IMPROVEMENT OF WEAR COMPONENT'S PERFORMANCE BY UTILIZING ADVANCED MATERIALS AND NEW MANUFACTURING TECHNOLOGIES: CASTCON PROCESS FOR MINING APPLICATIONS

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ABSTRACT

Michigan Technological University, together with The Robbins Group, Advanced Ceramic Research, Advanced Ceramic Manufacturing, and Superior Rock Bits, evaluated a new process and a new material for producing drill bit inserts and disc cutters for the mining industry. Difficulties in the material preparation stage slowed the research initially. Prototype testing of the drill bit inserts showed that the new inserts did not perform up to the current state of the art. Due to difficulties in the prototype production of the disc cutters, the disc cutter was manufactured but not tested. Although much promising information was obtained as a result of this project, the objective of developing an effective means for producing rock drill bits and rock disc cutters that last longer, increase energy efficiency and penetration rate, and lower overall production cost was not met.

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EXECUTIVE SUMMARY

Mining involves rock fragmentation in the form of drilling, cutting, digging, crushing, and grinding, under severe impact and wear conditions. The materials of construction and manufacturing processes to manufacture the tools used to fragment rock affect cost and performance, in terms of production rate and component life. Recently, new materials and manufacture techniques have been developed. Fiber reinforced ceramics, which can increase both the toughness and hardness of brittle materials, are very promising. A new manufacturing technique, developed by Michigan Technological University, combines casting, forging, powder metallurgy, bonding and coating in one process to produce high performance components from advanced materials with complex macrocomposite structures. This manufacture technique is suitable to produce key wear components with complex shapes and structures.

New materials (fibrous monoliths) and a new manufacturing technique (CastCon) present a great potential to improve performance of wear components. Key wear components, specifically drill bits and disc cutters, were selected to explore these new technical advances.

The objective of the project was to develop an effective means for producing rock drill bits and rock disc cutters that last longer, increase energy efficiency and penetration rate, and lower overall production cost based on the combination of Fiber Monolith (FM) and CastCon technologies.

Project team members included: Michigan Technological University's (MTU) Institute of Materials Processing and the Geological and Mining Sciences and Engineering department, The Robbins Group, Advanced Ceramics Research, Advanced Ceramics Manufacturing, and Superior Rock Bit.

Drill bit inserts were produced from the FM materials and tested in the field. Material testing initially showed promising results when comparing FM and conventional materials. The Vicker's hardness and fracture toughness was much greater with the FM material. However, this could not be repeated when various different FM and insert preparation methods were used. Inserts were put into a rotary tri-cone drill bit and used to drill taconite. Because the inserts did not show better wear resistance than the current state-of-the-art, this focus area did not progress to making drill bits using the CastCon process.

Manufacturing disc cutter sections was successfully tested on a small scale, using conventional and FM inserts. The next step was to manufacture full size disc cutters, using the CastCon process. The decision was made by the project team, together with DOE, to not use FM inserts in the prototype portion of this study. A variety of problems were encountered and solved during the prototyping phase, but an acceptable disc cutter was not produced.

In conclusion, although both the fiber monolithic and CastCon technologies showed promise, the project objective of producing longer lasting, lower cost drill bits and disc cutters was not met.

INTRODUCTION

Improving wear components performance by utilizing advanced materials and new manufacturing technologies was proposed to satisfy several research priorities described in the Mining Industry Roadmap for Crosscutting Technologies: 1) enhancement of exploration and resource characterization, 2) material wear properties, and 3) new materials for wear resistance. The reason given by the Roadmap for the materials research priorities is that the energy, safety, and cost benefits resulting from the use of new materials could be moderate to high. The targets of the materials research set by the Roadmap are to reduce operational downtime, increase energy efficiency, and improve use of consumables [1].

This project was initiated by Michigan Technological University (MTU). The Robbins Group, Advanced Ceramics Research and Superior Rock Bit were invited to participate. These organizations committed total 20% matching fund to support the project. The major experimental work was conducted by scientists, engineers, students and technicians at the Institute of Materials Processing of MTU. Our industrial partners also conducted great amount of experimental work, especially in FM sample fabrication, disc cutter prototyping and field tests. This research team kept a close collaboration during execution of the project by weekly conference meetings. Two professors from the Mining Department and Materials Science and Engineering Department of MTU joined the research to provide multidisciplinary expertise. This project also supported two Ph.D students.

Michigan Technological University is a well-established engineering research university in the Upper Midwest Region of the United States. Founded in 1885 as the Michigan School of Mines, it now supports eight mature fields of study in its College of Engineering, the largest of five colleges/schools within the University. Its combination of maturity and innovation draws faculty from across the continent to teach and research in an interdisciplinary environment.

The Robbins Company is an internationally recognized manufacturer of drilling and tunneling hardware for the mining and civil construction industries. Among the wide range of drilling and tunneling products produced by the Robbins Company were most of the machines that bored the English Channel Tunnel. The Robbins Group is a service firm spinoff from the Robbins Company. The Robbins Group worked in the team to develop the design of the disc cutter and select the appropriate materials for its fabrication.

Superior Bit Company. Frank Klima, President, will contribute his 20+ years of experience building and marketing rock-drill bits for the mining industry. A graduate of Michigan Tech, Mr. Klima has devoted his career to designing and manufacturing large-diameter blasthole bits suitable for the incredibly tough requirements of the Mesabi Iron Range. Together with Richard Gertsch, he will develop the design of the blasthole bit to be fabricated with the CastCon process.

PROJECT OBJECTIVE

The objective of the project was to develop an effective means for producing rock drill bits and rock disc cutters that last longer, increase energy efficiency and penetration rate, and

lower overall production cost based on the combination of Fiber Monolith and CastCon technologies.

BACKGROUND

The mining industry provides direct employment for roughly 355,000 people and indirect employment for an additional 5 million [7]. In 1997, mining produced $59.4 billion, including $27.1 B of industrial minerals, $12.4B of metallic minerals, and $19.9B of coal[7]. During the same period, $2.1B was spent on mining processing equipment[7]. The combined direct and indirect value from mining accounted 7% of the national GDP[7]. In addition, mined coal and uranium produce 77% of the electricity used in the United States[7]. The reduction in mining cost will make contribution to employ more people, and increase the nation GDP, and reduce the cost of electricity in the U.S.

Mining involves a great deal of rock fragmentation in the form of drilling, cutting, digging, crushing, and grinding. The tools that apply the fragmentation energy are subjected to severe impact and wear conditions. The materials of which these tools are made significantly affect cost and performance of the tools, in terms of production rate and component life. In many circumstances, the materials the tools are made of are the major factor to cause operational downtime for replacing consumables, to limit processing rate, and to affect overall production cost. For instance, geothermal drilling frequently encounters hard-rock, which reduces bit penetration rate and bit life, and raises the drilling cost. If both penetration rate and bit life could be doubled by using a diamond drill bit, an average reduction of 15% to 20% in drilling costs results[2-3].

In recent years, new materials and manufacture techniques have been developed by researchers in the area of materials and metallurgical engineering. One of the promising materials which could be used to improve both hardness and impact resistance of wear components is fiber reinforced ceramic. It is well known that hard materials posses good wear resistance, but are brittle with low fracture toughness. Improving toughness without sacrificing wear resistance has been very difficult. Recent development in fiber reinforced ceramics has shown the possibility of increasing both toughness and hardness of brittle materials.

A new manufacturing technique has been developed by Michigan Technological University. This manufacturing technique combines casting, forging, powder metallurgy, bonding and coating in one process to produces high performance components from advanced materials with complex macrocomposite structures. This manufacture technique is suitable to produce key wear components with complex shapes and structures.

The new materials and new manufacturing technique present a great potential to improve performance of wear components. This project was to select key wear components as the candidates to explore applications of these new technical advances. The key components should be so important that the mining processes using the equipment installed with the key components could benefit a great deal if the properties of these components can be improved.

ECONOMIC COMPETITIVENESS

The U.S. mining industry has been the world leader in producing and processing minerals and mineral products at competitive prices while mining employees earn an average of $44,000 per year compared with $29,000 per year of the average salary for all industries. The low cost coal and uranium produced by the mining industry keep the U.S. electricity cost among the lowest in the world. The low electricity cost helps to enhance the competitiveness of the entire U.S. industry. However, the mining industry has been facing and will continuously face keen competition in cost-effective exploration, mining and processing of mineral products. The minerals commodity market has to compete with both other domestic suppliers and suppliers from international market. The economic competitiveness of the U.S. mining industry depends on its ability to maintain lower production costs and increase efficiency. This requires to continuously find better methods, technologies and processes, which can maintain and improve safety and environmentally sound conditions while continuing to increase productivity and cut costs.

The proposed research is to explore the opportunities of applying new materials and manufacture technologies on the wear components used by the mining industry. As the first step, two key wear components used in exploration and mining will be focused on for the investigation. These key components play an important role to increase work efficiency, reduce operational downtime, and lower production cost in excavating. Successful research and consequent industrial applications of the new technology will have potential to double wear component's life and penetration rate and greatly reduce energy consumption and operation costs in exploration and mining.

As the second step, more wear components will be selected for research on utilizing advanced materials and new manufacture process to improve their performance, which will make greater contribution to improve the economic competitiveness of the entire mining industry.

The proposed method of combining advanced materials and new manufacturing technique to produce mining wear components is innovative method and will help the U.S. mining industry to maintain the world leader in crosscutting technologies.

SELECTION OF WORKING MODELS

A drill bit and a disc cutter were selected as working models for the project.

Drill bits

Drill bits are widely used in blast hole mining and mineral exploration. There are several types of drill bits. Conventional drill bits are manufactured in several steps: 1) manufacturing rollers, heads, or shanks by forging and machining; 2) manufacturing tungsten carbide cutters or inserts separately; 3) drilling and machining openings for insert in the bit body; and 4) mounting the cutters or inserts by brazing or friction. The process is expensive, time-consuming, and provides minimal confinement for the insert. Further, major tooling changes are required for changes in drill bit shape. Because the cutters and inserts on many roller cone drill bits and

percussion drill bits are placed by hand in holes machined in the bit body, the fit of each insert varies from insert to insert. Consequently, each insert experiences a different confinement stress. Also, controlled compressive stresses can increase insert strength by reinforcing the insert, which tends to be weaker in tension and shear than compression. If a better confinement technology were available, stronger inserts could result.

The drill bit's properties could be greatly improved by using fiber reinforced ceramics as the cutter or insert material, and providing better confinement on cutters or inserts as illustrated in Figure 1. The proposed manufacturing technique is described in Figure 2.

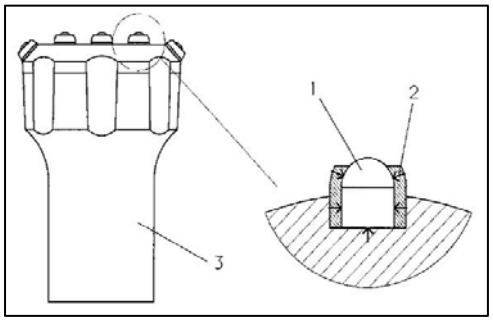

Figure 1. Schematic Illustration of a Percussion Rock Bit. Common in smaller diameter holes, the bit features a cast or machined bit body where inserts are placed in machined holes. We propose to develop a technology in which the insert (1), confinement substrate (2), and bit body (3) are HIPed in one step by the CastCon Process. (See Figure 2 for an illustration of how this bit is manufactured.)

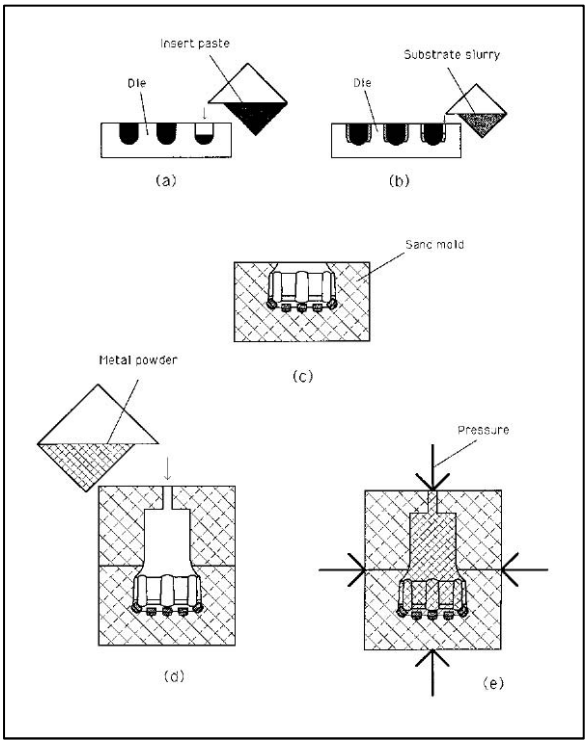

Figure 2. The CastCon Manufacturing Process for drill bit. This illustration shows the major steps in manufacturing a three-material drill bit. Steps may be added or deleted depending on the bit design and application required. a) A mold is prepared, a paste containing the nanometer diamond composite is introduced into the mold, and the paste cures to form preform inserts. b) After release from the mold in Figure 3a, a preform insert is placed in another mold for joining confinement substrate. The substrate slurry is then introduced into the mold, where it surrounds the insert and cures. c) The insert perform along with the confinement substrate perform are placed in the lower half of the sand mold, and the upper half of the sand mold is joined to the lower half. d) After joining the two halves of the mold, the metal powder that will comprise the bit body is poured into the mold. Note that the bit body can be assembled in layers, if differing material are needed for the bit application (e.g. the bit can graduate from a very hard face to a softer interior, if desired). e) The sand mold and the bit components are pressed and heated as a unit. At the end of the process, the nearly completed bit is produced. Sprue removal and machining of bearing surfaces or threads are all that remain.

Disc Cutters

Disc cutters are used on large cutterheads such as tunnel boring machines, raise borers, and shaft borers. High levels of force against the rock and a permanently sharp cutting profile are desired for maximum chipping ability. The former is governed mainly by the bearings in the disc mount, but the latter is limited currently by the properties of the disc material (4340 high strength steel). Sharp cutter profiles become dull extremely rapidly during use. Current art compromises with "constant cross section" profiles, which do not increase cutter bearing area (and thus do not lower applied pressure) significantly until a very large part of the cutter is worn away (Figure 4). The hardness of this high strength steel ranges between HRC 24-53, insufficient for long bit life. Brazing tungsten carbide inserts along the edge of the disc has not worked well, due to insufficient support provided for the inserts. The proposed new disc cutter and manufacturing technique are described by Figures 3 and 4.

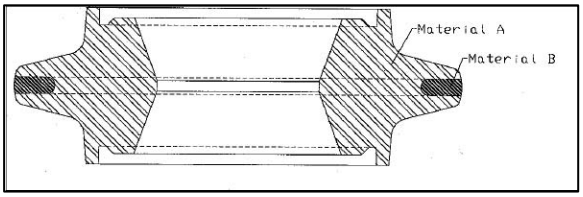

Figure 3. Proposed disc cutter structure. Material A will be selected from steel alloys, Material B will be selected from nanosized carbides, nitrides or diamond, and a third material could be inserted between Materials A and B.

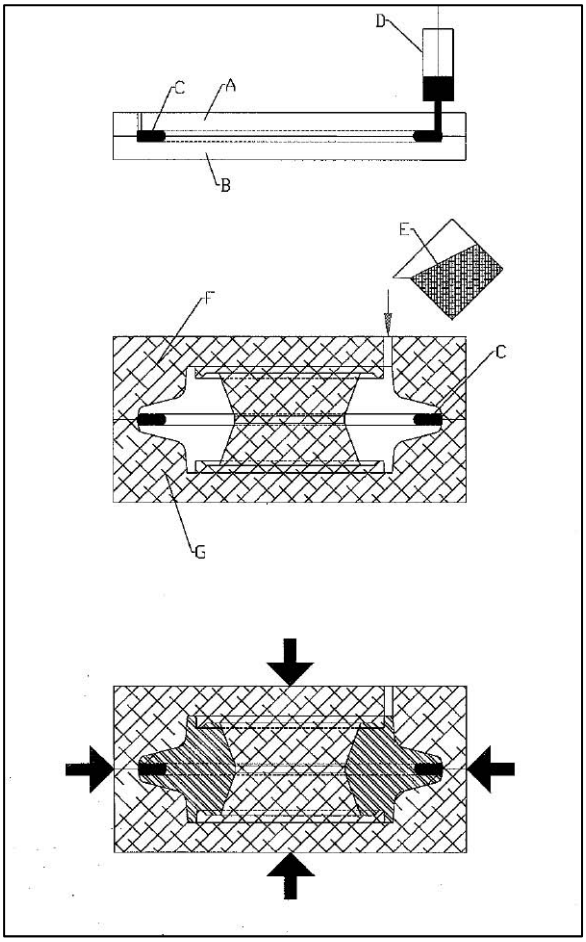

Figure 4. Proposed disc cutter manufacturing approach. A: top half die; B:bottom half die; C: paste made of nanosized hard material and a binder; D: injector to push the hard material paste into the die, forming a ring; E: powdered body material is charged into the sand mold with the hard material ring positioned in place; F: cope; G: drag; and the sand mold with a hard material ring and powdered body material inside is heated and pressed to produce the proposed disc cutter.

Fiber Monolithic Process and Materials

Adanced Ceramics Research developed the fiber monolithic process. It aims to produce a new class of structural ceramics which exhibit mechanical properties similar to continuous fiber ceramic composites, including high fracture toughness, damage tolerance and graceful failure. In this process, sets of inexpensive, thermodynamically compatible ceramic and/or metal powders are blended with thermoplastic polymer binders and then co-extruded to form a 'green fiber'. The green composite fiber may be wound, woven or braided into the shape of the desired component. The fabricated green components are then pyrolyzed to remove the polymer binder and sintered or hot-pressed to obtain the final FM product. FMs may have widespread applications as structural ceramics. Many components can be readily fabricated from the green materials using the process.

CastCon Process

The integrated manufacturing of rock drill bits and disc cutters as illustrated in Figures 1 and 3 is possible with the CastCon process. The CastCon process has been under development for more than eight years at Institute of Materials Processing (IMP) of Michigan Technological University (MTU). Two patents were issued in 1998[23-24] and several papers were published[25-28]. Exhaust valves, gears, surgical implants, propellers, wrenches, connecting rods, router cutters, plug cutters, twist drill bits, aluminium composite clad aluminium brake rotors, and tank track center guides have been successfully produced from 316 stainless steel, Co-Cr-Mo, Co 6, Ti-6Al-4V, D7 tool steel, C-276 Ni alloy, 718 super alloy, 17-4 dual phase steel, pure Ni, carbon steel, Al, Cu, WC, TiC, Ti_3Al, SiC, and Al_2O_3 powders. The papers and news releases by six magazines[29-34] on this process have attracted great interest from various industries. The CastCon process combines metal casting, forging, powder metallurgy, coating and bonding in one process to produce high performance components.

The major advantages of the CastCon process include:

- excellent shape forming capability inherited from the vast experience of sand molding techniques in the metal casting industry;
- good mechanical properties equivalent to forged parts resulting in low porosity and fine microstructure;
- great flexibility of producing a wide variety of metallic, intermetallic, ceramic and composite components by using various powdered materials and their mixtures;
- capability of producing macro composites by bonding different powders, or a powder to a solid, or a solid to a solid;
- a unique powder coating method for improving abrasive and corrosion resistances;
- a production process suitable for both single piece and high volume productions.

EXPERIMENTAL METHODS

This project considered two drilling technologies – drill bit inserts and disc cutters. The research efforts focused on the materials of construction (fiber monolith (FM)) and the method of manufacture (CastCon).

MATERIALS SELECTION

Current drill bit inserts are made of various grades of tungsten carbide (WC) cermet for working at different drilling conditions. Diamond clad WC inserts are also available. The project research group discussed the target insert material at the project kick-off meeting. We selected the FM WC/Co system as our material of choice because of its potential good combination of hardness and toughness and ACR's extensive experience in this material. The drill bit body is made of a forged tool steel.

Current rock disc cutters for tunneling machines have no insert and are made of 4340 high strength or H13 tool steel. The Robbins Group suggested working with H13 material and the research group decided to follow this suggestion.

FABRICATION OF WC FIBER MONOLITHIC INSERTS

The WC fiber monolithic (FM) specimens used for the drill bit inserts and disc cutters were produced by ACR and sent to Michigan Tech. These specimens were made using ACR's special organic binder system and extrusion process. During the project, changes were made to the insert materials, ACR fabrication process, and the binder removal and sample consolidation procedures, based on test results. Due to the proprietary nature of the ACR fabrication process, only a brief description of the final method is given below.

Green FM Precursor Fabrication

ACR's process starts with mixing selected powder/powders with an organic binder in a manner similar to that for metal or ceramic injection molding. Actually, the organic binder was not a single substance but consisted of a primary binder and several additives to improve mixing viscosity, binder and powder adhesion, mixture strength, and subsequent binder removal,

In this research, a WC/Co cermet system was used as the fiber or core material and either pure Co or WC/Co cermet acted as the matrix or shell material. To prepare for fiber manufacture, very fine WC and Co powders were mixed with an organic binder to form the fiber precursor. Similarly, the matrix material was prepared with a different ratio of Co powder and organic binder with or without WC powder.

The fiber precursor was extruded into a thin bar using a heated extruder at ACR. The matrix precursor was extruded separately into small and large tubes. The bars and tubes were cut into pieces. The bars were inserted into the small tubes one by one and then many small tubes were inserted into a large tube to form a fiber monolith. This was the first round of extrusion. This process was then repeated by extruding the fiber monolith into a bar and inserting those bars

into tubes. The process was repeated again to make the final fiber monolithic specimen. After completion of three rounds of extrusions, the large tube was finally extruded into a thick bar constructed of many WC/Co fibers in a pure Co or WC/Co matrix. The resulting thick bar or fiber monolithic precursor was then sent to Michigan Tech for further treatment.

Binder Removal and Insert Consolidation

The fiber monolithic precursors were cut or machined into specimens at Michigan Tech. A variety of methods were evaluated for binder removal and insert consolidation. The organic binder in the specimens needed to be removed by burn-out or evaporation up to 500°C, then the space left from the binder needs to be closed by sintering or hot pressing.

The binder burn-out was carried out in a vacuum/pressure furnace or a kiln vacuum system. The vacuum/pressure furnace is capable of vacuum level of 100 microns or argon pressure of 1500 psi. The binder burn-out was very difficult to do because cracks, distortion and expansion may occur in the specimens due to fast evaporation of the organic binder. Very slow heating rates of 1°C/min to 0.1°C/min were used in the experiments. Containment burn-out was tested to restrict expansion of specimens and provide support to specimens when the binder became soft. To execute containment, a specimen was buried in sand held by a container. The container was either open on top or closed with a stem. The container was heated in a vacuum furnace to remove the binder. The surrounding sand prevented the specimens from expanding and distorting. Different heating rates and sizes of sand were used.

Initially, two pressureless sintering tests were conducted by MTU in a vacuum furnace. The samples were thin pieces sliced from an ACR green bar. The first sample was heated at a rate of 1°C/minute to 500°C and held for 30 minutes to burn the organic binder, and heated continuously to 1100°C and held for one hour. The second sample was heated at a fairly slow rate of 0.5°C/minute to 700°C and held for 30 minutes, and heated continuously to 1100°C and held for one hour.

Further free standing pressureless sintering tests were conducted in a vacuum furnace by MTU using larger green bar samples (1.25” in height and 0.865” in diameter) at very slow burnout rates, as described below:

Procedure #1:

0.5ºC/min to 500ºC burnout, 5ºC/min to 1100ºC sintering

Procedure #2:

1. 0.1 degree per minute to 200ºC
2. One hour soak at 200º
3. 0.5 degree per minute from 200 to 300ºC
4. 0.1 degree per minute from 300 to 400ºC
5. 0.5 degree per minute from 400 to 500ºC
6. One hour soak at 500ºC
7. 5 degree per minute from 500 to 1100ºC
8. One hour soak at 1100ºC

ACR also conducted parallel burnout experiments in a vacuum environment.

ACR Experiment #1:	**ACR Experiment #2:**	**ACR Experiment #3:**
Heating rate: 1ºC/min	Heating Rate: 1ºC/min	Heating Rate: 1.25ºC/min
Soak Temperature: 200ºC	Soak Temperature: 150ºC	Heating Rate: 0.1042ºC/min
Soak Time: 6 hrs.	Soak Time: 6 hrs.	Soak Temperature: 150ºC
		Soak Time: 6 hrs.

ACR four day test

ACR tried a four-day vacuum burnout with the following profiles:

25°C to 80°C in 3 hrs,
80°C to 175° C in 16 hrs,
hold at 175°C for 8 hrs,
175°C to 300°C in 12 hrs,
hold at 300°C for 6 hrs,
300°C to 350°C in 6 hrs,
hold at 350°C for 6 hrs,
350°C to 400°C in 12 hrs,
400°C to 600°C in 12 hrs,
hold at 600°C for 6 hrs, and
gas backfilled cooldown.

The specimens become very weak after the binder is removed and need to be consolidated to full density to gain strength. There are three possible methods to achieve full density: sintering, sintering plus container-less hot isostatic pressing (HIP), and container HIP. The associated costs are low, medium and high. All three methods were tried in this research. In the first method, the specimen was heated to a consolidation temperature and held at temperature in a vacuum furnace. By referring to the literature regarding WC cermet and several trials, the consolidation temperature was determined to be 1350°C, which is close to the melting point of Co. In the second method, the specimen was heated to 1350°C and held at temperature in a vacuum mode, and then pressurized to 500 psi of argon at the temperature and held. It was hoped to achieve 95% density or isolate pores by sintering at 1350°C, and then the pressurized argon can act on the external surfaces of the specimen to close its internal porosity. In the third method, a specimen is surrounded by sand and encapsulated in a metal container.

Hot Pressing

Hot pressing is a process to press samples in a graphite or molybdenum die by one or two punches in vacuum or inert gas atmosphere at elevated temperatures. It is a high temperature unidirectional and mechanical pressing or a pressure assisted sintering process. In this research, we used a 20 ton vacuum hot press and a graphite die to consolidate WC and steel powders. It has a mechanical pump only and the vacuum level is normally less than 100 microns Hg.

In the first series of tests, the first sample was inserted in a graphite die with bottom and top punches and heated at a rate of 1°C/minute to 500°C and held for 30 minute and heated continuously to 1100°C. 4,000 psi pressure was then applied on the sample by a hydraulic cylinder through the top graphite punch and held at 1100°C for one and a half hour. The second hot pressing test was conducted at reduced hot pressing temperature of 950°C. Other conditions remained the same.

In the second series of tests, a previously heat treated sample was hot pressed to reduce porosity. The die assembly was heated in a hot press at 10°C/min from room temperature to 900°C and held for 1.5 hours. During heating, 4 ksi pressure was applied to the specimen through the bottom and top punches. The occurrence of porosity and cracks was evaluated. The specimen was then reheated at the same condition to 950°C and held for 1.5 hours, then evaluated. The specimen was reheated again to 1000°C, 1050°C, 1100°C and 1150°C and checked after each treatment.

ENCAPSULATION

A sand mold needs to be encapsulated in a metal container for HIPping in the CastCon process. We prepared two types of steel containers for making small samples and large parts in this project. The first container was small and consisted of a piece of 3" or 5" pipe with a bottom and a top covers. The bottom cover was welded on the middle pipe first. The sand mold was loaded into the uncovered container and closed with the top cover. The top cover was then welded on the container. The top cover had a steel stem welded on its center to provide access to the inside of the container. The second, larger container was made of 1/16" thick steel sheet. The steel sheet was cut, rolled and welded. It also had a stem welded on the top cover, as shown in Figure 5.


Figure 5. HIP can for disc cutters

To ensure a good air tight seal, all welding was done in a glove box with argon atmosphere. Excellent welding is required to prevent vacuum leaks. Can leakage occurred several times during the project and was a problem in making the discs. To ensure tight welds, we carefully cleaned the welding regions on the cans before welding using sand blasting and wiping with alcohol. We also did a vacuum leak check before finally sealing the cans.

The organic binder in the sand mold needs to be removed before sealing the container. Initially, binder burn-out was conducted in a vacuum furnace. Later, for the larger disc cutter samples, binder burn-out was conducted in a kiln using a mechanical pump connected with the stem of the container to extract the off-gases. The kiln has a temperature and time controller and is capable of maximum heating temperature to 1200°C. To reduce oxidation of cans in a conventional kiln without an inert or reducing gas protection, a carbon powder was used to cover and surround the cans. The binder burn-out and off-gassing temperature was also lowered to 500°C from 1100°C used in the vacuum furnace. The kiln binder burn-out procedure is given below.

- 5°C/min from 25°C to 200°C,
- held for 5 hours at 200°C,
- 5°C/min from 200°C to 500°C,
- held for 5 hours at 500°C, and
- furnace cooled.

The binder burn-out temperature varied from 500°C up to 1100°C in this project. The heating rate was 2°C to 5°C per minute from 25°C to 500°C and 10°C per minute from 500°C to 1100°C.

After the binder burn-out process in the vacuum furnace, the container was transferred into the glove box. The container was vacuumed with a mechanical pump to the vacuum level < 50 microns Hg in the glove box. The stem of the container was then clamped by a hydraulic press in the glove box and sealed by welding. The container is ready for consolidation by HIPping.

HOT ISOSTATIC PRESSING

HIP is a totally different piece of equipment from the hot press. HIP uses argon as the pressure transmitting medium. In HIP, samples are placed in a vessel full of argon. The argon is pressurized to 15 to 60 ksi and heated to elevated temperatures. The samples are then isostatically pressed.

The small containers were HIPped using MTU's Quick-HIP at 1100°C under 60 ksi for two hours. The large containers were sent to Bodycote Isostatic Pressing for HIPping at 1150°C under 15 ksi for four hours. Bodycote is a HIPping service company located at 155 River Street, Andover, MA 01810.

LABORATORY EVALUATION OF FM SPECIMENs

Microstructure evaluation and chemical analysis were conducted on the initial FM samples to determine why the binder removal and consolidation processes were not working as anticipated. Thermogravimetric analysis (TGA) was conducted to help determine why defects were forming in FM specimens. TGA determines the weight loss (evaporation rate) vs. temperature, and was conducted on a green FM sample at 170 torr with a heating rate of 1C°/min.

After consolidation, the fiber monolithic specimens were examined for their microstructure, porosity and mechanical properties and compared with those of a conventional WC cermet material. This laboratory evaluation provided an indication for performance during field testing.

Density determination

Density is important parameter to evaluate if a specimen reaches fully dense or not.

Remaining pores will significantly reduce mechanical properties of a material. It is commonly assumed that remaining pores will be isolated if a specimen's relative density is over 95%. Isolated pores in a specimen can be eliminated or closed by HIPping, i.e., applying pressurized gas on the external surfaces of the specimen at an elevated temperature. Therefore, density is an important parameter. In this project, all densities were measured based on Archimedes' principle following ASTM B 328.

Microstructure examination

The specimens were cut, polished and examined under a light microscope and a scanning electron microscope (SEM) with energy dispersive spectroscopy (EDS). To reveal their fiber structure, the specimens were cut either parallel or perpendicular to the fiber direction.

Mechanical property determination

The fiber monolithic WC is a very hard and tough material, and extremely difficult to machine. To simplify the mechanical property evaluation, we decided to determine the hardness and fracture toughness of the fiber monolithic WC and compare its properties with those of a conventional WC. Fiber monolithic WC has different properties parallel and perpendicular to the fiber direction. The properties in both directions were determined.

To determine hardness of the material, a FM WC specimen was cut along the fiber direction and also perpendicular to fiber direction. Both samples were polished and tested. Vickers hardness was testing using a micro hardness tester (1 kg load) and a Leco Model AVK-A hardness tester for 20 and 50 kg loads. To determine the final hardness number, five tests were conducted and averaged.

There are several approaches to determine fracture toughness of brittle materials, such as double torsion, indentation fracture, indentation strength, chevron notch, double-cantilever beam, single edge pre-cracked beam, and compression pre-cracking. Indentation fracture is a popular and low cost method:

$$K_{Ic} = 0.016 \frac{p}{C_o^{3/2}} \left(\frac{E}{H} \right)^{1/2}$$

where K_{IC}, P, C_o, E and H are the fracture toughness, the load, the crack length from the center of indent to the crack tip, the Young's modulus, and the Vickers hardness.

MACHINING

To produce drill bit inserts for field tests, the consolidated specimens were sent back to ACR and machined to specifications there.

The disc cutter prototype with WC inserts was sent to The Robbins Group for final

machining. Machining was difficult because the disc material was very hard. After discussion with the machinist, a fixture was developed to aid machining. The disc was machined to its near final dimensions

DRILL BIT INSERT FIELD TEST

Superior Rock Drill Bit Company provided a great help in testing the FM drill bit inserts. The inserts were placed on 16½ inch diameter rotary tri-cone drill bits manufactured by Superior Rock Drill Bit. Three inserts were placed on three 16 ½" diameter rotary tri-cone drill bits. The inserts were on the outermost row of the bit (i.e. the gauge row, also called the heel). The drills drilled taconite rock until the bits were completely worn out. Each test bit drilled in excess of 2,000 feet before the bit was discarded. Taconite is a very hard and abrasive rock, generally greater than 50 ksi uniaxial compressive strength, which presents a challenging environment for drilling.

FABRICATION OF CONVENTIONAL WC/CO DISC CUTTER INSERTS

Conventional WC/Co inserts are produced by sintering compacts of mixed WC and Co powder. We followed the same procedure to make the inserts for the 6.5" disc cutter prototypes. We purchased a ready mixed WC/Co powder (GWC-10, 90% WC and 10% cobalt) from Alldyne Powder Technologies. A die with a top and a bottom punch was designed and fabricated as shown in Figure 6a. The die was filled with the WC/Co powder mixture then pressed by a hydraulic press. The top piece in Figure 6b shows a compact ejected out of the die after hydraulic pressing. The compact was sintered at 1350°C for one hour in a vacuum furnace. The bottom piece in Figure 6b shows an insert after vacuum sintering. Ten inserts were then bonded together using epoxy to form a WC ring with the help of an auxiliary tool as shown in Figure 6c.

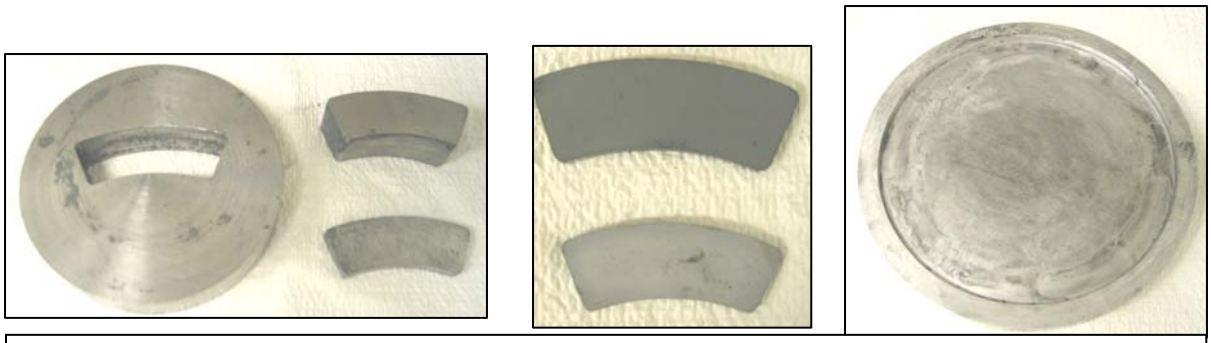

Figure 6. Conventional WC/Co disc cutter insert fabrication. (a) insert die with top and bottom punch; (b) top: WC/Co insert piece from die, bottom: WC/Co insert piece after sintering; (c) tooling to form insert ring for disc cutter

We found in experiments that the WC insert would react with steel at an elevated temperature, which may result in cracking of a WC insert. An intermediate coating could prevent this from happening. We used two coatings in this project. In the first coating, a 325 mesh CoCr powder was mixed with water and a glue to form a slurry. A brush was used to apply the slurry on the surfaces of an insert. After air drying, the CoCr powder adhered to the insert. In the second coating, a 5 micron Co powder was used. The finer powder gave a better control of the slurry viscosity and resulted in a more homogeneous coating.

HEAT TREATMENT

The H13 steel used in the disc cutters requires a heat treatment to gain high strength. The heat treatment was conducted in a vacuum furnace following the standard heat treatment procedure for this material. The heat treatment procedure is given below.

- heated at 600°C/hr from 25°C to 1020°C
- held for one hour at 1020°C for austenitizing
- argon quench from 1020°C to 25°C
- heated again at 600°C/hr from 25°C to 600°C
- held for two hours at 600°C for tempering
- furnace cooled to 25°C

FABRICATION OF ROCK DISC CUTTER SECTIONS

We divided the disc cutter prototyping into two phases. In the first phase, only sections of 6.5" and 17" rock disc cutters were made. The section samples have the same cross section shapes and dimensions as those of the circular disc cutters but are straight in length as shown in Figure xx. The purpose was to reduce the risk of failure and development costs. Full size prototyping is expensive because of the tooling, large amount of powder, model making, insert preparation, encapsulation, and HIPping. Section samples gave a good representation of real size prototypes.

The section samples were produced using the CastCon process. First, the aluminum patterns for making sand molds for 6.5" and 17" sections were designed and fabricated. To make a sand mold, the mixture of sand and a cold set organic binder was prepared and compressed against the pattern. After curing, the mold was separated from the pattern. A WC insert was produced and coated as described in **Fabrication of Regular WC Inserts**. The coated WC insert was placed in the mold and the mold was closed with a sand cover. A metal powder was then charged into the sand mold through a hole in the cover. The mold was encapsulated by a metal container which had a stem. The metal container was then heated in a vacuum furnace to burn the organic binder in the sand mold. After cooling, the metal container was vacuum-sealed and HIPped in the Quick-HIP furnace as described in **Hot Isostatic Pressing**. After HIPping, the metal container was opened to release the part.

ROCK DISC CUTTER PROTOTYPING

The Robbins Group gave great assistance in this work. They provided the drawing of currently used 6.5" rock disc cutter for a tunneling machine. To produce the cutter using the CastCon process, we designed an aluminum pattern (see Figure 7) according to the drawing, with an estimate of 12% linear shrinkage caused by powder consolidation. The pattern was modified several times after the first use to correct the initial shrinkage estimation.

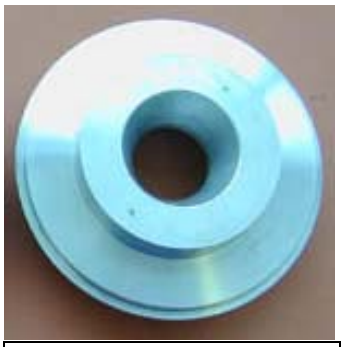

Figure 7. Disc Cutter Pattern Used to Manufacture Discs for Field Testing

The sand mold for making the 6.5" cutter prototypes consisted two pieces: top half and bottom half. Both half molds were made of sand and an organic binder mixture against the pattern. Green WC inserts were prepared by pressing WC powder in the insert die. The green WC inserts were prepared according to the method described in **Fabrication of Regular WC Inserts**. Metal containers were made of 1/8" thick low carbon steel by cutting and welding. To get ready for HIPping, a series of steps were followed. The circle WC insert was placed in the bottom half sand mold. The top half mold was glued with the bottom half mold. H13 powder was charged into the mold through a hole drilled on the top half mold. The mold was encapsulated in a metal container. The mold was heated in a kiln to remove organic binder. Various temperatures were used for this binder removal step, based on observations of the metal container and the resulting part. The metal container was sealed. The container was shipped to BodyCote for HIPping. The container was returned and opened to remove the disc cutter prototype.

Initially, phenol binder was used for sand molding. This binder needs sulfuric acid as a catalyst to cure the binder. In the prototyping tests, sulfur came out during HIPping and may form sulfide film on H13 powder surfaces. To reduce the sulfur contamination, we replaced the phenol binder with an oil based binder, LINO-CURE binder. The binder was ordered from Ashland Chemical. The sand and binder blended in the following ratio: 10% LINO-CURE A binder based on sand, 20% LINO-CURE C based on LINO-CURE A, 5% LINO-CURE B (catalyst) based on LINO-CURE A.

Binder burn-out was generally conducted in the same way. The can was heated in a box furnace first with a hose connecting the can's stem and a vacuum pump to evacuate the can. The can was heated according the procedure described below:

- 5°C/min from 25°C to200°C,
- held for 5 hours at 200°C,
- 5°C/min from 200°C to 500°C,
- held for 3 hours at 500°C, and
- furnace cooled.

After binder burn-out, the can was heated again for off-gassing in a vacuum furnace from room temperature to 1100°C according the procedure below:

- 10°C/min from 25°C to 500°C,
- 2°C/min from 500°C to 1100°C,
- held for 3 hours at 1100°C, and
- furnace cooled.

Then the can was sealed and shipped to Bodycote for HIPping.

We increased off-gassing temperature of cans from 500°C to 1050°C to reduce potential oxidation of H13 powder inside of a can. The off-gassing temperature was high and the samples were held for for 3 hours at that temperature. Severe oxidation took place on the can surface and scales formed. To reduce the risk of leaking due to potential oxidation through the thin steel, we identified a protective oxidation resistance glazing coating, named "CeramGuard" for heat

treating metal protection. This coating is supplied by A.O. Smith Corporation, Holton Road, Florence, KY 41042, Tel: 606-727-3500. CeramGuard is a group of coating for heat treating at different temperatures. We selected and ordered CG-27 according to A.O. Smith Corp's recommendation. CG-27 is supposed to be used around 1100°C. We applied this coating on the cans before off-gassing. To further reduce oxidation scaling, carbon was added into the off-gassing furnace to react with oxygen and reduce oxidation of cans.

During the prototyping trials, cracking and can distortion occurred. Several measures were taken to overcome these problems. In addition to the changes in binder and binder removal procedures, these measures included 1) examination of the vacuum system for off-gassing; 2) modification of the off-gassing system with a by-pass for argon purging a HIPping can before cooling; 3) quenching HIP cans to increase the cooling rate and reduce exposure time at most oxidation temperature ranges; 4) addition of more oxidation prevention agent into the sand mold; and 5) utilization of a metal mandrel to reduce shrinkage restriction of the disc center hole.

RESULTS AND DISCUSSION

This project focused on two drilling tools: drill bits and disc cutters. The drill bit portion of the project mainly focused on using a new fiber monolithic material as an insert to give better cutting performance. With the disc cutter, the focus was more on using a new manufacturing process, called CastCon, to make a better disc cutter. A number of problems were encountered in both the new material and the new process. These problems and the efforts to address them are described in this section.

FIBROUS MONOLITH (FM) MATERIAL PRODUCTION

This study used tungsten carbide and cobalt as the material to produce the fibrous monolith. As a result of this project, some aspects of the process for forming the initial FM performs were modified but the majority of this research focused on binder removal and material consolidation. This included looking for the most cost effective method to bring the FM material from a green preform to a ready-to-be-machined part.

The fibrous monolithic material was intended to be incorporated into both the drill bits and the disc cutters. However, based partly on the difficulties encountered in FM production and partly on the problems associated with CastCon operations, the FM disc cutter insert study was discontinued early in that portion of the program. For this reason, much of the discussion below relates to FM inserts for drill bits.

ACR fiber reinforced monolithic green preforms

Michigan Tech purchased tungsten carbide (WC) powder and cobalt powder and sent them to Advanced Ceramics Research. According to our collaboration agreement, ACR produced FM green bars and plates using these powders and their unique process and then shipped them to Michigan Tech for further experiments. The FM green bars were used for making the drill bit inserts and the plate inserts were tested for the rock disc cutter. The bars were about 5" long and 3/4" in diameter, the plates were about 2" wide, 3" long and 1/2" thick.

Two FM structures or compositions were designed and produced for this research. In each FM structure, the WC fibers, occupying 82.5 vol% of the monolith, were made of fine WC powder, cobalt powder and an organic binder. Each WC fiber in the monolithic was surrounded with a cobalt sheath which was made of cobalt powder and an organic binder. Table 1 gives the design structure and Figure 8 is a schematic drawing of the FM structure. ACR's process produces the fiber reinforced monolithic structure, but organic binder removal and consolidation are needed to make the monolithic preform strong.

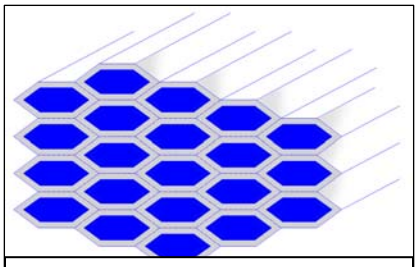

Figure 8. Schematic of the Core and Boundary Areas in a Fibrous Monolith. The test inserts were composed of a tungsten carbide core and a cobalt boundary

Table 1. Fiber Monolithic Composition	
Fiber Constituent	**Fiber Sheath Material**
90 vol% WC and 10% cobalt (82.5% of FM volume)	100% cobalt (17.5% of FM volume)
94 vol% WC and 6% cobalt (82.5% of FM volume)	100% cobalt (17.5% of FM volume)

Changes in the FM production process were made during the project, in response to problems encountered during binder removal and consolidation. Project participants identified three possible reasons for the problems encountered during binder burnout: polymer constituents of the binder system, high polymer loading, and the large size of the pieces, all of which contribute to binder removal problems. Selecting and optimizing the binder system would be a time consuming process involving many experiments with no guarantee of success. It was decided to try other possible improvements and avoid changing the binder system at this time. Two changes were made in the FM production stage to see if these had any impact on the problems.

Initially, ACR produced the FM green rods using two passes of extrusion, followed by a unidirectional warm pressing in a cylindrical die. It was possible that the unidirectional pressing lacked the transverse force necessary to ensure good bonding between fibers. ACR suggested the addition of warm isostatic pressing (WIP) after unidirectional pressing. The WIP was carried out at 100 psi and 140°C by a subcontract company. No obvious improvement was observed.

The fiber bundle surface may have less polymer due to nozzle “smearing” during extrusion. The subsequent unidirectional warm pressing does not create sufficient relative movement between adjacent fiber bundles; therefore bonding between fiber bundles may not be sufficient to hold them together during burnout, causing delaminating. For that reason, unidirectional warm pressing was replaced with a third pass of extrusion. The large deformation of fiber bundles may create better bundle bonding. A green rod was produced through three passes of extrusion by ACR. Again, no significant improvement was seen.

Since these FM production process changes had no apparent impact on the problems, efforts continued to modify the binder removal and consolidation process. Appendix A contains a list of the various FM samples that were generated during the project.

Pressureless Sintering

Pressureless sintering was our first choice for consolidating the FM inserts because of the potential low production cost. We hoped that the FM inserts could be heated in a single furnace to remove the organic binder and reach zero porosity as well.

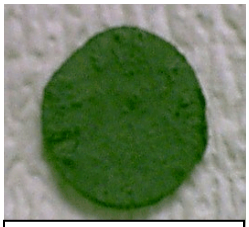

Figure 9. FM sample after pressureless sintering

Two pressureless sintering tests were conducted in a vacuum furnace. The samples were thin pieces sliced from an ACR green bar. The first sample was heated at a rate of 1°C/minute and showed bubbles and micro cracks on the surfaces. Figure 9 shows a picture of the sample after sintering. We believed that the heating rate was too high, which caused bubbling. The second sample was heated at a fairly slow rate of

0.5°C/minute. This sample also showed bubbles and micro cracks on the surfaces. These initial tests indicated difficulty in removing the binder without defects.

Hot pressing tests

If bubbles and cracks always occur during a binder removal process, hot pressing is a way to close the bubbles and cracks at an elevated temperature under pressure.

Binder Removal and Consolidation

Two hot pressing tests were conducted to combine binder removal and consolidation. The first sample was heated to 1100°C then pressed. After cooling and opening, the sample had to be scraped out of the graphite die. It appeared that the sample became a partial liquid or paste at some point during the hot pressing operation. The second hot pressing test was conducted at a reduced temperature of 950°C, other conditions remained the same. The second sample looked very similar to the first (see Figure 10). Neither case should happen because the melting point of the material is over 1200°C. The temperature reading was checked and it was correct.

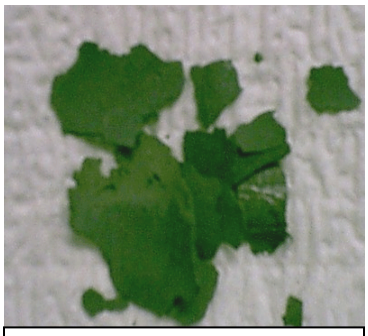

Figure 10. FM sample after hot pressing

One possibility was a high sulfur content in the organic binder which reacted with cobalt powder, forming a low melting temperature sulfide. When ACR prepared the FM material, organic binders were mixed with WC and cobalt powders to produce the fiber reinforcement structure. The residual sulfur from the organic binder might react with the cobalt matrix to form a low melting point sulfide.

To test this hypothesis, we examined the microstructure and sulfur content of Specimen ACR-B1-S-1 (a sample from the first sintering test). Two typical regions of Specimen ACR-B1-S-1 were examined. One region included a large hexagonal inclusion (greater than 1mm). Energy Dispersive Spectroscopy (EDS) analysis indicated that this inclusion contained 97.52% cobalt, 2.47% tungsten and 0.01% sulfur. The adjacent matrix contained 4.67% cobalt, 95.33% tungsten and 0.01% sulfur. Many small hexagonal cells were identified, which are the cobalt coated WC fibers. Many cracks were also seen. Figure 11 shows another region of inhomogeneous appearance. The hexagonal "inclusion" contained 13.03% cobalt, 86.95% tungsten and 0.02% sulfur. Many cracks and pores were seen. The examinations indicated that the sulfur content in the specimen was not high. The change in the FM during hot pressing was not caused by residual sulfur.

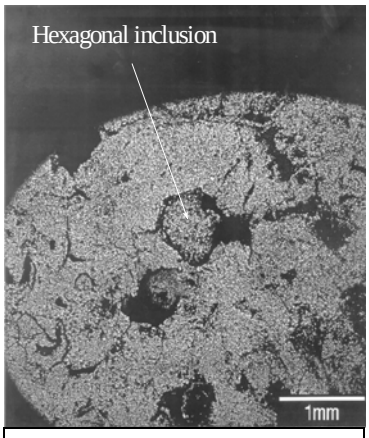

Figure 11. FM sample microstructure

The microscopic evaluation also indicated that there was some inhomogeneity in the specimen which should be controlled during preparation of the fiber reinforced material. The cracks could be caused by the internal stress between the fibers and the

matrix during heating due to different thermal expansion rates and binder evaporation. As a result of this observation the FM process changes noted earlier were implemented.

If residual sulfur did not cause the material changes at lower consolidation temperatures, another possibility was the low strength of the fiber reinforced material when the temperature range was between the binder melting point and binder evaporation. Although the pressing pressure was very light, it could be that the sample could not bear the weight of top graphite punch. We decided to separate the binder removal and the consolidation processes and try hot pressing again to consolidate the sample.

Consolidation Only

Specimen ACR-B1-S-1, which had been used in the pressureless sintering tests so the binder had already been burned off in the vacuum furnace, was used to perform the next series of hot pressing tests. The specimen was hot pressed to 900°C. Porosity and cracks remained in the specimen after the hot pressing. The specimen was then reheated at the same conditions to 950°C and held for 1.5 hours. Porosity and cracks still could be seen in the specimen. The specimen was reheated again to 1000°C, 1050°C, 1100°C and 1150°C and checked after each treatment. After hot pressing at 1150°C, the original cracks disappeared. The specimen (ACR-B1-HP-8) was then mounted and polished. Hardness and fracture toughness were then examined.

Vickers Hardness and Fracture Toughness

Vicker's hardness of Specimen ACR-B1-HP-8 was determined using a micro hardness tester (small load) and a regular hardness tester (larger load). This specimen had an average Vickers hardness of 1157, as shown in Table 2. The presence or absence of cracks radiating from the tips of the indent during the hardness tests is an indication of the fracture toughness of a material (indentation fracture method).

Table 2. Vicker's Hardness of ACR-B1-HP-8

Sample location	Micro hardness (1 kg load)	Hardness (20 kg load)	Hardness (50 kg load)
A	1274	1283	1118
B	1139	1095	1030
	No cracks	No cracks	No cracks
average Vicker's hardness of Specimen ACR-B1-HP-8: 1157			

To provide a comparison, we used data from two conventional WC/Co cermet hot pressing experiments previously conducted for other research, both at 1100°C and 4 ksi. One specimen was made of 50 vol% WC powder and 50 vol% cobalt powder. The other was 80 vol% WC powder and 20 vol% cobalt powder. Table 3 lists their Vickers hardness and fracture toughness. The FM specimen is harder than the 50/50 and 80/20 WC/Co materials. Figure 12 shows the indentation cracks in the 80/20 WC/Co specimen. Figure 13 shows the indent of the 20 kg load on the FM specimen; no cracks were found. Although fracture toughness cannot be determined by the indentation method, it is obvious that the fiber reinforced monolithic sample should have much higher fracture toughness than the conventional materials.

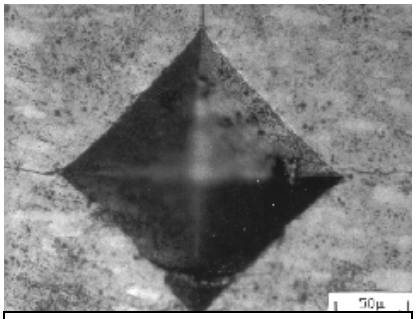

Figure 12. 80/20 WC/Co indent, has cracks (IMP-WC/Co-80/20-SW-HP)

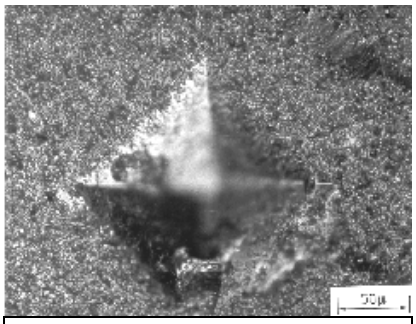

Figure 13. FM hardness indent, no cracks, 20 kg load (ACR-B1-HP-8)

The fiber reinforced monolithic specimen contains 82.5 vol% fiber which is made of 90 vol% WC and 10 vol% cobalt. In total (82.5% x 90% = 74.25%), the specimen contains 74.25 vol% WC and 25.75 vol% cobalt. In comparison with 80/20 WC/Co conventional material, the fiber reinforced monolithic is harder and has much higher fracture toughness in the fiber orientated direction.

Table 3. Comparison of Fiber Reinforced WC/Co and Conventional WC/Co Properties

Materials	Vickers Hardness	Fracture Toughness	Comments
50/50 WC/Co	909	6.95 MPa·$m^{1/2}$	Long indent cracks under 20 kg load
80/20 WC/Co	1042	11.87 MPa·$m^{1/2}$	Long indent cracks under 20 kg load
Fiber Reinforced WC/Co (74.25/25.75 WC/Co)	1157	n/a, but much higher	No indent cracks under 50 kg load

FM Material Fabrication

The hot pressed FM material showed reasonably good mechanical properties. However, hot pressing is used for single part consolidation and is not suitable for massive production. The next choice of pressure assisted consolidation is hot isostatic pressing (HIPping). It can treat hundreds or thousands of parts in one furnace run. Based on the experience gained from the hot pressing tests, it is better to separate the two processes of binder removal and pressure assisted consolidation. To make a FM insert strong enough for handling after its binder is removed, a pre-sintering step is necessary.

FM insert fabrication trial started with ACR's green bar manufacture. More cobalt and WC powder were purchased and shipped to ACR. ACR produced several pieces of 5" long and 3/4" diameter FM green bars and delivered to Michigan Tech. Michigan Tech cut the bars into pieces and machined the pieces to near button insert shape with consideration of later shrinkage.

The microstructure of the first batch of fiber reinforced material was not homogeneous. ACR changed the recipe and optimized the extrusion procedure for the second batch. The second batch contained 94 vol% WC + 6 vol% cobalt in the cells and 10 vol% cobalt in the shells. ACR

produced green bars 0.865" in diameter and 4" in length from the second batch of material. Cross-section examinations showed great improvement in extrusion homogeneity.

Binder removal and pre-sintering tests

Previous tests showed that binder removal and pressureless sintering resulted in bubbles and cracks on surfaces and bodies. However, we did not want to give up this effort because of the potential benefits of low production cost and difference in specimen shape.

The tests started with insert free standing trials. The inserts were heated in a vacuum furnace following a predetermined heat procedure. The binder in the fiber reinforced material needs to be removed by slow burning. The space left over by the binder need to be closed by sintering. A series of tests were conducted at very low heating rates and sintering at different temperatures to see if the bubbling and cracking problem can be solved.

Table 4 gives the binder removal and sintering test results. Slowing the heating rate did reduce cracking as seen in comparison of ACR-B2-SIN-1 with ACR-B2-SIN-2. However, cracks still appeared even at a very slow heating rate of 0.25°C/min. A high sintering temperature (1300°C) resulted in relatively large channels.

Table 4. Shrinkage and Porosity

Specimen	Processing	Ini. Thick in	Ini. Dia. In	Fin. Thick in	Fin. Dia. in	Thick Shrink %	Dia. Shrink %	Dry Wt g	Impreg. Wt g	Satura. Wt g	Wire Wt g	App. Poros. %
ACR-B2-SIN-1	25-500°C: 1°C/min 500°C: 30 minutes 500-1100°C: 10°C/min 1100°C: 60 minutes	0.235	0.865	0.233	0.820	0.85	5.2	16.4664	15.3867	16.9680	.2617	27.22
ACR-B2-SIN-2	25-500°C: 0.5°C/min 500°C: 30 minutes 500-1100°C: 10°C/min 1100°C: 60 minutes	0.235	0.865	0.229	0.800	2.55	7.5	16.4682	15.2670	16.7835	.2617	17.73
ACR-B2-SIN-3	25-500°C: 0.5°C/min 500°C: 30 minutes 500-1300°C: 10°C/min 1300°C: 60 minutes	0.235	0.865	0.208	0.708	11.49	18.15	16.3180	15.3612	16.3781	.2617	4.70
ACR-B2-SIN-4	25-500°C: 0.25°C/min 500°C: 30 minutes 500-1250°C: 5°C/min 1250°C: 60 minutes	0.25	0.865	0.221	0.723	11.60	16.42	17.3701	16.3890	17.5005	.3216	9.10
ACR-B2-SIN-5	25-500°C: 0.5°C/min 500°C: 30 minutes 500-1100°C: 5°C/min 1100°C: 60 minutes	0.25	0.865	0.242	0.802	3.59	7.28	17.5668	16.5334	18.1313	.3216	29.41
ACR-B2-SIN-6	25-500°C: 0.5°C/min 500°C: 30 minutes 500-1100°C: 5°C/min 1100°C: 60 minutes	1.25	0.865	1.162	0.822	7.04	4.97	87.9490	81.3095	90.8000	.3216	29.06

Pimples, cracks, delaminating and bloating could be worse in larger specimens. To investigate the problems, several specimens of 1.25" in height and 0.865" in diameter, were heated in a vacuum furnace using very slow burnout rates.

Serious cracking, delamination and bloating occurred during binder burnout and sintering (Figure 14). Although cracking, delaminating and bloating was seen in the smaller specimen, the defects in the larger specimen were much more profound. Container HIPping should be able to close internal voids such as cracking and delaminating, but is not effective in closing surface cracks. It is necessary to solve the burnout problem to make the FM material useful.

Figure 14. Cracking, delaminations, and bloating resulting from binder burnout and sintering.

It was projected that the cracking, delaminating and bloating were caused by escaping vapor during burnout of the polymer binder in the FM green material. Slower heating should lower the vapor evaporation rate and reduce the occurrence of those defects. But after a slow burnout and sintering test, there was no significant improvement.

ACR burnout tests

ACR also conducted parallel burnout experiments in a vacuum environment.

ACR Experiment #1: Excessive cracking, delamination between fibers, obvious bloating in the radial and along the height, as well as, "pimples" on the surface. Most distinctive crack measured 0.7087 in. and 0.0394 in. wide (Figure 15). Most of the damage occurring in binder burnout is occurring below 200ºC.

Figure 15. Sample from Exp #1. Obvious cracks, quite large and distinct. Also see cracks along bundle boundaries on top and bottom surfaces.

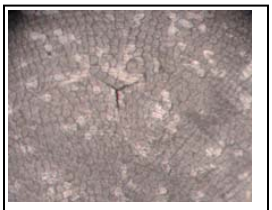

Figure 16. Exp #3, only noticeable bundle crack. Higher mag than Figure 9. Smaller microcracks probably exist.

ACR Experiment #2: Cracking and delamination between fibers not as pronounced as previous, bloating in the radial and along the height. Most distinctive crack measured 0.1969 in. and 0.0197 in. wide.

ACR Experiment #3: Very much improved. Few thin delaminations. Very small crack between fiber bundles (Figure 16). Cracks are thin but still quite long. Most distinctive crack measured 0.1969 in. and 0.0197 in. wide.

Dimensional changes in the specimens are given in Table 5.

Table 5. Dimension changes of specimens.

Experiment #	Change in Weight, g	Change in Height, in.	Change in Diameter, in.
1	-0.4350, (-0.77%)	0.0105, (1.05%)	0.0190, (2.44%)
2	-0.2380, (-0.42%)	0.0025, (0.25%)	0.0295, (3.79%)
3	-0.2600, (-0.46%)	0.0010, (0.10%)	0.0082, (1.06%)

In ACR's experiments, cracking and delaminating started below 200ºC. To determine the weight loss (evaporation rate) vs. temperature, a FM green material was sent to a lab to conduct a thermogravimetric analysis (TGA), running at 170 torr with heating rate of 1ºC/min. Figures 17 and 18 show the weight loss curves of the 94% WC + 6% Co material and the 94% WC + 6% Co (fiber) – Co (shell) FM material. The majority of weight loss took place in the temperature range from 350ºC to 600ºC. There was not much weight loss in the cracking and delaminating temperature below 200ºC according to the TGA curve. Therefore, escaping vapor was not the major cause of the burnout defects.

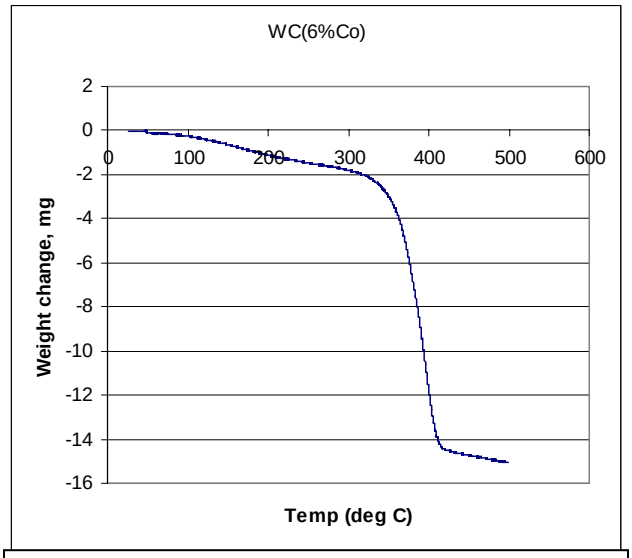

Figure 17. TGA analysis of 94% WC + 6% Co FM

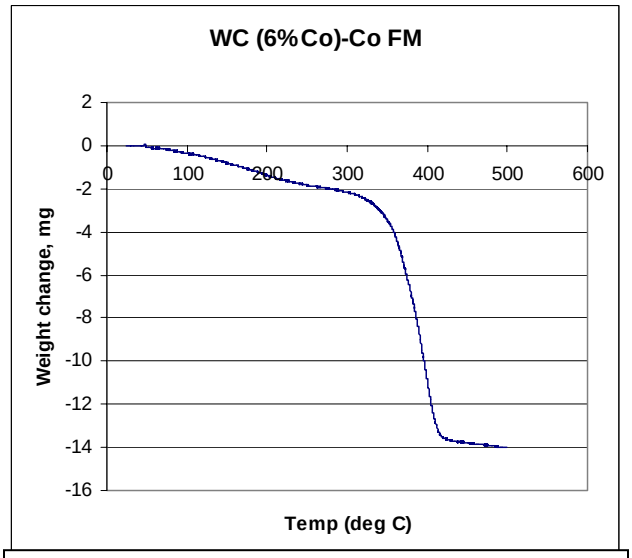

Figure 18. TGA analysis of 94% WC + 6% Co, with Co shell FM

It was probable that three reasons contributed to the burnout problem: improper binder system, large samples which hindered the escape of organic vapors, and inhomogeneity of the polymer binder surrounding the FM fibers. Switching binder would be a time consuming issue, involving many experiments with no guarantee of success. We decided to try other possible ways of improvement and avoid changing the binder system.

ACR four-day burnout test

ACR tried a four-day vacuum burnout test. The diameter of the part grew by 0.17% and the height increased by 4.5% during binder burnout. "Bloating" was evident; the part was no longer a right cylinder and tended to lean. The surface was smooth, with no pimples. There were no significant signs of out-gassing (gas entrapment) leading to bursting. (This defect looks distinctly different from the delams seen on this part.) There were obvious signs of fiber delamination around fiber boundaries. The fiber separation may be a direct result of the stress created as the sample bloated causing the fiber separation. Figure 19 shows the resulting sample.

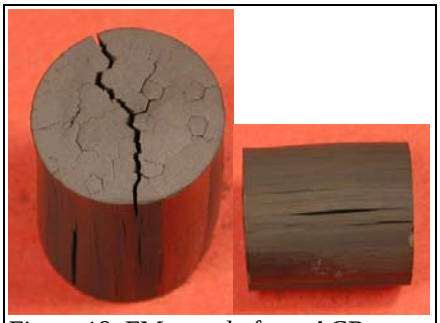
Figure 19. FM sample from ACR vacuum burnout test

Confinement burnout

Bloating and delaminating always occurred in FM burnout experiments, so something needed to change. Confined burnout could be an effective way to address these problems. In a confined burnout, a green FM piece was inserted into a steel container, surrounded with silica sand, and covered with a lid. The lid has a stem in the center. The entire container was heated in a vacuum furnace.

Figure 20 shows the result of confinement burnout of a button insert. No bloating, cracking and delaminating can be seen, a very encouraging result.

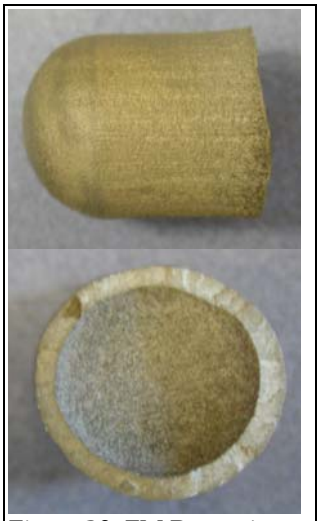

Figure 20. FM Button insert from confinement burnout

Thin plate burnout

Thin plate FM was needed as a hard insert for the disc cutters. Thin plate FM should have less tendency of burnout problem. A green FM plate of 3" x 2" x 0.5" was produced by ACR. This plate was cut and sliced into four pieces, with dimensions of 3" x 0.98" x 0.22". Two of them were burned and sintered in vacuum. One was laid on a flat graphite plate and the other was laid on a ceramic grill with hope of improving vapor escape. The third piece was burned and sintered in a steel container filled with sand (confinement burnout).

Figure 21 shows the burnout result of a thin plate laid on a flat graphite surface. Severe bloating, cracking and warping can be seen. Figure 22 shows the burnout result of a thin plate laid on a ceramic grill. Severe bloating, cracking and warping still occurred.

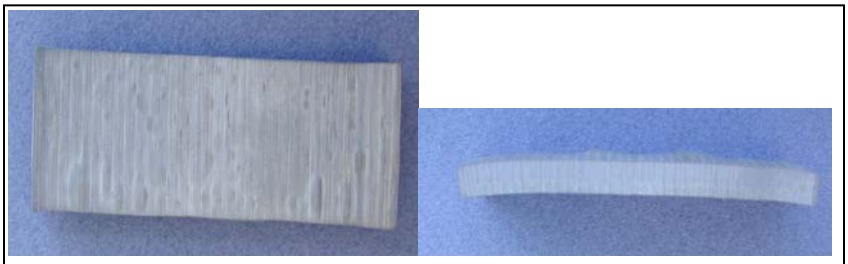

Figure 21. FM thin plate burnout on graphite

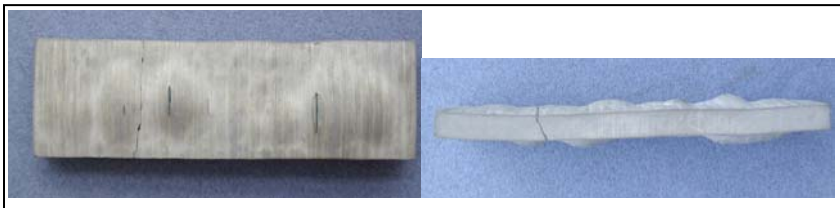

Figure 22. FM thin plate burnout on ceramic grill

Figure 23 shows the result of confinement burnout of the thin plate insert. The insert has no warping, no bloating, but many big cracks. The cracks seem different from previous burnout cracks and could be caused by restricted sintering, which means the shrinkage of the thin plate during sintering was blocked by the surrounding silica sand.

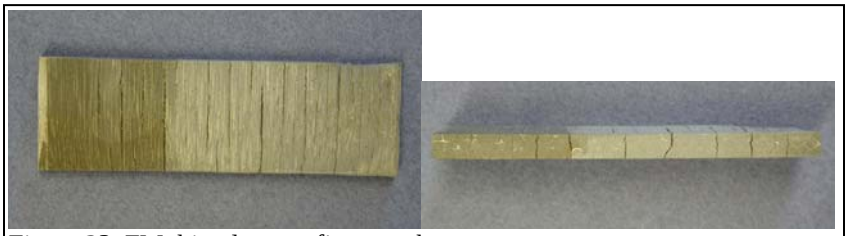

Figure 23. FM thin plate confinement burnout

HIPping

Hot pressing is limited by high temperature properties of die and punch materials. Pressure is normally less than 5 ksi. In this HIPping test, a button sliced from the 3/4" diameter fiber reinforced WC/Co preform (green piece), which was produced by ACR, was heated in a vacuum furnace from room temperature to 500°C at 1°C/min, held for 30 minutes, heated continuously to 1100°C at 10°C/min, held for one hour, and then furnace cooled. The sintered specimen was inserted into a steel container filled with sand. The steel container was then vacuumed and sealed. The container was HIPped in our Quick-HIP at 1100°C under 60 ksi. This specimen (ACR-B1-HIP-1) was cut, mounted, polished and examined.

Microstructure and mechanical properties of HIPped specimen

One half of specimen ACR-B1-HIP-1 was examined on the cross-section normal to the extrusion axis and the other examined on the cross section parallel to the extrusion axis. Each specimen was mounted, polished, tested using a Vicker's hardness tester and examined under a microscope.

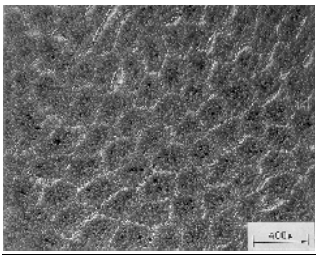

Figure 24. Hexagonal structure in HIPped FM

Figures 24 and 25 show the microstructure of the cross-section normal to the extrusion axis. The hexagonal WC/cobalt fibers are bundled by thin cobalt shells (Figure 1). Remaining pores can be seen in Figure 25, which mean that the HIPping temperature is not high enough to close the pores. Figure 26 shows the microstructure of the cross-section parallel to the extrusion axis. The cobalt lines (shells) are not parallel, the lines are much thicker in some regions and some lines are broken.

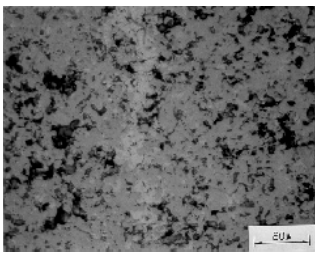

Figure 25. Remaining pores in HIPped FM

Table 6 gives the Vicker hardness test results. The hardness is lower than 1157 of the specimen produced by hot pressing. Microscopic observations of the indents show that many cracks were generated near the tips of indents or along the cobalt lines. Higher load indentation produced irregular indent shapes. It was obvious that some

material underneath the indents collapsed, which indicated that pores may exist there. In comparison with the specimen produced by hot pressing, the hot pressed specimen had no cracks after the indentation test but this HIPped specimen had many cracks. This means the HIPped specimen has much lower fracture toughness. All microstructure and mechanical test results indicate that the specimen is not fully dense and porosity is the main reason for the unacceptable mechanical properties. To solve the problem, increasing HIPping temperature should be the first choice to close the remaining pores.

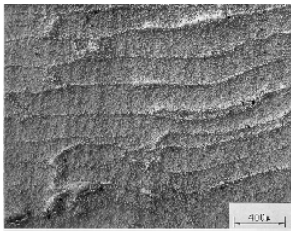

Figure 26. Microstructure parallel to extrusion axis in HIPped FM

Table 6. ACR-B1-HIP-1 Vicker's Hardness Test Results				
	Normal to Extrusion Axis		**Parallel to Extrusion Axis**	
	20 kg load	**50 kg load**	**20 kg load**	**50 kg load**
1	1213	1062	1154	925
2	1013	831	570	Collapsed
3	1063	1000	1134	Collapsed
4	1135		1105	
5	977		915	
Average	**1080**	**964**	**976**	

An experiment was conducted to find the appropriate HIPping temperature to consolidate the FM material. Specimens were prepared by slicing a green rod of Batch 2 FM material, which was produced by ACR. One specimen (ACR-B2-HIP-1) was 0.25" thick and 0.865" in diameter, and another (ACR-B2-HIP-2) was 1.25" tall and 0.865" in diameter. The smaller specimen was heated slowly in a vacuum furnace to burnout the binder and then heated continuously to an elevated temperature for sintering. After sintering, the specimen was inserted into a steel container and surrounded with sand. The container was covered using a steel lid with a steel stem in the center. The container was heated, vacuumed through the stem, sealed, and shipped to ACR. The HIPping was conducted at ACR in a mini HIP.

After HIPping, the container was returned to Michigan Tech, cut and opened. The specimen was checked for cracking, porosity, dimension change, microstructure, Vicker's hardness, and fracture toughness. The examination showed reasonably good mechanical properties.

Then the larger specimen was processed in the same way as the smaller specimen, using the identical burnout, sintering and HIPping parameters. After HIPping, the larger specimen was checked for appearance, dimension change and open porosity.

Table 7 shows the shrinkage and porosity for these specimens. They reached fully dense. Figure 27 shows the specimens. Figures 28 and 29 show the microstructure normal and parallel to the extrusion axis. No remaining porosity was observed. These results indicated that the HIPping temperature and pressure (1200ºC and 30 ksi) were appropriate. Table 8 gives the Vicker's hardness and fracture toughness test results for the smaller specimen. To compare with commercial WC insert's properties, a commercial 90% WC+10% Co insert was examined. Table 9 shows its hardness and fracture toughness. In comparison with the mechanical properties of

commercial WC material, the FM WC material shows better fracture toughness but lower hardness.

Table 7. Porosity and Shrinkage											
Specimen	Initial Thick In.	Initial Dia. In.	Final Thick In.	Final Dia. In.	Thick Shrink %	Diameter Shrink %	Dry Weight g	Impregnated. Weight g	Saturated Weight g	Wire Weight g	Approx. Porosity %
ACR-B2-HIP-1 (ACR-B2-SIN-5)	0.242	0.802	0.188	0.772	22.31	3.74	17.4827	16.5132	17.4828	0.3212	0.01
ACR-B2-HIP-2 (ACR-B2-SIN-6)	1.162	0.822	1.003	0.726	13.68	11.68	87.7304	81.5160	87.7304	0.2619	0
Processing Conditions	25-500ºCº: 0.5CºC/min 500ºC: 30 min. 500-1100ºC: 5ºC/min 1100ºC: 60 minutes HIPped at 1200ºC & 30 ksi										

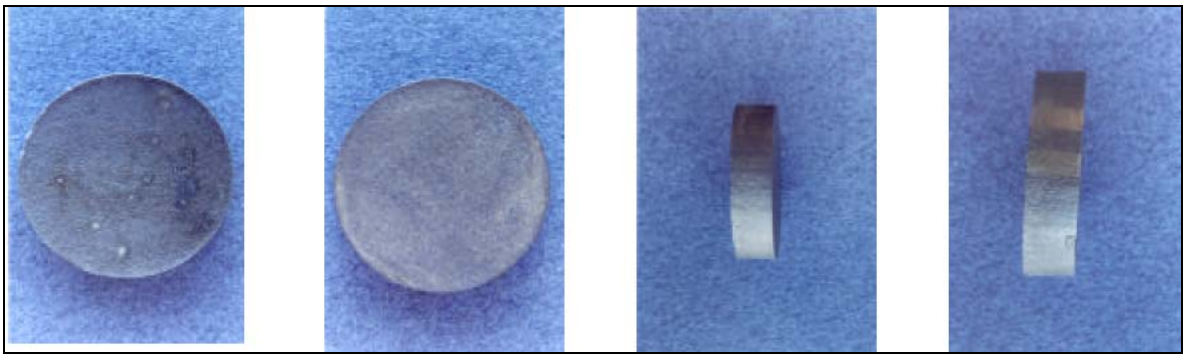

Figure 17. HIPped specimens ACR-B2-HIP-1 and ACR-B2-HIP-2

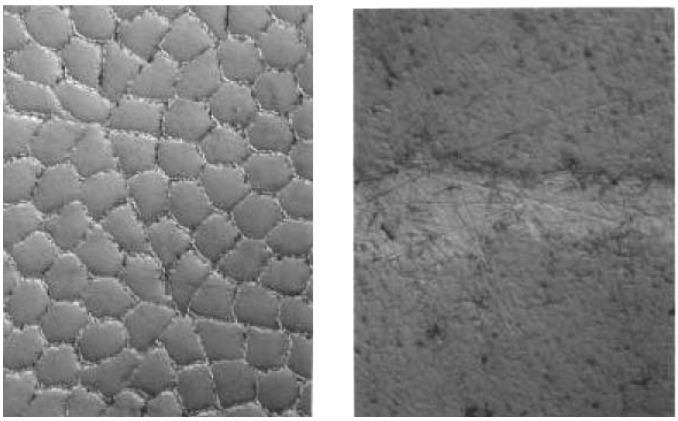

Figure 28. HIPped specimen microstructure normal to extrusion axis

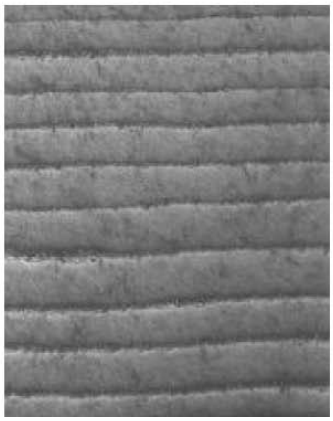

Figure 29. HIPped specimen microstructure parallel to extrusion axis

Table 8. FM Insert Hardness and fracture toughness.							
Sample	ACR-B2-HIP-1						
Date	Mar.18, 2002	P=load, N=newton	50kg	490.5N		E=	588.48 GPA
	Vicker's Hardness (Hv)	Direction	Index 50X	Crack 1 200X	Crack 2 200X	C_o (m)	Fracture Toughness $K=0.016 (P/C_o^{3/2})*(E/Hv)^{1/2}$ (MPa) $m^{1/2}$
Normal							
1	1026	Horizontal Vertical	30 32	32 16	0 0	3.61905E-04 3.42857E-04	8.72 9.46
2	1093	Horizontal Vertical	31 30	0 0	0 0	2.95238E-04 2.85714E-04	11.47 12.43
3	1061	Horizontal Vertical	32 30	10 22	4 0	3.38095E-04 3.38095E-04	9.50 9.66
4	1063	Horizontal Vertical	29 29	17 41	0 23	3.16667E-04 4.28571E-04	10.47 6.77
5	1097	Horizontal Vertical	29 30	6 16	0 0	2.90476E-04 3.23810E-04	11.73 10.30
Normal Average	1068						10.05
Parallel							
1	987	Horizontal Vertical	30 31	41 0	0 0	3.83333E-04 2.95238E-04	8.16 11.84
2	1037	Horizontal Vertical	30 29	15 10	0 32	3.21429E-04 3.76193E-04	10.36 8.23
3	937	Horizontal Vertical	30 30	42 0	37 0	4.73810E-04 2.85714E-04	6.09 12.43
Parallel Average	987						9.52
Total Average	**1027.5**						**9.78**

Table 9. Commercial Insert Hardness and fracture toughness							
Sample	Commercial Bit Insert						
Date	Mar. 26, 2002	P=load, N=newton	50kg	490.5N		E=	654.00 GPA
	Vicker's Hardness (Hv)	Direction	Index 50X	Crack 1 200X	Crack 2 200X	C_o (m)	Fracture Toughness $K=0.016 (P/C_o^{3/2})*(E/Hv)^{1/2}$ (MPa) $m^{1/2}$
Normal							
1	1231	Horizontal Vertical	29 29	12 5	20 24	3.52381E-04 3.45238E-04	8.74 9.01
2	1226	Horizontal Vertical	29 29	12 20	19 10	3.50000E-04 3.47619E-04	8.84 8.92
3	1233	Horizontal Vertical	29 29	19 23	16 15	3.59524E-04 3.66667E-04	8.47 8.23
4	1237	Horizontal Vertical	29 29	15 15	29 19	3.80952E-04 3.57143E-04	7.75 8.56
5	1222	Horizontal Vertical	29 29	18 15	22 20	3.71429E-04 3.59524E-04	8.10 8.48
Normal Average	1230						8.51

Continued Study of Confinement Burn-Out

The experiments conducted in the first confinement burn-out concluded that confinement burn-out can solve the cracking and delamination problem. This success raised a hope of container-less HIPping. In container-less HIPping, the FM inserts after burn-out are sintered to a relative density higher than 95%, or to the level that internal pores are isolated. The remaining pores are closed by high temperature and high pressure gas acting on the external surfaces of inserts during HIPping. Because there is no metal container and seal required, container-less HIPping lowers the manufacturing cost dramatically. In this effort, the experiments of confinement burn-out continued to find out the optimum burn-out and sintering conditions for achieving relative density higher than 95%.

Three green FM inserts were tested. The three inserts were produced by cutting and machining a FM green rod which was made by ACR through a three pass extrusion. The first insert was embedded in a clay crucible filled with silica sand. The confinement came from the weight of sand on top of the insert. The heating rate was 0.5°C/min from room temperature to 500°C, followed by a sintering to 1100°C for two hours. Figure 30 shows the appearance of the insert after burn-out and sintering. Bloating is observed

Figure 30. FM Insert after binder burnout and sintering in sand confinement

on the upper part of the insert which suggests that confinement by sand weight alone is not sufficient.

The second insert was embedded in a metal container completely filled with silica sand and covered by a metal lid. The confinement was introduced by the metal container's resistance to expansion. The burn-out rate was also 0.5°C/min from room temperature to 500°C, followed by sintering to 1100°C for two hours. Figure 31 shows the appearance of the insert after burn-out and sintering. From these figures, we can see that FM material penetrated into the surrounding sand and there are many large internal pores. We think that it is due to coarse surrounding sand (20-28 mesh) giving little resistance to the penetration of FM material into the sand interspaces.

Figure 31. . FM Insert after binder burnout and sintering in container confinement with coarse sand

The third insert burn-out and sintering process was the same as the second one except a much finer (100-150 mesh) graphite sand was used as the surrounding material. Figure 32 shows the resulting insert. There are no cracks or bloating. A density measurement revealed that the insert was not fully dense. The insert absorbed water. The insert was then sintered to 1200°C for one hour and the density checked after cooling. The insert still absorbed water. The insert was sintered to higher temperatures in 50°C increments, up to 1350°C for one hour. Then, the insert absorbed no water, assuming over 95% relative density. The insert was then sent to ACR for 1300°C and 30 ksi container-less HIPping.

Figure 32. . FM Insert after binder burnout and sintering in container confinement with finer sand

FM Material Container-less HIPping

An FM specimen after sintering at 1350°C for one hour reached the desired density (absorbing no water). This specimen was sent to ACM for container-less HIPping at 1300°C and 30 ksi. After HIPping, this specimen was returned to MTU (Figure 33). There was no noticeable difference in appearance between before and after HIPping. The specimen was cut and polished to expose its microstructure, as shown in Figures 34 and 35. Porosity obviously remained. The remaining porosity indicated that either HIPping did not help to consolidate the specimen further after sintering or that the remaining pores were still interconnected to external

Figure 33. FM insert after container-less HIPping

surfaces so that pressurized gas could not act on the specimen's external surfaces to build a pressure difference inside and outside the specimen during HIPping.

Figure 34. Microstructure of FM insert after container-less HIPping

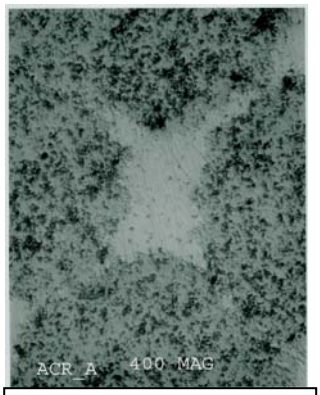

Figure 35. Microstructure showing porosity of FM insert after container-less HIPping

Another FM specimen was sintered at 1350°C for one hour in a vacuum furnace and followed immediately by a 50 ksi argon pressurizing at the same temperature for another hour in the same furnace. The specimen showed significant shrinkage after the sintering/low pressure HIPping, as shown in Figure 36. The specimen was cut and polished. It was surprisingly found that the FM microstructure disappeared, as seen in Figure 37. The WC grains seemed bigger than their original size and were distributed randomly as a conventional WC microstructure. This suggests that the WC particles recrystallized at the high temperature of 1350°C, which is near the melting point of the matrix cobalt. The specimen reached near full density.

These two experiments imply that the FM material cannot achieve both full density and maintain the FM microstructure using container-less HIPping. Container HIPping may be necessary to consolidate this type of material.

Figure 36. FM insert after sintering and low pressure HIPping

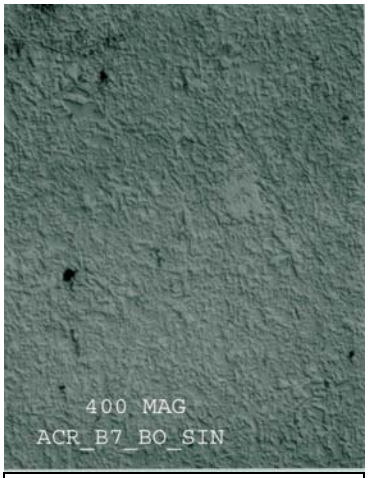

Figure 37. Microstructure of FM insert sintering and low pressure HIPping

DRILL BITS

One project objective was to develop an effective means for producing rock drill bits that would last longer, increase energy efficiency and penetration rate, and lower overall production costs. This was to be done based on a combination of fiber monolith and CastCon technologies. The fiber monolith was tested for the cutting inserts and these inserts were to be incorporated into the CastCon process testing to make a drill bit. Initial project efforts focused on producing inserts for testing, then shifted to developing cost effective production methods for the FM inserts, as described in the previous section. Ultimately, field testing showed that the FM insert did not perform acceptably and therefore use of the CastCon process to manufacture a drill bit which included the insert was not pursued.

Insert machining

Four HIPped inserts were sent to The Robbins Group and they arranged the grinding to specified dimensions (0.905" in height and 0.6895" in diameter, with a 60 degree tapered point). The grinding was done by a ceramic machining company in the Seattle area. One of the inserts had minor surface cracks. This insert was produced by free standing burnout and sintering, followed by container HIPping. The HIPping does not close all surface cracks generated during the free standing burnout. Figure 38 shows the machined insert.

Figure 38. Two Views of the Round Top Drill Bit Insert. The insert is typical of the cutting tools placed on rotary drill bits. (Orientation of the fibrous monolith was right to left, from the round top to the flat base.)

Hammer Test for Drill Bit Inserts

Tungsten carbide is a brittle material that is susceptible to shattering on impact. To test for threshold brittleness, WC insert manufacturers (e.g. Ingersol Rand) and WC insert users (e.g. the Robbins Company) employ a simple, qualitative, yet effective test: hit the WC insert with a heavy sledge hammer (~16 pounds). An insert that does not survive the impact will be judged overly brittle and suspect for drilling and rock cutting applications. An insert that survives the test will require further testing.

Figure 39. Close-up of the Hammer Induced Damage to the Bottom of the Insert. The damage appears to mostly occur in each fibrous monolith cell. Compare the damaged to the undamaged cells

One insert was subjected to several blows with a 16 pound hammer. The insert exhibited minor surface damage at the impact points (Figure 39). The damage appears to be related to the cells in the FM structure. The damage appears as pits that are approximately the same size and pattern as the intact cells.

Drill Bit Insert Field Tests

The field tests were assisted by Superior Rock Drill Bit Company. Three inserts were placed on three 16 ½ inch diameter rotary tri-cone drill bits (Figure 40) manufacture by Superior Rock Drill Bit. The inserts were on the outermost row of the bit (i.e. the gauge row, also called the heel). The drills drilled taconite rock until the bits were completely worn out. For example, the rotary cone bearing in one bit lost all of the bearing rollers. Additionally, all three drill bits had their heel rows completely worn away, and nothing was left of the inserts, either new or current art on those rows.

Figure 40. Drill Bit Used in Field Tests. The Superior Rock Bit test bit was 16.5 inches in diameter. The "C" carbide was the new art test insert.

Each test bit drilled in excess of 2,000 feet before the bit was discarded. Taconite is a very hard (generally greater than 50 ksi uniaxial compressive strength) and abrasive rock, which presents a challenging environment for drilling.

Test Results

The FM carbide-cobalt inserts wore away faster than current art inserts, although the wear rate was not significantly much higher (Figure 41). The relatively soft boundary seems to cause the wear. The boundary layer is composed of cobalt, which is significantly softer than the very hard tungsten carbide core.

Figure 41. Current Art and Fibrous Monolith Inserts on a Worn Superior Rock Bit. The FM insert in the center has worn more than the current art inserts, although no brittle failures have occurred like the small chip on the insert to the left of the test insert.

Figure 42. Highly Worn FM Insert on a Worn Superior Rock Bit Drill Bit. Compare the worn insert (center) to the surrounding current art inserts. The two current art inserts at the top exhibit brittle failures.

It is important to note that there were no brittle failures such a chipping, cracking, and spalling. The FM material exhibited no brittle failure. However, the pitting failures seen in the hammer tests were similar to the appearance of surface of the worn insert on the drill bit (Figure 42). Together, these wear and failure patterns suggest that FM cell boundaries are at least partially involved in the failure and pre-mature wear.

Failure Analysis of FM inserts

The field test showed that the FM inserts failed earlier than the traditional WC inserts in a pattern of pre-mature wear. There was a thought of C-porosity existing in the FM material and being the major cause of weak mechanical property.

This thought was raised because the organic binder in the green FM may leave carbon in the material after the organic binder is burned-out. ACM checked the microstructure of a finished insert.

The finished insert was WC(6%Co)-Co FM material HIPped under 30 ksi at 1200°C. With help from ACR, it was polished to reveal the FM macrostructure. Initial observations were: There is good extrusion and consistent macrostructure throughout the part. The part was consolidated well and of high density.

Upon polishing, the part was viewed under a microscope to see if residual carbon was present (Figure 43). Residual carbon is typically observed as black or dark spots. No significant amount of C-porosity was observed in the fiber monolith. In stating that, however, ACR does not have the ability to rate the amount of C-porosity present within the part as compared to the carbide industry. Each supplier of carbide standardizes a technique and reference from which a piece is quantified as being saturated by C-porosity, thus deemed unacceptable. However, the insert has less visible C-porosity than some of the sample inserts ACM have sectioned.

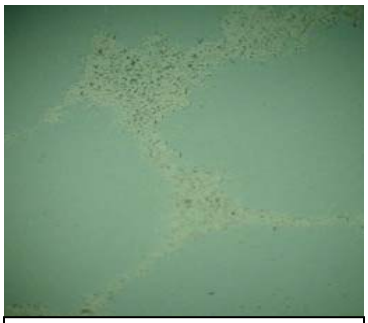

Figure 43. WC(6%Co)-Co FM after etching at 20X, to observe C-porosity in FM

The bottom surface of the insert was etched to reveal eta phase (darker spots in Figure 44). Eta phase is a carbon deficiency that results in more brittle carbide. As with excess C-porosity, eta phasing is also considered a defect in the carbide industry. There is very little eta phase present in the carbide core. However, there is eta phase present throughout the cobalt interface. This makes sense since there should be little or no carbon in the pure cobalt interface.

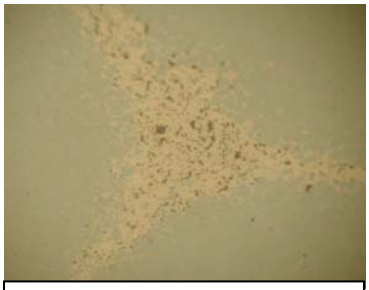

Figure 44. WC(6%Co)-Co FM after etching at 20X, eta phase (darker regions)

It is ACR's opinion that although C-porosity may be present, they do not think it was a major contributing factor. ACR does not have a valid method for quantifying C-porosity as industry and is basing its opinion on observation and comparison based on other FMs produced. It is also their opinion that the FM was of solid structural integrity, high density and regular FM structure. However, they think that the increased wear may be due to the soft and ductile nature of the cobalt interface. This material system may not be suitable for this application.

DISC CUTTER FABRICATION

The fabrication trial for the disc cutters used the CastCon process. The disc cutter fabrication was divided into two phases. In the first phase, only sections of 6.5" and 17" rock disc cutters were made. The section samples had the same cross section shapes and dimensions as those of the circular disc cutters but were short and straight. The purpose was to reduce the risk of failure and development costs. Full size prototyping is expensive because of the tooling, large amount of powder, model making, insert preparation, encapsulation, and HIPping.

Section samples gave a good representation of real size prototypes and material interaction problems were addressed at this smaller scale. However, nearly every aspect of scaling up to full size disc cutters encountered problems. Many of these were addressed during the research, but time and funding constraints halted the prototyping before all the problems were solved.

Fabrication of Disc Cutter Sections

The disc cutter sections were produced using the CastCon process. In the first trial, the WC/Co hardcore insert cracked after HIPping. One thought was that the differences of thermal expansion coefficient between the WC/Co insert and the adjacent body material created a large residual shear stress during cooling which caused cracking of the WC/Co insert. Subsequent experiments indicated that this assumption was wrong. The reaction of WC/Co with steel at high temperature was the real reason for the cracking.

In an effort to solve the cracking problem, several hot pressing tests were conducted with a WC/Co layer against different metal layers with or without an intermediate coating. In Figure 45, the WC/Co layer (with a long crack) is in direct contact with 316 stainless steel (top layer). Cracking occurred in the WC/Co layer. In the hot pressed specimen shown in Figure 46, WC/Co (bottom layer) was isolated from the 316 stainless steel with a CoCr layer and no cracking occurred. More tests indicated that a WC/Co layer would crack if in contact with a low carbon steel such as 316 SS, but cracking would not occur if the WC/Co layer was in contact with a high carbon steel such as D7.

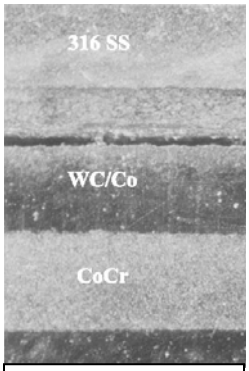

Figure 45. WC/Co without insulation, with crack in WC/Co layer (IMP-WC/Co-88/12-SW-HP-2)

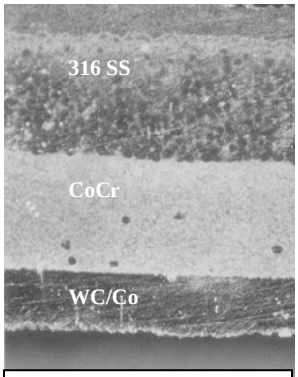

Figure 46. WC/Co with CoCr insulation, no cracking (IMP-WC/Co-88/12-SW-HP-3)

A microstructure examination of the contact region between the WC/Co layer and the 316 SS layer revealed a reaction zone. It appeared that the diffusion of carbon in the WC/Co layer to the 316 SS layer was the major cause of the cracking. If a third material isolated the two materials, cracking was avoided.

After the hot pressing tests, two more 6.5" disc cutter sections were produced to check whether the cracking problem was caused by a reaction, and fixed by the addition of CoCr insulation, or whether the cracks were a result of residual stresses due to differences in thermal expansion coefficients. One section had a WC/Co insert with a CoCr coating, and the second section had two separated WC/Co hard cores without a coating but with reinforcement tendons in the middle and two ends. The idea of adding the three reinforcement tendons was to generate residual compression stress during cooling to avoid cracking in the WC/Co hard cores. The steel reinforcement tendons have a larger thermal expansion coefficient than the WC/Co hard core material, therefore subjecting larger elastic shrinkage. Both sample sections were HIPped at 1100°C under 60 ksi. A large crack still occurred in the uncoated WC/Co hard core (see Figure 47). No cracks were found in the section where the WC/Co insert was coated with CoCr.

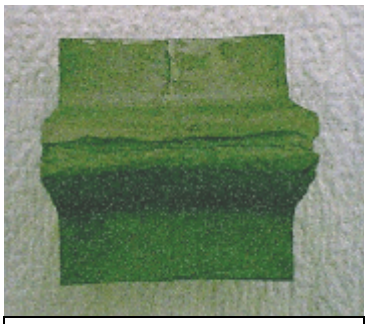

Figure 47. Large crack in WC/Co hardcore of 6.5" disc section prototype, (IMP-6.5Sec-HIP-5)

Three more 6.5" disc sections and one 17" disc section were produced out of H13 powder after we determined the solution to the cracking problem. The three 6.5" sections contained a green FM insert, a hot-pressed FM insert, and a pre-sintered traditional 90% WC + 10% Co insert respectively. The second insert was hot-pressed by ACR under 5 ksi at 1300°C. The third insert and the insert for 17" section were pre-sintered in a vacuum furnace at 1350°C for one hour. All FM inserts were coated with CoCr coating for insolation. Figure 48 shows the produced sections. There were no cracks in any of the sections and the fabrication was very successful.

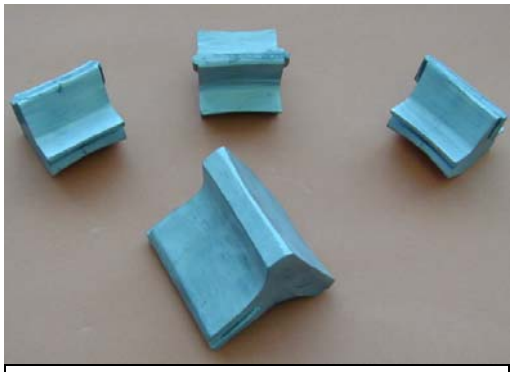

Figure 48. Finished Disc Cutter Sections

The sections were cut to expose their structures, as shown in Figure 49. The 17” section with the conventional WC insert is labeled **1**, a 6.5" section with a "green" FM WC insert **2**, a 6.5" section with hot pressed FM WC insert **3**, and a 6.5" section with sintered conventional WC insert **4**. Figure 50 shows a closer view of each section, focusing on the insert. Microscopic evaluation (Figure 51) showed that the "green" WC insert obviously shrank and the hot-pressed WC insert shows a relatively coarse FM structure. The sintered conventional WC insert in the 6.5” disc section shows homogenous microstructure and good bonding with the H13 body.

There was a concern that the heat treatment normally required for an H13 steel may break the bonding between the WC insert and the H13 body since WC and steel have very different thermal expansion properties. To test the concern, the 6.5" section with the sintered conventional WC insert was heat-treated in a vacuum furnace using the procedure described in the Experimental Methods. This part showed cracks in the WC insert and along the boundary between the WC insert and the H13 body (Figure 52). We concluded that a barrier material may be needed to coat the WC insert surface to isolate its contact with H13 body as a buffer to reduce the thermal shock due to the thermal expansion differences. Cobalt was selected as the barrier material because of its good match with both materials and no potential to react to form a brittle intermetallic compound.

Figure 49. Disc cutter section pieces, the 17” section with the conventional WC insert is labeled **1**, a 6.5" section with a "green" FM WC insert **2**, a 6.5" section with hot pressed FM WC insert **3**, and a 6.5" section with sintered conventional WC insert **4**

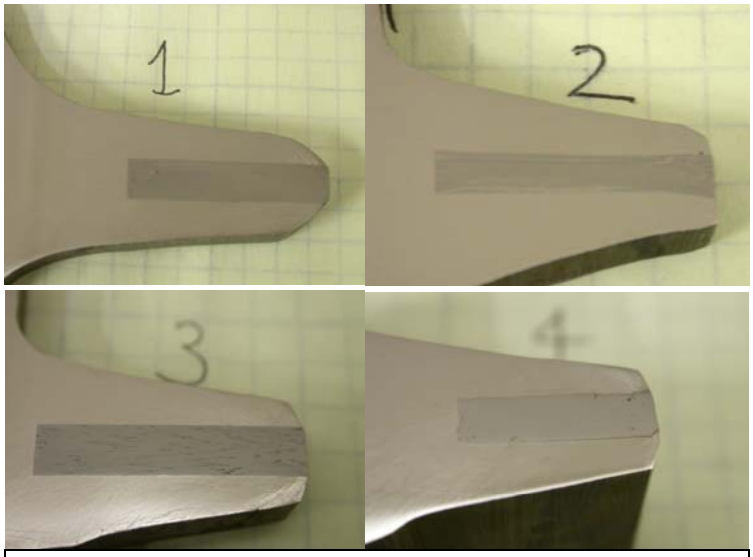

Figure 50. Closer views of disc cutter section pieces, focusing on the inserts.
17” section with the conventional WC insert is labeled **1**. A 6.5" section with a "green" FM WC insert **2** shows shrinkage. A 6.5" section with hot pressed FM WC insert **3** shows coarse FM structure. A 6.5" section with sintered conventional WC insert **4** shows good bonding.

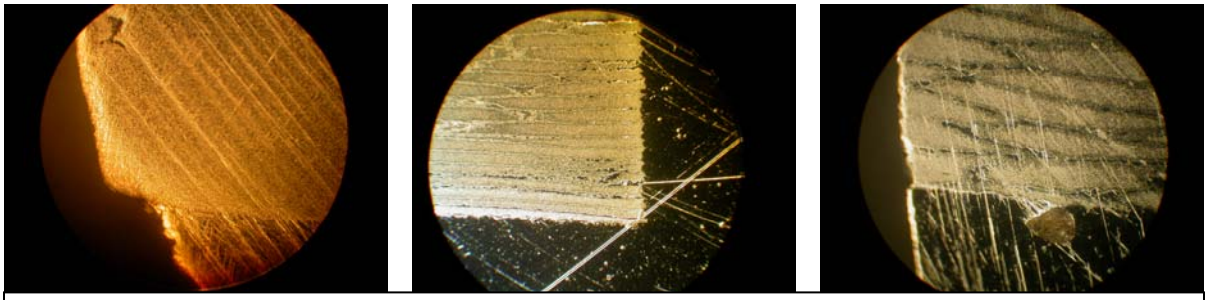

Figure 51. Microstructure of inserts in 6.5” disc cutter sections. Left – sintered conventional WC insert (**4**) shows homogeneous microstructure, Center – “green” FM WC insert (**2**) shows shrinkage, Right – hot pressed FM WC insert (**3**) shows coarse FM structure.

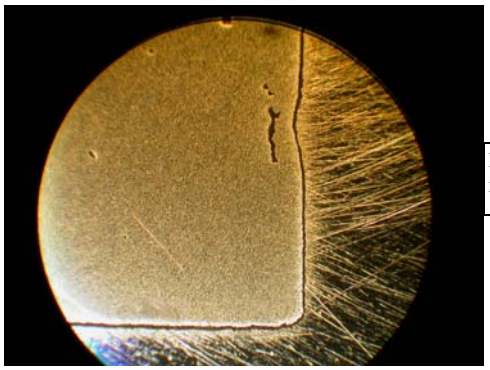

Figure 52. Microstructure of heat treated sintered conventional WC insert in 6.5” disc section (**4**) showing cracks.

6.5" Disc Cutter Prototype Manufacturing

Following the evaluation of the disc sections, prototype manufacturing of the 6.5” disc cutters began. At this point the project team, together with DOE, decided to drop the FM component in this portion of the project and focus on using conventional materials with the new CastCon process. The prototype manufacturing did not go smoothly. After five trials, each of which required modified tooling, additional metallic powder, mold making, insert preparation, encapsulation, and HIPping, the project funding and time had run out, without an acceptable 6.5” disc cutter prototype.

First prototyping trial

The first prototyping trial started with making three real size 6.5" disc cutters: one H13 only and two with WC inserts, using the CastCon process.

Three sand molds were made, two with WC ring inserts positioned in their centers. H13 powder then filled the rest of the sand molds. The sand molds were encapsulated in HIP cans and heated in a furnace to remove organic binder, then sealed. Unfortunately, the first can was blown badly due to the relatively high heating rate during the binder burn-out cycle (Figure 53). The heating rate was reduced when treating the remaining two cans. The cans still expanded slightly. The two cans were shipped to Bodycote for HIPping. The cans showed severe distortion after processing and the disc were distorted as well (Figure 54). Cracks can be seen on the H13 only disc cutter (Figure 55). Measurements indicated that the hub and cutting edge thickness needed to be increased.

Figure 53. Blown HIP can

Figure 54. Disc cutter from first trial

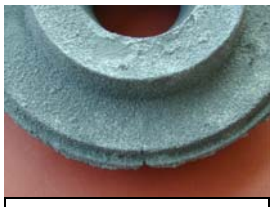

Figure 55. Crack on H13 only disc cutter

After examination of the cans, it appeared that the distortion was caused by expansion of cans during off-gassing in the vacuum furnace. During binder burn-out and off-gassing by heating, the gases in the sand mold were released and exhausted through a stem of the HIP can. The binder burn-out temperature went up to 1100°C in the final stage and the 1/16" thick steel of the HIP can was fairly soft at that temperature. If the gas generation rate is much higher than the rate at which gas escapes, the internal gas pressure of the can will be much higher than the outside pressure. We guessed that it was the case in our off-gassing process. The inside and outside pressure difference was greater than the strength of the can material at that temperature, resulting in can blowing or expansion. The can expansion generated free space, which allowed the mold sand to move in the can or to crack without constraint from the can. The sand mold movement and cracking destroyed the sand mold's capability of retaining the shape of its cavity which contained the metal powder.

Second prototyping trial

In the second trial, we reduced the heating rates during bind burn-out and off-gassing in an effort to solve the can distortion problem. The disc pattern was modified with four steel rings bonded on the needed surfaces to increase the hub and cutting edge thickness.

Figure 56. Blown HIP can with oxidation scaling

Both cans expanded slightly after high temperature off-gassing although a much slower heating rate was used. After HIPping, one can cracked and another blew (Figure 56). From our past experience, the cracked can was caused by free movement of graphite sand inside the can. A portion of the graphite sand moved over an insulation layer to contact directly with the steel can and reacted with the steel. The reaction formed a brittle iron carbide and the carbide cracked during cooling. The blown can was due to a small vacuum leak. The high pressure gas penetrated into the can during HIPping, and expanded the can upon a quick discharge of HIPping gas.

Figure 57. Rib defects on disc cutter

When removed from the cans, both discs showed severe defects as seen in Figure 57. It was obvious that both sand molds cracked and the metal powder flew into the big, thick cracks and formed the "ribs".

Third prototyping trial

Previous 6.5" disc prototyping trials encountered HIP can expansion or blowing problem during off-gassing in a vacuum furnace. We have decided to switch to a conventional kiln to do the binder burn-out and off-gassing job, using a vacuum pump to extract the volatiles and gases released from the sand mold inside the can.

The sand molds and HIP cans were made in the same way as before. To reduce oxidation of cans in a conventional kiln without inert or reducing gas protection, a carbon powder was used to cover and surround the cans. The binder burn-out and off-gassing temperature was also lowered to 500°C from 1100°C used in the vacuum furnace. After binder burn-out and off-gassing, the cans were sealed and shipped to Bodycote for HIPping.

Figure 58. Disc cutters from Trial 3

Two 6.5" disc cutters were made, one from H13 powder only and the other from H13 powder with WC inserts. The cans did not expand during binder burn-out and off-gassing. There were no leaks during HIPping. The discs after removal from the cans looked good as seen in Figure 58. The disc with WC inserts was sent to The Robbins Group for final machining. The disc was difficult to machine because the material is very hard. During the machining, two

problems were found. First, the disc hub was still not thick enough for machining to final specification. Second, micro cracks were present along the radial directions. The thickness problem can be solved by modifying the disc pattern in the future. We believed that the micro cracks were caused by the low temperature binder burn-out and off-gassing. From our past experience, the H13 powder surfaces could be contaminated if the off-gassing temperature is not high enough.

Fourth prototyping trial

Discs produced during the last trial were not thick enough on the disc hub section. Two 1/8" thick steel rings were prepared and bonded on the pattern hub to increase its thickness. We used the modified pattern to make the 6.5" discs this trial. Extra care was taken in preparing the HIP cans to guard against vacuum leaks.

The off-gassing temperature of cans was increased from 500°C to 1050°C in this trial to reduce potential oxidation of H13 powder inside the can. The off-gassing temperature was high and lasted for 3 hours at the temperature. Severe oxidation took place on the can surface and oxidation scales formed on the can. To reduce the risk of leaking due to potential oxidation through the thin steel, we identified an oxidation resistance glazing material, named "CeramGuard" for heat treating metal protection. We applied this coating to the cans before off-gassing. To further reduce oxidation scaling, fly ash residual carbon was added into the off-gassing furnace outside of the cans to react with oxygen and reduce can oxidation.

Figure 59. Disc cutter from Trial 4 with crack (center of photo, top to bottom)

The cans did not leak during HIPping. It showed that our measures to solve the can vacuum leak was effective. The discs produced using the modified pattern achieved fairly good near net shapes and were larger enough for machining to final specifications.

Although the off-gassing temperature was increased from 500°C to 1050°C, cracking still can be seen on the disc radial directions (Figure 59) and contact regions of H13 steel and WC inserts. There appear to be several factors contributing to the problem: 1) powder oxidation, 2) low ductility of H13 material, 3) residual stress caused by the shrinkage restriction of the disc center hole, 4) improper vacuum during off-gassing, 5) too slow cooling after off-gassing causing re-oxidation, and 6) too little oxidation prevention agent added into the sand mold.

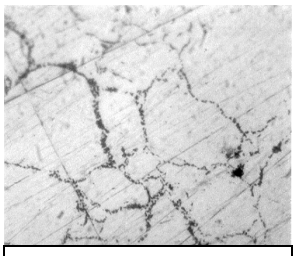

Figure 60. Microstructure examination revealed oxidation films on H13 powder surfaces

A sample was cut out of a disc and examined under a microscope. This examination revealed oxidation films on H13 powder surfaces (see Figure 60). Oxidation of H13 powder during off-gassing could be the major reason for the disc cracking.

Fifth prototyping trial

During this trial, several measures were taken to overcome the cracking problem encountered in previous prototyping experiments. The measures included 1) examination of the off-gas vacuum system; 2) modification of the off-gassing system with a by-pass for argon purging a HIP can before cooling; 3) quenching HIP cans to increase the cooling rate and reduce exposure time at most oxidation temperature ranges; 4) addition of more oxidation prevention agent into the sand mold; and 5) utilization of a metal mandrel to reduce shrinkage restriction of the disc center hole.

Two 6.5" disc cutter sand molds were made, canned, off-gassed, sealed, and processed. The resulting disc cutters still had cracks.

We were unable to fabricate an acceptable disc cutter using the CastCon process. There could be many reasons why it did not work, but, for whatever reason, the process currently does not give reproducible results. Project efforts focused on adjusting the process but there may be other issues. One problem could be the raw material powder used to form the disc cutters. An inexpensive powder was used which may not have been of the highest quality.

ESTIMATION ON DISC CUTTER MANUFACTURING COST

The basic steps of manufacturing the disc cutters using the CastCon process include die fabrication, WC insert fabrication, pattern making, sand molding, sand mold assembling, powder charging, container fabrication, binder burn-out, sealing, HIPping, cleaning, and final machining.

A steel die set is required to produce the green WC inserts by pressing WC powder in the die. A pre-mixed WC and Co powder mixture is pressed in the steel die to form a green WC insert. The green piece is then sintered in a vacuum furnace to form a strong WC insert.

A pattern is required to form the cavity of the sand mold. The cavity has the same shape as the disc cutter but is enlarged about 10% depending on the powder characteristics. The pattern can be made of wood, plastic and metal. The mixture of sand and binder are charged over the pattern and surrounded by a flask to form a drag or cope sand mold by compaction. Separation of the pattern and the sand mold forms the desired cavity. The WC inserts are set in the sand mold using glue. The drag and cope molds are bonded together to form an integrated mold by a binder. Dry metal powder is charged into the cavity of the sand mold through a hole.

A low carbon steel container is required to hold the sand mold. The sand mold is placed in the steel container and covered with a steel lid with a central stem. The stem is connected with a vacuum pump through a hose. The steel container is then heated to remove volatile materials such as an organic binder in the sand mold. The container is hermetically sealed after binder burn-out. The container is subjected high pressure and elevated temperature to consolidate the powders and to perform diffusion bonding. The container is opened and the resulting disc is sand blasted to remove any residual sand mold. The disc is machined to specifications.

Based on the manufacturing steps described previously for making the 6.5” disc cutters, preliminary cost estimates were developed. Table 10 lists the raw material costs and Table 11 gives the manufacturing costs.

Table 10. Estimated 6.5” Disc Cutter Raw Materials Costs

Material	Cost	Notes
H13 tool steel powder	$7.00/lb / $ 5.00/lb	Small quantity / large quantity
Cobalt alloy powder	$35.00/lb / $ 25.00/lb	Small quantity / large quantity
Tungsten carbide powder	$50.00/lb / $ 25.00/lb	Small quantity/minimum order (>150 kg)
Sand cost	$0.07	7 pounds per disc, $20 per ton
Binder	$0.05	
Steel container	$5.00	

The main body of the disc weighs about 8 lbs, or cost $40 to buy the powder assuming $5 per pound. The WC inserts weight about 1.8 pound, or cost $45 assuming $25 per pound. The total powder costs about $95 per disc. The cost of sand, binder and steel container add an additional $5.12 per disc to the cost.

Table 11. Estimated 6.5” Disc Cutter Manufacturing Costs		
Activity	**Cost**	**Notes**
Tooling: Pattern	$1.00	About $1,000, divided by 1,000 discs
Tooling: Die	$1.00	About $1,000, divided by 1,000 discs
Sand molding & WC insert fabrication	$5.00	Labor and equipment usage
Binder burn-out	$5.00	Labor and equipment usage
Encapsulation	$2.00	Labor and equipment usage
HIPping	$20.00	Commercial HIP chamber: 60" in diameter and 120" in height, $20,000 per run. Assuming disc can size: 8" in diameter and 4" in height, 1,000 discs per run
Cleaning	$2.00	Labor and equipment usage
Final Machining	$10.00	
Total Manufacturing Costs	**$46.00**	**Per disc**

Adding together the powder cost of $95, additional raw materials cost of $5.12, and the manufacturing cost of $46 per disc, the total preliminary estimated cost is $146.12 per disc.

CONCLUSIONS

The green fiber monolith (FM) material consists of an organic binder, a ceramic powder and a metal powder, which is similar to the composition of ceramic or metal injection molding materials. The difference is that the ceramic and metal powders in the FM material have a controlled arrangement to construct a fiber matrix microstructure. As with ceramic or metal injection molding materials, the organic binder in the FM must be removed before consolidation. It is not surprising that the binder removal by burning or evaporation is very difficult without cracking and other defects. However, the binder removal of this FM material proved to be much more difficult than is typical for a ceramic or metal injection molding material.

Containment binder removal is an effective way to remove the organic binder in the FM material at a relatively high heating rate without macro cracking.

Pressure-less sintering can not close the internal porosity of the FM material created by the binder removal. Pressure assisted sintering is necessary to consolidate the material to fully dense.

The consolidated FM material shows a relatively good combination of hardness and fracture toughness in the laboratory. However, the drill bit inserts made of FM material failed earlier than the inserts made of regular tungsten carbide in two field tests.

It appears that the 6.5" disc cutter with tungsten carbide inserts in the center of the cutting edge, for use in a tunneling machine, can be produced using H13 powder and the CastCon process. However, more auxiliary tooling, monitoring instruments and equipment need to be set up to have better control of the manufacturing process and parameters to reduce manufacturing defects. This enhanced disc cutter needs to be field tested to prove its superiority over the currently used single steel disc cutter.

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APPENDIX A
List of Specimens

Specimen Notation	Material Origin	Material Description	Processing
ACR-B1-S-1	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 1 .0°C /min
ACR-B1-S-2	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 0 .5°C /min
ACR-B1-HP-1	ACR	1ST WC/Co fiber monolithic	1100°C hot pressing, 4 ksi
ACR-B1-HP-2	ACR	1ST WC/Co fiber monolithic	950°C hot pressing, 4 ksi
ACR-B1-HP-3	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 900°C hot pressing
ACR-B1-HP-4	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 950°C hot pressing
ACR-B1-HP-5	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 1000°C hot pressing
ACR-B1-HP-6	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 1050°C hot pressing
ACR-B1-HP-7	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 1100°C hot pressing
ACR-B1-HP-8	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 1150°C hot pressing
ACR-B1-HIP-1	ACR	1ST WC/Co fiber monolithic	1100°C sintering, 1100°C HIPping,
IMP-WC/Co-50/50-SW-HP	IMP	Mixture of 50v% WC+50 v% Co, D7, sandwich structure	1100°C hot pressing, 4 ksi
IMP-WC/Co-80/20-SW-HP	IMP	Mixture of 80v% WC+20 v% Co, D7, sandwich structure	1100°C hot pressing, 4 ksi
IMP-WC/Co-88 /12-SW–HP-2	IMP	Mixture of 88v%WC+12v% Co, 316 SS, CoCr, sandwich	1100°C hot pressing, 4 ksi
IMP-WC/Co-88 /12-SW-HP-3	IMP	Mixture of 88v%WC+12v% Co, 316 SS, CoCr, sandwich	1100°C hot pressing, 4 ksi
IMP-H13-HIP-1	IMP	H13 powder, tensile bar	1100°C HIPping, 60 ksi
IMP-H13-HIP-2	IMP	H13 powder, tensile bar	1100°C HIPping, 60 ksi, heat treated
IMP-6 .5Se c-HIP-5	IMP	316 body, 50/50 WC/Co hardcore	1100°C HIPping, 60 ksi
IMP-6 .5Se c-HIP-7	IMP	H13 body, 50/50 WC/Co hardcore	1100°C HIPping, 60 ksi
IMP-6 .5Se c-HIP-8	IMP	H13 body, 88/12 WC/Co hardcore, CoCr isolation	1100°C HIPping, 60 ksi