



Friction and wear reduction properties of GNP nano-particles as nano-additive for Al-Si + Al₂O₃ composite/Chromium plated steel tribopair

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KEYWORDS	ABSTRACT
Friction Wear Nano-additive Lubricating oil	Engine component durability in terms of friction and wear urge for the growth of new engineering materials and nano-lubricants for reducing friction and wear losses. Tribological test are carried out on eutectic Al-Si with (0 – 10 wt. %) n-Al ₂ O ₃ composite using reciprocating tribometer under high applied load (100 N – 300 N) with stroke 2 mm, 30 Hz frequency and 120-meter sliding distance. Base PAO-4 lubricating oil, SAE20W50 commercial lubricating oil, PAO-4 + 0.5 wt. % GNP, PAO-4 + 0.5 wt. % GNP + 0.5 wt. % oleic acid (OA) are used as the lubricating oil for the present study. The tribo-test results suggest the COF, wear volume and wear rate significantly reduced for PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA in comparison to other lubricants. The friction and wear volume reduction percentage for PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil is in the range 56.82 % and 21.56 % respectively compared to base PAO-4 base and SAE20W50 commercial lubricating oil. The anti-friction and anti-wear properties of the GNP based lubricant is because of roll-ball bearing, mending mechanism as well as tribo-film formation on the sliding interface.

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1.0 INTRODUCTION

In today's world, one third of the energy consumed is because of the friction losses and excessive wear which contributes for the majority of the mechanical system failure (Sarkar and Clark, 1980; Cruse, 1997). The emerging demand to reduce friction and wear of the mechanical system open up the doors for the development of the lightweight materials and new innovative lubricating techniques (Cole and Sherman, 1995; Hirata, 2005). In the recent times, aluminum matrix composites have been used for various internal combustion engine applications because of their better tribological properties and fuel effectiveness (Taylor, 1998; Becker, 2004; Ali and Xianjun, 2015). Light weight Al-Si alloys proves to be an effective matrix material for the production of aluminum composites because of excellent thermal, mechanical and tribological properties (King, 1987; Surappa and Rohatgi, 1981). Inclusion of the hard phase reinforcements in the Al-Si matrix material enriched the mechanical and tribological properties, which make these composite applicable for various high end applications (Pagounis et al., 1996). Size, morphology, weight/volume concentration, fabrication route also causes the improvement in their tribological properties (Pagounis et al., 1996). Improvement in the lubricating oil performance is another new innovation which can also solve the problem of friction and wear in materials. Nano-particles as the additive in the lubricating oil have been in focus in recent days because of their unique and excellent anti-friction and anti-wear properties (Lee et al., 2009; Ahmadi et al., 2013). Dispersion stability is major problems which affect the use of these nano particle additives in the lubricating oil (Jatti and Singh, 2015). The suspended nano-additives are likely to settle down, agglomerate, form clusters in the lubricating oil which reduces the tribological performance of the lubricating oil (Hwang et al., 2008). Thus to reduce these problems regarding the use of nano-additives in the lubricating oil, surfactant are added along with the nano-additives particles. Surfactant not only reduces the settling behavior and reduce agglomeration in the lubricating oil but also improves the friction and wear behaviors performance of the nano-lubricant (Kamogawa et al., 2001; Samavti et al., 2012). Nano-additive based lubricants form an adsorbed layer on the metallic interface of the tribo-pair. In the current scenario, most of the additives used are phosphorous and sulphate based additives but they are environmentally hazardous. Now there is the demand of environmental friendly additives that must possess excellent tribological properties under wet lubricating conditions.

Various tribological studies have been investigated on the friction and wear performance of Al-Si alloy, Aluminum composites and different lubricants with or without nano-additive in the past have been studied. Novak et al. in their study use diamond nano-particles in paraffin oil and study its tribological properties. The result shows that diamond nano-particles not only exhibit excellent tribological properties but also increases the load bearing capacity of the lubricating oil by penetrating in the rubbing surface. Excellent ball bearing effect is shown by the nano additive diamond particles in the paraffin oil (Novak et al., 2014). Shen et al. in their study discuss the tribological behavior of spherical diamond nano-particles as additive in the base lubricating oil. It is reported that addition of the nano-diamond particles not only improves the tribological properties but also improves the viscosity of the lubricant (Shen et al., 2001). Pawlak et al. studied the tribological behavior of h-BN as the nano oil additive. It is observed that nano as well as micro h-BN improves the tribological properties of the lubricant (Pawlak et al., 2009).

Shahnazar et al. in their study report that boron compound as the nano-additive in the lubricant generate h-BN by tribo-chemical reaction which formed a tribo-layer on the interface (Shahnazar et al., 2016). Chen et al. in their study investigate eutectic Al-Si alloy with applied load 0.5 N under lubricating conditions. From this study it is observed that Silicon particles present in

the alloy increases the load bearing capacity, wear resistance of alloy. Ultra-mild wear regime occurs during tribo-testing (Chen et al., 2008). Moustafa et al. studied the wear loss of Al-Cu material. The tribo test is performed under dry test conditions at 10 N load, 3.6 m/s sliding rate. It is reported that the wear rate of the Al-Cu is 2×10^{-3} mm³/m. Wear rate diminished to 0.5×10^{-3} mm³/m when strengthened with 30% of Al₂O₃ fortification particles (Moustafa and Soliman, 1997). Yalcin et al. reported the wear rate of 18 mm³/m for A356 Aluminum which is diminished to 12 mm³/m for the composite sample with 5 wt. % SiC. The wear tests are performed under dry sliding conditions at 5 N load 0.4 m/s speed for 1000 m sliding distance (Yalcin and Akbulut, 2006).

Reihani et al. studied the wear performance of Al6061 with or without fortification. SiC is utilized as the strengthening particles. It is accounted that the wear volume is 3 mg for the Al6061 base alloy which is diminished to 1.2 mg for the composite material when strengthened with 30 wt. % SiC (Reihani et al., 2006). The wear tests are performed under rough wear test conditions at 150 N load and 2000 rpm. Kumar et al. in their examination on Al6061/SiC and Al7075/Al₂O₃ composite report that the composites show better wear obstruction. It is likewise revealed that the expansion of SiC particles demonstrated better wear obstruction in contrast with the Al₂O₃ particles (Kumar et al., 2010). Umanath et al. contemplated the dry sliding wear of aluminum strengthened with SiC and Al₂O₃ particles. It is established that composite indicated a critical improvement in the wear obstruction of the composite. It is observed that the composite improves the mechanical and wear conduct of the composite (Umanath et al., 2013).

In this current study, eutectic Al-Si + Al₂O₃ composite sample is fabricated using SPS route. Further the lubricating tribo-test is conducted on the composite/chromium-plated-chrome-steel tribo-pair with base PAO-4 lubricating oil, SAE20W50 commercial lubricating oil, PAO-4 + 0.5 wt. % GNP, PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil. This study focuses emphasis on the friction and wear reduction, influenced by GNP as lubricating additive and Oleic acid surfactant in the PAO-4 base lubricating oil. From the above literature, it is clear that no such particular research study reported for this composite sample with GNP and OA in the base PAO-4 lubricant.

2.0 MATERIALS & SAMPLE FABRICATION

Eutectic Al-Si alloy is used as the matrix powder having composition shown in table 1. The matrix material is having particle size 50 μm – 70 μm and 2.67 g/cm³ theoretical density. The primary reinforcement used in the study is n-Al₂O₃ with varying content (0-10 wt. %). The reinforcement particle size for the primary reinforcement is 20 nm with 3.95 g/cm³ of theoretical density. These materials procured from intelligent materials, India are used as the raw material for this study. The reinforcement is added in the different variable concentration (0, 2, 4, 6, 8, 10 wt. %) in the base matrix material. Base composition with no reinforcement is represented as BC, with C1, C2, C3, C4 and C5 as the composite sample with 2, 4, 6, 8, 10 wt. % reinforcement respectively are represented in the present study.

Table 1: Elemental composition of Al-Si alloy powder.

Al %	Si %	Fe %	Cu %	Zn %	Mn %	Mg %
Bal.	= 10.58	< 0.8	< 0.3	< 0.2	< 0.15	< 0.1

For sample fabrication the raw powders are first sonicated at 40 KHz frequency with ethanol as process control agent in the ultrasonic probe sonicated followed by milling/blending in the

high energy ball mill of planetary configuration. The powders are milled in the silicon nitride jar with 10 mm silicon nitride ball. Ball to powder ratio is 10:1 and other milling parameters are 240 rpm for 24 hours. The wet suspension of powder in ethanol is then dried in the vacuum evaporator and vacuum oven at 50° C to completely remove the moisture content from the milled powder. The blended powder is then ready for the fabrication process. The dried powder is poured in the graphite die and the samples are fabricated under argon atmosphere at 450° C temperature, with 100° C heating rate, 50MPa load, 10 min holding time and 150° C cooling rate. The samples are then removed from the die after fabrication and then prepared for the tribological testing. The fabricated samples are polished using SiC emery paper followed by the diamond paste on velvet cloth to get the mirror polished surface.

To prepare lubricating oil with nano-additive and surfactant in the PAO-4, 0.5 wt. % of GNP and OA is added to the base oil. The liquid sample is first magnetically stirred for 2 hours followed by ultrasonic mixing for 8 hours with controlled temperature. The Oleic acid is added to improve the dispersion stability of the nano-additives in the base lubricant, considering the problem of agglomeration of nano-additive in the lubricant.

3.0 TRIBOLOGICAL TESTING DETAILS

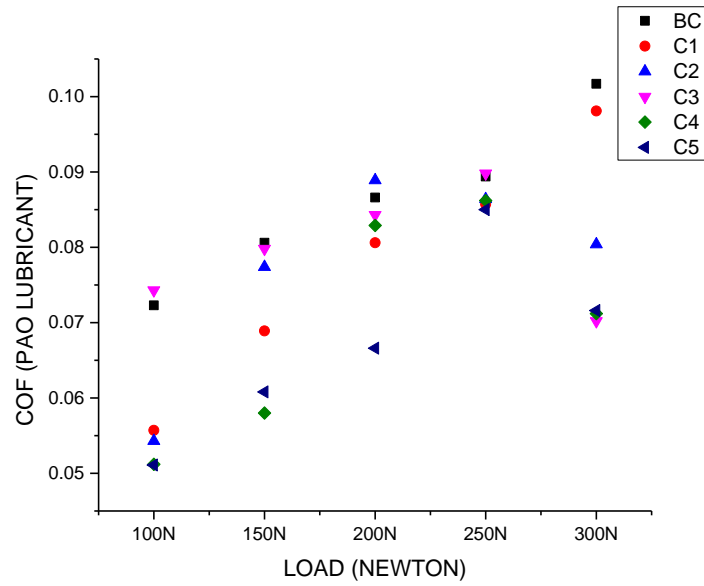
The tribological tests are conducted on R-tech universal tribometer of reciprocating configuration with ball-on-disc geometry. The test is conducted under variable load 100 N – 300 N with stroke 2 mm, 30 Hz reciprocating frequency, 120-meter sliding distance at room temperature. The load test is carried out for 1000 seconds. 1 to 2 drop of lubricant oil is applied on the highly polished sample before testing. Each test is carried thrice so as to reduce the experimental error. Before and after testing the samples are cleaned with acetone in the ultrasonic cleaners to remove the dirt, contamination and debris from the composite samples. 10 mm chromium-plated-chrome-steel-ball is used as the counter-body for the tribo-test. The tribo-test with different lubricating oil i.e. Base PAO-4, Commercial SAE20W50 oil, PAO-4 + 0.5 wt. % GNP, PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA are used which helps in base line comparison. After testing the wear volume is measured using 3D surface profilometer. Further to calculate wear rate equation (1) is used.

$$Wr = \frac{Wv}{SD \times L} \quad (1)$$

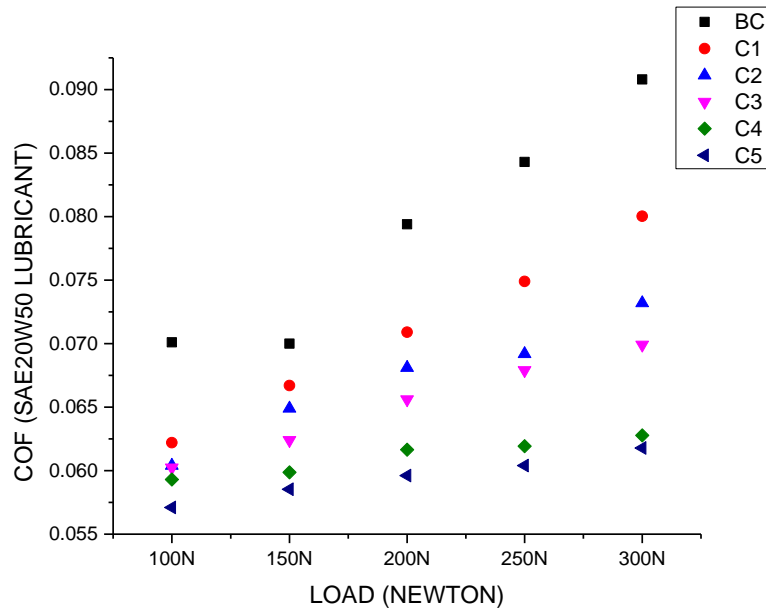
Wr: Wear Rate; *Wv*: Wear Volume; *SD*: Sliding Distance; *L*: Load.

4.0 FRICTION AND WEAR STUDIES AND ANALYSIS

From the experimental studies conducted on the tribo-pair it is clear that the factors i.e. composite composition, load, sample roughness, counter-body, lubricant as well as additives have a huge impact on the frictional properties of the tribo-pair. The tribo-test is conducted with four variable lubricants. For the tribo-test conducted with PAO-4 base oil and SAE20W50 commercial oil similar behavior with COF is observed as shown in Figure 1 (a) & (b) respectively.



(a)

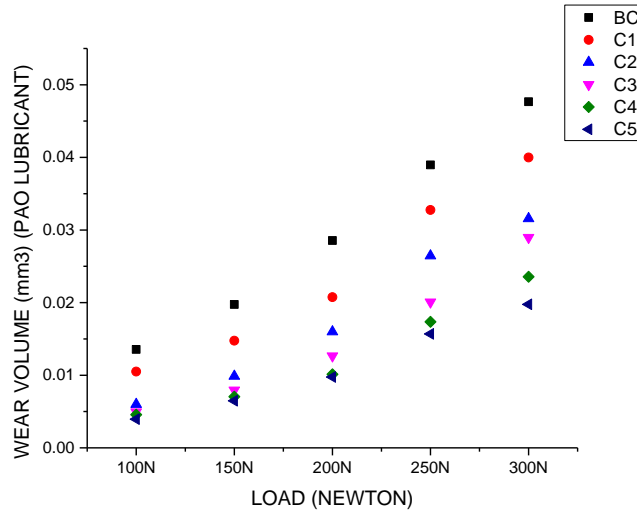


(b)

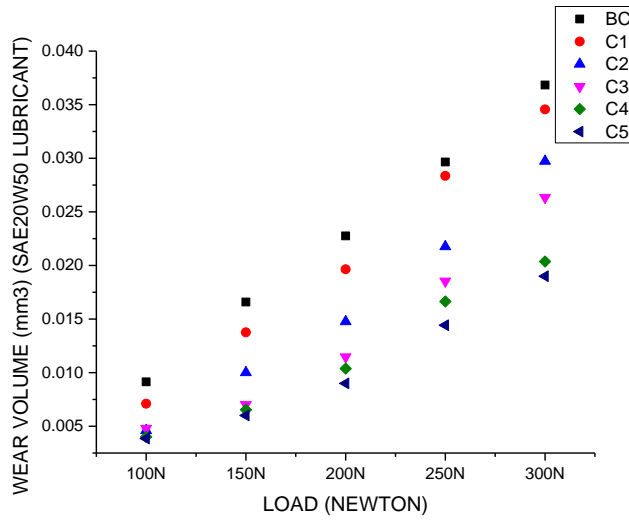
Figure 1: (a) COF v/s Load for PAO-4 base oil and (b) COF v/s Load for SAE20W50 oil.

The value for the COF increases with increasing load for all the composite samples. It is observed that the COF value for the SAE20W50 commercial lubricating oil seem to be less compared to base PAO-4 lubricating oil. In this case the sample composition also has an influence on the frictional behavior of the tribo-pair. It is observed that the base composition i.e. eutectic Al-Si alloy report maximum value for the COF as well as wear volume for the PAO-4 and SAE20W50 oil. This high value of COF for the base composition is attributed to the soft aluminum present in the alloy in large quantity. The aluminum asperities easily adhere during the testing that increases the value of COF as well as wear volume. It is observed that the inclusion of the hard phase ceramic reinforcement in the alloy matrix increases the frictional and wear resistance properties of the composite sample. This reduction in the COF and wear volume of composite sample is attributed to the hard phase reinforcement which improves the hardness thus indeed improves wear resistance of the fabricated samples.

Due to the inclusion of the $n\text{-Al}_2\text{O}_3$ in the eutectic Al-Si alloy the particles do not easily protrude from the sliding interface. This indeed prevent the further formation of asperities previously formed on the base composition due to abrasion as well as high solubility adhesion mechanism. Another fact that is responsible for the reduction of the friction and wear of the composite material is the coherent oil film thickness between the tribo-pair which actually reduces the direct metal-metal contact. That lubricating oil also reduce the interface temperature which is generated because of frictional heat causes the softening of the matrix material. Reduction in frictional heat ultimately helps to reduce the frictional traction and reduces the COF value with PAO-4 and SAE20W50 oil. The progressive behavior of the COF and wear volume with increasing load is attributed to the increased direct contact with the applied load. As the load increases the area of contact also increases at the nano-scale which increases the direct asperity-asperity contact. Due to high asperities contact the frictional force also increases leading to high value of the COF with load. Due to this high COF and high asperity contact, the interlocking between the interfaces increases which leads to adhesion and abrasion mechanism. Wear volume and wear rate graph for the PAO-4 and SAE20W50 oil with all composite samples are shown in Figure 2 (a, b) & 3 (a, b) respectively. Figure 2 and 3 report the similar trend for the wear volume removal and the wear rate of the composite sample under different lubricating oil conditions. Increase in the applied load leads to increase in the wear volume and wear rate of the base composite and composite samples. It is reported that there is a decrease in the wear volume and the wear rate with the increase in the nano reinforcement in the matrix material. Another important point comes from this particular study is that SAE20W50 lubricant shows reduced material removal and wear rate in comparison to the base PAO lubricant.

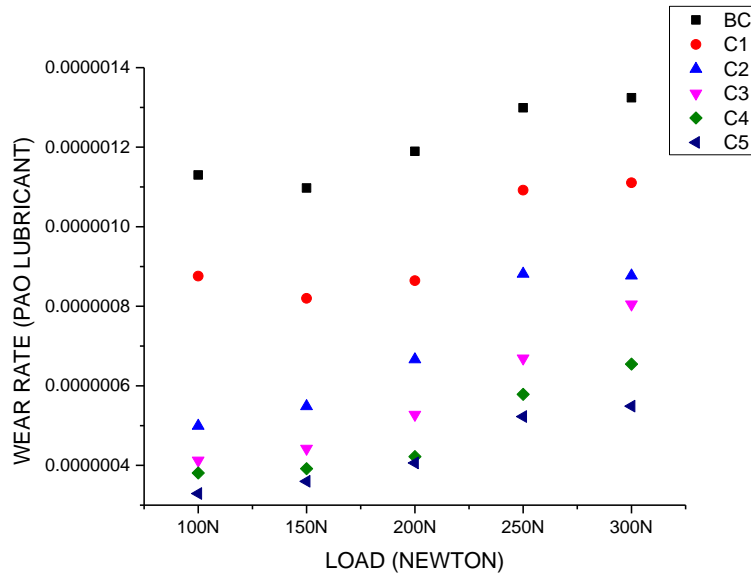


(a)

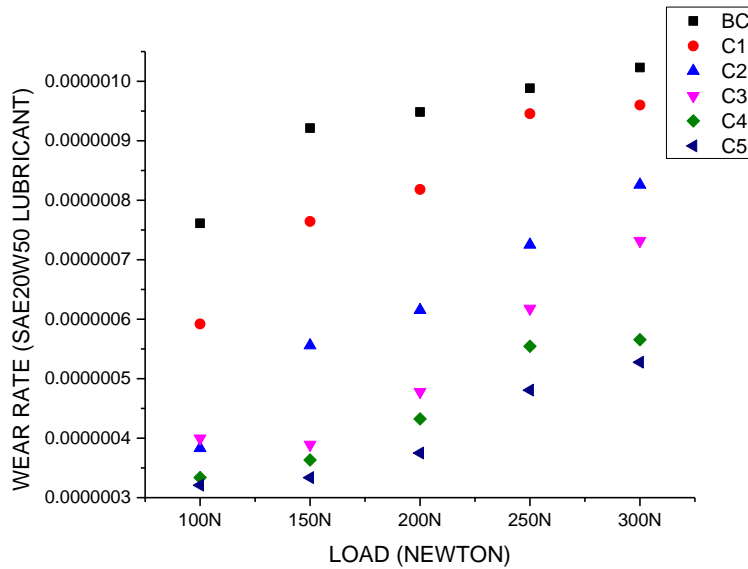


(b)

Figure 2: (a) Wear Volume v/s Load for PAO-4 oil and (b) Wear Volume v/s Load for SAE20W50 oil.



(a)



(b)

Figure 3: (a) Wear Rate v/s Load for PAO-4 oil and (b) Wear Rate v/s Load for SAE20W50 oil.

SEM & OM on the wear scar with PAO-4 base and SAE20W50 commercial lubricating oil are shown in Figure 4 & 5. Mild solubility adhesion, narrow grooves, furrows due to abrasion as well as delaminating wear at extremely high load due to severe plastic deformation are observed on the scar zone. For composite with high concentration of n-Al₂O₃, discontinuous oxide layer are

also observed which attributed to oxide layer patch damaged due to extremely high applied load. These oxide layer/ tribo-layer are also responsible for the reduction of wear volume and COF for the composite with high concentration of ceramic reinforcement. EDS analysis confirms that small amount of the material transfer is observed on the counter-body due to mild adhesion as shown in Figure 6. 3-D surface roughness analysis is made on the scar zone and is shown in Figure 7.

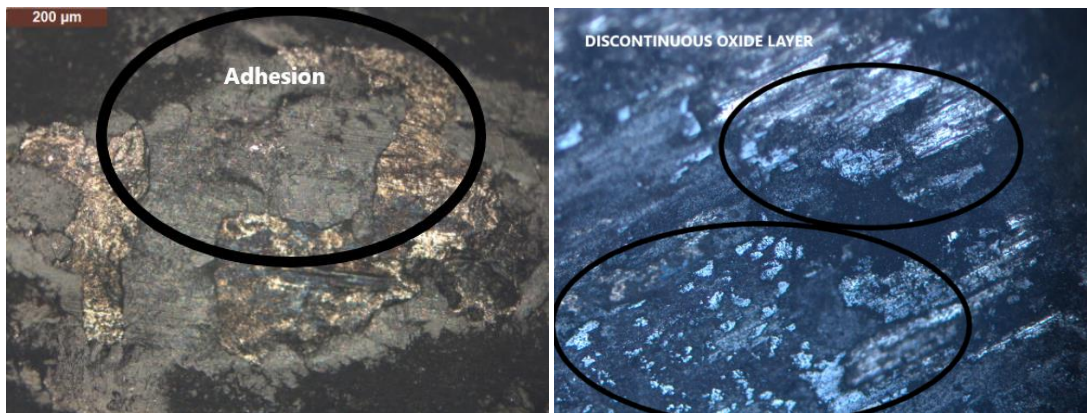


Figure 4: Optical micrographs of the wear scar with PAO-4 base and SAE20W50 commercial lubricating oil.

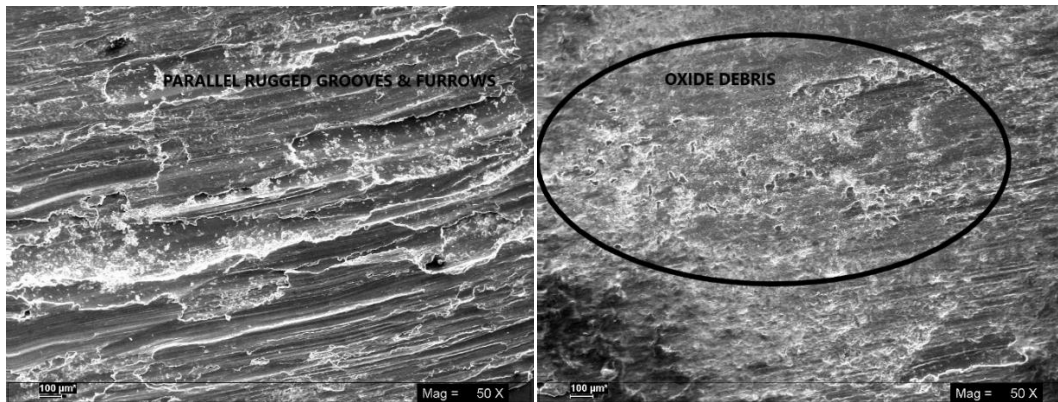


Figure 5: SEM micrographs of the wear scar with PAO-4 base and SAE20W50 commercial lubricating oil.

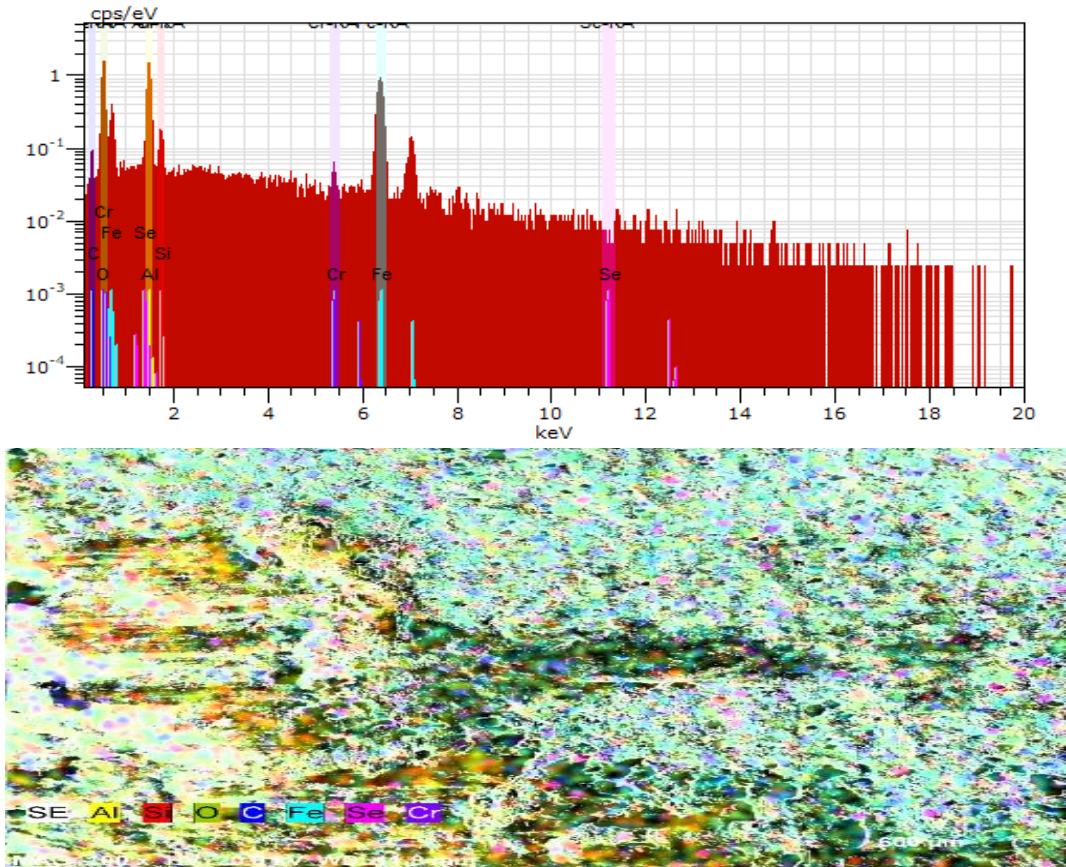


Figure 6: EDS analysis of the wear scar for PAO-4 base and SAE20W50 commercial lubricating oil.

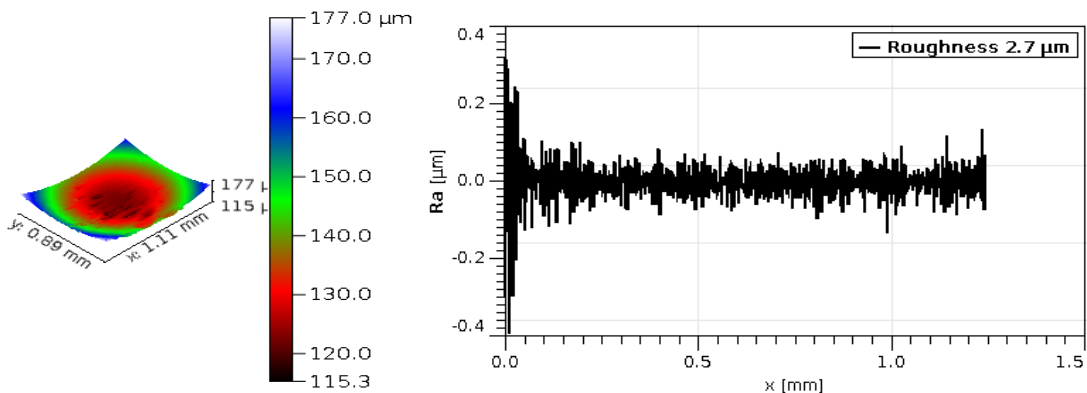


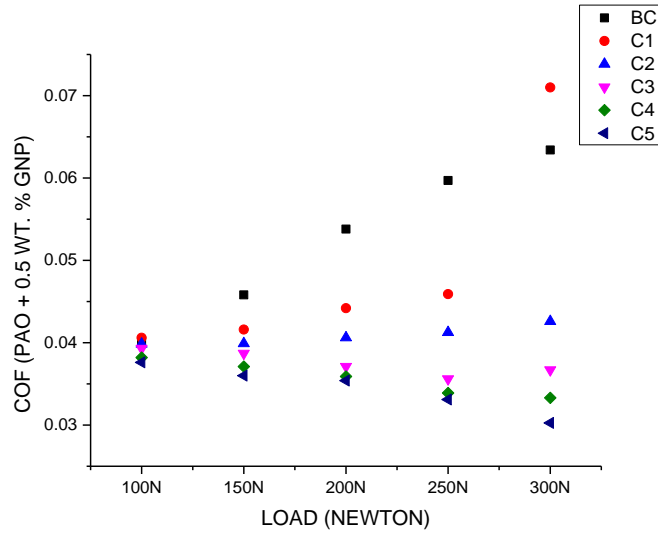
Figure 7: 3D surface roughness of the wear scar for PAO-4 and commercial lubricating oil.

However, for the PAO-4 + 0.5 wt. % GNP lubricating oil significant reduction in the COF as well as wear volume is observed compared to the base PAO-4 and commercial SAE20W50 lubricating oil. There is a progressive trend in the COF with increasing load for the base composition as well

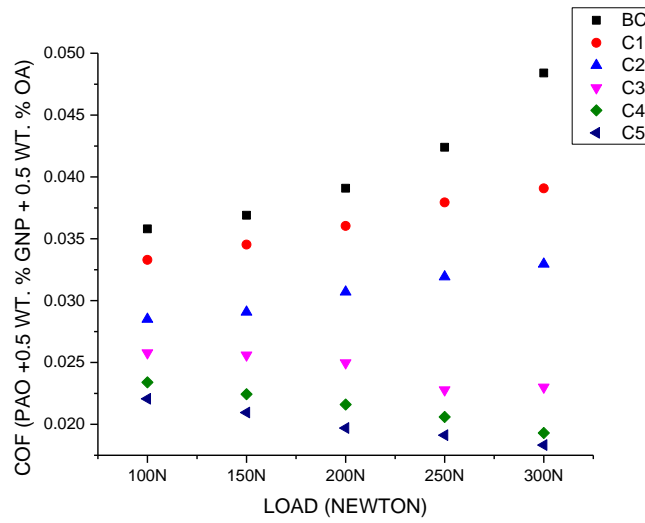
as for the composite sample up to 4 wt. % n-Al₂O₃, afterwards the COF value almost decreases with increasing load as shown in Figure 8 (a). Similar COF behavior for the PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil is observed as shown in figure 8 (b).

From the results it is observed that, COF value as well as wear volume is very less for these lubricating oil in comparison to base PAO-4 and SAE20W50 oil. The progression in the COF for the composite sample up to certain limit is attributed to the soft matrix material which increases the frictional traction hence increases COF. Beyond that limit the COF starts reducing, this reduction is attributed to two main reasons: (a). high percentage inclusion of hard phase n-Al₂O₃ reinforcement in the matrix material which increases the hardness indeed wear resistance of the composite sample. The hard phase ceramic particles increase the overall load bearing capacity of the composite thus help in reducing the COF with increasing load. (b). another mechanism responsible for the reduction of COF with increasing load is the presence of the GNP nano-additives particles and Oleic Acid as surfactant in the lubricant. GNP nano-additives particles are the lamellar structured particles with weak Vander wall force which can be easily sheared thus help in easy sliding of bodies in contact. GNP particles present in the lubricant also reduce the COF and wear volume by mending mechanism and nano ball-roll-bearing mechanism. The GNP particles settle down within the asperities of the contacting interface thus reduces the asperities contact at the nano-level that causes easy sliding at the interface. By nano-ball-roll-bearing mechanism the nano-additives particles convert the pure sliding mechanism to the rolling mechanism which also helps in reducing the COF and wear volume of the composite sample. The wear volume and wear rate for PAO-4 + 0.5 wt. % GNP and PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricant is shown in Figure 9 (a, b) & 10 (a, b) respectively.

Optical micrographs and SEM on the wear scar for the PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricant oil shows some obvious scratches as shown in Figure 11 & 12. SEM studies shows smooth surface because of less adhesion and abrasion mechanism. Delaminating pits observed on the scar zone due to plastic and shear deformation at extremely high sliding load. The presence of the GNP nano-particles in the oil help in formation of continuous low shear stress oxide lubricating smooth junction on the scar zone which indeed reduces the direct metal-metal contact that reduces the COF and wear rate. This smooth tribo-film helps to reduce the adhesion and abrasion mechanism due to reduced asperities. EDS analysis confirms the formation of graphene oxide layer on the scar zone as carbon and oxygen are present on the scar zone and is shown in Figure 13. This oxide layer also helps to improve the load bearing capacity of the composite sample. Raman analysis gives the evidence that the graphene oxide film is formed on the scar zone as D, G and 2D band are formed on scar zone as shown in Figure 14. 3D surface roughness of the wear scar is shown in Figure 15, which is evident that a smooth scar surface is formed even after the testing, attributed to self-polishing mechanism of the GNP particles present in the oil.

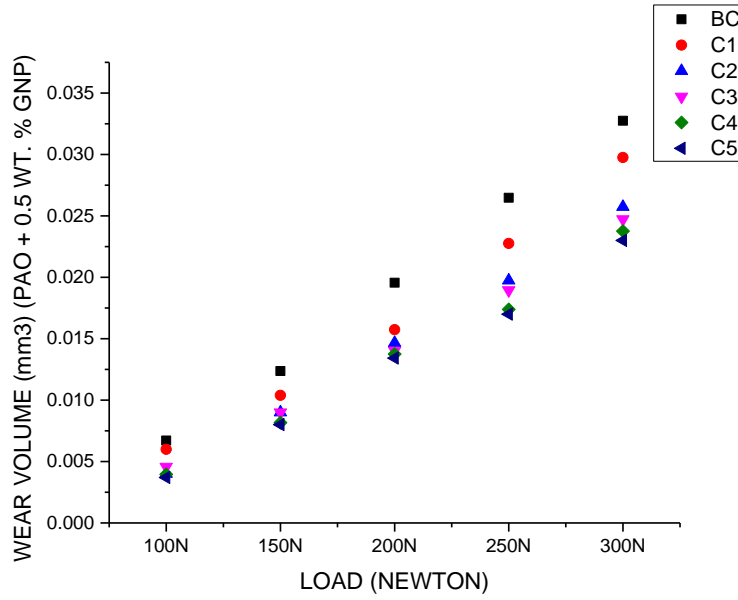


(a)

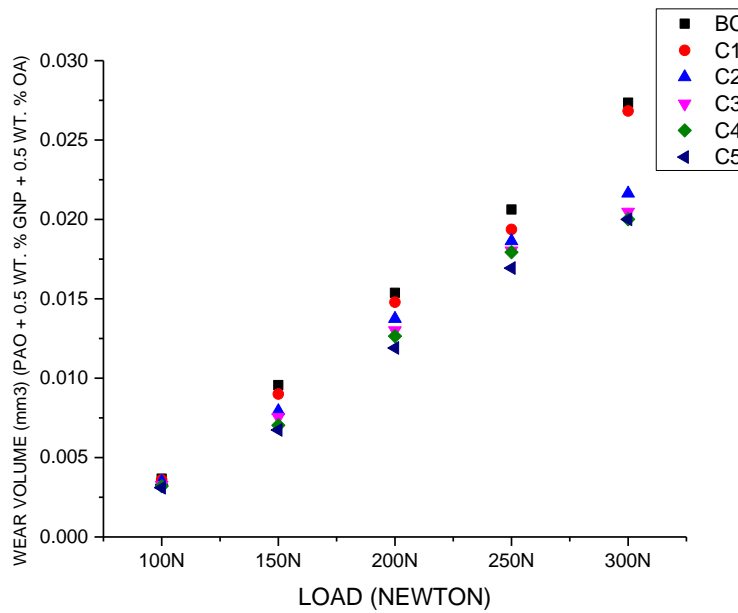


(b)

Figure 8: (a) COF for PAO-4 + 0.5 wt. % GNP and (b) COF for PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA.

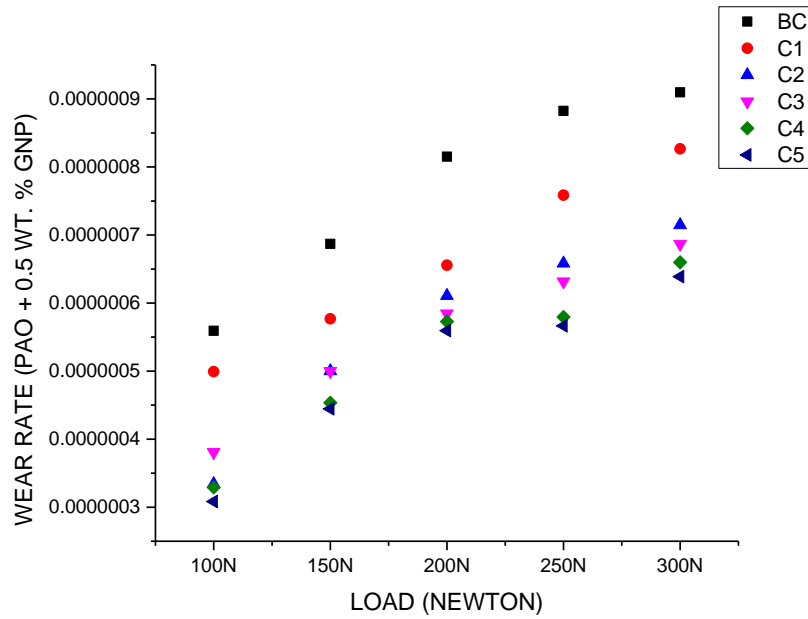


(a)

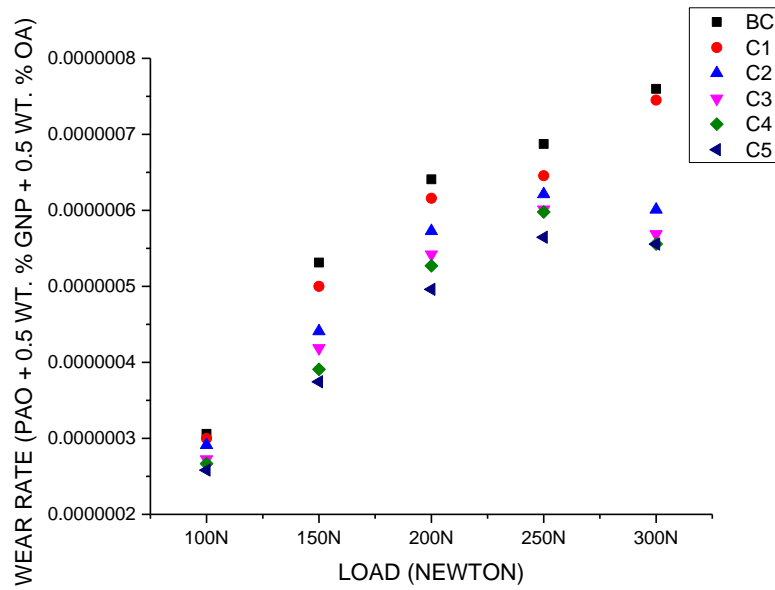


(b)

Figure 9: (a) Wear Volume for PAO-4 + 0.5 wt. % GNP and (b) Wear Volume for PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA.



(a)



(b)

Figure 10: (a) Wear Rate for PAO-4 + 0.5 wt. % GNP and (b) Wear Rate for PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA.

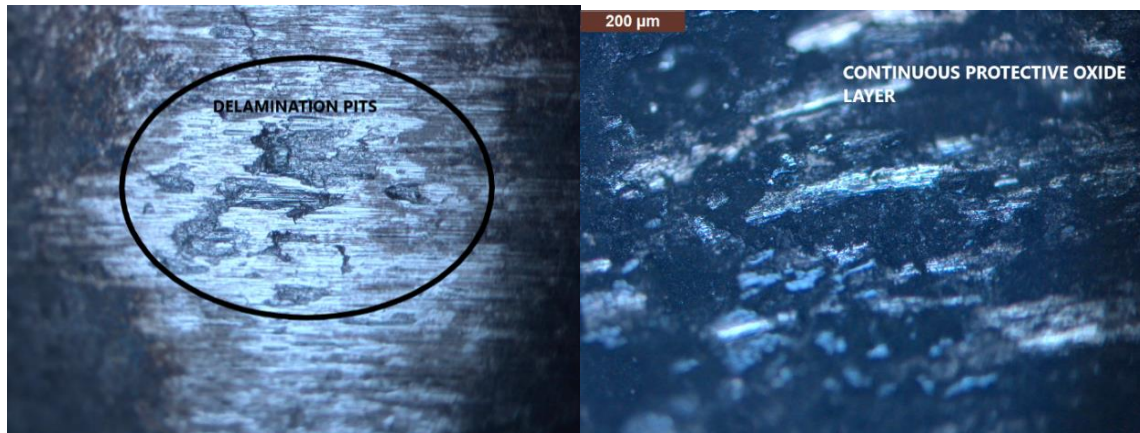


Figure 11: Optical Micrographs of the wear scar with PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil.

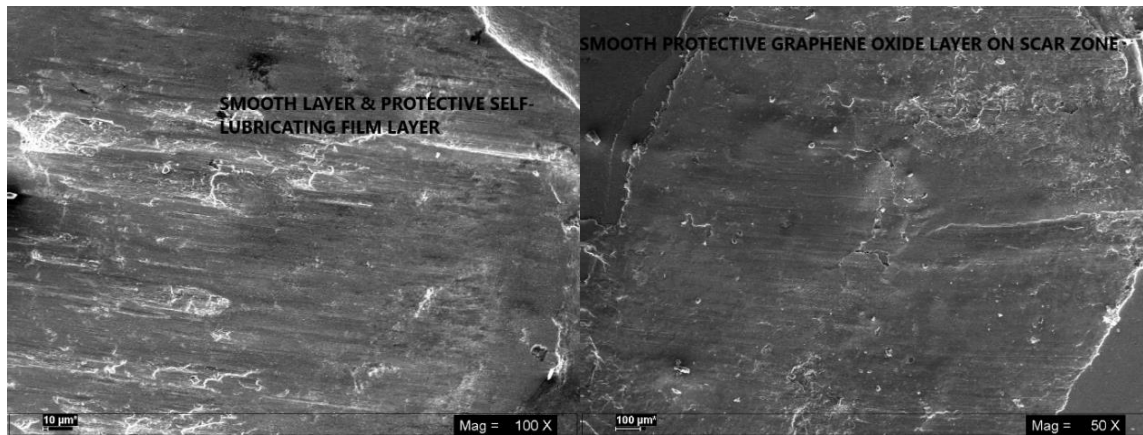


Figure 12: SEM Micrographs of the wear scar with PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil.

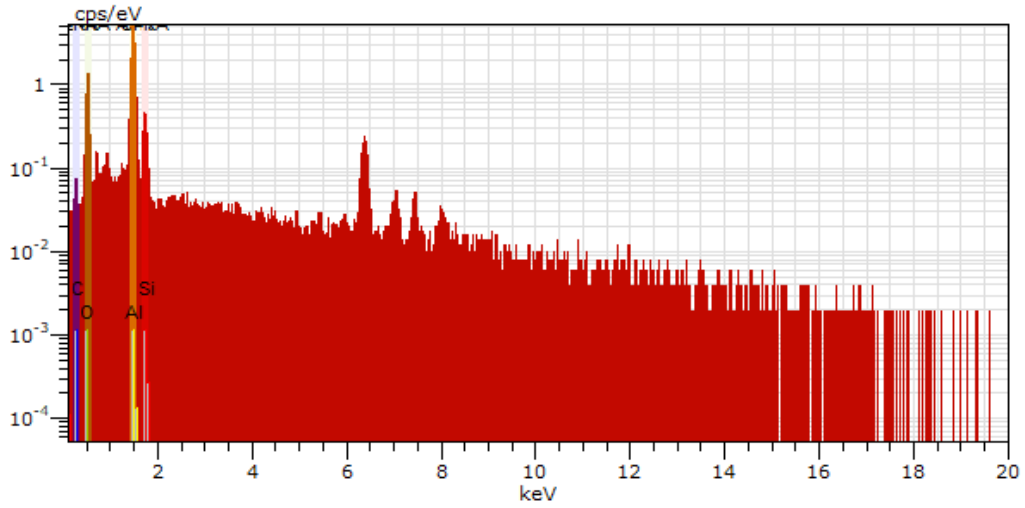


Figure 13: EDS analysis of the wear scar for PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil.

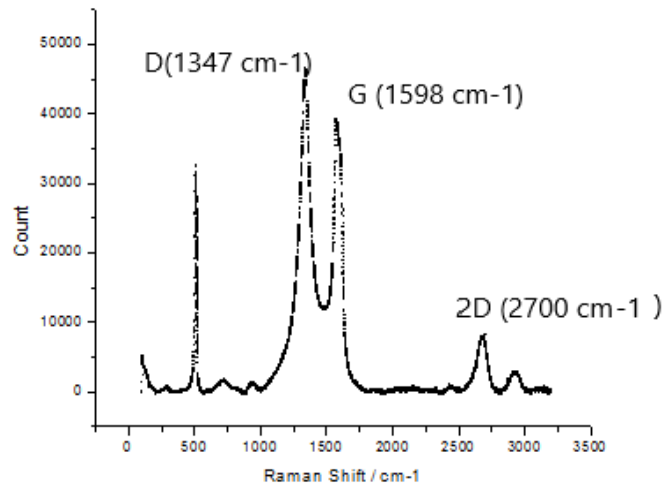


Figure 14: Raman Analysis of the wear scar with PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil.

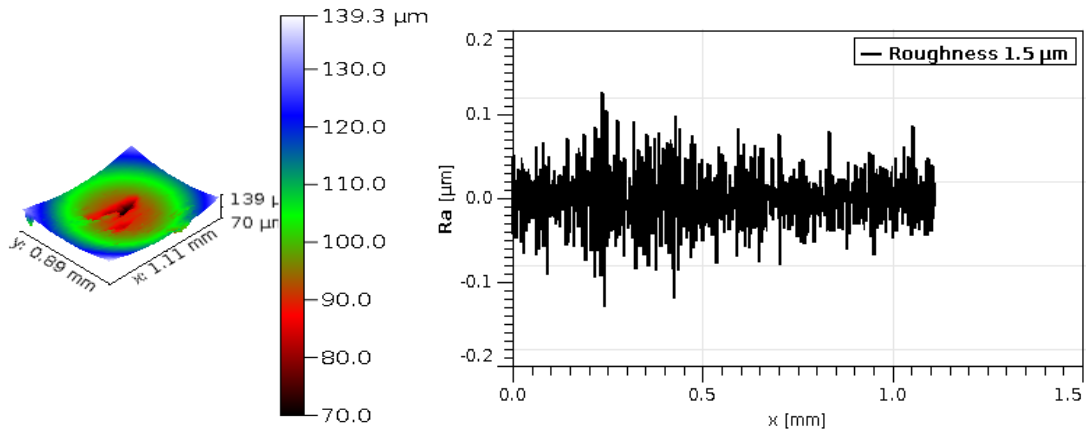


Figure 15: 3D surface roughness of the wear scar with PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricating oil.

5.0 CONCLUSION

The present study focus on reduction of friction and wear of tribo-pair, which is influenced by n-Al₂O₃ reinforcement in composite, and GNP nano-additives as well as oleic acid as surfactant in the base oil. The tribo-test is conducted at high applied load 100 N – 300 N for different oils. From the study the following are the conclusive points obtained.

- (a) Al-Si + 10 wt. % n-Al₂O₃ composite with PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricant exhibit exceptional tribological properties. As these composition sample along with the above stated lubricant shows minimum value of COF, minimum wear volume and wear rate. n-Al₂O₃ reinforcement restricts the wear propagation during the continuous sliding,

and the GNP along with OA in the lubricant improves the lubricity of the lubricant and reduce the agglomeration rate.

- (b) Increase in the ceramic reinforcement increases the hardness which improves the wear resistance of the composite. Reduction in the COF for the Al-Si + 10 wt. % n-Al₂O₃ composite with PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA is in the range from 56.82 % in comparison to the base oil. Wear volume reduction is in the range from 21 % in comparison to the base PAO-4 base oil.
- (c) Nano-additive particles in the lubricant govern the mending and nano-ball-roll-bearing mechanism which improves the lubricity and load bearing capacity of the lubricant.
- (d) Nano-additive particles present in the lubricant forms a low shear stress oxide smooth film layer on the scar zone which improves easy sliding hence reduces the COF and wear of the material.
- (e) Surface roughness of the wear scar for Al-Si + 10 wt. % n-Al₂O₃ composite with PAO-4 + 0.5 wt. % GNP + 0.5 wt. % OA lubricant is decreased by 44.44 % in comparison to base alloy for PAO-4 lubricant having average surface roughness value of 2.7 μm.
- (f) The smooth scar surface is attributed to the nano-additive self-polishing mechanism within oil. Overall, improvement in the friction and wear properties is observed which makes these compositions as well as nano-additive particles compatible for various tribological applications.

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