



# Investigation of the Interaction, Rheological and Tribological Properties of Bis(pinacolato)diboron with Lithium Grease

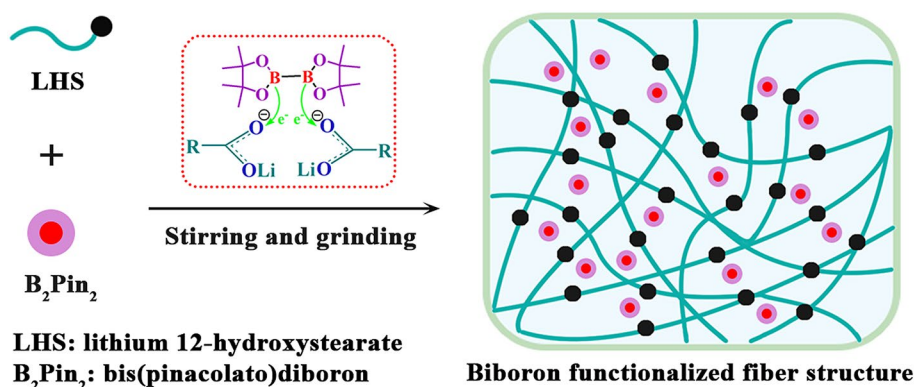
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## Abstract

In this article, Lewis acid–base complex of lithium 12-hydroxystearate (LHS) with diboron compound is formed by the introduction of bis(pinacolato)diboron ( $B_2Pin_2$ ) into lithium grease. The interaction between Lewis acid  $B_2Pin_2$  and Lewis base  $RCO_2^-$  of LHS is characterized by various techniques. Moreover, the rheological and tribological behaviors of the base grease are evaluated at low and moderate temperature. The results indicate that the addition of  $B_2Pin_2$  can noticeably enhance the rheological property of the base grease because the formation of Lewis acid–base complex is beneficial for improving the soap fiber structure strength, and  $B_2Pin_2$  could also help reduce the friction and wear of the grease during the sliding process, which likely owing to the boundary lubrication film generated by  $B_2Pin_2$  adsorption on the rubbing surface and tribochemical reaction between borate esters and steel surfaces. The improvement of mechanical stability and tribological properties is beneficial to increasing the grease service life.

## Graphical Abstract



**Keywords** Lithium grease · Diboron compound · Rheological · Friction and wear

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## 1 Introduction

Bearings are crucial tribological components of many types of machinery. From a small supermarket shopping trolley to huge power plants, a great number of light-duty as well as industrial equipment could not function without the use of bearings in some form. According to a recent study, at least 80% of bearing failures are related to lubrication [1, 2]. Therefore, lubrication plays a critical role in the life and performance of bearings in various applications. Grease is the most widely used lubricating material for bearings [3]. To date, up to 90% of bearings are lubricated with lubricating grease [4, 5]. Grease mainly consists of a base oil, a thickening agent, and multi-functional lubricant additives, with complex microstructure and physical and chemical properties [6]. In addition to the influences of chemical structure of base oil and additives on the lubricating performance of grease, the micro/nano structural of thickener fiber also has a significant influence on the tribological behaviors and mechanical stability of grease, both determine the grease service life, especially at low temperature and low speeds [7–9].

However, there is room to improve the mechanical stability (rheological parameters) and tribological properties of lubricating grease simultaneously, as increasing the rheological properties of grease usually do not prevent friction and wear reduction. For instance, the lubricating grease based on nano-montmorillonite and castor oil with higher thickener agent (commercial organo-montmorillonite Cloisite 20A) achieved higher friction coefficient at different normal loads (10 N and 30 N) [10]. Similar behavior was observed for oleogel-like dispersions containing ethylene vinyl acetate copolymer (EVA) and high-oleic sunflower (HOSO) vegetable oil [11]. The reason is that oleogels with lower viscosity and viscoelastic moduli are easier to generate a protective lubricating film in comparison with oleogels with higher viscosity and viscoelastic moduli [10]. Thanks to the good friction reduction and anti-wear (AW) properties, oxidation inhibition, and low toxicity, intensive research has been done on using borates as high-performance and eco-friendly lubricant additives [12–14], and their results indicated that the good tribological performance of borate is ascribe to the boundary lubrication film formed by borate esters adsorbed on the sliding surfaces and tribochemical reaction between

borate esters and steel surfaces [14]. In recent years, Xu et al. has investigated the Lewis acid–base interaction of tributyl borate (TBB) with lithium 12-hydroxystearate (LHS) and indicated that B atoms of TBB can serve as second-level linking points to enhance the structure strength of soap fiber and the colloidal stability of lubricating grease [15]. Moreover, the addition of TBB could improve the tribological performance of lithium grease at elevated temperature. Compared with TBB, diboron compound could contribute to more complex fiber structure by binding each boron atom to one carbonyl oxygen atom of  $\text{RCO}_2^-$  in LHS, resulting in marked improvement of the structure strength of lithium grease.

Organic diboron species have been used to modified semiconductor oxide materials due to their unique Lewis acid character and reducing ability [16–18]. Facilitated by the Lewis acid–base interaction between B center of the diboron reagent and the surface bridging  $\text{O}_{2c}$  of semiconductor oxidematerials (e.g.,  $\text{TiO}_2$ ,  $\text{ZnO}$ ,  $\text{SnO}_2$ ) nanoparticles, the adsorption of diboron species onto these nanoparticles results in spontaneous electron transfer, leading to various important transformations [16, 17]. This strategy offers an effective approach for functionalizing the fiber structure of lubricating grease. Herein, bis(pinacolato)diboron ( $\text{B}_2\text{Pin}_2$ ) was added into lithium-based grease to react with the carbonyl oxygen atom of  $\text{RCO}_2^-$  in LHS. The Lewis acid–base complex was characterized by several techniques, such as Fourier-transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), and Transmission Electron Microscopy (TEM). Furthermore, the rheological behavior and tribological property of the lubricating grease with different contents of  $\text{B}_2\text{Pin}_2$  were evaluated at temperatures below 70 °C.

## 2 Experimental Section

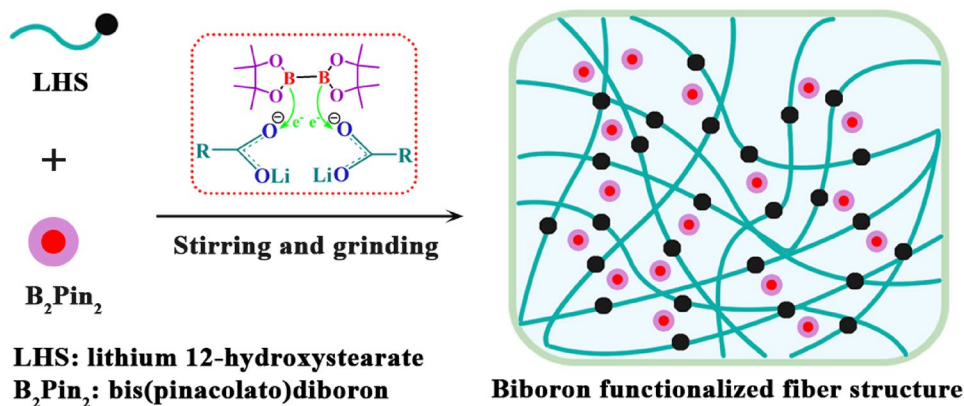
### 2.1 Materials

The lithium-based grease was prepared via the same method reported by Xu et al. [15, 19] and the weight percent of lithium soap was about 12 wt%. Then bis(pinacolato)diboron ( $\text{B}_2\text{Pin}_2$ , purity  $\geq 98\%$ , J&K Scientific Ltd., Beijing, China) was introduced into the base grease. After vigorous mechanical mixing, each sample was ground five times through a

**Table 1** Typical properties of lithium-based grease with varying content of  $\text{B}_2\text{Pin}_2$

| Properties         | 0 wt% | 1 wt% | 3 wt% | 5 wt% | 8 wt% | Test method |
|--------------------|-------|-------|-------|-------|-------|-------------|
| Cone penetration   | 249.5 | 247.1 | 247.4 | 248.2 | 248.8 | ASTM D217   |
| Oil separation (%) | 1.75  | 1.04  | 0.88  | 0.63  | 0.84  | ASTM D6184  |
| Roll stability     | 17.01 | 14.93 | 15.62 | 16.13 | 16.93 | ASTM D1831  |

**Scheme 1** Schematic illustration of the Lewis acid–base interaction of  $B_2Pin_2$  with LHS and the influence of  $B_2Pin_2$  on soap fiber structure of lithium grease



three-roll mill. Typical properties of the tested grease with various contents of  $B_2Pin_2$  are summarized in Table 1.

Thickener fibers in the greases could be obtained by the following method: 10 g grease was thoroughly washed with petroleum ether ( $3 \times 20$  mL) to remove the poly- $\alpha$ -olefin (PAO 10) base oil and washed several times with dichloromethane to remove the unreacted diboron. Then the resulting soap fibers (LHS +  $B_2Pin_2$ ) were dried under vacuum at  $50^\circ\text{C}$  overnight.

## 2.2 Sample Characterization

FTIR of the thickener fiber was performed with an IFS 66v/S using the KBr disk method. TEM images were taken on a JEOL JEM-F200 microscope in TEM mode as well as in annular dark-field (ADF) mode form, operated at 200 kV. XPS of the soap fiber was conducted on a Thermo Scientific K-Alpha spectrometer equipped with a monochromatic Al K $\alpha$  X-ray source (1486.6 eV) operating at 100 W.

Rheological measurements of the tested grease were conducted on an Anton Paar MCR 302 rheometer (Austria) using 1 mm gap for a plate-to-plate geometry (24.985 mm diameter). Oscillatory shear experiments were carried out at a fixed angular frequency ( $\omega = 10$  rad/s) and various shear strains (ranging from 0.01 to 100%) and temperatures (0, 30 and  $60^\circ\text{C}$ ) to evaluate the structure strength of thickener fiber during mechanical aging. Shear measurements were performed at different temperatures (0, 30 and  $60^\circ\text{C}$ ) and shear rates (0.01 and  $100\text{ s}^{-1}$ ) to reveal the evolution of viscosity as a function of time for the lubricating greases.

To investigate the tribological performances of  $B_2Pin_2$  as additives for the lithium-based grease, high contact stress ball-on-disc oscillating reciprocating tribological tests were carried out with an Optimal-SRV-IV tribometer. The AISI 52100 steel balls ( $\phi 10$  mm with hardness of about 626 HV) reciprocated against stationary AISI 52100 steel discs ( $\phi 24.00 \times 7.88$  mm, hardness 700 HV) at a stroke of 1 mm and an oscillation frequency of 25 Hz. The friction coefficient was collected automatically by a computer connected

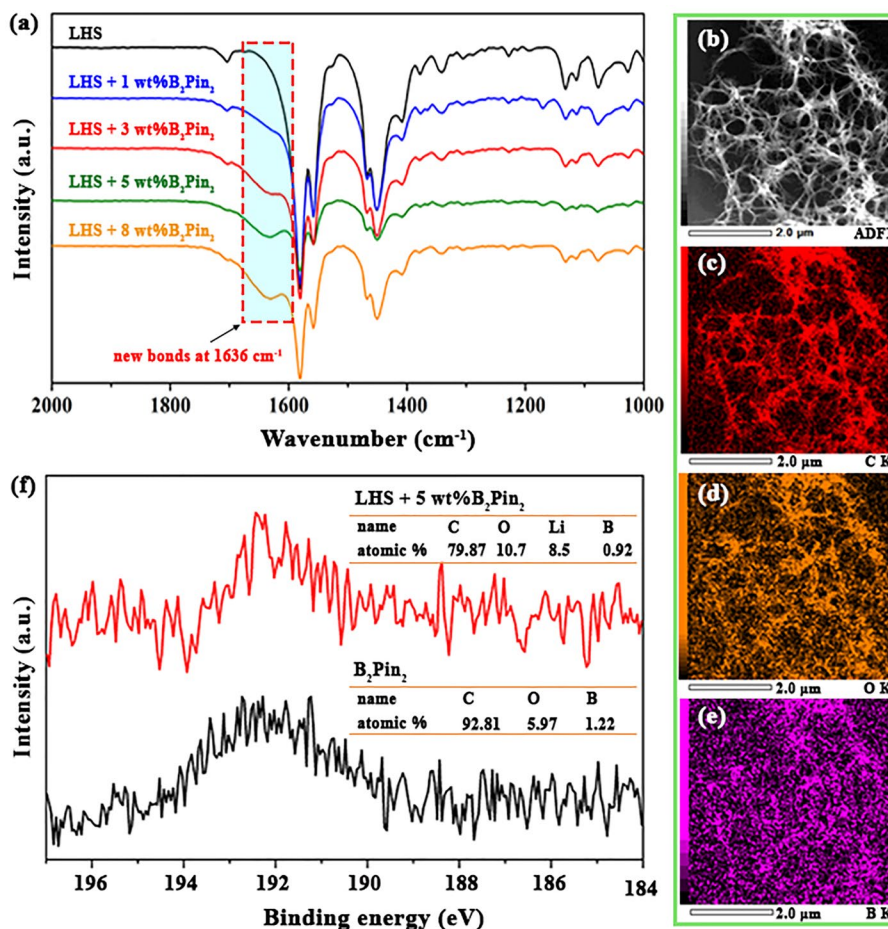
to the SRV tribometer. Wear volumes of the steel disc were measured with a MicroXAM 3D surface profilometer after each tribological test. The surface composition of the wear disc was obtained with XPS. Note that all experiments were repeated at least three times.

## 3 Results and Discussion

### 3.1 Spectroscopic Characterization

The proposed Lewis acid–base interaction of  $B_2Pin_2$  with LHS was shown in Scheme 1. Infrared band assignments of the thickener fibers are reasonable to confirm the formation of Lewis acid base complex (as shown by the red dashed box in Scheme 1). Figure 1a displays the IR spectra between  $800$  and  $2000\text{ cm}^{-1}$  of various soap fibers (LHS, LHS + 1, 3, 5, and 8 wt%  $B_2Pin_2$ ) isolated from the base grease containing different concentration of  $B_2Pin_2$ . Two strong peaks located at  $1580\text{ cm}^{-1}$  and  $1559\text{ cm}^{-1}$  in the spectrum of neat thickener fibers are assigned to  $CO_2^-$  stretching bonds of LHS. When the concentration of  $B_2Pin_2$  is 1 wt%, the IR spectrum of LHS + 1 wt%  $B_2Pin_2$  is similar with that of neat LHS, particularly no obvious  $CO_2^-$  stretching bonds change is observed. When the concentration of  $B_2Pin_2$  reaches over 5 wt%, a new broad peak is clearly seen to be centered at around  $1636\text{ cm}^{-1}$ , which may be due to the formation of Lewis acid–base complex between boron atom in  $B_2Pin_2$  and  $CO_2^-$  of LHS. In this reaction, the boron atom tends to donate an electron pair to the carbonyl oxygen atom resulting in electron-rich  $CO_2^-$  group, so the  $CO_2^-$  stretching band is broadened and shifts to higher frequency with increasing  $B_2Pin_2$  concentration. TEM characterization was performed to explore the influence of  $B_2Pin_2$  on the fibrous microstructure of lithium-based lubricating grease. Although the fiber structures of LHS and LHS + 5 wt%  $B_2Pin_2$  are similar to each other (Fig. 1b, the TEM image of the fiber morphology of LHS is not shown), the corresponding C, O, and B elemental mapping of LHS + 5 wt%  $B_2Pin_2$  (Fig. 1c–e)

**Fig. 1** **a** FTIR spectra of LHS and LHS with different concentration of  $B_2Pin_2$ . **b** TEM image of the fiber morphology of LHS + 5 wt%  $B_2Pin_2$  and the corresponding **c** C, **d** O, and **e** B elemental mapping. **f** B  $1s$  high-resolution XPS spectra for  $B_2Pin_2$  and LHS + 5 wt%  $B_2Pin_2$ ,



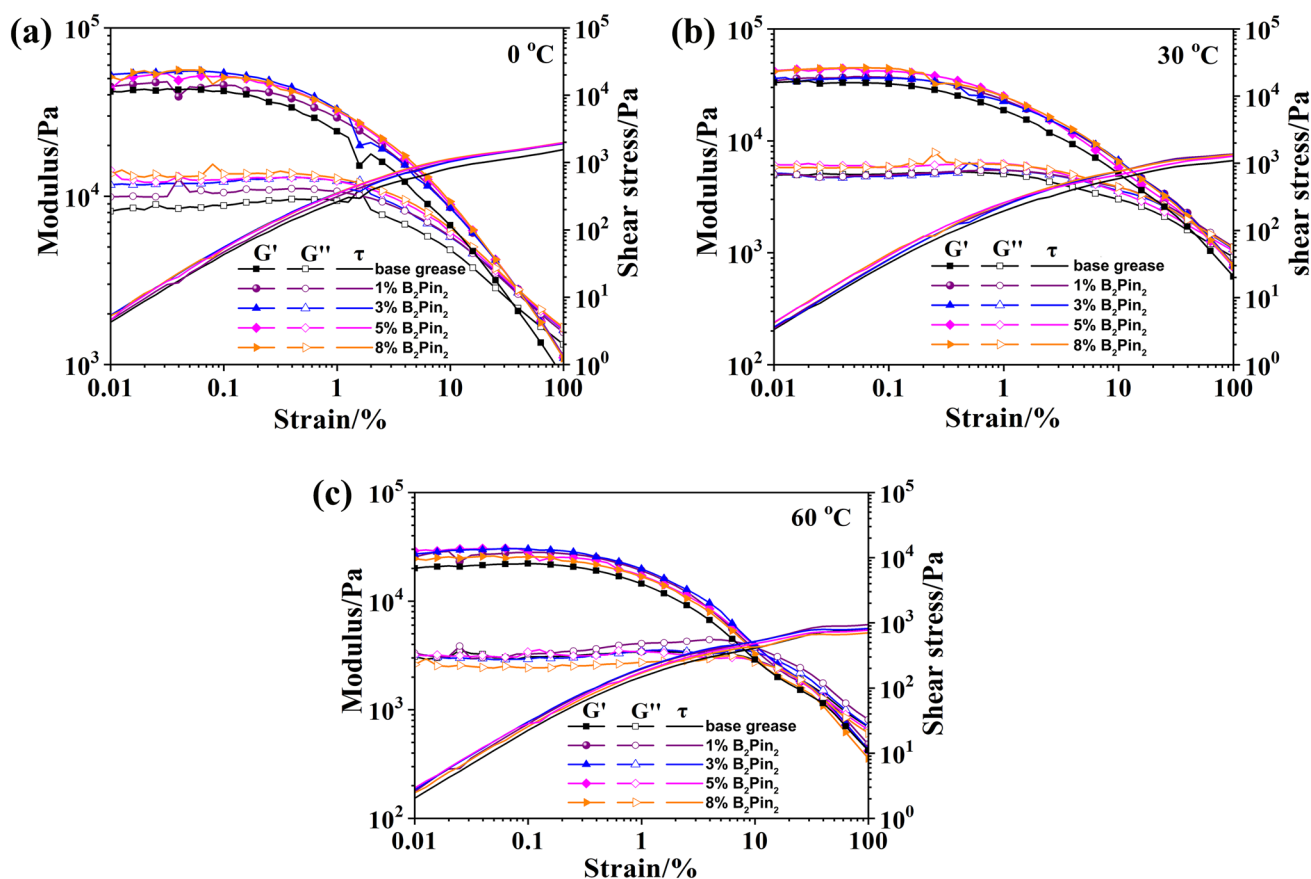
display that B phase mapping distributes over the area of C and O phase mapping, indicating boron containing layer on the surface of soap fiber structure. Additional evidence for the presence of  $B_2Pin_2$  in the soap fiber structure comes from the B  $1s$  XPS spectrum for LHS + 5 wt%  $B_2Pin_2$  (Fig. 1f). As expected, it can be observed that an obvious B  $1s$  peak located at about 192.3 eV, which is similar to that of pure  $B_2Pin_2$ . Quantitative analysis of LHS and LHS + 5 wt%  $B_2Pin_2$  is reported (inset Tables of Fig. 1f) and shows the boron level of 0.92 atomic% in LHS + 5 wt%  $B_2Pin_2$ , confirming the interaction of  $B_2Pin_2$  with LHS to form the Lewis acid–base complex in the lubricating grease.

### 3.2 Rheological Properties of Lithium Grease with Diboron

Shear can result in mechanical breakdown of the thickeners [20]. In order to study the reinforcing effect of  $B_2Pin_2$  on the fibrous structure of lithium grease, the rheological properties of fresh grease with varying  $B_2Pin_2$  content were evaluated by shearing the greases in a rheometer at low and moderate temperature. In oscillatory shear measurements, shear modulus and shear stress versus strain are reported in Fig. 2

for the base grease with 0, 1, 3, 5, and 8 wt%  $B_2Pin_2$  at 0 °C, 30 °C and 60 °C. When the modulus at strains outside the linear viscoelasticity (LVE) regime, all these greases exhibit transitions from  $G'$ -dominant to  $G''$ -dominant behavior, corresponding to the structure transformation from the solid-like to liquid-like, and the transformation point indicates the decomposition point of grease crosslink structure. It is evident that at all shear strains, both storage ( $G'$ ) and loss ( $G''$ ) for the grease with the addition of  $B_2Pin_2$  are markedly higher than for the base grease at different temperatures, particularly for the grease with 5 wt%  $B_2Pin_2$ . Meanwhile, the corresponding shear stress deduced from these measurements is also greater than the base grease. These results mean that the lithium lubricating grease with the addition of  $B_2Pin_2$  is both much stronger and more viscous than the base grease [21], which might be attributed to the formation of Lewis acid–base complex of LHS with  $B_2Pin_2$  and the enhancement of the structure strength of lithium grease. In addition, it is seen that 5 wt%  $B_2Pin_2$  is the optimum concentration to provide significant improvement for grease structure strength. In the shear experiment, evolution of viscosity with time at different shear rate and temperature for the base grease and the grease with 5 wt%  $B_2Pin_2$  is shown





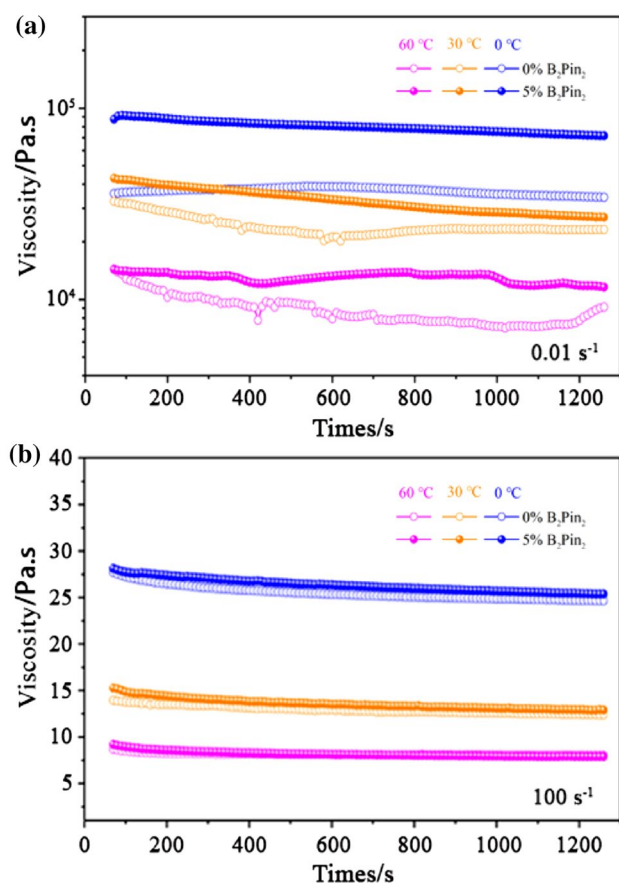
**Fig. 2** Storage modulus ( $G'$ ), loss modulus ( $G''$ ), and shear stress ( $\tau$ ) as a function of shear strain for lithium grease with various concentration of  $B_2Pin_2$  at the temperature of **a** 0, **b** 30, and **c** 60 °C

in Fig. 3. At low value of shear rate ( $0.01 \text{ s}^{-1}$ ), the viscosity of the grease with 5 wt%  $B_2Pin_2$  is substantially higher than the viscosity of the base grease (Fig. 3a), especially for the viscosity at low temperature (0 °C), which manifests a value around two orders of magnitude greater than that of the base grease, further demonstrating that soap fiber structure strength is significantly reinforced by the addition of  $B_2Pin_2$ . However, at high shear rate ( $100 \text{ s}^{-1}$ ), the grease with 5 wt%  $B_2Pin_2$  shows a slightly increase in viscosity compared to the base grease under low and moderate temperature. This can be explained by the fact that the grease thickener will not enter the contact surface but will be pushed to the sides at higher shear rate [22], leading to the formation of lubricating film primarily governed by the base oil [9].

### 3.3 Tribological Properties of Lithium Grease with Diboron

The lubrication performance of the lithium grease additized with  $B_2Pin_2$  were investigated by SRV under variable temperatures, frequencies and loads. Firstly, the friction coefficient of the base grease with 0, 1, 3, 5, and 8 wt%  $B_2Pin_2$

measured at a constant load of 200 N, a fixed frequency of 25 Hz, and different temperature of 30 and 60 °C are displayed in Fig. 4. It is seen that the base grease has a relatively long running-in time (around 0–300 s) with very high friction coefficient ( $> 0.2$ ) at 30 °C (Fig. 4a). However, the addition of  $B_2Pin_2$  can dramatically reduce the running-in period and shows lightly reduce friction coefficient. When the temperature is increased to 60 °C (Fig. 4c), the friction coefficients are noticeably lower than that obtained for the base grease. In particular, 5 wt%  $B_2Pin_2$  is the optimum concentration to reduce the friction coefficient (up to 19%) compared to that of 1 wt% (8%), 3 wt% (14%), and 8 wt% (16%)  $B_2Pin_2$ . Moreover,  $B_2Pin_2$  additized grease also exhibits exceptional AW performance, as shown in Fig. 4b and d. The addition of 5wt%  $B_2Pin_2$  reduced the wear volume of the base grease by 12 times and 3 times at 30 °C and 60 °C, respectively, providing stronger protection against wear than other concentrations of  $B_2Pin_2$ . Figure 5 also show the 3D morphology of the wear scars in Fig. 4b and d. Compared with the base grease, the addition of  $B_2Pin_2$  generated shallower and narrower wear scars, while the wear scars generated by base grease are deeper and wider, so it is significant



**Fig. 3** The evolution of shear viscosity for lithium grease and the grease with 5 wt% B<sub>2</sub>Pin<sub>2</sub> at shear rate of **a** 0.01 s<sup>-1</sup> and **b** 100 s<sup>-1</sup> at different temperatures

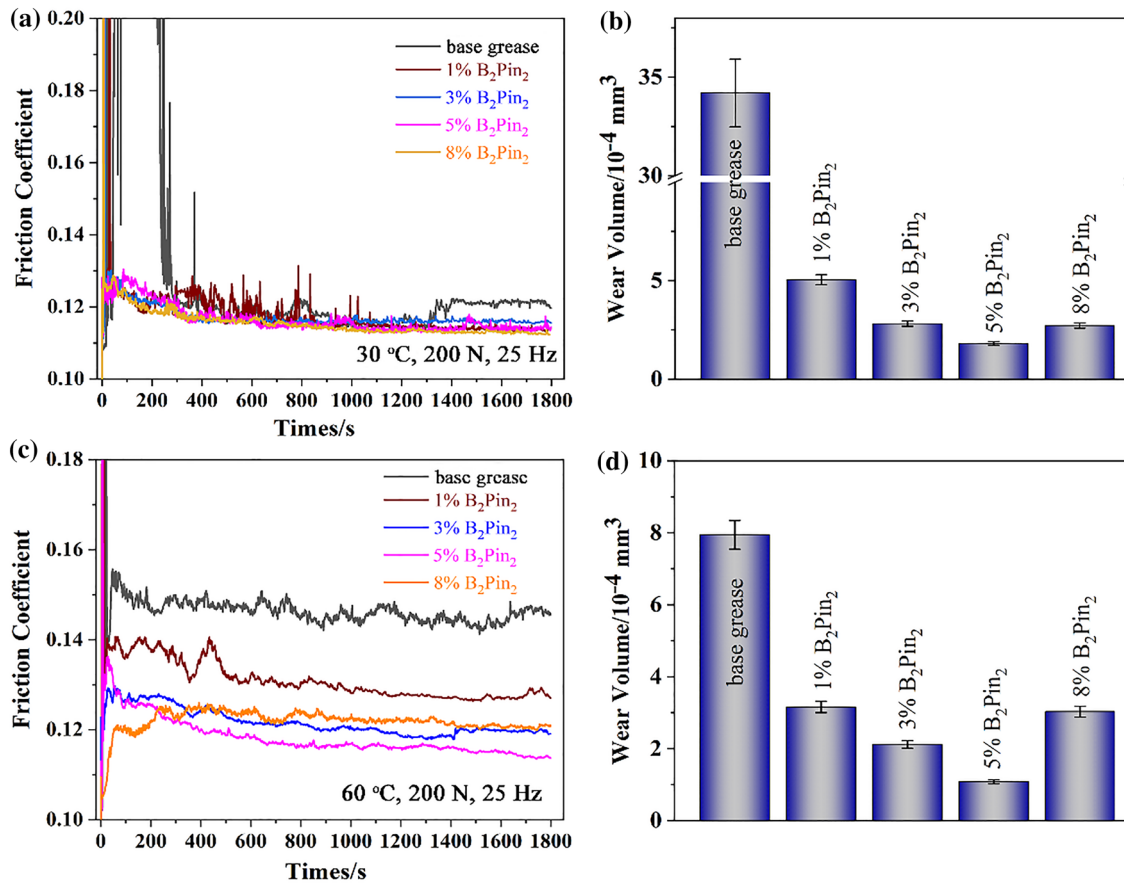
that the use of B<sub>2</sub>Pin<sub>2</sub> prevents the formation of wear scars larger and deeper. The excellent friction and wear reduction properties could probably be ascribed to the boundary lubrication film formed by B<sub>2</sub>Pin<sub>2</sub> adsorbed on the sliding surface and/or tribochemical reaction between borate esters and steel surfaces [12, 14].

The lubricating performance of 5 wt% B<sub>2</sub>Pin<sub>2</sub> added in the grease was further evaluated by changing the reciprocating frequencies and applied loads. As shown in Fig. 6a and b, the addition of 5 wt% B<sub>2</sub>Pin<sub>2</sub> could contribute efficiently to the friction reduction of base grease during frequency

ramp test from 10 up to 70 Hz stepped by 15 Hz at different temperatures, particularly as the frequency below 40 Hz. In addition, Fig. 6c and d display the load-carrying capacity of 5 wt% B<sub>2</sub>Pin<sub>2</sub> additivated grease increased remarkably from 100 and 150 N to 500 N with increase in applied load from 50 to 500 N at 30 °C and 60 °C, respectively. The improvement of anti-shear and load-carrying capacities of lithium grease may be attributed to the reinforcement of the grease structure strength and the formation of boundary lubrication film by the addition of B<sub>2</sub>Pin<sub>2</sub>.

### 3.4 Surface Analysis of Wear Scars Lubricated by Lithium Grease with Diboron

To investigate the friction reduction and AW mechanism of B<sub>2</sub>Pin<sub>2</sub> in lithium grease, high-resolution (HR) XPS spectra of B, O and Fe on the worn surface of the steel discs were obtained. As shown in Fig. 7a, the binding energies of B 1s on the worn scars lubricate by the base grease with the addition B<sub>2</sub>Pin<sub>2</sub> at 30 °C and 60 °C are similar to each other, and the peaks centered at 193.5 eV and 192.0 eV might be ascribed to B–B and B–O binds related to B<sub>2</sub>Pin<sub>2</sub> residue and B<sub>2</sub>O<sub>3</sub> [15, 23]. The O 1s XPS spectra for the wear scars lubricated with the base grease at 30 °C and 60 °C are also very similar, and one of them can be deconvoluted into five peaks, which appear at 529.1 eV, 529.8 eV, 530.8 eV, 531.7 eV and 532.7 eV, are identified to Fe<sub>3</sub>O<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub>, and LHS (Fig. 7b) [23]. After lubricating with the grease additived B<sub>2</sub>Pin<sub>2</sub> at 30 °C and 60 °C (Fig. 7b), the O 1s spectra including three and five peaks, which might be assigned to LHS (530.8 eV, 531.7 eV) and B<sub>2</sub>Pin<sub>2</sub> (532.6 eV) residues, and Fe<sub>2</sub>O<sub>3</sub> (529.8 eV), Fe<sub>3</sub>O<sub>4</sub> (530.1 eV), LHS (530.8 eV, 531.7 eV) and B<sub>2</sub>Pin<sub>2</sub> (532.6 eV) residues, respectively [23, 24]. In addition, the XPS spectra of Fe 2p display four peaks and five peaks when the lubricating grease with B<sub>2</sub>Pin<sub>2</sub> were evaluated at 30 °C and 60 °C (Fig. 7c), these peaks were corresponded to FeO (709), Fe<sub>3</sub>O<sub>4</sub> (709.8 eV), Fe<sub>2</sub>O<sub>3</sub> (710.8 eV and 724 eV), and Fe(OH)O (712 eV) [23, 25], respectively. These results demonstrate that the addition of B<sub>2</sub>Pin<sub>2</sub> in the lithium grease could adsorb on the contact surfaces and generate tribochemical products to form boundary lubrication film at 30 °C and 60 °C. The film contains FeO, Fe<sub>3</sub>O<sub>4</sub>, Fe<sub>2</sub>O<sub>3</sub>, Fe(OH)O, B<sub>2</sub>O<sub>3</sub>, and B<sub>2</sub>Pin<sub>2</sub> residue, which is beneficial to improving the tribological properties of the lithium grease during friction and wear process.



**Fig. 4** a, c Friction coefficient and b, d wear volume of steel discs lubricated by lithium grease and the grease with different concentration of B<sub>2</sub>Pin<sub>2</sub> at a, b 30 and c, d 60 °C (SRV load, 200 N; stroke, 1 mm; frequency, 25 Hz)

## 4 Conclusions

To sum up, the interaction of bis(pinacolato) diboron (B<sub>2</sub>Pin<sub>2</sub>) with lithium 12-hydroxystearate (LHS) in lithium grease was characterized, and the rheological and tribological performance of the grease additized with B<sub>2</sub>Pin<sub>2</sub> was investigated at low and moderate temperatures. The following conclusions can be drawn from the present study:

- (1) The formation of Lewis acid–base complex of LHS with B<sub>2</sub>Pin<sub>2</sub> was confirmed. In this reaction, each boron atom of B<sub>2</sub>Pin<sub>2</sub> tends to donate an electron pair to one carbonyl oxygen atom of RCO<sub>2</sub><sup>-</sup> in LHS, resulting in the combination of boron atom (Lewis acid) with the carbonyl oxygen atom (Lewis base).
- (2) Rheological analysis demonstrates that the combination of B<sub>2</sub>Pin<sub>2</sub> with LHS obviously reinforced the soap fiber structure strength of lithium grease at low temperature and low shear speed, and 5 wt% B<sub>2</sub>Pin<sub>2</sub> is the optimum concentration to provide the significant improvement.
- (3) Tribological experiments clearly show that the addition of 5 wt% B<sub>2</sub>Pin<sub>2</sub> reduced the friction coefficient up to 19% and the wear volume by around 12 times compared with the base grease and enhanced the anti-shear and load-carrying capacities of lithium grease at 30 °C and 60 °C, respectively.
- (4) The excellent tribological behaviors of B<sub>2</sub>Pin<sub>2</sub> in the grease are ascribed to the formation of boundary lubrication film generated by B<sub>2</sub>Pin<sub>2</sub> adsorbed on the rubbing surface and tribochemical reaction between borate esters and steel.

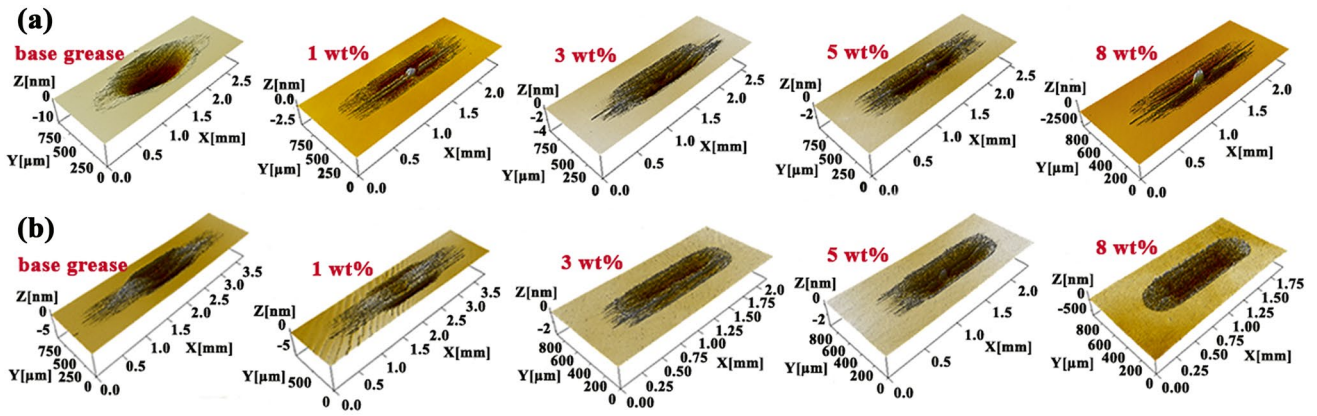


Fig. 5 The 3D optical microscopic images of wear track lubricated by lithium grease and the grease with different concentration of  $B_2Pin_2$  at a 30 and b 60 °C (SRV load, 200 N; stroke, 1 mm; frequency, 25 Hz)

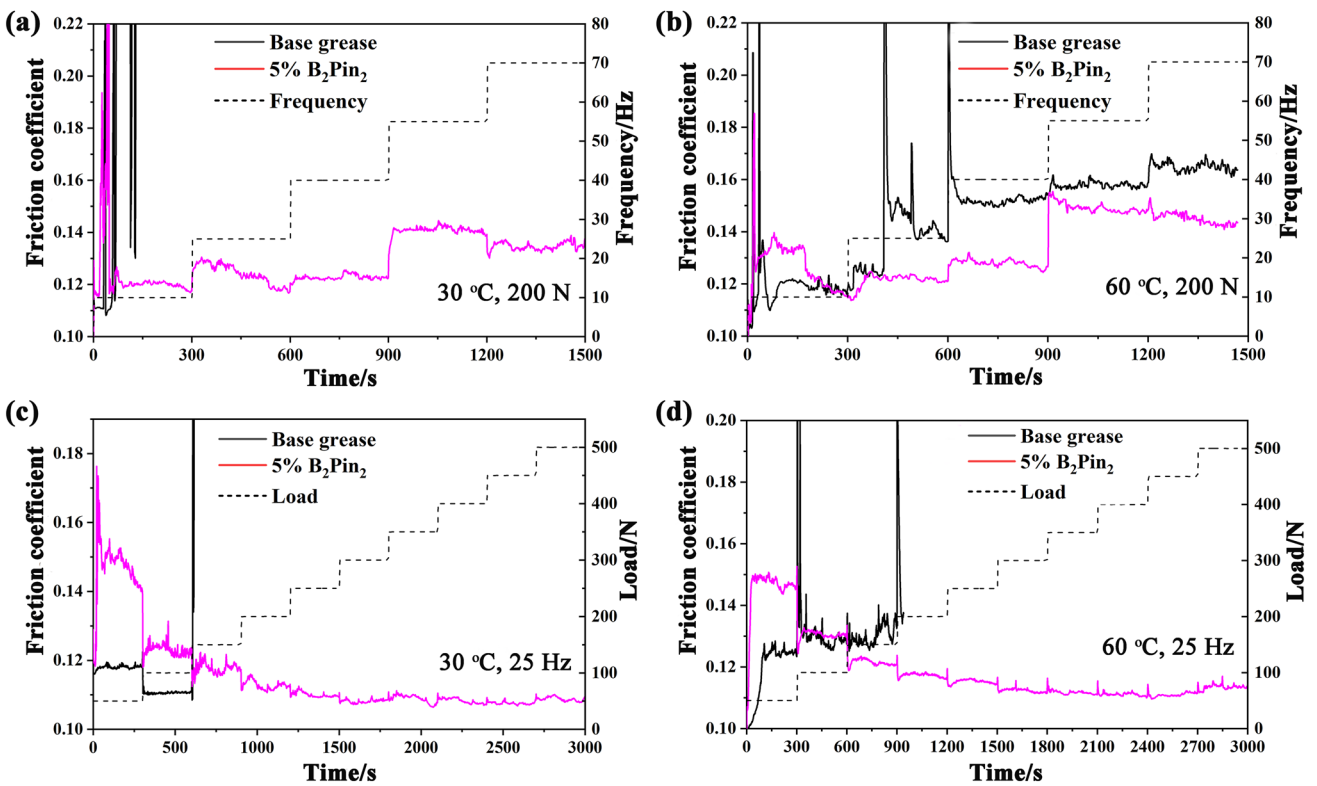
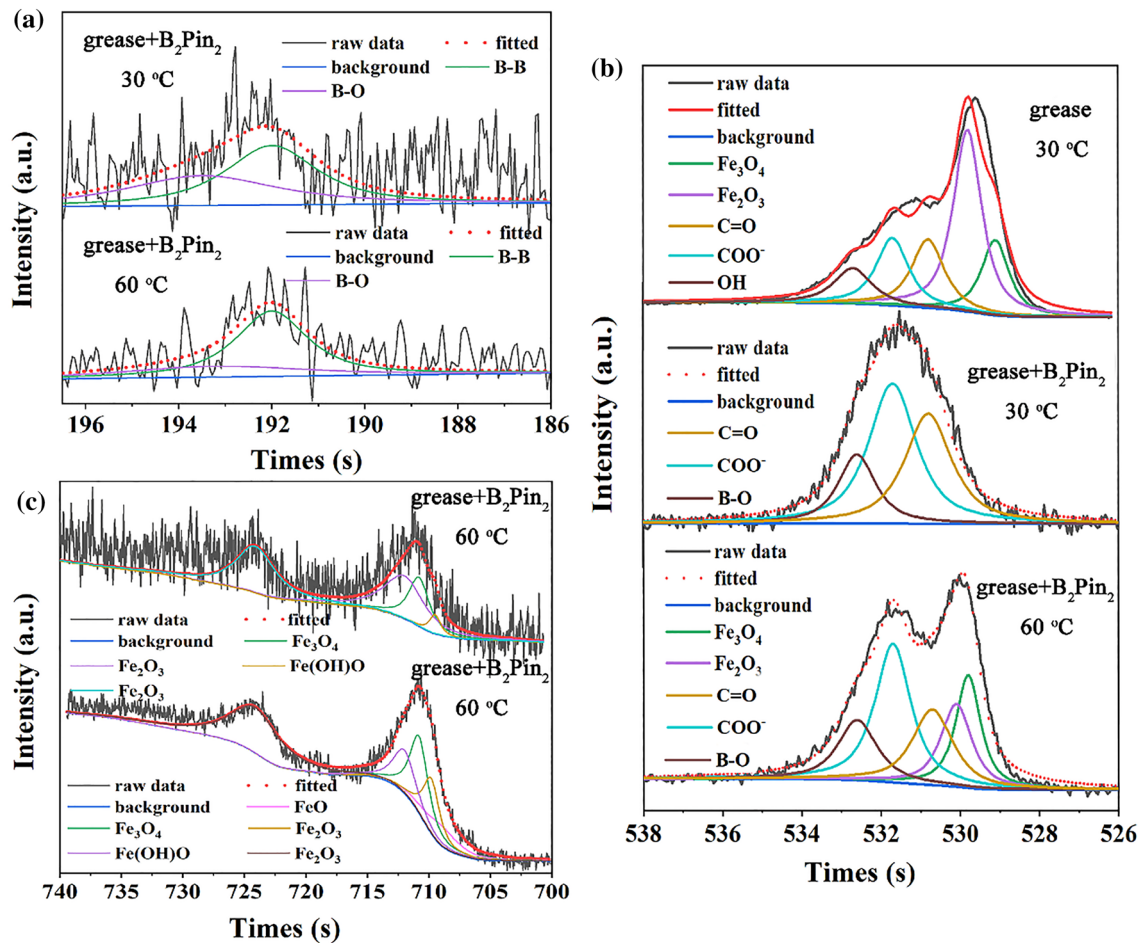


Fig. 6 The variation of friction coefficient as a function of time during a, b frequency load ramp test from 10 to 70 Hz and a c, d load ramp test from 50 to 500 N at a, c 30 and b, d 60 °C (SRV conditions

for frequency test: frequency, 10–70 Hz; load, 200 N; stroke, 1 mm, and for load ramp test: load, 50–500 N; stroke, 1 mm; frequency, 25 Hz)





**Fig. 7** High-resolution XPS spectra of **a** B 1s, **b** O 1s, and **c** Fe 2p for the steel discs lubricated by lithium grease and the grease with 5 wt% B<sub>2</sub>Pin<sub>2</sub> at 30 and 60 °C (SRV load, 200 N; stroke, 1 mm; frequency, 25 Hz)

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**Data Availability** Not applicable.

**Code Availability** Not applicable.

## Declarations

**Conflict of interest** The authors declare no competing financial interest.

**Ethical Approval** Not applicable.

**Consent to Participate** Not applicable.

**Consent for Publication** Not applicable.

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