RESEARCH PAPER

The Wear Behavior of a Cast Al-20wt%Mg$_2$Si Composite Thixoformed via SIMA

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Abstract: Aluminum matrix composites are candidate materials for aerospace and automotive industries owing to their specific properties such as high elastic modulus (E), suitable strength and low wear rate. The effect of thixoforming process on the wear behavior of an Al-Mg$_2$Si composite sample was studied in this paper. In thixoforming process, casting defects such as macrosegregation, shrinkage and porosity are reduced significantly. These advantages are sufficient to attract further studies on thixoforming operation. Thermal analysis of the composite sample, as-cast microstructure, wear surface and subsurface area of the thixoformed alloy were investigated. Wear behavior of the specimens were examined using a pin-on-disk machine based on ASTM-G99, at the applied loads of 25, 50 and 75 N and the constant sliding velocity of 0.25m/s. The worn surfaces and subsurfaces were examined by scanning electron microscopy (SEM). The experimental results indicated that the thixoformed specimens exhibited superior wear resistance than the as-cast alloy. Moreover, the dominant wear mechanism is an adhesive wear followed by the formation of a mechanical mixed layer (MML). However, a severe wear regime occurs in the as cast specimens compared to the thixoformed counterparts.

Keywords: Al-Mg$_2$Si Alloy, SIMA, Semi-solid forming, Thixoforming, Wear test.

1. INTRODUCTION

Aluminum matrix composites are widely used in aerospace and civil industries because of their low densities and a favorable combination of strength and resistance to corrosion [1]. Metal matrix composites (MMCs) are a class of materials that seek to combine the high strength and stiffness of a ceramic with the damage tolerance and toughness provided by a metal matrix [2]. As a new group of MMCs, in situ Al-Mg$_2$Si composites are used in many industrial applications. In situ Al-Mg$_2$Si composites has high potential as a wear resistant material because the intermetallic compound of Mg$_2$Si exhibits high melting temperature (1085 °C), low density (1.99×10^{3}kgm^{-3}), high hardness (4500 MNm^{-2}), low thermal expansion coefficient (7.5×10^{-6} k^{-1}) and excellent workability [3]. However, the primary Mg$_2$Si phases in the as-cast Al-Mg$_2$Si composites are usually very coarse and apparently, the presence of coarse second phases and their uneven distributions have a deteriorating effect on the room temperature mechanical properties. In

this regard, several modification methods have been proposed to optimize the morphology of second phases [4,5]. As the result of the modification, the Mg$_2$Si morphology changes from dendritic form to polygonal one [2,4,5].

Thixoforming is widely known as a technology that involves formation of metal alloys between solidus and liquidus temperatures. Thixoforming process produces less casting defects such as macrosegregation, shrinkage and porosity. These advantages have attracted more explorative work on thixoforming operation [2,4,5,6]. For the procedure to operate successfully, the microstructure of the starting material must consist of solid near globular particles surrounded by a liquid matrix and wide solidus-to-liquidus transition area [7,8] The aim of the semisolid processing is to achieve a fine globular structure [5,8]. Among the production methods, SIMA is an ideal candidate with significant commercial advantages of simplicity and low equipment costs [9]. The strain induced melt activation (SIMA) process has been used to enhance the mechanical properties of Al alloys in recent years. In this process strain

is stored in a billet and a globular structure is evolved by the strain energy in the billet after reheating. Parameters such as heating time, temperature and the degree of cold working are critical factors in controlling the semisolid microstructures in SIMA process [10] In addition, it omits the procedure of molten metal treatment, and is applicable for both low and high melting alloys [13-15]. Experimental results indicated that a non-dendritic microstructure could be obtained by the SIMA process in the alloy [16]. Many studies have been done on the wear regimes of Al-Si alloys [15,17,18]. Dyson has identified three regimes, scuffing, severe and seizure during wear process at the different applied loads [19]. Saghafian et.al [17] in a study on wear behavior of a thixoformed Al-25wt%Mg2Si composite reported that the dominant wear mechanism could be delamination wear normally associated with the formation of an MML containing pin and disc materials for all the applied loads. It is reported that the transition from mild wear has been designated as metallic wear, scuffing, seizure and sever regimes [16].

Depends on the basis of observations and analysis on the wear rates and worn surfaces, the wear mechanism of the Al-20wt%Mg$_2$Si was dominantly controlled by adhesive and minor delamination [8]. However, less work has been carried out on the wear behavior of Al-Mg$_2$Si composites. This work is an attempt to study the effect of thixoforming via SIMA on wear behavior of the Al-20wt%Mg$_2$Si composite.

2. EXPERIMENTAL PROCEDURE

Commercial cast Al alloy 413 (ingot) and pure magnesium (ingot, ≥98%purity) were used as starting materials. About 450g of molten Al alloy 413 was prepared using a resistance electric furnace and a graphite crucible. About 85 g of magnesium preheated at 300°C was added into the molten alloy at 720-750°C. After holding the molten alloy at this temperature for 15 minutes, it was poured into a steel die of 35mm diameter × 100mm height ×10mm thickness) at 700°C to produce in situ

Al-Mg$_2$Si composite ingots.

Subsequently, the ingot was cut into a series of disk specimens with the dimensions of 35mm in diameter and 15mm thickness, and then rolled to certain thicknesses at room temperature to achieve 5 and 10% reduction of area using a 50 ton rolling machine.

The eutectic and liquidus temperatures of the composite were determined by thermal analysis (TA) and its corresponding differentiated curve.

The rolled specimens were heated at 575°C for 45 min and subsequently thixoformed using a hydraulic press under 5ton force and 5 mm/s cross head speed. Heating time of specimens, the necessary force and cross-head speed was determined practically.

To measure the hardness of specimens, a Brinell hardness testing machine using a ball indentor of 2.5 mm in diameter at the applied load of 31.25 kgf was employed.

To examine the wear behavior of the prepared specimens, the thixoformed and the as-cast specimens were cut into the pins of 5mm in diameter and 10mm height. Steel disk AISI/SAE 52100 of 30mm in diameter and 10mm thickness with hardness 60-63 HRC was used as counterpart. keller reagent was used for etching in the metallographic process. Dry sliding wear test conducted using a conventional pin-on-disk testing machine based on ASTM-G99 and at the applied loads of 25, 50 and 75N. The sliding distance and velocity were 1000m and 0.25 m/s, respectively, and test was run at room temperature. Weight losses of specimens were measured by digital balance with ±0.1 mg precision. To determine the wear mechanism of the composite Specimens, wear surface and subsurface, and wear debris morphology, were studied using scanning electron microscope (SEM) linked with energy-dispersive spectroscopy (EDS).

3. RESULTS AND DISCUSSION

3.1. Chemical Analysis

Chemical composition of the manufactured Al-Mg$_2$Si composite is shown in table 1.

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Table 1. Chemical composition of produced ingot

Elements	Mg	Si	Cu	Fe	Mn	Zn	Al
Al-Mg₂Si Comp. (%Wt)	13.8	9.6	0.14	0.4	0.04	0.44	Rem.

3.2. Thermal Analysis

To determine the liquidus and eutectic reaction temperatures of the composite, thermal analysis (TA) method giving the cooling curve and then its corresponding differentiated curve was performed. Based on the results of this method, the eutectic and liquidus temperatures are 545 and

Fig.1. The cooling curve of the composite and its corresponding differentiated curve.

Now it is necessary to determine the desirable solid fraction for thixoforming process which is reported [12] to be 50-70vol%. Based on Thermocalc software giving the solid fraction versus temperature diagram, appropriate temperature to reach such volume fractions of solid is 575-585°C, Fig.2. Thermocalc is a thermodynamic calculation software for

Fig.2. The solid fraction-temperature diagram achieved by Thermocalc.

tackling mineral equilibria problems. It has two main components: the application itself, and the internally-consistent thermodynamic dataset it uses [10].

3.3. As-Cast Microstructure

The microstructure and EDS analysis of the as-cast Al-20wt%Mg₂Si composite Specimen, cast at 700°C are illustrated in Fig. 3 and table 2, respectively. As seen it consists of primary Mg₂Si particles, α-Al phase, binary eutectic of α-Al+Mg₂Si and Si particles present in eutectic mixture. Some needle like iron-rich intermetallics like π-Al₈Si₆Mg₃Fe and
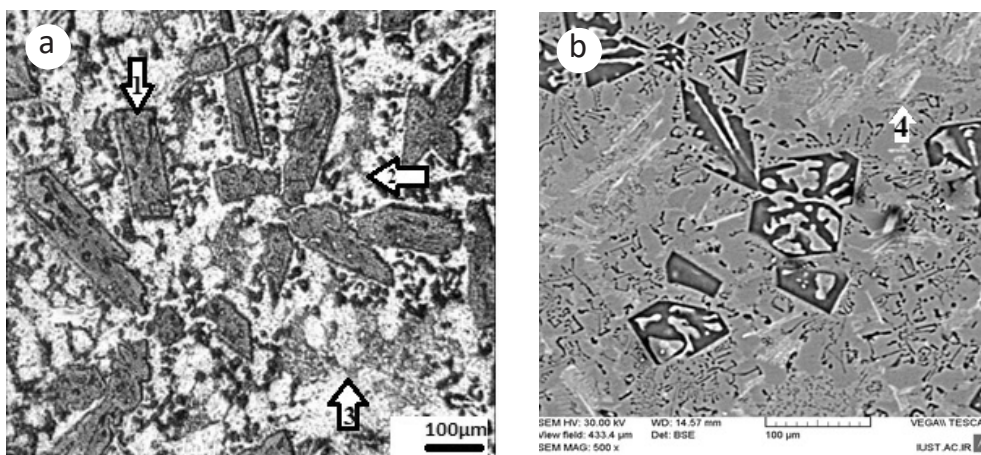
Fig.3. (a) Typical microstructure of the Al-20wt%Mg₂Si composite, as-cast at 700°C. (b) The same as (a) at higher magnification. 1-Primary Mg₂Si, 2-Binary $_{Eu}$, 3-Si$_{Eu}$, 4-Iron-rich intermetallic

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β-Al$_5$FeSi phases are also appeared in the final microstructure considering the chemical composition of the composite made [19].

Table 2. EDS analysis of (a) α-Al (b) Mg$_2$Si and (c) iron-rich intermetallics

Wt%	Mg	Al	Si	Fe
a	2.59	97.41	-	-
b	64.30	-	35.70	-
c	9.83	67.61	18.12	4.44

The volume fraction and mean particle size of Mg$_2$Si phase in the as-cast Specimen are 30vol% and about 105 μm, respectively, which were obtained via MIP software. The most important microstructural feature seen in Fig. 3 is undesirable shape and size of Mg$_2$Si particles associated with an uneven distribution deteriorating the mechanical properties such as strength and wear resistance. Hence, it was necessary to do some cold work (e.g. rolling) to break up these particles into smaller ones before applying the semisolid process. Although the smaller particles of Mg$_2$Si are resulted from cold working of the starting materials, the particles size distribution is not able to be changed in the cold worked state. In addition, the microstructure consists of cracks within the matrix along with undesirable fracture surface of the particles as result of applying cold working. To remove these defects and establish a continuous matrix containing the smaller Mg$_2$Si particles with an even distribution, applying a subsequent semisolid process is neces-

sary. Hence, the 5 and 10% cold worked specimens were heated at 575 °C for 45 min and then pressed.

3.4. Heating at 575 °C and Microstructure of Thixoformed Specimens

The desirable microstructure for semi-solid deformation process consists of spherical solid particles as small as 100 μm in a continuous liquid matrix. Liquid fraction in this process should be 30-50vol% [19]. The temperature equivalent to 50% volume fraction, calculated by Thermocalc software (Fig.2) using solid fraction versus temperature diagram, is 585 °C. However, based on the experimental results, the amount of solid fraction was smaller than expected for deformation process. Therefore, to attain a desirable structure and prevent excess liquid fraction, the Specimens were heated at a lower temperature of 575 °C for 45 minutes. Appropriate heating time was obtained experimentally.

Fig.4 shows the microstructure of two semi-solid heat treated specimens. As seen in Fig.4, much smaller particles of Mg$_2$Si with desirable morphology and even distribution is established within the matrix alloy in comparison with the as-cast specimens. This is, in fact, resulted from combination of the cold working process (breaking up the coarse particles) and applying the subsequent semisolid process followed by pressing, which is totally called thixoforming process. In other words, during heating stage of the process, the eutectic constituent of the composite is molten and injected by the subsequent pressing force into the cracks occurred

Fig. 4. The composite microstructure heated at 575 °C for 45 minutes with (a) 10% reduction of area (b) 5% reduction of area

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within the matrix and between the fractured particles. As result of which the fracture surface of the particles is also wetted by the melt, a continuous matrix containing much smaller particles with corners rounded. Fig. 4 also shows the effect of the primary cold working percent applied on the starting materials before semisolid stage of this process. As it seen clearly, applying a higher cold work percent (Fig. 4b) causes a smaller particle size. This can be attributed to the formation of the smaller fractured particles induced by applying a higher percentage of deformation to the materials.

Fig.5 shows the arrangement of Mg_2Si, iron rich intermetallic particles and globular α-Al grains in the thixo-formed Specimen. The eutectic Si phase and iron rich intermetallic particles are modified and transformed to the fine globular particles during semisolid treatment.

3.5. Evaluation of Hardness Test Results

Table 3. The results of Brinell hardness test

Production Process	Hardness (Brinell)
As-cast	71.1
SIMA	74.7
Thixo-form	80.3

The hardness values of the specimens under different conditions is given in table 3. As seen, the hardness of Specimens increased upon applying semisolid deformation treatment.

In the cast Al-Mg_2Si composites, high stresses were concentrated on the coarse Mg_2Si dendrites

and micro cracks simply originated from the primary Mg_2Si particles leading to inferior mechanical properties. In contrast, in the thixo-formed composites, the fine and globular particles of Mg_2Si experience less stress concentrations. Consequently, the probability of micro-crack formation effectively decreased in thixo-formed specimens and thus the hardness was improved [8].

This results also indicated that hardness of thixo-formed Specimen was higher than ones prepared through SIMA. This is because of elimination of shrinkage porosities by applying load during thixo-forming process.

3.6. Wear Behavior

The results for wear rate variations versus the applied load are shown in Fig.6. As it can be observed, wear rate increases with the applied load for all Specimens. It is also seen that wear rate of the thixo-formed composites is lower than the as-cast composites.
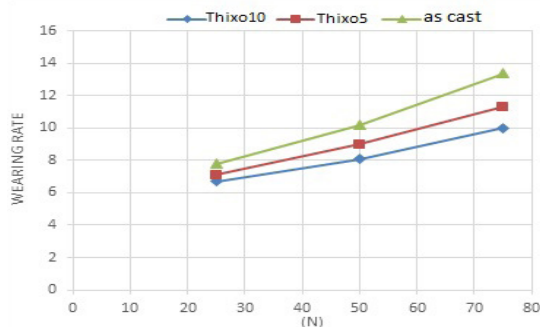

Fig. 6. Wear rate variations versus the applied load.

Fig.5. Distribution of α-Al and Mg_2Si particles in the composite heated at 575 °C for 45 minutes with 10% reduction of area (b) The same as (a) but at higher magnification. 1-Iron-rich intermetallic. 2- Si Eutectic.

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Improvement of hardness and wear properties of the thixo-formed specimens can be attributed to the following micro- structural evolutions:

1. Diminishing particle size of the primary Mg₂Si and elimination of sharp edges during rolling (one step in thixoforming). As particle size becomes smaller, existing cavities in Mg₂Si particles decreases. It is reported that these cavities act as the crack initiation places [2];

2. Formation of globular α-Al particles when reheating at semi-solid temperature;

3. Modification of eutectic Mg₂Si particles, eutectic Si particles and iron-rich intermetallics.

Applying load during wear test causes plastic deformation leading to break up of the coarse dendrites of Mg₂Si and therefore, increasing the wear rate of the cast specimens. But in the thixo-formed composites the globular and fine particles of Mg₂Si are not severely broken up by the load application, so that these hard particles decrease the surface plastic deformation of specimens and this means improvement of wear properties [2].

Improper distribution of the rough binary and ternary eutectic particles as well as needle-shaped intermetallics in the cast Specimen causes generation of stress concentration at the sharp edges of these particles when applying load affecting its mechanical properties.

3.7. Study of Worn Surfaces and Debris

To study the wear behavior of the specimens, the worn surfaces and sub-surfaces, morphology and chemical composition of wear debris were examined.

As shown in Fig.7, the worn surfaces of the Al-Mg₂Si alloy at the applied load of 25 N (both as-cast and thixo-formed specimens) contain some craters (peeled off regions). EDS analysis of these craters (table 4) shows the presence of pin and disc constituents (Al, Mg, Si and Fe) implying the formation of a mechanical mixed layer (MML) on the worn surfaces during wear process [17]. Considering EDS analysis of the wear debris collected from the same specimen (Fig.8) worn under the same condition (Table 5), it could be concluded that an adhesive wear followed by the formation of MML at the worn surface and its

subsequent delamination is the dominant wear mechanism. On the other hand, the presence of a higher level of iron in the crater areas (Table 5) can imply the fact that the thixo-formed specimen exhibited a superior wear resistant than the as-cast one [17].
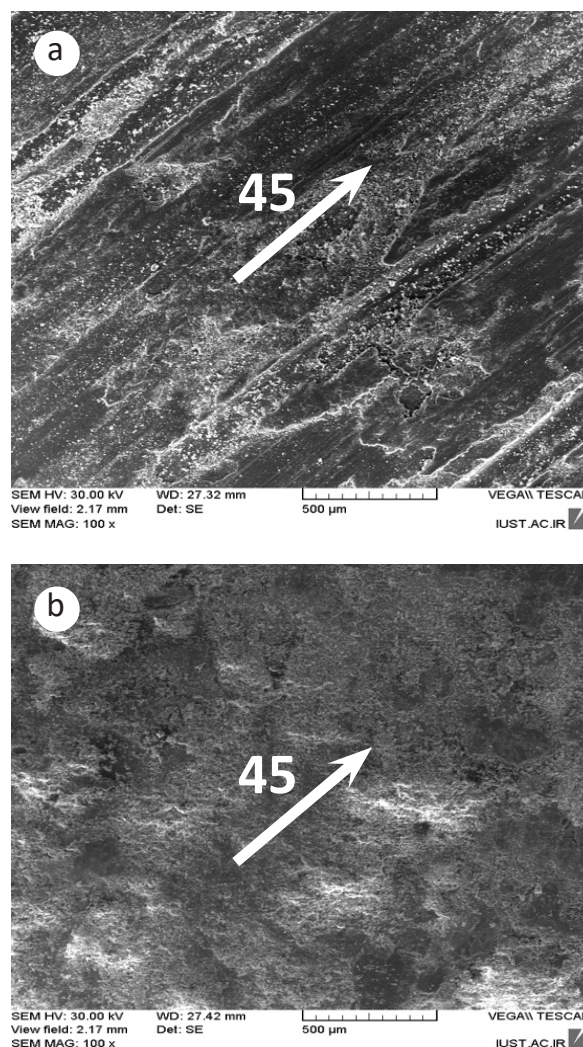

Fig.7. The worn surface of the Al-20%wtMg₂Si alloy at the applied load of 25 N (a) as-cast (b) thixo-formed.

Table 4. EDS analysis of the worn surface of the Al-Mg₂Si alloy at the applied load of 25 N (a) as-cast (b) thixo-formed with 10% reduction.

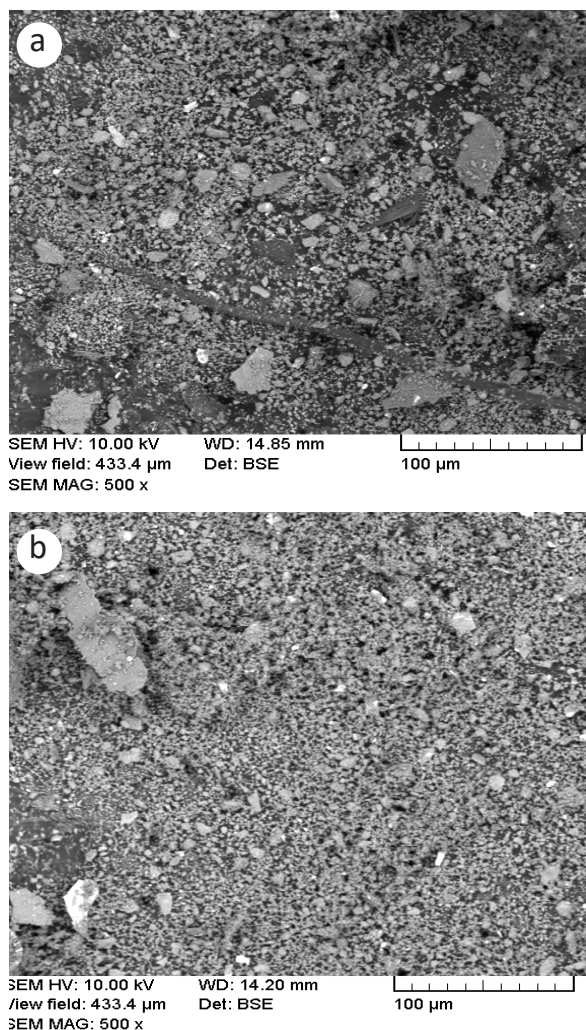| Wt%	O	Mg	Al	Si	Fe
a	34.96	5.02	46.25	5.93	7.84
b	46.73	4.26	30.67	4.39	13.94

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Table 5. EDS analysis of the debris collected from the specimens worn at the applied load of 25N (a) as-cast (b) thixo-formed with 5% reduction of area(c) thixo-formed with 10% reduction of area

Wt%	O	Mg	Al	Si	Fe
a	42.30	8.91	32.50	5.78	7.82
b	45.30	5.32	32.49	4.97	11.85
c	46.57	4.53	30.30	3.97	14.64

The presence of oxygen in the collected debris can be attributed to the formation of iron and aluminium oxides. However, because of the presence of the specimen elements content and lack of formation of a monolithic oxide layer, the oxidative wear could not be a dominant wear mechanism. The presence of a higher level of oxide content in the MML can improve the shear strength of this layer by acting as a binder. This, in turn, may reduce the fluctuation of friction coefficient variation seen in Fig.9.

At the higher applied load of 75N the severer wear was appeared. The plan views of the worn surfaces for both as-cast and thixo-formed specimens worn at the applied load of 75N are given in Fig.10. As clearly seen more damages are seen on the worn surfaces because of applying a higher load. In addition, the presence of craters containing debris particles on the worn surfaces (table 5), with a mixture of pin and disc components,(table 6) confirm that an adhesive wear associated with

Fig.8. Wear debris collected from the specimens worn at the applied load of 25N (a) as-cast (b) thixo-formed with 10% reduction of area.

Fig.9. Variation of friction coefficient with sliding distance at the applied load of 25 N:
a) as-cast b) after thixoforming with 10% reduction of area

the formation of MML could be the main working mechanism at this applied load. It is also seen that the Fe/Al ratio (table 6) increases after thixoforming the specimen. The smaller debris particle size (Fig. 11b) containing the higher Fe/Al ratio particles (table 7 b and c) imply that the thixo-formed specimens exhibit a superior wear resistance when compared to as cast Specimens. This could be further confirmed with a reduction in the friction coefficient average value, (Fig. 12) with narrower fluctuations showing a harder subsurface in the thixo-formed specimen.

Table 6. EDS analysis of the worn surface of the Al-Mg$_2$Si alloy at the applied load of 75 N (a) As-cast (b) After thixoforming with 10% reduction of area

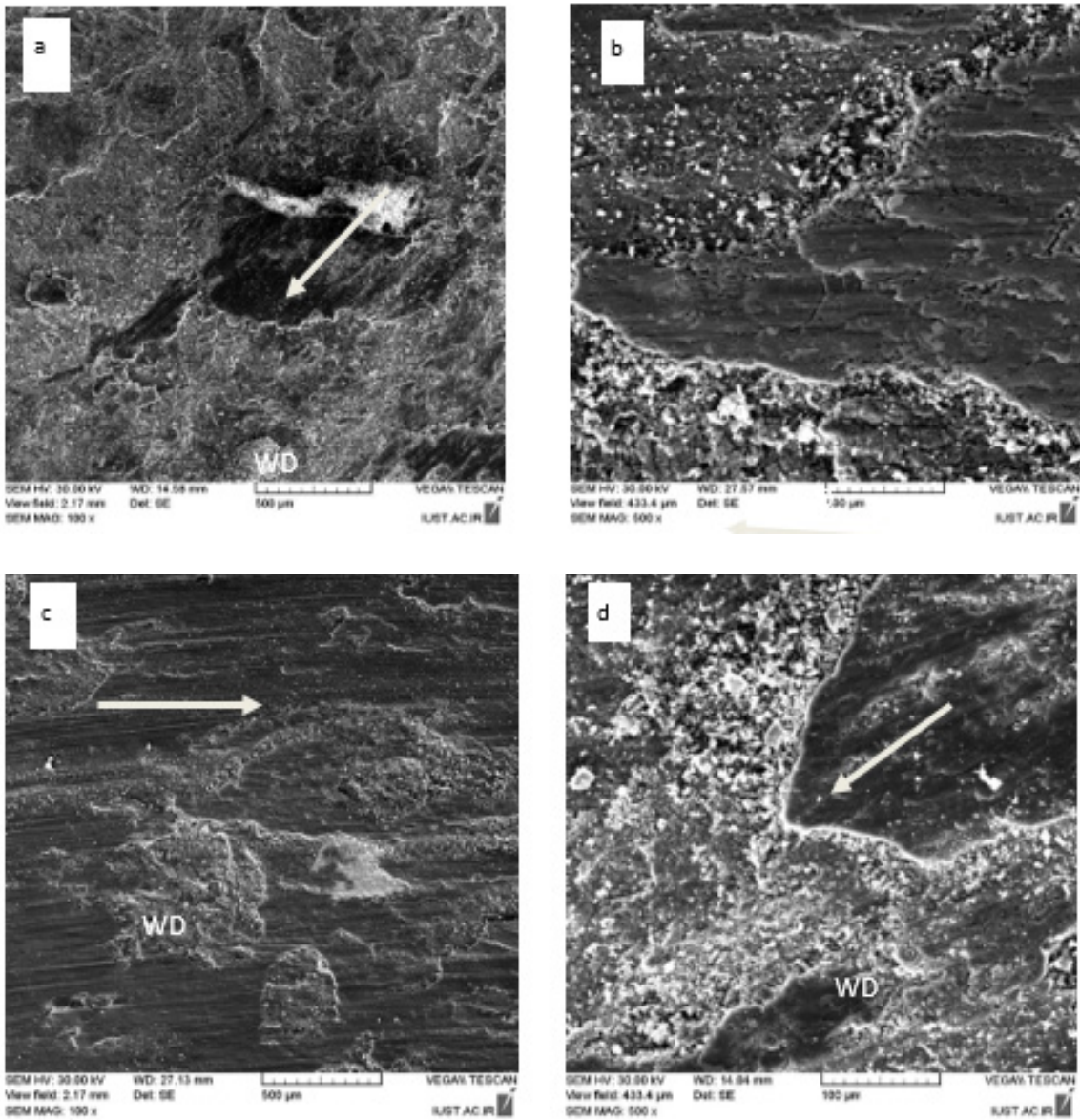| Wt%	O	Mg	Al	Si	Fe
a	43.66	5.29	29.48	3.70	17.87
b	15.17	3.53	37.18	2.51	44.61

Fig.10. The plan view of the worn surfaces of the Al-Mg$_2$Si alloy worn at the applied load of 75 N: (a) As-cast. (b) Higher magnification of (a). (c) After thixoforming with 10% reduction of area. (d) Higher magnification of (c).

Fig.11. Debris particles collected from the specimen worn at the applied load of 75N
(a) As-cast. (b) After thixoforming with 10% reduction of area.

Table 7. (a) EDS analysis of the debris collected from the specimen worn at the applied load of 75N a) as-cast (b) after thixoforming with 5% reduction of area (c) After thixofoming with 10% reduction of area.

	O	Mg	Al	Si	Fe
Wt%					
a	46.04	4.28	29.30	4.03	16.35
b	44.73	4.81	27.20	3.93	19.33
c	36.59	6.25	28.24	4.22	24.70

3.8. Study of Subsurface Area

To examine the location of crack initiation as well as the role of Mg_2Si particles, the subsurface area was studied by SEM. Fig.13 shows the subsurface layer of the specimens worn at the applied loads of 25 and 75N. As seen the depth of plastically deformed area increased with the applied load. The presence of subsurface cracks containing debris particles is consistent with the formation of MML occuring during an adhesive wear mechanism [17, 18].
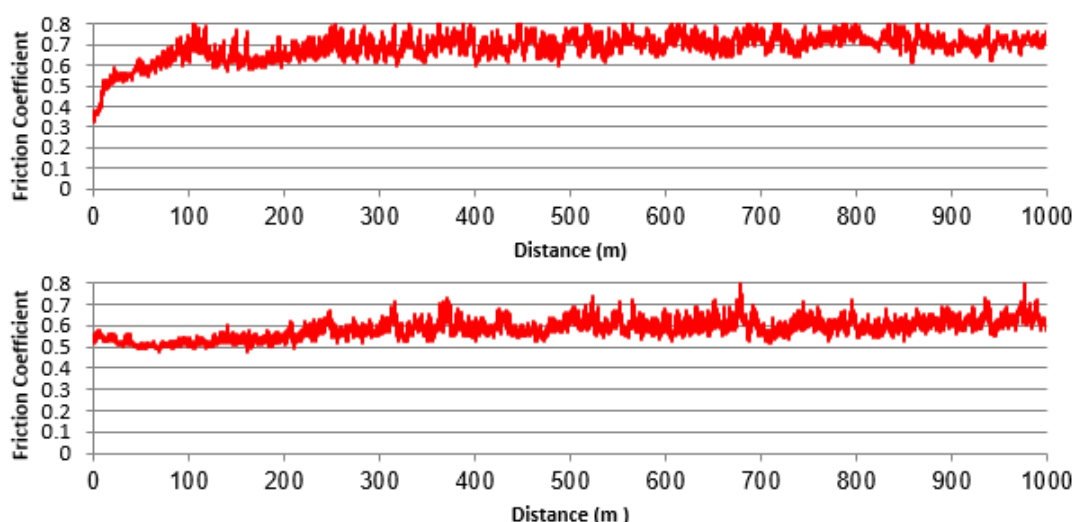

Fig. 12. Variation of friction coefficient with sliding distance at the applied load of 75 N:
(a) As-cast (b) After thixoforming with 10% reduction of area

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Based on the mentioned wear mechanism, formation of MML begins with initiation of micro cracks preferentially at the interface of matrix alloy/second phase and its further growth and then approaching the surface. The debris particles formed at the beginning of wear process enter the surface cracks and a sandwich form layer is formed upon the further sliding process.
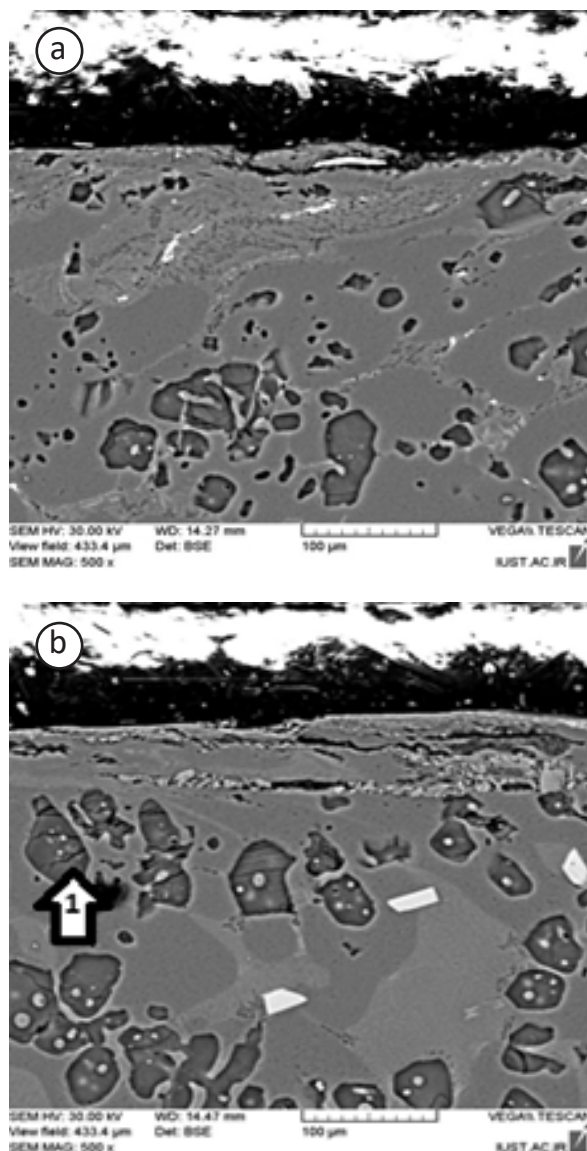

Fig.13. The subsurface area of the Specimens worn at the applied loads of (a) 25N and (b) 75N. Cracks in particle shown by arrow numbered 1. Formation of tribolayer (MML) during the wear process can be apparently seen.

4. CONCLUSIONS

Based on the results obtained from the current work, the main effects of applying thixoforming process on the microstructure and, therefore, wear behavior of the cast Al-20wt%Mg$_2$Si are as follows:

1. The morphology of primary Mg$_2$Si changes from the sharp edge particles into a semi spherical shape accompanied by a decrease in particle size.

2. Eutectic Mg$_2$Si particles, eutectic Si particles and iron-rich intermetallics are also modified.

3. Primary α-Al phase changes from a dendritic morphology into a globular when reheating at semi-solid temperature.

4. The microstructural features are further enhanced when increasing the reduction of area from 5 to 10%.

5. Microstructural modification specifically removing the sharp edges of the primary Mg$_2$Si particles gives rise to decreasing the stress concentration sites causing the initiation micro cracks. As result of this, the shear strength needed for wear resistance during sliding wear process increases.

6. A combination of the results of worn surface and subsurfaces, debris particles characteristics and friction coefficient's values and fluctuations imply that the dominant wear mechanism is an adhesive wear followed by formation of a tribolayer (MML). However, a severe wear regime occurs in the as cast specimens compared with the thixoformed counterparts.

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