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A Cylinder and Assembled Four-Block Type Tribo-Test: Novel Method to Study Tribo-Chemistry of Lubricant and Material

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A new tribo-tester was developed by modification of the “Falex Pin & Vee-block” type tribo-test (ASTM D 3233). The advantages of the present method are (1) specimen made of various materials is easily available and (2) the specimen is ready to surface analysis without any processing after the tribo-test. Utilities of the new tribo-test were demonstrated by comparing additive-material compatibility. The tribological properties of aluminum/aluminum contact were improved by phosphorus containing additives, whereas those of copper/copper contact were improved by sulfur containing additives. These results were well explained by tribo-chemistry based on the results of XPS analysis.

Keywords: additive-material compatibility, surface analysis, tribo-chemistry, boundary lubrication

1. Introduction

It is generally understood that anti-wear additives provide boundary film on rubbing surface through tribo-chemical reactions. The tribological properties and mechanism of additive have been well studied for steel surfaces. Recently, aluminum, copper, magnesium, titanium or other metal based alloys have been introduced as advanced tribo-materials. However, it is sometimes pointed out that the conventional anti-wear additives for steel are not always effective for other metal based materials. Two mechanisms are speculated for insufficient tribological properties, namely;

(1) Kinetic issue; insufficient reactivity of the additive with metal surfaces. Poor adsorption activity of the additive onto the surfaces is classified in this category.

(2) Thermodynamic issue; insufficient tribological properties of the tribo-chemically generated product, even if the reaction took place.

In this work, our attention was focused on compatibility of additive with tribo-materials in terms of tribo-chemistry. Chemical analyses of rubbed surface are essential for the purpose. At the beginning of this project, we designed a new tribo-test method to study tribo-chemistry of additive with materials. The requirements for tribo-test are as follows.

(1) Test specimen has to be prepared easily from various materials. In other words, preparation of test specimen needs only a simple processing.

(2) Rubbing flat surface(s), which is beneficial for surface analyses.

(3) Size of test specimen has to be small enough to input the chamber of surface analysis. Cutting process of specimen after tribo-test may damage the product of the tribo-chemical reaction.

A ball-on-flat type and related configurations seem to meet the above requirements. The ball surface is in contact against the flat surface throughout the test. On the other hand, the flat surface repeats contact and release during the test. Certain "static" chemical reaction (or non tribo-chemical reactions) may take place while the flat surface is exposed to the lubricant. It is difficult to distinguish the two reactions by surface analysis. Therefore, elimination of the potentiality of any side reactions is desired.

Our answer was a modification of the ASTM D 3233, so-called “Pin & Vee block” type tribo-test[1,2]. We designed a specimen socket that assembles Vee-shaped parts from two blocks. The configuration of the test is similar to the ASTM D 3233 test, as shown in Fig. 1. After the tribo-test, V-blocks were disassembled; whose flat surface was contacted against the cylinder throughout the test. Size of block is small enough to be inserted in a vacuum chamber of major surface analysis instruments. In this paper, we report applicability of the new tribo-test to evaluate compatibility of additive with materials under boundary conditions. The results are explained in terms of tribo-chemistry by XPS analysis.

Nomenclature

Abbreviation		Description
Chemicals	PAO	Additive-free synthetic hydrocarbon
	P-additive	Dibutyl phosphonate
	S-additive	Dibenzyldisplfide
Material	A-material	Alluminum alloy
	C-material	Copper alloy
	F-material	Bearing steel
Surface analysis	FS	Fresh surface
	OW	Outside wear track
	RS	Rubbed surface
	WP	Wear particle
	XPS	X-ray photoelectron spectroscopy
Others	μ	Friction coefficient
	ASTM	American Standard of Testing Materials
	JIS	Japanese Industrial Standard

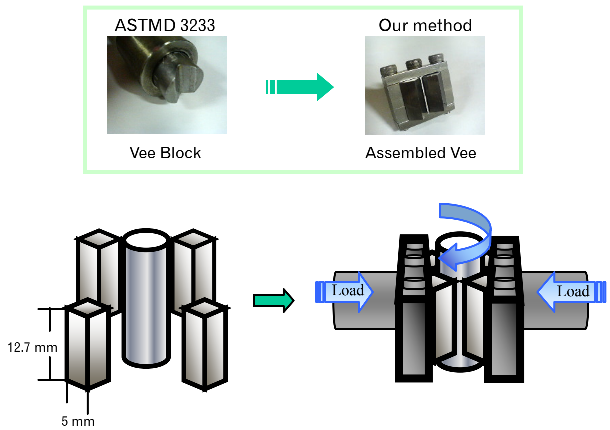


Figure 1 Outline of the tribo-test

Table 2 Conditions of the tribo-test

Operation parameters	Applied load [N]	49, 196
	Rotation of cylinder [rpm]	290
	Sliding velocity [$m \cdot s^{-1}$]	9.6E-02
	Oil temperature [℃]	20-25
	Oil supply [$mg \cdot s^{-1}$]	0.5
	Test duration [min]	20
Test cylinder	Material (JIS)	SUJ2, C6161, A5052
	size [mm]	Φ6.35 × 32 L
Test Block	Material (JIS)	SUJ2, C6161, A5052
	size [mm]	5.0 × 5.0 × 12.7

2. Experimental

The properties and contents of tribo-materials, and conditions for the tribo-test are listed in Tables 1-2. P-additive and S-additive were selected as representative additives for mineral oils. They were dissolved in PAO at the concentration of 60 $mmol \cdot kg^{-1}$. Viscosity of the base oil was 16.8 and 3.90 $mm^2 \cdot s^{-1}$ at 40 and at 100 °C, respectively. The sample oil was provided to the contact by a syringe throughout the tribo-test. XPS was conducted to investigate the chemical reaction of the additives. Samples for the analysis were prepared as follows.

(1) RS and OW: V-blocks were disassembled after the tribo-test. They were ultrasonically washed with hexane and then dried. Chemistry of the boundary film could be acquired, if the film was generated on RS. Products of static reaction of the additive with the material could be detected on OW, if the reaction took place without any tribological effects.

(2) WP: Oil drop from the contact was received in a Petri dish. Wear particle in the oil was collected by filtration. Wear of the boundary film affords the additive-derived compounds on WP.

(3) FS: PAO was provided to the contact and the resultant oil composed of wear particle was dropped into a solution of additive in a Petri dish. Wear particle was collected by filtration. The results are related to the reactivity of the additive with nascent surfaces.

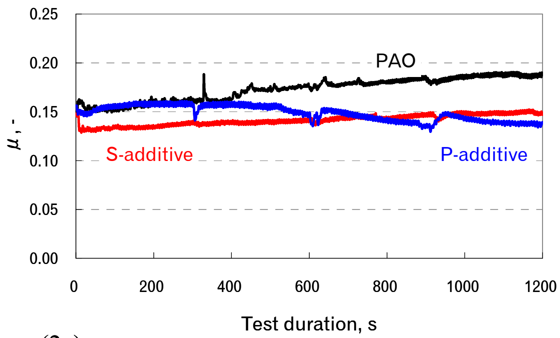
Table 1 Properties and contents of the material

Material		Contents of element, mass%								
Code	JIS	Fe	Cu	Al	C	Si	Mn	Cr	Ni	Zn
F-material	SUJ2	Balance			0.95-1.1	0.15-1.35	<0.5	1.3-1.6		
C-material	C6161	2.0-4.0	Balance	7.0-10			0.5-2.0		0.5-2.0	
A-material	A5052	<0.40	<0.10	Balance		<0.25	<0.10			<0.10

3. Results and discussion

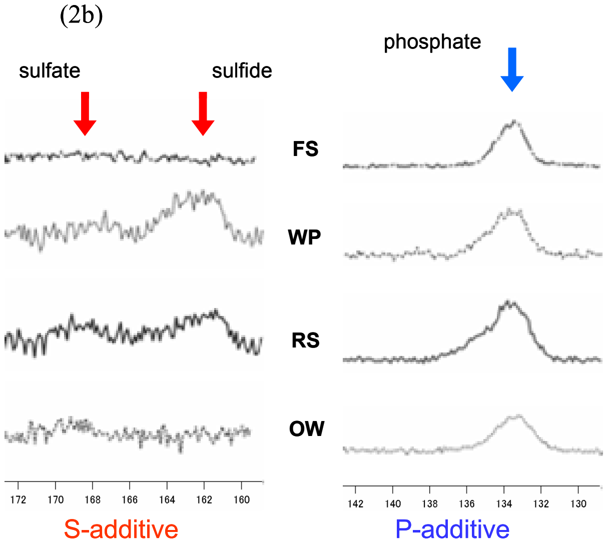
3.1. F-material

Friction test using a steel cylinder and steel blocks was examined to confirm the reliability of the new tribo-tester. The conventional additives reduced both friction and wear under these conditions. Iron phosphate or iron sulfide was found on RS by XPS analysis lubricated with the P-additive or S-additive, respectively (Figure 2). These results are in accordance with the literature[3]. Here we show the utility of the new test method for studying tribo-chemistry of additives.



(2a)

(2b)

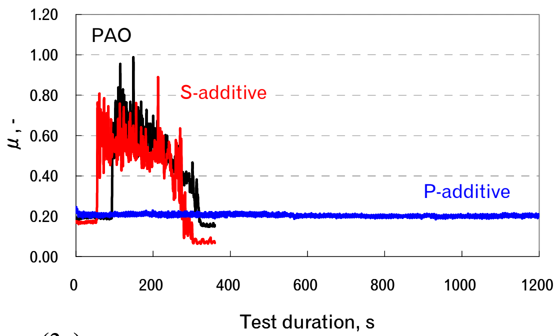


(2c)

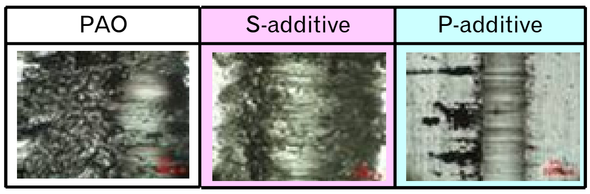
Figure 2 Results obtained from F-material at 196N
(a) friction trace (b) morphology of worn surfaces
(c) XPS spectrum

3.2. A-material

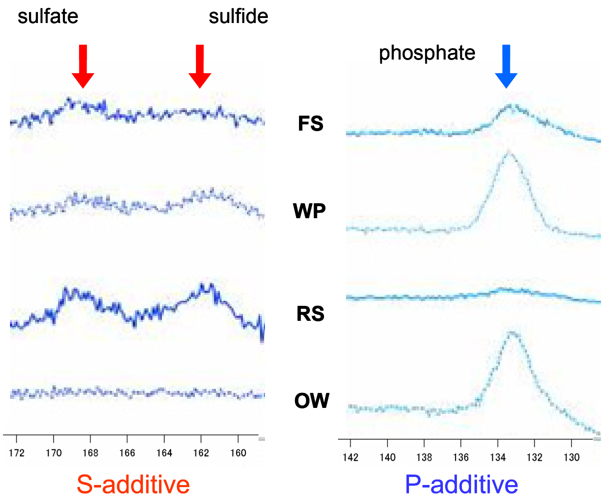
To the best of our knowledge, the tribological properties of aluminum alloy are still unsatisfactorily with the conventional lubricants. In fact, severe wear or seizure was observed by the additive-free base oil even at low load of 49 N. The test was aborted within several minutes. The S-additive did not improve the tribological properties at all. The P-additive prevented seizure and it reduced friction remarkably, as shown in Figure 3. However, the P-additive has insufficient anti-wear properties under these conditions in comparison with those for steel.



(3a)



(3b)



(3c)

Figure 3 Results obtained from A-material at 49N
(a) friction trace (b) morphology of worn surfaces
(c) XPS spectrum

A considerable amount of sulfides and sulfates were found on the RS while the tribological properties of the S-additive were poor. The results indicate that the S-additive undergoes the tribochemical reaction on aluminum surface whereas the tribological properties of the resultant product were poor. Phosphates were found after the tribo-test with P-additive. It should be noted that only a trace amount of phosphates was found on RS while considerable amount of those was detected on WP. The results also indicate that the resultant products possessed poor tribological properties. Therefore, insufficient tribological properties of the P-additive for aluminum alloy are considered as "thermodynamic issue". P-additive reacted with un-rubbed surfaces. On the other hand, tribological process was required for the reaction of the S-additive; almost no reactions were detected on OW.

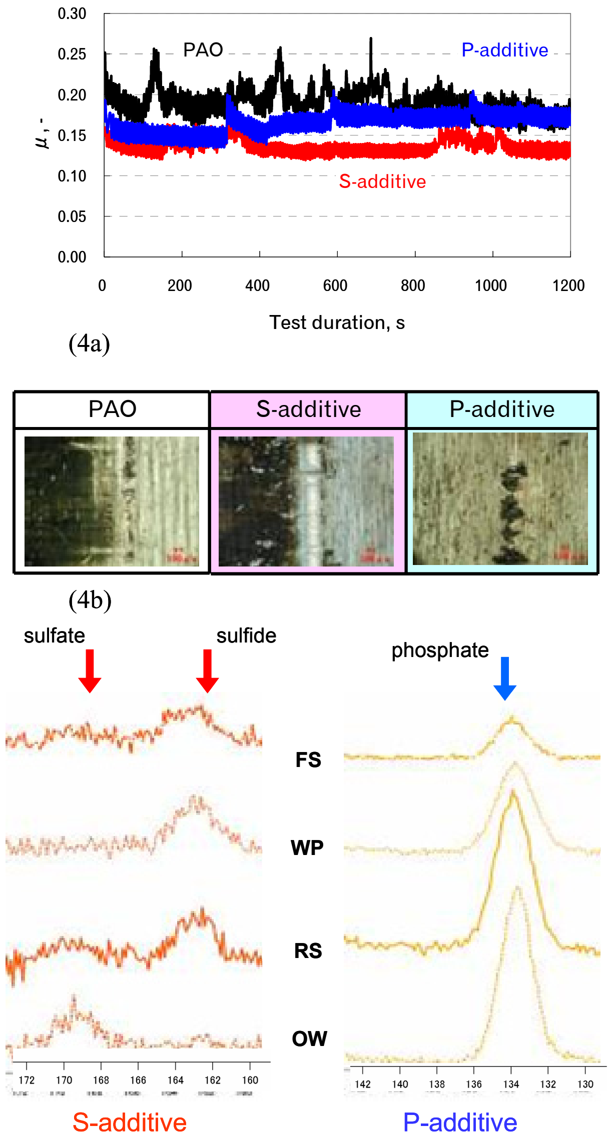


Figure 4 Results obtained from C-material at 49N
(a) friction trace (b) morphology of worn surfaces
(c) XPS spectrum

3.3. C-material

PAO exhibited friction fluctuation throughout the tribo-test. The S-additive reduced friction to considerable extent. It also provides a smooth worn surface, in comparison with the additive-free oil. The XPS spectra show the formation of sulfides on FS, on WP, and on RS. The results indicate that the tribo-chemical reaction of the S-additive affords sulfides on rubbing surface; thereby reduce friction. It should be noted that sulfates are major products on OW. Effects of tribo-chemistry on reaction of copper with the S-additive are of interests. The P-additive also reduced friction at the initial stage of the test. However friction coefficient gradually increased as test duration. The P-additive gave deposits on worn surfaces as shown in Figure 4b. The XPS spectra show the formation of phosphate on RS. Although considerable amount of phosphates retain on RS, friction reduction by the P-additive was little (Figure 4). These results suggest the "thermodynamic issue" for the combination of copper-alloy with the P-additive under these conditions.

4. Conclusions

A new tribo-test which evaluates compatibility of lubricant with tribo-material was developed. Specimen made of various materials for the test is easily available. Surface analysis of the resultant specimen is possible without any cutting process after the tribo-test. The present method provide powerful tool to study tribo-chemistry of lubricants with tribo-materials. As examples, poor tribological properties of the conventional additives composed of sulfur or phosphorus were investigated. Although the tribo-chemical reaction of the additive took place on aluminum or copper surfaces, the resultant products exhibited poor tribological properties under the tribo-test conditions.

5. Acknowledgements

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6. References

[1] Savin Sebastian, K. S. & Bhaskar, V. R., "A new design for the pin and V-blocks tribometer," Tribology International, 28, 4, 1995, 219-223.

[2] Blau, P. J., "Measuring Friction in the Laboratory," in "Friction Science and Technology," Mercel Dekker, New York, 1996.

[3] Pawlak, Z., "Tribochemical Nature of Antiwear Films," in "Tribochemistry of Lubricating Oils," Elsevier, the Netherland, 2003.