Metal-Matrix Composites and Thermal Spray Coatings for Earth Moving Machines
Final Report

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EXECUTIVE SUMMARY

One of the predominant operating costs and reasons for scheduled downtime in mining machines is to replace components which have undergone abrasive wear. In order to achieve a significant increase in abrasion resistance, cost effective advanced materials must be developed for application in mining. Complicating this effort are the diverse wear modes and conditions experienced from component to component and from mine to mine. Two processes explored to produce materials which will overcome these challenges are a hybrid pressure casting process for steel MMCs and thermally processed sprayed coatings.

Pressure casting of steel matrix composites was jointly pursued by Caterpillar Inc. and the University of California, Santa Barbara. Two hard particle systems were explored, (1) cermets (WC-Co and Mo-Fe-B) and (2) oxides (alumina and alumina-zirconia). In order to permit selective placement of these hard particles within a casting, processes were successfully developed to make hard particle preforms. In addition, processes were developed to coat particles, with the expectation that such coatings would aid infiltration. Of the coatings explored, none were found to be sufficient to withstand the casting conditions of the alumina based composites (due to melting and oxidation), and therefore no improvement in infiltration was realized. Without a mechanism to improve the infiltration characteristics of alumina based composites, the hybrid pressure casting process (in its current capacity) was unable to infiltrate oxide preforms. WC-Co was successfully coated with a boride coating which improved infiltration and reduced dissolution. However, even without the coating, the cermet based particles were readily infiltrated during pressure casting. Using a hot isostatic press, to mimic a pressure casting process, UC Santa Barbara was able to infiltrate uncoated alumina-based particles.

The fracture and wear characteristics of the resulting steel composite systems were characterized at UC Santa Barbara and DOE's Albany Research Center, respectively. The results for the cermet-based systems were mixed, with the gouging wear resistance meeting project goals for some systems, but not the impact wear resistance. The low impact wear resistance resulted from the dissolution of the particles and subsequent carbide precipitation in the steel matrix. The precipitation significantly increased the hardness and lowered the toughness of the matrix, resulting in the poor impact wear resistance. In contrast, there was minimal interaction with the alumina-based particles, which led to tougher matrices and tougher composites. Although not measured, it is suspected that this will result in good impact wear resistance. Measured gouging wear resistance exceeded project targets. Based on these observations, coupled with cost estimates for composite components, it was determined that the alumina-zirconia composites would make a viable composite system. However, the HIP based process with which this composite was made, is expensive and inefficient. For these composites to be used in production, further develop of a lower cost casting process (improved, higher pressure version of Caterpillar process) would have to be developed.

Arc lamp fusing of thermal spray coatings served to metallurgically bond the coating and substrate, thus allowing such coatings to survive high-stress loading under which conventional coatings would delaminate. Coating wetting and melting characteristics were optimized for arc lamp processing via chemistry variation. The coating compositions were designed to produce composites of hard, wear-resistant precipitates in a somewhat ductile matrix after fusing.

Knowledge was gained to allow the design of a functionally graded material coating in which the base layer(s) metallurgically bonded to the underlying substrate and absorbed solidification and coefficient of thermal expansion mismatch stresses, while the uppermost layer(s) proved extremely wear-resistant. Three successful candidate alloy systems for use in FGM designs were identified. These systems may be used alone or in conjunction with one or both of the other two. All three systems are believed to be sufficiently tailorable in composition to serve as bond coats and wear layers. High-stress third-body abrasion resistance gains in excess of 5X over carburized and hardened steels were realized.

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ABSTRACT

In an effort to realize minimum of a 2x increase in wear life of ground engaging components used on mining machines, two potentially cost effective processes were explored for the production of tailored, highly abrasion resistant materials: (1) hybrid pressure casting of steel composites, and (2) arc lamp fusing of thermal spray coatings. Steel composites comprised of cermet or oxide hard particles were successfully produced using pressure casting processes, although a cost effective process has not yet been identified for oxide particles. Both composites achieved project wear targets in high stress gouging wear, but the cermet composites did not meet the targets in impact wear, due to poor matrix toughness resulting from particle dissolution. Oxide composites had superior toughness and are expected to meet impact wear goals. Arc lamp processing of thermal spray coatings was successfully demonstrated to produce a metallurgical bond at the coating interface. Functionally graded materials were developed and successfully fused to allow for the accommodation of thermal process stresses in an intermediate layer. Ultimately, three functionally graded materials were identified as having high stress, three-body abrasion resistance sufficient to exceed project goals.

1.0 INTRODUCTION

1.1 Steel Matrix Composites

The wettability of hard particles by liquid metal plays a key role in metal matrix composite processing, especially in the pressure infiltration at elevated temperature. The transport of the liquid into the hard particles is driven by the pressure differential between the inside and outside body of the porous preform. The capillary pressure P_0 varies with the pore size d as follows:

$$P_0 = 2 \gamma \cos(\theta) / d$$

where γ is the surface tension of the liquid and θ is the contact angle. For a wetting system such as liquid steel/WC-Co particles, the capillary pressure P_0 is positive. Thus, even without applied pressure, the liquid steel could penetrate the WC-Co particles for a certain depth. This has been demonstrated at Caterpillar and elsewhere. As implied, although the liquid metal is pulled into the particles, there is a limit to the depths and quality, due to the solidification rate and the probability of trapping pockets of uninfiltreated particles. In a non-wetting system such as liquid metal/ Al_2O_3 , external pressure is needed to force the liquid into a porous body. Based on the equation above, the smaller the pore size, the higher the applied pressure that is required.

Part of the driving force for the wetting of the WC-Co particles is the reaction/interaction of the molten metal with the particles. In general, it has been found that the particle size must be kept above a certain size to prevent dissolution of a significant fraction of a particle. This size has been suggested as being approximately 5 mm or greater [1]. Unfortunately, this size is not necessarily optimal for abrasive wear as there is a larger gap between the particles, permitting

wear of the matrix. This leaves the particles at a level above the matrix where they may be vulnerable to impact damage, and therefore more rapid wear.

This investigation focused on the development of a hybrid pressure casting process, which is similar to the squeeze casting process used in casting aluminum and aluminum composites. Due to higher expected solidification rates in this process, it is anticipated that smaller WC-Co particles may be used. In addition, due to the application of pressure, it is expected that the particles will be fully infiltrated, creating a pore-free composite. Preliminary casting trials performed by Caterpillar showed, on a small scale, that these expectations could be realized for WC-Co particles [2]. Due to the application of direct pressure on the molten steel, it is expected that this process may be used to infiltrate oxide particles as well. Oxide particles, like alumina would provide a cost and weight advantage over WC-Co.

In this investigation the pressure casting of composites comprised of cermet (WC-Co and Mo-Fe-B) and oxide (alumina and alumina-zirconia) particles is explored. Resulting castings are evaluated using fracture toughness and abrasive wear testing to determine their potential for application on mining machines. Ultimately it is desired that a process and composite system be developed which will lead to a minimum of a 2x increase in component wear life.

1.2 Thermal Spray Coatings

This research effort was focused on creating highly abrasion resistant coatings that could be used in earth moving machinery. Thermal spray coatings are very attractive for such applications because of the freedom allotted with respect to chemistry. That is, most thermal spray processes use powdered feedstock rather than wire or wrought product, the chemistries of which are constrained by processing limitations. However, thermal spray coatings by nature rely on mechanical bonding of the coating to the substrate, thus making them susceptible to contact fatigue failures and gross delamination.

The ensuing results describe a method for transforming the mechanical bond of thermal spray coatings to a metallurgical bond. The apparatus used to fuse coatings was that of an inert gas plasma arc lamp, which consists of a light-focusing reflector around a water-cooled tube containing an inert gas thermal plasma. The focused light performed similar to a laser, but with a large line footprint rather than a small spot. Because of the much larger footprint afforded by the arc lamp, processing time was drastically reduced compared to using a laser for the same application.

2.0 EXPERIMENTAL

2.1 Steel Matrix Composites

2.1.1 Hard Particles

Since abrasion resistant particles for the composite systems will require both abrasion and impact resistance, two types of particles were explored, cermets and alumina-based grinding and blasting media. Tungsten Carbide – Cobalt (WC-Co) particles are the most commonly used

abrasion resistant particles for extreme service components. Typically the WC-Co particles are placed in high wear areas through incorporation in arc weld processes. Limitations of WC-Co include

- High Density: As a result of the high density of Co and W, the particles lead to composites with density higher than the parent steel matrix.
- Particle Dissolution: Due to the solubility of Co in molten iron and the ability of molten steel to dissolve WC, the dissolution of WC-Co has been a challenge in the production of composites containing WC-Co. In the patent for their composite wear blocks containing WC-Co particles, Kennametal points out that they use large (3-4 mm) particles explicitly to avoid excessive particle dissolution [ref].
- Strategic Elements: Although probably not quite as big of an issue as it once was, the use of strategic elements (W, Co), which are subject to price fluctuations dependent upon global supply and governmental stability, is undesirable.

Alumina addresses these issues by providing a low density particle with abundant elements. However, alumina may lack sufficient toughness to resist the severe wear and impact experienced in component service. In addition, being a metal oxide, alumina has poor wettability with molten steel, thus hindering molten metal infiltration and the attainment of an interfacial bond. The WC/Co and alumina-based particles used in this investigation are shown in Fig. 1.

Mo-Fe-B (MFB) particles, were explored as a potential compromise between the high density and cost of WC/Co and the poor wettability of alumina. These particles have a density closer to that of steel and are comprised of less expensive elements than those in WC-Co, and yet their abrasion resistance is similar to that of WC-Co.

2.1.2 Particle Coating

Given the poor wettability of the alumina particles and the dissolution challenge of the WC-Co particles, various particle coating schemes were explored. Initially Ni, Cu, and stainless steel coatings were investigated. These were applied to the particles using three different processes, (1) electroless plating (performed by Advanced Ceramics Inc.), (2) mechanical milling, and (3) chemical coating and reduction. Mechanical milling involves jar milling of particles with powders having the composition of the desired coating. Milling mechanically bonds the metal to the particles. Chemical coating involves the coating of particles with a metal salt (eg $\text{Cu}(\text{NO}_3)_2$ or $\text{Ni}(\text{NO}_3)_2$) and then reducing the salt in a hydrogen atmosphere furnace to leave just the metal. Although chemical coating has a number of advantages, emissions from the process may not be compatible with environmentally benign manufacturing goals.

Through work with the MFB particles, it was recognized that B makes a good wetting agent which may improve molten metal infiltration of the particles. FeB coated WC-Co particles were explored with the hopes that the boride coating would further improve infiltration and potentially reduce the dissolution of the WC-Co particles. The coating was applied by means of milling – 200 mesh FeB powder with (–12, +20) mesh WC-Co particles for 48 hours.

Fig. 1 Images showing four different abrasion resistant particles used in initial casting: (a) 0.7 – 1.2 mm alumina spheres (85% alumina, 15% amorphous), (b) 0.6 – 1.2 mm stoneblast (75% alumina, 25% ziconia), (c) 0.4 – 0.9 mm WC-6%Co, and (d) 0.6 – 1.2 mm WC-6%Co.

2.1.3 Hard Particle Preforms

Critical to the development of selectively reinforced components is the ability to keep the particles in groupings of a defined shape. Therefore, a focus of this study was to develop technology for the creation of hard particle preforms with sufficient porosity to permit molten metal infiltration.

Hard particle preforms were fabricated by applying powder metallurgy techniques. Hard particles were mixed with 10 vol.% organic binder. For those cases where the preforms were sintered and the hard particles were not coated, metal powder (like carbonic iron) was added to the preforms to facilitate bonding. The compositions for many of the preforms investigated are listed in Table 1. Mixtures were compacted by conventional uniaxial pressing. The added organic binder gave the preform sufficient strength to withstand careful handling. After compaction, the green parts were dried in an oven. Following drying, the preforms were either sintered or used as-dried (see Fig. 2). For sintering to take place an agent like the metal coating

or added metal powder was required. Although leaving the preforms unsintered would permit selective placement of the hard particle preforms, it does not ensure the particles would stay in place once molten metal infiltration starts and the binder begins to burn out, thus allowing the particles to become incorporated into the molten metal.

Table 1. Mixture Composition of Various Preforms

Preform Designation	Composition
Cu-coated Al_2O_3 preform	20wt. % metal powder + 4wt.% organic binder + Cu- Al_2O_3 particles, balance
Ni-coated Al_2O_3 preform	20wt. % metal powder + 4wt.% organic binder + Ni- Al_2O_3 particles, balance
Cu-coated WC-7Co preform	10wt. % metal powder + 3wt.% organic binder + Cu coated WC-7Co particles, balance
Ni-coated WC-7Co preform	10wt. % metal powder + 3wt.% organic binder + Ni-WC-7Co particles, balance
WC-7Co preform	10wt. % metal powder + 3wt.% organic binder + WC-7Co particles, balance
#27	100wt.% +20 mesh MFB particles + 5wt.% Binder
#28	100wt.% -20,+30 mesh WC particles + 2.5wt.% Binder
#29	100wt.% -12,+20 mesh WC particles + 2.5wt.% Binder
#30	100wt.% -20,+30 mesh WC balls + 2.5wt.% Binder
#31	50wt.% -20,+30 mesh WC particles + 50wt.% -20,+30 mesh WC balls + 2.5wt.% Binder
#36 & #37	66.6wt.% 430SS coated stoneblast particles +33.3wt.% -20, +30 mesh WC particles + 4.2wt.% Binder
#38 & #39	70wt.% 430SS coated alumina-zirconia particles +30wt.% -80 mesh MFB powder + 6.7wt.% Binder
#45-46	80wt.% (-12,+20) mesh WC + 20wt.% -20 mesh MFB particles + 2.5wt.% Binder
#47-49	80wt.% (-20,+30) mesh WC + 20wt.% -20 mesh MFB particles + 2.5wt.% Binder
#54, #68	100wt.% (-12,+20) mesh FeB powder coated WC particles + 2.5wt.% Binder

Fig. 2 Picture of tungsten carbide preforms used for casting infiltration.

The typical volume fraction of carbide based hard particle preform is around 60%. It is expected that this volume fraction can be reduced with only a small decrease in wear resistance, but a significant increase in toughness. Attempts to add steel powder into preforms to reduce the volume fraction of hard particles resulted in oxidation of the steel powder during casting and thus negatively impacted the infiltration and integrity of the composite. In order to prevent oxidation of the steel powder, graphite was added to the preform. The graphite reacts with the oxygen instead of the steel. In addition, by locally elevating the C content in the matrix, the

graphite may help inhibit dissolution of the tungsten carbide particles and promote precipitation of WC, rather than large carbon deficient carbides.

2.1.4 Ferrous Matrix

Two material compositions were used for the casting of the composites. For initial work, an SAE 4130 steel was melted from bar stock (4140 was used in casting trials at UC Santa Barbara). This steel was initially chosen because of its similarities with GET steels. However, due to the added complexity of this system associated with potential interactions of the alloying elements with the particles, it was decided to pursue a simpler steel system. As a result, the final casting trials were performed with SAE 1045, plain carbon steel.

2.1.5 Pressure Casting Tooling

Figure 3 shows a schematic drawing of the arrangement used for pressure casting. The apparatus consisted of an H13 tool steel die and punch. Within the steel punch was placed either a ceramic insert or a coated steel insert to provide protection for the die. Protection for the punch was accomplished using a ceramic plug, or a coated steel disk which was held on the end of the punch. In initial trials the plug and punch were placed into the die manually after the molten metal was poured in. In later trials, the punch was attached to the top of the press to decrease the time from pouring the metal to application of pressure. Fig. 4 shows an image of the actual pressure casting tooling sitting outside the press. The die sat on a block which permitted the application of a vacuum after molten metal was poured, to remove gas which might lead to trapped porosity (brass connector visible on block/base below die). The press was rigged with a hydraulic ejection station, such that the casting could be ejected/pushed up through the die after solidification. The four posts held the die in place during this operation and a ram extended from the bottom of the arrangement to the bottom of the die to transmit the ejection pressure.

2.1.6 Die Protection

Two different routes were explored for die protection, (1) a thin refractory coating which may or may not need to be reapplied after each casting, and (2) a thick refractory shell which would be used for a single casting, similar to the mold in investment casting. Initial casting trials were performed using the thick refractory shell concept (Fig. 4).

The challenge with using the thick refractory inserts for die protection is the cost of fabrication and the tolerance capabilities of ceramic processing. Given the short comings of these inserts, die coatings were explored which may be applied directly to the steel die and provide protection from welding and aid removal of the casting. Casting trials were performed to assess the coatings. In addition, coating cost and ease of application were considered.

2.1.7 Gravity and Pressure Casting

A 50 pound induction furnace was used at Caterpillar for melting steel. Prior to casting, the ceramic crucibles were preheated in air. The steel crucibles were also heated in air, but to a significantly lower temperature. For gravity casting trials, molten steel was poured directly into

a preheated ceramic or steel crucible in which a preform was placed in the bottom. For pressure casting trials, a preheated transfer ladle was employed. The pouring temperature of the molten steel was approximately 1700°C. In the case of pressure casting, after pouring, the whole tooling die system was moved into the press, and the working platform moved up until the top punch touched the molten steel. The top punch was self-aligned to the steel die. Pressure was applied up to around 97 MPa (14 ksi). In initial trials the pressure was held for about 60 seconds before being released. In later trials the pressure was only held for around 5 seconds in an effort to eliminate crack-like defects which were forming at the interface between the infiltrated preform and unreinforced matrix. After pressing, the die tooling was moved away from the top punch and the steel cup (or ceramic insert) together with the coupon was ejected. Following extraction, both the cup and casting were either allowed to slow cool or quenched in water.

Fig. 3 Schematic of the arrangement used for pressure casting trials at Caterpillar.

Fig. 4 Pressure casting tooling mounted on press slide. Also shown are (A) ceramic die insert and punch cap, (B) ceramic coated steel insert and ceramic punch cap and (C) punch used to generate infiltration pressures.

Pressure infiltration at UC Santa Barbara was accomplished using a hot isostatic press (HIP), configured to emulate squeeze casting. Efforts at UCSB focused on fabricating composites reinforced with the alumina and alumina-zirconia particles. Castings were made with both 4140 and 1045 steel matrices. Infiltration was accomplished by heating a crucible containing both particles and steel in vacuum. Once a temperature is attained where the steel is molten, gas pressure is applied. Since the molten metal creates a seal between the vacuum which exists at the hard particles and the applied gas pressure which exists above the molten metal, the molten metal infiltrates the preform. Aiding the infiltration is the fact that the particles are at the same temperature as the molten metal.

2.1.8 Heat Treatment of Cast Coupons

For the 4130 alloy steel, heat treatment was applied to the as-cast composite coupons. One sample (#45) was austenitized at 890°C (1634°F) for one hour and then quenched in oil, followed by tempering at 204°C (399°F) for one hour. Other casting samples were water quenched at about 816°C (1500°F) during cooling from casting to save time and energy.

The SAE 1045/carbide composite coupons were normalized at 860°C (1580°F) for 16 hours in a vacuum furnace, followed by slow cooling at a rate of 10°C (50°F) per minute. The 4140 and 1045 specimens fabricated at UCSB were normalized for 16 hours at 850°C (1562°F).

2.1.9 Abrasive Wear Testing

Abrasive wear testing was conducted at DOE's Albany Research Center (Albany, OR) on various composite samples. The composite configuration and tests conducted are indicated in Table 2. Three types of abrasive wear tests were conducted (1) dry-sand, rubber wheel (ASTM G65 Procedure B) which produces low stress scratching wear, (2) pin-abrasion, which produces higher stress gouging wear, and (3) impeller-abrasion, which produces high stress gouging and impact wear [3]. The pin-abrasion tests were conducted at three different loads to examine how the materials performed under different abrasive loads. Due to the limited number of samples available for a given condition and due to the nature of the tests, only one or two tests were conducted per condition, limiting the accuracy of the measurements. Regardless the data should provide an indication of the expected abrasive wear performance.

Table 2: Composite Test Samples and Tests Performed

Composite	Matrix/Condition	DSRW	Pin-Abrasion	Impeller-Abrasion
FeB coated WC-Co	Q&T 4130	X		X
WC-Co+5%MFB	Q&T 4130	X		X
FeB coated WC-Co	Normalized 1045		X	
WC-Co+5%MFB	Normalized 1045		X	
Alumina-Zirconia	Normalized 1045		X	
Alumina	Normalized 1045		X	
1045 Matrix	Normalized 1045		X	

2.2 Thermal Spray Coatings

As a first effort in 2001, materials demonstrated previously to have high abrasive wear resistance were plasma sprayed in air onto mild steel substrates and subsequently treated with the 300kW plasma arc lamp at Oak Ridge National Laboratory (ORNL). The rectangular substrate size (76.2mm x 25.4mm x 10mm) was chosen to allow for dry-sand/rubber wheel (ASTM G65 B) and pin-on-disk wear testing at Albany Research Center (ARC). Coating thickness and composition were selected to simulate layers within a functionally graded material (FGM) and arc lamp processing parameters were varied in an effort to achieve a metallurgical bond between the coatings and substrates. All plasma sprayed specimens were degreased, grit blasted with 20 mesh alumina grit, and cleaned with a solvent prior to spraying. A Metco 9MB plasma gun was employed for spraying.

Additionally, two coating compositions were applied by high-velocity oxy-fuel (HVOF) spraying onto case-hardened alloy steel substrates. These specimens were hollow cylinders (90mm O.D. x 60mm I.D. x 25.4mm), chosen to investigate geometric effects during arc lamp processing. HVOF sprayed specimens were degreased, grit blasted with 20 mesh alumina grit, and cleaned with a solvent prior to spraying. A Metco DJ HVOF gun was employed for spraying. All thermal spray coatings referenced in this report were produced at the Caterpillar Inc. Technical Center in Mossville, IL.

Arc lamp processing at ORNL was performed in a flowing Ar atmosphere to minimize possible oxidation of coating materials. Effects of pre-heating were explored by varying arc lamp power and number of passes across each specimen. Coating materials, thicknesses, and arc lamp parameters are shown in Table 3. Coatings underwent microhardness testing (Vickers, 500g) prior to and after arc lamp treatment.

Table 3. Coating characteristics and arc lamp processing parameters.

Coating Composition*	Coating Layer Thicknesses, μm	Substrate Shape	Arc Lamp Parameters**	
M4+30vol.%TiC	460	rectangular	HC	LC twice, then HC
M4+30vol.%TiC on 80/20 M4 +30vol.%TiC/M4	440 on 460	rectangular	HC	LC twice, then HC
60/40 M4+30vol.%TiC/M4	490	rectangular	HC	LC twice, then HC
M4	440	rectangular	HC	LC twice, then HC
M4 on 80/20 M4/A4650	520 on 500	rectangular	HC	LC twice, then HC
60/40 M4/A4650	520	rectangular	HC	LC twice, then HC
T15+30vol.%TiC	500	rectangular	HC	LC twice, then HC
T15+30vol.%TiC on 80/20 T15+30vol.%TiC/T15	520 on 520	rectangular	HC	LC twice, then HC
60/40 T15+30vol.%TiC/T15	560	rectangular	HC	LC twice, then HC
T15	490	rectangular	HC	LC twice, then HC
A4650	510	rectangular	HC	LC twice, then HC
CPT alloy	1010	rectangular	HC	LC twice, then HC
CrC-NiCr composite	500	hollow cylinder	---	LC twice, then HC
CrC-NiCr composite	380	hollow cylinder	---	LC twice, then HC
70/30 CPT alloy/NiCr alloy	380	hollow cylinder	---	LC twice, then HC

* Numbers indicate weight percents of components unless otherwise indicated.

** LC = 400A at 7mm/s traverse speed, HC = 900A at 8mm/s

The second effort consisted of twenty-two plasma sprayed coating compositions being fused with the plasma arc lamp at (ORNL) in 2002 and sent to ARC for ASTM G65 B wear testing. The coating compositions were designed to produce composites of hard, wear-resistant precipitates in a somewhat ductile matrix after fusing. Alloy and mixture compositions were varied systematically in order to determine both the most wear-resistant and the most compliant coating compositions. The goal of this particular effort was to gain enough knowledge to allow for the design of a functionally graded material (FGM) coating in which the first layer will metallurgically bond to the underlying substrate and absorb solidification and coefficient of thermal expansion (CTE) mismatch stresses, while the uppermost layer(s) will be extremely wear-resistant. The substrate onto which all coatings were sprayed was carburized and hardened, low-alloy steel (25mm x 25mm x 152mm) with a surface hardness of approximately 50HRC.

Building on knowledge previously gained from experiments involving arc lamp treatment of thermal sprayed coatings, a proprietary ternary alloy sintered from two binary alloys (CP and PT) was included in all coatings to provide specific coating wetting characteristics crucial to the fusing process. This proprietary ternary alloy will hereafter be referred to as CPT. Coating thicknesses ranged from 0.25mm to 0.8mm with the vast majority of coatings being approximately 0.75mm thick.

Again, arc lamp processing at ORNL was performed in a flowing Ar atmosphere to minimize possible oxidation of coating materials. Each coating composition was fused two different ways with the arc lamp; 2x 7mm/s at 500A, then 5mm/s at 900A and 7mm/s at 500A, 6mm/s at 650A, 5mm/s at 900A, and 6mm/s at 400A. Lamp passes prior to the 900A pass served to pre-heat the coating prior to fusing and the 400A pass after the fusing pass was added in an effort to relieve solidification stresses in the fused coating. It should be noted that manipulation issues with the arc lamp robot resulted in approximately a 25s delay between passes in all cases. Coatings were microhardness tested (Vickers, 300g) prior to and after arc lamp treatment and the results are shown in Table 4.

ASTM G65 B wear testing was performed on twenty-one fused coating compositions at the ARC. Fusing of the coatings was accomplished by usage of the plasma arc lamp at ORNL using the following parameters: 2x 7mm/s at 500A, then 5mm/s at 900A and 7mm/s at 500A. During fusing, the porosity and finely dispersed oxides inherent to plasma sprayed coatings agglomerate and rise toward the coating free surface. While some of these agglomerates float out of the coating, others are trapped throughout the coating thickness. Therefore, an apparent density calculation was performed for each coating through digital manipulation of optical micrographs. In order to conservatively estimate the wear resistance of these coatings, all agglomerates were considered to be voids.

The third coating fusing iteration consisted of fusing ten plasma sprayed FGM coatings detailed in Table 5. All coatings were designed with lower layers that metallurgically bond to the underlying substrate and absorb solidification and CTE mismatch stresses, while the upper layer(s) remain very hard and wear resistant. These coatings were deposited onto hollow carburized and hardened steel cylinders with geometries closely resembling Caterpillar Inc. machine components.

Table 4. Microhardness results for coatings in both the as-sprayed and fused conditions.

Coating Composition*	Microhardness $\pm\sigma$, HV _{300g} **		
	As-sprayed***	2x 7mm/s @ 500A & 5mm/s @ 900A	7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, & 6mm/s @ 400A
M4 – 20CPT	513 $\pm$ 113	801 $\pm$ 132	815 $\pm$ 125
M4 – 40CPT	565 $\pm$ 158	1066 $\pm$ 48	1056 $\pm$ 63
M4 – 60CPT	625 $\pm$ 182	1170 $\pm$ 61	1103 $\pm$ 127
M4 – 80CPT	753 $\pm$ 297	1236 $\pm$ 184	1270 $\pm$ 206
316L – 40CPT	391 $\pm$ 124	567 $\pm$ 37	641 $\pm$ 59
316L – 60CPT	541 $\pm$ 164	784 $\pm$ 109	854 $\pm$ 169
316L – 80CPT	903 $\pm$ 268	1166 $\pm$ 186	1124 $\pm$ 183
434L – 60CPT	266 $\pm$ 111	767 $\pm$ 127	663 $\pm$ 205
434L – 80CPT	457 $\pm$ 189	1017 $\pm$ 202	973 $\pm$ 132
NiCr – 60CPT	537 $\pm$ 51	512 $\pm$ 77	467 $\pm$ 113
NiCr – 80CPT	711 $\pm$ 167	849 $\pm$ 128	879 $\pm$ 199
NiCr – 90CPT	604 $\pm$ 65	1146 $\pm$ 114	1108 $\pm$ 225
Cr – 20CPT	600 $\pm$ 82	638 $\pm$ 175	590 $\pm$ 70
Cr – 40CPT	658 $\pm$ 114	729 $\pm$ 107	828 $\pm$ 100
Cr – 60CPT	787 $\pm$ 214	654 $\pm$ 112	691 $\pm$ 129
Cr – 80CPT	715 $\pm$ 255	961 $\pm$ 177	747 $\pm$ 161
50CP – 38PT – Cr alloyed	539 $\pm$ 155	891 $\pm$ 204	819 $\pm$ 146
53CP – PT	423 $\pm$ 23	745 $\pm$ 128	672 $\pm$ 100
Cr – 40(53CP – PT)	608 $\pm$ 91	not tested	797 $\pm$ 65
Cr – 60(53CP – PT)	646 $\pm$ 71	not tested	not tested
Cr – 80(53CP – PT)	625 $\pm$ 67	1136 $\pm$ 82	1244 $\pm$ 183
(M4 + 40v.Al ₂ O ₃)– 50CPT	451 $\pm$ 94	1006 $\pm$ 52	855 $\pm$ 130

* Numbers indicate weight percents of components unless otherwise indicated.

** Ten hardness tests were performed on a polished coating cross-section, unless otherwise noted.

*** Less than ten hardness tests were performed on a polished coating cross-section.

Table 5. As-sprayed FGM coatings on heat treated cylinders.

Cyl. No.	1st Layer Mat'l. & Δt	2nd Layer Mat'l. & $\Delta t, \mu\text{m}$	3rd Layer Mat'l. & $\Delta t, \mu\text{m}$	4th Layer Mat'l. & $\Delta t, \mu\text{m}$	5th Layer Mat'l. & $\Delta t, \mu\text{m}$	6th Layer Mat'l. & $\Delta t, \mu\text{m}$	7th Layer Mat'l. & $\Delta t, \mu\text{m}$	8th Layer Mat'l. & $\Delta t, \mu\text{m}$
1	316L – 40CPT, 290 μm	316L – 60CPT, 250 μm	316L – 80CPT, 520 μm					
2	316L – 40CPT, 80 μm	316L – 60CPT, 90 μm	316L – 80CPT, 360 μm					
3	M4 – 40CPT, 200 μm	M4 – 60CPT, 240 μm	M4 – 80CPT, 590 μm					
4	M4 – 40CPT, 120 μm	M4 – 60CPT, 120 μm	M4 – 80CPT, 330 μm					
4	NiCr – 60CPT, 230 μm	NiCr – 80CPT, 310 μm	NiCr – 90CPT, 580 μm					
6	NiCr – 60CPT, 140 μm	NiCr – 80CPT, 100 μm	NiCr – 90CPT, 400 μm					
7	NiCr – 60CPT, 140 μm	NiCr – 80CPT, 510 μm	M4 – 80CPT, 460 μm					
8	NiCr – 60CPT, 220 μm	NiCr – 80CPT, 320 μm	M4 – 80CPT, 560 μm					
9	316L – 40CPT, 110 μm	316L – 60CPT, 130 μm	M4 – 40CPT, 220 μm	M4 – 60CPT, 230 μm	M4 – 80CPT, 510 μm			
10	316L – 40CPT, 110 μm	316L – 60CPT, 110 μm	M4 – 60CPT, 120 μm	M4 – 80CPT, 150 μm	316L – 40CPT, 160 μm	M4 – 80CPT, 150 μm	316L – 40CPT, 150 μm	M4 – 80CPT, 190 μm

Each of the coatings was fused with identical processing parameters. A schematic of the fixturing used during the fusing operation and photographs of the set-up are shown in Figs. 5 and 6, respectively. A 11.5cm arc length was used with a reflector that allowed for a 4cm stand-off distance. The cylinders were rotated at approximately 1.5rpm (5.8mm/s surface speed) and water-cooled from the internal diameter (I.D.) during fusing. A quartz plate was supported between the arc lamp and cylinder to protect the lamp from smoke and unforeseen debris during processing. Inert gas (Ar) was flowed between the cylinder and quartz plate to expel any smoke evolved during fusing of the coatings. Each cylinder was allowed to complete three revolutions – one revolution each with arc lamp settings in the order of 650A, 300A, and 900A. Two of the

ten bushings were not completely revolved during the 900A pass due to failure of the arc lamp anode. These cylinders were cross-sectioned and evaluated via electron microscopy and microhardness indentation and will be discussed later.

Full-scale laboratory wear testing of seven coated (and fused) cylinders was completed. The testing consisted of sliding motion between the as-fused coating surface and a hardened steel (~50HRC) component with the negative shape of the coated cylinder. Both water and sand were injected between the sliding surfaces after each of 4,000 cycles. The cylinder (coating) weight loss was measured every 1,000 cycles and the mating component weight loss was measured after test completion. Three baseline tests were run with current production materials to provide a basis for comparison. Although not exact, comparisons in terms of weight loss are acceptably accurate considering the density of the current steel components and that of the coating are very similar. Abrasive wear testing details are provided in Table 6.

Fig. 5 Schematic diagram of the set-up used for fusing coatings on the outside diameter of hollow steel cylinders.

Fig. 6 Photographs of the cylinder fusing set-up at ORNL.

Table 6. Abrasive wear testing parameters.

Vertical Load, kN	54.3
Horizontal Load, kN	52.7
Resultant Load, kN	75.7
Initial Contact Length, mm	64
Avg. Cycle Time, s	10
Avg. Rotation Time, s	1
Avg. Water Pressure, kPa	114
Avg. Sand Flow, grains/min.	255

Arc lamp fusing of 16 FGM coatings, detailed in Table 7, was accomplished in the fourth iteration. Again, all coatings were designed with lower layers that metallurgically bond to the underlying substrate and absorb solidification and coefficient of thermal expansion mismatch stresses, while the upper layer(s) remain very hard and wear resistant. These coatings were deposited onto as-rolled 1018 steel tubes with the same O.D. as components used in some Caterpillar Inc. machinery.

The same fixturing was employed as described previously except an 11.5cm arc length was used with a reflector that allowed for a shorter, 1cm stand-off distance. The cylinders were rotated at approximately 1.3rpm (5.0mm/s surface speed) with a quartz plate supported between the arc lamp and bushing to protect the lamp from smoke and unforeseen debris during processing. Air was flowed between the tubes and quartz plate to expel any smoke evolved during fusing of the coatings. With the exception of one tube, each was allowed to complete one rotation for preheating and one for fusing. Cylinder no. 10F was only rotated approximately 180° during the fusing pass due to the breaking of the protective quartz plate.

Full-scale laboratory wear testing of four coated (and fused) cylinders was completed. Details on the coating designs are given in Table 8. One coating composition was selected to serve as a compliant base layer in an FGM design and was sprayed to approximately 0.75mm thick.

Table 7. As-sprayed FGM coatings descriptions and associated arc lamp processing parameters.

Cylinder No.	1st Layer Mat'l. & Thickness	2nd Layer Mat'l. & Thickness	3rd Layer Mat'l. & Thickness	Arc Lamp Current Settings (Preheat & Fuse)
1F	316L - 40CPT, 250µm	316L - 60CPT, 100µm	316L - 80CPT, 310µm	400A & 700A
2F	316L - 40CPT, 200µm	316L - 60CPT, 110µm	316L - 80CPT, 300µm	550A & 800A
3F	316L - 40CPT, 350µm	316L - 60CPT, 250µm	316L - 80CPT, 520µm	500A & 800A
4F	316L - 40CPT, 370µm	316L - 60CPT, 260µm	316L - 80CPT, 490µm	550A & 800A
5F	316L - 60CPT, 770µm			550A & 800A
6F	M4 - 40CPT, 230µm	M4 - 60CPT, 100µm	M4 - 80CPT, 280µm	500A & 800A
7F	M4 - 40CPT, 210µm	M4 - 60CPT, 110µm	M4 - 80CPT, 280µm	550A & 800A
8F	M4 - 40CPT, 340µm	M4 - 60CPT, 270µm	M4 - 80CPT, 500µm	550A & 850A
9F	M4 - 40CPT, 360µm	M4 - 60CPT, 280µm	M4 - 80CPT, 490µm	550A & 800A
10F	M4 - 40CPT, 600µm			550A & 800A
11F	NiCr - 60CPT, 390µm	NiCr - 80CPT, 330µm	NiCr - 90CPT, 440µm	550A & 800A
12F	NiCr - 60CPT, 370µm	NiCr - 80CPT, 250µm	NiCr - 90CPT, 510µm	550A & 800A
13F	NiCr - 60CPT, 350µm	NiCr - 80CPT, 40µm	NiCr - 90CPT, 250µm	550A & 800A
14F	NiCr - 60CPT, 250µm	NiCr - 80CPT, 90µm	NiCr - 90CPT, 310µm	550A & 800A
15F	NiCr - 80CPT, 640µm			550A & 800A
16F	M4 - 15FeSi, 630µm			550A & 800A

Table 8. Descriptions of fused FGM coatings tested for high-stress third-particle abrasion.

Cylinder No.	1st Layer Mat'l. & Thickness	2nd Layer Mat'l. & Thickness	3rd Layer Mat'l. & Thickness
3F	316L - 40CPT, 350μm	316L - 60CPT, 250μm	316L - 80CPT, 520μm
5F	316L - 60CPT, 770μm		
9F	M4 - 40CPT, 360μm	M4 - 60CPT, 280μm	M4 - 80CPT, 490μm
11F	NiCr - 60CPT, 390μm	NiCr - 80CPT, 330μm	NiCr - 90CPT, 440μm

3.0 RESULTS AND DISCUSSION

3.1 Steel Matrix Composites

3.1.1 Microstructure of Hard Particles Preforms

Sintered preforms were strong enough for transport and handling in the pressure infiltration process because of metallic bonds formed by the added metal powder. Fig. 7 shows a cylindrical Cu-coated alumina preform. The as-sintered preform is porous with many metallic bonds at the contact area of the particles. In this case the coating, which was applied by electroless plating, remained adhered to the particle surface and still fully coated the particle surface after sintering (Fig. 8). The mixed-in Cu powder formed metallic bonds between the coated particles. Sintered powder bridges were even observed between nontouching particles. In contrast, the coating layer formed by milling was discontinuous (Fig. 9). It appears that some of the coating detached from alumina particle surface during sintering. Coalescence of coated powder and added powder is observed after sintering, leaving portions of the particles uncoated.

Fig. 7 Cu-coated alumina preform made through powder metallurgy route.

Fig. 8 Surface of as-sintered preform comprised of alumina particles coated using electroless plating. The process creates a porous preform with metallic bond between the particles.

Fig. 9 Surface of a fractured Cu-coated alumina preform. The particles were coated through milling with Cu powder. The uncoated alumina surface results from the coating debonding during fracture and diffusion during sintering (migration driven by desire to reduce surface energy).

The fracture surface of an electroless coated Ni-alumina preform reveals decohesion between the ceramic particles and coating layer (Fig 10). Gaps existed between the ceramic particles and coating layer. This indicates that the cohesion between the alumina and nickel is poor. Fig. 11 shows the fracture surface of a mill-coated Ni-alumina preform. The structure is similar to that of the mill-coated Cu-alumina preform. However, a greater fraction of the surface remained

coated when Ni powder is used. This may be due to the fact that the particle size of the Ni powder is much finer than that of copper powder.

In brief, preforms may be successfully manufactured through powder metallurgy techniques. However, the sintering process appears to modify the particle coatings, leaving portions of the particle surface uncoated. Regardless, it was found that such coatings for alumina particles were ineffective at improving molten metal infiltration. Since the casting process is performed in air, the Ni and Cu coatings oxidize due to the heat of casting (heat of dies and molten metal) prior to infiltration. In addition, any attempts to provide adequate preheat for the preform, also resulted in oxidation. Ultimately this implies that alumina based particles must be infiltrated without coatings, thus increasing the preheat and pressure requirements for the particles. Without the coatings, it is also questionable whether preforms could be easily made. It is known that the spherical alumina particles will sinter together (due to the presence of a glassy sintering aid), however the ability to make preforms with the angular alumina-zirconia media would have to be investigated further.

Fig. 10 Surface of a fractured Ni-coated alumina preform. The particles were coated through electroless plating. The fact that there are uncoated regions on the alumina particles suggests that there is poor bonding between the Ni and alumina.

3.1.2 Gravity Casting

3.1.2.1 Oxide Hard Particles

For gravity casting (no pressure applied to molten metal), no infiltration occurred for those preforms containing alumina particles. Instead of being infiltrated the preforms broke up and the alumina particles floated to the top of the melt without being entrained.

Fig. 11 Surface of a sintered preform comprised of alumina coated with Ni via milling.

3.1.2.2 Cermet Particles

For the cermet particles significant infiltration was achieved, even without the application of pressure. Fig. 12 shows the cross section of a gravity cast WC-Co composite. The infiltration depth was up to 9mm. However, the hard particles were only partially infiltrated and porosity was observed throughout both the reinforced and unreinforced composite regions. The oxidation of the cobalt binder may have contributed to the incomplete infiltration. In addition, large pores and cracks were observed at the interface between base steel and composite materials. The cracks are likely due to the brittleness of the steel-carbide composite system, as described below.

Fig. 12 Macrostructure of #6 steel/WC composite. Liquid steel was poured at 1640°C into a 540°C preheated ceramic cup with no applied pressure. It shows a good infiltration depth but high porosity in the composite zone.

In contrast to the WC-Co composites, fully infiltrated cast coupons were obtained for steel-boride composite systems. Figure 13 shows an image of a whole cast coupon of #27B and cross sections of #27A. The liquid steel fully infiltrated the preform without any defects. The hard particles were uniformly distributed in the steel matrix. Parts of the hard particles were observed dissolved into steel, but not to the extent experienced by the WC-Co particles. The wettability of liquid steel on MFB is even better than that on WC-Co. The interface between the reinforced and unreinforced regions was smooth and continuous. No cracks or gaps were observed at the interface of steel and hard particles. However, due to the lower density of the MFB preforms, they tended to float during casting, (Fig. 13 (b)).

Applying vacuum during the gravity casting helps reduce blowholes and porosity in the cast coupons. The three sources which may cause the formation of blowholes and pores are

- 1) The decomposition of the organic binder, which generates hydro-carbon gas (it is believed that this is the main source for the pores in the reinforced regions and on the outside walls).
- 2) Turbulence of the liquid steel caused by pouring traps air in the steel.
- 3) Oxygen dissolved in the liquid steel at high temperature releases during solidification.

Fig. 13 Pictures of #27A and #27B steel/MFB composite coupons made by gravity casting, (a) whole and sectioned cast coupons, and (b) cross section macrostructure of #27A shows the floating of the MFB preform and partial dissolution of MFB particles.

3.1.2.2.1 Microstructure of Gravity Cast Steel-Carbide Composite

Figure 14 shows the dissolution of the cemented carbide particles into a steel matrix. The dissolution of the particles is caused by the melting of cobalt binder and the dissolving of the Co into the iron. With the binder phase dissolved and molten, the WC particles floated out away from center of the WC-Co particles. Hence, the particles lost shape quickly and the very fine WC grains dispersed into the steel matrix, as seen in Figure 14(a).

Fig.14 Micrographs showing the dissolution of the cemented carbide particles into a steel matrix: (a) dissolution of cemented particle caused by melting of the binder phase, (b) WC grain growth, (c) $(W, Fe)_6C$ precipitated at steel matrix, and (d) high magnification picture of the $(W, Fe)_6C$.

In the molten metal the WC grains started to dissolve as well. Tungsten carbide has a limited solubility in steel. Therefore, upon solidification they reprecipitated either as WC or $(W, Fe)_6C$. When the carbide precipitates as WC, it is manifested as growth of existing grains, as seen in Figure 14(b). However, when it precipitates as very large $(W, Fe)_6C$ (which is a carbon deficient η -phase) the precipitation is mostly distributed in the steel matrix, as shown in Figures 14(c) and (d). The $(W, Fe)_6C$ or η -phase is very brittle, and therefore detrimental to the toughness of the steel matrix. The combination of the dispersion of fine WC grains and η -phase in the steel matrix make the composite very brittle, and cracks were found in the as-cast steel/WC-Co composite samples and a result of process stresses (likely primarily thermal expansion mismatch stresses).

3.1.2.2.2 Microstructure of Gravity Cast Steel-Borides Composite

Figure 15(a) illustrates the morphology of the MFB-Fe cermet particles. The rectangular or angular shaped MFB ternary boride was bonded by iron. Fig. 15(b) shows an individual cermet particle distributed in the steel matrix. The edge of the particles show evidence of dissolution.

Fig. 15 Morphology of the MFB cermet particles and the MFB cermet-steel composite. (a) MFB cermet particle showing spherical pores which are a typical feature of as-sintered powder metal parts in the final sintering stage. (b) Edges of cermet particles showing evidence of dissolution.

Fig. 16 shows the dissolution of the MFB-Fe cermet particles in the steel matrix. The MFB is dissolved by eutectic reaction with matrix steel, as shown (Fig. 16(a)). A clear front edge of the reaction zone was found at the interface with the steel matrix. The steel matrix appeared to be only minimally affected by the dissolution of the hard particles unless expanding reaction zones fill all the inter-particle area. Fig. 16(b) shows the morphology of totally dissolved MFB-Fe hard particle (or a section through a fully dissolved region of particle). Upon the dissolution of the hard particles, eutectic microstructure replaces the composite structure of the original particle and the pores inside the particles are eliminated. When the composition of the eutectic was diluted by steel, the primary Fe-Mo-B solution began to precipitate from the eutectic matrix, as shown in Fig. 16(c). Fig. 16(d) is a further diluted microstructure of the Fe-Mo-B alloy.

In the development of the microstructure shown in Fig. 16, the Fe-MFB system could be seen as a pseudobinary eutectic system. The original MFB-Fe cermet could be seen as a hypereutectic structure that is characterized by very large proeutectic MFB borides with eutectic cells as a matrix. When liquid steel is encountered, a eutectic reaction occurs between liquid steel and the MFB borides. It is reported that the liquid phase formation temperature for the Fe-MFB is about 1052°C (1925°F), which is much lower than the melting point of the base steel. This feature is very beneficial to infiltration since the liquid solidification temperature is decreased from about 1538°C to 1051°C (2800°F to 1925°F) when the eutectic reaction takes place. If sufficient time exists prior to solidification, inter-diffusion results in higher Fe composition than that of eutectic. Hence, the proeutectic austenite dendrites will precipitate first, as seen in Figs. 16(c) and (d).

The dissolution of the MFB particles had a significantly negative effect on the composition of the matrix. An examination of the steel, even in unreinforced regions, shows B precipitated at grain boundaries. Such regions will significantly reduce the toughness of the composites. This issue will be discussed further in the next section.

Fig. 16 Micrographs showing the dissolution process of the MFB-Fe cermet particles in a steel matrix: (a) the eutectic reaction of the MFB with steel, (b) almost fully dissolved MFB-Fe cermet particle, (c) primary precipitated Fe-Mo-B solid solution from the eutectic matrix, and (d) further diluted microstructure of the Fe-Mo-B alloy.

3.1.2.2.3 Microstructure of Gravity Cast WC-Co Preforms Containing MFB particles or FeB coating.

Table 9 shows chemistries of cast coupons #45, #54, #68 using the standard 4130 alloy steel. The chemistry of base steel was affected not only by process control, but also by the composition of preforms. The high molybdenum and boron content of #45 was caused by the dissolution of MFB particles in a preform comprised of WC/Co and MFB particles. High boron content is detrimental to the toughness of the steel because the boron segregates to grain boundaries, leading to grain boundary embrittlement. The preforms in #54 and #68 were comprised of only FeB coated WC/Co particles. As a result, the boron content was 20 times less in #54 and #68 coupons than that in #45 coupon, and is in the range desired for boron treated steels.

Figure 17 shows the microstructure of #45 before and after heat treatment. It was observed that the matrix of the as-cast composite was primarily a eutectic of boride and steel, which formed due to the dissolution of MFB particles. Around the edge of tungsten carbide particles, fine complex boron-carbides precipitated, as seen in Figures 17 (a) and (b).

Table 9. Chemical Analysis of Casting Samples

Element	Chemistry Results, %			
	#45	#54	#68	Standard 4130 steel
Carbon – C	0.37	0.36	0.32	0.28~0.33
Manganese – Mn	0.41	0.31	0.47	0.40~0.60
Silicon – Si	0.14	0.05	0.17	0.15~0.30
Nickel – Ni	0.16	0.16	0.04	-
Chromium – Cr	0.96	1.05	0.82	0.80~1.10
Molybdenum – Mo	0.80	0.22	0.17	0.15~0.30
Copper – Cu	0.18	0.16	0.10	-
Aluminum – Al	0.02	0.146	0.00	-
Boron – B	0.05	0.0022	0.0025	-
Phosphorous – P	0.018	0.021	0.008	0.035 max
Sulfur – S	0.031	0.028	0.016	0.040 max

Figures 17 (c) and (d) show the microstructure development of #45 after heat treatment. More complex boron-carbide particles were precipitated from the composite matrix and around the edge of tungsten carbide particles. Significant grain growth was also observed for the precipitated boron-carbide or carbon deficient carbides. These coarse carbides are detrimental to the toughness of the matrix. The microstructure of the as-tempered sample is similar to the as-quenched one, possibly a result of autotempering resulting from residual heat in the particles after quench.

Figure 18 shows the microstructure of the base (unreinforced) steel of coupon #45. The microstructure of the base steel of the as-cast sample is composed of coarse bainite or acicular ferrite. While the microstructure of as-quenched sample appeared to consist of fine martensite with relatively small grain size. Boron was found to segregate at the grain boundaries.

The FeB coated WC-Co-steel composites contain microstructural features of both plain WC-Co composites and the mixed MFB and WC-Co steel composites. Figure 19 presents the microstructure of coupon #54 fabricated with an FeB coated WC-Co preform. It was found that a thin layer of complex boron-carbide was formed around the WC-Co particles, which helped to prevent the dissolution of the WC-Co particles. However, some large, carbon deficient carbides were also precipitated from the steel matrix, which is observed very often in WC-Co-steel composites. Fig. 19(b) shows the interface between the unreinforced base steel and the composite region, where it is clearly evident that reactions with the particles are dramatically affecting the steel composition and structure in the reinforced regions.

Fig. 17 Microstructures development of #45 gravity casting coupon indicated the precipitation and coarsening of complex carbides in the composite part: (a), (b) as-cast, (c), (d) as-quenched, and (e), (f) as-tempered.

Fig. 18 Microstructure development of the base steel in coupon #45 gravity cast sample, (a) as-cast, (b) as-quenched, and (c) as-tempered.

Fig. 19 Microstructure of coupon #54 made with a FeB coated WC/Co preform: (a) composite region, and (b) interface between composite and base steel.

3.1.3 Pressure Casting

3.1.3.1 Alumina Hard Particles

Attempts to pressure cast composites containing alumina hard particles, using the process developed at Caterpillar, failed to achieve any significant infiltration. Pressure casting trials were performed using spherical alumina particles coated with Cu or Ni through the electroless process. The coating was expected to improve the wetting behavior of liquid steel. However, the coating layer was oxidized instantly when the particles were transported into the preheated ceramic. It was observed that the metal coating came off the alumina particles near the steel/preform interface, as shown in Fig. 20. This is due to the melting of the copper coating, which does not wet or react with the alumina. The molten copper segregated into pores at the lower part of the preform because of the surface tension. Instead of a metal coating, a molten glass layer was found at the surface of alumina particles and it resulted in some degree sintering of the alumina particles (glass phase is from the binder used to create the 90% Al_2O_3 spheres). Further complicating infiltration is the rapid solidification of the steel. All these factors are detrimental to the liquid metal infiltration.

Fig. 20 Macrostructure of #11 steel/ Al_2O_3 composite. Liquid steel was poured at 1700°C into a 540°C preheated ceramic cup. The applied pressure was 31MPa. Little infiltration was observed because of poor wetting and rapid solidification of the liquid steel.

Researchers at UC Santa Barbara were able to infiltrate uncoated Al_2O_3 particles (Fig. 21). The particles were infiltrated using a hot isostatic press (HIP), where the particles and steel were heated up together in vacuum. Once the metal was molten, pressure was applied such that the molten metal was forced into the preform, which was still under vacuum due to the seal created by the molten metal. Complete infiltration was achieved.

Fig. 21 Steel/alumina composites created at UC Santa Barbara using a hot isostatic press. The composite on the left consists of the spherical alumina particles, and that on the right has the angular “stone-blast”, alumina/zirconia particles.

The initial set of composites made at UC Santa Barbara were cast using a 4140 alloy. SEM micrographs of cross-sections through the pressure cast alumina and alumina-zirconia composites are shown in Figures 22 and 23. The notable features include uniform distributions of the particles and complete infiltration of the compact by the molten alloy. In the alumina/steel composite, the particles had partially sintered at the contact points (Fig. 22(b, c)). Such sintering was not evident with the alumina-zirconia particles.

Fig. 22 Scanning electron micrographs showing uniform distribution of alumina particles in a steel matrix (a); sintering at the particle junctions (b,c); and complete melt infiltration, even in regions adjacent to the sintered contacts (c) and between closely spaced particles (d). (Image in (a) taken in backscatter electron imaging mode; (b)–(d) taken in secondary electron imaging mode.)

Fig 22 (cont.)

Fig. 23 Scanning electron micrographs showing distribution of alumina-zirconia particles in steel matrix (a, b); and complete infiltration of the steel melt between closely spaced particles (c) and into cracks within the particles (d). (Images taken in backscatter electron imaging mode.) There was no evidence of sintering at the particle contacts.

The matrix hardness measurements are summarized in Fig. 24. Prior to normalization, the matrix hardness was uniform within each specimen (at least over length scales greater than the indentation size, $\sim 100\mu\text{m}$) and essentially the same in the two specimens: $302\pm 9\text{ kg/mm}^2$ and $330\pm 30\text{ kg/mm}^2$ in the alumina-zirconia /steel and alumina/steel composites, respectively. Following normalization, the matrix within the steel/alumina-zirconia composite exhibited an $\sim 30\%$ increase in hardness (to $430\pm 32\text{ kg/mm}^2$). By contrast, that in the steel/alumina composite exhibited a 50% *decrease* (to $154\pm 36\text{ kg/mm}^2$). The origin of these opposing changes is surmised to be associated with a reaction between the steel alloy and the ceramic particles (dissolution of Zr). Differences in microstructures were also observed (Fig. 25).

Fig. 24 Summary of hardness measurements of the HIPed composites, before and after the normalization treatment (“Stoneblast” = alumina-zirconia).

The second group of composites cast at UC Santa Barbara again contained either spherical alumina or angular alumina/zirconia (stoneblast) particles. The matrix was chosen as SAE 1045, to eliminate the potential for alloy interaction with the particles. No special efforts were made to enhance melt infiltration. Infiltration was again accomplished using a hot isostatic press (HIP), configured to emulate squeeze casting. All specimens were normalized prior to testing. Resulting microstructures are evaluated in Sect. 3.1.3.3

Fig. 25 Optical micrographs showing differences in matrix microstructure in (a, b) the alumina/steel which has a ferrite-pearlite matrix and (c, d) the stoneblast/steel composites which has a bainitic matrix.

3.1.3.2 Cermet Hard Particle Composite

Pressure casting of cermet based composites was carried out at Caterpillar using the arrangement shown in Figs. 3 and 4. Generally improvements in casting integrity were noted with pressure casting. Notably, shrinkage cavities and porosity were almost completely eliminated with application of pressure.

Three fully infiltrated steel-carbides composite coupons obtained by pressure casting are shown in Fig. 26. The preforms for these three pressure cast samples were FeB powder coated WC-Co particles, WC-Co mixed with 5% fine MFB powder, and WC-Co mixed with coarse MFB particles. All three coupons had good shape on both top and bottom sides. However, some blowholes were still observed in cross section of the three coupons. A crack was also observed at the composite-steel interface, as shown in Fig. 27.

Fig. 26 Profiles of steel-WC/Co composite coupons made by pressure casting.

Fig. 27 Cross-section of pressure cast coupon #68 showing (a) the fully infiltrated composite and a blowhole near side wall, and (b) a higher magnification image showing the interface crack and micro-cracks in composite region.

One theory for the formation of the interfacial crack is that it was due to the differences in shear properties of the composite and unreinforced regions under the action of continuously applied high pressure through semi-solid and solid periods. Given the limitations of press control, one potential solution for this problem is to reduce the pressure holding time. In casting trials the pressure holding time was decreased from 60 seconds to 5 seconds. Examination of pressure cast coupons using this modified process revealed that the interfacial cracks were eliminated (Fig. 28).

Casting trials for the reduced volume fraction hard particle performs produced fully infiltrated composite coupons. The graphite does appear to provide oxidation protection for the steel powder during preheating and casting. In addition, reaction of graphite with the liquid steel improved the wettability of liquid steel on the steel powder and hard particles, thus assisting infiltration.

Fig. 28 Cross section pictures of #81 and #83 pressure casting coupons show elimination of large crack at interface between composite and base steel.

3.1.3.3 Microstructural Evaluation of Composites Pressure Cast with SAE 1045

SEM micrographs of cross-sections through the SAE 1045 composites as well as the neat 1045 steel matrix are shown in Figures 29–33. For both composites containing the WC-Co particles, there was significant dissolution of the particles by the steel (Figures 30 and 31). In addition to the presence of individual WC grains in the surrounding steel (e.g. Figures 30(e) and (f)), Fe levels in excess of 20% were detected within the particles at distances of a few hundred microns from the apparent particle/matrix interface (see, for example, the light gray phase, about 300–400 μm thick, around the particle in Figure 30(c)). The matrix microstructures differed significantly from the ferrite/pearlite structure characteristic of the neat matrix. Due to the dissolution of the WC grains and the added FeB or MFB phases, a significant fraction of carbides formed upon solidification. In addition, the matrix contained very little ferrite and was comprised of either very fine pearlite or bainite.

There was no significant interaction between the phases in the composites containing either alumina or alumina-zirconia particles (Figs. 32 and 33). The particles were uniformly distributed throughout the matrix (Figures 32(a) and 33(a)). The interfaces appeared distinct, without obvious dissolution of the particles or other chemical interactions (Figures 32(b, c) and 33(b, c)). Furthermore, the matrix exhibited a ferrite/pearlite microstructure, essentially the same as that of the neat alloy (compare Figures 32(d) and 33(d) with Figure 29). The only detectable interaction was in the form of trace amounts (about 1%) of Zr in the matrix in the composites containing alumina-zirconia particles. Furthermore, there was some impregnation of the melt into the pores within the latter particles. This feature is expected to enhance interface integrity through mechanical interlocking.

(a)

(b)

Fig. 29 SEM micrographs of the neat 1045 steel matrix after normalization, showing the expected ferrite/pearlite microstructure.

Fig. 30 SEM micrographs of the composite with FeB coated WC-Co particles. Note the extensive dissolution of the WC-Co.

Fig. 31 SEM micrographs of the composite with an WC-Co + 5%MFB particle perform. Note the dissolution of the WC-Co and the formation of unidentified matrix phases (likely from the dissolution of the MFB particles).

Fig. 32 SEM micrographs of the composite with spherical alumina particles, showing (a) uniform distribution of particles, (b,c) distinct interfaces at the steel/particle boundary without particle dissolution, and (d) ferrite/pearlite microstructure within the matrix, similar to that of the neat material (Fig. 29).

Fig. 33 SEM micrographs of a composite with alumina-zirconia particles, showing similar features to that in the alumina-containing composite, notably: (a) uniform distribution of particles, (b,c) distinct interfaces at the steel/particle boundary without particle dissolution, and (d) ferrite/pearlite microstructure within the matrix.

The effects of particle dissolution and chemical interactions with the FeB and MFB additions were manifested in significant increases in matrix hardness relative to the neat material (Figure 34). The matrix hardness in the MFB containing materials was about 5 times that of the neat steel. In the FeB material, it was more than twice as high. By contrast, both the alumina and alumina-zirconia containing composites exhibited hardness levels that were the same as that of the neat matrix, within the experimental scatter. This correlation is consistent with the noted similarities in the matrix microstructures.

Fig. 34 Summary of average matrix hardness within composites and of neat 1045 steel.

3.1.4 Fracture Toughness

Preliminary measurements of the mechanical performance of the HIPed composites (containing 4140 matrices) were made using single edge-notched flexure tests. The specimens were approximately 9 mm square in section and the notches were approximately half the edge length (~4.5 mm). The resulting load-displacement curves from two tests performed on each composite type are shown in Fig. 35. The alumina-zirconia/steel composite exhibited a significantly higher peak stress and a more rapid reduction in stress with displacement beyond the stress maximum. However, the work of fracture (obtained from the area under the load-displacement curve and the fracture area) was similar to that in the alumina/steel composite, falling in the range of ~12–19 kJ/m² (Fig. 36). The corresponding values for steady state fracture resistance as well as the nominal fracture resistances calculated at the load maximum are shown in Fig. 37. The steady state values are considerably higher than those at the load maximum: the latter being in the range of ~8–10 MPa√m and ~16–20 MPa√m in the alumina/steel and alumina-zirconia /steel composites, respectively. These differences are believed to be associated with the differences in matrix hardness and particle-matrix interaction.

A complementary measure of the performance is the net-section stress at the load maximum. The results are plotted against the matrix hardness in Fig. 38. The data appear to show a proportional relationship between notched strength and matrix hardness. At the simplest level, the notched strength in a ductile (notch-insensitive) metal might be expected to be comparable to

the yield strength, which in turn is given roughly by $H/3$ where H is hardness (in consistent units). Evidently this type of prediction overestimates the experimental measurements, by about a factor of 3 (Fig. 38). This is believed to be due to the fact that failure occurs along the particle-matrix interface (shown below), causing a reduction in the efficacy of load transfer from the matrix to the particles and hence a reduction in strength. Nevertheless, the data suggest an important effect of matrix hardness.

Fig. 35 Load-displacement response of composites in notched flexure tests (“stoneblast” = alumina-zirconia).

Fig. 36 Comparisons of work of fracture in the two composite types

Fig. 37 Estimates of fracture resistance of the composites, obtained from the load maximum (denoted K_o) and the work of fracture (K_{ss}).

Fig. 38 Effects of matrix hardness on the notched strength.

Fractography of the notched specimens reveals some plasticity within the steel and fracture along the particle-matrix interfaces. Some evidence of remnant steel on the particle surfaces was also seen. Fig. 39 shows a map of the opposing composite fracture faces from a test of an alumina-zirconia/steel composite. A “d” denotes debond, “f” indicates the particle fractured, and “m”

indicates that there is evidence of both particle fracture and debond. The presence of remnant steel on the particles and occurrence of particle fracture, suggest that the bonding of the steel to the particles is at least moderately good, and probably a significant fraction of the strength of the steel itself.

Fig. 39 Fracture map showing both halves of the fracture face from a test of an alumina-zirconia/steel composite having a 4140 matrix. A “d” indicates debond, an “f” indicates the particle fractured, and an “m” indicates an apparent mix of debond and fracture.

Further examination of the fracture surfaces from 4140 composites containing alumina and alumina-zirconia particles is provided in Figs. 40 and 41. Some evidence of remnant steel on the alumina particle surfaces was observed (Figs. 40(c, d)). This suggests that the bonding of the steel to the particles is at least moderately good.

Fracture toughness testing of composites having 1045 steel matrices was performed at UC Santa Barbara using notched 4-point bend coupons. The coupons were each about 10 mm x 10 mm in cross-section, with notches of half the depth. In order to understand the strength of the composites, unnotched bend bars were also tested.

The results from the notched and unnotched bend tests are shown in Figure 42. In the unnotched configuration, the WC/Co composites exhibited high strength (500-700 MPa) (Figure 42(a)). The FeB coated WC-Co containing composite fractured catastrophically, whereas the one having MFB mixed in a WC-Co preform showed a more significant post load-maximum “tail” in the load-displacement curve. The alumina and alumina-zirconia containing composites exhibited a lower peak stress but a subsequent “graceful” failure.

Fig. 40 (a, b) Low magnification images of the fracture surface of the alumina/steel composite. Remnants of steel on the alumina particles are seen in the backscatter images in (c) and (d).

Fig. 41 Low magnification images of the entire fracture surface of the alumina-zirconia/steel composite, in (a) secondary and (b) backscatter mode. The region at the top is part of the notch. The backscatter image shows the distribution of the stoneblast particles (medium gray) amongst the fractured steel matrix (light).

In the notched condition, the alumina-zirconia material displayed the best performance. Its strength was on par with the two WC-Co composites (300–400 MPa), and higher than that of the alumina composite (about 200 MPa). Furthermore, it exhibited the highest work of fracture: almost an order of magnitude greater than that for the WC-Co materials (Figure 43).

(a)

(b)

Fig. 42 (a) Unnotched and (b) notched bending response of the four composites. (S is span length)

Fig. 43 Work of fracture obtained from area under the notched specimen, load-displacement curves.

SEM observations of the fracture surfaces were broadly consistent with the mechanical measurements. Both of the WC-Co materials displayed planar fracture surfaces and extensive cleavage cracking within the matrix (Figures 44 and 45). The alumina composites failed by interface debonding followed by local plastic stretching of the intervening matrix ligaments (Figure 46(a)). These features are consistent with the low strength and the extended tail in the notched load-displacement response (Figure 42(b)). The alumina-zirconia composite failed by particle cracking followed by deformation of the matrix. Upon comparing matching fracture surfaces, there was evidence of steel within the fractured particles (see, for example, the particles denoted 3 and 4 in Figure 47).

Fig. 44 Fracture surfaces of the FeB coated WC-Co containing composites. (c) and (d) are of the same region, in backscatter and secondary electron imaging modes, respectively. Note the cleavage facets in the matrix region of (c).

Fig. 44 (cont.)

Fig. 45 Fracture surfaces of the WC-Co + 5%MFB containing composites at progressively increasing magnifications. Matrix fracture occurs predominantly by cleavage, as illustrated in (c) and (d).

Fig. 46 Matching fracture surfaces of the alumina containing composite, viewed in backscatter electron imaging mode. The numbers correspond to matching regions on the two fracture surfaces. The dark regions are alumina and the light regions are steel.

Fig. 47 Matching fracture surfaces of the alumina-zirconia containing composite, viewed in backscatter electron imaging mode. The numbers correspond to matching regions on the two fracture surfaces. The dark regions are alumina-zirconia and the light regions are steel. Most particles fail by fracture. The matrix "patches" on particle 4 on the left and particle 3 on the right are a result of steel infiltration into the particles.

3.1.5 Abrasive Wear Testing

Abrasive wear results for the composites in Table 2 were compared with the abrasive wear results measured for current premium direct hardened steel used in ground engaging tool (GET) applications. Table 10 presents the DSRW and impeller abrasion test results as a factor of improvement over the current GET steel. It is seen that in low stress scratching the improvement

in wear resistance is around 6 to 8x. In contrast, when impact and more severe gouging is introduced, material toughness becomes increasingly important. As a result the factor of improvement in impact-abrasion doesn't exceed 2X.

Table 10. Factor of wear improvement over current GET steel for DSRW and Impeller-Abrasion testing.

Composite	Matrix/Condition	DSRW	Impeller-Abrasion
FeB coated WC-Co	Q&T 4130	5.7	1.7
WC-Co+5%MFB	Q&T 4130	7.7	1.5

Unfortunately the data in Table 10 is derived from a limited number of measurements, due to the small number of samples produced. However the data is sufficient to provide some indication of performance trends. More thorough testing occurred using the pin-abrasion test with composites having a normalized SAE 1045 matrix. Given the size of the castings which UC Santa Barbara was able to pressure cast in their HIP, only the pin-abrasion test coupons could be extracted. As expected the composites provide a significant improvement over the neat matrix. The oxide based composites and the WC-Co+5%MFB/1045 composite all displayed similar wear resistance. In contrast, the FeB coated WC-Co reinforced composite displayed lower wear resistance than the other composites. The WC-Co reinforced composites appeared to be decreasing in relative wear resistance at higher test loads. One possible reason for this trend is the degradation and embrittlement of the WC-Co and WC-Co/Matrix interface during molten metal infiltration. Such degradation would be more likely to manifest itself at high abrasive loads.

When compared to GET steels, it is found that the oxide based composites are providing approximately 10x the wear resistance, while the WC-Co composites are providing 2x the wear resistance for FeB coated WC-Co and 5x for the WC-Co+5%MFB composites. Given that a component will only be selectively reinforced, a wear resistance of greater than 2x is required for doubling the component life. Therefore, based on the impeller-abrasion wear data and the pin-abrasion wear data, the FeB coated WC-Co composites in their current form are not providing adequate abrasive wear resistance to meet project goals. The wear resistance of the WC-Co+5%MFB reinforced composite would likely be able to produce a 2x increase in component life, however in a heavy impact application, this composite system would likely fall short of meeting component life targets. It is unknown how the oxide based composites would perform. However, given their higher fracture toughness, it is expected these composites would provide sufficient impact wear resistance to meet project goals.

Coupon wear surfaces were first examined in an SEM and then were metallographically mounted, ground and polished to reveal a cross section of the wear surfaces. Figure 48 shows that the neat matrix displayed relatively deep scratches, with material being removed along with the occurrence of a significant amount of plasticity in both the ferrite and pearlite phases (Figure 48(b)).

The wear processes for the WC-Co composites are shown in Figures 49 and 50. The particles are observed to be raised above the plain of the matrix and are therefore wearing at a slower rate than the matrix, thus reducing the overall wear rate of the composite by providing some protection to the matrix. In contrast to the neat matrix, only shallow scratches are observed in

the matrix of the WC-Co based composites and no significant subsurface matrix deformation is observed (Figures 49(e), 49(f), 50(e) and 50(f)). This is a result of the relatively high hardness of the matrix in these composites (Figure 34) and the high fraction of very hard carbides in the matrix which provide further wear resistance. Due to the carbide fraction in the matrix, the matrix wear process is likely dominated by microfracture of the carbides and localized plastic flow of the metallic matrix. The WC-Co particles appear to be worn by both scratching wear in the case of fine grain WC/Co particles (Figure 49(c) and (d)) and grain plucking in the case of coarse grain WC-Co (Figure 50(c) and (d)).

In the alumina and alumina-zirconia composites the particles provided substantial wear protection to the matrix (Figures 51 and 52). In these composites, the matrix was worn by a similar process to the neat matrix, given that they are of the same structure (Figures 51(e), 51(f), 52(e) and 52(f)). The scratches appear to be more shallow than those in the neat matrix, likely due to the protection provided by the particles. No scratches are observed on the particles. The particle wear mechanism appears to be primarily a result of microfracture and grain plucking (Figures 51(c), 51(d), 52(c) and 52(d)).

3.1.6 Process Cost Analysis

A cost analysis was performed for pressure cast steel MMCs. Moderate volumes were assumed, recognizing that composite components would likely only be used in severe mining applications. Two components were examined, (1) track-type tractor track shoe grousers, and (2) motor grader edges. For both components, the cost of the hard particle preform was the dominant cost. The use of a composite grouser would lead to a significant increase in undercarriage cost which may not be accepted by the customer. Given that the pin-abrasion (gouging wear) wear resistance is as much as 10x for WC-Co and oxide based composites, the life of the shoe would likely be dictated by the life of the steel plate, rather than the grouser. Ultimately additional analysis would be required to determine whether the purchase price for the composite shoes would justify the cost avoidance from multiple shoe replacements or regrousering.

For the motor grader edge, the estimated production cost is similar to or less than the current WC-Co tiled edge. The composite edge would provide improved toughness (particularly the oxide based composites) and a similar wear performance, and therefore could provide an advantageous, cost effective product. Although similar edges are not currently used as edges for buckets and dozers in mining, the MMC technology could be used to make such product. Overall, the analysis confirmed that a significant cost advantage could be realized if stoneblast (alumina-zirconia) particles could be used as the hard phase.

(a)

(b)

Fig. 48 Pin abrasion of neat matrix. (a) SEM image of wear surface showing deep gouges and extensive plastic flow. (b) Optical image of the cross section of a scratch showing localized plastic flow revealed through deformation of pearlite grains.

Fig. 49 Wear processes in Fe-B coated WC-Co composite where (a) and (b) show the general wear process where the hard particles (distinguishable in the backscattered SEM image shown in (b) as the light-colored phase) are providing some protection to the matrix, but are also experiencing some gouging abrasion, more closely shown in (c) and (d). The matrix (e) displays shallower scratches than seen in the neat matrix, largely due to the protection provided by the carbides in the matrix (light phase in (f)).

Fig. 50 WC-Co + 5%MFB composite where (a) and (b) show the general wear process with the hard particles (distinguishable in the backscattered SEM image shown in (b) as the light-colored phase) providing some protection to the matrix. In contrast to the case shown in Fig. 48, the WC-Co particle examined does not display any clear scratches (c). Instead the particles appear to be wearing by a progressive erosion of the cermet matrix and grain plucking (d). The matrix (e) displays shallower scratches than seen in the neat matrix, largely due to the protection provided by the carbides in the matrix (light phase in (f)).

Fig. 51 Wear processes in alumina-zirconia composite where (a) and (b) show the general wear process with the hard particles (distinguishable in the backscattered SEM (b) as the darker phase) providing significant protection to the matrix. The hard particles do not show any evidence of scratches/gouges (c). The particles appear to be wearing through microfracture and grain plucking (d). The matrix shows evidence of gouging wear and plasticity (e,f), with the gouges shallower than those in the neat matrix due to the protection provided by the hard particles.

Fig. 52 Wear processes in the alumina reinforced composite where (a) and (b) show the general wear process with the hard particles (distinguishable in the backscattered SEM (b) as the darker phase) providing significant protection to the matrix. The hard particles do not show any evidence of scratches/gouges (c). The particles appear to be wearing through microfracture and grain plucking, possibly aided by small scale plasticity (d). The matrix shows evidence of gouging wear and plasticity (e,f), with the gouges shallower than those in the neat matrix due to the protection provided by the hard particles.

3.2 Thermal Spray Coatings

For the first fusing iteration in 2001, a thermocouple was welded to the underside of one plasma sprayed rectangular substrate for preliminary arc lamp treatments. After a single pass with the arc lamp operating at 400A and at a traverse rate of 7mm/s, the substrate temperature rose from room temperature to approximately 360°C. An identical second pass raised the temperature to approximately 580°C. A third lamp pass at 900A and 8mm/s resulted in an approximate temperature of 950°C. It should be noted that manipulation issues with the arc lamp robot resulted in approximately a 25s delay between passes in all cases. Additionally, no coating containing the A4650 material was treated as a result of excessive smoke emanation from the coating during heating, which could have possibly harmed the lamp hardware.

Coatings were treated two separate ways with the arc lamp in an effort to understand the degree to which pre-heating of the coating prior to melting/fusing effects coating structure and coating-substrate bonding. Pre-heating was accomplished by twice scanning specimens at 400A and 7mm/s before a melting/fusing pass at 900A and 8mm/s. Separate specimens also underwent the melting/fusing pass without any pre-heating step.

As seen in Figs. 53 through 56, a metallurgical bond between the substrate and tool steel (M4 and T15) coatings was not achieved, even with the pre-heating step. However, the agglomeration of porosity is indicative of a majority of the coatings having been molten during processing. Images of coatings not having undergone the pre-heating step were also not metallurgically bonded to the substrate and are therefore, not shown. Only a small fraction of the TiC particles in the starting powders were present in the coating after spraying. This result is still under investigation, but it is believed that the TiC particles rebound from the substrate during spraying because they were not molten. One remedy for this could be to generate starting powders in which the tool steel and TiC are well bonded.

Figs. 57 and 58 show the CPT alloy as-sprayed and after arc lamp processing, respectively. By pre-heating the coating before the high power arc lamp pass, the coating appears to have been partially molten through its thickness and a metallurgical bond was achieved at the interface. Macroscopically, the coating surface was smoothed yet the edges were only slightly rounded, thus indicating the presence of both solid and liquid phases during arc lamp treatment. When treated in the absence of pre-heating steps, the coating completely separated from the substrate, likely due to solidification stresses, high temperature gradients, and/or differences in thermal expansion characteristics between the coating and substrate. Note that the as-sprayed coating thickness was approximately 1mm. However, during sectioning of the coating treated with pre-heating steps, part of the coating broke away and the remainder of the cross-section is shown in Fig. 58.

The 70/30 (weight ratio) mixture of the CPT alloy with an 80%Ni-20%Cr alloy also allowed for a metallurgical bond at the coating-substrate interface. However, significantly more porosity is seen in the coating after arc lamp processing than is typically seen in HVOF sprayed coatings. The origin of this porosity is believed to be the result of the agglomeration of intersplat porosity and oxide inclusions inherent to thermal spray coatings.

(a)

(b)

Fig. 53 Cross-section SEM image of the as-sprayed M4+30vol.%TiC on 80/20 M4 +30vol.%TiC/M4 coating at (a) low magnification and (b) higher magnification of the coating-substrate interface.

As seen in Fig. 59, a metallurgical bond appears to have occurred at the interface between the CrC-NiCr coating and substrate, but was partially fractured at some point during arc lamp

processing. Some cracking was also present in an orthogonal direction to the coating surface (Fig. 60). As with the CPT coating, this cracking is believed to have resulted from solidification stresses, high temperature gradients, and/or differences in coefficient of thermal expansion.

(a)

(b)

Fig. 54 Cross-section SEM image of the M4+30vol.%TiC on 80/20 M4 +30vol.%TiC/M4 coating after arc lamp treatment at (a) low magnification and (b) higher magnification of the coating-substrate interface. The coating was scanned twice by the lamp at 400A and a traverse rate of 7mm/s, then once at 900A and 8mm/s.

(a)

(b)

Fig. 55 Cross-section SEM image of the as-sprayed T15+30vol.%TiC on 80/20 T15 +30vol.%TiC/T15 coating at (a) low magnification and (b) higher magnification of the coating-substrate interface.

(a)

(b)

Fig. 56 Cross-section SEM image of the T15+30vol.%TiC on 80/20 T15 +30vol.%TiC/T15 coating after arc lamp treatment at (a) low magnification and (b) higher magnification of the coating-substrate interface. The coating was scanned twice by the lamp at 400A and a traverse rate of 7mm/s, then once at 900A and 8mm/s.

(a)

(b)

Fig. 57 Cross-section SEM image of the as-sprayed CPT coating at (a) low magnification and (b) higher magnification.

(a)

(b)

Fig. 58 Cross-section SEM image of the CPT coating after arc lamp treatment at (a) low magnification and (b) higher magnification of the coating-substrate interface. The coating was scanned twice by the lamp at 400A and a traverse rate of 7mm/s, then once at 900A and 8mm/s.

(a)

(b)

Fig. 59 Cross-section SEM image of the 70/30 CPT alloy/NiCr alloy coating after arc lamp treatment at (a) low magnification and (b) higher magnification of the coating-substrate interface. The coating was scanned twice by the lamp at 400A and a traverse rate of 7mm/s, then once at 900A and 8mm/s.

(a)

(b)

Fig. 60 Cross-section SEM image of the 500 μ m thick CrC-NiCr composite coating after arc lamp treatment at (a) low magnification and (b) higher magnification of the coating-substrate interface. The coating was scanned twice by the lamp at 400A and a traverse rate of 7mm/s, then once at 900A and 8mm/s.

Table 11 lists the microhardness of each coating as-sprayed and after arc lamp treatment(s). In the vast majority of cases, arc lamp processing increased coating hardness. This increase likely resulted from a combination of diffusion bonding between individual lamella in the coatings, reduction of microporosity, and coating homogenization. The CPT coating exhibited an extremely high hardness, but was softened significantly when mixed with only 30wt.% NiCr.

Table 11. Microhardness results for coatings in the as-sprayed and post-arc lamp treatment conditions.

Coating Composition*	Microhardness, HV _{500g} **		
	As-sprayed	After HC Arc Lamp Pass	After 2 x LC, then HC Arc Lamp Pass
M4+30vol.%TiC	334	457	853
M4+30vol.%TiC on 80/20 M4 +30vol.%TiC/M4	352	521	761
60/40 M4+30vol.%TiC/M4	307	544	882
M4	323	751	499
M4 on 80/20 M4/A4650	349	not treated w/ lamp	not treated w/ lamp
60/40 M4/A4650	448	not treated w/ lamp	not treated w/ lamp
T15+30vol.%TiC	388	453	798
T15+30vol.%TiC on 80/20 T15+30vol.%TiC/T15	437	681	700
60/40 T15+30vol.%TiC/T15	371	539	792
T15	358	338	551
A4650	229	not treated w/ lamp	not treated w/ lamp
CPT alloy	949	1270	1034 (1952***)
CrC-NiCr composite	not tested	not treated w/ lamp	1032
CrC-NiCr composite	not tested	not treated w/ lamp	932
70/30 CPT alloy/NiCr alloy	not tested	not treated w/ lamp	521 (446***)

* Numbers indicate weight percents of components unless otherwise indicated.

** Hardness tests were performed on a polished coating cross-section, unless otherwise noted.

*** Hardness tests were performed on top surface of coating.

Coating composition was found to play a major role in the adherence and macroscopic wetting characteristics during the second fusing iteration of this program. Fig. 61(a) shows a 434L stainless steel – 60wt.% CPT coating that “balled” somewhat during fusing due to surface tension and wetting effects. Fig. 61(b) shows a M4 tool steel – 60wt.% CPT coating that remained dimensionally stable during fusing. Of the compositions investigated, only the coatings containing 434L had unacceptable dimension changes during arc lamp treatment.

All fused coating compositions investigated achieved some degree of metallurgical bonding to the steel substrates as can be seen in Figs. 62 through 80. Coatings sprayed with additions of elemental Cr exhibited large amounts of oxide formation during fusing. The oxide, which often floated to the top of the coating as can be seen in Fig. 75(c), was determined to be very high in Cr content by energy dispersive spectroscopy (EDS). Minimal differences in microhardness and microstructure were observed in identical coating compositions, but fused with different arc lamp parameters. Therefore, only coatings treated with two pre-fuse and one post-fuse pass are shown.

As expected, coating hardnesses increased proportionally with CPT additions. In nearly all cases, arc lamp fusing increased coating hardness and oftentimes hardness was approximately doubled. Bulk and inter-lamellar oxides and porosity were also agglomerated during fusing. Some oxides remained at the coating-substrate interface and a minimal amount of grit blast media was also embedded in some specimen substrates.

No cracking was observed in any of the fused coatings. Three coatings, Cr – 40(53CP – PT), Cr – 60(53CP – PT), and NiCr – 90CPT cracked and/or spalled significantly during sectioning for metallography. This brittleness is of concern and somewhat inherent to extremely hard, fused materials that are under significant stress due to solidification, high temperature gradients during processing, and/or thermal expansion differences. In Figs. 52 through 70, the lightest gray particles are hard, complex ternary intermetallics, which was confirmed by x-ray diffraction. The dark gray, acicular structures are composed of primarily Cr and Fe and are another complex ternary intermetallic. In the case of coatings containing M4 tool steel, V and W are apparently dispersed throughout both the precipitates and matrix.

(a)

(b)

Fig. 61 Macroscopic images of the (a) 434L – 60CPT and (b) M4 – 60CPT coatings after arc lamp treatment. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 62 Cross-section SEM (compositional) images of the M4 – 20CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 63 Cross-section SEM (compositional) images of the M4 – 40CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 64 Cross-section SEM (compositional) images of the M4 – 60CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 65 Cross-section SEM (compositional) images of the M4 – 80CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 66 Cross-section SEM (compositional) images of the 316L – 40CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 67 Cross-section SEM (compositional) images of the 316L – 60CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 68 Cross-section SEM (compositional) images of the 316L – 80CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 69 Cross-section SEM (compositional) images of the 434L – 60CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 70 Cross-section SEM (compositional) images of the 434L – 80CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 71 Cross-section SEM (compositional) images of the NiCr – 60CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 72 Cross-section SEM (compositional) images of the NiCr – 80CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 73 Cross-section SEM (compositional) images of the NiCr – 90CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 74 Cross-section SEM (compositional) images of the Cr – 20CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 75 Cross-section SEM (compositional) images of the Cr – 40CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 76 Cross-section SEM (compositional) images of the Cr – 60CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 77 Cross-section SEM (compositional) images of the Cr – 80CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 78 Cross-section SEM (compositional) images of the 50CP-38PT-Cr alloy coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 79 Cross-section SEM (compositional) images of the Cr-80(53CP-PT) coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

(a)

(b)

(c)

Fig. 80 Cross-section SEM (compositional) images of the (M4 + 40v. Al_2O_3) – 50CPT coating after arc lamp treatment at (a) low magnification, (b) higher magnification of the coating-substrate interface, and (c) higher magnification of the middle of the coating. Little or no Al_2O_3 was deposited during spraying and is therefore, not seen in these cross-sections. The lamp scanning parameters for this coating were as follows: 7mm/s @ 500A, 6mm/s @ 650A, 5mm/s @ 900A, then 6mm/s @ 400A.

The wear testing results are shown in Table 12. In general, the wear resistance varied proportionally with the coating CPT alloy content. Two coatings, 316L stainless steel – 40wt.%CPT and Cr – 60wt.%(53wt.%CP – PT), exhibited particularly high volume losses of 61.9 and 66.9mm³, respectively. Because replicate runs were not possible, it remains unknown if these results are typical or anomalies. Twelve of the coatings exhibited an approximate volume loss of at most 1/5th that of H13 tool steel heat-treated to 47-48HRC.

Table 12. ASTM G65 B wear testing results from ARC.

Coating Composition*	Mass Loss, g	Apparent Porosity, %	Apparent Density**, g/cm³	Calculated Vol. Loss, mm³
M4 – 40 CPT	0.0529	4.1	7.58	7.0
M4 – 60 CPT	0.0303	4.8	7.49	4.0
M4 – 80 CPT	0.0285	4.0	7.52	3.8
316L – 40 CPT	0.4872	0.7	7.86	61.9
316L – 60 CPT	0.0617	6.1	7.40	8.3
316L – 80 CPT	0.0259	4.8	7.46	3.5
434L – 60 CPT	0.2124	2.6	7.60	28.0
434L – 80 CPT	0.0287	7.3	7.23	4.0
NiCr – 60 CPT	0.1270	6.1	7.61	16.7
NiCr – 80 CPT	0.0302	3.8	7.65	3.9
NiCr – 90 CPT	0.0331	6.5	7.36	4.5
Cr – 20 CPT	0.2530	3.7	7.05	35.9
Cr – 40 CPT	0.1100	14.0	6.4	17.2
Cr – 60 CPT	0.0785	5.7	7.13	11.0
Cr – 80 CPT	0.0494	7.5	7.1	7.0
50CP – 38PT– Cr alloyed	0.0580	12.7	6.75	8.6
53CP – PT	0.1655	9.1	7.09	23.3
Cr – 40(53CP – PT)	0.1967	11.8	6.56	30.0
Cr – 60(53CP – PT)	0.5054	---	assume 7.56	66.9
Cr – 80(53CP – PT)	0.1933	16.4	6.42	30.1
(M4 + 40v.Al ₂ O ₃)– 50 CPT	0.0760	2.0	6.96	10.9

* Numbers indicate weight percents of components unless otherwise indicated.

** ρ M4 = 7.97g/cm³

ρ CPT = approx. 7.8g/cm³

ρ NiCr = approx. 8.56g/cm³

ρ 316L = 8.0g/cm³

ρ 434L = 7.8g/cm³

ρ Cr = 7.2g/cm³

Photographs of the eight successfully fused cylinders from the third fusing iteration of this program are shown in Fig. 81. The black asperities on the coatings are relatively loosely adhered surface oxides, resulting from processing without the aid of a completely inert atmosphere and the floatation of interlamellar oxides inherent to thermal sprayed coatings. All coatings responded well to the arc lamp processing in terms of dimensional stability, despite being rotated and the flow of Ar gas across the molten or semi-molten surface. Every coating exhibited some degree of radial cracking after fusing and longitudinal cracking along the line at which the arc lamp was shut off. Previous microscopy indicated such cracks extend through the hardest coating layers, but arrest at the more compliant base layers or base metal.

A slightly too short arc length, rapid cooling from the cylinder I.D., and lower powder density at the extremities of the arc prevented metallurgical bonding at the coating edges. This result was visible with low magnification optical microscopy. However, scanning electron microscopy indicated that a significant amount of, if not complete, metallurgical bonding was achieved across the majority of the coatings widths as can be seen in Figs. 82 and 83.

As seen in Fig. 84, microhardness profiles of coated cylinder cross-sections indicated that internal water-cooling prevented significant tempering near the I.D. surfaces. Base steels immediately below the coatings were tempered considerably after the 650A arc lamp passes. However, these regions were similar in hardness to the as heat-treated cylinders after the 900A arc lamp pass. Refer to Fig. 35. It appears that the base steels near the coatings were re-austenitized and quenched from the internal water-cooling. Therefore, fusing of coatings onto cylinders prior to heat-treating may be a more desirable processing sequence from the standpoint of minimizing coating stresses arising from high temperature gradients.

Cylinder no. 2
(a)

Cylinder no. 3
(b)

Cylinder no. 4
(c)

Cylinder no. 5
(d)

Cylinder no. 6
(e)

Cylinder no. 7
(f)

Cylinder no. 9
(g)

Cylinder no. 10
(h)

Fig. 81 Photographs of post-fused FGM coatings on the outside diameters of eight cylinders.

(a)

(b)

Fig. 82 Compositional backscattered electron SEM images of the 316L – CPT FGM coatings after fusing at (a) approximately 6mm/s at 650A and (b) approximately 6mm/s at 650A followed by 6mm/s at 900A.

(a)

(b)

Fig. 83 Compositional backscattered electron SEM images of the NiCr – CPT and M4 – CPT FGM coatings after fusing at (a) approximately 6mm/s at 650A and (b) approximately 6mm/s at 650A followed by 6mm/s at 900A.

Fig. 84 Microhardness profiles of four cylinders after arc lamp processing. The baseline cylinder microhardness profile is included for reference. Lines are intended only a rough guide for the eye.

Fig. 85 Microhardness profiles of the outermost sections of the four arc lamp processed cylinders and the baseline (heat-treated) cylinder.

Photographs of the tested component are shown in Fig. 86. In all cases, a traditional wear scar in the coating was not evident. Instead, the coating surface merely “polished” during testing. Note that the radial cracks in the fused coating were a result of the fusing operation rather than wear testing. All but one of the coatings (cylinder no. 7) flaked to some degree during testing. However, two additional coatings flaked only in one small area each and therefore, were considered successful. In all cases where the portions of coating flaked, the coating debonded from the substrate rather than within the coating thickness.

The flaking of coatings was thought to be a result of too little metallurgical bonding between the coating and base material after arc lamp fusing. A representative compositional SEM image affirming this thought shown in Fig. 87. This issue was a result of slightly too short of an arc

length, rapid water-cooling from the bushing I.D., and lower power density at the extremities of arc.

The three cylinders that survived wear testing showed outstanding abrasion resistance while lengthening the wear life of the mating steel surface. The three successful coatings increased the wear lives of coated components by a factors of approximately 5.9 - 8.4X while maintaining or slightly increasing the life of the non-coated mating components. The as-fused coating surface proved to be smooth enough to prevent gouging of the softer mating surface. The increased wear life of the mating steel is thought to be the result of the bushing (coating) maintaining roundness rather than flattening through wear.

Cylinder no. 2, tested 1,000 cycles
(a)

Cylinder no. 4, tested 4,000 cycles
(b)

Cylinder no. 5, tested 1,000 cycles
(c)

Cylinder no. 6, tested 4,000 cycles
(d)

Cylinder no. 7, tested 4,000 cycles
(e)

Cylinder no. 9, tested 1,000 cycles
(f)

Cylinder no. 10, tested 1,000 cycles
(g)

Fig. 86 Photographs of coated (fused) cylinders after wear testing.

Fig. 87 Compositional backscattered electron SEM image of cylinder no. 2 after wear testing. The cross-sectional view of flaking coating clearly indicates unsuitable metallurgical bonding for the high-stress wear test.

The final fusing iteration of plasma sprayed coatings was performed with identical hollow cylinders, but without water-cooling the I.D. during fusing. Photographs of the fused coatings are shown in Fig. 88, with the exception of cylinder no. 4F, which was left at ORNL for analysis.

All coatings were lightly grit blasted to remove relatively loosely adhered surface oxides, which resulted from fusing without the aid of an inert atmosphere and the floatation of interlamellar oxides inherent to thermal sprayed coatings. All coatings responded well to the arc lamp processing in terms of dimensional stability.

In contrast to tubes that were internally water-cooled during fusing and reported on previously, circumferential and longitudinal coating cracking was not prevalent. Only cylinder no. 1F exhibited circumferential cracks that could be resolved by the unaided eye. This was likely the result of incomplete fusing of the coating due to a lower arc lamp current during the fusing pass than was used with any other specimen.

Cylinder no. 1F
(a)

Cylinder no. 2F
(b)

Cylinder no. 3F
(b)

Cylinder no. 5F
(d)

Cylinder no. 6F
(e)

Cylinder no. 7F
(f)

Cylinder no. 8F
(g)

Cylinder no. 9F
(h)

Cylinder no. 10F
(i)

Cylinder no. 11F
(j)

Cylinder no. 12F
(k)

Cylinder no. 13F
(l)

Cylinder no. 14F
(m)

Cylinder no. 15F
(n)

Cylinder no. 16F

(o)

Fig. 88 Photographs of fused coatings after light grit blasting to remove surface oxidation.

Wear testing equipment maintenance prevented the testing of all fused coatings. Also, misalignment of the coated cylinder and the mating steel component resulted in point loading (invalid testing) of two of the four coatings. The coatings that were point loaded cracked and partially flaked off of the substrate. A photograph of one successfully tested component (cylinder no. 3F) is shown in Figs. 89 and 90. As observed previously, a traditional wear scar in the coating was not evident. Instead, the coating surface merely “polished” during testing.

The flaking of coatings is thought to be a result of imperfect metallurgical bonding between the coating and base material after arc lamp fusing. Further optimization of the arc lamp fusing process is needed to achieve the desired degree of bonding.

The coating that survived wear testing showed outstanding abrasion resistance while lengthening the wear life of the mating steel surface. This coating increased the wear life of coated components by a factor of five while increasing that of non-coated mating component by 20%. As reported in previous testing iterations, the as-fused coating surface proved to be smooth enough to prevent gouging of the softer mating surface. The increased wear life of the mating steel is thought to be the result of the cylinder (coating) maintaining roundness rather than flattening through wear.

Cylinder no. 5F exhibited abrasion resistance nearly identical to current production carburized and hardened steel and resulted in no wear life change of the mating steel surface. This coating was tested for 8,000 cycles and the coating thickness was worn through to the steel substrate.

This marks the first time that a coating has been tested until complete wear-through. Noting that the purpose of this particular coating was to act as a tough, compliant base layer, the test result was considered highly successful. A digital image of the successfully tested component is shown in Fig. 91.

(a)

(b)

Fig. 89 Digital images of coated (fused) cylinder no. 3F (a) before and (b) after 7,000 wear testing cycles.

(a)

(b)

Fig. 90 Optical micrographs of coating no. 3F (a) as-fused and (b) after wear testing.

Fig. 91 Photograph of coated (fused) cylinder no. 5F after 8,000 wear testing cycles. The box highlights the area of complete coating wear-through.

4.0 CONCLUSIONS

4.1 Steel Matrix Composites

Ultimately two categories of materials and two sets of distinct conclusions came out of the Steel Matrix Composites effort. Steel composites reinforced with cermet (WC-Co or Mo-Fe-B mixes) hard particles were successfully pressure cast using the process developed at Caterpillar. However, solidification rates were insufficient to prevent dissolution of the particles. Not only did particle dissolution hurt the integrity and performance of the hard particles, but carbide precipitation in the steel matrix significantly lowered the fracture toughness of these materials. As a result of the matrix embrittlement and particle degradation, the wear results for these composites were mixed. Although some configurations met the project targets for high stress gouging wear, the targets were not met for impact abrasion due to the poor toughness of these composites.

In contrast, steel composites reinforced with alumina particles, particularly alumina-zirconia, consistently met project goals for high stress gouging wear. Given the good measured toughness of these materials, it is also expected that they would meet or exceed project goals for impact

wear applications. The improved toughness over the cermet based composites results from the fact that the particles have minimal interaction with the steel matrix, leaving the matrix ductile and tough. Unfortunately, no good mechanism was identified in this project for making hard particle preforms with the alumina-zirconia particles, and attempts to infiltrate the alumina particles with the Caterpillar process failed to obtain any infiltration. The hot isostatic process used to create make the alumina-based composites at UC Santa Barbara would not be production viable. However, it is felt that with more effective steel delivery (less heat loss), higher preheating temperature on the particles, and higher casting pressures it would likely be possible to produce alumina composites using the Caterpillar process.

Cost analysis further emphasizes the benefit of pursuing alumina-based composites. Due to the lower cost and weight of the oxide particles, components which would not likely be attractive when made with WC-Co particles, could be competitive when made with alumina particles. Ultimately, then, the goals of the Steel Matrix Composite effort were not met, however the steel/alumina-zirconia system could be developed into a cost-effective, highly abrasion resistant material, with further casting and preform process development.

4.2 Thermal Spray Coatings

Arc lamp processing serves to agglomerate porosity inherent to thermal spray coatings, increase coating hardness, and metallurgically bond the coating and substrate. The coatings melting and wetting characteristics have been optimized through compositional variation sufficiently to allow for dimensional stability and metallurgical bonding. Fluxing agents and melting point depressants seem to allow for better coating behavior during arc lamp treatment.

The number of candidate systems for usage in FGM designs was narrowed to three: M4-CPT, 316L-CPT, and NiCr-CPT. These systems may be used alone or in conjunction with one or both of the other two. All three systems are believed to be sufficiently tailorable in composition to be able to serve as bond coats and wear layers.

Although metallurgical bonding was readily achieved in a series of experiments, imbedded grit blast media could potentially reduce the bond strength of the coating. For experimental purposes, alumina grit can be used for substrate surface roughening, but chilled iron grit would likely be a better choice for coated machine components that will be subjected to high loads and impacts. Overall, arc lamp fusing of thermal spray coatings serves to metallurgically bond the coating and substrate such that very hard coatings can be employed in high-stress environments where conventional thermal spray coatings would delaminate.

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