

**AN INVESTIGATION INTO TRIBOLOGICAL
BEHAVIOUR OF Al-TiN-GRAPHITE
NANO-COMPOSITES FABRICATED BY
FRICTION STIR PROCESSING**

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Abstract

The present study aims to fabricate the surface aluminum-TiN-graphite composite through friction stir processing (FSP), and investigate the tribological features of the produced composite. Microhardness profiles, pin-on-disk test and

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both optical and electron microscopy are used to probe the characteristics of the surface composite. The results indicate that using the blend of TiN/graphite as reinforcing component rather than individual graphite or TiN improves the wear resistance of composite. According to the outcomes, existence of graphite hinders clustering of TiN particles and also lubricates the contact surface, all leading to a mild wear regime. It is also shown that wear resistance of the fabricated composite depends on the ratio of TiN to graphite, where the optimum resistance is obtained at a TiN/graphite volume ratio of 0.2. Moreover, it is observed that hardness distribution through the fabricated composite is not uniform, and a maximum hardness occurs below the surface, which does not shift by altering the TiN/graphite ratio.

1. Introduction

Metal matrix composites (MMCs) usually exhibit a higher strength, higher elastic modulus, and an improved tribological behaviour. Such characteristics make these composites promising structural materials for aerospace and automobile industries [14]. Interesting engineering features of aluminum along its relatively weak mechanical and tribological characteristics has made the aluminum matrix composites an engaging topic for researchers, where they can heal the weakness of aluminum matrix through reinforcing components. In recent decades, using graphite as reinforcing component in MMCs has been common to reduce the friction coefficient [3, 4]. Also, carried out researches suggest the constructive role of graphite in wear resistance of aluminum matrix composites, where development of a thin layer of graphite prevents an intense contact of sliding surfaces [19]. However, there are two known primary problems in composites containing graphite; firstly, distribution and non-wetting condition of graphite, which might aggravate the tribological properties of composite [21], and secondly, reduction of hardness and wear resistance due to excessive content of graphite [13]. To compensate these potential problems, hybrid composites in which a hard component like ceramics is added to graphite has been introduced [3, 4, 13, 14, 19, 21]. SiC [19], Al₂O₃ [3, 4, 13, 14, 19, 21], and Ti [21] are the main hard components having been investigated. Also, the ultimate features of a hybrid composite vary depending on kind of hard material, ratio of graphite to hard material, fabrication process, and applied parameters.

Friction stir processing (FSP) is a new method applied to produce surface composites based on principals of friction stir welding [10]. So far, several studies have been carried out to produce various surface composites through friction stir processing [2, 7, 17]. It has been shown that FSP can contribute to hardness of matrix through dynamic recrystallization and a consequent grain refinement [8]. Furthermore, these studies indicate that FSP is able to enhance the wear properties of Al alloys by a proper distribution of reinforcing components [18, 22]. Thus, the mentioned advantages have engaged industries to utilize the friction stir processing as an efficient method for fabrication of surface aluminum matrix composites.

The present study aims to utilize TiN as an excellent hard and lubricant component [6] in fabrication of Al-TiN-graphite composite through friction stir processing. The hardness distribution and wear properties of surface composite as two main tribological features have been investigated, and an attempt has been made to find the effect of TiN/graphite ratio on wear resistance of composite. The outcomes can be used to design a proper modified procedure for fabrication of Al-TiN-graphite surface composite by friction stir processing method.

2. Materials and Methods

Table 1 shows the chemical composition of Al6061 used in the current study to produce the aluminum matrix composite. Also, TiN nano powder with mean size of 50nm was added to graphite powder possessing an average size of 10-20 micron to form the reinforcing component of composite. As Figure 1(a) illustrates, the $100 \times 50 \times 20$ mm plates of aluminum were fixed in a flat position by a fixture during friction stir processing. The applied tool was made of hot working steel alloy, which its hardness reached 45HRC after heat treatment. Furthermore, as Figure 1(b) depicts the geometry of used tool, the tool was composed of a 20mm diameter shoulder and a cylindrical pin with diameter of 4mm and height of 3mm. Also, a uniform U-shape groove with depth of 2mm and

an opening of 1mm was made on aluminum plates along the direction of tool movement to put the powder mixture in it. To investigate the effect of TiN/graphite ratio on wear properties of fabricated composite, 5 various samples with different volume ratios of TiN/graphite were prepared. The utilized ratios are listed in Table 2. As Figure 1(c) illustrates, rotational and advancing speeds as main friction stir processing parameters were adjusted to 1800rpm and 80mm/min, respectively. Also, the tilt angle was kept at 3 degree during the fabrication process. It is noteworthy to mention that selected FSP parameters are proved to have an optimum performance in case of Al-Al₂O₃-graphite composite fabrication [14]. Furthermore, each sample was subjected to two passes of friction stir processing to achieve a better distribution of powder. Distribution of TiN/graphite powder was examined through scanning electron microscopy (SEM). Also, optical microscopy was utilized to observe the microstructure of composite, and cross-sectional hardness distribution of stir zone was evaluated by Vickers hardness test using a load of 200 grams and a dwell time of 15 seconds.

Table 1. The measured chemical composition of Al6061 through quantometer

Al	Mg	Si	Fe	Cu	Zn	Ti	Mn	Cr	Other
Balance	1.05	0.63	0.61	0.32	0.12	0.01	0.07	0.05	0.05

Table 2. The utilized volume TiN/graphite ratios for various samples

Sample	No. 1	No. 2	No. 3	No. 4	No. 5
TiN/graphite volume ratio	0	0.2	0.5	0.8	1

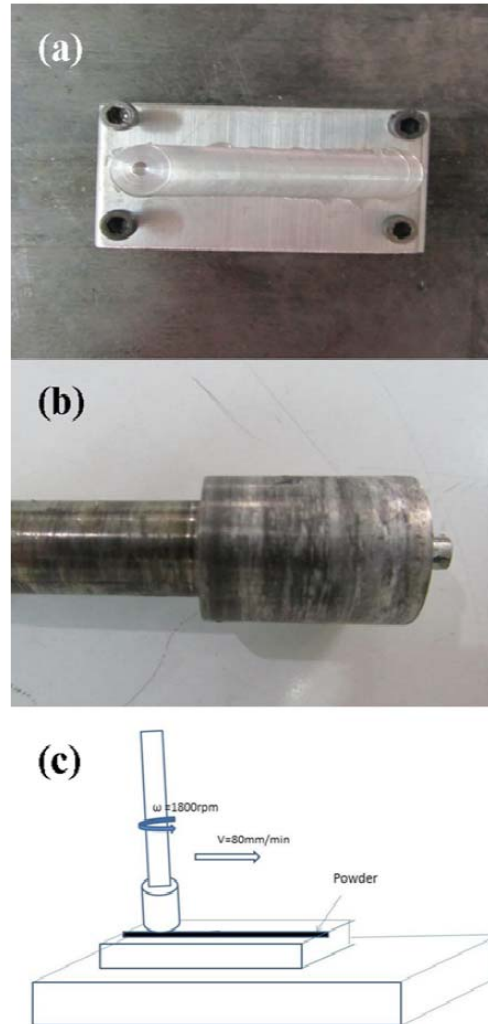


Figure 1. Schematic configuration of friction stir processing.

In order to assess the wear resistance of fabricated composites, the pin-on-disk test was applied, following The ASTM G99-04 standard. The pin-on-disk test was carried out under a load of 10N for a sliding distance of 1000m with a velocity of 0.5m/s at dry sliding condition. The mass loss of all wear samples was measured through weighing before and after wear test. In addition, the surface of worn-out samples was examined by SEM images.

3. Results and Discussion

As Figure 2(a) illustrates, three main distinct regions are detectable on cross section of friction stir processed aluminum: stir zone (SZ), which forms through extrusion of plasticized material; thermo-mechanically affected zone (TMAZ); and finally the base metal (BM) region in which no significant change has happened (Figure 2(b)). As it is seen in Figure 2(c) and (d), the dominant microstructural feature of TMAZ is a grain elongation caused by shear forces coming from stream of materials in the stir zone [5]. Since SZ is the only region in which the plasticized material can flow and distribute the reinforcing component, this area is a potential bed for composite development, and all investigations will be focused on the stir zone to characterize the formed composite. Figure 2(c) reveals a sharp boundary between stir zone and TMAZ, where existence of reinforcing particles can be introduced as primary reason for this phenomenon. Observing the stir zone at higher magnification indicates a severe grain refinement of aluminum matrix, which can be attributed to simultaneous dynamic recrystallization and pinning effect of reinforcing particles [9, 11]. Also regarding the size of particles, only TiN nano powder is expected to prevent the grain growth through pinning effect and graphite powder is not able to lock the grain boundaries on its own [15].

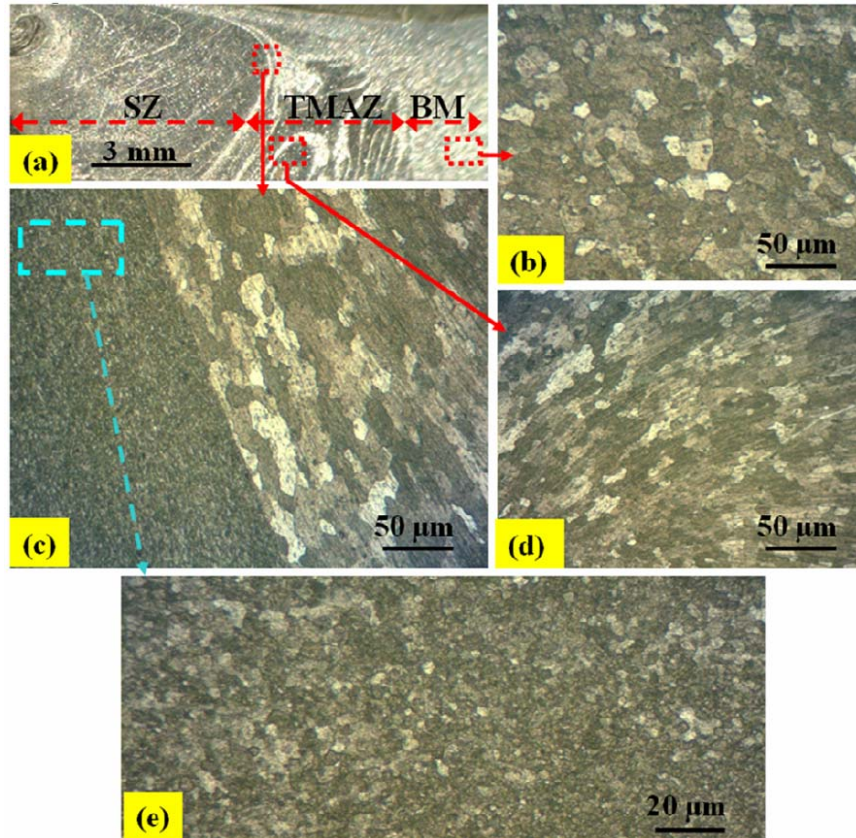


Figure 2. Cross-sectional view of a processed sample: (a) Macroscopic view of SZ, TMAZ, and base metal (BM); (b) microstructure of base metal; (c) the boundary between SZ and TMAZ; (d) microstructure of TMAZ; and (e) SZ in higher magnification (revealing a severe grain refinement in the SZ).

Proper distribution of nano hard particles and graphite powder without clustering has an important influence on mechanical and wear properties of composites. Moreover, a successful pinning effect requires a uniform distribution of TiN nano particles. Figure 3 shows the SEM micrographs of 4 samples with different volume ratios of TiN/graphite. As it is seen, in all studying values of TiN/graphite volume ratio, even by complete removal of TiN (Figure 3(a)), graphite particles are distributed

uniformly. However, results indicate that the same condition is valid for TiN powders, if graphite component exists. Comparison of samples No. 2 (Figure 3(b)) and No. 3 (Figure 3(c)) with sample No. 5 (Figure 3(d)) suggests the constructive role of graphite in prevention of TiN clustering. In another word, excessive increase of TiN/graphite ratio or complete removal of graphite powder from reinforcing mixture might results in clustering of TiN particles. Figure 4 depicts a cluster of TiN nano particles shown by a dashed circle in Figure 3(d). Figure 5 shows the hardness of fabricated composites at the top surface. Each value is average of 6 different measurements conducted at various areas. According to the obtained results, the hardness of all samples is higher than as-received aluminum matrix (32 Hv). This surface hardening can be ascribed to a combination of different reasons. Firstly, grain refinement of aluminum matrix due to friction stir processing (Figure 2), and secondly existence of reinforcing particles, which make the dislocations movement difficult [5, 15]. As a result, the indenter is not able to penetrate as much as it could do in an as-received aluminum matrix, and hardness value increases. Moreover, it is seen that raising the TiN/graphite ratio can boost the hardness of composite at the top surface. In another word, the TiN powder is more efficient than graphite component at raising the hardness of composite. This superior effect of TiN on hardness of composite can be explained through two main differences between TiN and graphite. Above all, the higher intrinsic hardness of TiN relative to graphite contributes to this phenomenon. Also, as it was discussed, the reinforcing component which can lock the grain boundaries through pinning effect is TiN nano powders, and increasing the TiN/graphite ratio escalates the efficiency of grain growth prevention.

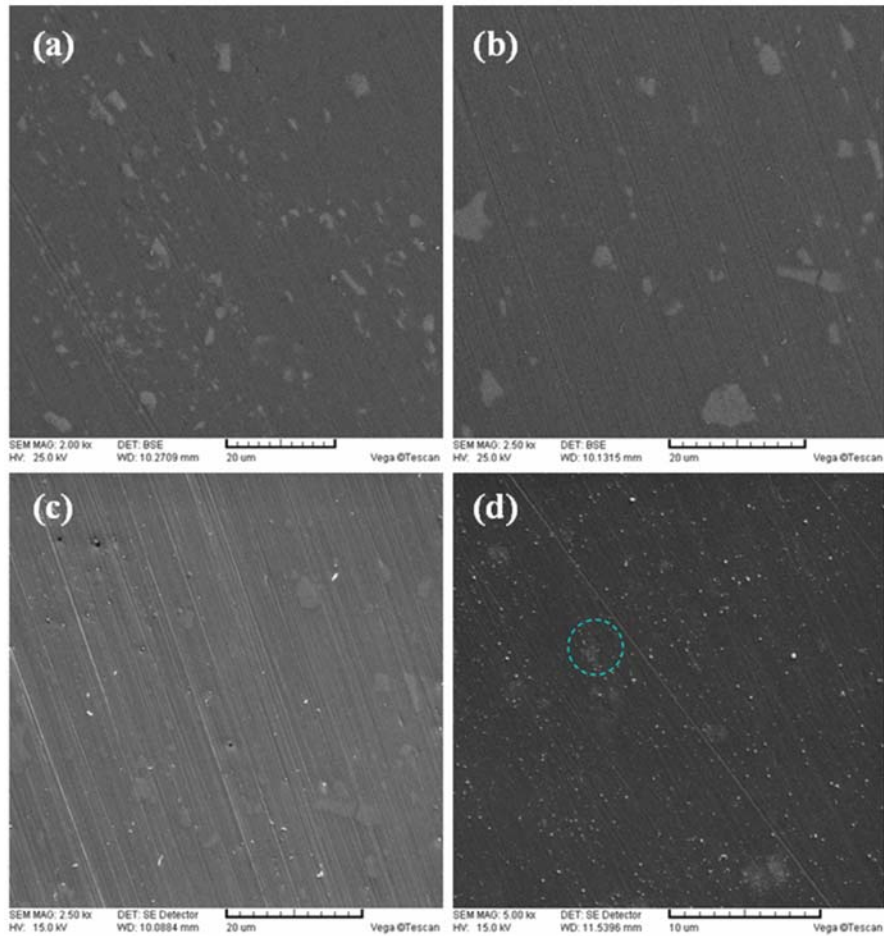


Figure 3. SEM images of various samples: (a) Sample No. 1 fabricated without TiN; (b) sample No. 2 containing 20% TiN; (c) sample No. 3 fabricated with 50% TiN; and (d) sample No. 5 produced without graphite. Although in all samples graphite is distributed uniformly, clustering of TiN powders is detectable in last case (without graphite).

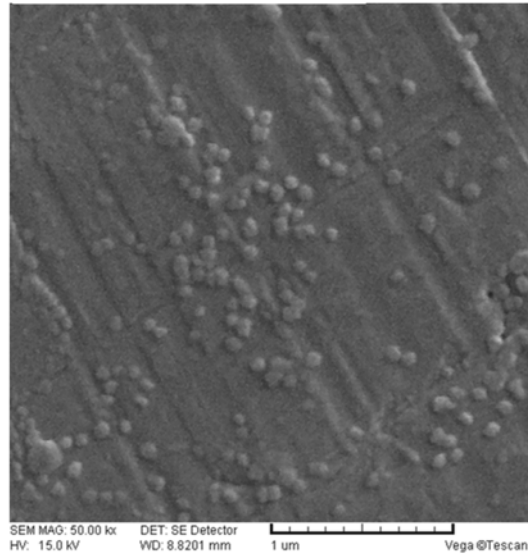


Figure 4. Close view of clustering in sample No. 5. The region is indicated in Figure 3 through dashed rectangle.

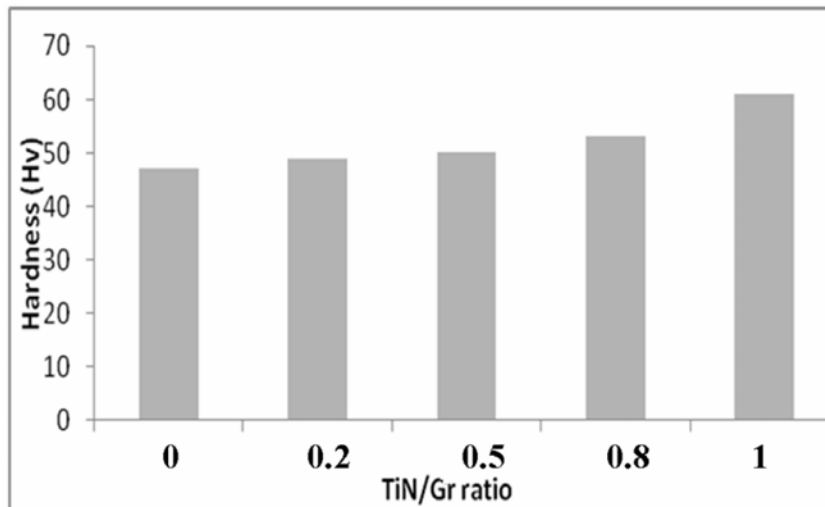


Figure 5. The average surface hardness of various studying samples with different ratio of TiN/graphite. The hardness rises by increasing the portion of TiN.

For evaluating the wear properties of fabricated samples, all of them were subjected to a pin-on-disk test. The weight loss of all samples after 1000m sliding is shown in Figure 6. The results obviously indicate that mixing the TiN and graphite powders as reinforcing components reduces the weight loss of composite. Such a reduction in weight loss of composite can be ascribed to combination of hardening effect of TiN and lubricating role of graphite powder as two essential factors for wear resistance in materials [1, 16]. For sample No. 1 fabricated without TiN powder, the lack of hardness results in a significant weight loss, and shortage of lubrication ends in a severe weight loss in composite No. 5 fabricated without graphite. Figure 7 depicting the worn surface of composites No. 1 and No. 2 confirms the constructive role of blending TiN and graphite in wear resistance of fabricated composite. As it is seen, by adding TiN to graphite-aluminum composite, the severe wear regime (Figure 7(a)) turns to a mild wear regime in which less surface damages are detectable (Figure 7(b)).

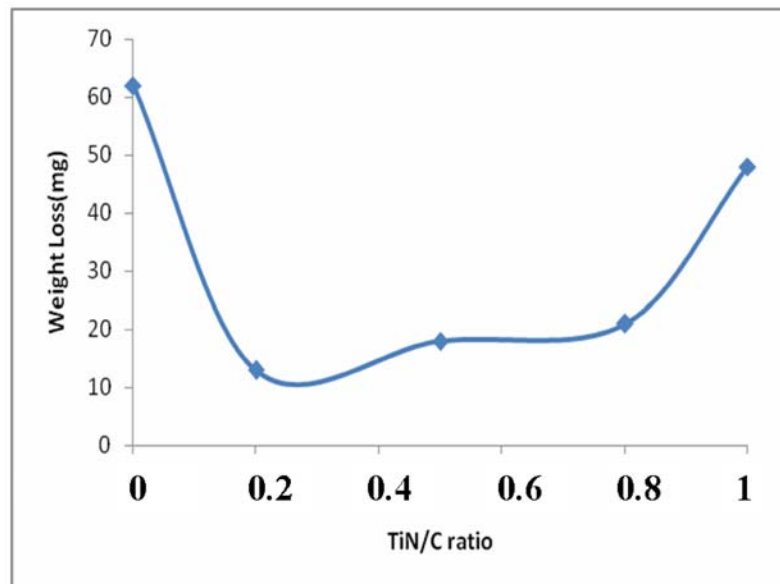


Figure 6. Weight loss of studying samples after a sliding of 1000m. The sample fabricated by 20% TiN shows the best result.

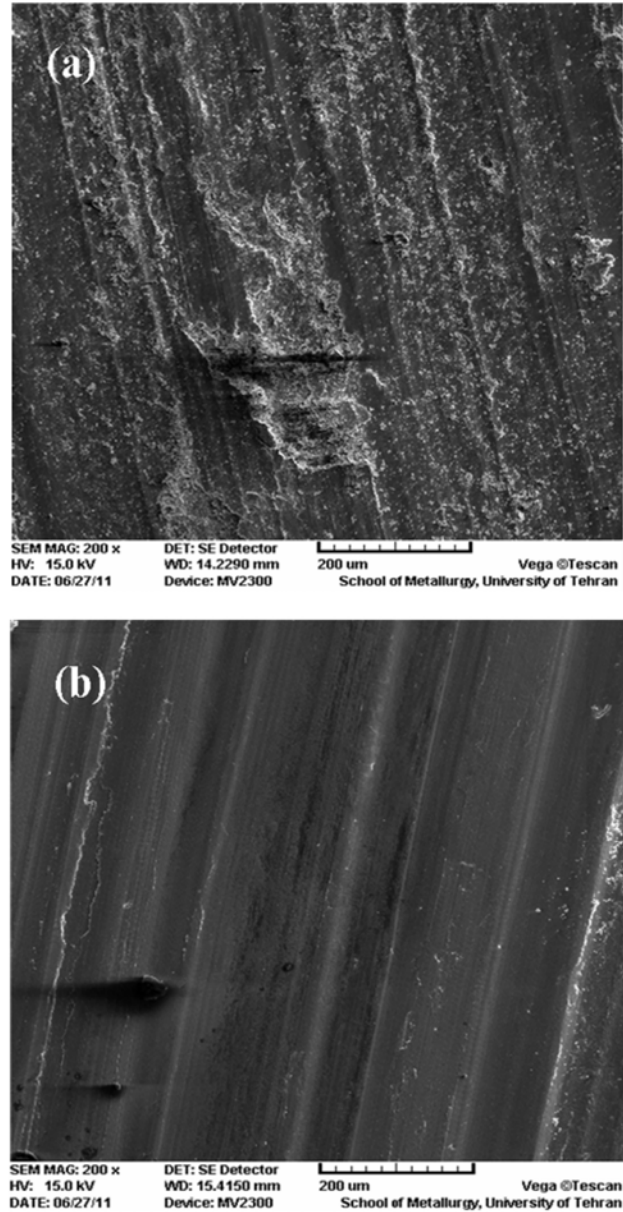


Figure 7. Worn surface of samples: (a) No. 1 fabricated without TiN; (b) No. 2 containing 80% graphite. In the former case, the severe wear regime is obvious, and the latter one shows a mild wear regime.

Figure 8 illustrates the measured friction coefficient of three different composites. This graph also suggests the influential role of mixing TiN and graphite together in reduction of friction coefficient of studying composite. Since the generated heat through friction is directly proportional to friction coefficient [23], the fabricated composite containing the mixture of TiN and graphite reduces the released heat of friction at the contact surface. This phenomenon along the lubricating role of graphite drops the temperature of contact surface. A critical temperature at which the mild wear regime transforms to a severe wear regime has been reported by Zhang et al. [20, 23]. This transition temperature can explain the observed reduction of weight loss by mixing TiN and graphite as reinforcing components. It appears that reducing the heat of friction through blending TiN and graphite powders together cools the contact surface below the transition temperature, and consequently, the mild wear regime occurs. If temperature of contact surface be considered as only dominant factor in wear behaviour of composite, then the graphite-aluminum composite must possess the least weight loss due to a superior lubrication. However, as results indicate, this composite has the highest level of weight loss among studying cases. It is shown that effect of hardness on wear rate of materials can not be ignored, where harder materials undergo milder wear. Thus, despite the fact that cooling the contact surface by adding graphite component can contribute to wear resistance, the hardness of composite must be also enhanced through addition of TiN nano powders. The microhardness profiles through the depth of samples indicate that adding the TiN ratio improves the hardness of composite at all areas (Figure 9). Also, it is seen that hardness distribution is not uniform through the composite, where the maximum value can be clearly detected at 2mm below the surface. This location showing the maximum hardness is independent of TiN ratio and does not shift in various studying samples. This feature implies that nonuniformity of hardness distribution can be explained by material flow

rather than TiN ratio. The upward flow of material in nugget zone besides the pressing force applied by shoulder accumulates the TiN powders underneath of surface [12], increasing the hardness of this region to a maximum value.

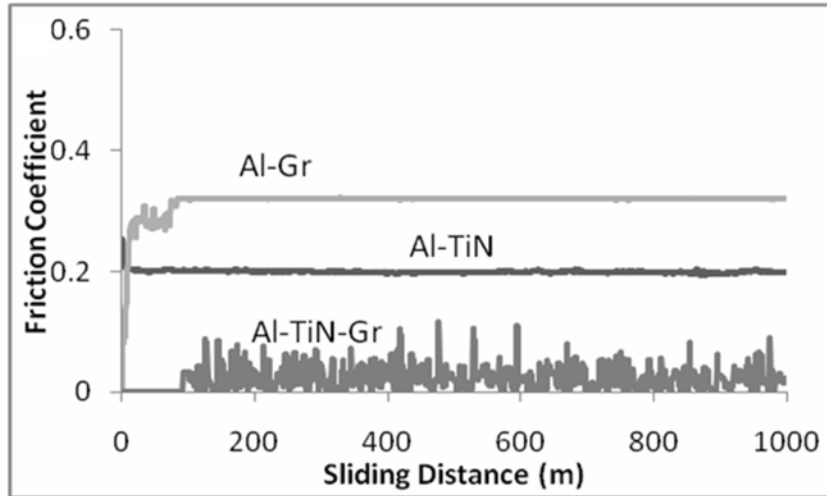


Figure 8. Measured friction coefficient through a sliding distance of 1000m. The composite containing 20% TiN shows the least friction coefficient.

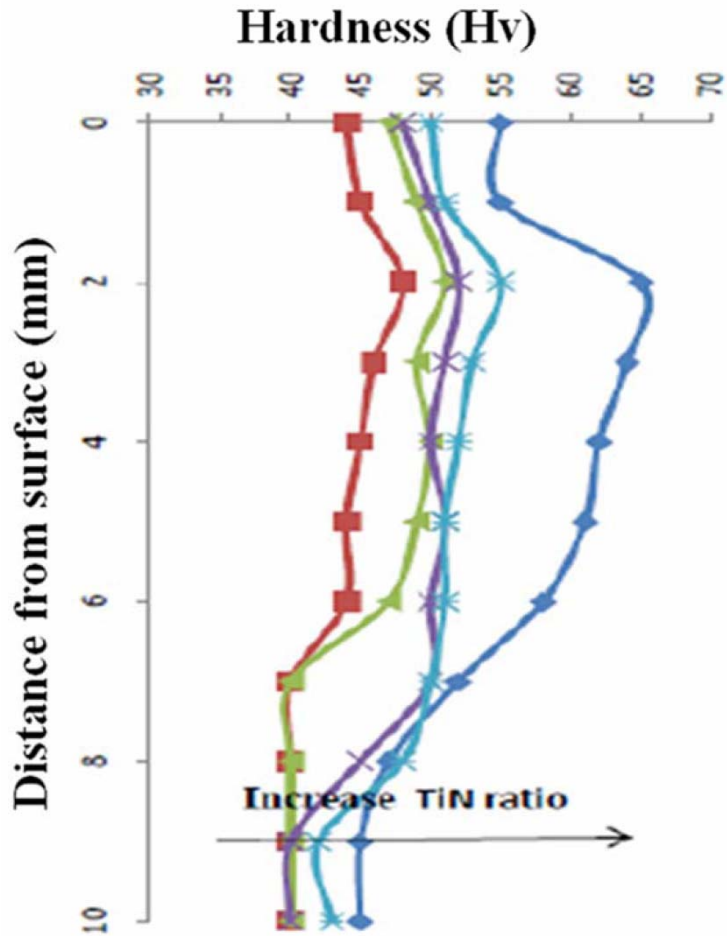


Figure 9. Distribution of hardness underneath of fabricated composites.

4. Conclusion

According to the obtained results, the tribological characteristics of aluminum-TiN-graphite composite fabricated by friction stir processing depend on ratio of TiN to graphite. It is also shown that coupling the graphite by TiN hinders the clustering of TiN nano powders and enhances the hardening effect of TiN particles. The carried out study reveals that mixing the TiN and graphite as reinforcing component improves the wear resistance of composite. The lubricating role of

graphite and the hardening effect of TiN powder are brought up as main reasons for observed features. The best wear resistance is obtained at TiN/graphite volume ratio of 0.2. Furthermore, it is indicated that hardness distribution through the fabricated composite is not uniform, and maximum hardness occurs at 2mm below the surface.

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