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Original Article

Impact of soot nanoparticle size and quantity on four-ball steel wear characteristics using EDS, XRD and electron microscopy image analysis

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ABSTRACT

The effect of soot contamination on the tribological performance of engine oil was investigated. Carbon black is introduced to simplify diesel engine soot contamination. Besides, the tribological performance issue is verified by a four-ball tribometer. The steel ball worn surfaces were studied by Scanning Electron Microscopy (SEM), Optical Microscope (OM) and Energy Dispersive X-ray spectroscopy (EDX). In addition, Transmission Electron Microscopy (TEM) was used to investigate the morphology and nanostructure of soot and carbon black. According to the four ball test results, the average wear scar diameter of steel ball tested with engine oil blended with N220, N330, N550 and N660 by 1% by weight is larger than that of pure engine oil by 26%, 38%, 41%, and 39%, respectively. The wear scar diameter tends to increase after blended larger size of carbon black particle. The steel balls tested with formulated engine oil without soot contamination and with soot contamination by 0.5 wt%, 1 wt%, and 2 wt% have average wear scar diameters of 621, 567, 784 and 894 nm, respectively. On the other hand, wear scar roughness of steel balls tested with formulated engine oil without soot contamination and with soot contamination by 0.5 wt%, 1 wt%, and 2 wt% were 2.28, 0.25, 1.49 and 1.76 μm, respectively.

Consequently, quantity of the soot nanoparticle approximately 0.5% by mass in engine oil significantly plays an important role in steel ball wear scar diameter and surface roughness reduction. Moreover, the agglomerated soot which is larger than the oil film thickness might block the lubricant from entering the contact. It leads to the breakdown of the oil film thickness resulting in increasing adhesive wear on the worn surface.

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1. Introduction

Internal combustion engine's soot is composed of the agglomeration of pure carbon atoms, which are the product of the incomplete combustion between hydrocarbon fuel and oxygen in the air. Due to the thermionic emission and electron capture, carbon atom is combined to be arranged in the hexagonal lattice of carbon 6, which is enlarged to become graphene carbon fringes. The graphene carbon fringes are then matched as carbon platelet and overlapped to be layered aggregate. When there are more layers, the layered aggregate carbon platelet will be extended into different circular directions. Owing to the limitations of electrostatic force of carbon charge, shear force by the flow of exhausted gas, drag force from Brownian motion of gas molecule, and gravitation of soot particle, these lead to the combination of the graphene carbon fringes, which have the diameter of no more than 80 nm, and are called primary single nanoparticle [1,2].

The single primary nanoparticle may be combined with two or more single primary nanoparticles to become agglomerated particles, which are like a cluster of grapes.

The nanostructure of soot particles is investigated by the Scanning Electron Microscopy (SEM) and the Transmission Electron Microscopy (TEM). According to the soot nanostructure analysis, it is found that the graphene carbon fringe has the length in the range of 0.1–7 nm. The image, which has an area of 100 nm squared, the width of 10 nm, and the length of 10 nm is transmitted by the high-resolution transmission electron microscopy. This image has been focused to measure the total length of carbon platelet per area. The analysis of carbon platelet with approximately 100 nm squared has been found the total fringe length of 190–230 nm. This can be used to calculate the carbon atom density of the soot which has 85–105 atoms per nanometer cubed. Moreover, the engineering carbon density in the soot has 1.75–1.85 g per centimeter cubed (g/cm^3) [4] The value of mechanical hardness of the soot can be measured by means of carbon Plasmon energy method. The value of electron energy loss spectra is measured by the high-resolution TEM. After that, the relationship between the engineering carbon density of soot nanoparticle and the hardness of soot nanoparticle is simulated. The hardness of different types of soot nanoparticles can be measured by the linear relationship between the carbon density and the hardness of glassy carbon, graphite, diamond-like carbons, and diamond. It is stated that the hardness of glassy carbon and diamond was approximately 250 kg/mm^2 and 10,000 kg/mm^2, respectively. It can be concluded from the result that each type of single soot nanoparticle has the value of hardness higher than that of metal. This indicates that soot nanoparticle is hard enough to destroy parts of the engine [5]. Concerning the deposition of the soot to a piston ring, a cylinder, and a lubricating oil bath, the researchers found that soot nanoparticles can flow to the close intake and exhaust valve set of the valve train including a variety of mechanism such as sliding, rolling–sliding and reciprocating contact. The soot particle has a direct effect on the engine wear [6]. According to the analysis of used lubricating oil from cars and trucks, it was found that there was a mixture of soot: approximately 0.7% [7]. In the consequences of previous review, it has been found that there were very few research trends on the study of quantity on steel ball wear mechanisms which is depended on the various properties of primary single soot's nanostructure. Moreover, this research work extended and tested the different conditions of soot contamination in engine oil under four ball tests with four types of carbon black or without carbon black at different weight ratio. This article aims to investigate the impact of soot nanoparticle size and quantity, which influences on the steel ball wear characteristics and mechanisms. The Four-ball wear tester is used to test the wear on the steel ball, which is immersed in engine oil. The soot nanoparticle, formed by an incomplete combustion between the molecules of fuel and oxygen in the engine combustion chamber, is simulated by using carbon black with single primary nanoparticle which has revealed the various sizes and different quantity of contaminants.

2. Experimental setup and methodology

The formulated SAE0W30 engine oil was used in this research. Table 1 shows physical and chemical properties of engine oil. The engine oil properties including viscosity, oxidation and nitration, and total base number (TBN) were measured according to ASTM-D445, ASTM-E2412M, and ASTM-D4739, respectively. The quantity of the metallic oil additives was measured by X-ray fluorescence spectroscopy (XRF). In addition, mean and minimum thickness of lubricant film (h_c) at various temperatures can be calculated by “Elastohydrodynamic Lubricant (EHL)” equation presented by Hamrock and Dowson [8] as shown in the following equation:

$$\frac{h_c}{R} = 2.69\left(\frac{U\eta_0}{ER}\right)^{0.67}(\alpha E)^{0.53}\left(\frac{W}{ER^2}\right)^{-0.067}\left(1-0.61e^{-0.73k}\right) \tag{1}$$

where η_0 is viscosity at atmospheric pressure of the lubricant, R is radius of curvature, U is entering surface velocity, E is Young's modulus, α is pressure–viscosity coefficient, W is contact load and k is elasticity parameter [9,10].

Table 1 – Properties of SAE5W30 engine oil.

Oil conditions			Oil additives		
Viscosity at 40 °C (cSt)	ASTM-D445	66.9	S	ppm	1570
Viscosity at 100 °C (cSt)	ASTM-D445	11.2	Ca	ppm	1020
Oxidation (Abs)	ASTM-E2412M	7.7	Zn	ppm	763
Nitration (Abs)	ASTM-E2412M	5.8	P	ppm	658
TBN (mg KOH/g)	ASTM-D4739	10.2	Mo	ppm	97

Oil conditions	Calculated Elastohydrodynamic lubricant oil film thickness (nm)	
	Mean	Minimum
Oil temperature of 40 °C	62.8	39.5
Oil temperature of 75 °C	40.2	25.1
Oil temperature of 100 °C	18.9	11.7

Table 2 – Properties of soot's primary single nanoparticle.

Properties of primary single soot nanoparticle	Type of primary single soot nanoparticle					
	Biodiesel Engine's Soot	Diesel Engine's Soot	N220	N330	N550	N660
Maximum diameter (nm)	57.5	58.2	49.0	74.4	83.4	120.2
Minimum diameter (nm)	18.1	16.1	11.3	9.4	15.9	14.9
Mean diameter (nm)	28.7	30.5	23.6	30.4	44.6	54.0

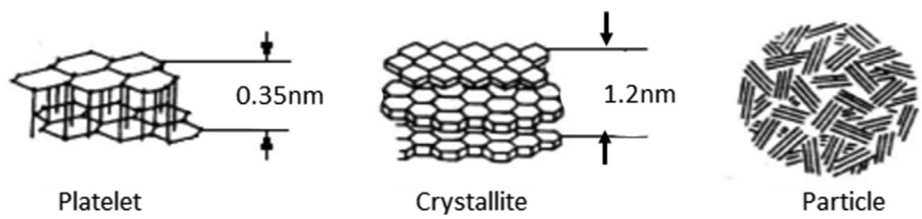


Fig. 1 – Depicts the dimension of carbon platelet, the distance between crystallite structure and combination of carbon fringes as a soot particle [3].

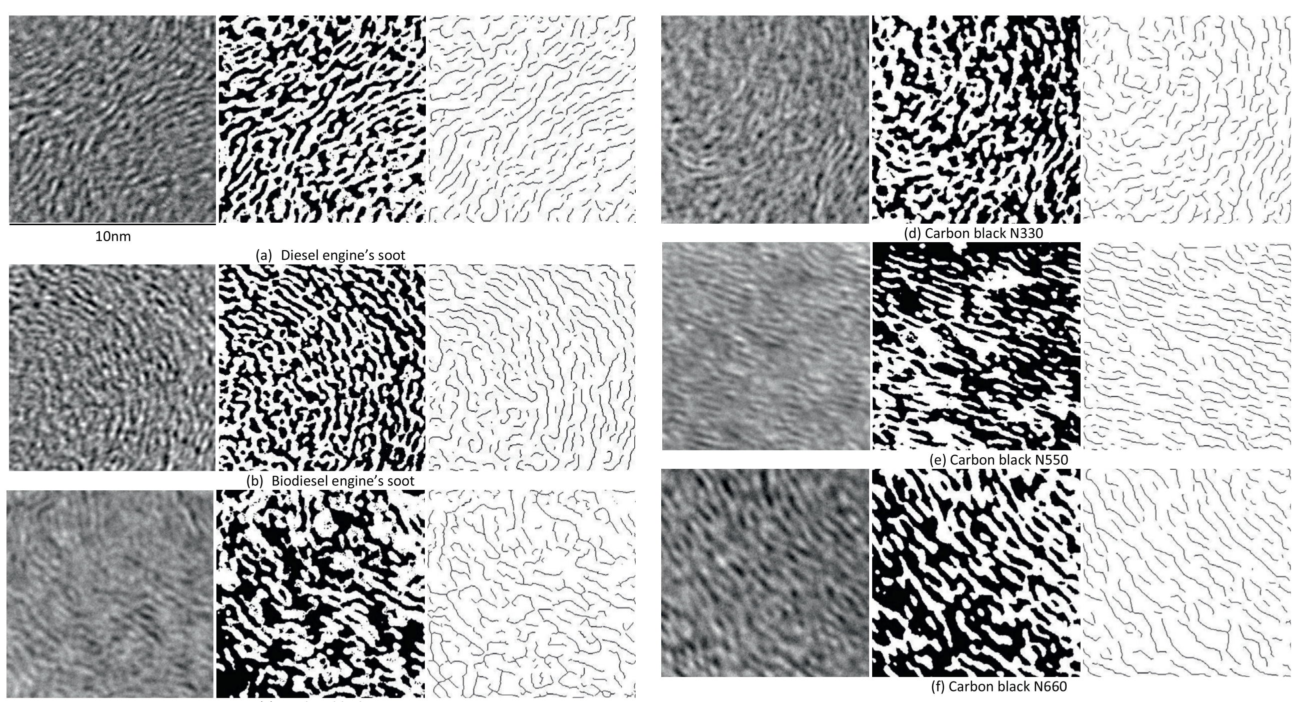


Fig. 2 – TEM micrographs of (left) 100 nm^2 focused area (middle) black-white (right) skeletonize images of (a) diesel engine's soot, (b) biodiesel engine's soot, (c) Carbon black N220, (d) Carbon black N330, (e) Carbon black N550 and (f) Carbon black N660.

The TEM (TEM, JEOL JEM-2010) was employed in this research to investigate the morphology and the nano-structure of soot and carbon black. The agglomerate and single primary particle of soot and carbon black have already discussed in the previous study of related article. Carbon black, the pure carbon, has physical characteristics similar to soot from engine combustion [11,12]. The better understanding of internal structure of Diesel or Biodiesel Engine's soot and carbon black indicate the important properties of carbon density, mechanical hardness of the soot and single primary particle size of soot contamination will affect on the lubricity of engine oil performance to the engine system wearing. It can be controlled the size and weight ratio of the carbon contamination in order to reduce the worn surface of the engine parts and to cover the engine life. The unburned hydrocarbon and the metal oxide ashes

Table 3 – Properties of primary single soot's nanostructure.

Properties of primary single soot nanostructure	Type of primary single soot nanoparticle					
	Biodiesel Engine's Soot	Diesel Engine's Soot	N220	N330	N550	N660
Total fringe length (nm/100 nm^2)	208	227	201	204	210	191
Total carbon atoms of 100 nm^2	14,700	17,696	13,818	12,916	12,878	11,110
Atom density (atom/nm^3)	90.59	103.12	91.94	88.90	86.64	87.31
Density (g/cm^3)	1.82	2.01	1.83	1.79	1.84	1.74
Hardness (GPa)	11.20	20.73	14.28	12.33	14.57	9.58

in the carbon black had already been extracted. The commercial carbon black has slightly different diameters of primary nanoparticle depending on its grade. The diameters of 200 samples of single nanoparticle of soot and carbon black were measured. The average diameters of carbon black N220, N330, N550, and N660 were 24, 30, 45, and 54 nm, respectively. The result indicated that the single primary particle size of carbon black N330 and N220 were close to the single primary particle size of biodiesel and diesel engine soot which were 29 and 31 nm, respectively, as shown in Table 2 (see Fig. 1).

Figure 2 shows the focused TEM images of soot and carbon black nanostructure. The images were analyzed by using image processing software (Image J). 100 nm^2 focused area of TEM micrographs of each carbon black primary nanoparticle, black and white image as well as skeletonized carbon black. Each of carbon fringe consisted of carbon atom from incomplete combustion product of hydrocarbon fuel. The TEM micrographs focused area was changed to be two color images of black and white. Then, it was converted to be a skeleton image by image processing software. From the skeletonized image, the line represented carbon crystallite which consisted of carbon fringe. Each of fringe was a pair of graphene sheet [6]. Number and length of the carbon platelet were measured. The result can be used for calculating the density which is in range of 1.79–1.01 g/cm^3 and the hardness can be estimated as shown in Table 3. Moreover, the lamellar nanostructure of carbon platelet characterized by TEM images of carbon black had an average fringe length of 1.07 nm and an average d-spacing of 0.37 nm. Table 4 shows the crystalline structure of carbon black including the interplanar spacing (d-spacing) which was characterized by X-Ray Diffraction technique using Bragg's Law and Scherrer's equations. The XRD result indicated that all the carbon black samples consisted of the lattice plane in graphite structure which has a d_{002} of around

Table 4 – XRD data analysis of carbon black samples.

Properties of carbon black nanostructure	Type of carbon black			
	N220	N330	N550	N660
Inter-planer spacing, d_{002} (nm)	0.36	0.36	0.36	0.36
Crystallite height, L_c (nm)	1.08	0.94	1.20	1.23
Crystallite width, L_a (nm)	5.28	4.70	5.09	5.47
Number of stacking of graphene layers, N	2.98	2.59	3.35	3.38

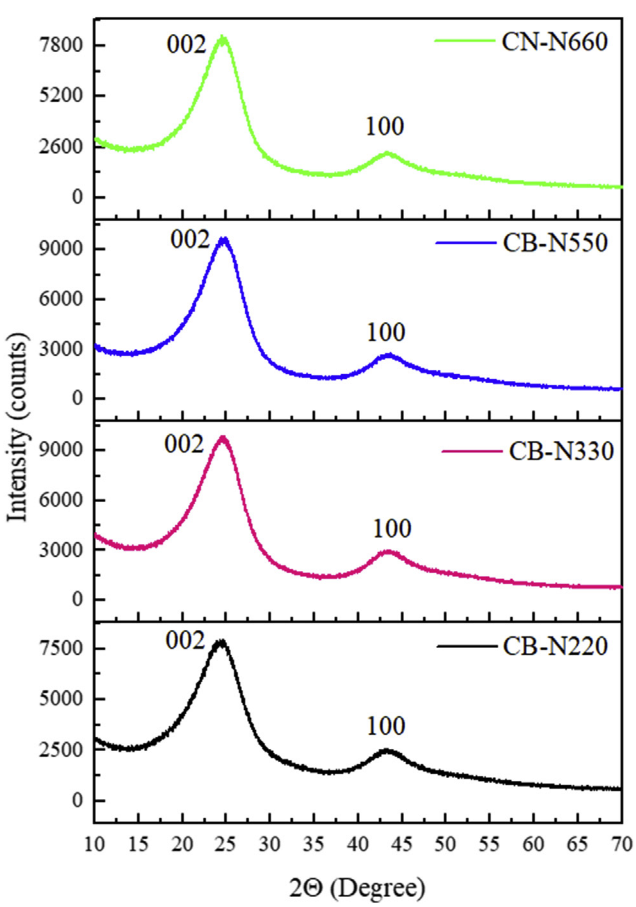


Fig. 3 – XRD spectrum of four types of carbon black samples.

Table 5 – Condition of soot contamination in engine oil for four-ball test.

Condition of soot contamination in engine oil for four-ball test	Percentage of oil and soot contamination (%)			
	Case 1		Case 2	
	Oil	Soot	Oil	Soot
Pure oil without soot contamination	100	0	100	0
1% CB-N220 contamination	99	1	–	–
0.5% CB-N330 contamination	–	–	99.5	0.5
1% CB-N330 contamination	99	1	99	1
2% CB-N330 contamination	–	–	98	2
1% CB-N550 contamination	99	1	–	–
1% CB-N660 contamination	99	1	–	–

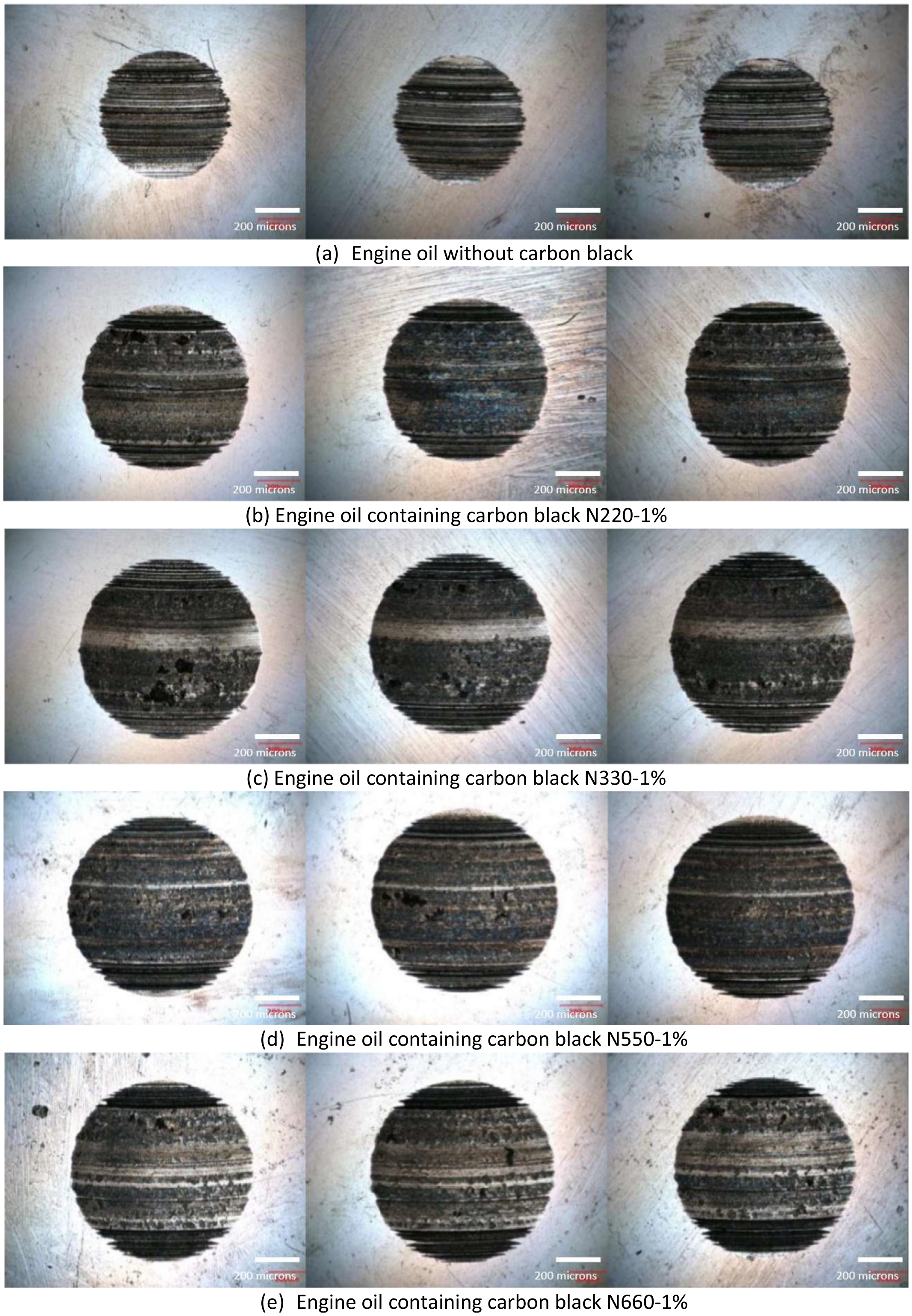


Fig. 4 – Wear scar and surface roughness of the tested balls for (a) oil without carbon black, (b) oil with carbon black N220-1%, (c) oil with carbon black N330-1%, (d) oil with carbon black N550-1% and (e) oil with carbon black N660-1%.

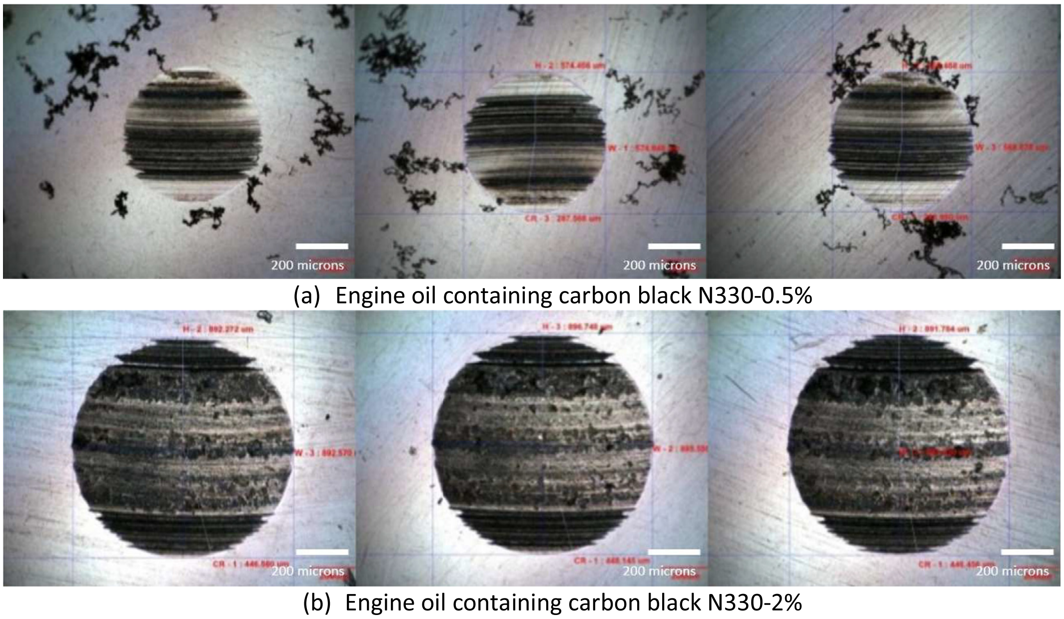


Fig. 5 – Wear scar and surface roughness of the tested balls for (a) oil with carbon black N330–0.5% and (b) oil with carbon black N330-2% by weight.

Table 6 – Impact of soot particle size and quantity on steel ball wear scar diameter and surface roughness.

Condition of soot contamination in engine oil for four-ball test	Average wear scar diameter (micron)	Average surface roughness (micron)
Pure oil without soot contamination	621	2.28
1% CB-N220 contamination	784	1.77
0.5% CB-N330 contamination	567	0.25
1% CB-N330 contamination	860	1.49
2% CB-N330 contamination	894	1.76
1% CB-N550 contamination	877	1.81
1% CB-N660 contamination	865	1.60

Table 7 – Impact of soot particle size and quantity on steel ball wear mechanisms.

Condition of soot contamination in engine oil for four-ball test	Steel ball wear mechanism (%)		
	Abrasive	Adhesive	Fatigue
Pure oil without soot contamination	78	18	4
Oil with 1% by weight of CB-N220 contamination	18	76	6
Oil with 0.5% by weight of CB-N330 contamination	43	56	1
Oil with 1% by weight of CB-N330 contamination	18	80	2
Oil with 2% by weight of CB-N330 contamination	16	83	1
Oil with 1% by weight of CB-N550 contamination	12	83	5
Oil with 1% by weight of CB-N660 contamination	13	84	3

0.36 nm, as shown in Fig. 3. The d_{002} refers to the d-spacing between two consecutive carbon layer (002) planes. The average d-spacing measured from TEM images agreed with the value obtained from the XRD result [13]. Former studies have shown that the interplanar spacing was used to authorize the degree of graphitization, hence, to provide the information of soot formation and growth [14].

The experiment for wear preventive characteristics was divided in two cases as shown in Table 5. First, in order to investigate the impact of soot particle size, different sizes of carbon black (N220, N330, N550 and N660) were used as a surrogate of soot. Each type of the carbon black was mixed in the engine oil by 1 wt.%. Second, different amounts of carbon black N330 (0.5 wt.%, 1 wt.%, and 2 wt.%) were blended to investigate the impact of soot quantity. The performance of fresh engine oil and soot contamination engine oil were tested by a four-ball wear tester following ASTM D-4172 standard. Four chrome alloy steel balls have a diameter of 12.47 mm and a surface roughness of 0.005 microns. Three lower balls were clamped and immersed in the evaluated lubricant. The fourth ball was pressed with force of 392 N and rotated at 1200 rpm. The lubricant was test at 75 °C for 60 min.

The wear scar diameter (WSD) of three stationary balls were measured by using optical microscope (OM) and the average scar diameter was used for comparing the lubricant performance. Three-dimensional optical microscope (3D-OM) was used to measure the roughness of the worn surface. The average roughness of each sample was calculated. In addition,

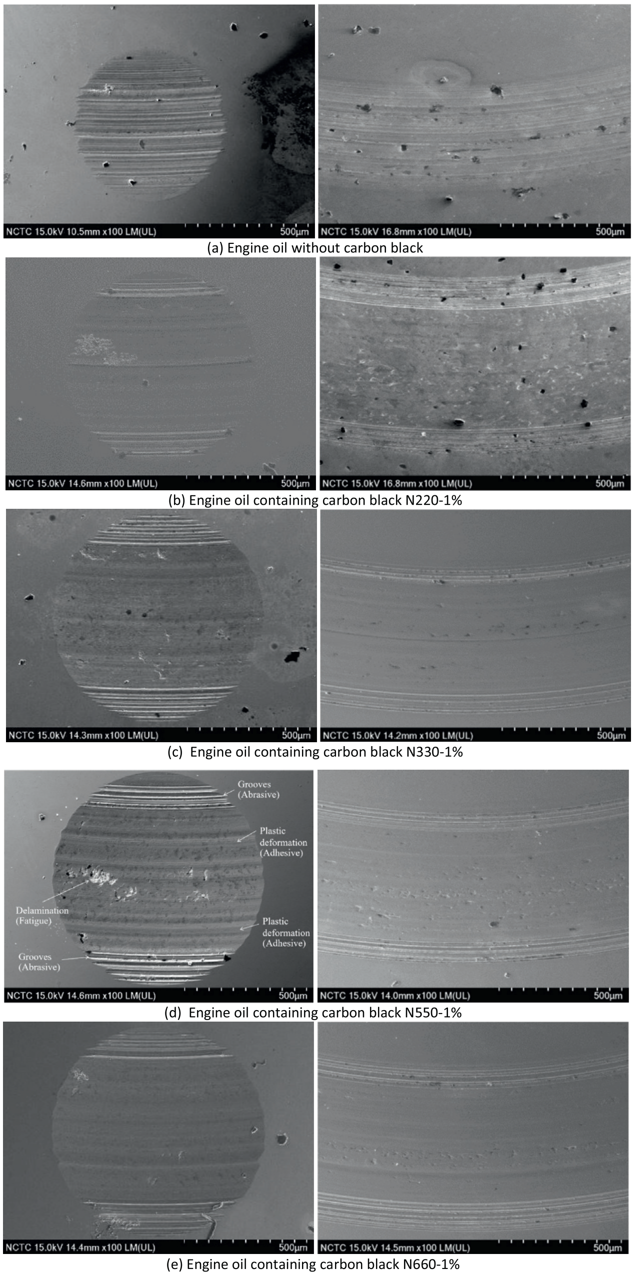


Fig. 6 – SEM micrographs of the lower and upper steel balls wear scar tested with engine oil (a) without soot, with carbon black (b) N220-1%, (c) N330-1%, (d) N550-1% and (e) N660-1% by weight.

worn surface of steel balls were analyzed by using a SEM and Energy Dispersive X-ray Spectrometer (SEM-EDX, SM-6610LV and X-MaxN 50).

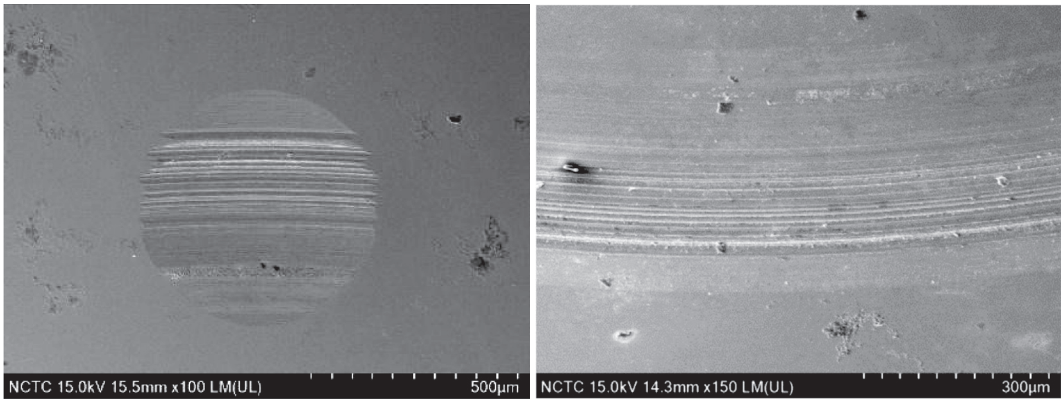
3. Result and discussion

Optical microscope images of steel ball worn surface are shown in Figs. 4 and 5. The average wear scar diameter and average surface roughness are shown in Table 6. The average wear scar diameter (average WSD) of steel ball tested with 1 wt.% of N220 contamination engine oil is larger than that of pure engine oil approximately by 36%; however, the average surface roughness is lower by 27%. Moreover, worn surfaces of the steel ball tested with 1 wt.% of N330, N550, and N660 also demonstrate an increase in average WSD and decrease in average surface roughness. On the other hand, engine oil blended with 0.5 wt. % of N330 causes average WSD and average surface roughness to decrease by 9% and 50%, respectively, when comparing with the worn surface of pure engine oil. The result show that an appropriate quantity of soot nanoparticle could lead to the reduction of steel ball wear scar diameter. With 1 and 2 wt.% of N330 addition to lubricant, average WSD and surface roughness increased significantly when comparing with 0.5 wt.% of N330 addition. It can be concluded that the quantity of soot nanoparticle of more than 1 wt.% addition can severely affect the steel ball wear scar.

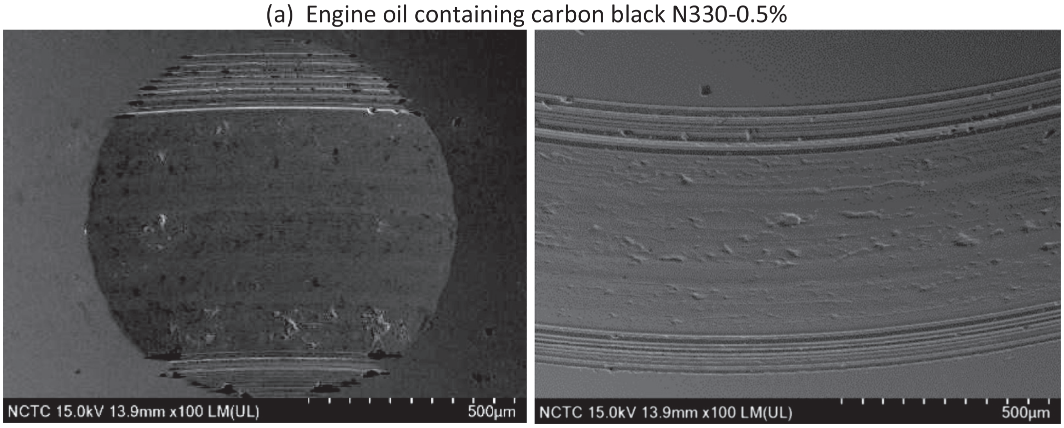
Various forms of wear are divided into three modes in lubricated contact [15,16]. First, adhesive wear occurs when two contact surfaces undergo relative movement with strong adhesion force between rubbing surfaces. Material transfer and plastic deformation can smoothen or roughen the surface [17]. Second, asperities on the hard surface or hard particles in the contacting surface abrade on the surface leaving the deep grooves along with sliding direction. It is described as abrasive wear [18]Third, in the surface fatigue contact, the surface is repeatedly loaded, resulting in crack initiation and delamination on the surface [19]. The possible wear mechanism was identified on the SEM images of the worn steel ball surface.

Figure 6 and Fig. 7 show SEM images of worn surface after four ball wear tests. The classification of wear mode on SEM image are shown in Fig. 5 (d). The center of three stationary ball SEM images were focused for wear mechanism analysis. They were coved with $10.5 \times 10.5\ \mu m^2$ square blocks (432 blocks). Each block was identified by wear mode on the worn surface and the average percentage of each wear mode was shown in Table 7. Worn surface tested with engine oil without carbon black contamination demonstrated the highest percentage of abrasive wear mode which fine and deep grooves are left on the steel ball surface (see Fig. 5 (a)). Figure 5 (b-e) show wear scar of steel ball tested with 1 wt.% of N220, N330, N550 and N660, respectively. Here, surface wear was mostly identified as an adhesive wear making up 76–84% of the total wear at the center of worn surface. The starvation of the engine oil at the contact surface due to the aggregate carbon black particles may prevent the oil from entering to the contact at the center of the steel ball. However, some grooves along with sliding direction also show on the upper and lower area of the circular wear scar in every case.

Figure 7 shows wear scar of the steel ball tested with engine oil blended with 0.5 wt.% and 2 wt.% of N330. The sample with the lowest percentage of N330 has the smallest wear size. It indicates that engine oil can lubricate efficiently on the contacting surface. Abrasive wear scar percentage of this sample is higher than that of the sample tested with 1 wt.% of



(a) Engine oil containing carbon black N330-0.5%



(b) The engine oil containing carbon black N330-2%

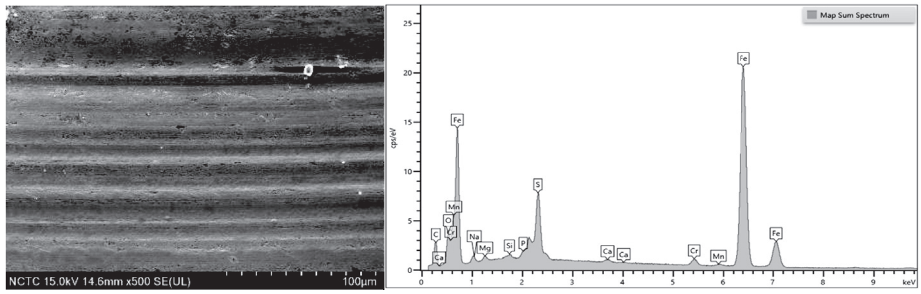
Fig. 7 – SEM micrographs of the lower and upper steel balls wear scar tested with engine oil with carbon black N330 (a) 0.5% and (b) 2% by weight.

N220, N330, N550 and N660. Grooves mainly appear on the center of the steel ball as seen in Fig. 7 (a). However, the wear scar of steel ball tested with 2 wt.% of N330 was observed to have the largest wear scar diameter and highest proportion of adhesive wear. Most of the carbon black particles are in the aggregate form which could block the lubricating oil. Therefore, soot concentration significantly effects the wear scar diameter and wear mechanism.

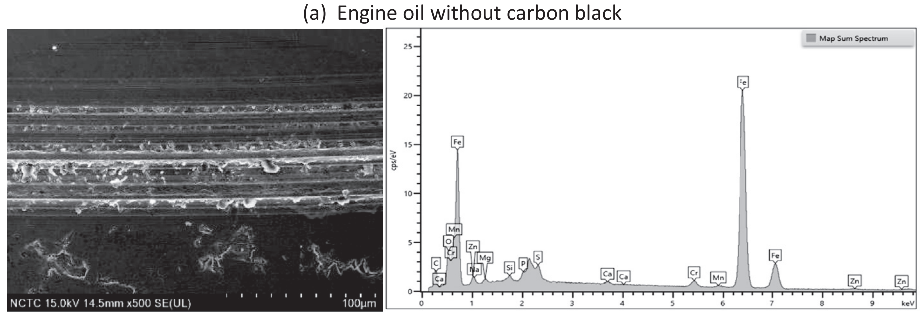
Figure 8 and Fig. 9 show SEM images (500x magnification) and EDX spectrum of worn surfaces. The quantity of the metal elements is shown on Table 8. The steel ball surface mainly consists of iron (Fe), carbon (C) and chromium (Cr). After the steel ball was tested with pure engine oil and soot contaminated engine oil, the EDX analysis show metallic additives on the worn surface such as zinc (Zn), phosphorus (P) and sulfur (S). In addition, adding carbon black into the engine oil led to increasing of C quantity on the worn surface as a result of accumulated carbon black on the steel ball surface. Nevertheless, carbon black with largest diameter (N660) was unable to reach the contact zone due to its size. It shows the amount of C was only 6.59%. The particles larger than the oil film thickness could not enter to the rubbing surface.

Table 8 – EDX elementally analysis on the steel ball wear scar surface.

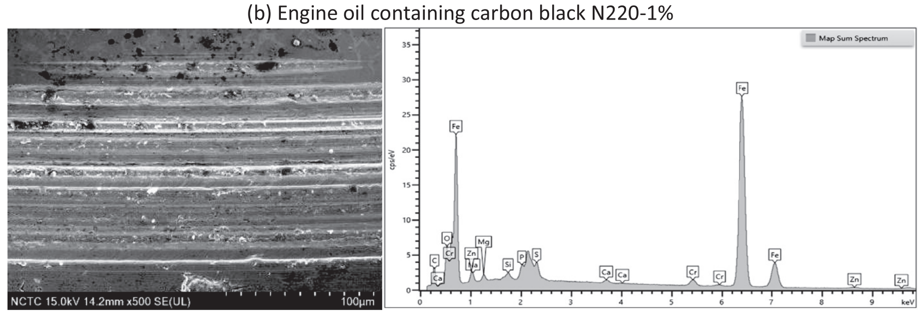
Steel ball wear scar surface	Elementally fraction (%)											
	Fe	C	O	S	Zn	Cr	P	Mg	Na	Ca	Si	Mn
Original steel ball surface	91.81	6.79	0	0	0	1.42	0	0	0	0	0	0
Pure oil without soot contamination	80.39	9.36	3.03	4.05	0	1.30	0	0.41	0.64	0.28	0.21	0.33
1% CB-N220 contamination	77.40	10.94	4.88	1.17	2.28	1.21	0.57	0.40	0.21	0.27	0.34	0.32
0.5% CB-N330 contamination	75.43	10.24	4.65	3.02	3.44	1.26	0.34	0.70	0	0.46	0.24	0.24
1% CB-N330 contamination	76.04	10.77	5.13	1.27	2.91	1.26	0.98	0.59	0.32	0.34	0.39	0
2% CB-N330 contamination	69.01	14.62	7.64	2.15	2.45	1.15	1.10	0.62	0.29	0.37	0.27	0.32
1% CB-N550 contamination	73.17	11.55	5.82	2.10	3.32	1.26	0.87	0.67	0.33	0.34	0.30	0.28
1% CB-N660 contamination	80.21	6.59	4.43	1.13	3.15	1.37	1.02	0.83	0.42	0.37	0.22	0.27



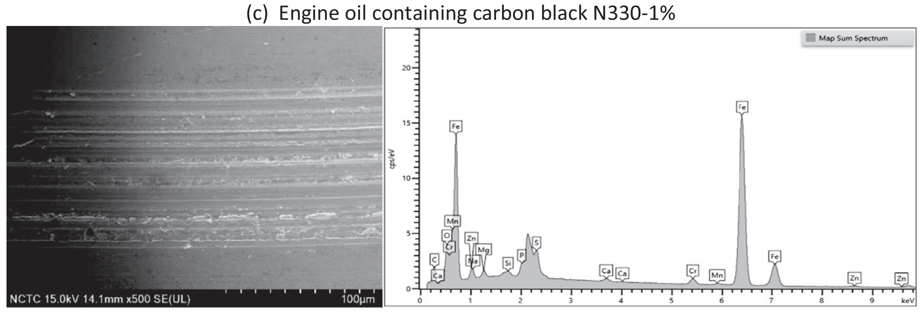
(a) Engine oil without carbon black



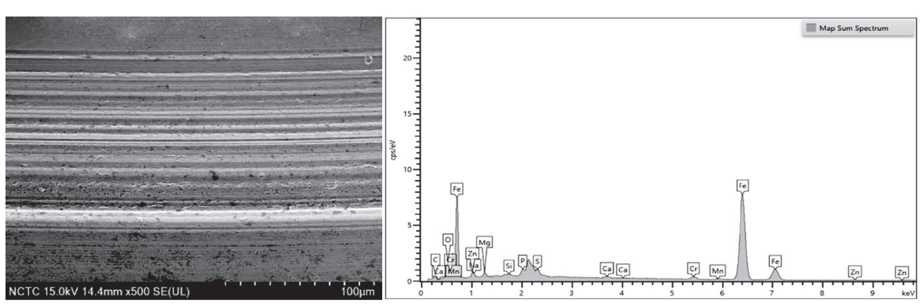
(b) Engine oil containing carbon black N220-1%



(c) Engine oil containing carbon black N330-1%

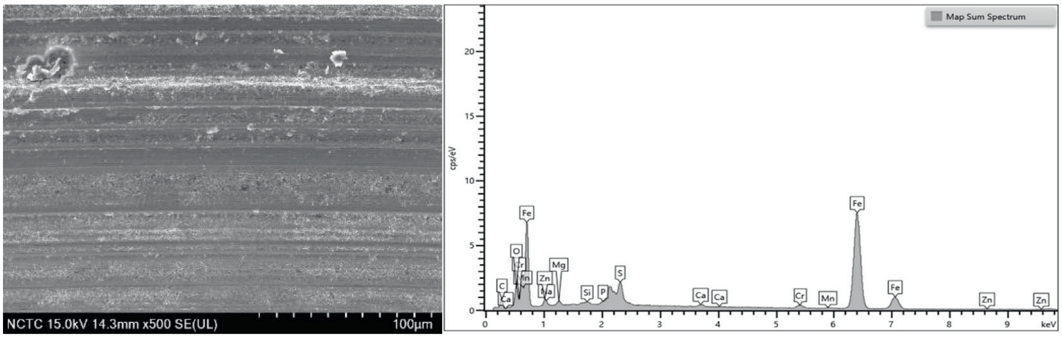


(d) Engine oil containing carbon black N550-1%

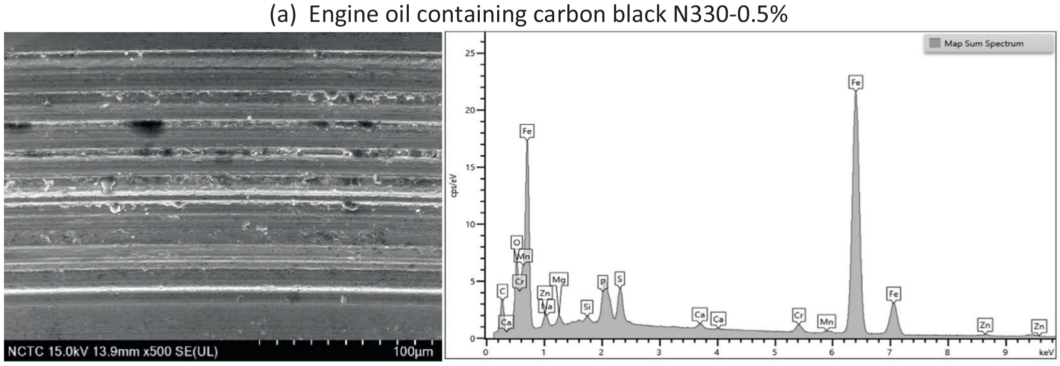


(e) Engine oil containing carbon black N660-1%

Fig. 8 – SEM micrographs and EDX analysis of the steel ball wear scar in the condition of engine oil (a) without soot, with carbon black (b) N220-1%, (c) N330-1%, (d) N550-1% and (e) N660-1% by weight.



(a) Engine oil containing carbon black N330-0.5%



(b) Engine oil containing carbon black N330-2%

Fig. 9 – SEM micrographs and EDX analysis of the steel ball wear scar in the condition of engine oil with carbon black N330 (a) 0.5% and 1% by weight.

4. Conclusion

The morphology and nanostructure of the single primary carbon black particles N220, N330, N550 and N660 were analyzed by the TEM and X-ray diffraction. It does not show much difference from soot particles emitted from diesel engine exhaust gas. The single primary particles of carbon black N220, N330, N550 and N660 have an average diameter of 24, 30, 45, 55 nm, respectively. The primary particle size of carbon black N330 is similar to the size of the soot particle from diesel engine exhaust (29–31 nm) which is smaller than the calculated oil film thickness. The average oil film thickness at 75 °C is 40 nm. Moreover, the hardness of carbon blacks and soot were estimated from the carbon density in their structure. The results show that carbon black N220, N330, N550 and N660 hardness are 14.28, 12.33, 14.57 and 9.58 GPa, respectively and soot's hardness are in range of 11.20–20.73 GPa. Their hardness is greater than the hardness of the steel ball which is 7.84 GPa.

The average wear scar diameter of steel ball tested with engine oil blended with N220, N330, N550 and N660 by 1% by weight is larger than that of pure engine oil by 26%, 38%, 41%, and 39%, respectively. The diameter of wear scar tends to increase with the size of the blended carbon black particle. Furthermore, the surface roughness of the worn surface of the steel ball tested with 1% by weight of carbon black contamination (N220, N330, N550 and N660) in engine oil is lower than that of without carbon black by 22%, 35%, 21%, and 30%, respectively. The steel ball surface may be polished by carbon black particles, which had higher hardness. Since carbon black particles can disperse throughout the engine oil and could be considered as a third body, friction was reduced during the rolling between work surfaces.

On the other hand, different quantities of carbon black N330 (0.5%, 1%, and 2% by weight) have dissimilar effects on the size of the wear scar of the steel ball. It was found that the average wear scar of steel ball tested with engine oil blended with 0.5% by weight of carbon black is lower than that of without carbon black by 9%, whereas the increasing percentage of carbon black (1% and 2% by weight) lead to the increment of wear scar diameter by 38% and 44%, respectively. The addition of approximately 0.5% by weight of carbon black in the engine oil has a significant effect on steel ball wear scar diameter and surface roughness reduction.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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