

## Research Article

# Friction Reduction Capabilities of Silicate Compounds Used in an Engine Lubricant on Worn Surfaces

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Effects of magnesium silicate and alumina dispersed in engine lubricant on friction, wear, and tribosurface characteristics are studied under boundary and mixed lubrication conditions. Magnesium silicate and alumina, henceforth called as friction reducing compounds (FRC), were dispersed in engine lubricant in very low concentration of 0.01% weight/volume. Four-ball wear test rig was used to assess friction coefficient and wear scar diameter of balls lubricated with and without FRC based engine lubricant. Scanning electron microscopy (SEM) equipped with Energy Dispersive X-ray (EDX) was used to analyse the tribosurface properties and elemental distributions on worn surfaces of the balls. Test results revealed that FRC based engine lubricant increases friction coefficient but marginally reduces wear scar diameter of new balls, whereas, test on the worn-out balls running on FRC based engine lubricants shows 46% reduction in friction coefficient compared to the new balls running on engine lubricants without FRC. Investigations on tribosurfaces with respect to morphology and elemental distribution showed the presence of Si and O elements in micropores of the worn surfaces of the balls, indicating role of FRC in friction coefficient reduction and antiwear properties. These FRC based engine lubricants may be used in the in-use engines.

## 1. Introduction

In the recent past a number of original equipment manufacturers (OEMs) recommended a lower viscosity grade engine lubricant to improve fuel economy of their vehicles. Effect of engine lubricant viscosity on fuel consumption was studied by many researchers and it has been reported that lower viscosity grade engine lubricants result in reduction in engine fuel consumption [1, 2]. But there are some concerns associated with the use of a low viscosity grade engine lubricant in terms of wear characteristics and durability of an engine. Most vulnerable parts of an engine prone to wear, associated with lower viscosity grade engine lubricants, are the surfaces operating in boundary lubrication regime such as cam and follower of valve train system, top dead centre (TDC), and bottom dead centre (BDC) of a cylinder liner. Therefore, these low viscosity grade engine lubricants require some sort of surface modifiers which must act on the tribosurfaces operating in boundary lubrication regime via physical or chemical adsorption and forming a protective tribofilm, able to reduce friction coefficient and preventing the excessive wear of the engine parts during engine operation.

During the last decade, many researchers have studied serpentine mineral, very similar to the FRC used in this study, as a potential surface modifier possessing excellent tribological characteristics. Serpentine mineral, basically a clay material, is stoichiometrically represented by the chemical formula of magnesium silicate hydroxide,  $Mg_6Si_4O_{10}(OH)_8$ . Serpentine-group minerals are composed of chrysotile, lizardite, and antigorite, and based on the analysis it emerged that these are either members of a complex solid solution series or are separate chemical species, but are not simply polymorphs [3]. Yuansheng et al. [4] had put some light on the basic structure of  $Mg_6Si_4O_{10}(OH)_8$ ; it was shown that Si-O tetrahedron plane and the Mg-O/OH octahedron plane form the layered structure via cross-linkage between them. A concept of *in situ* mechanochemical reconditioning of worn tribosurfaces of a locomotive diesel engine using serpentine powder added in engine oil in the ratio of 5 mg/mL, with particle size not larger than  $2\ \mu\text{m}$ , was also explained. Nanohardness of protective layer formed was reported to be twice as high as that of the substrate and surface roughness of  $0.0694\ \mu\text{m}$ , coefficient of friction between real piston ring and

cylinder bore, was stabilized to 0.005. Yu et al. [5] studied the compositions of a thin tribofilm generated on worn surface by using 1.5 weight% serpentine powders, with an average size of  $1\ \mu\text{m}$ , dispersed in SN 500 mineral base oil. It was reported that a nanocrystalline tribofilm was generated on worn surface with a thickness of 500–600 nm, mainly composed of  $\text{Fe}_3\text{O}_4$ ,  $\text{FeSi}$ ,  $\text{SiO}_2$ ,  $\text{AlFe}$ , and  $\text{Fe-C}$  compounds ( $\text{Fe}_3\text{C}$ ). Zhang et al. [6] investigated the tribological characteristics and self-repairing effect of 5% hydroxy-magnesium silicate (HMS) with average diameter about  $0.5\ \mu\text{m}$ , dispersed in gasoline engine lubricant SJ 10W40 on steel-to-steel friction pairs “plane on plane” configuration with various surface roughness; it was stated that wear resistance of HMS self-repairing material has a relationship with the surface roughness of friction pairs and the average diameter of self-repairing material particle. When the powder average diameter is close to the surface roughness value of friction pairs, the HMS self-repairing material shows friction reduction, antiwear, and self-repairing properties. Nan et al. [7] also used 0.5 wt% as an optimum concentration of ultrafine magnesium aluminum silicate powders, as lubricant additive in SRV oscillating friction and wear tester and found that ultrafine powder was very efficient in reducing friction and wear at different loads (10 N–100 N). Tribological characteristics of serpentine powder used in engine oil or base oil with respect to formation of tribofilm, surface modifying capabilities, reduction of friction coefficient, and wear was extensively studied by many other researchers [8–13]. Similar results on reduction of friction coefficients by 24.63% and diameters of friction spots by 41.88% as compared to the base oil were reported by Zhao et al. [14]. Lyubimov et al. [15] reported the merits of Kaolin based powder over serpentine powder in terms of tribological characteristics.

In contrast to other researchers, an alternate mechanism of the self-repairing function was studied by Yue et al. [16]. According to his work, aluminum and silicon elements of silicate particles were not found in the repaired layer, hypothesizing that the silicate particles did not participate in the formation of the repaired layer rather it act as a catalyst to promote a series of complex tribochemical reactions to form a regenerated layer with amorphous carbon structure on the worn surface under high-friction temperature and pressure in the friction and wear process.

Qi et al. [17, 18] proposed generation mechanism of tribofilm formed by nanoscale serpentine powder (average size  $< 100\ \text{nm}$ ) at a high temperature of  $400^\circ\text{C}$ . Effect of thermal activation on tribological characteristics of serpentine ultrafine powder added in liquid paraffin was studied by Yu et al. [19]; it was found that a temperature range from  $300^\circ\text{C}$  to  $600^\circ\text{C}$  increases the film forming ability of the serpentine and at higher temperature beyond  $850^\circ\text{C}$  the layer structure is destroyed and aggravated the friction and wear.

Based on the literature review it may be inferred that serpentine mineral was added in engine lubricant or base oil by researchers in concentration ranging from 0.025% to 5%, normal load applied in different tribometers was varying from 50 N to 200 N, and experiments were conducted, either at very high temperature ranging from  $300^\circ\text{C}$  to  $600^\circ\text{C}$  or at room temperatures. In this paper a four-ball wear test rig

was used to investigate the lubricity properties of FRC based engine lubricant, applied load on balls was around 490 N, and engine lubricant temperature was controlled at  $90^\circ\text{C}$ , simulating the boundary lubrication condition of a typical cam and follower of valve train system of an internal combustion engine. A very low concentration (0.01%) weight/volume of FRC (a mixture of Mg silicates hydroxide and alumina) was added in an engine lubricant SAE 5W-30, API SL/CF. SEM-EDX was used to examine and analyse the role of FRC in influencing surface properties of worn surfaces.

## 2. Experimental Set Up

**2.1. Engine Lubricants Sample Preparation.** Friction reducing compound was synthesized by crushing and mixing Mg silicates, alumina, and some catalyst together in pestle mortar. Chemically the main constituent of the FRC powder belongs to the serpentine family, which is stoichiometrically represented by the chemical formula of magnesium silicate hydroxide,  $\text{Mg}_6\text{Si}_4\text{O}_{10}(\text{OH})_8$ . The average particle size of FRC was around  $9\ \mu\text{m}$ . Engine lubricant sample for the experiments was prepared by adding 1 mg of FRC in 10 mL of engine lubricant, SAE 5W-30, API CF/SL. Engine lubricant sample comprising of FRC was prepared by first stirring it using a magnetic stirrer at 2000 rpm followed by an ultrasonic bath treatment for one hour, keeping bath temperature at  $50^\circ\text{C}$ . Following engine lubricants samples were used for the study:

Oil A, SAE 5W-30,

Oil A1, 0.1 mg of FRC added in 1 mL of SAE 5W-30.

It was also observed that by adding FRC in low concentrations of 0.1 mg/mL, it does not affect physicochemical characteristic of the engine lubricants adversely.

**2.2. Rolling Contact Fatigue Four Ball Test Rig.** The tribological performance of lubricants was carried out on a four-ball tribotester shown in Figure 1. The tribotester utilizes four-ball geometry in a trapezoidal form. The top ball is fixed into the spindle and rotates at the predefined speed, that is, 1200 rpm. The bottom three balls are fixed in a ball cradle filled with lubricant. The four balls make three-point contacts. The test conditions employed are given in Table 1. These operating conditions were slightly different from that prescribed in ASTM D 4172, applied load was kept as 50 kg instead of 40 kg, and oil temperature was kept as  $90^\circ\text{C}$  in place of  $54^\circ\text{C}$ , to adequately represent the boundary lubrication conditions of a typical cam and follower of valve train system of an internal combustion engine. Friction encountered within the contact is continuously monitored and recorded using the data acquisition software.

**2.3. SEM-EDX.** FESEM (Field Emission Scanning Electron Microscope) with EDX/EDS (Energy Dispersive X-ray/Spectroscopy) system was used to examine and analyse the morphology and elemental distributions of worn surfaces of the balls.

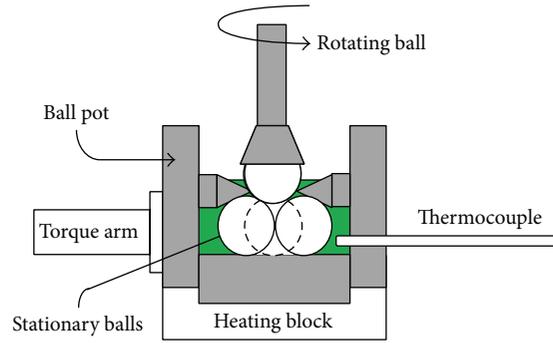


FIGURE 1: Four-ball tribotester.

TABLE 1: Test operating conditions of four-ball test rig.

1	Load (kgf)	50
2	Ball specimen diameter (mm)	12.7
3	Speed (rpm)	1200
4	Oil temperature ( $^{\circ}$ C)	90
5	Ball type	EN 31 steel
6	Ball roughness $R_a$ (microns)	0.6
7	Composition of EN 31 steel	C: 1%, Mn: 0.50%, Cr: 1.40%, Si: 0.2%

TABLE 2: Test matrix of the experiment.

Test number	Duration in min	Type of balls	Engine lubricants
1	60	New	Oil A
2	60	New	Oil A1
3	120	New	Oil A
4	120	New	Oil A1
5a	60	New	Oil A
5b	75	Balls used in test 5a	Oil A1

### 3. Test Matrix and Methodology

A comparative performance of the engine lubricants without FRC (Oil A) and with FRC (Oil A1) was conducted on a four-ball test rig with respect to friction coefficient, wear scar diameter, and surface properties. Test matrix of the experimental study is shown in Table 2. A total of five tests were conducted, first two tests were carried out on new balls using Oil A and Oil A1 each of one hour' duration, next two tests were conducted on new balls for two hours each to study the effect of extended duration on friction coefficient and wear, and last test was conducted in two phases, initially with Oil A (test number 5a) for one hour, then draining Oil A without disturbing the balls positions and refilling it with Oil A1 for carrying out the test 5b on same used balls till friction coefficient gets stabilized.

Bench marking of test rig in terms of friction coefficient and wear scar diameter of balls was done in test number 1 using Oil A on new balls for one hour' duration, followed

by test number 2 using Oil A1 on new balls for one hour. These tests were conducted in order to examine the efficacy of FRC, added in engine lubricant in very low concentration, on friction coefficient, wear scar diameter, and surface properties of new balls specimens.

In order to investigate the effect of test duration on friction coefficient, wear scar diameter, and surface properties, two more experiments (experiments numbers 3 and 4) were conducted on new balls using Oil A and Oil A1 for extended test duration of two hours each.

Finally, test number 5a was conducted on new balls using Oil A for one hour followed by test number 5b on same set of used balls (worn-out balls without changing the tribosurfaces of interacting balls) that were undertaken using Oil A1 until stabilization of friction coefficient. Friction coefficient stabilization was reached within 1 hour and 15 min (4500 sec). This test was carried out to investigate the effect of FRC added in engine lubricant, that is, Oil A1, on worn-out balls with respect to friction coefficient, wear scar, and surface properties.

Friction coefficient for each test was obtained from the measured value of friction torque of the contact surface geometry of the balls by a data logging system.

Wear scar diameter (WSD) in mm, for each oil sample, was calculated by taking the average worn scars of all three stationary balls. Morphology and elemental analysis of the worn surfaces of the balls was done by selecting a ball among the three stationary balls randomly followed by the SEM and EDX analysis on it, to understand the role of FRC on the surface properties.

### 4. Result and Discussion

**4.1. Friction Coefficient and Wear Characteristics.** Friction coefficients of the ball surfaces along with the standard deviation and wear scar diameter (WSD) results of all tests on rolling contact fatigue four-ball test rig are shown in Table 3.

**4.1.1. Effect of FRC on Friction Coefficient and WSD of New Balls.** It is observed from Table 3 that the addition of FRC in engine lubricant, even in very low concentration (0.01%), can influence friction coefficient of tribosurfaces. Wear scar diameters of balls run on both engine lubricants (Oil A and Oil A1) were almost similar during the one hour' test.

TABLE 3: Stabilized friction coefficient and wear scar diameter.

Test number	Duration in min	Types of balls	Engine lubricants	Stabilized friction coefficient	Standard deviations	Wear scar diameter (WSD), mm
1	60	New	Oil A	0.109	0.015	0.525
2	60	New	Oil A1	0.142	0.015	0.525
3	120	New	Oil A	0.098	0.009	0.650
4	120	New	Oil A1	0.131	0.015	0.600
5a	60	New	Oil A	0.109	0.012	0.660
5b	75	Balls used in test 5a	Oil A1	0.076	0.020	0.660

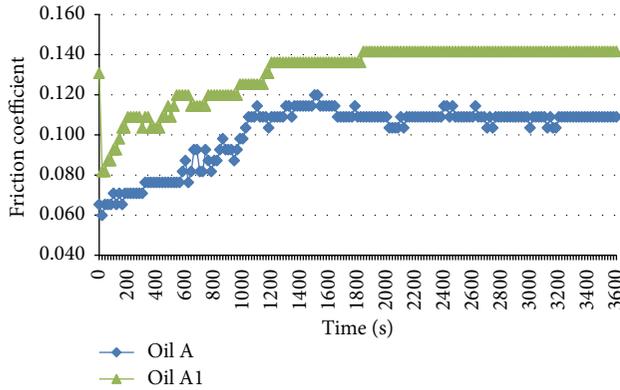


FIGURE 2: Friction coefficient variation with time using new balls (tests 1 and 2).

Figure 2 shows the comparative results of friction coefficient of new balls running on Oil A and Oil A1 each for one hour' duration. Comparative test results revealed that FRC based engine lubricant that is Oil A1 aggravates the friction between the interacting balls, resulting in higher values of friction coefficient as compared to Oil A, which showed lower values of friction coefficient, throughout the test. This may be explained with the fact that Mg silicate particles may act as abrasives on smooth surfaces of new balls having surface roughness values of  $R_a = 0.6 \mu\text{m}$ , which is much lower than the average size of FRC particles, that is,  $9 \mu\text{m}$ . Bigger diameter FRC particle may bring certain degree of grain abrasion during the test, which enhances frictional force between interacting surface of balls resulting in increase in friction coefficients. Hence, surface roughness and FRC particle size play a vital role in influencing friction; this finding is consistent with the study done by Zhang et al. [6]. Friction coefficient values varied from 0.082 to 0.142 for Oil A1 and for Oil A; it varied from 0.065 to 0.109 and according to Stribeck curve [20], friction pairs with friction coefficient  $\mu \geq 0.10$  were considered as operating in boundary lubrication conditions, and those with  $0.001 < \mu < 0.10$  were assumed to operate in mixed lubrication regime. So inferences may be drawn from these results that the lubrication regime of interacting balls transformed from mixed to boundary for Oil A1 owing to presence of FRC, whereas lubrication regime for Oil A in test 1 predominantly in mixed lubrication only.

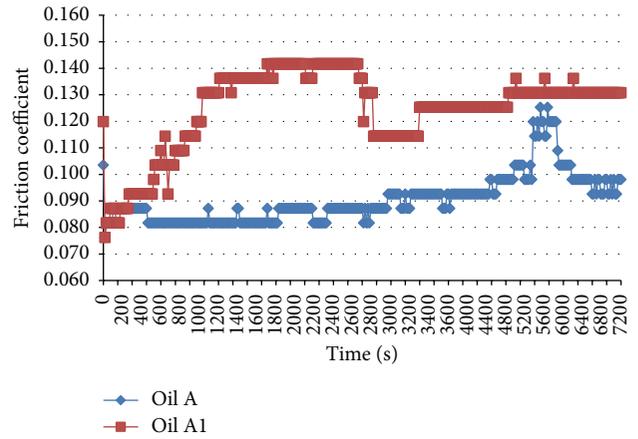


FIGURE 3: Friction coefficient variation with time using new balls (tests 3 and 4).

**4.1.2. Effect of Extended Test Duration on Friction Coefficient and WSD of New Balls.** Another set of test was conducted on new balls for extended time duration of two hours, lubricated with Oil A and Oil A1 (tests numbers 3 and 4). These tests were conducted to investigate the role of FRC in formation of protective film on tribosurfaces if any, which reduces the friction coefficient. It is observed from Figure 3 that the friction coefficient values for Oil A1 keep on increasing from 0.076 to 0.131, during the whole test and stabilized at 0.131 which was very close to the stabilized value of 0.142 obtained for Oil A1 during the one hour' test (test number 2). This increasing trend of friction coefficient value negated the idea of friction reduction capabilities of FRC. On the contrary it may be inferred from the results of tests numbers 2 and 4, shown in Figures 2 and 3, that FRC based engine lubricant enhances the friction coefficient of new balls under the prescribed operating conditions irrespective of test duration. Wear performance of FRC in terms of wear scar diameter (WSD) shows marginal reduction in WSD values, that is, from 0.650 for Oil A to 0.600 for Oil A1, as indicated in Table 3.

**4.1.3. Effect of FRC on Friction Coefficient and WSD of Used Balls.** Test 5 was carried out in two phases, 5a and 5b; during first phase, friction coefficient value which varied from 0.082 to 0.109 for Oil A as shown in Figure 4 and

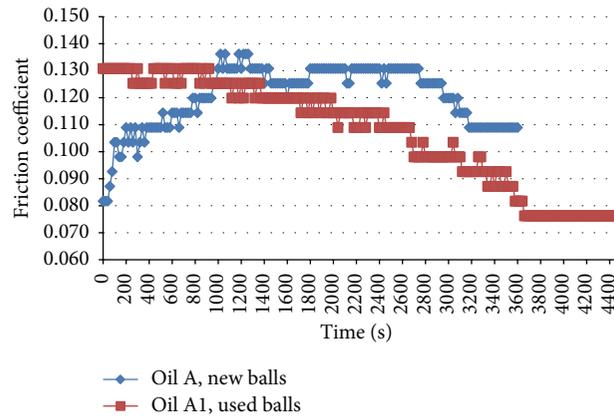


FIGURE 4: Friction coefficient variation with time using worn-out balls (tests 5a and 5b).

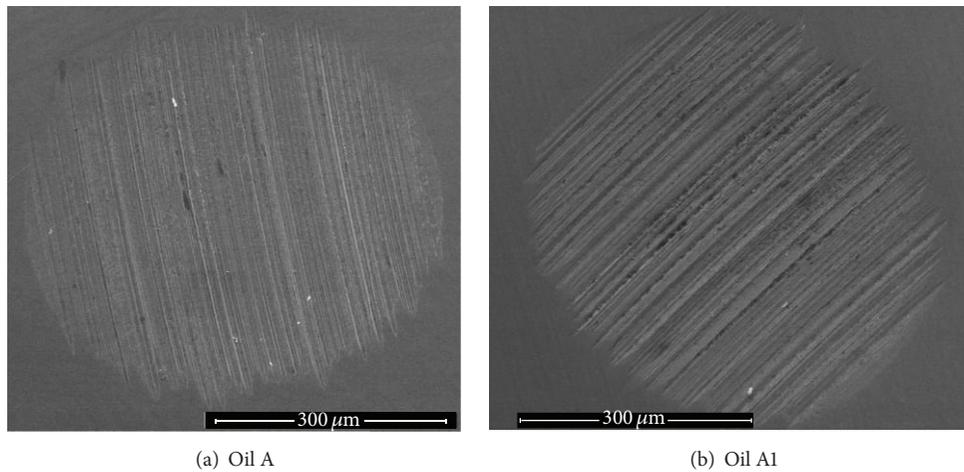


FIGURE 5: SEM images of ball after 1 hr test (a) ball run on Oil A and (b) ball run on Oil A1.

when Oil A1 was used in second phase of test on the same set used balls (worn surfaces), and friction coefficient values were continuously decreasing from 0.131 to 0.076 and stabilized at 0.076 after 75 minutes. By this test it was demonstrated that FRC based engine lubricants, that is, Oil A1, performed as an excellent friction reducing compound for the worn surfaces. Lubrication regime also transformed from boundary condition to mixed lubrication condition. It may be depicted from these results that FRC may acts as friction reducing compound when used on worn surfaces and significantly reduces the friction coefficient. Further it is hypothesized that on worn surfaces, FRC particles may get adsorbed and embedded into the micropits or furrows and showed some reaction activity on interacting surfaces culminating into much smoother surfaces and ultimately helps in friction reduction. It is assumed that some sort of friction reducing film may be formed on the interacting ball surfaces which help in friction coefficient reduction.

#### 4.2. Surface Morphology and Elemental Composition

4.2.1. *Effect of FRC on Surface Morphology.* SEM images of worn surface of balls run on Oil A and Oil A1 are shown in Figures 5(a) and 5(b). Certain degrees of furrow were

observed in the rubbing directions, but these rubbing marks are more pronounced and prominent in case of ball run on Oil A1. This may be explained with the fact that FRC particles, mainly silicates, may act as an abrasive on a smooth surface of EN31 steel ball under high load of 490 N which results in higher value of friction coefficient and large number of typical grain abrasion and fatigue wear.

Elemental composition of the highlighted surface shown in Figures 6(a) and 6(b) demonstrates the presence of various base elements of EN31 alloy steel balls like Fe, C, S, P, and Cr, whereas presence of Ca, O, and Zn on worn surface of ball may be originated from engine lubricant additives (antioxidant, detergent). Figure 6(a) shows the elemental composition of the ball run on Oil A and absence of Si from the worn surface indicates that the balls were run on engine lubricant (Oil A) without FRC. Presence of Si, O elements on the worn surface of the ball run on Oil A1, shown in Figure 6(b), confirms the role of FRC in influencing the new ball surface properties in terms of deterioration and abrasion of smooth surfaces.

4.2.2. *Effect of FRC on Surface Morphology during Extended Hour Test.* Figures 7(a) and 7(b) shows micrographs of worn

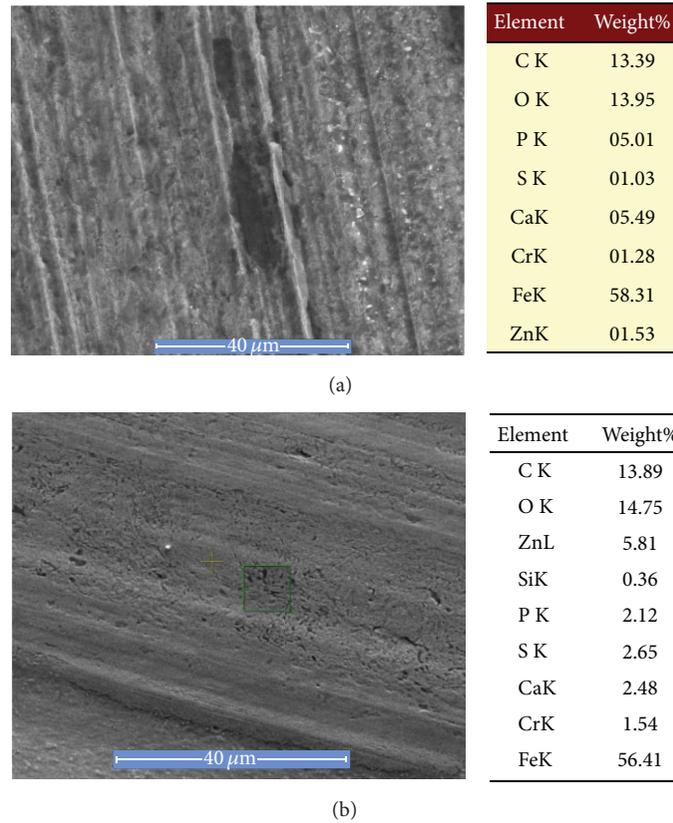


FIGURE 6: Elemental composition of the ball surfaces run on (a) Oil A and (b) Oil A1.

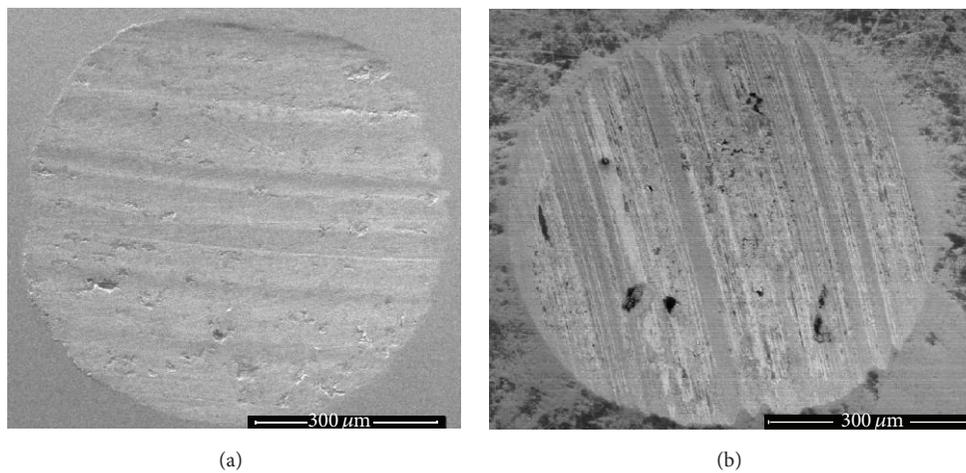


FIGURE 7: SEM images of ball after 2 hr test (a) ball run on Oil A and (b) ball run on Oil A1.

surface of the tests run on Oil A and Oil A1, respectively, for two hours each. Many deep scratches and adhesion phenomenon budding on worn surfaces of steel ball were observed for Oil A1, indicating the worn mechanisms mainly as abrasive wear and adhesive wear. On the contrary, lesser scratch marks were observed in the micrograph of ball surfaces run on Oil A, shown in Figure 7(b). This may be

explained with the fact that FRC was absent in Oil A, resulting in lesser abrasive wear on the worn surface.

4.2.3. *Surface Morphology of Balls Used in Tests Numbers 5a and 5b.* Figure 8 shows the micrographs of the worn surface, which are initially lubricated with Oil A using new balls for one hour followed by test on same set of balls (worn-out balls

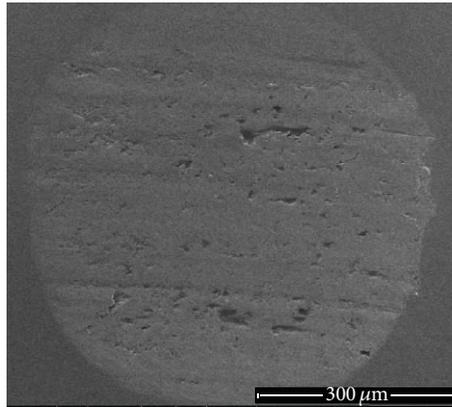


FIGURE 8: SEM image of a ball which initially run on Oil A for 60 min and then on Oil A1 without changing the surface contact for 75 min.

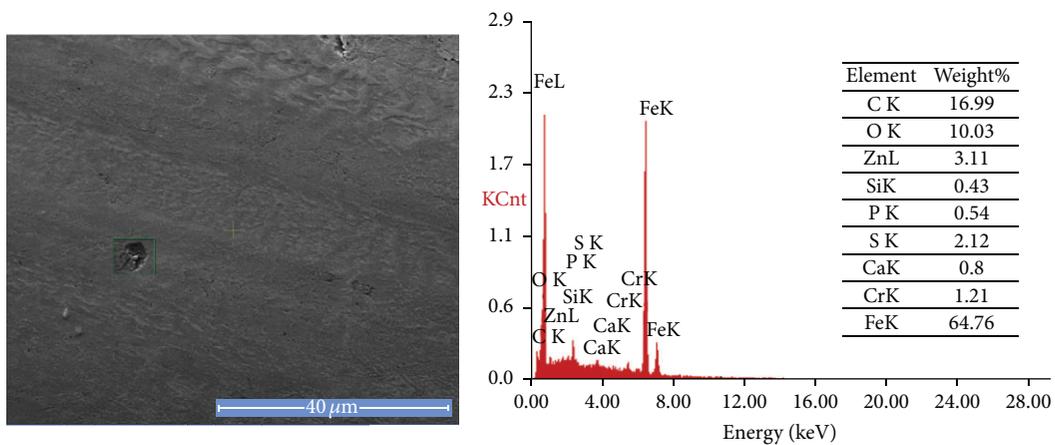


FIGURE 9: SEM image and elemental composition of surface of a ball which initially run on Oil A for 60 min and then on Oil A1 using same balls for 75 min.

without changing the tribosurfaces of interacting balls) using Oil A1 until stabilization of friction coefficient. The surface exhibits much smoother morphologies but a large number of micropits were also seen on the worn surface.

Figure 9 illustrated a typical elemental composition of one of the micropits on the worn surface. Presence of Si and O, in micropits, corroborates the role of FRC in friction reduction of interacting surfaces, consistent with the previous studies, highlighting the benefits of these micropits on the friction reducing mechanisms of surface texture [19, 21]. Micropits on friction surface can disarm abrasive dusts by entrapping them, thereby suppressing grain abrasion and adhesion. Besides, it is obvious that the micropits can act as oil reservoirs, which transport or retain oil to be released in emergency situations. The grinding action between tribosurface due to foreign particles removed from worn surface were captured by the micropits during the sliding and shearing of the worn surfaces, resulting in the reduction in abrasive wear of the contact surface.

Figures 10(a) and 10(b) describe the elemental distribution of the worn surface of the ball used for test run 5a and 5b and ball run on Oil A1 for one hour’ test. Presence of Fe

Si and O on the worn surfaces of both balls accounted for the possibility of some sort of ferrosilicate layer on tribosurfaces which may help in friction reduction.

### 5. Conclusions

This study revealed some of the important facts about the performance of FRC based engine lubricant with respect to friction, wear, and surface properties using a four ball tribometer. The following points may be concluded:

- (i) Engine lubricant with very low concentration of FRC influenced the friction coefficient.
- (ii) FRC based engine lubricant aggravates the friction between the interacting surfaces of new balls and lubrication regime transformed from mixed to boundary conditions during the test. SEM micrographs demonstrated it with the deep scratch marks in the rubbing direction when the FRC based engine lubricant was used.
- (iii) Friction reducing performance of FRC based engine lubricant on worn surfaces is phenomenal and during

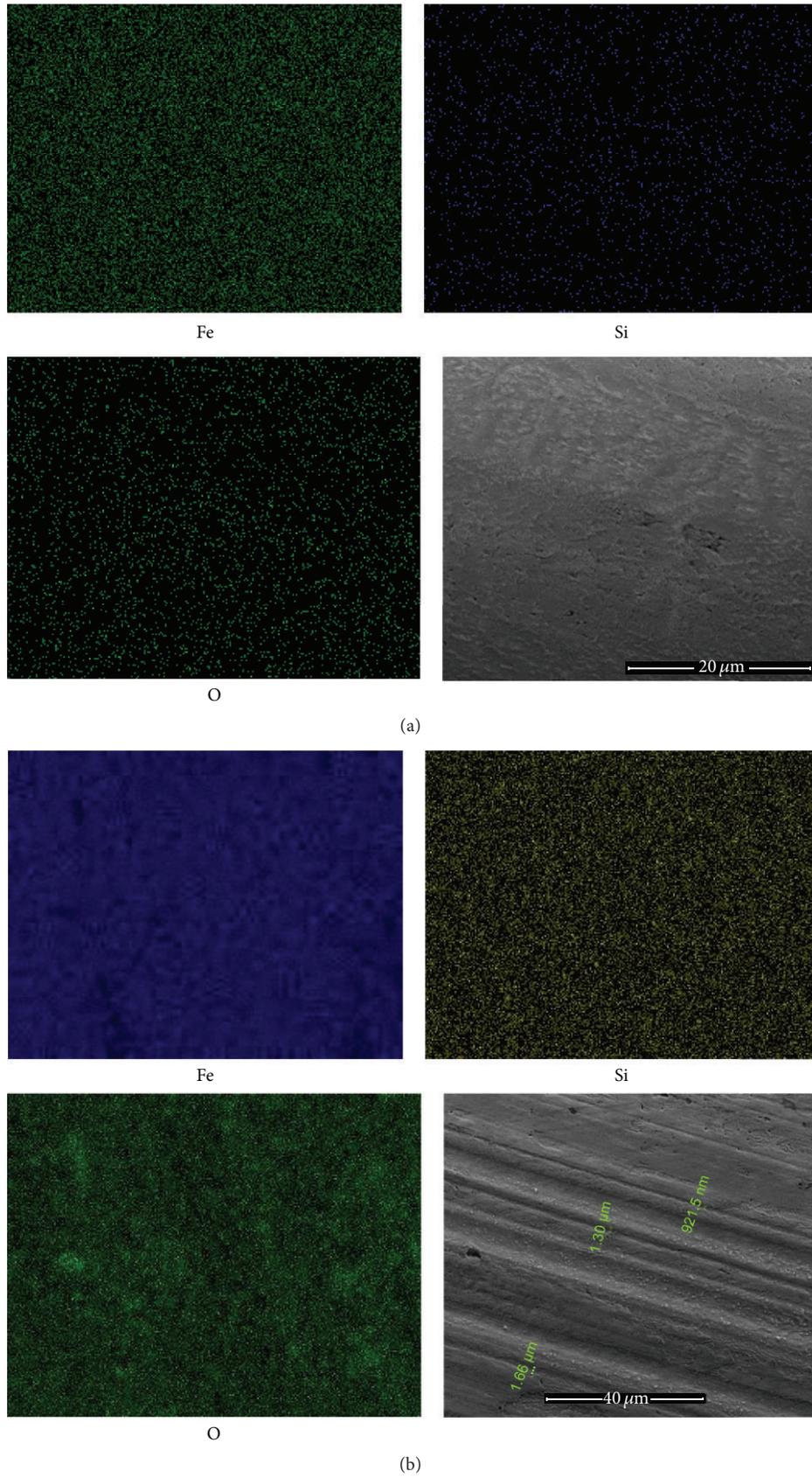


FIGURE 10: Worn surface mapping of the balls used in (a) tests numbers 5a, 5b and (b) test number 2 demonstrating the presence of Fe, Si, and O.

the test on worn surfaces, lubrication regime transformed from boundary condition to mixed lubrication condition.

- (iv) Presence of Fe, Si, and O on the worn surfaces of the used balls may account for the ferrosilicate layer on tribosurfaces.

## Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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