High-resolution X-ray Computed Microtomography Studies on Porous Coatings

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Introduction

X-ray computed microtomography (XCMT) studies were carried out to visualize and characterize the microstructures of three characteristic coatings applied in thermal barrier coatings (TBCs), solid oxide fuel cells (SOFCs), and prosthetic bone implants. The experiments focused on the quantitative characterization of porosity and its variation with thickness. Through these studies, we can further our understanding of the influence that environmental exposure has on relative changes in the microstructural features of these coatings. In the case of TBCs and SOFCs, we focused on quantifying the effects that increasing the temperature and time would have on microstructural changes in the coatings. In the case of bone implants, cell growth was the primary focus. The excellent energy tunability of XOR beamline 2-BM at the APS allowed us to characterize multilayered coatings of interest.

Methods, Materials, and Results

Ceramic TBC systems are widely utilized to protect high-temperature components in gas turbines from thermal oxidation and hot corrosion. The ceramic TBCs under investigation here are deposited by using electron-beam physical vapor deposition (EB-PVD). The EB-PVD process offers the opportunity of generating coatings with columnar microstructures having intercolumnar spacing across the thickness of the coating. These provide an inherently superior strain tolerance and thermal shock resistance, thereby resulting in significant lifetime enhancements. Since growth depends on competitive processes that can vary during deposition, variations in microstructure and texture are anticipated throughout the thickness of the coating. Conventional yttria-stabilized zirconia (YSZ) coatings were initially characterized. Because of the high-temperature conditions encountered, phase stability and sintering kinetics play a significant role in controlling the performance of these coatings [1-3].

Figure 1 shows changes in the microstructure (porosity) of these coatings with respect to the exposure temperature. It is evident that significant microstructural changes occur upon exposure to heat as a result of increased sintering kinetics/diffusion. It is also seen that this sintering/column bridging reduces porosity and hence results in a loss of strain tolerance. Also seen are regions of low porosity in the heat-treated coatings, indicating regions of column bridging/necking. The results show decreasing porosity with increasing temperatures, which is consistent with the higher elastic modulus measured on these coatings. However, upon long-term exposure above 1400°C, the tetragonal phase in the YSZ coatings destabilizes into cubic and monoclinic phases, resulting in significant micro-cracking and loss of the coating's mechanical integrity [2]. Hence, rare-earth-doped zirconates with pyrochlore structures are seen as potential candidates because of their low thermal conductivity, high-temperature-sintering resistance, and phase stability. Similar studies are being performed on these novel pyrochlore TBCs.

The second study involved process map studies of the production of reliable, porous titanium coatings for implant applications by vacuum plasma spraying [4]. Coatings with vastly different microstructures were deposited by using various feedstock particle sizes. They were compared with a reference titanium coating produced by using the powder metallurgy technique. Figure 2 shows one such microstructure with 55% to 60% porosity. It was found to heavily favor cell growth when compared with the sintered reference standard. Quantitative analysis of these sets of coatings is underway to better understand the pore connectivity, sizes, and porosity variation across the thickness of the coating. Experiments were also done to investigate and characterize a complete SOFC stack, consisting of an interconnect, a cathode, an electrolyte, and an anode. The system studied here is a porous nickel/YSZ cermet anode with a YSZ electrolyte and a porous $(La,Sr)MnO_3$ cathode. While porosity plays a crucial role in the operation of these SOFCs, the high operating temperatures (800-1000°C) of these structures cause microstructural changes. Hence, maintenance of the porous structure is important.

Discussion

A quantitative characterization of the microstructural features in porous coatings was demonstrated successfully

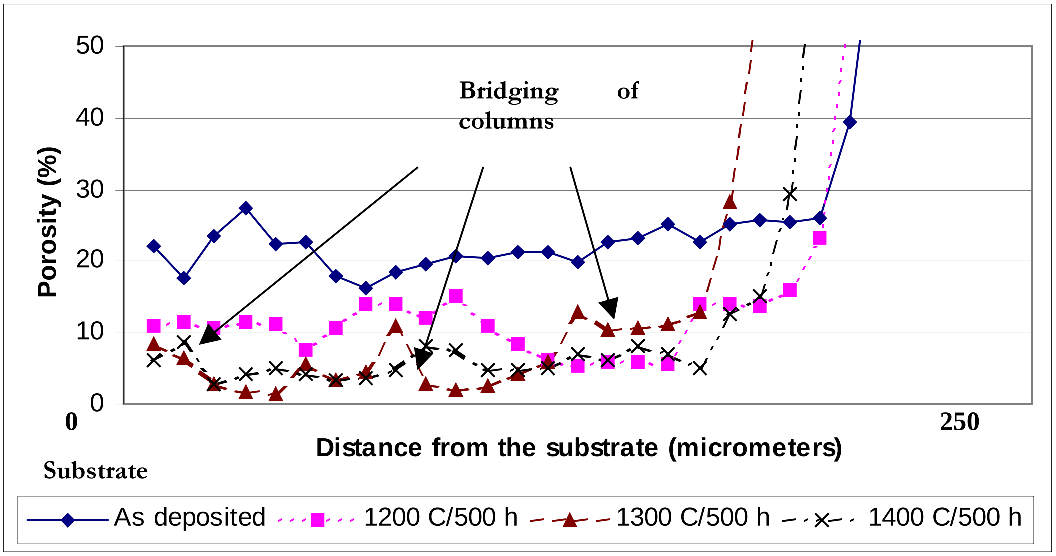

FIG. 1. Influence of exposure temperature on porosity variation along the thickness of the coatings.

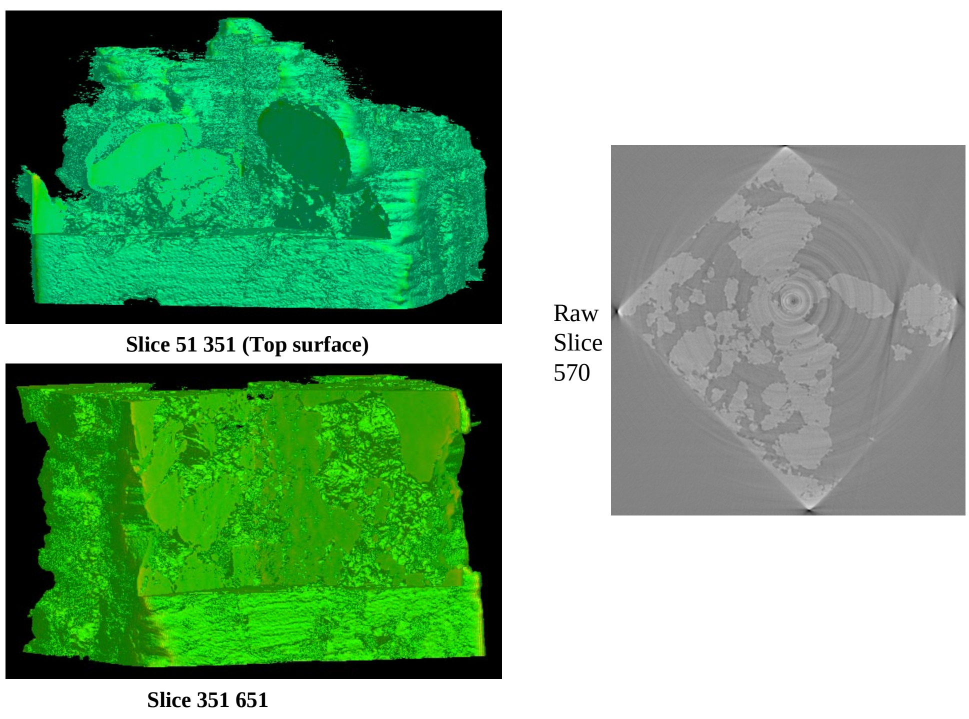

FIG. 2. XCMT of vacuum-plasma-sprayed titanium coating. Scale is approximately 600 μm^2 on a side.

in the case of EB-PVD TBCs, porous Ti implants, and SOFC materials by using the high-resolution XCMT technique. The results from a quantitative analysis of imperfections and results on microstructural changes upon environmental exposure (i.e., high-temperature exposure at different temperatures and times of exposure in the case of TBCs and SOFCs, and exposure to biofluids for cell growth in the case of porous implants) will help us comprehend coating behavior in service.

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