

Investigation of failure mechanism of thermal barrier coatings (TBCs) deposited by EB-PVD technique

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Abstract. Failure mechanism of thermal barrier coatings (TBCs) prepared by electron beam physical vapor deposition (EB-PVD) technique owing to formation of micro cracks was investigated. The TBCs were deposited on the Ni-based super alloy IN-100 and the micro cracks were observed within the top ceramic coat of thermally cycled TBCs at 1050°C. It was observed that these cracks propagate in the ceramic coat in the direction normal to interface while no cracks were observed in the bond coat. SEM/EDS studies revealed that some non-uniform oxides were formed on the interface between ceramic top and metallic bond coat just below the cracks. Study proposed that the cracks were initiated due to stress owing to big difference in Pilling-Bedworth ratio of non-uniform oxides as well as thermal stress, which caused the formation of cracks in top ceramic coat leading to failure of TBCs

1. Introduction

Demand for enhanced jet engine efficiencies has led to significant increase in combustion temperatures and operating pressures. These requirements have proved to be a big driving force to improve the super alloy technology, such as alloying, production techniques and different coating techniques. Consequently, an increase in turbine inlet temperature demands materials with higher melting temperatures i.e. intermetallics or ceramics. An intermediate development stage is the use of the super alloys with an oxidation resistant bond coating and a ceramic top coating, so-called thermal barrier coatings (TBCs). TBCs are layer systems deposited on thermally highly loaded metallic components, as for instance in gas turbines [1-4]. The TBC concept is valuable because the mechanical properties of the metallic super alloy are maintained and the alloy is protected against the high temperature due to the low thermal conductivity of the top ceramic coating. It is now well established that yttria partially stabilized zirconia (YSZ) TBCs due to low thermal conductivity can lead to significant improvements of turbine engine efficiency and hot component's life time [5]. The state-of-the-art TBC consists of two layers. Top ceramic coat, 6.5-8 wt.% Y₂O₃ partially stabilized zirconia (YSZ), protects the substrate from high temperature. Metallic bond coat MCrAlY (Where M indicates Ni, Co, Fe or their combination) is deposited next to substrate to protect it from oxidation and corrosion at high temperatures as well as it helps in reducing thermal expansion mismatch between the ceramic coat and the super alloy [6,7]. This bond layer also provides the mechanical

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bonding between the substrate and the top ceramic coat. This multilayer system is not thermally stable under long term exposure at elevated temperatures. A thermally grown oxide (TGO) is formed by oxidation at the bond coat/top ceramic coat interface. This scale consists mainly of alumina (Al_2O_3) formed by selective oxidation of aluminum from the bond coat. The thermally grown oxide at the bond coat/ceramic coat interface plays vital role for spallation and lifetime of TBCs.

Previous investigations mainly focused on plasma sprayed TBCs, the most prominent characteristics of which are the loss of adherence and spallation of ZrO_2 . The Use of EB-PVD method opened a new era of fabricating TBCs of very high quality. Schematic diagram of EB-PVD is shown in figure 1.

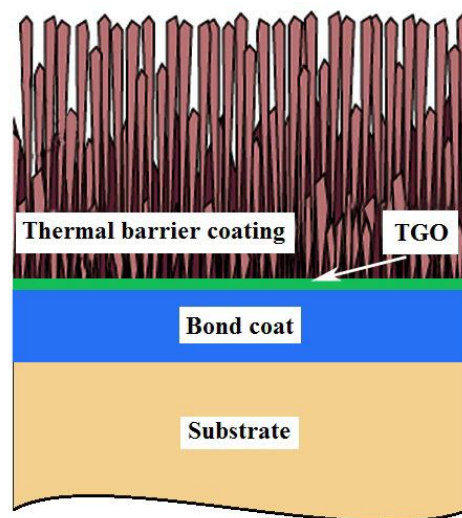


Figure 1. A schematic diagram of an electron beam physical vapor deposited YSZ coating with elongated pores aligned perpendicular to the substrate surface.

EB-PVD TBCs offer significantly extended lifetimes compared with APS TBCs due to its better controlled processing [8,9]. TBCs failure is expected to result either from stresses developed upon heating to elevated temperatures or from stresses developed upon cooling to ambient temperatures. Failure by either mode may be influenced or even controlled by the initial residual stress state of the coating or by the effects of degradation due to the thermally activated processes such as bond coat oxidation, or bond coat plastic deformation and phase transformation [10].

A large number of publications deal with the improvements in lifetime of the TBCs [10-14]. Significant advances in coating durability have been realized through improvements in coating materials and processing conditions for plasma spray and other coating techniques.

Future improvements will be greatly facilitated if coating failure mechanism is more rigorously investigated. Thus the purpose of this study is to elucidate the effects of oxidation of the metallic bond coat under cycling conditions on the failure mechanism of the TBCs.

2. Experimental

The specimens used for experimental purpose were cut from a turbine blade material IN-100 superalloy with nominal composition of 10Cr-60Ni-15Co-3Mo-5.5Al-4.7Ti-1.0V-0.15C-0.06Zr-0.015B. Specimens were ultrasonically cleaned in alcohol bath. At a constant temperature of the

substrate and working pressure in the chamber, the microstructure and porosity of ceramic ZrO_2 -(6.5-8) wt.% Y_2O_3 condensate were evaluated with respect to the deposition rate (V_d). After deposition of TBC, all the samples were then submitted to cyclic test to evaluate ceramic spallation and crack growth. Each cycle consisted of a 10 min heating up to 1050 °C, an exposure at 1050 °C for 1.5 hour, followed by a 10 min cooling in air. The specimens were then finely polished using standard metallographic methods. The microstructure of the TBCs was investigated with scanning electron microscopy (SEM) equipped with energy dispersive X-ray spectrometry (EDS). Elemental concentration analysis was made by using EPMA to acquire precise, quantitative elemental analysis and elemental variation in the critical regions identified from SEM observations.

3. Results and Discussion

3.1. Initial microstructure

Cross-sectional image of SEM morphologies of the sample under the as-deposited condition with $V_d=1.8$ are shown in figure 2. Figure shows the uniform flat surface morphology and the overall cross section of the specimen with a 110 μm thick YSZ top coating.

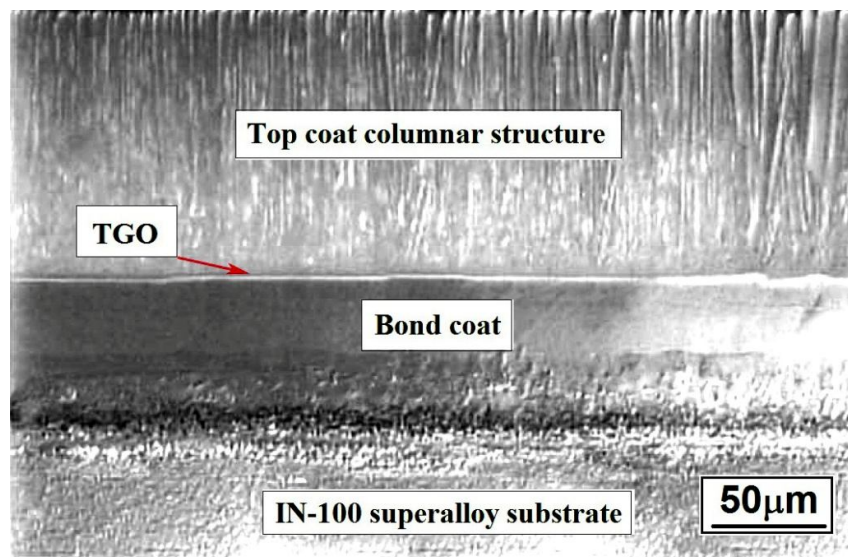


Figure 2. SEM cross-sectional image of IN-100 specimen coated with TBC exhibiting columnar structure.

The total thickness of TBC coating is about 150 μm with a bond coat of about 40 μm and a top ceramic coat of about 110 μm . The top ceramic coat exhibits typical columnar structure which is desirable owing to higher strain tolerance and dense structure.

3.2. Effect of deposition rate

A slight increase in V_d resulted in more dense layer of ZrO_2 formed on the bond coat-ceramic interface in the initial period of deposition, as well as a reduction of the total porosity of the ceramic layer. The formation of a denser ceramic layer with the increase of V_d is accompanied by the growth of the cross section of ZrO_2 columnar crystallites. The average diameter of the single crystallite is increased from 1.5-2 μm at $V_d = 1 \mu\text{m}$ per minute up to 3-4 μm at $V_d = 2 \mu\text{m}$ per minute. The preferred structural growth as observed and shown in Figure 1 with increasing V_d is also substantiated with the finding by previous researches (15-17).

3.3. Crack growth analysis as a function of thermal cycles

The cyclic temperature testing for ceramic spallation and crack growth study shows that the maximum thermal life occurs for ceramic top coat produced in the 1.2-2.0 μm per minute range when the micro porosity of the ceramic layer is 12-16%. Average crack length as a function of thermal cycles for the case of $V_d = 2 \mu\text{m}$ per minute is shown in Figure 3. It shows that the observed crack length increases approximately linearly with the number of thermal cycles, with slight slow rate in the thermal cycles between 30~50 showing crack arrest followed by accelerated crack growth in higher thermal cycles. Such behaviour is in compliance with the findings of fewer past studies (18, 19). Figure 4 shows the cross sectional image of the specimen coated with TBC after maximum cycles (80 cycles) considered in this study at 1050 $^{\circ}\text{C}$. From the figure, a micro crack can be observed in the top ceramic coat at the interface between the bond coat oxide layer and top ceramic coat. The micro cracks after initiation, propagate through the ceramic top coat perpendicular to the interface.

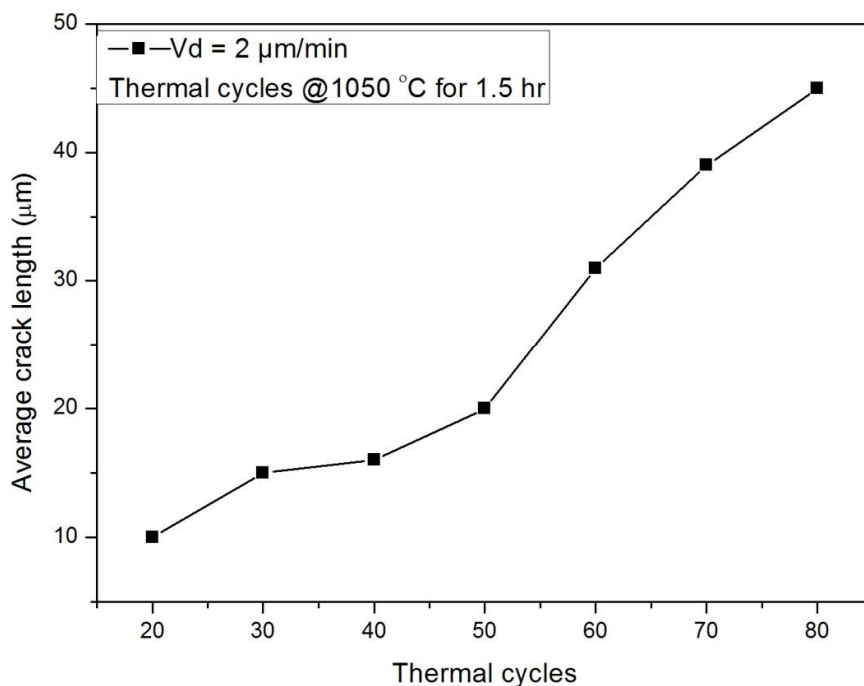


Figure 3. Average crack length as a function of thermal cycles.

Along a line normal to the interface, at about 8 μm from the crack, elemental concentration analysis was made by using EPMA. Figure 5 shows the elemental concentration profiles of Ni, Co, Cr and Al. Further analysis showed that just beneath the crack, Al element was found about 12wt. %, abnormally in higher content than the area nearby.

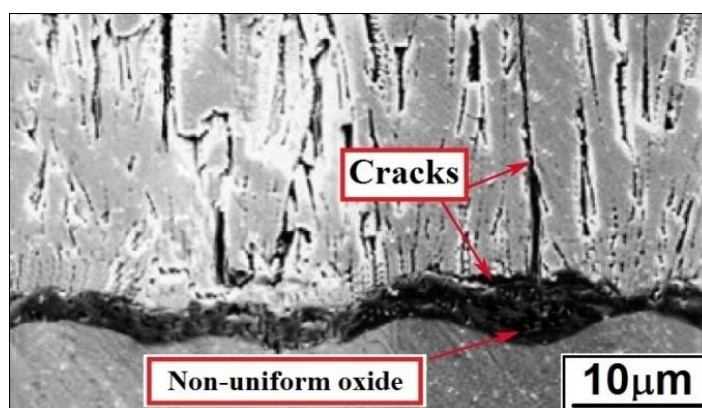


Figure 4. SEM cross-sectional image of thermally cycled IN-100 specimen coated TBC after 80 cycles at 1050 °C showing the crack.

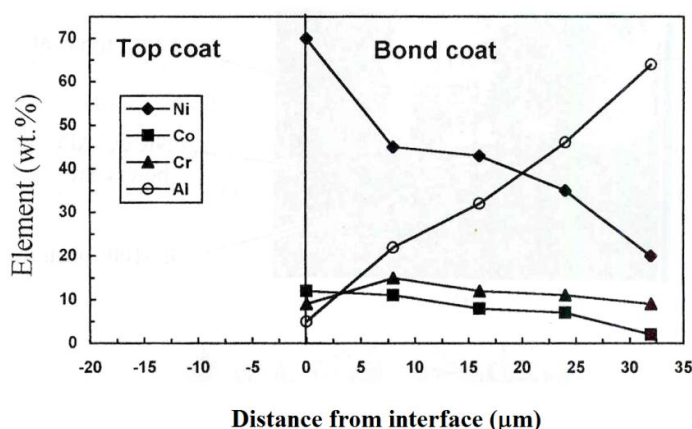


Figure 5. Elemental distribution perpendicular to the interface after thermal cycling of TBC coated specimen from 1050 °C.

3.4 Oxidation kinetics and damage mechanism

During high temperature oxidation, the oxygen from the atmosphere can easily diffuse to the metallic bond coat owing to the high porosity, segmentation and ionic conductivity of the zirconia top coat. The bond coat suffers with oxidation attack. An oxide layer builds up to the interface between the bond coat and the top coat and resulted volume expansion causes internal stress on the interface. Presence of higher Al content just beneath the crack indicates the formation of Al_2O_3 on the interface between the top coat and the bond coat. Formation of Cr_2O_3 and NiO on the interface, along with Al_2O_3 has also been reported elsewhere [13, 14, 20]. It is believed that the growth of Al_2O_3 on the interface is less detrimental to TBCs as compared to that of NiO , CoO or other oxides [14]. The formation of non-uniform oxides on the interface results into high growth stresses. First, the growth rate of Al_2O_3 is relatively slower than that of other oxides, e.g Cr_2O_3 , NiO and CoO [20]. Secondly, the PBR (Pilling-Bedworth ratio, volume of oxide : volume of metal) of Al_2O_3 is smaller than other oxides present on the interface (1.28 for Al_2O_3 and 1.65, 1.86, 2.07 for NiO , CoO , Cr_2O_3 respectively). Thus the growth stress of Al_2O_3 should be smaller as compared to that of non-uniform oxide scale [21]. Thirdly, growth of NiO or CoO occurs predominantly by cations diffusion through oxide scale and the

oxide growth stresses should be generated in the top coat-bond coat outgrown oxide scale interfacial region where spallation of the top coat initiated. However, for the growth of Al_2O_3 , oxide growth stresses are generated in the less critical Al_2O_3 bond coat interfacial region by inward diffusion of oxygen through Al_2O_3 scale [22]. Thus it is inferred that a non-uniform oxide formed on the surface of the bond coat leads to the generation of high growth stresses resulting in initiation of cracks in the top ceramic coat causing the failure of TBCs.

Several mechanisms of micro crack formation have been reported in the literature [23-25]. Movchan *et al.* [26] have suggested that in two layer metal / ceramic PVD coatings, the cracks initiate from the base metal and then spread out into the ceramic layer, finally causing the spallation of ceramic coat. Tsai *et al.* [27] found that the spallation of the top coat was initiated and propagated mainly by the cracks within the laser-glazed plasma sprayed top coat near the interface of the top coat /bond coat and same also verified by P. K. Wright. [28] Kh.G. Schmitt-Thomas *et al.* [13] and B.C. Wu *et al.* [21] have used the Al_2O_3 diffusion barrier layer between the bond coat and top ceramic coat to reduce the probabilities of initiation of cracks in the later.

From the observations noticed, cracks are likely first initiated from interface between the ceramic/oxidation layer (Figure 4) and then the non-uniform oxide layer forms between the bond coat and the top coat and the resulted volume expansion causes thermal stress at the interface. When the oxide layer has attained a critical thickness, cracks can first be observed. With increasing oxide thickness, the internal stress increases and the ceramic layer spalls off. According to the calculation, an oxide layer with a thickness of $3\mu\text{m}$ can produce about 12 MPa of oxidation stress. In case of formation of non-uniform oxides, stresses will be even much higher. Another factor is the thermal stress that can reach about 9.88 MPa. This is caused by the difference between the thermal expansion coefficients of the top and the bond coats when the specimens were cooled to between 700 and 600°C for stress measuring purpose. If the specimens would be cooled to room temperature for stress measuring purpose, a greater thermal stress can be expected.

Combined with the factors discussed above, the cracks in the TBCs produced by EB-PVD technique are first observed on the ceramic/bond coat interface owing to bond coat oxidation. These cracks then propagate along a direction normal to the interface in the ceramic layer. The growth of these cracks finally results in the spallation of top ceramic coat ultimately leading to the failure of the TBCs.

4. Conclusion

In this study the TBCs were prepared by EB-PVD method and the following observations about the formation of initial cracks have been made:

- a. In the thermally cycled TBCs, the cracks were observed to initiate from the interface between the ceramic top coat and the MCrAlY bond coat owing to the oxidation of bond coat. Cracks after formation, propagate in the ceramic coat in the direction normal to the interface. No cracks were observed in the bond coat.
- b. At a point just below the cracks, abnormal higher Al concentration was observed; it is believed that non-uniformed oxides formed at the interface caused the formation of micro cracks in the top ceramic coat.
- c. The techniques which can reduce the oxidation of metallic bond coat and avoid the formation of non-uniform oxides at the interface will be helpful to stop the initiation of cracks in the top ceramic coat and its spallation which ultimately causes the failure of the TBCs.

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