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Screening design of hard metal feedstock powders for supersonic air fuel processing

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ABSTRACT

Replacement of electrolytic hard chromium (EHC) method by Thermal Spray Technology has shown a growing interest the past decades, mainly pioneered by depositing WC-based material by conventional HVOF processes. Lower thermal energy and higher kinetic energy of sprayed particles achieved by newly-developed supersonic air fuel system, so-called HVAF-M3, significantly reduce decarburization, and increase wear and corrosion resistance properties, making HVAF-sprayed coatings attractive both economically and environmentally. In the present work, a first order process map has been intended via a full factorial design of experiments (DoE) to establish relationships between powder feedstock characteristics, such as primary carbides grain size, binder grain size and powder strength, and coating microstructure and mechanical properties. A second order process map was then established to study possible correlations between the deposit microstructural properties and their respective abrasion/erosion wear and corrosion performances.

1. Introduction

In the field of wear, erosion and corrosion applications, recent restrictions in the use of carcinogenic hexavalent form of chrome element has driven the need of replacing electrolytic hard chrome plating (EHC) by other material/process with equivalent tribological properties [1,2]. WC-based powder materials have been proposed as excellent candidates when thermally processed with high kinetic spraying systems [2,3]. Depending on the targeted industrial application, involving dry/wet abrasion/erosion wear, and associated corrosion load case, coating tribological responses to such environment has been largely investigated the past decades through the role of primary carbide grain size (CGS) of WC–Co and WC–CoCr feedstock materials [4–8]. Those studies highlighted the difficulty of minimizing carbide decarburization, being detrimental to the coating abrasion wear resistance, while spraying with air plasma spraying (APS), pulsed plasma spraying (PPS), high velocity oxy-fuel (HVOF) and activated combustion high velocity air fuel (AC-HVAF) technologies [9–14]. One of the latest low-temperature high-kinetic thermal spray processes, named as supersonic air fuel or HVAF-M3 system (UniqueCoat Technology), has since emerged as an interesting and promising alternative method to depose such temperature-sensitive material at even higher flame velocity [3]. Recent work on such HVAF–M3 system revealed the enhancement of coating tribological performances for wear and corrosion protection in the field of construction equipment and off-shore industries [15,16]. Coarsening of the CGS of initial powder feedstock was demonstrated to improve dry abrasion and erosion resistance. However the CGS appeared not to be the only powder features to explain such coating performances, but rather its ratio to the binder Grain Size (BGS), as well as the initial powder strength (PS). In the present work, a first order process map has been designed through screening objectives in order to study relationships between those factors and coating abrasion/erosion wear and corrosion performances, and to highlight the repeatability and reliability of the HVAF–M3 spraying system.

2. Experimental procedure

2.1. Feedstock materials and spray process

Pre-series commercial WC–CoCr powder feedstock materials were manufactured by Fujimi Incorporated (Japan). Ten powders, with a ($-30 + 10$) particle size distribution, were selected while varying their respective primary carbide grain size (CGS), binder grain size (BGS) and powder strength (PS), designated as main factors of a full-factorial design of experiments (Table 1). Domex355 coupons geometry—6 mm thick, were positioned on a rotating carousel, and coated by 15 sequential spray passes. Standard configuration of the

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Table 1
Design matrix of experiments—factor levels.

Powders reference	Commercial reference	Particle size	CGS	BGS	PS	Density
		(μm)	(μm)	(μm)	(MPa)	(g/cm^3)
KB1-1[a]	DTS-W999	−30 + 5	2.0	0.5	300	3.64
KB1-2[a]	DTS-W999	−30 + 5	2.0	0.5	300	3.64
KB2	DTS-W1000	−30 + 5	0.2	0.5	100	3.82
KB3	DTS-W1001	−30 + 5	4.0	0.5	100	3.20
KB4	DTS-W1002	−30 + 5	0.2	2.0	100	3.74
KB5	DTS-W1003	−30 + 5	4.0	2.0	100	3.40
KB6	DTS-W1005	−30 + 5	0.2	0.5	500	5.30
KB7	DTS-W1006	−30 + 5	4.0	0.5	500	5.24
KB8	DTS-W1007	−30 + 5	0.2	2.0	500	4.57
KB9	DTS-W1008	−30 + 5	4.0	2.0	500	4.81
KB10	DTS-W1009	−30 + 5	0.2	2.0	300	4.04

[a] Center points of the full factorial design of experiments.

HVAF–M3 system, i.e. operating the large combustion chamber, long nozzle and short axial powder injector, was utilized for depositing the powder feedstock materials with identical spraying parameters, previously developed by the authors for such feedstock chemistry [15,16]. The HVAF–M3 system was as well utilized for gritblasting the substrates prior to deposition, utilizing mesh 220 (−75 + 45) DURALUM White F220 (Washington Mills). Microstructure features, carbide decarburization, microhardness and abrasive wear and erosion resistance of resulting coatings were evaluated as responses, including respective corrosion resistance. Weighted distributions of carbide grain size and carbide contiguity in resulting coated systems were given a particular interest, in order to correlate coatings microstructure features to wear and corrosion performances.

2.2. Statistical models

In this work, design of experiments (DoE) was used to establish relationships between WC–CoCr feedstock characteristics and HVAF–M3 sprayed coatings properties. DoE is a standard statistical approach conventionally used to study relationships between process parameters and coating properties in thermal spray. The approach is usually a stepwise procedure starting with screening fractional or full factorial designs to response surface designs for optimization purposes. In this study, a full factorial design was selected since this design can gain valuable insight in how the specific powder feedstock characteristics can interact on several responses such as coating microstructure and tribological properties. It should be noted that quantification or discretization of all factors and responses is necessary when using DoE and that the results are dependent on the selected levels of the factors (Table 1). The investigation was performed utilizing the statistical software MODDE ©, MKS Umetrics AB, Sweden. A full factorial screening design comprising 11 experimental runs in total was performed in a random order to increase the model reliability, reproducibility and repeatability, including two center points, also called replicates. Multiple linear regression (MLR) was used to establish the relationships between the factors (CGS, BGS and PS) and the selected responses. Separate MLR models were derived for each response variable, to establish a best fit for the statistical representation of the significance of each factor and their eventual interactions.

2.3. Characterization methods

2.3.1. Coating microstructure

SEM micrographs of respective coatings cross sections were analyzed utilizing a TM3000-Tabletop Microscope (HITACHI). A specially developed image thresholding algorithm utilizing the image analysis Aphelion ® software was applied on 20 SEM pictures (X7000) per samples, in order to identify volume fraction of porosity and carbide/binder phases, as well as primary carbide size and carbide contiguity weighted-distributions respectively.

2.3.2. Micro hardness Vickers

Micro-Vickers hardness measurements were carried out on the polished cross-section of the coatings according to ASTM E384-10 with a Vickers indenter at a load of 100 g, 300 g and 500 g, and a dwell time of 15 s, using a Shimadzu Microhardness Tester. Respective microhardness values were calculated from averaging series of 20 indentations, and respective distributions were analyzed via boxplot representations in order to highlight outliers.

2.3.3. Phase analysis

X-ray diffraction analysis of powder feedstock and coated systems were carried out using an D500 Siemens diffractometer, with Cr source at (35 kV/30 mA) with $\lambda - k\alpha = 0.228$ nm, in order to evaluate the carbide retention index in coated systems following reference [5].

2.3.4. Abrasive wear resistance

Suga Abrasion test was conducted according to ASTM D6037 to investigate the abrasive wear resistance of the coating. The area (30 × 12 mm^2) of coated systems was worn on a SiC (F180) at a reciprocating velocity of 40 DS/min, under a constant load of 30.1 N, and respective volume wear loss was evaluated.

2.3.5. Erosion wear resistance

Blast erosion test was used to investigate erosive wear resistance, where the blast material of alumina F40, a blast angle of 30°, a blast distance of 50 mm and an air pressure of 0.4 MPa were applied.

2.3.6. Corrosion resistance

Neutral salt spray test (NSS) formalized as an ASTM B117 following the ISO 9227 standard was performed to evaluate the relative corrosion resistance of coated materials exposed to a salt spray (pH 6.5–7.2) preconditioned to the operating temperature of 35 ± 2 °C and fogging a 5% salt solution at a condensate collection rate of 1.0 to 2.0 ml/h per 80 m^2. Acetic acid salt spray (ASS) was used for more corrosive environments than the ASTM B117 Standard, according to ISO 16701. A 5% by mass solution of sodium chloride in 95% of ASTM D1193 Type IV water was used and the pH was adjusted with the glacial acetic acid between 3.1 and 3.3. This solution was then atomized to create a fog that has a condensate collection rate of 1.0 to 2.0 ml/h per 80 m^2, maintaining the exposure zone to 35 ± 2 °C. Sprayed samples were grinded or polished to a Ra of 0.1, edges were protected by specially designed tape, and as-polished surface was exposed to a maximal period of 216 h (NSS) and 80 h (ASS) respectively.

3. Part I: material characterization of responses

3.1. Microstructure investigation

Powder feedstock SEM analysis preliminary shows the main difference in particle morphology between fine (0.2 μm), medium (2.0 μm) and coarse (4.0 μm) primary carbides (CGS). The role of binder grain size (BGS) and powder strength (PS) is highlighting the level of carbide distribution, anchoring and embedment (Fig. 1). The different combinations lead to a sensitive decrease of deposition efficiency while combining coarser carbide and finer binder size, an effect amplified with lower powder strength. (Fig. 2). When coarser carbide size increases above the critical splat thickness, hard phases are likely rebounding off splat [8,15], acting as an amplified erosive media processed at supersonic speed. A specially designed Image Analysis procedure utilizing Aphelion software coupled with Matlab routines has been developed to evaluate respectively coating porosity, primary carbide grain size (CGS) and contiguity (CC), distributions (Fig. 2). The analysis was performed on one cross section for each sample over 20