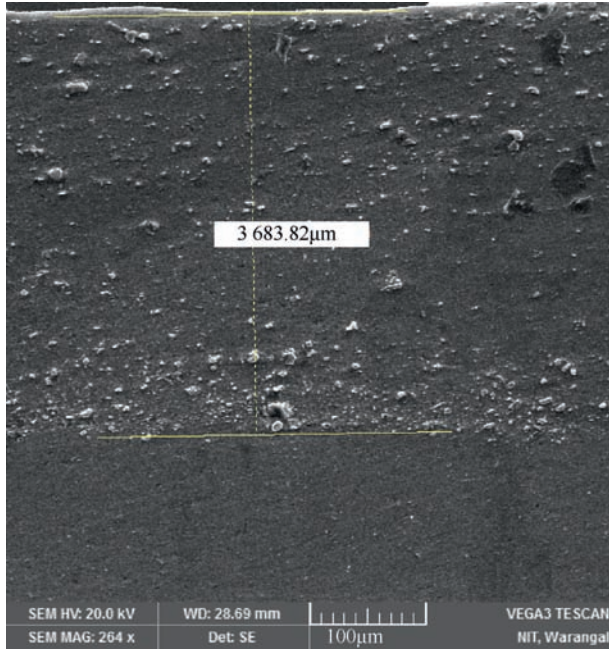


Fig. 2. SEM microstructure of Al/TiB<sub>2</sub> surface nano composite for depth of composite formation.

structure due to the restraint of grain boundary and the enhancement of the induced strain. The presence of TiB<sub>2</sub> particles is considered for more effective formation of fine grain structure due to the restraint of grain boundary and the enhancement of the induced strain [13–16]. However the higher hardness is achieved by the TiB<sub>2</sub> particles.

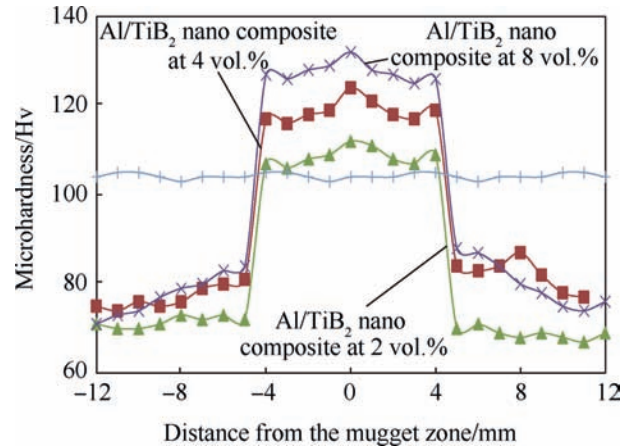


Fig. 4. Microhardness survey of Al/TiB<sub>2</sub> surface nano composites and as-received Al alloy.

### 3.3. Tensile properties

The Tensile properties of Al-TiB<sub>2</sub>-2 vol. %, Al-TiB<sub>2</sub>-4 vol. % and Al-TiB<sub>2</sub>-8 vol. % surface nano-composites and base metal Al alloy is existed in Table 3. All the tensile properties are shown graphically in Fig. 5. It is seen that all the tensile properties of Al nano surface-composites were decreased as compared with the base metal Al alloy. This is due to the existence of reinforcement particles might be restricting the grain boundary sliding and dislocations. Ultimately it leads to deteriorate the tensile properties [15,16]. It is observed that the increasing the volume percentage of TiB<sub>2</sub> particles, the UTS, YS and %EL immensely decreases. However, increasing the volume percentage of TiB<sub>2</sub> particles increases the

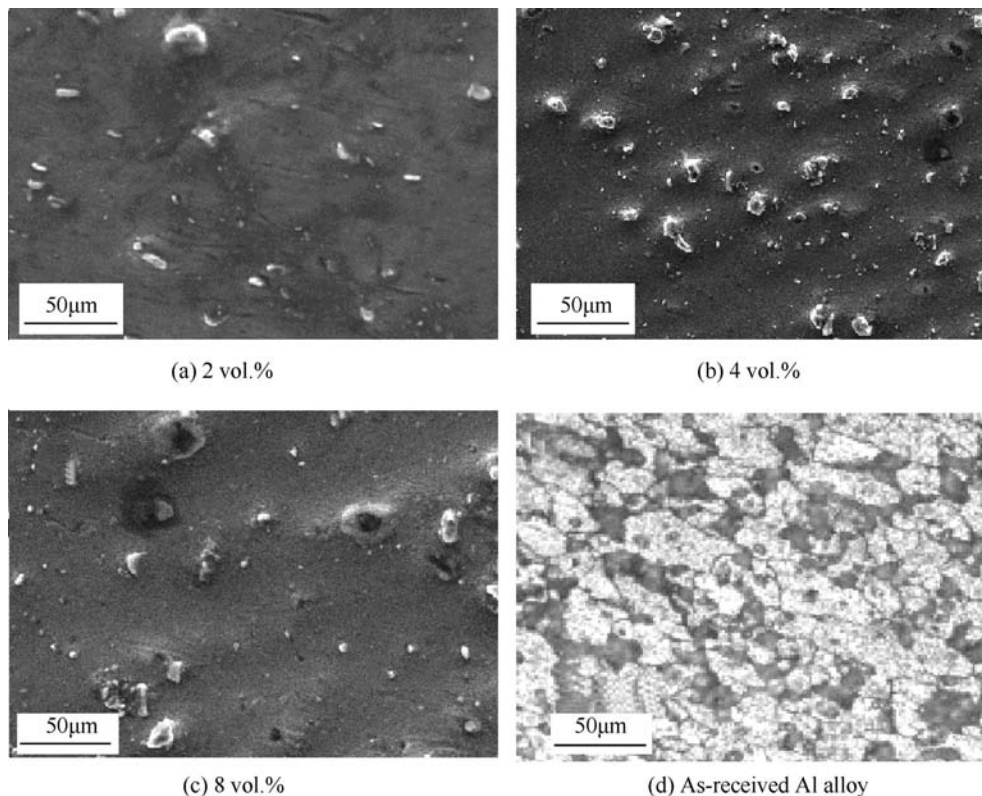


Fig. 3. SEM microstructures of Al/TiB<sub>2</sub> surface nano composites (a) 2 vol. %, (b) 4 vol. % and (c) 8 vol. %.

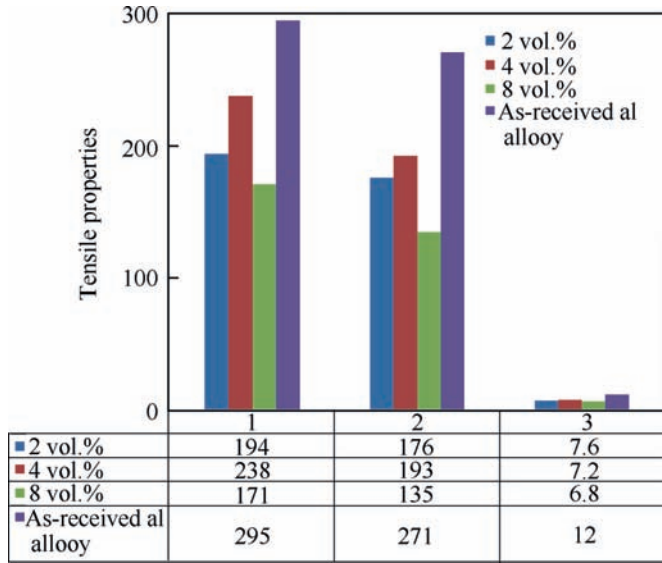


Fig. 5. Comparison of Tensile properties of Al/TiB<sub>2</sub> surface nano composites & d) As-received Al alloy.

interface area between the TiB<sub>2</sub> particles and Aluminum matrix due to low inter particles space area causes the agglomeration of TiB<sub>2</sub> particle results in low tensile properties [17]. The volume percentage of TiB<sub>2</sub> particles increases could be restrict the grain boundary sliding, dislocations and also the weak interfacial bond between the reinforcement particles and the matrix, finally it leads to deteriorate the tensile properties [18,19]. In other words

composite has incompatible deformation between the plastically deformed matrix and rigid reinforcement particles caused geometrical dislocations. Besides, increasing the volume percentage of TiB<sub>2</sub> particles increases the effective slip distance of dislocations during deformation, which leads to reduce elongation [20].

The SEM fractography of Al-TiB<sub>2</sub>-2 vol. %, Al-TiB<sub>2</sub>-4 vol. % and Al-TiB<sub>2</sub>-8 vol. % surface nano composites and base metal Al alloy is seen in Fig. 6. By observing the fracture surface of base metal Al alloy consist of voids and dimple which reveals the ductile fracture (Fig. 6 (d)). It is also observed that the extracting out of the reinforcement particles and minute dimples are seen in the rupture surface of Al-TiB<sub>2</sub>-2 vol. %, Al-TiB<sub>2</sub>-4 vol. % and Al-TiB<sub>2</sub>-8 vol. % surface nano-composites. These observations are further confirming the stated causes for lower UTS, YS and %EL of all the surface composites [17,18].

### 3.4. Wear properties

Wear rate versus the sliding distance of Al-TiB<sub>2</sub> surface nano-composites and base metal Al alloy was revealed in Fig. 7. It is shown that an increase in the volume percentage of TiB<sub>2</sub> particles which decreases the wear rate. Because of the enhanced hardness by the distribution TiB<sub>2</sub> particles and also which acts as load bearing particles. Wear rate is decreased by increasing the volume percentage of TiB<sub>2</sub> particles and also at larger volume percentage of TiB<sub>2</sub> particles increases the wear rate due to pulled out of hard nano-TiB<sub>2</sub> particles from the composite specimen pin during the wear process and which formed on the steel disc and acted as barrier. Further it converts the adhesive wear to abrasive wear which results in more amount of material wear-out from the composite specimen pin [21,22]. The lower wear rate is attained at a rotational speed of 1120 rpm, 4 volume percentage of TiB<sub>2</sub>.

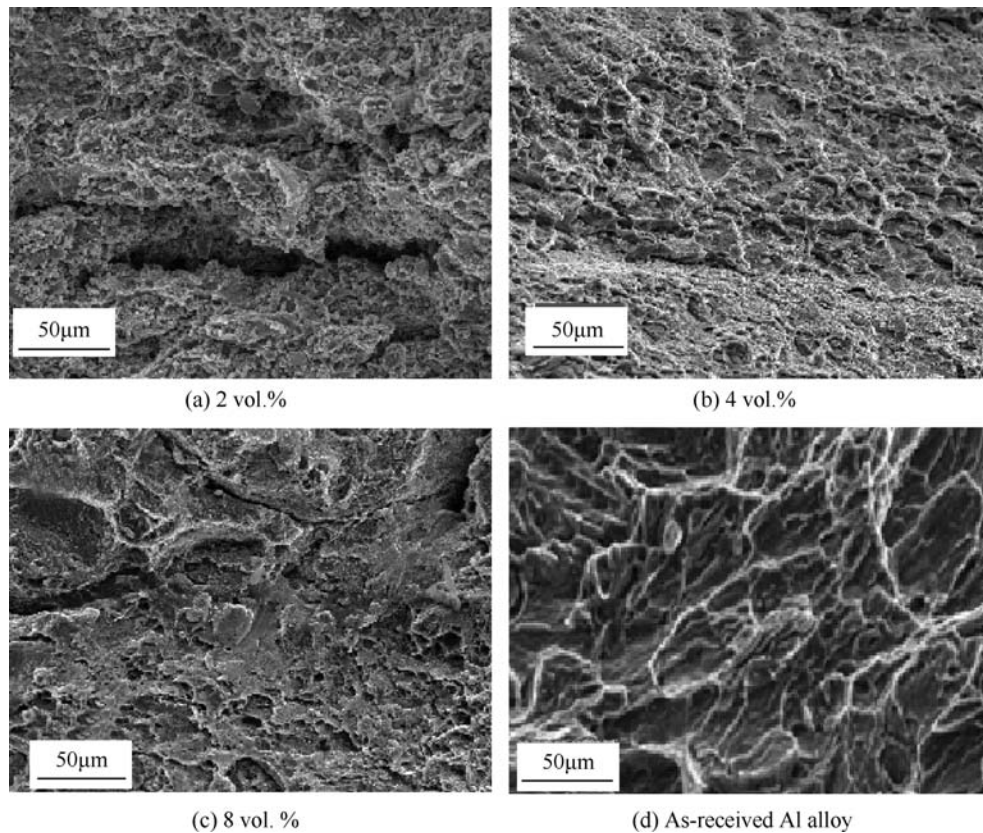
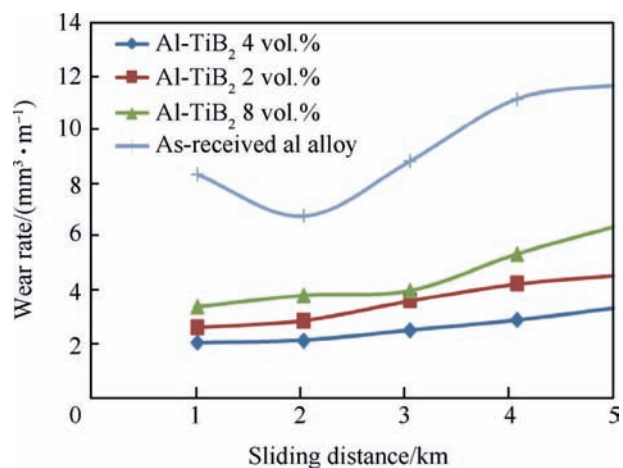


Fig. 6. Fracture features of Al/TiB<sub>2</sub> surface nano composites a) 2 vol. %, b) 4 vol. % and c) 8 vol. % and d) As-received Al alloy.

**Table 3**  
Tensile properties of Al/TiB<sub>2</sub> surface nano composites.

Al/TiB <sub>2</sub> surface nano composites	Ultimate strength (UTS)/MPa <sup>a</sup>	Yield strength (YS)/MPa <sup>a</sup>	Percentage of Elongation/(%EL) <sup>a</sup>
2 vol. %	194	176	7.6
4 vol. %	238	193	8.2
8 vol. %	171	135	6.8
As-received Al alloy	295	271	12

<sup>a</sup> Average of four values.

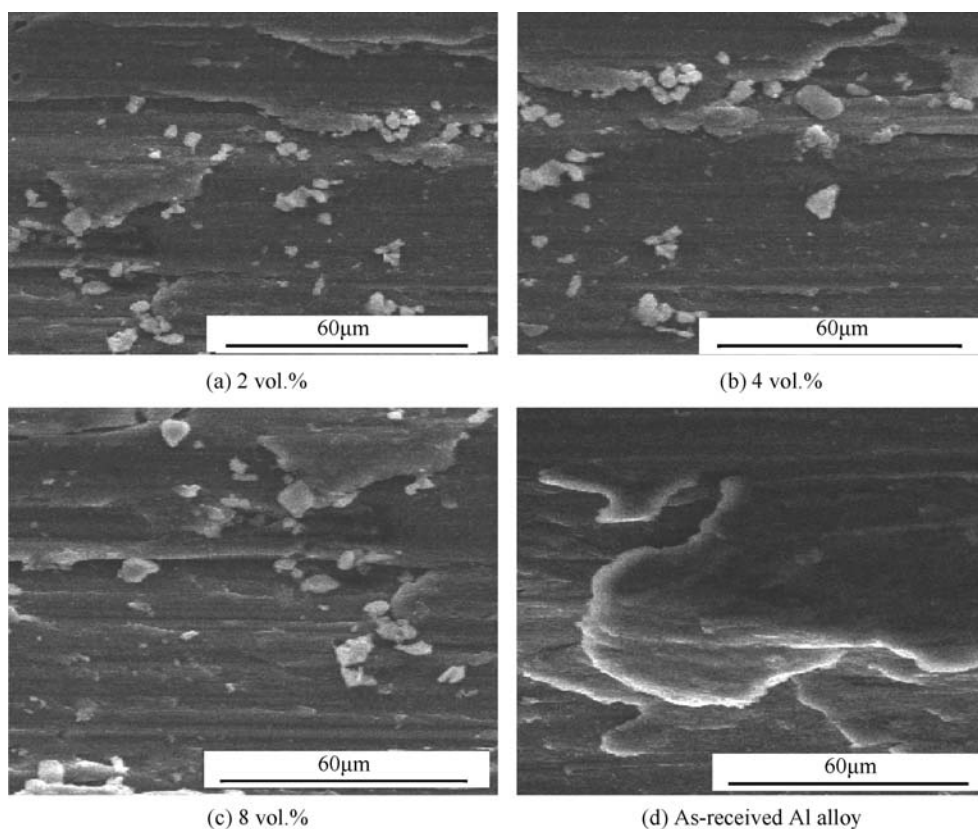


**Fig. 7.** Wear rate with respect to the sliding distance of Al-TiB<sub>2</sub> surface nano composites and as-received Aluminum alloy.

The wear morphology of Al-TiB<sub>2</sub> surface nano composites and base metal Al alloy is seen in Fig. 8. The presence of tribo mechanically mixed layer converts the wear manner as of two body to three body wear and decreases wear rate which acts as solid lubricant. It could be observed that the wear tracks are larger in the base metal Al alloy as compared to the Al-TiB<sub>2</sub> surface nano composites due to the occurrence of hard TiB<sub>2</sub> particles. However, the nonappearance of TiB<sub>2</sub> particles which causes the micro plough-up of surface in contact during wear to make irregular surface [22].

#### 4. Conclusions

The nano composite surface layer by reinforcing TiB<sub>2</sub> particles on 6061-T6 Aluminum Alloy via FSP successfully fabricated. Influence of nano-sized reinforcement particles of TiB<sub>2</sub> (average size is 35 nm) on microstructure, mechanical and tribological behaviour of 6061-T6 Al alloy surface nano composite prepared via Friction stir process was studied and the obtained conclusions are:



**Fig. 8.** Wear morphology of Al-TiB<sub>2</sub> surface nano composites a) 2 vol. %, b) 4 vol. % and c) 8 vol. % and d) As-received Al alloy.

- 1) The depth of surface nano composite layer is measured as 3683.82  $\mu\text{m}$ .
- 2) An expansion over volume rate of  $\text{TiB}_2$ , microhardness increments up to 132 Hv and also which may be higher over as-received Aluminum alloy (104 Hv).
- 3) It is seen that all the tensile properties of Al surface nano composites were decreased as compared with the base metal Al alloy.
- 4) It is found that high wear resistance exhibited at 4 volume percentage (vol. %) as compared with the 2 and 8 vol. %.
- 5) It is also found that the wear debris formation is higher at the 8 vol. % of Al- $\text{TiB}_2$  surface nano composite.

### Acknowledgements

The author(s) would like to express thanks the authorities of NIT-Warangal, DMRL-Hyderabad and RCI-Hyderabad for provided that the facilities to carried-out my research work. One of the authors *Dr. A. Devaraju* is thankful to the Principal and the management of **S R Engineering College, Warangal-Telangana State**, for their constant support during this work.

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