TECHNICAL REPORT
March 1 through May 31, 1995

Project Title: **DESIGN AND FABRICATION OF ADVANCED MATERIALS FROM ILLINOIS COAL WASTES**

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ABSTRACT

The main goal of this project is to develop a bench-scale procedure to design and fabricate advanced brake and structural composite materials from Illinois coal combustion residues. Scanning electron microscopy (SEM), differential scanning calorimetry (DSC), differential thermal analysis (DTA), and transmission-Fourier transform infrared (FTIR) were conducted on PCC fly ash (Baldwin), FBC fly ash (ADM, unit1-6), FBC fly ash (S.I. coal), FBC spent bed ash (ADM, unit1-6), bottom ash, and scrubber sludge (CWLP) residues to characterize their geometrical shapes, mineral phases, and thermal stability. Our spectroscopic results indicate that the scrubber sludge is mainly composed of a gypsum-like phase whose lattice structure is different from the lattice structure of conventional gypsum, and sludge does not contain hannebachite ($CaSO_3.0.5H_2O$) phase. In the second and third quarters the focus of research has been on developing protocols for the formation of advanced brake composites and structural composites. Our attempts to fabricate brake frictional shoes, in the form of 1.25 inch disks, from PCC fly ash, FBC spent bed ash, scrubber sludge, coal char, iron particles, and coal tar were successful. Based on the experience gained and microscopic analyses, we have now upscaled our procedures to fabricate 2.5 inch diameter disks from coal combustion residues. The SEM and Young's modulus analyses of brake composites fabricated at 400 psi < Pressure < 2200 psi suggest pressure has a strong influence on the particle packing and the filling of interstices in our composites. Our data suggest that FBC spent bed particles by themselves are not a suitable raw material for our brake shoe pads. However, this is not the case for PCC fly ash particles or for scrubber sludge particles. On sintering the brake composite, the scrubber sludge derived composite's Young's modulus decreased, while the opposite trend was seen for the PCC derived composite. The E values of our coal combustion residue derived brake shoe pads are comparable to those reported in the literature for automotive brakes formed from conventional raw materials.

EXECUTIVE SUMMARY

This project seeks to make use of an advanced, integrated approach for the design and fabrication of brake and structural composite materials from coal combustion residues and coal gasification by-products. The composite materials, which will use significant amounts of combustion residues (20 to 50 wt %) and coal chars (10 to 30 wt %), not only will alleviate the disposal costs and the potential environmental damage costs associated with residues but will also convert residues into high value structural composite materials.

In the first three quarters of this project, we have probed the structural and thermal behaviors of coal combustion residues and scrubber sludge which form the raw materials for our brake shoe pads and structural composites. In addition, we have explored and established protocols for the fabrication of brake composites. This was achieved by fabricating 2.5 inch (diameter) X 1 inch (thick) disks from PCC fly ash, scrubber sludge, and FBC spent bed ash. To characterize PCC fly ash, FBC fly ash (ADM, unit1-6), FBC spent bed ash (ADM, unit1-6), FBC fly ash (S.I. Coal), bottom ash, and scrubber sludge (CWLP), we undertook differential scanning calorimetry (DSC) measurements at $30°C < T < 600°C$ and differential thermal analysis (DTA) at $50°C < T < 1100°C$. Moreover, we subjected the residues to SEM and transmission-FTIR analyses. Based on these characterization data the following was concluded: (a) The PCC fly ash particles are spherical in shape and range from 0.2 μm to 15 μm. Because of their shape and since the particles are thermally inert up to 1100°C, the PCC fly ash is an excellent raw material for our brake and structural composite materials. The PCC fly ash is largely composed of oxides including hematite and magnetite. The presence of these two microwave lossy components makes sintering of materials containing PCC fly ash via microwave heating possible. (b) The scrubber sludge particles, in general, have a shape which is whisker-like, thus imparting it with fiber-like characteristics. Our FTIR analysis of the scrubber sludge sample revealed that CWLP sludge does not contain calcite, hannebachite ($CaSO_3.0.5H_2O$), or bassanite. Our analysis also showed that sludge is mainly composed of gypsum ($CaSO_4.2H_2O$). However, it must be cautioned that the sludge's gypsum lattice structure is different from the conventional gypsum lattice structure. Therefore, the scrubber sludge particles may not display the physical and chemical properties of conventional gypsum. Also, our DSC and DTA data suggested that before scrubber sludge particles can be used they must be heated to 200°C to evolve out the water associated with sludge. At $200°C < T < 1000°C$, our DSC and DTA data indicated the dehydrated gypsum is thermally stable, thus. very suitable for our composites. Therefore, the presence of scrubber sludge particles in our composites will provide mechanical strength to our materials. (c) The FBC fly ash and FBC spent bed bottom ash particles showed a considerable amount of fusion resulting in larger sized particles, i.e., 100 μm to 750 μm. The transmission-FTIR data on the two FBC fly ashes examined lead us to believe that the main constituents of FBC fly ash are anhydrite, lime, portlandite, calcite, hematite, magnetite, and various glass phases. The FBC spent bed ash is largely composed of anhydrite, lime, portlandite, calcite, periclase, hematite, and magnetite. The thermal stability characteristics of FBC fly ash and FBC spent bed ash, as determined by

DSC and DTA techniques, are such that they make these particles suitable raw materials for brake composite materials and structural composites. (d) The bottom ash particles contained a considerable amount of carbon and have a highly porous but glassy structure, thus, making the bottom ash particles an ideal filling material for our brake lining material and structural composites. (e) After our initial attempts to form structural composites, in the form of 1.25 inch disks, from PCC fly ash, FBC spent bed ash, scrubber sludge, coal char, and coal tar were successful, we upscaled the size of our structural composites to 2.5 inch diameter disk size. Typical disks are depicted in Figure 1.

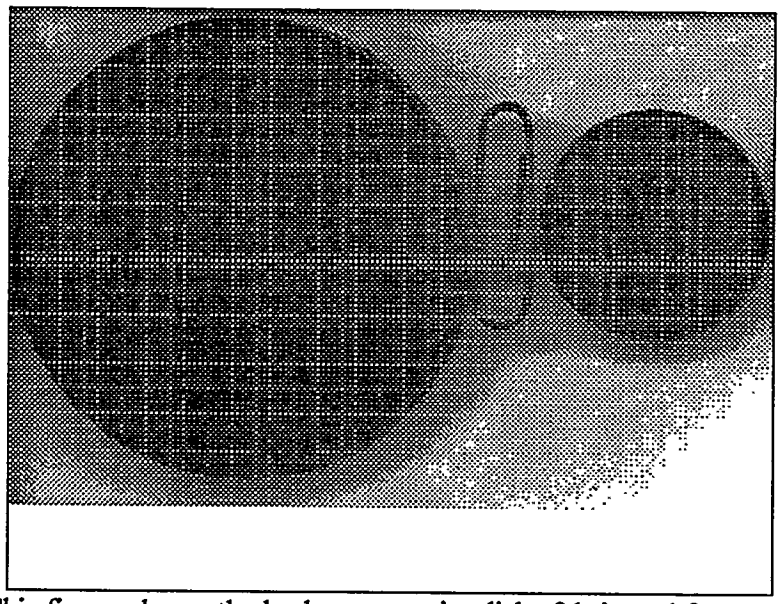

Figure 1. This figure shows the brake composite disks fabricated from PCC fly ash, FBC spent bed ash, scrubber sludge, and coal char.

Our microscopic results on the fabricated disks indicated that pressure had a strong influence on the pore structure of the fabricated material and pressures of 2100 psi or above should be used to control the porosity of the resultant composite. (f) The SEM and Young's modulus analyses on the brake composites fabricated from PCC fly ash, FBC spent bed ash, scrubber sludge, coal char, and iron particles as a function of pressure showed that higher pressures lead to better particle packing and better mechanical performance. (g) When only FBC spent bed ash particles are used along with coal char to form brake composite materials, the hot pressed composite collapses. However, this is not the case for brake shoe disks formed from either scrubber sludge particles or PCC fly ash particles. Therefore, it is concluded that FBC spent bed ash by itself is not a good filler material for the brake shoe pad. On the other hand, when the irregularly shaped particles of FBC spent bed ash are combined with scrubber sludge particles or PCC fly ash particles, the resultant material is expected to have better tribological behavior. (h) The hot pressed composite has considerable porosity which heals on subjecting the hot pressed composite to pyrolysis at 400°C. (i) In contrast to the carbon fibers when we used mineral fibers to form our brake shoe pads, excellent bonding occurred between the mineral fibers and resin matrix. This could be visualized by comparing Figures 2 and 3.

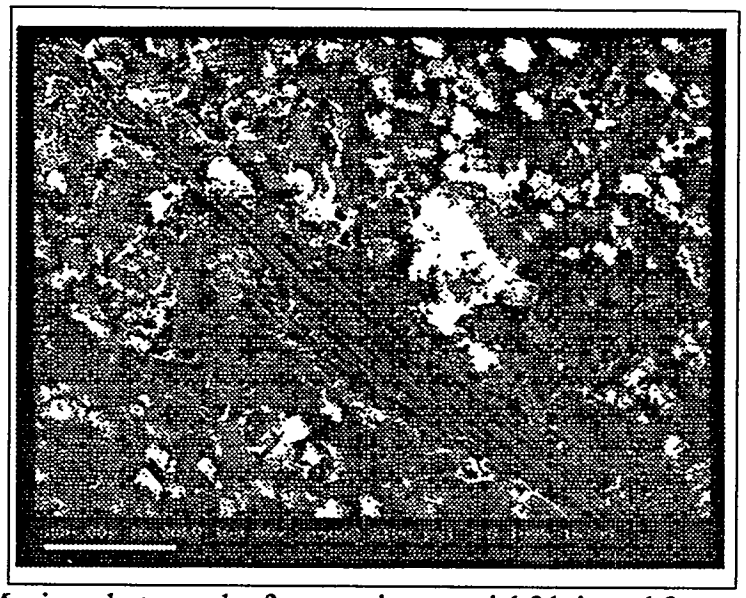

Figure 2. SEM microphotograph of composite material fabricated from scrubber sludge, FBC spent bed ash, coal char, and carbon fibers. Note the lack of bonding between the carbon fibers and matrix.

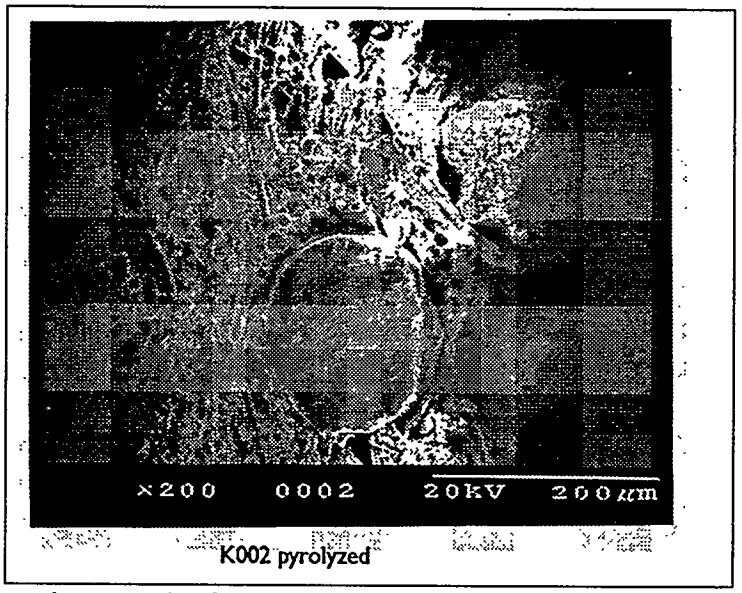

Figure 3. SEM microphotograph of composite material fabricated from scrubber sludge, FBC spent bed ash, coal char, and mineral fibers. Note the excellent bonding between the mineral fibers and matrix.

(j) On subjecting the hot pressed composites fabricated from scrubber sludge, coal char, and FBC spent bed ash particles to pyrolysis, the Young's modulus (E) value decreased. A typical result obtained from three point bend test is reproduced in Figure 4 showing the elastic behavior followed by the breakage of the composite material. The decrease in E value implies that the material becomes more brittle relative to the hot pressed sample. While this is also the case for composites fabricated from scrubber sludge, the behavior of PCC fly ash fabricated composites is exactly opposite where E value increased on

sintering. It should be pointed out here that the E values of our coal combustion residue derived brake shoe pads are comparable to those reported in the literature for automotive brakes formed from conventional raw materials. (k) Both DSC and FTIR results indicate that the resin material in the composite degrades on sintering the material at 400°C. Therefore, the brake shoe pad's mechanical strength should be enhanced by limiting the sintering temperature to 200°C < T < 400°C. This is reasonable since automotive brakes are exposed to temperatures up to 300°C.

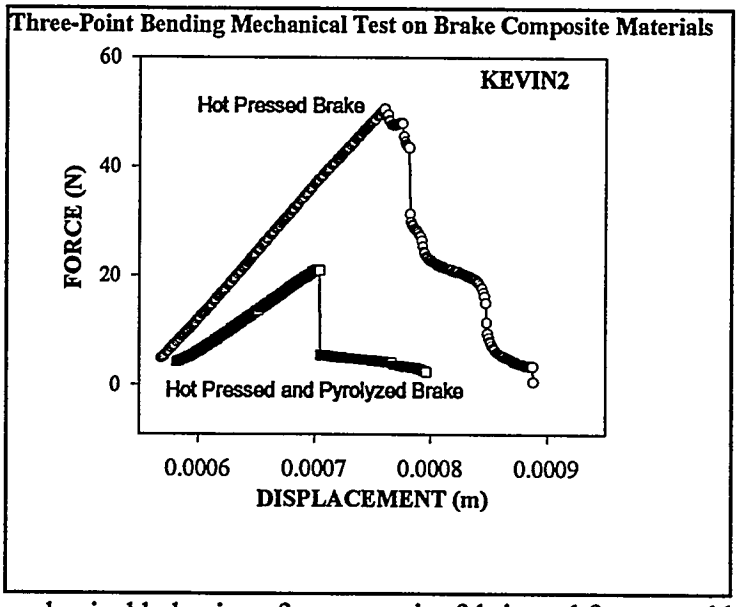

Figure 4. The mechanical behavior of a composite fabricated from scrubber sludge, FBC spent bed ash, and coal char.

OBJECTIVES

The main goal of this project is to develop a bench-scale procedure to design and fabricate advanced brake and structural composite materials from Illinois coal waste residues by utilizing the effective capability of fly ash and FBC residues to modify coal tar's thermosetting properties. The specific objectives of our project are: (1) to characterize both structural and thermal properties of various combustion residues and gasification residues from power plants using Illinois coals, (2) to evaluate how the glass transition temperature and the subsequent thermosetting behavior of processed coal tar are altered by additives such as fly ash residue and FBC residue, (3) to optimize the thermosetting properties of processed coal tars by altering the concentration and types of residue additives used such that the desired end product, i.e., advanced brake and structural composite materials, is fabricated, (4) to identify and to optimize the engineering steps required to fabricate the high value, advanced brake composite materials, (5) to ascertain and to optimize the engineering conditions under which high value, advanced structural composite materials for the automotive industry could be fabricated from coal combustion residues, (6) to characterize the fabricated advanced brake and structural composite materials for their suitability and performance as brake and structural materials, and (7) to undertake a systematic, economic analysis of our process and to present comparisons with competitive products. In the first year of the project the thrust of the work is to be directed towards the first four listed objectives.

INTRODUCTION AND BACKGROUND

Massive quantities of inorganic residues are generated when coal is gasified or burned, and when flue gases are scrubbed of sulfur dioxide. According to Golden of EPRI, the electric utilities in the USA produce about 90 million tons of coal combustion ash residues annually. It is believed that a typical 800 Megawatt power plant consumes about 7500 tons of coal and generates 1000 tons of fly and bottom ash per day. The Federal Clean Air Act Amendments of 1990 mandate that sulfur emissions by coal-fired industry be significantly reduced by the year 2000. Illinois coals contain high sulfur. The sulfur is in the form of inorganic minerals (chiefly pyrite) and is also organically bound. Therefore, continuous utilization of midwest coal will require scrubbing which will further enlarge combustion residue production. It is estimated that by the turn of century about 200 million tons of coal combustion residue will be produced annually. With the current cost of residue disposal expected to rapidly escalate from $10/ton to $30/ton by the year 2000, the economic stakes for the coal utilization industry are substantial. Consequently, the technologies which can convert combustion residues into high value, but economically sound, materials are of utmost importance. Our proposed research of converting combustion residues into advanced brake and structural materials precisely does that.

EXPERIMENTAL TECHNIQUES

Differential Scanning Calorimetry (DSC) Measurements: The thermal behavior of fabricated composites was recorded on a Perkin-Elmer DSC7 system, interfaced with a PC 486. The DSC was calibrated for temperature and enthalpy. The temperature calibration was performed by the two-point method, using the melting transitions of indium (430 K or 157°C) and zinc (693 K or 420°C). The accuracy in temperature between 300 K and 693 K , based on our calibration procedure, was estimated to be ±1 K. The enthalpy calibration was performed using indium heat of fusion as the standard. After the enthalpy calibration, the DSC data on zinc metal were re-recorded, and the observed enthalpy of the melting transition of zinc was consistent with the values reported in the literature. The conditions under which the instrument calibration was performed exactly matched the experimental run conditions, namely the scan rate of 20 K/min (20°C/min), nitrogen gas purge at 0.207 MPa (30 psi) pressure. Also, during both calibration and heating runs, the dry box assembly over the sample head was flushed with nitrogen gas to maintain thermodynamic equilibrium. Aluminum sample pans, in an unsealed mode, generally were used to probe the thermal behavior of brake composite materials. This was achieved by pushing down the top sample pan cover gently onto the bottom pan containing the sample. Typically 20 mg of sample were used for our DSC measurements.

Transmission-Fourier Transform Infrared (Transmission-FTIR) Measurements : This quarter we continued our efforts to characterize the composite materials fabricated from coal combustion residues (CCR) and flue gas desulfurization (FGD) residue obtained from the Mining Engineering (Southern Illinois University at Carbondale) Department's Combustion Residue Bank. The Transmission-FTIR data were collected on the composites by first crushing them to a fine powder. The KBr pellet technique was adopted to record the transmission-FTIR spectra of the composite materials. The spectra were obtained on a Nicolet IR-44 FTIR spectrometer equipped with a DTGS detector and interfaced with a PC computer. Typically, 100 single beam scans (interferograms) of the sample were collected. The reference interferograms were collected under identical conditions but without a sample in the beam. During this process, the spectrometer was continuously purged with dry nitrogen gas. With the use of the transformed single-beam sample and reference spectra, the spectra of the sample could be plotted as transmittance or absorbance. We used triangular apodization to transform the interferograms acquired at 4 cm-1 resolution. Therefore, the effective resolution of the spectra was approximately 6 cm-1. The KBr pellets were dried for 24 hours in a desiccator before the pellets were subjected to transmission-FTIR measurements.

Fabrication of Brake Composite disks : In the last six months, we have continued with our efforts to form brake composite and structural composite materials from coal combustion residues, scrubber sludge, and coal tar derived from Illinois coals. Generally, the following steps were executed for fabricating the composites:

(1) As a first step, the coal tar obtained from Illinois coals was processed to enhance its ash wetting properties.

(2) A dilute solution of the processed coal tar was formed with tetrahydrofuran. This product henceforth will be called "Tar-THF solution." The PCC fly ash combustion residue particles were combined with Tar-THF solution to form a colloidal structure which ensured that the residue particles were thoroughly coated with tar. The tetrahydrofuran was recovered from the colloidal structure by rotary evaporation, and the tar coated particles were dried in air. The dried particles had the appearance of a fine powder. This powder will be classified as "MAL101" powder.

(3) A similar colloidal structure, as described in step 2, was prepared with scrubber sludge particles. The powder obtained from the sludge particles will henceforth be referred to as "MAL201."

(4) The tar coated particles of the combustion residue were mixed with various ingredients as shown in Table 1 to form brake composite materials. These experiments were conducted to probe what role various combustion residues have in the formation of the composites and if any particular residue performs better than other residues.

(5) The tar coated particles of PCC fly ash, scrubber sludge, FBC spent bed ash, and iron were mixed with various ingredients as shown in Table 2 to fabricate brake composites.

(6) Mixtures listed in Tables 1 and 2 were accurately weighted and were ground in a mortar. The ground samples were thoroughly mixed to ensure the best achievable homogeneous distribution of the particles. A one and one quarter inch diameter pellet die was prepared by applying a thin coating of a standard release agent. The die was cured at 180°C (453 K) for one hour and cooled to room temperature. The samples mixtures were poured into the die and hot pressed at the desired pressure and temperature to form the primary composite skeleton.

(7) A procedure identical to step 6 was used to form 2.5 inch diameter composite disks except the die was of the appropriate size. It is worthwhile to remark here that the hot pressed sample must be cooled to room temperature before the sample is removed from the die. Failure to do so, our observations indicate, will result in the collapse of the composite structure. The hot pressed samples were examined for their thermal stability, structural behavior, and mechanical performance by conducting DSC, FTIR, SEM, and three point bend tests.

(8) The hot pressed composite materials were further processed to enhance their mechanical strength by curing the material at 673 K (400°C). To accomplish this the hot pressed brake skeletons were heated in an electric oven under the flowing nitrogen gas condition. The sample's temperature was raised at a rate of 1°C per minute and was held at 400°C for 30 minutes. After thermal soaking at 400°C, the composites were cooled to room temperature and were subjected to thermal stability, structural, and mechanical tests.

Scanning Electron Microscopic (SEM) Measurements: We collected SEM images on brake composite materials KEVIN1, KEVIN2, KEVIN3, KEVIN4, and KEVIN5. These data were collected with a view to understand how the nature of the combustion residue effects the composite. The SEM images were obtained using a Hitachi S-570 scanning electron microscope. A thin portion of the composite was cut and was glued on a SEM

high resolution stub. The composite samples were then heated at 60° C (333 K) for 24 hours. These steps were necessary to ensure that the composite would not detach from

TABLE 1
Various Mixtures Used To Fabricate Brake Composites at 180°C.

Sample I.D.	Ash Residue (wt%)	Coal Char (wt%)	Tar Coated Iron Parti-cles (wt%)	FRF Mineral Fibers (wt%)	Phenolic Resin (wt%)	Press-ure (psi)
KEVIN3	Scrubber Sludge 32.1	22.2	7.4	7.3	solid: 11.0 liquid:18.5	2,500
KEVIN4	PCC Fly Ash 35.5	24.5	12.3	15.5	solid: 12.3	2500
KEVIN5	FBC Spent Bed Ash 36.7	25.3	8.5	8.5	solid: 12.7 liquid: 8.5	2,500

the stubs when under electron beam and also would not decompose when under electron bombardment. The composite samples were sputter coated with a 400°A thin layer of gold to reduce sample charging. Generally, the SEM micrographs were collected using an accelerating voltage of 20 kV. The working distance used was in the range of 8 mm to 12 mm.

Mechanical Tests (Young's Modulus): The hot pressed and pyrolyzed brake composite materials, i.e., KEVIN2H, KEVIN2P, KEVIN3H, KEVIN3P, KEVIN4H, and KEVIN4P, were subjected to the three point bend test to determine their mechanical performance. It should be noted that we used designation H and P to classify composite samples which were only hot pressed and were hot pressed and pyrolyzed at 400°C, respectively. We used a diamond circular saw to cut rectangular strips of the sample having nominal thickness of about 0.2 inch and width of 0.4 inch from 2.5 inch diameter brake composite material. Nine samples were subjected to three point bend testing to determine the Young's modulus of our brake composite material. The samples were labeled as follows:

1. KEVIN2H1
2. KEVIN2P1
3. KEVIN2P2
4. KEVIN3H1
5. KEVIN3P1
6. KEVIN3P2
7. KEVIN4H2
8. KEVIN4P1
9. KEVIN4P2

The equipment used to measure the Young's modulus was the Instron Universal Testing Instrument (model 4206) located in the Mechanical Engineering Department. This system is a highly reliable precision system for evaluating the mechanical properties of materials. The system is designed to test materials under either tension or compression; and a complete range of extensometers, fixtures, and local cells are available. The equipment is controlled by a microprocessing unit which in turn is controlled by a computer for

actuation and data collection. The span length was fixed at 1.6 inch. The sample was supported by two cylinders below and one cylinder above the center of the specimen.

TABLE 2
Various Mixtures Used To Fabricate Brake Composites at 180°C and 2500 psi.

SAMPLE I.D.	Tar Coated Ash particles (wt%)	Coal Char (wt%)	Iron Particles (wt%)	FBC Spent Bed Ash (wt%)	Phenolic Resin (wt%)	FRF Mineral Fibers (wt%)
KEVIN1	Scrubber Sludge 32.3	29	9.7	9.7	14.5	4.8
KEVIN2	Scrubber Sludge 27.1	24.3	8.1	8.1	12.2	20.3
KEVIN3A	Scrubber Sludge 36.6	25.3	8.4	0	solid:12.6 liquid: 8.4	8.5
KEVIN3B	Scrubber Sludge 40.0	27.7	9.2	0	solid:13.8 liquid: 0	9.2
KEVIN3C	Scrubber Sludge 40.0	27.7	9.2	0	solid: 4.6 liquid: 9.2	9.2

A force was applied directly above the center of the specimen. A deflectometer was mounted directly below the center of the specimen to measure the deflection as the force was applied.

The Young's modulus, E, was determined by plotting the force versus deflection. In the region of the plot before the specimen breaks, the curve was linear and the behavior was elastic. The slope, S, of that linear region was used to determine the Young's modulus of the material, i.e.,

$$E = \frac{SL^3}{4wt^3} \quad , \qquad (1)$$

where L is the span length, w is the width, and t is the thickness of the sample.

RESULTS AND DISCUSSION

Effects of Residues on Brake Composites: This quarter we examined whether types of combustion residue have any effect on the structural and mechanical properties of the fabricated brake composite materials. To ascertain this, three composite materials were designed. In the first brake material, referred to as "KEVIN3" in Table 1, the surface treated scrubber sludge particles were used to fabricate our brake disk. In the second brake material, surface treated PCC fly ash was the main residue in the composite named KEVIN4. The third brake composite contained FBC spent bed ash as the major residue content of the composite classified as KEVIN5. Our efforts to form the composite using FBC spent bed ash as the main and the only residue ingredient in the composite were not successful as hot pressed material disintegrated once the material was removed from the die. However, this was not the case for KEVIN3 and KEVIN4. The hot pressed composites KEVIN3 and KEVIN4 were sintered under nitrogen gas at 400°C to further enhance the mechanical strength of these materials.

TABLE 3
The Young's Modulus of Various Brake Composite Materials

SAMPLE I.D.	COMPOSITE PYROLYZED AT 400°C	THICKNESS (inch)	WIDTH (inch)	YOUNG'S MODULUS, E, (GPa)
KEVIN2H1	No	0.219	0.16	5.6
KEVIN2P1	YES	0.23	0.15	3.3
KEVIN2P2	YES	0.23	0.15	3.1
KEVIN3H1	NO	0.19	0.15	5.3
KEVIN3P1	YES	0.19	0.16	2
KEVIN3P2	YES	0.19	0.14	3.9
KEVIN4H2	NO	0.2	0.17	2.2
KEVIN4P1	YES	0.2	0.16	4.5
KEVIN4P2	YES	0.21	0.19	4.5

Effects of Pyrolysis on the Brake Composites: As pointed out in the experimental section, we collected the SEM microphotographs on KEVIN2H, KEVIN2P, KEVIN3H, and KEVIN3P samples to understand how sintering affects the composite's structure. In Figures 5 and 6 we reproduce the SEM photos of KEVIN2 composite which was hot pressed and subsequently pyrolyzed at 400°C, respectively. It should be noted from these

figures that the hot pressed samples have much higher porosity than the pyrolyzed samples. This observation suggests that on sintering the composites the phenolic resin flows and fills the pores in the material. Figure 7 shows SEM microphotograph of a typical composite which has been hot pressed at 180°C. As can be seen, the mineral fibers are well dispersed in the residue matrix, thus, providing it with mechanical strength. However, the interesting point to note from the SEM photos presented in Figures 2 and 3 is the lack of bonding between carbon fibers and SC1008 resin and the excellent bonding between FRF mineral fibers and the SC1008 resin. The coal char particles, it appears, do not cross-link well with the matrix produced from SC1008 phenolic resin.

Mechanical Behavior: Our results from the three point bend test performed on KEVIN2, KEVIN3, and KEVIN4 composite material are summarized in Table 3 and Figures 4 and 8. The force-displacement behavior of KEVIN2 composite, depicted in Figure 4, shows initially an elastic behavior, i.e., as force increases displacement increases. In fact, there is a linear relationship between force and displacement. As the applied force continues to increase, the extensive deformation of the sample results in the breakage of the sample. It should be observed from Table 3 that the Young's modulus (E) of the composite decreases when the hot pressed disks were subjected to pyrolysis at 400°C. This is the case when composite materials were fabricated from scrubber sludge. However, on subjecting KEVIN4 to pyrolysis the E value of the material increased relative to the hot pressed sample. It seems that the composites formed from the scrubber sludge became brittle on sintering. The E values of our brake composite materials compare favorably with those reported for automotive brakes in the literature.

Thermal Behavior: Figure 9 reproduces the thermal behavior of KEVIN2 composite which was hot pressed (KEVIN2H), and the composite which was hot pressed and pyrolyzed at 400°C (KEVIN2P). The difference DSC curve obtained by subtraction, i.e.,

$$\text{Difference DSC curve} = \text{DSC curve (hot pressed)} - \text{DSC curve (hot pressed and pyrolyzed)} \quad (2)$$

is shown in Figure 9(c). As can be seen from the difference DSC curve, an endothermic peak is observed at 167°C. This implies that the hot pressed sample underwent a thermal event at around 167°C which was endothermic. We assign this endothermic peak to the desorption of water which was produced due to the cross-linking reaction of phenolic resin.. Three additional endothermic peaks were observed at 413°C, 474°C, and 518°C for the hot pressed composite relative to hot pressed and pyrolyzed sample. Since none of the residues used in the fabrication of KEVIN2, i.e., scrubber sludge and FBC spent bed ash, have any decomposition reactions at the aforementioned temperatures, we assign these thermal events to the degradation and decomposition reactions of the phenolic resin.

Structural Behavior: FTIR spectroscopic studies were undertaken to characterize how sintering of the hot pressed brake composite affects the structure of the material. Figure 10 depicts the FTIR spectrum of KEVIN2 composite which was formed by hot pressing at 180°C and the KEVIN2 composite which was hot pressed at 180°C and then pyrolyzed at

400°C. Both spectra are dominated by inorganic infrared bands. Two very strong bands were observed at 1157 and 1121 cm^{-1}. In addition, a doublet having frequencies 677 and 617 cm^{-1} was observed. These bands strongly suggest the presence of anhydrite ($CaSO_4$) in our composite implying that during hot pressing and subsequent pyrolysis of KEVIN2 the scrubber sludge maintains its chemical structural integrity. On subjecting the KEVIN2 hot pressed composite to pyrolysis at 400°C few changes occur in the infrared band frequencies especially those of organic bands. It should be noted that the aliphatic vibrational bands at 2960 - 2850 cm^{-1} lose intensity when the sample was pyrolyzed. In addition, the infrared band at 1509 cm^{-1} disappears on sintering the sample at 400°C. Therefore, it is reasonable to conclude that phenolic resin undergoes degradation reactions at 400°C, thus fixing the pyrolysis temperature to < 400°C.

CONCLUSIONS AND RECOMMENDATIONS

In the first three quarters of this project, we have probed the structural and thermal behaviors of coal combustion residues and scrubber sludge which form the raw materials for our brake shoe pads and structural composites. In addition, we have explored and established protocols for the fabrication of brake composites. This was achieved by fabricating 2.5 inch (diameter) X 1 inch (thick) disks from PCC fly ash, scrubber sludge, and FBC spent bed ash. This quarter we focused on ascertaining: (1) how individual residues affect the structure of the formed material and does any individual residue by itself have advantage over other residues, and (2) how sintering of the brake shoe pad affects its mechanical and structural properties. The following is concluded: (a) When only FBC spent bed ash particles are used along with coal char to form brake composite materials, the hot pressed composite collapses. However, this is not the case for brake shoe disks formed from either scrubber sludge particles or PCC fly ash particles. Therefore, it is concluded that FBC spent bed ash by itself is not a good filler material for the brake shoe pad. On the other hand, when the irregularly shaped particles of FBC spent bed ash are combined with scrubber sludge particles or PCC fly ash particles, the resultant material is expected to have better tribological behavior. (b) The hot pressed composite has considerable porosity which heals on subjecting the hot pressed composite to pyrolysis at 400°C. (c) In contrast to the carbon fibers when we used mineral fibers to form our brake shoe pads, excellent bonding occurred between the mineral fibers and resin matrix. (d) On subjecting the hot pressed composites fabricated from scrubber sludge, coal char, and FBC spent bed ash particles to pyrolysis, the Young's modulus (E) value decreased. This implies that the material becomes more brittle relative to the hot pressed sample. While this is also the case for composites fabricated from scrubber sludge, the behavior of PCC fly ash fabricated composites is exactly opposite where E value increased on sintering. It should be pointed out here that the E values of our coal combustion residue derived brake shoe pads are comparable to those reported in the literature for automotive brakes formed from conventional raw materials. (e) Both DSC and FTIR results indicate that the resin material in the composite degrades on sintering the material at 400°C. Therefore, the brake shoe pad's mechanical strength should be enhanced by

limiting the sintering temperature to 200°C < T < 400°C. This is reasonable since automotive brakes are exposed to temperatures up to 300°C.

DISCLAIMER STATEMENT

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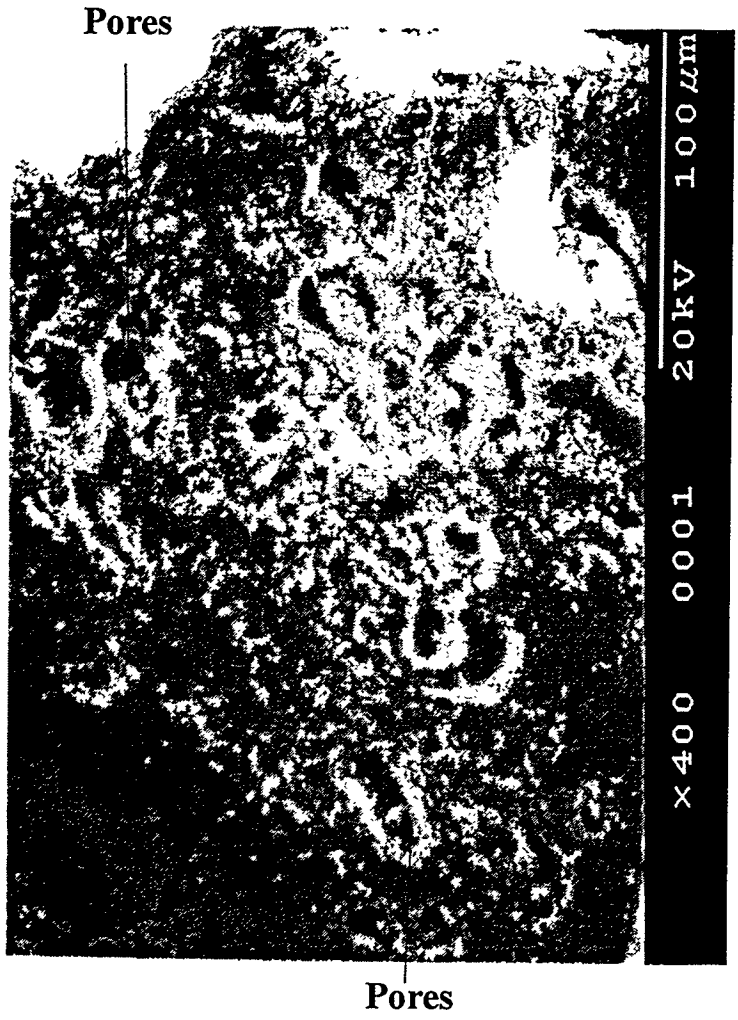
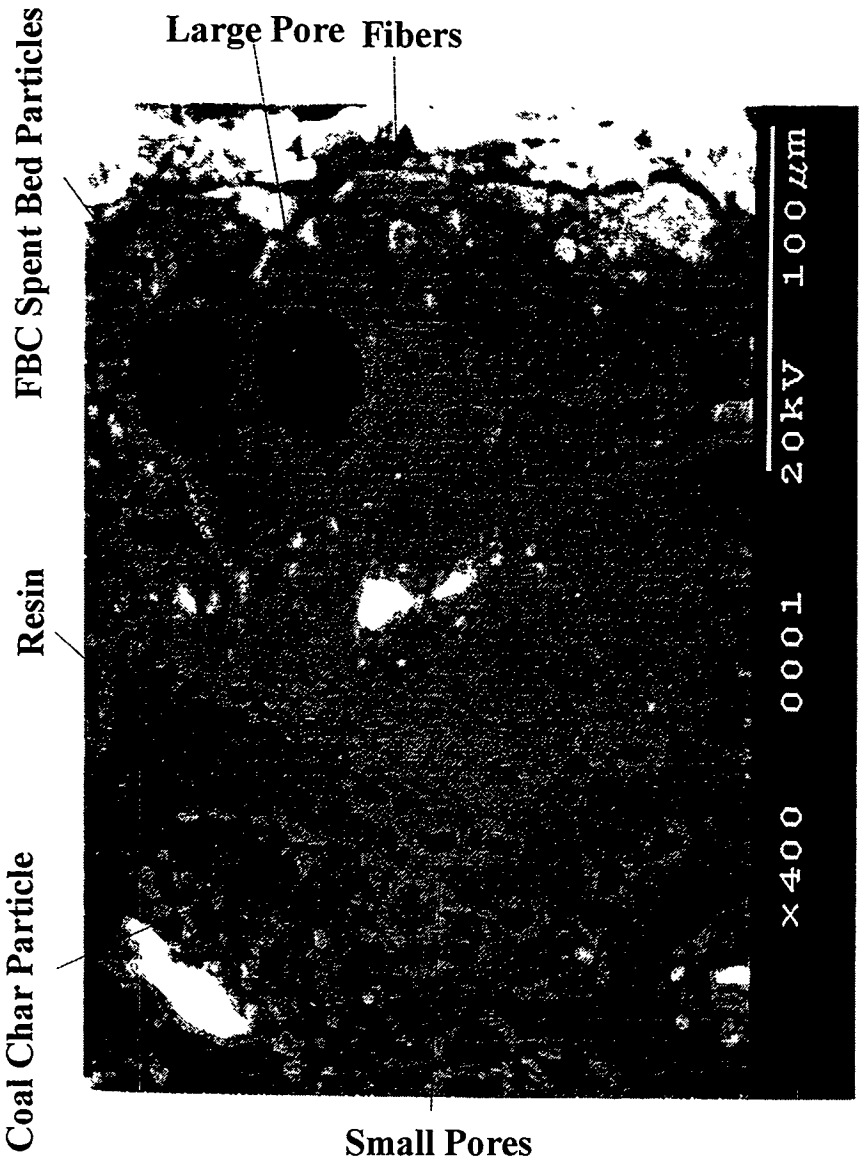
Figure 5.



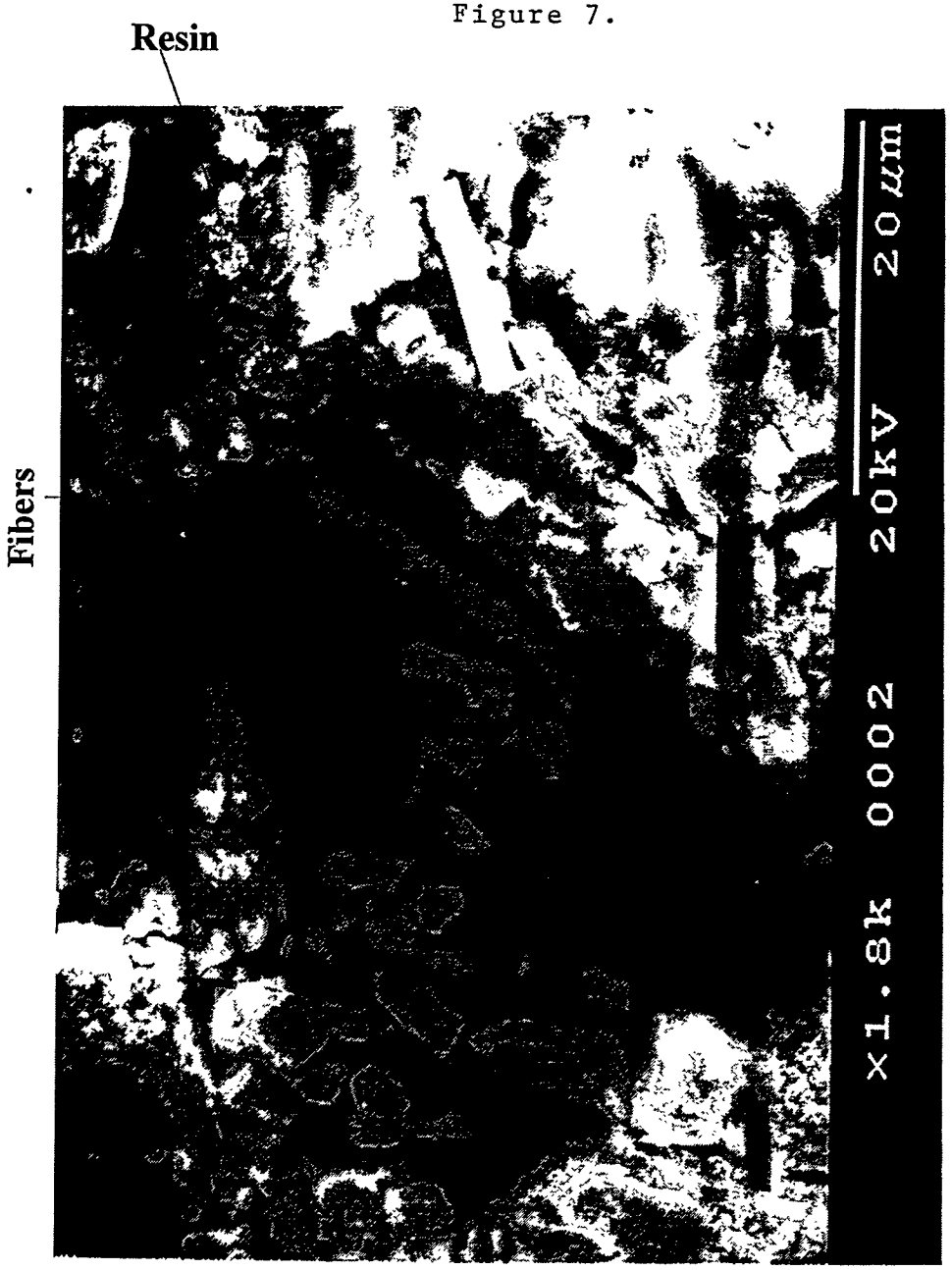
Hot Pressed KEVIN2 Composite. The Composite has not undergone any pyrolysis.

Figure 6.



SEM micrograph showing the KEVIN2 composite which was hot pressed at 180 C and then pyrolyzed at 400 C.

Figure 7.



Hot Pressed KEVIN2 Composite. The Composite has not undergone any pyrolysis.

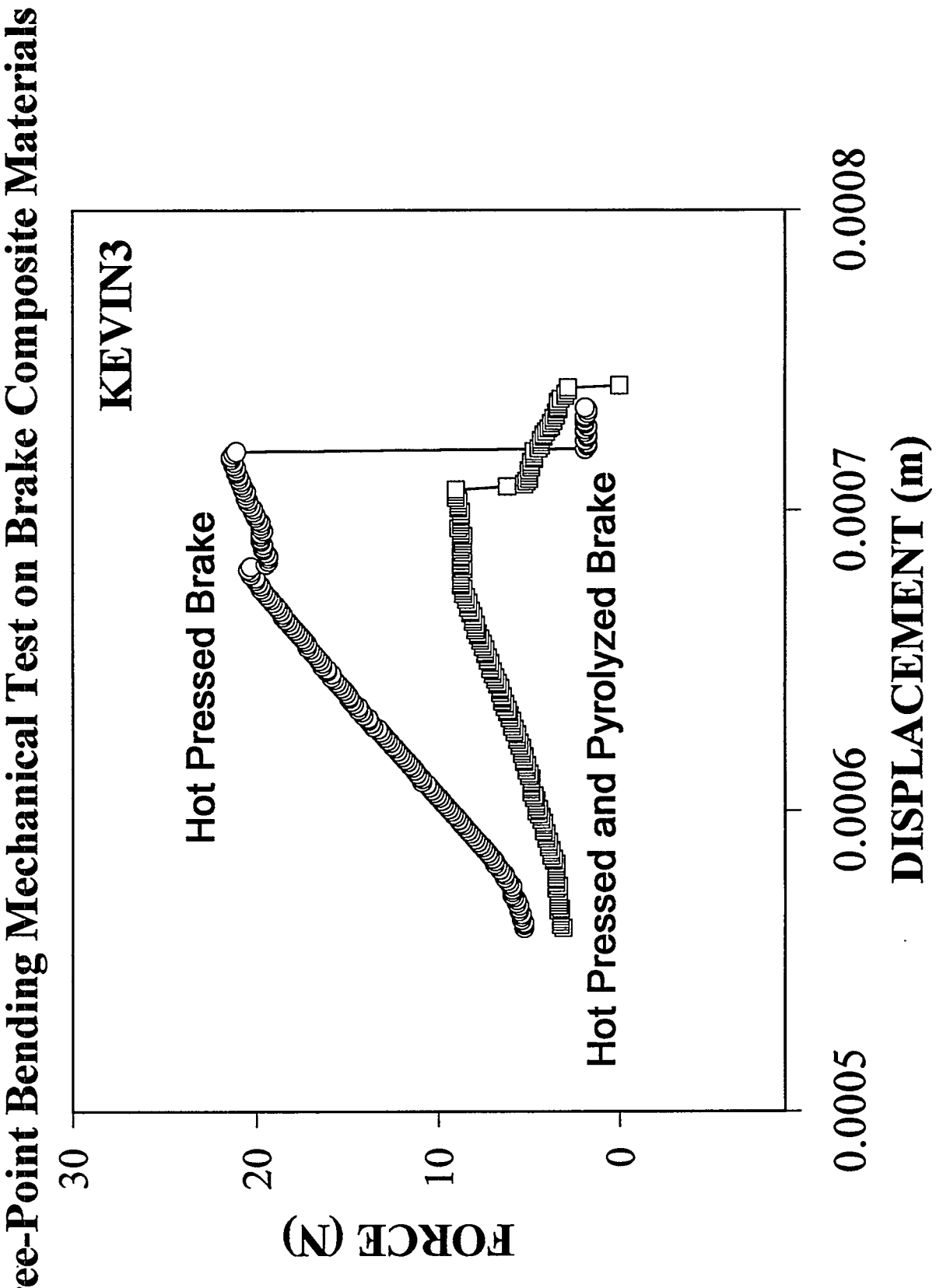

Three-Point Bending Mechanical Test on Brake Composite Materials
KEVIN3
Hot Pressed Brake
Hot Pressed and Pyrolyzed Brake
FORCE (N)
30
20
10
0
0.0005
0.0006
0.0007
0.0008
DISPLACEMENT (m)

Figure 8.

Figure 9.

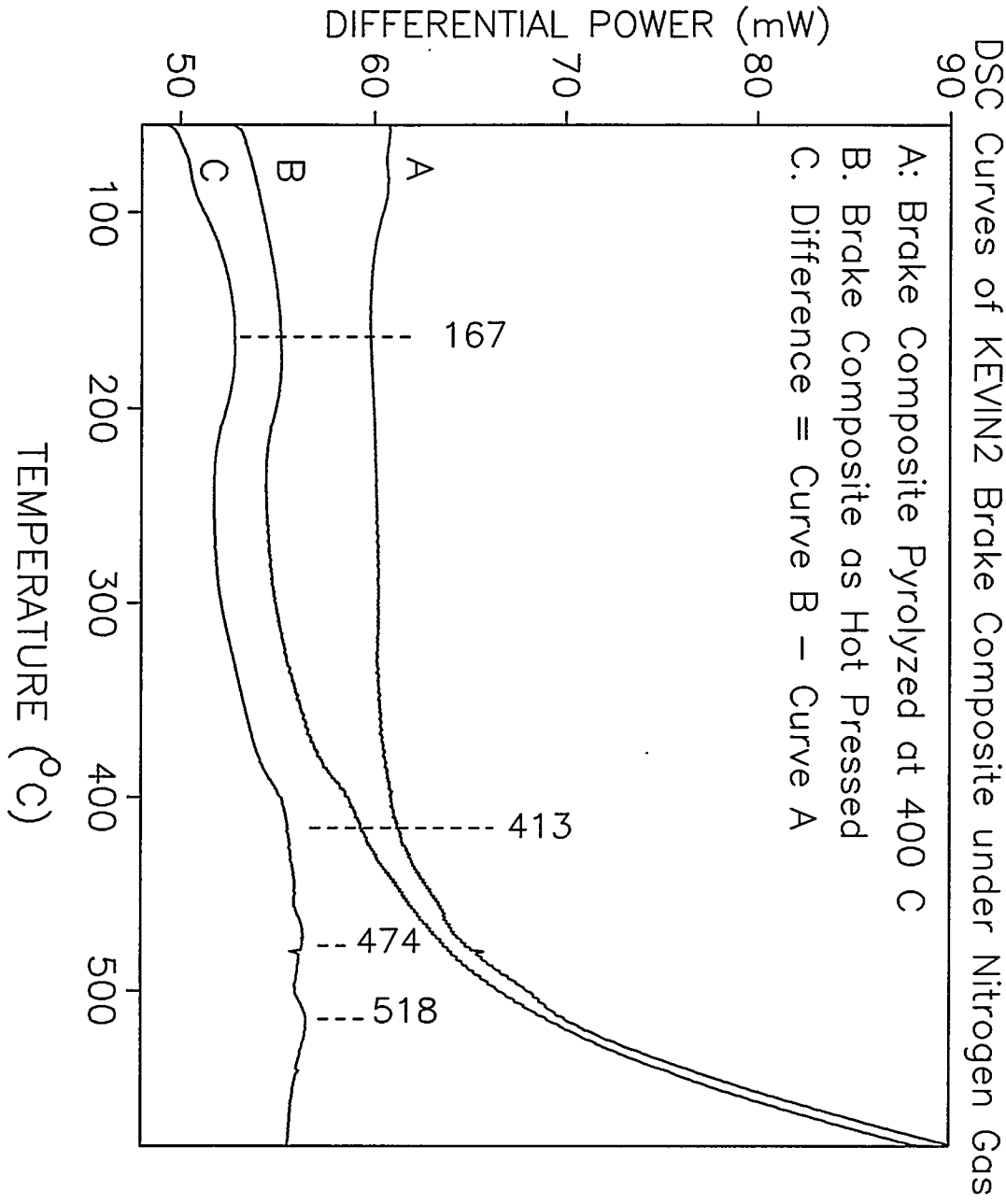
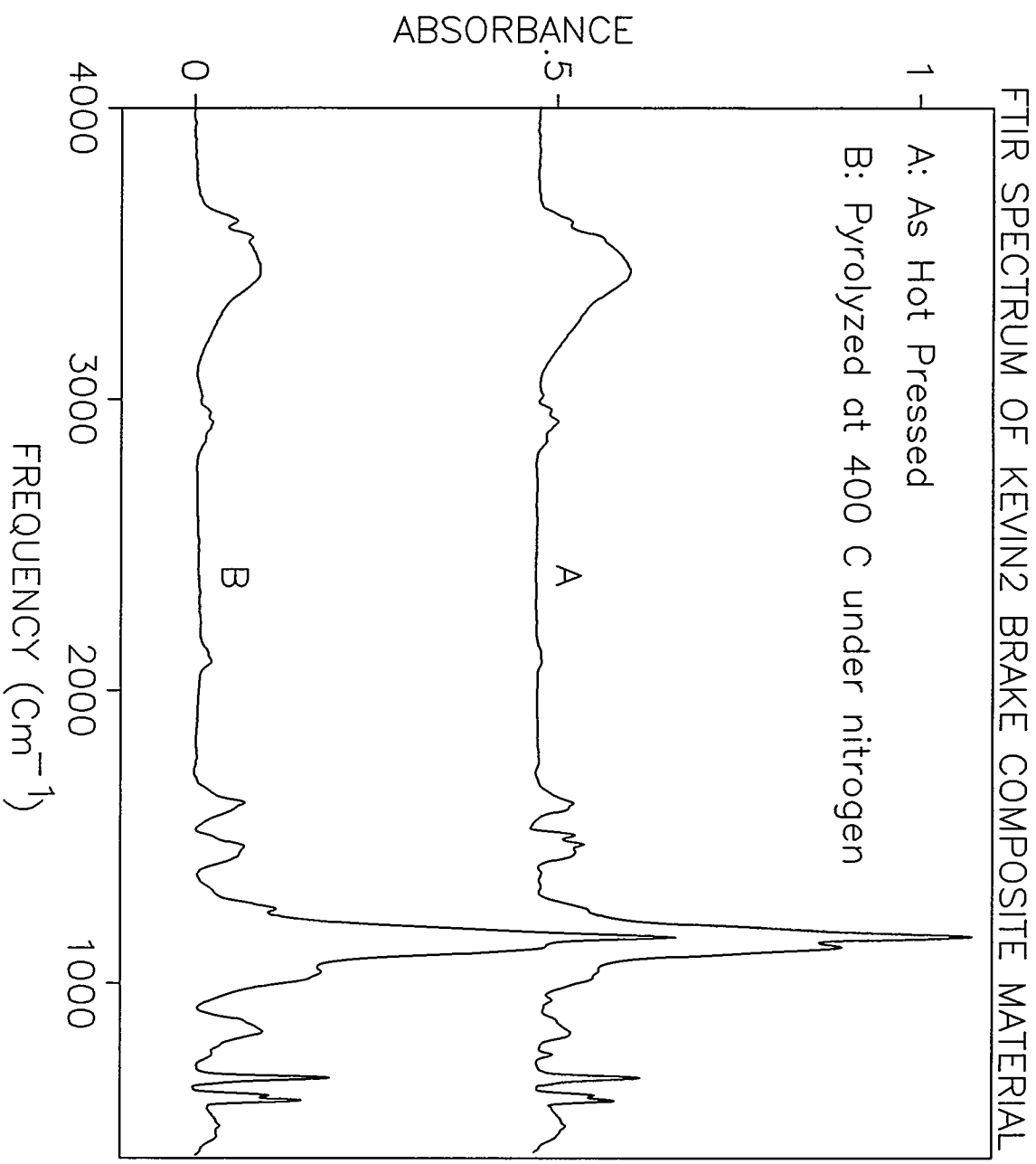

DSC Curves of KEVIN2 Brake Composite under Nitrogen Gas
A: Brake Composite Pyrolyzed at 400 C
B. Brake Composite as Hot Pressed
C. Difference = Curve B – Curve A
DIFFERENTIAL POWER (mW)
50
60
70
80
90
TEMPERATURE (°C)
100
200
300
400
500
A
B
C
167
413
474
518

Figure 10.


FTIR SPECTRUM OF KEVIN2 BRAKE COMPOSITE MATERIAL
A: As Hot Pressed
B: Pyrolyzed at 400 C under nitrogen
ABSORBANCE
1
.5
0
4000
3000
2000
1000
FREQUENCY (Cm⁻¹)
A
B

PROJECT MANAGEMENT REPORT
March 1 through May 31, 1995

Project Title: **DESIGN AND FABRICATION OF ADVANCED MATERIALS FROM ILLINOIS COAL WASTES**

DOE Cooperative Agreement Number:	DE-FC22-92PC92521 (Year 3)
ICCI Project Number:	94-1/3.1A-3M
Principal Investigator:	Vivak M. Malhotra, Department of Physics Southern Illinois University at Carbondale
Other Investigators:	Maurice A. Wright, Materials Technology Center, Southern Illinois University at Carbondale
Project Manager:	Dan Banerjee, Illinois Clean Coal Institute

COMMENTS

The project is progressing smoothly. In the last quarter, we conducted extensive scanning electron microscopy experiments at the Microscopy Center of the University. The bills are yet to arrive from the Center. Therefore, there will be higher expenditures next quarter. To further expedite the fabrication of the composites, three graduate students at half-time were hired for the summer.

DISCLAIMER

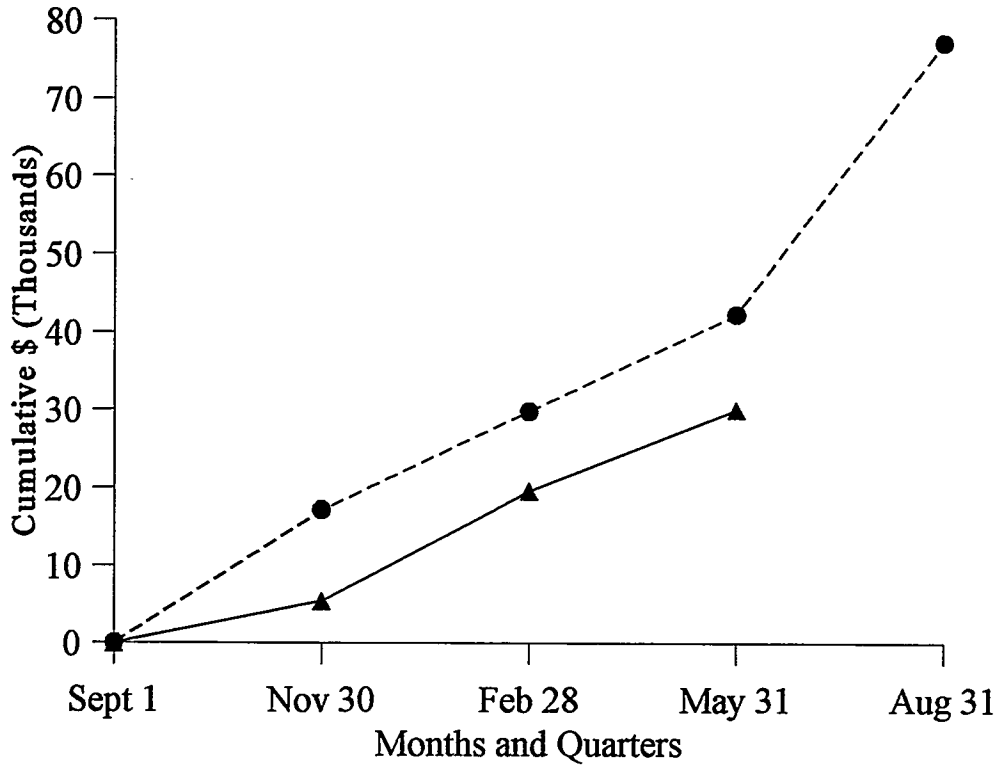
PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER

Quarter*	Types of Cost	Direct Labor	Fringe Benefits	Materials and Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Cost	Total
Sept. 1, 1994 to Nov. 30,1994	Projected	6,204	1,921	1,250	0	4,500	1,750	1,563	17,188
	Estimated	3,120	700	723	0	0	372	492	5,407
Sept. 1, 1994 to Feb. 28, 1995	Projected	12,408	3,842	2,500	300	4,500	3,500	2,705	29,755
	Estimated	8,856	1,685	1,539	0	4,500	1,224	1,780	19,585
Sept. 1, 1994 to May 31, 1995	Projected	18,612	5,764	3,750	600	4,500	5,250	3,848	42,324
	Estimated	15,176	2,696	1,619	0	4,500	1,424	2,542	27,957
Sept. 1, 1994 to Aug. 31, 1995	Projected	42,385	10,397	5,000	1,000	4,500	7,000	7,028	77,310
	Estimated								

*Cumulative by Quarter

CUMULATIVE COSTS BY QUARTER

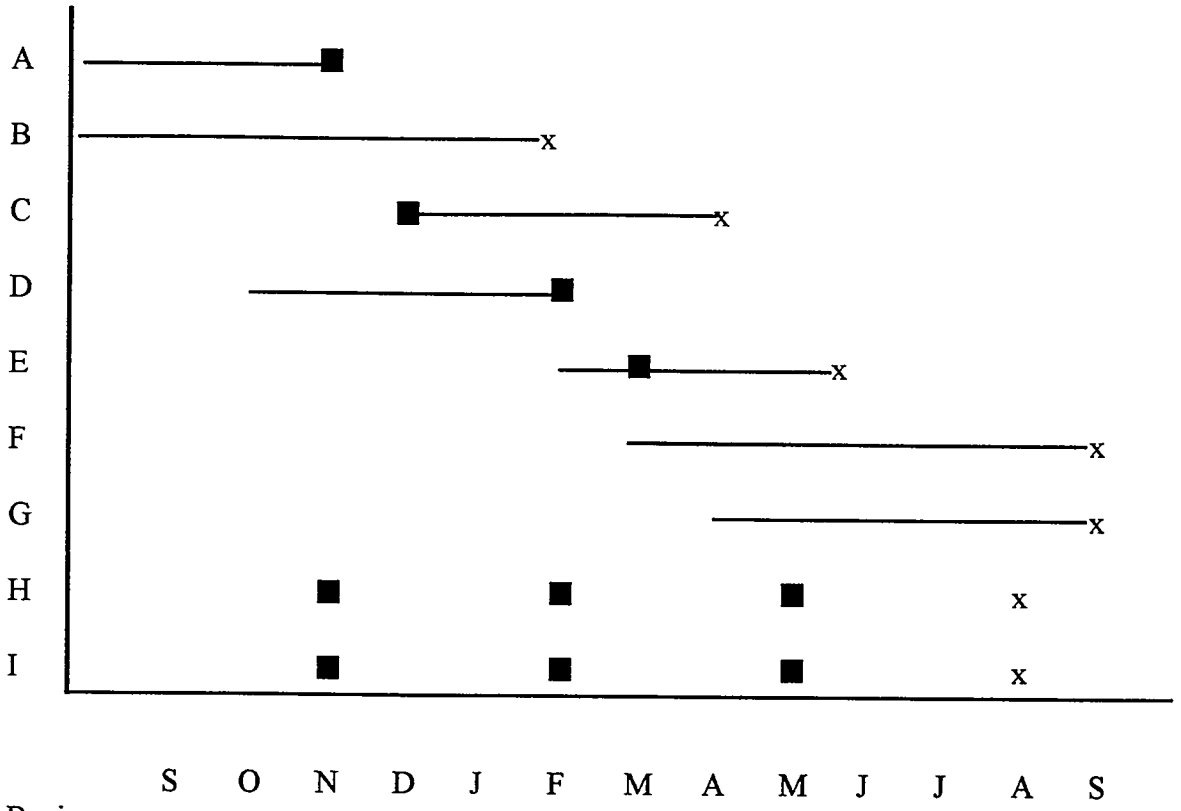
Design and Fabrication of Advanced Materials from Illinois Coal Wastes



● = Projected Expenditures - - - - - - -
▲ = Actual Expenditures _____

Total Illinois Clean Coal Instutute Award $77,310

SCHEDULE OF PROJECT MILESTONES



Hypothetical Milestones:

A: Order Equipment
B: Mineral Analyses of Residues
C: Thermal Stability of Residues
D: Altering Thermal Properties of Tar
E: Change Thermosetting Characteristics
F: Fabricate Brake Material
G: Fabricate Structural Material
H: Quarterly Technical Progress Reports
I: Quarterly Project Management Reports