Materials Research Express

PAPER

Failure behavior study of EB-PVD TBCs under salt spray corrosion and thermal shock cycles

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Keywords: EB-PVD TBCs, salt spray corrosion, thermal shock, failure behavior

Abstract

Thermal barrier coatings (TBCs) are the most used methods to protect the internal hot end components of aeroengines for their improved thermal efficiency. Because of the severe service environment of TBCs, their spalling failure poses a problem. In recent years, studies on the erosion resistance of TBCs in marine atmosphere have attracted much attention. The study made an initial attempt in building a simulation experiment of aeroengine TBCs under marine atmospheric circumstances to explore the effect of thermal shock on the performance of TBCs under salt spray corrosion. The experimental results show that electron beam-physical vapor deposition (EB-PVD) Y2O3-stabilized ZrO2 (YSZ), one of the mainstream TBCs technologies, produced 20% of the surface spalling area after 300 thermal shock cycles under the simulated experimental conditions of salt spray corrosion and thermal shock. By testing and analyzing the failure samples and combining the results of SEM, EDS, and XRD, it was found that the ceramic layer and thermally grown oxide (TGO) are partly dissolved, thus dispersing coatings’ particles, but the phase structure does not change significantly, indicating the fine anticorrosion property of EB-PVD TBCs.

1. Introduction

As functional ceramics, the concept of thermal barrier coatings (TBCs) was first proposed by National Aeronautics and Space Administration (NASA). This type of ceramic involves spraying a protective coating with high-temperature resistance and high thermal insulation on the surface of superalloy substrates [1–3]. TBCs have high porosity and low thermal conductivity, which can reduce the surface temperature of alloy and improve the thermal efficiency of engine. Therefore, TBCs are frequently used in key technologies such as aeroengine technologies, and they provide the most practical method to improve the service temperature [1, 2]. Studies show that the thermal-mechanical-chemical coupling is the root cause of spalling failure of TBCs. Because of the complex failure mechanism of TBCs, conventional mechanical experimental methods such as tension, bending, and loading are unable to reflect the spalling failure of TBCs well. The development of an experimental method in simulating the service environment of TBCs turns out to be an inevitable way to study the reliability of TBCs [4].

Recently, much attention has been paid to the corrosion of TBCs in marine salt spray climate. Under the marine climate, the corrosion effect of sea salt and moisture is worse, 3–4 times higher than that of land climate [5]. The aeroengines in marine climate sustain a lasting harsh environment of high humidity, salt spray, and other chemicals. In addition to high-temperature erosion and normal loss, it is also affected by this type of corrosive substances. Moreover, in most of the time, the aeroengines are in the state of parking. Some studies show that the corrosion damage of aeroengines after parking for a long time is worse than that in the working
When the aeroengines are working, TBCs are under the action of thermal-mechanical-chemical coupling. The term 'thermal' indicates a high temperature of above 1000 °C. The term 'mechanical' indicates the effect of the high-speed flame flow with various impurities on the coatings, and the term 'chemical' indicates corrosive substances [7]. Under the action of thermal-mechanical-chemical coupling, the damage speed and degree of TBCs are much higher than those under the action of simply thermal-mechanical coupling. Therefore, it is necessary to study the marine atmospheric corrosion resistance of aeroengine TBCs [8–16].

In a marine climate, TBCs are exposed to a dynamic high temperature and salt spray corrosion environment when the engine is working, known as thermal corrosion in the literature [17]. Thermal corrosion is an important form of high-temperature corrosion, which is caused by the reaction of metallic materials with the salt deposited on their surface in a high-temperature environment. At present, two methods for thermal corrosion experiments are known: 'salt coating' and 'salt dipping' [18, 19]. In the 'salt coating' method, salt is deposited on the surface of the coatings at a certain temperature or other processes, whereas in the 'salt dipping' method, the coatings are soaked in a saline environment, such as a saline solution, at a certain temperature. The commonly used molten salts are NaCl, Na₂SO₄, K₂SO₄, and V₂O₅ [20, 21]. The temperature of thermal corrosion cannot reach the normal working temperature of the engine, which is only 800 °C–1000 °C [22]. The cycle time of thermal corrosion is 2–10 h [23]. Three types of thermal corrosion experiments have been reported [24]: The first type of experiment involves pure molten salt corrosion without a substrate, which is mainly for new ceramic coatings. The second type of experiment involves pure molten salt thermal corrosion with a substrate, which is mainly used for the most mature Y₂O₃-stabilized ZrO₂ (YSZ) and other composite coatings. The third type of experiment involves the simulation of thermal corrosion in the marine atmospheric environment. The common method involves salt spray corrosion at room temperature, followed by ablation with high-temperature gas, cycling repeatedly until failure [17, 22]. Most of the above experiments are based on the assessment of the effects of different coatings, processes, type of salt, temperature, and time on the thermal corrosion products of coating materials, while studies combined with mechanical failure analysis are relatively fewer. When an aeroengine works, the coatings are in a dynamic environment of high temperature, erosion, and corrosion [25]. Therefore, it is very important to perform thermal corrosion simulation experiments of TBCs under the actual working condition [16]. In this study, we performed the first experiment of this type and demonstrated its design and execution. In addition, the original experimental results and data analysis are provided.

2. Experimental process

2.1. Test rig of thermal shock
A high-temperature combustion chamber and Laval chamber nozzle were used to build an experimental simulation platform that can simulate service environments of high temperature and erosion of TBCs. During the test, the samples were fixed on the fixture, and a gas gun produced a high-temperature gas of up to 2000 °C through the combustion reaction of oxygen and aviation kerosene. The flame temperature and velocity were controlled and adjusted according to the flow rate of gas. The high-speed gas ejected by the gun is fed to the samples through a closed chamber, and the temperature of the whole front edge of samples was measured in real time using a long-wave infrared pyrometer, as shown in figure 1. The surface temperature of samples was controlled between 1100 and 1200 °C. The sum of heating time and holding time was controlled to 150 s. Then, the ignition was turned off, cooling the back of the samples with compressed air to maintain the thermal gradient, and the cooling time was about 150 s [26, 27]. The whole process is termed as a thermal shock cycle.

2.2. Sample preparation
Studies show that the thermal corrosion life of YSZ (7–8 wt. % Y₂O₃) produced by electron beam-physical vapor deposition (EB-PVD), the mainstream technology of TBCs, is twice as long as that produced by atmospheric
plasma spraying (APS) \[17, 28\]. Therefore, EB-PVD YSZ was used as a test sample in this study. At the same time, to simulate the actual working condition of TBCs, the same material GH536 nickel base superalloy was selected as the substrate and converted into a disc shape with a diameter of 30 mm and a thickness of 3 mm to reduce the mechanical stress at the edge. Then, a NiCrAlY bonding layer with a thickness of 80 μm and YSZ ceramic layer with a thickness of 150–170 μm were successively deposited on a sandblasted substrate using EB-PVD technology \[29\], as shown in figure 2.

2.3. Simulation of marine salt spray corrosion

According to statistics, aeroengines are grounded most of the time, almost all the year round in a static marine climate environment \[6, 19\]. The marine atmospheric environment is very complex and varies with the difference in the longitude and latitude of the earth and coastal geographical conditions. The main corrosion particles in marine atmosphere are Na\(^+\), Cl\(^-\), SO\(_4^{2-}\), NO\(_3^-\), and NH\(_4^+\). Plasma particles produced by the reaction of SO\(_2\), HNO\(_3\), and N\(_2\)O\(_5\) with NaCl \[30–32\]. The actual service condition of aeroengines is in the ‘cycle state’ from normal temperature to a high temperature \[33\].

To simulate the static deposition corrosion state of aeroengines in marine climate and shorten the test period, ‘salt dipping’ method was selected. According to the literature, several substances such as NaCl, NaNO\(_3\), NH\(_4\)Cl, and Na\(_2\)SO\(_4\) play a major role in salt spray corrosion \[33\]. The solutes of various components are weighed in terms of proportion, and the pH of the solution is 3 \[34\]. The chemical composition of salt spray solution is shown in table 1. The solution is rich in Na\(^+\).

2.4. Thermal shock test

Salt spray corrosion (soaked in a salt spray solution) for 10 h and then thermal shock test for 30 cycles, defined as a thermal corrosion cycle hereof \[35\], cycling until failure. During the test, the high-temperature infrared

| Table 1. Chemical composition of salt spray corrosion solution. |
|---------------------------------------------------------------|
| The chemical composition | NaCl | NaNO\(_3\) | NH\(_4\)Cl | Na\(_2\)SO\(_4\) |
| Value (mol l\(^{-1}\)) | 0.1 | 0.01 | 0.01 | 0.01 |

Figure 2. EB-PVD YSZ samples: (a) NiCrAlY bonding layer with a thickness of 80 μm, (b) YSZ coatings with a thickness of 150–170 μm.

Figure 3. Two working stations of thermal shock test (the left working station is used for corrosion YSZ, the right working station is used for original YSZ).
The infrared thermometer is aimed at the surface of the coatings, monitoring the surface temperature of the samples dynamically and timely. At the same time, a YSZ sample without corrosion is studied in another station, as shown in figure 3. When the spalling area of the coatings reaches 20% [36], the thermal shock test is terminated.

2.5. Real-time monitoring of the defeats

During a high-temperature erosion test, it is difficult to observe the damage, including crack appearance and location of the coatings. The results show that the thermal diffusivity of coatings increased due to sintering at a high temperature, while the thermal diffusivity decreased due to the failure such as cracks and spalling. Therefore, the change in surface morphology of coatings under high-temperature shock can be recorded as visualized in infrared thermal images [37]. When the test object is motivated, the defects of object lead to an uneven conduction of heat generated by the motivation, which can be observed from the infrared radiation of the surface [38]. The surface radiation heat of TBCs with a complete structure is uniform, while the surface radiation heat is uneven when the microstructure changes. Owing to the correlation between infrared thermal images and defects, the infrared detection technology is applicable in predicting the defect expansion trend of TBCs [39]. Therefore, an infrared thermal imager (GF309) was used in this study to monitor the changes in coating morphology.

During the experiment, the generation and propagation of defects of the coatings and cracks produce abnormal infrared thermal images, as shown in figure 4. Figure 4(a) shows the macromorphology image recorded using a CCD camera. Figure 4(b) shows the infrared thermal image recorded using an infrared thermal imager, and the right side shows the temperature scale. The infrared thermal image clearly shows the defect area of YSZ samples, and the temperature in the defect area is significantly higher than that in the surrounding normal area. Thus, by infrared imaging, it is convenient to determine the spalling of coatings in real time.
3. Results and discussion

3.1. Macromorphologies of YSZ samples

To make this experiment representative, three groups of repeated tests were carried out for YSZ samples in this study, and the average number of cycles was considered as the results. It can be concluded that the average life of salt spray corrosion YSZ samples is far fewer than that of original YSZ samples under the same experimental conditions. When the samples reached the failure criteria, the average number of thermal shock cycles of the corrosion YSZ sample is 300 compared with 470 of the original YSZ sample, as shown in Figure 5.

The sample morphologies of the two stations are shown in Figures 6 and 7.

Figure 6 shows that the surface morphology of corrosion YSZ sample slowly changes under the thermal shock at 1200 °C. A small amount of spalling points begin to appear at the edge of coatings after 135 cycles. With the increase in cycles, the spalling area of coatings continuously increases. Until 270 cycles, the spalling area of sample surface is close to 5%, and the thermal insulation effect of coatings starts to weaken significantly. Until 300 cycles, the spalling area of sample surface is above 20%, indicating that the coatings have failed. Compared with figure 7, small spalling points began to appear on the surface of original YSZ sample after nearly 200 thermal shock cycles, and the spalling area of sample surface was less than 5% after 300 thermal shock cycles.

It is preliminarily determined that the failure of YSZ under thermal shock and salt spray corrosion is faster. The spalling position of original YSZ sample under thermal shock mainly starts from the edge of the sample as a result of thermal fatigue damage [36, 40]. However, corrosion YSZ under thermal shock is different; in addition to the above reasons, the penetration and corrosion of salt spray to the ceramic layer and bonding layer accelerate the failure of coatings.

3.2. SEM test of YSZ samples surface

While conducting the thermal shock and thermal corrosion tests, a group of samples was subjected to pure corrosion test. When the thermal corrosion sample failed, the corrosion time is 100 h, and the number of thermal shock cycles is 300.
For comparing the failure of YSZ samples under different working conditions, the samples were divided into four types: original YSZ, corrosion YSZ, thermal shock YSZ, and thermal corrosion YSZ. To distinguish the samples, the four types of samples are numbered, as shown in table 2.

To understand the failure mechanism of TBCs, the backscattered electron mode of SEM was used to study the microstructural degradation of TBCs. The samples were divided into small samples with a size of 10 mm × 4 mm × 3 mm using a precision cutting machine. The SEM morphologies of original YSZ sample (1#) and corrosion YSZ sample (2#) are compared in figure 8.

The surface of original YSZ sample (1#) shows columnar crystals with a 'clustered' shape, and gullies are present between the clusters. This is mainly because the coatings gradually shrink due to cooling during the manufacturing, resulting in tensile stress [38]. However, the surface morphology of corrosion YSZ sample (2#)}
changed significantly, and the columnar crystals became more compact, from 'clustered' to 'stacked,' which can be confirmed by the SEM cross-section morphologies shown in figure 10.

The SEM morphologies of original YSZ sample and corrosion YSZ sample after 300 thermal shock cycles (which are defined as thermal shock YSZ (3#) and thermal corrosion YSZ (4#) respectively) are compared in figure 9.

Figure 9 shows that the surface morphologies of thermal shock YSZ sample (3#) and thermal corrosion YSZ sample (4#) are different. Under corrosion and thermal shock cycles, the damage in thermal corrosion YSZ (4#) is much greater than that in thermal shock YSZ (3#). Thermal corrosion YSZ (4#) produced a large area of spalling, while thermal shock YSZ (3#) produced only a small area of spalling at the edge. Without corrosion, the damage of thermal shock YSZ (3#) is mainly caused by the sintering of coatings caused by thermal shock, and the spalling of coatings is gradually transitional from the surface. Thermal corrosion YSZ (4) has a large area of spalling and obvious 'gully' cracks, also known as interfacial cracks [41], making the spalling of ceramic layer along the cracks.

3.3. SEM test of YSZ samples cross-section

To better understand the micromorphology of coatings, the cross-section of samples was analyzed by performing SEM tests. The results are shown in figure 10.

The three-layer structure of TBCs can be observed from figure 10: Nickel base superalloy at the bottom, NiCrAlY metal bonding layer at the middle, and YSZ ceramic layer at the top [27]. By comparing figures 8 and 9 with figure 10, it was found that the surface morphology and cross-section morphology of the coatings match well.

Figure 10 shows that the columnar crystal edge has a feathery structure, and its growth and distribution are uniform [42]. The average thickness of ceramic layer is about 163 μm, and the existence of gaps between the columnar crystals can provide a channel for O₂, erosion flame flow, and corrosion substances. After being eroded by salt spray, the change in coatings is clearly observed. The gap between columnar crystals is no longer clear, and the feathery structure disappears. The columnar crystal became inclined and showed 'stacked.' The average thickness of ceramic layer after corrosion is about 155 μm. The bonding layer is also eroded, and its thickness is no longer uniform.

Under the action of high temperature, the interface between ceramic layer and bonding layer can form an oxide protective film of Al and Cr, namely, thermally grown oxide (TGO) [43]. After thermal shock cycles, the gap between columnar crystals is more obvious than before, which can be attributed to the sintering of ceramic layer and TGO growth. When TGO grows to a certain thickness, it will produce stress and lead to interfacial cracks [13]. The interfacial cracks will gradually extend to the coating surface, causing a partial fracture of columnar crystals or total fracture from the bonding layer. Under the joint action of TGO and sintering, the ceramic layer will be peeled off [41].
However, the morphology of thermal corrosion YSZ sample (4#) is different. The columnar crystals are almost bonded together, and the fracture of columnar crystals is more clearly observed. Pores are observed near the position of TGO, i.e., the coatings are dissolved under the erosion of salt spray and high temperature.

Based on the literature, three main reasons are observed for the damage of coatings: first, the dissolution of YSZ leads to the loosening of particles in the ceramic layer. Second, the dissolution of YSZ leads to stress between the columnar crystals and the formation of cracks [44]. Third, the dissolution of TGO loosens the parts present between the ceramic layer and bonding layer.

3.4. EDS analysis
The adhesion between crystals and coatings is very poor, and it is prone to be peeled off, thus destroying the integrity of oxide protective film. The generation of pores in the coatings is an important phenomenon in this experiment, and their composition can be analyzed by EDS, as shown in figure 11. The ceramic layer and bonding layer are separated, and the pores are located just at the junction of the ceramic layer and bonding layer. Zr, Cr, Al, O elements and trace Na accumulated beneath the pore, indicating that the ceramic layer and bonding layer are dissolved under the joint action of salt spray corrosion and thermal shock [45]. During thermal corrosion, TGO can effectively prevent the diffusion of corrosion substances to the bonding layer and lower the corrosion rate [35]. However, with the continuous erosion of corrosion substances, the oxide protective layer is dissolved. According to the literature, the corrosive substances on the coatings will generate Na₂O at a high temperature, and TGO will react with Na₂O and form Na₂CrO₄ and NaAlO₂ [44]. When the concentration of metal sodium salt reaches a certain degree, it will be saturated, precipitated, and crystallized at the interface. After the failure of TGO, the metals in the bonding layer will diffuse to the surface, and defects will appear in the bonding layer. At the same time, YSZ is also dissolved, forming Na, Zr, and O compounds as reported in the literature [44]. The dissolution disperses the particles of the coatings, and these defects aggregate to form pores. The pores cause great damage to the stable structure of coatings. The positions and contents of Zr, Cr, Al, O, and Na in the energy spectrum are consistent with those reported in the literature [44, 46].

3.5. XRD analysis
The XRD test results for the four types of samples are shown in figure 12. The YSZ coatings, mainly t'-phase, have a Y₂O₃ partially stabilized ZrO₂ structure [47, 48] with a preferred orientation at (200), which is a standard EB-PVD structure [49]. After salt spray corrosion, the diffraction intensity of (002) crystal plane increased because the preferred orientation of coatings is partially destroyed. At the same time, a small amount of NaCl phase is also observed on the surface. After thermal shock, the diffraction peak intensity of (111) surface of
coatings increased, indicating that the preferred orientation of t'-phase YSZ in the coatings is partially destroyed due to thermal-mechanical coupling [50, 51].

The XRD analysis of thermal corrosion YSZ (4#) shows that Na0.08Zr0.92O1.88 is formed, and the intensity of diffraction peak of thermal corrosion YSZ sample (4#) is weaker than that of original YSZ sample (1#). The higher the crystallinity, the higher the intensity, and the sharper the peak shape. The decrease in diffraction peak intensity indicates that the crystallinity of YSZ sample decreases because of the destruction of YSZ columnar crystals and formation of some products under the action of corrosion and thermal shock [44], consistent with the previous phenomenon and analysis.

4. Conclusions

In this study, an experiment was conducted for simulating aeroengine working condition in marine atmosphere to evaluate the corrosion resistance of TBCs prepared by EB-PVD. The following conclusions are drawn:

1. As the TBCs prepared by EB-PVD method have a columnar crystal structure, the corrosive substance can infiltrate along the columnar crystal gaps. The corrosion of salt spray on the coatings is not clearly observed in a short time; however, lasting exposure can cause corrosion and damage to the ceramic layer and bonding layer, resulting in the peeling and failure of coatings.

2. Under only thermal shock, the peeling of coatings is caused by the change in stress and composition structure caused by the growth of TGO and sintering of coatings. By combining thermal shock and salt spray corrosion, YSZ is damaged faster and more severely; unprotected oxides are formed in the ceramic layer, between the ceramic layer and bonding layer [52], resulting in the degeneration and failure of YSZ.

3. YSZ is mainly composed of t'-ZrO2. Under the action of corrosion and high-temperature shock, the diffraction peaks do not change obviously, i.e., the phase structure of YSZ does not change obviously, and the EB-PVD TBCs have good thermal-mechanical-chemical stability.
Acknowledgments

The author gratefully acknowledges the invaluable support from Xie Ying, Tang Taotao, Ma Dupei, Yan Jingjun, Wang Qiang, Liu Guolin, Zhou Jie, Chen Huanyu and Zheng Bangkun in the test. This work was supported by the National Natural Science Foundation of China (Grant Nos. 51971189) and Zhuzhou Joint Fund of Hunan Natural Science Foundation of China (Grant Nos. 2019J60011).

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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