Tribology Online
Japanese Society of Tribologists

Tribology Online, 6, 5 (2011) 247-250.
ISSN 1881-2198
DOI 10.2474/trol.6.247

Short communication

Lubricating Characteristic of Grease Composites with CNT Additive

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(Manuscript received 3 February 2011; accepted 17 June 2011; published 30 June 2011)

The friction at high speed and high pressure tends to cause the lowering of grease stickiness because the frictional heating occurs at the sliding surface, which will bring to the degradation of lubricity. In order to improve the lubricity, carbon nanotubes (CNTs), which has excellent heat conductivity, was added as an additive into the different types of greases. Two kinds of the CNTs, which were synthesized by a floating catalyst method at different annealing temperatures, were used. The sliding experiments were conducted to study the tribological performance of the grease composites by varying the added amounts of CNTs and were compared to the original greases. As a result, it was found that the CNTs improved both the friction and the wear properties. The optimum content of the CNTs in the grease composites was 0.3 mass%.

Keywords: grease, grease composite, carbon nanotube, friction, wear

1. Introduction

In current technology of additive requirement in grease of high performance and environmental friendliness, there seems to be an interest in moving away from the use of heavy metals[1)]. On this background nano-carbon allotrope such as fullerene, carbon nanohorns (CNHs) and carbon nanotubes (CNTs) has recently been paid an attention as additives in grease[2,3)]. The improvement in friction and wear for diester grease with 5 wt% fullerene were comparable to those for graphite and MoS_2. CNHs doped grease exhibited a good load carrying capacity[2)]. Its wear resistance performance was, however, less remarkable unless the CNHs are heat-treated[2)]. The CNTs exhibit extraordinary high strength and unique electrical conductive properties, and is also an efficient heat conductor[3-5)]. In our previous report[3)], when urea-based grease is mixed with both CNTs and MoS_2, it shows synergy effect in mutual improvement of lubricity, load-durability. Especially the merit of addition of CNTs in the grease is the thermal conductivity enhancement, which is 1.8 times as large as that of the grease without the CNTs additive[3)]. Recently, a key contributor of the thermal conductivity enhancement is attributed not to be Brownian motion but to be nanoparticle clustering[6)].

In order to conduct the further study and find the best combination between CNTs and greases, the present note reports the friction and the wear characteristics of different kinds of greases with and without two kinds of the CNTs.

2. Experimental

2.1. Grease composites

Three kinds of greases used in this work were commercially available, as shown in Table 1. The greases were lithium-based or complex based lithium greases having the good heat-resistance, water resistance, and mechanical stability.

For the CNTs additives, we used multi-walled carbon nanotubes; MWNT-7®, produced by HODOGAYA-KAGAKU Co. Ltd. The CNTs, synthesized by a floating catalyst method[4,5)], were further heat-treated at different temperatures of 2600 °C and 1200 °C, which were referred to CNT-A and CNT-B, respectively.

Table 1 Properties of commercially available greases used in this work

	Idemitsu Daphne Alphagel ET®	Idemitsu Daphne Eponecks SR®	Idemitsu Alvania Grease S2®
Base oil	ester	mineral	mineral
Thickener	Lithium soap	Lithium complex soap	Lithium soap
Penetration	250	370	283
Dropping point	190	300	181

Micro-Raman spectra of the two CNTs are shown in Fig. 1. Micro-Raman spectrum of the CNT-A exhibited G band at 1575 cm^{-1} due to the graphite structure, and D band at 1334 cm^{-1} due to the defect of graphite and amorphous. Here, we should note that G/D intensity ratio is convenient indicator of the graphitization[7]. The G/D value of the CNT-A was larger than that of the CNT-B, indicating that the products (CNT-A) prepared at 2600 ^{o}C for 15 min has relatively less defects in the graphite structure and thereby the higher crystallinity of the CNT-A.

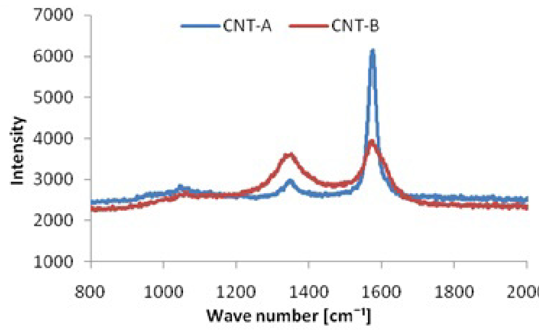

Fig. 1 Micro-Raman spectra of CNT-A and CNT-B

The FE-SEM photographs of the CNTs are shown in Fig. 2. It was observed that the two CNTs have very similar morphologies. Both CNT-A and CNT-B were about 150 nm in diameter and 10 - 20 μm in length. The density was 2.0 g/cm^3. The heat conductivity was around 1000 W/(m·K)[5].

The CNTs contents in the grease composites were varied from 0 mass% to 0.5 mass%. The mixture of the grease and the CNTs powder was put into a bowl of stainless steel, and then stirred using a hand-mixer until the mixture became homogeneous. The lubricity of these grease composites was investigated by sliding experiments.

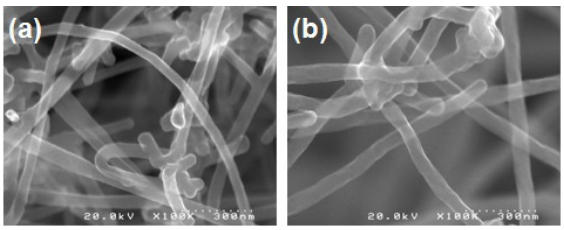

Fig. 2 FE-SEM images of (a) CNT-A and (b) CNT-B

2.2. Sliding experiment

The sliding experiment was carried out using a ball-on-disk type friction tester (FPR-2100; RHESCA Co. Ltd.) at room temperature. Both the ball and the disk were made of high carbon chrome steel of E52100, 100CrMn6 (SUJ2). The size of ball is 4.8 mm in diameter, and the size of disk is 25 mm in diameter and 5 mm in thickness. The load applied on the ball was 1.96 N or 19.6 N. Hertz pressures at 1.96 N and 19.6 N were 0.62 GPa, 1.35 GPa, respectively. The radius of sliding track was about 8 mm. The sliding speed was 0.25 m/s or 0.50 m/s. For each experiment, both the ball and the disk were pre-cleaned in ethanol using ultrasonic cleaning machine. The grease was then evenly pasted on the disk with the thickness of around 2 mm. The duration time of each sliding experiment was 1800 sec, therefore the actual sliding distance were 450 m and 900 m for the speed of 0.25 m/s and 0.5 m/s, respectively. 2 - 3 samples were used for each test, and the averaged value was reported. The wear scar on both the ball and the disk were observed by using an optical microscope. The Raman measurements were recorded on a Kaiser Optical Systems HoloLab 5000 apparatus (incident excitation: 532 nm from a Nd:YAG laser, Shimadzu Co., Ltd., Japan).

3. Results and discussion

3.1. Friction coefficient

Figure 3 shows the friction coefficient as a function of the CNTs contents in Alvania grease S2® composites at the load of 19.6 N and at the sliding speed of (a) 0.25 and (b) 0.5 m/s. Both CNT-A and CNT-B showed the similar tendency of the frictional characteristics, irrespective of the sliding speed. It was observed that the friction coefficient of the grease without CNTs was the highest value of 0.17. As the CNTs content increased from 0.1 mass% to 0.3 mass%, the friction coefficient decreased until the CNTs content was 0.3 mass%. At the CNTs content of 0.3 mass%, the friction coefficient exhibited the minimum value of 0.125. As compared to the grease without the CNTs, the friction coefficient was dramatically reduced by ca. 26%. The friction coefficient then increased with increasing the CNTs contents of 0.3 mass%. At the lower load of 1.96 N (not shown), the maximum friction coefficient was 0.102 without the CNTs, while the minimum was 0.09 with 0.3 mass% CNTs); the results showed the similar dependence of the friction coefficient against the CNTs content like as seen in Fig. 3.

Figure 4 shows a comparison of the friction test results for all greases composites. Among the grease composites tested, Alvania Grease S2® and its composites with the CNTs showed the lowest friction coefficient among other greases. In addition, other greases composites also showed the similar tendency of the friction coefficient to that of the CNTs-added Alvania Grease S2® composites; that is, the grease without CNTs showed higher friction coefficient than those of grease composites with the CNTs, whereas all grease composites with the CNTs showed the minimum friction coefficient at the 0.3 mass% CNTs content because of the increased shear deformation of the grease composite with higher CNT contents (above 0.3 mass%). Addition of either CNT-A or CNT-B at the optimum concentration of 0.3 mass% was found to be effective in reducing the mean friction coefficient.

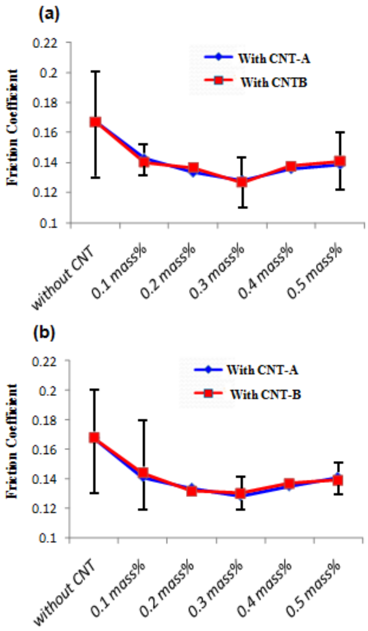

Fig. 3 Friction coefficient of Alvania Grease S2® composites with and without CNTs at the load of 19.6 N, sliding speed: (a) 0.25 m/s and (b) 0.5 m/s

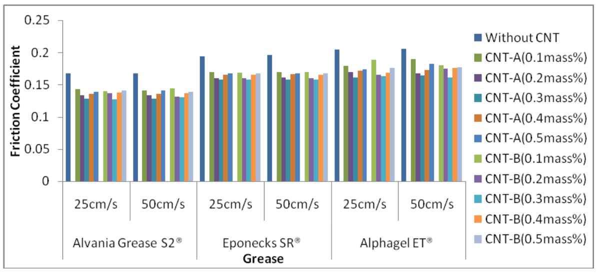

Fig. 4 Friction coefficients of three greases composites with and without CNTs at the load of 19.6 N

3.2. Wear property

Figure 5(a)-(d) shows the optical microscope images of the worn surface of the disk and the mating ball lubricated with Alvania Grease S2® composites with and without CNT-A at the load of 19.6 N. The wear damages lubricated in the grease composites with the CNT-A were mild as compared to that in the pure grease; their borderlines could be hardly recognized under the optical microscope. The wear damages of the ball lubricated with grease composite with 0.3 mass% CNT-A was milder than those of the ball lubricated with the pure grease. The wear damage of the ball lubricated with grease composite with the CNT-B also showed the similar trends to the case of grease composite with the CNT-A (not shown).

As seen in Fig. 5, the wear damage of the ball was a circular shape. The worn volume V was calculated from the radius of the wear damage. Then, the specific wear rate (W_s) of the ball was calculated by the relation of $W_s = V/(W \times L)$.

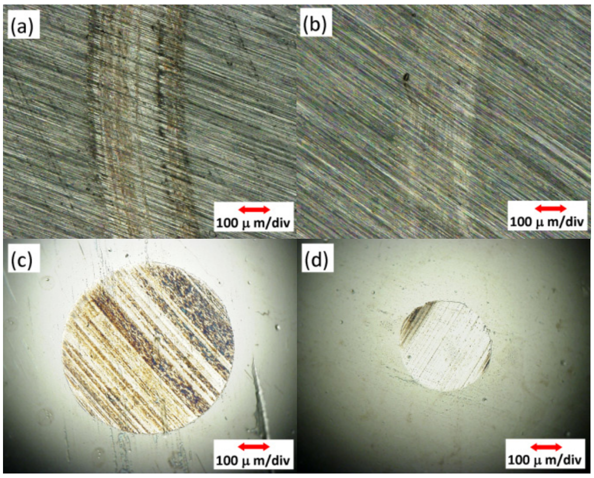

Fig. 5 Optical microscope images of (a) and (b): the wear track of the disk, (c) and (d): the mating ball in Alvania Grease S2® composites lubricated with Alvania®, respectively. (a) and (c): Alvania®, (b) and (d) Alvania® + CNT-A (0.3 mass%) at the load of 19.6 N and the sliding speed of 0.25 m/s

Figure 6(a) and (b) are the specific wear rate (W_s) of Alvania grease S2® composites with and without the CNTs at the sliding speed of 0.25 m/s and 0.5 m/s, respectively. It was found that the relationship between W_s and the CNTs contents showed the similar tendency to the friction coefficient, irrespective of the load and the sliding speed. The result indicates that at the present tests, the optimum concentration (0.3 mass%) of CNTs additive in the grease composites well operates to prevent both adhesion and adhesive wear at the real contact area without losing lubrication property of grease. The CNTs in the grease composites played a great role on W_s at higher load of 19.6 N. The decreased extent of the W_s in the grease composites with the optimum CNTs content was about one-fifth of the greases without CNTs at 1.96 N and one-tenth without CNTs at 19.6 N, respectively.

Figure 7 shows a comparison of the W_s for all greases composites at the load of 19.6 N. Eponecks SR® grease composites showed similar W_s behaviors to Alvania Grease S2® composites at sliding speed of 0.25 and 0.5 m/s, while the W_s of Alphagel ET® grease composites was higher than that of the former two.

Finally, micro-Raman spectral analyses of the worn grease composites in the disk wear scar were carried out. Figure 8 shows the typical spectra of the worn Alvania grease S2® composites with 0.3 mass% the CNT-A and CNT-B at the load of 19.6 N and the speed of 0.5 m/s. For the CNT-A, the G-band was clearly observed, while the D-band did not. Moreover, both the G and the D-bands were disappeared for the CNT-B. The results suggested that, as far as Raman spectral examination concerned, both the crystallinity of as-received CNTs and the degradation of the CNTs did not affect the friction and wear behavior during sliding experiments.

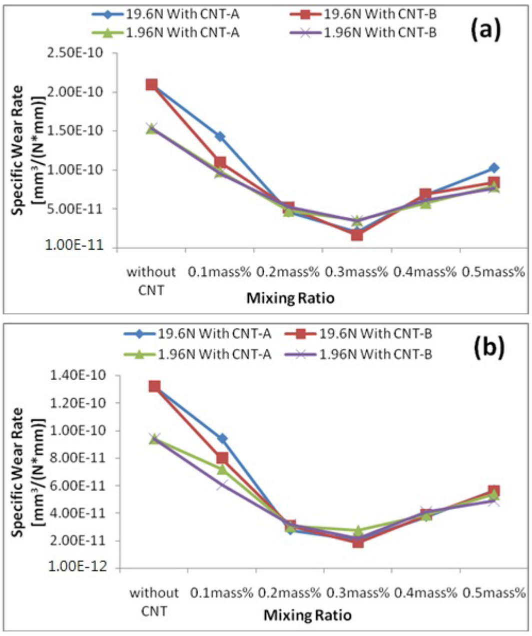

Fig. 6 Specific wear rate of Alvania grease S2® composites with and without CNTs, (a) sliding speed: 0.25 m/s, (b) sliding speed: 0.5 m/s

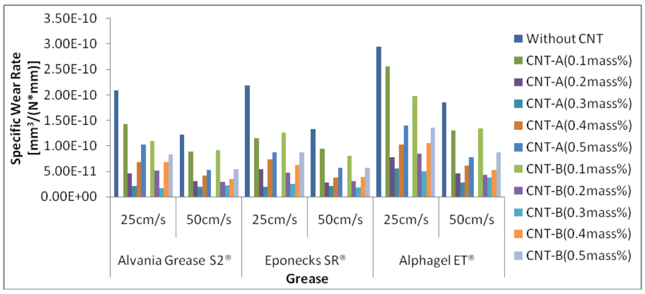

Fig. 7 Specific wear rate of all grease composites at the load of 19.6 N

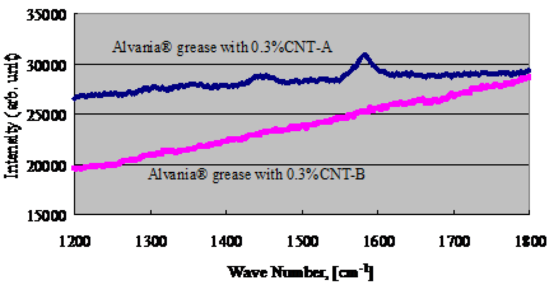

Fig. 8 Micro-Raman spectra of the worn Alvania grease S2® with 0.3 mass% CNT-A and CNT-B

4. Conclusions

This paper mainly concentrated on the possibility if the CNT, as an additive, is effective for the greases to improve their tribological performance. The results are summarized as:

1) Addition of the CNTs (both A and B) into the greases could reduce both the friction and the wear drastically.
2) The optimum amount of the CNTs additive was 0.3 mass%.
3) Among three kinds of greases tested, Alvania® Grease S2 and the grease composites showed the lowest friction and wear.
4) Both well crystallized CNT-A synthesized at higher temperature and poorly crystallized CNT-B synthesized at lower annealing temperature showed enough effectiveness in reducing both friction and wear.

5. Acknowledgements

We thank Dr. T. Tsukada, HODOGAYA CHEMICAL CO, LTD for providing CNT sample. This work was supported by CLUSTER, KAKENHI #21360074 and COE program of the Ministry of Education, Culture, Sports Science and Technology, Japan.

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