Environmentally-Compliant Corrosion and Wear Protection Coatings

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Abstract

Environmental, Safety and Health (ES&H) issues associated with many wear and corrosion resistant coatings have been identified on the Office of the Secretary of Defense's (OSD) Action and Watch lists. This list includes Electrolytic Hard Chrome (EHC) and tungsten carbide cobalt (WC-Co) coatings. Inovati[1] has been developing ES&H compliant coatings to replace these hazardous materials. Potential candidates identified as part of a United States Air Force Phase I STTR project included aluminum, iron, and titanium-based alloys formulated without constituents on the OSD Action/Watch list. The challenge for the research project was to select corrosion resistant alloys configured as Particle Reinforced Metal Matrix Composite (PRMMC) to permit application of wear resistant coatings using the Kinetic Metallization™ (KM) process[2] and equipment. The initial research work demonstrated that an iron-molybdenum alloy when formulated as a PRMMC coating had the potential to meet the ES&H requirements for alternative wear/corrosion resistant coatings comparable to the legacy EHC and WC-Co coatings. The Phase II STTR research continued the development activity with an amorphous iron alloy formulated with various hard phase constituents. This paper will present the results of the qualification tests performed on these ES&H compliant coatings and potential military and industrial applications.

Keywords: Non-Heavy Metal Materials, Amorphous Iron Coatings, Kinetic Metallization, Wear Resistant Coatings, Corrosion Resistant Coatings

Introduction

Coating materials and coating methods used to protect U.S. military aircraft and weapon systems from corrosion and wear need compliant alternatives in order to keep up with dynamic ES&H regulations. Multiple military repair facilities have been found to be non-compliant with OSHA exposure limits to toxic heavy metals such as hexavalent chromium and cadmium. Thus, the Air Force is supporting research to develop alternatives to the dangerous materials identified on the OSD Emerging Contaminants Watch or Action Lists (1).

Inovati was awarded a US Air Force Phase I STTR project (FA8650-15-M-5044) in 2015, and a follow-on Phase II STTR project (FA8650-16-C-5022) in 2016, to investigate alternative materials that could potentially replace Electrolytic Hard Chrome (EHC) and tungsten carbide

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[1] Inovati is DBA for Innovative Technology, Inc, PO Box 60007, Santa Barbara, CA 93160.

[2] Kinetic Metallization™ (KM) is solid-state process developed and patented by Inovati.

cobalt (WC-CO) coatings with ES&H compliant materials. Of the primary structural metals only alloys of Al, Fe, and Ti are not on either the Watch or Action Lists. Thus, we studied all three systems in Phase I with additions of hard-phase constituents also not identified on the OSD Emerging Contaminants lists.

The Phase I research results conducted by Inovati in collaboration with The Aerospace Corporation (STTR subcontracted Co-Investigator) demonstrated that an iron-based alloy (FeMo) coating co-deposited with a hard phase (SiC or Al2O3) PRMMC admixture had good prospects to provide an environmentally compliant alternative for EHC and WC-Co coatings. This PRMMC material when applied with the low temperature KM process yielded coatings with improved wear resistance comparable to High Velocity Oxygen Fuel (HVOF) WC-17Co coatings.

Prior research work performed by Hashimoto, et al. (2) demonstrated excellent corrosion resistance for amorphous FeMoPC alloy ribbons. Based on this historical information, Inovati elected to develop and test an amorphous-iron (FeMoPC) powder formulation in Phase II in order to enhance the corrosion resistant properties of the basic FeMo alloy system.

Kinetic Metallization™ (KM) (3,4) is a low temperature process that enables deposition of these novel coating materials without modifying the amorphous structure. This process also minimizes thermal oxidation of the coating materials and precludes thermal degradation of component material properties, while eliminating generation of toxic fumes frequently produced with high-temperature thermal spray processes such as HVOF.

The KM process has been shown to be less costly than HVOF for application of WC-Co wear resistant coatings (5). In fact, KM applied WC-Co was selected over HVOF to replace EHC coatings on aircraft hydraulic gear shafts (Macy and Ganenzle 2014) and has been more recently used to applied wear resistant coatings to turbo-pump parts for the commercial space industry. KM applied coatings also eliminate the need for hydrogen bake out of High Strength Steel (HSS) compared to electrolytic processes.

This paper summarizes the findings of the STTR Phase I & II research in collaboration with investigators at The Aerospace Corporation to continue research to initially qualify the FeMoPC-alloy PRMMC coatings a potential replacement for the hazardous EHC and HVOF WC-Co coatings currently used to protect military aircraft components and weapon systems.

Experimental Procedure

Study of Environmentally Compliant FeMo Alloys

ES&H compliant coating materials identified in Phase I of Air Force funded STTR included the iron-based (FeMo) alloys with moderate corrosion resistance. The focus of the Phase II research was to investigate the corrosion and wear resistance of an amorphous-iron PRMMC material when blended with an ES&H compliant hard-phase constituent (e.g., as Al_2O_3, SiC or B_4C).

Inovati discovered that iron powders alloyed with molybdenum and phosphorus could be produced as a corrosion-resistant matrix material. A rapid-solidification atomization process was used to produce an amorphous structure for the FeMoPC alloy. Alloying with molybdenum enables the formation of a hydrated iron oxy-hydroxide film to passivate the iron alloy in a corrosive environment as demonstrated by Hashimoto, et al. (2). To enhance the corrosion resistance of the FeMo alloy system, phosphorous was added, which also inhibits grain growth during rapid solidification of the powder. This approach was pursued in Phase II of the research work to optimize the corrosion resistance of the basic FeMo alloy system. The proprietary amorphous-iron powder selected for conducting this research work is designated as a FeMoPC alloy.

Kinetic Metallization Process and Equipment

Kinetic Metallization is a spray deposition process that enables coating of various materials at low temperatures. This solid-state spray deposition technology eliminates any hazards associated with the evolution of toxic fumes characteristic of some thermal spay technologies and precludes degradation of the component mechanical properties. Additionally, because the KM process does not use any chemicals it provides an opportunity for application of environmentally benign coating materials that will meet the dynamic ES&H regulations.

For this project, Inovati used the low temperature KM-Production Coating System (KM-PCS) shown in Figure 1 for deposition of the amorphous-iron coatings to preclude any grain growth of the coating materials during fabrication of test specimens. This system comprises a KM Control Cabinet with dual Powder Fluidizing Units (PFU) and an attached KM Nozzle Gun. A 6-axis robot for articulating the KM Gun while spraying coupons or parts was mounted within the spray enclosure, while a 7th rotation axis enables spraying rods, tubes, and other axisymmetric samples and parts.

Figure 1

Photograph of Kinetic Metallization-Production Coating System (KM-PCS)

Optimization of KM Process Parameters for Various Powder Formulations

Inovati developed powder specifications to establish the best-suited powder properties for the KM process. These specifications encompassed powder characteristics to ensure not only the highest quality of repair materials, but also the efficiency of the KM process. The powder specifications include:

- Particle size distribution < 20-µm for optimum wear resistant coatings
- Incorporation of reinforcement constituents of comparable size < 20-µm in the basic PRMMC powder formulations.

A parametric study was used to determine optimum conditions for spraying the various test powders. Variation of parameters for this purpose included:

- Gas type
- Spray pressure and temperature
- Powder pre-treatments and formulations with 2nd phase constituents
- Powder feeding rates
- Traversing speed of the deposition nozzle versus coating thickness

The KM nozzle produces a very directed and narrow jet, which requires overlapping of adjacent strokes to provide a contiguous and smooth layer of any material. In addition, the highest deposition efficiency of the KM process is obtained with the nozzle jet impinging perpendicular to the component surface to within a tolerance of ±15°.

The optimized KM spray parameters for deposition of the various PRMMC powder formulations are listed below in Table 1.

Table 1. Typical KM Spray Parameters

KM Parameter	Amorphous Fe-Based Materials
Helium Gas Pressure (psig)	60
Nozzle Throat Temperature (°C)	400
Deposition Temperature (°C)	200
Powder Feed Rate (g/m)	25-30
Translation Speed (in/s)	5
Nozzle Standoff (mm)	10

Results

Microscopic Analysis of Amorphous-Iron PRMMC Coatings

The Phase I work demonstrated that a FeMo/SiC PRMMC coating applied with the KM process had good potential as an environmentally compliant wear and corrosion resistant coating material. To enhance the properties of these FeMo—SiC materials, Inovati elected to pursue Phase II research work using an amorphous-iron metal matrix formulated with hard phase constituents (e.g., SiC, Al_2O_3, or B_4C). These coatings were expected to exhibit improved wear properties to potentially replace hazardous EHC and HVOF WC-Co coatings on high-strength steel (HSS) components.

The challenge for Phase II project was the development of wear resistant PRMMC's with hard phase concentrations (e.g., SiC, Al_2O_3, or B_4C) sufficiently high to enhance the wear performance. This required formulating powders with an ultra-fine particle size distribution (< 20 micrometers) for both the hard phase constituent and the ductile metal matrix. The ultra-fine particle size is also a prerequisite for obtaining wear resistant coating that can be ground and polished to finishes less than Ra of one micrometer.

Amorphous-iron alloy powders produced by a proprietary manufacturer were blended with 2nd phase powders (e.g., SiC, Al_2O_3, or B_4C) in a sonic blender for periods up to 15 minutes per batch to ensure uniform compositions. Typically, the SiC and B_4C powders were blended 50% by volume with the amorphous iron-based powder, while 30 volume percent was used for the Al_2O_3 formulations.

X-Ray Diffraction of Amorphous Powders and Coatings

Samples of amorphous-iron powders were delivered to our STTR partner, The Aerospace Corporation, for testing the amorphous characteristics of the KM powders via X-ray diffraction. Using their PANalytical X'Pert Pro diffractometer with copper X-ray radiation, the spectral results of these tests are displayed in Figure 2. The broadened peaks at approximately 44º and 77º compared favorably with the vendor supplied X-ray diffraction data and are characteristic of amorphous iron materials.

A sample of the amorphous-iron PRMMC coating deposited at KM helium gas operating temperatures of 870 ºC (deposition temperature of approximately 200 ºC) shows an amorphous-iron structure in X-ray diffraction (Figure 3) as compared to the BCC 440C crystalline steel.

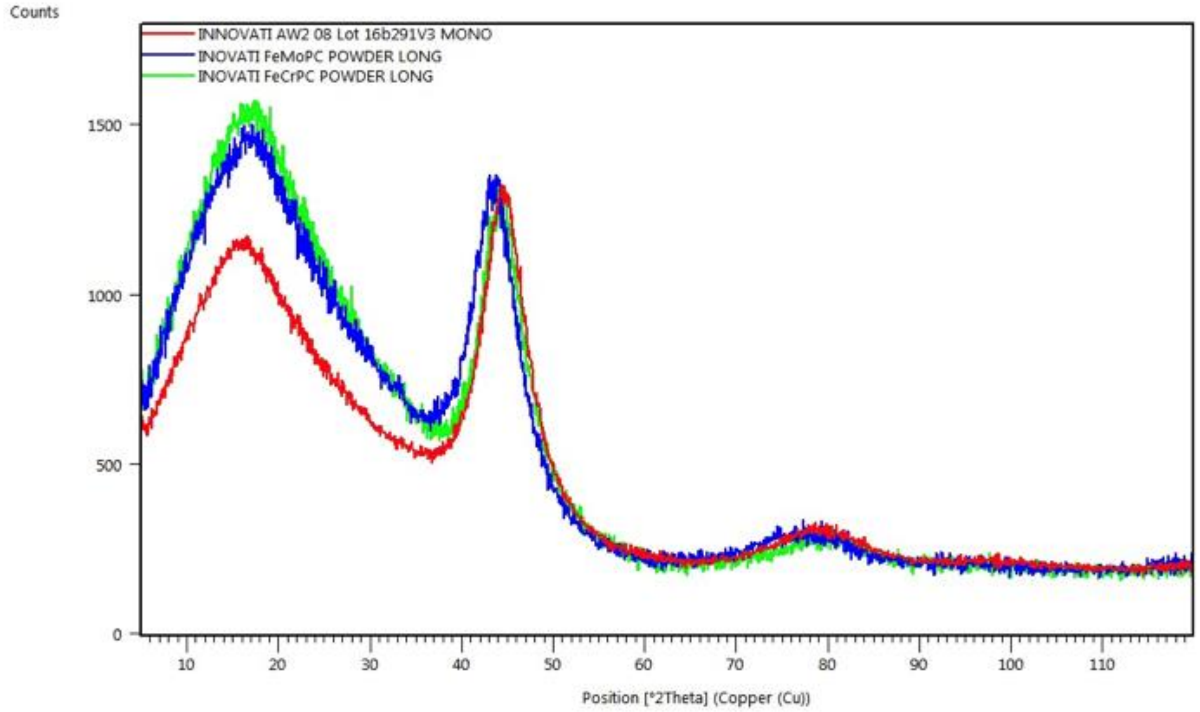

Figure 2

X-ray diffraction of three different amorphous-iron powders. Blue spectrum is that of the amorphous FeMoPC alloy. Other amorphous-iron chromium bearing powders (red and green spectra) were supplied for comparative analysis, but these materials were not relevant candidates for this project. The broadened peaks in the X-ray diffraction spectra at 44º and approximately 77º indicate an amorphous-iron structure with no crystallinity. The broad peak at 16º is a monochromator artifact but did not affect relevant spectral characteristics for identifying iron crystallinity or lack thereof.

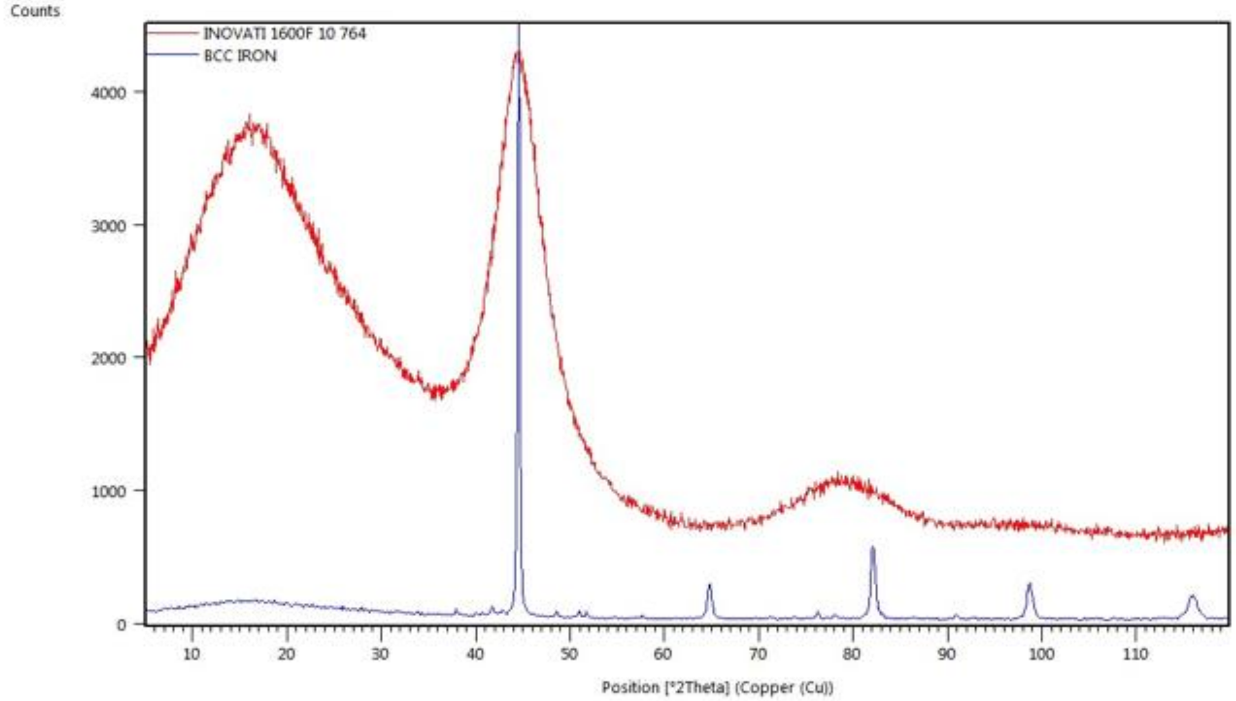

Figure 3

X-ray diffraction of amorphous FeMoPC alloy coating at KM gas temperature of 870 ºC. Figure shows that the amorphous structure is preserved at KM helium set-point gas temperature of 870 ºC (beam deposition temperature of 200ºC). Broadened peaks in the X-ray diffraction spectra at 44º and approximately 77º indicate an amorphous-iron structure with no crystallinity. The broad peak at 16º is a monochromator artifact but did not affect relevant spectral characteristics for identifying iron crystallinity or lack thereof. The amorphous FeMoPC coating results are compared to BCC iron shown by the blue trace.

Development of Environmentally Compliant Amorphous-Iron PRMMC Formulations

Scanning Electron Microscopy (SEM) images of amorphous-iron powder blended with silicon carbide and alumina are shown in Figure 4. The KM depositions indicate high densities of the composite coatings with bright phase indicating the amorphous-iron metal matrix. Concentrations of these second phase constituents can be varied to tailor the wear resistance of these composite coatings.

Scanning Electron Micrographs (SEM) of the amorphous-iron PRMMC coupons were cross-sectioned from coatings on wear bars for ASTM G-77 testing (Figure 4). To compare properties of the amorphous-iron coatings with conventional wear resistant coatings, a FAA certified job shop was enlisted to prepare HVOF (Jetcote 3000) control coupons with WC-17Co coatings. A SEM cross-section of the HVOF WC-17Co is compared to a WC-Co coating deposited with the KM process as shown in Figure 5. The Particle Size Distribution (PSD) for the amorphous-iron PRMMC material (Figure 4) is much coarser compared to the Kinetic Metallization of WC-Co HF-10-10 coatings, however future research work is planned to reduce the PSD of the amorphous-iron PRMMC coating materials for improve wear resistance.

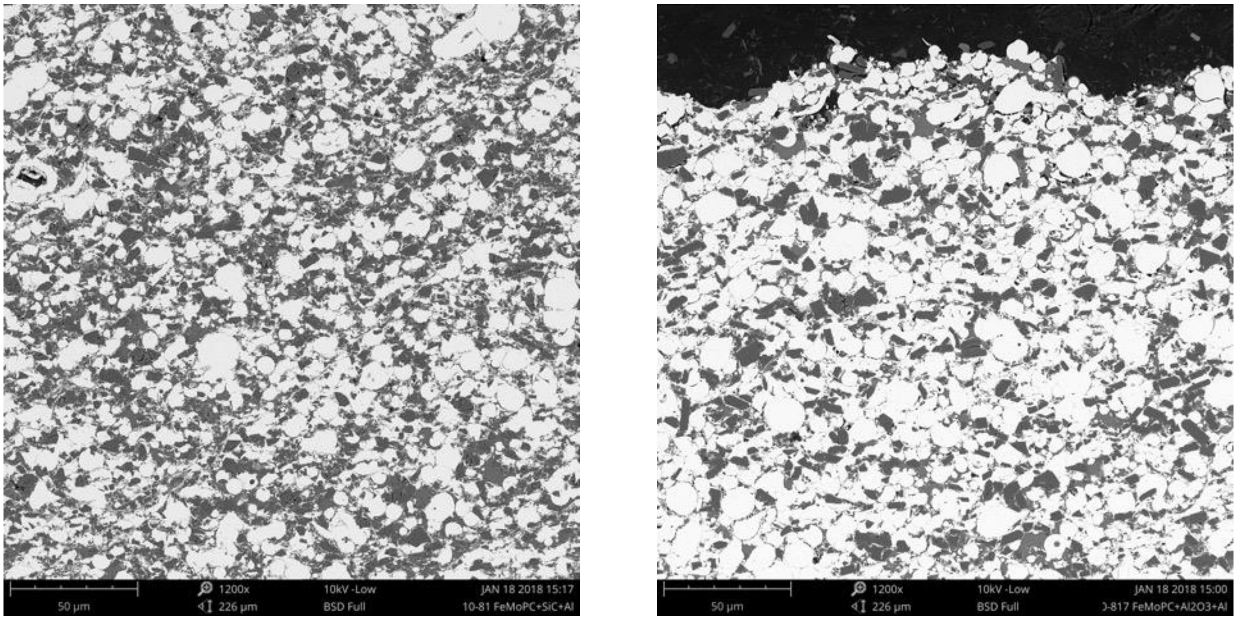

Figure 4

SEM of amorphous-iron/50vol%SiC (left) and amorphous-iron/30vol%Al_2O_3 (right)

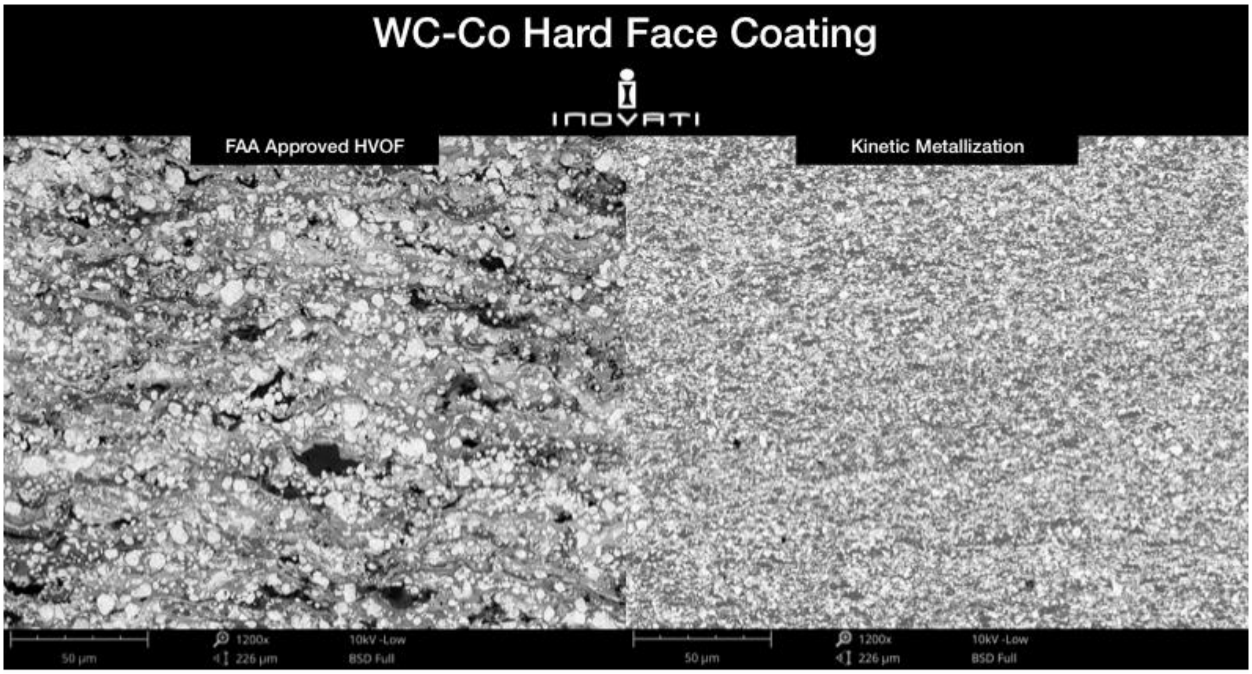

Figure 5

SEM micrograph of HVOF WC-17Co coating (left) compared to Kinetic Metallization of WC-Co HF-10-10 (right). These SEM micrographs are only used to compare a legacy wear resistant coating to amorphous-iron PRMMC coatings shown in Figure 4.

To improve lubricity of amorphous-iron PRMMC coatings molybdenum was blended with amorphous iron as shown in the SEM micrographs of Figure 6 with bright phase indicating the molybdenum particles. The concentration of Mo in the PRMMC can be also tailored to meet the wear requirements and potentially reduced friction of these environmentally compliant coatings.

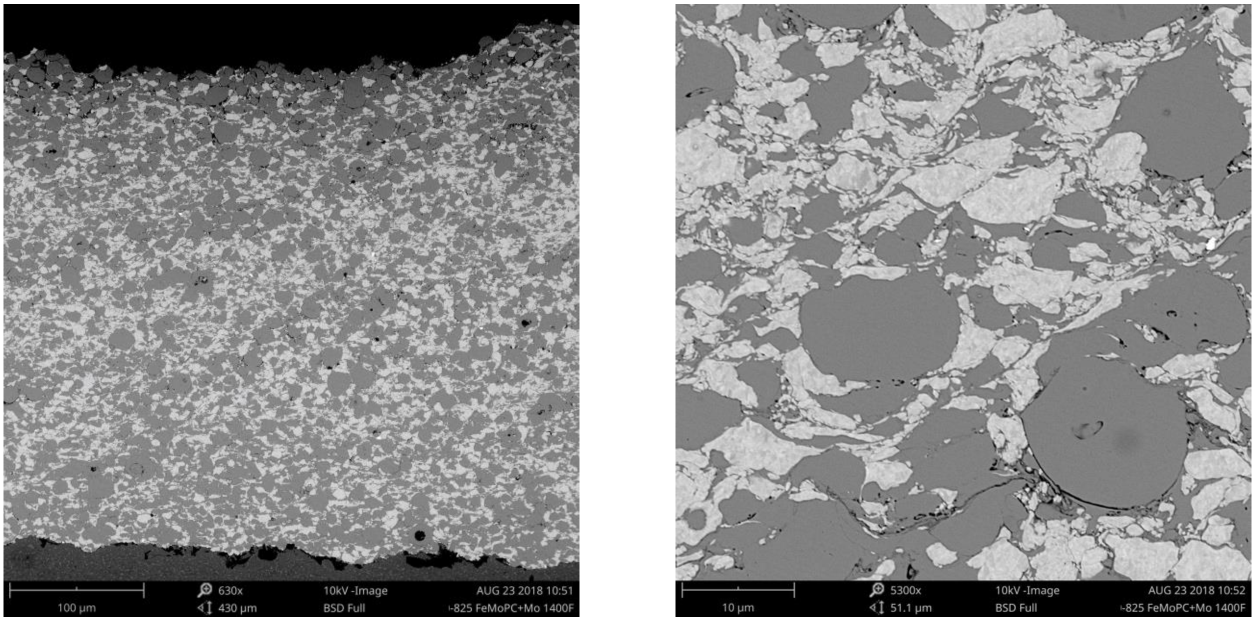

Figure 6

SEM Micrographs of amorphous-iron/50vol%Mo at 640X and 5300X, respectively.

Wear Testing of Amorphous-Iron and Legacy Coatings

Samples of amorphous-iron powders [FeMoPC and FeCrPC] blended with alumina were delivered to The Aerospace Corporation for conducting wear testing per ASTM G99. The results of these tests are displayed in Table 2 and show that the amorphous FeMoPC alloy formulation has the lowest wear rate compared to baseline amorphous FeCrPC material, which contains chromium as a Watch List contaminate (1). More importantly, these wear rates are two orders of magnitude lower for the FeMoPC/30%Al_2O_3 PRMMC coating than legacy HVOF WC-Co coatings reported in the literature at ~$5.0*10^{-08}$ mm^3/mm (6).

The coefficient of friction chart for the FeMoPC/30vol%Al_2O_3 PRMMC coating using a WC-Co ball is displayed in Figure 7.

Table 2. Results of Wear Test (ASTM G99)

Amorphous-Iron PRMMC Coating	Ball Material	Mean Wear Mark Width (mm)	Number of Cycle	Total Sliding Distance (mm)	Wear Volume (mm3)	Wear Rate (mm3/mm)	Mean Wear Rate (mm3/mm)	Steady State Avg Coefficient of Friction
FeCrPC-30%Al_2O_3	WC-Co	0.42	10000	439822	4.8E-04	1.1E-09	1.2E-09	0.51
	WC-Co	0.39	10000	298451	3.6E-04	1.2E-09		
	Sapphire	0.50	10000	392699	9.7E-04	2.5E-09	3.3E-09	0.60
	Sapphire	0.55	10000	345575	1.4E-03	4.1E-09		
FeMoPC-30%Al_2O_3	WC-Co	0.42	10000	439822	4.8E-04	1.1E-09	8.0E-10	0.63
	WC-Co	0.30	10000	298451	1.3E-04	4.0E-10		
	Sapphire	0.35	10000	392699	2.3E-04	6.0E-10	4.0E-10	0.56
	Sapphire	0.27	10000	345575	8.0E-05	2.0E-10		

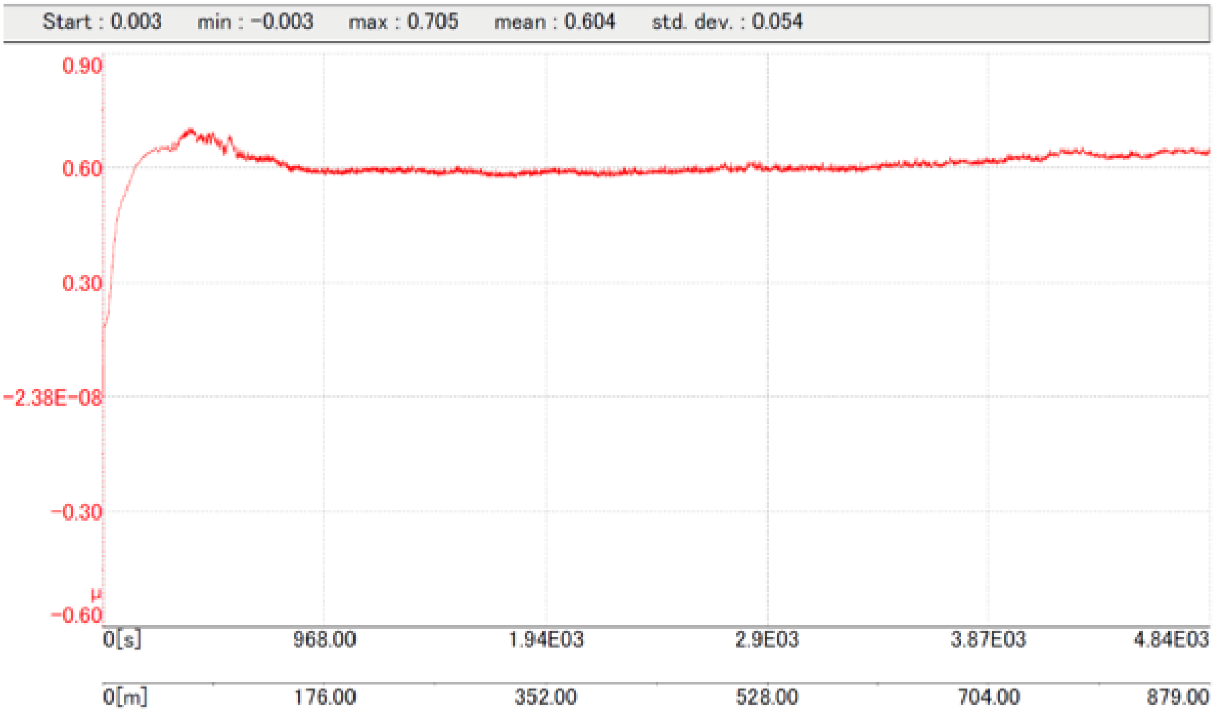

Figure 7

Coefficient of Friction for FeMoPC/30%Al_2O_3 measured with WC-Co Ball

Subsequent to the ASTM G99 pin-on-disc tests, Inovati elected to use the more aggressive ASTM G-77 wear test designed for oil field operations to obtain a better comparison with legacy coatings. The Aerospace Corporation enlisted the test capability of Wear and Friction Resources, LLC in Tomball, TX for performing coated block on 4140 steel ring tests. The ASTM G-77 wear test is a very aggressive sliding wear test, primarily used for ranking hard face coatings using in the mining and oil/gas industries. A more realistic test for aerospace applications may be ASTM G-133 or a reciprocating coated block on flat sheet, but this was not evaluated in the Phase II research.

Coated bars for conducting wear tests per ASTM G-77 included FeMoPC and FeCrPC alloy PRMMC formulations blended with SiC, B_4C, and Al_2O_3 hard phase constituents, and bars coated with legacy materials of EHC and HVOF WC-17Co.These wear test results are summarized in Table 3.

The combined mass loss values listed in Table 3 is important as it shows the mass loss wear rate of the coated block added to the mass loss wear rate of the 4140-steel rotating ring. The mass loss measurements reported in Table 3 represent the average of three separate measurements performed on the coating and block combinations. The standard deviations listed for the combined mass loss values shows significant variation, which indicates that the coatings with larger percent deviations did not perform as well and is consistent with a larger mass loss value. For the legacy coatings, the 4140-steel rings disintegrated more aggressively than the respective coated blocks and gave with larger variations in the measurements. These variations are not well understood but may indicate that the ASTM G-77 test methodology is not suitable for wear applications required to minimize wear rates of both the coating materials and the mating sliding surfaces.

In this case the wear resistance of the amorphous-iron PRMMC coatings are superior to the HVOF WC-17Co legacy coating, and at least comparable to the wear resistance of EHC. This wear performance could be improved by using a finer SiC particle size in the PRMMC coating and increasing the concentration of SiC in excess of 50vol%. The wear resistance of the amorphous-iron FeMoPC/30vol%Al_2O_3 coated block is somewhat lower than the SiC PRMMC, which is to be expected. Again, the Al_2O_3 PRMMC could be improved with higher concentrations of alumina.

Table 3. ASTM G-77 Average Wear Test Results

Coating Material	Wear Surface	Mass Loss (gm)	Combined Mass Loss (gm)
FeMoPC/50vol%SiC	Ring	0.045	0.06 ± 0.01
	Coated Block	0.009	
FeMoPC/50vol%SiC/1%Al	Ring	0.048	0.08 ± 0.04
	Coated Block	0.034	
FeMoPC/50vol%B_4C	Ring	0.070	0.08 ± 0.003
	Coated Block	0.011	
FeMoPC/30vol%Al_2O_3	Ring	0.060	0.09 ± 0.01
	Coated Block	0.032	
HVOF WC-17Co	Ring	0.194	0.20 ± 0.07
	Coated Block	0.004	
EHC	Ring	0.089	0.09 ± 0.05
	Coated Block	0.003	

Corrosion Testing of FeMoPC-Alloy PRMMC Coatings

Corrosion test specimens of amorphous-iron PRMMC were submitted to The Aerospace Corporation Materials Laboratory during the project to perform OCP and Tafel DC Polarization measurement using 3.5% NaCl electrolyte solutions. Figure 8 shows a typical Tafel measurements on the amorphous-iron coating coupons performed with a Princeton Applied Research (PAR) VMP2 Multichannel Potentiostat.

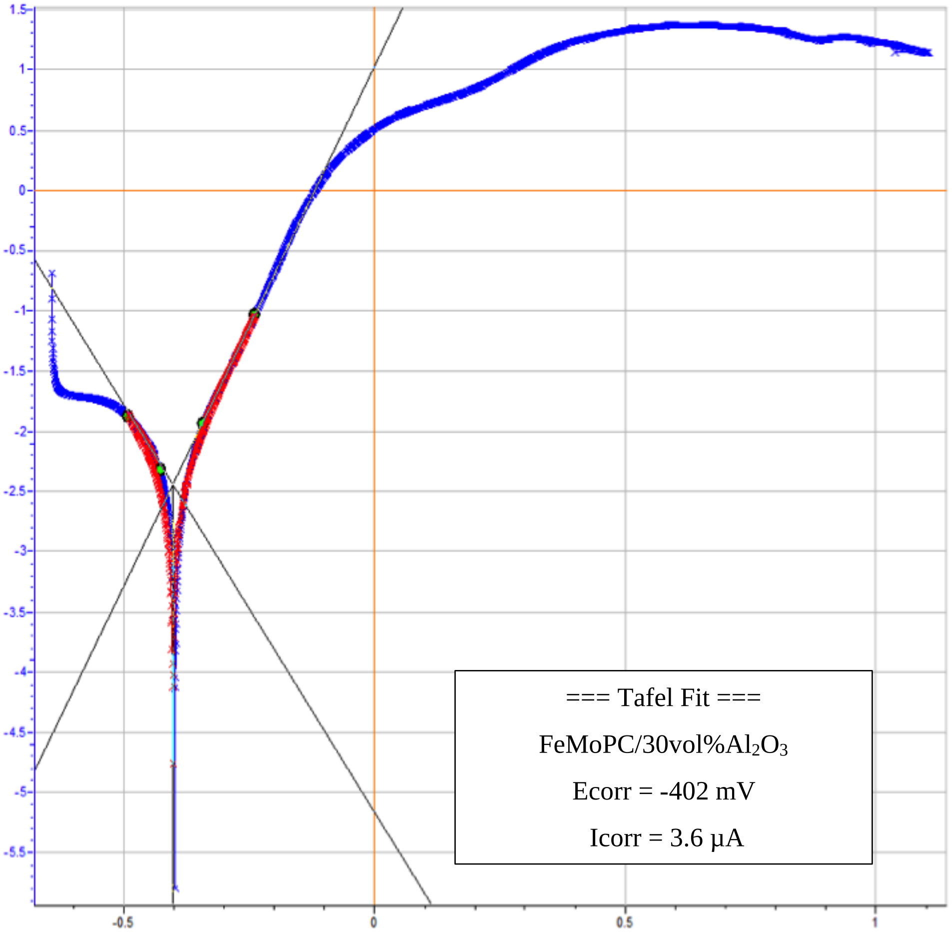

Figure 8.

Tafel DC polarization measurement performed on amorphous-iron PRMMC coatings

These results in terms of corrosion voltages and currents are listed in Table 4. The table also has preliminary estimates of the corrosion rates in terms of milli-inches/yr (mpy), where the coating density was not explicitly measured. It is interesting that the corrosion rates for the amorphous-iron PRMMC in 3.5% NaCl is somewhat lower than reference (7). Although the EHC coating has a very slightly lower Tafel corrosion current, the amorphous-iron coatings with PRMMC reinforcements are superior to HVOF WC-Co coatings on the steel control coupon. The low corrosion current measured for the KM HF-10-10 (WC-Co) coating is exceptional, but not relevant since this material is not considered an environmentally compliant candidate.

Table 4. Tafel DC Polarization Corrosion Test Results in 3.5% NaCl Solution

Coating Material	Tafel Corrosion Voltage Ecorr (mV)	Tafel Corrosion Current Icorr (µA)	Corrosion Rate Estimate (MPY) (milli-inch/year)
FeMoPC/50vol%SiC	-499	5.69	2.57
FeMoPC/30vol%Al_2O_3	-402	3.60	1.88
EHC	-629	4.80	2.45
HVOF WC-Co	-637	8.45	7.51
KM HF-10-10 (WC-Co)	-412	1.00	0.80
Carbon Steel	-543	27.93	14.12

ASTM B-177 Salt Fog Testing of Amorphous-Iron PRMMC Coatings

Additional corrosion testing of the amorphous-iron PRMMC coating was evaluated in comparison to legacy EHC and HVOF WC-Co coatings on 4130 steel substrates. Figure 9 shows the results of a 1000-hr exposure test to salt fog per ASTM B-177. Clearly, the amorphous-iron PRMMC coating is superior to WC-Co coating and competes with the corrosion performance of EHC coatings. Note, the EHC specimens exhibited some corrosion at the edges and the corner for holding the coated specimen in the electrolytic bath. Improved masking of these areas would have precluded red rust generation.

FeMoPC/50vol% SiC

HVOF WC-Co

Electrolytic Hard Chrome

KM HF 10-10 (WC-Co)

Figure 9

ASTM - B117 Salt Fog test results after 1000-hour exposure

Adhesion Testing of FeMoC-Alloy PRMMC Coatings

Adhesion test coupons were prepared by KM deposition onto steel substrates for adhesion strength testing at Inovati using an Elcometer Model F510-20T adhesion gauge with 10-mm diameter dolly buttons bonded to the coatings using high-strength epoxy (Loctite EA 9359.3). By optimizing surface preparation procedures, we have achieved an epoxy adhesion of 8200 ± 770 psi onto 6061Al a (see Table 5). The adhesion of the FeMoPC/50vol%SiC is slightly stronger or equivalent to the EHC, although all coatings failed by epoxy adhesion to the surface of the coating. None of the coatings failed within the coating material, and variations are attributed to the adhesion characteristics of the epoxy to the surface of the coatings.

Table 5. Adhesion Test Results with FeMoPC PRMMC and Legacy Coatings

KM Parameter	Adhesion Strength (psi)	Type of Failure
Epoxy Joint for Elcometer F510-20T 10-mm Stainless Steel Dolly on 6061Al	8200 ± 770	Epoxy Adhesion
FeMoPC/50vol%SiC	6600	Epoxy Adhesion
EHC	5900	Epoxy Adhesion
HVOF WC-17Co	7400	Epoxy Adhesion
HF-10-10 WC-Co	8500	Epoxy Adhesion

Summary

The objective of the F15A-T31 Air Force STTR project was to investigate and develop environmentally compliant inorganic materials for corrosion and wear protection of structural metals on military aircraft and weapon systems. Inovati down selected an amorphous-iron FeMoPC alloy metal matrix for test and evaluation as part of the Phase II STTR project. These ES&H compliant materials in combination with the low temperature spray deposition KM process was demonstrated to provide wear and corrosion resistant coating materials to potentially replace EHC and HVOF WC-Co protective coatings.

The results of the Phase II STTR work has successfully demonstrated that the FeMoPC/50vol%SiC and the FeMoPC/30vol%Al_2O_3 PRMMC formulations have promising wear and corrosion resistance as a benign environmentally compliant material. The wear resistance is superior to the legacy coating of HVOF WC-17Co, and comparable to the EHC coating. The Tafel DC Polarization measurements in 3.5% NaCl electrolyte solutions, indicate that the corrosion resistance is comparable to legacy EHC and superior to HVOF WC-17Co coatings. The adhesion strength of the FeMoPC/50vol%SiC PRMMC formulation was comparable to the legacy EHC. All of the adhesion tests conducted with the Elcometer Model F510-20T using 10-mm dollies failed in the epoxy with no cohesive or adhesive failures within the coatings.

Inovati is pleased to announce that the next phase of qualification testing for deployment of amorphous-iron PRMMC for wear and corrosion protection of military aircraft and weapon systems has been funded in FY2019 by the Office of the Assistant Secretary of the Army for Installations, Energy and Environment under the National Defense Center for Energy and Environment (NDCEE) project with small business matching funds from the US Air Force.

Inovati manufactures KM Systems, which are globally marketed to industry, academia, and government organizations. In 2014 Inovati was awarded a NAVAIR SBIR Phase II.5 contract (8) to deliver and install a KM System at the NAVAIR FRC-SW to repair aircraft components. Under a SBIR Phase III award a second KM System was delivered and installed at the FRC-SW production facility in July 2015. Components routinely repaired by FRC-SW have been returned to the fleet and are now flying.

Inovati plans to follow the same successful commercialization roadmap for deployment of these ES&H compliant coatings for protection of aircraft components and weapon systems following qualification to TRL 8 over the next two years.

Acknowledgments

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