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Influence of nano-serpentine mineral powder as a lubricating additive on the high-temperature tribological properties of metal friction pairs

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High-temperature tribological properties of 45 steel friction pairs were investigated, and nano-serpentine mineral powder (nano-serpentine), nano-serpentine mineral powder + nano-lamellar expandable graphite (Nano-EG + nano-serpentine) were used as mineral oil additives, respectively. By means of SEM, TEM and EDS, the worn surface morphology and composition distribution on the contact surfaces with different lubricating additives at high temperature were compared and analyzed. The results show that under high-temperature conditions, using nano-serpentine mineral powder and Nano-EG combined as lubricating oil additives, a self-repairing transfer film is formed. At an early high-temperature friction stage, the friction coefficient decreases with time, which leads to better anti-friction properties. As regards wear resistance, the high-temperature friction tribological properties of 45 steel with a Nano-EG + nano-serpentine additive were superior to those with only nano-serpentine mineral powder additive, which is mainly due to the combination of the good self-lubrication property of Nano-EG and the self-repairing effect of the nano-serpentine mineral powder.

Keywords: High temperature; Nano-serpentine; Surface morphology; Self-repairing film; Tribological properties

INTRODUCTION

High-temperature friction, as the name implies, refers to the friction and wear problems at high temperature, which mainly includes two aspects: one is the continuous operation of the machine at a higher temperature; the second is the machine working at a higher temperature periodically. Typical for the former case are aircraft engines, space vehicles and different metallurgical equipment. For the latter case, tribological problems usually appear in certain circumstances such as lean oil leakage/oil cutting-off. Especially, under insufficient lubrication conditions, the contact parts of relative motion may lead to temporarily high temperature.

Problems about high temperature friction and wear are mainly solved through surface engineering technology (PVD/PVC coating, laser cladding layer, carburizing/nitrogen technology, etc.), lubricating materials and wear-resistant materials. Especially, in the case of oil shortage, how to reduce the friction and wear of the kinematic pair is one of the research topics of high temperature tribological design, which is important not only for the normal operation of the machine, but also to the reliability and service life of the friction pairs.

Using surface engineering technology, Liu, Deng *et al.* co-workers studied the friction and wear properties of five PVD nitride coatings at 200-700ºC and found that the five kinds of coatings would cause oxidation wear at high temperature [1]. Of all coatings, AlTiN was most adapted to high temperature, high speed and heavy load. At the same time, the different hardness and chemical activity caused different wear mechanisms. The research on the high temperature friction and wear properties of Ni-P alloy coating by Li *et al.* showed that a Ni-P alloy layer prepared by electric brush plating technology could effectively improve the tribological properties of the specimen at 450 ºC [2]. Zhang and Ceng used grid laser quenching and low-temperature ion sulphurizing technology to treat 42MnCr52 steel surface. Because of the increase in hardness and oil storage function of the porous sulfide layer, processed samples have significant antifriction and wear resistance properties [3]. The friction and wear properties of Fe-Al/TiC laser cladding layer against Si3N4 ball were studied by Wang and Gao [4]. It was found that delamination wear is the high-temperature wear mechanism of the cladding layer.

From the lubrication technology and materials matching, Li and Xiong used laser surface texturing technology to etch micropores on the surface of nickel-based material in order to store lubricating oil, and utilized plasma surface alloying technology to diffuse Mo or multi-permeate Mo/N on the microporous surface to improve the surface wear resistance [5-7]. Sun and Qiao found that SiO_2 nanoparticles could significantly enhance the high-temperature wear resistance properties of rapeseed oil at 500 ºC [8]. The friction coefficient is only 0.16 and the wear loss is reduced by more than 80%. Yao and Yue found that friction and wear performances of double imidazole ionic liquid with alkyl chain are significantly better than with ether

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chain at 250 ºC. Shortening the chain length of double imidazole ionic liquid is beneficial to the tribological properties [9].

To improve the wear resistance of materials, TiB2-SiC multiphase ceramics was *in situ* synthesized by Zhou and Xiao [10]. The results showed that the physical properties and the high-temperature tribological properties of SiC ceramics are improved, and high temperature oxidation is the main wear mechanism. Through the research of the high-temperature friction and wear properties of PCD tools, Al_2O_3/TiC ceramic cutting tools and carbide tools, Zhang and Deng revealed the high-temperature friction and wear mechanism of typical hard-brittle material tools [11], and provided a theoretical basis for the design and selection of tool materials. High-temperature friction and wear behavior of M50 high-speed steel were investigated by Liu [12]. The results showed that the combined action of friction heat and ambient temperature causes a semi- molten state of the contact surfaces above 400 ºC. As an inevitable result, a layer of metal film is formed during the sliding process, which is the main reason for reducing the friction coefficient.

Nano mineral serpentine powder, as a lubricating oil additive, showed good tribological properties and its self-repairing behavior has been confirmed by some scholars [13-16]. In this paper, nano expanded graphite with lamellar structure (Nano-EG) was used as antifriction agent, nano mineral serpentine powder (nano-serpentine) as lubricating oil additive (self-repairing agent). The tribological properties of steel/steel friction pairs at high temperature were studied by corresponding experimental design in order to reduce friction and wear of metal pairs in case of oil cutting-off.

EXPERIMENTAL

Nano-serpentine powder was prepared by mechanical milling and Nano-EG by ultrasonic cavitation. A certain amount of nano-serpentine powder and/or Nano-EG was added to the base oil. By electromagnetic stirring the lubricating oil with nano additives was obtained. Figs. 1 and 2 show the TEM (JEM-2010, JEOL company, Japan) image if nano-serpentine and the SEM (S-4800 SEM, Hitachi company, Japan) image of Nano-EG, respectively. From Fig. 1 it is seen that the nano-serpentine powder mainly shows two kinds of shapes: one is approximately spherical (diameter is less than 100 nm), the other is a long column type (diameter is less than 50 nm). As can be seen from Fig. 2, the lamellar structure of expanded graphite is removed, and its thickness is less than 25 nm.

The lubricating oil with different additives was put into the oil tank located on the lower base of the testing machine, and then the friction pair was placed in a high-temperature insulation box for heating to 350ºC. The tribological tests were carried by using MMU-5G high temperature friction and wear tester (Jinan Jingcheng Test Technology Co.) at a load test of 200 N, 400 N and 800 N, and a speed of 346 r/min. The upper and lower specimens were 45 steel, respectively. The other test details can be found elsewhere [15, 16]. In order to reduce the dispersion of test results, each test was repeated three times under the same conditions. The antifriction and wear resistance of the friction pairs with different lubricating oil additives at high temperature were determined. Meanwhile, the wear mechanism was discussed by means of SEM and EDS.

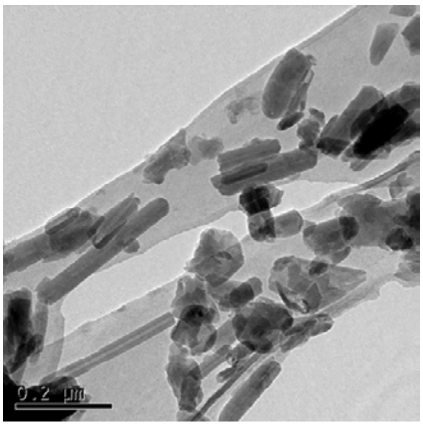


Fig. 1. TEM image of nano-serpentine powder

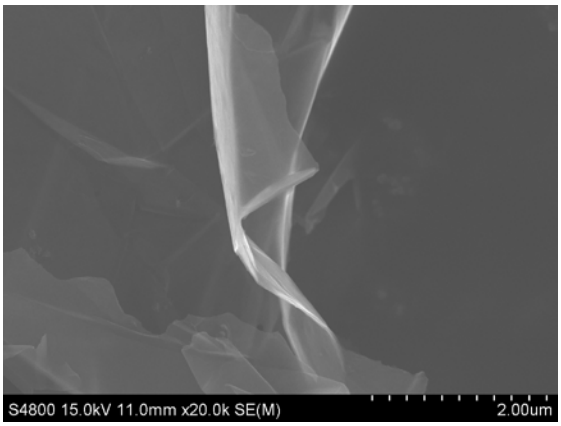
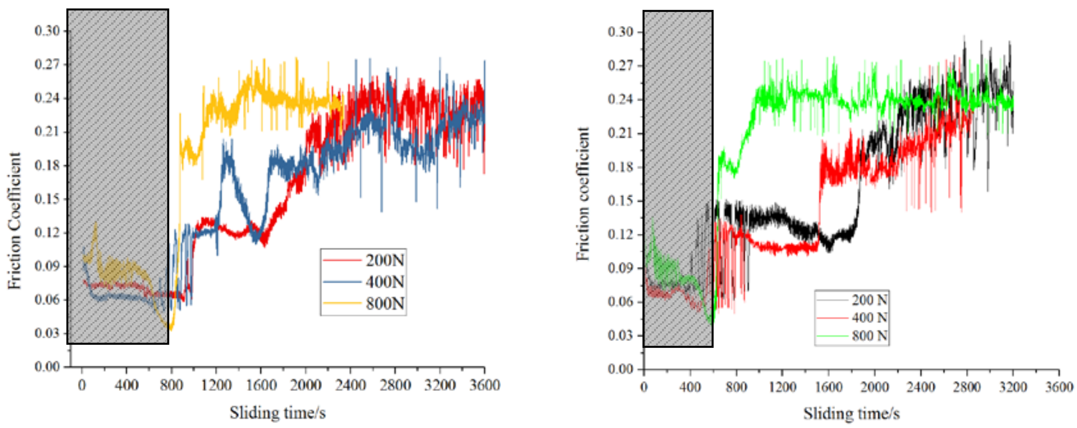


Fig. 2. SEM image of Nano-EG

RESULTS AND DISCUSSION

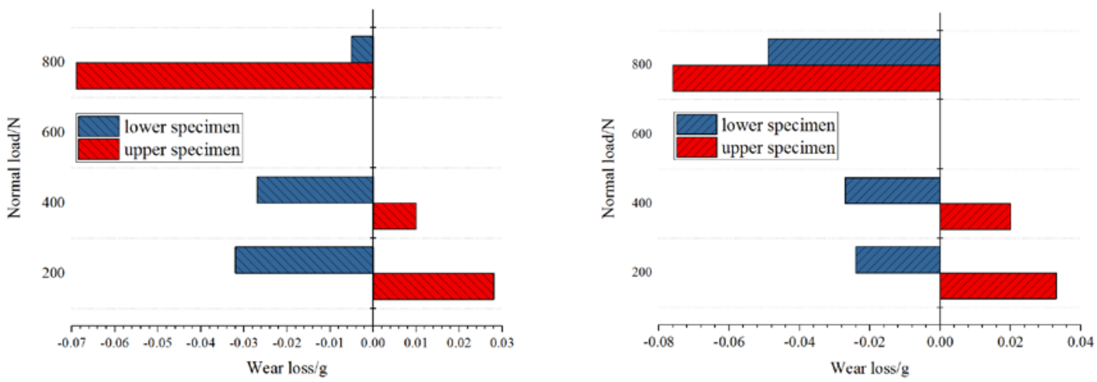
Fig. 3 shows the friction coefficient-time history curve of friction pairs with different additives. According to the figure, it can be seen that the friction coefficient is characterized by three obvious stages under high temperature. In the initial stage of wear, the friction coefficient is small and gradually decreases. After a period of time, the friction coefficient rises suddenly and enters a relatively stable platform. Then the friction coefficient continues to increase and fluctuates at higher values. Compared to Fig. 3a and 3b, where the additive is Nano-EG+ nano-serpentine, the duration of the first stage is longer than that of nano-serpentine additive, and the antifriction effect is better. At present, the global air disasters that caused by oil supply disruptions of aircraft engine lubrication system occur frequently [17-19]. When oil cutting-off of the aircraft lubricating system takes place, the sustained flight capability has become the focus of attention. After oil cutting-off, the carrying capacity of bearings under full load operation conditions is very important for the safety of the aircraft. For example, the turbine spindle bearings of aircraft in the design, it is required to work 30 s under oil cutting-off in normal flight conditions [20]. Through the tribological tests, the lubricating oil by adding nano repairing agent and self lubricant can not only effectively meet this requirement, but also greatly prolong the working time of the engine.

Fig. 4 illustrates the wear of the upper and lower specimens with different additives. The negative value means that the mass after the test is not reduced, but increased to some degree. It is obvious that under the conditions of low and medium load, there is a small amount of negative wear loss for the lower specimens and a small amount of wear loss for the upper specimens. However, under high load negative wear loss appears for both upper and lower samples. Compared with Fig. 4a and 4b, it is seen that when nano-serpentine is added, the wear loss of the upper and lower samples is higher under higher load. When the contact load was 800 N, the maximum negative wear loss of the upper specimens was registered, and the mass increase reached 0.069 g and 0.076 g, respectively.



a b

Fig. 3. History curve of friction coefficient-time with different additives: a) Nano-EG + nano-serpentine powder, b) nano-serpentine powder

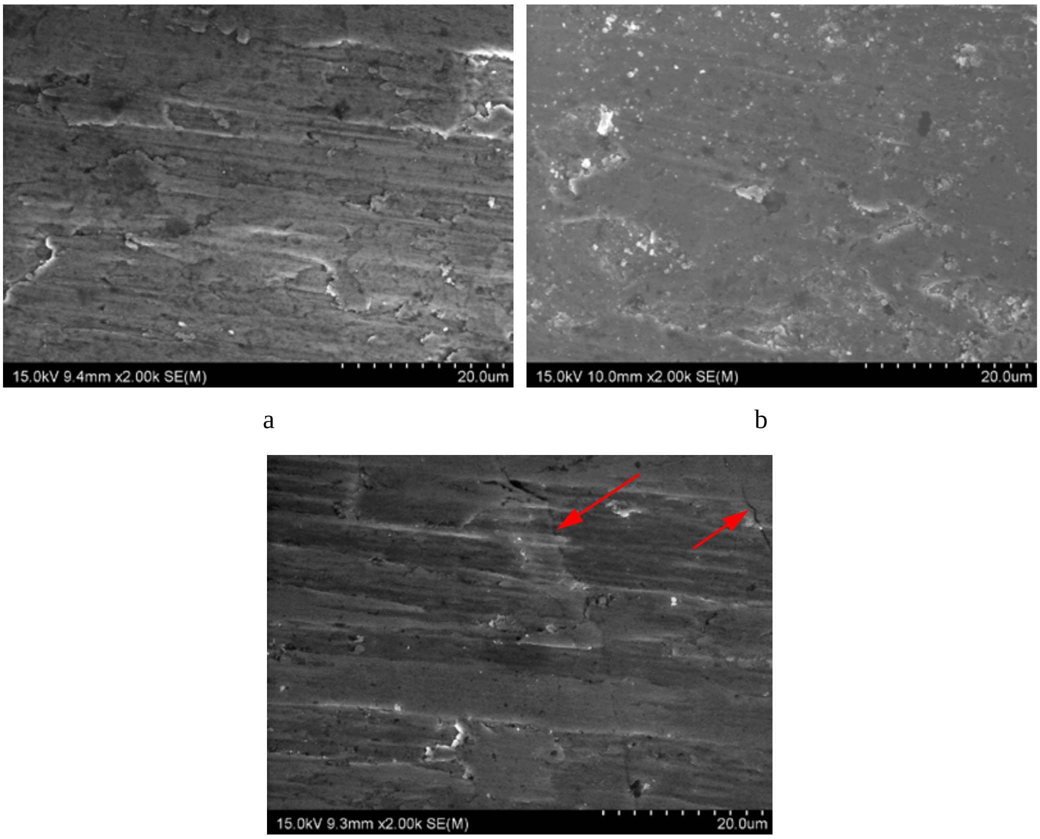


a b

Fig. 4. Comparison of wear loss of specimens with different additives: a) Nano-EG + nano-serpentine powder, b) nano-serpentine powder

Meanwhile, when the additive was Nano-EG + nano-serpentine or nano-serpentine, the mass of the lower specimens increased to 0.005 g and 0.051 g, respectively. This is because Nano-EG as self-lubricating additive could hinder the spreading of nano-serpentine on the contact surface so that the process of chemical reaction occurred on the contact surface [15, 16]. From the point-of-view of metal self-repairing and reduced wear of the surface, Nano-EG + nano-serpentine or nano- serpentine as lubricating oil additives not only did not lead to a phenomenon of obvious wear under the conditions of oil cutting-off at high temperature, but could also effectively improve the contact state and fit between friction pairs.

Fig. 5 shows the SEM image of the worn surface morphology when the additive is nano-serpentine. As shown in Fig. 5a, the surface wear is mainly based on a slight ploughing effect and adhesion under light load conditions. Under medium load, the wear morphology is smooth and the adhesion wear is the main wear form, as shown in Fig. 5b. When the load is large, the wear morphology of the specimens is mainly a plastic flow. Due to the heavy tangential force, the self-repairing film formed on the worn surface forms cracks (red arrows in the figure), as shown in Fig. 5c. Fig. 6 shows the SEM worn surface morphology when the additive is Nano-EG+ nano-serpentine. As can be seen from the figure, the addition of Nano-EG significantly improves the wear form of contact surface. At different loads, the wear surface is relatively smooth. When the load is small, the slight adhesive wear is the main, as shown in Fig. 6a).Under medium load, the worn surface is mainly composed of minor ploughing and a small amount of adhesion, as shown in Fig. 6b). When the load is large, the wear is changed into adhesive wear. Also, due to the larger tangential force the self-repairing film formed on the worn surface also appeared cracks (red arrow in the figure), as shown in Fig. 6c). Because of the addition of Nano-EG, the wear forms with different lubricating oil additive are different. Nano-EG with the self-lubrication property improves the wear resistance of the contact surface and also weakens the ploughing effect of nano-serpentine on the contact surface. Fig. 7 shows the local EDS energy spectrum analysis of the worn surface topography for different additives (Fig. 7a and b).



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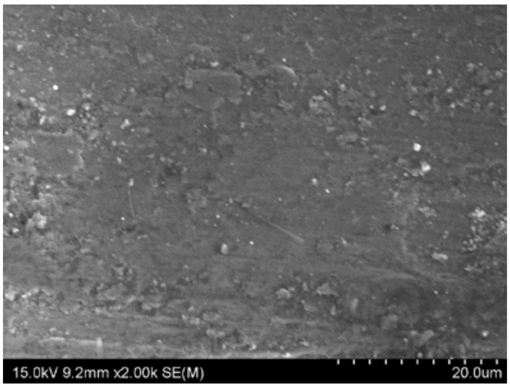
c

Fig. 5. Morphology of the worn surfaces with nano-serpentine additive at different loads: a) 200N, b) 400 N, c) 800N



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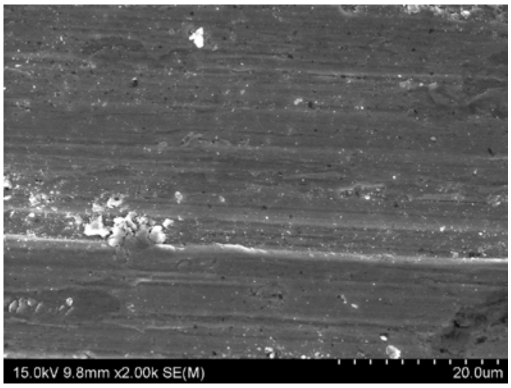

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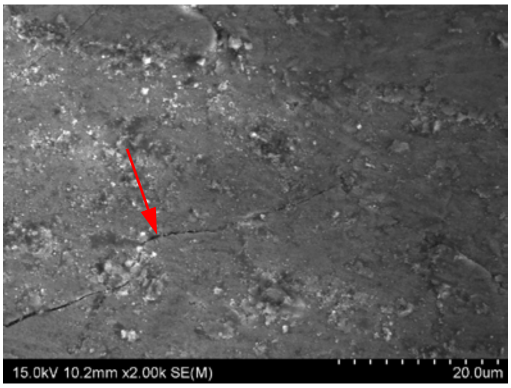

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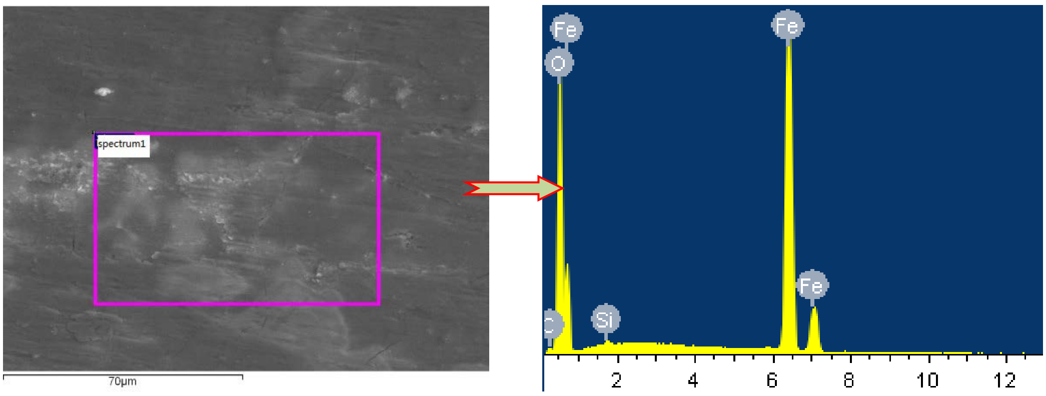
Fig. 6. Morphology of the worn surfaces with Nano-EG + nano-serpentine additives at different loads: a) 200N, b) 400 N, c) 800N



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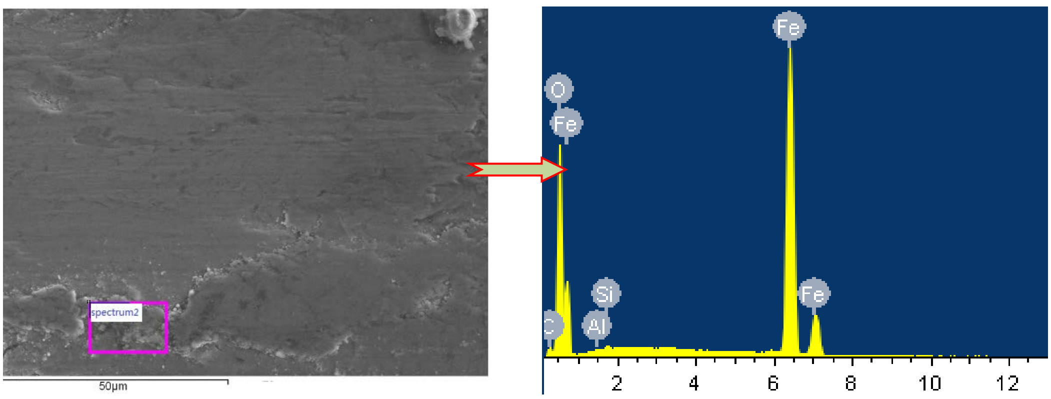

b

Fig. 7. Energy spectrum analysis of worn surface with different additives: a) nano-serpentine powder, b) Nano-EG + nano-serpentine powder

The characteristic element Mg of serpentine in energy spectrum not found, which indicates that in the friction test process, the magnesium iron has been replaced by iron [15, 16]. At the same time, oxygen atoms appear in the spectrum, which shows that the chemical reaction occurs in the process of friction. There are two possible ways to introduce oxygen atoms. One is decom- position product silica during heat treatment and hydrolysis of serpentine powder under high temperature condition, two is a chemical reaction between the oxygen atoms and iron. According to the friction coefficient curve mentioned above, the friction coefficient tends to relative stable value with the test time. The results show that the variation of the friction coefficient is mainly controlled by the chemical reaction at the friction interface. According to Fig. 4, the larger the external load is, the more easily the additives are deposited on the interface and the chemical reaction occurs on the friction surface. Therefore, varying degrees of mass increase for the upper and lower specimens appear.

CONCLUSIONS

The paper investigated the high temperature tribological properties of metal friction pair with Nano-EG and nano-serpentine mineral powder as lubricate additives. Some conclusions are as follows:

1) The addition of nano-serpentine mineral powder and Nano-EG into the lubricating oil could effectively improve the wear resistance of the metal friction pair under high temperature condition. More important, in the initial period of friction, the friction coefficient decreased with the running time, which is very beneficial to alleviate/reduce the friction and wear problems caused by oil cutting-off at short time under high temperature.

2) The varying degrees of wear loss for the upper and lower specimens appeared. Lubricating additive material was transferred to the contact surfaces, which is the nature of the wear resistance can be improved.

3) The high temperature tribological properties of metal friction pairs were influenced by kind of additives. When the nano-serpentine mineral powder was added, the wear form was mainly composed of plough and adhesive wear. However, when Nano-EG and nano-serpentine were added, slight adhesive wear was the main wear form.

4) With nano-serpentine used as lubricating oil additive together with Nano-EG, the tribological properties of the friction pairs at high temperature were superior to those with nano-serpentine mineral powder only. It is mainly because Nano-EG has good self-lubrication property (better antifriction effect), and nano-serpentine mineral powder has a self-repairing effect on the contact surface.

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ВЛИЯНИЕ НА НАНОСЕРПЕНТИНОВ МИНЕРАЛЕН ПРАХ КАТО СМАЗОЧНА ДОБАВКА ВЪРХУ ТРИБОЛОГИЧНИТЕ СВОЙСТВА НА ТРИЕЩИ СЕ ДВОЙКИ МЕТАЛИ

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(Резюме)

Изследвани са високотемпературните трибологични свойства на 45 триещи се стоманени двойки с използване на наносерпентинов минерален прах (наносерпентин) и смес от наносерпентин и наноламелен разширяем графит като минерални маслени добавки. С помощта на SEM, TEM и EDS е изследвана морфологията на износените повърхности и съставът им с използване на различни смазочни добавки при висока температура. Резултатите показват, че при високотемпературни условия с използване на смес от наносерпентин и наноламелен разширяем графит се получава саморегулиращ се трансферен филм. В ранен етап на високотемпературното триене коефициентът на триене намалява с времето, което води до по-добри антифрикционни свойства. По отношение на съпротивлението на износване, високотемпературните трибологични свойства на 45 двойки стомана с добавка от наносерпентин и наноламелен разширяем графит са по-добри от тези с използване само на наносерпентин, което се дължи на комбинацията от добри смазващи свойства на наноламелния разширяем графит и саморегулиращия ефект на наносерпентиновия минерален прах.