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Enhancing AW/EP property of lubricant oil by adding nano Al/Sn particles

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This paper presents results of experiments to enhance antiwear/extreme pressure (AW/EP) properties of a lubricant oil by adding metal nano particles. In this experiment, Al, Sn and Al + Sn nano-particles were selected as trial additives. The AW and EP properties were evaluated on Four-Ball test machine, while the feature and composition of the wear scar surface were investigated by scan electron microscope (SEM) and energy dispersion spectrum (EDS). The test results show that the AW and EP performance can be improved within a wide load range by adding Al + Sn nanoparticles. Analysis of the enhancement mechanism has also been conducted in this experiment and presented in this paper. It is found that nano-Sn particles can be deposited on the friction surface when the pressure was moderate and act as AW additive. It is also found that the nano-Al particles can be deposited under the condition of high load pressure and act as EP additive. Thus, the AW and EP properties of tested lubricant oil have been improved at the same time due to adding both Al and Sn.

KEY WORDS: nano-metal particles, lubricant additive, anti-wear, extreme pressure

1. Introduction

Nano-scale metal particles, which enjoy tiny size and other special physical properties, have drawn great attention in many engineering fields, including the lubricant industry. The emergence of nano-materials has provided a new approach to develop of lubricant additives. A lot of novel work has been done with enormous encouraging achievements [1–9]. Due to the difference of physical properties on nano scale, a certain kind of metal particle may function only within specific load range. In other words, a particular kind of metal particle may not be able to satisfactorily enhance AW/EP properties within a wide load range. However, simultaneous use of various kinds of nano-scale metal particles might be an effective way to overcome the shortcomings mentioned above. From this idea, nano-scaled Sn and Al particles have been selected as two candidate materials. After being characterized in morphology and dimensional size, they were added to a commercial lubricant oil both individually and as a mixture, and then were evaluated on a four-ball test machine. The test results have been analyzed with SEM and EDS to explain the enhancement mechanism of AW/EP property of the lubricant oil.

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2. Experimental

2.1. Nano-scale metal particles

The selection of metal particles was based on their physical properties, especially considering hardness and stability in open air. Preliminary trials show that Sn and Al particles are suitable for our experimental parameters. The Sn and Al particles used in these experiments are fabricated by arc plasma enhanced physical evaporation–deposition method. These particles display spherical morphology under transmission electron microscope (TEM), as shown in figure 1.

These particles are quite stable and inactive in open air. They can be heated up to about 500 °C (Al) and about 250 °C (Sn) in open air without being oxidized, as illustrated by differential thermal analysis (DTA) test in figure 2 and figure 3 displays the X-ray diffraction (XRD) patterns of Al and Sn particles with the estimation of particles size by Sherrer broadening [10]. The average diameters of the two kind of particles have been separately estimated as 53 nm (Al) and 217 nm (Sn).

2.2. Preparation of trial oil

The carrier oil used in these series of experiments is a commercial automobile engine oil SE15W/40. Al particles, Sn particles and Al + Sn (1:1, volume ratio) particles were dispersed into the carrier oil at the same percentage of 1% (vol.) by ultrasonic vibrator for 30 min at the temperature of 50 °C.

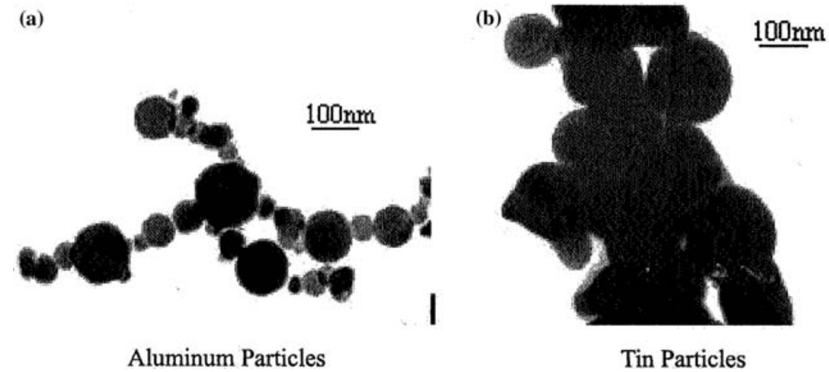

Figure 1. Nano-scale Al and Sn particles adopted in this experiment.

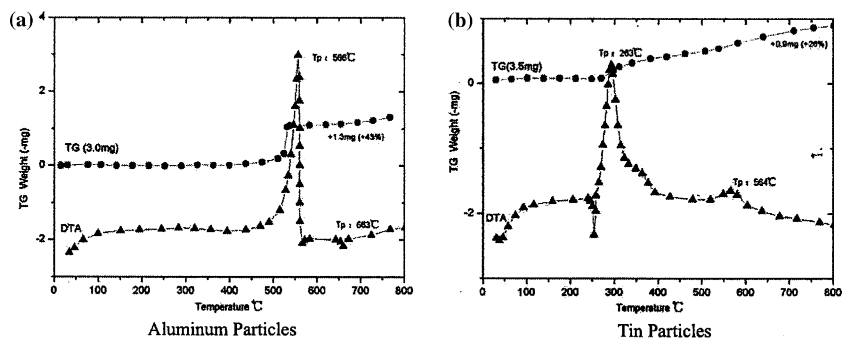

Figure 2. DTA curves reveal the thermal stability of nano-scale Al and Sn particles.

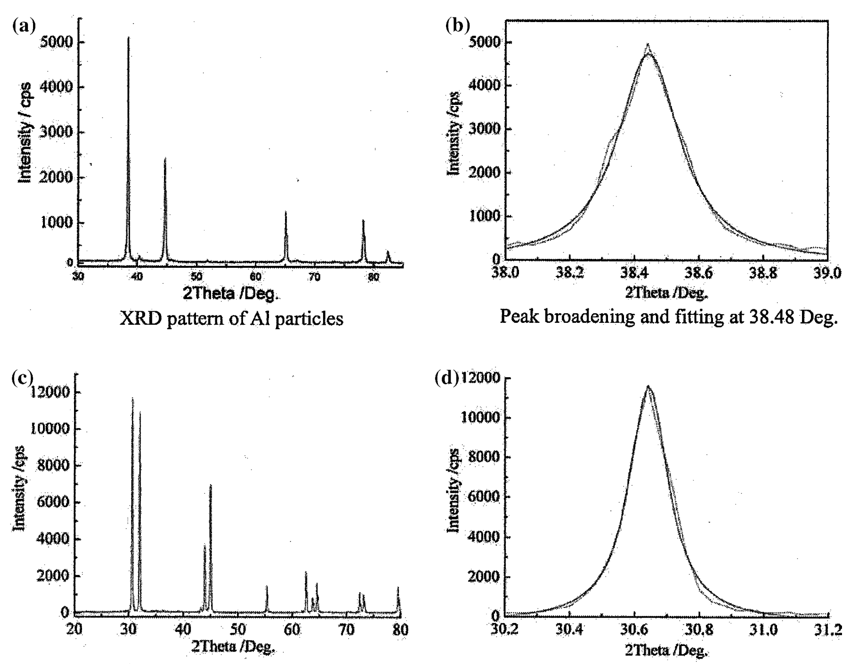

Figure 3. XRD patterns and peak broadening & fitting of Al and Sn particles.

2.3. Four-ball extreme-pressure test

The evaluation of the low wear and load-carrying properties of metal nano-particles added lubricant oil was done with four-ball method based on ASTM standard D2783. The reference oil was SE15W/40 automobile engine oil, the "blank oil" in this experiment. The three kinds of dispersed metal nano-particles were further diluted with the blank oil at volume ratio of 1:10, so the volume ratio of metal particles in each oil sample was 0.1%. The load was initially

Table 1.
Wear scar diameter of test balls.

Load /Kg Oil sample		Wear scar diameter/mm											
		45	77	95	105	109	114	119	126	130	140	160	180
SE15W/40	Min	0.31	0.39	0.41	0.42	0.43	0.44	2.20	2.50	2.54	2.70	2.88	3.20
	Mean	0.32	0.40	0.42	0.44	0.46	1.16	2.25	2.60	2.64	2.73	2.97	3.20
	Max	0.33	0.41	0.43	0.45	0.47	2.40	2.30	2.70	2.75	2.80	3.16	3.20
SE15W/40 + 1‰Sn	Min	0.25	0.34	0.36	0.39	0.40	0.40	1.95	2.50	2.50	2.74	3.16	3.10
	Mean	0.28	0.35	0.38	0.40	0.42	0.44	2.05	2.52	2.53	2.90	3.04	3.23
	Max	0.30	0.36	0.40	0.41	0.44	0.62	2.15	2.55	2.60	2.97	3.16	3.40
SE15W/40 + 1‰Al	Min	0.30	0.36	0.39	0.40	0.41	0.41	0.45	0.45	2.30	2.53	2.60	2.87
	Mean	0.31	0.38	0.40	0.41	0.42	0.43	0.46	0.47	2.40	2.60	2.68	2.96
	Max	0.33	0.40	0.42	0.42	0.43	0.44	0.47	0.48	2.50	2.68	2.77	3.10
SE15W/40 + 1‰Al/Sn	Min	0.25	0.34	0.39	0.40	0.43	0.43	0.43	0.45	2.10	2.30	2.35	2.75
	Mean	0.27	0.36	0.40	0.42	0.44	0.45	0.45	0.46	2.13	2.33	2.51	2.90
	Max	0.30	0.38	0.42	0.45	0.45	0.46	0.46	0.47	2.20	2.40	2.68	3.08

The Loads corresponding to cells with grey background denote the PB value of relevant oil samples.

applied at 45 Kg and gradually increased to 180 Kg. The rotating speed applied was 1450 rpm, and the time duration for each test was 10 s. The material of the test balls was GCr15A, with diameter of 12.7 mm and hardness of HRC 64–66. After each test, the diameter of wear scar was measured as set forth below in table 1. Figure 4 illustrates the wear scar diameter versus applied load of each trial oil. The maximum non-seizure load (PB) of each oil sample as listed in table 2 was derived from the relationship between wear scar diameter and applied load.

3. Results and discussions

3.1. *AW and EP performance of oil samples with metal nano-particles*

Comparing the two curves in figure 4(a), it can be seen that the wear scar diameters have been reduced in the tests with Sn particles as long as the load is no more than 114 kg. The average reduction of scar diameter is 10.5% and even reaches 12.5% in the case where the load is smaller than 77 kg. While considering the data in table 1, it can be seen in figure 4(a)

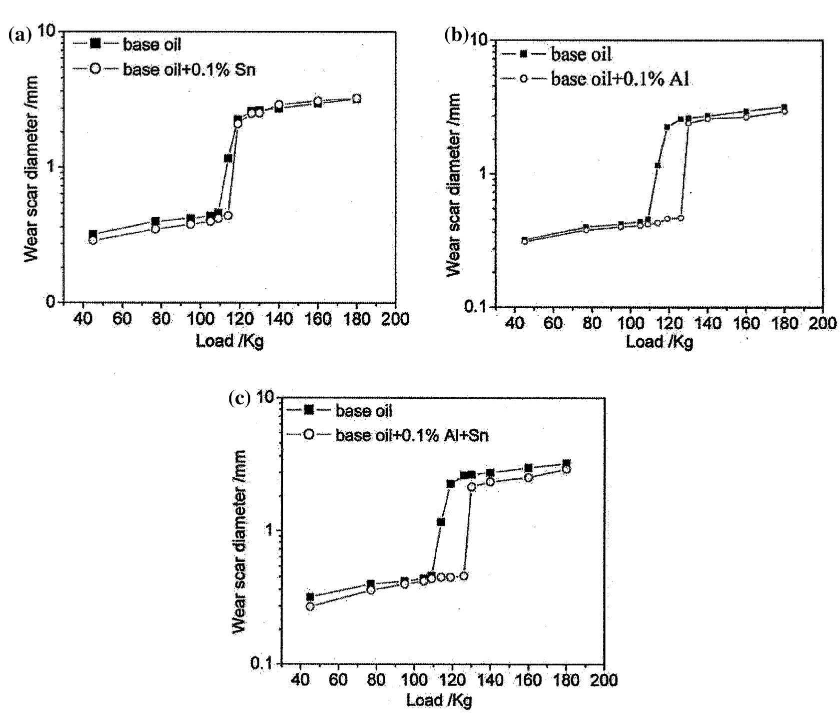

Figure 4. Comparison of wear scar diameter between metal nano-particles added oils and blank oil.

Table 2.
Non-seizure Load of oil samples (PB Value).

Oil sample	Blank SE15W/40	SE15W/40 + 1 Sn	SE15W/40 + 1 −AI	SE15W/40 + 1.A1/Sn
P_B/Kg	109	114	126	126?

that the PB value has been increased 5 kg from 109 to 114 kg, but the increase is not as much as had been expected. When the load increases to 126 Kg and more, the curve of the Sn particles enriched oil merges into that of the blank oil, which means the Sn particles have little effect on the EP property of the tested oil.

The trial result of the oil with Al particles is displayed in figure 4(b). For the condition in which the load is smaller than 114 Kg the AW property is barely improved, as the average reduction of scar diameter is merely 4% or so. But when the load is increased to 114 Kg and more, the EP capability of Al particles is observed. From figure 4(b), it is quite easy to see that the PB value has been increased 15 Kg from 109 to 126 Kg. After the load has been increased to 130 Kg and above, the average reduction of wear scar diameter has reached 8.6%, more than that (4%) in the low load range. That is to say, Al particles tend to have a largest effect in the EP load range.

As for the oil with both Al and Sn particles added, the combination has provided a significant performance enhancement. Besides the PB Value that has been enhanced from 109 to 126 Kg, which can easily be seen in figure 4(c), in the load range from 45 to 109 Kg, the diameter of the wear scars has been reduced on average 8.7%. In the load range from 126 to 180 Kg, the diameter of the wear scars has been reduced around 15%. So, the AW and EP properties have been enhanced at the same time in this sample oil.

3.2. SEM and EDS analysis

To get more understanding, the wear scar surface of the test balls lubricated with blank SE15W/40 oil were observed and analyzed with SEM and EDS. The micrographs of the wear scars with loads of 77 and 126 Kg were selected and respectively shown in figure 5(a) and (b). Due to the difference of the load applied, the scratch shown in figure 5(a) is very slight in several microns, while several hundreds of micron scratches could been seen in figure 5(b).

Figure 5(c) presents the SEM micrograph of wear scar surface of the test balls lubricated by the Sn enriched oil with the load of 77 Kg. Comparing figure 5(c) with figure 5(a), it can be seen that the wear scratch have been reduced quite a lot. The wear scar surface is quite flat and the scratching grooves have been turned into minor pits. The element mapping in figure 5(g) tells that deposited Sn particles fully filled wear scar area. The melting point of Sn is only 232 °C, with a Brinell hardness 51 MPa, so particles can be easily deformed and crashed, even melted at the contact points, and so fill the tiny grooves of the scar surface and partially prevent the scar surface from further severe wearing. While considering Sn particles which mostly reduce wear in the low load range (see figure 4(a), they may be utilized to promote the AW property of the lubricant oil.

The image in figure 5(d) reveals what happened at the wear scar surface after Al particles were added into the oil. When the applied load goes up to 126 Kg, the micrograph shows the wear scar has been improved significantly. Comparing with figure 5(b), there is nearly no scoring and instead only flat and shallow grooves can be seen; In figure 5(h), Al element has almost covered the entire tested area of the wear scar surface, which means there are lots of Al particles deposited on the wear scar surface. Al also belongs to the soft metal category, and its Brinell hardness is 245 MPa. So the Al particles behave in the same way as Sn particles do to reduce the wear scar diameter and promote the PB value of the lubricant oil (see figure 4(b)). Since Al is harder than Sn (245 MPa is >51 MPa), Al particles can hold much more load as an EP additive before they become crushed, which can explain why Al particles enhance more PB value and reduce scar diameter mostly in the high load range instead of in the low load range.

Figure 5(e) and (f) were taken from samples tested with Al/Sn particles respectively under load 77 and 126 Kg. While referring to figure 5(a) and (b), it can be easily seen that in both cases there are fairly good wear scar surfaces, only mild scratches, no serious wear. That means the combination of Al and Sn particles can provide an additive improvement. The AW enhancement is mainly provided by Sn particles, and the EP enhancement is definitely due to the contribution from Al particles.

Further measurements conducted by EDS as illustrated in figure 6 disclose more evidence for the enhancement mechanism of tribological properties. From Curve #1, when the applied load is less than that corresponding to point "A" (about 109 Kg), the content of Sn on the scar surface increases along with the increase in load, but it decreases after point "A", even though the load keeps increasing. This phenomenon can be explained as follows. At the contact point of the wear scar, there exists two

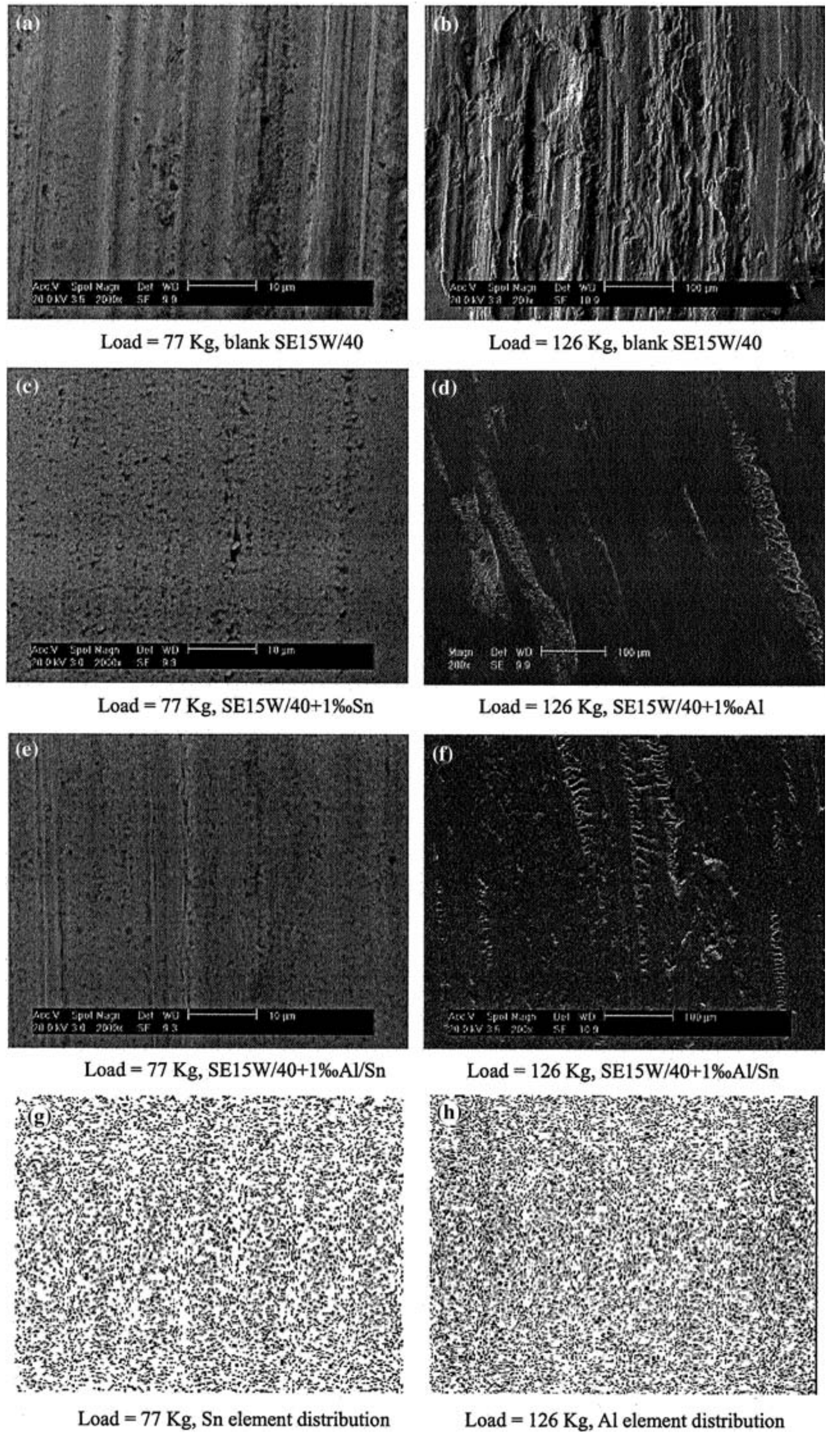

Figure 5. SEM images and element distribution of the wear scar surface.

opposite processes related to the rubbing of the metal particles friction-deposition, and friction-wipe off, both of which are decided by the pressure and wear at the contact point. Low load generates moderate pressure and less wear. So, as long as the load does not overtake the PB value, the concentration of the deposited Sn particles will be thicker than those that wiped off, and the higher the load is, the greater is the concentration of deposited particles are. From table 1, it can be easily found that the load (109 Kg) at point "A" is the PB value of blank oil. When the load rises above the PB point, the oil film will be broken and severe wear will occur, and thus cause more Sn particles to be wiped off of the surface than deposited. This tendency of curve #1 in figure 6 matches quite well with the relationship

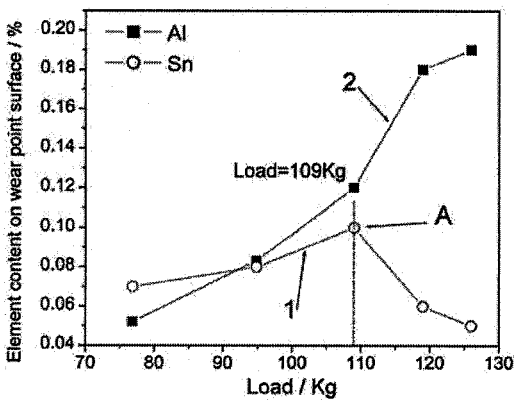

Figure 6. Elements content on wear scar surface vs. load.

between wear scar diameter and applied load when Sn particles are added, as shown in figure 4(a).

From Curve #2 of figure 6 when the load is less than that corresponding to point "A", the Al content increases along with the increase of the load, but the rate of increase is greater than that of Sn particles. The difference may due to Al's higher hardness. After point "A", the Al content continues to increase, even at a faster rate. This phenomenon can be explained as follows. When the load exceeds the PB point of the blank oil, serious wear and extreme pressure may destroy the alumina shell covering each Al particle, which is unable to be accomplished using the lower load. This will crush more Al particles and squeeze the particles into the tiny surface grooves. The tendency of increasing Al content displayed by curve #2 is consistent with the tribological behavior of the lubricant oil with Al particles presented in figure 4(b).

4. Conclusions

The following conclusions can be drawn from the experiments and analysis described above:

(1) The combination of nano-scale Al and Sn particles can obviously enhance the AW and EP properties of the lubricant oil. When the load is small (F $<$ 114 Kg), the nano-scale Sn particles play the role of AW additive; while when the force is large (F $>$ 114 Kg), the nano-scale Al particles act as an EP additive.

(2) It can be inferred that the nano-scale particles have been deposited on the surface of the wear scar and filled into the tiny grooves, whereafter these particles act as a barrier, separating the friction surface pair, reducing the wear, and subsequently postponing the occurrence of seizure.

References

[1] Y.Q. Xia and S.R. Kim, Lubricant. Sealing 3 (1999) 33.
[2] J.E. Mack and P.K. Mack, Lubricant additive, US Patent, No. 4204968.
[3] E.E. Strumban, Ultrafine powder lubricant, US Patent, No. 5S23006.
[4] Y.Q. Xia, J.Y. Ding and X.G. Ma, Lubricant Oil 13(6) (1998) 137.
[5] C.A. Melendre, A. Narayansamy and V.A. Maromi, J. Mater. Res. 4(5) (1989) 1246.
[6] F. He, Z.Y. Zhang and Y.F. Xiao, Lubricant. Sealing 5 (1997) 65.
[7] T. Hisakado, T. Tsukizoe and R. Yoshikawa, J. Lubr. Technol. Trans. ASME 105(2) (1983) 245.
[8] B.X. Jiang, B.S. Chen and J.X. Dong, Lubricant. Sealing 2 (1999) 50.
[9] Z.W. Ou, B.S. Xu *et al.*, Mater. Res. Guid. 14(8) (2000) 28.
[10] B.D. Cullity, *Elements of X-ray Diffraction*, 2nd edn. (Addison-Wesley, Reading, Massachusetts, 1978) p. 102.