Investigation of calcium phosphate (CaP) tribofilms from commercial automatic transmission fluids (ATFs) and their correlation with antishudder performance

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Abstract: The friction properties of wet clutches are highly dependent on the surface tribofilms formed by automatic transmission fluids (ATFs). Here, four commercial ATFs were evaluated with a disc-on-disc tribometer to study tribofilm formation on steel surfaces and the effects of tribofilms on the friction properties. The chemical composition, stoichiometry, structure, and thickness of the tribofilms were investigated by scanning electron microscopy (SEM), energy dispersive X-ray spectrometry (EDX), secondary ion mass spectrometry (SIMS), and X-ray photoelectron spectroscopy (XPS). Calcium phosphate (CaP) tribofilms form on the friction surface with all ATFs, which contributes to their antishudder characteristics. The thickness and surface coverage of CaP tribofilms are positively correlated with their antishudder properties.

Keywords: tribofilm; commercial ATF; antishudder; hydroxyapatite

1 Introduction

Wet clutches are used in automatic transmissions to transmit torque. The primary component of a wet clutch is the tribological paper and steel discs immersed in the ATF. During engagement of the wet clutch, the paper disc (static) and steel disc (rotating) are pushed together by force, and then friction between the discs eliminates the speed difference, and the two discs rotate at the same speed to reach a state of lock up. Thus, power is transmitted from an input to an output shaft [1]. This friction between surfaces is a crucial feature of automatic transmission performance. However, uncontrolled friction often causes shudder. For instance, when the dependence of the kinetic friction coefficient on the relative velocity (μ - v curve) is negative, steady sliding at the equilibrium point becomes unstable, and the instability generates shudder [2]. Stick-slip is another type of shudder that results

from the discontinuity between static and kinetic friction; stick-slip can arise when the static friction is higher than the dynamic friction [3, 4]. The tendencies of a wet clutch to experience both stick-slip and shudder are usually characterized by the μ - v curve. A positive slope of the μ - v curve is beneficial if stick-slip and shudder are to be avoided.

Tribofilms play a key role in controlling friction in boundary and mixed lubrications [5, 6]. Some lubricant additives, such as antiwear (AW), friction modifier (FM), and detergent, are known to chemically interact with metallic surfaces to form tribofilms, and the friction characteristics are affected by the tribofilms [7–13]. Tribofilms protect the substrate from excessive wear, reduce/increase the friction coefficient, and maintain good anti-seizure performance [14, 15]. Antishudder performance of wet clutches can be modulated by FMs and other additives in ATFs [16–18]. Our previous study found that a CaP tribofilm formed by FM and

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detergent affected friction characteristics [19, 20]. Zhao et al. [8, 21] investigated the chemical composition of tribofilms containing P and Ca on the steel friction plate of the clutch. Tohyama et al. [16, 17] found that the combination of Ca-containing FM and P-containing detergent provides good antishudder performance. Fitima et al. [18] also reported that Ca-containing detergents and FMs affected antishudder properties. However, these abovementioned studies are based on laboratory formulations; there are few studies on commercial ATFs and their actual performance.

Many analytical techniques are used to analyze tribofilms. Rydel et al. [6] studied the dependence between the morphology of the tribofilm and the microstructure of the underlying material by atomic force microscopy (AFM). Tipton et al. [22] characterized changes in tribofilm chemistry by scanning auger microscopy (SAM). However, SAM is not suitable for detecting tribofilms with a thickness of more than 200 nm. Auger electron spectroscopy (AES) was used for surface elemental mapping of tribofilms by Qu et al. [23]. In this study, XPS depth analysis is used, in which the chemical composition of tribofilm is determined down to a depth of approximately 900 nm. Moreover, the 2-D imaging of secondary ion mass spectrometry (SIMS) is a very effective way to observe the distribution state of elements on different layers in the tribofilm [24]. In addition, SIMS 2-D mapping can accurately measure the ion concentration in the micro-region, which serves as a primary method for comparing the thickness and distribution intensity of tribofilms.

Most of the studies discussed above used lab-formulated ATFs; however, many formulations cannot be applied in practice because of the poor compatibility of additives and other negative effects for ATFs. This study uses four commercial ATFs. SEM, EDX, SIMS, and XPS are used to comprehensively characterize the composition of tribofilms. Tribological testing is carried out to study the relationship between a tribofilm and its antishudder characteristics. Although the formulations are different, the antishudder properties can be primarily attributed to the CaP tribofilms formed by reactions among additives. The combination of XPS depth analysis and SIMS 2-D imaging confirms that thicker and more densely distributed tribofilm is beneficial to antishudder performance. These results

provide deeper insights into the actual working conditions of ATFs.

2 Experimental

2.1 Tribological testing

A Wazau tribometer (Wazau Co., Ltd., Germany), a miniature version of the LVFA [25], was used to evaluate the μ - v characteristics in this study (Fig. 1). In a 50 ml oil tank, a paper disc (BorgWarner 6100) is rotated on a static steel disc. The contact pressure, relative speed between friction couple, and the temperature of the oil were set according to the test procedure. The friction coefficient at various speeds, loads, and temperatures were recorded. The test procedure was carried out according to JASO M349 for LVFA [19, 20, 25]. The tribological testing for each condition was done in triplicate.

2.2 Test oil

Oils A, B, C, and D from four well-known ATF companies were purchased for this study. These oils were chosen based on their applicability and generality with high market share. The characteristics of the four ATFs are shown in Table 1. Oils A, B, and C passed the JASO standard to satisfy the antishudder requirement, while ATF-D only met the DEXRON III standard without antishudder characteristics.

ATFs were tested in the Wazau test rig to evaluate their friction performance. The μ - v curves and the steel discs from Wazau tests were analyzed.

Fig. 1 Wazau test rig.

Table 1 Characteristics of ATF A, B, C, and D.

Company	Oil A	Oil B	Oil C	Oil D	Test method
	A, USA	B, UK	C, USA	D, China	
Kinematic viscosity (100 °C) ($\text{mm}^2\cdot\text{s}^{-1}$)	6.177	6.143	6.006	7.916	ASTM D445
Viscosity index	154	150	151	202	ASTM D2270
Acid number ($\text{mg KOH}\cdot\text{g}^{-1}$)	3.29	1.28	1.69	1.16	ASTM D664
Density (20 °C) (kg/m^3)	845	844	845	846	ASTM D1298
Ba ($\text{mg}/(\text{kg}/\text{m}^3)$)	< 0.1	< 0.1	2.8	–	ASTMD5185
B ($\text{mg}/(\text{kg}/\text{m}^3)$)	129	68.9	106	164	ASTMD5185
Ca ($\text{mg}/(\text{kg}/\text{m}^3)$)	48	121	174	58.6	ASTMD5185
Mg ($\text{mg}/(\text{kg}/\text{m}^3)$)	< 0.1	0.35	0.56	–	ASTMD5185
P ($\text{mg}/(\text{kg}/\text{m}^3)$)	289	196	501	297	ASTMD5185
Si ($\text{mg}/(\text{kg}/\text{m}^3)$)	< 0.7	5.3	3.9	< 0.2	ASTMD5185
Zn ($\text{mg}/(\text{kg}/\text{m}^3)$)	< 0.4	< 0.3	2.73	< 0.6	ASTMD5185
Satisfied standards	JASO 1A	JASO 1A	JASO 1A	GM DEXRON III	–

2.3 Surface analysis

Surface analysis was carried out to determine the topography and chemical composition of tribofilms. The steel friction surfaces were analyzed by scanning electron microscopy (SEM, Philips XL-30TM), energy dispersive X-ray spectrometry (EDX, Philips XL-30TM) and X-ray photoelectron spectroscopy (XPS, Thermo Electron ESCA 250).

ToF-SIMS analysis was carried out (TOF SIMS V, ION-TOF GmbH, Muenster, Germany) to study the element distribution of the post-test steel surface tribofilm. Analysis was performed using a pulsed Bi⁺ ion high energy beam of 30 KeV with a current of 1 pA on a 500 μm $\times$ 500 μm scanned area. A 2-D SIMS image was generated by scanning the Bi₃⁺ primary ion beam over the sample (50 μm $\times$ 50 μm , 256 pixels $\times$ 256 pixels) 50 times using Surface Lab6 software (ION-TOF, Münster, Germany).

In the XPS detection, an X-ray spot size of approximately 500 μm was chosen. Peak detail spectra were obtained at pass energy of 50 eV. The hydrocarbon C1s peak at 284.6 eV was used as the binding energy reference. Depth profiles were performed with 3 kV Ar ion sputtering. A sputter time of 10 s roughly corresponds to a depth of 1 nm, with a systematic uncertainty of 50%. By successively sputtering off the top layers, the chemical composition of tribofilm was determined down to a depth of approximately 900 nm.

3 Results and discussion

3.1 Friction results

The durability of the antishudder property is an important criterion to evaluate the overall performance of an ATF formulation [20]. Figure 2 shows μ - v curve (40 °C) results of the four ATFs by the Wazau endurance-aging test. The sign of $d\mu/dv$ at $v = 0.3$ and 0.9 m/s on the μ - v curves shown in Table 2 is the judgment of antishudder property (JASO M349: 2001-5.1).

ATF-A shows a relatively good positive slope for its μ - v curve ($d\mu/dv$ (0.3 m/s) > 0.00, $d\mu/dv$ (0.9 m/s) > 0.00) at 0 and 24 h. The slope of 48 h μ - v curve in the speed region of 0.4–1.0 m/s becomes slightly negative ($d\mu/dv$ (0.9 m/s) < 0.00). Then, the 72 h result shows a negative friction curve slope ($d\mu/dv$ (0.3 m/s) < 0.00, $d\mu/dv$ (0.9 m/s) < 0.00), which means that the antishudder characteristic deteriorates between 48 and 72 h.

ATF-B exhibits the best antishudder durability among the four oils. It maintains $d\mu/dv$ (0.3 m/s) and $d\mu/dv$ (0.9 m/s) values above 0.0 for longer than 144 h. The μ - v curves from 0 to 144 h are all quite positive, as shown in Fig. 2(b). The slope of the 168 h μ - v curve, which shows a positive slope from the beginning, becomes negative when speed exceeds 0.3 m/s. The 192 h μ - v curve shows a steep negative slope for low speed.

Fig. 2 μ - v curves after endurance test for ATFs A, B, C, and D.

Table 2 $d\mu/dv$ at 0.3 and 0.9 m/s.

$d\mu/dv$	v (m/s)	0 h	24 h	48 h	72 h	96 h	120 h	144 h	168 h	192 h
ATF-A	0.3	+	+	+	—	—	—	—	—	—
	0.9	+	+	—	—	—	—	—	—	—
ATF-B	0.3	+	+	+	+	+	+	+	+	—
	0.9	+	+	+	+	+	+	+	—	—
ATF-C	0.3	+	+	+	+	+	—	—	—	—
	0.9	—	—	—	—	—	—	—	—	—
ATF-D	0.3	—	—	—	—	—	—	—	—	—
	0.9	—	—	—	—	—	—	—	—	—

The $d\mu/dv$ (0.3 m/s) values for ATF-C remain above 0.0 until approximately 96 h, but the degree of the positive slope for the low-speed region becomes eased with aging. The μ - v characteristics in the high-speed region have a negative slope after 24 h, and the negative slope value increases slowly with aging.

The slope of the μ - v curve for ATF-D appears negative from 0 hours (Fig. 2(d)). With increasing durability time, the degree of negative slope is rapidly deepened. Throughout the entire aging test, all the

$d\mu/dv$ (0.3 and 0.9 m/s) values are less than 0.

The antishudder durability of ATFs A and C is 48 and 96 h, respectively, which is less than that of ATF-B (168 h), while ATF-D has no antishudder property.

After 24 h of durability testing, ATFs A, B, and C possess antishudder characteristics. The slopes of the μ - v curves at 24 h for ATFs A, B, and C are positive while that of ATF-D is negative. The analyses of the steel surface after 24 h durability testing are presented in Section 3.2 below.

3.2 Steel surfaces analysis

3.2.1 SEM and EDX

The 24 h post-test steel disc surfaces for the four oils are shown in Figs. 3(a), 4(a), 5(a), and 6(a). SEM pictures of steel discs at the time of complete disappearance of antishudder characteristics (72, 192, and 120 h for

ATFs A, B, and C, respectively) are shown in Figs. 3(b), 4(b), and 5(b). Figure 6(b) is the SEM image of steel disc after 48 h of endurance-aging for ATF-D.

Black tribofilms are detected on the surface of the four ATFs (Figs. 3–6). Several points in the tribofilms are selected for EDX analysis, as shown in Fig. 7. The tribofilms of ATFs A, B, C, and D mainly contain Ca,

Fig. 3 SEM images of post-test steel disc surfaces after (a) 24 h and (b) 72 h Wazau durability tests for ATF-A.

Fig. 4 SEM images of post-test steel disc surfaces after (a) 24 h and (b) 192 h Wazau durability tests for ATF-B.

Fig. 5 SEM images of post-test steel disc surfaces after (a) 24 h and (b) 120 h Wazau durability tests for ATF-C.

Fig. 6 SEM images of post-test steel disc surfaces after (a) 24 h and (b) 48 h Wazau durability tests for ATF-D.

Fig. 7 EDX spectra of tribofilms on steel disc surfaces after 24 h Wazau durability tests: (a) ATF-A, (b) ATF-B, (c) ATF-C, and (d) ATF-D.

P, and O.

The 24 h steel surfaces of ATFs A, B, and C are distributed with bushy tribofilms, while the 24 h tribofilm of ATF-D is sparse (Figs. 3(a), 4(a), 5(a), and 6(a)). For ATF-A, the antishudder characteristic disappears after 72 h of endurance testing, and the negative slope of the 72 h μ - v curve is much lower than that of the 48 h μ - v curve. At the same time, the

CaP tribofilm at 72 h disappears (as shown in Fig. 3(b)). Similarly, at the end of ATF-B's durability test, the CaP tribofilm almost disappears when the μ - v curve suddenly reaches down to the maximum negative slope value at 192 h (as shown in Fig. 4(b)). This phenomenon is consistent with our previous research; that is, the CaP tribofilm contributes to maintaining the antishudder property [19]. The slope

of μ - v curves for ATF-C slowly decreases, and there is little difference in slope value every 24 h. Until the antishudder characteristic disappears at 120 h, a very sparse tribofilm remains on the steel surface (as shown in Fig. 5(b)). In the endurance-aging process of ATF-C, the slow decline of the μ - v curve slope may be related to the gradual disappearance of the CaP tribofilm.

The steel surface of ATF-D in Fig. 6 shows different features compared to the steel surfaces of the other ATFs. It has no antishudder characteristic at 0 h, and the CaP tribofilm on the steel surface is rare. After 48 h, the degree of negative slope deepens rapidly. The loss of antishudder is probably due to the disappearance of the CaP tribofilm.

3.2.2 SIMS

Figure 8 shows the Ca^+ 2-D distribution map on the steel surface after the 24 h endurance test for ATFs A, B, C, and D by means of ToF-SIMS. In order to eliminate the influence of surface impurities, samples were swept by oxygen for 200 seconds before detection. The MC and TC in Fig. 8 represent the highest Ca^+

concentration in the micro detection area and the total number of Ca^+ in the whole detection area, respectively. In the 2-D image, the greater the brightness of yellow, the higher the concentration of Ca^+ in the microsphere. Therefore, the distribution of Ca for ATF-B is the most intensive and homogeneous of the four samples. The Ca^+ concentration in the micro detection area is $B \text{ (MC 116)} > A \text{ (MC 93)} > C \text{ (MC 89)} > D \text{ (MC 16)}$, which indicates that the thickness of the tribofilm is $B > A > C > D$. From the MC value of A (MC 93), B (MC 116), and C (MC 89), it is speculated that they have similar tribofilm thickness. This will be verified in the following XPS depth analysis. However, by comparing the total number of Ca^+ (TC) of the four tribofilms, we conclude that the intensity of Ca^+ distribution of tribofilm B (TC 4.558 e^6) is much larger than that of tribofilm A (TC 1.138 e^6), C (TC 3.992 e^5), and D (2.64 e^5). These results suggest that the surface coverage of tribofilm B is greater than the others.

3.2.3 XPS

The XPS narrow spectra of $\text{Ca}2\text{p}$, $\text{P}2\text{p}$, and $\text{O}1\text{s}$ regions

Fig. 8 ToF-SIMS mappings ($300 \mu\text{m} \times 300 \mu\text{m}$) of Ca for post-test (24 h endurance) steel surfaces: (a) ATF-A, (b) ATF-B, (c) ATF-C, and (d) ATF-D.

obtained from steel surfaces after the 24 h durability test for ATFs A, B, C, and D are shown in Figs. 9–12. The Ca2p and P2p peaks detected almost match the peaks of different forms of calcium phosphate [26]. The O1s region obtained from ATFs C and D exhibit two components, indicating the presence of hydrogen phosphate groups in the tribofilms [27]. It can be inferred from these results that the components of these tribofilms are probably different forms of hydroxyapatite.

The elemental depth profiles of the tribofilms after the 24 h durability tests for ATFs A, B, C, and D are shown in Fig. 13 (a sputter time of 10 s roughly corresponds to a depth of 1 nm). It is observed that the P/Ca ratios of the three tribofilms have linear relationships (2.4:1 for ATF-A, 1:1 for ATFs B, C, and D) in the depth direction. This result verifies that the tribofilms are different forms of hydroxyapatite. The tribofilm thickness of ATFs A, B, and C exceeds 900 nm, while the thickness of ATF-D is only about

Fig. 9 Ca2p, P2p, and O1s XPS narrow spectra of ATF-A tribofilm.

Fig. 10 Ca2p, P2p, and O1s XPS narrow spectra of ATF-B tribofilm.

Fig. 11 Ca2p, P2p, and O1s XPS narrow spectra of ATF-C tribofilm.

100 nm. At the maximum value of XPS in depth detection (900 nm), the percentage of Fe is only 50% for tribofilm B, which is less than that of tribofilms A (80%) and C (80%), which means the thickness of tribofilm B is larger than the others. This may explain why ATF-B has excellent antishudder property, while ATF-D does not have any antishudder ability. A hydroxyapatite tribofilm with high thickness and high coverage is helpful for maintaining good antishudder performance and durability. The tribofilm is much like a surface texture produced by the chemical reaction of additives [28].

Fig. 12 Ca2p, P2p, and O1s XPS narrow spectra of ATF-D tribofilm.

4 Conclusions

In this study, SEM, EDX, SIMS, and XPS analysis were used to systematically study the composition of tribofilms generated from four well-known brands of ATFs. The components of the tribofilms are all forms of hydroxyapatite. Results of XPS depth analysis and SIMS 2-D imaging show that the thickness and distribution intensity of tribofilm have a positive correlation with antishudder performance.

Among the four ATFs, ATF-B forms the thickest and highest coverage CaP tribofilm on the steel surface

Fig. 13 XPS depth profile of the post-test (24 h durability) steel surface tribofilms for ATFs A, B, C, and D (etching rate = 0.1 nm/s).

with the best antishudder durability (168 h). The antishudder durability of ATF-A and C is 48 and 96 h respectively, probably due to their lower tribofilm coverage and depth, while ATF-D formed the thinnest CaP tribofilm with no antishudder property. These results suggest that CaP tribofilms with high thickness and high surface coverage maintain good antishudder performance and durability.

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