

Conditions for Transfer Film Formation and Its Effect on Friction Coefficients in NAO Friction Materials Containing Various Abrasive Components

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The non-asbestos organic friction materials with various abrasives such as ZrO_2 , $ZrSiO_4$, Al_2O_3 , Fe_3O_4 were manufactured and underwent friction tests to find conditions for transfer film formation and their effect on friction coefficients, and also the effect of different abrasive components was analyzed. Friction tests were composed of the effectiveness test and the fade & recovery test successively and counterpart disks were examined at each stage to find transfer film distribution changes. Friction surface observation by SEM revealed that the transfer film was formed not in the effectiveness stage which simulated the normal braking condition, but in the fade stage when temperature rose up to 600°C. The level of friction coefficients for most friction materials turned out to be opposite to the quantity of transfer film formation implying that the transfer film reduced the friction coefficient. High temperature condition was requisite for transfer film generation, but the transfer film was hardly formed irrespective of the magnitude of deceleration rate under moderate operating conditions where temperature stayed below 350°C. Among the friction materials tested, friction material which contained Al_2O_3 produced the least transfer film but showed the highest friction coefficient throughout the entire stages of the friction test.

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NOMENCLATURE

N = normal force applied to a brake pad

F = frictional force

μ = coefficient of friction

T = brake torque

A_f = percentage of friction surface covered with transfer film over the total friction surface under investigation, %

g = gravitational acceleration, 9.8m/s²

1. Introduction

One of the most frequently used brake systems consists of brake

pads and rotating disks, sometimes called rotors. The brake pad is composed of a friction material and a back plate which holds the friction material. And the friction between the friction material and highly rotating rotors induces deceleration of vehicles.

Friction materials can be classified as metallic, semi-metallic, low steel, non-asbestos organic (NAO) depending on constitute materials and manufacturing process. The NAO material is composed of several constituents such as fiber materials, binders, filler materials, abrasive materials and lubricating materials. Frictional characteristics of the friction materials vary with kinds and amount of constituents involved in the materials, and the abrasive components among all the constituent components are known to affect significantly the level of friction coefficient.¹ Considering that the friction coefficient is closely related to microscopic friction surface configuration, friction materials with various abrasives such as ZrO_2 , $ZrSiO_4$, Al_2O_3 , Fe_3O_4 respectively, were manufactured in the present study to study the effect of abrasives on

friction surface characteristics.^{2,3}

Several researches on friction surface observation reported the existence of a third body on surface of disks or pads called either friction film, oxidation film or transfer film which is believed to be formed during friction and to have combined components with those of disks and pads.⁴⁻⁹ The friction characteristics can be influenced by the existence of the transfer film therefore it is essential to know the effect of transfer films on friction. However, under which braking circumstances the transfer film would be formed or deteriorated has not been clearly known. Moreover, the effect of the transfer film formation on subsequent friction coefficient changes has been in dispute.

The main purpose of the present study is to seek for conditions for transfer film formation and its effect on friction coefficients in organic friction materials. For the purpose, friction tests composed of three successive stages; the effectiveness, the fade and the recovery stages were performed. Microscopic observation of friction surfaces on the counterpart disks was followed at all the friction stages to monitor development and deterioration of the transfer film, which was believed to reveal suitable conditions for transfer film formation. Effect of the transfer film formation on friction coefficients can be accessed by comparing the quantity of observed transfer film formation with friction coefficients in each stage of the tests. The secondary purpose of the present study is to access the respective effect of various abrasive components on transfer film formation and friction coefficients, which was originally aimed at development of friction materials with high friction coefficients.

Friction tests were composed of successive three stages of the tests; the effectiveness, the fade and the recovery tests, and three stages of counterpart disks used for testing the four different friction materials were collected to monitor the transfer film generation and deterioration.²⁻⁴ The effectiveness stage was intended to simulate normal braking conditions such as during street driving. Brake operations were applied at initial temperature of 80°C with various initial speeds and deceleration rates in the effectiveness stage. After the braking, the temperature remained below 350°C in most cases. The fade stage was intended to simulate harsh situations when a car is descending from a high mountain. The temperature rose up to 600°C due to consecutive braking operations during short time interval. In the fade stage, 10 consecutive braking operations with relatively high deceleration rate of 0.45 g were applied with short time interval of pauses, and friction coefficients decreased with increase in friction temperature. The recovery stage simulated the braking conditions when a car finished descending from a high mountain and temperature returned to normal. In the recovery stage which followed directly after the fade stage, more relaxed braking operations with long time interval of pauses resulted in decrease in temperature to normal.

Collected disks were sectioned carefully so as not to damage the friction surface developed during friction tests, and the specimens were hot mounted and polished for electron microscopic observation. Energy dispersive X-ray (EDX) was used when chemical composition analysis was needed to identify the existence of the transfer film on friction surface. Through the analysis of more than 100 pictures taken in each case of test stage, the fraction of area in friction surface occupied by the transfer film was measured.

Microscopic observation of disk surface in the three stages revealed

Table 1 Compositions of friction materials with 4 different abrasive components used in the present study

NAO Friction Material		%
Fiber material	Aramid, Mineral Fiber, Cu Wool etc.	15
Reinforcing material	K ₂ O ₆ TiO ₂	10
Abrasive material	ZrO ₂	9
	ZrSiO ₄	
	Al ₂ O ₃	
Binding material	Phenol resin	14
	Organic filler	Organic dust
Inorganic filler	BaSO ₄ , Ca(OH) ₂ etc.	29
Lubrication	FeS ₂ , Graphite etc.	7

quite different transfer film distributions for most friction materials tested, which implied that there are some favorable conditions for transfer film formation. Information on transfer film distribution deduced from the microscopic findings of each stage provided some of important conditions under which the transfer film can be generated or removed, and the effect of the transfer film formation on friction coefficient was investigated.^{2,3} Additionally, differences in distribution of the transfer film with various friction materials containing different abrasive components were linked to difference in friction coefficients.

2. Test Methods and Procedure

2.1 Friction material preparation and friction tests

Four friction materials which contain four different abrasive components of ZrO₂, ZrSiO₄, Al₂O₃, Fe₃O₄ respectively, with the same weight percentage of 9% were prepared while keeping the other constituents all the same. The basic compositions of the materials are given in Table 1. Rotors used as a counterpart material in the present test were made of grey cast iron with carbon content of 3.0-3.4%, Si of 1.9-2.4%, Mn of 0.5-0.9%, and P, S, Cr of less than 0.1%. The rotor matrix was pearlite with hardness level of HRB 87-98.

According to the classical theory on friction, the frictional force F is proportional to the normal force N , as $F = \mu N$, where μ is the coefficient of friction. Developing a friction material with high friction coefficients has been the major concern in the friction material industry. For a rotating disk vs. pad braking system as shown in Fig. 1, the equation for the coefficient of friction can be expressed as follows.

$$\mu = T/(N \cdot r) \quad (1)$$

Here, N is the normal force applied to a brake pad through hydraulic brake cylinder, $T (= F \cdot r)$ is the brake torque which corresponds to product of friction force F and r , where the moment arm r is a distance from the center of the rotor to the centroid of the brake pad. By measuring the brake torque at brake dynamometer, coefficients of friction can be obtained from Eq. (1). Friction force N can be varied by changing pressure in brake cylinder, and deceleration rate can be determined by V_0 / t , where t is the time period spent from initial velocity V_0 until stop.

Friction tests were conducted using 1/5 scale dynamometer in the modified JASO C406-P1 test mode as shown in Table 2.¹⁰ The friction

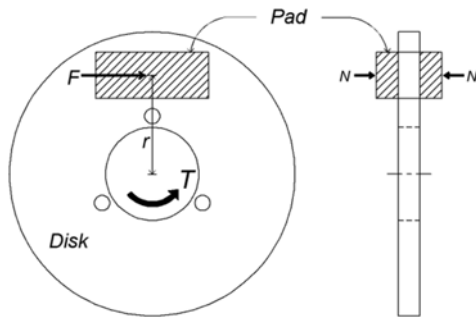


Fig. 1 Brake system for a rotating disk vs. pads

tests were composed of the effectiveness, the fade and the recovery tests. The effectiveness test consisted of breaking the pad sliding at some initial velocities at initial temperature of 80°C, with different deceleration rates from 0.1 g to 0.8 g, where g is gravity acceleration. The fade test consisted of braking with 10 consecutive decelerations with 0.45 g with interval of 35 seconds, while the recovery test following the fade test consisted of consecutive deceleration with 0.3 g with interval of 120 seconds.

Temperature during braking process was measured by K-type thermocouple installed at the center of the rotating disk. The thermocouple was located very close to the friction surface where air was blowing with constant speed of 11 m/sec.

2.2 Specimen preparation for SEM observation

Friction tests were performed successively, and three stages of counterpart disks used for testing four different friction materials were collected for microscopic observation to get the entire picture of how and when the transfer film was formed. As shown in Table 2, the three stages were composed of the effectiveness stage, the fade stage and the recovery stage. A total of 12 different rotors, in combination of 3 stages and 4 materials, were prepared and the rotors were cut by the diamond cut-off to have the size of 1 cm × 1 cm (rotor thickness of 0.7 cm) which was fit for the observation in the Scanning Electron Microscope (SEM). The feed speed of the specimen during cutting was small enough such as 0.005 mm/sec so as not to damage the preformed friction surface, and thus the possible heat deformation of the specimen during cutting was minimized. Afterwards, carbon fiber was decomposed on the friction surface with black resin during hot mounting process to prevent spread reflection of light at the friction surface. After the mounting, the specimen was grinded and polished. Sand papers of #600 and #2000 were used in the grinding process, and polishing disks in the range of 6 μm, 3 μm, 1 μm, 0.04 μm were used in the polishing process.^{2,3} Friction surface of the specimens were observed by the scanning electron microscope called FE-SEM (Field Emission Scanning Electron Microscope). Energy dispersive X-ray (EDX) was used to analyze the composition of transfer film on the friction surface.

3. Test Results and Discussion

3.1 Friction surface observation and transfer film distribution

Fig. 2 shows the microstructure of the friction surface which was

Table 2 Modified JASO C406-P1 test mode

No	Items	Velocity (km/h)		Interval (sec)	Temp. (°C)	Decel. (g)	N	
1	pre-burnish	50		120	-	0.3	10	
2	1 st effect.	50	100	-	80	0.1-0.8	E.1	
3	burnish	65		-	120	0.35	200	
4	2 nd effect.	50	100	130	-	80	0.1-0.8	E.1
5	re-burnish	65		-	120	0.35	35	
	base	50		-	80	0.3	3	
6	fade	100		35	60	0.45	10	
	recovery	50		120	-	0.3	13	
	e/point	100		-	60	0.45	2	

Table 3 Examples of chemical compositions of transfer film in the fade stage

	ZrO ₂	ZrSiO ₄	Al ₂ O ₃	Fe ₃ O ₄
C	17.1	22.9	18.4	19.2
O	14.7	17.2	19.3	16.3
Ba	9.8	10.6	12.6	15.4
Fe	45.3	43.7	40.8	41.5
Si	2.4	1.8	2.4	2
Al	0.1	0.2	1.2	0.4
Pt	2.0	2.6	-	-
S	3.0	0.3	2.5	-
Cu	5.6	-	1.2	-
Na	-	0.3	-	0.5
Ca	-	-	1.6	3.2
Mg	-	0.4	-	-

thought to represent the most typical configuration of each stage for various friction materials. In the figure, friction surfaces of two materials with abrasive components of ZrO₂ and Al₂O₃ were shown as representative cases. Microstructures of the friction surface for materials with abrasive components of ZrSiO₄ and Fe₃O₄ were similar to those of ZrO₂, hence omitted here for brevity.³ In each picture which had magnification of 5,000, the lower side of the boundary was the disk base material while the upper side was carbon fiber resin used for hot mounting. On the centerline of each picture which corresponds to the friction surface, some parts had film with thickness of around 1 μm while other parts did not have film with bare surface. To identify the film which seemed dubious either disk material, resin, or the third material, chemical composition of the vicinity of the boundary was analyzed during microscopic observation. Chemical composition of the disk material was 95% Fe, while that of the resin was 99% carbon. The composition of the film turned out to be a mixture of the friction material and the disk material, with high concentration of oxygen content sometimes. From this finding the transfer film was considered to be formed by mixture of debris from the friction material and disk material mechanically, and the high content of oxygen implied that the active oxidation process occurred under high temperature during friction. Table 3 shows typical examples of chemical composition of the transfer film in the fade stage for various materials tested.

Features of friction surface observation was that the transfer film was scarcely found at the effectiveness stages for all the friction materials, but at the fade test all the disk surface except for the Al₂O₃ case was covered with transfer film overall. At the recovery stage, the transfer film was found to diminish considerably compared to that at

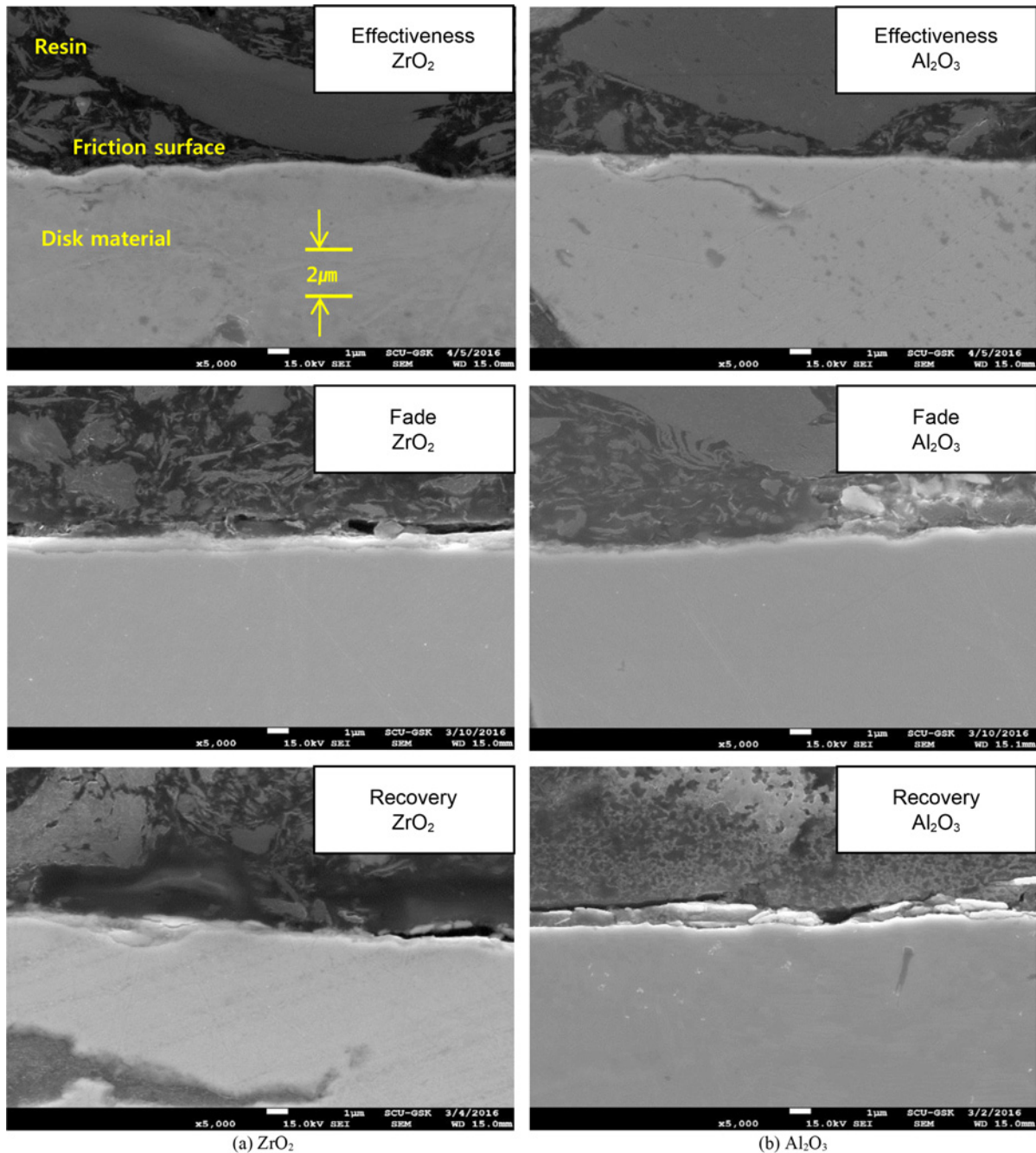


Fig. 2 Microstructure of the friction surface for various friction materials at effectiveness, fade, and recovery stage, respectively (Magnification 5,000)

the fade stage but still large amount compared to that at the effectiveness stage. To investigate the level of distribution of transfer film quantitatively, pictures such as in Fig. 2 were scrutinized if specific friction surface spot had transfer film or not. Data from more than 100 pictures in each case of the test were collected to calculate area fraction of friction surface covered with transfer film, A_f statistically.^{2,3} Here, A_f was defined as length summation of friction surface which is covered with transfer film over total length of friction surface under investigation. For instance, if a total of 0.3 cm portion among 1 cm friction surface under investigation was covered with the transfer film, A_f was 30 percent.

Fig. 3 shows A_f values at each stage of tests for 4 different friction materials. Low A_f values such as 2-3% in the effectiveness stage for most materials implied that the transfer film would not be built during normal braking condition as in the effectiveness test. The friction surface at the fade stage on the other hand, was covered with the transfer film overall with A_f values of 60-98% for most materials, which implied that the transfer film would be formed during harsh environment as in the fade test. At the recovery stage, A_f diminished by half compared to that at the fade stage for most materials, implying that part of the transfer film generated during the fade stage was removed during the recovery stage. Very few transfer film was found in all the stages for

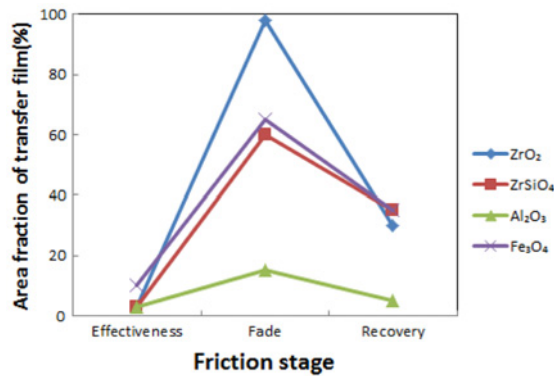


Fig. 3 Area fraction of the transfer film, A_f at each friction stage for 4 different friction materials

the material which contained Al_2O_3 abrasives.

3.2 Effect of friction temperature and deceleration rate on transfer film formation and deterioration

Transfer film distribution in Fig. 3 revealed that the film was formed not in the effectiveness stage but in the fade stage, and the film was removed gradually during the recovery stage. By examining the friction test surroundings performed, the conditions for the formation and deterioration of transfer film could be sought.

Fig. 4 shows coefficients of friction in the effectiveness test for four different friction materials, tested with different deceleration rates from 0.1 g, to 0.8 g under initial speed of 100 km/h. The initial speed was varied as 50, 130 km/h, but the friction coefficients showed practically no dependence on initial speed therefore these test results were omitted here. Shown in the picture below is the temperature right after each friction test. Temperature after friction was determined mainly by the initial speed, and the temperature level was below $350^\circ C$ even when the initial speed was 130 km/h in the effectiveness test. Recalling the scarce distribution of the transfer film in this test mode, it was worth to note that the transfer film was not formed at temperature below $350^\circ C$ in spite of fairly large amount of deceleration rate such as 0.8 g.

Fig. 5 shows variations of friction coefficients in the fade & recovery stages for four different friction materials, together with variations of temperature in friction materials during the test. Temperature changes were almost identical irrespective of friction materials. Due to nature of the fade test where successive braking was continued with short interval of pauses, temperature increased monotonically with successive braking. Recalling the prevailing transfer film at the fade stage in contrast to the scarce transfer film formation at the effectiveness stage, comparison of the two test surroundings would be essential to establish conditions for transfer film generation. In the effectiveness test deceleration rate varied from 0.1 g to 0.8 g under normal temperature below $350^\circ C$, while in the fade test deceleration rate was kept as 0.45 g under increasing temperature up to $600^\circ C$ or higher. Surprising finding from the two test surroundings was that high temperature condition was requisite for the transfer film generation but the quantity of deceleration rate was hardly effective under normal temperature below $350^\circ C$.

In the recovery test as shown in Fig. 5 temperature level at the start of the recovery test was around $350^\circ C$ and cooled down rapidly

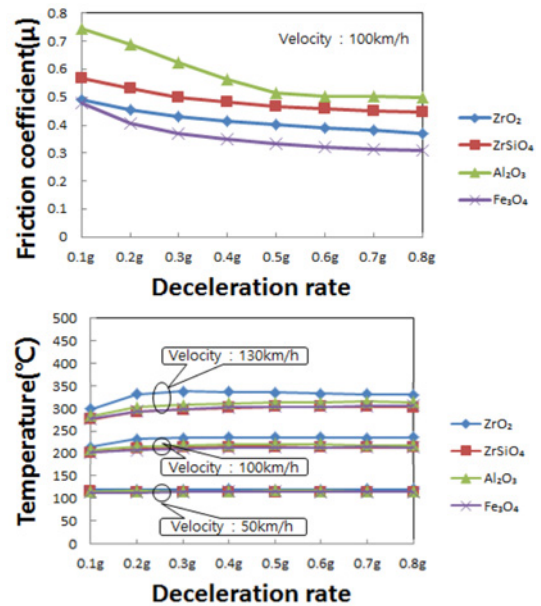


Fig. 4 Coefficients of friction in the effectiveness test for 4 different friction materials (upper figure), and temperature right after each friction test (lower figure)

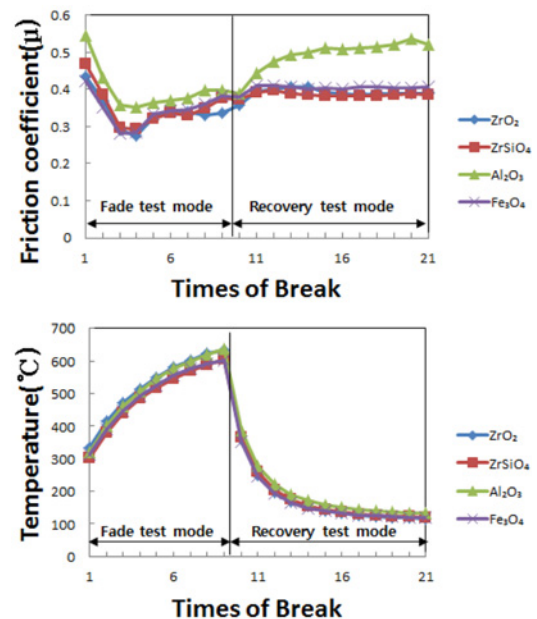


Fig. 5 Variations of friction coefficients in the fade & recovery tests for 4 different friction materials (upper figure), together with variations of temperature in friction materials (lower figure)

thereafter. The transfer film distribution in the recovery stage turned out to diminish considerably from the amount found at the fade stage, implying that the transfer film generated during the fade surroundings was removed during recovery test surroundings. Temperature level in the recovery test was similar to that in the effectiveness test, and the deceleration level of 0.3 g in the recovery test was rather milder than those of 0.1 g to 0.8 g in the effectiveness test. Findings above implied that in the normal braking conditions with moderate temperature the

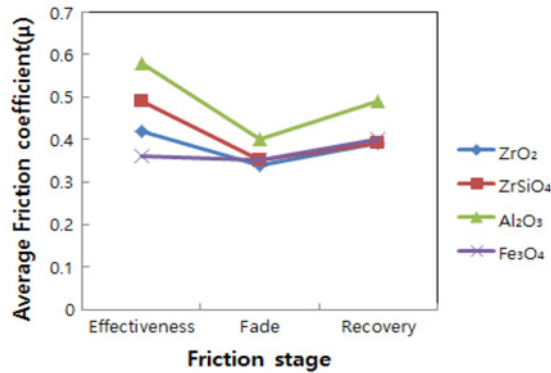


Fig. 6 Average friction coefficients at each friction stage for 4 different friction materials

transfer film tends to be removed rather than to be generated, and eventually would become extinct unless the pads meet another fade surroundings.

3.3 Effect of transfer film formation on subsequent friction coefficient

The average friction coefficients for four different friction materials at each friction stage were shown in Fig. 6. Combination of Figs. 3 and 6 revealed that the transfer film formation reduced friction coefficients from 0.35-0.65 in the effectiveness stage to 0.35-0.4 in the fade stage for most friction materials tested. In the recovery stage when the quantity of the transfer film distribution, A_f diminished by half of the fade stage, friction coefficients of most friction materials remained in the level of 0.4-0.5 which was lower than those in the effectiveness stage but higher than those in the fade stage. This again revealed that the transfer film formation reduced friction coefficients.^{2,3}

3.4 Effect of different abrasives on transfer film formation

Friction coefficient of friction material which contained Al_2O_3 was the highest among various friction materials tested in all the stages as shown in Fig. 6. Distribution of the transfer film in Fig. 3 showed that the material which contained Al_2O_3 had the least level of the transfer film distributions throughout all stages. In the recovery stage for instance, distribution quantity of the transfer film, A_f was 5% in the material which contained Al_2O_3 , while in the other materials A_f was 30-35%. This may agree with the findings that formation of the transfer film reduced friction coefficients.

The reason for the scarce distribution of transfer film in the material which contains Al_2O_3 among all materials can be sought by differences in hardness level of the abrasives. The Moh's hardness level of Al_2O_3 is over 12, while those of other materials are under 11.¹¹ The hard particle seemed to gnaw away the transfer film easily thus suppress the stacking of the transfer film. This seemed to explain why the friction material which contained Al_2O_3 showed considerable increase in friction coefficient with continued braking in the recovery stage as shown in Fig. 5. For friction materials containing Fe_3O_4 , ZrO_2 , $ZrSiO_4$ whose hardness levels were below Al_2O_3 , the transfer film were well developed and friction coefficients of these materials were lower than that of Al_2O_3 containing material.

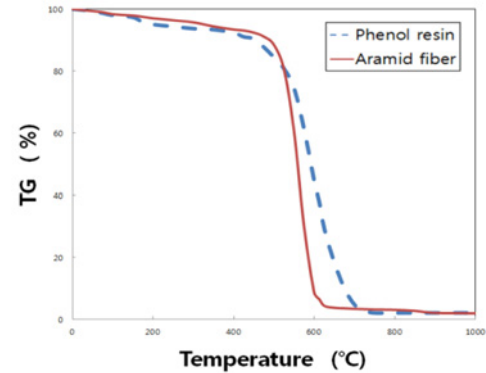


Fig. 7 Thermal-gravimetric weight percentage vs. temperature for phenol resin and aramid fiber

3.5 Reason for the active transfer film formation at high temperature

Fairly well developed transfer film at the fade stage for most of organic friction materials was attributed to the active thermal decomposition of pad material at high temperature. As shown in Fig. 5, temperature at the fade stage rose up to 600°C at which some components which comprise the friction materials were believed to decompose. Among various components that constitute the organic friction material, the weakest components at high temperature were known to be phenol resin or aramid fiber used in the study.^{11,12} Thermo-gravimetric (TG) analysis was performed by using TG-DTA equipment to investigate thermal decomposition of the components. Fig. 7 shows the TG weight percentage remaining versus temperature, for phenol resin and aramid fiber, respectively. The onset decomposition temperatures were a little over 500°C, and more than 50% of the weights were decomposed at 600°C for both of phenol resin and aramid fiber materials. Due to thermal decomposition of these components at high temperature, lots of debris generated from pads seemed to be stacked on the counterpart disk surface, which eventually would form transfer film.

4. Conclusion

In an effort to find the conditions for the transfer film formation and its effect on friction coefficients, the organic friction materials with various abrasives such as ZrO_2 , $ZrSiO_4$, Al_2O_3 , Fe_3O_4 respectively, were manufactured and underwent various friction tests. Friction tests were composed of the effectiveness, the fade and the recovery stages successively, and three stages of counterpart disks were collected. Collected disks were observed by SEM to find the transfer film distribution. From the analysis following conclusions were drawn;

For most of friction materials tested the transfer film was not found in the effectiveness stage which simulated the normal braking condition, but on the other hand the friction surface was covered with the transfer film overall at the fade stage when temperature rose up to 600°C and the coefficient of friction reached the minimum. At the recovery stage following the fade stage, the transfer film distribution quantity, A_f diminished by half compared to that at the fade stage for most materials.

Comparison of the transfer film distribution with friction test surroundings revealed that high temperature condition was requisite but the transfer film was hardly formed irrespective of the magnitude of deceleration rate under normal operating conditions where temperature stayed below 350°C.

Friction coefficients for most friction materials were the highest in the effectiveness stage, lowest in the fade stage, and in the middle in the recovery stage. Noting that the distribution quantity of the transfer film, A_f was opposite to the friction coefficient level, the transfer film turned out to reduce the friction coefficient.

Among the friction materials tested, friction material which contained Al_2O_3 component produced the least transfer film, but had the highest friction coefficient throughout all the tests. This was attributed to differences in hardness level of the abrasive components used in the study.

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