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Bond Coat Engineering Influence on the Evolution of the Microstructure, Bond Strength and Failure of TBCs Subjected to Thermal Cycling

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Different types of thermal spray systems, including HVOF (JP5000 and DJ2600-hybrid), APS (F4-MB and Axial III) and LPPS (Sulzer Metco system) were employed to spray CoNiCrAlY bond coats onto Inconel 625 substrates. The chemical composition of the bond coat powder was the same in all cases; however, the particle size distribution of the powder employed with each torch was that specifically recommended for the torch. For optimization purposes, these bond coats were screened based on initial evaluations of roughness, porosity, residual stress, relative oxidation, and isothermal TGO growth. A single type of standard YSZ top coat was deposited via APS (F4MB) on all the optimized bond coats. The TBCs were thermally cycled by employing a furnace cycle test (FCT) (1080°C-1 h – followed by forced air cooling). Samples were submitted to 10, 100, 400, and 1400 cycles as well as being cycled to failure. The behaviour of the microstructures, bond strength values (ASTM 633) and the TGO evolution of these TBCs were investigated for the as-sprayed and thermally cycled samples. The results showed that the bond strength values decrease significantly only when the TBC is about to fail (top coat spall off).

1 Introduction

Thermal barrier coatings (TBCs) are applied on the parts of the hot zones of gas turbines engines for aerospace and energy generation applications. These parts include the combustion chambers, transition ducts, vanes, turbine blades and afterburners [1, 2]. A thermally sprayed TBC system generally consists of a two-layered architecture, i.e., a ceramic top coat (e.g., ZrO_2 -7-8wt% Y_2O_3 , a.k.a. YSZ) and an MCrAlY bond coat (BC) (M = Ni and/or Co). This architecture is deposited on a Ni-based metallic superalloy substrate (e.g., Inconel 625). For turbine engines, the typical bond coat and YSZ top coat thickness ranges are 125-250 μm and 250-400 μm , respectively. Air plasma spray (APS) torches are the standard deposition systems for the YSZ top coat, whereas, different types of thermal spray torches can be employed to deposit the MCrAlY bond coat; which also includes APS, in addition to low pressure plasma spray (LPPS) and high velocity oxygen-fuel (HVOF). It is important to highlight that even within the same torch category, there can be torch design differences that translate into different torch performance levels and thereby coating performance, e.g., radial or axial powder injection for APS or fuel type for HVOF (e.g., kerosene or H_2).

This work had the major objective to investigate the effect of the CoNiCrAlY bond coat deposition torch on the TBC performance via furnace cycle testing (FCT).

2 Experimental Procedure

2.1 Substrate and bond coat

All TBCs in this study were deposited on Inconel 625 pucks (2.54 cm diameter x 1.5 cm height). Inconel 625 is a typical material employed in the combustion chambers, transition ducts and afterburners of gas turbines. The substrates were grit-blasted with alumina before bond coat deposition, which is a typical preparation step.

2.2 CoNiCrAlY bond coat

The nominal CoNiCrAlY bond coat composition was the same for all samples employed in this study (38.5wt%Co, 32wt%Ni, 21wt%Cr, 8wt%Al, 0.5wt%Al). The particle size distribution employed for each torch was recommended by the torch manufacturer. Table 1 summarizes the main characteristics of the CoNiCrAlY powders. Table 2 identifies the combinations of torches and CoNiCrAlY powders employed in this study. The bond coat thickness range was 150-200 μm .

Table 1. CoNiCrAlY bond coat powder summary.

CoNiCrAlY powder	Manufacturer	Nominal particle size range
CO-210-24	Praxair (Indianapolis, IN, USA)	-45 / +20 μm
Diamalloy 4007	Sulzer Metco (Westbury, NY, USA)	-45 / +15 μm
Amdry 9951	Sulzer Metco (Westbury, NY, USA)	-38 / +5 μm

Table 2. CoNiCrAlY deposition torch summary.

Technique / BC Torch	Manufacturer	CoNiCrAlY powder
<u>APS</u> F4-MB	Sulzer Metco (Westbury, NY, USA)	Amdry 9951
<u>APS</u> Axial III	Northwest Mettech (North Vancouver, BC, Canada)	CO-210-24
<u>LPPS</u> LPPS	Sulzer Metco (Westbury, NY, USA)	Amdry 9951
<u>HVOF</u> DJ2600-hybrid	Sulzer Metco (Westbury, NY, USA)	Diamalloy 4007
<u>HVOF</u> JP 5000	Praxair (Indianapolis, IN, USA)	CO-210-24

2.3 YSZ top coat

Just one type of YSZ powder was employed in this study (Metco 204NS, Sulzer Metco, Westbury, NY, USA). Nominal particle size distribution range: -125 / +11 μm .

This YSZ powder was sprayed using just one single APS torch (F4MB, Sulzer Metco, Westbury, NY, USA) for all bond coats. The spray set recommended by the torch manufacturer was employed to deposit all YSZ top coats. The YSZ top coat thickness range was 225–275 μm and 350–400 μm for the TBCs subjected to isothermal treatment and FCT, respectively.

2.4 Residual stress and roughness

The residual stress of the bond coats was qualitatively evaluated via the deflection of the Almen N strips. The roughness (R_a) values of the bond coats were measured by using a stylus profilometer.

2.5 Microstructural characterization

In order to better preserve the microstructure, the TBCs were vacuum impregnated in epoxy resin prior to cutting with a diamond saw for cross-section evaluation. The cut sections were re-mounted in epoxy resin and polished according to standard metallographic techniques.

The TBCs were evaluated via a scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectroscopy (EDX) module. The porosity measurements were performed by using image analysis at a magnification of 500 X.

2.6 Isothermal oxidation

For the screening process before FCT, the TBCs were exposed to an isothermal heat treatment in a box furnace at 930°C for up to 4000 h in air to evaluate thermally grown oxide (TGO) formation via SEM analysis.

2.7 FCT study

Prior to FCT, the selected TBCs were heat-treated under controlled conditions in vacuum (pressure <0.07 Pa) at 1080°C for 4 h. During FCT, the TBCs were plunged into a pre-heated furnace (in air) at 1080°C. The dwell time in the furnace was 1 h. The cooling was performed by removing the samples from the furnace to a cold zone with forced air cooling during 15 min to reach room temperature (RT).

Debonding/delamination of $\geq 20\%$ of the TBC surface area was considered the criteria of failure.

Macroscopic lifting (but coating still attached to the substrate) was also included in this criteria.

3 Results and Discussion

3.1 Primary screening

The bond coat powders were sprayed under a wide range of spray parameters in order to create coatings with different properties. Typically, between 3 and 6 bond coats were produced for initial screening. The particle temperature (T) and velocity (V) values were measured for some of the bond coats produced for

this study. A summary of the average T and V values (3000 particles) measured can be found in Figs. 1–2.

Fig. 1. Average T values for some of the bond coats produced for this study.

Fig. 2. Average V values for some of the bond coats produced for this study.

The graphs show that the bond coat powders were sprayed under a wide range of T & V average values from $\sim 1250^{\circ}\text{C}$ to $\sim 2400^{\circ}\text{C}$ and ~ 250 m/s to ~ 700 m/s, respectively. It has to be highlighted that not all bond coats produced for this study were included in Figs. 1–2, therefore, the range of particle T & V values may be wider than these currently reported.

The porosity values for all the BCs tested in the initial screening are found in Fig. 3. The porosity values exhibit a range from $\sim 0.5\%$ to $\sim 10\%$. As expected, LPPS BCs tend to exhibit the lowest values, whereas, APS BCs tend to exhibit the highest ones. The residual stress behaviour of some of the BCs produced for this study was evaluated via Almen N strips. Their deflections are shown in Fig. 4. The deflection values ranged from about -300 μm to about $+300$ μm , where negative values represent compressive residual stress and positive values represent tensile residual stress.

For the APS BCs evaluated, all residual stress values were tensile. All BCs sprayed using a kerosene-based HVOF torch (JP5000) exhibited compressive stresses. For a H_2 -based HVOF torch (DJ2600-hybrid), the resulting stresses were tensile, compressive or neutral depending on the particle T & V values.

The roughness (R_a) values for all the BCs tested in the initial screening are found in Fig. 5. BCs with roughness values varying from $\sim 3 \mu\text{m}$ to $\sim 8 \mu\text{m}$ were produced for this study.

Fig. 3. Porosity values for all the as-sprayed BCs produced in this study.

Fig. 4. Residual stress values for some of the BCs produced in this study.

It has to be highlighted that in addition to exhibiting different average porosity and roughness values, their standard deviation values also changed depending on the torch and also when the BC was sprayed using the same torch. Therefore, important and different microstructural/topological features were likely to be produced.

3.2 Secondary screening

Based on the data gathered in Figs. 1-5, the following BCs were selected for a secondary screening based on the isothermal treatment at 930°C in air for up to 4000 h (Table 3). The criteria for selection were based on including (i) 3 BCs for each technique/torch combination, (ii) a ranking of qualitative relative oxidation of the cross-section of the as-sprayed BC (evaluated via EDX – mapping at 500X mag.), in addition to a range of different values of (iii) porosity, (iv) Almen N deflection and (v) roughness. It is important to highlight that relative oxidation meant a relative ranking related to each group of technique/torch combination. Therefore, a “low relative

oxidation” in HVOF and LPPS BCs does not necessarily mean that both coatings exhibited a similar weight% or atomic% of oxygen content. It is important to point out that for all BCs listed in Table 3, a single type of APS YSZ top coat was applied on all samples for secondary screening.

Fig. 5. Roughness values for all the as-sprayed BCs produced in this study.

Table 3. BCs selected for secondary screening.

Technique / BC Torch	BC	Qualitative relative oxidation
APS F4MB	12	High
	15	Medium
	16	Low
APS Axial III	A	Low
	C	High
	17	Medium
LPPS SM LPPS	J	Low
	K	Low
	L	Low
HVOF DJ2600-hybrid	2	Medium
	4	Low
	5	High
HVOF JP 5000	X	Low
	7	Low
	10	Low

The relationship between TGO growth and as-sprayed BC roughness values for the TBCs isothermally heat-treated at 930°C for 500 h can be found in Fig. 6. It was observed that for all processes (except LPPS) the roughness values varied from ~ 4 to $6.5 \mu\text{m}$, and the TGO values were typically found within a range from ~ 2 to $3 \mu\text{m}$. The LPPS BCs, however, exhibited higher TGO thickness values. Thus, up to 500 h of isothermal exposure, there is not a significant difference among different processing techniques, except LPPS. The relationship between TGO growth and as-sprayed BC porosity values for the TBCs isothermally heat-treated at 930°C for 500 h can be found in Fig. 7. As seen in previous R_a results (Fig. 6), variations of BC porosity levels from ~ 1 to 10% do not lead to major changes in TGO thickness, except for LPPS BCs. It is hypothesized that the higher TGO thickness observed for the LPPS BCs is probably related to the lowest oxidation levels of all as-sprayed BCs, thereby

exhibiting higher amounts of non-oxidized Al in its structure to form the TGO upon thermal exposure. Consequently, the results of Figs. 1-7 showed that the initial TGO formation (930°C up to 500 h) seemed to be more dependent on the atmosphere where the coatings were deposited (i.e., absence of O₂), rather than particle T & V, porosity, residual stress and roughness values. Moreover, the LPPS BCs did not exhibit the typical lamellar microstructure, whereas, it was observed for the other BCs. Therefore, the porosity of the LPPS BCs was probably not surface-connected, thereby collaborating for yielding a thicker TGO.

Fig. 6. TGO thickness versus as-sprayed BC R_a after an isothermal exposure at 930°C for 500 h.

Fig. 7. TGO thickness versus as-sprayed BC porosity after an isothermal exposure at 930°C for 500 h.

The TGO thickness values after a 4000 h isothermal exposure at 930°C were measured and are described in Fig. 8. It is important to highlight that none of the TBCs failed from the isothermal exposure up to 4000 h. In addition, the TGO thickness values of the DJ and JP BCs were similar to those of HVOF-sprayed CoNiCrAlY isothermally exposed at 1000°C for 4000 h, as reported by Fossati et al. [3].

The TGO growth changed considerably even within the same type of torch/brand combination, such as for the APS-Axial III system. The majority of the TGO thickness values varied from ~4 to 6 μm. Higher TGO thickness is typically associated with TBC failure, however, just using a process like LPPS does not guarantee lower TGO growth rates. Therefore, the

results of Fig. 8 tended to show that the overall TGO growth under these conditions was relatively uniform up to 4000 h. As a consequence, isothermal treatment may not be an efficient or effective screening tool to predict TBC failure.

Fig. 8. TGO thickness for the torch type/brand combinations employed in this study after an isothermal exposure at 930°C for 4000 h.

Nevertheless, this study focused only on the TGO thickness and not on its composition and phase transformations. It has been shown by Chen et al. [4] that for APS and HVOF BCs after FCT, the alumina layer on the HVOF BC remained stable, whereas, that of the APS BC transformed into chromia and spinels. These phase transformations lowered the FCT life of the APS bond-coated TBC.

3.3 FCT

The BCs selected for FCT are listed in Table 3. The selections were based on the results obtained in Figs. 1-8 and other considerations, such as, the parameters recommended by the torch manufacturer for the specific torch/BC combination. A total of 6 torch/BC combinations were selected for TBC manufacturing and FCT.

It is important to point out that for each torch/BC combination, a total of 42 TBCs (i.e., BC + YSZ top coat) were produced. As 6 torch/BC powder combinations were employed, a total of 252 TBCs were produced for the FCT study. For each torch/BC combination, 6 TBCs were set aside: 1 used for metallography evaluation and 5 employed for bond strength testing based on the ASTM C633 standard. Prior to FCT, the bond strength values of as-sprayed and vacuum heat-treated TBCs were measured. During the course of the FCT, a series of 6 TBCs of each torch/BC combination were retrieved in an aleatory manner from the FCT rig for bond strength testing and SEM examination after 10, 100, 400 and 1400 cycles. The remaining TBCs were cycled up to failure.

The average of cycles to failure for the TBCs employed in this study is described in Fig. 9. All TBCs exhibited failure within the YSZ top coat and at the BC/top coat interface, as described by Rabiei and

Evans [5] and Wright and Evans [6]. Prior to failure (for SEM-analyzed TBCs), a horizontal crack was observed in the YSZ top coat parallel to the interface, adjacent to the TGO.

Table 3. BCs selected for secondary screening.

Technique	BC Torch	BC
APS	F4MB	12
	Axial III	A
		17
LPPS	SM LPPS	J
HVOF	DJ2600-hybrid	2
	JP 5000	X

Fig. 9. FCT summary for the TBCs tested.

It is possible to observe that, for the TBCs tested in this study, the two “highest FCT lives” and two “lowest FCT lives” each had one BC sprayed via HVOF and one sprayed via APS. The TBC for which the BC had been sprayed with the LPPS system exhibited an intermediate FCT life.

The difference between the FCT lives of the two APS BC-based systems (Axial III versus F4MB) may be partially explained by the following as-sprayed BC oxidation hypothesis. The axial feeding of the Axial III torch inserts the powder particles at the centre of the plasma jet where the plasma gases tend to shroud the powder particles against the atmosphere. For this torch, the particles tend to stay inside the centre plasma, relatively insulated from the air, until the moment they hit the substrate surface. In addition, the high H_2 flow rate employed with the Axial III (50 lpm) when compared to that of the F4MB (8 lpm) torch probably enhances the deoxidizing capabilities of the Axial III plasma, i.e., the reaction of the H_2 with O_2 molecules of the air to form H_2O vapour. The F4MB exhibits an external and radial powder injection. In order to have a “sufficient degree of melting” to form the coating, the particles must cross over the streamlines of the plasma jet, thereby being more exposed to the atmospheric air and more prone to as-sprayed oxidation.

The difference between the FCT lives of the HVOF BC-based TBCs (DJ versus JP 5000) probably can

not be explained on the oxidation issues. Both BCs sprayed with those torches naturally exhibit “low oxidation levels”. Nonetheless, one important point to highlight is the fact that the JP-X bond coat exhibited the highest roughness of all BCs employed in this study (Fig. 5). Therefore, the “low FCT performance” of this HVOF bond-coated TBC may be related to this issue. In addition, the as-sprayed DJ-2 BC was in “low” tensile stress, whereas, the JP-X, based on Fig. 4, was probably in “high” compressive stress. Although it is recognized that the low oxidation levels of the as-sprayed BCs are important for the improvement of TBC life (Chen et al. [4]), the FCT results (APS, HVOF and LPPS) showed that using a thermal spray system that yields low oxidation values in BCs alone does not necessarily guarantee improved TBC life.

Haynes et al. [7] have also shown that APS-BC TBCs can yield improved FCT lives when compared to LPPS-BC TBCs. It was reported that the coefficient of thermal expansion (CTE) issues had a significant influence on TBC life, by reducing stresses between the metallic substrate and the YSZ top coat. Patterson et al. [8] have also shown that APS-BC TBCs, in spite of significant oxidation during FCT, exhibited excellent thermal cycling life. This improved life was associated with reduced CTE mismatch. Saeidi et al. [9] measured the elastic modulus (E) values of HVOF and LPPS CoNiCrAlY BCs at high temperatures after annealing. The results show the E values of the HVOF-BCs are closer to the Inconel 625 substrate. The present authors hypothesized that an increased ceramic layer compliance from porosity and microcracking may also be involved.

The evolution of the bond strength values can be found in Fig. 10. The as-sprayed TBCs exhibited bond strength levels varying from ~22 to 37 MPa. After vacuum heat treatment pre-FCT, the data range was reduced to ~22-27 MPa. Consequently, the different BC spray processing techniques did not yield very significant differences in the bond strength values of the TBCs.

Fig. 10. The evolution of the bond strength values for the TBCs evaluated via FCT.

It was observed that the bond strength values for the TBCs subject to thermal cycling tended to slightly drop over time, i.e., they exhibited a relatively stable bond strength drop after the thermal cycling started. A major drop in the bond strength was noticed only when the TBC was about to fail, as observed for the DJ-2 and Axial III-17 TBCs at 1400 FCT cycles.

Moreover, the two highest as-sprayed bond strength values belonged to the DJ-2 and JP-X TBCs, respectively. Nonetheless, both TBCs exhibited radically different FCT lives (Fig. 9). Therefore, as-sprayed bond strength values do not seem to be an optimal indicator to predict the future TBC behaviour. The overall TGO that evolved in FCT is described in Fig. 11. At 1400 FCT cycles, when the TGO layers of the Axial III-A TBC reached a thickness value of $>10\text{ }\mu\text{m}$, it failed (Fig. 9).

Fig. 11. The evolution of the bond strength values for the TBCs evaluated via FCT.

The F4, JP and LPPS TBCs exhibited earlier failure, and if the data of their TGO formation of Fig. 11 is extrapolated, it can be inferred that the $10\text{ }\mu\text{m}$ value would be surpassed earlier on. The same type of extrapolation could be applied for the Axial III-17 and DJ-2 TBCs. Their failures occurred at ~ 1600 and ~ 1800 FCT cycles, respectively, and extra FCT cycles were needed to allow their TGO to surpass the $10\text{ }\mu\text{m}$ limit. Schmidt et al. [10] reported critical TGO thickness values of $8\text{--}11\text{ }\mu\text{m}$ for oxidation/fatigue thermo-mechanical loading of TBCs.

4 Conclusions

Although low oxidation levels in the as-sprayed BCs are considered of fundamental importance in TBC performance, just producing a low oxidized as-sprayed BC *per se* did not necessarily guarantee improved TBC life. Isothermal oxidation showed that the initial thickness of the TGO (930°C -500 h) was more dependent on the atmosphere where the coatings were deposited, rather than particle T & V, porosity, residual stress and roughness values. The overall isothermal TGO growth at 930°C -4000 h was relatively uniform for the majority of the BCs. Therefore,

isothermal treatment may not be an efficient screening tool to predict TBC failure. As-sprayed bond strength values are useful for quality control (QC) purposes, however, should not be used as a reliable indicator about the durability of a TBC. TBCs tend to exhibit a relatively stable bond strength drop after the thermal cycling started. Nonetheless, a major drop in the bond strength was noticed only when the TBC was about to fail. Moreover, during FCT the TBCs tended to fail after the TGO reached thickness levels higher than $\sim 10\text{ }\mu\text{m}$. The results of this study showed the importance of carefully selecting the most effective testing steps in order to predict the time-life of a TBC. In addition, this study indirectly highlighted the cost-effective issues, i.e., depending on the application, APS BCs can be technically and cost competitive.

5 Literature

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