The Effect of APS-HVOF Bond Coating Thickness Ratio on TBC Furnace Cycle Lifetime

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Abstract
Combinations of NiCoCrAlY APS and high-velocity oxygen fuel (HVOF) bond coatings were deposited on alloy 247 disk substrates with APS yttria-stabilized zirconia (YSZ) top coatings to assess the benefit of air plasma sprayed (APS) ‘flash’ bond coatings. Using 1-h cycles at 1100 °C in air with 10% H2O and HVOF-only and APS-only bond coatings as a baseline, it was found that APS flash coatings extended the average coating lifetime by 16% to 35% with the thicker flash coating performing best. Principal component analysis and energy dispersive spectroscopy (EDS) compositional mapping on coatings characterized after 0, 100, 300 and 500 cycles and after failure showed that the Al in the bond coatings was depleted due to both oxidation and back diffusion into the substrate. The APS-only bond coating had significant oxidation throughout the bond coating and was so depleted in Al, that Al diffused from the substrate to the coating. Residual stress maps of the thermally-grown alumina scale were obtained every 100 cycles using photo-stimulated luminescence piezospectroscopy (PLPS) revealing that the Thick Flash coating had the slowest rate of damage accumulation in the oxide scale.

Keywords Principal component analysis (PCA) · Furnace cycle testing (FCT) · APS flash coating · Alumina scale · TBC · HVOF bond coating

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Introduction

Natural gas-fired simple and combined cycle industrial gas turbines (IGTs) are the largest source of electricity in the US [1]. In a low-carbon future, IGTs may still be used to burn “green” hydrogen generated using renewables [2]. To maintain the turbine output with H₂ combustion, which has a lower energy density compared to natural gas combustion, the turbine inlet temperature could be increased, but this may impact durability and reliability, which are critical metrics for utilities and consumers. For the turbine hot section, a key materials technology to enable durability during higher temperature superalloy performance is thermal barrier coatings (TBCs) [3–5].

A current area of interest for TBC performance is the use of an air plasma sprayed (APS) MCrAlX (M=Ni and/or Co, X=Y, Hf and/or Si) flash coating applied over a dense high-velocity oxy-fuel (HVOF) coating. Such flash coatings have been shown to increase TBC lifetime in furnace cycle testing (FCT) on both flat disk and cylindrical rod specimens [6–9]. The enhanced performance has been attributed to improved micro-roughness of the surfaces resulting from the APS deposition process [9] and to the lower coefficient of thermal expansion of the APS layer vs. most superalloys, which acts as a mechanical buffer between the ceramic top coating and the substrate [10]. Additionally, the underlying HVOF layer provides an additional Al reservoir for enhanced oxidation resistance of the APS layer and further protects the substrate from oxidation attack [8].

This study was an initial attempt to determine whether the ratio of the HVOF layer to the APS layer could be optimized with respect to coating lifetime when compared to HVOF-only and APS-only bond coatings. Previously it was demonstrated that NiCoCrAlY flash coatings outperformed those containing both Y and Hf [7], so this study used only NiCoCrAlY powder for the APS and HVOF coatings. The coated superalloy disks were exposed using 1-h cycles in air with 10% H₂O at 1100 °C and (1) residual stress maps of the thermally-grown alumina scale were obtained every 100 cycles using photo-stimulated luminescence piezospectroscopy (PLPS) and (2) a time series of coating cross-sections were characterized using principal component analysis (PCA) of energy dispersive spectroscopy (EDS) maps to characterize the Al distribution in the coating as a function of exposure time.

Material and Methods

Three groups of six alloy 247 superalloy disks (1.59 cm diameter and 2 mm thick, all with the composition shown in Table 1) were coated on one side with an HVOF NiCoCrAlY bond coating using the powder composition listed in Table 1. An outer APS flash coating was then deposited on two set of these disks, hereafter referred to as Thin Flash and Thick Flash. One set of samples was not subsequently coated with a flash coating and so only had an HVOF coating. A fourth
| Material            | Ni  | Co | Cr  | Al  | Y   | Hf  | Si  | Ti  | W   | Ta  | Mo | C | Other (ppma) |
|---------------------|-----|----|-----|-----|-----|-----|-----|-----|-----|-----|----|---|--------------|
| Bond coating powder | 40.4| 19.6| 16.1| 23.9| 0.24| <   | 0.07| <   | 0.01| <   | <  | < | 13S          |
| Alloy 247           | 60.3| 9.9 | 9.7 | 12.6| <   | 0.047| 0.06| 1.3 | 3.2 | 1.0 | 0.45| 0.8| 19 Re, 6S    |
A batch of disks was coated with only an APS bond coating (APS-only). The starting bond coating powder size for both processes was $37.5 \pm 6.6 \mu m$. All the disks were annealed at $1080 \degree C$ for 4 h in a $10^{-4}$ Pa vacuum prior to the deposition of a ~200 $\mu m$ thick APS YSZ top coating.

Five specimens of each coating type were thermally cycled in air with 10 vol% $H_2O$ at $1100 \degree C$ to measure the TBC furnace cycle lifetime. The specimens were held on alumina rods using a Pt–Rh wire in an automated vertical furnace rig and each cycle consisted of 1 h at temperature followed by cooling for 10 min in ambient air to <30 $\degree C$. The samples were removed from the rig for inspection every 20 cycles with the failure criterion of >20% coating loss but failure typically was 100% of the YSZ coating spalling in one piece at the end of life. The sixth disk of each coating type was cycled in a similar manner but a portion of each coated disk was sliced off after 100, 300, and 500 cycles using a diamond saw for subsequent microstructural characterization. The same specimens were then cycled to and a portion of the disk was cut off at each stage to observe the microstructural evolution of the bond coating and oxide scale beneath the YSZ [11].

For characterization of the reaction products, specimens were mounted in epoxy, polished and imaged using light microscopy and scanning electron microscopy (SEM). Compositional maps were acquired using energy dispersive spectroscopy (EDS) (NSS, Thermo Scientific). Principal component analysis (PCA) within commercial software (Pathfinder, Thermo Scientific) was used to identify independent phases from the EDS maps. The phase maps for the oxides, pores and cracks were combined into one map which was then used as a template to set the Al atomic percentage maps to 0% for the non-metallic phases. This allowed the changes in the Al content only in the metallic phases to be observed.

Residual hydrostatic stress was non-destructively measured in the thermally grown $\alpha-Al_2O_3$ scale through the YSZ top coating after cycling using photo-stimulated piezospectroscopy (PLPS) as described elsewhere [12, 13]. A Raman microprobe (Dilor model XY800, Horiba Scientific, Edison, NJ) with an Ar$^+$ laser operating at 5145 Å was used to measure the same 500 by 670 $\mu m$ region with a step size of 10 $\mu m$ after every 100 cycles which allowed the local evolution of stress to be monitored during FCT on one specimen of each type of bond coating. Fractal analysis [9] and roughness measurements were performed on as-received cross-section images of each of the four bond coatings.

**Results and Discussion**

Figure 1 shows light optical microscope images of the as-sprayed (after vacuum annealing of bond coating) cross-sections of all four coatings. The HVOF-only coating appears dense across the entire bond coating as does the inner HVOF bond coating of the two flash coatings. The outer flash coatings contain internal oxidation that is oriented parallel to the coating surface and is typical of the APS process [14]. The APS-only bond coating also had internal oxidation between the splats oriented parallel to the surface. Figure 2 shows the starting thicknesses of the four bond coatings shown in Fig. 1. The HVOF-only and two flash coatings were of similar overall
thickness, while the APS-only coating was 20–40 µm thicker than the other coatings, which reflects the variability the process and was not intentional. As shown in Fig. 2, the flash coating thicknesses comprised 28% and 48% of the total coating thickness for the Thin Flash and Thick Flash coatings, respectively.

Figure 3 shows the roughness ($R_a$) and fractal dimension [9] at the YSZ/bond coating interface of all four as-received coatings measured on the cross-sectional images. The roughness of all four coatings fell within the same range of 4.4–5.2 µm which is significantly less than what was reported elsewhere for these
coatings [9, 10]. The fractal dimension \( D_f \) of the four coatings ranged between 1.08 and 1.12 which is also lower than values reported by Nowak, et al., on their flash coated sample [9]. The minimal difference in \( D_f \) among the four coatings in this work diminishes the confounding effect of \( D_f \) relative to other variables to be evaluated in the furnace cycle testing.

![Fig. 3](image1.png)

**Fig. 3** The starting roughness, \( R_\alpha \), and the fractal dimension \( (D_f) \) of the four bond coatings measured at the YSZ bond coating interface

![Fig. 4](image2.png)

**Fig. 4** TBC lifetimes (cycles to failure) following 1-h cycles in air + 10 vol% H\(_2\)O at 1100 °C. The error bars are one standard deviation of 5 specimens. The improvement from the HVOF-only and APS-only coatings to the flash coatings is indicated on the figure
Figure 4 shows the average lifetime for five specimens of each type of coating in 1-h cycles at 1100 °C in air with 10% H₂O. The best performing coating was the one with a Thick Flash coating, which had an average lifetime of 1104 ± 71 cycles which was an improvement compared to HVOF-only and APS-only coatings of 35% and 70%, respectively. The Thin Flash also improved average lifetime (by 16%) over the HVOF-only coating which was half of the improvement of the Thick Flash coating. The APS-only coating had the shortest average lifetime.

Using the same methodology applied in previous studies [7, 8], the mean hydrostatic compressive stress measured with PLPS in the thermally-grown Al₂O₃ scale is shown in Fig. 5. For all four coatings, the stress gradually decreased with thermal cycling due to damage accumulation in the scale as observed in previous studies [12, 15–17]. The rate of residual stress relaxation from fastest to slowest was HVOF-only, Thin Flash, Thick Flash and APS-only. The stress in the HVOF-only coating relaxed fastest because the dense bond coating was the least strain tolerant during thermal cycling and so cracking in the scale accumulated most rapidly, likely leading to a shorter lifetime compared to the two flash coatings. The APS-only coating had the lowest mean stress at 100 and 200 cycles which would indicate more scale cracking. However, after 600 cycles, it had the highest remaining mean hydrostatic stress.

Figure 6 shows cross-sectional back-scatter electron images of the four bond coatings in rows and the FCT duration (as-received, 100, 300 and 500 cycles, and failed) in columns. After 100 cycles, the two flash coatings and the APS-only coating show extensive oxidation of the inter-splat regions as O ingress proceeds along the

![Fig. 5 Mean compressive stress measured in the Al₂O₃ scale using PLPS versus number of 1-h cycles at 1100 °C of the four TBCs studied in this work. The measurements are the average of 3468 individual stress measurements, and the error bars are one standard deviation. Measurements were collected every 100 cycles, and the data points are shifted slightly for clarity]
metal-oxide interfaces. The APS-only coating thickens with further cycling leading to a noticeable 30% total bond coating thickness increase of the APS-only coating at failure. In contrast, the dense HVOF-only coating only oxidizes on the outer surface.

Fig. 6 Back-scattered electron images of HVOF-only, Thin Flash, Thick Flash and APS-only bond coatings from the top to the bottom rows. The columns show specimens following 0, 100, 300, and 500 cycles and after failure, in that order from left to right. The cycles-to-failure of these samples are listed on the images in the final column

Fig. 7 The Al atomic percentage in the metallic phases of the images shown in Fig. 6. The rows from top to bottom correspond to the HVOF-only, Thin Flash, Thick Flash and APS-only coatings. The columns from left to right are samples after 0, 100, 300 and 500 cycles, and after failure
Maps of the Al atomic percentage within only the metallic phases corresponding to the images of Fig. 6 are shown in Fig. 7. The range of the color bar (0 to 35 at%) was chosen to highlight different metallic phases with red being $\beta$-NiAl, green being $\gamma'$-Ni$_3$Al and blue being $\gamma$-Ni(Al). All four coatings start off at 0 cycles with significant $\beta$-NiAl and ~25 at.% Al, whereas the 247 substrate is green–blue indicating the $\gamma$/$\gamma'$ structure of this alloy. The initial increase in the Al content in the substrate adjacent to the bond coating as evidenced by the green layer is attributed to diffusion of Al from the bond coating into the substrate during the 4-h vacuum anneal at 1080 °C.

After 100 cycles of exposure at 1100 °C, several changes can be observed across the four coatings (Fig. 7, 2nd column of images). First, the APS-only coating has completely lost both the $\beta$-NiAl (red), and the $\gamma'$-Ni$_3$Al (green) metallic phases due to significant internal oxidation of Al to Al$_2$O$_3$ and other transient oxides across the entire coating cross-section. This has reduced the Al concentration in the APS-only coating to ~8 at% after only 100 h. Again, oxidation is able to proceed rapidly because of O diffusion along metal-oxide interfaces through the APS coating. Conversely, the dense HVOF-only and the two flash coatings still contain both $\beta$-NiAl and $\gamma'$-Ni$_3$Al after the same exposure; however, the $\beta$-NiAl phase is decreasing in abundance as the flash coating thickness increases. This shows how the HVOF inner layer is acting as an Al reservoir for the oxidation of the outer flash coatings. Also observable after 100 cycles is the increase in Al concentration in the substrate (it appears greener) in the three HVOF-containing coatings which indicates that Al is also being lost from the coating due to diffusion from the bond coating into the substrate during the exposure. However, the opposite happens with the APS-only sample with Al diffusing from the substrate into the bond coating. This difference will be discussed in more detail below.

As thermal cycling continues from left to right in Fig. 7, the Al concentration in all four coatings steadily decreases both due to oxidation and interdiffusion with the substrate. The thicker the APS layer, the faster the loss of Al from the HVOF layer. After 300 cycles, the Thick Flash coating has lost its $\beta$-NiAl (red) phase but still contains reserve Al to accommodate further oxidation of the flash coating. Both the HVOF-only and Thin Flash coatings have $\beta$-NiAl (red) remaining at failure (last column in Fig. 7). The longest-lasting Thick Flash coating retained some Al in the coating at failure but the retention of $\beta$ phase in the coating does not appear to be necessary to maintain YSZ adhesion.

Figure 7 also shows that the Al content of the metal within the flash regions is more depleted in Al (see Thin and Thick Flash at 100 and 300 cycles) than the HVOF layer. Also, the $\beta$-NiAl phase appears to coarsen with thermal cycling as shown by the increase in the size of the red grains from left to right in Fig. 7. Finally, the APS-only coating has only ~5 Al at% remaining at failure which will favor the formation of Ni-rich spinel-type oxides which are known to cause failure of TBC coatings because of the associated volume expansion [18–20].

Figure 8 shows line scans of the Al concentration in the substrate under the Thick Flash and APS-only coatings at 0 and 500 cycles. Initially, the Al concentration is ~11 at% for both coatings and does not vary from the bond coating interface into the substrate. After 500 cycles, the substrate adjacent to the Thick Flash coating and
extending a further ~150 µm under the coating has a higher Al at% indicating that Al has diffused from the bond coating into the substrate. Conversely, the substrate under the APS-only coating has lost significant amounts of Al due to diffusion into the bond coating. This is due to the extensive oxidation of the APS-only coating which reduces the Al concentration in the bond coating below that of the 247 substrate thereby favoring diffusion of Al from the substrate into the bond coating. This shows that a pure APS coating can degrade the substrate during high-temperature exposure and that a dense Al-rich HVOF layer is needed between the outer flash coating and the substrate to protect the substrate from the effects of oxidation. It appears that the Al reservoir of the 247 superalloy substrate extended the lifetime of the APS-only coating by supplying Al to it near the end of life. If this experiment were repeated with a superalloy substrate with lower Al content, it is likely that the difference between the 100%APS and flash coating lifetimes would be even larger.

Finally, Fig. 9 shows maps of Al₂O₃ (blue), Cr-rich spinel (white) and cracks/pores (green) in cross-sections of the four coatings after 500 cycles generated using PCA of the EDS maps and corresponding to images in Fig. 6. The volume of Al₂O₃ increases as the thickness of the APS layer increases with the APS-only coating being mostly Al₂O₃ after 500 cycles. The Cr-rich spinel oxides (white) were not observed for the HVOF-only coating but are present in small amounts in the two flash coatings. However, the APS-only coating has far more Cr-rich spinel, especially adjacent to the YSZ top coating. The spinel phase will form on bond coatings that have been depleted in Al and is indicative of the end of coating life [14]. Oxidation that results in a spinel phase produces a large volumetric expansion compared to Al₂O₃ which is deleterious to the mechanical integrity of the coating. The formation of this phase may explain the higher compressive stress in the Al₂O₃ scale observed for this coating compared to the other coatings in Fig. 5. The damage in this coating
is further evidenced by the large crack (green) that has formed in the APS-only coating at the bond coating/YSZ interface in this image. Conversely, the HVOF-only and two flash coatings have less cracking at the interface after 500 cycles.

These results confirm previous observations [5, 6] that the bi-layer flash coating structure with an inner HVOF layer confines the mixed metal-oxide structure to the outer portion of the coating and supplies Al to the metal in this layer. Once pathways for the Al to diffuse to the metal in the intermixed layer from the dense HVOF layer are cut off due to oxidation and cracking, the outer flash layer will become Al depleted and begin to form Cr- and Ni-rich oxides hastening failure [14]. This depletion is more striking in the APS-only coating which becomes rapidly depleted in Al and forms spinel-type oxides leading to earlier failure, Fig. 4. The APS-only coating also illustrates the importance of the inner HVOF layer for preventing Al depletion of the superalloy substrate with the bond coating acting as a protective coating that can be reapplied multiple times during the service life of the coated turbine component. The depleted superalloy with the APS-only coating was more heavily damaged making repair and recoating more difficult. The lifetime improvement imparted by the flash coating over the HVOF-only coating is likely due to the reduction in the thermomechanical mismatch stress between the top coating and the substrate caused by the intermingled oxide/metal flash coating. Further work is needed to explicitly demonstrate that the outer flash coating provides improved strain tolerance during thermal cycling leading to longer TBC life. For these FCT test conditions, the Thick

![Fig. 9 PCA of EDS maps of the four coatings following 500 cycles. The Al₂O₃, Cr-rich spinel and cracks/pores are shown in blue, white and green, respectively.](image-url)
Flash coating with a 50:50 HVOF/APS ratio resulted in the best performance but more prototypic testing in a thermal gradient is needed to determine the optimum ratio for service conditions.

**Conclusion**

The performance of four different YSZ-coated bond coatings (HVOF-only, Thin Flash, Thick Flash and APS-only) in furnace cycle testing was studied using 1-h cycles at 1100 °C in air with 10% water vapor. The bond coating with the highest average coating lifetime was the Thick Flash coating which had roughly 50:50 HVOF/APS layer ratio and increased coating lifetime 35% and 70% over the HVOF-only and APS-only coatings, respectively. In both cases, the outer APS layer converted into a convoluted mixture of interlocking metal and oxide that retained a higher residual stress in the alumina than a conventional HVOF-only coating. A time series of observations of the coating composition evolution illustrated that the APS-only coating quickly became depleted in Al due to internal oxidation, while the HVOF layer prevented oxide ingress while acting as an Al reservoir for the APS flash layer as well as preventing Al depletion of the superalloy substrate. The use of principal component analysis assisted in illustrating the temporal changes in the microstructure and chemical composition of the coatings.

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**Author contributions** Michael Lance and Bruce Pint wrote the main manuscript text. Michael Lance conducted the research. Ken Kane conducted the furnace cycle testing. All authors reviewed the manuscript.

**Declarations**

**Competing interests** The authors declare no competing interests.

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