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Experimental Investigation of the Friction Modifying Effects of Graphene and C60 Fullerene Used as Nanoadditives in Engine Lubricating Oil Performed on an Oscillating Tribometer

Csaba Tóth-Nagy[1], Ádam István Szabó[1*]

[1] Department of Propulsion Technology, Audi Hungaria Faculty of Automotive Engineering, Széchenyi István University, Egyetem tér 1, H-9026 Győr, Hungary
[*] Corresponding author, e-mail: szabo.adam@ga.sze.hu

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Abstract

The present article presents the results of tribological investigations performed on an off-the-shelf engine lubricant containing nanoadditives of multilayered graphene and C60 fullerene alternately. As anthropogenic CO_2 is believed to be highly responsible for global climate change, its emission is regulated in many countries. CO_2 emissions can be significantly decreased by improving the efficiency i.e. decreasing the losses in an engine. Hence reducing frictional losses was the ultimate scope of the investigations presented in this article. The experiments were carried out on an oscillating tribometer at the Department of Internal Combustion Engines at Széchenyi István University. The experiments showed that multilayered graphene in engine lubricant did not modify the friction coefficient inevitably (-1% to +4%). Fullerene nanoparticles, however, reduced the friction by 4–8%. The optimal fullerene doping quantity that resulted in the lowest friction showed to be at around 0.14 wt%.

Keywords

nano-additive, carbon, friction, engine, lubricant

1 Introduction

The development of newly engineered engine lubricating oils is a key factor for the production of cleaner internal combustion engines. The requirements are high: lower CO_2 and exhaust emissions, lower friction, and lower fuel consumption. One of the main roles of an engine lubricant is to reduce the friction in the internal combustion engine. Lower friction in engines results in higher power output, less fuel consumption and simultaneously lower emissions. Manufacturers use numerous additives to create an optimal engine lubricant. Papers report that there are nanosized particles (metallic or nonmetallic) that can work as a lubricating oil additive in tribological systems. These nanosized additives easily dissolve in oils and they can replace some of the chemical additives of the lubricant. Reports show that nanoadditives in engine lubricants can reduce the friction and wear of the components. Researchers reported on friction modifiers for engine lubricants, such as graphenes, fullerenes (Berman et al., 2014; Rasheed et al., 2016; Tang et al., 2014; Zhang et al., 2014) and on their role in tribological systems with various results (Dai et al., 2016).

Spear et al. (2015) investigated two-dimensional graphene sheets, graphene oxide (GO) and metallic nanoadditives (Y_2O_3, WS_2, αZrP, IF-MoS_2) and reported their wear decreasing (~95%) and friction decreasing (~12%) effects. Lin et al. (2011) showed that graphene platelets used as an additive in lubricating oils slightly reduced the friction coefficient and the wear rate. But chemically modified graphene platelets in reflux reaction showed the ability of improved dispersion in oil and had significant friction and wear reducing effects. Shahnazar et al. (2016) used pin-on-disc tribometer tests to show that 0.06–5 wt% of graphene used as an additive in base oil decreased the friction coefficient by ~80%. Berman et al. (2013a, 2013b) published the high antiwear and friction decreasing properties of graphene in steel-steel contacts. Marchetto et al. (2012; 2015) used microtribological experiments to show that a graphenized SiC surface had a three to five times lower friction coefficient than an ungraphenized one. There are numerous computational methods for investigating the frictional properties of graphene nanosheets such as tight-binding atomistic simulations (Bonelli et al.,

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2009), molecular dynamics simulations (Fang et al., 2015; Kavalur et al., 2017; Xu et al., 2012; Yang et al., 2017), finite element methods (Parashar and Mertiny, 2013), complemented with atomic force microscopy (Lee et al., 2010; Zeng et al., 2017) and micro-scale scratch tests (Shin et al., 2011). Simulation models showed promising friction and wear prediction results for experimental tests. Lee et al. (2017) and Liang et al. (2016) published on the applicability of water-based graphene doped lubricants and their tribological properties and reported that graphene doped lubricants showed 81% lower friction and 61% lower wear rate. Researchers investigated the properties of graphene adsorbed metal surfaces in tribological conditions and found that surface passivation with graphene can reduce friction by 88% in surface-surface contacts (Restuccia and Righi, 2016; Wintterlin and Bocquet, 2009).

Zhao et al. (2016) reported that multilayered graphene used as an additive in lubricating oil can slightly reduce friction but it performs relatively unstable tribological properties because of prevalent agglomerates and crystal defects.

Ginzburg et al. (2002) showed that C60 fullerene used as an additive in industrial oil can form a protective antiwear layer on the contact surfaces. Jaekeun Lee et al. (2009) reported, that C60 fullerene as an additive in refrigerant oil can decrease the friction coefficient effectively at low loads (<1600 N). Kwangho Lee et al. (2009) used a disc-on-disc tribometer to show that C60 fullerene doped refrigerating oil decreased friction by 90% when compared to raw oil. They concluded that the 0.1 vol% of C60 fullerenes in lubricating oil showed the best lubrication characteristics.

Inorganic fullerenes are the focus of interest of many researchers because inorganic fullerenes – MoS_2, TiS_2, WS_2, NbS_2 – have the same molecular structure and similar tribological properties as carbon-based fullerenes. Rabaso et al. (2014) used an oscillating tribometer to investigate the friction and wear decreasing effect of inorganic fullerene used as nanoadditive in PAO (poly-alpha-olefin) oil. Rabaso et al. (2014) concluded that inorganic fullerene can reduce friction significantly and has great anti-wear properties.

Based on the review of the existing scientific literature, it was assumed and expected that the considered forms of nanosized carbon allotropes would decrease the friction in a tribological system when used as an additive in engine lubricating oil.

2 Experiments

2.1 Materials

An off-the-shelf engine lubricant was selected for the experiments as a reference and base oil (Castrol EDGE 0W-30 fully synthetic engine lubricating oil "boosted with TITANIUM FST"). The graphene platelets used for nanodoping for basis oil were A-12 (from Graphene Supermarket) graphene nanopowder with a purity of >99.5%, its average thickness is <3 nm (3–8 monolayers) and its lateral dimensions were between 2–8 µm. The C60 fullerene used in the experiments was >99.5% pure (CAS 99685-96-8) from Tokyo Chemical Industry. Fig. 1 shows scanning electron microscopic (SEM) images of the used graphene platelets and C60 fullerene.

2.2 Sample preparation

Four different mixtures were determined by a mass fraction: 0 (reference oil, did not contain carbon nanoadditives), 0.05 wt% of nanoadditives, 0.1 wt%, 0.25 wt% and 0.5 wt%, respectively. The defined amount of graphene or C60 fullerene nanoparticles was added to the engine lubricant and an ultrasonic mixer was applied for 20 minutes at 50 °C to stir a perfect dispersion of nanoparticles in the lubricant. The dispersant content of the base oil provided a stable dispersion of nanoadditives in the engine lubricant during the experiment.

Graphene doped engine lubricant formed a monodispersion because of the relatively large particle size. In contrast to graphene, the C60 molecules are relatively small in size, approximately 1 nm including the external π electron cloud. The small molecular size allowed the lubricant to contain some fullerene molecules in solution form (1-decene is the main ingredient of the base oil) as Ruoff et al. (1993) reported the rest was presumably in a monodispersed form.

The specimens under investigation were a ball and a disc. Both were polished 100Cr6 (ISO 683-17:1999, 1999), which is an example of a cam and follower material combination. Fig. 2 shows a microscopic map, a microscopic section, and the surface parameters of the polished, test-ready plate. The microscopic measures were performed with a Leica DCM3D confocal microscope.

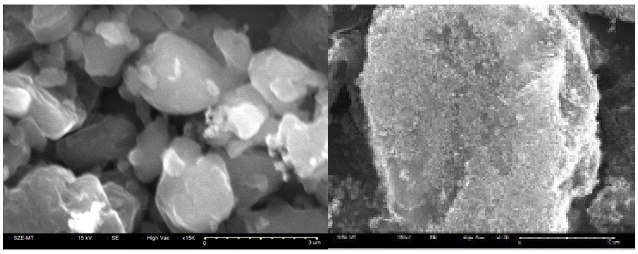
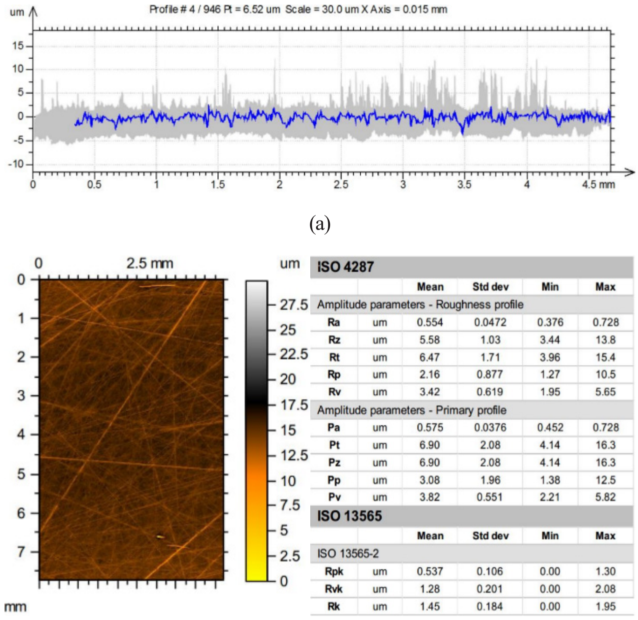

(a) (b)

Fig. 1 (a) SEM micrographs of the used graphene platelets; (b) C60 fullerenes



(a)

ISO 4287					
		Mean	Std dev	Min	Max
Amplitude parameters - Roughness profile					
Ra	um	0.554	0.0472	0.376	0.728
Rz	um	5.58	1.03	3.44	13.8
Rt	um	6.47	1.71	3.96	15.4
Rp	um	2.16	0.877	1.27	10.5
Rv	um	3.42	0.619	1.95	5.65
Amplitude parameters - Primary profile					
Pa	um	0.575	0.0376	0.452	0.728
Pt	um	6.90	2.08	4.14	16.3
Pz	um	6.90	2.08	4.14	16.3
Pp	um	3.08	1.96	1.38	12.5
Pv	um	3.82	0.551	2.21	5.82
ISO 13565					
		Mean	Std dev	Min	Max
ISO 13565-2					
Rpk	um	0.537	0.106	0.00	1.30
Rvk	um	1.28	0.201	0.00	2.08
Rk	um	1.45	0.184	0.00	1.95

(b) (c)

Fig. 2 (a) A section of the surface; (b) Microscopic map from the surface of the plate; (c) the surface parameters of the plate

2.3 Experimental equipment

The effect of lubricating oil quality is best assessed in a working engine itself. Zöldy (2021) developed a novel method to test lubricant in an engine. However, engine tests are time-consuming, costly, and result in an undesired environmental impact. However, more economical tests are available for testing the oil itself using tribometers to test the change of the lubricant effect (Tóth-Nagy and Szabó, 2022). Graphene doped engine lubricating oil was tested for its friction modifying effect in the tribology laboratories of the Department of Internal Combustion Engines at Széchenyi István University. The experiments were carried out on a linear oscillating tribometer. The test equipment for the experiments was the SRV linear oscillating tribometer by Optimol. The tribometer applied an oscillating ball pushed perpendicularly onto a plate. The normal force of the load and the relative motion between the ball and the plate cause friction and wear. The oscillating speed and stroke length of the tribometer as well as the normal load can be varied. The tribometer measures friction force with a torque sensor and load (normal force) with a force sensor. Fig. 3 shows the principle of the operation of the tribometer.

2.4 Experimental method

Before the experiments started, a drop of oil was applied to the center of the plate, and the tribometer heated up the system to 100 °C. Then the experiment started. The oscillating ball spread the lubricant evenly on the surface and lubricated the contact surfaces. The experiments were performed in two phases. The first phase of the experiment was a 30-second running-in process. The ball oscillated under a 50 N load at a frequency of 50 Hz with a stroke of 1 mm. The second phase started immediately after the first one and lasted for 2 hours. The second phase was performed at 200 N and 50 Hz at a 1 mm stroke. The whole test was performed at 100 °C, which is about the operating temperature of an engine lubricant.

The data acquisition system of the tribometer recorded data with a 1 Hz sampling time during the experiments. The computer calculated the friction coefficient from the recorded load and frictional torque parameters. The computer saved the value of the absolute integral because the frictional peak could give incorrect results. The value of the absolute integral can be calculated from the area enclosed by the curve of the friction coefficient. The absolute integral value shows an average value, while the peak value is always taken from the dead-point range where the coefficient of friction is the highest as shown in Fig. 4 The absolute integral value was used for the evaluations.

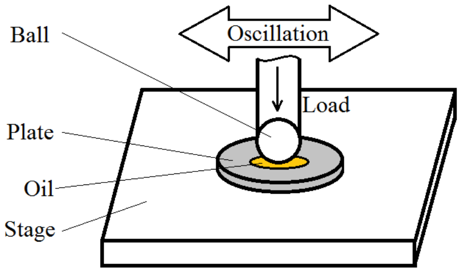


Fig. 3 Ball on plate arrangement of the SRV oscillating tribometer. The oscillating ball is pressed onto the surface of the plate. Load and friction force are measured

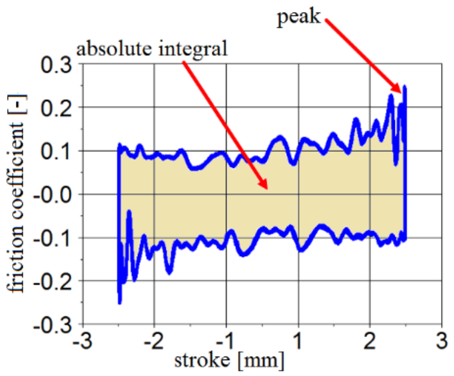


Fig. 4 The frictional peak and the absolute integral value during an experiment on the oscillating tribometer

2.5 Data analysis method

The friction coefficient and the load were recorded versus time. The friction modifying effect of carbon nanoadditives was represented by averaging all the valid test results of a particular mixture. To analyze the friction modifying effect of the carbon nanoadditives in lubricating oil, it was needed to investigate the friction coefficient at the end phase of the experiment. At this point, the specimen surfaces went through a regular running-in process. The friction coefficient slightly oscillated during the experiments, which is a normal phenomenon. Because of this phenomenon, it was necessary to take the average of the friction coefficient at the last 1 minute of the test run.

3 Results of the experiments

3.1 Results with graphene doped lubricant

Although the analysis considered the friction coefficient (average of the different test runs) of the examined lubricant as well as the average-by-time of the last 1 minute of this averaged friction coefficient, results showed similar trends, thus only the average of the last minute of the tests are presented here. The results of the experiments with graphene doped engine lubricant are presented in Fig. 5. The friction coefficient was lower only at the highest concentration (0.25 wt%). The application of the 0.05 and 0.1 wt% graphene doped lubricant slightly increased the friction coefficient. The friction coefficient showed a different behavior when 0.05 wt% graphene doped engine lubricant was applied. After the initial near-steady value of the friction coefficient, after about 2500 seconds, each curve clearly shows a decrease until the end of the experiment, except the curve of the 0.05 wt% graphene doped mixture. However, the results showed that the friction modifying effect of graphene is not obviously significant.

Multilayered graphene has a relatively large particle size as shown in Fig. 1, which may not allow the particles to enter the contact region. Furthermore, the high temperature raised the thermal activity of the graphene platelets, which presumably increased their ability to form agglomerations during the experiment. These phenomena resulted in an unstable outcome in the friction coefficient.

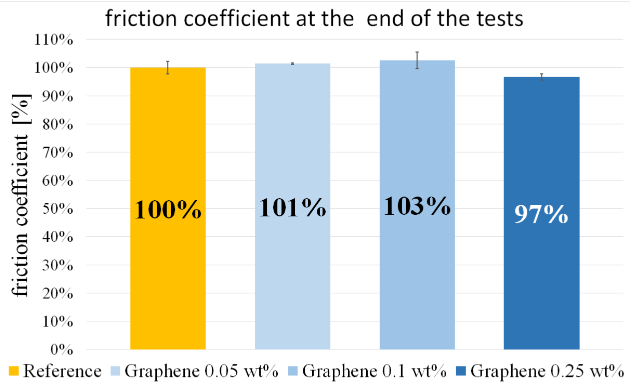


Fig. 5 The friction coefficient comparison of graphene doped oil averaged in the last 1 minute compared to the reference oil

3.2 Results with C60 fullerene doped lubricant

Fullerene showed to be adaptable for nanodoping because the relatively small size allowed the fullerene molecules to enter the contact region. Fig. 6 show the friction coefficient of fullerene doped lubricant. The friction coefficient was 4–8% lower at the end of the experiments for the doped lubricant than for the reference oil. This might be due to multiple reasons: the fullerene particles suspended in the nanooil have a spherical structure play a role as ball-bearings on the friction surfaces and allow less metal-to-metal contacts which were identified by the lower friction coefficient of the C60 doped lubricant compared to basis oil. Fullerene is believed to participate as one of the components in the tribofilm as well as a polishing agent due to its high hardness.

The optimum C60 content was determined based on the experiments. The trend-line of the average friction coefficient of each mixture of the last 1 minute at the end of the experiments showed a curve of a second degree. Fig. 7 shows the friction coefficient of the different mixtures and the trend line of the average friction coefficient of the reference and the C60 fullerene doped mixtures. The optimum point was defined from the trend line, which showed presumably the lowest friction coefficient with the used parameters to be 0.1011 at 0.142 wt% of concentration. This 0.142 wt% concentration of fullerene in engine lubricant confirms the scientific literature.

The friction coefficient increased when the mixture had a higher concentration of C60 fullerenes than 0.142 wt%. The increase in the friction coefficient is presumed to be

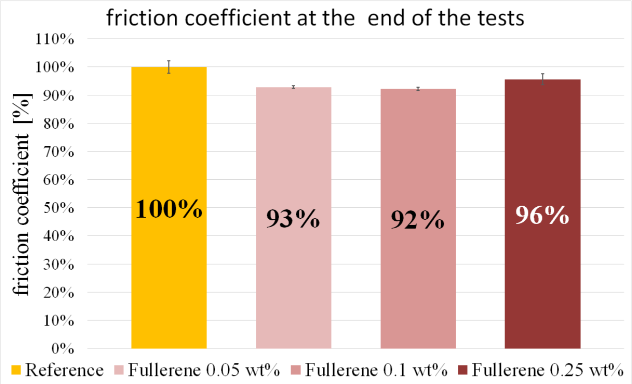


Fig. 6 Friction coefficients averaged in the last 1 minute of the experiments compared to the reference oil. All the concentrations of C60 fullerene in lubricating oil decreased the friction coefficient by 4–8%

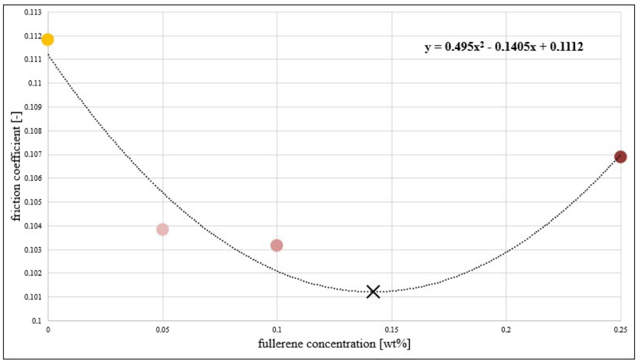


Fig. 7 Friction coefficients averaged in the last 1 minute of the experiments. The trend line shows the friction decreasing effect of the fullerene doped engine lubricant. The optimal mixture seems to be at around 0.14 wt% of fullerene content

due to the high concentration of nanoadditives in the engine lubricant that increased the viscosity of the mixture.

4 Conclusions

The current experimental study investigated the friction modifying effects of graphene platelets and C60 fullerene as nanoadditives in an off-the-shelf engine lubricating oil alternately. The experiments were carried out on an oscillating tribometer. The graphene nanosheets modified the friction coefficient by −1% to +4% when compared to the reference engine lubricant. No conclusions can be deducted regarding the friction modifying effect of graphene nanoadditives on an average scale based on the results of the present study. The possible reason can be that the graphene was claimed to be nanosized but in reality, the lateral extension of the graphene platelets applied was larger by 3 magnitudes. These large-sized particles also agglomerated and the increased particle size prevented graphene from entering into the contact region of the tribological system. Graphene seems to have no obvious trend in friction modifying effect.

C60 fullerene used as an additive in engine lubricant, however, decreased the friction coefficient by 4–8%, which is a significant decrease. Fullerene dissolved/mixed well into the lubricating oil without any other additives. The reduction in friction coefficient is presumably to be attributed to the fullerene molecules acting as rolling elements lowering the friction on the contact surfaces. Fullerene is also presumed to participate in tribofilm formation. Fullerene also acted as a polishing agent further reducing the friction. The optimal fullerene concentration from friction coefficient consideration showed to be around 0.142 wt%.

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