Study on the Characteristics of a TBC System Containing a PVD-Al Interlayer under Isothermal Loading

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Abstract: In this work, the oxidation behavior of an atmospheric plasma-sprayed thermal barrier coating (TBC) system with a thin Al physical vapor deposition (PVD) film deposited over the bond coat is discussed. The TBC consisted of: (i) CoNiCrAlY bond coat sprayed on the Inconel 600 substrate; (ii) a thin Al interlayer deposited by direct current DC magnetron sputtering; and (iii) yttria-stabilized zirconia (YSZ) sprayed as the top coat. Such thermal barrier coatings (Al-TBC) were isothermally oxidized at 1150 °C with different holding times, and then they were compared with the reference TBC (R-TBC) systems without an Al interlayer (R-TBC). Scanning electron microscopy with energy-dispersive X-ray analysis was used to study the oxide formation along the bond coat (BC) and top coat (TC) interface, as well as crack formation in the YSZ top coat. Then, using Image Analysis, the oxide formation and crack formation were characterized in all specimens after a slow heating and cooling cycle, and after 100, 300, and 600 h of isothermal exposure. The results showed that the Al-TBC system proposed here exhibits higher oxidation resistance at the bond coat and top coat interface, less crack formation in the YSZ top coat, and enhanced mechanical stability compared to the conventional TBCs. It was found that enrichment of the bond coat and top coat interface with Al limited the formation of detrimental transition metal oxides during isothermal loading. Finally, the corresponding failure caused by thermally grown oxide (TGO) phenomena is “mixed failure mode” for both studied TBCs.

Keywords: thermal barrier coatings; thermally grown oxides; isothermal exposure; crack formation; oxidation model

1. Introduction

Contemporary gas turbine technology development is largely dependent on progress in the field of thermal barrier coatings (TBCs). The current generation of TBC consists of two main layers: (i) a metallic bond coat (BC), which is sprayed directly onto the turbine blade; and (ii) a ceramic top coat (TC), which insulates the turbine components from high heat loads [1,2]. The BC layer is usually composed of MCrAlY (M = Ni, Co, or NiCo alloy) or (Ni,Pt) Al and provides effective protection for the substrate against oxidation at elevated temperature. It further compensates the thermal expansion gap between the super alloy substrates and the ceramic top coat [3,4]. The top coat is mostly composed of stabilized zirconia (rare-earth-doped zirconia, primarily with yttrium) [5,6].
Conventional TBCs have been in commercial use since the 1980s [7]. However, many related problems must be better understood and solved to increase the lifetime and efficiency of gas turbines. The research topics in the field of TBCs can be categorized in two groups: (i) finding new materials for thermal barrier coatings [8,9], and (ii) finding new technologies to produce coatings with improved properties at high temperature [10–12]. On the other hand, there are also interdisciplinary issues. One of them is the control of the formation of so-called thermally grown oxides (TGOs) and their influence on the BC/TC interface, which plays an important role in determining the lifespan of TBCs [13,14].

The TGO phenomena correspond to oxidation of the bond coat during the service. In cases of BC made of MCrAlY, TGOs usually consist of \( \alpha-Al_2O_3, NiO, Cr_2O_3, \) or spinel \((Co, Ni) (Cr, Al)_2O_3\) [15]. TGO growth and increasing stress levels in the BC/TC interface are considered as the most limiting factor for the TBC service time, the key element for enhancing the TBC service time is to control the oxide growth mechanism in the BC/TC interface [16,17].

In this study, the BC and TC interface in a reference atmospheric plasma-sprayed (APS) TBC system was enriched with a thin Al PVD film to increase the Al activity in this area, and hence promote the formation of \( \alpha-Al_2O_3\). \( \alpha-Al_2O_3\) is known to be chemically stable and provides low oxygen diffusivity; therefore, it slows down the TGO formation rate and enhances the durability of TBC systems. The TGO formation characteristics during isothermal loading were studied and compared to similar TBC systems without an Al interlayer. Finally, an oxidation model is introduced for TBC containing an Al PVD interlayer and compared with the reference TBC (R-TBC).

2. Materials and Methods

The TBC systems were deposited by atmospheric plasma spraying on Inconel 600 substrates (DIN 2.4816, UNS N06600). The system included a bond coat of CoNiCrAlY (CoNi32Cr21Al8Y0.5, 60.95.1, GTV GmbH, Luckenbach, Germany) and an yttria-stabilized zirconia (YSZ) top coat (ZrO\(_2\) + 8 wt.\% of Y\(_2\)O\(_3\), Oerlikon Metco 204NS, Westbury, NY, USA). The BC layer was sprayed by an F6 plasma torch (GTV GmbH) to reach a thickness of 150 µm. A thin Al layer was deposited by DC magnetron sputtering on the as-sprayed surface of the CoNiCrAlY BCs with a nominal film thickness of 2 µm. The sputtering was performed under working pressure of 0.8 Pa using an Al target (purity 99.999%, EVOCHEM Advanced Materials GmbH, Offenbach, Germany) and 50 sccm Ar (purity 99.9999%) as a sputtering gas. Then, the roughness raw bond coat surface and the Al-covered bond coats were measured using a contact stylus profilometer according to ISO 11562. Finally, 8YSZ coating was sprayed as a top coat using an SG-100 plasma torch (Praxair S.T., Indianapolis, IN, USA) with thickness of 150 µm. The reference TBCs, i.e., without an Al interlayer, were included in the research plan for comparison (Figure 1).

![Figure 1](image-url). The scheme presents the two TBC systems considered in this work: (a) R-TBC—the reference system, and (b) Al-TBC—system with an Al interlayer.

In the isothermal exposure tests, the specimens were submitted to the following treatments:
• Heating from room temperature (RT = 20 °C) up to 1150 °C with an estimated heating rate of +6 °C/min;
• Exposure with a dwell time of 0 (i.e., heated and directly cooled down), 100, 300, and 600 h;
• Cooling down to room temperature with an estimated cooling rate of −0.5 °C/min.

All tests were performed in a batch furnace (KT 65/230.19, Linn High Therm GmbH, Eschenfelden, Germany) under an air atmosphere.

After isothermal oxidation, all TBCs were metallographically prepared and investigated using scanning electron microscopy and energy-dispersive X-ray analysis (SEM/EDX, Zeiss VP 1455 incl. EDAX Genesis, Carl Zeiss Microscopy GmbH, Jena, Germany). The micrographs were then processed with “Fiji” software (open source, built on ImageJ2 core) in order to measure the TGO thicknesses in the BC/TC interface and crack lengths in the YSZ TC, after different thermal exposure times. The thickness of the thermally grown oxide layer in the BC/TC interface was calculated based on Equation (1) [18]:

\[
\delta_{eq} = \frac{\sum \text{cross sectional area of TGO}}{\text{cross sectional length of TGO}}
\]  

SEM images with magnifications of 100× and 400× were used to study the cracks and TGOs in the TBCs discussed here. The average values of 25 or 35 measurements, i.e., 25 or 35 micrographs depending on the SEM magnification, were used for crack length and TGO thickness measurements, respectively.

3. Results and Discussion

3.1. Microstructure of as-Sprayed TBC Systems

The microstructures of both TBC systems considered in this study, i.e., R-TBC and Al-TBC, are presented in Figure 2. The BC and TC layers show the typical microstructure of APS coatings. For metallic bond coats, well-flattened spatters are visible, although some single pores and oxide scales are visible as well. According to image analysis and EDX studies, the porosity was about 2% and the oxide content was 22%. The YSZ top coat also showed a typical lamellar structure with a pore content of about 7%. Both systems showed a good mechanical interlocking of the top coat to the bond coat layer.

![Figure 2. Backscattered scanning electron microscope images (BSD-SEM) presenting the as-sprayed coatings: (a) R-TBC and (b) Al-TBC.](image)

The Al interlayer (Figure 2b, dark line at the BC/TC interface) was neither destroyed nor diffused during the spraying of the outer YSZ layer. However, in some single places, the Al film was not continuous, resulting in single direct contact zones between the BC and TC in the as-sprayed condition (Figure 2b, labelled with circles). There could be several reasons for such Al PVD layer discontinuities: (i) local shadowing caused by the rough as-sprayed surface of the CoNiCrAlY bond coat; (ii) differences in the Al grain growth kinetics during the sputtering process; or (iii) local removal of the thin Al interlayer by impinging YSZ particles during top coat deposition. The bond coat’s surface roughness was not influenced by the deposition of an additional Al interlayer. Roughness measurements...
revealed $Ra = 7.1 \mu m$, $Rz = 48.6 \mu m$ for the bare bond coat surface, and $Ra = 7.2 \mu m$, $Rz = 50.5 \mu m$ for the Al-enriched bond coat.

3.2. Microstructure after Thermal Treatment

The cross-sectional microstructures of the TBC systems after one heating and cooling cycle are shown in Figure 3. The R-TBC system already showed the formation of typical TGOs in the BC/TC interface (Figure 3a), comprising a combination of $Cr_2O_3$, $\alpha-Al_2O_3$, $NiO$ and spinel oxides of the type $(Ni,Co) (Cr,Al)_2O_4$ [19–21]. In comparison, the Al-TBC system (Figure 3b) exhibited slightly different oxidation behavior. At the interface between the bond coat and top coat of the TGO layer, direct contact zones between metallic BC and YSZ TC (labelled with white rectangle in Figure 3b) and cavities (labelled with circles in Figure 3b) can be observed. The origin of formulation of cavities could be related to the vertical diffusion of aluminum into the bond coat during thermal exposure. The cavities did not result in significant weakening or separation of the BC and TC interface. This can be explained by numerous direct contact zones and sufficient mechanical interlocking between the subsequent TBC layers.

![Figure 3. Backscattered scanning electron microscopy images (BSD-SEM) presenting the coatings after one heating and cooling cycle: (a) R-TBC and (b) Al-TBC.](image)

The growth and chemical composition of oxides formed at the BC/TC interface in both TBC systems was studied by EDX and presented in a form of element mapping (presented in Figure 4). After one heating and cooling cycle, the bond coat and top coat interface of the reference TBC system revealed a TGO layer rich in Cr, Co, Al, and O. The interface of the Al-TBC system showed mainly Al and O. The Al interlayer exhibited two different effects: it partly melted and diffused into the bond coat; or, it oxidized and quickly formed the aluminum oxide at the top of the bond coat, which thickened during further isothermal exposure at high temperature.

The cross-sectional microstructures of both TBC systems subjected to 100, 300, and 600 h of isothermal exposure are presented in Figure 5. In general, the R-TBC system (Figure 5a,c,e), compared to the Al-TBC system (Figure 5b,d,f), showed extensive TGO formation (dark grey regions labelled with “TGO” in Figure 5) in the BC/TC interface. In addition, after 600 h of isothermal exposure, the BC microstructures of the R-TBC system revealed much higher oxidation levels than the BC microstructure of the Al-TBC system (Figure 5e,f).

In the Al-TBC system, after 100, 300, and 600 h of isothermal exposure (Figure 5b,d,f), the formation of a semicontinuous dark line along the CoNiCrAlY BC microstructure (on the CoNiCrAlY BC facing side and close to the BC/TC interface, labelled with arrows in Figure 5b,d,f) was observed. To investigate the elemental composition of the dark line, EDX mapping was performed for the Al-TBC system. The elemental distribution after 100 and 300 h of isothermal exposure is shown in Figure 6. According to EDX studies, the dark line is rich in “Al” and “O”. This may suggest that the Al layer diffuses gradually during isothermal exposure in the BC, and the dark line corresponds to the diffusion front of Al into the BC. This line also constitutes a kind of barrier for further oxidation of the CoNiCrAlY bond coat.
Figure 4. EDX mapping of the TBC systems after one cycle of isothermal exposure: (a) R-TBC and (b) Al-TBC. The arrows pointing upward indicate the TGO layer in the BC/TC interface.

Figure 5. TBC systems after isothermal exposure at 1150 °C: R-TBC (on the left) and Al-TBC (on the right). Dwell time: (a,b) 100 h of isothermal exposure; (c,d) 300 h of isothermal exposure; and (e,f) 600 h of isothermal exposure. The arrows pointing upward mark a dark line rich with “Al” and “O” along the CoNiCrAlY BC microstructures and close to the BC/TC interface on the CoNiCrAlY BC facing side; extended from [22].
3.3. TGO Thickness and Cracks Measurements

First, analysis of the maximum equivalent TGO thicknesses formulated in the BC/TC interface of both TBC systems was carried out. The effect of the dwell time duration at elevated temperature on the equivalent TGO thicknesses is shown in Figure 7a, for R-TBC and Al-TBC systems. As one may expect, the equivalent TGO thickness increased in both systems with the increasing holding time. However, this effect was much more pronounced in the conventional R-TBC system (Figure 7a). The maximum equivalent TGO thickness was much lower in the case of the Al-TBC system (achieving even a 50% reduction). After 600 h of isothermal exposure, it even reached 67 and 38 µm for the R-TBC and Al-TBC systems, respectively. The normal distribution, representing 35 measurements of the equivalent TGO thickness formed during 300 and 600 h of isothermal exposure, is shown in Figure 7b,c, for both TBC systems. After 300 and 600 h of isothermal exposure, the Al-TBC system showed an average equivalent TGO thickness of around 20 and 24 µm, respectively, in comparison to the R-TBC system with thicknesses around 28 and 41 µm. The results prove that the modified TBC system with an Al interlayer (Al-TBC), in comparison to the reference TBC system (R-TBC), exhibits smaller average equivalent TGO thickness in the BC/TC interface after isothermal exposure, which means that it is characterized by higher oxidation resistance in the BC/TC interface than conventional R-TBC systems. Moreover, the TGO thickness distribution is much narrower in the case of Al-TBC, which suggests that the oxidation process in that TBC system is more stable and predictable.

Quantitative analysis of the maximum horizontal crack lengths generated during isothermal exposure in both TBCs is presented in Figure 8. It was observed that the growth of TGOs and horizontal cracks are connected proportionally, which corresponds to the general knowledge about thermally sprayed TBCs. The correlation between holding time and maximum horizontal crack length in both TBCs is shown in Figure 8a. It can be observed that with the increase in the holding time at elevated temperature, the maximum horizontal crack length increased in both R-TBC and Al-TBC systems, but the effect was much more pronounced for the conventional TBC system, i.e., without an Al interlayer.
The maximum horizontal crack length was much lower in the case of the proposed Al-TBC system (up to a 50% reduction). Similarly, as in the case of TGO thickness, the normal distribution of the crack length was plotted based on 25 measurements. It is shown for both TBC systems in Figure 8b,c after 300 and 600 h of isothermal exposure, respectively. After 300 and 600 h dwell times, the Al-TBC system showed a slightly lower average horizontal crack length and a narrower crack length distribution.

Figure 7. Analysis of TGO formation: (a) the effect of dwell time duration at 1150 °C on the equivalent TGO thicknesses; (b) normal distribution of equivalent TGO thicknesses after 300 h; and (c) normal distribution of equivalent TGO thicknesses after 600 h of isothermal exposure.

Figure 8. The effect of holding time at 1150 °C on the maximum horizontal crack lengths in the R-TBC and Al-TBC systems: (a) distribution of the maximum horizontal crack length with respect to the holding time; (b) normal distribution of the maximum horizontal crack length after 300 h of isothermal exposure at 1150 °C; and (c) normal distribution of the maximum horizontal crack length after 600 h of isothermal exposure at 1150 °C.

The investigated microstructures during isothermal exposure are in accordance with the ‘isothermal failure mode’ showing crack paths across the TGO for both analyzed TBC systems, as described in the literature [23–25]. In Figures 7 and 8, one can see that the R-TBC system is characterized by more extensive thermally grown oxide formation in the bond coat and top coat interface and more evident delamination cracking along the YSZ TC near to the BC/TC interface, in comparison to the Al-TBC system. This proportional connection is a typical phenomenon and has been reported [26–29]. Based on this, the possibility to stabilize and slow down the TGO growth using an Al interlayer seems to be a promising approach to increase the lifetime of TBC systems. By enriching the BC/TC interface with Al, it appears possible to stabilize and prolong the so-called TGO steady-state growth stage, mainly including Al₂O₃ formation, and is known to influence the overall durability of TBC systems, as discussed by Chen [30]. However, the mechanism as to how
the Al interlayer limits TGO formation in the BC/TC interface is not presented in this study, and is the subject of ongoing research.

3.4. Crack Formation and Oxidation Sequence in TBC Systems

The as-sprayed R-TBC system revealed direct contact in the interface between the CoNiCrAlY bond coat and the YSZ top coat (Figure 9a). After preliminary isothermal exposure, the BC/TC interface mainly revealed the formation of an intrinsic and nonhomogeneous mixed oxide layer with single Al-enriched regions (Figure 9b). The oxide formation in the BC/TC interface increased the compressive stress level in the BC/TC interface. As a result, longer isothermal exposure caused further increases in oxide growth, raised this stress profile at the BC/TC interface, and resulted in crack initiation, as already described in the literature [31]. Afterwards, accelerated, e.g., by thermal expansion mismatch or sintering, the TBC failure started by opening and connecting the initially formed short horizontal cracks (Figure 9c). Finally, longer horizontal cracks were formed, and spallation of the TC layer occurred [25].

Figure 9. Schematic representation and comparison of the oxidation sequence for the TBC systems: (a–c) R-TBC; (d–f) Al-TBC. Mixed oxides are labelled in gray, Al-rich oxidase is labelled in black, and cracks are labelled with red.
A model for the oxidation sequence of the presented Al-TBC system is displayed in Figure 9d–f. The thin Al interlayer in the as-sprayed Al-TBC showed some single discontinuities, which corresponded to direct contact zones in the BC/TC interface (marked in Figure 9d). After the first isothermal cycle, a thin layer rich in metal oxides, oxide scale rich in “Al” and “O”, as well as cavities, were observed in the BC/TC interface (Figure 9e). Together with further exposure to isothermal oxidation, the formed cavities provided a free space for TGOs to grow freely without introducing additional stresses in the BC/TC interface. This proceeded until the volume of cavities was fully filled with oxides. Afterwards, single cracks started to form in the BC/TC interface as a result of additional stresses generated by gradually formed oxides. Longer exposure to elevated temperature stimulates the growth of oxides in the bond coat and top coat interface, as well as subsequent cracks in the YSZ ceramic layer. In addition to that conventional TGO growth mechanism [31], a semicontinuous Al- and O-rich front formed within the bond coat layer close to the BC/TC interface (Figure 9f), probably formed by Al that infiltrated the BC via particle interfaces or by diffusion after extended isothermal exposure.

4. Conclusions

The present study has clearly demonstrated that the discussed Al-TBC system containing a thin Al PVD layer on the CoNiCrAlY bond-coat has better oxidation resistance than the conventional R-TBC system. The reason was related to higher Al chemical activity in the BC/TC interface. This resulted in numerous effects during long-term exposure to elevated temperature, i.e., the formulation of cavities enabling reductions in stresses during TGO growth in the bond coat and top coat interface, extending the onset and steady state growth stage of the TGO, and the ability to form a protective α-Al₂O₃ layer as a barrier for further oxidation of the bond coat layer.

The investigated microstructures of both TBC systems reveal the so-called ‘mixed-failure mode’ after 600 h of isothermal exposure. The altered TBC with PVD-Al interlayers is characterized by a reduced equivalent TGO thickness and crack formation in the BC/TC interface during and after thermal treatment in comparison to the conventional R-TBC system. The proposed TBC systems comprising a PVD-Al interlayer are also characterized by a lowered tendency to form cracks within the YSZ TC during isothermal exposure tests. Accordingly, the conducted approach is understood as a promising and easy-to-perform possibility to enhance the service time of TBC systems in general.

These first results were gained only for the specific APS TBC systems. Further investigations using a broader range of materials, spraying methods, and parameters will be carried out in future research.

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