# Investigation of Tribological Properties of Lubricating Gear Oil With Abrasive Nano Additives

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***Abstract***

*Purpose: Lubricants prepared with metal oxide nanoparticle additives are found to have better tribological properties. In this work the widely used commercial SN lubricating oil is added with Al2O3, SiO2, TiO2 nanoparticle and the tribological characteristics are studied. Wear tests were conducted on plain oil and oil with nanosized Al2O3, SiO2, TiO2 additives using a four ball tribo-tester.*
*Design/methodology/approach: The wear images of the ball specimens were analyzed using confocal microscope to measure the topography of the wear parameters such as wear scar diameter, depth of wear, angle of the wear.*
*Findings: It is found that the gear oil with silica nanoparticles is found to have improved properties. Results show a considerable reduction in the values of wear scar diameter, wear angle and wear depth for the gear oil added with Al2O3, SiO2, TiO2 nanoparticles of 0.4 wt. %.*
*Originality/value: In the work carried out, a new innovative technological approach to analyze the tribological properties of lubricating oil by finding the wear scar diameter, wear angle and wear depth is reported.*
***Keywords: Confocal microscopy; Lubricating gear oil; Silica nanoparticles; Wear scar Topography***

## 1. INTRODUCTION

Lubricants prepared with nanoparticle additives are found to have better tribological properties than the one with oil soluble additives. These lubricants meet the modern demands like the ability to reduce frictional resistance, good anti-wear characteristics, heat absorbing capacity etc. In particular, the metallic oxide nano additives are found to influence effectively the tribological characteristics of the lubricant oil than the organic additives. Many metallic oxides such as Al2O3, TiO2, CuO, ZnO etc. [1-10] are investigated as lubricant additives. Xue et al. [1] reported on the surface modified titanium dioxide (TiO2) additive in liquid paraffin which displayed good load carrying capacity with better anti-wear and friction reduction properties. Hu et al. [2] found TiO2 additive in 500 SN oil to exhibit improved wear resistance, and decreased frictional coefficient values and correlated the anti-wear and friction reduction performance of nanoparticles with respect to the additive structure, particle size, and concentration. Yu [3] reported on the tribological property of engine oil with CuO and TiO2 nano particles as additives and found considerable reduction in the scar depth. Report on tribological properties of sun flower oil with an addition of 0.5 wt% zinc borate nano powder, done by Zhao et al. [4] exhibited reduction of friction coefficient to the maximum of 14 %. Arumugam et al. [5] reported that addition of 0.05 wt.% of TiO2 nanoparticles in rapeseed oil reduced frictional coefficient considerably and found the value to be between 18.4% and 19%. Verma et al. [6] reported on MoS2 nanoparticles as additives in paraphine oil and observed the wear scar diameter and friction coefficient to be low. Tarasova et al. [9] concluded that addition of ZnO with mineral oil exhibited good tribological character along with a good film forming capacity. The engine oil of SF grade, showed desirable tribological properties with CuO additives as reported by Padgurskas et al [8]. Chou et al. [9] and Lin et al. [10] reported the suspension property to be the key factor for marginal reduction in friction and wear for the chosen oil, which was achieved by ultrasonic vibration of additive in the medium. Many reports focused on improving load carrying capacity with nano silica in rapeseed oil, increasing viscosity with nano additives and significant reduction in friction coefficient with respect to the choice of nano additive [8]. For example, cobalt nanoparticle was reported to reduce the friction more efficiently than iron nanoparticles. Tribological properties of copper nanoparticles indicate it to be a best additive for lubricating oils. However, research on improvement of tribological properties of lubrication gear oil by using the cheapest metal oxide - silica in nanosize is not reported elsewhere in case of SN gear oil (standards of General Motors Type A suffix A). The durable lower viscosity oil with anti-wear additives that prevent wear, pitting, spalling, scoring and scuffing is more desirable in automobile applications in order to prevent equipment failure and downtime. In the work carried out, a new innovative technological approach to analyse the tribological properties of lubricating oil by finding the wear scar diameter, wear angle and wear depth is reported. As gear oils are subjected to rolling load, four ball tribo-testers are used to analyse the anti-wear performance of the gear oil. Analysis of wear scar is important to tribologists as it reveals the critical details on the level of wear the ball is subjected to different loading conditions. Conventionally the balls subjected to test are taken out and tested for only for wear scar diameter using an optical microscope. In the proposed approach confocal microscope is used to analyse the wear depth and wear angle.

## 2.PROCEDURE FOR PREPARATION OF SAMPLE

Gear oil of the current application meeting the standard of general motor's Type A Suffix A / Daimler Benz DBL 6623-10 is chosen for the study. The Al2O3, SiO2, TiO2 nanoparticles of an average size less than 50 nm are purchased from Sigma Aldrich. For good dispersion stability, the surfactants are mixed in a ratio of 2:2:1 and added with sodium dodecyl benzene sulfonate. A preferred HLB value for compatibility between the oil and the surfactant is also ensured. Al2O3, SiO2, TiO2 nanoparticles with different weight percentages – 0.2 wt.%, 0.4 wt.%, 0.6 wt.%, 0.8 wt.% and 1 wt.% are added to the lubricant oil and the mixture is kept at constant temperature of 70-80oC in a water bath for about twenty minutes. Further, the mixture is stirred in a magnetic stirrer at a constant temperature of 80oC for about 2 hours at a speed of 1300 rpm, to achieve good dispersion. Wear test is conducted in a four ball test apparatus (Figure 1) with specimens of balls prepared according to ASTM standards. The specimen balls are made up of Chrome alloy steel in conformity with AISI standard steel no. E-52100 and suitably polished. The composition of the ball is listed in Table 1.

**Figure. 1. Schematic diagram of four ball tester**

**Table 1. Chemical composition of E52100 steel**

| Cr | C | Mn | Si | Cu | Ni | P | S |
|---|---|---|---|---|---|---|---|
| 1.58 | 0.99 | 0.46 | 0.23 | 0.1 | 0.1 | 0.008 | 0.005 |

**Table 2. Sample combinations of $Al_2O_3$, $SiO_2$, $TiO_2$**

| S.No. | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|---|---|---|---|---|---|---|---|
| SAMPLE | oil | oil with surfactant | (0.2,0.2,0.2) | (0.2,0.4,0.4) | (0.2,0.6,0.6) | (0.2,0.8,0.8) | (0.2,1.0,1.0) |
| S.No. | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| SAMPLE | (0.4,0.2,0.4) | (0.4,0.4,0.6) | (0.4,0.6,0.8) | (0.4,0.8,1.0) | (0.4,1.0,0.2) | (0.6,0.2,0.6) | (0.6,0.4,0.8) |
| S.No. | 15 | 16 | 17 | 18 | 19 | 20 | 21 |
| SAMPLE | (0.6,0.6,1.0) | (0.6,0.8,0.2) | (0.6,1.0,0.4) | (0.8,0.2,0.8) | (0.8,0.4,1.0) | (0.8,0.6,0.2) | (0.8,0.8,0.4) |
| S.No. | 22 | 23 | 24 | 25 | 26 | 27 | |
| SAMPLE | (0.8,1.0,0.6) | (1.0,0.2,1.0) | (1.0,0.4,0.2) | (1.0,0.6,0.4) | (1.0,0.8,0.6) | (1.0,1.0,0.8) | |

Typically, an optical microscope of accuracy ± 0.01 mm is used to measure wear scar at the bottom of the three balls. In this work the topography is studied with a confocal microscope. Experiments are repeated for the oil without nanoparticles under typical conditions of testing. The average values of the trials are discussed in the analysis part. The lower balls subjected to tribo-testing develop wear scar which has a three dimensional topography. The topography of the wear can be best defined its diameter, depth of wear and the wear angle. In comparison with the

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conventional microscopy, confocal microscopy offers several distinct advantages. The most important capability of confocal microscopy is the ability to acquire high quality metallographic images with the capability to reconstruct the sectional plane information into three dimensional model. In a conventional microscopy the specimen gets illuminated as a whole where as in confocal microscopy a laser source illuminates a point on the specimen. The digital image collected in conventional microscopy has the problems of blurring and reduces the resolution of the image. In confocal microscopy the information of the specimen on the focal plane only reaches the photo detector and the information is reconstructed to form a 3D image. The recent advantages in the fields of microelectronics, laser radiation techniques and digital image processing have led to significant improvements in the field of confocal microscopy. In this paper confocal microscopy is used to study the wear topography.

## 3.RESULTS AND DISCUSSION

Figure 2 shows SEM micrograph of Al2O3, SiO2, TiO2 nano particles. The particles are spherical in shape and size lesser than 50 nm. An elemental composition of Al2O3, SiO2, TiO2 particle is observed using EDX showed the presence of Silicon and Oxygen. No peaks related to impurities are observed (Figure 3). Four Ball tester performs both Wear Preventative (WP) and Extreme Pressure (EP) analyses for measuring the wear and frictional properties of lubricants under sliding-on-steel test conditions.

In a typical four ball tribo-tester (Figure 1), three ½ inch diameter balls made of hardened steel Table 1 is rotating held in position by a fourth ball of same diameter steel ball. They are covered with lubricant oil and pressed into the cavity formed by the three clamped balls forming a three-point contact, and rotated for a set duration. Lubricants are compared for anti-wear characteristics using the average size of the scar diameters worn on the three lower clamped balls. The equipment has provisions to measure the normal load on the ball assembly and frictional torque using load cells.

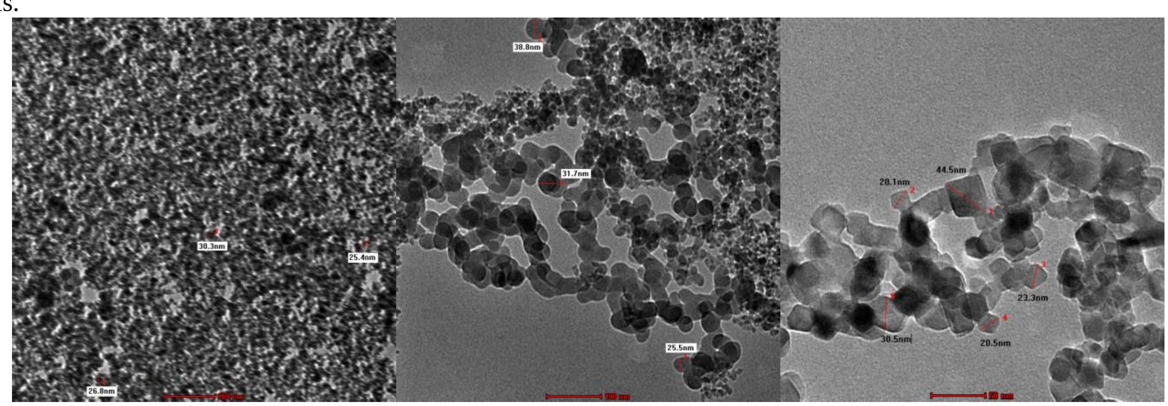


**Figure. 2. SEM micrographs of the $Al_2O_3$, $SiO_2$, $TiO_2$ nano particle**

### 3.1. EFFECT OF $AL_2O_3$, $SIO_2$, $TIO_2$ NANOPARTICLES ON COEFFICIENT OF FRICTION

Figure 4 shows that the friction coefficient of the oil as the function of time. As observed from the anti-wear test, the coefficient values vary with the weight percentage of the nano additive added to the oil, which is due to the changes in shear stress. The sample with 0.4 wt.% of Al2O3, SiO2, TiO2 nanoparticles is found to highly reduce the coefficient of friction (about 53 wt.%) when compared with oil without additive and other samples. Therefore 0.4 wt.% of Al2O3, SiO2, TiO2 added oil can be considered as best one with low shear force and improved load carrying capacity. Figures 5-8 show the wear scar topography obtained with confocal microscope. Balls subjected to tribo-testing with different weight percentages of nano additives are measured for the topographical information namely, wear scar diameter, wear depth and wear angle and are shown in Table 2. The influence of different % weight proportions of nano particles added with the lubricating oil on the wear topographical parameters are presented in the subsequent sections.

### 3.2. EFFECT OF $AL_2O_3$, $SIO_2$, $TIO_2$ NANOPARTICLES ON THE ANTI-WEAR PROPERTIES OF OIL

When compared to the base oil, 1 wt.% Al2O3, SiO2, TiO2 nanoparticle shows a wear reduction of 12.5-20%. Considerable reduction is also noticed in all the combinations of oil with additives over 0.2 wt.% to 1 wt.% loading. It is also observed that this wear scar grooves from the plain sample ball is not due to plastic deformation. The severity of the wear scar depends on wear scar diameter. Thus the nanoparticle oil seems to possess to a better wear resistance than the base oil. Some solid materials are found at the end part of the wear surface along rubbing direction and this could be due to the friction product of nanometre silicon dioxide on anti-wear properties. The wear reduction percentage has been analysed with respect to the loading concentration of nanoparticle in the oil. It is understood that when compared to base oil, the addition of silicon dioxide nanoparticle strengthens the wear resistance of the oil. It is obvious that at 0.4 wt.% of loading, the wear reduction is significant. After this, no significant degree of reduction is observed. If more than 1 wt.% of nanoparticles are added in the oil, agglomeration may occur which causes Al2O3, SiO2, TiO2 nanoparticles to increase their size beyond nano level. The shape of the particle may change from sphere to other form with angular features that promotes wear.

**Figure 3 image of wear topography obtained Material**

**Figure 4 image of wear topography obtained liqued**

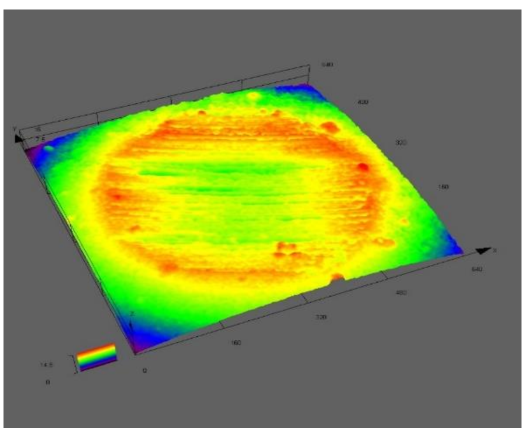

**Figure 5 image of wear topography obtained using confocal microscope**

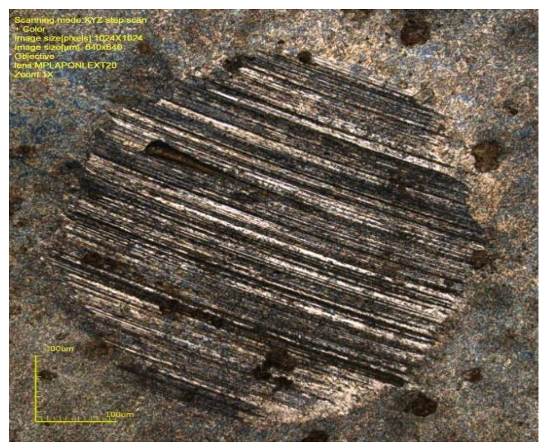

**Figure 6 image of wear topography obtained using confocal microscope .**

### 3.3 EFFECT OF $AL_2O_3$, $SIO_2$, $TIO_2$ NANOPARTICLES ON THE WEAR ANGLE

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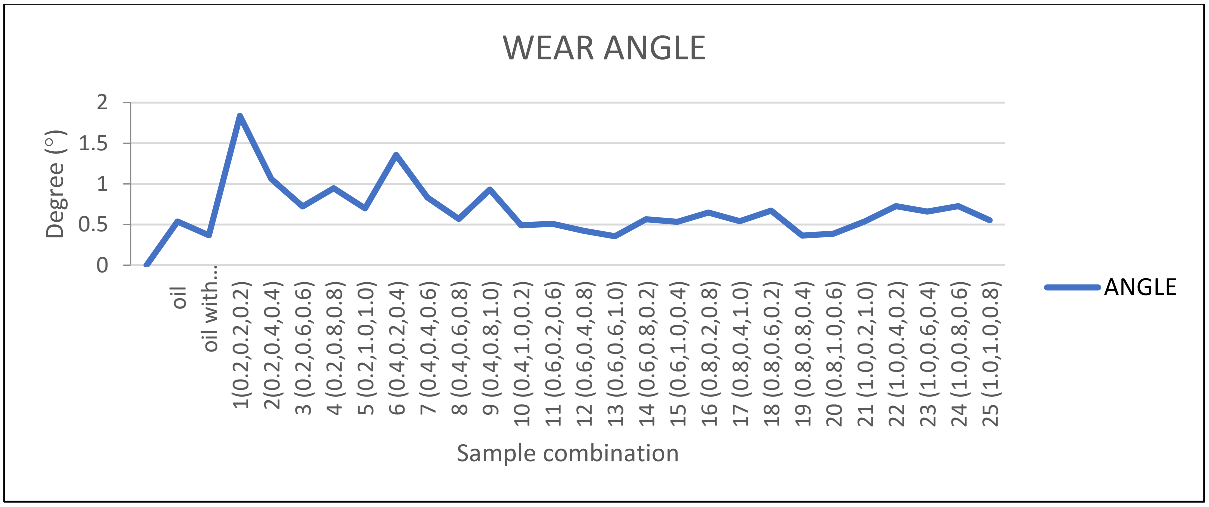


**Figure 7 Sample Combination analysis with response Angle Vs Degree**

Table 2 shows that the contact between balls during test condition is 0.625 higher than the other proportions. The wear angle decreased to 55.52% when 0.4 wt.%, nanoparticle added with oil. The addition of nanoparticle influenced the balls contact area, while simultaneously reducing the conduct angle wear scar diameter, wear depth and the wear angle. As the contact area of balls is reduced, the shear force and coefficient of friction also seem to reduce. Further, the addition of nanoparticle seems to increase the load carrying capacity.

### 3.4 EFFECT OF $AL_2O_3$, $SIO_2$, $TIO_2$ NANOPARTICLES ON THE WEAR DEPTH

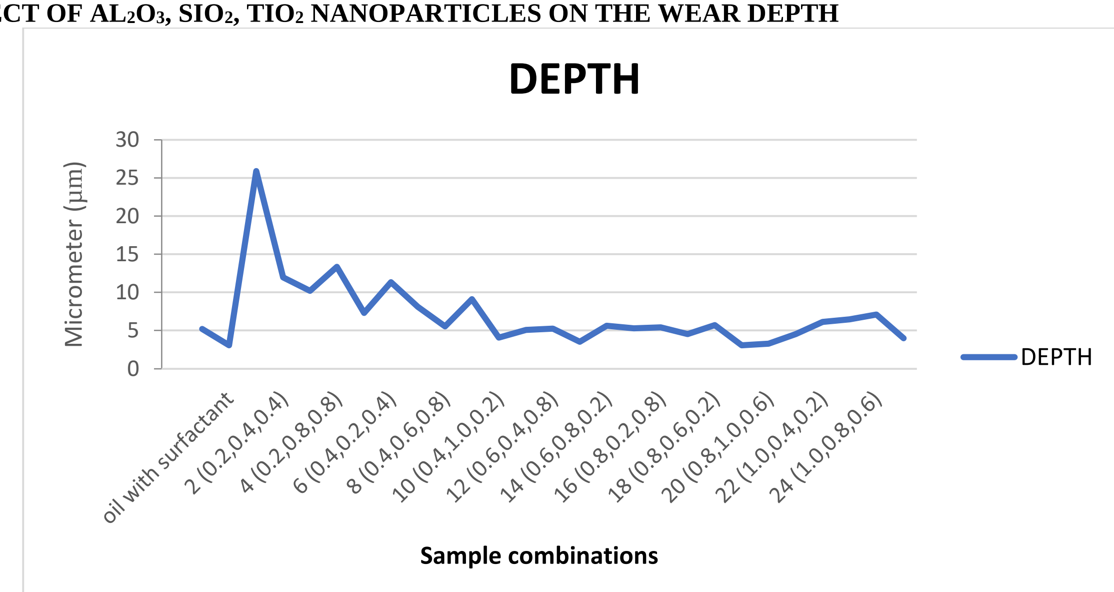


**Figure 8 Sample Combination analysis with response Depth Vs Micrometer**

Figures 5-8 show the wear depth of wear scar measured using confocal microscope. It is found to be 6.294 µm for the balls run with oil without additives. The depth of wear decreased to 4.1 µm (65.14% of wear depth reduction compared to oil without additive) after the addition of 0.4 wt.% Al2O3, SiO2, TiO2 nanoparticles to the oil. Even though, the other weight proportion of nanoparticle reduces the wear depth when compared to standard gear oil condition, the addition of 0.4 wt.% nanoparticle seems to provide a higher wear depth reduction. The filling of nanoparticles in the cleavage of the wear reduces the shear force and thereby, reducing the depth of wear, load carrying capacity and coefficient of friction.

### 3.5. EFFECT OF $AL_2O_3$, $SIO_2$, $TIO_2$ NANOPARTICLES ON THE WEAR SCAR DIAMETER

Figures 5-8 show the wear image measured on specimens obtained after testing with oil and oil with nanoparticle additives using confocal microscope. The microscopic results shown in Table 2 indicate that the gear oil contain the highest wear scar diameter of 576.663 micrometre. The 0.4 wt.% nanoparticle in oil gives lower wear scar diameter (that is 21.649 % of wear scar diameter reduction), when compared to the other proportions. However, it is noticed that the addition of nanoparticles in lubricating oil results in lesser wear scar diameter compared plain oil without nanoparticles.

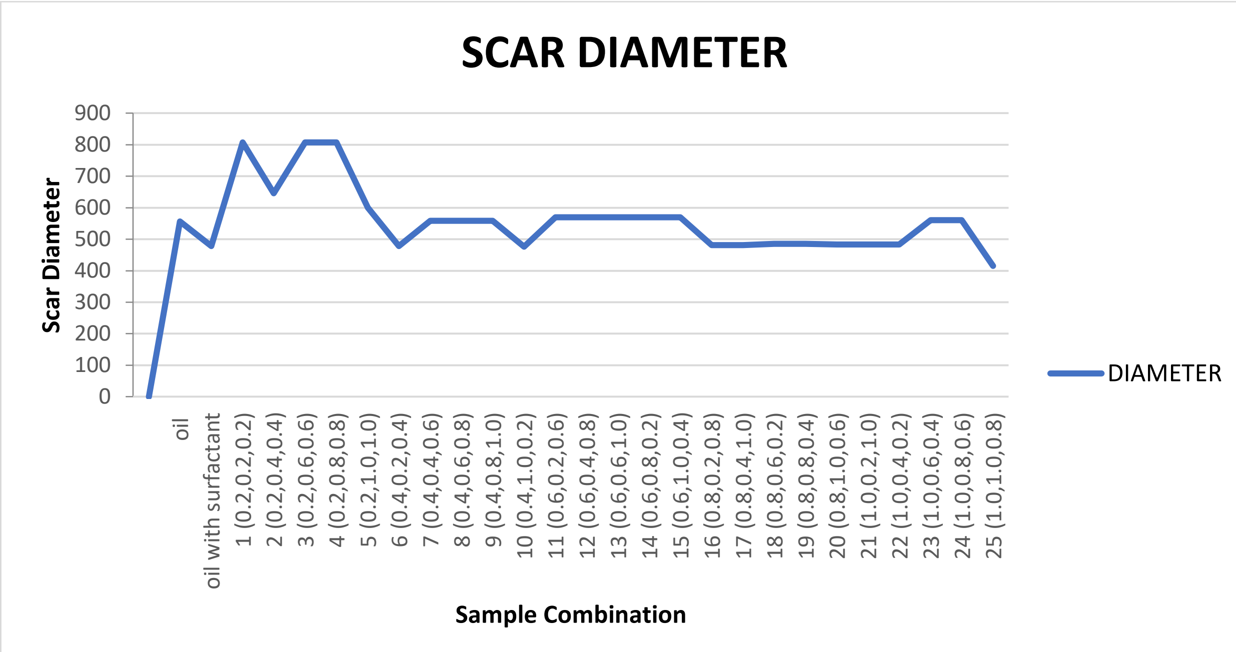


**Figure 9 Sample Combination analysis with response Diameter Vs Scar Diameter**

### 3.6. EFFECT $AL_2O_3$, $SIO_2$, $TIO_2$ NANOPARTICLES ON THE LOADING CARRYING CAPACITY OF THE OIL

The adding of 1 wt.% of Al2O3, SiO2, TiO2 nanoparticles to the oil seems to present maximum load carrying capacity of the oil with the weight of 392 N, as it corresponds to low friction coefficient (Figure 1). Further, the addition of Al2O3, SiO2, TiO2 nanoparticles (more than 1 wt. %) results in very high coagulation, even in the presence of surfactant and results in the increase of the diameter of wear scar. Whereas, in case of 0.4 wt.% of Al2O3, SiO2, TiO2 nanoparticles added oil, the friction coefficient is slightly higher than the above sample for the same loading capacity. Comparing all the samples,0.4 wt.% can be taken as optimized, owing to the low friction coefficient and proper dispersion.

### 3.7. TRIBOLOGY PROPERTIES OF $AL_2O_3$, $SIO_2$, $TIO_2$ NANOPARTICLES

The SEM image of the balls after cleaning with distilled water is shown in Figure 9. The SEM image of the oil without additive shows the presence of cleavages and larger scuffing width. After addition of 0.2 wt.% of Al2O3, SiO2, TiO2 nanoparticles as additive to the oil, the cleavage was minimized and it was completely absent in case of 0.4 wt.% of Al2O3, SiO2, TiO2 added oil. This was reflected by the smooth surface of the balls as shown in Figure 10. But further increase in wt.% of additives resulted in appearance of cleavages with notable scuffing width over the ball surface. Higher wt.% of additives not only resulted in increase in scuffing width, but also shows the presence of coagulated Al2O3, SiO2, TiO2 nanoparticles over the surface of the balls even after proper cleaning. The SEM analysis proves the 0.4 wt.% Al2O3, SiO2, TiO2 nanoparticle added oil to be the best one in terms of its tribological properties. After the oil with different wt.% of Al2O3, SiO2, TiO2 nanoparticle was stirred for 60 min, the balls were cleaned in the ultrasonic bath vibrator using distilled water. The SEM image corresponding to balls taken from 0.8 and 1.0 wt.% of Al2O3, SiO2, TiO2 added oil, clearly shows presence of Al2O3, SiO2, TiO2 nanoparticles over the wear scar, even after the balls were cleaned with distilled water. This shows that the nanoparticles were very well adhered over the balls. Here the anti-wear mechanism was formed due to the deposition of the Al2O3, SiO2, TiO2 nanoparticle acting as the third body which decreases shear stress [11]. This is due to the deposition of Al2O3, SiO2, TiO2 nanoparticle and this is its stability.

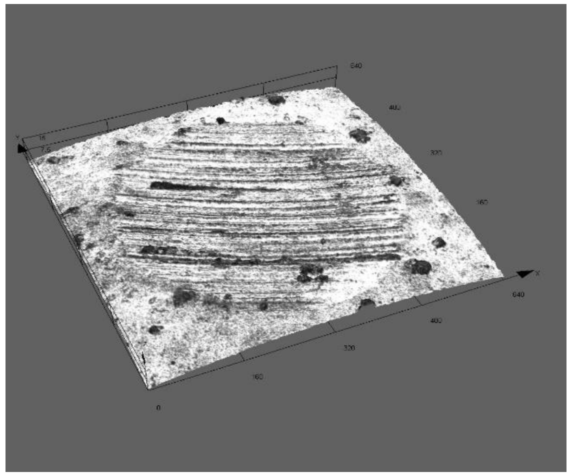

**Figure. 9. Image of wear topography obtained using a confocal microscope (0.8,0.2,0.8 wt.% $Al_2O_3$, $SiO_2$, $TiO_2$)**

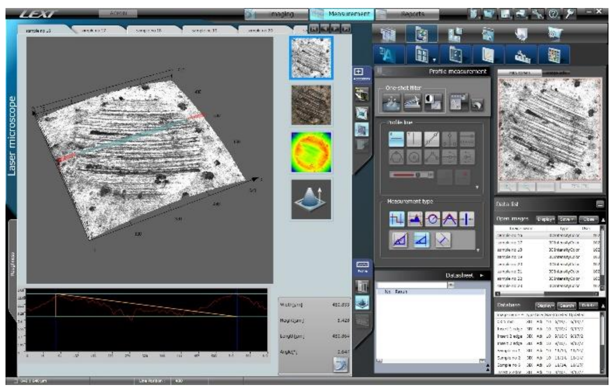

**Figure. 10. Image of wear topography obtained using a confocal microscope**

## 4. CONCLUSIONS

The standard gear oil of commercial interest with different load concentrations of Al2O3, SiO2, TiO2 nanoparticles are tested for its anti-wear properties using four ball tester. The scar diameter, depth and wear angle of the balls under test are reported successfully for the first time by using confocal microscopy. For sample with 0.4 wt.% of Al2O3, SiO2, TiO2 nanoparticle as additive, the average reduction in scar diameter, depth and wear angle are observed to be 21.65 %, 65.14%, and 55.52%, respectively which is a very minimal value when compared to oil without additive. The cleavages and scuffing width of the wear are found to be very less for the 0.4 wt.% of additive added oil, as proved by the SEM analysis. The wear mechanism behind the excellent tribological properties of 0.4 wt.% of additive added commercial oil is explained by the balls with stable Al2O3, SiO2, TiO2 nanoparticles over its surface even after several washing.

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