Project Title: EXTRUDED FIBER-REINFORCED CEMENT COMPOSITES CONTAINING FLY ASH

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ABSTRACT

The major goals of the present research are to: 1) develop extrudable compositions of fiber-reinforced cement composites that contain substantial quantities of Illinois fly ash; 2) identify the best compositions for targeted products and 3) promote the most promising products for commercialization. The existing technologies and materials for producing precast elements are not suitable for this market. With further development, the extrusion technology can overcome these limitations. This high value-added product is targeted for the industrial and residential construction industries, including applications such as roofing materials, exterior siding, floor tiles, and wall, ceiling, and floor panels. This year involves development and testing of different compositions and specimen cross-sections. Optimum material compositions and combinations of hybrid fibers are studied to enable tailoring of the composite performance. The use of hybrid fibers enables a reduction in the cost and an improvement of the performance to take full advantage of the beneficial aspects of fly ash. The effect of fly ash on the durability of composites was evaluated, as was the effect of fiber dispersion on composite performance.

In previous years, it was shown experimentally that fiber-reinforced cement composites containing large amounts of Illinois fly ash could be extruded. As much as 80% by volume of the cement could be replaced by the Illinois fly ash to produce extruded fiber-reinforced composites with high strength and toughness. High-performance-fiber-reinforced composites were developed that can be used for lightweight elements for the industrial and residential construction industries. The flexural performance of extruded composites can be controlled and optimized with the use of hybrid fibers. The addition of polyvinyl alcohol (PVA) fibers to a glass and polypropylene (PP) hybrid composite significantly increases both the strength and the toughness of the composite and produces a strain-hardening response. Moreover, the long-term durability of the glass:PVA:PP hybrid composites is better than that of the glass fiber composites and the PVA fiber composites. The addition of fly ash further improve the durability of most composites. The composite cross-section was shown to influence its mechanical properties. Cellular cross sections with circular openings performed better than the those with square openings when tested dry; however, when tested saturated, the reverse trend was observed.
EXECUTIVE SUMMARY

The overall goals of the present research are to develop and promote the use of extruded fiber-reinforced cement composite components for residential and industrial construction. These products include exterior panels for industrial buildings, roofing materials (tiles, shingles, corrugated sheets), exterior residential siding, indoor-outdoor floor tiles, indoor wallboard, and decorative indoor wall, ceiling and floor panels. This is accomplished by: 1) finding the optimal composition with the maximum allowable content of the Illinois fly ash; 2) evaluating hybrid fiber reinforcement, i.e., combinations of different types of fibers such as polypropylene (PP), glass, and polyvinyl alcohol (PVA) fibers, to reduce the cost and to tailor the composite performance for a specific application; 3) evaluating the durability of the composites to improve performance, especially of glass fiber composites, with the addition of fly ash and hybrid fibers; 4) optimizing the cellular cross-sections, which offer reduced weight and increased rigidity, to minimize element weight and improve performance; 5) identifying the best compositions, cross-section shapes, and long-term durability for targeted products in industrial and residential construction; 6) working closely with ACBM's Industrial Affiliates to transfer this technology to the market.

Fibers are incorporated in the brittle cement matrix to control cracking by bridging the cracks, to provide high ductility, and to improve impact resistance and tensile and flexural strength. The relationship between the elastic modulus of the fibers and the elastic modulus of the cement matrix (about 15-30 GPa) influences the mechanical performance of the composite. Hybrid composites containing two or more different types of fibers can be considered to control the cost and optimize desired properties of the composite by taking advantage of the different properties of different types of fibers.

Extrusion is a processing technique that has been shown to impart high performance characteristics to fiber-reinforced, cementitious materials. Extrusion is a forming process in which a highly viscous, plastic-like mixture is forced through a die, a rigid opening of desired cross-section. There is growing interest in the use of the extrusion process in the fiber-reinforced cement composites industry. Extrusion yields a dense matrix with a strong fiber-matrix bond and good fiber alignment. It also allows greater flexibility in element shape than is possible with standard board manufacturing processes. It is a potential candidate for low cost commercial applications, such as roofing tiles, flooring tiles, building panels, and pressure pipes.

The extrusion technology has been shown to be superior to conventional production methods for cementitious composites in that the extruded composite is stronger, more ductile, making it better able to withstand hazards such as hail and wind load, and largely impervious to water penetration, offsetting freeze/thaw concerns. The resulting composite has been evaluated by a major producer of industrial siding and found to be cost-competitive. The existing technologies and materials for producing precast elements

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are not suitable for the residential construction market, since the products are too heavy and not cost effective. This has prevented market penetration of these products. With its lightweight, high value-added elements, extrusion technology can provide a more competitive product.

The proper viscosity (rheology) is essential for successful extrusion. If the rheology is not ideal, defects can form during extrusion leading to reduced performance of the composite. The round-particle morphology of fly ash improves the rheology of the extrudate, in addition to the ecological and economical benefits of its use in cement-based systems. Partial replacement of cement with fly ash not only makes cement-based elements less expensive; it also reduces the total life cycle energy consumption by reducing the amount of cement needed.

A laboratory ram extrusion rheometer was used to extrude small-scale sheets and cellular cross-sections. Specimens containing Illinois Class F fly ash as a replacement for cement were prepared with different fibers, PVA, glass, polypropylene, and hybrid fiber combinations. In most cases, 70% by volume of cement was replaced by fly ash. For comparison, specimens without fly ash were also prepared. The fiber volume fraction (V_f) was 2% for PVA fibers and 5% for the glass, polypropylene and the hybrid fibers. All these composites were extruded as 4 mm sheets. PVA fiber composites with a fiber volume fraction of 3% and 60% by volume of the cement replaced by fly ash were prepared to study the different specimen cross-sections, including 4 mm and 8 mm thick sheets, a cellular cross-section with square openings, a cellular cross-section with circular openings and a cellular cross-section with square openings whose corners are rounded.

After extrusion, the specimens were covered with a plastic sheet for 1-3 days. Then they were steam cured at 90°C and 100% relative humidity (RH) for 2 days or moist-cured for 28 days in 100% RH at room temperature. After removal from the curing environment, the specimens were stored at 22°C, 50% RH for 24 hours, dried at 105°C for 24 hours, and stored at 22°C, 50% RH for another 24 hours before testing in flexure. Moist-cured specimens were also tested under a saturated, surface-dry condition to study the effect of moisture content on the mechanical behavior. These specimens were kept under moist curing conditions until immediately prior to testing. To study the effect of fly ash on the long-term durability of the composite, glass, PP, PVA, and hybrid composites both with and without fly ash were stored in an 80°C water bath for 6 weeks, after being moist cured 28 days, to accelerate aging. The mechanical performance was evaluated by determining the flexural strength and toughness of the composites using three-point bend tests.

It was found that mixtures containing substantial quantities of Illinois Class F fly ash are extrudable and provide enhanced flexural properties.

One of the main advantages of the extrusion technique is that a greater variety of cross-sections can be produced than is possible with the standard manufacturing processes. One of the goals of the present work is to produce lightweight elements for precast applications. For these applications, lightweight elements, such as cellular cross-sections
are important to reduce the cost of transportation and erection. A variety of cross-sections were extruded using a number of different mixtures to optimize mixture design and cross-section performance. It was found that the composite cross-section influenced its mechanical properties with sheets generally performing better than cellular cross-sections. Cracking at the corners of the cellular specimens tested dry was observed with a scanning electron microscope (SEM). Cellular cross-sections with circular openings performed better than those with square openings when tested dry. However, when the specimens were tested wet, the square opening performed better than the circular opening. The drying process may increase cracking at the corners of the cross-section leading to a reduction in mechanical performance. The circular opening may improve the mechanical performance of the composite, but it also increases the unit weight of the element, a critical consideration for precast panels. In an effort to reduce the element weight while improving performance, cellular cross-sections with square openings whose corners are rounded were extruded. The unit weight of this cross-section was similar to that of the plain square openings, but after a 2-day steam cure, the performance was similar as well. Therefore, further modification of the die design is needed to minimize cracking and reduce the element density.

The flexural performance of extruded composites can be controlled and optimized with the use of hybrid fibers. A hybrid composite performs better than composites reinforced with each of its constituent fibers, indicating that hybrid fiber composites do allow for tailoring of composite performance and improved performance over individual fiber composites. Hybrid composites containing glass and polypropylene fibers showed a significant drop in load-carrying capacity at a relatively small deflection after the glass fibers failed. Even with a minimal amount of glass fibers, the drop in strength exhibited by the composite was not desirable. The addition of PVA fibers to these glass/PP hybrid composites significantly increases both the strength and the toughness of the composite and produces a strain-hardening response. The primary drawback of PVA fibers is that they are significantly more expensive than polypropylene fibers. A 40:40:20 glass:PVA:PP ratio composite with a total fiber volume fraction of 5% was found to be the optimal hybrid combination because it gave high performance (similar to a 40:60 glass:PVA hybrid composite) with relatively low cost. SEM analysis was used to examine the fracture surfaces of the plain and hybrid composites to determine if there were differences in microstructure that might explain the improved performance of the hybrid composite. However, no significant differences were observed among the composites.

All hybrid composites were tested with 70% of the cement in the matrix replaced with fly ash. The most successful hybrid composites, 20:80 glass:PP and 40:40:20 glass:PVA:PP, were also tested without fly ash. The presence of fly ash improved the flexural performance of the glass and polypropylene composite, however it did not have a significant effect on the performance of the glass, PVA and polypropylene composite. Similar trends were obtained for steam-cured specimens and moist-cured specimens.

In general, composites without fly ash are more adversely affected by aging. The glass and glass:PVA:PP hybrid composites with fly ash show little change in strength but a
significant reduction in toughness with aging. Without fly ash, these composites show a reduction in strength and a much greater reduction in toughness. The polypropylene fiber composite with fly ash shows significant increases in both strength and toughness with aging. These increases are much less when fly ash is not present. The increase in strength and the reduction in toughness may be explained by an improvement in fiber-matrix bonds with aging, as the fibers fracture instead of pulling out at failure. The increase in bond strength may be a result of a decrease in porosity with aging. This decrease was observed in mercury intrusion porosimetry and SEM analysis of the glass fiber composite. Because the polypropylene fiber composite has a relatively poor fiber-matrix bond, the improvement in bond strength with aging is not great enough to cause fibers to fracture, which may explain the increase in strength and toughness with aging. The durability of a composite can be controlled by hybrid fiber combinations and, in most cases, improved with the addition of fly ash.

Glass fiber composites were prepared with bundled and dispersible glass fibers to examine the effect of fiber dispersion on composite performance. The dispersible fiber composite performed much better than the bundled composite at a lower fiber volume fraction. Good fiber dispersion may improve composite performance because the fiber-free, or unreinforced, areas of the matrix are smaller. The addition of fly ash further enhanced the improved performance of the dispersible glass composite, suggesting that fiber dispersion may be aided by fly ash.

This project has attracted commercial interest. A Colorado based start-up company, which is engaged in developing the concept of modular concrete housing, is interested in using extruded composites as its construction modules. ACBM has been working closely with this company to develop an extrudable mixture using a new rapid-setting cement. A significant amount of Illinois Class F fly ash is added to achieve an extrudable mixture.

**Summary and Conclusions** The flexural performance of extruded composites can be controlled and optimized with the use of hybrid fibers. Increasing the glass fiber content while reducing the polypropylene fiber content increases the hybrid composite strength over the polypropylene fiber composite. The addition of PVA fibers to a glass/PP hybrid composite significantly increases both the strength and the toughness of the composite and produces a strain-hardening response. The 40:40:20 glass:PVA:PP ratio composite was found to be the best combination because it gave high performance (similar to a 40:60 glass:PVA hybrid composite) with relatively low cost. A composite with cellular cross-sections was successfully extruded to produce lightweight elements for precast applications. It was found that the composite cross-section influenced its mechanical properties. The shape of the opening of a cellular cross-section as well as the moisture content of the element affects the mechanical performance. A circular opening performs better than a square opening when the composite is tested dry, but a square opening performs better than circular when tested wet. Hybrid fiber combinations can control the durability of a composite, and the addition of fly ash can make them more durable. Fiber dispersion can have a significant effect on performance. It can be concluded that the replacement of cement with fly ash is beneficial for extruded fiber-reinforced cement composites.
OBJECTIVES

The objectives of the present research are to develop and promote fiber-reinforced extruded composites containing large amounts of Illinois Class F fly ash for siding for industrial buildings and residential construction applications. These objectives are achieved by: 1) developing extrudable compositions of fiber-reinforced cement composites that contain substantial quantities of Illinois fly ash, 2) identifying the best compositions for targeted products, and 3) promoting the most promising products for commercialization. Both large-scale and small-scale cellular cross-sections will be modified and tested for use in residential and lightweight commercial construction applications. Extrusion of cellular cross-sections is important where reduction in total weight and increase in structural rigidity is necessary. Compositional development will focus on: 1) finding the maximum allowable contents of Illinois fly ash, 2) evaluating hybrid fiber systems, i.e., combinations of different fibers, 3) optimizing the mix design for each hybrid system and for large-scale and small-scale cellular cross-sections. Hybrid systems could allow a better control of the composite performance. For example, combining glass, polypropylene (PP) and polyvinyl alcohol (PVA) fibers could yield high strength and high ductility composites. Moreover, the cost could be reduced if inexpensive fibers such as polypropylene could replace some of the more expensive PVA fibers. Different hybrid combinations were examined. The impact properties and long-term durability of the composites will be evaluated.

The major statement of work tasks to meet these objectives are outlined below:

1. **Industrial collaboration**: Work with the manufacturer of commercial siding, the residential construction industry (with PCI), and fiber manufacturers. In addition, enlist ACBM Center’s industrial affiliates to determine the commercial feasibility of the most promising products. Receive feedback in the form of suggestions for modifications to enhance marketability, such as ways to reduce costs, or to enhance properties like nailability.

2. **Development of hybrid systems**: Optimize fiber combinations for different hybrid systems using mechanical properties and rheological data. Design and extrude different hybrid fiber compositions with glass and other fibers such as polypropylene and PVA. Evaluate extrudability using extrusion rheometry and mechanical and durability testing.

3. **Optimization of the Illinois Class F fly ash content**: Determine optimum fly ash contents and fiber combinations for each set of desired properties by starting with a base matrix composition (no fly ash) and testing a range of fly ash additions, directly replacing cement. Establish fly ash dosages and fiber combinations based on modulus of rupture (MOR), toughness, and density.

4. **Development of batch processing procedures**: Evaluate elements of the batch processing procedure, including the order of addition of components, the appropriate mixing order of wet and dry ingredients, the degree of dispersion of fibers, the level of shear necessary during mixing to disperse fibers fully without damage, the homogeneity of the mixture and the quantity and distribution of entrapped air. Adopt procedures that yield the most homogeneous mixture for each fiber system.
5. **Scale-up and die modification**: Modify both large-scale and small-scale cellular dies for use in lightweight construction for commercial applications, focusing on the junction points of the element.

6. **Mechanical Testing**: Evaluate flexural and impact behavior of the composite to determine its properties for different material compositions and specimen shapes. Calculate flexural strength, or MOR, and toughness.

7. **Durability testing**: Evaluate the effects of accelerated aging on composite strength. Examine the influence of moisture content of the specimen prior to testing.

8. **Microstructural correlation with properties**: Correlate microstructural features such as fiber alignment, size and distribution of flaws and pores, degree of microcracking, and fiber damage due to aging, fiber pullout, or fiber fracture with composition, processing conditions, and performance.

**INTRODUCTION AND BACKGROUND**

Fibers are incorporated in a brittle cement matrix to control cracking by bridging the cracks, to provide high ductility and to improve impact resistance as well as increase both the tensile and flexural strength. The relationship between the elastic modulus of the fibers and the elastic modulus of the cement matrix (about 15-30 GPa) influences the mechanical performance of the composite. Hybrid composites containing two or more types of fiber can be considered to control the cost and to optimize the desired properties of the composite by taking advantage of the different properties of different types of fibers.

High-performance-fiber-reinforced cement composites (HPFRCC) are defined as those with improved tensile properties (1). Extrusion is one processing technique that has been shown to impart high-performance characteristics in fiber-reinforced cementitious materials (2, 3). In general, extrusion is a forming process in which a highly viscous plastic-like mixture is forced through a die, a rigid opening having the geometry of the desired cross-section. It is widely used in the ceramic, clay, and polymer industries. There is growing interest in the use of the extrusion process for the fiber-reinforced cement composites industry. With properly designed dies and properly controlled material proportions, the fibers can be aligned in the load-bearing direction during extrusion. The matrix properties and fiber packing can be controlled to achieve low porosity and to improve the bond between the fiber and matrix. One of the main advantages of this technique is that a greater variety of element cross-sections can be produced than is possible with the standard manufacturing processes. In this way, complicated solid cross-sections and open or cellular cross-sections can be extruded, usually with only minor modifications in batch composition. Extrusion is a potential candidate for commercial applications, such as roofing tiles, flooring tiles, building panels, and pressure pipes. The proper viscosity is essential for successful extrusion. The mixture must be plastic (soft) enough to flow under pressure and pass easily through the die, but it must also be rigid enough to resist deformation and maintain its shape after exiting the die. If the rheology is not close to ideal, defects can form in the material during extrusion leading to reduced performance. The round-particle morphology of fly ash improve the rheology of the extrudate in addition to the economical and ecological
benefits of using fly ash in cement-based elements. Therefore, fly ash should be considered as a replacement for cement when developing mixtures for extrusion.

Partial replacement of cement with fly ash not only makes cement-based elements inexpensive, it also reduces the total life-cycle energy consumption by lowering the amount of cement needed (4). The energy requirement for cement production in the US is about 6 millions BTUs per ton of cement. Any major reduction in the production of cement would have a significant impact on energy use and CO₂ emissions. An already promising area where substantial reductions can be made is in the partial replacement of cement in concrete with fly ash. The use of fly ash as a partial replacement for cement in cement-based construction materials results in: 1) a significant energy saving due to a lower total energy per construction unit, 2) a lower CO₂ emission per construction unit, and 3) a significant reduction in land filling of the ash.

Summary of Previous Results
For the past two years, the ACBM Center has been involved in development of extrudable material compositions of fiber-reinforced cement composites containing substantial quantities of Illinois Class F fly ash. The Illinois fly ash was examined as a replacement for cement in composites reinforced with different types of fibers, including PVA, cellulose, glass, polypropylene, and acrylic (PAN) fibers, and hybrid fiber combinations. Illinois Class F fly ash was shown to improve significantly the flexural behavior of the extruded composite for all types of fibers (5). The greatest improvement was obtained for composites containing PAN fibers when 70% by volume of the cement was replaced by fly ash as presented in Figure 1. Moreover, as much as 80% by volume of the cement was successfully replaced by Illinois fly ash. Such a significant improvement with the addition of fly ash was not obtained for similar composites produced by the conventional casting process (6). The Illinois fly ash substantially improved the rheology of the freshly extruded mixtures. This led to a reduction in composite defects and enhanced the composite properties.

Cellular cross-sections were extruded successfully for lightweight elements. It was found that the composite cross-section influenced its mechanical properties. The large-scale cellular composite showed lower strength than the small-scale specimens. This was due to fiber clumping at the junction points of the cellular element as well as higher porosity of this composite. Further development of cellular cross-sections should continue to optimize their performance.

These encouraging results show that mixtures containing substantial quantities of Illinois Class F fly ash are extrudable and have high flexural properties. This indicates that the use of Illinois Class F fly ash is very attractive for extruded fiber-reinforced cement composites. The progress of the past year is given in this report.
EXPERIMENTAL PROCEDURES

Materials and Specimen Preparation
Specimens containing Illinois Class F fly ash as replacement for the cement were prepared and compared with composites without fly ash. Different types of fibers including PVA, glass and polypropylene fibers were added to the mixtures, individually and in hybrid combinations. The fiber properties are presented in Table 1. The composites were prepared with 70% by volume of the cement replaced with fly ash and without fly ash.

The hybrid fiber composites were examined by preparing various hybrid combinations by volume percentages of glass:PP fibers, 0:100, 20:80, 40:60, 60:40, 80:20, and 100:0, and of glass:PVA:PP fibers with volume percentages of 40:60:0, 40:40:20, and 20:40:40. In all cases except the 100% PVA composite, the total fiber content was 5% by volume. The fiber content of the PVA composite was 2% by volume. All of these composites were extruded as 4mm thick sheets.

The basic mixture designs, by volume, for the hybrid systems included 30% Class F fly ash, 13% cement, 12% silica fume, 5% fibers, 1% superplasticizer and 1% methyl cellulose with a water/cement ratio of 0.24, by weight. The glass fiber composite, the PVA fiber composite and the polypropylene fiber composite as well as the hybrid fiber composites with a 20:80 ratio of glass:PP fibers and a 40:40:20 glass:PVA:PP fibers were also examined without fly ash, i.e., 43%, by volume, of cement.

To examine the effects of fiber dispersion, two different types of glass fiber composites were extruded, both with and without fly ash. Glass fibers are generally packaged in bundles. These fibers can either remain in bundles after mixing, bundled, or separate from the bundles when water is added, dispersible. Bundled glass fibers were used in the glass fiber composite and in hybrid composites containing glass. For comparison, a glass fiber composite containing 3%, by volume, of dispersible glass fibers was extruded both with and without fly ash.

Different specimen shapes, sheets and small-scale cellular cross-sections, were extruded from a composite reinforced with PVA fibers, to understand better the influence of the specimen cross-section on the mechanical performances. These specimens were extruded for a variety of mixtures and fiber lengths to determine the best mixture for extruding different cross-sections. The base mixture had 60% by volume of the cement replaced by the Illinois fly ash, 3% by volume 2 mm PVA fibers, and 6% by volume silica fume. The second mixture had no fly ash. In the third mixture, the silica fume content was doubled to 12% by volume. The fourth mixture kept the silica fume content at 12% and increased the fly ash replacement to 70% by volume of cement. The fifth mixture was the same as the base mixture with 6 mm PVA fibers instead of 2 mm.

4 mm and 8 mm thick sheets and three small-scale cellular specimens, one with square openings, one with circular openings, and one with square openings whose corners are rounded, were extruded. The circular opening was used to minimize cracking observed at
the corners of the square opening. The rounded-square opening was designed to minimize cracking at the corners as well as to reduce the unit weight of the element. Not all cross-sections were extruded for each mixture. The cross-sections extruded for each mixture are shown in Table 2. All mixtures for all cross-sections were steam cured for 2 days and tested dry. Specimens of the base mixture were also moist cured for 28 days and tested wet and dry to determine the effects of curing and testing conditions on different cross-sections.

**Extrusion Equipment and Procedure**

A laboratory extrusion rheometer was used to extrude the small-scale specimens in order to evaluate the rheology of extrudable mixtures. This extruder is a ram extruder. A piston forces the material through a die.

The mixing procedure for the mixtures for all types of fibers was as follows. First the liquid phase was mixed with the fibers to get proper fiber dispersion. Then, the solid materials were added to the mixture. All the components were mixed together for about 10-15 minutes.

After being extruded, the specimens were covered with a plastic sheet for three days (one day for PVA fibers). Then they were exposed to different curing regimes, two days of steam curing at 90°C and 28 days of moist curing at 100% RH and room temperature. To examine the durability of the composites, all specimens with fly ash and plain fiber composites and the best hybrid composites without fly ash were subjected to six weeks of accelerated in an 80°C water bath after a 28-day moist cure.

The steam-cured, moist-cured, and accelerated-aging specimens were tested dry. Moist cured specimens of different cross-sections were also tested under saturated, surface dry conditions to study the effect of the specimen moisture content on mechanical performance. Specimens tested dry were prepared in the following manner. After removal from the curing environment, they were stored for 24 hours at 22°C and 50% RH. Then they were dried at 105°C for another 24 hours and cooled to room temperature over 24 hours at 22°C and 50% RH. The saturated specimens were kept in the curing environment until immediately prior to testing. Any surface moisture was removed with a paper towel.

**Flexural test**

Three-point flexural tests were performed to characterize the mechanical behavior of the extruded specimens. The span was four inches for the thin-sheet specimens and eight inches for the thick-sheet and small-scale cellular specimens. The three-point bending tests were conducted in stroke control, incrementing displacement, at the rate of about 0.00045 in/sec. The test results shown are an average of at least four specimens for the small-scale specimens and at least three specimens for the large-scale cellular specimens. Typical stress-deflection curves representing the flexural behavior of the composites are chosen for comparison for sheet and cellular cross-section specimens.
Microstructure characterization
Scanning Electron Microscopy (SEM) – The fracture surfaces of specimens tested in flexure were examined with a scanning electron microscope (SEM), in an attempt to characterize the microstructure of the composite. Fragments of specimens obtained after flexural tests were dried at 60°C and then coated with gold prior to observation.

Mercury Intrusion Porosimetry (MIP) - MIP was used to determine the pore size distributions for 28-day moist-cured and aged glass fiber composites both with and without fly ash. The porosity of these composites was analyzed using an Autoscan 33 MIP with maximum applied pressure of 223 MPa. Samples for the porosimetry were prepared by crushing a specimen and drying it for 24 hours at 110°C.

Industrial Collaboration
ACBM has been working closely with a Colorado-based start-up company that is developing extruded elements for modular housing construction. Initial work focused on developing a rheology, using a proprietary, rapid-setting cement, that would lend itself to the continuous extrusion process. Initial trials were made with a small, ram extruder.

RESULTS AND DISCUSSION

Industrial Collaboration
The project to develop extruded, fiber-reinforced cement composites containing fly ash has certainly attracted commercial interest. Initial interest was expressed by several ACBM Industrial Partners, those commercial concerns closest to ACBM’s research.

Initial work with a major manufacturer of commercial siding to assess the feasibility of extrusion to produce high-quality wallboard was complete. While not commercialized, this company remains interested in manufacturing by extrusion and has not ruled out utilizing this work as the basis for a new product offering.

A second company that produces cellulose fibers has also exhibited a good deal of interest in this work, because the extruded composite contains fibrous material in addition to fly ash. It has commissioned additional, proprietary research at ACBM that complements this work.

This extrusion work came to the attention of a Colorado-based startup company, Company X, which is engaged in developing the concept of modular concrete housing. Company X would like to use extruded composites as their construction modules. In May 2000, Company X commissioned ACBM to investigate the efficacy of their proprietary, fly-ash-based cement in the ACBM extrusion process. Much progress has been accomplished.

Initial work focused on developing a rheology, using a proprietary, rapid-setting cement, that would lend itself to the continuous extrusion process. Initial trials were made with a small, ram extruder to assess the mix. A significant fraction of the fly-ash cement, up to 50% by volume, was replaced with Illinois Class F fly ash to arrive at the final extrusion
mix. The fly ash was added primarily as a rheological aid. It also helps delay the set time so that laboratory extrusion can be performed. The fly ash also helps to reduce the material costs. The work progressed to the point where a laboratory-scale auger extrusion run was completed, producing extruded samples of cellular cross section of sufficient quality to be used as demonstration samples for Company X investor presentations.

Company X has received an 18-month NIST Partnership for Advancing Technology in Housing (PATH) grant for the development of "an entirely new, low-cost, lightweight, building material." Company X is committed to using extrusion in the production of their wall panels, and they are actively investigating the production of other shapes needed for complete modular home construction. Company X is on an aggressive timetable to purchase a commercial extruder and establish a pilot production plant. They are holding discussions with the Evanston director of DCCA's Illinois Technology Enterprise Corporation about locating the new business in Illinois.

**Effect of specimen cross-section**

One of the main advantages of the extrusion technique is that a greater variety of element cross-sections can be produced than is possible with the standard manufacturing processes. Complicated solid cross-sections and open or cellular cross-sections can be extruded, usually with only minor modifications in batch composition. One of the goals of the present work was to produce lightweight cement board elements for residential construction products. In this type of application, the cement boards are made in the factory and then transferred to the building area. A reduction in weight is important in order to reduce the cost of transportation and erection. To this end, lightweight, large cellular cross-section elements were extruded. Seven-foot long cellular cross-sections, which were light enough to be carried easily, were extruded successfully.

The composite cross-section can influence not only the element weight but also its mechanical properties. Figure 2 presents the flexural strength and toughness obtained for composites with a variety of matrices all reinforced with PVA fibers of sheets of different thickness and different small-scale cellular cross-sections. It can be seen that the specimen cross-section effects the performance of the composite. The different cross-sections performed differently for different matrices, but in general, the sheets performed better than cellular-sections.

Of the different mixtures tested, not one showed clearly superior performance for all cross-sections, and each cross-section responded differently to the changes in the mixture design. The composite with fly ash was stronger than the one without for all cross-sections and tougher for all except the thick-sheet specimen. The increases in strength and toughness were smallest for the square cellular section. The greatest increases in strength and toughness were observed in the circular-cellular section and the thin-sheet section, respectively. An increase in the fly ash content, from replacement of 60% of cement to 70% of cement by volume, resulted in significant increases in toughness but reductions in strength. The composite with a lower silica fume content, 6% versus 12% by volume, is tougher for all cross sections but shows little or no change in strength. The
composite with 2 mm PVA fibers was 50% stronger and tougher than the one with 6 mm PVA fibers.

Cracking at the joints of previously extruded square cellular specimens tested dry was observed by SEM, as shown in Figure 3. These cracks can lead to reduction in the mechanical performance. To minimize this cracking, the small-scale cellular die was modified and specimens with circular openings were extruded. Figure 4 shows a comparison between the flexural behavior of the two cellular openings tested both wet and dry after a 28-day moist cure. It is clear that, when fly ash was used, the circular opening cross-sections were stronger and tougher when the specimens were dry (Figure 4a). However, when the specimens were tested wet, the square opening performed better than the circular opening (Figure 4b). When the specimens were steam cured and tested dry, the square opening cross-sections were weaker but more ductile than the circular openings, as shown in Figure 4c. The process of drying the specimen may increase the cracking at the square joints negatively affecting mechanical performance. However, when the performance of the square openings is compared to the circular and rounded-square opening for other mixtures tested dry after a steam cure (Figure 2), the differences in performance are not consistent enough to suggest that drying is the only factor. It is also important to note that the flexural strength of specimens tested dry, for both openings, is greater than those tested wet, but the dry specimens are more brittle.

The circular opening may improve the mechanical performance of the composite, but it also increases the density of the element. For identical mixtures, the unit weight of the cross-section with circular openings is 0.44 g/mm, while the unit weight of the cross-section with square openings is 0.39 g/mm. Such increase in the element density is critical when precast panels are considered, as mentioned previously. In an effort to minimize cracking at the joints while maintaining a lower element density, a cellular cross-section with square openings the corners of which were rounded was extruded. This cross-section had a unit weight of 0.39 g/mm—the same as for a cellular cross-section with plain square openings. However, after a two-day steam cure, the performance of this cross-section was not significantly different from that of the plain square cellular section, as shown in Figure 4c. This may indicate that simply rounding the corners of the square cellular cross-section is not sufficient to prevent cracking there during the drying process, or that cracking at the corners is either not occurring or not the cause of the poorer performance of the square section. More testing is needed to determine if there is in fact a significant difference in performance between the circular opening and the plain and rounded square opening cross-sections. In addition, the fracture surfaces of these specimens should be examined using SEM to determine if there is cracking at the corners of the rounded square cross-section. Further development of the cellular cross-section design may also be necessary.

Hybrid Composites
As shown in previous work, composite performance is significantly affected by the type and amount of fibers used as reinforcement. Specific fibers can be chosen based on the performance requirements of a given application. Composite performance can be further
tailed by using two or more types of fibers as reinforcement in a hybrid fiber composite. The mechanical performance of the composite is influenced by the relationship between the elastic moduli of the cement matrix and the fiber as well as the quality of the bond between the fiber and the matrix. In previous work, polypropylene fibers, with a modulus of elasticity lower than that of cement and a relatively poor fiber-matrix bond, were combined with glass fibers, whose modulus of elasticity is greater than that of the cement matrix and that have a strong fiber-matrix bond, to produce a composite that was both strong and tough. Varying combinations of polypropylene and glass fibers with a total fiber volume fraction of 5% were tested in a cement matrix with 70% of the cement replaced with fly ash. It was shown that increasing the amount of polypropylene fibers while decreasing the amount of glass fibers resulted in an increase in composite toughness with a reduction in composite strength.

Because glass fibers are much stronger and more brittle than polypropylene fibers, all the hybrid composites containing these two fibers showed a significant drop in load-carrying capacity at a relatively small deflection after the glass fibers failed. The higher the glass content the greater the drop in strength. Even with a minimal amount of glass fibers, the drop in strength exhibited by the composite, as shown in Figure 5, was not desirable. In an attempt to avoid this drop, a hybrid composite reinforced with glass and PVA fibers was tested. The ultimate tensile strength of PVA fibers is much closer to glass fibers than the strength of polypropylene fibers is, but PVA fibers are more ductile than glass. This resulted in a tougher composite than the glass and polypropylene hybrid composites that showed strain hardening instead of a drop in strength after the tensile capacity of the glass fibers was reached. The primary drawback of PVA fibers is that they are significantly more expensive than polypropylene fibers. To reduce cost, a portion of the PVA fibers was successfully replaced with polypropylene fibers without a significant reduction in composite strength or toughness. The various glass, PVA, and polypropylene hybrid combinations tested are shown in Figure 6. The 40:40:20 glass:PVA:PP composite was chosen as the best combination because its performance is similar to that of the 40:60 glass:PVA hybrid composite while replacing some of the PVA fibers with less expensive polypropylene fibers. The improvement in performance of the hybrid composite as compared with plain fiber composites is shown in Figure 7. The hybrid composite performs better than composites reinforced with each of its constituent fibers, indicating that hybrid fiber composites do allow for tailoring of composite performance and improved performance over individual fiber composites. All hybrid composites were tested with 70% of the cement in the matrix replaced with fly ash.

The hybrids showed similar trends for steam-cured specimens and moist-cured specimens. However, the moisture content of the specimen during testing significantly affected performance. Flexural strength was higher for all composites tested dry, with greater improvement when the polypropylene content was higher than glass. This trend was reversed for toughness. Polypropylene fibers were more brittle when tested wet, while glass fibers tested wet were more ductile. A hybrid with a higher glass content was, therefore, tougher when tested wet.
The 20:80 glass:PP composite was also tested without fly ash to determine the effect of fly ash on composite performance. A comparison of the performance of this composite with and without fly ash after a 28-day moist cure, tested dry, is shown in Figure 8. As expected, the hybrid composite with fly ash was both stronger and tougher than the composite without fly ash. Both the plain polypropylene and the plain glass composites show improved performance with the addition of fly ash.

Scanning electron microscopy was used to compare the microstructure of plain and hybrid fiber composites both with and without fly ash by examining the fracture surfaces of these composites after testing in flexure. Microstructural differences could explain the differences in performance between plain and hybrid composites. Specimens that had been moist-cured for 28 days and tested dry were examined. The matrix was examined to look for differences in porosity and fiber dispersion. A qualitative assessment of the fiber to matrix bond strength was made based on the relative size of the gaps between the fiber and the matrix. The length of the fibers protruding from the matrix gave information on whether fibers pulled out or fractured at failure. Fiber surfaces were examined to look for fiber damage, the formation of fibrils on the fibers, or particles on the surface, which might influence performance. In general, the differences observed were neither consistent nor significant enough to draw conclusions about the differences in composite performance.

**Fiber Dispersion**

To examine the effect of fiber dispersion on composite performance and the effect of fly ash on fiber dispersion, glass fiber composites, with and without fly ash, were tested with two different types of fibers, bundled and dispersible. All the glass fibers used in this work were packaged in bundles. The dispersible glass fibers separated when added to the liquids of the mixture and dispersed throughout the matrix, while the bundled fibers did not. A comparison of the performance of glass fiber composites with bundled and dispersible fibers both with and without fly ash after a 28-day moist cure is shown in Figure 9. The dispersible fibers (Figure 9a) greatly outperform the bundled fibers (Figure 9b), especially when fly ash is added to the matrix. The dispersible glass fiber composites had a $V_f$ of 3%, while the bundled glass fiber composites had a $V_f$ of 5%. The dispersible glass composite without fly ash performed as well as the bundled glass fiber composite without fly ash, even with the lower fiber volume fraction. When fly ash was added to the matrix, the dispersible glass composite had comparable strength and was much more ductile than the bundled glass composite, again with a lower fiber volume fraction. These results clearly indicate that better fiber dispersion improves performance. If the fibers are well dispersed, then the unreinforced, or fiber-free, areas of the matrix will be smaller, preventing crack propagation and thus strengthening the composite. The significant improvement in performance of the dispersible glass composite with the addition of fly ash may be a result of a reduction in bond strength with the addition of fly ash, or it may suggest that fly ash serves as an aid to fiber dispersion.
Durability
After a 28-day moist cure, specimens were placed in an 80°C water bath for six weeks to accelerate aging. A comparison of the strength and toughness of glass, PVA, and polypropylene fiber composites and hybrid composites with and without fly ash after a moist-cure and after accelerated aging is shown in Figure 10. In general, the composites without fly ash were more adversely affected by aging. The glass and glass:PVA:PP hybrid composites with fly ash show little change flexural strength but a significant reduction in toughness with aging. Without fly ash, both these composites show a much greater reduction in toughness and a reduction in strength. The polypropylene fiber composite with fly ash shows significant increases in both strength and toughness with aging. These increases are much less when fly ash is not present. However, the PVA composite without fly ash was less affected by aging than the one with fly ash. There was no change in strength or toughness with aging, while the PVA composite with fly ash showed a significant reduction in toughness with aging.

The increase in strength and the reduction in toughness may be explained by an improvement in fiber-matrix bonds with aging, as the fibers fracture instead of pulling out at failure. The increase in bond strength may be a result of a decrease in the porosity of the composite with aging as shown by MIP, in Figure 11. Composites without fly ash have a lower porosity to begin with and exhibit a greater decrease in porosity with aging, which may explain the greater reduction in toughness with aging in composites without fly ash. Because the PP fiber composite has a relatively poor fiber-matrix bond, the improvement in bond strength with aging is not great enough to cause fibers to fracture instead of pulling out, which may explain the increases in both strength and toughness with aging. The extent of the changes in strength and toughness in the hybrid composites appears to depend on the amounts of each of the fibers present (Figure 10). The increase in strength with aging is greater for hybrid composites with larger amounts of polypropylene fibers. The reduction in toughness with aging in the hybrid composites decreases with smaller amounts of glass fibers and PVA fibers.

An examination of the microstructure of glass fiber composites with and without fly ash further supports the theory of increased bond strength with aging. Figure 12 shows micrographs of fiber-matrix bonds in these specimens. The fly ash particles are easily identified by their regular, spherical shape (Figure 12a & 12b). The gaps between the fibers and the matrix are larger in the specimens with fly ash than in those without fly ash. These gaps appear to be smaller after aging in both the specimens with and without fly ash is indicative of a weaker fiber-matrix bond. Some degradation of the fiber surface can be observed in the aged composite without fly ash (Figure 12d).

Impact Testing
The impact resistance of extruded fiber-reinforced cement composites is of particular importance in the potential commercial applications of such products. Impact tests were to be performed on extruded composites this year. However, the preparation of the impact-testing machine was much more complicated and required more labor than anticipated. As a result, impact tests have not yet begun. Instead the work this year has
focused on modifying the testing machine to record the information required from these tests. The machine must be instrumented so that load-displacement curves can be acquired during the test.

CONCLUSIONS AND RECOMMENDATIONS

Mixtures containing substantial quantities of Illinois Class F fly ash are extrudable and have high flexural properties. Most of the composites tested were stronger, tougher, and more durable when some of the cement was replaced with fly ash. This performance makes the use of Illinois fly ash very attractive for extruded fiber-reinforced cement composites.

A variety of matrices, all reinforced with PVA fibers, were tested to optimize mixture design for extrusion of different cross sections. While no one mixture gave the best performance for each cross-section, some general trends were observed. The addition of fly ash improved both the strength and toughness for all cross-sections and made the composites much easier to extrude. Composites with more fly ash, 70% replacement of cement by volume versus 60% replacement, were tougher but weaker. A higher silica fume content, 12% by volume instead of 6%, reduced the toughness of the composite substantially, but did not have a significant effect on strength. Shorter PVA fibers performed better than longer ones.

Cellular cross-sections were successfully extruded, providing potential lightweight elements for precast applications. However, these sections showed the worst performance out of a variety of cross-sections tested. Cracking at the joints of the cellular specimens was observed by SEM. To avoid this problem, a cellular cross-section with circular openings was extruded. These specimens performed better than those with square openings when tested dry, however, when the specimens were tested wet, the square opening performed better than the circular opening. The circular opening performed better than the square opening for all mixtures except the one without fly ash after when tested dry after a steam cure.

The circular opening may improve the mechanical performance of the composite, but it also increases the unit of the element. Such an increase in the element density is critical when precast panels are considered. In an effort to reduce cracking and maintain a lower element density a cellular cross-section with square openings with rounded corners was developed. This cross-section had a unit weight very close to that of the plain square opening cross-section. However, it showed no improvement in performance. Therefore, further modification of the die design is needed to minimize cracking and reduce the element density.

The flexural performance of extruded composites can be controlled and optimized with the use of hybrid fibers. The hybrid composite performs better than composites
reinforced with each of its constituent fibers. A glass and polypropylene hybrid is stronger than a plain polypropylene composite and tougher than a plain glass composite, but exhibits an undesirable drop in strength after the glass fibers reach their tensile capacity. A glass and PVA hybrid shows improved strength and toughness with a strain hardening response, however PVA fibers are significantly more expensive than glass or polypropylene. Some of the PVA fibers can be replaced with polypropylene, and the composite will maintain its high performance with reduced cost.

Hybrid fiber composite performance is not affected by different curing regimes. However, the moisture content of the specimen during testing does affect performance. Flexural strengths are higher when composites are tested dry, but the toughness depends on the amounts of each fiber present. A composite with greater polypropylene content is tougher when tested wet, while the reverse is true for a composite with greater glass content.

The replacement of some of the cement in the matrix with fly ash generally improves the performance of the extruded composite, but the effect is dependent on the fiber type. The plain fiber composites, all of the glass:PP hybrid composites and the 40:40:20 glass:PVA:PP composite were tested with 70% of the cement in the matrix replaced with fly ash and without fly ash. Generally, the addition of fly ash improved the strength and ductility of the composites.

Fiber dispersion has a significant effect on the performance of glass fiber composites. Composites containing glass fibers that remained bundled performed worse than composites reinforced with fibers designed to disperse throughout the matrix, both with and without fly ash. The addition of fly ash made the improvement in performance of the dispersible fibers over the bundled fibers even greater. This may be a result of reduced bond strength with the addition of fly ash, or it may suggest that fly ash serves as an aid to fiber dispersion. The influence of fiber dispersion on composite performance should be the same for any type of fiber. As the dispersion improves more of the matrix is reinforced, improving performance.

The durability of a composite can be controlled with hybrid fiber combinations and is affected by the presence of fly ash. The extent of the changes in strength and toughness in the hybrid composites appears to depend on the amounts of each of the fibers present. A hybrid composite containing a higher percentage of a more durable fiber, such as polypropylene, is less affected by aging than a composite with a higher percentage of a less durable fiber, such as glass. Composites without fly ash tend to be more adversely affected by aging. For instance, glass composites without fly ash show a greater reduction in strength and toughness with aging.

Future work will include testing hybrid systems tested in impact to determine how hybrid fiber combinations effect the impact resistance of a composite. Further modification of the cellular die design will continue to minimize cracking at the corners of the cross-section and reduce the element density. Collaboration with the industrial partner will continue to commercialize this development. In addition, ACBM will be promoting this
material and process for use in different building material products, such as roof tile, residential siding, and floor tile.

REFERENCES


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**Table 1: Properties of the various fibers**

<table>
<thead>
<tr>
<th>Type of Fiber</th>
<th>Tensile Strength (MPa)</th>
<th>Modulus of Elasticity (GPa)</th>
<th>Fiber Diameter (μm)</th>
<th>Fiber Length (mm)</th>
<th>Density (Kg/m³)</th>
</tr>
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<tbody>
<tr>
<td>Glass</td>
<td>3500</td>
<td>71</td>
<td>14</td>
<td>6</td>
<td>2680</td>
</tr>
<tr>
<td>Polypropylene (PP)</td>
<td>700</td>
<td>5</td>
<td>50</td>
<td>13</td>
<td>910</td>
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<tr>
<td>PVA</td>
<td>1900</td>
<td>41</td>
<td>14</td>
<td>2</td>
<td>1300</td>
</tr>
<tr>
<td>Acrylic (PAN)</td>
<td>300</td>
<td>8</td>
<td>37</td>
<td>6</td>
<td>1180</td>
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</table>

**Table 2: Cross-sections tested for various mixtures.**

<table>
<thead>
<tr>
<th>Mixture</th>
<th>4 mm sheet</th>
<th>8 mm sheet</th>
<th>Square cellular</th>
<th>Circular cellular</th>
<th>Rounded-square cellular</th>
</tr>
</thead>
<tbody>
<tr>
<td>60% FA, 6% SF</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>No FA, 6% SF</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>60% FA, 12% SF</td>
<td>X</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>70% FA, 12% SF</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>60% FA, 6% SF, 6 mm PVA</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>X</td>
</tr>
</tbody>
</table>

**Figure 1:** Flexural behavior of acrylic fiber composites with and without fly ash.
Figure 2: Flexural strength and toughness of PVA fiber composites of various cross-sections.
Figure 3. SEM micrograph of the corner of a square opening cellular composite.

Figure 4. Flexural behavior of cellular composites with circular, square, and rounded-square openings: a) after 28-day moist cure, tested dry; b) after 28-day moist cure, tested saturated; c) after 2-day steam cure, tested dry.
Figure 5. Flexural behavior of glass and polypropylene hybrid composites.

Figure 6: Flexural response of hybrid composites with various glass:PVA:PP combinations.
Figure 7: Flexural behavior of 100% glass, PVA and PP fiber composites compared with hybrid composite.

Figure 8. Behavior of 20:80 glass:PP hybrid composite with and without fly ash.
Figure 9. Behavior of glass fiber composites with and without fly ash: a) dispersible; b) bundled.

Figure 10. Performance of composites with and without fly ash after 28-day moist cure and 6 weeks accelerated aging: a) flexural strength, with fly ash; b) toughness, with fly ash; c) flexural strength, without fly ash; d) flexural strength, without fly ash.
Figure 11. Porosity of glass fiber composites with and without fly ash after a 28-day moist cure and accelerated aging.
Figure 12. SEM micrographs of aged and unaged glass fiber composites: a) 28-day moist cure, with fly ash; b) 6 weeks accelerated aging, with fly ash; c) 28-day moist cure, without fly ash; d) 6 weeks accelerated aging, without fly ash.