



# Oxidation and hot corrosion behavior of Al<sub>2</sub>O<sub>3</sub>/YSZ coatings prepared by cathode plasma electrolytic deposition



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## ABSTRACT

Al<sub>2</sub>O<sub>3</sub>/YSZ double layer TBCs dispersed with Pt particles were prepared by cathode plasma electrolytic deposition (CPED) on the high temperature alloy (0.1% C, 12% Co, 6.5% Cr, 6.2% Al, 5% W, 1% Mo, 1.5% Hf, 6.5% Ta, 0.01% B, balance Ni, wt%). High temperature cyclic oxidation at 1100 °C, hot corrosion at 900 °C and thermal insulation tests at 1100 °C were adopted to investigate the high temperature corrosion resistance of the coating. It is revealed that the mechanical property of composite coating is improved by the toughening effects of Pt particles. The Al<sub>2</sub>O<sub>3</sub>/YSZ double layer coatings dispersed with Pt possess well oxidation resistance and hot corrosion resistance, due to low oxygen diffusion rate and chemically inertness of the Al<sub>2</sub>O<sub>3</sub> layer.

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## 1. Introduction

Thermal barrier coatings (TBCs) have been extensively used in hot section components of gas turbines to increase turbine efficiency by elevating the operational temperature [1–3]. The application of the TBCs enables the engines to be operated at higher gas inlet temperature, giving rise to the improvement of the thrust-to-weight ratio and fuel efficiency of the engines [4–6]. A typical TBC system consists of a metallic bond coat and a ceramic top coat. The bond coat (usually MCrAlY, M=Ni and Co) protects the substrates from oxidation and improves the adhesion of the ceramic top coat to the metallic substrate [7]. YSZ ceramic has been used as top coat materials for decades due to low thermal conductivity, high phase stability, high thermal-expansion coefficient and high fracture toughness as compared to other ceramics. However there are shortcomings such as hot corrosion and spallation which reduce the durability of the TBCs. The element of Na, V and S can lead to premature degradation of TBCs by hot corrosion in typical gas turbine operating conditions. Molten salts (especially chlorides) lead to the formation of plentiful non-protective oxides on the bond coat surface resulting in failure of TBCs [8–14]. In addition, Coatings peel off from the substrate easily at high temperature, due to the thermal expansion mismatch.

Because of good physical and chemical properties of Al<sub>2</sub>O<sub>3</sub>, it has been considered as the most important ceramic material. Al<sub>2</sub>O<sub>3</sub> possesses high mechanical strength, chemical neutrality, low rate of corrosion in most environments and extremely low oxygen diffusion rate [15]. So Al<sub>2</sub>O<sub>3</sub> layer over the YSZ coating could prevent hot corrosion [16,17] and significantly reduce the infiltration of oxygen into the YSZ at the high temperature [18]. In addition, it is reported that the high-temperature spallation resistance of the coating has been improved significantly owing to the toughening effects of noble metal particles [19].

There are two main types of TBC manufacturing routes: air plasma spraying (APS) [20,21] and electron beam physical vapour deposition (EB-PVD) [22,23]. However, the co-deposition of Pt particles and coatings are achieved difficultly by APS and EB-PVD. Recently, our group has found that the Pt particles can be dispersed in ceramic coating easily by cathode plasma electrolytic deposition (CPED) technique, and the fracture toughness of ceramic coating is improved significantly [24,25].

In this study, Al<sub>2</sub>O<sub>3</sub>/YSZ double layer TBCs containing dispersed Pt particles were prepared by CPED on the high temperature alloy with bond coat. It is expected that such TBCs would possess well oxidation and hot corrosion resistance because of extremely low oxygen diffusion rate and good chemical stability of Al<sub>2</sub>O<sub>3</sub> layer, provide good thermal insulation because of low thermal conductivity of YSZ, and exhibit excellent mechanical properties because of the toughening effect of the Pt particles. We expect the results of

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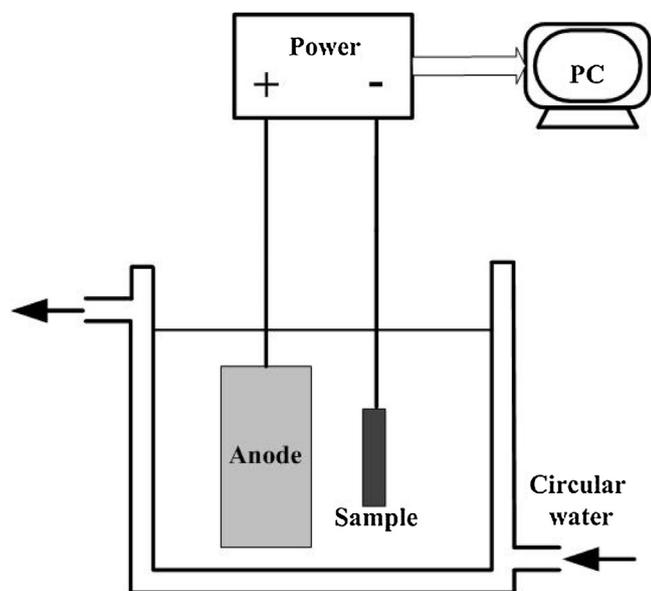


Fig. 1. The schematic view of the CPED device.

Table 1

The composition of the solution and CPED parameters to prepare  $\text{Al}_2\text{O}_3/\text{Y}_2\text{O}_3$  coatings.

Parameters	YSZ	YSZ-Pt	$\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$ -Pt
$\text{Y}(\text{NO}_3)_3$	0.2 mol/L	0.2 mol/L		
$\text{Zr}(\text{NO}_3)_4$	1.8 mol/L	1.8 mol/L		
$\text{Al}(\text{NO}_3)_3$			0.8 mol/L	0.8 mol/L
$\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$		0.2 g/L		0.2 g/L
PEG			20 g/L	20 g/L
Voltage	135 V	135 V	140 V	140 V
Duty	60%	60%	60%	60%
Frequency	500 Hz	500 Hz	500 Hz	500 Hz
Time	30 min	30 min	10 min	10 min

this study to promote the development of TBCs with novel structures and new manufacturing route.

## 2. Experimental procedure

### 2.1. Coating preparation

The schematic view of CPED device for preparing  $\text{Al}_2\text{O}_3/\text{YSZ}$  coatings is shown in Fig. 1. A platinum electrode was worked as anode with a dimension of  $120\text{ mm} \times 50\text{ mm} \times 0.3\text{ mm}$ . The samples of IC10 alloys (0.1% C, 12% Co, 6.5% Cr, 6.2% Al, 5% W, 1% Mo, 1.5% Hf, 6.5% Ta, 0.01% B, balance Ni, wt.%) were used as cathode  $15\text{ mm} \times 10\text{ mm} \times 2\text{ mm}$ . The bond coat of MCrAlY was deposited onto the samples with plasma spraying method. A pulsed electrical power supply (TN-KGZ01) was connected in the electrolytic bath. The YSZ and YSZ-Pt layers as TBCs were firstly prepared on the samples with bond coat, then the  $\text{Al}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ -Pt layer as top coats were prepared over the YSZ and YSZ-Pt coatings respectively. The compositions of the aqueous solution and CPED parameters to prepare ceramic coatings are listed in Table 1.

### 2.2. Coating characterization

The morphologies of  $\text{Al}_2\text{O}_3$ -Pt/YSZ-Pt coatings were investigated by scanning electron microscope (SEM, JMS-6480A) with an energy-dispersive spectroscopy (EDS) system. The phase structures were detected by X-ray diffraction analysis (XRD, PW 3710, Philips)

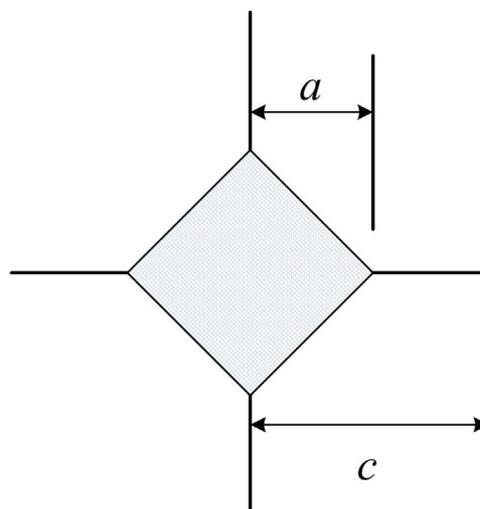


Fig. 2. Schematic of Vickers indentation setup showing characteristic dimension of crack length ( $c$ ).

at room temperature using nickel filtered  $\text{Cu K}\alpha$  radiation in the  $2\theta$  range of  $20$ – $90^\circ$  with a step size of  $0.02^\circ$ .

Hardness and elastic modulus of oxide coatings were detected by MTS NanoIndenter XP indentation tests by driving a diamond indenter into the coating surface, and the depth was kept as  $2000\text{ nm}$ . The Vickers indentation testing (Wolpert Wilson Instruments, 401MVD) was used to measure the indentation parameters of half crack length ( $c$ ) as shown in Fig. 2.

High temperature cyclic oxidation tests were carried out to investigate the kinetics of oxidation and spallation resistance of coatings in air furnace at  $1100^\circ\text{C}$  for  $200\text{ h}$ . Before the oxidation test, the mass of sample + crucible ( $W_0$ ) and the mass of crucible ( $W'_0$ ) were weighed by the analytical balance (BT 25S, Sartorius) with sensitivity of  $10^{-5}\text{ g}$ , respectively. Then the sample + crucible were put into the furnace, and taken out after  $10\text{ h}$ . After air cooling to the room temperature, the spalling oxide could be found in the crucible, the mass of sample + spalling oxide + crucible ( $W_1$ ) and the mass of spalling oxide + crucible ( $W'_1$ ) were weighed by the analytical balance again. The weight gain was equal to  $W_1 - W_0$ , and the spallation was equal to  $W'_1 - W'_0$ . Then this cycle was repeated  $20$  times.

The main compounds of corrosive salt in hot corrosion tests were mixed in weight ratio of  $55\text{ wt.}\% \text{V}_2\text{O}_5$  and  $45\text{ wt.}\% \text{Na}_2\text{SO}_4$ . The salt was spread over the surface of coatings in a  $3\text{ mg/cm}^2$  concentration leaving approximately  $2\text{ mm}$  from the edge to avoid edge effect. The samples were set in an electric furnace with air atmosphere at  $900^\circ\text{C}$  for  $200\text{ h}$ . At regular interval ( $10\text{ h}$ ), the samples were taken out, cooled down to room temperature and weighed by the analytical balance with sensitivity of  $10^{-5}\text{ g}$ .

The thermal insulation capability tests were carried out in the apparatus shown in Fig. 3. Two k-type thermocouples were used as sensors to record the temperatures on the coating surface ( $T_1$ ) and on the uncoated alloy surface ( $T_2$ ). Ar gas flowing at  $5\text{ L/min}$  was used to cool the alloy substrate. The thermal insulation capability was evaluated on the basis of the temperature drop,  $\Delta T = T_1 - T_2$ .

## 3. Results

### 3.1. Morphologies of the prepared coatings

Fig. 4 (the secondary electron SEM images) shows the surface and cross-sectional morphologies of YSZ and YSZ-Pt coatings prepared by CPED. It can be seen from Fig. 4( $a_1$  and  $b_1$ ) that the cracks

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