# Microstructure design for blended feedstock and its thermal durability in lanthanum zirconate based thermal barrier coatings

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#### Abstract

The effects of microstructure design on the lifetime performance of lanthanum zirconate (La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>; LZO) based thermal barrier coatings (TBCs) were investigated through various thermal exposure tests, such as furnace cyclic thermal fatigue, thermal shock, and jet engine thermal shock. To improve the thermal durability of LZO-based TBCs, composite top coats using two feedstock powders of LZO and 8 wt.% yttria doped stabilized zirconia (8YSZ) were prepared by mixing at different volume ratios (50:50 and 25:75, respectively). Also, buffer layers were introduced in layered LZO-based TBCs deposited by air plasma spray method. The TBCs with two buffer layers showed the best thermal shock resistance and thermal cyclic performance among all samples in all tests. For applications with mildly slow cooling rates, the thermal durability in single-layer TBCs is more effectively enhanced by controlling composite ratio in the blended powder, better than introducing a single buffer layer. For applications with fast cooling rates, the thermal durability can be effectively improved by introducing buffer layer than composite top coat, since buffer layer provides fast localized stress relief due to its high strain compliance. These research findings allow us to control the TBC structure and the buffer layer is efficient in improving thermal durability in the cyclic thermal exposure and thermal shock environments.

*Keywords*: Thermal barrier coating; Lanthanum zirconate; Layer structure; Composite; Thermal exposure.

#### 1. Introduction

Thermal barrier coatings (TBCs) have been widely employed to protect the hotsection components of gas turbines and accommodate the increase of turbine inlet temperature, resulting in the improvement of the generation efficiency [1–4]. Most of TBC systems are consisted of a superalloy substrate, an adhesive metallic bond coat, and a thermal insulating ceramic top coat. The typical top coat material in TBC is 7-8 wt% yttria doped stabilized zirconia (8YSZ), which is deposited by air-plasma spray (APS) or electron-beam physical vapor deposition [5]. However, the usage of YSZ has limitation to satisfy the demand for higher efficiency in gas turbines. At higher operating temperatures, YSZ based TBCs have a degradation by phase transition and microstructure densification. YSZ transforms from t phase to the tetragonal and cubic phase (t and c phases, respectively) during cooling process after higher operating temperatures above 1200 °C, and then to monoclinic (m) phase with a volume expansion of about 3–5 vol.%, resulting in the spallation or delamination of TBCs [6]. In addition, the poor sintering resistance of YSZ at higher temperatures reduces porosity in the top coat, deteriorating the thermal insulation ability of YSZ coating. It also increases the elastic modulus due to microstructure densification, causing the decrease of the strain compliance of YSZ coating [7–9].

There were many trials to overcome the limitation of current YSZ material, such as zirconates, perovskites, and hexaaluminates [6, 10–12]. Among them, zirconates which contain rare-earth elements have been investigated as a new material for advanced TBCs in higher operating temperatures above 1200 °C. Especially, lanthanum zirconate (La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>: LZO) has significantly lower thermal conductivities than YSZ. The thermal conductivity of LZO ceramic has a lower value of  $1.5 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$  for the bulk, as contrasted with 2.1 W·(m·K) <sup>-1</sup> for bulk YSZ [7]. The superior thermal insulation ability of LZO can be explained by two

reasons. LZO has a pyrochlore crystal structure which contains more oxygen vacancies than fluorite YSZ material, resulting in disturbance of lattice scattering which is the dominant mechanism of thermal conduction in ceramic material [6, 13]. The lattice scattering is also interrupted by the atomic weight difference between lanthanum and zirconium which are 138 and 91 g·mol<sup>-1</sup>, respectively. On the other hand, LZO is very stable and has no phase transition in the temperature range of 30-1400 °C [14].

Although the promising characteristics of LZO, however currently, the biggest challenge of LZO for application is the relatively poor lifetime performance than typical YSZ based TBC. LZO has lower coefficient of thermal expansions (CTEs, 9.1-9.7×10<sup>-6</sup>·K<sup>-1</sup> for LZO and 10.5-11.5×10<sup>-6</sup>·K<sup>-1</sup> for 8YSZ) and inferior fracture toughness than YSZ (~ 1.1 MPa·m<sup>1/2</sup> for LZO and ~ 2.0 MPa·<sup>1/2</sup> for 8YSZ) [6, 15]. The former results in severe CTE mismatch with typical bond coat materials. During operation, the delamination of LZO-based TBC was observed due to the stresses at the interface between the top and bond coat which is generated by CTE mismatch [14]. Besides, its low fracture toughness can't hinder crack propagation and delamination during operation [7, 16]. Vassen et al. performed thermal cyclic test to evaluate the improvement of thermal durability by introducing double-layer TBCs (8YSZ buffer layer and LZO top layer) in the range of temperature from 1200 °C to 1450 °C. The double-layer TBCs showed an enhanced thermal durability than LZO singlelayer TBC and similar results to signle-layer 8YSZ system at temperatures below 1300 °C [17]. Wang et al. compared the thermal shock behavior with single-ceramic-layer 8YSZ and double-ceramic-layer LZO/8YSZ coatings. The double-ceramic-layer coating had a longer thermal shock lifetime even than that of single-ceramic-layer coating, indicating the doublelayer structure is an efficient design to overcome shortcoming of LZO based TBC [18]. On the other hand, Wang *et al.* also studied the thermal cyclic behavior of TBC system with graded coating, the single-layer and double-layer coatings. The thermal cyclic lifetime of the graded coating was extended than that of the single and double layer coatings [19].

In this research, a new microstructure of LZO-based TBC was designed with the concept of layer structure using blended LZO and 8YSZ feedstock powders. The mixed LZO and 8YSZ powders with selected volume ratio allow to combine advantages of LZO thermal insulating performance and 8YSZ thermal durability. Moreover, buffer layers with the single and double structures were introduced between bond coat and blended top coat to enhance interfacial stability. The effects of architecture design on the thermal durability were investigated through furnace cyclic thermal fatigue (FCTF), thermal shock (TS), and jet engine thermal shock (JETS) tests. Also the microstructure evolution and mechanical property were observed and evaluated before and after thermal exposure tests. The thermal durability with structure design was discussed, based on the comparison of microstructure evolution and mechanical property before and after thermal exposure tests.

## 2. Experimental procedure

#### 2.1 Sample preparation

The disk shaped Ni-based superalloy (Nimonic 263, a nominal composition of Ni-20Cr-20Co-5.9Mo-0.5Al-2.1Ti-0.4Mn-0.3Si-0.06C, in wt.%, ThyssenKrupp VDM, Germany) was used as a substrate. The dimensions of the specimens were 25 mm in diameter and 5 mm in thickness. Before deposition of the bond coat, sand-blasting was performed on the surface of the substrate using Al<sub>2</sub>O<sub>3</sub> powder with a particle size about  $\sim$  420 µm. The feedstock powder of the bond coat was AMDRY 962 (hereinafter 962; Sulzer Metco Holding AG, Switzerland, nominal composition of Ni-22Cr-10Al-1.0Y in wt.% and particle size of 56-106 um). METCO 204 C-NS (hereinafter C-NS; Sulzer Metco Holding AG, Switzerland, particle size of 45–125  $\mu$ m) was employed as a feedstock powder for 8YSZ. LAO-109-1 (Praxair Surface Technologies, Indianapolis, IN) was the powder used to produce the LZO coatings. LZO and 8YSZ powders with different volume ratios of 50:50 and 25:75, respectively, were mechanically mixed by ball mill drive (NITBMT0202, NANOINTEC, Republic of Korea) for 4 h, and then used as a blended powder.

The thicknesses of the bond and top coats were approximately 150 and 430 µm, respectively. First, the bond coat was deposited by using a commercial APS coating method (9MB, Sulzer Metco, Switzerland), then the top coats including buffer layers were coated by using same equipment. Two types of single-layer top coat (SLC) without buffer layer were deposited using different blended compositions, and two types of blended double-layer top coat (DLC) with buffer layer were deposited using 8YSZ and LZO. In DLC, the buffer layers are different. One has only 8YSZ buffer layer and the other has two buffer layers of 8YSZ and blended composition of 25:75 vol.% in LZO and 8YSZ, respectively. The detailed designs are illustrated in Fig. 1. All coating processes were done within 2 h.

#### 2.2 Characterizations

To obtain both cross-sectional microstructure and mechanical properties before and after thermal exposure tests, each TBC sample was cold-mounted using epoxy resin. The mounted samples mounted into a fluid epoxy resin were polished using silicon carbide paper and polished using the 3 and 1 µm diamond pastes. The cross-sectional microstructure of TBC specimens was observed using a scanning electron microscope (SEM, JEOL Model JSM-5610, Japan). The chemical composition of each TBC system was determined using an energy dispersive spectrometer (EDS, Oxford Instruments, Oxford, UK). The mechanical properties were characterized by two kinds of indentation test, nano-indentation and microindentation. Localized elastic modulus, E, and hardness, H, were evaluated by nano-indentor (Nanoindenstruments, MTS Systems Corp., Eden Prairie, USA) with a Berkovich tip (radius of tip < 100 nm). The data were selected after indentation of up to 2000 nm depth. The global H values of the bond and top coats were determined using a micro indenter (HM-114, Mitutoyo Corp., Japan) with a Vickers tip for a load of 3N and holding of 15 s. To obtain reliable values, more than 10 points were tested for each Vickers hardness result. The size of the indentation impression was measured using SEM and all indentations were performed at room temperature.

## 2.3 Thermal exposure tests

A bottom-loading programmable cyclic furnace was used for FCTF tests. The tests were performed at a surface temperature of 1100°C with a temperature difference of 150°C between the top and bottom surfaces of specimen with a dwell time of 40 min, and then natural air cooling was allowed for 20 min at room temperature. The failure was defined as 25 % delamination of the top coat surface. The TS tests were carried out till delamination in a muffle furnace. When the temperature of the furnace reached to 1100°C, the samples were pushed into the furnace. In the TS tests, the samples were held for 40 min in the furnace and then directly quenched into water. Throughout the TS, the temperature of water was between 20 and 35°C. More than 50% of the region spalled in the top coat or cracked at the interface between the top and bond coat was adopted as the criterion for failure in water quenched samples. Additionally, to investigate the thermal shock and thermal cycling performance, JETS tests were conducted with using specially designed system; the center of top coat was exposed for 20 s by flame with the temperature of 1400 °C and directly quenched by nitrogen

gas for 20 s, respectively, followed by ambient cooling for 1 min. The failure criteria in the JETS tests were typically more than 20 % spallation of the TBC surface.

## 3. Results and discussion

#### 3.1 Characterization of as-coated samples

Cross-sectional microstructures and element analysis results of as-coated TBCs are shown in Fig. 2. The bond coat showed a typical microstructure prepared by APS method in all samples, which contains oxide materials and some pores. The top coat also showed intrinsic defects, such as pores, unmelted particles, and splat boundaries. The top and bond coats were well deposited with controlled thicknesses of  $350 \pm 80$  and  $140 \pm 10 \ \mu m$  in the top and bond coats, respectively, even though the sample C (Fig. 2C-1) shows a slightly thicker top coat than others. A thick coating thickness may cause the thermal and residual stresses, but the implication was negligible on thermal durability according to previous study [20]. Figures 2A and 2C are the SLC TBCs using the blended feedstock powders with volume ratio 50:50 and 25:75 of LZO and YSZ, respectively. In the microstructure images, Fig. 2A-1 shows more LZO content (white color) and less YSZ content (gray color) than Fig. 2C-1, consistent with the coating design shown in Fig. 1. The dense YSZ buffer layer could be observed in both DLC TBCs of Figs. 2B-1 and 2D-1. Each buffer layer was deposited with the thickness of 60 µm. In the DLC TBC of Fig. 2D-1, the second buffer layer with a volume ratio 25:75 of LZO and YSZ, respectively was inserted between the top coat and the first buffer layer. The TBCs did not show any delamination or cracking at the interface between the top and bond coats. All samples showed an irregular surface roughness at the interface. Also, indistinguishable interface without obvious large cracks were observed within top coats, indicating minimal thermal and residual stresses were imposed at the interfaces.

In Fig. 2, the white arrows indicate the guide line of EDS analysis inside each image. The element distribution for zirconium (Zr) and lanthanum (La) were different between the SLC TBCs and the DLC TBCs with the buffer layers. The existence of buffer layer above the bond coat and content of each element could be better identified through evaluating the distribution of Zr element, rather than La element, due to less content of La element and distinguishably large particle size of YSZ and LZO powders. On the other hand, the *E* and *H* values of each TBC from nano-indentation were summarized in bottom graphs. Both of *E* and *H* values had continuity across the sample thickness in the samples C and D. On the contrary, discontinuous values were observed at the interface between the bond and top coats in the samples A and B. The samples B and D suffered from more paths to form the designed layered structure, resulting in thermal and residual stresses between layers [21]. However, the obtained *E* and *H* values were not significant different in all samples, indicating that the influence of continuity or discontinuity could be negligible for lifetime performance in thermal exposure tests.

#### 3.2 Furnace cyclic thermal fatigue test

Equivalent operating hour (EOH) is commonly used for estimating lifetime in inspection of gas turbine system. Equivalent operating hour is a function of the operating hours, coefficient of correction, load rejection, equivalent hours for startup, trips, rapid load change, standby, fuel operation, etc. [19, 22] Based on the EOH, the conservation and inspection interval units are performed rather than actual running hours:

$$EOH = AOH + 20[\sum S_i + \sum LR_i + \sum T_i + \sum LC_i] \times F,$$

where EOH, AOH,  $\sum S_i$ ,  $\sum LR_i$ ,  $\sum T_i$ ,  $\sum LC_i$  and F represent the equivalent operating hours, actual operating hours, coefficient of correction, load rejection, trip, rapid load change, and

fuel factor (gas: 1.0), respectively. In this study, EOH was estimated as an approximate value by multiplying 21 with the number of cycles.

TCFT tests were performed for each sample to evaluate the thermal durability of newly designed TBCs till 1143 cycles (24,000 EOH). The numbers of cycle-to-failure and surface status are summarized in Table 1. The cross-sectional microstructures of each specimen after FCTF tests are shown in Fig. 3. The sample A (Fig. 3A) with 50:50 vloume ratio composite top coat was just delaminated after 540 cycles (11,340 EOH), showing the poorest thermal fatigue resistance. The delamination of samples B and C was occurred after 768 and 936 cycles (16,120 and 19,650 EOH), respectively. The improvement suggests that the thermal fatigue resistance can be effectively improved by controlling composition ratio than introducing a buffer layer. FCTF test has a relatively milder cooling rate than TS test. In FCTF tests, the interfacial residual stress had sufficient time to dissipate into a thick composite (sample C) coat, but was saturated in a thin buffer layer (sample B). The DLC TBC, the sample D survived after 1143 cycles (24,000 EOH) without spallation of the top coat, showing a superior thermal fatigue resistance. The densified microstructure of top coat was observed after the tests, showing less splat boundaries, and crack coalescence and growth compared to as-coated microstructure. FCTF tests of sample D suggest that combining graded composite top coat and buffer layer is an effective strategy to improve the thermal fatigue resistance performance.

The delamination was occurred within each top coat and/or the interface between the top and bond coats in the samples A, B, and C, as shown in Fig. 3. During FCTF tests, the development of TGO layer (black:  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) was observed at the interface of the top and bond coats in all samples, while the spinel composition (dark gray: Cr<sub>2</sub>O<sub>3</sub>, NiAl<sub>2</sub>O<sub>4</sub>, and other spinel structures) in the TGO layer was developed in the sample D only, owing to longer

thermal exposure time compared to the samples A, B, and C. If the thickness of TGO layer is greater than 10  $\mu$ m, the interface between TGO layer and top coat normally starts to be delaminated and shows a failure phenomenon. The formation mechanisms and composition of TGO layer have been well documented in literature [23, 24]. As a result, the improvement of lifetime performance can be obtained in actually applied TBC systems, resulted from introducing buffer layer and controlling composition ratio which can protect oxidation of bond coat and growth of TGO layer.

## 3.3 Thermal shock test

TS tests were performed for all samples, and the numbers of cycle-to-failure and surface status are summarized in Table 1. The SLC TBCs, the samples A and C were fully delaminated after 10 and 14 cycles, respectively. On the other hand, the DLC TBCs with buffer layer showed improved thermal shock resistance. The delamination of the DLC TBCs with the single and double buffer layers occurred after 29 and 54 cycles, respectively. Consequentially, thermal shock resistance would be effectively improved by introducing buffer layer between the top and bond coats. Our results suggest that, during high cooling rate such as TS tests, buffer layer provides localized stress relief due to its high strain compliance. By introducing buffer layer at the interface between the top and bond coats in SLC TBC, the magnitude of the thermal and residual stresses could be diffused quickly, which is important of applications with fast cooling/heating rates [21]. Particularly, the thermal and residual stresses imposed on the sample D could be distributed into three layers by introducing double buffer layers. Therefore, the DLC TBC with double buffer layers shows the most improved thermal shock resistance.

The cross-sectional microstructures of each TBC sample after TS tests are shown in

Fig. 4. The SLC TBCs, the samples A and C were delaminated just within 15 cycles, showing a thinner TGO layer than those in FCTF tests. This is due to different thermal exposure time between TCFT tests and TS tests. T DLC TBCs with buffer layer, the samples B and D were survived after 29 and 54 cycles, respectively. There were full delamination in the sample B and partial delamination in the sample D, with large horizontal cracks at the interface of the two buffer layers. The delamination in the samples B and D was created within the composite layer, due to the lower fracture toughness of LZO than that of 8YSZ. Also the CTE mismatch between the bond coat and the LZO was reduced by introducing the buffer layer [6, 15]. Therefore, CTE difference should not be the only reason for delamination of LZO based TBCs. Delamination can more easily occur in SLC TBCs was likely caused by a combination of its low fracture toughness and high stresses created by CTE mismatch.

In TS tests, before water-quenching, the whole temperature of samples was about 1100 °C. Compared with the LZO and YSZ based top coats, the bond coat and substrate have higher CTEs than the top coats, resulting in larger volume shrinkage of bond coat and substrate during quenching sequence. The in-plane compressive stresses and out-of-plane tensile stresses would be created, and small cracks and/or splat boundaries coalesced and propagated within top coat near the interface. The partial delamination resulted from horizontal crack growth can prevent the top coat from catastrophic full delamination in actual application. However, the bond coats in all samples were degraded more after TS tests than FCTF tests. In TS tests, the TGO layer consisted of Al<sub>2</sub>O<sub>3</sub> was easily cracked due to its low CTEs and brittleness in water quenching sequence [25]. The penetration of oxygen to the bond coat was difficult to prevent due to multiple cracks. On the other hand, development of uniform and thick TGO layer prevented the permeation of oxygen in FCTF tests, resulted

from slower cooling sequence than TS tests.

## 3.4 Jet engine thermal shock test and comparison

The numbers of cycle-to-failure and surface status in JETS tests are summarized in Table 1. The microstructures of samples after JETS tests are shown in Fig. 5. The first delamination was occurred in SLC TBC with 50:50 ratio in early stage of within 70 cycles with catastrophic failure, and then the sample B with 25:75 ratio was delaminated after 1022 cycles. As shown in Fig. 5, delamination happened near the interface between the bond and top coats in SLC TBCs, showing similar fracture position and behavior with prior microstructures after the TCFT and TS tests. However, the samples B and D with buffer layer survived after 2000 cycles, showing a superior thermal stability. During JETS tests, densification occurred from the surface of top coat, resulting in generation of vertical cracks in the samples B and D (indicated with arrows in Fig. 5), due to disappearance of splat boundary and pores. The growth and coalescence of horizontal cracks were observed at the interface between the buffer layer and the top coat in the sample B, and between the buffer layers in the sample D. This microstructure of vertical cracked TBC system can enhance the thermal durability with improved strain compliance during operations [26]. As a result, the DLC TBCs with buffer layer will show superior thermal durability due to its vertical cracks. Similar to previous results of the TCFT and TS tests, the results of JETS tests indicated that the buffer layer could diffuse the stresses caused by CTE mismatch and improve thermal durability of TBCs. On the other hand, the interfacial stability was obtained without any evident growth of TGO scale, even compared to the as-coated microstructures in Fig. 2. This interfacial stability is the result of thermal barrier ability of low thermal conductivity material and relatively short thermal exposure time compared to the FCTF and TS tests.

Since the sample D demonstrated the best thermal durability among all samples, its microstructure and Vicker's micro-hardness of as-prepared, after the FCTF, TS, and JETS tests are summarized in Figs. 6 and 7, respectively. After TCFT tests, densification in the top coat and cracks within the buffer layer were observed, while the top coat after TS tests contained a large horizontal crack which caused the partial delamination. After TS tests, the temperatures in the substrate and the bond coat were same with the top coat, resulting in the horizontal cracks due to a rapid shrinkage of the substrate and the bond coat caused by large CTE mismatch. However, JETS tests created vertical cracks (not shown in Fig. 6) by exposure of only top coat surface by direct flame and the horizontal cracks between the buffer layers.

Mechanical property evolution for the sample D is summarized in Fig. 7, before and after thermal exposure tests. In general, there was an increase of *H* values in whole coating layer. The main reason of increase is due to densification of the ceramic layer (buffer layer and top coat) and oxidation for the bond coat during thermal exposure. In the FCTF and TS tests, the increase of hardness values in buffer layer was less than those of top coat (composite layer) due to the position of buffer layer where can be protected from severe exposure. On the other hand, the increase in the top coat after TS tests was larger than that after FCTF tests. Even thermal exposure time of TS tests was shorter than that of FCTF tests, a dense microstructure with coalescence of cracks rather than splat boundaries and tiny pores was achieved, which increased hardness values. The hardness values after JEST tests were significantly increased rather than those after the FCTF and TS tests. This is mainly due to the higher testing temperature and direct flame exposure in JETS tests. The surface of TBC was exposed to high temperature flame of 1400 °C, resulting in severe sintering effect. One other explainable mechanism is similar with the microstructure difference after the FCTF and

TS tests, based on microstructure evolution. Consequentially, the microstructure evolution supports the observed mechanical property in the thermal fatigue and shock environments. The composite coatings with the double buffer layers can prevent a catastrophic failure with a superior thermal durability.

## 4. Conclusions

New La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> (LZO) based TBC architectures were designed using the blended LZO and 8YSZ feedstock powders and buffer layers, and prepared using air plasma spray method. The microstructure, thermal durability, and mechanical properties were systematically investigated. The conclusions are summarized below.

- (1) DLC TBC with blended 50:50 volume ratio of LZO and 8YSZ powders and with double buffer layers showed the most outstanding thermal durability among all samples in the FCTF, TS, and JETS tests. The improved thermal durability is due to the reduced thermal and residual stresses at the interfaces and stress dissipation in the composite top coat.
- (2) For application with mildly slow cooling rates similar to FCTF test, the thermal durability in SLC TBCs is more effectively enhanced by controlling composite ratio in the blended powder, which shows a better thermal durability than introducing a single buffer layer. This is due to the fact that, in FCTF tests, the interfacial residual stress has sufficient time to dissipate into a thick composite top coat, but is saturated in a thin buffer layer.
- (3) For applications with fast cooling rates similar to the TS and JETS tests, the thermal durability can be effectively improved by introducing buffer layer than composite top coat. Buffer layer provides fast localized stress relief due to its high strain compliance.

By introducing buffer layer at the interface between the top and bond coats, the thermal and residual stresses could be diffused quickly. The partial delamination of top coat in DLC TBC with double buffer layers would be caused by the coalescence and growth of horizontal cracks.

- (4) The vertical cracks in DLC TBCs which were generated during JETS tests could release the thermal and residual stresses induced by CTE mismatch.
- (5) The measured elastic moduli and hardness values are not significant different in all samples, indicating that the influence of continuity or discontinuity could be negligible for lifetime performance in thermal exposure tests.
- (6) Microstructure design through blending two feedstock powders and introducing buffer layer enhanced thermal durability. Thermal durability of LZO-based TBC can be improved through combining graded composite top coat and buffer layer. This research can provide important microstructure design information for future advanced TBC design.

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#### **Table captions**

Table 1. Summary of the numbers of cycle-to-failure and surface status in each thermal exposure tests.

#### **Figure captions**

Fig. 1. Schematic diagram of microstructure design for the sing layer and double layer TBCs. Fig. 2. Cross-sectional microstructure, element analysis results, and mechanical properties of as-coated TBCs: (A) SCL TBC of 50:50 composite without buffer layer, (B) BCL TBC of 50:50 composite with buffer layer, (C) SCL TBC of 25:50 composite without buffer layer, (D) BCL TBC of 50:50 composite with double buffer layer. Each number indicates microstructure, element analysis results, and mechanical properties.

Fig. 3. Microstructure after FCTF tests: (A) SCL TBC of 50:50 composite without buffer layer, (B) BCL TBC of 50:50 composite with buffer layer, (C) SCL TBC of 25:50 composite without buffer layer, (D) BCL TBC of 50:50 composite with double buffer layer.

Fig. 4. Microstructure after TS tests: (A) SCL TBC of 50:50 composite without buffer layer, (B) BCL TBC of 50:50 composite with buffer layer, (C) SCL TBC of 25:50 composite without buffer layer, (D) BCL TBC of 50:50 composite with double buffer layer. Each number indicates the low and high magnification microstructures, respectively.

Fig. 5. Microstructure after JETS tests: (A) SCL TBC of 50:50 composite without buffer layer, (B) BCL TBC of 50:50 composite with buffer layer, (C) SCL TBC of 25:50 composite without buffer layer, (D) BCL TBC of 50:50 composite with double buffer layer. Each number indicates the low and high magnification microstructures, respectively. Arrows indicate vertical cracks generated during JETS tests.

Fig. 6. Cross-sectional microstructures of BCL TBC of 50:50 blended powder with double

buffer layer before and after thermal exposure tests: (A) as-coated, (B) after FCTF test, (C) after TS test, and (D) after JETS test. Each number indicates the low and high magnification microstructures, respectively.

Fig. 7. Vickers hardness values of BCL TBC of 50:50 blended powder with double buffer layer before and after thermal exposure tests

Sample species	TCFT test/Status	TS test/Status	JETS test/Status
(A) SLC TBC (50% LZO : 50 % YSZ in volume)	540 cycles (11,340 EOH)/ Fully delaminated	10 cycles/ Fully delaminated	70 cycles/ Fully delaminated
(B) DLC TBC (50% LZO : 50 % YSZ in volume) with single buffer layer	768 cycles (16,128 EOH) / Fully delaminated	29 cycles/ Fully delaminated	2000 cycles/ Sound condition
(C) SLC TBC (25% LZO : 75 % YSZ in volume)	936 cycles (19,656 EOH) / Fully delaminated	14 cycles/ Fully delaminated	1022 cycle/ Fully delaminated
(D) DLC TBC (50% LZO : 50 % YSZ in volume) with double buffer layers	1143 cycles (24,000 EOH)/ Sound condition	54 cycles/ Partially delaminated	2000 cycles/ Sound condition

Table 1. Summary of the numbers of cycle-to-failure and surface status in each thermal exposure tests.