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Friction Mechanism of Nano-MMT/In Composite on Steel Ball Higher Pair

Yang Cao^a, Taoyue Yang*^b, Dabin Zhang^a, Quan Wang^c, Xiaohui Guo^c

^a College of Mechanical Engineering, Guizhou University, Guiyang, 550025, P.R.China,

^b Guizhou Vocational Technical Institute, Guiyang, 550023, P.R.China,

[°] Guangxi ZhongYan Ltd, Nanning Guangxi, 530001, P.R.China,

29050672@qq.com

In order to obtain the tribological properties of nano-montmorillonite (MMT) / In lubricant additive on steel ball tribo-pair. 1:1 MMT/In composite nano-powders were prepared by nano-MMT and nano-In modified with coupling agent KH550, and the nano-MMT/In lubricating oil dispersion system was prepared by 150N base opl containing 3 mass fraction of nano-MMT/In composite powders, and then took the dispersion effect by laser particle size analyzer, TEM and IR. The anti-wear and friction-reducing behaviors of that lubricating oil dispersion system were observed on the MMU-10G abrasive-wear tester with 45#steel tribo-pair, the morphology and the element of the worn surfaces were analyzed by SEM and EDX. The results showed that the grain size of the modified nano-MMT/In was smaller than the one without modification, and the modified nano-MMT/In had the good dispersion in the system; compare with the one in base oil system had declined by 22.54 %, and the wear spot diameter had declined by 12.62 %; The surface of sample after friction had created the self-repairing film that contain the characteristic element of MMT and In, that phenomenon was caused by the interaction of nano-MMT and nano-In.

1. Introduction

In the mechanical system, the higher pair friction exists widely, such as gear drive, ball bearings, etc. The performance of lubricating oil additives in the higher pair environment is an important index. Nano-mineral (Gao C P, Wang Y M, Pan Z D, 2014) (Zhang Z, Chen G R, Li H F et al, 2014) (Zhao F Y, 2014), nano-metal (Ruan T G, Zhou G Y, Xie X D et al, 2015) (Guo Z G, Xu J S, Gu K L et al, 2005) (Li Z, Ding X, Luo M et al, 2015) and composite nano-materials (Wu X M, Zhou Y K, Yang L et al, 2014) (Gao Y Z, Zhang H C, Wang L et al, 2005) have a lot of research on the four ball friction and wear testing machine for the tribological properties, and the better anti-friction and anti - wear effects are obtained. Montmorillonite (MMT) is a typical kind of layered silicate clay mineral (Jiang G L and Zhang P P, 2005), and Indium (In) is a kind of low-melting-point metal with square crystal structure. We have studied the tribological properties of nano MMT/In composite nano additives in low friction environment. However, the tribological properties of the higher pair friction are still to be studied, and there is still no relevant reports at home and abroad. So this article will study on the tribology performance of nano-MMT/In composite nano-material, as the lubricant additives, to steel ball friction pair, and analyze its tribology performance improvement and film-forming mechanism.

2. Experiment

2.1 Experiment materials and equipments

Material and reagents include: nano Montmorillonite (Zhejiang Fenghong Clay Chemicals Co., Ltd); nano Indium (Shanghai Chao Er Nano Technology Co., Ltd); KH550 silane coupling agent (chemically pure, Nanjing Shuguang Chemical plant); base lubricants (150N, Jiangsu Kunshan); absolute ethyl alcohol (chemically pure, commercially available).

Instruments and equipments include: MMU-10G friction-abrasion testing machine; IRAffinity-1 Fourier transform infared spectrometer; Winner801 laser particle size analyzer; JSM-6490LV scanning electron microscope; JEM-2000FXII transmission electron microscope.

2.2 Surface modification of MMT/In composite nano materials and the lubrication system preparation

2.2.1 Nano particle modification

Prepare enough 3 % wt KH550 ethanol solution for the surface modification of nano MMT and In, and then mix a certain amount of nano MMT with above-mentioned KH550 solution, and fully stir it for half an hour at a temperature of about 65 °C, eliminate the solution and make repeated extraction with ethanol, further eliminate the ethanol solution after extraction, dry the solid phase matters in the drying oven, obtaining the nano MMT modified by KH550. Prepare to obtain the nano in modified by KH550 with the same method. Finally, inspect the modification effects of the two with IR.

2.2.2 Preparation of nano MMT/In base oil system

Put KH550 modified nano MMT and In of equal mass respectively into the ethanol solution, forming dispersed systems by stirring and ultrasonic dispersion, and then mix and fully stir the two systems, after filtering and drying, the solid phase and liquid phase are separated, obtaining the blending composite nano MMT/In; prepare to obtain the composite nano MMT/In that isn't modified with the same method.

Add the above prepared two kinds of 1:1 MMT/In composite nano powder into the 150 N base oil by 3 % wt, and fully stir it for 30 minutes, and then disperse it with ultrasonic dispersion instrument for 30 minutes to make the oil sample system for experiment, among which, mark the surface modified nano MMT/In base oil system sample as KMIO, and the unmodified as MIO. Set aside the oil sample KMIO and MIO for 1 hour, and then carry out particle size analysis with Winner801 analyzer.



Figure 1: IR spectra of nano MMT and nano In

2.2.3 Modification effect analysis

The nano MMT itself has hydrophilic hydroxyl groups, while the powder form In may also act with water and form hydroxyl, thus the KH550 converts the hydrophilic surface to hydrophobic surface by reaction with their hydroxyls, realizing the purpose of modifying their performance. Refer to the Fig 1 for the FT-IR spectrogram of nano MMT and In before and after modification.

For Fig 1 (a), the MMT modified by coupling reagent also produces new absorption peaks at 2970 cm⁻¹ and 1338 cm⁻¹, among which 2970 cm⁻¹ is the characteristic peak of -CH2, -CH3, and 1338 cm⁻¹ is the characteristic peak of amide groups. For Fig 1 (b), the ln modified by coupling reagent also produces new absorption peaks at 2940 cm⁻¹, 2860 cm⁻¹ and 1404 cm⁻¹, among which 2940 cm⁻¹ and 2860 cm⁻¹ are the characteristic peaks of -CH2, -CH3, and 1404 cm⁻¹ is the characteristic peak of amide groups. These indicate that the modified MMT and ln surface produce chemical reactions with KH550, and their surface is coated by KH550 modifier.

Fig 2 (a) shows that the average particle diameter of the solid phase matters in oil sample MIO is 327.71 nm, with a dispersion index of 0.422, and particulate matters of a diameter larger than both nano-MMT and nano-In appear, indicating that nano-MMT and In have obvious agglomeration phenomenon in base oil. Fig 3(a) is the TEM picture of MIO system, in which the darker color shows the nano-In, the lighter color shows the nano-MMT, and the particle diameter of agglomeration formed by the two is larger. Fig 2 (b) shows that the average particle diameter of the oil sample KMIO is just 46.79 nm, with a dispersion index of 0.321, indicating that the

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modified nano particle maintains its original particle diameter after forming the dispersion system. It is also reflected from the TEM picture in Fig 3(b) that the surface modified nano-MMT and nano-In almost have no agglomeration phenomenon in the lubrication system, with favorable dispersing performance.







Figure 3: TEM of MIO and KMIO sample

2.3 Friction and wear test

The friction and wear test is carried out on the MS-10 four-ball friction and wear tester. The steel ball friction sample is divided into two groups to compare the friction and wear performance in the oil sample BO and KMIO. Other samples are HMB and HMMI. The test is conducted according to (SH/T0189-92) rules. The washing gasoline is used to clean the four test balls, upper ball fixture, oil cup and each part contacting the test oil carefully. The four balls are cleaned for 10min by using the petroleum ether in the ultrasonic wave, and then they are dried and put into the fixture and poured into the testing oil sample. 147 N load is imposed, and the temperature is risen to 75 ± 2 °C. After running for 60 min at the rotary speed of 1200 r/min, the oil cup is taken out, and the wear scar diameter of the lower steel ball is measured in the microscope.

Measure the wear scar diameter of each steel ball twice, once along the central ray of the oil cup, and the other vertical to it with the precision of 0.01 mm. Test the three steel balls for six times to calculate the mean value which is considered as the final test data. If the wear scar is ellipse, then test in the wear scar direction and the vertical direction once more.

Fill the tested data into the operation interface of the four-ball tester and the production test report, and get the mean friction coefficient. After the friction and wear test, the morphological analysis is conducted by using JSM-6490LV and SEM on the sample. The surface composition analysis is conducted by using EDX, thus elaborating the mechanism of the tribological properties.

3. Results and discussion

3.1 Wear scar diameter

Figure 4 is an optical picture of the lower static ball of the steel ball friction pair of two oil samples. As can be seen from the figure, the wear scar diameter of friction sample in the solid-phase nano-powder lubrication system is smaller than that in the base oil. The wear scar diameter of the HMB sample in the base oil is 0.753 mm, and the wear scar diameter of HMMI sample is 0.658 mm, reducing by 12.62 %. This indicates that the anti-wear property of MMT/In compound nano-powder oil sample is superior to that of the base oil.



(a) HMB Sample



(b) HMMI Sample

Figure 4: Wear scar diameter of each sample

3.2 Friction coefficient

The average friction coefficient of each friction sample in the four-ball test is shown in Table 1. The friction coefficient of oil sample with compound powder additive is 0.055, and the average friction coefficient of the friction sample in the pure base oil is 0.071, reducing by 22.54 %. It means the nanometer MMT and In in the lubrication system can reduce the friction coefficient.

Table 1: The average friction coefficient of e	each friction sample(µ)
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Sample	HMB	HMMI
the average friction coefficient /µ	0.071	0.055

3.3 Mechanical analysis

Figure 5 shows the SEM morphology of the surface after the friction sample (upper rotary ball) test. Figure (a) shows the friction scratch of the HMB sample is deep and dense with sharp crack edge and severe wear. Most wear scars of the HMMI sample surface shown in Figure (b) is light and the convex-concave contrast is narrow. The bottom of the wear scare groove is blur and seems to have covering masses. The superficial roughness has been reduced significantly, which suggests the covering film has been formed in the HMMI sample friction process.





(a) SEM of HMB sample worn surface

(b)SEM of HMMI sample worn surface

Figure 5: SEM micrographs of worn surface lubricated with different lubricants(upper sample)

Figure 6 shows the EDX analysis result of the friction surface of each sample. As can be seen, on condition of the dispersed lubrication of MMT/In nanometer compound powder, the sample surface contains elements from the lubrication system MMT/In different from the matrix, and the alloy film different from the matrix materials has been formed. The characteristic peak of AI, Mg and In elements has been added on the EDX, and the concentration of new elements can also be seen from the data in Table 2.



Figure 6: EDX characterizations of the upper sample surfaces

Generally, it is believed that the antifriction and anti-wear mechanism of nanometer additives on higher pair friction has the following few explanations: 1) The ball effect has been produced on the friction surface by the particle, and the effect is rather clear in the initial period of the friction operation; 2) The physical and chemical reaction occur on the nanometer additive and matrix surface to form the repairing film with low surface energy. The completeness and covering area can directly affect the friction coefficient; 3) The hard nanometer particle has the grinding and polishing effect on rough surface; 4) The shear strength of soft metal nanometer particle is low, which is easy to form the transfer film on the friction antithetical material. In this way, the friction will occur between the transfer film and the soft metal, thus reducing the friction coefficient.

Table 2: Elemental atomic percentage of the worn surfaces with different lubricants(%)

Element	С	Mg	Al	Si	Fe	In
HMB sample	22.55	0	0	0.16	59.26	0
HMMI sample	37.79	0.01	0.07	0.45	48.08	0.16

In higher pair friction sample, due to the short running time (60 min), it is hard to form clear alloying repairing film containing MMT characteristic elements formed by chemical reactions on the sample surface; In contrast, the heat coating film is easy to be formed by the soft metal. From the EDX analysis, we can see the content of In is higher than that of MMT characteristic elements, indicating the repairing film formed on the sample surface is mainly In. This is because the nanometer particle shear strength of In is low and is easy to form the film on the friction antithetical material. As a result, the anti-wear property can be enhanced. Meanwhile, soft metal In has strong ability to reduce the friction, so the friction coefficient can be improved. MMT hard-phase particle can produce certain ball bearing effect and grinding and polishing effect on the friction pair, so the friction coefficient can be reduced further. The MMT characteristic element can be tested on the friction surface, which means the MMT nanometer particle and matrix surface have had physical and chemical reactions with the nanometer In with the proceeding of friction, thus forming the repairing film with low surface energy.

4. Conclusions

(1) The nano MMT and nano In modified by KH550 disperse equally in the base oil, and there is no agglomeration phenomenon appeared in the lubrication system, the original grain diameter features of nano MMT and nano In can be maintained.

(2) MMT/In composite nano additives have remarkable anti-friction effects on steel ball friction pair, the average friction coefficient of sample HMMI reduced 22.54 % than that of sample HMB.

(3) MMT/In composite nano additives have self-repairing capability on steel ball friction pair. The wear spot diameter of sample HMMI reduced 12.62 % than that of sample HMB.

(4) In the lubrication system with MMT/In composite nano additives, by observation with SEM, there are self-repairing films of darker color formed on the surface of steel ball friction pair, by inspection with EDX, the surface composition contain the characteristic elements for MMT and In, indicating that there are self-repairing films formed on the surface of the test-piece, greatly improving the anti-friction and abrasion resistant performance of the friction pair.

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