

The tribological properties of zinc borate ultrafine powder as a lubricant additive in sunflower oil

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ABSTRACT

This paper presents an investigation on the tribological properties of zinc borate ultrafine powder (ZBUP) employed as a lubricant additive in sunflower oil. The stable dispersions of 0.5 wt%, 1 wt% and 2 wt% ZBUP in sunflower oil were achieved by using an ultrasonic homogenizer. Both a 4-ball tester and a pin-on-disc tester were employed to evaluate the anti-wear and friction reduction capabilities of ZBUP. Tribo-films with dark colour were generated on the worn surfaces and showed a good contrast with the substrate. The worn surface with different morphologies reflected as the colour alterations on the worn surface were observed when different lubricants were applied. The morphology and elemental analysis of the worn surfaces were studied using atomic force microscopy (AFM) and scanning electronic microscopy (SEM). Mechanical properties of the tribo-films and substrates were studied with a nano-indentation tester. Test results suggest that tribo-films generated on the worn surface have a relatively low hardness compared with the steel substrate. The substrates on the worn surfaces lubricated in sunflower oil with the powder demonstrated higher hardness than that of the substrate lubricated with pure sunflower oil due to the possible tribo-chemical reaction between the zinc borate additive and substrate. The combination of sunflower oil with 0.5% ZBUP has delivered the most balanced performance in friction and wear reduction. This study has demonstrated the possibility of application of this industrially applicable solid lubricant additive (zinc borate) with a decomposable vegetable based lubricant oil.

Keywords: *Zinc Borate Ultrafine Powder; Sunflower oil lubricant; Nano-indentation*

INTRODUCTION

The utilization of nano sized or submicron sized particles as an inorganic lubricant additive has received much attention over recent years, owing to their outstanding tribological properties and good environmental friendly feature compared with the traditional organic lubricant additives that contain P, S and Cl elements [1-6]. In particular, many studies have been conducted with borate nanoparticles. Tribological properties of titanium borate [7], ferrous borate, magnesium borate [8] and zinc borate [9] nanoparticles together with dispersing agent sorbitan monostearate have been investigated, and the outstanding lubricating performances were observed. However the expensive and complicated preparation process of nanoparticles hinders their mass application. Compared with nanoparticles, submicron sized particles have won their attention in industrial application due to relatively low cost and simple preparation process although submicron size particles are more thermodynamically unstable in liquid media.

Recent studies show that some of the submicron sized particles employed as lubricant additives have demonstrated outstanding tribological properties. Yu et al. [10] investigated the tribological behaviour of surface coated serpentine ultrafine powders (with average size of 1 μ m). The formation of a tribo film of multi-apertured oxide layer with excellent mechanical properties on the worn surface was observed. Z.S Hu et al. [7-8, 11] also observed the alteration of surface material composition caused by tribo-chemical reaction when borate lubricant additives were used, however the mechanical properties of the worn surfaces using borate lubricant additives have never been characterised.

In this study, a concept of “green” lubricant was investigated. As an attractive alternative to petroleum based oil, vegetable oil was selected as base oil due to its renewable property and environment friendly characteristics [12-13]. Friction reduction and anti-wear properties of sunflower oil with lubricant additive zinc borate were studied using both pin-on-disc and 4-ball tribo testers. The morphology and mechanical properties of worn surface were analysed using SEM and Nano-indentation facility.

MATERIALS AND EXPERIMENTAL APPARATUS

Materials

Commercial sunflower oil which has a viscosity of 27.2mPa.s at 40°C was used as the base oil without further treatment. Zinc borate ultrafine powder (Molecular Formula:

2ZnO · 3B₂O₃ · 3.5H₂O) with particle size of 500-800nm (Shandong Jiqing Chemical Co. Ltd, China) was employed, its TEM picture is shown in Fig.1.

Uniform and stable suspensions with three additive concentrations in weight ratio of 0.5%, 1% and 2% were prepared using an ultrasonic homogenizer (Sonics VC 750) for two minutes. No sedimentation of the additive in the suspensions was observed for 12 hours. The details of suspensions are shown in table 1.

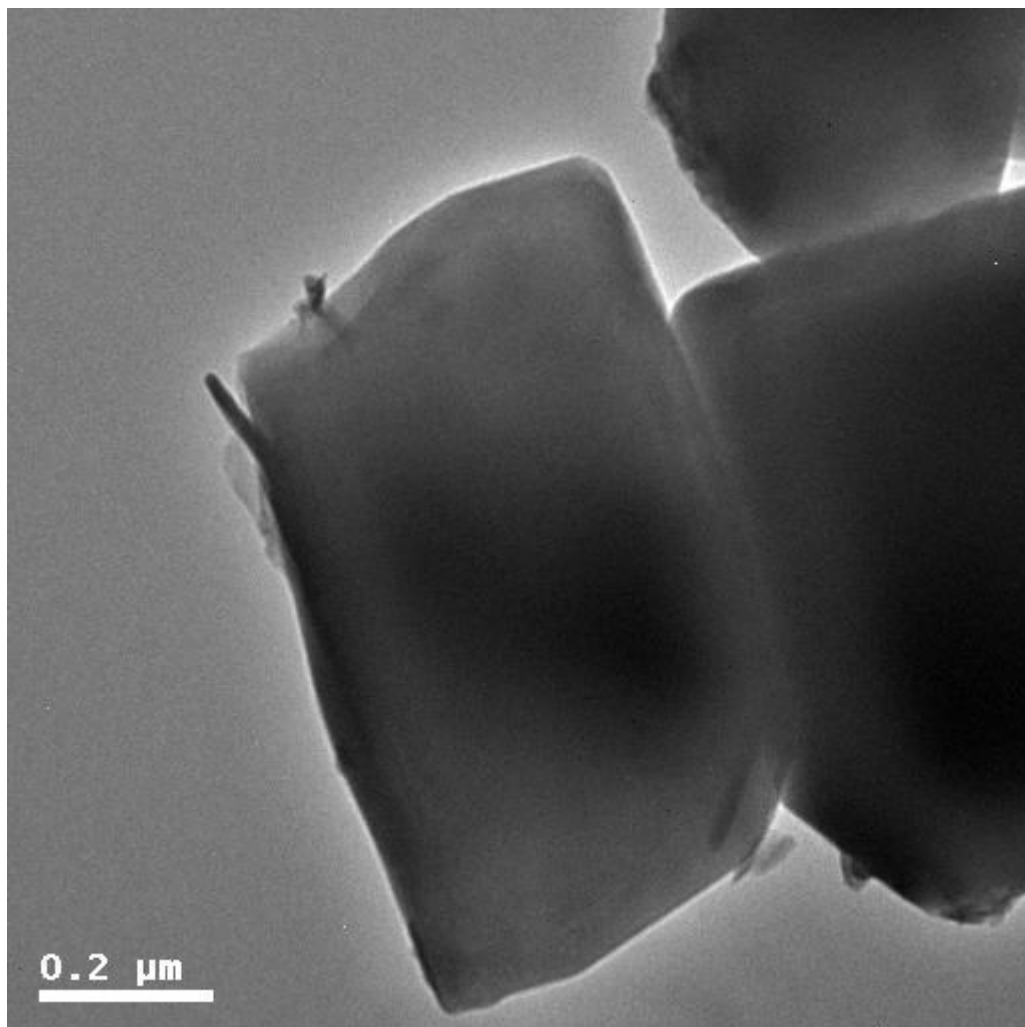


Fig.1 Typical TEM micrograph of ZBUP.

Table 1: Correspondence between suspensions and sample codes.

Concentration and content of suspension	Sample Code
0.5% wt ZBUP in sunflower oil	Sample I
1% wt ZBUP in sunflower oil	Sample II
2% wt ZBUP in sunflower oil	Sample III

Friction and Wear Tests

The friction reduction property of the ZBUP in sunflower oil was evaluated using a POD 2 Pin-on-disc tester (Teer Coatings Ltd.) which has a sensitivity of 0.02N on friction force measurement. All the tests were carried out within a boundary lubrication regime, with a sliding speed of 50mm/s for 30 minutes under 50N load at the ambient temperature of 22°C. The bearing balls of 5mm diameter used as the upper pin in the tests were made of AISI52100 chrome steel with HRC of 59-61. The disc was made of the same material, with 27mm diameter and 3mm thickness. Prior to a test, the disks were grounded and polished to a uniform surface roughness Ra of around 15nm. Both bearing balls and discs were cleaned with acetone in an ultrasonic water bath for five minutes before each test.

The anti-wear property of the ZBUP in sunflower oil was determined using a 4-ball tester. Bearing balls used in the test were also made of AISI 52100 chrome steel with a diameter of 12.7mm and HRC of 59-61. According to ASTM D4172 standard, wear scar diameters (WSD) of the balls were measured under following test conditions: 294N (load), 1200rpm (rotating speed), 30 minutes (experiment duration) and ambient temperature of 22°C. At the end of the test, WSD of the three lower bearing balls were measured using an optical microscope with accuracy of 1µm. The averages of three repeats of the experiments were conducted for each additive concentration to minimise the data scattering.

Surface Analysis

The morphology and the elemental distribution on the wear track of the tested sample discs were studied using JEOL 6100 scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS).

Mechanical properties of the worn surfaces were determined using a Nano-indentation/scratching tester (Micro Material Ltd). A Berkovich indenter with a tip diameter

of 50nm was employed throughout the experiment. For all indentation tests, a constant maximum indentation load of 5mN was applied with the loading/unloading duration of 15 seconds, and the initial load was set to be 0.05mN. Distance between each indent was not less than 15µm in order to avoid any possible interference between neighbouring indents. The micro-hardness of the discs measured with the Nano-indentation/scratching tester is 8.5GPa.

EXPERIMENTAL RESULTS

Friction Reduction Property

Friction coefficients of sunflower oil with various additive concentrations are illustrated in Fig.2. It can be seen that pure sunflower oil exhibited the highest friction coefficient using the Pin-on-disc tester. Sample I delivered the best friction reduction performance. Compared with using pure sunflower oil, more than 13% reduction of friction coefficient was observed by adding 0.5% ZBUP. Sample II demonstrated the similar level of friction reduction to sample III. Further increase of ZBUP concentration to 2% did not yield any extra improvement; on the contrary, both an increase of friction coefficient and an unstable lubricating performance were obtained.

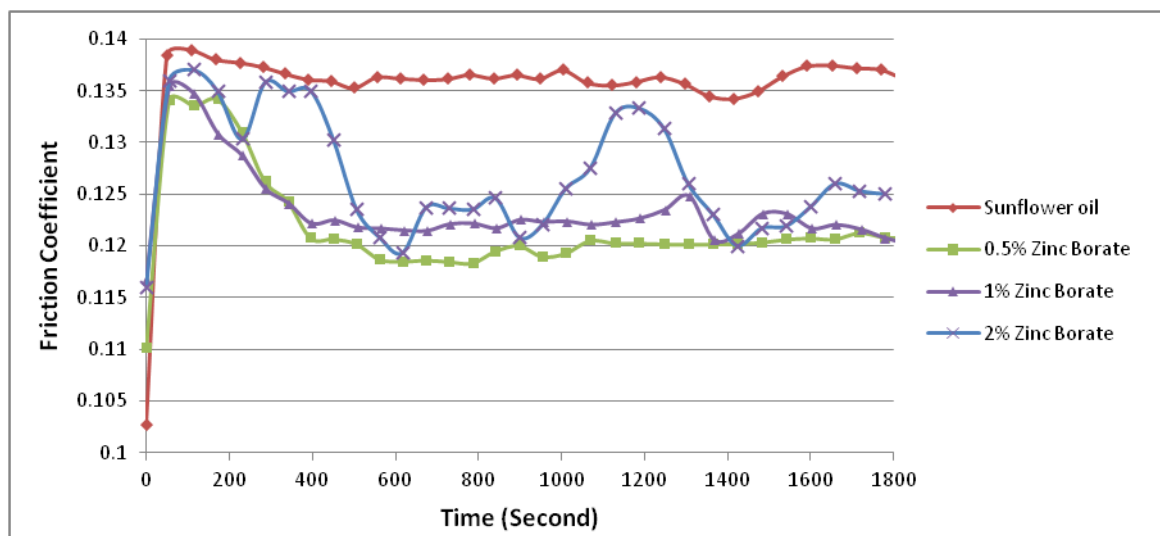


Fig.2 Friction coefficient as a function of testing duration.

Antiwear Property

Fig.3. shows the anti-wear property of sunflower oil with various concentrations of ZBUP. Anti-wear property of the lubricant is reflected by the wear scar diameter (WSD) of the three lower bearing balls in the 4-ball test and a smaller WSD suggests the superior wear

resistance. With the employment of ZBUP, WSDs of the lower bearing balls have been effectively reduced. The WSD is $437\mu\text{m}$ when pure sunflower oil was used as lubricant. This value was reduced to $397\mu\text{m}$ when Sample I was employed and a reduction of 10% was observed in comparison with using sunflower oil alone. It is clear that when no more than 0.5% ZBUP was used, WSD was reduced sharply, however the rate of reduction then slowed down when more than 0.5% of additive was applied.

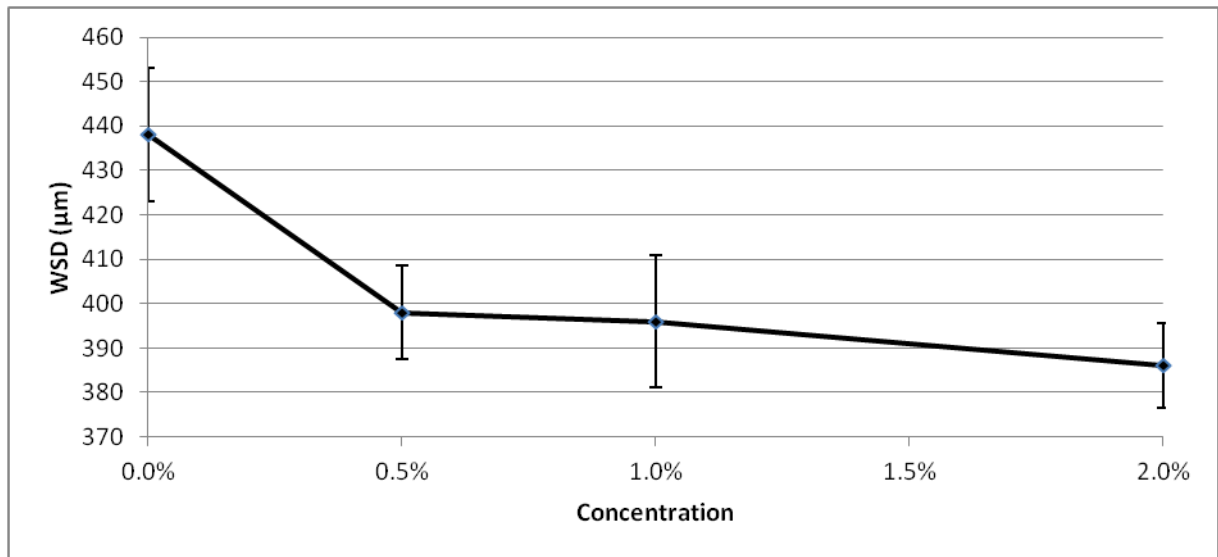


Fig.3 Wear scar diameter of the pins versus ZBUP concentrations.

Morphology Analysis by Optical Microscope

Wear tracks on AISI 52100 steel discs produced in pin-on-disc test were firstly assessed using an optical microscope. As shown in Fig.4, tribo-films appeared in dark colour can be observed on all the wear tracks. They demonstrate a clear contrast to substrate materials appeared in bright colour. A clear change of the morphology on the worn surfaces was observed when different concentrations of ZBUP were applied. Wear tracks (*b*) and (*c*) in Fig.4 were produced using sample I and II respectively. Compared with wear track (*a*), which was lubricated by pure sunflower oil, much less dark areas (tribo-films) can be found on track (*b*) and (*c*). A great amount of dark areas (tribo-films) were also observed on track (*d*) which is lubricated using sample III. It is evident that the appearance of dark area (tribo-films) is in coincidence with an increase of friction coefficient.

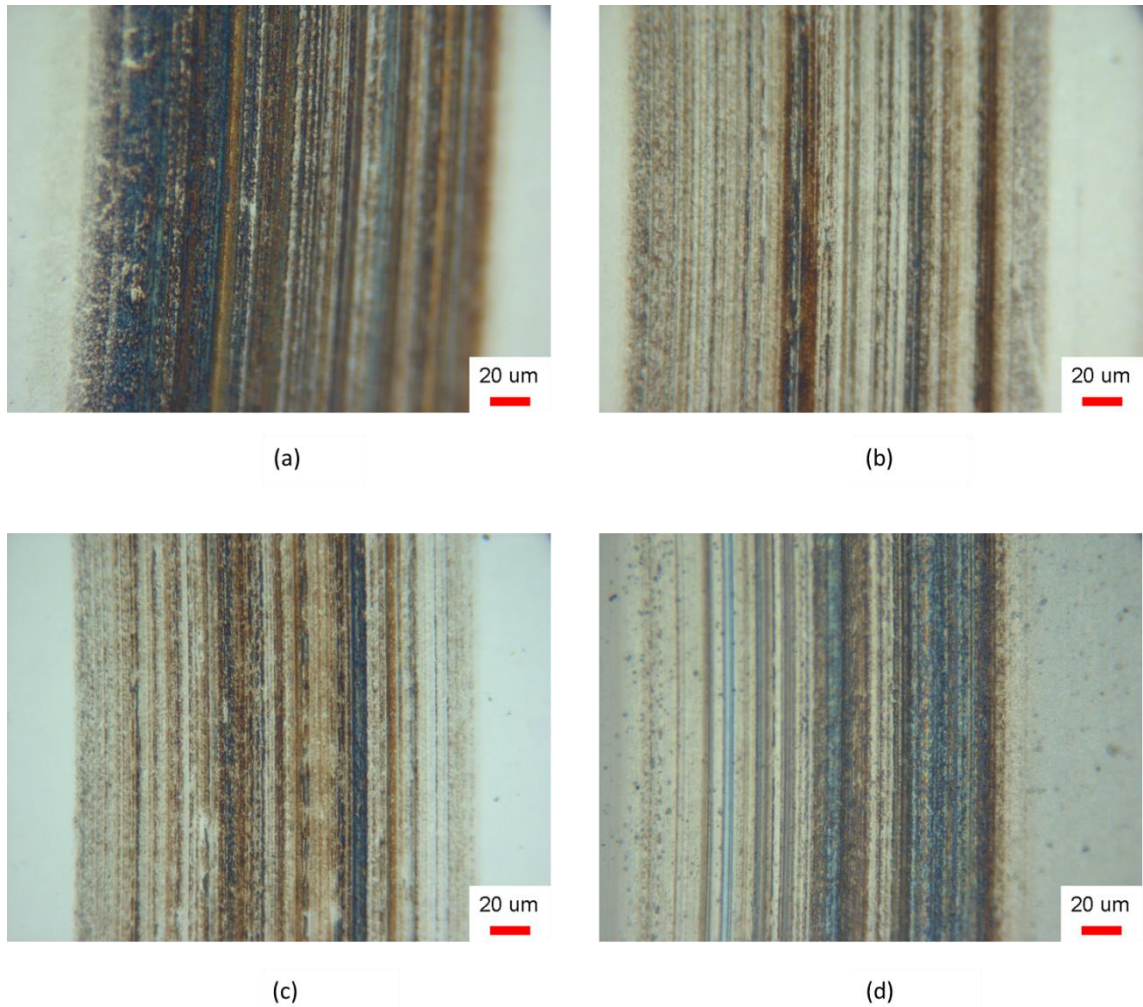


Fig.4 Optical micrographs of wear tracks on the discs lubricated using: (a) pure sunflower oil (b) Sample I (c) Sample II (d) Sample III.

Fig.5 shows the AFM morphologies of the tribo-films (dark area) generated on the wear tracks lubricated with different lubricant samples. Changes on the size and shape of the tribo-films were discovered when different lubricants were used. The tribo-films generated on the worn surface lubricated with pure sunflower oil, as shown in Figs.5(a-b), have a relatively big fragment size, the measured thickness of the tribo-films was around 800nm. When sample I was used as the lubricant as shown in Figs.5(c-d), the tribo-films discovered on the worn surface established a reduced fragment size, small patchy pieces of tribo-films with irregular and stretched shapes were found scattering over the worn surface. The thickness of the measured tribo-films was around 230nm. As shown in Figs.5(c-d), more homogenous and evenly spread tribo-films were found when sample II was employed as the lubricant. The tribo-films have a thickness of around 280nm, which is similar to the one generated by

sample I. The tribo-films generated by sample III, as shown in Figs.5(e-f), also have considerably bigger fragment size compared with the tribo-films generated by sample I and II. The thickness of the tribo-films around 360nm was recorded. It is evident that, to the tribo-films generated by sunflower oil containing ZBUP, the growths of the fragment size and thickness of the tribo-films were consistent with an increase of the zinc borate additive concentration in the lubricant base oil.

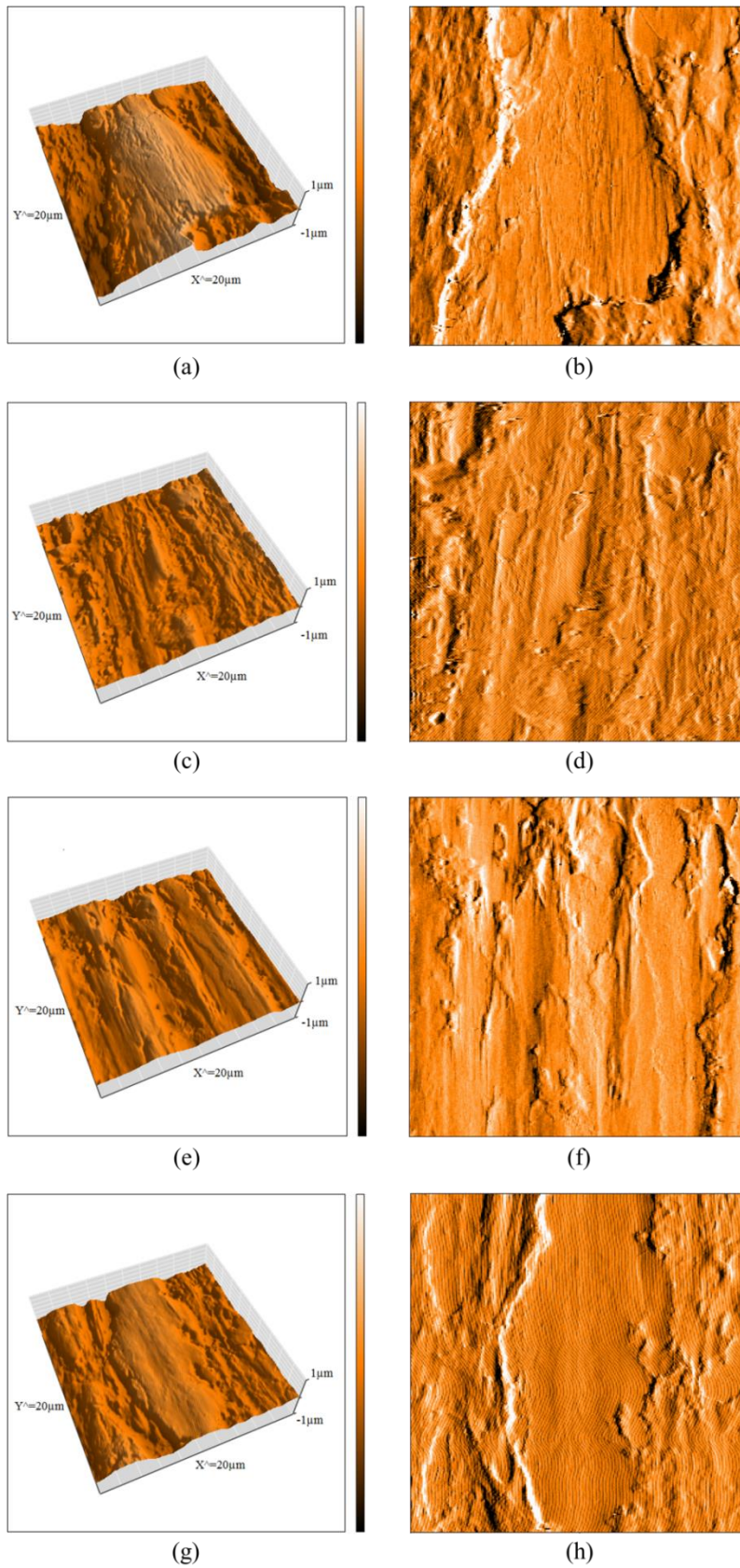


Fig.5 AFM surface topographic images of the worn surfaces lubricated by different samples: (a-b) pure sunflower oil, (c-d) sample I (e-f) sample II, (g-h) sample III.

Mechanical Properties of the Worn Surface

The nano-indentation tests were carried out at randomly selected positions in the bright (substrate) and dark (tribo-films) areas on a wear track. Corresponding load–depth curves of samples lubricated in sunflower oil with different concentrations of ZBUP are illustrated in Figs.6-9. Mechanical properties of the worn surface on the tested discs can be derived from further analysis of the load–depth curves. Hardness, in this case, was calculated from the results achieved. Hardness is reflected by the indentation depth when a fixed load applied. A higher value of indentation depth suggests a softer surface. As displayed previously in Fig.4, the surface properties of the wear track are not homogenous across its width span. In all experiments, indents on the substrate, as shown in Figs.6-9, have smaller indentation depth (Group **A**) than the indents performed on the tribo-films (Group **B**). Compared with load–depth curves of the surfaces lubricated by pure sunflower oil (Fig.9) and sample III (Fig.8), load–depth curves of the surface lubricated by sample I (Fig.6) and II (Fig.7) are less scattered. The fluctuation in local mechanical properties could be used to reflect the homogeneity of a worn surface on a contact track, since a good lubricant may reduce the variation or fluctuation in local mechanical properties and retain a relatively homogeneous worn surface [14]. The average hardness of both the substrate and tribo-films of all the samples are presented in Fig.10. Substrate and tribo-films on the worn surface have higher hardness values when sunflower oil containing ZBUP was used as the lubricant than those when pure sunflower oil was employed. As shown in Fig.10, the hardness of the substrate on the worn surface lubricated in the sunflower oil with ZBUP is relatively consistent. Compared with the result in pure sunflower oil, an average increase of 26% in hardness was observed with the application of zinc borate additive. It is suggested that the hardness increase of the substrate is the result from the tribo-chemical reaction and the iron boride generated on worn surface substrate may effectively influence its mechanical properties [9, 11, 15]. The hardness measurement of the tribo-films, however, did not show the similar consistency. When ZBUP was applied, the hardness of the tribo-films reduced with an increase of zinc borate additive contents. The tribo-films generated in sample I demonstrated the highest hardness of 9.3GPa, which was followed by 7.3GPa, the hardness of the tribo-films generated by sample II. Tribo-films generated in pure sunflower oil held the lowest hardness of 5.5GPa, which was close to 5.7GPa obtained with sample III.

A variation of the hardness values of the tribo-films was observed when different lubricants were used for the tests. Based on the morphology study of the worn surfaces, it is suggested

that the thickness and continuity of the tribo-films are responsible for such variations. It is clear that tribo-films generated on the worn surface have a lower hardness than that on the substrate. Compared with the thicker tribo-films generated in pure sunflower oil and sample III, the hardness values of the tribo-films generated in sample I and II are more likely to be interfered by the high hardness of the substrate due to their thin thickness. Therefore, a reduction of the measured hardness values of the tribo-films that associated with an increase of tribo-films' thickness can be observed.

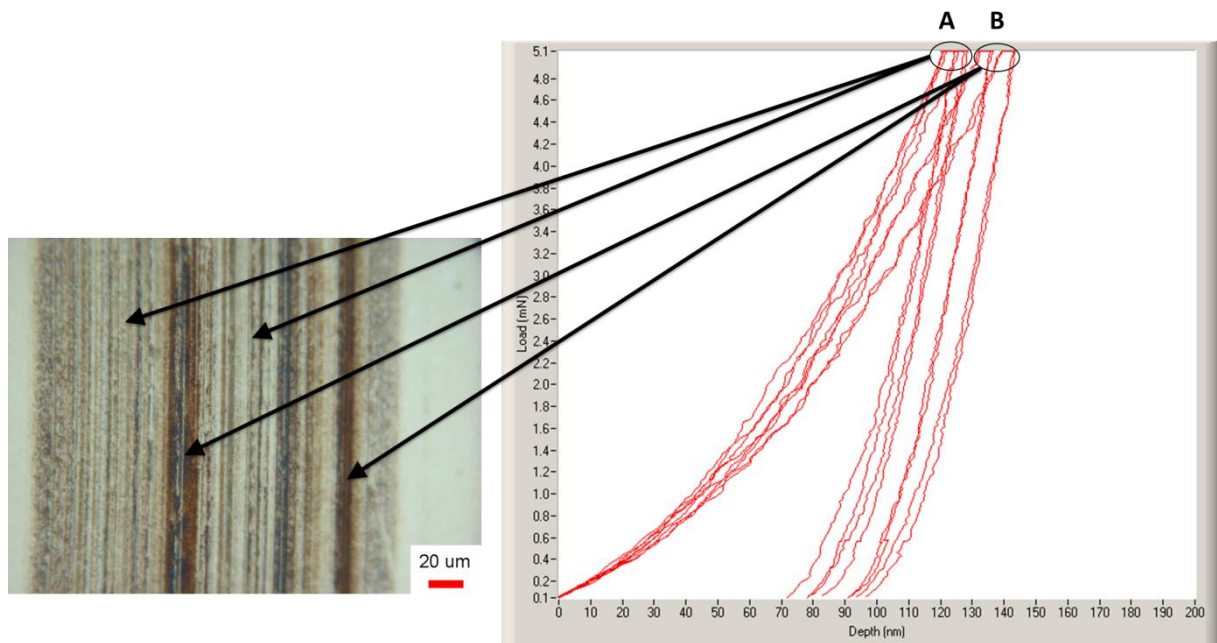


Fig.6 Typical Load depth curves of surface lubricated in sample I (with a maximum applied load of 5mN).

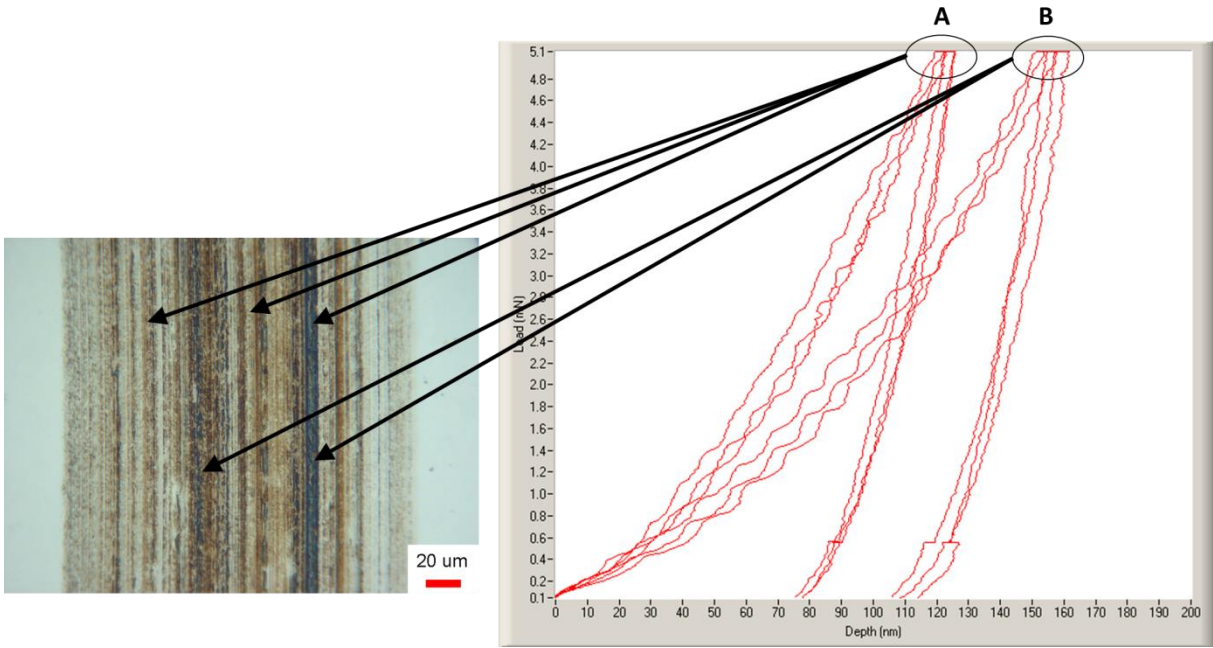


Fig.7 Typical Load depth curves of surface lubricated in sample II (with a maximum applied load of 5mN).

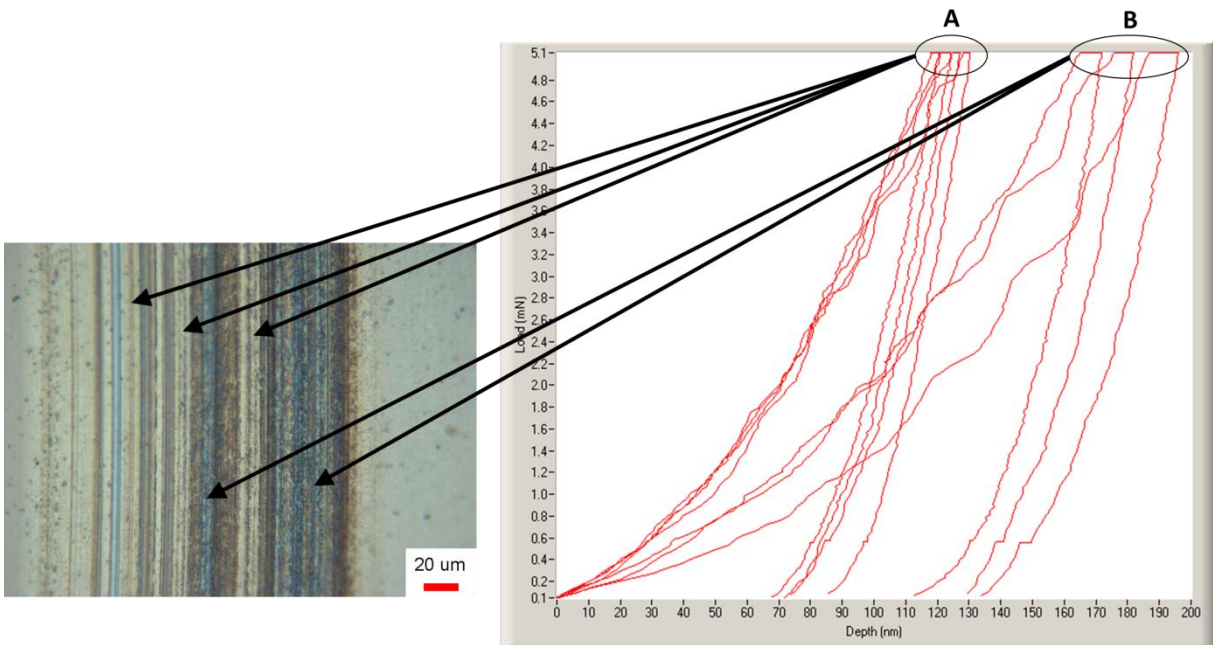


Fig.8 Typical Load depth curves of surface lubricated sample III (with a maximum applied load of 5mN).

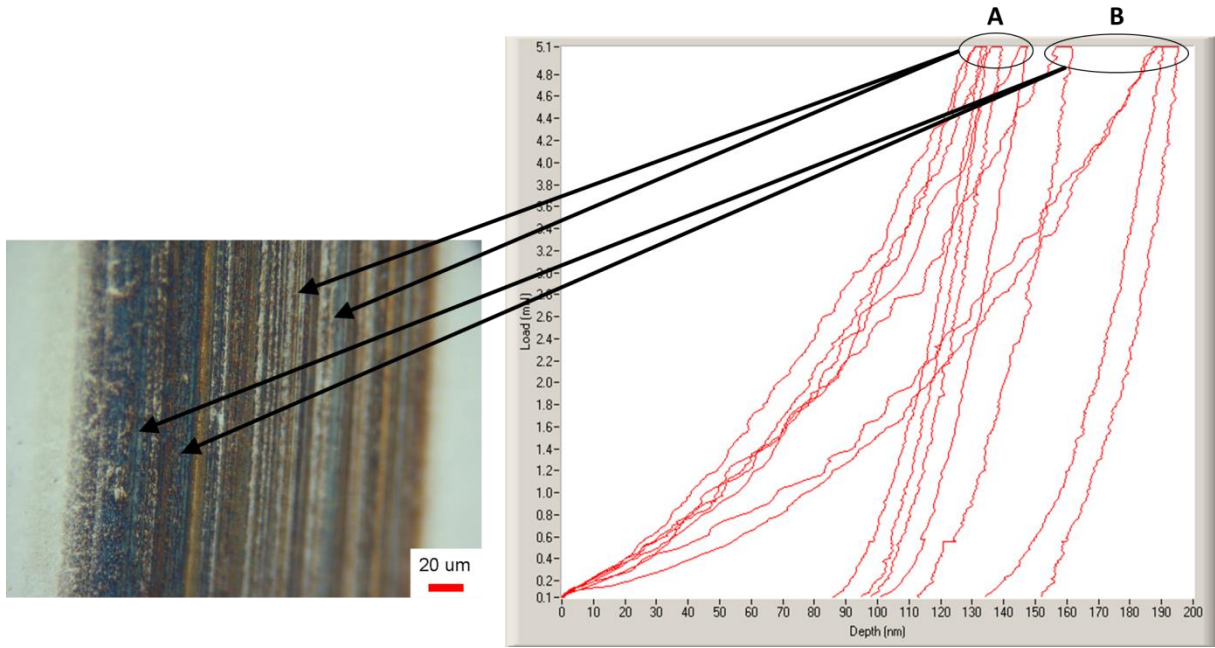


Fig.9 Typical Load depth curves of surface lubricated using pure sunflower oil (with a maximum applied load of 5mN).

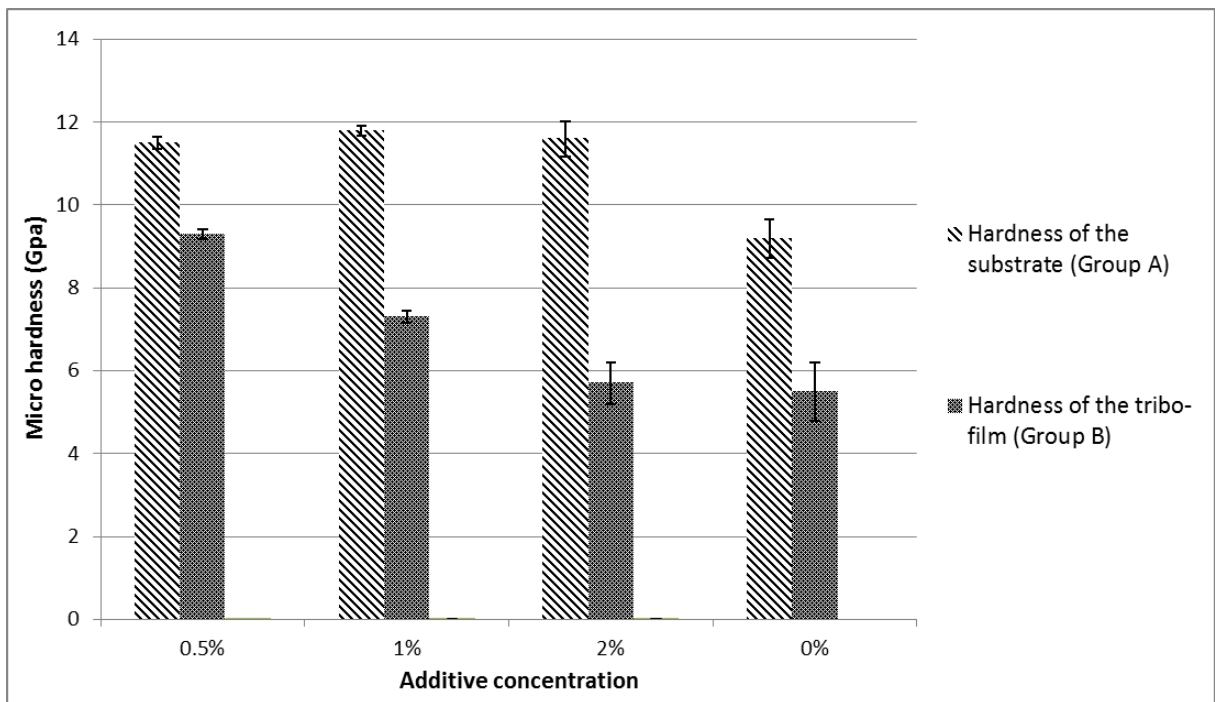
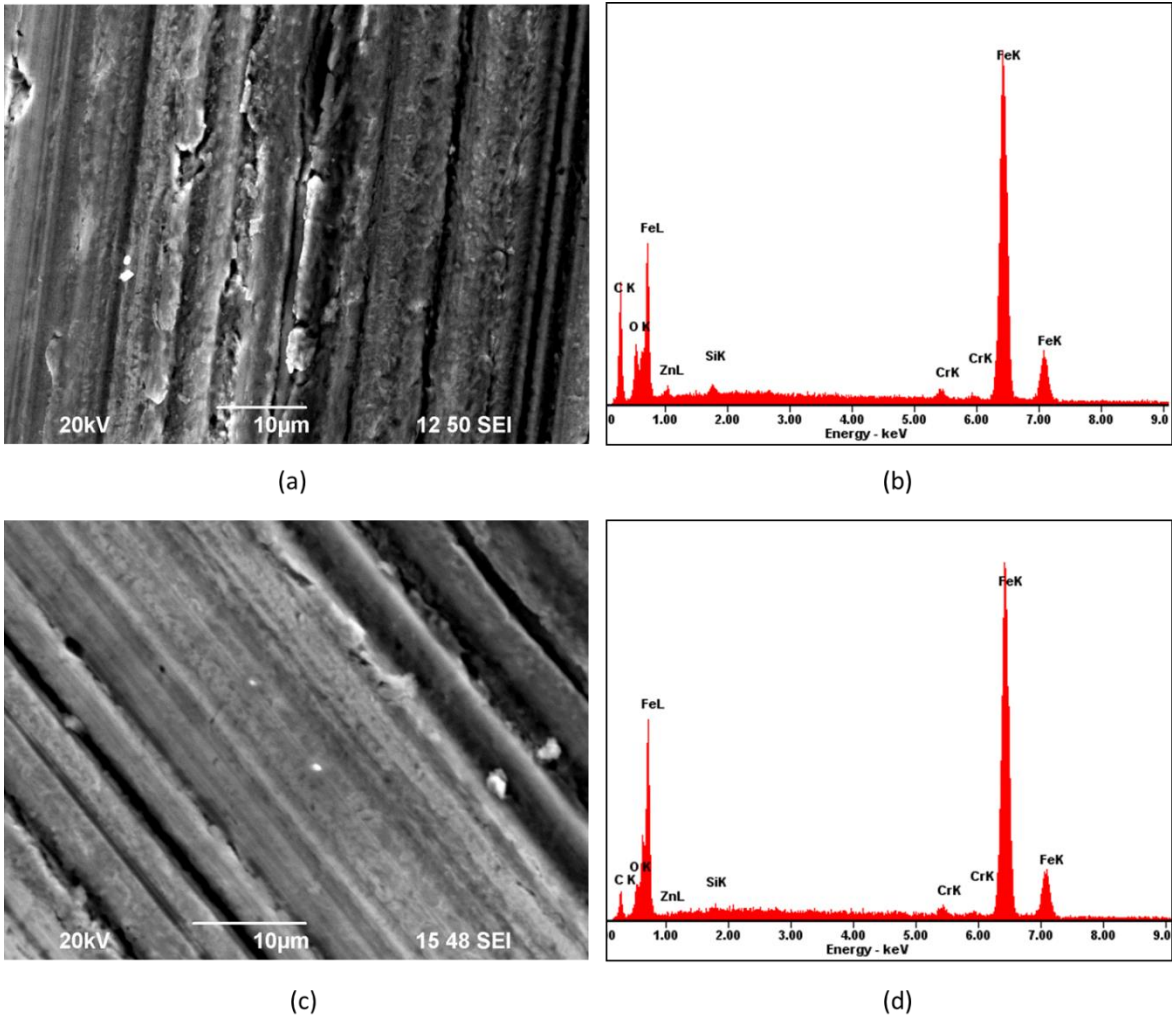


Fig.10. Surface hardness on the wear tracks of discs lubricated in sunflower oil with different additive concentrations (with a maximum applied load of 5mN).

SEM and EDS Analysis

The wear tracks on the discs were analysed under a scanning electron microscope (SEM) equipped with energy dispersive X ray spectroscopy (EDS). Typical SEM images and EDS analyses of the worn surface lubricated in sample III are shown in Fig.11 since the highest contrast of the dark (tribo-films) and bright (substrate) areas were observed. The tribo-films displayed in Fig.11(a) have a complex topography which make a good contrast with the substrate displayed in Fig.11(c) with smoother surface texture. The EDS patterns of the tribo-films and substrate are illustrated in Fig.11(b) and Fig.11(d) respectively and the associated quantified elemental analysis results are also given in Table 2. It is well understood that it is generally difficult to achieve an accurate Boron quantity directly using EDS due to its low atom weight, therefore Boron quantity is not given in the table. It can be seen that a great increase of content of Carbon, Oxygen and Zinc was found on the tribo-films compared with that on the substrate. The similar phenomenon was found on the wear tracks tested in all other lubricant samples with ZBUP. It is suggested that the Carbon is mainly contributed by decomposed lubricant base oil (sunflower oil). The Oxygen is possibly derived from zinc borate additive and metal oxides. The Zinc is also attributed to the ZBUP employed as the lubricant additive in sunflower oil. These results suggest that tribo-chemical reactions may have taken place during the sliding process due to the local high contact pressure and flash temperature caused by the collision and rupture of the asperities between the mating surfaces. Consequently the tribo-films appeared in dark colour were formed on the worn surface, and ZBUP is proven to be an important ingredient of the tribo-film formation.



**Fig.11. SEM micrographs and EDS patterns of worn surfaces lubricated in sample III:
(a-b) dark area of the wear track (c-d) bright area of the wear track.**

Table 2 EDS element analysis of the worn surface lubricated by sample III.

Element composition (wt%)	Bright area	Dark area
<i>C</i>	11.21	29.65
<i>O</i>	3.57	5.48
<i>Zn</i>	0.39	2.86
<i>Si</i>	0.45	0.58
<i>Cr</i>	1.22	0.95
<i>Fe</i>	83.16	60.49

DISCUSSION

Experimental data from the current study have demonstrated the excellent tribological properties of ZBUP employed as a lubricant additive in sunflower oil. Under a friction force, ZBUP is firstly entrapped and then deposited on to the contact interfaces due to shear effect. The third body effect of the ultrafine particles between two contact surfaces can reduce direct metal contact and consequently adhesion [16]. A reduction of adhesion is directly responsible for the reduction of friction and wear, especially at the beginning of the experiment when both friction and wear caused by adhesion were dominant. When ploughing and debris are generated at the contact interface, ZBUPs entrapped in the contact areas could deliver mechanical filling and polishing effects on the rubbed surface and reduce the mechanical lockup between the contact surfaces.

As the sliding continues, it is clear that tribo-chemical reactions took place due to the local high contact pressure and flash temperature caused by the collision and rupture of the asperities between the mating surfaces. Tribo-films appeared in dark colour were then generated on the worn surface, which have a lower hardness than that on the substrate material. It is evident that the properties of the tribo-films play an important role in the tribological performance. The outstanding friction reduction and anti-wear performances were observed when sunflower oil with 0.5% and 1% zinc borate powder was used as the lubricants, accompanied with the thin and patchy tribo-films with small coverage on the worn surface. The friction reduction performance of the two lubricants can be attributed to the diboron trioxide and iron boride formed by tribo-chemical reactions [7, 9, 15, 17-18]. The observed anti-wear performance can be associated with the high hardness of the substrate and the formation of tribo-films [19]. It is well understood that at the initial stage of sliding, wear occurs as the fatigue fracture of the deformed original asperities. This is then followed by a regime in which the wear loss is dominated by material delamination. For a homogeneous material the dislocations pile up beneath the surface layer, subsequently subsurface cracks nucleation and propagation take place when the critical shear stress is reached [20]. When the cracks reach a critical length and finally propagate to the surface, delamination (wear loss) begins. When a thin layer of tribo-film with low hardness and reduced modulus is generated on the hard substrate, dislocations will pile up at the interface between the tribo-film and the substrate. As the sliding continues, these dislocations escape through the surface of the tribo-film due to its small thickness [21]. As a result, the wear of material protected by tribo-film will be remarkably reduced or delayed. For the same reason, with an increase of additive

concentration, the anti-wear performance of the lubricants containing ZBUP was further improved due to the formation of more healthy and complete tribo-films. Interestingly, an increase of additive concentration demonstrated different effects on the friction reduction performance. A slight rise on friction coefficient was detected after the additive concentration was increased from 0.5% to 1%. When the additive concentration was further raised to 2%, the ability of friction reduction of the lubricant was weakened considerably. The possible reason for this is the intensified plastic deformation and mechanical lockup promoted by the thickened and better spread tribo-films. As a result of plastic flow, soft tribo-films easily pile up in front of the asperities and cause more resistance for manoeuvre.

Without ZBUP, the pure sunflower oil demonstrated the highest friction coefficient and the worst anti-wear behaviour. The morphology of the tribo-films and low hardness of the substrate could be responsible for the bad performance. The complex surface topography introduced by very thick tribo-films may also cause a barrier effect which leads to local breakage of oil film [22-23].

CONCLUSIONS

The tribological properties of ZBUPs employed as a lubricant additive in sunflower oil was investigated in this paper. Base on the above results and discussion, the follow conclusions can be drawn:

- ZBUP with a particle size of 500-800nm has demonstrated excellent friction reduction and antiwear property as a lubricant additive in sunflower oil. The most noticeable tribological improvement was observed when 0.5% ZBUP was applied, where more than 14% reduction on friction coefficient and 10% reduction on WSDs were found respectively.
- The outstanding tribological properties of ZBUP as lubricant additive in sunflower oil can be attributed to the increased hardness of the substrate and the formation of tribo-films due to the tribo-chemical reactions occurred on the worn surfaces. These tribo-films with content of Fe, O, C, Zn, and B elements have a smaller hardness than the substrate material.
- The changes on the size and profile of the tribo-films were discovered when the different lubricant samples were employed. The physical and chemical properties of the tribo-films play an important role in the tribological behaviour.

- The promising results from this study have demonstrated the possibility to provide an environmentally friendly lubricant using ZBUP as a lubricant additive in a vegetable based lubricant.

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