Wear Debris Analysis of Seizure Behaviors of PEEK Materials in Oil

Tomoharu Akagaki^{1)*}, Kazuma Kataoka²⁾ and Masahiko Kawabata³⁾

¹⁾Department of Mechanical Engineering, Hachinohe National College of Technology
16-1 Tamonoki, Uwanotai, Hachinohe-shi, Aomori 039-1192, Japan

²⁾Mitsubishi Automotive Engineering Co. Ltd.

1 Nakashinkiri, Hashimecho, Okazaki-shi, 444-8501, Japan

³⁾Tribotex Incorporation

45-7 Yamaguchi, Nagakusa-cho, Obu-shi, Aichi 474-0052, Japan

*Corresponding author: akagaki-m@hachinohe-ct.ac.jp

(Manuscript received 03 November 2015; accepted 08 July 2016; published 15 September 2016)

(Presented at the International Tribology Conference Tokyo 2015, 16-20 September, 2015)

Seizure behaviors of PEEK materials are studied using blocks on a ring wear tester under oil-lubricated conditions. The seizure processes are monitored with a particle counter to measure the number of wear particles in oil. Three kinds of block materials are tested: PEEK, PEEK composite filled with 30 mass% of carbon fiber, and white metal (WJ2). The ring is made of forged steel (SF540A). The sliding velocity is 15 and 19 m/s. The load varies between 294 and 1177 N. The ring temperature is measured with an alumel-chromel thermo-couple with a diameter of 0.5 mm, located 1mm below the frictional surface. Results indicate that the particle counter method can detect the seizure in the PEEK materials as well as in WJ2. However, the number of wear particle generated in the seizure of the PEEK materials is much less than in WJ2. It also increases in a mixed lubrication in WJ2. In contrast, wear particle is not detected appreciably in a mixed lubrication in the PEEK materials. Wear particles and wear scars on these materials are observed using a scanning electron microscope (SEM). The wear mechanisms in the seizure are discussed.

Keywords: seizure, wear debris analysis, particle counter, ring temperature, PEEK, PEEK composite, oil lubrication

1. Introduction

Poly-ether-ether-ketone (PEEK) is a high performance thermoplastic polymer and has some excellent mechanical and chemical properties such as high strength, high toughness, heat resistance, thermal stability, easy processing and chemical inertness [1]. As PEEK materials have also superior tribological properties, they have been applied to many machine elements [2-4]. Recently, PEEK composite bearing has been developed and rapidly replacing a traditional sliding bearing made of metal in a hydroelectric power plant [5-6]. A power breakdown in an electric power plant due to damages of PEEK composite bearing gives an immeasurable loss to a society strongly depending on an electricity. In order to prevent the power breakdown in a plant, it is important to detect quickly damages such as seizure and abnormal wear using condition monitoring techniques.

So far various monitoring and diagnostic techniques have been developed and applied to machine systems

and elements such as gas turbines, hydraulic systems, diesel engines, rolling bearings and sliding bearings mainly made of metals [7-10]. In contrast, there has been little work regarding the condition monitoring and the seizure behaviors of PEEK materials. This might be because PEEK composite bearing is relatively new and so its seizure has not been widely noted yet. Among some wear debris analyses, Ferrography is not an effective method to analyze non-magnetic particles such as PEEK materials, although it is very useful for magnetic particles. Although a spectrometric oil analysis program (SOAP) can detect many elements at high accuracy, it cannot detect carbon which is the major element of both PEEK material and carbon fiber's filler [11]. A particle counter method gives information on the number and size of wear particles in oil. It could be expected that the particle counter is useful for detection of both metal and polymer particles. However, a particle counter method has not been attempted to monitor the wear process of the PEEK materials.

The maintenance guideline for PEEK composite bearing has not been established yet. As a first step for

establishing the maintenance guideline, it is essential to correlate the seizure behaviors of PEEK materials with the characteristics of wear particles and the wear mechanisms. It is also important to monitor the seizure process using a monitoring technique and to examine whether it can detect the seizure.

In this study, the seizure behaviors of PEEK materials were studied at a high sliding velocity under oil-lubricated conditions. The seizure processes were monitored with a particle counter at a regular interval. For a comparison, the seizure process of white metal (WJ2) was also studied. Wear particles and wear scars were observed with a scanning electron microscopy (SEM) and the wear mechanisms were discussed.

2. Experimental apparatus and procedure

Experiments were conducted with block on a ring wear tester shown in Fig. 1 [12]. The testing materials are listed in Table 1. The ring was a forging steel (SF540A), with 130 mm diameter and 20 mm thickness. The ring surface was finished by cylindrical grinding after turning. Its surface roughness was $0.15 \pm 0.05 \mu\text{m}$ R_a . This roughness was decided so that lubrication modes could shift from hydrodynamic lubrication or

mixed lubrication to seizure at high sliding velocity. The block materials were unfilled PEEK (PEEK for short) and a composite filled with 30 mass% of PAN-based carbon fibers (PEEK composite). The carbon fiber was $8 \mu\text{m}$ in diameter and its length ranged from 50 to 200 μm .

For a comparison, the white metal (WJ2) was also tested. The blocks were 90 mm long, 10 mm wide, and 10 mm high. They were finished with an emery paper of #600. The experimental conditions are listed in Table 2. The sliding velocity was 15 and 19 m/s. The load was varied in the range of 294 to 1177 N. The lubricant used was a non-additive turbine oil (ISO VG46), which was supplied to the frictional surface at a flow rate of about 1 ml/s with a pump. The oil temperature was kept at $30 \pm 3^\circ\text{C}$ with a controller. The test duration was 1800 s. However, testing stopped when the seizure occurred. After the test, a profilometer was used to record the block's wear scar and to determine which of the block's profile was parallel to the sliding direction. In some tests, the surface roughnesses of their wear scars were also measured.

The ring temperature was measured with an alumel-chromel thermo-couple with a diameter of 0.5 mm, located 1 mm below the frictional surface. Figure 2 illustrates the measurement method of the ring temperature. The thermocouple was inserted into a

Fig. 1 Schematic diagram of experimental apparatus

Table 2 Experimental conditions

Sliding velocity (m/s)	15.0, 19.0
Load (N)	294 ~ 1177
Test duration (s)	1800
Lubricant	Non-additive turbine oil (ISO-VG46)
Flow rate (ml/s)	1.1
Oil temp. ($^\circ\text{C}$)	30 ± 3

Table 1 Properties of testing materials

	Material	Hardness	Surface roughness R_a (μm)
Ring	Forging steel (SF540A)	HV189 $\pm$ 8	0.15 ± 0.05
Block	PEEK	HRR126	0.15 ± 0.05
	PEEK composite (CF: 30 mass%)	HRR124	0.15 ± 0.05
	WJ2	HV26	0.25 ± 0.05

Fig. 2 Diagram showing measurement method of ring temperature

depth of 10 mm from the ring's side surface and fixed firmly with bolts and nuts. The frictional torque was measured with a torque-meter. The lubricating oil was delivered continuously with a pump to the particle counter to measure the number of wear particles at a regular interval. This system had the time lag of 1-2 minutes because it took 1-2 minutes before the oil reached the counter. The wear particle was separated from oil samples with a membrane filter having a pore size of 1 μm . Wear scars and wear particles were observed using the SEM.

3. Results and discussion

3.1. Seizure behaviors and ring temperature

Figure 3 illustrates the relationship between the friction coefficient and the run time at the sliding velocity of 19 m/s. The applied loads are 589 N for WJ2 and 1177 N for the PEEK materials. WJ2 transits to the seizure soon after the start of the test. In contrast, the PEEK materials transit to the seizure after sliding for a long time. The run time required for the transition to the seizure in the PEEK composite is longer than that in PEEK. Figure 4 illustrates the relationship between the friction coefficient and the ring temperature corresponding to the results shown in Fig. 3. In WJ2, the seizure occurs when the ring temperature exceeds about 80°C. In PEEK and the PEEK composite, the friction coefficient is small until about 100°C. Thereafter it increases gradually with the increase of the ring temperature. Finally, the seizure occurs when the ring temperature exceeds about 130°C for PEEK and about 180°C for the PEEK composite. Thus the seizure behaviors are strongly dependent on the ring temperature. It is expected that the real surface temperature is higher than the measured one in this study. Therefore, it is considered that the seizure occurs when the real surface temperature exceeds the glass transition temperature of 143°C in PEEK materials. At the temperature above the glass transition temperature, the real contact area increases due to the softening of the

Fig. 3 Relationship between friction coefficient and run time

Fig. 4 Relationship between friction coefficient and ring temperature

PEEK materials. As the results, the severe plastic deformation and flow occurs leading to the rapid increase of the friction coefficient. The ring temperature for transiting to the seizure in the PEEK composite is higher than that in PEEK. This might be because carbon fiber fillers contribute to improve the strength of materials and to increase the resistance to the plastic deformation and flow. Thus it is said that the PEEK composite has the superior seizure-resistant property.

3.2. Number of wear particles generated in seizure process

It is important for understanding these seizure processes to know lubrication modes. In order to apply the EHL theory to the results in this study and calculate a lambda ratio, the cross-sectional profile of the block's wear scar and the surface roughnesses of the ring and block were measured after the test. Figures 5(a,b) illustrate the cross-sectional profiles of the wear scars of the PEEK composite's block. Figures 5(c,d) illustrate the results in the block of WJ2. Table 3 summarizes the measurement results of the friction coefficient, the ring temperature and the surface roughness before and after the test. These results correspond to the profiles shown in Fig. 5.

Fig. 5 Cross-sectional profiles of ring and block: (a,b) PEEK composite's block; (c,d) block of WJ2

Table 3 Measurement results of friction coefficient and surface roughness after test

	Block material	Experimental condition	Friction coefficient	Ring temperature (°C)	Ring's surface roughness R_a (μm)	Block's surface roughness R_a (μm)	Comment
(a)	PEEK comp.	19 m/s 1177 N	0.02-0.03	13-140	(before) 0.21 (after) 0.22	(before) 0.14 (after) 0.15	Test was stopped before seizure.
(b)	PEEK comp.	19 m/s 1177 N	0.06-0.12	220	0.12 0.13	0.2 <u>1.4</u>	Seizure
(c)	WJ2	15 m/s 294 N	0.015-0.25	83	0.13 0.17	0.28 <u>0.08</u>	Mixed lubrication
(d)	WJ2	15 m/s 883 N	0.03-0.05	157	0.15 <u>0.46</u>	0.26 <u>0.7</u>	Seizure

Figure 5(a) is the experimental result when the test was stopped just before seizure. Then the friction coefficient increased gradually and the ring temperature was near the glass transition temperature. In the PEEK composite's block, the surface roughnesses of the ring and block after the test are almost the same as those before the test except only the block's roughness in the seizure, as shown in Table 3. The block's cross-sectional profile does not agree with that of the ring, as shown in Fig. 5(a). This means that the elastic contact predominates. In the seizure, the plastic contact is predominant as both the profiles agree well each other, as shown in Fig. 5(b).

In the block of WJ2, the ring's surface roughness is almost similar before and after the test when the friction coefficient is relatively small, as shown in Table 3. In contrast, the block's one becomes smooth. It is confirmed that the mixed lubrication is predominant, as the SOAP has detected the increase of tin element concentration. The iron element has not been detected at all. The measurement results of the surface roughness agree well with the SOAP's data. In the seizure, both the ring's and block's surface roughness becomes very rough due to transfer fragments on the ring and severe

damages on the block. As the cross-sectional profiles of the block in both cases agree well with those of the ring, the plastic contact is predominant, as shown in Figs. 5 (c,d).

These results suggest that EHL theory can apply to the case of the PEEK composite's block until seizure occurs without considering the changes of the surface roughness and the block's cross-sectional profile. In the case of WJ2, it may be possible to apply the EHL theory for rough estimation of lubrication modes as well as the case of the PEEK composite, as the block's wear scar is shallow and small, and only the block's surface roughness becomes smooth.

A lambda ratio [13] is calculated for discussion of the results; it is defined as $\lambda = \frac{h_{\min}}{(R_{q1}^2 + R_{q2}^2)^{1/2}}$, where

$h_{\min}$ is the minimum film thickness and R_{q1} and R_{q2} are the root mean square roughnesses of the ring and block, respectively. The minimum film thickness is calculated using the Dowson-Higginson formula [14]. Oil viscosity is calculated at the ring temperatures obtained from each test, and R_{q1} and R_{q2} are calculated from the arithmetic average roughness, R_a , where $R_q = 1.25 R_a$ [15].

Figure 6 illustrates the relationships between the friction coefficient, the ring temperature, the Λ ratio and the run time in the PEEK composite. The Λ ratio decreases gradually with the increase of the ring temperature due to the decrease of the viscosity. At the ring temperature of about 100°C, the Λ ratio becomes less than 3 and the lubrication mode changes from the hydrodynamic lubrication to the mixed lubrication. Even if the lubrication mode changes to the mixed lubrication, the friction coefficient maintains the low value of 0.01 until about 130°C. Thereafter it increases a little and finally the seizure occurs as the Λ ratio decreases to about 1 at about 180°C. It is well known that the boundary lubrication becomes predominant when the Λ ratio is less than 1. As shown in Table 3, the surface roughness of the block becomes rougher than that before the test when the seizure occurs. Therefore it is apparent that the Λ ratio is less than 1 in seizure. Thus, it is said that the boundary lubrication leads to seizure at high sliding velocity. It is also said that the PEEK composite has the excellent durability to maintain the low friction in the mixed lubrication at the high temperature.

Figure 7 illustrates the number of wear particles generated in the seizure process shown in Fig. 6. The particle sizes measured with the particle counter were in the range of 5 to 100 μm . In the mixed lubrication, the number of wear particles is not counted appreciably due to few generation of wear particles, although the data instantaneously scatters. When the lubrication mode transits from the mixed lubrication to the seizure, the number of wear particles increases rapidly to about 10^5 per 100 ml. Most of them are less than 15 μm in size. The similar analytical results were obtained in the seizure process of PEEK. It is strange that wear particles are not generated when the mixed lubrication is predominant in PEEK and the PEEK composite. This result is discussed based on the SEM observation results of the wear scar.

Figure 8 illustrates the relationships between the friction coefficient, the ring temperature, the Λ ratio and the run time in the seizure process of WJ2 at 15 m/s and 883 N. The Λ ratio decreases gradually with the increase of the ring temperature. The Λ ratio becomes less than 3 at about 60°C and the mixed lubrication becomes predominant. Then the friction coefficient fluctuates. When the Λ ratio becomes close to about 1 at 100°C, the seizure occurs. In the seizure of WJ2, the surface roughnesses of both the ring and block become very rough, as shown in Table 3. Therefore it is apparent that the Λ ratio is less than 1 in the seizure as well as in the PEEK composite's block.

Figure 9 illustrates the number of wear particles generated in the seizure process of WJ2 shown in Fig. 8. When the lubrication mode changes from the hydrodynamic lubrication to the mixed lubrication, the number of wear particles increases gradually to about 5×10^5 per 100 ml. As the seizure occurs, the number of wear particles increases rapidly to about 3.2×10^6 per 100 ml. Most of them are less than 15 μm in size. As the

Fig. 6 Relationships between friction coefficient, ring temperature, Λ ratio and run time in PEEK composite

Fig. 7 Relationships between friction coefficient, number of wear particles and run time in PEEK composite

Fig. 8 Relationships between friction coefficient, ring temperature, Λ ratio and run time in WJ2

seizure proceeds, the number of larger wear particles tends to increase. Thus the wear particles are generated in WJ2 clearly both in the mixed lubrication and seizure. The wear particles generated in the mixed lubrication can be regarded as a sign to indicate the direct contact leading to seizure at the high sliding velocity. Figure 10 illustrates the comparison of the number of wear particles generated in the seizure processes of three block materials. The number of wear particles generated in the seizure of the PEEK was about 5×10^5 per 100 ml.

Fig. 9 Relationships between friction coefficient, number of wear particles and run time in WJ2

Fig. 10 Comparison of number of wear particles generated in seizure process

It is the least in the seizure of the PEEK composite. In contrast, it is the most in the metal of WJ2. The difference might be because the PEEK materials have a gummy property at the high temperature. Therefore, it is supposed that the wear fragments do not easily detach from the wear scar. It is well known that the wear resistance of the PEEK composite is higher than that of the PEEK [12]. Thus the number of wear particles generated in the seizure of the PEEK composite might be less than that of the PEEK.

3.3. SEM observations of wear particles and wear scar

Figure 11 shows the SEM micrographs of the wear scar of the PEEK composite when the friction coefficient increases gradually in the mixed lubrication. The wear scar is smooth and any signs of the seizure are not observed, as shown in Figs. 11(a,b). The wear scar is uniformly covered with lamellar structures which probably are formed due to the mild plastic deformation and flow as the PEEK composite becomes soft at the high temperature. Clear evidences showing the generation of wear particles are not observed on the wear scar. This observation result agrees with the analytical result shown in Figs. (7,10). However, it is

Fig. 11 SEM micrographs of wear scar of PEEK composite in mixed lubrication before transiting to seizure: (a) overview; (b) enlargement of (a). The arrow indicates the relative direction of motion of the counterface

not clear in the PEEK composite that the wear particle is generated or not even if the lubrication mode is the mixed lubrication just before the seizure. Even if the wear particle is generated, it might be too tiny to detect using the particle counter.

Figure 12 shows the SEM micrographs of the wear scar of WJ2 in the mixed lubrication. The wear scar becomes smooth due to plastic deformation and flow as shown in Fig. 12(a). It seems that small wear particle are generated from layers formed by the ridge's deformation as shown in Fig. 12(b). As shown in Fig. 12(c), wear particles are also generated by cracking of smooth surface formed by plastic deformation. These wear particle sizes estimated are less than 10 μm and roughly agree with the result shown in Fig. 9.

Figure 13 shows the SEM micrographs of the wear particles generated in the seizure of the PEEK composite. Twisted and cylindrical wear particles are commonly observed as shown in Figs. 13(a-c). It is less than about 50 μm in size. Carbon fiber and ribbon-like wear particles are also observed, as shown in Figs. 13(b,c), respectively. A huge plate-like wear fragment

Fig. 12 SEM micrographs of wear scar of WJ2 in mixed lubrication: (a) overview; (b) enlargement of (a); (c) enlargement of (b). The arrow indicates the relative direction of motion of the counterface

Fig. 13 SEM micrographs of wear particles generated from PEEK composite in seizure

more than 500 μm in size remains on the wear scar, as shown in Fig. 13(d). At the front and lateral sides, many twisted and cylindrical bars similar to the wear particles shown in Fig. 13(a) are observed. Thus it is considered that the twisted and cylindrical wear particles have something to do with the huge plate-like wear fragment. In order to discuss the wear mechanism in the seizure, the wear scars were observed with the SEM. The results are shown in Figs. (14,15). As shown in Figs. 14(a,b), many ribbon-like and wire-like extrusions are observed at both the inlet and outlet positions. This is because these extrusions detach from the outlet position and adhere to the inlet position. Severe plastic deformation and flow causing the tearing fractures of the friction surfaces are commonly observed as shown in Fig. 14(c). The similar features was also observed in the case of the PEEK. Thus it is said that the seizure in the PEEK materials is characterized by the severe plastic

Fig. 14 SEM micrographs of wear scar of PEEK composite after seizure: (a) inlet position; (b) outlet position; (c) inside region. The arrow indicates the relative direction of motion of the counterface

Fig. 15 SEM micrographs of inside region in wear scar of PEEK composite after seizure: (a) overview; (b) enlargement of region indicated by red circle in (a); (c) enlargement of region indicated by yellow circle in (a); (d) enlargement of region indicated by blue circle in (a). The arrow indicates the relative direction of motion of the counterface

deformation and flow causing the tearing fracture. Figure 15 shows the inside region of the wear scar in the seizure of the PEEK composite. A deep and wide groove is observed, as shown in Fig. 15(a). Many carbon fibers are observed near the groove, as shown in Fig. 15(b). They gather densely due to the high temperature and pressure and seem to arrange in the frictional direction probably due to the plastic flow. Figure 15(c) shows the large fragment is likely to separate due to the tearing fracture and to generate the groove shown in Fig. 15(a). The huge wear fragment shown in Fig. 13(d) is probably generated in this process. When the huge wear fragment slides in the groove, many twisted and cylindrical bars may be produced from the interface between the wear fragment and the contact surface. Abundant twisted and cylindrical bars are certainly observed in the groove as well as the huge wear fragment, as shown in Fig. 15(d). Thus the twisted and cylindrical wear particles suggest that the severe plastic deformation and flow causing the tearing fracture is predominant in the seizure.

Figure 16 shows the wear scar and the wear particles generated in the seizure of WJ2. In the seizure, a large amount of transfer deposits from WJ2 was observed on the ring surface. On the wear scar of WJ2, the surface layers, probably formed due to the plastic flow and the back transfer, crack and separate as plates, as shown in Figs. 16(a,b). Plate-like wear particles having the same surface feature are observed as shown in Figs. 16(c,d). The size is mostly less than 50 μm . The SEM observation results suggest that the cracking and the brittle fracture are predominant in WJ2 and the wear particles detach easily from the surface. In contrast, the tearing fracture resulting from the severe deformation

Fig.16 SEM micrographs of wear scar of WJ2 and wear debris generated from WJ2 in seizure; (a) overview of wear scar; (b) enlargement of region indicated by blue circle in (a); (c) overview of wear debris; (d) enlargement of region indicated by yellow circle. The arrow indicates the relative direction of motion of the counterface

and plastic flow is predominant in the PEEK composite. The wear particles do not detach easily from the surface and tends to adhere again to the surface due to probably the gummy property or static electricity. The difference in the wear mechanism may lead to the difference in the number of the wear particles generated in the seizure process.

When the particle counter method is applied to analyze the seizure process of the PEEK composite, the results in this study show that there are two problems to be solved. One is the time lag of 1-2 minutes required for detecting wear particles, and the other is that wear particles might be not generated from the composite in the mixed lubrication. Although the time lag can be reduced, it is not avoidable because oil samples have to be sent to the counter. However, the particle counter measuring wear particles in a real time is more efficient than batch types of wear debris analyses. This study is the accelerated tests that the experimental conditions are very severe and certainly lead to seizure soon. If the growth of wear damages is not so fast, this method may be useful. In a practical situation, operating conditions are mild and decided to maintain low friction and wear for a long time. On the second problem, a lot of wear particles are generated from the ring in the mixed lubrication if the ring's surface roughness is more than about $0.5 \mu\text{m } R_a$ [12,16]. In contrast, wear particles are not appreciably generated from the ring when the ring is smooth. Wear particles generated from the composite are also observed although the amount of generation is a lot less than that of the steel ring. These results suggest that the smooth ring used in this study might not cause appreciably the composite's wear. Thus it is difficult for the wear debris analysis to detect the mixed lubrication

Fig.17 Seizure behaviors in PEEK composite

in the PEEK composite when the ring is smooth. Figure 17 illustrates the relationship between the friction coefficient and the ring temperature in the smooth ring. The same test was conducted four times. Although the data are a little scattered, there is a good correlation between the friction coefficient and the ring temperature. The seizure process strongly depends on the ring temperature and can be roughly divided into three regimes: hydrodynamic lubrication, mixed lubrication and seizure. Figure 17 suggests that the measurement of the ring temperature may be useful to detect the mixed lubrication. These are the next subjects in a future.

4. Conclusions

The seizure behaviors of the PEEK materials were studied at the high sliding velocity under the oil-lubricated conditions. The seizure processes were monitored with the particle counter. The following conclusions were obtained.

- (1) The particle counter method could detect the seizure in the PEEK materials as well as in WJ2. However, the number of the wear particle generated in the PEEK materials was much less than in WJ2.
- (2) The wear particle was generated in the mixed lubrication before transiting to seizure in WJ2. In contrast, it was not detected appreciably in the PEEK materials.
- (3) The PEEK composite had the excellent ability to maintain the mixed lubrication than WJ2 and the PEEK.
- (4) Twisted and cylindrical wear particles were commonly observed in the seizure of the PEEK materials. The severe plastic deformation and flow causing the tearing fracture were predominant in the seizure of the PEEK materials.

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