Anisotropic USAXS Studies of EB-PVD Microstructures

A. J. Allen,[1] J. Ilavsky,[1] A. Kulkarni,[2] H. Herman,[2] T. A. Dobbins,[1] P. R. Jemian[3]
[1]National Institute of Standards and Technology (NIST), Gaithersburg, MD, U.S.A.
[2]State University of New York (SUNY), Stony Brook, NY, U.S.A.
[3]University of Illinois at Urbana-Champaign, Urbana, IL, U.S.A.

Introduction

Modern, high-efficiency, gas turbine engines that are used to generate electricity operate at increasingly high temperatures, and thermal barrier coatings (TBCs) are needed to enable the blades and other components to withstand the extra heat. A multidisciplinary approach toward materials characterization is being used to explore the processing-microstructure-property correlations in both plasma-spray [1] and electron-beam physical-vapor-deposited (EB-PVD) [2] ceramic coatings. Ultrasmall-angle x-ray scattering (USAXS) is being combined with high-resolution x-ray microtomography in this effort. Experiments focused on quantitative characterization of the porosity (pore size distribution, orientation, and morphology) in these coatings and relative changes in the microstructure during thermal cycling, which affect the TBC lifetime.

EB-PVD has become a major means of depositing the ceramic TBC, which is typically yttria-stabilized zircoonia (YSZ), on the bond-coat and superalloy (blade) substrate. However, an optimized EB-PVD design depends on fully characterizing and understanding the complex anisotropic microstructures. Anisotropic USAXS capabilities have been developed at UNI-CAT to address exactly this kind of materials issue [2].

Methods and Materials

For anisotropic USAXS studies, side-reflection crystal optics were introduced to remove the intrinsic slit-smearing effect of the Bonse-Hart double-crystal scattering geometry and provide effective pinhole scattering. The anisotropy of the scattering is investigated by repeated USAXS scans over the range of scattering vector, Q, for different azimuthal orientations of the sample about the incident beam direction.

The EB-PVD samples were prepared by depositing YSZ from a rotating vapor plume onto a stationary substrate. A coating thickness of 0.5 mm was achieved by repeating the EB-PVD process. The coating and substrate were sectioned together to produce 0.2-mm-thick slices, and these were studied "in section" with the incident x-ray beam perpendicular to the slice. Three kinds of measurement were carried out: (1) USAXS scans as a function of Q for a given azimuthal angle to determine structure, (2) anisotropic scans of the azimuthal angle at fixed magnitude of Q to determine anisotropy at different scale lengths, and (3) USAXS imaging of the sample at different orientations and magnitude of Q (using the instrument's x-ray charged-coupled device [CCD] camera) to establish the location in the coating of particular anisotropic features.

Results

Figure 1 shows a typical anisotropic azimuthal scan at fixed $Q = 0.0006$ Å^{-1}, together with USAXS images of the EB-PVD sample for selected sample orientations. Features in the anisotropic scan can be associated with the unique EB-PVD columnar morphology, specifically, the intercolumnar pores (**I**) that are nearly perpendicular to the substrate and the intracolumnar pores (**C**) that are oriented at ± 30° or so to the column axes. At higher Q values, the fine intracolumnar pores become the dominant anisotropic scattering features, while the coarse intercolumnar pores are no longer apparent. After making allowance for effects due to the crystal optics, inspection of the USAXS images as a function of sample orientation reveals that the intercolumnar pore structure is more marked toward the base (B) of the coating, while the intracolumnar pore structure is more obvious toward the top (T). Upon thermal cycling of the coating (10×1150°C/0.5 h) to simulate typical service life conditions, some of the intracolumnar pores sinter away, while the intercolumnar pores become more ordered.

Figure 2 presents USAXS scans for particular orientations of Q with respect to the normal to the coating and also shows the apparent pore size distributions obtained from a maximum-entropy (MaxEnt) analysis. The apparent size distribution varies for different orientations because of the anisotropy of the microstructure (Fig. 3). Transmission electron microscopy (TEM) studies confirm that the 340 Å or 34-nm pores coalesce together to form planar voids that constitute the bulk of the anisotropic intracolumnar porosity. The anisotropy develops as the EB-PVD columns grow during deposition and arises because of effects on the vapor deposition of "shadowing" around the cone-shaped tops of the columns.

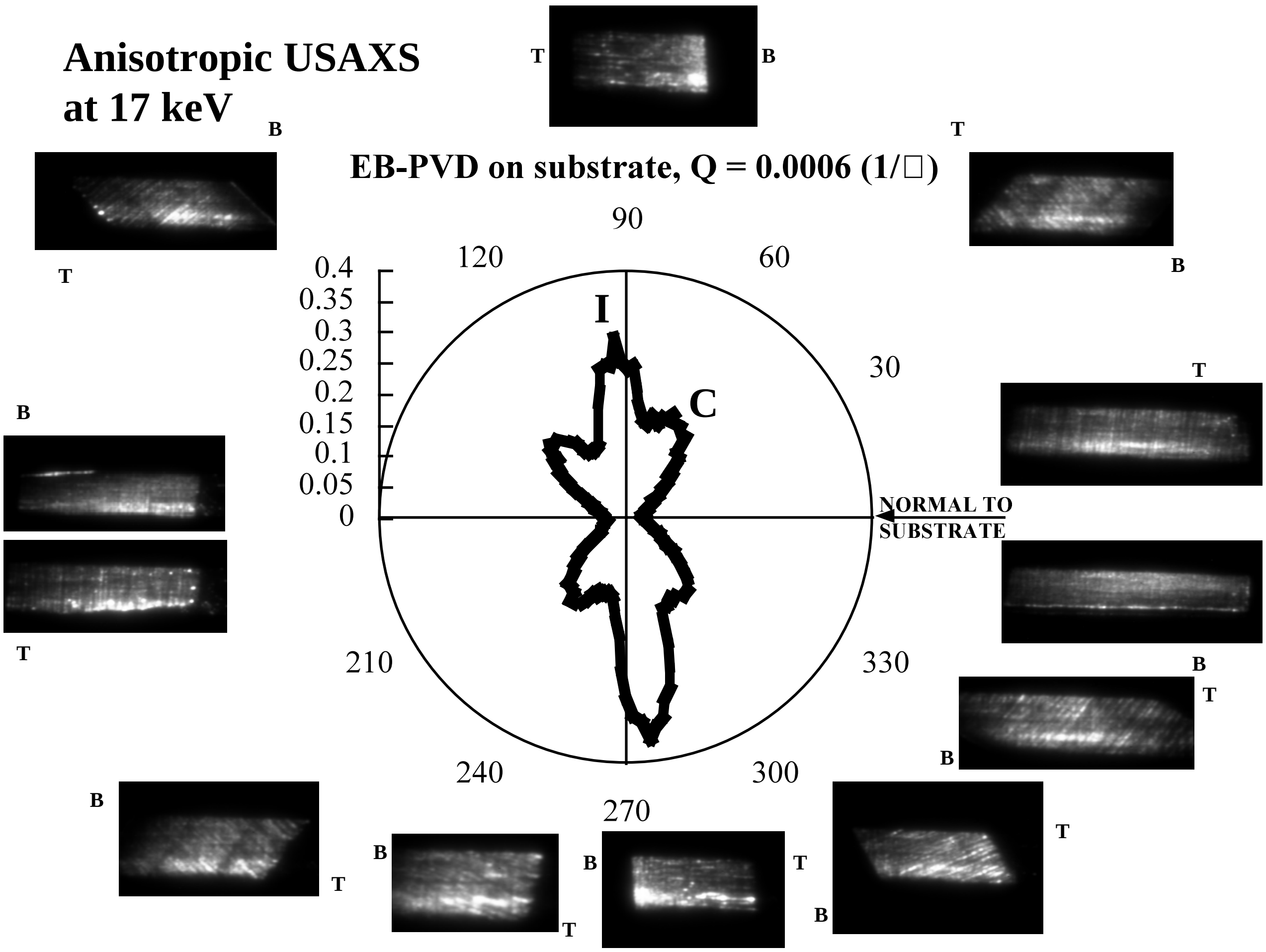

FIG. 1. Anisotropic USAXS intensity vs. azimuthal angle with USAXS images at selected orientations.

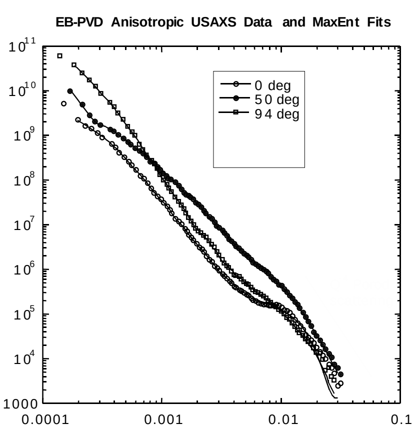

FIG. 2. Anisotropic USAXS scans for selected orientations of Q with respect to the normal to the substrate plane.

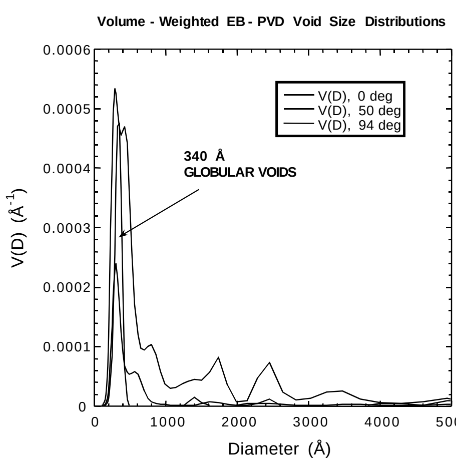

FIG. 3. Apparent MaxEnt size distributions corresponding to the anisotropic USAXS data shown in Fig. 2.

Discussion

With appropriate anisotropic microstructure models, the USAXS studies confirm that much of the total porosity is contained in the nanometer intracolumnar voids, even though it is the columns and intercolumnar voids that define the unique EB-PVD morphology. The studies also indicate that individual (globular) nanometer voids disappear almost entirely on thermal cycling. By quantifying the microstructure over the scale range from nanometers to micrometers, by following the changes during servicelike conditions, and by combining USAXS with microtomography and numerical modeling, the TBC property evolution may be predicted for different columnar microstructures. Hence TBC design may be optimized to take best advantage of superior EB-PVD tolerance against stress, corrosion, and thermal shock.

Acknowledgments

Use of the APS was supported by the U.S. Department of Energy (DOE), Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38. The UNI-CAT facility at APS is supported by the University of Illinois at Urbana-Champaign, Materials Research Laboratory (DOE, State of Illinois-IBHE-HECA, and National Science Foundation [NSF]), Oak Ridge National Laboratory (DOE under contract with UT-Battelle LLC), National Institute of Standards and Technology (U.S. Department. of Commerce), and UOP LLC. This research was also supported by the NSF MRSEC program at SUNY-Stony Brook under Grant No. DMR-0080021.

References

[1] J. Ilavsky, A. J. Allen, G. G. Long, P. R. Jemian, and J.-F. Bisson, “Studies of anisotropic void microstructures in YSZ deposits by small-angle x-ray scattering,” poster presented at Symposium P, Advances in Surface Engineering — Fundamentals and Applications, Materials Research Society fall meeting, Boston, MA (November 2001).

[2] J. Ilavsky, A. J. Allen, G. G. Long, and P. R. Jemian, “Effective pinhole-collimated ultrasmall-angle x-ray scattering instrument for measuring anisotropic microstructures,” Rev. Sci. Instrum. **73** (in press 2002).