CHAPTER IV

RESULTS AND DISSCUSSION

This research related to the preparation of lubricant which is suitable for use in FDB spindle motors for 2.5 inch HDD. As proposed in the previous chapter, the results will be shown and discussed in each part, respectively.

4.1 Investigation of the suitable base oils for finished lubricant preparation

4.1.1 Selection for type of ester base oils

Initially, type of ester base oils such as monoester, diester and polyol ester were investigated in terms of kinematic viscosity which should be in range of 8 – 10 mm^2/s. The kinematic viscosity for each type of ester base oil is shown in table 4.1.

Table 4.1 Kinematic viscosity of several ester base oils

Ester type	**Ester compound**	**Kinematic viscosity at 40 °C (unit :mm^2/s)**
Momoester	Methyl decanoate	3
	2-Ethyl hexyl oleate	29
Diester	Dimethyl adipate	8
	Bis(2-ethylhexyl)sebacate	12
Polyol ester	Glycerol monooleate	90
	Glycerol trioleate	230

According to table 4.1, the diesters have viscosity close to the range of 8 – 10 mm^2/s. Thus, the diesters are selected as candidates for further investigation as finished lubricant in FDB spindle motors for 2.5 inch HDD.

4.1.2 Selection for suitable diester base oils

The diester base oils as shown in table 4.2 were studied in terms of kinematic viscosity. The flowing time of each diester base oil was measured at 40 °C. Then, the kinematic viscosity was calculated using equation 4.1.

$$\nu = c \times t \qquad (4.1)$$

Where, ν = kinematic viscosity in mm^2/s

c = calibration constant of the viscometer in $(mm^2/s)/s$ unit

t = measured flow time in second unit

Example 1

Kinematic viscosity of dioctyl adipate at 40 °C:

$$\nu = 525.30 \text{ s} \times 0.01561 \ (mm^2/s)/s$$

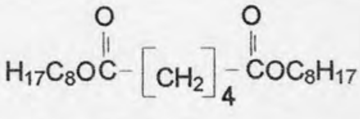
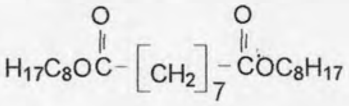
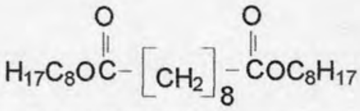
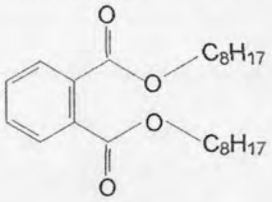
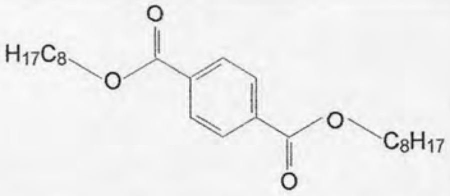
Where, ν = kinematic viscosity in mm^2/s unit

$c = 0.01561 \ (mm^2/s)/s$ at 40 °C

$t = 525.30$ second

The kinematic viscosity of other ester base oils were calculated by the same method as shown in example 1 and the results are summarized in table 4.2.

Table 4.2 Kinematic viscosity of diester base oils

Diester	**Structure**	**Kinematic viscosity at 40 °C (unit :mm^2/s)**
Dioctyl adipate (DOA)	 $H_{17}C_8O\overset{\overset{O}{\|}}{C}-\left[CH_2\right]_4-\overset{\overset{O}{\|}}{C}OC_8H_{17}$	8.2
Dioctyl azelate (DOZ)	 $H_{17}C_8O\overset{\overset{O}{\|}}{C}-\left[CH_2\right]_7-\overset{\overset{O}{\|}}{C}OC_8H_{17}$	11.8
Dioctyl sebacate (DOS)	 $H_{17}C_8O\overset{\overset{O}{\|}}{C}-\left[CH_2\right]_8-\overset{\overset{O}{\|}}{C}OC_8H_{17}$	12.5
Dioctyl Phthalate (DOP)		30.2
Dioctyl terephthalate (DOTP)		30.5

From table 4.2 it was obvious that the kinematic viscosity of DOA, DOZ and DOS base oils were possible to be used to prepare finished lubricants for FDB spindle motors in 2.5 inch HDD.

However, the DOZ base oil has much higher price than those of DOA and DOS base oils as shown in table 4.3. Therefore, the price of DOZ base oil may affect the FDB spindle motors production cost. For this reason, the DOA and DOS base oils were selected to prepare finished lubricants by varying percentage of blending as shown in table 4.4. Thus, the blend lubricating base oil would have viscosity in the range of 8 – 10 mm^2/s.

Table 4.3 Properties and prices of DOA, DOZ and DOS base oils

Item	**Unit**	**DOA** **(CAS no. 103-23-1)**	**DOZ** **(CAS no.103-24-2)**	**DOS** **(CAS no.122-62-3)**
Appearance	-	Colorless clear liquid	Colorless to light yellow clear liquid	Colorless clear liquid
Kinematic viscosity at 40 °C	mm^2/s	8.2	11.8	12.5
Specific Gravity	-	0.927 ± 0.003	0.918 ± 0.003	0.915 ± 0.003
Total Acid number	mg KOH /g	0.6 Max	0.8 Max	0.05 Max
Flash Point	°C	236	238	246
Pour point	°C	-62	-62	-62
Price	฿/kg.	4,480	20,679	3,270

In this research, mixtures of DOA and DOS base oils were designated as samples A to H and characteristic analysis results are represented in table 4.4.

4.1.3 Properties assessment for base oil samples

Initially, base oil samples as shown in table 4.4 were analyzed for kinematic viscosity which should be in range of 8 – 10 mm^2/s at 40 °C and weight loss by TG/DTA. The analysis results of these base oil samples are shown in table 4.4, and Fig.4.1 and 4.2.

Table 4.4 Kinematic viscosity and weight loss of base oil samples

Sample designation	**%Blending**		**Kinematic viscosity (unit : mm^2/s)**		**%weight loss (unit : %)**	
	DOA	**DOS**	**40 °C**	**100 °C**	**85 °C x 3 hrs**	**120 °C x 3 hrs**
A	20	80	10.64	3.21	0.198	3.394
B	30	70	9.82	3.14	0.205	3.596
C	40	60	9.76	3.10	0.251	3.708
D	45	55	9.64	3.08	0.266	4.402
E	50	50	9.41	3.02	0.279	5.774
F	55	45	9.34	2.98	0.302	8.683
G	60	40	9.22	2.94	0.407	10.874
H	70	30	8.69	2.85	0.863	13.070
DOA	100	0	7.66	2.32	0.907	17.964
DOS	0	100	12.24	3.23	0.074	2.956

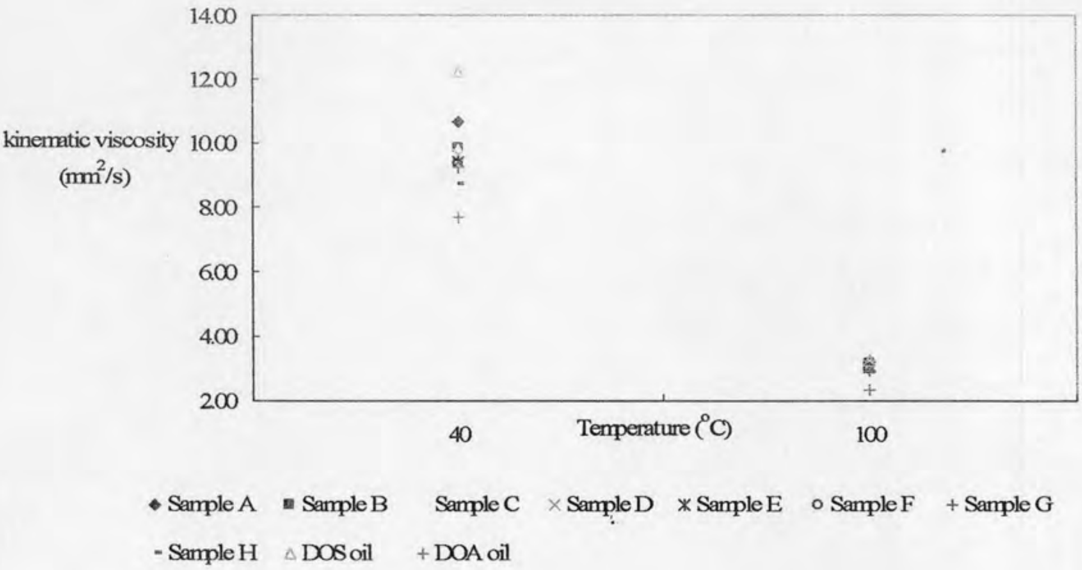


Figure 4.1 Plot of testing temperature versus kinematic viscosity of several base oil samples

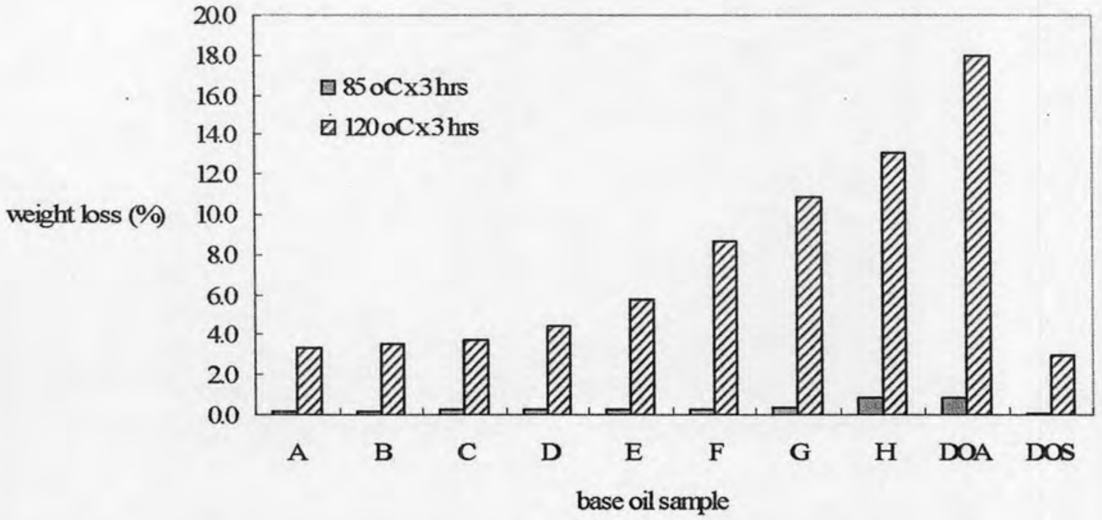


Figure 4.2 Plot of several base oil samples versus % weight loss

As shown in table 4.4, kinematic viscosity at 40 °C of sample B to sample H were within the target range (8 – 10 mm^2/s). However, sample B was likely to be out of target range when additives were added, so sample B was rejected. Besides, weight loss of sample H was very high when tested at 120 °C for 3 hours, so sample H was also rejected. Thus, samples C to sample G were selected to continue for further tests as shown in table 4.5, Fig.4.3 and Fig.4.4.

Table 4.5 Summarized results in all testing items of base oil samples

Items		Unit	Base oil sample				
			C	D	E	F	G
Total acid number		mgKOH/g	0.0495	0.0550	0.0573	0.0591	0.0664
Flash point		°C	240	244	242	238	240
Pour point		°C	below -60				
Moisture content		ppm	559	556	593	608	603
Specific gravity		-	0.9160	0.9168	0.9192	0.9213	0.9242
Corrosive outgas by Cu-Ag coupon test		-	Nondetection of any corrosive elements				
Corrosion by Cu plate test		-	1a				
Outgas analysis by GC/MS	85 °C x3 hrs	ng/g	841	953	1,235	1,301	1,366
	120 °C x3 hrs		1,957	2,348	2,403	2,516	2,653
Wear scar by four ball wear test		mm.	0.61	0.61	0.62	0.64	0.65
Friction coefficient by SRV	-20 °C	-	0.135	0.134	0.133	0.134	0.132
	50 °C		0.118	0.117	0.115	0.115	0.114
Oxidation induction time by RBOT		minute(s)	50	55	56	53	48

According to table 4.5, each result is discussed as following.

Total acid number (TAN)

Total acid number of each base oil sample was calculated by using equation A2 and the results are summarized in Fig. 4.3.

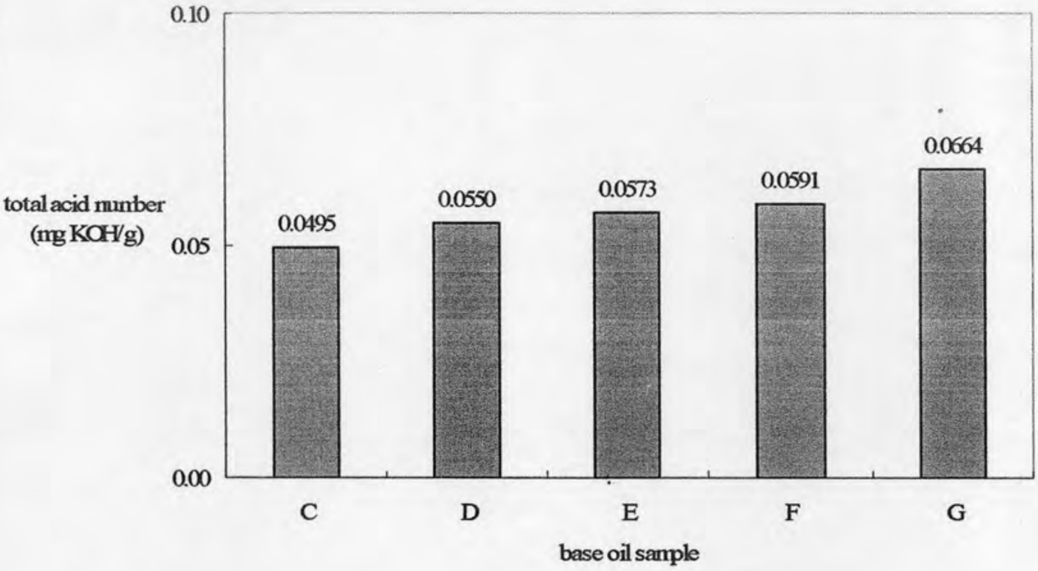


Figure 4.3 Plot of several base oil samples versus total acid number

The total acid number indicated that the acidity was slightly increased with increasing amount of DOA oil as in order of sample C to sample G. Because the acidic character of DOA oil is higher than DOS oil (refer table 4.3), so it might influent the total acid number of mixture samples C, D, E, F and G, respectively. However, total acid number were not significantly different.

Flash point

From table 4.5, flash point of each sample was not significantly different and all samples were still within acceptable difference (8 °C).

Pour point

These samples were prepared from DOA and DOS oil. Thus, flow ability at low temperature of all samples were excellent and also they were lower than detection limit of pour point instrument.

Remark : detection limit of pour point instrument is -60 °C.

Moisture content

Based on moisture content results as shown in table 4.5, all base oil samples had water content values not significantly different and they were still within acceptable difference (10%).

Specific gravity

Specific gravity increased from sample C to sample G with increasing amount DOA content in oil samples as shown in Fig. 4.4.

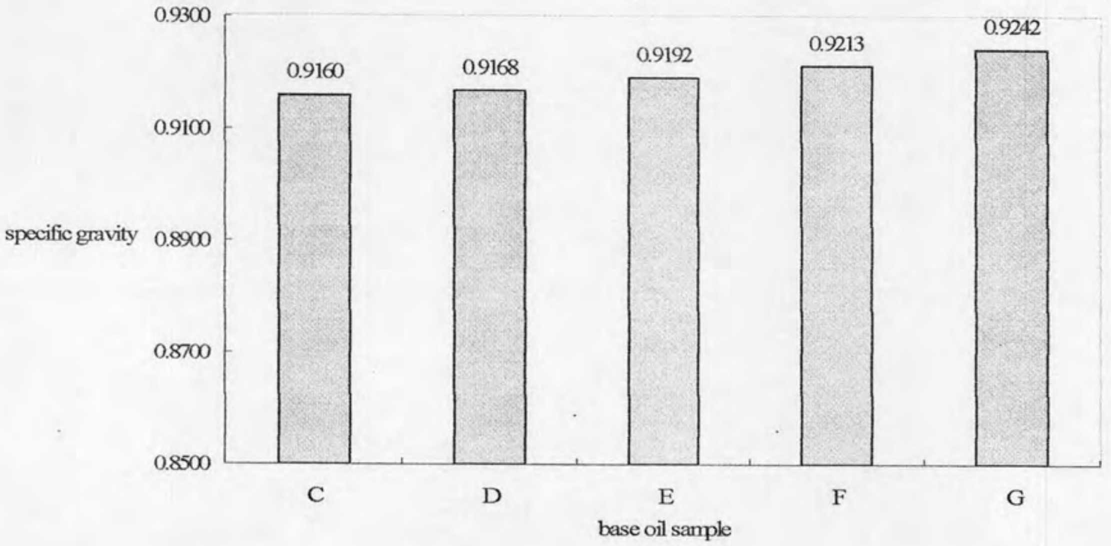


Figure 4.4 Plot of several base oil samples versus specific gravity

Cu-Ag coupon corrosion

After finished testing, surface of Cu and Ag were analyzed for corrosive elements such as sulfur which may be remained on their surface by SEM/EDX instrument. The spectra of Cu and Ag after tested of base oil samples are presented in Fig. 4.5 to Fig. 4.9.

Sample C

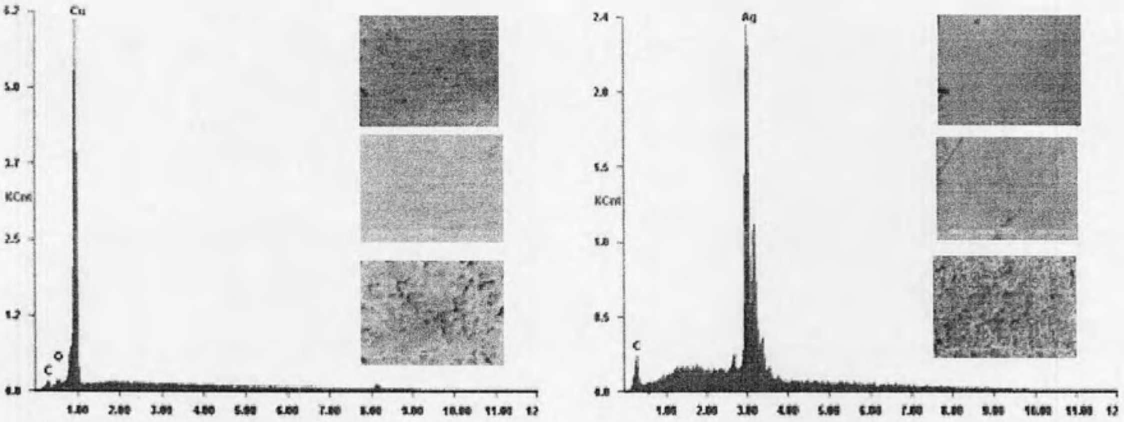


Figure 4.5 Surface analysis on Cu-Ag coupon of sample C by SEM/EDX

Sample D

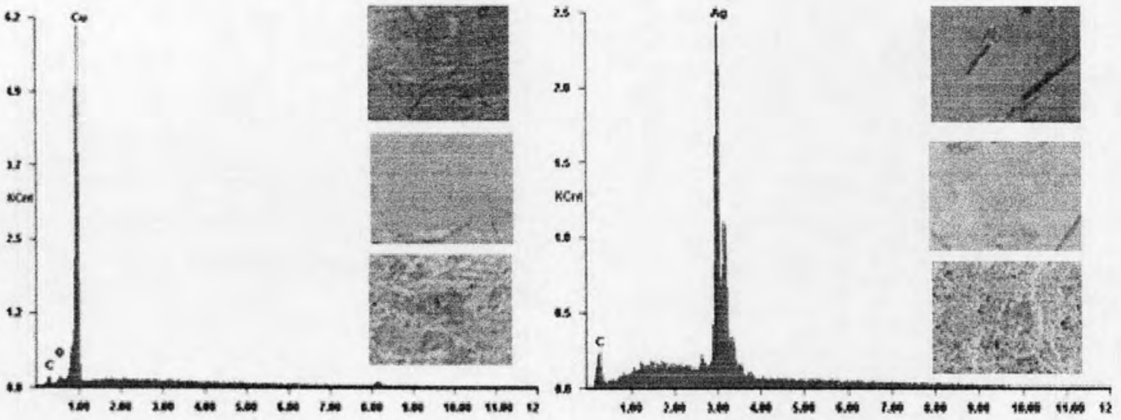


Figure 4.6 Surface analysis on Cu-Ag coupon of sample D by SEM/EDX

Sample E

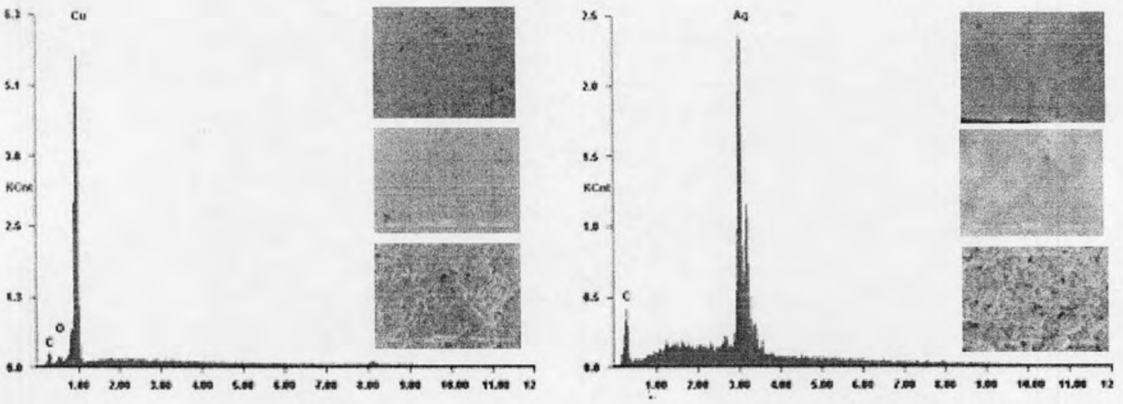


Figure 4.7 Surface analysis on Cu-Ag coupon of sample E by SEM/EDX

Sample F

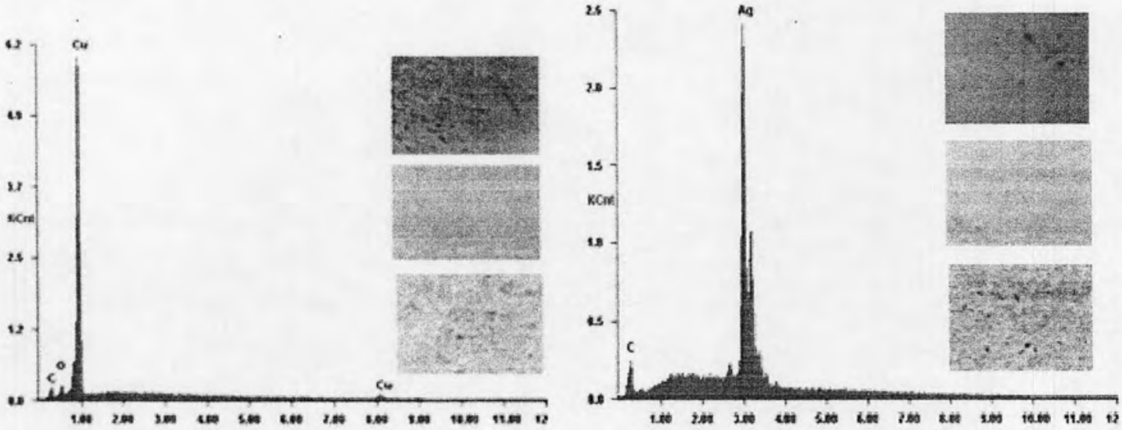


Figure 4.8 Surface analysis on Cu-Ag coupon of sample F by SEM/EDX

Sample G

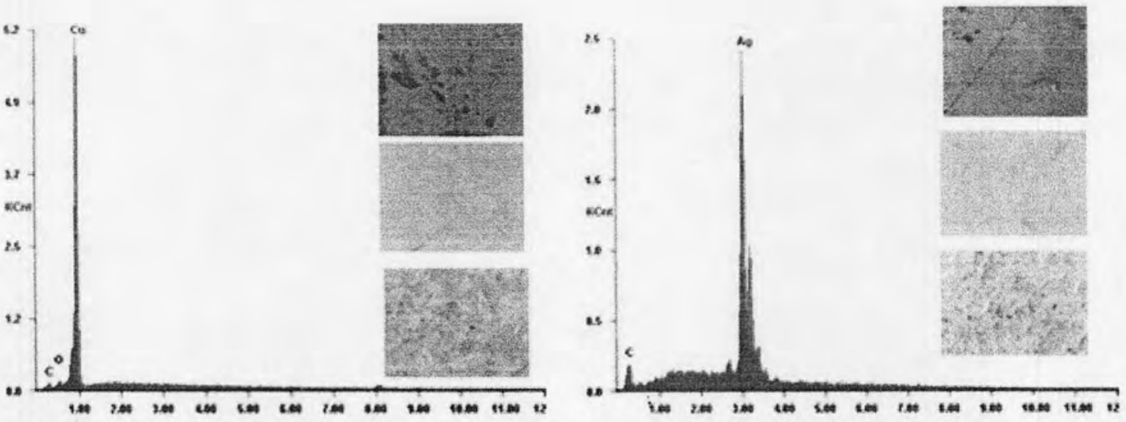


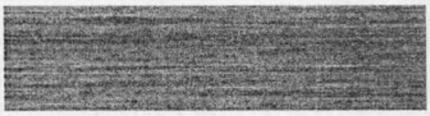
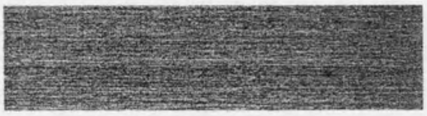
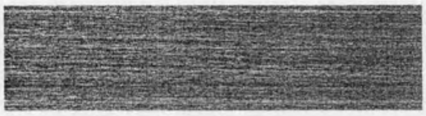
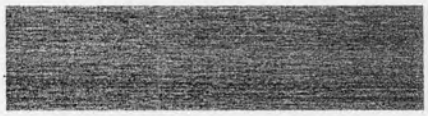
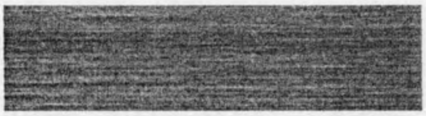
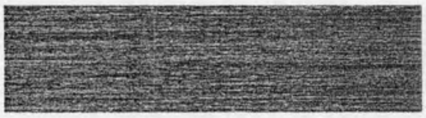
Figure 4.9 Surface analysis on Cu-Ag coupon of sample G by SEM/EDX

From surface analysis of each base oil sample by SEM/EDX instrument, it was found that there was no trace of sulfur corrosive outgas on surface of Cu and Ag coupon after tested with each base oil sample. Therefore, it could be concluded that these base oil samples did not contain any substances which might generate corrosive outgas to damage surface of bearing's components.

Cu plate corrosion

From Cu plate corrosion test result as shown in table 4.6, it was found that these base oil samples were in class 1 when compared with ASTM corrosion standard. Thus, these base oils would not cause any corrosion on surface of metal.

Table 4.6 Pictures of Cu plate corrosive results of base oil samples

Sample	Picture of Cu plate after tested	Corrosive level
Blank		-
C		1a
D		1a
E		1a
F		1a
G		1a

Outgas analysis

The outgas of base oil samples increased from sample C to sample G; respectively as shown in Fig 4.10. Presumably, it was from DOA evaporation.

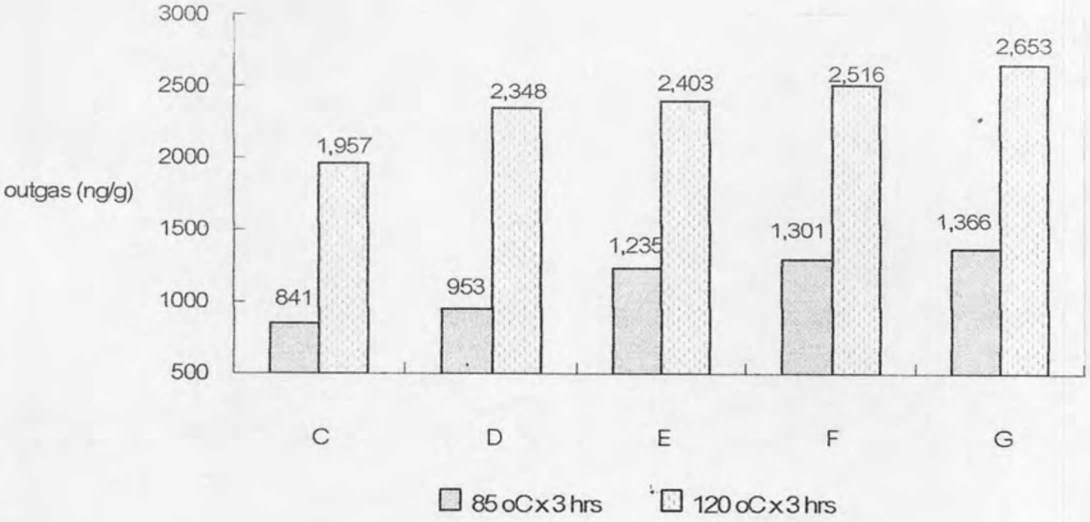


Figure 4.10 Plot of several base oil samples versus outgas

Wear scar by Four ball wear tester

The wear scar on surface of metal when each base oil sample was used to lubricate, were found to increase from sample C to sample G; respectively as shown in Fig. 4.11. It was resulted from evaporation lose, so lubricity performance was decreased. However, size of wear scar from all base oil samples were still within 10% acceptable difference, thus lubricity performance of all base oil samples were not significantly different as shown in table B1.

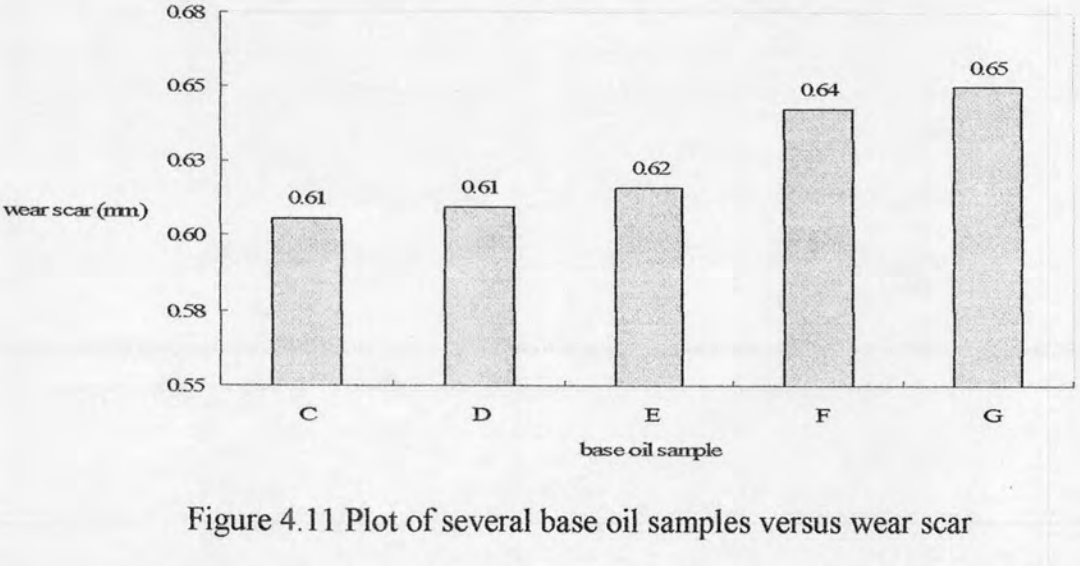


Figure 4.11 Plot of several base oil samples versus wear scar

Friction coefficient by SRV instrument

Friction coefficient of each base oil sample was measured and recorded by SRV instrument and typical result of friction coefficient is shown in Fig. B1 to Fig. B10. However, friction coefficient results as shown in table 4.5 of all base oil samples were not significantly different and they were still within 10% acceptable difference.

Oxidation induction time by RBOT instrument

Normally, acceptable difference allows ± 30 minutes when measuring oxidation induction time by RBOT. Thus, oxidation induction time of all base oil samples as shown in table 4.5, were not significantly different.

From table 4.5, the 4 suitable base oils were selected to test for the next step which was additive addition. The selection of suitable base oils were selected based on number of selection as summarized in Fig. 4.12.

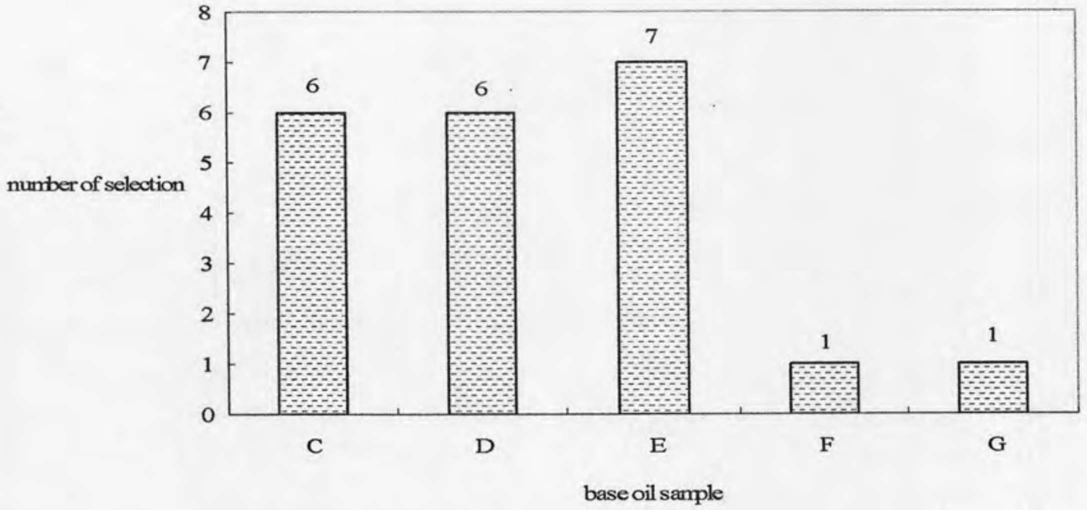


Figure 4.12 Plot of base oil sample versus number of selection

Samples F and G got the lowest selection items (only 1), thus both samples were eliminated.

4.2 Investigation of the suitable base oils for finished lubricants preparation

4.2.1 Finished lubricants preparation

In this step, the selected base oil samples' samples C to E were blended with several additives in different percentage as shown in table 4.7.

Table 4.7 Type and percentage of additives

Type of additive	% Blending of additive
Antioxidant	1%
Antiwear	0.5%
Metal deactivator	0.05%

The finished lubricants which were designated as samples I to III and two commercial finished lubricants which are currently use in FDB spindle motors are shown in table 4.8.

4.2.2 Properties assessment of finished lubricants

Initially, finished lubricants were analyzed for 2 items which are kinematic viscosity which should be in range of 8 – 10 mm^2/s at 40 °C and weight loss by TG/DTA. The testing results of finished lubricant samples were compared with those of commercial A and commercial B as shown in table 4.8.

Table 4.8 Kinematic viscosity and weight loss of finished lubricants

Sample designation	**Base oil sample**	**Kinematic viscosity (unit : mm^2/s)**		**%weight loss (unit : %)**	
		40 °C	**100 °C**	**85 °C x 3 hrs**	**120 °C x 3 hrs**
I	C	10.07	3.12	0.245	5.742
II	D	9.86	3.09	0.279	5.914
III	E	9.62	3.05	0.284	6.735
Commercial A	Ester	12.72	3.22	0.315	4.901
Commercial B	Ester	9.59	3.04	0.325	7.149

Remark :

1. Commercial A is lubricating oil which currently use in FDB spindle motors for 3.5 inch HDD.
2. Commercial B is lubricating oil which currently use in FDB spindle motors for 2.5 inch HDD.

From table 4.8, results of kinematic viscosity and % weight loss of each finished lubricant can be summarized in terms of charts as shown in Fig. 4.13 and Fig. 4.14, respectively.

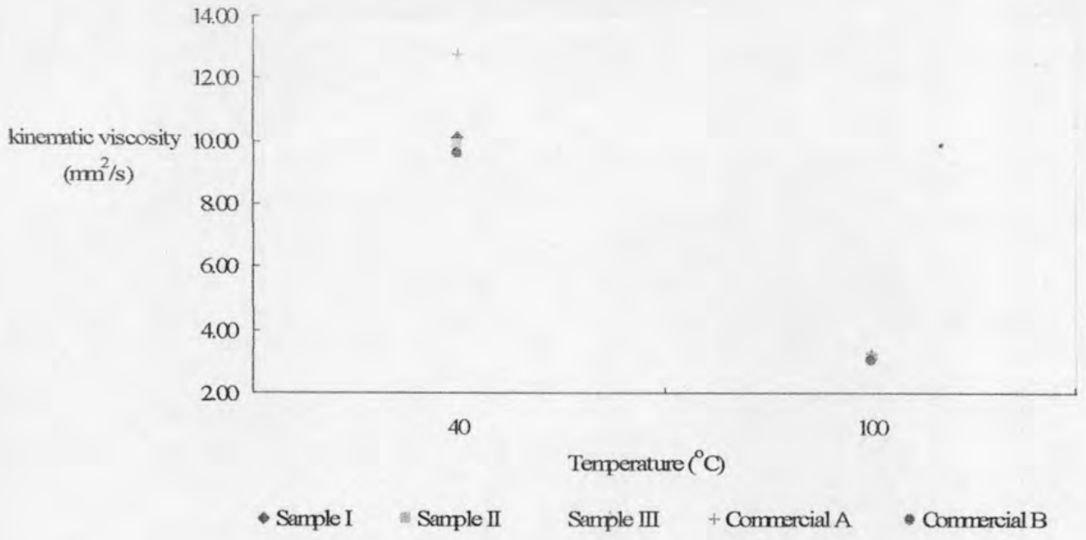


Figure 4.13 Plot of testing temperature versus kinematic viscosity of several finished lubricants

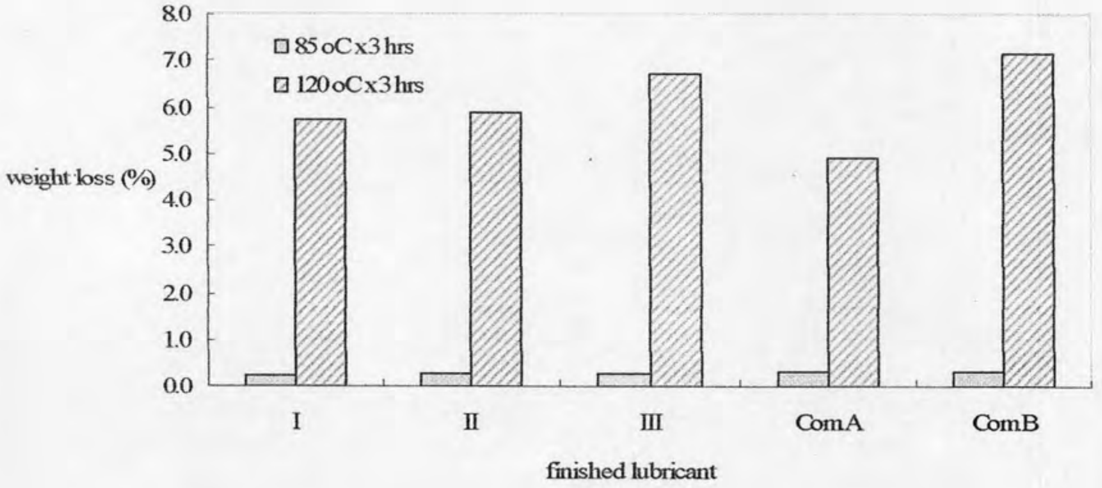


Figure 4.14 Plot of several finished lubricants versus % weight loss

According to kinematic viscosity at 40 °C of sample I, it was out of the target range (8 – 10 mm^2/s). Therefore, sample I was not suitable for use in FDB spindle motor for 2.5 inch HDD. Thus, samples II and III would be assessed in other properties.

To more understand the effect from chemical composition of each finished lubricant, the main chemical composition of each finished lubricant in samples II, III and commercial B were characterized by FTIR instrument and GC/MS instrument as shown in Fig. 4.15 to Fig. 4.20.

Sample II

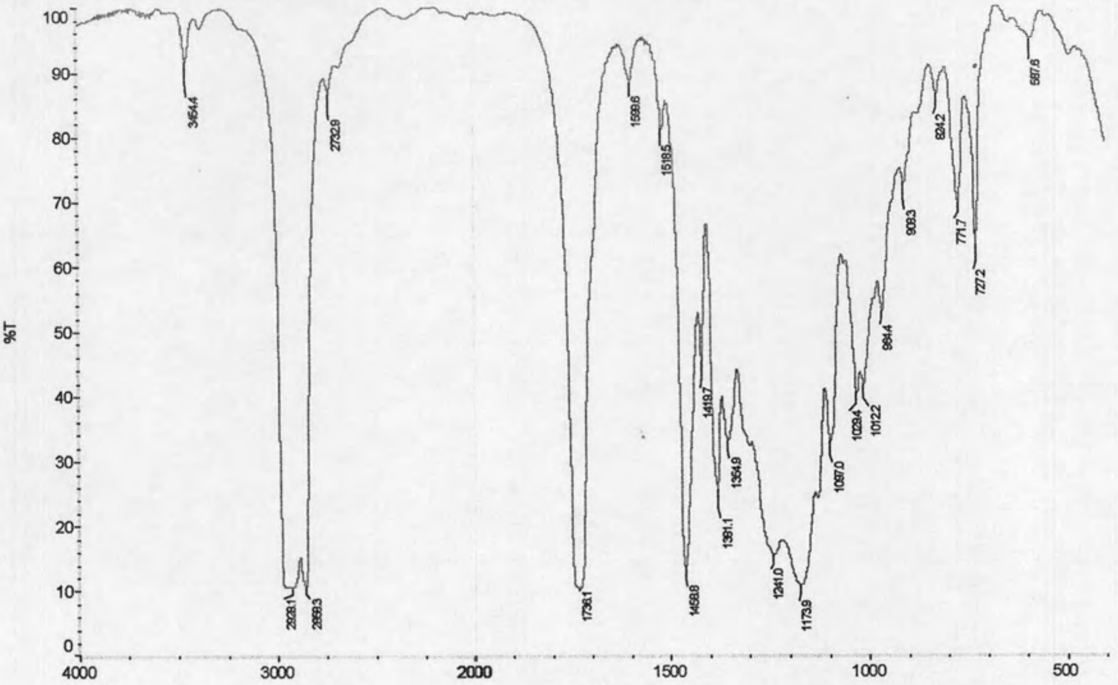


Figure 4.15 FTIR spectrum of sample II by FTIR

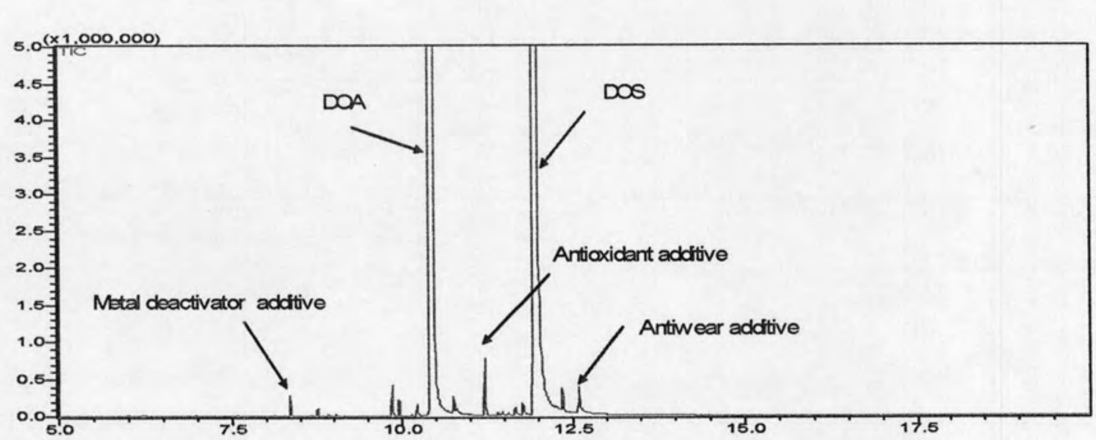


Figure 4.16 The total ion chromatogram of sample II by GC/MS

Sample III

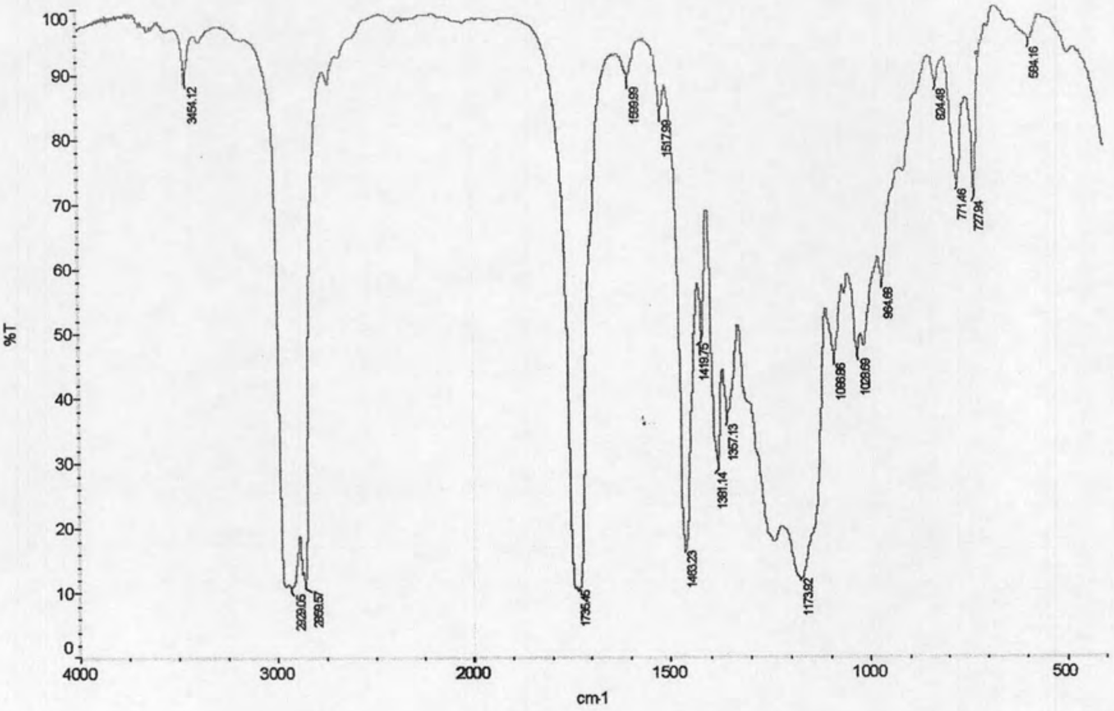


Figure 4.17 FTIR spectrum of sample III by FTIR

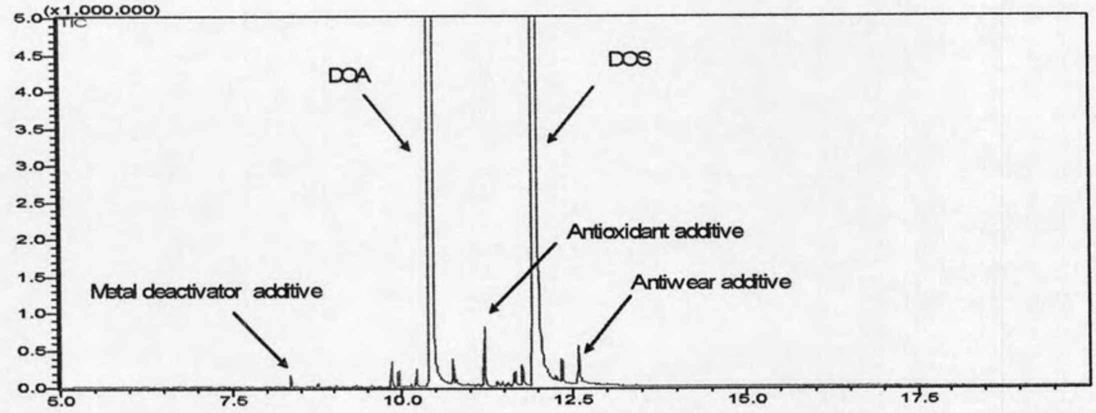


Figure 4.18 The total ion chromatogram of sample III by GC/MS

Commercial B

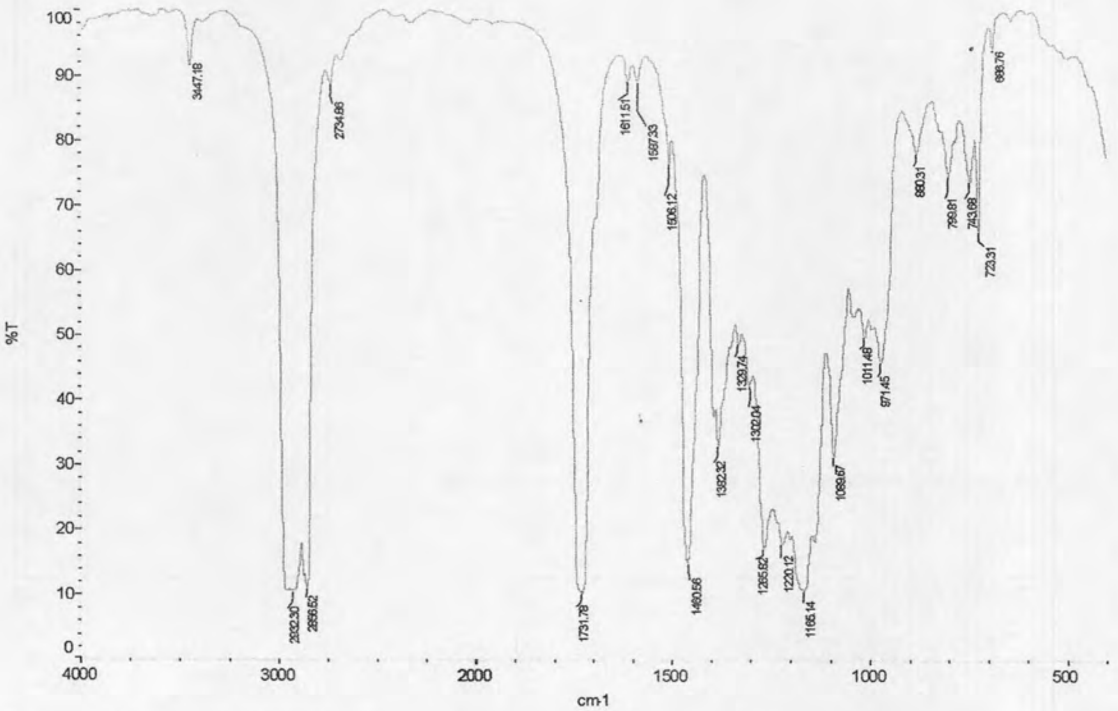


Figure 4.19 FTIR spectrum of commercial B by FTIR

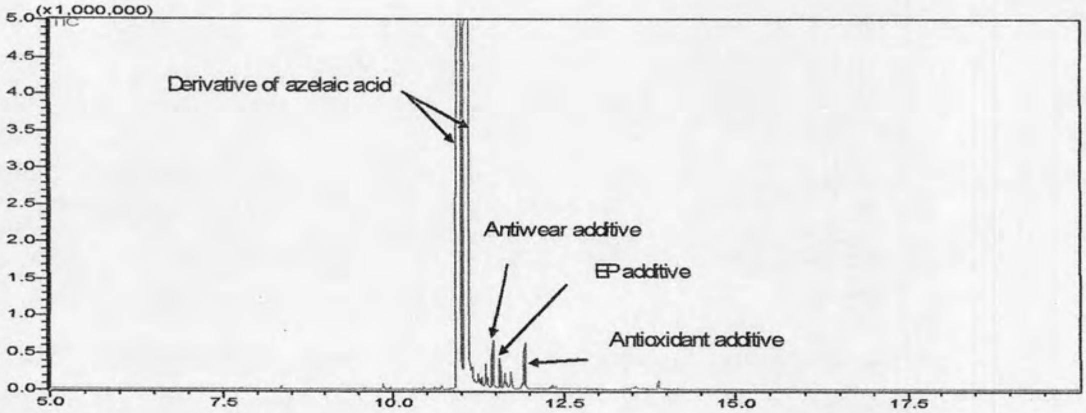


Figure 4.20 The total ion chromatogram of commercial B by GC/MS

According to IR spectra of samples II and III, the peak at wave no. 3,464 cm^{-1} and 1,599 cm^{-1} were assigned to be aromatic amine antioxidant additive. In terms of total ion chromatogram (TIC) from GC/MS analysis, DOA base oil was detected for the base peak at m/z = 171 and DOS base oil at base peak at m/z = 185. Moreover, alkylated diphenylamine as antioxidant additive, triphenyl phosphate as antiwear additive and tolutriazole compound as metal deactivator were also detected at base peak at m/z = 266, m/z =77, m/z =142, respectively.

In case of commercial B, IR spectrum showed characteristic peak of aromatic amine compound as antioxidant additive at wave no. 3,447 cm^{-1} and 1,587 cm^{-1}. Besides, FTIR also detected sulfurized ester as extreme pressure (EP) additive at wave no. 1,731 cm^{-1} and 1,611 cm^{-1}. GC/MS analysis indicated the derivative compound of azelic acid at base peak m/z = 171. Moreover, 4,4-dioctyldiphenylamine (antioxidant additive), thiazol compound (EP additive) and tricresyl phosphate (antiwear additive) were also detected at base peak m/z = 322, m/z = 213, m/z = 368, respectively.

The summary of analysis result of samples II and III are shown in table 4.9.

Table 4.9 Summarized results in all testing items of finished lubricants

Items		Unit	Finished lubricant		
			II	**III**	**Com.B**
Total acid number		mgKOH/g	0.0852	0.1214	0.1815
Flash point		°C	240	242	232
Pour point		°C	below -60		
Moisture content		ppm	711	689	654
Specific gravity		-	0.9188	0.9213	0.9182
Corrosive outgas by Cu-Ag coupon test		-	Nondetection of any corrosive elements		
Corrosion by Cu plate test		-	1b		2a
Outgas analysis by GC/MS	85 °C x3 hrs.	ng/g	1,056	1,372	1,630
	120 °C x3 hrs.		2,918	3,051	3,125
Wear scar by Four ball wear tester		mm.	0.40	0.37	0.30
Friction coefficient by SRV	-20 °C	-	0.110	0.108	0.098
	50 °C		0.107	0.105	0.094
Oxidation induction time by RBOT		minute(s)	1,253	1,269	1,495

Total acid number

Total acid number results are summarized as shown in Fig. 4.21.

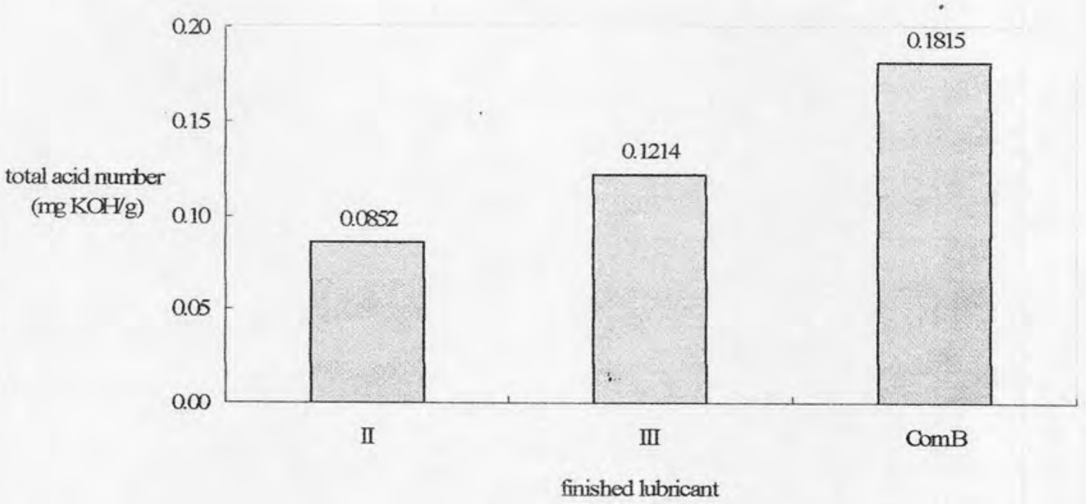


Figure 4.21 Plot of finished lubricants versus total acid number

The total acid number results indicated that the acidic property increased from samples II, sample III and commercial B; respectively. Sample III had higher acidic property than sample II because of base oil's character. In terms of commercial B which had the highest acidic property, this finished lubricant was presumably used high acidic type of extreme pressure additive such as sulfurized ester.

Flash point

Referring to table 4.9, flash point of commercial B was lower than those of other finished lubricants because of base oil's character.

Pour point

These finished lubricants were prepared from synthetic ester oil, thus flow ability at low temperature of all samples were excellent and also lower than detection limit of pour point instrument which is -60 °C.

Moisture content

Moisture content of all finished lubricants were not significantly different and they were still within 10% acceptable difference.

Specific gravity

Specific gravity increased from samples II, III and commercial B; respectively as shown in Fig. 4.22. In this matter, it may be explained by base oil's character which DOA has specific gravity higher than that of DOS and it also may be higher than that of derivative compound of azelic acid which is used as base oil for commercial B.

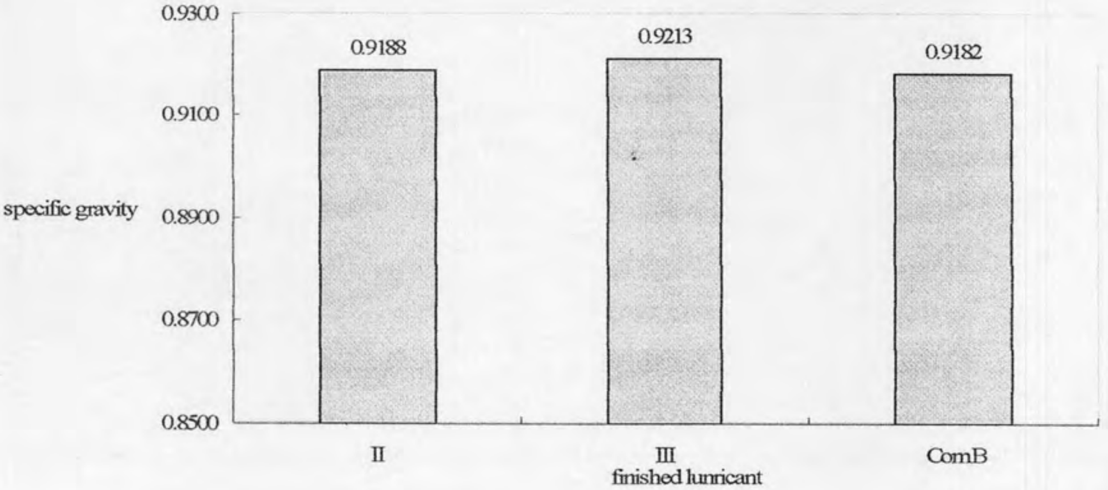


Figure 4.22 Plot of several finished lubricants versus specific gravity

Cu-Ag coupon corrosion

After finished testing, surface of Cu and Ag were analyzed for element such as sulfur element which may be remained on their surfaces by SEM/EDX instrument, if corrosion occurred. The spectra of Cu and Ag after tested with each finished lubricant are presented in Fig. 4.23 to Fig. 4.25.

Sample II

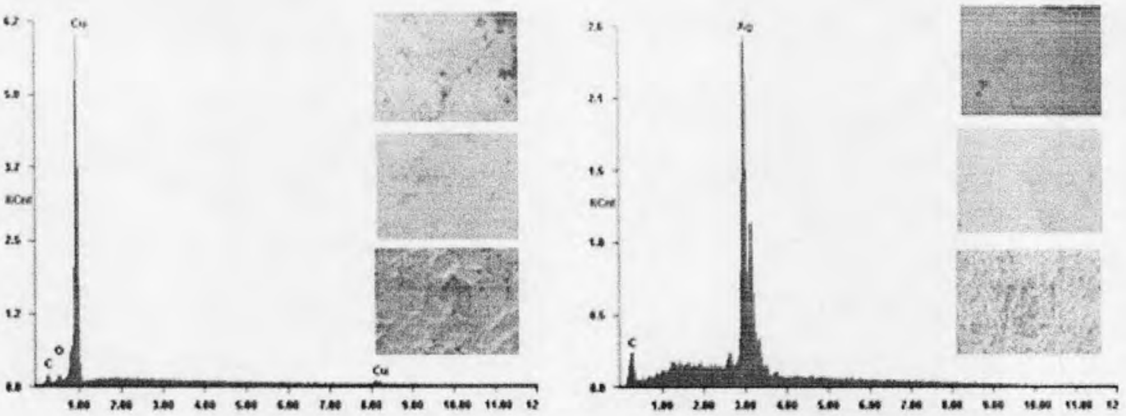


Figure 4.23 Surface analysis on Cu-Ag coupon of sample II by SEM/EDX

Sample III

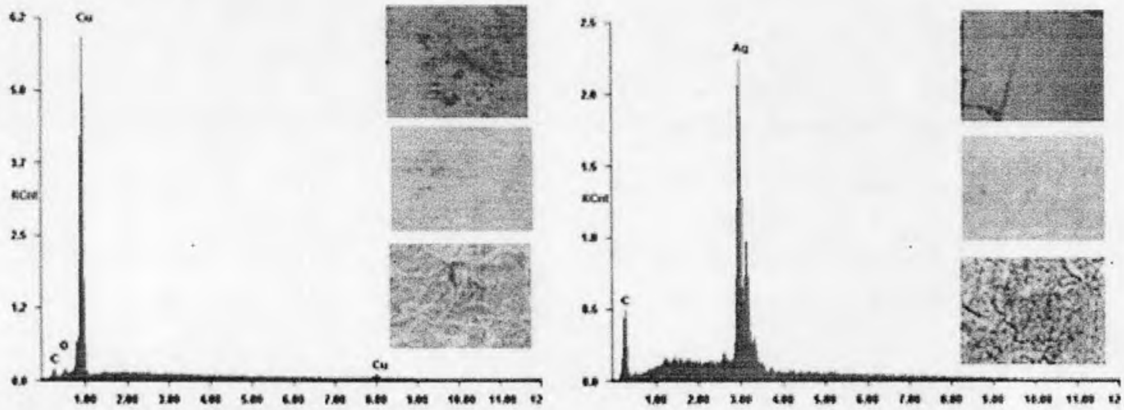


Figure 4.24 Surface analysis on Cu-Ag coupon of sample III by SEM/EDX

Commercial B

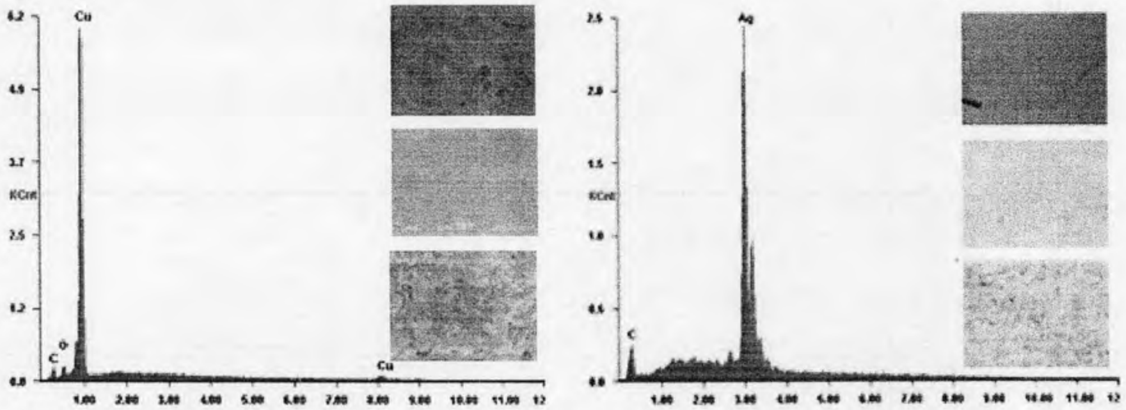


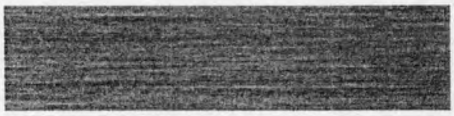
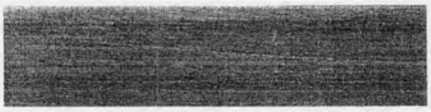
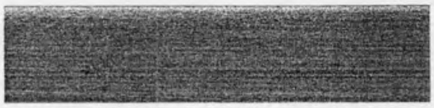
Figure 4.25 Surface analysis on Cu-Ag coupon of commercial B by SEM/EDX

According to surface analysis results, SEM/EDX instrument could not detect any sulfur corrosive outgas on surface of Cu and Ag coupon after test with all finished lubricants. Therefore, it could be concluded that the base oil or additives in these finished lubricants were not generated any corrosive outgas to damage surface of bearing's components.

Cu plate corrosion

From Cu plate corrosion test as shown in table 4.10, it was found that samples II and III were in class 1 when compared with ASTM corrosion standard. However, commercial B was in class 2 which could be from the effect of sulfurized ester use as EP additive. However, corrosion in class 2 was acceptable as noncorrosion on surface of materials.

Table 4.10 Pictures of Cu plate corrosive results of finished lubricants

Sample	Picture of Cu plate after tested	Corrosive level
Blank		-
II		1b
III		1b
Commercial B		2a

Outgas analysis

The outgas results as shown in Fig. 4.26 showed the tendency of increasing from samples II, III and commercial B; respectively. In case of samples II and III, it could come from DOA oil evaporation. For commercial B which had the highest outgas, it could be explained that this finished lubricant use more volatile base oil which was derivatives of azelic acid. The kinematic viscosity result also indicated that commercial B had lower viscosity than those of samples II, and III.

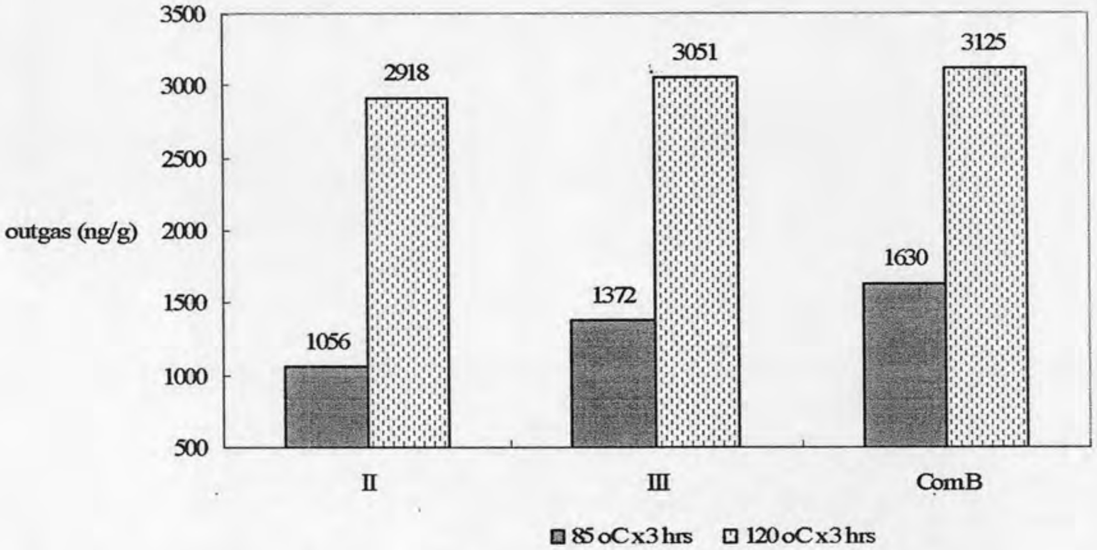


Figure 4.26 Plot of several finished lubricants versus outgas

Wear scar by Four ball wear tester

The wear scar were increased from samples II, III and commercial B; respectively as shown in Fig. 4.27. However, in case of samples II and III it was found that the wear scar of them were within 10% acceptable difference, so it could be concluded that samples II and III had the same performance of antiwear property. Moreover, size of wear scar when used samples II and III to lubricate on testing work piece was smaller than that of neat base oil. Commercial B was found to contain sulfurized ester (EP additive), so this additive could reduce metal-metal contacting. Wear scar of them are shown in table B1.

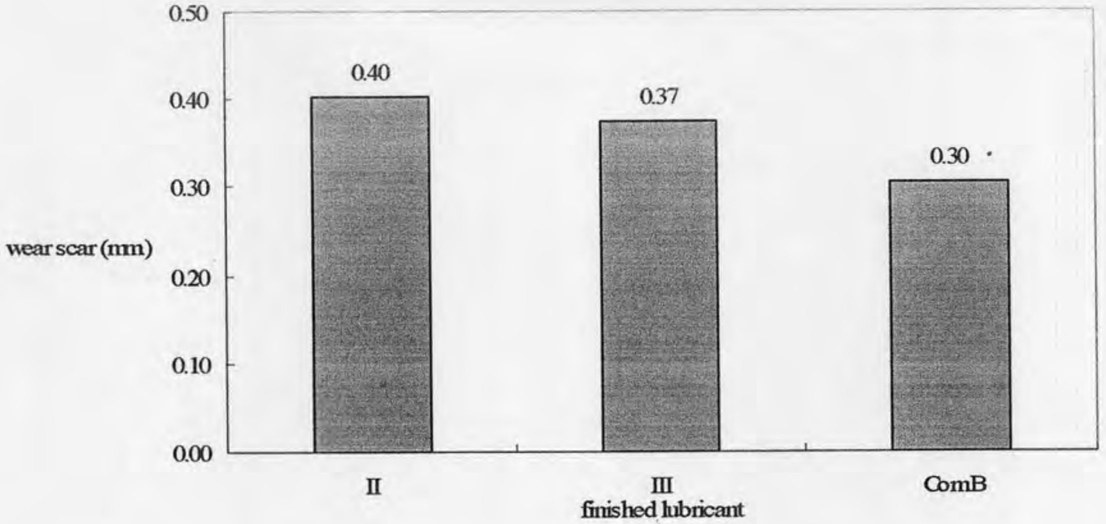


Figure 4.27 Plot of several finished lubricants versus wear scar

Friction coefficient by SRV instrument

The friction coefficient were decreased from samples II, III and commercial B; respectively. However, in case of samples II and III it was found that the friction coefficient value were still within 10% acceptable difference, so it could be concluded that samples II and III had the same lubricity performance. For friction coefficient of commercial B, it was found to be lower than those of samples II and III because it contained high lubricity performance additive such as EP additive. Thus, commercial B gave better lubricity than samples II and III.

Friction coefficient of each finished lubricant was measured and recorded by the program of SRV instrument and the typical results of friction coefficient are shown in Fig. B11 to Fig. B14.

Oxidation induction time by RBOT instrument

Based on oxidation induction time (OIT) as shown in Fig. 4.28, sample II had oxidative stability property poorer than those of sample III and commercial B; respectively. However, the oxidation induction time of both samples were not significantly different and within acceptable difference allows as ± 30 minutes. Probably commercial B contained another type of antioxidant additives, thus the oxidation property of it was better than those of samples II and III.

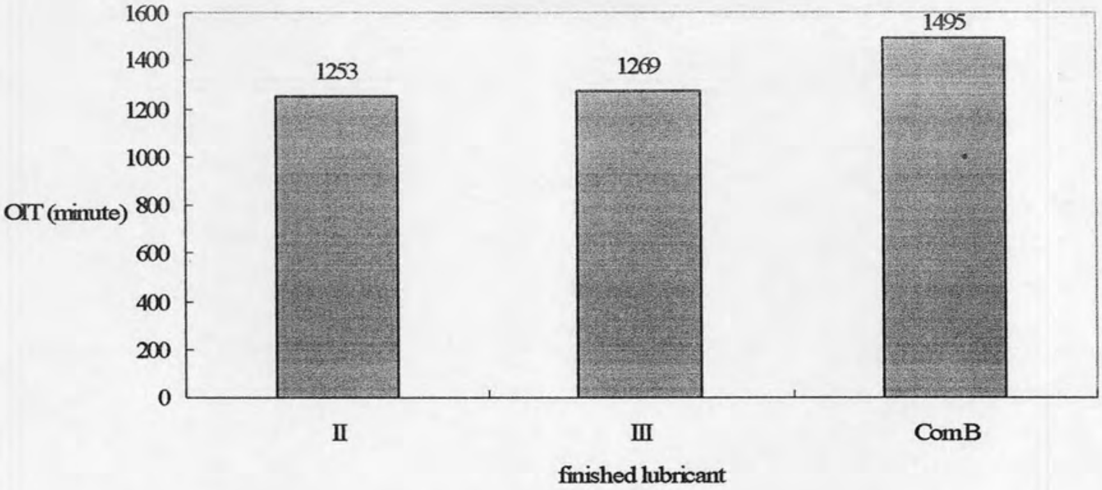


Figure 4.28 Plot of several finished lubricants versus OIT

Evaporation loss

As shown in Fig. 4.29, the percentage of evaporation loss of samples II and III were lower than that of commercial B as shown in table B2.

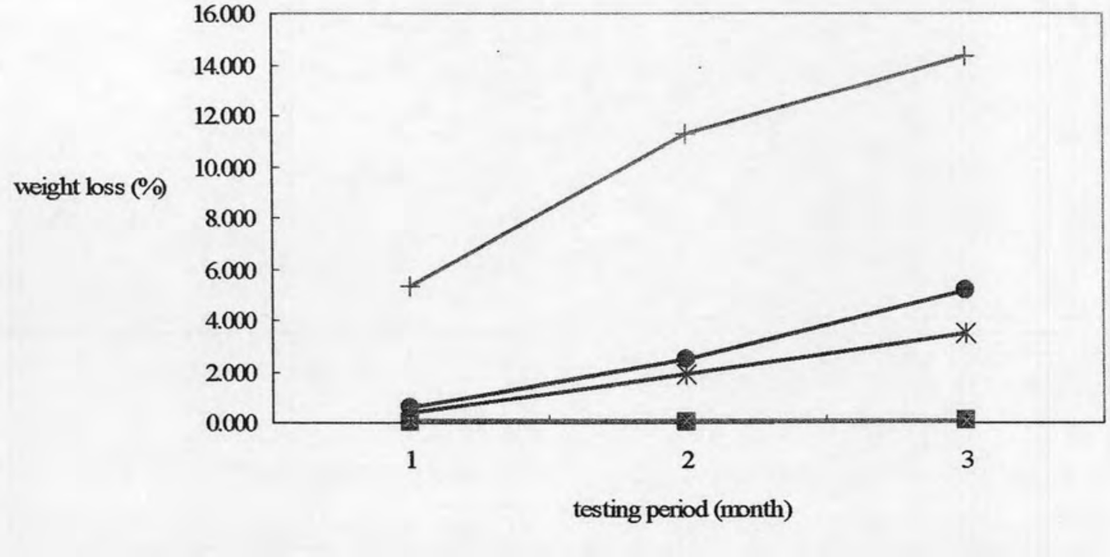


Figure 4.29 Percentage of weight loss at 60 °C and 120 °C for several testing period

Compatibility test

Bearing parts after compatibility test were subjected to surface analysis by microscope. Moreover, finished lubricants were also analyzed for total acid number and the results of these testing items are shown in table 4.11 and Fig. 4.30, respectively.

Table 4.11 Visual check of bearing part after compatibility test by microscope

Testing condition		**Finished lubricant**		
		Sample II	**Sample III**	**Commercial B**
60 °C	1 month	O	O	C
	2 months	O	O	C
	3 months	O	O	C
120 °C	1 month	O	O	C
	2 months	C	C	C
	3 months	C	C	X

Remark :

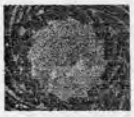
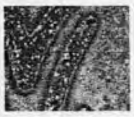
O represents no stain or discoloration on bearing part

C represents discoloration on bearing part

X represents some stain on bearing part

The picture of bearing parts before and after compatibility test are shown in table 4.12.

Table 4.12 Picture of bearing part after compatibility test at 60 °C and 120 °C by microscope

Condition test	Sample II		Sample III		Commercial B	
	x10 magnification	x100 magnification	x10 magnification	x100 magnification	x10 magnification	x100 magnification
before test						
60 °C x 1 month						
60 °C x 2 months						
60 °C x 3 months						
before test						
120 °C x 1 month						
120 °C x 2 months						
120 °C x 3 months						

From table 4.12, bearing part when tested at 60 °C for testing period of 3 months with samples II and III were found to contain no change. However, bearing parts that baked with commercial B at the same testing condition, were found to have color change. The color change of these bearing parts may be from sulfurized ester EP additive's effect on surface of the part. In case of high temperature testing at 120 °C, the bearing part that baked with samples II and III, were also showed color change.

Besides, finished lubricants after compatibility test were also tested for total acid number and it was found that commercial B had lower total acid number than samples II and III. Due to commercial B contained high performance antioxidant additive, so it could be more retarded oxidation reaction. Therefore, acidic products from commercial B should be also lower than those of samples II and III at both conditions as shown in table B3 and Fig. 4.30.

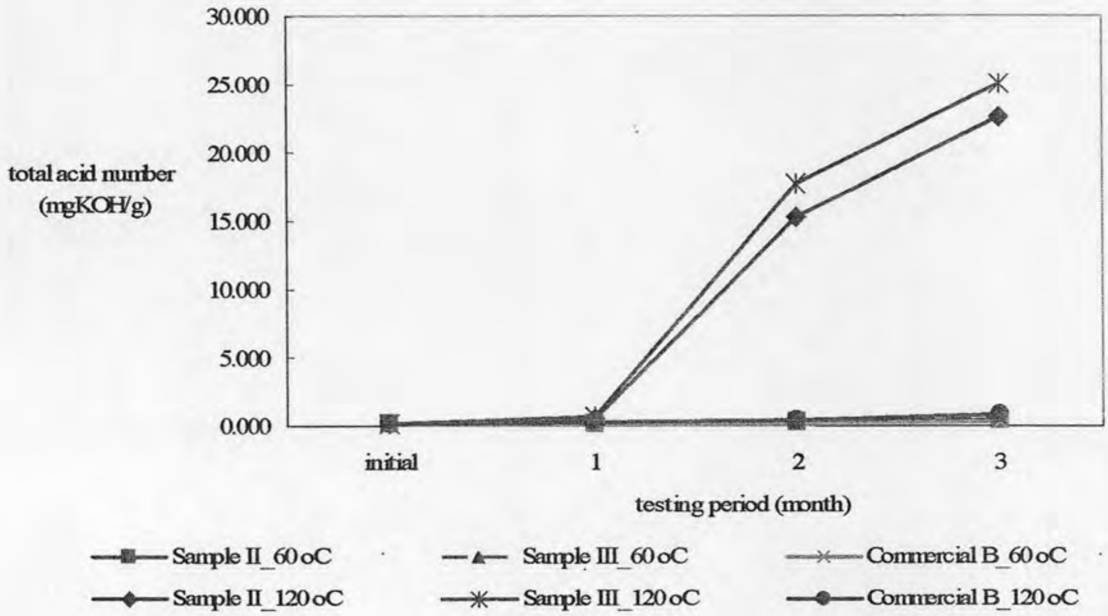


Figure 4.30 Total acid number of finished lubricants after compatibility test at 60 °C and 120 °C for three months testing period

Service life test

The finished lubricants were applied into FDB spindle motor for 2.5 inch HDD and then run in the control oven at 80 °C and 100 °C for 3 months. The life test result was presented in term of running current which motor used for running as shown in table B4 and table B5.

Both testing conditions at 80 °C and 100 °C are shown in Fig. 4.31 and table 4.13. It was found that the slope of commercial B was higher than those of samples II and III which means that commercial B consumed more current during testing for 3 months than samples II and III. It is possible that commercial B had high evaporation loss, therefore, viscosity was increased which resulted in using more power to run at boundary condition.

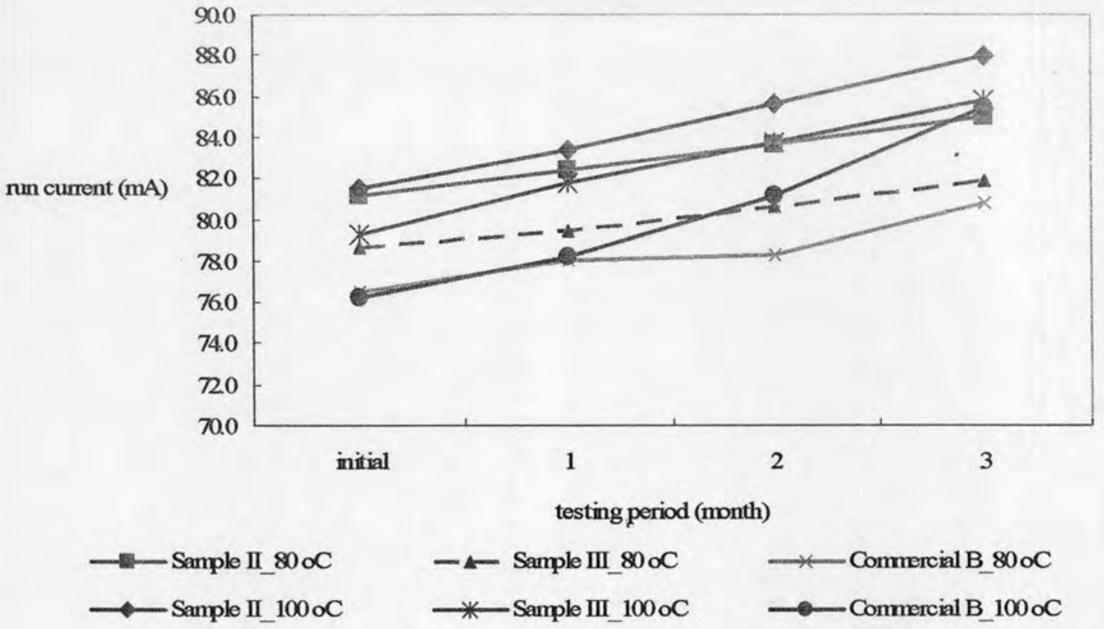


Fig.4.31 Plot of running current of motor at 80 °C and 100 °C versus several testing period of each finished lubricants

Table 4.13 Equation of graph for finished lubricant after life test

Testing condition	**Finished lubricant**	**Equation of graph**
80 °C	Sample II	y = 1.284X + 79.82
	Sample III	y = 1.102X + 77.35
	Commercial B	y = 1.320X + 75.07
100 °C	Sample II	y = 2.176X + 79.18
	Sample III	y = 2.134X + 77.28
	Commercial B	y = 3.058X + 72.58

According to results of wear scar and oxidation induction time as shown in table 4.9, samples II and sample III were poorer performances than commercial B. Therefore, these finished lubricant samples should be improved by adding more amount of antioxidant additive and antiwear additive which would explain in section 4.3.

4.3 Performance improvement of finished lubricant

Due to samples II and III having low performance on lubricity and oxidation stability properties, more antioxidant additive and antiwear were added into these finished lubricants.

4.3.1 Optimization of percentage of additive

This step was separated into 2 parts. First, only antioxidant additive was added at different percentage in sample II and then oxidation induction time and kinematic viscosity at 40 °C were measured. The testing results are shown in table 4.14. Next, antiwear additive was added at different percentage into sample II and then wear scar and kinematic viscosity at 40 °C were measured. The testing results are shown in table 4.15.

Table 4.14 Testing result of each sample after addition of antioxidant additive

Testing item	**% antioxidant additive in sample II**				**Com. B**
	1% (Original)	**1.5%**	**2%**	**2.5%**	
Oxidation induction time by RBOT (minute)	1,253	1,327	1,558	1,663	1,495
Kinematic viscosity at 40 °C (mm^2/s)	9.86	9.88	9.90	9.93	9.59

Table 4.15 Testing result of each sample after addition of antiwear additive

Testing item	**% antiwear additive in sample II**				**Com. B**
	0.5% (Original)	**1%**	**1.5%**	**2%**	
Wear scar by Four balls wear tester (mm.)	0.40	0.33	0.33	0.32	0.30
Kinematic viscosity at 40 °C (mm^2/s)	9.86	9.89	9.93	9.95	9.59

According to result of oxidation induction time and wear scar, oxidation stability property of sample II were increased with increasing percentage of antioxidant additive. However, kinematic viscosity was also found to increase when increasing the amount of additive. In term of wear scar results of each sample, it was not significantly changed when increased the amount of antiwear additive. Therefore, the optimization of antioxidant additive and antiwear were 2% and 1%, respectively.

Table 4.16 Type and percentage of additives in improving of finished lubricants

Type of additive	**% Blending of additive**	
	Previous (refer table 4.9)	**New addition**
Antioxidant	1%	2%
Antiwear	0.5%	1%
Metal deactivator	0.05%	0.05%

The improved finished lubricants with higher amount of antioxidant additive and antiwear were designated as samples V and VI.

4.3.2 Properties assessment for finished lubricants

Initially, samples V and VI were analyzed for wear scar which measured by four balls wear tester and oxidation induction time by RBOT instrument. The results were compared with those of commercial B.

Wear scar by Four ball wear tester

Because more amount of antiwear additive (from 0.5 % to 1%) were added to samples V and VI, so the lubricity performance was improved. Besides, wear scar of them were also nearly the same size of commercial B which were within 10% acceptable difference as shown in table 4.17.

Oxidation induction time by RBOT instrument

In term of oxidation stability, samples V and VI were found to endure oxidation reaction longer than that of commercial B as shown in Fig. 4.32.

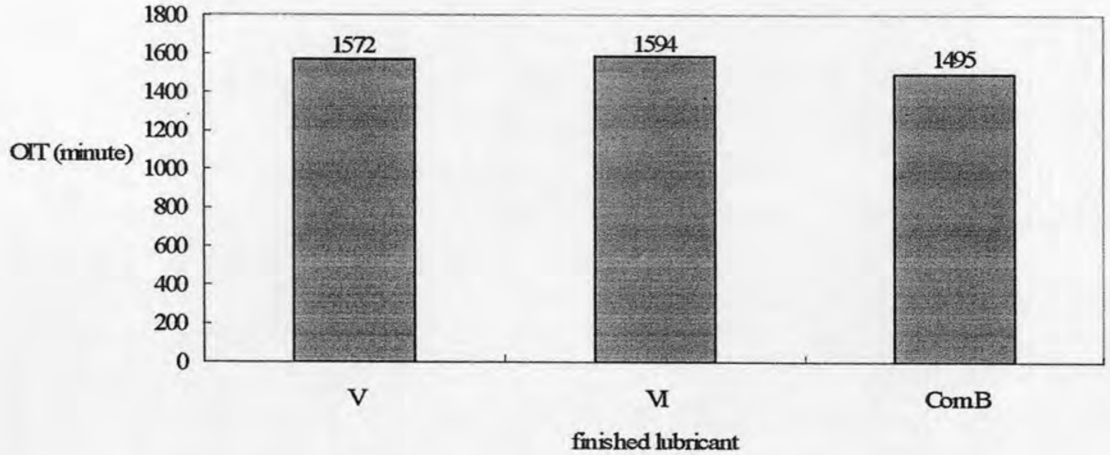


Figure 4.32 Plot of several finished lubricant versus OIT

Because the oxidation stability as shown by oxidation induction time of samples V and VI which were longer than that of commercial B, so it could be concluded that both samples had better performance than commercial B in term of service life.

Next, finished lubricants were analyzed for kinematic viscosity which should be in the range of 8 – 10 mm^2/s at 40 °C and weight loss and the results of finished lubricant samples and commercial B were compared as shown in table 4.17.

Table 4.17 Wear scar, kinematic viscosity and weight loss of finished lubricants

Sample designation	**Wear scar (unit : mm.)**	**Kinematic viscosity (unit : mm^2/s)**		**%weight loss (unit : %)**	
		40 °C	**100 °C**	**85 °C x 3 hrs**	**120 °C x 3 hrs**
V	0.33	9.91	3.10	0.281	6.254
VI	0.31	9.67	3.06	0.289	6.985
Commercial B	0.30	9.59	3.04	0.325	7.149

Because more amount of antioxidant and antiwear additives were added into samples V and VI, so their kinematic viscosity at 40 °C were slightly higher than those before improvement. From this matter, the effect on sample V was close the upper limit of target range. Therefore, sample V was not suitable for use in FDB spindle motor for 2.5 inch HDD. For this reason, sample V was rejected and only sample VI would be assessed for other properties as shown in table 4.18.

Table 4.18 Characteristic properties of finished lubricants

Items		Unit	Finished lubricant	
			Sample VI	**Commercial B**
Total acid number		mgKOH/g	0.1276	0.1815
Flash point		°C	242	232
Pour point		°C	below -60	
Moisture content		ppm	725	654
Specific gravity		-	0.9219	0.9182
Corrosive outgas by Cu-Ag coupon test		-	Nondetection of any corrosive elements	
Corrosion by Cu plate test		-	1b	2a
Outgas analysis by GC/MS	85 °C x3 hrs.	ng/g	1,394	1,630
	120 °C x3 hrs.		3,086	3,125
Friction coefficient by SRV	-20 °C	-	0.101	0.098
	50 °C		0.097	0.094

Cu-Ag coupon corrosion

After finished testing, surface of Cu and Ag were analyzed for corrosive element such as sulfur which may be remained on their surfaces by SEM/EDX. The spectra of Cu and Ag after tested of sample VI are presented as in Fig. 4.33 and the results were compared with that of commercial B as shown in Fig. 4.25.

Sample VI

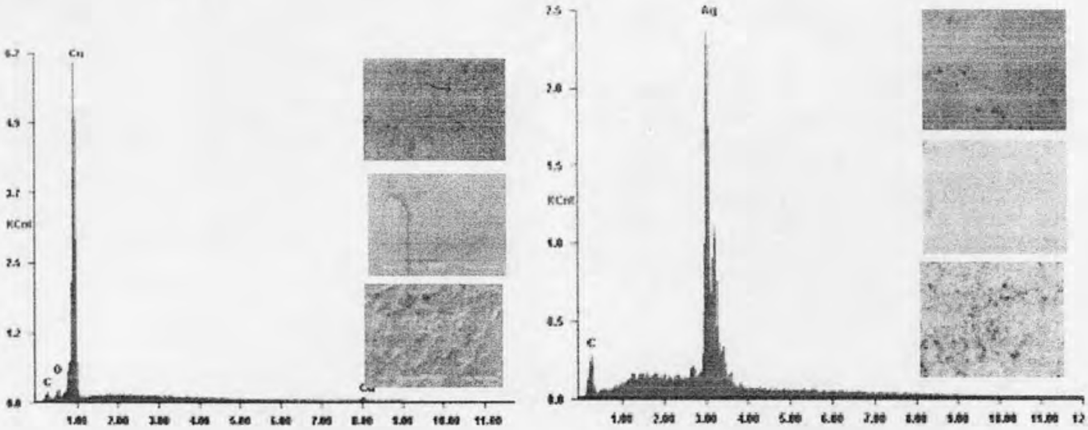
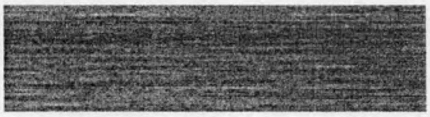
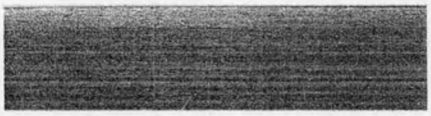


Figure 4.33 Surface analysis on Cu-Ag coupon of sample VI by SEM/EDX

According to surface analysis results, SEM/EDX instrument could not detected any sulfur corrosive outgas on surface of Cu and Ag coupon after tested with sample VI and commercial B. Therefore, it could be concluded that composition of base oil or additives in these finished lubricants were not generated any corrosive outgas to damage surface of bearing's components.

Cu plate corrosion

Table 4.19 Pictures of Cu plate corrosive results of sample VI

Sample	Picture of Cu plate after tested	Corrosive level
Blank		-
VI		1b

From Cu plate corrosion test result as shown in table 4.19, it was found that sample VI was in class 1 when compared with ASTM corrosion standard. However, commercial B was in class 2 which could be because of sulfurized ester EP additive. However, corrosion in class 2 was considered not to give any corrosion on surface of materials.

Outgas analysis

Commercial B had higher outgas than sample VI because this finished lubricant used more volatile base oil.

Friction coefficient by SRV

Base on results of friction coefficient (Fig. B15 to Fig.B18), both finished lubricants were not significantly different and friction coefficient of them were still with in 10% acceptable difference.

Therefore, it could be concluded that sample VI was improved to the same lubricity performance when compared with commercial B.

Evaporation loss

The percentage of evaporation loss (Fig. 4.34) of sample VI was lower than that of commercial B. Thus, it suggested that commercial B was contained base oil with lower viscosity, so it might be easier to evaporate than sample VI.

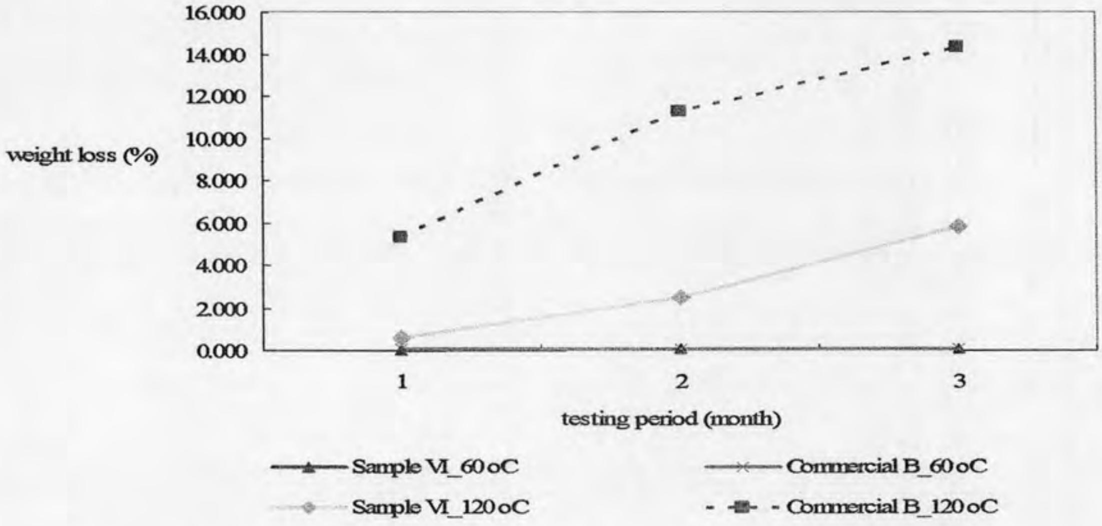


Figure 4.34 Percentage of weight loss at 60 °C and 120 °C for several testing period

Compatibility test

Surface of bearing part after compatibility test were analyzed by microscope. Moreover, finished lubricants were also analyzed for total acid number and the results of these testing items were shown in table 4.20 and Fig. 4.39, respectively.

Table 4.20 Visual check of bearing parts after compatibility test by microscope

Testing condition		**Finished lubricant**	
		Sample VI	**Commercial B**
60 °C	1 month	O	C
	2 months	O	C
	3 months	C	C
120 °C	1 month	C	C
	2 months	C	C
	3 months	C	X

Remark :

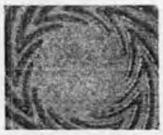
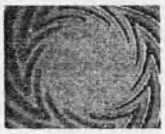
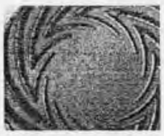
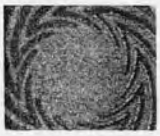
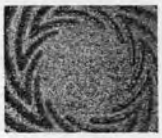
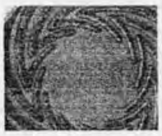
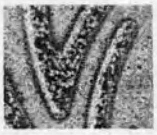
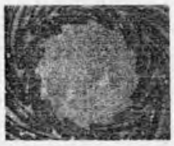
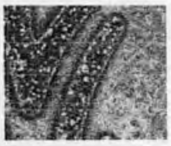
O represents no stain or discoloration on bearing part

C represents discoloration on bearing part

X represents some stain on bearing part

The picture of bearing parts before and after compatibility test are shown in table 4.21.

Table 4.21 Picture of bearing parts after compatibility test at 60 °C and 120 °C by microscope

Condition test	Sample VI		Commercial B	
	x10 magnification	x100 magnification	x10 magnification	x100 magnification
before test				
60 °C x 1 month				
60 °C x 2 months				
60 °C x 3 months				
before test				
120 °C x 1 month				
120 °C x 2 months				
120 °C x 3 months				

After baking at 60 °C for 2 months, it was found that the bearing part when tested with sample VI gave color change to light brown color. The color change of these bearing parts may be from the effect of triphenyl phosphate antiwear and phenyl amine antioxidant additives. Thus, the testing time which bearing part changed color was faster than sample III.

In case of high temperature testing as 120 °C, it was found that the bearing part that baked with sample VI, changed color as shown in table 4.21. In the area of color change, phosphorus element was detected which was possible from antiwear additive as triphenyl phophate compound, base on SEM/EDX result as shown in Fig. 4.35 to Fig. 4.38. However, bearing part baking with commercial B at 120 °C for 3 months gave stain on it and SEM/EDX detected sulfur element which possibly came from EP additive. In addition, phosphorus element was also detected which possibly came from tricresyl phosphate as antiwear additive.

Sample VI

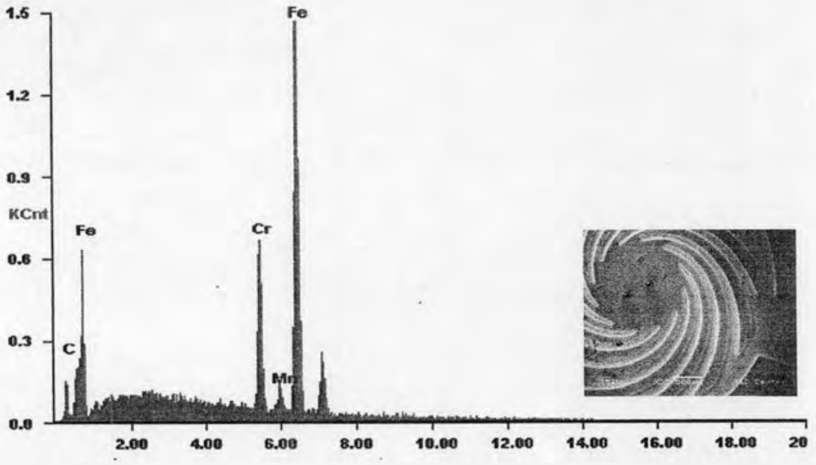


Figure 4.35 Surface analysis on bearing part before compatibility test of sample VI by SEM/EDX

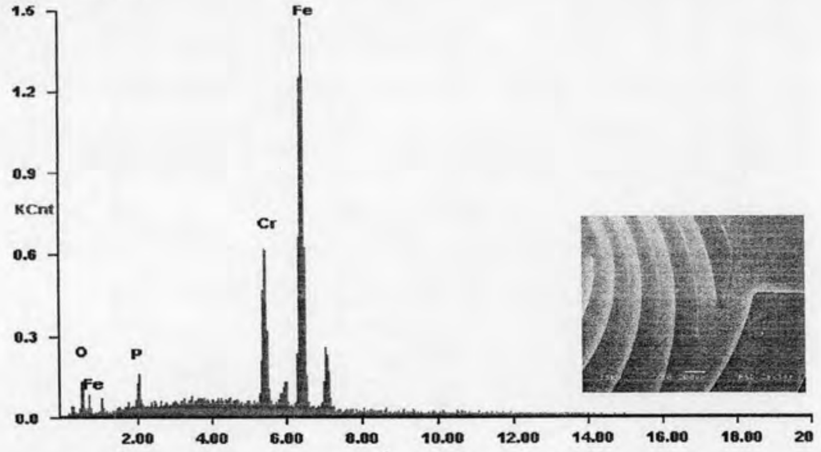


Figure 4.36 Surface analysis on bearing part after compatibility test at 120 °C for 3 months with sample VI by SEM/EDX

Commercial B

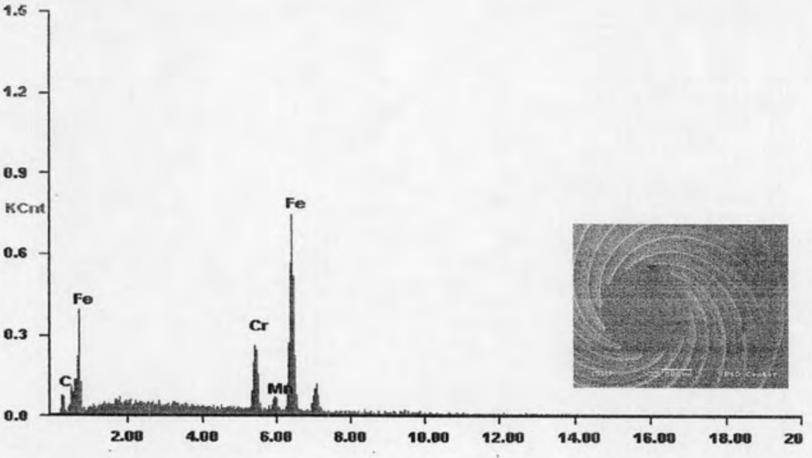


Figure 4.37 Surface analysis on bearing part before compatibility test of commercial B by SEM/EDX

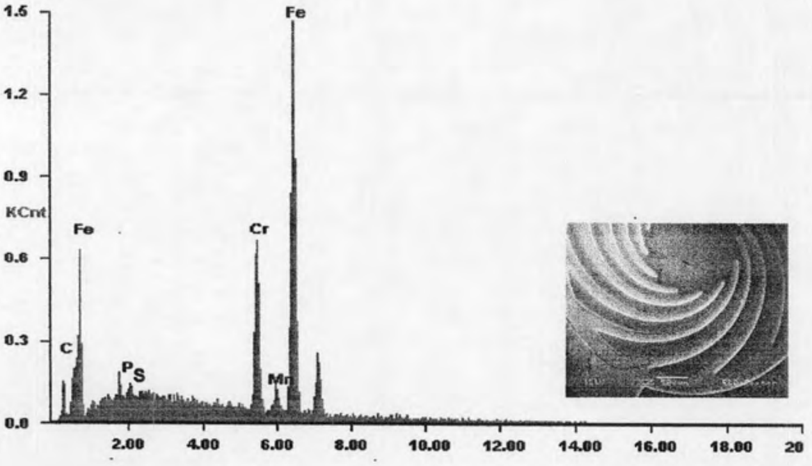


Figure 4.38 Surface analysis on bearing part after compatibility test at 120 °C for 3 months with commercial B by SEM/EDX

Besides, surface analysis of bearing parts after compatibility test, the finished lubricants were also analyzed for total acid number as shown in Fig. 4.39.

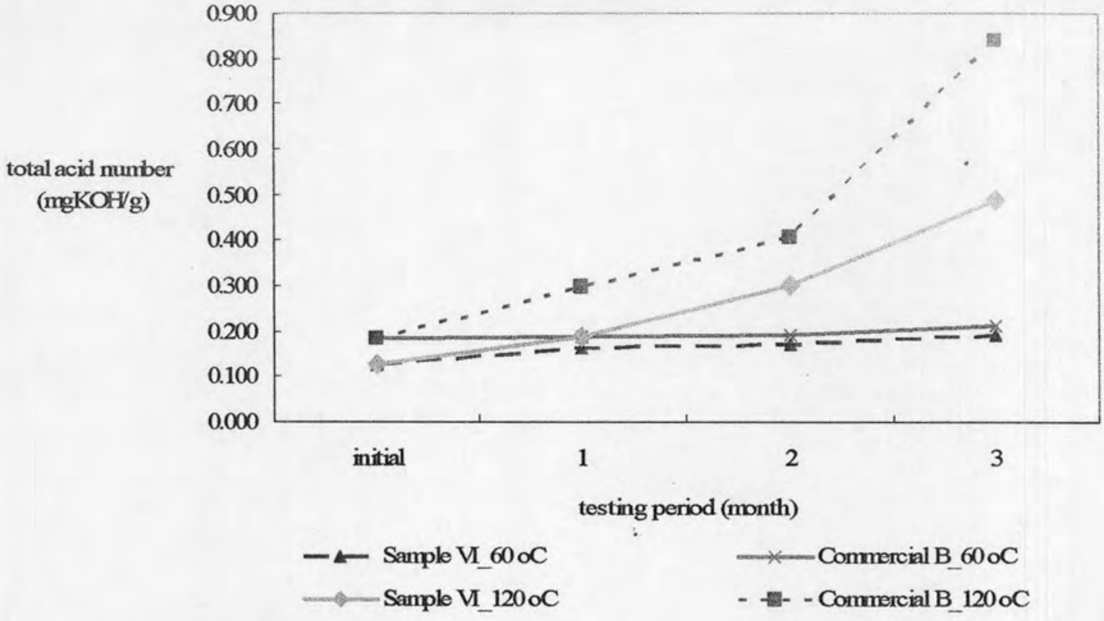


Figure 4.39 Total acid number of finished lubricants after compatibility test at 60 °C and 120 °C for three months testing period

Total acid number of sample VI was lower than that of commercial B because retarded oxidation reaction ability of sample VI was better than commercial B. Therefore, acidic products from sample VI should be lower than those of commercial B.

Service life test

The finished lubricants were applied into FDB spindle motor for 2.5 inch HDD and then run in the control oven at 80 °C and 100 °C for 3 months. The service life test result was presented in term of running current which motor used for running as shown in table B4 and table B5.

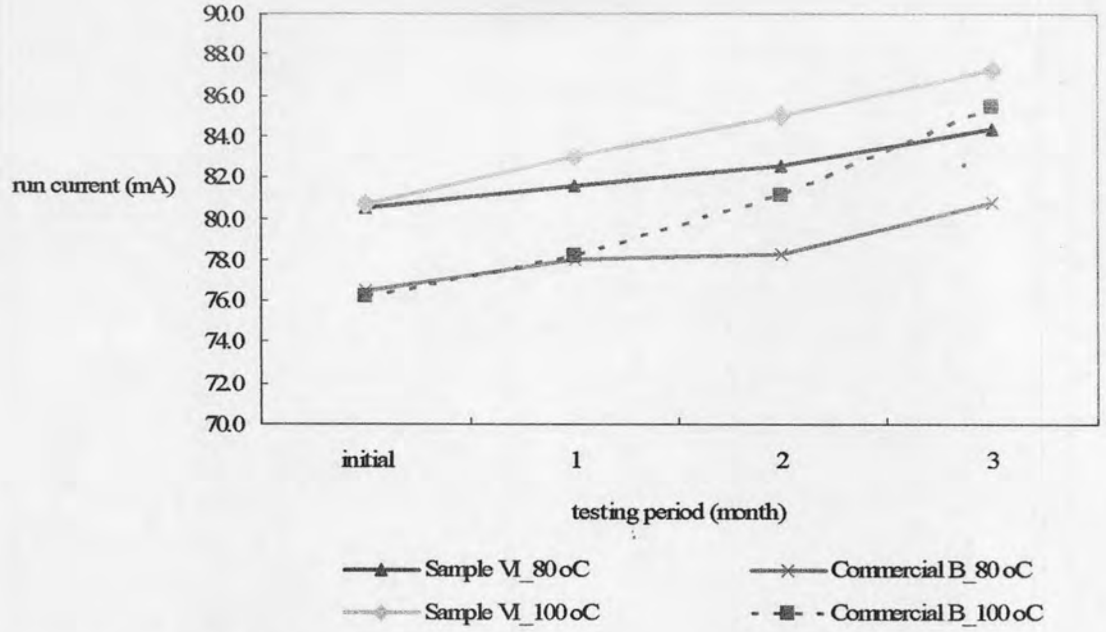


Figure 4.40 Plot of running current of motor at 80 °C and 100 °C versus several testing period of each finished lubricants

From Fig. 4.40 and table 4.22 for both testing conditions at 80 °C and 100 °C, the slope of commercial B was higher than that of sample VI which mean that commercial B was consumed more current during testing for 3 months than sample VI. It is possible that commercial B had higher evaporation loss. Therefore, viscosity was increased which resulted in using more power to run at boundary condition.

Table 4.22 Equation of graph for finished lubricant after life test

Testing condition	**Finished lubricant**	**Equation of graph**
80 °C	Sample VI	y = 1.252X + 79.12
	Commercial B	y = 1.320X + 75.07
100 °C	Sample VI	y = 2.148X + 78.62
	Commercial B	y = 3.058X + 72.58