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## PAPER

# Microstructure and wear characteristics of 1050Al/Fe surface composites by friction stir processing

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## Abstract

In this study, friction stir processing (FSP) is used for improvement of 1050 Al surface with Fe powder (100  $\mu\text{m}$ ) as reinforcement. The surface layer composites of 1050 Al/Fe powder were produced in different conditions of FSP. Results of SEM and EDS technique indicated that FSP enhances the production and bonding of intermetallic products. Moreover, mechanical test results also demonstrated that FSP increased the microhardness, tensile strength and the wear resistance of specimens as a result of surface composite and fine-grained structure on the surface of 1050Al alloy. The influence of rotational speeds on wear resistance was studied in terms of friction coefficients and weight losses. SEM micrographs of the worn surfaces were also studied to evaluate the wear mechanisms. Increasing the rotational speed to 1600 rpm was caused increasing of the hardness and wear resistance, simultaneously. Adhesive wear mechanism was decreased on the worn surface of F2-1600 samples due to uniform distribution of reinforcement particles during FSP. General investigations showed that the specimens in 1600 rpm condition have acceptable surface layer composite due to better wear resistant and mechanical properties.

## 1. Introduction

Today, various industries require materials with unique properties to better performance. In this regard, aluminum alloy is considered to be one of the most applicable. Because of high strength-to-weight ratio, these materials are widely used in aerospace and automobile industries [1]. In more industries, because of poor mechanical and surface properties, application of pure Al group (1XXX) is limited. On the other hand, surface property is important part of materials. Therefore, more researchers focused on surface modification of Al alloys by different methods [1, 2].

Friction stir processing (FSP) is one of new techniques for surface modification. FSP is a kind of friction stir welding, and it has the ability to improve the surface microstructure and produce the surface composite.

Studies show that produced composite layer on surface by FSP could be effective technique to improvement properties of Al alloys [1, 2]. FSP is a solid state process. It means that, without melting, simply by the process of warming and high deformation lead to severe changes on surface of samples. This process involves a non-consumable rotating tool which is slowly entered to the work piece by the machine and after sufficient heat generated by friction, it starts to move along the desired direction [2–4].

FSP is used in the space industries, automotive, marine industries, railways and etc in the United States of America [3]. FSP is a technique for fabrication of aluminum matrix composites that exhibit good wear resistance and high hardness. According to Mishra *et al* [2] aluminum composite with reinforcement particles exhibit high strength, and improved resistance to wear, creep and fatigue, thereby making them suitable for various industries. Wang *et al* [4] carried out FSP on the surface of Al with SiC particles and studied the mechanical properties. In another work, Marzoli *et al* [5] reinforced AA6061 with  $\text{Al}_2\text{O}_3$  particles via FSP and fabricated a good surface composite. You *et al* [6] produced an Al–CuO composite using FSP. In different aluminum

**Table 1.** The chemical composition of 1050 Al alloy.

Element	Al	Si	Fe	Bi	Cu	Mn
Percent%	99	0.04	0.12	0.1	0.005	0.001>

**Table 2.** The physical properties of iron (Fe) powder.

Density (gr/cm <sup>3</sup> )	Morphology	Color	Grain size ( $\mu$ m)
7/874	spherical	gray	100

composite, aluminum/Fe composite has special properties. The second phase is the main reason of latter properties. FeAl and Fe<sub>3</sub>Al are the intermetallic compounds of iron and aluminum. These compounds have various properties include high melting point, high hardness, low density and good resistance to corrosion and oxidation [7]. This composite have been produced by different methods include rapid solidification [8], mechanical alloying [9] and sever plastic deformation [10]. But the main problem of this method is limited size of final product [11]. FSP is a severe plastic deformation (SPD) technique used to produce *in situ* composite with different size. For example, Lee *et al* [11] were able to fabricate Al<sub>3</sub>Ti and Al<sub>13</sub>Fe<sub>4</sub> compounds in Al-Ti and Al-Fe mixed powder by FSP. They reported that *in situ* Al-Fe and Al-Ti reaction can be induced during FSP [11]. However, no comprehensive and systematic research has been done about production of *in situ* Al-Fe composite in bulk aluminum by FSP. For example, few researchers studied the mechanism of formation and wear resistance of this composite. Hence, the present investigation is an attempt to assess the effect of the rotational speed, on the mechanism of formation and wear resistance of *in situ* Al-Fe composite.

In this paper, commercially Al 1050 and Fe powder were used as the matrix and reinforcing particles, respectively. To decrease agglomeration of particles, two pass FSP was used on the Al sheet. Then microstructure and mechanical properties such as hardness, tensile strength and wear resistant of the surface composite were evaluated.

## 2. Experiments

In this study, Al 1050 plate was used as the base metal. The chemical composition of base metal is listed in table 1. The dimension of each sample was considered 200 × 60 × 6 mm<sup>3</sup> length, width and thickness respectively.

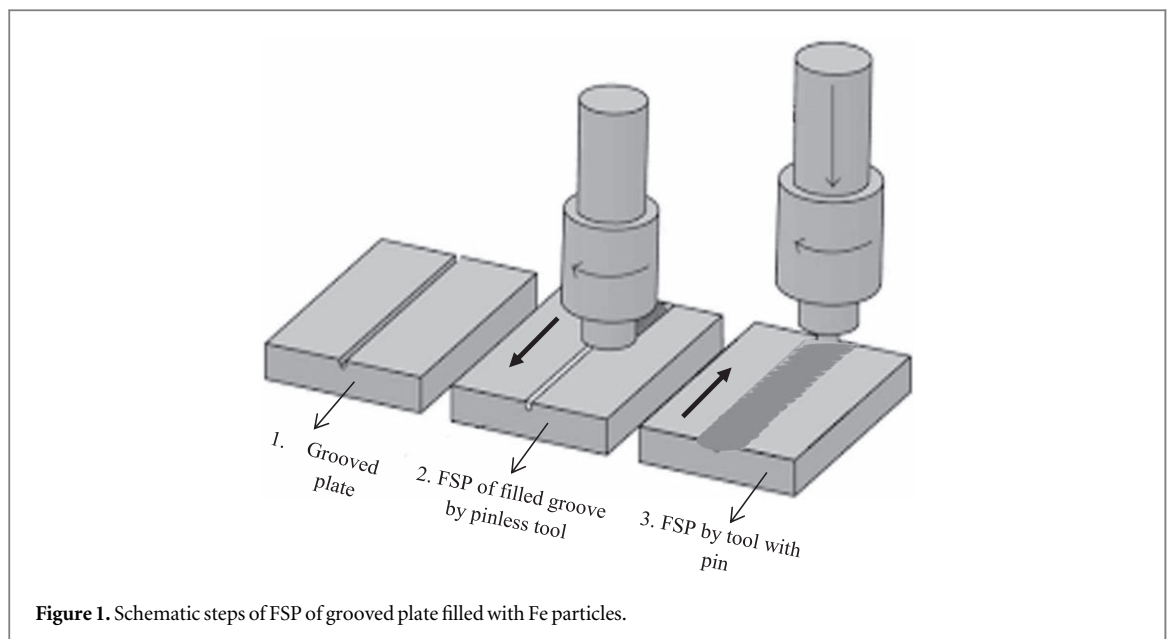
Pure iron (Fe) powder is also used as a reinforcing phase. Table 2 shows the physical properties of the powder. In this research, amount of Fe powder was 6%Vol of base metal. Before FSP, in order to produce the surface composite, the surface of plates was grooved. The width and depth of the groove were 2 mm and 3 mm respectively. The FSP was started by closing the groove using a pinless tool in order to prevent the particles from being scattered during FSP. The second stage was done by a H13 steel tool with the shoulder diameter of 20 mm and the cylindrical pin of 4 mm in diameter and 4.5 mm in height. Figure 1 illustrates schematic of FSP of the grooved plate. Figure 2 shows two tools (pinless tool and tool with pin) that were used in this study. The tool was tilted 3° from the plate normal direction during FSP. FSP was performed by a vertical milling machine. To improve the surface composite of Al plates, different condition of FSP was selected. According this, three rotational speeds include 1000 rpm, 1600 rpm and 2000 rpm were selected. For All specimens, FSP was applied in constant linear velocity of 50 mm min<sup>-1</sup> and two passes. The specification of specimens are given in table 3.

Scanning Electron Microscopy (SEM, Cambridge S360 model) and EDS analysis were used for microstructural study. Transverse tensile samples (loaded perpendicular to the FSP direction) were tested by Santam STA 150 machine. Vickers hardness was measured using a tester with 50 000 mg load and the dwell time of 15 s in points with 1 mm distance from the upper surface in the cross section and at the surface of the composite. A pin-on-disk apparatus was employed for the wear test under the load of 10 N, rotational pin speed of 50 mm min<sup>-1</sup>, traversal radius of 10 mm, and the total distance of 200 mm.

## 3. Results and discussion

### 3.1. Microstructure

The distribution of the Fe particles in nugget zone (NZ) is shown in figure 3. As be seen, the Fe particles are distributed in NZ. In addition, since the rotational speed of tool has a direct relation with intensity of deformation [1, 2], so that increasing the rotational speed to 1600 rpm and 2000 rpm would reduce the agglomeration of Fe particles. The SEM micrographs in figure 4, been focused on morphology of one Fe particle.



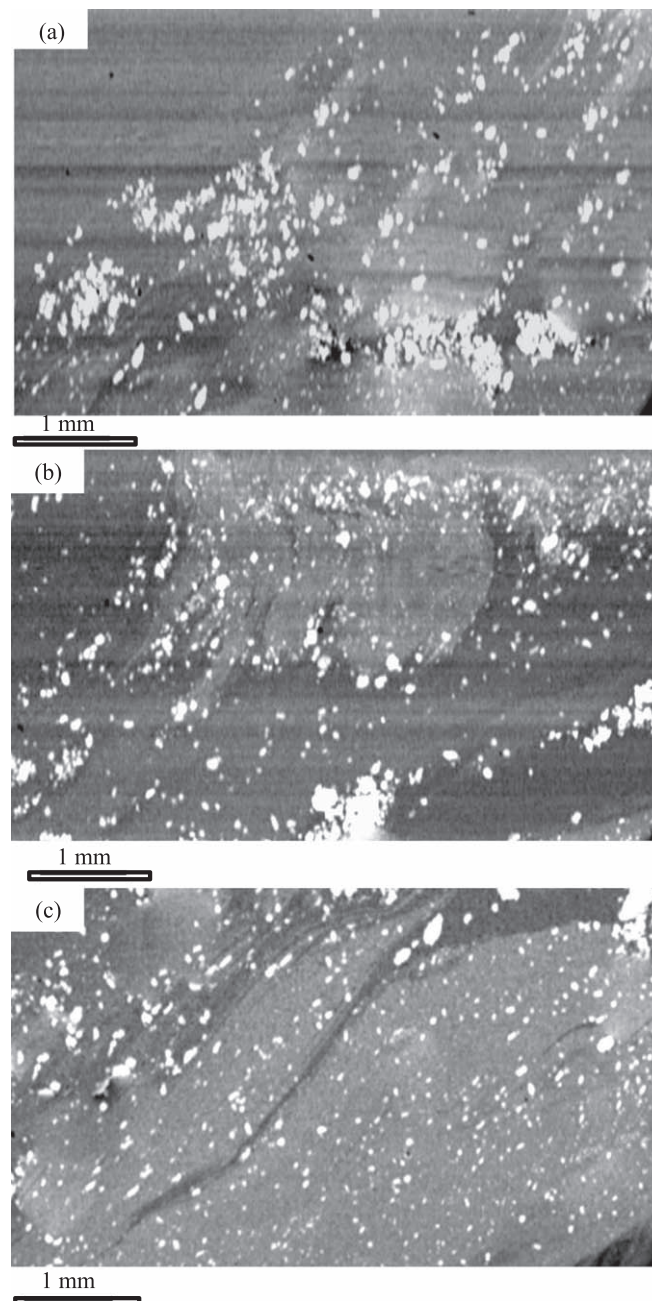
**Figure 1.** Schematic steps of FSP of grooved plate filled with Fe particles.



**Figure 2.** Tools of FSP (a) pinless tool (b) tool with pin.

**Table 3.** Specification of specimens.

Samples	Rotational speed (rpm)	Linear velocity (mm/min)	Number of pass	% Fe Particles (%Vol)
F2-1000	1000	50	2	6
F2-1600	1600	50	2	6
F2-2000	2000	50	2	6

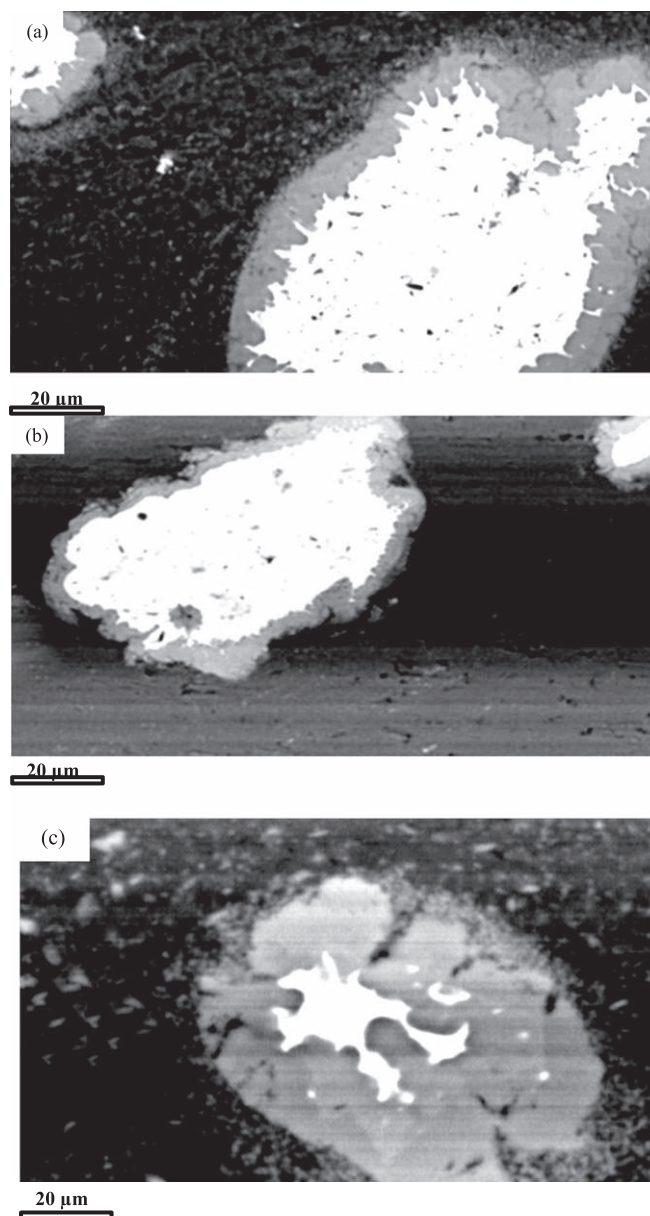


**Figure 3.** SEM images of distribution particles in different FSP conditions (a) F2-1000 (b) F2-1600 (c) F2-2000 specimens.

As shown in figure 4, each Fe particles are surrounded by distinct layer and new interface is created. The EDS analysis was applied to accurate investigation of this phenomenon. For this purpose, three areas were selected to EDS analysis as shown in figure 5. The results of EDS analysis are listed in figure 6. As can be seen, that the composition of A point is related to Fe powder. Chemical composition of B point shows that a new phase is presented in this area which contains iron and aluminum. The atomic percentage of elements displayed that the ratio of the atomic aluminum to iron is more than three times and it is consisted with the intermetallic compounds of  $\text{Al}_{13}\text{Fe}_4$ . It was revealed that the condition is provided for the formation of intermetallic compound. So, the Fe particles reacted with Al matrix and a new phase was formed between particles and Al matrix during the FSP. Increasing temperature and mechanical alloying during FSP [1] have been the main reason of this reaction. Similar results were obtained by Lee [11] and Adam [12] about Fe-Al composites.

Moreover, close comparison of figures 4(a)–(c) demonstrated that thickness of intermetallic compound were enhanced by increasing rotational speed of FSP. The increasing temperature with high rotational speed is the main reason of this result [11]. Therefore, rotational speed is effective parameter on morphology of intermetallic compound.





**Figure 4.** SEM images of Fe particles in the FSPed Al composite (a) F2-1000 (b) F2-1600 and (c) F2-2000 specimens.

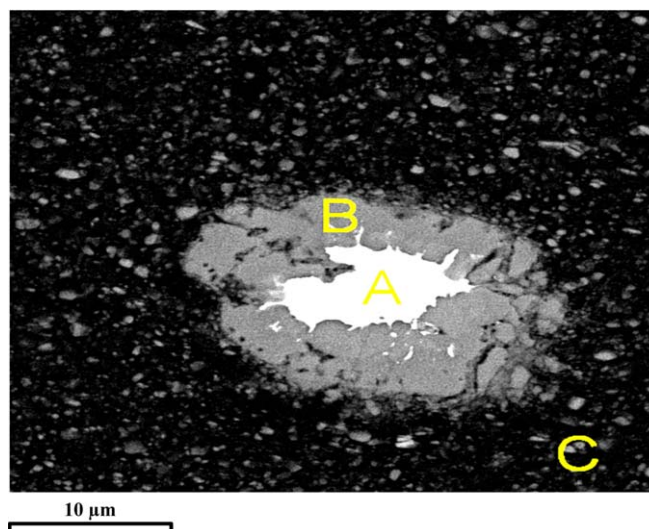
The F2-1600 specimen was selected for linear scan EDS analysis. The results of linear EDS are shown in figure 7. The results reapproved the formation of new phase between base metal and reinforcement particles. The atomic percentage of elements displayed that this combination is  $\text{Al}_{13}\text{Fe}_4$  intermetallic. From the literatures [13–17],  $\text{Al}_5\text{Fe}_2$  is the main intermetallic compound in Al-Fe composite by different solid state techniques. For example, lee *et al* claimed that  $\text{Al}_{13}\text{Fe}_4$  is the first phase in the reaction between Al and Fe. In the following, this phase reacted with Fe particle and  $\text{Al}_5\text{Fe}_2$  phase is formed [13]. However, due to the low temperature in short time during FSP, in our specimens no  $\text{Al}_5\text{Fe}_2$  compound was observed.

From the detailed observation of the microstructure of the surfaces, it may be concluded that the interface between Fe particles and Al matrix produced by two FSP passes in all rotational speeds. It seems that the use of iron nanoparticles can bring surprising results that will be addressed in future research.

### 3.2. Hardness

Figure 8 shows the microhardness results of Al/Fe composite surface. The microhardness result shows that hardness value in NZ is much higher than base metal. The distribution of Fe particles, formation of  $\text{Al}_{13}\text{Fe}_4$  phase and grain size refinement of Al are the main reasons of this result. The same result was reported in some research [13, 18, 19].

Advancing side (AS) and retreating side (RS) show similar distribution of Fe particles. So, the microhardness of AS is equal to RS. Similar to Barmouz *et al* [20]. Figure 8 does not show a Heat affected zone (HAZ) in these



**Figure 5.** SEM image of the selected particle for the EDS analysis after two passes FSP (F2-1600).

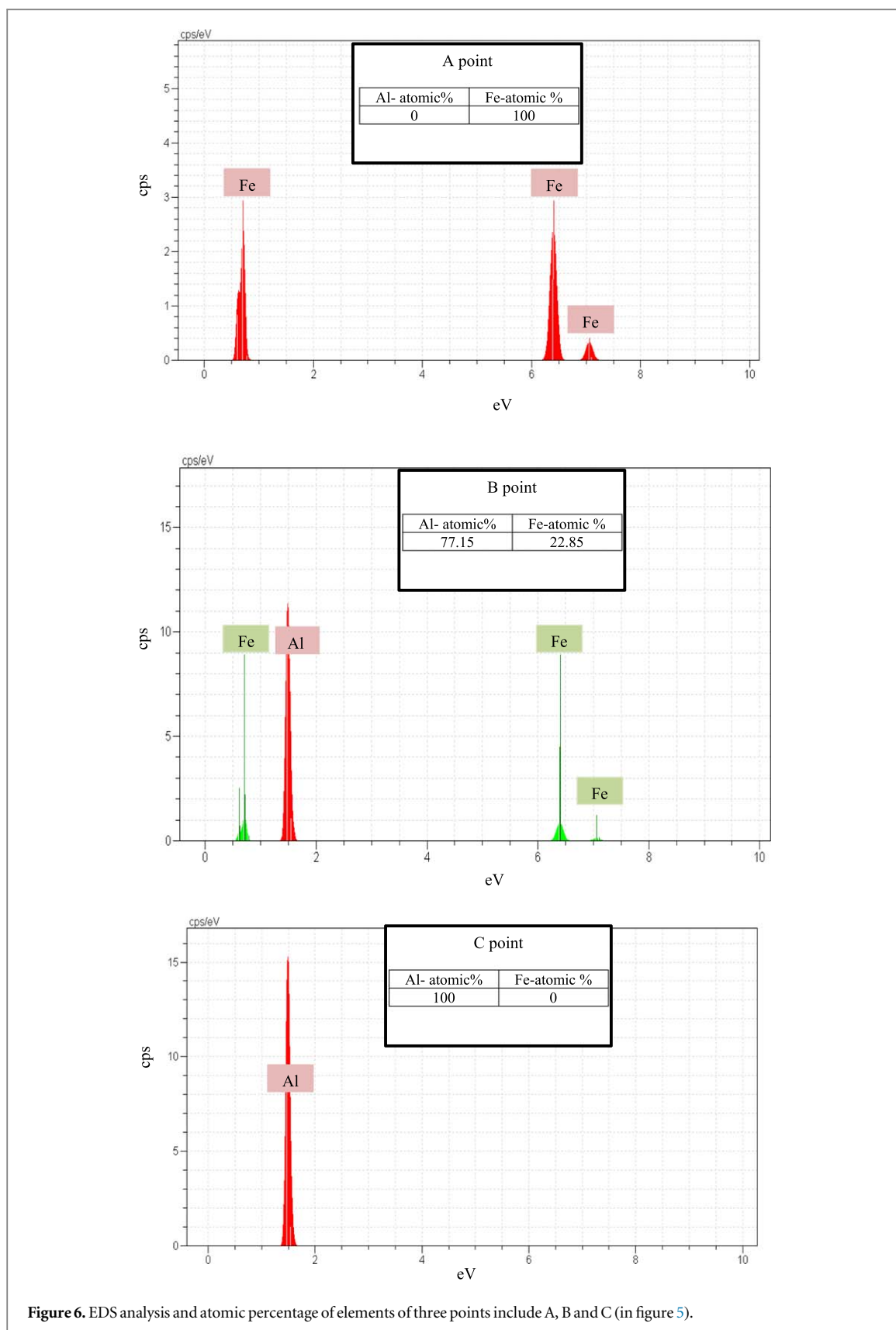
specimens. Why so, in the HAZ regions, the hardness usually lower than base metal but in the FSP technique due to specially conditions sometimes this region couldn't formation[1, 2].

Also, as it can be seen that the average hardness value in NZ of F2-1000 specimen is lower than F2-1600 and F2-2000 samples. This is because of the inadequate distribution of the reinforcement particles. By rotational speed increasing, the agglomeration of the particles was reduced in NZ and more uniform distribution would be occurred. Furthermore, the better conditions are provided to form intermetallic compounds by increasing temperature during high rotational speed. The same results were reported about SiC particles in copper matrix [18]. In the other hand, the reason of difference in the hardness can be attributed to the increasing of the size of the crystallized grains in the produced samples in rotational speed of 2000 rpm [21]. In general, the higher rotational speeds of the tool and the more friction with the substrate, were caused to increase of temperature [2]. In the same linear velocity, the sample subjected to 1600 rpm has lower temperature and smaller coaxial recrystallized grain. So, the higher hardness is produced in specimens with the 1600 rpm condition.

### 3.3. Wear test

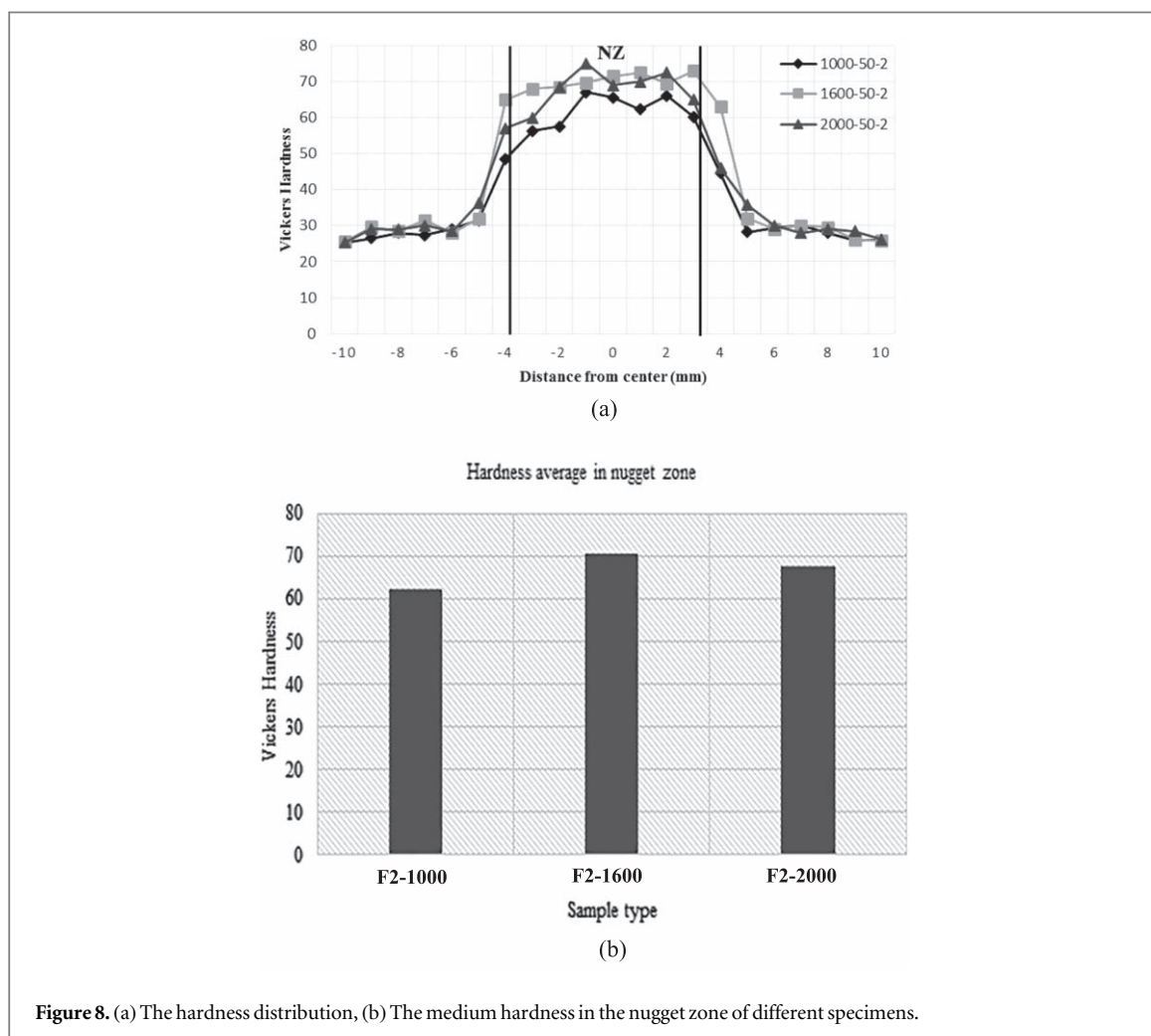
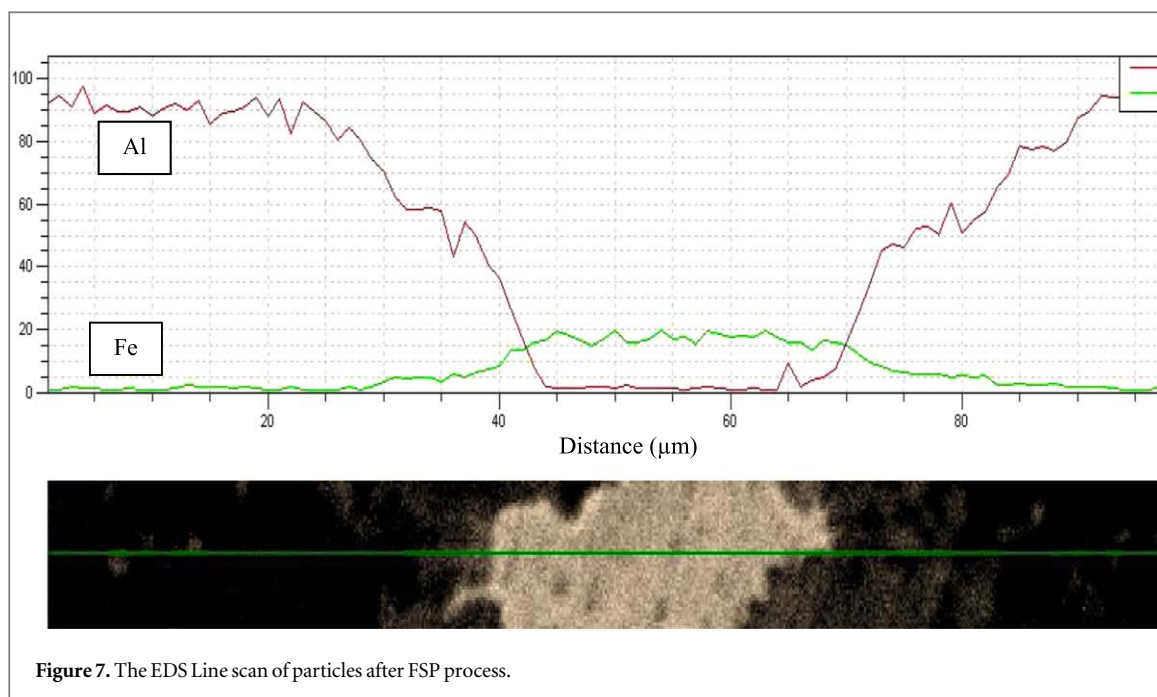
The results of wear test of all samples shown in table 4. According to this, the results of wear resistance of the samples are investigated. The lost weight during abrasion test and the average of friction coefficient were measured.

The weight loss and friction coefficient in the F2-1600 and F2-2000 specimens are lower than F2-1000 sample. This can be attributed to the higher hardness of these specimens and the decrease in contact between the specimen surface and tool due to the presence of Fe particles. Similarly, Wang *et al* [4] reported good wear properties in Al-SiC specimens produced with FSP. As regards to constant linear velocity, increasing the rotational speed was the main factor to improve wear properties of FSPed specimens due to better distribution of Fe particles. With regard to the distribution of Fe particles on the surface of the composite in F2-1600 and F2-2000, in compare to F2-1000, better wear properties are obtained. The average of the friction coefficient for different specimens is listed in table 4. The specimen with lower friction coefficient has low weight loss. The same results are reported by Izadi [22] and salehi [23] about Al-SiC composite by FSP. The high friction coefficient indirectly suggests that the mechanism of wear is predominantly adhesive in nature. The localized welding of the worn debris is the main reason of this phenomenon [24]. On the other hand, the friction coefficient was found to decrease to low value during the wear of F2-1600 specimen. The decreased friction of coefficient is attributed to decreasing localized welding of the worn debris. The uniform distribution of reinforcement Fe particles lead to decrease contact between pin and specimen surfaces during wear test. A detailed study of the SEM micrograph of the worn surfaces of all specimens were undertaken to understand the mechanism of wear. Figure 9 shows worn surface of F2-1000, F2-1600 and F2-2000 specimens. The F2-1000 specimen has deep grooves and delamination that implies the mechanism of wear is predominantly adhesive. Unlike F2-1000 sample, F2-1600 specimen has shallow groove that indicate the mechanism of wear is abrasive. Close comparison of micrographs illustrate that both abrasive and adhesive mechanisms are visible in F2-2000 specimen. From the detailed observation of friction coefficient and the micrograph of the worn surfaces, it may be concluded that the improved wear resistance of surface composite layer produced in F2-1600 as compared to



**Figure 6.** EDS analysis and atomic percentage of elements of three points include A, B and C (in figure 5).

that of F2-1000 and F2-2000 is attributed to increased hardness. The rate of wear increased in F2-1000 mainly because of increased coefficient of friction. On the other hand, in surface of F2-1600 specimen due to a comparatively lower coefficient of friction, adhesive wear decreased. Furthermore, the adhesive mode of wear is lower in the surface composite layer in F2-1600 specially.



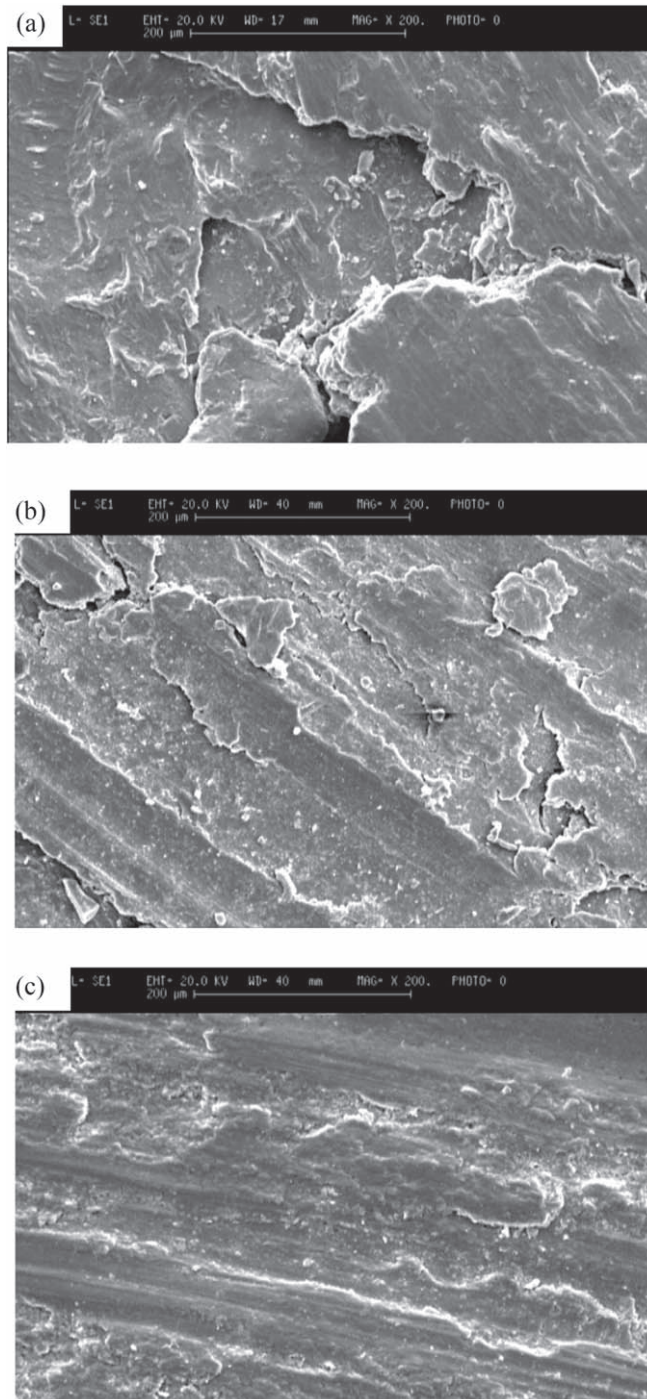
### 3.4. Tensile test

In table 5 the ultimate strength and the percentage of elongation is shown. According to the Hall-Pitch relation, by reducing the grain size, the amount of strength (UTS) of the produced composite is improved. On the other



**Table 4.** The lost weight and the coefficient of composite specimens.

Sample	F2-1000	F2-1600	F2-2000
Weight loss (gr)	0.0041	0.0032	0.0033
Average friction coefficient	0.43	0.36	0.39

**Figure 9.** SEM micrograph of worn surfaces (a) F2-1000 b) F2-1600 and (c) F2-2000 specimens.

hand, the distribution of Fe particles in soft Al matrix leads to increases the UTS of specimens. The F2-1600 specimen has produced more strength in compare to the F2-1000 produced sample. Therefore, the homogeneous distribution of Fe particles leads to increase of UTS of specimens. Some research are reported the same results [19]. The UTS of F2-2000 specimen was lower than F2-1600 due to grain growth.

**Table 5.** The ultimate strength and elongation of composite specimens.

Sample	F2-1000	F2-1600	F2-2000
UTS (MPa)	87.61	103.06	99.59
Elongation%	26.72	20.15	23.08

Table 5 shows the elongation results of all specimens. As it can be seen, that the elongation of samples decreased by increasing the UTS of specimens. In the other words, presence of Fe particles impedes the movement of the boundaries and reduces the amount of elongation.

## 4. Conclusion

In this work, the effect of Fe particles on the mechanical properties and the microstructure of Al matrix composite fabricated via two pass FSP were investigated in different rotational speeds include 1000, 1600 and 2000 rpm. The most important results are as follows:

- Conducting the FSP in the two pass prevented to pores formation.
- Distribution of Fe particles was improved with increasing rotational speed of FSP from 1000 to 1600 rpm.
- The intermetallic compounds were observed in FSPed specimens because of the high deformation and temperature during FSP process. In the other word, the Fe particles interaction with Al matrix and intermetallic interface was formed.
- Increasing rotational speed to 1600 rpm resulted in a higher hardness in NZ.
- The adhesive mode of wear is decreased in surface of F2-1600 specimen, because of uniform distribution of Fe particles by FSP.

More work is needed to optimize the processing parameters of FSP such as tool's profile and linear speed, in order to further improve the physical and mechanical properties of this composite.

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