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# A Collaborative Program for Development of Frictional Materials Using Industrial Wastes

Yoginder P. CHUGH, Peter FILIP, Samrat MOHANTY & Kok-Wai HEE
Center for Advanced Friction Studies, College of Engineering, Southern Illinois University, Carbondale, Illinois 62901, USA

ABSTRACT: *Current frictional materials, for example automotive brakes, consist of ingredients (abrasive powders, steel or copper chips, binders etc.) with very diverse physical, engineering and thermal properties. The authors believe that several of the problems associated with such frictional materials (low contact area, noise, vibration, temperature development and dissipation) can be overcome if ingredients have smaller diversity in their properties. Furthermore, the production cost of such materials can also be reduced through large volume use of industrial wastes readily available in most countries in the world.*

*Working in cooperation with industrial partners, the authors have made considerable progress toward development and characterization of such materials over the last 4-months. Additional studies are continuing to optimize composition of such frictional materials and develop commercially viable brake pads. Industrial partners will test these materials and modify them, in cooperation with authors, to implement their production and marketing. The collaborative program involves faculty and students with diverse backgrounds and student training is an integral part of the program.*

## 1 INTRODUCTION

### 1.1 Background and need for research in friction materials

Surface interactions between two touching solids are of universal importance since solids represent a substantial portion of our world. Here, we are concerned with the friction effects of surface interactions, which represent ability of touching solids to transfer tangential force at different applied normal loads. Associated effects of interest include wear and adhesion phenomena. Wear represents removal of material from the surfaces of one or both of the contacting bodies. Adhesion implies the ability of contacting solids to withstand tensile forces upon application of normal compressive forces. A well known topical area where friction, wear, and adhesion play major roles is in the use of friction materials for braking effort to control the speed of moving bodies; e.g. automobiles, trains, etc. The term "tribology" refers to the knowledge base concerned with the mechanical surface interaction phenomena in solids.

The processes of friction, wear, and adhesion are complex and involve properties of both contacting solids. The ability of the current knowledge base to predict these behaviors from the more fundamental engineering properties is limited and more research is needed in the area. It is however well established that these properties involve volume or bulk properties of the solids as well as the more intuitive contacting interface of these bodies or surface properties. Examples of bulk engineering properties include plasticity and yield behavior and their temperature dependence, elastic properties including ability to store elastic strain energy, and the more obvious thermal properties, including conductivity and diffusivity properties.

Surface properties of importance include ability to develop a surface film with properties different than those of the substrate, ability to absorb molecules from the environment, and surface energy of the bodies in contact. It is important to note that atomic bonds play a critical role in a solid and the friction and wear properties are related to these bonds in a rather complex manner not well defined by research to date.

A few metals and alloys will form very thin film in (≈10 -5mm) air while developing very different films of nitrides, sulfides, and chlorides in other environments. These films and their characteristics play an important role in surface interactions. In addition to the surface film, metals and non-metals alike develop an adsorbed film, which primarily consists of molecules of water vapor and oxygen and has thickness of about 3x10-7 mm. This adsorbed film can reduce surface interaction properties. Surface energy of the solids also plays a role in friction and wear properties but its role is not fully understood.

Crystal structure, chemistry of the oxide layer, and stacking fault energy also influence tribological properties. Hexagonal close-packed crystalline structure results in less friction and oxide layer stiffness must be appropriately matched to volume properties of the metal to achieve a homogenous solid lubricant layer and consistent friction and wear properties.

A review of pertinent literature indicates that tribological research is still not matured and considerable research is needed to resolve complex relationships that affect development of friction and wear properties.

### 1.2 Automotive Brake Assembly and Brake Pad Friction Materials: State- of- the- Art

A typical automotive brake assembly consists of a cast iron drum or disc mating a brake pad’s friction material to develop frictional resistance to control automobile speed. During the braking effort, frictional resistance converts kinetic energy into thermal energy, which must be appropriately dissipated to maintain stable performance of braking effort. Although extremely critical, tribological behavior of the automotive brakes is not well understood. In particular, tribological contact studies as they affect friction, wear and adhesion properties, and, noise and vibration are critical.

The drum or disc typically is grey cast iron with 3-4% carbon by weight. This material offers many of the properties desired such as mechanical strength and stiffness, wear and damping properties and thermal properties.

Organic brake pads, typically used for automotive brakes, are composites fabricated through hot compaction of 10-20 different components. These components generally fall in the category of binders, structural materials, fillers, and frictional additives to stabilize coefficient of friction. A characteristic friction material mix design is often developed empirically [1]. The trial and error strategy of friction material development has considerable drawbacks and significant effort has been concentrated on finding the relationship between formulation and the performance of friction materials [1]. Optimization of polymer matrix brake lining materials can be achieved using scientific approaches [9, 10]. Only a better understanding of complex processes, however, may help in improving the design and safety of friction materials.

Brake lining materials underwent a distinct development in the recent three decades [6-8]. This was related to an effort to develop optimal materials, which would reflect the requirements determined by transportation safety, economy, environmental concerns, and increased performance demand. As a rule, the brake lining material should have i) appropriate mechanical and thermal properties, ii) adequate and stable coefficient of friction (0.3 to 0.7) in a wide range of operating conditions (temperature, pressure, environment), iii) high resistance to wear and good compatibility with the rubbing counterpart. In addition, they should operate without pollutions and noise, and vibration and should be easily manufactured at low costs

### 1.3 Authors’ hypothesis

Most of the current industrial friction materials are composed of heterogeneous constituents, which are diverse in physical and engineering properties. Heterogeneity among the components leads to differential thermal properties, surface stress concentrations, localized wear properties, and noise and vibration problems.

The authors believe that if brake composites could be fabricated with fine-size constituents with small diversity in physical and engineering properties, we will have a more efficient brake assembly with less noise and vibration, and improved wear rates. Furthermore, efforts should be directed toward minimizing their cost through appropriate use of industrial wastes.

With the principal author’s background and experience in utilization of coal combustion byproducts (fly ash, bottom ash, scrubber sludge, etc), their utilization for industrial brakes fabrication was established as a high priority. These byproducts are fine grained (mean particle size 10-30 microns) and contain several of the chemical constituents (silicon oxide, aluminum oxide, iron oxide, magnesium oxide, unburnt carbon etc.) desired in a friction material. Limited effort had already been made to incorporate fly ash in brake pad composites [11, 12, 19], which pointed towards limited success and additional research efforts in this area. About 25% by weight of fly ash successfully incorporated in automotive pad compositions. The newly formulated brakes' test samples outperformed the commercially available original equipment materials [11, 12].

It was decided to attempt to increase fly ash percentage to 50-55%. In this regard, the low specific gravity of fly ash and its wide availability enhances its potential as a friction material constituent. Steel chips have mechanical and thermal properties that are very different than friction, filler, or binder components. It is our view that the size of the steel chips and their engineering properties significantly affect tribological properties, stick-slip phenomenon, and vibration and noise performance of brake pads. The use of smaller size aluminum and/or brass chips, which have engineering and thermal properties closer to other constituents, may provide a better brake pad. Since an aluminum company is located close to the university, the use of waste aluminum chips in brake pad composition was identified as another high priority task.

Since the market for friction materials for automotive brakes around the world is very large, and CCBs and waste metal chips are available throughout the world, it was thought that any improvement in weight, cost and friction properties through the use of proposed industrial wastes should result in commercialization and quick return on investment.

This paper is concerned with development of high quality frictional materials using industrial wastes, with emphasis on automotive brakes. This research was undertaken under the auspices of the Center for Advanced Friction Studies at Southern Illinois University at Carbondale (SIUC) that is funded by National Science Foundation, Illinois Department of Commerce and Economic Opportunity, and cooperating industrial partners. The theme of industrial wastes-based friction materials was selected because Illinois and the USA produces large amounts of coal combustion by-products (CCBs) such as fly ash, bottom ash, fluidized bed combustion fly ash; metal wastes of aluminum and brass are widespread, and some of the wastes from coal processing plants may have potential for use as an ingredient in automotive brakes fabrication.

## 2 EXPERIMENTAL

### 2.1 Sample preparation

An original equipment (Ford: Crown Victoria) brake lining sample was purchased from the manufacturer. This brake lining was used as a standard reference sample to compare to the developed materials and is referred to as sample A. Table 1 shows the formulation of the two samples containing as-received fly ash from a fluidized bed combustion (FBC) unit [11, 12]. The first formulation (without potassium titanate) is referred to as sample F and an improved formulation (with potassium titanate) is referred to as sample I.

All the constituents were weighed and mixed for 10 minutes using a commercial blender. The sequence of adding each constituent was as follows: pulverised mineral fiber (PMF), Twaron, Asbury graphite, copper, iron, steel, coke, Fiberfrax, magnesium oxide (MgO), sulfur powder, Barytes (BaSO4), antimony trisulphide (Sb2S3), Styrene Butadiene Rubber (SBR), phenolic resin, and fly ash.

The final dry mixture was poured into a cylindrical mold for FAST test sample and a specially made brake mold (typical for OEM Crown Victoria) for dynamometer test sample. Both molds were hot pressed at 180oC for 15 minutes to prepare test samples. Composition and fabrication density were varied and the results were evaluated based on unconfined compressive strength and stiffness. During the pressing process, pressure was released several times to release the gases that evolved from the cross linking reaction (polycondensation) of the phenolic resin. Post-curing was carried out by placing the cylindrical sample and cast brake pad in a preheated furnace (Fisher scientific Isotem Furnace) at a constant temperature of 180oC for four hours [16].

Chugh and Mohanty [23] pursued efforts in collaboration with Filip to develop a friction material utilizing high volume of fly ash and industrial waste aluminum chips. Various compositions were tried. The relative proportions of the ingredients in attempted mixes are also indicated in the Table 2.

### 2.2 Experimental procedures

The FAST machine uses a cast pearlitic gray iron disc (diameter of 300 mm) and the brake lining test sample with dimension of 12.7 x 12.7 x 5.00 mm. The test sample was mounted on the loading arm and pressed against the flat surface of the rotating disc. The rotating cast iron disc moved with a sliding speed of V = 7m/s for 90 minutes and the temperature increased from room temperature to around 300oC. Before FAST testing, the surfaces of the test samples and the cast iron discs were ground with 320-grid sandpaper. The load was set to achieve a constant friction force. The friction coefficient was calculated by measuring normal and tangential pressures every five seconds throughout the 90-minute test. Wear rate was calculated from weight loss and thickness reduction of the samples.

Two dynamometer test procedures were used in this research, i.e. AO4D and J2430 [22]. The dynamometer test procedure AO4D consists of (i) instrument check stops (ii) 50-kmph and 100-kmph effectiveness, (iii) burnish, (iv) fade, (v) recovery, and (vi) 3150C and 4250C wear test procedures. The burnish procedure, also known as bedding or run-in, establishes a uniform contact surface between the friction material and the rotor. A friction-stabilizing layer is usually developed during this procedure. Fade and recovery procedures were performed to determine high temperature performance. A friction material may lose effectiveness as the temperature increases (fade). The recovery procedure is used to identify whether a material can recover to the baseline characteristics after the temperature decreases. The temperature of the friction surface was monitored by K- type thermocouples embedded 1 mm below the friction surface of brake lining. The test procedure J2430 consists of (i) instrument check stops (ii) burnish, (iii) 50 kmph and 100 kmph effectiveness (iv) fade, (v) hot performance, (vi) cold performance, (vii) recovery, (viii) reburnish and (ix) final effectiveness cycles.

Two samples each of A, F and I were tested using FAST machine and full-scale dynamometer using the AO4D procedure. New discs and brake linings were used for each test. For each sample two tests were run on the full-scale dyno; one runs the entire test procedure and the other is terminated after the fade test procedure. After each test, wear debris was removed from the friction surface using adhesive tape. Square samples (12.7 mm X 12.7mm) were carefully cut off from the brake lining in order to avoid modification of the friction surface for analysis for repeated FAST test. High volume fly ash samples were tested using FAST machine and one composition was tested on full-scale dynamometer using J2430 procedure.

## 2.3 Characterization of the friction surface

The topography of a friction surface was analyzed using light microscopy (LM, Nikon FX135), scanning electron microscope Hitachi S2460N environmental (SEM), transmission electron microscope Hitachi H7100FA (TEM), and X-ray diffraction (Rigaku Dmax flash B diffractometer, CuKa radiation, Ni filter, 40KV, 30mA). Both electron microscopes were supported with energy dispersive X-ray analysis (EDX). Samples were cut from the brake lining before and after the friction test in order to compare the difference. For light and scanning electron microscopy, samples were embedded into a molding resin, either parallel or perpendicular with respect to the friction surface. The samples were ground and polished (Ecomet, Beuhler) prior to analysis.

For TEM observations, the friction surfaces were coated with liquid polyvinyl alcohol (15 wt%) (PVA) and dried for 24 hours. After drying, the friction layer was stripped together with PVA coating. The PVA was soaked in 10 ml of distilled demineralized water (DDW) and shaked vigorously to dissolve the PVA. The solution was centrifuged at 100 RPM for 5 minutes. The residue (brake-surface ``friction layer" fragments) was collected onto carbon coated Formvar holey films supported by 200 mesh gold grids [17].

## 2.4 Thermal properties

The mineral spirit technique was used to measure the density and open porosity of the samples. Differential Scanning Calorimetry (DSC) is a thermal analysis technique that was used to measure the specific heat of the samples. Laser flash method and a push rod-style dilatometer were also used in this study for measuring thermal diffusivity and thermal expansion of the samples. Thermal conductivity of the samples was calculated using density, specific heat and thermal diffusivity that were measured.

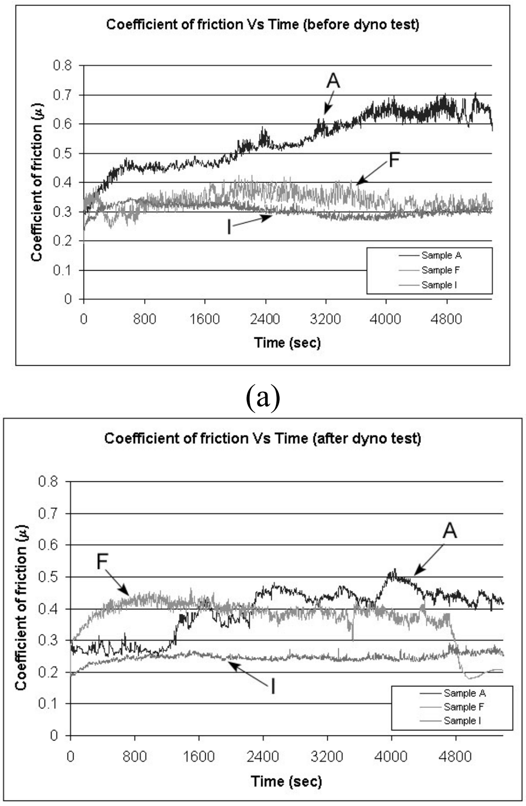
# 3 RESULTS AND DISCUSSION

## 3.1 Friction performance of A, F and I compositions (low fly ash)

*FAST testing results*: FAST tests were performed at a constant sliding speed to investigate the friction stability of the samples. The FAST results of samples A (Ford: Crown Victoria), F and I are shown in

Figure 1a. In the initial stage, the friction layer started to develop [18]. The coefficient of friction (μ) varied most significantly between 600 sec and 4200 sec of testing for all materials.

Sample A showed a significant increase in the friction coefficient. Such an increase is often associated with adhesion between the metal and abrasive material on the friction surface. This increase generally indicates unstable and aggressive friction. Some mild vibrations were observed at approximately 4100 sec of testing. The friction coefficient continued to increase until stabilization was reached at μ = 0.69 after 4200 sec of testing. High metal content in sample A dictated the character of the friction process.



(a)

(b)

Figure 1. The change of friction coefficient as a function of time for samples A, F and I (a) before and (b) after dynamometer test.

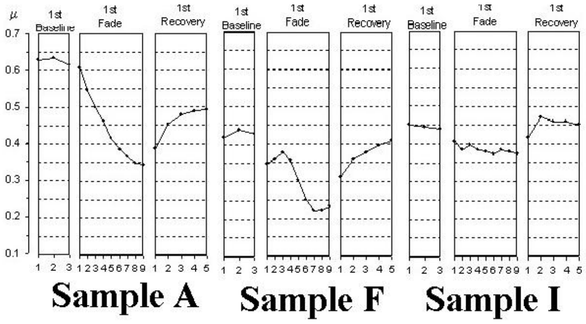
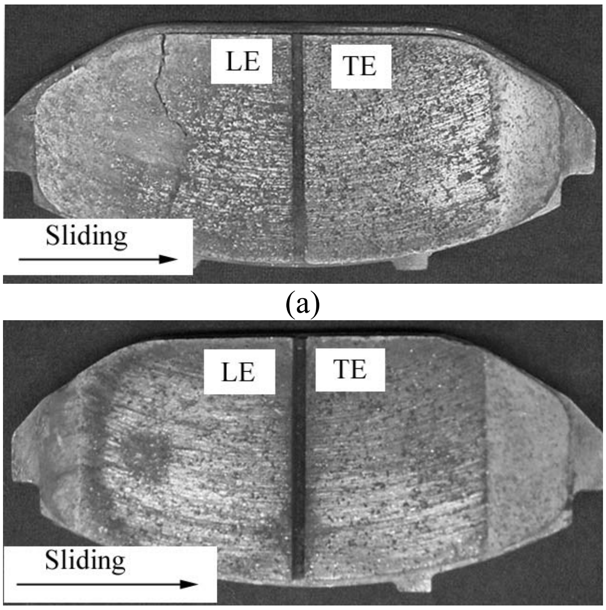


Figure 2. The dynamometer test results of samples A, F and I expressed as the average value of friction coefficient.



(a)

(b)

Figure 3. Surface of samples (a) F and (b) I brake lining after the dynamometer test (sliding direction is marked by arrow). The leading edge (LE) and trailing edge (TE) are marked on the pad.

In Figure 1a, sample F exhibited a sudden decrease in the friction coefficient after 200 sec of testing. This change was typically observed at the beginning of the test before a stable friction layer developed. It may also be a phenomenon associated with the evaporation of moisture from the sample [18]. The friction coefficient was stable after 600 sec of testing and remained at an average value of μ = 0.34. Some mild vibrations occurred between 600 sec and 4200 sec.

Sample I (with 25% fly ash) exhibited a relatively stable friction coefficient throughout the test. A stable friction layer was responsible for this behavior. Neither judder nor rapid changes of the friction coefficient occurred. Sample I completed the 90 minute test at a constant $\mu = 0.30$.

The FAST results taken after the dynamometer tests are shown in Figure 1b. The friction coefficient of sample A significantly decreased after the dynamometer test. The FAST test result demonstrated that sample A performed with unstable friction level. The performance changes are believed to be due to chemical changes in the friction layer. The friction coefficient of sample F increased slightly to 0.37 and the sample wore off before the test was completed. This indicates that the material property changed after the dynamometer test. The friction coefficient of Sample I decreased slightly after the dynamometer test. However, the friction coefficient exhibited stability throughout the entire test. It shows that sample I was able to maintain braking performance even after severe braking conditions.

Typical baseline, fade and recovery tests of samples A, F and I are summarized in Figure 2. The baseline effectiveness of sample A was exhibited in the baseline section of the test at the average value of 0.62. In the fade section, the friction coefficient of sample A dropped from 0.6 to 0.35. It was found that sample A had a significant reduction in friction coefficient at high temperature. This phenomenon is called thermal fade. Fade phenomenon is manifested as undesirable decrease in the coefficient of friction at high temperature [19, 20]. The recovery section is used to identify how quickly a material can "recover" to baseline characteristics. The value of 0.49 friction coefficient was recorded in the recovery section. Apparently, the friction coefficient was unable to recover back to the baseline value of 0.62.

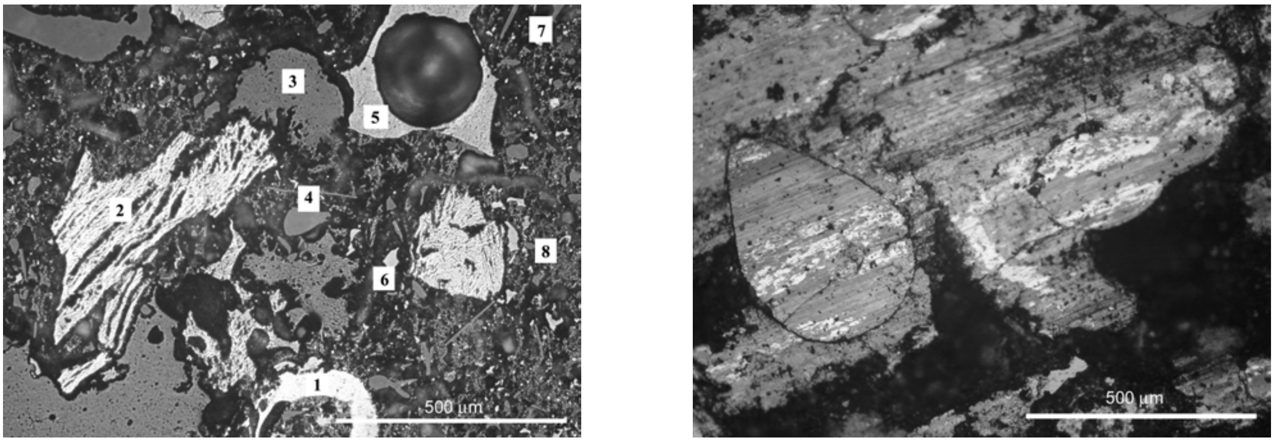
The average value of the friction coefficient for sample F was $\mu = 0.42$ in the baseline section. The friction coefficient of sample F slightly increased in the initial period at the fade section. However, this was followed by a gradual decrease and reached the lowest value of 0.23. Figure 2 demonstrates that sample F exhibited fade. The friction coefficient in the recovery section was similar to the baseline value of 0.4. It indicates that sample F is able to recover to baseline characteristic after fade.

The baseline friction coefficient of sample I was recorded at the average value of 0.45. Sample I exhibits a steady friction coefficient (average 0.38) throughout the fade section as the temperature increased. The friction coefficient increased back to the baseline characteristic of 0.45. The sample I was able to exhibit the same friction behavior even after a fade test. Samples A and F demonstrated fade, but the sample containing potassium titanate performed without fade.

The initial weight of samples F and I are shown in Table 3. These are approximately 78 grams lighter compared to the commercial brake lining material (sample A). This is because the relative quantities of metal were replaced with an equal volume of fly ash in the brake lining composition. This reduces cost by replacing expensive ingredients (like iron, steel chip, copper, brass and so on) with low cost material (fly ash). The wear rates were calculated as the weight loss after testing. As shown in Table 2, sample F exhibits high wear (approximately 50%) more as compared to samples A and I.

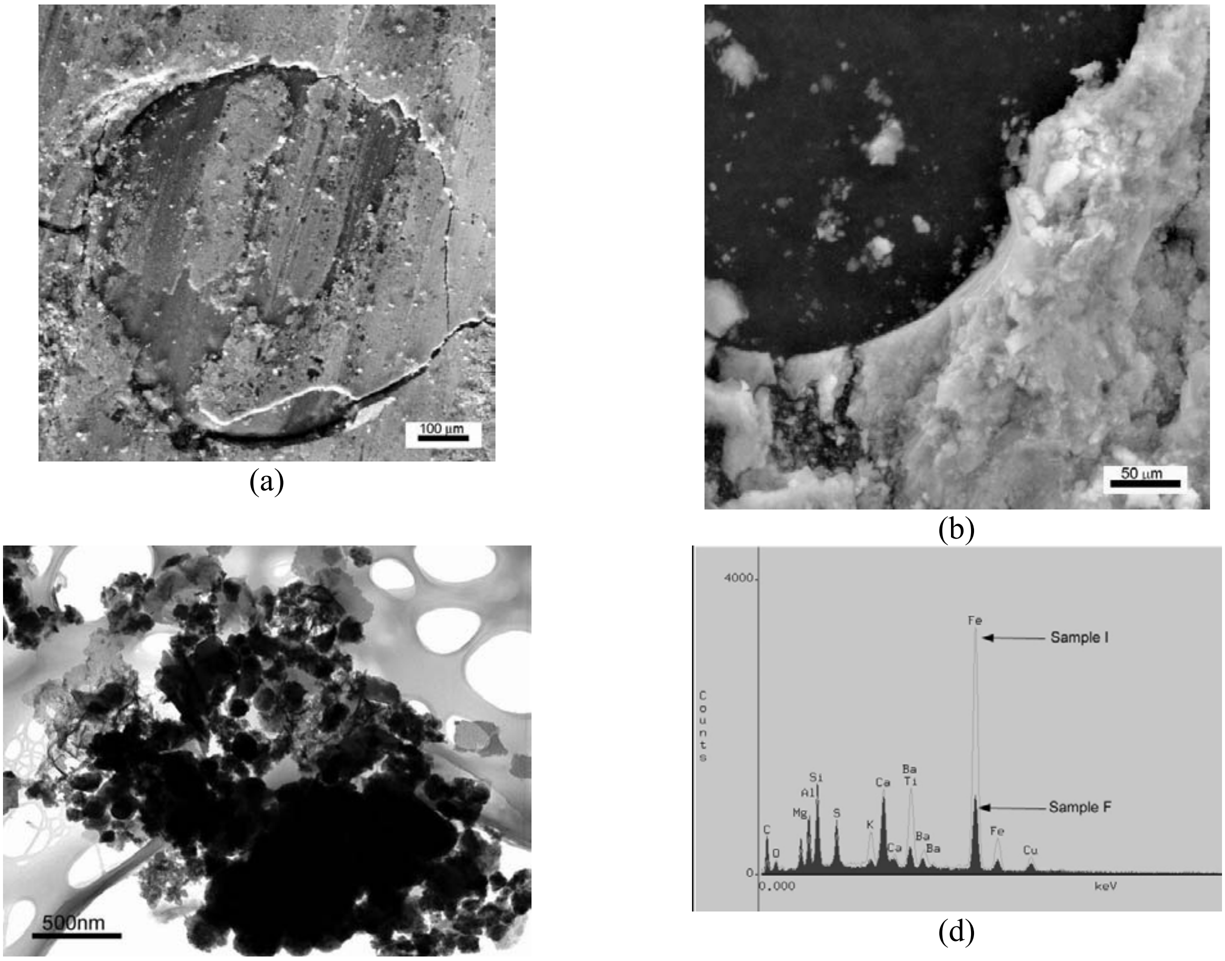
Figure 3a shows burn out resin binder and oxidation at the leading edge of sample F, which influenced the wear rate. When the pads heated up during braking, the resin tended to shrink and crack, and at very high temperatures transformed to carbon. Carbonized resins weaken the matrix and accelerate pad wear [11]. The glassy phases lost their support and were torn off from the surface by the shear force [12]. It is believed that the leading edge had more optimum contact with the disc, thus it wore out faster than the trailing edge. It appears in Figure 3b that the leading edge of sample I has more contact area than the leading edge of sample F. The wear rate was significantly improved by adding potassium titanate to sample I.

*Characteristics of friction surface of A, F and I compositions*: The microstructures of sample F before and after the dynamometer testing are shown in Figure 4. The light microscope image shown in Figure 4a represents the heterogeneous microstructure of sample F. The copper, coke particle, SBR, glassy particle, fly ash, BaSO4, fiber and mating with small particles are marked by number 1, 2, 3, 4, 5, 6, 7 and 8, respectively. As is apparent from Figure 4b, the surface is covered by a friction layer upon test termination. This layer is not continuous and is heterogeneous. Dark (black) areas shown in Figure 4b represent holes formed during the final stage of the dynamometer test. The contact surface is covered by bright and grayish friction layer. The friction layer carries and transfers the load to protect the underlying material spalling off and keeping the wear rate low [13].



(a) (b)

Figure 4. Surface of sample F (a) before and (b) after the dynamometer test



(a) (b) (c) (d)

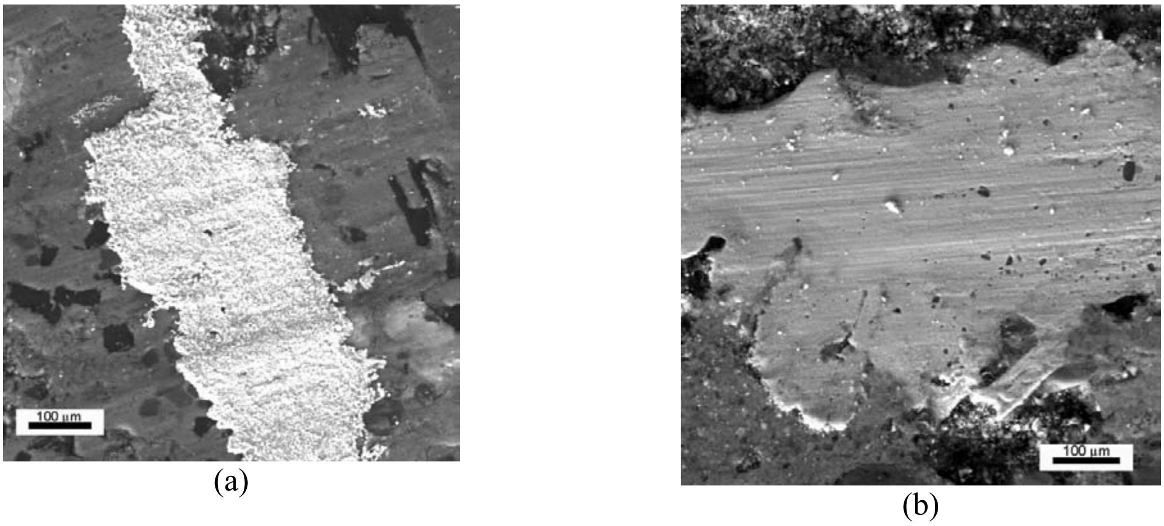
Fig. 5. Characteristic friction layer formed on the friction surface of sample I after testing. (a) SEM micrograph of a glassy phase, (b) Wear debris from the friction surface, (c) TEM micrograph of wear debris, (d) Typical EDX spectrum of sample I overlapping sample F.

The friction layer adhered to the contact area of the bulk material. When the friction material and cast iron disc rubbed against each other without a lubricant, wear particles formed protective layers on the surface [24]. Figure 5a shows a thick friction layer adhering onto the spherical glassy particle. The layer covering the friction surface diminishes the abrasive effect of the glassy phase. The friction layer eliminated the sharp edge and smoothed the friction surface. Glassy particles act as an abrasive element, and apparently scratch off material adhering to the cast iron disc counter face. Wear debris surrounding the spherical glass particle, removed using adhesive tape, is shown in Figure 5b. SEM analysis demonstrated that friction debris often collects around in relief glassy particles. The TEM bright field image (Figure 5c) shows the thin discontinuous friction layer consisting of particles with various shapes (round and angular), transparent and non-transparent attached to each other.

A typical EDX spectrum is shown in Figure 5d. A similar chemistry was observed in the friction layer covering the glassy phase, wear debris removed using adhesive tape and fine particles detected under the TEM microscope. The EDX spectrum revealed that it was dominated by the presence of iron and other elements such as magnesium, aluminum, silicon, sulfur, calcium, potassium, titanium and copper. Sample F contained very similar chemical elements in the friction layer as sample I but lacked potassium and titanium. This indicates that the 10-wt% of potassium titanate in sample I took part in forming the friction layer and improved the overall performance.

EDX spectrum in Figure 5d demonstrated that sample I contained higher amounts of iron and other elements than sample F on the friction layer. Oxygen and carbon content were also high in the friction layer, indicating the presence of oxide particles and degraded lubricant within the layer.

*Fade characteristics of A, F and I compositions*: After each test was terminated after fade procedure, the friction surface of samples F and I were subjected to SEM and EDX analyses. An SEM micrograph of sample F is shown in Figure 6a. This micrograph shows a friction layer formed with fine white particles covering the iron chip. EDX analyses showed the presence of antimony on the iron chip. Antimony trisulphide present in the matrix must have covered the iron chip in the friction layer and lubricated the brake lining. No antimony was present in the friction layer after a complete dynamometer test. It is believed that antimony forms primarily at high pressure and temperature. Obviously, the friction layer containing antimony is being removed by sliding at lower temperatures, and the newly formed layer does not have identical chemistry and structure [2]. The formation of the new friction layer without antimony results in the restoration of higher friction coefficient. The SEM micrograph in Figure 6b shows the friction layer smeared on the friction surface of sample I. The EDX results of sample I revealed similar chemical elements in the friction layer. In the presence of potassium titanate, antimony was not found in the friction layer and the fade phenomenon was prevented from occurring. This indicates that chemical changes on the brake surface are responsible for overall performance changes.

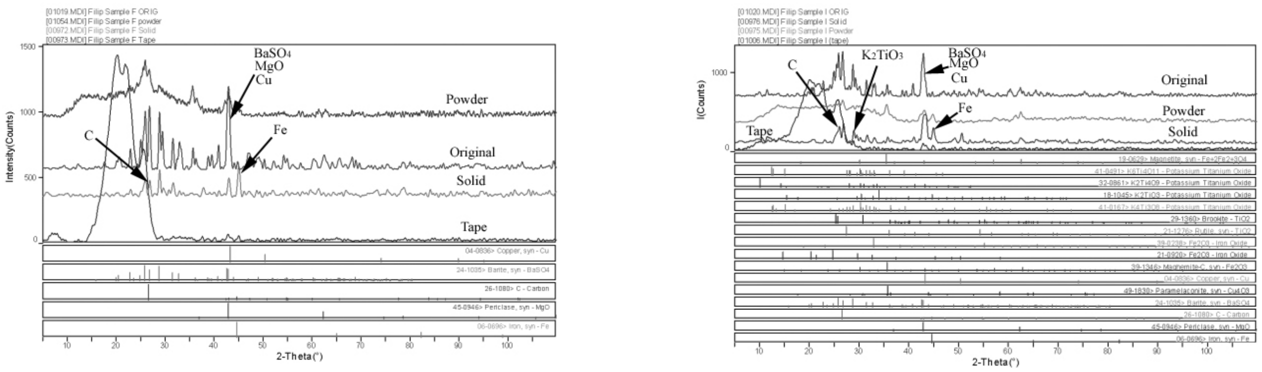


(a)

(b)

Figure 6. SEM micrograph of friction layer formed on the friction surface after fade testing of samples (a) F and (b) I.

The X-ray diffractograms of samples F and I are shown in Figure 7. The original sample before friction test is marked as ``original", collected friction dust is marked as ``powder", friction layer removed by an adhesive tape is marked as `Tape" and solid sample after friction test is marked as ``solid". It is clear that the composition of the friction layer significantly differs from the bulk material. X-ray diffraction revealed relatively high amounts of metallic Fe on the friction surface of samples F and I after the friction test (Figure 7a and 7b). This clearly proves that Fe chips present in the brake lining were easily deformed (smeared over the surface) and adhered to the friction surface. Iron in the friction layer was also transferred from the cast iron disc. It is impossible to distinguish between Fe transferred from the disc and the pad, respectively. Copper was transferred to the friction layer from the bulk of brake linings. Both, iron and copper were oxidized and formed iron oxide and copper oxide in the friction layer. When one component is present in the friction layer in different relative amounts than the disc or bulk material, it indicates that a chemical reaction occurred on the friction surface. A few types of potassium titanium oxide were detected on the friction surface of sample I (Figure 7b).



(a)

(b)

Figure 7. X-ray diffractogram of investigated samples after friction test, (a) sample F and (b) sample I.

The thermal conductivity of samples A, F and I were calculated using the Equation 1 below,

$$\kappa = \alpha \rho Cp \quad (1)$$

where $\alpha$ is the thermal diffusivity, $\kappa$ is the thermal conductivity, $\rho$ is the bulk density and Cp is the specific heat. Figure 8 shows the calculated thermal conductivities of samples A, F and I. The thermal conductivities of sample A decrease with increasing temperature. Sample I exhibited a high peak at the curing temperature between 100oC and 150oC. It possibly indicates that the sample may not have been fully cured. When the temperature reached 200oC, sample I had the same thermal conductivity as sample F. It indicates that potassium titanate did not change the thermal properties of sample I. Nevertheless, the friction layer that contains potassium titanate does improve the friction stability and fade resistance.

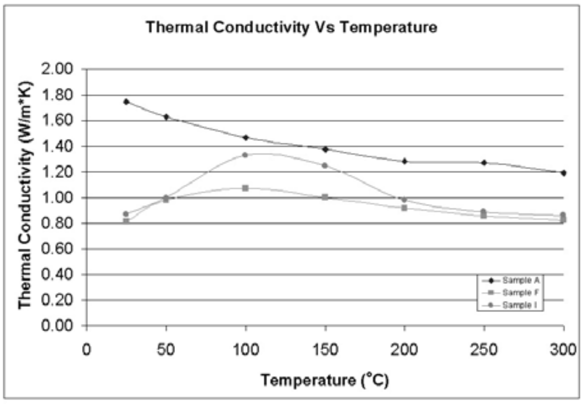


Figure 8. Thermal conductivity of samples A, F and I as a function of temperature

### 3.2 Friction performance of high volume fly ash compositions

FAST Testing Results: High volume FBC fly ash compositions are given in Table 4. The FAST results for FA(65)-RE(25)-GL(10) and FA(55)-RE(30)-GL(10) compositions are indicated in Figure 9. The results indicate fade in the coefficient of friction values after about 20 minutes, and initial high values are also variable. The initial variable peaks point towards the grinding of the asperities and the low friction coefficient pointed towards a general lack of reinforcement. Out of these two compositions, the FA(55)-RE(30)-GL(15) composition provided a superior performance and was selected as the composition for further optimization.

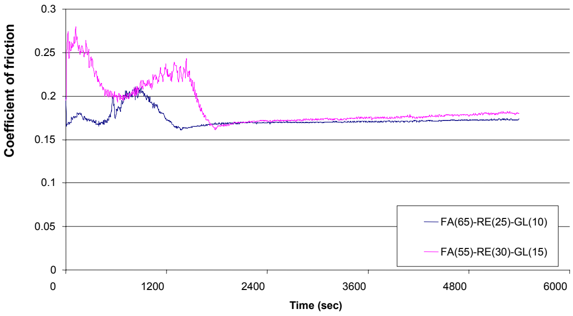


Figure 9. FAST results for high volume fly ash compositions

To overcome the reinforcement problem, aluminum chips were added as another key component. Aluminum chips were chosen due to their availability as an industrial waste, and comparable specific gravity, and specific heat capacity as the fly ash. This ensured a more homogeneous mix in terms of density and thermal properties. The oversized aluminum chips were removed and only -1 mm fraction was used for subsequent mix compositions. The aluminum chips were added in 2%, 5% and 10% by wt fractions of the whole composition. The FAST results for the above compositions are indicated in Figure 10. The 2% addition of aluminum chips did not significantly improve the friction behavior. There are initial variable peaks followed by drop in the friction coefficient. However, the 5% aluminum composition exhibited a consistent behavior, with the coefficient friction of about 0.22. The 10% aluminum composition exhibits a higher coefficient of friction (0.35 – 0.425) for about 60 minutes of the test duration before steeply dropping down to around 0.2.

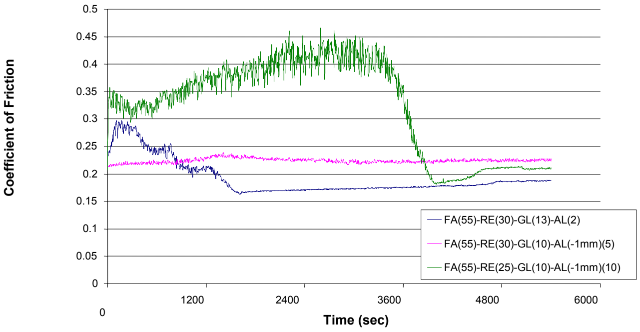


Figure 10. FAST results for compositions with varying amount of aluminum chips (-1mmx 0 size)

Based on the friction surface analysis of the previous compositions by SEM, the next logical step in the mix development was to reduce the size of the aluminum chips to limit the formation of glassy silicate on the chips and reduce the abrasiveness of the compositions. The 10% composition was selected due to the high initial coefficient exhibited in earlier studies. The aluminum chips used were -0.5 mm x 0 size and samples were subjected to FAST testing. The results obtained are presented in Figure 11. The FA(55)-RE(25)-GL(10)-AL(-0.5mm)(10) composition offered the best results so far with two of the three samples exhibiting friction coefficients in the range 0.35-0.4. The third sample failed halfway during the test due to chipping, which was attributed to improper fabrication.

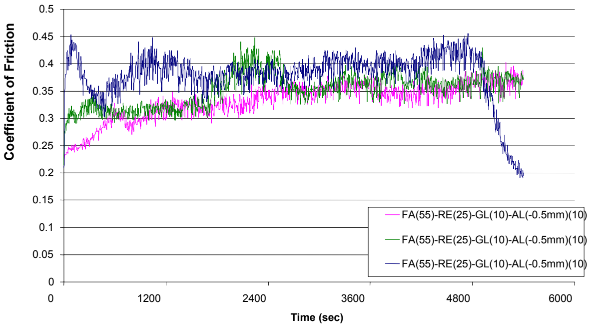


Figure 11. FAST results for compositions with varying amount of aluminum chips (-0.5mmx 0 size)

Further reduction of aluminum chips was done to see the effect on the coefficient of friction and -0.25mm x 0 size aluminum chips were used. The FAST results are presented in Figure 12. Though there was no discernible rise in the coefficient, the behavior was more consistent than earlier samples. Potassium titanate was used in the compositions (5% by weight) to improve the behavior, without any success.

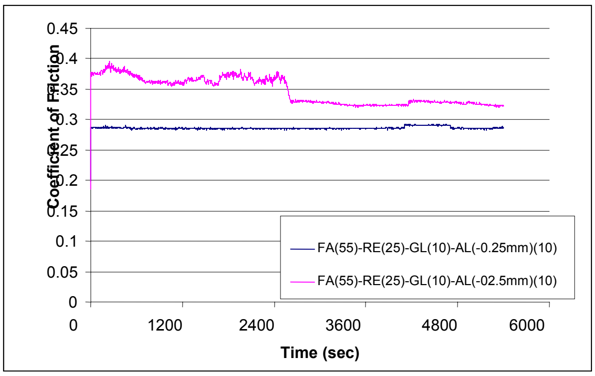


Figure 12. FAST results for compositions with varying amount of aluminum chips (-0.25mmx 0 size)

In addition to the compositions tested above, a high LOI (loss on ignition) and high iron F-fly ash was also used to make FAST samples and was designated as FA2 (fly ash 2). The mix compositions used are given in Table 2.Initial samples made with this ash and 5% and 10% aluminum chips (0.25 mm x 0) failed the FAST test with initial peaks followed by significant drops in the friction coefficient. However, the FA2 compositions without any aluminum exhibited satisfactory friction performance. The FAST results of two such compositions are presented in Figure 13. The two compositions FA2(55)-RE(30)-GL(15) and FA2(65)-RE(25)-GL(10) exhibited a consistent friction coefficient over the entire test duration with the former composition giving a superior performance.

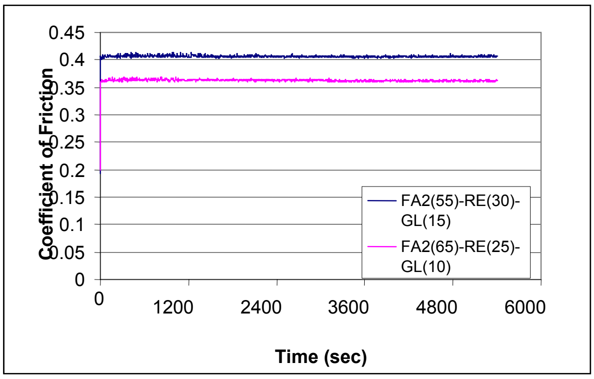


Figure 13. FAST results for compositions with FA2

*Dynamometer Testing Results*: The results offered by this composition prompted the usage of full-scale dynamometer to characterize the friction behavior. J2430 test procedure was used for this purpose and the results are discussed in the following section.

The J2430 dynamometer test procedure uses different cycles to assess the friction behavior of a brake pad. The effectiveness of the material is assessed in two distinct cycles named effectiveness and final effectiveness. The first effectiveness cycle (Figure 14) is preceded by instruments check up and burnish cycles. The coefficient of friction during the first effectiveness cycle dropped as the speed was increased to 100kph. The only encouraging fact observed was that the temperature did not rise very high as compared to commercial friction materials.

The final effectiveness cycle is preceded by five different cycles of hot performance, cooling performance, fade and reburnish. During the final effectiveness cycle too, the coefficient of friction was fairly consistent except for the 100-kmph range. Again the temperatures recorded were low for the friction material. During the dynamometer testing, heavy overtorques were witnessed during some of the cycles, which pointed towards adhesive friction properties.

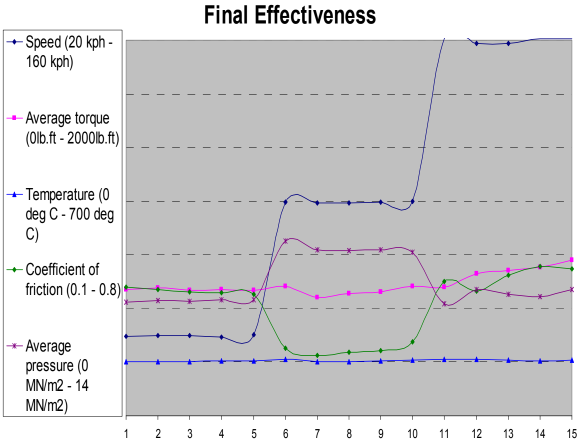


Figure 14. Dynamometer test results for composition FA(55)-RE(25)-GL(10)-AL(-0.5mm)(10)

### 3.3 Characteristics of friction surface of two high volume fly ash compositions

The 5% and 10% Al-chip composition samples were studied with SEM after they had undergone the FAST test. The results are presented in Figure 15. In the SEM analyses, friction layers formed on the surface of the FAST samples were studied for their composition to determine the causes behind the failure of the material from a friction point of view. The friction layer in the 10% aluminum chips composition has presence of higher aluminum in comparison to 5% aluminum composition. The presence of high silicon in both the compositions pointed towards the formation of a glassy silicate on the surface of aluminum chips, which is believed to have contributed towards the lowering of the coefficient of friction. Also, the presence of high iron in both the friction layers pointed towards the extra abrasiveness of the friction material, since the iron could have come only from the rotor.

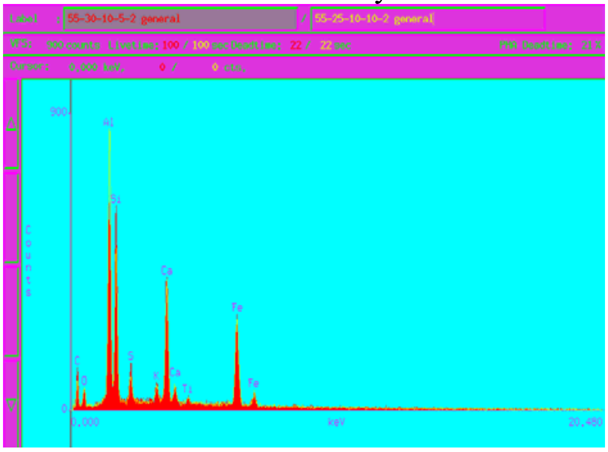


Figure 15. SEM analyses of friction layer

## 4 CONCLUSIONS AND FUTURE DIRECTIONS

Based on research completed to date, the following conclusions can be made.

1. It is possible to utilize over 45% of fly ash in the formulation of brake pad compositions. It may be possible to utilize an additional 10% of magnetite waste in automotive brake pads.
2. The authors' hypothesis that several of the problems associated with current frictional materials used for automotive brake pads can be overcome by utilizing ingredients with smaller diversity in their engineering and physical properties appears valid.
3. The number of ingredients used in automotive brake pads may possibly be reduced by utilizing selected industrial wastes. The production cost of such pads should also be lower than currently available commercial products.

4. The collaborative effort involving university professionals and industrial partners in this product development effort has been the key to successful developments to date.
5. Future planned efforts include optimization of brake pad compositions, developing commercially viable pads, and their laboratory and field testing by industrial partners.

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