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*Article*

# The Effect of Temperature on Lubrication Property with MoDTC-Containning Lubricant —Temperature Dependence of Friction Coefficient and Tribofilm Structure—

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## Abstract

The effect of temperature on the lubricity of molybdenum dithiocarbamate (MoDTC) was investigated by a ball on disk tribometer with lubricant oil containing MoDTC and calcium sulfonate (CaSU) at temperatures of 25°C, 40°C, 60°C and 80°C. The behavior of friction coefficient was closely dependent on the oil temperature. Friction coefficient decreased after a certain induction period and became to be constant at steady state. At higher temperature, the induction period became to be shorter, and the slope of decreasing friction coefficient became to be steeper. Friction coefficient at steady state decreased at higher temperature, and was changed reversibly when the oil temperature was changed. Oscillatory change in friction was observed when the oil temperature was changed periodically. Chemical analysis of the tribofilm with X-ray Photoelectron Spectroscopy (XPS) revealed that higher content of Mo(IV) as a component of molybdenum disulfide ($MoS_2$) was observed in the tribofilm formed at higher temperature. Highly oriented $MoS_2$ layer was observed with High Resolution Transmission Electron Microscopy (HR-TEM) in the tribofilm formed at 80°C. It was found that the structure of tribofilm is altered reversibly with oil temperature. A steady state model of tribofilm formation was proposed on the basis of observed results.

## Keywords

molybudenum dithiocarbamate, molybdenum disulfide, tribo chemistry, friction coefficient, tribofilm, X-ray photoelectron spectroscopy, high resolution transmission electron microscopy, temperature dependence, layer structure

## 1 Introduction

A reduction in friction coefficient has been one of the major issues concerning energy conservation as it was reported that positive economic effects that are the equivalent of about 2% of Japan's GNP could be expected by reducing friction and wear [1]. In the field of lubricants, low viscosity base oils can decrease viscous resistance in hydrodynamic lubrication [2,3] and the use of friction modifiers has been an effective solution to decrease friction in boundary lubrication.

Molybdenum dithiocarbamate, MoDTC as a typical friction modifier has been extensively studied and is known to effectively reduce friction by producing molybdenum disulfide, $MoS_2$ on the frictional surface through chemical reaction [4-6]. Yamamoto et al. reported that the production of $MoS_2$ can take place exclusively in frictional contact while only molybdenum trioxide, $MoO_3$ is produced in non-frictional contact [7]. Kano et al. suggested that an engine oil consisting of MoDTC and zinc dialkyldithiophosphate, ZDDP produces a $MoS_2$ compound having "inner-skin-layer" that effectively reduces friction [8].

Some studies on MoDTC, including the reports on the effect of material and coating on friction reducing properties of MoDTC [9-11], the synergetic effects by combining MoDTC with other additives [12-14], and so on, showed that lubricity of MoDTC is greatly affected by the condition and environment of lubrication tests. Although temperature is considered one of the important factors affecting its lubricity, previous studies dealt only with experiments at some different constant temperatures [4,15] but not with those under continuously changing temperature condition.

Therefore, this study aimed to evaluate the effect of temperature on the friction reducing properties of MoDTC as a friction modifier and to identify the temperature-dependent structure of the tribofilm in relation to friction reducing properties. In detail, the correlation between friction coefficient and tribofilm structure during testing at temperatures changing from 25˚C to 80˚C was evaluated. Furthermore, the effect of temperature was discussed by focusing on the dynamic behavior of tribofilm structure relative to the friction coefficient changing with an increase or decrease in oil temperature.

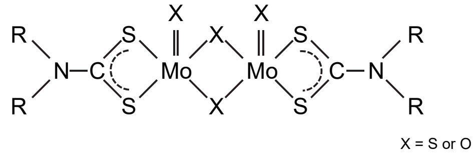
## 2 Experimental

### 2.1 Test oil

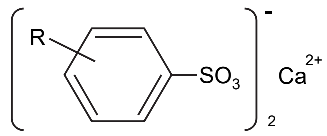
A test oil, polyalphaolefin oil with a kinematic viscosity of 7.94 $mm^2$/sec at 100˚C (PAO8), was prepared by adding commercially available oil-soluble MoDTC of Mo:220 ppm and neutral calcium sulfonate (CaSU) of Ca:120 ppm. Molecular structure of the additives are shown in Fig. 1. MoDTC was used in combination with CaSU to achieve a stable friction coefficient based on previous studies showing that the friction coefficient becomes unstable when MoDTC is used alone and that CaSU or ZDDP promotes MoDTC adsorption on the frictional surface by forming an underlayer leading to a stable friction coefficient [12,16].

### 2.2 Lubrication test

Lubrication tests were carried out using a ball on disk tribometer as shown in Fig. 2. The tribometer consists of an 8 mm diameter-ball and a 30 mm diameter- and 4 mm thickness-disk made of high-carbon chromium bearing steel, SUJ2 (AISI52100). The arithmetic surface roughness in a direction perpendicular to grinding marks is $Ra$=0.2~0.3 μm. The ball and disk were immersed in the test oil. The temperature was controlled by an oil bath connected to a chiller with error tolerance levels of ±0.5˚C and ±2.5˚C for constant temperature and changing temperature conditions, respectively. In most previous studies, the amount of test oil was probably too small to allow the oil temperature to be stable and properly controlled when the surface was exposed to a significant temperature rise caused by frictional heat. For this reason, our experiments were performed with a large amount of lubricating oil (about 400 ml) capable of providing enough heat capacity for heat generation in frictional contact so that a proper temperature control could be achieved. The oil temperature was measured about 1 cm away from the lubricated contact during testing, at which no temperature rise due to frictional heat was observed.




(a) Molybdenum dialkyldithiocarbamate (MoDTC)



(b) Calcium alkylbenzene sulfonate (CaSU)

Fig. 1 Chemical structures of MoDTC and CaSU

Table 1 summarizes test conditions. A load of 17 N (max.

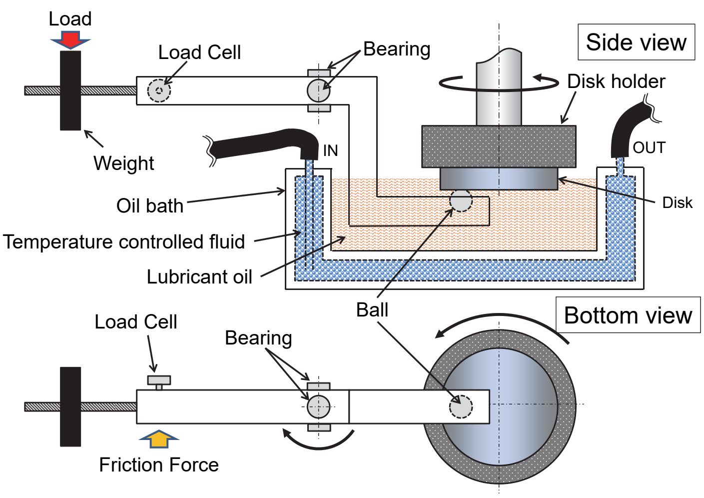



Fig. 2 Schematic illustration of ball-on-disk test rig

Table 1 Conditions for ball on disk test

| | Method 1 | Method 2 | Method 3 |
|---|---|---|---|
| Load, N | 17 | 17 | 17 |
| Sliding speed, m/s | 0.31 | 0.31 | 0.31 |
| Temperature, ˚C | RT (ca.25˚C)<br>40˚C<br>60˚C<br>80˚C | 60˚C → 80˚C → 60˚C → 40˚C | 40˚C → 80˚C<br>80˚C → 40˚C |

Hertzian contact pressure: 1.41 GPa) and a sliding speed of 0.31 m/s were applied to the disk and ball under three different temperature conditions as described below.

Test method 1 is characterized by four different constant temperatures: room temperature (about 25˚C), 40˚C, 60˚C and 80˚C.

Under test methods 2 and 3, the temperature was changed in a different sequence. As for test method 2, the test was started at 60˚C and the temperature was increased to 80˚C, brought back to 60˚C and decreased to 40˚C. All these temperature changes were made after confirming that the friction coefficient became stable. Under test method 3, the test was started at 40˚C or 80˚C and the temperature was increased to 80˚C or decreased to 40˚C after maintaining the initial temperature for 30 min. Then, the temperature was maintained for another 30 min to finish the test.

2.3 Lubrication test

High Resolution Transmission Electron Microscopy, HR-TEM and X-ray Photoelectron Spectroscopy, XPS were performed to examine the structure and composition of tribofilm.

A thin layer of approx. 10 μm width, 7 μm depth and 100 nm thickness was cut out of the tribofilm formed on the wear track of the tested ball perpendicular to the direction of friction using Focused Ion Beam System, as a specimen for HR-TEM observation and elemental analyses by Electron Energy Loss Spectroscopy, EELS as well as Energy Dispersive X-ray Spectrometry, EDS. In the qualitative analysis of S and Mo, S-L and Mo-K rays were used for EELS and EDS analyses, respectively.

XPS analysis was performed with monochlomated Al K$\alpha$ X-ray source for the area within 200 μmφ of the center of the wear scars. Mo3d curve fitting was performed by setting a binding energy difference between 3d5/2 and 3d3/2 as 3.13 eV and a peak area ratio as 3d5/2:3d3/2=3:2.

## 3 Results

3.1 Lubrication test : method 1 – constant temperature

Lubrication tests were carried out at four different constant temperatures (room temperature (about 25˚C), 40˚C, 60˚C and 80˚C) to study the temperature dependence of friction coefficient. Figure 3 shows friction coefficients as a function of temperature for the MoDTC-containing lubricant. At room temperature of about 25˚C, the friction coefficient gradually decreases similarly to its base oil alone suggesting that it decreases due solely to smoothing of the contact surface without any effects from the additive. At 40˚C or above, it decreases in three steps: (1) initial decline, (2) drastic drop and (3) steady state. Although the friction coefficient curves at 40˚C become fluctuant as time advances, those at 60˚C or above are relatively more reproducible showing nearly equal values for same temperature. The friction coefficients in the temperature range of this study were found to be lower at higher temperature being temperature dependent at steady state. As the test temperature increases, initial decline period before a drastic drop in friction coefficient became shorter and the friction coefficient curve became more steep, i.e. the rate of decline of friction coefficient became higher in drastic drop period.

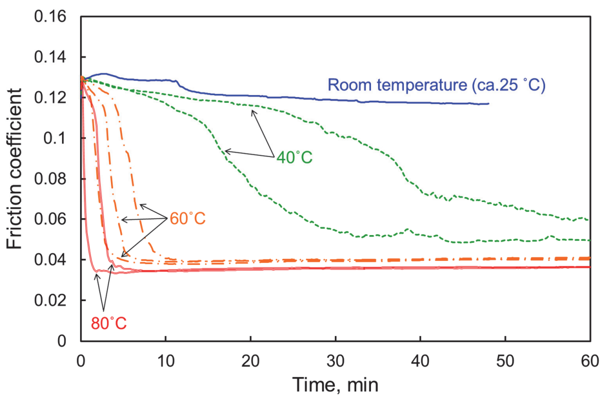



Fig. 3 Friction coefficient as a function of temperature for MoDTC-containing lubricant

3.2 Lubrication test : method 2 – changing temperature

The results of the lubrication tests by method 1 showed that the friction coefficient is highly sensitive to temperature. Subsequently, another series of lubrication tests were carried out under a changing temperature condition to study the dynamic behavior of friction coefficient. Figure 4 shows the results of the tests in which the oil temperature was changed stepwise from 60˚C to 80˚C, 60˚C and 40˚C. The drastic drop in friction coefficient at the onset temperature of 60˚C indicates that a tribofilm from MoDTC formed during the initial stage of friction. The friction coefficient of about 0.04 at 60˚C is almost the same as that at steady state in the constant temperature test at 60˚C as shown in Fig. 3. As the temperature increases to 80˚C when 20 min have passed, the friction coefficient slightly decreases and becomes stable at around 0.036 which is similar to that at 80˚C in the constant temperature test. As the temperature decreases to 60˚C, and then to 40˚C, the friction coefficient increases and becomes stable at around 0.04, and then further increases to around 0.05. These results confirm that the MoDTC-containing oil has a friction coefficient varying according to the oil temperature and the coefficient values are almost the same as those in the constant temperature tests as indicated with dashed lines in Fig. 4. It is worth noting that the friction coefficient fluctuates when the set temperature has been reached. This fluctuation will be further explained below.

Figure 4 shows that the temperature undergoes a sinusoidal variation or a ripple after once reaching the set temperature. Interestingly, at 80˚C, the friction coefficient corresponds closely to the ripple. The enlarged view of the friction coefficient data at 80˚C with the temperature axis displayed inverted in Fig. 5

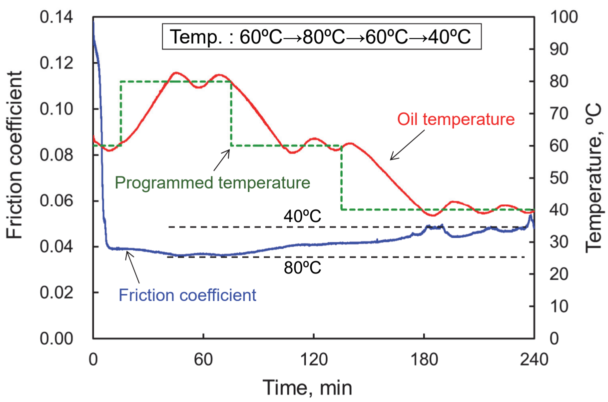



Fig. 4 Friction coefficient of MoDTC-containing lubricant when temperature was changed stepwise.
Broken lines show friction coefficient obtained at a constant temperature as shown in Fig. 3

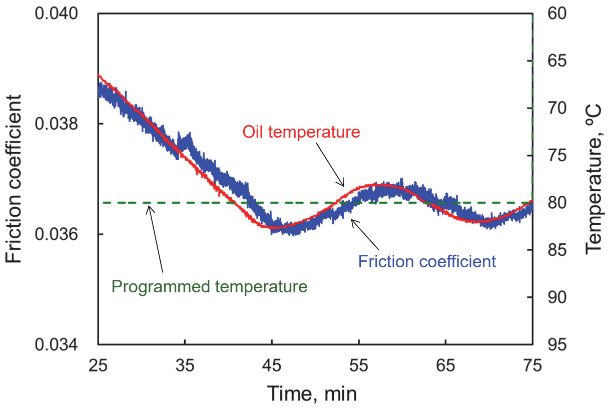



Fig. 5 Oscillatory behavior of friction coefficient according to temperature change

clearly shows how much they correspond with each other. The friction coefficient decreases correspondingly with increase in oil temperature and reaches a value of about 0.036 at 80˚C. It should be noted that the friction coefficient makes a sine curve corresponding to the temperature curve. The real oil temperature exceeds the set temperature while making a ripple following the sine curve. The friction coefficient at around 80˚C follows a very small temperature change of ±2˚C while exhibiting the same periodic change as the oil temperature, suggesting that the friction coefficient changes according to the effect of temperature change. More importantly, there is a phase difference or a time lag between two curves and the friction coefficient always starts to change about 1.5 min later than the temperature. The temperature is expected to change almost simultaneously at the measuring point and in the vicinity of lubricated contact because the measuring point is only about 1cm away from the lubricated contact and the disk rotation allows the oil to be sufficiently agitated. The friction coefficient is supposedly affected by a change in viscosity with almost no time lag. That means that the time lag of about 1.5 min is likely to be caused not by a change in oil viscosity resulting from temperature change, but by other factors such as a structural change in frictional contact. This will be discussed in the next section.

## 4 Discussion

Assuming that the temperature-dependent change of friction coefficient is caused not by a change in base oil viscosity, but by the effect of tribofilm formed through a tribochemical reaction in lubricated contact, frictional surfaces were analyzed to study the relationship between the surface structure and friction coefficient.

### 4.1 Surface analysis: constant temperature condition

The wear scars on the balls subjected to lubrication testing under three constant temperature conditions of room temperature (about 25˚C), 40˚C and 80˚C, were analyzed using HR-TEM and XPS to identify the structure of tribofilm.

The TEM images and EELS and EDS chemical mapping shown in Fig. 6 reveal the formation of tribofilms between the substrate and the protecting overcoat regardless of test temperature. By comparing these HR-TEM images, the tribofilm at 80˚C seems to have a structure composed of layers regularly aligned along the upper surface unlike those at 25˚C and 40˚C. Considering that the TEM and electron diffraction pattern analyses on wear debris performed by Grossiord et al. identified a monolayer $MoS_2$ film [6] and that in Fig. 6 (c) the interlayer distance is about 0.6 nm which is almost the same as that of $MoS_2$, 6.16 Å [17], this layered structure is likely to consist of $MoS_2$. In the results of elemental analyses as shown in the bottom part of Fig. 6, the higher intensity of white color means that the element is present at a higher rate. At 40˚C and 80˚C, unlike 25˚C, there is considerable overlap in the distribution of Mo, S and O which is indicative of the production of $MoS_2$ and $MoO_3$. In addition, these data show that the relative proportion of $MoS_2$:$MoO_3$ depends on temperature because S increases while O decreases as temperature increases.

XPS measurements were performed on the tested balls for the area within a radius of 100 μm from the center of the wear track to analyze the chemical structure of tribofilms. The measured Mo3d spectra in Fig. 7 show that the tribofilm at 40˚C exhibits three peaks characteristic of Mo(IV), Mo(VI) and S2s. Mo(IV) and Mo(VI) are probably derived from $MoS_2$ and $MoO_3$, respectively. The peak area ratio between Mo(IV) and Mo(VI)

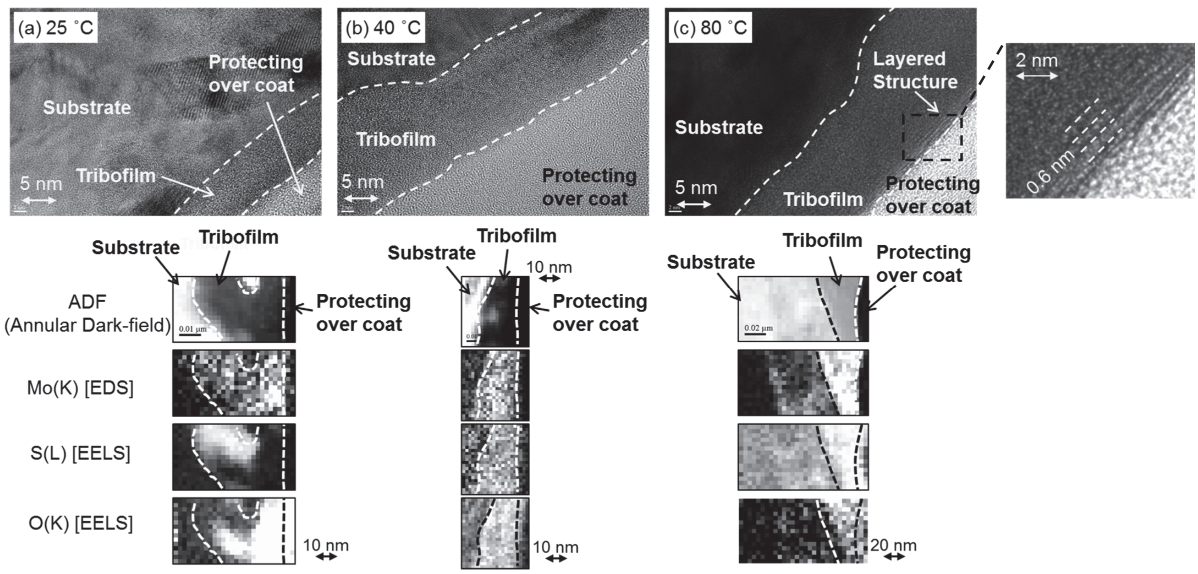



Fig. 6 TEM image and EELS analysis of tribofilm formed by lubrication with MoDTC-containing lubricant (a) 25˚C, (b) 40˚C, (c) 80˚C

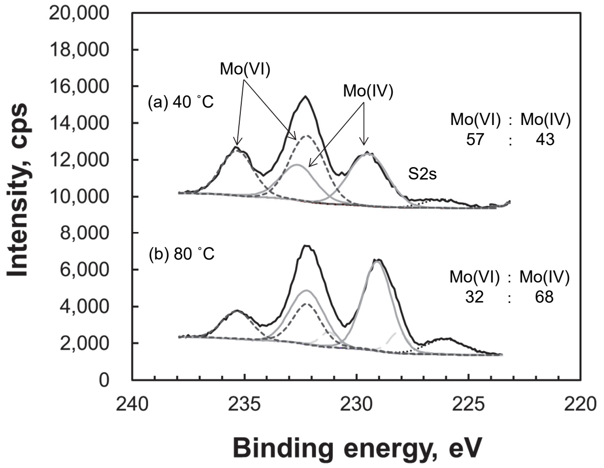



Fig. 7 XPS spectra (Mo3d) of tribofilm on the ball lubricated at (a) 40˚C and (b) 80˚C

determined according to the curve fitting procedure as in 2.3 is Mo(IV):Mo(VI)=43:57. Whereas, the tribofilm at 80˚C had a peak area ratio of Mo(IV):Mo(VI)=68:32, that means the ratio of $MoS_2$ increases at higher temperature.

Based on the XPS results, the higher $MoS_2$ ratio of the tribofilm at steady state at 80˚C would be one of the factors that decrease friction coefficient. The cause for the increased $MoS_2$ ratio at 80˚C will be discussed later. The fact that XPS confirmed the presence of $MoS_2$ while HR-TEM observations revealed no formation of $MoS_2$ layered structure is indicative of the presence of $MoS_2$ nanoclusters [18] at 40˚C. Thus, the $MoS_2$ structure with layers aligned along the lubricated surface at 80˚C, not like $MoS_2$ nanoclusters with slip planes arranged in random order, can enhance lubricity leading to a decrease in friction coefficient.

### 4.2 Surface analysis: changing temperature condition

In the lubrication tests under changing temperature condition (method 2), the friction coefficient changed in response to temperature change. In order to further examine the temperature-dependent structure of tribofilm, TEM and XPS analyses were performed on the tribofilms formed on the wear scar of the balls subjected to lubrication testing either at increased temperature (40˚C to 80˚C) or at decreased temperature (80˚C to 40˚C) as shown in Fig. 8. In the tests, the friction coefficients decreased as the oil temperature increased and vice versa. Same as in Fig. 5, the friction coefficient at around 80˚C followed a very small temperature change of ±2˚C and there was a phase difference between the friction coefficient and oil temperature, clearly demonstrating reproducibility of test results in which the friction coefficient underwent reversible change in response to temperature.

As shown in Fig. 9 (a), the HR-TEM image of the wear track on the ball tested at increased temperature reveals the layered structure in the tribofilm which most likely consists of $MoS_2$. On the other hand, the tribofilm formed during the test at decreased temperature also has the layered structure but it seems to be out of alignment or in a less dense arrangement especially near the upper surface. The layered structure in Fig. 9 (b) should be derived from $MoS_2$ produced at 80˚C because the HR-TEM image of Fig. 6 (b) shows no layered structure for the tribofilm formed during the constant temperature test at 40˚C. The layered structure being out of alignment in a less dense arrangement near the upper surface may be due to that the $MoS_2$ layered structure produced at 80˚C was damaged through

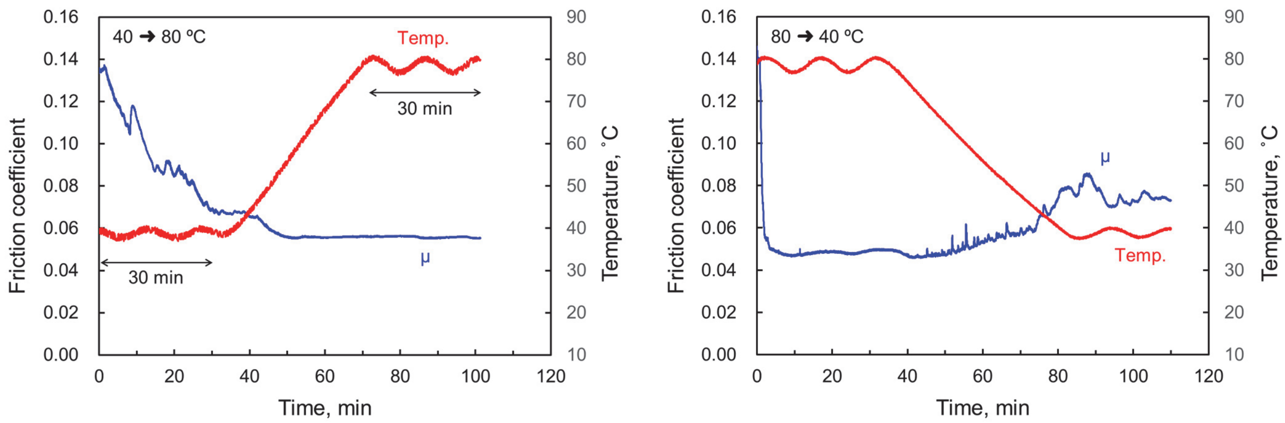



Fig. 8 Temperature dependence of friction coefficient under the condition of a stepwise temperature change

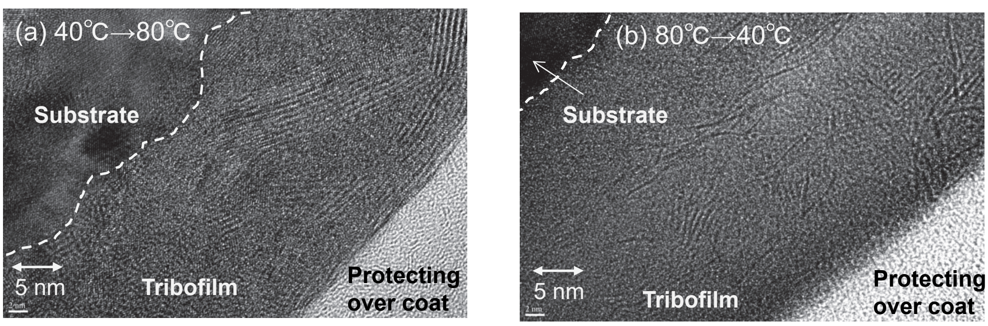



Fig. 9 TEM images of tribofilm formed at (a) 80˚C and (b) 40˚C after the lubrication test shown in Fig.8

shearing accompanied by adhesive wear at 40˚C.

Figure 10 shows XPS spectra of the wear tracks of the tested balls. Mo3d curve fitting was performed to determine the peak area ratio between $MoS_2$ and $MoO_3$. The tribofilm formed during the test at increased temperature (40˚C to 80˚C) is similar to that in the constant temperature test at 80˚C (Fig. 7 (b)) in both Mo3d spectrum shape and the peak area ratio obtained by curve fitting. Their peak area ratios between Mo(IV) and Mo(VI) are specifically 70:30 and 68:32, respectively. Whereas, the tribofilm formed during the test at decreased temperature (80˚C to 40˚C) is more similar to that in the constant temperature test at 40˚C (Fig. 7 (a)) and has a larger Mo(VI) peak area with a Mo(IV):Mo(VI) ratio of 32:68.

4.3 Steady-state model for tribofilm formation

The results of HR-TEM and XPS analyses, in which the tribofilm structure and the relative proportion of $MoS_2$:$MoO_3$ in the tribofilm were found to depend on the oil temperature at the point of lubrication, suggest that $MoS_2$ was produced on the frictional surface through tribochemical reaction of MoDTC while at the same time being removed by wear leading to the formation of a temperature-dependent tribofilm. In other words, the production of $MoS_2$ was facilitated at higher temperature but the $MoS_2$ tribofilm could not be formed in advance before it was partly worn off at lower temperature resulting in a smaller ratio of Mo(IV):Mo(VI). This high rate of $MoS_2$ production at higher temperature is supported also by the steeper slope during the period of drastic drop in friction coefficient at higher temperature as shown in Fig. 3. Thus, the tribofilm with a higher $MoS_2$ ratio could continue to stay on the frictional surface because at 80˚C $MoS_2$ was produced from MoDTC at a higher rate. Another point to note is that $MoS_2$ of high orientation was produced at 80˚C. The phase difference or time lag between temperature and friction coefficient in Fig. 5 can be attributed to the time needed for the change in the structure and composition of tribofilm associated with accumulation of $MoS_2$ through MoDTC reaction. It is suggested by the layered structure being out of alignment near the upper surface in Fig. 9 (b) that such a change in the structure and composition occured because of a dynamic behavior of tribofilm: a tribofilm was formed on the frictional surface, partly removed by being sheared and worn, and replaced with another tribofilm newly formed. The friction coefficient at steady state regardless of temperature can be described by a steady-state model that assumes that it depends on the tribofilm structure maintained by a balance between production and removal.

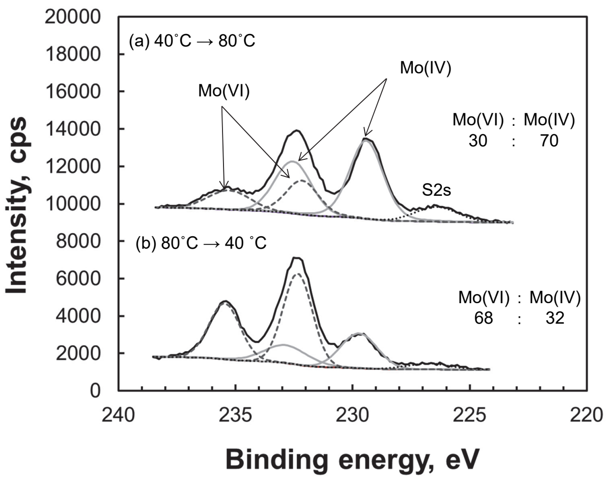



Fig. 10 XPS spectra of the test ball lubricated at (a) 80˚C and (b) 40˚C after the lubrication test shown in Fig. 8

## 5 Conclusions

With an aim to study the effect of temperature on the friction reducing properties of MoDTC as a friction modifier, lubrication tests at different temperatures ranging from room temperature (about 25˚C), 40˚C, 60˚C and 80˚C were carried out to evaluate lubricity of MoDTC based on friction coefficient measurements. The tribofilm formed in lubrication tests were analyzed by HR-TEM and XPS. The results of the lubrication tests, the HR-TEM observations and the XPS analyses are summarized as follows:

(1) Friction-reducing properties of MoDTC are highly dependent on the oil temperature. MoDTC provides a $MoS_2$ tribofilm with a structure and composition ratio depending on the oil temperature.

(2) The friction coefficient changes with change in oil temperature and decreases inversely with the temperature.

(3) The friction coefficient precisely follows a temperature change of about ±2˚C while exhibiting the same periodic change as the oil temperature. Considering that the friction coefficient always starts to change about one and a half minutes later than the oil temperature, it is believed that such a temperature-dependent change is caused not by a change in oil viscosity resulting from temperature change, but by a change in the structure and composition of the tribofilm.

(4) The XPS analysis showed that the tribofilm formed at higher temperature has a higher $MoS_2$ ratio. The HR-TEM observation revealed the tribofilm at 80˚C with a structure composed of aligned layers. The tribofilm structure is changed reversibly with the oil temperature.

(5) A steady-state model of the tribofilm is proposed: The friction coefficient at steady state depends on the tribofilm structure maintained by a balance between production of $MoS_2$ through MoDTC tribochemical reaction and removal of the tribofilm by wear.

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