

Investigation of Mechanical Property and Microstructure of Nanocomposite AZ31/SiC Fabricated by Friction Stir Process

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Abstract: The friction stir process (FSP) is a solid state process, which has been used to insert reinforcing particles into the structure of a material to create a composite with improved properties. Magnesium is a light structural metal that is increasingly used in the aerospace and automobile industries. In this research, SiC nanoparticles were added to AZ31 alloy using FSP in two overlaps of 100% and 50% passes. In 100% pass overlapping, nanoparticles were added in 4, 8 and 16 volume percentages and in 50% pass overlapping only nanoparticles in 4 volume percent were added. The FSP process performed as 4 consecutive passes in both overlaps along with rapid cooling. Microstructure, hardness and tensile strength of created composites were examined. The results suggested that adding reinforcing materials causes reduction in the size of the grains, uniformity of structure and increase in the hardness of material. SiC nanoparticles distributed uniformly through the AZ31 alloy. By increasing volume fraction of reinforcing materials, yield stress of the material increased but ultimate stress and formability properties reduced. In 50% overlapping state, the yield stress in directions, either parallel or perpendicular to the pin direction, increased rather than 100% overlapping state, but the ultimate stress and elongation properties reduced. This reduction was greater in the perpendicular direction relative to the pin direction.

Keywords: Friction Stir Process, Magnesium AZ31, Mechanical Strength, SiC Nanoparticles

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1 INTRODUCTION

Increasing material strength using changes in microstructure or adding reinforcing materials to their structure is one of the issues that researchers are working on it. Magnesium is the lightest structural metal that is taken particularly into consideration in various industries including automotive industry due to its low density and high specific strength [1]. For this reason, the researchers are attempting to increase its mechanical strength. One of the processes that is used to increase metal strength is friction stir process. This process is a solid state process which is used to change microstructure of materials. In this process a spinning tool which is composed of a shoulder and a pin, enters into the specimen and moves in an intended direction with constant traverse and rotational speeds. Simultaneously with traverse and rotational speeds, materials are being transmitted from the front of the pin to its back and they will be placed under extrusion and forging processes with high strain and strain rate along with dynamic recrystallization as an effect of this movement. The results provided by performing friction stir process on different metals including Aluminium and magnesium indicate that the process causes change in mechanical properties and subsequently it increases or decreases ultimate stress, yield stress and material formability and this is because of changes in material microstructure [2-6]. Regarding successful implementation of friction stir process on metals, researchers have lately focused their attention on using this process for adding reinforcing materials to the material structure and reviewing its mechanical properties. In the following the studies that have accomplished on Aluminium and magnesium in this field will be addressed.

Dolatkhah et al., [7] added two kinds of SiC sized 50 nm and 5 micrometers to Aluminum 5052 using friction stir process. They observed the size of Aluminum grains decreases and hardness and resistance to wear increases by decreasing the size of SiC particles and increasing the number of passes. Salehi et al., [8] created and reported composite Aluminum base 6061 with 50 nm particles of SiC in which increasing rotational speed and decreasing traverse speed is more appropriate for creating composite and in addition, threaded pin is better than square pin for creating composites. Choy et al., [9] added SiC particles to Aluminum 6061-T4 and reached a uniform distribution of SiC particles in Aluminum matrix. Another study also performed on adding nanoparticles Al_2O_3 [10], [11], composition B4C and tic [12] on Aluminum in which resistance to wear and microstructure was studied. Few studies were performed in the field of adding reinforcing materials to the magnesium that they will be referred in the

following. Morisada et al., [13] added SiC particles sized 1 micrometer to the alloy AZ31. They reported that during FSP process, SiC particles increase hardness and decrease grain size. Najafi et al., [14] used SiC particles sized 4 micrometers to create composite with alloy matrix AZ31. It is noted in their report that simultaneous use of SiC particles and cooling during FSP process causes the size of magnesium grains to decrease significantly and hardness to increase right after recrystallization. Asadi et al., [15] added SiC particles sized 5 micrometer to the alloy AZ91 and like other researchers they observed that these particles affect the reduction of size of grains and increase the hardness. Asadi et al., [16] used particles Al_2O_3 and SiC sized 30 nm in alloy AZ91 in another study and reported that SiC particles produce more hardness and mechanical strength rather than Al_2O_3 particles. They linked its reason to more tendency of Al_2O_3 particles to agglomeration. San et al., [17] used 40 nm particles of SiC for creating composite with alloy matrix AZ63. Their results illustrate that SiC particles cause increase in hardness, ultimate stress and material formability towards base material but elongation in two sample cases of FSP does not change with and without powder. Lou et al., [18] used combination of Al_2O_3 particles and carbon nanotubes (CNTs) with different percentages of both types of reinforcements in alloy AZ31 and they studied hardness and wear. In the field of using other reinforcing phases, study of Jiang et al., [19] can be referred in which they examined the effect of SiO_2 nanoparticles in alloy AZ31 on microstructure and hardness. After the process of friction stir of one pass, an area sized 10 to 14 mm will be affected using pin with a diameter of 8 mm and turns to fine-grained, this little area may not be proper for engineering applications [20]. Another method that is recently used is the use of overlap passes. In this method the fine-grained area of each pass overlaps the fine-grained area of next pass to a certain extent until the desired surface is created with arbitrary width. Overlap passes could be implemented using advance or retreating area. Gandra et al., [21] studied the effect of each of these overlaps over the microstructure and bending strength of Aluminum 5083 and found that overlap with advance side creates more uniform layer and overlap with retreating side creates a layer with higher strength but both had the same hardness distribution. The matter that should be taken under consideration in overlap passes is the way next pass begins. Does next pass have to begin right after the end of the pass or we should allow the temperature of specimen to be reduced down to room temperature and after that next pass be started? This subject was studied on the Aluminum 5086 in the research of Ramesh et al., [4]. They made overlaps in two cases. In first overlap, next pass starts right after

the end of each pass while in second overlap after the end of pass, the specimen is allowed to align its temperature with ambient temperature and after that next pass starts. The mechanical properties decrease in direction of the process implementation toward a case of one pass and even a raw material and they have far weaker results in the perpendicular direction to the process implementation. But in general the results of the case in which the sample is allowed to reduce its temperature down to room temperature and after that next pass starts was reported better. The only report on the effect of overlapping passes on the magnesium alloy AZ31 is provided by Venkateswarlu et al., [5]. The effect of overlapping with different percentages of 0, 50 and 100% is studied in this research.

As it is observed in the literature review, the researches performed on adding reinforcing particles to alloy AZ31 in 100% overlapping and specifically in 50% overlapping state for producing surface and examining the effect of these reinforcing particles on the mechanical properties of created composite are extremely low and this study was performed for this reason. Rapid cooling in fabrication of nano composite with FSP was not reported in other researches that is applied in this study. In this study, the reinforcing particles SiC are added to alloy AZ31 in three different volume percentages of 4, 8 and 16 using friction stir process in 100% overlapping state. Also for producing a big surface, SiC nanoparticles were added to the alloy AZ31 with 4% volume percent by 50% overlapping state. Regarding this matter that for creating a uniform distribution of reinforcing particles in base metal traverse speed should be low and rotational speed should be high, fast cooling were used during the process to prevent excessive increase of temperature in the piece. Microstructure, hardness and tensile strength is studied that we address to them in the following.

2 EXPERIMENTAL WORKS

Magnesium alloy AZ31 used in this research has percentages of elements in accordance with Table 1. Magnesium pieces were cut and prepared with 100×100 mm dimension with a thickness of 10 mm. SiC particles that were used with average size of 50 nm, are shown in Fig. 1-a. Used die during FSP process is shown in Fig. 1-b. This die contains a copper part (due to fast heat transfer between cooler material and workpiece) that AZ31 specimen is placed on it and cooling liquid passes through grooves which is embedded in the cooper. Heat transfer from the bottom of the pin to rear panel is the first stage of heat loss created in FSP process [22]. That is why this kind of cooling was used. Water at 10°C with 1.5 liter per minute of flow rate was used for cooling.

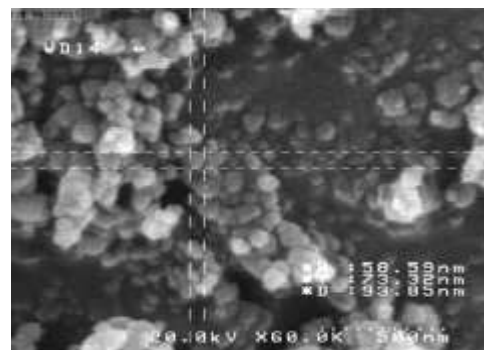
Table 1 The percentage of AZ31 constituent elements

Constituent elements	Percentage
Mg	95.5
Al	2.82
S	0.056
Cl	0.18
K	0.041
Ca	0.023
Mn	0.42
Fe	0.011
Cu	0.0045
Zn	0.94

The sizes of diameter of shoulder, pin, and pin length are respectively equal to 18, 7, and 4 mm and the thread pitch of pin is considered as clockwise. The process was accomplished with rotational speed and traverse speed of 1000 rpm and 28 mm/min in 4 consecutive passes, respectively. 100 and 50 percent overlaps were implemented according to Eq. (1) [21], [23]:

$$OR = l - \left[\frac{l}{d_{pin}} \right] \tag{1}$$

Where l is the distance between the centers of two consecutive passes, d_{pin} is the pin diameter, and OR is the overlap ratio.



a)



b)

Fig. 1 a) SiC particles, b) used die in FSP process

Reinforcing materials were added to AZ31 in 100% pass overlapping in three 4, 8 and 16 percent of volume percent. For this purpose, grooves were added on the surface of piece with width of 1 mm and depth of 1, 2 and 4. For 50% overlapping state in 4% of volume percentage, SiC nanoparticles were created in grooves according to Fig. 2. In which the distance between center and the center of these grooves is equal to pin diameter. The grooves were filled with the reinforcing phase and the surface was sealed using an instrument without a pin to avoid forcing the reinforcing materials from the grooves. FSP was carried out using an instrument with a pin in 4 consecutive passes with 100% overlap in conjunction with cooling.

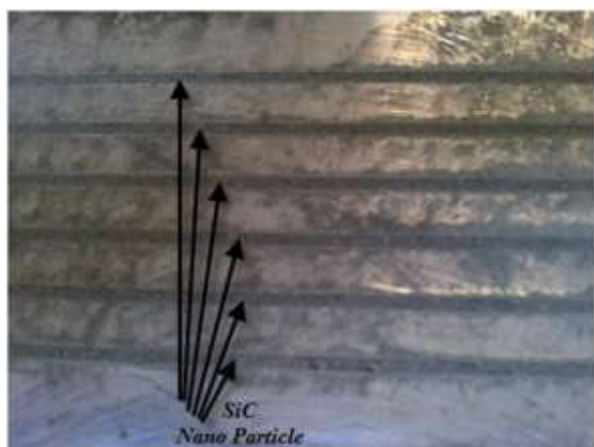


Fig. 2 Importing SiC nanoparticles into the grooves created in 50% overlapping state

According to Fig. 3 tension samples were separated in parallel and perpendicular to process direction in 50% overlapping state and only in parallel of process direction in 100% overlapping state using Electro Discharge Machine (EDM).

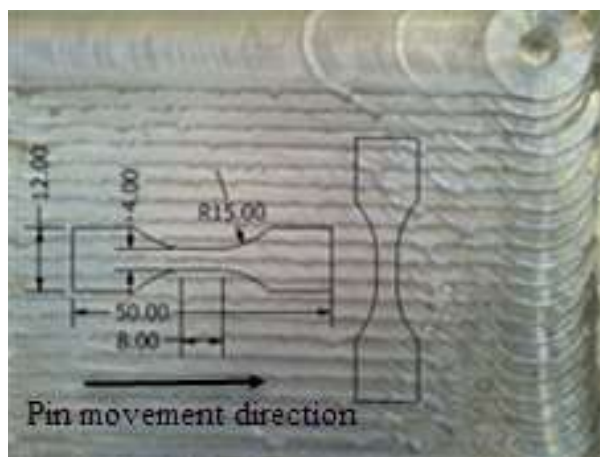


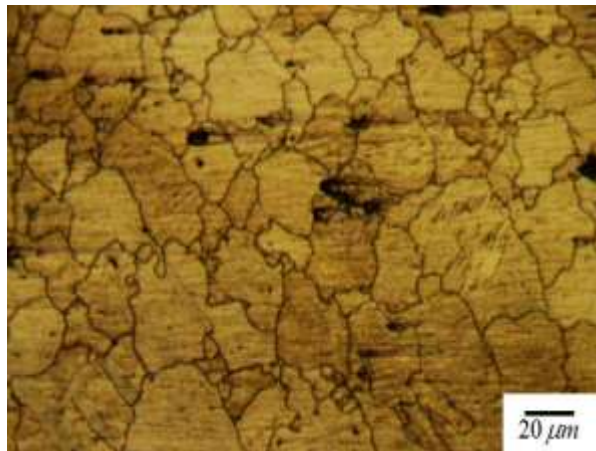
Fig. 3 Dimensions of the tension sample

These samples were polished in order to omitting surface effects. Tension tests were carried out using SANTAM machine under strain rate of 0.01(1/s). Microhardness of samples were measured under 100 grf and 15 second. After etching the surface of samples with a solution of 4.2 g Picric acid, 10 ml Acetic acid, 10 ml distilled water and 70 ml Ethanol, the microstructure of samples was observed using optical microscope and SEM were used in order to observe distribution of reinforcing phases within alloy AZ31.

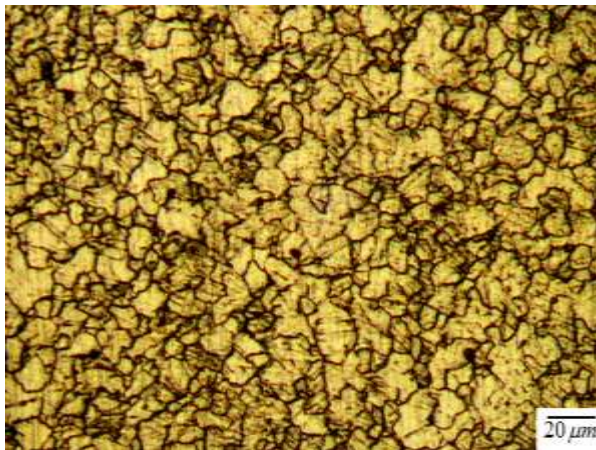
3 DISCUSSION: MICROSTRUCTURE

In Fig. 4 Photos taken by optical microscope and in Fig. 5 Photos taken by SEM are shown. Figures 4-a, 4-b, 4-c, 4-d, 4-e respectively are the microstructure of raw material, FSP areas without reinforcing nanoparticles, and ratios of 4, 8, and 16% of volume percent of SiC nanoparticles. The raw material has non-uniform structure with grains sized 18 micrometer. The average grain size of the AZ31 structure after 4 FSP passes and cooling was 6.4 μm without reinforcing materials, 2.04 μm with 4% SiC, 1.65 μm with 8% SiC, and 1.15 μm with 16% SiC. In the 50% overlap FSP with 4% of nano SiC particles the grain size was same as 100% overlap with 4% nano particles. As it is seen in Fig. 4, use of SiC nanoparticles makes the grain size of AZ31 structure to be reduced and more uniform. This fact is also mentioned by other researchers [13], [17], [24], [25]. By reducing the traverse speed of FSP process, the sizes of material grains without reinforcing phases and material consisting of reinforcing materials will be increased due to a great increase of sample temperature. In the case where the material consists of reinforcing materials, these materials prevent grain growth of grains even with high temperature in the piece [13]. In this research regarding the use of cooling during FSP process, overgrowth of sample without reinforcing phases is prevented too. Figures 5-1, 5-b, and 5-c demonstrate SiC particles distribution in states of 4%, 8% and 16% of volume in 100% overlapping state respectively and Fig. 5-d demonstrates SiC particles distribution in the state of 4% and 50% overlapping state within AZ31. As it is seen, SiC particles illustrate more dispersion within the material by increasing the volume percent. SiC particles are placed inside the Magnesium grains and between them and they have uniform distribution. This uniform distribution of SiC particles inside the grains and between them are also reported by other researchers [13], [16], [17]. In terms of nanoparticle distribution, the 50% overlapping state in volume percent of 4 is same as 100% overlapping state. The only difference between 50% and 100% overlapping FSP is emergence

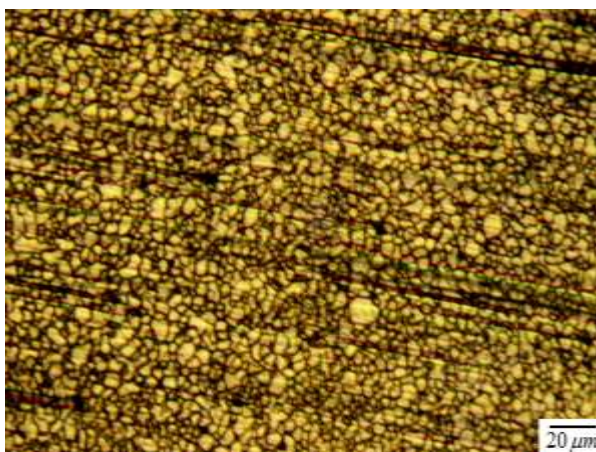
of transition zones between passes in which they have very small grains. This fact is shown in Fig. 4-f.



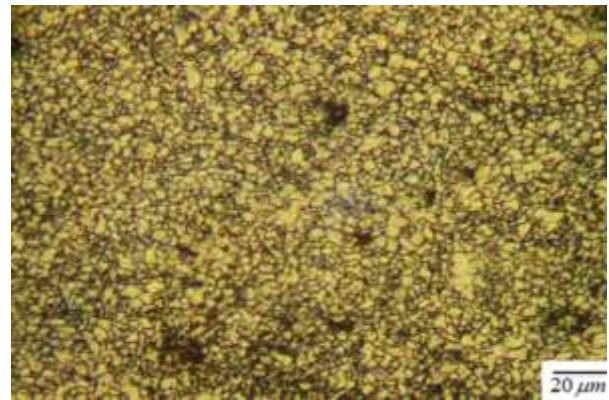
a)



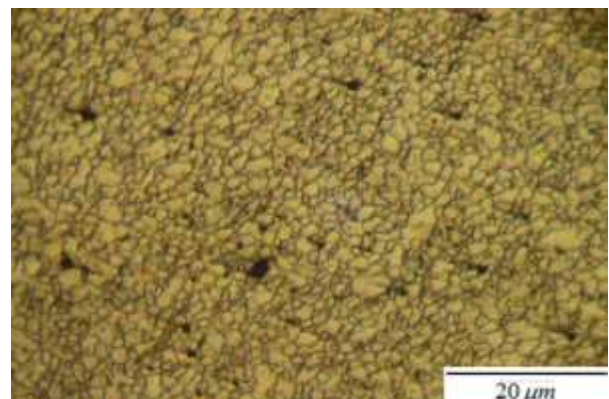
b)



c)



d)



e)



f)

Fig. 4 a) The structure of raw material. b), c), d), e) respectively the material structures after FSP without reinforcing materials with 4% SiC, 8% SiC, and 16% SiC. f) the transition zone between passes

4 DISCUSSION: MICROHARDNESS

In FSP process together with powder, three factors increase hardness, 1- refinement of the size of the grains, 2- emerging reinforcing phases into the matrix, 3- quench hardening due to the difference between thermal expansion coefficient of reinforcing materials and the matrix [7]. Hardness of different modes is illustrated in diagram of Fig. 6.

As it can be seen, the hardness is increased from 67 Vickers for raw material to 112 Vickers for composite containing 16% of SiC nanoparticles. According to Eq. (2) in which H is hardness and V is volume percent, by increasing the volume percent of reinforcing phases the hardness of composite will be increased [26].

$$H_{\text{composite}} = H_{\text{matrix}} V_{\text{matrix}} + H_{\text{particle}} V_{\text{particle}} \quad (2)$$

This matter is consistent with results presented in Fig. 6. it can link the hardness increase in nanocomposite containing SiC particles to uniform distribution of SiC nanoparticles within the matrix so that a structure with a smaller grain size will be established as a result. An increase in micro-hardness resulting from the uniform distribution of SiC nanoparticles has been previously reported [11], [15]. In a sample with 50% overlapping, the hardness distribution changes more than 100% overlapping state that this matter is justified by explanation were given about transition zones between passes in microstructure section.

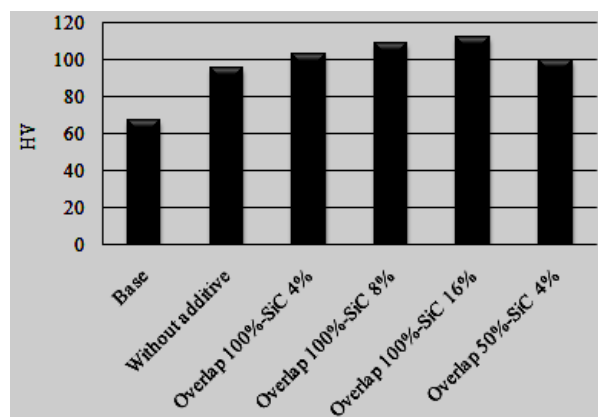


Fig. 6 hardness average values of raw material, 4 times FSP process without reinforcing materials and composite containing 4, 8, and 16 percent of SiC nanoparticles in 100% overlapping and containing 4% of SiC nanoparticles in 50% overlapping

5 DISCUSSION: TENSILE STRENGTH REVIEW

In Fig. 7, the stress-strain diagram, raw material, 1 time FSP process, 4 times FSP process without reinforcing materials and with 4, 8, and 16 percent of SiC nanoparticles in 100% overlapping are shown. In Table 2, numerical values of yield stress, ultimate stress, and elongation are given for different specimens. As it can be seen in Fig. 7, the FSP process without adding reinforcing materials causes reduce of the sample yield stress but increase of ultimate stress and its elongation. These results were observed by Mishra and Yuan [6] and also Daras et al. [2]. In a situation of

using 4 times FSP process (because the material structure has become more uniform with smaller size of grains), elongation and ultimate stress have higher values compared with 1 time FSP state. Also Venkateswarlu et al. [5] observed that by implementing 2 times of FSP process on the Magnesium AZ31, the structure becomes more uniform and ultimate stress and elongation increase. It can be seen in Fig. 7 that by increasing volume percent of SiC nanoparticles, the yield stress of material increases in comparison with the state of FSP without reinforcing materials, but elongation and ultimate stress does not seem to be increased and even by increasing the volume percent from 4 to 8 and 16, they will be decreased.

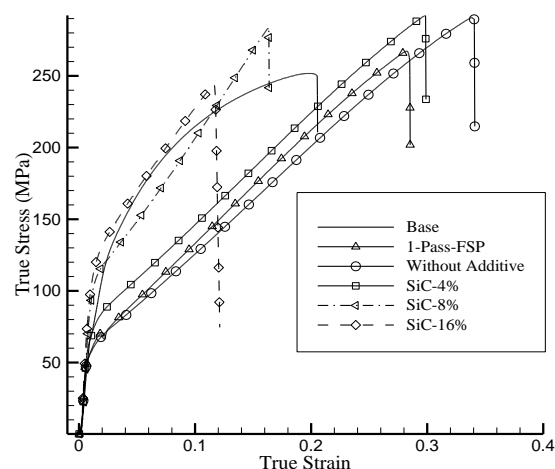


Fig. 7 The stress-strain diagram of raw material, the process of 1time FSP, 4 times FSP without reinforcing materials and with 4, 8, and 6 percent of SiC nanoparticles

Table 2 Numerical values of yield stress, ultimate stress, and elongation

	Yield Stress (MPa)	Ultimate Stress (MPa)	Elongation (%)
Base	110.14	251.80	22.82
Without additive-1Pass	56.71	267.35	33.01
Without additive-4Pass	57.30	290.70	40.63
Overlap 100%-SiC-4%	75.42	292.21	34.84
Overlap 100%-SiC-8%	105.17	283.83	17.80
Overlap 100%-SiC-16%	122.27	244.13	12.89
Overlap 50%-SiC-4%-Longitudinal	103.53	227.77	21.66
Overlap 50%-SiC-4%-Transverse	87.08	132.57	11.09

Increase of yield stress and decrease of elongation by increasing volume percent of SiC nanoparticles is also reported by Chawla and Shen [27]. SiC nanoparticles reinforce the matrix using two mechanisms. In the first mechanism, the stresses are transferred from the matrix to the nanoparticles. The aspect ratio of the nanoparticles enhancing the strength of the composite from this mechanism is low, but it has been reported that it has a specific influence on the increase in mechanical properties. The second mechanism is that the difference between matrix and nanoparticle cooling initiates dislocations around the nanoparticles, but the nanoparticles prevent dislocation. In this way, the nanoparticles increase the strength of the composite [27]. By increasing volume percent of nanoparticles, work hardening rate of material increases. Thereupon the stresses created around the nanoparticles, which are sites for stress concentration by themselves, are released to a lesser extent and that causes faster arise of cracks in the material and reduction of material elongation [27]. The properties of created composite with 4% of SiC nanoparticles are same as the state without reinforcing materials. San et al. [17] reported that if the volume of reinforcing materials is low, they would not have a significant impact on increasing the material strength. Increasing the yield stress as an effect of SiC nanoparticles can be justified by Eq. (3) in which σ_c is the yield stress of matrix, σ_o is the frictional stress, k is the gradient of Hall-Petch relation, d is the size of matrix grain, V_f is the volume percent of reinforcing materials, and S is aspect ratio [24].

$$\sigma_c = \left(\sigma_o + kd^{-1/2} \right) \left[\frac{V_f (s+4)}{4} + (1-V_f) \right] \quad (3)$$

According to this equation, decreasing the size of grain and increasing the volume percent will increase the yield stress which is compatible with results presented in this study.

In composites, the ultimate stress is more sensitive to microscopic defects of matrix in comparison with yield stress [10]. Around SiC nanoparticles, high stress concentration occurs and considering that the matrix cannot release its stress in these areas, cracks arise and cause rupture in the composite. Therefore, matrix cannot bear great stresses and the ultimate stress will be reduced. By increasing the reinforcing materials in matrix, distance between these materials will be reduced and stress relaxation will face far more problems and this matter causes reducing ultimate stress by increasing the volume percent of reinforcing materials [24]. Another reason that could be linked with reduction of elongation and ultimate stress is agglomeration of SiC nanoparticles. As it can be seen in Fig. 5, by increasing the volume percent, aggregation of SiC nanoparticles will be increased in the matrix. In 50% overlapping state towards 100% overlapping state,

the yield stress increased but the ultimate stress and elongation reduced in which this condition is weaker in sample tensions perpendicular to the pin direction. Decreasing mechanical properties in direction of process implementation also was reported by Ma et al. [20]. 50% overlapping causes average size of grains fine in passes transmission areas. This matter causes increasing the yield stress in direction of pin but it causes decreasing the properties in the perpendicular to pin direction due to non-uniformity in grain size.

6 CONCLUSION

In this study, SiC nanoparticles were added to magnesium AZ31 alloy using FSP process. FSP process was accomplished using 4 consecutive passes along with cooling. Microstructure, hardness and tensile strength of raw material, one pass of FSP, 4 passes of FSP without reinforcing materials, and 4 passes of FSP for 4, 8, and 16 volume percentages in 100% overlapping and in 4% volume percent in 50% overlapping were studied in which the following results were achieved:

- 1- FSP process makes the size of grains fine and also more uniformity. By increasing the number of passes, this uniformity and reduction of grain size will also increase and improve the mechanical properties.
- 2- Adding SiC nanoparticles prevents growing of the grain size and also makes the structure more uniform.
- 3- The hardness of the material increases by implementing FSP process and adding reinforcing phases. There is a direct correlation between this increase and the volume percent of reinforcing materials.
- 4- By performing 4 times of FSP process, SiC nanoparticles distribute uniformly in alloy AZ31.
- 5- By adding reinforcing materials, the yield stress increase but the ultimate stress and elongation decrease.
- 6- in 50% overlapping state in direction of the pin and perpendicular to pin movement, the yield stress increases compared with 100% overlapping state but the ultimate stress and elongation decrease. This matter is related to lack of uniformity in size distribution of grains. Considering that this lack of uniformity is more in the direction perpendicular to the pin movement, therefore mechanical properties decrease more in this direction.

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