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Three-dimensional X-ray micro-computed tomography of cracks in a furnace cycled air plasma sprayed thermal barrier coating

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Using X-ray micro-computed tomography, a total of 3 × 3 mm^2 area of an air plasma sprayed (APS) thermal barrier coating (TBC) was imaged with a resolution of 0.76 μm at 60% cyclic furnace life. Over 10,000 X-ray micrographs were used to extract the 3-D geometry of the cracks in the top coat. The true shape, center and location of the cracks with respect to bond coat peak–valley roughness features were obtained, which revealed the real nature of cracking characteristics of APS TBCs.

Keywords: Thermal barrier coating (TBC); Air plasma sprayed (APS); 3-D tomography; Image analysis; Cracking

Thermal barrier coatings (TBCs) are widely used by both the aviation and power generation industries to reduce the temperature experienced by the metallic components within the hot section of gas turbines and jet engines [1]. One of the most common types of TBCs is air plasma sprayed (APS) 7 wt.% yttria-stabilized zirconia (YSZ), which tends to fail during service due to progressive cracking. Cracking in APS TBCs has been studied extensively [2–4]. The cracking characteristics, such as crack shape, location, length and opening as a function of life fractions (hot time divided by total time to failure of a sample per test condition), can be used in fracture mechanics calculations of lifetime prediction as well as for developing coatings with optimized microstructure and bond coat roughness parameters. The most important information regarding the cracking of the APS TBCs are crack length, crack opening and the location of the crack center point with respect to peak–valley bond coat roughness features. A common method for investigating cracking of TBCs is by examining the interface cross-section via scanning electron microscopy (SEM) where a slab of sample is periodically sectioned during cyclic life testing while continuing to test the remaining of the sample. There are three major issues associated with sectioning methods: (i) the cross-section does not necessarily pass through the center point of the crack; (ii) the existence and position of peak–valley features behind a cross-section plane is unknown; and (3) data cannot be collected from a consistent location. For all these reasons, cracking characteristics must be collected from a large number of micrographs so that results can be concluded statistically. Obviously, this type of statistical dataset can be size dependent. Thus there are validity issues concerning cracking characteristics for which data is collected from 2-D micrographs and generalized to 3-D. In a previous study [5], over 1000 SEM cross-section micrographs were obtained by the sectioning method to characterize the cracking, oxidation kinetics, rumpling kinetics and failure mechanism as part of a cyclic furnace test program that was carried out at three temperatures and three cycle durations (three samples per test). Surprisingly, the result showed that cracking characteristics are approximately independent of temperature and cycle duration. In other words, crack length, ratio of crack opening to crack length and even per cent interface delamination as a function of life fraction stayed approximately the same. Only the rate of growth of cracks increased with temperature and inversely with cycle duration. This is very unexpected considering there was a 33 times difference between the longest to shortest life measured. Neither the true crack shape nor its relation to the bond coat geometry could be extracted in the previous study. All of this motivated us to measure the true shape of the cracks and their location in-situ (top coat in place with bond coat and substrate) and non-destructively by 3-D X-ray micro-computed tomography (μ-CT). The results obtained by sectioning are compared with 3-D X-ray μ-CT imaging results to assess the difference in cracking characteristics obtained by the two methods.

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The sample used here is an APS 7 wt.% YSZ, on an APS NiCoCrAlY bond coat and a nickel-base superalloy substrate. The overall shape of the sample is a disc of 25.4 mm in diameter. The nominal thicknesses are 0.254 mm top coat, 0.154 mm bond coat and 3.175 mm substrate. The sample was cyclically heat treated in atmospheric conditions, up to its 60% life fraction, along with samples tested in a previous study [5]. Due to a confidentiality agreement with the engine original equipment manufacturer the temperature and cycle duration of the test cannot be disclosed.

μ-CT is a computer-processed X-ray scanning method to generate 3-D imaging using multiple (10,000 in this study) 2-D radiographs in a manner analogous to medical computer axial tomography. This process allows for viewing virtual slices of a scanned region within an object. It is widely used in the medical field and the contrasting relies on the dissimilar X-ray attenuation coefficient for different materials (absorption contrasting) as well as phase of an X-ray wave as it traverses matter (phase contrasting) which can produce a unique grayscale contrast in a radiograph [6]. It should be noted that both the top coat material and substrate are "high-Z", meaning their elements such as Zr and Ni have high atomic number, Z, i.e. 91and 58, respectively. Therefore they have high attenuation coefficients, resulting in undesirably high absorption of X-rays during X-ray tomography. To achieve higher X-ray transmission, the entire heat-treated sample was mounted in a cold-mount low-shrinkage epoxy, EpoxySet (Allied High Tech Product, Inc., Rancho Dominguez, CA) and the substrate was ground down to 1 mm using a Beta-Vertex polisher (Buehler, Inc., Lake Bluff, IL). To avoid overheating the substrate during the grinding process, cold water was used as a coolant.

In order to look for cracks in the TBC in a non-destructive manner, 3-D X-ray μ-CT was performed using Versa-XRM-510 equipped with a 160 kV source (Xradia, Inc., now Zeiss, Pleasanton, CA). A large field of view (FOV) X-ray μ-CT was performed to map the entire 25.4 mm diameter sample to see where cracks nucleate and propagate with a resolution of 2 μm. On the other hand, small FOV X-ray μ-CT was carried out to look at the cracks that are in the range of 100–300 μm in length with a 3–5 μm opening at multiple locations within the sample over an area of at least 3×3 mm^2. The setting parameters for each scan type are shown in Table 1.

Image analysis was performed with Avizo Fire® software developed by FEI Visualization Sciences Group (Burlington, MA). In total, 10,179 X-ray micrographs were processed to identify separately every voxel (called segmenting) for: cracks, top coat material, bond coat, thermally grown oxide and substrate (referred to as labeling) using the watershed algorithm via the segmentation module [7]. 3-D surface meshes were generated by Delaunay triangulation using the Generate Surface module [7]. The centroid of each crack and its aspect ratio was found by computing the centroid and the principal axis using the Compute module [7]. It should be noted that for fewer than 5% of the cracks, the center of gravity was slightly outside the crack geometry because those cracks have a curved shape. In this case the center of the gravity was slightly shifted to be inside the crack. In addition, the location of the largest crack opening was found to be in agreement with the crack center of gravity due to the general shape of these cracks, which have the largest opening at the center and crack opening decreasing rapidly to zero toward the edges of the crack. The location of peaks and valleys were obtained by projecting the bond coat surface height contours onto a 2-D plane. Finally, the location of the crack center of gravity with respect to the peak–valley was obtained by the nearest-neighbor search algorithm, incorporated in MATLAB®, as described in Ref. [8].

Figure 1 shows an example of small FOV radiograph obtained by X-ray μ-CT without image processing; the quality of the image is comparable to that acquired using backscatter SEM. This enabled easier and faster segmentation of X-ray micrographs for such a large database. For visualization purposes, the 3-D reconstructed geometry of the cracks found in a typical region of this sample is shown in Figure 2a, which is based on stitching together two adjacent small FOV scans that have been segmented, labeled and the 3-D surface geometries generated. Each colored geometry is a different crack while the gray is the combined TGO-bond coat–substrate geometry and top coat is not displayed for simplicity of representation. As can be seen, there is a wide variation in crack shapes and sizes. Furthermore, the surface of the bond coat is very rough due to the nature of APS fabrication which is by solidification of molten droplets resulting in splats. The assumption of a simple, single, sinusoidal wave representation as the asperity used in nearly all models is far from reality. It can also be seen that the data form a single 2-D cross-section SEM

Table 1. Scan parameter for large and small FOV.

Scan type	Large FOV	Small FOV
FOV (mm)	2	0.6
Resolution (μm)	2	0.76
Projections	2401	3000
Angle range (°)	−98:+98	−96:+96
Binning	2	2
Exposure time (c)	2	15
Power setting (kV W^{-1})	150/10	90/8
Total time (h)	2	13:35
Filter	HE3	HE3

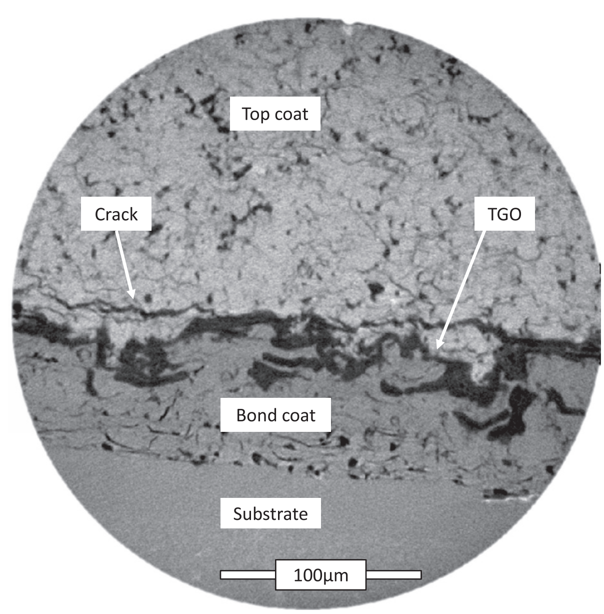

Figure 1. Small FOV radiograph obtained by X-ray μ-CT demonstrating the image quality generated without image processing.