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Microstructure of nanoporous yttria-stabilized zirconia films fabricated by EB-PVD

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Abstract

Microstructures of ZrO_2–4 mol% Y_2O_3 coating layers fabricated by EB-PVD are characterized. Coating layers are found to consist of porous columnar grains with a feather-like structure containing evenly dispersed nano-sized gaps. Nanopores less than 50 nm in diameter could also be observed within columnar grains. The columnar grain size and column width of the coating layers increased with increasing coating thickness and substrate rotation speed. The porosity of the coating layers also shows increases with increasing substrate rotation speed. Specimens rotated at 1 rpm are found to contain wavy or scalloped columnar grains. Specimens rotated at higher speeds are found to consist of straight columns with a banded structure.

Keyword: Electron beam physical vapor deposition (EB-PVD); Porosity; ZrO_2; Films

1. Introduction

Thermal barrier coatings (TBCs) of high melting point oxides on superalloy turbine parts have been used in gas turbines for some time now, leading to enhanced efficiency and performance of these devices.[1–5] A typical oxide for TBCs is partially stabilized zirconia containing 4 mol% yttria (4YSZ) because of its low density, low thermal conductivity, high melting point and good thermal shock resistance, i.e., its excellent erosion resistance properties.[6–8] Such coatings have generally been applied by plasma spraying or physical vapor deposition above a bond layer coated directly onto the turbine blade. This configuration improves the thermal efficiency of the turbine system because the low thermal conductivity of coating films allows the component to be used at higher temperatures.[9–11]

Recently, TBCs manufactured by electron beam physical vapor deposition (EB-PVD) have been favored because their unique microstructure offers the advantage of superior tolerance to mechanical strain and thermal shock at the high temperatures at which gas turbines are operated.[12–16] TBCs applied using the EB-PVD process have further advantages over the plasma spray process such as better erosion resistance, bonding strength and surface roughness of the coatings. However, the thermal conductivity of EB-PVD coatings is relatively high.

To improve the thermal performance of EB-PVD TBCs, detailed microstructural characterization of coating films is necessary because thermal properties depend strongly on microstructure. One of the advantages of using the EB-PVD technique to deposit oxide ceramics is that it is possible to vary the microstructure of the coating by controlling deposition process parameters such as coating chamber pressure, deposition rate, vapor incident angle (VIA), vapor incidence pattern (VIP) and substrate temperature. Several studies on the microstructures of YSZ films have been reported.[17–23] However, the influence of coating thickness or rotation speed on the microstructure of EB-PVD YSZ coating films has not been sufficiently explained in the literature.

The purpose of the present work is to characterize the influence of coating thickness and substrate rotation speed

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on the microstructure of YSZ coatings deposited by EB-PVD on zirconia substrates.

2. Experimental procedure

Coatings were deposited by EB-PVD using commercially available 4 mol% Y_2O_3-stabilized zirconia targets. Substrates of ZrO_2–4 mol% Y_2O_3 were prepared by pressureless sintering at 1600 °C in the shape of discs of 10 mm diameter and 2 mm thickness. No bond coat between top coats and substrates was applied. Before deposition of the top coats, the substrates were ultrasonically cleaned in acetone and then isopropyl alcohol. After drying, substrates were inserted into a special holder assembly and placed into a vacuum system.

The substrates were preheated at 900–1000 °C in a preheating chamber using graphite heating elements. The substrates were then moved to the coating chamber for deposition.

The substrates were mounted on revolving holder with horizontal axis normal to the ingot. They are positioned directly above the evaporation ingot and coating surface is horizontal in rotation axis. Electron beam evaporation using a 45 kW electron gun was carried out in the coating chamber under a vacuum of 10 Pa. The target material was heated above its evaporation temperature of 3000 °C, and the resulting vapors were condensed on stationary or rotating zirconia substrates. Oxygen gas flowing at 300 cm^3/min was fed into the coating chamber during deposition to control the oxygen deficiency of the zirconia. To determine the influence of the substrate rotation speed on the microstructure of the coating layers, coated specimens were formed at different rotation speeds, namely 0 (stationary), 1, 5, 10 and 20 rpm. Total coating thicknesses in the range of 20–700 μm were obtained. The substrate temperature was 950 °C in all conditions.

X-ray diffraction (XRD) was used to determine the crystal structures of the phases present and to determine if any preferred orientation developed in the coating layers. The porosity containing nanopores in the coated layers was performed using a mercury porosimeter. The microstructures of coated specimens were observed by SEM and analyzed by energy dispersive X-ray spectroscopy (EDS). The average size of column and pore of each coating layer was obtained using the intercept method on SEM micrographs.

3. Results and discussion

3.1. Influence of coating thickness on microstructure

Typical microstructures of the upper surfaces of coatings rotated at 5 rpm for different coatings thicknesses are shown in Fig. 1. The top surfaces of the coating layers consist of square-pyramidal or cone-like grains. The microstructure of the 500 μm thick coating contains larger grains than that of the 50 μm thick coating. In addition, gaps between the

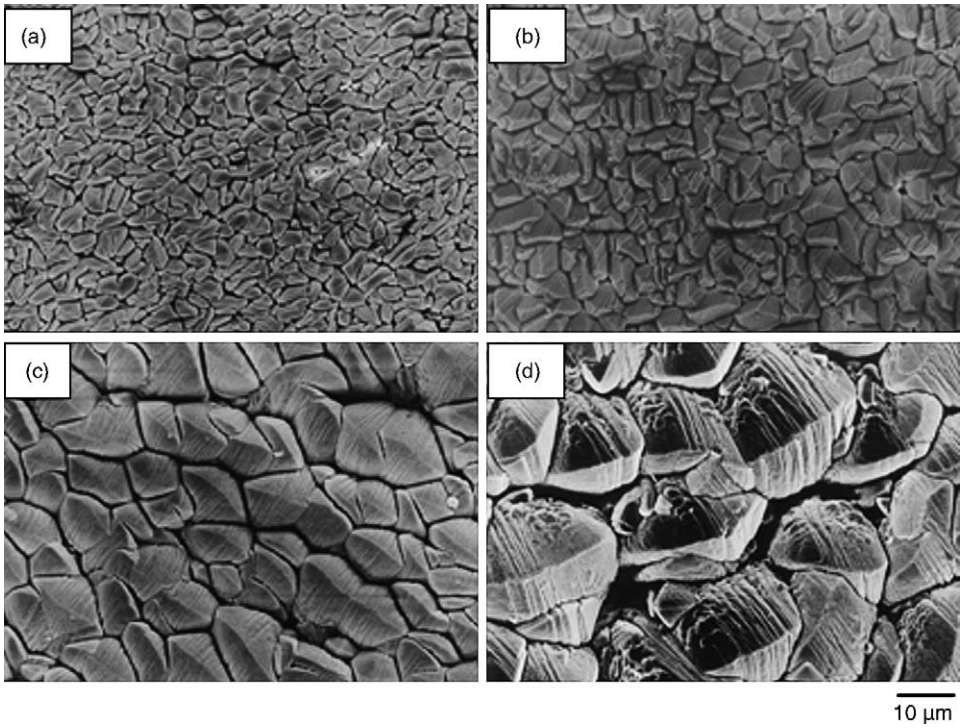

Fig. 1. SEM micrographs of surface regions of ZrO_2–4 mol% Y_2O_3 coating layers for different coating thickness obtained at 5 rpm: (a) 50 μm, (b) 100 μm, (c) 200 μm, and (d) 500 μm.