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Research Article

IMPACT OF SOOT ON METAL WEAR CHARACTERISTICS USING LASER DIFFRACTION SPECTROSCOPY

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ABSTRACT:
The characteristics of soot affecting on the abilities of lubricating oil and leading to result in metal wear was studied. Soot particle contamination was simulated using carbon black. Micro-nanostructure of particles were studied by electron microscope. The behavior was studied by means of a Four-Ball tribology test with friction and wear measured. Wear roughness in micro-scale was also investigated by high resolution optical microscope and 3D rendering optical technique. Based on tribology test, soot contamination in oil made more wear on tested steel ball and reduced friction during test comparing to the test with fresh oil. In conclusion, the appropriate particle size (20 nm - 80 nm of primary particles and 80 nm - 300 nm of agglomerated particles), which is near to oil film thickness between metal surface contacts, is the dominant cause of making wear. By the way, the too large particle size compared to oil film thickness will escape out and too small particle size will not effect on wear. Furthermore, high level of proper particle size (20 nm – 100 nm) contaminated in oil will increase probability of rubbing process.

Keywords: *Lubricating oil, Soot, Wear, Electron microscopy, Laser diffraction spectroscopy*

1. INTRODUCTION

Internal combustion engine produces soot as a result of incomplete fuel combustion. It has been shown that only some soot fraction which is produced within the engine reaches to the atmosphere through the exhaust pipe, studied by Daido *et al*. [1]. The remainder is deposited on the cylinder walls and piston heads. Soot which is retained in the engine, mainly in lubricant, few percent is attributable to blow-by gases. The remainder results from piston rings scraping away soot deposits in the cylinder, which then end up in the sump. Then it is transported around the engine where it can be entrained into component contacts. Ryason *et al*. [2] concluded that soot particles are abrasive because they were found to generate grooves and breakouts in metal surfaces. Ratoi *et al*. [3] showed that dispersed carbon black rapidly abraded additive reaction films. Aldajah *et al*. [4] and Yamaguchi *et al*. [5] found that the presence of soot particles reduces the thickness of anti-wear films and act as an abrasive element. George *et al*. [6] found that the soot contaminating in lubricant changes the chemical properties. The performance of anti-wear lubricant additive can also be negatively impacted and lead to increase wear and premature engine failure. Esangbedo *et al*. [7] concluded that severe oil thickening is linked to soot agglomeration in diesel engine oil. The ineffectiveness of dispersant to retard particle growth is attributed to poor soot functionalization.

Gautam *et al*. [8] studied the influence of soot on wear using reciprocating Ball-on-flat tribology test. The detrimental effect of soot is quite clear evident as the average wear with soot contamination is higher than wear without soot

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contamination. Aldajah *et al*. [9] had conducted a Four-ball tribology test. The samples lubricating oils were tested which are different in soot containing. They found that there appears to be no clear relationship between the soot content and steel ball wear volume. The fresh oil gives the result as near to zero wear volume, an oil adding soot made larger wear volume. Hu *et al*. [10] added Carbon Black into the base oil and formulated lubricant at mass percentages of 0 – 8% by weight. The kinetic viscosities of oil samples were measured using a viscosity meter at 40 ºC and 100 ºC. They found that the viscosity has slightly increased when raising the containing of Carbon Black. The average wear scar diameters of steel ball have increased among soot content increasing. The friction coefficient has trended increasing among the samples which have more content of carbon black. However, it has been noticed that the friction coefficient was small drop at low percent concentration of carbon black.

Truhan *et al*. [11] had tested a reciprocating Ring-and-flat bench test to demonstrated mechanism in cylinder liner. They found that the viscosity has been increased among the soot concentration is increasing. The chemical activities of soot particles and their reactions with additive prevent the formation of liquid boundary layers on metal surface so the surface becomes easier for scratching. They also found that wear rate of piston wall has trended to increase when soot content is raising but there is not affecting to the case of piston ring. They concluded the soot and particulate contents have a major effect on wear of the cast iron flat and less of an effect on the ring. Soejima *et al*. [12] had conducted the Cam-follower bench test with many conditions. They found that used oil has more wear than fresh oil, and also same trend as adding Carbon Black. The oil adding Carbon Black give the result in more damaged than fresh oil. In case of adding dispersant, they found the particle size is kept small but note that this case made more wear rate too. Jao *et al.* [13] reported that soot particles are sufficiently hard to abrade metal parts.

The properties of soot particle very much depend upon thermodynamic of combustion in the engine. The structural complexity of soot varies depending on type of engine and its operating conditions. Karin *et al*. [14,15,16] reported that the primary soot particles are typically spherical in shape using Transmission Electron Microscopy (TEM). The diameter of primary and agglomerate soot particles are in the range of 20 - 80 nm and 100 - 300 nm, respectively. Wear mechanism induced by the presence of soot is not successfully understood yet. Therefore, the impact of soot on metal wear mechanism is needed to investigate using advanced technique for better understanding.

2. Experimental Setup

In order to understand what behavior occurred in lubricating oil in diesel engine. The first step is trying to inspect the amount of soot and wear contamination in used oil of diesel passenger cars and trucks using ASTM E-2412M and ASTM D-6595, respectively. Used oil samples are kept from diesel engine crank case through oil level deep-strip hole. The volunteer cars and trucks are from different mile age and all vehicles are at end of oil change interval. Lubricating oil sampling equipment consist of vacuum pump, plastic tube and plastic bottle, as shown in Fig. 1. The oil samples are kept from 2,000 cc diesel engine of five passenger cars, 2,500 cc diesel engine of five passenger cars and 7,684 cc diesel engine of five heavy duty trucks. Second, the investigation by TEM (JEOL JEM-2010) is a study of physical properties of both carbon black and engine's soot, as shown in Fig 2. It presents the physical shape of single particle and also presents the complexity of their agglomerate.

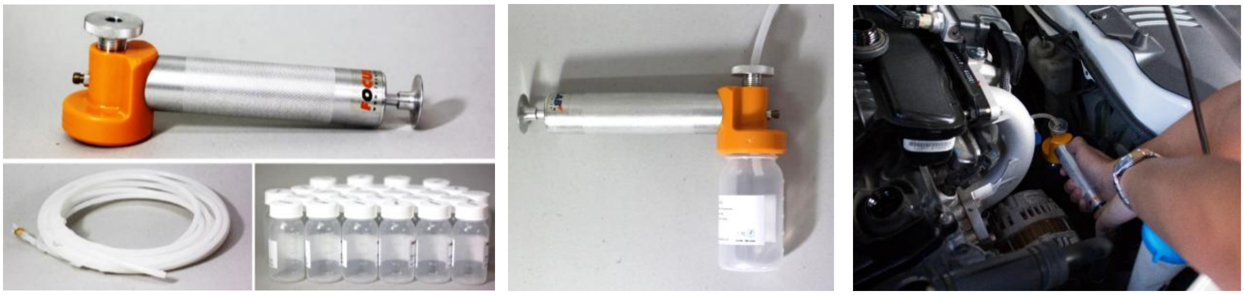


Fig. 1. Lubricating oil sampling equipment and sampling the lubricating oil from diesel vehicle.

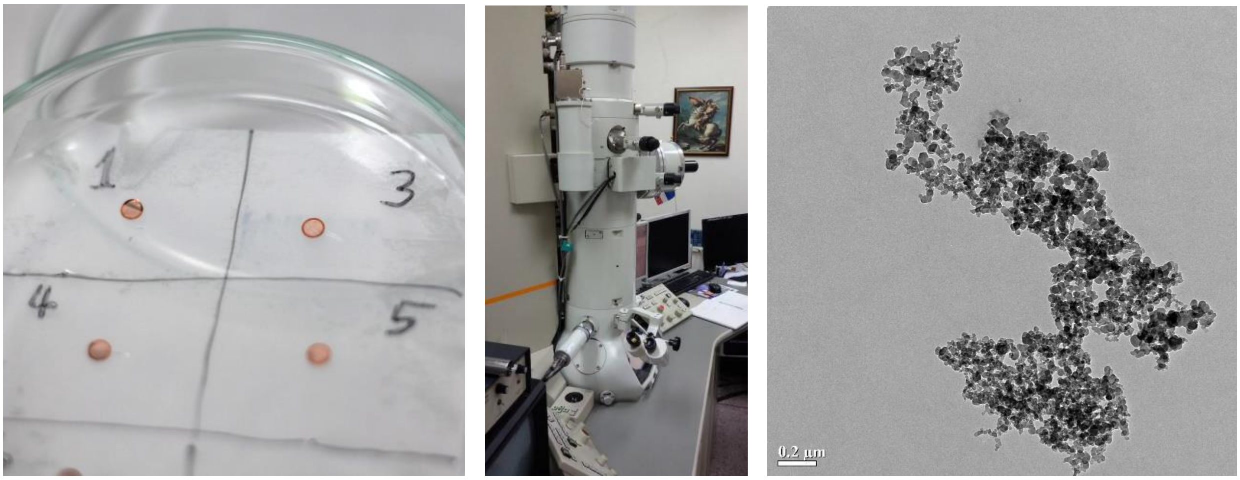


Fig. 2. Carbon black and soot powder prepared in copper grid for Transmission Electron Microscopy.

The main purpose of the third step is to analyze the statistical association between carbon black distribution and lubricating oil additive by means of Laser particle size detector. Behavior of soot dispersing in water, palm oil and formulated oil. Particle size distribution is investigated by high resolution optical microscope and laser diffraction technique. Formulated oil viscosity, oxidation, nitration, and contamination are also investigated. Oil additives in formulated oil are investigated by X-ray fluorescence (XRF). The final step is to measure wear due to carbon black by means of Four-ball tribology. The fresh lubricating oil and oil which were contaminated with carbon black at 1% weight samples were prepared. The lubricating oil samples were tested via using this tribology test method as follow the ASTM D-4172. Three 12.7 mm diameter steel balls are clamped together and covered with the lubricating oil sample to be evaluated. A fourth 12.7 mm diameter steel ball, referred to as the top ball, is pressed with a force of 392 N into the cavity formed by the three clamped balls for three-point contact. The temperature of the test sample is regulated at 75 ºC and then the top ball is rotated at 1200 rpm for 60 minutes. The testing results are compared by using the average size of the scar diameters worn on the three lower clamped balls, during testing. The wear scar results are observed by Optical Microscope (OM), firstly. After inspect by OM, wear that appear on both upper balls and lower balls are also inspected more by convert the captured images from optical microscope to black and white color and gray scale color for helping investigation morphology on wear scar. Moreover, 3D image rendering system is used to investigate wear shape and measure surface roughness. This test will help us to know more details in wear mechanism which occurred during test especially the test with soot contamination oil samples.

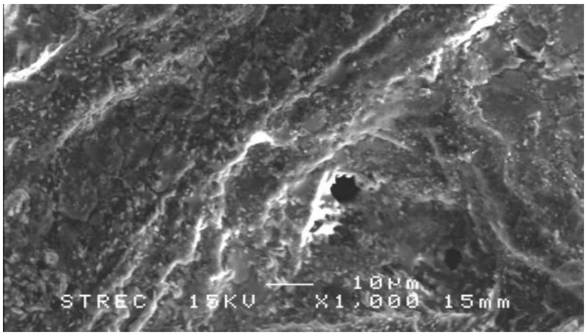
3. Results and Discussion

3.1 Soot and metal wear contamination in used lubricating oil

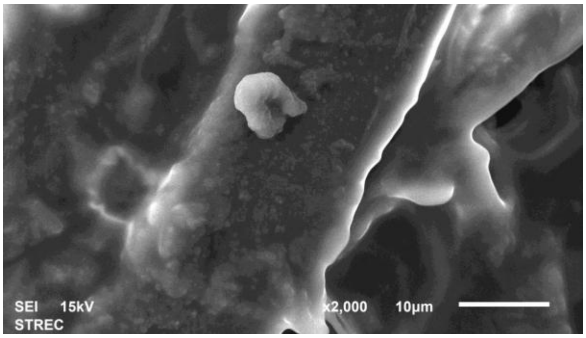
The oil interval changes of passenger car differ from truck where truck has more long last interval change than passenger car. Passenger car normally changes oil when oil age reach 10,000 km and 20,000 km for truck. This case is trying to investigate the amount of soot that contaminate in diesel engine oil at after end of interval change. The results are shown in Table 1. The average value of soot contaminations are 0.64%, 0.41% and 0.42% by weight for 2,000 cc, 2,500 cc and 7,684 cc engines, respectively. It is an average value of soot contamination in all samples of 0.49% by weight. This results indicated that, among oil using through end of oil life, soot contaminated in oil is less than 1% by weight. In past century, the amount of soot founded in engine reached in range of 3 - 5%. This may be the effect by fuel technology and engine development by manufacturer that trying to reduce the producing of particulate matter in the engine. The results have been noticed that the amount of metal wear in small engine is higher than large size engine. It may be because small engine has to operate in higher speed than large engine to reach maximum torque and power during running. Figure 3 shows Scanning Electron Microscopy (SEM) and OM images of soot particle in both accumulation (fine particle, PM2.5) and coarse (PM10) modes. Micro particle of metal wear also clearly observed by OM. The results show that iron is the highest percentage and followed by copper and aluminum respectively because most of engine parts are made by iron, especially in the parts which have more rubbing surface areas, such as a cylinder wall area.

Table 1: Soot and metal wear contamination in used oil

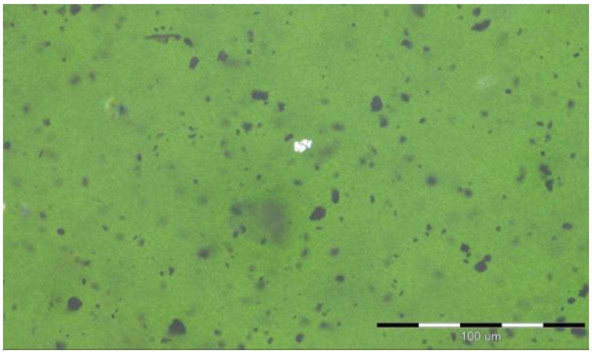
Group	Engine size (cc)	Amount (Vehicles)	Average soot contamination (% wt.)	Average wear contamination (ppm)	
				Total metal	Iron
1	2,000	5	0.64	87.3	58.8
2	2,500	5	0.41	38.1	19.4
3	7,684	5	0.42	11.7	4.4



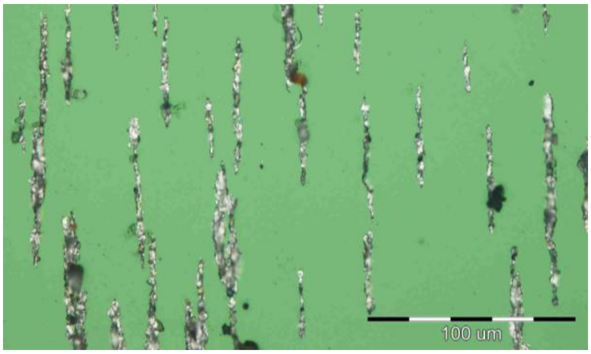
(a) Accumulation mode of soot particles



(b) Coarse mode of soot particles



(c) Coarse mode of soot particles

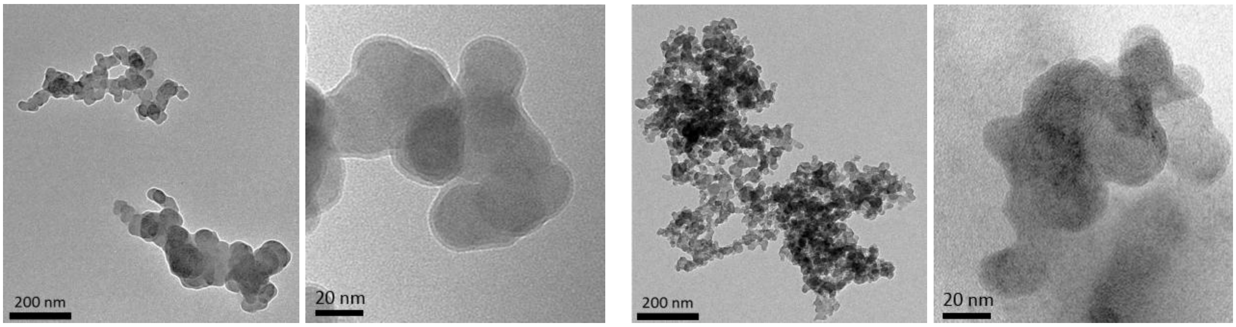


(d) Metal wear particles

Fig. 3. Soot and metal wear contamination in diesel engine's used lubricating oil using (a, b) Scanning Electron Microscope and (c, d) Optical Microscope.

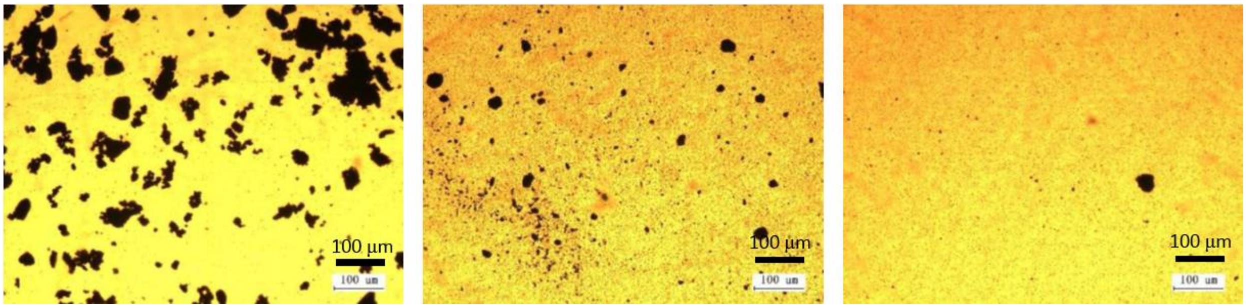
3.2 Nanostructure of soot particle emission

There are approximately 50 images captured of engine diesel's soot and commercial carbon black particles. The images present both primary single and agglomerates particles. The agglomerate diameter size is approximately 100 - 300 nm in both soot and carbon black. The primary single particle size are in range of 20 - 80 nm, as shown in Fig. 4. Most of soot are clearly presented smaller size than carbon black. The mean values of diesel soot and carbon black primary particle diameter are 30 nm and 58 nm, respectively. It showed that the commercial carbon black, representing to typical soot, average size is double to the average of diesel engine exhaust system soot. It could be an effect from carbon black production process. When commercial carbon black is produced in low temperature and low pressure atmosphere, it may become larger in size than the primary particle which is conducted in combustion chamber of internal combustion engines. In this research, soot particle contamination was simulated using carbon black to eliminate the impact of unburned hydrocarbon fuel (Soluble organic fraction) condense on the surface of soot (Pure carbon).



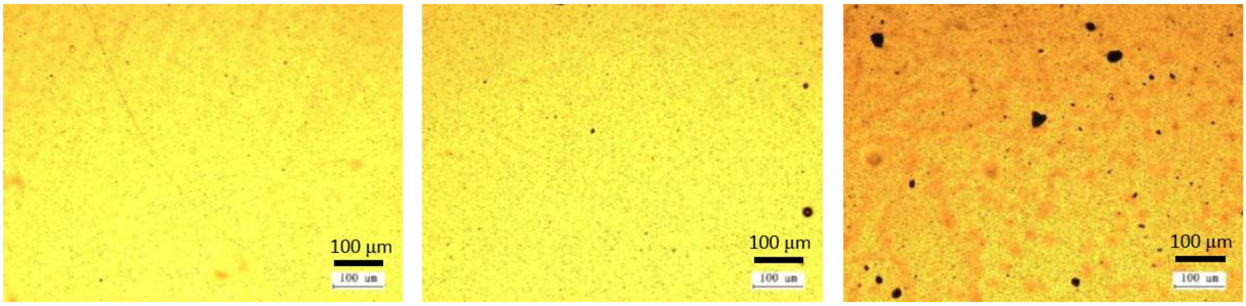
(a) Commercial carbon black (N330) (b) Diesel engine's soot fine particle

Fig. 4. TEM images of (a) carbon black and (b) diesel engine's agglomerate soot particles.



(a) Water (b) Palm oil (c) Formulated oil lubricant

Fig. 5. Optical images of 1% weight of carbon black particles distributing in different type of liquids.



(a) 0.25% weight (b) 0.5% weight (c) 2.0% weight

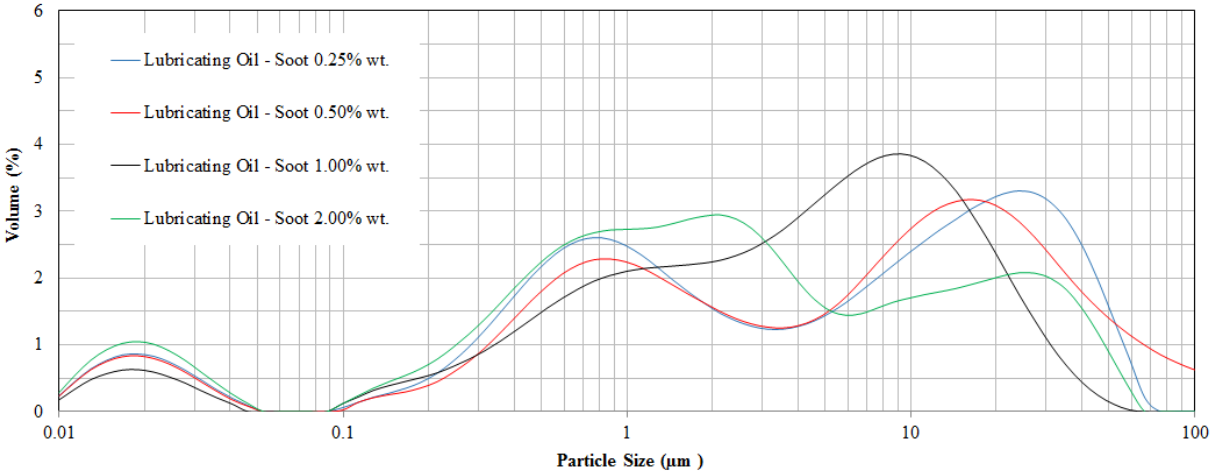
Fig. 6. Optical images of different carbon black particles quantity distributing in formulated oil lubricant.

Table 2: Oil Properties of Diesel Engine Lubricant Grade SAE 15W-40

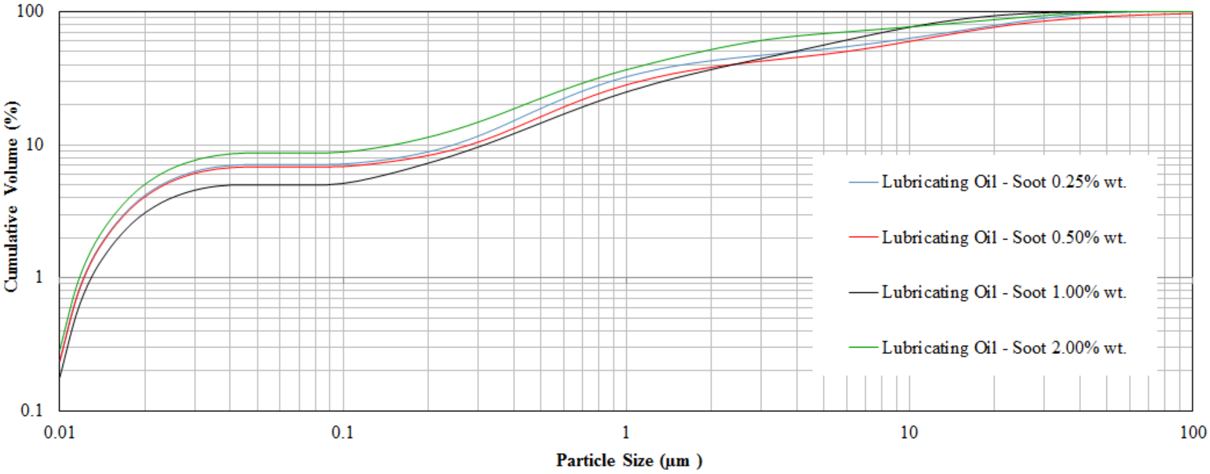
Oil conditions			**Oil additives**		
Viscosity (40°C)	cSt	104.2	S	ppm	0.822
Viscosity (100°C)	cSt	13.8	Ca	ppm	0.593
Oxidation	Abs	4.0	Zn	ppm	0.132
Nitration	Abs	5.5	P	ppm	0.128
TBN	Mg KOH/g	8.2	Cl	ppm	171.0

Table 3: Oil Properties of Diesel Engine Lubricant Grade SAE 15W-40 Mixed with Carbon Black

Oil condition		**Oil mixed with carbon black**				
		New oil	0.25% wt.	0.5% wt.	1.0% wt.	2.0% wt.
Viscosity (40ºC)	cSt	104.2	101.3	101.6	98.9	105.6
Viscosity (100ºC)	cSt	13.8	13.9	13.7	13.9	14.0



(a) Particle size distribution



(b) Particle cumulative volume

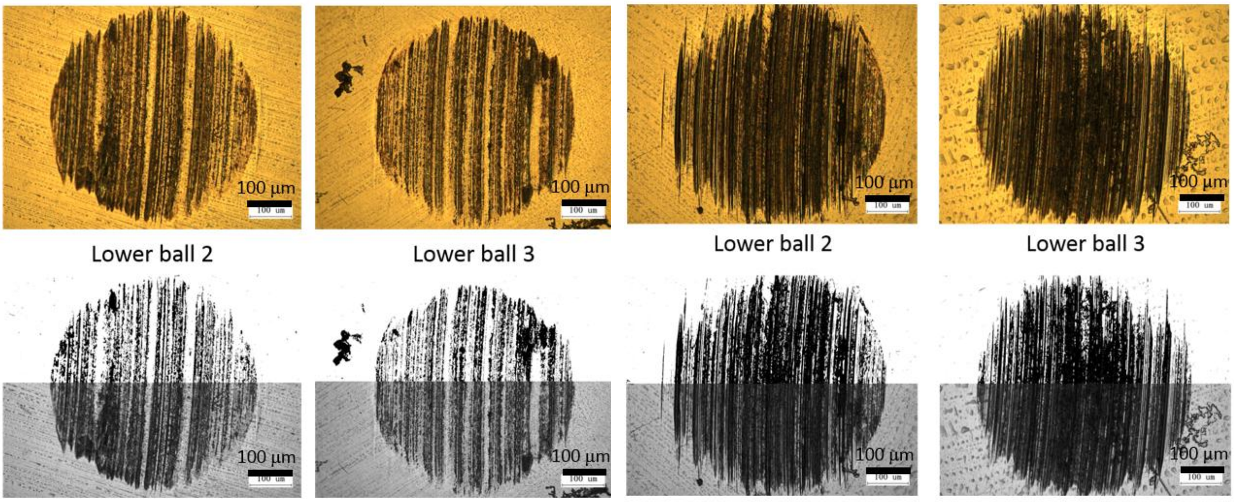
Fig. 7. Carbon black particle (a) size distribution and (b) cumulative volume in lubricating oil by Laser Particle Diffraction Spectroscopy.

3.4 Soot particle distributing in liquid

Table 2 shows oil properties of diesel engine lubricating oil which is viscosity grade as standard SAE 15W-40. There are several condition for investigating such as viscosity, oxidation, nitration, total acid number, and total base number. Oil additives in formulated oils are analyzed by using X-ray fluorescence (XRF). The viscosity of lubricant mixed with carbon black is shown in Table 3. It was clearly observed that the small amount of carbon black contamination is not significant impact on lubricant viscosity. Figure 5 shows the dispersing of carbon black 1% weight in water, palm oil and formulated oil lubricant. Results of the particle-dispersing observation by optical microscope are accordance with assumption before the experiment that soot dispersion is not similar at different liquid significantly. Additionally, the result presents the carbon black accumulates more clearly if its percentage by weight increases, as shown in Fig. 6. As expected, when looking at the sample of the carbon black-dispersing results in formulated oil lubricant, theirs dispersing is significantly better than in water and palm oil because an oil lubricant has an additive that help to disperse soot particle. After investigate the distribution of carbon black in liquid by using optical microscope, this case study is also investigated liquid sample mixed carbon black by laser diffraction technique. Figure 7 shows size distribution of carbon black powder mixing in formulated oil lubricant at 0.25%, 0.5%, 1% and 2% by weight. There are small primary particles which have the size of 10 - 60 nm. The first and second group of agglomerate particles are size of 0.1 - 3 microns and 3 - 80 microns.

3.5 Impact of soot on metal wear using four-ball tribology

The wear scar diameter was measured under optical microscope. Figure 8 shows the microscopy image of wear scars found on lower balls after 60 min running time in lubricating oil (a) without carbon black and (b) with carbon black. The results showed that wear diameter of four metal balls in case of testing with soot are larger than wear diameter of four balls without soot. Similarly, wear diameter of four balls in lubricant B with soot are larger than wear diameter of four balls without soot. The average wear diameter of balls in case of testing in lubricating oil without carbon black is 493 microns and in lubricating oil with carbon black is 537 microns. Figure 9 shows the surface image from 3D rendering optical microscope. They are helping to inspect morphology of wear scar surface and helping to understand wear mechanism of four-ball tribology system. Furthermore, this tool is also providing the roughness value which is a good information for calculating and helping defining the lubrication regime of the test. Roughness on wear scar of balls in case of testing in lubricating oil without carbon black is 0.07 microns and in lubricating oil with carbon black is 0.19 microns, as show in Fig. 9. It might be expected that carbon black particle can enter to the contact surface zone and then make wear on metal surface. However, primary and agglomerate particle size distribution might be directly impact on metal wear mechanism. Therefore, soot dispersant additives would be a key parameter of engine lubricating oil development. The samples with carbon black seem to be lower friction torque than the sample without carbon black, as shown in Table 4. The average friction torque in testing with lubricating oil is 2.01 N.mm and the average friction torque in testing in lubricating oil with carbon black is 1.62 N.mm. Figure 10 shows the comparison of particles size distribution before and after Four-ball tribology by Laser Particle Diffraction Spectroscopy. After Four-ball tribology, it was clearly observed that agglomerated carbon black and wear particle sizes are in the range of 10 – 100 nm and 100 nm - 7 microns.

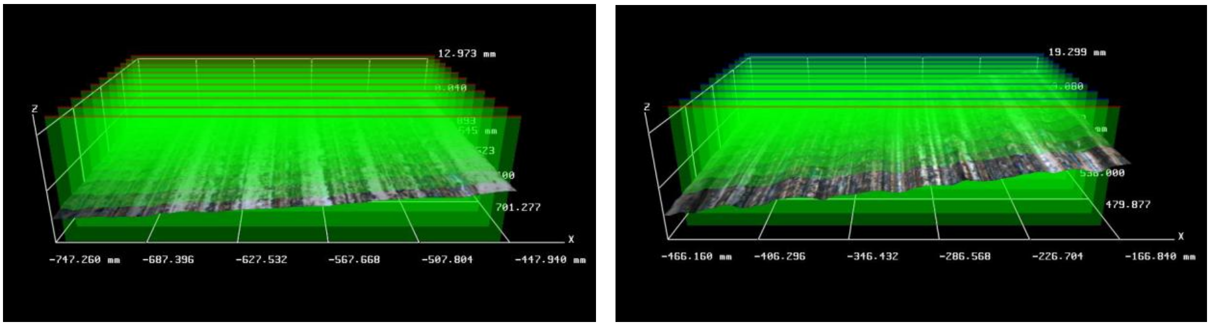


(a) Lubricating oil (b) Lubricating oil with carbon black

Fig. 8. Wear scar on steel ball of (a) lubricating oil and (b) lubrication oil with carbon black 1% wt.

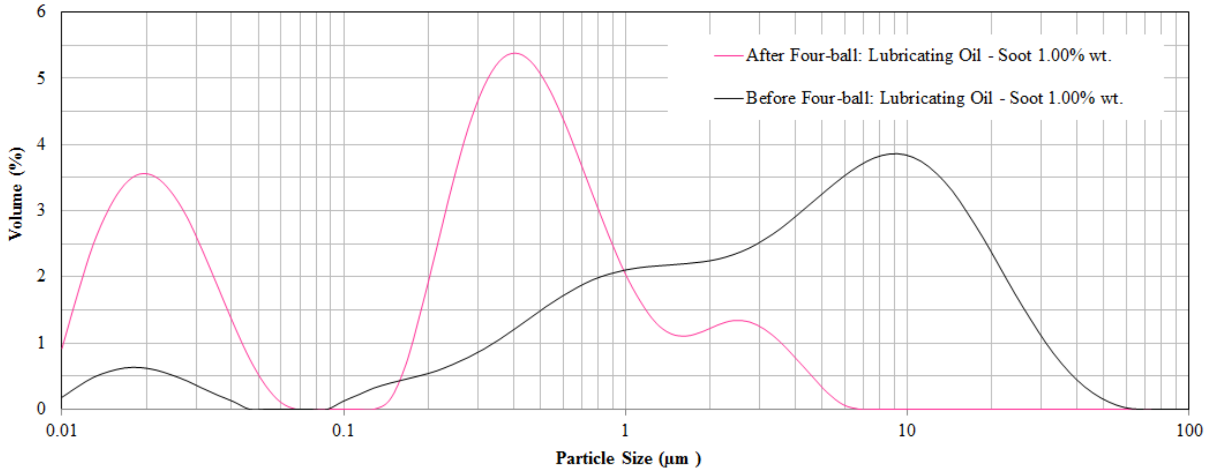
Table 4: Wear scar diameter, roughness and friction torque

Wear conditions		Lubricating oil	Lubricating oil with carbon black
Wear scar diameter	microns	493	537
Roughness	microns	0.07	0.19
Track density	line per mm	237	261
Friction torque	N-mm	2.01	1.62

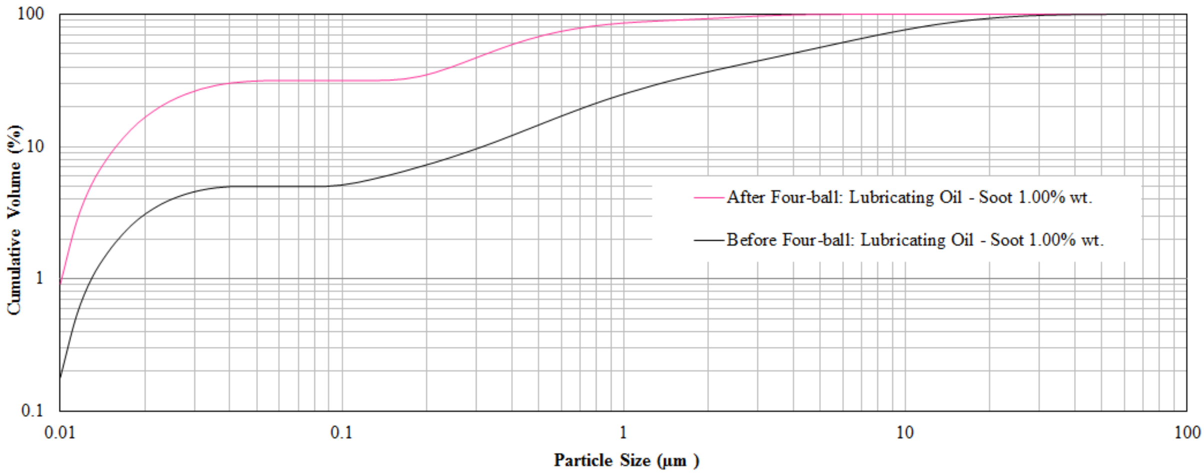


(a) Lubricating oil (b) Lubricating oil with carbon black

Fig. 9. Wear roughness on steel ball of (a) lubricating oil and (b) lubrication oil with carbon black 1% wt.



(a) Particle size distribution



(b) Particle cumulative volume

Fig. 10. Carbon black and wear particles (a) size distribution and (b) cumulative volume in lubricating oil before and after Four-ball tribology test by Laser Particle Diffraction Spectroscopy.

4. Conclusion

In case of investigating lubricant from passenger car and truck, it has been showed that soot was contaminating in range of 0 - 1.0% by weight in diesel engine used oil. The results also induced to show that soot may be the cause of increasing metal wear from engine parts. Carbon black can be used to be a simulant of soot because the both have a similar morphology. However, they still have small difference in the view point of the particle diameter size. The tested results presented the difference of soot particle size distribution in liquid that there are highly agglomerated in water, smaller level of agglomeration in palm oil, and well dispersing in formulated oil. Based on the four-ball tribology result, soot in lubricating oil may act as a rolling element and result in reducing friction between metal surfaces. Another idea is that soot may react with lubricating oil by both physical and chemical reaction and result in increasing oil film thickness between contact surfaces. The most significant result that the appropriate particle size (20 nm – 300 nm), which is near to oil film thickness between metal surface contacts, is the dominant cause of making wear. High level of proper particle size (20 nm – 100 nm) contaminated in oil will increase probability of rubbing process. Furthermore, soot dispersing oil additives might have a most significant effect on engine wear.

5. Acknowledgement

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