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# Effect of the size and morphology of particles dispersed in nano-oil on friction performance between rotating discs<sup>†</sup>

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### Abstract

Various carbon-based particles such as graphite, carbon black, graphite nanofibers and carbon nanotubes were dispersed in mineral oil to systematically examine the effect of the size and shape of particles on the properties of friction performance. As the results of friction tests using a disc-on-disc tribotester, the friction coefficient of a disc specimen was significantly reduced when nano-sized spherical particles were suspended in mineral oil. This was attributed to the presence of spherical nanoparticles, which prevented direct contact between frictional surfaces. However, the fibrous nanoparticles with high aspect ratios deteriorated the lubrication performance between friction surfaces due to a higher degree of agglomeration.

Keywords: Friction performance; Nanoparticles; Graphite; Carbon black; Graphite nanofibers; Carbon nanotubes; Nano-oil

#### 1. Introduction

Lubrication is an interdisciplinary science that involves physics, chemistry, materials, fluid mechanics and contact mechanics [1]. Numerous mechanical systems require a variety of functional lubricants to decrease the friction and wear of contacting surfaces as well as to significantly reduce the total energy consumed by mechanical systems. Many studies have focused on improving the lubrication performance of general lubricants. One approach is to incorporate particle additives into regular lubricants so that it can reduce the friction and wear of frictional surfaces. This simple approach has been applied widely in lubrication engineering and has contributed to lubrication enhancement [2].

Many studies have reported that the nanoparticle-dispersed lubricants are effective in decreasing the level of wear and friction. Various types of nanoparticles were used to prepare nano lubricants, including polymers, metals, and organic and inorganic materials [2-9]. Rico et al. [3] evaluated the extreme pressure and wear reduction properties of nano lubricants using polytetrafluoroethene (PTFE) nanoparticles in two different types of mineral oil with a kinematic viscosity of 70 mm²/s and 500 mm²/s at 40°C, respectively. The nano lubri-

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cants containing the PTFE nanoparticles provided better lubrication characteristics due to the shock-absorbing and reducing contact between the frictional surfaces. Liu et al. [2, 4] carried out the friction and wear tests using 500SN oil containing Al and Sn nanoparticles. The friction coefficient, extreme pressure, and wear rate of the specimen were measured using a pin-on-disc tribotester and a four-ball test machine. They reported that the antiwear and extreme pressure performance of nano lubricants were improved within a wide load range by the addition of Al and Sn nanoparticles. The non-seizure loads of Al- and Sn-based nano lubricants were increased from 109 kg<sub>f</sub> to 126 kg<sub>f</sub> and 114 kg<sub>f</sub>, respectively. Lee et al. [7] also examined the lubrication characteristics of fullerene-added oil by controlling the volume concentration of fullerene. They reported that the nano lubricants with higher fullerene concentrations had lower friction coefficients.

Many researchers have attributed the observed enhancement of nanoparticle-dispersed oil lubrication to a mending effect, protective film and ball bearing effect [2, 5, 8]. Liu et al. [2] suggested the mending effect to explain the lubrication mechanism. They dispersed 0.1 wt% copper nanoparticles into an SN500 base oil, and measured the friction coefficients of these nano lubricants at various temperatures ranging from 100°C to 900°C. The copper nanoparticles suspended in the lubricants agglomerated into clusters under high temperature and heavy load conditions. This agglomeration was attributed to the severe friction between contacting surfaces. During

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(a) Graphite (d=5 μm) (b) Graphite (d=450 nm) (c) Graphite (d=55 nm)







(d) Carbon Black

(e) Graphite Nanofiber

(f) Carbon Nanotube

Fig. 1. TEM images of carbon-based nanoparticles (where d is the primary size of nanoparticles employed).

friction, the clustered particles deposited and filled the grooves. On the other hand, Tao et al. [8] suggested that the nanoparticles had rolling effects (or ball bearing effects). They reported that spherical particles suspended in lubricants play the role of ball bearings (i.e., spacer) between the frictional surfaces.

This study examined the effect of the size and morphology of nanoparticles suspended in lubricating oils on the lubrication performance. Different nano lubricants were prepared by mixing raw mineral oil with various nanoparticles, including graphite, carbon black (CB), multi-walled carbon nanotubes (MWCNTs) and graphite nanofibers (GNFs). The lubrication performance of the various nano lubricants prepared in this study was evaluated using a disc-on-disc tribotester. The friction coefficient and temperature of the frictional surfaces were measured under various normal forces at a fixed rotation speed. The morphology of the frictional surfaces was examined by scanning electron microscopy (SEM).

## 2. Experimental

The experiments were divided into two parts. The first part was to analyze the tribological characteristics of the nanoparticle-dispersed lubricants (i.e., nano-oil) by measuring the friction coefficient and temperature. The second part was to examine the surface characteristics of the disc specimens tested. A disc-on-disc tribotester was used to measure the friction coefficient and temperature of frictional surfaces immersed in raw mineral oil or nano-oil.

The base lubricant used in this study was a commercial mineral oil (Supergear EP220, SK, Korea) with a specific gravity of 0.89 and a kinematic viscosity at 40°C and 100°C of ~220 mm<sup>2</sup>/s and ~19 mm<sup>2</sup>/s, respectively. Four different types of nanostructured materials, including MWCNTs, GNFs, graphite, and CB particles, were used to fabricate the nanooils with a total concentration of ~0.1 vol%.

Table 1. Summary of various nano-lubricants tested.

Sample	Particle type	Particle size	Volume fraction
RL	None	N/A	0.0 vol%
NL1	Graphite	5 μm	0.1 vol%
NL2	Graphite	450 nm	0.1 vol%
NL3	Graphite	55 nm	0.1 vol%
NL4	Carbon black	54 nm	0.1 vol%
NL5	Graphite nanofiber	d <sub>GNF</sub> : 140 nm L <sub>GNF</sub> : 25 μm	0.1 vol%
NL6	Carbon nanotubes	d <sub>CNT</sub> : 20 nm L <sub>CNT</sub> : 40 μm	0.1 vol%

In order to prepare a nano-oil with dispersion stability, al-kyl-aryl sulfonate ( $c_n u_{2n+1} \longrightarrow -so_i m$ ) was added to the mixture of nanoparticles and base lubricant at a surfactant: nanoparticles of 0.3:1 by weight, and sonicated with an ultrasonic homogenizer (SMT Company, UH-600S, 600W, 20 kHz) for 2 h. The nanoparticle surfaces were modified by the surfactant, which induced repulsive forces between nanoparticles suspended in the lubricant [3, 5]. Table 1 summarizes all the nano lubricants tested in this study.

A disc-on-disc tribotester, which is described in detail elsewhere Refs. [7, 10, 11] was constructed to evaluate the lubrication characteristics. Briefly, the tribotester consisted of an air cylinder, two load cells, a servo motor, a fixed plate and a rotating plate in the lubricant chamber. Two discs had ringshaped contacting surfaces and their contacting force was changed by a cylinder rod. The plates were made from grey cast iron (GC200, KS) without any treatment and were polished to a roughness of ~0.2 µm. The frictional surfaces of the fixed and rotating plates in the disc-on-disc tribotester were merged into the lubricating oil. During the frictional test, the nano-oil was supplied to the contacting surfaces through a gap between the frictional plates. A normal load was applied using the air cylinder system, and the speed of the rotating plate was controlled by a servo motor. The magnitude of the normal force of up to ~5,000 N was measured using a load cell installed under the air cylinder, and the friction force from a combination of the rotating motion (i.e., ~3,000 rpm) and normal load was measured using another load cell installed in the closed chamber [7].

The temperature of the lubricating oil is also an important factor in determining the lubrication characteristics. Therefore, the temperature was monitored using a thermocouple installed on the surface of the fixed plate. The lubrication tests were carried out at 35°C. Before the lubrication tests, the frictional plates had an aging time with a normal force of 100 N and a rotating speed of 500 rpm for 10 minutes. During the aging time, the temperature normally increased to  $\sim 40^{\circ}\text{C}$ . A normal force ranging from 500 N to 3,000 N was applied to the fixed plate under a fixed rotating speed of 1,000 rpm for a series of lubrication tests. The contacting surfaces of the fixed plates were observed by SEM (JEOL, JSM-6700F) to determine the

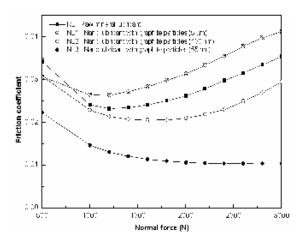


Fig. 2. Friction coefficients of the disc specimen as a function of the applied normal force at different particle sizes.

effect of the nano-oil on the roughness and wear pattern of the frictional surface.

#### 3. Results and discussion

The effect of the size of dispersed particles on the lubrication performance was examined by varying the primary size of the spherical-like graphite particles: 55 nm, 450 nm and 5 um. Fig. 2 shows the evolution of the friction coefficients as a function of the normal force and particle size of the graphite nanoparticles. The lubrication performance clearly improved with decreasing size of the particles suspended in the mineral oil. This suggests that nano-sized particles effectively play the role of ball bearings and reduce contact between frictional surfaces. However, the lubrication performance was worse than that of raw mineral oil when the average size of the suspended particles was greater than several micrometers. This was attributed to the particle size being larger than the thickness of the lubricating oil film, which is almost equal to the gap of the frictional surfaces and is strongly affected by the normal load, rotating speed and the roughness of the contacting surfaces. The thickness of the oil film for gear lubrication normally ranges from 0.5 to 5.0 µm. Therefore, micron-sized particles larger than the thickness of the oil film appear to disturb the smooth lubrication by increasing the roughness of the frictional surfaces. In addition, they accumulate at the inlet of the lubricating oil between the frictional surfaces and impede the inflow of lubricants [12].

The effect of the nanoparticle morphology on the lubrication performance was investigated. Fig. 3 shows the evolution of the friction coefficients of a disc specimen tested with variously shaped nanoparticles as a function of the normal force. The friction coefficient of the nano-oil was significantly lower than that of raw mineral oil, indicating that the nanoparticles are potential additives for enhancing lubrication performance. The friction coefficients of the CB- and graphite-added lubricants (i.e., spherical particles) were much lower than those of MWCNTs- and GNFs-added lubricants (i.e. fibrous particles).

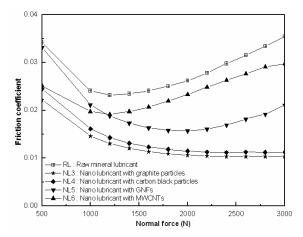


Fig. 3. Friction coefficients of the disc specimen as a function of the applied normal force at different nanoparticle shapes.

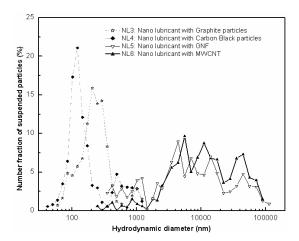


Fig. 4. Size distribution of the nanoparticles suspended in the nano-oils measured by DLS system.

This is presumably because spherical nanoparticles act more effectively as ball bearings between the frictional surfaces. In addition, spherical nanoparticles (i.e., zero-dimensional structure) have fewer contact points than fibrous nanoparticles (i.e., one-dimensional structure). Another possible reason is that the agglomeration of fibrous nanoparticles (i.e., MWCNTs and GNFs) in mineral oil must have occurred. These agglomerated fibrous particles did not appear to effectively play the role of ball bearings.

The latter can be verified by measuring the particle size distributions (PSDs) in the mineral oil. Fig. 4 shows the PSDs of various nanoparticles dispersed in mineral oil determined by dynamic light scattering (DLS). The measured PSDs using a DLS system do not correspond directly to the real nanostructures of the fibrous nanoparticles on account of their high aspect ratios. However, it can indicate the relative degree of nanoparticle agglomeration in a liquid medium [13, 14]. The average hydrodynamic sizes of spherical CB and graphite nanoparticles were 114.3 nm and 198.7 nm, which are several times larger than the primary sizes of each particle determined

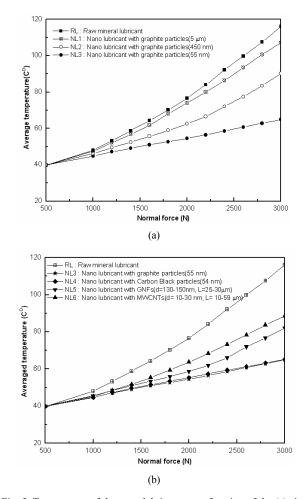


Fig. 5. Temperature of the nano lubricants as a function of the (a) size and (b) shape of nanoparticles dispersed in nano-oil.

by TEM (i.e.,  $\sim$ 50 nm), as shown in Fig. 1. This suggests that they are slightly agglomerated to some extent. However, the average hydrodynamic sizes of the fibrous MWCNT and GNF nanoparticles were determined to be  $\sim$ 5.36  $\mu$ m and  $\sim$ 10.04  $\mu$ m, respectively, indicating that these fibrous nanoparticles had agglomerated much more significantly than the spherical nanoparticles so that they were detected as micro-sized particles by the DLS system.

The temperature of the lubricants was measured by a thermocouple installed on the fixed plate in the disc-on-disc tribotester. The friction between the fixed and rotating plates increased the temperature of the lubricant, which decreased the viscosity of the lubricant. Figs. 5(a) and 5(b) show the lubricant temperature as a function of the normal force. In the case of the raw lubricant, the lubricant temperature increased with increasing applied normal force. As shown in Fig. 5(b), the average temperature of the CB and graphite nanoparticle added-lubricants was lower than that of the CNTs- and GNFs-added lubricants. This was attributed to less surface contact between the fixed and rotating plates due to the presence of spherical nanoparticle additives. The higher temperature of the agglomerated CNTs- and CNFs-added lubricants were attrib-

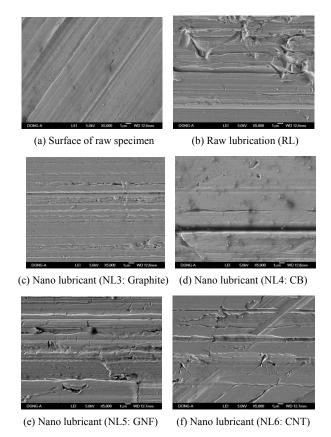


Fig. 6. SEM images of the frictional surfaces after the frictional tests with the various nano lubricants.

uted to the considerably higher contact area (several hundred times higher) formed by the 1-D structured nanotubes/nanofibers rather than the spherical particles. This is related indirectly to the temperature variation of microparticle and nanoparticle-added lubricants, as shown in Fig. 5(a). The decreased surface area of the particle-based ball bearings through the use of smaller particles reduced the level of friction so that the average final temperature of the lubricants was lower over the entire range of the normal load applied.

The frictional surface of the fixed plates was examined after a series of lubrication trials to test the hypothesis that spherical nanoparticles contribute to better lubrication performance than nanotubes, nanofibers, or microparticle additives. Fig. 6 shows the frictional surfaces after the lubrication tests with different shapes of nanoparticle additives. Fig. 6(a) shows the initial surface condition of the raw specimen. The inclined scratches with a surface roughness of  $\sim 0.2 \mu m$  were caused by the initial polishing process. Fig. 6(b) shows an SEM image of the frictional surface of the fixed plate after testing with the raw lubricant. The grooves, scars and wears formed uniformly over the entire frictional surfaces due to the severe metal contact between frictional surfaces. However, the CB and graphite nanoparticles-added lubricants maintained relatively smooth frictional surfaces with fewer scars and wear, as shown in Figs. 6(c) and 6(d), indicating that the presence of spherical nanoparticles reduced the metal contact significantly. Unlike

the spherical nanoparticle-added lubricant tests, nanotubes- or nanofibers-added lubricants produced deeper grooves and more severe scratches on the frictional surfaces, which suggest that the agglomerated fibrous nanoparticles deteriorated lubrication performance.

#### 4. Conclusions

This study compared the lubrication properties of raw mineral oil with those of nano-oils using a disc-on-disc tribotester, focusing on the influence of the size and shape of nanoparticle additives dispersed in mineral oil. The addition of nanoparticle additives to the lubricants enhanced the lubrication characteristics when compared to microparticle additives. The presence of nanoparticles between the frictional surfaces significantly reduced contact between the frictional plates by playing the effective role of ball bearings. In terms of the shape of particle additives, the nano-oils with the fibrous particle additives (e.g. CNFs/CNTs) showed higher friction coefficients than the nano-oils with spherical nanoparticle additives. This is presumably because the fibrous nanoparticles have higher aspect ratios, resulting in more contact between the frictional surfaces than spherical nanoparticles. Furthermore, the fibrous nanoparticles agglomerated more easily than the spherical nanoparticles so that the thickness of the agglomerated fibrous nanoparticles became larger than that of the lubricant film, resulting in an increase in surface roughness and inhibition of the inflow of lubricating oil at the inlet of the frictional surfaces.

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