Evaluation of hot corrosion of 8YSZ coatings elaborated by suspension plasma spraying

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Abstract. In this paper, the evaluation microscopic of hot corrosion of 8mol% Yttria Stabilized Zirconia (8YSZ) coatings was studied in the presence of V₂O₅ and Na₂SO₄ as corrosive molten salt, for 40h at 1050 ºC. First, the substrates of Inconel 718 super-alloy were sprayed with a NiCrCoAl-Y₂O₃ bond coat by atmospheric plasma spraying (APS). Then this bond coat was polished for elaborated the 8YSZ layer by suspension plasma spraying (SPS). The microstructure of the cross-section and surface of the coating was evaluated by scanning electron microscopy (SEM). After the hot corrosion test, the delamination of 8YSZ coatings was occurred in the ceramic layer due to the creation of stress resulting from the chemical reaction between the molten salts and the yttria (Y₂O₃) of 8YSZ coating at high temperature. According to EDS-SEM analysis, the evaluation of fractured sections of 8YSZ coating showed mainly the formation of crystals composed by Y, V, O and the surface was mainly composed by Zr and O. Those crystals can be related with the tetragonal phase of YVO₄, which they were commonly found by other researchers in studies of hot corrosion of YSZ-based TBCs when its surface reacts with the corrosive salts.

1. Introduction

Thermal barrier coatings (TBCs) systems are widely used to provide thermal and corrosion protections for the metallic hot-section components of gas turbines to achieve extended your durability [1–5]. A typical TBC system is composed by MCrAlY (M=Ni and/or Co) metallic bond-coat as an oxidation resistant layer and the yttria stabilized zirconia (YSZ) topcoat as a thermal insulation layer [1,4,6]. One of the most crucial bond coat properties is to improve the adherence between the metallic component (Ni-based superalloys) and ceramic coating, as well as the resistance to high-temperature oxidation and corrosion of TBC system [1,3]. Yttria stabilized zirconia (YSZ) material has been generally chosen for the ceramic topcoat, due to its low thermal conductivity and high thermal expansion coefficient, with the latter being approximately equal to that of the substrate [3]. TBCs are usually applied either by an atmospheric plasma spray (APS) or electron beam physical vapour deposition (EB-PVD). However, plasma spraying technology has enjoyed widespread acceptance thanks to the high cost-effective deposition conditions and deposition efficiency [1,2]. The other hand, the typical gas turbine operating conditions use of low quality fuels containing impurities such as Na, V and S, which it can lead to premature degradation of TBCs by hot corrosion [7].

Suspension plasma spraying (SPS), as a newly emerged technique of thermal spraying processes, has been intensity investigated to elaborated coatings (TBCs) with enhanced characteristics through submicron or nanometer particles [8–10]. The suspensions contain fine particles that are dispersed in a
liquid phase by mechanical or chemical methods. Then the suspension with particles is injected into the plasma through a liquid feedstock system as a liquid stream. Some particles with sizes less than 1µm are easily accelerated and then decelerated in the plasma jet, which requires a shorter standoff distance between the plasma torch exit and the substrate (≈9-20cm) than the conventional processes (≈3-7cm) [8]. Therefore, the substrate experiences a greater heat flux due to the shorter standoff distance. To achieve a desirable coating microstructure, injector properties and spray parameters must be accurately optimized in this SPS process [8]. In addition, previous works on TBCs elaborated by SPS have focused mainly on coating formation, thermal properties, thermal cycling, and isothermal oxidation [7]. 3, that SPS coating offers higher cycling durability that coatings elaborated by APS [7]. However, the hot corrosion behaviour of YSZ coatings elaborated by SPS has not yet been sufficiently studied.

The objective of this work is to evaluate the hot corrosion behaviour of 8mol% YSZ coating elaborated by SPS. The NiCrCoAl-Y2O3 bond coats were first deposited on a Inconel 718 superalloy substrate using APS process. Then the YSZ top coat was applied on the polished bond coat surface using the SPS technique. Finally, hot corrosion test was carried out in presence of V2O5 and Na2SO4 for 40h at 1050ºC.

2. Materials and method

Ni-based supper-alloy (Inconel 718) substrates in shape of disk with the dimension of 25mm in diameter and thickness of 5mm were grit blasted with alumina particles to create a desired surface roughness Rₐ profile of 4-5µm.

Atmospheric plasma spraying (APS) was used to elaborated NiCrAlCo–Y2O3 composition (Metco 461NS) bond coat of 250µm thickness using F4-MB torch (Sulzer-Metco, Wolhen, Suiza) with Multicoat system. Thereafter, the YSZ ceramic layer was applied over the polished bond coat through the SPS technique. SPS coating was elaborated using the same torch and Multicoat system with which was equipped with a liquid delivery unit developed by University of Limoges (France). 8mol% YSZ powder with d₅₀ of 360nm (d₁₀=230nm and d₉₀=560nm) supplied by UCM Advanced Ceramics was used to achieve the aqueous suspensions. The solid loading of the suspension was fixed at 20wt.% according to published works by researchers at the Université de Limoges (France) [11 –15] in which this value was found to be a good compromise between deposition efficiency and required plasma output. The Beycostat C213 with an amount of 2.1wt.% of YSZ powder was used for dispersing the particles in ethanol. Spray parameters for the APS and SPS coatings are shown in Table 1.

| Parameters                          | NiCrCoAl-Y2O3 | 8mol% YSZ |
|-------------------------------------|---------------|-----------|
| Spray distance (mm)                 | 100           | 30        |
| Intensity current (A)               | 500           | 500       |
| Internal diameter of the F4 torch (mm) | 7             | 7         |
| Plasma gas for APS, Ar-H2 flow rate (l/min) | 45-15        | –         |
| Plasma gas for SPS, Ar-He flow rate (l/min) | –             | 40-20     |
| APS powder feed rate (g/min)        | 25-30         | –         |
| SPS suspension feed rate (ml/min)   | –             | 24        |
| Inner diameter of the suspension injector (µm) | –             | 150       |

Morphological characterization of YSZ powder was carried out by field emission scanning electron microscopy (FE-SEM) microscope (JEOL 7400F equipment). The cross-section of 8YSZ coating was polished according the standard ASTM E1920-03 and was analysed by scanning electron microscopy (SEM) microscope (JEOL JSM-6490LV equipment). In order to evaluate the corrosion behaviour of the coating, 55wt.% V₂O₅ (Alfa-Aesar, 99.6% min) and 45wt.% Na₂SO₄ (Carlo Erba, Ref. 4330007) powders was selected as a corrosive salt. The corrosive salt was strewed over the surface of the coatings in a 25-30mg/cm² concentration, leaving approximately 3mm from the edge uncoated to
avoid the edge effect. The samples were pushed into the muffle furnace with air atmosphere at 1050°C for 40 hours and then cooled down inside the furnace. An accelerated hot corrosion test was carried out in this study as compared with those in the gas turbine applications; this study is somewhat comparable with similar investigations [10,16,17]. The surface of coating exposed to hot corrosion was analysed by EDS-SEM technique.

3. Results

The morphology of 8mol% YSZ powder is shown in Figure 1. This material presents particles with an irregular and angular shape that are highly correlated with its manufacturing method, which was by high energy milling process. The morphology of the cross-section of 8mol% YSZ coating was observed by SEM, as shown in Figure 2. This morphology is composed by a bimodal structure made of un-molten nanosized particles imbedded in a matrix of molten material, which is a typical characteristic of this kind of coatings [18]. This bimodal structure develops because after the liquid is evaporated in the plasma and the resulting particles or agglomerates may thus be partly melted or melted, yielding the end coating [19]. The thickness of SPS coating was 90±4µm.

![Figure 1. FE-SEM image of 8YSZ powder used to prepare the suspension.](image1)

![Figure 2. SEM images of polished cross-section of the 8YSZ coating.](image2)

The macroscopic image of the surface coating sample after 40h exposure to hot corrosion is shown in Figure 3(a). As observed, YSZ coating was delaminated, which occurred when the sample was cooled down inside the furnace until room temperature after 40h of the corrosion test. In other words, the coating was mainly detached by effect of molten salts, thermal stress and differential expansion between the ceramic coating and the bond coat. This depletion is similar to that found in the other work with similar conditions [7]. Figure 3(b) shows the SEM micrograph of a fractured section of YSZ coating after the hot corrosion test. As clearly seen, the rod-type crystals have formed on the coating surface. The EDS analysis of these points show that the coating surface (EDS 1) contains mainly Zr, O and with small amounts of Ni, Cr, V (Figure 3(c)) as well as the rod-type crystals (EDS 2) contain Y, V and O (Figure 3(d)). This rod-type crystal was formed as a result of the reaction of yttria of YSZ coating with the molten salt at elevated temperature.

Furthermore, the porosity and the reactive surface of coating allows the possibility of the penetration of the corrosion salt into the structure of the YSZ layer. Thus, salt can also react with the stabilizer of YSZ layer and cause the destruction and delamination of zirconia coating via thermal stress arising from the formation of rod-crystals and transformation of tetragonal to monoclinic zirconia. This behaviour is similar to that found by other researchers, especially in APS coatings [1,3,7]. In order to understand hot corrosion degradation mechanism of YSZ coatings under molten salts, it's essential to analyse the possible reactions that occur at high temperature. The appearance of
rod-crystals of Y-V-O elements found in EDS 2 (Figure 3) can be explained by the reactions presented in the Equation 1 and 2. This reactions, were also proposed by other researchers [1,3].

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\begin{align*}
V_2O_5(1) + Na_2SO_4(1) & \rightarrow 2NaVO_3(1) + SO_3(g) \\
YSZ(s) + 2NaVO_3(1) & \rightarrow ZrO_2(s)\text{(monoclinic)} + YVO_4(s) + Na_2O(1)
\end{align*}
\]  

At first, V_2O_5 could be first melting during heating because of its lower melting point (690°C). At \(\approx 610°C\), the liquid NaVO_3 could be formed by the reaction of molten salt (Na_2SO_4+V_2O_5) at the testing temperature of 1050°C, according to Equation 1. Finally, NaVO_3 reacted with the stabilizer Y_2O_3 of YSZ to form monoclinic ZrO_2, rod-crystals of YVO_4 and Na_2O (see Equation 2). In this study, Na was not detected in EDS 1. It is possible that Na_2O was sublimated at high temperatures during hot corrosion test.

4. Conclusion

In this work, the evaluation microscopic of the hot corrosion behaviour of 8mol% YSZ coating elaborated by suspension plasma spraying technique was investigated. The hot corrosion test was carried out using a mixture of V_2O_5 and Na_2SO_4 for 40h at 1050°C. The YSZ coating showed a bimodal distribution structure, which was composed of a dense area with semimolten nanometric particles and a lamellar zone with a higher degree melting. This bimodal structure is typical in coating elaborated by SPS.

The mechanism of hot corrosion of YSZ coating elaborated by SPS is similar to the coating deposited by APS. This mechanism consist of the infiltration of molten salt into the YSZ coating elaborated by SPS. Then, the molten salt cause formation of monoclinic-ZrO_2 and YVO_4 phases by interaction between YSZ layer and molten salt. The formation of monoclinic-ZrO_2 induces cracks in the zirconia phase of the top coat leading to the failure and delamination of YSZ layer in this study, especially when the coating was cooling after hot corrosion test.

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