

QUARTERLY REPORT #2

FIBROUS MONOLITH WEAR RESISTANT COMPONENTS FOR THE MINING INDUSTRY

2ND TECHNICAL QUARTERLY REPORT

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FIBROUS MONOLITH WEAR RESISTANT COMPONENTS FOR THE MINING INDUSTRY

ABSTRACT

A set of materials property data for potential wear resistant materials was collected. These materials are designated for use as the 'core' materials in the Fibrous Monolith structure. The material properties of hardness, toughness, thermal conductivity and cost were selected as determining factors for material choice. Data for these four properties were normalized, and weighting factors were assigned for each property to establish priority and evaluate the effects of priority fluctuation. Materials were then given a score based on the normalized parameters and weighting values. Using the initial estimates for parameter priority, the highest ranking material was tungsten carbide, with diamond as the second ranked material. Several materials were included in the trade study, and five were selected as promising 'core' materials to include in this effort. These materials are tungsten carbide, diamond, boron carbide, titanium diboride and silicon carbide. Work was initiated on a trade study to evaluate 'shell' materials. These materials will require the investigation of different material properties, including ultimate tensile strength, ductility, toughness, thermal expansion, thermal conductivity and compatibility during consolidation with the 'core' materials.

Kyocera Industrial Ceramics in Kyoto, Japan was visited, with the purpose of negotiating and signing the subcontract for Kyocera's participation on this program. An assessment was made on the testing and manufacturing capabilities of Kyocera and how such capabilities can be integrated into our development effort. Tours were conducted of Kyocera's machine tool production plant in Sendai, Japan, as well as their research and development facilities in Kagoshima, Japan. Kyocera's facilities include substantial materials characterization and testing capabilities at room and elevated temperatures, and manufacturing capabilities of thousands of parts/hr, all of which will be made available to us for use on this program as part of Kyocera's in-kind program cost share contribution. The Kyocera subcontract and the details of Kyocera's participation on this program were discussed and agreed upon during the two-day meeting (see Attachment A). Kennametal's Vice President and Chief Technical joined discussions regarding potential 3-way collaborations between Kyocera, ACR Inc. and Kennametal. This collaboration would involve the utilization of Kennametal's Rapid Omni-Directional Compaction Process (ROC Process) in the production of FM-based cutting tools. Kyocera and ARC Inc are in the process of evaluating the potential of this process in the fabrication of wear resistant composite tooling.

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INTRODUCTION

This program addresses the mining industry's need for improved components for wear resistance. The cost/performance ratio drives the application of components and materials used in mining applications. The mining industry traditionally had little use for advanced wear resistant materials due to their high cost relative to their improved durability. The goal of this program is to offer advanced wear resistant materials, in the form of fibrous monolith composites, that will overcome the cost/performance barrier traditionally associated with advanced materials and significantly increase the wear life of targeted components. Materials systems that exhibit promise as a crosscutting technology where resistance to wear is important will also be developed. Research will be performed on other applications, such as metal cutting tools, as crosscutting technologies are developed and translated into other industries.

The program is a collaborative effort of component manufacturers, end users, a national laboratory, and universities. The program will target three particular wear components which offer a broad cross-section of wear conditions and environments encountered in the mining industry. These components are: 1) drill bit inserts used for drilling blast holes and oil and gas wells, 2) dozer teeth used in a variety of earth-moving equipment, and 3) hydro cyclone apex cones, used in cyclone separators for sizing of crushed ore. As the program progresses these target items will be evaluated for appropriateness to the goals of the program. The program team will design fibrous monolith structures or coatings into existing components. The program team members will fabricate, inspect, and test the components in real operating environments. Team members will also develop process workbooks for fabricating fibrous monoliths, non-destructive evaluation of components, and modeling of composite/component behavior under typical stress and wear conditions. This body of knowledge will be used as a basis for future work.

Fibrous Monolith Composites

Fibrous monoliths (FMs) are a new and very versatile class of structural ceramics. They have mechanical properties similar to CFCCs, including very high fracture energies, damage tolerance, and graceful failures but can be produced at a significantly lower cost. Since they are monolithic ceramics, FMs are prepared using a simple process in which ceramic and/or metal powders are blended with thermoplastics and melt extruded to form a flexible bi-component 'green' fiber (**Figure 1**). These fibers can be compacted in the 'green' state to create the fabric of polycrystalline cells after sintering. The process is widely applicable, allowing the cell/cell boundary bi-component fibers to be made from any thermodynamically compatible set of materials available as sinterable powders. The scale of the macro-structure is determined by the green fiber diameter (cell size) and coating thickness (cell boundary). Once the green composite fiber is fabricated it can be wound or braided into the shape of the desired component using any conventional composite architecture. The thermoplastic binder is removed in a binder burnout step and is then hot pressed or sintered to obtain a fully dense component.

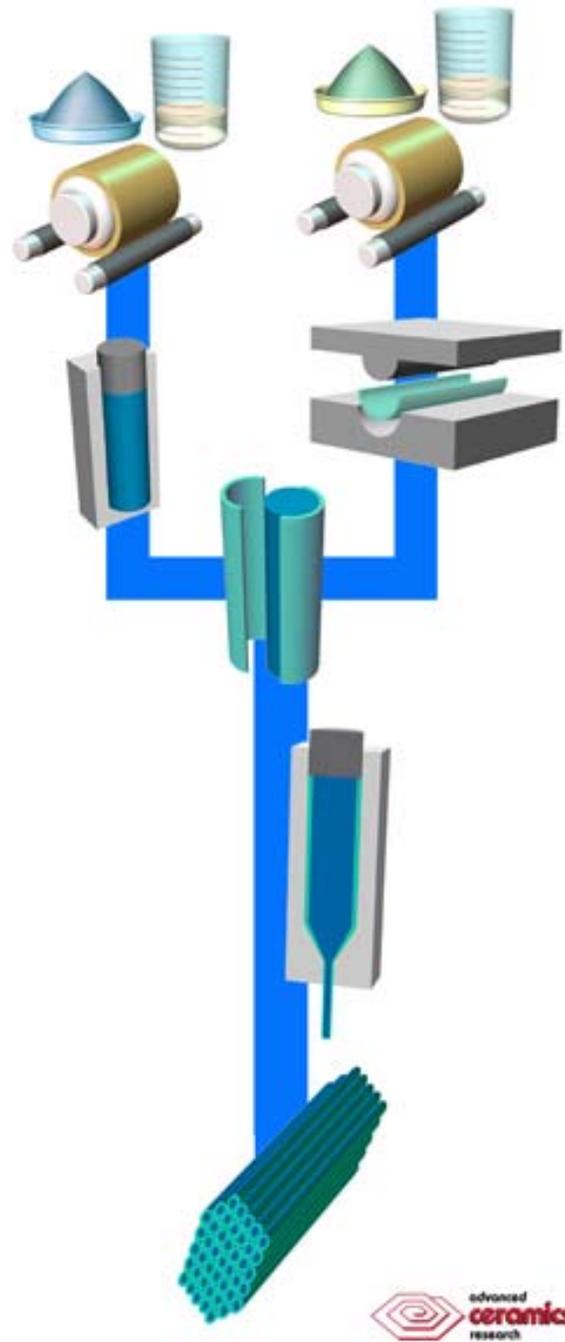


Figure 1. Illustration of the Fibrous Monolith co-extrusion process. Two ceramic and/or metal powders are blended separately with thermoplastics and plasticizers. The resulting mixtures are pressed into shells and rods. The shells and rods are laminated to form a composite feedrod that is then placed in a heated die and co-extruded. The resulting green coaxial filament is laid-up, wound or woven into the desired component. The component is then delubed to remove the plastics and then hot pressed or sintered to densify the composite.

When viewed perpendicular to the fiber direction after densification, the two phases that make up the architecture of a FM composite are a primary phase that appears as a hexagonal polycrystalline cell, separated by a thin and continuous secondary phase (cell boundaries) as shown **Figure 2**. Volume fractions of the two phases in an FM composite that result in the best composite properties are typically 75 to 90 % for the primary phase (polycrystalline cell), and 10 to 25% for the continuous phase (cell boundary). The cell phase is typically a structural ceramic, such as ZrC, HfC, TaC, Si₃N₄, SiC, ZrB₂, HfB₂, ZrO₂, or Al₂O₃, while the cell boundary phase is typically either a ductile metal, such as W-Re, Re Ni, Ni-Cr, Nb, or a weakly-bonded, low-shear-strength material such as graphite or hexagonal BN.

Past research has shown that the low shear strength cell boundaries such as BN and graphite accommodate the expansions and contractions during thermal cycling of the FM composite components, resulting in improved thermal shock resistance. From the mechanical behavior viewpoint, the BN or graphite cell boundaries enables non-catastrophic failure due to stress delocalization and crack deflection mechanisms (**Figure 3**). This has been successfully demonstrated previously at both room and elevated temperatures. In addition, the presence of a ductile or relatively ductile cell boundary phase greatly increases the damage tolerance and wear resistance of the Fibrous Monolith composite. For example, a diamond-based FM composite with a relatively ductile WC-Co interface forms a very wear resistant and damage tolerant composite that can be applied as a coating to drill bit inserts for use in rock drilling applications for oil, gas, and ore deposit exploration and production (**Figure 4**).

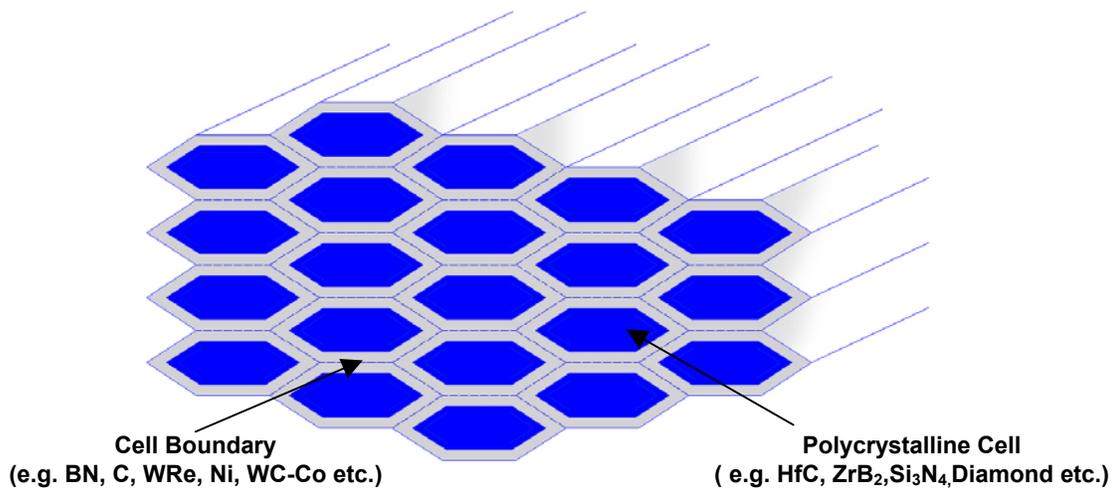


Figure 2. Schematic of a typical uniaxial Fibrous Monolith microstructure shown perpendicular to principal fiber direction.

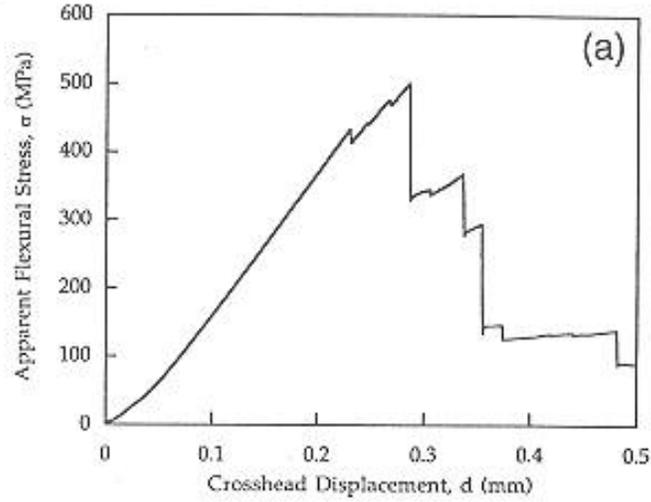


Figure 3. Typical flexural stress-strain curve for a silicon nitride/BN FM material.

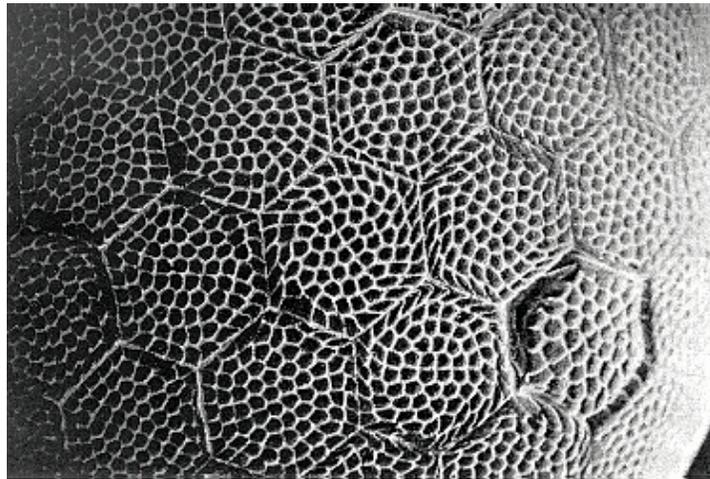


Figure 4. ACR's Diamond/ WC-Co FM composite applied as a coating on the surface of a WC drill bit insert (100x). Note the isolation of the darker material (Diamond) into discrete cells by the lighter contrast phase (WC-Co).

EXECUTIVE SUMMARY

This program addresses the mining industry's need for improved components for wear resistance. The cost/performance ratio drives the application of components and materials used in mining applications. The mining industry traditionally had little use for advanced wear resistant materials due to their high cost relative to their improved durability. The goal of this program is to offer advanced wear resistant materials, in the form of fibrous monolith composites, that will overcome the cost/performance barrier traditionally associated with advanced materials and significantly increase the wear life of targeted components. Materials systems that exhibit promise as a crosscutting technology where resistance to wear is important will also be developed. Research will be performed on other applications, such as metal cutting tools, as crosscutting technologies are developed and translated into other industries.

The program is a collaborative effort of component manufacturers, end users, a national laboratory, and universities. The program will target three particular wear components which offer a broad cross-section of wear conditions and environments encountered in the mining industry. These components are: 1) drill bit inserts used for drilling blast holes and oil and gas wells, 2) dozer teeth used in a variety of earth-moving equipment, and 3) hydro cyclone apex cones, used in cyclone separators for sizing of crushed ore. As the program progresses these target items will be evaluated for appropriateness to the goals of the program. The program team will design fibrous monolith structures or coatings into existing components. The program team members will fabricate, inspect, and test the components in real operating environments. Team members will also develop process workbooks for fabricating fibrous monoliths, non-destructive evaluation of components, and modeling of composite/component behavior under typical stress and wear conditions. This body of knowledge will be used as a basis for future work.

PROGRAM MANAGEMENT

During the week of April 9th to the 13th, ACR Inc Chief Operating Officer Mark Angier, Vice President of Marketing and Sales Matthew Pobloske, and Manager of Composite Ceramics Mark J. Rigali visited Kyocera Industrial Ceramics in Kyoto, Japan. The purpose of this trip was to negotiate and sign the subcontract for Kyocera's participation on this program. In addition, we had the opportunity to assess the testing and manufacturing capabilities of Kyocera and how such capabilities can be integrated into our development effort on this program.

The visit began with tours of Kyocera's machine tool production plant in Sendai, Japan as well as their research and development facilities in Kagoshima, Japan. Kyocera's R&D facilities include tremendous materials characterization and testing capabilities at room and elevated temperatures, all of which will be made available to us for use on this program as part of Kyocera's in-kind program cost share contribution. The machine tool manufacturing line allows Kyocera to manufacture thousands of ceramic inserts per hour. Resources in both facilities are available for composite development work on this program.

On Wednesday and Thursday (April 11th and 12th) we met with Kyocera executives Director and General Manager of the Legal Affairs Group Minoru Fujiyoshi, Manager of the Corporate Development Group Michio Ito, Deputy General Manager of Cutting Tools Division Eiji Umegae, and Legal Counsel Takeshi Kawano. The Kyocera subcontract and the details of Kyocera's participation on this program were discussed and agreed upon during the two-day meeting. Kyocera has agreed to participate by evaluating new compositions of fibrous monoliths for wear resistant applications and developing and applying testing and characterization techniques for fibrous monolith components (see Attachment A for details). On Friday April 13th Kennametal's Vice President and Chief Technical Officer David B. Arnold joined discussions regarding potential 3-way collaborations between Kyocera, ACR Inc. and Kennametal. This collaboration would involve the utilization of Kennametal's Rapid Omni-Directional Compaction Process (ROC Process) in the production of FM-based cutting tools. Kyocera and ARC Inc are in the process of evaluating the potential of this process in the fabrication of wear resistant composite tooling. At this time ACR Inc is pursuing the ROC process as a potential production consolidation method for the composites being developed on this program, and are in the process of securing ROC process equipment time at Kennametal for composite development experiments.

EXPERIMENTAL

Task 3. Develop Compositions of Fibrous Monoliths

a. Conduct material trade studies to select best materials for evaluation

CORE MATERIAL TRADE STUDY

A set of material property data for potential wear resistant materials was gathered by program team members at Advanced Ceramics Research (ACR). These materials represented the initial candidates for developing fibrous monolith (FM) drill bits for use in the mining industry. A trade study matrix was constructed using the material properties considered most important for the drill bit application, specifically, hardness, toughness, thermal conductivity, and cost. Each of these properties was normalized to the maximum value contained in the data set. All properties were ranked between 0 and 1, where 1 was the most desirable value. Because the cost values are inversely proportional to their desirability, the normalized cost number was subtracted from 1 so that the ranking order was consistent with the other parameters. To obtain an overall score for each material, we summed the normalized parameters after applying a percentage-weighting factor for each parameter. This method allowed some flexibility to adjust the weighting factors according to the priority of the property. For example, if hardness was considered to more important than toughness, the weighting factor for hardness could be increased and the weighting factor for toughness decreased. For materials that had a range of properties, the maximum and minimum values were input to show a possible range of expected performance in the trade study. Those materials with a range of values reported in the literature are listed as individual maximum and minimum rows in the trade matrix.

Based on experience and discussions with mining industry members, the initial weighting factors were set to the following values:

Parameter	Weight
Hardness	35%
Toughness	25%
Thermal Conductivity	15%
Cost	25%

As expected, the top ranked material using these weighting factors was tungsten carbide, with diamond as the second ranked material. The fact that the most commonly used material, tungsten carbide, and diamond are so closely ranked may have been an indication that the importance of cost was underestimated in our initial determination of the mining industry's perception of parameter weighting factors. There was a significant gap in the material scores after tungsten carbide and diamond, indicating that these two materials were clearly superior to the others when using these weighting factors. The top ten materials are listed in the following table with their weighted score.

Rank	Material	Score
1	WC maximum	0.543
2	Diamond maximum	0.543
3	WC/Co 10.1% 2.84 micron	0.409
4	Diamond minimum	0.402
5	B ₄ C maximum	0.394
6	TiB ₂ maximum	0.383
7	TiC maximum	0.381
8	SiC alpha maximum	0.380
9	B ₄ C minimum	0.377
10	WC/Co 10.1% 0.98 micron	0.376

To study the influence of cost on the material selection decision, the weighting factors were adjusted by increasing the performance parameters and decreasing the importance of cost, and vice versa. The following table lists the parameters used to study cost sensitivity.

Parameter	Performance Sensitive	Balanced	Cost Sensitive
Hardness	40%	35%	30%
Toughness	30%	25%	20%
Thermal Conductivity	20%	15%	10%
Cost	10%	25%	40%

With maximum emphasis on the importance of cost (40%), tungsten carbide was clearly above the other materials as the obvious material choice. This was not surprising, since in the cost sensitive mining industry tungsten carbide is the most common wear resistant material in use today. As the importance of the performance parameters was increased, diamond became the obvious material choice. As a wear resistant material, diamond has found a niche in the mining and drilling marketplace. High end drilling operations that can afford larger investments required to access resources, such as oil and gas drilling, utilize diamond drill bits. It should be mentioned, however, that diamond is only utilized where the most demanding drilling environments necessitate a high performance bit.

Performance Sensitive		Balanced		Cost Sensitive		
Rank	Material	Score	Material	Score	Material	Score
1	Diamond maximum	0.651	WC maximum	0.543	WC maximum	0.625
2	Diamond minimum	0.490	Diamond maximum	0.543	B ₄ C maximum	0.504
3	WC maximum	0.460	WC/Co 10.1% 2.84 micron	0.409	WC/Co 10.1% 2.84 micron	0.498
4	TiB ₂ maximum	0.321	Diamond minimum	0.402	SiC alpha maximum	0.497
5	WC/Co 10.1% 2.84 micron	0.320	B ₄ C maximum	0.394	TiC maximum	0.495
6	B ₄ C maximum	0.285	TiB ₂ maximum	0.383	ZrO ₂ cubic maximum	0.491
7	WC/Co 10.1% 0.98 micron	0.280	TiC maximum	0.381	B ₄ C minimum	0.489
8	WC/Co 5.1%	0.277	SiC alpha maximum	0.380	Al ₂ O ₃ maximum	0.488
9	WC/Co 7.6%	0.275	B ₄ C minimum	0.377	WC minimum	0.485
10	TiC maximum	0.267	WC/Co 10.1% 0.98 micron	0.376	ZrO ₂ cubic minimum	0.480

One of the main benefits of the fibrous monolith composite structure is its increased toughness. Hardness is desired for wear resistance, but very hard materials tend to fail catastrophically. By using the FM composite structure ACR plans to increase toughness and improve the overall performance of the wear components. For this reason it was decided to use the trade study matrix to look at the relationship between hardness and toughness. As with the cost analysis, where we adjusted the importance of cost up and down, we varied the importance of hardness and toughness. Starting with an equal weighting of importance, the importance of hardness was increased while toughness was decreased and all other parameters were held constant. The weighting factors are listed in the following table.

Parameter	Balanced	Harder	Hardest
Hardness	30%	40%	50%
Toughness	30%	20%	10%
Thermal Conductivity	15%	15%	15%
Cost	25%	25%	25%

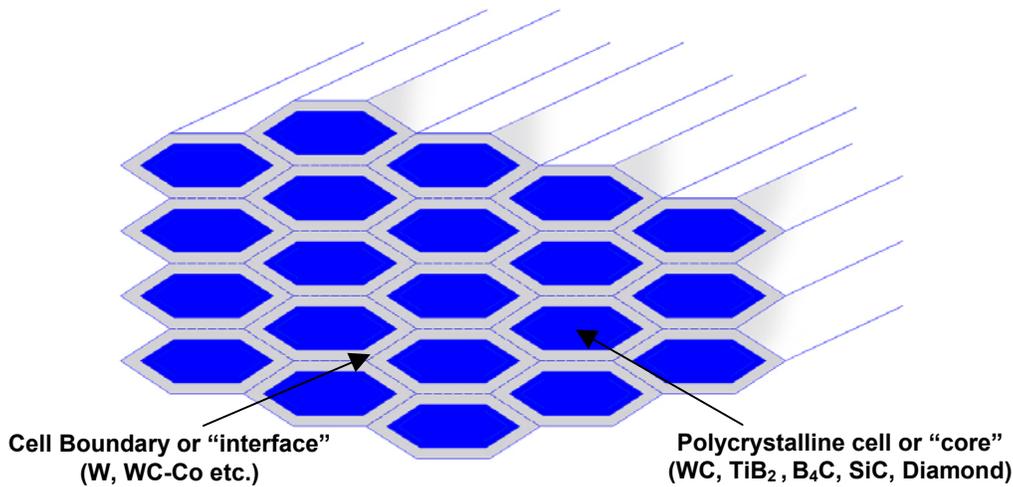
It is clear that as the hardness parameter becomes more important in the trade study, diamond moves to become the overwhelming choice. The mining industry's current choice, tungsten carbide, is a distant second when hardness is the most important factor. Boron carbide is another material with good scores in this hardness versus toughness trade. Even though boron carbide scores drop as the importance of hardness increases, it does not drop as rapidly as all the other materials in the study, thus holding its ranking at 4th in all three cases.

Balanced Harder Hardest						
Rank	Material	Score	Material	Score	Material	Score
1	WC maximum	0.585	Diamond maximum	0.584	Diamond maximum	0.667
2	Diamond maximum	0.501	WC maximum	0.500	Diamond minimum	0.466
3	WC/Co 10% 2.84 micron	0.438	Diamond minimum	0.423	WC maximum	0.415
4	B ₄ C maximum	0.399	B ₄ C maximum	0.390	B ₄ C maximum	0.381
5	WC/Co 10% 0.98 micron	0.396	TiC maximum	0.381	TiC maximum	0.380
6	ZrO ₂ cubic partially stabilized	0.392	WC/Co 10.1% 2.84 micron	0.380	SiC alpha maximum	0.375
7	WC/Co 5.1%	0.392	TiB ₂ maximum	0.378	TiB ₂ maximum	0.369
8	WC/Co 7.6%	0.390	SiC alpha maximum	0.378	B ₄ C minimum	0.357
9	TiB ₂ maximum	0.388	B ₄ C minimum	0.370	Cr ₂ O ₃ maximum	0.354
10	B ₄ C minimum	0.383	Al ₂ O ₃ maximum	0.362	Cr ₂ O ₃ minimum	0.353

After reviewing these results, the materials for this phase of the program were narrowed to 5 choices. ACR will work with at least three of these materials when attempting to develop FM systems based on these core materials. The five materials are:

- 1) Tungsten carbide
- 2) Boron carbide
- 3) Titanium diboride
- 4) Diamond
- 5) Silicon Carbide

Tungsten carbide is such a widely used material that ACR would want to use this material in early trails even if the material had not ranked high in the trade study. Since boron carbide was the next best material when cost sensitivity is concerned it should also be included in the early trails. Titanium diboride was one of the highest ranked materials in performance so it should be included. Diamond is typically a high cost material, but the overwhelming indications that expected performance would exceed all other materials dictates that it be included. Diamond has an added difficulty due to the high pressure processing requirements. In spite of this hurdle, ACR wants to keep this material on the initial list in the event that ACR gains access to diamond consolidation equipment through one of the program partners. Silicon carbide is included since the material scored well in the cost sensitivity trade. However, it may be a more appropriate material for apex cones in the next phase of the program. For this reason, silicon carbide is included in the list and will be tested in this phase if one of the other materials falls out.



In addition to the Fibrous Monolith potential core materials discussed earlier, work is underway to prepare an interface trade study to select appropriate interface materials to be used with the selected core materials. The material requirements include: high ultimate tensile strength, high ductility and/or toughness, coefficient of thermal expansion, thermal conductivity and compatibility of the interface's sintering/consolidation temperature with the selected core materials. Cost will be considered, although FM composites are not as cost sensitive to interface selection because the interface typically makes up a much smaller portion of the composite (10-20 volume %).

Because of the success of the diamond/WC-Co FM system in the oil and gas drill bit insert application, and the high rating of both the WC and diamond in the trade study, the first system selected for development in the mining drill bit insert is diamond/WC-Co. Diamond/WC-Co FM inserts have been fabricated and sent to Kennametal for evaluation, and the results of the testing are expected to be available in September (see 1st Quarterly Technical Report for details). Results will be included in the next quarterly progress report.

Task 3. Develop Compositions of fibrous monoliths

No work was performed on this task in the current quarter. Once the interface materials are selected we will formulate a test matrix and begin composite fabrication of FM systems using the core materials selected above.

Kennametal is expected to complete wear testing of the FM samples (fabricated and delivered under this task at the end of the first calendar quarter in September. We will report on the results of testing in the 3rd Quarterly Technical Progress Report.

PLANS FOR THE NEXT REPORTING PERIOD

1. Complete the FM trade study (Task 2a).
2. Complete composition development of Fibrous Monoliths for the drill bit application (Task 2b).
3. Complete the development of fabrication process parameters of Fibrous Monolith compositions selected for the drill bit insert application (Task 3a).
4. Continue densification process optimization of Fibrous Monolith compositions selected for the mining drill bit insert application (Task 4a).
5. Begin laboratory testing of FM compositions developed for drill bit insert application (Task 5)

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AMMENDED KYOCERA STATEMENT OF WORK

EXHIBIT B

ADVANCED CERAMICS
RESEARCH

STATEMENT OF WORK

FIBROUS MONOLITH WEAR RESISTANT
COMPONENTS FOR THE MINING INDUSTRY

ACR Confidential

STATEMENT OF WORK

FIBROUS MONOLITH WEAR RESISTANT COMPONENTS FOR THE MINING INDUSTRY

MINING INDUSTRY OF THE FUTURE CROSSCUTTING TECHNOLOGIES

PROGRAM SUMMARY

This program addresses the mining industry's need for improved components for wear resistance. The cost/performance ratio drives the application of components and materials used in mining applications. The mining industry traditionally had little use for advanced wear resistant materials due to their high cost relative to their improved durability. The goal of this program is to offer advanced wear resistant materials, in the form of fibrous monolith composites, that will overcome the cost/performance barrier traditionally associated with advanced materials and significantly increase the wear life of targeted components. Materials systems that exhibit promise as a crosscutting technology where resistance to wear is important will also be developed. Research will be performed on other applications, such as metal cutting tools, as crosscutting technologies are developed and translated into other industries.

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The Department of Energy's (DOE) Office of Industrial Technology (OIT) funds this program with considerable cost share from several of the industrial partners on the program. This statement of work defines the tasks to be performed by Kyocera Corporation.

PROGRAM OBJECTIVES

OBJECTIVE # 1

Meet the objectives defined in this statement of work.

OBJECTIVE #2

Evaluate new compositions of fibrous monoliths for wear resistant applications.

OBJECTIVE #3

Demonstrate improved testing and characterization techniques for fibrous monolith components.

PROGRAM TASKS

1. OBJECTIVE #1 - PROGRAM MANAGEMENT

- a. Prepare and deliver monthly status reports (objective #1)

Kyocera program management shall prepare monthly status reports to provide ACR information on Kyocera development efforts, technical results and cost summary as required by ACR. Kyocera shall provide the report in Kyocera's format. ACR will be responsible for preparing and submitting all reports to DOE.

- b. Travel (objective #1)

At discretion of Kyocera, Kyocera program management and technical staff will travel to support the program including, as necessary, monitoring laboratory and field tests, and participating in program meetings at ACR.

2. OBJECTIVE #2 - EVALUATION OF COMPOSITIONS OF FIBROUS MONOLITHS

- a. Analysis and evaluation of material properties at ACR's request (objective #2)

Kyocera technical staff will evaluate material candidates for high wear resistant fibrous monolith in mining applications. Material candidates will include a wide range of ceramic and metallic materials that demonstrate the desired properties. Trade studies will evaluate the materials based on the desired features for each of the potential applications, for example impact toughness, high-temperature capability, and compressive strength.

3. FABRICATION OF TEST SAMPLES

- a. Fabricate fibrous monolith samples (objective #3)

At the instruction of ACR regarding the manufacturing method or process, Kyocera technical staff will fabricate material samples for laboratory testing.

- b. Characterize fibrous monolith properties (objective #3)

At the direction of ACR, Kyocera technical staff will conduct material properties testing to characterize the selected candidate materials. Kyocera technical staff shall perform laboratory testing of fibrous monolith test samples, including but not limited to static properties, such as flexural strength, hardness, coefficient of thermal expansion, thermal conductivity, fracture toughness and wear tests.

- c. Prepare and deliver laboratory test report (objective #3)

Kyocera technical staff shall prepare and deliver to ACR a laboratory test report. The lab test report should include a description of the materials tested, and the results of testing. The lab test report shall be in Kyocera format.