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In situ transmission electron microscopy of high-temperature degradation of yttria-stabilized zirconia thermal barrier coatings



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ABSTRACT

The initial stage of degradation process of yttria partially stabilized zirconia thermal barrier coatings during heating at 1000–1450 K were observed in situ by transmission electron microscopy. The observation directly demonstrated that the formation of several kinds of oxides and resulting void growth started above 1000 K in the bond-coat layer near the interface with the thermally grown oxide layer, leading to the development of voids over the entire interface due to void coalescence. Such development of voids corresponds to the generation and propagation of thermally grown cracks, i.e., to the initial spallation of the coating.

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In yttria partially stabilized zirconia (YSZ) thermal barrier coatings used in environments where temperatures exceed 1000 K, such as jet engines, a metallic layer, i.e., a bond-coat (BC) layer, is introduced between the outermost surface YSZ layers, i.e., top-coat (TC) layers, and the substrates to improve the adhesiveness of the TC layers and the oxidation resistance of the substrates. However, BC layers lead to the formation of thermally grown oxide (TGO) layers with a thickness of 0.5–10 μm between the TC and BC layers [1–4], by which various phenomena related to the spallation of the coating occur near the TGO layer. Examples of such phenomena include crack formation due to the growth of the TGO layers [5,6], rumpling of the interfaces [7], oxidation of Cr and Ni–Co [6–8], and void formation [7,9,10]. In previous investigations on coating degradation, spallation mechanisms have been proposed on the basis of static observations of the coating textures before and after spallation [5]. In situ transmission electron microscopy (TEM) observations of the structural variation in coatings at high temperatures at which the coating is actually used enable us to investigate phenomena related to the spallation process and mechanism, providing guidance for the design of materials to suppress the degradation of thermal barrier coatings. We have developed an in situ high-temperature TEM technique using a 2000-K-class heating stage [11]; this method can directly elucidate the degradation process of YSZ thermal barrier coatings at temperatures where they are actually used.

YSZ thermal barrier coatings were prepared by successive deposition of BC and TC layers onto a substrate as follows. A CoNiCrAlY (bal. Co–32Ni–21Cr–8Al–0.5Y (wt%)) BC layer of 70- μm thickness was deposited onto a Ni-based superalloy (IN 738 LC) using vacuum plasma spraying. A YSZ (ZrO_2 –4 mol% Y_2O_3) TC layer of 200- μm thickness was subsequently deposited onto the BC layer via electron beam physical vapor deposition in a vacuum with residual gaseous molecules containing a small amount of oxygen molecules, i.e., a partial pressure of 1 Pa while the substrate was heated at 1223 K. Sample pieces for TEM and scanning electron microscopy (SEM) were cut and mechanically polished to observe the cross section of the TC/BC interfaces of the thermal barrier coating. The interface regions were further thinned to 100 nm thickness for TEM observation using a focused-ion-beam method.

The TEM sample was mounted onto the 2000-K-class sample heating holder [11], and the holder was inserted into the sample chamber of the transmission electron microscope (JEOL, JEM-2011KZ). The sample was heated inside the microscope, and the structural variations were observed in situ and recorded with a charge-coupled-device camera with a frame rate of 6.25 image-frames per second. We carried out two heating cycles. The sample temperature was increased from room temperature to 1360 K within 17 min and was then decreased to room temperature within 2 min. Subsequently, the temperature was increased again from room temperature to 1450 K within 10 min and maintained at 1450 K for 10 min. The temperature was then decreased to room temperature within 2 min. Elemental analysis of the coating

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was carried out using energy-dispersive X-ray spectroscopy (EDX); the X-ray spectrometer was attached to a scanning electron microscope (FEI, Helios NanoLab 600i).

Fig. 1 shows a SEM image of the cross section of an interface of the YSZ thermal barrier coating. The upper part of the image shows the YSZ TC layer, in which columnar structures are formed along the direction perpendicular to the interface to reduce thermal strain during thermal protection [2]. Note that a 500-nm-thick TGO layer was formed between the TC and BC layers.

Fig. 2 shows a time series of TEM images detailing the structural variation in the TC/TGO/BC interface during heating (see Movie 1 in Supplementary data). In the as-prepared state, the TC and TGO layers were closely bonded (Fig. 2(a)). In contrast, voids smaller than several tens nanometers in width are observed at the TGO/BC interface (arrows in Fig. 2(a)). When the sample was heated to 1000 K, these voids grew (Fig. 2(b)). When the heating temperature was increased to 1360 K, new voids formed and the void growth accelerated along the TGO/BC interface plane (Fig. 2(c)). As the temperature was further increased to 1450 K, the acceleration of the void growth led to the coalescence of voids, i.e., the development of voids of several micrometers in width over the entire TGO/BC interface (Fig. 2(d)). Such development of voids over the entire interface in the BC layer corresponds to the generation and propagation of thermally introduced cracks. Note that no microstructural variation inside either the TC or the TGO layer was observed. Thus, the in situ TEM observation directly shows that the initial stage of the coating degradation was caused by spallation at the TGO/BC interface as a result of the development of voids over the entire interface.

Fig. 3 shows electron diffraction patterns of the BC region before and after heating. Before heating (Fig. 3(a)), the diffraction pattern corresponds to a Co–Ni–Cr solid solution (γ phase) (unmarked hkl indices

in Fig. 3). The lattice parameter of this γ phase is 0.37 nm, which is 3% larger than that of the γ phase produced such that only the BC layer was deposited onto the substrate with the same composition ratio, i.e., without subsequent deposition of the TC layer and the corresponding formation of the TGO layer [12]. Although CoNiCrAlY BC layers have been reported to comprise a γ -phase matrix and NiAl (β -phase) precipitates [12,13], β -phase precipitates are not observed in the present sample (Fig. 3(a)). After heating (Fig. 3(b)), the intensity of the diffraction spots corresponding to the γ phase decreases to less than that of the sample before heating. Instead, new diffraction spots corresponding to several oxides, including α -Al₂O₃, NiCr₂O₄, and Y₂O₃, as shown in Fig. 3. The emergence of these diffraction spots implies that these oxides were formed from the γ phase. Similar results have been reported in previous studies on BC layers with the same composition [14].

Fig. 4 shows the results of EDX elemental analysis of the TC, TGO, and BC layers before and after heating. The peaks in the spectra of the TC and BC layers correspond to those of the raw material components (TC: Zr, O, Y; BC: Co, Ni, Cr, Al, Y) (the peaks indicated by triangles in Fig. 4). The other elements (unmarked peaks in Fig. 4) are those introduced through atomic diffusion during deposition, indicating that atoms mutually diffuse among the TC, TGO, and BC layers. The same elements in the TC layer are observed after heating. In the BC layer, although the same elements are observed after heating similarly, the intensities of the Al, Ni, and Cr peaks decreased. In contrast, the intensities of the Cr peaks in the spectrum of the TGO layer are substantially lower after heating.

The spallation of YSZ thermal barrier coatings has been attributed to crack generation at the TGO/BC interfaces as a result of heterogeneity in the TGO layer, i.e., non-uniform growth of the TGO layer [5,15,16]. In the present study, the formation and growth of several kinds of oxide particles, e.g., α -Al₂O₃, NiCr₂O₄, and Y₂O₃, in the BC layer were confirmed, indicating that the TGO layer grew into the BC layer. Such oxide formation is heterogeneous and introduces strains at the TGO/BC interfaces. The content ratios of Al, Ni, and Cr in the BC layer decreased after heating, as shown in the elemental analysis results (Fig. 4). This corresponds to outward diffusion of these elements to the TGO/BC interface, followed by the formation of α -Al₂O₃, NiCr₂O₄, and Y₂O₃. The Cr content ratio in the TGO layer also decreased after heating, implying the outward diffusion of Cr from the TGO layer. Such composition and changes in the BC and TGO layers introduce strains at the TGO/BC interfaces, promoting the formation and growth of voids. Therefore, both the heterogeneous oxide formation at the TGO/BC interface during heating and the variation in the lattice parameters of the BC and TGO layers due to atomic diffusion introduce strains at the interfaces. These strains is inferred to promote the formation and growth of voids at the BC layer along the TGO/BC interfaces, corresponding to the generation and propagation of thermally introduced interfacial cracks. The present in situ observation directly reveals that such void growth led to the interfacial spallation, i.e., the initial stage of the degradation of the YSZ thermal barrier coating.

The void formation at the TGO/BC interfaces is explained on the basis of the inward diffusion of oxygen in TGO to the TGO/BC interfaces and the outward diffusion of Al in TGO to the TC/TGO interfaces; when the supply of Al in BC to the TGO/BC interfaces is insufficient, voids are generated [17–21]. We compare void sizes in YSZ thermal barrier coatings using NiCoCrAlY (bal. Ni-20Co-20Cr-8Al-0.5Y (wt%)) BC layers [7], similar composition to that of the present BC layer. After a long thermal treatment, i.e., 314 cycles of 40 min heating at 1394 K, of this YSZ thermal barrier coating, voids larger than several tens micrometers are formed [7]. This void size is much larger than that observed in the present study (several micrometers). This is because the present heating period is very short (10 min at 1450 K). Thus, the void formation and growth observed in the present study corresponds to the initial stage of the formation of several tens micrometer voids after long heating periods. In particular, because small voids of several micrometers or less form faster, they may generate and coexist after long heating periods.

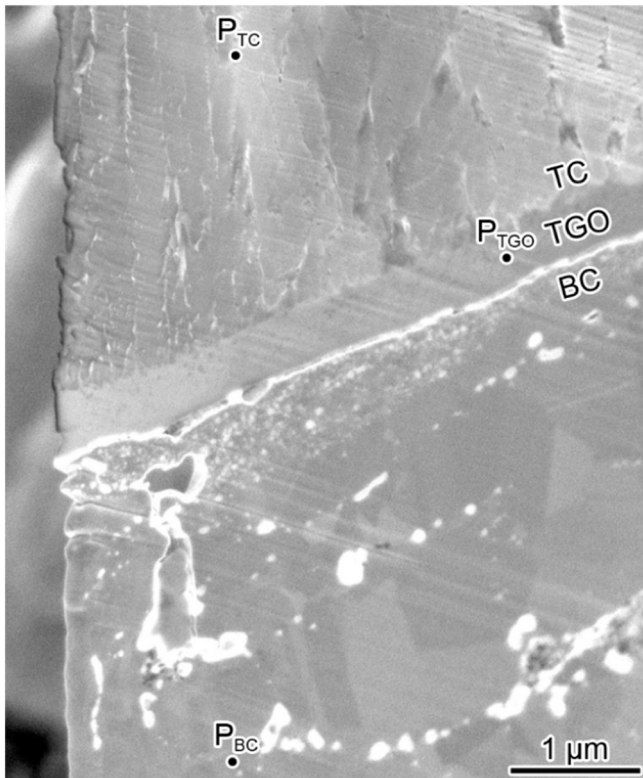


Fig. 1. SEM image of the cross section of a TC/TGO/BC interface in an as-prepared YSZ thermal barrier coating. The upper side in the image is the YSZ TC, and the lower side is the CoNiCrAlY BC. TGO is formed between the TC and the BC. The points indicated by P_{TC} , P_{TGO} , and P_{BC} are the EDX elemental analysis points in the TC, TGO, and BC layers, respectively (see Fig. 4).

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