Improvement of Wear Component's Performance by Utilizing Advanced Materials and New Manufacturing Technologies: CastCon Process for Mining Applications

Quarterly Technical Progress Report

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Abstract

The fibrous monolith material was successfully consolidated in both the hot press and the hot isostatic press. Initial evaluations indicate the material will have a very high fracture toughness and be very hard. Tungsten carbide was successfully consolidated in an H13 tool steel with the incorporation of a Co-Cr layer between the WC and the steel.

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Executive Summary

The last report summarized problems in consolidating the fibrous monolith material and consolidating tungsten carbide in a hard tool steel. In this reporting period, these problems were largely solved. The accomplishments of this reporting period were

1. successful hot-press consolidation of the fibrous monolith material into a hard (1200 Vickers) and tough material (no cracks were formed during the Vickers test). This indicates a material with very high fracture toughness combined with a very hard material.
2. successful hot isostatic press consolidation of the fibrous monolith material. Using the CastCon process, a section of the fibrous monolith was formed and is in preparation for testing. Although untested, the material has already exhibited a very high resistance to grinding.
3. a series of tests resulted in a successful consolidation of a 5.5 by 1.5 by 0.5 cm piece of tungsten carbide (WC) in an H13 tool steel. Past consolidation attempts resulted in cracks in the WC. The cause was leaching of the WC into the steel; this was eliminated with a Co-Cr layer between the WC and the steel.

Two major tasks will be addressed next:

1. refine manufacturing and consolidation of the fibrous monolith material. Although the cobalt and WC material exhibits excellent properties, uniformity of the material will be improved.
2. manufacture prototype drill bit inserts mount the inserts on a 16 inch diameter roller cone drill bits, and test the inserts by drilling taconite in an operating iron mine.

Problem Analysis

We encountered a problem of consolidating fiber reinforced WC/Co monolithic material in the last reporting period. The material was flowed out of the graphite die during hot pressing at relatively low temperatures. When ACR prepared the material, organic binders were mixed with WC and cobalt powders to produce the fiber reinforcement structure. We postulated thought that residual sulfur from the organic binder might react with the cobalt matrix to form a sulfide of low melting point. This was not the case. To test the sulfur hypothesis, we first examined the microstructure and sulfur content of Specimen ACR-B1-S-1. This specimen was the one resulted from the first sintering test (as shown in Figure 2 of the last quarterly report). Two typical regions of Specimen ACR-B1-S-1 were examined. Figure 1 is a SEM (scanning electron microscopy) picture of Region one. This region includes a large hexagonal inclusion (greater than 1mm). EDS (energy dispersive spectroscopy) analysis indicates that this inclusion contains 97.52% cobalt, 2.47% tungsten and 0.01% sulfur. Adjacent matrix contains 4.67% cobalt, 95.33% tungsten and 0.01% sulfur. Many small hexagonal cells can be identified, which are the cobalt coated WC fibers. Many cracks also can be seen. Figure 2 shows another region of inhomogeneous appearance. The hexagonal “inclusion” contains 13.03% cobalt, 86.95% tungsten and 0.02% sulfur. Many cracks and pores can be seen.

In conclusion, sulfur content in the specimen is not high. The problem of material squeezing at lower consolidation temperature was not caused by residual sulfur. The real reason is not clear. The specimen indicates that there are 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. Pressure assisted consolidation is necessary.

Since residual sulfur is not the reason to cause the material squeezing at lower consolidation temperatures, another possibility could be the low strength of the fiber reinforced material when the temperature range is between binder melting and binder evaporation. The low strength couldn't bear the weight of top graphite punch, although it is very light. We decided to separate the binder removal and the consolidation processes and try hot pressing again.

Figure 1. Region one, SEM (ACR-B1-S-1).

Figure 2. Region two, SEM (ACR-B1-S-1).

Results and Discussion

Hot Pressing Tests

Hot pressing is a process to press samples in a cylindrical die by one or two punches in vacuum or inert gas atmosphere at elevated temperatures. Therefore, it is a unidirectional and mechanical pressing. Specimen ACR-B1-S-1 after the examination as mentioned above was used to perform the hot pressing tests. This specimen was previously heated in a vacuum furnace from room temperature to 500°C at 1°C/min to burn organic binders, held for 30 minutes, heated continuously to 1100°C at 10°C/min, held for one hour, and then furnace cooled. To hot press this specimen it was inserted into a graphite die with a bottom punch and a top punch. 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. Porosity and cracks remained in the specimen after the hot pressing. The specimen was then reheated at the same condition to 950°C and held for 1.5 hours. Porosity and cracks still can be seen in the specimen. The specimen was reheated again to 1000°C, 1050°C, 1100°C and 1150°C respectively. After 1150°C hot pressing, the original cracks disappeared. The specimen (ACR-B1-HP-8) was then mounted in Beuhler Transoptic, polished by a diamond disc and diamond paste. Microstructure, hardness and fracture toughness were then examined.

Microstructure

Figure 3 shows a regional microstructure of the fiber reinforced WC/Co specimen (ACR-B1-HP-8) after hot pressing. The cells or WC fibers are about 250μ in hexagonal shape and insulated by a thin cobalt shell. The cross section of the specimen was not homogeneous (Figure 2). Particularly, a big inclusion can be seen in Figure 4.

Figure 3. Fiber reinforced WC/Co, (ACR-B1-S-1).

Vicker hardness and fracture toughness

Vicker hardness of Specimen ACR-B1-HP-8 was determined using a micro hardness tester (small load) and a regular hardness tester (larger load).

a) Micro hardness (load: 1 kg)

Spot 1: Hv 1274

Spot 2: Hv 1139

Figure 4. Big inclusion in fiber reinforced WC/Co, (ACR-B1-S-1).

No crack was found at the tips of the diamond indent as shown in Figure 5.

b) Regular hardness (load: 20 kg, Leco Model AVK-A)

Spot 3: Hv 1283

Spot 4: Hv 1095

No crack was found at the tips of the diamond indent as shown in Figure 6.

c) Regular hardness (load: 50 kg, Leco Model AVK-A)

Spot 5: Hv 1118

Spot 6: Hv 1030

No crack was found at the tips of the diamond indent in Figure 7. The specimen presents an average Vicker hardness of 1157.

Conventional WC/cobalt cermet and fiber reinforced WC/cobalt cermet comparison

The Institute of Materials Pressing conducted two conventional WC/Co cermet hot pressing experiments previously, 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 1 lists their Vicker hardness and fracture toughness. The fiber reinforced WC and cobalt monolithic specimen is harder than the 50/50 and 80/20 WC/Co materials. Figure 8 and 10 show the cracks in the 50/50 and 80/20 WC/Co specimens. The cracks were generated at the tips of the indent during the hardness tests at 20 kg load. The cracks were used to measure the fracture toughness using the indentation fracture method. Figures 5 to 7 show the indents of 1 kg, 20 kg and 50 kg loads on the fiber reinforced monolithic specimen. No cracks were found. Although fracture toughness can't be determined by the indentation method, it is obvious that the fiber reinforced monolithic sample should have much higher fracture toughness.

The fiber reinforced monolithic specimen contains

Figure 5. Micro hardness indent, 1 kg load, no cracks, (ACR-B1-HP-8).

Figure 6. Hardness indent, 20 kg load, no cracks, (ACR-B1-HP-8).

Figure 7. Hardness indent, 50 kg load, note the absence of cracks, (ACR-B1-HP-8).

82.5 vol% fiber which is made of 90 vol% WC and 10 vol% cobalt. In total ($82.5\% \times 90\% = 74.25\%$), 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.

Figure 8. 50/50 WC/Co indent, has cracks, (IMP-WC/Co-50/50-SW-HP).

Figure 9. 80/20 WC/Co indent, has cracks, (IMP-WC/Co-50/50-SW-HP).

Figure 10. 80/20 WC/Co indent, has cracks, (IMP-WC/Co-50/50-SW-HP).

Table 1. Comparison of fiber reinforced WC/Co and conventional WC/Co

Materials	Vicker Hardness	Fracture Toughness	Indent Cracks
50/50 WC/Co	909	$6.95 \text{ MPa}\cdot\text{m}^{1/2}$	long under 20 kg load
80/20 WC/Co	1042	$11.87 \text{ MPa}\cdot\text{m}^{1/2}$	long under 20 kg load
Fiber Reinforced WC/Co (74.25/25.75 WC/Co)	1157	n/a, but much higher	no under 50 kg load

Fiber reinforced WC/Co HIPping

After successful consolidation of the fiber reinforced WC/Co by hot pressing, HIPping (hot isostatic pressing) was conducted. HIP is a totally different equipment from 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. Hot press 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 pressure. Figure 11 shows the specimen removed from the steel container after HIPping. This specimen will be cut, mounted, polished and examined.

Figure 11. HIPped fiber reinforced WC/Co specimen, (ACR-B1-HIP-1).

H13 HIPping

Based on our discussion with Robbins Group, H13 was selected as the disc cutter body material. H13 powder was purchased and two specimens were produced by HIPping at 1100°C under 60 ksi pressure. These two specimens will be heat-treated, machined to make tensile bars and tested for their mechanical properties.

Figure 12. HIPped H13 specimens, (IMP-H13-HIP-1 & 2).

Solution of WC/Co cracking

It was found that WC/Co hardcore insert cracked after HIPping. We thought that the differences of thermal expansion coefficient between the WC/Co material and the adjacent body material caused large residual shear stress during cooling. The residual shear stress caused cracking of the WC/Co. Recent experiments indicated that the assumption was wrong. The reaction of WC/Co with steel at high temperature was the real reason to cause cracking. If a third material can isolate the two materials, cracking can be avoided as shown in Figures 13 and 14. In Figure

Figure 13. WC/Co cracking without insulation, (IMP-WC/Co-88/12-SW-HP-2).

13, WC/Co layer (with a long crack) directly contacts with 316 stainless steel (top layer). The cracking occurred in the WC/Co layer. In the hot pressed specimen shown in Figure 14, WC/Co (bottom layer) was isolated from 316 stainless steel with a CoCr layer. No cracking occurred.

6.5" disc cutter section prototype

To ensure the WC cracking was under control, three 6.5" disc cutter section prototypes HIPped at 1100°C under 60 ksi using the CastCon process. A long crack can be seen in the WC/Co hardness core of the prototype shown in Figure 15. The prototype shown in Figure 16 has two separated WC/Co hard cores with three 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 reinforcement tendons of steel have larger thermal expansion coefficients than the WC hard core material, therefore subjecting larger elastic shrinkage. A big crack was still found in the left WC/Co hard core. The single WC/Co hard core of the prototype shown in Figure 17 was coated with a third material, CoCr. No cracks were found in the prototype.

A summary of the samples produced to date is given in Table 2.

Figure 14. WC/Co with insulation, (IMP-WC/Co-88/12-SW-HP-3).

Figure 15. Big crack in WC/Co hardcore of 6.5" disc section prototype, (IMP-6.5Sec-HIP-5).

Figure 16. Crack in WC/Co hardcore of 6.5" disc section prototype with reinforcements, (IMP-6.5Sec-HIP-7).

Figure 17. No crack in WC/Co hardcore of 6.5" section prototype with insulation, (IMP-6.5Sec-HIP-8).

Table 2. Summary of specimens

Specimen Notation	Material Origin	Material Description	Processing
ACR-B1-S-1	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 1.0°C/min
ACR-B1-S-2	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 0.5°C/min
ACR-B1-HP-1	ACR	1 ST WC/Co fiber monolithic	1100°C hot pressing, 4 ksi
ACR-B1-HP-2	ACR	1 ST WC/Co fiber monolithic	950°C hot pressing, 4 ksi
ACR-B1-HP-3	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 900°C hot pressing
ACR-B1-HP-4	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 950°C hot pressing
ACR-B1-HP-5	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 1000°C hot pressing
ACR-B1-HP-6	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 1050°C hot pressing
ACR-B1-HP-7	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 1100°C hot pressing
ACR-B1-HP-8	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 1150°C hot pressing
ACR-B1-HIP-1	ACR	1 ST WC/Co fiber monolithic	1100°C sintering, 1100°C HIPping,
IMP-WC/Co-50/50-SW-HP	IMP	Mixture of 50v% WC+50v% Co, D7, sandwich structure	1100°C hot pressing, 4 ksi
IMP-WC/Co-80/20-SW-HP	IMP	Mixture of 80v% WC+20v% 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-6.5Sec-HIP-5	IMP	316 body, 50/50 WC/Co hardcore	1100°C HIPping, 60 ksi
IMP-6.5Sec-HIP-7	IMP	H13 body, 50/50 WC/Co hardcore	1100°C HIPping, 60 ksi
IMP-6.5Sec-HIP-8	IMP	H13 body, 88/12 WC/Co hardcore, CoCr isolation	1100°C HIPping, 60 ksi

Conclusions

1. Fibrous monolithic WC/cobalt cermet is tougher than the conventional WC/cobalt cermet with equivalent or higher hardness.
2. A layer of third material is required to isolate a steel body material and a WC insert for preventing the steel and WC reaction at elevated temperatures.