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SEMI-ANNUAL REPORT #1

FIBROUS MONOLITH WEAR RESISTANT COMPONENTS FOR THE MINING INDUSTRY

1ST TECHNICAL SEMI-ANNUAL REPORT

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Fibrous Monolith Wear resistant components for the Mining Industry

ABSTRACT

Published mechanical and thermal properties data on a variety of materials was gathered, with focus on materials that have potential with respect to developing wear resistant and damage tolerant composite for mining industry applications. Preliminary core materials of interest include but are not limited to: Diamond, Tungsten Carbide and Cemented Tungsten Carbides, Carbides of Boron, Silicon, Titanium and Aluminum, Diboride of Titanium and Aluminum, Nitrides of Aluminum, Silicon, Titanium, and Boron, Aluminum Oxide, Tungsten, Titanium, Iron, Cobalt and Metal Alloys. Preliminary boundary materials of interest include but are not limited to: W metal, WC-Co, W-Co, WFeNi, and Mo metal and alloys.

Several FM test coupons were fabricated with various compositions using the above listed materials. These coupons were consolidated to varying degrees by uniaxial hot pressing, then cut and ground to expose the FM cell structure. One promising system, WC-Co core and WFeNi boundary, was consolidated to 97% of theoretical density, and demonstrates excellent hardness. Data on standard mechanical tests was gathered, and tests will begin on the consolidated test coupons during the upcoming reporting period.

The program statements of work for ACR Inc. and its subcontractors, as well as the final contract negotiations, were finalized during the current reporting period. The program start date was February 22nd, 2001. In addition to the current subcontractors, Kennametal Inc., a major manufacturer of cutting tools and wear resistant tooling for the mining industry, expressed considerable interest in ACR's Fibrous Monolith composites for both machine and mining applications. At the request of Kennametal, ARC Inc fabricated and delivered several Fibrous Monolith coupons and components for testing and evaluation in the mining and machine tool applications. Additional samples of Diamond/Tungsten Carbide-6%Cobalt Fibrous Monolith were fabricated and delivered for testing Kennametal's Rapid Omni-directional Consolidation (ROC) Process. A meeting was held with Kennametal Inc. September 27th, 2001. At this meeting, Kennametal expressed interest in working with ACR on three mining tool applications including roof bits, point attack tooling and drill bit inserts. In addition, there is considerable interest in developing FM composite machine cutting tools, which involves crosscutting technology. In addition to the discussions on business development, Kennametal reported on testing performed on ACR's FM composites the first quarter of this year.

ACR Inc. also visited Tribocor Inc. of Houston TX to discuss the possibility of teaming to consolidate diamond/WC-Co composite coatings. Diamond-based composites require special high pressure consolidation equipment and Tribocor has expressed an interest in providing diamond powder preparation and consolidation services, to enable the mass-production of a low cost diamond-based FM composite products including drill bit inserts and point attack tools. Tribocor has agreed to perform consolidation of diamond/WC-Co FM coated inserts to verify their consolidation process and produce test pieces that we can press into mining drill bits for field testing.

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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, which 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 into 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.

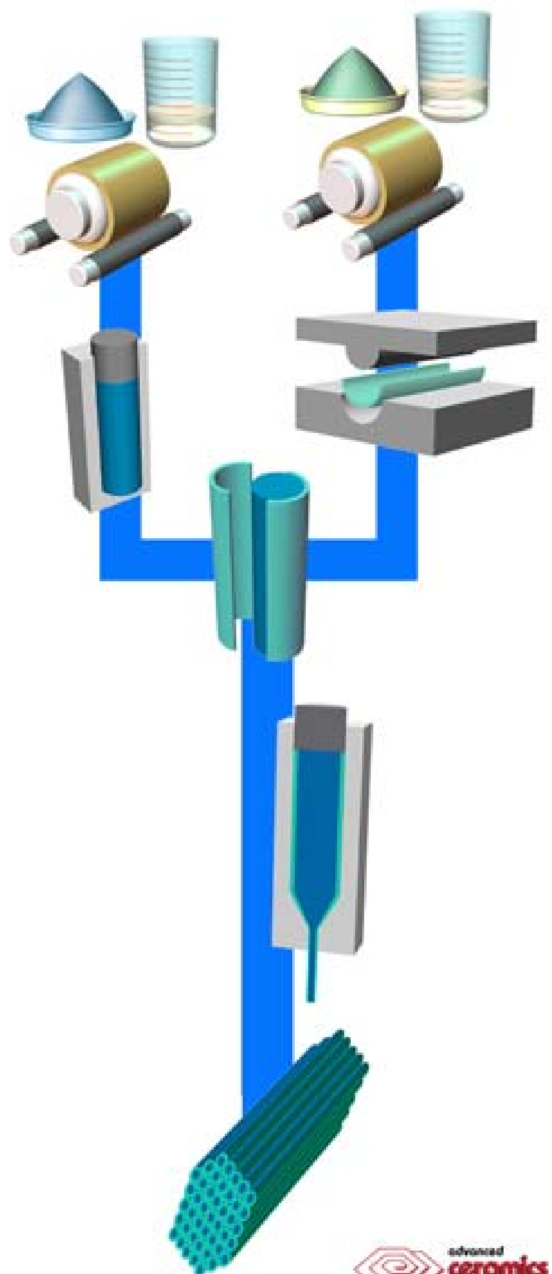


Figure 1. Illustration of the Fibrous Monolith co-extrusion process. Two ceramic and/or metal powders are blended separately with thermoplastics and plasticizers. The resulting mixtures are pressed into shells and rods. The shells and rods laminated to form a composite feedrod that is then placed in 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, Si_3N_4, SiC, ZrB_2, HfB_2, ZrO_2, or Al_2O_3, 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**).

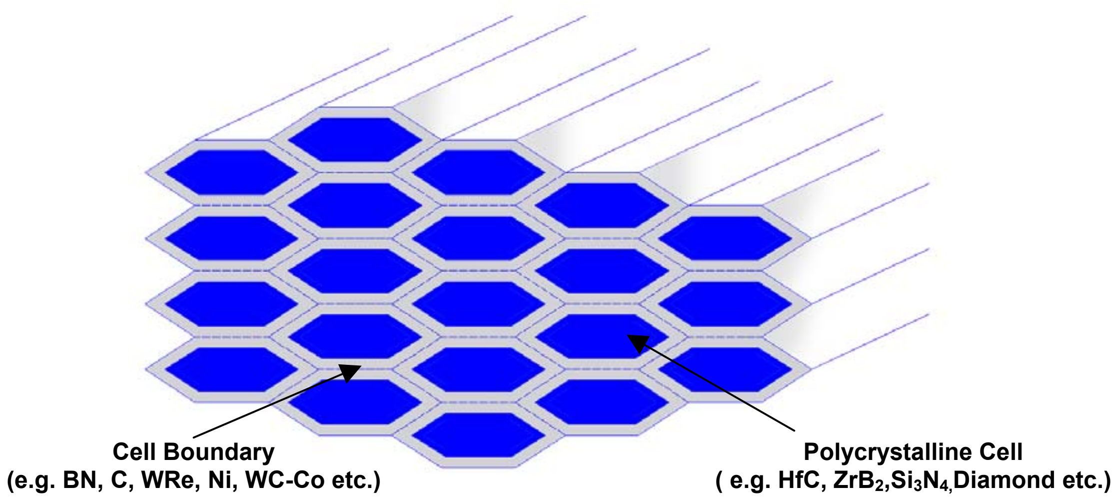


Figure 2. Schematic of a typical uniaxial Fibrous Monolith microstructure shown perpendicular to principal fiber direction.

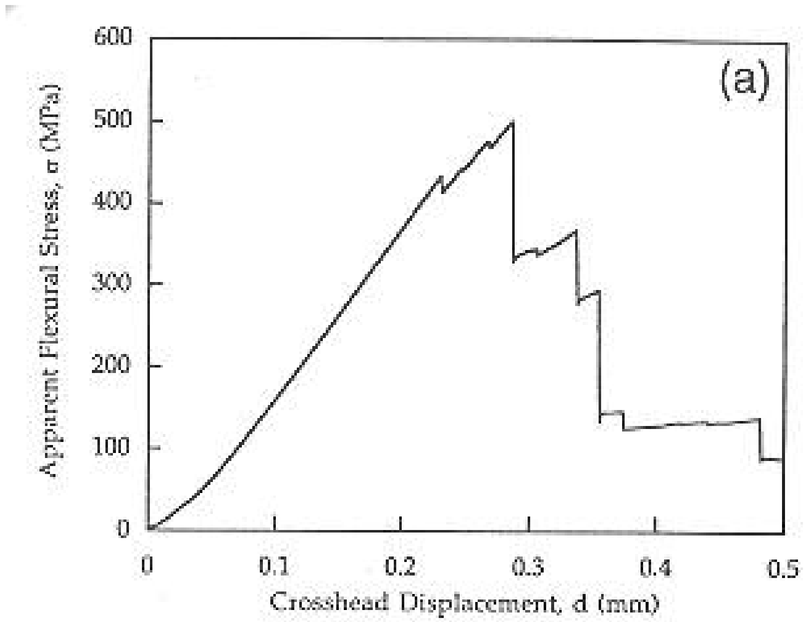


Figure 3. Typical flexural stress-strain curve for a silicon nitride/BN FM material.

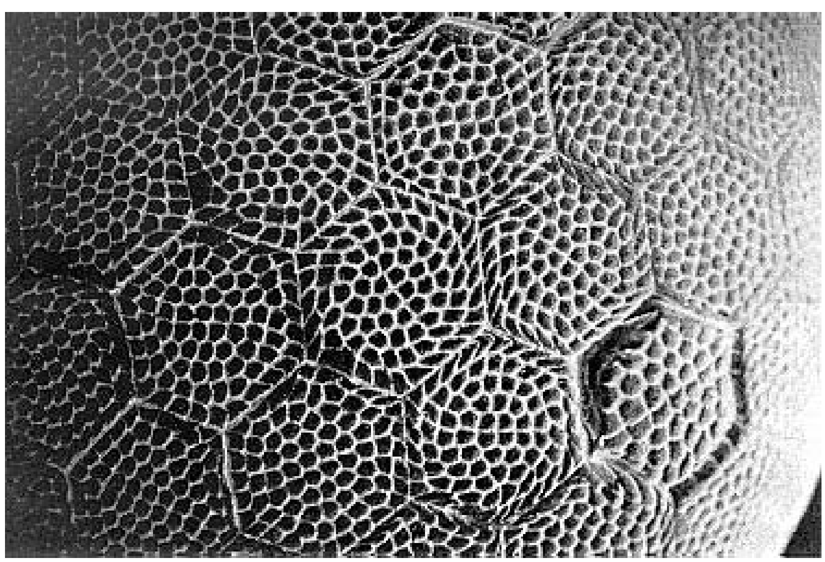


Figure 4. ACR's Diamond/ WC-Co FM composite applied as a coating on the surface of a WC drill bit insert (100x). Note the isolation of the darker material (Diamond) into discrete cells by the lighter contrast phase (WC-Co).

EXECUTIVE SUMMARY

During the first six months of this program, work was performed on finalizing the program statements of work for ACR Inc. and its subcontractors, as well as the final contract negotiations. The program start date was February 22nd, 2001. 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). Kennametals's Vice President and Chief Technica Officer 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.

In addition to 3-way collaborations with Kyocera, Kennametal Inc. has expressed considerable interest in our Fibrous Monolith composites for their own machine and mining tool applications. At the request of Kennametal, ARC Inc fabricated and delivered several Fibrous Monolith coupons and components for testing and evaluation in the mining and machine tool applications. Additional samples of Diamond/Tungsten Carbide-6%Cobalt Fibrous Monolith were fabricated and delivered for testing Kennametal's Rapid Omni-directional Consolidation (ROC) Process. ACR visited Kennametal on September 27th at which time they shared results of tests performed on our samples. ACR and Kennametal also began discussions regarding the co-development of FM cutting tool and mining tools including metal cutting inserts, conical point attack tool inserts, and drill bit inserts.

A set of materials property data for potential wear resistant materials was collected and analyzed. The materials of interest included but were not limited to: Diamond, Tungsten Carbide and Cemented Tungsten Carbides, Carbides of Boron, Silicon, Titanium and Aluminum, Diborides of Titanium and Aluminum, Nitrides of Aluminum, Silicon, Titanium, and Boron, Aluminum Oxide, Tungsten, Titanium, Iron, Cobalt and Metal Alloys. These materials are designated for use as 'core' and 'shell' materials in the Fibrous Monolith structure. The material properties of hardness, stiffness, tensile strength, transverse rupture strength, toughness, thermal conductivity, coefficient of thermal expansion 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, and titanium diboride. Work was also completed on the trade study to evaluate 'shell' materials. The selected shell materials include tungsten carbide-cobalt, tungsten-metal alloys, molybednium-metal alloys, and high-strength-steel alloys.

Efforts to develop, fabricate, and consolidate FM compositions utilizing the selected core and shell materials are well underway. Several FM systems including diamond/WC-Co, TiB_2/WC-Co, B_4C/WC-Co, and WC-Co/W-Ni-Fe now been fabricated and consolidated. Early results indicate that the diamond/WC-Co and WC-Co/W-Ni-Fe FM systems have excellent potential as effective wear-resistant coatings for the mining drill bit insert application.

In parallel with the composite development and fabrication effort, a test matrix for the evaluation of these composites for drill bit inserts application has also been compiled. The test matrix was developed with input from several sources including information provided by Kennametal, searches of the National Institute of Standards Technology ASTM literature database (www.astm.org), previous research performed in cooperation between ACR and Argonne National Laboratory and reviews of other drill bit manufacturer's datasheets.

PROGRAM MANAGEMENT

Meetings with Kennametal in Latrobe, PA, Kyocera in Sendai, Japan, Tribocor Technologies Inc. in Houston, TX and Dennis Tool, in Houston TX scheduled to take place in mid-September were postponed due to air travel restrictions resulting from the terrorist attacks on Washington D.C. and New York, NY on September 11th, 2001. All of these meetings were rescheduled for late September (Tribocor and Kennametal) and October (Kyocera, and Dennis Tool).

The meeting with Kennametal Inc. was rescheduled and took place on September 27th, 2001. ACR participants included Randy Cook, (Product Development Engineer), program PI Dr. Mark J. Rigali (Manager of Composite Ceramics), and Matthew Pobloske (Vice President of Marketing and Product Development). Kennametal participants included David B. Arnold (Vice President and Chief Technical Officer), Bernard North (Director of Materials and Process Development), John S. VanKirk (Director of analysis and Performance Technology Center), Ted R. Massa (Manager of Steel and Non-metalcutting Product Development), and Dr. Pankaj Mehrotra (Manager of Ceramic Technology). Kennametal expressed interest in working with ACR on three mining tool applications that include:

1. Roof Bits
2. Point Attack Tooling
3. Drill bit inserts

In addition, there is considerable interest in developing a cross-cutting technology: FM composite machine cutting tools. Kennametal has agreed to provide raw materials and guidance on consolidation to produce FM cutting tool prototypes. In exchange for this ACR will fabricate the prototypes for testing by Kennametal. This is a 5-billion-dollar/year market that represents a significant business opportunity for ACR's FM composite technology. ACR and Kennametal are now in the process of developing an agreement for collaborative research and development in all four of these business areas. This may include Kennametal's participation as a partner on the current DOE program. In addition to the discussions on business development, Kennametal briefed ACR on testing performed on the FM composites we provided to them in the first quarter of this year. A detailed discussion of the test results and conclusions is presented in the Experimental Section of this report.

ACR Inc. also visited Tribocor Inc. of Houston TX to discuss the possibility of teaming to consolidate diamond/WC-Co composite coatings. Diamond-based composites require special high pressure consolidation equipment and Tribocor has expressed an interest in providing diamond powder preparation and consolidation services, to enable the mass-production of a low cost diamond-based FM composite products including drill bit inserts and point attack tools. Tribocor has agreed to perform consolidation of diamond/WC-Co FM coated inserts to verify their consolidation process and produce test pieces that we can press into mining drill bits for field testing.

EXPERIMENTAL

Task 2. Develop Compositions of Fibrous Monoliths

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

CORE MATERIAL TRADE STUDY

ACR gathered published mechanical and thermal properties data on a variety of materials with potential application to developing wear resistant and damage tolerant composite for mining industry applications. The primary focus in this quarter was to gather data and compile a materials property database for the mining drill bit insert application. The gathered data will be used to select the best materials for Fibrous Monolith composite development for this application. Factors such as cost and FM processability will be considered in addition to material properties such as density, melting point, ultimate tensile strength, toughness, thermal conductivity and transverse rupture strength. Preliminary materials of interest include but are not limited to:

1. Diamond
2. Tungsten Carbide and Cemented Tungsten Carbides
3. Carbides of Boron, Silicon, Titanium and Aluminum
4. Diborides of Titanium and Aluminum
5. Nitrides of Aluminum, Silicon, Titanium, and Boron
6. Aluminum Oxide
7. Tungsten, Titanium, Iron, Cobalt and Metal Alloys

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. These properties were normalized to the maximum value contained in the data set for each of the materials of interest. 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_4C maximum	0.394
6	TiB_2 maximum	0.383
7	TiC maximum	0.381
8	SiC alpha maximum	0.380
9	B_4C 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 max.	0.651	WC max.	0.543	WC max.	0.625
2	Diamond min.	0.490	Diamond max.	0.543	B_4C max	0.504
3	WC max.	0.460	WC-10%Co 2.84 μm	0.409	WC-10%Co 2.84 μm	0.498
4	TiB_2 max.	0.321	Diamond min	0.402	α-SiC max.	0.497
5	WC-10%Co 2.84 μm	0.320	B_4C max.	0.394	TiC max.	0.495
6	B_4C max.	0.285	TiB_2 max.	0.383	ZrO_2 cubic max	0.491
7	WC-10%Co 0.98 μm	0.280	TiC maximum	0.381	B_4C min.	0.489
8	WC/Co 5.1%	0.277	α-SiC max.	0.380	Al_2O_3 max.	0.488
9	WC/Co 7.6%	0.275	B_4C min.	0.377	WC min.	0.485
10	TiC max	0.267	WC-10%Co 0.98 μm	0.376	ZrO_2 cubic min.	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%**

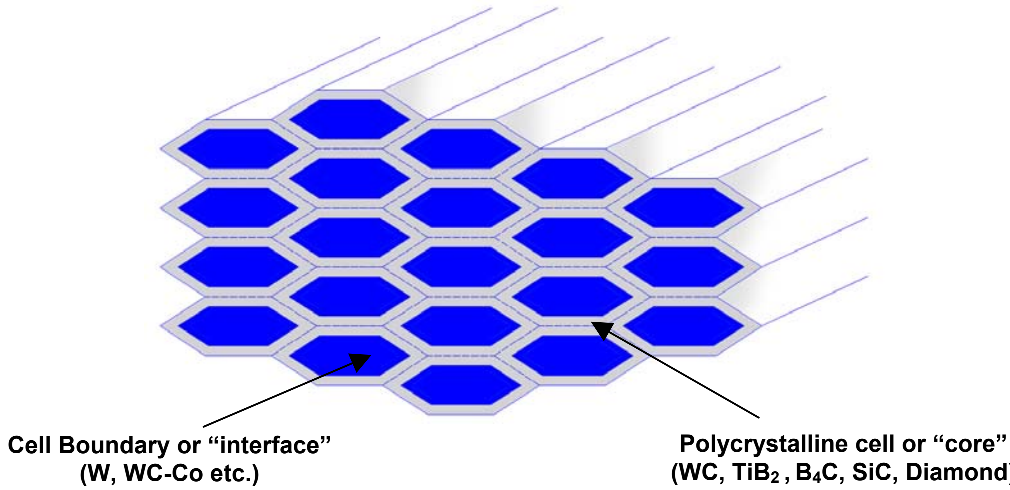
It is clear that as the hardness parameter becomes more important in the trade study, diamond moves to become the overwhelming choice. The mining industry's current choice, tungsten carbide, is a distant second when hardness is the most important factor. Boron carbide is another material with good scores 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 it's ranking at 4th in all three cases.

	Balanced		Harder		Hardest	
Rank	Material	Score	Material	Score	Material	Score
1	WC max.	0.585	Diamond max.	0.584	Diamond max.	0.667
2	Diamond max	0.501	WC max.	0.500	Diamond min.	0.466
3	WC-10%Co 2.8μm	0.438	Diamond min	0.423	WC max.	0.415
4	B_4C max.	0.399	B_4C max.	0.390	B_4C max.	0.381
5	WC-10%Co0.98 μm	0.396	TiC max.	0.381	TiC max.	0.380
6	Cubic ZrO_2	0.392	WC-10%Co2.8μm	0.380	α-SiC max.	0.375
7	WC/Co 5.1%	0.392	TiB_2 max.	0.378	TiB_2 max.	0.369
8	WC/Co 7.6%	0.390	α-SiC max.	0.378	B_4C min.	0.357
9	TiB_2 max.	0.388	B_4C min.	0.370	Cr_2O_3 max.	0.354
10	B_4C min.	0.383	Al_2O_3 max.	0.362	Cr_2O_3 min.	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.



The interface trade study to select appropriate materials to be used with the selected core materials has also been completed. The most important material requirements for the interface include: high ultimate tensile strength and high thermal conductivity. Hardness and elastic modulus are also factored in but considered less important for the FM interface. Hard materials were selected for the core and the purpose of the interface is to provide the toughness and tensile strength that the hard, high modulus core materials inherently lack. For drill bit inserts, the friction of drilling through rock in most mining applications generates a considerable amount of heat that must be dissipated and conducted away from the insert tips, hence a high thermal conductivity in the interface is also desired especially in light of the relatively low thermal conductivity of two of the core materials (B_4C and TiB_2). Compatibility of the interface's sintering/consolidation temperature with the selected core materials is also required. In addition, a relatively small difference between the coefficient of thermal expansion (CTE) between the core and interface is also desired to minimize the creation of residual stress in the part after densification at elevated temperatures. Cost was also 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 %). As with the core material trade study the thermal and mechanical properties of potential interface materials were normalized to the maximum values within each category and ranked between zero and one, where one is the highest and most desirable value. The initial weighing factors were set with a heavy weighting towards ultimate tensile strength and thermal conductivity as well as a balanced set of weighing factors shown below.

Parameter	High UTS, K	Balanced
Hardness	**10%**	**20%**
Elastic Modulus	**10%**	**20%**
Ultimate Tensile Strength, UTS	**40%**	**30%**
Thermal Conductivity, K	**40%**	**30%**

Rank	High UTS, K	Score	Rank	Balanced	Score
1	W metal max.	0.84	1	WC-Co alloy max.	0.73
2	WC-Co max.	0.69	2	W metal max.	0.71
3	W-Co alloy max.	0.68	3	W-Co alloy min.	0.59
4	W metal min.	0.58	4	W metal min.	0.54
5	W-Ni-Fe alloy max.	0.54	5	WC-Co max.	0.51
6	Ti-Zr-Mo alloy max.	0.53	6	W-Ni-Fe alloy max.	0.49
7	Ti-Zr-Mo alloy min.	0.51	7	Ti-Zr-Mo alloy max.	0.47
8	Fe-Ni-Co alloy (Aermet)	0.51	8	Ti-Zr-Mo alloy min.	0.46
9	Mo metal	0.44	9	Fe-Ni-Co alloy	0.44
10	WC-Co min.	0.43	10	Mo metal	0.40

As can be seen from the interface material rankings, tungsten based materials (W metal, its alloys and WC-Co) achieved the highest scores followed by molybdenum alloys (Ti-Zr-Mo alloys**)** and high tensile strength steels such as Aermet. Our initial development effort will focus on fabricating TiB_2, B_4C, WC and Diamond based FMs with W and WC-Co interfaces. Because B_4C requires a relatively high consolidation temperature (1900 –2100 C), W alloys and WC-Co may not be appropriate interface materials. Pure W metal, which sinters at similar temperatures, may be the only interface material choice, of those materials listed above, for combination with B_4C.

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, we will also explore this system for development in the mining drill bit insert is diamond/WC-Co. As we noted in the Quarterly reports (1^{st} and 2^{nd}) Diamond/WC-Co FM inserts have already been fabricated and sent to Kennametal for evaluation. Kennametal reported the results of their testing to ACR on September 27^{th} during a visit to the Kennametal Corporate Research and Development Facilities in Latrobe, PA.

Task 2. Develop Compositions of fibrous monoliths

b. Composition development

Work performed under Task **2b** during the first six months on the this program initially focused on finalizing the program statements of work for ACR Inc. and its subcontractors as well as the final contract negotiations. In addition to its current subcontractors, Kennametal Inc., a major manufacturer of cutting tools and wear resistant tooling for the mining industry, expressed considerable interest in ACR Inc's Fibrous Monolith composites for both machine tools such as end-mill bits as well as mining "point attack tools" used on long-wall mining equipment.

At the request of Kennametal, ARC Inc fabricated a set of Fibrous Monolith coupons and components for testing and evaluation in the mining and machine tool applications (**Figure**

1). Kennametal requested 7 to 10 0.5" x 0.5" x 0.2" thick squares of each of the following systems:

1. Silicon Nitride/Boron Nitride (Si_3N_4/BN)
2. Zirconium Diboride/Boron Nitride (ZrB_2/BN)
3. Hafnium Carbide/Tungsten-3.6%Rhenium alloy (HfC/WRe)
4. Titanium Diboride/Alumina (TiB_2/Al_2O_3)

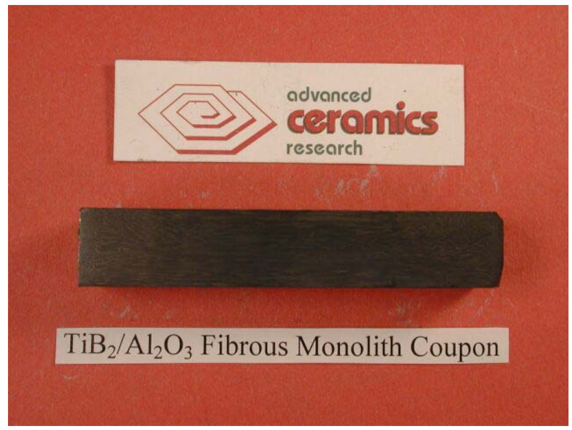


Figure 1. 3" x 0.5 x 0.5" ZrB2/BN (left) and TiB2/Al2O3 Fibrous Monolith coupons fabricated for testing and evaluation by Kennametal. The photo at left shows a 0.25" thick slice cut from the FM coupon.

The initial test results on the coupons provided to Kennametal for the metal machine tool application were disappointing. The FM insert samples all chipped during preparation for testing and as a result the Kennametal engineers were unable to produce any testable inserts from any of the samples sent by ACR. Despite this initial setback, both Bernard North (Director of Materials and Process Development) and Dr. Pankaj Mehrotra (Manager of Ceramic Technology) remain very interested in the FM technology. The principal concern expressed was that the BN and WRe interfaces may be too brittle for metal cutting tools. After a long discussion about suitable materials, for machine tool applications, Kennametal agreed to send us samples of the Al_2O_3 and TiCN powders currently used in the fabrication of their Al_2O_3 –TiCN composite metal cutting tool. We will use their powder to fabricate Al_2O_3/TiCN FMs for direct comparison to their current Al_2O_3 –TiCN product. We expect to receive the powders from Kennametal within the next several weeks and if we are successful in producing an improved metal cutting tool insert using FM technology, it will open a large business opportunity for ACR in a "cross-cutting" industry.

In addition to the test coupons, Kennametal expressed considerable interest in the Diamond/Tungsten Carbide-Cobalt FM system originally developed for Smith International Inc. (SII) as a coating for oil and gas drill bit inserts. These FM coatings were originally developed with Smith International Inc. (SII) on a DOE funded program [5] to produce toughened coatings for drill bit inserts used in oil and gas exploration. With the cooperation of SII, one dozen 3/8-inch WC drill bit inserts coated with Diamond/Tungsten Carbide-6%Cobalt Fibrous Monolith (Figure 2) were fabricated and delivered to Kennametal in late February. The drill bit inserts were fabricated using SII's high-pressure consolidation techniques and delivered to Kennametal for wear testing and comparison to their standard WC-based point attack tool inserts.

ACR Inc. was also asked to provide an additional one dozen unconsolidated Diamond/WC-Co coated inserts for consolidation using Kennametal's Rapid Omni-directional Consolidation (ROC) Process. The ROC process is a hot isostatic press adapted to rapidly consolidate large numbers WC metal-cutting and mining-tool inserts. Kennametal intends to compare the wear performance of the diamond/WC-Co FM inserts consolidated by SII to those consolidated using their ROC process. Successful consolidation using the ROC process will mean that densification of diamond/WC-Co system is amenable to the large volume production process currently used in the mining industry by Kennametal for the fabrication of WC-based point attack tools.

The Diamond/WC-Co samples were consolidated by the ROC process onto WC inserts in mid-March. Preliminary observations indicate that the ROC process partially densified the Diamond/WC-Co coating but the coating surface was irregular, having a "wavy" or crenulated appearance. Full densification of the diamond-based FM system using the ROC process for a mining application appears unlikely at this time because the ROC process cannot reach the pressures necessary to promote the diamond to diamond bonding required to form wear and damage tolerant coating. For this reason we will focus our efforts in high-pressure consolidation and develop partnerships with companies with such capabilities including Smith Tool, Dennis Tool, Tribocor Inc. and Phoenix Crystal.

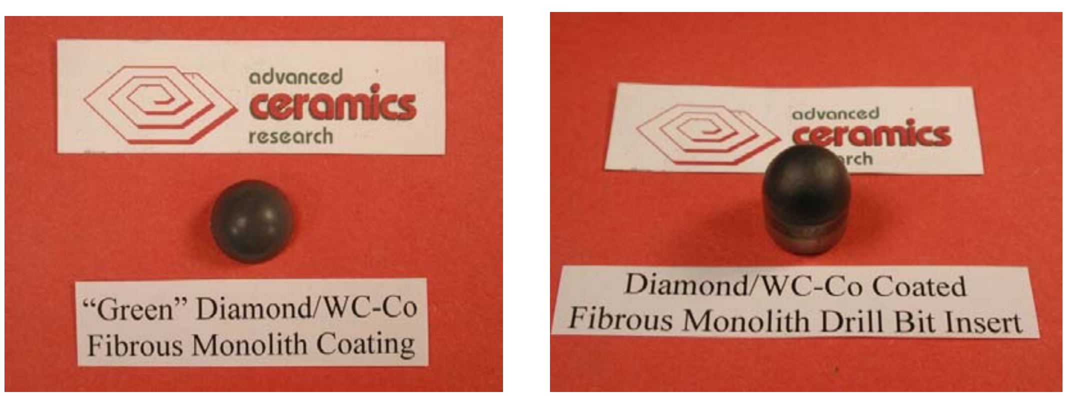


Figure 2. Diamond/WC-Co Fibrous Monolith coating (left) and a consolidated Diamond WC-Co coating on a 3/8 WC oil drill bit insert.

Kennametal has completed the initial testing of the hemispherical domed 7/16 inch diameter cellular diamond/WC-Co FM coated inserts in a conical tool application using their drum tool tester. In addition to the FM insert, Kennametal ran a standard Kennametal K3560 tool in the drum tool tester for comparison. The FM and K3560 inserts were run against hard sandstone at 90 rpm (990 feet per minute tip speed.) Approximately 230 linear feet of rock were cut at a traverse rate of 5.0 surface feet per minute and a 0.20 inch depth-of-cut. The test was halted when the standard Kennametal K3560 tool began to show excessive wear and steel wash. Kennametal estimates that a total of approximately 4140 'impacts' per tool were experienced during the test. The K3560 tip, measured by volume of material removed, was approximately 60-70 percent worn. At the end of the test, the Diamond/WC-Co FM coated button tips were visibly worn, with a wear band extending around the flank of the dome. One of the tips was EDM cross-sectioned and the wear was measured under a microscope. The wear varied across the dome cross-section surface, as expected based on the cutting action in the test. At the location of maximum wear, ~ 0.014 inch of the 0.020 inch original cellular thickness was removed. On the opposing side of the cross section, the wear was approximately 0.008-0.010 inch. Based on this very preliminary result, it appears that the Diamond/WC-Co FM material provides a performance advantage of approximately 2-4 times the conventional K3560 in abrasive wear testing against hard sandstone. This 2-4x factor is below that seen with similar testing on conventional PCD's, however it appears that the cellular PCD probably has better fracture toughness and resistance to cracking than traditional PCD.

A second test was run at 56 rpm with a 5ft/min traverse and a cutting depth of 0.2 inch for 853 linear feet over the same hard sandstone. Accounting for irregularities in the rock, this test produced approximately 7600 'impacts' per tool. At these lower rpm, the Diamond/WC-Co FM coated button showed much less wear as compared to the FM coated button run at 90 rpm. From the minimal amount of material lost during testing, it is estimated that a performance advantage of at least 10x times over the conventional K3560 tool was achieved.

Examination of the sectioned surfaces both of the diamond-WC-Co inserts by optical microscopy revealed noticeable differences in the orientation and uniformity of the cellular structure between the tips from the first and second tests. The tip from the second test at 56 rpm had a very uniform cellular structure, whereas the first tool displayed a very irregular WC-Co cell boundary that appeared to be missing in the section of the tool adjacent to the wear surface. These irregularities were likely caused by inconsistencies during co-extrusion of the FM filament in green processing and may be responsible for the poor performance of the button at high rpm. Kennametal is in the process of repeating the tests at low and high rpm using carefully selected inserts that have been examined before testing to insure that they exhibit a regular FM structure. Overall, Kennametal is impressed with the performance of the FM inserts in these preliminary tests and has expressed considerable interest in the continued development of these coatings for drill bit inserts, point attack tools and roof bits.

Task 3. Develop Compositions of fibrous monoliths

a. Fabrication Process Development

Upon completion of the trade study, the selected materials were obtained and suitable thermoplastic-ceramic blends were developed for fibrous monolith co-extrusion (see **Figure 1** for a pictorial representation of this process). This development process is iterative and often 5-10 attempts are required to achieve an extrudable blend for a given thermoplastic-ceramic FM system. The process involves blending the individual ceramic powders with thermoplastic melt-spinnable polymer binders and plasticizers, using a high shear mixer, to form a smooth, uniformly suspended mixture. Since the mixers we use have fixed volume reservoirs, the recipes devised to produce batches of the thermoplastic/ceramic blends are formulated on a volumetric, as opposed to a mass, basis. A typical blend consists of 50 to 62 vol. % of the ceramic powder, 37 to 50 vol. % of the thermoplastics, and 0 to 12 vol. % of the plasticizers. The core and interface of a fibrous monolith composite are blended separately and pressed into feedrod and shells respectively, then warm-laminated to form a green composite feedrod. We can produce 'green' composite feedrods with the following core/shell volume ratios: 90/10, 82.5/17.5, 80/20, 70/30, 60/40 and 50/50. The composite feedrods are extruded to form a coaxial FM filament using ACR's computer numerically controlled (CNC) ball-screw extruder. By producing thermoplastic/ceramic blends of the cores and shells with the appropriate rheological properties, the extruded filaments maintain the core/shell volume ratio of the original feedrod. The filaments are then wound, woven or laid-up in the desired architecture, warm laminated and then delubed in a burnout furnace to remove the polymers while maintaining the unique FM architecture. After binder burnout the samples are consolidated using pressureless-sintering, hot-isostatic-pressing or uniaxial-hot-pressing to form the final dense component.

For the initial development work on this program, the following formulations have been developed as "working recipes" for boron carbide, titanium diboride, tungsten carbide, and a W-Ni-Fe alloy (**Tables 1-4**).

Previous work by ACR Inc. and The University of Michigan [5] has shown that a core/shell volume ratio of 82.5/17.5 co-extruded to 2 mm provides the best combination of composite strength and toughness in applications where wear resistance is required. For this reason we have selected these parameters to begin our development efforts on this program. Co-extrusion of the FM systems (**Table 5**) was performed 2 mm filament was cut into 3" lengths and then laid-up uniaxially in a 1" x 3" die to form a ~0.5 inch thick 1" x 3" FM coupons. These coupons are placed in a binder burnout furnace to remove the polymers and then densified by hot pressing. The establishment of optimal consolidation conditions by hot pressing, hot isostatic pressing, or pressureless sintering involves the systematic variation of temperature and pressure, as well as the possible addition of sintering aids (**Task 4**), and is described in detail below.

Table 1. Co-extrudable Boron Carbide-Polymer 'Core' recipes.

Working B_4C Brabender Recipes		
Type	**B_4C 'Core' material**	
Recipe No.	ECT-01	
Material	**Density (g/cc)**	**Volume %**
B_4C	2.520	50.00
Polymers	1.000	40.00
Plasticizer	1.000	10.00
		100.00

Table 2. Co-extrudable Titanium Diboride 'Core' recipes.

Working TiB_2 Brabender Recipes		
Type	**TiB_2 'Core' material**	
Recipe No.	ECT-02	
Material	**Density (g/cc)**	**Volume %**
TiB_2	4.520	55.00
Polymers	1.000	40.00
Plasticizer	1.000	5.00
		100.00
Type	**TiB_2 + (10% Ni Sintering Aid) 'Core' material**	
Recipe No.	ECT - 05	
Material	**Density (g/cc)**	**Volume %**
TiB_2 + 10vol% Ni	4.918	55.00
Polymers	1.000	36.00
Plasticizer	1.000	9.00
		100.00
Type	**TiB_2 + (5% Al_2O_3 Sintering Aid) 'Core' material**	
Recipe No.	ECT - 05	
Material	**Density (g/cc)**	**Volume %**
TiB_2	4.520	50.00
Al_2O_3	3.990	5.00
Polymers	0.930	37.00
Plasticizer	1.100	8.00
	2.892	100.00

Table 3. Co-extrudable Tungsten Carbide-Polymer 'Core' and 'Shell' recipes.

Working WC Brabender Recipes		
Type	**WC-3%Co 'Core' material**	
Recipe No.	ECT-06	
Material	**Density (g/cc)**	**Volume %**
WC-3%Co	14.950	50.00
Polymers	1.000	42.00
Plasticizer	1.000	8.00
		100.00
Type	**WC-6%Co 'Shell' material**	
Recipe No.	ECT-07	
Material	**Density (g/cc)**	**Volume %**
WC-6%Co	14.960	50.00
Polymers	1.000	45.00
Plasticizer	1.000	5.00
		100.00

Table 4. Co-extrudable Tungsten-Nickel-Iron alloy 'Shell' recipe.

Working W-Ni-Fe Brabender Recipes		
Type	**W-Ni-Fe 'Shell' material**	
Recipe No.	ECT-01	
Material	**Density (g/cc)**	**Volume %**
W-Ni-Fe	15.42	54.00
Polymers	1.000	34.00
Plasticizer	1.000	12.00
		100.00

Task 4. Densification Process Development

a. Densification Process Optimization

Sample fabrication during the reporting period included several 1" x 3" uniaxial Fibrous Monolith test coupons of various compositions. These test coupons are characterized in **Table 5** and illustrated in the photographs in **Figure 4**.

Table 5. FM Samples Fabricated and Consolidated During the Reporting Period

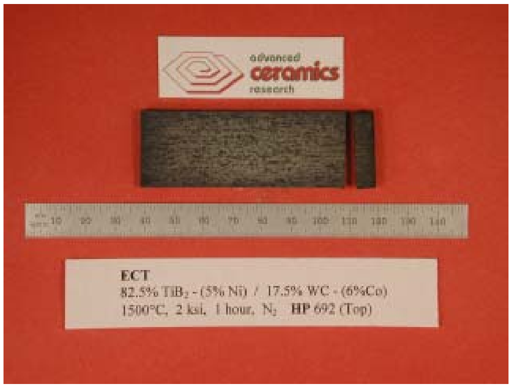
FM Composition 82.5% core / 17.5% shell (volume/volume)	Temperature (°C)*	Pressure (PSI)	Measured Bulk Density (g/cc)	Theoretical Density (g/cc)	% Full Theoretical Density
TiB_2 / WC-6%Co	1500	2000	4.348	6.080	71.5
B_4C / W-Ni-Fe	1500	2000	3.685	4.535	81.3
TiB_2-10%Ni / WC-6%Co	1500	2000	4.155	6.541	63.5
TiB_2-5%Al_2O_3 / WC-6%Co	1500	2000	4.803	6.184	77.7
WC-6%Co / W-Ni-Fe	1550	2000	14.562	15.032	96.9
TiB_2 / WC-6%Co	1550	2000	4.817	6.541	73.6
TiB_2-5%Al_2O_3 / WC-6%Co	1600	2000	4.386	6.168	71.1
TiB_2-10%Ni / WC-6%Co	1600	2000	3.984	6.541	60.9

*All samples were hot-pressed for 1 hour at the temperatures denoted.

Figure 4. FM Test Coupons Fabricated During the Reporting Period

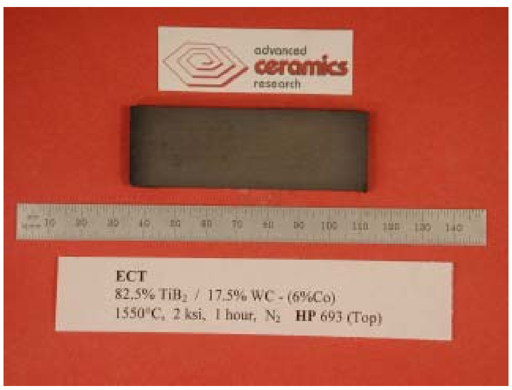
82.5% B_4C / 17.5% W-Ni-Fe

82.5% TiB_2 / 17.5% WC-6%Co



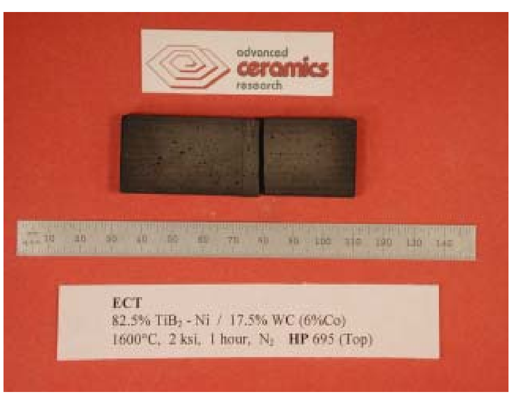
82.5% TiB_2-5%Ni /17.5% WC-6%Co

82.5% TiB_2-5%Al_2O_3/17.5% WC-6%Co



82.5% TiB_2 / 17.5% WC-6%Co

82.5% WC-6%Co / 17.5% W-Ni-Fe



82.5% TiB_2-Ni / 17.5% WC-Co (6%)

82.5% TiB_2-Al_2O_3 / 17.5% WC-Co

Physical characterization of these hot pressed FM composites was performed using optical microscopy and Archimedes density measurements (as reported in **Table 5** above). The physical appearance of the fabricated billets varied widely due to the constituent materials and range of firing temperatures. Several test coupons were only partially consolidated, and demonstrated friable structures that were easily broken by hand. Other test coupons were dense, possessing metallic luster and a visually observable intact cell structure. The sample with the highest measured theoretical density is the WC-6%Co / W-Ni-Fe FM sample hot pressed at 1550°C. Because of its hardness, the sample proved to be extremely difficult to polish in preparation for microscopic observations. However, a photomicrograph was obtained, after a considerable polishing effort, showing the presence of a distinct FM cellular structure. The high density, high apparent hardness, and presence of a distinct FM structure make the WC-6%Co / W-Ni-Fe system very promising for mining wear applications, especially as coatings for WC drill bit inserts.

Examination of the other systems listed above revealed that they all have well defined FM structures. However, the presence of voids and pores is indicated by their low measured densities, between 60 and 80%, as well as the observation of pores and voids in the samples during optical microscopy. Efforts to obtain fully dense TiB_2 and B_4C-based FM systems by refining the consolidation pressure, temperature and sintering aids will continue as these systems also have considerable promise in improving the wear-life of a variety of mining components.

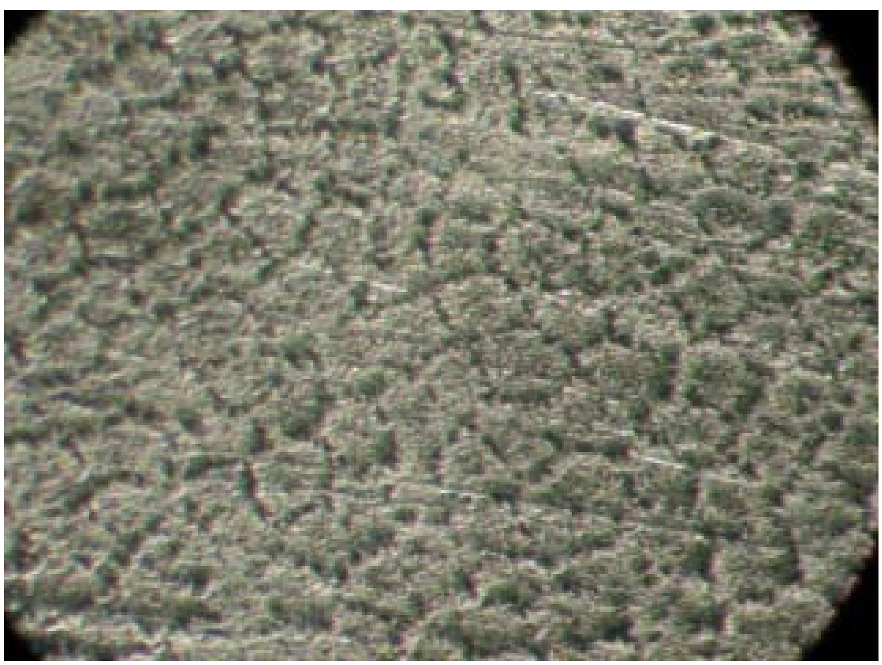


Figure 5. Photomicrograph of WC-6%Co / W-Ni-Fe FM system consolidated at 1550 C. The hexagonal WC-6%C cells (gray) are isolated by thin Wi-Ni-Fe boundaries. Note that the rough polish of the sample partially obscures the FM structure.

Task 5. Fabrication of Test Samples

a. Fabricate Fibrous Monolith Samples

ACR Inc. has compiled a preliminary set of tests, listed below, for mechanical and wear property evaluation for the composites we are developing on this program. Starting with a search of the database available at the ASTM website (www.astm.org) for ceramic wear-related testing documents specific to the testing of Advanced Ceramics, a list of candidate ASTM standards was generated. These standards were reviewed to determine applicability for the FM composite materials being developed on this program. In addition, the testing requirements of Kennametal and Smith Industries were reviewed to determine if the testing performed by our mining industry customers is applicable. The list below includes testing requirements from both industrial and ASTM standards.

Transverse rupture strength

- Three point load similar to ASTM standard B 406-96
- Requires 5 specimens.

Modulus of elasticity

- Resonance method similar to ASTM standard C 1198-96
- Mass of at least 5 grams required.

Compressive strength

- Determined by pressing a right circular cylinder sample between two tungsten carbide blocks held in alignment by an outer sleeve assembly

Relative impact resistance

- Determined by dropping a standard weight on the free end of a test specimen held in a cantilever beam fashion. The height of fall at which the specimen breaks is recorded.

Endurance limit

- Established using the rotating beam method.

Dry abrasion resistance

- Dry sand / rubber wheel abrasion resistance similar to ASTM G65-00e1.

Tensile strength

- Thin ring specimens 2 inches O.D. X 1.9 inches I.D. X .5 inches are subjected to internal hydraulic pressure.
- Tensile strength is typically 45 to 50 % of the transverse rupture strength

PLANS FOR THE NEXT REPORTING PERIOD

1. Complete composition development of Fibrous Monoliths for the drill bit application (Task 2b).

2. Complete the development of fabrication process parameters of Fibrous Monolith compositions selected for the drill bit inset application (Task 3a).

3. Continue densification process optimization of Fibrous Monolith compositions selected for the mining drill bit insert application (Task 4a).

4. Analyze preliminary drill bit design using material properties (Task 6a).

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4. G.E. Hilmas, A. Brady and J.W. Halloran, "SiC and Si_3N_4 Fibrous Monoliths: Non-Brittle Fracture From Powder Processed Ceramics Produced by Coextrusion,"; pp. 609-614 in Ceramic Transactions-Vol. 51 *Ceramic Processing Science and Technology*. Edited by H. Hausner and G. Messing, American Ceramic Society Westerville, OH, 1995.
5. "Development of Advanced Fibrous Monoliths," Advanced Materials Partnership program, a DARPA funded, DOE managed program, Cooperative Agreement No. DE-FC02-96CH10861 between DARPA and ACR.

ATTACHMENT A

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, which 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.

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.