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SEMI-ANNUAL REPORT #4

FIBROUS MONOLITH WEAR RESISTANT COMPONENTS FOR THE MINING INDUSTRY

4TH TECHNICAL SEMI-ANNUAL REPORT

Reporting Period: 8/23/02 to 2/22/03

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ABSTRACT

During the reporting period, work continued on development of formulations using the materials down-selected from the initially identified contenders for the fibrous monolith wear resistant components. In the previous reporting period, a two-stage binder removal process was developed that resulted in prototype parts free of voids and other internal defects. During the current reporting period, work was performed to characterize the two-stage binder removal process for WC-Co based FM material systems. Use of this process has resulted in the fabrication of defect free sintered WC-Co FM bodies, with minimal free carbon porosity and densities approaching 100% theoretical. With the elimination of free carbon porosity and other binder removal process related defects, development work focused on optimizing the densification and eliminating defects observed in WC-Co based FM consolidated by pressureless sintering. Shrinkage of the monolithic core and shell materials used in the WC-Co based FM system was measured, and differences in material shrinkage were identified as a potential cause of cell boundary cracking observed in sintered parts. Re-formulation of material blends for this system was begun, with the goal of eliminating mechanical stresses during sintering by matching the volumetric shrinkage of the core and shell materials. Thirty-three 7/8" drill bit inserts (WC-Co(6%)/WC-Co(16%) FM were hot pressed during the reporting period. Six of these inserts were delivered for field-testing by Superior Rock Bit during the upcoming reporting period. In addition, Al_2O_3/Al_2O_3-TiCN FM cutting tool inserts were fabricated, and cutting tests performed.

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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.

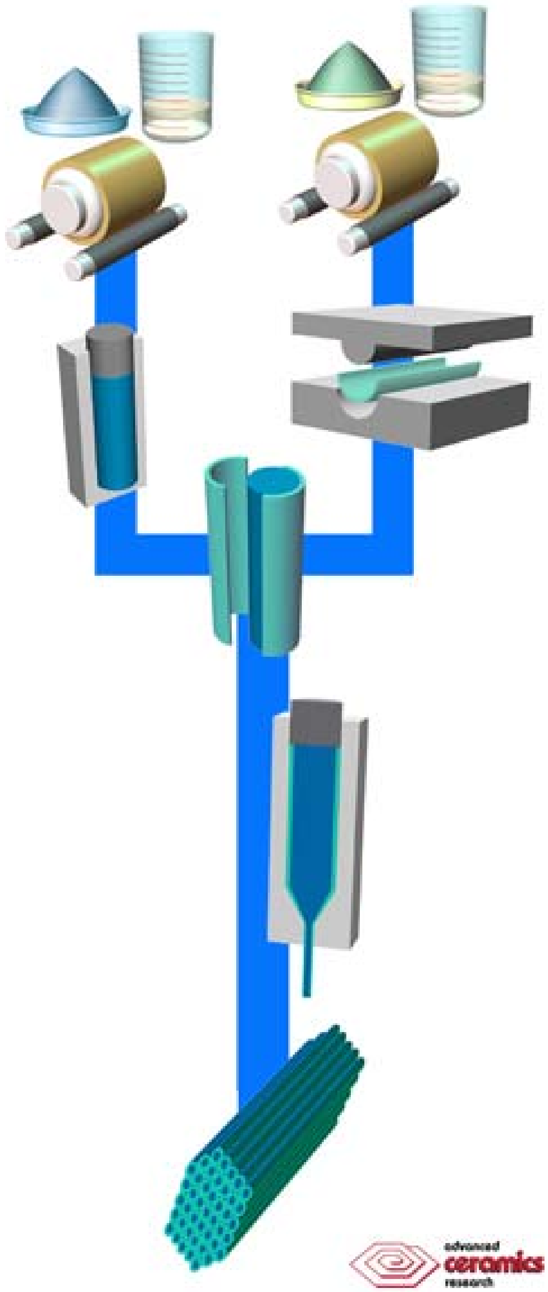

Figure 1. Illustration of the Fibrous Monolith co-extrusion process. Ceramic and/or metal powders are blended separately with thermoplastics and plasticizers. The resulting mixtures are pressed into shells and rods. The shells and rods laminated to form a composite feedrod that is then placed in a heated 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**).

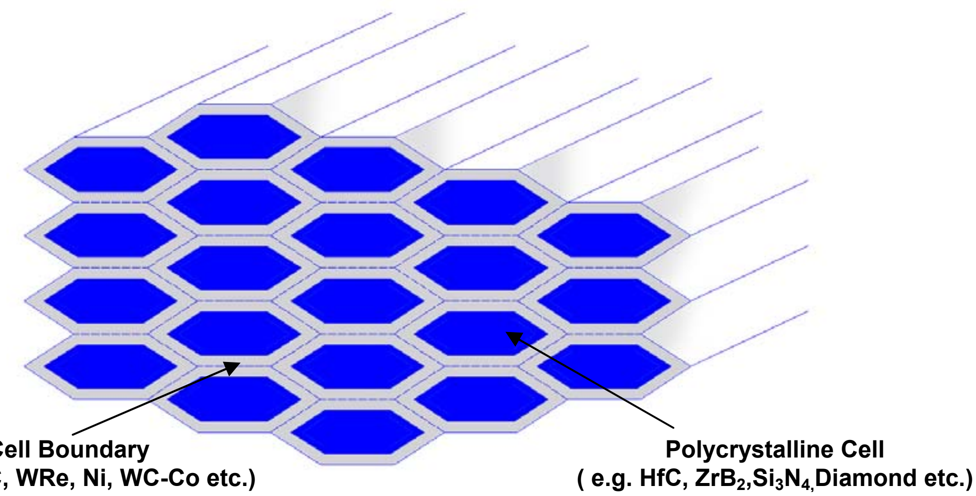

Figure 2. Schematic of a typical uniaxial Fibrous Monolith microstructure shown perpendicular to principal fiber direction.

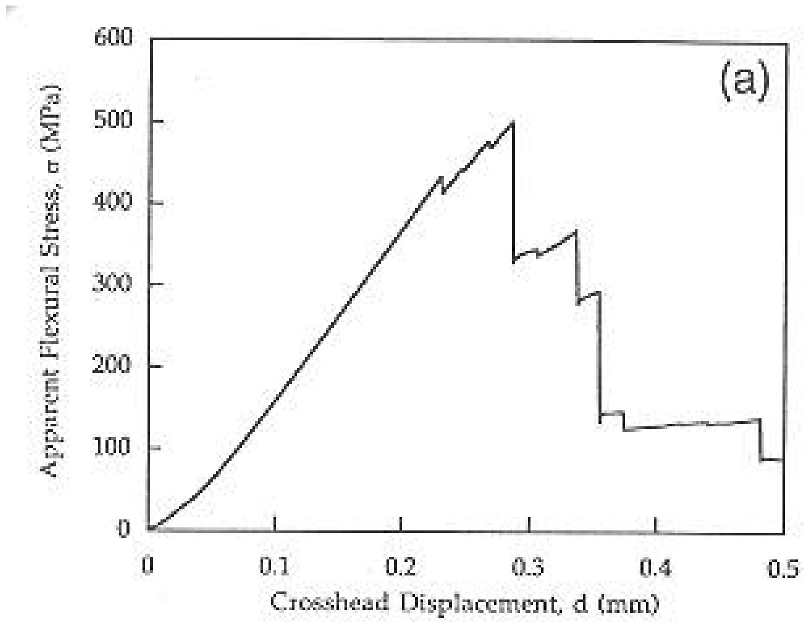

Figure 3. Typical flexural stress-strain curve for a silicon nitride/BN FM material.

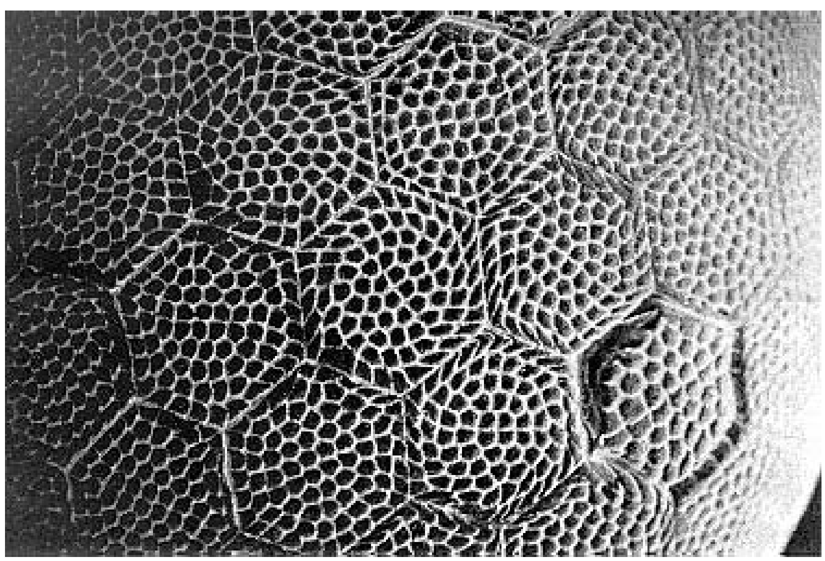

Figure 4. ACR's Diamond/ WC-Co FM composite applied as a coating on the surface of a WC drill bit insert (100x). Note the isolation of the darker material (Diamond) into discrete cells by the lighter contrast phase (WC-Co).

EXECUTIVE SUMMARY

During the reporting period, work was performed to characterize the two-stage binder removal process for WC-Co based FM material systems. Use of this process has resulted in the fabrication of defect free sintered WC-Co FM bodies, with minimal free carbon porosity and densities approaching 100% theoretical. In addition, shrinkage of the monolithic core and shell materials used in the WC-Co based FM system was measured, and differences in material shrinkage were identified as a potential cause of cell boundary cracking observed in sintered parts. Re-formulation of material blends for this system was begun, with the goal of eliminating mechanical stresses during sintering by matching the volumetric shrinkage of the core and shell materials. This development will allow the fabrication of prototype components for field-testing using the pressureless sintering process.

The component fabrication effort remained focused on drill bit inserts, conical and radial tool inserts and wear plates/inserts for earth moving equipment. Thirty-three 7/8" drill bit inserts (WC-Co(6%)/WC-Co(16%) FM were hot pressed during the reporting period. Six of these inserts were delivered for field-testing by Superior Rock Bit during the upcoming reporting period. In addition, Al_2O_3/Al_2O_3-TiCN FM cutting tool inserts were fabricated, and cutting tests performed.

Meetings with Kyocera Corporation took place in December, at ACR. ACR participants included program PI Dr. Mark J. Rigali (Manager of Composite Ceramics), Mike Fulcher, (Research Engineer), Ken Knittel (Research Engineer) along with ACM personnel Joe Halloran (Development Engineer). On the Kyocera side the participants were Junichi Imada Manager, Tatsuyuki Nakaoka Materials Development, and Daisuke Shibata Materials Development. Both Kyocera and ACR personnel presented recent results of materials development for WC-Co based FMs, as well as other fibrous monolith material systems of interest to both parties.

PROGRAM MANAGEMENT

The integration of partners into the program has required significant travel in order to build relationships and work toward agreement on the pursuit of materials, approaches and intended outcomes for the Fibrous Monolith Wear Resistant Components.

Dr. Zak Fang, University of Utah

Dr. Zak Fang, professor at the University of Utah in Salt Lake City, began work on development of densification processes (**Task 4**) during the reporting period. Dr. Fang is formerly of Smith International, and was an active participant in the development of diamond/WC-Co Fibrous Monolith materials for oil and gas drilling applications. Dr. Fang is well recognized as an expert in the field of WC-Co and other hardmetal materials. Several meetings were held with Dr. Fang over the course of the reporting period, both in Utah and at ACR's facilities.

Dr. Greg Hilmas, University of Missouri-Rolla

Work was begun during the reporting period on green processing and binder removal (**Task 3**) by Dr. Greg Hilmas, professor at the University of Missouri-Rolla. Dr. Hilmas is formerly of ACR, and was an active participant in the development of fibrous monolith material technology, including diamond/WC-Co Fibrous Monolith materials for oil and gas drilling applications. Dr. Hilmas is well recognized as an expert in the field of fibrous monolith materials. Several meetings were held with Dr. Hilmas over the course of the reporting period, both in Missouri and at ACR's facilities.

Kyocera Corporation

Meetings with Kyocera Corporation took place December 2002, at ACR. ACR participants included Mike Fulcher, (Research Engineer), Ken Knittel (Research Engineer) and program PI Dr. Mark J. Rigali (Manager of Composite Ceramics). Joe Halloran, of Advanced Ceramics Manufacturing was also in attendance.

Those present from Kyocera were Junichi Imada (Manager), Tatsuyuki Nakaoka (Materials Development), and Daisuke Shibata (Materials Development). Tours of the ACR's facility were conducted. Presentations by Kyocera Sendai personnel included topics on WC-Co, Si_3N_4/BN and diamond/WC-Co FM development. Lengthy discussions were held on the topics of formulation, green processing and manufacturing, with emphasis on binder removal processing and ideas for improved manufacturing techniques.

Dr. Mark Rigali, Mike Fulcher and Ken Knittel made presentations about the state of research and materials systems up to that point. The tests planned to evaluate the FM materials were discussed. Processing improvements, such as continuous co-extrusion, binder removal improvements and consolidation process improvements were also discussed, including aspects such as technical difficulties and possible equipment availability.

EXPERIMENTAL

Task 2. Develop Compositions of Fibrous Monoliths

During the second reporting period, materials were down-selected from the original trade study based on the results of preliminary consolidation investigations. The down-selected material systems are listed in **Table 1**. Efforts during the reporting period were focused on the development of these material systems for mining applications.

Table 1 – Down-Selected Core and Interface Materials for FM Development

Core Material	Interface Materials
Tungsten Carbide-Co(6%)	Cobalt metal
Tungsten Carbide-Co(6%)	Tungsten Carbide-Co(16%)
Diamond	Tungsten Carbide-Co(14%)
Alumina	Alumina-TiCN

During the reporting period, ACR began investigating material sources and potential benefits of WC-based FM systems with core to shell cobalt ratios other than 6%:16%. Because the difference in mechanical properties between the core and shell are what give FM materials their enhanced wear resistance properties, it has been suggested that increasing the cobalt ratio from the current 6%:16% to 6%:20%, 6%/30% or 3%/16% will result in a subsequent increase in wear resistance. As time and resources permit, samples of WC-based FM materials will be fabricated and tested for mechanical properties, and potentially fabricated into prototypes for the appropriate field-testing applications.

Task 3. Develop Fabrication Process Parameters of Fibrous Monoliths

Fabrication process parameters for fibrous monoliths include thermoplastic blending, core and shell molding, core and shell co-extrusion, coupon fabrication, and binder removal. Extrudable formulations for all the materials listed in **Table 1** were developed during the first twelve months of this effort, and work during the previous reporting period focused on the development of optimized binder systems and binder removal conditions. Based on this development effort, a two-stage, low temperature vacuum followed by high temperature binder removal in a reducing (Ar/H_2) atmosphere was developed. During process development, the two-stage process demonstrated a high yield of bloat free parts, as well as reduced free carbon levels within the range necessary to prevent the formation of residual free carbon (also called "carbon porosity") during consolidation. The two-stage binder removal process has become ACR's standard process for fabricating WC-Co containing parts, and has been successfully used to fabricate parts (e.g. test coupons, rod stock, inserts) that have sintered densities in excess of 99%.

In order to further characterize and optimize the two-stage binder removal process, experiments were begun to both calculate and measure free carbon levels in WC-Co FM samples with varying final soak (550 °C) times. The final soak temperature was selected based on published literature in which it was determined that at 550 °C the kinetics for carbon reduction by H_2 were most favorable. WC-Co FM cylindrical samples 1 cm in diameter and 1 cm tall were fabricated and used for the characterization experiment. The low temperature vacuum stage of the binder removal process was held constant, and the final soak time of the high temperature stage (**Table 2**) was varied from 4-24 hours in increments of 4 hours. The free carbon was then calculated using the weight of each sample, and the known binder formulation and loading. A chart of the calculated percentage of organic material removed is shown in **Figure 5**. The data demonstrates the expected trend, with the calculated percentage removal of organic material from the FM samples decreasing with decreasing final soak times. Additional process runs with final soak times of 0 and >24 hrs are planned, and will be carried out during the next reporting period. In addition, samples from each of the runs have been sent out for LECO carbon analysis, to verify the calculated data. This information will be presented, with the calculated values, in the next technical report.

Table 2 – High Temperature Ar/H_2 Atmosphere Binder Removal Profile

Start Temp	End Temp	Heating Rate	Soak Time
RT	325 °C	30 °C/hr	8.0 Hours
325 °C	550 °C	30 °C/hr	24 Hours
550 °C	RT	Unforced Cooling	N/a

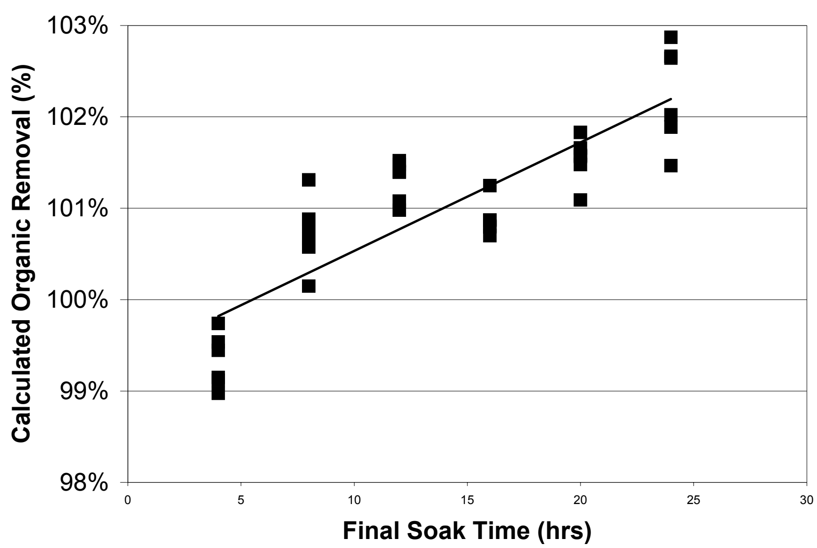

Figure 5 – Calculated organic removal from WC-Co FM samples with varying binder removal profile final soak (550 °C) time in Ar/H_2 atmosphere.

While the trend of decreasing organic removal with decreasing final soak times was expected based on published literature and past experience, the calculated values of greater than 100% organic removal were unexpected. There are several possible explanations for this observation, including measurement error, formulation error, or potential loss of Co from the samples during binder removal. Calculations made using monolithic WC-Co(6%) and

WC-Co(16%) material samples fabricated to determine material shrinkage during sintering (see **Task 4**) have shown that in samples of core material weight loss during binder removal is essentially 100%, however, the weight loss in the shell material is significantly higher at 102.5%. Because the shell material has significantly more cobalt than the core material, it appears as if cobalt loss is the likely cause of the extra weight loss in both the monolithic and FM samples. A cobalt loss of 0.01 g during binder removal would account for the increase in calculated organic removal percentage seen for the binder removal process with a 24 hour final soak time. This is approximately 1.5% of the total cobalt in each sample, and is not expected to have a major impact on the mechanical or wear properties of the WC-based FM system. Such loss may also be expected in conventional monolithic cemented carbides as well.

<u>Summary of Work performed at UMR</u>

A subcontract was put in place during the previous reporting period with Dr. Greg Hilmas, professor at the University of Missouri-Rolla. Dr. Hilmas is formerly of ACR, and was an active participant in the development of fibrous monolith material technology, including diamond/WC-Co Fibrous Monolith materials for oil and gas drilling applications. Dr. Hilmas is well recognized as an expert in the field of fibrous monolith materials, and has been enlisted to support the program in the area of formulation and binder removal process development.

To date, UMR's has made progress on this program in two areas. The first of these involved developing powder binder compositions that can be successfully co-extruded and undergo binder burnout to remove the organic binder in the components. The powder binder blends to be used had to serve three basic functions. First, the binder system had to be able to accommodate a high (>55%vol) loading of powder and still be developed as a uniform blend. The binder system that was chosen is able to accommodate 60%vol powder. Second, the powder binder system had to provide successfully co-extruded FM architectures. With the developed binder system, UMR has been able to manufacture fibrous monolithic structures using the co-extrusion process. UMR has also been able to develop a binder burnout profile that completely removes the polymer, and leaves no externally visible defects.

The second part has focused on the production of laminate samples using alternating layers of the core and shell material from the co-extrusion process. This study was undertaken to create a clearer understanding of the role of Co content on the mechanical behavior of the WC-Co FM systems. The success of the WC-Co based FM material system in the mining tool bit application depends greatly on the manner in which cracks interact with the core/shell materials and the interface between them. In order to study this aspect of the program, it was determined that laminate samples would provide a good method for testing how cracks would interact with the core and shell and their interface. In order to produce these samples, sheets of material were pressed using similar compositions to those used for co-extrusion. These sheets were then laminated together into a large disk, or square. Sample bars were then cut from the large laminate. After undergoing binder burnout the samples were then sintered. All samples that have been sintered to date show some form of minor cracking, either visible on the outside, or in the center of the bar after sectioning.

Work in the next reporting period will be focused on altering the binder burnout and sintering profiles to eliminate the formation of flaws in the laminated samples. Once this has been accomplished and the laminated samples can be successfully burned out and sintered, mechanical testing will be performed. Specifically, UMR will be performing flexural strength and fracture toughness measurements in four-point bending along with hardness measurements by indentation.

Task 4. Densification Process Development

WC-Co based FM systems

Densification studies are being performed in order to optimize conditions and develop processes for the fabrication of fully dense ceramic parts. Incomplete densification can be detrimental to hardness, fracture toughness and transverse rupture strength, which are all important factors determining the performance of the mining components under development. Full densification of the WC-Co materials is critical to the performance of these materials in applications for drill bit inserts, wear surfaces, and hydrocyclone cones. One process being evaluated for consolidation of this system is pressureless sintering. Pressureless sintering is the most cost effective consolidation process, and can be used to fabricate large batches of parts. Experiments to optimize pressureless sintering for the WC-Co based FM were designed and carried out during the reporting period, both at ACR and the University of Utah (Dr. Zak Fang). This process is used extensively in industry for densifying WC-Co based materials. Specific experiments carried out included time, temperature, and heating rate evaluations, as well as measurements of individual material shrinkage during sintering at varying temperatures.

Using samples fabricated for the binder removal study discussed in **Task 3**, evaluations of sintering rate, soak time and temperature were performed. Initial sintering experiments were carried out using an alumina crucible and lid containing a small alumina crucible filled with Co metal. The reservoir of Co metal is used to prevent excessive evaporation of cobalt from the WC-Co FM parts during sintering by creating a partial pressure of Co at the sintering temperatures. Results of initial experiments confirmed the expectation that part density increased with increasing soak temperatures and time, and appeared to be unaffected by the heating rate of the furnace during sintering. Upon cross sectioning of the sintered samples, a significant number of parts contained small cracks, which at first appeared to be associated with bundle boundary separations during binder removal. Bundle boundary separations are typically caused by poor or incomplete lamination of the individual filament during lamination of the green part. Microscopic observation of the cracks at 100X, however, showed that the cracks were actually along both the filament and cell boundaries (**Figure 6**). Because the cell boundaries are formed at a relatively high pressure (~10X green lamination) during the second pass extrusion process, cracks along the cell boundaries are most likely not formed during binder removal, and likely formed due to excessive mechanical or thermal stresses during sintering. Additionally, after several sintering runs it was observed that the alumina crucible was cracked and had a high surface roughness that was not present when the crucible was first used. Based on discussions with Dr. Fang, it was determined that the alumina crucible was dissolving at the sintering temperatures of the WC-Co FM, and use of the alumina crucible was halted.

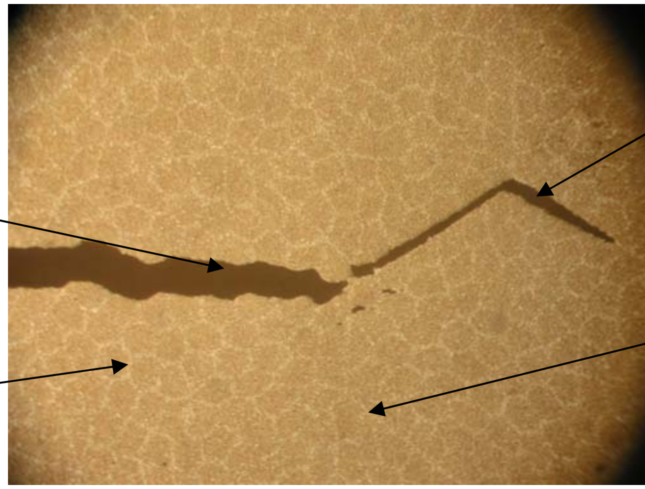

Figure 6 – Examples of cell and filament boundary cracking in sintered WC-Co based FM samples.

To determine if the use of the alumina crucible was the root cause for the cracks observed after sintering, a new graphite crucible was purchased. The new sintering set-up is identical to the old, with graphite replacing alumina. A small number of parts were sintered in the graphite crucible, at two different heating rates. The two heating rates used were the standard fast heating rate (2700 °C/hr) used for earlier sintering experiments at ACR, and a rate (300 °C/hr) suggested by Dr. Fang (see discussion of U of Utah work) that produced crack free samples. A summary of these results is presented in **Table 3**. Based on the observance of cracks in one of the samples sintered using the highest heating rate, it was determined that the cracks seen in parts from earlier sintering runs were not due to the use of the alumina crucible.

Table 3 – WC-Co FM samples sintered using different heating rates

Sample #	Heating Rate (°C/hr)	Soak Temp.	Soak Time	Density	Cracks
1	300	1350 °C	60 minutes	14.61	No
2	300	1350 °C	60 minutes	14.65	No
3	2700	1350 °C	60 minutes	14.62	No
4	2700	1350 °C	60 minutes	14.54	Yes

Once the alumina crucible had been eliminated as a possible source for the boundary cracking observed in sintered WC-Co based FM samples, it was postulated that the cracks are a result of mechanical stresses caused by a difference in shrinkage between the core and shell materials during sintering. This hypothesis is based on comments from WC-Co material suppliers, in this case Kennametal, with respect to differences in material shrinkage with differing Co percentage. Corroborating this hypothesis in the observation of large domains of cobalt (called "pools") in samples sintered at the University of Utah using the slower heating rate (see discussion below), which are most likely cracks that have been healed by the flow of cobalt during the longer sintering run. To verify that the shrinkage of monolithic WC-Co(6%) and WC-Co(16%) were different, an experiment to measure the individual shrinkage of core and shell materials was designed and begun during the reporting period. Samples of monolithic WC-Co(6%) and WC-Co(16%) 1 cm in diameter and 1 cm high were prepared, using the standard EVA-wax formulations and the standard two-stage binder removal process. These samples were then sintered at varying temperatures using the rapid heating profile, and then the volume was measured by water immersion and compared with the green material volume measured using the same technique. Data on the shrinkage of the two monolithic materials is presented in **Figure 7**.

From **Figure 7**, it is clear that at all the sintering temperatures investigated, the shrinkage of the shell material is significantly (2-2.5%) higher than that of the core material. In the current green formulation, the solids loading for both materials is 58%, which would result in a shrinkage of 42% assuming ideal mixing of the powders and polymer. The shrinkage of the core material is very close to 42%, however the ~45% shrinkage of the shell material may be enough to cause significant mechanical stresses and cracking of the FM parts during sintering. To eliminate the heating rate as the cause of the shrinkage difference, an additional sintering run will be made using the slower heating profile. If the additional run confirms the difference in material shrinkage, work to reformulate the green formulations and produce materials with closely matched shrinkages will be performed during the upcoming reporting period. Also during the upcoming reporting period, both monolithic and FM samples will be fabricated and sintering experiments performed at both ACR and U of Utah.

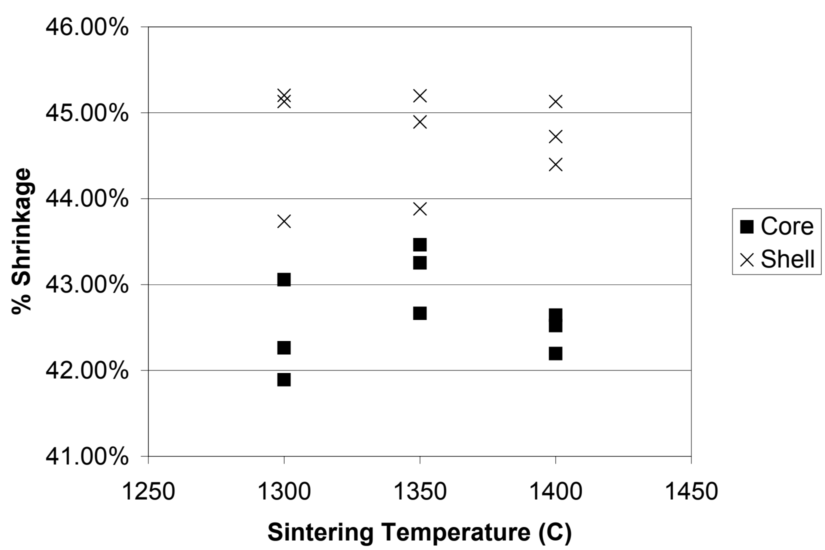

Figure 7 – Shrinkage measurements on monolithic WC-Co(6% and 16%)

<u>Summary of Work performed at U of Utah</u>

Work at U of Utah is focused on the effect of sintering and the diffusion of cobalt during sintering of the WC-Co FM system. While it is known that diffusion of cobalt across the cell/boundary interface is a function of the sintering temperature, the role of various process and material parameters (e.g. particle size and porosity) on the diffusion kinetics of cobalt needs to be further investigated and understood. Preliminary experimental studies of the sintering behavior of WC-16%Co/WC-6%Co FM composite were carried out during the reporting period. These studies were focused on varying the sintering temperature to determine the influence of sintering temperature on the diffusion kinetics of cobalt in the system. Samples were sintered in a vacuum furnace at different sintering temperatures, using a profile with one, two or three elevated soak temperatures. Sintered samples showed an increase in density with sintering temperature. Micro-hardness measurements also showed an increase in Vickers hardness values with sintering temperature. At sintering temperature of 1320 °C and above, a difference in micro hardness values of about 100 between cell and boundary was observed, with the cell having higher values compared with the boundary. Similar to the micro hardness values, the macro hardness values also showed an increase with sintering temperature. In all cases, the cellular structure of the FM (WC-16%Co/WC-6%Co) was preserved during liquid phase sintering. In the next reporting period, additional sintering experiments will be performed to determine the extent of diffusion of cobalt before the onset of liquid phase sintering. Results of this study will enable the prediction of final cobalt concentrations based on starting cobalt percentages in the core and shell materials, and will allow for the design of final mechanical properties of the WC-Co based FM systems to met specific application requirements.

<u>Al_2O_3-based Systems</u>

In the previous technical report, development of consolidation conditions that produced Al_2O_3 based samples with >99% theoretical density was discussed. Al_2O_3 based FM materials are being pursued at the request of Kyocera, an industrial partner on this program and Kennametal, a customer of ACR. In additional to the mining applications, such as roof bit inserts, Kennametal is interested in the FM systems as a way to potentially improve material performance over the currently available monolithic metal cutting inserts. Alumina based materials are currently in use for machining cast gray iron. It has been proposed that the FM structure will create a more durable, longer lasting machine tool insert than available with conventional monolithic materials. While there is widespread use of this machining technique, substantial market share is currently dominated by two or three manufacturers.

Task 5. Fabrication of Laboratory Test Samples

<u>Testing underway</u>

<u>WC-Co based FM systems</u>
During the reporting period, ACR completed fabrication of a abrasion testing system based on ASTM Standard B661 for testing high stress abrasion resistance, and input from Dr. Fang at the university of Utah. A photo of the testing machine is given in **Figure 8**. Samples of ACR's hot pressed WC-Co based drill bit inserts, as well as commercial available WC-Co bit inserts for comparison, have been fabricated and will be tested using this machine. Results from these tests will be presented in the next technical report.

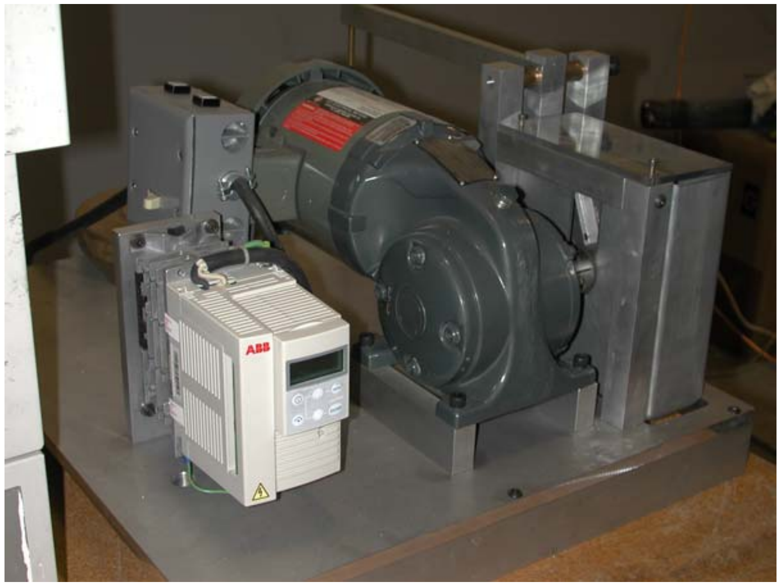

Figure 8 – High stress abrasion testing machine.

Al_2O_3 based FM systems

Cutting tool tests on Al_2O_3 based monolithic and FM systems were performed at Competitive Engineering in Tucson, AZ during the reporting period. This work is being undertaken by Advanced Ceramic Manufacturing (ACM) as part of the industrial cost share commitment to this program. Several rectangular test inserts were prepared, using both ACR and ACM material formulations, and compared with a baseline Kennametal K090 Al_2O_3-TiC commercially available insert. The test parameters are given in **Table 4**. Results of the testing indicated that both the monolithic (~50% of K090) and FM (~20% of K090) systems still require additional development work to meet the baseline performance established using the K090 insert. ACM plans to continue this development effort during the upcoming reporting period.

Task 6. Fabrication of Drill Bit Inserts

With the development of improved thermoplastic blends and binder removal processes, work was begun during the previous reporting period to fabricate large drill bit inserts for field-testing. A total of 33 inserts were hot pressed during the reporting period. Photographs of sample inserts before and after centerless grinding are presented in **Figure 9**. A cross section of one of the inserts that was sectioned for abrasion testing is shown in **Figure 10**. Six inserts were delivered for field-testing to Superior Rock Bit Company, Virginia, MN. Frank Klima of Superior has agreed to provide field-testing of inserts for no charge. Results from this field-testing will be discussed in the next technical report. The remainder of the insert will be machined as necessary to meet upcoming field testing needs.

Figure 9 – Hot pressed WC-Co(6%)/WC-Co(16%) FM drill bit inserts before (left) and after (right) centerless grinding.

Figure 10 – Cross section of hot pressed WC-Co(6%)/WC-Co(16%) FM drill bit insert at 5X (left) and 50X (right).

As additional improvements are made to the thermoplastic blend formulations and the binder removal (**Task 3**) and sintering processes (**Task 4**), inserts will be fabricated for consolidation by sintering. Consolidation by sintering represents a considerable cost benefit, when compared to hot pressing, and is the current practice in industry for consolidation WC-Co monolithic inserts. It is expected that sintered inserts suitable for field-testing will be fabricated during the upcoming reporting period.

PLANS FOR THE NEXT REPORTING PERIOD

1. Complete pressureless sintering process optimization of Fibrous Monolith compositions selected for the mining drill bit insert application (Task 4).
2. Begin testing of WC-Co based FM samples using high stress abrasion wear tester (Task 5).
3. Field test Fibrous Monolith drill bit inserts at Superior for evaluation of mechanical properties and field performance (Task 6).
4. Fabricate WC-Co FM samples and provide prototypes for additional field-testing by Eagle Innovations (pump seals) and RA-TECH (stabilizer inserts) (Task 6).
5. Develop additional mining industry field testing contacts and fabricate samples for field-testing (Task 6).

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