Sintering-induced delamination of thermal barrier coatings by gradient thermal cyclic test

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Abstract
Lifetime is crucial to the application of advanced thermal barrier coatings (TBCs), and proper lifetime evaluation methods should be developed to predict the service lifetime of TBCs precisely and efficiently. In this study, plasma-sprayed YSZ TBCs were subjected to gradient thermal cyclic tests under different surface temperatures, with the aim of elucidating the correlation between the coating surface temperature and the thermal cyclic lifetime. Results showed that the thermal cyclic lifetime of TBCs decreased with the increasing of surface temperatures. However, the failure modes of these TBCs subjected to thermal cyclic tests were irrespective of different surface/BC temperatures, that is, sintering-induced delamination of the top coat. The thickness of thermally grown oxide (TGO) was significantly less than the critical TGO thickness to result in the failure of TBCs through the delamination of top coat. There was no phase transformation of the top coat after failure. In contrast, in the case concerning the top coat surface of the failure specimens, the elastic modulus and microhardness increased to a comparable level due to sintering despite of the various thermal cyclic conditions. Consequently, it is conclusive that the failure of TBCs subjected to gradient thermal cyclic test was primarily induced by sintering during high-temperature exposure. A delamination model with multilayer splats was developed to assist in understanding the failure mechanism of TBCs through sintering-induced delamination of the top coat. Based on the above-described results, this study should aid in facilitating the lifetime evaluation of the TBCs, which are on active service at relatively lower temperatures, by an accelerated thermal cyclic test at higher temperatures in laboratory conditions.

KEYWORDS
layered ceramics, sinter/sintering, thermal barrier coatings (TBC), thermal shock/thermal shock resistance

1 | INTRODUCTION

Thermal barrier coatings (TBCs) are widely used in both aircraft engines and land-based gas turbines to protect metallic components from high-temperature environments.1–3 A typical TBC system consists of a superalloy substrate, a MCrAlY(M=Ni, Co, NiCo, CoNi) or NiAl-based bond coat (BC), a thermally grown oxide (TGO) inevitably grown on BC surface, and an air plasma-sprayed (APS) or electron-beam physical-vapor deposited ceramic top coat (TC).4–6 APS top coat generally presented a lamellar microstructure wherein intersplat pores...
are connected with intrasplat cracks. Consequently, this unique porous structure contributes to approximately half more drop as respect to its corresponding bulk material in the through-thickness direction.\textsuperscript{7} The state-of-the-art top coat TBC material is 7-8 wt% yttria-stabilized zirconia (8YSZ), primarily due to its low thermal conductivity, high toughness, and high coefficient of thermal expansion.\textsuperscript{2}

As a multilayer system with BC, TGO, and top coat stacking sequentially on substrate, the delamination-resistance of TBCs is highly relevant to the performance of all constituent parts. It is believed that the inducements of TBC failure are as follows: (1) the thickening of TGO,\textsuperscript{8-10} (2) the phase transformation induced volume expansion of top coat;\textsuperscript{11,12} (3) the sintering of top coat.\textsuperscript{13} All these three types of failure mechanisms can be attributed to the large strain and stress resulting from the meta-stable phases and microstructures mentioned above.

The thickening of TGO results in a large growth of strain and stress, which could induce TBCs failure. When the TGO thickness increases to above 5.5 \textmu m, the stress in TGO can be as high as 3-4 GPa. Consequently, new cracks meandered through the top coat as well as the TGO and expanded along the interfaces to approximately several hundred micrometers.\textsuperscript{9} Based on mechanics analysis, Rabiei and Evans showed that the cracks in the TBCs as well as in the interface remain isolated with small sizes when the TGO thickness is below a critical value of 5.5 \textmu m. In other words, TBC appears to be irrespective of the thinner TGO. Dong et al.\textsuperscript{8} conducted a gradient thermal cyclic test at a surface temperature of 1150°C and indicated that the thermal cyclic lifetime of TBCs decreases sharply from 2000 cycles to 400 cycles when the TGO thickness increases from 5 to 7.7 \textmu m. This clearly suggested that when the TGO thickness is smaller than the critical value of 6.0 \textmu m, TGO thickening is not dominantly responsible for the TBCs failure.

Phase transformation of top coat could be another reason accounting essentially for the failure of TBCs. YSZ generally exists as three different phases: monoclinic, tetragonal, and cubic depending on the composition and the temperature. During plasma spraying, the stacked splats are formed by successive impaction of molten or partial molten particles on a substrate, following by flattening, rapid solidification, and cooling. As a result, nontransformable tetragonal phase (t'-phase) is often retained in plasma sprayed YSZ coatings at room temperature.\textsuperscript{14,17} However, t'-phase is a thermodynamically metastable phase and would transform to cubic phase (c-phase) and monoclinic phase (m-phase) during long-term thermal exposure.\textsuperscript{18} The phase transformation from t-phase to m-phase accompanies 3%-5% volume expansion, which may cause the failure of TBCs.\textsuperscript{19}

Sintering of the top coat could also be an essential reason for TBCs failure. When TBCs are exposed to high temperature with extended periods, sintering of the top coat would occur inevitably. Both the intersplat pores and intrasplat cracks might be healed up, leading to significant stiffening of the top coat. This has a detrimental effect on the strain tolerance of top coat.\textsuperscript{15,20} In addition, the sintering of top coat also leads to an increase in thermal conductivity, resulting in distinct degradation on the thermal barrier performance.\textsuperscript{14}

Given the fact that TBCs may experience different service conditions under various applications, it is necessary to develop an evaluation approach of the thermal cyclic lifetime by taking the above-described failure modes of TBCs into consideration. Isothermal cyclic test, wherein the temperatures of the top coat and substrate stay the same, is one of the popular approaches to evaluate the lifetime of TBCs. However, the oxidation rate of BC increases dramatically when the test temperature exceeds a certain degree (i.e., 1075°C).\textsuperscript{21,22} Moreover, the isothermal cyclic test cannot embody the fundamental thermal barrier role of TBCs since the effects of all factors on the lifetime are integrated, thus being difficult to separate.\textsuperscript{23} To better simulate the real service condition of TBCs, gradient thermal cyclic tests attract more and more attentions.\textsuperscript{22-25} Given that the thermal cyclic lifetime may be several years or more than thousands of thermal cycles, it could be extremely difficult to evaluate the lifetime of TBCs directly at the real service conditions. Therefore, to develop systematic evaluation methods of TBCs lifetime based on accelerating methods, relationship between the service temperatures and the lifetime of TBCs should be comprehensively established.

In this study, TBC samples were subjected to gradient thermal cyclic test with different surface temperatures. The microstructure, mechanical properties were determined to investigate the cracking behavior. The sintering-induced failure modes were further discussed. These would benefit the development of the failure evaluation of TBCs.

2 EXPERIMENTAL PROCEDURE

2.1 Preparation and characterization of TBCs

Commercially available YSZ powder (Sulzer Metco 204B-NS, −75+45 \textmu m, Westbury, NY, USA) was used to deposit the ceramic top coat by a APS system (GP-80, Jiu-Jiang, China). The morphology of the YSZ powders was shown in Figure 1. The spray parameters were shown in Table 1. The thickness of top coat was approximately 500 \textmu m. Prior to the deposition of the top coat, a MCrAlY BC was deposited on a nickel-based superalloy (Inconel
A commercially available NiCoCrAlTaY powder (Amdry 997, –37+9 μm, Sulzer Metco) was used as the BC material in this study. The BC was deposited to approximately 100 μm by a low pressure plasma spraying (LPPS) system. Table 2 shows the spraying parameters of LPPS. Before the deposition of top coat, the as-prepared samples with bond coat were subjected to preheat-treatment by two different stages, namely, a prediffusion procedure (1080 °C + 4h, O2 ≤ 0.01 ppm) following by a preoxidation procedure (1080 °C + 4h, O2 ≈ 10 ppm).

The cross-sectional microstructure of the coating was investigated using a scanning electron microscope (SEM) system (TESCAN MIRA 3 LMH, Czech Republic). Before obtaining the section of samples for a metallographic examination, the samples were infiltrated with epoxy adhesive under vacuum to protect the coatings from any artifact destruction during the sample preparation. The phase composition of YSZ coatings before and after thermal cyclic test was characterized by a X-ray diffraction (XRD, PANalytical X’pert PRO, Netherlands) system. The porosities of the coatings were determined by image analysis. The cross-sectional Vickers hardness was determined by a micro-Vickers Indenter (BUEHLER MICROMET5104, Akashi Corporation, Japan). The hardness tests were performed at a test load of 300 g and holding time of 30 s. The in-plane Young’s modulus of top coat was measured by a Knoop indentation method. The parameters and the test system were the same with the measurement of micro-hardness.

### 2.2 | Thermal cyclic test for TBCs

Thermal barrier coatings have been widely used to provide thermal insulation to metallic components from the hot gas in gas turbines. The top coats are exposed to extremely high temperature to provide a major reduction on the surface temperature (approximately 100°C to 300°C dependent on various service conditions) of the underlying superalloy. A gas burner test setup was designed for the gradient thermal cyclic test (Xi’an Jiaotong University, China) to simulate the actual service condition of TBCs. Three different combinations of preset temperature, namely, 1250 °C/950 °C, 1300 °C/1000 °C, and 1350 °C/1050 °C were used to carry out the gradient thermal cyclic tests, with the aim of elucidating the correlation between the lifetime of TBCs and exposed temperatures. The temperature combination described above refers the surface temperature of top coat and its corresponding BC temperatures.

In this study, one thermal cycle was defined as follows: 50 s heating, 250 s holding, and 120 s cooling. In detail, the TBC surface was heated by a propane and oxygen flame to the presetting temperatures in 50 s, following by holding for 250 s. While, the backside of the TBC was cooled by compressed air to keep a thermal gradient. Then, the specimen was rapidly cooled down to room temperature in 120 s by compressed air from both the TBC surface and backside.

The temperatures of the specimen at surface and backside were monitored by a noncontact infrared thermometers with wavelengths of 8-14 and 1.6 μm, respectively. The emissivity for YSZ and superalloy substrate was calibrated to be 1 and 0.91 according to their wavelength, respectively. The lifetime of TBCs was defined as the thermal cyclic lifecycles when 10% area delamination of the top coat was observed.
## RESULTS AND DISCUSSION

### 3.1 Lifetime of TBCs

Figure 2 shows the lifetime of TBCs at different combinations of presetting temperature surface temperatures under gradient thermal cyclic test. Two to three samples were tested for each group, and the lifetime is the mean value, the error bars represent standard deviations on sets of 2-3 samples, the error bars represent standard deviations. It can be found that the lifetime decreased with the increase in thermal cyclic temperature. The average lifetime was $121 \pm 7$ cycles when the surface temperature of top coat was $1250^\circ C$. When the surface temperature increased to $1300^\circ C$, the lifetime of TBCs reduced to $91 \pm 6$ cycles. The drop of the lifetime of TBCs was even more severe (i.e., only $21 \pm 3$ cycles) when the surface temperature further increased to $1350^\circ C$. Hence, it is possible to conclude a higher temperature often accelerates the failure of TBCs.

The temperature labels refer to the top coat surface temperature. It was possible to observe a multilayer delamination phenomenon when failure occurred. The delaminated layers had a combined thickness of tens of micrometers and were composed of multiple splats. Figure 3 shows the surface photographs of the TBCs when failure occurs under different combinations of presetting temperature, and the failure parts were marked with dash line. In detail, Figure 3A shows the photograph of a TBC sample after 1 cycle at $1250^\circ C$. It can be found that the sample surface was intact, which is almost the same with the other conditions after the first cycle. After thermal cyclic test for 118 cycles at $1250^\circ C$ (see Figure 3B), 91 cycles at $1300^\circ C$ (see Figure 3C), and 21 cycles at $1350^\circ C$ (see Figure 3D), the delamination area reached about 10% of the sample surface. Therefore, the corresponding cycles were recorded as the lifetimes of TBCs.

### 3.2 Cross-sectional microstructure of TBCs

To understand the multilayered splats delamination phenomenon of the tested TBC samples, the cross-sectional microstructure of YSZ coatings for the failure TBCs were examined by SEM, as shown in Figure 4. It can be seen from Figure 4A-C that the thickness of top coat at some localized regions was smaller than the original 500 $\mu m$, which meant that delamination of the top YSZ coating occurred. In addition, some large lateral cracks can also be observed around the delamination failure area. The thickness of the delaminated layer was tens of micrometers. This is distinctly thicker than the individual splats with only approximately 1-2 $\mu m$. No large cracks were observed around the TGO. Therefore, it is reasonable to believe that the delamination of top coat is essentially responsible for the failure of TBCs at different gradient thermal cyclic temperatures in this study.

### 3.3 TGO thickness

Usually, TGO growth is considered as one of the dominant factors leading to the failure of TBCs. Thus, the TGO growth behavior in the present TBC samples was examined. It was found from Figure 4 that no obvious cracks were observed around the TGO, suggesting that the TGO may not be responsible for the failure of TBCs in this study. As a further step, TGO thickness was determined by the cross-sectional SEM micrographs before and after thermal cyclic test, and

![Figure 2](image2.png) **FIGURE 2** Lifetimes of thermal barrier coatings at different conditions. The temperature labels refer to the top coat surface temperature [Color figure can be viewed at wileyonlinelibrary.com]

![Figure 3](image3.png) **FIGURE 3** Macrographs of the thermal barrier coatings after different cycles to failure under different conditions at high temperatures. (A) 1 cycle at $1250^\circ C$, (B) 118 cycles at $1250^\circ C$, (C) 91 cycles at $1300^\circ C$, (D) 21 cycles at $1350^\circ C$ (c and cs represent cycle and cycles, respectively. Blue dash lines represent the delamination regions) [Color figure can be viewed at wileyonlinelibrary.com]
each value was the mean thickness of 10 different regions of TGO, the error bars represent standard deviations. As shown in Figure 5, the TGO thickness in the as-sprayed TBCs was 0.32 ± 0.04 μm, resulting from the preheat treatment mentioned in Section 2.1 (see Figure 5A). After thermal cyclic test until failure, the TGO slightly thickened with respect to that in the as-sprayed TBC. The thicknesses of TGO was 0.57 ± 0.08 μm, 0.54 ± 0.09 μm, and 0.56 ± 0.13 μm at the corresponding surface temperatures of 1250°C, 1300°C, and 1350°C, respectively. Although the TGO thicknesses after thermal cyclic test were thicker than that of the as-prepared samples, they were significantly smaller than the reported critical value to induce a complete delamination of top coat, that is, 5-6 μm.8,9 It is clear that the slightly thickening of TGO is not the dominant factor responsible for the failure of TBCs in this study.

3.4 | Phase compositions of the top coat

Given the fact that phase transformation might be a reason to induce the spallation of top coat,11 the phase compositions of the top coat after TBC failure were examined, as shown in Figure 6. It can be found that the phase compositions of the YSZ coatings before and after thermal cyclic test were mainly nontransformable t-phase. From the detailed analysis in a diffraction angle range from 27 to 32 degrees, a very little fraction of monoclinic phase can also be detected even for the as-sprayed coatings. However, it seemed that there was no increase in the diffraction peaks of monoclinic phase after gradient thermal cyclic test. It was possible to observe that there was no distinct phase transformation after thermal cyclic test at different test conditions. Therefore, it evidently proved that no phase transformation of the top coat occurred. Consequently, the phase transformation may not be the dominant factor responsible for the failure of TBCs in this study.

3.5 | Sintering of the top coat

During service, high-temperature exposure with extended periods often leads to inevitably sintering of the ceramic top coat.26 The detailed sintering behavior of TBCs has been intensively investigated.15,27 However, different regions of the top coat along through-thickness direction are exposed to different temperatures under gradient thermal cyclic test. The inner temperatures of YSZ coating decrease gradually from surface region to the interface between top coat and BC. To reveal the inhomogeneous sintering behavior across the thickness of the ceramic top coat, the unpeeling cross sections of the ceramic coatings were divided into three regions according to their distance away from the TGO/BC interface. These different regions were labeled as “Out”, “Middle”, and “In” regions, as shown in Figure 7A, the typical cross-sectional SEM images of the unpeeling region of the failure samples (eg, 21cycles at 1350°C). It is known that, air-plasma-sprayed top ceramic coat is typically lamellar structure stacked by a series of splats. However, for the samples after thermal cycling to failure (eg, 21cycles at 1350°C), as shown in Figure 7B near the coating surface, there has been extensive healing of microcracks near the coating surface. Lots of lamellar structure disappears because of sintering. The lamellar structured ceramic coating evolved into a denser one. Figure 7C shows the high-magnified microstructure of the “Middle” region, and there remain some lamellar pores because of lower temperature in this region. As for the “In” region shown in Figure 7D, the coating keeps typical
lamellar structure with lots of lamellar pores and cracks. In cross-sectional images (Figure 7) obtained using backscattered electron detector, pores appear very dark contrast, which permits the pores to be distinguished and quantified by image analysis. Cross-sectional images at 5000× magnification were used to obtain the porosity using image J software, and 10 images were used for each region of the coatings. The detailed porosities of different regions after thermal cycles are shown in Table 3. In the porosity in “In” region decreased from 13.3±1.4% to that of 7.3±1.0% in “Out” region.

Figure 8 shows the high-magnification fracture surface images of the peeling coatings before (A, as-sprayed) and after failure (B, 21 cycles@1350°C) near coating surface. It can be seen from Figure 8, grain growth is obvious, but the specific grain size cannot be accurate measured because of the shading of adjacent grains.

Figure 9 shows the elastic modulus (E) and Vickers hardness (Hv) of different regions before and after the thermal cyclic test. The Knoop indentation method was used to measure the elastic modulus of ceramic coatings. By measuring the elastic recovery of the residual surface impression of the indentation long and short diagonals (with half lengths a' and b', respectively), the modulus E can be estimated by Equation 1.28

$$E = \alpha \times \left( \frac{b}{a} - \frac{b'}{a'} \right)^{-1} \times Hv$$

where \(\alpha\) is a constant (\(\alpha=0.45\)), a and b are the half lengths of the long and short diagonals before elastic recovery,
b/a=1/7.11 for a perfect indenter, $H_v$ is the Vickers hardness. For each result in Figure 9, average value of 15 valid indentations was measured and calculated by Equation 1, and the error bars represent standard deviations on sets of 15 measurement points. Since the microstructure and mechanical properties of the as-sprayed top coat were independent with regions, the elastic modulus at different regions was comparable, that is, 62 GPa. However, after gradient thermal cyclic test, the elastic modulus presented a differential distribution. Although the elastic modulus not completely presented a gradient distribution, but the observed trend of increasing stiffness from “in” to “out” regions after thermal cycle is nevertheless quite clear, since the local temperatures in top YSZ coat decreased from the “Out” region to the “In” region. In addition, the coating hardness also presented the similar tendency upon gradient thermal cyclic test as respect to the elastic modulus. Since substrate thickness is much larger than top coat, the thermal mismatch induced strain ($\varepsilon$) and stress ($\sigma$) during thermal cyclic test can be roughly calculated by Equations 2 and 3 as follow,

\[
\varepsilon = (\alpha_{\text{Sub}} - \alpha_{\text{TC}}) \times \Delta T \tag{2}
\]

\[
\sigma = E \times \varepsilon = E \times (\alpha_{\text{Sub}} - \alpha_{\text{TC}}) \times \Delta T \tag{3}
\]

where $\Delta T$ is the temperature difference during thermal cycle, $\alpha_{\text{TC}}$ and $\alpha_{\text{Sub}}$ is the thermal expansion coefficient of top coat and substrate, respectively.

In this study, TBCs experienced thermal cycles including a temperature increasing ($\Delta T$) of >1000°C and a temperature decreasing as well. The thermal expansion coefficient of YSZ ($\alpha_{\text{TC}}$) is about $11 \times 10^{-6}$/K, the superalloy($\alpha_{\text{Sub}}$) is about $15 \times 10^{-6}$/K, and the elastic modulus is as high as 90 GPa, the mismatch stress is estimated to be >360 MPa. It has been reported that, for YSZ coating on a zirconia substrate (for examining sintering stress), the in-plane stress induced by sintering is 0.3 MPa. Therefore, the mismatch stress is much greater than sintering stress. Sintering induces the increase in elastic modulus further increase the mismatch stress, and the failure of TBCs in this study is dominated by mismatch induced stress strengthened by sintering hardening.

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**TABLE 3** Porosity of different regions across thermal barrier coating thickness

<table>
<thead>
<tr>
<th>Porosity (%)</th>
<th>As-sprayed</th>
<th>After failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Out</td>
<td>13.4±0.8</td>
<td>7.3±1.0</td>
</tr>
<tr>
<td>Middle</td>
<td>13.4±0.8</td>
<td>10.8±0.8</td>
</tr>
<tr>
<td>In</td>
<td>13.4±0.8</td>
<td>13.3±1.4</td>
</tr>
</tbody>
</table>

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**FIGURE 7** High-magnification images of different regions after failure (21 cycles at 1350°C) [Color figure can be viewed at wileyonlinelibrary.com]
Although the thermal cyclic lifetime and thereby the high-temperature exposure durations for these three test conditions were significantly different, the measured mechanical properties of the top coat were almost the same. The elastic modulus of the “Out” regions reached approximately 90 GPa at different thermal cyclic conditions. Similarly, the corresponding “In” and “Middle” regions at different conditions were comparable to each other. This was also applicable to the Vickers hardness. Therefore, it clearly suggested that the failure of TBCs occurred through the delamination of top coat, when the elastic modulus of ceramic coatings increased to nearly the same elastic modulus of 90 GPa from the original 60 GPa at as-sprayed state. Owing to the fact that a higher temperature often accelerates the sintering process, the thermal cyclic lifetime is shortened. In brief, a certain sintering level could be the essential reason responsible for the failure TBCs under the gradient thermal cyclic test.

The sintering process of TBCs in different conditions can be summarized schematically in Figure 10. Based on the sintering of top coat surface induced delamination, a lifetime assessment method for TBCs could be established. For example, the initial elastic modulus was 60 GPa at the as-sprayed state, while it finally reached about 90 GPa near the surface regions after different thermal cyclic tests when failure occurred. The sintering process could be associated with the increase in elastic modulus at top coat surface from 60 GPa to 90 GPa. At 1250°C, the sintering-induced failure finished in 121 thermal cycles. However, at higher temperatures, like 1300°C or even 1350°C, the sintering process is accelerated. Namely, the sintering effect on failure at a higher temperature in each cycle is larger than that at a relatively lower temperature. It has been reported that, for YSZ top coats heat treated 10 h (comparable in this study) at 1400°C, the elastic modulus (obtained via four-point bending) reached to 60-70 GPa, considering that the elastic modulus of their as-sprayed coating is about 17 GPa, the stiffness data in this study are reasonable.

This means that the lifetime of TBCs exposed at relatively lower temperatures could be predicted by an accelerated thermal cyclic test at higher temperatures with acceptable durations in laboratory conditions.

### 3.6 Multisplat layered delamination analysis

After thermal cyclic tests at different conditions, at least three aspects can be clearly distinguished. To begin with, the maximum thickness of TGO is about 0.57 μm, being far less than the critical value of 5-6 μm to induce a completely delamination of the top coat. Secondly, the top
ceramic coatings showed no obvious phase transformation. Third, dependent with the regions along through-thickness direction, high-temperature exposure induced distinct changes in mechanical properties of the top coat. The significantly stiffened top coat dominates the delamination of TBCs.

To sum up, a model concerning the cracking delamination could be developed, as shown in Figure 11. The APS ceramic coatings typically exhibit a lamellar structure through the successive stacking of individual splats (see Figure 11F). From a microscopic perspective, the coating exhibits a connected pore network consisting of intersplat pores and intrasplat cracks, as shown in the inset of Figure 11. During the rapid cooling stage, the elastic modulus and temperature gradient may make the coating surface region shrink rapidly. The localized shrinkage produces large tensile stress on top coat. In the case of a pre-existing crack located near coating surface, as shown in Figure 11B, the crack firstly connects with the coating surface in the weak bonding part due to sintering effect, as shown in Figure 11C. Following on, the crack continues to extend, as shown in Figure 11D. However, it is worth noting that there are two types of routes for crack extension. One is the propagation along intersplat pores, which results in the delamination of individual splats. The other one is multisplats layered delamination, which results in the delamination of a layer with a thickness of tens of micrometers, as shown in Figure 11E. Therefore, it is necessary to understand the competition between these two types of delamination modes.

For both intersplat pores and large lateral pores, as shown in Figure 11F, the crack propagation is a result of pore boundary tearing under the shear stress induced by surface shrinkage. The crack propagation occurs when the strain energy release rate is larger than the critical interfacial crack energy or fracture toughness. Therefore, the competition between the cracking driving force and the cracking resistance is discussed from two aspects in following parts, that is, strain energy release rate and localized fracture toughness of the top coat.

The strain energy release rate, \( G_h \), can be obtained as follows:

\[
G_h = \int_0^h \frac{E_x \varepsilon^2}{2(1-v)} dx \tag{4}
\]

where \( h \) is the distance from the top coat surface to the crack tip (either large lateral crack shown in Figure 11F or the intersplat pores), \( E_x \) is the local elastic modulus at the crack tip, \( \varepsilon \) is the strain, \( v \) is the Poisson’s ratio.

It can be seen from Equation 4 that \( G_h \) increases with the increase of \( h \). Furthermore, the sintering-induced gradient distribution of mechanical properties makes the \( G_h \) increase more significantly. Therefore, the energy release rate for the layered crack tip (see Figure 10C) is significantly greater than the individual intersplat pore tip. The interfacial crack energy or fracture toughness (\( G_{hc} \)) is also locally enhanced by the sintering-induced gradient mechanical properties. As discussed in the Section Acknowledgments above, the elastic modulus of top coat increased because of high-temperature sintering. Moreover, the elastic modulus increased faster at the regions closer to the top coat surface due to the temperature gradient across the
coating thickness. Although it is quite difficult to directly obtain the local interfacial crack energy of the relatively thinner top coat with different values across the coating thickness, it is reasonable to state that, the local fracture toughness of the top coat increases faster at the regions closer to top coat surface due to the temperature gradient across the coating thickness. Consequently, the cracking becomes more readily with the increase in distance from top coat surface.

Based on the analysis above, it is clear that the multisplats layered delamination (see Figure 11) is preferential because of both relatively larger strain energy release rate and relatively lower interfacial crack energy. The mechanics analysis further suggests that the lifetime of TBCs exposed at relatively lower temperatures could be evaluated by higher temperatures, with the aim to accelerate the sintering-induced failure procedure under thermal cyclic tests in laboratory conditions.

4 | CONCLUSIONS

In this study, gradient thermal cyclic tests with different combinations of surface/BC temperatures were conducted, with the aim to simulate and evaluate the lifetime of APS TBCs in laboratory conditions. The following conclusions could be obtained from this work:

1. The thermal cyclic lifetime decreased with the increase in surface temperature of top coat from 1250°C to 1350°C. However, these samples under different experimental conditions exhibited a same failure mode, that is, multilayered splats delamination.

2. The maximum thickness of TGO in failed TBC samples was 0.57 μm, being far less than the critical value causing the failure of TBCs through complete delamination of the top coat. In addition, there was no obvious phase transformation during thermal cyclic test. Therefore, the TGO thickening and phase transformation were not the essential reasons responsible for the failure of TBC in this study.

3. The mechanical properties present a gradient distribution across the thickness of top coat, due to the temperature gradient in burner thermal cycling. In addition, despite of the different temperatures at top coat surface, the mechanical properties increased to a comparable level when the failure of TBCs occurs. Therefore, sintering dominates the failure of TBCs in this study.

4. A model concerning the multisplats layered delamination was proposed to explain the failure behavior of top coat. Due to the shrinking-stress generated at the top coat surface, the lateral cracks connect with coating surface, following by propagation until the final multisplats layered delamination.

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