



Wear behavior of Fe₃Al-TiN-TiB₂ HVOF coatings: A comparative study between *in situ* and *ex situ* powder processing routes



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ABSTRACT

In the present study, the tribological properties of High Velocity Oxy-Fuel (HVOF) coatings prepared from Fe₃Al-based composite powders were investigated. The iron aluminide matrix of the composite powders was reinforced with TiN and TiB₂ particles made using two different processing routes: a) an *in situ* method where fine ceramic particles were formed in the matrix by the reaction between Ti and BN, and b) an *ex situ* method where preformed coarse TiN and TiB₂ particles were added to the matrix. The tribomechanical performance of the coatings was assessed using indentations and pin-on-disc wear tests. Compared to *ex situ* samples, the Fe₃Al-based coatings strengthened with *in situ* ceramic particles exhibit higher microhardness and wear resistance regardless of the sliding velocity. The presence of voids, cracks and scratches/grooves in the wear track of the *in situ* coatings and the coating material transferred to the corresponding counterpart suggest that coatings with fine reinforcing particles fail predominantly *via* delamination and adhesive wear mechanisms. In the case of the *ex situ* coatings, the presence of a significant amount of hard ceramic particles within the wear track indicates that abrasive wear plays a dominant role in the degradation mechanism. Oxidation wear also contributed to material removal at high sliding velocity since transfer materials inside the wear track contain a high oxygen content compared to the unworn region regardless of the coating type.

1. Introduction

Metal matrix composites (MMC) have been widely recognized as materials of interest for wear protection due to their special microstructure and excellent mechanical properties [1]. The combination of the metallic matrix and the secondary phases give rise to enhanced properties of the resulting material [2]. MMC are promising potential candidates for replacing conventional bulk steels or hard chrome in many areas of applications such as aerospace, automotive, hydroelectricity or manufacturing industries [3].

Among potential matrix candidates, special attention has been given to low-cost and low-density materials such as iron aluminides. These materials are environmentally friendly and demonstrate good corrosion and oxidation resistance at high temperature [4]. However, their poor mechanical and tribological performances have restricted their use in the fields where wear-resistance is particularly important. It has been shown previously, that the modification of microstructure of iron aluminides leads to improvement of mechanical properties [5,6].

Iron aluminides are thermodynamically compatible with several borides, carbides, nitrides and oxides [7,8]. The iron aluminide (Fe₃Al) MMC including ceramic inclusions exhibit unique microstructure and excellent mechanical properties such as high hardness, high strength and high fracture toughness [9,10].

High-Energy Ball Milling (HEBM) is widely used to manufacture MMC materials [11,12]. In this process, powder particles are plastically deformed, fragmented and repeatedly cold-welded in order to form materials with fine and homogeneous microstructure in which secondary phases are uniformly dispersed. The technique allows one to manufacture composites with a relatively defect-free matrix/reinforcement interfaces [11].

Thermal spray techniques are often used for producing thick coatings that exhibit interesting wear resistance characteristics [13]. In many applications subjected to hostile environmental conditions, thick protective coatings are preferred compared to thin films prepared using PVD or CVD for extended lifetime protection. Among the fabrication techniques, High Velocity Oxy-Fuel (HVOF) deposition can be used to obtain dense protective coatings on different types of

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substrates [14]. In the high-pressure HVOF process, such as the one used in this work, oxygen and kerosene are mixed and ignited to form a hot and high-pressure gas into which powder particles are injected. The particles are heated and expelled at about three times the speed of sound toward the substrate, and coatings are formed from the successive buildup of melted or semi-melted splats [14].

Ball milling followed by subsequent heat treatment has been used to prepare Fe₃Al-based composite powders reinforced with *in situ* formed ceramic particles. Specifically, it has been demonstrated that the microstructure and the mechanical properties of the hybrid Fe₃Al/(AlN, Fe₂B) powders could be preserved during the HVOF deposition [8]. Improved tribomechanical properties of these coatings were attributed to the fine dispersion of *in situ* precipitated AlN and Fe₂B particles within the Fe₃Al matrix. Using an *ex situ* approach, Peng et al. [15] have shown that the room temperature toughness and flexural strength of the ball milled then hot-pressed Fe₃Al-based composite were improved by the addition of preformed ZrO₂ particles. The low thermal expansion mismatch between the Fe₃Al matrix and the coarse ZrO₂ particles, and the stress-induced transformation of ZrO₂ particles were reported to be particularly beneficial [15].

Both *in situ* and *ex situ* approaches lead to enhanced mechanical and tribological properties of Fe₃Al-based composites. However, little has been done to sufficiently identify which fabrication route can effectively enhance the tribomechanical properties of such materials.

In the present work, we compare the microstructure and the mechanical properties of the Fe₃Al-based composite coatings prepared using powders with *in situ* strengthened and *ex situ* preformed ceramic particles namely titanium nitride and titanium diboride. The wear behavior of the HVOF coatings are compared and discussed with respect to different sliding test conditions.

2. Materials and experimental methods

2.1. Powder preparation

In the *in situ* approach, the initial powder mixture consisted of 70 wt% of pre-alloyed iron aluminide (Fe₃Al: 97.5% pure with ~2% of Cr from Ametek), 10 wt% of boron nitride (BN: 98% pure from Lower Friction) and 20 wt% of titanium (Ti: 99.4% pure from Alfa Aesar). The mean particle size of BN and Ti was 5 and 150 μm, respectively. This powder mixture was milled for 10 h using the HEBM technique, and is labeled M30 in the following text. More details on the fabrication of the composite powders are described in [16].

Following the initial HEBM, the as-milled M30 powder; was heat-treated in a furnace at 1000 °C for 2 h under vacuum (10⁻⁶ mbar) to form TiN and TiB₂ particles in the matrix; it is labeled AM30 in the following text below. The heating rate was 7 °C/min, and a cooling rate of 4 °C/min was used from 1000 °C to 500 °C. Thereafter, the heat-treated powder was re-milled at high energy (1000 rpm) for 5 h; it is labeled In-M30. This fabrication approach is referred to as the *in situ* synthesis route because the hard ceramics (TiN and TiB₂) are formed during the powder fabrication process. The re-milling time of 5 h was chosen in order to obtain a reasonable amount of powder (after sieving) with an average particle size between 20 and 50 μm which is adequate for the HVOF deposition.

During the *ex situ* approach, X-ray diffractograms of the AM30 samples were used to evaluate the amount of TiN and TiB₂ materials in the composite powder. An equivalent amount of preformed titanium nitride (TiN: 99.8% pure) and titanium diboride (TiB₂: 99.5% pure) particles from Alfa Aesar was ball milled at high energy (1000 rpm) with the Fe₃Al powder. The initial size of the TiN and TiB₂ particles was < 10 μm and 44 μm, respectively. The powder mixture is labeled Ex-M30 in the following text. To monitor the evolution of the Ex-M30 particle size as a function of milling time, a small amount of powder

Table 1
Experimental conditions for HVOF deposition.

Spray parameters	Value
Standoff distance (cm)	38
Nozzle length (cm)	15
Nozzle diameter (mm)	11
Chamber pressure (kPa)	700
Oxygen flow rate (slpm)	890
Kerosene flow rate (l/h)	23.5
Equivalent oxygen/kerosene ratio	1.1

(~1g) was collected every hour and analyzed using the Horiba LA 900 Particle Size Analyzer. The HEBM of the Ex-M30 powder was stopped (after 4 h) when the average particle size of the as-milled powder was identical to that of the In-M30 powder (*i.e.* 30–40 μm).

2.2. Coating preparation

The HVOF coatings were fabricated using a Praxair J-P 8000 HP-HVOF system mounted on an ABB robot arm and controlled by a computer using the deposition conditions described in Table 1. The composite powders were deposited onto flat 304 stainless steel substrates previously sandblasted using 60 grits alumina particles and cleaned with methanol. The HVOF gun was scanned at a speed of 10 cm/s for a total of ten passes. The gun was displaced by 10 mm after each pass. The coatings are labeled with a “C” in front of the names of the corresponding powder. For example, In-CM30 is referred to the HVOF coating prepared from the In-M30 composite powder. More details on the HVOF coating process can be found in [8].

2.3. Microstructural analysis

X-ray diffractograms of composite powders and of their corresponding HVOF coatings were obtained using a Brüker D8 DISCOVER diffractometer in a $\theta/2\theta$ configuration. The diffractometer, operated at 40 kV and 40 mA, was equipped with a copper source (λ : 1.5406 Å), a Gobel mirror, a divergent slit of 0.6 mm and a LynxEye detector set with soller slit (2.5°). The diffractograms were acquired from 20° to 90° in 2 θ , with a step size of 0.051° and a time per step of 0.5 s. The phase quantification was obtained by Rietveld refinement using Topas 4.2 software (Brüker AXS).

Surface morphology and chemical composition of the powders and the HVOF coatings were obtained on polished cross-sectional surfaces using a 7200 JEOL scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS Inca X-max 80 Oxford) operating at 15 kV.

2.4. Mechanical characterization

The depth-sensing indentation measurements on the surface and on the cross-section of the HVOF coatings were performed using a Triboindenter (TI950 Hysitron) equipped with a Berkovich diamond tip calibrated with a quartz standard. Matrices of 20×20 points evenly spaced by 5 μm were used with a trapezoidal load function: the load was linearly increased up to a maximum of 2 mN for 5 s, kept constant for 2 s, and linearly decreased for 5 s. The maximum penetration depth was about 150 nm. Measurements were obtained from two distinct and randomly chosen regions of the polished surfaces of the specimen (because of the heterogeneity of the coating), and a minimum of 300 indents was considered for statistical analysis. The nanoindentation technique was chosen in order to measure the mechanical properties of

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