

# Tribological performance of nanometer samarium borate<sup>①</sup>

HUANG Wei-jiu(黄伟九)<sup>1</sup>, WANG Jiu(王九)<sup>2</sup>, WANG Ying-fang(王应芳)<sup>1</sup>,  
LI Wei-li(李慰立)<sup>1</sup>, CHEN Bo-shui(陈波水)<sup>2</sup>

(1. Department of Mechanical Engineering, Chongqing Institute of Technology,  
Chongqing 400050, P. R. China;

2. Department of Petroleum Applied Engineering, Logistic Engineering College,  
Chongqing 400016, P. R. China)

**[Abstract]** Nanometer crystal samarium borate with a particle size of 20~ 40 nm was prepared using replacing solvent drying technique. The wear resistance and load-carrying capacity of 500SN base oil could be improved and the friction coefficient could be decreased by the addition of nanometer samarium borate. But the dosage of samarium borate nanoparticles had to be controlled at a relatively low level, a higher concentration of nanoparticles was not of beneficial to the tribological performance of the oil. The optimal dosage of nanometer samarium borate is 1.0%. Tribochemical reactions took place in the tribological process, which resulted in the formation of deposition products including diboron trioxide and disamarium trioxide. Fe<sub>2</sub>B and FeB were also found on the wear scar. The improvement of tribological properties of the oil comes from the formation of deposition layer and permeating layer.

**[Key words]** nanometer particle; samarium borate; tribology; action mechanism

**[CLC number]** TH 117.2

**[Document code]** A

## 1 INTRODUCTION

It is well known that solid lubricants, such as MoS<sub>2</sub> or graphite, possess good tribological properties. A number of researchers have added them to lubricating oils in order to improve the tribological performance of oils, but the results have not always been satisfactory. Some results indicated that it was helpful to reduce the wear and friction between the rubbing surface by dispersing the powder in oil<sup>[1, 2]</sup>. However, some negative results, such as an increased wear rate and lubricant starvation, have also been reported<sup>[3, 4]</sup>. It was concluded that the effectiveness was seriously dependent on the particle size. Recently, much attention has been paid to the preparation and applications of nanometer particles. Investigations on using nanometer-sized particles (for example, C<sub>60</sub>, CeF<sub>3</sub>, graphite, and diamond) as additives for lubricating oils have been carried out, and the results indicated that it was beneficial to improve the tribological properties of oils by dispersing nanoparticles in oils<sup>[5~ 10]</sup>.

The present paper aims at preparing nanometer samarium borate using the replacing solvent drying technique<sup>[11]</sup> and studying its tribological properties as a lubricating oil additive.

## 2 EXPERIMENTAL

### 2.1 Preparation of nanometer samarium borate

The disamarium trioxide was dissolved by chlorhydric acid, and was made up to 1 mol/L samarium chloride solution, then 40 mL 1 mol/L samarium chloride was added to 60 mL 1 mol/L borax aqueous solution with stirring. After filtering, the precipitate was washed five times with 50 mL distilled water. The precipitate and 200 mL *n*-butanol were added in the flask and distilled. The cooled water was separated from the *n*-butanol in the oil-water separation tube and *n*-butanol was returned in the flask. Stopped the distillation until that the boiling point of the solution or the volumes of the water in oil-water separation tube stopped increasing. The *n*-butanol was then removed by distillation, and nanometer samarium borate was gained after drying.

### 2.2 Measurement of tribological properties

Nanometer samarium borate and dispersing agent sorbitol monostearate were added in 500SN base oil with heating and stirring. The load carrying capacities and antiwear properties were evaluated on MQ-800A four-ball tribotester at 1 450 r/min, room temperature. The maximum non-seizure load was determined according to the national standard method GB3142-82. The wear scar diameter of the steel ball after test duration of 30 min was also measured. The balls (diameter of 12.7 mm) in the tests were made of GCr15 bearing steel with HRC of 59 to 61. Friction coefficient of oils were measured under a constant load of 300N using HQ-1 ring-on-block tribotester,

① **[Foundation item]** Project (59875083) supported by the National Natural Science Foundation of China

**[Received date]** 2001- 01- 10; **[Accepted date]** 2000- 03- 24

where the ring was quenched CrWMn steel ring (0.9% ~ 1.2% Cr, 0.9% ~ 1.05% C, 1.2% ~ 1.6% W, 0.8% ~ 1.1% Mn, 0.15% ~ 0.35% Si) of diameter 49.24 mm, height 12.7 mm, hardness 62 (HRC) and a surface roughness of  $R_a = 0.27 \mu\text{m}$ , which was rotating against 45 steel block (12 mm × 6 mm × 4 mm) with a hardness of 44.8 (HRC) and a surface roughness of  $R_a = 0.35 \mu\text{m}$ . The rotating speed of ring was 1500 r/min.

### 2.3 Characterization

Morphology and size of samarium borate were measured using a JEM-2000 EX transmission electron microscope, XRD spectra of the nanometer particle was measured with a Philips Xpert-MPD X-ray diffractometer. X-ray photoelectron spectroscopy (XPS) was conducted with a PHI-1600 X-ray photoelectron spectrometer. The upper ball used for XPS analysis was washed ultrasonically with petroleum ether and dried after the four-ball test. The  $\text{MgK}\alpha$  radiation was used as the excitation source at pass energy of 50 eV. The binding energy of C1s (284.6 eV) was used as the reference.

## 3 RESULTS AND DISCUSSION

### 3.1 Characterization of nanometer samarium borate

The morphology of samarium borate particles prepared with the replacing solvent drying technique is shown in Fig. 1. The size of the particles can be seen to be of about 20~50 nm. No marked coagulation is found. XRD spectrum of the particle is given in Fig. 2. Some sharp peaks are shown, which indicates that the particles are microcrystals.



Fig. 1 Morphology of nanometer samarium borate particle Magnification

### 3.2 Effect of nanometer samarium borate on maximum non-seized load of oil

The effect of the concentration of samarium borate nanoparticles on maximum non-seized load ( $F_B$ ) was studied, and the results are shown in Fig. 3. It is found that samarium borate nanoparticles exhibit

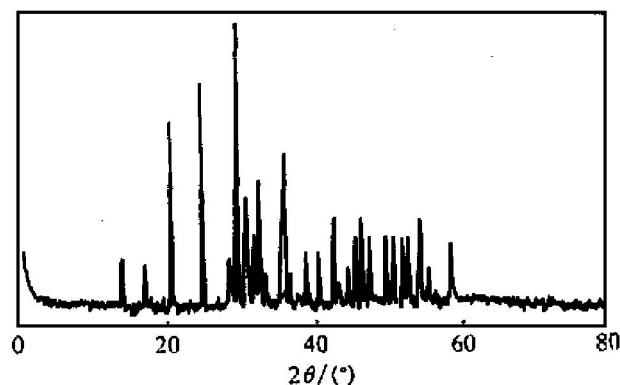


Fig. 2 XRD spectra of nanometer samarium borate particle

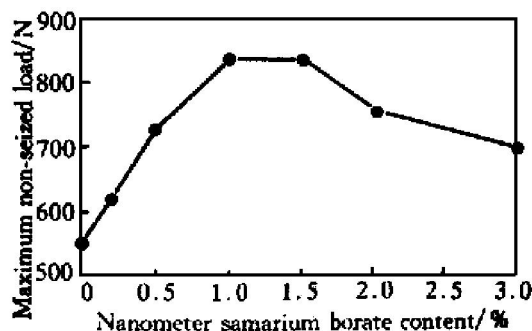
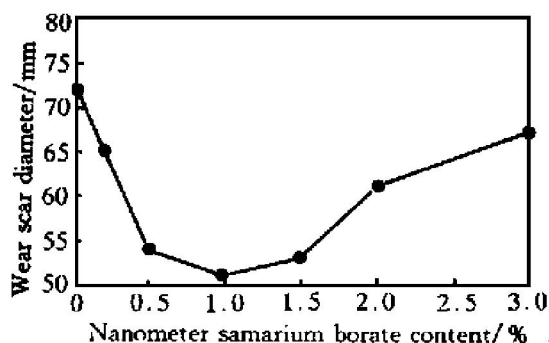


Fig. 3 Effect of concentration of samarium borate nanoparticles on maximum non-seized load

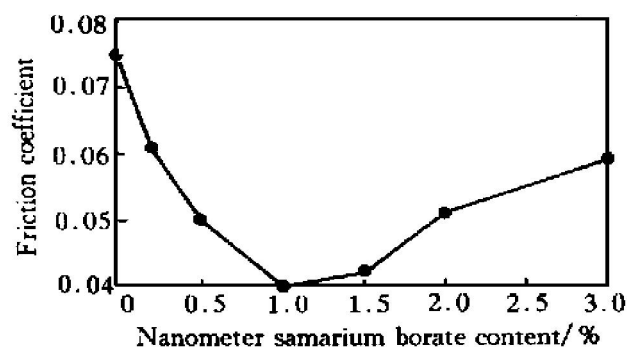
good load-carrying capacity. The  $F_B$  value of base oil is 549N and is not changed by the addition of 1.0% dispersing agent sorbitol monostearate. With the concentration in the range 0~1.5%, the  $F_B$  value increases with the concentration of samarium borate nanoparticles, and  $F_B$  value goes from 549N (base oil) to 834N. However, when the concentration of samarium borate nanoparticles is above 1.5%, the  $F_B$  value reduces sharply. These results indicate that the load-carrying capacity of the oil could be improved effectively by dispersing samarium borate nanoparticles in the oil, with the dosage of samarium borate nanoparticles being in a suit range.

### 3.3 Effect of nanometer samarium borate on anti-wear and friction-reducing behaviour

The effects of the concentration of samarium borate nanoparticles on wear scar diameter and friction coefficient are shown in Figs. 4 and 5, respectively. It can be seen that the wear scar diameter and friction coefficient could be reduced by dispersing samarium borate nanoparticles in oil. When the concentration of samarium borate nanoparticles is of about 1.0%, the smallest wear scar diameter and friction coefficients are obtained. When the concentration of samarium borate nanoparticles is increased, the wear scar diameter and the friction coefficient gradually increases. These results reveal that a higher dosage of samarium borate nanoparticles is disadvantageous in terms of the



**Fig. 4** Effect of concentration of samarium borate nanoparticles on wear scar diameter at load of 392 N and time of 30 min



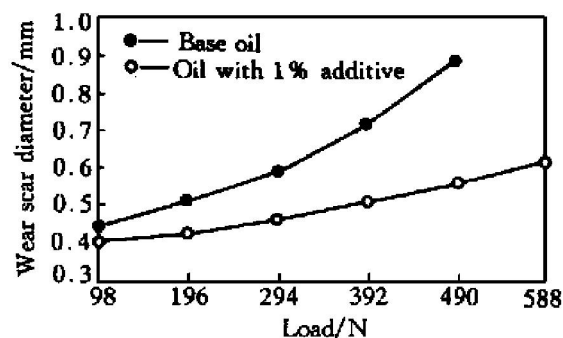
**Fig. 5** Effect of concentration of samarium borate nanoparticles on friction coefficient at load of 300 N and time of 30 min

antiwear and friction-reducing behaviour of oils.

As discussed above, it can be reasonably said that samarium borate nanoparticles possess good tribological performance, but the dosage of these nanoparticles in oil should be strictly controlled at a relatively low level (1.0% ~ 1.5%). Any higher dosage of nanoparticles worsens the tribological properties of the oil, including load-carrying capacity, antiwear and friction-reducing properties. The reason may be that nanoparticles have very high surface energy, and a strong tendency to aggregate into larger particles. At higher concentration, the probability of coalescence of the nanoparticles is also increased, and larger particles could be yielded easily in the testing process. These larger particles could act as abrasives to increase wear and friction on the rubbing surfaces.

### 3.4 Effect of load on antiwear property of oil containing samarium borate nanoparticles

The AW properties of the samarium borate nanoparticles in base oil as the function of load are shown in Fig. 6. The results indicate that the wear scar diameter of base oil increases significantly as the load increasing, but the wear scar diameter of oil containing 1.0% samarium borate nanoparticles increases tardily under different applied loads, which imply that the antiwear property of base oil is greatly improved by the addition of samarium borate

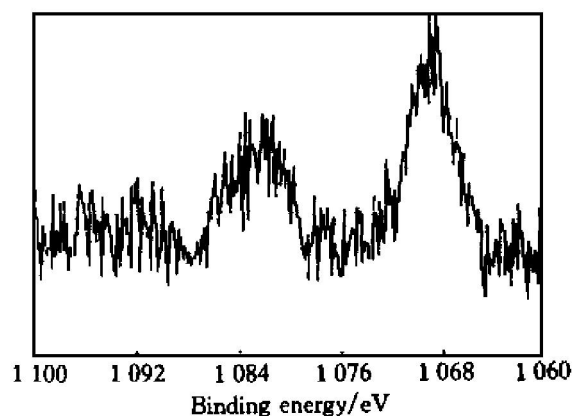


**Fig. 6** Effect of load on wear scar diameter at time of 30 min

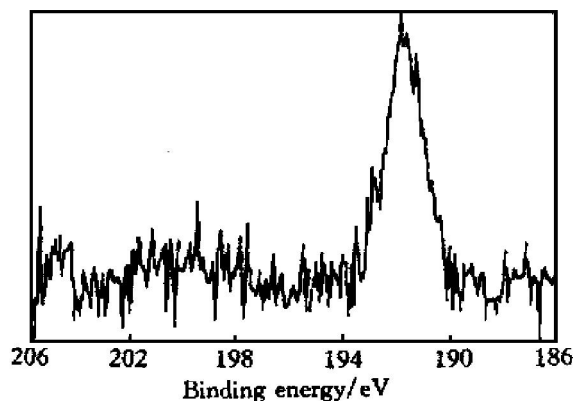
nanoparticles.

### 3.5 Discussion of antiwear mechanism of samarium borate

After a four-ball test at 588N for 30 min in the oil with 1.0% nanometer samarium borate, element composition of the wear scar was analyzed using XPS. Oxygen, boron, samarium, carbon and ferrous were detected. Fig. 7 shows that the binding energy of Sm 3d on the rubbing surface is evident at about 1 083.2 eV. the standard binding energy of Sm 3d in  $\text{Sm}_2\text{O}_3$  is 1 083.4 eV, respectively<sup>[12]</sup>, which implies that some samarium borate may be decomposed to disamarium trioxide during tribotesting. XPS spectrum of the boron is shown in Fig. 8. The binding energies indicate that boron exists mainly in the form of diboron trioxide (192 eV),  $\text{Fe}_2\text{B}$  (188.4 eV) and  $\text{FeB}$  (187.9 eV). An antiwear mechanism of the additive, therefore, can be deduced from all the above characterizations. First of all, the nanoparticles fill into the valley of the roughness peak on the rubbing surface, which may act as tiny ball bearings and provide an easily sliding layer. Then the samarium borate may further decompose to give diboron trioxide and disamarium trioxide. A further tribochemical reaction between the diboron trioxide and substrate ferrous may produce  $\text{Fe}_2\text{B}$  and  $\text{FeB}$ . It is the deposition layer containing the nanoparticles, diboron trioxide and disamarium trioxide that provides the oil with its



**Fig. 7** XPS spectra of samarium on rubbed surface



**Fig. 8** XPS spectra of boron on rubbed surface

excellent antiwear and friction-reducing properties because of low shear forces. And the permeating layer containing  $\text{Fe}_2\text{B}$  and  $\text{FeB}$  gives the rubbing surface higher load-carrying capacity probably due to a higher hardness<sup>[13]</sup>. The cooperative action between the deposition layer and permeating layer may play an important role in improving the tribological properties of the lubrication oil.

#### 4 CONCLUSIONS

Nanometer crystal samarium borate with a particle size of 20~40 nm was prepared using replacing solvent drying technique. The wear resistance and load-carrying capacity of 500SN base oil can be improved and the friction coefficient can be decreased by the addition of nanometer samarium borate. But the dosage of samarium borate nanoparticles has to be controlled at a relatively low level, a higher concentration of nanoparticles is not of beneficial to the tribological behavior of the oil. The optimal dosage of nanometer samarium borate is 1.0%. Tribochemical reactions take place in the tribological test, which results in the formation of deposition products including diboron trioxide and disamarium trioxide.  $\text{Fe}_2\text{B}$  and  $\text{FeB}$  are also found on the wear scar. The improvement of antiwear properties of the oil comes from the formation of the deposition layer and permeating layer.

#### [ REFERENCES ]

- [ 1 ] Nakahara T. Effect of molybdenum sulfide dispersed in oil on friction in journal bearings [ J ]. *Toraiborajisuto*, (in Japanese), 1991, 36(4): 289–295.
- [ 2 ] Khokhlow A A. Antifriction additive for lubricating oils [ P ]. SU 1635904, 1991.
- [ 3 ] Wan G T Y, Spikes H A. The behaviour of suspended solid particles in rolling and sliding elastohydrodynamic contacts [ J ]. *Tribo Trans*, 1988, 31(1): 12–21.
- [ 4 ] Bart Z W J. Some investigations on the influence of particle size on the lubrication effectiveness of molybdenum disulfide [ J ]. *ASLE Trans*, 1972, 15(2): 207–215.
- [ 5 ] Shebalin A I. Lubricating composition with a solid friction modifier [ P ]. WO 9104311, 1991.
- [ 6 ] Gupta B K, Bhushan B. Fullerene particles as an additive to liquid lubricants and greases for low friction and wear [ J ]. *Lubr Eng*, 1994, 50(7): 524–528.
- [ 7 ] DONG Jun-xiu, HU Ze-shan. A study of the antiwear and friction-reducing properties of the lubricant additive, nanometer zinc borate [ J ]. *Tribo Int*, 1998, 31(5): 219–223.
- [ 8 ] QIU Sun-qing, DONG Jun-xiu, CHEN Guo-xu. Tribological properties of  $\text{CeF}_3$  nanoparticles as additives in lubricating oils [ J ]. *Wear*, 1999, 230: 35–38.
- [ 9 ] FAN Jing-lian, HUANG Bai-yun, QU Xuan-hui. W-Ni-Fe nanostructure materials synthesized by high energy ball milling [ J ]. *Trans Nonferrous Met Soc China*, 2000, 10(1): 57–59.
- [ 10 ] CHEN Zhao-hui, CHEN Zhen-hua, ZHOU Duo-san, et al. Application of nanocrystalline  $\text{MnNi}_5$  for activation of  $\text{ZrCrNi}$  hydride electrodes [ J ]. *The Chinese Journal of Nonferrous Metals*, (in Chinese), 1999, 9(1): 65–68.
- [ 11 ] HU Ze-shan, DONG Jun-xiu, CHEN Guo-xu. Replacing solvent drying technique for nanometer particle preparation [ J ]. *J of Colloid and Interface Science*, 1998, 208: 367–372.
- [ 12 ] Moulder J F, William F S, Peter E S, et al. *Handbook of X-ray Photoelectron Spectroscopy* [ M ]. New York: Perkin Elmer corporation, Physical Electronic Division, 1979. 215–237.
- [ 13 ] DONG Jun-xiu, CHEN Guo-xu, LUO Xiu-ming. A new concept-formation of permeating layers from non-active antiwear addition [ J ]. *Lubr Eng*, 1994, 50(1): 17–22.

( Edited by LONG Hua-zhong )