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The role of transfer layers on friction characteristics in the sliding interface between friction materials against gray iron brake disks

M.H. Cho[a], K.H. Cho[a], S.J. Kim[b], D.H. Kim[c] and H. Jang[a,]*

[a]*Department of Advanced Materials Engineering, Korea University, 5-1, Anam-dong, Seongbuk-gu 136-701, Seoul, South Korea*
[b]*Materials Research Team, Advanced Technology Center, R&D Division for Hyundai Motor Company & Kia Motors Corporation, Gyeonggi-Do 445-706, South Korea*
[c]*Advanced Material Research Team, R&D Center, Hankook Tire Co., Daejeon 305-343, South Korea*

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The effect of transfer layer formation on friction performance was studied using a brake friction material containing 15 ingredients. Based on a base formulation, 13 friction material specimens containing different relative amounts of ingredients were produced and they were tested on gray iron disks using a small-scale friction tester. A non-destructive four-point probe technique to measure electrical resistance of the thin film was used to estimate the transfer layer thickness. Results showed that the transfer layer formation was highly dependent on the relative amount of ingredients in the friction material and temperature. Among various ingredients, solid lubricants and iron powders increased the transfer layer thickness but no apparent correlation between transfer layer thickness and the coefficient of friction was found. Strong influence from individual ingredients was observed, dominating the friction characteristics during sliding. On the other hand, the thick transfer layers on the disk surface tended to reduce the friction material wear and the amplitude of the friction coefficient oscillation during sliding.

KEY WORDS: transfer layer, brake performance, friction materials, friction oscillation, wear

1. Introduction

Two surfaces in physical contact normally experience the transfer of atoms when they are rubbed against each other. The direction and amount of atomic flux during the material transfer is determined by bonding characteristics of the atoms at contacts, chemical and electrostatic states of mating surfaces, and local stress distribution at the junctions [1–3]. The material transfer at the rubbing interface is vigorous at dry sliding conditions and it often forms transfer layers on the sliding surfaces. The transfer layer mediates the interaction of the two surfaces during sliding and the tribological properties at the sliding interface can be changed significantly by the presence of the transfer layer.

In the case of a sliding situation involving composites, it turns into more complicated situations in terms of material transfer at the sliding interface. This is because the composite contains multiple constituents having different properties and morphology. Automotive friction materials sliding on a gray iron disk is a typical example of the multi-component composite material since they contain more than 10 ingredients to meet requirements for safe and comfortable brake performance [4,5]. The transfer layer on the gray iron disk, therefore, contains numerous constituents and it is known that the transfer layer plays crucial roles in brake performance. The transfer layer produced during brake applications ranges from submicron to several hundred micron in thickness and highly dependent on the braking condition and ingredients in the friction material [6–11].

Several experimental works were performed to investigate the role of transfer layers on friction and wear of brake friction materials and gray iron disks considering physical and chemical properties of the two mating materials. Rhee *et al.* [6] reported that the transfer layer on the disk surface from semi-metallic friction materials tended to generate squeal noise when the stable transfer layer exists in the temperature range of 100–200 °C, suggesting that the transfer layer may be not desirable for brake noise. They also correlated the cohesive and adhesive strength of friction film with propensity of friction film formation, suggesting that the thickness and uniformity of the transfer layer changed with the sliding condition and the layer thickness affected wear rate. They also reported that the composition of the transfer layer changes through the thickness of the layer [8]. Wirth *et al.* [9] investigated the composition of the disk surface after brake tests and suggested that the friction performance was affected by the composition of the transfer layer and the thickness of the transfer layer did not affect the friction characteristics. They also reported that the presence of the stable transfer film improved the wear rate. On the other hand, Jain and Bahadur [12] reported that the coefficient of

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*To whom correspondence should be addressed.
E-mail: hojang@korea.ac.kr

friction changed according to the transfer layer formation and the transfer layer thickness reached a steady state during the test. Smurugov [13] showed that the transfer layer formed at an initial stage affected the friction characteristics significantly and further thickness increase had little influence on the friction properties. Filip *et al.* [10] also reported that the structure and composition of the transfer layer were significantly different from the bulk friction materials. Friction films on the commercial pad surface were studied by Eriksson *et al.* [14,15] using light optical interferometry and other analytical techniques and they found that the plateau formation in the friction film was closely related with squeal generation and wear process of friction materials. The friction film on the semi-metallic pad was investigated by Österle *et al.* [11,16] using focused ion beam (FIB) techniques and transmission electron microscopy (TEM) and they reported multiple layers in the friction film on the pad surface.

While the detailed mechanism of transfer layer formation has not been fully understood yet, it is generally accepted that the transfer layers are formed on the gray iron disk surface by adhesion and compaction of wear debris and solid lubricants in the friction material having polarity on the metal surface are considered to play an active role in the attachment of wear debris on the gray iron disk surface [17,18]. It is also known that the composition and thickness of the transfer layer can be changed by the physical and chemical reactions occurred at the sliding interfaces. Therefore, the thickness of the transfer layer seems not sustained at a certain thickness but keeps changing according to the temperature, pressure, and environmental conditions at the friction interface. However, systematic approaches to investigate the transfer layers considering the ingredients and braking conditions are limited despite the crucial role of the transfer layers in wear resistance of friction materials and friction characteristics including noise propensity and roughness of a brake system.

In this work, the role of friction material ingredients on transfer layer formation was studied by employing a four-point probe resistance measurement technique to estimate transfer layer thickness on the disk surface instead of using destructive metallographic practices. Using a friction material containing typical ingredients of a commercial brake friction material, we also studied the possible correlation between transfer layer thickness and various aspects of friction performance such as the coefficient of friction, oscillation of the friction coefficient, and wear resistance.

2. Experimental procedure

Friction material specimens used in this experiment were produced based on a non-asbestos organic type brake friction material containing 15 ingredients (table 1). Based on the base formulation in the table 1, thirteen friction material specimens (1 original and 12 modified specimens) were produced. Modified specimens were produced by increasing the amount of one ingredient by 100 vol.% while the amounts of the other ingredients were proportionally decreased based on the initial formulation. Among 15 ingredients, quantities of 12 ingredients were changed for modified specimens and the amounts of other 3 ingredients (barite, calcium hydroxide, and vermiculite), that were considered as relatively inactive for friction characteristics, were fixed in this study. Friction material specimens were manufactured according to the conventional procedure for a commercial brake pad for a mid-size passenger car. The manufacturing procedure comprised mixing, hot pressing, and heat treatment and the detailed procedure for manufacturing a commercial brake friction material can be found elsewhere [4,19].

Friction tests were carried out in a constant interval test mode. A detailed test procedure used in this work is given in table 2. Friction tests were performed using a small-scale friction tester, which consisted of a rotating gray iron disk and two pieces of friction materials pressed against the disk using a hydraulic press (figure 1). Friction tests were carried out in a room maintaining temperature at 23 ± 5 °C and relative humidity at 55 ± 10%. The gray iron disk was 12 cm in diameter and 2.5 cm in thickness and showed A-type graphite morphology on a pearlite matrix. The size of the friction material specimen was 2×2×1.2 cm and the total apparent contact area on the disk was 8 cm^2. The composition of the gray iron disk is given in table 3. Wear rate of the friction material was obtained by

Table 1.
Ingredients in the friction material used in this work.

Classification	Ingredients [vol.%] (Mohs hardness)	Vol.%
Reinforcing fibers	Aramid pulp [5](7), Steel fiber [4](5.5), Mineral fiber [6](6)	15
Binders	Straight novolac resin [6](2.6), Epoxy modified phenolic resin [6](2.9)	12
Lubricants	Graphite [8](2), Molybdenum Disulfide [2](1.5)	10
Abrasives	Zircon [2.5](7), Magnesia [2.5] (6)	5
Friction modifiers	Cashew particles [8](2.2), Iron powder [2](4.5), Rubber powder [5](2.5), Calcium hydroxide [2](1.5), Barite [25](3.5), Vermiculite [16](1.5)	58

Numbers in the square brackets and parenthesis indicate the vol.% in the base formulation and Mohs hardness of ingredients.

Table 2.
Friction test procedure used in this study.

1. Burnishing: Initial brake temperature (IBT) = 100 °C, Speed = 6.9 m/s, Pressure = 0.82 MPa, Time = 600 s (10 s drag–10 s interval, 30 times)
2. Constant Interval Test: IBT = 100 °C, Speed = 6.9 m/s, Pressure = 0.82 MPa, Time = 600 s (10 s drag–10 s interval, repeated 30 times)

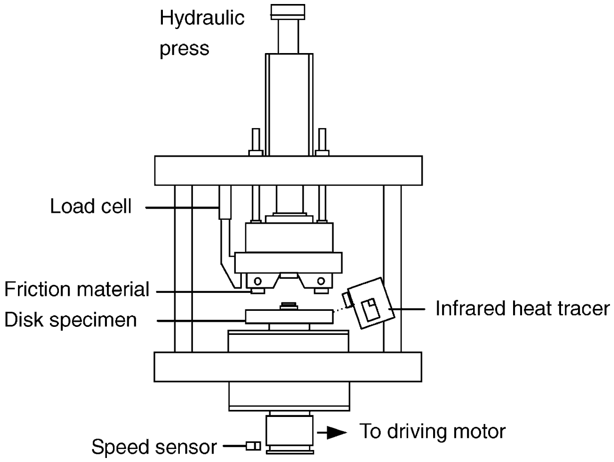

Figure 1. A schematic diagram of the friction tester used in this study.

normalizing the wear amount (mm^3) by friction energy involved at the sliding interface (Nm). This is because the wear rate of the friction material is determined by the friction energy required to stop a vehicle and the friction energy is determined by the inertia of a moving vehicle during brake applications. For the wear tests, the temperature was maintained at 250 ± 20 °C by controlling the sliding speed in the range of 1.5–4.0 m/s. Temperature of the disk was measured using an IR heat tracer (3M IR-16). The test was carried out in a drag mode at 0.7 MPa of pressure and the sliding distance was set at 3000 m.

The transfer layer thickness on the disk surface was measured using a four-point probe technique of measuring electrical resistance of a film [20]. This was based on the fact that the transfer layer on the disk surface was a thin multiphase nano-composite representing the constituents of the friction material and gray iron and that electrical resistance of the non-asbestos organic friction material was high. The high electrical resistance of the non-asbestos organic friction material was ascribed to the binder resins (resistivity: $\rho = 1.0\times10^{10}$–$1.0\times10^{16}\ \Omega$ m) and inorganic ingredients with high electrical resistance. The transfer layer, on the other hand, showed resistivity in the range of 10^{-1}–$10^{1}\ \Omega$ m according to the composition and thickness of the transfer layer. This is because the high conducting constituents such as steel, graphite, and iron, play major roles in determining the electrical resistance of the film. Electrical resistance of the transfer layer was measured through the thin silver film coated on top of the transfer layer in the size of 7×7 mm. A relationship between the transfer layer thickness and electrical resistance was obtained by measuring the electrical resistance of a series of transfer layers having different thickness using a friction material with a base formulation. The thickness of the transfer layer was measured by using a scanning electron microscope (SEM) after polishing the cross section of the disk. The transfer layer thickness was obtained by averaging the electrical resistance from 10 different areas equally spaced in a sliding track on the disk surface. Figure 2 shows an SEM micrograph exhibiting the transfer layer on the disk surface after a friction test. It shows a silver coat, a transfer layer, and a typical microstructure of the gray iron substrate. An approximate linear relationship between the transfer layer thickness and electrical resistance of the transfer layer was obtained (figure 3): Resistivity = 6.21 Ω m. Although the linear relationship in the figure 3 was obtained from the base formulation, the same resistivity was employed to estimate the transfer layer thickness throughout this study to avoid repetitive metallographic processes and observations. The deviation of resistivity due to the compositional difference of the modified friction materials was less than ± 10% since the amounts of the ingredients changed for modified friction materials were small. Despite inherent errors, this method turned out to be a reasonably reliable technique to estimate the transfer layer thickness without destroying the disk specimens. This method, however, was not applicable when the thickness of a transfer layer was less than 10 μm due to roughness of the disk surface.

3. Results and discussion

3.1. *Effect of sliding temperature on transfer layer formation*

Material transfer at the sliding interface is a dynamic event and is determined by mechanical and chemical interactions at the rubbing surfaces. Although experimental evidence for the mechanism of transfer layer

Table 3.
The composition of gray iron disk used in this work (wt.%).

C	Si	Mn	Cr	Cu	S	P	Sn	Mo	Fe
3.6	1.8	0.5	0.2	0.25	0.08	0.09	0.05	0.05	Bal.

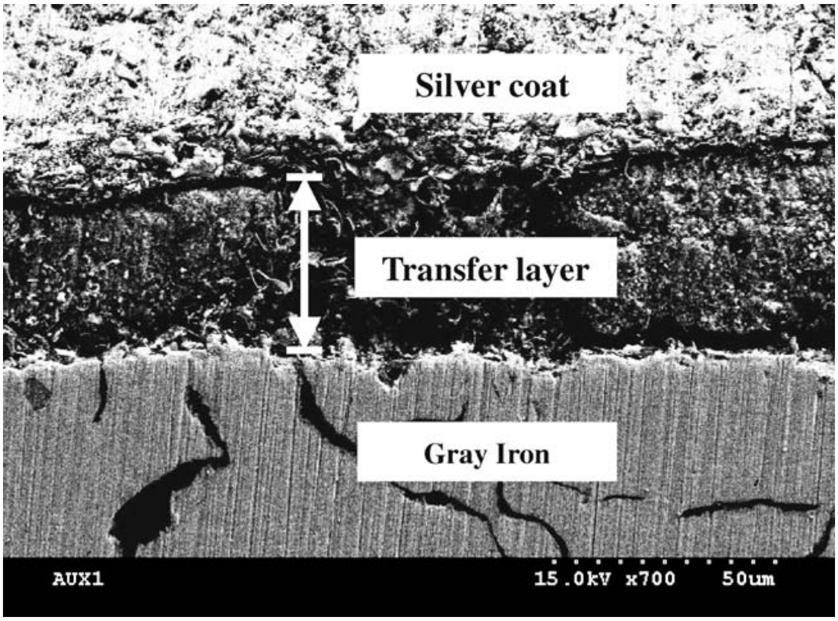

Figure 2. An SEM micrograph of a cross section of a disk specimen showing transfer layer and silver metal layer coated to measure electrical resistance of the transfer layer.

formation during brake applications is still fragmentary, it is believed that the transfer layer is transient and its thickness is strongly affected by the temperature at the sliding interface. In order to investigate the effect of sliding temperatures on the formation of the transfer layer on the gray iron disk, the transfer layer thickness was measured after carrying out friction tests at several different temperatures using a base formulation (specimen A) (figure 4). The figure indicates that the transfer layer thickness is sensitive to the temperature The transfer layer thickness increased up to approximately 250 °C and decreased rapidly beyond 300 °C. This result suggests that, at low temperature, the transfer layer is formed by mechanical attrition and simultaneous attachment of wear debris from friction materials onto the disk surface. On the other hand, the increase of the transfer layer thickness at elevated temperatures indicates active chemical adhesion of wear debris on the gray iron counter surface. The removal of the transfer layer at the temperature above 300 °C in figure 4, on the other hand, is attributed to the loss of adhesion in transfer layer due to thermal decomposition of organic binders and solid

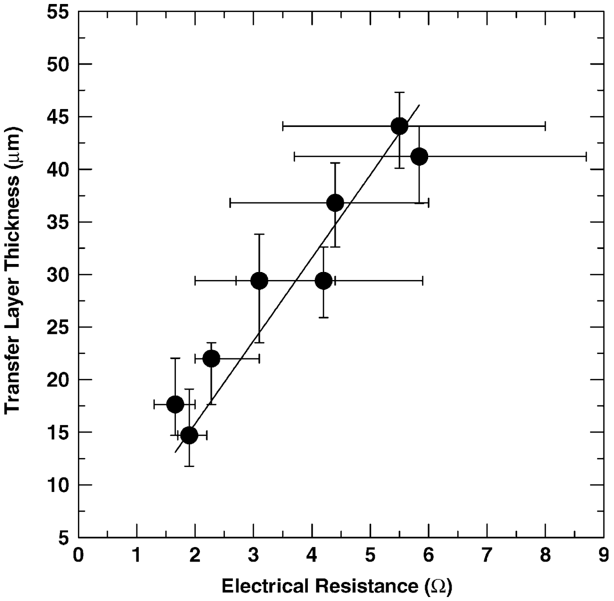

Figure 3. The relationship between electrical resistance and the transfer layer thickness. An approximate linear relationship (resistivity = 6.21 Ω m) was found in this work.

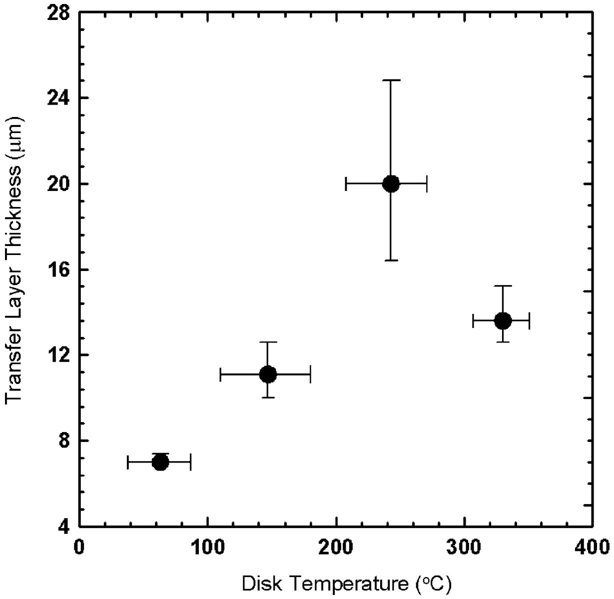

Figure 4. The thickness of transfer layers as a function of disk temperature.

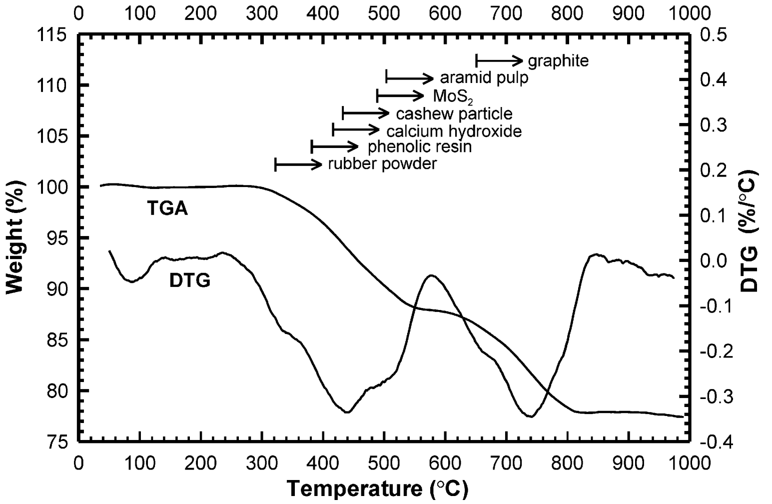

Figure 5. Thermogravimetric (TG) and differential thermogravimetric (DT) analysis of the friction material A (base formulation) used in this study. Decomposition temperatures of the degradable ingredients were indicated.

lubricants [8,20,21]. Thermogravimetric analysis (TGA) of the friction material was carried out to find a correlation of the transfer layer thickness with thermal decomposition of the ingredients in the friction material (figure 5). It shows that a major weight reduction begins at 300 °C and it continues up to 500 °C before the second major weight reduction takes place near 600 °C. Differential thermogravimetric analysis (DTG) also indicates that the thermal decomposition of the ingredients takes place in two different temperature ranges. The first weight reduction in the figure 5 is attributed to the thermal decomposition of organic binders, aramid pulp, and MoS_2. The second major reduction mainly represents the oxidation of graphite [22]. This result supports the fact that the decrease of transfer layer thickness above 250 °C in the figure 4 is ascribe to the first major decomposition of the ingredients in the figure 5. The temperature deviation between the decomposition temperature from thermal analysis and the temperature showing destruction of transfer layers suggests that the flash temperature at the sliding interface is much higher than the bulk temperature measured in this experiment [1]. A similar result was reported in the study of friction and wear of simplified formulations containing phenolic resins as a binder and aramid pulps as a reinforcement [23]. It shows that the thermal decomposition begins at 250 °C and peak reduction in weight takes place at approximately 500 °C, resulting in the loss of friction force at this temperature range.

3.2. *Effect of ingredients on transfer layer formation*

Relative influence of the ingredients on transfer layer formation was examined by measuring the transfer layer thickness on the disk after friction tests using friction materials containing different amounts of ingredients. The friction test was carried out using the friction materials containing double the amount of each ingredient based on the basic formulation. The bulk temperature on the disk surface was maintained in the range of 250 ± 20 °C by controlling the sliding speed. Figure 6 shows the relative influence of the ingredients on the transfer layer formation when 1 vol.% of each ingredient is increased. The relative influence from each ingredient was obtained by normalizing the difference of transfer layer thicknesses by the vol.% increase of each ingredient. For instance, in the case of specimen C, the relative influence (-1.08 μm/vol.%) was obtained from the thickness difference divided by the increment of steel fibers in the specimen C in vol.%, that is; -1.08 (μm/vol.%) = (16.6–20.9 μm)/4 vol.%.

The result shows that solid lubricants and iron powders promote the transfer layer formation. The formation of transfer layers with solid lubricants is attributed to the polarity of the lubricant molecules on the gray iron surface [17,24]. The figure also suggests that iron powders tend to build up thicker transfer layers implying that fine wear particles from soft iron (ferrite) adhere well to gray iron disks [1]. On the other hand, abrasive ingredients with high hardness such as magnesia and zircon tended to decrease the transfer layer thickness. Thin transfer layers were also observed in the case of using friction materials containing extra mineral fibers, aramid fibers, steel fibers, and etc., due to their high hardness. Rubber particles also showed tendency of decreasing transfer layer thickness because the resilience of the rubber disturbs the compaction of wear debris on the disk surface.

3.3. *Effect of transfer layer on friction coefficient and friction oscillation*

When transfer layers are present on the disk surface, the friction coefficient is determined mainly by the interfacial layers at the rubbing surface since they play a major

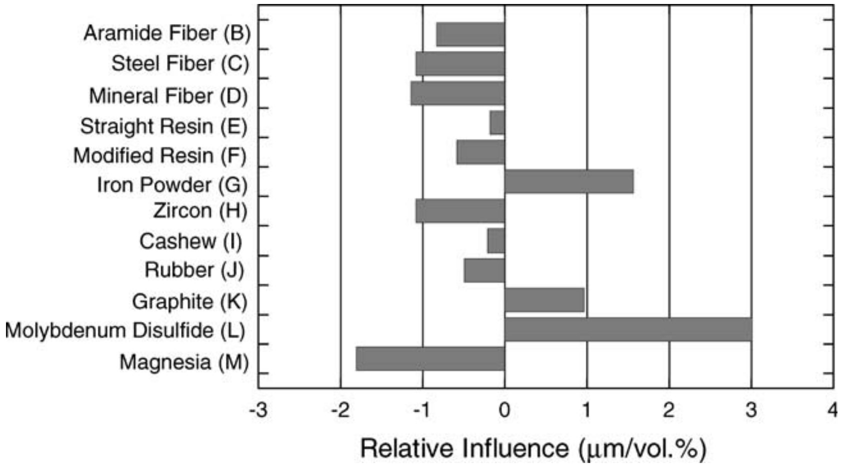

Figure 6. Relative influence of ingredients on the generation of transfer layer on the disk surface. The relative influence was obtained by normalizing the difference of transfer layer thicknesses from base and modified formulations divided by vol.% of ingredients.

role in determining the resistance to the sliding action. The resistance to the sliding motion, in this case, can be determined by three different components. They are interfacial strength of the layer between a friction material and the transfer layer, shear strength of the transfer layer, and adhesive strength at the interface between a gray iron surface and a transfer layer. The relative importance of the three factors in determining the friction characteristics is dependent on the type and size of ingredients in the friction material and the sliding conditions. In particular, the role of transfer layers on the friction characteristics can be diminished when large wear debris or hard particles bigger than the dimension of the transfer layer thickness are present at the rubbing surface.

In this study, the effect of transfer layer thickness on the coefficient of friction and amplitude of friction force oscillation was studied. Figure 7 shows the coefficient of friction as a function of transfer layer thickness. The figure shows no apparent relationship between transfer layer thickness and the coefficient of friction. On the other hand, it indicates that the friction materials containing extra volumes of hard abrasives such as magnesia and zircon show high levels of the friction coefficient. This implies that the hard abrasive particles destruct transfer layers locally and increase the coefficient of friction by scratching the disk surface in a two body abrasion mode. On the other hand, when extra solid lubricants are included in the friction material, low levels of friction coefficients are obtained. This is because the thick transfer layers prevent the direct abrasion of the hard abrasive particles and wear debris on the disk surface and, in this case, the resistance to the sliding is mainly provided by the shear strength of the transfer layer.

The influence of the transfer layer thickness on the intensity of the friction force oscillation was also examined in this work to elucidate the possible correlation between the transfer layer and brake-induced vibration (brake judder). In general, the fluctuation of friction force during sliding is ascribed to both uneven height (runout or waviness) of a sliding surface and transient surface artifacts. When the friction test is carried out on a rotating disk, it generates cyclic friction force oscillation and surface artifacts produce irregular fluctuation of friction force. The effect of disk runout and the transfer layer on the intensity of friction force oscillation is carefully analyzed in this study since this is equivalent to the effect of disk thickness variation (DTV) and transfer layers on the brake torque variation during braking in a disk brake system.

Based on the assumption that the runout of the disk can be reduced by developing a transfer layer on

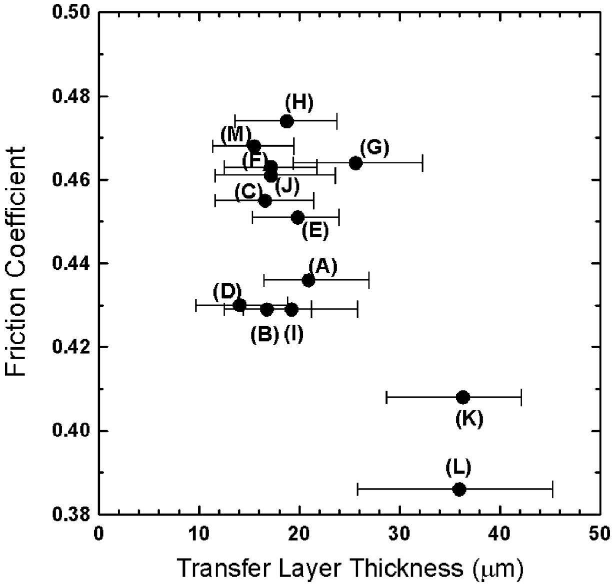

Figure 7. The friction coefficient of friction materials plotted as a function of transfer layer thickness. The letters in the data points indicate the specimen with extra ingredient in the Fig. 6: A: Basic Formulation, B: Aramid Fiber, C: Steel Fiber, D: Mineral Fiber (Rockwool), E: Straight Resin, F: Epoxy Modified Resin, G: Iron powder, H: Zircon, I: Cashew, J: Rubber, K: Graphite, L: MoS_2, M: MgO.

the disk surface, the formation of transfer layer and its preservation have attracted much attention. The postulation of the reduction of disk runout by transfer layer formation was examined in this work by using friction materials exhibiting different transfer layer thickness. The test was carried out at 250 ± 20 °C using a disk with the same amount of runout (15.4 μm). Figure 8 shows the amplitude of friction coefficient as a function of transfer layer thickness on the disk surface. The figure shows that the transfer layer tends to reduce the amplitude of the friction coefficient. On the other hand, strong influence from the tribological properties of individual ingredient is observed. In particular, bigger amplitudes were observed in the cases of using specimen D (extra mineral fiber) and F (extra resin).

The transfer layer, however, was delaminated at higher temperatures due to thermal decomposition of the ingredients and, in this condition, the amplitude of the friction coefficient increased. This is because the high temperature sliding often produces transient patchy transfer layers on the disk surface, leading to random fluctuation of friction coefficients [25]. This situation was normally accompanied by excessive friction material wear and occurred when the bulk temperature of the disks reached higher than 400 °C.

3.4. The effect of transfer layer on wear of friction materials

Wear of the friction material is mainly affected by the type of ingredients and braking conditions. This is because the friction material wear is highly dependent on the inherent wear resistance of individual ingredients and thermal decomposition. Durability of commercial friction materials during service is normally determined by the specific wear rate, which is the wear amount per energy used to snub and stop a vehicle. Therefore, the temperature, which is proportional to the friction energy at the sliding interface, is an important factor for assessing the wear resistance of the friction material and the transfer layer also closely related with the wear rate since the thickness of the transfer layer changes with temperature. In particular, the influence of the transfer layers on wear rate of the friction material, therefore, has attracted much attention among many aspects of transfer layers on various brake performances. Although much speculation has been spread among brake related research communities, experimental evidence about the role of transfer layers on friction materials wear was not found in the literature. A part of the reason for the lack of experimental results on this important issue is ascribed to the difficulty of measuring transfer layer thickness on the gray iron disk surface.

Figure 9 shows the specific wear rate as a function of transfer layer thickness. By controlling the sliding speed, tests for wear rate was carried out at 250 ± 20 °C, which was the temperature range for optimal condition for transfer layer generation during drag. A conclusive correlation between specific wear rate and transfer film thickness was not found in this study. Instead, strong influence from the tribological properties of individual ingredient was observed. Solid lubricants exhibited low wear rate with thick transfer layers and the iron powder helped to reduce the wear by developing transfer layer on the disk surface. On the other hand, other ingredients showed strong influence from the tribological properties of each ingredient. In particular, it was interesting to

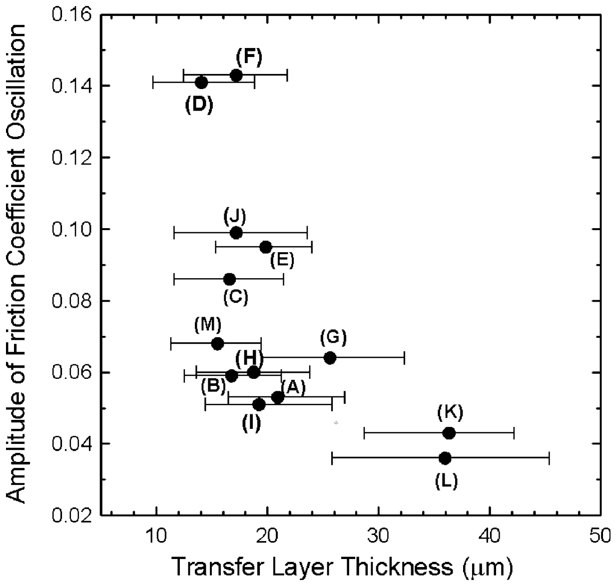

Figure 8. The amplitude of friction coefficient as a function of transfer layer thickness on the disk surface. The transfer layers were developed during sliding tests at 250 °C and the thickness measurements were carried out at room temperature after the test.

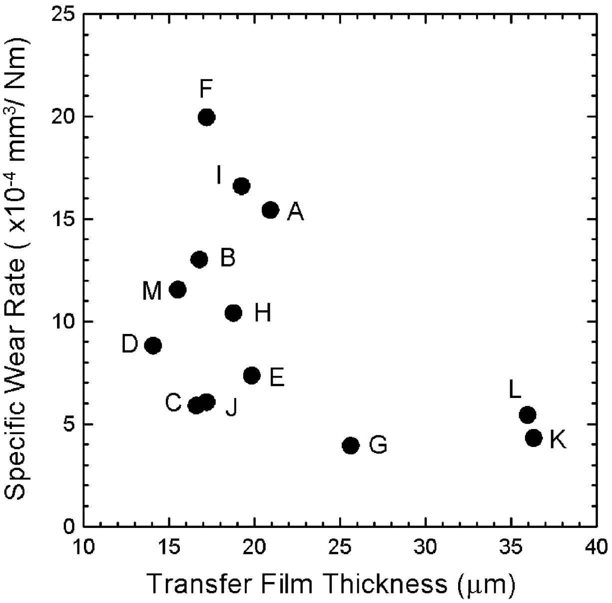

Figure 9. Specific wear rate as a function of transfer layer thickness. The wear rate was obtained by normalizing the wear amount by friction energy involved during sliding.

observe that the epoxy modified resin, which was often partially substituted with the straight resin to improve heat resistance [26], increased the wear rate of the friction material. From these results it was found that the transfer layer thickness had little effect on the wear of friction materials unless it maintained a certain thickness.

4. Conclusions

The effect of transfer layers on friction characteristics was examined with different friction materials using a small-scale tribotester. Transfer layer thickness was measured by a non-destructive four-point probe electrical resistance measurement technique. Transfer layer thickness was affected by temperature and the amount of ingredients and it showed no direct correlation with the friction characteristics. Stronger influence from the composition of the transfer layer, that is proportional to the relative amount of constituents in the friction material, was observed. Conclusions from this study are as follows:

(1) The transfer layer thickness varied with temperature. The thickness was increased up to approximately 250 °C, and decreased rapidly beyond 300 °C due to the thermal decomposition of ingredients.
(2) The thickness of the transfer layer was strongly affected by the relative amount of ingredients. In particular, solid lubricants and iron powders were effective for developing a thick transfer layer.
(3) Friction coefficient was independent of transfer layer thickness but intensity of friction oscillation was reduced when the transfer layer was thick.
(4) Wear rate of friction material was independent of the thickness of the transfer layers.

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