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# Oxidation behaviour of CoNiCrAlY bond coats produced by plasma, HVOF and cold gas dynamic spraying

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#### ABSTRACT

This paper examines and compares the microstructure and oxidation behaviour of CoNiCrAlY coatings manufactured by the APS, HVOF and CGDS deposition techniques. The coatings microstructural features were characterized by means of SEM and XRD analyses. Coating samples were then subjected to isothermal heat treatments at 1000 °C. Oxide growth rates were obtained from a series of mass gain measurements while oxide scale compositions were determined from SEM, XRD and EDS analyses. Results obtained in this study show that the as-sprayed CGDS and HVOF coatings exhibit similar microstructures, whereas the APS coating features high levels of visible defects and oxide content. Oxidation experiments revealed low oxide growth rates for both the CGDS and HVOF coatings as a result of low porosity and oxide content. The oxide scale on the CGDS and HVOF coating after 100 h of oxidation were composed mainly of alumina without the presence of detrimental fast-growing mixed oxides. The presence of Cr<sub>2</sub>O<sub>3</sub> and dispersed NiO was however also observed for the HVOF coating. As expected, the APS coating featured the onset of mixed oxides in the early stages of oxidation. From these results, it appears that potential improvements to the bond coat oxidation behaviour can be achieved using low-temperature processing methods such as CGDS.

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#### 1. Introduction

Increasing demands for higher gas turbine engine performance have led to the development of thermal barrier coating (TBC) systems applied to the engine's hot-components. TBCs typically consist of an underlying MCrAlY bond coat with an yttria partially stabilized zirconia ceramic top coat. The latter acts as a thermal insulator whereas the bond coat promotes bonding between the part and the top coat and provides protection against oxidation and hot-corrosion. The development of a "prime-reliant" TBC system, where life expectancy of the coating system does not govern that of the turbine blade, has yet to be achieved due to premature failure of the ceramic top coat. Upon exposure to high-temperature gases, a thin oxide scale, the thermally grown oxide (TGO), forms at the bond coat/top coat interface and continues to grow in thickness during thermal cycling [1]. Rapid and uneven growth of the TGO leads to localized stress concentrations where cracks can nucleate and initiate the failure dynamics. A dense and uniform  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> TGO scale is desirable [2] as it has a slow growth rate and inhibits further oxidation due to its low diffusivity [3]. Other oxides that may form in the TGO include NiO or mixed spinel-type oxides which have high growth rates and thus are

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detrimental as they form protrusions in the TGO that initiate failure mechanisms of the top coat [1].

Bond coats are typically manufactured using air-plasma spraying (APS), low-pressure plasma spraying (LPPS) and high-velocity oxygen-fuel spraying (HVOF) [4-6]. The predominant drawback to these techniques is that their inherent high temperatures inevitably lead to changes in the coating microstructure, namely oxide inclusions. Shibata et al. [4] deposited MCrAlY bond coats and reported values for coating oxide content of 0.16. 0.94 and 1.8 wt.% for LPPS, HVOF and APS respectively. Coatings with higher initial oxide content were shown to experience increased oxide growth rates when subjected to isothermal oxidation testing, thereby promoting the onset of premature spallation of the top coat. Tang et al. [7] deposited MCrAlY coatings by HVOF and demonstrated that inprocess surface oxidation is detrimental to the TGO growth mechanism as it promotes the onset of undesirable fast-growing nonalumina oxides that form protrusions and accelerate the TBC failure mechanisms.

Cold gas dynamic spraying (CGDS) uses kinetic energy rather than thermal energy to produce coatings [8]. In this process, fine powder particles are accelerated in a supersonic flow and undergo severe plastic deformation upon impacting the substrate to form a coating. This technique has been used to produce coatings from various materials with different types of microstructures including conventional, nanocrystalline, amorphous and metastable structures [8–20]. CGDS operates at much lower temperatures than thermal spray

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processes and consequently its coatings exhibit no grain growth [11]. In addition, it uses inert gases which hinder in-process particle surface oxidation [9,10]. These advantages make CGDS an interesting alternative for the deposition of bond coats.

CoNiCrAlY coatings have been manufactured by CGDS to verify their feasibility and investigate whether microstructural changes occurred throughout the deposition process [21]. The present study investigates and compares the oxidation behaviour of CoNiCrAlY coatings manufactured by CGDS, HVOF and APS. These coatings were subjected to isothermal oxidation testing and characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS). Mass gain measurements of the oxidized coating samples were carried out to assess oxide growth rates.

#### 2. Experimental procedure

#### 2.1. Feedstock material

The feedstock material considered in this study is a commercially available CoNiCrAlY alloy (Sulzer Metco) with a nominal composition of Co-32Ni-21Cr-8Al-0.5Y (wt.%). This gas atomized powder has a spherical morphology and is available in two different particle size ranges: AMDRY 9951 particles are finer (5-37  $\mu$ m dia.), whereas AMDRY 9954 is generally coarser (11-62  $\mu$ m dia.). The AMDRY 9951 feedstock was used to manufacture the CGDS coatings due to aerodynamic considerations [9]. Conversely, coatings were manufactured by thermal spray techniques using the AMDRY 9954 feedstock according to the powder manufacturer's recommendations.

#### 2.2. Deposition techniques and equipment

#### 2.2.1. CGDS coatings

The CGDS CoNiCrAlY coatings were manufactured using the cold spray system developed at the University of Ottawa Cold Spray Laboratory. Details of this CGDS system can be found elsewhere [21]. For the present study, helium was used as the main propellant gas with a nozzle inlet gas temperature and pressure of 550 °C and 2.0 MPa respectively. In comparison, current commercial CGDS systems can operate with helium at temperatures and pressures of up to 800 °C and 4.0 MPa respectively [22]. This suggests that the CGDS process has not been stretched to its limit in the present study and that further coating optimization is likely possible. Coatings were manufactured onto grit blasted aluminum 6061 substrates and were then removed from the substrates prior to analysis and oxidation testing, as detailed in a subsequent section.

#### 2.2.2. Thermal spray coatings

Two common thermal spray techniques were considered in this study for the deposition of CoNiCrAlY coatings. Firstly, coatings were manufactured by APS using a SG100 plasma torch (Praxair) with Ar and He as the primary and secondary gases respectively. Secondly, coatings were deposited by HVOF using a DJ2600 torch (Sulzer Metco) with  $H_2$  and  $O_2$  as combustion gases. Deposition of the coatings by both thermal spray techniques was carried out at Vac Aero International Inc. in Boucherville, Quebec, Canada. The specific deposition parameters for both the HVOF and APS coatings are presented in Table 1.

#### 2.3. As-sprayed coating characterization

The coatings produced were investigated qualitatively and quantitatively in order to assess their properties in the as-sprayed condition using a SEM (Zeiss, model Evo10). Prior to microscope investigation, the coating samples were sectioned and polished using standard metallographic techniques. SEM images were used to

#### Table 1

Deposition parameters for the thermal sprayed CoNiCrAlY coatings.

HVOF	
Spray gun	DJ-2600
Oxygen pressure (psi)	170
Hydrogen pressure (psi)	142
Air pressure (psi)	100
Stand-off distance (in.)	11
Powder feedrate (lbs/h)	7.0
APS	
Spray gun	SG-100
Current (A)	800
Voltage (V)	39
Primary gas Ar pressure (psi)	50
Secondary gas He pressure (psi)	110
Stand-off distance (in.)	5
Powder feedrate (lbs/h)	3.0

identify visible defects such as cracks and undeformed particles. The coating phase composition was also investigated by means of XRD analysis using a Philips X-Pert model PW 1830 generator diffractometer with CuK $\alpha$  radiation. Detailed scans with step size of 0.01° and step time of 2s were conducted with 2 $\theta$  values ranging from 20° to 85°. EDS analysis was also carried out to evaluate the as-sprayed coatings oxide content. Finally, an optical microscope equipped with the Clemex Vision Lite image analysis software was used to evaluate coating porosity.

#### 2.4. Oxidation experiments

The samples used for the oxidation experiments consisted of freestanding  $15 \text{ mm} \times 15 \text{ mm} \times 0.5 \text{ mm}$  CoNiCrAlY flakes prepared from the coating samples manufactured by CGDS, HVOF and APS. Coatings were initially cut to size and then sectioned from the substrates. The sectioned surface was then polished using 320-grit SiC paper in order to remove any remaining substrate material and obtain free-standing coating samples. These samples were then subjected to isothermal heat treatments at 1000 °C in static atmospheric air. Samples were placed in the pre-heated furnace and then allowed to soak for durations of 5, 25, 50 and 100 h respectively. Following each heat treatment, the samples were removed from the furnace and allowed to cool to room temperature. Each specimen was weighed before and after the oxidation experiments using a digital weighing scale (Sartorius Extend – model ED124S). The mass gain measurements are reported as the increase in mass normalized per unit area for each sample. Following each heat treatment, samples were also subjected to XRD analysis to investigate the oxide composition and phase content as well as the overall oxide growth dynamics. Finally, SEM images of the samples' oxidized surface were used to examine the oxide morphology while cross-section images were taken in order to assess the thickness and growth of the oxide scale and depletion zone.

#### 3. Results and discussion

#### 3.1. As-deposited coating characterization

Fig. 1 shows SEM images of the as-deposited CoNiCrAlY coatings manufactured by (a–b) CGDS, (c–d) HVOF and (e–f) APS at  $100 \times$  and  $400 \times$  magnifications (Fig. 1b(ii) and d(ii) were taken at  $2500 \times$  magnifications). The coating porosity analysis and oxide content measurement results for each deposition technique as well as for the original feedstock powder are summarized in Table 2.

The CGDS coating (Fig. 1a–b) exhibits limited porosity and crack content. Porosity measurements for the CGDS coating resulted in an average porosity of  $1.0 \pm 0.3$ %. The pores found within the CGDS coating are typically small in size with an average equivalent diameter of  $1.5 \,\mu$ m. This is attributed to insufficient localized plastic deformation of

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