

# Orthogonal Experimental Design Applied for Wear Characterization of Aluminum/C<sub>sf</sub> Metal Composite Fabricated by the Thixomixing Method

Ebrahim Akbarzadeh<sup>1,\*</sup>, J.A. Picas<sup>2</sup>, M.T. Baile<sup>2</sup>

<sup>1</sup> Young Researchers and Elites club, Science and Research Branch, Islamic Azad University, Tehran, Iran

<sup>2</sup> Light Alloys and Surfaces Treatment Design Centre (CDAL), Universitat Politècnica de Catalunya (UPC), 08800 Vilanova i la Geltrú, Barcelona, Spain

\*Ebrahim.akbarzadeh@estudiant.upc.edu

Tel: +34 93 896 7216, Fax: +34 93 869 7200

## Abstract

High silicon content aluminum alloy (hypereutectic) possess good tribological characteristics with low coefficients of friction, when embedded with short carbon fiber (C<sub>sf</sub>), making this composite a good material choice where good wear and high strength properties are required in light weight components..

There is no previously published information available, to the knowledge of the authors, regarding the influence of wear parameters and their interactions on the tribological behavior of C<sub>sf</sub> reinforced metal matrix composites. In this study a Taguchi design of experiment (DoE) was conducted to optimize and analyze the effects of the wear parameters on the tribological properties of Al/C<sub>sf</sub> metal matrix composite.

A novel thixomixing method which was used to process the metal within the semisolid state was employed to embed short carbon fibers homogenously into the metal matrix. The influences of the sliding speed, applied load and volume fraction, of C<sub>sf</sub> on the specific wear rate and coefficient of friction were examined, with each of these input parameters tested at three levels(0, 4.2, 8.1 %vol.). The results were indicated that Al/C<sub>sf</sub> composite had better tribological properties than Al alloy due to which contains carbon as solid lubricant. According to the statistical analysis, the influence of volume fraction of carbon fiber on wear parameters was ranked first; so the load and sliding speed are at the following rankings. The contribution percentage for each parameter was determined by the analysis of variance. The relatively good interfacial adherence of carbon fiber and matrix alloy were demonstrated. The coherent and adherent graphite-rich layer on the worn surface was characterized using scanning electron microscopy (SEM).

*Keywords: Wear; Aluminum; carbon fiber; thixomixing; coefficient of friction; Design of Experiment.*

## Introduction

Modifications of bulk properties, surface treatments and application of coating have been carried out for reduction of wear of components. Aluminum metal matrix composites (AMMCs) can be used as lightweight materials in aerospace or automobile industries such as piston or cylinder liners due to high

1 strength, good wear resistance, high thermal conductivity and low coefficient of friction [1]. Addition of  
2 carbon fiber improves the machinability and wear resistance similarly with graphite particulates which  
3 provide the good tribological properties with an excellent lubricity and lower coefficient of friction [2,3].  
4 The good mechanical strength and high wear resistance are not only essential for aluminum matrix  
5 composites while self-lubrication properties are also desirable [4]. Owing to these benefits, short carbon  
6 fiber ( $C_{sf}$ ) can be utilized as reinforcement into aluminum silicon and provides high toughness and  
7 strength [5,6]. In wearing of metal-to-metal contact, the carbon fiber plays a role as solid lubricant based  
8 on its graphite microcrystalline composition. The surface of Al/ $C_{sf}$  deformed plastically and the  $C_{sf}$  milled  
9 on the metal surface as small particles and reduced the friction.

10 Most of the previous methods such as powder metallurgy [7], stir casting [8] or squeeze casting  
11 infiltration [9] were not effective to disperse and distribute the carbon fibers into metal matrix. The  
12 thixotropic nature could be useful for dispersion and distribution of particles or even whiskers in the  
13 matrix. The viscosity of slurry is related to the fraction of solid, solid size, shear and temperature. In this  
14 mixing process, viscosity of slurry decreases rapidly during the initial period of shearing, due to the  
15 dissociation of agglomerates of solid particles which are weakly bonded leads to trapping the liquid  
16 within the inter-aggregate spaces. The thixomixing process is theoretically designed based on application  
17 of high shear stress ( $\tau$ ) on the carbon fiber bundles. The semi-solid thixomixing prevents the  
18 reinforcement components to either floating or sinking during the process. The ideal key in this method is  
19 to apply sufficient high shear stress and embed fiber clusters in the semisolid slurry of aluminum and  
20 disperse them in the matrix significantly. Some of few attempts for dispersion of fibers and fragment of  
21 agglomerates have been unsuccessful in the liquid condition of metal [10,11]. They can be described  
22 simply as, solid particles of initial  $\alpha$ -phase are surrounded by liquid eutectic under shearing agitations, the  
23 weakly bindings between solid agglomerations are broken and the viscosity decreases rapidly in early  
24 stages. The temperature of slurry effects on the solid fraction in addition, it will change on the viscosity  
25 [12]. Stirring of partial solidified aluminum produces high shear stress to disperse and displace the short  
26 carbon fibers into the matrix individually, and then revealed a uniform microstructure. It has been  
27 proposed to stirring of slurry which improves incorporation of the short fibers into the alloy while is  
28 solidifying. Semi-solid method has been assumed for longer freezing range alloys which gather energy  
29 savings and good particle dispersion. This method requires break-down of dendritic microstructure of the  
30 feedstock material to globular features which leading to thixotropic properties.

In this present work, the tribological property of hypereutectic Al/C<sub>sf</sub> composite fabricated by a novel thixomixing was studied. Thixomixing process could be effective method to provide a good interfacial adherence due to high silicon content in the slurry, as well as there is no need to apply external coating on the fibers. Due to the lower temperature of process than other fully liquid methods, the aluminum carbide does not form during fabrication. The main and interaction effects have been analyzed by Taguchi design of experiment to find out the effect of carbon fiber, load and speed of sliding and their percentage contribution on wear parameters of Al/C<sub>sf</sub> composite. The similar studies on wear properties using the design of experiment method were conducted with interesting and applicable results [13,14]. Friction and wear behavior of the developed composites was discussed. However, the information regarding to development and characterization of tribological behavior of short carbon fiber reinforced hypereutectic aluminum matrix composites are not studied.

## 2. Materials and Experimental

### 2.1. Test materials

A hypereutectic aluminum silicon alloy (4047); was chosen as matrix with chemical composition Al-11.2Si-0.7Mg-0.65Mn-0.13Cu-2.4Fe-0.1Zn-0.2Ti, in wt. %. The carbon fiber was originally made of polyacrylonitrile (PAN) by Japan Toray Co. were chopped into 5±1 mm length then the agent sizing on the fibers removed by heating in 500 °C for 30 minutes in aerated furnace. The chemical structure of fiber is similar to the graphite [15], consisting of graphene layers stacked parallel on each other [16]. The carbon fiber has high chemical resistance at high temperature [17] but the fibers are fragile. Physical properties of carbon fiber have shown in Table 1.

**Table 1** Physical properties of carbon fiber

| Type  | Diameter<br>( $\mu\text{m}$ ) | Tensile Strength<br>(MPa) | Tensile Modulus<br>(GPa) | Elongation<br>(%) | Mas per Unit Length<br>tex (g/1000m) | Density<br>(g/cm <sup>3</sup> ) |
|-------|-------------------------------|---------------------------|--------------------------|-------------------|--------------------------------------|---------------------------------|
| M40JB | 6                             | 4400                      | 377                      | 1.2               | 454.5                                | 1.75                            |

A novel and specific mixer was designed based on thixotropic property of aluminum, and was made of H13 steel alloy. In the thixomixing process, semi-solid slurry is being sheared and reinforcements (carbon fibers) are dispersed and distributed homogenously into the matrix without any external coating onto the short fibers. One of the advantages of the thixomixing process is to provide good interfacial adherence

1 between aluminum and carbon fiber. This method is similar to Searle rheometer in which a distance gap  
2 of 2 mm between the die walls provides a suitable shear rate ( $\dot{\gamma}$ ) and shear stress (Fig. 1). First, the die  
3 was pre-heated to 700 °C and the molten hypereutectic aluminum silicon alloy was poured into the die.  
4 Based on the diagram of the solid fraction versus the temperature for the 4047 aluminum silicon alloy, a  
5 suitable temperature to achieve appropriate solid fraction for thixomixing was determined to be 560°C.  
6 The chemical resistivity of carbon fiber conforms to the processing temperature of thixomixing. When the  
7 temperature of the die, rotor, and aluminum was maintained at 560°C, the chopped carbon fibers were  
8 added gradually to the mixer with continuous mixing for 10 minutes. Finally, the rotor was removed from  
9 the die and the fabricated composite was cooled to room temperature with no need to recast the samples.  
10 The coupons were cut at an appropriate size from the product of the mixer. The samples were prepared  
11 between a 4.2 and 8.1% volume fraction of the carbon fiber for wear tests. All dendritic structures were  
12 degraded into fine and small grain sizes and converted into spherical shapes due to high shearing load  
13 produced by the mixer.  
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## 28 *2.2. Wear testing*

29 Samples were cut off and the surfaces were polished with abrasive emery paper of 600, 800 and 1000  
30 grades sequentially. The Brinnell hardness measurements were performed by an average of 6 points at  
31 different regions on both aluminum alloy and developed composite using a load of 60 kg for duration of  
32 30 s. A micro-hardness tester (Struers- Duramin 20) was utilized based on standard ASTM E 92-82 using  
33 100 g loads at 10 s time for all the tests. The density measurements were determined using a precision  
34 digital electronic balance (accuracy of 0.0001 mg) and the standard Archimedes principle for both cast  
35 alloy and the fabricated composites with different volume fraction of carbon fiber. Hardness and density  
36 values of the samples were tabulated in Table 2. The pin-on-disc test equipment with counterpart WC-6%  
37 Co spherical pin with diameter of 3 mm (CSEM Tribometer) was utilized during the study. All tests were  
38 carried out in dry conditions without liquid lubrication under 24% relative humidity rate, at 0.05, 0.1 and  
39 0.2 cm/s, the loads of 5, 10 and 20 N with three levels of carbon fiber content, until 10<sup>4</sup> laps completed  
40 according to standard ASTM G-99 of testing. Each composite sample had a rectangular shape of 2×2 cm  
41 and the wear track was a circle with 6 mm diameter. The specific wear rate was calculated using the  
42 transverse area of the worn channel, which was measured by a profile-roughness measuring unit.  
43 Generally, the wear rate ( $W$ ) mm<sup>3</sup>/Km was calculated by using the equation (1),  
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$$W = \Delta V / D \quad (1)$$

where  $\Delta V$  is the volume loss ( $\text{mm}^3$ ) and  $D$  is the sliding distance (Km). The following equation was used to calculate the specific wear rate of the specimen affected by the chemical composition of the exposed material and the environment of the exposure.

$$k = \frac{\Delta v}{F \cdot \Delta s} \quad (2)$$

where  $k$  is wear factor or specific wear rate,  $\text{mm}^3/\text{N.m}$ ,  $\Delta v$  is volume loss,  $\text{mm}^3$ ,  $\Delta s$  is sliding distance,  $m$  and  $F$  is load,  $N$ . It must be mentioned that the equations 1 and 2 were deviations of Archard equation for sliding wear.

The results of coefficient of friction and the specific wear rate of samples were determined and imported to software for further analysis. A total of 9 experimental runs must be conducted and repeated twice times to remove any errors or mistakes, using the combination of levels for each control factor. Analysis of variance and determination of interaction effects and of each erosion parameter was performed by using MINITAB v.16. The worn surfaces and wear debris were characterized using scanning electron microscopy (Jeol JSM-5600) with energy dispersive X-ray (EDAX) analysis.

### 3. Results and Discussion:

#### 3.1. Influence of testing parameters on the main effect plot

Physical properties such as density and hardness values of successful sample after thixomixing were illustrated in Table 2. Differences between theoretical and experimental density refer back to the porosity which exists in the samples.

**Table 2** Physical and Hardness properties of the samples

| Sample                                       | Theoretical Density ( $\text{g}/\text{cm}^3$ ) | Experimental Density ( $\text{g}/\text{cm}^3$ ) | Hardness (HBS) | Micro-hardness (HV0.1) |
|--|--|---|----------------|------------------------|
| $\text{Al}_{\text{hyper}}$                   | 2.6  | 2.59  | 70.1           | 51.4                   |
| $\text{Al}_{\text{hyper}}+4.2\% \text{ C}_f$ | 2.57   | 2.55  | 74.8           | 56.1                   |
| $\text{Al}_{\text{hyper}}+8.1\% \text{ C}_f$ | 2.53   | 2.51  | 85.4           | 59.3                   |

Taguchi method is used in experimental design and provides a simple, powerful tool to determine optimal and effective parameters. In this study, a loss of function was used to determine the quality of a characteristic that was converted into a signal-to-noise ratio (S/N) to reduce the number of interactions.

Table 3 shows each test parameters and arrangement based on Taguchi method for design of experiment.

1 Three different quality characteristics can be calculated based on S/N ratios, namely “Nominal is the  
2 best”, “Larger is better” and “Smaller is better”. Our objective is to minimize the values for specific wear  
3 rate and coefficient of friction to the possible minimum limit so; the S/N ratios for three factors at three  
4 levels are calculated by selected smaller-is-better characteristic:  
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$$\frac{S}{N} = -10 \log \frac{1}{n} (\sum y^2) \quad (3)$$

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10 Where, n is number of observations and y is the response value. The main effect plots on both specific  
11 wear rate and coefficient of friction were plotted for the mean values of S/N ratio, is shown in Fig. 2. The  
12 impact of the three factors at three levels can be studied by using an L9 orthogonal array and reducing the  
13 number of interactions evolved to nine. In general, the best value for each study parameter is the higher  
14 S/N values on the main effect graph, which is an easy way to determine optimal testing conditions [18].  
15 Increasing the volume fraction of carbon fibers in aluminum composites and increase the S/N ratio in the  
16 main effect plot result in a lower specific wear rate (Fig. 2a). But increasing the load and sliding speed  
17 does not significantly affect the wear rate in the main effect plot as indicated in Fig. 2 due to the near-  
18 horizontal line for both mentioned parameters in this type of plot. Wear loss for this composite did not  
19 increase with increases in the applied load and sliding speed, which pertain to the wear debris retained on  
20 the worn surface that covers a greater area of contact. The increase in load causes more erosion and the  
21 formation of wear debris, which in turn leads to the enhancement of forces that impact the wear debris to  
22 form a resistant layer. The detached material during the wear process cannot be eliminated but can be  
23 reduced at appropriate chemical composition and/or a harder microstructure. The increasing of volume  
24 fraction of carbon fiber leads to decrease of wear loss by covering more area of contact, but the applied  
25 load and speed, which indicated twofold action, first remove more materials from the surface in  
26 consequence decreases wear rate by forming debris as a layer on the surface [19]. The wear mechanism is  
27 classified as abrasive wear, erosive wear, adhesive wear, fatigue wear [20]. The adhesive wear  
28 mechanism was proposed for Al/C<sub>sf</sub> composite due to some interaction through the process.  
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49 The main plot of coefficient of friction was exhibited in Fig. 2b and the influence of testing parameters  
50 clearly demonstrated. The S/N ratio corresponding to the coefficient of friction increased with increasing  
51 of volume fraction. The developed composites revealed lower values of coefficient of friction when  
52 compared to the matrix alloy, which indicated the self-lubrication effect of grinding fibers on the surface  
53 and formed the graphite-rich layer. When the counterpart slides over the sample with carbon fibers in the  
54 composite get sheared and ground continuously, at higher load levels which get squeezed between two  
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counting surfaces. The very fine particles of carbon were squeezed out and formed an anti-friction layer with appropriate properties mainly on the coefficient of friction. The adherent tribo-film reduces the coefficient of friction at different sliding speeds, but the film may be broken at very high sliding speeds which are not interested in his study. The coefficient of friction is detected lower when the load of sliding increased in the most studied materials, for instance Ureña et al. [21] have been reported, the lower values of coefficient of friction for carbon fiber-6061 aluminum composite, whereas the fiber was coated with copper and nickel by electrolysis. The high inclination from 0% to 4% in Fig. 2b shows high influence of carbon content in the composite on the coefficient of friction. In addition, increasing of sliding speed and load cannot change the frictional coefficient remarkably in this composite. Consequently, the superior improvement of wear parameters of Al/C<sub>sf</sub> composite versus the matrix alloy can be attributed to:

-enhancement of hardness as plastic deformation resistance and toughness caused to increase of wear resistance.

-No pull out of carbon fibers even at high sliding speeds, shown a good adherence between carbon fiber and matrix alloy which plays a key role in composite materials.

Instead of, the most of individual fibers, is fragmented and ground onto the surface during wear process and formed a graphite tribo-film, which has high load carrying capacity and high lubricity [22]. The high load capacity of carbon fiber will result in reduced plastic deformation and the size of worn particles while sliding at high loads. The larger worn particles indicated high plastic deformation and high wear rate. The improvement of wear resistance with the presence of good bonding at the interface of matrix and fiber has been reported [3,23].

-In addition, some authors reported the formation of mechanical mixed layer by comprising of possible metallic or non-metallic oxides with fine carbon particles [24]. This sticky fine mixture formed a layer and reduces the direct contact between a pair of sliding interfaces and decrease results to lower coefficient of friction. The mechanical mixed layer formation increased with the increasing of volume fraction of carbon fiber in developed composite.

-Good thermal conductivity of carbon fibers (8W/mK, longitudinal) [25] helps to more heat dissipation, and the softening effects will be retarded despite of high velocity of sliding or loads.

**Table 3** Experimental design parameters and results using an L9 (3<sup>3</sup>) orthogonal array

| Run | Volume Fraction (%) | Speed of Sliding (cm/s) | Load (N) | Specific Wear Rate (mm <sup>2</sup> /N *10 <sup>-13</sup> ) | Friction Coefficient | S/N Ratio for Wear Rate | S/N Ratio for Coefficient |
|-----|---------------------|-------------------------|----------|---|----------------------|-------------------------|---------------------------|
|-----|---------------------|-------------------------|----------|---|----------------------|-------------------------|---------------------------|

|    |   |    |    |               |                 |         |  | of Friction |
|----|---|----|----|---------------|-----------------|---------|--|-------------|
| W1 | 0 | 5  | 5  | 5.429 / 5.204 | 0.3931 / 0.3871 | 65.3056 |  | 8.48060     |
| W2 | 0 | 10 | 10 | 5.567 / 5.502 | 0.4417 / 0.4401 | 65.1896 |  | 8.51937     |
| W3 | 0 | 20 | 20 | 5.866 / 5.813 | 0.4776 / 0.4137 | 64.7120 |  | 8.74022     |
| W4 | 4 | 5  | 10 | 4.927 / 4.792 | 0.3708 / 0.3789 | 66.1483 |  | 9.06319     |
| W5 | 4 | 10 | 20 | 5.104 / 5.241 | 0.3673 / 0.3319 | 65.6117 |  | 9.17541     |
| W6 | 4 | 20 | 5  | 5.033 / 4.984 | 0.3365 / 0.3743 | 66.0484 |  | 9.18715     |
| W7 | 8 | 5  | 20 | 3.316 / 3.221 | 0.3262 / 0.3483 | 69.8402 |  | 9.25538     |
| W8 | 8 | 10 | 5  | 3.367 / 3.132 | 0.3428 / 0.3367 | 70.0836 |  | 9.16405     |
| W9 | 8 | 20 | 10 | 3.753 / 3.204 | 0.3745 / 0.3387 | 69.8861 |  | 8.95637     |

**Table 4** Analysis of variance (ANOVA) for S/N ratios

| Responses                            | Source             | DF | Seq. SS | Adj. SS | Adj. MS | F      | P     | Contribution (%) |
|--------------------------------------|--------------------|----|---------|---------|---------|--------|-------|------------------|
| Wear Rate <sup>1</sup>               | Volume of Fraction | 2  | 28.6373 | 28.6373 | 14.3186 | 177.42 | 0.006 | 96.72            |
|                                      | Speed of Sliding   | 2  | 0.6437  | 0.6437  | 0.3219  | 3.99   | 0.200 | 2.17             |
|                                      | Load               | 2  | 0.1654  | 0.1654  | 0.0827  | 1.02   | 0.494 | 0.55             |
|                                      | Error              | 2  | 0.1614  | 0.1614  | 0.0807  |        |       | 0.005            |
|                                      | Total              | 8  | 29.6078 |         |         |        |       | 100              |
| Coefficient of Friction <sup>2</sup> | Volume of Fraction | 2  | 5.5506  | 5.5506  | 2.7753  | 18.45  | 0.048 | 82.46            |
|                                      | Speed of Sliding   | 2  | 0.2488  | 0.2488  | 0.1244  | 0.83   | 0.547 | 3.69             |
|                                      | Load               | 2  | 0.6306  | 0.6306  | 0.3153  | 2.10   | 0.323 | 9.36             |
|                                      | Error              | 2  | 0.3008  | 0.3008  | 0.1504  |        |       | 4.46             |
|                                      | Total              | 8  | 6.7308  |         |         |        |       | 100              |

1)  $S = 0.284089$  R-Sq = 99.45% R-Sq(adj) = 97.82%  
2)  $S = 0.387826$  R-Sq = 95.53% R-Sq(adj) = 82.12%

### 3.2. Interaction plot

Fig. 3 shows the interaction plots which belongs to the parameters in wear process that effectively impress each other as determining roles. The failure of one factor in the response to the same effect on various levels of another factor is called an interaction. As a general, when the interaction lines of a parameter are parallel with other parameter lines, indicated no interaction in against point of view when they have different inclinations, can be detected with interaction. In Fig. 3a shows that the wear rate was increased with increasing of sliding speed and load while the carbon fiber does not exist in sample, whereas the presence of carbon fiber does not change the wear rate with increasing of speed and load. The wear rate followed the same trend when the volume fraction increased regarding the different levels of sliding speed and load. Fig. 3b shows the interaction between parameters on the responses of coefficient of friction. At sliding speed of 10 and 20 cm/s of and high values of load, the coefficient of friction has shown different values due to strong interaction effect between two sliding parameters. The interaction of load and sliding speed on the wear rate and coefficient of friction was determined clearly. The differences in value of responses can be seen clearly in the samples contains carbon fiber and the samples without them. The highest inclination of volume fraction of carbon fiber was appeared as the most significant factor on both wear rate and the coefficient of friction of the composite but has no interactions with the



1 other parameters in wear rate. However, the friction was influenced by variations of sliding distance and  
2 load, because the increasing of applied load leads to stabilization of tribo-film, especially at high load,  
3 and it was destroyed at high sliding speed. The trend of interaction plot was conformed to the information  
4 achieved from main effect plot. In composite of Al/C<sub>sf</sub> produced by thixomixing, the presence of carbon  
5 content has an individual effect on specific wear rate and also on friction coefficient, which improved the  
6 wear resistance of composite when the speed of sliding and load of wear increased.  
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### 11 12 13 14 *3.3. Analysis of Variance*

15 The analysis of variance (ANOVA) table was desirable to understand and determine the order and the  
16 impact of various factors such as volume fraction, load, sliding speed and their interactions. The values of  
17 ANOVA for the prepared thixomixed aluminum/carbon fiber composites were tabulated in Table 4, based  
18 on the degree of freedom (D), the sum of the square (SS), the percentage of the contribution (P), and F-  
19 statistics (F) to evaluate the source of variation during the wear process. The p-value use to detect the  
20 significance of a factor to compare with alpha value of 0.05, so the factor is significant when the p-value  
21 is lower than 0.05. The F-value means that parameters in experiment show a significant effect on optimal  
22 characteristic when the F bigger than 4. The statistical calculation was accomplished by determining the  
23 total variability of the S/N ratios. The effect of order of parameters on wear rate and coefficient of friction  
24 is relatively easy to identify. The Contributions of each design parameters and the errors calculated by  
25 sum of the squared deviations (SS) from total mean values of signal to noise ratio. A very high impact of  
26 volume fraction of carbon fiber on the wear rate (96.72%) and friction coefficient (82.46) and relatively  
27 low influence of load (0.55%) on wear rate were measured (Table 4). It might be observed also the  
28 influence of sliding speed and load on coefficient of friction for this composite.  
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44 The average of each response for each level of the factors has been shown in the response table (Table 5)  
45 which includes a ranking of important factors. The ranking assigned to the important factors and have  
46 been detected by comparison to the relative Delta statistics, which is the difference between averages  
47 (=highest average - lowest average) for each factor. The optimum testing conditions were investigated as  
48 the volume fraction of carbon at 8.1%, load of 5 N and sliding velocity 5 cm/s for the best wear resistance  
49 and the best friction values for the thixomixed Al/C<sub>sf</sub> composite.  
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59 **Table 5** Response table for S/N ratios  
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|                         | Level | Volume fraction | Speed of Sliding | Load  |
|-------------------------|-------|-----------------|------------------|-------|
| Specific Wear Rate      | 1     | 65.07           | 67.10            | 67.15 |
|                         | 2     | 65.94           | 66.96            | 67.07 |
|                         | 3     | 69.94           | 66.88            | 66.72 |
|                         | Delta | 4.87            | 0.22             | 0.42  |
|                         | Rank  | 1               | 3                | 2     |
| Coefficient of Friction | 1     | 8.580           | 8.933            | 8.944 |
|                         | 2     | 9.142           | 8.953            | 8.846 |
|                         | 3     | 9.125           | 8.961            | 9.057 |
|                         | Delta | 0.562           | 0.028            | 0.211 |
|                         | Rank  | 1               | 3                | 2     |

### 3.4. Confirmation test

The final step was to verify the improvement of the quality characteristic using the optimal levels of the design parameters (A3B1C1). The estimated S/N ratio  $\eta$  using the optimal level of the wear process can be calculated as;

$$\eta = \eta_m + \sum_{i=0}^q (\eta_i - \eta_m) \quad (4)$$

where  $\eta_m$  is the total mean of the S/N ratio,  $\eta_i$  is the mean S/N ratio at the optimal level and  $q$  is the number of the main design parameters that significantly affect the performance characteristic.

Statistical analysis can predict the S/N ratio based on the optimal testing parameters which achieved from the last section. The S/N ratio was found for specific wear rate and coefficient of friction to be 70.21 and 9.77 dB, respectively. According to the experimental results obtained from the new practical test, the corresponding values for the same responses are 3.12 and 0.323 (Table 6). This table shows a comparison of values of specific wear rate and coefficient of friction of the predicted and experimented using the optimal parameters. The experimented wear parameters have shown a better improvement than minimum wear parameters at the initial trial (A3B2C1), which meant that the wear process modified for better application. This powerful method of analyzing can be developed for optimizing the other parameters which influence on the wear process. The key role of carbon fiber in reducing of wear parameters was proved in conformation tests of thixomixed Al/C<sub>sf</sub> composite.

**Table 6** Results of the confirmation tests

| Level  | Minimum Initial Responses | Optimal Wear Parameters |            |
|--|---------------------------|-------------------------|------------|
|  |                           | Experiment              | Prediction |
|  | A3B2C1                    | A3B1C1                  | A3B1C1     |
| Wear Rate (mm <sup>3</sup> /N *10 <sup>-13</sup> ) | 3.132                     | 3.128                   | 3.001      |
| S/N Ratio  | 70.08                     | 70.09                   | 70.21      |
| Coefficient of Friction                            | 0.3367                    | 0.3238                  | 0.3203     |
| S/N Ratio  | 9.16                      | 9.79                    | 9.77       |

### 3.5. Microstructural Analysis and Wear Mechanism

1 The initial microstructure of hypereutectic Al/Si alloy was prepared by thixomixing method which is  
2 shown in Fig. 4. The fiber has been dispersed and distributed homogenously in the matrix which shows  
3 relatively good adherence at the interface. During this novel process of compo-casting, some porosities  
4 and air holes were arrested in composite which are indicated clearly, but it can be removed by further  
5 metal shaping such as extrusion or forging. Hypereutectic Al/Si alloys have shown the good tribological  
6 properties, due to containing wear resistance elements such as silicon [26]. It has been reported that  
7 thixoforming methods can increase the wear resistance [27]. But, the presence of iron in the matrix alloy  
8 is not negligible in current thixomixing process due to the dissolution of Fe from the die to the composite  
9 during compo-casting process (Fig. 4b). The  $\beta$ -Al<sub>5</sub>FeSi intermetallic precipitated and forms a needle-like  
10 structure which is hard and brittle and good site for micro cracks initiated, at end increases the wear rate  
11 [28]. The modification elements such manganese (0.6 wt.% ) has been reported to change the flake like  $\beta$ -  
12 intermetallic into star-like  $\alpha$ -intermetallic, and reduce the deleterious effect of Fe and reduce the wear rate  
13 [29,30]. However, the presence of iron in the Al/Si alloy improves the high temperature properties and the  
14 thermal stability of the alloy [31].

15 Fig. 5a shows typical SEM micrographs of the worn surface of the Al-4.2% C<sub>sf</sub> composite clearly  
16 exhibited the deep, permanent grooves related to high speed of sliding and also the tribo-film formed on  
17 the surface. These wear mechanisms were produced by the ploughing and formed the relatively deep  
18 grooves made by two asperities as counter pin and the hardened worn debris such as silicon or metal  
19 oxides. During the milling, the loose small particles were packed tightly layer over layer and an adherent  
20 smooth graphite-rich film formed over the track area. The micro-hardness values increased from 56 HV  
21 for un-tracked area of composite to 67 HV for track area which enhanced 20%, on the other hand the  
22 hardness was increased just 6% of the matrix samples when the micro-hardness increased from 51 to 54.3  
23 for un-tracked and tracked area, respectively. Some of the residual worn debris which remained unpacked  
24 contains smaller amounts of oxide particles and small size microcrystals of graphite were at the surface  
25 eventually leads to exhibit the slight plastic deformation. The smooth surface at tracking area and the  
26 smaller size of debris are cause of the carbon content and the lower coefficient of friction. It has been  
27 reported that the temperature in contact area is an important influential parameter on the wear mechanism  
28 during the wear test [18]. When the coefficient of friction decreased the temperature will be decreased so  
29 the adherence between two interfaces, reduced. However, the worn particle sizes become smaller at a  
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1 lower temperature because of the high thermal conductivity of composite and solid lubrication of carbon  
2 content in the composite. In Fig. 5b the lighter region at the micrograph is belonging to iron intermetallic  
3 with some carbon fibers remained on the wear track. In spite, the absence of any coating on the carbon  
4 fiber, the good adherence to matrix and no pull out of the fiber was shown in the microscopy illustrations.  
5 The wear track areas at lower resolution have been shown in Fig. 6a and 6c while the difference in the  
6 wear mechanism illustrated clearly. Wear process produces heat and has been dissipated much more when  
7 carbon fiber presented in composite samples and slightly plastic deformation will occur. The coherent  
8 surface in track area without any detachments exhibited in Fig 6a and 6b, in contrast a severe plastic  
9 deformation undergone in Fig.6c and big detachments revealed the softening effect due to the  
10 enhancement of temperature. While the wear is processed, the dislocations were accumulated under the  
11 track area and formed the cracks at a certain depth which is parallel to the surface, so they become  
12 stretched and expand to the critical length. Finally the wear surface between expanded cracks and surface  
13 is being peeled like wear debris and the fresh layer under it oxidized as shown in Fig. 6c. The morphology  
14 of the worn surface revealed that some materials were removed due to abrasive wear. Fig. 6d shows the  
15 cross sectional area of Al-8.1% C<sub>sf</sub> composite worn surface under sliding speed 20 cm/s and load 10 N.  
16 This microscopy illustration proves the presence of carbon fiber at wear track and good adherence to the  
17 matrix which were not pulled out. Additionally, the fibers have been ground and the very small size  
18 graphite particles were tightly packed with small loose oxide particles and formed an adherent film over  
19 the worn surface. The asperities of the hard worn debris and counter pin caused the formation of grooves  
20 like ploughing and can be seen in the cross sectional view.

21 Fig. 7 shows the EDAX pattern of the worn surface of matrix alloy and composite sample contains 4.1  
22 Vol.% of C<sub>sf</sub>. The EDAX of the track area of composite sample shows that C and O are present, as Fig. 7b  
23 represents. In the EDAX spectrum of the C<sub>sf</sub>/Al (Fig. 7b), the peak belongs to O appeared lower than that  
24 of the matrix alloy (Fig. 7a), because of the low temperature caused by the C<sub>sf</sub>. Fig. 8 shows the mapping  
25 EDAX of a worn track area of hypereutectic silicon aluminum alloy/C<sub>sf</sub> composite. The mapping EDAX  
26 analysis contains bright and dark spots which reveal them to be rich of the individual elements in that  
27 point. The carbon and oxygen in worn surface came from the carbon fiber, which ground on tracking area  
28 and the oxidized debris which was composed of materials from composite and oxidation of aluminum by  
29 air.

## Conclusion

1 This experimental work has been carried out based on the Taguchi method which is an efficient  
2 methodology for optimization and analysis of results. The novel thixomixing has been conducted to  
3 produce hypereutectic aluminum/carbon fiber composite which is applicable for lightweight components  
4 with self-lubrication and good tribological properties. Thixomixing can be utilized to fabricate aluminum  
5 metal composite reinforced by SiC, SiO<sub>2</sub> and alumina powder. The following conclusions may be drawn  
6 for studying factors on the wear parameters:  
7

8 -The presence of carbon fiber can reduce the specific wear rate and coefficient of friction due to forming  
9 rich-graphite layer in the wear track and increase the thermal conductivity and reduce softening effect.  
10

11 -The thixomixing provides relatively good adherence at the interface of C<sub>sf</sub> and matrix alloy. No pull out  
12 of carbon fiber was investigated and was ground onto the metal surface which affect as solid lubricant.  
13

14 -The developed composite shows a lower influence on the coefficient of friction and wear rates when  
15 compared to the matrix alloy while the load and sliding velocities changed. The coefficient of friction  
16 decreased initially and then increased after an optimal level. However, the thixomixed composite  
17 represented a lower coefficient of friction and wear rates for comparing the matrix alloy at all levels of  
18 loads and sliding speeds were studied.  
19

20 -According to ANOVA, the carbon fiber volume fraction has the most significant effect on the wear  
21 parameters. The contribution percentages are 96.72% and 82.46% for specific wear rate and coefficient of  
22 friction, respectively. The optimal conditions for best results have been predicted and the confirmation  
23 experiments are conducted. The wear parameters were improved comparing with the obtained initial  
24 values.  
25

26 - In order to find out the wear mechanism, the surface deformations of the worn surfaces were compared  
27 with SEM views. The SEM morphology of the worn surface indicated the smooth and high graphite layer  
28 in the track area with the small debris particles causing low plastic deformation.  
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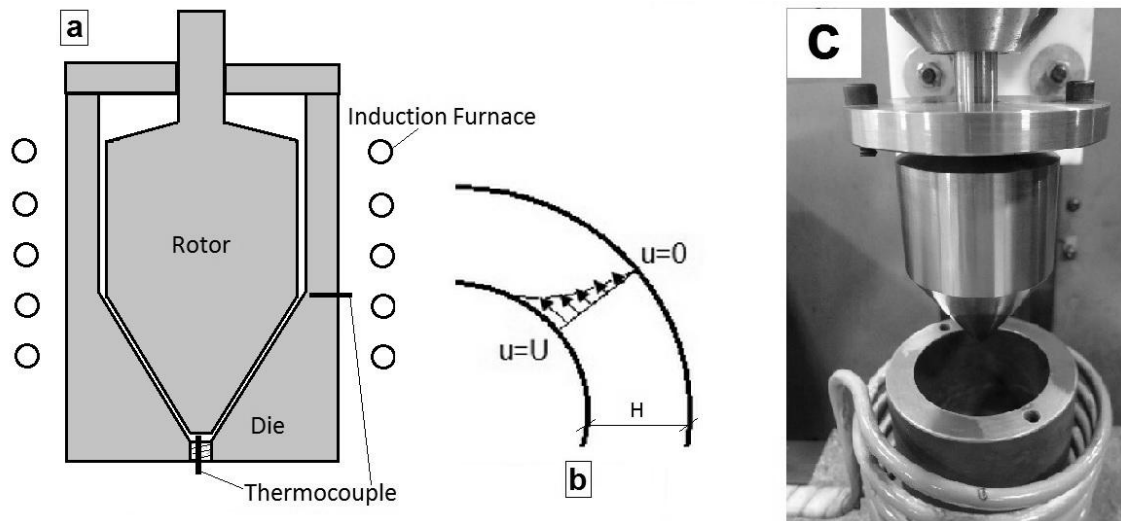
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33 for their kind and helpful collaboration regarding this study.  
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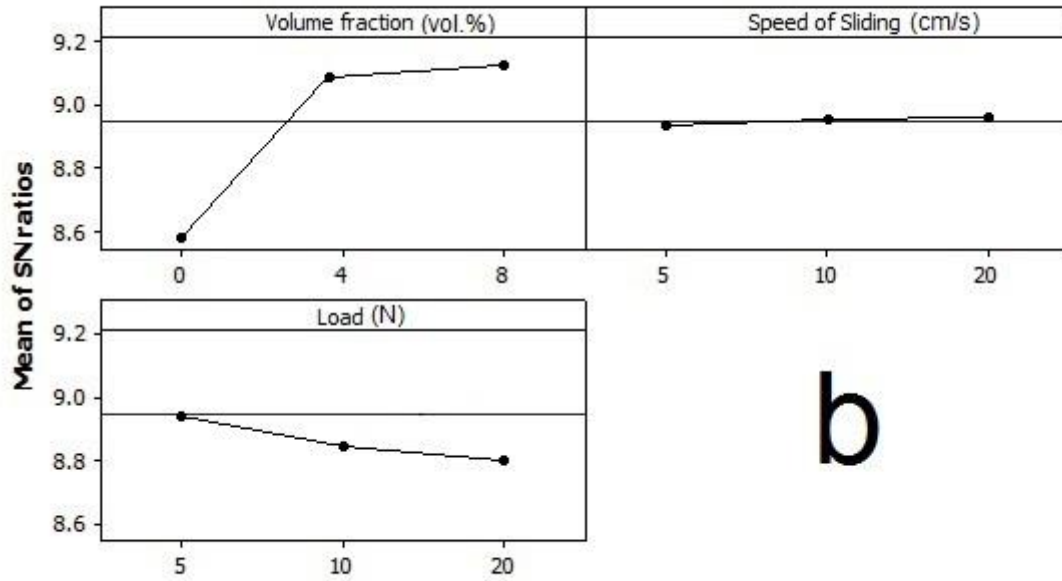
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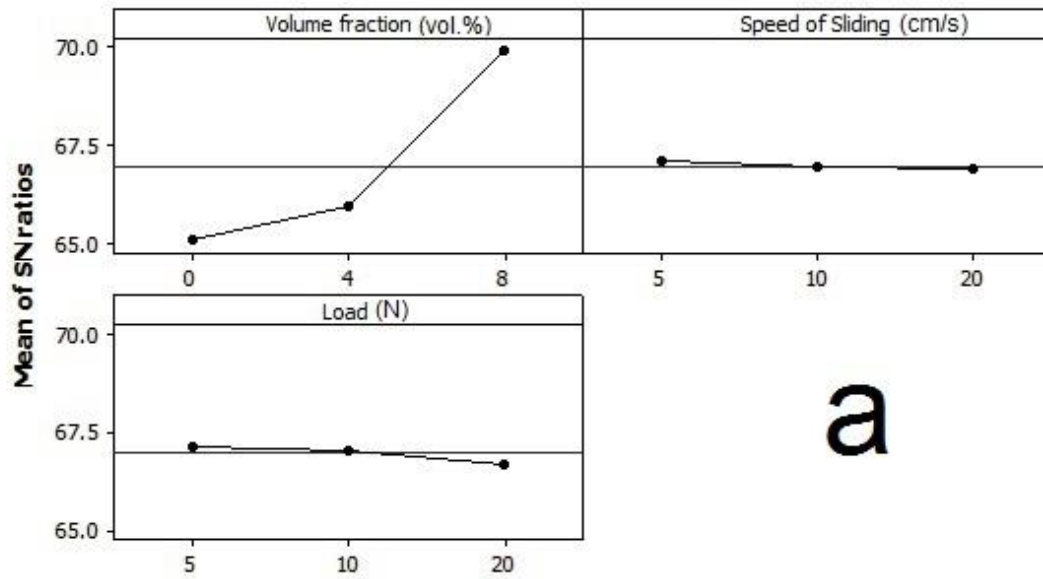


**Fig. 1** A schematic illustration of designed thixomixer, (a) the inner part (Rotor) is rotating and the outer cylinder (Die) is stationary (b) schematic of flow in present system in the gap between two parallel plates and, (c) real image of mixer.



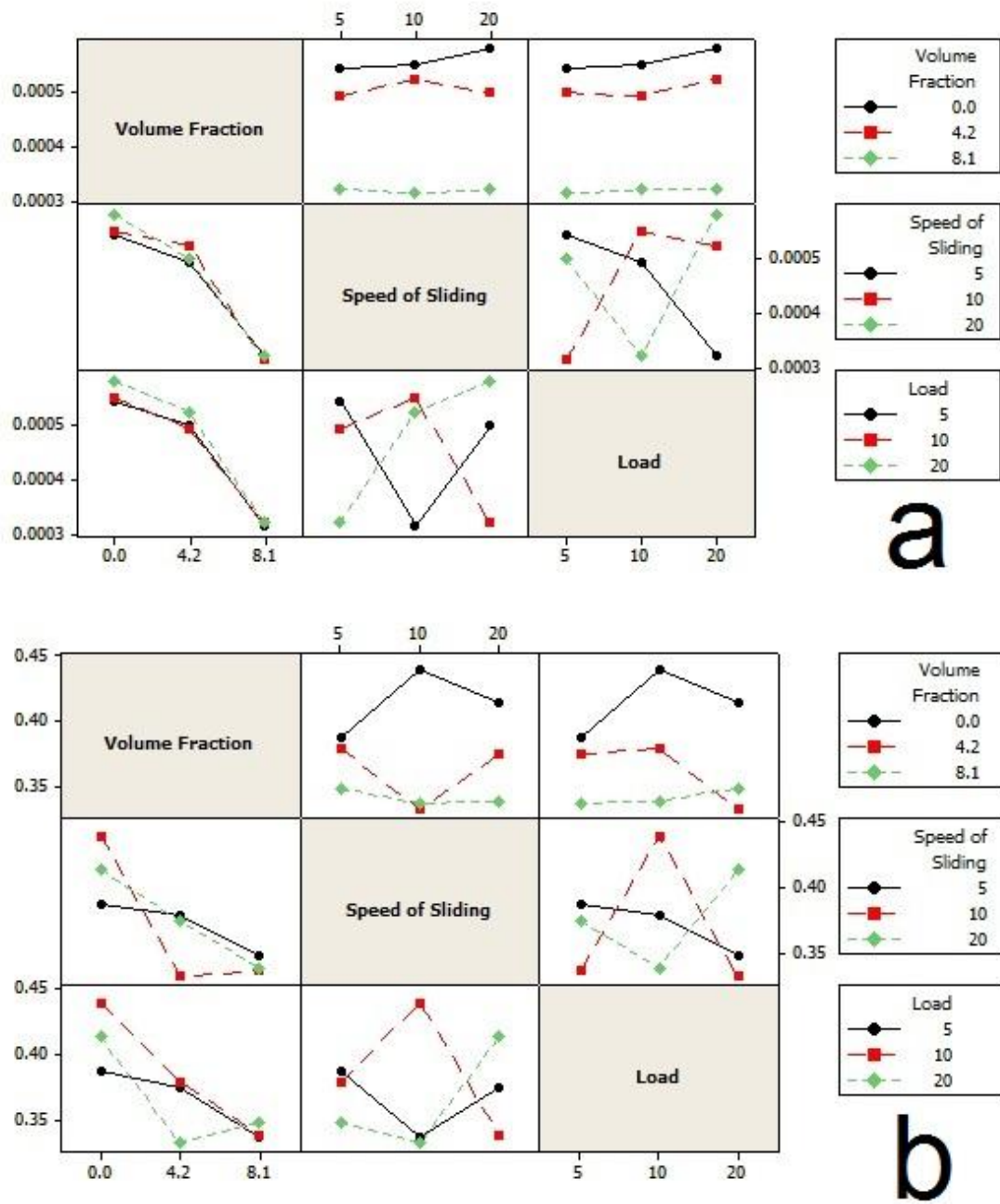


Signal-to-noise: Smaller is better

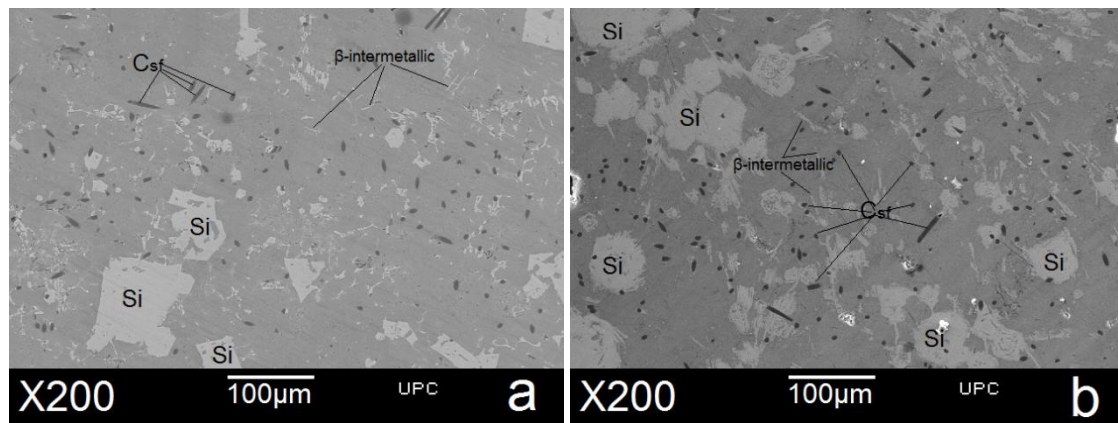


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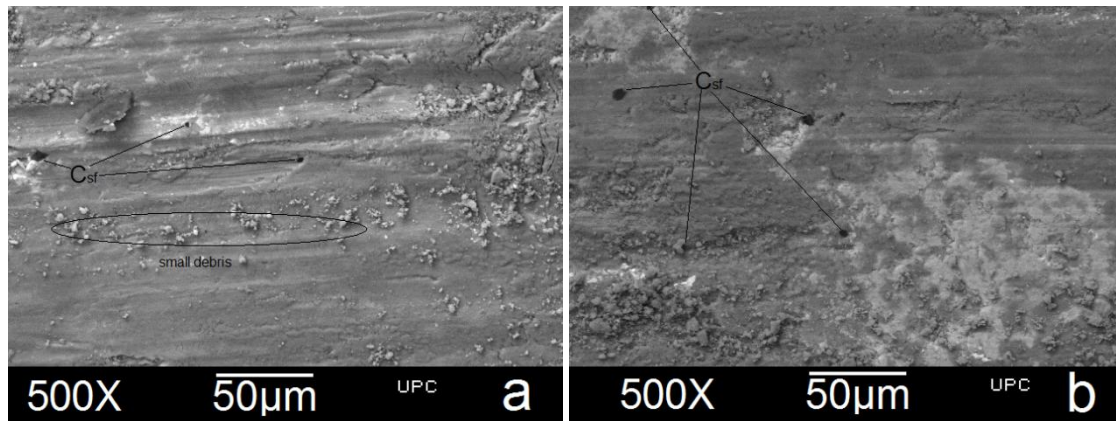
**Fig. 2** the main effect plots for (a) specific wear rate and (b) Coefficient of friction



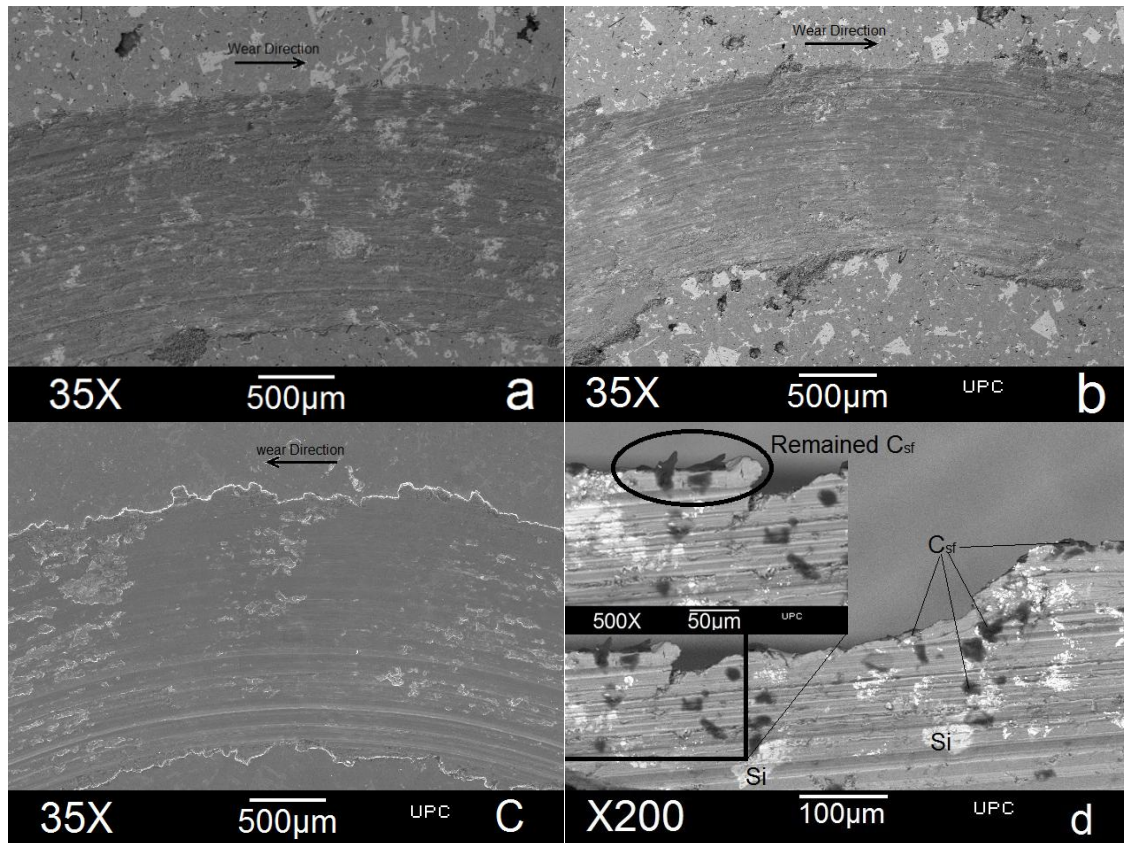
**Fig. 3** Interaction plots for (a) specific wear rate and (b) Coefficient of friction (m)



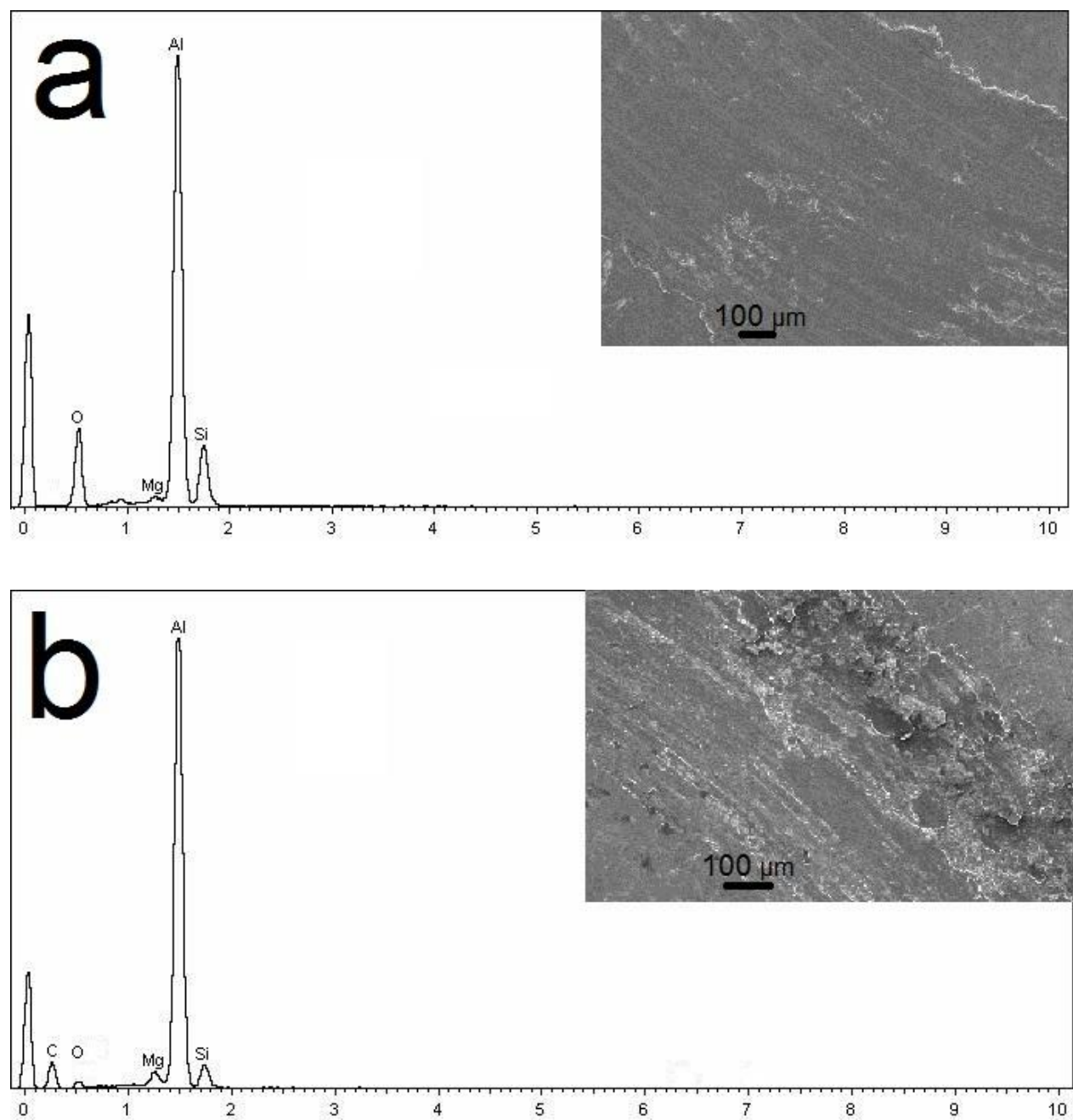
**Fig. 4** SEM morphologies of samples before wearing (a) Al-4.2% C<sub>sf</sub> composite (b) Al-8.1% C<sub>sf</sub> composite.



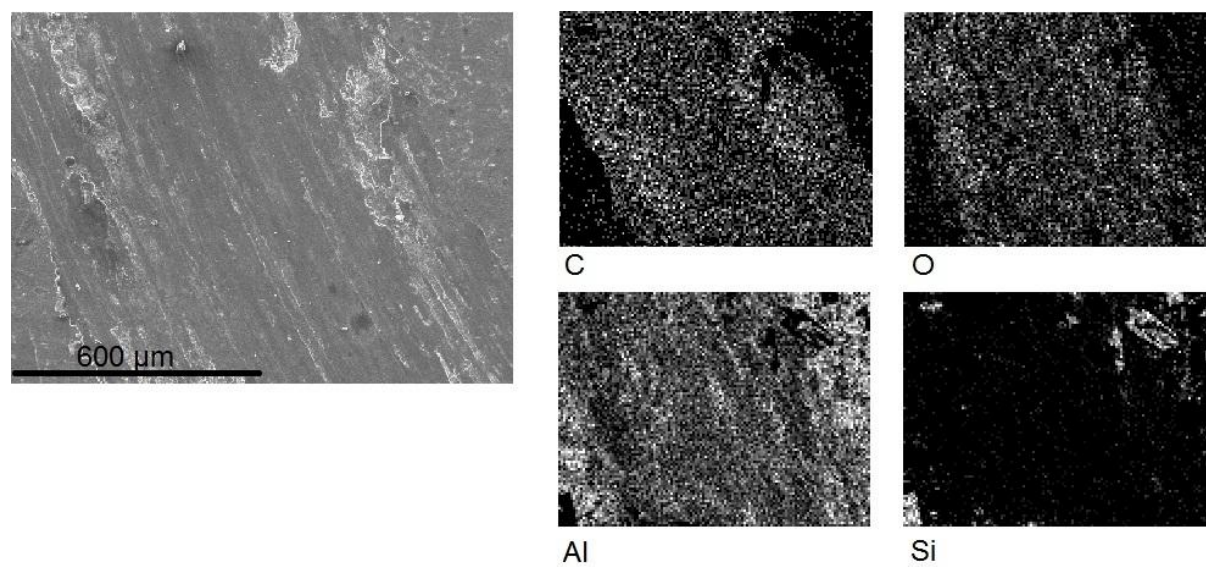
**Fig. 5** SEM morphologies of (a) Al-4.2% C<sub>sf</sub> composite at sliding speed 20 cm/s and load 5 N (b) Al-8.1% C<sub>sf</sub> composite at sliding speed 10 cm/s and load 5 N.



**Fig. 6** SEM morphology of worn track surface for (a) Al-8.1% C<sub>sf</sub> composite at sliding speed 20 cm/s and load 10 N (b) Al-4.2% C<sub>sf</sub> composite at sliding speed 20 cm/s and load 5 N (c) matrix alloy without carbon fiber (d) cross section of worn track of Al-8.1% C<sub>sf</sub> composite.



**Fig. 7** EDAX with SEM pattern of worn surfaces area of a) hypereutectic silicon aluminum as matrix alloy and b) hypereutectic silicon aluminum/ C<sub>sf</sub> 4.1 Vol.% composite



**Fig. 8** Element distribution from mapping EDAX of worn surface of aluminum/4.1 Vol.% C<sub>sf</sub> Composite