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Influence of EP Film Properties on Anti-scoring Performance of Gears*

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Using a power circulating gear testing machine, the influence of EP film properties on the anti-scoring performance was examined in air and nitrogen gas environments. An electron probe microanalyser and a transmission electron microscope are mainly used to determine the chemical composition and the structure of EP film on tooth surfaces.

The findings are as follows:

- 1) In the case of sulphur-additive (dibenzyl-disulfide), EP film is much formed, and the chief compounds in EP film are FeS and FeS₂. FeS is observed more often in nitrogen gas environment than in an air one.
- 2) In the case of phosphorus-additive (dialkyl-phosphate), the compounds of Fe₃P and FePO₄ in EP film show a favorable effect for anti-scoring performance as well as the chemical polishing effects.

1. Introduction

In order to improve the scoring capacity of a gear, many additives have been used. The reacting mechanism to steel of these EP(extreme pressure) additives and the effect of environments on the reaction have been clarified fairly by experiments using adsorption test, four-ball test, two-cylinder test etc., but studies using a practical gear are very few⁽¹⁾.

In gear meshing, the contact time ratio is small and the contact is not continuous. Therefore, it seems to be probable that the result of gear test differs from other testing results. The present scoring test was done in air or nitrogen gas environment, using liquid paraffin as a base oil, which was added with a small amount of dibenzyl-disulfide as a sulphur-type additive or dialkyl-phosphate as a phosphorus-type additive. The difference of EP film formation on a tooth surface in each condition described above was measured using EPMA equipment and the crystal structure in surface EP film was analysed by means of the electron diffraction using an electron microscope. Therefore the objective of the present investigation is to study the effect of the kind and grade of the EP film on the scoring capacities and wear resistance capacities.

2. Test gear and lubricant

The material of test gear is

SCM21H. The chemical composition is shown in Table 1. Table 2 shows the dimensions of the test gears.

The lubricant is liquid paraffin containing some additives, their compositions and physical properties being shown in Table 3.

3. Test rig and experimental method

As the test rig, a power circulating gear drive testing machine was used. The driven pinion was rotated at a speed of 6,000 rpm. In gear testing, the bath lubrication was used and the environment in a

Table 1 Chemical compositions of gear material

Chemical composition wt % Material	C	Si	Mn	P	S	Cr	Mo
SCM21H	0.16	0.25	0.80	0.016	0.010	0.98	0.18

Table 2 Test gears

Item	Pinion	Gear	Item	Pinion	Gear
Number of teeth	18	40	Tooth width (mm)	6	
Module	4		Length of action (mm)	17.288	
Cutter pressure angle (°)	20		Contact ratio	1.464	
Addendum modification coefficient	+0.4	-0.5	Backlash (mm)	0.34	
Pitch circle diameter (mm)	72	160	Gear finish	Reishauer ground	
Outside diameter (mm)	83.2	164	Grade of accuracy	2nd class of JIS	
Center distance (mm)	116		Material	SCM21H Carburizing and quenching	

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Table 3 Physical properties and compositions of testing lubricant

Item	Dialkyl-phosphate additive oil	Dibenzyl-disulfide additive oil	Item	Dialkyl-phosphate additive oil	Dibenzyl-disulfide additive oil
Specific gravity	0.863	0.865	Total acid number (mgKOH/g)	0.9	0.01
Flash point (°C)	204	204	Saponification value (mgKOH/g)	0.9	3.6
Viscosity- 37.8°C ity(cSt)98.9°C	31.96 5.277	32.27 5.254	Water content (Wt %)	—	—
Viscosity index	103	103	Sulphur content (Wt %)	0.0017	0.59
Pour point (°C)	-12.5	-12.5	Phosphorus content (Wt %)	0.044	—
Residual carbon (Wt %)	0.09	0.00			
Ash content (Wt %)	0.003	0.002			

Base oil : 150SUS liquid paraffin.

1. Pressure cylinder for nitrogen gas,
2. Pressure reducing valve,
3. Pressure gauge, 4. Inlet-changing valve,
5. Upper inlet, 6. Inlet in oil bath,
7. Thermocouple, 8. Thermometer, 9. Heater,
10. Rheostat.

Fig.1 Gear box and inlet of nitrogen gas

- q. Load sharing ratio,
R. Equivalent radius of curvature,
Vs. Relative sliding speed,
θ. Flash temperature rise.

Fig.2 Calculated value of flash temperature rise

Table 4 Load stage

Load stage	Tooth load kgf/mm	Max. Hertzian stress kgf/mm ²	Load stage	Tooth load kgf/mm	Max. Hertzian stress kgf/mm ²
1	3.91	41.04	1	24.83	103.42
2	5.27	47.65	2	26.92	107.69
3	6.71	53.76	3	29.05	111.87
4	8.25	59.61	4	31.22	113.97
5	9.85	65.14	5	33.43	120.00
6	11.53	70.48	6	42.63	135.51
7	13.27	75.61	7	52.36	150.18
8	15.07	80.57	8	62.57	164.18
9	16.92	85.37	9	73.20	177.57
10	18.82	90.04	10	84.25	190.51
11	20.78	94.61	11	95.66	203.00
12	22.78	99.06	12	107.43	215.12

gear box was kept in air or nitrogen gas. In the case of measurement in air, a pair of gears are rotated without loading for one minute at a rate of four times during temperature rise. The temperature becomes constant at 70 °C, and then the test gears are loaded to the desired amount and are rotated for five minutes. In the case of testing in nitrogen gas, nitrogen gas is allowed to flow in for one minute from an upper inlet (5) with a pressure of 0.6 kgf/cm², as shown in Fig.1, and then a cork is put into the center hole of each gear shaft. Subsequently the gas pressure is reduced to 0.1 kgf/cm² and the gas flows out from the inlet in the oil bath (6) during the gear drive test.

The oil temperature and drive time are the same as those in air. The residual oxygen concentration in the gear box can be measured by gas chromatography (carrier gas is helium). In this case, the residual oxygen concentration is 600 ppm. As a reference, the oxygen concentration in the used nitrogen gas is 200 ppm.

Klaus et al.⁽²⁾ have reported after a four-ball test that the diameter of wear scar in the environment of residual oxygen concentration of 5,000 ppm was clearly smaller than that in air. Therefore, in the gear test also, the effect of oxygen on

scoring failure may be clarified sufficiently with the above mentioned concentration. The tooth load step was set such that the difference of the flash temperature calculated by Blok's equation was constant between different steps. The tooth load and maximum Hertzian pressure at each load step are shown in Table 4.

As an example, the surface temperature rise, the relative sliding speed, the relative radius of curvature and the load sharing ratio at load step P-5 are shown in Fig. 2. After a pair of gears are tested at each load step, the profile change, the change of surface roughness and the cumulative weight loss due to wear were measured.

The tooth of the pinion is driven to a load step just before scoring has occurred and then the weight concentrations of sulphur and phosphorus on the tooth surfaces are measured by means of EPMA. Next EP compound film of the tooth surface is transferred on a replica by the extractive replica method. An electron diffraction photograph is taken with the electron microscope and then the structure of crystals is analysed.

In the present experiment, the scoring of tooth surface was decided by the formation of typical scuffing marks which are characterized by local welds, but the wear accompanying a gradual change of tooth profile was not deemed as a scoring. The experimental procedure for the extractive replica method is as follows. The surface of gear tooth, which has been cut, is firstly etched with a corrosive agent (3 % nital) in a period of 15~20 sec. and then the carbon film is evaporated on the tooth surface with thickness of about 400 Å by means of a vacuum evaporation equipment. Next, a carbon film is cut into a square with a side length of 1 mm, and after etching this sample with the same corrosive agent, the surface layer of tooth is transposed on carbon film and the film square is then separated from the tooth. This film prepared by the procedure described above becomes a sample for electron diffraction test.

For investigating the effect of sulphur and phosphorus components contained in steel of gear material, an unused gear was tested for the measurement by EPMA and an electron diffraction test with extractive replica method. In the results, there was observed no effect of contained components.

Fig. 3 Cumulative weight loss

Therefore it can be considered that the measured values in the present experiment are the values of EP film formation on the surface.

4. Experimental results and discussion

4.1 Cumulative weight loss and ratio of scoring area

Figure 3 shows the cumulative weight loss in air and nitrogen gas environments to the dibenzyl-disulfide additive oil (this is called "S type additive oil" for short in the following) and dialkyl-phosphate additive oil (this is called "P type additive oil" for short in the following). The weight loss before the starting stage of scoring is the same for each type and is considerably small, except S type additive oil in nitrogen gas environment.

However, the weight loss shows a rapid rate of increase at the starting stage of scoring. These phenomena can be explained as follows. When the scoring occurs by a rupture of EP film, its load stage dominates the extent of scoring area and depth, so the weight loss depends on the scoring load stage. But the weight loss corresponds to the same order by the EP film function before the starting stage of scoring. The weight loss in the case of S type additive oil in nitrogen gas environment differs from that in other cases due to the difference in characteristic properties of EP film. In the present experiment, it is considered that the starting stage of scoring can be decided by the appearance of welding. From the welded area, the ratio of scoring area is calculated. The ratio of scoring area is about 62 % in the case of P type additive oil and about 44 % in the case of S type additive oil regardless of the environments, as shown in Fig. 4. According to the experimental result⁽³⁾ in non-additive liquid paraffin of the same viscosity, the ratio of scoring area differs depending on the environment, i.e. about 55 % in air environment and about 70 % in nitrogen gas environment.

The results that the ratio of scoring area does not correspond to the cumulative weight loss and the ratio of scoring area differs depending on the environments in the case of non-additive liquid paraffin, indicate that if a considerably thick or

Fig. 4 Starting point of scoring and spread ratio of scoring area

strong EP film is formed, the scoring area will be restricted only to a zone of high load sharing ratio and high sliding speed, resulting in a decrease in the scoring area. Therefore, it is considered that the EP film in the case of S type additive oil is thicker or stronger than that of P type additive oil. On the other hand, since an oxide film can be formed easily in air environment in the case of non-additive liquid paraffin, the oxide film acts as an anti-friction agent and brings about a small scoring area, as pointed out by Bjerk⁽⁴⁾ from the result of a roller test.

4.2 Variation of tooth profiles

Figure 5 shows the variation of tooth profiles correlated with the relative position from an initial profile with conditions. The variation of tooth profile is considerably small, except in the case of S type additive oil in nitrogen gas environment.

Very slight piling up of profile due to plastic flow of surface layer is observed at the dedendum surface of the wheel and the wear is observed at the addendum surface of the pinion at the load stage of P-6 of S type additive oil in air environment and P-2.4 of P type additive oil in air environment.

On the other hand, the wear on the dedendum surface of the wheel and on the addendum surface of the pinion is remarkable at all load stages of P-2.4 ~ P-12 in the case of S type additive oil in nitrogen gas environment.

Next, the variation of tooth profiles is decided by the degree of failure at the surface layer. In the case of P type ad-

ditive oil, irrespective of the environments, the variation of tooth profile is remarkable at the zone of one pair teeth contact. The wear of tooth profile of the wheel is also remarkable in nitrogen gas environment. In the case of S type additive oil, the variation of dedendum profile of the pinion is remarkable in air environment (P-9). The same tendency is observed at the load stage P-12 in nitrogen gas environment also. Therefore, the load is supported with the EP film and the EHL (elastohydrodynamics lubrication) oil film in the low load stages (if we calculate using Dowson's equation, EHL oil film thickness at P-2.4 load stage is about 5×10^{-6} cm), but with an increasing tooth load, the predominant action changes from an adsorbed EP film to a compound EP film. A scoring failure can be prevented due to wear of this compound, but the weight loss increases.

Such wear is observed also in P type additive oil, but the intensity is lower than that in S type additive oil. An improvement of surface roughness by the action of chemical polishing during the load increment reduces the wear, as shown in Fig. 6. In S type additive oil, a black film is formed on the tooth surface during the load increment regardless of the existence of oxygen, resulting in a discoloration of lubricant. The suspended matter in lubricant was analysed by the emission spectrochemical analyser. As a result of analysis of the suspended matter in lubricant, not

Fig.5 Profile variation in various testing conditions

Fig.6 Variation of tooth surface roughness (P type additive oil, in air) (Scoring occurs at P-8)

(a) In air environment

(b) In nitrogen gas environment

Fig.7 Sulphur concentration on tooth surface

(S type additive oil, 20kV, 0.05μA)
 0.5:0~0.5Wt%, 1.0:0.5~1.0Wt%,
 2.0:1.0~2.0Wt%, 3.0:2.0~3.0Wt%,
 0.1:0~0.1Wt%, 0.2:0.1~0.2Wt%,
 0.4:0.2~0.4Wt%, 0.6:0.4~0.6Wt%.

carbon but iron was detected. Such result proves that an increase in scoring limit is caused by the ease of the wear of compound EP film in S type additive oil, especially in nitrogen gas environment.

4.3 EPMA testing result

Figure 7 shows the curves of equal weight concentration of sulphur obtained from EPMA (electron probe microanalyzer) test on tooth surface of a gear. The gear was driven to a stage just preceding the scoring stage. Figure 7(a) shows the result in air environment and Fig.7(b) shows the result in nitrogen gas environment. The contour lines of sulphur weight concentration were drawn according to the results of analysis, as shown in Fig.8, which had been carried out along six lines arranged 1 mm apart.

Figure 9 shows an example of the line analysis of phosphorus weight concentration and Fig.10 shows the contour lines of phosphorus weight concentration. In the analysis of sulphur the accelerating voltage of the electrons was kept at 20 kV, but in the case of phosphorus the voltage was set at 10 kV, considering that the mean weight concentration of tooth surface decreases as the voltage becomes higher⁽⁵⁾. Each concentration of sulphur and phosphorus is detected to be nearly in proportion to the

flash temperature rise by Blok's equation. Examined in detail, the concentration at tooth top becomes higher than that at the highest point in the zone of one pair contact. It can be considered that the above mentioned tendency is due to the variation of tooth surface roughness. The cumulative weight loss and the profile variation vary according to the properties of EP film as described in Paragraphs 4.1 and 4.2. From these characteristics and the results of EPMA test, we get the following conclusions.

From the interesting facts that the profile variation is large and the sulphur concentration is low in the case of S type additive oil in nitrogen gas environment, it seems that the scoring can be prevented by the wear of compound EP film. The profile variation is smaller and the sulphur concentration is higher in the case of S type additive oil in air environment than in nitrogen gas environment. These results support an idea that the wear can be prevented by the adsorbed EP film until a considerably high load stage and the sulfide EP film is formed⁽⁶⁾ at the real contact point where the flash temperature becomes higher. In the case of S type additive oil, regarding the difference between the scoring limit load in air environment and in nitrogen gas environment, this can be explained by the oxide film as well as the thermal stability of sulphur type additive and sulphur compound in the same idea as reported by Toyoguchi et al.⁽⁷⁾. Namely, because the adsorption of sulphur type additive is more powerful on the oxide film of iron than on the iron surface, the formation of adsorbed EP film is promoted very much by the environment of oxygen or air.

Fig.8 Sulphur concentration
(S type additive oil, in air)

Fig.9 Phosphorus concentration
(P type additive oil, in air)

(a) In air environment

(b) In nitrogen gas environment

Fig.10 Phosphorus concentration on tooth surface
(P type additive oil, 10kV, 0.05μA)
0.4:0.4~0.4Wt%, 0.6:0.4~0.6Wt%,
0.8:0.6~0.8Wt%, 1.0:0.8~1.0Wt%,
1.2:1.0~1.2Wt%, 1.4:1.2~1.4Wt%.

However, since the sulphur type additive and the sulphur compound decompose and then oxidate at temperature above a certain limit, the sulphur compound formed on the iron surface increases in quantity in the environment of rare oxygen. In the present study, the calculated value of the surface temperature is also about 200 °C at the load stage P-8 just ahead of the scoring limit load stage and this temperature is close to the transient temperature 230 °C as suggested by Toyoguchi et al.. So, the formation of the compound EP film in nitrogen gas environment becomes violent and it is useful to the prevention of scoring. In air environment the decomposition and the formation alternately occur repeatedly. Namely, the compound EP film decomposes and then an oxide film is formed. Next, the additive is adsorbed on the oxide film and then again a compound EP film is formed. The effect of adsorbed EP film here is predominant and the profile variation before the occurrence of scoring is small.

In the case of P type additive oil in both environments of air and nitrogen gas, the phosphorus concentration detected by EPMA is lower than that in the case of S

type oil in air environment, and then the scoring limit load and the profile variation are small. Regarding the above results, it is assumed that the scoring limit load and the wear resistance capacity increase mainly due to the chemical polishing and the adsorbed EP film. The profile variation is large and the scoring occurs easily in the zone of one pair contact, because of the high temperature and the thermal decomposition of phosphorus compound EP film. This idea is supported by the following results: Figure 11(a) and (b) show the second electron images in the zone of one pair contact on tooth surface in the case of S type additive oil in nitrogen gas environment and of P type additive oil in nitrogen gas. It is clear that the top part of surface roughness was completely worn out at the middle part of Fig. 11(a), but the locally damaged parts of EP film were in metallic contact as shown in Fig. 11(b). In these secondary electron images, we can not observe locally dense clusters of the luminous spot, due to the chemical reactant product, as shown in wear traces of a four ball test⁽⁸⁾. Figure 12 shows the characteristic X-ray images. The density of luminous spot at the top part of tooth is generally higher than that in the zone of one pair contact, and it is intuitively clear that a compound EP film is uniformly formed with a temperature rise.

4.4 Electron diffraction photograph by means of electron microscope

Using the replicas prepared by the above method and an electron microscope (the accelerating voltage of 100 kV), we can get the penetrative photomicrographs and the electron diffraction patterns shown

(a) S type additive oil

Transverse oscillating curve in the figure shows the surface roughness.

(b) P type additive oil

Fig.11 Secondary electron images in zone of one pair contact (in nitrogen gas)

(a) Near the tooth top

(b) In zone of one pair contact

Fig.12 Characteristic X-ray images (sulphur)

in Fig.13. In the case of S type additive oil, FeS_2 of a face centered cubic system, FeS_2 of a rhombic system and FeS of a hexagonal system are observed. Here the replicas are collected three for each of the three teeth and the observation is taken at more than 10 points in each replica. The structure of FeS is recognized more often in nitrogen gas environment than in air environment. Regarding the compound EP film of S type additive oil, from the present study we can state as follows: It has been reported⁽⁹⁾ that the scoring limit load increases with the melting point of surface materials in contact. FeS has small sliding resistance and high melting point (about $1,200^\circ\text{C}$), but FeS_2 has low melting point (about 500°C).

The crystal system of FeS was observed more frequently than the crystal system of FeS_2 in nitrogen gas environment. Therefore, in this case the shearing strength decreased, the wear occurred easily. As a result, a scoring could not occur. In air environment, the adsorbed EP film plays an important role until higher load stage as described above. When the adsorbed EP film decomposes, the scoring occurs easily. Even if the compound EP film is formed, the content of FeS_2 which has a low melting point is predominant. Therefore the scoring limit load becomes lower than that in nitrogen gas environment.

Other EP film formation may yield sulfuric acid compounds. Because the iron sulfate hardly acts as EP film⁽⁸⁾ and these crystal structures are complex, these compounds are not analysed in the present paper.

Figure 14 shows the penetrative photomicrographs and the electron diffraction patterns for P type additive oil in nitrogen gas environment. Here, Fe_3P of a tetragonal system is observed. Further, for P type additive oil in air environment, instead of phosphide, iron phosphate FePO_4 of a hexagonal system is observed.

It has been reported about the tappet testing result by Barcroft et al.⁽¹⁰⁾ that in the environments of air, as an oxide functions as the catalyst, phosphate compounds are predominant in the product on iron surface. In the present study, it is considered also that FePO_4 was only observed in air environment, but in nitrogen gas environment a eutectic product of Fe_3P and Fe which has a low melting point acts as an EP film, as reported by Beeck et al.⁽¹¹⁾. Moreover, irrespective of environments of air and nitrogen gas, the profile variation is large in the zone of one pair contact after the scoring has occurred. These facts may be reasonable, taking the following results into consideration: The eutectic product loses the activity as an

Fig.13 Photomicrographs and electron diffraction patterns (5,000 times)

Fig.14 Photomicrographs and electron diffraction patterns (5,000 times)

EP film at high temperature, because the melting point of this product is lower than that of S type additive oil. And the quantity of product is little, as evidenced by the measuring results by EPMA.

5. Conclusions

From the experimental results of the cumulative weight loss, the profile variation, EPMA test and the electron diffraction test, the following conclusions can be drawn.

Considering the difference between the effects of P type additive and S type additive, the effect of P type additive is an improvement of lubricating ability mainly due to the chemical polishing and the increment of wear resistance capacity by the adsorbed EP film. The compound EP film is easy to decompose thermally and then the quantity of the compound products decreases. Further, since the melting point is low, the scoring limit load is not so large.

The effect of S type additive can be recognized in some improvement of the wear resistance capacity by the adsorbed EP film, but this additive is characterized by the fact that the scoring limit load increases due to the wear of the compound EP film, as confirmed by emission spectrochemical analysis. About the effect of environments, we can state as follows. In the environment of nitrogen gas with S type additive oil, because the loading capacity of scoring limit increases as a function of compound EP film which contains a lot of crystal structure of FeS which has high melting point and low shearing strength, the scoring damage is not so serious. In the environment of air with S type additive oil, however, the loading capacity is lower than that of nitrogen gas, because the compound

FeS of low melting point is predominant in EP film and it decomposes at comparatively low temperature.

In environment of air with P type additive oil, the function of phosphate compounds formed easily because of the oxide acting as the catalyst, as well as the function of adsorbed EP film, causes an increase in the scoring limit load.

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References

- (1) Barcroft, F.T. and Daniel, S.G., Proc. Instn. Mech. Engrs., Vol.179, Part 3J (1964), p.265.
- (2) Klaus, E.E. and Bieber, H.E., ASLE Trans., Vol.7, No.1 (1964), p.1.
- (3) Terauchi, Y. et al., Memor. Facul. of Engg. Hiroshima Univ., Vol.7, No.1 (Ser. 21), (1979), p.21.
- (4) Bjerk, R.O., ASLE Trans., Vol.16, No.2 (1973), p.97.
- (5) Terauchi, Y. et al., Preprint of Jpn. Soc. Mech. Eng., No.740-15 (1974), p.61.
- (6) Forbes, E.S. and Reid, A.J.D., ASLE Trans., Vol.16, No.1 (1973), p.50.
- (7) Toyoguchi, M. et al., Bull. JPI, Vol.2 (1960), p.50.
- (8) Allum, K.G. and Forbes, E.S., ASLE Trans., Vol.11, No.2 (1968), p.162.
- (9) Rozeanu, L., ASLE Trans., Vol.16 (1973), p.115.
- (10) Barcroft, F.T. and Daniel, S.G., Trans. ASME, Ser.D, Vol.87, No.3 (1965), p.761.
- (11) Beeck, O. et al., Proc. Royal Soc. London, A, Vol.177, No.968 (1940), p.103.